PROGRESS REPORT OF SOAP STOCK COMMITTEE 1937-38

The committee's time has been devoted to study of the Official and Optional Official methods, rule 276, sections 2 and 3, respectively, for the determination of total fatty acids in soap stock. Particular attention has been given acidulated soap stock from soya bean and corn oils. Ten samples of soap stock from soya bean and cotton seed oils, and acidulated soap stock from cotton seed, soya and corn oils have been analyzed in the chairman's laboratory. Some of these have been analyzed by the entire committee.

Using the Official method, we have conducted experiments in which the fatty acid cake was dried at room temperature and at 50-55 F. With the Optional Official, or wet extraction method, we have extracted the fatty acids with warm petroleum ether, used varying quantities of solvent, and held the solvent in contact with the fatty acids overnight.

Results to date indicate that both methods as now written give satisfactory results on the materials that we have analyzed. However, it appears advisable to re-write the methods, clarifying the wording, and making the directions more specific in several instances. A draft of the methods containing the proposed changes is now being studied by the committee, but we wish to accumulate more data before making our recommendations to the Society. A complete report will be given at the Spring meeting.

W. J. REESE,
C. P. LONG,
E. R. BARROW,
A. D. RICH,
J. J. LAPPEN,
W. T. WATKINS, Chairman.

REPORT OF THE GLYCERIN ANALYSIS COMMITTEE AMERICAN OIL CHEMISTS' SOCIETY, OCTOBER, 1937

FOR several months your committee has been engaged in coöperative work on the determination of the per cent glycerin yield of oils and fats. For natural oils of high quality glycerin yield may be ascertained fairly closely by calculation from ester value but for low grade stocks, which have undergone partial hydrolysis, or superglycerinated fats, glycerin yield must be estimated by direct methods of analysis.

The method selected for study involved a saponification of 5.000 gms. of sample with 5 ml. aqueous, 50% KOH. The oil and caustic are stirred in a beaker until a homegeneous paste is obtained and heated for four 15-minute periods in an oven at 105° C., stirring after each heating. The saponified mass is dissolved in water, decomposed with dilute H_2SO_4, cleared by heating and filtered through a wet paper into a volumetric flask from which an aliquot portion of suitable size may be taken with a pipette for dichromate oxidation.

Four oils were chosen for coöperative work, all of high quality: coconut, cottonseed, hydrogenated cottonseed and tallow. Eleven laboratories reported analyses by the method described, several even trying other methods for purposes of comparison. The results, on the whole, were surprisingly good, only two members of the committee having difficulties sufficient to impair the accuracy of their analyses seriously, though most of them appear to have had trouble with imperfect and incomplete saponification.

Though the proposed method is believed by several members to be satisfactory in the hands of an experienced analyst, the consensus of opinion appears to be that the uncertainty of complete saponification is so great that the method should not be recommended for adoption by the Society. It is expected that this work will be continued during the coming year, with the investigation embracing one or more other methods proposed by members of the Committee. In future work it is desirable to include low grade stocks which may necessitate purification of the glycerin solution before oxidation.

The Committee has under consideration the International Committee Methods of Analysis for Crude Glycerin. It is expected that action will be taken in the near future on the question of recommending certain of these methods to the Uniform Methods Committee.

A. O. C. S. Glycerin Analysis Committee—1936-1937

Ralph W. Bailey
Stillwell & Gladding, Inc.,
130 Cedar Street,
New York, N. Y.

H. C. Bennett
Los Angeles Soap Company,
617 East First Street,
Los Angeles, Calif.

W. H. Burkhart
Hecker Products Corporation,
Soap Division,
5300 Holabird Avenue,
Baltimore, Md.

J. E. Doherty
Lever Brothers Company,
Cambridge, Mass.

Chas. G. Gundel
Fels & Company,
Philadelphia, Pa.

L. F. Hoyt
Larkin Company, Inc.,
Buffalo, N. Y.

W. J. Reese
Colgate-Palmolive-Peet Company
Kansas City, Kans.

M. L. Sheely
Armour Soap Works,
Chicago, Ill.

B. S. Van Zile
Colgate-Palmolive-Peet Company
105 Hudson Street,
Jersey City, N. J.

C. C. Zeigler
Swift & Company,
Union Stock Yards,
Chicago, Ill.

J. T. R. Andrews, Chairman
Procter & Gamble Company,
Ivorydale, Ohio.

NOTICE

Standard Crude Glycerin Samples

In 1929 the Soap Section undertook the preparation and analysis of a Standard Crude Glycerin to be used as a check on the accuracy of such analyses in commercial transactions. Sixteen laboratories participated in the analytical work in which an almost unbelievable accuracy was attained. A four-ounce bottle of this Standard Crude Glycerin, with its accepted analysis by International Standard Methods printed on the label, may be obtained from Mr. J. C. P. Helm, 509 Tchoupitoulas Street, New Orleans, Louisiana, at a cost of only one dollar.

This notice is to emphasize the availability of these Standard Crude Glycerin samples, and even if your laboratory has had no difficulty in check analyses, the Standard should be on hand for check purposes in the event of controversies. Also, for those who are striking glycerol balances in the operations of their crude and refinery plants, such a standard should be of inestimable value.

ABSTRACTS

Oils and Fats

Edited by
M. M. PISKUR and RUTH LINDAHL

The spoilage of fats and oils. The Kreis reaction and its carrier. Richard Neu. *Chem.-Ztg.* 73, 733-36 (1937).—Neu reviews the literature on the subject of rancidity of fats, giving a bibliography of 36 references. He presents a new method for carrying out the Kreis test which yields a clear solution of the epihydrin-aldehyde-phloroglucinol condensation product. This solution, it is claimed, can be used directly for spectrographic analyses. The method is as follows: "Two cc. of the fat to be examined are placed, drop by drop, on 10-12 grams of granular silica gel contained in a porcelain dish about 5 cm. in diameter. (Standard Gel of the Silica Gel G.M.B.H., Berlin was used.) The fat is added in such a manner that absorption of the gel takes place immediately without wetting the walls of dish more than necessary. When this has been carried out correctly (which can be done with ease) one obtains a granular material which is then placed in a calcium chloride tube, a piece of glass wool having previously been placed in the base of the bulb. The upper part of the tube is then closed with a cork containing a glass tube of 1-2 mm. diameter, bent at right angles to the tube containing the sample. The bottom of this tube, which is drawn out to a fine capillary, dips in a 0.2% solution of phloroglucinol (prepared by dissolving 0.2 gm. pure phloroglucinol in 100 ml. of alcohol or of 20% HCl). The tube containing the sample may be cooled with cold water. The amount of phloroglucinol solution used is varied according to the degree of decomposition of the sample under examination, the volume being varied so that a color of sufficient depth for spectrographic examination is obtained (that is, the absorption band must be visible). In general, cells of sufficient width and permitting lengthwise examination are used, or one may use the absorption vessels recommended by Baly or Pulfrich. A vigorous stream of moist hydrochloric acid vapor is then passed down through the tube. The moist HCl stream is obtained by passing a stream of CO_2 which has been dried by conc. H_2SO_4 through a wash flask containing 25 cc. of conc. HCl (sp. gr. 1.18) and through a drying tower containing pumice stone which has been soaked in conc. HCl. Shortly after the stream has begun to pass through the tube, the solution at the bottom becomes more or less colored, depending upon the degree of decomposition of the sample. It is well to cool the absorption flask with ice water. The solution will be absolutely clear and show no traces of fat droplets even after bubbling the gas through for 1½ hours. The colored solutions can be examined directly in a spectroscope. The colors are stable for 1 hour. It is important, however, that the specified amounts of HCl be adhered to strictly, as otherwise stability of color is adversely influenced and the condensation may not be complete." Neu points out that the merit of this method lies in the large surface of fat which is exposed by adsorption on silica gel. He calls attention to the fact that no photographs of the adsorption bands of rancid oils have been published and fills the gap in the literature by supplying two spectral photographs covering rancid olive oil, C_6—C_{10} fatty acids, and epihydrin aldehyde.

Cooked butter. W. Ritter. *To be published in Deutsche Lebensm. Rundschau.* Ritter discusses several phases of the manufacture of cooked butter in Switzerland. (1) *Methods of Manufacture:* The usual process for preparation of cooked butter consists in removing the water by evaporation; in this process the buttermilk constituents settle out in insoluble form and the fat can be easily separated. Other processes separate the fat at temperatures below 50°C. by allowing product to stand and then separating the fat. In the first process, a fat insoluble residue which can be used for feed preparation is obtained. In the latter process, a watery layer which is useless is obtained. (2) *Changes During Cooking:* The butter first melts and a cloudy system is produced. When boiling begins a strong foam usually forms, later giving way to quiet boiling. The mixture soon clarifies with temp. rising to 105-110°C. A second foam develops which in some instances turns brown. The residue which separates is sometimes coarse and flocculent and at other times fine and crystalline. (3) *Lecithin in Cooked Butter:* Cooked butterfats, which have been obtained by separation at low temp. (below 50°C.), contain no lecithin, while those obtained by boiling contain lecithin. It appears that the lecithin at first goes into the insoluble residue from which it is later dissolved into the fat at the higher temperatures and toward the end of the process. (4) *Water Content of Cooked Butter:* Due to solubility of water in butterfat, it is practically impossible to reduce moisture below 0.1%. Moisture is an important consideration in rust which develops in tin cans containing the cooked butter, and in autoxidation of the fat. (5) *Differences Between Melted and Cooked Butter:* "Melted" butter is fat removed at low temperature and "cooked" butter is that removed by boiling or evaporation of water. Melted butter usually has the odor of the raw material