mg./day/in.^{2}/in. thickness). For the series of foams from elaidinized castor oil, water-absorption values markedly decreased from 411 to 155% as oil content increased to 80%. This is a like trend but lower than the 515 to 170% range obtained for the analogous foams from castor oil. Results of water-vapor penetration tests, ranging from 236 to 545 mg./day/in.^{2}/in. thickness, indicated an increasing trend as oil content increased. Aging caused a slight increase in these values. The foams from the formulation containing 60% of elaidinized castor oil were the most resistant to water-vapor penetration.

Summary

The preparation and properties of two series of castor oil urethane foams, one from castor oil and the other from elaidinized castor oil, were investigated. The first series of foams was made from prepolymers containing 60% of castor oil prepared at increasing temperature levels to vary the degree of crosslinking in the final foams. These foams had lower tensile strengths than observed for a previously prepared foam of 60% castor oil and did not show significant differences in water resistance as crosslinking varied. They were increased nearly 100% in compressive strength with increased crosslinking and had very good shrinkage characteristics as values of only 1 to 2% were obtained.

A second series of foams was prepared from 50, 60, 70, and 80% of elaidinized castor oil to compare with foams from a similar series from castor oil. This series of foams of 50 to 80% elaidinized castor oil contents was similar in density (1.7 to 6.7 lbs./cu. ft.), had improved shrinkage characteristics (11, 1, 3, and 4%, respectively), showed increased compressive and tensile strengths (up to 12.1 p.s.i. at 50% compression modulus and 34.7 p.s.i. ultimate tensile for the 60% foam formulation), and had better water-resistance properties (411 to 155% *vs.* 515 to 170% water absorption) than the analogous foams from castor oil. In general, humid aging only slightly affected the values obtained for the foams and was significant in only a few instances, *e.g.,* decreased tensile in the elaidinized castor oil series.

Thus increasing crosslinks in the foam apparently did not improve water resistance but did improve shrinkage characteristics in addition to some increased strength properties, as would be anticipated. Foams from elaidinized castor oil, while similar in density and foaming characteristics to analogous foams from castor oil, exhibited less shrinkage and improved water-resistance.

Acknowledgment

The authors wish to acknowledge the assistance of J. P. Moreau for preparing and testing some of the foams, of V. P. Whitley for some of the oil analyses, and of The Baker Castor Oil Company for the castor oil used.

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[Received April 20, 1959]

Second Interim Report of the A.O.A.C.-A.O.C.S. Crude Fiber Liaison Committee

THE INITIAL INTERIM REPORT of the A.O.A.C.-A.O.C.S. Crude Fiber Liaison Committee was published in the May 1959 issue of the Journal of the A.O.A.C.(1). In this initial report the need for improvement in the precision and accuracy of the current official crude fiber method was discussed. The method itself was also discussed in detail, indicating those areas in the method where deviations or lack of operator techniques could result in loss of accuracy and precision.

In this initial report it was announced that a collaborative study comparing the official method (2, 3) with the Oklahoma State Filter Screen method (4) and a method involving the use of a Buchner Funnel (5) would be conducted by the Liaison Committee. Prior to the submitting of samples, a fourth method was submitted to the Liaison Committee by F. W. Quackenbush and E. D. Schall of Purdue University. This method utilizes a Shimer filtering funnel and, for the purposes of identification, will be referred to as the Purdue Method. The committee was intrigued by the apparent simplicity of the filtering operation using the Shimer Funnel and decided to include this method as a part of the collaborative study. Thus a total of four methods was compared.

In order to obtain complete data, that is, within-laboratory and between-laboratory precision, the A.O.C.S. statistical design was used. This requires two operators in each laboratory to run each sample by each method in duplicate on two different days. Twelve of the committee members agreed to participate, and four samples including a 50% soybean oil meal, a cottonseed meal, a dairy feed, and alfalfa meal were submitted to each laboratory. Using the design described above, each collaborator reported 16 results on each method, or a total of 64 results. More than 750 results were obtained and subjected to statistical analysis.

The methods as they were used in this collaborative study are described below. It will be noted that the collaborators were permitted some options in the choice of equipment and reagents. These are explained in the initial Interim Report.

Methods

The following reagents and apparatus were common to all methods used in the collaborative study, and in the interest of brevity only the special items will be listed under each test method.

A. Reagents

1. Sulfuric acid solution containing 1.25 g. H_2SO_4 per 100 ml.
2. Sodium hydroxide solution containing 1.25 g. NaOH per 100 ml.
   NOTE: The strength of the acid and alkali solutions must be accurately checked.
3. Asbestos, acid-washed, long fiber. Ignite for 1 hr. in a muffle furnace at 650°–700°C. Store in a closed container to avoid contamination. Asbestos cleaned from ignited crucibles may be used over for subsequent fiber determinations.
4. Ethyl alcohol 95%.
5. Ethyl ether A.C.S. grade or petroleum ether A.O.C.S. specification H 2-41.
6. Dow Corning antifoam AF emulsion.

B. Apparatus

1. Extraction apparatus with condenser to fit a 600-ml. tall form beaker and a hot plate adjustable to a temperature that will bring 200 ml. of distilled water at 25°C. to a rolling boil in 15 ± 2 min.
2. 600-ml. tall form lipless beakers.
3. Alundum crucibles No. 5204 porosity RA 766 (minimum weight 15 g. — maximum 20 g.) or Gooch crucibles, 40-ml. capacity, Coors No. 270, size No. 4A.
4. Air oven maintained at 130° ± 2°C.
5. Electric muffle furnace with rheostat control and pyrometer.
6. Desiccator with efficient desiccant. (Calcium chloride not satisfactory, drierite 4–8 mesh is satisfactory.)
7. Filtering device consisting of a suction flask with a suitable holder for the alundum or Gooch crucible (Filter ring Sargent No. 2 with 60° fluted funnel is satisfactory).
8. Apparatus designed to preheat alkali, acid, and wash water.

C. Preparation of Sample

1. To eliminate the possibility of fineness of grind affecting the method precision, all samples used in this study were ground before they were sent to the collaborators.

Method I. Official A.O.C.S.—A.O.A.C. (modified to incorporate ideas presented in initial report of Liaison Committee)

A. Reagents

1. through 6. as listed for all methods.

B. Apparatus

1. through 8. as listed for all methods.
9. Filtering cloth, of such character that no appreciable solid matter passes through when filtration is rapid. No. 40 filtering cloth made by National Filter Cloth and Weaving Company recommended.
10. Fluted glass funnel.

C. Procedure

1. Extract 2 g. of the air-dry material with ethyl or petroleum ether, or use residue from oil determination (A.O.C.S. Official Method Ba 3-38), and transfer residue, together with *ca.* 0.5 g. of asbestos, to 600-ml. beaker.
2. Add 200 ml. of boiling H_2SO_4 and 1 to 2 drops of Dow Corning AF emulsion. Place the beaker and contents on the hot plate, preheated to temperature specified in B-1. Reflux for 30 min. at this temperature, rotating periodically to keep any solid material from adhering to sides of the beaker.
3. At expiration of 30 min. remove flask, filter immediately through filtering cloth in fluted funnel, and wash with boiling H_2O until washings are no longer acid.
4. Wash charge and asbestos back into the 600-ml. beaker, using exactly 200 ml. of the boiling NaOH solution to effect the transfer.
5. Replace beaker on the hot plate and boil for exactly 30 min.
6. At expiration of 30 min. remove beaker and immediately filter through a Gooch crucible prepared with an asbestos mat or through an alundum crucible.
7. Wash residue with 25 ml. of boiling H_2SO_4 solution (1.25%). Follow with boiling H_2O and finally with 15 ml. of alcohol.
8. Dry crucible and contents in oven at 130°C. for 2 hrs., desiccate, and weigh.
9. Place in muffle at 650°C. for 30 min., remove, desiccate, and weigh. Record loss in weight as crude fiber.

D. Calculations

$$\text{Crude fiber, } \% = \frac{\text{loss in weight} \times 100}{\text{weight of sample}}$$

Method II. Oklahoma State Filter Screen

A. Reagents

1. through 6. as listed for all methods.

B. Apparatus

1. through 8. as listed for all methods.
9. Filter screen (Figure 1) attached to a vacuum and compressed air by means of a two-way stopcock. Suction should contain reservoir to catch filtrates.
   NOTE: Filter screen shown in Figure 1 is simplest design. A filter screen attached to the head by a threaded ring was fabricated and used by some of the collaborators. This permitted removal of the screen for easy cleaning.

C. Procedure

1. Follow C-1 Method I.
2. Follow C-2 Method I.
3. Remove at the end of the 30-minute period and insert the filter screen (with suction on) with the face of the screen just under the surface of the liquid. Keep the screen in this position until the liquid is completely removed.

   Without breaking the suction or raising the screen, immediately add 50–75 ml. of boiling water and remove by means of suction as before. Repeat with three more 50-ml. washings.
   NOTE: All washings should be made as rapidly as possible to prevent the mat from becoming dry, thereby slowing down the filtration rate.
4. Carefully remove and invert the screen holding it high enough to drain all the water from the line. Return screen to the beaker, turn off the suction, and allow the compressed air to pass back through the screen to loosen the asbestos sample mat. (If compressed air is not available, this operation can be accomplished by blowing back through the filter screen.)
5. Insert a spatula beneath the loosened edge of the mat, and allow it to drop into the beaker. Follow with 200 ml. of boiling 1.25% NaOH, washing the filter screen free of any adhering particles with this solution. Digest as before for exactly 30 min.
6. Remove at the end of the digestion period and filter as shown in paragraph 4. Without breaking the suction add 25 ml. of the boiling dilute H_2SO_4 (1.25%) for the initial wash. Complete the washing operation with boiling water as shown in section C, paragraph 5.
7. Transfer the digest to an alundum or Gooch crucible by means of a water-wash bottle. Wash free of water by filling the crucible twice with 95% alcohol.
8. Follow C-8 Method I.
9. Follow C-9 Method I.

D. Calculations

Follow Method I.

Method III. Buchner Funnel

A. Reagents

1. through 6. as listed for all methods.

B. Apparatus

1. through 8. as listed for all methods.
9. No. 40 filtering cloth made by the National Filter Cloth and Weaving Company, 1717 Dixwell Avenue, Hamden, Conn. Cut cloth in oval about 6 x 7 in. Stitching will prevent ravelling.
10. Buchner Funnel, 4-in., preferably polyethylene adapted to fit filtering device indicated in B-7.

C. Procedure

1. Follow C-1 Method I.
2. Follow C-2 Method II.
3. At the expiration of 30 min., remove the flask and filter through cloth which has been fitted into the Buchner funnel so that the filtration is speeded by suction. Wash with boiling water until washings are neutral.
4. Wash the charge and asbestos back into the flask with 200 ml. of boiling NaOH solution (1.25%).

5. Attach the flask to the condenser and boil for exactly 30 min.
6. At the expiration of 30 min. remove flask and filter immediately through a Gooch crucible prepared with an asbestos mat or through an alundum crucible.
7. Wash the crucible and contents with 25 ml. of boiling dilute H_2SO_4 (1.25%). Follow with three 50-ml. washings of boiling water. Wash the crucible and contents free of water by filling twice with 95% alcohol.
8. Follow C-8 Method I.
9. Follow C-9 Method I.

D. Calculations

1. Follow Method I.

Method IV. Purdue Method (using Shimer filter assembly)

A. Reagents

1. through 6. as listed for all methods.
7. Filter aid, homogenized glass wool. Add 10 g. of Pyrex Brand Glass Wool (Corning Glass Works) to 500 ml. of water in Waring Blendor. Homogenize until a smooth blend is obtained (2 to 4 min.). Remove water, and dry the filter aid at 130°.

B. Apparatus

1. through 8. as listed for all methods.
9. Filtering device, either individual filter flasks or manifold with suitable connection to permit use of Shimer filter.
10. Shimer filter with porcelain disc (Figure 2), A. H. Thomas, No. 5314-S.

NOTE: Prepare an asbestos mat 3–6 mm. thick on the porcelain disc by pouring a water suspension of the asbestos onto the filter and then applying suction.

C. Procedure

1. Follow C-1 Method I.
2. Add 1.0–1.5 g. of the glass wool, 200 ml. of boiling 1.25% H_2SO_4, and 1–2 drops Dow Corning AF emulsion. Place the beaker and contents on the extraction apparatus with the hot plate at the temperature specified in B-1.
3. Continue the boiling for exactly 30 min., rotating periodically to keep any solid matter from adhering to the sides of the beaker.
4. Remove after 30-min. period and pour beaker contents into funnel above Shimer filter (with previously prepared asbestos mat: see B-10) with suction applied. Rinse beaker thoroughly with hot water. After liquid has passed through filter, wash mat and digest thoroughly with hot water.
5. Add 200 ml. of preheated 1.25% NaOH to each beaker, and heat to boiling on digestion apparatus.
6. Disassemble filtering apparatus, and push a rod through the bottom of the Shimer filter to force the asbestos mat and sample beyond the rim of the filter, partially exposing the porcelain disc. Remove the asbestos mat and sample from the disc and transfer to the boiling NaOH.

NOTE: Addition of a boiling stone to the alkali will prevent bumping.

7. At the end of the digestion period pour the beaker contents into the funnel above the Shimer filter (with asbestos mat previously prepared: see B-10). Rinse with 25 ml. of hot acid (1.25% H_2SO_4). Complete washing with hot water. Remove water with two 25-ml. rinsings of 95% alcohol.
8. Transfer the asbestos mat and sample to a crucible in the manner described in D6. Dry crucible and digest in an air oven at 130°C. for 2 hrs.

NOTE: A regular Coors crucible may be used instead of a Gooch or alundum since no filtering is done in this step of the operation.

9. Follow No. 9 Method I.

E. Calculations

1. Follow Method I.

Discussion and Results

The results obtained from each collaborator are tabulated in Tables I through IV. Also shown in these tables are the standard deviations obtained on each sample by each method.

Table V gives a summary of the statistical calculations. The statistical design gives us the answers for laboratories, operators, and days. The variance due to days (which includes the analytical error) is com-

TABLE I
Crude Fiber in Cottonseed Meal Sample

Collaborator	No. 1 modified official		No. 2 Oklahoma State filter screen		No. 3 Buchner funnel		No. 4 Purdue University Shimer filter	
1	7.7	7.9	7.6	7.6	7.6	7.9	7.9	8.3
	8.2	8.2	7.6	7.7	7.7	8.1	8.7	8.6
2	8.4	8.4	8.5	8.6	8.2	8.9	10.9[a]	11.7[a]
	8.4	8.3	8.7	9.1	9.3	9.2	10.4[a]	10.3[a]
3	8.5	8.3	8.8	8.8	8.3	8.3	8.3	7.9
	8.2	8.8	8.5	8.8	8.2	8.4	8.3	8.1
4	6.9	7.0	8.3	8.3	7.4	7.5	8.4	8.3
	6.9	6.8	8.6	8.3	7.3	7.2	8.5	8.6
5	8.0	8.1	8.0	8.3	8.4	8.2	7.9	7.9
	8.2	8.0	7.5	8.2	8.6	7.6	8.0	8.2
6	7.7	8.4	7.8	7.5	8.6	8.8	8.2	8.7
	8.4	8.5	7.6	8.2	8.4	8.3	8.7	8.7
7	7.7	7.5	8.2	8.3	8.6	8.7	7.9	8.2
	7.9	7.9	8.3	8.2	8.1	8.3	7.9	8.1
8	8.6	7.8	8.2	8.5	8.0	8.1	9.2	9.3
	7.8	8.2	7.5	8.7	7.9	7.5	9.7	9.5
9	8.8	8.6	8.8	8.6	8.5	8.5	....	....
	8.5	8.7	8.7	8.6	8.6	8.8	....	....
10	9.5	7.6	8.4	8.4	8.1	9.3	8.1	8.0
	9.0	8.3	8.3	8.3	8.0	8.8	8.4	7.9
11	8.2	8.3	8.6[b]	8.5[b]	8.8	8.7	8.5	8.4
	8.5	8.3	8.9	8.8	8.6	9.2	8.1	9.2
12	8.04	9.36	8.42	8.57	8.63	8.84	8.84	8.32
	8.08	8.50	8.53	8.45	8.90	9.30	8.96	8.54
Average	8.16		8.32		8.35		8.43	
Std. dev.								
Within-labs	0.37		0.24		0.33		0.26	
Between-labs	0.57		0.42		0.55		0.48	

[a] Not used in calculations.
[b] Violent bumping during digestion.

TABLE II
Crude Fiber in 50% Soybean Oil Meal Sample

Collaborator	No. 1 modified official		No. 2 Oklahoma State filter screen		No. 3 Buchner funnel		No. 4 Purdue University Shimer filter	
1	2.5	2.5	2.7	2.7	2.4	2.3	2.5	2.2
	2.6	2.4	2.3	2.5	2.2	2.4	2.6	2.8
2	2.6	2.8	2.8	2.8	3.0	3.3	3.2	3.2
	2.5	2.7	3.2	2.8	3.1	3.0	3.4	3.2
3	2.9	2.8	2.9	2.8	2.9	2.8	2.7	2.8
	2.7	3.0	2.7	2.8	2.8	2.9	2.6	2.7
4	2.3	2.3	2.6	2.6	2.6	2.6	2.8	2.8
	2.2	2.1	2.5	2.6	2.5	2.6	2.8	2.8
5	2.8	2.8	2.4	2.6	2.0	2.5	2.3	2.5
	2.3	2.7	2.6	2.4	2.6	2.5	2.2	2.6
6	2.9	3.0	2.6	2.9	2.7	2.8	2.8	2.8
	2.8	3.1	2.8	3.2	2.9	3.2	2.7	3.2
7	2.6	2.3	2.4	2.3	3.2	3.2	2.7	2.7
	2.5	2.4	2.6	2.4	2.5	2.7	2.6	2.6
8	3.6	2.6	2.5	2.6	2.8	2.6	2.9	2.9
	2.7	2.6	2.5	2.7	2.7	2.8	2.7	3.2
9	2.6	2.7	2.7	2.8	2.8	2.8	....	....
	2.8	2.9	2.8	2.8	2.8	2.8	....	....
10	3.0	2.5	2.3	2.6	2.5	2.6	2.6	2.5
	2.5	2.6	3.1	2.9	3.1	2.7	2.7	2.6
11	2.5	2.6	3.0	2.8	2.8	2.7	3.0	3.2
	2.9	2.8	2.8	2.7	2.6	2.6	....[a]	....[a]
12	2.57	2.57	2.55	2.15	2.65	2.65	2.64	2.70
	2.53	2.50	2.63	2.72	2.63	2.80	2.57	2.78
Average	2.65		2.68		2.72		2.74	
Std. dev.								
Within-labs	0.20		0.18		0.18		0.15	
Between-labs	0.27		0.21		0.25		0.27	

[a] Analyst unable to filter.

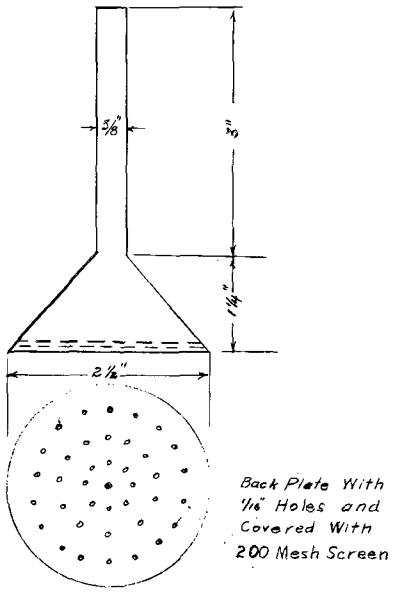

FIG. 1. Filter screen for Oklahoma State method.

bined with the variance due to operators to give the within-laboratory variance. The variance due to laboratories is added to the within-laboratory variance to give the between-laboratory variance. These are reported as standard deviations (standard deviation is the square root of the variance). In Table VI the within- and between-laboratories variances for the four meals are combined to give the final within- and between-laboratories variance for each method. The agreement within- and between-laboratories is calculated from these values in accordance with the statistical method recently adopted by the A.O.C.S. The

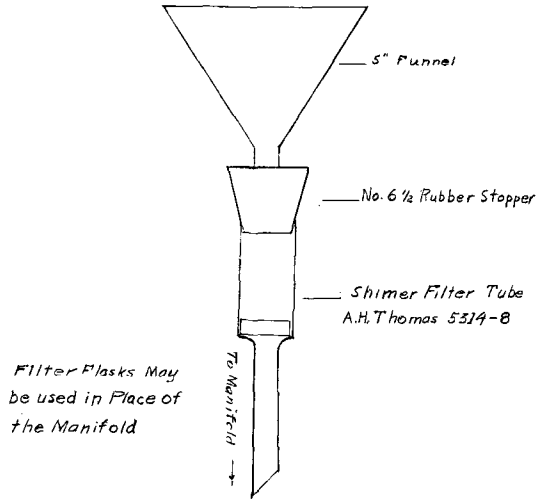

FIG. 2. Shimer filter assembly for Purdue method.

high fiber in the alfalfa meal distorts the variances; therefore calculations are also shown, excluding the alfalfa meal results.

To compare the accuracy of the method the averages of the results of the various methods are checked for differences by means of the "t" test. Some samples showed significant differences between the methods. However on the combined data there are no significant differences between the methods; the averages are statistically equivalent. Variances of the results of the various methods are checked for differences by means of the "F" test. The methods do show a difference in variability, as checked out by the "F" test. The Buchner funnel method showed the least variability, followed in order by the Purdue

TABLE III

Crude Fiber in Dairy Feed Sample

Collaborator	No. 1 modified official		No. 2 Oklahoma State filter screen		No. 3 Buchner funnel		No. 4 Purdue University Shimer filter	
1	8.7	8.7	8.8	8.4	8.8	8.9	8.6	8.9
	8.9	9.4	8.6	9.4	8.6	9.0	9.1	8.9
2	9.2	9.4	9.6	9.7	9.8	9.7	10.5	10.9
	9.0	9.4	9.6	9.6	8.8	9.5	9.4	9.9
3	9.3	9.2	9.5	9.4	9.2	9.3	8.9	9.0
	9.1	9.5	9.3	9.6	9.2	9.2	8.8	8.9
4	8.2	8.3	9.4	9.3	8.6	8.7	9.2	9.2
	8.1	8.0	8.9	9.0	8.7	8.8	9.4	9.3
5	8.8	8.6	8.3	8.4	9.1	8.7	8.9	9.0
	9.0	8.7	8.8	8.6	8.9	8.4	9.2	8.6
6	9.3	8.7	9.2	9.2	8.8	9.5	9.4	9.6
	9.1	9.7	9.5	9.3	8.9	9.2	9.4	9.4
7	8.5	8.8	9.2	9.2	9.1	9.1	8.6	8.7
	8.5	8.6	9.1	9.0	8.8	8.9	9.0	8.9
8	10.3	9.3	9.1	9.8	9.0	9.4	9.5	9.8
	8.9	9.2	8.5	9.4	8.5	8.9	10.0	9.5
9	9.8	9.4	9.2	9.3	9.1	8.9	....	....
	9.4	9.4	9.2	9.0	9.2	9.0	....	....
10	9.9	10.1	9.0	9.0	8.9	8.8	10.6	9.0
	9.0	9.0	9.3	8.9	8.6	9.0	9.0	8.9
11	8.7	8.9	9.4[a]	9.4[a]	9.0	9.1	9.0	9.0
	8.9	8.9	9.3	9.1	9.0	8.8	9.5	9.5
12	8.71	9.08	8.88	8.81	8.89	9.09	9.06	9.30
	8.76	8.71	9.09	8.78	9.07	9.25	8.89	9.05
Average	9.02		9.13		9.00		9.26	
Std. dev.								
Within-labs	0.35		0.25		0.24		0.39	
Between-labs	0.49		0.36		0.29		0.52	

[a] Violent bumping during digestion.

TABLE IV

Crude Fiber in Alfalfa Meal Sample

Collaborator	No. 1 modified official		No. 2 Oklahoma State filter screen		No. 3 Buchner funnel		No. 4 Purdue University Shimer filter	
1	22.6	23.0	22.4	22.4	23.1	22.7	22.7	23.5
	23.7	23.2	23.3	23.0	23.4	24.0	23.8	24.2
2	24.0	24.6	24.9	24.4	24.1	24.6	25.2[b]	25.2[b]
	25.2	24.7	25.2	24.9	23.0	24.0	23.9[b]	25.3[b]
3	24.5	24.3	25.1	24.6	24.4	24.3	23.9	23.7
	24.4	25.0	25.7	25.4	24.3	24.2	23.3	23.3
4	22.3	22.4	24.1	24.0	24.0	24.0	24.1	23.8
	22.4	22.4	23.9	24.1	22.8	22.9	23.9	24.1
5	24.6	24.2	23.9	24.2	23.7	23.7	23.5	23.4
	23.6	23.7	22.5	22.7	23.7	23.2	23.6	23.8
6	23.4	23.6	23.1	23.7	23.8	23.6	24.5	24.5
	24.0	24.6	22.6	23.7	23.8	23.7	23.9	24.2
7	23.7	23.0	24.1	24.1	23.4	24.5	23.1	23.2
	23.1	23.1	24.0	23.6	24.2	24.0	23.8	23.4
8	25.6	23.5	23.5	24.1	23.4	24.0	23.9	24.8
	23.2	24.0	23.1	24.7	23.1	23.1	24.2	24.0
9	25.1	24.6	24.8	24.7	24.1	24.6	....	....
	24.3	24.7	24.4	24.5	24.5	24.6	....	....
10	24.6	23.1	23.1	23.5	23.4	23.3	23.3	23.8
	24.0	24.7	23.9	24.2	22.9	24.2	23.4	23.4
11	24.1	23.9	24.9[a]	24.4[a]	25.1	24.6	24.0	23.5
	24.3	24.2	24.1	24.3	24.7	24.8	24.4	25.1
12	27.5	24.5	24.4	24.5	24.9	32.6	24.5	25.2
	24.1	29.3	24.2	26.2	24.0	24.9	23.8	24.7
Average	23.93		24.00		23.85		23.77	
Std. dev.								
Within-labs	0.58		0.52		0.43		0.42	
Between-labs	0.80		0.84		0.62		0.51	

[a] Violent bumping during digestion.
[b] Not used in calculations.

TABLE V

Summary of Statistical Calculations

	Methods			
	No. 1 modified official	No. 2 Oklahoma State filter screen	No. 3 Buchner funnel	No. 4 Purdue University Shimer filter
Average results				
Cottonseed meal	8.16	8.32	8.35	8.43
Soybean oil meal	2.65	2.68	2.72	2.74
Dairy feed	9.02	9.13	9.00	9.26
Alfalfa meal	23.93	24.00	23.85	23.77
Standard deviation within-labs.				
Cottonseed meal	0.37	0.24	0.33	0.26
Soybean oil meal	0.20	0.18	0.18	0.15
Dairy feed	0.35	0.25	0.24	0.39
Alfalfa meal	0.58	0.52	0.43	0.42
All samples	0.40	0.33	0.31	0.32
Excluding alfalfa meal	0.31	0.22	0.26	0.29
Standard deviation between-labs.				
Cottonseed meal	0.57	0.42	0.55	0.48
Soybean oil meal	0.27	0.21	0.25	0.27
Dairy feed	0.49	0.36	0.29	0.52
Alfalfa meal	0.80	0.84	0.62	0.51
All samples	0.57	0.51	0.46	0.46
Excluding alfalfa meal	0.46	0.34	0.39	0.44

method, the Oklahoma State Filter Screen method, and the Modified Official method. However these differences, while statistically significant in some cases, are actually quite small and do not provide a clear-cut basis for deciding which method is best.

Conclusions

The results obtained in this collaborative study show that none of the methods tested offer sufficient advantages in precision and accuracy to warrant selection as an official method to the exclusion of other methods studied.

Also the precision of all methods tested show these methods to be inadequate for checking crude fiber specification limits set up by the N.S.P.A. on soybean oil meal. This statement would also apply to any other product where specification limits on crude fiber are narrower than the precision shown in Table VI.

Ease and speed for manipulation are also an important criterion for choosing an official method. A poll of committee members showed no definite preference for any one method studied; every method received the support of two or more of the committee members.

TABLE VI

A.O.C.S. Precision Calculations

Agreement within labs.
(95% confidence limits)
(Two single determinations in one lab. should not differ by more than)

Method	No. 1	No. 2	No. 3	No. 4
All samples	1.11	0.91	0.86	0.89
Excluding alfalfa	0.86	0.62	0.71	0.79

Agreement between labs.
(95% confidence limits)
(Single determinations in two labs. should not differ by more than)

Method	No. 1	No. 2	No. 3	No. 4
All samples	1.58	1.41	1.27	1.27
Excluding alfalfa	1.28	0.95	1.08	1.26

The Liaison Committee is still hopeful that a method can be devised that will give a precision in line with trade specifications and practices. The committee will continue to work toward the development of such a method.

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[Received August 25, 1959]

Quantitative Fatty Acid Analysis of Vegetable Oils by Gas-Liquid Chromatography[1,2]

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THE DEVELOPMENT of gas-liquid chromatography (G.L.P.C.) by James and Martin (6, 7) and subsequent applications of other workers (3, 4, 5, 8, 9, 16, 18) made possible the rapid separation of micro quantities of fatty acid esters. The separations were based mainly on chain length, using liquid phases such as silicone and Apiezon greases. The technique was utilized successfully in this laboratory for the quantitative determination of C_{16}, C_{18}, C_{20}, C_{22}, and C_{24} fatty acid esters in rapeseed oil (2). The quantitative aspects were checked by using known mixtures of pure fatty acid esters which showed that the G.L.P.C. results were accurate to within one unit percentage.

The successful separation of saturated and unsaturated fatty acid esters by Orr and Callen (14) with polyester liquid phases and the further developments by other workers (1, 10, 11, 12, 13, 17) led to the complete analysis of fats and oils on a micro scale. Investigations in this laboratory on fatty acid compositions of vegetable oils, animal fats, and partially hydrogenated oils showed good agreement between measured iodine values and those calculated from G.L.P.C. data and led to the work presented herein. The fatty acid composition of six commercial vegetable oils and two

[1] Issued as N.R.C. No. 5373.
[2] Presented at 32nd annual fall meeting, American Oil Chemists' Society, October 20–22, 1958, Chicago, Ill.
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