Finally, it was necessary to establish whether the phosphorus content obtained by the two methods of extraction were genuine phosphatides. The quantities obtained were naturally rather small but it was possible to refine some of the crude material by repeated precipitation with acetone from ether solution. Of this refined material 0.092 grams gave 3.39% P, an indication that the substance is nearly a pure plant phosphatide. Another sample, not so highly refined, was analyzed for phosphorus and nitrogen to establish the P:N ratio. The waxy, brown ether soluble substance contained 1.81% P and 0.95% N, showing that it was only of about 50% strength. The P:N ratio of 1.1:1 is a very satisfactory result considering the small quantities of material available.

It was not originally intended to go further into the question of caffeine extraction, but as it was relatively easy to extract, it was considered worthwhile to investigate the question further, especially as the usual caffeine extraction is rather tedious and frequently gives rise to colored products. The caffeine extracted with alcohol-benzene crystallizes out in faintly yellow crystals in a nearly pure state. The yields of caffeine in Table 1 correspond to the figures of many previous authors. Only in one case was the residual caffeine left after the alcohol-benzene extraction investigated. After the usual light petroleum and alcohol-benzene extraction a sample of unroasted Cameroon coffee was treated in the usual way for the extraction of caffeine and only yielded 0.18% of caffeine. It therefore seems probable that the alcohol-benzene would remove all the caffeine if the extraction were carried out long enough.

Conclusion

Fresh green coffee beans contain a rather high percentage of phosphatides in the light petroleum extract as well as in the subsequent alcohol-benzene extract. Nearly all these phosphatides are destroyed by roasting. By using alcohol-benzene as a solvent the major portion—if not all—of caffeine is extracted also and can easily be isolated in a pure state.

I desire to express my thanks to Charles M. Caines who has been conducting a good deal of the analytical work.

REFERENCES

1. H. Meyer and Eckert, Monatshefte *31*, 1227 (1910).
H. A. Schnette, M. A. Cowley and C. Y. Chang, Journal A.C.S. *56*, 2085 (1934).
A. Heiduschka and R. Kuenn, Journal Practische Chemie *139*, 269 (1934).
K. H. Bauer and R. Kuehn, Fette and Seifen *45*, 229 (1938).
A. Steger and Ivan Loon, Raceuil Travaux Chim. *53*, 28 (1934).
R. D. de G. Paula, Annal. Asso. Qin. Brasil *2*, 57 (1943).
2. Koenig, Die Menschlichen Nahrungs und Genussmittel *1*, 1116 (1933).

Report of the Committee on Analysis of Commercial Fats and Oils

Fat Stability Test

The Committee has not done any collaborative work on the fat stability test during the past year. However, some work has been going on in one of the laboratories, investigating some of the variables involved in the test. There is nothing definite to report at this time.

Unsaponifiable Matter

A group of samples was prepared by adding known quantities of various types of unsaponifiable matter to coconut oil. Coconut oil was used because of its natural low content of unsaponifiable matter. These samples were submitted to a subcommittee who determined the unsaponifiable matter by several selected methods. The results are shown in Table 1.

TABLE 1

Laboratory	Method	% Unsaponifiable Matter in Refined Coconut Oil				
		Refined Coconut Oil	Plus 2% Cholesterol	Plus 2% Lanolin	Plus 2% Lecithin	Plus 1% Denaturing Oil
1	Kerr-Sorber (a)	0.60	0.68	2.46	1.85	1.12
2	Kerr-Sorber (a)	0.27	1.98	1.19	0.33	0.90
3	Continuous (b)	0.27	2.10	0.83	0.35	0.70
4	Continuous (b)	0.32	2.20	1.21	0.23	0.38
5	S. P. A. (a)	0.25	2.17	1.28	0.34	0.78
6	S. P. A. (a)	0.28	2.19	1.35	0.28	0.25
2	S. P. A. (a)	0.55	2.49	1.50	0.62	0.61
5	F. A. C. (b)	0.19	2.07	1.06	0.26	0.84
6	F. A. C. (b)	0.20	1.92	1.03	0.26	0.44

(a) Ethyl ether.
(b) Petroleum ether.

Attempts to use ethyl ether with the continuous and F.A.C. methods were generally unsatisfactory, due to the formation of emulsions.

In the case of the original oil all methods gave results within reasonable agreement. When unsaponifiable matter was added, there was a tendency for the SPA method to yield the highest results although there was little to choose between it and the continuous method. Denaturing oil is not completely recovvered by any method.

A comparison was made between vacuum and air oven drying. The results appear in Table 2:

TABLE 2

Sample	% Unsaponifiable Matter After Drying in	
	Air oven at 101-103°C. for 15 minutes	Vacuum oven at 70°C. for 30 min. and an absolute pressure of about 112 mm. of Hg.
Refined coconut oil	0.19	0.19
Refined coconut oil	0.25	0.25
Plus 2% cholesterol	2.07	1.95
Plus 2% cholesterol	2.18	2.17
Plus 2% lecithin	0.25	0.25
Plus 2% lecithin	0.37	0.30
Plus 2% lanolin	1.06	1.01
Plus 2% lanolin	1.28	1.28
	Air Oven at 101°C. for 15-20 minutes	Vacuum oven at 56°C.. and an absolute pressure of 4 mm. Hg. to constant weight
Soap	0.66	0.72
Brown grease	2.00	2.40
Light grease	0.86	0.91
Brown grease	1.88	1.90
Yellow grease	0.70	0.70
Yellow grease	0.96	0.93

Further work will be done on drying methods with special reference to the recovery of denaturing oils because it is probably at this point that this material is lost. Further work will also be done on the comparison of methods.

Congeal Point

Continuing the work started last year on the congeal point, another group of samples was submitted to a subcommittee for trial with some modification of the methods used last year. The results are shown in Table 3:

TABLE 3

Laboratory	Samples 16	17	18	19
	°C.	°C.	°C.	°C.
1	30.2	31.0	32.3	29.6
2	30.6	31.8	33.0	31.2
3	31.3	31.8	34.0	31.4
4	31.9	36.3	37.4	35.7
5	30.0	30.4	30.5	28.8

Although in a few instances there was agreement between different members, generally speaking there was no improvement over results previously reported [Oil & Soap, *22*, 101-107 (1945)]. Further study of this method will be necessary before any recommendation can be made. A motion was made, seconded, and carried at the November committee meeting to issue the statement that the committee has found none of the present methods satisfactory and does not recommend their use for the evaluation of solid fats.

TABLE 4

Spectrophotometric Analyses

A.O.C.S. Collaborative Samples—Soap

Laboratory	Non-Conjugated Linoleic	Non-Conjugated Linolenic	Non-Conjugated Arachidonic	Conjugated Diene	Conjugated Triene	Conjugated Tetraene
			Soap No. 1			
6	2.22	0.86*	0.18	0.61	0.007	0.001
4	1.90	0.33	0.18	0.66	0.01	0.00
1	1.88	0.36	0.17	0.63	0.006	0.0008
2	1.72	0.33	0.13	0.57	0.007	0.000
7	1.98	0.31	0.15	0.61	0.01	0.000
8	2.04	0.39	0.14	0.65	0.012	0.0004
9	2.07	0.42	0.18	0.64	0.009	0.000
10	1.93	0.38	0.15	0.64	0.01	0.001
Average	1.97	0.35	0.16	0.63	0.009	......
Std. deviation	0.14	0.036	0.019	0.028	......	......
			Soap No. 2			
6	2.20	0.82*	0.14	0.57	0.008	0.001
4	2.12	0.32	0.20	0.62	0.01	0.000
1	2.07	0.46	0.12	0.61	0.009	0.0008
2	1.85	0.31	0.16	0.51	0.008	0.000
7	2.26	0.36	0.18	0.56	0.01	0.000
8	......	......	......	......	......	......
9	2.28	0.47	0.19	0.59	0.011	0.000
10	2.18	0.36	0.16	0.60	0.01	0.001
Average	2.14	0.38	0.16	0.58	0.009	......
Std. deviation	0.14	0.063	0.026	0.035	......	......
			Soap No. 3			
6	2.38	0.65*	0.16	0.60	0.008	0.001
4	1.93	0.32	0.19	0.63	0.01	0.000
1	2.07	0.34	0.22	0.62	0.009	0.001
2	1.73	0.28	0.20	0.53	0.005	0.000
7	2.07	0.37	0.17	0.59	0.01	0.000
8	......	......	......	......	......	......
9	2.05	0.45	0.18	0.59	0.008	0.001
10	2.11	0.38	0.17	0.61	0.01	0.001
Average	2.05	0.36	0.18	0.60	0.009	......
Std. deviation	0.18	0.052	0.02	0.03	......	......
			A.O.C.S. Collaborative Samples—Tallow			
			Tallow No. 1			
6	2.95	0.44	0.06*	0.53	0.02	0.002
4	2.71	0.34	0.22	0.57	0.01	0.000
1	2.70	0.29	0.21	0.55	0.013	0.002
2	2.44	0.34	0.20	0.53	0.01	0.000
7	2.69	0.33	0.21	0.58	0.01	0.000
8	2.86	0.41	0.20	0.55	0.013	0.002
9	2.72	0.43	0.21	0.56	0.013	0.001
10	2.64	0.45	0.19	0.58	0.01	0.001
Average	2.73	0.38	0.21	0.56	0.012	......
Std. deviation	0.14	0.057	0.01	0.025	......	......
			Tallow No. 2			
6	2.10	0.47	0.09*	0.90	0.01	0.001
4	1.74	0.30	0.18	0.87	0.01	0.000
1	1.94	0.28	0.18	0.79	0.011	0.0006
2	1.63	0.37	0.21	0.76	0.01	0.000
7	1.86	0.30	0.15	0.83	0.01	0.000
8	......	......	......	......	......	......
9	1.80	0.42	0.15	0.82	0.007	0.001
10	1.74	0.36	0.13	0.81	0.01	0.000
Average	1.83	0.36	0.17	0.83	0.01	......
Std. deviation	0.14	0.065	0.026	0.044	......	......
			Tallow No. 3			
6	2.22	0.55*	0.06*	0.70	0.01	0.001
4	2.16	0.28	0.24	0.73	0.01	0.000
1	2.20	0.24	0.21	0.67	0.010	0.001
2	1.91	0.30	0.19	0.64	0.01	0.000
7	2.19	0.30	0.19	0.71	0.01	0.000
8	......	......	......	......	......	......
9	2.14	0.42	0.21	0.70	0.009	0.001
10	2.11	0.37	0.18	0.71	0.01	0.000
Average	2.13	0.32	0.20	0.70	0.01	......
Std. deviation	0.11	0.060	0.02	0.028	......	......

NOTE: Results marked with an asterisk not included in average or standard deviation.

F.A.C. Color Comparator

Mr. Whyte of Colgate-Palmolive-Peet Company has constructed and submitted another model of his proposed FAC color comparator. However, it has not been examined yet by all members of the subcommittee so that no action can be taken.

Stability of Sodium Thiosulfate Solution

The stability of sodium thiosulfate solution as influenced by the various suggested stabilizing agents is under investigation. The storage tests are not yet complete so that no conclusion can be made now.

TABLE 5

Tallow Refining—Bleaching Tests

Laboratory	1			2			3			4			5		
	FAC	Bleach Y	Bleach R	FAC	Bleach Y	Bleach R	FAC	Bleach Y	Bleach R	FAC	Bleach Y	Bleach R	FAC	Bleach Y	Bleach R
4	11A	8	1.2	15	30	3.2	9	30	2.4	15	30	5.6	13	7	1.3
2	11A	8	1.2	15	25	2.5	7	20	2.0	15	30	5.8	13	7	1.6
8	11A	8	1.2	15	22	3.2	7	17	2.0	15	30	4.9	13	7	1.7
9	11B	10	1.4	15	20	2.6	7	20	2.4	17	41	6.6	11	7	1.9
7	11B	9	0.9	11B	26	2.6	7	17	1.7	11B	50	5.4	11	9	0.9
Average	....	....	1.2	....	....	2.8	....	....	2.1	....	....	5.7	....	....	1.5
Std. deviation	....	....	0.16	....	....	0.31	....	....	0.27	....	....	0.56	....	....	0.35

Laboratory	6			7			8			9			10			11		
	FAC	Bleach Y	Bleach R	FAC	Bleach Y	Bleach R	FAC	Bleach Y	Bleach R	FAC	Bleach Y	Bleach R	FAC	Bleach Y	Bleach R	FAC	Bleach Y	Bleach R
4	9	6	0.6	11A	5	1.6	9	8	1.3	13	25	2.3	9	10	1.7	11	7	0.6
2	9	7	0.9	11A	8	1.6	9	9	1.4	13	20	2.5	9	15	2.0	11	8	0.9
8	9	7	1.0	11A	8	1.7	9	9	1.6	13	17	2.1	9	20	1.6	11	8	1.1
9	9	7	1.3	11A	7	1.7	11	10	1.9	11	13	2.0	11	10	2.0	11	6	1.1
7	9	7	0.7	11A	11	1.1	9	12	1.2	13	18	1.8	9	13	1.3	11	7	0.7
Average	....	....	0.9	....	....	1.5	....	....	1.5	....	....	2.1	....	....	1.7	....	....	0.9
Std. deviation	....	....	0.24	....	....	0.23	....	....	0.24	....	....	0.24	....	....	0.26	....	....	0.20

FAC color determined on original sample as submitted.

Standard Potassium Dichromate

A few years ago this Committee strongly urged the U. S. Bureau of Standards to prepare and maintain a standard sample of potassium dichromate to serve as a standard for oxidimetry and iodimetry. Such a sample is now available and may be purchased from the U. S. Bureau of Standards at Washington, D. C.

Spectroscopic Methods

A subcommittee undertook to analyze a group of soap and tallow samples by the spectroscopic method of the Rubber Reserve Company. The results obtained are shown in Table 4. A committee has recently been appointed on spectroscopic methods with R. C. Stillman as chairman, and any further work of this kind will be referred to that Committee.

Tallow Refining-Bleaching Test

Work was started this year in an effort to find some better means of evaluating fats from the standpoint of color for soap production. This began with a collaborative study using a method proposed by L. B. Parsons [Oil & Soap, *20*, 256-7 (1943)]. This method is not concerned with refining loss, but only with the bleached color. The data obtained on a series of samples appear in Table 5.

These samples were bleached with an activated bleaching earth. It is evident from these data that there is no correlation between the FAC color of the original fat and the color after refining and bleaching. Further work will be done this coming year on this method.

Rewriting Methods

During the past year a subcommittee has worked extensively with the editor of the Analytical Methods in reviewing the methods of the entire section on Sampling and Analysis of Commercial Fats and Oils for the new book of methods which is in preparation.

M. L. LAING
E. W. BLANK
E. W. COLT
F. G. DOLLEAR
J. FITELSON
J. C. KONEN
C. P. LONG
J. E. MARONEY
R. T. MILNER
L. B. PARSONS
H. A. SCHUETTE
L. M. TOLMAN
F. C. WOEKEL
V. C. MEHLENBACHER,
chairman

The Evaluation of Bleaching Earths*

The Adsorptive Capacity of Some Bleaching Earths of Various pH for Chlorophyll in Soybean Oil

HERBERT F. HINNERS, JUSTIN J. McCARTHY, and RAYMOND E. BASS
Lever Brothers Company, Edgewater, N. J.

IT is generally recognized that few, if any, types of bleaching earths are equally well suited for the adequate color reduction of all kinds of oils. Nearly all bleaching-earth manufacturers produce several types of adsorbents for industry, at times recommending a particular type for a specific oil. Assuming that the bleaching-earth producers know how to prepare a type of earth which is especially suitable for a very specific group of oils, it is still almost totally unknown why an earth prepared in a specific manner is suited for one type of oil and at the same time, *ineffective* for others. It is also interesting to note that this individual suitability of bleaching earths is sometimes very selective. An earth designated by the manufacturer for one group of oils will bleach some oils of this type to a greater extent than other oils of the same type (1).

Parsons (2) suggested that the decolorization of oils by fuller's earth was due to adsorption. Subsequent work has led to the general acceptance of this point of view. Many of the earlier adsorption studies were correlated by Rogers, Grimm, and Lemmon (3) who showed that in bleaching experiments, using a number of petroleum products and several different types of adsorbents, that Freundlich's (4) equation applies accurately when the amount of color removed is used as a measure of the amount of material adsorbed.

In attempting to follow this procedure in the decolorization of vegetable oils, one is confronted with the problem of determining the true color of the unbleached and bleached oil. Obviously, the Lovibond system of color matching, as is universally used today in the vegetable oil industry, is inadequate in this respect for it is impossible to obtain a satisfactory match by a combination of red and yellow glasses for an oil, such as soybean, which contains varying amounts of red, yellow, and green pigments (5). The values of a true color must be additive in the sense that the color, C_m of a mixture of two oils having colors C_a and C_b will be given by the equation:

$$C_m = \frac{C_a V_a + C_b V_b}{100}$$

where V_a and V_b respectively are percentages (by volume) of the oils whose colors are C_a and C_b (6). Therefore, in the present work, observations have been confined to the chlorophyll band of the spectrum, whereby more accurate and comparative values could be obtained spectrophotometrically to satisfy the conditions of the above equation.

The chemical properties of bleaching clays, natural and artificially activated, as well as activated carbons, have been studied extensively by many investigators, and it has been generally concluded that in most instances the bleaching action must be determined empirically because there appeared to be no definite relationship to chemically determinable values.

Parsons (7), however, in discussing the properties of fuller's earths, pointed out that all earths which had been found valuable for bleaching showed a distinct so-called "acid reaction." If a wetted sample was touched to neutral litmus paper, the paper turned

---

* Presented at the 19th annual Fall Meeting, American Oil Chemists' Society, Nov. 7-9, 1945, Chicago, Ill.