

# COOPERATIVE ANALYTICAL WORK of the American Oil Chemists Society\*

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Some years ago an eminent president of the American Chemical Society said, in effect, that few if any worthwhile contributions had ever been made to the science by other than those who had been trained in the rigorous school of the analytical laboratory.

I do not believe any small scientific society can equal the record of The American Oil Chemists' Society in the value of its contributions to an industry through the medium of the analytical laboratory. I also realize that I am speaking today to that branch of our society whose work is principally that of research chemistry, but I doubt if there is an individual present who has not served his full apprenticeship as an analytical chemist. It should be interesting information to you, therefore, to know something of the great volume of research work which has been conducted in the analytical laboratories of this society. I say "research work" advisedly for, while the improvement of laboratory efficiency is the main purpose of cooperative work, it is equally true that analytical methods are best perfected by this means.

Those in charge of our cooperative work have constantly kept in mind its research value in selecting the type of samples for check purposes. Over a period of years practically every variation of cottonseed, cottonseed meal and cottonseed oil has been represented in these samples. This has resulted in many discoveries which, in importance, go far beyond mere analytical methods. The work has not only been a decided contribution to science but has been largely responsible for the coordinating of laboratory and factory to the extent that the analyses of samples can accurately represent the monetary value of products.

While there has been sporadic and somewhat unorganized collaborative work since the founding of the original society in 1908, the service as



THOMAS C. LAW

a regular function really had its beginning in 1915 when Dr. F. N. Smalley, then Chief Chemist of the Southern Cotton Oil Company at Savannah, Georgia, started sending out weekly samples of cottonseed meal to a selected group of laboratories. For several years Dr. Smalley financed and personally handled this service as just one of his many valuable contributions to the industry and his fellow chemists. At his untimely death in 1921 no more appropriate memorial could have been established by his friends and this society than to carry on this work under his name as a major function of the society's service. It was enlarged to include all member laboratories that desired to participate. The regulatory chemists of the various state departments were invited to join without expense to them. So, this year, we really begin the twentieth series of cooperative cottonseed meal samples. For the past

ten years the laboratories participating have averaged over 75 in number. During these ten years nearly 25,000 check samples of cottonseed meal have been handled by the Smalley Foundation. The answer as to whether this work has been worth while is found in its record of proficiency. It can be said, without fear of contradiction, that no record of cooperative analytical work can be found which will equal that of this Foundation in close agreement of results. It also determined definitely the proper method for oil and nitrogen content of cottonseed meal.

Second only to the Smalley Foundation, in number of samples and record of efficiency, is the collaborative work on the laboratory refining of crude cottonseed oil. This was first undertaken by the original Chemists' Committee of the Interstate Cotton Seed Crushers' Association 25 years ago. In latter years it has been carried on jointly by the Refining Committee of the society and the Chemists' Committee of the association.

It would be impossible even to estimate the value to the industry that has resulted from standardizing laboratory refining methods. Only a few years ago hundreds of arbitrations were held each season. Today, there is an occasional arbitration on the interpretation of a rule but arbitration on quality of products is almost unheard of. The saving in dollars and cents, though quite large, is insignificant as compared with the good will and better understanding which now exists due almost entirely to the success of collaborative work. The only discordant note to be found is that we have done such a good job that referee analyses are no longer needed, and the commercial chemist is fast becoming a useless relic to be stored away in a dark corner on the shelves of scientific progress.

The third and most complicated series of check samples was made

\*A paper presented at the eighth fall meeting of the American Oil Chemists' Society in Chicago, October 11, 1934.

necessary when a system of seed grading was advocated by the U. S. Department of Agriculture. No finer example of real cooperative analytical research work can be found than that conducted by the laboratories of this society on cottonseed for the past four years. The keen interest, extreme care and actual pride which each collaborator has taken in this work is truly remarkable. A series of 30 samples was sent out each season to around 40 participants. Of a total of about 5,000 samples we find practically none unreported.

In a seed analysis there are five variables, each of which can be affected by the slightest deviation of method and by numerous outside influences. Four years of this collaborative effort have solved most of the problems which at times appeared to be insurmountable. The test for free fatty acids in the extracted oil is not yet as perfect as we would have it, but the results of a certain group of collaborators are most satisfactory.

The following figures will give some idea of what this contribution of service has actually cost the laboratories participating. Last season actual expense funds paid out were:

Smalley Foundation, 47 Laboratories at \$15.00 each.....	\$ 705.00
Cottonseed and Cottonseed Oil, 50 Laboratories at \$50.00 each	2,500.00
<b>Total .....</b>	<b>\$ 3,205.00</b>
Conservative value of analytical work—	
Cottonseed, 1,264 analyses at \$2.50 each .....	3,160.00
Cottonseed Meal, 1,405 analyses at \$3.00 each.....	4,215.00
Cottonseed Oil and Soapstock, 296 analyses at \$5.00 each .....	1,480.00
<b>Total .....</b>	<b>\$ 8,855.00</b>
<b>Grand total .....</b>	<b>\$12,060.00</b>

Over a four year period the laboratories of this society have paid in expenses and contributed in analytical services easily \$50,000.

An accurate record has been kept of the work each season and while statistics are rather uninteresting this paper would not be complete without a detailed account of at least one season's record.

A comparative system of grading was worked up from the record of the three series of cooperative samples analyzed during the season of 1932-33. First a plan of calculating an accepted average was adopted

for each test, then fair tolerances plus or minus the accepted averages were established. When the total points off from the accepted averages plus or minus the tolerances were determined a factor was selected based on the general average of the season. A passing mark of

80 was adopted and the average calculated to a mark of 90. This plan in detail for each series is given herewith, also a record of the season 1933-34 on each of the three series.

The system in detail is as follows:

**COTTONSEED**

All results except ammonia reported to first decimal.  
Deductions based on influence of each test upon final grade and value.

Test	Tolerance Allowed	Deduction by Points
Oil .....	0.2± Accepted Average	0.1 for each 0.1± Tolerance
Ammonia .....	0.05 Accepted Average	0.02 for each 0.01 Tolerance
F. F. Acids —		
Below 2.5% .....	0.2 Accepted Average	0.1 for each 0.1 Tolerance
2.5% to 5.0%.....	0.3 Accepted Average	0.1 for each 0.1 Tolerance
5.0% to 7.5% .....	0.5 Accepted Average	0.3 for each 0.5 Tolerance
Over 7.5% .....	1.0 Accepted Average	0.5 for each 1.0 Tolerance
Moisture .....	0.3 Accepted Average	0.05 for each 0.1 Tolerance
Error in calculations .....		0.3 When effects grade 1 or less 0.5 When effects more than 1
Not observing rules.....		0.4 When effects grade 1 or less 0.7 When effects more than 1

Average points off on 30 samples is 4.88.  
On basis of satisfactory passing grade of 80 a fair average would be 90. Therefore a factor of 2 is adopted.  
 $100 - (4.88 \times 2) = 90.24$   
Results of 43 laboratories: 11 over 95. 13 between 90 and 95. 14 between 80 and 90. 5 below 80.  
A passing mark of 80 was recommended for approved and designated laboratories.

**COTTONSEED CAKE AND MEAL**

All results reported to second decimal.

Test	Tolerance Allowed	Deduction by Points
Oil .....	0.10± Accepted Average	0.1 for each 0.01± Tolerance
	0.30 Accepted Average	No. 12
Ammonia .....	0.05 Accepted Average	0.1 for each 0.01 Tolerance

Average points off on 30 samples is 4.78.  
On basis of satisfactory passing grade of 80 a fair average would be 90. Therefore a factor of 2 was adopted.  
 $100 - (4.78 \times 2) = 90.44$   
Results of 45 laboratories: 28 over 95. 5 between 90 and 95. 8 between 80 and 90. 4 below 80.  
A passing mark of 80 was recommended for approved and designated laboratories.

**COTTONSEED OIL AND SOAP STOCK**

All results reported to first decimal. Deductions based upon influence of each test upon final grade and value.

Test	Tolerance Allowed	Deduction by Points
F. F. acids .....	0.1± Accepted Average	0.1 for each 0.1± Tolerance
Refining loss .....	0.3 Accepted Average	0.1 for each 0.1 Tolerance
Color .....	0.3 Accepted Average	0.05 for each 0.1 Tolerance
Bleach .....	0.2 Accepted Average	0.05 for each 0.1 Tolerance
Soap stock .....	0.3 Accepted Average	0.1 for each 0.1 Tolerance

Average points off 0.88 on 14 samples equivalent to 1.89 on 30 samples. On basis of a satisfactory passing grade of 80 a fair average would be 90. Therefore a factor of 5 was adopted.  $100 - (1.89 \times 5) = 90.55$ .  
Results of 26 laboratories: 17 over 95. 6 between 90 and 95. 2 between 80 and 90. 1 below 80.  
In view of the smaller number of samples it was necessary to balance with a recommendation of a passing mark of 90 for approved laboratories.

NATIONAL COTTONSEED PRODUCTS ASSOCIATION

Compilation of total points off from accepted average plus tolerance and official grades on the thirty samples of cottonseed meal. Smalley Foundation, 1933-34

Approved Laboratories NCPA				Designated Laboratories NCPA			
Chemist Number	Oil	Ammonia	Total Grade	Chemist Number	Oil	Ammonia	Total Grade
3	0.01	0.01	0.02 99.60	1	0.00	0.00	0.00 100.00
8	0.01	0.11	0.12 97.60	2	0.04	0.05	0.09 98.20
9	0.00	0.15	0.15 97.00	4	0.00	0.03	0.03 99.40
12	0.00	0.00	0.00 100.00	7	0.01	0.02	0.03 99.40
13	0.05	0.01	0.06 98.80	10	0.00	0.49	0.49 90.20
14	0.00	0.24	0.24 95.20	11	0.06	0.03	0.09 98.20
15	0.02	0.01	0.03 99.40	17	0.44	0.23	0.67 86.60
16	0.00	0.09	0.09 98.20	21	0.10	0.75*	0.85 83.00
18	0.00	0.00	0.00 100.00	25	0.00	0.00	0.00 100.00
22	0.15	0.06	0.21 95.80	29	0.00	0.16	0.16 96.80
24	0.00	0.15	0.15 97.00	30	0.53*	0.17	0.70 86.00
27	0.30	0.06	0.36 92.80	43	0.03	0.06	0.09 98.20
28	0.03	0.01	0.04 99.20	52	0.00	0.53*	0.53 89.40
48	0.07	0.73*	0.80 84.00	53	0.00	0.02	0.02 99.60
49	0.06	0.13	0.13 97.40	54	0.20	0.62*	0.82 83.60
51	0.00	0.02	0.02 99.60	60	0.24	0.37	0.61 87.80
55	0.00	0.24	0.24 95.20	80	0.09	0.09	0.18 96.40
†57	0.00	0.01	0.01 99.80	†81	0.24	0.59*	0.83 82.20
68	1.18*	0.31	1.49 70.20	...	...	...	...
59	0.00	0.02	0.02 99.60	...	...	...	...
†61	0.02	0.80*	0.82 83.00	...	...	...	...
65	0.00	0.00	0.00 100.00	...	...	...	...
69	0.01	0.13	0.14 97.20	...	...	...	...
70	0.37	0.12	0.49 90.20	...	...	...	...
71	0.00	0.07	0.07 98.60	...	...	...	...
72	0.01	0.89*	0.90 82.00	...	...	...	...
75	0.00	0.00	0.00 100.00	...	...	...	...
78	0.17	0.29	0.46 90.80	...	...	...	...
†79	0.19	0.22	0.41 91.60	...	...	...	...
Average	0.09	0.17	0.26 94.80	...	0.11	0.23	0.34 93.20
Average * out	0.05	0.095	0.145 97.10	...	0.085	0.12	0.205 95.90

Official method of grading approved by Chemists Committee and Executive Committee.

Tolerance plus or minus accepted average: Oil—0.10. Ammonia—0.05.

Deductions: Oil—0.10 for each 0.01 above or below tolerance limit.

Ammonia—0.10 for each 0.01 above or below tolerance limit.

Grade: 100.00 minus (total deduction × 2).

†Based on 29 samples. ‡Based on 28 samples. \*Over 0.50 points off.

No deductions on Numbers

Oil tests..... 1-4-9-10-12-14-16-18-24-25-29-49-51-52-53-55-57-59-65-71-75..... 21

Ammonia tests 1-12-18-25-65-75 ..... 6

Classification of laboratories on basis of season grades

Grade	Numbers	
100.....	1-12-18-25-65-75	6
98-100.....	2-3-4-7-11-13-15-16-28-43-51-53-57-59-71	15
95-98.....	8-9-14-22-24-29-49-55-69-80	10
90-95.....	10-27-70-78-79	5
85-90.....	17-30-52-60	4
80-85.....	21-48-54-61-72-81	6
Below 80.....	58	1

NATIONAL COTTONSEED PRODUCTS ASSOCIATION

Compilation of total points off from accepted average plus or minus tolerance, and official grades on six samples of crude cottonseed oil and two samples of soap stock for season 1933-34.

Chemist Number	Total Points Off						Incomplete Grade	Official Grade
	Free F. Ac.	Rfg. Loss	Color	Bleach	Total F. Ac.	Errors		
*1	0.00	0.00	0.05	0.00*	0.15	0.00	0.20	99.00
†2	0.00	0.00	0.00	0.00	0.00†	0.00	0.00	100.00
3	0.10	0.10	0.00	0.00	0.00	0.00	0.20	99.00
4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	100.00
5	0.00	0.00	0.00	0.00	0.00	0.00	0.00	100.00
6	0.00	0.00	0.00	0.00	0.00	0.00	0.00	100.00
†7	0.00	0.10	0.00	0.05	†...	0.00	0.15	99.25
†8	0.20	0.00	1.55	0.75	2.40†	0.00	4.90	75.50
*†9	0.20	0.20	0.20	*...	†...	0.00	0.60	97.00
10	0.00	0.00	0.00	0.00	0.39	0.00	0.39	98.05
†11	0.20	0.10	0.30	0.05	3.72†	0.00	4.37	78.15
12	0.20	0.70	0.25	0.10	0.00	0.00	1.25	...
13	0.00	0.00	0.00	0.05	0.66	0.00	0.71	96.45
14	0.00	0.00	0.10	0.10	0.13	0.00	0.33	98.35
15	0.10	0.00	0.00	0.00	0.00	0.00	0.10	99.50
16	0.00	0.00	0.00	0.00	0.00	0.00	0.00	100.00
17	0.00	0.00	0.15	0.00	0.03	0.00	0.18	99.10
18	0.00	0.20	0.00	0.00	1.42	0.00	1.62	91.90
19	0.10	0.00	0.40	0.20	0.24	0.00	0.94	95.30
20	0.00	0.10	0.00	0.10	0.00	0.00	0.20	99.00
†21	0.00	0.00	0.00	0.15	0.00†	0.00	0.15	99.25
22	0.00	0.30	0.00	0.05	0.00	0.00	0.35	98.25
23	0.00	0.00	0.05	0.05	0.00	0.00	0.10	99.00
24	0.00	0.20	1.05	0.55	0.00	0.10†	1.90	90.50
*†25	0.00	0.60	0.05	*...	†...	0.00	0.65	96.75
26	0.00	0.00	0.35	0.05	0.13	0.00	0.53	97.35
27	0.00	0.00	0.25	0.00	0.57	0.00	0.82	95.90
†28	0.40	0.00	0.00	0.05	0.00†	0.00	0.45	97.75
29	0.00	0.00	0.05	0.05	0.00	0.00	0.10	99.50
†30	0.00	0.00	0.00	0.00	0.06†	0.00	0.06	99.70
31	0.00	0.00	0.00	0.00	0.27	0.00	0.27	98.65
†33	0.40	0.20	0.70	0.00	0.43	0.20§	1.93	90.35
34	0.20	0.00	0.10	0.00	0.20	0.00	0.50	97.50
35	0.00	1.10	0.00	0.00	0.00	0.00	1.10	94.50
40	0.10	0.00	0.15	0.00	0.00	0.00	0.25	98.75
*†43	0.00	1.10	0.15	*...	†...	0.00	1.25	93.75
*†45	0.00	0.10	0.25	*...	†...	0.00	0.35	98.25
*†46	0.00	0.00	0.15	*...	†...	0.20¶	0.35	98.25
47	0.30	3.30	0.00	0.15	0.71	0.40¶	4.86	75.70

Official method of grading:

Test	Tolerance allowed
Free Fatty Acids .....	0.1
Refining Loss .....	0.3
Color, red .....	0.3
Bleach, red .....	0.2
Total Fatty Acids .....	0.3

Deduction by points

Test	Deduction by points	Tolerance
Free Fatty Acids .....	0.1 for each 0.1 ±	Tolerance
Refining Loss .....	0.1 for each 0.1	Tolerance
Color, red .....	0.05 for each 0.1	Tolerance
Bleach, red .....	0.05 for each 0.1	Tolerance
Total Fatty Acids .....	0.1 for each 0.1	Tolerance

Grade: 100.00 minus (Total deductions × 5).

\*Tests missing. †Reports missing. (Not following rules: ‡Reporting free fatty acids. §Grading. ¶Proper amount of lye.)

Classification of laboratories on basis of Official Grades.

Grade	Numbers	
100.....	4-5-6-16	4
98-100.....	3-10-14-15-17-20-22-23-29-31-40	11
95-98.....	13-19-26-27-34	5
92-95.....	12-35	2
Below 92.....	18-24-47	3

NATIONAL COTTONSEED PRODUCTS ASSOCIATION

Compilation of total points off from accepted average plus tolerance and final official grades on the thirty samples of cottonseed co-operative work, 1933-34.

Chemist Number	Points Off from Accepted Average Plus Tolerance						Official Grade
	Oil	Ammonia	F. F. Ac.	Moisture	Errors	Rules	
1	1.10	0.24	1.10	0.00	0.00	0.00	2.44
2	1.00	0.18	0.40	0.00	0.30	0.40	2.28
3	0.80	0.24	0.70	0.00	0.00	0.40	1.64
5	0.80	0.10	1.20	0.00	0.00	0.40	2.50
6	3.70	0.98	3.40	0.25	0.60	0.00	8.93
7	0.70	0.26	1.00	0.05	0.60	0.00	2.61
8	2.20	0.38	2.70	0.80	0.00	0.00	6.08
9	2.20	0.52	4.20	0.05	0.60	0.40	7.97
10	1.30	0.28	1.50	0.10	0.10	0.00	3.28
*11	4.40	1.82	3.70	0.00	0.00	0.00	9.92
12	3.10	0.46	1.10	0.55	0.20	0.00	5.41
13	2.40	0.40	1.10	0.10	0.00	0.00	4.00
14	0.60	0.18	0.10	0.30	0.00	0.00	1.16
15	2.60	0.50	0.70	0.45	0.00	0.00	4.25
16	1.60	0.86	1.30	0.30	0.00	0.00	4.06
17	2.90	0.28	1.50	0.85	0.50	0.40	6.43
18	1.30	0.74	2.70	0.30	0.00	0.00	5.04
19	0.50	0.42	2.30	0.05	0.00	0.00	3.27
20	1.40	0.16	0.20	0.15	0.30	0.00	2.21
21	2.60	1.14	1.60	0.15	0.40	0.00	5.89
22	0.10	0.50	1.90	0.00	0.00	0.00	2.50
23	1.80	0.06	0.40	0.05	0.00	0.00	2.31
24	1.20	0.10	1.10	0.00	0.00	0.00	2.40
25	3.10	0.52	0.80	0.00	0.30	0.00	4.72
26	2.60	0.58	0.80	0.30	0.10	0.40	4.78
27	3.30	0.26	3.80	0.20	0.30	0.00	7.86
28	0.90	1.06	1.30	0.00	0.00	0.00	3.26
29	2.10	0.60	0.60	0.05	0.00	0.00	3.35
30	1.30	0.10	2.60	0.05	0.60	0.00	4.65
31	0.60	0.36	1.50	0.20	0.00	0.40	3.06
32	4.30	0.68	3.10	0.40	0.50	0.40	9.38
33	1.30	0.40	1.20	0.15	0.00	0.40	3.45
34	0.70	0.10	0.60	0.00	0.00	0.40	1.40
35	1.10	0.62	2.50	0.00	0.00	0.00	4.22
36	0.60	0.14	0.10	0.00	0.00	0.00	0.84
37	2.20	0.14	1.50	0.10	0.10	0.00	4.04
38	3.20	0.26	1.80	0.90	0.30	0.40	6.86
39	1.20	0.02	1.40	0.50	0.00	0.00	3.12
40	1.30	0.02	0.30	0.45	0.50	0.06	2.57
42	0.40	0.14	1.50	0.15	0.00	0.00	2.19
43	2.00	0.76	0.90	1.85	0.00	0.40	5.91
†45	1.90	0.12	1.80	0.50	0.00	0.40	4.72
†46	0.40	0.12	1.50	0.10	1.40	0.40	3.92
Average	1.76	0.42	1.53	0.25	0.15	0.11	4.22

Classification of collaborators on basis of season grades:

Grade	Numbers	Total
95-100	1-2-3-5-14-20-22-23-24-34-36-42	12
90-95	7-10-13-15-16-19-25-26-28-29-30-31-33-35-37-39-40	17
85-90	8-12-17-18-21-38-43-45	8
80-85	6-9-27-32	4
Below 80	11-46	2

\*Based on 19 samples. †On 25 samples. ‡On 10 samples.  
 There are no perfect records on oil, ammonia and free fatty acids.  
 Off on only one sample Off on only two samples

Oil ..... Number 22 Numbers 2-14  
 Ammonia ..... Numbers 23-39-40 Numbers 14-34  
 Free fatty acids..... Numbers 2-14-23-36-40 Number 20

SOME

BIOCHEMICAL ASPECTS

OF FATS. PART I

By William E. Anderson and Harold H. Williams\*

(FROM THE DEPARTMENT OF PHYSIOLOGICAL CHEMISTRY, YALE UNIVERSITY, NEW HAVEN, CONN.)

A discussion of the biochemical aspects of lipids must necessarily include the components which resist the action of alkali—in other words, the unsaponifiable material. A few years ago Drummond (1) remarked:

“From the standpoint of the biochemist, the study of the unsaponifiable constituents of the natural oils and fats is a veritable El Dorado for those prepared to undertake the very difficult task of attacking the problems that await solution in this field of research.”

The diversity of substances present in the unsaponifiable portion of the mixture commonly called fats is indicated by the presence of hy-

drocarbons, alcohols, carotinoids, vitamins, and sterols. Whereas most of the animal and vegetable fats consist almost entirely of glycerides and a small percentage of unsaponifiable matter, the liver oils of some sharks contain as high as 90 per cent of unsaponifiable lipid. On the other hand, Chibnall and Channon (2) found the ether extract of the leaf cell cytoplasm of spring cabbage to contain only about 27 per cent of fatty acids.

Since the discovery of carotene and ergosterol as precursors of vitamin A and D, respectively, we recognize that all unsaponifiable components may be of the utmost biological significance. However, it is beyond the scope of this dis-

ussion to deal with each substance individually. The unsaponifiable lipid is a common constituent of protoplasm. The various components of the unsaponifiable fraction, however, are not common to all living matter. Some are peculiar to the plant, others to the animal kingdom; sterols on the other hand, are common to plant and animals alike, being, respectively, represented by phytosterols and cholesterol. This discussion will be confined to some of the biochemical aspects of cholesterol since this substance is today occupying an important place in biochemical investigation.

The ubiquitous occurrence of this substance in the structure of the animal cell is indicative of its im-

\*Sterling Fellow, 1934-35.