

Highlights of the 36th Fall Meeting—II Outstanding Technical Program Reviewed

(The second and last of a series, beginning in the November issue, designed to offer a detailed review of the 36th AOCs Fall Meeting.)

Symposia Highlight—Sixteen-Session Series One Hundred and Four Papers Presented

Soon after the first call for technical papers to be delivered at the Toronto Meeting, it became obvious that this Program was to be not only the largest in AOCs history, but certainly the most varied—offering presentations of interest to virtually every element of the fat and oil field. Yet, the number—and indeed the quality—of technical papers cannot alone be the measure of a successful program. The factors of pre-planning and organization dominate as proper balance is sought. Much credit is due the Toronto Program Committee which, under the direction of H. W. Lemon, was braced to meet the expected tide of enthusiasm, and which prepared and presented a program wherein each and every interest was looked after from beginning to end. Initial apprehension and reservation, due to the size of the program, quickly turned to praise as those attending found a steady series of interesting and well-attended sessions, no matter what particular objectives they individually sought. Conflicts in programming were virtually non-existent, and time was utilized to the utmost.

Highlighting the program were two symposia: "In Vivo Antioxidants and Polyunsaturated Acid Metabolism" and "Symposium for Producers." Authors from nineteen States, Canada, England, Poland, Japan, Holland, and Mexico presented one hundred and four papers. Presiding at the sixteen technical sessions were: Sven Young, F. H. Lehberg, Raymond Reiser, Paul Sheffer, C. Y. Hopkins, H. K. Hawley, O. S. Privett, A. J. Stirton, S. F. Herb, W. O. Lundberg, H. J. Dutton, R. T. O'Connor, E. I. Birnbaum, L. R. Dugan, H. G. Willsie, R. A. Burt, and H. H. Engel.



OPENING TECHNICAL SESSION—Sven Young presiding.
Left to right: H. R. Sallans, H. S. Hissocks, A. A. McKerrigan, W. D. Raymond, and Sven Young.

Tropical and Canadian Oils; UK Edible Fat Industry

The opening Technical Session offered discussions of oils and oilseeds in the Tropics and in Canada, and a review of the edible fat industry in Great Britain.

W. D. Raymond, Tropical Products Institute, London, described the production of palm oil, palm kernels, and groundnuts in Africa, illustrating his talk with color slides. He showed how improved methods and better control of lipolysis, chemical hydrolysis, and the action of moulds are improving the quality of the oils.

E. S. Hiscocks, Director, Tropical Products Institute, emphasized the danger of mould growth. A toxic substance termed "Aflatoxin" produced by the growth of *Aspergillus flavus* on groundnuts has been isolated. This substance has been responsible for the deaths of domestic animals and birds fed the meal after oil extraction from mouldy seeds.

A. A. McKerrigan, J. Bibby and Sons, Ltd., Liverpool, discussed the edible fat industry in Great Britain and compared it with that in the United States. Great Britain depends upon imports for her edible oil supply and problems are created by price fluctuation. Ingredient fats are more numerous and varied than in the U.S.

H. R. Sallans, Prairie Regional Laboratory, Saskatoon, described research leading to the development of oil seed crops in Canada, particularly on the Prairies. Rapeseed has become an important crop. It is produced in more northerly areas than sunflowers, another Canadian crop. Both Argentine and Polish varieties of rapeseed are grown, but the latter is preferred. Extensive plant breeding experiments are in progress, and they have yielded a rapeseed oil containing no erucic acid.

Dr. Sallans stressed the need for an oil seed crop with a high protein content.

Panel Discussion: The Role of Fats and Oils in Nutrition with Particular Reference to Cholesterol Metabolism

W. O. Lundberg: A prolonged change in cholesterol metabolism may not necessarily affect the development of atherosclerosis. Dietary cholesterol may affect the level of cholesterol in the blood. It is known however, that blood cholesterol levels are also influenced by synthesis of cholesterol in the body. This synthesis in the body is in turn affected by dietary factors.

Corn oil in the diet of experimental animals brings about a lowering of cholesterol in the blood but a simu-

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taneous increase in cholesterol in the liver and other organs. Feeding unsaturated fats to pigs and other experimental animals resulted in an immediate drop in blood cholesterol of the animals. After continued feeding, however, blood cholesterol gradually increased until it equalled that of control animals on normal diet.

Total caloric intake may be just as important as the amount of fat in the diet for the control of blood cholesterol levels.

The relation of diet to coronary heart disease is still poorly understood. On the basis of present scientific evidence there is no justification for recommending drastic diet changes to the general public.

Raymond Reiser: Fats have a definite function in the diet, such as taste, giving a feeling of satiety, etc.

Persons on a fat free diet gradually lose the ability to utilize fats. Essential fats must be included in the diet. Dietary unsaturated fatty acids increase the requirements in the body of vitamin E and antioxidants.

Excessive dietary fat of any kind is undesirable.

F. H. Mattson: Polyunsaturated fatty acids are not essential fatty acids in the classical sense. Much higher concentrations are intended when discussing the control of blood cholesterol levels.

Butter and other short chain fatty acids cause rise in cholesterol.

In making comparisons between diet constituents, the total caloric content as well as total cholesterol content must be constant. Statistical analysis of data should be carried out. It is not possible to lower blood cholesterol levels more than about 20% by means of diet changes.

J. D. Justice: Dr. Justice compared diets containing *trans* vs. *cis* fatty acids. Diets contained safflower oil and hydrogenated safflower oil. Using human subjects (prison convicts), no significant differences were noted. In rabbits and other small animals fed *cis* and *trans* acids, no significant effect was noted in *post-mortem* examination of organs.

J. M. R. Beveridge: In addition to the nature of fatty acids and glycerides in the diet of humans, some factors in the non-glyceride fraction are also of significance in their effect on blood cholesterol levels.

Ingested cholesterol is important in the control of blood cholesterol.

K. F. Mattil: A direct causal relationship between diet and heart disease has not been proven. Only large, carefully controlled tests with humans can answer this question. The large-scale, long-term, controlled dietary study planned by Dr. Page of Cleveland Clinic is open to criticism because of poor planning. A major part of the diet to be used will be safflower oil which is not obtainable in commercial quantities. The diet should contain fats readily obtainable by the general public to be practical and economical.

C. G. King (recapitulation): Food fats have still not been definitely proven to be responsible for atherosclerosis. High levels of blood cholesterol are a factor in atheros-

clerosis in many cases, but there are also other important factors. The body manufactures cholesterol. Blood cholesterol rises in response to tobacco smoking, anger, or nervous excitement. Diversity of food, eaten in moderation, including fats, is the best defense. Gorging at meals is bad and tends to speed clotting time. Long gaps between meals are not good since they encourage gorging. Frequent meals are better.

In Vivo Antioxidants and Polyunsaturated Fatty Acid Metabolism

A. L. Tappel, Food Science and Technology, University of California, reported that mitochondria have been studied as examples of membrane structures. The mitochondria require polyunsaturated fatty acids but not any specific fatty acid. Peroxidation of the polyunsaturated fatty acids is very damaging to membranes *in vivo*. Natural defenses include vitamin E, sulfur amino acids, and selenium compounds.

L. J. Machlin, Monsanto Chemical Co., in discussing the biological consequences of feeding polyunsaturated fatty acids to antioxidant-deficient animals, pointed out that a combination of high levels of polyunsaturated fatty acids and low levels of lipid antioxidants gives a variety of symptoms in experimental animals. The incidence of encephalomalacia in chicks is related to peroxidation of polyunsaturated fatty acids in the cerebellum.

Regarding the dietary antioxidants in young swine, E. G. Hill noted that high levels of polyunsaturated fatty acids cause erythrocyte hemolysis in tissue peroxidation. The latter is prevented by low levels of vitamin E or santonin, but prevention of erythrocyte hemolysis requires much higher levels of antioxidants.

J. E. Oldfield, Oregon State University, discussed the effects of *in vivo* antioxidants in feeding oils to swine. Lard, fresh menhaden oil, and menhaden oil oxidized under controlled conditions with and without added antioxidants were fed at the 10% level to pigs. The oxidized menhaden oils lowered food intake and efficiency of feed conversion proportional to the degree of oxidation. Addition of antioxidants improved the rates of gain although not to the level of the lard group.

J. G. Bieri, National Institute of Arthritis and Metabolic Diseases, described studies on the metabolic effects of selenium and vitamin E with rats and chicks. Incubation of tissue homogenates from vitamin E deficient chicks with ingested selenium showed antioxidant action on the rate of tissue lipid peroxidation. Studies on coenzyme A levels in chick liver showed relationships between selenium, vitamin E and the sulfur amino acids. Feeding of selenium or α -tocopherol to chicks offset the toxicity and deaths which appeared to be caused by addition of antioxidants at relatively high dietary levels.

P. P. Nair, Biochemistry Research Division, Sinai Hospital of Baltimore, Inc., showed the separations and quantitative determinations of vitamins E and K using GLC. A mixture of two silicone polymers was found to give the best separations of tocopherol, α -tocopherol quinone, and α -tocopherol hydroquinone. Derivatives of the tocopherols



PANEL DISCUSSION—F. H. Lehberg presiding. Left to right: F. H. Lehberg, K. F. Mattil, Raymond Reiser, W. O. Lundberg, C. G. King, moderator, J. D. Justice, F. H. Mattson, and J. M. R. Beveridge.



IN VIVO ANTIOXIDANTS AND POLYUNSATURATED ACID METABOLISM SYMPOSIUM, Technical Sessions A and E; Raymond Reiser presiding. Seated: B. F. Craig (left), and Raymond Reiser. Standing: (left to right) L. J. Macklin, E. G. Hill, A. L. Tappel, P. P. Nair, J. G. Bieri, and J. E. Oldfield.

were prepared and characterized by gas liquid chromatography. The sensitivity of this technique was discussed in relation to analysis of tissue.

Symposium for Producers

F. W. Holm, Corn Products Co., discussing the problem of Personnel, stated that for many years we were ennobled by training, believing the acquisition of knowledge and skill would result in high productivity. More important is the extent to which training is put to productive use, e.g., Job Instructor Training, Job Methods Training, and Job Relations Training.

Deep sociological changes taking place throughout the world in the past decade resulted in a more intimate and continuing interest and awareness in the group process and individual relationships. The important power relationship was the transfer of responsibility back to the line organization. *Objectives, Expectations, and Accountability* have become an integral part of management vocabulary. Important in these new evaluations are the superior-subordinate relations. The superior must be aware of his values, objectives, and the reaction and inter-action of others with him; the subordinate's continuous task is self-development.

R. T. Forest, Lever Brothers, Ltd., reporting on the ever-widening field of Communications, noted that the chief area of interest is the *intra-personal* communication within the individual which in turn has an important bearing on the effectiveness of the *inter-personal* exchanges of ideas and information.

Basic communication is a message to be transmitted. Better understanding of our thought processes, past experience, and attitudes have an important bearing upon our perception of the communication. Research work has thrown new light on communications, distortions of which may result from improper observations and description and the inadequacy of the language we use to convey. Much is lost when conveyed to a second, third, and fourth party when relying on words alone. Relationship as well as details of fact must be included in communications. It must be a two-way process; the receiver being able to ask questions or feed back information.

In group discussions, proper statement of the problem, avoidance of forming evaluations in the early stages before all the facts are in, and "brainstorming" of evaluations are important.

In inter-personal communications, assessing what people actually say does not always communicate what they are trying to say. Personal, psychological, and environmental factors may have an inhibitive effect. Misunderstanding can arise from bad listening habits. Also, the feeling of subject matter and delivery are important in communications.

It is important to the superior that the subordinate has a clear understanding of his job. There should be an efficient flow of communication up, down, and across the organization to convey information to points where decisions are made. Periodic discussions on various levels within the

organization and subsequent communication of progress and problems within each group to higher levels keep management informed on all aspects. Communications is one of the decisive processes in organizational coherence not only within itself but also in its relationship to the outside world.

H. E. Marxhausen, Cargill, Inc., pointed out that Technical Safety is a broad, encompassing term. Safety lies in two areas—first, in the physical plant and second, with personnel.

The following factors play an important part in the physical plant safety: Maximum protection against fire, explosion, personal injury, injury to general health of employees, location of a building in relation to others, design, climatic conditions, and building ventilation.

Adequate fire protection is a necessity. This includes: proper number, size, and type of fire extinguishers; automatic sprinklers or deluge system if there is an adequate water supply, or foam system is highly recommended; and a trained fire brigade, no plant being too small for this protection.

Technical safety in many cases is not given adequate attention by personnel, a safety program usually being pushed into background. Top management must be convinced of the need for a safety program and there should be constant personnel training and follow-ups.

The importance the Technical Safety Committee and various subcommittees played in the revisions of the NFPA standards was stressed and highly commended.

W. J. Johnson, The Buckeye Cellulose Corp., stated that accident prevention in industrial laboratories is largely a job of "selling" safety and providing the necessary train-



SYMPOSIUM FOR PRODUCERS, Technical Session B—P. R. Sheffer presiding. Left to right: P. R. Sheffer, W. J. Johnson, and F. W. Holm. (Not pictured: R. T. Forrest.)

ing and equipment to do the job. A continuing interest and attention to matters of safety by all supervisors is essential. A balance of both oral and written instruction is called for in terms that can easily be understood. The most common hazards to be guarded against in any laboratory were found to be toxicity, chemical burns, fire, electrical shock, and glass cuts. Each laboratory should develop two check lists for safety: 1) a list of safety equipment, and 2) safety procedures.

The importance of safety precautions and good house-keeping being exercised was emphasized in the use of inflammable materials, corrosive materials, solvent vapors and gases, chemical dusts, mercury, cyanosis, cyanides and other poisons, and disposal of laboratory wastes. Design and layout of equipment and furniture play a vital part in the safe operation of any laboratory. Safety comes from a proper attitude of mind and depends on the cooperative effort of each individual.

In discussing instrumentation, W. F. Bollins, Swift & Company, noted that it has been used, in one form or another, in the oil and seed processing industry from its very beginning. The amount of instrumentation used has steadily increased. In oil seed processing the technology has developed from small-scale batch operation to large-scale continuous operations. In the earlier processing plants instrumentation used was limited principally to thermometers and pressure gauges.

In later developments semi-continuous and continuous cooking were coupled with hydraulic pressing and later

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news a- poppin'

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with continuous screw pressing, steam pressure controllers being used to control steam jacket temperatures. Instrumentation used has also progressed in that better temperature controllers are used. Other instrumentation which has helped in developing the continuous process has been the electrical interlocking of materials-handling equipment, electrical overload protection devices, and the use of ammeters.

The solvent extraction process has also developed through a number of stages from more or less simple batch processes to the modern continuous process. The important role that instrumentation has played in this development was thoroughly reviewed.

Todd Frohman, International Business Machines Corp., reviewed the controlling of industrial processes through the use of digital computers. By simple instrumentation connection of the computers to industrial processes, these "Logic Boxes" will function as the "perfect operator" in all phases of a particular process. In addition, they will decide what can be done to the process to make more money.

Industry is finding that both in the fields of research and actual manufacture of the product, "on-line" digital computers are paying their way. By controlling all phases of an operation and rapid communication of facts, management can now make the quick decisions so necessary to dynamic operations.



SYMPOSIUM FOR PRODUCERS, Technical Session F—P. R. Sheffer presiding. Seated, left to right: W. F. Bollens and P. R. Sheffer. Standing, left to right: N. H. Moore, H. E. Marxhausen, W. R. Wingard, L. T. Frohman.

Digital computers can now be applied to fat rendering, pilot plant, and starch hydraulics operations. Efficient data collection, controlling multi-variables in more than one process and the rapid communication of facts, have made digital computers invaluable in these processes.

On the subject of construction, N. H. Moore, N. Hunt Moore & Assoc., and K. W. Becker, Blaw-Knox Company, emphasized the element of safety in engineering and construction of solvent-extraction plants. Choosing the proper location requires consideration of elevation, winds, special hazards, drainage, housing for the solvent area, and rules of proper ventilation and explosion relief.

The main consideration is in the protection of equipment and personnel from the weather. The design of the foundation for both the building and the equipment is an important factor in the safety of a plant and a complete soil test should be run to determine the proper design.

The design of the equipment for safe operation, maintenance and sanitation should be carefully considered. Electrical planning and maintenance are very important. The use of a consulting engineer for National Electric Code standards is recommended. Every effort should be made to protect operating personnel against injuries due to falling. An emergency water supply for a solvent plant is very important in case of sudden shut-down by electrical failure and other emergencies.

Sanitation and housekeeping should be given top consideration in the arrangement of equipment. A plant that can be easily kept clean is one that will be kept clean.

M. R. Wingard reviewed the methods of reducing utility

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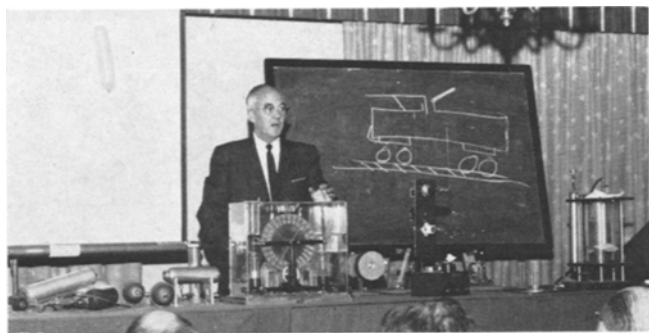
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costs by equipment and plant design, noting that the greatest possibilities lie in the extraction area.

The bean desolventizing operations produce vapors containing large quantities of latent heat. It is only a matter of finding ways to use it before consigning it to cooling water. Slides were shown illustrating in effect potential savings in heat conservation from the operation. The savings obtainable are shown in the construction of a new plant where a new boiler or cooling tower may be eliminated.

The economics involved in such reduction in utility costs are excellent since capital investment is negligible. However, a price may be involved inasmuch as some operations may require more consideration being given to design, preparation, operators, operating variables, consistency, conditions of cooling tower, and drainage, as well as working knowledge of cause and effect. Under the worst conditions the economics and potentials are excellent. In some plants as much as \$50,000 to \$60,000 per year savings have been realized. The price lies in operating care and exploration of the possibilities and making the most of them.

Mr. H. H. Engel, U. S. Department of the Interior, Bureau of Mines, gave a demonstration of the dangers of fire by static electricity, dust and gas explosions, that was both entertaining and scientific. The potential danger of gasoline was stressed, and also the explosive properties of gas-air mixtures and of dust-air mixtures. All demonstrations were very spectacular.



SPECIAL DEMONSTRATION, Technical Session P—H. H. Engel, presiding.

Structure and Composition

The use of modern techniques was well illustrated in the first three papers which dealt with the isolation of new and rare acids. K. L. Mikolajczak, Northern Utilization Research and Development Division, reported a high content of 11-eicosenoic acid (more than 40%) in three seed oils not previously studied, including one species of Compositae.

Occurrence of *trans,trans*-10,12-octadecadienoic acid, an isomer of linoleic acid, in a seed oil was described by C. Y. Hopkins, National Research Council of Canada. The acid was isolated and characterized.

Toru Takagi, in work carried out at Nagoya University, Japan, and the National Research Council, Saskatoon, showed that the seed oil of the primitive species, *Podocarpus nagi*, contains 20% of 5,11,14-eicosatrienoic acid. Identification was made by the newer techniques of partial oxidation and partial hydrogenation, followed by gas chromatography.

Carter Litchfield, A. & M. College of Texas, reported that isomerization of oleic acid by selenium or nitrous acid gives a mixture containing 75–80% of *trans* acid, not 67% as generally believed. A similar result was obtained with linoleic and linolenic acid, although some by-products were formed with selenium.

Retention times in gas chromatography were discussed by R. G. Ackman, Fisheries Research Board of Canada. He found that position isomers of monounsaturated acids provide retention time data which aids in the identification of more highly unsaturated acids.



STRUCTURE AND COMPOSITION, Technical Session C—C. Y. Hopkins presiding. First row, left to right: Elizabeth M. Kirby, K. L. Mikolajczak, C. Y. Hopkins, and Toru Takagi. Second row, left to right: C. C. Litchfield, R. G. Ackman. (Not pictured: H. Susi and C. R. Eddy.)

M. L. Blank, Hormel Institute, spoke on the use of thin layer chromatography in structure determination of glycerides and lecithins. It is clear that notable progress has been made in this difficult field.

Three speakers told of advanced work in infrared spectroscopy. H. Susi, Eastern Regional Research Laboratory, made use of both the 1.5 μ and 3 μ regions for qualitative determination of 1,2- and 1,3-diglycerides. C. R. Eddy, Eastern Regional Research Laboratory, described an infrared study of the rotational isomers in hydroxylic fatty compounds and the effect of hydrogen bonding in stabilizing some of them. Elizabeth M. Kirby, Ontario Research Foundation, demonstrated that both wagging and rocking vibrations give useful information on chain length and on double bond configuration of fatty acid salts.

General Session

A method for the quantitative analysis of butterfat triglycerides using gas-liquid chromatography was described by A. Kuksis, Queen's University. Thirty-one triglyceride peaks were recognized in butterfat and the results suggested that a specific triglyceride distribution occurs which deviates from random. The separation of triglycerides by liquid-liquid partition chromatography was outlined by E. G. Hammond, Iowa State University. The details for applying this system to the separation of model simple triglycerides and cocoa butter were described.

Another novel method of separating triglycerides as their mercuric acetate adducts was reported by J. A. Inkpen, Purdue University. After forming the mercuric acetate adducts of the triglycerides, they were separated on an alumina column using ethyl ether-acetate acid as the eluting solvent. The glycerides are separated substantially into classes according to their degree of unsaturation although there was some overlapping because of tailing between fractions.

T. J. Potts, Ralston Purina, gave a very clear presentation on why the neutral oil analysis should replace the cup loss method for trading soybean oil. It was shown that a lack of correlation between the cup loss method and the neutral oil analysis was inherent in the cup loss method.



GENERAL SESSION, Technical Session D—H. K. Hawley presiding. Seated, left to right: H. K. Hawley, Arnis Kuksis, E. G. Hammond, and T. J. Potts. Standing, left to right: S. F. Herb, J. A. Monick, N. H. Kuhrt, and C. F. Smullin. (Not pictured: J. A. Inkpen.)

The following formula was proposed for trading soybean oil: Premium = (5.00 - 1.00 neutral oil loss) 0.01 × \$10.00.

An elegant method for the determination of epoxyoleic acid by gas-liquid chromatography was introduced by S. F. Herb, Eastern Regional Research Laboratory. Methyl epoxyoleate was altered or decomposed during gas-liquid chromatography on polyester, Carbowax 20M and Apiezon "L" columns, but not on a silicone rubber (SE-30) column. By chromatographing the sample on a polyester column as well as on the SE-30 column (which does not separate fatty acids of the same chain length) all the fatty acid components of the oil were determined quantitatively.

J. A. Monick, Colgate-Palmolive, showed a relationship between the smoke point (temperature at which decomposition products become visible) of a superglycerinated shortening and free glycerol content. A method was developed for the continuous removal of glycerol from commercial monoglycerides with a thin-film evaporator where the residence time in the film evaporator was 11-26 seconds. The operation was carried out at 340F and 7 mm Hg absolute pressure, which reduced the glycerol content to 0.3%.

The composition, physical properties and applications of conjoined crystals (a new and improved food emulsifier) was the topic of Noel H. Kuhrt, Research Laboratories, Distillation Products Industries. The conjoined crystals are a mixture of saturated monoglyceride and the corresponding monoester of propylene glycol. This mixture disperses readily in water and retains this property for 2½ years. Since it readily disperses foam, it is used extensively in low calorie fruit fluffs, sponge cakes, and other products.

C. F. Smullin, Atlas Chemical Industries, Inc., described a method for the analytical estimation of Span 60 emulsifier in cake mixes and baked cakes. A gas-chromatographic method which measures isosorbide and a paper chromatographic method which is based on the isolation of 1,4 sorbitan with subsequent periodate oxidation, and the measurement of formaldehyde spectrophotometrically with chromotropic acid were outlined. The average recoveries of the emulsifier were 97%, with an average deviation from the mean of ± 5% absolute.

TLC Film—Overflow Attendance Requires Second Showing

A novel feature of the meeting was the motion picture in color on "Thin Layer Chromatography" shown by O. S. Privett. The setting was the Hormel Institute Laboratories and the film showed the practice of T.L.C. including such features as various spreaders and spray reagents as well as the techniques of sample application and development of plates. The overflow attendance at the first showing warranted a second session on Wednesday. This showing was also "standing room only."

Detergents and Surfactants

The application of flame spectrophotometry to the analysis of soaps and detergents for sodium, potassium and



"Standing room only" is all that is left in the British Columbia Room in the Royal York Hotel, where O. S. Privett presided at the early part of Session E on Tuesday, Oct. 2nd. A motion picture produced by Dr. Privett entitled "Thin-Layer Chromatography Basic Techniques and Research Applications" was shown, and then rescheduled for the many people unable to see the first run.



DETERGENTS, Technical Session G—A. J. Stirton presiding. First row: E. C. Beck, Eric Jungermann, A. J. Stirton, R. R. Mitchell, and E. A. Setzkorn. Second row: L. E. Weeks, J. W. McClutcheon, G. J. Stockburger, and M. T. Atwood.

magnesium cations was described by E. C. Beck, Armour and Company. Problems associated with the analysis of magnesium in the presence of sodium and potassium were outlined and the feasibility and reliability of the methods illustrated.

The physical requirements for detergent tablets were discussed by E. Jungermann, Armour and Company. Test procedures were given covering the testing for compressive strength, impact strength and rate of disintegration in water. The influence of processing and formulation variables on these characteristics was discussed by the speaker.

A paper by D. E. Herring, E. R. Howard Limited, England, was read by R. B. Mitchell, Lever Brothers Limited. He reported on the use of dimidium bromide and disulphine-blue as a mixed indicator in titrating anionic detergents with cationics to give contrasting colors between the water and chloroform and allow the end point to be traced in either phase.

A comparison of methods for following the biodegradation of detergents was presented by E. A. Setzkorn, Continental Oil Company. Surface tension measurements, colorimetric methods, Warburg manometric procedure and gas-liquid chromatography were used in studies relating to the bio-oxidation of straight and branched chain alkyl benzene sodium sulfonates and alkyl sulfates. The merits of the different analytical procedures were defined as they applied to the detergents under review.

A. J. Stirton, Eastern Regional Research Laboratory, reported on the effect of change in chemical structure on surface active properties of straight chain sodium alkane sulfonates of 12-18 carbon atoms and sodium 1-hydroxy, 2-alkane sulfonates with even carbon atoms from 12-18.

L. E. Weeks, Monsanto Chemical Company, proposed the use of ethylene glycol as the principal carrier solvent along with a small quantity of water for the rapid determination of free unsulfonated oil in neutralized sulfonated detergent. It is applicable to both the higher molecular weight sulfonates as well as dodecyl benzene sulfonate and gives rapid results making it especially suitable for process control as compared with steam distillation or extraction procedure.

Certain engineering factors pertaining to the sulfonation of fatty alcohols or their derivatives with chlorosulfonic acid were presented by J. W. McCutcheon, John McCutcheon, Inc. The construction and operation of a typical batch plant was discussed along with possible alternate approaches in the processing as necessitated by economic or engineering requirements.

G. J. Stockburger, Atlas Chemical Industries, dealt with the acid and base-catalyzed additions of ethylene oxide and propylene oxide to the isomeric butyl alcohols and higher aliphatic alcohols.

M. Attwood, Continental Oil Company, described the "Alfol" straight-chain alcohols and their commercial application. He visualizes phthalate ester plasticizers, alcohol sulfates and ethylene oxide adducts as offering important fields for these interesting alcohols. Many other applications were described including their use in the preparation of amine oxides, alkyl chlorides, mercaptans, nitriles and quaternary ammonium salts.



CHEMICAL REACTIONS, Technical Session H—S. F. Herb presiding. Seated: Abner Eisner, S. F. Herb, and J. Devine. Standing: A. N. Wrigley, J. P. Friedrich, M. J. Diamond, D. J. Moore, J. S. Showell, and D. C. Malins.

Chemical Reactions

The papers in this session dealt with the formation of new and unusual compounds from C-18 unsaturated fatty acids and other olefins.

J. P. Friedrich, Eastern Regional Research Laboratory, reported on the preparation of cyclic, unsaturated adducts of monoenes with conjugated dienoic and trienoic acids. Ethylene reacts readily with two or more conjugated double bonds, α -olefins react with difficulty and internal olefins are unreactive.

From unsaturated C-18 fatty acid methyl esters, D. J. Moore, Northern Regional Research Laboratory made azelaaldehydic acid acetals their vinyl esters and polymers of the esters. These are new compounds, and this is the first reported application of the vinyl ester synthesis to carboxylic acids containing an acetal function.

M. J. Diamond, Western Regional Research Laboratory, gave convenient methods for the preparation of phosphate and phosphite esters of various long-chain hydroxy fatty acids. Phosphorous esters are potentially useful in plasticizers, surface coatings, and hydraulic fluids.

Abner Eisner, Eastern Regional Research Laboratory, discussed the addition of phenols, aryl thiols, and aromatic acids to the double bond in oleic acid, cyclohexene and polyenoic acids using methane-sulfonic acid as catalyst solvent. Positional isomers are formed with, respectively, the aromatic ring, sulfur (thioether), and oxygen (ester) attached to the alkyl chain.

Good yields of fatty dichlorocyclopropanes were obtained by A. N. Wrigley, Eastern Regional Research Laboratory, from reactions of dichlorocarbene with unsaturated hydrocarbons and fatty esters of *cis* and *trans* configuration. The ring is opened and β -chlorallylic ethers (or alcohols) of longer chain length are produced on treatment with silver nitrate and alcohols (or water).

John S. Showell, Eastern Regional Research Laboratory, used perchloric acid to give a quick, one-step method, with high yield of γ -stearolactone from oleic acid. The effect of selected reaction variables was studied, and the intermediates, etc. formed were found to be isomers with changes in the position and the configuration of double bonds, polymers (C-O and C-C), other lactones, and addition products with water.

Donald C. Malins, U.S. Fish and Wildlife Service, stated that acetyl nitrate reacts quantitatively with methyl oleate forming isomeric nitro, acetoxy-nitro, and nitro-nitrate derivatives. These, in turn, are useful intermediates for

synthesizing the amino, amino-acetate, and amino-alcohol derivatives.

J. Devine, Unilever Research Laboratory, gave a report on the preparation of N-alkyl amides from α -olefins and alkyl nitriles by the Ritter reaction.

Biology and Nutrition

Hans Kaunitz, Department of Pathology, Columbia University, described further experiments with rats fed purified diets containing dihydroxystearate, long chain saturated glycerides from cocoanut oil and linoleic acid. Rats appeared normal after 1 year but showed a slight weight depression. Liver and adrenals were heavier but testicular fat pads were lighter than control rats of same weight, suggesting a decreased neutral fat deposition.

L. R. Dugan, Jr., Department of Food Science, Michigan State University, combined lipolysis, chromatographic separation and GLC analysis to elucidate the structure of high melting glycerides associated with the milk fat globule membrane. He showed that the GS₃ content was 71.2% and GU₂ negligible. Of the mixed glycerides, beta position was primarily occupied by a saturated fatty acid and while random distribution was observed with respect to S and U acids, the individual acids were not.

M. Kuchmak, Department of Food Science, Michigan State University, described the fractionation of phospholipids of hog muscle tissue on silicic acid columns. Identity and content of each fraction was accomplished by I. R.

spectrophotometry and for the cephalins, TLC analysis. Compared to gravimetric and phosphorus content methods, the above was found to be more precise and judged to be more accurate. Dr. Kuchmak is to be commended for his masterful handling of an embarrassing malfunction of the projector.

K. K. Carroll, Collip Medical Research Laboratory, University of Western Ontario, described the GLC of fat soluble vitamins using glass columns packed with 3% SE-30 or 3% QF-0065 on siliconized gas Chrom P with Barber Coleman Model 10 chromatograph and radium ionization detector. *Trans* vitamin A and the acetate showed evidence of alteration during chromatography. As polarity of the column increased, retention times decreased. During the discussion, the author stated that beta and gamma tocopherols came very close together and that cholesterol had to be separated prior to GLC analysis. Interest in vitamin analysis is due to the observation that 10% uric acid in diets produced sterility, decreased semen production and testes degeneration in male rats, and increased the vitamin E requirement.

J. C. Alexander, Procter and Gamble Co., showed evidence
(Continued on page 20)



BIOLOGY AND NUTRITION, Technical Session I—W. O. Lundberg presiding. Seated: Haus Kaunitz, W. O. Lundberg, Mary E. McKillican, and V. Mahadevan. Standing: L. G. Dugan, Jr., Myron Kuchmak, K. K. Carroll, B. E. Brown, and W. E. Scott.



BIOLOGY AND NUTRITION, Technical Session M—L. R. Dugan, Jr. presiding. Seated: L. R. Dugan, Jr., and Joyce L. Beare. Standing: H. J. Thomasson, J. G. Hill, B. M. Craig, and Herman Schleuk.

(Continued from page 17)

for the need and significance of careful diet handling for experimental animals. In diets containing polyunsaturated oils, daily preparation and feeding of diets as well as antioxidants and refrigeration, as opposed to the conventional weekly mixing and twice-weekly feeding, produced superior growth rate and higher efficiency of feed conversion. The results were highly significant after only four weeks. Considerable discussion was evoked, pro and con with respect to the criticism of other reported methods.

V. Mahadevan, The Hormel Institute, described experiments in which 8- to 12-week old weanling miniature pigs, after a 3-week low fat basal diet, were fed diets containing 0.5% cholesterol and either 15% tallow (saturated) or 15% safflower oil (unsaturated). Significant differences in blood coagulation were observed using various methods. Discussion resulted concerning the feasibility of describing diets saturated and unsaturated when tallow and safflower fats were different in many other respects.

B. E. Brown, Department of Food Chemistry, University of Toronto, was able to maintain soybeans in a mold-free condition for periods up to 9 weeks at relative humidities between 75% and 95%. Continuous increases in free fatty acid content were observed, and a decrease in the fat-soluble non-glyceride matter. There was no measurable change in the triglyceride composition. Rapid loss of viability was observed in the absence of mold.

Mary E. McKillican, Food Research Institute, Canadian Department of Agriculture, described the quantitative separation of "free" and "bound" endosperm lipids by silicic acid columns. TLC resolved the resultant lipid fractions into components which were analyzed for FA composition by GLC.

W. E. Scott, Eastern Regional Laboratory, showed that when ground *vernonia anthelmintica* seeds were incubated and aged in a water-saturated nitrogen atmosphere changes in oil composition occurred, as evidenced by solubility change, increase in FFA content and I.V., and decrease in epoxy content. The change appeared due to conversion of epoxyoleic acid to (+) - threo-12, 13-dihydroxyoleic acid. The isomer, (-) - threo-12, 13-dihydroxyoleic acid was also prepared by acetylation of the oil.

H. J. Thomasson, Unilever Research, delivered two papers. The first described methods employed in their investigation of essential fatty acids. The delivery was highlighted by the use of excellent color slides. The second dealt with efforts to link dietary fat with initiation and progression of arterial lesions in rabbits. Lesion development was classified as to degree of severity and well illustrated by colored slides. A diet containing 40% of total calorie content as coconut oil produced marked occurrence atherosclerosis, whereas only slight occurrence resulted from 40% total calorie as supplied by rice, and very little from 40% corn oil. The foregoing diets (soybean oil was used in lieu of corn oil) were supplied to rabbits after atherosclerosis was induced by 0.2% cholesterol diets. A very slight progression of the lesions was observed with the soybean oil diet, somewhat greater with the rice diet and marked progression with the coconut oil diet. Histological evidence of foam cell changes in the lesions was given.

Joyce Beare Rogers, Canadian Food and Drug Directorate, concluded that there is a minimum proportion of saturated acids for the most acceptable mixture of fatty acids in the diet of the rat, after observing the food intake, body weight and proportions of linoleic and arachidonic acids in the liver of rats fed diets containing rapeseed oil in which additional saturated fats were supplied by palm oil, bayberry tallow or olive oil.

B. M. Craig, Prairie Regional Laboratory, presented an "Engineer's approach to a nutritional problem," in the relationship between dietary fat and body fat of the animal. By a knowledge of the FA composition of the synthesized fat (lipogenesis) dietary fat and body fat, calculations were made and proportions of depot fat supplied by these two sources were given.

H. Schlenk, The Hormel Institute, supplied labelled methyloleate (C_{11}) orally to male, 28-day rats and found 10% of the radioactivity in carcass FA's and 6% in organ FA's. Analysis of methylesters of organ FA's as well as similar experiments using labelled heptadecanoate and 10-nonadecanoate verified that only part of the hexadecanoate specific activity arises from oleic acid.

J. G. Mill, Queens University, concluded from experiments with students fed fat-free diets or diets providing 45% of calories in the form of corn oil or butterfat that the total lipid level of the red blood cell remains constant under a wide variety of conditions including large variations in the lipid content of the surrounding medium and that the cell membrane integrity depends upon this constancy.

Hydrogenation

Using the methyl esters of refined soybean oil, H. S. Weber, Armour Research Foundation, described a method used for determining the selectivity of hydrogenation catalysts. A standardized laboratory hydrogenation followed by chromatographic analysis of the products was used to determine the relative reaction rates of the linolenic and linoleic fractions. The results were then expressed in terms of a selectivity index S_L . Classification based on the selectivity index S_L was found useful in the evaluation development of catalysts, and in particular catalysts which could be used for the selective hydrogenation of the linolenic acids of soybean oil for reducing undesirable flavor reversion in salad and cooking oil products.

Using the method described above, C. H. Riesz, Armour Research Foundation, presented data evaluating a variety of commercial hydrogenation catalysts. Platinum, palladium, and rhodium catalysts demonstrated high selectivities ($S_L = 2.4 - 2.7$) while nickel catalysts showed S_L values generally less than 2.0 with the unsupported Raney-type giving the best results. Reaction rates and *trans*-isomer formation were also determined, results summarized as follows:

| Catalyst | Rx rate | Isomers | Selectivity (S_L) |
|----------|---------|---------|-----------------------|
| Pd | High | Poor | Best |
| Pt | Low | Good | High |
| Rh | Low | Poor | High |
| Ni | High | Best | Good |

Supported nickel catalysts were also tested and found comparable in performance with commercial nickel catalysts.

Catalysts prepared with molecular sieve carriers demonstrated some interesting results. Nickel molecular sieve catalysts provided selectivities in the range of 2.0-2.3 S_L , low isomers (6%), and good reaction rates at relatively low temperatures (60-80C), while platinum and palladium molecular sieve catalysts showed lower selectivities as well as lower isomer formation compared to their commercial types.

It is postulated that the molecular sieve, by virtue of its particular porous structure (4 to 13 angstrom units pore diameters), surrounds and spaces the catalyst particles into a pattern which favors a more selective reaction.

H. J. Dutton, Northern Regional Research Laboratory, described an analog computer used to correlate experimental data in the study of hydrogenation reactions for the

(Continued on page 44)



HYDROGENATION, Technical Session J—H. J. Dutton presiding. Seated: C. D. Evans, S. S. Chang, H. J. Dutton, and C. H. Riesz. Standing: R. O. Feuge, D. E. Bitner, W. K. Rohwedder, H. S. Weber, and Ahmed Mabrouk.

purpose of determining specific rate constants. Digital computers which have previously been used for such work did not provide a convenient enough adjustment of rate constants in kinetic equations to get an acceptable fit for experimental data for reactions where "isolinoate" and the "oleate shunt" need to be considered.

The analog computer developed and used for this purpose provides essentially an electronic network analogous to the chemical reaction being studied. Specific reaction rates are determined by a simple adjustment of potentiometers, which are the analogs of the rate constants for which a required fit is required between calculated and experimental data. An oscilloscope used in conjunction with the computer further enhances the utility of this system by presenting composition vs. time or composition vs. degree of saturation in the form of curves plotted on rectangular coordinates which can easily be photographed for detailed study and record.

Isotopic effects during catalytic hydrogenation were discussed by E. D. Bitner, Northern Regional Research Laboratory. Methyl oleate was catalytically hydrogenated with mixtures of hydrogen, deuterium, and tritium gases for the purpose of determining if one or the other of the gases was preferentially absorbed; i.e., if there was any isotopic effect. The necessary laboratory equipment system used for this study included such items as a mass spectrometer analyzer and ion chamber.

Results of the work indicate that both tritium and deuterium may be used as tracers in the study of hydrogenation reactions without the complication of isotopic effects. A possible explanation for this behavior of tritium and deuterium is that their isotopic effects on solubility are small at the atmospheric pressure and 40C temperature conditions of these experiments.

W. K. Rohwedder, Northern Regional Research Laboratory, investigated the use of deuterium as a tracer in the study of hydrogenation reactions, to determine the extent

of exchange taking place between carbon bonded hydrogen and the deuterium tracer gas.

Methyl oleate was catalytically deuteriated to the stearate. The results analyzed by mass spectrometer showed that some molecules contained 3, 4, 5, and up to 11 atoms of deuterium indicating that extensive exchange takes place. A small number of saturated molecules were also found with no deuterium at all in their structure. As expected, the largest number of molecules contained 1 and 2 deuteriums. With methyl stearate catalytically exposed to deuterium gas, however, no exchange was found to have taken place.

A process for winterizing specially hydrogenated soybean oils for the purpose of removing the linolenic content and thereby improving the quality for salad oil use has been developed and is presently in commercial use.

C. D. Evans, Northern Regional Research Laboratory, described an investigation conducted for the purpose of determining optimum conditions for hydrogenating and winterizing to produce high yields of low linolenic content oils.

Samples of soybean oil were hydrogenated under selective and nonselective conditions to iodine values between 85 and 115 units. Oil of 7% linolenic content was found to yield

92% oil of 3.1% linolenic
85% oil of 2.0% linolenic
and 75% oil of 1.0% linolenic

Generally, 90% yields were had from 110 I.V. oils, and 70% yields from 90 I.V. oils, and also *trans*-isomers were found to increase linearly to about 30% at 90 I.V.

Solvent winterization was also conducted using acetone, which is a selective solvent for unsaturated molecules, and yields were generally increased by an average of 10% over ordinary winterization. Complete removal of linolenic fractions was effected by solvent winterization under conditions of 80% yield. For a similar yield by ordinary winterization approximately 1.5% linolenic fraction would remain in this liquid oil.

A. F. Mabrouk, Northern Regional Research Laboratory, noted that experimental results of the homogeneous catalytic hydrogenation of sodium sorbate indicate a high degree of selectivity at the 4,5 double bond of the sorbic acid. Hydrogenation using pentacyanocobaltate II catalyst reduced the sorbate to hexenoate. Gas liquid chromatography was used for analysis of the reaction products which were found to contain

80% 2-hexenoic acids
19% 3-hexenoic acids
1% 4-hexenoic acids
Trace sorbic acid

Caproic acid was not found in the reaction products either during prolonged hydrogenation or with increase in the amount of catalyst used.

T. L. Ward, Southern Regional Research Laboratory, reported an investigation to determine the optimum conditions for the hydrogenation of cottonseed oil in order to completely destroy cyclopropene groups and avoid simultaneously hydrogenating or isomerizing linoleic or oleic groups.

It was found that optimum conditions for one series of tests were 105C temperature, 20 psig. pressure and 0.1% nickel catalyst, causing the complete destruction of cyclopropene as indicated by a negative Halphen test. The oil showed a reduction of 2.8 units I.V. and a formation of only 1.7% *trans*-isomers. No conditions could be found which yielded an oil with a negative Halphen test and completely avoided the formation of *trans*-isomers. Filterability and yields of subsequently winterized oils were found to be unaffected by the hydrogenation treatment employed.

S. S. Chang discussed the isolation and analysis of volatile decomposition products formed during typical commercial hydrogenation of soybean oil, to determine the source of the characteristic hydrogenation odor of hardened oils. The quantity of volatiles recovered was found to be proportional to the peroxide values of the oils before hydrogenation. However oils heated in the absence of the

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OXIDATION AND STABILITY, Technical Session K—R. T. O'Connor presiding. Left to right: F. D. Hill, B. Weinberg, R. T. O'Connor, D. H. Saunders, and W. D. Pohle. (Not pictured: Ahmed Mabrouk.)

nickel catalyst showed a much lower quantity of volatile decomposition products and also lacked the characteristic odor.

The odor of the isolated volatiles disappeared when they were passed through a column packed with celite impregnated with 2,4-dinitrophenylhydrazine and phosphoric acid, due to the formation of hydrazones. The hydrazones were subsequently separated into five fractions by chromatographic means. These fractions were then hydrolyzed and it was found that the ketone and aldehyde fractions were the ones responsible for characteristic hydrogenation odors.

Oxidation and Stability

F. D. Hill, Department of Dairy and Food Industry, Iowa State University, in reporting on the identification of the oxidized-metallic and grassy flavor components of autoxidized milk fat, outlined the methods of isolation of these substances, which were aldehydes and ketones. Vinyl amyl ketone accounts for the metallic flavor; and for the grassy flavor, the authors isolated various components from the autoxidation of methyl linolenate. The 2,6-*trans-cis* nonadienal showed a grassy flavor. Sulfhydryl groups acted as antioxidants, and influenced the flavor development.

B. Weinberg, Canada Packers, Ltd., gave evidence that various types of methyl silicones promoted the oxidative breakdown of hydrogenated shortenings at temperatures close to 200C. Only when the fat was very stable did this breakdown not occur. Silicones also failed to protect the non-hydrogenated oils, but greatly reduced gum formation. The harmful action of silicones seemed to be largely independent of the added level. All silicones proved to be excellent defoamers.

D. H. Saunder, Eastern Regional Research Laboratory, showed that in the presence of histidine the rate of linoleate autoxidation with sodium dodecyl sulfate was about double that of potassium palmitate and sodium myristate, and all anionic emulsifiers tended to promote the pro-oxidative action of histidine. When non-ionic emulsifiers were applied, histidine showed only slight pro-oxidative activity. The anionic emulsifier decreased the rate of oxidation with increase in concentration, while the non-ionic emulsifier showed no such effect. The effect of certain salts on the rate of oxidative action of histidine depended on the type of emulsifier used. The strong pro-oxidative action of the ferric iron-histidine complex on methyl oleate and linoleate was demonstrated.

A. F. Mabrouk, American Meat Institute Foundation, University of Chicago, reported the investigation of the effect of various carbohydrates present in meat on the kinetics of autoxidation. It was found that with a decreasing number of carbon atoms in the carbohydrate, the rate of autoxidation of methyl linoleate increased, reaching a maximum in the case of glyceraldehyde. The configuration of the carbohydrate had but a slight effect on the rate of oxidation. When the primary alcohol group was oxidized to a carboxyl group, the effect was the opposite. The anhydro sugars also decreased the rate of autoxidation.

W. D. Pohle, Swift and Company, discussed the modification of the ASTM Oxygen Bomb Method by the addition of a copper catalyst. This reduces the time required. The new method has been approved by ASTM. It is faster than the AOM method, and provides good results.

General Session

Charles Pratt, Savannah State College, described the separation, by paper chromatography, and the identification of four of the flavonol glycosides in cottonseed oil and meal. These flavonoid compounds probably occur in the kernel and not in the hull.

W. A. Pons, Jr., Southern Regional Research Laboratory, reported on the removal of red and green pigments from cottonseed oil with different adsorbents and different bleaching times. The red pigments are removed by normal and sulphurous acid modified alumina which give oils comparable in taste, stability, and rate of hydrogenation etc., to oils bleached with conventional Fullers earth.

The isolation of high purity fatty acids was outlined by O. S. Privett, The Horuel Institute, using a two-stage process with various forms and combinations of adduct formation, crystallization, distillation, and chromatography.



GENERAL SESSION, Technical Session L—E. I. Birnbaum. Left to right: Charles Pratt, W. A. Pons, E. I. Birnbaum, Audrey T. Gros, B. de Vries, and F. B. Padley. (Not pictured: O. S. Privett.)

Polyunsaturated methyl esters are best separated on silver nitrate columns.

For the preparation of mono- and diglycerides, Audrey T. Gros, Southern Regional Research Laboratory, recommended direct esterification with *p*-toluenesulfonic acid catalyst and continuous removal of water by azeotropic distillation. Simple esterification predominates with some intra- and interesterification occurring.

B. de Vries discussed the separation of methyl esters, triglycerides and sterols according to the number, position, and configuration of the double bonds. This was accomplished with silica impregnated with silver nitrate in column and thin-layer chromatography.

F. B. Padley, Unilever Research Laboratory, analyzed natural fats and oils by enzyme hydrolysis and by thin-layer chromatography, and quantitative data from the latter was obtained by photodensitometry of charred silver nitrate impregnated silica layers, and the results agreed with those from enzyme hydrolysis.

Unit Processes

H. Niewiadomski, Technical University, Gdansk, Poland, reported it was not necessary to completely remove phosphorous and sulfur compounds from rapeseed oil to obtain satisfactory hydrogenation of the oil. From results obtained it was anticipated that in the case of sound, mature seed only degumming of the crude oil would be required prior to hydrogenation.

J. R. Reynolds, Saskatchewan Wheat Pool, described the effect of moisture and temperature during cooking of rapeseed, prior to extraction, on quality of the oil with particular emphasis on the hydrogenation characteristics. With no addition of moisture to the seed during cooking and at temperatures not exceeding 220F, oil was obtained which hydrogenated satisfactorily. This type of cook produced material readily handled in a filtration-extraction plant and the resulting meal showed no deleterious effects in feeding trials with mice.

G. C. Mustakas reported on improvements on the previously described method for obtaining essential oil, bland oil, and bland meal from mustard seed. A reduction in the conversion time for enzymatic release of the essential oil plus atmospheric steam stripping after conversion resulted in quantitative recovery of the essential oil.



UNIT PROCESSES, Technical Session N—H. G. Willsie presiding. Seated: R. S. Wayman, H. G. Willsie, and H. Niewiadomski. Standing: J. R. Reynolds, George Hoh, David Barlow, and G. C. Mustakas.

Shorter heating periods throughout the process gave a significant improvement in protein meal quality.

G. L. K. Hoh, E. I. du Pont de Nemours, presented data on the oxidation of tertiary amines with hydrogen peroxide in water and non-aqueous solvents and peroxy acids under various reaction conditions. The preferred system was hydrogen peroxide in water with addition of water during the reaction to prevent gelation. Yields and efficiencies were substantially improved by removal of impurities in the initial amine, which, if present, consumed a portion of the peroxide.

D. O. Barlow, E. I. du Pont de Nemours, discussed factors involved in the repeated use of ion exchange resin in epoxidation of oils. Wide clearance, recessed impeller, and centrifugal pumps were preferred for transfer of the resin oil slurry. Separation of the resin by centrifugation resulted in lower oil retention and less resin degradation than filtration. Methods for maintaining the desired concentration of water and acetic acid were outlined.

General Session

An analytical method for estimating abietic and neobietic acids was described by W. S. Vought, Jr. These acids can be separated from fatty acids by esterifying the latter with methanol and extracting the rosin soaps with water. Subsequently the rosin acids are formed and they are estimated by measuring their absorption at 241 and 250 millimicrons.

G. E. Graham reported that the curing of oxidized drying oils for use as linoleum binders can be reduced from 60 days to 4 days by the use of polymethylol phenols as accelerated curing agents. The compound which gave the best results was the tetramethylol of Bisphenol A.

W. S. Vought presented a paper by J. S. Heckles, R. H. Reiff, and F. H. Byers on the analysis of linoleum binders. The method was described in detail and followed more or less classical procedures of saponification of the binder and separation of fillers by filtration. Beta sitosterol is determined by precipitation as the 1-1-digtonide, and ligno-ceryl alcohol is separated on an alumina column. Fatty acids, rosin acids, glycerol, and pentaerythritol are also determined.

C. K. Lyons reported that the use of blown castor oil in place of castor oil in the polyol mixture to produce rigid urethane foams increased the compressive strength of the foams. The increase in strength is in proportion to the degree of oxidation.



GENERAL SESSION, Technical Session O—R. A. Burt presiding. Left to right: R. B. Sumantri, C. K. Lyan, R. A. Burt, B. M. Craig, A. P. Tullach, W. S. Vought, Jr., and G. E. Graham. (Not pictured: R. W. Poe.)

R. W. Poe showed that much better separation of mixtures of 1-olefins, from C₆ to C₁₈ in chain length, was obtained when the temperature of operation of the column was programmed from 75°C to 230°C at 9°C per minute than when the whole operation was performed at a uniform temperature. Results obtained by this means were more precise than those obtained by means of the mass spectrometer.

R. B. Sumantri presented experimental data of vapor pressures and vapor-liquid equilibrium conditions for caproic, caprylic, capric, lauric, and myristic acids and binary mixtures of adjacent members. It was shown that this data could be predicted by means of the Fenske-Myers and Supina graphical methods and extensions of them. These methods were suggested in cases where it is not possible to obtain data by experimental means.

B. M. Craig reported that a procedure formerly used to oxidize oleic acid and methyl oleate with tertiary butyl chromate or chromate-pyridine complex could be applied to the oxidation of the methyl esters of linoleic, ricinoleic, and linolenic acids to produce various keto compounds. However, methyl eleostearate and methyl acetyl ricinoleate were not affected by chromate-pyridine complex. In some cases where oxidation does take place it does so with explosive violence unless the temperature of the reaction mixture is carefully controlled.

A. P. Tullock described how hydroxy fatty acid glycosides of sophorose are produced by fermentation of long-chain hydrocarbons such as fatty acids or vegetable and animal fats, and how these hydroxy acids can be converted to dicarboxylic acids.

JAOCS Thanks Session Reporters

JAOCS wishes to express its sincere gratitude to those who volunteered to report on the Technical Sessions of this memorable Fall Meeting. The presentation of the foregoing technical review was made possible through the prompt and accurate efforts of the following individuals:

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- B. Weinberg, Canada Packers, Ltd.
- C. G. Youngs, Prairie Regional Laboratory
- W. N. B. Armstrong, Department of Chemistry, Ontario Research Foundation
- B.M. Craig, Prairie Regional Laboratory, Saskatoon

New Literature . . .

(Continued from page 32)

MIXING EQUIPMENT CO., INC., has published a 20 page brochure (Catalog B-540) giving helpful tips on such subjects as how to avoid vortexing, how to do two jobs with one mixer, how to dissolve light powders, and proper mixer mounting and positioning. (170 Mt. Read Blvd., P. O. Box 1370, Rochester 3, N.Y.)

SPENCER KELLOGG, Division of Tectron, Inc., now has available the 1962 Edition of their Technical Service Bulletin containing recently up-dated formulation, manufacturing, and test exposure data on their water-soluble linseed oil vehicle, Linaqua. (Technical Service Dept., 120 Delaware Ave., Buffalo 5, N.Y.)

WILKENS INSTRUMENT AND RESEARCH, INC., has announced their Gas Chromatography Catalog and Accessory Sheet that is available upon request. (P. O. Box 313, Walnut Creek, Calif.)