

Oct. 18, 1955

D. C. ROBERSON ET AL

2,721,165

CONTROL OF WAX DEOILING

Filed July 9, 1951

3 Sheets-Sheet 1

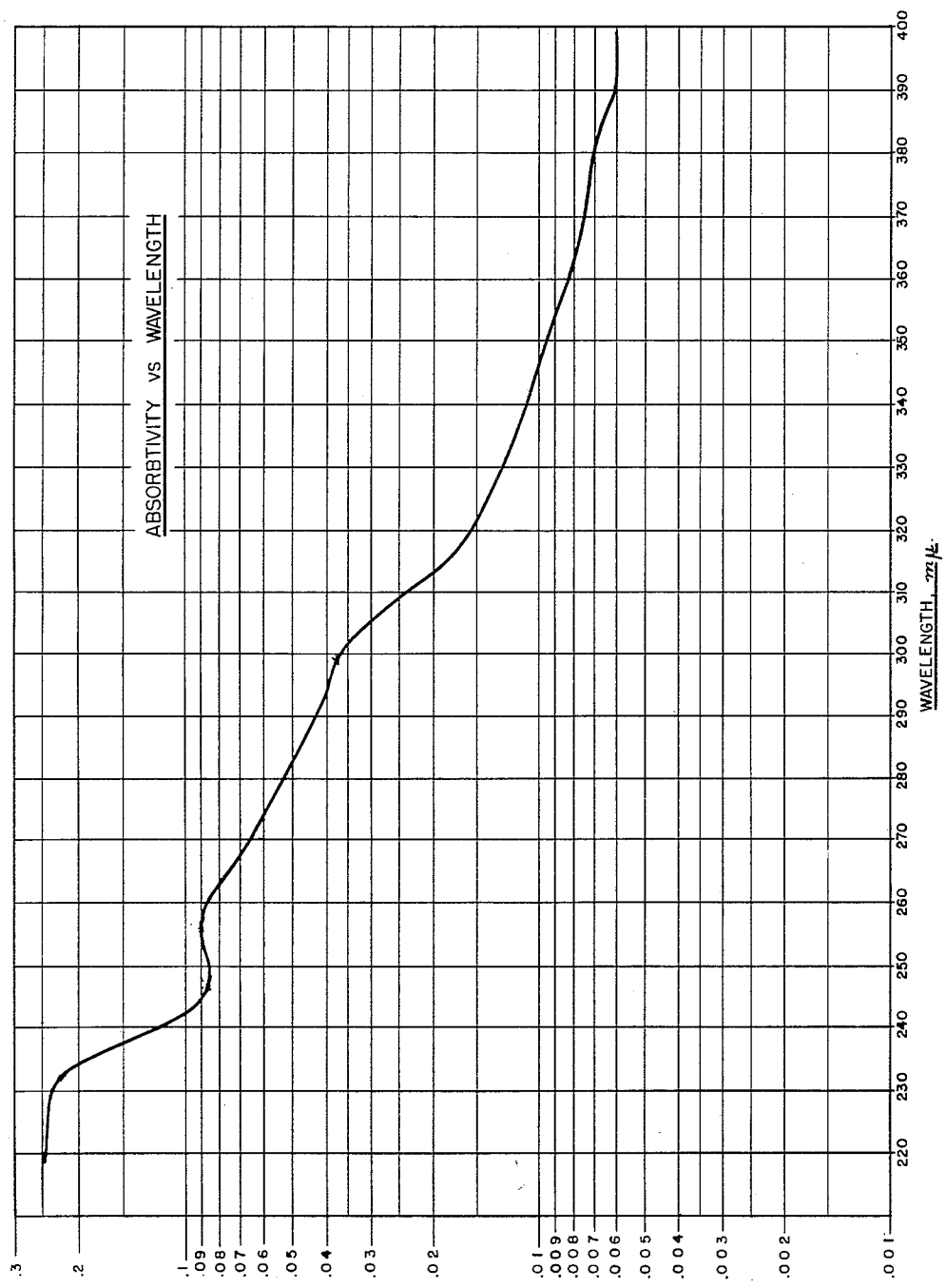


FIG. 1.

ABSORBANCE, A

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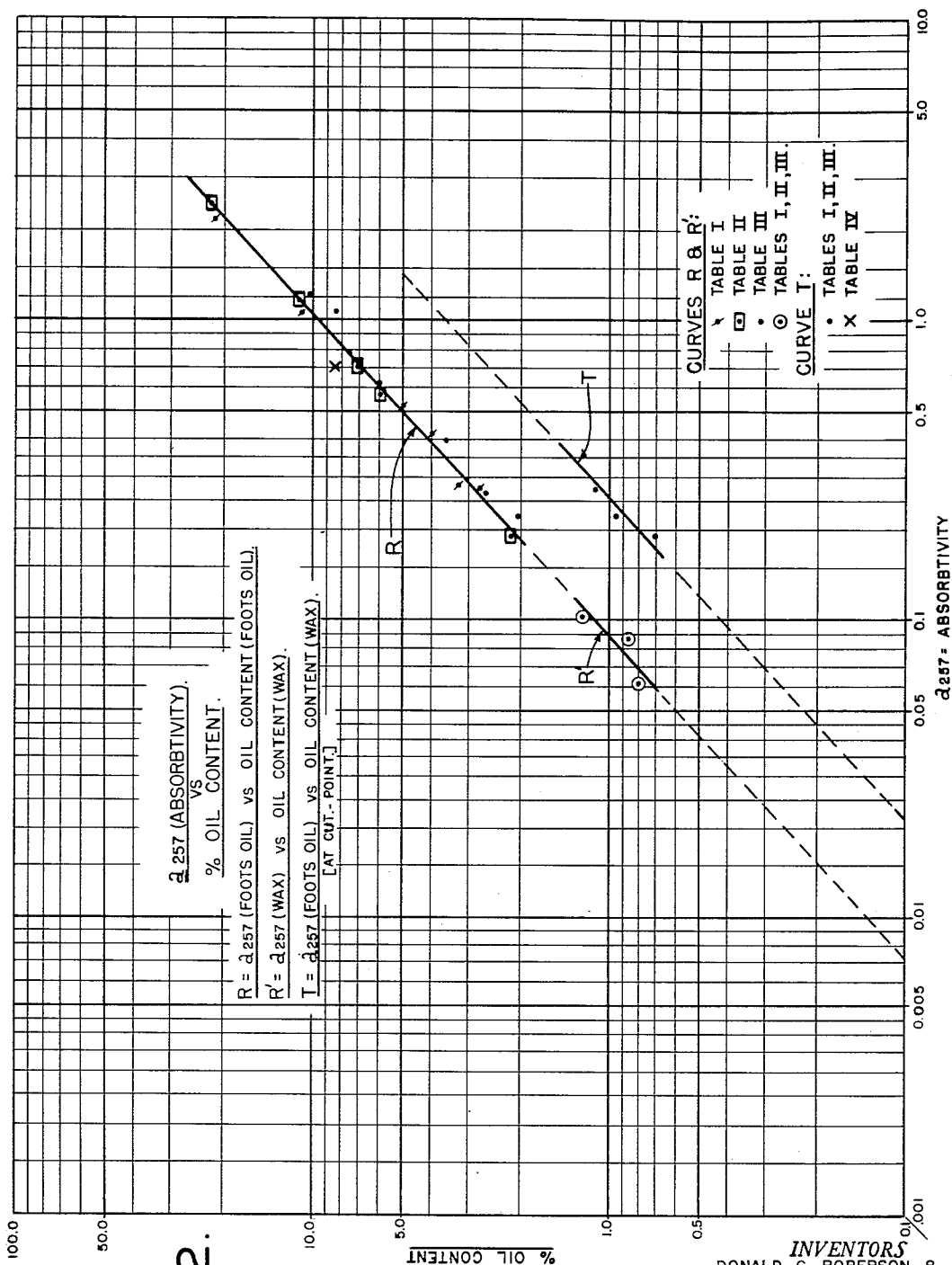


FIG. 2.

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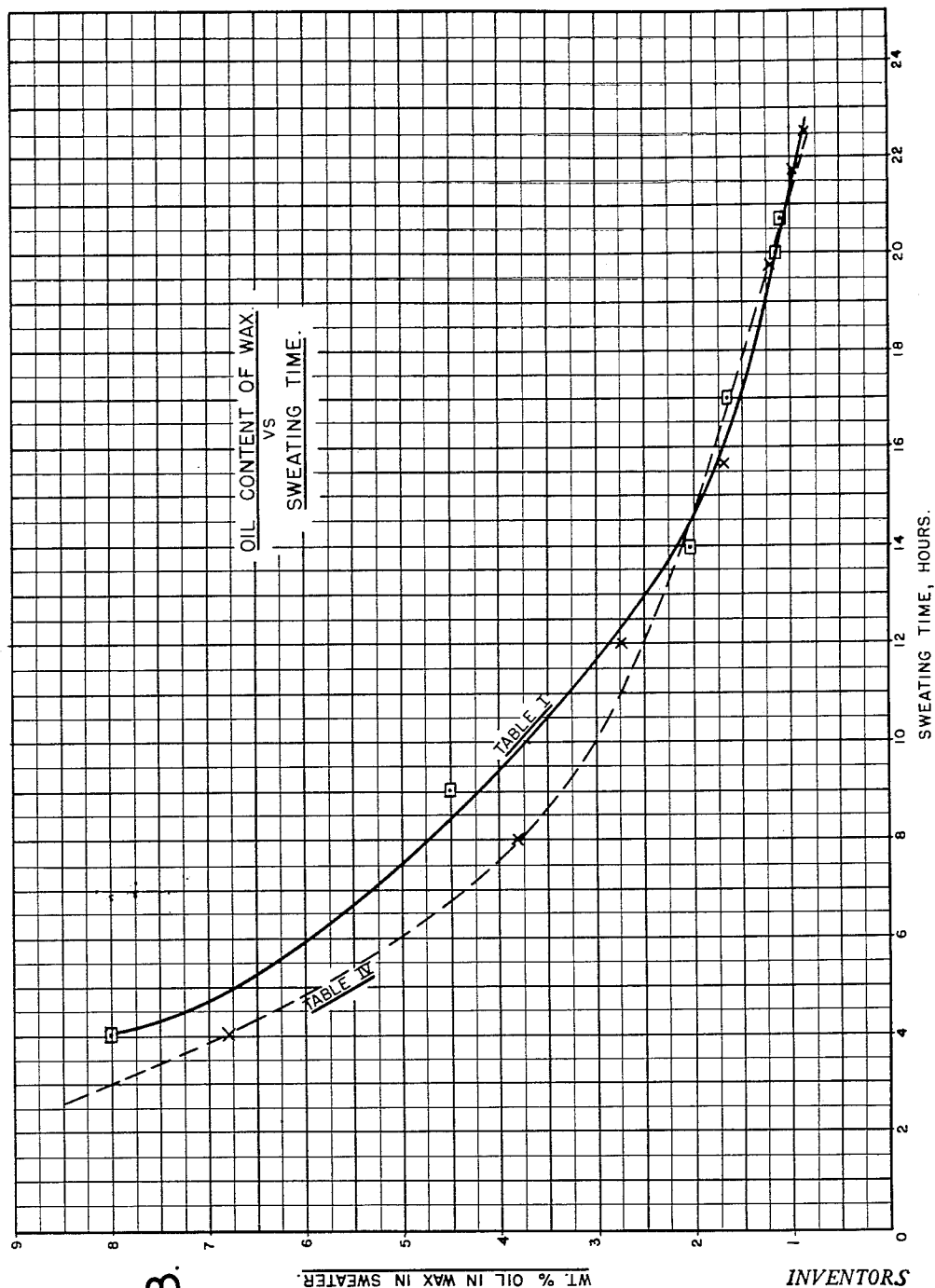


FIG. 3.

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1

2,721,165

## CONTROL OF WAX DEOILING

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Application July 9, 1951, Serial No. 235,842

2 Claims. (Cl. 196—20)

The present invention relates to a method of determining at any time in the de-oiling of wax the oil content of the wax and thereby of controlling the de-oiling of wax in a manner to assure obtaining a finished wax product having a predetermined low oil content.

De-oiling of oil-wax mixtures is generally accomplished in either of two conventional manners. The most common method is known as "sweating" in which oil is removed or "sweated" from a batch of the crude wax stock by slowly raising the temperature of the wax in a container and draining off the exuded oil until only a desired amount of oil remains in the wax. Another method is the removal of undesirable oil by means of solvent extraction. While the present process is applicable to either type of operation, attention is directed foremost to the sweating type of operation since it presents particular problems of obtaining uniform samples of wax for observance in determining the point at which to terminate the de-oiling treatment.

Wax sweating operations have generally been carried out in one of two types of apparatus. These are the pan and the tank sweaters. In each of these types, the method is substantially the same and involves chilling a warm molten liquid oil-wax mixture to a semi-solid state and then slowly raising the temperature, during which time a liquid which is known as foots oil, drains off the stock in the sweater. The desired wax product remains in the sweater.

In the present discussion and claims the "oil content" is determined by the standard ASTM test method D721-49 described in the American Society for Testing Materials Handbook (1949), page 981, in which the "oil content" is defined as the amount of a sample which is fluid at  $-25^{\circ}$  F. when in solution with methylethylketone.

It requires an experienced operator to know at what time a sweating operation should be terminated and the wax removed from the sweater. It is impracticable to remove and analyze samples of the wax remaining in the sweater at intervals during the process, not only because of the inaccessibility thereof but also because of a sample taken from any particular location in the sweater is not necessarily representative of the remainder of the wax. It is, therefore, the usual practice to analyze successive samples of the foots oil which drains from the wax.

One method employed by experienced operators is that of pouring a sample of the foots oil into a melting-point wax bath, allowing it to cool and solidify so that a cake of wax about  $7'' \times 1\frac{1}{2}'' \times 1\frac{1}{2}''$  is formed, and examining the cake by transmitted light. If the operator observes a greenish tinge, it indicates to him that the wax in the sweater requires further sweating. Experience has taught operators that the absence of the greenish tinge is indicative of a product which possesses desirable color and color stability and also has or approaches the proper melting point. The absence of a tinge also indicates a low oil content which in turn provides a product of desirable tensile strength. If the sample cake is substan-

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tially colorless, the operator then determines its melting point by a suitable method such as the ASTM method D87-42 described in ASTM Standards Handbook (1949), page 698 or the Saybolt procedure. If the melting point meets the specifications, the operator will then terminate the sweating operation.

It has now been found that the oil content of the wax in the sweater may be readily and easily determined at any particular time by determining the absorptivity of a sample of the foots oil at a particular wavelength of light as explained more fully below. Thus, it is observed that when the absorptivity of oil-wax mixtures for ultra-violet (u. v.) light having wavelengths between 240 and 350 millimicrons is plotted against the wavelengths, each oil-wax mixture will have a characteristic peak absorption at a particular wavelength somewhere within that range, i. e., it will absorb a greater amount of light of that particular wavelength value between 240 and 350 millimicrons than it will at higher or lower wavelengths within that range. This absorptivity peak may occur at a different wavelength for different oil-wax stocks but always occurs at the same wavelength for different oil-wax samples taken from stocks of the same type and source. It has also been found that there is a correlation between the numerical value of absorptivity of the foots oil for light of that particular wavelength and the oil content of the wax in the sweater; that is, there is a direct relationship between the oil content values of the wax in the sweater and the absorptivity values at the peak of absorptivity for various foots oil samples. This makes it possible, in accordance with the invention, to determine the u. v. absorptivity of foots oil samples (a simple and quick procedure) and from the determination to ascertain the oil content of the wax in the sweater. In this way, the sweating can be terminated when the oil content of the wax in the sweater reaches a desirably low figure. Such a resulting wax of low oil content will possess desirable properties of improved tensile strength, and color stability. Thus an oil-wax stock can be readily sweated to a predetermined value below that initially contained in the stock or to an oil content less than some specified value, such as 5 per cent. The same determination may be made of the oil removed by solvent extraction after the oil is recovered from the solvent and the oil content of the wax obtained from a similar but not necessarily identical correlation, as more fully explained below. This procedure and control for sweating operations is described below in connection with the drawings in which:

Figure 1 is a graph, illustrating the absorptivity values at various wavelengths of light of an oil-wax stock and showing the characteristic absorptivity peak of the particular wax;

Figure 2 is a graph on logarithmic plot illustrating three relationships between (a) absorptivity versus oil content of successive samples of a foots oil stream from a particular stock—curve R; (b) absorptivity versus oil content of wax within the sweating equipment from the same stock—curve R'; and (c) absorptivity of the foots oil stream versus the oil content of wax in the sweater from identical stock—calibration curve T; and

Figure 3 is a plot of time of sweating versus oil content of wax in sweater.

The respective relationships illustrated in Figure 2 are plotted on logarithmic scale purely for convenience in the conventional manner to represent the relationships as straight line curves. It will be readily appreciated that the same relationships could be represented by a curve on a linear scale.

Referring to Fig. 1, the absorptivity values (a) were calculated from data observed for a number of settings of a spectrophotometer using a sample of a foots oil

stream from a particular oil-wax stock and varying the wavelengths between about 240 and 350 millimicrons. In making the comparison, the  $a$  values were calculated from the relation

$$a = \frac{A}{c \times l}$$

where  $A$  is the absorbance of the sample which is determined as the logarithm to the base 10 of the reciprocal of the transmittance—that is, the ratio of the rate at which energy is transmitted in a beam of radiant energy (e. g., ultraviolet light) through a sample of the solution to the rate at which radiant energy is supplied to the sample;  $c$  is the concentration of the sample solution in grams per liter; and  $l$  is the length in centimeters of the sample through which the beam of light passes.

A definite absorptivity peak was observed at a particular wavelength which is readily ascertained from a plot of the rate as shown in the diagram plotted as Figure 1. It was observed that a peak of absorptivity occurred at the same wavelength for a number of samples of the sweat stream from the same type of wax stock.

The ASTM weight per cent oil content was determined for the same foots oil samples on which the absorptivity was computed. A plot of the absorptivity values of the foots oil samples at the peak wavelength, when plotted against the ASTM oil content of the samples foots oil gives a smooth curve. When plotted on a logarithmic scale the curve is a straight line curve such as curve R of Fig. 2 (absorptivity values of sweat stream versus oil content of sweat stream).

From the same control batch of wax, the absorptivity values of a series of sweated refined wax samples containing various observed amounts of oil in the sweated product were likewise determined and plotted on the same logarithmic plot against the oil content; this plot was also a straight line curve ( $R'$ ) which was found to be on the same line as curve R but falling at lower values of percentage oil content as shown in Fig. 2 as curve  $R'$ . This curve of absorptivity versus oil content of the wax being a lower extension of the curve of the absorptivity versus oil content of the sweat stream taken from the same type of stock in a sweating operation, demonstrates the direct relationship between the properties of the foots oil stream and the wax within the sweater from which the foots oil is taken. The curves R and  $R'$  actually may be joined and extended at both ends as illustrated in Fig. 2. The resulting curve  $R-R'$  (consisting of the sections R and  $R'$  and extrapolations) is designated in the balance of the disclosure as the "u. v. curve" and points read from the curve are "u. v. oil content." These values from the u. v. content curve are more nearly accurate for all correlations than the calculated values of ASTM oil content since the factors through which errors may be introduced in the ASTM test method far exceed those of the present spectrophotometric determinations. Thus the necessity or making a quantitative separation which is difficult and time consuming, measuring the wax sample only to one-tenth of a gram, dissolving the sample in methyl-ethylketone, taking a sample at a specified temperature from a body of wax of continuously changing temperature, and other steps in the ASTM test method introduce errors. On the other hand, in the spectrophotometric system there is no necessity for such separation, the sample is weighed to the fourth decimal place and is diluted and transferred but once (into the spectrophotometer cell). Furthermore, the operator need measure only one physical property in this latter method. Experience has shown that the measurements are more accurate and the handling of the sample simplified with resulting smaller chance for error.

Furthermore, a third plot on the same logarithmic paper of values of absorptivity of the foots oil samples versus the oil content of the wax removed from the sweater at the same time for different sweating operations on the same stock (for example, at the cut points—that is, the points at which sweating of several different batches

of the same stock is terminated) also gives a straight line curve, such as curve T of Figure 2, which is found to be parallel to both curves R and  $R'$  above. This is done for a series of samples of wax to obtain values of absorptivity of foots oil samples versus oil content of wax to demonstrate that a plot of these values is also directly related to both of the curves R (absorptivity versus oil content of foots oil samples) or  $R'$  (absorptivity versus oil content of wax in sweater). The propriety of the parallel relationship between curves  $R-R'$  and T is further strengthened by numerous other observations in which the oil-wax stocks are first treated by various refining procedures such as acid-treating to reduce the aromatic content and then the absorptivity versus oil content relationships observed. The respective curves are always in parallel relationship, being displaced to the left. The degree of shift depends upon the nature of the treatment. The curve T is now established as a base calibration curve for other batches of stock of the same type.

It follows therefore, that, for any subsequent sweating operation upon a different batch of stock of the same type as the control batch only a single point of absorptivity of foots oil versus oil content of wax in the sweater need be observed. This pair of values may preferably be taken at the cut point of a preliminary sample sweat. The locus of that point (absorptivity values of the foots oil versus the oil content of the wax) on a graphic plot will be on a calibration curve T for that particular batch of wax stock, which curve T is, as shown, parallel to either of the curves R or  $R'$ , above. Thus a curve T may be drawn through the single determined point and parallel to either of the curves R or  $R'$  and this curve T will be the calibration curve for subsequent portions of the same batch of stock. The simplest of the two possible curves (R and  $R'$ ) to determine will, of course, be the one of absorptivity versus oil content of the foots oil sample (R) and thus the points on curve R will ordinarily be determined. The calibration curve T can then be drawn through the single point parallel to the predetermined curve R. The oil content of the wax is the sweater at various points before the cut point can be also determined by reading from curve T the value opposite the observed absorptivity of the foots oil sample taken at that point.

The operator may now quickly determine the oil content of the wax in the sweater at any time during the sweating operation of all portions of the batch subsequent to the preliminary run by the expedient of observing with the aid of a spectrophotometer the absorptivity at a particular wavelength of light of a foots oil sample and, by reference to the established calibration curve T, can know the oil content of the wax in the sweater. He will then be able to know the point in the operation at which to terminate the treatment. In conducting the sweating operation, the operator is also enabled to know in advance, by reference to the calibration curve, the value of absorptivity of the foots oil stream to watch for in order to obtain a finished wax of the predetermined and desired oil content.

Similarly, with a wax stock from a different crude source, a calibration curve may be established by determining the wavelength at which the characteristic absorptivity peak is found, then running a preliminary sweat of a sample, determining the absorptivity at the peak wavelength and oil content of a series of foots oil samples, plotting and drawing a curve through these values, determining the oil content of the wax at the cut point of the control sweat, plotting and drawing a curve parallel to the first curve through the point represented by the absorptivity of foots oil and oil content of wax (both at the cut point); the latter curve is then the calibration curve for that particular wax stock.

Similar development of a calibration curve and its utility in controlling the de-oiling operations may be made for solvent de-oiling operations wherein the ab-

sorptivity of the oil extracted from oil-wax mixtures may be observed.

The present invention, therefore, provides a method of determining the oil content of wax during de-oiling operations and a method of de-oiling oil-wax mixtures wherein control over the method is exercised by determining the absorptivity of successive samples of the foots oil or extracted oil for light at an observed characteristic wavelength between 240 and 350 millimicrons. It is possible, by means of this control method, to control the de-oiling operation with such precision that the oil content of the wax being de-oiled, when the operation is terminated, will be within 0.05% of the amount desired. Thus, the de-oiling operation can be observed at regular intervals and terminated when the oil content of the wax is found to be of a predetermined and commercially acceptable value.

The reason for the agreement between the correlation of the absorptivity values of samples of the foots oil or extracted oil and the oil content of the refined wax is not entirely understood but it may be surmised that the aromatic content of the oil in the wax and in the oil samples accounts for the peak values of absorptivity and that there are at all times aromatics present in the oil content to give the characteristic peak. However, regardless of any theory the discovery provides an adequate and advantageous method for controlling the de-oiling of wax stocks in the preparation of refined waxes of any desired final oil content.

The method of control may be carried a step further using as a basis the system disclosed herein. The time interval between the beginning of the de-oiling operation and the taking of the respective foots oil or extracted oil samples can be recorded and, as each sample is examined for determination of the oil content of the wax, these values may be plotted as in Fig. 3—time versus oil content. From a plot of a series of sets of values a smooth curve may be drawn as illustrated in Fig. 3. By extrapolating the curve a fairly accurate time may be rapidly estimated for the termination of the de-oiling operation and samples of the foots oil or extracted oil may be taken and observed at more frequent intervals as the time for termination of the treatment approaches so that the most desired point for the oil content of the refined wax and thus for the termination of the de-oiling operation may be observed closely. Here the relationship is plotted on linear scale although, if desired, it can also be represented on a logarithmic scale.

The method of this invention has many important advantages. Not only is the method of control more accurate than methods heretofore known but it is also much faster and simpler. The ASTM method for determining the oil content of a paraffin wax requires from 3 to 8 hours. An analysis of the oil samples for absorptivity of ultraviolet light of a given wavelength and thereby the oil content of the wax in the sweater in accordance with the present invention requires only about 20 to 30 minutes. Not only is the operator enabled to know the time at which the wax in the sweater will meet the desired and predetermined specifications of oil content and thereby tensile strength for the commercial trade, but he can know the proper time to terminate the operation in order that loss of desirable wax will be minimized in the oil stream being withdrawn, since it is recognized as inevitable that a relatively small but increasing amount of the desirable wax content will be taken from the wax stock with the oil stream near the end of the de-oiling operation particularly in the case of sweating when, of course, the wax stock in the sweater has been elevated in temperature to release the heavier undesired oils. Thus the operator can select that point in the operation at which the wax will possess just the desired properties for a finished wax product but still will not be treated so long as to result in loss of product.

In the operation of the present process for sweating an

oil-wax mixture, it has been found advantageous to provide a circulating stream of heated water through a plurality of relatively small pipes located throughout the tank type of sweater. Since it is desirable for the wax stock and the temperature to be elevated slowly at a constant rate, it has been found that these desirable conditions may be obtained by providing an automatic temperature control on the circulating stream of water. This may consist of a time-temperature controller which may actuate a diaphragm control valve which in turn governs the rate at which pressurized and superheated steam is introduced into a circulating stream of water. Thus the amount of superheated steam and thereby the temperature of the circulating water may be gradually increased at a constant predetermined rate with respect to length of time of processing or treatment. In this manner, substantially all of the lighter undesirable oils will be removed before the progressively heavier undesirable oils are removed from the body of wax in the sweater. In this way a more effective sweating and uniform finished product may be obtained.

#### EXAMPLE

A 3-gram sample of a crude paraffin wax known to have an oil content of 2 to 10% by weight is made into a 100 cc. solution with pure iso-octane. The 100 cc. solution is poured into a clean, dry, tared 100 cc. volumetric flask. The optical density or absorbance of the sample for light having wavelengths within the range of 240 to 350 millimicrons is then determined with a spectrophotometer such as a Beckman Model DU Photoelectric Quartz Spectrophotometer in known manner. In the following table the values of "A" as defined heretofore were measured using an iso-octane solution of the stock at a concentration (c) of 2.907 grams of wax per liter and an optical path (d) in the spectrophotometer of 0.998 centimeter, the values of "a" were calculated from the relationship described hereinbefore and the following results were obtained:

Table A

| Wavelength, mu | Absorbance<br>A | Absorptivity<br>a |
|----------------|-----------------|-------------------|
| 220            | .719            | 0.248             |
| 222            | .701            | 0.242             |
| 224            | .701            | 0.242             |
| 226            | .700            | 0.241             |
| 228            | .701            | 0.242             |
| 232            | .690            | 0.238             |
| 236            | .669            | 0.231             |
| 238            | .611            | 0.210             |
| 240            | .594            | 0.184             |
| 242            | .447            | 0.154             |
| 244            | .359            | 0.125             |
| 246            | .262            | 0.090             |
| 248            | .248            | 0.086             |
| 250            | .251            | 0.087             |
| 252            | .250            | 0.090             |
| 254            | .253            | 0.091             |
| 256            | .262            | 0.090             |
| 258            | .254            | 0.088             |
| 260            | .244            | 0.084             |
| 262            | .222            | 0.077             |
| 264            | .194            | 0.067             |
| 266            | .173            | 0.060             |
| 268            | .156            | 0.054             |
| 270            | .142            | 0.049             |
| 272            | .123            | 0.042             |
| 274            | .108            | 0.037             |
| 276            | .097            | 0.033             |
| 278            | .051            | 0.018             |
| 280            | .038            | 0.013             |
| 282            | .025            | 0.010             |
| 284            | .024            | 0.008             |
| 286            | .020            | 0.007             |
| 288            | .018            | 0.006             |
| 290            | .018            | 0.006             |
| 300            | .018            | 0.006             |

It was found that, for the crude wax thus tested, the curve obtained by plotting a values against wavelength as illustrated in Fig. 1, had a peak absorptivity at 257 millimicrons. Similar tests with other samples of wax from the same source showed that all waxes from that source had a maximum absorptivity at 257 millimicrons. Consequently, the determination thus far described need

be made only once so long as the wax sweated comes from the same source.

A commercial wax sweater was then filled with crude wax similar to that of the sample and the sweating was carried out in the conventional manner. Samples of the foots oil were taken after 0, 4, 9, 14, 17, 20 and 20¾ hours. The absorptivity values of aliquot portions of these samples for light having a wavelength of 257 millimicrons were determined in a Beckman Model DU Photoelectric Quartz Spectrophotometer in the same manner as previously described, and recorded in column 2, Table I appearing hereinafter. The remaining portions of these samples were tested for correlation by the conventional ASTM testing methods to determine their oil content and the results recorded in column 3 of Table I. The values thus obtained were plotted on logarithmic graph paper with the ASTM oil content in per cent as the ordinate and the absorptivity ( $a_{257}$ ) as the abscissa.

The experiment was repeated on the same wax stock in two other tests and the values recorded in Tables II and III hereinafter as columns 2 and 3 respectively. These values were likewise plotted on Fig. 2 and the curve R drawn as shown. This curve R represents the mean of the points plotted of absorptivity versus ASTM oil content of the foots oil samples and is designated the "u. v. oil content" curve. Due to the many factors which cause the ASTM calculations to digress slightly as explained before, the u. v. content values are of greater accuracy.

In addition in Tables I, II and III, there are included the observed absorptivity of the wax and the ASTM oil content of the wax in the sweater at the cut points (the points at which the sweating is terminated). This absorptivity of the refined wax, when plotted against the ASTM oil content of the wax, gives the straight line curve R', which is actually an extension of curve R.

Curves R and R' are joined and extended as shown in Fig. 2 to emphasize further the parallelism and identity of relationship between the characteristics of the foots oil stream and the wax in the sweater.

Similarly the values from the curve R' for the oil content of the wax in the sweater represent the "u. v. oil content" of the wax and the values for the wax at the cut points of the sweating runs is recorded in column 5 of Tables I, II, and III, respectively. These values are subject to less error than the ASTM values from which they differ slightly.

When the u. v. oil content values of the wax in the sweater at the cut point (recorded in columns 5 of Tables I, II and III) is plotted against the absorptivity of the foots oil sample at the same point and a curve is drawn through the points, the curve T of Fig. 2 is obtained. This curve T is found to be parallel to the curve R—R' and to correlate the absorptivity of the sweat stream with the oil content of the wax in the body of the sweater at the time the sweat stream sample was obtained. The data above referred to in the Tables I, II and III are as follows:

Table I

| Sweating Time, Hours | Foots Oil |                  | Refined Wax in Sweater |                              |                                   |                          |
|----------------------|-----------|------------------|------------------------|------------------------------|-----------------------------------|--------------------------|
|                      | $a_{257}$ | ASTM Oil Content | $a_{257}$              | U. V. Oil Content (curve R') | Oil Content (calibration curve T) | ASTM Oil Content (calc.) |
| (1)                  | (2)       | (3)              | (4)                    | (5)                          | (6)                               | (7)                      |
| 0.....               | 1.090     | 10.88            | -----                  | -----                        | -----                             | -----                    |
| 4.....               | 2.200     | 21.37            | -----                  | -----                        | 8.01                              | -----                    |
| 9.....               | 1.228     | 10.22            | -----                  | -----                        | 4.57                              | -----                    |
| 14.....              | 0.528     | 4.99             | -----                  | -----                        | 2.05                              | -----                    |
| 17.....              | 0.426     | 4.00             | -----                  | -----                        | 1.68                              | -----                    |
| 20.....              | 0.282     | 3.26             | -----                  | -----                        | 1.14                              | -----                    |
| Cut Point            |           |                  |                        |                              |                                   |                          |
| 20¾.....             | 0.276     | 2.78             | 0.103                  | 1.13                         | 1.11                              | 1.24                     |

Table II

| Sweating Time, Hours | Foots Oil |                  | Refined Wax in Sweater |                              |                                   |                          |
|----------------------|-----------|------------------|------------------------|------------------------------|-----------------------------------|--------------------------|
|                      | $a_{257}$ | ASTM Oil Content | $a_{257}$              | U. V. Oil Content (curve R') | Oil Content (calibration curve T) | ASTM Oil Content (calc.) |
| (1)                  | (2)       | (3)              | (4)                    | (5)                          | (6)                               | (7)                      |
| 0.....               | 2.436     | 22.00            | -----                  | -----                        | -----                             | -----                    |
| 5.....               | 1.182     | 11.08            | -----                  | -----                        | -----                             | -----                    |
| 10.....              | 0.576     | 5.9+             | -----                  | -----                        | -----                             | -----                    |
| Cut Point..          | 0.191     | 2.17             | 0.062                  | 0.71                         | 0.78                              | 0.80                     |

Table III

| Sweating Time, Hours | Foots Oil |                  | Refined Wax in Sweater |                              |                                   |                          |
|----------------------|-----------|------------------|------------------------|------------------------------|-----------------------------------|--------------------------|
|                      | $a_{257}$ | ASTM Oil Content | $a_{257}$              | U. V. Oil Content (curve R') | Oil Content (calibration curve T) | ASTM Oil Content (calc.) |
| (1)                  | (2)       | (3)              | (4)                    | (5)                          | (6)                               | (7)                      |
| 0.....               | 1.090     | 9.36             | -----                  | -----                        | -----                             | -----                    |
| 5.....               | 0.624     | 5.99             | -----                  | -----                        | -----                             | -----                    |
| 10.....              | 0.402     | 3.60             | -----                  | -----                        | -----                             | -----                    |
| 13.....              | 0.266     | 2.62             | -----                  | -----                        | -----                             | -----                    |
| Cut Point            |           |                  |                        |                              |                                   |                          |
| 14½.....             | 0.224     | 2.06             | 0.086                  | 0.96                         | 0.91                              | 0.87                     |

Summarizing this explanation, the values in columns 2 and 3 are plotted to give the curve R; the values of columns 4 and 7 are plotted to give the curve R' of Fig. 2; the values in columns 5 of each table are plotted against the absorptivity of the foots oil stream at the cut point (column 2) to give the relationship represented by curve T, which is found to be parallel to R and R', differing by a constant factor which shifts the curve to the right on the graphic plot. Thus, the value of the oil content of wax in the sweater may be determined at any time during the sweating operation by examining a sample of the foots oil at that time and the value determined from curve T. These values for the wax at the cut point are shown in columns 6 of Tables I, II and III and it is seen how closely the values for oil content from curve T agree with the u. v. content shown in columns 5 of each table. In Table I, the value of the oil content in the wax sweater is read from calibration curve T at each time the foots oil sample is examined. The successive values are recorded in column 6 and are plotted against the time of sweating in Fig. 3. The value of oil content at 0 hour is not recorded as it differs but little from that when temperature has been raised slightly at the end of 4 hours and the reading is more accurate after the sweating treatment has been in operation a short time. The time-oil content relationships as recorded in Table I and plotted in Fig. 3 can be obtained for operations in Tables II and III also, if desired.

In a subsequent operation, the values reported in Table IV below were observed. The absorptivity of the foots oil at the cut point was observed to be 0.212 and the value from the calibration curve (column 3) indicated that the oil content of the refined wax in the sweater at this point would be 0.86. This is close and within practical limits with the u. v. oil content value of 0.72 and even nearer the actual calculated ASTM value of 0.855 which is a difference of only 0.005 from the value as determined from the established calibration curve T. The agreement between values determined from the calibration curve and the actual values of the oil content of the wax is very close and well within a permissible difference for all practical purposes.

Table IV

| Sweating Time,<br>Hours | Foots<br>Oil,<br>$a_{257}$ | Refined Wax in Sweater                          |           |                                          |                                   |
|-------------------------|----------------------------|-------------------------------------------------|-----------|------------------------------------------|-----------------------------------|
|                         |                            | Oil<br>Content<br>(calibra-<br>tion<br>curve T) | $a_{257}$ | U. V.<br>Oil<br>Content<br>(curve<br>R') | ASTM<br>Oil<br>Content<br>(calc.) |
| (1)                     | (2)                        | (3)                                             | (4)       | (5)                                      | (6)                               |
| 0                       | 3.442                      | 12.3                                            |           |                                          |                                   |
| 4                       | 1.854                      | 6.8                                             |           |                                          |                                   |
| 8                       | 1.010                      | 3.82                                            |           |                                          |                                   |
| 12                      | 0.718                      | 2.75                                            |           |                                          |                                   |
| 15½                     | 0.432                      | 1.09                                            |           |                                          |                                   |
| 19½                     | 0.306                      | 1.22                                            |           |                                          |                                   |
| 20½                     | 0.280                      | 1.12                                            |           |                                          |                                   |
| 21½                     | 0.234                      | 0.95                                            |           |                                          |                                   |
| Cut Point 22½           | 0.212                      | 0.86                                            | 0.664     | 0.72                                     | 0.855                             |

The above example shows that the time to terminate the sweating in order to obtain a wax product of proper properties, may be ascertained readily by examining the absorptivity of a sample of the foots oil stream and referring to the calibration curve T to determine if the wax has at that point the desired and predetermined oil content to meet the specifications.

The values of time at which foots oil samples were taken were recorded in column 1 and these values were plotted in Fig. 3 against the oil content of the wax in the sweater as it is determined (from calibration curve T) at each examination of a foots oil sample. From the resulting curve (Fig. 3) it can readily be seen that, by extrapolating the curve beyond values actually observed after several hours, it is possible to determine approximately the time at which the sweating operation should be terminated to yield a product of predetermined oil content as read along the ordinate axis of the plot.

The preceding description has been described with special reference to a control of the sweating of oil-wax stocks to obtain a refined finished wax of predetermined oil content by observance of the absorptivity of samples of foots oil from the sweater and reference to a calibration curve.

With slight modifications a different calibration curve may be established and a similar method of control obtained in solvent de-oiling where the absorptivity of the oil removed from the oil-wax sample could be correlated with the oil content left in the wax and the operation controlled by determining the absorptivity of successive samples of solvent-removed oils and thereby observing the oil content of the wax.

It is evident that numerous modifications will immediately appear to those skilled in the art upon reading the foregoing description. All such modifications are intended to be included in the scope of the invention as defined in the following claims.

We claim:

1. The method of preparing finished wax of a predetermined oil content which comprises the steps of (1) sweating an oil-wax mixture, (2) withdrawing a sweat stream sample, (3) passing through said sample ultra-violet light of that wavelength between about 240 and 350 millimicrons which corresponds to the characteristic absorptivity peak of said sample and noting the absorptivity value of the sample at that wavelength, (4) repeating steps two and three until the absorptivity of the sweat stream sample reaches a value which corresponds to the predetermined oil content of the wax within the sweater as shown by a preestablished correlation between the oil content of the wax and the absorptivity of the sweat stream sample, and (5) then immediately terminating the sweating.

2. A method according to claim 1 in which the predetermined oil content of the wax is below 5%.

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