no recommendation can be offered for the reasons previously stated.

On several occasions complaints have been heard that our present hull analysis is inaccurate and presents a false picture. With this in mind, the committee compared the present official method with one in which the hulls were treated in an identical manner as the seed; that is, dried, fumed, ground, and both oil and ammonia determined. The results proved conclusively the weakness of the present method. Oil and ammonia results were consistently from two to three times as great as under the present method. Moreover, these losses check very closely mill operations and the method has the added advantage that the value of both the cake and oil lost in the hulls above the theoretical, can be easily shown. The expense of the test is obviously about that of a seed analysis and while agreeing to its superiority, the committee did not wish to recommend it without further study. It is our intention to present this problem to the Crude Mills Operations committee for further study in order to determine if, in the opinion of the mills themselves, the added advantages are worth the additional cost of the test.

The other test studied this season was that of the lint determination of seed. This is easily the most inaccurate test now described in our methods. While we can not hope for too great accuracy in this test, due to the difficulty in getting representative samples, nevertheless preliminary work has shown that a great improvement may be made. Our study included both the method used by Dr. Freyer, which is an adoption of the present test, and one in which a five gram sample is fumed, the lint removed by rubbing, and dried. The lint is found by subtracting the moisture loss from the total loss in weight. This method has the disadvantage of using a small sample and the advantage of no loss of hull. Additional comparative tests are necessary before either can be recommended.

The committee feels that while we can make no definite recommendations, we have at least raised several pertinent questions for the consideration of the Society, and which we hope will assist the committee following us to make some material improvements in our methods of analysis.

WAXES IN THE CANDLE INDUSTRY

By L. W. GELLER*

DURING recent years much work has been done to find a synthetic wax or resin capable of hardening and improving the various grades of paraffin for the production of a better and less expensive candle wax.

For a better understanding of the problem, commercial candles can be divided into five principal groups:

1. The first group contains all the candles which are consumed in glasses or in jars made from any kind of fireproof plastics.
2. The second group consists of the colored and decorative candles.
3. The third group contains the beeswax or religious candles.
4. The fourth contains all the white household molded candles.
5. In this group are the small birthday and taper candles.

For the production of candles there are two waxes in common use—paraffin and beeswax. Stearic acid, the commercial form of which is a mixture of palmitic and stearic acids, is an important substance for the candle manufacturing. This fatty acid, while not a wax, behaves like a candle wax as far as the burning property is concerned, being consumed like paraffin or beeswax.

By mixing the waxes with other materials, attempts have been made to improve the quality of the candle waxes. These added materials include:

1. Natural hard waxes, such as carnauba and montan wax.
2. Synthetic waxes, such as the IG-waxes, or waxes made from palmityl (cetyl), stearyl, oleyl alcohols, or their homologues and derivatives.
3. Substances related to fatty acids, especially palmitic, stearic and oleic acids, as follows:
    a. Amido fatty acids and alkyl or aryl amido fatty acids, such as Stearic amid ($C_{17}H_{35}$-CO.NH_2), stearic and palmitic anilid ($C_{17}H_{35}$ CO-NH.C_6H_5) ($C_{15}H_{31}$CO-NH.C_6H_5), oleic benzidid ($C_{17}H_{33}$CO-NH.C_6H_4-C_6H_4.NH-CO $C_{17}H_{33}$), etc.
    b. High molecular ketones, obtained from stearic, palmitic, oleic and other fatty acids. Such are stearone ($[C_{17}H_{35}]_2CO$), palmitone, oleone ($[C_{17}H_{33}]_2CO$), etc.
4. Hydrogenated oils and **fats.**
5. Substances foreign to the properties of candle wax or stearic acid, such as benzo-naphthol.[1]
6. Coating substances, consisting of synthetic and natural resin.[2]

All these materials have been used with the purpose of hardening and improving the dripping property of the candles.

Before going into further details we must consider what the hardening of a candle wax means. Generally, it is believed that the hardening of a candle wax is achieved by raising its melting point. In fact, that is not the case, especially with the candle waxes. To the waxes belongs the property of softening before they reach their melting point; and often the difference between the softening and melting point is equal to 10-20° C.

It is clear that for a candle the softening point, and not the melting point, is the property which determines the degree of hardness. Accordingly, a hardening agent for a candle wax will be an addition agent which raises the softening point of the original wax and, correspondingly, the bending point of a candle made from it.

The melting point may be determined by bringing a sample of about 30 grams of melted wax in a test tube of 4 inches in length and 1 inch of diameter. The tube is then closed by a cork in which is inserted a thermometer subdivided into fifths of a degree C. The tube is brought into an empty bottle of about 5 inches in height and 3 inches in diameter, closed by a cork provided

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with an opening in the center, into which fits the test tube. The apparatus with melted wax is then shaken until the wax in the tube starts to solidify by showing a visible turbidity. At this point the temperature is taken and the highest degree constant for 30 seconds is the melting point.

The bending point is the temperature at which a candle softens and starts to bend. For this purpose the candle to be tested is kept in a thermostat for about seven hours at a constant temperature.

As already mentioned, the following materials have been tried as hardening agents for candle waxes: stearic acid, montan wax, IG-S wax, high molecular alcohol waxes, hydrogenated oils and fats, synthetic and natural resins.

Out of all these substances only the stearic acid can be called a real hardening agent for the candle waxes. It does not imperil and destroy the burning qualities of the candle by producing smoke, or by burning wet, or by dripping, and even improves their burning quality. The stearic acid due to its crystalline form, though it raises the softening point, will lower the melting point of paraffin.[3]

Wax mixture.	Melting point, ° F.
Paraffin 100%	125.6 °
Paraffin 98% + stearic acid 2%..	125.2 °
Paraffin 90% + stearic acid 10%.	122.7 °
Paraffin 86% + stearic acid 14%.	122.0 °
Paraffin 80% + stearic acid 20%.	121.1 °
Paraffin 75% + stearic acid 25%.	120.6 °

Carnauba wax acts differently; it raises the melting point of the wax but has no effect on its softening point or on the bending point of the candle. One of the reasons why it does not affect the softening point is because it cannot be added in a larger quantity than 2% to any candle stock. Any amount of carnauba wax exceeding this quantity will make the candle smoke, burn wet and burn with too large a flame. All of these effects are serious defects.

Wax mixture.	Melting point, ° F.
Paraffin 100%	125.6 °
Paraffin 98% + carnauba wax 2%.	134.6 °
	..f..

Montan wax has no effect on the softening or bending point of the stock. However, it is often added to give to the candle an opaque appearance. Due to its objectionable odor and, as in the case of carnauba wax, because it produces smoky flame, the added amount should not exceed 2%.

During the last five years the hydrogenated oils and fats have been successfully introduced on the market especially in connection with scale paraffin wax.[4]

For candle manufacturing there are in common use three different grades of paraffin:

1. Scale wax, called crude paraffin wax.
2. Paraffin wax of melting point 125°, and
3. Paraffin wax of melting point 130-135.°

Scale paraffin wax is used only in candles which are consumed in glasses or in vessels made from fireproof plastics. These candles, being wide in diameter (1¼-4 inches) and relatively low in height (½-7 inches), will not bend in warm weather but will soften and, of course, will lose more or less their original shape. To avoid to some degree this deficiency, stearic acid or a mixture of harder paraffin and stearic acid is added. Better results have been obtained by substituting for part or all of the stearic acid hydrogenated oils or fats which are of high melting point. The hydrogenated products mix well with paraffin and, during the burning in glasses, do not separate. The results obtained with carnauba wax, montan wax, IG-S wax and high molecular fatty alcohol waxes are not satisfactory. During the burning they separate out of the paraffin mixture, producing a heavy crust on the wall of the glasses, especially during cold weather, obstructing the flame or even breaking the glasses; besides, they reduce the burning quality of the candles considerably.

These kinds of candles are very much in use for churches, camps, hospitals, homes, and lately as perfumed and disinfectant candles. Their size corresponds to the time to be burned, i.e., from two hours to eleven days. The time is controlled not only by the size of the candle but also by the size and chemical treatment of the wick.

Not the same can be said about the second group, the colored candles. They are, with some exceptions, hand-made, and then they are dipped in colored hot wax. Here the wax composition of the inner candle is different from that of the coating or dipping wax. One of the best and cheapest waxes for the inner candle is a high melting point paraffin containing a small amount of stearic acid. Other waxes than stearic acid do not improve the quality of the candle. Montan and carnauba burn wet and smoky, IG-S wax and high molecular fatty alcohol waxes burn wet and do not show the expected improvement; besides, they are not white and are too expensive. The dipping wax, in which the dyestuff is dissolved, is of a different composition from that of the inner candle. The paraffin predominates and the stearic acid is present in such quantity as is necessary to dissolve the dyestuffs. The brightness of the color is accentuated by the softness of the beeswax in combination with a very small amount of hard wax.

In the third group there are the beeswax candles. Being for religious purposes, they must contain a certain amount of pure beeswax, e.g., 51%, 60%, 75%, etc. The rest of materials to 100% consists of paraffin and stearic acid. These candles are generally hand-made but they also can be molded.[5] The hand-made candles cannot contain much of a hardening agent on account of cracking during manufacturing, but the molded candles can be hardened successfully by adding to the mixture a small amount of difficultly saponifiable carnauba wax esters.[6] This wax in combination with a high melting paraffin should improve also the wet burning and dripping of beeswax candles. Other waxes or resins, natural or synthetic, do not improve the qualities of the beeswax candles.

The household candles are made from paraffin, alone or in admixture with stearic acid, which may be added to 60-70%. They are molded candles.

The fifth group contains the birthday candles and tapers, which consist of pure paraffin in admixture with a small amount of stearic acid.

SUMMARY

1. The waxes in use for candles consist of paraffin and, in special cases, of beeswax.
2. Stearic acid is the only known hardening agent for paraffin. It raises the bending (softening) point and lowers the melting point. It can be used in any quantity without impairing the burning quality of the candle.
3. No other wax, natural or synthetic, can be substituted for stearic acid as a hardening agent for candle stock. Other waxes generally injure the burning quality of the candle or fail to produce any improvement, being at the same time more expensive than stearic acid.
4. The hydrogenated oils and fats serve as hardening agents for paraffin, especially for scale paraffin wax, and are used chiefly for candles which are consumed in glasses.
5. Synthetic or natural resins can be used in form of coatings only

and are used chiefly for decorative candles.

6. No known synthetic hardening agent for candle wax can be satisfactorily substituted for stearic acid, even in such a mixture as 95% paraffin (MP 135°) and 5% stearic acid.

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(2) Jaeger, A., to Selden Co. U. S. Patent 1959164. (May 15, 1934.)
(6) Luedecke, Wachse, page 40.
(3) Luedecke, Wachse, page 108.
(1) Seifensieder Zeitung, Vol. 44, 1933, page 791.
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REPORT OF THE OLIVE OIL COMMITTEE

By M. F. LAURO, Chairman

PRESENTED AT THE 26TH ANNUAL MEETING, AMERICAN OIL CHEMISTS' SOCIETY, MAY 23-24, 1935

As may be remembered, this committee undertook the drafting of certain specifications for Olive Oil and for Olive Oil Foots. We had a precedent for this in the work of the American Society for Testing Materials, in establishing values for linseed, soya bean and perilla oils. We had a stimulus for this in the increasing demand by the trade in these commodities for more definite and modern standards. At present neither text nor authority supply satisfactorily the needed information.

As a start, the following preliminary classification of values was sent around to each member for his comment and criticism.

For SPANISH, GREEK AND ITALIAN OLIVE OILS (comprising by far the largest group of oils being imported into this country):
oils being imported into this country):

	Normal or Recommended Limits	Outside Limits
Specific gravity at 15.5 C.	0.915 to 0.920	......
Iodine value (Wijs)	82 to 86	79 to 90
Saponification value	189 to 195	188 to 196
Titer of the fatty acids	17 to 26 C.	......
Unsaponifiable matter (FAC)	Maximum 1.5%	......
Crismer test	69 to 71 C. plus or minus 0.5	
Halphen, Villavecchia and Bellier tests to be negative.		
Coin or Silver Benzoate tests and the Beilstein test to determine extracted oils.		
For ALL FOOT OR SULPHUR OILS (irrespective of origin):		
Iodine value (Wijs)	80 to 86	77 to 90
Saponification value	188 to 195	185 to 200
Titer of the fatty acids	16 to 23	......
Unsaponifiable matter (FAC)	Maximum 2.25%	......
Ash content	Maximum 0.20%	0.25%
	to consist chiefly of iron oxide and only traces of lime, etc.	

Replies were received from time to time. And since the publication in the November, 1934, issue of our OIL AND SOAP of the committee report directing attention to its work and inviting comment from the trade as well as from chemists, I have had also some very interesting letters from dealers and sources outside the membership.

Dr. Jamieson states he would consider 90 as a proper outside limit for the iodine value of Italian and Spanish olive oils exported to this country, but for Tunisian and Dalamatian oils the limit should be 94. He also directs attention to the fact that the majority of iodine values given in the literature were determined either by the old Hubl method or that of Hanus, that the Wijs figures are some 2 to 4 points higher at times, and that therefore the values which I gave may be somewhat low. Agreeing with him that 86 as a normal upper limit for olive oil is too low, I would change this to 88. There is no question that the matter of method for obtaining iodine numbers is one to take into account in arriving at our limits. On page 93 of his text on "Vegetable Fats and Oils" we have the analysis by him of some authentic samples representing certain types of olive oils, as follows:

	Californian	Italian Bitonto	Spanish Borjas	Tunisian Sousse
Iodine No. (H)	85.1	84.4	83.7	86.0
Sap. No.	190.6	190.6	192.4	193.6
Unsap. matter	1.0%	1.1%	0.8%	0.8%
Acid value	1.5	1.8	1.8	1.9
Sp. Gr. 25/25 C.	0.9119	0.9120	0.9116	0.9131

Mr. Dickhart gives the interesting information that he had outlined rules, some time ago, for use by the Olive Oil Association in transactions in olive oil products. He would classify olive oil by reviving the four grades, namely, Edible, Commercial, Technical and Olive Oil Foots. The edible grade would follow the U. S. Pharmacopeia requirements, except that the iodine value be determined by the Wijs method and be not less than 82 nor more than 85, that the specific gravity at 15.5 C. be from 0.914 to 0.919, the titer 17 to 26, coin, etc., tests negative. For commercial grades, moisture and impurities must not be over 2 per cent and free fatty acidity as oleic must be under 5 per cent. The technical oils must show either a positive coin test or the copper wire (Beilstein) test, to indicate that they are extracted olive oils. And olive oil foots must be oil extracted from olives or the pulp of same with carbon bisulphide or other (halogenated) solvent, and to grade "Prime Green," must be of a natural green color, not dyed, and must saponify with 20 degrees Beaumé lye to a soap of a definitely green coloration.

Dr. August Gill gave his approval to the proposal to draw up specifications and considered the limits first tendered as being reasonable ones.

M. L. Sheely supplied data representing 40 samples of olive oil