MANUFACTURE OF PARAFFIN WAX
FROM PETROLEUM

By
RALPH H. ESPACH
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INTRODUCTION

Paraffin wax is an interesting and valuable product obtained in the refining of many crude petroleums. Petroleum when refined is separated into a number of fractions by vaporization and fractionation. One of the most important of these is a lubricating-oil fraction from which wax, if present, must be removed before the manufacture of satisfactory lubricants is possible. Most of the Eastern, Mid-Continent, and Rocky Mountain petroleums contain wax, while the majority of the Gulf coast and many of the California petroleums are free of wax. The removal of wax from lubricating-oil fractions is a rather expensive refining operation. The equipment necessary for wax removal is costly, but it has long life and its maintenance costs are low. The equipment for finishing the crude wax into commercial products is not costly compared to that for removing the wax from the oil. As the cost of wax removal is chargeable to the lubricating oil, the actual refining of the crude wax into commercial products should be profitable even with the low prices for wax that have obtained for several years. The increase in the price of wax (more than double—low 2.087, high 4.275 cents per pound for 124–126 a. m. p., white crude scale, f. o. b. New York) during 1933 greatly stimulated wax-manufacturing activities. The amount of paraffin wax and lubricants produced in the United States during the past 10 years 3 and the price of wax (representing the yearly average of the weekly prices in the National Petroleum News for 124–126 a. m. p. Pennsylvania white crude scale wax, f. o. b. New York) are given in the following table:

Table 1.—Paraffin-wax and lubricant production in the United States, 1925–34

<table>
<thead>
<tr>
<th>Year</th>
<th>Wax (pounds)</th>
<th>Price (cents per pound)</th>
<th>Lubricant (barrels)</th>
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<tr>
<td>1925</td>
<td>590,577,000</td>
<td>5.607</td>
<td>31,055,000</td>
</tr>
<tr>
<td>1926</td>
<td>645,815,000</td>
<td>5.312</td>
<td>32,293,000</td>
</tr>
<tr>
<td>1927</td>
<td>584,347,000</td>
<td>3.488</td>
<td>31,721,000</td>
</tr>
<tr>
<td>1928</td>
<td>630,144,000</td>
<td>4.381</td>
<td>34,658,000</td>
</tr>
<tr>
<td>1929</td>
<td>630,074,000</td>
<td>3.565</td>
<td>34,359,000</td>
</tr>
<tr>
<td>1930</td>
<td>547,688,000</td>
<td>2.941</td>
<td>34,301,000</td>
</tr>
<tr>
<td>1931</td>
<td>477,406,000</td>
<td>2.143</td>
<td>26,794,000</td>
</tr>
<tr>
<td>1932</td>
<td>458,923,000</td>
<td>2.076</td>
<td>22,437,000</td>
</tr>
<tr>
<td>1933</td>
<td>469,566,000</td>
<td>2.972</td>
<td>23,775,000</td>
</tr>
<tr>
<td>1934</td>
<td>468,726,000</td>
<td>3.607</td>
<td>26,294,000</td>
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* Work on this manuscript completed June 1934.
† Associate petroleum engineer, U. S. Bureau of Mines Petroleum Experiment Station, Bartlesville, Okla.
Although wax is a necessary byproduct in the manufacture of most lubricating oils it also has major economic importance.

The production of commercial wax is closely related to the production of lubricants, because of the necessity for removing wax from lubricant stocks, but it does not necessarily follow production of lubricants. The market demand for wax determines whether the crude wax is refined further into commercial waxes or whether it is used as cracking stock in conversion processes for the production of motor fuel. Thus, the quantity of wax produced, as shown in table 1, could be increased should demand warrant. It is of interest that the quantity of wax exported from the United States in 1931 and all previous years for which there are records always has exceeded the domestic demand (imported wax included). Since 1931 the amount of wax exported has been less than the domestic demand for wax, in 1933 amounting to 70 percent of the domestic demand. The amount imported has been small (not over 8 percent of the United States production), but it consists of the higher-melting-point grades.

Practically all of the imported wax comes from the Netherland East Indies and British India.

Since paraffin wax has convenient melting points, will bend, and is tenacious at ordinary temperatures, does not deteriorate, is impervious to water, and has a high dielectric strength, it is used extensively in the manufacture of candles; the impregnation of waxed papers; the coating of paper cartons (butter, cheese, ice cream), drinking cups, milk bottles, and milk-bottle tops; electrical insulation; waterproofing; the impregnation of match tips; floor and furniture polishes; laudering; the protection of preserves and jams from fermentation; coatings for cheeses to improve their appearance and prevent mold, evaporation, and shrinkage; the lining of butter tubs; coatings for beer vats and barrels (vinegar, cider, alcohol, whisky, molasses, and sauerkraut); coatings for meats, sausages, and other products which must be prevented from drying; protective wax dressings for burns; the manufacture of artificial flowers; etching glass; miners' lamps and marine bunker lights; waxing yarns in the textile industry; stuffing or loading for leather in tanneries; and for numerous other materials and purposes.

ACKNOWLEDGMENTS

The Bureau of Mines desires to express appreciation of the willing cooperation of the wax-manufacturing petroleum refiners who gave the Bureau's representative access to their refineries and supplied information on their methods of wax-distillate production and paraffin-wax manufacture.


Special recognition for helpful information and a critical review of the manuscript is due Will M. Morgan, Carbondale Machine Co.
The author also wishes to thank the following persons for reviewing and criticizing the manuscript: Dowell Gray, B. H. Lincoln, and Walter Miller, Jr., of the Continental Oil Co., and W. W. Leach, P. L. Smith, T. S. Richardson, C. M. Fabian, and R. R. Jackson, of the Magnolia Petroleum Co.

This work was done under the general supervision of R. A. Cattell, chief engineer; H. C. Fowler, former acting chief petroleum engineer; and H. H. Hill, former chief petroleum engineer of the Petroleum and Natural Gas Division, Bureau of Mines, and under the direct supervision of N. A. C. Smith, superintendent of the Petroleum Experiment Station, Bartlesville, Okla. The assistance of H. P. Rue, A. J. Kraemer, H. M. Smith, J. W. Horne, O. C. Blade, and J. M. Seward, of the Petroleum and Natural Gas Division of the Bureau of Mines, who critically reviewed the manuscript and helped in its preparation, is gratefully acknowledged. This Bureau of Mines project is a continuation or extension of work begun by L. D. Wyant and L. G. Marsh.

**SCOPE OF REPORT**

In the refining of wax-bearing crude petroleums by certain methods of processing, three materials, referred to in refining terminology as waxes, are encountered: Paraffin wax, slop wax, and petrolatum. The wax obtained from the lubricating-oil fraction of the crude oil, termed "wax distillate," when properly processed is referred to as paraffin wax. After the removal of the wax-distillate fraction in the distillation of a crude oil by certain operating methods, a slop-wax-distillate fraction is obtained. The wax in this fraction is referred to as slop wax and has little, if any, value, the whole fraction usually being used as cracking stock. If, however, market demand should justify processing this fraction, satisfactory commercial wax could be obtained from this source. Wax is encountered along with the heavy lubricating-oil fraction at or near the end of the crude-oil distillation and must be removed from this fraction to permit the manufacture of satisfactory lubricants. In the separation of this wax from the oil fraction some oil remains associated with the wax. The mixture is called "petrolatum." The amount of petrolatum available for commercial products exceeds the demand for such products, although it has a rather wide range of uses, and consequently much of it is disposed of as cracking stock.

It is the first one of these "waxes", paraffin wax, that is of greatest commercial importance, and this report proposes to give a detailed picture of the manufacturing process employed and equipment used in its production.

The information given herein was derived from data accumulated in 1929 and 1930, when 30 wax-manufacturing refineries located throughout the United States (California and the Rocky Mountain region excepted) were visited. These 30 refineries had an approximate total wax production of over 75 percent of the annual United States production at that time.

The wax-manufacturing process and the equipment employed are essentially the same at present as in 1929 and 1930. However, the increased use of good fractionation in the distillation of the crude
oil, especially in distillation under greatly reduced pressures (when preparing the wax distillate), is resulting in high-viscosity wax distillates that make the chilling and pressing operations so difficult that it sometimes is necessary to mix diluents (light petroleum fractions) with the distillate to lower its viscosity and make subsequent processing easier. Solvents also are being employed in the removal of wax from the distillate. The use of solvents involves some changes in the manner of handling the distillate and the slack wax.

PARAFFIN WAX

Paraffin wax is a mixture of hydrocarbons, solid at ordinary temperatures and usually obtained from petroleum. It is colorless or white, more or less translucent, without odor or taste, and greasy to the touch. It has a crystalline structure, but this is not observed readily except after crystallization from a solvent. The crystallization of wax has been studied extensively, and in later years the work, among others, of Padgett, Hefley, and Henrikson,4 Carpenter,5 Buchler and Graves,6 Rhodes, Mason, and Sutton,7 Tanaka, Kobayashi, and Ohno,8 Ferris, Cowles, and Henderson,9 and Katz10 has added much to the knowledge of wax crystallization.

PROPERTIES

The physical properties of paraffin wax vary, depending upon the material of origin and the process of manufacture. Some petroleums give only high-melting-point waxes; others give both high and low ones. The process of manufacture, especially the method of distilling the oil to obtain fractions that contain wax, can change greatly the characteristics of the wax.

MELTING POINT

The main commercial property of paraffin wax is its melting point; the higher the melting point the more valuable the wax. The range in melting point of the bulk of commercial paraffin waxes is from 118° to 136° F. However, waxes of as low as 108° F. and as high as 160° F. find commercial uses. Wax is a mixture of various hydrocarbons of different melting points, and the manufacture of the wax is such that all the waxes are solidified; then the lower-melting-point ones are melted or "sweated" out of the solid wax cake, leaving a porous, fibrous mass of higher-melting-point waxes. The melting point of a commercial paraffin wax signifies the temperature at which the mixture of hydrocarbons will liquefy.

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To determine the melting point of wax it is melted and allowed to cool, a time-temperature curve being kept on the cooling. The various hydrocarbon mixtures gradually solidify so that, while one temperature may be determined as the solidification point of the wax (when most of the mixture of hydrocarbons solidifies), this temperature is not the solidification point of all the hydrocarbons in the wax. The temperature curve shows that a range of solidification points is really determined, which depends upon the homogeneity of the hydrocarbons in the wax. For a temperature-time curve on cooling a melted wax certain conditions of temperature are obtained, and the drop in temperature of the wax is then observed. A slight decrease in the rate of cooling will be noticed, indicating the beginning of solidification of some of the hydrocarbons (latent heat of fusion being given up). Then the temperature will remain nearly constant for a time as the major part of the wax crystallizes, after which the rate of cooling will again increase. The temperature at which most of the wax crystallizes is called the melting point of the wax. (See p. 4.) The more closely the wax has been fractionally sweated the more homogeneous it becomes and the more nearly does the melting-point determination represent the actual melting points of the hydrocarbons in the wax. Where several cuts are made in the sweating operation and these cuts are sweated further, the finished wax will contain hydrocarbons of closer melting points than a wax with the same melting point but which is the direct product of only one sweating.

When waxes from petroleum are analyzed, hydrocarbons are found that have melting points ranging roughly from 100° to 200° F. The hydrocarbons in waxes, although of wide melting-point range, have been found to belong mainly to the normal and isoparaaffin series of hydrocarbons with the formula of $C_nH_{2n+2}$ and to range from $C_{15}H_{33}$, melting point 80.5° F., to $C_{48}H_{88}$, melting point 182.0° F., in Salt Creek (Wyo.) crude oil and from $C_5H_{14}$, melting point 106° F., to $C_{15}H_{30}$, melting point 206° F., in Burmah crude oil. As compared with these, Buchler and Graves found in slack wax from Salt Creek crude, pressed at 0° F., hydrocarbons from $C_{15}H_{33}$, melting point 80.5° F., to $C_{32}H_{66}$, melting point 156° F. Carpenter found hydrocarbons from $C_{25}H_{44}$, melting point 106° F., to $C_{34}H_{70}$, melting point 161° F., in refined paraaffin wax from Burmah crude oil.

The work of Ferris, Cowles, and Henderson, also of Clark and Smith, using slack wax from Mid-Continent crude, indicates that in addition to the normal paraaffin hydrocarbons petroleum waxes contain a number of homologous series. Clark and Smith state that Mid-Continent paraaffin wax apparently contains about 65 percent of normal paraaffins and at least 20 percent of isoparaaffinic material.

Carpenter suggests the possibility of the presence of isomers of the normal paraaffins or even of saturated naphthenes of general formula $C_nH_{2n}$.

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11 See footnote 6.
12 See footnote 5.
13 See footnote 9.
COLOR AND APPEARANCE

The color and appearance of commercial waxes are important factors in the value of the wax. If the wax is not white or colorless, disregarding the property of translucency, oil is present. Yellow scale wax contains up to 3 percent of oil which imparts a yellowish tinge to the wax. The presence of oil in wax should be kept at a minimum in most cases. Some uses for wax permit an oil content as high as 3 percent, but usually it is kept under 1 percent and in most refined waxes under 0.5 percent. Although no standard testing method has been adopted, the color of wax usually is determined by the Lovibond tintometer, using the standard amber (500 series) Lovibond glasses and a cell length of 6 or 12 inches in which the melted wax is contained and which is to be compared and matched with the standard glasses. In general, refined waxes have a color of one-half or less using a 6-inch cell, and semirefined and white scale waxes have a color of 1 or more. These color numbers should be about double when 12-inch cells are used.

In terms of the Saybolt chromometer, which is used almost universally for lighter oils and which also is used sometimes for determining the color of waxes, the color ranges from 20 to 25, most refined waxes being near 25. This is "water white," as the descriptive term is commonly used.\(^{15}\) The wax is in a melted state when its color is determined by either the Lovibond tintometer or the Saybolt chromometer. It should not be heated higher than necessary to keep it fluid until it can be removed from the cell, 150° to 160° F. being as high as is usually necessary. If the wax contains any suspended particles or moisture this should be removed by filtering the melted wax before it is put in the cell.

The stability of the color of a wax is as important for many uses as the color at the time the wax is made. Waxes can be manufactured water white but upon remaining in storage for a time gradually change in color. If they are exposed to light this action is accelerated greatly. The change in color, a gradually increasing yellow tinge, is attributed to the small amounts of unsaturated hydrocarbons left in the wax, which become dark in color upon polymerizing. Since there is usually a considerable interval between the manufacture of the wax and the time it reaches the consumer color stability receives considerable attention. The matter of color stability is entirely one of proper refining and can be controlled by the removal of hydrocarbons that may later polymerize.

Much can be learned about a refined wax from its appearance. Two waxes of the same melting point and color can appear quite different when cooled and solidified under the same conditions. One may be translucent, while the other is opaque or even white and chalky. According to the work of Mittler and Lichtenstern\(^{16}\) the opaque appearance is attributable to the oil content of the wax. They showed that the wax would be translucent, slightly mottled, or milky, depending on the oil content. These appearances were obtained by the addition of 0.25, 0.50, 1.0, and 3.0 percent, respectively.

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of oil obtained from the wax in its refinement to a $135^\circ$ F. melting-point, translucent, refined wax. When an equal weight of this same translucent wax was heated with a translucent wax of $108^\circ$ F. melting point the resulting wax was still translucent. It is known, however, that translucent paraffin waxes are made with 1-percent and higher oil and moisture contents. It would seem more likely that an explanation other than that of oil content would be more satisfactory in accounting for the appearance of wax.

Occluded or dissolved air or gas in the melted wax may form minute bubbles when the wax solidifies, and these will give the wax an opaque appearance. The rate at which a melted wax solidifies can be responsible for the degree of translucence. The slower the wax is cooled the more translucent the solid wax. Moreover, a cloudy wax can be made more translucent simply by heating it for a time at a temperature several degrees below its melting point. This clearing up and formation of translucent wax as a result of slower cooling are attributable to the formation and growth of larger wax crystals.

Probably the factor of greatest importance in influencing the appearance of a wax is the relative amount of the higher waxes, iso-paraffins, or other hydrocarbons such as the "soft-wax" impurity of Buchler and Graves, the "ceresine" of Sachanen, Zherdeva, and Vasilyev, and the "asphaltic material" of Padgett, Hefley, and Henriksen. These materials are considered capable of affecting the wax crystallization and by so doing influence the appearance of the solidified wax. The opinion is general that, disregarding the factors of occluded air and rate of cooling, the more sharply fractionated waxes form more translucent wax cakes on solidifying than the waxes from roughly separated distillates containing heavy ends and a greater abundance of the materials mentioned. Wax cakes from such distillates are more likely to be mottled or white. It has been found that waxes from distillates of increasingly higher boiling-point range have increasingly a more mottled appearance. The appearance of a wax can be used to a degree as a measure of its hardness and tensile strength. Usually the translucent waxes are harder, more solid, and of greater tensile strength than opaque or white waxes. The appearance of the fractured surface of a broken wax cake, whether rough, chalky, and crumbly, or firm, translucent, and with good needle or fibrous structure is a guide to the tensile strength of the wax. The tensile strength of a wax is a property of considerable importance to manufacturers of waxed papers.

**TENSILE STRENGTH**

For more information on certain characteristics and qualities of paraffin waxes, data relative to the tensile strength of waxes are obtained by certain refiners and manufacturers. No standard method of test is as yet general practice. Since the tensile-strength characteristic of a wax apparently gives some insight into how the wax will function when used as a coating on paper or possibly as candles.

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17 See footnote 6.
19 See footnote 4.
and other fragile products and since such information probably will become more important as time progresses, the following method of test, as carried out by one of the larger wax-manufacturing refiners, is given and with it a discussion of what the test might indicate.\footnote{Communication from P. L. Smith, Magnolia Petroleum Co., May 18, 1934.}

To obtain tensile-strength data on waxes it is necessary to have certain equipment and a method of test so standardized that duplicate results can be obtained readily by different laboratories. As a result of considerable experimental work, both with various types of equipment and different methods of tests, the following procedure has been adopted, and it is thought that concordant and reproducible results can be obtained regardless of the time or place of testing.

The equipment consists of a Perkins tensile tester manufactured by B. F. Perkins & Son, Inc., Holyoke, Mass.; 8 steel molds; 8 aluminum or brass plates at least 1 inch larger than the molds; 1 burner and tripod with asbestos-wire gauze or an electric hot plate with asbestos paper; 1 water bath kept at 70° F.; 1 cold room adjusted to 70° F. ± 2° F. in which the air is maintained at a relative humidity of approximately 50 percent; steam bath, thermometers, and beakers for melting and heating the wax to 230° F.

The Perkins tensile tester (see fig. 1) was designed originally to determine the tensile strength of cloth and paper. However, clamp jaws have been fitted to the machine in which molded wax briquets fit snugly. The testing machine is equipped with a hand wheel and handle which is revolved at a rate of 12 r. p. m. The machines can also be equipped with a small motor and reduction gears. The motorized equipment was found to be essential to eliminate sudden variations in the speed of the wheel causing erratic results.

A minimum of 8 steel molds is necessary, as 8 determinations are made on a given wax sample. A minimum of 3 sets or 24 molds is necessary when a number of waxes are tested daily. It is of prime importance that each mold be made accurately so that the wax briquets fit the jaws on the machine perfectly. For instance, a few thousands of an inch variation in the dimensions of the inside corners of the shank on the mold may make a difference of 8 to 10 pounds in the tensile strength of the wax. The aluminum or brass plates are used as a base for the mold on which to pour the melted wax. It has been found that a paper or cardboard base contains enough moisture to lower appreciably the tensile strength of the wax. The brass plates should have a thin coating of mercury amalgam on the side next to the wax to prevent sticking; however, this is not imperative. The plates should be large enough so that they extend one-half inch beyond the molds on all sides. This facilitates handling and results in less breakage when the wax briquets are removed.

The asbestos-wire gauze on the gas flame or the asbestos paper on the electric heater is necessary to prevent local overheating when the temperature of the wax is increased to drive out the moisture and the air. The electric heater with the cover of asbestos paper is recommended, since the temperature can be controlled more readily.

The cold room is essential to reliable results. Experimental work indicated that the tensile-strength tests cannot be made on an open laboratory table. Seasonal variations in temperature cause even a
Figure 1.—Machine for determining tensile strength of paraffin waxes.
high-tensile-strength wax to show erratic and poor results. A cold room is not satisfactory unless the humidity of the air in it has been lowered so that no moisture collects on the molds. Any of the modern air-conditioning machines now on the market will be satisfactory for such a purpose. The cold room is lined with cork and 10 by 7 by 9 feet in size. An automatic control holds the temperature of the room at 70° F. ±2° F., with a relative humidity of approximately 50 percent.

The procedure for tensile-strength determinations is as follows: A sample of the wax to be tested, equivalent to approximately 300 cm³ of liquid wax, is melted in a 600-cm³ pyrex beaker in a steam bath or by heating it with gas very carefully on an asbestos wire gauze. The steam bath is favored even though a longer time is required. When completely melted the sample is heated slowly, either by a gas flame on a gauze or by an electric hot plate covered with a thin sheet of asbestos paper, until a temperature of 280° F. has been reached. The liquid is stirred carefully with a thermometer at this temperature to dispel any moisture or air which may be present in the wax. Presence of moisture is indicated by a cracking sound and by the appearance of small, colorless globules in the bottom of the beaker.

During the period of heating, the steel molds, after having been thoroughly wiped off with a cloth, are allowed to cool to 70° F. in the air. It has been suggested that the molds be cooled in water at 70° F. for 30 minutes. It has been found, however, that due to the very small amount of moisture left on the mold after drying it as completely as possible with a cloth the tensile strength is consistently lower than when the same wax is molded in air-cooled molds.

It is better to cool the molds in position on the aluminum or amalgamated brass plates to prevent further handling with moist hands. Both molds and plates must be perfectly dry and be maintained as closely as possible at 70° F.

Enough of the melted wax at 230° F. is then poured into eight cooled molds so that the contraction of the wax on cooling will not cause a hollow place in the shank. After cooling 2 hours at the room temperature of 70° F., the excess paraffin above the mold is shaved off smooth. Then the molds are removed, and the wax briquets are placed in water at 70° F. for 15 minutes, or until they have been adjusted to that temperature. They are then broken in the tensile tester by revolving the wheel at 12 r. p. m. This causes the two jaws to be pulled apart. The pressure registered on the gage at the breaking point is noted for each briquet broken.

The average variation allowable in the breaking-point pressures is ±10 percent of the average of the eight determinations. Consequently all results which fall either above or below such limits are discarded. The remaining results are averaged again for the final result of the tensile strength of the wax. Actually, this pressure represents the force required to break a one-fourth-square-inch surface of wax; nevertheless this is taken as standard and is not adjusted to pounds per square inch.

The question naturally arises as to why waxes should have approximately the same melting point and at the same time vary widely in tensile strengths. Figure 2 is a photomicrograph of a
high- and low-tensile strength, 130° to 133° F. melting-point wax. Both waxes will show approximately the same oil and moisture content. Of course, the larger crystals in the low-tensile-strength wax are possible through the incorporation of certain amounts of oil not capable of being detected by the A. S. T. M. oil-and-moisture determination. (See footnote 46, page 98.) It is evident that there are certain chemical characteristics in various waxes which cause a difference in tensile strength. A wax that is polymerized readily to form color and odor compounds in the presence of air and sunlight and forms insoluble bodies of high melting points after such exposure will generally give low-tensile-strength results.

The 132° F. melting-point, 70-tensile strength wax shown in figure 2 had an excellent sun stability and a smooth, translucent appearance in the original cake. The 30-tensile-strength sample turned dark brown after being exposed to the sun for 10 days and developed a disagreeable, penetrating odor. The original cake looked milky and crumbly.

Aside from the chemical nature, the tensile strength will also vary considerably on waxes of the same melting points, depending upon the amount of oil present. Small quantities of spindle oil of 200 seconds viscosity were mixed with a 60-tensile-strength wax, and a curve was drawn showing the effect of this addition of oil on the tensile strength of the wax. Figure 3 shows that even with 0.015 percent of oil present the tensile strength drops from 60 to 50 pounds. With 0.3 percent of oil, which is well within the allowable variation in the determination of expressible oil and moisture in paraffin waxes, the tensile strength dropped to 18 pounds. Thus

Figure 3.—Graph showing relation between tensile strength and percentage of oil in a 60-tensile-strength wax.
small amounts of oil that will not be indicated by the A. S. T. M. tentative method of test for expressible oil and moisture in paraffin waxes will affect seriously the tensile strength of the wax. The melting points, as shown on the curve, were not affected by the addition of oil. In conclusion, then, the tensile-strength determination will indicate either that the wax contains small amounts of oil or that it is chemically unstable.

**OIL AND MOISTURE CONTENT**

Another factor in the value of a wax is its oil and moisture content. Earlier in the history of the wax-manufacturing industry this factor was measured quantitatively by an oil and moisture test of the amount of liquid that could be pressed out of a 30- to 35-gram sample of small flakes or thin shavings of the wax by a pressure of 900 pounds per square inch applied in a wax-testing press.21

With waxes containing some oil, such as scale waxes, this test produces results that can be duplicated, but with waxes having less than 1 percent oil the test has little value. Due to developments in the wax industry the quality of the wax produced improved, and with the dry, refined waxes of today the press method has become of little value. A similar test, except that 2.5 to 3.0 ml of wax is molded into a given shape and subjected to a pressure of 1,000 pounds per square inch, is used at present, mainly because of the lack of a more satisfactory and generally acceptable test. The direct refractometer method 22 of determining the oil content of paraffin wax is used by one or more large wax-manufacturing refiners. This method establishes a relationship between the index of refraction and the percentage of oil in a wax sample by means of a specially designed chart and two determinations of indexes of refraction.

With certain exceptions, paraffin wax should be tasteless and odorless. These properties are important when the wax is to be used for coating papers and containers for foods. Taste and odor usually result from oil and/or chemically unstable materials in the wax.

**SOLUBILITY**

The fact that wax is less soluble in various substances than is the oil with which it is associated in oil-wax mixtures has been applied in various methods of determining the wax in oil-wax mixtures. It is also beginning to have considerable commercial importance. The higher the melting point of a wax from a given source the less soluble it is in a given solvent. The solvents used today are of value, not because of their ability to dissolve wax but rather because of their differential solubility for the wax and the oil associated with the wax at temperatures within the range of wax-plant processes. Oil-wax mixtures, or oil containing wax in solution, are mixed with the solvent, usually at a temperature above the melting point of the wax, and both the oil and wax go into solution. The solution is then cooled (to 40° F. down to below 0° F.). At these temperatures

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the wax crystallizes out of the solution and can be removed by settling or filter pressing. The oil still remains in solution and is recovered by distilling off the relatively volatile solvent.

Considerable work has been done on the solubility of wax in various solvents. The work of the following investigators is the most important of that published: Wyant and Marsh,23 Henderson and Ferris,24 Sullivan, McGill, and French,25 Smith,26 Weber and Dunlap,27 Poole and collaborators,28 and Carlisle and Levine.29

Numerous and divergent groups of solvents might be used for dewaxing oil. The list of such solvents given herein is only partly complete but includes those that are already in use and those that offer possibilities—liquefied propane and butane, light petroleum fractions (liquid at atmospheric temperatures and pressures), acetone, butanol, nitrobenzene, trichloroethylene, methyl ethyl ketone, ethyl and butyl acetates, ethylene chloride, methylene chloride, isopropyl alcohol, chlorobenzene, mixtures of acetone and benzol, toluol and butanol, and acetone and butanol.

As stated above, the use of solvents is beginning to have considerable importance. For a number of years one refiner has been using a benzol and acetone mixture to remove wax from the wax distillate. This solvent process has recently been described by Govers and Bryant.30 A more recent process makes use of propane or butane in which part of the solvent is evaporated adiabatically to self-refrigerate the solvent and distillate for crystallization of the wax.31

**SEPARATION FROM PETROLEUM**

**IN WELLS, PIPE LINES, AND CRUDE-OIL STORAGE**

Petroleum is a liquid mixture of numerous hydrocarbons that may or may not contain those which, when separated from the mixture, are solid wax. So-called "paraffin" and "intermediate-base" crude oils contain waxes held in solution. The amount and melting points of these waxes in oil appreciably influence the characteristics of the oil. Some petroleums give little evidence of any wax content until the oil temperature is reduced, at which time the wax will start crystallizing, making the oil more viscous. Others contain wax in such amounts that the methods of oil production cause the wax to crystallize out. Thus rod waxes are deposited in the well casings, tubing, and open hole while the oil is coming to the surface from the oil reservoir.

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Due to the change in the nature of the crude oil as it leaves the oil sand and comes to the surface, it becomes less efficient as a solvent and the higher-melting-point waxes, being less soluble than lower-melting-point waxes in a solvent, crystallize out of the oil. Reistle\textsuperscript{32} discusses this and other factors that control the separation of wax from crude oil during production. Thus some waxes are separated from the oil in the process of taking the oil from the oil reservoir.

Crude oil usually is run to storage tanks for various periods before being processed in a refinery. During this time some of the lighter constituents of the oil are apt to be lost by evaporation, and the ability of the oil to act as a solvent for waxes is reduced. If the wax content of the oil is sufficient some of the wax will deposit out in the storage tanks. Another, and probably more important, factor in the separation of wax from crude oil is any reduction in temperature of the oil. Thus, oil left in storage during periods of lower temperature may deposit wax, the amount depending upon the nature of the oil, the amount of wax, and the melting point of the wax in the oil.

Pipe lines transporting crude oil from wells to storage and refineries may also have waxes deposited in them. The conditions of temperature and decreased solubilities described above are also responsible for this wax separation. These waxes are considered nuisances and sources of trouble, and very little if anything is done commercially with them.

\textbf{AT REFINERIES}

After the crude oil arrives at the refinery it is processed into various commercial products. The number of different products available depends upon the nature of the crude oil and the processing equipment at the particular refinery. Only a relatively small number of refineries in the United States have the necessary equipment and process various crude oils into practically all of the more common products available from crude oil. Of about 475 petroleum refineries in the United States, only about 75 plants manufacture wax, and of these only a small fraction furnish all melting-point grades of paraffin wax.

Crude oil is subjected to a distillation process and separated into various fractions. The boiling points of the waxes in crude oils are such that the waxes appear in the fractions ranging from heavy gas oil to the heavy lubricant fractions of the crude oil. The lower-boiling-point waxes thus are associated with the lighter oils, and as the oils become heavier the waxes have higher boiling and melting points. The paraffin wax usually is separated from the crude in the wax-distillate cut. This cut or fraction is by no means standardized, and its characteristics are apt to depend upon the crude oil from which it is taken and the equipment with which it is produced. This wax-distillate cut usually is made as heavy as possible. The controlling factor in the production of the wax distillate is the ability subsequently to chill and press the distillate in such a manner as to remove the wax readily from the oil. Due to the type of equipment


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and method of processing, as well as the nature of the crude oil, the character of the wax distillate may vary widely.

Once a satisfactory wax distillate is available the general procedure for removing its wax content, which might be around 10 percent, is somewhat as shown in the generalized flow chart, figure 4, or as follows: The distillate is taken from the refinery run-down tankage and cooled to the desired temperature by pumping it through a number of heat exchangers and then through chilling machines. Cold calcium chloride brine is circulated through the chillers, or
ammonia is expanded directly into them. Heat from the distillate is absorbed by the chilling medium, and as the distillate becomes colder the wax begins to crystallize out of the oil. When the distillate has been chilled sufficiently, the amount of chilling depending upon how low a cold test is desired on the finished oil, the distillate, now a slushy mixture of oil and wax crystals, is pumped to a filter press, where under the influence of pressure the oil is forced through canvas sheets, and the wax, unable to penetrate the sheets, remains behind, forming a cake. These filter presses thus cause the separation of the wax and most of the oil. The oil passing through the filter plates is referred to as the pressed distillate, and since its wax content has been sufficiently reduced it is available for further processing into lubricating-oil stocks. The wax left in the filter presses is termed "slack wax" and represents about one-fifth of the wax distillate; it is still composed of approximately one-half wax and one-half oil.

When the distillate is chilled to the desired low temperature and all the crystallized wax removed in one filter-press operation, such procedure is referred to as single pressing. Quite often the filter pressing is done in two steps and is then referred to as double pressing. The distillate will be chilled to a given temperature and filter pressed and the wax, which has crystallized at that temperature, removed from the distillate. The oil is chilled further and the additional wax crystallizing out of the oil removed by a second filter-pressing operation.

Somewhere in the processing cycle of finished waxes an acid treatment is given the material. This treatment may be given the distillate, the slack, the scale, or the finished waxes; consequently, while it is given the slack wax in figure 4, there is no standard place in the wax-manufacturing cycle where the acid treatment is applied.

The slack wax must be processed further to remove its oil content before the wax has any commercial value. The slack wax is removed from the presses and conveyed to a tank where it is melted. The liquid slack wax is then pumped to sweaters, where the wax is chilled into a more or less solid mass and then slowly heated. During the latter operation the oil and lower-melting-point waxes gradually leave the fibrous mass of higher-melting-point waxes. This sweating process is continued until the wax left in the sweaters is of the desired quality. It is then melted, usually purified by filtering, molded into cakes or "chipped" into thin sheets, and barreled, crated, or sacked for shipment. Large wax consumers in the United States may also receive their wax from the refiner in tank cars.

Solvents are playing a part in the removal of paraffin wax from petroleum fractions and probably will continue increasingly to do so. The solvents used depend upon their relative greater solubility for the oil than for the wax at reduced temperatures. A number of solvents dissolve both the oil and wax at or above room temperatures but lose their solubility for wax as the temperature is lowered, until at 0° F. the wax is practically insoluble while the oil continues to remain in solution. This property is used in removing wax. Benzol and acetone mixtures have been used since 1927 in removing wax from wax distillates.\[^{35}\] The benzol-acetone solvent, about 35

\[^{30}\] See footnote 30.
percent acetone and 65 percent benzol, is mixed with the wax distillate in a ratio of about 3 or 4 parts of solvent to 1 of distillate. This mixture is chilled to a little below the temperature of the cold test desired on the finished oil, at which stage the oil remains in solution in the solvent while the wax has crystallized out. The mixture is pumped through a filter press which removes the wax and permits the oil solution to pass through. The solvent is removed later from the oil by distillation, leaving an oil which is free from wax that will crystallize out at a temperature little above that at which the oil-wax solvent mixture was filtered. The wax from the press is freed of any solvent by distillation, leaving a slack wax which is ready for sweating. Propane, under pressure to keep it liquid, also is used as a solvent for removing wax from the oil. The use of propane will be described later.

**WAX DISTILLATE**

**DISTILLATION OF PETROLEUM FOR WAX-DISTILLATE Manufacture**

Under the present methods of production practically all of the paraffin wax is obtained from the wax-distillate fraction of the crude; however, there is apt to be a large amount of wax in the fractions heavier than wax distillate. This wax is of such crystalline nature that it cannot be separated from the oil and recovered with the equipment now in use for paraffin-wax manufacture. A possible exception to this is the wax in some slop-wax-distillate cuts. Slop-wax-distillate cuts, if made at all, are usually only a few percent in volume of the oil charged and, if the wax is to be removed, must be specially processed. The method of distillation employed to separate the wax-distillate fraction from the remainder of the petroleum plays an appreciable part in the characteristics and yields of the wax obtained from the distillate and the more important factor of the quality and yields of lubricant stocks in the distillate. Moreover, very little, if any wax distillate is manufactured for the wax it contains. However, in its manufacture the character of the wax distillate is governed by the ability of the refiner to remove a large portion of its wax content. What the refiner wants in the wax distillate is its lubricant-stock content. The manufacture of wax can be said to be secondary or in some cases a necessary evil. Occasionally conditions may warrant the processing of distillates solely for the wax content.

From the foregoing it is not to be construed that wax cannot be manufactured advantageously under proper conditions. Of necessity it must be removed from the lubricant stocks. The impure wax so removed can be processed with little additional equipment, mostly sweating equipment, into waxes of commercial value. Wax has commanded a low price for a number of years (see table 1, p. 1). Refined waxes, however, have ranged from 1 to 3 cents more per pound, depending upon the grade. At these low prices many refiners have still been able to show an earning from their wax plants. Since the middle of 1933, with advancing wax prices, much more interest has been shown in the manufacture of wax.

A wax-distillate fraction can be obtained from most wax-bearing crudes by any one of several distillation methods, namely, batch,
WAX DISTILLATE

Continuous, vacuum, coking, or rerun. The quality and quantity of wax from a distillate manufactured by these methods can be made very similar if adequate equipment, especially for fractionation, is employed.

**BATCH METHOD**

Because of the equipment employed, batch operation generally tends to yield a poor quality and yield of wax distillate, and the wax from this distillate will not have the quality and yield (in reference to the crude oil) that might be had from distillates produced by other methods. It is not to be inferred, however, that any distilling method ultimately produces an inferior quality of wax. Regardless of the quality of the crude wax from the distillate it usually can be processed by repeated sweatings (provided it can be sweated), acid treating, and filtering to produce a similar grade of wax to that made from a distillate obtained by any other distilling method. With batch distillation the yield of scale or finished waxes is apt to be low, and the wax probably will require more treatment before being finished. With batch distillation it is more difficult to make a sharp separation between the maximum wax-distillate cut and subsequent cuts. Consequently more waxes that press poorly, if at all, usually find their way in with wax that is pressable. Moreover, there are apt to be more coloring materials and wax-crystallizing inhibitors in the wax-distillate cut than in a similar cut made under different processing methods. The presence of the poorly pressable waxes reduces the amount of pressable wax distillate obtainable and thus decreases the wax yield. Coloring materials and tars in poorly made distillates are a detriment to sweater operation and yields. They are likely to settle out in the pans and hold oil and, when the wax in the pans is melted down at the end of a sweat, come off with the wax and tend to give it too high an oil and moisture content. The presence of coloring materials requires the use of more sulphuric acid treatment or filtration through fuller’s earth or bone char to give the proper color and color stability to the finished wax.

**CONTINUOUS METHOD**

Continuous distillation methods using good fractionation permit the manufacture of good-quality, pressable wax distillate because of better adaptability of fractionating equipment to this method of distillation than to the batch method. With the ability to separate the lubricating-oil fractions of the crude oil into sharp cuts, it is evident that a pressable wax distillate can be made which contains (1) most of the waxes in such form that they can be handled in the presses and (2) a minimum amount of those waxes that do not press readily. Consequently, an increased quantity of wax should be obtainable. The crude slack wax should also contain less oil because its crystalline nature is such that the oil can pass through the wax mass more easily. The coloring materials in the wax should be reduced because better fractionation has decreased the inclusion of heavier fractions in the wax-distillate cut. Continuous distillation includes that of both single flashing and multiple flashing of the crude oil. In single-flash operation the crude oil is heated to the highest temperature desired
and then vaporized, the wax-distillate cut being separated from the other material in the crude oil by fractionation in several trays of a bubble tower or some similar fractionating column. In multiple-flash operation the crude oil is heated in successive stages, during one of which the wax distillate is separated from the other lubricating stocks by means of a fractionating tower in which the wax-distillate vapors can be subjected to more efficient fractionation because the number of different hydrocarbons present in the tower is less than in the wax-distillate section of a single flash tower and their composition is more nearly that of the desired fraction to be taken from the tower, thus approaching more closely an ideal tower. However, in actual practice, generally speaking, the yields and quality of slack wax from either method of flashing are apt to be the same.

**VACUUM METHOD**

Vacuum distillation methods are used essentially to produce the lubricating oils in the heavier fractions of the crude oil. The use of reduced pressures during distillation of these fractions permits the use of lower temperatures in distilling the oil. Thus, the lubricating oils can be manufactured without as much danger of overheating the oil as when ordinary pressures are used, and the disintegration of some of the wax and the oil by cracking, with the production of undesirable materials, can be greatly reduced. The effect of vacuum on the yields and quality of the wax distillate is thus to increase them. The increased yield of wax distillate from cutting deeper into the crude oil increases the yield of wax. Moreover, with less undesirable materials in the oil and wax, less refining is necessary.

**CORKING OR RERUN METHOD**

Rerun distillation usually plays a part in the manufacture of wax distillates when any of the previously mentioned methods of distillation do not include sufficient fractionation to make a well-cut wax-distillate fraction or when the distilling and fractionating equipment is so operated that the lubricant distillates require additional processing before the wax can be removed from them. During the processing of crude oils that are run to cylinder-stock bottoms it may be imperative to rerun the wax-distillate fraction. In running to cylinder-stock bottoms it is desirable to protect them from excessive heat treatment. This may result in a poorly prepared wax-distillate fraction. If this fraction is made rather heavy or subjected to poor fractionation, rerunning may be necessary. The same is true of the wax distillate from crude oils that are run to flux bottoms. Crude oils are apt to be run to flux bottoms when the latter can be made into road oils or converted into asphalts. As a result, it is undesirable to subject the oil in the still to cracking temperatures, and the wax distillate is likely to be of poor pressing quality. Under such conditions of cylinder-stock and flux-bottom distillations the wax-distillate fraction is rerun, during which it is subjected to slight "cracking." The distillate is cracked just enough to permit the contained wax upon chilling to crystallize readily into such form that it is removable by filter-pressing.
When crude oils are run to coke it is usually unnecessary to rerun the wax-distillate cut because the wax in the distillate can be prepared properly for pressing. A heavier cut following the pressable wax distillate often is taken from the stills and rerun to get its wax in pressable condition. Rerunning this heavier cut furnishes a pressable wax-distillate cut that contains heavier lubricating-oil fractions than the usual wax-distillate cut. The rerunning operations on wax distillate, which are usually carried to coke, are undesirable from the lubricating-oil standpoint because the quality and quantity of oil are decreased in consequence; however, they are desirable from the wax-removal standpoint. The filter presses in use at refineries are of such design that only the large, more or less well-defined, crystal forms of wax can be easily separated from the oil. The heavier portion of the wax distillate and subsequent lubricating-oil cuts contain wax of very small crystalline structure—so-called "amorphous" wax. Subjecting this oil to a slight thermal decomposition or cracking changes the crystalline form of the wax so that it can be removed more readily by the pressing equipment and, when removed, is of a quality easier to process. Thus rerunning certain heavy wax distillates produces increased yields of a better-quality wax. It is not to be concluded that cracking increases the wax content of a crude oil. On the contrary, cracking will decrease the total wax in a crude oil, as it will decompose wax. Considering only the crystalline wax that can be produced by the refinery pressing and sweating operations, the use of slight cracking will produce more wax than if it were not employed. Cracking results in an increase of wax that is readily removable by filter-pressing, and it is the slack wax which is the base for the manufactured paraffin wax. If total wax contents were figured by solvent methods, it would be found that the content would be decreased by cracking.

CHARACTERISTICS OF GOOD-PROCESSING WAX DISTILLATE

The main characteristic of a good wax distillate is the nature and crystallizing character of the wax contained in the oil at the temperature at which it is desired to filter the cold oil-wax mixture. There is little in the appearance of a distillate to indicate whether its wax content will or will not be in a desirable crystalline form for ready removal by filtering. The color of a distillate bears no relation to its wax-removal qualities. It may be light or dark, yet its wax may be removed easily in both cases. Light-color distillates result either from the use of little cracking or good fractionation in their manufacture. As the amount of cracking is increased the distillates become darker until some are quite black. This color results either from finely divided carbon particles in the distillate or from the polymerized products formed in the cracking operation. Generally, little fractionation is applied to stills used for cracking wax distillate; consequently, such distillates usually are dark. Fractionation is applied to distillates made under conditions of continuous operation and produces a lighter-color oil for a given viscosity due to the elimination of some of the asphaltic and polymerized materials from the distillate. While color may mean little as to the relative ease of removing the wax from the oil, yet it does have a
bearing on the subsequent purification of both the wax and oil. Dark distillates produce waxes that usually require treatment to decolorize them. If the coloring matter is small particles of carbon, they are likely to plug the filter presses, making it more difficult to press such distillates. Other coloring materials act as wax-crystalizing inhibitors and as such are a detriment to filter-pressing. They must be removed by washing or acid treatment before the wax can be finished. The pressed oil also will require more chemical treatment to bring the darker oil to proper color.

**CONTROL METHODS USED FOR WAX DISTILLATE**

The industry uses a number of tests by which the wax distillate can be analyzed as to its pressability, which is the main factor to be considered because most refiners operate to obtain the maximum wax distillate consistent with their methods of wax-plant operation. This means that the distillate usually is cut to the point where it is still possible to press it but where any increase in the heavy ends would make it difficult and impracticable to put the distillate through the presses. The methods used by refiners to control the quality of the distillate may be listed as follows: Gravity, still temperatures, viscosity, photomicrographs, and distillation range. These methods are studied by the individual refiner with respect to the equipment being used, the crude oil being processed, and the manner in which the distillate is being produced, until one or more of the tests gives sufficient information on which he can rely to obtain a pressable distillate. In some cases the operator of the equipment samples and tests the oil stream as it comes from the distillation unit. In others, samples are taken from the distillate stream and tested in the plant laboratory. There is much to be said in favor of the operator testing the wax-distillate stream and being held responsible for the proper operation of the stills to produce a distillate of the desired quality.

**GRAVITY**

A gravity determination is usually the first information obtained as to the quality of a wax distillate. The operator takes samples of the distillate at regular intervals as it comes from the still. Previous information on the pressability of the distillate and sweat-ability of the slack wax from laboratory tests has indicated the gravity range that can be included in the wax-distillate cut for the particular crude being processed. Gravity alone is relied upon little as a measure of the wax-distillate quality, and usually it is supplemented by one or more of the tests that are discussed later.

The gravity of the pressable distillate from Pennsylvania crudes is usually around 34° A. P. I. With continuous operation, using shell stills, the stream from the stills is cut into wax distillate at about 37° to 39°, and all other streams down to 32° are run into the distillate. All streams below approximately 32°, if any, are run to slop until specifications are met on the residuum. The raw distillate will average about 35° to 36°, which when rerun will yield around a 34° pressable distillate. When the distillate cuts are being taken off, steam may be used in large amounts to protect the cylinder-stock bottom. When this is done it is usually necessary to rerun
the distillate or a part of it (the lower-gravity A. P. I. part when the distillate is made up of a number of streams from several stills). If not as much steam is used, a pressable distillate cut of about 34° can be obtained. With pipe stills and efficient fractionating equipment this gravity could be lowered somewhat, providing the cylinder-stock yield would not be affected.

The gravity of the pressable distillate from average Mid-Continent crude is lower than that from Pennsylvania crude and runs about 30° to 31° A. P. I. The streams from the various towers or stills in a continuous hook-up with good fractionation, from about 33° down to 28° to 29°, are put into pressable wax distillate. If any lower-gravity streams are included with these the distillate usually is rerun. Depending upon operating conditions, streams as low as 24° may be included in the raw distillate. One refiner running batch and to coke cuts the stream from 32° to 24° into raw wax distillate. The distillate is rerun to a 28° pressable distillate. If the production of wax is of some importance at a plant, the wax distillate usually will be of the higher gravity. The lower-gravity distillates are rerun to a pressable cracked-wax-distillate cut with a gravity of about 30°.

Slop-wax distillate, or the cut run off after pressable wax distillate is cut out at approximately 28° to 29°, may be run down to gravities ranging from 18° to 24° A. P. I. or higher. Rerunning is necessary to make this distillate pressable and usually is done in a batch operation. The stream from this distillation is cut into cracked wax distillate from about 34° to 35° down to 28° to 29° with the whole distillate averaging 31° to 32°.

Pipe-still and bubble-tower installations produce pressable distillates of about the same gravities, namely, 30° to 31° A. P. I. The slop cuts from such installations generally are rerun before the wax can be removed.

STILL TEMPERATURES

Closely allied with controlling the wax-distillate stream by gravity is its control by still temperatures. One refiner running Mid-Continent crude by the batch method and to coke cuts into wax distillate at approximately 660° F. and continues the wax-distillate stream until about 800° F. when the distillation is almost complete. Should the distillate, of around 30° A. P. I. gravity, 70 viscosity, Saybolt Universal at 100° F., and 250° F. flash, become difficult to handle in the wax plant this trouble is rectified by cutting the stream into and out of the distillate at different temperatures. At one refinery where paraffin-distillate streams are taken from four shell stills equipped with towers in a continuous battery, the stills are fired at 630° to 685° F. with increasing steam quantities of 7.5, 10, 12.5, and 15 pounds per barrel of total wax distillate taken off the stills. However, these temperatures and steam quantities may be changed somewhat so as to maintain a balance between them and obtain the desired quality of distillate. In general, temperatures of 600° to 700° F. are required to distill off the wax distillate, the temperature used depending upon the amount of steam employed. If the throughput on a battery of stills is increased it is usually necessary to increase the temperatures on the various stills in order to get off the distillate.
A 500-barrel-per-day increase when 6,000 to 7,000 barrels are run will require still temperatures several degrees higher. On shell stills in a continuous battery vacuum may be applied to those stills producing the lubricating-oil distillates. Where lubricant stocks are taken from good lubricant-producing crude oils at one refinery, the distillate stills are kept at about 580°F., and the heavier stocks are brought over with the use of more steam in those stills.

On cracking or rerunning distillations one refiner fires at approximately an increase of 25° per hour to 600° F. when the cracked wax distillate starts over; from 600° to 650° the stills are fired at an increase of 10° F. per hour; and from 650° to off, which is never over 685° F., at an increase of 5° F. per hour. If it were desired to crack the distillate hard the temperature would go to 700° to 720° F., but such operation would be detrimental to some of the lubricant stocks. Another refiner employing a continuous-shell-still battery and a bubble tower on the wax-distillate fraction maintains still temperatures on Oklahoma City and Seminole crude oils of 630° to 640° F., and the tower top is held at 540° for the Seminole and 570° for the Oklahoma City oil. In addition, 65 pounds of steam are used in reducing 1 barrel of Oklahoma City crude oil to stock and 45 pounds of steam in reducing 1 barrel of Seminole crude oil to fuel oil.

When distillates are made from pipe-still units employing single-flash systems the charge is heated from 750° to 800° F. At the point of distillate take-off from the tower the temperature will range from 550° to 575° F. In vacuum units the charge will be heated to about the same as above, but the temperature in the tower at the point where the wax distillate is taken off will be a little above 500° F.

**Viscosity**

Probably the most universal test applied to wax distillates to control their pressing quality is that of determining their viscosity. As stated before, most refiners operate to obtain the maximum of pressable distillate. Consequently the point of maximum viscosity is given most consideration. A review of the operation of most of the wax-manufacturing refineries in the United States shows the average maximum viscosity of the pressable distillate to be 70 seconds Saybolt at 100° F. This viscosity, however, is not the maximum obtainable for a pressable distillate. The viscosity of the distillate depends largely upon the amount of fractionation to which it has been subjected. With stills operated batch and not equipped with towers some refiners find it difficult to get a pressable distillate above 65 seconds in viscosity. Moreover, with shell stills operated continuously and equipped with efficient fractionating columns viscosities as high as 90 seconds can be obtained and the distillate still be pressable. An interesting example of the effect of fractionation on the possible increase in viscosity of a distillate is given here. A wax distillate was produced by one refiner from several stills in a battery of shell stills. The distillate stills were equipped with very small towers containing a few baffle plates, inadequate for the size of the still and of doubtful value. The distillate from these stills had to be rerun to obtain a 48 to 52 viscosity, 30° A. P. I., pressable distillate. Later
the same stills were equipped with an efficient modern bubble tower of sufficient capacity. The distillate produced overhead from this tower is pressable, of 80 to 90 seconds viscosity and a gravity of 30°.

The effect of proper fractionation was most beneficial.

It might be well to mention three additional benefits derived from the use of good fractionation. First, the increased viscosity of the distillate permitted an increase of lubricant stocks; the old distillate (pressed) yielded 20 to 25 percent of 200-viscosity stocks, and the new distillate (pressed) could yield 45 percent of the same viscosity. Actually, however, 33 percent of a 250- to 290-viscosity stock was made. Secondly, the amount of chemical refining was reduced by the better fractionation. Eighteen pounds of acid were used per barrel of reduced, raw pressed distillate of 200 viscosity, made under the old set-up. With the installation of a satisfactory tower only 14 to 15 pounds of acid per barrel are used on a 330-viscosity, reduced, raw pressed distillate. Third, the filter yield per ton of fuller's earth, when finishing the scale wax from the wax distillate, was greatly increased from about 10 to almost 40 barrels of scale per ton of earth.

With pipe stills and bubble towers of efficient design and operation it is admittedly easy to produce pressing distillates of 90 viscosity. It is stated that viscosities of 100 or over can be obtained on pressable wax distillates with the use of efficient fractionating equipment. However, in a survey of most of the wax-manufacturing refineries it was found that most of the wax distillates were produced from shell stills either in batch or continuous operation and that it was necessary to hold the viscosity of the distillate down to about 70 if the distillate was to be pressable.

PHOTOMICROGRAPHS

One method of controlling the pressable quality of the wax distillate is to observe the wax crystallization in a sample of the distillate under a microscope and, if desirable, to photograph the crystals formed under definite and set conditions. The method employed consists of using a microscope of about 50 to 100 magnification equipped with Nicol prisms and a water-jacketed hot stage that permits keeping the distillate at various temperatures so that the formation of the wax crystals can be observed as the temperature of the distillate sample is decreased. The temperature of the oil sample is reduced at a definite rate, and when the temperature is reached that is judged proper for comparison of the crystallized wax in the various samples the operator notes the appearance and characteristics of the wax crystals. A photomicrograph may be taken and the picture kept for reference. The appearance of the crystals will differ according to the characteristics of the oil in which the wax crystallizes. A standard series of photomicrographs covering all conditions of wax crystallization can be prepared; any observed crystallization can be referred to this for comparison. After some study and correlation of the wax crystallization with the manner in which the distillate presses, a certain kind of wax crystallization is set up as a limiting type above which the distillate will not press satisfactorily and below which it is pressable. With this decided the operator need only observe the type of crystalliza-
tion and compare it with the limiting type to determine whether the distillate is pressable or not and how it shall be controlled, whether it is possible to cut deeper or necessary to decrease the distillate cut, or whether to cut certain streams from the still or stills in or out of wax distillate.

This method of control was evolved primarily by members of the research department of the Tide Water Oil Co., who have developed it to the point where the plant operators use the method as their main control on the distillate. Davis and Campbell have described excellently the use of photomicrographs in controlling the quality of wax distillates. Figure 5, taken from their work, shows a series of photomicrographs that might be used for control purposes. They were made as follows: An Oklahoma crude oil was topped to remove all light oils up to the normal wax-distillate fraction. A distillation of the topped crude oil was made under 3 mm absolute pressure, cuts being made every 2.2 percent on the original crude. These cuts were combined and the physical characteristics as shown in table 2 determined. Photomicrographs were made of the crystal structure of various combined fractions.

**Table 2.—Physical characteristics of consolidated fractions**

<table>
<thead>
<tr>
<th>Combined cuts</th>
<th>Percent of crude</th>
<th>Distillation end point under 40 mm pressure, °F.</th>
<th>Viscosity at 100°F.</th>
<th>Combined cuts</th>
<th>Percent of crude</th>
<th>Distillation end point under 40 mm pressure, °F.</th>
<th>Viscosity at 100°F.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 to 6</td>
<td>13.2</td>
<td>572</td>
<td>62</td>
<td>1 to 10</td>
<td>22.9</td>
<td>637</td>
<td>90</td>
</tr>
<tr>
<td>1 to 7</td>
<td>15.4</td>
<td>590</td>
<td>67</td>
<td>1 to 11</td>
<td>24.2</td>
<td>652</td>
<td>100</td>
</tr>
<tr>
<td>1 to 8</td>
<td>17.6</td>
<td>698</td>
<td>71</td>
<td>1 to 12</td>
<td>26.4</td>
<td>676</td>
<td>111</td>
</tr>
<tr>
<td>1 to 9</td>
<td>19.8</td>
<td>618</td>
<td>81</td>
<td>1 to 13</td>
<td>28.6</td>
<td>702</td>
<td>125</td>
</tr>
</tbody>
</table>

In figure 5 the crystal structure shows a uniform diminution in size of the plate crystals through the series 1 to 11. Series 1 to 12 and 1 to 13 picture distinct malicrystalline forms. The scale shown is a photomicrograph of a millimeter scale, the smallest subdivision being 0.01 mm. A study of the pressability of distillates correlated with their crystal-structure characteristics will serve as a basis for judging later the quality of distillates in the plant.

Figure 6, table 3, and the following discussion also are taken from the work of Davis and Campbell. Figure 6 shows the crystal structure of six commercial pressing stocks from various Mid-Continent refineries. The scale shown is the same as that in figure 5. Stock 1 shows excellent large-plate crystals, considering the high end point. This distillate usually is diluted to a viscosity of 52 to 55 seconds at 100°F, for pressing. Stock 2 shows excellent crystal structure. Stock 3 gives a good filtering rate with a high neutral yield. It is diluted to a viscosity of 55 to 60 seconds at 100°F, for pressing. Stock 4 is a relatively low-wax-content distillate showing fair crystal formation and giving good press capacity and neutral

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Figure 5.—Wax crystals from consolidated cuts.
Figure 6.—Characteristic commercial paraffin distillates.
yields. Stock 5 is of the mixed-crystal type, showing poor fractionation. The crystal structure shows a scattering of fair-size but poorly formed plates with a background of minute plates. This distillate gives low pressing rates and low neutral yields. Stock 6 is a poorly fractionated product giving very small, 0.01- to 0.05-mm, crystals. The pressing rates and neutral yields are very low. This distillate is diluted to a viscosity of 64 to 68 seconds at 100° F. for low-temperature pressing and pressed straight for high cold-test oils. Table 3 gives some of the physical tests on these distillates.

Table 3.—Physical characteristics of various commercial wax distillates

<table>
<thead>
<tr>
<th>Pressing stock no.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gravity, ° A. P. I.</td>
<td>34.2</td>
<td>32.0</td>
<td>31.0</td>
<td>31.4</td>
<td>31.7</td>
<td>31.6</td>
</tr>
<tr>
<td>Flash point, ° F.</td>
<td>80</td>
<td>170</td>
<td>300</td>
<td>280</td>
<td>320</td>
<td>315</td>
</tr>
<tr>
<td>Viscosity at 100° F.</td>
<td>68</td>
<td>70</td>
<td>94</td>
<td>95</td>
<td>88</td>
<td>75</td>
</tr>
<tr>
<td>Cold test (solid point), ° F.</td>
<td>66</td>
<td>65</td>
<td>74</td>
<td>42</td>
<td>56</td>
<td>60</td>
</tr>
</tbody>
</table>

Distillation range at 40 mm, ° F.:
- Initial boiling point: 256 450 256 312 306
- 10-percent point: 344 312 476 395 394 418
- 30-percent point: 465 330 493 416 420 445
- 50-percent point: 494 473 509 436 453 469
- 70-percent point: 511 496 526 461 479 491
- 90-percent point: 527 518 541 490 503 513
- 90-percent point: 544 539 557 524 523 533
- 70-percent point: 564 561 573 557 543 554
- 50-percent point: 588 585 593 590 562 580
- 30-percent point: 622 611 615 623 590 616
- End point: 648 632 637 643 625 652

<table>
<thead>
<tr>
<th>Wax content, percent</th>
<th>8.75</th>
<th>6.75</th>
<th>7.66</th>
<th>1.68</th>
<th>5.77</th>
<th>4.80</th>
</tr>
</thead>
<tbody>
<tr>
<td>Melting point of wax, ° F.</td>
<td>118</td>
<td>123</td>
<td>123</td>
<td>119</td>
<td>124</td>
<td>123</td>
</tr>
<tr>
<td>Percent raw 220 viscosity neutral from pressed distillate</td>
<td>50</td>
<td>48</td>
<td>50</td>
<td>51</td>
<td>33</td>
<td>39</td>
</tr>
</tbody>
</table>

The use of photomicrographs has been discussed with a number of refiners that manufacture wax, and their comments are somewhat as follows: It should be a good method because it depicts what will take place when the oil is chilled for wax crystallization, if a refiner were assured of the same type of crude oil for processing. However, where the crude oil entering a plant is always changing, the characteristics of wax crystallization will differ due to the nature of the oil, and the still operator will be at a loss unless he has a guide as to the type of crystallization to be expected from each oil. Some refiners feel that it is unnecessary to resort to this method of testing when a viscosity determination affords them a satisfactory means of control. Moreover, they feel it would be difficult to get their plant operators to be competent in judging small differences in the type of wax crystallization. It would seem to the writer that, since this method of control actually provides direct information on the nature of the wax crystallization in a distillate, it should be the most useful test for judging the quality of a wax distillate.

**DISTILLATION RANGE**

The boiling-point range of the hydrocarbons in a wax distillate is used as a guide to the quality of the distillate, primarily, however, as a guide to the yield of lubricating oil. The range of the lighter fractions is not as important as is the range of the heavier
fractions in determining the ease with which the distillate can be put through the presses and its wax removed. Laboratory distillation of the distillate usually is carried on under reduced pressure, as otherwise cracking might occur, in which case the distillation would have little value. The distillation range alone may not give a satisfactory test for the distillates pressability, as some refiners find that of two distillates with the same boiling-point range one might exhibit very poor qualities in wax-plant operations. Apparently one or more of the other characteristics—viscosity, gravity, cloud point, and flash and fire points—needs to be considered with the boiling-point range. Davis and Campbell\(^{35}\) state that, "in addition to the yield of neutral oil, the distillation range of a pressing stock for a given degree of fractionation determined the crystal structure of the distillate."

On average Mid-Continent crude the distillate of about 70 seconds viscosity and 68 cloud will have an initial boiling point of approximately 310° F. and an end point of 575° F. at 10 mm absolute pressure. These temperatures when converted to atmospheric pressures would mean an initial boiling point of about 570° and an end point of about 870° F.\(^{36}\) It should be evident that similar viscosity distillates may have quite different boiling-point ranges depending upon how wide or narrow the cut is made. Table 3, page 25, gives the distillation range of several wax distillates, and their pressability is discussed on page 24. Samples of wax distillates analyzed by the writer several years ago showed wide temperature ranges between the 90-percent and end-point distillation points. Distillates that pressed well had ranges comparable to the first four stocks given in table 3. Distillates from batch operation had ranges of temperature greater than those for stocks 5 and 6, in some cases over twice as great. Much difficulty was experienced in removing the wax from these distillates. One refiner, processing Mid-Continent crude, states that if the end point of the distillate does not exceed 570° F. at 10 mm (about 650° F. at 40 mm) the distillate will process satisfactorily. Another refiner uses the 95-percent point instead of the end point. The distillate is obtained from a continuous battery of shell stills equipped with partial condensers, and generally on Mid-Continent crude, if the 95-percent point does not exceed 550° F. at 10 mm (about 630° F. at 40 mm), the wax-plant operations can be carried on satisfactorily. This refiner states that under certain operating conditions this point may vary almost 50° F. either way and the distillate still be satisfactory, such other characteristics as viscosity, color, and appearance permitting this variation. The distillation range should be a pertinent test to apply to a distillate in judging its quality.

**TREATMENT GIVEN WAX DISTILLATES**

Wax distillate may or may not be given some treatment (hot-water wash, sulphuric acid treat, or filtration) before it arrives at the wax plant for chilling and pressing. The distillate from the stills is kept warm in tanks equipped with steam coils to permit easier handling, from which it is usually sent to the wax plant for wax removal. In

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35 See footnote 34.
some cases, however, it is treated before this is done. Any treatment considered necessary for the distillate usually can be traced to a poorer quality of distillate produced as a result of the method of processing to which the oil had been subjected. Clean, clear, not easily oxidizable distillates, such as are apt to be produced from vacuum units with good fractionation, are considered by the majority of refiners not to need treatment before chilling and pressing. Distillates that have been cracked rather heavily are full of carbon particles and are dark or darken rapidly after coming from the still, and ones that are murky and cloudy from water particles held in the oil coming from the steam used in the distillation are sometimes considered to need treatment.

However, in one or two cases the writer has noticed that distillates that were clear, clean, and of good color were treated before being chilled and pressed instead of subsequently in the wax-plant operations. One refiner felt it best to treat even a very good distillate for the following reasons: (1) The distillate throughput in barrels per press is increased from 5 to 10 percent. (2) Less press-blanket cleaning is required. Untreated distillates caused press-blanket cleaning every 2 months or so; treated distillates extend the period to 6 months or more. (3) Storage and transfer tanks have less tank bottoms to be cleaned. (4) Repairs to blankets and presses are considerably less. (5) One to two percent larger lubricating-oil yields are obtained at the pressed-oil reducing stills from the treated than from the untreated distillate. In acid treatment of a distillate the gas-oil fraction and the foots-oil fraction unquestionably receive unnecessary treatment. These fractions are disposed of primarily as cracking stock and in fuel oil. However, the resulting increase in treating cost is said by this refiner to be outweighed considerably by the total savings accruing from the five items mentioned.

Average and poor distillates are treated to take out the fine particles of carbon that clog the press blankets, reduce the throughput of presses, and otherwise increase the costs of pressing. Good, clean distillate makes all subsequent processing easier and tends, as one refiner said, to offset slips and the human element in subsequent operations. When the distillate is treated no treatment is given the slack, scale, or refined waxes except filtering the final product, if necessary, to obtain the desired color. If the distillate is not treated, it is customary to treat the slack, scale, or refined wax and to filter the finished plant product through fuller's earth or bone char. The neutral lubricating-oil stocks contained in the wax distillate are treated with acid before finishing whether or not the distillate is treated. Refiners treating the wax distillate feel that such procedure reduces the amount of acid necessary to treat the neutral stock.

Wax distillate usually contains water unless it remains in storage for several days, during which time the water may settle out. For this reason the distillate when treated with sulphuric acid is contacted with the acid in two or three stages. Five pounds of 66° B. sulphuric acid per barrel of distillate can be considered an average figure for the total acid used in treating wax distillates. Quantities as low as 3 and as high as 8 pounds are used, depending upon the quality of the distillate. Amounts above 6 pounds, however, are
unusual. The first stage, or "dump", is called a "water cutter", about 1/4 to 1 pound of acid being used per barrel of distillate. This initial small amount is forced into the agitator full of distillate, after which it is agitated thoroughly and mixed with the oil by blowing air up through the oil and acid. The cutter acid unites with any water in the oil, so that when the subsequent larger acid dump makes contact with the oil there will be no water present to dilute the acid and reduce its effectiveness. If the distillate has been well settled it may not be necessary to add the acid in more than one dump. The main acid treat of 4 to 5 pounds per barrel of oil is charged to the agitator, and air is blown through the charge until the oil has been well contacted with the acid. After blowing, the contents of the agitator are allowed to stand for about 2 hours, and a sludge settles out which is withdrawn. During this operation some refiners prefer not to have the temperature of the distillate exceed 90° F. Orifice mixers have been used to contact the distillate and acid in lieu of agitating the mixture with air, and it is reported that the use of only one-half the customary amount of acid is necessary.

After removal of the sludge the distillate is sprayed with water to wash it free of fine acid particles. The distillate may be left in the same agitator or transferred to another to be neutralized. Care must be exercised to insure well neutralized and treated distillates if excessive corrosion of press plates and rotting of press blankets are to be avoided. Either caustic soda or soda ash is used as the neutralizing agent. The caustic solutions range in strength from 2° to 4° B., strengths of 3° B., generally being used. The weak caustic solutions are used to avoid emulsifying the distillate. Caustic strengths above 4° B. are considered unsatisfactory. It seems to be the opinion that the more fine acid particles and sulphonated products left in the distillate as a result of the acid treat the more difficulty can be expected from emulsification in the neutralizing wash. Some refiners prefer soda ash because it is a weaker base and consequently reacts less violently in neutralizing acids. Soda ash of about 11/2° to 2° B. strength is used. The amount of neutralizing agent generally employed is about 1 barrel to 20 barrels of distillate.

After the neutralization wash the distillate usually is heated to 120° to 130° F. by steam coils in the agitator and then washed with hot water of about the same temperature. One barrel of water to fifteen barrels of oil is sprayed over the oil and settles through it, after which the hot oil-water mixture is agitated by blowing with air until the caustic or soda has been washed out of the oil. Neutralizing agents, as well as any acid particles, must be removed thoroughly from the oil. Otherwise caustic coming in contact with the cotton press blankets causes them to become very hard and as a result less effective as filtering media. Sometimes the water and distillate are not blown with air, as simply spraying the oil with water is considered to wash it sufficiently. The mixture is allowed to settle from 5 to 10 hours, during which time the temperature of the oil may be raised to 150° F. to facilitate separation of the water which is then withdrawn. The distillate should then be clear and bright. If not, moisture is present, and the distillate is then blown with air until the oil is bright. It is then ready for the wax plant proper.
CHILLING WAX DISTILLATE

The first major step in the manufacture of wax is its actual separation in a crude state from the oil with which it is associated. Advantage is taken of the fact that the wax is increasingly insoluble in the oil as the temperature of the oil-wax mixture is lowered. From temperatures of 80° F. and lower the waxes of various melting points begin to crystallize out of solution. The temperature of 80° F. (cloud point) refers to pressable wax distillates. Heavier distillates show higher cloud points because the waxes in them have higher melting points; these distillates are not processed as such in the wax plants because of the trouble encountered in removing the wax. They can, however, be diluted with lighter petroleum fractions, usually naphtha, and then processed. Distillates of viscosities up to approximately 100 at 100° F. are reduced to viscosities of 50 to 70 at 100° F. The quantity of diluent used is 7 to 15 percent by volume. Too large a percentage of diluent is unsatisfactory in that the diluent then dissolves too much of the wax. Distillates that could be processed satisfactorily for average cold-test oils without the use of diluents are processed for zero cold-test oils more easily when diluents are used, permitting better operating conditions. The use of diluents makes it possible to single-press in some cases where double-pressing ordinarily would be employed. The naphtha diluent used can be of various boiling-point ranges. At one refinery 150° F. i. b. p., 450° F. e. p. naphtha is used; at another 200° F. i. b. p., 425° F. e. p. naphtha is used.

The wax distillate is pumped through heat exchangers or chillers to reduce its temperature to such a point that the oil, when freed of some of its wax, will have a desired pour point. This point may be so low that it is necessary to chill and press in two steps. Where the distillate comes to the plant at an elevated temperature and where a cooling medium is available, the oil usually is put through heat exchangers of various types before it is put through the chillers.

WAX-CHILLING EQUIPMENT

Heat exchangers can be employed economically whenever a colder medium is available to cool initially the wax distillate. The distillate comes to the wax plant at temperatures ranging in different refineries from 80° to 110° F. Temperatures of 95° to 100° F. are more common. Where cold water is available the distillate may be pumped through pipe coils submerged in water and the temperature reduced to 75° to 80° F. In one case temperatures as low as 50° F. were obtained by prechilling with water. Instances have been noted where the conventional, highly efficient shell and tubular type heat exchangers are used and the distillate is reduced to 75° F. Cold-pressed oil from the wax presses also is available for chilling. It may be run to tanks filled with pipe coils through which the wax distillate is pumped; it may be used in the common double-pipe heat exchangers; or it may be charged to standard double-pipe chilling machines (described later) in the same way that brine is charged in the main chilling operation. Cold-pressed oil prechillers reduce the distillate to temperatures of 70° to 75° F. In one instance tempera-
tures as low as 40° to 50° F. were obtained, using cold-pressed oil. It should be remembered that, when a distillate is prechilled to below its cold test with the resultant precipitation of wax, provision should be made for a surface-cleaning device. Otherwise the pipes will become coated with wax and soon become inefficient. Prechilling reflects itself in lower wax-plant-refrigeration requirements.

Chillers are made purposely for reducing the distillate temperature and handling the resultant slush of congealing wax in relatively viscous oil. In the United States several kinds of chilling machines are in use, but in all the basic idea of passing the distillate through a pipe or shell surrounded by the refrigerant, with provision for removing wax accumulation, is the same. The essential difference in the machines is the size of the pipe or shell through which the oil circulates and whether the distillate travels horizontally or vertically. The double-pipe horizontal and the 5-foot (diameter) by 20-foot (vertical) shell, so-called “Gray” chillers, are those in general use.

DOUBLE-PIPE HORIZONTAL CHILLING MACHINES

Double-pipe horizontal chilling machines consist of a number of sections of two different-size concentric pipes properly fitted to-

![Diagram](image-url)

**Figure 7.—Sectional view of double-pipe horizontal chiller.**

tgether at their ends with heavy fittings. These direct the flow of the distillate and refrigerant through the chiller. A picture of an uninsulated 5-pass chilling machine is shown in figure 8. The cooling medium flows through the annular space between the two pipes, of which the outer is usually 8 inches and the inner 6 inches. The inner pipe contains a conveyor and cleaning device fitting as closely as possible to the pipe walls. Figure 7 is a sketch of a section of a chilling machine showing the concentric pipes and the conveyor. The device aids in the progress of the chilled and viscous solution through the pipe and in preventing an accumulation of wax on the pipe walls. Chilling machines in general use are composed of 10 or 12 sections, as described, supported on channel-iron stands, 2 sections wide, connected together as a hairpin, and 5 or 6 sections high. The chillers are more commonly called “five- or six-pass machines,” a pass representing the movement of the oil down one section and back another before it moves up or down into another tier. The pipes are about 30 feet long, and a 6-pass chilling machine will have about 636 square feet of chilling surface, occupy a 40- by 3-foot floor space, and be 9 feet high. The conveyors are of the screw type, fitted with shafts which project through stuffing boxes on the end fittings, and they usually rotate at 8 r. p. m. They are driven by
Figure 8.—Five-pass, double-pipe, horizontal chilling machine, showing speed reducer.
sprockets fitted to the conveyor shafts at the driving end of the machine. A set of outboard bearings is provided to support the outer end of the conveyor drive shafts. A heavy-link belt chain runs over the sprockets, obtaining its motion from a speed-reducing device which in turn usually is driven by a belt from an individual motor or from a line shaft.

If several chillers are to be operated, it is considered better design to have one prime mover to drive all the chillers from one line shaft. Where a large number of chillers are in use they are often in groups of 4, 6, or 8, and a separate motor is used to drive a line shaft for each group. Where individual motors are used for each chiller they must be large enough to take care of any overloading due to conveyors sticking, wax plugging the oil stream, or ice forming from water that might be in the distillate. Stoppage of the scrapers can easily freeze the chillers, with attendant difficulties. It is more economical in prime movers to use one large motor or other power unit that is large enough to care for any overloading at one time on part of the chillers. It is unlikely that all the chillers in a group will become overloaded at any one time. To offset this advantage of a single large power unit there is the disadvantage that should the unit stop the whole plant must shut down. However, if the prime mover is a motor, probably the individual motors also would be down. All plants should be equipped so that if the conveyors in a chiller stop the cooling medium can be shut off and warm distillate charged to the chiller to raise its temperature and prevent freezing, then returned to the wax-distillate storage tank.

All chillers should be well insulated; if not, the cooling medium is exposed to the distillate on one side and to the air in the room on the other. The temperature differential between the refrigerant and the air in the room usually is large, and excessive heat can be absorbed from the room, reducing the effectiveness of the chiller. In one satisfactory method of insulating chillers they are covered with molded cork, then encased in a waterproof housing in which granulated cork mixed with paraffin is tamped to fill any voids, completely surrounding the chiller sections and fittings. Wood housing is not desirable unless the wood can be kept dry, as otherwise it rots easily. Sheet-metal housing, unless corrosion-resisting, is not as desirable as transite, which does not deteriorate easily. It is preferable to have the outside covering or housing of an insulated chiller as nearly airtight as possible to prevent the infiltration of air and subsequent condensation of moisture within the chiller insulating materials. Figure 9 is a sketch of one type of well-insulated chilling machine.

Often the chillers are left bare, and an attempt is made to insulate the room itself. In such instances the entire machine soon is covered by an ice cake which retards the rate of heat transfer, so can be said partly to insulate itself. However, the insulating efficiency of ice is very poor compared with that of cork. The thermal conductivity of ice of 0.92 density is 22.0 [0.192×10^{-3}, B. t. u., ft.\(^{-2}\), sec.\(^{-1}\) (°F., in.\(^{-1}\))\(^{-1}\)] compared with 0.42 [0.92×10^{-3} B. t. u., ft.\(^{-2}\), sec.\(^{-1}\) (°F., in.\(^{-1}\))\(^{-1}\)] for cork of 0.16 density at 32° F.\(^{37}\) Although this would indicate that ice transmits heat 50 times as readily as cork, actually the difference would not be so great because the ice about a chiller

contains air which would lower its conductivity appreciably. The rooms are insulated best by lining them with cork-board or rock-wool composition materials. In several instances false walls and ceilings have been built with ground cork, hair felt, or rock wool packed between them and the walls and ceiling. Special care must be exercised to make the insulation waterproof if it is to be of value. Air inflation should be stopped. For economy of refrigeration, such insulation should be considered only when there are few chillers in a plant and they are in the same room with the presses. Then the absorption of heat from the room, as well as from the distillate, may be justified in view of the desirability of keeping the pressrooms at low temperatures. If the chillers are alone in a room or rooms it is more desirable to insulate the chillers and disregard the room.

As an example of refrigeration loss due to the absence of insulation, one refiner with a small wax plant having 24 sections of chilling
equipment and cooling 500 barrels of distillate per day down to 15° F. stated that, as a result of the chillers being uninsulated and in an uninsulated room, over 10 tons of refrigeration per day were being lost. At another plant where approximately 5,000 barrels of distillate were chilled in thirteen 6-pass chillers and 3 Gray chillers (described later), the 6-pass chillers were uninsulated, the Gray chillers were covered with only 2 inches of hair felt, the brine lines had only 2 inches of hair felt, and the chiller rooms were uninsulated; 400 tons of refrigeration were actually needed. The 6-pass chillers were then insulated with hair felt and cork and encased in transite boxes. The Gray chillers and the brine lines were covered with another 2 inches of hair felt, making 4 inches of insulation, and the chiller room was insulated with transite sheets in back of which hair felt had been packed. This insulation reduced the refrigeration requirements of the plant to 250 tons per day. Refrigeration is estimated to cost 50 to 80 cents per ton. It is evident that proper insulation of chilling equipment should be given more than slight attention.

Comment on the double-pipe, horizontal type of chillers is directed to the high pressures under which they operate, part of which is built up in the machine by the viscous distillate moving back and forth through the sections and part by the screw conveyors. Any pressure drop through a chiller represents energy expended. If the speed of the screw conveyors is not synchronized with the pumping velocities of the distillate, pressure will be built up. One manufacturer is producing a spiral ribbon-type scraper as a substitute for the screw-type conveyor. The screw-type conveyor attempted to keep the pipe walls free from wax and to aid in forcing the oil-wax mixture through the chiller. The new type apparently provides for scraping the walls of the pipe free of wax, for breaking up wax accumulations, and for mixing in the pipe with a possible increase in the heat-transfer rate; it depends upon the pumping velocity to keep the distillate moving through the chiller. This type of conveyor should reduce friction losses in the chillers and require less power. It is reported to be responsible for a pressure reduction of 100 to 200 pounds in certain cases, and instead of 8 r. p. m., as in the case of the screw conveyors, the new scraper can operate at as high as 24 r. p. m. Both these factors should permit greater output than that of chillers equipped with screw-type scrapers.

The fact that double-pipe chillers are built to operate under high pressures is of advantage in that only one oil-charging pump is required to handle the wax distillate. A survey of 25 wax-manufacturing refineries showed approximately 118,000 square feet of installed chillers, of which the double-pipe type amounted to 54 percent and the Gray type to 46 percent. More recently, however, new installations seem to favor both horizontal and vertical double-pipe chillers.

Another comment is that the inner pipes are never scraped entirely free of all deposited wax because, due to their construction, the conveyors theoretically never touch the pipe walls. Although it is possible to get a thin film of wax over part of the inside pipe surface, there is little chance of any thick deposit because the heavy pipe will not deform or lose its circular shape. Paraffin wax has a rather low
thermal conductivity, about 4½ times that of cork, or 2.3
\[0.192 \times 10^{-3} \text{ B. t. u., ft}^{-2} \text{ sec}^{-1} (\text{°F.}, \text{ in}^{-1})^{-1}\] at 86°F. Consequently, any wax left on the metal surface of a chilling machine of any type will greatly retard the possible rate of chilling.

In crystallizing a wax out of an oil solution the rate of chilling affects the size of the crystals. Large wax crystals are desirable for best pressing operations with attendant good cold test on the pressed oil. When the distillate is chilled rapidly (referred to as "shock chilling") the distillate will not process as satisfactorily as when it is chilled more slowly. One manufacturer of refrigeration machinery and chilling machines states that the coefficient of heat transfer in double-pipe horizontal chillers using direct ammonia expansion and new-type scrapers is over twice that using brine and old-type scrapers. As a result the mean-temperature difference between oil and refrigerant and the possibility of shock chilling can be reduced.

**GRAY CHILLERS**

Gray chilling machines are vertical tank chillers 5 feet in diameter by 20 feet high and are used in many refineries. Figure 10 is a picture of a Gray chiller showing the scraping mechanism, brine space, insulation, tangential brine inlet, and distillate outlet. This 5- by 20-foot shell is surrounded by another slightly larger one with about a 3-inch space between them. These shells have common top and bottom plates to which they are riveted. Suitable openings for a manhole, distillate, and brine inlets and outlets are provided. The distillate enters the chamber at the top and leaves through an outlet fitting at the bottom. The cooling medium enters the annular space between the shells near the bottom through tangential fittings, circulates around the shell, and leaves at the top in a similar manner. The inner shell is equipped with a scraper mechanism consisting of a shaft, extending the length of the chiller, to which arms are attached. Bars carrying scrapers are fastened at the ends of these arms. The bronze scrapers are thrown against the inside wall of the tank as the mechanism revolves. A suitable driving-gear arrangement is attached to the shaft where it extends through a stuffing box in the top of the tank. The walls of the chiller are scraped free of wax twice per revolution of the shaft. Thus the building up of a wax-insulating layer and the attendant retarding of chilling are eliminated. Two bottom scrapers are provided for directing any accumulation of wax toward the distillate outlet at the center.

Gray chillers are easily insulated. The bottom rests on cork board, while the sides and top are insulated with granulated cork held in place by oak lagging. One Gray chiller has about 314 square feet of chilling surface and a distillate capacity of 300 to 500 barrels per day. It is rated about equivalent to a 6-pass (12 sections), double-pipe, horizontal chilling machine.

It is said of Gray chillers that two pumps are required to handle the distillate and that one of these is pumping chilled distillate which might have a detrimental effect on the wax crystals as a result of the wire drawing of the pump valves. The shell of Gray chillers may develop uneven places, possibly due to subjection of pressure

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Figure 10.—Sectional view of Gray chilling machine.
Figure 11.—12-inch-diameter, 5-section, wax-distillate chilling machine.
CHILLING WAX DISTILLATE

or of partial vacuum. In such cases the scrapers would not be able
to clean the wax from the metal surfaces which would have reduced
heat-transfer efficiencies, depending upon the depth of wax remain-
ing in any bulges or recesses. The Gray chiller does not lend itself
to the application of direct ammonia-expansion refrigeration, as do
double-pipe chillers. Gray chillers operate at relatively low pres-
sures with low power consumption and very little pressure loss.

OTHER CHILLING MACHINES

Two other machines that are manufactured embody the same me-
chanical principles as the Gray chillers just described. There are,
however, very few such installations. Practically all the wax plants
in the United States are equipped with double-pipe horizontal and
Gray chilling machines. In construction and operation these ma-
chines are similar to the Gray chiller, except that the inside shells
are either 20 or 12 inches in diameter, and a number of these are
hooked together to form one unit. The 20-inch-diameter machine is
made of two steel pipes, one within the other, both riveted at the
ends to steel flanges. The sectional units are vertical and are joined
by conduits through which the distillate and wax are conveyed from
one shell to the next. The screw conveyors with which the conduits
are equipped are used to assist the passage of the distillate from one
shell to another and to prevent freezing of the conduits. The wax-
scraping mechanism is similar to that of the 5-foot-diameter Gray
chillers except that springs are attached to the scrapers to keep them
pressed against the pipe walls. The distillate travels from the top
to the bottom of the first shell, then through a conduit to the bottom
of the second shell, thence upward, crossing from the top of the sec-
ond shell to the top of the third shell, and then downward. Brine
travels countercurrent to the distillate in the annular space between
the two shells. The scrapers are driven as in the 5-foot-diameter
Gray chillers and the conveyors by chains and sprockets.

The 12-inch-diameter chiller is of the same general design as the
20-inch one but is of heavier construction to operate under pressure
so that the direct passage of the distillate to the filter presses can
proceed as in the double-pipe horizontal chilling machines. Figure
11 pictures a 12-inch-diameter, 5-section, wax-distillate chilling ma-
chine showing a cross-section of one of the chiller sections and how
the chiller can be insulated. The scraper mechanism is the same
as that in the 20-inch unit. It revolves at a speed of approximately
50 r. p. m. This chiller can be built in any size, 5 sections provid-
ing 312 square feet of cooling surface, which is comparable to that
of one 5-foot-diameter Gray chiller. The screw conveyors of the
20-inch chillers are omitted in the 12-inch one. This type of chil-
er can be insulated effectively and boxed in, as is done with the
double-pipe horizontal chillers.

A distinctly different type of chilling machine, the Moore-Burnmah
oil cooler, is in use in a number of refineries outside of the United
States. Figure 12 shows the construction details of this type of
cooler. It is a continuously operating oil cooler consisting of an
upright series of 12 shallow, circular, cast-iron cells bound together
by vertical tiebolts held in open lugs for the easy removal and re-
Figure 12.—Moore-Burma patent oil cooler.
placement of any part of the machine. Each cell comprises two chambers, upper and lower; the upper one is closed by the bottom of the cell immediately higher when the machine is assembled. This chamber is 4 feet 3 inches in internal diameter, 4½ inches deep, and accurately machined throughout. The wax distillate flows through the upper chambers in series from top to bottom. Brine flows through the lower closed-in chambers, also in series but countercurrent to the distillate. Each cell is provided with a suitable cast-iron, double-ended scraper arm to which are attached cast-iron knife-edge blades on top, bottom, and ends. These are kept in proper adjustment by springs pocketed in the arm. The arms are keyed to a vertical central shaft which extends up through the tier of cells and which is worm-gear-driven. The entire assembly is supported by a frame and base plate. The cooler occupies a floor space of 6 by 7 feet and is over 13½ feet high. This machine has 390 square feet of heat-exchanging surface and is rated at a capacity of 475 barrels per day, cooling from 70° to 20° F.

**OPERATION OF CHILLING MACHINES**

The first operation in the wax plant is chilling the wax distillate to the desired temperature. It might be well to state here that the three operations of chilling, pressing, and sweating are far from standardized and seem to depend entirely upon the equipment. Some refiners prechill their distillate where heat-exchanger equipment and cooling mediums are available. Some refiners press on long cycles or use high pressures; others with limited pressing capacity or with presses and blankets that cannot be made tight under high pressure resort to short pressing cycles or lower pressures. Sweating procedure permits a great number of variations, usually as a result of the equipment it is necessary to use.

The oil is picked up from the distillate tanks by reciprocating steam pumps or, more commonly, by vertical tripless plunger pumps and charged to the chillers. It is common practice where possible to use one large prime mover, such as a large motor, steam engine, or gas engine, to drive one shaft from which all the pumping equipment is operated and, if possible, the chillers.

Vertical plunger pumps are most satisfactory for the high pressures encountered when the distillate goes directly from the chillers to the presses. Where numerous chillers are in operation one tripless pump supplies several chillers. Reciprocating steam pumps are used where the oil is simply pumped through the chillers. Such pumps are not considered desirable for feeding the presses. Because of the pressures at which the presses are operated, it is felt that reciprocating pumps produce shocks from pressure fluctuations that are conducive to poor pressing operations. If possible, one large pump usually handles all the oil charged to the chillers. In choosing the size of a distillate pump that feeds through the chillers to the filter presses, consideration should be given to the fact that when a new press is put on the line it will take a much larger volume of oil. Gray chillers do not require pressures over 50 to 75 pounds, and either plunger or reciprocating pumps are satisfactory to feed the chiller. The distillate charged to the chillers has temperatures of 75° to 110° F. At most plants it will range from 95° to 100° F.
Where water is available for prechilling the temperature of the distillate is reduced 25° to 30° F. One plant with a large supply of cold water gets as high as 40° of cooling. Where cold water is not available, cold-pressed oil may be used and a drop of as much as 60° obtained. Generally, however, the temperature is reduced about 35° F. Wax distillate cooled in water prechillers usually is reduced to 75° to 80° F. and in cold-pressed oil chillers to 65° to 70° F.

The distillate, whether prechilled or not, is chilled to the desired temperature by brine or by the direct expansion of ammonia. The degree to which it is cooled depends upon whether the oil is single-pressed or double-pressed. If single-pressed, the oil will be chilled about 2° F. below the cold-test temperature desired for the pressed oil. The finished commercial oils will have a cold test 5° to 10° F. higher than this. If double-pressed, the oil will first be chilled to whatever temperature may be deemed advisable at the particular plant, which usually depends upon how the wax separates in the second and cold presses, the ease or difficulty of removing it, and the equipment (especially presses) available for the two pressings. At plants making oils having a 20° cold test the temperatures range from 30° to 60° F., but the general temperature at which the wax distillate is warm-pressed, first pressing, is approximately 50° F. After chilling, the oil is pressed at once, and this warm-pressed distillate is run to a tank and charged to other chillers as soon as possible. The oil usually gains about 10° F. and enters the cold chillers around 60° F. The distillate is chilled to temperatures ranging from 10° to 20° F., depending upon the cold test desired for the finished oils. Within the last few years “zero to 10 cold test” oils have been manufactured. For these oils it is general practice to double-chill and press to remove the wax.

The usual temperatures at which the first or warm pressing is done range from 35° to 40° F. It seems inadvisable to go much below 35° F. for the warm pressing because, when cold pressing, the wax becomes difficult to press and sweat with satisfactory yields. For cold pressing the distillate, which has increased 15° to 20° F. in temperature over that at which it was warm-pressed, is chilled to temperatures of −10° to 0° F. Under some conditions, however, the wax can be removed satisfactorily in one chilling and pressing operation. In this connection it might be well to mention the use of Parafflow.30 Recently, this material has been made available to the industry. The addition of Parafflow to oils pressed at 10° to 15° F. gives the oil a cold test as low as that of oils pressed at −5° to 0° F. As a result the manufacture of low-cold-test oils has been made easier, because better wax is obtained in the presses and better swearer yields are obtained when pressing at 10° to 15° F. than at −5° to 0° F.

The amount of equipment required to chill the distillate varies, due to the operating conditions at different refineries. The volumes of oil and brine circulated and the oil and brine temperatures influence the amount of equipment needed. A study of the chilling capacity of Gray chillers, as operated in four large refineries, shows that 1 square foot of chilling surface yields, on an average, 57 barrel-degrees

per 24 hours. This figure was obtained by multiplying the daily distillate throughput in barrels by the degrees temperature drop and dividing by the square feet of chilling surface. The amount of chilling at the various plants ranged from 40° to 60°. The mean temperature difference between the distillate and the refrigerant ranged from approximately 30° to 40° F., averaging 35° F. at the four plants. The actual average refrigerant temperatures were: Inlet 0° F. and outlet 7° F. The actual average oil temperatures were: Inlet 65° F. and outlet 19° F. A study of the chilling capacity of double-pipe horizontal machines as operated in five refineries, several of which were large plants, shows that 1 square foot of chilling surface provides about 25 barrel-degrees per 24 hours. The amount of chilling at the various plants ranged from 35° to 80°. The mean temperature difference between the distillate and the refrigerant ranged from approximately 30° to 45° F., averaging 39° F. A study of similar double-pipe installations at five plants using direct ammonia expansion shows that 1 square foot of chilling surface yields 44 barrel-degrees per 24 hours. Due to lack of information on the pressures to which the ammonia was expanded it is impossible to give the mean temperature differences between distillate and refrigerant. Thus, from plant data, approximately 1,800 barrels per 24 hours can be chilled 10° by a Gray chiller which has about 314 square feet of chilling surface, or 1,600 barrels per 24 hours can be chilled 10° by a 6-pass, double-pipe, horizontal chiller having about 636 square feet of chilling surface. Direct ammonia expansion instead of brine chilling in the latter type of machine apparently increases the figure to approximately 2,800 barrels.

Chilling equipment is arranged in several ways. In plants where Gray chillers are used and a large number is needed, sets of 6 to 8 machines usually are connected in series. The oil is split into several streams, each going through a set of chillers. At 1 or 2 plants they were hooked up in series of 3. Small plants, of course, may use only 1 or 2 machines hooked up in parallel or in series. With double-pipe horizontal chillers this arrangement does not hold. Most of the plants operate them in parallel. One plant, warm-chilling 12,000 barrels of distillate, splits this amount into 16 streams, each going through a 6-pass chiller. However, a few plants arrange the machines in series of 2 or 3, 3 being the greatest number observed in one hook-up.

**REFRIGERATION**

In refinery practice ammonia is used as the refrigerant, generally in the absorption type of machine. This type of machine has proved very satisfactory in that it is simple to operate and can be run in some refineries with available exhaust steam. However, with the advent of low pressing temperatures and the fact that cooling water temperatures are apt to be higher than desirable, exhaust steam has been superseded by high-pressure steam. Moreover, in the newer installations, compression machines are generally used. Complete information on ammonia refrigeration can be found in the literature.

The ammonia is expanded and chills a calcium chloride brine which is circulated through the wax-distillate chilling machines. There are a number of plants, however, that expand the ammonia
directly into the chillers. By so doing, the brine and its system of heavily insulated pipes, tanks, cooler, and pumps are eliminated. The use of direct expansion reduces maintenance, decreases power consumption, and permits smaller equipment in refrigerating and chilling machinery than that used on brine systems. Direct expansion permits good operating control and from an over-all standpoint reduces the cost per ton of refrigeration; it is also more efficient than the use of ammonia to chill a brine which in turn chills the oil. Consequently, directly expanded ammonia permits a greater volume of oil to be handled by a chiller. With the advent of low-cold-test oils and the necessity for very low temperatures, there has been a decided trend toward the use of direct expansion. This is especially true at centrifuge dewaxing plants, where temperatures as low as $-60^\circ$ F. are reached.

The amount of refrigeration required for a given amount of chilling can be calculated. A survey of the installed tonnage of refrigeration compared with the amount of wax distillate chilled per day at refineries throughout the United States gives results as follows: 1 ton of refrigeration is provided for 10.7 barrels of distillate per day, which is an average for 16 plants. At individual plants the figures ranged from 5 to 16 barrels of distillate per ton of refrigeration, but the majority were 8 to 12 barrels per ton. At plants where direct expansion was used the amount of refrigeration was slightly less than that above. The average of five such plants was found to be 1 ton of refrigeration for 11.5 barrels of distillate per day. Individually, all five plants varied only slightly from this figure, none by more than a barrel. A more important figure, which takes into consideration the degree of chilling as well as the volume of oil chilled, was arrived at from the plant data and represents the work done per ton of refrigeration. The plant data show that 1 ton of refrigeration does 716 barrel-degrees per 24 hours, which means that 1 ton will chill 10 barrels $71.6^\circ$ F. in 24 hours. To compute this, the daily throughput in barrels was multiplied by the temperature drop in degrees and divided by the tonnage of refrigeration at the plant. Any prechilling from cold water was disregarded, but any from cold-pressed oil was taken into consideration. This figure, 716, is an average of 15 plants. Individual plants had figures ranging from 350 to 1,000, although the majority were between 675 and 925. Similar data collected on four plants using direct ammonia expansion gave an average of 732 barrel-degrees per 24 hours.

In the operation of refinery refrigeration units the brine temperatures vary at the different plants. At some plants the brine temperature is varied with the season; at others, where large quantities of distillate are double-chilled and pressed, brines of different temperatures are employed for the hot and cold chillers. The average brine temperature can be considered to be around $0^\circ$ F. The brines at most plants range from $-5^\circ$ to $2^\circ$ to $3^\circ$ F.; but a few plants prefer to operate with cold brines of $-15^\circ$ to $-20^\circ$ F., while a very few plants use brine temperatures of $8^\circ$ to $10^\circ$ F. Two large refiners who double-chill and press when manufacturing neutral lubricating oil use relatively warm brines for chilling the oil for the warm pressing and a brine $40^\circ$ colder for cold chilling; for example, the warm brine is at $20^\circ$ F. and the cold brine at $-20^\circ$ F., or they are at
40° F. and 0° F. The temperature of the brine must, of course, be
colder than the temperature of the chilled distillate. The amount of
chilling necessary depends upon the relative rates at which the brine
and distillate are circulated through the chillers. Cold brine may be
necessary when the capacity of the chilling equipment is small for
the amount of oil handled, at which time greater temperature differ-
entials are required. The brine usually is circulated by either cen-
trifugal pumps or vertical plunger-type pumps. Centrifugal pumps
are considered more satisfactory because of the even, steady flow of
the pump discharge.

Plants using direct expansion of ammonia chill to the desired
temperatures and at the desired rates by controlling the pressures
when the ammonia is expanded. Pressures of 3 to 10 pounds on the
suction side of the compressors correspond to temperatures of −20°
to −5° F, in the chillers where the ammonia is expanded.

In addition to other parts, an absorption refrigeration machine
has four sets of coils: The condenser, absorber, weak-liquor cooler,
and rectifier, which may be of four types—shell and coil, atmospheric,
tubular, and double pipe. A refiner installing such equipment should
consider the type of coils used. The refining industry generally em-
ploy the only two, atmospheric or double-pipe coils, depending upon
the quality of the water available. The coldest water available at
any refinery ordinarily is used at the refrigeration plant, as it pro-
vides economical operation and maximum refrigeration.

By far the greatest number of refrigeration plants are equipped
with atmospheric coils, because the coldest supply of water is gener-
ally from a river, a lake, or an ocean inlet in which considerable silt,
mud, and dirt and occasionally a high percentage of soluble salts are
usually present. With such water, atmospheric coils are preferable
to double-pipe coils since they can be kept more efficient and require
less cleaning. If double-pipe coils were used it would be necessary
to be continually cleaning out materials that settle or deposit and
adhere to the walls or even plug the pipes. Refrigeration coils are
essentially heat exchangers, and any formation of scale or depositi-
on of silt greatly affects the rate of heat transfer and therefore the
capacity and efficiency of the machine.

Atmospheric coils rust and scale, and in many refineries this is
removed twice a year. It should be done at least once a year. Some
refiners paint the coils after such cleaning, believing that they will
not rust as easily as when left unpainted. In addition, small plant
life, such as algae, which covers the coils must be combated at
refineries where the cooling water becomes warm during the summer
months. When such growths, silt deposits, etc., occur in harmful
amounts they should be removed. Some large plants (about 1,000
tons of refrigeration) employ one or two men constantly to keep the
colts clean.

Double-pipe coils are used where good, clean water is available,
usually from wells. In one refinery where the water carries a little
silt the double-pipe coils must be cleaned every 2 or 3 weeks. At an-
other refinery where the water is fair the coils are cleaned twice a
year, a tube cleaner being run through the pipes. By this method the
colts are kept in good condition. One refiner believes double-pipe
colts thus cleaned are superior to atmospheric coils which rust easily
and are rather difficult to clean. Double-pipe installations look cleaner and neater.

In compression refrigerating machines approximately one-fifth of the coil surface is used that would be required in absorption machines, which is a decided advantage where the refiner is confronted with contaminated cooling water with resulting severe corrosion of coils.

Recently a new method of providing refrigeration has been employed in a plant dewaxing distillate from which motor oils are produced of viscosities ranging from S. A. E. 20 to S. A. E. 60. In this plant liquefied propane under pressure is used as a solvent for the oil. Part of the low-boiling solvent is evaporated adiabatically, thus producing a low temperature and causing the wax to crystallize. A brief description of the plant and process of refrigeration, taken from the paper by Bahlke, Giles, and Adams,49 is somewhat as follows:

Liquefied propane from a storage vessel is mixed with the oil from a tank and the combined stream pumped through a heater in which the mixture is heated with steam to a temperature of about 180° F. The stream is then discharged into one of the chilling vessels or drums until it is about three-fourths full, then diverted to another chilling vessel. Chilling in the vessel is accomplished largely by evaporation of part of the propane but is aided somewhat by slow introduction of cold propane (−40° F.) which has been used to wash the filter cake in the wax filter. The evaporated propane is compressed, condensed, and returned to the propane storage vessel. In operating the plant the chilling drums are charged in rotation; thus one may be filled while the second is being chilled, the third being ready for discharging to the cold-oil storage drums. A complete cycle of charging and chilling requires about 90 minutes. The final chilling temperature is −40° F.

**FILTER-PRESSING CHILLED WAX DISTILLATE**

**FILTER-PRESSING EQUIPMENT USED**

Filter-pressing the chilled wax distillate in the wax-manufacturing process is carried along with the chilling procedure just described. Rather, chilling the distillate is contingent upon filtering it. The pumps charging the oil to double-pipe horizontal chillers can build up pressures of 800 or more pounds and, in addition to forcing the chilled oil and condensed wax through the chillers, force them into a header or headers that extend into the pressrooms. Where Gray chillers are used, press pumps take the oil-wax mixture from the chillers and put it into the press headers. The chilled oil and wax in these headers are necessarily under pressures great enough to permit satisfactory operation of the filter presses. Because of these pressures single-acting, positive-displacement, plunger-type, triplex power pumps ordinarily are used.

The filter presses used by the refining industry to separate the wax from the oil in the chilled oil-wax slush coming from the chillers are

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Figure 13.—Phantom view of a filter press, showing wax conveyor and oil drain below press.
essentially of one type—horizontal, hydraulic, plate filters. Figure
13 is a phantom view of a filter press, showing the wax conveyor and
oil drain below the press. These presses consist of a series of plates
and rings arranged so that a space is formed between each two plates
when they are pressed together tightly by a hydraulic ram and then
held by steel tierods and nuts. The chilled wax distillate comes into
each space, and the oil flows through the canvas blanket and outward
toward the plate edges. The crystallized wax remains behind on the
canvas. The plates are circular and range in diameter from 27 to 48
inches, but the latter size is practically always used. The plates and
loose rings (most refineries use these) are supported on two large-
diameter steel side rods which tie together the ram cylinder and sta-
tionary platen. The ram, stationary platen, follower platen, side
rods, and tierods form the filter-press frame and are supported by
cast-iron cradles or columns resting on the floor. The length of the
cylinder and the piston stroke depend upon the number of plates and
rings in the filter press. An 11-foot cylinder having a 10-foot piston
stroke is furnished with a 525-plate press. When the plates are set
on the side rods the follower platen is set up by the ram, squeezing
the plates firmly together, then the nuts on the tierods are tightened,
holding the plates firmly together. The ram is operated hydraulically
and serves only to force the plates together when the filter is made
up and to compress them again after the press is full so as to unscrew
the nuts on the tierods and back off the follower platen.

These presses are operated at pressures up to 500 pounds. High
pressures also are used in making up a press; consequently presses
are fabricated of heavy castings, and the rods are of large diameter.
The ram operates under pressures of 700 to 1,000 pounds per square
inch in making up and relieving the presses. The piston rod trans-
mitting this pressure to the platen is of steel 8 inches in diameter and
10 to 14 feet long, depending upon the number of plates in the
press. The two side rods upon which the plates rest are 4 1/4 inches
in diameter and up to 60 feet long for 500-plate presses. There are
8 tierods 3 inches in diameter, their length depending upon the num-
ber of plates in a press; in a 500-plate press they are about 45 feet
long. The platens, between which the plates are held together, are
heavy iron castings with 8 holes to take the tierods and 2 holes to
take the side rods. The stationary platen is fitted and drilled in the
center for a 2-inch pipe connection through which the press is
charged with distillate from the press header. The follower platen
slides on the two side rods when the nuts on the tierods are removed
and the platen is pulled back.

Under the press framework is a trough which catches the oil as it
runs out of the filter-press plates at their peripheries and carries it
to a pressed-oil tank. There is also another trough beneath the press
into which the wax falls when it is broken out of the press. A helical
conveyor operates in this trough and carries the pieces of wax out
into a tank where they are melted down by steam coils preparatory
to further processing. The oil trough is movable and is under the
press while oil is running off the press, but it is drawn aside to per-
mit the wax to drop into its trough when the press is being cleaned.
FILTER-PRESS PLATES

The filter-press plates are of vital importance to the filtering process, and exceptional care should be used in the manufacture and handling of them. These plates are of two general types, the loose-ring and the riveted-ring type. The loose-ring plate seems to be preferable to the industry, although a number of refiners maintain that the riveted type is superior. Figure 14 shows the various parts going into the construction of loose-ring filter-press plates. A press ring such as is put between each plate in a loose-ring filter press also is shown. Figure 15 shows in detail the difference between loose-ring and riveted-type plates.

The loose-ring plates are made of solid-steel center plates three-sixteenths inch thick having projecting lugs for supporting them on the two side bars of the press frame and a center hole 6 inches in diameter for the passage of the distillate. On either side of the center plate is a no. 16 U. S. gage steel disk, perforated with small holes on close centers and having a hole in the center slightly larger than the hole in the center plate. The perforated disks are covered with a blanket made of 12/0 hydraulic cotton duck (approximately 3 pounds per square yard), and all parts of the assembled filter plate are held together by 12 rivets and 24 buttons. The blankets are held together at their periphery by wire clips or twine. At the center they are
LOOSE-RING TYPE

RIVETED-RING TYPE

Figure 15.—Details of filter-press-plate construction.
well sewed together. The buttons on each plate are in the same location, thus they come directly opposite each other when the plates are put in a press. Great care should be exercised to have these buttons of proper thickness so that they will not come in contact before the joint is made at the outer edge of the plate with the ring. It is also important that there is no great amount of clearance, as otherwise the plates would tend to dish when the press is put on the line. All parts of filter-press plates should be made to gage and be interchangeable. To provide a space between these filter plates, rings are placed between the plates, forming a joint at the edge of the plate. These rings are of the same diameter as the plates, presenting a face 1\(\frac{1}{4}\) inches wide to the plates and having a thickness of \(\frac{1}{4}\) to 1 inch, depending upon the thickness of wax cake desired. However, practically all rings are one-half inch thick, which will allow for a one-half-inch wax cake. Each ring is supported in the same way as the plates; that is, a lug is attached to each side which assures proper alignment when assembled with the plates in the press. While rings one-half inch thick are more or less standard, one or two refiners use \(\frac{1}{4}\)- and \(\frac{3}{8}\)-inch rings, and several are using 1-inch rings in their warm presses.

The riveted-ring plate uses the same center plate and perforated plates as the loose-ring type. To provide a space between the plates for the wax to accumulate two rings of the same outside diameter as the plate, with a face somewhat wider than that of the loose ring, beveled on the inside-diameter edge and \(\frac{1}{4}\) or \(\frac{3}{8}\) inch thick depending upon the desired wax-cake thickness, are fastened to the outside of the perforated plates by rivets passing through the center plate, making the 3 plates and 2 rings into one assembly. A 12/0 hydraulic duck blanket covers the plate, as in the loose-ring type. Buttons of the same type and number are riveted to the fabricated plate as described. The joint between plates is thus formed by the canvas blanket of one plate at its periphery being forced tightly against the canvas cover of the next plate.

One or two variations have been observed in riveted-type plates. In one case the one-fourth-inch beveled steel rings are replaced by circular 12/0 duck (filter-press blanket material) strips built up to the desired thickness (about one-fourth inch) and riveted to the center and perforated plates, thus acting like metal rings in forming a space between any two plates. It should be stated, however, that the plates were equipped with twelve 4-inch-diameter buttons or about four times the usual button area on a plate. This was probably necessary to provide stability to the press. In another case the plates were equipped with twelve 3-inch-diameter beveled buttons on each side. These are riveted to the metal plates before the canvas blanket covers the plate. The blanket then covers the buttons as well as the riveted rings.

The large number of refiners using presses equipped with loose rings compared with the few using riveted-type plates would seem to indicate that the loose-ring type is superior. The arguments advanced in favor of the respective plates are about as follows:

The riveted type has one joint per plate and the loose ring two, consequently there is less chance of leakage with the riveted type.
The riveted type has a canvas-to-canVAS joint which makes up tighter than a metal-to-canVAS joint, reducing further the possibility of leakage.

The riveted type has a wider blanket-to-blanket joint because of the increased face of the riveted ring over the loose ring.

The riveted type does not require the handling of a ring with each plate, probably saving labor; however, the riveted-type plate is heavier than the loose-ring type, which tends to offset this advantage. One refiner states that 30 to 45 minutes in dumping time have been saved as a result of using riveted plates.

The riveted-type plate may require less attention when the press is set up due to elimination of the rings.

The riveted type is considered to cause greater wear and tear on the blankets as a result of having to insert breaking bars between the two tightly packed canvas plate blankets. When the press is broken down for removal of wax the bars usually are inserted at the same place each time, and the canvas becomes worn through at this point. To overcome this objection one refiner satisfactorily breaks the plates by inserting the breaking bars between the lugs of the plates. This may not be feasible in all cases because the plates are too strongly held together by the wax.

The riveted type is considered to be more easily torn by the spud bars in cleaning the wax off the plate along the bottom half near the plate edges. Should the spud, as it is scraped along the canvas, strike the riveted ring it may cut through the canvas. To make the canvas rise gradually from the plate face to cover the ring, the ring is beveled, but enough obstruction is presented so that a spud bar can cut the canvas. On a flat plate this is not so apt to happen, as the spud would pass over the end of the plate.

Loose-ring-type plates require less labor in repairing if warped or buckled, for in straightening out the center plate it is not necessary to cut off the two rings riveted to the center and perforated plates.

Some refiners maintain that one or the other of the two types of plates buckles more easily. The writer believes that such a condition bears no relation to the type of plate but is directly traceable to the buttons on the plates; the rings, whether loose or riveted, are considered fixed, and the thickness of the buttons depends upon them. Ice and its effect on press-plate buckling are not considered, as this is not the fault of any particular type of plate so much as of the procedure in wax-plant operation. Both types of filter-press plates are said by different refiners to give less trouble from the press plates as a whole bulging up when the press is made up. The writer believes that this condition is not due to any type of filter-press plate but rather to the buttons on the plate. If less clearance is provided at the buttons than at the plate edges at the rings, whether the latter are riveted or loose the buttons will be subject to greater compressive forces than the rings, resulting in a greater tendency for the press to buckle.

With the riveted type of plate and the blankets tightly stitched or wire-clipped at the plate edges, there is a probability of the stitching or clips pulling out from tension caused by pressure of the wax cake.
forcing the blanket tightly against the plate. Then the blankets fall away from the plate, making it inconvenient when cleaning presses or removing plates for repairs.

One refiner who uses loose-ring plates feels that riveted plates would be advantageous because of the impossibility of the rings falling off the two supporting side rods and getting down into the spiral conveyers, thus possibly jamming the system and making it necessary to cut the ring out of the conveyer, with the attendant interruption in the pressing cycle.

Riveted-type plates in which the canvas blanket covers the buttons as well as the rings permit blankets to be repaired more easily than those in which the buttons are riveted to the plate with the blankets under them. It is not necessary to cut off 12 rivets to remove the canvas; and it is claimed that, whereas one man could remove old blankets and place new ones on only 6 plates per day when it was necessary to cut off buttons, one man could handle 24 plates per day when the buttons could be neglected.

These opinions represent the “pros and cons” of the refining industry on the merits of the two types of plates. Although all the opinions have merit, it seems that the loose-ring press is used more generally because the maintenance cost of the blankets is lower. This is no small item to be considered by refiners with filtering areas of 100,000 to 800,000 square feet. Either type of press can be made tight if proper consideration is given to clearances and uniformity in all details of plate construction.

There are, however, factors to be considered in filter-press plate design which are of more importance than the type of plate used and of equal importance to any type of press plate. First and of prime importance is the design of the perforated plate on either side of the center plate. Upon it may depend the efficiency of the press, in that it may retard the possible filtering rate, produce oily wax cakes, and necessitate excessive filtering pressures with concomitant operating difficulties. The perforated plate is punched with holes that are usually one-eighth inch in diameter; some plates may be punched with holes a trifle larger, others with holes as small as one-sixteenth inch. These holes usually are on one-half-inch centers and are staggered. Some plates have the holes on centers as short as one-fourth to five-sixteenths inch. The perforated plate supports the canvas blanket and permits the oil that passes through the canvas to go through the one-eighth-inch holes in the plates and then travel to the edge of the plate and run off the press. Due to the pressure which forces the blanket against the plate, the size of perforation should not be over one-eighth inch, otherwise trouble may be experienced from the canvas being forced into the perforations. When a plate is punched as much as possible, smaller holes permit more hole area, thus affording greater opportunity for the oil to get past the perforated plate.

The oil travel is such that once it reaches the perforations it must be permitted to travel to the edge of the plate. To do this it is necessary to raise the perforated plate somewhat from the center plate, or if lying perfectly flat on the center plate some means must be used to permit the oil after getting through the perforations to
get to the plate edges. Unless one or the other is done the pressing operation will not be entirely successful. Poor press efficiency results from improperly perforated plates. Originally the plates were punched from one side, and slight burrs were formed on the underside of the punched plate. These were sufficient, when the rough side of the perforated plate was placed next to the center plate, to keep the perforated plate off the center plate, and since the edges of the burrs were not all of the same height it permitted the oil to go through the perforations and travel between the perforated plate and the center plate out to the edges and drop off the filter. The writer has noticed that where such plates have been used for many years and have been removed and repaired many times these burrs tend to flatten down or wear or break off. With such a condition one usually finds operating difficulties. Presses have been observed in which most of the cakes were dry, but a few were quite oily and sticky. Upon examination such oily cakes were found to have been caused by perforated plates without any burrs, allowing the plate to fit closely to the center plate. Moreover, where presses were made up mostly of such plates it was noticed that excessive pressures had to be used to get the press or presses to flow at desirable rates. It is not to be inferred, however, that the pressures used in pressing operations are always attributable to the condition of the perforated plates. The wax quality and blanket condition must not be overlooked.

Various methods are used to insure that a perforated plate will stand away from the center plate. Plates are punched so that the holes have heavy burrs on one side. In some cases every third or fourth row of holes is punched so as to leave larger burrs than the others. One method used is to make every other row of holes with a punch which has a shoulder on it and which, after punching a hole, presses down or cups the hole. The plate then is held away from the center plate by these rows of beaded or cupped holes. Another method used on plates for warm filter presses to increase throughput, or on perforated plates which are quite smooth on the side that would be next to the center plate, is to place a wire screen, no. 5 mesh and of no. 16 Old English gage wire, 0.065 inch in diameter, between the perforated and the center plates. In this way the oil can find egress to the edge of the press plate. Still another method is to take the solid center plate and groove it with a number of radial channels. These grooves are in addition to the use of burried perforated plates and aid the flow of oil to the plate edge; they also provide some oil a way of travel if the perforated plate lies more or less flat against the center plate.

The number of buttons, their size, and care in their manufacture and installation are of importance. The volume of open space between plates taken up by buttons represents volume that is replacing wax. In other words, if buttons were not necessary the filtering area and the wax capacity of a plate could be increased 2½ percent based on the standard 48-inch plate with twelve 2-inch buttons. Most refiners use plates with twelve 2-inch buttons on a side. This number and size seem more or less standard. Sometimes, however, the size has been reduced. In one plant twelve 1¼-inch buttons were used successfully and, in another, twenty buttons, 1½ inches in
diameter. In other installations the size of the buttons has been increased, in one case to 3 inches and in another to 4 inches, 12 buttons being used in each. Ordinarily button sizes larger than 2 inches and greater in number than 12 per side are not warranted. With the 4-inch button the plate was of the riveted type, and the rings were made of blanket material as previously described. Such construction may not have given solidity to the plate edges and may have made necessary the larger buttons. In this case probably about 10 percent of the volume between plates is taken up by buttons. If due consideration were paid to having uniform plates probably fewer buttons than twelve 2-inch ones could be used satisfactorily on one side of a press plate. One refiner whose slack wax from the cold presses was oily and sticky and who operated the presses at 350 to 400 pounds removed the outer 8 buttons, retaining only the 4 center 2-inch buttons, to permit easier removal of wax. It was stated that these wax press plates do not buckle and that the press makes up satisfactorily.

Another factor of prime importance in press-plate construction is uniformity in thickness of the plate at the buttons and at the rings. It is questionable whether enough consideration is given this factor by most refiners in repairing and rebuilding their plates. Today some buttons are manufactured that are machined to within 0.001 to 0.002 inch variation because it is believed by the manufacturer that such accuracy is necessary. Such refinement is not warranted if proper consideration is not given the perforated plates, for example, if one plate stands away from the center plate more than the other due to the condition of the burrs or type of punching. Precaution should be used when changing perforated plates on a press plate to see that both the perforated plates are the same distance from the center plate. Then buttons of uniform thickness will produce a satisfactory press plate. Bushings, cut out of old press blankets, should be used under the buttons with care, as their promiscuous use can go far toward causing press-plate buckling and leakage.

The buttons are held to the plate by a rivet passing through the three plates, the canvas, and the buttons. When the press plate is repaired it is necessary to drive out these rivets. One refiner, who considers this undesirable, substituted machine screws for rivets and threaded one of the buttons. When the plates are repaired the buttons are easily removed.

In designing plates for a loose-ring filter press consideration should be given to the lugs that are placed on the loose rings. Formerly it was the general practice to take a piece of soft, flat, steel bar and form it into a ring, then to cut grooves diametrically opposite each other to provide slots into which the lugs would be inserted and riveted to the ring. Due to handling and jarring, these lugs might work loose, and in some cases presses have been observed to leak at the loose lugs. To eliminate this, some refiners are welding the lugs to the rings, making them integral.

**AMOUNT OF FILTERING AREA**

The amount of pressing equipment required for a wax plant depends primarily upon the amount of distillate to be pressed. A
guide to the amount of pressing surface required can be obtained from a study of what various refineries find necessary to carry out their operations. Where double pressing is employed the number of square feet of filtering surface per barrel of distillate per day averages 45. This is an average of six plants, of which some were operating on Pennsylvania and some on average Mid-Continent crude. One plant not considered with those above was operating on 28 square feet per barrel per day. This plant was using 1/4-inch rings in its presses; the others were using 1/2-inch rings. This may account for the apparently better filtering rate. The figures for the six plants varied little from the average. The distillate filter-pressing capacities of these plants ranged from 400 to 12,000 barrels per day. A point of interest in regard to the amount of filtering area was noticed; the amount in the cold presses was two to three times the amount in the warm presses. Where single pressing is employed the square feet of filtering surface per barrel of distillate per day averaged 46, practically the same as that for double pressing. This figure is an average of 16 plants, of which all but one operate on average Mid-Continent crude. The figures for the various plants ranged from 20 to 90 with over two-thirds of the plants ranging from 35 to 64. The filter-pressing capacities of these plants ranged from 140 to 13,000 barrels of distillate per day. A standard 48-inch filter-press plate with twelve 2-inch buttons on each side provides about 21.7 square feet of filtering surface. Therefore, the average wax-manufacturing refinery has pressing equipment slightly in excess of two standard 48-inch plates for each barrel of wax distillate processed per day.

The relation of the slack-wax content of the distillate to the number of square feet of filtering surface per barrel of distillate per day is as follows: 7 plants with distillates of 13 to 18 percent slack-wax content had an average of 36 square feet; 6 plants with distillates of 19 to 23 percent slack-wax content had an average of 45 square feet; 3 plants with distillates of 24 to 29 percent slack-wax content had an average of 51 square feet. These are average figures, for in a number of plants with slack-wax contents ranging from 10 to 30 percent little difference existed in the filtering area provided per barrel of distillate. The same holds true for the condition of the slack as expressed by its oil content. The oil content of the slack at the various plants ranged from 30 to 60 percent, yet the amount of filtering area provided bore little relation, if any, to this factor.

BREAKING AND "SPUD" BARS

After the filter presses have been “on the line” for a given time and the filtering rate has become too slow and the press is full of slack it becomes necessary to cut them off the line and open the press to remove the slack. Due to the operating pressures at which the wax is forced against the canvas blankets, the wax is apt to adhere tightly to the blankets. Moreover, any water on or in the press will be frozen. Consequently, considerable force must be exerted in breaking apart the plates in a press preparatory to “spudding” or removing the wax from the canvas blanket of the filter plate. To do this “breaking bars” are employed. Breaking bars are used where the filter-press plates are stuck together tightly, and when the plates
are broken loose a lighter “spud” can be used to clean off the wax. If the plates do not adhere too tightly to one another the same spud bar that cleans the plate is used to break the plates loose. Breaking bars are of heavier stock than the spudding bars. They are made from ¾-, ½-, 1-, and 1¼-inch steel stock, with a handle formed out of the stock at one end and the end to be inserted between the plates flared out to a lip 1, 2, or 3 inches wide, wedge-shaped in cross-section to permit entrance between plates. The edge should be kept dull to minimize cutting of the canvas. Due to the force necessary to pry apart plates that are stuck together tightly, considerable wear takes place on the blankets at this point of entry. Several refiners have substituted a steel blade for the wedge-shaped tip on the breaking bar. The blades are 4 to 5 inches wide, 15 to 20 inches long, and taper until they are rather thin at the ends. These enter between the ring and the plate more easily than do the blunter breaking bars, and the pressure is transmitted over a wider area, thus being less severe on the canvas. The blades are welded to bars which have one end bent, forming a handle. Breaking bars as well as spud bars are about 30 inches long.

Spud bars vary more in design than breaking bars. Breaking bars must be heavy in order not to break when in use. If they break, serious injury may result to the press cleaner. Spud bars should be as light as possible, as the efficiency of the press cleaners can then be kept at a higher level during their working day. Heavy spud bars tire the cleaners and reduce their efficiency. The average spud bar is of ½- to ¾-inch stock with a handle formed on one end, a flared tip 2 to 3 inches wide on the other, and about 30 inches long over all. Several plants use ¾-inch stock, and several have tips as wide as 4 inches. Usually spud bars of the size and description just given are used in plants where they also serve as breaking bars. Figure 16 shows a press being dumped; the spud bars used also serve as breaking bars.

In order to lighten these bars various expedients are used. The spud bars may have a wooden handle with a one-sixteenth-inch steel blade 4 inches wide and 6 inches long attached to it, or it may be made of a hollow steel tube with a handle welded on one end and a 2-inch, soft-steel, rounded tip welded on the other. The tips on all spuds should be rounded somewhat and sharp edges and corners eliminated. A number of refiners use wooden paddles to break the filter-press plates and to remove the wax from them. Where press plates break easily (and in such cases the wax cake usually breaks away easily from the canvas) wooden paddles can be used advantageously. The paddles, made of hickory or maple, are about 30 inches long and have a blade about 3 or 4 inches wide and 12 inches long. One refiner stated that maple was superior to other woods in that it did not splinter as much. The paddles are sharpened when the edge becomes battered and too thick to enter easily between the filter plates. This can be done about six times before the paddle becomes too short to be usable.

**FILTER-PRESSROOM INSULATION**

Filter presses should be housed in well-insulated rooms for efficient refrigeration. If presses are in rooms having much higher temperatures than the desired cold test of the pressed oil, it will be necessary
to chill the wax distillates much colder to obtain the cold test than if the rooms were at temperatures near that of the desired cold test. When putting a filter press on line the first oil through will be warmer as a result of cooling the press, which of course gives the oil a decidedly higher cold test. With the advent of low-cold-test lubricating oils it was found imperative in their manufacture to keep pressroom temperatures low. At the temperatures formerly maintained in the pressrooms it was difficult, if not impossible, to get the low cold test required, even though the wax distillate had been chilled much lower than was deemed necessary.

Where relatively few presses are needed it is probably desirable, judging from the operations at many such plants, to have them in separate rooms. Where many presses are needed these are grouped in larger rooms. Presses filtering oils of the same temperature can best be grouped together. If oils of different temperatures are filtered the presses handling them can be placed in separate rooms where the temperatures can be controlled with reference to that of the oil. As stated before, these rooms should be insulated. The walls and ceiling can be lined with sheet cork or sheet material employing rock wool as a base. Rock wool, ground cork, or hair felt can be packed between transite sheets and the walls or ceiling. Cork blocks or hair-felt blankets can be attached to the walls and ceiling. Insulating brick of various makes can be used as partitions between the rooms or as linings for outside walls.

In addition to insulating the pressrooms, provision should be made for chilling them, not depending upon the cold filter press itself to absorb the heat from the room. Chilling the room to the desired temperature can best be accomplished by circulating some cold brine direct from the refrigeration units through pipe coils in the pressroom. The brine is then returned to the storage tanks of the refrigeration plant. Where higher-brine pumping pressures are not apt to be built up, cold brine from the wax-distillate chillers might be used before being returned to the refrigeration plant. If direct ammonia expansion is employed refrigeration coils of this type can be installed in the room. The amount of chilling and size of coil necessary can be figured when the structural details, dimensions, and insulation of the pressroom are known. However, coils of six 1-inch pipes, about the length of the pressroom, on each wall near the ceiling seem to chill satisfactorily a room containing one press. Where several presses are in one room the coils can be supported on a vertical metal frame between each press. It probably is not desirable to have the coils hanging horizontally from the ceiling and over the presses. In large rooms the brine coils should be made of pipe larger than 1 inch.

With double filter pressing the necessity for chilling the warm pressroom is not as great as with cold pressing. All pressrooms, however, should be insulated. One refiner making zero-cold-test oils keeps the pressroom at 15° F., which was probably the coldest temperature observed. At all other refineries the pressroom temperature ranged from 15° to 50° F. or higher when little, if any, consideration was given to temperatures.

Where diluents or solvents that are volatile are used and their concentration in the air of the pressrooms is apt to become too
great, the use of air conditioning has been instituted. In such cases fresh air is forced over refrigerant coils and discharged into the pressrooms.

**OPERATION OF FILTER PRESSES**

The operation of filter presses varies mainly with the pressures carried on them and the length of time they are left on the line. After a press has been emptied and any defective plates have been replaced the press is set up with a hydraulic ram. In the setting up of a press the ram piston is actuated by pressures ranging from 750 to 1,000 pounds per square inch. This pressure is transmitted to the rings, filter-plate edges, and buttons, forcing them tightly together. The nuts on the 8 tierods are tightened, then the pressure in the ram is reduced. The filter plates are then held together tightly by the tierods. A 2-inch pipe lateral extends from the chilled wax-distillate header to the center of the stationary platen of each press. A control valve is placed in this line. When the press is ready this valve is partly opened, permitting the oil to enter the empty filler and travel from plate to plate through the hole left in the center of the plates. When the wax distillate fills the press and the pressure begins to build up, the oil starts filtering through the canvas blankets, leaving behind any crystallized wax. At first the oil filters through rapidly, but as soon as the wax builds up in a layer over the blankets the rate of filtering drops. When the space between the plates is full of wax it is difficult for the wax distillate to reach much of the blanket, and the small streams of pressed distillate practically cease to run out from the edges of the plates.

In the pressing operations at some refineries four steps may be recognized: (1) Bringing up to pressure, (2) on the line, (3) drying out, and (4) removing the wax or dumping the press. At other refineries 3 or only 2 of these may be employed. To illustrate, after the valve on the lateral from the header to the press is cracked to allow oil to enter the press, the pressure on the press is gradually increased to 300 to 350 pounds, the normal pressing pressure, for 6 hours. The press may run for 10 or more hours at this normal pressure until it is considered full of wax. Then the pressure may be raised to 450 to 500 pounds for an hour or more to force out some of the oil occluded in the wax and make the wax cake drier. Then all pressure is taken off the press, and it is opened up to remove the wax.

"Bringing the press up to pressure"; that is, slowly bringing the pressure up to that in the distillate header over a period of several hours, is practiced at a number of refineries. Most refiners, however, take little more time than the necessary hour or more to fill the press with oil before opening the control valve wide or putting the press on the line. The procedure of "bringing a press up to pressure" varies widely. Some plants take 6, 8, or 10 hours to do this, during which time the control valve on the oil line to the press is gradually opened and the pressure in the press gradually builds up to the header pressure. Others use an individual pump for bringing the press up to line pressure in about 2 hours and then cut the press into the line. Still others raise the press pressure at definite increments, such as 20 or 40 pounds per hour, until the line pressure is reached. One plant brings the pressure up at 20 pounds per hour for about
20 hours and then cuts into the line pressure at 450 pounds, leaving it there for another 18 hours. Others, operating a number of presses in a group, have one press being cleaned out, the next at 100 pounds, the third at 200 pounds, and the fourth at 275 pounds or line pressure; when the last press is ready to be cleaned the pressures are stepped up on the others, and the one just cleaned is charged with distillate if this has not already been done. One refiner operating four or more presses in a group, with a line pressure of 325 pounds, brings an empty press up to 250 pounds in about an hour and keeps it at this pressure until the next one in the group has been cleaned and is ready to take oil. At this point the press at 250 pounds is raised to the line pressure and remains there until ready to be cleaned.

Filter presses are operated at various "normal pressing" pressures; the general practice is 300 to 350 pounds. A very few plants operate at 200 to 275 pounds, and several operate at 400 to 450 pounds. The pressure used seems to depend upon the quality of the wax and the rate at which filtering must be done. For example, one refiner found little difference in the quality of the wax cake when pressing at 300 or 400 pounds pressure. The time factor, however, was important. If the press at 300 pounds was left on the line less than a given time the wax cake would be soft and oily. Another stated that wax cakes of equal quality were obtainable at pressures from 150 to 350 pounds. The throughput of the press, however, was greatly reduced at the lower pressures. Another stated that satisfactory wax cakes are obtained at 275 pounds and that it is not necessary to have 300- and 400-pound pressures. For some waxes, at least, it seems that there is a minimum pressure at which a satisfactory wax cake can be produced in a press. Any greater pressures will not affect the wax cake materially. Consequently, the lowest pressure is used at which the volume throughput can be obtained. Since the wax distillates usually manufactured have as high viscosity as they can and yet be pressable and the slack wax sweatable, the slack wax in the presses is more apt to be oily and pasty instead of the hard, dry, crystalline wax possible if the distillate did not have as high a viscosity. As a result, to get a desirable rate of throughput and to force out of the wax cake as much oil as possible, filter-press pressures can be considered high compared with what they might be.

The drying-out step in pressing is carried out at very few refineries because it is generally felt to be unnecessary. When the throughput diminishes to uneconomical operation the filter press is cut off the line. It is then ready to be dumped, using breaking and spud bars as previously described. Uneconomical operation is considered by one refiner to be 5 to 7 barrels per hour for 500-plate presses. Another considers a rate of 2 barrels per hour low enough to discontinue filtering.

In general, the pressing cycle, or the time from a press taking oil until it is charged with oil again after cleaning, is about 24 hours. At 23 plants this actually ranges from 15 to 60 hours, averaging 28 hours, which is high owing to two plants which run on 60-hour cycles. Plants operating with double pressing are about equally divided in number between those having the cycle of operation of their warm and cold presses the same and those having the cold-press cycle shorter, usually about one-half the warm-press cycle.
A question of some importance in the pressing operation pertains to what effect, if any, a reduction in pressure on a press, while filtering, has on the operation of the press and the slack-wax quality. In general, it is reported that any appreciable pressure drop which exists for a time during the pressing operation will have a deleterious effect on subsequent operation of the press and that under such conditions the press usually is taken down and the wax removed instead of the press being brought up to pressure again in an attempt to continue filtration. Should the pressure on a press be reduced greatly for only a short time, such as 10 to 15 minutes, it is reported that little effect is noticeable on the press operation when the pressure is brought back. Should pressures be reduced to half of what they are or less and remain so for several hours it is reported that the presses fail to function properly when the pressure is again raised to normal. The press seems to seal up, in that the throughput is greatly reduced, and the wax cakes will remain oily regardless of the pressure subsequently used or the length of time it is applied.

USE OF HOT WATER FOR CLEANING PRESSES

When a refiner has a large number of presses the labor involved in removing the wax from them assumes sizable proportions if the usual procedure of press dumping is followed. To reduce this operating cost, one large refinery handling 13,000 barrels of distillate per day has used a novel method which has proved most satisfactory. The method has been in operation many years and is patented in all phases. Hot water is employed to melt the wax in the press, which is very seldom taken down. The presses are of the riveted-ring type, forming one-half-inch cakes. The plates themselves are of different design from the standard type. Two holes in the plate are diametrically opposite one another, one about 2 inches in diameter as near the bottom as possible and the other about 3 inches in diameter in the upper part. Thus when the press is made up there are two channels extending the length of the press, one 3 inches in diameter near the top and one 2 inches in diameter near the bottom. The arrangement of such a press and the hot-water lines are shown in figure 17. The chilled wax distillate, 15° to 16° F., enters through the 3-inch channel, and the unit operates as a standard press until it is full of wax, at which time it is shut down. Then the tierod nuts are loosened 1 inch, and hot water is forced into the press through the 3-inch channel just as the distillate was. This water soon cools and is permitted to pass out at the other end. As the water coming out of the press increases in temperature, a valve on the cold-water outlet line is gradually closed until, when the water comes through hot, the valve is completely closed. Then hot water is forced into the press from this end also. The water, with the cold-water drain line shut off, gradually builds up pressure and starts melting down the wax around the 3-inch holes, causing it and the water to pass through the blankets and run off into the trough below. Thus the hot water melts all the wax between the plates. Both the water and wax pass through the filter blankets. The plates were designed as they are because, when the standard type is used, the hot water entering through the center 2-inch hole would not melt the wax in the upper part of the press.
Consequently the water is put in near the top and on settling to the bottom will melt out the whole wax cake. The 2-inch channel at the bottom permits the water to drain out after the wax is removed. What little may remain will not cause any difficulty.

The amount of hot water used is 5 to 6 times the amount of wax removed. This mixture goes to three tanks 25 feet in diameter by 25 feet high in series, and the wax is floated off at the top, practically all coming out in the first tank. The wax rises from the water as it flows from one tank to the next, and as the wax reaches the top it automatically drains away. The wax from the three tanks runs together to storage, where it awaits sweating.

After each melting-down, hot water is sprayed onto the press from a perforated 2-inch pipe which extends the length of the press and above it. In this way the equipment is kept clean. The press is drained for several hours, during which time it cools somewhat. The plates are then set up and the tierod nuts tightened. The press is again ready for filtering. As the press has been heated during cleaning out and is considerably warmer than the chilled oil, the first oil through should be rechilled and repressed until the press is cooled to a satisfactory temperature.

CLEANING FILTER-PRESS PLATES

After filter presses have been operated for several months, in some cases as long as a year, a gradual dropping off is noticeable in the amount of distillate that can be put through the presses in a given cycle under the same operating conditions. The filter becomes sluggish, as though the amount of filtering area were reduced. Examination of the blankets at such a time shows the interstices to be plugged with fine particles of carbon or gummy, sticky wax and presenting a hard, more or less impervious, well-packed surface. To get such presses functioning properly again they must be cleaned. However, with the advent of well-fractionated uncracked wax distillates, such as those produced from vacuum units, the need for blanket cleaning is being eliminated. The impregnation of the
blankets with carbon particles and amorphous gummy waxes, closing the canvas pores and materially reducing the press throughput, is not experienced to any degree comparable to what it was. Consequently, one finds plants in which filter-press plates have not been cleaned for years, which, however, had to be cleaned regularly when older distillation equipment was used.

The plates are cleaned either in place or are taken out of the press. The cleaning agents are hot oils of lower viscosity than the wax distillate, steam, and hot water. When it is necessary to clear presses this is done about once or twice a year. In most cases cleaning of the plates is done in place. Where oil is employed, gas oil of about 35° to 36° A. P. I. gravity is used. It is heated to 150° to 160° F. and pumped into the press after it has been made up following removal of wax. The hot oil melts any wax held in the canvas, causing it to pass through and out of the filter like pressed-wax distillate. In loosening and melting away this wax much of the fine black carbon material is loosened and, with the interstices in the cotton duck opened somewhat, passes through the blanket. In some instances it has been found difficult to clean out a whole press by pumping hot oil in through the 2-inch inlet. A third or a half of the press would become clean and would permit all the oil that could be charged through the inlet to pass through the first third or half of the press. To overcome this difficulty one refiner places two special oil-inlet rings at different points in the press before it is made up for cleaning. They are 2 inches thick and have a 1½-inch pipe connection. The hot oil enters at these two points as well as at the usual inlet. In this way the oil is distributed more evenly throughout the press, and less hot oil is needlessly passed through the press before all plates have been cleaned.

One refiner using steam encountered much the same trouble as was experienced with hot oil; that is, only part of the plates were cleaned when the steam entered one end of the press. Accordingly a perforated pipe, made up in short sections that screwed together, was run through the center of the press and steam turned into the pipe. The steam then comes out all through the press. After 3 hours the steam is shut off, the press is opened, the water is allowed to drain out, and the plates are allowed to cool off for about 8 hours. The press is then made up and is again ready for service.

When the plates are removed from a press for cleaning they usually are stacked vertically in steam ovens holding about 150 plates. One method of operation is to turn live steam into these ovens for a number of hours when the wax is melted out of the plates, then to permit them to drain and dry before being returned to the press. Another method requires a cycle of operation of approximately a day. The plates are stacked in an oven with about a 1-inch space between them. They are held on V-shaped racks to which closed steam coils are attached. Above the plates are perforated hot-water coils. Hot water at 150° F. is sprayed down onto the blankets for about 6 hours. At the end of this time the plates are free of wax, and much of the carbon is washed away. After the wash steam is turned into the closed heating coils, and the blankets are dried for about 18 hours. Then the plates are dry and ready to be placed in a press.
Opinions differ as to whether hot water and steam or hot oil is best to use in press-plate cleaning. The differences arise from what effect either might have on the life of the canvas blankets. Some refiners using hot oil feel that the use of steam on the plates is detrimental. One refiner, when steaming out certain presses, noticed that the blankets seemed to rot around the buttons where the canvas had turned black. This condition was not obtained when cleaning was done with hot oil. Other refiners believe that steam is not detrimental. One stated that it gave a cleaner press than hot oil and that no increase in the rotting of the canvas had been noticed. The blankets on the cold presses at this refinery were removed about every 3 years, when they had deteriorated enough to be uneconomical for this service. They were then placed on the warm presses, where the service was not as severe.

Another factor to be considered in cleaning press plates is illustrated by the experience of a refiner who formerly cleaned the press plates with hot oil. He discontinued this practice because, after cleaning, the presses had to be in service for about a week before the desired cold test could be obtained on the oils. It was said that cleaning apparently opened the canvas and permitted some wax to pass through, mainly that which was apt to be of small crystal size. Small leaks also develop or show up in the blanket, and only after several days do they begin to plug with wax. Then the pressed oils begin to have the desired cold test. Probably in addition to these factors, the wax distillate processed by this refiner is of better quality than that formerly processed, consequently the presses can function properly without what had probably been established press-cleaning practice.

The life of the hydraulic duck blankets used in the wax filter presses is of some importance. The canvas is almost always made of tightly woven cotton threads, 19 fibers per thread, about 13 threads per inch, and weighing about 3 pounds per square yard. Data from a number of plants indicate that the life of blankets ranges from about 3 to 10 years. Three years is a short life. At most plants it is necessary to discard the canvas after 6 to 10 years. It is difficult to say why blankets in some plants last longer than others. Probably of prime importance to refiners who chemically treat the wax distillate is the effect of any chemicals left in the oil. Where no treatment is given the distillate the major factor in the life of a blanket is the wear and tear to which it is subjected when the plate is broken out of the press and the wax removed from it. Sulphur compounds in an oil may have some effect. Steam or hot water, used in blanket cleaning and combining with possible products in the oil to form weak acids, probably accounts for the statement of some refiners that these cleaning agents are detrimental to the life of the blankets.

A method of prolonging the life of press blankets has been developed within recent years, and such treated blankets are rapidly replacing the plain duck blankets. The duck is given a thin coat of a liquid copper compound that adheres firmly to the cotton fibers on the outside of the woven duck. This coating only covers the exposed cotton, for when the duck is cut it is evident that most of the cotton is untouched. The coating is not deep enough or continuous
over the interstices in the duck to retard the flow of oil through the blanket. As a result of this treatment the wax does not adhere so tightly to the blankets, and the small wax crystals do not penetrate so easily. Consequently presses are easy to break, and the wax is removed easily with resultant shorter press-cleaning time. Since there is little plate scraping to be done the wear on blankets from spud bars is decreased greatly and the life of the blanket increased. Since the wax does not penetrate the blanket, press-plate efficiency is increased.

**FILTER-PRESSING WITH SOLVENTS**

The use of solvents in the removal of wax from distillates presented a problem in filter pressing that had to be overcome before such methods could be employed satisfactorily. The solvents used are quite volatile, and as a result equipment different from that previously discussed in this chapter must be employed. The filter used at one plant is of a rotating-leaf pressure type, completely and tightly enclosed to avoid costly solvent losses and to protect operators against solvent vapors. The filter consists of a rotating filtering element housed in a horizontal pressure-tight cylinder. Figure 18 shows the construction of this type of filter. The oil-solvent-wax mixture enters the filter body under pressure and fills the cylinder. Four 4-inch inlets are at the bottom, and through them the mixture reaches all parts of the filtering element at equal pressure. The filtering element consists of 21 leaves, 48 inches in diameter, mounted at regular intervals on a 6-inch hollow shaft, the distance being determined by the thickness of wax cakes to be built upon the filter leaves. Each leaf
is composed of two round, perforated plates held about one-fourth inch apart by a coarse screen. The plates and screen are fabricated into a rigid unit by a ring at the outer edge and a hub in the center. Filter cloths are clamped on either side of this leaf by means of clamping rings. The hub is bored and key-seated to fit the hollow shaft. The leaf is divided into four sectors, each having a port into the shaft through a small tube. The oil-solvent solution, after passing through the filter cloths and the perforated plates, travels along the coarse screen and through the small tube into the hollow shaft. The four small tubes extend to the center of the shaft and, when a press is drained preparatory to dumping, trap the oil in the shaft and keep it from returning to the sectors as they revolve.

The hollow shaft receives the filtered solution discharged from the individual leaves, supports the weight of the leaves, and transmits the rotating drive. Gaskets are used to make pressure-tight joints between adjacent leaf hubs, and flanged couplings threaded on both ends of the shaft clamp all the hubs firmly together. The leaves are held in transverse alinement by bars fastened to their outer edges and parallel to the shaft. The hollow shaft is coupled to a drive spindle at one end and to a hollow filter-discharge spindle at the other. Stuffing boxes at these points prevent leakage. By disconnecting the filtering element at the couplings, it can be replaced without disturbing the main filter piping.

The filter body is divided on the horizontal center line into two halves with inspection doors in the upper section. The bottom of the shell is trough-shaped and contains a right- and left-hand screw conveyor which carries the dried wax to a central 12-inch discharge opening. A low drain connection is also provided for withdrawing all the liquid from the filter before the cake is dried and discharged.

It is necessary to drain whatever liquid is retained within the filter body before the filter is dumped. When a filter is taken off the line, inert or oxygen-free flue gas under pressure is admitted into the top, forcing all of the unfiltered mixture out of the filter. This pressure is maintained until all the liquid is also driven from the wax cakes on the leaves. These cakes are from $\frac{3}{4}$ to $1\frac{1}{2}$ inches thick. After the cakes are thus dried the wax outlet valve is opened and the pressure released, at which time the wax cake breaks free from the filter leaf and falls into the bottom of the filter. The helical conveyors are started, and the wax is discharged through the wax valve and carried away through a large pipe and conveyor to the slack-wax sump for subsequent processing.

Another type of filter used in a plant employing solvents is one in which rectangular filter plates are placed vertically in an upright cylindrical shell with a steep cone bottom. The chilled distillate-solvent mix is forced under 50 pounds pressure into the shell from the bottom, the oil-solvent solution passing through the filter plates and out of the filter to a tank, leaving the wax built up as a cake on the plates. When the desired cake thickness is reached the filter is shut off and the excess mixture drained from the shell. The wax cake is then washed with solvent to remove all oil, then the excess solvent is drained to storage. A back blow of solvent vapor through the filter leaves breaks off the wax cake; and it falls into the conical
bottom of the shell, is discharged into a pipe equipped with a screw conveyor, and is carried to storage tanks for solvent recovery and subsequent wax processing.

**SLACK WAX**

Slack wax is the crude wax produced by chilling and filter-pressing wax distillate, and at room temperatures it is a soft solid holding a large proportion of oil. Slack waxes have widely varying characteristics which are traceable to the method of wax-distillate manufacture and to possible treatment given the distillate. Slack wax of almost any characteristic can be derived from the same crude, depending on the crude processing. The nature of the crude, primarily its wax content, also influences the nature of the slack wax.

**SLACK-WAX CHARACTERISTICS**

The yield of slack wax from wax distillates ranges from 10 to 25 percent. The average from some 20 plants was 18 percent. The wide range in slack yield cannot be attributed to the crude, for average Mid-Continent oil processed by different refiners gave yields varying as widely as that just stated. The range can be ascribed to the method of processing. The refiner's operations are influenced by such considerations as the following: Whether he is interested in lubricating oils only and whether or not wax is to be considered in the light of just an impurity in the oil which must be removed and then thrown into cracking stock; what cold test he wants the finished marketable oil to have; whether he is interested in the wax as well as the oil; and to what degree he will process the wax into the various trade products. Such motives affect the quantity and quality of slack wax.

The amount of slack, however, does not necessarily indicate the yield of commercial wax that may be obtained from a distillate. Slack wax contains a large percentage of oil, entrained and sealed in the wax cake during filter pressing, which will range from 30 to above 60 percent. Thus a yield of 14 percent slack of 30-percent oil content represents more wax than a yield of 18 percent slack of 60-percent oil content, and there is less material to care for in the sweating operations. The amount of oil in slack is due primarily to the crystalline nature of the wax. It either presents a more or less porous cake through which the oil can travel or a tightly closed mass which seals in the oil. Moreover, faulty filter plates prevent the oil, if once through the wax cake, from getting out of the press. Although slack waxes have oil contents of 30 to more than 60 percent, difficulty is experienced in sweating those having the higher content. A large number of refiners do not care to produce slack wax having more than 50 percent oil. The average oil content of the slack wax that could be processed subsequently at 15 refineries is 44 percent.

When wax distillates are double-pressed it would seem that the warm pressing should yield drier wax cakes. This does not necessarily hold true. Some plants report that the warm-pressed slack is drier than the cold-pressed slack; other plants report the opposite. The percentage of slack obtained in either pressing seems to have no
effect on the oil content of either the warm- or cold-pressed slack. Where double pressing is employed the slack from both pressings usually is mixed for subsequent processing. In some cases the warm- and cold-pressed slack may be sweated separately to scales of different melting points, which can be blended to meet demand. Occasionally plants are operated so that the amount of either the warm or cold slack wax is relatively small, is very difficult (sometimes impossible) to sweat, and if mixed with the other slack impairs its sweating too greatly. In such cases these slack waxes are used as cracking stock for high-pressure cracking units or are redistilled in so-called "cracking stills" (primary distillation equipment) along with unpressable distillates to change their wax crystallization characteristics.

The melting point of slack waxes ranges from 98° F. to as high as 120° F. The majority of refineries report melting points of 100° to 105° F. Single-pressing plants average 103° F. Double-pressing plants have warm-pressed slack from 106° to 120° F. melting point and cold-pressed slack from 98° to 102° F., which when mixed have melting points about the same as the average for single-pressing plants.

The melting points of slack wax generally become higher the lower the yield from the distillates. For example, at plants employing single pressing, one obtains a 10-percent slack yield of 112° F. melting point, at another a 14-percent yield has a 106° F. melting point, and at others a 16- to 19-percent yield has a 102° to 104° melting point. At plants using double pressing one gets a 5-percent yield of warm-pressed slack, melting at 112° to 116° F., and a 13-percent yield of cold-pressed slack, melting at 100° F. At another plant the melting point of the first 5 percent on the distillate is 118° F. and the next 14 percent 102° F. Slack waxes at different refineries may have about the same melting points and yet have widely varying oil contents.

Slack waxes differ widely in color. Some range from slightly dirty yellow to almost black and others from clear golden yellow to dark olive. Where the distillate has been treated or well-processed the colors of the slack are light and clear, but where it has been severely cracked and little time has been provided for any settling the cakes are all shades of gray, and some are so dark as to appear almost black. This results from the carbon particles having filtered out of the distillate along with the wax. As stated in the section on wax distillate, some refineries treat the distillate primarily to obtain clean slack so that all wax processing beginning with pressing will be made easier.

A characteristic of slack wax that affects the amount of labor required in filter pressing is the manner in which the wax breaks out of the press. Some cakes come loose from both blankets when a plate is broken loose from the press and the wax is held in the ring. Other cakes adhere to one of the blankets. In some cases one insertion of the spud between the cake and the plate is sufficient to break the wax loose in one large piece. In other cases this must be repeated several times. Most of the time, however, the cake adheres to the blankets rather strongly. In such instances considerable force must be applied to break plates from the press and effort
exercised to scrape the blanket clean. Sometimes the cake splits in two or breaks up in pieces adhering to both blankets, due to the center of the wax cake being too oily. The center usually is wetter than the outside of the cake, and some cakes seem to be no more than a crust of relatively dry wax filled with a soft, mushy, oil-wax mixture. In extreme cases the whole cake, often called “cheese”, is not much more than a sticky, greasy mass. This characteristic of the slack wax is probably due entirely to the quantity of the wax-distillate cut taken from the crude and to its processing. When the wax breaks in pieces on each blanket the work of press cleaning is greatly increased over that ordinarily encountered.

QUALITIES FOR GOOD SWEATING

Slack wax should have certain qualities to insure that it can be sweated. Of major importance is the nature of the wax crystals that will form when the melted slack is chilled in the sweater; they should be long, interlacing, and fibrous, forming a network throughout the slack mass. Such crystals, like a mat of excelsior, constitute the body of the slack mass and hold the lower-melting-point waxes that crystallize. It is these long-needle crystals that maintain a wax cake during the sweating and remain as scale when this is completed. Without them the whole slack-wax mass would gradually melt down and carry away the higher-melting-point waxes with the result that little would remain in the sweaters.

In general, when slack-wax cakes break with fine crystalline faces or are unduly crumbly, it evidences the likelihood of a poor-sweating slack in that the higher-melting-point waxes are of small crystalline size and therefore are easy to run off with the lower-melting-point waxes and oil during sweating. Slack wax should be, relatively speaking, tough and not too crumbly. A fractured cake should show a somewhat rough face, indicative of long, fibrous crystals. A wax probably will sweat satisfactorily if a handful gathered from the slack when it is being solidified at the beginning of sweating (and is still mostly liquid) resembles a fibrous, long-needled mat from which the oil drains easily and which can be squeezed in the hand, driving out the oil and leaving a clean, rather white, relatively oil-free mass. The solidification of a good-sweating slack seems to progress gradually. First the excelsior-type mat of good, crystalline wax forms, then it fills in with the lower-melting-point waxes, all of which seal in some oil. Poor-sweating slack “sets up” rather rapidly once it starts solidifying. The total chilling period does not necessarily differ, however, from that of good slack. When a handful of poor-sweating slack wax is taken from the pans about the time it starts solidifying, one finds it to be of finer material that lacks strength, settles down, and does not have the porous nature of a mat of long-needled crystals. Compared to a mat of excelsior for a good-sweating slack, a poor one might be pictured as a coarse, fibrous wood pulp. The oil drains slowly from the wax, tending to carry away some of the crystals, and when a handful is squeezed much of it runs out between the fingers; what wax is left is not as free of oil as a good-sweating slack should be.

Slack wax should be cleaned as much as possible before sweating is attempted. Where the wax is dark due to the presence of carbon
particles they should be allowed to settle out before the wax is sweated. If not, the operation will be affected, and the sweaters and lines will plug.

The oil content of a slack wax may affect its “sweatability.” Waxes with oil contents of 55 to 60 percent and above are apt to be difficult if not impossible to sweat. High oil content and poor crystalline structure go more or less hand in hand and doubtless account for the unsuitability of high-oil-content slack wax for sweating.

TREATMENT GIVEN SLACK WAX

Of 25 refiners who manufactured commercial waxes, 9 did not treat the wax except to improve color by filtration of the plant product, 5 treated the wax distillate, 7 the slack wax, and 4 the scale or refined waxes. One wax manufacturer subjected the slack to a light distillation (distilled off some of the light oil) before sweating and then chemically treated the scale.

If the distillate has been chemically treated, the slack wax is not. Treatment given slack is of three kinds. The simplest is hot-water washing and settling. The most used is contacting with sulphuric acid, with subsequent neutralization and water washing. The third is subjecting the wax to a slight distillation.

When subjected to hot-water washing the melted slack is pumped to a tank. Here about 1 barrel of hot water at approximately 175° F. is sprayed over every 3 to 5 barrels of liquid slack. Then steam is forced up through perforated pipes in the bottom of the tank and agitates the mixture for about half an hour. The slack is allowed to settle for 10 to 15 hours, during which time all water, dirt, and carbon particles drop out; the clear slack is then run to the sweaters or to tankage. In one case where the warm-pressed slack was relatively dirty and full of carbon and the cold-pressed slack was clean, the former was treated with acid while the latter was clean enough to necessitate only a hot-water wash before sweating.

When slack wax is given an acid treatment the procedure followed depends upon the amount of water in the slack. If there is an appreciable amount the acid is added to the wax in increments; that is, a little is added first, referred to as a “cutter”, to remove the water, after which the major treat is given. When the slack can be kept in a tank in a melted condition for 1 or 2 days, the water, “B. S.”, and dirt can settle out, and the necessity for a cutter is eliminated. When the wax is melted down after being removed from the filter presses and in a short time is charged to the sweaters, a cutter usually is employed. The total amount of acid used depends upon the condition of the slack. Consequently, the amount of the cutter at different plants ranges from 1/2 to 1 1/2 pounds of 66° B. sulphuric acid per barrel of wax. After the slack is pumped to the agitator and, if necessary, brought up to temperature, the acid cutter is added. The contents are agitated by blowing for about 1/2 to 1 hour while the temperature is maintained at 115° to 125° F. It is deemed advisable not to heat above 125° F. when acid-treating, as the wax might be injured; moreover, at 125° F. the slack is low enough in viscosity to permit contacting with the acid. After blowing, the sludge and acid are allowed to settle, the time required depending upon the agitator capacity of a plant; 3 to 4 hours
usually is sufficient. However, at one plant the mixture settles for 12 hours. The sludge is then withdrawn from the agitator and the main acid treat given.

Provided the main treat is not too large it is given in one dump, otherwise two are used. The acid used is 66° B. sulphuric acid. Good slack wax is treated with as little as three-fourths pound of acid per barrel. In general, however, a total of 5 to 7 pounds per barrel is used and given in two dumps of equal amount. In the main treat the acid is added to the wax and agitated with air for about 1 hour. Light treats are agitated for as low as one-half hour. At the end of the agitation the slack is settled for 3 to 6 hours, and the acid and sludge are withdrawn. If the main treat is given in one dump, the slack usually is allowed to settle for 8 to 10 hours after the sludge is withdrawn before it is pumped to the neutralizing agitator. If the main treat is given in two dumps the second follows after the settling period of the first. It is again agitated for about an hour and then settled for about 12 hours; then the sludge and acid are withdrawn, and the slack is transferred to the neutralizing agitator. When the acid is added in one dump without a cutter the same procedure is followed, except that the period of agitation may be increased to 3 or 4 hours. In one case strong acid of 98-percent strength is used instead of the more usual 66° B., 93-percent acid. The same settling period of 8 to 12 hours is used.

After the acid treat the slack is transferred to a neutralizing agitator. Here it is contacted with a weak caustic soda (NaOH) solution the strength of which is determined by the possibility of emulsion formation. Usually caustic soda of 2 1/2° to 5° B. strength is used. However, one refiner uses one-eighth pound of caustic as strong as 16° B. per barrel of slack. Sometimes even weak caustic may be considered too active a neutralizing agent, and soda ash (Na₂CO₃) of 1½° to 2° B. strength is used. The amount of soda is about 2 percent by volume. Apparently the strength of the neutralizing agent that can be used without emulsifying the wax and forming soaps depends upon the degree of freedom of the slack wax from any acid particles. If any peppy sludge is noticed in the slack after the acid-treat settling period the wax should not be sent to the neutralizing agitator because of the possibility of emulsion formation. If all such sludge is out of the wax there is little likelihood of trouble with the neutralizing wash. Usually the caustic is added to the slack in the agitator. One refiner, however, feeds it into the suction side of the wax-transfer pump (from the acid to the neutralizing agitator), thus mixing them. Generally the caustic and slack are agitated with air for an hour or more, then washed with hot water and allowed to settle. The temperature of the slack is increased by steam coils in the agitator to around 140° F., and that of the hot water sprinkled down through the wax is 160° to 180° F. which will cause the wax to get as hot as 160° F. during the water wash. Usually about 1 barrel of hot water is used for every 3 barrels of slack. However, the amount of wash water at various refineries ranges from 15 to 40 percent of the volume of wax. If the water has been pumped in and not sprayed, the mixture will have to be agitated longer to insure washing. After the water wash the wax and water are allowed to settle for 8 to 16 hours, the water is withdrawn, and
the wax is then pumped to tankage ready for sweating. If, however, some moisture still remains after the settling period, the slack is usually blown for about 2 hours with air to dry it.

In connection with water washing or acid treatment, some operators notice a small layer of "bad wax" just above the water line and under the main body of good, clean slack. The amount of this slippery, soapy mixture of wax and water is never large—only a few barrels in a thousand—and always is less than 1 percent. The degree of fractionation given the wax distillate is said to determine the amount of this "bad wax." It has been noticed that pressing difficulties have been encountered where this substance has later been found to be present. This material is usually put back into untreated slack and is cleared up in the next treat.

Slack wax may be subjected to a light distillation. The slack is run to a shell still, and about 20 percent of it (or the light feet oil) is flashed off at a temperature of 500° to 550° F. using very little steam. The bottoms, or 80 percent of the slack, are put in an agitator and washed with an equal amount of hot water to remove any B. S. and other impurities. After settling, the slack is ready for sweating. It is claimed that, as a result of the distillation and the elimination of some feet oil, the character of the slack is changed to give it better sweating qualities.

SWEATING

Slack wax, as removed from the filter presses, contains from less than 80 to more than 60 percent of oil. The wax is composed of a large number of paraffin hydrocarbons, and while it might be referred to as a scale wax of a certain melting point (according to the test) actually it contains many waxes melting over a rather wide range. It is the purpose of the sweating operation to remove the oil and, if desired, to separate the wax into fractions of various melting points. The sweating operation is a heat treatment of the slack wax. The principle of operation is based on the fact that the various waxes making up the slack wax have progressively higher melting points. When most of the oil has been removed from crude scale, any further sweating into various cuts is essentially a process of fractional fusion. The sweating operation consists of chilling the warm, liquid slack wax until it is solid (this will suffice to convey the idea; however, in practice it seldom is chilled to a hard, solid cake), then slowly raising its temperature, during which time the entrained oil drains away. As the slack becomes warmer the lower-melting-point waxes melt and run off, leaving the desired product. It is believed that, upon chilling, the higher-melting-point paraffins form a mass of needlelike interlacing crystals to which other waxes adhere, both entraining some oil. When the wax is heated some of it melts, opening the interstices and sealed-up pores or voids and permitting the oil to flow out and drain off.

It is of interest to mention, in connection with the sweating of waxes, that different methods have been studied and tried. Of these, perhaps the method employing the principle of continuous sweating has been given most consideration. Such a principle was prompted by the desire to overcome inefficiencies in the utilization of heat and to shorten the time required for sweating. In general, the
wax would be poured into containers on an endless-belt arrangement passing through a chilling or solidifying room, then travel through a chamber or chambers in which increasingly higher temperatures were maintained and in which the purely sweating part of the process would take place. As the wax left these chambers it would be prepared to the desired oil and moisture content and melting point and then discharged from the traveling belt or bucket which would travel on to be recharged. Patents have been issued on such methods, but it is believed none is now used in the manufacture of wax.

Another interesting development, which may become of value where large amounts of wax are processed, is the application of distillation and efficient fractionation to the processing of waxes. Patents have been issued covering the use of distillation and fractionation to cause a separation of the waxes somewhat similar to that obtained by sweating. Possibly such processing could be developed to the point where, with certain types of wax charging stock, it could be fractionated into finished waxes.

**SWEATING EQUIPMENT**

Sweating equipment, or sweaters, is essentially of two types, the pan and the tank. A third type, the sweating stove, is used in numerous foreign refineries. The names describe the sweaters quite satisfactorily. In the pan type there are a number of horizontal pans in tiers. The tank type is essentially a tank filled with vertical tubes and horizontal plates. The sweating stove is a cylindrical shell in which a number of cells are placed. The pans in a pan sweater are placed in a sweating house or "oven", which is an essential part of the installation. Tank and stove sweaters are complete in themselves.

**PAN SWEATERS**

Pan sweaters seem to be built according to the requirements of the individual refiners and consequently vary widely in size and design. Figure 19 is a picture of a typical pan sweater with eight pans in a single stack oven. There is a wide difference in the size of pan-sweater installations, depending on individual conditions, recent installations tending toward larger sweaters. The capacities range from less than 100 to as high as 3,200 barrels. The majority of sweaters handle 300 to 500 barrels per charge. The pans range from 6 to 20 feet in width and 20 to 60 feet in length; the majority are 8 to 10 feet wide. Those 6 or 8 feet wide usually are 20 to 30 feet long, and those 10 feet wide and larger are 30 to 60 feet long. The depth of the pans from the screen to the top of the pan is 4 to 18 inches, depending on the thickness of cake to be sweated. The pans are arranged in stacks or tiers of 6 to 15 pans, which are usually placed singly or doubly in the ovens. However, large-capacity ovens often contain 4 stacks. The pans usually are supported by a framework of I-beam columns and crossbeams of channel iron. The distance

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from the top of one pan to the bottom of the next one above ranges from 8 to 18 inches but is usually about 1 foot. A point to remember in designing a stack of pans is that this space is valuable for air circulation over each pan. Quite often this space of 8 to 18 inches is partly filled (in some cases 50 percent or more) with a 4-, 6-, or 8-inch channel iron. Consideration should be given this feature to avoid unduly blocking the air currents.

The pans must be so designed that the water, oil, and waxes can drain out of them. Those about 20 feet in length drain to one point. The bottom may slope toward the center or toward one side at the middle. A draw-off line extends from these points to a draw-off manifold. If the pans are longer, the bottoms are divided into 10- to 15-foot sections, making 2 to 6 divisions. These sections usually drain to the center or in a few cases to the side at the center. Where pans are 20 feet wide they are divided further into two parts across their width.

Figure 20 is a sketch of a stack of pans in each of which is shown one detail, the parts shown making up a complete pan. Each pan contains a screen, a closed pipe coil, and a perforated pipe coil. The screen supports the wax cake, the coil pipes cold water during
chilling and warm water during sweating, and the perforated coil admits open steam into the pan and under the cake when the product of the sweat is being melted down.

The cold water for chilling the wax comes from the plant system, and no particular equipment is necessary except a control valve. The water at one refinery was very corrosive and dirty and frequently plugged the pan coils. Cold oil is now used instead of water as a cooling agent in the chilling operation at that plant. The wax cakes sweated are relatively thin (4 inches), and the coils are not used during sweating, oven heat alone being utilized.

Hot water for sweating, however, requires additional equipment which should be provided for each oven or group of ovens that are operated as one. Usually there is a tank equipped with open or closed steam coils that heat the water. A circulating pump is required for pumping the water through the coils in the pans, and where the coil outlets are open and the flow can be observed the water then returns to the tank by gravity feed. The temperature of the hot water is usually controlled manually by admitting steam into the tank; however, the temperature rise can be regulated automatically.

Screens.—Screens in the pans usually are made of one-fourth-inch-mesh brass screen of about no. 16 gage wire, although galvanized wire screen is also used. One-eighth-inch-mesh and even as fine as 10-mesh brass screens are used. One refiner believed the one-eighth-inch size was too fine because dirt and gumming residues would settle out and be retained on the wire, causing the cake to sweat out spotted. On the other hand, one-fourth-inch screen would permit the wax to settle below the screen meshes because of the greater heat content and conductivity of the wire, compared with that of the air around the cake, and the melting of the wax in contact with the wire. This settling would allow some of the slack to drop into the intermediate cut being sweated off. This refiner was using a 6-mesh screen of no. 18 wire. The screens usually are bolted to the sides of the pans and in small pans carry the weight of the wax cake. In larger pans the screen rests on another heavy one, such as 1½-inch mesh, or is supported at places throughout the pan by small bar-size tees or by angles or channels bolted to the sides of the pan.

A few refineries use perforated steel plates in place of screens. They are of about the same material and design as ordinarily used in the wax filter-press plates, that is, 16-gage material, punched with three-thirty-seconds-inch holes on three-sixteenths-inch centers. One refiner preferred them to screens because, during the summer months, some of the screen wires would cut through the wax and cause it to break off into the intermediate cut which was not so apt to happen with plates. Another refiner, however, stated that screens were superior, as they remained clean while the plates clogged.

One observation made at a number of plants was that the screens were not level and bulged, owing to not being drawn tightly across the pan, with the result that varying cake thicknesses were sweated. This condition leads to poorer yields.

Coils.—All sweater pans have closed pipe coils in them for chilling and sweating the wax. These coils usually are of three-fourths-
inch galvanized pipe, but larger pan installations may use 1-inch pipe. One plant observed employed 1¼-inch pipe. Sizes larger than three-fourths inch ordinarily are found where corrosion and the possibility of plugging from dirty water is encountered and where the water must travel through a long continuous coil.

Coils are arranged in a wide variety of ways depending upon the thickness of cake sweated, simplicity of construction, ease of control, and other factors. One feature, probably the prime one, which is considered by some refiners and should be by all, is that the coils should be designed so that, as nearly as possible, their temperatures in the wax cake will be the same at all places. It is evident that if a large pan was filled with one continuous coil the temperatures at the inlet and outlet would vary considerably. With such coils, the wax on the inlet side is chilled harder than that on the outlet side.

Placing of the coils depends upon the thickness of the wax to be sweated. When cakes are under 8 inches thick the coils are placed in one horizontal plane above and near the screen, but when 8 to 16 inches thick they are at two levels, one near the screen and the other some distance higher, depending on the thickness of the cake. This arrangement provides for more even heat transmission to or from the wax. When coils are at two levels in pans they may be of the hairpin type, in which the pipe runs from a header at one height, traverses the length of the pan, and returns to a header at the other level. Three-fourths-inch pipe hairpins usually are put on 3- or 4-inch centers and 1-inch hairpins on 6-inch centers. The coils may also be separate units at each elevation, like those described below. The lower coil is usually about 1 to 2 inches above the screen and the upper one 4 to 8 inches, depending upon the thickness of the cake. One installation for an 8-inch cake used less pipe surface on the upper coil which had 8-inch centers than on the lower one, which was ¾-inch pipe on 3- to 4-inch centers. Such an arrangement depends upon whether the top coil is to be used for chilling only and whether the heat for sweating the upper part of the cake is to come from the hot air in the oven or from the coils.

Pipe coils are of many designs, probably so as to furnish nearly uniform temperatures in all parts of various-sized sweater pans. The simplest is the continuous type, in which the pipe begins at one corner and goes back and forth the length of the pan, ending at the other corner of the same end of the pan at which it started. If one such coil involves too much travel for the water, two coils may be used, each beginning at the center of one end, traveling back and forth the length of the pan, and ending in a corner at the same end. At one refinery the water inlet and outlet headers are placed outside and at the ends of the 10- by 40-foot pans, which have 4 coils of about 5 runs each. Another type of coil consists of headers placed at opposite ends and connected by numerous lengths of three-fourths-inch pipe. In this type the water travels only the length of the pan. A modification of this type has two inlet headers across the center of the pan, and the water flows toward an outlet header at each end. Here the water travels only half the length of the pan. All coils of these types are set 1 inch to 2 inches above the screen and are supported by small steel members tied into the
sides of the pans or resting on the screen. The coils may also be hung from small steel members resting on the edges of the pan.

Underneath the screen on the bottom of the pan is a coil or coils, perforated with small holes, through which live steam is introduced below the wax cake when it has been sweated and is ready for the "melt-down."

**Headers.—**Closely adjacent to the stack or stacks of pans and extending alongside are headers for hot and cold water, steam, and sweated products (draw-off headers). If the oven contains two or four stacks the headers usually are grouped between them at the center of the oven. For long pans there may be several draw-off headers along the sides. The lines from the hot- and cold-water headers to the coils are equipped with control valves. The outlet of a water coil should be designed to permit observation of the water flow. Otherwise, there is no way to tell how much is going through any one coil. The outlet can discharge openly into funnels placed along a vertical outlet header, or each coil can be piped to a drum or a trough where the amount of water can be observed. Flow indicators have also been used for this purpose. The open discharge probably is the most satisfactory means of ascertaining both the cold- and hot-water flow. In one instance where the water flow could not be observed it was approximated by feeling the wax cake and estimating its temperature. This is not apt to be satisfactory.

The wax-charging header and laterals should be arranged so that, after the pans are filled, the warm slack or other waxes in the pipes drain back to their respective tanks. The draw-off headers are so arranged that each section of a pan drains into them. The discharge line from the pan to the draw-off header may be equipped with a valve for closing but generally is so arranged that it can be raised or lowered to hold or empty the contents of the pan. Sometimes, in a stack of pans these lines are held together by an arrangement whereby they operate in unison. If there are a number of sections in a pan, a draw-off header usually is provided for every two sections. These headers are designed in a number of ways, as illustrated in figure 21. One design (A) consists of cast-iron sections with four receiving cups cast to a central connecting pipe. Another (B) consists of a piece of 8-inch pipe with a number of lips welded to it where holes have been cut in the pipe. Still another type uses tangential tees coupled together with pipe nipples (C). In a fourth design (D) all draw-off lines are run down to a trough at a level that permits the lowest pan to drain into it. Then a glance at any group of pipes will tell how the sections they drain are sweating.

**Ovens.—**Because of the nature of the operations to be carried on within them, the sweating ovens and buildings must be designed with conflicting considerations in mind. The building should conserve as much heat as possible during the sweating period; on the other hand, it should permit rapid cooling of the pans and wax during chilling. The sweater ovens are subjected to repeated periods of heating and cooling; in addition, the temperatures at various places about the stack or stacks of pans should be as nearly uniform as possible during the chilling and sweating cycles. The buildings usually are of brick or hollow tile, with or without any windows and the ends made of large doors. One or two doors take up most of the end area, extend-
ing as high as the stack or stacks of pans in the oven. The oven should be placed in line with the prevailing winds of the summer months. The chilling problem is less important during the colder months. To take advantage of air currents, some ovens are built with the floor 6 to 10 feet above ground, permitting housing-in under the sweater for various purposes. The sweater run-down tanks or hot-water equipment can be conveniently located here. In several instances this space is used as a steam cellar where the steam coils that heat the oven are placed.

The size of the ovens is governed by the size and number of pans in a stack and the number of stacks in an oven. A space of 4 or 5 feet is left between the sides of the pans and the oven walls, and there are 3 or 4 feet between stacks. These spaces should be large enough to allow passage and to accommodate the various manifolds that may be installed between the pans. They should also be sufficient to permit circulation of air between the stacks. The lowest pan usually has 3 to 4 feet clearance off the floor, although some plants have only 2 feet. The highest pan in a stack should be 4 feet below the oven roof; however, in many plants this distance is only 3 feet and in some 1 to 2 feet. Since the hot air in the oven tends to rise to the top and the hot air from the oven walls circulates across the top pans more than the others, it is inadvisable to have the stack closer to the roof than 3 feet; 4 feet probably approaches the optimum distance. When the wax is close to the roof, it sweats to a greater degree than when lower, and much good scale or finished wax may be lost into intermediates.

The actual height of an oven depends upon the number of pans in a stack, which ranges from 6 to 15, and upon the depth. No particular number of pans can be said to be in more general use than another. Regardless of their individual depth, the number seems to be determined by the sweater capacity required by the plant. One refinery sweating thin (3- to 4-inch) cakes had what might be termed a “double-decked oven” consisting of two stacks of 15 pans each, one above the other with some distance between. In this instance

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**Figure 21.—Draw-off header designs:** A, Cast-iron sections bolted together; B, pipe with lips welded at proper places; C, tangential tees and pipe nipples; D, individual lines to trough.
the total height did not exceed that of many deeper pan installations. The height of an oven should be considered if means for positive air circulation are not to be employed. From floor to ceiling the usual distance is 20 feet; however, some ovens are as high as 32 feet. When heated only by steam coils on the walls, tall ovens will show too great temperature differentials between the top and bottom pans.

Sweater ovens usually, but not necessarily, have 1 or 2 large doors in each end which are opened wide to cool the oven and chill the wax by permitting the relatively cool air to circulate through the oven. The doors should be designed so that they are slightly higher than the top of the stack to provide equal cooling facilities for each pan. Where the doors are lower the pans above them will remain in more or less hot, pocketed air and will not chill equally with those below. As the sweating cycle proceeds, these pans are subjected to a higher temperature than the lower ones; in consequence their yields usually are very poor. Several aids to open-end circulation will be mentioned.

Ventilators, skylights, and trapdoors, all of which can be opened during the cooling cycle and closed during the sweating cycle, are satisfactory aids in cooling. A review of a number of plants using such openings in the roofs of sweater ovens indicates that they have an area of 3 to 10 percent of the total plan area of the oven, the average being 6 percent. Another aid to cooling a sweater is the use of windows and doors in the sides and ends, near the bottom, and/or near the top of the oven. The greater window area is usually along the top. The area of such ventilators on the sides and ends of the ovens varies, depending upon the size of the large doors, the amount of skylight and trapdoor area in the roof, and any other artificial means of air circulation. A review of a number of ovens, where windows and small doors are used as an aid in cooling, shows that the area in one side and end of an oven occupied by the windows and doors ranges from 3 to 8 percent of the total area.

Due to the more or less haphazard and at times decidedly inadequate method of wax chilling accomplished by whatever natural air currents can be induced to pass through the warm oven, mechanical means are employed to improve the rate and uniformity of chilling. These methods may be such that the large end doors can be eliminated. Forced air circulation is employed by 1 or 2 refiners as an aid to rapid, even chilling of the wax in the pans. Water coils are used at the same time. In one installation, fans on the sweater roof force air down through ducts and across every pan in the stack. The air is discharged at one end of the pans and travels between pans toward the other end. Large doors in the ends of the sweater oven are not used, and the air escapes through trapdoors in the roof. In another establishment, two concrete ducts are provided in the floors of the oven and covered with small, removable concrete slabs. Air is forced through these ducts and discharges into the oven where the slabs have been removed. In addition, the sweater has large doors at the end and a number of windows in the sides near the top.

To heat the sweater ovens during the sweating cycle proper, all ovens are equipped with steam coils, usually hung on the sides and made of horizontal runs of pipe extending the length of the oven.
With exhaust steam 3- or 4-inch pipe is generally used, but with live steam 2-inch or smaller pipe may be employed. Sometimes these coils are divided into two sets on a side to provide flexibility of heat control. In such cases, one set may be hooked up for live steam. Exhaust-steam coils are usually made with wide-pattern return bends and in many cases extend over practically the whole wall area of the two sides.

The number of square feet of coil surface used in an oven varies widely. Data from a number of plants indicate that, when exhaust steam is used, 1 square foot of coil surface is installed for 10 to 15 cubic feet of oven volume. When live steam is used, 1 square foot of coil surface is installed for 25 to 40 cubic feet of oven volume. It must be remembered that the heat losses due to the oven construction, its condition, and the rate of the sweating cycle all influence the amount of coil surface to be installed and doubtless account for the range of the figures just given.

Oven temperatures.—A temperature difference usually exists in various parts of an oven during the sweating cycle, due to infiltration of air from doors and windows and to cooling of the air by the cold walls and doors. Such temperature variations should be reduced as much as is economically possible. Where the plant product is yellow scale only and slight attention is paid to any intermediate cuts, there is little economic justification for making the oven temperature almost uniform throughout. Where, however, all the plant products are highly refined waxes and the intermediate cuts are sweating and re-sweated, until at some plants the process seems almost endless, there is ample justification for much work in improving oven-temperature conditions during the sweating cycle.

Various methods are used for producing and controlling efficient air circulation in an oven in the endeavor to have temperatures more uniform. One method is to place a sheet-metal partition 4 to 6 inches in front of the steam coils and the same distance from the wall. The partition extends from about a foot above the floor up to the highest pipe, which is not over 8 feet. Thus, a draft is created past the coils, resulting in better air circulation than if the partition were not used.

Another method is to place the heating coils in a cellar. This installation is made only when the floor is 5 to 10 feet off the ground. A number of vertical steam coils are placed in the cellar so that the hot-air column ascends along the sides of the pan stacks and between them. The oven floor is covered with steel grating, and movable solid-steel sheets are placed on the grating to protect the underside of the lowest pan and to regulate the amount of hot air and the place at which it ascends. It is a decided advantage on windy, stormy days to be able to control the place at which the hot-air currents are admitted into the oven. Another similar method is to place in the cellar a horizontal 4-inch coil extending over the whole cellar area and divided into two parts. Each part of the coil covers one half of the cellar so that the hot air in the oven will be better regulated. There is a concrete floor under each stack of pans, but the rest of the space is covered with grates through which the hot air rises.

One method, which doubtless produces less temperature variation than any discussed so far, uses circulating fans in the oven. For
example, an oven 22 feet wide, 30 feet long, and 20 feet high, with steam coils on the walls, contains two stacks of pans. In the top of the oven between the stacks are eight 16-inch ventilator-type fans; 4 take suction from one side and 4 from the other side of the oven. Each fan discharges the air into a large, galvanized, sheet-iron elbow turned down. Thus eight streams of hot air are blown downward between the stacks and dissipated by passing across all the pans. The cooled air is then heated again as it comes in contact with the steam coils on the walls.

Some operators have questioned why such consideration should be given temperatures throughout an oven or, to quote one, "What's the difference if the top pans sweat out too much and the bottom ones not enough and remain too wet? They get mixed when melted down, and the final product is satisfactory." If the maximum yield of wax with a minimum of sweating and the quality of the product are to be considered in the light of economically feasible sweating, the matter of as uniform oven temperatures as possible cannot be neglected. When tests are run on ovens it is not uncommon to find temperature differences as high as 25° F. The oven last described had temperature differences of 20° to 25° F. when operated without the fans, but with them the maximum variation in any part of the oven was 2° F.

A similar study was made a number of years ago by Bureau of Mines engineers of the temperatures in various parts of ovens during routine sweating runs at two Mid-Continent refineries. In one instance the temperatures were taken at regular hourly periods at 4 places above the wax in the top pan and at 4 places above the wax in the bottom pan. The room temperature as given by the regular sweater thermometer was also noted. In another instance the temperatures were taken above the wax near the ends of the top, center, and bottom pans. The sweater thermometer was also read. The temperatures in various parts of the ovens differed considerably—at one time during one of the sweats by 18° to 20° F. Thus, overheated spots existed where much of the good wax was sweated off.

Too often sweaters are operated by the temperature as given by the usual thermometer hung just inside one of the small entrance doors. An instance is cited of the advisability of selecting the proper location for the sweater thermometer. The sweater thermometer near the entrance door read 124° F., which was taken as the operating temperature, while at the same time the temperature above the wax in one of the pans was consistently 135° to 140° F., a 10° to 15° F. increase. The yield of wax from this particular pan was very low.

The experience of one refiner in locating the most satisfactory place for the sweater thermometer is interesting. The oven had steam coils on each side wall and contained one stack of pans. A reference point was chosen as that of the air space close to and alongside the stack of pans about half-way up and near the center of the stack. Experiments were conducted by obtaining temperatures at this point and at different places in the oven and comparing them. It was noted that during the first 6 to 8 hours of the 24-hour sweating period the temperatures at the top of the oven were not quite as

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high, within 3° to 4° F., as at the reference point, but during the latter half of the sweat they were 3° to 4° F. hotter. No information about the temperatures at the lowest pan was available. The sweater thermometer customarily hung on the oven wall rather close to the steam coils where it showed temperatures as high as 148° F. when a thermometer at the reference point read about 130° F. When the correct temperatures at the pans were used in controlling the sweat a more satisfactory and much better sweating operation was reported, with increased yields and improved quality of products. Subsequently a recording thermometer was installed with the bulb inside the oven at the desired place and the recorder outside where it could be easily read. One refiner believed that the temperature throughout an oven could vary as much as 20° F. but that this condition would exist only at the beginning of a sweat and that, at the end, temperatures would not vary over 3° to 5° F. While this may be true of some well-designed sweaters, nevertheless some tests show that the greatest differences occur toward the middle and latter part of the sweat, when intermediates are being sweated off.

Recording thermometers do away with the necessity of continually entering the hot oven and give the plant superintendent an actual record of how the sweater operates. The bulb can be placed where the temperature is the most representative instead of in a handy place such as near the door of the oven, as is the case with most sweater thermometers. Recording thermometers indicate the reasons why a sweater suddenly shows a poor yield. Formerly the plant superintendent had to be satisfied with the reply of the operator who often found it convenient to cover up some mistake by the remark. "I don’t understand it; it must be the nature of the wax."

**TANK SWEATERS**

Tank sweaters derive their name from their appearance. Tank sweaters are not used by as many refiners as are pan sweaters. Of 25 wax-manufacturing refiners, 17 used pan and 5 used tank sweaters entirely, while 3 used both types. The greater use of pan sweaters cannot be attributed to any particular superiority over the tank type. In some respects the latter type probably is superior. Refiners using tank sweaters are able to produce the same products, crude or refined, that others do with pan sweaters. One refiner, however, sweats slack to scale in a tank sweater and finishes the scale in pan sweaters because the latter can be controlled better. In this plant the water is very dirty and corrosive, causing the tubes in the tank sweater to plug and corrode. Consequently, in a tank sweater having many tubes plugged off and others partly so, uneven distribution of heat is inevitable, and poor sweating conditions result. Another refiner, using both types, states that just as good results are obtained with tank sweaters as pan sweaters. Moreover, tank sweaters are easier and cheaper to operate but are somewhat harder to cool. Their capacity usually is larger, ranging from about 200 to 2,750 barrels but mostly from 800 to 1,300. The capacity is generally proportionate to the tank diameter, since there seems to be one dimension in common, namely, the total depth of wax, which
is usually 13 feet. However, one large sweater had a 15-foot wax bed, and a battery of small sweaters had 12-foot beds. The diameters of tank sweaters range from 12 to 50 feet.

Tank sweaters are set on foundations 6 to 12 feet high, depending on the elevation of run-down tanks and on the use made of the space underneath where pumps and run-down tanks can be satisfactorily housed. The sweaters are insulated to insure uniform chilling and sweating and to conserve heat. For this purpose hollow

![Figure 22: Sketch of tank sweater showing some details.](image)

tile, placed so that there is about a 2-inch dead-air space between it and the tank, is generally used.

Figure 22 is a sketch of a tank sweater, showing some of its details. Tank sweaters are essentially tanks filled with a large number of small vertical tubes and horizontal wax supports. There is a tube sheet 1 to 2 feet from the bottom (2 feet is more desirable) and another 12, 13, or 15 feet higher, with 1 to 2½ feet of tank shell extending above this to the top. To these tube sheets 1- or 1¼-inch tubes are fastened, 1¼-inch ones being used more generally. The
tubes are on 4- to 6-inch centers, in different sweaters, depending on the distance between trays and the individual refiner's design. The number of tubes may range from 1,500 in small sweaters to about 15,000 in large ones.

Horizontal perforated plates are installed between the tube sheets every 4 or 6 inches apart, corresponding to the distance of the tube centers. These plates are usually of 16-gage material punched full of three-thirty-seconds-inch holes on three-sixteenths-inch centers so as to allow them to slip over the vertical tubes. Spools 4 or 6 inches long support these plates and are slipped over about every tenth tube; thus the wax in the sweater is essentially broken up into a number of 4- or 6-inch cakes. The opinion of one refiner, who operates a large number of tank sweaters, as to what happens during sweating is that the oil coming from the cakes percolates through the wax to the plates, and most of it works over to the vertical tubes and then runs down these to the sweater bottom. Some oil will, of course, filter through more than one cake, but it will not travel very far before it gets to a tube.

![Diagram of tank sweater with downward flow of water through tubes.](image)

**Figure 23.** Sketch of tank sweater with downward flow of water through tubes.

Water is passed through the tubes for chilling and sweating. In all cases the cold water is pumped into the bottom chamber, and it is important that as uniform a flow as possible be maintained up through all tubes. For this purpose the water may be admitted through a number of 4-inch inlets spaced throughout the bottom. Above these inlets deflector plates or baffles may be installed to diffuse the incoming stream more evenly. Some sweaters, however, have only one 6- or 8-inch water inlet in the bottom. A head of water ordinarily is carried over the tubes on all sweaters and ranges from 3 inches to 1 foot at different plants. The experience at one refinery with such cold-water circulation indicated that possibly the water channeled up through some tubes more than others. To obviate this difficulty and get a slightly colder chilling water a rather novel sweater was designed. Heat distribution during the sweating period was also taken into consideration. The sweater has an annular water space around the sides of the tank, and the water flows down through the tubes, up through the annular space between the two shells, and out at the top. Figure 23 is a sketch of this sweater,
which is 50 feet in diameter. The water is pumped first through a set of spray nozzles above the sweater and under a roof with which the sweater is equipped. The water is thus cooled somewhat before it flows down through the tubes. It comes up around the wax-filled tank, flows out into a trough at the top, and goes back to the plant system. The water flows through the tubes as a result of the pressure which equals the difference in water-head and water-outlet levels.

The water chambers in the sweater bottoms usually are equipped with steam coils to heat the water for the sweating period. However, separate water tanks with steam coils also are used. The water generally is circulated by a pump which takes suction from the overflow and discharges into the chamber, where it receives heat. In some cases, however, pumps are eliminated, and dependence is placed on the water rising through the tubes after it becomes heated in the chamber. Positive circulation by pumps probably is more satisfactory, in that the distribution of heat is apt to be more uniform. Provision is made for admitting live steam into the wax chamber to melt the wax after the sweat is over. The tubes in a tank sweater can be cleaned if enough dirt and scale accumulate to retard the flow of water or hinder transmission of heat. One refiner uses a small tube cleaner on the end of a pole and runs this down through the tubes.

SWEATING STOVES

The Alanmor sweating stove is used in a number of British-controlled refineries in Burma, Assam, Rumania, and Netherland East Indies. These stoves are of two types, the cell and the coil. They are made in different sizes, but the larger ones will be described here. The cell type is formed of a steel shell 12 feet in diameter and 20 feet high, in which 31 water-jacketed steel cells are fitted. Details of an Alanmor stove are shown in figure 24. The lowest cell forms the stove bottom and supports the others. An opening is provided from top to bottom on the shell with access doors and sight glasses. On each side of the opening are water inlet and outlet headers with lines and control valves to each cell. Two flat, circular plates, forming the top and bottom of adjacent wax cells and held apart by a 1-inch flat steel ring at their circumference, make a cell 1 inch deep in which the water travels. Division bars within are so arranged that the water will have to travel back and forth to insure even distribution. The upper side of the top plate of each cell has small steel bars welded across it on which rests an expanded metal sheet with a wire screen above it. The sheet and screen support the wax during the sweating period, and the space created by small bars under the sheets provides drainage. The underside of the bottom plate of each water cell has a series of transverse fins welded to it which rest on the expanded metal sheet below, thus supporting the tier of cells. The main purpose of the fins is to transfer heat from the circulating water to the wax. The fins are 5 inches deep and on 3-inch centers; thus a 5-inch wax cake is sweated. Within the shell access opening is a vertical wax header through which the wax is charged to each cell. Air vents are provided to release the displaced air from each cell. When a sweat is melted down the wax
Figure 24.—Alanmor patent wax-sweating stove.
discharges into the same header used for charging. The stove is insulated to provide better control and conservation of heat.

The coil type of “Alanmor” stove is essentially a tank divided into a number of horizontal compartments filled with pancake-type pipe coils. The steel shell is 12 feet in diameter and 20 feet high and is divided into 7 compartments, each 30 inches deep, containing 14 horizontal coils of three-fourths-inch pipe. The bottom of each compartment sags toward the center to effect drainage. On each compartment bottom there is a one-fourth-inch-mesh expanded metal sheet supported on studs. A wire screen covers the expanded metal sheet. In the center of the stove is a vertical pipe for charging and running off the sweated fractions. The stove is equipped with water inlet and outlet headers, a steam header, and an air-relief header; it is also insulated.

In view of the construction of these stoves, in which steel sheets are welded and riveted or a large quantity of relatively small pipe is used, corrosion may become a serious factor in the life of the equipment or dirty water may impair efficiencies and even plug the coils. At one plant it was necessary as a result of corrosion to operate these stoves with closed water circuits. The water is in a closed system of heat exchangers, deoxygenators, tanks, and the stoves themselves. Three such systems are provided for a group of stoves—one for cold, one for medium, and one for hot water. They are manifolded together at each stove and can be operated so as to give the desired temperature in the stoves.

The stoves are operated by the amount and temperature of the water circulated through the cells and permit operation similar to that of tank sweaters. Information on runs, yields, and quality of products is available in an article by H. L. Allan.43

OPERATION OF SWEATERS

PAN SWEATERS FOR SLACK WAX

After the sweater pans have been “melted down” the oven doors are opened and a new sweating cycle is begun. The end doors and any windows or ventilators are opened to cool the oven. The bottom of each pan is flooded with water to the level of the screens, then the warm liquid wax is pumped into the pans to a depth which will give the desired thickness of wax cake. At any one refinery the thickness of the cake depends upon a number of factors, chief of which are the depth of the pans, the equipment on hand, and the capacity of the sweater compared to the amount of wax to be sweated. The ability to control satisfactorily temperature conditions in the wax should also be a major factor in determining the size of cake that can be sweated in any particular oven; 6- and 8-inch cakes generally are sweated, but 4-, 5-, 7-, and 9-inch cakes are each handled by a number of refiners; 10-, 12-, and 16-inch cakes also are sweated, but usually only on slack wax, and the same generally holds true for 8- and 9-inch cakes.

At one refinery where slack was sweated in cakes 6, 10, and 12 inches thick in different ovens it was found that, under the same operating conditions, the yield of the same-quality wax in all cases was 4 percent less from the 10- and 12-inch cakes than from the 6-inch cakes. At another refinery sweating 6- and 9-inch cakes it was found that the 6-inch cake sweated better than the 9-inch. When a 9-inch cake of a certain stock is sweated compared with a 6-inch cake of the same stock, and when a product of the same quality is sweated in both cases it was noticed that a 5- to 8-percent lower yield was obtained with the 9-inch cake. The 6-inch cakes also produced better intermediates because their oil content was lower, as the oil could percolate through the 6-inch cake faster. In general, these observations and opinions are held by most refiners. Increased depths of wax mean greater distances for the oil to percolate through the wax, taking with it some good wax. If products of equal quality are obtained, there are greater temperature differentials in thicker cakes between the source of heat (hot-water coils and hot oven air) and places in the wax cake, which tend to cause lower yields. If cakes of the thickness sweated at the two refineries mentioned above could be sweated in pans equipped with proper coils and controlled in conjunction with the oven air, so that all cakes had similar temperature differentials due to having the same depth of wax to be heated by any one tube, then, disregarding what wax would be carried off in solution in the oil, it is quite likely that equal yields would be obtained. At one refinery equal yields from sweating 4- and 8-inch cakes are reported under certain conditions. Due to the fact that thinner cakes sweat faster, the 4-inch cake sweaters sometimes are operated to get the same volume throughput as the 8-inch cake sweaters. Under such conditions the 4-inch cake yields may be less than the 8-inch cake yields. This, however, is probably due to crowding the sweating cycle excessively on the smaller cake ovens.

Slack waxes are run into the sweater pans at a temperature of around 125° F., for, as previously stated, their melting points at various refineries range from 98° to 120° F. When some intermediates are sweated, this temperature may be higher. After charging the liquid wax in each pan is chilled as rapidly as possible. During the summer little attention is paid to the rate of chilling other than to increase it as much as possible. In the colder months of the year care must be exercised to prevent excessive chilling of parts of the cake. Chilling any part of the cake to a lower temperature than that necessary to obtain desirable cake conditions results only in increasing the sweating cycle and using additional heat to bring the temperature of the wax cake back to that at which chilling should have stopped. The wax cakes should be chilled, also sweated, with as uniform temperature conditions as possible throughout the cake. In the winter the top of the cake can be chilled too hard, likewise the bottom where the wax comes in contact with too cold water in the pans or in the coils. If the bottom of the cake is chilled too hard it will be difficult for the oil to percolate through. In winter one refiner uses water at 100° F. in the bottom of the pans to avoid excessive chilling. If the top is too hard the oil tends to sweat out and collect in pools on top of the cake and stay there, even after
the sweater drips have been cut from foots oil into intermediate wax. In addition, such chilling will cause the sweat to start poorly and sluggishly and require several hours of the cycle to be spent getting the sweater on stream. Proper operation of oven doors, windows, and ventilators and control of cooling water temperatures prevent overchilling.

Slack waxes rarely need to be chilled hard. Often they are chilled only until the sweater is full of a mass of wax crystals and much of the oil is still free and distributed throughout the pan. If a handful of the pan contents is then taken out, most of the oil will drain freely from the wax, which is a crystalline mass of sufficient strength and structure to retain its shape but full of interstices. As a result, the chilling process itself separates a large part of the oil from good-sweating slack wax. This fact is evidenced by the rush of foots oil when the drain lines are lowered at the beginning of the sweating period proper.

Most refiners chill the slack to 80° or 85° F. In summer nearly all the chilling is done by the water coils, and generally these are not so cold but what the wax can be chilled as rapidly as possible. One refiner states that it should not be necessary to chill any colder than 10° below the melting point of the slack. This practice would raise the chilling temperature to 90° or above. At another plant the contents of the pans are chilled until they do not feel hot to the touch, or to about 100° F. In such instances large quantities of oil will come to the top, but the wax will have crystallized and settled to the screen and water. When chilling 10-, 12-, and 16-inch thicknesses of slack wax, even to the lower temperatures of 80° and 85° F., large quantities of oil remain. The refiners who use these thick cakes for slack believe that this method of chilling is decidedly advantageous, for in a relatively short time they are able to get rid of most of the foots oil, which runs off as soon as the pan draw-off lines are lowered. Cakes under 8 inches thick seem to chill harder; at least, large amounts of free oil are not noticeable in the pans. However, the temperatures to which these cakes are chilled are not apt to be under 80° to 85° F. Some refiners chill the wax until the cake is hard, others until it feels dry to the touch, and still others until the oil is sealed in sufficiently so that, when the pan draw-off lines are lowered to withdraw the water, no oil will run off until the sweating period starts. Still others prefer to obtain a soft, spongy, granular wax cake having a relatively hard, firm bottom around the coils and a relatively hard top.

The sweating period proper begins when the wax has been chilled, every draw-off line lowered, and the water drained from the pans. The amount of foots oil coming off at this time will depend upon how solidly the wax has been chilled, usually being considerable if the slack cake is thick. The doors, windows, and ventilators are closed; steam is turned into the heating coils in the oven; and warm water is circulated through the coils in the pans. Where the wax has been chilled rather hard, the sweat usually is started with a rush; that is, the oven and circulating water are brought up to a predetermined temperature as rapidly as possible to get the sweater on stream (to have the desired amount of "drips" coming off). "Drips" is the name given to the stream of oil and wax coming from the pans. The drips are practically all oil at the beginning of a sweat and prac-
tically all wax near the end. When there is free oil in the pans at the start there is no occasion for beginning the sweat "with a rush." The water and oven temperatures can be brought up at the desired rate, for the sweater is essentially already on stream.

Sweating methods.—The sweat is carried out in a number of different ways.

(1) A uniform temperature increase is employed throughout the sweating period.

(2) The oven and water are brought up to a given temperature and held there during the whole sweat.

(3) Predetermined temperatures are established at which the foots oil and intermediate cuts are to be sweated off, and the water and oven are brought to these temperatures as soon as possible and held there until the cuts are off.

(4) A period of gradual temperature rise is combined with a period of "soaking" at a constant temperature.

Of these methods, the first probably is the most desirable, for there is likely to be less temperature differential between the water or oven and places in the wax cake farthest from the water coil or cake surface. Some differential must, of course, exist, but when the temperature rise is not uniform there are times in the sweating period when there is a wide temperature difference between water or room and parts of the cake, causing local overheating of the wax close to the heating surface.

Of the four methods of "running down a sweat" the first is employed more than any of the others. Nearly always the temperature is raised at a rate of 2° F. per hour. If the cake has been chilled rather hard the temperature of the water and room may be brought up to 95° to 100° F. as rapidly as possible to get the sweater started, after which the temperature increase is uniform. If the cake is soft and oily the temperature is increased at the usual rate. As the temperature increases slowly the sealed-up interstices and pores or voids gradually open up and permit the oil to percolate out through the cake. The foots oil will be off in 4 to 8 hours, at which time the water in the coils will be around 95° to 100° F. One refiner reports a temperature of 110° F. for the water and 120° F. for the oven. The temperatures at which refiners cut the sweater drips from foots oil into intermediates are determined by the quality of foots oil desired. The foots oil at different refineries is not the same, as will be discussed later, nor is the intermediate cut the same. When the drips are about to be cut into intermediate, one refiner opens a small door, thereby checking the rise in temperature, possibly even slightly lowering it. The sweating is retarded somewhat by this action, and it is claimed the foots oil sweats out cleaner and clearer, indicating a lower wax content or a sharper oil-wax separation at this point.

Since most installations have the pans well equipped with coils, the main source of heat for sweating is the hot water. Another source is the steam coils in the oven. The amount of water-coil surface, the amount of wax, and the area of the wax surface differ at various refineries. Consequently, the oven temperatures may be kept as near the hot-water temperature as possible or, as in most cases, about 10° F. higher than the water temperatures. In some plants oven temperatures are 20° F. higher. Quite often the temperature differential varies, becoming greater as the sweat progresses. It
is difficult to give any more concrete data on oven temperatures than that just cited. Some refiners pay little attention to the oven temperatures other than to keep them somewhere near that of the water in the coils. On the other hand, a very few use oven heat entirely in sweating; that is, the coils in the pans, if any, are not used.

The operation of the sweater with reference to where the foots oil and intermediates are cut is controlled by either the cloud point (often referred to as "cold test") or the melting point of the drips. As the drips continue to run off these physical characteristics change, due at first to the increasing wax content and then to the increasing melting points of the waxes. Each refiner decides at what point (for example, 90° F. cloud point) the stream is rich enough in high-melting-point waxes to warrant it going into intermediate, later to be resweated under different temperature conditions to recover the wax. Thus the drips are diverted from foots oil into an intermediate cut. The method of heating the wax cake has no influence on this control point. Drips are cut into intermediate at cloud points ranging from 80° to 90° F., mostly, however, nearer 90° F., and at melting points ranging from 85° to 100° F., but mostly from 90° to 95° F. One refiner controls the point of cutting the drips into intermediate by the volume of foots oil sweated off. Another uses a short-cut control method of simply inserting a finger in the drip stream, and whenever the drips have a high enough melting point to harden on the operator's finger they are cut into intermediate. Such a control method can be recommended only as a rough guide.

As the sweating proceeds the drips from the wax cake contain less oil and more wax. The melting points of the waxes coming off increase until, at the end of the sweat (determined by the quality of the scale left in the pans), they are usually within 10° F. of the melting point of the scale. The time required for getting the intermediate wax sweated off ranges from 6 to 16 hours, 8 to 12 hours being more usual. At this time the hot water in the pan coils will be 120° to 126° F. and the room temperature 130° to 145° F. As stated previously, these room temperatures cannot be definitely set for all plants, but the figures given are for the more general practice. At one refinery, where the sweating is done by the hot air in the oven, the temperature does not go over 120° F. In another, the water in the coils does not exceed 110° F. at the end of the sweat, but the room temperature reaches 140° F. At still another plant a small door is opened to cool the oven slightly and slow up the sweater. This is also done when cutting the drips out of foots oil and was discussed in that connection.

The usual procedure in sweating slack is to split the drips into a foots oil and an intermediate cut. Occasionally everything sweated off is run together. Once in a while the drips are separated into three fractions, a foots and two intermediates. One refiner, operating this way, cuts from foots into one intermediate at 90° to 95° F. melting point on the drips and into the second intermediate at 108° to 110° F. melting point. When the drips reach a melting point of 115° to 120° F. the sweat is stopped, leaving 125° to 126° F. melting-point scale. The sweat is run to this rather high melting scale so
that the refiner can get a good yield of 130° to 133° F. refined waxes without too much resweating.

Several means are used for determining when the sweat is over and the scale satisfactory for melting down. The refiner usually knows what cloud point or melting point of the drips indicates that the scale wax in the pans is satisfactory. Consequently, the "melt-down" of a sweat often is controlled by these points, which usually range from 108° to 116° F. The range of melting and cloud points is due primarily to the melting point and oil content desired for the scale wax, which properties may vary at different refineries. Quite often the point of melt-down is controlled by the quality of the scale, determined by taking samples from the pans and obtaining the melting points and oil and moisture contents. This control is usually employed when the refiner sells crude or white scale wax which must meet a given oil and moisture content. When the scale is resweated to refined waxes, it is not necessary to be so careful in controlling the melt-down point. The somewhat yellow color of scale wax will vary in proportion to its oil content. Experienced operators can usually estimate very closely the oil content of a scale wax by its appearance. As a substitute for the tests on oil and moisture content, some refiners use the color of the scale as a control for terminating the sweat and melting down.

In the second method of operating a sweat the oven and hot water in the coils are brought to a given temperature and held there until the sweat is through. This method is used by only a few refiners, and they generally employ oven heat alone. Usually the room is heated to 124° to 128° F. as rapidly as possible, or at a rate of 8° to 10° F. per hour, and held at this temperature until the scale left in the pans is satisfactory. Intermediates may not be taken, in which case whatever sweats off is run into one cut. The period for sweating by this method ranges from 20 to 30 hours.

The third method of sweating makes use of raising the water and oven temperatures to a given point as soon as possible and holding it there until the feet oil is off, then quickly raising the temperatures to another point and keeping them there until the sweat is over. The water is heated to around 100° F. and the room anywhere from 100° to 120° F., depending upon conditions discussed previously. These temperatures are held for a number of hours until the desired cloud point or melting point is obtained on the drips. When the drips are cut into intermediate the water temperature is increased to 116° to 118° F., although sometimes it is not raised higher than 110° F. The room temperature may be about the same as the water or as high as 20° F. above that of the water. These temperatures are maintained until the scale is satisfactory, that is, until it has the desired melting point or oil and moisture content.

The fourth method of sweating is used about as much as the first. The temperatures are brought up at a uniform rate, usually about 2° F. per hour and, at a certain stage, are held for sometime until the drips are ready to be cut into intermediate; then the temperatures are again raised slowly to a given point, where they remain until the sweat is finished. When operating under this method the water in the coils is heated from 80° to 85° F. to about 100° F. at a uniform rate. In some cases the sweat is started at 95° F., and
the water then reaches 105° F. The temperatures of 100° or 105° F. are maintained for several hours. It usually requires about 10 hours to get the fooots oil off. After the drips are cut into intermediate the water temperature is again raised at a uniform rate to 110° to 120° F. The oven temperatures may be from 120° to 140° F. The time required to get the intermediate off is 16 to 24 hours. More time is taken in sweating off the intermediates because often the scale must be finished, as regards oil and moisture content, for market without additional sweating. Moreover, carefully controlled, slower sweating toward the end of the period will cause sharper intermediate-scale separation, resulting in less reswearing of intermediates and higher-melting-point scale. When the drips are about ready to be cut into intermediate the operation need not be controlled as finely as later near the end of the sweat. The control on the drips, cloud or melting point, oil and moisture content, color, etc., is the same as that discussed with the first method.

The total sweating cycle (charging, chilling, sweating, and melting down) at various refineries ranges from 24 to 120 hours. At the majority of plants it is about 36 hours in winter and 48 in the summer. The difference between summer and winter at plants sweating the same size cake the year around is due to the difficulty of chilling the wax in summer. It is not uncommon to have the chilling period doubled, or even more, during the summer. Thus, plants with 6-, 8-, or 10-hour chilling periods in the winter require 15 to 24 hours in the summer. The size of cake also influences the length of the sweating cycle. The cycles employed at one refinery on the Atlantic seaboard during late spring, using different size cakes, are of interest in this respect. The sweater size, cake thickness, chilling period, and total sweating cycle are given in table 4.

<table>
<thead>
<tr>
<th>Sweater capacity (barrels)</th>
<th>Cake thickness (inches)</th>
<th>Total sweating cycle (hours)</th>
<th>Chilling period (hours)</th>
</tr>
</thead>
<tbody>
<tr>
<td>320</td>
<td>6</td>
<td>40</td>
<td>12</td>
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<td>8</td>
<td>72</td>
<td>24</td>
</tr>
<tr>
<td>3,200</td>
<td>16</td>
<td>120</td>
<td>72</td>
</tr>
</tbody>
</table>

PAN SWEATERS FOR SCALE AND INTERMEDIATES

When scale and intermediates are sweated, somewhat different temperatures and sweating periods are employed from those used for slack wax. If a refiner has sweaters in which wax cakes of different thicknesses are sweated it is general practice to use thinner cakes on scale and intermediates and thicker cakes on slack. The temperature differentials between the wax and the heating medium are apt to be smaller with thinner cakes, and since the melting-point range of the waxes in the various cuts is shorter than in the slack wax more care must be exercised in sweating scale and intermediates if the amount of resweating is to be as small as possible.
The scale or intermediate is charged to the pans and chilled with cold water, but since the wax has a higher melting point than slack the chilling temperatures are not as low. One refiner uses a cooling medium about 8°F lower than the melting point of the wax. Moreover, the water in the pans is heated to about 100°F to prevent chilling the bottom of the wax cake too much. With the temperature of the medium only 8°F lower than the melting point of the wax the cake does not become solid, and the intermediates and oil can percolate through. Another refiner chills the wax to a semisolid, spongy mass and tries not to get the cake so hard that he cannot push his fingers through it.

When scale is sweated the water usually is started through the coils at about 110°F to 115°F. The temperature may be as low as 102°F and as high as 118°F, depending upon the melting point of the scale and its oil content. The room temperatures are quickly brought up to that of the water and then raised more rapidly until, when the water reaches about 125°F, the room is usually approximately 140°F.

In sweating scale and intermediates, it is frequently the practice to take off only one cut, leaving the finished product in the pan. However, the majority of refiners split each sweat into three fractions. The actual break-up of the scale and intermediates in sweating presents a wide range of possibilities. It almost seems that each refiner, because of the products desired, their quality, the equipment available, and other special considerations, has an individual way of disposing of and finishing the products obtained from slack. Consequently, several sweating procedures of typical operations are given here. They are most easily explained by diagrammatic flow charts, as shown in figures 25 to 30, inclusive.

The actual temperatures and control conditions employed by one refiner on a 125°F to 126°F melting-point scale wax of 2½ to 3 percent oil content are as follows: At the start the water in the coils

![Flow chart of sweating operations](image-url)

Depending on how the intermediate is cut, product here can be anything from 114°-116° m.p. match wax to 120° scale.

Figure 25.—Flow chart of sweating operations; 120° F. m. p. scale or lower made at this plant. Percentage figures refer to yields.
is at a temperature of 118° F. and in about 10 hours is brought to 122° to 123° F. The room temperature is raised to 120° F. as soon as possible, then to a maximum of 130° F. during the 10 hours. At the end of this time the melting points on the drips are 123° to 124° F., and one intermediate cut is then off. The drips are cut into another intermediate. The water temperature is raised very little during the next 8- to 10-hour period of the sweat, but the room temperature may go to 140° F. At the end of that time the drips have a melting point of 125½° to 126° F., and a refined wax of 130° F. melting point is left in the pan. It is noted that the rate of temperature rise is slower than for slack—about 1° F. less per hour. The total cycle, including chilling, charging time, etc., is 36 hours.

Another refiner starts the sweat with water at 100° F. and raises it 2° F. per hour to 130° F., at which temperature it is held until the melting points on the drips are 125° F. and the refined wax remaining in the pans has a melting point of 130° to 132° F. and a satisfactory oil content.
The major factor to be considered when scale and intermediate sweating operations are finished is the oil content of the product, which usually governs the melt-down point. If the oil present is over the amount allowed the sweat is continued, even though some good wax will be run off into an intermediate cut. The sweating cycle on scale ranges from 30 to 60 hours, of which the sweating period proper requires 20 to 36 hours.

TANK SWEATERS

In the operation of tank sweaters the wax is pumped into the tank at the top and fills it gradually. Before the wax is put in some refiners pump in water 4 to 6 inches deep, depending upon the perforated plate spacing, so that after the wax is chilled the water can be drawn off, and a space is left in the bottom of the sweater into which the oil will drain. It seems that this is more desirable than to fill the sweater completely with wax, as probably very little of the wax between the first perforated sheet and the bottom of the sweater is left after a sweat is over.

The wax in the sweaters is chilled as rapidly as possible by passing water up through the tubes. Since outside temperatures have little effect on such sweaters, compared to pan sweaters, and since the tubes are spaced uniformly, it should be possible to chill the wax without getting it too hard. However, it is necessary to insure even distribution of the chilling water. Some refiners have noticed channeling and have guarded against it by using distributors and baffles in the water chamber. Slack usually is chilled to about 80° F. As
Figure 29.—Flow chart of sweating operations; percentage figures refer to yields.

Figure 30.—Flow chart of sweating operations; cuts not named are intermediates. Separate numbers refer to melting points of cuts. Percentage figures refer to final yields.
soon as the wax is chilled steam is admitted for heating the water in the chamber.

One method of sweating employed is that in which the rate of production of drips is kept as constant as possible. Hot water at 130° F. is circulated up through the tubes at once. At first there is a rush of oil; however, this soon slackens. At this point the temperature of the water is lowered somewhat and the amount circulated reduced. Then, as the drips continue to come off, the temperature of the water and the volume circulated are increased to produce a uniform stream of drips, the size of which controls the sweat. The water temperature is soon 130° F. again, at which it remains until about 4 to 5 hours from the end of the sweat, when it is raised gradually to 135° to 140° F. The sweating period is then completed. One refiner, operating this way, believes that the temperature of the water can be as hot as 20° F. above the melting point of the scale wax and still not hurt the operation. However, 10° to 15° F. is the usual differential. The scale from the above operation has a 120° F. melting point, and the temperature of the water, as stated, is 135° to 140° F. At this plant all the drips below 90° F. in melting point go to foots oil and all above, until the sweat is through, to one intermediate, which is resweat for 120° to 122° F. melting-point scale. This same refiner has tried various methods of operating tank sweaters—by uniform temperature increase, by increasing temperature in jumps and holding at each for a while, and by using a constant temperature. The operation described belongs to the last method, and as good results are obtained by it as by any other method.

This refiner noticed that, under certain operating conditions, the sweated wax in the top of the sweater was honeycombed with numerous channels as though some of the wax had melted. This condition was thought to have been caused by heat "banking up" in the top of the sweater. A new-type sweater, described on page 79, was designed, in which both the chilling and sweating water were caused to pass down the tubes. The result was that there was no overheating in the top of the sweater, and an even distribution of water down each tube was insured.

Most refiners operate their tank sweaters with a gradual temperature rise in the water of about 2° F. per hour. The drips are left in foots oil to the 90° F. melting point, then cut to intermediate and left until they have a melting point of 110° to 118° F. Drips of 115° to 118° F. will leave a scale of 125° to 130° F. melting point; those of 110° to 112° F. will leave a scale of 120° to 122° F. melting point. A 1,300-barrel sweater will get the foots oil off in about 8 hours and the intermediate in 10 to 14 hours. The final water temperatures are not over 126° to 130° F. One refiner controls the water temperature by the melting point of the drips. At first the water is 2° to 3° F. hotter than the melting point of the drips, at the middle of the sweat it is about 5° F. hotter, and at the end it is from 12° to 15° F. hotter. A thermometer was placed in the wax bed 3 feet from the bottom by one refiner who wished to be more certain of the actual temperature conditions of the wax in a sweater. Chilling was stopped at a wax temperature of 87° F.; then heating was started.
and continued as rapidly as possible to 90° to 95° F., after which an even rise of 2° F. per hour was followed to 114° to 119° F., when 124° to 126° F. melting-point scale was left. The rate was reduced to 1° F. per hour for the last 3 hours of the sweat. If higher rates than 2° F. per hour had been used at the beginning the intermediate cut would not be as easy to sweat, although the scale would be just as good. Because of this difficulty a rate was used of not over 2° F. If the rate was lower than 2° F. the operation was deemed uneconomical.

The sweating cycle on tank sweaters is 30 to 40 hours and is more or less uniform the year around. It may be a few hours longer in summer, because the cooling water is somewhat warmer than in the winter, but since the wax is not cooled below 80° F. there is not much difference. Chilling will require 12 to 16 hours and sweating about 18 hours.

Scale wax and intermediate cuts are resweated to about the same extent as they are with pan sweaters, and the products made are the same.

SWEATING EFFICIENCY

Of interest in sweating operations, regardless of type of equipment used, is a study of the amount of sweating done to get a given yield of finished wax. A figure indicating the total number of barrels of wax sweated and resweated to obtain 1 barrel of finished wax can be used as a measure of the efficiency of the operations. Such a figure will depend on the sharpness of the separation obtained in sweating off cuts. It was reported at one plant that intermediate cuts were sweated and resweated until the yield of wax was only 10 percent of the charge. Figures 25 to 30, inclusive, show the amount of recycling and resweating that is done. With sweating carried on to yields as low as 10 percent and with cuts not well separated, a large volume of recycling can be done.

Data on a large plant whose operations were similar to those shown in figure 28, taken from yearly figures on the amount of finished waxes compared with the amount of sweating done, indicate that for each barrel of finished wax 8.37 barrels were sweated. Moreover, for every barrel of slack sweated 1.93 barrels of materials were processed after the slack sweat. Data from another plant, whose operations were similar to those shown in figure 27, indicate that for each barrel of finished wax 7.02 barrels were sweated, also that for every barrel of slack wax sweated 1.58 barrels of materials were processed after the slack sweat. One plant operating as shown in figure 25 sweated 3.73 barrels to obtain 1 barrel of scale. Another plant handled 3.50 barrels to get 1 barrel of scale. It is impossible thus to compare one plant with another unless they operate alike and on the same quality of slack wax. For instance, the number of barrels sweated per barrel of finished product for a plant operating as in figure 25 is influenced greatly by the quality of the slack and finished product. An analysis from time to time of the amount of sweating done at any plant will give a figure that measures its efficiency.
About 10 years ago Bureau of Mines engineers\textsuperscript{44} analyzed some slack waxes from 13 refineries to determine the amount of wax in the samples and compared these data with the actual plant data on yields from slack sweating as reported by the refiners. The amount of wax in the slack was determined by an acetone solvent method.\textsuperscript{45} The yield of a given melting-point scale reported by the refiner, divided by the amount of the same melting-point wax found by analysis, was considered to be the efficiency of the plant when sweating slack. The efficiencies ranged from 23 to 85 percent and averaged 57 percent. This study indicated that much of the wax had to be recycled through the plant, in many cases being lost.

**PRODUCTS OF SWEATING**

**FOOTS OIL**

As previously stated, slack waxes have oil contents ranging from 30 to more than 60 percent. Most of this oil comes off in the first part of the sweat and is termed “foots oil.” The melting point of the foots-oil cut at several plants ranges from $85^\circ$ to $90^\circ$ F. The separation of the foots oil is not sharp enough, due to the nature of the sweating process, but that it contains some wax. Consequently, if sufficient equipment is available this oil is processed to recover the wax. One refiner experimenting with foots oil mixed it with equal volumes of pressed oil and chilled and re-pressed it, obtaining a yield of about 25 percent (figured on the foots oil) slack wax. This experiment substantiates information from some other plants that foots oil contains about 8 percent scale wax. The amount of foots oil in slack wax charged to the sweaters ranged from 35 to 65 percent. The majority of 16 refiners report yields of 45 to 55 percent. The average of these 16 plants is 49.5 percent. At plants where no intermediate cuts are taken and where everything off until the sweat is through is termed “foots” the amount may reach 70 percent. However, as there is no definite point at which refiners see fit to split the drips into foots and intermediate the percentages of both vary widely.

Fofts oil is handled in a number of ways. Most of the refiners return the foots oil to wax distillate to be rechilled and re-pressed. It may be returned either to the raw or cracked wax distillate. (Some plants subject the wax distillate from the primary distillation to light “cracking” to obtain a better-pressing distillate.) However, the foots oil may be mixed with a slop cut from the primary distillation, cracked with it, then chilled, and finally pressed for wax removal. One refiner charges foots oil to a still and reduces it to 50-percent bottoms with steam to avoid decomposition. The bottoms are then sweated and yield 48 to 50 percent of $118^\circ$ F. melting-point wax. This bears out the experiments of another


refiner. The wax thus recovered is mixed with the regular plant slack wax and processed. The 50 to 52 percent of oil from the sweat is used as pressure-still cracking stock. Some refiners accumulate the foots oil from various operations and sweat it. A slack wax is obtained that is mixed with the slack from the presses. When foots oil is sweated thick bodies of oil can be processed. No difficulty is experienced in chilling too hard since the foots oil does not cake up. Due to favorable conditions the wax crystallizes out in good needle form and permits easy and rapid sweating. Most of the oil drains away when the water is withdrawn from the pans, and the sweating cycle can be short as it is sweated to a relatively low temperature. A large number of refiners dispose of the foots oil by using it for pressure-still cracking stock.

INTERMEDIATE WAX

The properties of the intermediate cut from the slack sweating have wider variations than do those of foots oil, because both the beginning and the end of the cut are determined by varying operating conditions and by the products desired. The amount of this cut ranges from 12 to 30 percent of the slack when finishing a sweat to 120° to 124° F. melting-point scale and increases to 35 percent for 126° to 130° F. melting-point scale. The average amount of the intermediate cut at 13 refineries running to 120° to 124° F. melting-point scale is 18 percent of the slack. The melting point of the intermediate wax at several refineries ranges from about 100° to 110° F. The oil content of the average intermediate cut is about 50 percent.

The intermediate cut is processed by being sweated either by itself or by mixing it with the slack wax. If the cut is sweated by itself, subsequent intermediates and products are usually combined with similar ones from the resweating of scale and sweated to the various melting-point refined-wax products. Figures 25 to 30 show how intermediates are processed at several refineries.

SCALE WAX

Scale wax is the product obtained from sweating slack wax. Scale wax has a yellow tinge as a result of its slight oil content. At the end of a sweat the scale in the sweaters looks porous, and when some is picked up it breaks away with a fibrous, stringy surface. Generally speaking, slack of 100° to 105° F. melting point and 44 percent oil content is reduced to scale of 122° to 124° F. melting point and 2 percent oil content by sweating. The term "scale" refers not so much to a wax of any particular melting point as it does to a wax whose oil content has been reduced to 3 percent and in most cases to 2 percent or less. Scale waxes have melting points ranging from 118° to 130° F. By far the greatest number of refiners sweated slack to a scale of 120° to 122° F. or 122° to 124° F. melting point. The average yield at 14 refineries sweating to 120° to 124° F. melting-point scale is 33 percent of the sweater charge. The individual plants had yields ranging from 25 to 45 percent. The range in yields, while affected by the sweating technique and control exercised, probably is due to the quality of slack which, as previously discussed, varies widely.
The amount of oil in the slack, ranging from 30 to 60 percent, naturally affects the yield, since scale waxes have a relatively low and uniform oil content. When slack is sweated to a 118° F. melting-point scale the yield is somewhat higher (around 35 percent); when sweated to 126° to 128° or 128° to 130° F. melting point the yield can be expected to be lower. Two plants producing 126° to 128° F. melting-point scale report 30-percent yields.

Scale wax, if sweated originally to a low enough oil content, can be filtered and marketed as crude scale, or it can be resweated to refined waxes of all grades, from match waxes of 108° to 110° F. melting point to waxes of 132° to 134° F. melting point. Figures 26 to 30 furnish some information on how scale is resweated and how the intermediates are combined and sweated to commercial products. Crude scale wax as sold usually is of two grades, yellow and white, and each grade usually has two melting-point ranges. Scale sweated to a 2 percent or lower oil content can be sold directly as yellow crude scale and generally has a melting point of 124° to 126° F. This grade is used where an oil content of 1 or 2 percent and a petroleum odor or taste are not objectionable. White crude scale is the yellow crude scale that has been filtered through fuller’s earth or bone char to a desired color. If, as a result of filtering, the color has been removed sufficiently and if the oil content of the scale is under 1 percent the scale is sometimes referred to as a semirefined wax. White crude scale usually is made in 122° to 124° F. and 124° to 126° F. melting-point grades. It is ordinarily colorless, odorless, and tasteless when first manufactured but may not remain so. Crude scale waxes are sold, first, on their color, whether yellow or white, and then on their melting point. The color to which white scale is finished is determined by the use to which the purchaser wishes to put the wax and probably more often by the refiner’s self-imposed standards. The color of white scale wax usually is 1 or a little above on a 6-inch cell (between 2 and 3 on a 12-inch cell) of a Lovibond tintometer with standard 500-series glasses, or approximately an 18–20 color using a Saybolt chromometer.

**REFINED WAXES**

The refined waxes are so termed because they have been sweated and processed until, in addition to having the proper melting-point range and being practically free of oil, they are odorless and tasteless and will remain so for a long time. They should be colorless and very stable to light and heat; otherwise color will develop after exposure to either. The color of refined wax, if it can be said to have any, is usually about one half on a Lovibond 6-inch cell. However, some refiners finish their refined waxes to a color of 3. Where the Saybolt chromometer is used refined waxes have a color of about 25.

Refined waxes are made up into a great number of grades, determined by the melting point of wax desired by the individual purchasers. The melting points of refined waxes range from 105° to 108° F. to as high as 132° to 134° F. The largest volume sold is usually of 118° to 120°, 122° to 124°, and 130° to 132° F. melting point. There are no standard grades to which wax must conform, nor is it likely that there are any specific uses requiring only one
grade. Consequently, there is considerable overlapping. Grades of 105° to 118° F. melting points usually are referred to as match waxes, and for about every 2° of melting-point range up to 134° F. another grade of wax is available. Thus the National Petroleum News lists in its “Wax Markets” fully refined waxes of the following grades: 123° to 125°, 125° to 127°, 128° to 130°, 130° to 132°, 133° to 135°, and 135° to 137° F. American melting point.

The American melting point is arbitrarily taken as 3° F. higher than the A. S. T. M. paraffin-wax melting point.46 The term “American melting point” (from the “American” method of determining wax melting points) probably came into usage as a result of comparison with the “English” method and the English melting point. The English method was more satisfactory and was standardized and adopted by the American Society for Testing Materials. Melting points by the A. S. T. M. method D87–22 give results about 3° F. lower than the old “American” method. The term “American melting point” continues to be used, however, but is now 3° F. higher than the result obtained by the A. S. T. M. method and the “English” method.

While many grades of wax are sold most refiners make only two or three. Even those who can furnish all grades—and there are comparatively few—do not carry all of them in stock but blend certain grades to furnish intermediate grades or operate their sweepers to produce wax to meet specifications. Certain grades are in greater demand than others. The following approximate figures on the monthly production of wax give an index of the grades and the relative amounts of each that may be produced at large wax-manufacturing refineries:

<table>
<thead>
<tr>
<th>Scale</th>
<th>Tons</th>
<th>Melting point</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>105°–108° F.</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>117°–120° F.</td>
<td></td>
</tr>
<tr>
<td>300</td>
<td>118°–120° F.</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Refined wax</th>
<th>Tons</th>
<th>Melting point</th>
</tr>
</thead>
<tbody>
<tr>
<td>400</td>
<td>122°–124° F.</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>127°–129° F.</td>
<td></td>
</tr>
<tr>
<td>125</td>
<td>132°–134° F.</td>
<td></td>
</tr>
</tbody>
</table>

Refined waxes are white or colorless, more or less translucent, and feel slightly greasy. Good, refined, solidified wax shows a translucent rather than an opaque whiteness. The wax must be free from oil if it is to be odorless and tasteless. The oil content is measured by a test for the expressible oil and moisture.47 The well-known press method is used to determine the oil and moisture in scale and refined paraffin waxes of melting points at or above 108° F. It has been established that the expressible oil and moisture content do not bear a direct relation to the actual oil content of a given sample of paraffin wax. However, it is extremely difficult to determine actual oil content by any known method. The above method has been in almost universal use for many years and doubtless comes nearer to giving the information desired than any other test devised so far.

By this method refined waxes usually will show from zero to less than 0.5 percent oil and moisture content.

In general, the yield of refined waxes ranges from 80 to 95 percent of the yield of scale wax, depending upon the degree and efficiency of sweating. Since, as previously stated, the average 120° to 124° F melting-point scale yield is 33 percent of the charge to the sweaters, the average refined-wax yield is 27 to 31 percent of the slack. Since individual plants have scale yields ranging from 25 to 45 percent it is likely that some plants have refined-wax yields higher than 31 percent.

**TREATMENT GIVEN SWEATED WAX**

**CHEMICAL TREATMENT**

Makers of refined waxes resort to some kind of chemical treatment in the manufacturing process to produce a finished wax of the desired qualities. Such treatment makes use of sulphuric acid to remove coloring matter and unstable constituents. A final filtration is given the wax to remove foreign particles and water and to improve the color. The chemical treatment of wax distillate and slack wax has been discussed. Some refiners treat either the scale or refined waxes. Of 25 wax-manufacturing refiners consulted, 1 treated the finished wax with acid, 3 treated the crude scale before sweating to refined grades, 7 treated the slack wax before finishing to scale or refined waxes, 5 treated the wax distillate which was subsequently processed to scale or refined waxes, and 9 did no chemical treating. Virtually all refiners filtered the wax, scale or refined, when finishing the product.

Crude scale wax is treated in much the same manner as distillate and slack but must be heated to around 150° F. It is reported that higher temperatures seem to “burn” or “scorch” the wax. The scale is kept at this temperature in insulated agitators. The amount of acid used in treating depends upon the quality of the scale. One refiner employs a cutter of 1 pound per barrel followed by the main treat of 3 pounds per barrel. Another treats with 10 to 15 pounds per barrel in one dump. After the acid has been added to the wax and thoroughly agitated it is allowed to settle for a time, and the acid and sludge are drawn off. Any acid in the wax is then neutralized with soda ash or caustic soda, the strengths of which vary, depending upon what the different refiners have found advisable to use. In one instance 1 gallon of 5° B. soda ash is added per barrel of wax treated. In another, 10° to 15° B. caustic soda is used. The wax is then washed by spraying hot water down through it until it becomes neutral, after which it is settled for 8 to 12 hours until it is clean and free of water. It is then ready to be sweated further. One refiner treats scale by charging it to an insulated tank, stirring and heating it with live steam for 2 to 3 hours, then allowing the wax to settle for 6 hours. It is claimed that the live steam cleans the wax, takes out a slight distillate odor, aids in settling out dirt and grit, and eliminates the usual slimy collection that accumulates on top of the filters.

Most refiners hesitate to treat finished waxes with acid because of the possibility of injuring their quality by “burning” or “scorch-
ing.” However, finished waxes can be treated, for at one large refinery finished waxes are treated at 180° F. with 20 to 30 pounds per barrel of 66° B. sulphuric acid in one pump. Practically 80 percent of the acid is not affected and after the treat remains in the bottom of the agitator to be drawn off and used again. The use of this large amount of acid results in excellent contact between the wax and acid and assures a very clean product with a stable color. It is claimed that this wax will last for years without changing in color or acquiring an oily odor. The sludge from such treating is very dilute. Since this sludge must be disposed of and since the acid-recovery plant at this refinery operates better on a dilute sludge, it is advisable from a sludge-disposal standpoint to use a large amount of acid. After the sludge and acid have been withdrawn the wax is allowed to settle for 8 to 12 hours to insure settling out of the acid and sludge particles. Any acid in the wax is then neutralized with a 4° B. solution of caustic soda. The amount of caustic consumed in neutralizing the wax is about one-half pound per barrel. After neutralization the wax is washed with about 25 percent of its volume of water sprayed down through it. The wax is then settled for 16 to 24 hours to remove the water.

FILTERING THROUGH FULLER’S EARTH

All wax except yellow scale is filtered to improve its color. Most refiners use fuller’s earth, but three or four use bone char. The fuller’s earth used by the various refiners is granular and of three sizes. The majority use a 30/60 earth (maximum and minimum grain size), some a 15/30 earth, and others a 60/90 earth. The finer sizes are more efficient owing to their greater surface area, but the degree of fineness that can be employed is limited by such mechanical difficulties as the rate of wax filtration and the losses to be encountered in revivification of the earth. More detailed information on the physical properties, analyses, tests, uses, and revivification of fuller’s earth is given by Hatmaker and Middleton.48

Fuller’s earth is practically always revivified, after it has lost its decolorizing power, by passing it through a kiln or furnace to burn out the adsorbed materials taken from the wax or lubricating oils. This revivification can be repeated numerous times before it is necessary to discard the earth. Since the earth can be revivified it is possible to use several grades or “burns” of earth in filtering waxes.

Practices differ as to the earth used for filtering waxes. Some refiners place the earth in wax service and keep it there until it is discarded. Others use it first in either wax or lubricating-oil service then later in the other service; in some cases it is used first for lubricating oil, then for wax, and then for oil again. More refiners use new earth on wax than any other grade or “burn.” These refiners employ new earth in their percolating filters, and when the wax begins to come through at a darker color than desired the filter is cut out of service, dumped, and the earth revivified. After revivification, the earth is placed in lubricating-oil service. In one or two

PRODUCTS OF SWEATING

plants this second burn or no. 2 earth is also used on waxes. The reasons given for using new earth on waxes are that the yield (barrels of wax filtered to color) per ton of earth is greater on new than on reburned material and results in less filter washing and dumping, also that earth in wax service is less damaged than in oil service; that is, it can be revivified to a greater efficiency than earth that has been used on lubricating oils.

The next largest group of refiners use new earth for filtering light lubricating oils, such as spindle oil, and on oils to be finished to a light color, then, after revivifying it, they use it on wax. Some of the refiners, using no. 2 earth on wax, return it to lubricating-oil service as no. 3 earth after it has been used on wax and revivified. At one plant nos. 2 to 5 earth, inclusive, were used on wax, and earth older than no. 5 was returned to heavy-oil service. These refiners use reburned earth in wax service because they want the more efficient earth to finish certain oils and in one or two instances to eliminate an earthy taste and odor which, it is claimed, new earth imparts to the wax. Other refiners using new earth do not experience this difficulty with an earthy taste and odor. Possibly the wax may be affected thus by earth from some sources or its method of preparation. One refiner noticed that freshly filtered wax occasionally acquired a slight taste or odor from new earth which, however, disappeared after the wax had stood in a tank for several days.

Some refiners use new earth on wax and continue to use it reburned 6 to 8 times, after which it is discarded. At a few plants old earth is used, in one no. 8 burn is used which, after it is spent, is thrown away.

Each time the earth is used and revivified its efficiency as a color-removing agent is reduced. In addition there is a loss due to the earth being gradually broken up into small particles termed “fines”, which are screened out and discarded. It seems likely that the type of earth-revivifying furnace and its operation, as well as the use to which the earth has been put, contribute to the degree of lessened efficiency after each burning and to the production of “fines.”

The revivifying furnaces are of three types:

(1) A vertical or angle gravity-drop furnace, in which the earth drops vertically through baffled passages or slides over a series of slopes and meets hot gases from a burner at the bottom of the furnace. The impurities held by the pores of the fuller’s earth particles are burned out, and the earth leaves the furnace freed of most of them. This type of furnace has been practically superseded by the following types.

(2) A rotary-kiln type, consisting of a long, cylindrical chamber installed at a slight angle to the horizontal, which receives the spent earth fed in at the elevated end. Due to the rotation of the kiln the earth gradually advances to the lower outlet. As the earth works down along the kiln it meets and comes in contact with a countercurrent of hot gases which burn out the hydrocarbon material removed from the wax. This type of furnace is used extensively in the industry. Figure 31 shows a rotary kiln used for revivifying fuller’s earth. The small kiln beneath the burner cools the hot earth discharged from the burner. The bins and conveyors are used for handling the earth.
(3) The multiple-hearth roasting oven, which was designed originally for roasting ores and concentrates in the metallurgical industry but within the last decade has been applied to the revivification of fuller's earth, for which duty it seems admirably suited. A large number of refiners now use multiple-hearth furnaces. Figure 32 shows such a furnace. The earth fed in at the top is propelled first inward and then outward across 6 to 8 succeedingly lower hearths. A countercurrent of hot air and gases admitted to several hearths from oil- or gas-fired combustion towers alongside the burner sweeps across the hearths, freeing the earth of its adsorbed materials.

Wax-percolating filters are cylindrical vertical tanks with manholes at each end and with false perforated bottoms placed above the bottom manholes or outlets to retain the fuller's earth yet permit the melted wax to pass. These are of various sizes, holding 1 1/4 to 25 tons of earth or about 4 feet in diameter by 8 feet high to 10 feet in diameter by 20 feet high. The larger plants generally use 10-, 15-, 20-, and 25-ton filters; smaller plants use 1 1/2-, 2 1/2-, 3-, 4-, and 5-ton filters.

There is considerable variation in the amount of wax that can be filtered through a given amount of earth before the latter becomes so contaminated with impurities that the wax drops below a given color. This variation is a result of the quality of the earth, whether new, second burn, etc., as well as the cleanness of the wax to be filtered. The filter-yield figures from 14 plants on which these data were obtained ranged from 15 to 100 barrels of wax per ton of earth (weight of wax figured as 275 pounds for a 42-gallon barrel or 6.5 pounds per gallon). Most of the refiners, however, reported yields of 20 to 50 barrels of wax per ton of earth. Two plants where old reburned earth was used reported yields of 15 to 20 barrels per ton. One wax plant formerly had exceedingly poor wax distillate to process and old, poorly designed, inefficient sweatners; and as a result the filtering yield on scale wax was as low as 10 barrels per ton. After efficient fractionating equipment had been installed, on the same wax-distillate-producing shell stills this plant, using the same sweatners, increased the filtering yield practically fourfold. One plant that treats scale wax with live steam before filtering and finishing obtained filtering yields of 100 barrels per ton of new earth. One refiner who uses earths nos. 2 to 5 on wax stated that on filtering 118° to 120° F. melting-point scale yields of 32 barrels per ton were obtained with no. 2 earth, 28.5 barrels per ton with no. 3 earth, and 25 barrels per ton with no. 5 earth.

Wax is filtered hot through fuller's earth. It is heated to temperatures of 140° to 180° F., depending upon the melting point of the wax.

FILTERING THROUGH BONE CHAR

Bone char is made by the destructive distillation of the bones of domestic animals. Like fuller's earth, it is ground to varying degrees of fineness. Bone char was originally used almost exclusively in filtering mineral oils and petrolatum. It has almost been superseded by fuller's earth. Bone char usually is revivified in R. S. Kent bone-char revivifiers and apparently is reconditioned each time sufficiently so that it can be used repeatedly. The Kent revivifier con-
Figure 31—Rotary kiln, showing discharge end.
The molding press shown in figure 33 is made up of molding frames and cooling plates arranged alternately on channels between stationary and movable heads, the latter actuated by a hydraulic ram. Each molding frame holds two cakes. The sides of the frames and cooling plates extend above the cake-molding area and form an open reservoir along the top of the press when it is set up. The cooling plates are hollow and fitted inside with an arrangement of baffles. Each plate is connected to a water-supply pipe, located underneath, by means of rubber hose, so that the plates can be moved without breaking the connections. The water enters the plate at the bottom near one side and is discharged at the bottom near the other side, circulating up and down between the baffles to give maximum cooling. The water is discharged directly into the plant sewer system or, by a second set of rubber-hose connections, to a waste pipe. The usual capacity of the presses is 200 cakes; that is, they have 100 molding frames and 101 cooling plates and consequently handle a full ton of wax at each charge. However, the number of molding frames in a press varies. One refiner uses 144 in each machine.

The press operates as follows: The cooling plates and molding frames, placed alternately between the stationary and movable platens, are set up by the hydraulic ram which actuates the movable platen. Cold water is then circulated through the plates, and the molten filtered wax, piped from elevated storage tanks over the molding room to the top of the presses, is run into the press as rapidly as possible, filling the molds and part of the reservoir above them (providing for wax shrinkage upon cooling). After the wax has become sufficiently chilled so that no further shrinkage will take place, the surplus on the top is scraped off. The movable platen is then pulled back by the ram when the molds and plates are separated, and the wax cakes are readily removed. The molding cycle is 2½ to 3 hours with an average melting-point wax and water at 70° F. Refiners using warmer water report 3 to 3½ or 4 hours, or 6 to 8 dumps per press per day. One refiner reports operating on a 1½-hour cycle with cold water.

The other method of molding wax is referred to as the "pan method." At one plant using such equipment, aluminum pans, each holding about a 10-pound cake, are held in concrete troughs and partly immersed in cool water, which is circulated in the troughs. The troughs are arranged in stacks seven tiers or shelves high. Each stack is long enough to accommodate 10 pans and consequently has a capacity of 70 pans. A number of stacks are lined up in a row, and a number of rows are housed in one large molding room with a passage between every two rows. A traveling automatic filler hangs from a rail above each passage and fills two pans in each shelf of a stack, or a total of 14, at one time. The molten wax from storage above the molding room enters an automatic filling chamber which, when it contains 10 pounds, shifts itself off. For each discharge nozzle there are two such measuring devices, so that one is filling while the other is discharging into a pan. The pans are "turned-over" about three times a day, the total daily output of wax being approximately 35 tons. After years of use the pans at this plant were still smooth and not bent or distorted.
Figure 33. Ten-pound-cake, wax-molding press.
FIGURE 34.—Wax chipping or barreling machine.
Success in molding satisfactory wax cakes in pans, according to the refiner whose molding plant has just been described, is due to having the wax at the proper temperature when it is poured into the pans. This temperature should be 145° F. (118° to 120° F., 122° to 124° F., and 130° to 132° F. melting-point waxes are molded at this plant), then the wax cake will not stick to the aluminum pan but will shrink and break away upon chilling and, when the pan is inverted, will fall out.

At another plant using aluminum pans and molding 20-pound cakes the pans were filled by a mechanism somewhat similar to the one just described, but the chilling was done by the atmosphere. Molding was extremely slow under these conditions. Moreover, many of the wax cakes were difficult to break loose from the pans, and it was necessary to strike them against the rack to break out the cake. Such handling bent and distorted the pans and resulted in malformed wax cakes.

As soon as the wax is caked it is placed in boxes or bags for shipment. One company uses bags holding 132, 160, and 220 pounds. The 132- and 160-pound sizes are made of double 16-ounce burlap and the 220-pound size of double 10-ounce burlap. These three sizes are used in export shipments. Quite often for domestic shipments the burlap bags are lined with cotton cloth to insure cleanliness. The bags are packed and sewed by hand. Some caked wax is placed in wooden cases, but the large majority is shipped in burlap bags.

**BARRELING**

Most of the wax not molded into cakes is put through a wax-chipping machine and barreled or bagged for shipment. The chipping machine is essentially a chinning machine which changes the hot, melted wax into a warm, pliable, semisolid sheet that is easily packed in barrels or bags, where it slowly solidifies to a compact mass. A chipping machine is shown in figure 34. It is a slowly revolving, hollow, cylindrical drum, rotating in a horizontal plane on a hollow shaft. The drum, 3 feet in diameter and usually 5 feet long, is mounted above a shallow pan so as to dip about one-half inch into a constant-level bath of melted wax. Cold water enters through the hollow shaft and keeps the drum cool. At some refineries relatively warm brine from the wax plant is circulated through the drum. As the cool cylinder revolves, a film of wax adheres to it. This film, about one-sixteenth inch thick, becomes sufficiently solid in three-fourths revolution of the drum to be scraped off in a flexible sheet by means of a knife scraper pressing against the drum. The thin sheet of wax is still warm and falls through a guiding chute into a barrel or sack. An attendant packs the barrel or sack tightly with as much soft wax as possible. Occasionally a mechanical packer is used to fill the barrels. A wax-chipping machine with a 3- by 5-foot drum is rated at a capacity of about 6 barrels per hour, which can be varied by changing the temperature of the cooling medium and the speed of the drum. Double chipping machines are also manufactured which have drums 8 to 10 feet long and fill 2 barrels or bags at one time.

The barrels used for shipping wax are not necessarily of the same size nor are all packed alike. In general, a slack barrel will contain
about 240 pounds of wax. The barrels for waxes of 118° to 120° F. melting point or higher need not be tight, but those for shipping match waxes of 115° F. melting point or lower should be tight.

OTHER METHODS

Wax may be shipped in tank cars. Such shipments are usually interrefinery or to large consumers with facilities for melting and handling 8,000- to 10,000-gallon lots at their plants.

MOLDING INTO SMALL CAKES

Most of the wax manufactured in the United States is handled by one or more of the methods already described. However, some wax, probably less than 1 percent, is made into small one-fourth-pound cakes for household use and into candles. Relatively few wax manufacturers supply the demand for these small cakes, and only 2 or 3 refiners manufacture candles. These small one-fourth-pound cakes are molded in presses that are similar in principle to the large 10-pound-cake molding presses described previously. In one such press (see fig. 35) each mold frame has a capacity of 10 small cakes; in a standard machine there are 60 frames and 61 cooling plates. Thus 600 cakes, or 150 pounds of wax, can be molded at each charge. The wax cakes when broken out of the press are put into 1-pound packages and cased for shipment.

In another type of one-fourth-pound molding machine the melted wax is poured into slightly tapering molds between which cold water is circulated. The bottoms of the molds are movable but fit closely to the walls, thus preventing the wax from escaping. An elevating mechanism pushes the bottoms up into the molds and breaks the wax cakes loose so that they are accessible. This machine requires no ram.

MOLDING INTO CANDLES

Candles are made by pouring melted wax into candle-molding machines which are somewhat similar in principle to the one-fourth-pound cake molding machine just described. Figure 36 shows a candle-molding machine. Several hundred molds (cast of high-grade tin) are built in a heavy cast-iron mold box through which cold water is circulated around the molds. These molds are traversed by pistons which form the bottom of the mold and have the shape of the candle tip. These pistons are attached to an elevating frame, and after the wax is chilled the candles are forced out of the mold, lifted into receiving racks, and clamped there. The wicks are fed from spools through the center of each piston and held taut between the candles in the receiving racks and the tight-fitting holes in the pistons until the next run of candles has solidified, when those in the racks are cut loose. Wick is made of cotton, braided or unbraided, especially treated with chemicals so that it will be consumed completely in burning without smoking.

If the molded white candles are to be colored they are hand-dipped in dyeing vats and become covered with a thin film of colored wax. If special finishes requiring higher-melting-point waxes are to be given candles they are dipped in waxes such as carnauba and sperma-
Figure 35.—Quarter-pound-cake, wax-molding press.
Figure 36.—Candle-molding machine.
ceti. Spermaceti is an animal wax obtained from sperm oil taken from the blubber and head cavity of the sperm whale. Carnauba wax is a vegetable wax which forms as a coating on the leaves of a species of palm. When long, slender tapers are made, it is necessary to finish them by hand-dipping the molded candle several times in higher-melting-point waxes. Higher-melting-point waxes add mechanical strength to the candles, which are naturally rather fragile and are easily bent or misshaped. The sheathing of higher-melting-point waxes about the molded taper also aids in keeping the taper from dripping when lighted because the sheathing does not liquify as readily as the candle proper; forming a cupped edge which holds the melted wax until consumed. Considerable technique must be developed in the manufacture of candles, especially the finer kinds. At one large refinery about 4,000,000 pounds and at another about 2,200,000 pounds of wax a year are made into candles.

**WAX-PLANT ARRANGEMENT**

The arrangement of wax-plant equipment from the distillate tanks through the finished-product loading platform usually is influenced by other considerations than those relating purely to the wax plant. If considered for itself alone the plant would be compact and all wax operations carried on in equipment assembled and housed in one section of the refinery. The wax must be handled hot to stay liquid and be pumped about the plant. Consequently only a few short wax lines are desirable. In very large wax plants, where it is possible to have the treating equipment, filtering plant, and earth or bone-char burners solely in wax service, an ideal lay-out is feasible. In small plants, where the amount of wax processed does not warrant separate treaters, filters, earth-handling equipment, etc., it is more economical and convenient to pump the wax to where the plant agitators and filters, with earth conveying and burning systems, are located and then to the barreling and packaging house alongside a railway spur.

One finds few complete, well-laid-out wax plants. The larger plants were not designed and built at one time and have been added to at intervals. Small wax plants do not warrant compact arrangement. Wax plants should be designed so that the flow of material requires the least pumping and that one process follows directly after another. A rather well arranged wax plant is shown in figure 37.

In such an arrangement the distillate is processed to remove the wax in one large building. The refrigeration equipment is housed at one end of the building with the coils on the second floor. The brine travels direct to the chillers; some may go to keep the press-rooms cool and then return to insulated storage tanks. The wax distillate is obtained from tanks close by and is pumped direct through the chillers and into headers in the pressrooms. The equipment is laid out so that it can be added to easily, keeping the same arrangement. Any additions to refrigeration, chillers, and presses would have to be on the left side, the tankage on the right preventing any increase in this direction. The presses are so arranged that they can be taken out through the end wall if necessary. Doors could be installed in the outside wall opposite each press ram, but because of
The desirability of insulating the pressroom and because of the infrequent necessity for removing rams it is probably more desirable to build this solid, arranging the brickwork so that a hole can be made easily opposite each ram when necessary. Tanks for pressed oil and wax are placed in galleries at the end of the pressroom and partly below ground level. A filter-press-plate and blanket-repair room is close at hand. The pressed oil is pumped from the small gallery tanks to large tanks close by, whence it is pumped to the lubricating-oil plant. As much of the oil as possible is pumped through heat exchangers, to aid in chilling wax distillate or in lowering the pressroom temperature, before it is pumped to the lubricating-oil plant. The slack wax is pumped from the gallery tanks to storage in part of a battery of insulated tanks standing between the two buildings. At one end of this battery of tanks are insulated acid, neutralizing, and water-wash agitators for treating the wax.

![Diagram of wax plant](image)

**Figure 37.—Arrangement of compact wax plant.**

The sweater building has a pump room in the center and the necessary pan sweaters on each side. The pump room houses all the pumping equipment for this part of the plant and the necessary hot-water systems for sweating. Sweater run-down tanks for foot oil, intermediates, and finished waxes are below ground, alongside the sweater building and near the pump room. At one end of the sweaters are housed two wax filters. The filtered wax gravitates to tanks and is pumped to elevated storage tanks above the molding, barreling, and packing room, at the side of which is a shipping platform alongside a spur track, or close to a wharf if the wax usually is shipped by water. An actual plant arranged approximately as described processed about 4,000 barrels of Mid-Continent wax distillate (around 500 barrels of slack) per day, with an output of 250 or more barrels of finished cake wax.

A plant such as this presents one feature (the handling of fuller’s earth) that under some conditions can be improved upon in larger
plants. When old earth is used on wax and is discarded after becoming "spent" such a lay-out is very satisfactory. Where the earth is reburned after becoming unsuitable a possible improvement can be made. If the amount of earth used per day was enough to keep a roasting furnace operating a furnace could be installed to one side of the filter house with earth bins above the filters and the furnace, and conveying equipment could be installed in the filter and burner house for handling the earth. A serious objection, however, to putting a burner at such a place arises from the possibility of contaminating the filtered waxes with dust and "fines" coming from the burner. The two wax filters in the foregoing arrangement do not use enough earth to keep a revivifying furnace operating. Consequently the filters must be charged manually and the used earth transported to another part of the refinery to the burner storage bins for revivifying. If bone char were used in the filters, continuous conveying equipment and a char burner could be installed for handling and revivifying the char, as a bone burner handles about 5 tons per day while a multiple-hearth furnace handles 75 to 100 tons of fuller's earth per day.

At most plants, however, the refrigeration machines, chillers, and presses are close together. The sweaters may or may not be near at hand. For treating, the wax is usually pumped to the central agitator treaters. For filtering, it is usually pumped to the filter house containing all the plant filters; after filtering, it is pumped to a wax molding, barreling, and storage building.

PERSONNEL

The personnel of a wax plant depends upon the size of the plant in terms of distillate handled and wax produced, the amount and type of equipment considered necessary to process the distillate and wax, and the quality and nature of the wax made. Information was obtained from several refineries on the number of operators and the labor required in their wax plants, and these figures were averaged in determining the values given below. In analyzing this information the data were divided into two groups, one for plants running from 1,000 to 2,000 barrels of wax distillate per day and one for plants running from 5,000 to 13,000 barrels of distillate per day. For plants running less than 1,000 barrels of distillate a day it is difficult to obtain information on the amount of labor required, since the amount of distillate and wax handled is not sufficient to keep men occupied full time on the various wax-plant processes. The number of man-days of 24 hours required for refrigeration, chilling, and pressing in 1,000- to 2,000-barrel plants is 2.87 per 1,000 barrels of distillate; in 5,000- to 13,000-barrel plants this number is reduced to 1.87 per 1,000 barrels of distillate. In the calculation of these figures a man, such as a treater, working an 8-hour shift is considered as 0.33 man-day. It will be remembered that in the section on pressing an instance was cited of a refiner handling 13,000 barrels of distillate a day, whose filter-press construction was such as to eliminate the need for press cleaners. At this refinery the number of man-days required for refrigeration, chilling, and pressing is as low as 0.38 per 1,000 barrels. Where formerly 100 men were re-
quired on more than 100 presses when the presses were of the usual design, now only 6 men are employed.

The number of man-days (of 24 hours) required for sweating, treating, pumping, filtering, and caking in plants of 5,000 to 13,000 barrels of distillate is 1.14 per 1,000 barrels of distillate. In these plants most of the wax is made into 10-pound cakes and a small amount into one-fourth-pound cakes. Thus, the total wax-plant labor in the large refineries is 3.01 man-days per 1,000 barrels of distillate. The data on the 1,000- to 2,000-barrel distillate plants were not adequate to warrant any deductions on the number of men required for the various processes from sweating to barreling. The figure 3.01 man-days per 1,000 barrels of distillate in the larger plants is the sum of the figures obtained for each wax-plant process. These processes and the percentage of labor used by each are as follows: Refrigeration and chillers, 12; pressing, 50; sweating, 14; treating, pumping, and filtering, 6; molding, weighing, and packing, 18.

Reviewing the data in the light of the amount of wax produced, one finds that the large plants require 4.0 to 5.5 man-days to produce 10 tons of refined caked wax, the average of five large plants being 4.87. The data on the 1,000- to 2,000-barrel distillate plants, while not adequate to be directly comparable with those of the 5,000- to 13,000-barrel plants, indicate that about the same number of man-days are required to produce 10 tons of wax. The 1,000- to 2,000-barrel plants require more man-days per 1,000 barrels of distillate for refrigeration, chilling, and pressing than do the 5,000- to 13,000-barrel plants, but the small plants do not as a rule finish the wax any further than scale and ship in barrels or tank cars. Consequently, fewer men are required for sweating, treating, pumping, filtering, and caking at the smaller plants. This accounts for the fact that the average figures for both sizes of plants are about the same.
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**Notes:**
- The index contains entries related to various aspects of wax processing and properties, including chemical characteristics, distillation, and processing methods.
- Specific pages are referenced for detailed information on these topics.