for pilot-plant extraction of the seed; and to V. L. Frampton for his suggestions and encouragement.

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Third Interim Report of the A.O.C.S.-A.O.A.C. Crude Fiber Liaison Committee, 1959-1960

THE SECOND INTERIM REPORT of the Crude Fiber Liaison Committee was presented at the October 1959 meetings of the American Official Agricultural Chemists, published in full in the Journal of the American Oil Chemists' Society (1), and summarized in the Journal of the A.O.A.C. (2). This report covered a collaborative study by the committee, utilizing four different methods for filtering the crude fiber after the digestion. It was concluded that none of the filtering devices tested showed sufficient advantage in accuracy or precision to warrant selection as a standard procedure. The Crude Fiber Committee however agreed that the screen was preferable to the cloth as a filtering medium. This limited the selection to the Oklahoma State Filter Screen and a new device submitted by Mr. Entwhistle of the California State Department of Agriculture and designated as the California State Modified Buechner Funnel. This device consists of a two-piece, 7-cm. diameter, polyethylene Buechner Funnel, into which a 200-mesh stainless steel screen has been heat-sealed. The device appeared to incorporate all of the desirable features of the Oklahoma State Filter Screen, the Buechner Funnel Method, and the Purdue Method. Because of these interesting features the committee decided to conduct another collaborative study, comparing the California State Modified Buechner Funnel against the Oklahoma State Filter Screen.

Twelve laboratories participated in this study, and six samples were submitted, including meat scraps, yeast, 44% soybean oil meal, cottonseed meal, mixed feed, and alfalfa. The A.O.C.S. statistical design was used, giving a total of 48 results from each laboratory for a grand total of 576 results. The results of this study are summarized in Table I; each figure shown is an average of four determinations.

A statistical analysis was made of these results. The standard deviations and 95% confidence limits obtained on each sample are shown in Table II. It will be noted that the yeast and alfalfa samples show standard deviations considerably higher than any of the other products analyzed. Yeast is normally difficult to analyze for fiber, and it is not surprising that precision obtained on this sample was poor. Likewise high fiber content of the alfalfa sample will affect the precision. In drawing conclusions on the adaptability of the method, these two samples might logically be eliminated.

Table III expresses precision of the methods on the basis of a 95% confidence limit. In addition to the results obtained in this collaborative study, we have included in Table III the results of the previous collaborative study as reported in the Second Interim Report.

A serious disadvantage to both the Oklahoma and California Method is the relatively large quantities of asbestos which must be employed to obtain rapid and efficient filtration. Preliminary investigations by some of the collaborators gave evidence that there is a loss in weight in the asbestos during the incinera-

TABLE I

Collaborative Data Comparing Oklahoma Filter Screen with California State Modified Buechner Funnel

Laboratory	Meat scraps		Yeast		S.B.O.M.		Cottonseed meal		Mixed feed		Alfalfa meal	
	O[a]	C	O	C	O	C	O	C	O	C	O	C
1	2.10	2.25	4.06	4.61	6.34	6.12	12.17	12.14	4.92	5.12	24.61	24.00
2	2.10	2.14	4.63	4.50	6.04	5.70	11.01	11.78	4.91	5.00	24.67	24.73
3	2.21	2.47	6.33	6.46	6.61	6.63	12.23	12.24	5.27	5.30	25.44	25.48
4	2.03	2.18	5.63	5.70	5.98	6.10	11.60	11.35	4.85	4.85	24.60	24.25
5	1.88	2.05	3.05	4.48	6.00	6.10	11.45	11.50	4.85	5.05	24.43	24.65
7	1.93	2.13	4.04	4.18	6.10	6.28	12.25	12.05	4.94	4.83	24.11	24.34
8	1.99	2.09	4.64	5.13	6.18	6.10	11.69	11.41	4.93	4.99	24.40	24.22
9	2.17	1.91	4.56	5.09	6.13	5.89	11.62	11.24	4.84	4.78	24.59	24.42
10	2.14	2.15	5.13	4.85	6.25	6.24	11.90	11.70	5.18	5.23	25.23	25.33
11	2.00	1.91	3.90	4.03	6.08	5.85	11.81	11.17	5.04	4.93	24.57	24.16
12	2.18	2.33	5.86	5.63	6.62	6.81	11.79	12.16	4.95	5.23	24.65	24.84
13	1.85	2.08	4.63	6.15	5.98	6.15	11.30	11.75	4.85	5.18	23.63	24.88
$\bar{x}$	2.05	2.14	4.69	5.07	6.15	6.16	11.80	11.71	4.96	5.05	24.57	24.66

[a] O—Oklahoma Filter Screen. C—California State Modified Buecher Funnel.
Note: Each result shown is the average of four determinations.

TABLE II

Standard Deviations and 95% Confidence Limits on Individual Samples

	Standard Deviation				95% Confidence Limits			
	Within labs.		Between labs.		Within labs.		Between labs.	
	Okla.	Calif.	Okla.	Calif.	Okla.	Calif.	Okla.	Calif.
Meat scraps	.14	.17	.17	.21	.40	.47	.48	.57
Yeast	.49	.51	.99	.89	1.36	1.41	2.76	2.47
44% S.B.O.M.	.15	.32	.23	.39	.43	.89	.62	1.09
Cottonseed meal	.24	.31	.36	.45	.65	.87	1.00	1.25
Mixed feed	.15	.12	.18	.21	.40	.34	.50	.57
Alfalfa meal	.48	.51	.61	.59	1.34	1.42	1.68	1.62
All samples	.32	.36	.52	.51	.87	.99	1.43	1.42

TABLE III

95% Confidence Limits Within and Between Laboratories on All Samples

	1959				1960	
	Modified Official	Okla. Screen	Buechner Funnel	Purdue Shimer	Okla. Screen	Calif. Buechner
Agreement within laboratories						
All samples	1.11	0.91	0.62	0.89	0.87	0.99
Less alfalfa	0.86	0.62	0.71	0.79	.....	.....
Less alfalfa and yeast	.....	.....	.....	.....	0.48	0.69
Agreement between laboratories						
All samples	1.58	1.41	1.27	1.27	1.43	1.42
Less alfalfa	1.28	0.93	1.08	1.26	.....	.....
Less alfalfa and yeast	.....	.....	.....	.....	0.68	0.92

tion that affects the accuracy of the crude fiber determination. The committee agreed that this apparent asbestos "blank" should be investigated and, if possible, eliminated. Rather than conduct collaborative work at this point, it was decided that investigational work by individual laboratories would be more fruitful. The following assignments were made: a) survey of asbestos used by the Liaison Committee members, by R.E. Anderson of the Archer-Daniels-Midland Company; b) survey of asbestos in crude fiber determination by F.W. Quackenbush of Purdue University; and c) effect of solvents on bound moisture in asbestos by J.P. Hughes of the Southern Utilization Research and Development Laboratory. These men have completed their assignments, and the results were reported at the October 1960 meetings of the A.O.A.C.

It is evident from the last two collaborative studies conducted by the Liaison Committee that little, if any, improvement can be expected in the precision of the Crude Fiber Method beyond what has been accomplished to date. We believe the committee has sufficient data at hand to write a method that will prevent the wide discrepancies in results which were noted by R.T. Doughtie Jr. in the A.O.C.S. Smalley Check Sample Program. We do not believe however we shall ever be able to attain a precision that will permit commodity trading on the basis of .1 or .2% crude fiber.

K.E. HOLT, chairman

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Some Effects of *y*-Radiation or Linoleate Peroxidation on *a*-Tocopherol[1]

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When *a*-tocopherol was irradiated in isooctane, the main product appeared to be a 5-exo-methylene tocopher-6-one derived by the abstraction of two hydrogen atoms from tocopherol. When tocopherol was irradiated in tributyrin, transesterification was found to be a major reaction. Results with three solvents show that the irradiation products of tocopherol are complex and dependent on the solvent.

In peroxidizing linoleic acid, *a*-tocopherol was oxidized to *a*-tocopheryl quinone, but no radical-tocopherol addition products were detected.

SOME of the most important reactions of tocopherols, the major lipid antioxidants in nature, are those with free radical intermediates of lipid peroxidation. When biological systems are subjected to ionizing radiations, there are similar reactions between free radicals and tocopherols. An important part of the damage of ionizing radiation to living organisms (7) and to food products (17), especially meats (6), is *via* free radical lipids. Besides its importance in protecting against radiation damage, tocopherol is the most labile of the fat-soluble vitamins (12).

There is little information available on these reactions between tocopherol and free radicals. One of the most significant studies is that of Inglett and Mattill (9,10). They reported on the products which they isolated after reaction of *a*-tocopherol (Ia in Figure 1) and 2,2,5,7,8-pentamethyl-6 hydroxychroman (Ib) with the relatively stable benzoyloxy and *t*-butoxy radicals. Most of these could be explained as addition products of the chromanoxy free radical (II) or the rearranged radical (III). They postulated that tocopherylquinone arose in their system through hydrolysis of the benzoyloxyl adduct of II,2,a. One product which Inglett tentatively charac-

[1] Supported in part by the Quartermaster Food and Container Institute for the Armed Forces.

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