REPORT OF COMMITTEE FOR THE STUDY OF PAPER AND INKS USED IN SOAP WRAPPERS 1935

THE report of this committee for 1935 will necessarily be brief, since it covers only a plan of proposed collaborative work. Unavoidable circumstances have prevented the chairman from initiating the work sooner.

In conformity with the recommendations included in last year's report of this committee as published in OIL AND SOAP, Vol. 12, pp. 48-51 (March, 1935), work on soap wrappers is planned as follows:

1. Tests on several samples of plain unprinted paper.
    - (a) Spot test, with caustic alkali of five different strengths.
    - (b) Extraction method for alkali resistance.
    - (c) Soap contact tests on these same unprinted papers with three types of white soap, viz.: white floating soap, filled white laundry soap, and uncolored milled toilet soap.
2. Tests on printed soap wrappers. As suggested in the 1934 report of this committee, it would be highly desirable to subject to collaborative tests by members of this committee specimens of printed soap wrappers known by factory experience to give satisfactory or unsatisfactory results in use. Testing of such wrappers by the alkali spot test and by different soap contact tests according to detailed methods suggested by Mr. Sheely, Mr. King, and Mr. Bennett, as published in the 1934 report, should furnish valuable information.

It is hoped that the results of the collaborative work as outlined may permit this committee to recommend to the next meeting of the Society a tentative method, or methods, for testing soap wrappers.

The 1935 membership of the Soap Wrapper Committee is as follows:

Allen Abrams, H. C. Bennett, W. H. Burkhart, T. L. Crossley, J. E. Doherty, W. H. Graebner, M. H. Ittner, A. E. King, Frank Libby, N. J. Neubauer, A. S. Richardson, M. L. Sheely, L. F. Hoyt, chairman.

ABSTRACTS

Oils and Fats

Edited by
W. F. BOLLENS and M. M. PISKUR

Progress in treatment of vegetable fat source material (1930-1935). R. Strauss. *Fettchem. Umschau* 42, 219-29 (1935). Review of patents on extn. of vegetable oils.

Mechanism of autoxidation of ether. A. Reicke and R. Muster. *Angew. Chem.* 49, 101-3 (1936). The mechanism of peroxide formation and oxidation in ethers is reviewed. It is suggested that a study of the autoxidation of ether may lead to obtaining more exact information on the rancidification of fats and oils.

Lecithin and hydroquinone as antioxidants for vitamin A. H. N. Holmes, R. E. Corbet and E. R. Hartzler. *Ind. & Eng. Chem.* 28, 133-5 (1936). Hydroquinone and lecithin were studied as antioxidants for vitamin A in halibut liver and in cod liver oils at room temperatures and higher. Each affords protection for the vitamin, the degree varying with the concn. of the antioxidant. The combination of the two, however, affords a remarkable protection which is much greater than would be expected from additive effects.

The determination of the acetyl number of fats. II. Carrying out the acetylation. K. Täufel, M. de Mingo and H. Thaler. *Fettchem. Umschau* 42, 141-4 (1935); C. A. 30, 887. Three acetyl no. methods were compared. The Franchimont gave the most nearly theoretical results, while the "Wizoff" and Verley and Bolsing were inferior for hexa-, tetra- and di-hydroxystearic acid. All methods gave concordant results with castor oil and grapeseed oil.

The spoiling of fats and biochemical reactions involving fats. K. Täufel. *Fettchem. Umschau* 42, 164-8 (1935).—This review covers a no. of recent developments which reveal possible relationships between the oxidation of unsatd. fatty substances and the β-oxidation of fats *in vitro* on the one hand and the mechanism of fat metabolisms and similar biochem. reactions on the other. Glyceride hydrolysis is also discussed both as a purely chem. and as a biochem. process. Over 30 references. (C. A. 30, 888.)

New method for determining acid number of oils. Fedchenko and Kashin. *Masloboĭno Zhirovoe Delo* 11, 498 (1935).—The method makes it possible to obtain accurate results without the use of alc. and Et_2O. Weigh a sample of oil in a flask (15-18 cm. high, 2 cm. in diam., neck bore 1 cm. in diam.), add 15-20 cc. of 10% NaCl in H_2O and titrate with 0.1 *N* KOH in the presence of phenolphthalein to a faint rose that does not disappear with energetic shaking for 1 min. The method gave good results with black cottonseed oil. (C. A. 30, 892.)

A note on the polyethenoid acids of the n-Octadecane (C_{18}) series present in aquatic animal oils. T. G. Green and T. P. Hilditch. J. Soc. Chem. Ind. 55, 4-8T (1936). The investigation was made for the purpose of obtaining quant. data on the relative amts. of polyethenoid C_{18} acids present in marine animal oils.

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The unsatd. C_{18} acids of whale oil consist of about 90% oleic acid and isomers of same, 3% tetraethenoid and the remaining 7% may contain octadecadienoic acids, but only small amts. of ordinary linoleic acid; linolenic acid is absent. For cod liver oil the results were 70% oleic and isomers of same, 10% tetraethenoid stearidonic acid. The remaining 20% may be di- or tri-ethenoid in character, but linolenic is not present and ordinary linoleic, if present, is in extremely small amts. The unsatd. C_{18} acids from fat of grass fed carp contained 11 and 6% resp., of linolenic and linoleic acids.

Softening of linseed oil films. C. H. Rose and D. S. Bolley. Ind. & Eng. Chem. 28, 115-8 (1936). Linseed oil films which are dried and aged under a sunlamp at 78° F. and 50% relative humidity exhibit an aftersoftening action. When such films are extracted with acetone, maximum dryness is attained. On aging, the oils soften and the solid phase changes so that the film reverts to a condition of almost complete solubility in acetone, which indicates that syneresis is not the principal cause of the softening of linseed oil films. The less saturated linseed oils give dried films which soften less and are more insoluble in acetone. This shows that the composition of an oil affects not only the rate of drying but also the colloidal changes in the dried oil film. The presence of drier not only increases the drying rate but also delays the after-softening and decreases the degree of after-softening; likewise the acetone solubility is delayed and somewhat decreased. Linseed oil, when pigmented with white lead, gradually attains an acetone insolubility of 60-70% which does not decrease on further aging.

Polymerization of fatty oils. IX. The hydrogenation of polymerized ethyl linolenate. Alph. Steger and J. van Loon. *Rec. trav. chim.* 54, 750-6 (1935); cf. *C. A.* 29, 5293^7.—Et linolenate was prepd. and then polymerized until further treatment produced no change. High-vacuum distn. gave the following fractions: (1) up to 95°, 1.4; (2) 95-100°, 30.7; (3) 100-180°, 7.2; (4) 180-200°, 21.8 and (5) residue 38.9%. Fraction 1 was composed of decompn. products. Fraction 2 did not contain polymers, yet it could not be hydrogenated to Et stearate. The satd. acid derived from fraction 2 after hydrogenation is attacked by Bertram's $KMnO_4$ soln. This indicates the acid is not a fat acid. From the hydrogenated product of fraction 2 an acid whose formula corresponded to $C_{18}H_{34}O_2$ was sepd. by the Twitchell Pb salt method. These properties together with phys. characteristics indicate a compd. of cyclic structure. The fractions 4 and 5 contained the polymers. These could not be hydrogenated to an I no. of O. Oxidation of the hydrogenated product of these fractions according to the Bertram method yielded a residue of cyclic compds. Conclusion: By polymerization of Et linolenate at 300° in a current of CO_2 2 reactions take place: (*a*) A polymerization and (*b*) a cyclization of linolenic acid into a nonpolymerizable compd. which after hydrogenation corresponds to the formula $C_{18}H_{34}O_2$. Both reactions occur simultaneously until no linolenate remains. This explains why linolenic acid cannot be completely converted to polymers. **X. The polymerization of ethyl linolenate.** *Ibid.* 756-9.—Et linolenate was polymerized at 300° for 6, 10 and 14 hrs. I no. (Wijs and Br methods), thiocyanogen no. and $n^{20}/_D$ are reported on each product. The Wijs I no. with varying periods of contact, thiocyanogen no. and $n^{20}/_D$ are reported on the high-vacuum distillate to 100° and the residue of each product. Substitution of I was evident in the residues and was greater with longer polymerization time; while in the distillate substitution of I was not detectable. Conclusion: High polymers and low mol.-wt. isomers of Et linolenate are formed on polymerization. The high polymers increase with the time of heating. (C. A. 30, 890.)

Hydration of sunflower oil. M. Bauman, I. Grabovskiĭ, F. Vishnepol'skaya and G. Gushchin. *Masloboĭno Zhirovoe Delo* 11, 477-9 (1935).—The best results in clarifying sunflower oil were obtained by stirring the oil, pressed or non-pressed, at 70° first with 1% of 3% NH_4Cl for 20 min. and then with 1% of 0.25% HCl, followed by salting out with 2.5% NaCl at 68°, settling for 16 hrs. and filtering at 40°. The product after 8-10 months of storage formed only 1-1.5% of white sediment and showed no change in acidity. (C. A. 30, 890.)

Influence of the nature of a carrier on catalyst activity. E. Botkovskaya and P. Artamonov. *Masloboĭno Zhirovoe Delo* 11, 468-51 (1935). Expts. in comparative hydrogenation of vegetable oils in the presence of Ni catalyst pptd. on silica gel and on kieselguhr and tripoli of various origins, with and without preliminary treatment with H_2SO_4 and Na_2CO_3, disclosed that contrary to the general theory the best carriers are those contg. a max. of Al and Fe oxides and a min. of SiO_2. Further evidence was furnished by the use of new carriers, such as clays rich in Al_2O_3 and Fe_2O_3. Thus by the use of a clay of the compn. SiO_2 59.76, TiO_2 0.51, Al_2O_3 18.5, Fe_2O_3 8.1, CaO 0.79, MgO 2.66, K_2O 5.08, Na_2O 0.16, P_2O_5 0.16 and SO_3 0.19%, a fat mixt., m. 61° was obtained, i.e., a considerably greater activity of Ni than when pptd. on kieselguhr. The practical interpretation of the results is that the catalytic activity of Ni decreases with the greater contents of SiO_2, sol. in Na_2CO_3, in the carrier and increases with its increased soly. in H_2SO_4. The presence of P in a carrier has no posionous effect on the catalyst. (C. A. 30, 892.)

Selective hydrogenation (of vegetable fats). E. Etinburg, B. Sterlin and B. Krushevskiĭ. *Masloboĭno Zhirovoe Delo* 11, 471-3 (1935).—The selective process of hydrogenation of cottonseed and sunflower oils was studied by heating the oils in an autoclave at 160°, 180° and 200° with 0.1, 0.2 and 0.4% Ni pptd. on kieselguhr and a H current at a rate of 2, 3, 4 and 6 l./min. The samples taken at definite intervals were tested for the m. p., I and thiocyanate nos. and contents of solid acids. The selective process of hydrogenation was detd. by the contents of satd. acids formed and the linoleic acid unchanged. Under all

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conditions of hydrogenation of cottonseed oil the selectivity was greater at a higher temp. (200°). With the increased ratio of either Ni or H the rate of hydrogenation increases and the degree of selectivity correspondingly declines. A selective hydrogenation can be effected not by any definite optimum system but by an optimum correlation of the single factors in the process. A min. of the catalyst and a max. of H are required; the converse is true. At a certain correlation of the catalyst and H the temp. of the reaction can be lowered without affecting the selectivity of hydrogenation: in the presence of 0.1% and 0.15% Ni (based on the wt. of oil) and H rate of 4 l./min. the selectivity is of the same character as that at 180° (for 0.1%) and 160° and 180° (for 0.15%). The same is true with 0.2% Ni and H rate of 3 l./min. For the selective hydrogenation of sunflower oil more active conditions are required. Thus, at a rate of 6 l./min. of H the selective character of hydrogenation is greater than that of cottonseed oil.
(C. A. 30, 889.)

New British Chemical Standards for Vegetable Oils. Perfumery & Essential Oil Record 26, 11, 440 (November, 1935). Six new British Standard Specifications have just been issued in connection with a comprehensive series of nationally agreed standards for vegetable oils at present being drawn up by the British Standards Institution.

These specifications are for coconut oil, ground nut oil, olive oil, rape seed oil, linseed oil, for general purposes and sampling of fats and fatty oils.

The specifications have been prepared by the special Vegetable Oils Committee of the Chemical Division of the British Standards Institution. This committee, which includes representatives of trade interests as well as the Government Departments concerned, has been actively working for the past two years under the chairmanship of Mr. E. R. Bolton, F.I.C., M.I. Chem. E. Limits are laid down in the specifications for impurities, color, refractive index, iodine value, saponification value and acidity, etc., whilst standard methods for determining these properties are included in appendices.

The details of the tests adopted have been arrived at after careful examination of existing methods of analysis, particularly from the standpoint of accuracy and reliability, and in many cases experimental work has been carried out by members of the committee in practical verification of the methods. The limits specified have been fixed by the principal users and suppliers.

British Standard Specification No. 627 for the Sampling of Fats and Fatty Oils deals with the general precautions to be taken and the procedure to be adopted in the sampling of oils and fats in packages or in bulk. Methods are given for the collection of the gross sample and for the preparation from the gross sample of the final sample. Recommended suitable forms of sampling instruments are also given. The specification should serve a very useful purpose as a guide to the sampling of fatty oils and forms a valuable reference in respect of the newly issued specifications.

Other specifications are nearing completion for castor oil, tung oil, refined cottonseed oil, crude palm kernel oil, crude maize oil, and sesame oil.

Hydrogenation of fats with copper-nickel catalyst. V. Vasil'ev. *Masloboĭno Zhirovoe Delo* 11, 444-8 (1935).—Exptl. evidence of the advantages of using Cu-Ni catalyst in the hydrogenation of refined and crude oils confirms the results of U. S. practice.
(C. A. 30, 889.)

Purification of Fatty Acids. *Oil and Colour Trades Journal* 88, 1930, 1089 (October 11, 1935). Keffler has studied the purification of oleic acid by fractional distillation of its esters ("Bull. Soc. Chim. Belge," 1935, p. 425). The most commonly employed method for the preparation of oleic acid consists of the saponification of olive oil, the liberation of the fatty acids with an acid, and the solid acids separated from the liquid acids by taking advantage of the differences in solubility of their salts in appropriate solvents. The acids thus prepared are converted into the methyl or other esters and then fractionally distilled. Attempts have been made to prepare pure oleic acid along these lines. After precipitating the solid acids from the alcoholic solution as the lead salts, the liquid acids remaining in solution were converted into the lithium salts, which were then recrystallized from 80 per cent alcohol. Finally the methyl, ethyl, and propyl esters were obtained, and fractionally distilled to eliminate the remaining impurities. Contrary to what has been stated in the literature, it is shown that a single distillation of the esters cannot be considered satisfactory where the oleic acid is to be used for extremely accurate determination, as, for example, in the determination of the heats of combustion of the acid and esters, and that even after several such fractionations there is still a possibility of the oleic acid not being absolutely pure. The author gives schematic diagrams of the method of purification used and follows the purity by means of the heat of combustion of the ester being prepared.

Conversion of cottonseed oil into a lard-like fat mixture by the method of conjugated hydrogenation. V. Puzanov and G. Ivanova. *Masloboĭno Zhirovoe Delo* 11, 365-8 (1935).—By the method of Lyubarskiĭ (*C. A.* 27, 1533) of catalytic conjugated hydrogenation an appropriate mixt. of cottonseed oil and EtOH was autoclaved in the presence of Al-Ni (72% Al, 28% Ni) at 190-200° and 23-7 atm. for 30 min., giving a snow-white fat mixt. of normal taste with the compn. and consistency of lard, acidity 3.73 mg., m. 32.2° I no. 74.2 and Hehner no. 96.
(C. A. 30, 889.)

PATENTS

Fats. Harburger Oelwerke Brinckman & Mergell. Ger. 617,520, Aug. 20, 1935 (Cl. 23*a*. 2). Addn. to 612,716 (*C. A.* 29, 6454^5). Clear odorless fats are obtained by treating fatty materials with CH_2Cl_2 and driving off the latter by steam in the presence of water and an adsorbent such as fuller's earth.
(C. A. 30, 893.)

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Wool-fat alcohols. Deutsche Hydrierwerke A.-G. (Ludwig Prigge and Karl Hennig, inventors). Ger. 617,975, Aug. 30, 1935 (Cl. 12o. 5.02). Alcs. incapable of sapon. are obtained from wool fat and other high-mol. wax-like substances by sapong. the wool fat, etc., treating the product with at least 30% of a soap with no more than 18 C atoms in the mol., and finally distg. Thus, wool fat is sapond. with NaOH and water and palmitic soap, obtained by treating palm oil with NaOH, added. The mixt. is distd. with superheated steam to give a product of wool-fat alc. (C. A. 30, 893.)

Purifying fatty oils. Karl Merck, Louis Merck, Wilhelm Merck and Fritz Merck (trading as (firm of) E. Merck). Brit. 434,432, Sept. 2, 1935. Tunny-fish liver oil is freed from undesirable color, taste and odor by treating at ordinary temp. with aq. or alc. alkali and then sepg. the purified oil. The alkali removes the larger proportion of free fat acids without sapon., or loss of vitamin A. (C. A. 30, 894.)

Oils; vitamins. Aktieselskabet Ferrosan. Brit. 433,930, Aug. 22, 1935. The vitamin-contg. unsaponifiable components of fats and oils are extd. or concd. by sapong. the oil with a soln. of NH_3 or alkali metal hydroxide in a H_2O-sol. org solvent, e.g., Me_2CO, MeOH, EtOH, cooling or standing to sep. the soap and acidifying the residual soln. to ppt. fat acids from the soap which remains dissolved, said acids collecting as a layer with the unsaponifiable components. (C. A. 30, 894.)

Neutral bleached edible oils and fats. Wilhelm Gensecke (to American Lurgi Corp.). U. S. 2,003,076, May 28. After sepg. the albuminous and mucilaginous matter from a colored edible oil or fat such as a crude maize oil, the free fat acids are sepd. by neutralization with alkali, and the oil or fat is heated to about 280° for a sufficient time to effect bleaching, free fat acids being formed and distd. off in a vacuum with the aid of an inert gas.

ABSTRACTS

Soaps

Edited by **M. L. SHEELY**

Medicinal Soaps. P. Martell, Allgem. Oel-u. Fett. Ztg. 32, 366-8 (1935). Tar and S-containing soaps are recommended for skin diseases, dandruff and falling hair. The S-containing soaps are also recommended for use by workers in metal industries, especially those who work with lead compounds. The addition of $HgCl_2$ to soap has the disadvantage of producing a grayish off-colored product; however, this soap finds application in disinfection of hands before operations and also for disinfecting surgical instruments. Other antiseptic soaps mentioned contain phenols, salicylic acid, formaldehyde or peroxides.
(C. A. 30, 1, 318, January 10, 1936.)

Lathering Power of Soaps. P. N. Das Gupta, Indian Soap Journal, 2, 2, 2, 1935. P. N. Das Gupta discusses the lathering power of soaps made from numerous oils and combinations of oils. The article contains a very useful tabulation of lathering indices.
(American Perfumery and Essential Oil Review, 31, 3, 98, November, 1935.)

The Oils, Fats and Soaps Industries in 1935. R. Furness, M.Sc., The Industrial Chemist XII, 132, 23 (January, 1936). It has been found by the I. G. Farbenindustrie A. G. that a product from paraffin wax (or other suitable mineral oil distillate) obtained by air oxidation at 120 to 150° C. in the presence of a suitable catalyst such as, for example, a manganese soap, can be made to furnish reasonably high quality soap-making fatty acids. The process consists in dissolving the oxidation product in a suitable solvent and removing therefrom, by means of saponifying alkali solutions, the fatty acid components.

The unreacted wax, etc., together with the oxidized products which are unsuitable for soap-making, remain in the solvent and may be recovered for re-use. The soap solution may be suitably worked up to relatively high quality products.

Again, with a view to improving the yields of useful detergent materials, the same company takes the crude mass resulting from the oxidation of wax, mineral oil distillates, etc., and subjects it to catalytic hydrogenation under conditions of high temperature and pressure such as are employed, for example, in ammonia synthesis, coal liquefaction, etc. By these means, which are now of course technically feasible, the aldehydic and ketonic portions of the oxidized hydrocarbons are converted into alcohols, whilst, although it is possible also to reduce the carboxylic compounds present in the oxidation product to alcohols by this catalytic process, the fatty acids remain as such. Thus, from an original hydrocarbon mixture, primarily converted by oxidation into a complex mixture of alcohols, esters, ketones, aldehydes, acids, anhydrides, etc., together with unconverted hydrocarbons, a hydrogenated product is obtained consisting mainly of alcohols, acids and unchanged hydrocarbons. This latter mixture is now sulphonated in such a way that only the alcoholic groups are affected so that the subsequent treatment of the sulphonated mass with alkali solution leads to the formation of a solution of alkali soaps and alkali salts of the sulphuric acid esters of higher alcohols—the unreacted hydrocarbons being removed for further