# Report of Committee on Analysis of Commercial Fats and Oils-1939-40

THE Committee on Analysis of Commercial Fats and Oils is ready to report the results of its investigation of six methods and recommends the adoption of these. These are the Titer, the Modified Gardner Break test, the Detection of Tristearin in lard, the Villavecchia test for sesame oil, calculations for the Hydroxyl Value and the Smoke, Flash, and Fire points. A large amount of data have been collected in the study of these methods. This work has extended over a period of two to three years.

### Titer

The Committee has approved vertical stirring. The advantages of this over horizontal stirring are that it gives a sharper end point, is more convenient, and can easily be made mechanical by adapting a suitable motor and coupling arrangement to the stirrer.

The specifications for the titer thermometer have been revised. The new thermometer is more easily read, covers a greater range, and has been made partial immersion.

The Committee has found that a differential temperature of 10°C. between the titer point and the bath is insufficient for low titers. It is therefore recommended that this be increased.

The Committee wishes to emphasize the fact that no changes have been made in the titer determination which will give results different from those that might be obtained with the horizontal stirring method providing the latter is correctly performed. The committee believes that the proposed modification will make it easier for different chemists to obtain uniform and consistent results.

### Detection of Tristearin in Lard (Boemer No.)

The Committee has found that the F.A.C. capillary tube method for melting points yields consistent and satisfactory results with both the glycerides and the fatty acids. Since this method is already official with the American Oil Chemists' Society and the American Chemical Society, and also because it is more convenient than the use of a sulphuric acid bath, we recommend its adoption for this determination.

Further work has been done on the crystallization from acetone at 30°C. This work has indicated that if care is taken, sufficient crystals of a definite composition can be obtained. The method has been rewritten for the purpose of clarifying some of the details.

### Smoke Point

Several years ago the A.S.T.M. Cleveland Open Cup apparatus was adopted for use in the Smoke, Flash and Fire Point methods. We now suggest that these methods be rewritten so that they will be in accord with the latest revision of the A.S.T.M.

It is recommended that when the methods for the Smoke, Flash and Fire Points are prepared for publication in the A.O.C.S. methods, that they be allowed to remain together as they are herein written. The methods at present in the A.O.C.S. booklet are separated by the procedure for F.A.C. colors.

### Villavecchia Test

The Villavecchia test for the qualitative detection of sesame oil was investigated and approved. This method is already official with the Association of Official Agricultural Chemists.

### Modified Gardner Break

The Modified Gardner Break Method for soybean oil was studied at the request of the Uniform Methods and Planning Committee of the American Oil Chemists' Society. This method is of old standing and has been previously studied by others. Only two minor changes are recommended. A tall form beaker helps to prevent loss from foaming. The thermometer recommended will insure better control of the temperature.

### Hydroxyl Value

The Committee recommends that the method for calculations of Hydroxyl value be included with the Acetyl Value determination. References to the details of these calculations may be found at the end of the method which is attached.

### Color Reading

The Committee has approved the use of the one inch column in the Lovibond system of color reading for oil and fat samples which cannot be read in a 5¼ inch column. This item has been referred to the Color Committee for further handling.

### International Fat Commission Program

Unfortunately, the disturbances in Europe have held up our program of cooperation in the study of fat and oil analysis with the International Fat Commission. It is hoped that this may be resumed in the future.

### General

Because of the large amount of work ahead, this committee has been enlarged during the past year. The committee's schedule for future work is as follows:

1. Study of a method for the quantitative determination of peanut oil.
2. Study of a method for the determination of insoluble bromides.
3. Study of the various moisture methods for fats and oils.
4. Study of methods for the determination of unsaponifiable matter.
5. Study of methods for the determination of cholesterol and phytosterol.
6. Further study of the F.A.C. color standards.
7. Some study of iodine number determinations.

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## TITER

### Solidification Point of the Fatty Acids

**Reagents**

1. Glycerol Caustic. Dissolve with the aid of heat, 250 gms. of solid potassium hydroxide in 1,250 gms. of glycerine (dynamite or C.P. grade). To avoid foaming, do not heat above 135-145°C.
2. Sulphuric Acid. 30% by weight of sulphuric acid. This may be readily prepared by adding 16 ml of sulphuric acid (sp.gr.1.84) to 70 ml. of water.

**Apparatus**

1. Two liter Griffin low form beaker.
2. Wide mouth bottle, capacity 450 ml, height 190 mm, inside diameter of neck, 38 mm.
3. Test tubes, length 100 mm. diameter 25 mm, with or without rim. These tubes may have an etched mark extending around the tube at a distance of 57 mm from the bottom to show the height to which the tube is to be filled.
4. Saponification vessel. This may be a flask, beaker or casserole of a convenient capacity. The form of this vessel is not important so long as it is satisfactory for the saponification.
5. Laboratory thermometer, 0-150°C.
6. Stirrer. 2-3 mm outside diameter, one end bent in the form of a loop of 19 mm diameter. Glass, nichrome, stainless steel or monel wire may be used. The upper end can be formed to accommodate hand stirring or attached to a mechanical stirrer.
7. Titer thermometer:

Type: Etched stem glass.

Liquid: Mercury.

Range and subdivision: Minus 2 to plus 68 degrees centigrade in 0.2 degree graduations.

Total Length: 375 to 385 mm.

Stem shall be constructed of suitable thermometer tubing with a diameter of 6 to 7 mm. May be plain front or of the magnifying lens type. Red reading mercury is preferable but not obligatory.

Bulb: Corning normal or equally suitable thermometric glass. Diameter not less than 5.5 mm but not greater than that of the stem. Length 15 to 25 mm.

Distance to minus 2 degree mark from bottom of bulb: 50 to 60 mm.

Length of graduated scale: 300 mm minimum.

Length of unchanged capillary between lowest graduation mark and bulb 13 mm minimum.

Expansion chamber: To permit heating the thermometer to at least 85°C.

Length of unchanged capillary between uppermost graduation mark and expansion chamber: 10 mm minimum.

Top Finish: Glass ring.

Tubing above mercury shall be evacuated or filled with nitrogen or other suitable inert gas.

Graduation: All lines, figures, and letters to be clear cut and distinct. Each degree mark to be longer than the remaining lines. Graduations to be numbered at each multiple of 2 degrees.

Immersion: 45 mm.

Marking: "F.A.C. Titer Test," serial number, and the manufacturer's name or trade mark shall be etched upon the stem. The words 45 mm immersion shall also be etched upon the stem, and a line shall be eched around the stem 45 mm above the bottom of the bulb.

Scale Error. The error at any point on the scale, when the thermometer is standardized at 45 mm immersion, shall not exceed 0.2 degrees C.

Standardization: The thermometer shall be standardized at intervals of approximately 10 degrees C. and for an average temperature of the emergent mercury column of 25 degrees C.

Case: The thermometer shall be supplied in a suitable case on which shall appear the marking: "F.A.C. Titer Test," minus 2 degrees to plus 68 degrees graduations.

Note: For the purpose of interpreting these specifications, the following definitions apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a micrometer.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

**Preparation of the Fatty Acids**

1. Weigh 110 gms. of glycerol caustic into the saponification vessel, stir while heating to 150 C., add 50 ml of melted fat and reheat. In some cases a little additional caustic may be found necessary to insure complete saponification.
2. Continue stirring, being careful not to heat above 150°C., until completely saponified (see note 1).
3. Cool slightly, add 200-300 ml of water and after the solution of the soap add 50 ml of the sulphuric acid, stirring during the addition. After separation of the fatty acids, more water may be added if desired and boiling continued until the fatty acids are completely melted and clear. If a gas burner is used for heating, the water level should be high enough to prevent scorching on the sides of the dish.
4. The aqueous layer containing the sulphuric acid may be removed from under the fatty acid layer by an appropriate siphon. Again add water and boil two or three minutes, making sure that all of the fatty acids are melted and clear. High melting point fats are sometimes slow to melt and clear. The fatty acid layer should be carefully inspected while it is quiet, to be sure all has melted.
5. Siphon off water again and repeat, if necessary, with water as under *4* until wash water is neutral to methyl orange.
6. Carefully remove fatty acids so as not to include any water. Filter these while entirely melted through any rapid filtering paper. Heat the filtered acids on a hot plate to 130°C. to remove traces of moisture and pour into the test tube. Fill the latter to a height of 57 mm from the bottom. The sample should not be held at 130°C. nor should it be reheated to this temperature more than once. If excessive moisture is present, the acids should be decanted after having stood for a few minutes refiltered and reheated. The acids must be thoroughly dry.

**Solidification of Fatty Acids**

1. Fill and adjust the temperature of the water bath. The temperature of the water should be 20°C. for all samples having titers of 35°C. or higher, and 15 to 20°C. below the titer point for all samples with titers below 35°C. The water level should be 1 cm above the sample level.
2. Place the test tube containing the fatty acids in the assembly as shown in the drawing. Insert the titer thermometer to the immersion mark and so that it will be equidistant from the sides of the tube.
3. Stir with the stirring rod in a vertical manner at the rate of 100 complete up and down motions per minute. The stirrer should move through a vertical distance of about 3.8 cm. The stirring may be performed by mechanical means by attaching a small motor with suitable reducing gears to the stirring rod. The agitation should be started while the temperature is at least 10°C. above the titer point.
4. Stir at the directed rate until the temperature remains constant for 30 seconds, or begins to rise in less than a 30-second interval. Discontinue stirring immediately and observe the increase in temperature. Report as the titer the highest point reached by the ther-

mometer. Duplicate determinations are normally expected to agree within 0.2°C.

**Notes**

1. Saponification is usually indicated by a change in the appearance of the mass which finally becomes homogeneous. Frequently saponification is indicated by a thickening or increase in the viscosity of the mass, which again thins out after the reaction is complete. Also, soap bubbles begin to form and rise from the sample after the reaction is complete. Familiarity with the test and the change which takes place during saponification usually enables one to determine the proper end point. Attention should be called to the fact that under some abnormal conditions, the above mentioned indications may not be reliable so that considerable care should be exercised to insure complete saponification. The committee has investigated a number of proposed tests for complete saponification but up to the present time, none has been found which is reliable under all circumstances.

2. Caustic soda cannot be substituted for caustic potash in the glycerol method. This method is quick and satisfactory. If an alternate method of preparing the fatty acids is desired, the following may be used: Saponify 50 grams of fat with 60 ml of a solution of 2 parts of methanol to 1 part of 50% sodium hydroxide. Dry, pulverize, and dissovle the soap in 1,000 ml of water in a porcelain dish and then decompose with 25 ml of 75% sulphuric acid. Boil the fatty acids until clear oil is formed and then collect and settle in a 150-ml beaker and filter into a 50-ml beaker. Heat to 130°C. as rapidly as possible with stirring, and transfer, after cooling somewhat, to the usual 1 by 4-inch (2.5-cm x 10-cm) titer tube. The method of taking the titer, including handling the thermometer, is the same as that described in the standard method.

## MODIFIED VILLAVECCHIA TEST (A.O.A.C.)

### Qualitative Detection of Sesame Oil

**Reagents**

1. C. P. Hydrochloric Acid (sp.gr.1.19).
2. Villavecchia Reagent. Add 2 ml of C.P. furfural to 100 ml of 95% ethyl alcohol.

**Procedure**

1. Mix 10 ml of the sample with an equal volume of the hydrochloric acid.
2. Add to this mixture 0.1 ml of the Villavecchia reagent and shake well for 15 seconds.
3. Note the color of the lower layer as soon as possible after the emulsion has broken. If no pink to crimson color appears, the test may be reported negative at that point. If any color is observed in the lower layer, add 10 ml of distilled water, shake again and observe the color as soon as separation has taken place. If the color persists, report the test as positive. If the color disappears, sesame oil is not present.

**Notes**

1. Furfural gives a violet color with hydrochloric acid, therefore, it is necessary to use the dilute solution specified. It is advisable to read the color as soon as possible so that the pink color, if present, may be observed before it is masked by the development of other non-characteristic colors.

2. It is advisable, with the Villavecchia test as with others of a similar nature, to run control samples using as standards oils of known composition.

3. The test is applicable to hydrogenated as well as unhydrogenated sesame oil although not with the same degree of sensitiveness. The committee has found that as little as 0.25% of sesame oil can be detected, but is of the opinion that this limit should be accepted with reservations. It is the considered judgment of the committee that there is every assurance that at least 0.5% of sesame oil is detectable and that the lower limit with respect to the fully hydrogenated oil is 1%.

4. The sensitivity of the Villavecchia test to small quantities of sesame oil may be improved by increasing the amount of Villavecchia reagent up to 1 ml. However, doing this hastens the rate of development as well as the amount of non-charactertistic colors that are formed. Therefore, if greater amounts of the reagent are used, relatively greater care must be taken in the observation of the final color.

## ACETYL AND HYDROXYL VALUES

### Acetyl and Hydroxyl Values

The acetyl value is defined as the number of milligrams of potassium hydroxide required for the neutralization of the acetic acid obtained on saponifying one gram of an acetylated fat or wax, and is a measure of the hydroxyl content of the sample. However, in using the Andre-Cook formula it must be remembered that the calculations are based on the weight of acetylated fat. The hydroxyl value may be defined as the number of milligrams of potassium hydroxide equivalent to the hydroxyl content of the sample. The hydroxyl value is based on the weight of the unacetylated fat.

**Reagents**

1. C. P. Freshly Distilled Acetic Anhydride, (99-100%).
2. 0.5 Normal Hydrochloric Acid.
3. Alcoholic Potassium Hydroxide: Dissolve 40 grams of pure potassium hydroxide in 1 liter of 95% redistilled ethyl alcohol. The alcohol should be redistilled from potassium hydroxide over which it has been standing for some time, or with which it has been boiled for some time, using a reflux condenser. The solution must be clear and the potassium hydroxide free from carbonates.

**Procedure**

a. Acetylation (A.O.A.C. Method): Boil 50 ml of the sample with 50 ml of acetic anhydride under a reflux condenser for 2 hours. Pour the mixture into 500 ml of distilled water in a beaker and boil for 15 minutes while bubbling a stream of carbon dioxide through the solution to prevent bumping. Siphon off the water, add 500 ml more water and boil again for 15 minutes. Repeat the siphonation and boil for 15 minutes with a third 500-ml portion of water. Allow the mixture to cool and separate the aqueous layer, which should be neutral to litmus. Transfer the acetylated sample to a separatory funnel and wash with two 200-ml portions of warm water. Separate as much of the water as possible, add 5 grains of anhydrous sodium sulphate to the acetylated sample, and let stand for 1 hour, agitating occasionally to assist the drying. Filter through a dry folded filter, preferably in an oven heated to 100-110°C., and keep the filtered sample in the oven until the sample is completely dry. The acetylated product should be clear and brilliant.

b. Determination of Saponification Number: Weigh accurately about 5 grams of the filtered sample into a 250-300 ml Erlenmeyer flask. Pipette 50 ml of the alcoholic potassium hydroxide solution into the flask, allowing the pipette to drain for a definite time. Connect the flask with an air condenser and boil until the fat is completely saponified (about 30 minutes). Cool and titrate with the 0.5 N hydrochloric acid, using phenolphthalein as an indicator. Calculate the saponi-

fication number (milligrams of potassium hydroxide required to saponify 1 gram of fat). Conduct one or two blank determinations, using the same pipette and draining for the same length of time as above. The saponification numbers are to be determined on both acetylated and unacetylated portions.

**Calculations**

1.

a = ml hydrochloric acid required to titrate blank.
b = ml hydrochloric acid required to titrate sample.

$$\text{Saponification number} = \frac{(a - b) \quad 28.05}{\text{Weight of sample}}$$

2.

S = Saponification number before acetylation.
S′ = Saponification number after acetylation.
A = Acetyl Value

$$A = \frac{S' - S}{1 - 0.00075S'}$$

H = Hydroxyl Value

$$H = \frac{S' - S}{1 - 0.00075S}$$

To calculate the Acetyl Value from the Hydroxyl Value.

$$A = \frac{H}{1 + 0.00075H}$$

**Notes**

1. Determination of acetyle value by the filtration method or distillation method is extremely difficult and unreliable. The Andre-Cook saponification method yields accurate and concordant results on fats and oils containing stable hydroxyl groups such as castor oil, and is by far the simplest and easiest to manipulate. For blown oils and others having unstable hydroxyl groups none of the methods commonly used yields concordant results.


REFERENCES

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## SMOKE, FLASH AND FIRE POINTS

### Applicable to Animal and Vegetable Oils and Fats

**Smoke:**

**Apparatus**

1. Cleveland Flash Cup; A.S.T.M. Designation: D 92 — 33.

The Cleveland open cup is made of brass and shall conform to the dimensional requirements prescribed in Table I. The beveled edge of the cup shall be at an angle of approximately 45°. There may be a fillet of approximately 0.397 cm in radius inside the bottom of the cup.

2. Heating Plate: A metal place, 0.635 cm in thickness, and 15.24 cm in width for supporting the flash cup. The plate shall be of brass cast iron, wrought iron or steel. In the center of the plate there shall be a plane depression 0.079 cm in depth, and of just sufficient diameter to fit the cup. There shall be a circular opening 5.50 cm in diameter, cut through the plate, centering with the center of the above mentioned depression. The plate shall be covered with a sheet of hard asbestos board ¼ inch in thickness, and of the same shape as the metal plate. There shall be cut in the center of the asbestos board a circular hole just fitting the cup. Heat may be supplied from any convenient source. The use of a gas burner, electric heater, or alcohol lamp is permitted, but under no circumstances are products of combustion or free flame allowed to come up around the cup. The source of heat shall be centered under the opening in the plate and shall be of a type that will not produce local superheating. If a flame heater is used, it may be protected from drafts or excessive radiation by any suitable type of shield, that does not project above the level of the upper surface of the asbestos board.

TABLE I.—DIMENSIONAL REQUIREMENTS FOR CLEVELAND OPEN FLASH CUP

| | Inches | | | Centimeters | | |
|---|---|---|---|---|---|---|
| | Min. | Normal | Max. | Min. | Normal | Max. |
| Inside diameter immediately below filling mark | 2-15/32 | 2- 1/2 | 2-17/32 | 6.27 | 6.35 | 6.43 |
| Outside diameter below flange | 2-21/32 | 2-11/16 | 2-23/32 | 6.75 | 6.83 | 6.91 |
| Inside height from center of bottom to rim | 1- 9/32 | 1- 5/16 | 1-11/32 | 3.25 | 3.33 | 3.41 |
| Thickness of bottom | 7/64 | 1/8 | 9/64 | 0.28 | 0.32 | 0.36 |
| Distance from rim to filling mark | 23/64 | 3/8 | 25/64 | 0.91 | 0.95 | 0.99 |
| Distance lower surface flange to bottom of cup | 1- 7/32 | 1- 1/4 | 1- 9/32 | 3.10 | 3.18 | 3.26 |
| Vertical distance upper surface flange to rim | 7/64 | 1/8 | 9/64 | 0.28 | 0.32 | 0.36 |
| Thickness of rim | 5/64 | 3/32 | 7/64 | 0.20 | 0.24 | 0.28 |
| Width of lower surface of flange | 9/16 | 19/32 | 5/8 | 1.43 | 1.51 | 1.59 |

3. Thermometer, A.S.T.M. Open Flash.

| | E 1 (11C — 39) | E 1 (11F — 39) |
|---|---|---|
| Liquid | Mercury | Mercury |
| Filling above Liquid | Nitrogen gas | Nitrogen gas |
| Temperature Range | —6 to +400°C. | +20 to 760°F. |
| Subdivisions | 2 C. | 5°F. |
| Total Length | 303 to 307 mm. | |
| Stem Diameter | 6.0 to 7.0 mm. | |
| Bulb Diameter | Not greater than stem | |
| Bulb Length | Not over 13 mm. | |
| Bottom of Bulb to Graduation Line at | —6°C. | +20°F. |
| Distance | 40 to 50 mm. | |
| Top of Thermometer to Graduation Line at | +400°C. | +760°F. |
| Distance | 30 to 45 mm. | |
| Top Finish | Red glass ring | Red glass ring |
| Longer Graduation Lines at each | 10°C. | 10°F. |
| Graduations Numbered at each Multiple of | 10°C. | 20°F. |
| Immersion | 25 mm. | 1 inch |
| Special Marking on Thermometer | 25 mm. Imm ASTM Open Flash | 1 in. Imm ASTM Open Flash |
| Scale Error at any point up to when standardized shall not exceed | 372°C. 1°C. | 700°F. 2.5°F. |
| Test for Permanency of Range | Subject to 360 to 370°C. for 24 hrs. | Subject to 680 to 700°F. for 24 hrs. |
| Marking on Case | ASTM Open Flash —6 to +400° | ASTM Open Flash +20 to +760°F. |
| Standardization | The thermometer shall be standardized at the ice point and at intervals of approximately 50°C. or 100°F. for 25 mm. or 1 in. immersion and for the following temperatures of the emergent mercury column: | |

| Thermometer Reading | Average Temperature of Emergent Mercury Column | Thermometer Reading | Average Temperature of Emergent Mercury Column |
|---|---|---|---|
| 100°C. | 44°C. | 200°F. | 110°F. |
| 150°C. | 54°C. | 300°F. | 129°F. |
| 200°C. | 64°C. | 400°F. | 150°F. |
| 250°C. | 77°C. | 500°F. | 175°F. |
| 300°C. | 91°C. | 600°F. | 205°F. |
| 350°C. | 108°C. | 700°F. | 240°F. |

4. Cabinet: This shall be constructed of the materials and in accordance with the dimensions indicated in Figure I.

**Procedure:**

1. Fill the cup with the sample so that the top of the meniscus is exactly at the filling line of the cup. Adjust the position of the apparatus so that the beam of light is directed across the center of the cup. Suspend the thermometer in the center of the dish with the bottom of the bulb approximately 0.635 cm (¼ in.) from the bottom of the cup.

2. Heat the sample rapidly to within approximately 75°F. of the smoke point. Thereafter regulate the flame so that the temperature of the sample increases at a rate of not less than 9 or more than 11 degrees Fahrenheit per minute. The smoke point is taken as the temperature at which the sample gives off a thin bluish smoke continuously.

**Notes:**

1. In some cases a slight puff of smoke appears before the sample begins to smoke continuously. This is to be disregarded.
2. It is essential that the cup be kept entirely clean and free from any substances which might cause smoke to appear ahead of the true smoke point.

## FLASH AND FIRE

**Apparatus:**

1. The apparatus including the thermometer for the flash and fire points is identical with that used for the smoke point except that the cabinet is not used.

**Procedure:**

1. The thermometer shall be suspended or held in a vertical position by any suitable device. The bottom of the bulb shall be approximately 0.635 cm (¼ in.) from the bottom of the cup, and above a point half way between the center and back of the cup.
2. The cup shall be filled with the sample to be tested in such a manner that the top of the meniscus is exactly at the filling line at room temperature.
3. The test flame shall be approximately 0.397 cm (⅛ in.) in diameter. The test flame shall be applied as the temperature read on the thermometer reaches each successive 5°F. mark. The flame shall pass in a straight line (or on the circumference of a circle having a radius of at least 15 cm) 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 approximately 1 sec.
4. The sample shall be heated at a rate not exceeding 30°F. rise per minute until a point is reached approximately 100°F. below the probable flash point of the sample. Thereafter the rate of heating shall be decreased and for at least the last 50°F. before the flash point is reached, the rate shall not be less than 9°F. nor more than 11°F. rise per minute.

**Flash Point**

5. The flash point shall be taken as the temperature read on 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.

**Fire Point**

6. After determining the flash point, the heating shall be continued at the specified rate of 9 to 11°F. per minute, and application of the test flame shall be made at the specified intervals until the oil ignites and continues to burn for a period of at least 5 sec. The method of application of the flame shall be the same as for the flash point. The temperature read at the time of the flame application which causes burning for a period of 5 sec. or more shall be recorded as the fire point.

**Notes**

1. The flash point and fire point tests shall be made in a room or compartment free from air drafts. The operator shall avoid breathing over the surface of the sample. It is desirable that the room or compartment be darkened sufficiently so that the flash may be readily discernible.

## MODIFIED GARDNER BREAK TEST
### Applicable to Crude Soybean Oil

**Reagents**

1. C. P. Hydrochloric Acid (sp. gr. 1.19*).
2. C. P. Carbon Tetrachloride.

**Apparatus**

1. Beaker—Electrolytic Type, capacity 180 ml.
2. Thermometer, A.S.T.M. Open Flash.

| | E 1 (11C — 39) | E 1 (11F — 39) |
|---|---|---|
| Liquid | Mercury | Mercury |
| Filling above Liquid | Nitrogen gas | Nitrogen gas |
| Temperature Range | —6 to +400°C. | +20 to 760°F. |
| Subdivisions | 2°C. | 5°F. |
| Total Length | 303 to 307 mm. | |
| Stem Diameter | 6.0 to 7.0 mm. | |
| Bulb Diameter | Not greater than stem | |
| Bulb Length | Not over 13 mm. | |
| Bottom of Bulb to Graduation Line at | —6°C. | +20°F. |
| Distance | 40 to 50 mm. | |
| Top of Thermometer to Graduation Line at | +400°C. | +760°F. |
| Distance | 30 to 45 mm. | |
| Top Finish | Red glass ring | Red glass ring |
| Longer Graduation Lines at each | 10°C. | 10°F. |
| Graduations Numbered at each Multiple of | 10°C. | 20°F. |
| Immersion | 25 mm. | 1 inch |
| Special Marking on Thermometer | 25 mm. Imm ASTM Open Flash | 1 in. Imm ASTM Open Flash |
| Scale Error at any point up to | 372°C. | 700°F. |
| When standardized shall not exceed | 1°C. | 2.5°F. |
| Test for Permanency of Range | Subject to 360 to 370°C. for 24 hrs. | Subject to 680 to 700°F. for 24 hrs. |
| Marking on Case | ASTM Open Flash -6 to +400° | ASTM Open Flash +20 to +760°F. |
| Standardization | The thermometer shall be standardized at the ice point and at intervals of approximately 50°C. or 100°F. for 25 mm. or 1 in. immersion and for the following temperatures of the emergent mercury column: | |

| Thermometer Reading | Average Temperature of Emergent Mercury Column | Thermometer Reading | Average Temperature of Emergent Mercury Column |
|---|---|---|---|
| 100°C. | 44°C. | 200°F. | 110°F. |
| 150°C. | 54°C. | 300°F. | 129°F. |
| 200°C. | 64°C. | 400°F. | 150°F. |
| 250°C. | 77°C. | 500°F. | 175°F. |
| 300°C. | 91°C. | 600°F. | 205°F. |
| 350°C. | 108°C. | 700°F. | 240°F. |

3. Porcelain Crucible, Bitumen Type A.S.T.M. Test D 4-27, 1938.
   - Diameter at top ..... 4.4 cms.
   - Diameter at bottom ..... 3.6 cms.
   - Depth ..... 2.5 cms.

**Procedure**

1. Heat a sufficient amount of the well mixed sample to 75°C. (167°F.) and maintain at this temperature for about 5 minutes so as to melt all fat soluble particles which may be present.
2. Weigh 25 gms. into the designated beaker, add three drops of the hydrochloric acid and stir in thoroughly. Suspend the thermometer in the center of the oil acid mixture so that the bulb is completely immersed but not touching the bottom of the beaker. Apply heat so that the temperature rise will 74 to 79.5°C. (165-175°F.) per minute.

Caution: Do not stir or otherwise disturb the sample after heating has begun. Heat to 289°C. (550°F.) and withdraw the flame. Cool to about 25°C. (77°F.) either in a water bath or in air.

3. After cooling add, while stirring, 50 ml of the carbon tetrachloride to dissolve the oil. Allow to stand for one hour, stirring at 15 minute intervals so as to be sure and remove any oil which may be occluded to the surface of or within the separated material.
4. The crucible should be prepared with medium fibre asbestos and the pad must be thick enough to

prevent breaking through. The mat should be washed with water and alcohol and dried to constant weight before using.

5. Filter the sample with the aid of a vacuum, using a policeman to remove any traces of break from the beaker. Wash the break on the crucible well, using not less than 5, 20-ml portions of carbon tetrachloride. The crucible should drain dry after each addition and the vacuum should be broken when each new portion is added. This precaution is advisable to insure removal of all traces of oil. Dry the crucible at 105°C. to constant weight and determine the weight of the residue on the crucible.

**Calculations**

1. % Break = Weight of Residue × 4.

## DETECTION OF FOREIGN FATS CONTAINING TRISTEARIN IN UNHYDROGENATED PORK FATS (MODIFIED A.O.A.C.)

### Applicable to the Detection of Beef Fat in Lard and Sometimes Referred to as the Boemer Number

**Reagents**

1. C. P. Acetone.
2. Alcoholic Potassium Hydroxide, 0.5 Normal.
3. Hydrochloric Acid, (1+1).

**Apparatus**

1. Centrifuge tube, 100-ml, or glass stoppered cylinder of the same capacity.
2. Melting point tubes: Capillary glass tubing, inside diameter 1 mm, thin wall, convenient length 5 to 8 cm.
3. Thermometer: Any convenient thermometer of suitable range but with 0.1 or 0.2°C. subdivision.

**Procedure**

*Crystallization of the Glycerides:*

1. Weigh 20 gms. of the filtered sample into the centrifuge tube or cylinder. Adjust the temperature of the acetone to 30°C. and use at this temperature throughout. Add the acetone to the sample to the 100-ml mark. Shake until a thorough mixture results and allow to stand for about 18 hours at a temperature of 30°C. plus or minus 2°C.

2. Place the tube in a suitable centrifuge, whirl for 5 minutes and pour off the supernatant liquid. If a centrifuge is not available a 100-ml cylinder is used in which case the supernatant liquid must be siphoned off.

3. Add another 20-ml portion of the acetone to the crystals, shake, centrifuge and decant or siphon as above.

4. Repeat operation (3), this time mixing well and pouring through a qualitative filter paper. Complete the transfer of the crystals and wash the contents of the filter paper with 5 small portions of acetone.

5. Apply a vacuum to remove as much of the acetone from the crystals as possible. Remove the paper from the funnel, place on a dry smooth surface and break up any lumps with a spatula. Allow to dry thoroughly. The temperature of the glycerides must not be elevated to the melting point in drying because this will materially influence the final results. After drying, comminute the mass and determine the melting point as directed below.

**Preparation of the Fatty Acids**

1. Remove a sufficient amount of the glycerides for the melting point determination and transfer the remainder into a 500-ml Erlenmeyer flask. Add 100 ml of the alcoholic potassium hydroxide. Place a small funnel in the neck of the flask to prevent loss on boiling and saponify by boiling for 1 hour.

2. Add 100 ml of water to the soap solution and evaporate on the steam bath to remove as much of the alcohol as possible. Transfer to a 500-ml separatory funnel. Add a sufficient amount of water to bring the total quantity used to about 250 ml. Neutralize with the hydrochloric acid (1 + 1) to separate the fatty acids using a slight excess. Add 75 ml of ethyl ether and shake.

3. Draw off the aqueous layer and wash the ether layer at least three times with water or until the washings are neutral to methyl orange. Withdraw the ethyl ether extract, filter, evaporate the ether on the steam bath and dry the fatty acids at 100°C. for a few minutes. Protect the fatty acids at all times from ammonia fumes.

**Determination of the Melting Point**

1. The glyceride melting point tubes must be sealed at one end before introducing the crystals. The crystals are inserted through the open end and forced down into the closed end with a small glass rod or wire.

2. The fatty acid melting point tubes are prepared by dipping the open tubes into the melted acids so that the sample stands about 1 cm. high in the tube. This end of the tube is then sealed in a gas flame.

3. The tubes containing the fatty acids are allowed to stand for one-half hour in ice water or held in a refrigerator overnight (4 to 10°C.).

4. Fasten the melting point tubes containing the fatty acids and the glyceride crystals to the thermometer by a rubber band or any other convenient means. They should be adjusted so that the section of the tubes containing the samples are adjacent to the bulb of the thermometer. Suspend the thermometer in a beaker of water (suitably agitated) so that the bottom of the bulb of the thermometer is about 3 cm below the level of the water. The temperature of the water at this time must be at least 10°C. below the melting point of the sample. Heat the water at such a rate that the temperature wi l increase at about 0.5°C. per minute. The melting point is that point at which the samples become clear and liquid. The melting point determination of the glycerides and fatty acids should be made at the same time.

**Calculations**

1. If the melting point of the glycerides plus twice the difference between the melting point of the glycerides and the melting point of the fatty acids, is less than 73°C. the lard is regarded as adulterated.

B. N. = Boemer Number
A. = Melting Point of the Glycerides
B. = Melting Point of the Fatty Acids

B. N. = A + 2 (A-B).

**Notes**

1. If the quantity of crystals obtained from 20 gms. is insufficient this amount may be increased providing the acetone is increased proportionately.

2. The committee's investigation has indicated that 10 per cent beef fat can be detected with certainty and many times amounts smaller than this, even down to 5 per cent, can be found.

3. Tolman and Robinson have pointed out that this method is not applicable to hydrogenated pork fats.

4. The results on cooperative samples have indicated that if the melting point of the glycerides alone is used as a criterion, some pure samples of lard might be reported as adulterated. Therefore, use of this value alone is not recommended.