Oil Chemists' Official Committee Reports

Full Texts of Findings of Many Committees to be Presented at the Annual Convention, May 13 and 14

Report of F. F. A. Committee

DURING recent years there have been but few cases where buyer's and seller's chemists have not agreed on the free fatty acid content of crude oils. And so it was felt that the method of analysis for free fatty acid of crude oils described in Sec. 4, Rule 272, was giving satisfaction, and needed no investigation. In the case of refined oils there have been many differences over the free fatty acid content. The nature of the complaints have been such as to indicate that the method as written under Rule 274, Section 4, does not yield results of sufficient accuracy on refined oils of low acidity. So the work of the committee confined itself to finding the cause of the discrepancies, and to evolving a method which should so specify the minute details of the simple titration as to yield results reproducible on the same oil in different laboratories.

At the start, the opinions of the committee members were compiled for the information of all on the committee. It was found that the committee members did not agree on the details, and so a method was drafted which would clarify some of the details of the published method, and not conflict with the assembled opinions of the individual committee members. Four check samples were first sent out by the members to each other; a sample of refined cottonseed oil, two samples of refined corn oil from different makers, and a sample of P.S.Y. cottonseed. No other freshly refined vegetable oils seemed to be readily available. The results on the check samples, and an independent investigation of some of the details, brought out the following points:—

(1) Results are as accurate with tenth normal sodium hydroxide as with twentieth normal.

(2) A sample of 56.4 grams gives a slightly lower result than a sample of 28.4 grams, indicating that not only is the percentage effect of any contaminants reduced, but also the effect of the variation in color at start and finish of the titration is lessened.

In this connection it was found that where the same stopper and flask is used repeatedly without cleaning, that the few drops of oil remaining in the flask sets off the pink color by identical backgrounds at the start and finish of the titration.

(3) It makes no difference whether an Erlenmeyer flask or four ounce bottle be used in the titration except that a four ounce bottle is a bit small for a sample as large as 56.4 grams.

(4) Though it is usually more convenient to pipette a sample, the rules should specify a weighed sample for the sake of accuracy.

(5) Investigation disclosed the fact that an ordinary cork itself when cut in small pieces and digested in hot formula 30 alcohol will require some alkali for neutralization, doubtless due to the presence of a slight amount of resins. For that reason either a rubber stopper or a cork that has been seasoned should be used where great accuracy is desired.

(6) Hot titration is much better than cold titration. The temperature of the water bath must be below the boiling point of the alcohol. For that reason the temperature of the water bath in which the flask is warmed should be about 60° to 65° C. When the titration is made hot the time for the settling of the oil is reduced, and a more definite determination of the first appearance of a permanent pink color is obtained to match the pink tint existing at the start of the titration.

* * *

The method was then modified as given later in the report in accordance with the above points, and another check sample of salad oil was distributed to the committee members for titration. The results were as follows:

SAMPLE NO. 5

Trial	Result	Member
1	.030	W. H. D.
2	.032	"
3	.030	"
4	.034	"
1	.0312	J. D. E.
1	.030	R. C. H.
2	.0275	"
3	.025	"
4	.025	"
5	.020	"
6	.030	"
1	.0275	R. U. N.
2	.0250	"
3	.0257	"

1	.019	M. M. D. and G. W. F.
2	.020	
3	.020	
4	.020	"
5	.022	"
6	.022	"
7	.024	"

Grand Average:—.0257%
Greatest Variation from Average:— .0083%

The greatest variation from the average result was less than .01%. Two of the members obtained checks within .0001% of each other.

In order to make a further test of the method four different samples were made up using fresh corn salad oil and enough olive oil elaine of about 95% oleic acid content to slightly alter the free fatty acid. The samples were filled into four ounce bottles and marked A, B, C, D, E, F, G, and H. The analyst making the titration by the tentative method had no idea as to which two, if any, were duplicates.

The results were as follows:

	Duplicates		Error
A	.018%;	G .015%	.003%
B	.022%;	F .023%	.001%
C	.022%;	H .020%	.002%
D	.069%;	E .066%	.003%
	Average Error		.002%
	Maximum Error		.003%

Apparently the method of titration will give results reproducible to within a few thousandths of one percent, which should be close enough for all practical purposes.

It is recommended by your committee that the official method for the determination of free fatty acid in refined oils be altered to read substantially as follows:

"Put about 50 ml. of alcohol (Formula 30) into a clean dry 150 ml. flask and add a few drops of refined oil. Add 2 ml. of 1% phenolphthalein solution and place in water at 60 to 65° C until warm, and titrate with O.1N NaOH solution shaking vigorously until a faint permanent pink color is obtained. Weigh 56.4 gm. of the refined oil into the neutralized alcohol and titrate with occasional warming and violent shaking of the mixture until a faint permanent pink color is obtained in the supernatant alcohol of the same intensity as before adding the oil. Multiply the number of ml of O.1N NaOH by 0.05 and report as percentage free fatty acid expressed as oleic."

M. M. DURKEE, *Chairman*
R. U. NORRIS
W. H. DICKHART
J. D. EVANS
R. C. HATTER

Moisture Committee Report

The Moisture Committee this year did not send out any cooperative samples. The oven and method recommended by the Moisture Committee were adopted last year by the American Oil Chemists' Society but through an oversight the method and oven were not referred to the Chemists' Committee of the Cottonseed Crushers' Association and therefore it was not compulsory for various laboratories to use the oven during the past season. There were, however, some laboratories which purchased the oven and used it but unfortunately specifications as furnished by the Moisture Committee were not strictly adhered to and in some instances the ovens furnished certain laboratories leaked and were unsatisfactory. This was due principally to two things; first, the specification as to the weight of metal to be used was not followed, and second, the seams were not brazed. The Committee took these matters up with the manufacturers and we now have assurance that these ovens in the future will be brazed and will be absolutely guaranteed against leaks. The manufacturers claim, however, that it will be necessary to increase the price $25.

Although the Committee did not send out any cooperative samples, the Chairman was furnished with the numbers of three laboratories which handled the Smalley check meal samples in these ovens. The tabulation given below shows the results obtained.

	COLLABORATOR		
Sample	No. 1	No. 2	No. 52
1.	7.87	7.91	7.85
2.	9.35	——	——
3.	8.80	——	8.63
4.	8.20	——	7.91
5.	8.55	——	8.47
6.	8.31	8.09	7.62
7.	7.91	7.90	8.33
8.	7.42	7.63	7.68
9.	6.79	6.74	6.61
10.	7.14	6.90	6.93
11.	6.61	6.68	6.59
12.	6.84	6.64	6.45
13.	6.80	6.78	6.50
14.	6.35	6.38	6.28
15.	6.24	6.20	——
16.	6.32	6.32	6.38
17.	6.31	6.24	6.16
18.	6.05	6.06	6.07
19.	5.60	5.86	6.21
20.	6.05	6.10	6.35
21.	6.05	6.18	6.65
22.	6.56	6.52	7.31
23.	5.58	6.18	5.89
24.	6.01	6.04	6.02
25.	6.14	6.26	6.30
26.	6.82	6.59	6.87
27.	6.28	6.24	6.41
28.	6.41	6.30	6.36
29.	6.11	6.40	6.30
30.	6.90	6.40	6.74

These results are remarkably close in the opinion of the Committee and further confirm the value of this oven and method in obtaining uniform results. Next year the Committee hopes to have more data to present because the method and oven have been approved by the Chemists' Committee and many more laboratories will doubtless be equipped with this oven.

W. H. IRWIN, *Chairman*

Report of Seed Analysis Committee

A large amount of work has been done by the Members of this Committee in an effort to find whether the results obtained by the present Official Method were true indications of the value of the seed. However, this work has not been carried to a point where any definite proposal to change the Method or substitute another can be made. There are two points, however, where the Committee feels that correction in the present method of calculating the yields should be made.

FIRST: A correction of about two-tenths of one percent or four pounds of oil should be deducted from the total oil shown to take care of the oil left in the hulls.

SECOND: A uniform basis should be adopted upon which all seed should be calculated where the foreign matter determination has not been made due either to a small sample or because it has not been requested. This basis should be clearly shown on the report.

Shortly after our work was started the Department of Agriculture appointed a Committee to investigate the whole question of cottonseed sampling, grading and analysis, and it was the unanimous opinion of our Committee that we could best serve the Industry by turning over all our results to this Department of Agriculture Committee and offering them our fullest cooperation in every way. This has been done and should the Society wish to continue the Committee or appoint a new one we feel that the Members will always be ready to try out any suggested improvements in the present method or any new method that may lead to a better evaluation of cottonseed.

C. H. COX, *Chairman*

April 22, 1929.

Report of Methods Committee

The work of rewriting the methods was begun last year by a Committee of which Mr. Cluff was Chairman, but owing to Mr. Cluff's illness he asked the writer to undertake the rewriting and publication of the methods. This work has been completed and the methods have been published in loose leaf form by the Lefax Company, at an approximate expense to the Society of $760 for 2,500 sets of methods and 100 leather binders. The methods as published are strictly chemical and physical methods and contain no references to the Rules of The Cottonseed Crushers' Association. In addition to the methods printed in the Chemists' Chapter of the Rules of The Cottonseed Crushers' Association, the methods as published contain all of the methods of the Fat Analysis Committee of the American Oil Chemists' Society and the American Chemical Society revised to January 1, 1929.

These methods may be obtained in pamphlet form through the Secretary of The Society for 50 cents each or may be obtained in a leather back loose leaf binder of convenient size for $1.50. We have also made arrangements with the Lefax Company so that in the future will print 2,500 single sheets at a cost of $15.25 for straight printed matter or $19.75 for tabular matter. This will enable The Society to keep the methods up to date, insert new methods, etc., at a very small cost. The Committee wishes to urge upon each member of The Society that he purchase at least one or more of these sets.

W. H. IRWIN, *Chairman*

Schwartz Laboratories, Inc., Chemists and Engineers, announce the removal of their offices and laboratories from 113 Hudson Street to 202 East 44th Street, New York City.

The Industrial Chemical Sales Company, manufacturers of Nuchar, announce the issue of a new booklet dealing with the application of activated carbons to the refining and bleaching of vegetable oils, fats and greases. They will gladly mail a copy of the booklet to anyone interested. Their address is 230 Park Avenue, New York City.

The Supreme Court of Georgia has ruled that the state chemist cannot give a certificate of an analysis of a fertilizer sample for use as evidence in a court, unless that sample actually has been taken from the fertilizer at issue by a qualified state inspector. The decision was in a case of the Southern Cotton Oil Co. against T. B. Raines, appealed from a district superior court.

The New York Burlap & Jute Exchange, organized recently to provide an exchange trading in future contracts in burlaps, jute and other fibres is reported to be nearly ready for its official opening.