Report of Olive Oil Committee for 1930-1931

The Detection of "Sulfur" Oils in Olive Oils, Test for Halogenated Solvents and Iodine Number Determination

By M. F. LAURO, *Chairman*

THE following report submitted by the committee on olive oil of the American Oil Chemists' Society shows the benzoate test to be superior to the coin test for the detection of sulfur oils in olive oils, and recommends the Beilstein test for the detection of halogenated solvents in fatty oils in general. The report in full follows:

Individual Opinions

ANALYSTS 1, 2 and 4 concur in the sensitivity of the silver benzoate test when conducted according to the above modification. Lauro believes, however, that it is then too sensitive and there is the possibility of condemning a pressed oil. For this reason, the test as given by him was purposely restricted as to the later addition of the reagent and to the temperature of 150° C. so as to exactly parallel the results given by the coin test, so used on a thousand or so samples with check results. I would accordingly recommend the test as it stands. There is no need of going into the quantitative aspect as it involves some danger of uncertainty. Analyst 4 corroborates this by his statement that No. 1 oil contains substance giving a coloration similar to No. 2 sample under the modified procedure.

Analyst 6: "Coin test does not possess sufficient reliability to justify its use. Is reliable only when sulfur is contained in the oil in excessive quantities, much higher percentages than are necessary to detect by silver benzoate."

Analyst 7: "I think that this silver benzoate test is a very delicate and very satisfactory one. Certainly it proved to be much more efficient than the coin test in my hands.

All agree that at best, the coin test barely detects a 10% mixture of olive oil foots. See also report of the Committee of 1927-28, L. M. Roeg, *Chairman.*

Since a recovered oil, known in the trade as industrial oil, made from refined olive oil foots, contains a few hundredths of one-percent of sulfur due to the carbon bisulfide used for its extraction, a ten-percent admixture of this industrial oil in pressed olive oils would mean a very small amount of sulfur. I, therefore, feel it unwise to try to make the benzoate test conform to the detection of this minute amount or

Tabulation of Results.

Samples:
1. Pressed Olive Oil
2. Same with 10% olive oil foots added
3. Chlorine-extracted olive oil
4. Industrial olive oil (sulfur-extracted oil)

Tests:
The Beilstein test for halogen
The Coin test for sulfur or its compounds
The Silver Benzoate test for sulfur or its compounds

Report:

CHEMIST	Sample nos. and results #1 Coin	#1 Benz.	#2 Coin	#2 Benz.	#3 Beilstein	#4 Coin	#4 Benz.	Iodine No.
1.	Neg.	Neg.	Neg.	Neg.°	Positive	Pos.	Pos.	80.4
2.	Neg.	Neg.	Neg.	Slight°	Positive	Pos.	Pos.	80.7
3.	Neg.	Neg.	Neg.	Slight	Positive	Pos.	Pos.	79.2
4.	Neg.	Neg.	Slight	Neg.°	Positive	Pos.	Neg.°	
5.	Neg.	Neg.	Slight	Slight	Positive	Pos.	Pos.	81.3
6.	Neg.	Neg.	Slight	Positive	Positive	Pos.	Pos.	80.8
7.	Neg.	Neg.	Neg.	Positive	Positive	Pos.	Pos.	81

° Positive results obtained by the Benzoate test when the reagent was added to the oil in the beginning and then heated. The test is then rendered very sensitive. It was this modification that the committee of 1927-8 had found as I did, that as little as 1% of extracted oil could be detected.

Laboratory Bleaching Technique for Fatty Oils*

Control of Moisture Content of Vital Importance in Obtaining Results Comparable to Plant Operation

By J. T. R. ANDREWS and R. G. FOLZENLOGEN

THE fatty oil industry has long been cognizant of the fact that the bleaching of refined vegetable oils, such as cottonseed, with fuller's earth is considerably more efficient when performed on a factory scale than when carried out in the laboratory. The official bleaching test of the A.O.C.S., which is practically identical with that of the National Cottonseed Products Association (Rule 275, Sec. 2), makes allowance for this discrepancy between plant and laboratory by prescribing the use of 6.0% earth, an amount which is greater than that ordinarily used in the commercial bleaching of cottonseed and similar refined oils, but which is agreed to by oil technicians because they know that the laboratory bleached color can be duplicated in the plant with only about half of this earth usage. The official bleaching test was studied recently by Ma and Withrow[1]. In general, they found its main features justified. An excellent bibliography is appended to their paper.

THE official laboratory bleach test is made by heating 300 grams of refined oil in a refining cup to 120° C.; 6.0% of the official fuller's earth is added and the mixture is stirred mechanically at 250 r.p.m. for five minutes without allowing the temperature to drop below 105° C. The bleached oil is filtered through paper and the color of the clear oil is determined by matching a 5¼" column with Lovibond red and yellow type glasses.

In order to bring plant and laboratory conditions of time and temperature more closely together 105° C. and 15 minutes were selected for our first experiments. The official bleaching apparatus operated at 250 r.p.m. and 300 gram samples of oil were employed for most of our work. An open cup or beaker was used in all experiments except the few specifically described as made in covered vessels. We were fortunate in directing our early experiments to an investigation of the role of moisture in bleaching and the results obtained were so startling that the development of this idea constituted the remainder of our program.

Moisture Loss a Factor

IT WAS thought that in the laboratory, with only a 300 gm. sample in an open cup, drying would take place much more rapidly than in the plant where 50,000 to 80,000 lbs. of oil are agitated in a closed U-tank. Accordingly, comparative bleaches were made with and without addition of 1.0% of water (Table I) which showed that 3.0% earth + 1.0% of water is almost, but not quite, equal in bleaching power to 6.0% earth without water. Further experiments (Table II) showed that the optimum amount of water to be added is not proportional to the quantity of earth. For most efficient bleaching on a 300 gram scale,

TABLE I
Bleaching Refined Cottonseed Oil With and Without Added Water
(105°C.—15 minutes—open cup—original color 6.2 R)

% English Earth	Color, Lovibond Red: No Water	1.0% Water
1.0	5.0	4.1
2.0	4.2	3.3
3.0	3.4	2.8
4.0	3.1	2.5
5.0	2.9	2.3
6.0	2.7	2.2

TABLE II
Bleaching Refined Cottonseed Oil With Varying Amounts of Added Water
(105°C.—15 minutes—open cup—original color 7.5 R)

	Color, Lovibond Red			
% English Earth	1.0	2.0	4.0	6.0
% Water				
0.00	4.5	3.1	2.3	2.0
0.20	4.1	2.9	2.1	2.1
0.50	3.7	2.7	2.0	2.0
0.75	3.4	2.4	2.0	1.7
1.0	3.3	2.3	2.0	1.8
1.5	3.3	2.4	2.0	1.9
2.0	3.4	2.4	2.1	1.9
3.0	4.7	3.8	2.3	2.1
4.0	5.4	3.8	3.1	2.3

*Presented at Fall Meeting, American Oil Chemists' Society, Chicago, Nov. 14, 1930.

[1] Ind. & Eng. Chem. Analytical Ed. Vol. 2, pp. 374-77 (October, 1930).

the water added should be about 1.0% of the weight of oil taken. Without pausing to exhibit data, some of our other findings may be summarized as follows:

1. There apparently exists an optimum intensity of agitation above and below which poorer bleaching results. Speed is not a critical factor between 150 and 250 r.p.m.
2. Best results are obtained by mixing the earth with the cold oil and adding the water before heating is begun.
3. Added moisture improves the bleaching efficiency of earth and carbon mixtures, e.g. English + Darco.
4. Oil may be humidified during bleaching by use of steam which is effective but difficult to control.
5. Free fatty acid percentage does not increase during bleaching by the moisture method with English earth or Carlton earth.
6. A blanket of inert gas (CO_2) is not beneficial and when circulated over the surface of the oil it apparently defeats in part the object of added moisture.
7. Filtration of the refined oil through paper removes excessive moisture and conditions it for bleaching by the moisture method.
8. On freshly refined oils, no difficulty was experienced in getting a smooth and apparently homogeneous suspension of earth in oil after 1.0% of water had been added, but on oils which had been dried and filtered through kieselguhr in a press a considerable tendency for the earth to agglomerate into small balls was noted. This condition was attributed to removal of soap from the oil by adsorption on the guhr and, as the trouble was prevented entirely by the addition of from 0.125 to 0.25% of 10% soap solution, this surmise was probably correct.
9. Dehydration, either by oven drying or by desiccation over H_2SO_4, lowers bleaching power to a marked degree in the case of English earth. By humidification, bleaching efficiency can be completely restored. The same observations, to a somewhat less extent, are true of all other earths which we have studied over a period covering the past three years. It should be noted that maximum bleaching efficiency with English, Carlton and Filtrol is reached at about 20% moisture content. (Tables III-A, B & C.)

TABLE III-A

Bleaching Refined Cottonseed Oil
With English Earth of Variable Moisture Content
(105°C. — 15 minutes—open cup — no added water — 2.68% dry earth)

Earth Treatment	% Moisture in Earth	Color, Lovibond Red Oil No. 1	Color, Lovibond Red Oil No. 2
Color before Bleaching.......		8.3	8.8
Dried at 105°C. 16 hrs.......	0.0	4.9	5.2
Over conc. H_2SO_4 16 days....	6.1	4.2	
Earth as received............	10.8	3.7	3.8
Over 50% H_2SO_4 16 days....	13.8	3.4	
Over water 16 days.........	19.8	3.1	3.4
Moistened with water, mixed and allowed to stand several days	25.6		5.4
	31.7		6.6
	36.3		7.9

TABLE III-B

Bleaching Refined Cottonseed Oil
With Carlton Earth of Variable Moisture Content
(105°C.—15 minutes — open cup — no added water — 2.75% dry earth)

Earth Treatment	% Moisture in Earth	Color, Lovibond Red
Color before bleaching......		8.8
Dried 105°C. 16 hrs........	0.0	4.2
Earth as received..........	8.3	3.4
Moistened with water, mixed and allowed to stand several days	19.3	3.3
	26.2	3.6
	31.7	4.5
	38.2	6.3

TABLE III-C

Bleaching Refined Cottonseed Oil
With Filtrol of Variable Moisture Content
(105°C.—15 minutes—open cup—no added water)

Earth Treatment	% Moisture in Earth	Color, Lovibond Red	
% Dry Earth used in Bleach..		1.28	2.57
Color before bleaching........		8.8	8.8
Dried 150°C. 16 hrs..........	0.0	4.3	2.6
Earth as received............	14.4	3.9	2.4
Moistened with water, mixed and allowed to stand several days	19.4	3.8	2.5
	26.0	4.2	3.1
	31.8	4.4	3.3
	37.4	5.3	3.6

10. With proper moisture control, laboratory bleaching with English earth is equal to or better than plant bleaching in efficiency (Table IV-A).

TABLE IV-A

Plant vs. Laboratory Bleaching of Refined Cottonseed Oil with English Earth

Color Refined Oil	% English Earth	Color Plant Bleached	Laboratory Color No Water	Laboratory Color 1.0% Water
8.5	2.4	3.9	4.7	3.6
6.2	2.1	3.4	4.2	3.1
8.6	2.3	3.8	4.4	3.3
10.0	2.3	3.6	4.4	3.6
9.0	2.3	3.5	4.3	3.4
8.3	1.9	3.2	4.4	3.3
8.6	2.9	3.1	3.5	3.0
8.0	1.9	3.4	4.2	3.3
7.9	1.8	4.0	4.1	3.4
Average Color		3.5	4.3	3.3

With due precaution to prevent loss of moisture during laboratory bleaching, the efficiency obtained is approximately the same as that of the plant but not quite as high as that obtainable in the laboratory when optimum moisture conditions prevail (Table IV-B).

All of the conclusions herein presented were drawn from experimental bleaches on plant refined cottonseed oil. A few experiments on refined tallow and coconut oil indicate that similar results may be expected on other fatty oils. It should be noted that these conclusions apply primarily to laboratory bleaching under the conditions described.

At this point, the question may be asked "Cannot plant as well as laboratory bleaching be improved by proper moisture control?" Our observations have led us to the opinion that under ordinary conditions this is unlikely. The optimum moisture for plant scale bleaching is much lower than 1.0%; probably nearer 0.1-0.3% and most plant refined oil direct from the settling tanks is of practically optimum moisture content for bleaching on a factory scale. In a few cases, perhaps, such as in the case of oils which have been dried for storage, moisture control may have a plant application, but we are not optimistic over the possibility of its general extension into plant practice.

Effect of Temperature

ON BLEACHES made below 100° C. there is apparently no benefit derived from added moisture. With increasing temperature, the

TABLE IV-B

Plant vs. Laboratory Bleaching of Refined Cottonseed Oil with English Earth

Color Refined Oil	% English Earth	Color Plant Bleached	No Water	Laboratory Color Covered	1.0% Water
8.2	2.3	3.5	4.1	3.4	3.3
7.7	1.8	3.3	4.6	3.8	3.7
7.9	1.8	3.8	4.7	3.8	3.5
6.4	1.5	4.0	4.4	3.7	3.3
Average Color		3.7	4.5	3.7	3.5

moisture method improves in efficiency, reaching a maximum at about 135° C., where 5 minutes has been found ample time. At either 135° C. for 5 minutes or at 105° C. for 15 minutes, the results obtained by the moisture method using 3.0% English earth are only slightly inferior to bleaches made by the A.O.C.S. official method using 6.0% earth (Table V).

TABLE V

Comparison with A.O.C. S. Official Bleach Test

Color Refined Oil	Official Bleach 6% Earth; 120°-105° C. 5 minutes	Color, Lovibond Red 3% Earth; 1% Water; 105° C. 15 minutes	3% Earth; 1% Water; 135° C. 5 minutes
10.4	4.1	4.4	4.4
16.8	5.4	5.7	5.2
8.2	2.1	2.3	2.1
6.9	2.1	2.2	2.1
6.4	2.1	2.2	2.3
33.0	10.2	11.2	10.8
8.2	3.2	3.6	3.6
Average Color	4.2	4.5	4.4

General Conclusions

IN EXAMINATION of a considerable number of bleaching earths, we have found very few laboratory bleach tests which are not improved by the addition of water and practically none which are injured unless the refined oil is already excessively wet. In order to make an intelligent comparison of the bleaching efficiency of earths, the laboratory test should simulate the essential conditions prevailing in actual plant practice and we claim that no test which ignores the important function of moisture will meet this requirement. By the official test, or any test in which the laboratory moisture loss is not kept at a minimum or replaced by added water, positively erroneous conclusions can be drawn regarding the relative bleaching efficiencies of two earths under factory conditions and this is especially true in case one of these earths is more sensitive to moisture control than the other.

In conclusion, it is suggested that the official bleaching method of the A.O.C.S. be modified in the way we have demonstrated as effective in putting laboratory and plant bleaches on a more comparable basis. The modified method, making use of moisture control, will enable laboratory tests on both earths and oils to be translated directly into an accurate forecast of plant scale bleaches.

Report of the Committee to Rewrite the Methods

THE CHANGES made last year in the methods, and new methods covering cottonseed made necessary the reprinting of eighteen pages of the methods. This work was done by the Lefax Company at a total cost to the Society of $82.50 for two thousand sets.

Our methods, as now being sent out, are revised to August 1, 1930.

The only recommendation this committee has to offer is that the methods covering Soap also be printed in the Official Methods.

W. H. IRWIN, *Chairman.*

Chicago, April 13, 1931.

Laboratory Construction Company of Kansas City, Missouri, reports the sale of nitrogen apparatus to Wilson and Company of Oklahoma City, and Cudahy Packing Company of Kansas City, Kansas. Also, the complete installation of Laboratory Equipment Tables and Kjeldahl Nitrogen Apparatus for the three laboratories of the Food and Dairies Division, State of Illinois at Springfield, Illinois.

The Research Association of British Paint, Colour and Varnish Manufacturers have recently issued a Review, giving abstracts of current literature relating to the paint, color and varnish industries.

Carbohydrates Affect Mill Values of Cottonseed

The Relation of Percentages of Oil, Ammonia and Meats in Whole Cottonseed to the Carbohydrate Content of Meats

By G. S. MELOY

THE oil and the protein in cottonseed are found in the kernels or meats. In the natural state the hulls or seed coats, contain no oil, and less than one-half of one per cent of ammonia, indicating less than 0.4 per cent of nitrogen, or 2.5 per cent of protein. The crushers of cottonseed therefore depend upon the meats for the production of oil and for the protein content of the cake.

The oil content of pure cottonseed meats varies from below 28 per cent to above 40 per cent; and the protein content varies from below 30 per cent to above 52 per cent. In combination the oil and protein together form from below 70 per cent to above 80 per cent of the meats. The residue, composed of carbohydrates, moisture and other minor materials, therefore forms from less than 20 per cent to more than 30 per cent of the meats. The differences in the content of these materials, in the meats of different lots of cottonseed, is therefore a factor of value in the seed.

The meats content of different lots of cottonseed varies from below 40 per cent to above 56 per cent of the whole seed, due to differences in the thickness and density of the hulls, but chiefly to differences in the vegetation development and density of the meats themselves.

Variations in the meats content of cottonseed present problems of separating meats from the hulls. The lower the percentage of meats the greater the difficulty of separation, both because of the lower specific gravity of the meats and also because of the larger portion of hulls, to be separated from the meats. Differences in the meats content of various lots of cottonseed is therefore a factor of value in the seed.

In the accompanying tables percentages of oil and ammonia in whole cottonseed are grouped in columns under various percentages of meats. In the left hand column are given the percentages of oil and protein in the meats, corresponding with and resulting from the combinations of the percentages of oil, ammonia and meats in the whole seed. In Table I are grouped analyses of whole seed in which the combined oil and protein in the meats equal 70 per cent of the meats; in Table 2 the combined oil and protein equal 74 per cent of the meats; in Table 3 the combined oil and protein equal 76 per cent of the meats, and in Table 4 the combined oil and protein equal 80 per cent of the meats.

Similar tables may be prepared showing practically every possible combination of oil, ammonia and meats percentages found in whole seed and the corresponding oil and protein content of pure meats. These tables, however, are sufficient for this discussion. Tables 1 and 4 represent practically the extremes of variations, while Tables 2 and 3 represent the modal variations of normal cottonseed.

The average oil content of cottonseed, 19 per cent, and average ammonia content, 3.50 per cent, is found in the column 50 per cent meats, Table 2. In the left hand column it is seen that these percentages of oil, ammonia and meats in whole seed indicate that the percentage of oil in the meats is 38 and the percentage of protein 36, making a combination of 74 per cent of the meats.

These tables show that when the percentage of oil in whole cottonseed remains constant, increase in the ammonia content is the result of increases in the meats content and is coincident with increase in the content of materials in the meats other than protein and fat. Also that increases in both oil and ammonia in whole seed result from increases in the meats content and may or may not be coincident with increase of material in the meats other than protein and fat, depending upon the ratio of the increase in ammonia.

The tables also furnish a basis for visualizing the composition of cottonseed in connection with various growing conditions. For instance, typical combinations of oil and ammonia in seed produced in the drought area of Arkansas, Mississippi and Tennessee, during the past season, are found in the columns of meat content between 40 and 45 per cent, Tables 3 and 4, indicating a retarded or arrested vegetative development of the kernels or meats.

Typical combinations of oil and ammonia, in seed produced in northwestern Texas and western Oklahoma, are to be found in the columns of percentages of meats above 50 and generally also in Table 1 in which the combined oil and protein is only 70 per cent of the meats and are therefore concurrent with a high content of non-protein or fat materials in the meats, chiefly carbohydrates.

These significant differences in the carbohydrate content of cottonseed meats doubtless have marked influence on the color and refining loss of oil and possibly also on the color of the cake. High carbohydrate content may indicate lower cooking temperatures.

Variations in the content of carbohydrates in cottonseed kernels has apparently received very little consideration in past studies of the composition of cottonseed.

TABLES SHOWING THE RELATION BETWEEN THE PERCENTAGES OF OIL, AMMONIA AND MEATS IN WHOLE SEED AND COMBINED OIL AND PROTEIN IN MEATS

Table 1

Oil & Protein totaling 70% of meats		Percentages of meats in whole seed: 42		44		46		48		50		52		54		56	
Oil Per cent	Protein Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent
28	42	11.76	3.43	12.32	3.60	12.88	3.76	13.44	3.92	14.00	4.09	14.56	4.25	15.12	4.41	15.68	4.57
29	41	12.18	3.35	12.76	3.51	13.34	3.67	13.92	3.83	14.50	3.99	15.08	4.15	15.66	4.30	16.24	4.46
30	40	12.60	3.27	13.20	3.42	13.80	3.58	14.40	3.73	15.00	3.89	15.60	4.05	16.20	4.20	16.80	4.36
31	39	13.02	3.19	13.64	3.33	14.26	3.49	14.88	3.65	15.50	3.79	16.12	3.94	16.74	4.10	17.36	4.24
32	38	13.44	3.10	14.08	3.25	14.72	3.40	15.36	3.55	16.00	3.70	16.64	3.84	17.28	3.99	17.92	4.14
33	37	13.86	3.02	14.52	3.16	15.18	3.31	15.84	3.45	16.50	3.60	17.16	3.75	17.82	3.89	18.48	4.03
34	36	14.28	2.94	14.96	3.08	15.64	3.22	16.32	3.36	17.00	3.50	17.68	3.64	18.36	3.78	19.04	3.92
35	35	14.70	2.85	15.40	2.99	16.10	3.13	16.80	3.27	17.50	3.40	18.20	3.54	18.90	3.67	19.60	3.82
36	34	15.12	2.77	15.84	2.91	16.56	3.04	17.28	3.17	18.00	3.31	18.72	3.44	19.44	3.57	20.16	3.71
37	33	15.54	2.70	16.28	2.82	17.02	3.95	17.76	3.08	18.50	3.21	19.24	3.33	19.98	3.47	20.72	3.60
38	32	15.96	2.61	16.72	2.74	17.48	2.86	18.24	2.99	19.00	3.11	19.76	3.23	20.52	3.36	21.28	3.49
39	31	16.38	2.53	17.16	2.65	17.94	2.77	18.72	2.89	19.50	3.02	20.28	3.14	21.06	3.26	21.84	3.38
40	30	16.80	2.45	17.60	2.57	18.40	2.69	19.20	2.80	20.00	2.92	20.80	3.04	21.60	3.15	22.40	3.27

Table 2

Oil & Protein totaling 74% of meats		Percentages of meats in whole seed: 42		44		46		48		50		52		54		56	
Oil Per cent	Protein Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent
28	46	11.76	3.76	12.32	3.94	12.88	4.11	13.44	4.29	14.00	4.47	14.56	4.66	15.12	4.83	15.68	5.01
29	45	12.18	3.67	12.76	3.85	13.34	4.02	13.92	4.20	14.50	4.38	15.08	4.55	15.66	4.73	16.24	4.90
30	44	12.60	3.60	13.20	3.77	13.80	3.94	14.40	4.11	15.00	4.28	15.60	4.45	16.20	4.62	16.80	4.79
31	43	13.02	3.51	13.64	3.68	14.26	3.84	14.88	4.01	15.50	4.18	16.12	4.35	16.74	4.51	17.36	4.68
32	42	13.44	3.43	14.08	3.60	14.72	3.76	15.36	3.92	16.00	4.09	16.64	4.25	17.28	4.41	17.92	4.57
33	41	13.86	3.35	14.52	3.51	15.18	3.67	15.84	3.83	16.50	3.99	17.16	4.15	17.82	4.30	18.48	4.46
34	40	14.28	3.27	14.96	3.42	15.64	3.58	16.32	3.73	17.00	3.89	17.68	4.05	18.36	4.20	19.04	4.36
35	39	14.70	3.19	15.40	3.33	16.10	3.49	16.80	3.64	17.50	3.79	18.20	3.94	18.90	4.10	19.60	4.24
36	38	15.12	3.10	15.84	3.24	16.56	3.40	17.28	3.55	18.00	3.70	18.72	3.84	19.44	3.99	20.16	4.14
37	37	15.54	3.03	16.28	3.16	17.02	3.31	17.76	3.45	18.50	3.60	19.24	3.75	19.98	3.89	20.72	4.03
38	36	15.96	2.94	16.72	3.08	17.48	3.22	18.24	3.36	19.00	3.50	19.76	3.65	20.78	3.65	21.28	3.92
39	35	16.38	2.86	17.16	2.99	17.94	3.14	18.72	3.27	19.50	3.40	20.28	3.54	21.06	3.67	21.84	3.82
40	34	16.80	2.77	17.60	2.91	18.40	3.04	19.20	3.17	20.00	3.17	20.80	3.44	21.60	3.57	22.40	3.71

Table 3

Oil & Protein totaling 76% of meats		Percentages of meats in whole seed: 42		44		46		48		50		52		54		56	
Oil Per cent	Protein Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent
28	48	11.76	3.92	12.32	4.11	12.88	4.29	13.44	4.48	14.00	4.67	14.56	4.85	15.12	5.04	15.68	5.23
29	47	12.18	3.84	12.76	4.02	13.34	4.21	13.92	4.39	14.50	4.57	15.08	4.75	15.66	4.94	16.24	5.12
30	46	12.50	3.76	13.20	3.94	13.80	4.11	14.40	4.28	15.00	4.47	15.60	4.66	16.20	4.83	16.80	5.01
31	45	13.02	3.67	13.64	3.85	14.26	4.02	14.88	4.20	15.50	4.38	16.12	4.55	16.74	4.73	17.36	4.90
32	44	13.44	3.60	14.08	3.77	14.72	3.94	15.36	4.11	16.00	4.28	16.64	4.45	17.28	4.62	17.92	4.79
33	43	13.86	3.51	14.52	3.68	15.18	3.84	15.84	4.01	16.50	4.18	17.16	4.35	17.82	4.51	18.48	4.68
34	42	14.28	3.43	14.96	3.60	15.64	3.76	16.32	3.92	17.00	4.09	17.68	4.25	18.36	4.41	19.04	4.57
35	41	14.70	3.35	15.40	3.51	16.10	3.67	16.80	3.83	17.50	3.99	18.20	4.15	18.90	4.30	19.60	4.46
36	40	15.12	3.27	15.84	3.42	15.56	3.58	17.28	3.73	18.00	3.89	18.72	4.05	19.44	4.20	20.16	4.35
37	39	15.54	3.19	16.28	3.33	17.02	3.49	17.76	3.64	18.50	3.79	19.24	3.94	19.98	4.10	20.72	4.24
38	38	15.96	3.10	16.72	3.24	17.48	3.40	18.24	3.55	19.00	3.70	19.76	3.84	20.52	3.99	21.28	4.14
39	37	16.38	3.03	17.16	3.16	17.94	3.31	18.72	3.45	19.50	3.60	20.28	3.75	21.06	3.89	21.84	4.03
40	36	16.80	2.94	17.60	3.08	18.40	3.22	19.20	3.36	20.00	3.50	20.80	3.65	21.60	3.78	22.40	3.92

Table 4

Oil & Protein totaling 80% of meats		Percentages of meats in whole seed: 42		44		46		48		50		52		54		56	
Oil Per cent	Protein Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent	Oil Per cent	NH_3 Per cent
28	52	11.76	4.25	12.32	4.45	12.88	4.66	13.44	4.85	14.00	5.06	14.56	5.26	15.12	5.46	15.68	5.67
29	51	12.18	4.16	12.76	4.37	13.34	4.56	13.92	4.76	14.50	4.96	15.08	5.16	15.66	5.35	16.24	5.56
30	50	12.60	4.09	13.20	4.28	13.80	4.47	14.40	4.67	15.00	4.86	15.60	5.06	16.20	5.25	16.80	5.45
31	49	13.02	4.00	13.64	4.20	14.26	4.38	14.88	4.57	15.50	4.77	16.12	4.96	16.74	5.15	17.36	5.34
32	48	13.44	3.92	14.08	4.11	14.72	4.29	15.36	4.48	16.00	4.67	16.64	4.85	17.28	5.04	17.92	5.23
33	47	13.86	3.84	14.52	4.02	15.18	4.21	15.84	4.39	16.50	4.57	17.16	4.75	17.82	4.94	18.48	5.12
34	46	14.28	3.76	14.96	3.94	15.64	4.11	16.32	4.28	17.00	4.47	17.68	4.66	18.36	4.83	19.04	5.01
35	45	14.70	3.67	15.40	3.85	16.10	4.02	16.80	4.20	17.50	4.38	18.20	4.55	18.90	4.73	19.60	4.90
36	44	15.12	3.60	15.84	3.77	16.56	3.94	17.28	4.11	18.00	4.28	18.72	4.45	19.44	4.62	20.16	4.79
37	43	15.54	3.51	16.28	3.68	17.02	3.84	17.76	4.01	18.50	4.18	19.24	4.35	19.98	4.51	20.72	4.68
38	42	15.96	3.43	16.72	3.60	17.48	3.76	18.24	3.92	19.00	4.09	19.76	4.25	20.52	4.41	21.28	4.57
39	41	16.38	3.35	17.16	3.51	17.94	3.67	18.72	3.83	19.50	3.99	20.28	4.15	21.06	4.30	21.84	4.46
40	40	16.80	3.27	17.60	3.42	18.40	3.58	19.20	3.73	20.00	3.89	20.80	4.05	21.60	4.20	22.40	4.36

Smalley Foundation Analytical Results

Report on Cooperative Cottonseed Meal Analyses Conducted by American Oil Chemists' Society, 1930-31

By A. W. PUTLAND, *Chairman*

THE tables attached to this report summarize the results of the cooperative analytical program of the Smalley Foundation for the past year. The program was concluded, as usual, with thirty samples. There were 99 collaborators participating, as compared to 96 for the season 1929-1930, and 102 for the season 1928-1929.

In Table No. 1 we show the standing of the 45 collaborators who reported oil determinations on all samples. In the two preceding years 45 and 46, respectively, reported oil determinations on all the samples.

Table No. 2 shows the standing of the 71 collaborators who reported ammonia results on all samples. This number compares with 75 and 74, respectively, for the two preceding seasons.

Table No. 3 gives the average for both oil and ammonia for the 45 collaborators who reported on both oil and ammonia on all samples. In the two preceding seasons 45 and 46 collaborators reported oil and ammonia on all samples.

The winning collaborators are as follows:—

The Battle Cup for the highest efficiency in the determination of both oil and ammonia on all samples is awarded to Analyst No. 19, Dr. W. F. Hand, State Chemist, Mississippi A. and M. College, whose average is 99.889 per cent. The average efficiency is slightly less than that of last year obtained by the Southwestern Laboratories with an average efficiency of 99.95½ per cent, but somewhat higher than that obtained by the winner the year previous. The certificate for second place goes to Analyst No. 49, The Southwestern Laboratories of Dallas, Texas, with an efficiency of 99.866. It is interesting to note that last year the Southwestern Laboratories was awarded the cup for an efficiency of 99.956½ per cent, while this year the same laboratory was awarded second prize with an efficiency of 99.866 per cent. It is also interesting to note that Dr. W. F. Hand, who was awarded second place last year with an efficiency of 99.926 per cent, has been awarded first place this year with an efficiency of 99.889 per cent. The efficiency of the work this year appears to be less than that of last year, a possible reason of which will be offered later. The winner of the cup this year, Dr. W. F. Hand, also won the cup in 1926-27.

The certificate for the highest efficiency in determination of the oil only is awarded to Dr. W. F. Hand, State Chemist, Mississippi A. & M. College, whose average is 99.866 per cent. The certificate for second place goes to Mr. D. B. McIsaac, International Vegetable Oil Company, Savannah, Ga., analyst No. 4, with an efficiency of 99.804 per cent. The percentage of the winner last year was 99.938 and for second place 99.918.

The certificate for the highest efficiency in the determination of ammonia is awarded to Analyst No. 14, Mr. Geo. K. Redding, The Larrowe Milling Company, Rossford, Ohio, with an average of 99.980 per cent and the certificate for second place goes to No. 49, The Southwestern Laboratories, Dallas, Texas. The foregoing comparisons show that the percentage efficiency for both oil and ammonia, as well as for the combined oil and ammonia work, is slightly less than for last year.

There have been comparatively few complaints from the collaborators regarding the samples this year. This in spite of the fact that some abnormal meal samples were sent out. These abnormal samples possibly explain the slightly less efficiency obtained by the collaborators this year than that obtained last year. The few complaints were registered against including the results of the sample which contained an unusually high oil content. The Committee felt that our official method should provide for high or low oil content samples. If it does not, then it is not a method. We, therefore, ruled that this and other samples would be included in the final results. The Smalley Foundation Cooperative analytical program is not a contest but a means of checking our official methods in the hands of numerous operators as well as checking our individual work.

In concluding this report your Committee feels that the Society owes Mr. Thos. C. Law a tremendous debt for his care and attention in preparing and mailing the samples.

Personnel of Committee:—N. C. Hamner, L. C. Haskell, H. C. Moore, L. B. Forbes, E. A. Butt, G. K. Witmer, A. W. Putland, Chairman.

TABLE NO. I

Analyst No.	Points off	% Efficiency
19	28	99.866
4	41	99.804
93	45	99.784
49	50	99.761
31	52	99.751
80	53	99.746
86	54	99.741
58	62	99.703
7	63	99.697
37	69	99.668
84	77	99.631
43	80	99.617
9	81	99.611
40	83	99.602
13	84	99.596
26	96	99.539
70	97	99.534
15	101	99.516
6	103	99.506
53	108	99.472
69	113	99.458
92	113	99.458
2	119	99.429
79	124	99.409
35	127	99.39
77	132	99.365
83	132	99.365
3	136	99.347
71	139	99.332
60	141	99.321
56	149	99.284
58	149	99.284
81	150	99.279
10	155	99.256
75	159	99.221
5	162	99.221
74	186	99.106
59	193	99.072
66	196	99.058
20	202	99.029
45	214	98.971
62	311	98.51
83	340	98.36
72	672	96.77

TABLE NO. II

Analyst No.	Points off	% Efficiency
14	5	99.98
49	7	99.972
34	10	99.959
55	10	99.959
5	14	99.9424
89	15	99.938
53	16	99.9325
93	18	99.925
86	18	99.925
84	20	99.9168
61	20	99.9168
19	21	99.913
71	22	99.909
80	22	99.909
4	30	99.874
59	30	99.874
10	31	99.871
58	33	99.863
60	33	99.863
8	33	99.863
68	36	99.850
32	36	99.850
36	37	99.846

TABLE NO. II (Cont'd.)

Analyst No.	Points off	% Efficiency
33	38	99.842
7	39	99.837
2	40	99.833
43	41	99.829
24	42	99.825
26	43	99.821
39	43	99.821
9	44	99.817
74	45	99.813
66	47	99.805
31	47	99.805
70	48	99.8
38	49	99.796
40	51	99.788
81	53	99.780
91	54	99.775
35	56	99.768
54	57	99.766
76	58	99.756
15	61	99.744
3	63	99.738
75	67	99.721
18	67	99.721
27	67	99.721
69	68	99.719
6	68	99.719
56	72	99.700
92	74	99.692
65	78	99.674
37	81	99.796
83	84	99.65
72	86	99.641
30	89	99.630
22	93	99.612
13	94	99.608
16	106	99.558
88	106	99.558
73	116	99.517
77	125	99.482
62	128	99.457
25	129	99.462
21	130	99.46
45	131	99.455
79	151	99.37
20	153	99.362
17	163	99.333
1	166	99.309
64	348	98.548

TABLE NO. III

Analyst No.	% Efficiency
19	99.889
49	99.866
93	99.854
4	99.839
86	99.833
80	99.827
31	99.778
68	99.776
84	99.775
7	99.767
37	99.732
43	99.723
9	99.714
53	99.702
40	99.695
25	99.680
70	99.677
2	99.631
15	99.630
71	99.620
62	99.612
13	99.602
60	99.592
69	99.588
5	99.581
35	99.579
92	99.575
58	99.573
10	99.563
3	99.542
81	99.529
56	99.492
59	99.473
75	99.471
88	99.461
74	99.459
66	99.431
77	99.422
79	99.387
20	99.145
83	99.005
62	98.988
45	98.758
72	98.205

Notes of the Industry

Large Whaler Makes Port

On Saturday, April 18th, the Sir James Clarke Ross docked at Pier 20, Staten Island, New York, after a most successful voyage to the Antarctic seas. The ship is the largest cargo vessel afloat and has the additional distinction of being the largest and most modern factory ship in operation.

The modern whale handling and oil rendering equipment installed in the Ross has reduced the time of processing a whale to half of what was formerly required for a mammal of equal size. The quality of the oil obtained is very much improved, also. The processing is very simple in procedure, consisting essentially of live steam pressure rendering (60 lbs. pressure) of the blubber, meat and bone, following sedimentation of the oil for the removal of moisture. The oil is then pumped to the vessel's own storage tanks.

The Sir James Clarke Ross has deck room for handling six whales simultaneously, with three steam winches for hoisting the whales from the water through a large stern entrance chute onto the deck. There are thirty bone digesters and eight large rendering cookers for handling blubber and meat, together with a sufficient number of settling tanks to keep the rendering tanks free for continuous operation. Belt conveyors are used throughout the ship for the transport of solid materials. The bone oil is kept separate from the blubber and meat oil in the storage tanks.

The cargo landed by the Ross on this trip totaled 18,600 tons of oil. The shop could have brought a larger amount but the consignee's storage facilities were filled to capacity by this amount.

In the whaling operations in the Antarctic the whalers' crew are paid on a profit-sharing and bonus basis. The highest individual returns are amassed by the harpoon gunners, upon whose accuracy of aim the success or failure of a whaling voyage is so dependent. One of the Ross's harpoon gunners earned a total of $35,000 on the last voyage.

The members of the Mayonnaise Manufacturers Association have voted almost unanimously in favor of the Association's simplification program which contemplates the reduction of the number of sizes (not styles) of standard mayonnaise containers.

Mayonnaise Research Progresses

At a meeting of the Research Committee of the Mayonnaise Manufacturers Association, held in New York on April 9th, many problems of interest to manufacturers of mayonnaise were discussed. The Chairman of the committee, D. M. Gray read an interesting report relative to experiments on resistance to corrosion by several well-known anti-corrosive metals and alloys. The use of sugars other than sucrose in mayonnaise was discussed. L. B. Kilgore, Research Fellow of the Association, described the "Plumit," a newly developed instrument for the determination of relative density of mayonnaise mixtures. He further discussed the research plan of the Fellowship, enumerating the divisions of the contemplated work as follows:

1. Evaluation of emulsifying materials. 2. Effect of acidity on the development of rancidity in oils. 3. Color tests on fats and oils for the determination of incipient rancidity and constituents of oils producing such color reactions. 4. Methods of analysis. Determination of cholesterol volumetrically and by the zanthate method. 5. Practical tests of the value of malt sugar and other materials advocated for the improvement of mayonnaise. 6. Research on new methods of analysis. 7. Preparation of a rating or scoring card for the evaluation of commercial mayonnaise. 8. Bibliography.

According to the Georgia Agricultural Experiment Station, the varieties of soy beans adapted to the southeastern states are distinctly different from those cultivated in the north. The three varieties said to be most suitable for growth in Georgia are the Otootan, Laredo and Biloxi. The Experiment Station will furnish specifications of these varieties to those interested.

The dates and place for the Fifth Annual Convention of the Mayonnaise Manufacturers Association have been set. The meeting will be held at the Hotel Stevens, Chicago on October 26-27-28. Plans are already under way for the program. Frank Honicker is Executive Manager of the Association with offices at 1500 Walnut St., Philadelphia.

Referee Board Report

DURING the year 1930-31 the Referee Board granted certificates to only two new referees—William Black, Augusta Ga., and Clinton Morris, of the Morris-Flinn Laboratories, Macon, Ga. The following referees were re-certified for 1930-31:

Limited Referee Certificates. (All Products of the National Cottonseed Products Association). E. G. Williams, New Orleans, La.; G. K. Witmer, Battle Laboratories, Montgomery, Ala.; N. C. Hamner, Southwestern Laboratories, Dallas, Tex.; R. H. Fash, Fort Worth Laboratories, Fort Worth, Tex.; P. D. Cretien, Texas Testing Laboratories, Dallas, Tex.; F. B. Porter, Fort Worth Laboratories, Fort Worth, Tex.; T. C. Law, Law & Company, Inc., Atlanta, Ga.; P. McG. Shuey, Shuey & Co., Savannah, Ga.; F. R. Robertson, Houston Laboratories, Houston, Tex.; L. B. Forbes, L. B. Forbes Laboratories, Little Rock, Ark.; E. H. Tenet, L. B. Forbes Laboratories, Little Rock, Ark.; H. M. Shilstone, New Orleans, La.; J. C. P. Helm, New Orleans, La.; J. R. Mays, Barrow-Agee Laboratories, Shreveport, La.; E. R. Barrow, Barrow-Agee Laboratories, Memphis, Tenn.; G. W. Agee, Barrow-Agee Laboratories, Memphis, Tenn.; B. L. Caldwell, Barrow-Agee Laboratories, Jackson, Miss.; A. H. Preston, Southwestern Laboratories, San Antonio, Tex.; R. G. Huffman, G. W. Gooch Laboratories, Los Angeles, Cal.; S. Lomanitz, General Laboratories, Oklahoma City, Okla.; H. M. Bulbrook, Industrial Laboratories, Fort Worth, Tex.; F. C. Schilling, Industrial Laboratories, Fort Worth, Tex.; C. W. Rice, C. W. Rice & Co., Columbia, S. C.; N. E. Katz, Meridian, Miss.; D. C. Picard, Picard Laboratories, Birmingham, Ala.; W. J. Bramblett, San Antonio Laboratories, San Antonio, Tex.; F. Paquin, Galveston Laboratories, Galveston, Tex.; Curtis & Tompkins, Ltd., San Francisco, Cal. Limited to Cottonseed Cake, Meal & Feed Products. R. M. Chapman, Indiana Laboratories, Hammond, Ind.; S. W. Wiley, Wiley & Co., Baltimore, Md. Limited to Fish & Vegetable Oil, Cake & Meal. Laucks Laboratory, Seattle, Washington. All Products covered by the Methods of the American Oil Chemists' Society. H. P. Trevithick, Bureau of Chemistry, New York, N. Y.

So far this season the Referee Board has sent out two samples of crude oil and one sample of yellow oil as check samples to the several Referees. On the first sample of crude oil twenty-four out of twenty-eight laboratories check within + or —.5% of the refining loss and twenty-one within .5 on color. Thirteen check within .2% on refining loss and sixteen within .2 on color.

On the second sample of crude oil sent out twenty-three out of twenty-eight check within .5% on refining loss and seventeen within .5 on color. Seventeen check within .2% on refining loss but only six come within .2 of the average on color.

On the sample of refined oil sent out for color reading, out of a total of thirty-one figures, twenty-three were within .5 red and fifteen were within .2 red.

These figures would appear to indicate that refining loss results, on the whole, are in very good agreement, but that there is a wide range in color reading. The Committee feels that this check work is important and should be continued more extensively then has been done in the past.

W. H. Irwin, *Chairman.*
W. R. Stryker
L. C. Haskell
H. Aspegren
F. Paquin

Chicago, April 13, 1931.

Attempts to establish a tung oil industry on a commercial footing in New Zealand are being followed with great interest in that dominion. Until a few years ago, the growing of the tung oil tree was confined to China, but the American plantations are new reaching the profit-earning stage and it is from these that the promoters of the New Zealand schemes have secured their seeds.

Commercial planting in New Zealand has not extended over a long enough period for the trees to have reached the nut-bearing stage, but there is every indication from the growth already made that both the soil and the climate are well suited to their needs. Two large companies have started operations this year in the north of the dominion, and efforts are being made to interest farmers in the surrounding districts to take up the cultivation of the trees as side-lines to their normal activities. There are indications, therefore, according to the expectations of the promoters, that New Zealand will have a new industry of prime importance.

Olive Oil Report

(*From Page* 181)

to reject the coin test because of its failure to show that amount. That is why, in this report I have omitted tests made on smaller amounts, on which there are conflicting opinions. If we try to make the test quantitative, we are apt to defeat the purpose. A positive test on the coin or the benzoate is clear enough evidence of the presence of sulfur extracted oils, even though a negative test may not prove that such oils are not there.

We have all agreed on the Beilstein test. We have reached substantial agreement on the iodine test on sample No. 4, and, therefore, conclude that the method of the A.O.C.S. is satisfactory as applied to olive oils, with the time element a factor in the achievement of closely agreeing results.

We, therefore, recommend to the consideration of the Uniform Methods and Planning Committee, the incorporation into the methods sanctioned by the A.O.C.S., the following tests:

1. The Beilstein test for the detection of halogenated solvents in oils, (this for oils in general, not just for olive oil).
2. The Silver Coin test for "Sulfur Olive Oils" or Carbon Bisulfide Extracted Olive Oils.
3. The Silver Benzoate test for "Sulfur Olive Oils" or Carbon Bisulfide Extracted Olive Oils.

These tests are herewith appended, as recommended.

1. *Beilstein Test for the detection of halogenated solvents in oil.* A copper wire that has been previously heated in the Bunsen flame until no green color is imparted, is then dipped into the oil to be tested. It is then placed in the flame. Any traces of chlorine in the oil will give a strong green color to the flame.

2. *Coin Test for Sulfur Olive Oils (Carbon bisulfide extracted).* Pour approximately 20 cc. of the oil to be tested into a large test tube. Suspend a bright new dime by means of a glass rod so shaped for the purpose, or upon broken pieces of glass, in such a way that it is completely surrounded by the oil. The test tube is then placed in an oil bath and so heated that the oil in question reaches a temperature of 210° C., within one-half hour. Maintain this temperature for at least ten minutes longer. After cooling, the coin is examined for color. If it is definitely tarnished or blackened, the test is positive and indicates the presence of sulfur compounds in the oil tested.

3. *Silver Benzoate Test for Sulfur Olive Oils (Carbon bisulfide extracted).* Heat 5 cc. of the oil to be tested in a small test tube placed in an oil bath already at a temperature of above 150° C. so that the oil in the test tube reaches this 150° C. in five minutes at the most. This is to prevent scorching of the oil or undue contact with heat, as the silver benzoate test is a sensitive one. The tube is removed from the bath. A pinch of powdered silver benzoate is dropped into the oil and shaken at once. Any sulfur compounds present will almost immediately darken the oil appreciably as compared against the same oil untreated with benzoate but heated at the same time to the same degree of temperature.

Fish Oil for Poultry

The development of biologically tested sardine oil for use as a substitute for codliver oil in poultry feeding has been undertaken on the Pacific coast. As yet no commercial quantities of the oil are on the market, but at least one producer and one large dealer are working on the idea. It is understood that tests conducted by the producer, a large California fishing concern, have been very satisfactory and the head of the fish oil department of the large dealer mentioned is now in New York, presumably in the interests of further development work along this line.

It was stated by one man closely connected with fish oils that the sardine oil so tested and found satisfactory for poultry use will easily bring 50 cents per gallon, against approximately $1.50 per gallon for poultry codliver oil and 22 cents per gallon for ordinary sardine oil.

The Standards and Research Committee of the Mayonnaise Manufacturers Association held its spring meeting at the Hotel New Yorker, New York, on Thursday, April 9. The subjects discussed included metal corrosion, corn sugar in mayonnaise, colloid mills, the Kreis test, standard methods of analysis and the mayonnaise research fellowship.

In the production of synthetic fats, a mixture of fatty acids containing about 35-65% of saturated fatty acids and the balance unsaturated fatty acids may be esterified by heating with a deficiency of glycerol (suitably under reduced pressure). Brit. Pat. No. 331,880.