Report of Smalley Foundation Committee—1947-48

THE SMALLEY COMMITTEE was reorganized two years ago and three subcommittees set up to carry on specific functions. The chairmen of the subcommittees make up the membership of the general committee. After the season started there appeared to be a demand for some check work on drying oils, so with the approval of the president a subcommittee was established to pioneer some check work on these products.

It was our hope this year to present certificates for outstanding work on cottonseed, soyabeans, peanuts, and the vegetable oils. This year for the first time certificates will be presented to the top two collaborators in the cottonseed and soyabean series. The peanut check work has not reached a degree of accuracy that would meet the high standards of the Society. An effort will be made during this meeting to work out an equitable method of grading the vegetable oil results in order that consideration may be given to the presentation of certificates next year.

The chairman of the various subcommittees are hereby listed:

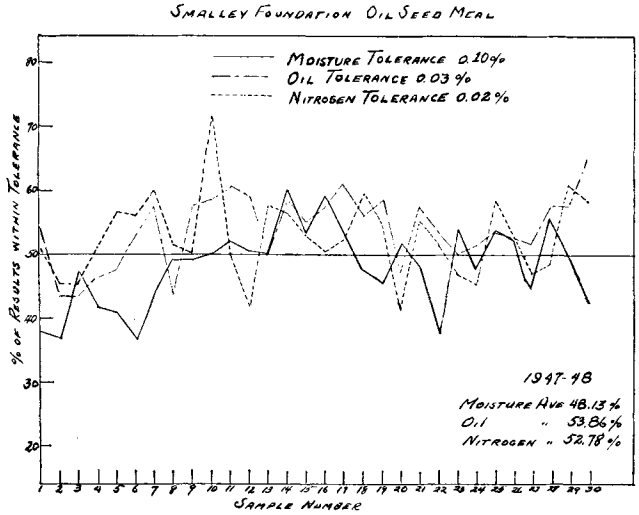
Subcommittee on Oil Seed Meal, R. W. Bates
Subcommittee on Oil Seeds, R. T. Doughtie
Subcommittee on Vegetable Oils, A. S. Richardson
Subcommittee on Drying Oils, Francis Scofield

Each subcommittee chairman will present a report on the activities of his committee.

R. W. BATES, chairman

SUBCOMMITTEE ON OIL SEED MEAL

WE are presenting herewith the 30th report of the activities of the Subcommittee on Oil Seed Meal. Every year some progress has been reported on the accuracy of the work. Again this year we can report this to be true. In all the work we feel that the results are unusually good. To this report is attached a graph



that has been prepared showing the number of collaborators (based on the percentage of the total) who were within the recognized tolerance of the accepted average. The general average on all samples has also been calculated. These values are as follows:

On Moisture 48.13% were within the tolerance of 0.10%
On Oil 53.86% were within the tolerance of 0.03%
On Nitrogen 52.76% were within the tolerance of 0.02%

This is a strong indication that the official tolerances bear the proper relationship to the accuracy of the three determinations. Further, we believe that a record of this type should be a part of future reports. While the proficiency of the winners is some indication of either improvement or non-improvement of the results, we believe that these calculations more clearly show the whole picture. It would be quite gratifying next season if the average for the moisture determination could be raised above 50%. It is also our recommendation that next season the moisture results be reported to the first decimal place only. It is the consensus of those consulted that moisture results reported to the second decimal place are not justified by the accuracy of the method.

During the year we asked two qualified statisticians to evaluate our method of calculation of the accepted average. They both informed us that the method in use is statistically sound. In this report we are including four tables indicating the standing (percentage) of the members participating. These may be listed as follows:

Table I lists the collaborators who reported moisture results on all samples.
Table II lists the collaborators who reported oil results on all samples.
Table III lists the collaborators who reported nitrogen results on all samples.
Table IV lists the collaborators who reported oil and nitrogen results on all samples.

Those listed with an asterisk contained some reports that were received late.

1. The award of the American Oil Chemists' Society Cup for the highest proficiency in the determination of both oil and nitrogen will be shared by

D. B. McIsaac, Kershaw Oil Mill, Kershaw, S. C.
M. A. Clark, Hartsville Oil Mill, Hartsville, S. C.

The per cent proficiency attained by these two men was 99.987%. Last year the value was 99.972%.

2. Certificate for second place will be awarded to

A. G. Thompson, Jr., Southern Cotton Oil Co., Columbia, S. C.

who attained a proficiency of 99.981%. Last year this value was 99.941%.

The other winners were:

	% Proficiency	Corresponding Value Last Year
Determination of Nitrogen:		
1st. M. A. Clark, Hartsville Oil Mill, Hartsville, S. C.	99.990	99.980
2nd. D. B. McIsaac, Kershaw Oil Mill, Kershaw, S. C.	99.986	99.974
2nd. P. D. Cretien. Texas Testing Laboratories, Dallas, Tex.	99.986	99.974
Determination of Oil:		
1st. A. G. Thompson, Jr., Southern Cotton Oil Co., Columbia, S. C.	99.995	99.968
1st. E. H. Tenent, Woodson-Tenent Laboratories, Memphis, Tenn.	99.995	99.968
2nd. G. K. Witmer, Battle Laboratories, Montgomery, Ala.	99.988	99.963
2nd. D. B. McIsaac, Kershaw Oil Mill, Kershaw, S. C.	99.988	99.963
Determination of Moisture:		
1st. R. C. Pope, Pope Testing Laboratories, Dallas, Tex.	99.948	99.912
2nd. N. C. Hamner, Southwestern Laboratories, Dallas, Tex.	99.940	99.834

In every case the winner attained a higher rating as measured by the per cent proficiency calculation than did the winners last year. Certificates will be presented to all the winners listed. This year again the A.O.C.S. Cup becomes the permanent possession of one of the winners. D. B. McIsaac won it in 1932-33 and 1943-44. According to the rules it becomes his property. A total of 88 collaborators were enrolled, and, as usual, 30 samples were distributed.

We again call attention to the preparation and distribution of the samples. The American Oil Chemists' Society should appreciate the tremendous contribution T. C. Law of Law and Company, Atlanta, Georgia, has made toward the success of this collaborative work through his careful handling of this phase.

TABLE I

Determination of Moisture

Analyst No.	Points Off	Per Cent Efficiency
22	11	99.948
6	13	99.940
42	21	99.902
34	30	99.860
56	39	99.818
74	44	99.794
9	45	99.790
39	66	99.692
15	69	99.678
51	74	99.654
19	75	99.650
35	77	99.640
67	78	99.635
24-62	81	99.621
66	82	99.617
48	83	99.612
1	84	99.607
29	86	99.598
8	98	99.542
23	103	99.519
18	106	99.505
21	110	99.485
63	113	99.471
25	117	99.453
33-41	121	99.435
37	124	99.421
30	126	99.411
77	129	99.397
7	134	99.373
12	139	99.351
40	146	99.317
44	150	99.299
52	154	99.281
82	156	99.271
16	164	99.235
58	165	99.229
14	170	99.205
17*	188	99.121
78*	191	99.107
60	193	99.099
27	194	99.093
26	199	99.071
11-13	211	99.014
50	215	98.995
10	266	98.756
45	298	98.608
57	304	98.580
70*	326	98.476
43	349	98.370
76	375	98.248
59*	490	97.711
54	496	97.683
64	517	97.584
65	534	97.505
32	559	97.388
20	673	96.855
75	741	96.537
73*	798	96.271
81*	921	95.694
68	944	95.588
47*	951	95.556
53	1022	95.224

* Reports received late.

TABLE II

Determination of Oil

Analyst No.	Points Off	Per Cent Efficiency
19-57	1	99.995
23-48	2	99.988
14	3	99.983
66	4	99.977
67	10	99.942
7-22-24-76	11	99.935
18-29	12	99.930
1-6	13	99.925
10	14	99.918
39-44	15	99.913
51	18	99.895
26	21	99.878
40	24	99.860
9-41	26	99.848
21	35	99.795
8-25	37	99.785
13-15	38	99.778
59*	39	99.773
12	41	99.761
17*	47	99.726
63	48	99.720
82	49	99.715
62	50	99.708
50-77	53	99.691
35	56	99.673
52-60	58	99.663
27	59	99.656
11	63	99.633
58	64	99.628
75	65	99.621
56	66	99.615
42-68	72	99.580
74	84	99.511
33	87	99.493
20	95	99.446
53	100	99.418
30	110	99.359
64	116	99.324
45	118	99.313
37	127	99.261
43	164	99.046
54	185	98.922
65	116	99.324
78*	219	98.724
47*	262	98.474
32	418	97.565
73*	425	97.523
34	511	97.023
81*	1017	94.074

* Reports received late.

We are again including in our report a list of the previous winners 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 Labs., Memphis, Tenn.
1921-1922	Battle Labs., Montgomery, Ala.
1922-1923	Battle Labs., 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 Labs., 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 Labs., Los Angeles, Calif.
1929-1930	Southwestern Labs., Dallas, Texas

TABLE III

Determination of Nitrogen

Analyst No.	Points Off	Per Cent Efficiency
14	2	99.990
48-67	3	99.986
6-66	4	99.981
29	6	99.971
19-26-76	7	99.967
11	9	99.957
1-23	10	99.952
10	11	99.947
40	13	99.938
24	14	99.932
12-52	16	99.924
15	17	99.918
18-20-25	18	99.914
57-58	19	99.909
50-51	20	99.904
22	21	99.899
16	30	99.856
35	31	99.852
41	33	99.842
39	35	99.832
8	36	99.827
7-54	38	99.817
27	39	99.813
37-74	41	99.803
60	42	99.798
53	44	99.788
64	46	99.780
46	49	99.765
2	50	99.760
33-68	52	99.751
30	57	99.726
63	66	99.683
43	68	99.673
62	73	99.650
56	76	99.636
9	77	99.630
82	79	99.621
17*	80	99.615
45	87	99.582
42-75	88	99.578
65-73*	91	99.564
36	92	99.558
21	122	99.414
31	126	99.395
32	129	99.381
59*	131	99.371
77	147	99.294
70*	166	99.204
13	174	99.165
47*	175	99.160
81*	189	99.093
34	205	99.016
78*	213	98.978
44	238	98.858
3	246	98.819

* Reports received late.

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 Labs., Dallas, Texas
1936-1937	N. C. Hamner, Southwestern Labs., 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. I. Rettger, Buckeye Cotton Oil Co., Memphis, Tenn.
1942-1943	Barrow-Agee Labs., Memphis, Tenn.
1943-1944	D. B. McIsaac, Kershaw Oil Mills, Kershaw, S. C.
1944-1945	W. W. Wynn, Jr., Barrow-Agee Labs., Cairo, Ill.
1945-1946	L. B. Forbes, L. B. Forbes Labs., Little Rock, Ark. Russell Haire, Planters Mfg. Co., Clarksdale, Miss.
1946-1947	Russell Haire, Planters Mfg. Co., Clarksdale, Miss.
1947-1948	D. B. McIsaac, Kershaw Oil Mill, Kershaw, S. C. M. A. Clark, Hartsville Oil Mill, Hartsville, S. C.

R. R. HAIRE — L. H. HODGES
R. T. DOUGHTIE — H. C. BLACK
T. C. LAW — R. W. BATES, chairman
T. L. RETTGER

TABLE IV

Determination of Oil and Nitrogen

Analyst No.	Per Cent Efficiency
14-48	99.987
19	99.981
66	99.979
23	99.970
67	99.964
6	99.953
57	99.952
29-76	99.951
1	99.939
24	99.934
10	99.933
26	99.923
18	99.922
22	99.917
51	99.900
40	99.899
7	99.876
39	99.873
25	99.850
15	99.848
41	99.845
12	99.843
8	99.806
50	99.798
11	99.795
52	99.794
58	99.769
35	99.763
9	99.739
27	99.735
60	99.731
63	99.702
20	99.680
62	99.679
17*	99.671
82	99.668
68	99.666
74	99.657
56	99.626
33	99.622
21	99.605
53	99.603
75	99.600
42	99.579
59*	99.572
30	99.543
37	99.532
77	99.493
13	99.472
45	99.448
65	99.444
44	99.386
54	99.370
43	99.360
64	99.264
78*	98.851
47*	98.817
73*	98.544
32	98.473
34	98.020
81*	96.584

* Reports received late.

SUBCOMMITTEE ON OILSEEDS

DURING the 1947-48 season the American Oil Chemists' Society offered three check series on oilseeds, namely cottonseed, soybeans, and peanuts.

On the cottonseed series of 10 samples a total of 45 chemists participated. Early in the series one collaborator withdrew and another failed to report on any sample. Grades received by the remaining 43 chemists averaged generally higher in efficiency than in prior years. Results reported showed excellent agreement on all factors with very few exceptions, and less errors in calculations, methods of reporting results, etc., were noted than heretofore. Two analysts received a final grade of 100.00% to end in a tie for first place. These analysts were George K. Witmer, Battle Laboratories, Montgomery, Ala., and C. L. Williams, Jackson Cotton Oil Mill Laboratory, Jackson, Tenn. Second place also resulted in a tie with two analysts receiving a final grade of 99.88%. These analysts were Edgar H. Tenent, Woodson-Tenent Laboratories, Memphis, Tenn., and Paul D. Cretien, Texas Testing Laboratories, Dallas, Tex.

On the soybean series of 10 samples a total of 25 chemists participated with one chemist withdrawing early in the series. Grades received by these collaborators, with few exceptions, averaged higher than during the previous year, and agreement between various analysts on oil results calculated to a 14% moisture basis was generally excellent. Variations occurring in moisture results were disturbing even though the allowed tollerance was increased from plus or minus 0.3% to plus or minus 0.5% from the accepted averages. First place on the series was attained by L. R. Brown, A. E. Staley Manufacturing Company, Decatur, Ill., with a grade of 99.4% while second place was made by George K. Witmer, Battle Laboratories, Montgomery, Ala., and R. C. Pope, Pope Testing Laboratories, Dallas, Tex., who tied with a grade of 98.8%. The results reported by analyst No. 21 were completely disregarded in arriving at the final grades, and no grade was assigned him due to his not following any recognized method of analysis on the samples. Had a grade been awarded him, it would have been considerably less than the recognized standard of 90.0% (82.6%).

On the peanut series of 7 samples a total of 18 chemists participated. Early in the series one chemist withdrew. (This analyst was the same one who withdrew from the cottonseed and soybean series). While the results on this peanut series were more uniform than on the series of a year ago, there is still considerable room for improvement. It is believed that the adoption of the peanut shaving machine to replace the food chopper grinder will, in the future, enable the collaborators to show results on identical samples which will be in closer agreement. The results on moisture results between collaborators have shown decided improvement and, on the whole, have indicated remarkable agreement. This improvement in results can be contributed largely to the work and recommendations of the Seed and Meal Analysis Committee under the general direction of T. H. Hopper and the cooperation of the members of his subcommittee. Chemist No. 1 received first place on the final grades for the series with a grade of 99.12%. This chemist is Thomas C. Law, Law and Company, Atlanta, Ga. Second place, with a grade of 98.36%, was made by Thomas B. Caldwell, Law and Company, Wilmington, N. C.

Final standing of the various collaborators (identification numbers on different series are not the same) on the several check series is shown below:

COTTONSEED SERIES

Place	Grade	Chem. No.	Place	Grade	Chem. No.
1	100.00	21-34	17	95.44	3-19
2	99.88	16-29	18	95.20	6-24
3	99.70	14	19	94.60	39
4	99.16	15	20	94.54	32
5	98.80	13	21	94.18	43
6	98.68	22	22	94.00	10-36
7	98.08	42	23	93.88	35
8	97.96	40	24	93.40	12-31
9	97.60	1-17	25	92.68	9
10	97.00	11	26	91.24	20
11	96.88	44	27	90.82	4
12	96.76	2-18	28	88.12	28
13	96.64	23	29	87.52	45
14	96.04	25	30	87.28	30
15	95.80	8	31	84.94	27
				Less than	41-33-5
16	95.50	7	32	80.00	26-38

SOYBEAN SERIES

1	99.4	20	9	93.4	5-7-13
2	98.8	10-11	10	92.8	2
3	98.2	23	11	90.4	14
4	97.6	16-17	12	89.2	24
5	97.0	9	13	88.6	8-19-25
6	96.4	4-12-15	14	87.4	3
7	95.8	6	15	79.0	18
8	94.0	1	....	.....	....

PEANUT SERIES

1	99.12	1	9	91.35	18
2	98.36	7	10	91.00	14
3	97.20	6	11	89.76	8
4	96.08	2	12	89.44	10
5	96.00	9	13	88.96	5
6	94.28	3	14	82.70	12
7	92.48	4-16	15	81.06	17
8	91.36	15	16	77.88	11

For several years we have used two or three sets of duplicate samples on the cottonseed series, such duplicates being prepared at the same time and sent out to collaborators from six to eight weeks apart. Results reported by the collaborators have shown remarkable agreement on all factors. This fact is most complimentary to the method used in the preparation of multiple samples from the same bulk sample as developed by Thomas C. Law, Law and Company, Atlanta, Ga., and his associates. On the 1947-48 series the following sets of duplicates were used: samples Nos. 5 and 8, 6 and 9, 7 and 10. Accepted averages of the samples as reported were:

	% F.M.	% Oil	% Ammonia	% F.F.A.	% Moisture
Set A: Sample No. 5.....	0.1	18.4	4.00	0.8	9.9
Sample No. 8.....	0.1	18.4	3.99	0.8	9.9
Set B: Sample No. 6.....	0.2	18.7	3.93	2.2	9.3
Sample No. 9.....	0.2	18.7	3.94	2.5	9.2
Set C: Sample No. 7.....	0.4	19.0	4.19	0.7	8.5
Sample No. 10.....	0.4	19.0	4.18	0.8	8.6

It will be noted on the above sets of duplicates that all factors show remarkable agreement. On Set B we have the widest range in F.F.A. results. However, sample No. 9 showed the expected rise in F.F.A. percentage between dates of the analysis of the two samples.

During the soybean series this season duplicate samples were also used—samples Nos. 5 and 6, 7 and 10. Very good agreement was obtained on both sets. The accepted averages of the samples as reported were:

CHECK OIL SAMPLES

Iden. No.	Deduction Points								Grade		
	Cottonseed Oil				Soybean Oil						
	F.A.	Loss	Color	Total	F.A.	Loss	Color	Total	C.S.O.	S.B.O	Both Oils
1*	0	.3	.6	.9	.3	0	0	.3	85.0[a]	96.7[a]	90.8
2	0	0	0	0	0	0	0	0	100.0	100.0	100.0
3	0	0	0	0	.3	.1	.1	.5	100.0	94.4	97.2
4	0	0	.1	.1	0	0	.1	.1	98.9	98.9	98.9
5	0	0	0	0	0	0	0	0	100.0	100.0	100.0
6	0	0	0	0	0	.6	.1	.7	100.0	92.2	96.1
7	0	0	1.0	1.0	0	.2	.2	.4	88.9	95.6	92.2
8	0	0	0	0	.9	.3	.1	1.3	100.0	85.6	92.8
9	0	0	.3	.3	0	.1	0	.1	96.7	98.9	97.8
10	0	0	0	0	0	0	0	0	100.0	100.0	100.0
11	.3	0	.3	.6	0	.6	0	.6	93.3	90.0[a]	92.0
12	0	0	.1	.1	0	.5	1.1	1.6	98.9	82.2	91.6
13	0	0	0	0	0	.7	.1	.8	100.0	91.1	95.6
14	0	0	.1	.1	0	0	.1	.1	98.9	98.9	98.9
15	.3	0	.5	.8	.9	0	.4	1.3	91.1	85.6	88.3
16	0	0	.1	.1	0	0	.2	.2	98.9	97.8	98.4
17	0	0	.3	.3	0	0	.3	.3	96.7	96.7	96.7
18	0	0	.4	.4	0	0	.7	.7	95.6	92.2	93.9
19	2.1	0	0	2.1	0	.5	0	.5	76.7	94.4	85.6
20	.3	.2	.1	.6	0	0	0	0	93.3	100.0	96.7
31	0	0	0	0	0	.5	0	.5	100.0	94.4	97.2
32	0	0	.1	.1	0	0	0	0	98.9	100.0	99.4
33	0	0	.7	.7	0	0	0	0	92.2	100.0	96.1
34	0	0	.1	.1	.3	0	0	.3	98.9	96.7	97.8
35	0	0	.1	.1	0	.7	.2	.9	98.9	90.0	94.4
36	0	0	.1	.1					98.3[a]	.....[b]	
37*	0	.4	.1	.5	0	.6	0	.6	94.4	93.3	93.9
38	1.2	.6	.4	2.2					75.6	.....[b]	
39	0	.2	.3	.5	0	0	0	0	94.4	100.0	97.2
40	.3	.2	.1	.6	.3	.1	.3	.7	93.3	92.2	92.8
41	0	0	0	0	0	.1	0	.1	100.0	98.9	99.4
42	0	.1	.9	1.0	0	.6	0	.6	88.9	93.3	91.1
43	0	.1	0	.1	0	0	0	0	98.9	100.0[a]	99.3
44*	0	0	.2	.2	0	.9	1.6	2.5	97.8	72.2	85.0
45	.3	.1	.2	.6	0	0	0	0	93.3	100.0	96.7
46*	0	.4	.3	.7	0	0	.3	.3	92.2	96.7	94.4
48	0	0	.2	.2	0	.2	0	.2	96.7[a]	96.7[a]	96.7
49	.3	.1	0	.4	0	0	0	0	95.6	100.0	97.3
50	0	0	0	0	0	1.0	.1	1.1	100.0	87.8[a]	93.9
51	0	0	.2	.2	0	.9	0	.9	97.8	90.0	93.9
52	0	.2	.6	.8	0	1.0	0	1.0	91.1	88.9	90.0
53*	0	0	0	0	0	1.5	0	1.5	100.0	81.3	91.2
54*	0	0	0	0	0	0	0	0	100.0	100.0	100.0
55	0	0	0	0	0	0	0	0	100.0	100.0	100.0
56	0	0	0	0	0	0	0	0	100.0	100.0	100.0
57	.3	0	.2	.5	0	.4	.2	.6	94.4	93.3	93.9
58*	0	0	.3	.3	0	.2	0	.2	96.7	97.8	97.2
59	0	.8	.4	1.2	0	.3	1.4	1.7	86.7	81.1	83.9
60*	0	.5	.3	.8	.3	0	0	.3	91.1	95.0	92.7
61	0	0	0	0	0	0	0	0	100.0	100.0	100.0
64	.6	.4	.6	1.6					82.2		
65*	.3	.4	.2	.9	0	.4	0	.4	90.0	93.3	91.3

[a] Based on two reports only.

[b] No grade is assigned on either oil unless more than one report was received.

* *In addition to the missing reports, the following deficiencies were noted:* Chemists No. 1, 37, 44, 46, 53, 54, 58, 60, and 65, one missing test each; chemist No. 65, two missing tests.

	% Moisture	% Oil	Obtained Beans from
Set A: Sample No. 5	8.5	17.7	Iowa
Sample No. 6	8.4	17.8	
Set B: Sample No. 7	10.6	17.7	New York
Sample No. 10	10.7	17.8	

G. CONNER HENRY
R. T. DOUGHTIE, JR., chairman

SUBCOMMITTEE ON CHECK OIL SAMPLES

AGAIN three samples each of crude cottonseed oil and of crude soybean oil have been distributed for collaborative refining tests, and in the continued absence of any better plan, the collaborators have been "graded" on both oils according to the method previously used for the refining tests on cottonseed oil. The results are shown in the accompanying table.

Grading System

Test	Tolerance	Deductions
F.F.A.	±.1	.3 for each .1% outside tolerance
Loss	±.3	.1 for each .1% outside tolerance
Color (Red)	±.3	.1 for each .1% outside tolerance

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 on settlement results for loss and for color, 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; settlement loss and color are fixed by the trading rules for cottonseed oil and reported by collaborators.

Full credit has been given for all reports mailed by collaborator before mailing of the mimeographed tabulation. Corrections without penalty are made for missing reports. Similar corrections are made for missing tests only in case report failed to include per cent free fatty acid and at least one result on refining loss and color.

F. G. DOLLEAR
F. R. EARLE
A. S. RICHARDSON, chairman

SUBCOMMITTEE ON DRYING OILS

EARLY in the 1947 season there was considerable interest in the Smalley Committee establishing a subcommittee to sponsor check work on drying oils. A committee was established and two sets of drying oils were distributed. Each set consisted of four

samples. The samples were analyzed for Color, Refractive Index, Specific Gravity, Acid Value, and Iodine Number. Twenty-four laboratories participated in the work. The results of the first set were tabulated and sent to the various participants. No attempt was made to treat the results statistically. The results in general were very creditable. The results of the second set are not available at this time.

We do not feel that the data on the samples should be listed at this time but hope that subsequent reports may carry information summarizing the deviations recorded. It is our feeling that substantial progress has been made in this work and it is our hope to continue it in 1948-49.

D. S. BOLLEY R. L. TERRILL
J. C. KONEN FRANCIS SCOFIELD, chairman

Arylstearic Acids From Oleic Acid. Variables Affecting the Yield and Properties*

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NUMEROUS references show the past and current interest in the reaction of an aromatic compound with an olefinic carboxylic acid such as oleic acid. The olefinic acid, the aromatic compound, and the condensing agent may be chosen from a variety of possibilities. In the present paper arylstearic acids, many of them new, prepared for the purpose of cooperation with the Naval Research Laboratory in the evaluation of lubricating oil additives are described. Observations regarding the nature of the reaction, with special attention being given to the use of oleic acid and of "iso-oleic" acid relatively free from linoleic acid, are also reported.

The arylstearic acids, which are prepared by the use of condensing agents of the type of aluminum chloride, are usually viscous oils which do not readily crystallize. This is believed due in part to their composition as a mixture of several isomers, the aryl radical being attached at different aliphatic carbon atoms in the fatty acid chain. Condensing agents of this type may cause migration of the double bond in oleic acid (5, 7, 21, 26), and if this occurs in the Friedel and Crafts reaction, formation of several isomeric arylstearic acids may be expected. Moreover, when substituted aromatic compounds are used in the synthesis, several isomers may be formed by attachment of the oleic acid to different points of the aromatic ring.

We have recently found that under certain conditions arylstearic acids which are solid at room temperature may be isolated in crystalline form. Thus far, we have obtained these solid crystalline acids most readily when using aromatic hydrocarbons which serve to restrict the possible number of isomers. Thus, the first solid crystalline arylstearic acid which we have isolated is obtained by the condensation of p-xylene with oleic acid. Solid products are also obtained in low yields by the condensation of p-chlorotoluene, o-xylene, o-chlorotoluene, and benzene with oleic acid.

Arylstearic acids prepared from different aromatic compounds differ in physical and chemical properties. The reaction by which they are prepared is a means of obtaining a selected saturated arylaliphatic carboxylic acid, and it was of interest to further explore the possibilities with readily available aromatic hydrocarbons.

Yield of Arylstearic Acids

Table I presents the yield and some of the analytical and physical data for 26 arylstearic acids. The reaction conditions were similar to those previously described (32, 33). Generally a 5 to 7 molar ratio of the liquid aromatic compound was used, and a 1.1 molar ratio of the metal halide was added in portions to the oleic acid solution. After all the metal halide had been added and had dissolved, the warm reaction mixture was heated to 80°, and then cooled and hydrolyzed in dilute hydrochloric acid. A petroleum ether or a mixture of a petroleum ether and o-dichlorobenzene was used as a solvent for reactions with solid (as well as for some liquid) aromatic compounds.

For the most part commercial oleic acid was used, having an average composition of 70% oleic, 15% linoleic, and 15% saturated fatty acids. The arylstearic acids were vacuum distilled at temperatures from about 220° to 280° at 0.4 mm. The yield was based on the oleic acid content.[3]

The yield, which depends on the aromatic compound, was greatest for technical m-xylene (92.4%) and the simpler alkylbenzenes. The yield was lower for aromatic chloro compounds, for compounds used in low molar ratios, for compounds which could easily undergo side reactions with aluminum chloride, and for arylstearic acids of higher molecular weight which could not be as readily vacuum distilled.

Aluminum bromide and zirconium chloride in place of aluminum chloride slightly improved the yield of phenylstearic acid (experiments 2 and 4). The yield of dimethoxyphenylstearic acid (experiment 25), and of xenylstearic acid (experiment 33), recorded in Table I, was increased to 40.5% and to 51.3%, respectively, when zirconium chloride was used as a catalyst in place of aluminum chloride.

An oleic acid of 95% purity (35) did not improve the yield, but the products had notably less color (experiments 5, 6, 9, 12, 13, 14, 15, 16, 17, 19, 20, 21, 22, 26, 29, 30, 32, 33). In experiment 14 a practically colorless xylylstearic acid was obtained from a solid

* Presented at the 39th Annual Meeting of the American Oil Chemists' Society, New Orleans, La., May 4-6, 1948.
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[2] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Dept. of Agriculture.
[3] Yields previously reported (32, 33), were based on a 100% content of oleic acid in commercial oleic acid and should be multiplied by a factor of about 1.4.