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Report of the Committee on Analysis of Commercial Fats and Oils

Fall Meeting, November 1948

Determination of Free Fatty Acids for Refining Test

THE present method for the determination of the free fatty acids in crude vegetable oil, Ca 9a-41, page 4, D (for refining test) specifies the use of a 7.05-gram sample inspection of free acid content. Since many crude oils run too low for this method to be entirely satisfactory, the Committee recommends that Method Ca 5a-40 be used instead and that Method Ca 9a-41 be rewritten accordingly.

SUBCOMMITTEE ON F. A. C. COLOR METHOD:

(E. W. Blank, Chairman)

Copies of a questionnaire relating to the F. A. C. Color Method were submitted to approximately 50 users of the F. A. C. Color Standards. Much of the criticism of the F. A. C. Color Standards centers around the 11A, 11B, and 11C standards. A suggestion has been made that these standards be redesignated. Your chairman has been unable to obtain any unanimity of opinion on this proposal. It may be pointed out that irrespective of how the standards are designated, the problem of interpretation remains a personal one. The problem was thoroughly discussed in a meeting of the subcommittee at New York and plans made for proceeding with the problem.

SUBCOMMITTEE ON DETERMINATION OF PEROXIDE VALUES:

(A. R. Baldwin, Chairman)

Three separate studies of the Wheeler method for determining peroxide values have been made by the collaborating groups. The extent of agreement within and among several laboratories was evaluated in the first test. The data shown below indicate that agreement on duplicate samples within each laboratory was very good and that all results agreed surprisingly well.

The second series was designed to note the effects of varying the reaction time from one to five minutes and of varying the interval between water addition and titration. Differences between values obtained at one- and five-minute reaction times were not as large as were expected. However, in general, the peroxides appeared to be somewhat higher after five minutes of reaction than after one minute. There was greater variation among laboratories on corn oil than on lard, but the duplicability within individual laboratories again was very good with an average difference between duplicates of about 0.5 Me/Kg.

Peroxide Values of Oxidized Fats
(Unaltered Wheeler Method)

Collaborator	1	2	3	4	5
Peroxides (Me/Kg)					
Lard	25.8	27.0	26.5	25.2	24.5
	26.1	26.0	24.5	25.0	24.7
	26.4	25.7	25.5	24.9	24.5
	26.5	25.0	25.3	25.4	.....
			25.6		
Ave.	26.2	25.9	25.5	25.1	24.6
Corn oil	21.8	23.3	23.4	20.7	19.7
	22.2	23.0	23.1	20.9	20.0
	22.0	22.9	23.4	20.9	20.2
	22.1	22.0	23.4	21.5	.....
			23.2		
Ave.	22.0	22.8	23.3	21.0	20.0

The lapsed time between water addition and titration up to five minutes had no effect on the peroxide values of lard, but wide variation was found between titration immediately and after five minutes for corn oil. Titration immediately after water addition would seem to be indicated from these results.

The third series of samples was distributed for the purpose of studying the effects of sample size (1.0 to 10.0 g.) on peroxide values. Several reports in the literature have indicated that with increased sample size the apparent amount of peroxides is reduced significantly. For both lard and corn oil the peroxide values, when 10 grams of fat were used, ranged 10 to 20 per cent lower than when only one gram of fat was analyzed. In fact, sample size thus far in the investigation of the Wheeler peroxide method appears to be the most significant variable.

SUBCOMMITTEE ON UNSAPONIFIABLE MATTER:

(C. P. Long, Chairman)

During the year, cooperative work was done on two samples of tallow. These were analyzed by the A. O. C. S. Method Ca 6a-40 and the modified S. P. A. ethyl ether method official for the A. O. A. C.

The results by both methods show rather wide spreads and are not such or sufficient to justify any recommendations for changes in methods.

The committee recommends that cooperative work on the method be continued.

SUBCOMMITTEE ON ANALYSIS OF DRYING OILS:
(J. C. Konen, Chairman)

The Drying Oil Subcommittee is actively considering eight analytical methods as follows: Ash, Diene Value, Flash and Fire Point, Hydroxyl Value, Iodine Value, Non-Volatile, Saponification Value, and Viscosity. Of these methods, three are generally acceptable to members of the subcommittee and are being edited prior to final submission as tentative methods. These include Flash and Fire Point, Saponification Value, and Viscosity. The methods for Diene Value and Hydroxyl Value are now being checked collaboratively by members of the subcommittee; and if reproducibility of results is obtained, the methods will be submitted as tentative methods. Methods for Ash and non-Volatile content are still in preliminary stages.

Tentative Method Ka 7-48

Flash and Fire Points

Open Cup Method

Definition: This method determines the temperature at which the sample will flash and burn.

Scope: Applicable to natural and synthetic drying oils and their fatty acids, except those which for any reason flash below 300°F.

A. APPARATUS:

1. Thermometer, A. O. C. S. Specification H 5-40.
2. Cleveland open flash cup, A. S. T. M. Designation D 92-33, constructed of brass and conforming to the dimensional requirements prescribed in Table I. The beveled edge of the cup is at an angle of ca 45°. There may be a fillet of ca 5/32 inch (3.97 mm.) in radius inside the bottom of the cup.
3. Heating plate, constructed of brass, cast iron, wrought iron, or steel, 1/4 inch (6.35 mm.) thick and 6 inches (152.4 mm.) in diameter. There is a plane depression 1/32 inch (0.79 mm.) deep in the center of the plate with diameter just sufficient to fit the cup and centered with a circular opening cut through the plate, 2 3/16 inches (55.0 mm.) in diameter. The plate is covered with a sheet of hard asbestos board 6.35 mm. thick and of the same shape as the metal plate.
4. Heat source, gas burner, alcohol lamp, or electric heater with rheostat control. Whatever form of heat is used, under no circumstances are the products of combustion or free flame allowed to come up around the cup. If a flame heater is used, it may be protected from drafts or excessive radiation with any suitable shield that does not project above the upper surface of the asbestos board. The heat source is centered under the plate opening and must not produce local superheating.
5. Metal flame test burner.

B. PROCEDURE:

(a) *Flash Point*

1. The flash and fire point determinations are carried out in a room or compartment free from air drafts and darkened sufficiently so that the flash is readily discernible. Avoid breathing over the surface of the sample.
2. Fill the cup with the oil or melted fat sample so that the top of the meniscus is exactly at the filling line. Suspend or secure the thermometer in a vertical position with the bottom of the bulb ca 1/4 inch from the bottom of the cup and in a position half way between the center and back of the cup.
3. Heat the sample at a rate not to exceed 30°F. (16.7°C.) rise per minute to within ca 100°F. (55.6°C.) of the flash point. Thereafter regulate the rate of heating so that the temperature of the sample increases 9° to 11°F. (5° to 6.1°C.) per minute.
4. Apply the test flame which is ca 1/8 inch (3.17 mm.) in diameter as the temperature reaches each successive 5°F.(2.8°C.) mark. Pass the flame in a straight line or on the circumference of a circle having a radius of at least 150 mm. (ca 6 inches) across the center of the cup and at right angles to the diameter passing through the thermometer. The test flame shall, while passing across the surface of the sample, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup shall be ca 1 second.
5. The flash point is the temperature indicated by the thermometer when a flash appears at any point on the surface of the sample. The true flash must not be confused with a bluish halo that sometimes surrounds the test flame.

(b) *Fire Point*

1. Continue the heating, after the flash point determination, as directed in paragraph 3 and in the manner prescribed for the flash point until the fire point is reached.
2. The fire point is the temperature indicated by the thermometer when application of the test

TABLE I

Dimensional Requirements for Cleveland Open Flash Cup

	Inches			Millimeters		
	Minimum	Normal	Maximum	Minimum	Normal	Maximum
Inside diameter immediately below filling mark	2 15/32	2 1/2	2 17/32	62.7	63.5	64.3
Outside diameter below flange	2 21/32	2 11/16	2 23/32	67.5	68.3	69.1
Inside height from center to bottom of rim	1 9/32	1 5/16	1 11/32	32.5	33.3	34.1
Thickness of bottom	7/64	1/8	9/64	2.8	3.2	3.6
Distance from rim to filling mark	23/64	3/8	25/64	9.1	9.5	9.9
Distance, lower surface flange to bottom of cup	1 7/32	1 1/4	1 9/32	31.0	31.8	32.6
Vertical distance, upper surface flange to rim	7/64	1/8	9/64	2.8	3.2	3.6
Thickness of rim	5/64	3/32	7/64	2.0	2.4	2.8
Width of lower surface of flange	9/16	19/32	5/8	14.3	15.1	15.9

flame causes burning for a period of at least 5 seconds.

Tentative Method Ka 8-48

Saponification Value

Definition: The saponification value is the amount of alkali necessary to saponify a definite quantity of the sample. It is expressed as the number of milligrams of potassium hydroxide required to saponify 1 gram of the sample.

Scope: Applicable to all natural and synthetic drying oils and their fatty acids.

A. APPARATUS:

1. Erlenmeyer flasks, Corning Alkali Resistant, Kimble Resistant or equivalent, 250 or 300 ml.
2. Air condensers, minimum 650 mm. long.
3. Water bath or a hot plate with variable heat control.

B. SOLUTIONS:

1. Hydrochloric acid, 0.5 N, accurately standardized.
2. Alcoholic potassium hydroxide, place a few g. (5 to 10) of KOH in a 2-liter flask and add from 1 to 1.5 liters of 95% ethyl alcohol (U. S. S. D. Formulas 30 and 3A are permitted) and boil on a water bath under a reflux condenser for 30 to 60 minutes. Distill and collect the alcohol. Dissolve 40 g. of potassium hydroxide, low in carbonate, in 1 liter of the distilled alcohol, keeping the temperature below 15.5°C. (60°F.) while the alkali is being dissolved. This soln. should remain clear.
3. Phenolphthalein indicator soln., 1% in 95% alcohol (see E, 1).

C. PROCEDURE:

1. Melt the sample if it is not already liquid (see E, 2) and filter through filter paper to remove solid impurities and traces of moisture. The sample must be completely dry. Drying oils of high viscosity should not be filtered.
2. Weigh a sample of such size that the back titration is 45 to 55% of the blank. This is usually 4 to 5 g. Add 50 ml. of the alcoholic KOH with a pipet and allow the pipet to drain for a definite period of time.
3. Prepare and conduct blank determinations simultaneously with the sample and similar in all respects.
4. Connect the air condensers and boil gently but steadily until the sample is completely saponified. This usually requires ca 30 minutes for normal samples. Be careful that the vapor ring in the condenser does not rise to the top of the condenser or there may be some loss.
5. After the flask and condenser have cooled somewhat, but not sufficiently to jell, wash down the inside of the condenser with a little distilled water. Disconnect the condenser, add ca 1 ml. of indicator and titrate with 0.5 N HCl until the pink color has just disappeared.

D. CALCULATION:

$$\text{Saponification value} = \frac{28.05\ (\text{titration of blank} - \text{titration of sample})}{\text{Weight of sample}}$$

E. NOTES:

1. A "masked phenolphthalein indicator" may be used with off-color materials. Prepare by dissolving 1.6 g. of phenolphthalein and 2.7 g. of methylene blue in 500 ml. of alcohol (U. S. S. D. Formula 30 or 3A). Adjust the pH with alcoholic alkali solution so that the greenish-blue color is faintly tinged with purple. Color change is from green to purple when going from acid to alkali.
2. When the saponification value is required on the fatty acids, the preparation and separation are performed as directed in A. O. C. S. Official Method, Cd 6-38.

Tentative Method Ka 6-48

Viscosity

Definition: The viscosity of a liquid is the resistance experienced by one portion of a liquid in moving over another portion of the liquid. The absolute unit of viscosity in the C. G. S. system is the poise which is expressed as dyne-seconds/sq. cm. The method determines the kinematic unit of viscosity, the stoke, which is equal to poises divided by density.

Scope: Applicable to all natural and synthetic drying oils.

A. APPARATUS:

1. Gardner-Holdt sample tubes (see D, 1). These are clear glass flat-bottom tubes 10.75 mm. ± 0.025 mm. inside diameter, 12.3 mm. outside diameter, and 112 mm. ± 0.05 mm. long. A pair of etched lines located 5 mm. and 13 mm. from the open end of the tube determines the volume of sample and the size of bubble.
2. Gardner-Holdt bubble type viscosity standards (see D, 2). The comparison in order of viscosities is given below for the Varnish Series, A to T; the Heavy Bodied Series, U to Z_6; the Rubber Series, Z_7 to Z_{10}; and the Lithographic Series, 000 to 8:

Gardner-Holdt Designation	Viscosity in Stokes	Time of Bubble Travel in Sec.	Gardner-Holdt Designation	Viscosity in Stokes	Time of Bubble Travel in Sec.
A	0.50	.....	O	3.70	5.4
B	0.65	.....	P	4.00	5.8
C	0.85	.....	Q	4.35	6.4
D	1.00	1.467	R	4.70	6.9
E	1.25	1.83	No. 00	4.80	7.0
F	1.40	2.05	S	5.00	7.3
G	1.65	2.42	T	5.50	8.1
No. 000	1.80	2.64	U	6.27	9.2
H	2.00	2.93	No. 0	8.00	11.7
I	2.25	3.30	V	8.84	13.0
J	2.50	3.67	W	10.70	15.7
K	2.75	4.03	X	12.90	18.9
L	3.00	4.4	No. 1	14.40	21.1
M	3.20	4.7	Y	17.60	25.8
N	3.40	5.0	Z	22.70	33.3
No. 2	23.50	34.5	No. 5	120.0	176.0
Z_1	27.0	39.6	Z_6	148.0	217.1
No. 3	34.0	49.8	No. 6	200.0	293.4
Z_2	36.2	53.1	Z_7	388.0	569.2
Z_3	46.3	67.9	Z_8	590.0	865.5
No. 4	62.1	91.1	Z_9	855.0	1254.3
Z_4	63.4	93.0	Z_{10}	1066.0	1563.8
Z_5	98.5	144.5	No. 8	1250.0	1833.7

3. Constant temperature bath 25°C. ± 0.1° C. (77°F. ± 0.2°F.).

B. PROCEDURE:

1. Pour the sample into the Gardner-Holdt tube up to the lower of the two tube markings.
2. Insert a cork of suitable size to the upper tube marking. It is essential that the air bubble in

the sample tube be the same size as in the standard tubes.

3. Place the sample and standard tubes having approximately the same rate of bubble rise in the constant temperature bath at 25°C. Hold in the bath until the temperature of the contents of the tube is 25°C. If necessary adjust the level in the sample tube so that the air bubble will be of the same size as that in the standard tubes.
4. Hold the sample tube and standard tube in a vertical position with the cork end at the top and then rapidly invert them together. Using the bottom meniscus for comparison, select the standard tube in which the air bubble rises at a rate most nearly corresponding with the rate of the air bubble rise of the sample.

C. CALCULATIONS:

1. Express the viscosity by the Gardner-Holdt letter designation or by the stoke viscosity corresponding to the standard tubes (see A, 2).
2. Express the viscosity of extremely high viscosity usually expressed in time of bubble travel in seconds or in stokes by dividing the time in seconds by 1.467 (see D, 3).

D. NOTES:

1. Sample tubes are available from H. A. Gardner Lab., Inc., Apparatus Division, 4723 Elm Street, Bethesda, Maryland. Two grades are available, Grade B for general control work as specified under A2, and Grade A for most accurate measurements having an internal diameter of 10.75 ± 0.005 mm. and other measurements as specified for Grade B tubes.
2. Standard viscosity tubes are available from the Gardner Laboratory.
3. In determining the time of bubble travel in seconds, the bubble is started from the cork and the time interval is measured on a stopwatch until the bubble just begins to flatten against the glass end of the specimen tube.

SUBCOMMITTEE ON ANALYSIS OF LECITHIN
(J. K. Gunther, Chairman)

Collaborative analyses on the determination of the acid value and phosphorus content of soybean lecithin were carried on during the year. However, no recommendations were being made except that the work be continued for another year.

Flash and Fire Method Cc 9-40 has been found unsatisfactory for solvent extracted oils which contain sufficient solvent to cause them to flash below 275° or 300°F. Accordingly, a closed cup method is proposed similar to the A. S. T. M. procedure using the Pensky-Martens tester. This method is submitted tentatively for the reason that some method for testing low flash point oils is urgently needed by the industry. However, further investigation is to be done by a subcommittee before the status of this method is changed. Method Cc 9-40 (open cup) will be restricted to those oils which flash above 300°F.

Tentative Method Cc 9b-48

Flash Point

Modified Closed Cup Method, A. S. T. M. Designation D 93-46

Definition: This method determines the temperature at which the sample will flash when a test flame is applied under the conditions specified for the test.

Scope: Applicable to animal, vegetable, and marine fats and oils which for any reason flash at temperatures below 300°F.

A. APPARATUS:

1. Pensky-Martens closed cup flash tester, A. S. T. M. Designation D 93-46, complete. Either hand or motor stirring is allowable, but the latter is preferred.
2. Thermometer, A. S. T. M., P. M., high range (10F-39).
3. Thermometer, A. S. T. M., P. M., low range.

B. PROCEDURE:

Caution: Samples should be kept at as low a temperature as possible from the time they are drawn until they are tested and they should be tested as promptly as possible. Results may not check after samples have stood around at room temperature for 24 hours.

1. Fill the cup (see Note 1) with the oil or fat sample so that the top of the meniscus is exactly at the filling line, place the lid on the cup and properly engage the locating devices. Insert the thermometer and suspend so that the bottom of the bulb is exactly 1¾ inches (4.445 cm.) below the level of the rim of the cup, which corresponds to the level of the lower surface of the portion of the lid inside the rim.
2. Light the test flame and adjust so that it is the size of a bead 5/32 in. (3.97 mm.) in diameter.
3. Heat the sample so that the temperature increases not less than 9°F.(5°C.) nor more than 11°F.(6.1°C.) per minute. During the heating, turn the stirring device from one to two revolutions per second.
4. At 30°F.(14.5°C.) below the actual flash point, but at least at 170°F., discontinue stirring and apply the test flame by operating the device which controls the shutter and lowers the test flame into the shutter opening. Lower the test flame in ½ second and leave in a lowered position for 1 second, then quickly return to the raised or high position. As soon as the test flame has been returned to the high position, resume stirring.
5. Repeat the application of the test flame as the temperature reaches each successive 10°F. mark.
6. The flash point is the temperature indicated on the thermometer at the time of the flame application that causes a distinct flash in the interior of the cup. The true flash must not be confused with the bluish halo that sometimes surrounds the test flame.

C. NOTE:

1. It is imperative that the apparatus, especially the cup, be scrupulously clean and free from any foreign substances.

A. R. BALDWIN, E. W. BLANK, E. L. BOLEY, D. S. BOLLEY, L. R. BROWN, C. A. COFFEY, G. A. CRAPPLE, GEORGE CRUMP, F. R. EARLE, H. T. IVESON, F. G. DOLLEAR, JACOB FITELSON, J. K. GUNTHER, J. C. KONEN, M. L. LAING, C. P. LONG, W. O. LUNDBERG, J. E. MARONEY, L. B. PARSONS, H. S. SCHUETTE, FRANCIS SCOFIELD, F. C. WOEKEL, V. C. MEHLENBACHER, chairman.