
American Oil Chemist's Society

45th

FALL MEETING



PROGRAM



OCTOBER 3-6, 1971

Chalfonte-Haddon Hall Hotel

Atlantic City, New Jersey

MONDAY AFTERNOON—OCTOBER 4

1:30 P.M.—Vernon Room

SESSION A—SYMPOSIUM: LIPIDS IN METABOLIC STRESS

Chairmen—Donald Therriault and B. Issekutz

- 1:30 1. FREE FATTY ACID METABOLISM DURING STRESS: EXERCISE, ACUTE COLD EXPOSURE AND ANAPHYLACTIC SHOCK
Pavle Paul and W.L. Holmes, Lankenau Hospital
- 2:00 2. LIPID METABOLISM DURING COLD EXPOSURE AND DURING COLD ACCLIMATION
Jean Himms-Hagen, University of Ottawa
- 2:30 3. EFFECT OF REGULAR MUSCULAR ACTIVITY ON LIPID METABOLISM IN RATS
Vlado Simko, Cornell University
- 3:00 4. HUMAN SKELETAL MUSCLE ADAPTATIONS TO EXERCISE STRESS
T.E. Morgan, L.A. Cobb, Russell Ross, University of Washington; F.A. Short, Swedish Hospital
- 3:30 5. CAUSE OF EXHAUSTION IN PROLONGED EXERCISE AND THE EFFECT OF METHYL-PREDNISOLONE (MP)
B. Issekutz, Jr., Dalhousie University
- 4:00 6. INTRAMUSCULAR ENERGY SOURCES IN DOGS DURING HEAVY PHYSICAL WORK
D.G. Therriault, G.A. Beller, J.A. Smoake and H.L. Harley, U.S. Army Research Institute of Environmental Medicine
- MONDAY AFTERNOON—OCTOBER 4
1:30 P.M.—Pennsylvania 1 Room
SESSION B—LIPID ANALYSIS
Chairman—Frank Luddy
- 1:30 7. GAS LIQUID CHROMATOGRAPHIC FRAGMENTATION OF SEED OIL DIGLYCERIDES ON POLYESTER LIQUID PHASES
A. Kuksis, O. Stachnyk and L. Marai, University of Toronto
- 1:50 8. A RAPID METHOD FOR DETERMINING ANTI-OXIDANT ACTIVITY AND FAT STABILITY
D.L. Berner, J.A. Conte and G.A. Jacobson, Campbell Institute for Food Research
- 2:10 9. EFFECTS OF SYNERGIST AND TEMPERATURE ON ANTI-OXIDANT FUNCTION
D.L. Berner and G.A. Jacobson, Campbell Institute for Food Research

- 2:30 10. STUDIES ON THE BIOSYNTHESIS OF CHLOROSULFOLIPIDS
T.H. Haines and Carolyn Mooney, City College of New York

- 2:50 11. SOLUBILIZATION OF FLAVOR MOLECULES IN LIQUID AND LIQUID CRYSTALLINE PHASES OF WATER, MONO- AND TRIGLYCERIDES
Stig Friberg and Inga Wilton, Swedish Institute of Surface Chemistry

- 3:10 12. LIPID AND PROTEIN COMPETITIVE REACTIONS IN DRY SYSTEMS
G.V. Rao, Far-Mar-Co, Inc., and L.R. Dugan, Jr., Michigan State University

- 3:30 13. STEROL ADDITIVES AS POLYMERIZATION INHIBITORS FOR FRYING FATS
R.J. Sims, J.A. Fioriti and M.J. Kanuk, General Foods Corporation

- 3:50 14. THE FATE OF TRIGLYCERIDE HYDROPEROXIDES DURING CATALYTIC HYDROGENATION
N.E. Bednarczyk, Nabisco Research Center, and S.S. Chang, Rutgers University

- 4:10 15. FUNGI PATHOGENIC TO INSECTS: THE NEUTRAL AND POLAR LIPIDS OF ENTOMOPHTHORA CORONATA
T.E. Bruszewski and R.O. Mumma, Pennsylvania State University

- 4:30 16. THE STRUCTURE OF AN ORNITHINE-CONTAINING LIPID FROM THIOBACILLUS THIOOXIDANS
H.W. Knoche, University of Nebraska, and J.M. Shively, Glemson University

- 4:50 17. THE FATTY ACIDS OF GLOMERELLA CINGULATA
Gregory E. Anekwe and Bharat C. Dubal, Tuskegee Institute

- 5:10 18. LONG CHAIN HYDROCARBON COMPOSITION OF CITRUS JUICE SACS
Steven Nagy and Harold Nordby, U.S. Fruit and Vegetable Products Laboratory

MONDAY AFTERNOON—OCTOBER 4

1:30 P.M.—Pennsylvania 3 Room

SESSION C—SYMPOSIUM: FATS AND OILS IN THE FOOD INDUSTRY

Chairman—T.J. Weiss

- 1:30 19. MARGARINE AND MARGARINE OILS
L.H. Wiedermann, Kraftco Corporation
- 2:00 20. CHIP FRYING OILS

R.L. Vandaveer, D.E. Banks and T.P. Dornseifer, Frito-Lay, Inc.

- 2:30 21. FAT SYSTEMS FOR BAKERY PRODUCTS
R.R. Baldwin, P. Baldry and R.G. Johansen, ITT Continental Baking Company, Inc.

- 3:00 22. FATS AND PROCESSES USED IN MANUFACTURING CHOCOLATE AND COMPOUND COATINGS: A REVIEW
G.M. Johnston, M&M/Mars, Division of Mars, Inc.

- 3:30 23. FOOD EMULSIFIERS: SCIENCE AND ART
N.H. Nash and L.M. Brickman, Drew, Division of Pacific Vegetable Oil Corporation

- 4:00 24. ANTI-OXIDANTS FOR FOOD FATS AND OILS
E.R. Sherwin, Eastman Chemical Products, Inc.

- 4:30 25. PRODUCT DEVELOPMENT AS INFLUENCED BY THE FEDERAL FOOD, DRUG AND COSMETIC ACT
R.J. Meyer, Food and Drug Administration

MONDAY AFTERNOON—OCTOBER 4

1:30 P.M.—Pennsylvania 2 Room

SESSION D—CHEMICAL DERIVATIVES

Chairman—Gerhard Maerker

- 1:30 26. KINETICS OF COPPER CHROMITE HYDROGENATION IN SOYBEAN AND LINSEED OILS: EFFECT OF PRESSURE
P.Y. Vignerson, S. Koritala, R.O. Butterfield, and H.J. Dutton, Northern Regional Research Laboratory

- 1:50 27. HYDROGENATION AND DEUTERATION OF CONJUGATED ISOMERS OF LINOLEATE AND LINOLENATE WITH PALLADIUM, PLATINUM, NICKEL AND LINDLAR CATALYSTS
Sambasivarao Koritala, C.R. Scholfield, E. Selke and H.J. Dutton, Northern Regional Research Laboratory

- 2:10 28. ALKOXYLATION OF N-ALKYL ETHYLENE DIAMINE
W. Rosenblatt, A. Stefcik, C. Bluestein and Y.K. Kim, Witco Chemical Corporation

- 2:30 29. EPIMINOSTEARATES FROM UNSATURATED FATS
T.A. Foglia and P.A. Barr, Eastern Regional Research Laboratory

- 2:50 30. PREPARATION AND REACTIONS OF 1-AZIRENES DERIVED FROM FATTY ACIDS

- T.A. Foglia, P.A. Barr and G. Maerker, Eastern Regional Research Laboratory
- 3:10 31. DIRECT ESTERIFICATION OF SODIUM CIS 9-(10)-EPIMINOOCYDDECANOATE
A. Bilyk, A. Eisner and G. Maerker, Eastern Regional Research Laboratory
- 3:30 32. ESTERIFICATION AND TRANSFERIFICATION OF 9-(10)-CARBOXYSTEARIC ACID AND ITS METHYL ESTERS: KINETIC STUDIES
E.J. Dufek, R.O. Butterfield and E.N. Frankel, Northern Regional Research Laboratory
- 3:50 33. POTENTIAL INDUSTRIAL DERIVATIVES OF METHYL 9-(10)-FORMYLSTEARATE: ACETALS, ETHERS AND POLYMERS
R.A. Awl, E.N. Frankel, E.H. Pryde and J.C. Cowan, Northern Regional Research Laboratory
- 4:10 34. PLASTICIZING ACTION AND CRYSTALLINITY IN POLYMERS INCORPORATING FATTY MONOMERS
A.N. Wrigley and E.F. Jordan, Jr., Eastern Regional Research Laboratory
- 4:30 35. POLYESTERAMIDES FROM LINSEED AND SOYBEAN OILS FOR PROTECTIVE COATINGS: ALKYD-TYPE POLYMERS
Wilma J. Schneider, L.E. Gast and J.C. Cowan, Northern Regional Research Laboratory
- 4:50 36. KINETICS OF THE REACTION OF ISOPROPENYL ACETATE AND STEARIC ACID TO FORM ISOPROPENYL STEARATE
M. Kozempel, J.C. Craig, Jr., H.I. Sinnamon and N.C. Aceto, Eastern Regional Research Laboratory
- 5:10 37. POTENTIAL LUBRICANTS FOR CONTINUOUS CASTING OF STEEL: POLYOL ESTERS OF PARTIALLY HYDROGENATED SOYBEAN ACIDS
E.W. Bell, J.C. Cowan and L.E. Gast, Northern Regional Research Laboratory
- MONDAY AFTERNOON—OCTOBER 4**
1:30 P.M.—Garden Room
- SESSION E-1—SYMPOSIUM: NEW EDIBLE OILS**
- 1:30 38. SUNFLOWERS: AMERICA'S MOST NEGLECTED CROP
J.A. Robertson, Russell Agricultural Research Center
- 2:00 39. SAFFLOWER: AN ANCIENT OIL CROP FINDING ITS PLACE IN TODAY'S MARKETS
- Glenn Fuller, Western Regional Research Laboratory
- 2:30 40. SESAME: CURRENT KNOWLEDGE OF COMPOSITION AND USE
C.K. Lyon, Western Regional Research Laboratory
- 3:00 41. PRESENT STATUS OF GENETIC RESEARCH ON SESAME
D.M. Yermamos, University of California
- MONDAY AFTERNOON—OCTOBER 4**
3:30 P.M.—Garden Room
- SESSION E-2—SYMPOSIUM: STATISTICS IN RESEARCH, QUALITY CONTROL AND MANUFACTURING**
- Chairman—H.L. Andrews
- 3:30 42. RESPONSE SURFACE METHODS FOR EXPLORING MULTIFACTOR SPACE
L.B. Hare, Hunt Foods and Industries
- 4:15 43. DESIGN OF EXPERIMENTS WITH MIXTURES
R.D. Snee, E.I. DuPont de Nemours & Company
- 4:45 44. DESIGN FOR FACTORIAL EXPERIMENTS
H.P. Andrews, Rutgers University
- TUESDAY MORNING—OCTOBER 5**
9:00 A.M.—Vernon Room
- SESSION F—SYMPOSIUM: BIOCHEMICAL AND CLINICAL ASPECTS OF THE USE OF FAT EMULSIONS IN PARENTERAL NUTRITION**
- Chairman—Werner Fekl
- 9:00 45. UPTAKE OF BLOOD TRIGLYCERIDES BY VARIOUS TISSUES
R.O. Scow, National Institutes of Health
- 9:30 46. EXPERIENCES WITH FAT EMULSIONS IN LONG TERM COMPLETE PARENTERAL NUTRITION
H.C. Meng, Vanderbilt University Medical School
- 10:00 47. SIGNIFICANCE OF THE RATIO OF FAT, CARBOHYDRATES AND AMINO ACIDS IN COMPLETE PARENTERAL NUTRITION
K.L. Heller, University of Frankfurt/Main, West Germany
- 10:30 48. DEPLETION OF ESSENTIAL FATTY ACIDS AFTER LONG TERM FAT FREE PARENTERAL NUTRITION
- D.A. Coats, Royal Australasian College of Surgeons
- 11:00 49. THE USE OF FAT EMULSIONS FOR THE TOTAL INTRAVENOUS FEEDING OF THE INFANT
A.G. Coran, National Naval Medical Center
- 11:30 50. THE ROLE OF FAT AS CALORIE SOURCE IN PARENTERAL NUTRITION
K. Schultis, University of Giessen and "Rastpfuhl" Hospital, West Germany and H. Beisbarth, 5 1/2 Langemarckplatz, 852 Erlangen, West Germany
- TUESDAY MORNING—OCTOBER 5**
8:30 A.M.—Pennsylvania 1 Room
- SESSION G—GENERAL: ANALYTICAL TECHNIQUES**
- Chairman—David L. Berner
- 8:30 51. CHECKING TEMPERATURES IN LABORATORY INSTRUMENTATION
W.H. King, Southern Marketing and Nutrition Research Division
- 8:50 52. NMR CHEMICAL SHIFT REAGENTS IN STRUCTURE DETERMINATION OF LIPID DERIVATIVES: I. BACKGROUND AND PRINCIPLES
D. Swern and J.P. Wineburg, Temple University
- 9:10 53. NMR CHEMICAL SHIFT REAGENTS IN STRUCTURAL DETERMINATION OF LIPID DERIVATIVES: II. SCOPE AND LIMITATIONS
J.P. Wineburg and D. Swern, Temple University
- 9:30 54. TRANSIENT NMR SIGNALS FROM SOLID AND LIQUID FATS
G.A. Pearsyn, The Praxis Corporation
- 9:50 55. DETERMINATION OF RESIDUAL ACETONE AND ISOPROPANOL IN OILSEED MEALS AND FLOURS BY DIRECT GAS CHROMATOGRAPHY
H.P. Dupuy, and Sara P. Fore, Southern Marketing and Nutrition Research Division
- 10:10 56. DETERMINATION OF MESITYL OXIDE AND DIACETONE ALCOHOL IN OILSEED MEALS AND FLOURS
Sara P. Fore, H.P. Dupuy and E.T. Rayner, Southern Marketing and Nutrition Research Division
- 10:30 57. A RAPID METHOD FOR DETERMINING PROTEIN IN SOYBEAN MEAL
B.D. Deacon, U.S. Department of Agriculture
- 10:50 58. COMPOSITIONAL STUDIES ON HYDRO-

- GENERATED OILS CONTAINING C-22 FATTY ACIDS**
H.B.S. Conacher and B.D. Page, Department of National Health and Welfare
- 11:10 59. **GAS CHROMATOGRAPHIC DETERMINATION OF CIS AND TRANS IN MONOENE AND DIENE FATTY ESTERS**
E.A. Emlen, Northern Regional Research Laboratory
- 11:30 60. **CHAR-ASHING OF GLYCERIDE OILS BEFORE DETERMINATION OF THEIR COPPER AND IRON CONTENTS BY ATOMIC ABSORPTION**
C.D. Evans, G.R. List and L.T. Black, Northern Regional Research Laboratory
- TUESDAY MORNING—OCTOBER 5**
8:45 A.M.—Garden Room
- SESSION H—SYMPOSIUM: FLAVOR RESEARCH IN FATS AND FAT BEARING FOODS (I)**
Chairman—T.H. Smouse
- 8:45 **INTRODUCTORY REMARKS**
T.H. Smouse, Clayton Research Center
- 9:00 61. **PHYSIOCHEMISTRY OF FLAVOR**
R.J. Henkin, National Institute of Health
- 9:30 62. **APPLYING SUBJECTIVE FLAVOR TESTS**
Jean F. Caul, Kansas State University
- 10:00 63. **FLAVOR POTENTIATION**
R.L. Wasson, Monsanto Company
- 10:30 64. **GRAS LIST REGULATIONS**
E.J. Merwin, McCormick & Company, Inc.
- 11:00 65. **SOYBEAN PROTEIN FLAVOR COMPONENTS: A REVIEW**
J.C. Cowan, J.J. Rackis and W.J. Wolf, Northern Regional Research Laboratory
- 11:30 66. **ENZYMATICALLY PRODUCED FLAVORS FOR FATTY SYSTEMS**
J.H. Nelson, Dairyland Food Labs., Inc.
- TUESDAY MORNING—OCTOBER 5**
9:00 A.M.—Solarium
- SESSION I—SYMPOSIUM: LIPIDS OF AQUATIC ANIMALS (I)**
Chairman—D.C. Malins
- 9:00 **INTRODUCTORY REMARKS**
D.C. Malins, National Oceanic and Atmospheric Administration
- 9:05 67. **AQUATIC FATTY ACIDS AND MAN**
R.G. Ackman, Fisheries Research Board of Canada Halifax Laboratory
- 9:35 68. **METABOLISM OF NEUTRAL LIPIDS IN SQUALUS ACANTHIAS**
J.R. Sargent, R.R. Gatten and R. McIntosh, University of Aberdeen, Scotland
- 10:05 69. **METABOLISM OF OLEYL ALCOHOL AND OLEYL ACID IN FISH**
D.M. Sand, C.H. Rahn and H. Schlenk, Hormel Institute
- 10:35 70. **THE MOLECULAR STRUCTURE OF TRIACYL GLYCEROLS RESISTANT TO PROCINE PANCREATIC LIPASE**
Usha Varanasi and D.C. Malins, National Oceanic and Atmospheric Adm. National Marine Fisheries Service, Pioneer Research Laboratory
- 11:05 71. **STUDIES ON HIGHLY UNSATURATED TRI-GLYCERIDES AND PHOSPHOLIPIDS**
N.R. Bottino, Texas A&M University
- TUESDAY AFTERNOON—OCTOBER 5**
1:30 P.M.—Vernon Room
- SESSION J—SYMPOSIUM: NUTRITIONAL PROPERTIES OF DIETARY FATS**
Chairmen—H. Kaunitz and K. Lang
- 1:30 72. **THE TOXICOLOGY OF FATS AND OILS**
K. Morgareidge, Food and Drug Research Laboratories, Inc.
- 2:00 73. **INFLUENCE OF NINE DIETARY FATS ON CARDIAC FIBROSIS AND CALCIFICATION IN RATS**
H. Kaunitz and Ruth E. Johnson, Columbia University
- 2:30 74. **A POSSIBLE METHOD FOR THE DETECTION OF NITROSAMINES IN FATS AND OILS**
Liselotte Hedler, University of Freiburg, West Germany
- 3:00 75. **CARDIAC LIPIDS IN RATS AND GERBILS FED OILS CONTAINING C-22 FATTY ACIDS**
Joyce L. Beare-Rogers, Food and Drug Research Laboratories
- 3:30 76. **LONG TERM NUTRITIONAL STUDIES OF FRYING FATS**
Knoerd Lang, University of Mainz
- 4:00 77. **THE PHYSIOLOGICAL EFFECTS OF WHEAT GERM OIL ON HUMANS IN EXERCISE**
T.K. Cureton, Jr., University of Illinois
- 4:30 78. **DIETARY PROTEIN, MEDIUM CHAIN TRIGLYCERIDES AND CHOLESTEROL METABOLISM**
H. Fisher and J.J. Kenney, Rutgers University
- TUESDAY AFTERNOON—OCTOBER 5**
1:30 P.M.—Windsor Room
- SESSION K—SYMPOSIUM: LIPID METHODOLOGY: I. ISOTOPIC TRACERS IN BIOLOGY**
Chairman—F.L. Snyder
- 1:30 79. **RADIOASSAY OF ³H, ¹⁴C, ³⁵S AND ³²P: LIQUID SCINTILLATION**
D.L. Horrocks, Argonne National Laboratory
- 2:00 80. **HEALTH PHYSICS PRACTICES IN LABORATORIES USING ³H, AND ¹⁴C-LABELLED TRACERS**
R.J. Cloutier and J.D. Berger, Oak Ridge Associated Universities
- 2:30 81. **STABLE ISOTOPE TRACER METHODOLOGY: INSTRUMENTATION AND APPLICATION TO HUMAN METABOLIC STUDIES**
P.D. Klein, and Patricia A. Szczepanik, Argonne National Laboratory
- 3:00 82. **THE MEASUREMENT OF LIPID TURNOVER IN NIQUE MEMBRANES BY A DOUBLE ISOTOPE TECHNIQUE**
Ten-ching Lee and Fred Snyder, Oak Ridge Associated Universities
- 3:30 83. **PREPARATION, ANALYSIS AND RADIOLYSIS OF LABELED COMPOUNDS**
L.E. Geller, New England Nuclear Corporation
- 4:00 84. **PRINCIPLES IN THE SYNTHESIS OF ¹⁴C-LABELED LIPIDS**
Claude Plantadosi, University of North Carolina
- 4:30 85. **TRITIUM LABELING OF LIPIDS**
T.L. Mounts, Northern Regional Research Laboratory
- TUESDAY AFTERNOON—OCTOBER 5**
1:30 P.M.—Garden Room
- SESSION L—SYMPOSIUM: FLAVOR RESEARCH IN FATS AND FAT BEARING FOODS (II)**
Chairman—T.H. Smouse
- 1:30 86. **USE OF LABELED COMPOUNDS TO STUDY**

- THE MECHANISM OF FLAVOR FORMATION IN OXIDIZING FAT**
S.T. Michalski and E.G. Hammond, Iowa State University
- 2:00 87. VARIOUS INTERACTIONS IN CHOCOLATE FLAVOR**
P.G. Keeney, The Pennsylvania State University
- 2:30 88. OXIDATION OF FISH OILS**
W.W. Nawar, University of Massachusetts and A.C. Noble, University of Guelph
- 3:00 89. THE CORRELATION OF FLAVOR SCORES TO CHEMICAL TESTS FOR A SERIES OF SOYBEAN OILS OF DIFFERENT OXIDATION HISTORIES**
T.H. Smouse, J.E. Covey and J.K. Maines, Anderson Clayton Foods
- 3:30 90. FLAVOR LOSS STUDIES ON HEAT PROCESSED FOODS**
R.J. Eiserle and J.A. Rogers
- 4:00 91. ROOM ODOR EVALUATION OF OILS AND COOKING FATS**
C.D. Evans, Kathleen Warner, G.R. List and J.C. Cowan, Northern Regional Research Laboratory
- 4:20 92. CATALYTIC DECOMPOSITION OF AUTOXIDIZED UNSATURATED FATTY ACIDS**
W.I. Kimoto and A.M. Gaddis, U.S. Department of Agriculture

TUESDAY AFTERNOON—OCTOBER 5

- 1:30 P.M.—Solarium**
- SESSION M—SYMPOSIUM: LIPIDS OF AQUATIC ANIMALS (II)**
- Chairmen—Usha Varanasi and D.C. Malins
- 1:30 INTRODUCTORY REMARKS**
- 1:35 93. COMPARATIVE ASPECTS OF STEROLS OF MARINE INVERTEBRATES**
D.R. Idler, L. Safe and P. Wiseman, Fisheries Research Board of Canada
- 2:05 94. SOME ASPECTS OF PHOSPHOLIPID METABOLISM DURING DEVELOPMENT OF THE BRINE SHRIMP, ARTEMIA SALINA**
R.D. Ewing, Oregon State University, and F.J. Finamore, Oak Ridge National Laboratory
- 2:35 95. STUDIES ON MARINE ORGANISMS: PROBING STRUCTURAL FEATURES OF BIOLOGICAL MEMBRANES WITH NITROXIDES**
W.T. Roubel, Pioneer Research Laboratory
- 3:05 96. HETEROGENEITY OF LIPID COMPOSITION**

- WITHIN THE CEPHALIC MELON TISSUE OF THE PILOT WHALE**
G. Wedmid, C. Litchfield, Rutgers University, R.G. Ackman and J.C. Sipos, Fisheries Research Board of Canada
- 3:35 97. CHOLESTEROL SYNTHESIS AND HYPERBARIC OXYGEN IN SWIMBLADDERS OF ABYSSAL AND SHALLOW FISHES**
C.F. Phleger, Scripps Institute of Oceanography
- 4:05 98. EFFECT OF DIETARY LINOLENIC ACID AND DODECAHEXAENOIC ACID ON GROWTH AND FATTY ACID COMPOSITION OF RAINBOW TROUT (SALMO GARDNERI)**
T.C. Yu, and R.O. Sinnhuber, Oregon State University

WEDNESDAY MORNING—OCTOBER 6

- 9:00 A.M.—Pennsylvania 2 Room**
- SESSION N—SYMPOSIUM: FATS AND OILS IN PHARMACEUTICALS AND COSMETICS**
- Chairman—S.D. Gershon
- 9:00 99. THE ROLE OF FATS AND OILS IN PHARMACEUTICALS**
J.B. Jerome, American Medical Association
- 9:30 100. THE ROLE OF FATS AND OILS IN COSMETICS**
M. Berdick, Chesebrough-Pond's Inc.
- 10:00 101. PHARMACOLOGY OF FATS AND OILS**
S. Carson, Food and Drug Research Laboratories, Inc.
- 10:30 102. ANALYSIS OF OIL BASED PHARMACEUTICALS**
E. Smith, FDA, Division Drug Chemistry
- 11:00 103. EMOLLIENT OILS**
H.D. Hamilton, Drew, A Division of Pacific Vegetable Corporation
- WEDNESDAY MORNING—OCTOBER 6**
8:30 A.M.—Garden Room
- SESSION O—SYMPOSIUM: ADDITIVES IN FATS AND FAT BEARING FOODS**
- Chairman—David Firestone
- 8:30 104. REVIEW OF GAS CHROMATOGRAPHIC APPLICATIONS IN THE FAT-SOLUBLE VITAMIN FIELD**
A.J. Sheppard, A.R. Prosser and W.D. Hubbard, Food and Drug Administration

- 9:00 105. APPLICATION OF THE FOOD ADDITIVE AMENDMENT TO FAT AND OIL BEARING FOODS**
A.T. Spilner, Jr., Food and Drug Administration
- 9:30 106. GAS CHROMATOGRAPHIC DETERMINATION OF ANTIOXIDANTS IN FATS AND OILS**
Miss E. E. Oja, Food and Drug Directorate
- 10:00 107. FOOD ADDITIVE REGULATIONS: INDUSTRY VIEWPOINT ON CURRENT PROBLEMS**
K.E. Mulford, Atlas Chemical Industries, Inc.
- 10:30 108. METHODOLOGY FOR EMULSIFIERS**
F.J. Baur, The Procter & Gamble Company
- 11:00 109. NUTRITIONAL QUALITY IN FORMULATED FOODS**
O.C. Johnson, Food and Drug Administration
- 11:30 110. METHOD FOR DETECTING CHEMICALLY INDUCED MUTATIONS IN FOOD ADDITIVES**
M.S. Legator, Food and Drug Administration

WEDNESDAY MORNING—OCTOBER 6

- 9:00 A.M.—Solarium**
- SESSION P—SYMPOSIUM: LIPID METHODOLOGY: II. APPLICATIONS OF COMBINED GAS CHROMATOGRAPHY-MASS SPECTROMETRY**
- Chairman—J. Throck Watson
- 9:00 111. A COMPARISON OF ELECTRON IMPACT AND CHEMICAL IONIZATION MASS SPECTROMETRY IN COMPOUNDS OF BIOLOGICAL INTEREST**
H.M. Fales, and G.W.A. Milne, National Heart and Lung Institute
- 9:30 112. APPLICATIONS OF A GAS CHROMATOGRAPH-MASS SPECTROMETER-COMPUTER ANALYTICAL SYSTEM IN STEROID STUDIES**
E.C. Horning and M.G. Horning, Baylor College of Medicine
- 10:00 113. GAS PHASE ANALYSIS OF PHOSPHOLIPIDS**
Catherine Fenselau and J.H. Duncan, Johns Hopkins University School of Medicine
- 10:30 114. A DIGITAL COMPUTER SYSTEM FOR ON-LINE DATA REDUCTION WITH A COMBINED GAS CHROMATOGRAPH-MASS SPECTROMETER**
C.C. Sweeley, B.D. Ray, W.I. Wood, J.F. Holland and M.I. Krichevsky, Michigan State University
- 11:00 115. PROSTAGLANDIN ANALYSIS: SURVEY OF DERIVATIVES USED FOR IDENTIFICATION AND**

**DETECTION BY GAS CHROMATOGRAPH-
MASS SPECTROMETER-COMPUTER SYSTEM**
J. Throck Watson and B.J. Sweetman, Vanderbilt
University School of Medicine

WEDNESDAY MORNING—OCTOBER 6

9:00 A.M.—Pennsylvania 1 Room
**SESSION Q—SYMPOSIUM: PROCESSING OF
EDIBLE AND INEDIBLE FATS AND OILS**

Chairmen—Karl Klein and E.I. Marshack

9:00 116. **CONTINUOUS MISCELLA SOAPSTOCK ACID-
ULATION PROCESS**
L.S. Crauer, The DeLaval Separator Company

9:30 117. **RECENT DEVELOPMENTS IN THE CHILLING
AND CRYSTALLIZATION OF FATS AND OILS**
J. Walsh, Contherm Corporation

10:00 118. **COMPUTER MONITORING OF A MINI-
REFINERY**
E.D. Bitner, J.O. Ernst and H.J. Dutton, Northern
Regional Research Laboratory

10:30 119. **NEW DEVELOPMENTS IN CONTINUOUS EX-
TRACTOR DESIGN**
D. Wolfgang Kehse, Gesellschaft Fur Olmuhlen-
inrichtungen M.B.H.

11:00 120. **CONTACTING AND DISPERSION EMPLOYING
KENICS MIXERS**
A.R. Macdonald, Kenics Corporation

11:30 121. **NEW DEVELOPMENTS ON A CONTINUOUS
HYDROGENATION PROCESS**
R.A. Coombes and R.A. Zavada, Blaw-Knox

WEDNESDAY MORNING—OCTOBER 6

9:00 A.M.—Pennsylvania 3 Room
**SESSION R—SYMPOSIUM: THE WORLD SUPPLY
OF EDIBLE OILS AND PROTEINS (I)**

9:00 122. **WORLD PRODUCTION TRENDS—EDIBLE FATS
AND OILS**
S. Mehr, USDA Foreign Agricultural Service

9:30 123. **INFLUENCE OF WORLD OIL CONSUMPTION
ON EDIBLE PROTEIN SUPPLY**
F.R. Sentil, USDA Agricultural Research Service

10:00 124. **PROJECTION AND PROSPECTS FOR COTTON-
SEED**
C.M. Cater, Texas A&M University

10:30 125. **PROJECTION AND PROSPECTS FOR PEANUTS**

C.T. Wilson, Virginia Polytechnic Institute and
State University

11:00 126. **PROJECTION AND PROSPECTS FOR SUN-
FLOWER SEED**
D.E. Gandy, Agronomist, National Cottonseed
Products Association

11:30 127. **PROJECTION AND PROSPECTS FOR COCO-
NUTS**
W.C. Labys, University of Rhode Island

WEDNESDAY AFTERNOON—OCTOBER 6

2:00 P.M.—Garden Room
SESSION S—SURFACTANTS

Chairman—Abner Eisner

2:20 128. **MIXTURES OF SOAP AND LIME SOAP
DISPERSING AGENTS AS REPLACEMENT FOR
PHOSPHATE BUILT DETERGENTS**
R.G. Bistline, W.R. Noble and J.K. Weil, Eastern
Marketing and Nutrition Research Division

2:20 129. **CHEMICAL AND TOXICOLOGICAL STUDIES
ON N-ALKYL HYDROXAMIC ACIDS**
R.F. Addison and G.L. Fletcher, Fisheries Re-
search Board of Canada, Marine Ecology
Laboratory, Bedford Institute

2:40 130. **PROBLEMS IN THE DETERGENT INDUSTRY
AND THEIR TECHNICAL SOLUTIONS**
A. Davidsohn, Ballestra S.P.A. Milano/Italy

3:00 131. **REVERSE PHASE THIN LAYER CHROMA-
TOGRAPHIC SEPARATION OF OXYETHYLATED
ALKYL SULFATE SURFACTANTS**
A.C. Breyer and Miss Marsha Millie, Beaver
College

3:20 132. **DEPENDENCE OF ULTRACENTRIFUGAL STA-
BILITY OF OIL-IN-WATER EMULSIONS ON
EMULSIFIER CONCENTRATION**
R.D. Vold and K.L. Mittal, University of Southern
California

3:40 133. **SOUND AND SAFE DETERGENT BUILDERS**
H.C. Silberman and E. Jungermann, Armour-
Dial, Inc.

4:00 134. **PERFORMANCE OF NONPHOSPHATE AND
PHOSPHATE HEAVY DUTY LAUNDRY DETER-
GENTS**
R.C. Steinhauer, A.J. Wysocki and E. Junger-
mann, Armour-Dial, Inc.

4:20 **OPEN FORUM: PROBLEMS IN THE SURFAC-
TANT INDUSTRY**

WEDNESDAY AFTERNOON—OCTOBER 6

2:00 P.M.—Pennsylvania 3 Room

**SESSION T—SYMPOSIUM: THE WORLD SUPPLY
OF EDIBLE OILS AND PROTEINS (II)**

2:00 135. **PROJECTION AND PROSPECTS FOR RAPESEED
AND MUSTARD SEED**

R. Ohlson, Ab Karlishamns Oljefabrik

2:30 136. **PROJECTION AND PROSPECTS FOR HAR-
VESTED FISH**

L.M. Sprague, University of Rhode Island

3:00 137. **PROJECTION AND PROSPECTS FOR ANIMAL
FATS AND MEATS**

G. Borgstrom, Michigan State University

3:30 138. **PROJECTION AND PROSPECTS FOR SOY-
BEANS**

E.R. Leng, University of Illinois

WEDNESDAY AFTERNOON—OCTOBER 6

2:00 P.M.—Solarium

**SESSION U—SYMPOSIUM: LIPID METHOD-
OLOGY: III. THIN LAYER CHROMA-
TOGRAPHIC TECHNIQUES**

Chairman—J.C. Touchstone

2:00 139. **QUANTITATIVE THIN LAYER CHROMATOGRA-
PHY OF GANGLIOSIDES**

S.R. Max, University of Maryland School of
Medicine, R.A. Snyder and R.O. Brady, NINDS,
National Institutes of Health

2:30 140. **DETERMINATION OF POLYMER MOLECULAR
WEIGHT DISTRIBUTION BY THIN LAYER
CHROMATOGRAPHY AND DIRECT SCAN-
NING DENSITOMETRY**

E.P. Otocka, Bell Laboratories

3:00 141. **THIN LAYER CHROMATOGRAPHIC METHODS
IN ANALYTICAL TOXICOLOGY**

Paul Schweda, Office of the Chief Medical
Examiner

3:30 142. **QUANTITATIVE ASPECTS OF THIN LAYER
CHROMATOGRAPHY IN AIR POLLUTION
MEASUREMENTS**

T.W. Stanley, Environmental Protection Agency

4:00 143. **RAPID DETERMINATION OF THE SPECIFIC
ACTIVITY OF ISOTOPIC MATERIAL USING
THIN LAYER DENSITOMETRY**

J.C. Touchstone, W. Wortmann, M. Kasparow
and T. Murawec, University of Pennsylvania

WEDNESDAY AFTERNOON—OCTOBER 6
2:00 P.M.—Pennsylvania I Room
SESSION V—GENERAL: LIPID METABOLISM

Chairman—G.A. Jacobson

- 2:00 144. **LIPID PEROXIDATION IN ERYTHROCYTES FROM VITAMIN E DEFICIENT RATS**
Myra O. Barker and Myron Brin, Hoffmann-LaRoche Inc.
- 2:20 145. **RADICAL ADDITION OF LINOLEIC ACID HYDROPEROXIDE OR ITS METHYL ESTER TO α -TOCOPHEROL OR ITS MODEL COMPOUND, 2,2,5,7,8-PENTAMETHYL-6-HYDROXYCHROMAN**
H.W. Gardner, G.W. Grams and K. Eskins, Northern Regional Research Laboratory
- 2:40 146. **LACK OF UNIVERSALITY OF THE RESISTANCE TO AUTOXIDATION OF CHOLESTERYL ESTERS IN AQUEOUS COLLOIDAL SUSPENSION**
L.N. Norcia, Temple University School of Medicine, and V. Mahadevan, V.A. Hospital
- 3:00 147. **LIPID CHANGES IN BEAGLE DOG LUNGS AFTER INHALATION OF ^{134}Ce FUSED MONTMORILLONITE CLAY AEROSOLS**
R.C. Pflieger, H.C. Redman and S.A. Benjamin, Lovelace Foundation
- 3:20 148. **ACTIVATION OF TRANS-UNSATURATED LONG CHAIN FATTY ACIDS BY RAT LIVER SUBCELLULAR FRACTIONS**
Kenneth Lippel, U.S. Department of Agriculture
- 3:40 149. **EFFECTS OF ORAL CONTRACEPTIVES ON**

GLYCERIDES AND CHOLESTERYL ESTERS OF HUMAN CERVICAL MUCUS
E.J. Singh and J.R. Swartwout, The University of Chicago

- 4:00 150. **METABOLISM OF α -MONOPALMITIN BY RAT LUNG IN VITRO**
M.C. Wang and H.C. Meng, Vanderbilt University Medical School
- 4:20 151. **SHORT METHOD FOR THE DETECTION OF CHICK EDEMA FACTOR IN FATS, OILS AND FATTY ACIDS BY ELECTRON CAPTURE GAS CHROMATOGRAPHY**
N. Kins and J. Berandy, Drew, Division of Pacific Vegetable Oil Corporation
- 4:40 152. **PLASMA MEMBRANE FRACTIONS FROM NORMAL AND ESSENTIAL FATTY ACID DEFICIENT EHRLICH ASCITES TUMOR CELLS**
H.L. Creinin and K.A. Narayan, University of Illinois

TUESDAY MORNING—OCTOBER 5

9:00 A.M.—Pennsylvania Room

SESSION W—SYMPOSIUM: ENVIRONMENTAL SCIENCE AND INDUSTRIAL PROCESSING

Chairman—G.M. Kreutzer

- 9:00 153. **CENTRIFUGAL APPLICATION RELATING TO FATS AND OILS WASTE STREAM**
A.E. Zambone, Sharples Corporation

- 9:30 154. **POLLUTION CONTROL FACILITIES AT A GLIDDEN-DURKEE REFINERY**
T.A. Walsh, Glidden-Durkee Division, SCM Corporation

- 10:00 155. **METHOD OF CONTROLLING SULFURIC ACID FUMES FROM AN INDUSTRIAL OPERATION**
F.E. Sullivan, Frank E. Sullivan Company

- 10:30 156. **YOUR AOCS COMMITTEE ON POLLUTION PROBLEMS**
Clifford Haysley, Glidden-Durkee Division, SCM Corporation

- 11:00 157. **REVIEW OF HEXANE-EXTRACTABLE MATERIALS AS RELATING TO PRESENT EFFLUENT STANDARDS**
G.M. Kreutzer, Swift and Company

- 11:30 158. **USE OF IMPROVED CURRENT IN WASTE TREATMENT**
D.R. Erickson, Swift and Company

TUESDAY AFTERNOON—OCTOBER 5

1:30 P.M.—Pennsylvania Room

SESSION W—SYMPOSIUM: ENVIRONMENTAL SCIENCE AND INDUSTRIAL PROCESSING

Chairman—G.M. Kreutzer

- 1:30 159. **AIR POLLUTION REGULATIONS AND IMPLEMENTATION PLANS: PAST, PRESENT AND FUTURE**
Francis Scofield and D.S. Ring, Ring & Scofield Consultants

ABSTRACTS OF PAPERS

significant morphometric increases ($p < 0.05$) in numbers and cross-sectional areas of mitochondria and a decrease in surface to volume ratio. The increased ability of trained muscle to oxidize substrates is accompanied by a twofold increase in intracellular resting glucose and significant increases in glycogen synthetase (glucose-phosphate dependent) and hexokinase ($p < 0.025$). Adequate amounts of other glycolytic enzymes were present but were not increased in the exercised muscle. Intracellular triglyceride, carnitine and acyl carnitine transferase were present in large amounts and did not respond to training. In vitro studies of fatty acid into intracellular tri-glyceride in response to exercise training. Fatty acid entry were stimulated three- to fivefold by addition of glucose. It therefore appears certain that exercise training increases mitochondrial oxidative capacity.

probably in the muscles, and the cold-acclimated animal differs from the cold-exposed animal in being able to switch on non-shivering thermogenesis via activation of the sympathetic nervous system. The maintenance of this adaptation for non-shivering thermogenesis in tissues other than the brown adipose tissue itself depends upon the brown adipose tissue. The adaptation disappears if the brown adipose tissue is removed; the adaptation does not develop if the normal proliferation of mitochondria in the growing brown adipose tissue is inhibited (with oxytetracycline) during acclimation of rats to cold. The mechanism by which the brown adipose tissue exerts this second function is at present unknown.

3
EFFECT OF REGULAR MUSCULAR ACTIVITY ON LIPID METABOLISM IN RATS. VLADO SIMKO, Sage Hospital, Ithaca, N.Y. 14850.

Technological progress affects the ratio between the energy storing fat mass and the energy consuming muscle mass in the body. Our experiments on rats attempted to clarify the effect of regular muscular activity on lipid metabolism. No changes in blood cholesterol were found in rats exposed to 1 hr of swimming and during the 5 hr following this exercise. However, exercise consistently lowered the total liver cholesterol in rats and we could reproduce this phenomenon in several studies even when the type and duration of exercise and the type of diet widely differed. This effect of exercise seems not to be mediated through alteration in synthesis of cholesterol. Exercising rats incorporated $1\text{-}^{14}\text{C}$ -acetate into liver cholesterol at a higher rate, which may be considered a compensation for the lowering of liver cholesterol by exercise. In subsequent experiments we studied the effect of exercise on the total bile production collected for 24 hr. In both the short-term exercise study (rotating drum) and prolonged swimming (105 days) study, the 24 hr bile excretion produced a significant drop in liver cholesterol only in exercising rats. Exercising rats excreted less cholesterol and more deoxycholic acid in bile than control animals. This may be of considerable importance to the fate of cholesterol, since the reconversion of choleliths to cholesterol is impossible. Untrained rats responded to physical exercise with a higher and prolonged rise of plasmatic free fatty acids. Exercising animals displayed a higher proportion of saturated fatty acids in depot fat triglycerides. Liver tri-glycerides on the other hand, had a lower proportion of saturated fatty acids and a higher proportion of monoenes and polyenes. Exercise may preferentially release polyenes from the depot fat, which may in turn promote the transport and catabolism of cholesterol.

4
HUMAN SKELETAL MUSCLE ADAPTATIONS TO EXERCISE STRESS. T.E. MORGAN, L.A. COBE, RUSSELL ROSS and F.A. SROET, University of Washington, Seattle, Wash. 98105.

During the stress of fasting, careful studies have shown that muscle alters its utilization of substrates to spare glucose and to depend largely upon oxidation of fatty acids and ketones. Patterns of substrate utilization by muscle in other stress states are not as clearly understood. Studies of the stress of muscular exercise indicate that muscle free fatty acid utilization increases during acute exercise. In these studies as work load increases mitochondrial oxidative capacity or ATP generation appears to limit oxidation of substrates. We have studied the muscle adaptations induced by long term exercise in 10 male human volunteers who exercised one leg by 2 hr of submaximal bicycle pedalling daily for one month. This training increased capacity to exercise on the bicycle ergometer. At the end of the month, both quadriceps femoris muscles were biopsied under local anesthesia. Mitochondria isolated from both exercised and contralateral nonexercised muscles were shown to have normal respiratory control and tightly coupled phosphorylative mechanisms. Mitochondrial oxidative capacity increased 52% ($p < 0.05$) in response to exercise training and was paralleled by highly significant increases in mitochondrial protein, oxidative enzymes and muscle phospholipid. Mitochondrial numbers and size were estimated by electron microscopy; there was

1
FREE FATTY ACID METABOLISM DURING STRESS: EXERCISE, ACUTE COLD EXPOSURE AND ANAPHYLACTIC SHOCK. PAUL PAUL and W.L. HOLMES, Lankenau Hospital, Philadelphia, Pa. 19151.

The rates of turnover and oxidation of plasma free fatty acid (FFA) were determined in unanesthetized dogs during exercise, acute cold exposure and anaphylactic shock with the aid of technique involving the continuous infusion of albumin bound $1\text{-}^{14}\text{C}$ -palmitate and the simultaneous measurement of O_2 uptake, CO_2 output, and the specific activity of CO_2 and FFA in normal untrained dogs. Plasma FFA supplied 20-30% of the energy expenditure during short leg 70-90% of the energy derived from the same sources. Acute cold exposure (4-5°C for 120 min) increased the participation of FFA in energy metabolism. During the control phase, plasma FFA level was $0.61 \mu\text{Eq/ml}$ with a turnover rate of $17.6 \mu\text{Eq/kg}$ and contributed which 22% was immediately oxidized and contributed 24% to the exhaled CO_2 . The same dogs exposed to acute cold increased their FFA level and turnover rate to $35.2 \mu\text{Eq/ml}$ and $28.0 \mu\text{Eq/kg}$ min, respectively, of which 35% was immediately oxidized contributing 46% to the exhaled CO_2 . A concomitant decrease in RQ was observed. During anaphylactic shock, plasma lactic acid concentration was high while FFA level and turnover rate were reduced greatly and the fraction of FFA turnover immediately oxidized was depressed markedly, i.e., 3-9% of turnover. When sodium lactate was infused to induce a blood level comparable to that seen in anaphylaxis, a marked decrease in the level and turnover rate of FFA was found. However, the fraction of turnover oxidized remained the same as during the preinfusion period (range of 21-40%). Results similar to those seen with lactate were obtained with respect to the per cent of FFA turnover oxidized when nicotinic acid, an agent which also reduces FFA level and turnover, was infused. The administration of exercise or norepinephrine during anaphylactic shock also interfered with FFA oxidation whereas exogenous lactate had no effect on this oxidation. The effect of a deficiency of pancreatic and thyroid hormones on FFA metabolism were investigated in dogs in which these endocrine glands were extirpated. These results will be discussed in relation to those observed during stress.

2
LIPID METABOLISM DURING COLD EXPOSURE AND DURING COLD ACCLIMATION. JEAN HIMMS-HAGEN, University of Ottawa, Ottawa, Ontario, Canada K1N 6N5.

Two lipid-containing tissues are important in cold exposure (exposure to cold of animals not previously living in the cold) and in cold acclimation (the adaptive state achieved when animals have lived in the cold for several weeks): these are the white adipose tissue and the brown adipose tissue. The white adipose tissue serves as a store of readily mobilized substrate (free fatty acids, FFA) for calorogenesis in other tissues during cold exposure, principally for shivering thermogenesis in muscle. The mobilization of the stored lipid is brought about through activation of the sympathetic nervous system by the cold stress. The brown adipose tissue has two functions in cold exposure and in cold adaptation, both quite distinct from the function of the white adipose tissue. These functions are heat production and the maintenance of the adipose tissue as cold. The triglycerides stored in the brown adipose tissue are mobilized as FFA, also via activation of the sympathetic nervous system, but the FFA are used primarily within the brown adipose tissue itself. The FFA are the agents which switch on the calorogenesis in the brown adipose tissue (via a primary understood form of "loosening" of the coupling of oxidative phosphorylation); they also serve as the substrate for the calorogenesis. The heat-producing function of the brown adipose tissue occurs in both cold-exposed and in cold-acclimated animals; it is of greater importance in the latter because this tissue normally grows in response to cold. Much of the heat production in cold-acclimated animals (nonshivering thermogenesis) occurs outside the brown adipose tissue itself, most

5
CAUSE OF EXHAUSTION IN PROLONGED EXERCISE AND THE EFFECT OF METHYLPREDNISOLONE. B. ISSEKUTZ, JR., Dalhousie University, Halifax, N.S., Canada.

Earlier studies on men and dogs showed that prolonged exercise caused a continuous rise of plasma FFA because of progressively increasing release of FFA from the adipose tissues. In dogs, in the third hour of exercise (treadmill run on 15% slope, 100 m/min) the FFA turnover is high enough to cover 80-90% of the energy expenditure. Studies with ^{14}C -glucose revealed that the hepatic glucose output cannot cover more than 10-20% and the rate of breakdown of muscle glycogen could provide fuel for an additional 20-25% of the energy expenditure. At time of exhaustion the rate of mobilization of energy sources exceeded the energy requirement by about 30%. On a molar basis, the rate of mobilization of carbohydrate to that of FFA is about 2:1. Exhaustion is usually caused by the decrease of plasma glucose, and a total of about 4 g glucose is produced by 100 g liver. Exercising dogs treated with methylprednisolone (MP) for two to three days prior to exercise had a glucose turnover rate about twice that of the controls, and ran twice as long. The total glucose production of these livers was 16/100 g. Doubly labeled experiments with $2\text{-}^3\text{H}$ -glucose and $14\text{-}^1\text{C}$ -lactate or ^{14}C -uric acid revealed that only 10% of this plasma glucose arose from lactate and not more than a maximum of 15% could have been derived from protein during the exercise. Both this onset of hypoglycemia and the rise of FFA were considerably delayed. The high rate of glucose turnover in MP treated exercising dogs is probably due to the fact that MP treatment greatly potentiates the effect of epinephrine on hepatic glycogenolysis. The initial glycogen content of the liver is one of the most important factors in limiting the duration of exercise and the decrease of plasma glucose plays an important role in facilitating the release of FFA.

6
INTRAMUSCULAR ENERGY SOURCES IN DOGS DURING HEAVY PHYSICAL WORK. D.G. THERIAULT, G.A. BELLER, J.A. SMOKE and H.L. HARTLEY, U.S. Army Research Institute of Environmental Medicine, Natick, Mass. 01760.

The nature of the fuel utilized by the contracting musculature during work has been a subject of debate for many years. In the past, many believed carbohydrate to be the sole fuel for muscular activity. It subsequently has been shown that both carbohydrate and lipid serve as fuel for contracting muscles. The source of these energy substrates must be either fat or glycogen in the muscles themselves, or fatty acids and glucose mobilized from other tissues, and carried to the muscle via the circulation. Although skeletal muscle is known to contain a wide variety of lipids, little is known concerning their physiological actions in the function of this tissue. Current thinking reflects a belief that the intracellular skeletal muscle lipids are utilized as a reserve energy source during exercise. The evidence, however is only suggestive. Methods for the quantitative isolation and detailed analysis of skeletal muscle lipids from small biopsy specimens have been applied to the

showed similar, but more pronounced behavior. Certain synergists, such as ascorbic acid, citric acid, or EDTA, that were effective at 110 C or 25 C failed to give comparable effects at 5 C or -20 C. In searching for an explanation of these observations, the concentrations of primary antioxidant and synergist in fat were varied simultaneously. With the AOM, maximum antioxidant activity was found to occur with specific ratios of synergist-antioxidant. The ratio associated with maximum antioxidant activity was found to be dependent on the nature of the antioxidant. The implications of these observations in the stabilization of oil-bearing food products and in the prediction of the composition of antioxidant mixtures are discussed.

10

STUDIES ON THE BIOSYNTHESIS OF CHLOROSULFO-LIPIDS. T.H. HAINES and CAROLYN MOONEY, City College of New York, N.Y. 10031.

Two series of chlorosulfolipids have been isolated from the phytoergolate, *Ochromonas danica*. One is a series of C₁₄-alkylidial disulfates with from zero to six chlorines replacing hydrogens on the chain. This series contains chloro groups in varying numbers and in various positions, corresponding to those of the hexachloro compound: 2,2,11,13,15,16-hexachloro-1,14-dicosanediol disulfate. The second is a corresponding series of C₁₆ carbon chain 1,15-diol disulfates. In this latter series the respective compounds are derivative to: 2,2,12,14,16,17-hexachloro-1,15-tetrasoanediol disulfate. We have established that ¹⁴C-acetate serves as a precursor for the biosynthesis of these compounds. Studies on the incorporation of acetate, formalin, stearate and oleate have yielded information about the biosynthetic patterns. Acetate and laurate are incorporated into stearate and oleate are apparently not incorporated. The implications of these data, and other data on related precursors, for the biosynthetic pathways, will be discussed.

11

SOLUBILIZATION OF FLAVOR MOLECULES IN LIQUID AND LIQUID CRYSTALLINE PHASES OF WATER, MONO- AND TRIGLYCERIDES. S. FRIBERG and L. WILTON, The Swedish Institute for Surface Chemistry, Stockholm, Sweden.

The solution and solubilization of flavors in liquid and liquid crystalline phases in systems of water, liquid fats and food emulsifiers have been investigated by means of phase equilibrium determinations. The results have been compared to those from investigations of the site of the flavor molecule in fat emulsions. The utility of the liquid crystalline phase as a model to give information about the conditions at the interface is discussed.

12

LIPID AND PROTEIN COMPETITIVE REACTIONS IN DRY SYSTEMS. G.V. RAO and L.R. DUGAN, JR., Michigan State University, East Lansing, Mich. 48823.

Model freeze-dried food systems prepared from defatted beef muscle fiber, phosphatidylethanolamine (PE), hydrogenated phosphatidylcholine, and nonanal in various combinations were stored at RH 14% and ambient temperature for 30 days. Chloroform-methanol soluble components were extracted and separated into elements to define the lipid-browning products. The extracted fibers were analyzed for amino acid content. Typical lipid-browning products were found in systems containing PE, PE plus nonanal and hydrogenated PE plus nonanal. Amino acid changes in the muscle fiber were noted in the systems containing PE or when nonanal was a component. Greatest changes were in lysine, methionine, phenylalanine and tyrosine. Decrease in methionine content was believed to be due to oxidation reactions. Decrease in other amino acids and lysine in particular was probably due to aminocarbonyl reactions from added aldehyde groups. Side products generated by oxidation of the PE during storage. Other than in the case of methionine, there was less loss of amino acids in fibers when the system contained PE. The competition of amino groups from the PE had a sparing effect on carbonyl-amino reactions involving the proteins. Lipid-browning reactions may thus have protein-sparing activity in stored dry food systems.

13
STEROL ADDITIVES AS POLYMERIZATION INHIBITORS FOR FRYING FATS. R.J. SIMS, J.A. FIORITI and M.J. KANUC, General Foods Corp., Tarrytown, N.Y.

The unsaponifiable fraction from *Vernonia*, antihelmintic seed oil functions as a polymerization inhibitor for polyunsaturated frying oils. Two sterols, vernosterol and a avenasterol were shown to be the active components responsible for this effect, although squalene also makes a small contribution. The degree of protection observed was similar to that obtained with methyl silicone. Since the effect is obtained only when the oil is quiescent, the mechanism may involve formation of a protective film at the oil-air interface. Additional examples of active unsaponifiable oil fractions include olive oil and wheat germ oil. Contrary to expectations, β -sitosterol, the major sterol of olive oil, had no effect. Neither stigmasterol or lanosterol seemed to influence oxidation rate, and ergosterol appeared slightly prooxidant. Addition of these unsaponifiable fractions (0.10-0.5% level) or 0.1% of avenasterol or vernosterol to safflower seed oil minimizes loss of polyunsaturates, darkening and increase in viscosity during heating at frying temperature. These effects are similar to those reported earlier by Chang with the α -sitosterols.

14

THE FATE OF TRIGLYCERIDE HYDROPEROXIDES DURING CATALYTIC HYDROGENATION. N.E. BENDASOYK and S.S. CHANG, Nabisco Research Center, Fair Lawn, N.J. 07410.

Most edible oils, when they are hydrogenated, develop a characteristic off odor. The volatile compounds responsible for this odor have been characterized and their formation has been attributed to decomposition of preformed hydroperoxides. The present study was concerned with determining the chemical nature of the nonvolatile compounds resulting from hydroperoxide decomposition under conditions of catalytic hydrogenation. Purified triolein was autoxidized to a peroxide value of 820 mEq/kg. The hydroperoxides were concentrated via silicic acid chromatography, and this highly oxidized fraction was hydrogenated at 175 C, 10 psi of hydrogen, and 0.25% nickel as catalyst. Preliminary fractionation of the methyl esters of the hydrogenated oxidized fraction was effected by urea adduct formation. The adductable compounds amounted to 50% of the esters and consisted of the expected products of linoleate hydrogenation. Further fractionation of the urea filtrate was carried out on silicic acid columns. Chemical characteristics of the 17 fractions obtained were determined by following analyses: molecular weight, elemental composition, IR and UV absorption, number of double bonds, hydroxyl content, carbonyl content, ester content and presence of ring structures. As indicated by the character of the products formed, decomposition via free radical intermediates, which yields oxygen-linked dimers, is the dominant mechanism of hydroperoxide breakdown under hydrogenation conditions. Reduction of hydroperoxides to alcohols is also an important reaction, while thermal decomposition, which yields carbon to carbon-linked dimers, is of minor importance.

15

FUNGI PATHOGENIC TO INSECTS. THE NEUTRAL AND POLAR LIPIDS OF ENTOMOPHTHORA CORONATA. T.E. BRUSZWSKI and R.O. MUMMA, Pennsylvania State University, University Park, Pa. 16802.

The polar and neutral lipids of *Entomophthora coronata* have been qualitatively and quantitatively examined with a 28-day growth period. The lipids were characterized with the use of thin layer column and gas chromatography, selective sprays, s.p., and ¹⁴C-labeling and mass spectrometry. The neutral lipids consist of monoglycerides, diglycerides, cholesterol, free fatty acids, triglycerides and cholesterol esters. The polar lipids are very unusual for fungi. Only 1% of the total lipids of the polar lipids are phospholipids (PE, LPE, PC, LFC and Sp) and their relative concentrations decrease with age. The organism contains a significant concentration of glycolipids which include cerebrosides. To our knowledge, this is the first report of the occurrence of cerebrosides in fungi. Many of the polar lipids, present in trace quantities, give a positive ninhydrin reaction.

study of the fate of muscle lipids during heavy physical work. Muscle triglyceride is indeed a source of energy for muscle contraction. Furthermore, measurements of free fatty acid A-V differences during moderate and heavy exercise support the belief that the intensity of work in relation to the individual aerobic power affects the percentage contribution of fat in the fuel of free fatty acids, a carbohydrate being a major source of energy. The closer the subject works to his maximum, the more important is carbohydrate.

7

GAS LIQUID CHROMATOGRAPHIC REACTIONATION OF SEED OIL DIGLYCERIDES ON POLYESTER LIQUID PHASES. A. KURSKI, O. STACHNYK and L. MAMAI, University of Toronto, Toronto, Ontario, Canada.

Mixtures of diglycerides derived from maize, flax and other seed oils by hydrolysis with pancreatic lipase have been effectively resolved according to their molecular weight and degree of unsaturation by gas chromatography of their trimethylsilyl ethers or acetates on 180 x 0.3 cm i.d. glass columns containing 3-6% polyester packing. Using organosilicone polyesters (EGSS-X, EONSS-M) diglycerides with 0-6 double bonds were recovered as separate peaks within 30 min at 250 C when run as the silyl ethers. The conventional polyesters (DEGS, EGS, BDS, NGS and CHDMS) required 20-30 C higher temperatures for a similar elution. Because of higher thermal stability, the succinates of neopentylglycol and cyclonexanediol gave the longest lasting packings and the best chromatograms. Glass columns appeared to minimize the decomposition of the liquid phase, while helium as carrier gas gave higher column efficiencies than nitrogen. The 1,2 (3,3)-diglycerides were eluted ahead of the corresponding 1,3-diglycerides on all columns. Under optimum conditions these separations could be performed with a sensitivity and accuracy approaching that commonly obtained in the analysis of fatty acid methyl esters. Obvious applications in the analysis of fatty acids are the analyses of molecular species of glycerophosphatides and triglycerides.

8

A RAPID METHOD FOR DETERMINING ANTIOXIDANT ACTIVITY AND FAT STABILITY. D.L. BREWER, J.A. COMBS and G.A. JACOBSON, Campbell Institute for Food Research, Camden, N.J. 08101.

The acceleration of lipid oxidation by hemin was used to determine antioxidant activity and either fat or oil stability. The test system consisted of a fat or oil emulsion dispersed in phosphate buffer (pH 7.2). The temperature of the system was maintained at 45 ± 1 C. The antioxidant was added to the fat before emulsification. The rapid rate of oxygen uptake which occurred on the addition of hemin to the emulsion was measured with a Beckman Oxygen Analyzer and was recorded automatically. All fats and oils tested exhibited a typical induction period, which could be increased by the addition of an antioxidant. This increase in the induction period was related to antioxidant activity. The age and purity of the hemin solution, the concentration of hemin in the emulsion, the age and pH of the emulsion, the peroxide value of the fat, and the temperature affect the induction period or the rate of oxygen uptake, or both. For a given set of conditions, the induction period was reproducible, although the rate of oxygen uptake was not. The activities of several antioxidants (BHA, propyl gallate, TBHQ and "Topanol") and synergists (ascorbic acid, citric acid and EDTA), determined by the hemin-catalyzed oxygen uptake method, are compared with those obtained by the AOM.

9

EFFECTS OF SYNERGIST AND TEMPERATURE ON ANTI-OXIDANT FUNCTION. D.L. BREWER and G.A. JACOBSON, Campbell Institute for Food Research, Camden, N.J. 08101.

A number of commercial, synthetic, primary antioxidants were found to exhibit varying responses when tested in fat at temperatures ranging from 110 C (AOM) to -20 C. The responses were antioxidant at 110 C (AOM) and 25 C, but sometimes slightly prooxidative or only slightly antioxidant at 5 C or -20 C. Natural antioxidant extracts from spices

properties to present margarine, but with a fat level of about 40%; need for new food standards of identity to cover other fatty foods, etc.

26

KINETICS OF COPPER CHROMITE HYDROGENATION IN SOYBEAN AND LINED OILS: EFFECT OF PRESSURE, P.V. VIGNERON, S. KOETALA, R.O. BUTTERFIELD and H.J. DUTTON, Northern Regional Research Laboratory, Peoria, Ill. 61604.

Although the prime impetus for study of copper catalysts in the selective reduction of linoleic acid in soybean oil is recent economic developments raise the possibility that hydrogenated linseed oil could also be used as an edible oil. Two levels of hydrogen pressure were used to hydrogenate soybean and linseed oils; their effects on kinetic pathways, *trans* content and distribution of residual double bonds were investigated. The course of the reactions was studied by removing, at intervals, a sample corresponding to a small iodine value drop; constituents were determined by the analytical methodology previously described. Increasing hydrogen pressure increased the rate of reduction and decreased the concentration of conjugated dienes. The linoleate-to-monoene amount is enhanced by raising the pressure although the pathway that still predominates for linseed oil hydrogenation is linoleate to conjugated dienes to monoenes.

27

HYDROGENATION AND DEUTERIATION OF CONJUGATED ISOMERS OF LINOLEATE AND LINOLENATE WITH PALLADIUM, PLATINUM, NICKEL AND LINDLAR CATALYSTS, SAMBASIVARAO KORTELKA, C.A. SCHOLFIELD, E. SELKA and H.J. DUTTON, Northern Regional Research Laboratory, Peoria, Ill. 61604.

Conjugated isomers of linoleate and linolenate were reduced with palladium, platinum, nickel and Lindlar catalysts at atmospheric hydrogen or deuterium pressure. After the products were separated, positions of their double bonds were determined by ozonolysis. Palladium and platinum catalysts reduced β -oleostearate directly to monoene. Nickel reduced β -oleostearate chiefly by 1,2-addition and to a lesser extent by 1,4- and 1,6-addition, whereas Lindlar catalyst reduced by 1,2- and 1,6-addition only. All catalysts reduced conjugated diene isomers of linoleate by both 1,2- and 1,4-addition, with nickel being somewhat more preferential for 1,2-addition. Selectivity for the catalytic reduction of dienes to monoenes decreased in the order nickel, palladium and platinum. Lindlar catalyst neither isomerized nor reduced the monoenes that formed during hydrogenation. Nor did palladium and platinum isomerize conjugated dienes and trienes during their reduction, but nickel and Lindlar catalysts isomerized them slightly. Some deuterium was found in unreacted conjugated diene and triene where nickel or Lindlar catalysts were used but none with palladium and platinum. Deuterated products contained a wide range of isotopic isomers with some having as many as 81 deuterium atoms. This wide deuterium distribution resulted from: exchange followed by addition; addition followed by exchange; and exchange-addition-exchange reactions.

28

ALKOXYLATION OF N-ALKYL ETHYLENE DIAMINE, W. ROSENBLATT, A. STARCK, C. BUNESIN and Y.K. KIM, Wico Chemical Corporation, Oakland, N.J. 07436.

The alkoxylation of a new *N*-alkyl-substituted diamine, Witcamine ED-16, obtained by reacting *Cusar* chloroparmins and ethylene diamine was studied. Ethoxylation in both laboratory glassware and Parr autoclave equipment readily produced 20 mole adducts without alkaline catalysis. The reaction was initiated by the formation of the three mole adduct at 160°C and subsequent addition of the alkylene oxide at 90°C. Adducts with mole ratios greater than 20 required an alkaline catalyst. Preparation of propoxylate derivatives necessitated addition of a relatively high (2.5-3%) concentration of caustic catalyst. Satisfactory results were obtained at 185-190°C and 40-50 psi. An evaluation of the surfactant properties of the alkoxyates and their correlation with structure and composition was obtained. Data on solubility, surface tension, density, refractive index, pH and foam characteristics are presented. Polyurethane foams were prepared from several of the alkoxyates.

29

EPIMINOSTEARATES FROM UNSATURATED FATS. T.A. FOGLIA and P.A. BARR, Eastern Marketing and Nutrition Research Division, ARS, USDA, Philadelphia, Pa. 19118.

The present work describes our further efforts to develop new methods for the preparation of the epimino derivatives of oleic alcohol and methyl oleate by direct procedures. The procedure utilized for the epimino preparation was the addition of the electrophilic reagent iodine oxide to the unsaturated compound followed by reductive cyclization to the desired epimino compound. Several hydrolytic reducing agents and direct hydrogenation over noble metal catalysts were investigated to determine the optimum conditions for preparing the epiminos. In comparison of the various methods studied demonstrated that the best yields of the epimino derivatives were obtained with lithium aluminum hydride, whereas direct hydrogenation gave the poorest yield. The versatility of the β -iodo side adducts as precursors for organic synthesis was demonstrated by a study of their reaction with trialkyl phosphites. The interaction of the β -iodo side adduct with a trialkyl phosphite underwent a spontaneous Arbuzov type reaction to the di-*n*-alkylphosphonaziridyl derivative of the starting olefin in good yield.

30

PREPARATION AND REACTIONS OF LAZIRENES DERIVED FROM FATTY ACIDS. T.A. FOGLIA, P.A. BARR and G. MAERKER, Eastern Marketing and Nutrition Research Division, ARS, USDA, Philadelphia, Pa. 19118.

l-Azirene derivatives of fatty acids, which contain an unsaturated nitrogen bridge at the chain location formerly occupied by a double bond, are a new class of fatty acid compounds. The *l*-azirene derived from methyl oleate (diagram) was prepared, and some of its reactions were studied.

N



The preparation of this novel fatty acid derivative was accomplished in a three-step synthesis with overall high yields. Addition of the electrophilic I_2N_2 to methyl oleate yielded the β -iodo azide derivative of stearic acid. Treatment of this compound with base resulted in dehydrohalogenation and gave methyl 9(10)-azido-9-octadecenoate. Solution photolysis of the latter vinyl azide yielded the *l*-azirene. Reduction of the *l*-azirene with metal hydrides gave the known epimino derivative of octadecanoic acid. Other reducing agents were unsuccessful in preserving the three-membered ring and gave amines as products. Reaction with aqueous mineral acids caused the dimerization of the *l*-azirenes with formation of tetrasubstituted pyrazines. The *l*-azirenes were attacked by acid chlorides and hydrochloric acid to give chlorinated amides as principal products.

31

DIRECT ESTERIFICATION OF SODIUM *cis*-9,10-EPIMINO-OCTADECANOATE. A. BILYK, A. EISNER and G. MAERKER, Eastern Marketing and Nutrition Research Division, ARS, USDA, Philadelphia, Pa. 19118.

Previous work at this laboratory has demonstrated that some epiminoester derivatives such as soaps have potential utility as lubricant materials. However, until recently a similar evaluation of epiminoester esters was not possible because of the difficulties involved in their preparation by conventional acid catalysis. Compounds containing the aziridine ring have been known to be unstable in the presence of mineral acids since they have a tendency to undergo polymerization. Additionally, with internal aziridine rings, cyclic opening reactions are possible as a result of nucleophilic attack by generated carboxylic acids from the epimino soap. In the present study, it was determined that high yields (86-89%) of methyl *cis*-9,10-epiminooctadecanoate, with a minimum of attendant byproducts could be obtained by heating the corresponding sodium soap with methanol in the presence of certain Lewis acid catalysts under carefully controlled conditions. Temperature, duration of heating period, and catalyst concentration were the variables studied for each catalyst. The catalysts evaluated were: boron trifluoride etherate, methanesulfonic acid, sulfuric acid, perchloric acid, fluoroboric acid and *p*-toluenesulfonic acid. Amino ethers and amino esters are

among the byproducts formed in small amounts during the esterification reaction.

32

ESTERIFICATION AND TRANSESTERIFICATION OF 9(10)-CARBOXYSTEARIC ACID AND ITS METHYL ESTERS. KINETIC STUDIES. E.J. DUFFEC, R.O. BUTTERFIELD and E.N. FRANKEL, Northern Regional Research Laboratory, Peoria, Ill. 61604.

9(10)-Carboxystearic acid and its monomethyl ester prepared by oxidation of hydroformylated oleic acid and methyl oleate were esterified with 1-butanol, 2-methoxyethanol, 2-chloroethanol, 2,2-dimethylpentanol and 2-ethylhexan-1-ol. Rate studies showed that the diacid was esterified when sulfuric acid was the catalyst to the alkyl 9(10)-carboxystearate approximately 20 to 30 times faster than the alkyl 9(10)-carboxystearate esterified to the diacid ester. Methyl 9(10)-carboxystearate was transferred to the alkyl carboxystearate about two times faster than the alkyl carboxystearate is esterified to the diacid ester. Esterification rate is highly dependent on catalyst concentration. Steric hindrance in 2,2-dimethylpentanol and the electrophilic character of 2-methoxyethanol retard the rate of reaction. Esterification with 2-chloroethanol yields, in addition to the expected diesters, products formed from the diesters by further reaction with 2-chloroethanol. The four possible diesters of methanol and dimethylpentanol that were prepared are being evaluated as lubricants and plasticizers.

33

POTENTIAL INDUSTRIAL DERIVATIVES OF METHYL 9(10)-FORMYLSTEARATE ACETALS, ETHERS AND POLYMERS. R.A. AWL, E.N. FRANKEL, E.H. PAYNE and J.C. CORWAN, Northern Regional Research Laboratory, Peoria, Ill. 61604.

Six acetal derivatives from methyl 9(10)-formylstearate and the enol ether derived from the dimethyl acetal were prepared in 90-98% purity and evaluated as potential PVC plasticizers. These compounds were low melting (below -80°C), and were generally compatible as secondary PVC plasticizers. They imparted superior low temperature properties when compared to dioctyl phthalate alone. The acetals and both alkenyl and alkyl ethers were characterized thermally, spectroscopically and chromatographically. A tough, rubber-like poly(amide-acetal) of 12 repeating units and a low molecular weight, viscous poly(ester-acetal) were prepared from the pentaerythritol acetal. Both polymers crosslinked with acid at high temperatures. Unlike the usual high-melting polyamides with glass transitions near 50°C, the poly(amide-acetal) was low melting (38-52°C) and showed a possible glass transition at -6°C (differential thermal analysis).

34

PLASTICIZING ACTION AND CRYSTALLINITY IN POLYMERS INCORPORATING FATTY MONOMERS. A.N. WAGLEY, E.F. JORDAN, JR., Eastern Marketing and Nutrition Research Division, ARS, USDA, Philadelphia, Pa. 19118.

A summary is made of glass or flex temperature-lowering and crystallization effects by some fatty components of polymers. When polyacrylonitrile was plasticized by copolymerization of its monomer with fatty acrylamides lowering of flex temperature was described by a modified Fox equation: $1/T_g = K_m + 1/T_g^0$ in which m was the fraction of plasticizing comonomer. Crystallization obscured the flex temperature of polyacrylonitrile homopolymer, but extrapolation to $m=0$ indicated its hypothetical value. The value of K increased with the length of the side chain of fatty comonomer. The glass or brittleness temperatures of series of homopolymeric *n*-alkyl methacrylates, acrylamides, acrylates, styrenes and alkenes defined with the increase in the length of the number of flexible bonds in the side chain. Plots of various series met at about -111°C, so that, in Fox relation terms, comonomers with long fatty side chains were superior as plasticizers to shorter side chain comonomers. Measurement of the crystallization of polyalkyl acrylates, acrylamides and vinyl esters by differential scanning calorimetry resulted in scanning curves that became narrower as saturated side chains became longer. Phase diagrams of homopolymers of different series showed isomorphism. Glass temperatures of C_{18} homopolymers were often half of melting temperatures, obeying a generalization normally applicable to

symmetrical polymers. These results demonstrated that the crystallization resided in the saturated fatty side chains. Heats of fusion per side chain methylene were therefore calculated. Values were similar to that reported for the alpha hexagonal to liquid transition of alkanes. Homopolymers with oleyl side chains were amorphous. Among random copolymers having as crystalline component octadecyl acrylate and as amorphous component methyl, ethyl, butyl, octyl or oleyl acrylate, ΔH_f varied as the weight fraction of crystalline component. At constant mole fraction, melting point declined with increase in log side chain length of amorphous component.

35

POLYESTERAMIDES FROM LINSEED AND SOYBEAN OILS FOR PROTECTIVE COATINGS: ALKYD-TYPE POLYMERS. WILMA J. SCHNEIDER, L.E. GAST and J.C. COWAN, Northern Regional Research Laboratory, Peoria, Ill. 61604.

By a simplified synthesis, 35 "alkyd" polyesteramides and urethane derivatives have been prepared and their film properties evaluated in the laboratory. This synthesis involves aminolysis of linseed or soybean oil with diethanolamine to produce a mixture of N,N-bis(2-hydroxyethyl) fatty amide (HEFA), glycerol, mono- and diglyceride and unreacted diethanolamine. Direct esterification of this mixture with diisocyanate or anhydrides forms hydroxy-terminated prepolymers of any desired hydroxyl level. Subsequent reaction with tolylene diisocyanate forms urethane-polyesteramides. The entire procedure takes only 8 to 10 hr and avoids the isolation and purification of HEFA. Air-dried films of these alkyd urethane-polyesteramides exhibit improved properties over the more linear products prepared previously; they dry faster to harder films, resist 5% hydrochloric acid for slightly longer times; and show excellent xylene resistance, much improved alkali resistance and good adhesion to metal. Film properties of these experimental urethane-polyesteramides compared well with those of four commercial alkyd resins, a linseed-urethane oil and a soy-urethane spar varnish.

36

KINETICS OF THE REACTION OF ISOPROPENYL ACETATE AND STEARIC ACID TO FORM ISOPROPENYL STEARATE. M. KOZEMPEL, J.C. CRAIG, JR., H.I. SINNAMON and N.C. ACOTRO, Eastern Marketing and Nutrition Research Division, ARS, USDA, Philadelphia, Pa. 19118.

A research program to develop an economically feasible process for making isopropenyl stearate has been undertaken. To this end an engineering study of the reaction kinetics was made. A mathematical kinetics model for the reaction of stearic acid with isopropenyl acetate to form isopropenyl stearate is presented. The model is a sufficiently accurate representation of the data to permit the engineering design of a continuous reactor and aid in scale-up. Though the reaction is reversible, the kinetics can be described mathematically most simply by a pseudo first order irreversible reaction model. The model predicts the rate of formation of isopropenyl stearate as a function of time, temperature, catalyst concentration and equilibrium concentration of isopropenyl stearate. Equilibrium is determined from an empirical equation as a function of the ratio of the original concentration of the reactants and is unaffected by temperature. The model predicts that the reaction can reach equilibrium in 10 min or less in relatively simple equipment.

37

POTENTIAL LUBRICANTS FOR CONTINUOUS CASTING OF STEEL: POLYOL ESTERS OF PARTIALLY HYDROGENATED SOYBEAN ACIDS. E.W. BELL, J.C. COWAN and L.E. GAST, Northern Regional Research Laboratory, Peoria, Ill. 61604.

In recent years continuous casting of steel into billets and slabs without going through the ingot stage has drawn widespread attention in the steel industry. Mold lubrication is vital in the continuous casting process to prevent sticking, to aid heat transfer, and to avoid heavy scum pickup. Crambe and rapeseed oils have been used extensively as lubricants. Because these vegetable oils are in short supply and because continuous steel casting is on the increase, a survey of possible substitute lubricants has been made. Trimethylololthane,

trimethylolpropane, trimethylolbutane and pentaerythritol esters of partially hydrogenated soybean fatty acids, derived from commercially hydrogenated soybean oils, have been readily prepared in good yields. Preliminary measurements of viscosities, smoke, flash and fire points of these polyol esters indicate that they are possible candidates for use as prime lubricants in the continuous casting of steel.

38

SUNFLOWERS: AMERICA'S MOST NEGLECTED CROP. J.A. ROBERTSON, Russell Research Center, Athens, Ga. 30604.

It has been said that the sunflower, *Helianthus annuus*, is one of America's most neglected natural resources. Although the sunflower is a native American wildflower, the significant developments in sunflower-breeding which led to its establishment as a cultivated crop occurred abroad. Limited acreage of confectionery and bird feed sunflower varieties has been grown in Minnesota and North Dakota for several years. However, recent interest in the United States in sunflowers as an oilseed crop has been due to the Russian's developing high yielding, high oil varieties. In 1967, these varieties were introduced into the United States from Canada for the first time. Sunflower oil is an excellent cooking oil. It also finds use as a salad oil and for making margarine, shortening and mayonnaise. The oil from seed grown in the northern United States typically contains 70% linoleic acid and has a high ratio of polyunsaturated to saturated fatty acids. This makes it desirable as an edible oil in light of evidence linking saturated fats to high blood cholesterol and incidence of heart disease. In contrast, sunflower oil from seed produced in the South generally contains 40-45% linoleic acid. Preliminary studies indicate that the southern oil with lower content of linoleic acid is more stable than northern oil. The effect of planting dates, location, and environmental temperatures on the fatty acid composition of northern and southern sunflower oils is reported. Studies also show that potato chips fried in sunflower oil are more flavorful after storage for four weeks than similarly treated chips fried in cottonseed-corn oil mixtures. Sunflowers also offer a promising new source of protein for both humans and livestock. The protein is highly digestible (90%) and possesses a high biological value (60%). Protein isolates prepared by the conventional method of extraction are greenish in color due to oxidation of chlorogenic acid. However, research underway indicates that development of a nearly white isolate is possible. Analysis of chemical, physical and organoleptic properties indicate that defatted sunflower kernels can be used in various food products. Defatted meal also can be used as a wheat substitute in bread and other bakery products.

39

SAFFLOWER: AN ANCIENT OIL CROP FINDING ITS PLACE IN TODAY'S MARKETS. GLENN FULLER, Western Regional Research Laboratory, ARS, USDA, Berkeley, Calif. 94710.

Safflower has been a useful oilseed crop since early historic times. Safflower oil began to achieve widespread use as an edible oil in the United States in the late 1950's because of its high polyunsaturate content. Even today much of its use and its ability to compete with other oils is based on its high content of linoleic acid. There are still many varieties of safflower available, however. Examination of the world collection known, brought forward a variety of safflower (IV=90) in which the oleic-linoleic acid ratio was essentially reversed from that of the normal variety (IV=144). A single gene was responsible for this reversal, an introduction of a new high oleic safflower came quite rapidly. Oleic safflower is reaching new markets quite different from those of ordinary safflower. A third type has also been found by Knowles to be of intermediate iodine value. The applications, economic considerations, and possible new uses of these oils will be discussed.

40

SESAME: CURRENT KNOWLEDGE OF COMPOSITION AND USE. C.K. LYON, Western Regional Research Laboratory, ARS, USDA, Berkeley, Calif. 94710.

Because of its good taste and outstanding stability, sesame oil has long been one of the most desirable edible vegetable

oils. Sesame meal is a valuable supplement for food and feeds because of the high methionine content of its protein relative to other oilseed proteins. Production of sesame seed in the United States has been small but is expected to increase with success in the development of nonshattering varieties. Current information on the composition, properties, processing and use of sesame will be discussed.

41

PRESENT STATUS OF GENETIC RESEARCH ON SESAME. D.M. YERMANOS, University of California, Riverside, Calif. 92502.

This paper will discuss current research on genetic aspects of sesame, and the potential for establishing it as a commercial crop.

42

RESPONSE SURFACE METHODS FOR EXPLORING MULTIFACTOR SPACE. LYNN B. HARE, Hunt Foods and Industries, Fullerton, Calif. 92634.

Abstract not available at press time.

43

DESIGN OF EXPERIMENTS WITH MIXTURES. R.D. SMITH, E. I. Dupont de Nemours and Co., Wilmington, Del. 19898.

Abstract not available at press time.

44

DESIGN FOR FACTORIAL EXPERIMENTS. HORACE P. ANDREWS, Rutgers University, New Brunswick, N.J. 08903.

Scientific investigations, whether in research and development laboratories, pilot plants, or in production for the improvement of processes, can make wide use of factorial experiments. These permit the efficient simultaneous investigation of several factors (two or more) and their interrelationships in a single experiment or a series of specially-related experiments. They are discussed in some detail, with particular emphasis on graphical concepts in the design and analysis of experiments for the exploration of factor space.

45

UPTAKE OF BLOOD TRIGLYCERIDES BY VARIOUS TISSUES. R.O. SCOW, National Institutes of Health, Bethesda, Md. 20014.

Blood triglycerides (TG) are carried in the blood stream in chylomicrons and very low density lipoproteins, which range in size from 0.08 to 0.6 μ and also contain small amounts of phospholipid, cholesterol and protein. Electron microscopic studies indicate that these particles do not cross the capillary wall in most tissues. There is considerable evidence that blood TG are hydrolyzed to FFA during uptake and that this process is catalyzed by lipoprotein lipase (LPL). The enzyme is found in all tissues that utilize circulating TG and its activity in individual tissues varies with nutritional and physiological states that affect TG uptake. LPL activity and TG uptake in adipose tissue, for example, are both decreased by fasting, diabetes and lactation, and increased by refeeding and insulin. Lactation also increases LPL activity and TG uptake in mammary tissues. The concurrent decreased activity in adipose tissue and increased activity in mammary tissue serve to divert blood TG from storage in adipose tissue to milk production in mammary tissue. Perfused rat adipose tissue has been used in this laboratory, to study the relationship between LPL activity and TG uptake. Studies with doubly labeled chylomicron-triglyceride showed that hydrolysis occurs outside of the blood stream and that two thirds of the FFA released are incorporated into tissue TG and the rest are released to the blood. Infusion of heparin caused immediate release of LPL to the blood and decreased the amount of chylomicron-TG hydrolyzed in the tissue. Electron microscopic cytochemical studies of adipose tissue perfused with chylomicrons showed that hydrolysis of blood glyceride by LPL occurs within the capillary endothelial cells and in the subendothelial space near the pericytes but not in the capillary lumen nor near the fat cells. The results indicate that the fatty acids of blood TG taken up by the

tissue cross the capillary endothelium as glycerides and FFA, and cross the extravascular space as FFA. Preliminary results indicate that a similar uptake may occur in mammary tissue and muscle. Although earlier studies suggested that intact blood TG might be taken up by liver parenchymal cells, recent experiments in several laboratories have shown that blood TG are hydrolyzed to FFA in other tissues before their fatty acids are taken up by the liver.

46

EXPERIENCES WITH FAT EMULSIONS IN LONG TERM COMPLETE PARENTERAL NUTRITION. H.C. MENG, Vanderbilt University Medical School, Nashville, Tenn. 37203.

It seems apparent that fat, as a dietary component, is an essential nutrient. It furnishes not merely calories which may be replaced by carbohydrate, but also provides other essential functions for body economy. In parenteral nutrition given no fat, concentrated carbohydrate solution has to be administered to furnish adequate calories and to maintain reasonable intake of fluid volume; central vein delivery is necessary. Fat emulsion can be infused into a peripheral vein without producing damages to the vessel. Furthermore, reports indicate that polyunsaturated fatty acids are needed in long term parenteral nutrition especially in infants and children. This review will present evidence showing that intravenously administered fat emulsions are biologically utilized in metabolic studies and in long term parenteral nutrition. Other problems such as oil or fat in the formulation of fat emulsions, rationales in the use of fat emulsions in parenteral nutrition, and the possible pharmacological effects of fat emulsions will also be discussed.

47

THE SIGNIFICANCE OF THE RATIO OF FAT, CARBOHYDRATES AND AMINO ACIDS IN COMPLETE PARENTERAL NUTRITION. K.L. IKLUBE, University of Frankfurt/Main, West Germany.

In two experimental series, dogs received 5 gm or 9 gm fat intravenously per kg body weight and day, and in addition minerals and vitamins. The test lasted 20 to 32 days. In all animals, a considerable hyperlipemia developed which was much more marked when soybean oil emulsions were used than when cotton seed oil emulsions were given. During the first 10 days, the weight of the animals hardly decreased although the nitrogen balance was extremely negative. Then, however, increasingly severe disturbances occurred. Anemia developed in all animals. Most of the animals showed bloody mucous secretions from the mucous membranes of the nose, the conjunctiva and the intestine. Autopsy of the organs revealed massive fat storage, especially in kidney, lungs, spleen and liver. Fat storage was also found in placentas of pregnant rats and dogs when fat was infused in a dosage corresponding to 100 gm fat in a man of 60 kg body weight. In contrast to this, fat storage could be found in the human placentas neither histologically nor chemically when women were given 50 gm fat intravenously during delivery. Although a considerable increase of maternal fat fractions in serum resulted, a similar increase could never be observed in the child. Finally, the problem of practical importance was checked in which way parenteral nutrition must be composed with regard to calories, amino acids, carbohydrates and fats in order to obtain a most favorable nitrogen balance. We found that the best nitrogen balances were always obtained when the amount of calories given corresponded to 1% of the basal turnover and when 20% of the calories were derived from amino acids, 30% from fats and 50% from carbohydrates.

48

DEPLETION OF ESSENTIAL FATTY ACIDS AFTER LONG TERM FAT FREE PARENTERAL NUTRITION. D.A. COATS, Royal Australasian College of Surgeons, Melbourne 3000, Australia.

Linoleic acid deficiency in adult man may cause no symptoms except in conditions of stress such as after injury or in the postoperative period. In these circumstances, there may be delayed convalescence with impairment of wound healing, the occurrence of a skin rash on the trunk and limbs, failure of erythropoiesis and an associated thrombocytopaenia due to failure of platelets to bud off from megakaryocytes in bone

marrow. These features may first become manifest during long term fat free parenteral nutrition and may be reversed by giving intravenous linoleic acid at a rate of about 0.1 g/kg body weight/day. Linoleic acid deficiency may be diagnosed by the nearly complete disappearance of arachidonic acid serum and its replacement by $\Delta^5, 8, 11$ eicosatrienoic acid. Further biochemical features of the syndrome will be discussed.

49

THE USE OF FAT EMULSIONS FOR THE TOTAL INTRAVENOUS FEEDING OF THE INFANT. A.G. CORAN, Naval Medical Research Institute, Bethesda, Md. 20014.

A new program for the parenteral feeding of postoperative newborns will be presented. The regimen is based on a synthetic amino acid solution and a soybean oil fat emulsion. This feeding has two major advantages over existing hypertonic glucose as the major source of calories. First, glucose tolerance with glucosuria and osmotic diuresis is eliminated, second, the reduced hypertonicity due to the liberal use of the isotonic fat emulsion allows the feeding program to be administered entirely through peripheral veins for extended periods of time. This feeding program has been shown to sustain retention of nitrogen and minerals in amounts and ratios similar to those found in normal newborns, even in the immediate postoperative period. In the initial phase of this study, 32 newborns were treated in this way following surgery for life-threatening congenital anomalies; 26 survived. Since this first evaluation, over 75 infants have been placed on this intravenous feeding regimen.

50

THE ROLE OF FAT AS CALORIC SOURCE IN PARENTERAL NUTRITION. K. SCHULNIS and H. BRISBAETH, Institut für Experimentelle Ernährung e.V., Erlangen, West Germany.

When the human maintenance requirements are met parenterally over a longer period, it has been proposed that the portion of fat should amount to approximately 30% of total calories. Parenteral nutrition is most frequently used in surgical patients who often need even an increased supply of calories. As a parameter for caloric balance of the organism, an equilibrated nitrogen balance may be taken. In the early postoperative period, however, fat infusions did not result in additional increase of nitrogen retention. This is possibly due to the fact that under the influence of traumatic stress the turnover of endogenous fat has already been activated at its maximum. Balance studies before and after surgery are to elucidate this relation further.

51

CHECKING TEMPERATURES IN LABORATORY INSTRUMENTATION. W.H. KING, Southern Marketing and Nutrition Research Division, ARS, USDA, New Orleans, La. 70119.

Occasionally it becomes necessary, or desirable, to check temperatures at important points in the gas chromatograph and other laboratory instruments to confirm or eliminate suspicion that the instrument indicators are not functioning properly. Some of the newer instruments have only over- and under-indicators for the temperatures set manually by dials or by automatic programmers. Some of the instruments are difficult or impossible to check with thermometers, requiring special probes which can be inserted at the proper point and which have external readouts. This paper presents a brief description of the newer instrumental techniques of temperature measurement and recording, together with the time-honored classical methods and adaptation of modern equipment to those methods. Details are given from our experience in obtaining reliable laboratory indications and records at various points in laboratory instrumentation by the simplest and least expensive means available.

52

NMR CHEMICAL SHIFT REAGENTS IN STRUCTURE DETERMINATION OF LIPID DERIVATIVES. I. BAK, G. WENZEL, AND PRINCIPLES, DANIEL SWERN and J.P. WENZEL, Temple University, Philadelphia, Pa. 19122.

¹H NMR is an extremely useful technique for the structural determination of many types of organic compounds, and it

has been widely used by lipid chemists. The technique, however, is severely limited in scope and utility with most long chain compounds because the majority of the chain methylene protons are, for all practical purposes, magnetically equivalent. These protons produce a broad, featureless signal of overlapping resonances which precludes proton identification, counting, and determination of coupling constants. Recently, it has been demonstrated that complexes of certain rare earth elements particularly cerium(III) and praseodymium(III), markedly expand the NMR spectra of compounds containing cerium and praseodymium groups with nonbonding electrons that can coordinate with the rare earth metal. The protons are expanded because the magnetic environment of the protons near the coordination site is different from the environment of distant protons, and simple first order signals are frequently obtained. In rare earth metal complexes are referred to as chemical shift reagents (csr). The chemical shift displacement is directly related to the distance between the protons in question and the complexed metal atom; the smaller the distance the greater the shift. The magnitude of the displacement of chemical shift is also related to the molar ratio of csr to substrate, and is generally optimized as the ratio approaches 1. In many cases a linear relationship is observed between the effected chemical shift and the molar ratio of csr to substrate. Complexes containing Eu (III) and Pr (III) complement each other since, relative to tetramethylsilane, Eu complexes shift proton signals downfield and Pr complexes shift them upfield. Some typical NMR spectra with and without csr of compounds useful as models for lipid derivatives will be described. The magnitude of displacement in chemical shifts in the presence of csr by different functional groups with nonbonding electrons will also be discussed.

53

NMR CHEMICAL SHIFT REAGENTS IN STRUCTURAL DETERMINATION OF LIPID DERIVATIVES. II. SCOPE AND LIMITATIONS. J.P. WENZEL and DANIEL SWERN, Temple University, Philadelphia, Pa. 19122.

We are currently investigating the scope and limitations of using chemical shift reagents (csr) to expand the amount of structural information that can be derived from the NMR spectra of saturated, unsaturated and chain substituted fatty alcohols, amines, acids, methyl esters and related derivatives; mono-, di- and triglycerides; phospholipids; and other long chain compounds. In this paper, we shall discuss the scope and limitations of using csr with long chain compounds to determine the position of double bonds, chain branching and substituents; NMR spectra will be presented to illustrate the relevant points.

54

TRANSIENT NMR SIGNALS FROM SOLID AND LIQUID FATS. G.A. PERRY, The Fraxis Corporation, San Antonio, Tex. 78298.

The importance of a rapid solid fat determination in the fats and oils industry is well known. Several new instruments having this capability have recently become available. In particular, the development of inexpensive and yet reliable NMR instruments have made this approach practical for plant quality control use. The most common of these instruments is the wide-line instrument which uses the steady state NMR technique. Another NMR technique is being used, namely the transient NMR technique which offers certain basic advantages and is of particular interest here. Solid and liquid signals from several hydrogenated fats and oils and consumer products will be discussed. The application to the determination of the degree of hydrogenation of oils will also be discussed.

55

DETERMINATION OF RESIDUAL ACETONE AND ISOPROPANOL IN OILSEED MEALS AND FLOURS BY DIRECT GAS CHROMATOGRAPHY. H.P. DUFREY and SARA P. FORB, Southern Marketing and Nutrition Research Division, ARS, USDA, New Orleans, La. 70119.

A simple and rapid procedure was developed for the determination of residual acetone and isopropanol in oilseed meals and flours. A small sample of meal or flour is placed between glass wool plugs in the liner of the injection port of a gas

chromatograph. A small aliquot of water is added on top of the sample, and the liner is inserted in the injection port which is sealed with the septum nut. The flow of the carrier gas sweeps the residual solvent and other volatiles rapidly into the gas chromatographic column which is temperature programmed to resolve the solvent and other materials. The concentration of residual acetone and isopropanol is easily determined by measuring the area of the appropriate peak of the chromatogram and comparing it with a calibration chart. This technique also permits detection of acetone condensation products, e.g., diacetone alcohol, if they are present.

56

DETERMINATION OF MESITYL OXIDE AND DIACETONE ALCOHOL IN OILSEED MEALS AND FLOURS. SARA P. FOXE, H.P. DUBEY and E.T. RAYNER, Southern Marketing and Nutrition Research Division, ARS, USDA, New Orleans, La. 70119.

Two methods are described for the determination of mesityl oxide and diacetone alcohol in oilseed meals and flours which were extracted with acetone. One is a modification of the Todd azotropic distillation procedure. The other is a simple direct gas chromatographic procedure. A small sample is inserted into the liner of the injection port, and the acetone condensation products (mesityl oxide and diacetone alcohol) are eluted from the sample and swept onto the column by added water and the flow of helium carrier gas. The mesityl oxide, diacetone alcohol and other volatiles are resolved by temperature programming. A number of samples were analyzed by both techniques. The results are compared and the relative merits of the two procedures are discussed.

57

A RAPID METHOD FOR DETERMINING PROTEIN IN SOYBEAN MEAL. B.D. DEACON, Market Quality Research Division, ARS, USDA, Beltsville, Md. 20705.

Preliminary findings indicate that protein content of soybean meal may be determined in less than 10 min. by use of a modified biuret procedure. Finely ground soybean meal and half normal sodium hydroxide in 30% isopropanol alcohol is stirred with copper hydroxide. A significant portion of the protein did not dissolve at room temperature, but stirring at 70°C dissolved approximately 95%. The absorbance of the filtered solution is measured at 550 nm and the protein estimated from a calibration chart. Analysis of 24 samples of soybean meal obtained from several sources and ranging from 42% to 54% protein content showed a coefficient of correlation of 0.931 and a standard error of estimate of $\pm 0.41\%$ when compared with the Kjeldahl protein.

58

COMPOSITIONAL STUDIES ON HYDROGENATED OILS CONTAINING C₁₈ FATTY ACIDS. H.B.S. COMACHER and R.D. PAGE, Department of National Health and Welfare, Ottawa, Ontario, Canada.

Recent nutritional studies on liquid or partially hydrogenated oils containing C₁₈ fatty acids have indicated abnormal cardiac lesions in the rat. Although the composition of the liquid oils is well documented, information on that of the hydrogenated oils used in Canada is lacking. This paper describes results from analytical studies on the composition of partially hydrogenated rapeseed, herring, whale and seal oils. The method includes an initial separation of the methyl esters by preparative gas liquid chromatography (GLC) into fractions of equal chain length. Each fraction is subsequently studied in detail by GLC, argentation thin layer chromatography, IR, and UV spectroscopy, and oxidative degradation. The results are discussed in relation to the compositions of the liquid oils.

59

GAS CHROMATOGRAPHIC DETERMINATION OF C₁₈ AND C₁₉ MONOENE AND DIENE FATTY ESTERS. E.A. EMMEN, Northern Regional Research Laboratory, Peoria, Ill. 61604.

A gas chromatographic (GC) method has been developed by which the per cent of *cis*- and *trans* in monoene and diene C₁₈ fatty esters can be quantitatively determined. To separate

cis- and *trans*-monoene and diene isomers with packed GC columns the fatty esters were first stereoselectively epoxidized with peracetic acid. The epoxidation procedure is simple and *cis*- and *trans*-epoxides can be analyzed without isolation from the reaction mixture. Positional and geometric isomerization of the double bond does not occur during the epoxidation reaction. Synthetic mixtures containing *cis*- and *trans*-A₆, A₉ and A₁₂ C₁₈-monoene isomers were separated into *cis* and *trans* fractions with no interference due to the double bond position. The A₁₅ *trans*-monoene isomer does overlap the *cis* peak and complicates the analysis. Diene mixtures containing *trans*-*trans*-, *cis*-*trans*-, and *cis*-*cis*-9,12-octadecadienes were also successfully analyzed by GC after epoxidation with peracetic acid. Each diene isomer forms two epoxy diastereomers. The *cis*-*cis*-dioxyacetate diastereomers are separated and the first of these peaks overlaps the *cis*-*trans*-dioxyacetate peak. However, the total percentage of *cis*-*cis*-dioxyacetate can be calculated since the ratio of the two *cis*-*cis*-dioxyacetate diastereomers is constant. The GC analyses utilize standard stabilized ethylene glycol succinate columns and requires no exotic equipment, reagents or unusual techniques.

60

CHAR-ASHING OF GLYCERIDE OILS BEFORE DETERMINATION OF THEIR COPPER AND IRON CONTENTS BY ATOMIC ABSORPTION. C.D. EVANS, G.R. LUST and L.T. BLACK, Northern Regional Research Laboratory, Peoria, Ill. 61604.

Trace amounts of copper and iron were determined by a char-ashing technique on a sample of molecular-distilled oil, copper-hydrogenated oils and on salad oils containing added copper chromite or copper salts. Char-ashing is a total carbonization of the oil in a Vycor dish on a hot plate with sufficient heat to distill destructively the oil (approximately 300°C), but at a temperature low enough so that the oil does not catch fire. Char-ashing, coupled with the atomic absorption analysis, gives reproducible results on a salad oil of 0.025 ± 0.002 ppm copper and 0.082 ± 0.012 ppm iron. Agreement was within 1% to 2% between char-ashing and the direct solvent method when levels of the two trace metals were high enough to be analyzed by direct atomic absorption analysis. Copper can be analyzed at levels of less than 10 ppb in edible oils by this char-ashing technique.

61

PHYSICO-CHEMICAL ASPECTS OF FLAVOR PERCEPTION. R.I. HENKIN, National Heart and Lung Institute, Bethesda, Md. 20014.

Normal perception of flavor depends upon the presence of normal taste and smell acuity. Absence or diminution of either sensory modality interferes with the ability to obtain flavor from food or drink. Both taste and smell depend upon two classes of physicochemical events called peripheral and neural events. Neural events depend upon depolarization of receptor nerves, conduction of impulses to the central nervous system, and integration of impulses into meaningful bits of information. Peripheral events depend upon receptor events and are primarily biochemical in nature. For taste, these events occur during the interaction (binding) of tastant and protein(s) at the membrane of the receptor cell(s) of the taste bud and are controlled, in part, by the interaction between thiol and transition metal ions. Conditions which increase thiol content or decrease metal content at the receptor diminish taste acuity and thereby decrease flavor perception. For smell, these events occur during the interaction (binding) of odorant and protein(s) at the membrane of the cilia of the mitral cell(s), presumably the membrane of the cilia of the mitral cells of the olfactory epithelium. These receptors are embedded in the olfactory epithelium. The interaction is controlled, in part, by the presence of vitamin A alcohol at the membrane. Conditions which decrease vitamin A alcohol content at the receptor diminish olfactory acuity and thereby diminish flavor perception. Maintenance of normal physicochemical balances of these chemical moieties is needed to preserve normal flavor perception.

62

APPLYING SUBJECTIVE FLAVOR TESTS. J.F. CAULI, Kansas State University, Manhattan, Kan. 66502.

No one method of "taste testing" is applicable to all odor and flavor problems. Selection of the optimal method(s) is based on understanding what information each method can provide and what information is needed. Underlying the methodology are certain principles that are universal to all such testing, plus those related to the specific type of product, in this case, fats and fat-bearing foods.

63

FLAVOR POTENTIATION. R.L. WASSON, Monsanto Company, St. Louis, Mo. 63166.

A specific ingredient in any food system can affect flavor in one of three ways: it can add one or more character notes to the flavor; it can produce no perceptible change; it can alter the flavor without being recognized itself. Flavor potentiation is produced by ingredients which fall into the third class of activity. A wide range of ingredients have been found to be active as flavor potentiators in certain food systems. Studies of natural product ingredients have led to the development of a series of proprietary new potentiator systems which produce significant flavor changes in certain oil-based food products. These materials have been termed time factors, fullness effect, and mouthfeel factor. Several examples of the flavor changes produced by these potentiators will be discussed along with examples using other potentiators. Studies have yielded good information about complex flavor systems and should lead to a better understanding of some of the mechanisms involved in taste perception.

64

GRAS LIST REGULATIONS. E.J. MEEWIN, McCormick and Co., Inc., Cockeysville, Md. 21030.

A review of the GRAS list regulations must start with the conception in the passage of the Food, Drug and Cosmetic Act of 1958, and the subsequent regulations issued in the Federal Register. Important, even major aspects, are the interpretations by various groups, some soundly based and carefully worked out, others based on misconceptions of the intent of the law, or the nature of safety evaluation. The nature and status of reviews of levels and quantities of GRAS items will be discussed.

65

SOYBEAN PROTEIN FLAVOR COMPONENTS: A REVIEW. J.C. COWAN, J.J. RACKIS and W.J. WOLF, Northern Regional Research Laboratory, Peoria, Ill. 61604.

This report on the flavor components of soybean protein products examines recent work including: studies on sensory evaluation of commercial flours, concentrates and isolates; extraction of flavor components from soybean flakes with hexane-alcohol azeotropic mixtures; the application of proteolytic enzymes to improve flavor; and the effect of lipoxigenase on soy beverage flavor.

66

ENZYMATICALLY PRODUCED FLAVORS FOR FATTY SYSTEMS. J.H. NELSON, Dairyland Food Labs, Inc., Waukesha, Wis. 53186.

Technology has been developed for the production of flavor systems via the controlled enzyme modification of fats. Lipases and esterases from various sources are utilized. Fats modified include milk fat, poultry fat and meat fat. A wide range of flavor profiles can be produced via control of enzyme, fat, enzyme modification conditions, and processing prior to and subsequent to enzyme modification. Applications for the flavor systems include margarine, imitation dairy products, confections and prepared foods.

67

AQUATIC FATTY ACIDS AND MAN. R.G. ACKMAN, Fisheries Research Board of Canada, Halifax, Nova Scotia, Canada.

In round figures, world fish harvests can be set at just over 60 million metric tons a year. Man kind probably consumes about one million metric tons of aquatic fats and lipids in a year in fish and fishery products, including crustacean and mollusc lipids, and nearly another million metric tons in the

form of refined and hydrogenated edible fats of marine origin. The latter are now virtually all fish oil, with only a little whale oil. The purpose of this paper is to contrast the properties and fatty acid compositions of natural and processed aquatic fats and to examine their contributions to the dietary fat intake of certain populations.

68 METABOLISM OF NEUTRAL LIPIDS IN *SQUALUS ACANTHIAS*. J.R. SARGENT, R.R. GATTEN and R. MCINTOSH, University of Aberdeen, Scotland, U.K.

Neutral lipids from *Squalus* liver contain approximately 60% triacylglycerols (TAG), 85% alkyl diacyl glycerols (ADG) and small amounts of wax esters (WE) and cholesteryl esters (CE). Muscle neutral lipids contain about 80% TAG, 20% ADG, and traces of WE and CE. Serum neutral lipids, however, contain 35% of TAG, 19% of ADG, 33% of WE and 14% of CE. Cell free preparations of liver catalyze the biosynthesis of TAG, WE and ADG by energy dependent processes. The rate of TAG synthesis is some three times the rate of WE synthesis and at least 10 times the rate of ADG synthesis. While fatty alcohol is incorporated directly into the alkyl linkage of ADG in liver, the major route of fatty alcohol metabolism in liver is direct incorporation into wax esters and oxidation to fatty alcohol by an NAD dependent, essentially irreversible process. Turnover studies with (3H) oleyl alcohol injected intravenously into *Squalus* show that, after 24 hr, free fatty acids (FFA) TAG and ADG in liver contain 1.33%, 0.77% and 0.42% of the injected dose of radioactivity, respectively. In muscle FFA, TAG and ADG contain 19.4%, 1.45% and 0.40% of the dose, respectively, after 24 hr. Negligible radioactivity is found in fatty alcohol in these tissues. All neutral lipid fractions are labelled in serum and turned over completely within 48 hr. FFA and TAG (but not 0.13% of the injected dose after 24 hr) are much more heavily labelled than other serum lipids. Factors operating to cause a rapid turnover of TAG in *Squalus* under conditions where ADG is relatively conserved will be discussed.

69 METABOLISM OF OLEYL ALCOHOL AND OLEIC ACID IN FISH. D.M. SAND, G.H. RAHN and H. SCHLENK, The Hormel Institute, Austin, Minn. 55912.

The metabolism of these compounds was studied by feeding them in labeled form to female gourami (*Tetraodon lineatus*). Within 8 to 6 hr most of the dietary oleyl alcohol is oxidized to acid which is incorporated into triacylglycerols of intestinal lipids. A much smaller portion of the ingested oleyl alcohol appears as the alcohol moiety of the wax esters in the intestinal lipid. Apparently, this portion has been directly esterified. Wax esters are present also in the blood lipid for about 16 hr after ingestion of oleyl alcohol. However, wax esters are not found in intestinal or blood lipids when oleic acid is fed. After 36 hr, about 75% of the absorbed 18:1 compound is found as alcohol and acid in roe wax esters. This amount of the dietary alcohol or acid is incorporated mainly into immature eggs. Most of the mature eggs of the roe, which are laid within two weeks after feeding, have lower specific radioactivity than the eggs remaining in the fish. Highest specific radioactivity was found in eggs laid four weeks after feeding. When radioactive eggs are fertilized, virtually all of the 18:1 radioactivity is lost within three days. This rapid catabolism of 18:1 in the developing embryo and fish is in strong contrast to the extremely slow turnover which had been found for 18:1 in the adult fish.

70 THE MOLECULAR STRUCTURE OF TRIACYL GLYCEROLS RESISTANT TO PORCINE PANCREATIC LIPASE. USHA VARANASI and DONALD C. MALINS, Pioneer Research Laboratory, Seattle, Wash. 98102.

Recent work from this laboratory (Varanasi and Malins, 1970, 1971) revealed the presence of large amounts of iso-valeric acid and long chain iso acids in the lipids of acoustic tissues of marine porpoises. We report that porcine pancreatic lipase hydrolysis of triacylglycerols of the melon followed by thin layer chromatography resulted in the isolation of a resistant fraction containing 85 mole per cent of total iso

acids. Studies on the molecular structures indicated that these triacylglycerols contained primarily two moles of isovaleric acid and one mole of long chain iso acids. The occurrence of two isovaleryl chains on the glycerol moiety severely restricts the possible number of isomeric triacyl glycerols. Thus, these triacylglycerols are significantly different from those of most animals that have complex mixtures of acids on each position of glycerol. Studies on the structures of lipase resistant triacylglycerols from acoustic and nonacoustic tissues will be discussed.

71 STUDIES ON HIGHLY UNSATURATED TRIGLYCERIDES AND PHOSPHOLIPIDS. N.R. BOVINO, Texas A and M University, College Station, Tex. 77843.

Tri-glycerides and phospholipids of liver and blubber of a Set whale caught in Antarctic waters were fractionated according to their degree of unsaturation by newly developed techniques of silver nitrate thin layer chromatography (TLC). Whole liver and blubber triglycerides were composed of at least seven distinct fractions. Saturated and monoenoic fatty acids were present in all fractions whereas groups of polyunsaturated fatty acids of equal degree of unsaturation appeared in individual fractions only. Whole liver phosphatidyl choline (PC) and their component molecular species were each separated by additional hydrolysis of the fractions with phospholipase A₂ from snake venom. Whereas the molecular species present in PC were different from those in PE, a common pattern, based on the degree of unsaturation of the component fatty acids, was apparent. An attempt was made to mimic the whale triglycerides by feeding pigs a diet containing shrimp as the only lipid source. The silver nitrate TLC analysis of the pig adipose tissue triglycerides showed that whereas the triglycerides of pig and whale were different, the patterns of distribution of their fatty acids were similar.

72 THE TOXICOLOGY OF FATS AND OILS. KENNETH MORGAREIDCK, Food and Drug Research Laboratories, Inc., Waverly, N.Y. 14892.

This title does not imply that any major class of foods is unique in attracting the attention of toxicologists. In addition to a number of inherent properties of special interest to nutritionists, physiologists and pharmacologists, edible fats and oils are derived from our now contaminated biosphere and, hence, may serve as carriers for a variety of unintentional residues including pesticides and industrial contaminants carried by air and water or deposited in soils. The relative importance of such residues as potential health hazards is emphasized by the dominant position occupied by fats in human diets. Furthermore, no other class of foodstuffs has been so thoroughly manipulated, altered, rearranged and modified by chemists, who boast of tailor-made fats to meet any food processor's need. The unique physical properties of these materials are important in terms of texture, palatability and flavor of foods in general. However, much work has been done and more is needed to relate the altered molecular configurations of these water insoluble materials to their biological absorption and metabolism. The same concern applies to degradation and oxidation products of fats subjected to high temperatures for prolonged periods of time. Finally, some attention must be paid to those naturally occurring substances which are either associated with fats or comprise constituent moieties of lipid molecules that have known toxic properties. Some of these are fatty acids of peculiar configuration; others are lipoproteins; and some are simply hitch-hiking fat-soluble organic compounds. These instances only underline the fact that not all natural substances are fit for food.

73 INFLUENCE OF NINE DIETARY FATS ON CARDIAC FIBROSIS AND CALCIFICATION IN RATS. HANS KAUNZ, and RUTH E. JOHNSON, Columbia University, New York, N.Y. 10032.

Groups of 40 male rats of the Charles River CD strain were fed, from weaning, purified diets containing 20% of either fresh or mildly oxidized cottonseed oil, olive oil, corn oil, soybean oil, chicken fat, beef fat, butter, lard, or a

saturated medium chain triglyceride mixture, MCT. Complete autopsies were carried out on all rats to determine terminal pathological lesions. There were significant differences among the groups fed different fats with regard to neoplastic and degenerative diseases. Hearts frequently showed focal fibrosis, and the degree of this fibrosis was classified from 0-4+ in a blind test. Combined data from corresponding groups given fresh and oxidized fats with regard to 3+ and 4+ cardiac fibrosis are as follows: corn oil, 57/79; cottonseed oil, 50/80; soybean oil, 41/79; olive oil, 43/79; MCT, 20/40; lard, 35/79; beef fat, 36/80; chicken fat, 31/80; butter, 26/79. A chi square analysis (8 d.f.) gave a $P < .001$. Some of these H & E stained sections (particularly those with 3+ and 4+ fibrosis) showed what appeared to be calcification. Therefore, sections from hearts with marked focal fibrosis were stained for calcium with Carr's stain (chloranilic acid). This calcification, which occurred in the muscle fibers rather than in the fibrotic foci, was also graded from 0-4+ in a blind test. The incidence of 3+ and 4+ calcification was as follows: corn oil, 31/56; olive oil, 23/48; cottonseed oil, 23/49; beef fat, 15/36; soybean oil, 13/41; chicken fat, 8/30; butter, 5/26; lard, 4/34. Chi square analysis (8 d.f.) gave a $P < .001$. Thus, approximately the same fats which had induced a higher incidence of severe focal fibrosis were associated with more severe muscle fiber calcification even when only hearts with comparable focal fibrosis were studied.

74 A POSSIBLE METHOD FOR THE DETECTION OF NITROSAMINES IN FATS AND OILS. LISELOTTE HEDLBE, Abteilung für Experimentelle Therapie der Universität Freiburg, Freiburg, West Germany.

It has been reported that rats fed soybean oil in a purified diet have a higher incidence of malignant neoplasms than do rats fed some other fats. Therefore, a study was undertaken to determine whether nitrosamines, which are known carcinogens could be responsible for the increased tumor rate. Soybean oil was transmethylyated and treated with urea to remove the urea-adduct-forming esters. The non-adduct-forming material was subjected to preparative thin layer chromatography in silicic acid and three zones (R_F values 1-4, 4-8 and 8-10), were scraped off separately and extracted with ethyl acetate. The extracts were steam-distilled in the presence of NaOH and methylene chloride extracts of the concentrated distillates were examined by gas chromatography with a flame ionization detector and nitrogen detector. The extracts from the original R_F 4-8 and 8-10 zones gave peaks corresponding to dimethyl, diethyl, dipropyl and dibutyl nitrosamines (DBNA) with the ionization detector, and these peaks were increased by addition of the corresponding nitrosamines. However, with the nitrogen detector, only a peak corresponding to DBNA could be detected after the extracts had been concentrated and diluted in hexane; this peak was increased by addition of DBNA. For final identification, the extracts must be further purified and a derivative formed, such as 5-nitro-2-hydroxy-benzyl-derivative after reduction to the corresponding hydrazine.

75 CARDIAC LIPIDS IN RATS AND GERBILS FED OILS CONTAINING C₈ FATTY ACIDS. JOYCE L. BEARE-ROGERS, Food and Drug Research Laboratories, Ottawa, Canada.

Erucic acid of rapeseed oil or castor oil increased the amount of cardiac fatty acids. Whole or seal oils low in C₈ fatty acids produced little effect on the amount of lipids in the heart. After prolonged feeding on this oil, liquid rapeseed oil partially hydrogenated herring oil, the incidence of cardiac necrosis was high. Gerbils receiving a diet containing 6% erucic acid showed a peak in lipid deposition at four days. These animals appeared to be suitable for testing the early myocardial influences of various C₈ fatty acids.

76 LONG TERM NUTRITIONAL STUDIES OF FRYING FATS. KONRAD LANG, Schwarzwaldrasse 71, 7812 Bad Krozingen, West Germany.

At a previous meeting of this society, nutritional studies of oils used for deep frying were reported. In long term

feeding tests, rats received soybean oil and partially hydrogenated peanut oil, which had been used for frying fish at a level of 10% in the diet. The oils had been used for frying fish at 175°C in an electrically heated stainless steel fryer under practical industrial fish frying conditions. In the first generation 772 weaning rats, divided into eight groups were given the fats for their entire life span. Compared with the controls, there were no differences with regard to growth with food efficiency, reproduction and pathobiology of the main organs. It was especially striking that the survival rate in the earlier periods of the experiment was significantly higher when heated fats were given. In the meantime, the same result has also been obtained with a second generation. After 33 months of feeding, the survival rate was significantly higher in those groups receiving the heated fats. The examination of the fats as 40-13 polycyclic hydrocarbons revealed that their concentration had largely decreased in the heating process.

77

THE PHYSIOLOGICAL EFFECTS OF WHEAT GERM OIL ON HUMANS IN EXERCISE. T.K. CURETON, JR., University of Illinois, Champaign, Ill. 61820.

Forty-two studies have been conducted using children, young adults and middle-aged men: 21 experiments used parallel matched groups, one on wheat germ oil (WGO) and another on placebo; 8 experiments used training to a plateau and then introduction of the WGO to see if any further gain resulted; 11 experiments used single cases, each his own control, alternating a year on and a year off, or on some other substance. The feeding was 10 X 6 min (or 20 X 3) capsules of Vobin WGO, fresh, whole, unboiled, daily for 4 to 20 weeks in various experiments, taken usually with a half pint of milk at the end of each day's workout (WGO = 1050 mg of oil, 2.64 mg of tocopherol, capsulated at 0°C). The content is estimated: 52.3% linoleic acid plus 0.011% of octacosanol (C₂₈H₅₈OH) in refined corn oil, vitamin E and 11.8% palmitic acid, 28.1% oleic, 3.5% linoleic and also unassignable fraction of phosphatides, phytosterols and other unidentified substances. Placebos used were refined corn oil, cottonseed oil and deuterated lard with vitamin E added to equal that in WGO. The results show statistically significant effects on several types of endurance, total body reaction times, pre- and post-exercise intervals, brachial pulse waves, pulse rate tests, pre-exercise intervals (ICP) of the ECG, basal metabolism, flicker fusion frequency, oxygen intake and delt tests, were used with some subjects improving and others not. Results indicate that six weeks or more of such feeding produces improved performances, improved nervous economy and stability of metabolism, advantageous in physical fitness work.

78

DIETARY PROTEIN, MEDIUM CHAIN TRIGLYCERIDES AND CHOLESTEROL METABOLISM. HANS FISHER and J.J. KENNEY, Rutgers University, New Brunswick, N.J. 08903.

On a normal diet, adequate in protein and containing corn oil or oleic acid, there is considerable cholesterol absorption in the chicken as deduced from plasma and liver cholesterol measurements. If medium chain triglycerides (MCT) are substituted for corn oil, there is essentially no increase in plasma or liver cholesterol level even when high dietary levels of cholesterol are fed. Reducing the level of dietary protein causes a sharp increase in utilization of dietary cholesterol as seen from increased plasma and liver cholesterol levels. This effect is particularly striking for diets containing MCT. The relationship of these findings to bile acid metabolism and sterol synthesis and excretion will be discussed.

79

RADIOASSAY OF ³H, ¹⁴C AND ³H AND ³H LIQUID SCINTILLATION D.L. HORROCKS, Argonne National Laboratory, Argonne, Ill. 60439.

The extensive use of radioactive tracers has created many problems in methods of radioassay. Of particular difficulty are methods for the radioassay of tracers which are pure beta emitters. Those which emit only low energy beta particles are the most difficult to measure. Liquid scintillation

techniques have proven to be the best method for the radioassay of these low energy beta emitters. The organic chemistry most commonly used in biological and radiochemistry experiments are ³H, ¹⁴C, ³⁵S and ³²P, which are all pure beta emitters. Liquid scintillation techniques have been developed for the radioassay of these tracers in a wide variety of samples. Liquid scintillation systems are available which will measure ³H with counting efficiencies of 50-60% in unquenched hydrocarbon samples or up to 15% for emulsions containing 25-40% water. Higher energy beta emitters can be measured with higher counting efficiencies: ¹⁴C at 90%, ³⁵S at 90% and ³²P at 100% in unquenched homogeneous samples. Liquid scintillation techniques are available which allow for the measurement of two tracers in the same sample and labeling. The counting efficiency for a given nuclide can be altered by impurities in the sample. Changes in counting efficiency due to sample changes can be determined on each sample by use of external gamma ray sources which do not alter the sample. For low level counting the actual background is very important and has to be determined as a function of the amount of sample quenching also.

80

HEALTH PHYSICS PRACTICES IN LABORATORIES USING ³H AND ¹⁴C-LABELLED TRACERS. R.J. CLOUTIER and J.D. BENOISE, Oak Ridge Associated Universities, Oak Ridge, Tenn. 37830.

Tritium and ¹⁴C are often considered relatively safe radionuclides to handle because their permissible body burdens are orders of magnitude greater than those for most other radionuclides. This is reflected in the U.S. Atomic Energy Commission regulations that do not require a license for the use of amounts up to 1000 microcuries of ³H and 100 microcuries of ¹⁴C. Although many tracer studies are performed with much smaller quantities, poor control of even these small amounts can result in contamination, causing an internal radiation hazard or the loss of valuable experimental data. This paper discusses the methods of handling radioactive materials that minimize the risks of contamination, and it emphasizes the needs for proper storage, secondary containers, monitoring air and surfaces, and analysis of bioassay samples for measuring body burdens. The user's responsibilities for licensing, waste disposal and transportation are also included. Experience at a laboratory handling both tracer and curie amounts of ¹⁴C and ³H required for labeling and synthesizing compounds and methods of accident prevention and decontamination is described.

81

STABLE ISOTOPE TRACER METHODOLOGY: INSTRUMENTATION AND APPLICATION TO HUMAN METABOLIC STUDIES. P.D. KLEIN and PATRICIA A. SZCZEPANIK, Argonne National Laboratory, Argonne, Ill. 60439.

Although stable isotopes of hydrogen, carbon and nitrogen were the original tracers used in the study of metabolic processes their use was superseded by the beta-emitting isotopes ³H and ¹⁴C when the latter became available because of the greater ease of detection of radioactive tracers. In recent years, two avenues of research have joined in the development of new sensitive and simplified means of measuring the stable isotopes content of molecules of biological and clinical interest. The first of these is the growing awareness of metabolic disorders in the human fetus, newborn infant and young child which, if diagnosed, can be successfully treated. Such diagnoses and the antecedent research require the use of nonradioactive tracers. The second avenue of research has been the development of the gas chromatograph-mass spectrometer which permits the facile introduction of biological compounds into a mass spectrometer, and whose development was followed by the accelerating voltage alternator (AVA) of Svedey et al. that permits comparison of two ion intensities. The Argonne gas chromatograph-mass spectrometer-integrating voltage alternator system has been designed to carry out analyses of ³H-, ¹⁴C-, and ³H-labeled metabolites of clinical interest. Features of its design include stable and reproducible focusing, precision attenuation of the more abundant mass, and direct conversion of ion intensities to digital values. In the first year of operation, it has been used to measure bile acid kinetics in human studies of infants, young children and siblings of patients with gall stones. It has been applied

to the analysis of labeled barbiturates and to kinetic studies of deuterium-labeled Krebs cycle intermediates. The operating experience and practical applications of this instrument system indicate that stable isotopic tracers are not only feasible but offer several substantial advantages over radioactive tracers in human metabolic studies.

82

THE MEASUREMENT OF LIPOID TURNOVER IN MEMBRANES BY A DOUBLE ISOTOPE TECHNIQUE. TEN-CHING LEE and FRED SNYDER, Oak Ridge Associated Universities, Oak Ridge, Tenn. 37830.

The double-labeling procedure that was originally devised by Arias, Doyte and Schimke (J. Biol. Chem. 244:3803, 1969) was used to examine relative rates of degradation of the lipid constituents of the endoplasmic reticulum in rat liver. With this technique, it is possible to determine a radioactivity decay curve of a biological component in the same animal by administering the ¹⁴C and ³H form of a single precursor at two different times. If ³H radioactivity represents the initial time point and ¹⁴C radioactivity represents the decay time point, the component with the most rapid rate of turnover will have the highest ratio of ³H/¹⁴C. The principle, assumptions and limitations of this method will be discussed. Using the double-labeling procedure, we found that the relative rates of degradation for phospholipid classes in the rough microsomes are the same as those in the smooth microsomes. However, the different phospholipid species are degraded differently within a given submicrosomal fraction. Furthermore, acyl moieties of phosphatidylcholine and phosphatidylethanolamine esterified at the 1 position turn over faster than those esterified at the 2 position. The results obtained are consistent with the concept that the endoplasmic reticulum is a mosaic array of structural lipoproteins and enzymic proteins in a dynamic state of heterogeneous turnover.

83

PREPARATION, ANALYSIS AND RADIOLYSIS OF LABELLED COMPOUNDS. L.E. GELLER, New England Nuclear Corporation, Boston, Mass. 02118.

The historical developments in the field of radiochemicals will be discussed briefly. Chemical and biosynthetic methods for the preparation of both carbon-14 as well as tritium will be presented. These methods will include tritium exchange labeling and the advantages and disadvantages of generally labeled compounds to investigators in the field. Illustrative examples for the analysis of labeled compounds will include autoradiography, gas chromatography with mass and activity detectors and reverse isotope dilution analysis. The discussion of synthesis and analysis leads into the topic of storage and decomposition. Since investigators utilize their supply of radiochemicals over a period of time it is important to understand the factors involved in radiochemical decomposition and the methods available for maximizing shelf life.

84

PRINCIPLES IN THE SYNTHESIS OF ¹⁴C-LABELLED LIPIDS. CLAUDE PLANTADOSS, University of North Carolina, Chapel Hill, N.C. 27514.

A description will be given of procedures and techniques for the chemical synthesis of the following labeled compounds: DL-α-1-¹⁴C-batylyl alcohol, DL-β-1-¹⁴C-batylyl alcohol, DL-α-1-¹⁴C-chimyl alcohol, DL-β-1-¹⁴C-chimyl alcohol, L-α-1-¹⁴C-chimyl alcohol, DL-α-1-¹⁴C-hexadecyl 2,2-dimethoxy-3-hydroxypropane and its keto analog. All the products were satisfactorily prepared in good yields and in high specific activity. The trifluoroacetic anhydride method of esterification was used to prepare a number of labeled esters of glyceryl ethers.

85

TRITIUM-LABELLING OF LIPIDS. T.L. MOUNTS, Northern Regional Research Laboratory, Peoria, Ill. 61604.

Elucidation of metabolic pathways, chemical reactions and biosyntheses has been greatly enhanced by the use of radioisotopes. Because of its short half-life and low radiation energy, tritium has become an important tool in lipid research. Lipids have been labeled with tritium by a variety

of techniques including chemical synthesis, catalytic reductions, exchange reactions and radiation-induced reactions. These techniques are described from both a historical and current aspect. Specific examples of labeling procedures are cited, as well as final purity of product evaluation. Current developments in final purity analysis instrumentation and procedures are also presented. The latest application of tritium at this laboratory is as a tracer in biological investigations and catalytic hydrogenation of lipids.

86

USE OF LABELED COMPOUNDS TO STUDY THE MECHANISM OF FLAVOR FORMATION IN OXIDIZING FAT. S.T. MICHALSKI and E.G. HAMMOND, Iowa State University, Ames, Iowa 50010.

A number of scission products, some of which are potent flavor compounds, have been identified in oxidized fats and oils. Studies on the mechanism of formation of these compounds is hampered by the low yields in which they are produced and the lack of suitable methods for quantitative analysis. For these reasons current suggestions about mechanism are based primarily on speculation or experiments carried out under extreme conditions of temperature or concentration. Compounds labeled with carbon-14 offer approaches to the study of some of these mechanisms. One mechanism that frequently has been proposed is that hydroperoxides decompose to give primary scission products that in turn may undergo further oxidation and degradation. To test this, hexanal-2-O¹⁴, 1-octene-3-one-4-C¹⁴ and 1-octene-3-ol-4-C¹⁴ were synthesized and added to soybean oil at concentrations of about 1 ppm. The soybean oil was allowed to oxidize at temperatures ranging from 25 to 60 C, and the fate of the labeled compounds was followed by molecular distillation and gas chromatography of the distillate by formation and thin layer chromatography of derivatives. The results indicate that hexanal is converted to hexanoic acid in low yields and that 1-octene-3-ol is converted to 1-octene-3-one. No degradation to shorter chain compounds was evident under the conditions we used.

87

VARIOUS INTERACTIONS IN CHOCOLATE FLAVOR. P.G. KERRY, Pennsylvania State University, University Park, Pa. 16802.

The published list of volatiles identified in cocoa beans and products derived from them includes more than 300 compounds. Recent attention has centered on the aroma compounds generated by roasting since it is during this process that the typical aroma of chocolate is developed. While changes involving cocoa lipids undoubtedly contribute to flavor, the origins of a large number of key aroma compounds can be traced to the sugars and amino acids in cocoa beans and their reaction products. Cocoa fat is especially important in respect to the perception of chocolate flavor. The distribution of volatiles between the continuous lipid phase and polar components and adsorptive surface phenomena are important considerations. The remarkable oxidative stability of cocoa butter is worthy of note in any discussion of flavor in chocolate products.

88

OXIDATION OF FISH OILS. W.W. NAWAR and A.C. NOBLE, University of Massachusetts, Amherst, Mass. 01002.

Fish oils are unique in that they contain characteristic long chain highly unsaturated fatty acids. Detailed information regarding the autoxidation of nonconjugated fatty acids containing more than three double bonds is scarce. For our study we selected methyl docosahexanoate as typical of fish oil esters. After autoxidation products were separated by gas chromatography and identified by mass spectrometry, IR and GC retention characteristics. The compounds identified include aldehyde esters, keto esters, hydrocarbons, shorter chain methyl esters, polyunsaturated aldehydes, saturated and unsaturated furans, and a number of cyclic esters. Mechanisms are proposed to account for the formation of most of the compounds identified.

89

THE CORRELATION OF FLAVOR SCORES TO CHEMICAL

TESTS FOR A SERIES OF SOYBEAN OILS OF DIFFERENT OXIDATION HISTORIES. T.H. SKOUSE, J.E. COVAT and J.K. MANES, W.L. Clayton Research Center, Richardson, Tex. 75080.

Many objective tests have been proposed and utilized to predict the oxidative or flavor stability, or both, of fats and oils. Some are believed to give an indication of the oxidative history of an oil before refining, others have been shown to follow high temperature oxidation that occurs during frying, and some give an indication of the volatile decomposition products that accumulate during hydroperoxide dismutation. The object of this study was to produce soybean oil samples having a range of slight to moderate autoxidation histories. Then the accumulated peroxides in all samples were determined and the volatile flavor components were removed by a high vacuum counter-current steam deodorization. By aging all samples under identical conditions for various lengths of time, the flavor score spread was obtained and regression analyses were performed on these flavor scores to seven commonly used chemical tests. Many of the tests investigated are utilized in the fat and oil industry for buying and selling specifications.

90

FLAVOR LOSS STUDIES ON HEAT-PROCESSED FOODS. J.A. ROGERS and R.J. EISELE, Fritzsche Bros. Inc., New York, N.Y. 10011.

Essential oils and oleoresins derived from spices and herbs, by steam distillation in the former case or by solvent extraction in the latter case, are increasingly more important as flavoring constituents in heat-processed foods. Steam-distilled oils, when used for such purposes, tend to steam distill during the heat processing due to moisture which is present. Oleoresins which contain the essential oil and natural fixatives tend to depress such volatization and are preferred as flavoring materials in such cases. Subjective flavor panel evaluations will be reported showing the significance between oleoresins and essential oils as well as the corresponding spice or herb. Instrumental evaluation including gas chromatography and thin layer chromatography will compare essential oils produced by direct steam distillation vs. those isolated from oleoresins. The difference in composition which can be related to the improved flavor effect from oleoresins will be discussed.

91

ROOM ODOR EVALUATION OF OILS AND COOKING FATS. C.D. EVANS, KATHLEEN WARNER, G.R. LIST and J.C. GOWAN, Northern Regional Research Laboratory, Peoria, Ill. 61604.

Panel evaluations have been made of room odors developed by edible oils and cooking fats heated to frying temperatures. All vegetable and mixed fat shortenings and oils of different iodine value and from special processing were investigated with and without added stabilizers. When silicones were added to frying fats, room odor scores markedly improved. Addition of antioxidants had little if any effect. Certain added antioxidant cleavage products had little effect on odor scores at levels where they were easily detected in taste tests. To be discernible in room odors, these additives had to be present at levels approximately 100-fold greater than their taste thresholds. Panel results show that the undesirable frying odors contributed by unhydrogenated soybean oil to mixtures with other oils could be detected at levels above 20%. As the level of soybean oil was lowered further, the room odor scores of oil mixtures improved perceptibly. Soybean oil hydrogenated with different catalysts to various iodine levels gave better odor scores as the level of linolenic acid decreased.

92

CATALYTIC DECOMPOSITION OF AUTOXIDIZED UNSATURATED FATTY ACIDS. W.I. KIMOTO and A.M. GADIRI, Eastern Marketing and Nutrition Research Division, ARS, USDA, Beltsville, Md. 20705.

The monocarbonyl products (alkanals, 2-enals, and 2,4-dienals), which were formed from autoxidized triolein, methyl linolenate and methyl arachidonate in presence of acid-washed Fuller's earth or cupric stearate at 85 C, from ferrous chloride or hemin, in 70% tertiary butanol at room temperature, or

on heating at 165 to 200 C, were determined. In the presence of Fuller's earth, no 2-enals were obtained from triolein, and of the total monocarbonyls, only 4% 2,4-heptadienal from methyl linolenate and 6% 2,4-decadienal from methyl arachidonate were formed. Treatment with metal catalysts or heating produced a substantial increase in the proportions of unsaturated monocarbonyl products, as indicated by the formation of 67-86% 2-enals from triolein, and 50-54% 2,4-heptadienal and 23-32% 2,4-decadienal from methyl linolenate and methyl arachidonate, respectively. The data suggest a selective hydroperoxide scission mechanism and support previous observations with autoxidized linoleate.

93

COMPARATIVE ASPECTS OF STEROLS OF MARINE INVERTEBRATES. D.R. IDLER, L. SAFF and P. WISEMAN, Fisheries Research Board of Canada, Halifax, N.S., Canada.

Marine invertebrates frequently contain complex mixtures of sterols varying in length of side chain and in site of nuclear or side chain (or both) unsaturation. This is particularly true of the more primitive species. The evolutionary higher organisms tend toward a single sterol cholesterol. With recent advances in gas liquid chromatography (GLC) and mass spectrometry, sterols of representative phyla have been re-examined; these studies have clarified the status of several sterols and sterol mixtures and led to the identification of a new marine sterol. The occurrence of cholesterol as the major sterol in several Porifera (sponges) is unique. Sponges also contain sterols not yet found in other invertebrate phyla, e.g., neospongsterol (22-dehydrocampesterol) and chondrilesterol. Considerable variability has been observed in sterol content. Centaurea, corals, jellyfish, sea-anemones) Gupta and Scherer (1969) examined three zoanthids and found that spongasterol was the principal sterol. *Lathus confertus*, 22,25-dihydrobrassicasterol of *Phyllozoo tuberculosa*, and *Polythoa sp.* contained a single sterol, 24-methylcholesterol. Bergmann first reported the presence of a unique Δ^5 -sterol, fergosterol, in a gorgonian (coral). Its structure has recently been established as (22R, 23R, 24R)-22,23-methylene-23,24-dimethylcholesterol (Ling *et al.*, 1970). Echinoderms (e.g., star fish, sea urchin) sterols are also complex; asteroids (e.g., sea star) and holothurians (e.g., sea cucumber) contain predominantly Δ^5 -sterols while echinoids, opuloids and crinoids contain Δ^6 -sterols. Chitons (class Amphineura) are unique among the molluscs in that Δ^5 -cholesterol is the principal sterol. Pelecypods (bivalves, e.g., oyster, clam, scallop) and gastropods (snails) contain complex mixtures of as many as 15 sterols in a single species, with gastropods having a greater percentage of cholesterol. Sterols of the scallop, *Placopecten magallanicus* have been separated by preparative GLC and identified by mass spectrometry. This has resulted in the identification of two new marine sterols, a Δ^5 -sterol, 22-trans-24-norcholesta-5,22-dien-3 β -ol (Idler *et al.*, 1970) and 22-cis-cholesta-5,22-dien-3 β -ol (Idler and Wiseman, 1971). Cholesterol is the principal sterol of all species of Crustacea (e.g., crab, lobster, snail) examined; in several species it is the only sterol. When present, deamosterol is the second major sterol. Several minor sterols have been detected by GLC.

94

SOME ASPECTS OF PHOSPHOLIPID METABOLISM DURING DEVELOPMENT OF THE BRINE SHRIMP *ARTEMIA SALINA*. R.D. EWING and F.J. FINAMORE, Oak Ridge National Laboratory, Oak Ridge, Tenn. 37830.

Microsomal preparations from larvae of *Artemia salina* contain an enzyme that rapidly converts CMP into CDP-choline. We conclude this enzyme is CDP-choline: 1,2-diglyceride choline-phosphotransferase (EC 2.7.8.2) and that the steady state of the reaction at low concentrations of CMP favors formation of CDP-choline. The enzymatic activity is completely dependent upon the addition of CMP and metal ions; no other cytidine compounds can substitute for CMP in the reaction. Furthermore, a stoichiometric relationship exists between the amount of phosphorus lost from phosphatidyl choline and the amount appearing in CDP-choline indicating phosphatidyl choline is the source of phosphocholine in formation of CDP-choline. The same microsomal preparations synthesize phosphatidyl choline using CDP-(¹⁴C)-choline as substrate, and incorporation of the choline moiety from CDP-choline into

phosphatidyl choline is stimulated by the addition of solubilized 1,2-diglycerides derived from soybean lecithin. A comparison of both enzymatic activities in the microsomal preparation under optimal conditions for each, reveals that the rate of synthesis of CDP-choline from phosphatidyl choline is about twice the rate of synthesis of phosphatidyl choline from CDP-choline.

95

STUDIES ON MARINE ORGANISMS: PROBING STRUCTURAL FEATURES OF BIOLOGICAL MEMBRANES WITH NITROXIDES. W.T. ROUBAL, Pioneer Research Laboratory, Seattle, Wash. 98102.

Dilute solutions of alkyl-substituted nitroxide compounds are stable, relatively inert, and show sharp, well-resolved and simple paramagnetic resonance spectra. When nitroxides are attached to biomolecules, however, simple spectra are no longer recorded; instead, due to the sensitivity of nitroxides to their environment, spectra undergo marked changes. Nitroxides, therefore, are novel substances for use as probes for elucidating structural features of biomolecules. Maleimide, iodacetamide, carboxyl, and isocyanate-containing nitroxides are very useful for investigating protein and enzyme structures. While nitroxide-labeled lipids and application in studies of lipid-protein interaction and in studies of membranes. Basic underlying theory together with studies devised for investigating structure, lipid-protein interaction, and association of exogenous substances with cellular constituents in fish and other organisms is discussed.

96

HETEROGENEITY OF LIPID COMPOSITION WITHIN THE CEPHALIC MELON TISSUE OF THE PILOT WHALE. GEORGE WEDMID, CARTER LITCHEFIELD, Rutgers University, New Brunswick, N.J. 08903, R.G. ACKMAN and J.C. SIPOS.

Dolphins, porpoises and toothed whales possess a large, fat rich body called a "melon" located in the forehead directly in front of the skull. This melon is thought to be an acoustical transducer for transmitting ultrasonic pulses into the surrounding water for the purpose of echolocation. The melon is usually spoken of as a homogeneous organ; however, dissection reveals that it is not morphologically distinct but gradually blends into the surrounding tissue. To better understand the acoustical function of the melon, we have examined the positional variations in lipid composition within the large melon of the pilot whale (*Globicephala melaleuca*). A 10 mm thick vertical slice was cut from the center of the frozen head of an adult male pilot whale. The slice was then sampled at 18 points based on a 50 X 50 mm grid pattern. The lipids were extracted from each sample and examined for lipid class, molecular species, fatty acid and fatty alcohol composition using thin layer and gas liquid chromatography. Results show that wax esters and triglycerides of both iso-valeric and long chain acids are present in all samples but in varying proportions. Saturated isovaleryl wax esters and diisovaleryl triglycerides predominate in the center and bottom of the melon near the upper jawbone. Concentrations of unsaturated wax esters and triglycerides containing only long chain acids are greatest at the front, top and back of the melon where it merges into other tissues. Between these two extremes there is a gradual change of composition. Hence the acoustical function of the pilot whale melon must be reconciled with a gradient rather than a homogeneous composition.

97

CHOLESTEROL SYNTHESIS AND HYPERBARIO OXYGEN IN SWIMBLADDERS OF ABYSSAL AND SHALLOW FISHES. C.F. PHILGORE, Scripps Institute of Oceanography, La Jolla, Calif. 92037.

A massive cholesterol accumulation has been discovered in oxygen-filled swimbladders of a number of abyssal benthopelagic fishes. These fish include three species of *Coryphaenoides* (a Macrourid), *Acanthopagrus* (a Gadid) and *Bassogadus digitatus* (a Brodid). This nonphysiological accumulation of free cholesterol comprises up to 49% of lipid in a fatty foam deposit (2-5% body volume) located in the swimbladder interior. Cholesterol is synthesized in the swimbladder gas gland of *Coryphaenoides* and various shallow fishes, from acetate-1-¹⁴C. Cholesterol synthesis rates were greater in the

gas gland than in the liver of all fish tested. It is facilitated by pressure (5,000 and 10,000 psi) in contrast to total lipid synthesis, which is retarded by pressure. A search has been made for the site of the pressure effect on cholesterol synthesis. One approach has been to administer tritiated squalene-2,3-oxide to excised tissues of these fishes, and study its cyclization to lanosterol as a function of pressure. Squalene oxidation to squalene-2,3-oxide has also been studied at different oxygen tensions, to look for a possible oxygen effect.

98

EFFECT OF DIETARY LINOLENIC ACID AND DOCOSENAIC ACID ON GROWTH AND FATTY ACID COMPOSITION OF RAINBOW TROUT (*Salmo gairdneri*). T.C. YU and R.O. SINNHUBER, Oregon State University, Corvallis, Ore. 97331.

Pure methyl linolenate (18:3 ω 3) and docosahexaenoate (22:6 ω 3) were incorporated in semipurified diets at several levels and fed to trout previously maintained on a fat free diet. After 14 weeks the weight gain and feed conversion of the fish on each diet were calculated. The fatty acid composition of the lipid from each group of fish was analyzed by gas liquid chromatography. The results showed that both 18:3 ω 3 and 22:6 ω 3 fed at the 1% level supported maximum growth of the fish. A decrease in growth rate was observed in fish on diets containing 0.5% of either 18:3 ω 3 or 22:6 ω 3. The control group fed a fat free diet exhibited a shock syndrome, poor appetite and a very slow growth rate. The fatty acid analysis revealed that a high percentage of eicosatetraenoic acid (20:3 ω 3) accumulated in the phospholipid fraction of the lipid of the fat free diet fish. The 20:3 ω 3 content was lowered when either 18:3 ω 3 or 22:6 ω 3 were included in the diet. In addition, the analysis showed that the dietary 18:3 ω 3 was rapidly converted by the fish into 22:6 ω 3. A high concentration of 22:6 ω 3 was found in the phospholipid. However, 22:6 ω 3 fed to the fish remained unchanged. No retroconversion of this fatty acid was observed.

99

THE ROLE OF FATS AND OILS IN PHARMACEUTICALS. J.B. JAKOMEK, American Medical Association, Chicago, Ill. 60610.

The role of fats and oils in pharmaceuticals continues to change. One measure of this change is given by an examination of various national compendia which highlight changes within our own country over the years. These changes will be compared with those taking place elsewhere. This paper will also examine some factors which influence the current supply of various pharmaceutical fats and oils as well as the factors of demand presently operating in this area. Attention will be given to the categories of pharmaceutical products utilizing fats and oils and conservative projections of future use, where feasible, will be attempted.

100

THE ROLE OF FATS AND OILS IN COSMETICS. MURRAY BERRIOR, Cheesbrough-Pond's Inc., Clinton, Conn. 06419.

The earliest emollients in the history of cosmetics were the naturally occurring animal fats and vegetable oils. These provided soothing and smoothing action on the skin, and grooming effects on head and beard hair. For the most part, other problems limited the interest in oils derived from fish. With the increasing sophistication of these materials, the shortcomings of natural fats and oils were overcome in several ways: increased stability through use of antioxidants, reduced odor through improved processing, improved stability through chemical modification, and substituted diversity through preparation of derivatives, and substitution of mineral oil. Today, the most important single cosmetic use of an unmodified natural fat or oil is that of castor oil as the base for lipsticks.

101

PHARMACOLOGY OF FATS AND OILS. STEVEN CARSON, Food and Drug Research Laboratories, Inc., Maspeth, N.Y. 11378.

To a greater extent than is represented by other classes of chemical materials, fats and fatty acids may produce

biological effects by a number of diverse actions. Their unique properties, either in the unsubstituted form or as substituted entities, may yield biological actions either as a direct pharmacological consequence or the resultant of unusual surface properties affecting penetration or availability by potent agents. The relationship of direct and passively resultant effects on the animal organism by several routes of administration will be discussed in terms of their selection for a variety of technological applications.

102

ANALYSIS OF OIL-BASED PHARMACEUTICALS. EDWARD SMITH, Bureau of Drugs, FDA, Washington, D.C. 20204.

Oils of both mineral and vegetable origin are used in the formulation of pharmaceuticals for their solvent properties and because of their effect on drug release. In most cases the analysis of these pharmaceuticals requires the separation of the drug substance from the oil components prior to the quantitation of the drug substance. When the physical properties of the drug substance are quite similar to those of components of the oil, the separation of the drug becomes involved. Column partition chromatography provides an excellent means of separating steroids from the vegetable oil vehicles used in injectables. Both reverse phase and direct phase techniques are applied to the separation of the steroid esters from the glyceride, sterol and triterpenoid fractions of the oil as well as from any unsaturated sterol. Illustrations will be provided to demonstrate the application of thin layer chromatography, paper chromatography and gas chromatography to the analysis of oil-based pharmaceuticals. The introduction of new surfactants and other adjuncts to topical preparations poses a complex problem. Analytical methodology which was adequate for older formulations is not suitable for the newer formulations. Examples of recently developed analytical methods will be discussed.

103

EMOLLIENT OILS. H.D. HAMILTON, Drew-A Division of Pacific Vegetable Oil Corporation, Bountown, N.J. 07005.

Emollients are substances employed in maintenance of smoothness, softness and pliability of skin surfaces. They offer protection against dryness and irritation by exerting a barrier effect to excessive moisture escape from the dermis. Emollients may have characteristic surface activity of themselves or be used in conjunction with surfactants which enhance their penetrating and barrier effects. Emollients have fat-like properties, and in fact are frequently based upon fats and oils or fatty acid derivatives. The emphasis in this paper will be on such emollients. A review will be presented of lipid substances that have been employed for emollient effect. In vivo and in vitro evaluation methods will be discussed. Practical utilization of emollient oils in cosmetic formulations will be illustrated.

104

REVIEW OF GAS CHROMATOGRAPHIC APPLICATIONS IN THE FAT SOLUBLE VITAMIN FIELD. A.J. SHEPPARD, A.R. PROSSER and W.D. HUBBARD, Division of Nutrition, FDA, Washington, D.C. 20204.

The application of gas chromatography as an analytical tool for the determination of the fat soluble vitamins has yet to be utilized to its full potential. The methods for menadione and vitamin E are well developed for the measurements of these vitamins in pharmaceutical products. Suitable internal standards have now been identified for menadione and vitamin E. The vitamin D content of pharmaceutical products has been measured by gas chromatography; however, an isomerization reaction is necessary to avoid hydrolysis and the sample cleanup procedures need to be improved. The gas chromatographic analysis of vitamin A in general has not proven to be a very satisfactory method. Considerably more research and developmental work is required before a routine, satisfactory gas chromatographic analysis for vitamin A can be achieved for even the relatively uncomplicated pharmaceutical products. Some workers have reported satisfactory determinations of the carotenes by gas chromatography. Vitamin K₁ analysis by gas chromatography has not progressed to any satisfactory extent. The application of gas chromatography to the analysis

of the vitamin content of biological materials has not developed to any large extent, although some gas chromatographic techniques have been used successfully for the determination of blood and liver levels of vitamin E. The problems of sample preparation, sample purification and sample concentration are today the major hurdles to be overcome before any really widespread application of gas chromatographic analysis of the fat soluble vitamins in biological materials can be realized.

105

APPLICATION OF THE FOOD ADDITIVE AMENDMENT TO FAT- AND OIL-BEARING FOODS. A.T. SPIKER, JR., Division of Regulations and Petitions Control, FDA, Washington, D.C. 20204.

Abstract not available at press time.

106

GAS CHROMATOGRAPHIC DETERMINATION OF ANTI-OXIDANTS IN FATS AND OILS. E.E. OJA, Food and Drug Directorate.

A procedure is described which is suitable for rapid simultaneous analysis of butylated hydroxytoluene, the two isomers of butylated hydroxyanisole, mono-tertiary butylhydroquinone, propyl gallate and nordihydroguaiaretic acid employing gas chromatography for quantitation and thin layer chromatography (TLC) for confirmation. The TLC system is the two-dimensional system described by Sahasrabudhe, and the procedure for extraction of antioxidants from fats, and oils is modification of his method. For quantitation, trimethylsilyl derivatives of the antioxidants are formed and relative responses compared to that of methyl pentadecanoate. Sample chromatograms and recovery data are given. Some recent developments in the analysis of antioxidants are reviewed briefly.

107

FOOD ADDITIVE REGULATIONS: INDUSTRY VIEW-POINT ON CURRENT PROBLEMS. KENNETH E. MUIRHEAD, Atlas Chemical Industries, Inc., Wilmington, Del. 19899.

The advancement of science frequently results in previously unknown discoveries. Also, scientific work developing new facts is sometimes reported along with completely unwarranted conclusions as to the implications of the new facts. Where such discoveries automatically make a food additive illegal under the law regardless of its safety, or where unwarranted conclusions relate adversely to the safety or nutritional value of foods or food additives, the resulting publicity makes the reasonable and rational application of our already difficult food law almost impossible.

108

METHODOLOGY FOR EMULSIFIERS. F.J. BAUER, Procter and Gamble Company, Cincinnati, Ohio 45224.

From the standpoint of amount consumed and rate of production increase, emulsifiers constitute the most important class of food additives used in this country. The presently used emulsifiers are covered in terms of quality control, determination in foods, analytical techniques used and official methodology.

109

NUTRITIONAL QUALITY IN FORMULATED FOODS. O.C. JOHNSON, Division of Nutrition, FDA, Washington, D.C. 20204.

Consumer interest in the nutritional quality of foods has increased in recent years. The Food and Drug Administration has initiated a number of activities which are intended to help assure the consumer that the foods which make up the American diet are nutritious. This applies to both new and traditional foods. A major activity is the development and evaluation of labeling which describes the nutritional qualities of food products. This includes labeling that indicates the nutrient content, as well as that which reports on specific properties such as fatty acid composition. Nutrient labeling which calls attention to positive nutritional aspects of foods will also increase interest in the addition of nutrients to foods. There will be a need to determine the availability of added nutrients, particularly in the more complex food mixtures.

The improvement of the protein quality of cereal grain and bean foods through the addition of amino acids is another area of concern. The safety and effectiveness of added amino acids must be determined before such fortification is put into general usage.

110

METHOD FOR DETECTING CHEMICALLY-INDUCED MUTATIONS IN FOOD ADDITIVES. M.S. LEGATOR, Bureau of Foods, FDA, Washington, D.C. 20204.

From the initial discovery of chemically-induced mutations 30 years ago, up to very recent times, geneticists have utilized these agents as tools to describe the genetics of their systems (i.e., microorganism and Drosophila). Although the public health hazard aspect of chemically-induced mutations was appreciated by many toxicologists and geneticists, the lack of relevant methods in mammals precluded the screening of drugs, food additives and pesticides for mutagenicity. Within the last few years, practical procedures in mammals have been developed that can be used to screen a variety of chemicals for mutagenic activity. Three procedures run currently should detect the majority of mutagenic agents. These three methods include: *in vivo* cytogenetics, in which a number of different experimental animals can be used and both germinal and somatic cells can be evaluated for chromosome abnormalities; dominant lethal test, in which male animals are treated with the compound and sequentially mated with females; pregnancy is interrupted at mid-term and the uterus is examined for preimplantation loss and early deaths (This is a genetic test that is incompatible with the survival of the conceptus and the time at which the effect occurred during spermatogenesis can be detected); the host-mediated assay, in which an indirect indicator such as a microorganism is employed in a host to detect point mutations after the host has had an opportunity to either detoxify or potentiate the compound. Although a small number of chemicals have been evaluated by these methods, several examples are available of commonly-used agents that were mutagenic in one or more of the three procedures. In addition to detecting active mutagenic agents, the correlation between carcinogenic and mutagenic agents has become increasingly more evident with the use of these procedures.

111

A COMPARISON OF ELECTRON IMPACT AND CHEMICAL IONIZATION MASS SPECTROMETRY IN COMPOUNDS OF BIOLOGICAL INTEREST. H.M. FALMS and G.W.A. MILNE, National Heart and Lung Institute, Bethesda, Md. 20014.

Over the years, electron ionization (electron impact) spectra have been used most successfully to determine structures of a wide variety of organic compounds. Success of the method has been largely due to the extensive studies on different compound types carried out by chemists and spectroscopists. Nevertheless, extensive rearrangements often occur with scrambling both of hydrogen and carbon and even today new ones are encountered (trimethylsilyl migrants, etc.). In chemical ionization, discovered by F.H. Field and D. Munson at Esso, ions are formed by collisions with other reagent ions. In this case, the energy transferred can be more easily predicted at a high and low energy ions may be used, i.e., H⁺ and CH₃⁺, respectively. Under these conditions intense ions are found at (M+H)⁺ and the fragmentation processes strongly resemble those found in acid-catalyzed solution chemistry, most ions being even electron ions. On the other hand, if charge exchange processes are studied by utilizing gases such as N₂ or He⁺, the spectra involve odd electron ions and resemble ordinary electron ionization spectra. Several examples from the field of natural products will be shown illustrating the former type of CI spectra.

112

APPLICATIONS OF A GAS CHROMATOGRAPH-MASS SPECTROMETER-COMPUTER ANALYTICAL SYSTEM IN STEROID STUDIES. E.O. HOBNING and M.G. HOEMING, Baylor College of Medicine, Houston, Tex. 77025.

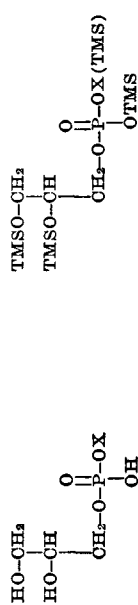
A gas chromatograph-mass spectrometer-computer analytical system equipped with a chemical ionization source was used to obtain mass spectra for derivatives of human steroids. The

carrier gases were methane and helium; the spectra obtained for steroids under these conditions were compared with the corresponding electron ionization spectra. Metabolic profile separations were also carried out with this system. Major and minor components of mixtures of biological origin were identified by direct comparison of spectra and by programmed data analysis.

113

GAS PHASE ANALYSIS OF PHOSPHOLIPIDS. CATHERINE FENSELAU and J.H. DUNOAN, Johns Hopkins University, Baltimore, Md. 21205.

Procedures have been developed which allow mixtures of phospholipids containing a variety of polar head groups to be identified by gas chromatography-mass spectrometry. Long chain acyl groups are removed by selective hydrolysis. The GPX compounds are filtered through ion exchange resin to regenerate acids from sodium salts, and the resulting neutral molecules are converted to per-trimethylsilylated derivatives.



OX = Hydroxyl
glycerol
ethanolamine
N,N-dimethylethanolamine
serine
inositol

The products of the silylation reaction can be analyzed easily using the direct probe inlet system, or, with more care, using a gas chromatographic inlet system. Derivatives comprising combinations of methyl esters and trimethylsilyl ethers have also been investigated. These offer the advantages of lower molecular weights and simpler fragmentation. Applications of the method will be discussed.

114

A DIGITAL COMPUTER SYSTEM FOR ON-LINE DATA REDUCTION WITH A COMBINED GAS CHROMATOGRAPH-MASS SPECTROMETER. C.C. SWEELEY, Michigan State University, East Lansing, Mich. 48823, B.D. RAY, W.I. WOOD, J.F. HOLLAND and M.I. KRICHVANSKY.

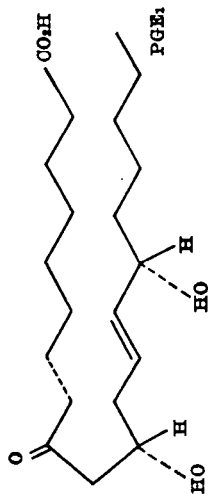
A small digital computer system has been developed for on-line data acquisition and reduction of information from a single-focusing mass spectrometer. The computer configuration includes 8K words of 12-bit length and 64K words of disk storage, two magnetic tape transports, a 12-bit analog-to-digital converter with multiplexor, and output to a teletype writer and a printer. Peak intensities and magnetic field strength values (Hall Effect probe) at the summits of peaks are determined in real time from data acquired at a sampling rate of 26 KHz. Subroutines are used subsequently for conversion of Hall Effect values to exact masses, exact masses to nominal masses, background subtraction, normalization, output to magnetic tape of finished mass spectra, and preparation of tabular data and bar graph plots. Up to 62 scans can be stored in a single run, using variable scan speeds with rates as high as 0.8 sec for a range from m/e 0 to 500. Calibration has been achieved up to m/e 860 and is normally carried out daily by an automatic routine utilizing any selected reference compound.

115

PROSTAGLANDIN ANALYSIS: SURVEY OF DERIVATIVES USED FOR IDENTIFICATION AND DETECTION BY GAS CHROMATOGRAPH-MASS SPECTROMETER-COMPUTER SYSTEM. J. TROCK, WATSON and B.J. SWEEFMAN, Vanderbilt University, Nashville, Tenn. 37203.

Intensified interest in the physiological and pharmacological roles of prostaglandins has created a need for reliable means for their analysis. Since these potent hormones occur only in trace amounts in typical biological samples, vapor-phase

methods must be considered as a means for quantification but this necessitates that functionalities in the prostaglandin molecules must be modified by suitable derivatization reactions. In the case of prostaglandins in the E series, which have the reactive β -keto system, extensive derivatization has been required involving esterification of the carboxyl group using diazomethane, followed by formation of the O-methyl oxime of the ketone and finally by acylation or silylation of the secondary hydroxyl groups. Unfortunately, the O-methyl oximes occur as pairs of enantiomers which are resolvable on gas liquid chromatography and in many cases, this complicates an already difficult separation problem. These problems, together with an assessment of the utility of other related derivatives (including deuterium analogs), will be discussed with emphasis on those which are particularly suitable for quantification by multiple ion detection with on-line data acquisition and reduction.



116
CONTINUOUS MISCELLA SOAPSTOCK ACIDULATION PROCESS. L.S. CRAVER, The De Laval Separator Co., Poughkeepsie, N.Y. 12602.

Cottongseed miscella soapstock is continuously acidulated and separated in a two-stage hermetic system into two phases: acid oil-hexane and acid water. Hexane is recovered for reuse in a stripping section. At the established processing parameters, there is a high yield of a quality acid oil.

117
RECENT DEVELOPMENTS IN THE CHILLING AND CRYSTALLIZATION OF FATS AND OILS. JOHN WALSH, Contherm Corporation, Newburyport, Mass. 01950.

A critical discussion is given of full size production equipment over extended operation on shortening, creamed and flaked, or soft margarine for container filling, on margarine printing in Morpac Benli equipment with the idea of chilling after the quiescent period for printing of a coder, timer product than is now the rule. Included in the research is cooling shortening for pies in particular, to a colder limit than is now the rule for producing cold products with good creaming qualities for frozen pastry products.

118
COMPUTER MONITORING OF A MINI-REFINERY. E.D. BITNER, J.O. ERNST and H.J. DUTTON, Northern Regional Research Laboratory, Peoria, Ill. 61604.

A laboratory scale pilot plant is under construction so that process can be studied. Unit operations planned for this refinery are continuous alkylrefining, bleaching, hydrolyzing and deodorizing. The alkylrefining step, similar to one in the Swedish Zenith process, is already in operation. Also, oil color, sodium content and free fatty acid concentration are automated and both crude and refined oils are being analyzed. These analyses will also be performed at later stages of processing on the bleached, hydrogenated and deodorized oils. Under Phase I of implementing computer control, electronic signals from the analytical transducers are sent to the computer via appropriate interfacing hardware and results are returned to the operator via teletype. Phase II will involve monitoring by the computer but with instructions to an operator, and Phase III involves "closing the loop"; that is, having the computer directly control the entire process.

Development of transducers, software and hardware such as is described here for the mini-refinery should facilitate subsequent implementation of computer control on plant scale.

119
NEW DEVELOPMENTS IN CONTINUOUS EXTRACTOR DESIGN. WOLFGANG KEHSE, Gesellschaft Fur Olmuhlenrichtungen, Hamburg, Germany.

The Carroussel extractor with stationary screen bottom with conical slits, and a rotating cell wheel above is a well known type of extractor. This type extractor has the advantage that the number of movable parts within the solvent vapor space is much less than the respective number of parts of all other known extractor types. Operational experiences show that Carroussel extractors need less maintenance and suffer less wear and tear. Using the constructive features of the Carroussel extractor an important development was made: all bearings with sliding frictions within the solvent vapor filled space were eliminated. The rotor is now without support in the center and driven by elements assembled around the circumference. Every element is driven by one hydraulic motor. In case of a mechanical breakdown, one of these driving elements can be exchanged while the extractor continues in operation. Extractors of this new type will be available in diameters from 6-45 ft and will process up to 3,000 tons of soybeans per 24 hr. The housing has to carry only its own weight and therefore it can be of a very light construction. Other units, however, have also been built, which are operating at pressure conditions of 60 psig, e.g., extractors for liquorice roots. These units also work continuously; properly prepared material is continuously fed into the pressure-space and is continuously discharged from there. The stationary slit bottom design with the conical slits can also mean the lower rotor edges against the slit bottom. By these means the miscella chambers can be separated pressurewise from the other extraction space. This makes it possible to maintain a pressure difference between the space above and the space below the material bed. The features of the extraction material often necessitate such a fine crushing that the normal percolation by gravity is no longer possible. By means of a pressure difference between the space above and below the material bed, however, i.e., either by application of overpressure in the upper space or a slight vacuum in the lower space, such materials will give a good percolation rate again. An important case of application for this specific kind of extraction is the direct extraction of rape seed without prepressing, which necessitates an extremely fine flaking that would, under normal circumstances, not allow sufficient percolation of solvent. The application of a pressure difference of over 7 psig will remove oil from rape seed below 1% residual oil content by direct extraction, and within reasonable extraction time. This may be applicable for the direct extraction of cotton and sunflower seed.

120
CONTACTING AND DISPERSION EMPLOYING KENICS MIXERS. A.R. MACDONALD, Kenics Corporation.

Abstract not available at press time.

121
NEW DEVELOPMENTS ON A CONTINUOUS HYDROGENATION PROCESS. R.A. COOMBS and R.A. ZAVADA, Blaw-Knox, Pittsburgh, Pa. 15222.

Abstract not available at press time.

122
WORLD PRODUCTION TRENDS—EDIBLE FATS AND OILS. SPANLEY MEHR, USDA, Foreign Agricultural Service, Washington, D.C. 20250.

Abstract not available at press time.

123
INFLUENCE OF WORLD OIL CONSUMPTION ON EDIBLE PROTEIN SUPPLY. F.R. SENTI, USDA, AHS, Washington, D.C. 20250.

Abstract not available at press time.

124

PROJECTION AND PROSPECTS FOR COTTONSEED. C.M. OATTEK, Texas A and M University, College Station, Tex. 77843.

Abstract not available at press time.

125

PROJECTION AND PROSPECTS FOR PEANUTS. C.T. WILSON, Virginia Polytechnic Institute and State University, Blacksburg, Va. 24061.

Abstract not available at press time.

126

PROJECTION AND PROSPECTS FOR SUNFLOWER SEED. D.E. GANDY, National Cottonseed Products Association, Memphis, Tenn. 38112.

Abstract not available at press time.

127

PROJECTION AND PROSPECTS FOR COCONUTS. W.C. LABAY, University of Rhode Island, Kingston, R.I. 02881.

Abstract not available at press time.

128

MIXTURES OF SOAP AND LIME SOAP DISPERSING AGENTS AS REPLACEMENT FOR PHOSPHATE-BUILT DETERGENTS. R.G. BISTLINE, W.R. NOBLE and J.K. WELCH, Eastern Marketing and Nutrition Research Division, ARS, USDA, Philadelphia, Pa. 19118.

To be useful as a replacement for phosphate-built detergents, soap must be formulated to prevent lime soap curd formation in wash and rinse cycles and to maintain high detergent efficiency at low concentration levels. Lime soap dispersing agents have been found to maintain the dispersal of lime soap curds through both wash and rinse cycles. Washing tests were carried out on standard soiled cloth in 300 ppm hard water with mixtures of soap and a number of lime soap dispersants. Esters of α -sulfonylated tallow fatty acids, 2-sulfated oxyalkylated tallow alcohols, sulfated alkanolamides, 2-sulfoethyl esters of tallow fatty acids and N-methyl, N(2-sulfoethyl)tallowamide gave good washing results when combined with soap at the 5% level and showed further improvement as the level of additive was increased to 20%. Small amounts of sodium oxyacetate, sodium citrate, sodium silicate or sodium tripolyphosphate increased the detergency of combinations of soap and lime soap dispersing agents. A study of ternary diagrams obtained from single washings of a standard soiled cotton indicated that optimum detergency was usually obtained when the mixture contained 80% soap, 10% lime soap dispersant and 10% builder. Single washing tests with standard soiled swatches failed to demonstrate the known superiority of phosphate-built syndet systems over other inorganic systems. Since this superiority was shown by the six cycle multiple soil-wash procedure of Schwartz, all important formulations were checked with the latter method.

129

CHEMICAL AND TOXICOLOGICAL STUDIES ON γ -ALKYL HYDROXAMIC ACIDS. R.F. ADDISON and G.L. FLETCHER, Bedford Institute, Dartmouth, N.S., Canada.

Medium chain γ -alkyl hydroxamic acids (R-CO-NHOH; R = n-C₆-n-C₆) are potentially useful as flotation agents. Since after their application they may enter lake or river systems, their toxicity to fish has been examined. A commercial hydroxamate mixture, prepared from a low boiling coconut oil ester fraction (3-C₆-n-C₆) and used as its dimethyl ammonium soap, proved toxic to trout at the 5 ppm level. Summated industrial treatment (air blowing at pH 9) did not reduce its toxicity. Experiments with synthetic hydroxamic acid analogues showed that toxicity was not attributable to the dimethyl ammonium moiety, was probably not due to liberation of hydroxylamine (itself toxic) through hydrolysis, but was probably a feature of the hydroxamate structure. Variation in toxicity with chain length in R was observed. The chemical, physical and toxicological features of hydroxamic acids are discussed.

130
PROBLEMS IN THE DETERGENT INDUSTRY AND THEIR TECHNICAL SOLUTIONS. A. DAVIDSOHN, via Varese 13, CH-6600 Locarno, Switzerland.

This paper deals with problems arising from the necessity to formulate and process detergent products which, on the one hand, have maximum detergent efficiency and, on the other hand, correspond to the demands of the "environment." Biodegradable detergent raw materials such as linear alkylates, and especially fatty alcohols (natural and synthetic), ethoxylated fatty alcohols, are making it necessary to modify sulfonation processes, and to develop special sulfonation raw materials are giving rise to problems in the diverse systems to produce syndets in powder form either by spray-drying, spray-mixing, dry neutralization, and in combination processes of these diverse systems. The need to produce ternary mixtures of, e.g., nonionics, anionic sulfonation products, and soap, causes special difficulties in spray-drying operations by stickiness of product, buildup on the walls of the spray tower, clogging of cyclones etc. The tendency to employ high percentages of nonionics in detergent powders, especially more biodegradable types of nonionics, which are generally more heat sensitive, increase these difficulties. In this paper, methods are proposed to overcome these difficulties, and special methods are described to regulate the density of powder products by special combination processes, which combine atomization with a special spray-mixing process, which has the advantage of incorporating high percentages of practically all types of nonionics (especially heat-sensitive biodegradable types, such as alcohol-ethoxylates, fatty acid alkylolamides, etc.). The process is adaptable for dry neutralization of diverse sulfonates as well, alone or in combination with fatty acids, which are transformed into soap during the process. The process makes it possible for the detergent manufacturer to formulate his detergent powder with maximum detergent efficiency and maximum biodegradability without encountering technical problems in the production process.

131
REVERSE PHASE THIN LAYER CHROMATOGRAPHIC SEPARATION OF OXYETHYLATED ALKYL SULFATE SURFACTANTS. MARSHA MILLIE and A.C. BREYER, Beaver College, Glenside, Pa. 19038.

A series of oxyethylated alkyl sulfate surfactants were separated by reverse phase thin layer chromatography. The separations were designed on the basis of a systematic study of the variation of the R_f values of the anionic sulfate derivatives with sample size, nature of the absorbent solvent composition, and the nature of the reverse phase. The best separation was obtained with inactivated Brinkman slumping H plates impregnated with 3% dodecanol-ethanol (v/v) at 27°C using a methanol-ammonium hydroxide (6:4 v/v) solvent system.

132
DEPENDENCE OF ULTRACENTRIFUGAL STABILITY OF OIL-IN-WATER EMULSIONS ON EMULSIFIER CONCENTRATION. R.D. VOID and K.L. MITTAL, University of Southern California, Los Angeles, Calif. 90007.

The rate of loss of oil from 50% Nujol-50% water emulsions stabilized with varying concentrations of Tween 20 or Triton X-100 was determined by ultracentrifugation at 39,460 rpm. Similar measurements were made on 50% olive oil-50% water emulsions stabilized with sodium dodecyl sulfate (SDS). In all cases, the stability increased with increasing initial concentration of the emulsifier similar to the behavior of the previously studied Nujol-water-SDS emulsions below the critical micelle concentration of SDA. However, in contrast to the behavior of Nujol-water-SDS emulsions, in the present systems the rate of separation of oil decreased with time of ultracentrifugation (i.e., with decrease in the quantity of residual emulsified oil). The data are discussed in terms of their bearing on selection of criteria for quantitative comparison of emulsion stability and of their use in attempting to identify the rate-determining step in the demulsification process.

133
SOUND AND SAFE DETERGENT BUILDERS. ERIC JUNGBERMANN and H.C. SILBERMAN, Armour-Dial, Inc., Chicago, Ill. 60608.

The impact of detergent builders on our environment is receiving a great deal of attention lately. The question of adequate sewage treatment in view of contemplated changes in domestic wastes composition is a matter of concern for sanitary specialists and lawmakers alike. The present paper explores possible consequences if phosphates are replaced by carbonates in household detergents. Studies have been conducted to assess the influence of alkalinity and of carbonates on aquatic sewage treatment processes, on algal growth, and on aquatic toxicity. The paper will review the state of knowledge and present our findings.

134
PERFORMANCE OF NONPHOSPHATE AND PHOSPHATE HEAVY DUTY LAUNDRY DETERGENTS. R.C. STRUBHAUER, A.J. WYSOCKI and E. JUNGBERMANN, Armour-Dial, Inc., Chicago, Ill. 60608.

The performance characteristics of a nonphosphate detergent product based on a sodium carbonate and liquid sodium silicate builder system have been compared with the leading phosphate-built products. Both standard laboratory and extensive consumer tests were carried out. Laboratory results were obtained over a wide range of water hardnesses and under a variety of test conditions. Data on detergent, soil redeposition, optical brightening, inorganic ash build-up and consumer reactions will be presented. Some data will also be included comparing the safety features of the phosphate vs. the nonphosphate products.

135
PROJECTION AND PROSPECTS FOR RAPESEED AND MUSTARD SEED. RAGNAR OHLSON, AB Karlshamn Oljefabrik, Karlshamn, Sweden.

Abstract not available at press time.

136
PROJECTION AND PROSPECTS FOR HARVESTED FISH. M. SPRAGUE, University of Rhode Island, Kingston, R.I. 02881.

Abstract not available at press time.

137
PROJECTION AND PROSPECTS FOR ANIMAL FATS AND MEATS. GEORG BOGSEFOM, Michigan State University, East Lansing, Mich. 48823.

Abstract not available at press time.

138
PROJECTION AND PROSPECTS FOR SOYBEANS. E.R. LENG, University of Illinois, Champaign, Ill. 61820.

Abstract not available at press time.

139
QUANTITATIVE THIN LAYER CHROMATOGRAPHY OF GANGLIOSIDES. S.R. MAX, R.A. SNYDER and R.O. BRADY, University of Maryland School of Medicine, Baltimore, Md.

Gangliosides are important constituents of the brain and of many extraneural tissues. Because they are affected in certain diseases and probably play a significant role in membrane phenomena, the quantitative determination of these substances in tissues under various conditions is of great importance. In this lecture, we will compare procedures which have been employed for the quantitative analysis by thin layer chromatography of these complex glycolipids, with particular emphasis on two which we consider to be most useful. The first procedure, described by MacMillan and Wherrett (J. Neurochem. 16:1621, 1969), involves visualization of separated gangliosides on the silica using the resorcinol spray reagent (Svennerholm, B.B.A. 24:604, 1957), scraping silica containing resorcinol-positive material (gangliosides) from the plate, and reaction with resorcinol reagent followed by extraction

of the chromophore and determination of optical density spectrophotometrically. The second technique also employs the resorcinol spray reagent but involves direct spectrophotometric determination of gangliosides on the thin layer plate with the Zeiss M4 QIII chromatogram spectrophotometer. This procedure has recently been applied (Max, Nelson and Brady, J. Neurochem. 17:1517, 1970). Both methods are rapid, sensitive and precise. We will describe each in detail and will compare the results of ganglioside analyses employing them.

140

DETERMINATION OF POLYMER MOLECULAR WEIGHT DISTRIBUTION BY THIN LAYER CHROMATOGRAPHY AND DIRECT SCANNING NEUTRON TOMOGRAPHY. E.P. OROOKA, Bell Laboratories, Murray Hill, N.J. 07974.

The molecular weight distribution of a commercial polystyrene is determined by thin layer chromatography (TLC) and direct scanning densitometry. The results of the TLC experiment are in good agreement with those derived by gel permeation chromatography. A number of other experiments are described which examine the mechanism of fractionation in the TLC of polymers. Both adsorption and solubility mechanisms appear important.

141

THIN LAYER CHROMATOGRAPHIC METHODS IN ANALYTICAL TOXICOLOGY. PAUL SCHWEDA, III Penn Street, Baltimore, Md. 21201.

The usefulness of thin layer chromatography for toxicological investigations is critically discussed in regard to the type of coatings, choice of solvent systems, mode of development, detection limits and methods for spot locations. Problems of positive identification of narcotics and dangerous drugs in body fluids of living persons as well as in autopsy material are stressed. A discussion of quantitative and semiquantitative analysis in situ and on eluted samples concludes the critical review.

142

QUANTITATIVE ASPECTS OF THIN LAYER CHROMATOGRAPHY IN AIR POLLUTION MEASUREMENTS. T.W. STANLEY, Environmental Protection Agency, Research Triangle Park, N.C.

Thin layer chromatography (TLC) has been used to separate complex mixtures of organic extracts of suspended airborne particulate matter. This technique has been applied to air pollution measurements to determine many toxic organic compounds present in ambient air and emission sources. One-dimensional separations, using adsorbents such as silica gel, cellulose and acetylated, cellulose have been used to separate phenols, azaheterocycles, polynuclear aromatics and oxygenated aromatics. These rapid separations of classes have resulted in routine analytical methods for the quantitative determination of benzo(a)pyrene, benz(c)acridine(s), acridine, carbazole(s), phenanthrene, and 7H-benz(4,5)anthracen-1-one in polluted atmospheres. Using simple elution techniques followed by spectrophotometric or spectrophotofluorimetric measurements, far less than one nanogram amounts have been fairly accurately determined. Two-dimensional separations using a mixed adsorbent layer of silica gel and acetylated cellulose has been used to determine internal concentrations of pyrene, benzo(a)pyrene, benzo(k,h,i)perylene, and benzo(k)fluoranthene in a polluted urban atmosphere. Peak concentrations, averaging 18-30 $\mu\text{g}/\text{m}^3$ for the well known carcinogen, benzo(a)pyrene, were found to occur between 7 and 9 A.M. A second peak, concentration one fourth that of the 7-9 A.M. peak, was found to occur between 4-6 P.M. Using direct spectrophotometry, no elution was necessary and sensitivities of .01 nanograms were possible with little loss in accuracy when unknowns were compared to pure standards used in the same system. This same two-dimensional separation on a mixed adsorbent layer has been used to determine five polynuclear aromatics found in organic extracts of particulate matter collected from automobile exhaust emissions. TLC has proven to be a valuable tool for assessing air quality and determining the contribution of various sources to the pollution of our atmosphere.

for microsomal or mitochondrial activation of *trans* acids are higher than the Km values required for activation of the corresponding *cis* acids. Also, all *trans* retinoate d- α -tocopherol acid succinate or rhodogeroate were not activated themselves but extensively inhibit activation of *cis* acids and linoleate while not affecting activation of *trans* monounsaturated acids or palmitate. Other detergents such as Triton-X-100 or sodium deoxycholate were shown to inhibit or activate acyl-CoA biosynthesis depending on the concentrations of detergent. The amount and type of protein and the fatty acid configuration and concentration used in the incubation medium. The presence of two long chain activating enzymes in rat liver microsomes and mitochondria is suggested; one enzyme appears to activate saturated and *trans* monounsaturated fatty acids and a second enzyme apparently catalyzes the activation of *cis* mono-, di- and polyunsaturated acids and *trans,trans* linoleic acids.

146
LACK OF UNIVERSALITY OF THE RESISTANCE TO AUTOXIDATION OF CHOLESTERYL ESTERS IN AQUEOUS COLLOIDAL SUSPENSION. L.N. NOZOLA, Temple University School of Medicine, Philadelphia, Pa. 19140, and V. MAHABDEVAN.

Early studies have shown that free cholesterol is readily autoxidizable by aeration in aqueous colloidal suspension at 85°C and that esterification of cholesterol with some long chain fatty acids causes complete inhibition of this autoxidation. More recently (JAOS 42:847, 1965) it was shown that cholesterol linoleate shows a different behavior in that the cholesterol moiety is as readily autoxidizable as free cholesterol itself. We wish to report at this time that introduction of an oxygen functional group in the acyl chain of a cholesterol ester does not render the cholesterol susceptible to this type of autoxidation. We have studied several such compounds. Furthermore, we have found that the tetrahydrocannabinol derivative of cholesterol linoleate shows a remarkable and unusual susceptibility to autoxidation while cholesterol ricinoleate itself is completely resistant. The OH-protected cholesterol ricinoleate autoxidizes slowly compared to free cholesterol but with considerably more extensive conversion to autoxidation products. It appears likely that the behavior of the OH-protected compound is a striking and unique example of the influence of a molecular hydrophobicity change, quite far removed from the reactive center which effects the orientation of the molecule in a colloidal system, in a manner which renders the reactive center susceptible to autoxidation.

147
LIPID CHANGES IN BEAGLE DOG LUNGS AFTER IRRADIATION OF ¹⁴CE-FUSED MONTMORILLONITE CLAY AEROSOLS. R.C. PRAGER, H.C. RADMAN, and S.A. BENJAMIN, Lovelace Foundation for Medical Education and Research, Albuquerque, N.M. 87108.

Three groups of Beagle dogs (four animals per group) were exposed to a ¹⁴Ce-fused montmorillonite clay aerosol and cumulated by two, four and six months postinhalation approximately 17,000, 26,000, and 44,000 rads to the lung, respectively, when the groups were sacrificed. This resulted in a progressive radiation pneumonitis. At monthly intervals, certain members of a group were anesthetized and their lungs (diaphragmatic lobe) intubated with a Metras catheter. Isotonic saline (100 ml) was introduced and withdrawn three times (fresh solution each time). The recovered lavage solutions were centrifuged and lung lining cells and pulmonary surfactant isolated, lyophilized and lipid-extracted. At sacrifice, the lungs were quickly obtained, minced, lyophilized and lipid extracted. Total lipids from all samples were gravimetrically determined and individual compounds were identified, isolated and quantitated by thin layer chromatography. The quantity of lipid present in whole lung tissue, pulmonary lining cells and surfactant was observed to increase as a function of radiation dose. More specifically, the neutral lipids (triglycerides and sterol esters) accounted for most of the increase of the total lipids. However, the quantity of phosphatidyl choline also increased with radiation dose but to a lesser degree than the neutral lipids.

148
ACTIVATION OF TRANS-UNSATURATED LONG CHAIN FATTY ACIDS BY RAT LIVER SUBCELLULAR FRACTIONS. KENNETH LIPPEL, Human Nutrition Research Division, ARS, USDA, Beltsville, Md. 20705.

Several differences have been observed in the parameters that influence activation of *cis* and *trans* unsaturated long chain fatty acids. First, the fatty acid to protein ratio required for maximum rat liver microsomal or mitochondrial activation of the *trans*-monounsaturated acids elaidate or *trans*-palmitate was similar to the ratio required for maximum activation of the *cis*-unsaturated fatty acids oleate and *cis*-stearate or of the *trans,trans* diunsaturated linoleic acid. However, when the substrate was a fatty acid-albumin complex rather than free fatty acid, the optimal fatty acid to protein ratio for *cis* or *trans* unsaturated acids or for palmitate maximum acyl-CoA synthesis all fall in the same range. Second, the Km values for coenzyme A and ATP required

143
RAPID DETERMINATION OF THE SPECIFIC ACTIVITY OF ISOTOPIC MATERIAL USING THIN LAYER DENSITOMETRY. J.C. TOUCHSTONE, W. WORTMANN, M. KASPAROW and T. MURAWKO, University of Pennsylvania Hospital, Philadelphia, Pa. 19104.

A problem of analysis of radioactive materials isolated from biological sources lies in identification of trace amounts of substances. The conventional method for indication of purity of an isolated compound seems to be recrystallization to constant specific activity. A simple and faster method for determination of specific activity by thin layer chromatography (TLC) has been developed. With silica gel containing a phosphor, direct quantitation by densitometry of fluorescence quenching is possible. A direct relationship exists between the amount placed on the plate and instrumental response. Quantitation of radioactivity was performed by scintillation after elution and removing an aliquot of the sample. Specific activity was determined after each separation in four solvent systems. A deviation of more than 5% indicated the impurity of the substance. The method is limited to substances absorbing UV light. Silica gel containing phosphomolybdic acid was used for nonlight absorbing compounds. Heating of the plate after development produced a blue color which was quantitated by spectrodensitometry (Schoeffel Model 3000).

144
LIPID PEROXIDATION IN ERYTHROCYTES FROM VITAMIN E DEFICIENT RATS. MYRA O. BARKER and MYRON BRAN, Hoffmann-La Roche Inc., Nutley, N.J. 07110.

Erythrocytes taken from rats maintained on a vitamin E deficient diet were incubated at 37°C for 2 hr in a phosphate saline buffer under a nitrogen atmosphere. Three different lipid peroxide-generating systems were used: dialuric acid in solution, gaseous hydrogen peroxide, or a peroxide-generating system consisting of glucose oxidase and glucose. Hemolysis was 90% complete at the end of 2 hr in samples from E deficient rats and essentially zero in samples from E supplemented controls. Following incubation, lipids were extracted from the stroma and subjected to two-dimensional thin layer chromatography for quantitation of products. Disappearance of polyunsaturated fatty acids was quantitated by gas chromatography of the methyl esters of total stroma phospholipids. Of the three peroxidizing systems, glucose oxidase-glucose produced the most consistent results as measured by polyunsaturated fatty acid disappearance.

145
RADICAL ADDITION OF LINOLEIC ACID HYDROPEROXIDE OR ITS METHYL ESTER TO α -TOCOPIEROL OR ITS MODEL COMPOUND, 2,2,5,7,8-PENTAMETHYL-6-HYDROXYCHROMAN. H.W. GARDNER, G.W. GRAMS and K. ESKINS, Northern Regional Research Laboratory, USDA, Peoria, Ill. 61604.

Linoleic acid hydroperoxide or methyl linoleate hydroperoxide react anaerobically with α -tocopherol or its model compound 2,2,5,7,8-pentamethyl-6-hydroxychroman to form principally an addition compound of 85% reactants. The reaction was catalyzed by 1.28 X 10⁻³ M Fe(II) or photoactivated flavin (0.01%). The presence of oxygen in the reaction terminated the addition, and resulted in the formation of quinones from α -tocopherol or its model compound. The addition compound synthesized from 2,2,5,7,8-pentamethyl-6-hydroxychroman and linoleic acid hydroperoxide (a 4.9 to 1 ratio of 18-hydroperoxy-*cis*-9, *trans*-12-octadecenoic acid and 9-hydroperoxy-*trans*-10-*cis*-12-octadecenoic acid) was used to solve the structural details of the bridging function. Three isomers of the addition compound (methyl esterified) were isolated and identified. In order of decreasing abundance the isomers were methyl 11-(2,2,5,7,8-pentamethyl-6-oxychroman)-*cis*-12,13-epoxy-*trans*-9-octadecenoate, methyl 11-(2,2,5,7,8-pentamethyl-6-oxychroman)-*trans*-12,13-epoxy-*trans*-9-octadecenoate, and methyl 11-(2,2,5,7,8-pentamethyl-6-oxychroman)-*cis*-9,10-epoxy-*trans*-12-octadecenoate. The mechanism appeared to be free radical addition brought about by the catalytic formation of alkoxy radicals from the hydroperoxide and chromanoxyl radicals from α -tocopherol or its model.

19%, 36%, 55%, 62% and 68% of the initial amount of MP was hydrolyzed and released into the medium at 5, 15, 30, 60 and 120 min, respectively. In 1 hr, 2.1% of the initial amount of MP and 2.5% of PA were oxidized to CO₂, and 18.5% of MP and 18.1% of PA were converted to lipids. There was no significant difference between the patterns of lipids incorporation from MP and PA. It is concluded that rat lung is capable of utilizing MP and converting it to other lipids, predominantly PL. The progressive decrease in tissue FFA and corresponding increase in PC, DG and TG suggest that FFA is a hydrolytic product of MP and is reesterified to DG, TG and PO and excessive tissue FFA is released into the medium. The similarity in tissue lipid patterns in experiments using MP and PA indicate that the pathway for PL synthesis from these two substrates is the same after hydrolysis of MP. The rapid increase in tissue and release of FFA into the medium suggest the presence of an active MP-hydrolyzing system in the rat lung and that the hydrolytic reaction is not a rate-limiting step for uptake and metabolism of MP.

151

SHORT METHOD FOR THE DETECTION OF CHICK EDEMA FACTOR IN FATS, OILS AND FATTY ACIDS BY ELECTRON CAPTURE GAS CHROMATOGRAPHY. N. KINS and J. BARANDY. Drew—Division of Pacific Vegetable Oil Corporation, Boonton, N.J. 07005.

This paper describes a short method for the detection of Chick Edema Factor in plant oils and fatty acids. While the existent official method is time consuming, this method requires less than 3 hr to analyze one sample, and even less time per sample with batch analysis. Complete assessment of this short method by industry and the F.D.A. is necessary before it may be considered for adoption as an official method. This method consists of two parts. The first part is a preparation of a sample for the subsequent second part, gas chromatography. The difference between this short method and the official method is in the first, preparative part, the parameters of the second part (gas chromatography) are the same as described in the official method. Samples are prepared for the gas chromatographic analysis by subjecting them to the double preliminary sulfuric acid cleanup and a caustic wash extracting the chick edema factor each time with trimethyl pentane. The final extract is washed with sulfuric acid again and examined by the electron capture gas chromatography. Gas chromatographic peaks with retention times 78, Aldrin (R_f values) between 3 and 45 are indicative of the presence of chick edema factor.

152

PLASMA MEMBRANE FRACTIONS FROM NORMAL AND ESSENTIAL FATTY ACID DEFICIENT EHRLICH ASCITES TUMOR CELLS. H.L. Chabrin, Lever Brothers Company Research and Development Center, Edgewater, N.J. 07020, and K.A. NAMAYAN.

Unexpected difficulties were encountered in the use of the procedure of Kamat and Wallach for the isolation of surface membrane from Ehrlich ascites tumor cells. Therefore, we have developed a modification of the procedure of Neville for rat liver and have obtained two fractions with high Na⁺-K⁺-Mg²⁺-ATPase activity. The procedure involved homogenization of the cells in 1.0 millimolar bicarbonate buffer followed by sucrose gradient centrifugation of a 1,500 xg precipitate. The fractions floating on top of sucrose solution of *d* 1.16 and the interphase between *d* 1.16 and 1.18 were considered to be rich in plasma membrane fragments as judged by their ATPase activity. The fractions were characterized by their enzyme activities (succinic dehydrogenase, DPNH diaphorase and ATPase), lipid to protein ratios, cholesterol and phospholipid content, and fatty acid composition. Nuclei, mitochondria and microsomes were isolated by conventional methods and their chemical composition and enzyme activities were compared with the various density fractions. This procedure was also used to obtain the plasma membrane fraction from cells grown in essential fatty acid deficient mice. These membranes had decreased levels of linoleic and arachidonic acids with increased levels of palmitoleic, oleic and eicosatrienoic acids. The ATPase activity of the *d* 1.16 float and the 1.16-1.18 interphase were lower in the essential fatty acid deficient fractions as compared to corresponding values obtained with normal cell fractions. An interesting observation of these studies was that the density fraction isolated from essential fatty acid deficient, in contrast to normal tumor cells, had substantially higher percentages of cholesterol.

153

CENTRIFUGAL APPLICATION RELATING TO FATS AND OILS WASTE STREAM. A.E. ZAMBONE, Sharples Corporation. Abstract not available at press time.

154

POLLUTION CONTROL FACILITIES AT A GLIDDEN-DURKEE REFINERY. T.A. WALSH, Glidden-Durkee Division, SCM Corporation. Abstract not available at press time.

155

METHOD OF CONTROLLING SULFURIC ACID FUMES FROM AN INDUSTRIAL OPERATION. FRANK E. SULLIVAN, Frank E. Sullivan Company. Abstract not available at press time.

156

YOUR AOCs COMMITTEE ON POLLUTION PROBLEMS. CLIFFORD HAYSLEY, Glidden-Durkee Division, SCM Corporation. Abstract not available at press time.

157

REVIEW OF HEXANE EXTRACTABLE MATERIALS AS RELATING TO PRESENT EFFLUENT STANDARDS. GEORGE M. KERUZZER, Swift and Company. Abstract not available at press time.

158

USE OF IMPROVED CURRENT IN WASTE WATER TREATMENT. DAVID R. ERIKSON, Swift and Company. Abstract not available at press time.

159

AIR POLLUTION REGULATIONS AND IMPLEMENTATION PLANS: PAST, PRESENT AND FUTURE. FRANCIS SCORFIELD and D.S. KING, King and Scofield, Washington, D.C. 20036.

With the enactment of the 1970 amendments to the Air Quality Act of 1967, the rules of the game were substantially altered. Specifically, the establishment of Air Quality Standards was transferred from the various states to the federal government, and the enactment and enforcement of "Implementation Plans" by which these standards might be achieved is left to the states. These implementation plans are in process of development but will not be final until early in 1972. In general, the states seem to be going two different ways. Some are following Los Angeles, using modifications of Rule 68 while others are aiming at the general reduction of emissions of all organic solvents. The implications of both approaches to the coatings solvent extraction, dry cleaning, and other industries, will be discussed.

