The Atlanta contingent was lined up for a group shot since the 1950 spring meeting will be in their hands: Stanly P. Stephenson Jr., E. C. Ainslie, Mrs. and Mr. G. Conner Henry, Mrs. and Mr. Dan Lee Henry, and Mrs. Ainslie

R. T. Doughtie Jr. *(center)*, chairman of Smalley subcommittee on oil seeds, chats with C. D. Lowry Jr. *(left)* of Chicago and Gail Erickson of Albany, Ga.

New Sources, Extraction Processes Discussed at New Orleans Meeting

THE proceedings at the 40th annual meeting and technical sessions of The American Oil Chemists' Society held at New Orleans May 10-12, 1949, reflect the character and spirit of this rapidly growing organization. The technical sessions demonstrated the high order of scientific ability of the membership, their steady exploration of new fields, and their readiness to exploit new tools as they appear. In their handling of controversial matters involving differences of scientific opinion, committee heads and members discussed the problems in such a spirit of mutual respect and understanding as to lead the observer to suspect that the code of ethics of the society is unnecessary.

Most of the technical papers can be roughly classified in three categories: 1. papers dealing with the investigation of new sources of fats, oils, and fatty acids, 2. those dealing with solvent extraction processes, and 3. those concerned with synthesis and reactions.

New Sources

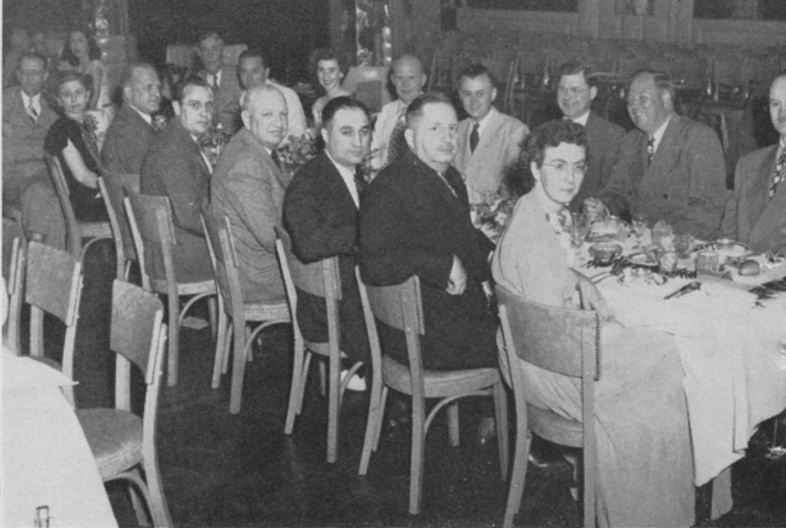
The Southern Regional Research Laboratory reported further progress in developing the technology of rice bran oil which constitutes a potential source of 30 to 50 million pounds for the U.S.A. At the present time one large and two smaller plants are extracting the oil in California, and one large plant is being built in Houston, Tex. Only a small amount of the oil is being recovered at the present time.

Loeb, Morris, and Dollear showed how drying and storing bran at a low moisture content was the most effective means in minimizing the development of free fatty acids, which is a major difficulty in the processing of rice bran oil. Both "converted" and white milled rice bran were studied. Swift, Fore, and Dollear reported the processing characteristics of the hexane extracted oil which constituted 13.8 to 17% of the bran. Eight crude oils with free fatty acids ranging from 2.0 to 4.9% gave losses of 12.0 to 20.5% by the laboratory cup method for hydraulic cottonseed oil. (Wesson losses on 5 crude oils ranged from 7.4 to 10.1%.) The colors of the refined oils varied from 35 yellow, 4.5 red to 70 yellow, 9.5 red, and

Inquiring about the total registration for the 40th annual meeting are *(left to right)* the new Journal editor, A. R. Baldwin, and two members of the Journal Committee, A. E. Bailey and T. L. Rettger, with the two reporters for the meeting: R. J. Vander Wal and H. T. Spannuth of Chicago

Gathered together to congratulate the newly elected vice president, J. R. Mays Jr. *(extreme right)*, are his colleagues from various independent laboratories: *seated*—E. H. Tenent, C. L. Manning, and E. R. Hahn; standing—G. Conner Henry, O. E. Wilkins, Dan Lee Henry, and G. W. Agee

A nicely balanced table group at the banquet comprises Mr. and Mrs. M. Gray Boulware, *beginning at the left;* W. D. Hutchins and Miss Claudine Hutchins, Mr. and Mrs. E. R. Cousins, Mr. and Mrs. Salvatore Milazzo, W. Reid Stryker, Mrs. and Mr. S. C. Murphy, Mrs. and Mr. John B. Geiger, Mrs. and Mr. Porter Williams, and Mrs. J. J. Ganucheau



Something happened to the paper on which the names for people at this table were written so it has not been possible to identify all of them. Among them *(foreground)* are Mrs. G. A. Crapple, A. A. Robinson, Crapple, J. H. Kirby in order. Across the table toward the left end are Mrs. and Mr. E. R. Hahn

after the official A.O.C.S. bleach, from 15 yellow, 1.5 red to 35 yellow, 3.2 red.

The A.O.M. keeping quality, after deodorization, varied from 17 to 29 hours, after hydrogenation from their original value of 103 to an iodine number of 66, and after deodorization the A.O.M. varied from 225 to 460 hours. This is about twice the value secured by a similar treatment for cottonseed oil.

The C_{22} and C_{24} dicarboxylic acids were shown by Schuette and Christensen to be present to the extent of 2.1 and 3.2%, respectively, in Japan wax which is the fruit-coat fat of a sumac, *Rhus succedena L.*

Hussain and Dollear characterized the oil from the kernels of lalob fruit, *Balanities aegyptiaca,* from Sudan, Africa. An average of 46% of the oil was found in the kernel which constitutes 9 to 10% of the whole fruit. By hexane extraction, a light colored oil was secured which showed 38.4% linoleic, 35.2% oleic, and 21.4% saturated acids by the iodine-thiocyanogen method. The ultra-violet absorption method showed only a trace of linolenic acid.

A pilot plant process using isopropanol as a solvent for rice bran was described by Meinke, Holland, and Harris. In this process 5 to 6 parts of solvent to bran are used to recover, in the syrup phase, 63% of the vitamins, which include biotin, folic acid, riboflavin, pantothenic acid, pyridoxine, thiamine, niacin, and inositol. The proposed process recovers an edible grade of vitamins and, at the same time, reduces the tendency of the bran to go rancid while recovering the oil.

Extraction Processes

Harris, Hayward, and Lamb of Texas A. & M. secured from cottonseed, on a pilot plant scale, an isopropyl alcohol miscella containing considerable quantities of fatty acids, phospholipids, raffinose, gossypol, and related color bodies admixed with the oil. Phase diagram studies were made choosing refined oil and oleic acid as key components with isopropanol-water and isopropanol-water-hexane.

In another study on hexane extraction Spadaro presented pilot plant data with slides in color for fractionation of cottonseed so as to remove 99% of

Another table of guests at the Society banquet comprises the following roster: *(left to right)* Enno Schoenhardt, Wilma A. Guice, Irving Rusoff and Mrs. Rusoff, Mrs. M. L. Karon and Mr. Karon, F. G. Dollear, Fred G. T. Menezes, S. A. Hussain, Mary Bruier, Marcel D. Webber, Mrs. Webber, Leah Castillon, Pat Von Der Haar, and E. C. Volkert

Distinguished by the Smalley cup is the table where sits R. W. Bates, Mrs. Bates, and R. T. Doughtie Jr. *on the left.* With them, in order, are the following: Lawrence Hodges, Mrs. C. K. Wiesman, Mr. Wiesman, E. F. Binkerd, R. J. Buswell, R. J. Vander Wal, Mrs. Vander Wal, Mrs. Russell Haire, Elizabeth Haire, W. Doss Lumpkin, Peggy Haire, and Russell Haire

the pigment glands. The process included material preparation, disintegration of the cottonseed flakes (either defatted or undefatted) in a solvent slurry followed by settling or centrifuging at 62 times gravity to separate the meal and pigment glands. The fines from the miscella are separated by pressure filtration or centrifuging at 1,500 times gravity.

Sweeney and Arnold described the 25-ton per day commercial plant which uses trichloroethane to extract soybean oil as developed at Iowa State College. The extractor is the lower section of a continuous loop of circular tubing through which the flakes are moved by a special chain conveyor, countercurrent to the solvent flow. The extracted flakes are carried into the upper run of the loop, which is steam-jacketed, to preheat them before they are dropped into steam-jacketed drying tubes. The miscella is concentrated in a climbing film evaporator with final stripping in a packed column. The solvent in the oil can be reduced to less than 50 p.p.m.

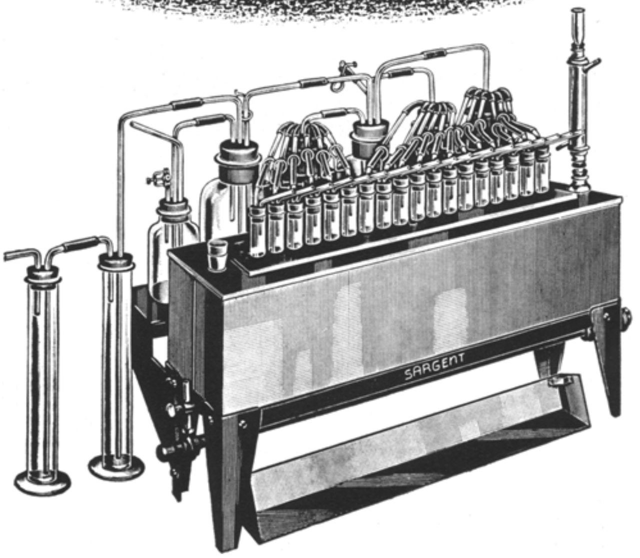
Prior difficulty encountered in the industry in hydrogenating trichloroethane extracted oil was directly attributable to faulty operation. As high as 1 to 2% of the solvent was noted in some oil shipments. Adequate methods are available for measuring the quantity of trichloroethylene below 10 p.p.m. No fire hazard is associated with the process. It was reported that no difficulty from toxicity of the solvent has been encountered in practice and that adequate controls are provided.

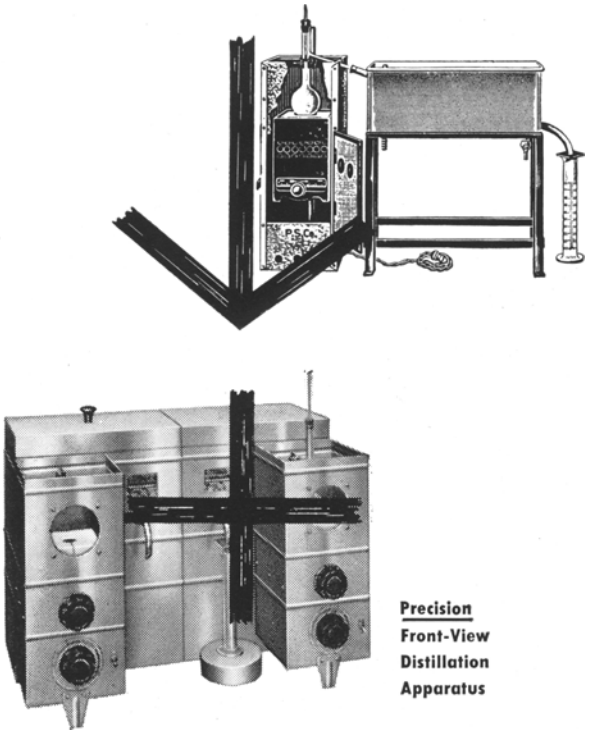
Norris, Mattil, and Lehman showed the retarding effect of trichloroethylene on hydrogenation which is associated with the presence of trichloroethylene. No residual effect on the oil is noted if the solvent is removed. Quantitative studies on adding relatively high dosages of from .01 to 0.1% trichloroethylene to refined and bleached oils showed catalyst poisoning in laboratory, pilot plant, and plant equipment. The solvent is lost through vaporization during refining, water washing, and bleaching. The amount lost, which may be 80% or more, depends upon actual operating conditions as these affect the degree of volatilization of the solvent. By concentrating the trichloroethylene distillation with kerosene and using infra-red absorption, 1 p.p.m. of trichloroethylene was detected.

Synthesis and Reactions

Another paper by Bailey dealt with the kinetics of hydrogenation. The analytical data of Bailey and Fisher, Oil & Soap *23*, 14-18, 1946, was re-examined with appropriate equations derived from consecutive monomolecular reactions. In the hydrogenation of linseed oil the comparative rates for different acids were found to be for linolenic, linoleic, isolinoleic, and oleic, 100, 40, 5, and 1.3, respectively, for "selective" conditions; and 100, 60, 20, and 8, respectively, for "non-selective." During non-selective hydrogenation approximately 24% of the linolenic acid went to linoleic, 65% to isolinoleic, and 11% directly to oleic, while with selective hydrogenation the corresponding percentages were none to linoleic, 54% to isolinoleic, and 46% to oleic.

Gros and Feuge showed, under conditions studied, that molecular portions of methyl and ethyl ester and glycerol reach an equilibrium at 70% triglyceride. By using an excess of glycerine and stripping with steam, approximately 90% triglycerides were secured.





Complete reaction was not secured between methyl and ethyl esters when reacted with triacetin and sodium methoxide as a catalyst. Reactions were studied at 200°C. and 50 mm. of mercury pressure. A hot water condenser was used.

Teeter, Kini, Myren, and Cowan prepared condensation products of one mole methyl linoleate and two moles of itaconic anhydride, and with 2 moles of citraconic anhydride. A reaction product of one mole of ester to one of the anhydrides was separated by molecular distillation from the non-volatile polymeric adducts. After heating 1 to 3½ hours at 170°C. with ethylenediamine, the nonvolatile polymers gave gels. Using 0 to 5% dimethyl aniline, methyl linoleate was condensed with equimolecular amounts of maleic anhydride. The higher amount gave a larger portion of a solid heteropolymer and a sharply decreased yield of the liquid adduct.

Teeter and Jackson studied the reaction products of equimolecular portions of methyl oleate and butyl hypochlorite and their behavior toward alcoholic silver nitrate and thermal dehydrohalogenation. Similar studies were made with methyl 9,10-dichlorostearate and with the chlorides from methyl ricinoleate and methyl 12-hydroxystearate. The results showed the presence of allylic chloride, a non-allylic monochloride, a saturated dichloride and unchanged methyl oleate.

Miscellaneous

Papers not classified in the three broad groups included a very interesting one by Dutton, Lancaster, and Brekke on the structure of soybean glycerides. It bears directly upon the recent discussions along these lines recorded in The Journal of the American Oil Chemists' Society.

Fractionation of soybean oil was studied in the light of calculated triglyceride structures of a "typical" soybean oil consisting of 14% saturated, 9% linolenic, 33% oleic, and 44% linoleic acids when the acids were assumed to be randomly or evenly distributed. Fractionations were made by adsorption, liquid-liquid extraction, and crystallization. The significant conclusion was that the efficiency of present fractionations does not equal the limit set by the actual distribution. Serious doubt is thrown on the theory of random distribution.

Samples of textile materials from peanut protein were distributed with the presentation of the paper on the industrial utilization of peanut protein by Arthur. He described procedures for evaluating peanut protein for industrial uses such as adhesive paper coatings, textiles, water-churned paints, and synthetic fibers through various physical tests. In extracted meals, unlike the expeller meals, more than 90% of the protein can be secured in a non-denatured state which makes the protein more valuable for the above uses.

Laboratory apparatus of one- and/or two-liter capacities for pressure reactions up to 1,000 p.s.i. were described by Binkerd, Vander Wal, Van Akkeren, and Hoppe. Provision has been made for the admission of gas, removal of liquid or gaseous samples during hydrogenation, and agitation and cooling by means of an internal coil.

Although carbon is commonly used to absorb biologically active compounds, it possesses catalytic activity and also reducing ability. Data were presented by Hassler showing this effect of adsorption on treated oils as revealed by spectrophotometric measurement, and also its reduction of ferric chloride.

That spectrophotometry is an increasingly useful tool in the chemistry of fats and oils is shown by its frequent mention in the reports. One instance is the paper by O'Connor, Field, Jefferson, and Dollear showing its utilization in a study of the processing of vegetable oils. Another is in the work reported by Castillon, O'Connor, Hall, and Miller on cottonseed pigments.

O'Connor, Field, Jefferson, and Dollear showed the effect of refining, bleaching, and deodorization on the visible and ultra-violet spectra of cottonseed, soybean, peanut, sesame, okra seed, and rice bran oils. It was shown that the absorption in some oils, which has frequently been attributed to chlorophyll, is neither the alpha nor beta form but another similar pigment.

Pons and Guthrie reported a method for determining free gossypol in cottonseed meals. Extraction by shaking with 70% aqueous acetone for one hour was followed by filtration and colorimetric analysis with p-anisidine. This method compares favorably with that of Smith.

Castillon, O'Connor, Hall, and Miller showed the effect of storage at 38, 77, and 85°F. on three purebred varieties of cottonseed, *G. hirsutum,* grown under similar environmental conditions. The significant change was the decrease in gossypol and the increase in gossypurpurin, which was greatest for the highest storage temperature. The gossypurpurin made the test with antimony trichloride non-specific for gossypol which was extracted by alkali from the non-acid yellow pigment.

Schuette presented an analysis of condelilla wax.

H. T. SPANNUTH
R. J. VANDER WAL

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National Bureau of Standards

Publications issued by the Bureau of Standards and now available at 10c each are entitled "Kinetic Study of the Reaction of Carbon Adsorbents with Oxygen," William V. Loebenstein, RP1948; "Treatment of Leather with Synthetic Resins," Rene Oehler and Timothy J. Kilduff, RP1951; "Rate of Shrinkage of Tendon Collagen Heat, Entropy, and Free Energy of Activation of the Shrinkage of Untreated Tendon; Effect of Acid, Salt, Pickle, and Tannage of the Activation of Tendon Collagen," Charles E. Weir, RP1947.

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The only apparatus made exclusively for determining evaporation rates has been developed by PRECISION SCIENTIFIC COMPANY, Chicago, Ill. The "Precision"-Shell Evaporometer can be used most advantageously by researchers in organic chemistry, the petroleum industry, and the paint and varnish industry. Another apparatus developed by Precision Scientific Company is the "Precision" Cryoscopic Molecular Weight Apparatus, used for determining the molecular weight of liquids or solids by the freezing-point method and can also be used for rapidly determining the freezing-point of liquids or for determining cooling curves of liquids.