Soap Section Committee Report

American Oil Chemists' Society, New Orleans, La., May 9, 1930
Presented Before Twenty-first Annual Convention

By A. K. CHURCH, *Chairman*

DURING the year the committee has completed its work on the analysis of the A. O. C. S. crude glycerin standard sample. The accepted analysis reflects accurately, we believe, within the limits of the method, the correct analysis of this sample when carried out by the International Acetin Method for crude glycerin adopted 1911. Never before has there been available in the United States a crude glycerin sample of authenticated analysis. The committee feels that such a sample will be found of value to all laboratories engaged in glycerin work. This A. O. C. S. standard crude glycerin sample is for sale by J. C. P. Helm, Sec., A. O. C. S., 705 Tchoupitoulas St., New Orleans, La., at one dollar a four-ounce bottle. The complete analysis is printed on the label. A detailed report of the committee's work on this sample appeared in *"Oil & Fat Industries,"* Dec., 1929.

The Soap Section Committee recommends that in the printed standard methods of the A. O. C. S. reference be made to the IAM for crude glycerin as the method for crude glycerin analysis adopted by our Society.

Standardization of the Acid Reagent

IN CONNECTION with work on the crude glycerin sample, methods of standardization of the acid reagent used were discussed at considerable length, two methods being presented in detail in the report of the committee's work published in *Oil & Fat Industries,* Dec., 1929. It is the consensus of opinion of the members that explicit directions for standardizing the acid are inadvisable for the reason that qualified chemists know how to prepare standard acids accurately—if they do not, they are not competent to carry through the Acetin Method. Also the I. S. M. Committee of 1911 did not consider it necessary to recommend a standard method.

On the other hand a minority opinion has been expressed that it is generally admitted that identical and specific procedures tend towards closer checks between different laboratories and encourage more careful and accurate work in all laboratories. That this is the age of standardization in all things and especially in methods of physical and chemical tests. That if it be desirable, for example, to recommend a standard method for the simple operation of determining the free fatty acids in an oil, it would seem decidedly more desirable and important to recommend a specific method of preparing the standard acid used in the free fatty acid determination, since the correctness of the FFA determination depends primarily on the correctness of the standardization of the acid.

A specific method for standardizing acid employed in oil, soap, and glycerin analysis, using the A. O. C. S. standard sodium carbonate now available and of which there is an ample supply, might be worth considering by the Uniform Methods Committee.

The laboratories that sent in results on the soap follow: (The laboratories are not listed in the order of the analyses.)

Fels & Co., Philadelphia, Pa.; La France Mfg. Co., Philadelphia, Pa.; Lever Brothers Co., Cambridge, Mass.; Lever Brothers, Ltd., Toronto, Canada; Larkin Co., Inc., Buffalo, N. Y.; Kirkman & Son, Brooklyn, N. Y.; Dr. Foster D. Snell, Brooklyn, N. Y.; Swift & Co., Chicago, Ill.; Mellon Institute, Univ. of Pittsburgh, Pittsburgh, Pa.; Curtis & Tompkins, San Francisco, Cal.; Procter & Gamble, Ivorydale, Ohio; The Palmolive Co., Milwaukee, Wis.; Colgate-Palmolive-Peet Co., Berkeley, Cal.; Los Angeles Soap Co., Los Angeles, Cal.

The standard methods for the analysis of soap adopted by the American Chemical Society and approved by our own Society, were employed in this work. The results are, in respect to some items, in your chairman's opinion, unsatisfactory. Mr. Peterson, Sec. of the Soap Section, comments, in part, as follows:—

Soap Sample

FOURTEEN laboratories submitted analyses of the soap sample, one laboratory sending two, each by a different analyst, making fifteen results received. The analyses follow:

A. O. C. S. Standard Soap Sample Calculated to Dry Basis

	1	2	3	4	5
Per Cent Fatty Anhydrides	85.68	85.94	86.10	86.03	86.45
Per Cent Fatty Anhydrides	10.07	10.01	10.11	10.07	10.095
Per Cent Combined Alkali (Na_2O)	0.92	0.15	0.60	0.74	0.312
Per Cent Combined Alkali (Na_2O)	0.67	0.93	Nil	N.F.	0.01
	87.27	87.02	86.70	86.77	86.772
Per Cent Glycerol	0.32	0.35	0.38	0.405	0.415
Per Cent Free Caustic Soda (NA_2O)	Nil	Trace	Nil	Nil	Trace
Per Cent Free Fatty Acids as Oleic	Nil	None	Nil	Nil	None
Per Cent Silica (SiO_2)	1.36	1.37	1.48	1.46	1.551
Per Cent Carbon Dioxide in IIA	0.21	0.22	0.21	0.061	0.091
Per Cent Total Alkalinity of Matter Insoluble in Alcohol calc. as Na_2O	0.77	0.82	0.81	0.88	0.83
Per Cent Sulphates calc. as Sodium Sulphate (Na_2SO_4)	Nil	0.04	0.03	0.009	0.04
Per Cent Chlorides calc. as Sodium Chloride (NaCl)	0.22	0.20	0.27	0.223	0.283
	100.22	100.03	99.99	99.878	100.077
Constants of Total Fatty Acids including unsaponified and unsaponifiable:					
Titre	40.4	40.48	40.45	40.4	39.90
Saponification Value	207.2	206.55	203.7	204.7	204.75
Iodine Number	59.8	60.60	60.1	59.86	57.77

	10	11	12	13	14	Ave.
Per Cent Fatty Anhydrides	86.38	86.04	86.15	86.30	85.80	86.29
Per Cent Combined Alkali (Na_2O)	10.07	11.01	9.93	10.11	10.22	10.13
Per Cent Unsaponifiable Matter	0.46	0.19	0.15	0.18	0.43	0.44
Per Cent Unsaponified Fat	0.01	0.10	0.15	0.28	0.57	0.20
	86.85	86.33	86.45	86.76	86.80	86.93
Per Cent Glycerol	0.38	0.336	0.33	0.43	0.25	0.35
Per Cent Free Caustic Soda (NA_2O)	Nil	0.05	Trace	Nil	0.062	Trace
Per Cent Free Fatty Acids as Oleic	Nil	Nil	Nil	Nil	Nil	Nil
Per Cent Silica (SiO_2)	1.56	1.482	1.66	1.44	1.40	1.39
Per Cent Carbon Dioxide in IIA	0.075	Trace	0.06	0.23	—	0.136
Per Cent Total Alkalinity of Matter Insoluble in Alcohol calc. as Na_2O	0.81	0.421	0.73	0.75	0.720	0.780
Per Cent Sulphates calc. as Sodium Sulphate (Na_2SO_4)	0.039	0.049	0.025	0.06	0.04	0.026
Per Cent Chlorides calc. as Sodium Chloride (NaCl)	0.25	0.214	0.26	0.27	0.21	0.246
	100.034	99.892	99.445	100.05	99.702	99.938
Constants of Total Fatty Acids including unsaponified and unsaponifiable:						
Titre	40.30	40.75	40.20	40.0	39.00	40.21
Saponification Value	204.77	199.95	203.4	200.9	204.30	203.95
Iodine Number	59.48	60.74	—	55.5	63.80	59.65

Standard Soap Sample Calculated to Dry Basis

	6	7	7a	8	9
Per Cent Unsaponifiable Matter	87.10	86.31	86.22	87.48	86.40
Per Cent Unsaponifiable Matter	10.04	10.00	10.03	10.17	10.08
Per Cent Unsaponified Fat	0.78	0.41	0.61	0.21	0.44
Per Cent Unsaponified Fat	Nil	Nil	Nil	0.22	0.02
	87.88	86.72	86.83	87.91	86.86
Per Cent Glycerol	0.37	0.32	0.35	0.24	0.35
Per Cent Free Caustic Soda (NA_2O)	0.009	0.02	.017	None	Nil
Per Cent Free Fatty Acids as Oleic	Nil	Nil	Nil	None	Nil
Per Cent Silica (SiO_2)	1.28	1.46	1.37	0.48	1.52
Per Cent Carbon Dioxide in IIA	—	0.34	0.31	0.17	0.072
Per Cent Total Alkalinity of Matter Insoluble in Alcohol calc. as Na_2O	0.92	0.87	0.83	0.75	0.80
Per Cent Sulphates calc. as Sodium Sulphate (Na_2SO_4)	Nil	0.009	.010	None	0.041
Per Cent Chlorides calc. as Sodium Chloride (NaCl)	0.25	0.20	0.23	0.35	0.26
	100.749	99.939	99.977	99.97	99.983
Constants of Total Fatty Acids including unsaponified and unsaponifiable:					
Titre	40.2	40.5	40.4	40.55	39.75
Saponification Value	200.85	204.9	204.8	203.80	204.90
Iodine Number	—	60.7	60.4	59.50	57.20

" x x x the results are far from satisfactory with respect to agreement in some of the individual items, while in others the agreement seems to be fairly close. Would this not seem to indicate that the methods of some of these items are at fault?

"It seems to me that the laboratories who have worked on this sample are capable of close agreement where the methods in use are capable of producing checking results. This is borne out by the work on the glycerin sample where very good agreement was obtained."

The chairman feels that improvement of the present standard method for determining the unsaponifiable matter and the unsaponified fat in soap is indicated as desirable by the above results.

Standardization of Reagents for the International Acetin Method for Crude Glycerin

DURING the course of our work on the crude sample your chairman's attention was called by a committee member to the fact that if the true glycerol, organic residue, and ash figures of the accepted analysis were added together, and the moisture determined and added thereto, instead of totaling approximately 100 the sum amounted to only about 98.5% showing about 1.5% unaccounted for. This discrepancy was confirmed in the chairman's laboratory on the determination of the water in our standard crude glycerin sample. Then a sample of C. P. glycerin certified to by the British Executive Committee (see below) and employed in England for the standardization of reagents used in the International Standard Methods for crude glycerin was procured. Its specific gravity was determined; also the total acetylizable as glycerol by the Acetin Method; also the glycerol by the bichromate method. The percent of glycerol from the sp. gr., using the Bosart & Snoddy tables, was 90.40; by the Acetin Method 88.86%. Here again is an unaccounted for difference of about 1.5%. The percent of glycerol by the bichromate method was 90.39 confirming the results from the sp. gr. tables.

Throughout the world crude glycerin is bought and sold on the International Acetin Method analysis. In the United States, we have thus far stuck to the use of a standard acid as the control agent, as originally specified by the International Committee in 1911. In Great Britain, however, the British Executive Committee under date of January, 1914, in "Supplement No. 1 (I. S. M. 1911)" in respect of the "Standardisation of Re-Agents" recommend that "The final standardisation is to be carried out upon Chemically Pure Glycerin in which the water content has been accurately determined." Chemically Pure Glycerin of standard quality is supplied bearing the seal and label of the British Executive Committee.

At the time the unaccounted for loss by the Acetin Method was brought to your chairman's attention he was not acquainted with the action of the British Committee in 1914 in adopting the use of C. P. Glycerin for standardizing the re-agents used in the Acetin Method. He has since been informed that the reason for this action by the English chemists in 1914 was the unaccountable loss noticed by them when using the Acetin Method as promulgated in 1911. So it would appear that we have rediscovered this same unaccounted for loss not realizing that it had already been discovered and action taken upon it by our English brothers.

This unaccounted for loss by the Acetin Method is now before the Soap Section Committee for their consideration. Some members have expressed the opinion that the Acetin Method is simply a measure of the OH groups whether these be present as glycerol or other compounds. Therefore, how can any sample of C. P. Glycerin be selected for standardization purposes with the assurance that it is absolutely definite in its glycerol content inasmuch as there may be other OH groups present? Again, since the Acetin Method is used as a standard for glycerol in buying and selling, the question whether it gives a true glycerol content or not is not important commercially but only of academic interest. The question, therefore, seems to be, so some of the committee have expressed themselves, whether the Committee as a whole would be sufficiently interested in an academic problem to spend much time on it.

On the other hand, other members have expressed a desire to investigate this apparent discrepancy; and most of the laboratories have indicated a willingness to work on this problem to the extent their facilities and time permit. Your chairman is one of those who cannot feel comfortable until this unaccounted for loss shown by the Acetin Method, when using standard acid as the controlling re-agent, is explained, or, if not explained, rectified.

The chairman and secretary wish to thank each member of the committee and each cooperating laboratory for the work done and the fine spirit of co-operation manifested.

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Cottonseed Analysis

(*From Page 292*)

tube and place a layer of absorbent cotton on the plate. Place the meats in the prepared tube and pour sufficient portions of petrolic ether on the meats to extract at least 5 grams of oil. Receive the extract in a tared flask. Evaporate the ether from the oil on a steam bath. Care must be taken to see that all the ether is removed from the oil. Weigh the oil, add 30 cc. neutralized alcohol (Formula 30) and titrate the free fatty acid of the oil with standard alkali, using phenolphthalein as the indicator. (0.1 N alkali is used if f. f. a. if low, but 0.25 N is used for oils with f. f. a. above 3 per cent). The addition of a small amount of petrolic ether before titration makes the end point sharper. The titration is performed in a flask which is shaken vigorously during the titration, the end point being taken when a permanent pink is obtained which persists for at least one minute.

$$\text{Per cent F. F. A.} = \frac{28.2 \times \text{Normality of alkali} \times \text{cc. used}}{\text{weight of oil}}$$

California Olive Oil

(*From Page 296*)

that California oils be further studied along these lines, with the idea of calling attention to cases of considerable variation from the average; second, that the average figures of 80 yellow—5 red—2 blue for color, 15-16 for relative viscosity, and 1.0 per cent. for free fatty acid be considered as tentative standards for the purpose of such investigation; and third, that a slight but distinct positive Kreis test be considered tentatively as maximum permissible rancidity for the purpose of such investigation.

References

[1] Kerr, R. H. Jour. Ind. Eng. Chem., *10*: 471-475 (1918).

[2] Issoglio, G. Ann. Chim. Applicata, *6*: 1-18 (1916); Atti. Accad. Sci. Torino, *51*: 582-605; Chem. Abs. *10*: 2943 (1916). (Original not seen.)

[3] Fellenberg, T. von Mitt. Lebensm, Hgy. *15*: 198-208 (1924); Chem. Abs. *18*: 3731 (1924). (Original not seen.)

[4] Vintilesco, I., and Popescu, A. Bul. Acad. Sci. Roumaine, *4*: 151-157 (1915); J. Pharm. Chim. *12*: 318-323 (1915); Chem. Abs. *10*: 646 (1916). (Original not seen.)

Soap Chemists' Report

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The committee and cooperating laboratories follow:

Ralph W. Bailey, Stillwell & Gladding, Inc., New York City; Chas. J. Gundel, Works Chemist, Fels & Co., Philadelphia; L. F. Hoyt, Manager, Research Dept., Larkin Co., Inc., Buffalo; Martin H. Ittner, Chief Chemist, Colgate-Palmolive-Peet Co., Jersey City; H. J. Morrison, The Procter & Gamble Co., Ivorydale; W. D. Richardson, Chief Chemist, Swift & Co., Chicago; M. L. Sheely, Chief Chemist, Armour Soap Works, Chicago; M. L. Sheely, Chief Chemist, Armour Soap Works, *Babbitt Lab.*, Jersey City; H. P. Trevithick, Chief Chemist, New York Produce Exchange, New York City; R. B. Trusler, Industrial Fellow, Mellon Institute, Pittsburgh; H. C. Bennett, Chief Chemist, Los Angeles Soap Co., Los Angeles; V. K. Cassady, Chief Chemist, The Palmolive Co., Milwaukee; Curtis & Tompkins, San Francisco; M. R. Dickson, Chief Chemist, Colgate - Palmolive - Peet Co., Berkeley; M. M. Durkee, The A. E. Staley Mfg. Co., Decatur; F. E. Joyce, Haskins Bros. & Co., Omaha; A. J. Harvey, Technical Director, Lever Bros., Ltd., Toronto; John Ornfelt, LaFrance Mfg. Co., Philadelphia; Foster D. Snell, Brooklyn; W. J. Reese, Chief Chemist, Colgate - Palmolive - Peet Co., Kansas City; Wm. A. Peterson, Chief Chemist, Kirkman & Son, Brooklyn, Secretary, Soap Section, A. O. C. S.; A. K. Church, Chief Chemist, Lever Brothers Co., Cambridge, Chairman, Soap Section, A. O. C. S.

The Institute for Commercial Expansion of the Ministry of Agriculture, Industry and Commerce of Brazil has issued a very complete discussion entitled "The Babassu Nut," published in Portuguese and English, which gives extensive information on the occurrence in Brazil of the Babassu palm, the products which may be developed from the palm and the conditions surrounding its exploitation. The booklet is well prepared and attractively printed. It is profusely illustrated with half-tones and contains maps of the babassu-producing areas, together with many beautifully designed colored charts setting forth exports of babassu products in recent years and imports of various commodities which may be affected by the development of the babassu industry.

Referee Applicant

Mr. Clinton Morris, of Morris-Flinn Company, Macon, Georgia, has applied to the American Oil Chemists' Society for Referee Chemist certification on all products covered by the Rules of National Cottonseed Products Association. (*First Publication*)