this conclusion may be erroneous; and the method has tremendous possibilities for extending our knowledge of oil colors.

We hope we have given you some insight into the Color and Bleach Method of the American Oil Chemists' Society, the merits, the shortcomings, and what still needs to be done. Perhaps the most difficult part of arriving at proper color methods is the failure of people to understand just what color is and how it should be measured. This paper may not have taught you much about color, but it will have been worthwhile if it has interested you enough to dig up the facts so you can reach your own conclusions on a scientific basis.

Melting Point, Solidification, and Consistency

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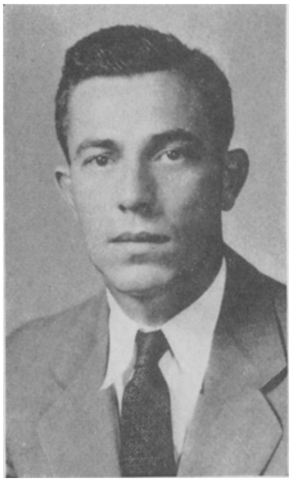
SINCE THE INTRODUCTION of the all-hydrogenated shortenings to replace the compound type, and the great increase in margarine use, methods for controlling the so-called plasticity or plastic range of the fats have been devised. The data obtained from melting points, solidification, and consistency tests are related in determining the plasticity or plastic range of the fats. Unfortunately very little published material is available however on many of these tests that are being used by the processors of fats today.



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Melting Points

The melting point is the temperature at which a solid assumes the liquid condition. With pure compounds the melting point is sharp and a well defined point. Inasmuch as fats and oils are a complex mixture of compounds, they do not have definite melting points but pass through a gradual softening before becoming completely liquid; therefore the melting point must be defined by the specific conditions of the method.

The Capillary Method (1) is essentially the method used by the organic chemist for determining the melting points of the pure organic compounds. In this method capillary tubes, 1 mm. inside diameter, are filled to a height of 10 mm. with the melted fat, the end is sealed, and the fat is allowed to stand at 40° to 50°F. for 16 hrs. They are then attached to the thermometer and placed in a bath which has been adjusted to 8° to 10°C. below the melting point. The bath is heated at the rate of 0.5°C. per minute. The melting point is taken as the temperature at which the fat becomes completely clear. This method is very unreliable, and even the same operator will have trouble checking his own results.

The Wiley melting point (2) is the most widely used and gives the most useful and reproducible data of any of the methods. The determination of melts by dilatometric methods gives more accurate results, but this method is not used extensively for melting points. In the Wiley melting point a disk of the fat 3/8 in. in diameter and 1/8 in. thick is solidified and chilled in a metal form for 2 hrs. or more. The disk is then suspended in an alcohol water bath of its own density and slowly heated while being stirred with a rotating thermometer. The melting point is taken as the temperature at which the fat disk becomes completely spherical.

Most laboratories and different companies have developed their own special apparatus and operational conditions in running the Wiley melting point. Various methods of determining the end-point are in use, such as the flap-over, tapping the sphere, which if it is at the end-point, will return to its spherical shape immediately, etc. Under carefully controlled conditions an analyst should be able to check results within plus or minus 0.2°C. In order to check with other laboratories each analyst must adhere closely to the standard method.

As an example of the accuracy of the Wiley method, a series of samples were run by seven different analysts in our laboratory. Of the seven analysts only one ran Wiley melts as a routine test, and with the other six it was only an occasional operation. The results are given in Table I.

As can be seen from the above table, the average difference between duplicate samples by the same analyst is 0.2°C. The difference between the highest value and lowest value is sample number one—0.6°C., sample number two—0.6°C., and sample number three—0.4°C. While these values may not be ideal, they are within a satisfactory and useful range for control laboratory purposes.

A series of samples sent out to subcommittee members by J. P. Hughes, chairman, were run in five different laboratories. Duplicates of each sample were run by two analysts on two different days, giving a total of eight determinations from each laboratory on each sample. From the data in Mr. Hughes' report the highest and lowest values reported for sample number one are 96.8°F. and 94.3°F., sample number two—106.0°F. and 103.1°F., sample number three—125.0°F. and 118.4°F. The range of 2.5°F. and 2.9°F. on samples number one and two are within a range that would be expected. The range of 6.6°F. on sample number three is explained by the fact that it is a formulation which exhibits the phenomenon of double melting points. One laboratory obtained good checks on sample number three at around 119°F. while another laboratory got equally as good checks at around 124°F. and the other three laboratories were between these two points. The checks obtained on all samples within a laboratory were very good

TABLE I

Comparison of Wiley Melting Points of Hydrogenated Vegetable Shortening by Different Analysts

Sample	Analyst No. 1		No. 2		No. 3		No. 4		No. 5		No. 6		No. 7	
No. 1	46.0°C.	46.2	45.9	46.0	46.4	46.4	45.6	45.8	46.2	46.2	46.4	46.2	45.6	45.8
No. 2	36.8°C.	37.0	36.8	36.8	37.0	37.2	36.6	36.8	37.0	37.0	37.0	37.2	36.8	36.6
No. 3	41.2°C.	41.2	41.4	41.2	41.4	41.2	41.2	41.0	41.4	41.2	41.4	41.4	41.0	41.2

with the average variation of four duplicate samples being in the range of 0.3°F. to 0.8°F. for all laboratories.

There are various other softening and slipping point methods, such as A.O.C.S. method Cc-4-25, which is used to a limited extent.

For work where an exact melting point is desired, the determination should be made by dilatometry. For most control work the accuracy of the Wiley melting point would be sufficient. The Wiley melting point in conjunction with the refractive index has been used for many years and is still widely used as a control for hydrogenation. For control purposes the long chilling can be eliminated, and the sample chilled quickly and run immediately. If the conditions of hydrogenation of a specific oil are established and closely controlled, the Wiley melting point can be a very useful and quick control for the hydrogenation process. The Wiley melting point is a very reliable control in the blending of oils.

Solidification Points

The titer or solidification point of the mixed fatty acids is the oldest of the fat characteristics used for control purposes. It is still used widely in the inedible fat industry but is seldom used in the edible fat industry. It furnishes no useful indication of the consistency of a plastic fat.

The titer test (3) determines the solidification point of the fatty acids. A titer tube is filled to the 57-mm. mark with dried fatty acids and suspended in an air bath, which in turn is surrounded by a water bath of 15° to 20°C. below the titer. The sample is stirred until the temperature begins to rise or remains constant for 30 seconds, after which the stirring is stopped and the end-point is taken as the maximum temperature to which the fat is carried by the heat of crystallization.

The Setting or Congeal Point gives the solidification point of the fat itself rather than the separated mixed fatty acids. It is used quite extensively on both shortening and margarine oils. The methods used are not standardized. The following method used in our laboratory is typical of the methods in use.

A sample of fat is dried at 130°C., cooled to 60°C., and a titer tube is filled three-fourths full of the sample. The sample is cooled rapidly (9°C. per minute) to within 1 to 2°C. of the estimated setting point by immersing it periodically in a 10°C. water bath while stirring it by hand. The outside of the titer tube is dried quickly and placed in the air bath of the Titer Tester, which is surrounded by a water bath 5°C. below the estimated setting point. The titer thermometer is centered in the tube with the bulb about 1 in. from the bottom and the mechanical agitator is started. The agitation is continued until the temperature stops falling and remains constant for 30 seconds. The highest point of temperature rise is taken as the setting point. The rise should be at least 0.8°C., and if it is less than 0.8°C., the determination should be repeated after adjusting the setting point bath temperature to give the proper cooling rate and degree of super-cooling, which is necessary to make the fat crystallize rapidly enough to show a good rise in temperature, when agitation is stopped.

Variation of the congeal or setting point, such as the Cloud Point test or the "C" number test, are used to a limited extent. In the Cloud Point test a sample of fat is stirred while being cooled, and a light beam of known intensity is passed through the fat falling on a photocell and the output read on a microammeter. In the "C" number test a tube of melted fat is stirred up and down by means of a dasher type stirrer while being cooled. When the fat solidifies to a point that the tube rises with the plunger and breaks the electrical contact, the temperature at this point is taken as the "C" number.

The values from congeal or setting points are more closely related to consistency than the melting points, provided the approximate selectivity of hydrogenation is known. For checking results the specific conditions of the congeal or setting point must be defined.

Congeal or setting points probably have more importance in the margarine type of oil. The congeal point and the Wiley melting point are kept as close together as possible with the spread between these being approximately 10°C. The congeal point is also used as one of the values for evaluating catalyst performance.

Consistency Measurements

The property of plasticity or consistency is regarded as essential to the performance of certain classes of fats. The class of butter, margarine, or table spreads depends on the consistency of the fat in its ability to be spread on bread. Other products in which consistency is highly important are the shortenings used in baked products.

The factor most directly influencing the consistency of a fat is the proportion of material in the solid phase. Fats have sufficient body to hold their shape with a solid content of approximately 5% by weight and become hard and brittle when the solid content is around 50%. A fat will have the best consistency for mixing and working within a solid content of about 15 to 25%. A small change in the solid content will have a great effect on consistency.

Many of the methods for melting point, congeal point, etc., are useful only because they yield values that can be correlated with the consistency of the fat. Probably the first method used for checking consistency was thrusting the finger into the product and squeezing it through the fingers. There is no standardization of devices or methods for measuring consistency, and practically every manufacturer of shortening has his own method.

Probably the most widely used instrument for consistency measurement is the penetrometer, either a mi-

TABLE II

Effect of Chilling Temperatures on Penetration Values of Hydrogenated Vegetable Oils

Sample	Penetrations (3 cups, 3 penetrations each) Chilling Temperature 12°F.									Average Penetration
1	77	78	82	77	77	78	75	79	82	78
2	66	70	71	66	67	69	67	68	70	68
3	35	36	36	36	39	34	36	36	35	36
4	37	36	40	35	36	37	37	37	34	37
5	27	29	30	27	29	30	28	28	29	29
6	37	37	36	34	37	39	37	38	39	37
	Chilling Temperature 7°F.									
1	77	75	76	75	77	76	75	77	74	76
2	66	70	67	64	65	69	67	64	70	67
3	36	35	35	36	33	37	35	37	39	36
4	34	35	39	35	34	36	34	35	37	35
5	29	27	31	28	29	28	27	29	28	28
6	37	38	39	33	38	39	37	37	39	38
	Chilling Temperature —3°F.									
1	80	81	77	79	80	81	80	76	74	79
2	70	69	66	69	66	72	69	73	68	69
3	37	37	38	37	36	35	36	33	35	36
4	34	36	38	34	35	37	33	35	34	35
5	25	27	29	31	29	27	31	28	28	28
6	33	35	36	38	37	40	34	35	33	36

cropenetrometer or an A.S.T.M. grease penetrometer. Feuge and Bailey (4) modified and standardized the micropenetrometer for consistency measurements.

Clardy, Pohle, and Mehlenbacher (5) describe a shortening consistometer which indicates the relative pressure necessary to force the product through an opening in the end of a plunger. The chief advantage they claim for the instrument is that it might be applied directly to the original container.

The method used in our laboratory is probably typical of most of the penetrometer tests used. The Precision Universtal model penetrometer is used in our laboratory. The needle used is our own design 24-g. needle. Our method is used on both the melted fat and the packaged products. In the melted penetrations regular 1-oz. tin ointment cups are filled with the melted fat. The cups are placed in a metal tray on the floor of the refrigerator freezing compartment with the control set to hold the air temperature in the compartment at 15°F. ± 5°F. The cups of fat are left in the freezing compartment 1 hr. The cups are then transferred to the desired constant temperature baths for 1 hr., after which the cups are transferred to the penetrometer stand and four penetrations are taken as quickly as possible about midway from the center to the rim. The penetration is made by raising the tip of the needle 100 penetrometer scale units (10 millimeters) above the fat surface and releasing it for 1 second to give an instantaneous plunge. The penetration of the fat is recorded as the average of the four penetrometer readings.

TABLE III

Penetration Values of Different Laboratories

Sample		Lab. No. 1	Lab. No. 2	Lab. No. 3	Lab. No. 4
No. 1	50°F.	48	50	45	56
	60°F.	64	64	60	65
	70°F.	87	83	79	82
	80°F.	103	94	97	94
	90°F.	144	138	124	128
No. 2	50°F.	60	54	56	58
	60°F.	80	75	75	78
	70°F.	110	111	97	104
	80°F.	122	129	134	138
	90°F.	165	163	200	170

The package penetrations are run by the same procedure except that the cups are taken from the package and placed directly in the desired constant temperature bath. In filling the package penetration samples, holes are drilled in the bottom of the 1-oz. cups, and the cups are forced down into the packaged product. The cups are removed by cutting around them with a spatula and removing a cone of the product, then cutting it off at the edge of the cup to prevent disturbing the product as much as possible.

A series of tests on the effect of the temperature at which the samples are set up in our penetration method was made. Samples were chilled for 1 hr. in a tray surrounded by an alcohol-water mixture at the following temperatures: 12°F., 7°F., and —3°F. The results are given in Table II. A review of Table II results shows good reproducibility and agreement of results at all three temperatures. To show the reproducibility between laboratories, Table III shows penetration results on check samples sent out to our laboratories.

While the time required for the penetration test makes it unacceptable for a quick control for hydrogenation, it is still quite valuable as a final control for hydrogenation. From the penetration values of the previous batches, conditions can be adjusted to correct any variation from the specification. In the blending of oils, where the time element is not as important, penetration values along with solid content and Wiley melting points are our primary controls. The package penetrations are used to control the plasticizing and tempering of the finished products. From previous experiences, conditions of plasticizing and tempering can be set up which will hold penetration values within narrow limits.

Summary

There are probably as many methods or variations of methods for melting points, solidification, and consistency as there are producers in the fats and oil industry. I have tried to review the more important ones. The interpretation of results and importance attached to each test will vary widely from producer to producer. My presentation and interpretation have been primarily of methods used by our company and related to the shortening field. In our experience the data obtained from Wiley Melting Points, Penetration Values, and % solids from dilatations are the primary controls for the consistency of our products.

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