

method is still the most satisfactory. However, a canvas of the Committee members during the present year developed the fact that practically all laboratories were using a slight modification, namely, bringing the alcoholic filtrate to incipient boiling before titration. A vote was taken and as a consequence, the Committee recommends modifying the method to include this procedure. As a matter of record the method on page 4 will then read as follows:

"Free Alkali or Free Acid. Heat the filtrate from the above nearly to boiling, add 0.5 c.c. of a 1% alcohol solution of phenolphthalein, and titrate with standard acid or alkali solution, and calculate the alkalinity to sodium hydroxide (or potassium hydroxide) or acidity to oleic acid."

Moisture in Paste Soaps Containing Glycerine

Several members of the Committee have pointed out that the oven method on these types of soaps is not satisfactory if accurate results are desired, since more or less glycerine is distilled off, depending upon the length of time the sample is left in the oven. Cooperative work on a sample of this type of soap was carried out several years ago when the distillation method for moisture was shown to be satisfactory. Consequently, the Committee recommends including a note to

this effect in the present method.

For record purposes the preliminary comments to this method on page 3 will be changed to read:

"Moisture. The oven method given below is generally applicable to all soaps. Experience has shown, however, that certain exceptions to this method must be made if accurate results are desired. These exceptions include:

- (a) For soaps containing appreciable amounts of sodium silicate the distillation method is preferred.
- (b) Soaps of linseed and other oxidizing oils absorb oxygen and if the oven method is used may gain in weight near the end of the test. Therefore, either an inert atmosphere or vacuum oven should be used. The distillation method is also applicable to these types of soaps.
- (c) Soaps containing appreciable amounts of glycerine, such as cold made and semi-boiled, (including paste soaps) usually give high results by the oven method. The distillation method is preferred for most accurate results on these types of soaps."

Minor Change in the Method for Rosin

An omission has occurred in the first printing of the Modified Wolff

Method for rosin. On page 6-a, under "Second Esterification" which proceeds as per "First Esterification," the final ether extract is obtained, and ether evaporated on the steam bath. At this point the method should have stated that alcohol should be added and the solution titrated as described. In order to clarify the procedure, the paragraph should read as follows:

"Second Esterification: Cool and dissolve the residue in 20 c.c. of absolute ethyl alcohol and then proceed as above under 'First Esterification.' Add 30 c.c. neutral alcohol (94% or higher) and titrate rosin and rosin soap as desired, using phenolphthalein as indicator."

The remainder of the paragraph remains unchanged.

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ABSTRACTS

Oils and Fats

Edited by

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The spoilage of fats and structural material by heat and light in regard to economy and life. I. H. Schmalfluss, H. Werner and A. Gehrke. *Fette u. Seife*. 43, 211-4 (1936).—A review, the major portion of which deals with authors' research on ketone rancidity.

Does babassu oil menace butter? Anon. *Food Indus.* 8, 617; 653 (1936).—Nowhere near the amt. of babassu oil that would be necessary to put it on a par with coconut oil in volume of consumption is available.

Unified work of D. G. F. I. communication: The determination of unsaponifiable. H. P. Kaufmann. *Fette u. Seifen*. 43, 218-22 (1936).—The petroleum ether, centralburo and English methods for detn. of unsapon. were compared in several laboratories. Conclusion: The petrol. ether method is quick and it yields good results for fats with low unsapon. percentage, however, it is not generally applicable to fats of high unsapon. percentage. Using ether as a solvent it becomes generally applicable, but requires more time and is tedious. It is recommended that the petrol. ether

method be retained for fats with low unsapon. percentage. It was suggested that in further work on the English method that 5 g. samples instead of the usual 2 g. be used. Further work will be on the comparison of the methods on difficulty saponifiable fats contg. small amts. of unsapon.

Hydroxylated acids of fats: An improved method of determination. P. G. Hafner, R. H. Swinney and K. S. West. *J. Biol. Chem.* 116, 691-697 (1936).—The authors present a modified West-Hoagland-Curtis method for detn. of Ac. no., which is claimed to be more convenient and accurate. It has been shown, apparently for the first time, that a number of the common animal and vegetable fats contain small but easily detectable, amts. of hydroxylated acids. Some Ac. values detd. are: Butter 2.7, castor oil 125.6, coconut 1.3, corn 3.6, cottonseed 4.4, lard 1.5, linseed raw 5.3, linseed boiled 7.9, neat's-foot 9.0, olive 3.1, peanut 2.6, and salmon 3.7.

Testing edible oils. H. Jesser and E. Thomas. *Angew. Chem.* 49, 846-7 (1936).—Color reactions of 23 oils were obtained by the 3 following procedures:

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(1) 1 cc. of oil in CHCl_3 or benzol is treated with 1.5 cc. of 30% SbCl_5 . (2) 1.5 cc. of oil are treated with 1 cc. AsCl_3 soln. and (3) 0.1 cc. of oil is treated with 2 cc. of a mixt. of acetic acid anhydride and chloroform (1:1) and 0.05 cc. concn. H_2SO_4 is added. The colors produced by the various oils are tabulated. It is especially noteworthy that the blackish brown color reaction of soybean oil lends itself to detection of adulteration of other oils with the soybean oil.

The occurrence and possible significance of some of the minor component acids of cow milk fat. T. P. Hilditch and H. Paul. *Biochem. J.* **30**, 1904-13 (1936).—Detailed analyses of the component acids of typical cow milk fat was made by the fractional distn. of the methyl esters method. Complete data are presented. There is no detectable amount of any unsaturated acid of lower molecular weight than $\Delta^{9:10}$ -decanoic acid in butter fat, and the positions of the double bond, relative to the carboxyl group, are the same in the decenoic, tetra-, hexa- and octa-decenoic acids of butter fat. It is suggested that these observations are in harmony with the hypothesis put forward in earlier communications, namely, that the lower saturated glycerides of milk fats have been produced from preformed oleo-glycerides, and that these minor, lower unsaturated components may represent degradation products of oleo-glycerides which have escaped complete saturation to lower saturated groups.

The fat and phosphatide content of wheat germ. Bruno Rewald. *J. Soc. Chem. Ind.* **55**, 1002-3 (1936).—Nearly 63% of the phosphatides in the wheat are "bound"; the total amount of phosphatides is 0.611%; 80% are of the lecithin types and 20% are of the cephalin type. Furthermore, the amount of petrol-ether soluble fat is 6.70% and another amount of 0.85% fat can be extracted in a second extraction with a mixture of solvents, the total amount of fat is 7.55%.

Methylene-blue induction period and virginity of olive oils. B. B. Cunningham and L. G. Saywell. *Food. Res.* **1**, 457-464.—This work may be briefly summarized as follows: To date no infallible test for virgin olive oil has been reported. The natural anti-oxidant content of a non-treated oil, although markedly affected by most refining treatments, varies so widely that it cannot be used as a basis of a test for virginity. It does not appear likely that a substance such as virgin olive oil, defined upon a basis of its manner of preparation rather than its physical or chemical properties, can be uniquely and quantitatively estimated by any physical or chemical test.

Twitchell reagents. XVI. Relation between the constitution of fat acids and their darkening on heating with Twitchell reagents. Kyōsuke Nishizawa and Kazuo Matsumoto. *J. Soc. Chem. Ind., Japan* **39**, Suppl. binding 257-8 (1936).—Fat acids were heated in vacuum for 4-5 hrs. at 95° with 0.5% of reagent. Kontakt and Twitchell (naphthalene) prepn. caused a greater color increase than Idrapid, Divulson-D and Pfeilring, when heated with or without the presence of air. Stearic acid darkened slightly under any condition. The OH-group protected fat acids from darkening. The isomerization from oleic to elaidic acid

greatly increased the resistance to darkening. Liquid, unsatd. acids are the primary cause of coloring in the splitting by Twitchell reagents, and the darkening is greatly intensified by the presence of air in the Twitchell process. (*Chem. Abs.*)

The effect of temperature on the viscosity of fats and fatty acids. G. B. Ravich. *Kolloid-Z.* **76**, 341-5 (1936).—The viscosities of natural and hydrogenated oil from sunflower seeds, and of linseed, cottonseed, seal, dolphin, mustard, rapeseed and castor oils were detd. with a capillary viscosimeter (with an accuracy of 0.5%) at various temps. between 0° and 100°. The exptl. temp. slopes of the viscosities of these oils were in agreement with the Fulcher-Tammann equation: $\log \eta = [C/(t + t_0)] + \log \eta_0$. The viscosities decreased with increase in I nos. of the oils. At temps. near 0° the viscosity of the oil appeared to be a good indicator of its degree of satn. Linseed and dolphin oils showed anomalous viscosities at 0°. (*Chem. Abs.*)

Fat metabolism in fishes. X. Hydrogenation in the fat depots of the tunny. J. A. Lovern. *Biochem. J.* **30**, 2023-2026.—Fats from various parts of tunny fish were examined. Fats from all parts were characteristic of the tunny family. There were differences in content of certain acids and in the C_{18} acids. It is shown that the content of stearic acid is inversely proportional to the degree of unsatn. of the remaining C_{18} acids.

The phospholipid fatty acids of muscle. R. H. Snider. *J. Biol. Chem.* **116**, 503-510.—The nature of the fatty acids found in the phospholipid of muscle as shown by the ratio of saturated to unsaturated acids and by the iodine number of the unsaturated acids was found to be quite constant over a considerable spread of muscles in a variety of animals both exercised and unexercised. Data show the effect of exercise is not reflected in the degree of unsaturation of the component fatty acids of muscle phospholipid.

White rats as experimental animals in studies on the soft-fat problem. H. E. Robinson, R. E. Gray and R. C. Newton. *Food Res.* **1**, 413-418 (1936).—Rats fed on diets known to produce from firm to very soft hog carcasses show strikingly parallel body-fat formation. Saturated fats of high melting point and low iodine number tend to offset the effects of soy-bean and peanut oils on the body fat of the rat. Rats are apparently a satisfactory experimental animal to use in studying the soft-fat problem.

The specific dynamic action of butter fat, and of superimposed sugar. J. R. Murlin, A. C. Burton and W. M. Barrows, Jr. *J. Nutr.* **12**, 613-643 (1936). cf. following abstract.—The specific dynamic action of butter fat, eaten as heavy cream, was studied in nine human subjects by means of the new semi-automatic respiration calorimeter, supplemented by the Tissot-Haldane method. Amounts of fat contained in the test meal varied from 80 to 171 gm. S. D. A. experiments occurred on the fourth or fifth day of an all-cream diet. With six subjects sugar (sucrose or glucose C. P.) was superimposed on the high fat at an "early" interval (3 to 5 hrs.) after the fat meal was taken and the same amt. again at a "late" interval (11

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to 15 hrs.), for the purpose of testing its combustibility. The dynamic response to the sugar was of two types: (a) complete summation to the fat metabolism, and (b) an increase greater than that due to the same amt. of sugar fed alone; the difference is reckoned as fat metabolism. The extra heat may come in part from oxidation of ketone bodies induced by the sugar. With three subjects the sugar, glucose, fructose and sucrose, respectively, was given twice at equal intervals after last fat, and the combustibility and dynamic effects compared.

The rate of ketogenesis in human subjects on high fat diets, as influenced by different sugars. J. R. Murlin, E. S. Nasset, W. R. Murlin and R. X. Manly. *J. Nutr.* 12, 645-670 (1936).—Sugar causes a marked reduction in total ketosis within 3 to 4 hours. Sucrose is somewhat more effective in doses up to 50 gm.; glucose more effective in 100 gm. doses. Fructose stands between the other two. A "late" dose of sugar following an "early" dose was much more effective than the "early" one in reducing ketogenesis. Also it increased carbohydrate combustion more. This is interpreted as due to replenishment of glycogen in the liver, possibly because of insulin production stimulated by the early dose. Glycogen formation seems to be as important as combustion in the antiketogenic action of sugar. Quite possibly the presence of a certain minimal "normal" content of glycogen in the liver is a condition precedent to the normal oxidation of fatty acids. There is somewhat more evidence, however, that this normal content of glycogen prevents the formation of ketone substances in the liver.

Studies on ketosis. VIII. Quantitative studies on the oxidation of the ethyl esters of the fatty acids. H. J. Deuel, Jr., L. F. Hallman, J. S. Butts and S. Murray. *J. Biol. Chem.* 116, 621-639 (1936).—The administration of ethyl acetoacetate, ethyl butyrate, or ethyl caproate to fasting rats results in a uniform ketonuria which is somewhat lower than that produced by their sodium salts. More than twice the ketonuria was observed after the administration of the ethyl esters of caprylic, capric, lauric, and myristic acids as was found in the acetoacetate controls. This indicates that two fragments capable of forming acetone bodies are produced per molecule of fatty acid. It is postulated that palmitic, stearic and oleic acids break up into at least three fragments per molecule which are capable of transformation to acetone bodies. No appreciable ketonuria followed the administration of the ethyl esters of propionic, valeric, heptonic, pelargonic or undecylic acid.

Introduction of silicon into fats. G. Klein and H. Nienburg. *Ber.* 69B, 2066-8 (1936).—It was thought that the introduction of Si into the large fat mols. would not only make the resulting silicic acid compds. more stable to hydrolysis but that, on account of their lipid nature, they might be readily absorbed by tissues and thus improve the resistance and proliferation tendency of connective tissue. On heating 70g. of a-monostearin (4 mol.) with 10 g. of $\text{Si}(\text{OEt})_4$ (1 mol) for 5 hrs. in a sealed tube at 140° and distg. off the alc. from the reaction mixt. in vacuo at 100°, 67 g.

of a crude melt was formed which gave, on recrystn. from benzine, colorless crystals of a-monostearin orthosilicate, $\text{C}_{84}\text{H}_{164}\text{O}_{16}\text{Si}$, m. 70°. Similarly were prepd. the orthosilicates of a, b-stearin (I), a-mono-olein, a-monobutyryn and a, b-dibutyryn, $\text{C}_{156}\text{H}_{300}\text{O}_{20}\text{Si}$ (m. 68°), $\text{C}_{84}\text{H}_{156}\text{O}_{16}\text{Si}$ and $\text{C}_{44}\text{H}_{76}\text{O}_{20}\text{Si}$. These glyceride orthosilicic esters have almost the same phys. properties, particularly m. ps. and acly. relations, as the original glycerides. The combining relationship, of 4 mols. of glyceride to 1 mol. of $\text{Si}(\text{OEt})_4$ is maintained even when an excess of $\text{Si}(\text{OEt})_4$ is used. An attempt to combine 1 mol. of a, b-stearin with 1 mol. of $\text{Si}(\text{OEt})_4$ gave I, m. 68°.

Some characteristics of the residue from the cracking of castor oil. A. A. Vernon and K. K. Ross. *J. Amer. Chem. Soc.* 58, 2430-2433 (1936).—Castor oil was submitted to pyrolysis at various temps. in a heated silica tube. The acrolein and heptaldehyde were removed and the I No., Ac. value, sapon. value, and acid value of the residue was detd. Undecylenic acid was submitted to pyrolysis under the same conditions. The results may be explained by assuming that acrolein splits off first and then the molecule breaks down to give heptaldehyde. The latter reaction requires a higher temp.

PATENTS

Extraction of oil. C. F. Christensen. Norw. 56,981, Aug. 3, 1936. Finely divided whale blubber or the like is indirectly heated in a closed container and is maintained in circulation through a pipe external to the containers. The app. consists of a closed container with a pump and an external circulating pipe, the latter being so dimensioned that it contains only a small proportion of the material at any time. Around the wall near the top of the container where the material enters, is arranged a distributing device for the material. (*Chem. Abs.*)

Separating unsaponifiable from saponifiable material such as that contained in sperm oil, wool fat, etc. Max Schellmann and Hans Franzen (to I. G. Farbenind. A.-G.). U. S. 2,056,984, Oct. 13. Saponifiable constituents are converted into an aq. mixt. of Mg, Ca and K soaps in such relative proportions that the m. p. of the crude saponification product, when in an anhyd. state, is below 150°, and volatile unsaponifiable matter is sepd. from the aq. soap mixt. by distn. (*Chem. Abs.*)

Fractionating suints. Metallgesellschaft A.-G. Fr. 799,793, June 19, 1936. Suint is subjected under normal pressure to a slow distn. accompanied by the introduction of steam, the fatty acids and nonsaponifiable substances being thereby removed and the neutral constituents split into fatty acids and alcs. The nonsaponifiable substances present in the distn. product thus obtained are eliminated, the fatty acids having been previously sapond., by a distn. under high vacuum and at as low a temp. as possible, and, finally, the fatty acids from the previous decompn. of the nonsaponifiable substances are collected by a fresh distn. under high vacuum. (*Chem. Abs.*)