$$A = \frac{m}{100}y + \frac{d}{100}z$$

$$B = \left(\frac{100}{100-p}\right)\left(\frac{m}{100}v\right)$$

$$C = \left(\frac{100}{100-p}\right)\left(\frac{m}{100}y\right) + \left(\frac{100}{100-p}\right)\left(\frac{d}{100}z\right)$$

Alternatively the hydroxyl number of the original sample can be determined and used with A, B, and C in several possible combinations of three to obtain additional solutions for p, m, and d. When the acid number of the sample is small and there is reason to believe that inert substances are not present to any appreciable extent, the sum of p, m, d should approach 100%. Further, when this is the case, p = 100-e, d = e-m, and m = e-d.

Summary

The products obtained by esterification of polyethylene glycols with fatty acid, or by means of the reaction of ethylene oxide with fatty acids, can be analyzed for their content of monoester, diester, and unreacted polyethylene glycol by taking advantage of the extractability of the polyethylene glycol with water. A hot salt solution is used to insure selective extraction. Saponification and hydroxyl numbers are used to calculate the composition of the mixture; the molecular weights of the acid and glycol are presumed to be known.

Acknowledgment

The authors are indebted to A. Anderle, C. C. Arsement, and L. Donn for assistance during this investigation.

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Report of the F.A.C. Subcommittee on Dilatometry, 1956-1957

THE DILATOMETRIC METHODS SUBCOMMITTEE of the Fat Analysis Committee was established in 1953 to select a standard dilatometric method. Dilatometry is used extensively to compare the solid-liquid phase relationships of fats, based on the specific volume change that occurs when fat goes from a solid to a liquid state. A standardized procedure is needed because the dilation values depend not only on the composition of the fat but also on the manner in which the fat is solidified and conditioned.

The following is a summary of the collaborative work which led to the method that is being recommended by the subcommittee. The results are reported as solid fat index values, which are equivalent to the melting dilation in milliliters per 1,000 g. of fat. The melting dilation is the total dilation minus the dilation caused by the thermal expansion of the fat and indicator fluid.

First Series of Collaborative Samples

Because dilation values are to some extent empirical, it was decided to determine first how well the different laboratories agreed with each other. Therefore each of the eight laboratories represented on the subcommittee was asked to analyze three check samples by its own laboratory method. The results fell into two general groups.

SOLID FAT INDEX

	Sample	10°C.		21.1°C.		33.3°C.	
		Av.	Range	Av.	Range	Av.	Range
Group 1 (3 labs.)	1	29.1	28.6–29.6	22.2	21.6–22.7	7.1	7.0– 7.3
	2	28.5	27.8–29.5	18.3	17.6–19.1	10.4	9.9–11.0
	3	42.8	42.1–43.0	25.7	25.3–26.3	4.4	4.3– 4.5
Group 2 (5 labs.)	1	33.0	32.5–33.5	24.6	23.8–25.3	7.6	7.2– 8.0
	2	36.3	35.0–36.9	24.2	23.4–24.8	10.9	10.6–11.6
	3	50.0	49.0–51.0	32.3	31.7–33.5	4.9	4.2– 6.7

Sample 1: Prime steam lard.
Sample 2: Soybean oil shortening.
Sample 3: Soybean margarine oil.

The laboratories whose results were in group 1 included a tempering step in the conditioning of the fat. Those whose results were in group 2 did not. There were variations in the fat conditioning procedure in each group however.

Group 1

10 or 15 min. at either 0°C. or −5.3°C.
30 min. at 26.7°C.
15 min. at either 0°C. or −5.3°C.

Group 2

Either 70, 90, or 120 min. at 0°C.

There were also differences in the dilatometers that were used.

Number	Sample size	Confining fluid
5	9 g.	Water
1	6 g.	Water
1	3 g.	Mercury
1	4 g.	Alcohol-water

Second Series of Collaborative Samples

The following variations in the conditioning of the fat were studied with the second series of samples.

Procedure A

15 min. at 0°C.
30 min. at 26.7°C.
15 min. at 0°C.

Procedure B

15 min. at −5°C.
30 min. at 26.7°C.
15 min. at −5°C.

Procedure C

90 min. at 0°C.
(3 laboratories studied the effect of 30, 60, 90, and 120 min. at 0°C.)

Dilation readings were taken at 10°C., 21.1°C., and 33.3°C. when they were considered constant. The

time required for constant readings at these temperatures was also reported. The results are given in the following tables.

SOLID FAT INDEX

	Sample	10°C. Av.	10°C. Range	21.1°C. Av.	21.1°C. Range	33.3°C. Av.	33.3°C. Range
Procedure A (6 labs.)	4	40.1	38.8–40.7	21.2	20.7–22.0	10.1	9.5–10.5
	5	33.0	32.1–33.8	20.6	20.1–21.4	8.6	8.4– 9.0
Procedure B (6 labs.)	4	40.8	39.5–42.4	21.3	20.6–22.3	9.8	9.4–10.4
	5	33.2	32.5–33.9	20.5	20.1–21.0	8.5	8.1– 8.8
Procedure C (7 labs.)	4	47.2	46.2–48.0	28.7	27.9–30.2	11.8	11.6–12.1
	5	38.0	36.1–39.9	26.0	25.0–26.7	8.6	8.3– 8.9

VARIATIONS OF PROCEDURE C (3 LABORATORIES)

Chill time	Sample	10°C. Av.	10°C. Range	21.1°C. Av.	21.1°C. Range	33.3°C. Av.	33.3°C. Range
30 min.	4	44.0	42.8–45.0	27.5	26.8–28.5	11.5	11.3–11.6
60 min.	4	46.1	45.2–47.2	27.9	27.6–28.3	11.7	11.6–11.8
90 min.	4	47.0	46.2–47.7	28.3	27.9–29.4	11.7	11.7–11.7
120 min.	4	47.2	46.7–47.9	28.4	28.3–28.6	11.7	11.5–12.0
30 min.	5	36.5	35.6–37.2	25.1	24.9–25.2	8.4	8.1– 8.8
60 min.	5	37.2	35.9–38.5	25.4	24.9–25.9	8.5	8.3– 8.8
90 min.	5	37.3	36.1–38.3	25.5	24.9–25.7	8.5	8.3– 8.7
120 min.	5	37.9	36.4–39.6	25.7	25.2–26.5	8.6	8.5– 8.7

(Sample 4: mixture of hydrogenated animal and vegetable fats.)
(Sample 5: hydrogenated vegetable oil.)

TIME REQUIRED FOR CONSTANT READINGS (MINUTES)

	Sample	10°C. Average	10°C. Range	21.1°C. Average	21.1°C. Range	33.3°C. Average	33.3°C. Range
Procedure A	4	41	25–65	33	25–55	30	25–35
	5	37	25–55	30	25–40	33	25–40
Procedure B	4	40	25–60	35	25–65	29	25–30
	5	39	25–60	34	25–60	38	25–50
Procedure C	4	29	15–40	29	25–35	27	15–35
	5	50	15–80	35	25–50	30	25–40

RESULTS OF 3 LABORATORIES STUDYING EFFECT OF CHILL TIME IN PROCEDURE C

	Chill time	Time required for constant readings: 10°C. Average	10°C. Range	21.1°C. Average	21.1°C. Range	33.3°C. Average	33.3°C. Range
Sample 4	30	52	40–65	40	30–50	40	30–55
	60	40	30–60	43	30–65	40	30–55
	90	35	30–40	30	25–35	32	30–35
	120	37	30–50	37	35–40	38	35–40
Sample 5	30	62	60–65	43	40–50	35	30–40
	60	63	60–70	50	40–60	43	40–45
	90	60	40–80	42	35–50	37	30–40
	120	75	65–80	60	40–80	43	40–50

Differences in the results obtained by Procedures A and B were not significant, and it was decided to eliminate Procedure B from further collaborative work because of its requirement of a —5°C. constant temperature bath.

No definite relationship between the results and the time required for constant readings could be established. The results indicated that changes in readings after 30 min. were slight. In general, shorter equilibrium times were reported for the smaller dilatometers.

Third Series of Collaborative Samples

It was decided that Procedures A and C should receive further study with everyone using the same type of dilatometer. Five of the eight laboratories were already using the same kind of dilatometer. This was available as a regular stock item and was chosen for the remainder of the collaborative program.

The dilatometer, the procedures for preparing the sample, filling of the dilatometer, and correcting for the thermal expansion of the fat and confining fluid were essentially the same as they appear in the attached method.

SAMPLE 6 (6 LABORATORIES)

	Solid index: Average	Solid index: Range	Time for constant reading: Average	Time for constant reading: Range
Procedure A				
10°C.	27.6	26.4–28.3	23	14–37
21.1°C.	14.3	13.8–14.6	28	15–33
26.7°C.	10.0	9.8–10.1	22	15–28
33.3°C.	3.5	3.2– 3.6	24	19–30
Procedure C				
10°C.	32.8	31.6–33.7	23	15–45
21.1°C.	18.5	17.6–18.9	36	18–47
26.7°C.	10.6	10.1–11.0	35	18–43
33.3°C.	3.7	3.5– 3.8	27	17–35

(Sample 6 was a hydrogenated vegetable oil.)

Here again no satisfactory relationship between equilibrium time and dilation values could be established, and it was decided that readings would be taken at 30 min. The following results are typical.

Procedure A: Time	Procedure A: Solid fat index	Procedure C: Time	Procedure C: Solid fat index
15	13.9	18	18.6
20	14.5	31	18.9
25	14.6	35	18.7
25	14.5	40	18.3
28	14.5	45	18.4
33	13.7	47	17.6

It was hoped that a standard correction for the thermal expansion of fat could be established. However better reproducibility was obtained when the experimental thermal expansion in each determination was used to calculate the solid fat index.

EXPERIMENTAL THERMAL EXPANSIONS

range: .00810 to .00860 ml./g./°C.
average: .00836 ml./g./°C.

The solid index values (duplicate determinations) at 10°C. calculated with the experimental and average thermal expansion corrections were as follows.

	Experimental Correction		Average Correction	
Procedure A	26.2	26.5	27.1	27.7
	28.1	28.5	29.2	29.0
	28.0	28.0	27.9	26.7
	28.1	28.1	27.8	27.7
	26.7	27.2	26.8	27.4
	28.2	28.1	27.8	28.4
Procedure C	31.2	32.0	32.4	33.1
	33.4	33.2	33.6	33.5
	33.8	33.7	33.8	33.9
	33.4	33.5	33.2	33.4
	32.6	32.3	32.5	32.6
	33.0	33.1	32.6	33.0

Fourth Series of Collaborative Samples

Agreement was reached on all of the major details of the method except the procedure for conditioning the fat. Each laboratory was then asked to have two analysts make duplicate determinations on two different days, using Procedures A and C, so that a good statistical evaluation could be made.

Sample 7 was used for a preliminary check on the method as written. Samples 8 and 9 were used for the more extensive analyses. The results and a statistical analysis by H. P. Andrews are given in the following tabulations.

SAMPLE 7 (PROCEDURE A)

Solid Fat Index

Laboratory	10°C.		21.1°C.		26.7°C.		33.3°C.	
	Av.	Range	Av.	Range	Av.	Range	Av.	Range
7	37.1	36.9–37.3	21.2	21.0–21.4	14.8	14.5–15.0	4.4	4.2–4.6

(Sample 7: Hydrogenated vegetable oil.)

SAMPLE NO. 8[a]

(Average of Duplicate Determinations)
Procedures A and C

Laboratory	Analyst	Day	Solid fat index							
			10°C.		21.1°C.		26.7°C.		33.3°C.	
			A	C	A	C	A	C	A	C
1	1	1	33.4	38.6	20.7	26.4	16.6	17.9	8.5	8.5
		2	33.7	38.9	21.3	26.3	17.0	17.4	8.7	8.5
	2	1	33.5	39.9	20.8	26.4	16.9	17.8	8.7	8.6
		2	33.4	39.3	20.6	26.2	16.9	17.6	8.6	8.4
2	1	1	33.4	38.9	20.9	26.3	16.4	17.4	8.5	8.6
		2	33.4	39.2	20.6	26.6	16.5	17.9	8.6	8.6
	2	1	33.8	39.5	20.6	26.7	16.6	17.5	8.5	8.7
		2	33.2	39.1	20.6	26.4	16.1	17.4	8.4	8.8
3	1	1	32.3	38.7	20.1	26.0	16.3	17.6	8.7	8.8
		2	32.4	38.4	20.4	25.8	16.4	17.4	8.6	8.5
	2	1	34.0	40.0	20.5	26.1	16.5	17.6	8.4	8.4
		2	34.1	40.1	20.6	26.2	16.7	17.6	8.5	8.4
4	1	1	33.9	39.6	20.8	26.3	16.7	17.7	8.4	8.3
		2	33.7	39.5	20.6	26.3	16.4	17.4	8.3	8.3
5	1	1	33.6	39.5	20.1	26.2	16.2	17.6	8.4	8.8
		2	33.6	39.2	20.4	26.3	16.3	17.6	8.4	8.9
	2	1	33.6	39.1	20.5	26.2	15.9	17.6	8.5	8.7
		2	33.2		20.4		16.6		8.6	
6	1	1	32.3	38.8	20.5	26.3	16.4	17.8	8.3	8.7
		2	32.4	38.6	20.7	26.4	16.5	17.8	8.3	8.2
	2	1	33.0	39.8	21.1	26.9	16.7	18.3	8.5	8.6
		2	33.1	40.0	21.0	26.8	16.6	17.7	8.4	8.6
7	1	1	32.9	38.6	21.0	26.5	16.8	17.9	8.5	8.6
		2	33.0	38.5	21.0	26.6	16.9	17.7	8.6	8.6
	2	1	32.9	38.6	20.9	26.9	17.0	17.9	8.5	8.5
		2	33.0	38.4	21.1	26.7	17.0	17.9	8.8	8.8

[a](Sample 8: 50% lard and 50% tallow.)

SAMPLE 9

(Average of Duplicate Determinations)
Procedures A and C

Laboratory	Analyst	Day	Solid Fat Index							
			10°C.		21.1°C.		26.7°C.		33.3°C.	
			A	C	A	C	A	C	A	C
1	1	1	42.2	48.6	29.1	35.6	25.8	27.1	16.2	16.0
		2	42.1	48.6	29.7	35.3	26.1	26.8	16.2	16.0
	2	1	42.2	49.7	29.5	36.5	26.0	27.7	16.4	16.4
		2	41.9	49.3	29.2	36.3	25.8	27.7	16.2	16.4
2	1	1	42.1	50.0	29.4	36.4	25.7	27.0	16.2	16.4
		2	42.8	50.6	29.6	36.9	25.9	27.5	16.6	16.5
	2	1	42.5	50.1	29.2	36.6	25.3	27.1	16.2	16.2
		2	42.1	50.4	29.1	36.5	25.8	27.0	16.0	16.3
3	1	1	41.1	49.0	28.8	36.2	25.4	27.5	16.0	16.5
		2	41.3	49.1	29.0	36.4	25.6	27.5	16.0	16.2
	2	1	43.1	50.4	29.2	36.6	25.9	27.7	16.2	16.5
		2	43.2	50.5	29.4	36.7	26.0	27.7	16.2	16.4
4	1	1	42.6	50.4	29.5	36.3	26.0	27.5	16.1	16.0
		2	42.7	49.8	29.2	36.4	25.9	27.3	16.0	16.1
5	1	1	42.5	50.5	28.8	37.0	25.3	28.1	16.3	16.8
		2	42.4	50.5	28.7	37.2	25.1	28.3	16.1	17.1
		1	42.3	50.3	29.1	36.8	25.3	27.8	15.7	16.7
		2	42.3		29.2		25.6		16.2	
6	1	1	42.3	48.8	29.5	36.5	25.9	27.5	16.1	16.3
		2	41.6	48.4	29.4	36.3	25.7	27.8	15.9	16.2
	2	1	42.2	50.3	29.4	36.9	25.8	28.1	16.0	16.4
		2	42.1	49.8	29.5	36.8	25.7	27.5	15.8	16.4
7	1	1	41.9	49.6	29.6	36.7	26.1	27.9	16.3	16.4
		2	42.1	49.3	29.5	36.7	25.9	27.8	16.4	16.3
	2	1	41.9	49.3	29.6	36.9	25.8	27.9	16.1	16.2
		2	42.4	49.4	29.6	37.2	26.0	27.9	16.5	16.3

(Sample 9: 94% hydrogenated vegetable oil, 6% high melting mono- and diglycerides.)

Dr. Andrews also computed the 95% probability limits for a number of analytical situations.

SUMMARY OF MEANS

Procedures A and C

	Solid Fat Index							
	10°C.		21.1°C.		26.7°C.		33.3°C.,	
	A	C	A	C	A	C	A	C
Sample 8 Laboratory								
1	33.50	39.21	20.84	26.35	16.85	17.70	8.59	8.53
2	33.40	39.19	20.63	26.50	16.38	17.56	8.49	8.70
3	33.20	39.34	20.36	26.06	16.44	17.56	8.53	8.55
4	33.80	39.55	20.70	26.30	16.55	17.55	8.35	8.30
5	33.49	39.27	20.34	26.25	16.24	17.63	8.45	8.82
6	32.69	39.31	20.85	26.60	16.55	17.94	8.35	8.56
7	32.91	38.55	20.99	26.69	16.90	17.86	8.55	8.64
Average	33.42	39.20	20.67	26.39	16.55	17.69	8.47	8.59
Sample 9 Laboratory								
1	42.08	49.06	29.35	35.93	25.91	27.38	16.21	16.21
2	42.35	50.31	29.30	36.64	25.64	27.16	16.21	16.36
3	42.15	49.78	29.08	36.50	25.70	27.63	16.10	16.41
4	42.65	50.10	29.35	36.35	25.95	27.40	16.05	16.05
5	42.34	50.42	28.91	37.05	25.31	28.08	16.05	16.87
6	42.04	49.34	29.40	36.68	25.74	27.75	15.93	16.36
7	42.06	49.44	29.54	36.90	25.94	27.88	16.29	16.35
Average	42.24	49.78	29.28	36.59	25.74	27.61	16.12	16.37
Over-all standard deviation	.53	.66	.32	.39	.32	.33	.19	.20

SUMMARY OF VARIANCE COMPONENTS. SAMPLES 8 AND 9

Procedures A and C

	10°C.		21.1°C.		26.7°C.		33.3°C.	
	A	C	A	C	A	C	A	C
Duplicates	.05558	.0596	.0204	.0211	.0231	.0174	.0144	.0142
Days	.0217	.0200	.0148	.0111	.0279	.0311	.0139	.0104
Total within analyst	.0775	.0796	.0352	.0322	.0510	.0485	.0283	.0246
Std. deviation	.28	.28	.19	.18	.23	.22	.17	.16
Between analysts	.2101	.3582	.0208	.0628	—	.0145	—	.0077
Total within lab.	.2876	.4378	.0560	.0950	.0510	.0630	.0283	.0323
Std. deviation	.53	.66	.24	.34	.23	.25	.17	.18
Between labs.	—	—	.0463	.0611	.0511	.0463	.0072	.0089
Total	.2876	.4378	.1023	.1561	.1021	.1093	.0355	.0412
Std. deviation	.53	.66	.32	.39	.32	.33	.19	.20
Coefficient of variation	1.4%	1.5%	1.3%	1.2%	1.5%	1.5%	1.5%	1.7%

1. Two single determinations by an analyst run on different days should not differ by more than approximately 2.8% of the value for Procedure A and 2.5% of the value for Procedure C.
2. Separate determinations by two different analysts in a laboratory should not differ by more than approximately 3.4% of the value for Procedure A and 3.6% of the value for Procedure C.
3. Separate determinations run in two different laboratories should not differ by more than approximately 4.1% of the value by Procedure A and 4.2% of the value by Procedure C.

Although Procedure C always gave higher dilation values than did Procedure A, the percentage of difference was not the same for all samples.

SOLID INDEX AT 21.1°C. (AVERAGES)

Sample	Procedure C	Procedure A	% Difference
1	24.7	22.7	8.1
2	24.2	18.7	22.7
3	32.6	26.5	18.7
4	28.7	21.2	26.1
5	26.0	20.6	20.7
6	18.5	14.3	22.7
7	27.1	21.2	25.4
8	26.4	20.7	21.6
9	36.6	29.3	19.9

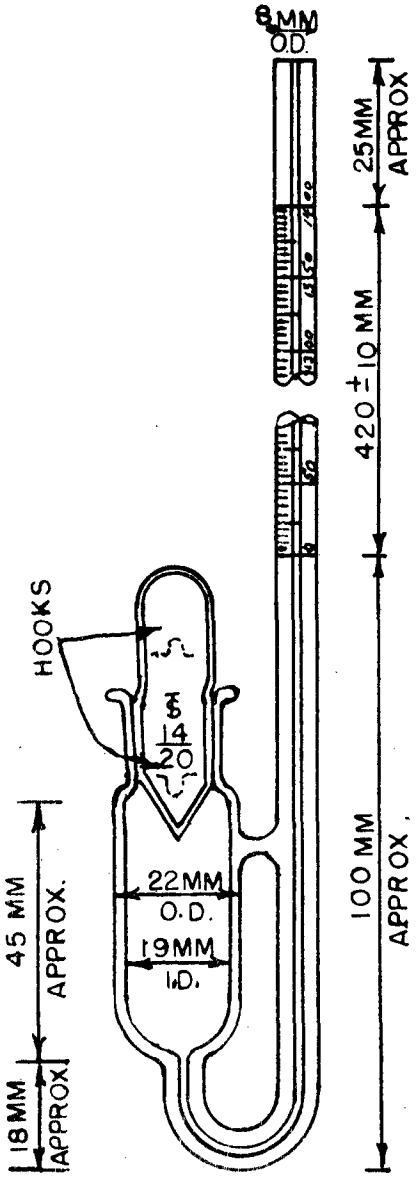

FIG. 1.

After deliberating on the results of the collaborative work and the relative merits of Procedures A and C, the subcommittee recommended that the attached method, which includes Procedure A, be submitted. A majority of the subcommittee felt that by including a tempering step one more nearly simulates the phase relationships normally found in plastic fats.

W. Q. BRAUN, chairman
W. F. SCHROEDER
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Solid Fat Index

Definition. The solid fat index is an empirical measure of the solid fat content. It is calculated from the specific volumes at various temperatures, utilizing a dilatometric scale graduated in units of ml. × 1000. Results are therefore expressed as melting dilation in ml. per kg. of fat.

Scope. Applicable to margarine oils, shortenings, and other fats with a solid index of 50 or less at 10°C., the method is empirical, and departures from the procedure may cause variations in results.

A. APPARATUS

1. Pyrex dilatometers constructed in accordance with the specifications in the diagram. The stem should be made from precision-bore, capillary tubing graduated in 0.005-ml. increments from 0 to 1.400 ml. with an over-all accuracy of at least ± 0.005 ml. The scale should be marked 0 to 1,400 in intervals of 50. The dilatometers should have identification numbers on the stems and stoppers.
2. Springs to fasten dilatometer stoppers.
3. Thermometer clamps for holding the dilatometers in the constant temperature baths.
4. Constant-temperature, water baths accurate to ± 0.05°C., equipped with means for adequate circulation. Solids indices at 10°, 21.1°, 26.7°, 33.3°, and 37.8°C. are commonly used to characterize shortenings and margarine oils. Therefore the baths required would be 0°, 10°, 21.1°, 26.7°, 33.3°, 37.8°, and 60°C. (See Note 1).
5. Vacuum pump capable of reducing pressure to 2 mm. Hg or less.
6. Pyrex 2 mm. I. D. capillary, 2-way stopcock with a burette tip.
7. Fisher Pyseal cement or equivalent.

B. REAGENTS

1. Potassium dichromate indicator solution *ca.* 1% in distilled water.
2. High vacuum grease—silicone type.
3. Petroleum solvent: A.O.C.S. Specification H2-41.
4. Distilled mercury.

C. CALIBRATION. All new dilatometers should be checked for accuracy.

1. Thoroughly clean and dry the dilatometer.
2. Clamp the dilatometer securely in an inverted position.
3. Clamp the capillary stopcock in place at the end of the dilatometer stem, and make a seal with Pyseal cement.
4. After the cement has hardened, immerse the tip of the stopcock into a reservoir of clean mercury which is at room temperature.
5. Using vacuum, draw the mercury into the dilatometer stem until the calibrated portion is full.
6. Successively withdraw .200-ml. portions of mercury into a tared 50-ml. beaker and record the weights.
7. Calculate the true volume in ml. contained in each measured scale-interval as follows (See Note 2).

$$\frac{\text{weight of mercury}}{\text{final scale reading} - \text{initial scale reading}} \times \frac{\text{sp. vol. of mercury at } T_R}{1{,}000}$$

where T_R is room temperature
1 ml. = 1,000 in scale reading

D. PROCEDURE

a) *Filling the Dilatometer*

1. Deaerate about 50 ml. of the indicator solution for 3 min. in a 250-ml. filter flask or a strong oil sample bottle at a pressure slightly above the vapor pressure of the solution at the temperature of deaeration. (The vapor pressure of water at 25°C. is 24 mm.). The indicator may also be deaerated by vigorous boiling for 15 min. at atmospheric pressure but should be cooled to room temperature before use.
2. Heat sample to 80°C. and deaerate in a 250-ml. filter flask or strong sample bottle at a pressure of 2 mm. Hg until no more gas bubbles are seen and for at least 2 min. The sample must be maintained in a liquid state and agitated vigorously during deaeration.
   Note. The indicator and sample should be used as soon as possible after they have been deaerated. The fat must be completely melted. Even slight crystallization occludes air.
3. Pipette 2 ml. of the indicator solution into the dilatometer bulb. Lubricate the stopper lightly with silicone grease, and weigh the assembled dilatometer to the nearest 0.01 g. on a torsion balance.
4. Carefully overlay the indicator with the sample and fill until the sample overflows. Insert the stopper so that the indicator solution rises to approximately the 1,200 mark of the stem when the stopper is securely sealed. The reading should be 1,200 ± 100 at 60°C.; if not, the determination should be started over.
5. Wash the fat from the outer surface of the dilatometer with petroleum solvent. Attach the retaining springs and reweigh the dilatometer when the solvent has evaporated.

b) *Measurement of Thermal Expansion*

1. Immerse the dilatometer to the 300 mark in the 60°C. bath and record reading after 15 min. Rechecks of the 60°C. reading at the end of the determination should agree with the 60°C. reference reading. Significant variations indicate faulty technique.
2. Transfer the dilatometer to the 37.8°C. bath, and immerse to the 300 mark. Read level of indicator at intervals of 5 min. until the change is less than 2 units in 5 min. Record the readings.

   Note. It is necessary for the sample to be completely melted at the lower temperature. If any seeding or clouding of the sample occurs, the sample must be remelted in the 60°C. bath, and the temperature of the other bath must be raised. If the reference bath temperatures are changed, appropriate substitution must be made in the calculations.

c) *Conditioning of the Sample*

1. Transfer the dilatometer to the 0°C. bath, and immerse to the 300 mark and hold for 15 min.
2. Transfer to a 26.7°C. bath, and hold for 30 min.
3. Transfer back to 0°C. bath, and hold for 15 min.

   Note. If an ice bath is used, provisions should be made for adequate water-circulation.

d) *Measurement of Dilation*

1. Transfer the dilatometer from the 0°C. bath to a bath at the lowest desired temperature. Immerse to the 300 mark, and record reading at 30 min.
2. Repeat at the next highest temperature and so on until readings have been obtained at all of the desired temperatures.

E. CALCULATIONS

1. Solid fat index at temperature T is
   (total dilation) − (thermal expansion) × (60 − T)
   where
   T is observed temperature
   Vc(T) is volume correction for expansion of glass and water at T
   R(T) is dilatometer reading at T
   W is weight of sample.
2. Thermal expansion of sample per degree C in ml./kg. is
$$\frac{R(60) - R(37.8) - Vc\ (37.8)}{W \times (60 - 37.8)}$$
   (See Notes 3 and 4)
3. Total dilation between T and 60 C. in ml./kg. is
$$\frac{R(60) - R(T) - Vc(T)}{W}$$

VOLUME CORRECTIONS (Vc)

Bath temp. °C.	60°C. Reading				
	1,000	1,100	1,200	1,300	1,400
0	22.0	20.3	18.6	16.9	15.2
5	22.2	20.5	18.7	17.0	15.3
10	21.8	20.1	18.4	16.7	15.1
15	21.0	19.5	17.8	16.2	14.6
20	19.8	18.4	16.8	15.3	13.8
25	18.4	17.0	15.6	14.1	12.7
30	16.6	15.3	14.0	12.7	11.4
35	14.4	13.3	12.2	11.1	10.0
40	12.0	11.0	10.2	9.2	8.3
45	9.4	8.7	8.0	7.2	6.5
50	6.6	6.1	5.6	5.1	4.5
55	3.2	3.0	2.8	2.5	2.3
60	0	0	0	0	0

F. REPRODUCIBILITY. Collaborative studies have shown that the following reproducibility can be expected:

1. two single determinations made on different days by an analyst should not differ by more than approximately 2.8% of the value;
2. separate determinations by two different analysts in a laboratory should not differ by more than approximately 3.4% of the value; and
3. separate determinations in two different laboratories should not differ by more than approximately 4.1% of the value.

G. NOTES

1. The basic procedure described above is applicable at temperatures other than those specified, and the committee recognizes that sometimes such deviations are necessary. These depend on the composition and the character of the fat. It is hoped however that within limits a uniform temperature range may become established in the industry. Meanwhile further work is planned in this direction.
2. In order to meet the specifications of this method, the dilatometer scale must be accurate to 0.005 ml. or less (1 scale graduation) from 0 to 1,400. It is necessary to draw correction curves from the calibration data for those dilatometers which do not meet specifications, and corrected readings must be used to calculate the solid fat index.
3. Vc from the table represents the combined corrections for the expansion of glass and water and applies to Pyrex glass only. If dilatometer is constructed of glass other than Pyrex, the corrections must be redetermined.
4. The normal liquid thermal expansion is 0.83 − 0.85 ml./kg. If determined values differ from this, it is advisable that they be rechecked carefully.

Report of the F.A.C. Total Neutral Oil Subcommittee 1956-1957

THE TOTAL NEUTRAL OIL SUBCOMMITTEE of the Fat Analysis Committee of the American Oil Chemists' Society was appointed in 1953 to select a standard method for the determination of total neutral oil.

Three methods were considered for study by the subcommittee: a modification of the Wesson method, J. Oil and Fat Industries, *3,* 297–305 (1926); modifications of the chromatographic method as proposed by Linteris and Handschumaker, J. Am. Oil Chemists' Soc., *27,* 260–264 (1950), and the crude oil impurities technique, which is an estimate based on the summation of the acetone-insoluble, free fatty acids, and moisture content of the sample. The latter technique was discarded as a possible method because it was not a single procedure. The chromatographic and Wesson techniques were studied quite extensively by the subcommittee.

In 1954 a sample of crude cottonseed oil was analyzed by the subcommittee, using the Wesson method and the chromatographic method. Each collaborator ran the chromatographic method, using the same alumina as well as his own supply of alumina. The statistical analysis of the 1954 study indicated that the precision of the Wesson method and the chromatographic method was comparable and that the agreement among laboratories using their own alumina for the chromatographic method was satisfactory.

In 1955 a "nested design" was used by the subcommittee to compare the Wesson method with the chromatographic method proposed by Archer-Daniels-Midland, using six different crude oils. The statistical analysis of the 1955 study indicated that the precision of the chromatographic method was as good as, if not superior to, the Wesson method. Since the majority of the subcommittee members favored the