

absorption peak at 281  $m\mu$  which was then used as a basis for analysis ( $E_{1cm}^{1\%}$  for the sample used was 116). After water partition 91% of the original dissolved guaiac still remained in the fat phase. Again this relative insolubility in water correlates well with the carry-over of its antioxidant effect to baked goods (Table I). It should be pointed out in order to explain the apparent discrepancies between these and previously reported stability data for gum guaiac (1) that in the present work the antioxidant was added merely by stirring it into warm lard. In the work of Higgins and Black (1) the gum was incorporated into deodorized lard by means of an acetic acid solution which greatly enhances its effectiveness (8). Failure to recognize this effect may explain why various investigators have obtained unsatisfactory results when using gum guaiac to stabilize lard.

6. N.D.G.A. was also determined on the basis of its absorption curve which peaked at 283  $m\mu$  ( $E_{1cm}^{1\%} = 188$ ). Again, its 90% retention in the fat phase correlated with its carry-over into pie crusts (Table I).

### Discussion

The data assembled in Table I appear to substantiate the hypothesis of Richardson, Grettie, and Newton that phenolic antioxidants which are relatively less soluble in water than in fat on a partition basis such as found in a bakery composition will carry their stabilizing properties into the fat in the baked goods; conversely, those antioxidants which were more soluble in water evidently are extracted from the fat phase and consequently are unable to exert their stabilizing effect in the final product.

On this basis a laboratory test is proposed for the evaluation of the probable baking carry-over of an antioxidant, to be used as a substitute for baking tests when the latter are not feasible or practicable. The test is based upon the partition of the antioxi-

dant between the fat in which it is dissolved and an equal volume of hot water. If, after thorough agitation of the two phases, a relatively large proportion of the antioxidant remains in the fat phase, it can then be assumed that the antioxidant will carry its effectiveness into baked goods, providing, of course, that it is not destroyed by heat or by one of the constituents of the baking composition.

For most phenolic antioxidants ultraviolet absorption spectroscopy appears to be the most convenient method for the determination of the amount of antioxidant in the two phases. When a spectrophotometer is not available, colorimetric analyses may readily be developed, as was shown above for gallacetonein.

### Summary

Verification is offered for the hypothesis that the ability of an antioxidant to stabilize the fat in baked goods is a function of its solubility characteristics. Thus, to be effective in baked goods, an antioxidant may not be sufficiently more soluble in water than in fat that it will be washed out of the fat by the moisture in the other ingredients. On the basis of this observation a laboratory method involving the partition of antioxidants between equal amounts of fat and hot water has been developed. Good correlation has been found between this laboratory test and actual bakeshop results.

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## Report of the Smalley Foundation Committee 1946-1947

**A** YEAR ago the Smalley Foundation Committee was enlarged to include all types of collaborative work to improve analytical results. As a result it was divided into three sub-committees:

1. Sub-Committee on Oilseed Meal, R. W. Bates, chairman
2. Sub-Committee on Crude Vegetable Oils, A. S. Richardson, chairman
3. Sub-Committee on Oilseed, R. T. Doughtie, Jr., chairman

Each of these sub-committees has worked faithfully in getting out samples and checking results, and it was thought best to allow each sub-committee chairman to make a report of the work of his group at this time. As chairman of the Smalley Foundation Committee, I want to express my thanks and appreciation for the careful work done by each of these sub-committees.

J. J. VOLLERTSEN, general chairman.

**W**E are presenting herewith the 29th report of the Sub-Committee on Oilseed Meal of the Smalley Foundation Committee of the American Oil Chemists' Society. During these past twenty-nine 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 higher than last year.

As usual, 30 samples of cottonseed meal were distributed to the collaborators. Last year we recommended that, in order to obtain better results in the determination of moisture, certificates be awarded to the collaborators having the highest and next highest averages in the work for the year.

There are attached to this report five tables indicating the standing in percentage of the members taking part. Table No. I gives the standing of 48 collaborators who reported moisture determinations on all samples. Table No. II gives the standing of 49 collaborators who reported oil results on all samples. Table No. III gives the standing of 52 collaborators who reported nitrogen on all samples. Table No. IV gives the standing of 49 collaborators who reported on 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 Founda-

TABLE NO. I.  
Determination of Moisture.

Analyst No.	Points Off	Per cent Efficiency
27.....	18	99.912
16.....	34	99.834
20.....	39	99.809
11.....	48	99.765
38.....	80	99.609
28.....	84	99.590
50.....	90	99.560
40.....	95	99.535
13-16.....	96	99.531
47.....	105	99.487
10.....	109	99.468
24.....	116	99.433
17.....	123	99.399
45.....	129	99.370
42.....	130	99.365
44.....	133	99.351
55.....	135	99.340
54.....	138	99.326
1.....	142	99.307
52.....	143	99.301
33.....	144	99.296
62.....	149	99.271
34.....	152	99.257
32.....	161	99.213
29.....	166	99.189
7.....	169	99.175
6.....	170	99.169
19.....	193	99.057
4.....	198	99.032
26.....	221	98.920
48.....	235	98.852
41.....	236	98.846
21.....	237	98.842
22.....	240	98.827
49.....	248	98.788
23.....	257	98.743
36.....	306	98.505
15.....	319	98.442
30.....	376	98.163
78.....	439	97.855
66.....	503	97.543
69.....	531	97.405
56.....	538	97.371
39.....	804	96.071
46.....	988	95.172
64.....	1279	93.750
51.....	1504	92.651

tion work. In Table No. V we give 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.

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 Analyst No. 13, Russell Haire, Planters Manufacturing Company, Clarksdale, Miss., with an average of 99.972%. According to our rules, when a collaborator attains the highest average on three different occasions, the cup becomes his permanent property. As this is the third time Russell Haire has been in this position the cup will be turned over to him at this meeting. The average efficiency is higher than that of last year, which was 99.959%. The certificate for second place goes to Analyst No. 4, Thomas Weiss, Chickasha Cotton Oil Laboratory, Chickasha, Oklahoma, who has an efficiency of 99.941%, as compared with 99.943% for last year.

The certificate for the highest efficiency in the determination of moisture is awarded to Analyst No. 27, G. K. Witmer, Battle Laboratories, Montgomery, Ala., with an average of 99.912%. The certificate for second place goes to Analyst No. 16, H. M. Bulbrook, Industrial Laboratories, Fort Worth, Texas, with an average of 99.834%.

The certificate for the highest efficiency in the determination of oil is awarded to Analyst No. 27, G. K. Witmer, Battle Laboratories, Montgomery, Ala., with an average of 99.968%. The certificate for second place goes to Analyst No. 13, Russell Haire, Planters Manufacturing Company, Clarksdale, Miss.,

with an efficiency of 99.963%. It should be borne in mind that a  $\pm 0.03\%$  tolerance was used on the determination of oil this year instead of  $\pm 0.02\%$  used in the past. Actually the results on oil are poorer than last year. The two leading collaborators last year were off 7 and 9 points. If  $\pm 0.02\%$  tolerance had been used this year the winning collaborators would be off 12 and 17 points. We feel that the use of the increased tolerance has brought the nitrogen and oil results more nearly into balance.

The certificate for the highest efficiency in the determination of nitrogen is awarded to Analysts No. 13 and 42, Russell Haire, Planters Manufacturing Company, Clarksdale, Miss., and T. L. Rettger, Buckeye Cotton Oil Company, Memphis, Tenn., who also won this award last year, with an average of 99.980% as compared to 99.975% for last year. The certificate for second place goes to Analyst No. 38, P. D. Cretien, Texas Testing Laboratories, Dallas, Texas, with an average of 99.974%, as compared with 99.966% for last year.

While the results on moisture are an improvement over what they have been heretofore, they are not entirely satisfactory. We selected samples 3, 5, 10, 16, 22, 28, and 29 entirely at random and determined the percentage of the results that were within the  $\pm 0.1\%$  tolerance. On sample No. 3 only 30.1% were within the tolerance while on sample No. 28, 62.5% were within the tolerance. This improvement was gradual through samples 5, 10, 16, and 22. No. 29 compared favorably with No. 28. It can thus be readily seen that improvement has been shown.

We believe that moisture determinations, more than oil and nitrogen, are greatly dependent on the analyst "knowing his oven" rather than on the type of oven used (this of course, within reasonable limits).

We made another approach to the study of improvement whereby we selected samples No. 3 and 28

TABLE NO. II.  
Determination of Oil.

Analyst No.	Points Off	Per cent Efficiency
27.....	6	99.968
13.....	7	99.963
4.....	8	99.957
11.....	13	99.932
7-34.....	14	99.925
47.....	19	99.900
21.....	22	99.884
41.....	24	99.873
23-38.....	27	99.857
30-45-62.....	28	99.852
6-20.....	29	99.846
10-24.....	30	99.841
22.....	32	99.830
78.....	34	99.820
32.....	41	99.782
19-46.....	42	99.777
33.....	48	99.746
16.....	51	99.730
55.....	53	99.719
15.....	58	99.693
48.....	59	99.687
42.....	60	99.682
28.....	61	99.677
57.....	65	99.655
40.....	71	99.623
29-50.....	73	99.614
52.....	78	99.587
66.....	86	99.544
54.....	87	99.539
39.....	88	99.534
60.....	89	99.558
26.....	90	99.523
36.....	105	99.443
69.....	119	99.369
17.....	120	99.364
44.....	131	99.305
49.....	144	99.237
56.....	146	99.226
1.....	175	99.073
51.....	180	99.046
64.....	214	98.866

TABLE NO. III.  
Determination of Nitrogen.

Analyst No.	Points Off	Per cent Efficiency
13-42.....	4	99.980
38.....	5	99.974
20.....	7	99.965
32-55.....	9	99.955
7-47.....	10	99.950
24.....	12	99.940
34.....	14	99.929
4.....	15	99.925
78.....	17	99.914
27.....	18	99.910
64.....	20	99.899
66.....	24	99.880
40.....	25	99.875
11-45.....	26	99.869
6-46-57.....	27	99.865
48.....	28	99.860
22.....	36	99.819
39-62.....	37	99.815
23.....	38	99.809
28-33-60.....	39	99.804
50.....	40	99.800
76.....	41	99.794
36.....	44	99.779
16-35-44.....	45	99.774
69.....	46	99.770
17.....	50	99.749
29.....	52	99.740
19.....	63	99.684
52.....	64	99.680
21.....	65	99.674
49.....	69	99.654
15.....	70	99.649
71.....	73	99.634
1.....	74	99.628
30.....	84	99.579
10.....	87	99.564
26.....	93	99.534
54.....	96	99.519
41.....	111	99.443
56.....	125	99.373
51.....	177	99.112

and calculated the Standard Deviation of the mean. The Standard Deviations on these two samples were as follows (Analyst 77 on No. 3 was eliminated):

No. 3—Standard Deviation.....0.214%

No. 28—Standard Deviation.....0.208%

Thus one of the better and poorer samples show about the same deviation. This is due to the fact that on No. 3 most collaborators missed the accepted average by a few tenths, but only 20 were within 0.1% and on No. 28 forty collaborators were within 0.1% of the average, but the 24 who were not within this limit missed by a greater margin. We do not believe that the use of the Standard Deviation is a practical measure of improvement.

We attempted to correlate the various standings with the type of oven used. We believe that the results would be more interesting if we had received replies from all collaborators on the type of oven in use. There seemed to be three general types of response:

1. Use of forced draft oven
2. Use of air oven (gravity convection)
3. No response or information.

It is interesting to note that in the first ten all reporting used a forced draft oven. Analyst No. 28 (7th) and No. 50 (8th) did not indicate oven used.

In the last ten (ratings 47 to 56) 2 air ovens were used, 2 used a forced draft and 6 did not report the oven used.

To correlate type of oven used with results we should have a response from all concerned as to the type of oven used; we hope to obtain this information next year. We hope that greater attention will be paid by the collaborators to the moisture determination with a view to improving our results.

For many years Thomas 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. P. 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

TABLE NO. IV.  
Determination of Oil and Nitrogen.

Analyst No.	Per cent Efficiency
13.....	99.972
4.....	99.941
27.....	99.939
7.....	99.938
34.....	99.927
47.....	99.925
38.....	99.916
20.....	99.906
11.....	99.901
24.....	99.891
32.....	99.869
78.....	99.867
45.....	99.861
6.....	99.856
55.....	99.837
62.....	99.834
23.....	99.833
42.....	99.831
22.....	99.825
46.....	99.821
21.....	99.779
33.....	99.775
48.....	99.774
57.....	99.760
16.....	99.752
40.....	99.749
28.....	99.741
19.....	99.731
30.....	99.716
66.....	99.712
50.....	99.707
10.....	99.703
29.....	99.677
39.....	99.675
15.....	99.671
60.....	99.666
41.....	99.658
52.....	99.634
36.....	99.611
69.....	99.570
17.....	99.557
44.....	99.540
26-54.....	99.529
49.....	99.446
64.....	99.383
1.....	99.351
56.....	99.300
51.....	99.079

TABLE NO. V.  
Special Table.

Analyst No.	Points Off	Per cent Efficiency
Determination of Moisture		
9.....	30	99.853
2.....	119	99.418
8.....	145	99.292
61.....	191	99.066
14.....	217	98.940
3.....	246	98.783
58.....	272	98.670
68.....	295	98.559
63.....	540	97.361
Determination of Oil		
61.....	23	99.878
9.....	24	99.873
14.....	74	99.607
58.....	78	99.587
2.....	81	99.571
8.....	118	99.375
3.....	138	99.268
68.....	488	97.413
63.....	1230	93.480
Determination of Nitrogen		
8.....	7	99.965
9.....	12	99.940
3.....	24	99.880
61.....	26	99.869
58.....	29	99.854
2.....	34	99.830
14.....	67	99.665
68.....	96	99.519
63.....	314	98.425
Determination of Oil and Nitrogen		
9.....		99.907
61.....		99.874
58.....		99.721
2.....		99.701
8.....		99.670
14.....		99.636
3.....		99.574
68.....		98.466
63.....		95.953

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, Illinois

1945-1946 { L. B. Forbes, L. B. Forbes Lab's., Little Rock, Ark.  
          { Russell Haire, Planters Mfg. Co., Clarksdale, Miss.

1946-1947—Russell Haire, Planters Mfg. Co., Clarksdale, Miss.

R. R. HAIRE	THOMAS C. LAW
F. F. HASBROUCK	T. L. RETTGER
L. H. HODGES	R. W. BATES, chairman

## SUB-COMMITTEE ON OILSEEDS

During the season of 1946-47 the Sub-Committee on Oilseeds of the Smalley Foundation Committee offered check series on cottonseed, soybeans, and peanut samples. The series on cottonseed and on soybeans comprised of 10 samples each while the peanut series comprised of 7 samples. At the completion of each series grades were calculated for each collaborator, such grades being based on a scale of deductions on points outside of tolerances allowed and errors in reporting, to denote the degree of efficiency attained by the individual analyst.

The accompanying tables, Nos. 1-2-3, show the ratings of each of the collaborators on the particular series of samples reported. The identification numbers of the various chemists on each series are not identical, but are the numbers assigned to each collaborator for each separate series of samples.

It is interesting to note that no individual chemist attained the highest efficiency on more than one particular series, and only two chemists received a rating in the first three places on two of the series of samples.

On the cottonseed series, chemists Nos. 9-14-22-23 made grades of 100.00% to end in a four-way tie for first place. These chemists were E. H. Tenent, Memphis, Tenn.; R. C. Pope, Dallas, Texas; Thomas B. Caldwell, Wilmington, N. C.; and Thomas C. Law, Atlanta, Ga. Second place, with a grade of 99.04%, was attained by analyst No. 32, G. Worthen Agee, Memphis, Tenn., while third place went to analyst No. 10, W. N. Kesler, Little Rock, Ark., with a grade of 98.80%.

On the soybean series, first place, with a grade of 100.00%, was earned by chemist No. 25, Paul D. Cretien, Dallas, Texas; second place went to analyst No. 7, R. H. Fash, Fort Worth, Texas, with a grade of 99.10%; and third place, with a grade of 98.47%, was earned by chemist No. 22, G. K. Witmer, Montgomery, Ala.

On the peanut series, chemist No. 9, G. K. Witmer, Montgomery, Ala., made first place with a grade of 99.41%; second place went to chemist No. 7, N. C. Hamner, Dallas, Texas, with a grade of 98.91%; third place was attained by analyst No. 10, Thomas B. Caldwell, Wilmington, N. C., with a grade of 97.82%. Chemists Nos. 1 and 4 were not awarded grades on the peanut series due to a single error in the extreme

TABLE NO. 1  
(Cottonseed Grades)

Chemist No.	Grade	Rating
9-14-22-23.....	100.00	1
32.....	99.04	2
10.....	98.80	3
13.....	97.84	4
11.....	97.60	5
5.....	96.76	6
27.....	96.46	7
18.....	96.40	8
28.....	95.74	9
6.....	95.44	10
34.....	95.08	11
12.....	94.90	12
40.....	94.78	13
7.....	94.24	14
25.....	93.88	15
37.....	93.40	16
17.....	93.34	17
3.....	92.62	18
24.....	92.56	19
33.....	92.44	20
20-36.....	92.38	21
15.....	91.78	22
1-35.....	91.30	23
30.....	90.76	24
31.....	90.04	25
29.....	89.80	26
2.....	89.20	27
21.....	88.96	28
19.....	87.52	29
39.....	86.44	30
4.....	86.38	31
26.....	85.24	32
16.....	76.12	33
38.....	59.92	34
8.....	24.82	35

TABLE NO. 2  
(Soybean Grades)

Chemist No.	Grade	Rating
25.....	100.00	1
7.....	99.10	2
22.....	98.47	3
2-6-14-17.....	98.20	4
5-8.....	95.95	5
15.....	95.50	6
3-11-19.....	94.60	7
1.....	94.15	8
20-21.....	93.25	9
9.....	92.80	10
16.....	92.35	11
18.....	90.10	12
13.....	89.65	13
24.....	77.95	14
23.....	76.60	15
12.....	67.15	16
4.....	64.00	17
10.....	61.75	18
27.....	58.87	19
26.....	50.50	20

being made by each chemist on samples Nos. 3 and 1, respectively, which errors caused their calculated grades to be so low that it was not considered as indicative of their regular work as indicated by the results on the other samples of the peanut series.

On the cottonseed series,, 75% of the analysts maintained grades of 90.00% or better; on the soybean series 70.4% of the analysts made grades of 90.00%

or better; and on the peanut series only 57.1% of the analysts attained grades of 90.00% or better.

C. G. HENRY

R. T. DOUGHTIE, JR., chairman

SUB-COMMITTEE ON CHECK OIL SAMPLES

The first act of the newly appointed sub-committee was to poll the sentiment of the 1945-6 collaborators on the desired number of check-oil samples. Twenty-

TABLE NO. 3  
(Peanut Grades)

Chemist No.	Grade	Rating
9.....	99.41	1
7.....	98.91	2
10.....	97.82	3
14.....	97.35	4
8.....	97.27	5
12.....	95.97	6
3.....	92.23	7
11.....	90.09	8
13.....	80.13	9
2.....	79.84	10
5.....	77.19	11
6.....	75.89	12
1 } .....	.....*	.....*
4 } .....	.....*	.....*

\* See explanation in discussion of grades to account for no grades being awarded.

A.O.C.S. CHECK OIL SAMPLES—FINAL GRADES—SEASON 1946-47

Identification No.	Deduction, Points								Grade		
	Cottonseed Oil				Soybean Oil				C. S. O.	S. B. O.	Both Oils
	F. A.	Loss	Color	Total	F. A.	Loss	Color	Total			
1.....	0	.2	.19	.39	.3	0	0	.3 <sup>a</sup>	95.7	95.0 <sup>a</sup>	95.4
2.....	0	.2	0	.20	0	.5	.6	1.1	97.8	87.8	92.8
3.....	.3	1.0	0	1.30	0	0	0	0 <sup>a</sup>	85.6	100.0 <sup>a</sup>	92.8
4.....	.3	.9	.57	1.77	0	0	0	0 <sup>a</sup>	80.3	100.0 <sup>a</sup>	90.2
5.....	0	.2	.1	.30	0	.5	0	.5	96.7	94.4	95.6
8.....	0	0	0	0 <sup>b</sup>	.....	.....	.....	.....	.....	.....	.....
9.....	0	1.02	.2	1.22	0	.1	0	.1	86.4	98.9	92.7
10.....	.3	.2	.1	.60	0	.2	0	.2	93.3	97.8	95.6
11.....	0	.2	.1	.30	0	.4	0	.4	96.7	95.6	96.2
12.....	0	.54	.26	.80	0	.5	.1	.6	91.1	93.3	92.2
13.....	0	.2	.3	.50	0	.1	0	.1	94.4	98.9	96.7
14.....	0	.36	0	.36	0	.4	0	.4 <sup>a</sup>	96.0	95.6 <sup>a</sup>	95.8
16.....	0	.30	.75	1.05	0	0	0	0 <sup>b</sup>	83.3	.....	.....
17.....	.3	0	.51	.81	0	0	.2	.2	91.0	97.8	94.4
21.....	.3	.18	.06	.54	.6	.2	0	.8	94.0	91.1	92.6
23.....	0	0	.25	.25	0	.5	0	.5	97.2	94.4	95.8
25.....	.9	.5	.3	1.70	0	.4	.3	.7	81.1	92.2	86.7
27.....	0	.49	.13	.62	.....	.....	.....	.....	93.1	.....	.....
28.....	.6	.19	.8	1.59	0	.4	.4	.8	82.3	91.1	86.7
30.....	0	0	.33	.33	0	0	.5	.5	96.3	94.4	95.4
32.....	0	0	.1	.10	0	0	.2	.2	98.9	97.8	98.4
33.....	.3	.39	.65	1.32	0	.1	0	.1	85.3	98.9	92.1
35.....	.9	0	.59	1.49	0	.7	0	.7	83.4	92.2	87.8
40.....	0	.1	.2	.30 <sup>a</sup>	0	.3	1.0	1.3	95.0 <sup>a</sup>	85.6	90.3
50.....	0	0	.2	.20	0	.2	0	.2	97.8	97.8	97.8
51.....	1.0	.27	.38	1.65 <sup>b</sup>	0	.2	0	.2 <sup>b</sup>	.....	.....	.....
52.....	0	.99	.13	1.12	0	1.0	0	1.0 <sup>a</sup>	87.6	83.3 <sup>a</sup>	85.5
53.....	.6	.36	.71	1.67	0	.3	1.4	1.7	81.4	81.1	81.3
54.....	0	.6	.1	.70	0	0	.1	.1	92.2	98.9	95.6
55.....	0	.1	.06	.16	0	.8	0	.8	98.2	91.1	94.7
56.....	0	.27	.16	.43	0	.2	0	.2	95.2	97.8	96.5
57.....	0	0	0	0	0	.7	0	.7	100.0	92.2	96.1
58.....	0	.39	.25	.64	0	0	0	0	92.9	100.0	96.5
59.....	.6	1.4	.23	2.23	0	0	.4	.4	75.2	95.6	85.4
60.....	0	0	0	0	0	.3	0	.3 <sup>a</sup>	100.0	95.0 <sup>a</sup>	97.5
61 <sup>c</sup> .....	0	.....	.....	.....	0	.....	.....	.....	.....	.....	.....
62.....	0	.1	0	.10	0	.1	0	.1	98.9	98.9	98.9
63.....	0	.8	0	.80	0	0	0	0	91.1	100.0	95.6
64.....	.6	.5	.2	1.30	0	.3	.1	.4	85.6	95.6	90.6
66.....	0	.92	.98	1.90	0	.5	.6	1.1	78.9	87.8	83.4
67.....	0	0	.13	.13	0	0	0	0	98.6	100.0	99.3
68.....	.3	0	.3	.60	0	.9	.2	1.1	93.3	87.8	90.6
69.....	0	0	0	0	0	.1	.1	.2	100.0	97.8	98.9
70.....	0	.1	.32	.42	0	.1	.3	.4 <sup>a</sup>	95.3	93.3 <sup>a</sup>	94.3
71.....	0	0	0	0	0	.1	0	.1 <sup>a</sup>	100.0	98.3 <sup>a</sup>	99.2
72.....	0	.1	0	.10	0	.9	0	.9	98.9	90.0	94.5
73.....	0	0	.43	.43	0	0	0	0	95.2	100.0	97.8
74.....	0	0	0	0	0	.2	0	.2	100.0	97.8	98.9
75.....	0	0	0	0	0	0	0	0	100.0	100.0	100.0
77.....	0	0	0	0	0	0	0	0	100.0	100.0	100.0
78.....	.3	0	.2	.50	0	0	1.5	1.5	94.4	83.3	88.9
80.....	0	.2	0	.20 <sup>b</sup>	.....	.....	.....	.....	.....	.....	.....

<sup>a</sup> Based on two reports only. <sup>b</sup> Based on one report only. <sup>c</sup> Reported per cent free fatty acid only.

three replies revealed the preference summarized below on cottonseed and soybean oils, with negligible demand for any other oil samples.

	Cottonseed Oil	Soybean Oil
Highest number favored.....	10	6
Lowest number favored.....	3	0
Average number favored.....	4.2	3.5

While four samples of each of the two oils could be justified by this poll, a majority of the collaborators favored continuing the Referee Board's practice of distributing three samples of each, and this plan was followed again for the past year. Two degummed soybean oil samples were distributed in order to increase the information available to the Refining Committee on results with the tentative method for refining this oil.

In the absence of any better suggestion for tabulating the season's work, collaborators have been "graded" on both oils by the method previously used for the refining tests on cottonseed oil. The tabulation of grades has been furnished to the collaborators and to others closely concerned with the work, and may be considered as a continuation of the present work.

F. G. DOLLEAR  
R. T. MILNER  
A. S. RICHARDSON, chairman

### Grading System

Since there is no approved method for grading our check tests on soybean oil, the above tabulation has no official status. The undersigned has arbitrarily applied to the soybean oil samples the same system which has in recent years been used for grading collaborators on cottonseed oil refining tests, as explained more fully below.

Test	Tolerance	Deductions
F. F. A.....	±.1	.3 for each .1% outside tolerance.
Loss, not over 9%.....	±.3	.1 for each .1% outside tolerance.
Loss, over 9%.....	±.4	.9/x for each .1% outside tolerance.
Color (Red), not over 7.6.....	±.3	.1 for each .1% outside tolerance.
Color above 7.6 Red.....	±.4	7.6/x for each .1% outside tolerance.

x = accepted average corrected to nearest whole number.  
Limit of deduction for one determination on one sample = 1.

$$\text{Grade} = 100 - \frac{100 \times (\text{Total Deductions})}{3 \times (\text{Number of samples})}$$

Grades are based as usual on settlement results for loss and for color of refined oil. I.e., the collaborator's settlement result is compared with the settlement result picked from the averages. The settlement loss is simply the lowest loss for soybean oil, and settlement loss and color are fixed by the trading rules for cottonseed oil.

Full credit has been given for all reports received late due to circumstances beyond control of the collaborator.

A. S. RICHARDSON, chairman

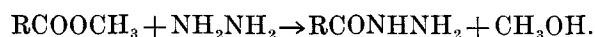
## Hydrazides of *n*-Aliphatic Acids<sup>1</sup>

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IN the course of an investigation of the composition and nature of the fission products of oxidized fatty esters, it was necessary to identify the *n*-aliphatic acids present in their mixtures. Usually these acids were obtained in the form of esters and often in rather small amounts. It was desirable to have recourse to a derivative that could be prepared directly from the ester in good yield and which would differ sufficiently from adjacent members in melting point and melting point depressions to permit accurate identification of the product or mixture of products.

The work of Sah (6) indicated that the monoacyl hydrazides might be satisfactory derivatives for this purpose, especially since the hydrazides may be analyzed for nitrogen by volumetric methods. The reaction of fatty acid esters with hydrazine may be illustrated by the following equation in which R can be an alkyl or aryl group:



Several of the hydrazides of the *n*-aliphatic acids have been prepared (3,6,7) previously as indicated in Table 1. The present series includes the hydrazides of the homologous series, valeric acid through lauric acid as well as those of myristic, palmitic, and stearic

acids. Efforts were made to prepare the hydrazides of oleic and elaidic acids. Contrary to the report of Hanus and Vorisek (3) elaidic acid formed a hydrazide in good yield. Oleic acid on the other hand underwent reduction and yielded mainly the hydrazide of stearic acid. A number of diacyl hydrazines of the *n*-aliphatic acids were also prepared but they did not possess sufficiently large differences in melting points between adjacent members of the homologous series to render them satisfactory as characterizing derivatives. The hydrazides can be prepared for purposes of identification with as little as 20 mg. of the parent esters.

### Experimental

**Materials.** In most cases, the saturated fatty acids and esters were obtained from Eastman Kodak Company. They were "white label products" and were used without further purification.

In a few cases the esters were prepared from commercial fatty acids by the method described by Bauer (1). Briefly the procedure consisted of treating commercial fatty acids with concentrated sulfuric acid, removing the sulfo derivatives by repeated washing with water, esterifying with methanol, and fractionally distilling the methyl esters. The fractions conforming most nearly to the calculated values for the saponification equivalent were selected for further purification either by redistillation (liquid

<sup>1</sup> Presented at the 38th Annual Meeting of the American Oil Chemists' Society, New Orleans, Louisiana, May 20-22, 1947.

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