Report of the Smalley Foundation Committee
1945-1946

WE are presenting herewith the 28th report of the Smalley Foundation Committee of the American Oil Chemists' Society. During these past twenty-eight years considerable progress has been made in the accuracy of the determination of Oil and Nitrogen on cottonseed meal. The results obtained in practically all determinations were slightly lower than last year. It must be understood, in gauging the accuracy of the results a difference of two points in either direction from the average is permitted without a deduction from the grade. We might add that the results obtained are so nearly perfect that a few hundredths of a per cent higher or lower than in any previous year means very little as far as accuracy is concerned.

As usual, thirty samples of cottonseed meal were distributed to the collaborators.

There are attached to this report four tables indicating the standing in percentage of the members taking part. Table No. I gives the standing of 49 collaborators who reported oil determinations on all samples. Table No. II gives the standing of 55 collaborators who reported nitrogen results on all samples. Table No. III gives the standing of 49 collaborators who reported oil and nitrogen on all samples. In these tables we have taken into consideration the results of those reports which were received within the time specified in our original announcement of the Smalley Foundation work. In Table No. IV we have given the standing of those collaborators who reported on all samples, but some of whose reports were received too late to be included under the rules.

TABLE NO. I.

Determination of Oil.

Analyst No.	Points Off	Per Cent Efficiency
56	7	99.963
43	9	99.952
32	13	99.931
24-41	14	99.925
31-47	15	99.920
2-49	22	99.883
25	26	99.861
6	28	99.851
11-74	33	99.824
9	37	99.803
8-21-82	38	99.797
35	39	99.792
57	41	99.781
7-26-45	46	99.755
53	48	99.744
59	50	99.733
40	52	99.723
23	53	99.717
10-22	54	99.712
13-33	61	99.675
60	66	99.648
27	70	99.627
34	74	99.605
12	75	99.600
4	78	99.584
67	80	99.573
42	81	99.568
30	82	99.563
66	94	99.499
29	95	99.493
5-58	97	99.483
37	98	99.477
3	102	99.456
77	107	99.429
54	117	99.376
36	122	99.349
72	127	99.323
71	161	99.141

TABLE NO. II.

Determination of Nitrogen.

Analyst No.	Points Off	Per Cent Efficiency
6	5	99.975
43	7	99.966
8-24	8	99.960
32-56	9	99.955
49	10	99.951
31-63-66	11	99.945
13-40	12	99.940
15	13	99.936
59	15	99.925
37	16	99.921
9-25-26-74	17	99.915
10-21-54	18	99.910
47	20	99.900
12	22	99.891
53	24	99.880
41-82	26	99.870
57	29	99.855
1-5	31	99.846
4	32	99.840
42	33	99.836
22	34	99.831
36-45	37	99.816
3	38	99.810
11	41	99.795
77	45	99.776
62-67	52	99.742
2	53	99.736
7-27	54	99.731
64	55	99.727
23	59	99.706
34	60	99.701
29	66	99.671
58	73	99.637
72	78	99.612
30	82	99.592
71	83	99.586
33	85	99.577
35	92	99.541
60	104	99.482
39	115	99.428

For the first time in the history of the check meal work of the Smalley Foundation Committee two collaborators tied for the highest efficiency in the determination of both oil and nitrogen. It will probably be necessary for them to share possession of the cup for six-month periods during the next year as no other provision has been made for a situation of this kind.

The winning collaborators are as follows:

The "American Oil Chemists' Society Cup" for the highest efficiency in the determination of both Oil and Nitrogen on all samples is awarded to Analysts No. 43 and 56, Russell Haire, Planters Manufacturing Company, Clarksdale, Miss., and L. B. Forbes, L. B. Forbes Laboratories, Little Rock, Ark., with an average of 99.959 per cent. The average efficiency is higher than that of last year, which was 99.957 per cent. The certificate for second place goes to Analysts 24 and 32, E. H. Tenent, Woodson-Tenent Laboratories, Mem-

phis, Tenn., and D. B. McIsaac, Kershaw Oil Mill, Kershaw, S. C., who had an efficiency of 99.943 per cent, as compared with 99.950 per cent for last year.

The certificate for the highest efficiency in the determination of Oil only is awarded to Analyst No. 56, L. B. Forbes, L. B. Forbes Laboratory, Little Rock, Ark., with an average of 99.963 per cent, as compared with 99.973 per cent for last year. The certificate for second place goes to Analyst No. 43, Russell Haire, Planters Manufacturing Company, Clarksdale, Miss., with an efficiency of 99.952 per cent as compared with 99.937 per cent for last year.

The certificate for the highest efficiency in the determination of Nitrogen is awarded to Analyst No. 6, T. L. Rettger, Buckeye Cotton Oil Company, Memphis, Tenn., with an average of 99.975 per cent as compared with 99.976 per cent for last year. The certificate for second place goes to Analyst No. 43, Russell Haire, Planters Manufacturing Company, Clarksdale, Miss., with an average of 99.966 per cent, as compared with 99.972 per cent for last year.

The moisture results obtained on the Smalley Foundation check meal samples have always been very erratic. In our last report we suggested that perhaps some incentive should be given in the form of a certificate to those who obtain the best results of this determination. We did not press this matter at the time because we understood that a committee had been appointed to study the subject of moisture in seeds and cottonseed meal. This committee has now turned in a report and if this is approved at the present meeting we would suggest that a definite method be given to all collaborators for determining moisture and that a certificate be awarded to those who excel in this work.

TABLE NO. III.

Determination of Oil and Nitrogen.

Analyst No.	Per Cent Efficiency
43-56	99.959
24-32	99.943
31	99.933
49	99.917
6	99.913
47	99.910
41	99.898
25	99.888
8	99.879
74	99.870
9	99.859
21	99.854
26	99.835
82	99.834
40	99.832
59	99.829
57	99.818
53	99.812
10	99.811
2-11	99.810
13	99.808
45	99.786
22	99.772
12	99.746
7	99.743
66	99.722
4-23	99.712
42	99.702
37	99.699
27	99.679
35	99.667
5	99.665
67	99.658
34	99.653
54	99.643
3	99.633
33	99.626
77	99.603
36	99.583
29	99.582
30	99.578
60	99.565
58	99.560
72	99.468
71	99.364

TABLE NO. IV.

Special Table.

Analyst No.	Points Off	Per Cent Efficiency
Determination of Oil		
28	32	99.829
76	113	99.397
Determination of Nitrogen		
28	24	99.880
19	37	99.816
69	41	99.795
76	45	99.776
14	119	99.407
Determination of Oil and Nitrogen		
28		99.855
76		99.587

For many years Mr. Thos. C. Law has prepared and distributed our samples at considerable inconvenience to himself. We again call attention to this as we feel that the Smalley Foundation Committee and the American Oil Chemists' Society should realize the tremendous contribution which he is making toward the success of this collaborative work.

We are again including in this report a list of the previous winners of the highest award for both oil and nitrogen. They are as follows:

1918-1919—G. C. Hulbert, Southern C. O. Co., Augusta, Ga.
1919-1920—G. C. Hulbert, Southern C. O. Co., Augusta, Ga.
1920-1921—C. H. Cox, Barrow-Agee Lab's., Memphis, Tenn.
1921-1922—Battle Lab's., Montgomery, Ala.
1922-1923—Battle Lab's., Montgomery, Ala.
1923-1924—L. B. Forbes, Memphis, Tenn.
1924-1925—E. H. Tenent, International Sugar Feed Co. No. 2, Memphis, Tenn.
1925-1926—Battle Lab's., Montgomery, Ala.
1926-1927—W. F. Hand, Miss. State College, State College, Miss.
1927-1928—E. H. Tenent, International Sugar Feed Co., Memphis, Tenn.
1928-1929—Geo. W. Gooch Lab's., Los Angeles, Calif.
1929-1930—Southwestern Lab's., Dallas, Texas.
1930-1931—W. F. Hand, Miss. State College, State College, Miss.
1931-1932—J. N. Pless, Royal Stafolife Mills, Memphis, Tenn.
1932-1933—D. B. McIsaac, International Veg. Oil Co., Savannah, Ga.
1933-1934—W. F. Hand, Miss. State College, State College, Miss.
1934-1935—W. F. Hand, Miss. State College, State College, Miss.
1935-1936—N. C. Hamner, Southwestern Lab's., Dallas, Texas.
1936-1937—N. C. Hamner, Southwestern Lab's., Dallas, Texas.
1937-1938—W. F. Hand, Miss. State College, State College, Miss.
1938-1939—W. F. Hand, Miss. State College, State College, Miss.
1939-1940—A. G. Thompson, Jr., Southern C. O. Co., Columbia, S. C.
1940-1941—Russell Haire, Planters Mfg. Co., Clarksdale, Miss.
1941-1942—T. L. Rettger, Buckeye Cotton Oil Co., Memphis, Tenn.
1942-1943—Barrow-Agee Lab's., Memphis, Tenn.
1943-1944—D. B. McIsaac, Kershaw Oil Mills, Kershaw, S. C.

1944-1945—W. W. Wynn, Jr., Barrow-Agee Lab's., Cairo, Ill.
1945-1946—L. B. Forbes, L. B. Forbes Lab's., Little Rock, Ark.
Russell Haire, Planters Mfg. Co., Clarksdale, Miss.

R. R. HAIRE
F. F. HASBROUCK
L. H. HODGES
T. C. LAW
R. C. POPE
J. J. VOLLERTSEN, chairman.

CORRECTION

The words linoleic and linolenic were interchanged in the equations and sample calculations shown on page 142 of the May, 1946 issue of Oil & Soap in the article on "Applied Ultraviolet Spectrophotometry of Fats and Oils," according to the author, B. W. Beadle, American Meat Institute, Chicago.

Comparison of a Simplified, Quantitative Kreis Test With Peroxide Values of Oxidizing Fats*

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THE two chemical tests most widely employed in recent years to indicate rancidity in fats are (1) estimation of the peroxides by titration of the iodine liberated and (2) estimation of the color produced by reaction between oxidizing fats and phloroglucinol in acid solution (Kreis test).

Earlier work on the Kreis test has been well summarized by Lea (1). One of the chief objections to the test in its original form is the fact that the color development took place in a two-phase system and that the color was often distributed between the acid and the ether phases, in an irregular manner. Attempts to make the test quantitative in this form were not highly satisfactory (2). Walters, Muers, and Anderson (3) succeeded in developing the color in a single phase by dissolving the phloroglucinol in amyl acetate and substituting trichloracetic for hydrochloric acid. They found the test to be highly sensitive as compared to the older Kreis method and subject to quantitative measurement in a Zeiss-Pulfrich photometer.

White (4) compared the Walters, Muers, and Anderson method with peroxide oxygen values and with other methods of estimating rancidity on bacon fat. He found that the modified Kreis test was the most sensitive, gave excellent precision, and best correlation with the peroxide value. However, he concluded that the peroxide test was somewhat easier to apply.

Pool and Prater (5) developed a simplified modification of the Walters, Muers, and Anderson procedure, using glacial acetic acid in place of amyl acetate. The essential features of their method were obtained by private communication some months before its publication. In the present paper the simplified procedure has been applied to a number of oxidizing fats, and the results have been compared with peroxide values and iodine numbers on the same fats.

Analytical Methods

Peroxide Value. The Wheeler method (6) was used with minor modifications. The results were found to be more reproducible if the period of standing was increased to 10 minutes before titrating with thiosulfate. Stansby (7) has emphasized the importance of using the same weight of fat in this test; 0.5 gm. were used throughout. The peroxide numbers were expressed as millimols of peroxide per 1,000 gm. oil, i.e., peroxide number =

$$\frac{0.5\ (\text{ml. thio. used in titration})\ (\text{normality of thio.}) \times 1{,}000}{\text{wt. of fat}}$$

Iodine Numbers. The Hanus method as outlined by Woodman (8) was used.

Modified Kreis Test. Since this test as carried out differed in a number of minor details from that later published by Pool and Prater (5), the exact procedure used is described here.

Reagents. (A) Thirty gm. trichloracetic acid plus 100 ml. glacial acetic acid. (B) One gm. phloroglucinol plus 100 ml. glacial acetic acid (see note 6 on the method).

Procedure. Aliquots of chloroform solutions containing 0.2 gm. of fat (see Note 3) were transferred to colorimeter tubes and made up to 3 ml. with chloroform. Six ml. of reagent A and 1 ml. reagent B were added, the tube shaken (note 5), allowed to stand exactly 15 minutes at 37° C. (note 1), and cooled 3 minutes. A blank containing the fat but omitting the phloroglucinol and using 7 instead of 6 ml. of reagent A was prepared at the same time and treated in the same way (note 2).

The color was read in an Evelyn photoelectric colorimeter, using a 540 filter and setting the instrument at 100% transmission with the blank. The Kreis value was expressed as the optical density (i.e., 2-log galvanometer reading) divided by .02 (the concentration of fat in gm. per ml. of solution in the colorimeter tube).

NOTES ON THE KREIS METHOD

1. *Time and Temperature of Color Development.* The conditions for color development specified above were chosen arbitrarily. As pointed out by Walters, *et al.* (3) the red color does not reach a maximum at any time or temperature but changes gradually to a yellow color. Hence, the absolute values recorded for the Kreis test depend upon the time and temperature chosen for color development. The temperature effect

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