TABLE 4.3
Evaluation of the Ruggedness of the Filter Bag Technique Using Youden's Ruggedness Test with Seven Variables at Two Levels[a]

No.	Variables	Level 1 lc	Level 2 Cap	Overall comparison lc	Cap	diff
1	Sample size (g)	0.8–0.9	1.2–1.3	22.24	22.33	0.09
2	Predry time (h)	2.5	3	22.30	22.27	–0.03
3	Predry temperature (°C)	98	102	22.26	22.31	0.05
4	Extraction time (min)	25	35	22.32	22.25	–0.07
5	Extraction temperature (°C)	89	94	22.32	22.26	–0.06
6	Postdry time (min)	25	35	22.28	22.30	0.02
7	Predry time (°C)	98	102	22.32	22.26	–0.06
	Significant at the 5% level diff =					0.31

No.		Chicken thighs, cooked lc	Cap	diff	Hot dogs lc	Cap	diff	Corn lc	Cap	diff
1	Sample size	34.01	34.03	0.02	45.61	45.71	0.10	14.31	14.20	–0.11
2	Predry time	33.99	34.04	0.05	45.69	45.63	–0.06	14.20	14.32	0.12
3	Predry temperature	34.05	33.99	–0.05	45.67	45.64	–0.03	14.30	14.21	–0.09
4	Extraction time	34.04	34.00	–0.04	45.60	45.71	0.11	14.31	14.20	–0.11
5	Extraction temperature	34.05	33.99	–0.06	45.64	45.67	0.03	14.25	14.26	0.01
6	Postdry time	4.02	34.02	0.00	45.66	45.65	–0.01	14.31	14.20	–0.11
7	Predry time	33.96	34.08	0.13	45.63	45.69	0.06	14.23	14.29	0.06
	Significant at the 5% level diff =			0.16			0.42			0.27

No.		Soybeans[b] lc	Cap	diff	Potato chips lc	Cap	diff	Cattle feed lc	Cap	diff
1	Sample size	22.04	21.31	–0.73	36.44	36.45	0.01	3.31	3.24	–0.07
2	Predry time	21.58	21.77	0.18	36.46	36.43	–0.02	3.29	3.25	–0.04
3	Predry temperature	21.62	21.72	0.10	36.45	36.44	–0.01	3.31	3.23	–0.08
4	Extraction time	21.47	21.88	0.41	36.43	36.46	0.04	3.26	3.29	0.03
5	Extraction temperature	21.49	21.86	0.38	36.42	36.47	0.05	3.24	3.30	0.06
6	Postdry time	21.68	21.67	0.00	36.46	36.43	–0.04	3.23	3.32	0.09
7	Predry time	21.75	21.60	–0.15	36.43	36.46	0.04	3.25	3.30	0.05
	Significant at the 5% level diff =			0.28			0.42			0.27

No.		Poultry feed lc	Cap	diff	Dog food lc	Cap	diff	Ground beef lc	Cap	diff
1	Sample size	4.34	4.40	0.06	3.96	4.00	0.03	36.99	36.85	–0.14
2	Predry time	4.40	4.34	–0.06	3.94	4.02	0.08	36.92	36.93	0.01
3	Predry temperature	4.36	4.38	0.02	4.00	3.96	–0.04	37.06	36.79	–0.27
4	Extraction time	4.34	4.39	0.05	3.94	4.02	0.07	36.88	36.97	0.09
5	Extraction temperature	4.38	4.35	–0.02	3.96	4.00	0.05	36.92	36.93	0.01
6	Postdry time	4.42	4.31	–0.12	3.96	4.00	0.04	36.94	36.91	–0.03
7	Predry time	4.30	4.43	0.12	3.92	4.04	0.11	36.85	36.99	0.14
	Significant at the 5% level diff =			0.32			0.26			0.16

[a]lc, low level; Cap, high level effect on % fat.
[b]Soybeans were not preheated before grinding.

TABLE 4.4
Evaluation of the Precision and Relative Accuracy of the Filter Bag Technique Compared with the Conventional Method[a]

Sample	Conventional % Fat/oil	Conventional SD	FBT % Fat/oil	FBT SD
Rice hulls	0.3	0.07	0.2	0.08
Soybean meal	1.4	0.01	1.7	0.05
Pig starter	1.8	0.05	1.9	0.11
Chick grower	2.2	0.10	2.2	0.10
Cattle feed	2.7	0.10	2.8	0.08
Corn	3.0	0.07	3.5	0.12
Chicken breast	3.2	0.07	3.1	0.05
Blueberry muffin	4.6	0.41	4.7	0.39
Oatmeal	5.9	0.08	5.7	0.21
Brownie mix	8.8	0.07	8.5	0.15
Turkey	8.9	0.11	8.7	0.07
Fish meal	9.9	0.07	9.8	0.16
Ham	10.6	0.03	10.9	0.11
Soybeans	21.3	0.08	21.0	0.44
Horse feed	22.1	0.18	22.2	0.05
Tortilla chips	24.2	0.22	24.2	0.26
Ground beef	28.4	0.16	28.6	0.23
Chicken thighs	29.1	0.09	29.2	0.13
Sausage	36.4	0.35	36.7	0.62
Safflower	40.4	0.22	39.5	0.20
Canola	41.4	0.07	41.7	0.12
Cheese Curls	43.3	0.06	43.2	0.29
Average	15.9	0.12	15.9	0.18

[a]n = 5.

Multilaboratory FBT Study. A summary of the data from the multilaboratory study is presented in Table 4.5. This study was a good test of the repeatability of the FBT because of the more extensive replication. The analysis of five samples was replicated four times in 13 laboratories. An average pooled SD of $S_r = 0.28$ was found, indicating good repeatability within laboratories among the five samples. The reproducibility among laboratories averaged $S_R = 0.60$. The study demonstrates that the FBT can be replicated among laboratories with good precision. The FBT values were in good agreement with the conventional method with all five sample types. The results also indicate that the Youden's Ruggedness Test was a good predictor of the laboratory reproducibility.

FBT Collaborative Study. A summary of the collaborative FBT results for the 28 samples as 56 blind duplicates is presented in Table 4.6. The results indicated that there was excellent agreement among laboratories analyzing the samples with the FBT. The reproducibility between laboratories was $S_R = 0.43$ and the repeatability

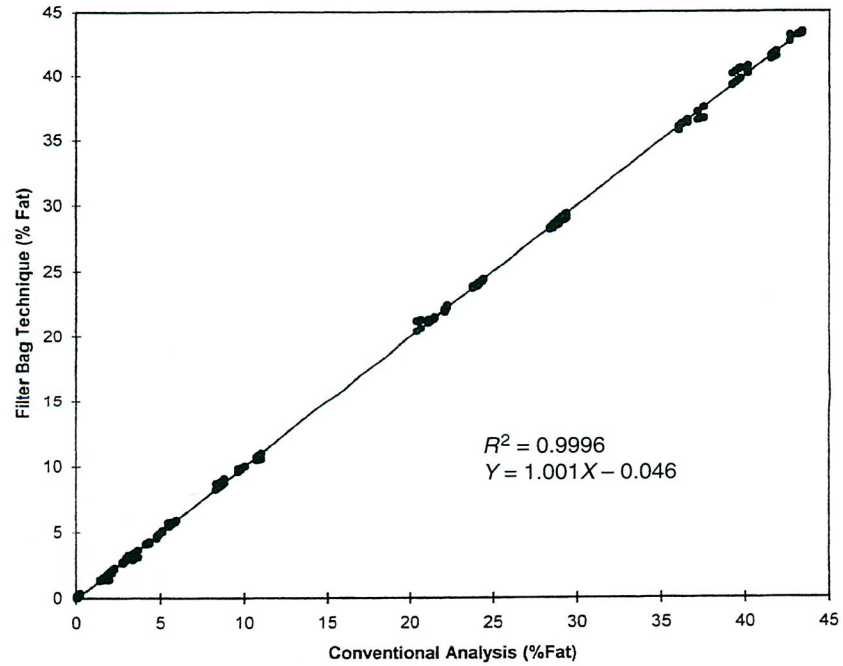

Fig. 4.11. Regression analysis of the accuracy of the FBT analysis of fat/oil relative to the conventional method in 22 samples each replicated five times.

within the laboratories was $S_r = 0.31$ (Table 4.7). The mean values for the 28 samples from the collaborating laboratories were not significantly different ($P < 0.001$) in a regression analysis from the average of the AOCS Certified laboratories using AOCS Ba 3–38 and AOAC 920.39. The Regression Plot (Fig. 4.12) illustrates the excellent correlation ($R^2 = 0.9990$) between the AOCS Certified laboratories and the collaborating FBT laboratories. The regression line passes through the origin, indicating that there is no bias between methods. The results confirm that the FBT method was capable of generating accurate data, relative to the Official AOCS method, in a variety of independent laboratories around the world.

The AOCS Method Am 2–93 (equivalent to the FOSFA method) yielded values that were higher than the FBT values for soybeans (1%), safflower (2%), and canola (6%). Am 2–93 generally yields higher values for oilseeds compared with other AOCS official methods due to the multiple grindings and extractions that are part of the method. Although the high temperatures of the FBT greatly accelerate the extraction kinetics, further experimentation is required to determine whether extended periods of time will remove matrix-bound oil without further grinding.

TABLE 4.5
Comparison of the Conventional Method with the Filter Bag Technique Performed in 13 Collaborating Laboratories on 5 Samples

	Ground beef		Cheese curls		Soybeans		Whole corn		Horse feed	
Filter bag method	Average	SD	Average	SD	Average	SD	Average	SD	Average	SD
Laboratory #										
1	22.7	0.19	41.4	0.25	20.7	0.12	3.2	0.11	21.7	0.20
2	22.4	0.20	41.4	0.18	21.0	0.19	3.3	0.16	21.7	0.16
3	21.4	0.60	40.3	0.55	20.8	0.56	3.2	0.03	21.8	0.08
4	23.0	0.16	41.7	0.17	21.3	0.33	3.7	0.27	21.9	0.13
5	22.1	0.25	41.0	0.44	19.6	0.43	3.0	0.69	21.1	0.36
6	21.8	0.25	39.4	0.54	20.8	0.15	3.1	0.11	21.3	0.10
7	22.8	0.24	41.6	0.46	21.1	0.13	3.3	0.10	22.1	0.08
8	22.4	0.45	41.6	0.25	21.0	0.46	3.1	0.38	21.7	0.12
9	22.7	0.25	41.5	0.09	22.0	0.07	3.7	0.15	22.3	0.16
10	22.8	0.04	42.0	0.17	20.5	0.24	3.1	0.19	21.4	0.32
11	22.5	0.23	41.1	0.20	20.0	0.36	3.3	0.15	22.1	0.25
12	21.9	0.53	40.4	0.27	20.9	0.35	2.3	0.18	21.8	0.17
13	22.7	0.16	41.9	0.18	21.8	0.17	4.2	0.22	22.3	0.16
S_r		0.31		0.32		0.31		0.27		0.20
S_R		0.56		0.80		0.72		0.50		0.41
Average	22.4		41.2		20.9		3.3		21.8	
Conventional method average	23.0	0.28	40.5	1.00	20.7	0.08	3.4	0.07	22.2	0.47

[a]n = 5. r, reproducibility value; R, reproducibility value; S_r, repeatability of the SD; S_R, reproducibility of the SD.

TABLE 4.6
Results of the International Collaborative Study of the Reproducibility of the Filter Bag Technique with Twenty-Eight Samples Analyzed as Blind Duplicates

		A	B	C	D	E	F	G	H	I	J	K	L	M	N
Lab. #	Rep.	(% Fat/oil)													
1	1	5.4	8.6	21.1	38.4	1.6	3.0	3.2	3.0	5.8	2.2	6.4	22.6	1.9	19.5
	2	5.7	8.7	20.9	38.4	1.7	3.1	3.3	3.1	5.7	2.1	6.2	22.8	1.7	19.6
2	1	5.9	8.8	21.4	40.2	2.0	3.6	3.7	3.5	6.0	3.5	7.0	22.8	2.3	20.4
	2	7.2	9.2	21.9	39.9	2.1	4.5	4.6	3.5	6.4	2.7	6.5	23.2	2.9	19.5
3	1	5.7	8.5	20.6	39.3	1.4	3.5	3.3	3.2	5.8	2.3	6.2	21.9	2.2	20.3
	2	5.9	8.6	20.8	39.3	1.5	3.8	3.1	3.3	5.2	2.0	6.5	21.9	1.9	19.9
4	1	5.6	9.0	21.0	39.2	1.4	3.0	3.5	3.3	5.9	2.4	6.0	22.6	1.9	19.7
	2	5.8	8.6	20.9	38.7	1.6	3.3	3.2	3.0	5.6	1.9	6.2	22.7	1.8	20.1
5	1	6.3	8.9	21.0	38.9	1.9	3.6	3.4	3.3	5.4	2.4	6.0	22.7	2.6	18.5
	2	6.2	8.9	20.1	38.3	1.5	3.6	3.3	3.1	5.6	2.7	5.8	22.5	2.1	19.4
6	1	4.8	8.3	20.2	39.0	1.2	2.9	2.9	2.8	5.2	1.9	6.1	22.4	2.2	19.8
	2	5.7	8.5	20.1	39.1	1.2	3.0	3.3	3.2	5.4	1.8	6.1	22.7	2.2	19.4
7	1	5.2	8.7	21.0	39.9	1.7	3.1	3.0	3.2	5.6	2.0	6.2	22.9	2.3	20.2
	2	5.3	8.5	20.7	39.7	1.5	3.2	3.1	3.2	5.6	2.2	6.2	22.7	2.3	19.9
8	1	5.1	9.0	20.3	38.5	1.5	3.1	2.5	3.1	3.5	1.5	5.7	23.3	2.9	20.5
	2	5.1	8.6	14.2	25.6	1.8	3.2	2.7	3.6	5.4	2.9	6.6	22.7	2.9	19.7
9	1	6.2	9.2	21.7	42.8	1.7	3.1	3.5	3.2	5.9	2.9	6.3	22.6	2.2	20.6
	2	5.5	9.0	21.3	42.9	1.8	3.2	3.5	3.3	5.7	3.1	6.3	22.9	2.2	20.1
10	1	5.6	8.6	20.6	38.5	1.3	2.9	3.8	6.0	5.6	2.2	6.1	22.6	2.1	20.2
	2	5.4	9.0	20.7	37.9	1.6	3.1	3.3	3.4	5.7	2.3	6.4	22.6	2.1	20.1
11	1	6.4	8.9	21.4	41.1	1.8	3.3	3.2	3.0	5.5	3.0	6.6	22.6	2.5	19.8
	2	6.4	9.3	22.2	40.5	1.7	3.1	3.1	3.1	5.5	2.8	6.3	22.9	2.5	20.7
12	1	6.2	8.3	20.5	38.3	1.9	3.2	8.3	4.6	5.2	2.1	6.3	22.7	2.7	20.3
	2	6.1	8.2	19.6	38.3	2.1	4.3	3.7	3.0	5.5	4.8	6.9	22.6	1.8	20.0

(Continued)

TABLE 4.6
(*Cont.*)

	Rep.	a	b	c	d	e	f	g	h	i	j	k	l	m	n
1	1	6.5	23.6	2.9	11.0	23.7	2.9	19.2	22.9	31.6	40.3	25.3	2.9	31.0	2.4
	2	6.6	23.8	3.0	10.2	24.1	2.4	18.9	21.9	32.1	39.5	25.6	3.2	31.7	2.0
2	1	7.5	24.0	3.6	12.2	24.0	3.2	20.3	22.6	31.4	40.1	26.2	3.6	30.9	2.8
	2	6.4	23.5	3.3	11.3	23.9	2.9	19.9	22.5	31.5	40.8	25.5	4.0	31.0	3.0
3	1	6.8	24.1	3.5	11.4	24.4	3.2	19.4	20.9	31.9	39.3	26.8	4.0	31.3	2.5
	2	6.8	23.8	3.1	11.8	23.7	3.0	20.0	21.8	32.2	38.5	25.9	3.0	31.2	2.4
4	1	6.5	23.6	3.1	11.4	23.9	2.1	19.0	21.3	32.2	39.4	25.6	3.5	30.6	1.8
	2	6.7	24.0	2.9	11.4	23.8	2.7	18.8	21.8	31.0	39.1	25.6	3.4	30.2	2.2
5	1	6.7	23.2	3.1	10.9	23.1	2.5	18.5	22.3	31.9	38.5	25.3	3.1	29.4	2.0
	2	6.6	23.3	2.8	10.8	23.5	3.0	19.2	21.8	31.4	38.7	24.5	3.4	29.2	2.4
6	1	6.9	23.5	3.2	11.6	23.6	2.6	19.1	22.9	30.6	39.1	25.3	2.8	29.6	1.8
	2	6.2	23.3	2.9	11.3	23.6	2.6	18.5	23.2	28.0	38.7	25.0	3.3	30.3	1.6
7	1	6.8	23.8	3.3	11.5	23.5	3.1	19.6	16.5	31.7	39.3	26.4	3.6	30.7	2.2
	2	6.8	23.8	3.2	11.5	23.4	3.0	19.5	10.6	32.1	39.7	26.2	3.6	30.9	2.3
8	1	7.2	24.1	2.3	11.6	23.9	3.3	20.0	18.0	31.9	39.6	25.8	3.3	30.1	2.3
	2	6.8	23.5	2.8	1.4	24.1	2.3	3.5	22.6	33.4	39.3	25.6	2.8	29.8	3.0
9	1	6.8	24.0	3.3	11.2	23.9	2.9	20.2	22.7	32.0	40.1	25.7	3.8	30.9	3.1
	2	6.9	23.9	3.4	11.7	23.4	2.8	20.6	24.1	32.6	40.0	25.7	4.1	30.0	3.3
10	1	6.9	24.1	3.2	11.6	23.9	3.0	19.3	23.4	32.8	39.8	25.7	3.7	31.3	1.9
	2	6.1	23.9	3.2	11.8	24.2	2.5	18.8	23.1	32.3	39.9	26.3	3.0	30.8	1.6
11	1	6.9	24.0	3.4	11.8	24.4	3.1	20.2	23.5	32.2	39.9	26.3	3.9	30.9	2.6
	2	6.9	23.7	3.1	12.2	24.4	2.9	19.6	22.4	32.3	39.4	26.0	3.3	30.8	2.6
12	1	6.7	23.9	4.0	11.9	20.2	4.6	19.6	22.4	32.0	39.9	29.4	3.0	31.4	1.8
	2	7.3	23.7	3.6	11.5	24.0	3.0	18.5	1.7	31.8	39.6	26.3	3.9	29.7	1.5

[a]Sample ID: A, oatmeal; B, brownie mix; C, soybean A; D, canola; E, soybean meal; F, corn A; G, poultry starter; H, cattle feed; I, pig starter; J, alfalfa; K, cat food; L, cookies; M, breakfast cereal; N, tortilla chips; a, dog food; b, crackers; c, turkey; d, ham; e, beef ground; f, chicken breast; g, soybean B; h, safflower; i, potato chips; j, hot dog; k, sausage; l, corn B; m, cheese curls; n, corn silage.

TABLE 4.7
A Summary of the Statistical Analysis of the International Collaborative Study of the Filter Bag Technique for the Proposed Official AOCS Method, "Rapid Determination of Oil/Fat Utilizing High Temperature Solvent Extraction"[a]

Sample ID[b]	A	B	C	D	E	F	G	H	I	J	K	L	M	N
Number of laboratories	12	12	11	9	12	12	11	10	11	11	12	11	12	12
Number of replicates	24	24	22	18	24	24	22	20	22	22	24	22	24	24
oil/fat %														
Collaborative average	5.8	8.7	20.9	39.0	1.6	3.3	3.3	3.2	5.6	2.4	6.3	22.7	2.3	19.9
Certified laboratories average[c]	5.7	8.7	21.1	39.7	1.6	3.6	3.5	3.0	5.5	2.2	6.2	23.1	2.3	20.0
AOCS Am 2-93 average for oilseeds			21.9	44.8		3.6								
Repeatability														
S_r[d]	0.36	0.20	0.35	0.23	0.14	0.31	0.24	0.18	0.20	0.39	0.27	0.20	0.26	0.39
RSD_r	6.2	2.3	1.7	0.6	8.5	9.5	7.3	5.6	3.6	16.1	4.2	0.9	11.4	2.0
r	0.99	0.56	0.98	0.65	0.39	0.88	0.68	0.51	0.56	1.08	0.75	0.56	0.72	1.09
Reproducibility														
S_R	0.54	0.31	0.63	0.68	0.27	0.42	0.42	0.20	0.28	0.50	0.30	0.20	0.36	0.48
RSD_R	9.4	3.5	3.0	1.7	16.3	12.7	12.6	6.1	5.0	20.7	4.7	0.9	15.7	2.4
R	1.52	0.86	1.76	1.90	0.75	1.18	1.16	0.55	0.78	1.39	0.83	0.56	1.00	1.35

(*Continued*)

TABLE 4.7
(Cont.)

Sample ID	a	b	c	d	e	f	g	h	i	j	k	l	m	n
Number of laboratories	12	10	12	9	11	11	11	9	11	12	11	12	12	12
Number of replicates	24	20	24	18	22	22	22	18	22	24	22	24	24	24
oil/fat %														
Collaborative average	6.8	23.8	3.2	11.6	23.8	2.8	19.4	22.5	32.0	39.5	25.7	3.4	30.6	2.3
Certified Laboratories average[b]	6.9	24.0	3.2	11.3	23.5	2.7	19.7	23.2	32.0	39.0	25.0	3.7	30.5	2.3
AOCS Am 2-93 average for oilseeds							20.8	24.7						
Repeatability														
S_r	0.35	0.23	0.21	0.30	0.24	0.33	0.38	0.53	0.48	0.35	0.34	0.39	0.48	0.23
RSD_r	5.23	0.96	6.57	2.59	1.01	11.89	1.97	2.36	1.49	0.89	1.33	11.48	1.59	9.87
r	0.99	0.64	0.58	0.84	0.67	0.94	1.07	1.49	1.34	0.98	0.96	1.10	1.36	0.63
Reproducibility														
S_R	0.35	0.23	0.34	0.30	0.36	0.33	0.62	0.83	0.52	0.59	0.51	0.41	0.69	0.51
RSD_R	5.23	0.96	10.84	2.59	1.49	11.89	3.19	3.69	1.61	1.49	1.98	11.93	2.27	22.45
R	0.99	0.64	0.96	0.84	0.99	0.94	1.73	2.33	1.45	1.65	1.43	1.14	1.94	1.44

[a]Included in the summary is a comparison of the Filter Bag Technique with the results of analysis by AOCS Certified Laboratories using official AOCS or AOAC methods.
[b]See Table 4.6 for sample ID.
[c]AOCS Official Methods Ba 3-38, AOAC 920.39 or equivalent.
[d]r, repeatability value; R, reproducibility value; S_R, reproducibility of the SD; RSD_R, reproducibility of the relative SD.

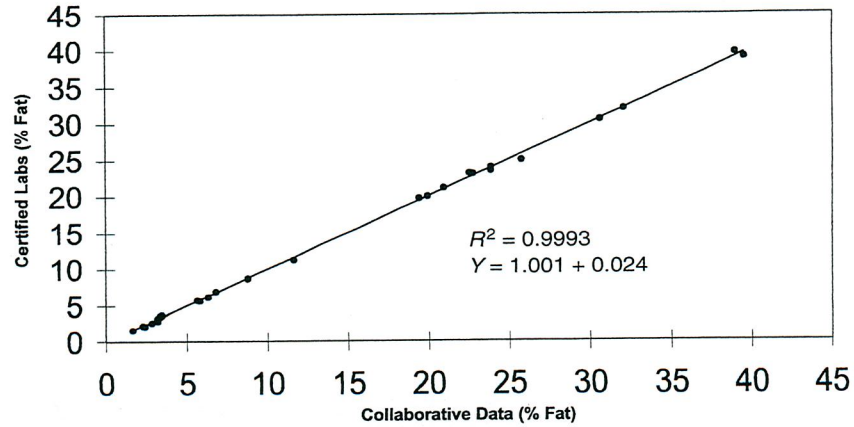

Fig. 4.12. Regression analysis of the accuracy of the analysis of fat/oil in 28 samples presented as 56 blind duplicates to an international group of collaborating laboratories using the FBT protocol relative to official methods.

The intralaboratory study and two collaborative studies provide a large body of evidence supporting the accuracy and precision of the FBT relative to Official Methods. These studies also show that the FBT can be applied to a wide range of samples. By utilizing high-temperature extraction and filter bags, this method is capable of rapidly processing large numbers of samples.

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