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**PARAFFIN AND CONGEALING-OIL PROBLEMS**

**BY**

**C. E. REISTLE, Jr.**

**WITH A CHAPTER ON A LABORATORY STUDY  
OF ROD WAXES**

**BY**

**C. E. REISTLE, Jr. and O. C. BLADE**

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between the Bureau of Mines, Department of Commerce,  
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# PARAFFIN AND CONGEALING-OIL PROBLEMS<sup>1</sup>

By C. E. REISTLE, Jr.<sup>2</sup>

## INTRODUCTION

The physical and chemical nature of many crude oils is such that they often deposit waxy material commonly called paraffin, congeal at normal winter temperatures, or are naturally so viscous that they present many difficult problems while being produced, transported, and stored. These problems have long been recognized as particularly aggravating and difficult to control or prevent. Mention of these troubles can be found in the earliest discussions of production problems,<sup>3</sup> but little work has actually been done to establish the factors responsible for the deposition of paraffin and to determine methods for properly handling congealing and viscous oils.

Some phases of the problems are encountered in nearly all of our major producing areas. Atmospheric temperature is a dominant factor in the troubles due to deposition of paraffin and the congealing of certain oils after their removal from the wells. Naturally the greatest difficulties of this character are experienced in areas where the winters are long and severe, as in the Rocky Mountain and north-eastern Appalachian districts. Atmospheric temperature, however, has no bearing on deposition of paraffin in the wells and producing sands.

The most pronounced troubles from congealing oil and deposition of paraffin are encountered in oil fields that produce paraffin or mixed-base crude oils having a pourpoint of 20° F. or above; however, paraffin troubles often are serious in fields where the pourpoint of the oil is much lower, such as the Grass Creek and Elk Basin fields, Wyoming, which produce oil having a pourpoint below 5° F. Analyses of 319 crude oils taken from major oil fields of the United States<sup>4</sup> show that 52 fields produce oils having a pourpoint of 20° F. or above, 13 of which have a pourpoint above 40° F. During the winter months it is often a serious problem to handle these oils in the flow lines and pipe lines besides the year-round troubles occasioned by the deposition of paraffin in the wells.

Difficulty is also experienced in producing and handling viscous naphthene-base oils when they have a Saybolt Universal viscosity of 1,000 seconds or more at 100° F. Pipe-line pressures and efficiencies are appreciably affected when the Saybolt Universal viscosity is

<sup>1</sup> Work on manuscript completed April, 1931.

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<sup>3</sup> Bone, J. H. A., *Petroleum and Petroleum Wells*: Philadelphia, 2d ed., 1865, p. 26.

<sup>4</sup> Smith, N. A. C., and Lane, E. C., *Tabulated Analyses of Representative Crude Petroleum of the United States*: Bull. 291, Bureau of Mines, 1928, 69 pp.

greater than 250 seconds. There are approximately 50 fields in the United States producing oils having a Saybolt Universal viscosity greater than 250 seconds at 100° F., and 21 of the fields produce oil with a Saybolt Universal viscosity of 1,000 seconds or more at 100° F.

Lack of information on the problems of handling congealing oils and oils from which paraffin deposits, and inadequate or faulty methods of preventing them, not only cost the petroleum industry large sums of money each year but frequently reduce the rate of recovery and the ultimate amount of oil that can be taken from the sand reservoir by present production methods.

#### ACKNOWLEDGMENTS

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In presenting this report the writer wishes to explain that many of the methods described are not original but are the practices developed and used in different localities. It is hoped that a discussion of their merits and a broader distribution of the knowledge on this subject will prove of value to the industry.

#### SCOPE OF REPORT

This report is in two parts—the major or first part is a discussion of the results obtained from field studies of the factors responsible for the deposition of paraffin and the congealing of oil and of practical methods of combating these problems. The second part of the

report deals with the analyses of crude waxes or paraffin obtained from different representative crude oils. It has been possible from the investigations that were made to determine the relative importance of the various factors and to develop methods of controlling conditions so that the paraffin and congealing-oil troubles could be modified or prevented. In many instances it was found impracticable or impossible to prevent the deposition of paraffin; it was then necessary to use methods for removing the paraffin at regular intervals. Numerous methods have been developed for this work and they are discussed with the view of emphasizing their relative value and practicability.

Very little has been published on the characteristics and constituents of petroleum rod waxes, and it is hoped that the information presented in the second part of the report will add to the knowledge on the subject.

A bibliography of the articles and books the writer found useful in his work is given at the end of the report.

## **DIFFERENT PHASES OF PARAFFIN, CONGEALING-OIL, AND VISCOUS-OIL PROBLEMS**

### **PRODUCTION PROBLEMS**

The accumulation of paraffin within the oil string of wells producing paraffin or mixed-base crude oil constitutes a great production problem. Usually the paraffin is deposited as a hard, granular coating generally extending to the top of the well from a depth where the temperature of the surrounding formation is approximately the same as that at which the oil is saturated with wax. Many conditions or factors affect the deposition of paraffin and in some wells it has been found deposited throughout the entire length of the oil string. Deposited paraffin gradually closes the pipe unless it is removed frequently or prevented from accumulating. In natural-flowing and gas-lift wells paraffin acts as a choke, which gradually decreases the production and in time completely stops the flow of oil unless removed. In gas-lift wells the accumulation of paraffin can be detected readily by a gradual increase in the input gas pressure required to flow the well; in natural-flowing wells it is more difficult to detect, as the decline in production caused by the paraffin is often mistaken for the normal decline of the well and this results in loss of a portion of the available production.

Paraffin deposited in the tubing of pumping wells does not affect immediately the quantity of oil produced, because the pump will continue to deliver approximately the same amount of oil until the opening in the pipe is greatly reduced. As the opening in the pipe becomes smaller, however, more power is required to pump the well; and the additional resistance to flow of oil up the tubing causes more rod failures, split tubing, and worn pump parts. When the wells are pumped by electric units the additional load often causes the motors to overheat, which shortens their life. It is therefore advisable, if the deposition of paraffin can not be prevented, to watch wells that "paraffin up" and remove the deposited paraffin at regular intervals.



Viscous oils, when flowing naturally or with the gas lift, require more energy to bring the oil to the surface because of the greater resistance offered by the oil; it is also necessary to use more power when lifting viscous oil with mechanical pumps. Neither of these conditions can be remedied without changing the character of the oil by heating, using "wet gas," or adding a lighter oil fraction to reduce the viscosity of the oil.

#### SAND PROBLEMS

The deposition of paraffin on the face of the producing sand or in the pores of the sand body, while not as common an occurrence as the accumulation of paraffin in the oil string, is a much more serious and difficult problem to detect or remedy. Usually this problem is more predominant in fields that have passed their flush production period. Paraffin accumulations in the sand or on the face of the sand also cause a gradual decline in the production of oil and gas that is often mistaken for the natural decline of the well, and unless this condition is corrected loss of production is incurred and the sand may be damaged permanently.

Although viscous oils do not deposit plugging material in the pores of the sand, they do offer greater resistance to movement, and it is difficult to obtain a satisfactory recovery of oil from the sand in a reasonable time. In producing viscous oils the problems do not pertain to the removing of materials which interfere with the flow of oil, but rather to changing the character of the oil or the natural conditions in the sand in order that oil will flow more rapidly into the well.

#### TRANSPORTATION PROBLEMS

Atmospheric and ground temperatures are responsible for most of the troubles that occur in the transportation of oils that congeal or deposit paraffin and in pumping viscous oils through long pipe lines. When the congealing or viscous oils are cooled their viscosity increases rapidly, and they often require such a high pressure that it is impracticable or impossible to move them through the lines without first reducing their viscosity by heating or mixing them with lighter gravity oils.

Although deposits of paraffin on the interior walls of pipe lines seldom plug the lines, a very small quantity deposited decreases the efficiency of the lines appreciably; consequently, it is advisable to remove such paraffin at frequent intervals. The deposition of paraffin in field-flow lines is often a serious problem, and it is not uncommon to have these lines completely plugged with paraffin or congealed oil. Frequently it is uneconomical to bury field lines or to provide means for heating them, and in some fields during cold weather the production of oil is often stopped because it is impossible to keep the field lines open. When the accumulation of paraffin can not be prevented without great expense precautions should be taken to remove it at regular intervals so that the line will not become plugged.

### STORAGE PROBLEMS

Congealing and viscous oils give very little trouble in storage but often require heating before they can be moved from storage. Heating increases evaporation losses unless the tanks are vapor tight. Paraffin often separates from the oil and settles to the bottom, forming waxy "tank bottoms" which should be put back in solution by heating before the oil is drawn from the tank. Where emulsion is present also in the oil, the tank bottoms may require special treatment before they can be mixed satisfactorily with the good oil. These bottoms usually contain a large percentage of wax, and because of their unfavorable appearance they are often drained into sumps and burned or disposed of otherwise. This practice, however, is a waste of crude oil because these tank bottoms can usually be treated profitably and "run" with the pipe-line oil.

### SPECIAL PROBLEMS

In some fields where water is produced with oil enough mineral salts crystallize or are deposited in flow lines to cause considerable trouble. Mineral salts sometimes completely plug the tubing and working barrels, and they sometimes coat the face of the producing sand and retard the rate of flow of oil into the well.

Cavings, loose sand, and precipitated mineral matter are often a major factor in the troubles encountered because they greatly increase the bulk of paraffin deposits and tend to plug the producing sand.

### CHARACTERISTICS OF DEPOSITED PARAFFIN

The deposits of crude wax or paraffin that accumulate in flow lines, in oil strings of wells, and on the face and in pores of the producing sand consist of very small wax crystals that tend to agglomerate and form granular particles of wax about the size of the grains of ordinary table salt. Deposited paraffin also contains gums, resins, asphaltic material, crude oil, sand silt, and in many instances water,<sup>5</sup> in addition to the wax crystals. The deposit varies in consistency from a mushy liquid to a firm hard wax, depending primarily upon the amount of oil present. Because the oil gradually "sweats" or drains from the wax particles, the hardness of the paraffin and the quantity of oil associated with the wax depends upon the length of time accumulation has taken place and the manner in which it was deposited. Table 1 shows that the melting point of paraffin taken from wells in different fields varies widely. The percentages of oil, gums, resins, and asphaltic material in some crude paraffins are given in Table 2. Very little loose sand or cavings were present in the wells from which the paraffin samples were taken, but it was not uncommon to find 50 or 60 per cent of loose sand or shale in the deposited paraffin in other samples.

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<sup>5</sup> Mills, R. van A., *The Paraffin Problem in Oil Wells: Rept. of Investigations 2550*, Bureau of Mines, 1923, 11 pp.

TABLE 1.—*Oil and silt content of crude paraffins*

Field and State	Producing sand	Melting point, °F.	Per cent by weight	
			Oil	Silt, sand, water, etc.
Salt Creek, Wyo.....	2d Wall Creek.....	165.4	35.0	2
Do.....	do.....	154.6	38.0	15
Lance Creek, Wyo.....	Dakota.....	181.0	24.0	4
Panhandle, Tex.....	Granite Wash.....	141.2	25.0	7
Marietta, Ohio.....	Macksburg Stray.....	135.2	30.0	6
Clay District, W. Va.....	Big Indian.....	161.9	32.0	2
Bradford, Pa.....	3d Sand.....	132.1	33.0	9
South Mountain, Calif.....	.....	158.6	20.0	12
Church and Fields pool, Tex.....	.....	164.6	38.0	3

TABLE 2.—*Oil, wax, gums, resins, and asphaltic material present in crude paraffin after removing suspended material*

Sample	Per cent by weight			
	Oil	Wax	Gums and resins	Asphaltic material
Panhandle, Tex.....	25.0	58.0	16.0	0.5
West Virginia.....	32.0	51.0	15.0	None.
Pennsylvania.....	33.0	57.0	8.0	.1
Salt Creek.....	38.0	48.0	11.0	2.2

The solubility of different fractions of paraffin wax in oil decreases with an increase in melting point and molecular weight,<sup>6</sup> therefore the higher melting-point waxes present in the oil separate first from solution. The wax fractions found in paraffin range from the lowest to the highest melting-point fractions present in the oil; the quantity of the different melting-point fractions in the deposited paraffin depends, therefore, upon the conditions that caused its separation and deposition. Fractions of wax with melting points ranging from 100° to 191° F. were found in different paraffins. The paraffins found in different wells in the same field will differ considerably in the amount of different melting-point fractions of wax, as the conditions causing their deposition vary with each individual well.

### FACTORS GOVERNING SOLUBILITY AND DEPOSITION OF PARAFFIN

The wax, gums, resins, and asphaltic material (paraffin) deposited or separated from the crude oil during production and transportation were originally in true solution in the oil. The alteration of certain conditions destroys the equilibrium so that the oil can no longer hold them in solution and causes separation and deposition. Certain definite factors are changed during the production and transportation of crude oil and affect the solubility of one substance in another. Preliminary laboratory and field investigations indicated that several of these factors are important in controlling the separation and

<sup>6</sup> Sullivan, F. W., jr., and McGill, W. J., Solubility of Paraffin Wax in Oil: Ind. and Eng. Chem., vol. 19, No. 9, 1927, pp. 1042-1045.

deposition of paraffin. The first of these factors is temperature, and in solutions of wax in oil determinations showed that the solubility of the solute in the solvent decreases with a decrease in temperature. The second factor controlling the separation and deposition of paraffin is the relative proportion of solvent to solute. At any definite temperature and pressure there is a fixed ratio between solvent and solute in a saturated solution, and if a portion of the solvent is removed from the system a proportionate quantity of solute will separate from the solution.

The third factor is the pressure exerted on the solution. As a general rule, the solubility of solids in liquids decreases with an increase in pressure where the system obeys Raoult's law or deviates from it in a positive direction;<sup>7</sup> however, if the system deviates in a negative direction from Raoult's law, increasing the pressure in most cases will increase the solubility. A solution of wax in oil deviates in a positive direction from Raoult's law, therefore the solubility of paraffin in crude oil is decreased with pressure. The fourth factor controlling the solubility of paraffin in crude oil is the amount of gas in solution. Theoretically, gas in solution may increase or decrease the solubility of paraffin in oil, depending entirely upon the resulting solvent formed by the dissolved gas and oil. If the dissolved gas in the oil is a better solvent for paraffin than the oil itself, the solubility of the wax will be increased, whereas if the dissolved gas is a poorer solvent for the paraffin the solubility will be decreased. The fifth factor is the presence of water, but the effect of water on the solubility of paraffin in oil can not be expected to be appreciable due to the fact that water is almost insoluble in either oil or wax. The sixth factor is the action of gums, resins, and colloidal asphaltic material in the oil, which undoubtedly exert an influence on the growth of the wax crystals when they separate from the oil. The seventh factor that affects the solubility of paraffin in crude oil depends upon the quantity of fine sand and silt in the oil.

Besides the seven enumerated factors that control or exert an influence on the solubility of paraffin in crude oil, certain other conditions cause the accumulation of paraffin after it has been precipitated from solution in the oil.

Before the problem of preventing or correcting paraffin troubles in the field is undertaken some knowledge of the effect of altering the factors and something of their relative importance must be at hand. The complex nature of crude oil and the different fractions of wax present made the determination of these facts a difficult problem, but after considerable preliminary work methods and apparatus were developed that proved satisfactory and gave the desired laboratory data.

#### **METHODS AND APPARATUS USED FOR DETERMINATIONS OF SOLUBILITY**

The writer believes that many readers of this report will not particularly care to read a description of the laboratory procedure followed and the apparatus used; therefore this information has been placed in the appendix, and only the results are discussed in the following pages.

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<sup>7</sup> Hildebrand, Joel H., *Solubility*: Am. Chem. Soc. Monograph, Chem. Catalog Co., New York, 1924, pp. 61, 149, 161.

## EFFECT OF TEMPERATURE

The solubility of a given solid in a liquid increases with an increase in temperature in almost all types of solution, but the extent of solubility varies greatly with different substances. Knowing the effect of temperature changes on the solubility of wax in crude oil is important, because the temperature of the oil changes appreciably

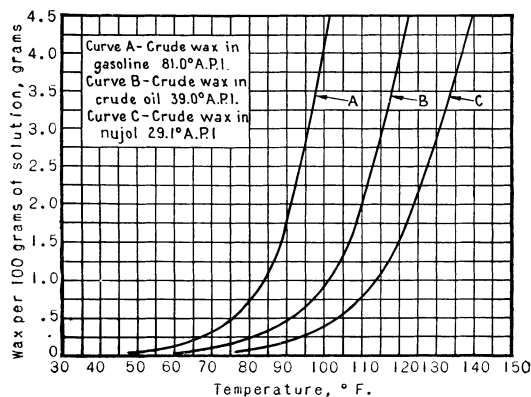


FIGURE 1.—Solubility of paraffin in crude oil—39° A. P. I.; gasoline 81° A. P. I., and Nujol 29.1° A. P. I.

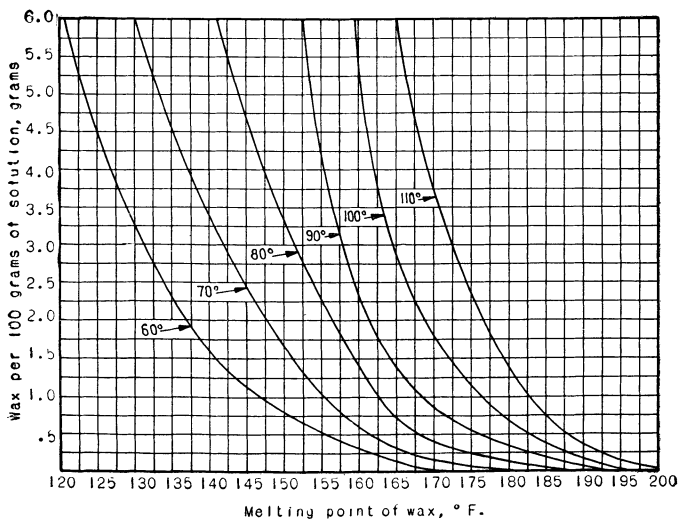


FIGURE 2.—Solubility of waxes of different melting points made from paraffin in crude oil, 39.0° A. P. I.

during its production and transportation. Furthermore, the solubility of different waxes in a given solvent, at a definite temperature, decreases as the molecular weight and melting point increase.<sup>8</sup> Curves showing the increase in solubility of crude wax, or paraffin,

<sup>8</sup> Sullivan, F. W., jr., McGill, W. J., and French, A., Solubility of Paraffin Wax in Oil: Ind. and Eng. Chem., vol. 19, No. 9, November, 1927, pp. 1042-1045. Weber, Paul, and Dunlap, H. T., Solubility of Paraffin Wax in Pure Hydrocarbons: Ind. and Eng. Chem., vol. 20, No. 4, 1928, p. 384.

in crude oil and other solvents and the solubility of waxes of different melting point, obtained from the rod waxes as determined in the laboratory, are given in Figures 1 and 2. Apparently the temperature of the oil is an important factor in the deposition of wax, and the data presented by the curves emphasize the low solubility of the waxes of higher melting point at temperatures often encountered in the production of crude oil. The curves also show that cooling of the oil-wax solution rapidly decreases the solubility of wax until low concentrations of wax are reached, where a further decrease in temperature has only a very slight effect on the amount of wax that can be held in solution.

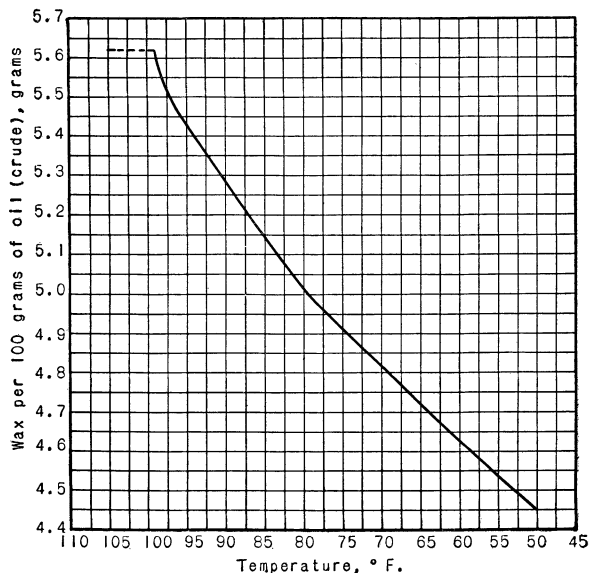


FIGURE 3.—Effect of temperature on natural wax content of 39.0° A. P. I. Salt Creek crude oil

The effect of cooling on the wax content of Salt Creek crude as taken from the well is shown by Figure 3. The sample of oil used contained 5.6 per cent of wax by weight, which is somewhat higher than the average for the field. The laboratory method used shows that the average Salt Creek crude contains approximately 5 per cent of wax. The difference in wax content of the sample and the average of the field was due to the fact that the sample of oil was taken from the well through a line containing some loosely deposited paraffin which probably washed into the sample. The total wax content of the sample was made up not only of the waxes of higher melting point, found in the deposited paraffin, but also of waxes of lower melting point. The solubility of the crude wax in wax-free crude of the same gravity agrees closely with the amount of wax that separates from the natural crude. For example, according to the determinations made in the laboratory 0.26 part of wax per 100 parts of oil by weight settled out of the natural crude (fig. 3) when it was cooled from 89° to 79° F., and 0.25 part of wax per 100 parts of oil by weight were dissolved in wax-free crude oil when it was heated from 79° to 89° F. (fig. 1).

Although the amount of wax that settles out of crude oil when the temperature is decreased may seem insignificant, it is surprisingly large even in a small well. For example, assume a 30-barrel well in the Salt Creek field which produces oil containing 5 per cent of wax, and assume that the oil is chilled to a temperature of 70° F., which is not unusual, as can be seen from Tables 5 and 6 (p. 31). Figure 3 indicates that the oil can hold only 4.82 per cent of wax at 70° F.; therefore, 0.18 per cent of wax (16.5 pounds of wax per day) will separate from the oil. If only half of this wax accumulated in the well, as usual with 30 per cent of oil, it would amount to approximately 1.6 gallons a day. In 20 days enough paraffin would deposit to fill completely 176 feet of 2-inch tubing without allowing for the volume taken up by sucker rods.

#### EFFECT OF ASPHALTIC MATERIALS, GUMS, AND RESINS

Wax crystallizes out as small particles when the temperature of crude oil is lowered enough to cause some of the wax to separate from solution. Apparently, the rate of cooling has little effect on the size of the particles, and other substances present in the crude oil exert an influence which prevents the growth of the wax crystals. If the oil is agitated or if it is not cooled sufficiently to throw out enough wax to become solid, the small wax crystals agglomerate into particles or grains about the size of table salt. Figure 4, *A*, shows the nature of wax crystals when first precipitated from crude oil, and Figure 4, *B*, shows the crystals after they have coagulated to form larger grains.

If crude oil is cooled without agitation, the small wax crystals form an interlocking network that supports the liquid constituents,<sup>9</sup> and a temperature is finally reached at which the oil will not flow when tilted to a horizontal position in a bottle. This temperature is called the "pourpoint." If the oil is agitated during the cooling or after the pourpoint is reached, it becomes liquid again but very viscous, due to the presence of the suspended wax crystals. The effect of temperature on the viscosity of several paraffin and mixed-base oils is shown in Figure 35, page 99. At approximately the temperature at which the wax starts to settle out the oil increases sharply in viscosity, when viscosity is determined by the Saybolt viscosimeter, while oils that do not contain wax increase gradually in viscosity. The writer and others believe that the viscosity, as indicated by the Saybolt instrument, is not satisfactory for crude oils containing wax crystals, as this instrument does not give a true measurement of the viscosity when suspended wax is present in the oil and gives misleading values when used for pipe-line calculations. It is suggested that a MacMichael viscosimeter or some form of mobilometer would give better values for this type of calculation than the Saybolt viscosimeter.

Heating crude oils to a temperature well above that at which all of the wax, asphaltic material, gums, and resins are completely

<sup>9</sup> Gurvich, L., The Problem of Hardening of Products Containing Paraffin: *Neftjanoe i slancevoe Chozhajstvo*, vol. 7, 1924, pp. 350-351.

soluble in the oil will appreciably lower the pourpoint.<sup>10</sup> An excellent discussion of this subject is given by Vietti.<sup>11</sup> The results obtained by Vietti show that the pourpoint of Panhandle crude can be lowered from 60° to 0° F. by heating the oil to 115° F., or above, and that the oil slowly returns to its original pourpoint on standing. Vietti also discusses the use of various chemicals offered to the industry for lowering the pourpoint and concludes that they are not satisfactory for that purpose. In studying the results obtained by heating oils the writer observed that oils from which the asphaltic materials, gums, and resins were removed did not respond to such heating and that the pourpoint was not affected.<sup>12</sup> It was noted, however, that the pourpoint of Lance Creek, Wyo., Wheat Basin, Mont., and other crude oils, which do not contain an appreciable quantity of asphaltic materials, were not affected by heating. The conclusion was therefore obvious that the asphaltic materials and gums when in solution act as a retarding agent to the crystallization of the wax and that they separate slowly from solution in the oil when cooled.

To substantiate these conclusions further, the action of the asphaltic materials, gums, and resins on the crystalline nature of wax was studied under a microscope. Figure 4, *A*, and 4, *B*, shows the crystalline structure of the wax as it normally separates from crude oil; and Figure 5, *A*, shows the crystalline structure of the wax that separates from the same crude oil after the gums, resins, and asphaltic material had been removed by fuller's earth. Figure 6, *A*, shows a pure wax fraction prepared from crude rod wax by removing the gums, resins, and asphaltic material with fuller's earth and removing the oil by recrystallization from acetone and ethylene dichloride. Figure 6, *B*, shows the same wax with a small portion of the material removed by the fuller's earth added back to the pure wax. The samples for Figure 6, *A*, and 6, *B*, were prepared in an identical manner and are of the same magnification ( $\times 50$ ).

#### EFFECT OF EVAPORATION OF VOLATILE CONSTITUENTS

There is a continuous loss of volatile liquid constituents from crude oil after it enters the well and even while it is in the producing sand. Loss of the lighter constituents of the oil reduces the quantity of wax that the oil can hold in solution at a definite temperature (1) by reducing the volume of the oil, resulting in less solvent being available to dissolve the same quantity of wax, and (2) by decreasing the solubility of the wax in the oil, because wax is more soluble in the lighter constituents of crude oil than in the heavier ones.<sup>13</sup> Figure 7 shows curves of the solubility of crude wax

<sup>10</sup> Gurvich, L., Properties of Paraffin-Containing Petroleum Products: *Petrol. Ztschr.*, vol. 19, 1923, pp. 183-185. Tychinin, B. G., Pavlova, S. N., Gershon, G. I., and Malinina, V. S., Structure and Properties of the Paraffin-Containing Petroleum and Mazout of Grosny: *Nefljanoe i slancevoe Khoziastvo*, vol. 6, 1924, pp. 838-854.

<sup>11</sup> Vietti, W. V., and Oberlin, W. A., Problems Encountered in Handling Panhandle Crude: *Petrol. Devel. and Technol.*, *Am. Inst. Min. and Met. Eng.*, 1927, pp. 269-284.

<sup>12</sup> Chernoshukov, N., Adsorption of Resins by Paraffin Wax and Solidification of Paraffin-Containing Products: *Nefljanoe i slancevoe Khoziastvo*, vol. 8, 1925, pp. 640-646.

<sup>13</sup> Weber, Paul, and Dunlap, H. T., Solubility of Paraffin in Pure Hydrocarbons: *Ind. and Eng. Chem.*, vol. 20, No. 4, 1928, pp. 383-384.



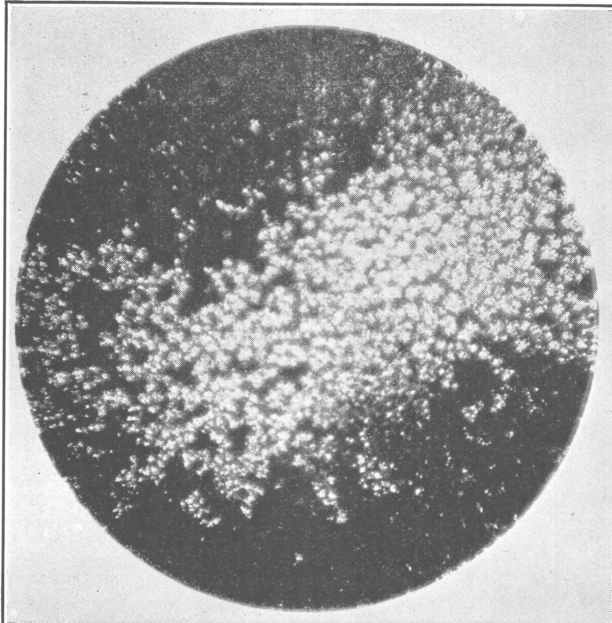
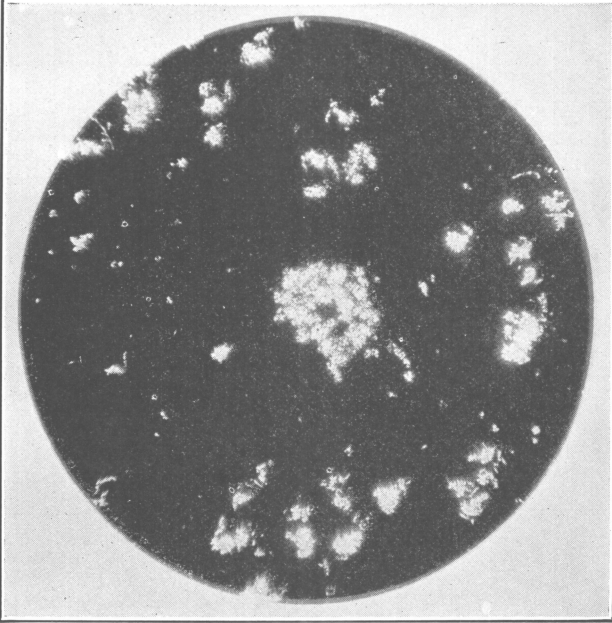
*A**B*

FIGURE 4.—*A*, Natural wax freshly precipitated from crude oil; *B*, natural wax crystals coagulated after standing in crude oil. Magnification,  $\times 50$

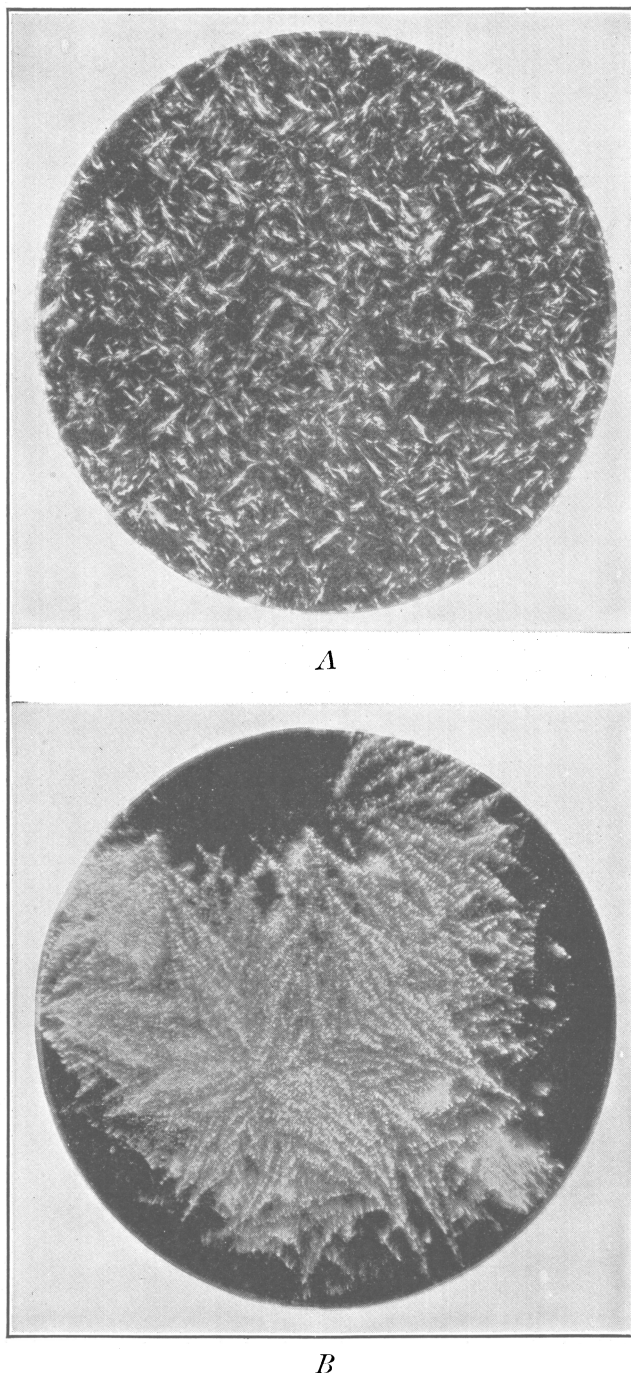


FIGURE 5.—*A*, Natural wax crystals precipitated from crude oil after treatment with fuller's earth; *B*, crystalline nature of rod wax from Salt Creek, Wyo., crude. Magnification,  $\times 50$

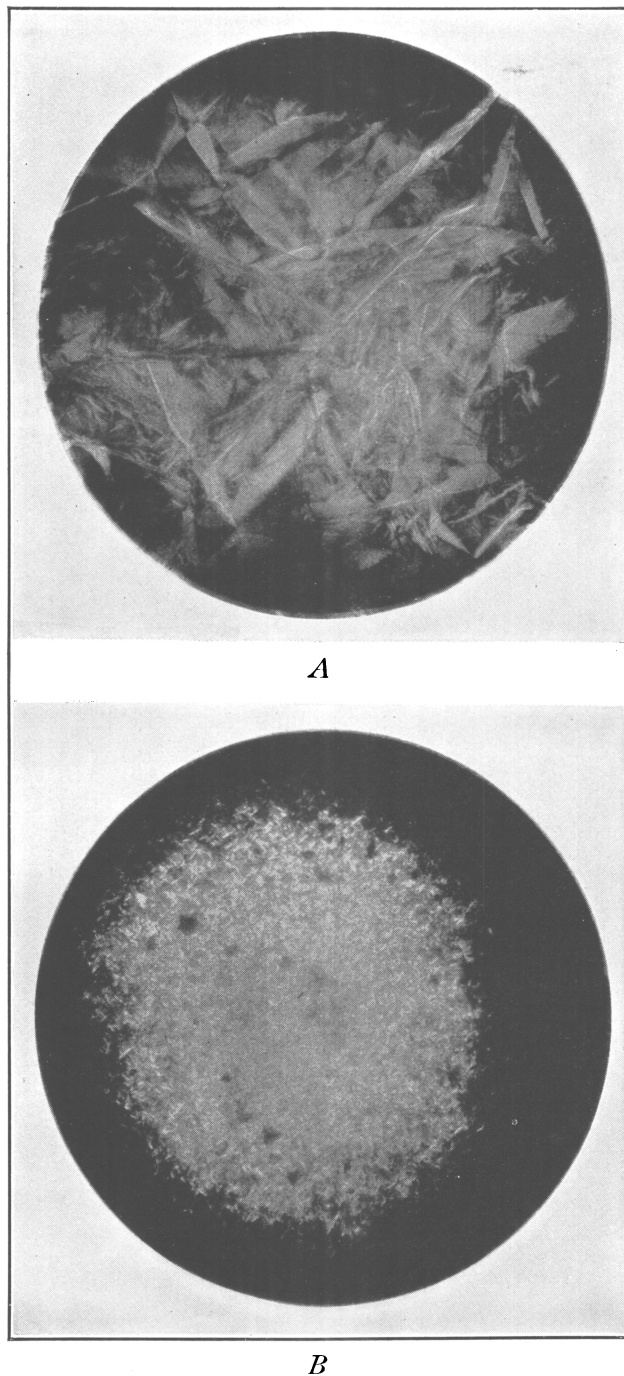


FIGURE 6.—*A*, Pure wax fraction from rod wax after treating with fuller's earth; *B*, same wax fraction with material removed by fuller's earth present. Magnification,  $\times 50$ .

in various fractions of crude oil at 100° and 90° F.; and the solubility of crude wax in gasoline (81° A. P. I.), in crude oil (39° A. P. I.), and in Nujol (29° A. P. I.) at different temperatures is shown in Figure 1 (p. 8). The solubility of wax differs noticeably as the fractions change from the lighter gasoline fractions, which probably con-

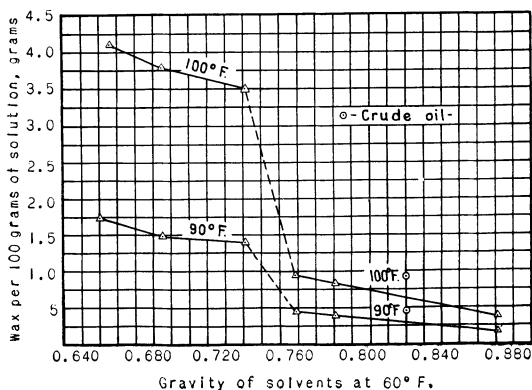


FIGURE 7.—Solubility of crude rod wax in various fractions of crude oil

sist of straight-chain saturated-paraffin hydrocarbons, to the heavier hydrocarbons, which are undoubtedly of saturated monocyclic and polycyclic series.<sup>14</sup> Although the wax in the crude paraffin increases in solubility as the fractions become lighter, the asphaltenes become less soluble in the lighter fractions of crude oil and are prac-

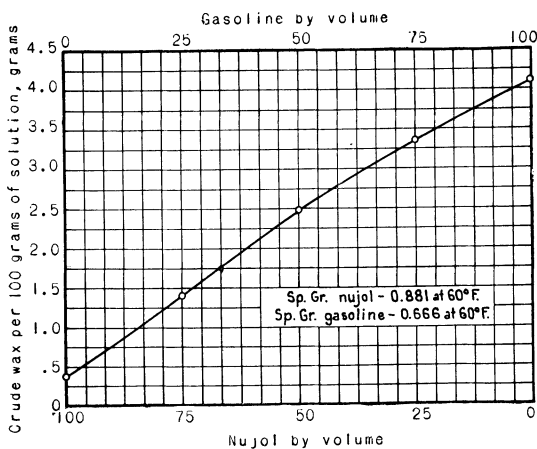


FIGURE 8.—Solubility of crude wax in mixtures of light and heavy fractions of crude oil at 100° F.

tically insoluble in fractions lighter than hexane. The solubility of wax in mixtures of two different fractions of crude oils is approximately the same as in either solvent alone and is proportional to the quantity of each solvent present. The solubility of wax in different mixtures of gasoline and Nujol is shown in Figure 8.

<sup>14</sup> Brooks, B. T., *Chemistry of the Nonbenzenoid Hydrocarbons*: Chem. Catalog Co., New York, 1922, pp. 13-51.

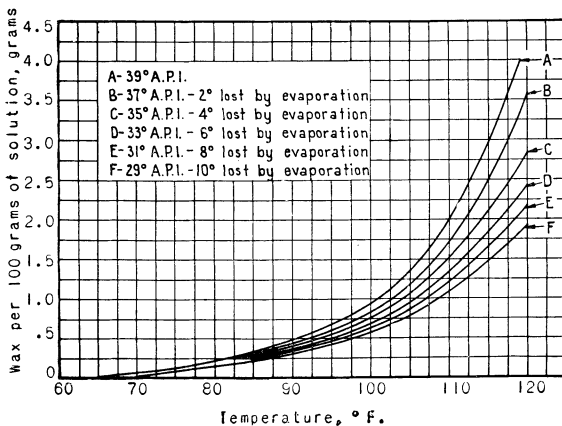


FIGURE 9.—Effect of evaporation and loss of volatile constituents on solubility of crude rod wax in crude oil

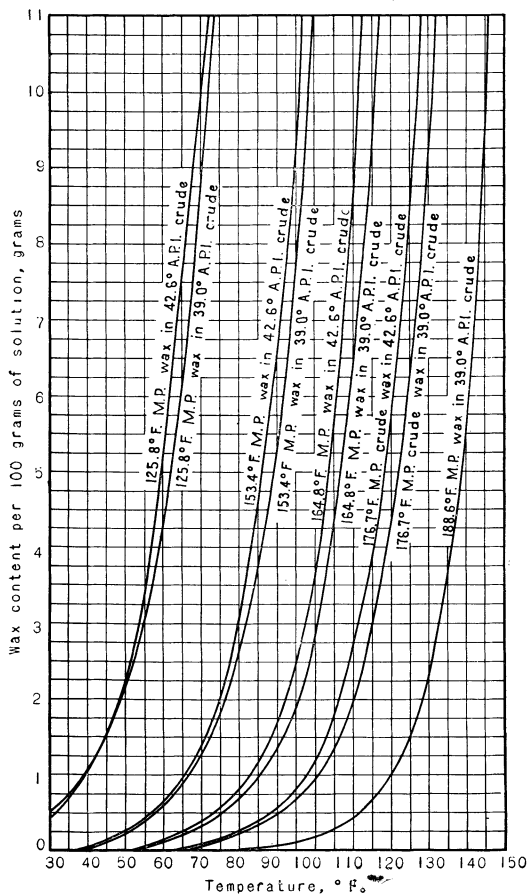


FIGURE 10.—Solubility of wax in 42.6° A. P. I. and 39.0° A. P. I. wax-free crude oil

The rate of decrease in the solubility of wax in crude oil due to evaporation of the volatile constituents is shown in Figures 9 and 10; besides decreasing the solubility of the wax in the oil with increasing loss by evaporation, the quantity of solvent for the same amount of wax is decreased. Using the formula

$$X = \frac{d_2 - d_1}{d_2 - d_3},$$

given by Wiggins,<sup>15</sup> the decrease in volume can be calculated approximately.  $X$  is the proportion of the original volume lost by evaporation,  $d_1$  the original specific gravity,  $d_2$  the specific gravity after evaporation has taken place, and  $d_3$  the specific gravity of the portion lost through evaporation. If the specific gravity of the portion lost by evaporation is assumed to be 0.666 and the volume of the original 39° A. P. I. oil remaining after a loss by evaporation of 2° A. P. I. is calculated, it is found that 5.7 per cent by volume is lost by evaporation or, in other words, that 94.3 per cent of the oil remains as a solvent for the wax. To obtain the percentage by weight it is necessary to use the formula:

$$\frac{d_2 \times \text{percentage of original volume}}{d_1} = \text{percentage of original weight.}$$

It therefore follows that a barrel of 39° A. P. I. oil that has lost 2° A. P. I. in gravity by evaporation can only dissolve 95.3 per cent as much wax by weight, as shown in Figure 9, because in addition to being a poorer solvent only 95.3 per cent of a barrel of oil by weight remains to act as a solvent. A 4° loss in A. P. I. gravity would result in the original volume being reduced 10.9 per cent, leaving 91.3 per cent by weight to act as a solvent. Therefore, only 91.3 per cent of the wax shown by curve *C*, Figure 9, could be dissolved by a barrel of 39° A. P. I. oil after it has lost 4° in A. P. I. gravity.

The loss of volatile constituents is a factor in the separation of wax from solution in crude oil; and, although not as important as temperature, under certain conditions it may be a major factor in the deposition of paraffin, especially in fields where gas-drive or pressure-restoration work is being undertaken. If a considerable portion of the gas by-passes and flows through the sand, it will remove a large quantity of the volatile constituents from the oil remaining in the sand, which would result in paraffin crystallizing from the remaining oil.

#### EFFECT OF PRESSURE, GAS, AND AIR IN SOLUTION

When pressure is applied to a solution of a solid in a liquid it will tend to decrease in solubility when the system obeys, or deviates in a positive direction from, Raoult's law.<sup>16</sup> Solutions of wax in oil are of this type as they deviate from Raoult's law in a positive direction. Pressure therefore decreases the solubility of the wax in oil.

<sup>15</sup> Wiggins, J. H., Evaporation Loss of Petroleum in the Mid-Centinent Field: Bull. 200, Bureau of Mines, 1922, p. 15.

<sup>16</sup> Hildebrand, Joel H., Solubility: Am. Chem. Soc. Monograph, Chem. Catalog Co., New York, 1924, pp. 160-163.

Laboratory experiments indicated, however, that the change in solubility even at a pressure of 1,000 pounds per square inch is small and can be disregarded as a factor affecting the solubility of paraffin in crude oil. Brooks<sup>17</sup> states that a compressed gas has a noticeable solvent power for a solid only when the gas compressed to the liquid state is of such a character as to dissolve the solid to a marked degree, because at the critical point the physical properties of gas and liquid become identical, and the solubility curves of gas and liquid should merge smoothly into each other. Paraffin should therefore show some solubility in natural gas but practically none in air.

To determine the solubility of paraffin in dry natural gas a number of determinations were made with gas at a pressure of 1,000 pounds per square inch. The composition of the gas was as follows:

	Per cent		Per cent
Carbon dioxide-----	0.0	Propane-----	1.55
Nitrogen-----	3.24	Butane-----	.81
Oxygen-----	.1	Pentane and higher-----	.1
Methane-----	87.4		
Ethane-----	6.8		100.00

Paraffin was found to be practically insoluble in dry natural gas at pressures up to 1,000 pounds per square inch. Paraffin was also found to be insoluble in air under any pressure up to 1,000 pounds per square inch. A summary of the results obtained from experiments made to determine the effect of dissolved air and dry natural gas on the solubility of paraffin in oil is given in Table 3. Apparently, the dissolved gas and air act as if they were present in the system as liquids and tend to change the solvent power of the oil for paraffin. When air was present in the various solvents the solubility of the wax decreased. The results show further that paraffin is insoluble or much less soluble in air than in any of the solvents used. Natural gas, however, apparently can dissolve paraffin to some extent when dissolved in oil. In the solubility experiments performed dissolved dry natural gas decreased the solubility of paraffin in the lighter fractions in which paraffin was more soluble than 2.8 to 2.9 grams per 100 grams of solvent, whereas it increased the solubility of paraffin in heavier solvents in which paraffin was less soluble than 2.8 to 2.9 grams per 100 grams of solvent. In lighter crudes, containing a high percentage of the fractions in which paraffin is readily soluble, dissolved gas slightly decreases the solubility of paraffin, whereas in the heavy crudes dissolved gas might increase the solubility of paraffin. Furthermore, the increase or decrease in solubility due to dissolved gas was directly proportional to the pressure at which the gas was held in solution—that is, to the quantity of gas in the solution. The decrease or increase in solubility of paraffin obtained by using dry natural gas and air, however, was so small in comparison to the effect of temperature, or loss in volatile constituents, that it is questionable if gas in solution plays more than a minor part in the separation or deposition of paraffin.

<sup>17</sup> Brooks, B. T., *Chemistry of the Nonbenzenoid Hydrocarbons*: Chem. Catalog Co., New York, 1922, pp. 13-51.

TABLE 3.—*Solubility of crude rod wax in various fractions of crude oil with and without gas and air in solution*

Solvent	Specific gravity of solvent at 100° F.	Grams of rod wax dissolved by—					Effect on solubility
		100 grams of solvent	100 grams of solution of solvent and gas at 1,000 pounds pressure	100 grams of solvent saturated with gas at 1,000 pounds pressure	100 grams of solution of solvent and air at 1,000 pounds pressure	100 grams of solvent saturated with air at 1,000 pounds pressure	
Gasoline fraction, boiling point, 73° to 84° F.	0.608	3.95	2.40	2.92	2.89	3.16	Decreased by gas. Decreased by air.
Gasoline fraction, boiling point, 139° to 149° F.	.675	4.71	3.72	4.27	3.79	3.92	Do.
Gasoline fraction, boiling point, 194° to 232° F.	.723	4.66	4.10	4.53	3.91	4.01	Do.
Kerosene fraction, boiling point, 350° to 400° F.	.785	2.84	2.85	3.04	2.45	2.49	Slightly increased by gas. Decreased by air.
Steam-stilled fraction.....	.806	2.41	2.63	2.79	2.26	2.29	Increased by gas. Decreased by air.
Nujol.....	.867	1.34	1.41	1.46	1.22	1.23	Do.
		Grams of natural paraffin dissolved in—					
		100 grams of crude oil	100 grams of crude oil and dissolved gas at 1,000 pounds pressure	100 grams of crude oil saturated with gas at 1,000 pounds pressure	100 grams of crude oil and dissolved air at 1,000 pounds pressure	100 grams of crude oil saturated with air at 1,000 pounds pressure	
Crude oil.....		5.6	5.43	5.74	5.40	5.45	Increased by gas. Decreased by air.

## EFFECT OF WATER

A number of solubility determinations with and without water in the oil-wax system showed conclusively that water does not increase or decrease the solubility of wax in oil. One would expect this because water is practically insoluble in oil and wax. Water, however, does enter as a factor in the deposition of wax by affecting other conditions. For example, in the production of oil from wells where considerable trouble is encountered with the deposition of paraffin it is not uncommon for the problem to be practically eliminated when the well starts to make an appreciable quantity of water, due to the fact that water has about twice the specific heat of oil and therefore contains heat enough to prevent cooling of the water and oil to a point where an appreciable quantity of wax will separate from solution. Water also furnishes an additional volume of liquid and increases the velocity of the oil flowing through the pipe, so that there is less opportunity for the paraffin to become attached to it. In some wells water emulsifies with the oil and the emulsion settles out with the wax, thus increasing its volume, but this fact has minor importance in the elimination of troubles due to paraffin.



**EFFECT OF FINE SAND AND SILT**

The amount of wax that separates from solution in the crude oil due to various conditions is in no way affected by the presence of fine sand and silt. These substances, however, often act mechanically to increase greatly the trouble from deposition of paraffin. The fine particles of sand and silt act as a nucleus for the cohesion of the small wax grains suspended in the oil into larger grains which separate more readily from the oil. The fine particles also accumulate with the paraffin and greatly increase its bulk. In some wells the paraffin consists of 50 to 75 per cent of clay, fine sand, or silt.

**CONDITIONS FAVORING ACCUMULATION OF PARAFFIN AFTER SEPARATION FROM SOLUTION IN OIL**

Although wax may separate from solution in the oil, due to certain conditions, it will not necessarily be deposited on the walls of the sand, pipe, rods, and other objects. In fact it would be logical for the wax to remain suspended in the oil and be carried along with it, because it has a specific gravity only slightly greater than that of the oil. This ideal condition often exists, and in some wells little or no paraffin trouble is experienced. In the laboratory and in the field certain other conditions caused or were responsible for the accumulation of paraffin after it separated from solution in the oil.

Assuming, of course, that the oil contains free wax crystals that have separated from solution due to causes previously discussed, the following conditions are chiefly responsible for its accumulation. The most important factor causing precipitated paraffin to accumulate on a surface is alternate coating and draining of the surface by the oil.<sup>18</sup> When a surface is intermittently coated with oil the film of oil left on the surface drains away until it is too thin and its movement too slow to carry the soft gummy wax particles in suspension. These particles become firmly attached while the oil continues to drain from the surface. If the surface is cooler than the saturation point of the oil additional wax separates from the oil film. When the surface is much cooler than the oil a thicker film of oil remains after each coating because the oil is more viscous. As a result a greater number of wax particles are deposited. The film of oil also exposes a large surface to evaporation, and the lighter constituents evaporate, concentrating the solution of wax in the remaining oil and causing the deposition of additional quantities of wax.

When a surface is continually in contact with the oil, at approximately the same temperature, the formation of a film and the draining of the oil away from the wax particles are prevented; also, there is less tendency for the wax particles to become attached to the surface. For example, in a pipe or container where the oil is not in motion the wax crystals remain suspended in the oil or settle slowly to the bottom, but they do not become attached to the surfaces with which they come in contact because of the oil that surrounds them.

In certain flowing-oil wells where excessive quantities of gas are produced the oil occurs as a mist or as small droplets of oil suspended

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<sup>18</sup> Mills, R. van A., *The Paraffin Problem in Oil Wells: Rept. of Investigations 2550, Bureau of Mines, 1923, 11 pp.*

in the gas. In such wells the pipe is coated with a film of oil only; and, if the film contains wax crystals, they usually become firmly attached to the pipe.

If the pipe or surface is much cooler than the oil the wax in the oil in contact with the surface is precipitated from solution and becomes attached to the surface. Where oil is in motion, therefore, wax becomes attached to the walls of the pipe under certain conditions, and the quantity deposited is apparently governed by the velocity of the oil. The precipitation on a cold surface of wax from oil in motion generally occurs where the movement of oil in pipes is not turbulent but is more nearly a streamline flow and where the temperature of the pipe is materially less than that of the oil. Under conditions of streamline flow the movement of the oil film adjacent to the pipe is relatively slow and is such that the precipitated wax has an opportunity to become attached firmly to the pipe. When the oil approaches or is in turbulent flow, however, the motion of the oil in contact with the pipe is rapid, and wax seldom becomes attached to the pipe.

The small wax crystals that are precipitated from solution in the oil exhibit an attraction for each other and tend to join and form larger granular particles, as shown by the photomicrographs in Figure 4, *A*, and 4, *B* (p. 12). The growth of the granular particles is hastened by agitating the oil, and the size and quantity of the particles depend upon the number of wax crystals present and the time of agitation. The larger particles of wax are deposited more easily on a surface when it is alternately coated with oil. The large granular particles of wax also form in storage tanks, if the oil is not so viscous as to prevent settling of the wax particles.

#### **CONDITIONS IN WELL AND SAND WHICH MAY CAUSE SEPARATION OF PARAFFIN FROM SOLUTION IN OIL**

Laboratory studies of the factors governing the solubility of wax in crude oil indicate that the two most important conditions are the temperature of the oil and the percentage of loss of the volatile constituents. It is therefore essential to consider how these conditions may be altered in the sand and well to prevent the separation of wax from solution in the oil.

Oil in its movement through the sand to the top of the well is subjected to cooling and evaporation due to several causes which vary to a certain extent with the underground equipment, method of completing the well, character of the sand reservoir, and manner in which the oil is produced. The most important conditions affecting the separation of paraffin from the crude oil are the following: (1) The cooling produced by the gas in expanding through an orifice or restriction; (2) cooling produced as a result of the gas expanding, forcing the oil through the sand to the well and lifting it to the surface; (3) cooling produced by radiation of heat from the oil and gas to the surrounding formations as it flows from the bottom of the well to the surface; (4) cooling produced by dissolved gas being liberated from solution; (5) change in temperature produced by intrusion of water; and (6) loss in volume and change in temperature due to the evaporation or vaporization of the lighter constituents.

**COOLING PRODUCED BY EXPANSION OF GAS THROUGH ORIFICE OR RESTRICTION**

Theoretically, if a perfect gas expands without doing work there is no change in temperature. The expansion of a gas that deviates from a perfect gas and expands without doing work, however, is accompanied by a slight cooling due to the so-called Joule-Thomson effect. The amount of cooling, however, is so small that it usually can be disregarded. An expanding gas must do work to cause a decrease in temperature. One form of work that the gas may do is in the sand or well and that produces a certain amount of cooling is due to the gas expanding through an orifice or restriction in which a portion of the thermal energy of the gas is consumed in accelerating and increasing the velocity or the density of the gas or both. The decrease in temperature or cooling, however, is localized to the point where the expansion takes place, and the actual loss in thermal energy (B. t. u.) is small, because it is concentrated at one place; however, very low temperatures may exist at that point. If the velocity of the gas decreases after passing the orifice or restriction, its temperature rises again, due to the conversion of the kinetic energy of the gas into thermal energy.

The pore spaces on the face of the sand in the well act as orifices, and the expansion of the gas as it leaves the sand and enters the well may be sufficient (when a high differential exists between the pressure in the sand and the pressure in the well) to cause appreciable cooling at the face of the sand, resulting in the separation of paraffin from solution in the oil. When the well contains enough liquid to cover the face of the sand the gas probably expands through the sand pores and produces cooling at that point; however, the velocity of the gas is decreased rapidly as it passes through the oil, thus converting the kinetic energy of the gas into thermal energy. This conversion of energy heats the oil, transfers the heat back to the face of the sand, and prevents low temperatures at the face of the sand.

In the oil string of a normal flowing well the gas does not expand rapidly at one point, and no cooling of this nature should take place. In gas-lift wells where the gas is injected into the oil string through foot pieces or small openings, or where there are restrictions in the pipe through which the gas rapidly expands, cooling may take place. When oil that contains a high percentage of wax is produced by the gas-lift method it is not advisable to have jets or restrictions in the oil string that will cause cooling of the gas, because paraffin will often accumulate where gas expansion occurs.

**COOLING PRODUCED BY EXPANSION OF GAS WHILE FORCING OIL THROUGH SAND AND LIFTING IT TO SURFACE**

When oil is flowed through the sand and from the well by the natural gas associated with it or raised to the surface by the gas lift the gas expands and supplies the energy to move the oil. The expansion of the gas consumes some of the thermal energy of the gas and lowers the temperature of the oil. This conversion or use of thermal energy supplied by the gas and oil varies with the amount of work done by the gas in flowing the oil. There are three general forms of expansion of gas in which it performs work—adiabatic,

polytropic or exponential, and isothermal. The expansion in the sand or well is some form of polytropic or exponential expansion expressed by the equation:

$$W = \frac{P_1 V_1 \times 144}{s-1} \times \left[ 1 - \left( \frac{P_2}{P_1} \right)^{\frac{s-1}{s}} \right].$$

Expressing the expansion as a function of temperature, the following equation holds true:

$$T_2 = T_1 \left( \frac{P_2}{P_1} \right)^{\frac{s-1}{s}},$$

where

$W$  = work done, foot-pounds,

$s$  = exponent of expansion (ratio of the specific heats of the gas),

$P_1$  = initial pressure at bottom of well, pounds per square inch absolute,

$P_2$  = pressure at top of well, pounds per square inch absolute,

$T_1$  = temperature at bottom of well, °F. absolute, and

$T_2$  = temperature at top of well, °F. absolute.

The exponent  $s$  varies with the type of expansion from 1.0 for isothermal expansion to  $K$  (the ratio of the specific heat of the gas at constant pressure to specific heat of the gas at constant volume) for adiabatic expansion;  $K$  for natural gas ranges from 1.2 to 1.3. The type of gas expansion in the sand and in the well is governed by the pressure drop, temperature of the oil and gas, ratio of oil to gas produced, radiation of heat through the formations, and evaporation of the oil. The more heat supplied by the oil and by radiation from the formation the more nearly the resulting expansion of the gas will approach isothermal.

#### COOLING PRODUCED IN SAND

At a distance from the well the pressure gradient in the sand decreases very slowly, but close to the well a very sudden decrease in pressure occurs and the velocity of the gas and oil is high. Uren<sup>19</sup> gives a formula for calculating the theoretical pressure drop in the sand at different distances from the well; and Figure 11, which was taken from his report, illustrates the pressure conditions surrounding a producing well. Regardless of the accuracy of the formula or the constants used in deriving this curve the fundamental pressure conditions within a sand reservoir undoubtedly approach those shown in the curve, and obviously a large percentage of the pressure drop between the normal formation pressure and the pressure at the bottom of the well occurs within a short distance from the well. The pores in the sand adjacent to the well where the most rapid decline in pressure occurs undoubtedly act as orifices through which the rapid expansion of the gas occurs. The expansion of gas and the transformation of thermal energy into kinetic energy as a result of expanding through these orifices is accompanied by an additional change of thermal energy into kinetic energy by the gas expanding in some form of polytropic expansion and accelerating and forcing the oil and gas through the relatively short distance in the sand. This expansion undoubtedly results in cooling

<sup>19</sup> Uren, L. C., Increasing Production of Petroleum by Increasing Diameter of Wells: Trans. Am. Inst. Min. and Met. Eng., 1364G, 1924, pp. 2-5.

of the oil and sand adjacent to the well. The larger the pressure drop and the greater the amount of work required to force the oil and gas into the well the greater the amount of cooling. Under certain conditions the radiation of heat through the sand and the heat carried by the oil will probably be sufficient to cause expansion of the gas approaching isothermal conditions and prevent the cooling produced by the gas in expanding and forcing oil through the reservoir sands. Under certain conditions low temperatures are known to exist in the sand adjacent to the well. H. R. Pierce, consulting engineer with Dunn-Lewis, observed that in certain gas wells producing some water, by gradually increasing the amount of gas

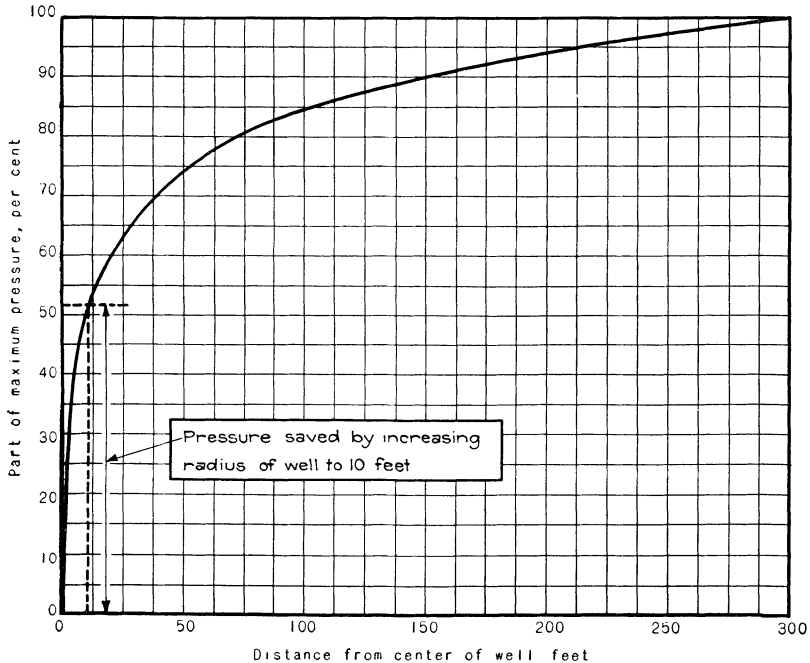


FIGURE 11.—Theoretical pressure gradient in sand, after L. C. Uren

taken from these wells by enlarging the orifices at the wellheads and decreasing the back pressure on the wells, a condition was reached where a further reduction in back pressure resulted in a decrease in the amount of gas delivered from the wells. When the size of orifices was then decreased and the back pressure increased the production of gas slowly increased to the original quantity for that particular orifice, indicating, possibly, that a flow condition was established in the sand which produced sufficient cooling to freeze the water accompanying the gas and plug the sand.

It is impossible to determine the exact conditions that will produce sufficient cooling to lower the temperature in the sand and congeal the oil or cause the precipitation of paraffin from the oil, but such results undoubtedly will be produced under certain conditions. In most wells the form of expansion and cooling can be modified by increasing the back pressure on the well, which in turn will decrease the differential pressure between the sand and well.

## COOLING PRODUCED IN WELL

When oil and gas flow from the bottom of the well to the surface the oil is cooled as a result of the gas expanding in some form of polytropic expansion and doing the work of flowing the oil and gas to the surface. The form of polytropic expansion occurring in the well depends upon the ratio of oil to gas, the temperature and pressure at the bottom of the well, and the loss of heat due to radiation in the surrounding formations and evaporation. The type of polytropic expansion, however, can be changed and can be made more nearly to approach isothermal expansion by decreasing the ratio of gas to oil, because the heat or thermal energy available (supplied chiefly by the oil) per cubic foot of gas will be greater when the quantity of gas produced per barrel of oil is reduced.

The temperature and pressure of the oil and gas as it reaches the surface with any definite type of expansion will depend upon the amount of energy consumed by friction and other factors within the well. By the use of Table 4 (p. 26), which was calculated from the equation

$$W = \frac{P_1 V_1 \times 144}{s-1} \times \left[ 1 - \left( \frac{P_2}{P_1} \right)^{\frac{s-1}{s}} \right]$$

and Figure 10, taken from Nowels's report,<sup>20</sup> which was calculated from the formula

$$T_2 = T_1 \left( \frac{P_2}{P_1} \right)^{\frac{s-1}{s}},$$

a more definite idea of the amount of energy consumed can be obtained. Assume a well with a pressure of 600 pounds per square inch and a temperature of 100° F. at the bottom, producing a constant volume of oil and gas. The available thermal energy and the loss of energy due to radiation and evaporation definitely establish the type of polytropic expansion. Assume, for illustration, that in this particular well the thermal energy is sufficient to cause a polytropic expansion in which exponent  $s$  is 1.020. If the work required from each cubic foot of gas, in expanding from a pressure of 600 pounds per square inch, to flow the well is 270,419 foot pounds, it is seen from Table 4 that the gas and oil will reach the top of the well at a pressure of 10 pounds per square inch gage. From Figure 12 ( $s$  equals 1.020) at a pressure of 10 pounds per square inch the temperature at the top will be 65.5° F. If the oil string and other conditions in the well are changed so that the energy per cubic foot of gas required to flow the well is 143,397 foot pounds, then from Table 4 the oil and gas will reach the top at a pressure of 100 pounds per square inch, and from Figure 12 ( $s$  equals 1.020), at a pressure of 100 pounds per square inch, the temperature at the top of the well will be 82° F. The example shows that to minimize the cooling it is advisable to produce a well in such a manner that the least amount of work will be consumed in lifting the oil to the surface. To operate a well so that the least amount of energy will be consumed in flowing the well the proper size oil string must be used.

<sup>20</sup> Nowels, K. B., Thermodynamics of Flowing Wells: Rept. of Investigations, Bureau of Mines, in preparation.

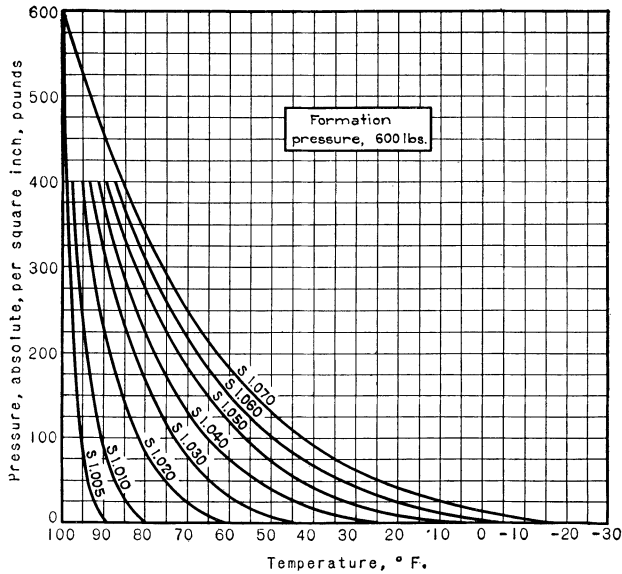


FIGURE 12.—Decrease in temperature of a gas when expanded by different types of polytropic expansion. Calculated by K. B. Nowels

TABLE 4.—Foot-pounds of work per cubic foot of gas—polytropic expansion<sup>1</sup>

Exponent, (S)	Reser-voir pressure, P <sub>1</sub>	Pressure at surface, P <sub>2</sub>								
		0	10	20	50	100	200	300	400	500
1.005	1,500	994,613	884,246	811,831	678,780	556,634	422,274	339,826	280,062	233,821
	1,000	610,769	537,710	488,030	397,438	315,612	225,020	169,455	131,505	99,359
	600	325,742	281,484	251,388	196,507	146,938	92,057	58,421	34,521	15,933
	200	81,620	66,162	55,650	37,100	19,168				
1.010	1,500	978,253	870,940	800,488	670,491	550,308	417,911	336,772	277,880	231,858
	1,000	600,540	527,482	480,724	393,054	312,690	223,559	168,034	129,868	97,818
	600	321,316	277,943	248,732	195,622	146,053	92,000	58,400	34,521	15,817
	200	81,002	65,544	55,341	36,482	17,622				
1.020	1,500	947,500	845,639	778,350	653,805	538,094	409,841	330,883	273,518	228,368
	1,000	581,984	513,162	467,866	383,849	305,530	219,029	165,842	127,121	96,656
	600	312,287	270,419	242,448	181,196	143,397	90,375	57,713	34,079	15,379
	200	79,147	64,152	54,259	35,863	18,751				
1.030	1,500	918,199	821,428	757,302	637,992	526,461	402,207	325,285	269,229	225,024
	1,000	564,585	499,720	456,079	375,228	299,685	215,474	163,310	124,199	95,341
	600	303,908	263,780	236,989	187,360	140,830	89,048	57,004	33,636	15,269
	200	77,395	62,906	53,280	35,286	18,653				
1.040	1,500	890,408	798,417	737,180	622,778	515,246	394,737	319,814	265,012	221,770
	1,000	548,669	486,466	444,780	366,973	293,841	211,869	160,874	123,615	94,172
	600	296,000	257,319	231,649	183,584	138,396	87,808	56,297	33,282	14,910
	200	75,746	61,679	52,342	34,750	18,427				
1.050	1,500	864,005	776,496	717,953	608,197	504,460	387,550	314,524	260,955	218,553
	1,000	533,706	473,886	433,938	358,980	288,113	208,333	158,449	120,278	91,813
	600	288,352	251,264	226,461	179,972	136,015	86,498	55,553	32,875	14,815
	200	74,114	60,504	51,402	34,225	18,179				
1.060	1,500	838,914	755,557	699,610	594,223	494,034	380,577	309,362	257,014	215,463
	1,000	519,031	461,900	423,544	351,362	282,687	204,953	156,150	120,278	91,813
	600	281,026	245,413	221,513	176,517	133,734	85,271	54,865	32,600	14,752
	200	72,567	59,366	50,508	33,735	17,962				
1.070	1,500	815,072	735,583	682,051	580,636	484,063	373,852	304,397	253,202	212,445
	1,000	505,147	450,436	413,657	344,001	277,476	201,683	153,944	117,149	89,569
	600	274,023	239,816	216,765	173,177	131,536	84,091	54,197	32,144	14,605
	200	71,064	58,265	49,643	33,248	17,755				
1.080	1,500	792,337	716,514	665,283	567,922	474,404	367,308	299,583	249,471	209,501
	1,000	491,793	439,446	404,085	336,890	272,397	198,462	151,703	117,149	89,569
	600	267,332	234,437	212,197	169,963	129,389	82,940	53,530	31,788	14,461
	200	69,617	57,204	48,802	32,771	17,537				

<sup>1</sup> Calculated by K. B. Nowels.

McWilliams<sup>21</sup> discusses this subject and shows that there is a optimum velocity of flow at which the least energy is required to flow the oil and gas through the pipe. If the volume of oil and gas is greater or less under the existing pressure conditions than can be carried by the pipe at this optimum velocity of flow, then more energy will be required. The optimum velocity of flow can be maintained or approached by the use of the proper size of pipe. If the size of the oil string is slightly smaller than that required to carry the oil and gas at the optimum velocity, under the existing pressure conditions, back pressure can sometimes be applied at the top of the pipe to decrease the volume of the gas accompanying the oil sufficiently to establish the optimum velocity of flow. McWilliams states that a small back pressure applied at the top of the well under these conditions does not appreciably affect the pressure at the bottom of the well and sometimes actually reduces it. When the diameter of the pipe is larger than that required to carry the oil and gas at the optimum velocity, the size of the pipe must be reduced if efficient operation is to be obtained.

It was stated previously that the smaller the amount of gas produced per barrel of oil with the same pressure and temperature conditions at the bottom of the well, the more nearly the expansion in the well would approach isothermal conditions, because more thermal energy would be available per cubic foot of gas. It can be seen from Table 4 and Figure 10 that the nearer the expansion in the well approaches isothermal expansion the smaller is the temperature drop for the same amount of work. It is more important, therefore, to prevent waste of energy in lifting oil as the expansion approaches the adiabatic condition so as to prevent excessive cooling.

These data are based upon the assumption that the wells are flowing steadily and not by heads. When a well flows by heads the oil is often lifted a number of times and dropped back into the well before sufficient oil accumulates to permit a flow. Each time the oil is lifted it is cooled by the transfer of heat to the gas which is expanding and lifting the oil. When such a condition exists the oil is cooled much more than it would be if a steady flow was maintained.

Cooling due to expansion of gas in doing work takes place only in wells where the oil is lifted by the gas and does not enter into the problem of mechanically pumped wells where the gas is separated from the oil and does no work in lifting the oil.

#### **RADIATION OF HEAT FROM OIL WHILE FLOWING FROM WELL**

The radiation of heat to the surrounding formations is a more or less fixed condition for each well and can not readily be altered by different production methods. The radiation of heat from one substance to another is generally accepted for practical purposes as directly proportional to the temperature difference, which in an oil well is the difference between the temperature of the oil and the surrounding medium. The radiation per B. t. u. per square foot of exposed surface, per hour, per degree temperature difference between pipe and various substances varies appreciably. For example, the

<sup>21</sup> McWilliams, J. R., *Handling Natural Flowing Wells in the Mid-Continent Fields*: Bull. Am. Petrol. Inst., vol. 10, No. 2, January, 1929, pp. 45-55.



rate of radiation from a pipe in contact with water is approximately eight times as great as if it is in contact with air. The rate of radiation of heat from a pipe in contact with dry earth is less than if it is in contact with air. This rate increases rapidly with an increase in the moisture content of the earth, approaching that of water. The radiation of heat to gas or air which is in motion is more rapid than gas or air which is not in motion. The loss of heat or the temperature drop of the oil flowing through a pipe can be calculated approximately, providing certain factors are known, from the following equation derived from the fundamental theory of heat transfer.

$$\log_{10} \frac{t_1 - t_a}{t_x - t_a} = \frac{600 DKX}{WQC},$$

where

- $t_1$  = temperature of oil at sand, °F.,
- $t_a$  = temperature of the surrounding medium (ground), °F.,
- $t_x$  = oil temperature at any distance  $X$ , °F.,
- $X$  = depth of well, miles,
- $D$  = outside diameter of pipe, inches,
- $Q$  = rate of flow, 42-gallon barrels per hour,
- $W$  = weight of liquid, pounds per 42-gallon barrel,
- $C$  = specific heat of liquid, B. t. u. per pound per 4° F., and
- $K$  = coefficient of heat loss, B. t. u. per hour per square foot of outside pipe surface per °F. difference in temperature between liquid and air.

The formula expressed in terms of temperature at the top of the well is as follows:

$$t_x = (t_1 - t_o) 10^{-\left(\frac{600 DKX}{WQC}\right)} + t_o,$$

where

- $t_x$  = temperature of oil and gas at any point  $x$  miles from initial point, °F.,
- $t_o$  = temperature of surrounding earth, °F.,
- $t_1$  = initial temperature of oil or sand temperature, °F.,
- $D$  = outside diameter of pipe, inches,
- $K$  = coefficient of heat transfer, B. t. u. per hour per square foot of pipe surface per °F.,
- $X$  = distance, miles,
- $W$  = weight of one 42-gallon barrel of oil, pounds,
- $Q$  = rate of flow through line, barrels per hour, and
- $C$  = specific heat of oil, B. t. u. per pound per °F.

or,

$$t = t_o + (t_1 - t_o) e^{-Y},$$

where

$$Y = \frac{SKX}{CM},$$

- $t$  = temperature of oil and gas at any point  $x$  feet from initial point, °F.,
- $t_o$  = temperature of air, ground, or surrounding medium, F.,
- $t_1$  = initial temperature, °F.,
- $e$  = base of Napierian logarithm,
- $S$  = circumference of pipe, feet,
- $K$  = coefficient of heat transfer, B. t. u. per hour per square foot of pipe surface per °F.,
- $X$  = distance from initial point, feet,
- $C$  = specific heat of oil, B. t. u. per pound per °F., and
- $M$  = rate of flow through line, pounds per hour.

The accuracy of the assumption of proportionality between heat transfer and temperature difference may perhaps be questioned; however, experience obtained from pumping hot oil in pipe lines is

insufficient to establish any better basis for calculation on account of the variation in some of the "constants" of the formula, especially the coefficient of heat transfer  $K$ . To obtain a more definite idea of the value of  $K$  actual temperature measurements were made in a number of wells in the Salt Creek field, Wyoming.

The apparatus used to make temperature measurements in the well was developed by the electrical department of the Midwest Refining Co. and consisted of a coil of fine copper wire inclosed in an oil-tight tube connected to a cable which could be lowered into the well. Operation of the apparatus was based upon the change in electrical resist-

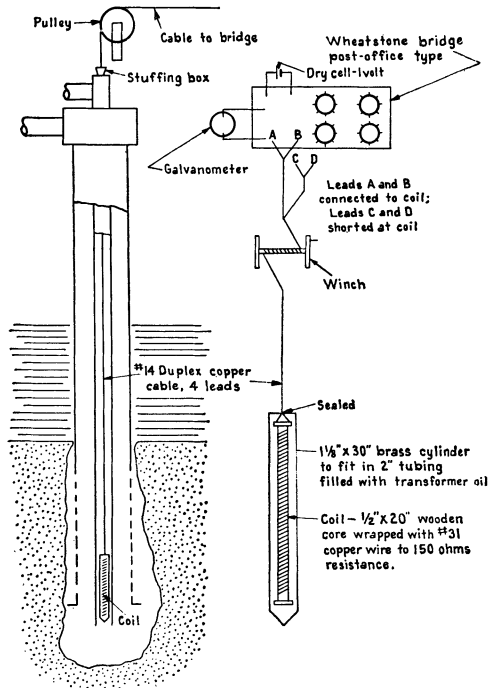


FIGURE 13.—Electrical-resistance pyrometer for measuring well temperatures

ance of the coil due to variations in temperature. The coil was calibrated in the laboratory, and a curve was developed to show its resistance at different temperatures. To eliminate the change in resistance of the copper cable due to changes in temperature a 2-lead cable was run into the well similar to the one to which the coil was attached. However, the second 2-lead cable was "shorted" or connected to itself at the bottom so that its resistance could be measured. In operation the resistance coil was lowered to the point at which a temperature reading was desired and the resistance measured by a Wheatstone bridge of the post-office type.

The resistance of the shorted cable was also measured and subtracted from the total resistance of the coil and cable, and the temperature was then read from the curve which had been obtained by calibrating the apparatus in the laboratory. Figure 13 is a diagram

of the instrument. It was found that temperature readings accurate to plus or minus 1° F. could be obtained with the apparatus when checked with an apparatus similar to that used by Van Orstrand<sup>22</sup> of the United States Geological Survey. Making temperature observations in oil wells with the electrical instrument is much more rapid than using maximum thermometers and is much more practical from a field standpoint. The use of electrolytic nickel wire in the construction of the coil would probably give a still more accurate apparatus.

The data obtained from the use of the electrical apparatus and those obtained by Van Orstrand made it possible to calculate the variation of the earth's temperature with depth at Salt Creek field. In the center of the field the temperature increases about 1° F. for every 30 feet of depth and, near the edge of the producing area, about 1° F. for every 40 feet of depth. A number of temperatures at the top and bottom of wells are given in Tables 5 and 6. With these temperature data it was possible to calculate a value for constant  $K$ . For mechanically pumped wells the loss of heat and the corresponding temperature drop of the oil are due undoubtedly to the radiation of heat to surrounding medium. Therefore, by use of the formula

$$Q' = aKT''Lt'$$

given by Barrett,<sup>23</sup> for heated oil lines, the actual value of  $K$  was calculated for these wells.

$$K = \frac{Q'}{aT''Lt'}$$

where

$K$  = radiation from pipe, B. t. u. per hour per square feet per degree temperature difference between pipe and air,

$Q'$  = quantity of heat radiated from pipe of length  $L$  in time  $t$ , B. t. u.,

$a$  = area of 1 foot of pipe, square feet,

$L$  = length of pipe, feet,

$t'$  = time required for oil to pass through  $L$  feet of pipe, hours, and

$T''$  = mean temperature difference between oil and surrounding medium calculated by the following formula:

$$T'' = \frac{T_1 - T}{\log e \left[ \frac{T_1 - T_0}{T - T_0} \right]}$$

where

$T_1$  = temperature of oil at beginning,

$T$  = temperature of oil at end of length  $L$ , and

$T_0$  = temperature of surrounding medium.

From Table 6 it can be seen that the value of  $K$  varies, due no doubt to the conditions existing between the tubing and the casing and the conditions of the surrounding medium in contact with the casing. If the surrounding medium in contact with the casing is water the radiation of heat will be more rapid than if it is moist or wet earth.

<sup>22</sup> Orstrand, C. E. Van, Apparatus for the Measurement of Temperatures in Deep Wells by Means of Maximum Thermometers: Econ. Geol., vol. 19, No. 3, 1924, pp. 229-247.

<sup>23</sup> Barrett, L. L., Pipe-Line Transportation of Hot Oil: Chem. and Met. Eng., vol. 24, No. 26, 1921, pp. 1148-1152.

TABLE 5.—Temperature data on flowing wells in the Salt Creek field, Wyoming

[Oil string, 2-inch tubing and packer; mean annual earth temperature, 46.7° F.]

Well	Temperature of sand, °F.	Depth, feet	Temperature of oil at casing head, °F.	Daily production, barrels	Gas per barrel of oil, cubic feet	Gasoline content of oil, gallons per minute	Pressure, at casing head, pounds per square inch	Remarks
8A, NW. ¼ sec. 13, T. 40 N., R. 79 W.	98.0	1,934	56	40	7,789	2.57	7	-----
16A, NW. ¼ sec. 13, T. 40 N., R. 79 W.	99.0	1,970	55	125	2,703	2.61	54	Test 1.
	99.0	1,970	69	109	2,198	3.06	297	Test 2.
	99.0	1,970	66	243	2,848	2.62	66	Test 3.
31A, SW. ¼ sec. 30, T. 40 N., R. 79 W.	96.0	1,836	81	575	1,085	1.74	76	-----
34A, SE. ¼ sec. 25, T. 40 N., R. 79 W.	95.0	1,745	75	269	1,057	1.77	41	-----

TABLE 6.—Temperature data on pumping wells in the Salt Creek field, Wyoming

[Size of tubing, 2-inch upset; mean annual earth temperature, 46.7° F.]

Well	Sand temperature, °F.	Temperature of oil at casing head, °F.	Daily production, barrels	Depth, feet	K	Remarks
<i>Data, December, 1926</i>						
19A, SE ¼ sec. 13, T. 40 N., R. 79 W.	98	72.0	95	1,972	-----	Pumping part time.
18A, SE ¼ sec. 13, T. 40 N., R. 79 W.	97	59.5	7	1,914	-----	Do.
30A, NW ¼ sec. 13, T. 40 N., R. 79 W.	97	80.0	118	1,919	-----	Do.
35A, NE ¼ sec. 1, T. 40 N., R. 79 W.	101	83.0	65	2,004	-----	Do.
26A, NE ¼ sec. 1, T. 40 N., R. 79 W.	101	69.0	16	1,967	-----	Do.
2A X, NW ¼ sec. 36, T. 40 N., R. 79 W.	94	82.0	270	1,597	1.14	Pumping steady 24 hrs.
35A, SW ¼ sec. 25, T. 40 N., R. 79 W.	94	74.0	25	1,620	-----	Pumping part time.
13A, SW ¼ sec. 25, T. 40 N., R. 79 W.	96	76.0	13	1,578	-----	Do.
18A, SE ¼ sec. 25, T. 40 N., R. 79 W.	94	80.0	60	1,658	-----	Do.
9A, SE ¼ sec. 25, T. 40 N., R. 79 W.	96	77.0	90	1,672	-----	Do.
20A, SE ¼ sec. 25, T. 40 N., R. 79 W.	98	82.0	170	1,688	0.80	Pumping steady 24 hrs.
22A, SE ¼ sec. 25, T. 40 N., R. 79 W.	98	78.5	190	1,850	1.42	Do.
27A, SE ¼ sec. 25, T. 40 N., R. 79 W.	100	70.5	35	1,826	-----	Pumping part time.
36A, SE ¼ sec. 25, T. 40 N., R. 79 W.	97	65.0	35	1,910	-----	Do.
1A, NE ¼ sec. 36, T. 40 N., R. 79 W.	100	69.0	50	1,911	-----	Do.
21A, NE ¼ sec. 36, T. 40 N., R. 79 W.	100	64.0	5	1,765	-----	Do.
25A, NE ¼ sec. 36, T. 40 N., R. 79 W.	100	68.5	25	1,850	-----	Do.
<i>Data, May, 1929</i>						
9A, SE ¼ sec. 25, T. 40 N., R. 79 W.	96	71.5	19	1,672	-----	Do.
20A, SE ¼ sec. 25, T. 40 N., R. 79 W.	96	81.0	103	1,688	0.54	Pumping steady 24 hrs.
36A, SE ¼ sec. 25, T. 40 N., R. 79 W.	97	69.0	12	1,910	-----	Pumping part time.
35A, NE ¼ sec. 1, T. 40 N., R. 79 W.	101	76.0	10	2,004	-----	Do.
22A, SE ¼ sec. 25, T. 40 N., R. 79 W.	98	87.0	185	1,850	0.54	Pumping steady 24 hrs.

The average value for  $K$  can be used in the following formulas, from page 28, to calculate the final temperature of the oil in a flowing well with tubing, due to radiation of heat to the surrounding medium.

$$t_x = (t_1 - t_0) 10^{-\left[\frac{600 DKX}{WQC}\right]} + t_0 \text{ or } t = t_0 + (t_1 - t_0)e^{-Y}.$$

In wells flowing through casing in contact with the surrounding medium, the value of  $K$  can not be obtained by experimental data taken from the well, and a value must be assigned that has been taken

from other experimental data. In pipe lines,  $K$  ranges from 0.2 to more than 1. The lower values are associated with dry, well-drained soil, and the higher values are those obtained when the pipe line traverses swampy ground. A value for  $K$  in a well flowing through casing would therefore depend upon the condition of the surrounding medium. The writer believes, from a study of pipe-line and pumping-well data, that 0.9 is a fair value for  $K$  in the average well flowing through casing.

The following examples illustrate the derivation of the constant  $K$  from pumping-well temperature data and the computation of heat losses from radiation to a surrounding medium in flowing wells. Constant  $K$  can be calculated for pumping wells by using the formulas on page 30, because the only loss of heat in pumping wells is that by radiation. For example, substituting the values taken for well 22A, SE.  $\frac{1}{4}$  sec. 25, T. 40 N., R. 79 W., May, 1929 (Table 6), in the following formulas,  $K$  equals 0.540.

$$K = \frac{Q'}{aT^n L t'} \quad \text{and} \quad T^n = \frac{T_1 - T}{\log e \left[ \frac{T_1 - T_0}{T - T_0} \right]}$$

where

$$T_0 = \frac{98 + 46.7}{2} = 72.3^\circ \text{ F.} \quad T_1 = 98^\circ \text{ F.} \quad T = 87^\circ \text{ F.}$$

$$T^n = \frac{98 - 87}{\log e \frac{98 - 72.3}{87 - 72.3}} = 19.7^\circ \text{ F.}$$

$$Q' = 185 \times 42 \times 6.9 \times 0.5 \times 11 = 294,877 \text{ B.t.u.}$$

$$L = 1,850 \text{ feet} \quad t' = 24$$

$$K = \frac{294,877}{0.6219 \times 19.7 \times 1,850 \times 24} = 0.5421.$$

To calculate the decrease in temperature of the oil due to radiation of heat the following formula can be used:

$$t_x = (t_1 - t_0) 10^{-\left[ \frac{600 D K X}{W Q X} \right]} + t_0.$$

Substituting the data for well 22A, SE.  $\frac{1}{4}$  sec. 25, T. 40 N., R. 79 W. (Table 6), the theoretical temperature  $t_x$  can be determined.

$$t_1 = 98^\circ \quad t_0 = \frac{98 + 46.7}{2} = 72.3 \quad D = 2.375$$

$$K = 0.54 \quad X = \frac{1,850}{5.280} = 0.3503 \quad W = 42 \times 6.9 = 290$$

$$Q = \frac{185}{24} = 7.7 \quad C = 0.5$$

$$t_x = (98 - 72.3) 10^{-\left[ \frac{600 \times 2.375 \times 0.54 \times 0.3503}{290 \times 7.7 \times 0.5} \right]} + 72.3$$

$$t_x = 87.8^\circ \text{ F.}$$

This calculated temperature compares favorably with the actual temperature at the wellhead—87.0° F.

In flowing wells the oil is cooled not only by radiation of heat to the surrounding formations but also by other factors. The theoretical decrease in temperature due to radiation of heat can be calculated, providing the well is flowing steadily and not by heads with long periods of surging between flows. For wells flowing through tubing  $K$  should be approximately the same as for pumping wells.

If the data given in Table 5 are used and  $K$  is assumed to be 0.54, the theoretical decrease in temperature of the oil due to radiation of heat to the surrounding formations for well 31A, SW.  $\frac{1}{4}$  sec. 30, T. 40 N., R. 79 W., would be found as follows:

$$t_1 = 96^\circ \quad t_0 = \frac{96 + 46.7}{2} = 72.3 \quad D = 2.375$$

$$K = 0.54 \quad X = \frac{1836}{5280} = 0.3477 \quad W = 42 \times 6.9 = 290$$

$Q$  = barrels of oil per hour plus gas per hour in equivalent of barrels of oil. The weight of 1 cubic foot of gas is assumed to be 0.0657 pound and 290 pounds of gas equivalent to 1 barrel of oil

$$Q = \frac{575}{24} + \frac{1085 \times 575 \times 0.0657}{24 \times 290} = 29.9$$

$$C = 0.5$$

$$t_x = (96 - 71.3) 10^{-\left[\frac{600 \times 2.375 \times 0.54 \times 0.3477}{290 \times 29.9 \times 0.5}\right]} + 71.3$$

$$t_x = 93^\circ \text{ F.}$$

The decrease in temperature due to radiation would be  $96^\circ - 93^\circ$ , or  $3^\circ$  F. The actual temperature of the oil at the casing head was  $81^\circ$  F., which indicates that  $12^\circ$  of cooling was due to work performed by the gas in lifting the oil and to other causes.

Changing the size of the oil string or using tubing in place of casing, which is in contact with the surrounding earth, causes little change in the value of constant  $K$ . It is true apparently that the radiation of heat is slightly more rapid from smaller pipes under ideal conditions, but this difference does not seem large enough to alter the value of  $K$  due to the other conditions. The most practical way to decrease or minimize the loss of heat to surrounding formations is by choice of the proper size of oil string to obtain the most rapid movement of oil from the well with efficient operation from a standpoint of work required. With oil strings of proper diameter the length of time that the oil is subjected to loss of heat by radiation is reduced, and as radiation of heat is proportional to the velocity or time required for the oil to flow through the pipe less heat will be lost from the oil where it moves more rapidly through the pipe. For example, if 4-inch tubing instead of 2-inch tubing had been used in the well given previously, the theoretical decrease in temperature would have been  $5.7^\circ$  F.

$$t_x = (96 - 71.3) 10^{-\left[\frac{600 \times 4.50 \times 0.54 \times 0.3477}{290 \times 29.9 \times 0.5}\right]} + 71.3$$

or

$$t_x = 90.3 \text{ for 4-inch tubing.}$$

The temperature drop would be  $96^\circ$  F. minus  $90.3^\circ$  F., or  $5.7^\circ$  F. The temperature drop for 2-inch tubing in this well was  $3^\circ$  F. The temperature drop is almost twice as great for 4-inch tubing and shows that decreasing the velocity by using larger tubing increases the loss of heat by radiation.

Whereas the greater part of the cooling in a flowing well is the result of the expansion of gas while lifting oil, radiation of heat to the surrounding formations plays an important part in the cooling of oil in pumping wells, especially those that are being pumped intermittently.

### COOLING PRODUCED BY LIBERATION OF GAS FROM SOLUTION

Although the writer did not perform any experiments to determine the amount of cooling caused by gas being liberated from solution, he believes from general observations in the laboratory that this cooling is negligible and plays no part in the problem of paraffin deposition.

### EFFECT OF WATER PRODUCED WITH OIL ON TEMPERATURE IN WELL

Laboratory and field observations show conclusively that the presence of water does not change the solubility of wax in oil, but certain temperature conditions may be produced by the intrusion of water which sometimes affect the deposition of paraffin.

The entrance of edge water or top water into the oil-bearing portion of the reservoir sand may result in congealing of the oil and deposition of paraffin in the pores of the sand, provided the temperature of the water is below the point where the oil is saturated with wax; but this condition is unusual, as the temperature of the water entering the sand is usually above the temperature at which the oil becomes saturated with paraffin.

The entrance of water into the well, particularly bottom water, usually decreases the quantity of paraffin deposited in the oil string of the well. Field observations showed that when a well started to produce water the temperature of the liquid at the top of the well increased. This increase in temperature is due to the fact that, although the temperature of the water and oil in the sand is no greater than it was before the entrance of water, the specific heat of water is twice that of oil, and water therefore contains twice the thermal energy per pound that oil does. The temperature of the water would therefore be reduced only half as much as the oil by the same loss of heat. Besides supplying more available heat, water also increases the volume and velocity of flow, thereby allowing less time for radiation of heat to the surrounding formations. Unfortunately, the writer was unable to obtain any actual temperatures of wells before and after they started to produce water, but the fact that the temperature does increase is so well known that additional data seem unnecessary. Although water helps prevent the accumulation of paraffin in some wells by retarding cooling of the oil, other difficulties encountered with the production of water with oil offset the advantages that might be obtained by the use of water as a remedial measure in preventing deposition of paraffin.

### EFFECT OF EVAPORATION OF LIQUID CONSTITUENTS

The crude oil and gas in the reservoir sand are originally in equilibrium. The oil is saturated under the existing conditions of pressure and temperature with the gaseous constituents, and the partial pressure exerted by the vapor of each liquid constituent is equal to that liquid's vapor pressure. When the reservoir is penetrated by a well equilibrium conditions are destroyed because of the removal of oil and gas and some additional gas that is liberated from solution in the oil remaining in the reservoir. During production the volatile constituents continually tend to vaporize enough to reestablish the

equilibrium between their partial pressure and vapor pressure. The vapor pressure of the lighter constituents which constitute a major portion of the crude oil lost by evaporation is shown in Figure 14, and the partial pressure of different percentages of a gas in a mixture of gases at various pressures is given in Table 7. There is a critical temperature for each gas above which it can not be liquefied at any pressure, but below this temperature the gas will liquefy at a pressure equal to or in excess of its vapor pressure. If the quantity of any liquid constituent in a gas and oil mixture is insufficient to create enough vapor so that its partial pressure will be equal to its vapor pressure, the constituent will not condense and exist in the system as a liquid but will be entirely vaporized. However, a certain quantity of the gas may be held in solution in the liquid. For

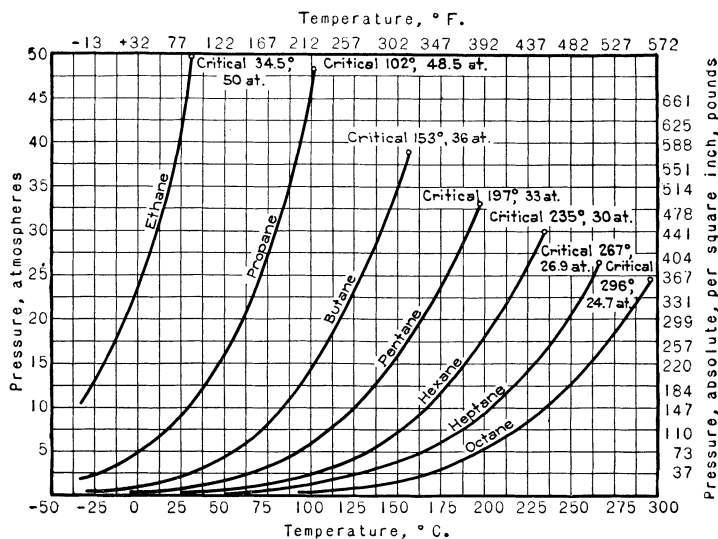


FIGURE 14.—Vapor pressure of saturated paraffin hydrocarbons, after Hamor and Padgett

example, consider a mixture of methane and butane at a pressure of 515 pounds per square inch absolute and a temperature of 100° F. At this temperature no amount of pressure would liquefy the methane because the temperature is above the critical temperature of methane, which is approximately minus 115° F.

Some gaseous methane may, however, be held in solution by the butane, which can be liquefied because its critical temperature is approximately 307° F. If there is only enough butane to make 5 per cent of the gaseous mixture the partial pressure of the butane vapor will be 26 pounds per square inch, or below the vapor pressure of butane, which is approximately 53 pounds per square inch at 100° F.; consequently, the butane will exist in the mixture only as a gas. If, however, there is enough butane to supply more vapor than 10.1 per cent of the total gas mixture the partial pressure of the butane will be above 53 pounds per square inch, and the additional butane (in excess of that required to make 10.1 per cent of the total vapor) will be in the liquid phase.



TABLE 7.—Partial pressures for various percentages of gas in gas mixture<sup>1</sup>

Gas by volume in total mixture, per cent	Pressure on total mixture, pounds																		
	1,515	1,415	1,315	1,215	1,115	1,015	915	815	715	615	515	415	315	215	115	90	65	40	15
	Partial pressure, pounds																		
5.-----	76	71	66	61	56	51	46	41	36	31	26	21	16	11	6	5	3	2	0.75
10.-----	151	141	131	121	111	101	91	81	71	61	51	41	31	21	11	9	6	4	1.5
15.-----	227	212	197	182	167	152	137	122	107	92	77	62	47	32	17	14	10	6	2.25
20.-----	303	283	263	243	223	203	183	163	143	123	103	83	63	43	23	18	13	8	3.0
25.-----	379	354	329	304	279	254	229	204	179	154	129	104	79	54	29	23	16	10	3.75
30.-----	454	424	394	364	334	304	274	244	214	184	154	124	94	64	34	27	20	12	4.5
35.-----	530	495	460	425	390	355	320	285	250	215	180	145	110	75	40	32	23	14	5.25
40.-----	606	566	526	486	446	406	366	326	286	246	206	166	126	86	46	36	26	16	6.0
45.-----	682	637	592	547	502	457	412	367	322	277	232	187	142	97	52	41	29	18	6.75
50.-----	757	707	657	607	557	507	457	402	357	307	257	207	157	107	57	45	33	20	7.5
55.-----	833	778	723	668	613	558	503	448	393	338	283	228	173	118	63	50	36	22	8.25
60.-----	909	849	789	729	669	609	549	489	429	369	309	249	189	129	69	54	39	24	9.0
65.-----	985	920	855	790	725	660	595	530	465	400	335	270	205	140	75	59	42	26	9.75
70.-----	1,060	990	920	850	780	710	640	570	500	430	360	290	220	150	80	63	46	28	10.5
75.-----	1,136	1,061	986	911	836	761	686	611	536	461	386	311	236	161	86	68	49	30	11.25
80.-----	1,212	1,132	1,052	972	892	812	732	652	572	492	412	332	252	172	92	72	52	32	12.0
85.-----	1,287	1,202	1,117	1,032	947	862	777	692	607	522	437	352	267	182	97	77	55	34	12.75
90.-----	1,363	1,273	1,183	1,093	1,003	913	823	733	643	553	463	373	283	193	103	81	59	36	13.5
95.-----	1,439	1,344	1,249	1,154	1,059	964	869	774	679	584	489	394	299	204	109	86	62	38	14.25
100.-----	1,515	1,415	1,315	1,215	1,115	1,015	915	815	715	615	515	415	315	215	115	90	65	40	15.0

<sup>1</sup> Calculated by K. B. Nowels.

If "dry" gas or gas containing less vapor of the liquid constituents than that required to create a partial pressure equal to their vapor pressure at any particular temperature is introduced into a gas and oil mixture the liquid constituents will evaporate until they have formed enough vapor in the system by evaporation to create a partial pressure equal to their combined vapor pressure.

These conditions are continually taking place in the sand and in the well after a reservoir has been tapped. The pressure on the oil and gas is reduced as it flows from the sand to the well and up to the surface. There is, therefore, constant evaporation of the liquid constituents to supply more vapor and maintain a partial pressure equal to their vapor pressure. Moreover, the gases of the noncondensable and condensable hydrocarbons are liberated from solution<sup>24</sup> as the pressure declines, and the released gases reduce the concentration of the vapors of the liquid constituents thus reducing their partial pressure; consequently, additional liquid must vaporize to maintain the partial pressure equal to the vapor pressure.

In gas-lift wells the additional gas used for lifting the oil also reduces the vapor concentration of each of the liquid constituents, and unless the total pressure is increased evaporation takes place to restore the equilibrium between the partial pressure of the vapor and the vapor pressure of the liquid.<sup>25</sup>

In natural-flowing and gas-lift wells the evaporation of liquid constituents (natural gasoline) continues while the oil is flowing from the sand to the top of the well, and due to the agitation and the resultant intimate mixing of the oil and gas conditions probably approach the theoretical in which the partial pressure of the vapor of each component in the gas is equal to its vapor pressure as a liquid. In pumping wells there is little or no evaporation of the liquid constituents after they enter the tubing, as most of the gas is separated from the oil before it enters the tubing and only the liquid phase is present. This condition, of course, exists only in pumping wells in which the gas separates from the oil at the bottom of the well and not in wells that are "agitated" and in which the gas is allowed to flow with the oil or wells in which gas is injected into the tubing to help lift the oil to the surface.

Evaporation of the liquid constituents of crude oil reduces the temperature of the oil, due to the latent heat of vaporization required to change the liquid constituents from the liquid to the vapor state. The amount of heat thus required is probably so small that it is supplied by the formation while evaporation takes place in the sand or at the bottom of the well; consequently, the temperature of the oil is changed slightly or not at all. In the casing or tubing, however, where the surrounding medium is cooler than the oil, this vaporization consumes some of the thermal energy of the oil, and the temperature of the oil is reduced. The latent heat of vaporization of different boiling-point fractions of crude oil is given in Table 8. These data were taken from an article by Leslie, Geniesse,

<sup>24</sup> Dow, D. B., and Calkin, L. P., Solubility and Effects of Natural Gas and Air in Crude Oils: Rept. of Investigations 2732, Bureau of Mines, February, 1926, 13 pp.; Mills, R. van A., and Heithecker, R. E., Volumetric and A. P. I. Changes Due to the Solution of Gas in Crude Oils: Rept. of Investigations 2893, Bureau of Mines, October, 1928, 15 pp.

<sup>25</sup> Neal, R. O., Effect of Gas Lift on Oil and Gas: Oil and Gas Jour., vol. 26, No. 39, February, 1928, pp. 120-121.

Legatski, and Jagrowski<sup>26</sup> and represent the results of experimental work with a paraffin-base petroleum. The latent heat of vaporization decreases with increase in pressure and becomes zero at the critical temperature and pressure. The heat of vaporization of a number of paraffin hydrocarbons at different pressures, calculated by Hildebrand's modification of Trouton's rule, is given by Wilson and Bahlke.<sup>27</sup> A copy of the curves is shown in Figure 15.

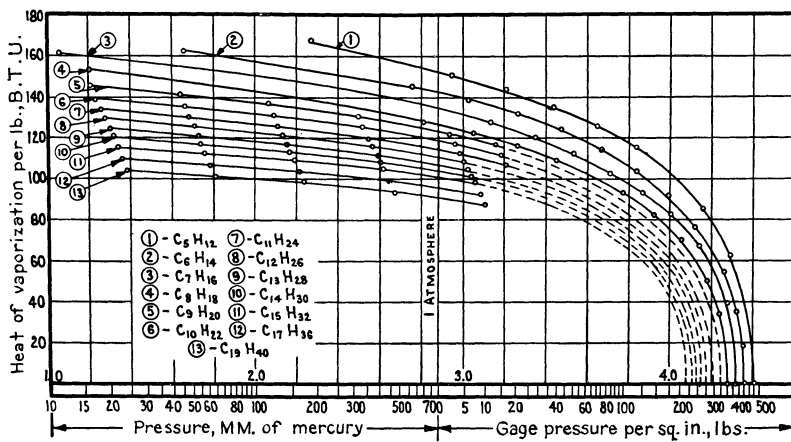


FIGURE 15.—Latent heat of vaporization of saturated paraffin hydrocarbons, after Wilson and Bahlke

TABLE 8.—Latent heat of vaporization of distillates from paraffin-base petroleum

[From article by Leslie, Geniesse, Legatski, and Jagrowski]

Boiling point of fraction, °F.	Specific gravity, 60°/60° F.	B. t. u. per pound	B. t. u. per gallon, 60° F.	B. t. u. per barrel (42 gallons), 60° F.
150.....	0.678	142.4	805	33,800
200.....	.720	134.1	805	33,800
250.....	.731	125.7	766	32,200
300.....	.755	117.4	738	31,000
350.....	.775	109.0	704	29,600
400.....	.788	100.6	661	27,800
450.....	.805	92.2	618	26,000
500.....	.815	83.9	569	23,900
550.....	.822	75.1	517	21,700
600.....	.823	67.1	460	19,350

The approximate cooling of the oil due to evaporation of the lighter constituents can be calculated if the quantity of lighter constituents evaporated from the oil is known. For example, a gas-lift well produced oil of 37.1° A. P. I. gravity with a gasoline content of 12.6 gallons per barrel, and an offsetting well which was pumping "on the beam" produced oil of 38.2° A. P. I. gravity with a gasoline content of 13.6 gallons per barrel. If it is assumed that the oil at the bottom of the wells was the same, the difference in gasoline con-

<sup>26</sup> Leslie, E. H., Geniesse, J. C., Legatski, T. W., and Jagrowski, L. H., Latent Heats of Vaporization of Distillates from Paraffin-Base Petroleum: *Ind. and Eng. Chem.*, vol. 18, No. 1, 1926, pp. 45-46.

<sup>27</sup> Wilson, R. E., and Bahlke, W. H., The Physical Properties of the Paraffin Hydrocarbons: *Ind. and Eng. Chem.*, vol. 16, No. 2, 1924, pp. 115-122.

tent indicates that 1 gallon of gasoline per barrel of oil evaporated from the crude while it was flowing from the bottom to the top of the gas-lift well. As has been shown, the latent heat of vaporization decreases with an increase in pressure and therefore would be somewhat less in the well than at atmospheric pressure. Assuming that the latent heat of the gasoline evaporated in this well under the existing pressure conditions was 650 British thermal units per gallon, which is probably a very conservative value, the amount of cooling due to evaporation can be calculated. The weight of 1 barrel of 38.2° A. P. I. gravity oil is approximately 294 pounds. Assuming that 2,000 cubic feet of gas, specific gravity 0.9, was produced and circulated per barrel of oil the gas would weigh approximately 130 pounds, making a total of 424 pounds of oil and gas to be cooled per barrel of oil produced. The specific heat of the total mixture would therefore lower the temperature 3.1° F.

The loss of heat due to evaporation of the lighter constituents is probably sufficient to cool the oil 2° to 4° F. for each gallon of liquid constituents that vaporize per barrel of oil while the oil is flowing from the well. When the pressures in the oil string of the well approach 400 to 500 pounds per square inch, the latent heat of vaporization probably will be so low that there will be little cooling due to evaporation. In wells in which the pressure at the top of the well is high and the gas-oil ratio is low the amount of liquid evaporated from the oil will also be negligible, and very little cooling will take place. Where the gas-oil ratio is high, however, evaporation and cooling undoubtedly occur. In the early life of the Panhandle field dry gas from a sand above the oil entered many of the wells, resulting in the production of oil with gas-oil ratios of 10,000 to 20,000 cubic feet per barrel. The dry gas removed 0.5 to 1.5 gallons of gasoline per thousand cubic feet of gas from the oil while it was flowing from the well and caused the loss of 5 to 10 per cent of the oil by evaporation and cooling of 10° to 20° F.

Cooling and the loss of liquid constituents by evaporation are negligible factors in the sand, or in properly operated pumping wells, and can be reduced in natural-flowing or gas-lift wells by efficient lifting of the oil and by preventing excessive gas-oil ratios.

#### **MANNER IN WHICH PARAFFIN ACCUMULATES IN WELL AND SAND**

It was found in the laboratory and in the field that the separation of wax from solution in the oil and the presence of wax crystals were not sufficient to cause accumulation of paraffin on pipe or other objects. Many wells produced oil containing suspended wax in large quantities without paraffin accumulating, whereas in other wells paraffin accumulated rapidly. The deposition of paraffin depends upon conditions that cause wax to separate from solution in the oil and those that cause the wax to build up on objects with which it comes in contact. In the oil string of a well the following factors are usually responsible for the deposition of paraffin on the pipe: (1) The alternate coating of the pipe and draining of the oil, (2) the presence of only a film of oil in contact with the pipe while the well is flowing, and (3) the contact of the oil with a cold surface.

**ACCUMULATION DUE TO INTERMITTENT COATING OF SURFACE**

The alternate coating of the pipe with oil is usually the most important cause of deposition of paraffin. The manner in which the paraffin attaches itself to the pipe when this condition takes place has been discussed previously. In wells that flow by heads there is a constant coating and draining of oil on the surface of the pipe, consequently heading wells offer the best conditions for the separation and accumulation of paraffin. The oil in such wells is being lifted and dropped by the gas until enough has accumulated to cause a flow, and each time the oil is lifted it gives up some of its thermal energy and volatile constituents to the gas. Radiation of heat to the surrounding earth also takes place. Therefore, when the oil reaches the surface it is much cooler than if the wells were flowing constantly. These conditions result in rapid accumulation of paraffin. When the period between heads is short the trouble is less severe, because the oil film does not have time to drain away from the wax particles and leave them attached to the pipe. The conditions causing separation of paraffin from solution in the oil are also modified in wells flowing by short heads. Intermittently operated gas-lift wells also produce the same conditions as heading wells, except that the oil is not being lifted, cooled, and evaporated between flows. In agitated pumping wells coating and draining take place where gas associated with or coming out of solution in the oil causes it to flow from the tubing at intervals instead of pumping steadily.

The temperature of the pipe and the amount of gas flowing during the intervals between flows also affect the film of oil and may cause additional paraffin to crystallize from solution; however, deposition of paraffin by intermittent coating of the pipe rarely extends below the point where the temperature of the pipe and surrounding medium is above that at which the oil is saturated with wax.

**ACCUMULATION DUE TO FORMATION OF FILM OF OIL**

The actual flowing conditions of oil and gas vary in different wells. Some wells flow a solid stream of oil; others flow alternate slugs of oil and gas. In still other wells where excessive gas breaks up the oil into finely divided particles, the oil is produced as a mist suspended in the gas. If the oil is produced as a mist, which is carried along by the high velocity of the gas, only a film of oil adheres to the pipe, due to the oil mist coming in contact with it. If the film of oil contains free wax crystals or particles that have separated from solution in the oil and is not thick enough to carry the wax particles in complete suspension, the particles come in contact with the pipe and due to their soft, gummy nature become firmly attached to it.

**ACCUMULATION DUE TO CONTACT WITH COLD OBJECTS OR SURFACES**

Wax seldom accumulates on cold surfaces in the flow string of oil wells except at points of unusual cooling, such as restrictions in the pipe, jets, and orifices through which the gas expands. The accumulation of paraffin is usually rapid at such points.

## CAUSES AND METHODS OF OPERATION FOR PREVENTING ACCUMULATION OF PARAFFIN

Paraffin troubles in oil-producing wells can be successfully overcome in two general ways: (1) By applying proper production methods which prevent or decrease the accumulation of paraffin; and (2) by removing the paraffin at regular intervals before it accumulates sufficiently to retard the rate of oil production. The accumulation of paraffin can often but not always be prevented by changing the conditions within the well and the manner in which the oil is produced. If the troubles due to the accumulation of paraffin can not be eliminated by changing the diameter of the flow string, altering the amount of back pressure held on the well, or otherwise changing the gas-oil ratio and method of production, heat or mechanical methods of preventing or removing the deposited paraffin can be used. Methods of preventing the accumulation of paraffin, other than by the correct operation and control of the conditions within the well, must be continued as long as paraffin accumulates and are often very expensive.

Methods of removing the paraffin at regular intervals are usually most practical where the accumulation of paraffin can not be prevented by proper control of the conditions within the well and are discussed later.

### CAUSES AND PREVENTION IN OIL STRING OF FLOWING WELLS

Preventing the accumulation of paraffin in the oil string of flowing wells is of major importance to many operators because of the resultant helpful effect on production of oil. The factors responsible for the accumulation of paraffin in flowing wells can often be controlled and paraffin troubles eliminated or modified. These changes frequently increase the rate of production and general efficiency of well operation.

In flowing wells the utilization of thermal energy by the gas in expanding and lifting the oil, the radiation of heat to the surrounding formations, and the evaporation of some of the liquid constituents cool the oil and cause wax to be precipitated from solution. The evaporation of liquid constituents decreases the actual volume of liquid available to keep the wax in solution. If wax accumulates in the oil string not only must the paraffin be precipitated from solution, but also conditions must be favorable for the paraffin to become attached to the pipe.

The conditions mainly responsible for the accumulation of paraffin in natural flowing wells are the alternate coating and draining of the pipe, when the well is flowing by heads, or the presence of a film of oil on the pipe, when the oil is produced with an excessive quantity of gas and carried in small droplets or as a mist suspended in the gas. The manner in which these conditions cause the paraffin to accumulate on the pipe has been discussed previously.

The accumulation of paraffin can be modified or prevented in natural flowing wells by keeping the wax in solution in the oil or by avoiding conditions that cause the precipitated wax to become

attached to the pipe. To prevent excessive cooling and evaporation and the precipitation of wax from solution in the oil, the oil must be produced with a minimum of gas and a steady flow maintained. This will tend to eliminate or modify conditions that cause the precipitated wax to become attached to the pipe. These conditions can usually be regulated by the use of proper size tubing, a flow bean, back pressure, or a combination of these. Proper regulation of back pressures in flowing wells usually requires considerable experimentation on each well because it is generally impossible to predetermine the exact pressure conditions, size of tubing, and other factors necessary to obtain the desired results. Even after conditions are established in the well to prevent the accumulation of paraffin they will usually have to be altered from time to time as the rate of oil and gas production changes.

The production and clean-out records of a number of wells in the Salt Creek field substantiate these conclusions. The wells in this field were generally completed with  $5\frac{3}{16}$  or  $6\frac{5}{8}$  inch casing and were allowed to produce through the casing until they paraffined up or stopped flowing from other causes. Usually, the wells produced for six or eight months before paraffin troubles were experienced. If the paraffin was then removed from the casing and the wells tubed the trouble due to paraffining was generally eliminated until the production again declined to the point where the wells flowed by heads. It was then necessary to clean out the wells at regular intervals or pump them to prevent further trouble from paraffin. The history of well 2A, NE.  $\frac{1}{4}$  sec. 25, T. 40 N., R. 79 W., is typical of these conditions. This well was completed in April, 1921, and produced 1,440 barrels of oil per day through a  $6\frac{5}{8}$ -inch casing. Early in 1924 production had declined to the point where the well started flowing by heads and finally paraffined up completely. In October, 1924, 2-inch tubing was placed in the well and it flowed steadily for several months before starting to flow by heads. The well paraffined up in April, 1925, and from April until June paraffin had to be removed every two weeks. In July the well was equipped for pumping. Since then the use of a gas anchor below the working barrel has eliminated paraffin trouble.

Another example of the effect of flowing by heads on paraffin accumulation was obtained by Nowels<sup>28</sup> in his experiments with flowing wells in the Salt Creek field, Wyoming. Well 34A, SE.  $\frac{1}{4}$  sec. 25, R. 40 N., T. 79 W., when producing through 2-inch tubing on a 17-day test, averaged 216 barrels of oil per day with a gas-oil ratio of 444 cubic feet of gas per barrel of oil. This gas contained 5.8 gallons of gasoline per thousand cubic feet, calculated from total recovery by the charcoal method. The oil flowed steadily under these conditions, and no indications of paraffin accumulations were noted. The well was then partly closed in by use of a smaller flow bean, so that it only produced 100 barrels of oil a day. In four days production declined to 24 barrels per day, with a gas-oil ratio of 500 cubic feet of gas per barrel of oil. The gasoline content of the gas was 6.01 gallons per thousand cubic feet. The following day the well was completely

<sup>28</sup> Nowels, K. B., Some Methods of Producing Flowing Wells in the Salt Creek Field and Their Effect on Gas-Oil Ratios: Rept. of Investigations 2833, Bureau of Mines, 1927, pp. 24-25.

paraffined up. During the 5-day period that the well produced by heads the oil that flowed from the well was subjected to more cooling and evaporation as a result of flowing by heads and the increased gas production. The well was later produced "wide open" again through the 2-inch tubing and produced an average of 269 barrels of oil per day with no signs of paraffin trouble.

In some fields the high gas-oil ratios are often responsible for paraffin troubles in the wells. The excessive production of gas causes a large loss of volatile constituents from the oil with excessive cooling, and usually the oil rises in the well in the form of a mist which leaves a film of oil in contact with the oil string and causes paraffin to accumulate rapidly. In the Panhandle field, Texas, gas-oil ratios of 10,000 to 20,000 cubic feet per barrel of oil were not uncommon. A satisfactory method of preventing high gas-oil ratios was to land the oil string below the gas horizon and shut the gas off by cement or a packer. Reducing gas-oil ratios usually helped to prevent the accumulation of paraffin. Trouble, however, was still experienced in wells that were producing by heads instead of flowing steadily.

Loose sand and cavings, although not responsible for the accumulation of paraffin, tend to be deposited with the wax and aid in increasing its bulk rapidly. In wells where paraffin troubles can not be eliminated it is advisable to prevent cavings and loose sand from entering the well, as paraffin accumulations often consist of 50 per cent sand and silt. Inserting a screen or perforated liner after cleaning the well is often the best method of preventing loose sand and cavings from entering the well.

To prevent paraffin troubles in flowing wells, they must therefore be operated so that cooling and evaporation of the oil are minimized and those conditions prevented that favor the accumulation of paraffin after it has separated from solution in the oil.

Although it is often possible to prevent or greatly modify the accumulation of paraffin by control of the factors discussed, obviously, incorrect methods will increase the trouble. There are always wells that it is either impossible or impractical to produce so as to prevent trouble. In such wells the deposition of paraffin must be prevented or removed by other than production-control methods.

#### CAUSES AND PREVENTION IN GAS OR AIR LIFT WELLS

The deposition and accumulation of paraffin in the oil string of gas or air lift wells are generally due to the same conditions that exist in natural flowing wells. In wells where jets, orifices, nozzles, or foot pieces are used paraffin often accumulates at points of local cooling, owing to the rapid expansion and acceleration of the gas as it passes through the restricted openings.

To prevent or reduce trouble from accumulations of paraffin in gas-lift wells so that there will be a minimum of cooling and evaporation the same precautions must be observed as in the operation of natural flowing wells. The volume and input pressure of the gas and the size of tubing must be selected so that conditions of minimum cooling and evaporation can be maintained. The oil should also be flowed through the tubing instead of the annular space between the tubing and the casing to reduce the amount of heat lost



from the oil by radiation to the surrounding formations. Maintaining a rapid flow of oil and gas through the tubing also facilitates the removal of accumulated paraffin. The excessive use of gas, resulting in excessive cooling and evaporation, and production of the oil as a mist, which leaves only a film of oil in contact with the pipe, cause most paraffin troubles in gas and air lift wells. The intermittent flowing of wells seldom causes trouble unless there are long intervals between flows, but even then the trouble is seldom serious because oil is not being lifted and dropped by the gas between periods of flow as it is in heading natural flowing wells.

Obviously, in many gas-lift wells, as in natural-flowing wells, it is impractical or impossible to control flowing conditions and prevent accumulation of paraffin, and the problem must be combated by other means.

#### CAUSES AND PREVENTION IN MECHANICALLY PUMPED WELLS

Pumping wells can be classified roughly in two groups: (1) Those producing during the early life of the field or during the transition period when the wells can no longer be produced satisfactorily by natural flow or gas-lift methods and (2) old, settled pumpers that produce only a small quantity of oil and are generally operated only part time. The two types of wells present different problems in the accumulation of paraffin and will, therefore, be discussed separately.

##### PUMPING WELLS IN TRANSITION PERIOD

During the transition period certain pumping wells paraffin up rapidly while others give little or no trouble, depending upon the manner in which they are operated. Although the daily accumulation of paraffin in the wells does not appreciably affect the amount of oil recovered, paraffining of the tubing must be prevented or the accumulation of paraffin removed before it plugs the tubing completely. Otherwise, sucker rods will part, pump cups wear excessively, and valves and working barrels fail to function properly; also, the tubing may split. If the wells are pumped electrically the accumulation of paraffin in the tubing may cause overloading and failure of the pumping motors. In any event, paraffining of pumping wells results in expensive replacement of equipment and loss of production while the wells are shut down for repairs.

The separation of paraffin from solution in the oil in pumping wells is due primarily to the reduction in temperature of the oil, accompanied by evaporation in some cases. The cooling is almost entirely due to the radiation of heat to the surrounding formations, and the slower the movement of the oil the more nearly it will approach the earth temperature. The temperature of the oil at the top of a number of pumping wells is given in Table 5, page 31. Table 5 shows that the wells of larger production, and consequently more rapid movement of oil, have a higher temperature at the wellhead. The loss of heat from oil by radiation has been fully discussed previously.

In some wells the oil is subjected to evaporation while at the bottom of the well, due either to the application of vacuum to the well or to the escape of gas which is produced with the oil. Evaporation

or loss of volatile constituents lowers the solvent power of the oil for wax, and the wax separates from solution at a higher temperature. However, in addition to cooling effect in pumping wells, the evaporation of volatile constituents from the oil, and the separation of wax from solution in the oil, other conditions must exist in the well that will cause the paraffin to become attached to the walls of the pipe and the sucker rods; otherwise, the paraffin will remain suspended in the oil and flow with the oil from the well.

The major cause of the accumulation of paraffin in wells during their early pumping life is the manner in which the oil flows from the well. During field investigations in Wyoming, Texas, and California it was observed that paraffin accumulated in certain pumping wells that were "agitated" or flowed oil by heads from the tubing, whereas wells that pumped steadily and thus maintained the tubing completely full of oil seldom gave paraffin trouble. If oil enters the tubing saturated with gas at a pressure higher than atmospheric, either because oil tends to remain supersaturated with gas or because oil with gas in solution enters the pump under pressure due to a fluid level much higher than that of the pump, the gas will remain in solution until the oil is lifted to a point where the hydrostatic pressure of the oil above is less than the pressure at which the gas is held in solution. Above this point the movement of the sucker rods usually agitates the oil enough to cause liberation of the gas from solution in the oil, which in turn flows or at least assists in flowing the oil from the tubing. The action is usually progressive, and as a certain amount of oil is flowed from the tubing it lowers the pressure on the remaining oil and liberates more gas, thus causing additional oil to flow from the tubing. This procedure often continues until the tubing is emptied to a depth of 700 to 1,000 feet or more. A well that operates in this manner is said to be "agitating." The pressure chart of well 23A in Figure 17, A, shows periods during which the oil flowed by agitation from a pumping well. During the intervals in which no pressure is indicated the oil was entering the tubing, and where pressures are shown on the chart the oil was flowing from the tubing.

Frequently, the oil-well pumps are operated at such speeds that they remove the oil more rapidly than it can enter the wells and gas is pumped into the tubing, resulting in additional agitation, liberation of gas, and flowing of oil from the tubing. This "agitation" or flowing by heads causes alternate coating of the walls of the pipe and presents an ideal condition for the accumulation of paraffin.

The accumulation of paraffin in pumping wells can be prevented by heating the oil and oil string or by eliminating conditions that cause the paraffin to become attached to the pipe. Pumping wells are much easier to control and produce so as to prevent paraffin troubles than flowing wells. In pumping wells the temperature of the oil, the amount of liquid constituents lost by evaporation, and consequently the amount of paraffin that will be precipitated from solution in the oil while it is being pumped from the well, can not be altered so easily by operation or change of equipment as in flowing wells, but the flowing by heads or agitation that causes the paraffin to become attached to the pipe can largely be prevented.

It has been pointed out that to decrease the loss of heat to the surrounding formations the velocity of the oil should be as high as practical. High flowing velocities can be maintained by the use of the smallest tubing practical, considering the quantity of oil to be lifted and the pumping equipment to be used. For example, the velocity of the oil will be approximately two and one-half times as fast through 2-inch as through 3-inch tubing. Calculations showing the reduction in the loss of heat by radiation by increasing the velocity through the use of smaller pipe have been given previously. (See p. 33.)

Eliminating agitation or producing by heads, which causes the wax to become attached to the pipe, is of the greatest importance in stopping or greatly modifying the accumulation of paraffin in pumping wells. Even though the oil is above the temperature at which it is saturated with wax and contains no free wax in suspension when it flows from the well, the remaining film of oil may be cooled by the formations surrounding the oil string to the point where wax is precipitated out of solution in the oil and becomes firmly attached to the pipe. The accumulation is much more rapid, however, when the oil contains free wax in suspension.

The methods that the writer found particularly satisfactory for preventing the accumulation of paraffin by agitation or flowing by heads are the application of back pressure to the tubing at the top of the well and the use of gas anchors. The application of back pressure on the tubing increases the pressure on the oil in the tubing, and when this pressure is greater than that at which the oil is saturated the gas is prevented from coming out of solution. The pressure is only exerted upon the oil in the tubing and has no effect upon conditions at the bottom of the well. Back pressure is particularly satisfactory in wells where a pressure of 50 or 60 pounds per square inch will prevent the gas from being liberated from the oil, although it is often advisable to use this method even where much higher pressures are necessary. A pressure of 50 or 60 pounds per square inch is roughly equivalent to pumping 39° A. P. I. gravity oil an additional 140 to 160 feet, and the increased strain on the pumping equipment and the work required to pump the oil are usually negligible. Higher back pressures of course require more power and efficient stuffing boxes around the polish rods.

In several pumping wells that were producing by heads in the Salt Creek field paraffin accumulated so rapidly that it was necessary to pull the rods at intervals of 10 to 14 days. After these wells were equipped with pressure regulators and sufficient back pressure was held on the tubing to prevent "agitating" the trouble from paraffin was eliminated. The detailed results obtained with well 23A, NE. 1/4 sec. 25, T. 40 N., R. 79 W., are given in Table 9. (Fig. 16 shows the manner in which the recording temperature and pressure gages were installed.) The production and the amounts of paraffin which accumulated in the well before and during the test are given in Table 9. Figure 17, A, is a typical gage chart showing the pressure at the tubing head before back pressure was applied. Although the pump was operating continuously, the chart shows that the well actually flowed its production at intervals and did not produce any oil between flowing periods. After a back pressure of 33 pounds per

square inch was applied, the well produced steadily during the entire 24 hours. Figure 17, *B*, is a 24-hour pressure chart of the well after back pressure had been applied. The temperature of the oil was approximately 60° F. before the back pressure was applied and 65° F. afterwards. The oil contained considerable free wax in suspension even after the back pressure was applied, but conditions were unfavorable for it to accumulate inside the pipe. Paraffin accumulated at the rate of approximately 6 pounds per day before the back pressure was applied (see Table 9), and after the application of back pressure stopped agitation in the well the amount of accumulated paraffin was almost negligible.

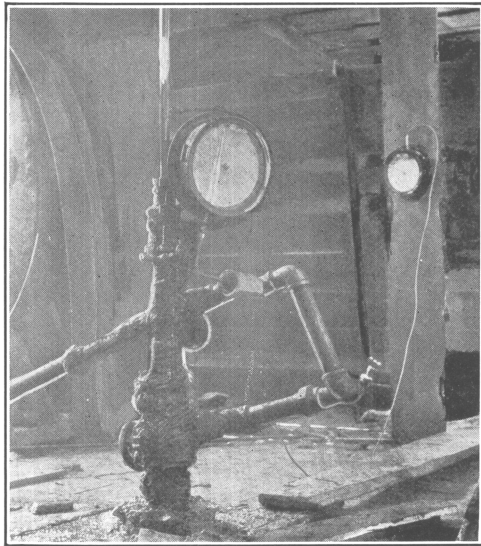


FIGURE 16.—Well equipped with pressure and temperature gages for studying effect of different methods of production on accumulation of paraffin

TABLE 9.—Production record of test well 23A, NE. ¼ sec. 25, T. 40 N., R. 79 W., Salt Creek, Wyo., before and after application of back pressure to tubing

Date	Production, barrels	Remarks	Date	Production, barrels	Remarks	
Apr. 16	31	Rods pulled and paraffin hook used—125 pounds paraffin removed.	July 7		Rods pulled and paraffin hook used—77 pounds paraffin removed.	
17			8	37		
18	39			22		Rods pulled and paraffin hook used—114 pounds paraffin removed.
23	35					
30	36					
May 1		Rods pulled and paraffin hook used—124 pounds paraffin removed.	31		Rods pulled and paraffin hook used—88 pounds paraffin removed.	
2	48	Rods pulled and paraffin hook used—60 pounds paraffin removed. Vacuum 20 inches for 8 days—9 days without vacuum.	Aug. 1	35	Back pressure 33 pounds on tubing head. Stroke shortened.	
5	36		5	33		
10	37		8	27		
15	34		10	32		
18	36		15	28		
			20	35		
19	32	25	34			
25	37		29		Rods pulled and paraffin hook used—10 pounds paraffin removed.	
30	34					
June 1	32	Rods pulled and paraffin hook used—114 pounds paraffin removed.	Sept. 2	32	No production record from Sept. 5 to Sept. 16.	
2			5			
5	31		15		Rods pulled—no appreciable quantity of paraffin found.	
12	27		16		Tubing pulled and gas anchor installed, also connected casing head to gas gathering system. No paraffin trouble since that time.	
17	28					
		Rods pulled and paraffin hook used—16 pounds paraffin removed.				

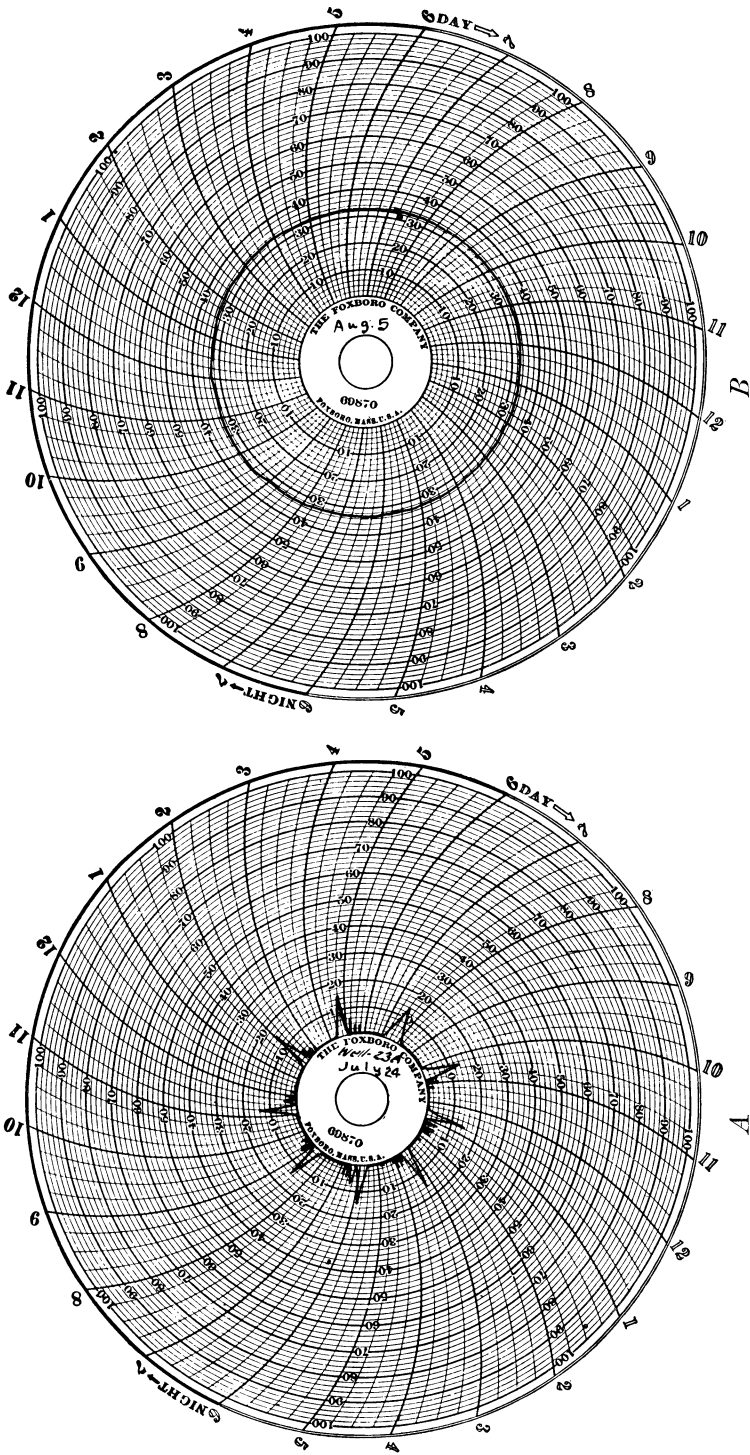


FIGURE 17.—A. Pressure-gage chart from well 23A without back pressure applied to tubing; B, pressure-gage chart from well 23A with back pressure on tubing

A number of wells in the Salt Creek field were equipped with 75-pound-per-square-inch pressure-relief valves placed on the flow lines at the tanks, and this hook-up successfully prevented the accumulation of paraffin in the tubing of the wells and in the flow lines.<sup>29</sup> Application of back pressure by means of pressure-relief valves on the flow lines at the tanks is satisfactory for preventing the accumulation of paraffin in pumping wells that agitate or flow by heads and can be used whenever the required back pressure is not too great to cause an excessive load on the equipment. The main disadvantages are the cost and upkeep of the pressure regulators and stuffing boxes and the additional load on pumping equipment.

The correct use of a properly designed gas anchor will, in many instances, prevent agitation or flowing by heads by removing the gas held in the oil as a supersaturated solution. The two types of gas anchors that have proved particularly satisfactory for this purpose are shown in Figure 18. The function of the gas anchor is to cause enough movement or agitation of the oil to liberate the free gas associated with the oil and any gas held in it as a supersaturated solution. Any type of gas anchor that will separate the gas from the oil is satisfactory. To obtain satisfactory results through the use of gas anchors they must be located at a point in the well where

the oil inlet is approximately at the fluid level, and the fluid inlet to the anchor should never be submerged below the normal fluid level if satisfactory removal of the gas is to be expected.

Gas anchors have no value in wells where the fluid level is maintained above them because when thus submerged the oil enters the gas anchor saturated with gas under the hydrostatic pressure and the gas is liberated from the oil in the upper part of the tubing,

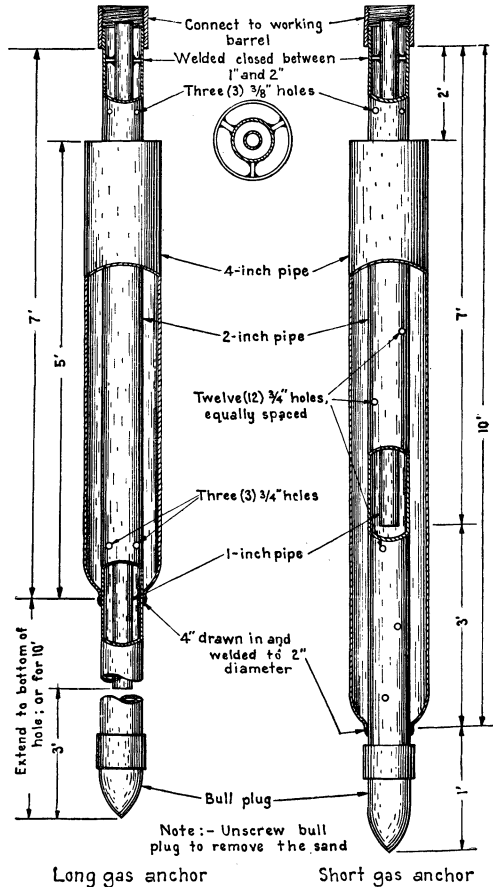


FIGURE 18.—Gas anchors used at Salt Creek, Wyo.

<sup>29</sup> Wood, F. E., Young, H. W., and Buell, A. W., Handling Oils and Paraffin in Salt Creek Field: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., New York, 1929, p. 265.

causing the well to flow by heads. Gas anchors also have little or no value in wells where a high back pressure is held on the sand or where the gas produced with the oil is not removed from the well between the tubing and the casing. The use of gas anchors is therefore limited to wells where the fluid level is maintained fairly constant and wells in which very little or no pressure is maintained on the sand by holding pressure at the casing head. Many of the pumping wells in Salt Creek that paraffined up rapidly were equipped with gas anchors, and in those wells in which "agitation" was prevented the paraffin trouble was eliminated.

TABLE 10.—*Production record of test well 25A, NW. ¼ sec. 2, T. 39 N., R. 79 W., Salt Creek, Wyo., before and after using gas anchor*

Date	Production, barrels	Remarks	Date	Production, barrels	Remarks
Jan. 5	10	Well cleaned out.	Apr. 6	-----	Rods pulled and parffin removed.
17	10		7	41	
20	-----		11	-----	Rods and tubing pulled and paraffin removed; gas anchor installed.
24	50				
31	41				
Feb. 6	66	Rods pulled and paraffin removed.	Apr. 12	50	
14	41		20	60	
17	37		26	44	
23	26		28	-----	Rods pulled—no paraffin—new cups used.
Mar. 2	37	Rods pulled to remove paraffin.	May 4	44	Do.
3	-----				
8	63				
15	63				
16	-----				
		Rods pulled to remove paraffin and replace cups.			
22	40				
28	-----				
31	-----				
Apr. 1	76	Rods pulled for paraffin. Rods pulled to replace cups.	June 7	42	
			14	40	

The production and clean-out record of well 25A, NW. ¼ sec. 2, T. 39 N., R. 79 W. (Table 10), is typical of the results obtained with gas anchors. In this well the gas anchor successfully removed enough gas from solution in the oil at the bottom of the well that it pumped steadily and had no tendency to agitate or flow by heads. When the oil reached the surface it contained an appreciable quantity of free paraffin that had separated from solution, but conditions in the well were unfavorable for paraffin to become attached to the pipe, and it therefore remained suspended in the oil.

In some wells gas anchors remove considerable of the gas in the crude oil but not enough to completely stop the well from agitating or pumping by heads. In such wells it is necessary to use a pressure regulator and maintain a back pressure on the tubing; however, the removal of part of the gas from the oil by use of a gas anchor decreases the amount of back pressure necessary on the tubing, as part of the dissolved gas, particularly that held in a supersaturated state of solution, is removed from the oil at the bottom of the well.

Back pressure or gas anchors, when properly used, will practically prevent the accumulation of paraffin in pumping wells where agitation or flowing by heads is responsible for the accumulation of paraffin. Nevertheless, in some wells enough paraffin will accumulate on the rods and tubing (in spite of all precautions to prevent it),

due to excessive cooling or other conditions, to warrant its removal from the tubing at long intervals.

In the Salt Creek field, Wyoming, and the Panhandle field, Texas,<sup>30</sup> wire lines with several stands of steel sucker rods on the bottom to furnish enough weight to keep the line taut are sometimes used in place of steel sucker rods. Wire lines vibrate sufficiently to cut the paraffin from the pipe and prevent it from accumulating; however, the use of wire lines as substitutes for sucker rods is not entirely satisfactory. Considerable trouble is often encountered when "wickers" from the worn line collect in the pump and cut the valves and cups. Furthermore, wire lines also cut the tubing more rapidly than rods.

In many wells rod guides are used as scrapers to remove the paraffin from the walls of the pipe, but they have proved unsatisfactory because the paraffin is only removed from the pipe by each guide for a distance equal to the length of the pumping stroke. In the Panhandle district, Texas, washers are sometimes welded to the sucker rods, but the practice is not satisfactory because welding apparently weakens the rods and causes excessive rod failures. The casting of babbitt buttons<sup>31</sup> on the rods proved more satisfactory than welding washers to the rods and prevented the accumulation of paraffin when the buttons were placed close enough together to scrape the entire inner surface of the tubing.

#### PUMPING WELLS IN SETTLED PERIOD

Settled pumping wells are wells that produce oil from sands in which the rock pressure has declined to a point where all or almost all of the original gas energy has been exhausted and the production of oil is small but fairly constant. Such wells generally produce 5 to 30 barrels of oil a day, and many of the small producers are pumped only a portion of the 24 hours. The oil contains insufficient gas in solution to flow by heads from the tubing as it nears the surface; therefore, it flows steadily from the tubing with each stroke of the pump.

The accumulation of paraffin in settled wells is normally negligible because of conditions within the well and because of the small amount of oil produced furnishing only a small amount of paraffin available for deposition. Generally, the movement of oil in the tubing of settled pumping wells is slow, and the temperature of the oil at the top of the tubing is therefore lower than in larger pumping wells. The temperature usually approaches the temperature of the coolest earth formations surrounding the well. Although the cooling effect of the formations above the oil sand results in separation of wax from solution the wax normally remains suspended in the oil and gives little or no trouble in the tubing.

Considerable trouble from accumulations of paraffin occurs in wells that are pumped part of the time only because the valves in almost all oil-well pumps leak sufficiently, after being in service for a time, to allow the oil to drain from the tubing while the well is not pump-

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<sup>30</sup> Vietti, W. V., and Oberlin, W. A., *Handling Congealing Oil and Paraffin: Petrol. Devel. and Technol.*, Am. Inst. Min. and Met. Eng., New York, 1927, pp. 269-284.

<sup>31</sup> Smith, L. E., "Buttons" on Sucker Rods Keep Tubing Free from Paraffin: *Nat. Petrol. News*, vol. 20, No. 50, Dec. 12, 1928, p. 55.



ing. This leakage results in alternate coating and draining of the pipe and rods with oil and causes paraffin to accumulate on the walls of the tubing and rods. The greater the number of intervals that the well is pumped per day the more rapid the accumulation of paraffin, unless the pumping equipment in the well is in good mechanical condition and prevents the tubing from draining during the interval that the well is shut down.

There is always a tendency for a film of oil in contact with the pipe to congeal or become viscous when the pipe is cooler than the oil, especially when the temperature of the pipe is below or near the pour-point of the oil. The thickness of the film of oil and the quantity of wax that will separate from solution depend upon the temperature of the pipe and the velocity of the oil. When the flow of oil is slow and the temperature of the pipe is below the saturation temperature of oil and wax a thick viscous film of slow-moving oil is in contact with the pipe, and some of the wax in the film becomes attached to it. The oil cools, and while it is standing in the pipe wax separates from solution. When movement again takes place the entire mass moves, leaving perhaps a small amount of wax attached to the pipe. When the oil is flowing rapidly through the pipe its tendency to form a film in contact with the pipe is greatly reduced or perhaps eliminated entirely, and less paraffin becomes attached to the pipe. Regardless of the thickness of the oil film and the velocity of flow of oil in the tubing, however, a small amount of paraffin is always deposited on the rods and tubing due to suspended wax particles coming into contact with these surfaces with sufficient force to adhere. The amount of paraffin that accumulates due to these and possibly some unobserved conditions is usually so small in settled pumping wells that it seldom has to be removed except when it is necessary to pull the rods for other causes.

#### CAUSES AND PREVENTION OF PARAFFIN DEPOSITION ON FACE OF PRODUCING SAND

The accumulation of paraffin on the face of the producing sand decreases the available surface from which the oil can flow into the well <sup>32</sup> and gradually decreases the production of oil. This decrease may be mistaken for the normal decline of the well. This problem is a serious one because it often occurs in old wells of small production, in which it is often doubtful whether the increased production to be obtained by removing the paraffin will justify the expense incurred. It is sometimes argued that the accumulation of paraffin on the upper part of the sand is advantageous, when gas is being injected into the sand, because it tends to prevent by-passing of the oil and escape of the gas into the well. It is questionable, however, if such a haphazard method of preventing by-passing of the gas is advisable, and it is probable that more damage is done by the paraffin in preventing oil from entering the well than good is accomplished by preventing the escape of excessive quantities of gas. Although it is impossible to know definitely the exact conditions at the bottom of a well and the manner in which paraffin is deposited certain logical

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<sup>32</sup> Mills, R. van A., *The Paraffin Problem in Oil Wells: Rept. of Investigations 2550*, Bureau of Mines, December, 1923, 11 pp.

conclusions can be drawn from data obtained from laboratory and field studies of the factors affecting the solubility and deposition of paraffin.

It is obvious that for paraffin to accumulate on the face of the sand the oil must be either cooled or subject to evaporation, as laboratory and field experiments have proved that these are the two main factors controlling the solubility of paraffin in oil. Conditions that favor the accumulation of free paraffin on the face of the sand must also exist in the well. That paraffin does accumulate on the face of the sand and in the sand adjacent to the well is definitely known because the pore spaces in pieces of sand and sand cores removed from different wells have been plugged with paraffin. The increased production obtained when methods for removing paraffin have been applied to certain wells demonstrates further that paraffin accumulates in the sand about certain wells.

The temperature of the sand and the character of the oil before it enters the well during the earlier life of a field is generally such that it is not saturated with wax, and unless conditions exist which produce sufficient cooling and evaporation of the oil so that it is saturated with wax the oil will hold all of its wax in solution and have the ability to dissolve additional wax at the sand temperature. As a field becomes older the lighter constituents are constantly being removed from the oil in the sand, and the oil becomes more nearly saturated with wax before it leaves the sand. Field investigations have shown that paraffin will accumulate on the face of the sand if the sand is exposed by maintaining the fluid level at or near the bottom of it. When the sand is exposed in small wells the oil drains down over the face of the sand, and conditions for evaporation by the gas produced with the oil are ideal. The accumulation of paraffin on the face of the sand is more rapid when the film of oil flowing over the sand is thin enough so that the precipitated particles of wax are not easily carried in suspension. Furthermore, the surface of the sand will be cooled by evaporation and by the gas expanding through the pore openings in the sand. Lowering the fluid level below the top of the sand in older fields gradually depresses the oil surface in the sand near the well and reduces the amount of oil flowing from the upper part of the sand. The amount of gas flowing through the upper part of the sand is increased, and there is more cooling and evaporation of the oil remaining in or flowing from that part of the sand. Figure 19, taken from Uren's article on fluid level<sup>33</sup> in settled pumping wells, illustrates the theoretical effect of different fluid levels in the well upon the oil content of the sand adjacent to the well.

The alternate covering of the sand and evaporation of the oil when wells are pumped intermittently and the lowering of the fluid level to the bottom of the sand during the pumping period may or may not cause paraffin to accumulate on the face of the sand, depending primarily on the nature of the oil. If the oil entering the well is not saturated with wax at the well temperature, paraffin will not build up on the sand face even if the oil flowing from the top of the sand is evaporated and cooled sufficiently to deposit some paraffin on the

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<sup>33</sup> Uren, L. C., Significance of Fluid Level in Oil-Well Pumping: *Trans. Am. Inst. Min. and Met. Eng.*, vol. 71, 1925, p. 1301.

upper part of the sand when alternately covered and exposed, because the temperature of the oil as it accumulates in the well is raised to approximately the sand temperature and when the fluid covers the face of the sand while the well is not operating the warmed oil redissolves the precipitated paraffin. As a field becomes older the oil in the sand gradually approaches the saturation point in wax content, and it therefore has less ability to dissolve any paraffin which is deposited when the face of the sand is exposed. In

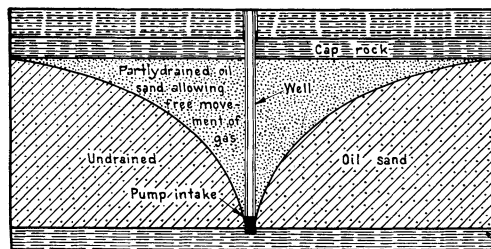
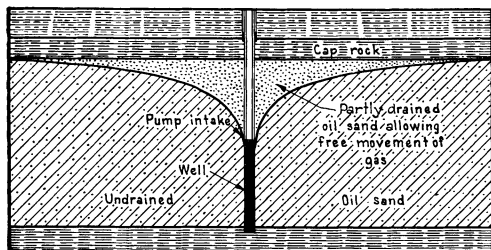
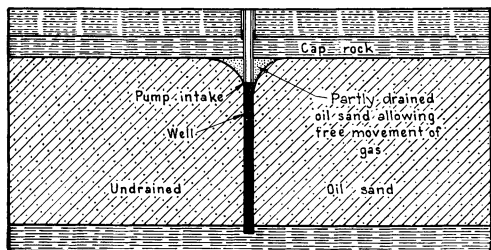


FIGURE 19.—Hypothetical effect of different fluid levels on saturation of sand adjacent to the well, after Uren

intermittently pumped wells it is advisable to keep the sand covered at all times if the accumulation of paraffin is to be prevented. As an example of such a condition J. T. Montgomery, of the Marion Oil Co., told the author of a number of wells he was operating near Core, W. Va., in which the fluid level had been alternately raised and lowered, due to intermittent pumping, until he felt sure that part of the sand was paraffined. He washed these wells by circulating gasoline and raised the pumps so that the fluid level was maintained near the top of the sand. Within a short time the production of oil, which had decreased immediately after the fluid level was raised, was more than it had been for several years. In some parts of the Burburnett and Electra fields, Texas, the sand face was also found to be paraffined up due to alternate exposing of the sand and application of a high vacuum. Similar conditions are found in many old fields.

The use of vacuum increases evaporation of the oil in the sand adjacent to the well, particularly in the partly drained portion, and increases that from the film of oil draining down over the face of the sand when low fluid levels are maintained. Lindsly and Berwald<sup>34</sup> cite an example of the accumulation of paraffin on the sand and the loss of production in the Shaffer Run pool, Pennsylvania, as typical of the effect of vacuum in certain wells. The loss of volatile con-

<sup>34</sup> Lindsly, B. E., and Berwald, W. B., Effect of Vacuum on Oil Wells: Bull. 322, Bureau of Mines, 1930, pp. 24-25.

stituents due to the use of vacuum in the Red River area of Oklahoma is also worthy of mention. The pumping wells in the Red River area were operated with a vacuum of 20 inches of mercury on the casing head, when all the operators agreed to lower the vacuum. It was gradually decreased to a vacuum of 15 inches of mercury without any appreciable loss in production; at the same time the gravity of the oil was increased from 39° to 42° A. P. I., indicating that the evaporation from 42° to 39° A. P. I. was taking place in the well or near the face of the sand, for if the gravity of all the oil in the sand had been lowered by the use of vacuum there would be no increase in gravity of the oil when the vacuum was decreased. The application of vacuum to sands in which the oil contains appreciable quantities of paraffin undoubtedly favors the separation of wax from solution in the oil and its deposition in the sand adjacent to the well.

In many wells the face of the sand is often plugged with paraffin that has been forced down the tubing or casing while paraffin accumulations were being removed with tools. It was not uncommon to remove 500 to 1,000 pounds of paraffin from the casing of flowing wells in Salt Creek field. Unless the proper tools and care are used, the paraffin that has been scraped off will be forced down to the bottom of the well and will plug the sand. The improper application of heat or chemicals for cleaning tubing and casing will often cause the melted paraffin to run down and coat the face of the sand if it is not covered with oil. When the sand is covered with oil any melted paraffin will remain suspended or dissolved in the oil and cause no damage.

The accumulation of paraffin on the face of the sand can best be prevented by keeping the fluid level at or near the top of the sand. In maintaining the fluid level at the top of the sand the production may be decreased in some wells; but unless a material loss in production occurs it is advisable to follow this practice as insurance against paraffin troubles, especially in wells where the exposed face of the sand paraffins up rapidly. In no well where there is danger of paraffin accumulation should the fluid level be lower than half the thickness of the sand, because additional lowering will seldom increase the production.<sup>35</sup> When the fluid level is maintained so that the sand is covered the oil flows directly from the sand into a body of oil in the well, greatly modifying the degree of cooling and evaporation of the oil, because it is not exposed as a thin film over a large area.

Elimination of the film of oil greatly reduces the possibility of an accumulation of paraffin on the sand. Fluid levels can be maintained at the top of the sand or at any other point by placing the intake to the pumps at the desired level. However, it is often desirable to pump the liquid from the bottom of the well to remove any water that might accumulate, and this can be accomplished by the use of a fluid-level regulator or flood nipple. A satisfactory flood nipple can be made by drilling one or more small holes, depending upon the length of stroke and speed of the pump, in a nipple attached to the bottom of the working barrel. This nipple should be placed

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<sup>35</sup> Uren, L. C., Significance of Fluid Level in Oil Well Pumping: Trans. Am. Inst. Min. and Met. Eng., vol. 71, 1925, p. 1301.

at the desired fluid level with sufficient pipe attached to reach approximately to the bottom of the well. In operation insufficient liquid can enter through the small holes in the nipple to supply the pump, and the additional liquid will be taken in from the bottom of the well. When the fluid level is reduced below the openings enough gas enters to destroy the suction lift of the pump on the liquid in the pipe and stop the removal of liquid. It is undoubtedly better practice in many wells to pump wells continuously, using a stroke and speed only slightly greater than those required to lift all the available oil, with the intake or fluid-level regulator placed so that the fluid level can not be lowered below the top of the sand. By maintaining the fluid level and pressure on the sand constant the maximum amount of oil should be recovered with a minimum amount of paraffin and other production troubles.

#### CAUSES AND PREVENTION OF PARAFFIN ACCUMULATION IN SAND

The accumulation of paraffin in the sand prevents the movement of oil toward the well and may completely shut off the flow of oil through part of the sand. This type of paraffin accumulation is difficult to detect, and as in accumulations of paraffin on the face of the sand the resulting decrease in oil production is often mistaken for natural decline of the well. This results in a smaller total recovery of oil and leaves the sand partly or completely plugged so that the oil can not later be removed by pressure-restoration or gas-drive methods without first opening up the passageways in the sand by using heat or other methods of liquefying or dissolving the paraffin.

The accumulation of paraffin in the sand is the result of the cooling effect caused by the expansion of the gas associated with the oil and the evaporation of some of the lighter constituents of the oil. There is also the possible cooling of the sand as a result of large quantities of cold water forced into the sand or entering the sand through faulty well casing; however, any material decrease in temperature is exceedingly doubtful, due to radiation of heat from the surrounding formations, with the possible exception of the sand close to the point where the water enters.

The oil in the reservoir sands is known to lose gradually some of its lighter constituents in addition to losing some of the gas held in solution. For example, the decrease in gravity of the oil in the Salt Creek field, Wyoming, from January, 1926, to October, 1929, was as follows: Weighed gravity of total production January, 1926, 37.8° A. P. I.; January, 1927, 37.69° A. P. I.; January, 1929, 36.71° A. P. I.; and October, 1929, 36.44° A. P. I. In addition to the loss in gravity the initial boiling point of the oil increased from 98° to 103° in 1922, to 116° F. in 1924, and to 142° to 144° F. in 1926. These facts show conclusively that there is a gradual loss of volatile constituents from the oil remaining in the sand, even in a field where no vacuum has been used. It is doubtful, however, if there will be sufficient evaporation of the main body of oil in the saturated part of the sand under natural conditions to cause the separation of wax from solution in the oil during the economic life of the field. It is

possible, however, that enough evaporation will take place in the partly drained area in the upper part of the sand adjacent to the well, and particularly where the fluid level is maintained below the top of the sand, to cause separation of wax, coagulation of the oil, and plugging of the sand pores.

The cooling of the sand by expanding gas moving the oil to the well—a condition that becomes pronounced close to the well—will have an increasing effect on the deposition of paraffin as the oil loses its lighter constituents. The expansion of gas through the partly drained area of the sand probably causes greater cooling and evaporation of the oil than in the undrained area, and there is more of a tendency for the partly drained part of the sand to become plugged with paraffin.

The evaporation of the lighter constituents is increased when vacuum is applied to a sand. Vacuum also produces more cooling and more favorable conditions for the precipitation of paraffin; however, vacuum has an appreciable effect on the oil in the sand only in wells where the sand is exposed, and the effect extends back an appreciable distance from the well only in very porous sands.

The injection of dry gas into the sand may also cool the oil and cause evaporation of the lighter constituents of the oil adjacent to the gas-intake well. The possibility of creating very low temperatures in the sand adjacent to wells in which gas at high pressure is being injected is discussed by Osgood,<sup>36</sup> who estimates the possibility of temperatures as low as  $-84^{\circ}$  F. being produced about injection wells. The author has observed that frequently during the injection of high-pressure gas into a new intake well in the Salt Creek field the well took large volumes of gas for a short period, followed by a rapid decline in the amount of injected gas, after which the volume injected gradually increased again. In some wells, however, the volume did not increase appreciably after once declining.

As pointed out by Osgood, a possible explanation for the behavior of the wells is that the injection of gas produces a low temperature in the sand adjacent to the well and causes congealing of the oil and separation of paraffin. When the flow of gas is reduced by this congealed oil and precipitated paraffin in the sand, the cooling produced by the expansion of gas is greatly diminished and the sand is then warmed due to radiation of heat from the surrounding formations. The oil then becomes fluid, again dissolves the paraffin, and again permits the gas to enter the sand rapidly. If the oil is forced back into the sand before the cooling is sufficient again to congeal it or cause the precipitation of paraffin, the sand will continue to take large volumes of gas because the greatest expansion of gas and cooling, when gas is injected into the sand, occurs close to the face of the sand. When enough gas can enter the sand through unplugged pore spaces to maintain the temperature of the sand below the congealing point of the oil by the expansion of the gas, the congealed oil very probably will remain congealed and will plug a portion of the sand. In some wells paraffin may be precipitated from solution in the oil by the cooling, plug part of the pores of the sand,

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<sup>36</sup> Osgood, W. H., Some Subsurface Problems of Applied Thermodynamics: Oil Weekly, Aug. 22, 1927, p. 60.

and cause the liquid constituents to flow away from the plugged portion of the sand. In wells where this occurs the paraffin will not redissolve, even if the sand temperature is raised to normal.

The prevention of paraffin deposition by proper production methods resolves itself into a problem of preventing excessive evaporation and cooling of the oil in the sand, which can best be accomplished by maintaining the fluid level in the well near the top of the sand so that there will be a minimum of drained or partly drained sand exposed to the well through which the gas from the oil-saturated portion of the sand can escape. Escape of gas from partly drained sands results in excessive cooling of the oil and sand and evaporation of the oil in the partly saturated portion of the sand. The gas also fails to force or carry its full quota of oil into the well. Casing or packing off the upper part of the sand and correct use of back pressure will prevent the escape of gas through the partly drained portion of the sand and, instead, will cause the gas to carry a maximum amount of oil into the well, thus decreasing the evaporation and cooling of the remaining oil in the sand and the danger from paraffin.

When gas is injected into the sand to restore the reservoir pressure or to increase production by gas-drive methods, the congealing of oil adjacent to the intake well can be prevented by injecting the gas at low pressure and gradually increasing the pressure as the oil is forced out of the sand adjacent to the well where the major cooling undoubtedly takes place. The injection of butane or light hydrocarbon liquids to wash the oil back from the sand about the well is also often advisable. Furthermore, it is often good practice to use high fluid levels or back pressure, or to shut off the upper part of the sand to prevent by-passing of the injected gas and excessive evaporation of the oil in the partly drained area of the sand. In fields where the oil that remains in the sand is saturated or nearly saturated with wax the gas or air should be returned to the sand without removing the gasoline vapors. If it is not practical or economical to return "wet" gas to the reservoir sand, at least all or a portion of the more volatile constituents that have only a limited market value should be returned.<sup>37</sup> In making gasoline from natural gas it is generally necessary to fractionate or "weather" a large quantity of the more volatile constituents from the crude natural gasoline to obtain a satisfactory product.

During 1926 in the Salt Creek field approximately 112,800 gallons of gasoline were extracted daily from the gas produced with the oil; and 7,200,000 cubic feet of gas per day, consisting chiefly of propane, butane, and pentane, with some higher and lower constituents, was weathered from the gasoline after its extraction from the gas and burned in a flambeau. The weathering-plant gas had no commercial value at that time, and it was generally thought that it would be difficult and impractical to return it to the sand. Recently, however, the engineers of the major company operating in the field made a detailed study of the problem of utilizing the waste weathering-plant gas and developed a satisfactory method of putting it back into the partly depleted sand. During 1929 approximately 30,000,000 cubic

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<sup>37</sup> National Petroleum News, Butane from Gasoline Plant Used in Repressuring: Vol. 20, No. 24, June 13, 1928, p. 68.

feet of gas, of which approximately 4,000,000 cubic feet was from the stabilizers, was returned to the sand daily. When the fractions that

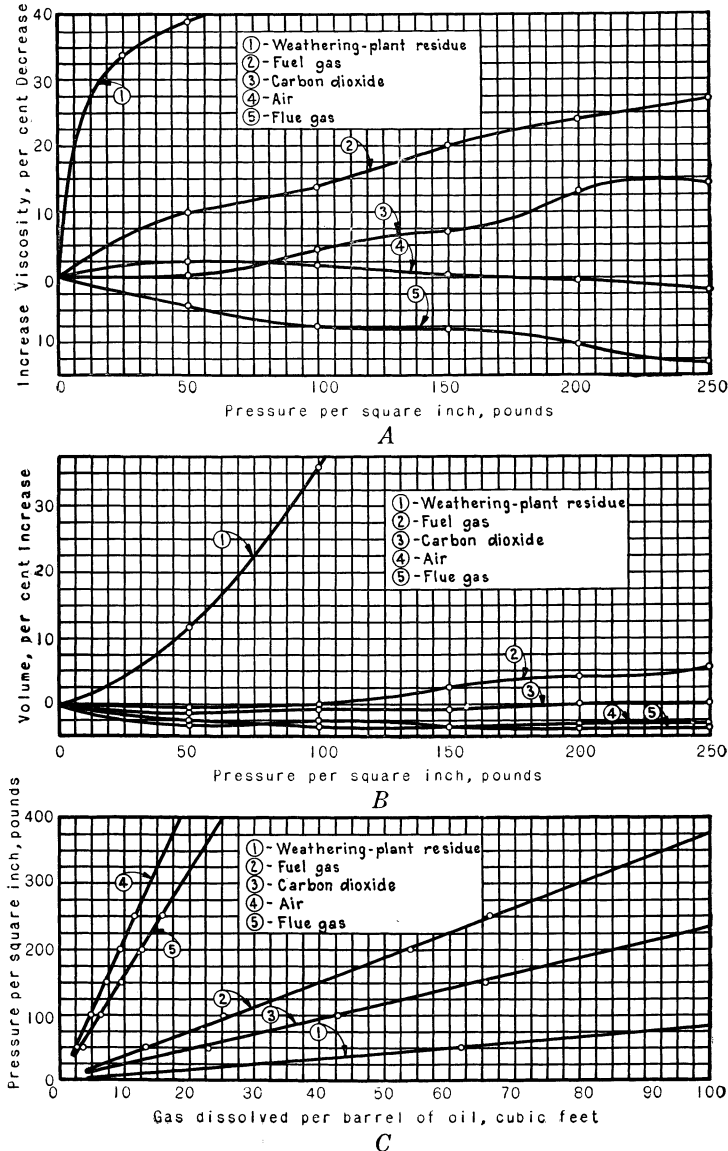


FIGURE 20.—Solubility of weathering-plant gas and other gases in crude oil and their effect on volume and viscosity of the oil, by Buell: *A*, Effect of saturation with gases under various pressures on viscosity of Salt Creek crude oil, 36° A. P. I., temperature 100° F.; *B*, effect of saturation with gases under various pressures on volume of Salt Creek crude oil, 36° A. P. I., temperature 100° F.; *C*, solubility of gases in Salt Creek crude oil at various pressures. All volumes corrected to 60° F. and 15.025 pounds pressure

are weathered from natural gasoline are mixed with crude oil they lower the viscosity and surface tension of the oil and appreciably increase its solvent power for paraffin. By returning weathering-



plant gas to the sand the operator can prevent the waste of the lighter fractions of the natural gasoline, reduce the probability of paraffin being deposited in the sand, and lower the viscosity and surface tension of the oil so that it will flow through the sand more rapidly and leave less oil on the surface of the sand grains. The solubility of gas from the stabilization-tower and other gases and their effect on the volume and viscosity of Salt Creek crude, as determined by A. W. Buell of the Midwest Refining Co., are shown by curves in Figure 20.

#### **METHODS FOR PREVENTING AND REMOVING ACCUMULATIONS OF PARAFFIN BY OTHER THAN CONTROL OF OPERATING CONDITIONS**

Where it is impractical or impossible to control the factors that cause paraffin to accumulate in the well and in the reservoir sand other means of preventing or removing the paraffin must be used. Operators should select a method that will satisfactorily accomplish the desired results with a minimum of expense and loss of production. The selection of the method should therefore be governed primarily by the results to be accomplished and the possibility of using available or easily constructed equipment.

The methods generally used to remove accumulations of paraffin are: (1) Those that remove the paraffin by mechanical equipment and the use of scrapers, explosives, and compressed air; (2) those that remove the paraffin by the use of solvents that dissolve the paraffin; and (3) those that use heat, which melts the paraffin and reduces it to a liquid so that it can easily be removed with the oil. The methods are not all equally applicable or satisfactory, and each has its own special advantages and disadvantages. In some wells a combination of two or more of the methods gives the most satisfactory results.

#### **MECHANICAL METHODS FOR REMOVING PARAFFIN FROM WELLS**

Mechanical methods offer an economical and satisfactory means for removing paraffin that has accumulated in the oil string, due either to careless operation or unavoidable conditions. After paraffin is removed with scrapers or similar tools an increase in production should not be expected, unless the accumulation of paraffin reached the point where it materially restricted the flow of oil in the oil string. The removal of paraffin does, however, insure the operator against paraffin troubles and loss of production for a period of time.

#### **STANDARD TOOLS**

The regular clean-out string of standard tools, consisting of socket, jars, stem, and bit, can be used advantageously for removing paraffin from the casing of flowing wells, but it is not advisable to use these tools except when a high formation pressure is available. The general procedure in cleaning a string of casing in which paraffin has accumulated is to run the tools far enough into the casing to cause the formation of a plug of paraffin. The tools are then withdrawn, and the formation pressure is allowed to build up and blow the paraffin plug from the well. Figure 21 shows a plug of paraffin

being blown from a well in the Salt Creek field. The formation pressure at this well was approximately 250 pounds per square inch. Care should be taken not to form so large a plug of paraffin that it can not be blown from the well by the available pressure, but if small plugs are formed and the method is repeated a number of times good results can be expected. If too large a plug is accidentally formed and the pressure is insufficient to blow the plug from the well it then becomes necessary either to drill through the paraffin or to follow the more satisfactory method of using a paraffin hook or other special tools. Sometimes it is inadvisable to drill through the paraffin because drilling often forces some of the paraffin down to the bottom of the well where there is danger of it being forced into the pores of the sand and clogging the fluid passageways.

#### PARAFFIN HOOK

The use of a paraffin hook is a positive method for removing large quantities of accumulated paraffin from the casing or tubing of flowing wells, and it is particularly applicable for removing paraffin from the tubing mechanically pumped wells. Figure 22 shows the design and construction of paraffin hooks for use on standard tools and sucker rods. In removing paraffin from the casing of wells the hook is usually run in place of the bit on a standard string of tools. Care should be taken not to run the hook so far into the paraffin that it will be difficult to pull out the paraffin. When paraffin is removed from tubing the hook is usually run on a  $\frac{1}{2}$  or  $\frac{5}{8}$  inch wire line, using a sinker bar to force it through the paraffin. In pumping wells the rods are pulled and the hook run on  $\frac{1}{2}$  or  $\frac{5}{8}$  inch wire line or on the rods.



FIGURE 21.—Paraffin being blown from well after forming bridge with standard tools

#### PARAFFIN KNIFE OR FLOW DEVIL

The paraffin knife<sup>38</sup> or flow devil<sup>39</sup> is particularly adaptable for removing paraffin from the oil string of natural flowing and air or gas lift wells. In fact, it is more satisfactory for this work than

<sup>38</sup> Bennett, E. O., and Sclater, K. C., Some New Aspects of the Gas Lift: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., 1926, pp. 124-129.

<sup>39</sup> Vietti, W. V., and Oberlin, W. A., Problems Encountered in Handling Panhandle Crude: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., 1927, pp. 271-284. Reistle, C. E., jr., Methods and Tools for Removing Paraffin from Flowing Wells: Rept. of Investigations 2302, Bureau of Mines, 1927, 3 pp.

the paraffin hook, provided the casing or tubing is not almost or completely plugged. This tool does not actually remove the paraffin from the well but instead cuts it loose from the pipe and permits the flowing oil to carry the loosened paraffin from the well. The paraffin knife or flow devil is not satisfactory for removing the paraffin from the tubing of pumping wells because it does not actually remove

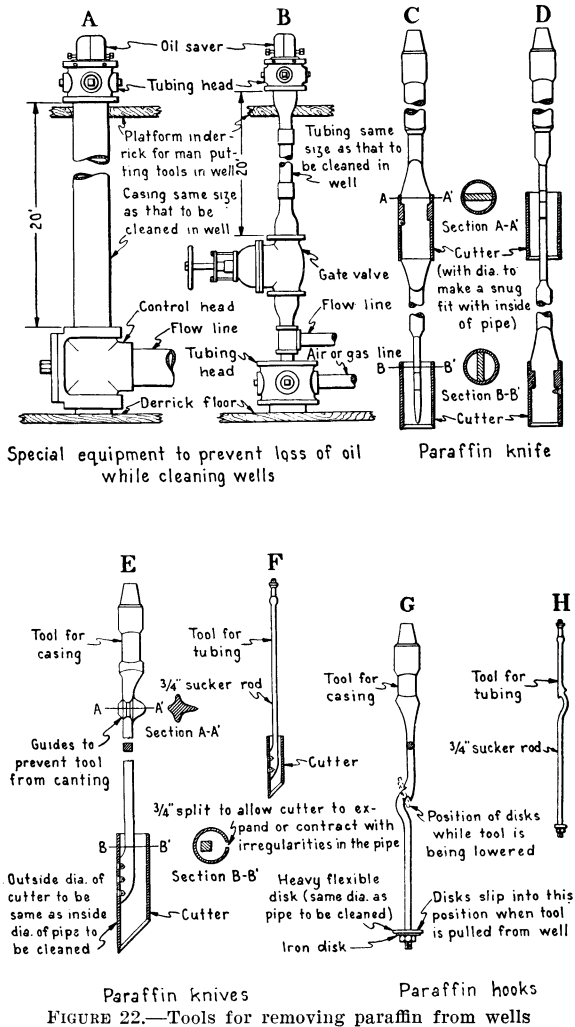


FIGURE 22.—Tools for removing paraffin from wells

the paraffin, and the loosened paraffin plugs the tubing below and clogs the pump when the rods and cups are run back into the well.

The paraffin knife or flow devil should be run into flowing wells frequently to prevent large quantities of paraffin from accumulating, as it is difficult for the ascending oil to remove large quantities. Care should also be taken not to run the tool too rapidly because there is danger of cutting more paraffin from the pipe than can be carried from the well by the oil, thus forming a bridge and possibly plugging the oil string.

The construction of the paraffin knife or flow devil is shown in Figure 22. This instrument is usually run into the well on a  $\frac{1}{2}$  or  $\frac{5}{8}$  inch line, using a short stem or sinker bar to force it through the paraffin against the velocity of the rising oil.

The use of a special wellhead (fig. 22), as suggested by the author in a previous report,<sup>40</sup> is particularly advisable with the paraffin knife or flow devil and in some wells with the paraffin hook. The special head eliminates waste of oil and coating of the derrick, workmen, and equipment with paraffin and oil. Figure 23 shows the dirty condition of the derrick and equipment when the paraffin knife, flow devil, swab, or paraffin hook is run into the well without using the special head.

#### OTHER TOOLS

In addition to standard tools, paraffin hooks, and paraffin knives or flow devils to remove accumulated paraffin from oil wells there are undoubtedly other tools which function in a similar manner and are perhaps equally satisfactory for removing paraffin. Rod guides, metal buttons cast or welded to the rods, and similar equipment for removing and preventing paraffin in pumping wells are not particularly satisfactory unless they are placed on the rods at intervals slightly less than the length of the pumping

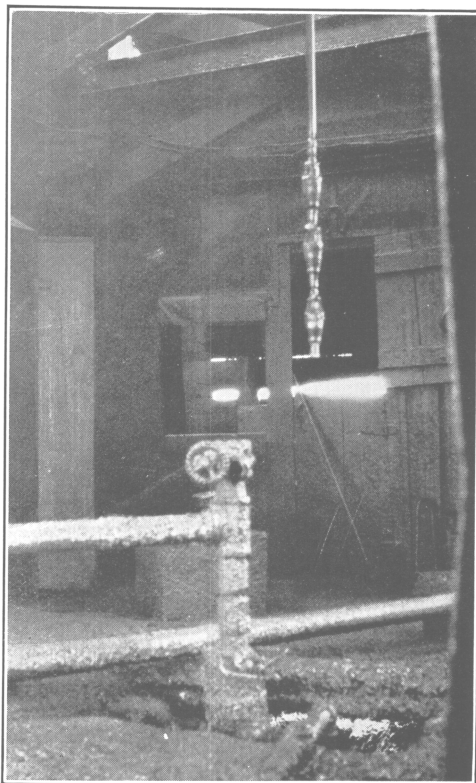


FIGURE 23.—Derrick floor and equipment coated with paraffin and oil after cleaning tubing without use of special head

stroke and extend to the depth at which the paraffin stops accumulating. The entire surface of the pipe on which paraffin accumulates will then come into contact with the buttons or guides and will scrape clean. The tools described are adequate to solve all problems encountered where tools are required to remove paraffin.

When rods and tools are pulled from a well while paraffin is being removed, the rods or line usually bring up considerable paraffin that has been scraped from the walls of the pipe. Frequently, the floor man scrapes the paraffin from the rods and line by gripping them loosely between his hands. This practice is dangerous because wickers are always present on steel rods and wire lines. The use of

<sup>40</sup> Reistle, C. E., jr., Work cited.

a piece of soft rope, as shown in Figure 24, or a section of an automobile tire casing,<sup>41</sup> is much better.

Some operators use steel-floor grating around the casing head, as shown in Figure 25, to prevent oil, paraffin, sand, or other material from dropping into the cellar. Disposing of these substances in that way provides a clean, safe floor around the casing head for workmen

to stand on while running tubing and rods, cleaning out, and doing other work. A concrete cellar connected with a concrete sump at the side of the derrick is an added precaution taken by some operators to lessen the fire hazard and collect the waste oil.



FIGURE 24.—Safe method of stripping paraffin from sucker rods

#### PORTABLE HOISTS FOR OPERATING TOOLS

Producing wells are seldom equipped for running tools into the oil string, and the portable hoist offers a satisfactory and economical method for operating tools and removing paraffin. Any type of portable hoist may be used, provided it has sufficient power to pull the paraffin and tools from the pipe. In the Salt Creek field portable electric hoists (fig. 26) have been found very efficient for operating cleaning-out tools. Two types of electric hoists are

used: (1) Those with the entire unit mounted on a 1½-ton truck for rod-pulling jobs and for removing paraffin from pumping wells, the equipment consisting of a 20-horsepower slip-ring, hoist-type motor driving a single-drum-type hoist with a capacity of 1,800 feet of ½-inch cable, 1,150 feet of ⅝-inch cable, or 800 feet of ¾-inch cable; and (2) a heavier hoist powered by a 40-horsepower motor of the same type mounted on a 3½-ton truck. This outfit is used especially for pulling tubing and cleaning out paraffin and sand from wells.<sup>42</sup>

<sup>41</sup> Miller, H. C., Safe Practices at Oil Derricks: Tech. Paper 419, Bureau of Mines, 1927, p. 33.

<sup>42</sup> Peake, A. W., and Prior, F. O., Use of Electricity in Wyoming Oil Fields: Petrol. Dev. and Technol., Am. Inst. Min. and Met. Eng., 1927, p. 210.

The cost of operating cleaning-out tools with portable hoists is approximately \$23.50 a day, allotted as follows:

Labor :	
Foreman-----	\$5.00
Winch operator-----	5.00
Helper-----	4.00
Depreciation, interest on investment, and maintenance-----	7.50
Power and gasoline-----	1.50
Miscellaneous-----	.50
<hr/>	
Total-----	23.50

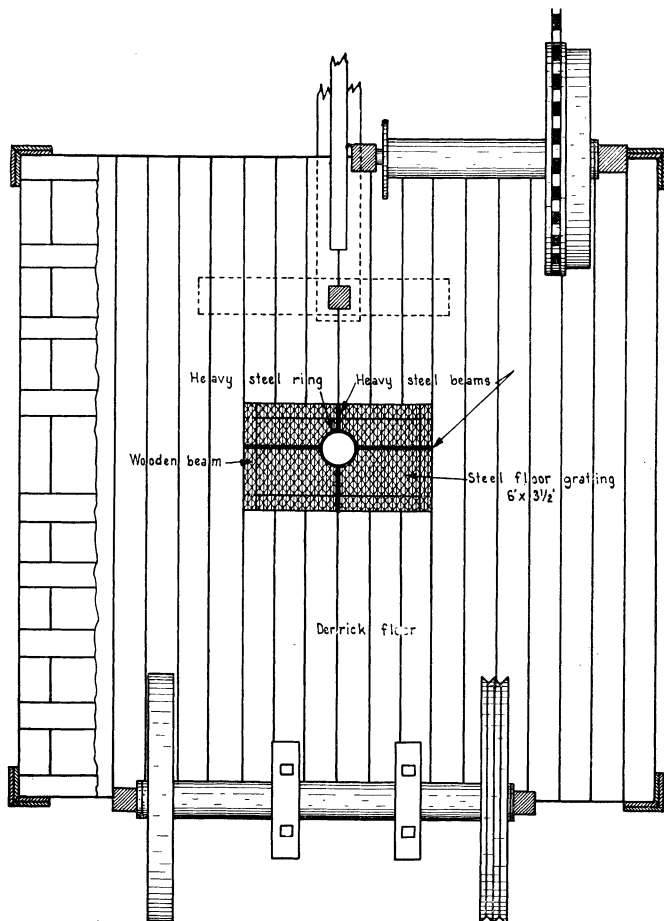


FIGURE 25.—Steel flooring surrounding wells to eliminate the accumulation of paraffin and oil on derrick floor

Eight crews in the Salt Creek field pulled rods and ran paraffin hooks in 370 wells, 2,000 feet in depth, in 28 days—an average of 1.65 wells per day per crew—at a cost of \$14.25 per well.

If portable hoists are used for running paraffin knives or flow devils in flowing wells equipped with special heads for “lubricating in” the tools, they can be used in running the tools in 6 to 10 wells per day at a maximum cost for removing paraffin of \$3.95 per well.

The use of hoists for pulling tubing and cleaning out paraffin and sand represents an appreciable saving over the use of clean-out tools and rigging up the well preparatory to running the tools. For example, the average duration of clean-out jobs, using portable hoists, in the Salt Creek field, was five days during December, 1928, whereas the average time for rigging up and cleaning out with tools was 10 days, or just twice as long. The average daily cost of rigging

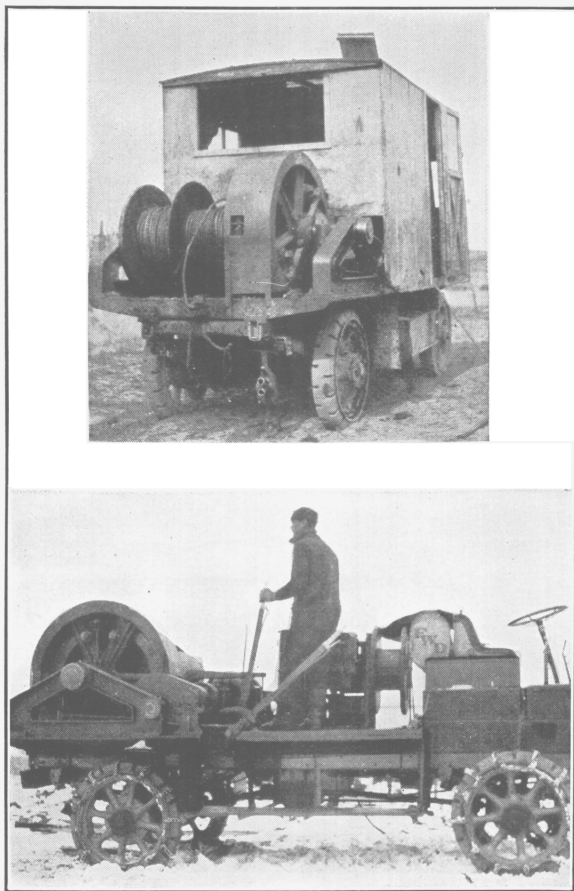


FIGURE 26.—Portable electric winches for removing paraffin from wells in Salt Creek field, Wyoming

up, using electric power and standard tools, was estimated at approximately \$23.97, or a total cost per job of \$239.70, compared with \$23.50 a day or a total cost of \$117.50 using portable hoists, according to H. S. Bossart, Midwest Refining Co.

#### COMPRESSED AIR OR GAS

The use of compressed air or gas for removing paraffin from the oil string or casing is seldom advisable or practical,<sup>43</sup> except where

<sup>43</sup> Wood, F. E., Young, H. W., and Buell, A. W., Handling Congealing Oils and Paraffin in the Salt Creek Field, Wyoming: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., 1927, p. 261.

high-pressure gas or air is available without additional expense. In the Elk Basin field, Wyoming, high-pressure gas was used to remove paraffin from the casing and tubing of a number of wells, but in many wells the paraffin was carried to the bottom of the well and apparently forced into the sand because the production after the use of gas was less than before attempts were made to remove paraffin. To regain the original production it was necessary to remove the paraffin from the face of the sand by washing with hot kerosene and shooting and in some wells by scraping it off with tools. When compressed air or gas is used to remove paraffin the string of pipe through which the air or gas is injected must be free of paraffin; otherwise, the air or gas will force the paraffin to the bottom of the well and plaster it on the sand face. In most wells paraffin tools are much more satisfactory than air or gas.

#### MECHANICAL METHODS FOR REMOVING PARAFFIN FROM THE SAND

Paraffin on the face and in the pores of the sand can not be removed by mechanical methods unless the sand in which it is deposited is also removed. The mechanical methods so far devised to remove accumulations of paraffin from the sand about the well only apply to removing the paraffin on the face of the sand or in close proximity to the well. Usually other methods, to be discussed later, for removing the paraffin from the face and pores of the sand are more economical and satisfactory.

#### STANDARD TOOLS

The usual method of cleaning out wells with standard tools to loosen accumulated sand and silt and mix it with water and oil so that it can be bailed from the well does not apply to removing paraffin deposited in the pores of the sand. In some wells the tools may cut enough sand away to remove any paraffin deposited on the face of the sand, but usually it is doubtful. If hot oil, hot water, or a good paraffin solvent is used in connection with tools, the paraffin on the face of the sand may be removed but not that deposited in the sand. Paraffin in the sand and, in most wells, on the face of the sand can not be removed satisfactorily by the use of tools and the standard methods of cleaning out wells.

#### SAND REAMERS

At least one oil-tool supply company sells a reamer for cutting away the surface of the sand to remove deposited paraffin and increase the diameter of the hole in the sand. The reamer consists of a large steel shaft 2 to 6 feet in length and covered with sharp cutting points. The reamer is run into the well on the end of tubing or small-diameter drill pipe and is connected to it by a swivel socket. When the pipe is rotated rapidly centrifugal force holds the reamer against the sand. Reaming is begun at the bottom of the sand, and when the reamer reaches an almost horizontal position the tubing is raised slowly until the top of the sand is reached. After the reamer is used, it is advisable to clean out the loose sand and paraffin before the well is put back on production. The company that markets the reamer recommends the use of compressed air to remove



the cuttings and clean the well; however, a sand pump, bailer, or other tools may be used if desired. In old fields where the pressure in the sand is low, care should be taken not to force water, paraffin, or silt back into pores of the sand and permanently close the drainage channels into the well.

The sand reamer can be used only in wells where the producing formation is exposed; it can not be used in wells where screened or perforated pipe is set opposite the producing sand unless the screened or perforated pipe is first removed from opposite the sand. The sand reamer has no value where the hole has previously been enlarged by caving or explosives to a diameter greater than it can cut. After the hole is once enlarged and the paraffin removed by the reamer method it can not be enlarged further, unless a longer cutting arm is used; therefore, paraffin can only be removed a limited number of times by this method. Although the sand reamer has been used successfully in shallow wells where the sand is soft it is questionable whether it will function equally satisfactorily in deep wells or in hard sands.

In wells in which the sand reamer actually cuts away the sand and enlarges the diameter of the hole the production of oil generally increases, due to the removal of the paraffin which plugged a portion of the sand and to the increase in the diameter of the well.<sup>44</sup> The cost of using this method ranges from \$300 to \$1,500 per well, depending mainly upon the depth of the well and the hardness and thickness of the sand.

#### HYDRAULIC METHOD

Uren<sup>45</sup> describes a method of cutting away the face of the sand by the hydraulic action of a high-pressure jet of oil. This method would also apply to removal of paraffin deposited on the face of unconsolidated sand or in close proximity to the well. The proposed equipment consists of two strings of pipe, one inside the other. The outer string has a jet at the bottom and is packed off from the smaller string. The oil is forced down between the two strings of pipe and jetted against the sand, and oil and loose sand is lifted by gas or air or flowed under its own pressure to the surface through the smaller pipe. The pipe is rotated during the operation of jetting off the face of the sand, and it can also be raised or lowered to insure cutting the entire surface of the sand.

It is questionable whether this method is practicable for removing paraffin from the sand except where the operator also wants to increase the diameter of the well. Even then it only applies to soft or loosely consolidated sands and can not be used where screened or perforated pipe is in place opposite the producing sand. The cost would be greater than other methods of removing paraffin from the face and pores of the sand.

#### COMPRESSED AIR OR GAS

In a number of fields compressed air or gas, especially where it is available without additional expense for compressing it to the

<sup>44</sup> Uren, L. C., Increasing Production of Petroleum by Increasing Diameter of Wells: *Trans. Am. Inst. Min. and Met. Eng.*, 1925, pp. 1276-1301.

<sup>45</sup> Uren, L. C., *Work cited.*

desired pressure, is used for cleaning wells. On some properties portable compressors have been used. The use of compressed air or gas for removing paraffin dates back to the earliest operations of oil wells. In 1865 Bone<sup>46</sup> discussed the removal of paraffin from the casing of wells by means of compressed air forced down a smaller pipe inserted in the casing and called an "ejector." Although the compressed-air or gas method of removing paraffin from producing wells has been tried for over 70 years it is questionable whether paraffin can be removed satisfactorily by this method. In any event, serious damage to the oil sand may result if the operation is performed improperly.

Where compressed air or gas is used to remove the accumulated paraffin the paraffin should first be loosened from the face of the sand with a reamer, solvents, or heat and held in suspension or solution in the liquid at the bottom of the well. The compressed air or gas can then be applied and the loosened or dissolved material blown from the well. If too great pressure is applied or if pressure is held on the sand too long, paraffin, fine sand, and silt may be forced into the sand pores and plug the sand; this should be guarded against.

#### EXPLOSIVES

The advisability of using explosives to remove paraffin from the face and pores of the producing sand depends primarily upon the manner in which the explosive will affect the sand. Unless the surface and that part of the sand containing the paraffin is shattered, loosened, or broken away from the main body of the sand so that it can later be removed from the well, the force of the explosion may force the paraffin more compactly into the pores of the sand and thereby aggravate instead of correct the trouble due to paraffin. Obviously, this method does not apply to wells in which screened or perforated pipe is opposite the producing formation.

The heat generated by explosives may in some instances be sufficient to melt or cause the oil to dissolve the paraffin on the face and in the pores of the sand, but this is questionable. The effect of shooting on the temperature of several wells in the Salt Creek field was determined with an electric pyrometer. A small pumping well, 26A, SW.  $\frac{1}{4}$  sec. 24, T. 40 N., R. 79 W., having a total depth of 1,572 feet, was shot at 1,544 to 1,558 feet with 20 quarts of solidified nitroglycerin. Immediately after the shot the temperature at the sand was 132° F., 32° F. above the normal temperature. One hour and twenty-five minutes later the temperature of the sand was 130° F., and 40 feet of fluid had accumulated in the hole. A large pumping well—25A, SW.  $\frac{1}{4}$  sec. 27, T. 40 N., R. 79 W., 2,414 feet deep—was shot at 11 a. m. with 20 quarts of solidified nitroglycerin placed 2,405 to 2,397 feet away, and at 4 p. m. it was reshot with 10 quarts of solidified nitroglycerin placed 2,392 to 2,381 feet away. The temperature at the sand immediately after the last shot was 120° F., with 400 feet of fluid in the hole, and as the normal sand temperature was 112° F. the explosives raised the temperature 8° F. It is doubtful if the small increase in temperature measured in these two wells would be sufficient to remove paraffin from the pores of the

<sup>46</sup> Bone, J. H. A., *Petroleum and Petroleum Wells*: 2d ed., Philadelphia, 1865, p. 27.

sand, except perhaps close to the well. Possibly, the temperature of the sand in small wells will be raised more than in large wells, because less liquid flows through the sand into small wells.

The main object of using explosives for removing paraffin is to blast off the face of the sand so that a fresh surface not filled or coated with paraffin will be exposed. The results obtained differ with the character of the sand. It is impossible in many wells to shatter the surface of the sand satisfactorily, and shooting apparently causes fissures or cracks that extend back into the sand and thereby open new drainage channels through which increased production flows without removal of paraffin from the sand.

The use of solvents to remove paraffin from the face of the sand is advisable before shooting, as it precludes the danger of plastering the surface of the sand and filling the pores with paraffin if the shot fails to shatter the surface. It is also advisable to cover the sand with a paraffin solvent, such as gasoline, kerosene, or fresh oil, so that if any of the solvent is driven back into the pores of the sand it will aid in removing precipitated paraffin.

The action of explosives in any particular sand can not be definitely predicted until several wells have been shot; therefore, the operator should experiment with one or two wells and determine definitely that the use of explosives will satisfactorily accomplish the results desired before explosives are used extensively. The cost of using explosives depends upon the quantity used and the amount of work necessary to clean the well after shooting. In 1930 a 40-quart shot in Salt Creek cost approximately \$285; a 20-quart shot, \$230. It is often advisable to clean out pumping wells before shooting and after shooting to leave the loosened material in the well, thus placing the well immediately on production, to obtain the maximum amount of oil resulting from the stimulating effect of the shot. In general, paraffin can be removed with less expense by other methods, and usually the increase in production due to the stimulating effect of the shot and the increase in size of the hole, in addition to the removal of paraffin in the sand, will offset the expense and justify the use of the method.

#### SOLVENTS FOR REMOVING PARAFFIN FROM WELLS

The use of solvents, such as gasoline, kerosene, and benzol, is probably one of the oldest methods of removing paraffin from oil wells. Solvents can be used in air or gas lift wells and pumping wells, regardless of whether screened or perforated pipe is set opposite the producing sand, but they can seldom be used in natural flowing wells because it is generally impracticable or impossible to put liquid solvents into wells that are flowing.

In gas-lift wells the solvent is usually injected into the well with the input gas. In the Panhandle field<sup>47</sup> hot kerosene was injected into a number of wells, using six to eight drums per well. Not only was the paraffin removed satisfactorily, but in some wells oil production was increased. Natural gasoline or motor gasoline can also be used to remove paraffin accumulations in oil wells, and it is a much better solvent than kerosene for paraffin.

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<sup>47</sup> Vietti, W. V., and Oberlin, W. A., Problems Encountered in Handling, Panhandle Crude: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., 1927, pp. 269-284.

A number of tests were conducted on pumping wells in the Salt Creek field,<sup>48</sup> using raw natural gasoline and drip gasoline to dissolve accumulated paraffin. The general method followed was to lubricate the gasoline into the well between the tubing and casing and to circulate the oil for six to eight hours by connecting the tubing into the casing. The results obtained from two of the tests are shown by Table 11 and 12. This method removed paraffin from the tubing satisfactorily, but it was necessary to circulate the solvent and crude oil to dissolve the paraffin that goes into solution very slowly or to heat the solvent before adding it to the well. Heating is impracticable when natural or drip gasoline is used. There was little or no difference in the production of pumping wells before and after dissolving the paraffin accumulations, because the paraffin that accumulates in tubing usually has no effect on production until it actually interferes with the operation of the pump. In some wells the cups in the pumps failed rapidly after solvents were used, probably because the sand that was held in the paraffin on the walls of the pipe settled to the bottom of the tubing and into the pump when the paraffin dissolved and released the sand.

TABLE 11.—*Production record of test well 6, SW. ¼ sec. 36, T. 40 N., R. 79 W., Salt Creek, Wyo., before and after using solvent*

Date	Production, barrels	Remarks	Date	Production, barrels	Remarks
Jan. 3	21		May 11	9	
9	21		12	3	
17	27		13	4	
23	48		14	14	
30	20		16	8	Rods pulled May 16 and 45 pounds of paraffin removed, 66 per cent clay.
Feb. 7	17				
17	27		17	2	
20	25		18	9	
28	6		18	8	
Mar. 7	8		20	9	
12	19		21	6	
21	5		22	9	
28	7		23	-----	No record.
Apr. 4	5		24	-----	Do.
11	15		25	15	
18	8	Rods pulled Apr. 17, and 100 pounds of paraffin removed.	25	8	
19	8		27	10	
20	9		28	10	
21	13		29	8	Rods pulled May 29 and 44 pounds of paraffin removed, 60 per cent clay.
22	-----	Well dead.			
23	-----	Do.			
24	-----	Do.	30	8	
25	8		31	8	
26	-----		1	6	
27	9		2	6	
28	10		3	8	
29	2		4	9	
30	0	Rods pulled Apr. 30 to remove paraffin and start test.	5	6	
May 1	0	No record.	7	3	
2	0	Do.	8	16	
3	12		9	9	
4	3		10	9	
5	9		11	7	Well treated with 2 barrels of gasoline in casing and 1 barrel in tubing, June 11.
6	4				
7	4		12	10	
8	13		13	9	
9	8		14	9	Rods pulled June 14, and 3 pounds of paraffin removed.
10	8				

<sup>48</sup> Reistle, C. E., jr., *Methods of Dealing with Paraffin Troubles Encountered in Producing Crude Oil*: Tech. Paper 414, Bureau of Mines, 1928, pp. 22-27.

TABLE 12.—*Production record of test well 12A, SE. ¼ sec. 13, T. 40 N., R. 79 W., Salt Creek, Wyo., before and after using solvent*

Date	Pro- duction, barrels	Remarks	Date	Pro- duction, barrels	Remarks
Jan. 3	115		May 17	50	
9	101		18	56	
14	92		19	0	No record.
22	104	Rods pulled Jan. 21, and paraffin removed.	20	53	
			21	58	
29	89		22	51	
Feb. 7	15	Rods pulled Feb. 2, and paraffin removed.	23	54	
			24	56	
13	76		25	50	
18	28		26	54	
25	79	Rods pulled Feb. 25, and paraffin removed.	27	54	
			28	53	
Mar. 4	100		29	53	
10	73	Rods pulled Mar. 11, and paraffin removed.	30	55	Rods pulled and 15 pounds of paraffin removed.
17	69				
24	68		Rods pulled Mar. 25, and paraffin removed.	June 1	0
Apr. 2	63	Rods pulled Mar. 31, and all paraffin removed to start test.	2	0	Do.
9	55		3	0	Do.
13	51		4	0	Do.
19	49	Rods pulled Apr. 18, and 40 pounds of paraffin removed.	5	49	
			6	76	
20	62		7	56	
21	63		8	51	
22	56		9	55	
23	60		10	0	No record.
24	64		11	54	Well treated with one-half barrel of gasoline in tubing and one-half barrel in casing, June 11.
25	60				
26	57		12	58	
27	64		13	53	
28	43		14	54	
29	58		15	7	Well off 19 hours while rods were being pulled; 5 pounds of paraffin removed.
30	60				
May 1	55		16	60	
2	52	Rods pulled and 40 pounds of paraffin removed.	17	54	
			18	54	
3	60		19	54	
4	53		20	54	
5	60		21	54	
6	0	No record.	22	54	
7	79		23	49	
8	68		24	53	
9	75		25	50	Well treated with 2 barrels of gasoline in casing and circulated 2 hours.
10	42				
11	77		26	56	Rods pulled; 4½ pounds of paraffin removed June 26.
12	52				
13	56		28	57	
14	57				
15	63				
16	58	Rods pulled May 16, and 40 pounds of paraffin removed.			

The advisability of using solvents to remove paraffin from the oil string depends primarily upon the cost of the solvent used. Inasmuch as the solvent is recovered as crude oil the value of the additional oil recovered should be deducted from the gross cost of the solvent. In most pumping wells paraffin can be removed from the oil string more economically by use of a paraffin hook operated by a portable hoist.

#### SOLVENTS FOR REMOVING PARAFFIN FROM SAND

Solvents can be used satisfactorily for removing paraffin deposited on the face of the sand, but they will not remove paraffin deposited in the pores of the sand unless heated or forced into the sand. Furthermore, the use of solvents will not prevent the accumulation of paraffin on the face of the sand, and paraffin will continue to accumulate unless corrective methods are followed after it is removed.

To remove paraffin successfully from the face of the producing sand, enough solvent should be introduced into the well to cover the entire sand when mixed with the accumulated oil. Gasoline is not only a good solvent, but it is usually preferred to other solvents for this work because it is available at a moderate cost. The solubility of paraffin in gasoline is shown in Figure 2, page 8. To remove paraffin from the face of a producing sand it is essential that the solvent be in contact with the paraffin for at least several hours and preferably longer. In sands where the formation pressure is low the solvent can often be forced back into the sand by adding enough of it to build up a hydrostatic pressure greater than the rock pressure or by applying gas or air pressure to the well at the wellhead after the solvent has been added. Forcing the solvent back into the sand tends to dissolve and remove the paraffin that is deposited in the sand pores. In wells where a vacuum has been applied to the producing sand the solvent will often be drawn into the sand pores by the vacuum in the sand, and there will be no need of creating a high hydrostatic pressure or applying back pressure.

The oil being produced from the sand will often dissolve the paraffin accumulated on the face of the sand without a solvent being added, if allowed to accumulate and build the fluid level up above the top of the sand. Vietti<sup>49</sup> states that this method proved satisfactory in a number of wells in the Panhandle district, Texas. In one well the oil was allowed to stand for four days, and the average daily oil production increased from 45 barrels to 130 barrels for the two weeks after the removal of paraffin.

The use of gasoline, kerosene, hot oil, or organic liquids as a solvent for removing paraffin from the face of the sand is often satisfactory and economical, and in most wells natural gasoline and kerosene have proved the cheapest and best solvents.

#### REACTION OF CHEMICALS

The use of strong acids, such as hydrochloric and sulphuric, as solvents for paraffin in oil wells is not recommended because paraffin is a relatively stable compound and will not be affected by such compounds. Furthermore, acids may attack and do considerable damage to the equipment in the well and may decrease the permeability of the sand<sup>50</sup> by changing the character of the cementing material in the sand.

In some fields water produced with the oil deposits mineral salts in the well. Mills<sup>51</sup> found that the use of hydrochloric acid would remove the mineral deposits, but he emphasizes the danger of using sulphuric acid due to the possible deposition of the insoluble calcium, strontium, and barium sulphates in the pores of the sand.

Lye or other strong bases have no value for dissolving paraffin, although these basic chemicals do not tend to damage the sand.<sup>52</sup>

<sup>49</sup> Vietti, W. V., Work cited.

<sup>50</sup> Nutting, P. G., Some Physical Problems in Oil Recovery: Oil and Gas Jour., vol. 28, No. 27, Nov. 21, 1929, pp. 44-45.

<sup>51</sup> Mills, R. van A., The Paraffin Problem in Oil Wells: Rept. of Investigations 2550, Bureau of Mines, December, 1923, 11 pp.

<sup>52</sup> Nutting, P. G., Work cited. Bechtel, L. L., and Blenkarn, W. O., Water and Lye Solution Cleans Wells of Paraffin; Oil Weekly, vol. 49, No. 7, May 4, 1928, p. 39.

## HEAT FOR REMOVING PARAFFIN

Increasing the temperature of crude oil increases the solubility of paraffin, and the two become miscible in all proportions as the temperature approaches the melting point of the wax fractions in the paraffin. Figure 2 (p. 8) shows the effect of temperature on the solubility of paraffin in crude oil. The application of heat will also cause the paraffin to become a liquid if the temperature of the oil is high enough. There are many methods of furnishing heat to the oil and paraffin to dissolve the paraffin and remove it from the oil string. A number of these will be discussed.

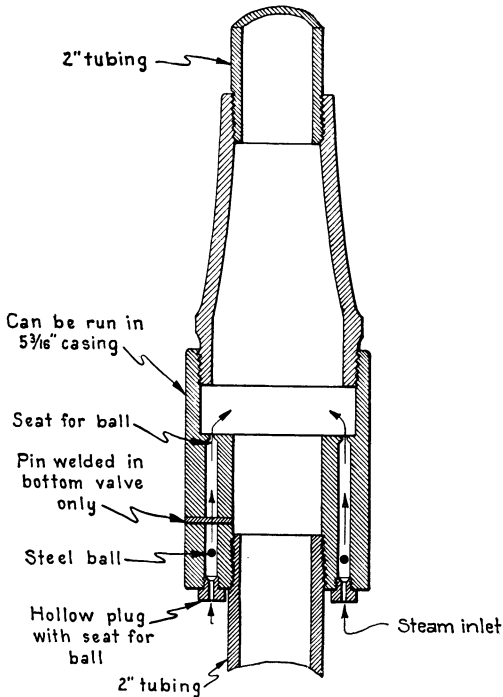


FIGURE 27.—Valve for injecting steam into oil string

## STEAM FOR REMOVING PARAFFIN FROM WELLS

Steam is successfully used to heat and remove paraffin in the oil string of flowing wells, and a number of methods have been developed to meet the variety of conditions met. All of the methods depend upon transmitting heat to the oil and paraffin in the oil string and melting and dissolving the paraffin so that it can be flowed with the oil from the well.

The simplest method of heating the oil and paraffin in the oil string is to inject steam into the space between the oil string and casing and allow the condensate to accumulate in the well and be produced with the well. This method can

only be applied in tubed wells where the formation pressure is less than the available steam pressure and where the condensed steam does not emulsify with oil to any great extent or damage the sand and cause caving of the exposed formations in the well.

It is advisable to apply sufficient steam to heat the oil and the oil string to a temperature of 135° to 145° F. for a depth somewhat below the bottom of the accumulation of paraffin. The length of time and amount of steam required to melt the paraffin depend upon the conditions in the well and can be determined only by experimentation. Usually the application of steam to a well for three to six hours is sufficient to melt the accumulated paraffin.

There are a number of different types of valves similar in principle to that shown in Figure 27 for injecting the steam into the oil

string<sup>53</sup> of flowing wells. The valves are usually spaced at intervals of approximately 200 feet on the oil string and extend below the paraffin accumulation, so that when steam is injected into the space between the oil string and casing it will blow into the oil string after enough pressure is built up to overcome the hydrostatic pressure. For example, at 400 feet, in a well producing 39° A. P. I. gravity oil, the steam pressure would have to be greater than 143 pounds per square inch at the valve before it would operate. It is questionable whether these valves actually produce any better results than those obtained by injecting the steam directly into the space between the casing and tubing without them.

The apparatus shown in Figure 28 was devised and used by K. C. Sclater, former petroleum engineer for the Oak Ridge Oil Co., Santa Paula, Calif., for removing paraffin from the oil string of flowing wells. This method removes the paraffin satisfactorily, but its principal disadvantage is that production of the well must be stopped while the paraffin is being removed, and if the well does not start to produce oil immediately after the valve is opened following steaming the melted paraffin accumulates and congeals below the valve.

Although steam coils will not produce as efficient a heat transfer to the oil and oil string as injecting the steam directly into the space between the casing and oil string, they can be used in all types of wells regardless of the pressure conditions existing in the casing or tubing. Steam coils also eliminate the danger of condensed steam damaging the well or causing emulsion troubles. A number of different types of coils are shown in Figure 29. These coils were used by K. C. Sclater in experimenting with the steam and hot oil for removing the paraffin from flowing wells in Santa Paula, Calif. The main objection to coils in which the condensed steam is returned to the surface is that they can only be used to a depth at which the available steam pressure can lift the column of condensed water. A 200-foot head of water, for example, exerts a pressure of approximately 87 pounds per square inch at the base of the coil, and it would therefore be necessary to have approximately 90 pounds of steam pressure to insure operation of a coil extending 200 feet into the well.

The use of steam for removing paraffin from the oil string of natural-flowing and gas-lift wells is not as economical or practical as the use of a paraffin knife or flow devil operated by a portable hoist. The number of wells that can be heated by one boiler is limited due to the length of steam line required, and assuming that 10 wells can be satisfactorily reached by steam lines from one boiler the interest and depreciation on a 40-horsepower boiler and boiler house is approximately \$4 per day or a fixed charge of \$120 a month. If each well required four hours steaming a week and two wells could be steamed at a time the boiler would have to be operated two and one-half 8-hour days each week or 10 days a month, requiring the labor of one man at \$4 per day, fuel at \$4 per day, and miscellaneous expense \$0.50 per day, or a total of \$8.50 per day or \$85 a month. Eighty-five dollars operation expenses plus \$120 depreciation and interest on investment makes a total of \$205 a month,

<sup>53</sup> Oil Field Engineering, Paraffin Removed Without Interrupting Production: Vol. 6, No. 2, Aug. 1, 1929, p. 18.



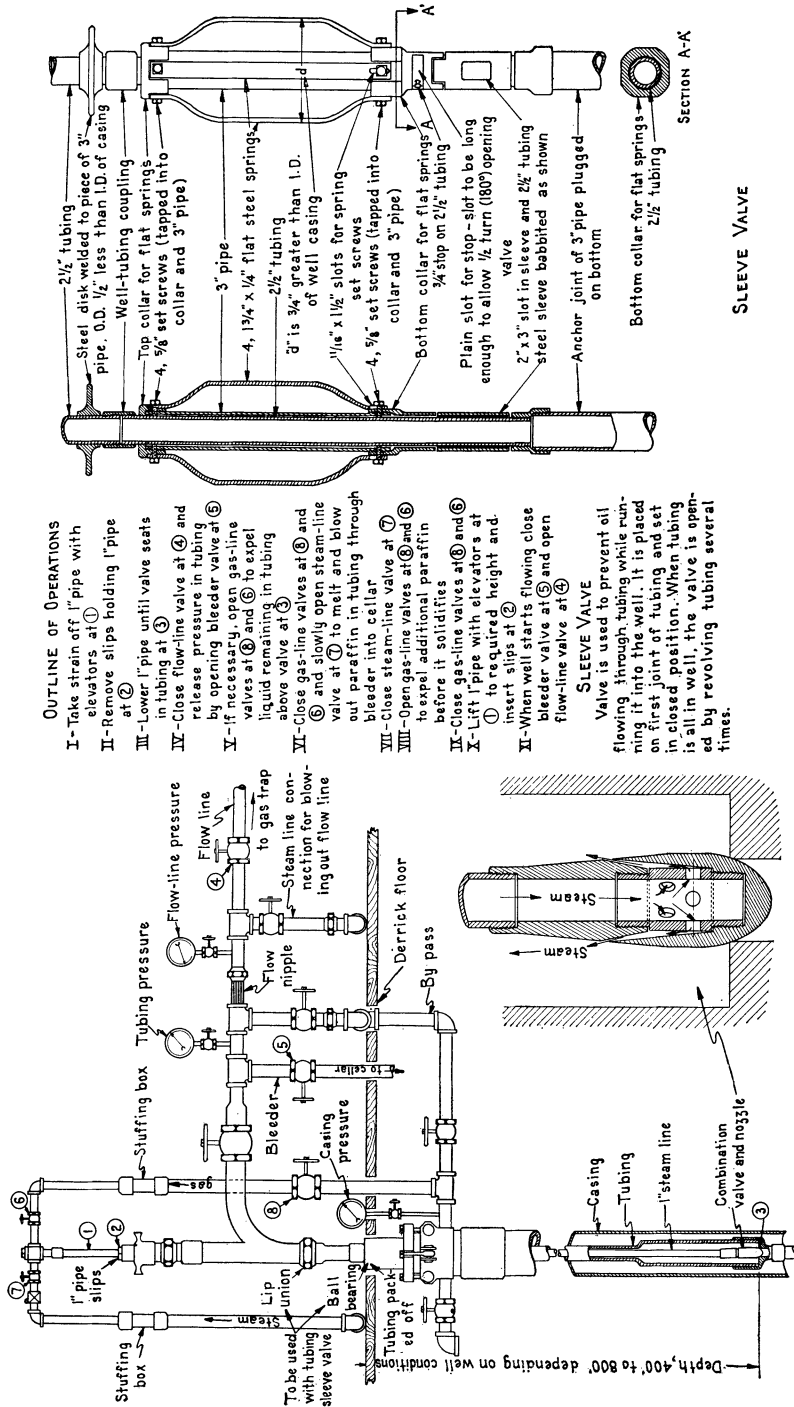


FIGURE 28.—Apparatus used by K. C. Sclater in experimenting with steam for removal of paraffin from oil string of flowing wells

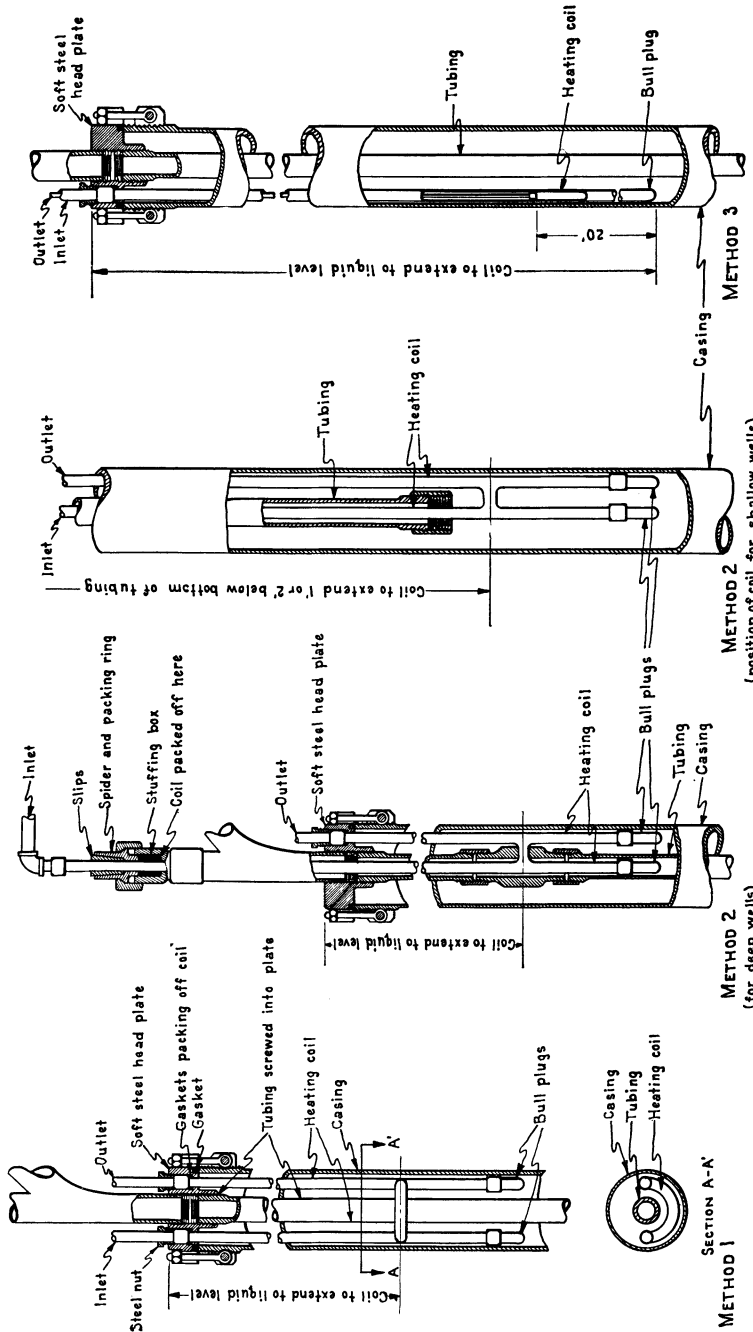


FIGURE 29.—Steam coils used by K. C. Schlater in experiments for the prevention of paraffin accumulations in the oil string

or \$20.50 per well. This figure does not take into consideration the cost of laying pipe to the well, putting coils into the well, or other expenses that would undoubtedly run the cost up to more than \$25 per well. The same results could be obtained with portable hoists and a paraffin knife or flow devil at a cost of \$3.95, as previously stated, each time the well is cleaned, or a total of \$15.80 per well per month. In some fields, however, where steam is available without additional expense, paraffin might be removed economically by steaming.

From the standpoint of cost the use of steam for removing paraffin from pumping wells is seldom satisfactory, except where steam is available without additional expense. In most pumping wells, pulling the rods and using a paraffin hook will prove more economical than steaming.

#### STEAM FOR REMOVING PARAFFIN FROM SAND

The application of steam heat to the producing sand is often a practical method of heating the sand and removing paraffin in shallow wells. Since trouble from paraffin deposited on the face and in the pores of the sand is encountered more generally in older fields where the fluid level has been maintained near the bottom of the sand and all the wells are mechanically pumped or intermittently pumped with compressed air, steaming is common in old fields. Furthermore, paraffin trouble in the sand is primarily a small-well problem and necessitates a careful study of the methods to be employed, the expenses to be incurred, and the increase in production to be expected before the attempt is made to repair small wells by injecting steam to melt the accumulated paraffin.

The general procedure when steam is used to remove paraffin from the sand of pumping wells is to raise the pump or place a flood nipple or fluid-level regulator so that the fluid level will be maintained at the top of the sand. A small (1 or 2 inch) string of pipe is then run to the bottom of the sand to carry the steam. The well is pumped during the steaming process to remove the accumulated paraffin, oil, and water. It is usually advisable to add several barrels of kerosene to small wells while steaming them. Steaming can be used for removing paraffin on the face of the sand and in close proximity to the surface of the sand, but it does not create a temperature high enough to effect the removal of paraffin very far back in the sand. In fields where a considerable footage of uncased formations is exposed in the well and in some producing sands the use of steam causes caving, which results in more expensive operations due to the necessity of cleaning out the loose material in the well. In such fields the cost of cleaning out a well after steam is used to remove paraffin is usually prohibitive. Steaming the sand usually requires a day for steaming and three days to pull and rerun the tubing and clean the well. The cost of steaming and cleaning-out work ranges from \$50 to \$100, depending upon the cost of pulling and running the tubing and the cost of steam, but this cost is usually offset by an increase in production when paraffin is removed from the face and pores of the sand. The heat also reduces the viscosity of the oil in the sand adjacent to the well and liberates additional gas from the oil, all of which aids in increasing the oil production. Unless the increase in

production more than offsets the cost of using steam it should not be used.

Superheated steam is used in the patented Larsen process.<sup>54</sup> The steam is run into the well through a  $\frac{3}{4}$ -inch line inside of a 2-inch line for insulation. During the steaming operation the well is also

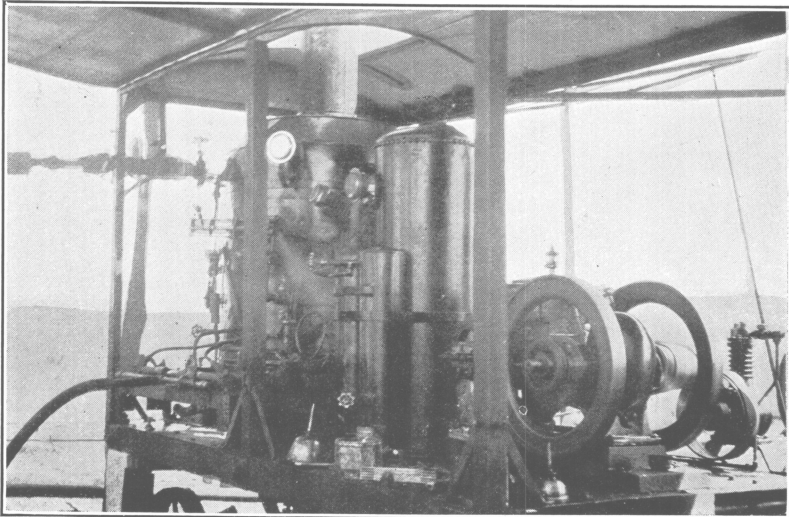
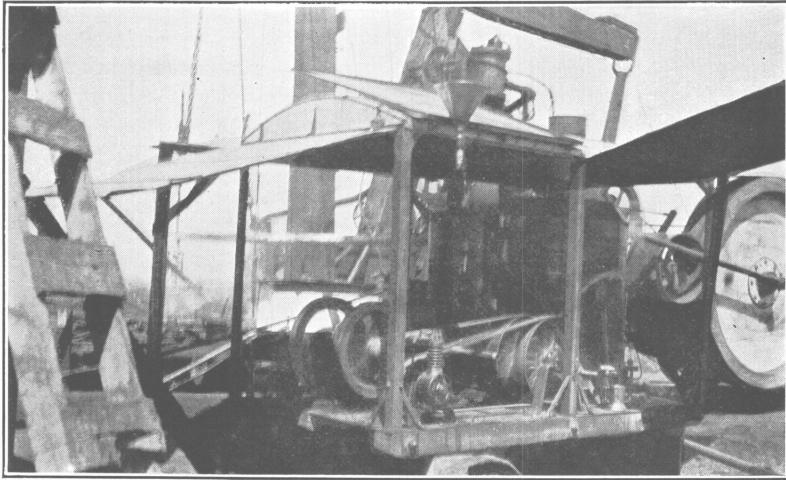


FIGURE 30.—Special boilers for generating high-pressure superheated steam

equipped with tubing and a pump to remove the water, oil, and paraffin accumulating in the bottom of the well. Cups of special composition are necessary in the pump to withstand the heat, or else a pump must be used that does not require cups.

In the Larsen process the steam is generated in a special boiler (fig. 30, *A* and *B*) capable of evaporating 85 pounds of water per

<sup>54</sup> Reistle, C. E., jr., *Methods of Dealing with Paraffin Troubles Encountered in Producing Crude Oil*: Tech. Paper 414, Bureau of Mines, 1928, pp. 33-34.

hour and superheating the steam to a temperature of 1,000° F. under a pressure of 1,500 pounds per square inch. Steaming is continued for 12 to 36 hours, depending upon the amount of paraffin in the well.

In the tests conducted in Salt Creek field it was found that the steam loosened the shale and bentonite present in the producing sand, causing considerable cavings and mud to accumulate in the well, and necessitated cleaning it out with tools. The temperature of the sand in one well after steaming was found to be 230° F., whereas the normal sand temperature is 103° F. This high temperature was sufficient to insure the removal of all paraffin in the sand adjacent to the well, and an increase in production was obtained in the several wells treated; however, the increase in oil production was not sufficient in any well to offset the expenses incurred in steaming the well. The expenses were very high, due mainly to the length of time the wells were steamed and the necessity of removing the loosened sand and cavings after steaming. The production figures for two wells in the Salt Creek field before and after steaming are given in Tables 13 and 14.

TABLE 13.—*Production record of test well 7A, NE. ¼ sec. 13, T. 40 N., R. 79 W., Salt Creek, Wyo., before and after steaming with superheated steam*

Date	Pro- duc- tion, barrels	Remarks	Date	Pro- duc- tion, barrels	Remarks
Feb. 15	24		Mar. 10	0	Steam lines put in.
16	27		11	0	
17	24		12	0	
18	21		13	17	Steaming.
19	16		14	7	Steaming off.
20	28		15	31	Do.
21	32		16	27	Do.
22	5		17	0	Steaming.
23	19		18	20	Steaming off.
24	21		19	11	Do.
25	28		20	15	Steaming.
26	20		21	0	Well cleaned out and new pump
27	19				run.
28	17		22	0	Do.
Mar. 1	19		23	0	Do.
2	29		24	0	Do.
3	29		25	50	
4	24		26	44	
5	27		27	40	
6	21		28	35	
7	24		29	32	
8	21		30	22	
9	29		31	33	

Heating the oil sand with superheated or low-pressure steam is only advisable in wells where the steam will not damage the sand or cause caving, which necessitates subsequently cleaning out the wells with tools. It is always advisable to experiment with two or three wells to determine definitely the results that can be expected from steaming before making extensive preparations for the application of steam for the removal of accumulated paraffin. Obviously, it is also a waste of time and money to heat a sand with steam unless there are good indications that the sand is paraffined up.

TABLE 14.—*Production record of test well 19A, SE. ¼ sec. 13, T. 40 N., R. 79 W., Salt Creek, Wyo., before and after heating with superheated steam*

Date	Pro- duc- tion, barrels	Remarks	Date	Pro- duc- tion, barrels	Remarks
Mar. 11	42		Apr. 6	52	Steam off.
12	47		7	47	Steaming.
13	47		8	47	Steaming equipment removed.
14	50		9	52	
15	52		10	47	
16	52		11	52	
17	66		12	52	
18	68		13	47	
19	58		14	52	
20	37		15	47	
21	58		16	42	
22	26		17	39	
23	50		18	26	
24	26		19	26	Changed pump, and cleaned out sand with sand pump.
25	52				
26	10	Rerunning tubing and steam- ing.	20	0	
		Do.	21	110	
27	0	Do.	22	136	
28	58	Steam off.	23	126	
29	31	Steaming.	24	115	
30	21	Do.	25	97	
31	42	Steam off.	26	84	
Apr. 1	63	Do.	27	92	
2	47	Do.	28	71	
3	66	Do.	29	63	
4	26	Do.	30	60	
5	63	Do.			

## CHEMICALS

As previously mentioned, there are two types of chemicals that are available to the industry for combating paraffin troubles. The one in which the chemical is supposed to prevent the deposition of paraffin, or increase the solubility of paraffin, has no particular value unless added continuously to treat the oil that is flowing into the well.

The second group of chemicals includes those that generate heat due to a chemical reaction that takes place in the well when water is added or when different chemicals are mixed, and they are of value for removing paraffin under certain conditions. A number of chemical compounds can be used to generate heat in oil wells, and several different ones are on the market for removing paraffin. The most important of these compounds consists of lye and aluminum filings which when mixed with water react violently, oxidizing the aluminum and generating approximately 700 British thermal units per pound of lye and aluminum used. One advantage of using the lye and aluminum compound is that the products of the reaction are liquid and strongly alkaline and will not damage the sand or reduce its permeability<sup>55</sup> as might happen with acids. A chemical reaction that has been used in some experimental work is the result of oxidation of aluminum as in the Thermite process,<sup>56</sup> in which the oxida-

<sup>55</sup> Nutting, P. G., Physical Problems in Oil Recovery: Oil and Gas Jour., Nov. 21, 1929, pp. 44-45.

<sup>56</sup> Wood, F. E., Young, H. W., and Buell, A. W., Handling Congealing Oils and Paraffin in Salt Creek Field: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., 1927, p. 266.

tion of aluminum is effected by igniting a mixture of iron oxide and aluminum. One disadvantage of this process is that the resulting products are solids which are sometimes difficult to remove from the well. There is some difficulty also in causing this reaction to take place under conditions found in oil wells, and due to the rapidity and violence of the reaction it is not always possible to utilize the heat produced. It is questionable whether the method can be used widely for removing paraffin from oil wells.

Mills<sup>57</sup> suggests the use of calcium carbide and water as a means of generating heat. The reaction of calcium carbide and water generates approximately 200 British thermal units of heat per pound, and it is therefore much less efficient than the reaction of aluminum and lye and iron oxide and aluminum compounds; moreover, the cost of materials is much greater. Mills also suggests the use of sodium peroxide, which reacts violently upon the addition of water and generates considerable heat with the liberation of oxygen. If sodium peroxide were used with aluminum it would in all probability produce more heat than any of the chemical reactions mentioned, but sodium peroxide is difficult to store without deterioration and is very dangerous to handle. Sodium peroxide also generates heat and liberates oxygen, and it bursts into flame when brought in contact with water or damp objects and therefore constitutes a great fire hazard. The use of strong acids and acids with other chemicals is not advisable, because they damage the metal equipment of the well and affect the producing sand adversely.

Doubtless, other chemical reactions might generate enough heat to melt deposited paraffin in oil wells, but only the more important have been discussed, mainly because they can be obtained as commercial chemicals at a nominal cost. Workmen should be cautioned to use the utmost care in using chemicals to prevent them from coming in contact with the body or clothes. Strong chemicals generally cause serious body burns, and if some of the chemical gets into the eye blindness may result. The hazards of handling chemicals are so great that only experienced workmen should be allowed to use them. It is advisable, whenever chemicals are handled, to wear goggles, rubber gloves, shoes, and an apron for protection.

#### REMOVAL OF PARAFFIN FROM OIL STRING BY HEAT-FORMING CHEMICALS

The use of chemicals for removing paraffin from the tubing and casing of wells is not generally practicable, mainly because it is difficult to cause the chemical reaction to take place in the tubing or casing and to melt the paraffin. Even then the paraffin often recongeals before the oil carries it from the well. If the reaction takes place in the bottom of the well most of the heat is dissipated before it reaches the paraffin deposits in the oil string, unless a large quantity of chemicals is used. Furthermore, it is "penny-wise and pound-foolish" to use large quantities of expensive chemicals when it is much cheaper to pull the rods and use a paraffin hook in pumping wells or to run a paraffin knife or flow devil in flowing wells. The cost of enough chemicals to insure the removal of paraffin from the

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<sup>57</sup> Mills, R. van A., *The Paraffin Problem in Oil Wells: Rept. of Investigations 2550*, Bureau of Mines, 1923, 11 pp.

tubing of pumping wells is seldom less than \$50, and in many wells there is an added cost due to the chemicals damaging the cups and necessitating pulling of the rods. Frequently, also, sufficient sand is loosened to require a cleaning-out job with tools, a sand pump, or compressed air.

#### REMOVAL OF PARAFFIN FROM SAND BY HEAT-FORMING CHEMICALS

Paraffin deposited on the face of the sand, and in screened or perforated pipe set opposite the sand, can be satisfactorily removed from wells that are not flowing by the proper use of sufficient heat-forming chemicals. In a flowing well the chemicals are removed from the well before they have an opportunity to function properly.

When chemicals that generate heat are used to melt paraffin on the face of the sand, the fluid level in the well should be raised to the top of the sand to distribute the heat to the entire surface of the sand. The fluid level may be raised by allowing the well to stand for a time or by adding enough oil, kerosene, or gasoline to bring the fluid to the desired level. When heated by chemicals the oil will then dissolve the paraffin from the face of the sand and keep the paraffin in solution or suspension until it is pumped from the well. If the well is virtually free of oil when the chemicals are used, the heat may not reach all of the sand, and the paraffin may recongeal before it is removed from the sand. Usually, the most economical method used to introduce heat-forming chemicals into pumping wells is to pour the chemicals into the well between the casing and tubing and wash them down with water without removing the tubing and pump. If there is sufficient oil in the well to cover the pump the heat from the chemicals will not damage it.

Enough chemicals should be used to raise the temperature of the oil to 140° to 160° F. If the aluminum-lye compounds are used, 1 pound of chemicals will generate 700 British thermal units, which will raise the temperature of 1 barrel of oil approximately 5° F. If the sand temperature is 100° F. it will require enough chemicals to heat the oil about 60°, or approximately 12 pounds per barrel of oil in the well, not taking into consideration the radiation or loss of heat to the sand, which will consume at least 50 per cent of the heat generated, making it necessary to use 18 pounds of chemicals for each barrel of oil in the well. If there are 5 barrels of oil in the hole in the sand 90 pounds of chemicals should be used. The requirements, of course, vary with the temperature of the sand. The author believes that it is a mistake to add excessive quantities of water to the chemicals because additional heat will be required to raise the water to the desired temperature. Hot water should always be used if available. It is exceedingly doubtful if sufficient heat can be generated with chemicals to heat the sand an appreciable distance from the well, but it is reasonable to suppose that the hot oil will dissolve some of the accumulated paraffin back in the body of the producing sand. Just how far can only be conjectured.

A well should be allowed to stand without pumping for one to three hours after chemicals are added to insure complete solution of the paraffin on the face of the sand. If allowed to stand for a much longer period than three hours the sand may cool sufficiently



to cause reprecipitation of some of the paraffin. The cost of using chemicals to heat the sand ranges from approximately \$50 to several hundred dollars, depending upon whether it is necessary to subsequently pull tubing or clean out the well with tools.

#### HOT GAS FOR REMOVING PARAFFIN

The application of heat to the oil string<sup>58</sup> by the injection of hot gas is only practical in gas-lift wells. The usual procedure is to heat the gas near the well with steam-heat exchangers or gas-fired heaters. The amount of heat that can be transmitted to the well in this manner is limited, because of the relatively small heat capacity of gas and the radiation of heat to the surrounding earth. For example, 1,000 cubic feet of gas of a specific gravity 0.6 (air=1) would carry approximately 20 to 25 British thermal units of heat energy per degree Fahrenheit, whereas it requires approximately 145 British thermal units to raise the temperature of 1 barrel of 39° A. P. I. gravity oil 1° F. Only part of the heat carried by the gas would be effective in heating the oil string, because a large portion of the heat is lost by radiation to the surrounding formations. Bennett<sup>59</sup> found in the Panhandle field, Texas, that by heating the intake gas to 160° to 180° F. the deposition of paraffin could be prevented in the upper 100 to 150 feet of tubing. In some wells where large volumes of gas are circulated enough heat may be supplied by heating the gas to a temperature of 200° or more to prevent paraffin accumulations to a depth of 500 to 700 feet. The use of hot gas to remove paraffin deposited on the face of the producing sand, however, is unsuccessful because a large part if not all of the heat of the gas is dissipated by radiation to the surrounding formations and to the oil in the oil string before the gas reaches the sand.

#### CIRCULATION OF HOT OIL FOR REMOVING PARAFFIN

The circulation of hot oil for removing paraffin from the oil string and sand has proved satisfactory in some fields. Knox<sup>60</sup> reports the successful removal of paraffin and stimulation of production by heating a portion of the production and returning it to the well. When hot oil is returned to the sand through the space between the tubing and casing it rapidly loses its heat to the surrounding formations and, unless large quantities are circulated, seldom reaches the sand at a temperature much above that of the surrounding formations. For example, using the formula given by the writer in the discussion of radiation of heat to the surrounding formations (see p. 28), the circulation or introduction of 10 barrels of oil an hour, heated to 200° F., into a well 1,600 feet deep in the Salt Creek field

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<sup>58</sup> Baker, Warren, Simple Heater Corrects Paraffin Trouble in Gas-Lift Wells: Oil Weekly, vol. 53, No. 26, April, 1929, p. 56. Cold or Hot Gas: Oil and Gas Journal, July 19, 1928, p. 64. Miller, H. C., Gas-Lift Method of Flowing Oil Wells: Bull. 323, Bureau of Mines, 1930, p. 36.

<sup>59</sup> Bennett, E. O., and Sclater, K. C., Some New Aspects of the Gas Lift: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., 1926, pp. 127-128.

<sup>60</sup> Knox, W. J., Cleaning Oil Wells with Heated Oil: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., 1925, pp. 174-178.

through a  $5\frac{3}{16}$ -inch casing, where the average temperature of the surrounding formations is  $75^{\circ}$  F., would result in the oil reaching the sand at a temperature of not more than  $110^{\circ}$  to  $115^{\circ}$  F., not considering the radiation of heat to the returning oil in the tubing. If the oil is introduced through the tubing, the loss of heat will be much less, and the oil will reach the sand at a temperature of approximately  $145^{\circ}$  to  $150^{\circ}$  F. It would be impossible, however, to circulate the oil in this manner with the pumping equipment in the well unless two strings of tubing were used. When hot oil is circulated in a well by being introduced into the casing, the temperature of the oil as it leaves the well through the tubing is misleading. This temperature is partly the result of radiation of heat from the oil going down the casing to the oil coming up the tubing, which occurs in the upper part of the well and is not the result of pumping hot oil from the bottom of the well. If the fact that the well acts as a heat exchanger is considered it can readily be seen that the hot oil flowing down the well will have transferred a great part of its heat to the surrounding formations and the returning oil in the tubing before reaching the sand.

The following method of circulating hot oil was used in the Salt Creek field. Tubing and rods were removed from the well, and the tubing was rerun with jets on the bottom to force the oil out against the sand. The oil was heated to  $160^{\circ}$  to  $180^{\circ}$  F. in a 50-barrel tank equipped with steam coils and was forced down the tubing by a steam pump. Sufficient oil was used to establish circulation, and the oil was returned to the surface through the casing and back into the tank. The oil was circulated at the rate of approximately 75 barrels per hour, and it carried all the loose sand and cavings to the surface where they settled out in the heating tank. The oil production of the well was almost doubled at first and then declined rapidly to an average production of 4 or 5 barrels more than before the hot oil was used, indicating that the hot oil not only removed the paraffin from the face of the sand but also caused a small permanent increase in production. Undoubtedly, the large increase in oil production following the heat treatment of the well was caused by the pressure readjustment in the sand while the oil was circulated, as heat reduced the viscosity of the oil and liberated additional gas from solution in the oil in the sand. The production record of the test before and after circulating the hot oil is given in Table 15. The cost of the method was approximately as follows: Pulling and running tubing, \$50; moving tank and pump to location, \$10; steam, \$20; loss in gravity of circulating oil, \$10; labor, \$20; and miscellaneous, \$10; or a total of \$120. This expense plus the loss in production while the well was shut down was more than the increase in production was worth. Ordinarily, however, the well should not have been shut down for more than three days, which would have reduced the cost of operation and loss in production about one-third. The jet method of washing a sand with hot oil is seldom economical in small wells where only a small increase in oil production is obtained.

TABLE 15.—*Production of test well 3D, NW. ¼ sec. 13, T. 39 N., R. 79 W., at Salt Creek, Wyo., before and after circulating hot oil*

Date	Pro- duc- tion, barrels	Remarks	Date	Pro- duc- tion, barrels	Remarks
Sept. 5	22		Sept. 26	0	
7	23		27	0	Replacing pumping equipment.
8	23		28	46	
9	23		29	36	
10	22		30	30	
11	22		Oct. 1	30	
12	22		2	23	
13	23		3	25	
14	23		4	28	
15	23		5	28	
16	23		6	28	
17	23		7	27	
18	23		8	24	
19	23		9	26	
20	23		10	27	
21	23		11	27	
22	0	Pulling tubing.	12	27	
23	0	Running tubing with jets.	13	26	
24	0	Jets plugged—tubing pulled.	14	27	
25	0	Circulating hot oil—160° F.			

## HOT WATER FOR REMOVING PARAFFIN

The procedure in using hot water to melt and remove accumulated paraffin on the face of the producing sand is similar to that in using hot oil. Water contains approximately twice as much heat energy as oil of the same temperature and therefore reaches the sand at a higher temperature and produces more heating at the sand. This advantage and the fact that in heating the oil the lighter constituents vaporize and are lost, thus decreasing the commercial value of the oil used in the cleaning-out operation, make the use of water preferable. Bechtel and Blenkarn<sup>61</sup> have used this method extensively in pumping wells of the Panhandle district, Texas, and have obtained satisfactory results. The procedure followed after pulling the rods and standing valve was to heat 150 to 200 barrels of water to 190° F. or more in a tank equipped with open steam coils and to run the heated water into the well through the tubing in order to prevent excessive cooling of the water due to radiation in the well. If there is a large quantity of paraffin in the tubing it should be removed with a paraffin hook before the hot water is run into the well. Water produced from the sand or fresh water with lye added was used, because fresh water alone has a tendency to dissolve certain minerals and otherwise damage the producing sand in the Panhandle field. The lye had little or no effect on the paraffin and acted merely as a protective agent for the sand. After the hot water had been added to the well the rods were run and the well pumped.

The results obtained by washing several wells in the Panhandle district with hot water are given in Table 16. It is always advisable to remove the water from the well before it has cooled to the sand temperature. This can easily be accomplished, as the water remains fairly hot for several hours. In districts where the application of

<sup>61</sup> Bechtel, L. L., and Blenkarn, W. O., Water and Lye Solution Cleans Wells of Paraffin: Oil Weekly, May 4, 1928, pp. 39-42.

hot water causes the producing sand to cave and disintegrate the use of compressed air or gas for removing the water and sand is advisable, if compressed air or gas is readily available without too great an expense; otherwise, the expensive operation of pulling the tubing and cleaning out is necessary.

Removing paraffin from the face of the sand and from the sand adjacent to the well with hot water offers a satisfactory and inexpensive method, if the water does not damage the sand or cause sufficient caving to necessitate cleaning out with tools. Cleaning wells by the hot-water method, as described, costs approximately \$30 to \$50 per well, if steam is available from a fired boiler near the well and if the well does not need cleaning with tools after washing. Moving a boiler to the location or cleaning out with tools will greatly increase the expense.

TABLE 16.—*Production record of wells washed with hot salt water in the Panhandle field, Texas*<sup>1</sup>

Well No.	Washing	Date	Hot water used, barrels	Lye, cases	Oil, barrels per day		
					Before	After	Increase
C3	First	Mar. 24, 1928	150	None.	66	60	<sup>2</sup> 6
C4	do	Sept. 28, 1927	225	None.	27	150	123
Do	Second	Jan. 16, 1928	150	6	64	97	33
Do	Third	Apr. 3, 1928	200	None.	15	60	45
C5	First	Apr. 14, 1928	150	None.	18	68	50
D2	do	Oct. 14, 1927	<sup>3</sup> 10	3	70	100	30
H2	do	Nov. 10, 1927	250	<sup>4</sup> 240	30	35	5
H3	do	Jan. 20, 1928	<sup>3</sup> 10	3	14	20	6
J1	do	Feb. 7, 1928	300	6	7	15	8
T2 <sup>5</sup>	do	Oct. 9, 1927	125	None.	170	160	<sup>2</sup> 10
T2	Second	Jan. 12, 1928	200	6	18	78	60
R1	First	Jan. 14, 1928	20	4	25	25	0
R1	do	Oct. 18, 1927	100	None.	60	90	30
W2	Second	Jan. 21, 1928	90	6	26	44	18

<sup>1</sup> Taken from Water and Lye Solution Cleans Wells of Paraffin, by L. L. Bechtel and William O. Blenkarn.

<sup>2</sup> Decrease.

<sup>3</sup> Steam down tubing 3 hours.

<sup>4</sup> Pounds of sal soda.

<sup>5</sup> The well was plugged with paraffin two days before the washing so there was really not the decrease shown.

#### FLAME FOR REMOVING PARAFFIN

The fact that a flame can be maintained at the bottom of small wells if a combustible mixture of gas and air is supplied is sometimes utilized for heating the producing sand. In 1920 Mills experimented with some wells in the Marietta district, Ohio, and succeeded in starting a fire at the sand in one of the wells. The air to maintain combustion was supplied from the sand as the sand was being repressured with air. By a coincidence a combustible mixture of gas and air was entering the well from the sand and this was ignited by lowering oil-saturated burning waste into the well. The well was allowed to burn for 24 hours, then the fire was extinguished by pouring several buckets of water into the well. The oil production of the well was doubled by this heat treatment. After this experiment another well was "burned out" by supplying air from

the surface through tubing and igniting it in the same way. The fire was maintained in the well for several hours, and the subsequent oil production was more than doubled.<sup>62</sup>

The Hope Natural Gas Co.<sup>63</sup> has carried on considerable experimental work in connection with the flame method of removing paraffin from oil wells and has obtained some very satisfactory results. Gas and air are carried to the bottom of the well through tubing, on the bottom of which is a specially designed burner. A pilot burner at the top of the tubing is also kept burning to indicate that a combustible mixture of air and gas is being forced into the well. The flame is ignited by lowering a special lighted fuse into the well. A method using an electrical apparatus for igniting the flame has also been perfected.

The use of a flame at the bottom of the well applies mainly to small wells in which only a relatively small quantity of oil enters the well during the burning operation, although it is theoretically possible to maintain a flame when submerged in oil. The flame heats the sand and removes any paraffin accumulated on it, and it undoubtedly heats the sand for a considerable distance from the well. The tarry residuum of heavy oil and paraffin in the well and on the sand, after the burning, is flushed from the sand and mixed with fresh oil coming from the sand while it is still hot. It is advisable to pour several barrels of fresh oil or kerosene into the well after burning and while the sand is still hot to aid in dissolving the tarry residuum and paraffin material and to pump the well immediately after the burning operation has been completed.

Heating the sand by the flame method undoubtedly removes paraffin and stimulates production, but the intense heat on the face of the sand often loosens large quantities of sand which accumulate in the well and have to be cleaned out with tools or compressed air; the additional expense of cleaning out often offsets the value of the increased production. The burning method is primarily of value in heating the sand of small wells, especially those wells in which heating with a flame does not disintegrate the sand and cause extensive caving; however, caving can be controlled to a great extent by not using too hot a flame or keeping it in operation for too long a time. It is questionable whether this method can be used in wells where screened or perforated pipe is set opposite the sand, for in all probability the flame would seriously damage the screened or perforated pipe.

The expense of applying heat to the producing sand by means of a flame on a property where compressed air and gas are available consists mainly of pulling and running the tubing, laying gas and air lines to the well, and the labor of one man to control the gas-air mixture entering the well. A small portable compressor is often used to supply the air where only compressed gas is available.

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<sup>62</sup> Mills, R. van A., *The Paraffin Problem in Oil Wells: Rept. of Investigations 2550*, Bureau of Mines, 1923, 11 pp.

<sup>63</sup> National Petroleum News, *Hope Tries Flame in Well to Remove Paraffin*: Vol. 15, No. 34, Aug. 22, 1923, p. 91. Smith, T. E., *Successful Method of Melting Paraffin in Wells Found by Hope Company*: Nat. Petrol. News, Jan. 28, 1925, pp. 5-56, 59-60.

## ELECTRIC HEATERS FOR REMOVING PARAFFIN

Electric heaters<sup>64</sup> for heating the oil and sand in a well offer a number of operating advantages that can not be obtained with other methods of heating. A number of different heaters have been developed for use in oil wells, and although it is impossible to describe or refer to the respective merits of each heater, the fundamental information obtained by experimenting with several types has made it possible to draw certain definite conclusions regarding the use of electric heaters for removing paraffin in oil wells.

The most serious trouble experienced in the use of heaters is in the unsatisfactory construction of power leads or cables which can not withstand the severe conditions encountered in the well. The rubber insulation commonly used will resist water satisfactorily, but it deteriorates rapidly when brought in contact with oil; on the other hand, varnish insulation fails when in contact with water.

To the author's knowledge, the most satisfactory cables so far perfected have the following specifications: Cable A, 1-conductor cable, No. 4 A. W. G. strand; 37 strands of 0.036 bare copper wire; 0.094 varnished cambric; two cotton braids varnish-treated; covered with two wraps of 0.02-inch brass tape; weight, 680 pounds per 1,000 feet. Cable B, 1-conductor cable, consisting of 19 strands of No. 16 bare copper wire, double lapped with varnished cambric tape; covered with three asbestos braids covered with special rubber tapes inclosed in steel-armored cable; outside diameter, 54/32 inches. Cable C, 2-conductor cable, consisting of No. 2 stranded bare copper wire insulated with rubber compound; cable filled and jacketed with a special rubber compound; varnished cambric tapes applied with oil-resistant varnish; cotton braids applied with oil-resistant varnish inclosed in steel-tape armor. These three cables have proved more or less satisfactory, but there is still room for improvement in their construction. The cost of such cables ranges from \$0.40 to \$1 per foot, and they can be obtained in any desired length.

It is apparent from the use of different types of cables that the best results are obtained when the cables are attached by clips to the tubing so that it is not necessary for them to support any great amount of weight. T. J. Murphy, of the Westinghouse Electric Co., suggests the use of conduit with the power leads running down through the center, with special tight connectors and joints where the different sections of conduit are fastened together. This suggestion seems to have considerable merit, and in the author's opinion a conduit so made could probably be developed into the most satisfactory type of power "lead-in" yet available.

In the experimental work in the Salt Creek field single-conductor cables were used for one conductor of the electric circuit, and the tubing was used for the other conductor. It was necessary to use a nonmagnetic armor on the cable to prevent loss of power due to

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<sup>64</sup> Bignell, L. G. E., *Electric Heaters Remove Paraffin: Oil and Gas Jour.*, Nov. 14, 1929, p. 42. Powers, John, *Removing Paraffin Deposits from Wells with Electric Heaters: Nat. Petrol. News*, July 4, 1928, pp. 80-82. Wood, F. E., Young, H. W., and Buell, A. W., *Handling Congealing Oils and Paraffin in Salt Creek Field of Wyoming: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng.*, 1927, p. 266.

induction. The line loss was approximately 4 kilowatts when the electric heater was operated at a depth of 2,000 feet. It was found advisable to use a transformer between the power supply and the heater to prevent stray currents that might affect other electrical equipment.

In the construction of electric heaters for use in oil wells the commercial or standard factory-fabricated heating elements are recommended. These units can be obtained in different capacities; but those of 1, 2½, and 5 kilowatt capacity, inclosed in iron sheaths which can be welded or screwed into the heater body, are the most satisfactory.

Heaters often burn out when there is no oil in contact with the heater to convey the heat away from the heating elements, and in some wells sand collects around the heater and causes the elements to burn out. It is advisable, therefore, to use an automatic switch or circuit breaker to shut off the current when the heater becomes overheated.

#### ELECTRIC HEATERS FOR REMOVING PARAFFIN FROM TUBING OR CASING

The use of electric heaters for removing paraffin from the tubing or casing of flowing wells generally is not economical, because the paraffin can be removed from such wells more economically by a paraffin knife, flow devil, or paraffin hook. Although the accumulation of paraffin can be satisfactorily prevented in most pumping wells by correct operation the electric heater can often be used to advantage in pumping wells in which the accumulation of paraffin can not be prevented.

Heaters to prevent the deposition of paraffin in the oil string of pumping wells should be placed on the tubing just below where the paraffin starts to accumulate. Figure 31 shows an experimental heater. The main difficulty in attempting to melt accumulations of paraffin in the oil string with electrical heaters is to obtain a satisfactory heat transfer between the heater and the oil in the necessarily short length of the heater. The heat generated by a 10-kilowatt heater should theoretically raise the temperature of 10 barrels of oil 46° F. in one hour; but the efficiency of heat transfer is usually 50 per cent or less, so that 15 kilowatts of power or more will be required.

The best method of employing the electric heater to remove paraffin from the tubing of pumping wells is to use a heater capable of raising the temperature of the oil almost to the melting point of the wax in order to insure a rapid solution of the wax by the oil, then to operate the heater at intervals of one or two weeks depending upon the rate at which the paraffin accumulates. The continuous operation of a smaller heater, with capacity to heat the oil sufficiently to prevent the deposition of paraffin, is usually too expensive. The following data are given as an example of the cost of operating electric heaters: A 10-kilowatt-hour heater was placed at a depth of 700 feet on the tubing of a 100-barrel well. The oil normally reached the surface at a temperature of 65° to 71° F.; and with the heater in continuous operation the oil reached the surface at a temperature of 83° F., which was sufficient to prevent the deposition

of paraffin. The heater cost approximately \$150 and the cable, \$280; depreciation on this equipment of 100 per cent in 12 months makes a fixed monthly charge for equipment of \$36. At a cost of  $1\frac{1}{4}$  cents per kilowatt-hour for electric power the heater used \$3 worth of

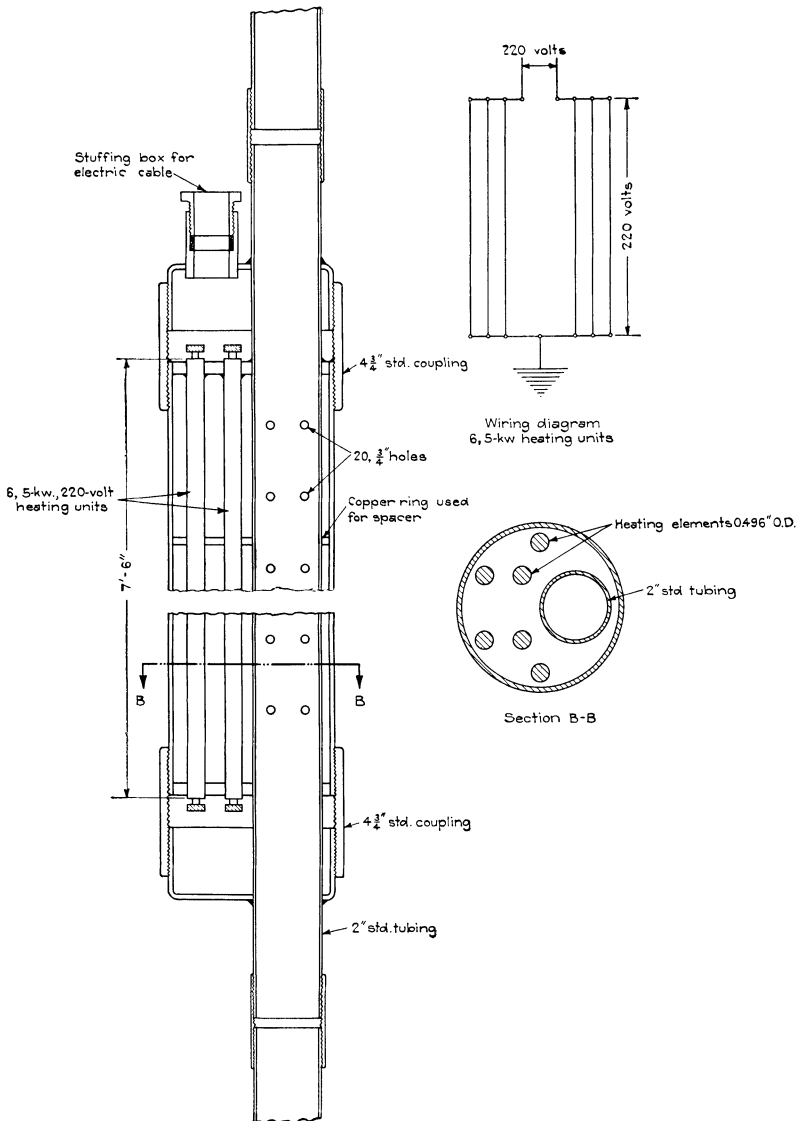


FIGURE 31.—Experimental electric tubing heater

power a day or \$90 a month; maintenance and miscellaneous expense approximated \$10 a month, making a total cost of \$136 a month for continuous operation of the electric heater. Moreover, the rods in the well would in all probability have to be pulled once a month to replace the cups. Replacing the cups would be taken care of when



the paraffin was removed by pulling the rods and using a paraffin hook, but at best there would be an average monthly additional cost of \$14 for pulling the rods and a loss of four hours' oil production which in a 100-barrel well would amount to 16.6 barrels. If oil is worth \$1.50 a barrel the loss due to the well being down would be \$25, making the total monthly cost of continuous electric heating \$175. The same well could be kept in excellent condition by pulling the rods and using a paraffin hook once a week. The cost of pulling rods and using a paraffin hook is \$14.25, making the total cost of such work \$57 a month. If the well lost four hours' production each time the paraffin hook was used this loss would amount to 66 barrels a month, which with oil worth \$1.50 a barrel would be equivalent to a monetary loss of \$99, making a total cost of \$156 a month as against \$175 for continuous electric heating.

A 30-kilowatt-hour heater raised the temperature of the oil in another 100-barrel pumping well from 65° to 105° F. at the tubing head, and the oil was hot enough to dissolve the deposited paraffin in the tubing completely when operated for 24 hours four times a month. The heater cost approximately \$200 and the cable \$280. If the value of this equipment depreciates 100 per cent in 12 months the monthly charge for equipment is \$40. The heater consumed \$9 worth of power each 24 hours or \$36 a month; maintenance and miscellaneous expenses approximated \$10 a month, making a total operation cost of \$86 a month. In addition to this cost the rods would have to be pulled once each month to replace the cups, at a cost of \$14 plus the loss of four hours' production, or 16.6 barrels, which at \$1.50 a barrel would amount to \$25. The total cost of the method would then be \$125, which is \$31 a month cheaper than pulling the rods four times and \$50 a month less than running a small heater continuously, provided there was a loss of four hours' production each time. If no loss of production is incurred as a result of pulling the rods, due to the oil filling up the well, pulling rods would be \$46 a month cheaper than using the intermittent electric heater. Intermittent operation of a larger heater is therefore more satisfactory from a cost standpoint than using a smaller heater continuously. Obviously, however, in wells where there is no loss of daily oil production as a result of pulling the rods or where it is only necessary to remove the paraffin once or twice a month the use of a paraffin hook will be much cheaper.

When electric cables and heaters are perfected so that their useful life is several years or longer, the cost of maintenance and depreciation will be greatly reduced, and they will then offer a more practical solution to the paraffin problem. It is questionable, however, if there is a real need for electric heaters to remove paraffin from the oil string of pumping wells, because paraffin troubles can usually be prevented or modified by operating wells so that the paraffin that does accumulate can be removed economically with the paraffin hook when the rods are pulled for other maintenance requirements.

#### ELECTRIC HEATERS FOR REMOVING PARAFFIN FROM SAND

During 1927 and 1928 the Midwest Refining Co. conducted a number of experiments, using electric heaters to heat the producing sand. Through the courtesy of the company officials, the author was per-

mitted to observe the experiments and was given a copy of the data obtained. One of the test wells, equipped with an electric pyrometer for measuring the temperature at the bottom of the well, is shown in Figure 32. The specifications and construction of the experimental heaters are shown in Figure 33.

Seventeen wells were selected as test wells; but due to operation troubles encountered when the wells were being prepared for the heaters seven of the tests were abandoned, leaving 10 wells in which the heating tests were actually completed. All of the wells were settled pumpers, producing from the second Wall Creek sand, in which gas-pressure restoration operations had been in effect for several years. Before the test the fluid level in all the wells had been maintained at or near the bottom of the sand, and the operators believed that the upper part of the sand was paraffined up. Each test was conducted as follows: The well was cleaned out with a sand pump and a new pump installed on the tubing, after which a daily gage of the production was taken for at least two weeks preceding the use of the heater. The tubing was then pulled, and the pump was examined for any wear or defects and repaired or replaced if necessary before the heater and electric pyrometer were run into the well. The heater was then turned on and a temperature of approximately 225° F. maintained at the bottom of the well for one week while the well was being pumped. The well was then allowed to stand two days without pumping so that hot oil could accumulate and dissolve any paraffin on the face of the sand; then it was pumped for another week with the heat on, after which the tubing in all wells except in tests 5, 6, 9, and 10 (Table 17) was pulled and the heaters removed. The heaters that were left in the four wells were subsequently operated one or more days a week. Well 6A, SW.  $\frac{1}{4}$  sec. 25 (Table 17), was particularly responsive to heating, and whenever the production began to decline the heat was turned on for 48 hours to increase the production again. Table 17 gives the results of the tests. The probable oil recovery without heat was calculated by multiplying the average daily production before heating by the number of days the well produced after heating. This method of comparing the total production

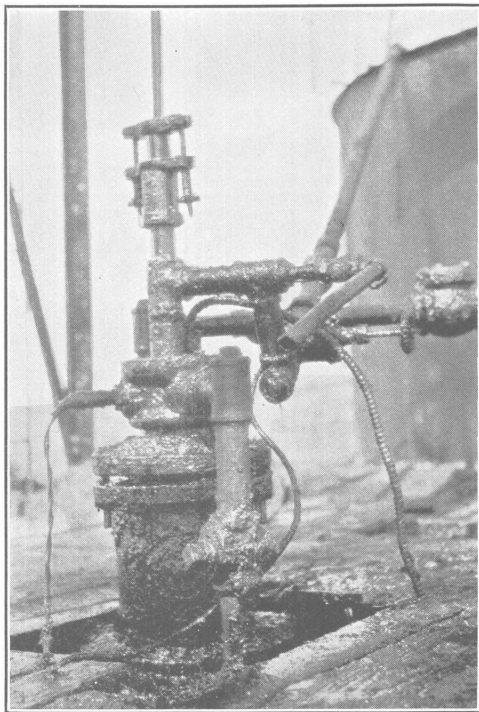


FIGURE 32.—Well equipped with electric heater and electric temperature-measuring apparatus

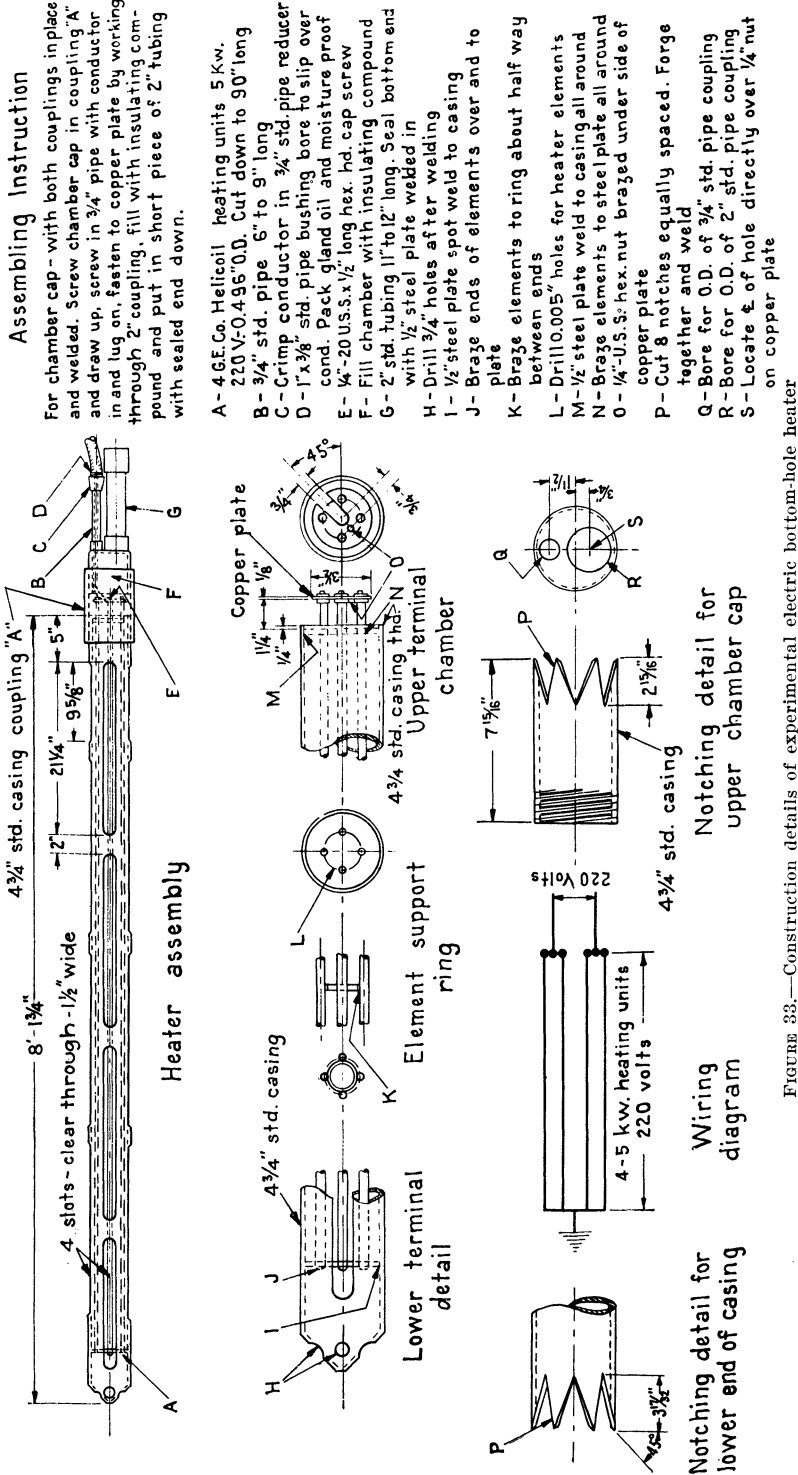


FIGURE 33.—Construction details of experimental electric bottom-hole heater

increase due to heating does not take into consideration the normal decline of the well, and the increase in production due to heating was perhaps greater than the table indicates.

Two wells (tests 4 and 7) whose daily average oil production was less after heating were producing larger quantities of gas than the other wells, and from experience it appears that wells making large quantities of gas are not generally benefited by heating. The indicated loss in production in those wells was probably due to the normal decline of the well and not to the heat treatment. It is apparent from Table 17 that certain wells responded to heating and justified the expense incurred, whereas the production in other wells did not seem to be appreciably affected by heat treatment. Whether or not the production of a well can be stimulated by the application of heat can only be determined by tests on each well. More satisfactory results might have been obtained by the alternate application of heat with the fluid level in the well at the top of the sand.

The temperature of the oil in well 18 A, SE.  $\frac{1}{4}$  sec. 25, the temperature at the casing head, and the well's production after turning off the heat (which had maintained the oil at a temperature of 228° F. for nine days) are given by the curves in Figure 34. The production of the well remained fairly constant after discontinuing the heat treatment, although the temperature of the oil at the bottom of the well declined rapidly for the first 12 hours and then slowly for seven days, when it reached the normal sand temperature. While the heater was in operation the temperature of the oil at the tubing head was 80° to 83° F., and this temperature declined in proportion to the bottom-hole temperature when the use of the heater was discontinued. The tests indicated that, when the temperature at the bottom of a well was 200° F. or more, the oil reached the surface at a temperature warm enough to preclude the danger of any paraffin being deposited in the tubing; in fact, the oil could dissolve any paraffin present in the tubing.

TABLE 17.—Results of heating sand at Salt Creek with electric heaters

Test No.	Well No.	Average daily production before heating		Total kilowatt-hours used in heating	Cost of power at 1½ cents per kilowatt-hour	Average daily production after heating		Probable total recovery if not heated, barrels	Actual total recovery after heating, barrels	Total gain in production after heating, barrels	Increase in production, per cent
		Days	Production, barrels			Days	Production, barrels				
1	19A, NE. $\frac{1}{4}$ sec. 1	19	22.0	4,920	\$73.80	58	25.8	1,276	1,494	218	17.2
2	18A, SE. $\frac{1}{4}$ sec. 25	18	31.6	4,443	66.65	27	32.1	857	867	10	1.6
3	36A, NW. $\frac{1}{4}$ sec. 34	43	14.5	6,080	91.20	89	19.6	1,280	1,744	464	35.2
4	3A, NE. $\frac{1}{4}$ sec. 34	15	30.7	5,924	88.86	110	25.8	3,377	2,835	-542	-16.0
5	4A, SW. $\frac{1}{4}$ sec. 6	8	3.6	9,200	138.00	46	7.1	173	328	155	97.2
6	8A, SW. $\frac{1}{4}$ sec. 6	8	6.0	11,288	169.32	155	8.9	930	1,385	455	48.3
7	16A, SW. $\frac{1}{4}$ sec. 6	8	7.0	9,384	140.76	155	6.0	981	930	-51	-11.1
8	21A, SW. $\frac{1}{4}$ sec. 6	9	3.8	3,596	53.94	154	3.8	585	585	0	0
9	6A, SW. $\frac{1}{4}$ sec. 35	14	3.4	25,172	377.58	219	10.2	745	2,238	1,493	200.0
10	16A, NE. $\frac{1}{4}$ sec. 1	55	8.3	12,960	194.40	61	11.1	506	678	172	33.7

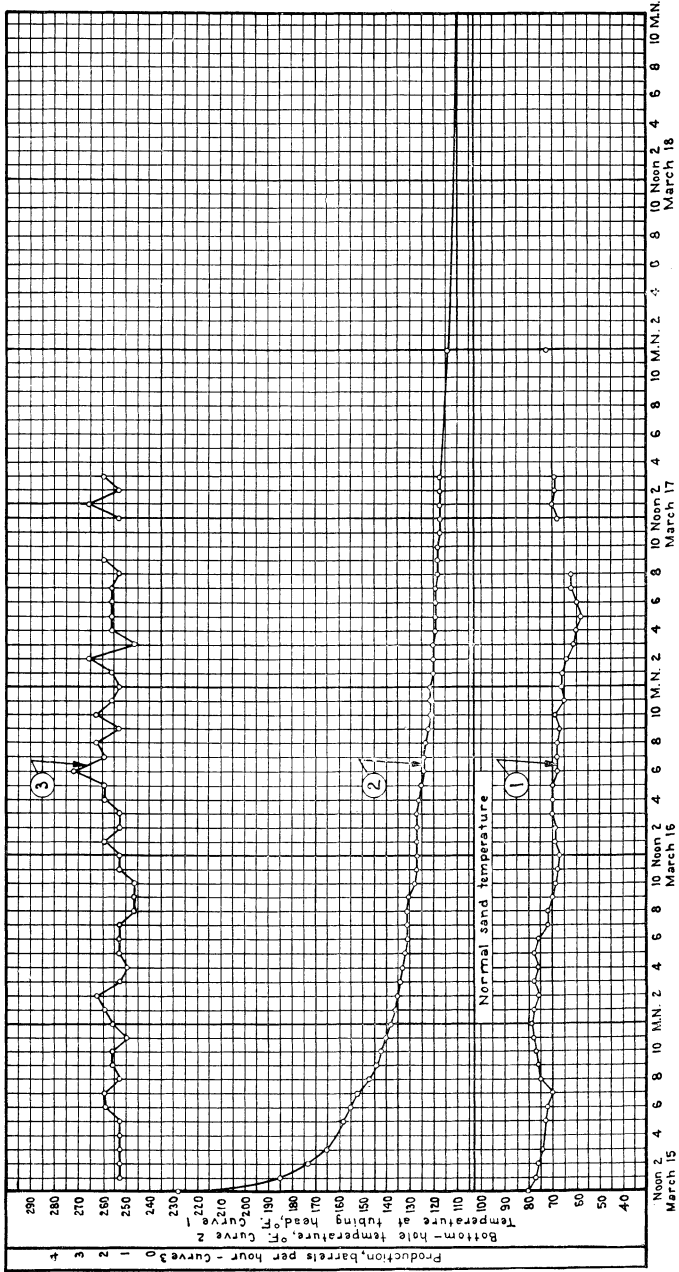


FIGURE 34.—Temperature decline of well after heating nine days at 228° F.

The tests indicated that when an electric heater is used it should be placed at the bottom of the well, where it will heat and clean the sand and stimulate production, instead of on the tubing, where the application of heat will only remove and prevent the accumulation of paraffin in the tubing.

The approximate cost of operating electric bottom-hole heaters similar to those used in the Salt Creek tests is as follows: The heater and cable should be depreciated 100 per cent in a year and a half, although they will probably last somewhat longer. The heater costs approximately \$200 and the 2,000-foot cable \$800, making a total cost of \$1,000 for equipment, which if depreciated 100 per cent in 18 months would amount to \$55.55 a month; and maintenance and installation costs are approximately \$10 a month, making a fixed charge of \$65.55 a month. If the heater was operated for 48 hours twice a month the cost of power at 1½ cents per kilowatt-hour would be \$14.45. The total cost of operating an electric heater is therefore \$80 a month, or \$2.67 a day, or the equivalent of 53 barrels of oil a month at \$1.50 a barrel. To offset the cost of electric heaters, therefore, the production should be increased at least enough to produce \$80 worth of additional oil per month. In the author's opinion the use of the electric apparatus would not be justified, under the conditions stated, if the value of the increased oil production is not more than twice the amount of the expenses incurred in the heating operation; this margin is necessary to offset the cost of experimental work and the expenses incurred in wells that will not respond to the heat treatment.

In test 1, Table 17, the oil production of the experimental well was increased 3.8 barrels a day, which at \$1.50 a barrel amounted to \$5.70. The duration of the test on this well was 58 days, and the increase in production during that time had a gross value of \$330.60; \$73.80 worth of power was used, and the depreciation and maintenance on the heater from the previous estimate were \$111.45, making a total expense of \$185.25. The net profit due to use of electric heaters was therefore \$145.35.

When electric heaters are used for heating the sand and removing paraffin it is advisable to maintain the fluid level at the top of the sand, at least when the heaters are in operation, so that the heat will be transferred to the entire surface of the sand more readily. It is questionable whether temperatures in excess of 200° F. are necessary, as this temperature insures the melting of all paraffin on the face of the sand and for a considerable distance back in the sand from the well. Higher temperatures often cause crumbling of the sand and necessitate a cleaning-out job.

All wells will not give increased oil production after electric heaters are used. Probably, the sand in the wells that failed to respond to heat treatment in the tests was not paraffined up; however, in wells where the face of the sand tended to paraffin up or in wells where the paraffin was accumulating in the sand, the use of electric heaters solved the paraffin problem and stimulated production. The author believes that it is best to use heaters that can be run into the well on the tubing and permanently left in the well as part of the standard equipment, so that the sand can be heated and the paraffin in the tubing removed at proper intervals.

## METHODS FOR INCREASING PRODUCTION OF VISCOUS OILS

The production of the heavy naphthene-base crude oils found in many localities presents problems somewhat similar to those encountered in the production of congealing oils, except that it is necessary to lower the viscosity of the naphthene-base oils rather than to prevent the separation of wax from solution in the oil. Application of heat lowers the viscosity of the naphthene-base oil and often results in large increases in production. Some of the previously discussed methods of heating the producing sand and the oil string, used to remove and prevent paraffin, also apply to reducing the viscosity of heavy naphthene-base oils. The effect of temperature on the viscosity of heavy naphthene oils is shown by the curves in Figure 35.

### STEAM FOR REDUCING VISCOSITY

The use of steam coils in natural flowing wells decreases the viscosity of the oil in the oil string and reduces the resistance to the flow of oil into the well, which often results in an increase in production. The use of steam, however, is limited, because steam can not be used at great depths owing to loss of heat by radiation and to the high pressure required to return the condensed steam to the surface. It is generally inadvisable to allow steam or condensate to mix with heavy oils because of the stable emulsions usually formed.

### HEATED GAS FOR REDUCING VISCOSITY

In gas-lift wells the use of heated gas results in heating the oil in the oil string to a depth of several hundred feet, thereby lowering the viscosity of the oil and the pressure required to lift it from the well.<sup>65</sup> It is very doubtful, however, if any of the hot gas actually reaches the sand, except in the very shallow wells. The temperature of the oil leaving the well often misleads the operator into thinking that he is heating the sand, whereas actually the oil flowing from the well is being heated in the upper part of the oil string, and very little or no heat is reaching the sand. This failure to heat the sand is usually true of all methods of heating where the heat is introduced between the casing and tubing.

### ELECTRIC HEATERS FOR REDUCING VISCOSITY

The use of electric heaters at the bottom of the well is probably one of the most promising methods of increasing the production of viscous oils. Heaters similar to those used for heating the sand and removing paraffin could be used for this work, and the cost of operation would be approximately the same. This field of investigation merits considerable study as to its usefulness in the viscous oil fields.

In general, any method of heating that can be economically applied to the oil while the well is in operation will benefit the production of viscous oils.

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<sup>65</sup> Miller, H. C., *Mechanical Installations for Gas-Lift Pumping as Practiced in California Oil Fields: Petrol. Devel. and Technol.*, Am. Inst. Min. and Met. Eng., 1927, pp. 85-100.

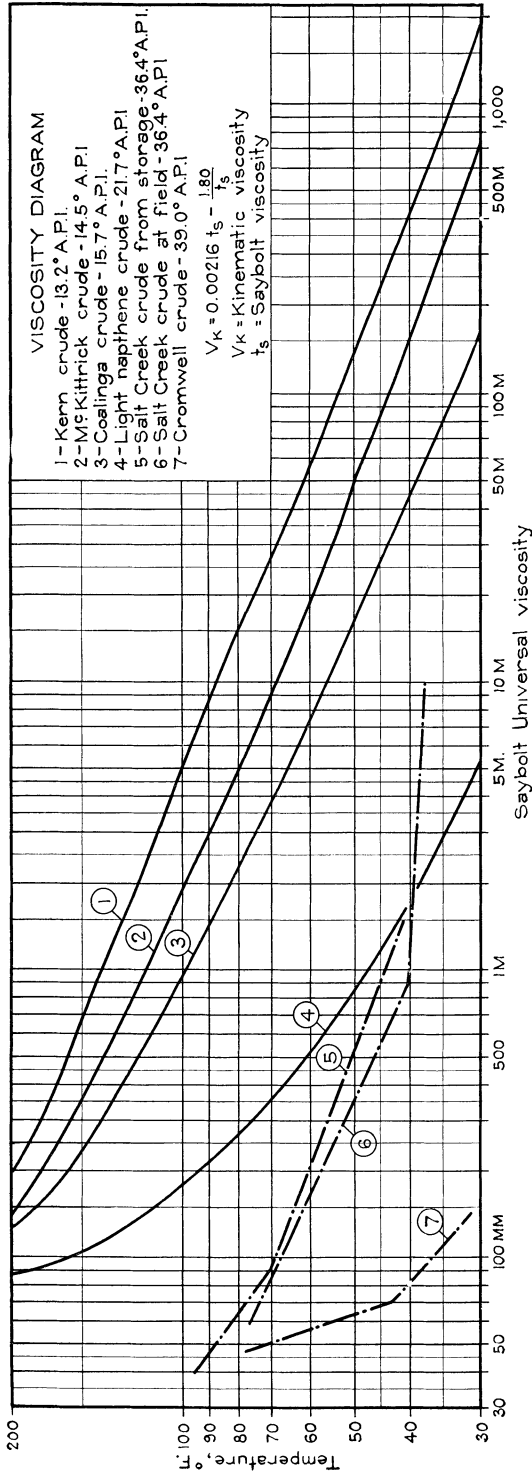


Figure 35.—Viscosity of naphthene and mixed base crudes at different temperatures



**GAS AND VOLATILE LIQUID FRACTIONS OF CRUDE OIL**

When natural gas or the volatile fractions of crude oil, such as propane, butane, and pentane, are dissolved in heavy viscous oils, the viscosity of the oils is lowered appreciably.<sup>66</sup> The effect of dissolved stabilizing plant gas (propane, butane, and pentane) on the viscosity of crude oils is shown in Figure 20 (p. 59). In the production of viscous oils by the gas lift it is often good practice to use "wet" gas that contains a large percentage of natural gasoline, because vapors of the liquid constituents in the gas are absorbed by the oil and lower its viscosity and reduce the intake-gas pressure required to flow the oil to the surface.

When gas is returned to the sand, as in repressuring or gas-drive operations, it is also advisable to return as much of these liquid constituents of natural gas to the sand as possible.

**CAUSES AND METHODS FOR PREVENTING CONGEALING OF OIL AND ACCUMULATION OF PARAFFIN IN FIELD FLOW LINES AND PIPE LINES**

Two distinct pumping problems are encountered in the pipe-line transportation of crude oil containing a high percentage of wax. One is due to the congealing of the oil when cooled, and the other is due to the deposition of paraffin on the walls of the pipe; in both, cooling of the oil is the major factor responsible for the troubles encountered.

When crude oil containing wax is cooled the wax that separates from solution rapidly increases the viscosity of the oil; and if sufficiently cooled the oil becomes very viscous or jellylike, and it is impractical to supply sufficient pressure to move it through the pipe. Cooling of the oil during transportation through pipe lines is due primarily to climatic conditions and radiation of heat to the surrounding earth. If oil is kept in motion the temperature at which it congeals is much lower than when it is allowed to stand still, as the crystals of wax interlock and form a supporting network which prevents movement of the liquid constituents of the oil.

Heating mixed-base crude oils to a temperature of 140° to 160° F. lowers the pourpoint or temperature at which the oil congeals 15° or 20° F. for a period of time usually lasting from 24 to 48 hours. This phenomenon, which has been discussed previously (p. 11), has made it possible to transport oils during the winter months that congeal at relatively high temperatures, such as the Panhandle crude, which has a pourpoint of 55° to 66° F.

Paraffin will only accumulate on the walls of the pipe when the oil has been cooled to a point where some of the wax has separated from solution and certain other conditions exist; otherwise, the paraffin particles remain suspended in the oil and are carried along with it. As in casing and tubing, the most favorable condition for the accumulation of paraffin on the interior walls of flow lines occurs where the oil is alternately flushed from the line by gas that accom-

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<sup>66</sup> Dow, D. B., and Calkin, L. P., Solubility and Effects of Natural Gas and Air in Crude Oil: Rept. of Investigations 2732, Bureau of Mines, 1926, 13 pp. Beecher, C. B., and Parkhurst, I. P., Effect of Dissolved Gas Upon the Viscosity and Surface Tension of Crude Oil: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., 1926, pp. 57-63.

panies the oil. Then the walls are alternately coated and drained, and the suspended wax particles in the film of oil become firmly attached to the pipe. Another condition that causes deposition of paraffin in trunk pipe lines and some flow lines is the slow movement of oil through cold lines. When the oil is moving slowly through the lines, as in streamline flow, there is a film of oil in contact with the pipe which moves very slowly and is cooled to the approximate temperature of the pipe. If the temperature of the pipe approaches or is below the congealing point of the oil the film contains a large quantity of free wax, and the movement is so slow that the wax becomes attached firmly to the pipe. If the flow of oil approaches or is turbulent only a very thin film of oil moves in a streamline manner along the pipe walls, but it has a higher velocity than the film along the wall when the entire body of oil is moving in streamline flow.<sup>67</sup> During turbulent flow, therefore, the free paraffin remains suspended in the oil, and only a very small or negligible quantity accumulates on the pipe.

Several examples of the rates of deposition of paraffin for various conditions of flow were studied, and the most conclusive data were obtained on two parallel 4-inch lines running from the Long Beach field, California, to a near-by refinery. The oil was an intermediate or mixed-base crude. One of the lines was operated at full capacity, with the oil flowing through at least part of the line under conditions favoring turbulent flow; the second line was operated continuously but carried only a small quantity of oil under conditions favoring streamline flow. This condition existed for several months during the winter until it was necessary to increase the quantity of oil transported by the second line. It was then found that in the last few miles of the line paraffin had accumulated and restricted the size of the line to about three-fourths inch in diameter, causing the oil to move in turbulent flow. After the line was cleaned out by pumping hot distillate through it a much larger quantity of the same oil was pumped through the line at or approaching a turbulent flow, and a year later when the line was cut for inspection no paraffin had accumulated. The other line operating with a large quantity of oil at all times had no paraffin trouble. Engineers of the Sinclair Pipe Line Co. have also observed similar conditions in some of their Oklahoma lines.

#### FIELD FLOW LINES

Usually it is uneconomical to bury field-flow lines deep enough to protect them from the effect of atmospheric temperature changes, and they are laid on the surface of the ground where they will be easily accessible. Surface lines are subjected to severe temperature changes that cause congealing of the oil and precipitation of paraffin from solution in the oil, so that under favorable conditions paraffin accumulates on the pipe.

Intermittent flowing of oil and gas through lines should be avoided to prevent the accumulation of paraffin. If flowing wells produce intermittently the flow of oil through the lines can be maintained steady by placing gas separators adjacent to the well, with provisions

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<sup>67</sup> Heltzel, W. G., Fluid Flow and Friction in Pipe Lines: Oil and Gas Jour., Oct. 7, 1926, pp. C-158-C-171.

for cleaning out the short line from the well to the separator. Often after the oil leaves the gas separator, especially when the separator is operated at a pressure of 15 or more pounds per square inch, enough gas is liberated from the oil to cause intermittent flushing of the lines. Such flushing can sometimes be prevented by maintaining 10 or 15 pounds pressure per square inch on the line by means of a pop valve on the line at the tanks, thus holding the gas in solution in the oil until it reaches the tanks. Moreover, the oil from many pumping wells contains considerable gas in solution, which in many instances causes flushing of the flow lines, and this condition can also be prevented by placing pop valves or pressure regulators on the flow line at the tanks. These precautions will usually prevent the deposition and accumulation of paraffin in the flow line, except in very small wells where the deposition is due to the very slow movement of the oil. Accumulation under such conditions can seldom be prevented, and the paraffin must be removed

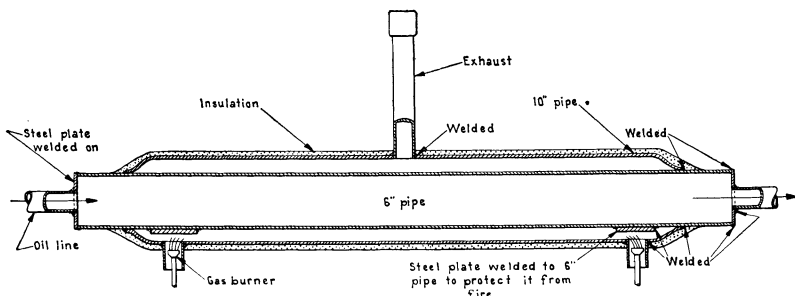


FIGURE 36.—Gas-fired heater for field flow lines

at regular intervals. Congealing of the entire body of oil in flow lines can not be prevented except by heating or preventing the cooling of the oil.

#### STEAM

The most common method of removing paraffin and congealed oil from flow lines is by the use of steam. In the Salt Creek field, Wyoming, for example, many of the field lines are blown out several times during the winter to remove the accumulated paraffin, using steam from boilers at the tank stations throughout the field. In the Panhandle district, Texas, steam lines are often laid parallel with the flow lines, and both are inclosed in wooden boxing;<sup>68</sup> then, whenever it is necessary to remove paraffin or congealed oil, steam is run through the lines. Another method used in the Panhandle field is to inject steam directly into the flow lines at a point near the wellhead. Steam is economical if readily available from boilers near the well; otherwise, the cost is usually prohibitive. The use of steam, however, is generally an expensive method of cleaning lines, due to the labor required to connect the lines to the boilers and the cost of the steam.

<sup>68</sup> Vietti, W. V., and Oberlin, W. A., Problems Encountered in Handling Panhandle Crude: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., 1927, pp. 269-284.

## GAS-FIRED HEATERS

Gas-fired or steam heaters placed in the lines near the wells are economical appliances for heating the oil to prevent it from congealing and for dissolving accumulated paraffin in flow lines. Figure 36 shows a drawing of a gas-fired flow-line heater.

## ELECTRIC HEATERS

The electric heaters in Figures 37 and 38 were used on the flow lines of some of the wells in the Salt Creek field, Wyoming. The

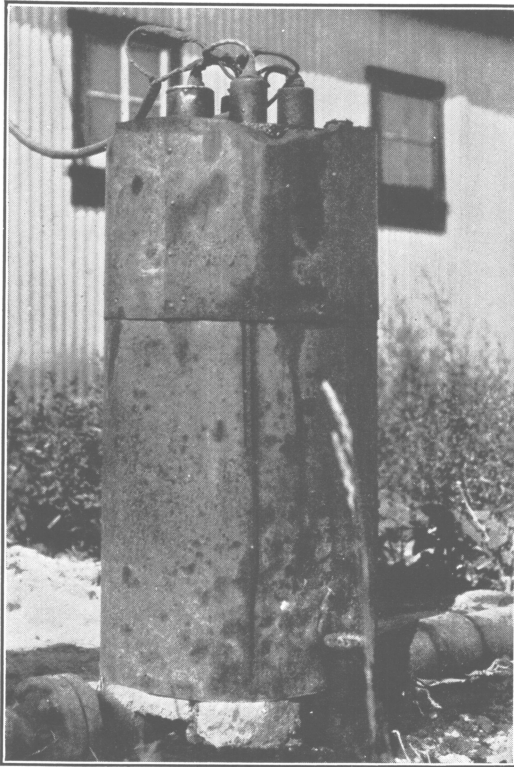


FIGURE 37.—Electric flow-line heater

heaters had a capacity of 5 and 10 kilowatt-hours, depending upon the amount of oil to be heated. Experience showed that it was advisable to equip the heaters with automatic cut-outs to prevent them from burning out, due to overheating, if movement of the oil should stop. Electric heaters can be used to particular advantage on isolated wells when they are equipped with automatic temperature regulators which turn on whenever the temperature of the oil approaches the congealing point. The approximate cost of operating a 10-kilowatt-hour heater is \$3.60 a day and that for the 5-kilowatt-hour heater \$1.80, based on power costs of  $11\frac{1}{2}$  cents per kilowatt-hour. Generally it is unnecessary to operate the heaters for more

than a portion of the day, as the lines are usually heated by the sun enough to prevent congealing of the oil. The depreciation and maintenance charges on the heaters are small, as they require little attention and have a life of three or four years. A heater similar

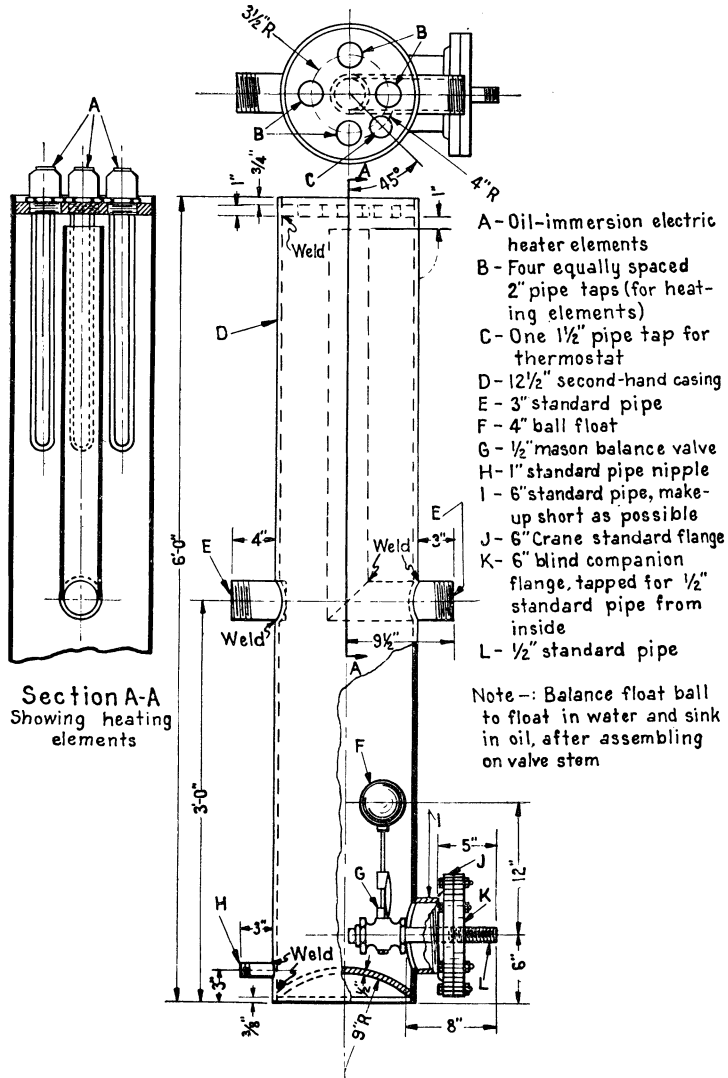


FIGURE 38.—Electric flow-line heater

to that shown in Figure 37, equipped with automatic cut-out and temperature regulator, costs approximately \$200; and depreciating the cost of the heater in three years makes the fixed cost, including interest on the money invested and maintenance, about \$7 per month, or \$0.24 a day. The total daily cost of operating the heaters is there-

fore \$3.84 for the 10-kilowatt-hour heater and \$2.04 for the 5-kilowatt-hour heater. This high cost of operation makes the use of heaters uneconomical except on the flow lines of isolated wells.

SCRAPERS

Pipe-line scrapers are valuable in removing paraffin from flow lines. Flow lines should be laid without sharp bends so that scrapers can be run through them when experience has shown that the flow lines are likely to paraffin up. Figure 39 shows a common type of pipe-line scraper. Scraper boxes should be placed at each end of the line so that scrapers can be inserted and forced through the lines without wasting oil. A very satisfactory scraper box is shown in Figure 40. To insert the scraper, valve 3 is opened and valves 1 and 2 are closed, the bull plug is removed, and the scraper is placed in the scraper chamber. The bull plug is then replaced, and valves 1 and 2 are opened and valve 3 is closed to permit the oil to force the scraper along the line. While the scraper is being run, valve 3 of the scraper box at the receiving end of the line is closed, and valves 1 and 2 are open so that the scraper will enter the chamber with the paraffin ahead of it. Valve 3 is then opened, valves 1 and 2 are closed, and the scraper is removed. Where considerable hard paraffin has accumulated ahead of the scraper the bull plug on the receiving scraper box is usually removed and the paraffin run into a pit. Scrapers should be used often enough to prevent a large accumulation of paraffin. Scraping is a very economical method for removing

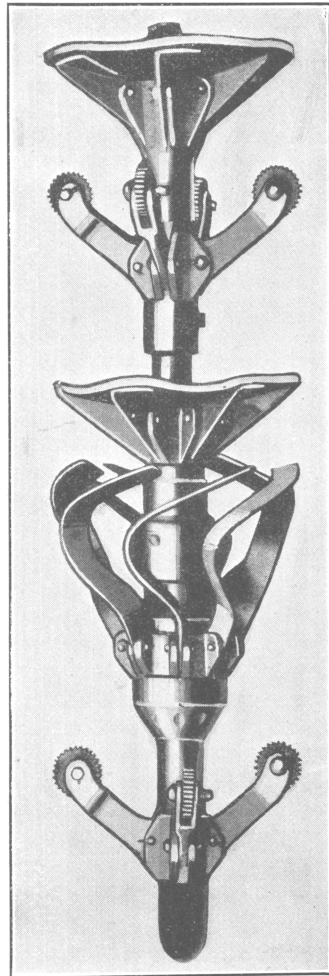


FIGURE 39.—Pipe-line scraper

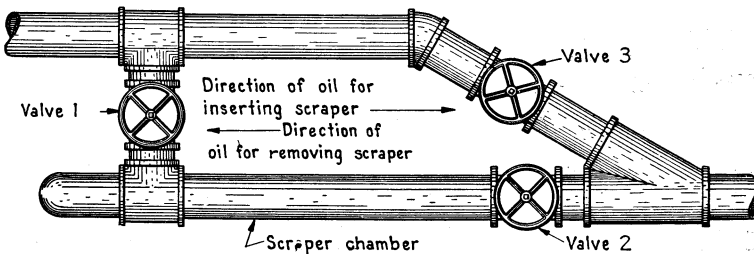


FIGURE 40.—Scraper box for inserting scraper in field flow lines

deposited paraffin from flow lines but is of no value for removing oil that has completely congealed in the line.

#### TANKS ADJACENT TO WELLS

Placing small flow tanks of 250 to 500 barrels capacity adjacent to the wells tends to eliminate the problem encountered when oil congeals in areas where winters are long and severe. The oil can then be run from the tanks during the day when the temperature has moderated; or, if necessary, the oil may be heated in the tanks before moving. The short flow line connecting the tank and well should be equipped so that a scraper on the end of a rod can be used by the pumper before enough paraffin accumulates to plug it. No trouble should be encountered from congealing oil in such a short line.

#### TRUNK PIPE LINES

In the transportation of crude oil in trunk pipe lines trouble is often encountered from the high viscosity or congealing of the crude as a result of cooling. The viscosity of paraffin or mixed base crudes that contain an appreciable quantity of wax increases slowly when they are cooled until a point is reached where a portion of the wax is separated from solution, after which any additional cooling causes a sharp increase in viscosity. This fact is illustrated by the curves in Figure 35 (p. 99). If congealing oils are not kept in motion after the critical temperature is reached they become solid and congeal due to the formation of a network of wax crystals which tend to support the liquid constituents of the oil and if cooled sufficiently become semisolid even if kept in motion. It is almost impossible to reestablish movement of oil in a pipe line after the oil has congealed unless heat is applied.

#### BLENDED WITH LIGHTER CRUDE OILS AND WASTE PRODUCTS FROM GASOLINE PLANTS

The viscosity of crude oils can be reduced and congealing prevented by mixing the oil with lighter crudes or with lighter fractions of the crude oil. In many areas natural gasoline and distillate are mixed with the crude to allow transportation from the field to the refinery and at the same time reduce the viscosity or prevent congealing of the oil. Undoubtedly, the waste products from the manufacture of natural gasoline, such as propane and butane, which are commonly referred to as weathering-plant gas, could be utilized economically for reducing the viscosity of heavy oil when they are available at little or no cost. The effect of dissolved weathering-plant gas on reducing the viscosity of Salt Creek crude is shown by the curves in Figure 20 (p. 59).

#### HEATING OIL

Heating is the method generally used for reducing the viscosity of viscous and congealing oils where it is impractical to mix them with lighter oils or light petroleum products. The viscosity-temperature curves of a number of congealing and viscous oils are shown in Figure 35 (p. 99). The heating of paraffin or mixed-base crude

oils often materially lowers the pourpoint or temperature at which the oils congeal for a period of time, depending upon the characteristics of the oil.<sup>69</sup> Laboratory experiments indicate that the gums, resins, and asphaltic material which also separate from the oil on cooling retard the crystallization of wax when redissolved by heating and are responsible for temporarily lowering the pourpoint. Vietti found that heating Texas Panhandle crude to 140° F. lowered the congealing or pourpoint from 55° to 0° F. for 12 to 20 hours and that several days elapsed before the oil regained its original pourpoint. Crude oils, such as Pennsylvania and West Virginia paraffin-base oils and the Lance Creek and Grass Creek crudes of Wyoming, that contain very small quantities of gums, resins, asphaltic material, and other similar substances, do not show as definite a lowering of the pourpoint after heating as the intermediate or mixed-base crudes.

Heltzel<sup>70</sup> states that it is often more economical to use larger lines and pump the oil cold when moderately viscous oils are being handled than to use small-diameter lines and heat the oil. If conditions of turbulent flow can be maintained through the lines, the viscosity of oil does not affect the pumping pressure, as friction is independent of viscosity under conditions of turbulent flow; only in streamline flow is viscosity an important consideration. However, highly viscous oils can not be pumped with sufficient velocity through practical-size lines, with economical pump pressures, to establish turbulent flow, and heating must be substituted. The conditions necessary to establish turbulent flow can be determined from Durand's<sup>71</sup> modification of the D'Arcy formula:

$$p = \frac{f o u^2 L}{288 g D}$$

where

$f$  is a function of  $\frac{D v o}{u}$

$u$  = viscosity, absolute units,

$o$  = density, pounds per cubic foot,

$D$  = inside diameter of pipe, feet,

$L$  = length of pipe, feet,

$v$  = velocity, feet per second, and

$p$  = loss in pressure head, due to friction, pounds per square inch.

The critical point where the régime of flow changes from streamline to turbulent flow occurs near the point where the ratio  $\frac{D v o}{u}$  has a value of 2,000. The transition between streamline and turbulent flow, however, is not sharp, and it is considered better to assume streamline flow for all conditions where the value of the ratio  $\frac{D v o}{u}$  is less than 2,500.

It is advisable to heat congealing or viscous oils only to the point where the mean viscosity of the oil between pump stations will be low enough to permit the available pump pressures to establish tur-

<sup>69</sup> Vietti, W. V., and Oberlin, W. A., Problems Encountered in Handling Panhandle Crude: Petrol. Devel. and Technol., Am. Inst. Min. and Met. Eng., 1927, pp. 275-278.

<sup>70</sup> Heltzel, W. G., Fluid Flow and Friction in Pipe Lines: Oil and Gas Jour., p. C-168, Oct. 7, 1926, revised and republished Oil and Gas Journal, June 5, 1930, p. T-203.

<sup>71</sup> Heltzel, W. G., Work cited; Durand, W. F., Hydraulics of Pipe Lines: Jour. Elec. vol. 44, 1920, pp. 434-439; Barrett, L. L., Pipe-Line Transportation of Hot Oil: Chem. and Met. Eng., vol. 24, No. 26, p. 1148.



bulent flow, or at least prevent congealing of the oil. During the winter months in some areas it is advisable to heat the oil high enough at intervals of several stations to lower the pourpoint.

Heltzel<sup>72</sup> points out that heating oil above the point necessary to reduce the viscosity enough to establish turbulent flow results in a very minor reduction in pump pressures and only supplies additional heat that is lost in the first few miles of line. Heating congealing oils will not prevent solidification of the oil if the line is shut down for any length of time, but it will insure a lowering of the pourpoint for 24 to 72 hours. When it becomes necessary to shut down a line carrying congealing oils, in which the temperature will be below the pourpoint of the oil, it is advisable to flush the line with a noncongealing crude oil, gasoline, naphtha, gas oil, or water. In starting a line to carry congealing oil it is customary to pump hot water or noncongealing oil through the line to heat it, starting with cold liquid and increasing the temperature gradually.

Although paraffin or mixed base crude oils seldom require heating above 130° F. to reduce the viscosity and prevent congealing, heavy naphthene oils must often be heated to 160° to 180° F. to pump them at all. In many instances it is impractical to heat such oils sufficiently to reduce the viscosity to the point where turbulent flow can be maintained. The loss of heat is approximately proportional to the difference in temperature between pipe and the surrounding earth or air, and the hotter the oil the more heat is lost until a point is reached where it becomes more economical to increase the pumping pressure than to increase the temperature of the oil.

#### METHODS FOR CALCULATING CAPACITY AND HEAT LOSSES OF HEATED PIPE LINES

A discussion of pipe-line design would justify a report in itself, so that an attempt will be made to discuss only the calculations involved in determining the capacity and heat losses of a simple line. In the solution of problems relative to heating of oil and spacing of pump stations for viscous or congealing oils the following steps are necessary:

1. The construction of a curve showing the temperature of the oil as a function of distance traveled.
2. The construction of curves showing the viscosity and density of the oil function of distance traveled.
3. The construction of curves showing the values of the ratio  $\frac{Dvo}{u}$  and of  $f$  as a function of the distance.
4. The construction of a curve showing the elevation or profile of the line expressed in pounds per square inch pressure equivalent to the feet of oil head for that elevation as a function of distance.
5. The construction of a curve showing the pressure in the pipe line as a function of the distance. This curve is the hydraulic gradient. The point of intersection of the hydraulic gradient with the profile pressure curve or the line representing zero pressure gives the location of the next pumping station.

To calculate the temperature of the oil at any point in the line a logarithmic mean-temperature formula similar to that generally

<sup>72</sup> Heltzel, W. G., Fluid Flow and Friction in Pipe Lines: Oil and Gas Jour., Oct. 7, 1926, p. C-168, revised and republished in Oil and Gas Jour., June 5, 1930, p. T-203.

used in many heat-transfer calculations is satisfactory, provided the heat loss is assumed to be directly proportional to the difference in temperature between the pipe and the surrounding medium. The following formula used by several major pipe-line companies, which is derived from the fundamental theory of heat transfer, is believed to be accurate enough for the problem:

$$\log_{10} \frac{t_1 - t_a}{t_x - t_a} = \frac{600 d K X}{W Q C},$$

where

$t_1$  = liquid temperature at hot end of line, ° F.,

$t_a$  = air temperature (the mean, for the time of year in question, should be used in the case of buried lines), ° F.,

$t_x$  = liquid temperature at any distance  $X$  miles from hot end of line, ° F.,

$d$  = outside diameter of pipe, inches,

$Q$  = rate of flow, 42-gallon barrels per hour,

$W$  = weight of liquid, pounds per 42-gallon barrel,

$C$  = specific heat of liquid, B. t. u. per pound per 1° F., and

$K$  = coefficient of heat loss, B. t. u. per hour per square foot of outside pipe surface per 1° F. difference in temperature between liquid and air.

The formula can also be expressed as:

$$t_x = (t_1 - t_a) 10^{-\left[\frac{600 d K X}{W Q C}\right]} + t_a,$$

or

$$X = \frac{W Q C \log_{10} \left[ \frac{t_1 - t_a}{t_x - t_a} \right]}{600 d K}$$

The following statement by a prominent pipe-line engineer with regard to the use of a formula of this type summarizes the possible errors:

The entire accuracy of the assumption of proportionality between heat transfer and temperature difference may perhaps be questioned, but ordinary experience in pumping hot oil is apparently not sufficient to establish any better basis for calculation on account of enormous variations in some of the "constants" of the formula, especially the coefficient of heat transfer  $K$ .

In any given case there is also a question as to the proper value to assign to  $t_a$ . In considering a buried line,  $t_a$  has sometimes been taken as the ground temperature some distance from the line and frequently as the mean air temperature at the time of year under consideration. The ground may be warmer than the air during some months, due to absorption of direct radiation from the sun, whereas the ground temperature tends to be rather uniform for a considerable depth, and the value of  $t_a$  can probably be estimated to within 10° F. Solar radiation would be important in a bare line laid above ground; but such lines are not ordinarily used, and the error resulting from estimating the value of  $t_a$  would usually be on the side of safety.

The principal error in using the formula is in the selection of proper value for  $K$ . Barrett<sup>73</sup> suggests a value of 0.9 for well-drained soil, but this value is somewhat high for warmer climates such as California, Oklahoma, and Texas. A value of 0.4 for summer conditions and 0.8 for winter conditions gives values closer to actual operating results. In wet, poorly drained soils  $K$  would have a

<sup>73</sup> Barrett, L. L., Pipe-Line Transportation of Hot Oil: Chem. and Met. Eng., vol. 24, No. 26, 1920, p. 1148.

value between 1 and 1.5 and of approximately 2 for bare pipe exposed to the air. Values of  $K$  for lines insulated with 85 per cent magnesia laid above ground should be between 0.4 and 0.5.

A specific example to illustrate the application of the curves for determining the location of pump and heating stations when a viscous naphthene-base crude is handled is given in the following problem. Assume that a 14.5° A. P. I. gravity oil is being pumped through a line with an outside diameter of 8 inches and an inside diameter of 7.62 inches; the rate of pumping is 25,000 barrels per day; the mean air temperature is 70° F.; the available pump pressure is 800 pounds per square inch; and the heater capacity is sufficient to raise the temperature of the oil to 160° F. at the pump station. To calculate the temperature-distance curve it is advisable to divide the line into 2-mile sections and compute the temperature drop for each section by using the heat-loss formula. The temperature at the end of the first 2 miles would be:

$$\begin{aligned}
 t_x &= (t_1 - t_a) 10^{-\left[\frac{600 d K X}{W Q C}\right]} + t_a \\
 t_1 &= 160^\circ \quad X = 2 \\
 t_a &= 70^\circ \quad W = 338 \\
 d &= 8 \text{ inches} \quad Q = 1,042 \\
 K &= 0.4 \quad C = 0.5 \\
 t_x &= (160 - 70) 10^{-\left[\frac{600 \times 8 \times 0.4 \times 2}{338 \times 1,042 \times 0.5}\right]} + 70 \\
 t_x &= 155^\circ \text{ F.}
 \end{aligned}$$

The temperature-gradient curve in Figure 41 was obtained by repeating the calculation for 2-mile intervals.

The viscosity and density of the oil at various temperatures must be obtained from laboratory data, and from these data and the temperature-gradient curve the absolute viscosity and the density-gradient curves can be calculated. If the viscosity is given in Saybolt Universal time it can be converted into absolute viscosity in foot-poundal-second units ( $u$ ) by the following formula:

$$\text{where} \quad \frac{u}{o} = 0.00000237 - \frac{0.00194}{t}$$

$o$  = density, pounds per cubic foot, and  
 $t$  = Saybolt Universal time, seconds

If the density of the oil is given in degrees A.P.I., it can be converted into density in pounds per cubic foot ( $o$ ) by the following equation:

$$o = \frac{64.2 \times 141.5}{131.5 + ^\circ \text{A.P.I.}}$$

The curve showing the value of the ratio of  $\frac{Dvo}{u}$  is then calculated:

$D$  = inside diameter of pipe, feet,  
 $v$  = velocity of oil, feet per second,  
 $o$  = density of oil, pounds per cubic foot, and  
 $u$  = absolute viscosity, foot-poundal-second units.

The curve showing the value of the friction coefficient  $f$  is then constructed by Durand's<sup>74</sup> formula,  $f = \frac{64}{Dvo} u$ , which holds true for

<sup>74</sup> Durand, W. F., *Hydraulics of Pipe Lines*: Jour. Elec., vol. 44, 1920, pp. 434-439.

streamline flow where the value of the ratio  $\frac{Dvo}{u}$  is less than 2,000.

The value of  $f$  for that part where the ratio  $\frac{Dvo}{u}$  is greater than 2,000 must be taken from experimental data and that given by Heltzel.<sup>75</sup> The data given in Figure 42 are considered to be reliable.

A curve showing the ground elevation at each point on the line expressed in pounds per square inch equivalent to the static head

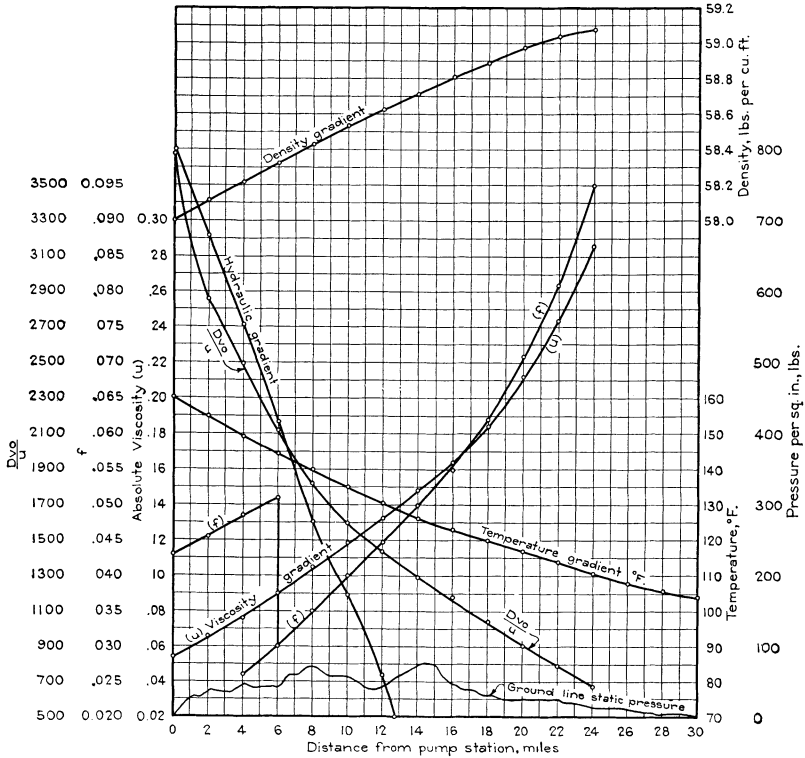


FIGURE 41.—Calculation necessary for designing heated-oil pipe line

of oil for that elevation above that of the pump station is then drawn. This curve can be calculated by the following formula:

$$P_s = E \times 0.4332 \times \text{specific gravity of oil,}$$

where

$P_s$  = static pressure of oil equivalent to elevation of point on pipe line, and

$E$  = elevation of point on pipe line, feet above pump station.

The curve showing the pressure in the pipe line, or the hydraulic gradient as a function of distance, can then be calculated using the

<sup>75</sup> Heltzel, W. G., Fluid Flow and Friction in Pipe Lines: Oil and Gas Jour., Oct. 7, 1926, pp. C-168-C-170, revised and republished in Oil and Gas Jour. June 5, 1930, p. T-203.

formula  $P = \frac{fo v^2 L}{288gD}$  developed from Durand's<sup>76</sup> modification of the D'Arcy formula:

$P$ =loss in pressure head due to friction, pounds per square inch,  
 $f$ =coefficient of friction,  
 $o$ =density of oil, pounds per cubic foot,  
 $v$ =velocity, feet per second,  
 $L$ =length of pipe, feet,  
 $g$ =acceleration due to gravity (32.2 feet per second), and  
 $D$ =inside diameter of pipe, feet.

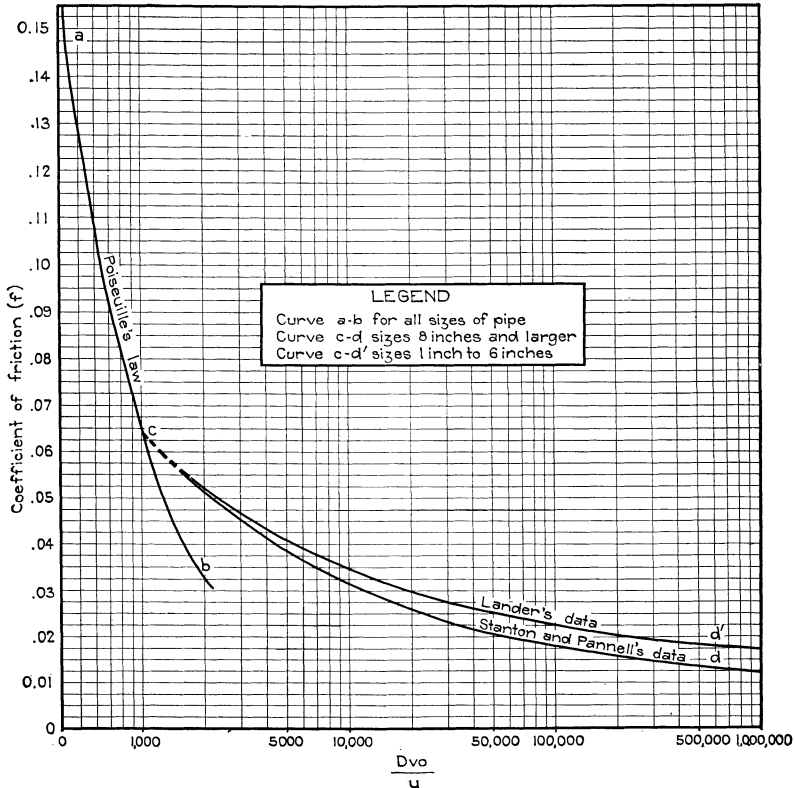


FIGURE 42.—Coefficient of friction  $f$ , after Heltzel

In calculating the hydraulic gradient it is advisable to determine the pressure drop for 2-mile intervals using the average values of  $f$  and  $o$  for each section. The pressure drop for the first 2 miles of the problem under discussion would be:

$$P = \frac{0.044 \times 58.06 \times (5.12)^2 \times 10,560}{288 \times 32.2 \times 0.635}$$

or

$P$ =pounds per square inch, or the pressure in the line at the end of the first 2 miles would be 800—120 or 680 pounds per square inch.

<sup>76</sup> Durand, W. F., *Hydraulics of Pipe Lines*: Jour. Elec., vol. 44, 1920, pp. 434—439.

The calculation for  $P$  is repeated until the hydraulic gradient curve (pressure in pipe line) either intersects the ground-line static pressure curve or becomes zero. In this particular example (see fig. 41) the hydraulic gradient curve intersects the ground static curve 12.1 miles from the pump station, at which point another pump station will be required.

In designing a line a number of different curves must be constructed, using different temperatures, pressures, and pipe sizes to determine the most economical operating conditions. Unfortunately, the amount of oil that can be put through a line at a definite pressure and temperature can not be calculated directly from the present flow formulas, as they can not be solved for velocity or quantity of oil carried because the coefficient of friction  $f$  is a function of the density, velocity, and viscosity of the oil flowing through the pipe. It is therefore necessary to calculate the capacity of each size of pipe with different pressure and temperature conditions. A graphical method for the direct solution of this problem is given by Wolf.<sup>77</sup>

The value of heating the oil to reduce the viscosity and raise the hydraulic gradient a sufficient amount to clear the top of a hill and maintain the full capacity of the line is shown in Figure 43. If the oil was not heated but was maintained at an average temperature of 100° F. the capacity of the line would be decreased as the hydraulic gradient fell below the top of the hill when the line was operated at full capacity, and it would either be necessary to locate another pump station at this point to maintain the full capacity of the line or to increase the initial pump pressure. Heating the oil to 140° F. decreases the viscosity sufficiently to raise the hydraulic gradient above the pressure required to force the oil over the hill, and no reduction in the capacity of the line will be encountered with the original pump pressure.

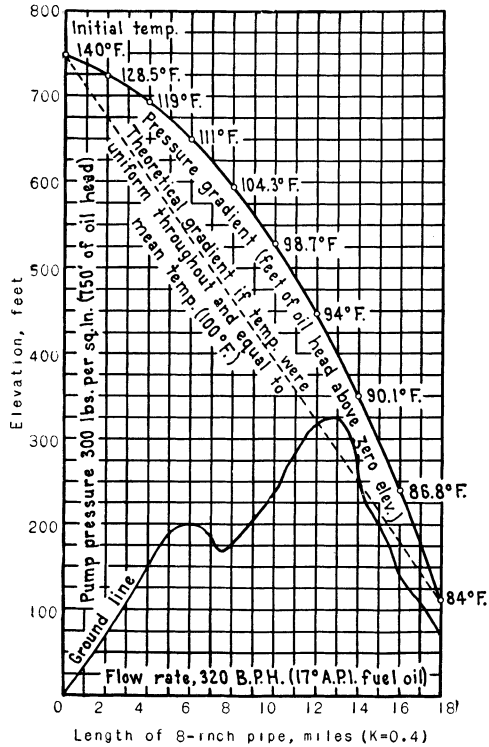


FIGURE 43.—Value of heating oils to clear hills or high spots in pipe line carrying viscous or congealing oils

<sup>77</sup> Wolf, Oscar, The Design of Oil Trunk Pipe Lines: Oil and Gas Jour., Aug. 29, 1930, pp. T-163-T-195.

The effect of heating a viscous oil on the capacity of a 10-inch line 16 miles long, handling oil of 16.5° A. P. I. gravity, is illustrated in Figure 44. The coefficient of heat loss ( $K$ ) was assumed to be 0.75 and the mean earth temperature 50° F. Curve 1 shows the pump pressures and capacity of the line with the pump station delivering oil at 140° F. to a cold line. If a pump pressure of only 350 pounds per square inch is available 150 barrels of oil per hour is all that can be put through the cold line. A pressure of 415 pounds per square inch will be required to force enough oil through

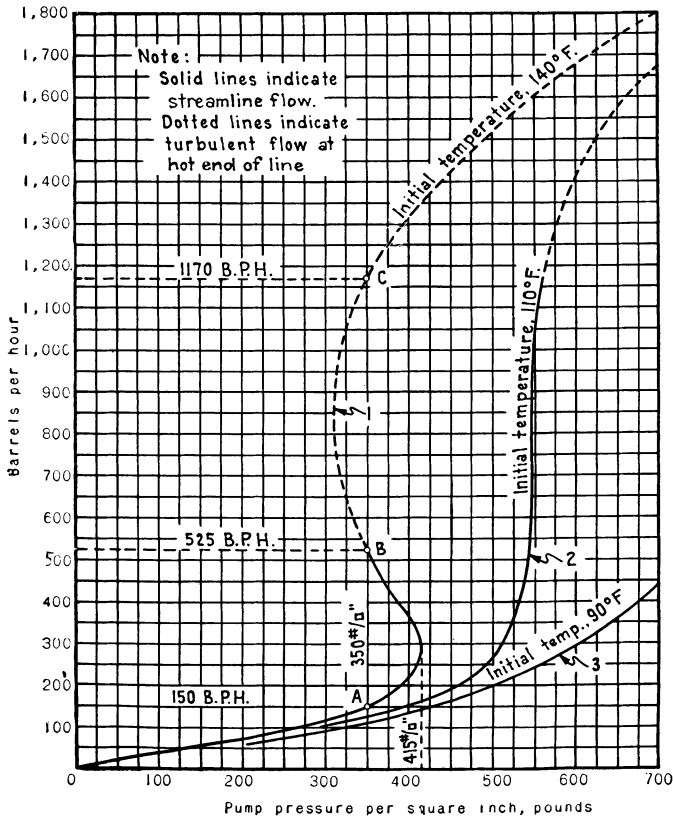


FIGURE 44.—Effect of heating viscous oils on capacity of pipe lines

the line so that the oil will heat the line sufficiently to allow higher pumping speeds, after which the line will continue to become warmer until equilibrium is reached, and 1,170 barrels of oil can be pumped per hour at a pump pressure of 350 pounds per square inch. If a pump pressure of 415 pounds is not available, the same results can be accomplished by heating the oil or running hot water or hot light oil through the line until it is heated. Curves 2 and 3 show the pump pressures and capacity of the line starting with a cold line and with the initial temperature of the oil 110° and 90° F.

The effect of using looped or double lines for hot oil is shown in Figure 45. Curve (1) shows the capacity and pumping pressure

of the 10-inch line used in the previous discussion starting with a cold line and an initial oil temperature of 140° F. Curve (2) shows the capacity and pumping pressure of a "looped" or double 8-inch line for 8 miles on the hot end of the line and then for 8 miles of single 10-inch line, starting with a cold line and an initial oil temperature of 140° F. Curve (3) shows the capacity and pumping pressures of the same line with the looped section placed on the cold end of the line. The curves show that placing a loop on the cool end of the line increases the capacity of the line over that of a single

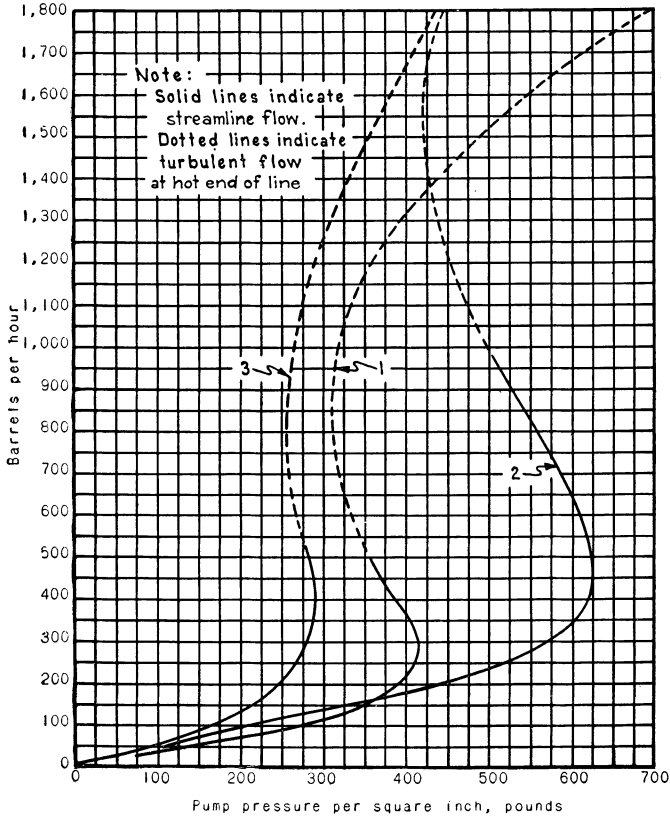


FIGURE 45.—Value and operation of looped lines for transmission of heated viscous oils

10-inch line, while under certain conditions placing the loop on the hot end of the line actually reduces the capacity and increases the pumping pressure over that of a single line. The reduction in capacity when a loop is placed on the hot end is due to increased radiation of heat and cooling of the oil as a result of the slower velocity and greater radiation area in the looped section, which increase the viscosity of the oil more rapidly. At high velocities the capacity of the line with the looped section on the hot end is greater than that of the single line and approaches that of the line with the looped section on the cold end.



The most practical method of heating oil at pipe-line pumping stations is by the use of steam-heat exchangers; however, electric heaters and hot gas heaters using the exhaust gases from Diesel engines have been used in some instances. Engineers are not in

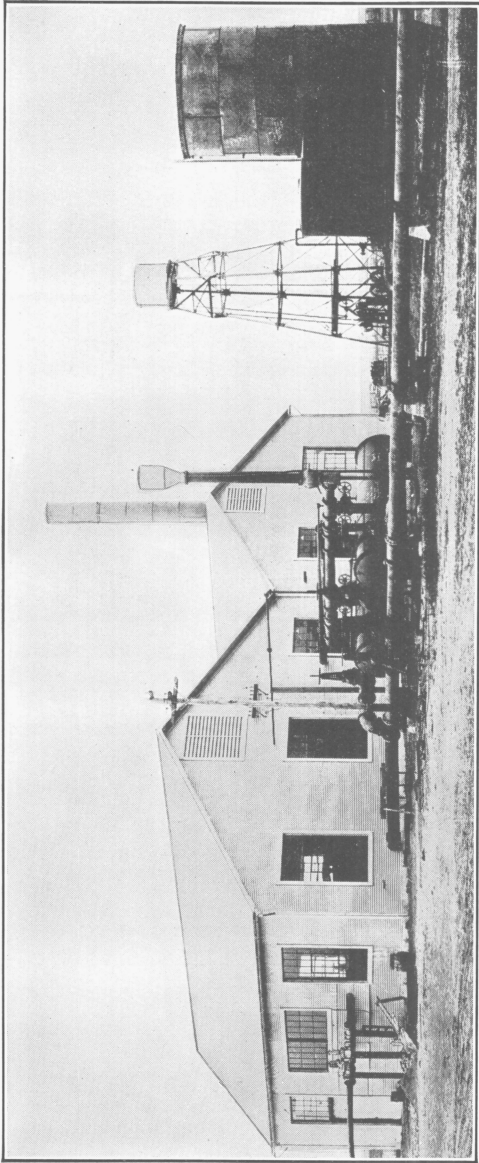


FIGURE 46.—Low-pressure pipe-line oil heaters

accord as to the advisability of using steam-powered prime movers for pumping the oil and utilizing the exhaust steam for heating the oil or of using Diesel or electric powered pumps and generating the steam for heating the oil separately. Undoubtedly the use of steam for pumping and heating will bring about economical operation if the station is a permanent installation and if the oil must be heated during the entire year. Diesel or electric powered plants with auxiliary steam-heating plants are more economical where heating is only necessary during part of the year. In handling fuel oils or oils that will not vaporize appreciably, large low-pressure heaters (fig. 46) designed for use with low-pressure or exhaust steam can be used on the intake side of the pumps.

When light crudes are handled it is necessary to use high-pressure heaters placed on the pressure side of the pumps; otherwise the oils vaporize and cause gas locking of the pumps. A commercial installation of heaters for pumping high-gravity crudes is shown

in Figure 47. The heaters are designed for high or low pressure steam.

Heating pipe lines often results in excessive expansion and contraction of the line, and unless this has been taken into consideration when the line is being constructed trouble will be encountered each

time the line is heated and cooled. It is considered advisable when starting up a line for use at high temperature to bring the temperature of the line gradually to the operating temperature by first pumping warm water or warm nonviscous oils through it. Figure

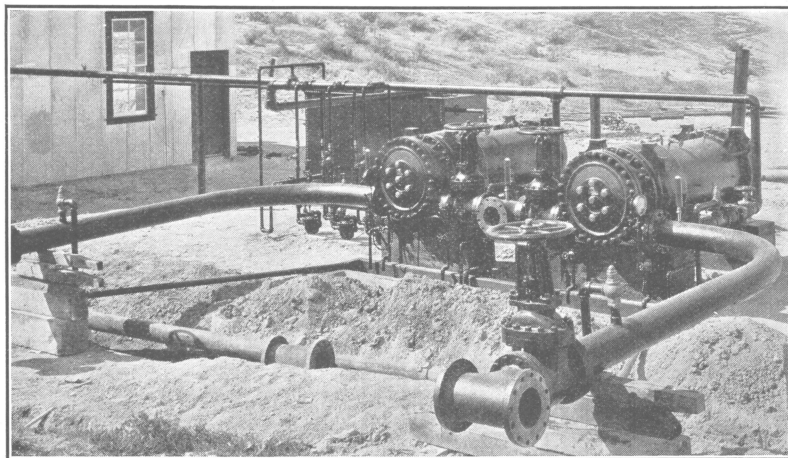


FIGURE 47.—High-pressure pipe-line oil heaters for volatile oils

48 shows the expansion of pipe with change in temperature.<sup>78</sup> Expansion joints materially reduce the trouble due to the expansion of pipe lines, and if ordinary precautions are observed when constructing lines for transporting hot oils no trouble will be encountered.

#### REMOVAL OF PARAFFIN FROM TRUNK PIPE LINES

Although the accumulation of wax on the walls of pipe lines seldom becomes very thick when the flow of oil through the line is turbulent or is approaching turbulence a small amount of paraffin will in time accumulate, and if the lines are operated at reduced capacity paraffin tends to accumulate rapidly. When paraffin accumulates it is necessary to clean the lines at regular intervals to prevent plugging of the lines. Monthly removal of the small quantity of paraffin that accumulates in one of the trunk lines between Oklahoma and Chicago, in which the oil flows turbulently or approaches this form of flow, increases the efficiency of the line from 2 to 5 per cent.

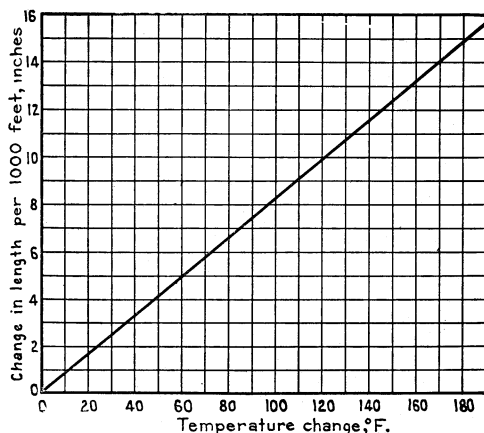


FIGURE 48.—Theoretical expansion of steel pipe when heated

<sup>78</sup> Pound, J. H., Expansion in Long Pipe Lines: Oil Weekly, vol. 53, No. 8, May 10, 1929, pp. 21-23, 101-104.

The most satisfactory method of cleaning pipe lines in which the accumulation of paraffin has not become too thick is by the use of the pipe-line scraper.<sup>79</sup> (See fig. 39.) This scraper is forced through the line by the movement of the oil and forces the paraffin ahead of it. Welded lines that have been put together without thought of using scrapers may contain beads or "icicles" on the inside of the welds that will cut the scrapers to pieces. Beads or "icicles" in welded lines can be prevented by running a cutter through the lines after each joint is welded. It is also important to eliminate all sharp bends that might prevent free passage of the scraper through the line. Scraper boxes at all manifolds and stations facilitate the insertion and removal of the scrapers. The cost of using scrapers at regular intervals is negligible, and they are a very cheap insurance against plugged lines.

When pipe lines become so badly filled with paraffin that it is impossible to clean them with a scraper, the paraffin must be dissolved or melted by running kerosene, distillate, hot crude oil, or hot water through the line or the line must be cut at intervals and steamed out. Such methods are expensive and emphasize the value of using pipe-line scrapers at regular intervals.

#### METHODS FOR HANDLING CONGEALING-OIL AND PARAFFIN DEPOSITS IN STORAGE TANKS

Paraffin and mixed-base crude oils that have a high pourpoint or "cold test" often congeal in the flow and field storage tanks and must be heated before they can be pumped. Pipe-line companies usually require that such oils be heated to at least 90° F. to insure complete settling of any water and B. S. (emulsions) that are in suspension in the oil. It is also advisable to heat such oils so that any paraffin that has separated from the crude will be put back into solution; otherwise, it accumulates in the bottom of the tanks with the cut oil and requires special treating to recover it as pipe-line oil.

#### HEATING OIL IN TANKS

When large quantities of oil are handled in tank batteries the most economical method of heating the oil is to use steam. A small (45-horsepower) boiler located at the tank battery will usually supply enough steam to heat the tanks with steam coils and to clean out the flow lines and heat the oil for treating emulsions if present. On old leases, where the production of oil is small and there is no constant demand for steam, individual gas-fired heaters are generally the most economical. Heaters can also be used for heating the oil for treatment with chemicals to "break down" emulsions. Several types of individual tank heaters suggested by R. T. Bright, formerly of the Geological survey at Muskogee, Okla., are shown in Figure 49.

In the Salt Creek field 250 and 500 barrel tanks heated by electricity are located at many wells to eliminate flow-line troubles due to congealed oil. Figures 50 and 51 show the method of installing the electric heaters in the tank. When the atmospheric temperature

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<sup>79</sup> Oil and Gas Journal, Pipe-Line Scrapers for Many Uses: Aug. 30, 1928, p. T-173.

is zero and the oil is heated to 100° F. approximately 65 per cent of the heat was found to be lost by radiation in a 250-barrel tank. The cost of power for heating a 250-barrel tank of oil under such

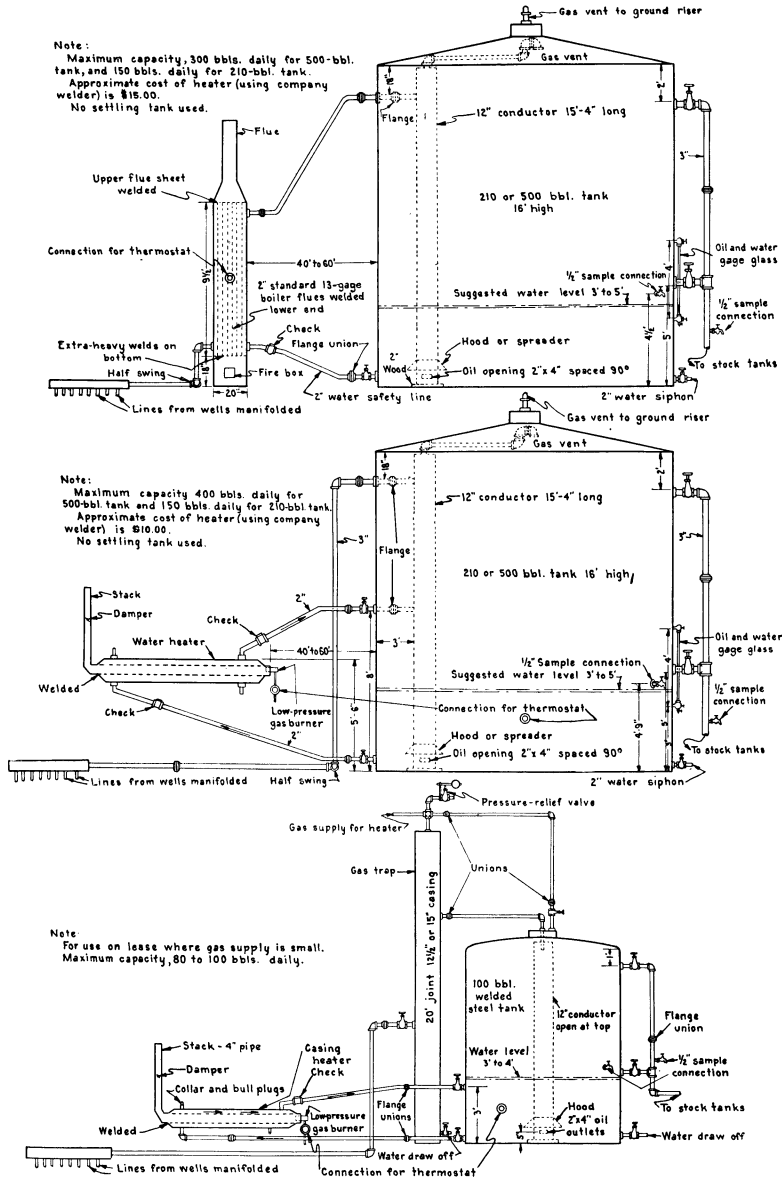


FIGURE 49.—Gas-fired heaters for field storage tanks

conditions is \$10.80, based on a power cost of 1½ cents per kilowatt-hour. Undoubtedly, gas-fired heaters would usually be more economical, but electric heaters are invaluable for isolated wells where long gas lines would have to be laid and maintained.

## RECLAMATION OF TANK BOTTOMS

Unless the oil that contains precipitated wax is heated in the tanks before it is removed the wax gradually accumulates and forms

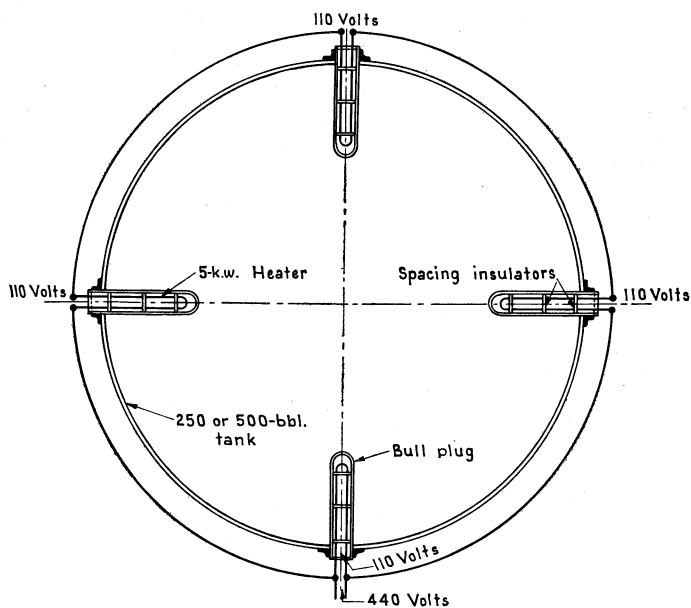


FIGURE 50.—Method of installing electric heaters in oil-storage tanks

“tank bottoms,” which can readily be put back into solution by heating when the tank is full of oil. The general procedure in many fields is to allow the wax to accumulate in field-storage tanks

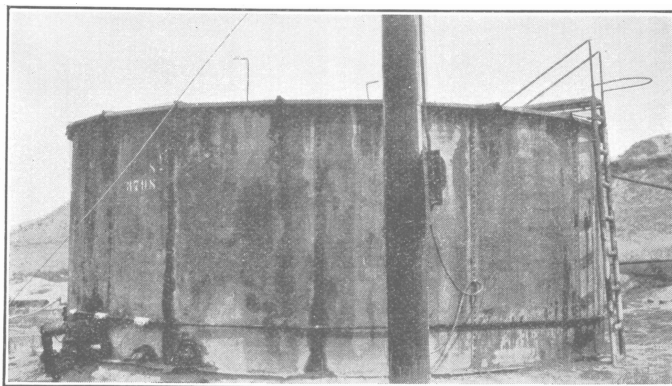


FIGURE 51.—Tank equipped with electric heaters

to a depth of about 1 foot before heating the oil in the tank, to eliminate the necessity of steaming the tank each time oil is removed. When emulsion is present with the accumulated paraffin in tank bottoms it often becomes necessary to draw off the settlings at regu-

lar intervals and to treat the emulsion, but frequently tank bottoms are run into sumps and burned. This process is wasteful, and the oil and wax contained in these tank bottoms can usually be profitably reclaimed and mixed with fresh oil. Figure 52 shows a tank battery with equipment for treating tank bottoms. The wax and emulsion are run into a wooden sump box where they are steamed and chemicals added if necessary. The water (and frequently some very stable emulsion) is drained off into a pit; and the reclaimed oil, which usually has a large amount of wax in solution, is pumped into the small tank where it is mixed with fresh oil before going into the pipe line.

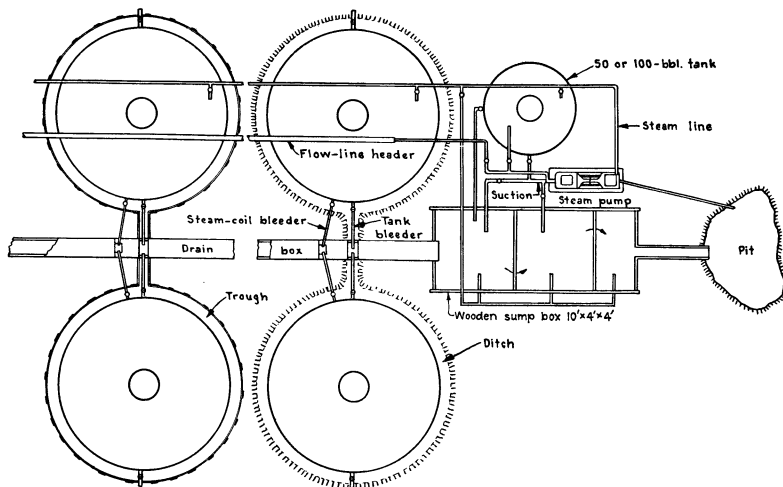


FIGURE 52.—Treating plant at field tank station

### SALVAGING PARAFFIN AND WASTE OIL

In fields where paraffin troubles are encountered in the production of oil, oil and paraffin in considerable amounts generally accumulate around the wells as a result of pulling rods and tubing. If this material is allowed to accumulate on the derrick floor and in the cellar of the well, accident and fire hazards are increased. By using steel grating, as shown in Figure 25, p. 65, the oil and wax will drop into the cellar and from there the accumulated oil and other material can be pumped or flowed by gravity into a small sump at the side of the derrick. It is not absolutely necessary to use steel grating for flooring, as the oil will drain through the cracks in a wooden floor, but the grating gives a better foothold for workmen and a cleaner derrick. The waste oil can then be collected from the sumps and derrick cellars by a tank wagon and portable pump and taken to the field tanks, or, if necessary, to a central treating plant to remove any water before running it into the pipe line. Five or ten barrels of oil frequently flow from the tubing while rods and tubing are being pulled, and the investment necessary to construct the collecting basin and sump will return a good profit.

### SPECIAL PROBLEMS

In some localities mineral salts are deposited in oil wells and oil strings, due to their precipitation from the water that is produced with the oil.<sup>80</sup> The deposition of calcium carbonate from the water entering the well occurs because most frequently soluble calcium bicarbonate is converted into insoluble calcium carbonate by the liberation of carbon dioxide. Other mineral salts precipitate from oil-field waters, also; and in the Eldorado field, Kansas,<sup>81</sup> for example, barium and strontium sulphate were deposited in the tubing of wells due to the mixing of two different waters that entered the wells. In the Eldorado field, deposits of mineral salts accumulated rapidly and for a long time were removed by pulling the rods and tubing. Later, the deposition of mineral salts was prevented by excluding one of the waters from entering the well. In certain wells mineral salts are also deposited in the well by gas produced with the oil, as a result of the evaporation of the water that carried them.

The problems brought about by the deposition of minerals in oil wells are with few exceptions of minor importance and do not seriously affect the production of oil. It is, of course, advisable at all times to prevent the entrance of water into producing wells and indirectly to prevent the deposition of mineral matter in the well.

### SUMMARY

The data obtained in this laboratory and field study of the conditions causing or favoring the deposition of paraffin from certain types of crude oils and difficulties encountered in the production, transportation, and storage of congealing or viscous oils have shown that certain methods of operation will prevent or modify the troubles. Although in actual operation it is often impractical or impossible to control these conditions and entirely prevent the troubles, they can usually be greatly modified or overcome by the use of other methods.

In discussing methods of correction and prevention, the author has attempted to present the advantages and disadvantages of each method and the results that might be expected. The selection of any method depends primarily upon the nature of the problem and the available equipment. No one method will prove economical for all conditions, and the operator should consider these facts and the costs entailed before making a selection.

The problem of the accumulation of paraffin in the oil string of wells is one that usually can be corrected by careful operation of the well. In wells where it is impossible or impractical to prevent the accumulation of paraffin by proper operating methods, it is advisable to remove the accumulation of paraffin in the oil string periodically with tools. Methods for heating the oil string and oil to prevent the accumulation of paraffin in the oil string are usually less satisfactory as regards costs than the use of special paraffin-removing tools.

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<sup>80</sup> Mills, R. van A., Paraffin Problem in Oil Wells: Rept. of Investigations 2550, Bureau of Mines, December, 1923, 11 pp.

<sup>81</sup> Mills, R. van A., Protection of Oil and Gas Field Equipment Against Corrosion: Bull. 233, Bureau of Mines, 1925, p. 60.

Correct operation of wells will greatly modify the problems of paraffin accumulation on the face and in the sand; however, where paraffin has accumulated or can not be prevented from accumulating, heating the sand and the use of solvents are the most satisfactory paraffin-removal methods, although tools or explosives for removing the face of the sand are sometimes used to advantage. In sands where gas or air is being returned the injection of gas from stabilizers or of the lighter liquid constituents of natural gasoline, or the recirculation of the gas from which the gasoline vapors have not been removed, will often prevent deposition and remove deposited paraffin.

The handling of congealing and viscous oils is primarily a problem of preventing excessive cooling, and where this can not be accomplished the oil must be heated. Different methods of heating and of preventing cooling have been discussed. Local conditions, the nature of the oil, and the available equipment are generally the deciding factors governing the selection of the method most advisable for handling such oils.





# LABORATORY STUDY OF ROD WAXES

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By C. E. REISTLE, jr., and O. C. BLADE

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## INTRODUCTION

In reviewing the literature pertaining to the study of waxes and the solid constituents of crude oil the authors found that most of these investigations had been devoted chiefly to the study of the chemical and crystalline characteristics of the wax components of "paraffin" or "rod wax" and that very little information was available which would be applicable or useful in making a study of the problems encountered with deposition of "paraffin" or rod wax in the production of crude oil. It seemed advisable, therefore, to conduct a laboratory study, in addition to actual field experiments, to determine the following facts: (1) The nature of the rod-wax oil solution; (2) the factor or factors, and their relative importance, responsible for the separation of rod wax from solution in the oil; (3) the general characteristics of the precipitated rod wax; and (4) the nature of the various constituents found in rod waxes precipitated from different types of crude oils. The results of the laboratory investigation dealing with the nature of rod-wax crude-oil solutions, and the factors causing the separation of rod wax from solution in the oil and their application to the paraffin problems encountered in the production of the crude oil, have been discussed in Part I of this bulletin. The analyses of the different rod waxes and a description of their constituents were not discussed, however, and although the data are limited and of minor importance with respect to production problems it is believed advisable to include this information in the report to give more complete information on the nature and composition of rod waxes.

## REVIEW OF LITERATURE

The crystalline behavior and chemical composition of waxes found in different crude oils have been sources of much discussion, and a number of conflicting theories have been advanced to explain their composition or crystalline peculiarities. The most important of the theories are as follows:

(1) Zalozeicki,<sup>82</sup> after a study of petroleum waxes and ozocerite, concluded that both crystalline and amorphous waxes are present in crude petroleum and that in the distillates from petroleum the wax is of a crystalline nature. He therefore postulates the theory that the amorphous waxes (protoparaffins), probably branched chain,

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<sup>82</sup> Zalozeicki, Z., Paraffin in Petroleum: Ztschr., angew. Chem., vol. 3, 1888, pp. 261-318.

are converted into the crystalline waxes (pyroparaffins), straight-chain hydrocarbons, by distillation. Marcusson<sup>83</sup> apparently substantiates these conclusions. Using pure samples of paraffin wax and ceresin wax of the same melting point Marcusson found that the molecular weight, index of refraction, and specific gravity of the ceresin wax were higher than those for the paraffin wax, and he therefore concluded that the ceresin was a branched chain or isoparaffin. He found that the ceresin wax was similar to the amorphous wax in petroleum, and careful distillation of the ceresin made it possible to produce a crystalline wax.

(2) Gurwitsch,<sup>84</sup> in studying the crystalline behavior of petroleum waxes with the aid of a petrographic microscope and polarized light found that petroleum waxes, including petrolatums, which had not been distilled or recrystallized by the use of solvents were crystalline. His conclusions were that Zaloziecki's amorphous wax (protoparaffin) was not actually amorphous, but that the crystalline development had been retarded and altered by the crystallizing medium or oil and, possibly, by the presence of colloidal material. The fact that the crystallizing medium and the presence of colloidal material do influence the crystalline behavior of the petroleum waxes has been substantiated by Gordon;<sup>85</sup> Sachnen;<sup>86</sup> Padgett, Hefley, and Henriksen;<sup>87</sup> and Tanaka, Kobayashi, and Ohno.<sup>88</sup>

(3) Buchler and Graves<sup>89</sup> were able to prepare by careful fractionation and recrystallization of waxes from Salt Creek (Wyo.) crude oil wax fractions having physical and chemical characteristics in close agreement with values given for the pure paraffin straight chain  $C_nH_{2n+2}$  hydrocarbons prepared by Krafft.<sup>90</sup> In addition to this normal paraffin series of crystalline waxes they found associated with the wax a substance or series of substances that they term "soft wax." This substance is probably the same as the "mineral jelly" or amorphous wax noted by Carpenter.<sup>91</sup> They showed that the presence of this so-called "soft wax" produced a marked effect on crystalline behavior of the pure straight-chain hydrocarbons and concluded that although the viscosity of the crystallizing medium and the solubility and molecular weight of the wax might govern the crystalline behavior of the wax to a certain extent the vital or most important factor was the quantity of soft wax present.

(4) Ferris, Cowles, and Henderson<sup>92</sup> obtained, by fractional distillation and recrystallization of wax from scale wax, slack wax,

<sup>83</sup> Marcusson, J., *Chem. Ztg.*, vol. 38, 1914, pp. 73; vol. 39, 1915, pp. 578-613; *Ztschr. angew. Chem.*, vol. 37, 1924, p. 473.

<sup>84</sup> Gurvich, L., *Scientific Principles of Petroleum Technology*: Chapman & Hall, London, 1926, pp. 15 and 450; *Properties of Paraffin-Containing Products*: *Petrol. Ztschr.*, vol. 19, 1923, pp. 183.

<sup>85</sup> Gordon, P. F., *Paraffin Crystallization*: *Jour. Soc. Chem. Ind.*, vol. 42, 1923, pp. 405 T; Gordon, P. F., and Marshall, A. C., *The Separation of the Components of Petroleum*: *Jour. Soc. Chem. Ind.*, vol. 46, 1927, pp. 304-306 T.

<sup>86</sup> Sachanen, A., *Solidification of Paraffin-Containing Products*: *Petrol. Ztschr.*, vol. 22, 1926, pp. 484.

<sup>87</sup> Padgett, F., Hefley, D., and Henriksen, A., *Wax Crystallization*: *Ind. and Eng. Chem.*, vol. 18, No. 8, 1926, pp. 832-835.

<sup>88</sup> Tanaka, Y., Kobayashi, R., and Ohno, S., *Studies on Crystallization of Paraffin*—Parts I, II, and III: *Jour. Faculty Eng., Tokyo Imperial Univ.*, Tokyo, Japan, vol. 17, No. 15, November, 1928, pp. 278-282.

<sup>89</sup> Buchler, C. C., and Graves, G. D., *The Petroleum Waxes*: *Ind. and Eng. Chem.*, vol. 19, No. 6, 1927, pp. 817-824.

<sup>90</sup> Krafft, F., *Preparation of Pure Paraffin Hydrocarbons*: *Ber. deut. chem. Gesell.*, vol. 19, 1896, pp. 2223; vol. 29, 1896, pp. 1323; vol. 40, 1907, pp. 4783.

<sup>91</sup> Carpenter, J. A., *The Physical and Chemical Properties of Paraffin Wax, Particularly in the Solid State*: *Jour. Inst. Petrol. Technol.*, vol. 12, 1926, pp. 288-315.

<sup>92</sup> Ferris, S. W., Cowles, H. C., Jr., and Henderson, L. M., *Composition of Paraffin Wax*: *Ind. and Eng. Chem.*, vol. 21, No. 11, 1929, pp. 1090-1092.

foots oil, and sweat oil found in Mid-Continent petroleum, six fractions of wax having the same 50 per cent boiling point. These six waxes, however, had decreasing melting points and increasing density and indexes of refraction. Their conclusion was that, although the major portion of the wax in petroleum might consist of the straight-chain  $C_nH_{2n+2}$  series there was also evidence of waxes composed of other types of hydrocarbons, probably of the branch-chain paraffin series. These conclusions are somewhat substantiated by Marcusson.<sup>93</sup>

#### SELECTION OF SAMPLES

Samples of rod wax and crude oil were obtained from most of the major fields where paraffin troubles are encountered. It was impractical to make a detailed study of every one of the large number of samples; therefore, the Bureau of Mines Hempel distillation<sup>94</sup> of the crude oils from which the rod waxes were precipitated was used as a basis for classifying the rod waxes into groups produced from oils of similar characteristics. After the classifications were made the rod waxes from Bradford field, Pennsylvania, Clay district, West Virginia, Panhandle district, Texas, and Salt Creek field, Wyoming, were selected for the detailed analyses. These samples represented different types of crude oils and the major producing districts in which paraffin problems were encountered in the production of crude oil.

#### ANALYSES OF ROD WAXES

##### DESCRIPTION OF SAMPLES

The sample from the Bradford district was very soft, nongranular-appearing wax that had a melting point of 132.1° F., which is low for the average rod wax. It was yellow and contained a small amount of suspended water and sand. The rod wax from the Clay district, West Virginia, was of a stiff viscid consistency, granular in appearance, and had a melting point of 161.9° F. It was yellowish-green and contained a little suspended water and sand. The Panhandle rod wax, which had a melting point of 141.2° F., was of very stiff and viscid consistency and appeared granular. It was greenish-black and contained some sand and water in suspension. The Salt Creek rod wax had a melting point of 154.6° F. and was of a semi-fluid consistency with a granular appearance. It was dark green and contained suspended sand and water.

##### FRACTIONATION OF ROD WAXES

It was necessary to develop a satisfactory method for fractionating the rod wax because the object of the analyses was to determine the character and approximate quantity of the various substances present. In view of the fact that distillation might alter the characteristics and composition of the gums, unsaturated material, and possibly the wax<sup>95</sup> it was thought advisable to fractionate the crude

<sup>93</sup> Marcusson, J. The Composition of the Solid Hydrocarbons of Petroleum, Paraffin, and Ceresin: Mitt. kgl. Materialprüfungsamt, vol. 33, 1917, pp. 415-426.

<sup>94</sup> Dean, E. W., Hill, H. H., Smith, N. A. C., and Jacobs, W. A., The Analytical Distillation of Petroleum and Its Products: Bull. 207, Bureau of Mines, 1922, 82 pp.

<sup>95</sup> Zulozicki, Z., Paraffin in Petroleum: Ztschr. angew. Chem., vol. 3, 1888, pp. 126-261-318.

rod wax by the use of selective solvents. Accordingly, acetone was used for the preliminary fractionation, although other solvents could probably have been used with as good or better results.

The crude rod wax was first centrifuged while melted to separate the suspended water, sand, and as much as possible of the free crude oil that was held mechanically in association with the precipitated wax. After being centrifuged the rod wax was melted on a steam bath, and approximately 300 grams was slowly poured into 15 liters of cold acetone, thus causing the wax to form a spongy mass offering a large surface for the extraction of the remaining oil. The acetone and wax were refluxed over a steam bath for 48 hours, cooled to 30° F., filtered, and the insoluble material washed with 15 liters of fresh acetone chilled to 30° F. The material that was insoluble in acetone was practically free from oil and liquid constituents and consisted of the waxes, resins, gums, and asphaltic material. The insoluble material was melted on a steam bath and weighed after all traces of acetone had been removed with a vacuum pump. This weight was then considered as the weight of the oil-free rod wax sample. The acetone containing the soluble material was distilled off, leaving as a residue the crude oil removed from the wax. The oil-free rod wax was remelted and poured into 15 liters of cold acetone to obtain the wax again in a spongy finely divided state. The acetone was distilled off until only 2 liters remained, after which the wax and acetone were refluxed over a steam bath for 24 hours and then filtered while hot. The hot acetone filtrate was cooled to 30° F., filtered, and the insoluble waxy material designated as fraction F1. The filtered acetone was distilled, and the soft brown gum or resin that remained was designated as residue R1. The rod wax remaining insoluble after refluxing with 2 liters of hot acetone was again refluxed with a larger amount of acetone for 24 hours, and the procedure previously described was followed which resulted in fraction F2 and acetone soluble residue R2. This procedure was repeated, using enough acetone to procure approximately 5 per cent fractions. When only about 10 per cent of the original oil-free rod wax remained undissolved by acetone it was refluxed with 15 liters of petroleum ether (b.p., 100–150° F.). The soluble material was called the last "F fraction." The insoluble material was called asphaltenes.

The F fractions were composed of the crystalline and "soft wax," with some gums and resins held in solution or absorbed by the wax. They were dissolved in petroleum ether and treated with 200-mesh fuller's earth until colorless. The fuller's earth was removed by filtration, and the petroleum ether was then distilled off, using a high vacuum to remove the last traces of petroleum-ether vapor. The resulting fractions were white and were composed of crystalline and "soft waxes." The gums and resins absorbed from the F fractions by the fuller's earth were recovered by extraction of the fuller's earth in a Soxhlet extractor, using petroleum ether and then benzol as solvents. The small quantity of wax absorbed by the fuller's earth was extracted by the petroleum ether and returned to the wax fractions. The benzol dissolved the gums and resins and removed them from the fuller's earth. These gums were apparently similar to the R fractions and were therefore added to them.

The melting points of the white wax fractions were then determined, and groups of two or three of the F fractions were combined, the number depending upon the relationship of the melting points. The fractions were combined so that the samples for recrystallization would be larger and a smaller number of samples would have to be run. The combined fractions were recrystallized from solution in ethylene dichloride at room temperature or above, and the waxes that recrystallized from solution were called the "CF fractions." The material that remained in solution in the ethylene dichloride was recovered by distillation and was designated the "SCF fractions." Sometimes the fractions were recrystallized four or five times, in which case all the material remaining soluble in the ethylene dichloride was combined to form the SCF fraction.

#### DISCUSSION OF RESULTS

The quantity, physical properties, and characteristics of the different fractions separated from the rod waxes were determined. The results are summarized in Tables 18 to 21, inclusive.

#### WATER, SAND, SILT, AND MINERAL SALTS

The water, sand, silt, and mineral salts found in the rod wax are not considered in the following discussion, because they have no particular bearing on the composition of the rod wax that actually separates from solution in the oil and are present in the rod wax only as a result of being mechanically deposited with the wax as they are produced with the oil.

#### CRUDE OIL

The oil found in rod waxes is crude oil mechanically held or trapped between the wax grains and removable by high-speed centrifuging or filter pressing. There may be some oil actually in solution or absorbed by the wax crystals, but the amount is small compared to the quantity of oil held mechanically in the rod wax. The quantity of oil in the four rod waxes analyzed ranged from 24 per cent in the Panhandle wax to 38 per cent in the Salt Creek wax. The amount of oil present in a rod wax depends upon the manner in which the wax was deposited and the corresponding time of deposition. For example, the presence of a large quantity of oil (30 to 50 per cent) in a rod wax indicates that the wax has been deposited in a short time or has not stood for any great length of time without being submerged in an excess of crude oil, whereas the presence of only a small quantity of oil (10 to 30 per cent) indicates that the rod wax was deposited over a long period of time or under conditions where the oil could drain away from the accumulated wax.

TABLE 18.—*Summary of analysis of rod wax 1 from Bradford district, Pennsylvania*

[Melting point of crude rod-wax sample, 132.1° F. (55.8° C.); weight of crude rod-wax sample, 303 grams; weight of oil-free rod-wax sample, 202 grams, 67 per cent; weight of crude oil removed from rod wax, 101 grams, 33 per cent]

FRACTIONS SEPARATED FROM ROD WAX WITH HOT ACETONE AND PRECIPITATED AT 30° F. TO FORM CRUDE-WAX FRACTIONS

No.	Weight, grams	Oil-free rod wax, per cent	Acetone, used, liters	Color	After removing gums and resins with fuller's earth			Melting point, ° F.
					Weight, grams	Oil-free rod wax, per cent	Color	
F1	17.8	8.8	0.5	Light brown	15.3	7.6	White	138.0
F2	10.7	5.3	.5	do	10.2	5.0	do	138.4
F3	11.1	8.0	1.0	do	10.6	5.2	do	139.7
F4	8.2	4.1	1.5	do	7.9	3.5	do	141.7
F5	13.9	6.9	2.0	do	13.2	6.5	do	145.5
F6	12.8	6.3	2.5	do	12.6	6.2	do	147.5
F7	11.0	5.4	3.0	do	10.9	5.4	do	148.8
F8	11.3	5.6	3.5	do	10.8	5.3	do	150.5
F9	13.8	6.8	4.5	do	13.6	6.7	do	151.9
F10	12.1	6.0	6.0	do	11.5	5.7	do	153.5
F11	9.9	4.9	7.0	do	9.1	4.5	do	154.4
F12	10.4	5.1	8.0	do	10.0	5.0	do	155.4
F13	8.1	4.0	9.0	do	7.8	3.9	do	155.7
F14	6.7	3.3	10.0	do	6.3	3.1	do	157.6
F15	8.9	4.4	74.0	do	8.5	4.2	do	157.9
F16	12.5	6.2	Residue.	Dark brown	6.6	3.3	Opaque yellow	129.2
Total	179.2	88.7			164.9	81.6		

GUMS<sup>1</sup> AND RESINS SOLUBLE IN ACETONE AT 30° F. PLUS THOSE REMOVED FROM CRUDE-WAX FRACTIONS WITH FULLER'S EARTH

No.	Weight, grams	Oil-free wax, per cent	Color	No.	Weight, grams	Oil-free wax, per cent	Color
R1	2.5	1.2	Dark brown.	R10	1.5	0.7	Dark brown.
R2	1.8	.9	Do.	R11	1.4	.7	Do.
R3	3.5	1.7	Do.	R12	1.2	.6	Do.
R4	2.1	1.0	Do.	R13	1.4	.7	Do.
R5	2.4	1.2	Do.	R14	1.6	.8	Do.
R6	1.7	.8	Do.	R15	1.6	.8	Do.
R7	1.2	.6	Do.	R16	2.0	1.0	Do.
R8	1.9	.9	Do.				
R9	1.5	.7	Do.	Total	29.3	14.5	

<sup>1</sup> Soft viscid gum; these gradually became more viscid and harder until for R16 gum is very hard.

TABLE 18.—*Summary of analysis of rod wax 1 from Bradford district, Pennsylvania—Continued*

CRUDE-WAX FRACTIONS COMBINED AND RECRYSTALLIZED WITH ETHYLENE DICHLORIDE

No.	Crude-wax fractions combined	Total crude-wax fractions, per cent	Color	Melting point, ° F.	Index of refraction, 183° F. (84° C.)	Density 183° F. (84° C.)	Crystalline habit	Physical characteristics
CF1 <sup>2</sup>	F1-F2	8.7	White	145.2	1.4294	0.7721	Large plates and needles.	Hard wax.
SCF1 <sup>3</sup>	do	2.8	do	119.9	1.4377	.7883	do	Soft wax.
CF2	F3-F4	9.7	do	145.8	1.4302	.7733	do	Hard wax.
SCF2	do	4.3	do	121.4	1.4415	.7956	do	Do.
CF3	F4-F6	12.0	do	149.2	1.4306	.7735	do	Hard, brittle wax.
SCF3	do	2.7	do	131.2	1.4417	.7960	Smaller plates, more needles.	Hard wax.
CF4	F7-F8	12.9	do	150.9	1.4325	.7785	Large plates and needles.	Hard, brittle wax.
SCF4	do	2.2	do	136.1	1.4428	.7981	Smaller plates and needles.	Hard wax.
CF5	F9-F10	13.1	do	153.0	1.4344	.7820	Large plates and needles.	Hard, brittle wax.
SCF5	do	4.0	do	141.5	1.4429	.7983	Small plates, mostly needles.	Hard wax.
CF6	F11-F12	8.7	do	155.3	1.4369	.7865	Plates and needles	Do.
SCF6	do	.9	do	144.2	1.4445	.8014	Needles and plates	Do.
CF7	F13-F14-F15	11.1	do	157.3	1.4414	.7955	do	Do.
SCF7	do	1.8	do	146.5	1.4497	.8113	do	Soft wax.
CF8	F16	4.4	Opaque white.	133.2	1.4575	.8265	do	Soft, gummy wax.
SCF8	do	.7	do	119.8	1.4588	.8290	do	Do.

<sup>2</sup> CF fractions are those which crystallized from the ethylene dichloride.

<sup>3</sup> SCF fractions are those which remained in solution in the ethylene dichloride.

	Per cent
NOTE.—Total white wax fractions.....	81.6
Total gums and resins.....	14.5
Total "asphaltenes".....	.2
	96.3
Loss.....	3.7
	100.0



TABLE 19.—*Summary of analysis of rod wax 2 from Clay district, West Virginia*

[Melting point of crude rod-wax sample, 161.9° F. (72.1° C.); weight of crude rod-wax sample 297 grams; weight of oil-free rod-wax sample, 202 grams, 68 per cent; weight of crude oil removed from rod wax, 95 grams, 32 per cent]

FRACTIONS SEPARATED FROM ROD WAX WITH HOT ACETONE AND PRECIPITATED AT 30° F. TO FORM CRUDE-WAX FRACTIONS

No.	Weight, grams	Oil-free rod wax, per cent	Acetone, used, liters	Color	After removing gums and resins with fuller's earth			
					Weight, grams	Oil-free rod wax, per cent	Color	Melting point, ° F.
F1	8.9	4.4	2.0	Light brown	7.7	3.8	White	135.8
F2	7.7	3.8	5.0	do	6.3	3.1	do	151.0
F3	8.2	4.1	10.0	do	6.5	3.2	do	160.8
F4	11.5	5.7	20.0	do	10.3	5.1	do	167.8
F5	11.5	5.7	60.0	do	10.2	5.0	do	169.1
F6	11.7	5.8	75.0	do	11.2	5.5	do	172.2
F7	12.2	6.0	90.0	do	11.2	5.5	do	172.8
F8	13.7	6.8	105.0	do	12.6	6.2	do	174.1
F9	8.8	4.4	105.0	do	7.5	3.7	do	175.1
F10	9.5	4.7	60.0	do	8.8	4.4	do	175.9
F11	8.5	4.2	60.0	do	7.8	3.9	do	176.5
F12	8.4	4.2	75.0	do	7.8	3.9	do	176.8
F13	7.5	3.7	65.0	do	6.9	3.4	do	177.3
F14	7.5	3.7	75.0	do	6.9	3.4	do	176.8
F15	8.2	4.1	75.0	do	7.6	3.8	do	175.2
F16	8.7	4.3	111.0	do	6.4	3.2	do	175.2
F17	9.0	4.5	116.0	do	8.3	4.1	do	175.8
F18	7.3	3.6	116.0	do	6.8	3.4	do	175.8
F19	5.9	2.9	15.0	do	5.5	2.7	do	179.6
F20	12.3	6.1	Residue		5.6	2.8	do	178.6
Total	187.0	92.6			162.9	80.6		

<sup>1</sup> 75 per cent acetone; 25 per cent ethylene dichloride.

GUMS<sup>2</sup> AND RESINS SOLUBLE IN ACETONE AT 30° F. PLUS THOSE REMOVED FROM CRUDE-WAX FRACTIONS WITH FULLER'S EARTH

No.	Weight, grams	Oil-free wax, per cent	Color	No.	Weight, grams	Oil-free wax, per cent	Color
R1	5.5	1.7	Dark brown.	R12	0.5	0.2	Dark brown.
R2	3.0	1.5	Do.	R13	2.5	1.2	Do.
R3	2.2	1.1	Do.	R14	3.7	1.8	Do.
R4	1.7	.8	Do.	R15	1.9	.9	Do.
R5	2.6	1.3	Do.	R16	2.8	1.4	Do.
R6	1.1	.5	Do.	R17	1.9	.9	Do.
R7	.4	.2	Do.	R18	2.8	1.4	Do.
R8	.5	.2	Do.	R19	1.1	.5	Do.
R9	1.0	.5	Do.	R20	1.3	.6	Do.
R10	.8	.4	Do.				
R11	.2	.1	Do.				
Total				Total	37.5	18.6	

<sup>2</sup> R1, soft oily gum; R2, soft gum; these gums gradually became harder and more viscid until for R20 gum is very hard and brittle.

TABLE 19.—*Summary of analysis of rod wax 2 from Clay district, West Virginia—Continued*

CRUDE-WAX FRACTIONS COMBINED AND RECRYSTALLIZED WITH ETHYLENE DICHLORIDE

No.	Crude-wax fractions combined	Total crude-wax fractions, per cent	Color	Melting point, ° F.	Index of refraction, 183° F. (84° C.)	Density, 183° F. (84° C.)	Crystalline habit	Physical characteristics
CF1 <sup>3</sup>	F1-----	3.4	White...	141.6	1.4342	0.7838	Large plates, folding, and needles.	Soft, oily wax.
SCF1 <sup>4</sup>	...do-----	1.7	...do-----	111.5	1.4404	.7935	...do-----	Do.
CF2	F2-F3-----	7.2	...do-----	160.5	1.4337	.7797	...do-----	Hard wax.
SCF2	...do-----	2.3	...do-----	107.6	1.4515	.8148	Needles and folded plates.	Soft wax.
CF3	F4-F5-----	13.5	...do-----	168.6	1.4339	.7809	Large plates, folding, and needles.	Hard wax.
SCF3	...do-----	1.1	...do-----	151.9	1.4471	.8063	Needles and folded plates.	Do.
CF4	F6-F7-----	14.9	...do-----	171.8	1.4344	.7824	Large plates, folding, and needles.	Hard, brittle wax.
SCF4	...do-----	1.0	...do-----	161.6	1.4431	.7987	...do-----	Hard wax.
CF5	F8-F9-----	12.5	...do-----	173.7	1.4359	.7848	...do-----	Hard, brittle wax.
SCF5	...do-----	.3	...do-----	163.8	1.4434	.7992	...do-----	Hard wax.
CF6	F10-F11-----	10.9	...do-----	175.5	1.4372	.7871	...do-----	Hard, brittle wax.
SCF6	...do-----	.5	...do-----	167.5	1.4441	.8006	Needles, folding, and small plates.	Hard wax.
CF7	F12-F13-F14	14.0	...do-----	176.7	1.4386	.7890	Sm aller plates and needles.	Do.
SCF7	...do-----	1.1	...do-----	168.4	1.4413	.7952	...do-----	Do.
CF8	F15-F16-F17-F18-F19.	9.4	...do-----	176.4	1.4422	.7972	...do-----	Do.
SCF8	...do-----	2.3	...do-----	168.2	1.4445	.8013	Small plates and needles.	Hard, gummy wax.
CF9	F-20-----	2.7	...do-----	180.5	1.4436	.7996	...do-----	Do.
SCF9	...do-----	1.2	...do-----	176.0	1.4446	.8015	...do-----	Do.

<sup>3</sup> CF fractions are those which crystallized from the ethylene dichloride.  
<sup>4</sup> SCF fractions are those which remained in solution in the ethylene dichloride.

NOTE.—Total white-wax fractions-----	80.6
Total gums and resins-----	18.6
Total "asphaltenes"-----	.0
	99.2
Loss-----	.8
	100.0

TABLE 20.—*Summary of analysis of rod wax 3 from Panhandle district, Texas*

[Melting point of crude rod-wax sample, 141.2° F. (60.7° C.); weight of crude rod-wax sample, 296.3 grams; weight of oil-free rod-wax sample, 225.0 grams, 75.8 per cent; weight of crude oil removed from rod wax, 71.6 grams, 24.2 per cent]

FRACTIONS SEPARATED FROM ROD WAX WITH HOT ACETONE AND PRECIPITATED AT 30° F. TO FORM CRUDE-WAX FRACTIONS

No.	Weight, grams	Oil-free rod wax, per cent	Acetone used, liters	Color	After removing gums and resins with fuller's earth			
					Weight, grams	Oil-free rod wax, per cent	Color	Melting point, ° F.
F1	13.2	5.9	2.0	Light brown	13.1	5.8	White	145.0
F2	9.5	4.2	2.0	do	9.0	4.0	do	149.8
F3	11.2	5.0	4.0	do	11.1	4.9	do	154.2
F4	11.3	5.0	5.0	do	10.8	4.8	do	157.2
F5	11.5	5.1	6.0	do	10.9	4.8	do	158.8
F6	9.7	4.3	7.0	do	8.7	3.9	do	160.4
F7	9.9	4.4	10.0	do	7.6	3.4	do	162.3
F8	11.5	5.1	15.0	do	11.1	4.9	do	163.6
F9	11.3	5.0	20.0	do	10.9	4.8	do	165.4
F10	12.2	5.4	30.0	do	11.7	5.2	do	167.0
F11	11.9	5.3	40.0	do	11.1	4.9	do	168.2
F12	13.4	6.0	50.0	do	12.0	5.3	do	170.1
F13	13.4	6.0	60.0	do	11.5	5.1	do	172.2
F14	11.9	5.3	80.0	do	9.7	4.3	do	172.2
F15	11.6	5.2	105.0	do	6.2	2.8	do	172.2
F16	28.0	12.4	Residue	Very dark brown	15.7	7.0	Light yellow	172.1
Total	201.5	89.6			171.7	76.3		

GUMS<sup>1</sup> AND RESINS SOLUBLE IN ACETONE AT 30° F. PLUS THOSE REMOVED FROM CRUDE-WAX FRACTIONS WITH FULLER'S EARTH

No.	Weight, grams	Oil-free wax, per cent	Color	No.	Weight, grams	Oil-free wax, per cent	Color
R1	4.2	1.9	Light brown.	R10	3.0	1.3	Light brown.
R2	2.4	1.1	Do.	R11	3.0	1.3	Do.
R3	2.3	1.0	Do.	R12	2.7	1.2	Do.
R4	1.0	.4	Do.	R13	2.8	1.2	Do.
R5	1.1	.5	Do.	R14	3.3	1.5	Do.
R6	1.7	.8	Do.	R15	4.3	1.9	Do.
R7	1.4	6.0	Do.	R16	7.1	3.2	Do.
R8	3.0	1.3	Do.				
R9	1.3	.6	Do.	Total	44.5	19.8	

<sup>1</sup> R1, soft oily gum; R2, soft gum; these gums gradually became harder and more viscid until for R16 gum was very hard and brittle.

TABLE 20.—*Summary of analysis of rod wax 3 from Panhandle district, Texas—Continued*

CRUDE-WAX FRACTIONS COMBINED AND RECRYSTALLIZED WITH ETHYLENE DICHLORIDE

No.	Crude-wax fractions combined	Total crude-wax fractions, per cent	Color	Melt-ing point, ° F.	Index of refraction, 183° F. (84° C.)	Den-sity, 183° F. (84° C.)	Crystalline habit	Physical characteristics
CF1 <sup>2</sup>	F1-F2	8.4	White	150.2	1.4300	0.7732	Large plates, folding, and needles.	Hard wax.
SCF1 <sup>3</sup>	do	1.2	do	138.3	1.4395	.7917	do	Soft wax.
CF2	F3-F4	12.4	do	156.7	1.4316	.7767	do	Hard, brittle wax.
SCF2	do	2.4	do	129.3	1.4334	.7797	do	Soft wax.
CF3	F5-F6	10.0	do	159.7	1.4323	.7778	do	Hard, brittle wax.
SCF3	do	.4	do	139.4	1.4337	.7805	do	Hard wax.
CF4	F7-F8	10.4	do	163.1	1.4335	.7809	do	Hard, brittle wax.
SCF4	do	.4	do	152.2	1.4410	.7947	do	Hard wax.
CF5	F9-F10	11.2	do	165.9	1.4345	.7832	do	Hard, brittle wax.
SCF5	do	.8	do	156.9	1.4404	.7935	do	Hard wax.
CF6	F11-F12	13.8	do	169.4	1.4368	.7864	do	Hard, brittle wax.
SCF6	do	2.7	do	164.6	1.4383	.7894	Needles, small plates, and folding.	Hard wax.
CF7	F13-F14-F15	13.8	do	172.2	1.4390	.7907	do	Do.
SCF7	do	2.6	do	164.0	1.4421	.7969	do	Hard, gummy wax.
CF8	F16	7.0	do	172.1	1.4503	.8126	Needles, very small plates, and folding.	Do.
SCF8	do	3.0	Yellow	169.4	1.4532	.8181	do	Do.

<sup>2</sup> CF fractions are those which crystallized from ethylene dichloride.  
<sup>3</sup> SCF fractions are those which remained in solution in ethylene dichloride.

	Per cent
NOTE.—Total white wax fractions	76.3
Total gums and resins	19.8
Total "asphaltenes"	.2
	96.3
Loss	3.7
	100.0

TABLE 21.—*Summary of analysis of rod wax ¼ from Salt Creek, Wyo.*

[Melting point of crude rod-wax sample, 154.6° F. (68.6° C.); weight of crude rod-wax sample, 309.4 grams;<sup>1</sup> weight of oil-free rod-wax sample, 190.5 grams, 62 per cent; weight of crude oil removed from rod wax, 118.9 grams, 38 per cent]

FRACTIONS SEPARATED FROM ROD WAX WITH HOT ACETONE AND PRECIPITATED AT 30° F. TO FORM CRUDE-WAX FRACTIONS

No.	Weight, grams	Oil-free rod wax, per cent	Acetone used, liters	Color	After removing gums and resins with fuller's earth			Melting point, ° F.
					Weight, grams	Oil-free rod wax, per cent	Color	
F1-----	14.5	7.6	1.0	Light brown-----	14.0	7.3	White-----	142.6
F2-----	8.4	4.4	1.5	do-----	8.1	4.3	do-----	139.4
F3-----	13.2	6.9	3.0	do-----	12.7	6.7	do-----	143.8
F4-----	10.1	5.3	4.0	do-----	9.3	4.9	do-----	150.9
F5-----	10.6	5.6	5.0	do-----	9.8	5.1	do-----	152.9
F6-----	11.4	6.0	6.0	do-----	10.6	5.6	do-----	154.0
F7-----	9.4	4.9	7.0	do-----	8.6	4.5	do-----	156.9
F8-----	8.0	4.2	8.0	do-----	7.2	3.8	do-----	158.5
F9-----	8.6	4.5	11.5	do-----	7.8	4.1	do-----	160.5
F10-----	9.1	4.8	16.0	do-----	8.5	4.5	do-----	162.2
F11-----	8.5	4.5	21.0	do-----	8.0	4.2	do-----	165.3
F12-----	10.9	5.7	26.0	do-----	10.5	5.5	do-----	165.6
F13-----	10.1	5.3	45.0	do-----	9.6	5.0	do-----	171.4
F14-----	10.8	5.7	75.0	do-----	10.1	5.3	do-----	176.3
F15-----	8.1	4.3	Residue--	Dark brown-----	7.1	3.7	do-----	176.7
Total--	151.7	79.6	-----	-----	141.9	74.5	-----	-----

<sup>1</sup> 75 per cent acetone; 25 per cent ethylene dichloride.

GUMS<sup>2</sup> AND RESINS SOLUBLE IN ACETONE AT 30° F. PLUS THOSE REMOVED FROM THE CRUDE-WAX FRACTIONS WITH FULLER'S EARTH

No.	Weight, grams	Oil-free rod wax, per cent	Color	No.	Weight, grams	Oil-free rod wax, per cent	Color
R1-----	7.2	3.8	Brown.	R10-----	1.0	0.5	Brown.
R2-----	3.5	1.8	Do.	R11-----	1.2	.6	Do.
R3-----	4.5	2.4	Do.	R12-----	6.9	3.6	Do.
R4-----	1.9	1.0	Do.	R13-----	2.5	1.3	Do.
R5-----	1.8	.9	Do.	R14-----	2.8	1.5	Do.
R6-----	2.0	1.0	Do.	R15-----	1.0	.5	Do.
R7-----	1.5	.8	Do.				
R8-----	1.3	.7	Do.	Total-----	40.3	21.2	
R9-----	1.2	.6	Do.				

<sup>2</sup> Soft oily gum; these gums gradually became harder and more viscid until for R15 gum was hard and brittle.

TABLE 21.—Summary of analysis of rod wax 4 from Salt Creek, Wyo.—Contd.  
CRUDE-WAX FRACTIONS COMBINED AND RECRYSTALLIZED WITH ETHYLENE  
DICHLORIDE

No.	Crude-wax fractions combined	Total crude-wax fraction, per cent	Color	Melting point, ° F.	Index of refraction, 183° F. (84° C.)	Density, 183° F. (84° C.)	Crystalline habit	Physical characteristics
CF1 <sup>3</sup>	F1-F2-F3	12.3	White	146.6	1.4299	0.7734	Large plates, folding, and needles.	Soft wax.
SCF1 <sup>4</sup>	do	5.0	do	119.9	1.4393	.7913	do	Soft, oily wax.
CF2	F4-F5	13.1	do	153.3	1.4316	.7765	do	Hard wax.
SCF2	do	2.3	do	140.1	1.4394	.7915	do	Do.
CF3	F6-F7	13.9	do	155.6	1.4331	.7793	do	Hard, brittle wax.
SCF3	do	1.4	do	142.4	1.4430	.7985	do	Hard wax.
CF4	F8-F9-F10	15.4	do	160.8	1.4344	.7813	do	Hard, brittle wax.
SCF4	do	2.8	do	153.6	1.4363	.7855	Needles, small plates, folding.	Hard wax.
CF5	F11-F12	10.0	do	167.2	1.4358	.7839	Large plates, folding, and needles.	Hard, brittle wax.
SCF5	do	3.6	do	158.0	1.4403	.7933	Needles, small plates, folding.	Hard wax.
CF6	F13-F14	9.5	do	174.9	1.4371	.7868	do	Do.
SCF6	do	5.2	do	164.4	1.4416	.7985	do	Hard, gummy wax.
CF7	F15	4.4	do	179.5	1.4413	.7952	do	Hard wax.
SCF7	do	1.0	Opaque.	163.6	1.4517	.8125	do	Hard, gummy wax.

<sup>3</sup> CF fractions are those which crystallized from the ethylene dichloride.

<sup>4</sup> SCF fractions are those which remained in solution in the ethylene dichloride.

NOTE.—Total wax fractions	Per cent
Total gums and resins	74.5
Total "asphaltenes"	21.2
	3.0
	98.7
Loss	1.3
	100.0

A large quantity of oil present in the rod wax tends to make it soft, semifluid, and more readily affected by heat, regardless of the melting point of the waxes present, because the oil dissolves and softens part of the wax when the temperature is increased. If very little oil is present the rod wax is hard and less plastic, and an increase in temperature does not have much effect on the plasticity until it approaches the melting point of the solid waxes present.

#### GUMS AND RESINS

The gums and resins found in the rod waxes are reddish-brown unsaturated compounds originally in solution in the crude oil. The gums and resins from the four samples of rod wax had the same general characteristics and seemed to belong to the same or similar types of unsaturated hydrocarbons. Probably a part of these gums and resins were sulphur and oxygen compounds.<sup>96</sup> In the fractionation of the rod waxes the gums decreased in solubility and increased in hardness. The more soluble gums and resins were of a soft, oily consistency and not viscid; as the fractions became more insoluble the gums became harder and more viscid, and the last fractions were hard and brittle although on warming they became soft but very

<sup>96</sup> von Pilat, K., and Dukett, J., Resinification of Paraffin Oils: Erdöl u. Teer, vol. 2, 1926, p. 571.

viscid. These gums, as will be discussed later, were found to exhibit a very marked effect on the crystalline behavior of the wax fractions obtained from the rod waxes.

The rod wax from the light-colored paraffin-base, Bradford (Pa.) crude oil contained 14.5 per cent of gums and resins; the rod wax from the slightly darker paraffin-base crude oil from Clay district, West Virginia, contained 18.6 per cent of gums and resins; the rod wax from the dark-green paraffin-base crude oil from the Panhandle district, Texas, contained 19.8 per cent of gums and resins; and the rod wax from the dark-green mixed-base crude oil from Salt Creek, Wyo., contained 21.2 per cent of gums and resins.

The presence of gums and resins in rod waxes probably is due partly to their absorption<sup>97</sup> by the wax as it crystallizes from solution and is also a result of decreased solubility due to the same conditions which cause separation of the wax from solution in the oil. These gums and resins can be redissolved in crude oil by increasing the temperature, and they are also soluble in the lighter fractions of crude oil.

#### " ASPHALTENES "

The quantity of " asphaltenes " present in the rod waxes was small, especially in the waxes from the paraffin-base crudes. The quantity of " asphaltenes " in the Bradford (Pa.) rod wax was 0.2 per cent; in the Clay district (West Virginia) rod wax, a trace; in the Panhandle district (Texas) rod wax, 0.2 per cent; and in the Salt Creek (Wyo.) rod wax, 3 per cent. Because " asphaltenes " are insoluble in acetone the solvent was used to remove the oil from the wax, and because the quantity of " asphaltenes " found in the wax was approximately the same as would be present naturally in the quantity of oil removed from the wax the authors believe that the " asphaltenes " were not actually precipitated from the crude oil with the other constituents of the rod wax but were in solution in the crude oil associated with the rod wax and that they did not form a part of the actual solid constituents.

#### WAX

The most important and by far the largest constituent of all the rod waxes was wax. The total wax content in the four rod waxes was as follows: Bradford, Pa., 81.6 per cent; Clay district, West Virginia, 80.6 per cent; Panhandle district, Texas, 76.3 per cent; and Salt Creek, Wyo., 74.5 per cent. The rod waxes were fractionated into 15 to 20 fractions, as previously described, and the gums and resins were removed from these fractions leaving the " total wax." After the melting point was determined, the fractions were combined as shown in Tables 18, 19, 20, and 21, to reduce the number of fractions and to increase the size of the samples. The index of refraction and melting point of the combined fractions, particularly the more insoluble ones, showed such a wide divergence from the values of Buchler and Graves,<sup>98</sup> that it was thought ad-

<sup>97</sup> Chernoshukov, N., Adsorption of Resins by Paraffin Wax and Solidification of Paraffin-Containing Products: *Neftjanoe i slancevoe Chozjajstvo*, vol. 8, 1925, pp. 640-646.

<sup>98</sup> See footnote 8, p. 8.

visible to recrystallize all the fractions in ethylene dichloride to determine if the divergence was due to the presence of "soft-wax" impurities in the wax or if the waxes found in rod wax were different from those of Buchler and Graves. Two series of fractions were obtained—those crystallized from the ethylene dichloride, or CF fractions, and those recovered from the ethylene dichloride solution by distillation of the solvent, or SCF fractions. In some

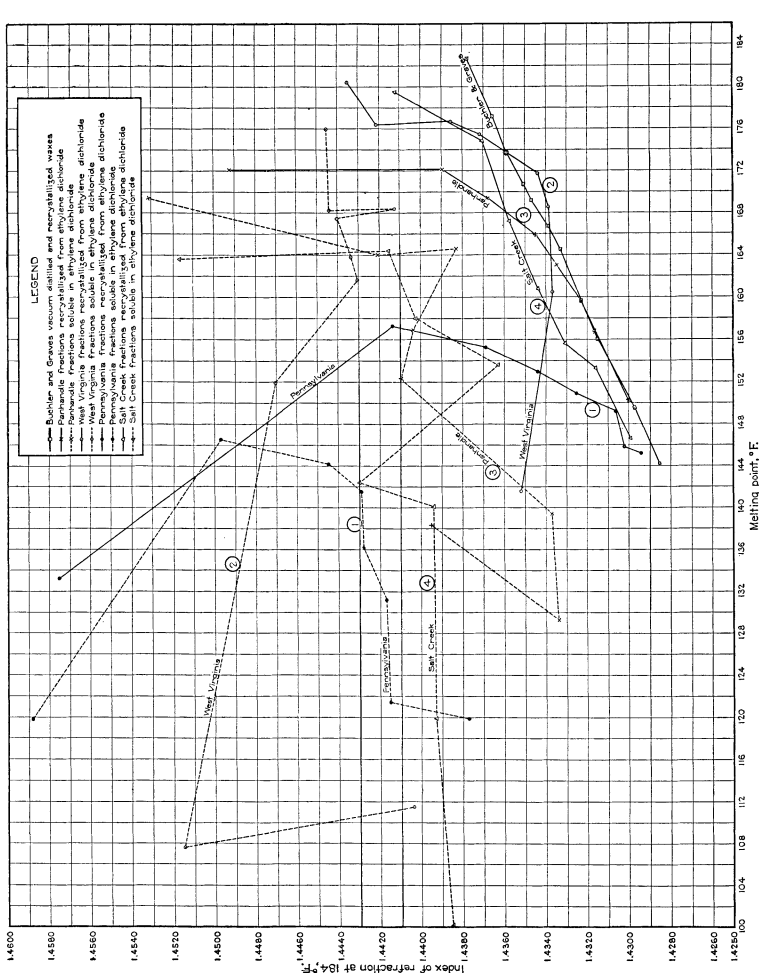


Figure 53.—Relationship between melting point and indexes of refraction of wax fractions taken from rod wax

cases the recrystallization was repeated several times. The color, index of refraction, density, and crystalline behavior of the fractions were then studied.

The relationship between the melting points and indexes of refraction of the fractions (CF 1, 2, and 3, from rod wax 1; CF 2, 3, 4, 5, and 6, from rod wax 2; CF 1, 2, 3, 4, and 5, from rod wax 3; and CF 1, 2, 3, 4, 5, and 6, from rod wax 4), plotted on coordinate paper and shown by Figure 53, indicates that these fractions are



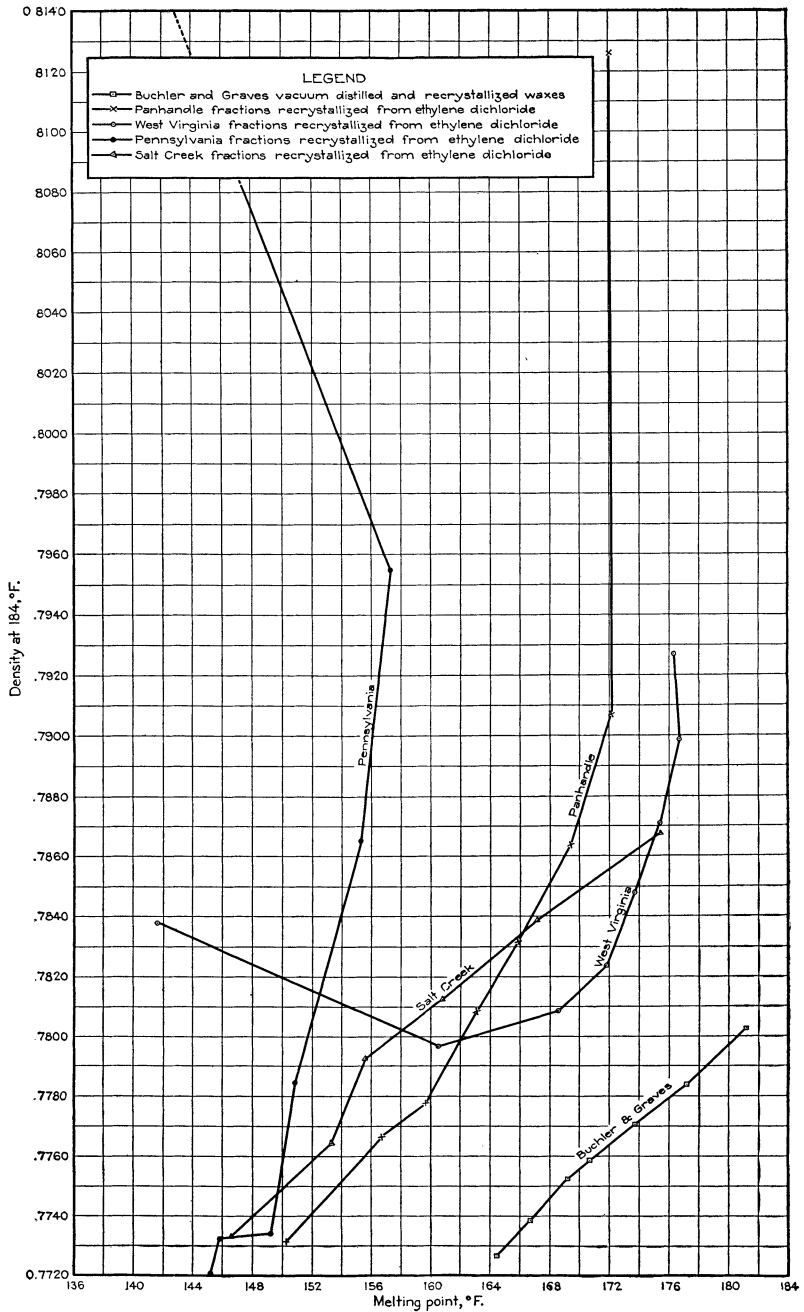


FIGURE 54.—Relationship between melting point and density of wax fractions taken from rod wax

probably similar to the crystalline waxes obtained by Buchler and Graves<sup>99</sup> and that, by further crystallization, the curve showing the properties of these waxes would fall much nearer the curve of Buchler and Graves. The fractions are therefore probably members of the straight-chain saturated-paraffin series,  $C_nH_{2n+2}$ . The relationship between melting point and density, as shown in Figure 54, is not as close as that of the same properties determined by Buchler and Graves, but the difference is probably the result of the difference in the methods used in determining the density of the waxes. The method used by the authors is given in the Appendix of the report.

Fraction CF 1 from rod wax 2 had an index of refraction considerably higher with respect to melting point than the Buchler and Graves values, probably owing to the known presence of an appreciable quantity of oil which was not completely removed from the rod wax before the first fractionation was made. If this oil had been removed, the values would probably more closely approach the Buchler and Graves values.

The fractions CF 4, 5, 6, 7, and 8, from rod wax 1; CF 7, 8, and 9, from rod wax 2; CF 6, 7, and 8, from rod wax 3; CF 7, from rod wax 4; and the SCF fractions from all the rod waxes show marked divergence in the relationship between melting point and index of refraction from the same value of Buchler and Graves for the straight-chain paraffin hydrocarbons. The last fraction of wax obtained from each rod wax was very gummy and had a much higher index of refraction and density than any of the other fractions, and it seemed to be composed primarily of the "soft wax" discussed by Buchler and Graves or "mineral jelly" described by Carpenter.<sup>1</sup> This gummy material is much less soluble than the crystalline waxes; and it is therefore most predominant in the last fraction or residue, although it is also found in increasing quantities in the other fractions. Its high index of refraction and density at least partly account for the increasing divergence of the higher melting-point wax fractions from these same values for the waxes of Buchler and Graves; however, in fractions CF 4, 5, 6, 7, and 8, from rod wax 1, and in many of the SCF fractions the divergence of their melting point and index of refraction from the same values for the Buchler and Graves waxes is not due entirely to the presence of "soft wax" or "mineral jelly." In view of the hard, crystalline nature of these fractions and the fact that, in CF 5, 6, and 7, from rod wax 1, repeated recrystallizations did not tend to bring their index of refraction and melting point any nearer the values of Buchler and Graves, it seems likely that some waxes are present in the rod wax that belong to a series other than the homologous straight chain  $C_nH_{2n+2}$ , which is more in accordance with the findings of Ferris, Cowles, and Henderson.<sup>2</sup>

The physical characteristics of the SCF fractions are very erratic, because these fractions were not recrystallized after the removal of the CF fractions, and they are therefore composed of most of the "soft wax" contained by the original F fraction and varying amounts of the crystalline waxes.

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<sup>99</sup> See footnote 8, p. 8.

<sup>1</sup> See footnote 10, p. 11.

<sup>2</sup> See footnote 11, p. 11.

## CRYSTALLINE BEHAVIOR OF WAX FRACTIONS

The crystalline characteristics of the wax fractions with and without a solvent were studied under a microscope. In the preparation of the slides for the crystalline study without a solvent hanging-drop slides were made, and these were cooled from a temperature 10° F. above the melting point of the wax to room temperature in an oven in which the temperature dropped 1° F. in 10 minutes. The solvent used in studying the crystalline behavior of the waxes was 98 per cent ethyl alcohol. Concentrations of 1 part of wax in 100, 500, 1,000, 2,000, and 10,000 parts of alcohol were used. The solutions of wax and alcohol were heated to 158° F. (70° C.) and then cooled in the oven at the rate of 1° F. in 10 minutes.

Typical illustrations of the crystals produced by the slow cooling of the CF wax fractions without a solvent are shown in Figures 55, *A* and *B*; 56, *A* and *B*; and 57, *A*, *B*, and *C*. These crystals were predominantly bladelike crystals or large plates with folding<sup>3</sup> and well-formed needles. The formation of needles or sharper folding of the plates apparently becomes more pronounced as the melting point and quantity of waxes, other than the normal straight-chain series and "soft wax," increase. The fraction SCF 8 from the Bradford wax, in which a large quantity of the "soft wax" impurity was present, has the appearance of petrolatum, as may be noted in Figure 57, *C*. No large plates, foldings, or needles were seen; and very small plates seem to predominate. The individual crystals are small and well formed. They are very thin, rhombic plates having angles of 70° and 110°, similar to those shown in Figure 58. Usually, a profusion of very small individual crystals, similar to those shown in Figure 59, were found on the cover glass adjacent to the larger hanging drops of wax. These small crystals were identical in optical characteristics and shape to the wax crystals obtained by Tanaka, Kobayashi, and Ohno<sup>4</sup> when crystallizing paraffin wax from absolute alcohol. Tanaka and his coworkers concluded that the fundamental and most stable arrangement of paraffin molecules produces the diamond-shape rhombic crystals with angles of 70° and 110°, while the less stable arrangement is the needle form. They also believe that the needle is a multiple twining or parallel growth of the rhombic plates in the direction of the 70° angle while Rhodes, Mason, and Sutton<sup>5</sup> conclude that the needle form of crystal is the result of folding or curling of the plate crystal.

The optical properties and crystal system of the wax crystals, regardless of whether they were plate or needle form, were similar for all the CF and SCF fractions, irrespective of their source or of the quantity of "soft wax" present. The crystals show a distinct biaxial interference figure, optically positive with 2 *V* approximately 25° to 30° and 2 *E* approximately 45°. The large plates when viewed flatwise are isotropic or exhibit only a very weak double refraction, but when viewed edgewise or inclined they are strongly

<sup>3</sup> Rhodes, F. H., Mason, C. W., and Sutton, W. R., *Crystallization of Paraffin Wax: Ind. and Eng. Chem.*, vol. 19, 1927, pp. 935-938.

<sup>4</sup> Tanaka, Y., Kobayashi, R., and Ohno, S., *Studies on Crystallization of Paraffin: Jour. Faculty of Eng., Tokyo Imperial Univ.*, vol. 17, No. 15, Tokyo, Japan, November, 1928, pp. 275-293.

<sup>5</sup> Rhodes, F. H., Mason, C. W., and Sutton, W. R., *Crystallization of Paraffin Wax: Ind. and Eng. Chem.*, vol. 19, No. 8, August, 1927, pp. 935-938.

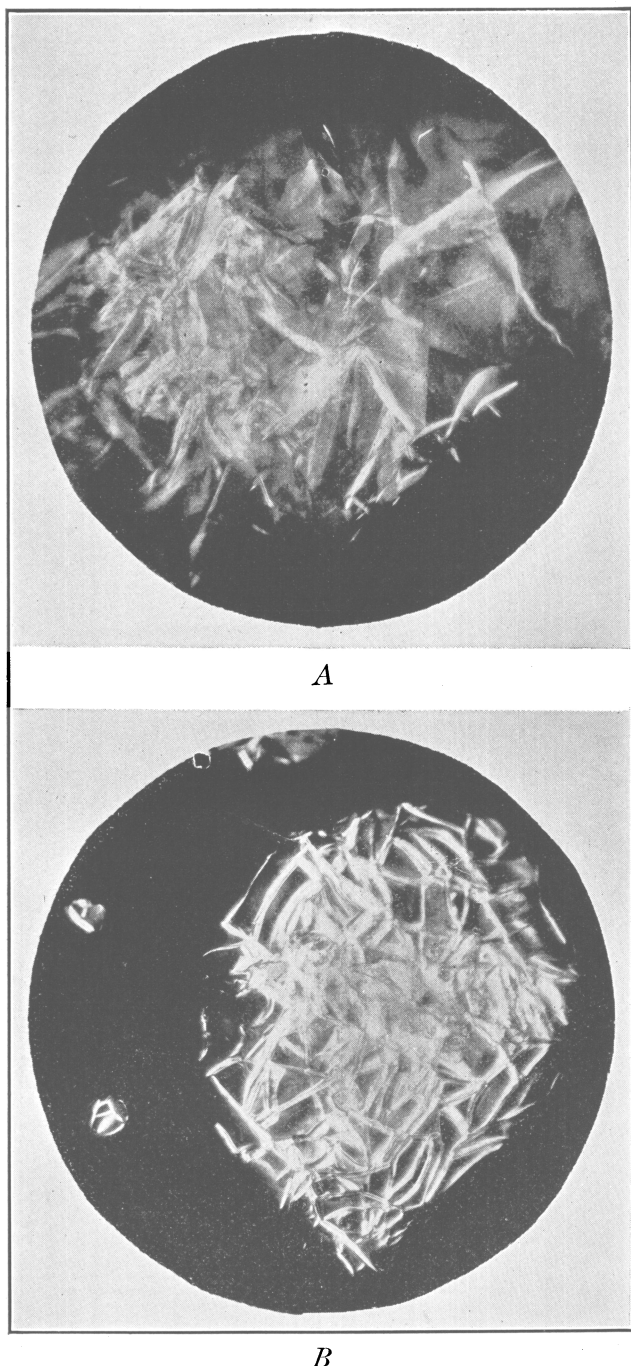


FIGURE 55.—Fractions of rod wax crystallized without presence of solvent: *A*, CF 1 rod wax No. 2; *B*, CF 2 rod wax No. 1. Magnification,  $\times 50$

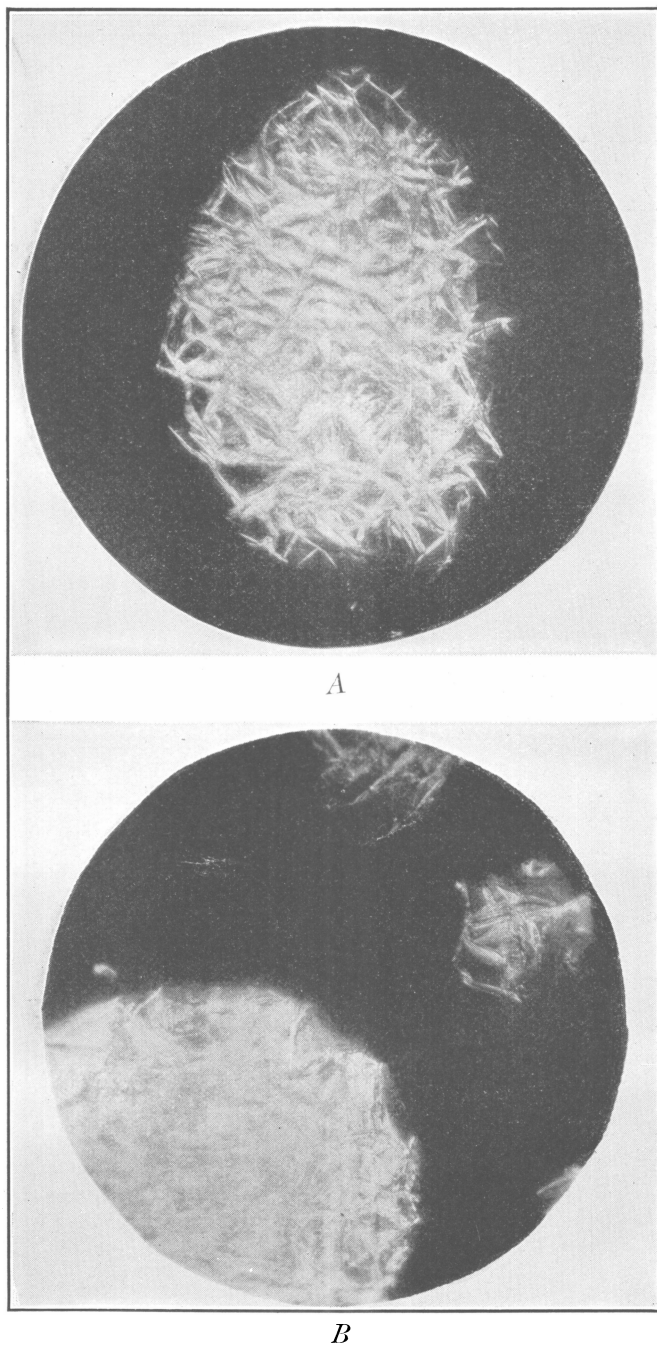


FIGURE 56.—Fractions of rod wax crystallized without presence of solvent: *A*, CF 3 rod wax No. 2; *B*, CF 3 rod wax No. 4. Magnification,  $\times 50$

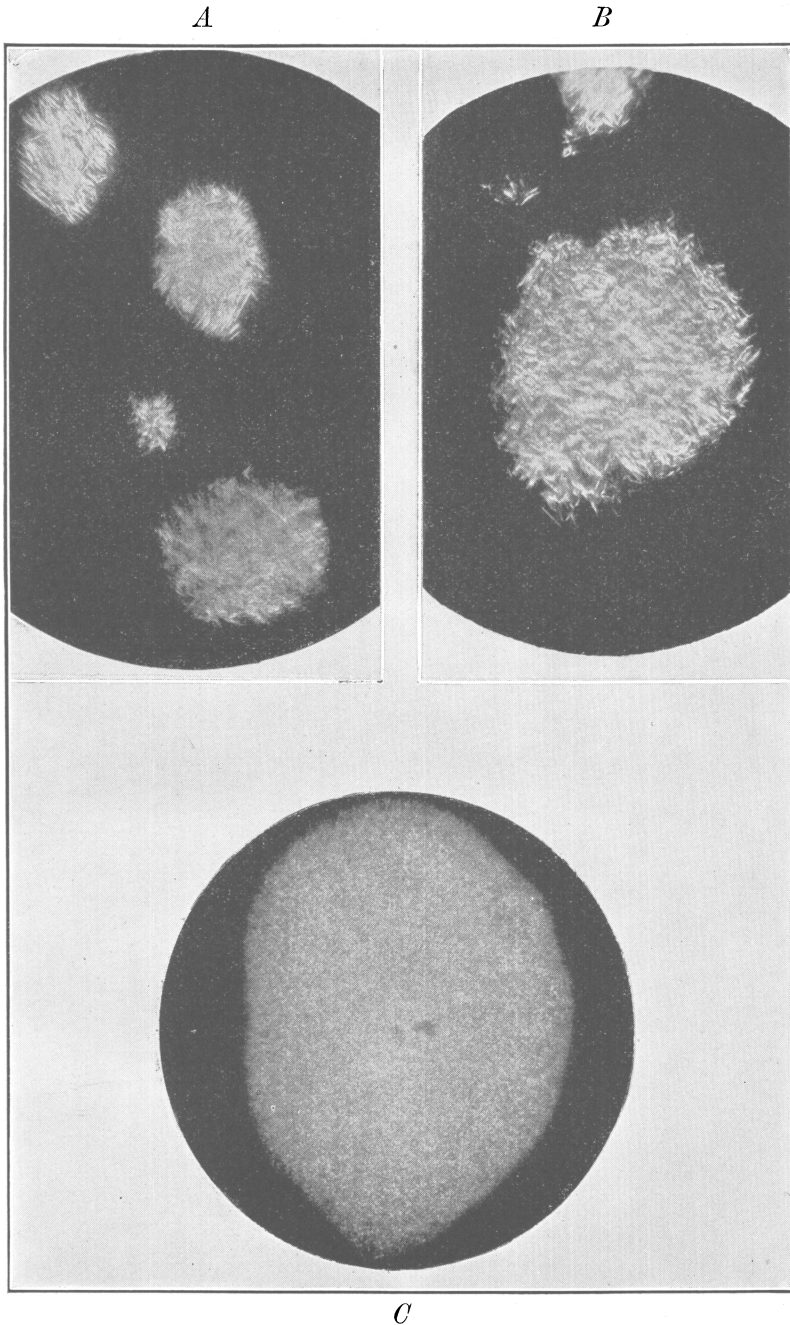


FIGURE 57.—Fractions of rod wax crystallized without presence of solvent: *A*, CF 7 rod wax No. 2; *B*, CF 7 rod wax No. 1; *C*, CF 8 rod wax No. 1. Magnification,  $\times 50$

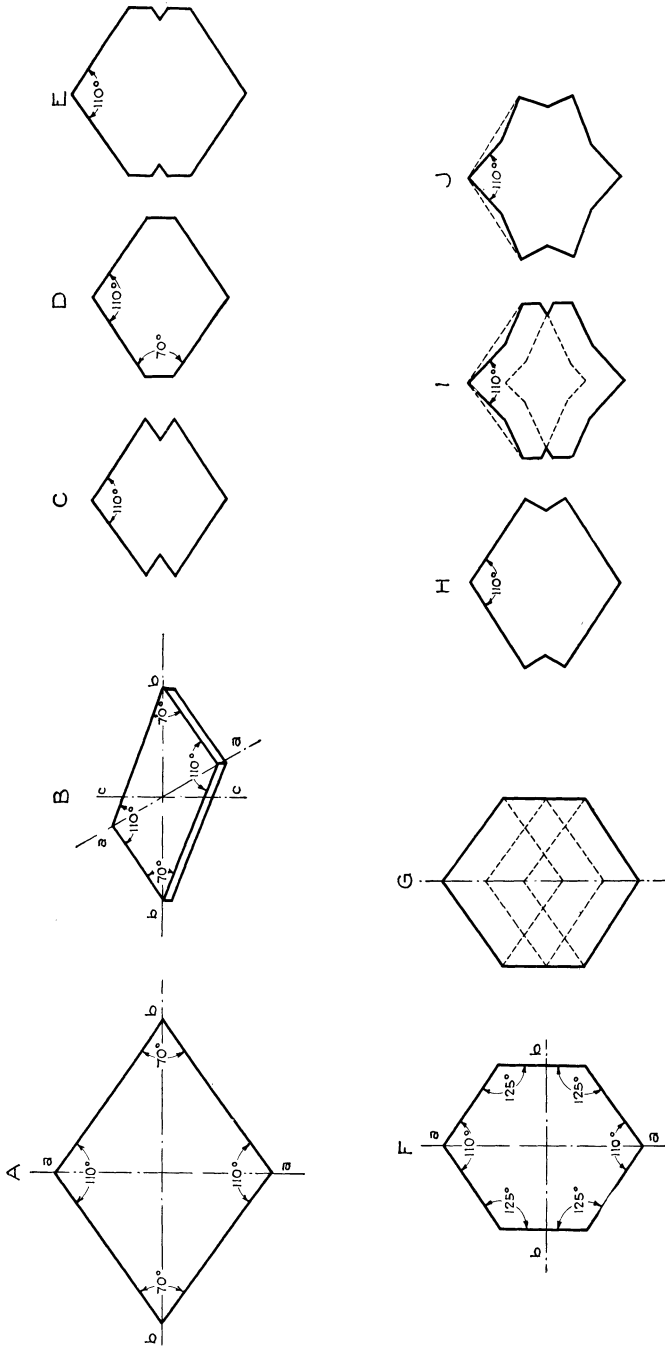


FIGURE 58.—Theoretical crystalline form of plate wax crystals, after Tanaka, Kobayshi, and Ohno

double refractive or anisotropic and exhibit parallel extinction. The small individual rhombic plates are double refractive in all three positions.

The crystallization of the paraffin wax fractions from the more concentrated alcoholic solutions resulted in needle forms of crystals as shown by Figure 60, *A* and *B*, while the more dilute solutions produced very thin rhombic plates shown in Figures 61, *A* and *B*, and 62, *A* and *B*. The plates were usually a complex form of a hexagonal shape and not the simple rhombic form with angles of

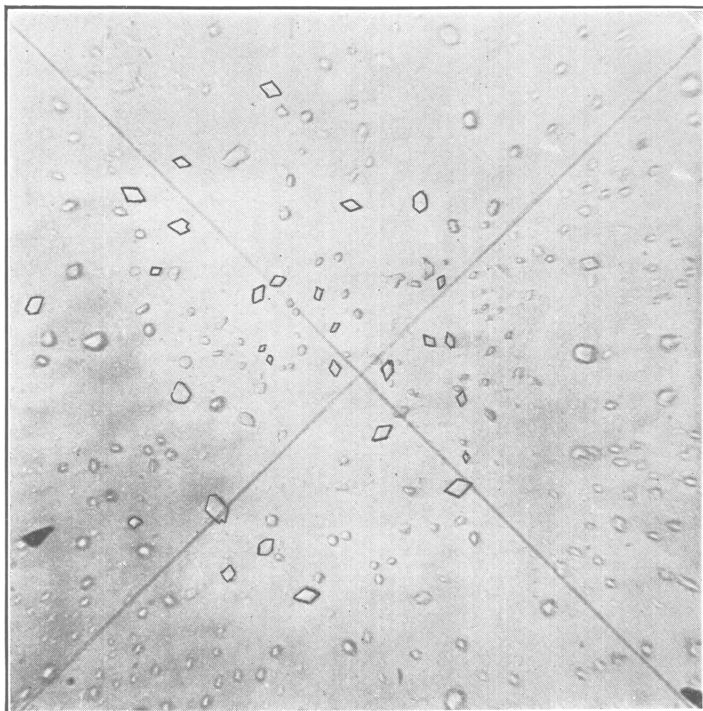


FIGURE 59.—Small individual crystals formed on cover glass from CF 1 rod wax No. 1. Magnification,  $\times 210$

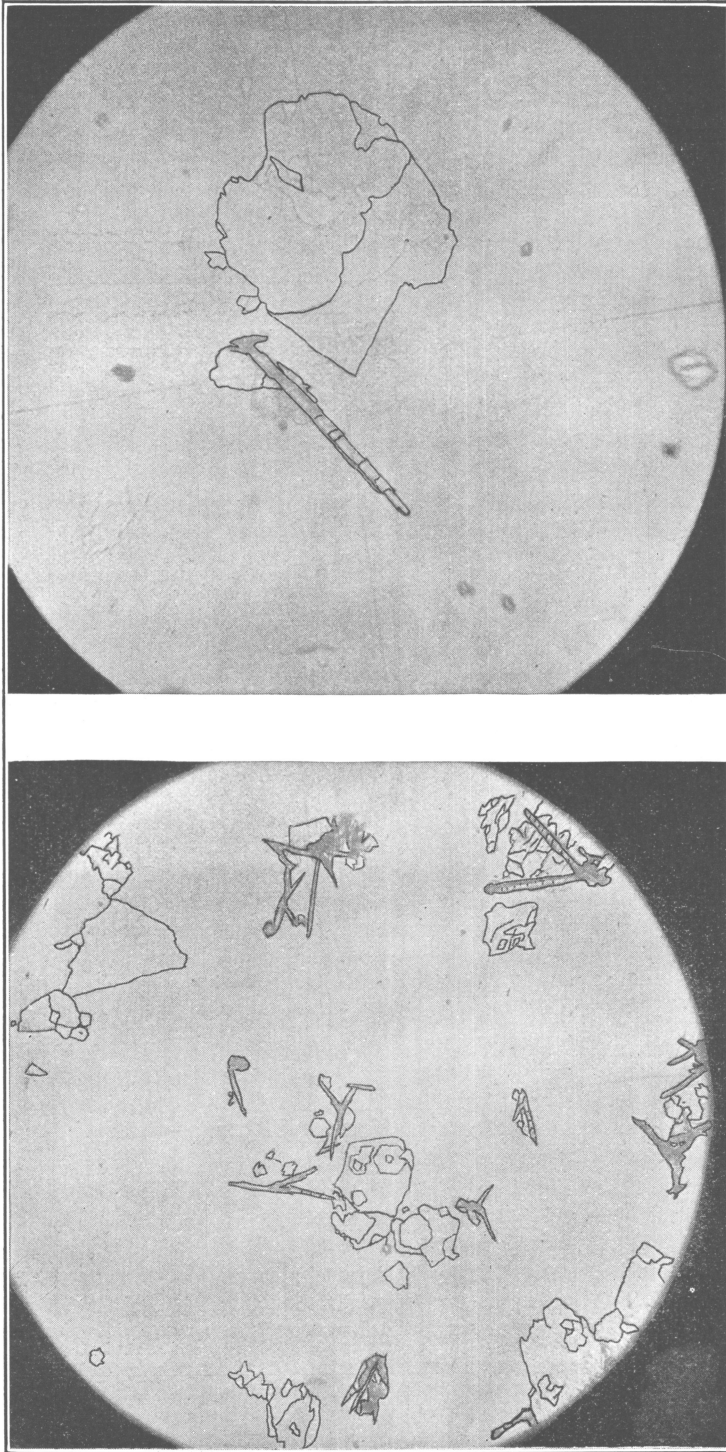
$70^\circ$  and  $110^\circ$ . At first inspection they appear to belong to the hexagonal crystal system, but measurement of the angles shows two angles of  $110^\circ$  and four of  $125^\circ$ . Consequently, they belong to the rhombic crystal system.

The probable structure of these complex plate crystals starting with the fundamental rhomb as given by Tanaka<sup>6</sup> and his coworkers is shown by Figure 58.

The behavior of the paraffin waxes and the formation of the needle and plate crystals seem to be more than an alteration of "habits" or change in the relative growth of the different crystal faces. It seems probable that the presence of "soft wax" impurity favors the forma-

<sup>6</sup> See footnote 21, p. 27.





A

B

FIGURE 60.—Fractions of rod wax crystallized from solutions of wax in absolute alcohol; A, CF 1 from rod wax No. 1, 1 part wax, 250 parts absolute alcohol, magnification  $\times 50$ ; B, CF 1 from rod wax No. 1, 1 part wax, 250 parts absolute alcohol, magnification,  $\times 200$

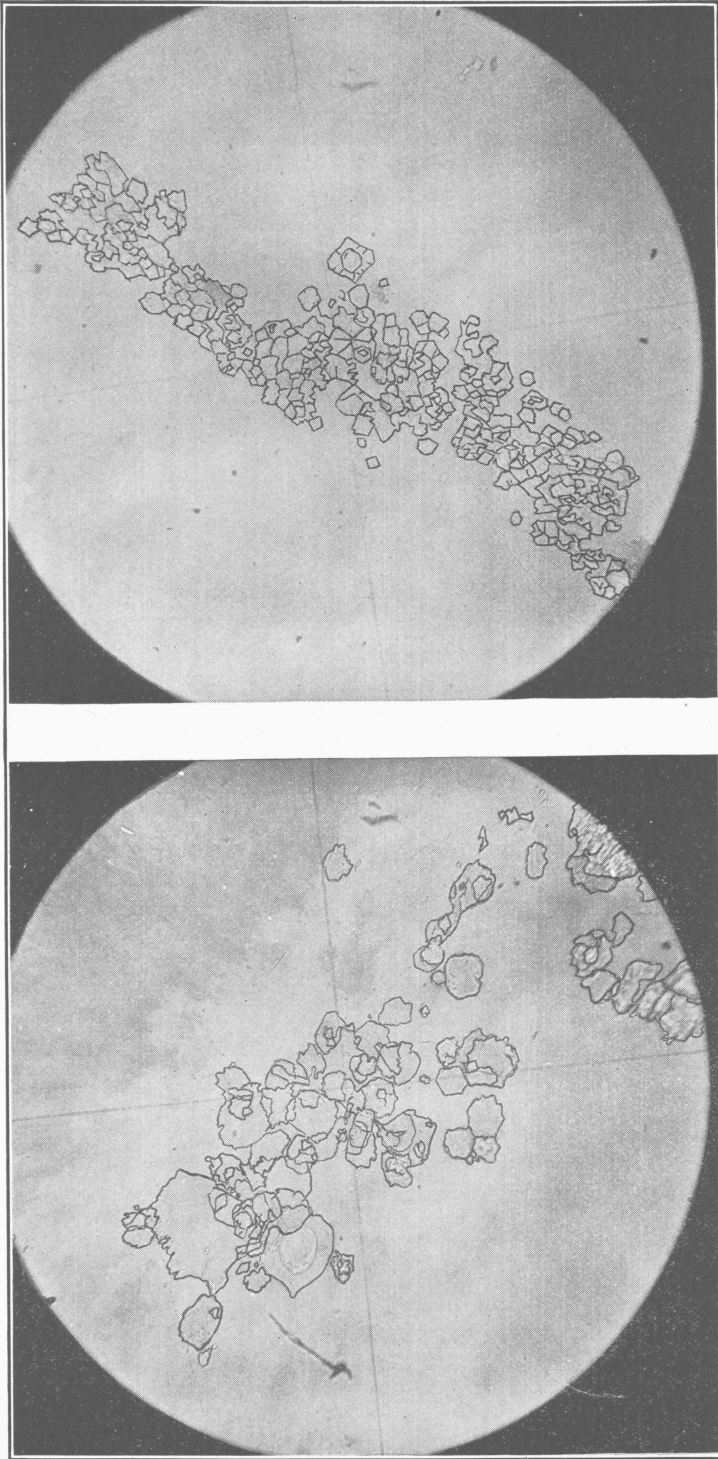


FIGURE 61.—Fractions of rod wax crystallized from solutions of wax in absolute alcohol; *A*, CF 1 from rod wax No. 1, 1 part wax, 1,000 parts absolute alcohol; *B*, CF 1 from rod wax No. 1, 1 part wax, 500 parts absolute alcohol. Magnification,  $\times 50$

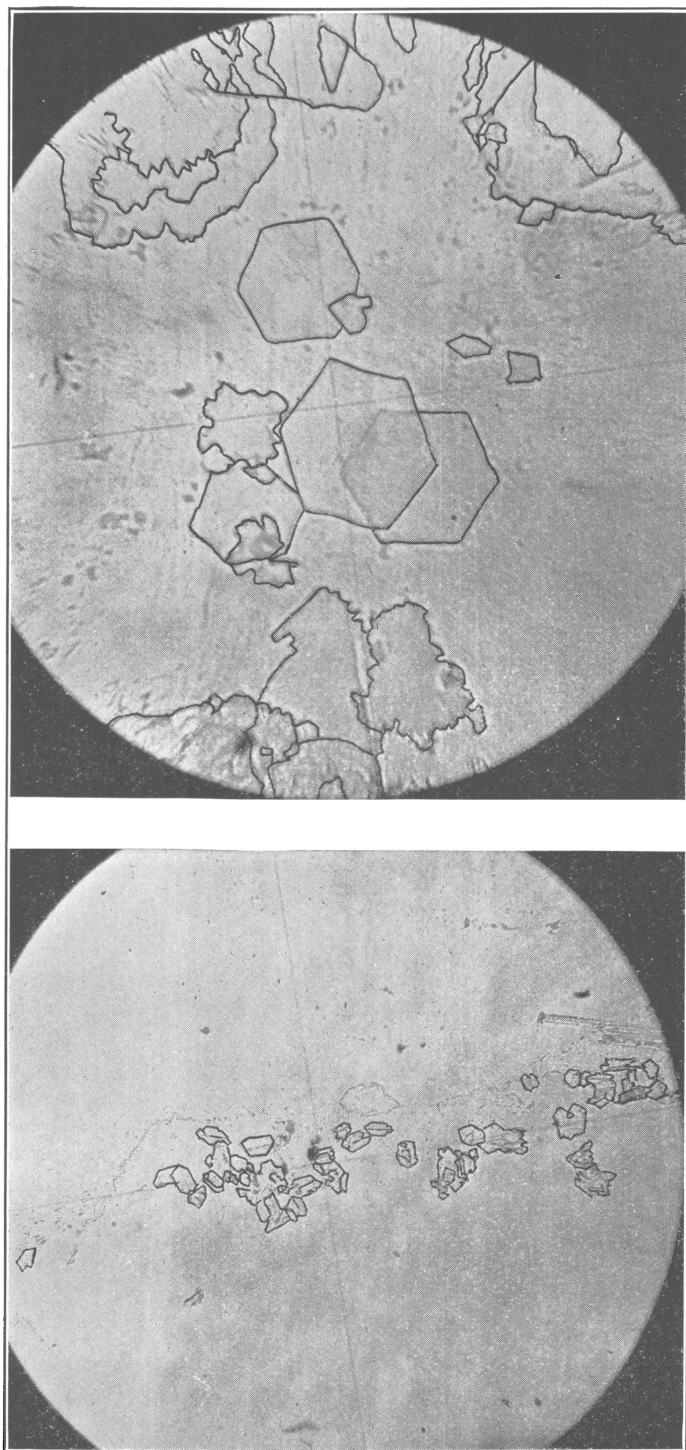
*A**B*

FIGURE 62.—Fractions of rod wax crystallized from solutions of wax in absolute alcohol: *A*, CF 8 from rod wax No. 2, 1 part wax, 250 parts absolute alcohol, magnification  $\times 50$ ; *B*, CF 1 from rod wax No. 3, 1 part wax, 500 parts alcohol, magnification,  $\times 200$ .

tion of needle crystal, in accordance with the theories of Buchler and Graves, because needle crystals or foldings occur in the large drops of wax crystals on the hanging drop slides, produced from the waxes in the absence of solvent, and in the more concentrated alcoholic solutions and because individual plate crystals are obtained on the cover glass of the hanging drop slides, possibly as a result of vaporization and condensation of pure wax from the larger drops, when heated in the oven previous to cooling, and from the more dilute alcoholic solutions. It was noted, however, that in the presence of large quantities of soft wax, such as the last fraction from each rod wax, the wax crystals were small plates. It was also evident that the viscosity and chemical characteristics of the medium from which the wax was crystallized and the presence of colloidal material produced changes in the crystalline habit of the wax. An example of the effect of gums and resins is shown in Figure 6, *A* and *B* (p. 14). These waxes are the same wax, but that in Figure 6, *A*, is the pure wax, whereas that in Figure 6, *B*, had a small quantity of the gums and resins added which had been removed previously from the wax by fuller's earth. Both crystallizations were made on the same slide under the same conditions.

#### SUMMARY

From the analyses of rod waxes it is evident that they are composed of substances precipitated from the oil as a result of disturbing the solution equilibrium and not as a result of a chemical reaction. The most important and largest components of rod waxes are the crystalline waxes. Although most of the crystalline waxes undoubtedly belong to the  $C_nH_{2n+2}$  homologous straight-chain paraffin series, there was also evidence, particularly in the Bradford rod wax, of the presence of crystalline waxes belonging to other series, probably isoparaffins or branch-chain series. In addition to these crystalline waxes there is also a quantity of "soft wax" or "mineral jelly" in rod wax. These waxes crystallized in the rhombic system to form plates or needle crystals, depending apparently upon the presence of "soft wax," gums, resins, colloidal material, and the viscosity of the crystallizing medium. The rhombic plate with angles of  $70^\circ$  and  $110^\circ$  is probably the true form of the pure paraffin wax crystal. The number of different individual compounds and types of waxes present in the rod wax depends upon the character of the crude oil and the factors causing the separation of the rod wax from solution in the oil. The waxes having the highest melting point and molecular weight present in the oil are the first to separate from solution, and the quantity and amount of the waxes of lower melting point or molecular weight present in the rod wax depend on the extent to which the oil was cooled or evaporated while deposition took place.

The gums and resins are precipitated from solution in the oil as a result of a decrease in their solubility, due to the same factors that cause the precipitation of the crystalline wax and to the absorption of these substances by the crystalline wax. Gums and resins are part of the little-known highly unsaturated constituents present in crude oils and probably consist of oxygen and sulphur compounds.

The "soft wax" or "mineral jelly" is present in the rod wax in only small proportions. "Soft wax" appears to be a very viscid liquid, or jelly, less soluble in acetone and alcohol but more soluble in ethylene dichloride than the crystalline wax. The density and index of refraction of the "soft wax" are much higher than those of the crystalline wax, which seems to indicate an unsaturated composition. The presence of "soft wax" in the rod wax can be accounted for in the same manner and as a result of the same conditions that are responsible for the presence of gums and resins.

## APPENDIX

### DESCRIPTION OF APPARATUS AND METHODS USED FOR SOLUBILITY DETERMINATIONS

As a result of the analysis of the rod waxes it was apparent that the physical and chemical characteristics of oils and paraffins from different fields were related closely enough so that information obtained from studying the solubility of one rod wax and oil would apply generally to all of them. It was decided therefore to confine the solubility determinations to a paraffin and oil from the Salt Creek field, Wyoming, which is near the Laramie office of the Bureau of Mines where the laboratory work was done, because the Salt Creek field is one of the major oil fields in the United States in which paraffin troubles are an important problem. The crude paraffin was taken from the casing of a flowing well and contained approximately 38 per cent of oil and some silt and sand. Its melting point was 165.4° F. The oil was removed from the sample of paraffin by repeated crystallizations from acetone. The oil-free paraffin consisted mainly of wax with a melting point above 150° F., gums, resins, and asphaltic material. It was found necessary to remove the small quantity of lower-melting-point waxes present before the paraffin could be used for solubility determinations, because the lower-melting-point waxes were very soluble in crude oil and would fractionate out, giving misleading results. The oil-free wax was therefore separated into two parts by use of acetone. One part consisted of the lower-melting-point fractions and represented 16 per cent of the original wax. It had a melting point of 138.6° F. The other part, which was 84 per cent of the original oil-free wax, had a melting point of 176.7° F. and contained 19 per cent of gums, resins, and asphaltic material.

The solubility of the different wax fractions and of the crude paraffin was determined by dissolving a weighed amount of wax in a known quantity of solvent and determining the temperature at which the wax started to separate from solution. This method, which is commonly referred to as the cloud-point method, has been used by other investigators in their study of wax solubility and found to be satisfactory.<sup>7</sup> To obtain a wax-free crude oil for the solubility work it was necessary to dewax a sample of the Salt Creek crude. Dewaxing was accomplished by distilling the gasoline, kerosene, and gas-oil constituents from the crude by using a vacuum during the latter part of the distillation and then taking the residue and diluting it with twenty times its volume of a mixture of 25 per cent ethylene dichloride and 75 per cent acetone. The mixture of acetone and ethylene dichloride was a good solvent for oil at 32° F.; however, wax, gums, and resins become practically insoluble in it at

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<sup>7</sup> Sullivan, F. W., McGill, W. J., and French, A., *Solubility of Paraffin Wax in Oil*: Ind. and Eng. Chem., vol. 19, No. 9, November, 1927, pp. 1042-1045.

this temperature and crystallize so that they can be separated readily from the solution by filtration. The natural crude wax or paraffin was separated from the oil in this manner and recrystallized twice to remove the small quantity of oil that it contained. The oil was added to the oil and ethylene dichloride; acetone solution and the acetone and ethylene dichloride were then distilled from the oil, using a large and efficient fractionation column to prevent any appreciable quantity of the oil from being carried over with the solvent. The dewaxed oil was separated into two portions; the gasoline, kerosene, and gas-oil fractions were then added to form two crudes, one having a specific gravity of 0.813 at 60° F. (42.6° A. P. I. gravity) and the other having a specific gravity of 0.830 at 60° F. (39° A. P. I. gravity), which was the gravity of the original crude. Both oils had cloud points below -10° F., which indicated that practically all the wax had been removed.

When the solubility of wax in different solvents under pressure and with gas and oil in solution was being determined the cloud-point method was impracticable, and it was necessary to develop a method by which a sample of the saturated solution could be analyzed for the wax content.

The apparatus finally constructed consisted of a steel container with a capacity of approximately 200 cubic centimeters and strong enough to withstand a working pressure of 2,000 pounds per square inch. The containers were fitted with tops provided with two valves. The construction was such that pressure could be applied to the contents of the container and samples could be removed. Tubes of 3 and 5 cubic centimeters capacity for taking and weighing samples were made from an aluminum alloy. These tubes were also capable of withstanding a pressure of 2,000 pounds per square inch. Drawings of the apparatus are shown in Figure 63. A constant-temperature water bath with manifold connections for supplying the pressure to the solubility containers was used, and a large centrifuge was equipped with automatic temperature control so that the solubility containers could be centrifuged to remove suspended wax from the solution before removing a sample. When nonvolatile solvents at atmospheric pressure were used the samples were drawn off into glass-stoppered U tubes. The aluminum weighing tubes were used for both volatile solvents and dissolved gas present in the solution.

The following procedure was adopted: The ratio of wax to solvent altered the apparent solubility of the wax somewhat, because it was composed of several fractions with different solubility.<sup>8</sup> It was therefore necessary to use the same relative proportion of wax to solvent in all determinations; the proportion used was exactly twice the amount held in solution, as determined by the cloud-point method, without pressure or gas and air in solution. This proportion gave figures higher than those for the cloud-point method, but the different determinations were uniform and comparable. The wax and solvent were placed in the solubility container in the constant-temperature bath, which was set at the desired temperature. When pressure was applied or gas was added to the oil-wax solutions the containers were connected to the manifold by flexible copper

<sup>8</sup>Weber, Paul, and Dunlap, H. T., Solubility of Paraffin Wax in Pure Hydrocarbons: Ind. and Eng. Chem., vol. 20, No. 4, 1928, p. 384.

tubing and the desired pressure supplied. The solution was held at the bath temperature for 48 hours and shaken at 30-minute intervals during the day to insure a saturated solution. At the end of 48

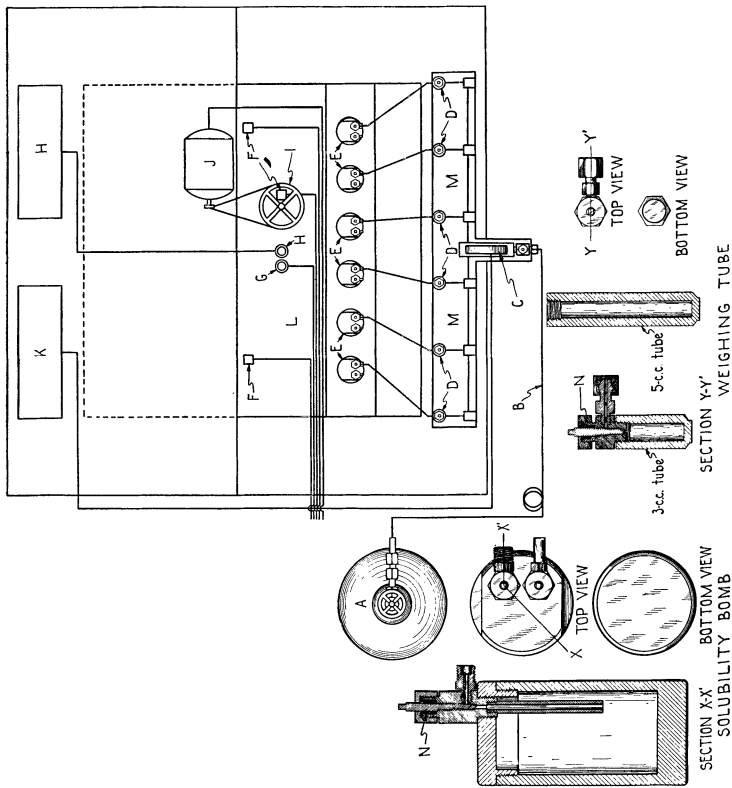
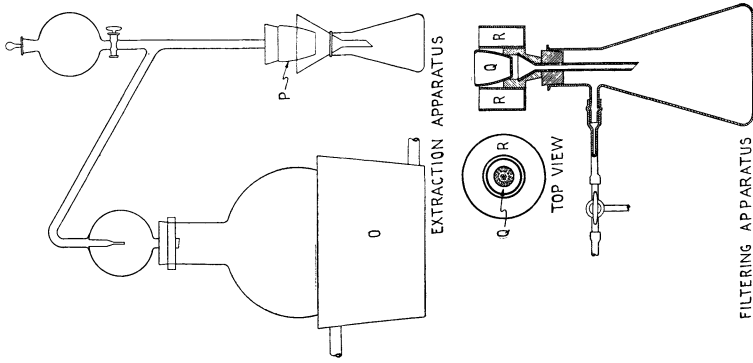


FIGURE 63.—Solubility apparatus

hours the containers were centrifuged for 45 minutes at 3,400 revolutions per minute, with the temperature in the centrifuge maintained at the same temperature as the bath. After centrifuging, the con-



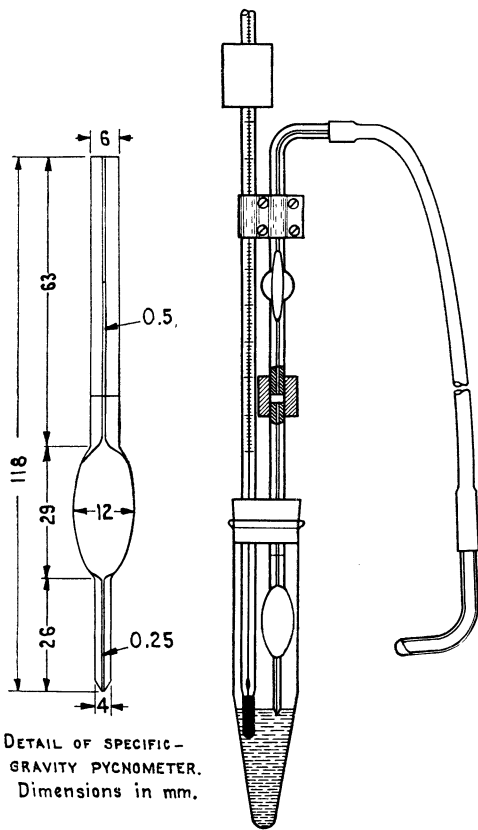
tainers were again placed in the bath, and the samples were withdrawn into weighed glass U tubes or into the aluminum weighing tubes and their weight determined. They were then transferred into flasks for determining the wax content. When volatile solvents were used the samples were transferred into weighed 100-cubic centimeter Erlenmeyer flasks, using petroleum ether as a wash, and the solvent was removed on a steam bath using a high vacuum to remove the last traces. The flasks were then weighed, and the amount of wax was determined. The specific gravity of the wax and solvent had been determined previously by the usual laboratory methods. The wax content of the sample could then be calculated as grams of wax per 100 grams of solvent, or cubic centimeters of wax per 100 cubic centimeters of solvent. This method proved very accurate and could be checked within  $\pm 1/5$  of 1 per cent.

When nonvolatile solvents, such as crude oil or other heavy-oil fractions were used as solvents for the wax a more complicated procedure was necessary. The weighed sample taken from the solubility containers was washed into a 2,000-cubic centimeter Erlenmeyer flask using 100 cubic centimeters of petroleum ether (boiling between 100° and 160° F.) for each gram of sample when crude oil was the solvent. One-tenth gram of fresh fuller's earth per gram of sample was then added and well shaken to remove asphaltic material and gums. The small quantity of earth used did not appreciably affect the wax content due to its selective tendency for gums, oil, and asphaltic material.<sup>9</sup> This procedure was necessary as the gums, resins, and asphaltic material present in the crude oil, in addition to those in the crude wax, formed a gummy precipitate that could not be filtered from the special solvent. To calculate the amount of crude wax dissolved in such cases it was necessary to consider the fact that 19 per cent (gums and resins) of the dissolved crude paraffin was removed by the fuller's earth. The earth was then removed by filtering and washing the oil with additional petroleum ether through a Buchner funnel. When refined oil fractions were used, fuller's earth was unnecessary. The light gasoline was then distilled off and a vacuum applied; the heavy oil and wax were left in the flask. To the oil and wax in the flask 50 cubic centimeters of a mixture of 25 per cent ethylene dichloride and 75 per cent acetone per gram of sample was added as a solvent, and this was refluxed on a steam bath for 15 minutes to insure complete solution of the wax and oil in the solvent. The solution was then cooled to 30° F., which caused the wax present to crystallize from it. It was then filtered through a Gooch crucible, using an asbestos mat as the filtering medium, and washed with 25 cubic centimeters of chilled solvent.

The wax was removed from the Gooch crucible by forcing hot ethylene dichloride vapors through the crucible and by adding 5 or 10 cubic centimeters of hot ethylene dichloride to the crucible; the solution of wax in ethylene dichloride was collected in a weighed 100-cubic centimeter Erlenmeyer flask, and the ethylene dichloride was removed by distillation, using a vacuum to remove the last traces. The weight of the remaining wax could then be determined.

<sup>9</sup> Diggs, S. H., and Bachler, C. C., Determination of Oil in Paraffin Wax: *Ind. and Eng. Chem.*, vol. 19, No. 1, January, 1927, p. 125.

The apparatus used for filtering the wax and removing it from the crucible is shown in Figure 63. With crude oil, where the gums, resins, and asphaltic material were removed by fuller's earth, it was necessary to divide the weight of wax obtained by 81 and multiply by 100 to correct the value for the 19 per cent of gums, resins, and asphaltic material removed by the fuller's earth. Although this procedure seems long and complicated, it produced satisfactory results after the technique of the various operations was thoroughly developed, and the results checked regularly within  $\pm 0.25$  of 1 per cent wax content.



DETAIL OF SPECIFIC-  
GRAVITY PYCNOMETER.  
Dimensions in mm.

ASSEMBLED APPARATUS  
FIGURE 64.—Density apparatus

#### DESCRIPTION OF APPARATUS AND METHOD USED FOR DETERMINING DENSITY OF WAXES

The apparatus consisted of a modified Drushel pycnometer, a thermometer graduated in tenths of a degree, a 50-cubic centimeter centrifuge tube, a glass stopcock, capillary glass tubing, and rubber tubing assembled as shown in Figure 64. The volume of the pycnometer was determined by calibration with water at  $84^{\circ}$  C., or  $183^{\circ}$  F. (the temperature at which the density of the waxes was to be determined) by the same method used in determining the density of the waxes.

The wax sample was placed in the glass centrifuge tube, melted in a steam bath, and all dissolved air removed by use of a vacuum pump. The thermometer and pycnometer were inserted as shown in Figure 64, care being taken not to allow the end of the pycnometer to touch the wax; the apparatus was then immersed in a glass water bath, maintained at a constant temperature of  $183^{\circ}$  F. ( $84^{\circ}$  C.), to a depth sufficient to cover the entire pycnometer. When the temperature of the wax was constant at  $183^{\circ}$  F. ( $84^{\circ}$  C.) the tip of the pycnometer was immersed in the wax; and it was filled by suction to the calibration point, the stopcock closed, and the tip of the pycnometer slowly removed from the wax. The entire apparatus was then withdrawn from the bath and allowed to cool; the slight excess of wax on the tip of the pycnometer was removed, and the pycnometer was weighed. As the volume of the pycnometer was

known the density could then be calculated. This method produced very accurate results after the technique of the different operations was mastered. Care had to be taken not to fill the pycnometer above the calibration and not to immerse the point of the pycnometer too deep in the wax. The main difficulty encountered was in the calibration of the pycnometer with water, and an average of a number of determinations was used. The four different pycnometers, after calibration with water, were checked with the same wax, and a variation or error in density of not more than 0.0002 was found.

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