

New Orleans Meeting Marks Fiftieth Milestone

THE 50TH ANNIVERSARY MEETING of the American Oil Chemists' Society, held at the Roosevelt Hotel in New Orleans, La., April 20-22, 1959, featured an outstanding program, consisting of 59 technical papers presented by authors from 11 states and seven foreign countries. Highlighting the program were two symposia covering "Fifty Years of Fat and Oil Research in Selected Countries of the World" and "Fifty Years of Progress in the Chemistry and Technology of Fats and Oils." Topics of great interest to oil chemists, such as oil processing, plastic fats, isomerization during hydrogenation, analytical methods, gas-liquid chromatography, infrared spectroscopy, distribution of glycerides, chemical reactions, and polymers were discussed.

The technical papers were contributions of two universities, nine foreign and domestic government agencies, six research institutions, and 19 industrial laboratories. The excellent technical program was organized by Robert T. O'Connor, Southern Utilization Research and Development Division, U.S.D.A.; and presiding at the eight technical sessions were Leo A. Goldblatt, E. F. Pollard, Robert T. O'Connor, Edward A. Gastrock, James J. Spadaro, James Hugh Brawner, Harold J. Deobald, and Raiford Holmes, respectively.

Fifty Years of Fat and Oil Research in Selected Countries of the World

The research objectives of each of six foreign countries have been dictated by the immediate national fat and oil economy and, particularly in Germany, have changed abruptly at the beginning and end of each world war. In discussing the research in Britain, J. A. Lovern, Department of Scientific and Industrial Research, Food Investigation, Torry Research Station, Aberdeen, Scotland, emphasized four fields of fundamental research to which the British had made major contributions: a) the extensive systematic research by Hilditch and his school on the fatty acid and glyceride compositions of natural fats and oils; b) the contribution to our knowledge of polymorphism; c) the proof of structure of natural unsaturated fatty acids; and d) the development of methods of synthesizing even the more complex of these acids. Charles Paquot, National Center of Scientific Research, Bellevue, France, traced the advances in industrial fat and oil processing in France and the introduction of continuous processes, particularly in the manufacture of soap and detergents. He discussed in some detail the recently developed commercial process for preparing the polyamide, Rilsan, from 11-amino-undecanoic acid derived from castor oil.



FIFTY YEARS OF PROGRESS—Seated are H. J. Harwood (left), D. H. Wheeler, and H. E. Robinson; standing, R. T. O'Connor, chairman, D. S. Bolley, and R. A. Duncan.



FIFTY YEARS OF RESEARCH—Speakers at this symposium are (front row) A. R. Baldwin for H. P. Kaufmann, Germany; Charles Paquot, France; L. A. Goldblatt, chairman; J. M. Martinez-Moreno, Spain; (second row) J. A. Lovern, Scotland; H. J. Dutton, U.S.A.; H. W. Lemon, Canada; and D. H. S. Horn, South Africa.

As H. P. Kaufmann, University of Münster, Münster, Germany, was unable to attend, his paper was read by A. R. Baldwin. It enumerated the many important fundamental and technical advances resulting from the work of various German chemists and emphasized the sudden marked changes in the direction taken by research under stress of national emergencies. H. W. Lemon, Ontario Research Foundation, Toronto, Canada, reported that until recently most of the oil and fat research in Canada has been performed in government laboratories and has been practical in nature. The over-all objective is to lessen Canada's dependence on imports, especially edible oils, and to utilize to the best advantage domestic oils, such as soybean, sunflower, safflower, and rapeseed oils. Considerable attention has therefore been directed to the "reversion" problem.

According to D. H. S. Horn, South African Council for Scientific and Industrial Research, Pretoria, South Africa, organized research in that country began in 1939 with a systematic attack on the chemistry of pilehard and other marine oils. In the past 10 years considerable fundamental work has been done on the analysis and structure of a number of the less common fatty acids and other constituents of seed oils, cutin, cane wax, and wool wax. The paper by J. M. Martinez-Moreno, Instituto de la Grasa, Sevilla, Spain, read by H. J. Dutton, pointed out that only about 50% of the Spanish scientific papers on oils and fats have been reported in Chemical Abstracts. Since the olive is their most important source of oil and plays an important part in their economy, it is not surprising that most of their research has dealt with practical problems on the extraction and processing of olive oil.

Fifty Years of Progress in the Chemistry and Technology of Fats and Oils

Progress in the chemistry of fats and oils, applications of fatty acids, technology of edible fats and oils, utilization of inedible fats and oils, and the production of soaps was reviewed in the second symposium. D. H. Wheeler of General Mills reported that considerable advances have been made in fractional distillation; crystallization; isomerization; liquid extraction; autoxidation; epoxidation, oxidative cleavage; thermal polymerization; structure analyses; vapor phase, elution, development and paper chromatography; ultraviolet, infrared, and x-ray diffraction analyses; and chemical reactivity of the carboxyl group of fats and oils, or their fatty acids and derivatives. While soaps, greases, and candles were the chief uses of fats and

oils 50 years ago, H. J. Harwood of Armour and Company pointed out that now there are major outlets in rubber compounding, protective coatings, plasticizers, surface-active agents, lubricants, and synthetic fibers. The most important developments in the fats and oils industry since 1920 are as follows: a) catalytic hydrogenation of fatty acids and their esters between 1920 and 1930; b) fractional distillation (with corrosion-resistant alloy stills) of fatty acids and their esters between 1930 and 1940; c) continuous splitting of triglycerides to produce fatty acids and glycerol, solvent crystallizations, and preparation of nitrogen-containing derivatives of fatty acids between 1940 and 1950; d) oxidative cleavage and preparation of derivatives between 1950 and 1959. He indicated that more fundamental research on fats and oils is needed.

Herbert E. Robinson of Swift and Company stated that technology in the last 50 years has provided sources of edible fats other than butter and lard. Hydrogenation and deodorization of vegetable oils have produced palatable shortenings which have high concentrations of vitamin E and linoleic acid. Isomeric fats are satisfactorily utilized without deleterious effects. Heredity was cited as the most probable cause of arterial defects. High cholesterol blood levels merely indicate physical deterioration.

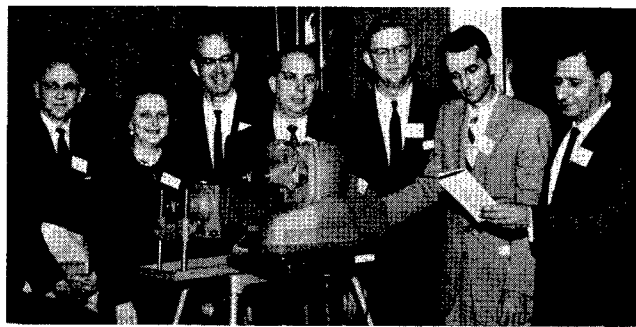


SESSION B—Facing E. F. Pollard, chairman, are J. K. Sikes, E. M. Meade, S. Kaloyreas.

Don Bolley of Baker Castor Oil Company, in delivering the paper of Francis Scofield of the National Paint, Varnish and Lacquer Association, outlined the history of the inedible fats and oils industry, principally the protective coatings industry, which began at about the same time the American Oil Chemists' Society was founded. Inedible oils were first used as the prime paint component; later they were used with phenolics. Still later they were used to prepare alkyds. Now they are being used to prepare oil alkyds for latex flat paints, which are competing with water-thinned, synthetic rubber-based latex paints. A resumé on the consumption of castor oil in the past 50 years was presented. Although it was first used for medical and cosmetic purposes, detergents and lubricants, its derivatives are widely used now as plasticizers, lubricants, synthetic resins, and organic intermediates. Castor oil was first dehydrated in 1928; now dehydrated castor oil is used chiefly in protective coatings. Recently it has been



MORE SPEAKERS—At the podium is E. A. Gastrock, chairman of Session D. In front row are H. M. Truax, I. A. MacDonald, and K. W. Becker; in back, J. Furman L., L. A. Baumann, W. A. Singleton, M. M. Mattikow, and C. M. Doyle.



SESSION E—From left to right these speakers are R. J. Vander Wal, Miss Wilma Guice, E. R. Cousins, R. J. Wrobel, W. O. Lundberg, W. A. Pons Jr., and the chairman, J. J. Spadaro.

used in the preparation of polyurethanes for many applications.

R. A. Duncan of the Procter and Gamble Company mentioned that there were only four different kinds of soap 50 years ago. Phase diagrams were not available, and there was a lack of scientific knowledge of soap-making techniques. In 1930 the batch-type process was replaced with a continuous soap-making process, thereby producing a better product much more quickly. Synthetic soaps have been prepared from sulfated alcohols, alkyl and aryl sulfonates, and now synthetics comprise 75% of soap sales.

Norwood Jatho Jr., U. S. Department of Labor, outlined American manpower needs through 1965. The growth in white-collar occupations, particularly in professional and technical fields, will continue. Lewis A. Bauman, Agricultural Marketing Service, U.S.D.A., reported results of a large-scale study of the keeping quality of raw linseed oils, which indicated that 77% of approximately 490 million pounds stored for periods up to five years met Federal Specifications and that the oils which did not meet specifications had a decreased value of only about 0.6¢ per pound on a 14¢ market. None of the oils stored in the warmer southern areas met specifications. Oils stored best in large, filled tanks, and top oil stored better than bottom oil. The results confirmed the suspected unreliability of the heated and chilled foots test as measures of nonoil constituents.

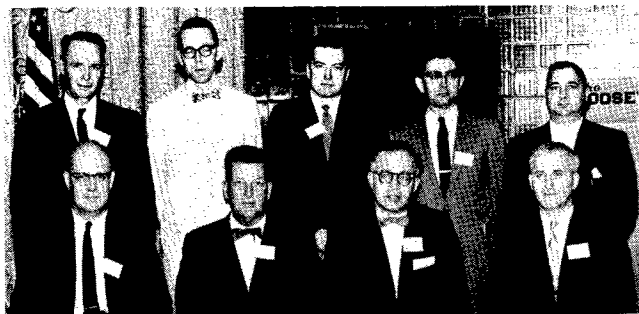
Gossypol

To elucidate the color fixation in crude cottonseed oil Walter A. Pons Jr., Southern Utilization Research and Development Division, presented data on a kinetic study of the rate of fixation of gossypol added to purified cottonseed oil under aerobic conditions. The reaction is dependent on temperature and is of the second order. Two molecules of gossypol undergo reaction simultaneously. In another report Mr. Pons described a process for recovery of gossypol from cottonseed gums obtained by water-washing of crude cottonseed oil. Recoveries were 41% of gossypol of 98% purity and 36% of gossypol of 99% purity. A phosphatide residue, low in gossypol content and suitable for feed purposes, was a by-product of the process.

Improved Processes and Equipment

A commercial installation of the filtration-extraction process for extraction of sunflower seed was described by J. Furman L. of Cia. Productura Nacional de Aceites S.A., Santiago, Chile. Extracted meal averaged 1.7% residual oil content at a hexane-to-meats ratio of 1.7. Data were presented to show that this process conducted on a bench-scale correlated closely with an industrial scale and enabled pre-establishment of optimum operating conditions prior to plant operation.

The "C.S.A." Process for refining fatty oils was described by Morris Mattikow of Refining Unincorporated, along with special mixers and proportioners that are employed. Plant data showed that the "C.S.A." Process is particularly applicable to oils of 1.8 to 8.0% free fatty acids, yielding lower losses than the modified soda ash process.



SESSION F—In the front row are T. F. Waters, J. H. Brawner, C. F. Smullin, S. F. Herb; in back, K. E. Holt, J. S. Showell, A. J. Fenton, L. R. Dugan Jr., A. Maggiolo.

Also described was the "Ammonia" Process with hermetic centrifuges for refining soybean and corn oil. Advantages of this method are removal of all gums, with minimum damage to phosphatides. Also discussed was a pilot-plant process for combining refining and water-washing of degummed oils in one step in a Podbielniak contactor. Collin M. Doyle of Podbielniak Inc. described a single machine used for degumming, soda ash and caustic refining, or re-refining-water washing of vegetable oils. Performance data were presented for a number of different vegetable oils. James K. Sikes, Plains Cooperative Oil Mill, demonstrated the desirability of integrating oil mill processing.

Kenneth W. Becker, Blaw-Knox Company, reviewed and compared the advantages and disadvantages of batch, semi-continuous, and continuous commercial systems for oil deodorization. Increasing demand for bland oils of highest color and flavor stability has forced accelerated trends towards use of initial de-aeration, lower absolute pressure, higher temperature, zones for retention and more intimate steam contact, addition of emulsifiers and antioxidants, and stainless steel equipment. Necessary for processing economy has brought about reduced operator attention, minimum down-time, lower entrainment losses, and lower utilities costs.

Working and Consistency of Plastic Fats

The design, construction, and operation of a pilot-plant size plasticizer for shortening and margarine was discussed by R. W. Bates of Armour and Company. Data were presented to show optimum operating conditions and variables affecting the air content and plasticity of the plasticized fat. Comparative penetration values and performance data were given. A continuous, worked-consistency meter for measuring changes of consistency of shortening during working was described by Dale V. Stingley, also of Armour and Company. Data were presented on a series of shortenings tested at 70°F. and 92°F., showing the relationship of Solids Index to worked consistency. Wilma A. Guice of the Southern Utilization Research and Development Division, discussed the influence of composition and tempering of confectionery fats as evaluated with a hardness tester developed at that Laboratory.

In two papers concerning the commercial formulation of shortenings with mono- and diglyceride emulsifiers, H. Mack Truax and Ira A. MacDonald, Atlas Powder Company, outlined the statistical design and reported results of experiments in which such factors as mono- and diglyceride contents of emulsifier, iodine values of the emulsifier, usage levels, and tempering of the chilled and plasticized shortenings were evaluated against performance criteria, such as water absorption, icing volume, and cake-baking characteristics.

Isomerization During Hydrogenation

E. R. Cousins, Southern Utilization Research and Development Division, reported that temperature had a more marked effect than other variables on the migration of double bonds during the hydrogenation of an oleoyl group. In general, for all variables, the migration of the double bond was always greater under those conditions that de-

creased the availability of hydrogen at the catalyst surface. The effects of variation in pressure and temperature on the formation of *trans*-isomers during hydrogenation of cottonseed and soybean oils were illustrated by Dale V. Stingley of Armour and Company.

William F. Barstow, Southwest Industries Inc., described the latest developments in equipment for the generation of inert gases (nitrogen, carbon dioxide, and combinations of the two) practically free of oxygen and combustible impurities and with moisture content as low as 5 p.p.m. and latest units for generation of nitrogen and hydrogen (or mixtures) from ammonia. Significant reductions have been realized in initial, operating, and maintenance costs. Also discussed were improvements in gas analyses and safety instruments for measuring and recording contents of oxygen, hydrogen, and combustibles.

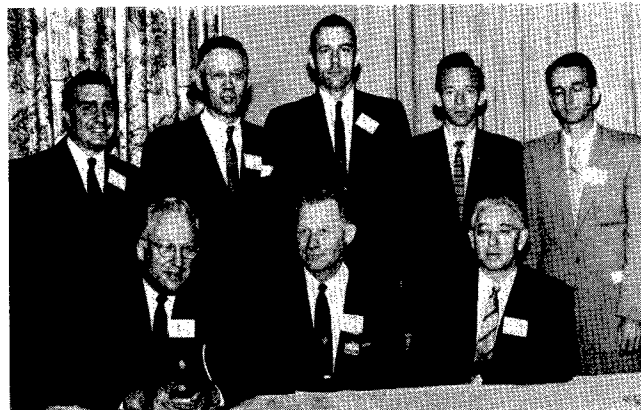
Fractionation of Fatty Acids

A novel process for low-temperature fractional solvent crystallization of fats and fatty acids was explained by W. A. Singleton, Chemetron Corporation. The process uses acetone or an acetone-like solvent with a crystal modifier. The combination yields nonviscous slurries at extremely low solvent ratios. Cardinal features are reduced equipment size and costs and operating costs lower than those of existing systems. Operating and cost data on several fats and tallow fatty acids were presented and discussed. E. M. Meade, The Meade Laboratory and Process Company, Unionville, Ontario, Canada, described a technique for the production of high oleic and high linoleic acid fractions from tall oil by the precipitation of oleic acid as acid soap from polar solvents. The process could provide an easily absorbed 200 million pounds a year of highly unsaturated acids and a challenging addition of 150 million pounds to the annual oleic acid production.

The theory of the washing process was covered by H. Lange of Henkel and Cie, GMBH, Düsseldorf, Germany.

Analytical Methods

A number of papers concerning analytical methods were presented. T. F. Waters described the purpose, rules, and scoring system of the between-laboratory analytical accuracy program used by the Procter and Gamble Company. Recommendations were made for the use of a common method based on this program for the several industry-wide competitive analytical programs now extant. K. E. Holt, Archer-Daniels-Midland Company, presented details on an empirical procedure for the determination of water-dispersible protein in soybean oil meals and flours. Data were given to show within-laboratory and between-laboratory precision of the method. A new constant for a pure compound, the specific molecular constant (in millimoles per gram), which is the reciprocal of the molecular weight, was proposed by John S. Showell of the Eastern Utilization Research and Development Division, U.S.D.A. This constant permits the reporting of all analytical data on a single uniform scale and permits interrelation of data



SESSION C—In front are H. J. Dutton, H. J. Deobald, chairman, C. Y. Hopkins; in back, F. E. Luddy, W. C. Ault, W. R. Noble, R. H. Purdy, and W. A. Pons Jr.

for complex mixtures without any further manipulations and knowledge of constants and definitions. L. R. Dugan, American Meat Institute Foundation, described a new method for the quantitative determination of butylated hydroxyanisole. The determination is based on the measurement of the absorption of the specific and sensitive reddish-purple color resulting from the reaction of BHA with diazotized sulfanilic acid in alkaline solution. The average recovery was 96.74%. A new rapid physicochemical test for the identification and quantitative detection of foreign fats used to adulterate milk fat was disclosed by Socrates A. Kaloyereas of Louisiana State University.

A. Maggiolo, The Welsbach Corporation, described an ozone number apparatus to determine the unsaturation values of fatty acids and oils. The results indicated 95% of the true olefinic value. A. J. Fenton, The Procter and Gamble Company, stated that a reliable analytical method has been developed for the determination of *cis* unsaturation in oils by measurement of a near infrared absorption band at 2.14 microns.

Progress in Chromatographic Separations

Charles F. Smullin, Atlas Powder Company, described the development of a silica gel column chromatographic procedure for the determination of free glycerol as well as mono-, di-, and triglycerides in monoglyceride mixtures. Rearrangement of the monoglycerides to form glycerol and diglycerides occurs with unsatisfactory lots of silica gel. S. F. Herb, Eastern Utilization Research and Development Division, stated that there was good agreement between the analyses of the methyl esters of vegetable fats and oils by gas-liquid chromatography and ultraviolet spectrophotometry in conjunction with iodine values. Francis E. Luddy, of the same laboratory, described the preparation and purification of fatty acid methyl esters—an important step in connection with gas-liquid chromatography. A chromatographic method for the separation of polyunsaturated fatty acid methyl esters was described by W. Stoffel, The Rockefeller Institute for Medical Research. A series of new polyunsaturated fatty acids were isolated from the body of the menhaden fish. According to H. J. Dutton, Northern Utilization Research and Development Division, U.S.D.A., acetonitrile-pentane-hexane comprised a system for the preparation of pure polyunsaturated fatty acid methyl esters by countercurrent distribution. An automatic recording refractometer simplified the operation.

J. M. Martinez-Moreno, Instituto de la Grasa, Sevilla, Spain, reported on the characteristics of membranes to protect emulsions of olive oil, including their semicrystalline phases and selective adsorption of heavy metal cations.

W. O. Lundberg, The Hormel Institute, described experiments designed to examine the influence of essential fatty acids on the *in vivo* conversion of palmitic to C_{18} acids. A definite role of essential fatty acids in fat biosynthesis was not established.

Esterification

J. D. Bradner, Atlas Powder Company, outlined a method for calculating the relative esterifiability of primary and secondary hydroxyl groups of glycerol. Esterification of the primary hydroxyls was favored. L. H. Dunlap of the Armstrong Cork Company stated that zinc acetates and salicylates were the most effective catalysts among those studied for the esterification of oleic acid with mono- and polyfunctional alcohols. The rate constant increased linearly with the concentration of the zinc ion.

R. J. Vander Wal, Armour and Company, proposed a method for calculating the proportions of glyceride types and isomeric forms in some C_{16} - C_{18} fats from the total saturated acyl groups and the percentage of saturated groups in symmetrical monoglycerides derived from the fat by pancreatic lipase hydrolysis.

C. Y. Hopkins, National Research Council, Ottawa, Canada, pointed out that epoxy acids are constituents in the glycerides of seed oils of the Malvaceae and related families and that possible larger amounts exist in other species.



SESSION II—At the left of the chairman, R. L. Holmes, are D. A. Yeadon, E. T. Roe, L. H. Dunlap, L. L. Gelb, and G. C. Mustakas; seated are H. P. Dupuy, H. M. Teeter, and W. G. Bickford.

Nuclear magnetic resonance was suggested as the most promising method of establishing their presence. R. H. Purdy, Pacific Vegetable Oil Company, traced the development of safflower as a commercial field crop and mentioned the uses for the oil and the meal.

Wilfred R. Noble, Eastern Utilization Research and Development Division, outlined a method for isolating hydroxy acids by low-temperature acidulation and fractionation of wool wax acids and their methyl esters. Partitioning the methyl esters between heptane and a mixture of ethanol, methanol, and water produced 25% hydroxy ester in the alcohol fraction and 70% nonhydroxy ester in the heptane fraction; the remaining 5% contained a hard, transparent, colored material. E. W. Maurer of the same laboratory described the preparation and properties of octadecylsulfuric acid from tallow.

Structure of Ricinelaidic Acid

Evidence was presented by W. G. Bickford, Southern Utilization Research and Development Division, to show that, in the β -hydroxy-ene system of ricinelaidic acid, the hydroxy group lies above the ethylene plane with the hydrogen oriented toward the double bond. In this confirmation the system is stabilized by intramolecular hydrogen bonding between the hydroxy group and the π electrons of the ethylene bond. H. P. Dupuy, also of the Laboratory, said that a small amount of water retarded the formation of polyacrylonitrile during the cyanoethylation of some ricinoleic acid derivatives and that any polyacrylonitrile formed was readily separated by pouring the cyanoethylated mixture into diethyl ether. The course of the cyanoethylation reaction and the purification of the product was followed by infrared analysis as the nitrile group in these compounds can be determined quantitatively.

D. A. Yeadon, Southern Utilization Research and Development Division, told of the preparation and testing of two series of urethane foams from castor oil. Foams of untreated castor oil made to contain increasing degrees of crosslinking were about equivalent in water adsorption; foams prepared from castor oil modified by elaidinization had improved water-resistance properties in addition to some increased strength properties. Improved shrinkage characteristics were observed for the series of urethane foams described.

E. T. Roe, Eastern Utilization Research and Development Division, showed that the acid-catalyzed addition of phenols and phenyl ethers to oleic acid proceeded through a carbonium ion mechanism. A strong acid cation exchange resin and 95% sulfuric acid were used as catalysts. No clean-cut advantage between condensing agents could be shown.

Soybean Vinyl Ether Copolymers

An improved process for the production of water-white copolymers of conjugated soybean vinyl ethers and isobutyl or ethyl vinyl ethers was described by G. C. Mustakas, Northern Utilization Research and Development Division. Turbo-agitation, a rapid quenching of the reaction with

absolute methanol, and the removal of the catalyst by precipitation with calcium hydroxide were described as being responsible for the improved products. The determination of the molecular weight distribution of several soybean vinyl ether copolymers was described by H. M. Teeter of the same Laboratory. An integral fractionation technique involving the precipitation of the polymer fractions from dilute benzene with increasing amounts of methanol was found superior to a cumulative volume technique. It was found that the distribution of the molecular weights of the polymers approximates the "most probable" distribution. L. L. Gelb, Senior Fellow of the National Renderers Association working at Eastern Utilization Research and Development Division, reported that the magnitude of some physical properties of copolymerized mixtures of epoxidized lard or soybean oil and a commercial diglycidyl ether of bisphenol A decreased uniformly as the weight percentage of epoxidized lard or soybean oil increased. He also pointed out that the addition of a tertiary amine to the epoxidized soybean oil would significantly decrease the curing time and increase the heat-distortion temperature. A cost saving is realized when epoxidized lard is used in place of soybean oil; up to 20% of an epoxidized lard may be used with little change in the properties of the resins.

AUDREY T. GROS *et al.*

New Orleans Draws 635

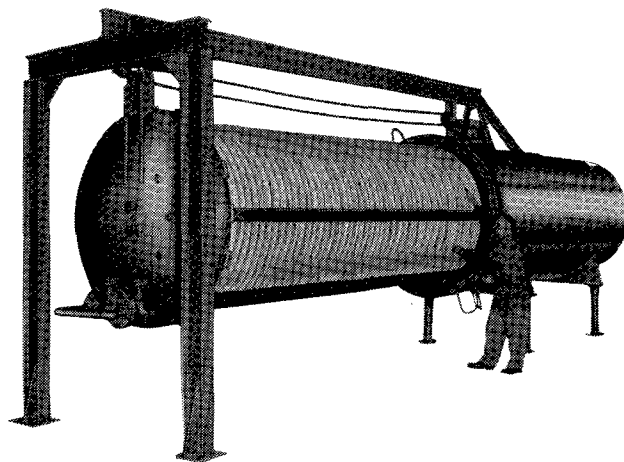
THE GOLDEN ANNIVERSARY meeting of the American Oil Chemists' Society in New Orleans was a smash success in every way, with the gold color as a keynote in printed program, banquet decorations, and so on. The Roosevelt even re-did its chairs in gilt for the banquet hall. Attendance reached a new high for a spring meeting: 635, of whom 412 were members, 68 nonmembers, 129 ladies, and 26 guests.

The total figure represented 29 different states and the District of Columbia as well as 13 foreign countries. Illinois led with 83, Louisiana ranked next with 77, and other sizable delegations were 33 from Ohio, 30 from New York, 42 from Texas, 24 from Tennessee, 23 from Minnesota, 21 from Pennsylvania, 15 from New Jersey, 12 from Missouri, and 11 from Kentucky. Foreign countries represented with one each were Argentina, Australia, Chile, Finland, France, Germany, Norway, Scotland, South Africa, Spain, and Turkey. Mexico sent two and Canada nine.

Sponsors of the meeting were the following: Archer-Daniels-Midland Company, Armour and Company, Distillation Products Industries, French Oil Mill Machinery Company, Lever Brothers Company, Procter and Gamble Company, Spencer Kellogg and Sons Inc., and Wesson Oil and Snowdrift Company.



NEW ORLEANS COMMITTEE—Assembled on the staircase at the Roosevelt are (front) C. L. Hoffpauir, H. L. E. Vix, Mrs. R. O. Feuge, R. T. O'Connor; (back), W. S. Singleton, T. H. Hopper, J. J. Ganuchau, F. C. Magne, and N. B. Knoepfler.



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