

of the samples. It was decided at the meeting last year that no samples would hereafter be discarded.

Judging from the comments of the collaborators, the samples this year, with the possible exception of No. 25, have been generally very uniform. A number of collaborators expressed the belief that this sample was not uniform. This was confirmed later by several of the collaborators who exchanged samples with each other and found quite different results than on their own samples. There has been very little cause, however, for criticism of the samples, which have been generally very satisfactory.

There have been more instances this year of collaborators failing to receive samples on time. This has kept several who otherwise would have reported on all 30 samples from being included in the final reports. This is partly, however, the fault of the collaborators in failing to notify Mr. Bailey in time to have another sample mailed.

Only about half of the collaborators responded to the Chairman's request published on report No. 30 that they figure up their number of points off on each sample and advise the Chairman of their findings. The Chairman has not double-checked the results except those who have earned certificates. It would seem that in view of the time required by a Chairman of any of our active committees in the conduct of work of this kind that the collaborators might reply to requests of this kind, which indicates a willingness to cooperate, and will make the work much more enjoyable to all.

Two members of the Ammonia Committee have suggested that check samples be continued throughout the year, but that they be not sent so often during the summer months. The Chairman does not concur in this recommendation, and does not believe that the Society will, for many obvious reasons.

The Ammonia Committee during the past year has undertaken no additional research or study of methods, and has nothing in addition to report.

Committee: H. C. MOORE (Armour Fert. Works, Chicago), L. B. FORBES, C. A. BUTT, JOHN MALOWAN, C. H. COX.

SOAP STOCK COMMITTEE REPORT 1923-24

BY A. A. ROBINSON

The committee this year divided its work into three sections:

First was a continuation of the work begun by last year's committee on the F. A. C. Committee (American Chemical Society) method for unsaponifiable as applied to soap stock.

Second, it tried out two methods, or rather two modifications of one method for Free Oil in soap stock.

Third, two proposed changes in the official method for total fatty acids

were investigated. These changes consisted in (a) Drying the fatty acids under an inert gas such as carbon dioxide, and (b) Baking the saponified mass, just after the alcohol is driven off, at 120°C. for two hours. This latter was suggested by an article by Dr. Steipel in *Zeit d. Ol u Fett Ind.*, vol. 41, 1921, in which he stated that this is necessary to get rid of certain alkali-combining, resin-like oxy fatty acids, which would otherwise be determined as fatty acids. These substances are not only worthless as soap, themselves, it being impossible to salt them out, but they also render an equal quantity of soap of fatty acids incapable of being salted out.

SAMPLE No. 1 CORN OIL SOAP STOCK

	Unsaponifiable matter %	% Free oil		Official method		Total fatty acids Drying under CO ₂		Baking at 120°C.		T. F. A. by separatory funnel method
		KOH	NaCl	%	I No.	%	I No.	%	I No.	
Moore	4.49	15.13	16.39	50.83	...	51.09	...	50.92
Long	4.98	14.90	16.77	51.27	...	51.53	...	51.80	...	51.39
Reese	5.00	14.51	15.81	51.91 ^a	...	50.58 ^{a,b}	...	52.31 ^a
Tompkins	4.62	15.14	15.19	51.90	...	51.92	...	52.03
Irwin	5.00	14.90	15.40	51.13	...	50.98	...	51.10
Robinson	4.87	15.00	15.35	51.60	...	51.41	...	51.34

SAMPLE No. 2 SOYA BEAN OIL SOAP STOCK

Long	1.40	7.42	7.72	37.98	...	37.47	...	37.61	...	37.80
Tompkins	1.08	9.35	9.15	39.25	121.0	39.08	131.0	38.95	121.0	...
Reese	1.71	8.35	8.03	40.06	70.5	37.83	121.0	38.77	94.5	...
Bosart	2.13	8.59	7.80	39.80	134.6	39.80	135.1	39.50	127.8	...
Irwin	1.34	8.05	8.11	37.30	128.8	37.30	130.4	37.10	121.0	...
Strack	1.32	8.96	8.29	37.93	123.3	38.02	127.9	37.91	127.0	...
Robinson	1.14	7.72	7.79	37.87	...	37.65	...	37.81

^a Wet extraction method used.

^b Dried under natural gas.

Unsaponifiable Matter

The results on unsaponifiable matter obtained on the sample of corn oil soap stock showed fairly close agreement. Those obtained on the Soya Bean Soap stock were not as good. However, the F. A. C. Method has for years proven so successful on Fats of all kinds, that your committee believes that it will be satisfactory for soap stocks. The method is simple, fairly rapid and gives concordant results in the hands of different analysts. Your committee recommends that it be made official for soap stocks. The F. A. C. Method for unsaponifiable matter is as follows:¹

Determination

“Weigh 5 g. (±0.20) of the prepared sample into a 200cc. Erlenmeyer flask, add 30 cc. of redistilled 95% Formula 30 alcohol and 5 cc. of 50% aqueous potassium hydroxide and boil the mixture for one hour under a reflux condenser. Transfer to the extraction cylinder and wash to the

¹ *J. I. E. C.*, 11, 1164 (1919).

40cc. mark with redistilled 95% ethyl alcohol. Complete the transfer, first with warm then with cold water, till the total volume amounts to 80 cc. Cool the cylinder and contents to room temperature and add 50 cc. of petroleum ether. Shake *vigorously* for one minute and allow to settle until both layers are clear, when the volume of the upper layer should be about 40 cc. Draw off the petroleum ether layer as closely as possible by means of a slender glass siphon into a separatory funnel of 500cc. capacity. Repeat extraction at least four more times using 50 cc. of petroleum ether each time. Wash the combined extracts in a separatory funnel three times with 25cc. portions of 10% alcohol, shaking vigorously each time. Transfer the petroleum ether extract to a wide-mouth tared flask or beaker and evaporate the petroleum ether on a steam-bath in an air current. Dry as in the method for moisture and volatile matter. Any blank must be deducted from the weight before calculating unsaponifiable matter. Filter and wash free from the insoluble residue, if any, evaporate and dry in the same manner as before. The committee wishes to emphasize the necessity of thorough and vigorous shaking in order to secure accurate results. The two phases must be brought into the most intimate contact possible, otherwise low and disagreeing results may be obtained. When the unsaponifiable matter runs over 5%, more extractions are recommended."

Free Oil

The method for free oil investigated was one which was tried out by the committee for 1920-21. It is as follows:

"A," weigh 10 gm. of the well mixed sample into an extraction cylinder. Dissolve in 80 cc. of 50% alcohol, made up from redistilled alcohol. Add 50 cc. of petroleum ether, boiling below 65°C. Shake thoroughly, then add 10 cc. of 14% solution of potassium hydroxide and shake vigorously for one minute. Allow to settle until both layers are clear, draw off the petroleum ether layer as closely as possible with a glass siphon, into a 500cc. separatory funnel. Repeat the extraction at least four times, using 50 cc. petroleum ether each time.

Wash the combined extracts in the separatory funnel three times with 25cc. portions of 10% alcohol, shaking vigorously each time. Transfer the petroleum ether extract to a tared soxhlet flask, and evaporate the petroleum ether on the steam-bath. Dry in oven at 105°C. to constant weight. This weight includes neutral glyceride and unsaponifiable matter. The latter is subtracted, giving the weight of neutral oil.

"B," the same as above except that 10 cc. of sodium chloride is used in place of the 10 cc. of 14% potassium hydroxide. In this case, the dried oil is dissolved in a little petroleum ether, an equal volume of neutral alcohol added and the free fatty acids present titrated with 0.1N alkali,

using phenolphthalein as an indicator. The amount found is deducted from the weight of neutral oil, free fatty acids and unsaponifiable matter.

This method "B" was found to be unsatisfactory due to the difficulty of getting two clear layers when extracting with petroleum ether. Practically all the collaborators reported that they had trouble in getting check results by this method.

Method, "A," seems to offer possibilities of being satisfactory. The results obtained were fairly close, and not much difficulty was experienced in making the determination. However, the question arises as to whether or not the 14% potassium hydroxide used caused a loss of neutral glyceride by saponification. Your committee does not believe it advisable to recommend the adoption of this method until that point has been investigated.

Total Fatty Acids

An examination of the results obtained on the total fatty acid determinations, shows clearly that there is no value to either of the proposed modifications. No lower results were obtained when the fatty acids were dried under CO₂, and the iodine absorption value of the fatty acids was not materially changed. This confirms the results obtained a few years ago, when it was found that it was not necessary to dry the fatty acids from crude cottonseed oil in an inert atmosphere, due to the protective action of certain impurities present.

Baking the soap mass at 120° for two hours was without effect on the amount of total fatty acids determined.

In the course of the year's work a matter, which the committee had not considered, was brought to its attention. That is the substitution of a wet extraction method for total fatty acids other than cocoanut and palm kernel. The method now official is slow and for that reason is not used by many laboratories in cases not involving settlement. In addition, on soap stocks from the more liquid oils, such as soya bean, corn and linseed, the present official method can be followed only with the greatest difficulty. A chilled work room is necessary if the fatty acids are to be solidified and kept so until the filter paper is dry.

Enough experimental work has been done in the past to prove beyond a doubt that a wet extraction method is capable of as great accuracy as the present method. This was brought out by the report of the committee for 1920-21, at which time Mr. Long was chairman. However, as additional data, in the present report, Mr. Long has shown the results obtained by a wet extraction method, while all of Mr. Reese's results were obtained by wet extraction.

In view of this, the committee wishes strongly to urge the adoption of a wet extraction method for total fatty acids in the place of, or, at any rate, in addition to the method now official.

The method that it proposes for adoption is as follows:

Total fatty acids.

NOTE: Excepting Copra and Palm Kernel Soap Stock.

Weigh out from weighing bottle 8 or 10 grams of the well mixed sample of soap stock, or 4 to 5 grams of acidulated soap stock, and transfer to a 400cc. beaker. Saponify with a excess of alcoholic potassium hydroxide. After saponification is complete heat on a steam-bath, with stirring until all the alcohol is driven off.

When the alcohol is evaporated, add 100 cc. of water and heat until the soap is dissolved. Wash the contents of the beaker into an extraction cylinder (a 250cc. glass stoppered cylinder) with hot water taking care not to exceed 130 cc. total volume in the cylinder. Acidify with dilute hydrochloric acid (1:1), carefully avoiding too large an excess. Mix gently by rotating the cylinder. When the cylinder has cooled to 50°C. add 100-125 cc. redistilled petroleum ether boiling below 65°C. It is not necessary for the fatty acids to have cleared thoroughly. Stopper cylinder and shake, then allow to stand until the petroleum ether layer has separated. Siphon off this petroleum ether layer through a 9 cm. filter paper to a 400cc. beaker. Make at least four more extractions using 25-30 cc. of petroleum for each extraction. Allow filter paper to drain well, then wash with a spray of petroleum ether from a wash bottle, until all the fatty acids are extracted. Add all the petroleum ether extractions to the first extraction in the 400cc. beaker. Evaporate partially and transfer to a tared 150cc. Soxhlet flask on the steam-bath, washing all traces of the fatty acids from the beaker with petroleum ether from a wash bottle. Evaporate all petroleum ether and heat in oven at 105°C. to constant weight. Report as per cent, "total fatty acids."

This is essentially the same method as is now official for cocoanut soap stock and should give equally satisfactory results.

Summary

1. The committee recommends that the F. A. C. Method for unsaponifiable matter be made official for soap stocks.

2. The committee does not recommend any method for free oil in soap stocks. However, it found the method, "A," as described above, to give promise and advises that work on it be continued in the future.

3. Drying the total fatty acids under CO₂ is not necessary, even with such easily oxidized fatty acids as those from corn and soya bean oils.

4. Baking the saponified mass at 120°C. is not necessary.

5. The committee recommends that a wet extraction method for total fatty acids in cottonseed and other soap stocks be made official.

The committee wishes to express its appreciation of the valuable colla-

boration given it by Mr. Irwin, of Swift & Co., Dr. Bosart of Procter & Gamble and Mr. Strack of the Southern Cotton Oil Co.

Committee: A. A. ROBINSON, Wilson & Co., Chicago Ill., C. P. LONG, W. J. REESE, G. A. MOORE and P. W. TOMPKINS

COLOR OF OIL AND MEAL COMMITTEE REPORT

BY DAVID WESSON

During the past year the chief efforts of the Committee have been to find out by means of comparative work whether the Eastman colorimeter can be made to supersede the Lovibond glasses and the type of tintometer now being used with artificial light.

Along in December, Mr. Morrison was requested to send several samples obtained from coöperative work to several laboratories which possessed Eastman colorimeters. The plan was for each laboratory to read the samples and then send them to the next laboratory, so that all the readings might be made on the same identical samples. Instructions were sent out to read the samples at the same time with the Lovibond glasses and report them. Owing to various causes the samples started on their rounds in December, did not reach the Chairman of this Committee until some time in March. The following tabulation of results shows big differences between different laboratories whether they work with the Lovibond glasses or with the Eastman colorimeter:

Description of sample	Lovibond reading		Eastman readings		
	Lovibond marked Red	Lovibond found Red	Red	Yellow	Neutral
Analyst 2, P. and G. Dallas					
Oil No. 3					
P. and G.....	16	15	41	50	36
Armour and Co.....	..	14	48.9	50	35.1
Southern Cotton Oil, Savannah.....	39.4	50	35.6
Wesson, New York.....	..	13.3	38.8	50	29.3
Analyst 4, Tilson Lab. Houston					
Oil No. 3					
P. and G.....	16	16.2	42	50	33
Armour and Co.....	..	13.6	49.3	50	35.3
S. C. O. Co., Savannah.....	39.8	50	34.8
Wesson, New York.....	..	13.5	39	50	28
Analyst 27, Swift and Co., Chicago					
14° Lye, Oil No. 3					
P. and G.....	14.2	16.8	41	50	36
Armour and Co.....	..	13.6	48	50	34.6
S. C. O. Co., Savannah.....	38.6	50	34.4
Wesson, New York.....	..	13.3	39.4	50	28