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2,383,535

PROPANE FRACTIONATION OF HEAVY OILS

Filed Oct. 27, 1941

2 Sheets-Sheet 1

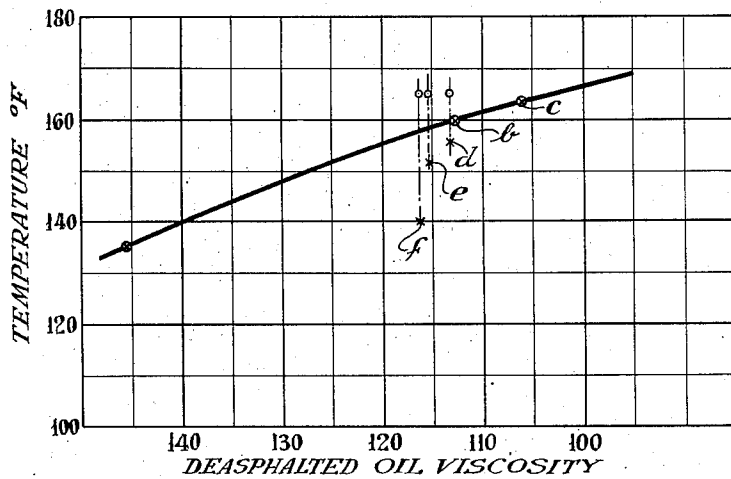


Fig. 1.

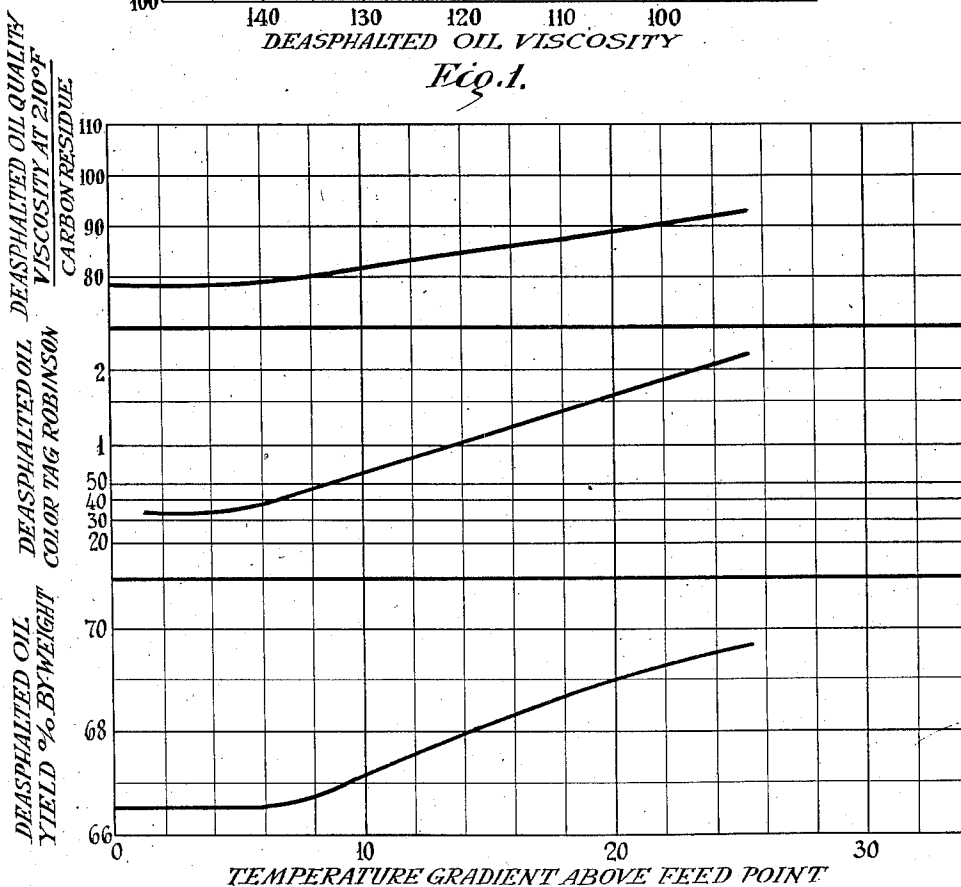


Fig. 2.

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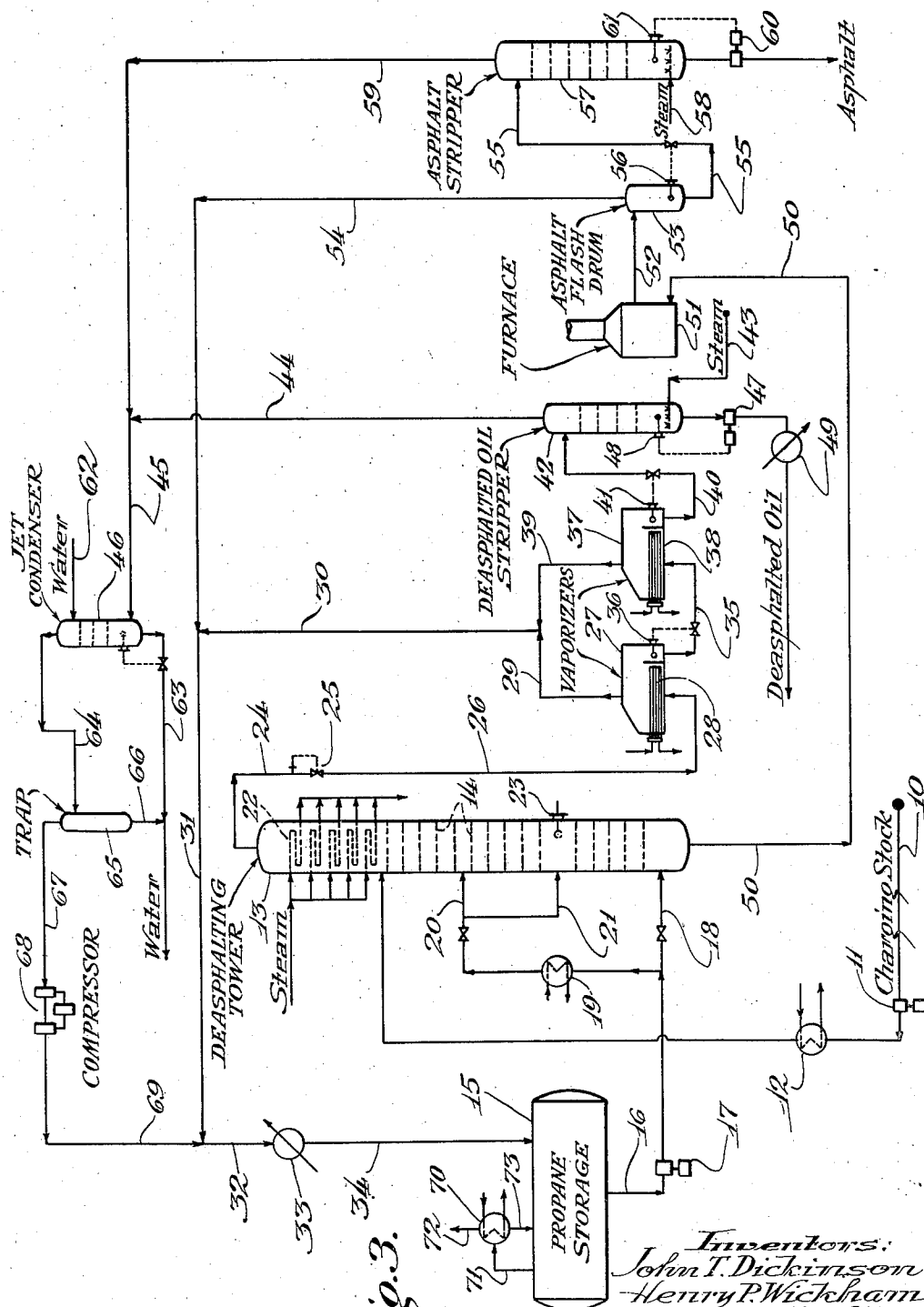
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UNITED STATES PATENT OFFICE

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PROPANE FRACTIONATION OF HEAVY OILS

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11 Claims. (Cl. 196—13)

Our invention relates to propane fractionation of heavy oils and it pertains more particularly to improved methods and means for simultaneously increasing the yield and quality of deasphalted oil obtainable from a reduced crude.

While the invention relates primarily to the deasphalting and fractionation of petroleum oils, particularly residual stocks, it should be understood that the invention is applicable to shale oils, oils produced by the hydrogenation of carbonaceous materials, oils produced by synthesis, from carbon monoxide and hydrogen (the so-called Fischer liquids such as Kogasin), etc. Many features of the invention are likewise applicable to animal oils such as lard oil, whale oil, fish oil, cod-liver oil, etc. and to vegetable oils such as soy bean oil, cottonseed oil, linseed oil, etc. Also, the invention is applicable to the fractionation of fatty acids derived from animal and vegetable oils and to the fractionation of resins and resin acids.

It has been known for many years that propane is a unique and outstanding agent for separating asphalt, resins, etc. from heavy lubricating oils, for separating oils from waxes and for separating oils, fats, waxes, resins, etc. into components of different physical and chemical properties. Generally speaking, propane is not an extractive solvent of the type exemplified by sulfur dioxide, phenol, furfural, dichlorethyl-ether, etc. but is a precipitant the solvent properties of which change very rapidly and very radically with changes in temperature, particularly within the range of from about 100 to 200° F. An object of our invention is to utilize the unique properties of propane in a fractionation system more effectively and efficiently than they have ever been used before. In other words, our object is to secure the maximum of benefits and advantages obtainable by the use of propane and equivalent precipitants in the fractionation of heavy petroleum oils or other substances fractionatable by propane.

A further object of our invention is to provide an improved system for fractionating lubricating oils or other substances in counter-current towers. Counter-current propane deasphalting in towers has been suggested by Bahlke (U. S. 2,086,487), Voorhees (U. S. 2,079,886), Anné (U. S. 2,213,798), etc., and countercurrent propane fractionation of oils in a tower has been described by Whiteley et al. (U. S. 2,110,845) but none of such processes has been commercially successful. An object of our invention is to provide improvements in such countercurrent propane fractionation processes and to place such

processes on a commercial basis. Our object is to correlate operating conditions, particularly operating temperatures at various points in the system with propane-to-oil ratios, oil charge rates, propane charge rates, position of interface between liquid phases, etc., so that maximum yields of valuable products can be obtained at a minimum cost without sacrificing product quality and with actual improvement in product quality.

A further object of our invention is to provide a method and means for obtaining increased yields of deasphalted oil in a propane deasphalting system without increasing deasphalted oil viscosity and without degrading its color or quality.

Heretofore, increases in yields have only been obtainable by increasing viscosity and sacrificing color, carbon residue, and other desirable properties. Our object is to provide a method and means for obtaining increased yields of a deasphalted oil of given viscosity and for simultaneously obtaining a deasphalted oil of better color, lower Conradson carbon, and more desirable properties than have heretofore been obtainable. In other words, our object is not only to obtain high yields but is also to obtain oils of higher quality than have ever before been obtained by any propane fractionation process heretofore known.

A further object of our invention is to provide improved methods and means for obtaining desired temperatures or temperature gradients in various parts of a countercurrent propane fractionation system. Heat exchange surfaces are undesirable in such systems because precipitated material tends to adhere to such surfaces and to prevent efficient heat transfer. Our object is to avoid the use of heating coils in all parts of a countercurrent propane fractionation tower which are below the point of oil inlet. Other objects of the invention will be apparent as the detailed description thereof proceeds.

In a preferred embodiment of our invention as applied to an East Texas residuum, we employ a countercurrent baffled tower operating under a pressure of about 450 to 500 pounds per square inch. One volume of charging stock is introduced at an upper point in said tower which is sufficiently spaced from the top of the tower to provide a reflux zone between the tower top and the oil inlet. In this reflux section of the tower we maintain a temperature gradient of at least 5 and preferably more than 10° F. In one specific example where we are deasphalting a 15% East Texas residuum to obtain about 70% of deasphalted oil, the charging stock inlet may be about

three-fourths of the way up in the tower so that the reflux zone is about one-fourth of the tower volume. In this specific example we use a tower temperature of about 150° F. at the point of oil inlet and a tower-top temperature of about 165° F. About 4 volumes of propane are introduced at a point near the base of the tower at such temperature that the tower bottom will be at a temperature of about 120° F. Two additional volumes of propane are introduced at a point about one-fourth of the way up in the tower at such temperature that at this point the tower temperature will be about 136° F. Another two volumes of propane are introduced at a point about half way up in the tower and at such temperature that the tower temperature at this point will be about 143° F. An important feature of this system is that at the base of the tower we employ the lowest temperature, the lowest propane concentration and the lowest upward flow velocity. As we proceed upward we employ higher propane concentrations, higher temperatures, and higher flow velocities so that in effect we not only maintain a temperature gradient in the tower but we also maintain a "propane concentration" gradient.

As applied to the propane-rich phase the propane concentration or propane-to-oil ratio is greatest at the lowest point in the tower at which propane is introduced. The exact propane concentrations at various points in the tower will depend, of course, upon total amount of propane employed, the points at which propane is introduced, the nature of the oil undergoing fractionation and the temperature gradient imposed. However, higher propane-to-oil ratios are required for effecting phase separation of relatively non-viscous oils than are required for effecting the separation of a highly viscous or asphalt phase. In countercurrent tower operation we have found that effective stripping of the asphalt or lower phase may be obtained by introducing only a part of the propane at the bottom of the tower, thus providing relatively low liquid flow rates in the tower at this point. We have found that more propane is required at intermediate and upper points in the tower to provide the necessary propane-to-oil ratio for the desired phase separation. By introducing propane at one or more intermediate points in the tower at a temperature which is substantially higher than the tower temperature at the point of introduction, we not only obtain the desired increase in tower temperature at the point of introduction but we simultaneously obtain the desired increase in propane-to-oil ratio.

Where the lower or propane-lean phase is not too viscous we prefer to maintain the interface level in the tower at a distance from the bottom of the tower which corresponds roughly to the percentage of charging stock withdrawn from the base of the tower. In other words, if 25% of the charging stock is withdrawn from the base of the tower we prefer an interface level about one-fourth of the way up in the tower. If half the product is taken overhead and half as bottoms, we prefer to introduce the charging stock at a middle point in the tower. If the bulk of the product is withdrawn as bottoms we prefer to maintain the interface at a point nearer the charging stock inlet, but in this case it may be necessary or desirable to increase the size of the reflux zone above the point of oil inlet.

Where extremely viscous products such as asphalt are removed from the bottom of the tower,

practical considerations may require that the interface level be maintained at or below the lowest point of propane inlet because asphaltic material can flow downwardly through the very fluid propane stream much more readily than introduced propane can flow upwardly through a viscous asphalt stream. When only a small amount of the propane is introduced at the base of the tower it may be possible and desirable to introduce a part of the propane below the interface level. In the preferred example hereinabove referred to the interface is about one-fourth of the way from the bottom of the tower so that asphalt will be in a continuous phase in the tower bottom for countercurrent washing with that portion of the propane that is introduced at the base of the tower. The lower temperatures and low flow rates in the lower part of the tower insure solution of desired oil components in the upflowing propane stream. The high temperature which prevails in the upper part of the tower tends to precipitate both asphaltic material and heavy oil fractions but the combined effect of temperature and propane concentration in the upper portion of the tower tends to selectively precipitate only the asphaltic components so that high yields of high quality oils of high viscosity are taken overhead and a substantially oil free asphalt of high specific gravity is removed from the base of the tower.

We have found that horizontal spaced baffles, preferably slat baffles, occupying within the approximate range of 30% to 70%, in other words about 50%, of the cross-sectional area effectively prevent short circuiting and provide for the necessary intimacy of contact. The heating coils employed in the upper reflux section may be spaced to serve the function of such baffles. However, in the upper part of the tower the difference in specific gravity between the propane-rich phase and the propane-lean phase is relatively small and we, therefore, prefer to allow at least 70% free space in the tower in order to prevent "flooding" and to provide for effective separation. In fact, the upper part of the tower may be of larger diameter than the lower part of the tower on account of the lower specific gravity differential which exists between the two phases in the upper part of the tower.

The invention will be more clearly understood from the following detailed description read in conjunction with the accompanying drawings wherein:

Figure 1 is a chart illustrating the effect of tower-top temperatures and temperature gradients on deasphalted oil viscosity;

Figure 2 is a chart illustrating the effects of temperature gradients in the reflux section of the tower on yield, color, and quality of deasphalted oil, and

Figure 3 is a schematic flow diagram of a commercial deasphalting plant embodying the features of our invention.

Before describing the commercial plant we shall point out the remarkable and unexpected results obtainable by using temperature gradient of at least about 5 to 10° F. in the upper part of a propane deasphalting tower and above the point of oil inlet. In a system wherein the oil feed stock is introduced into a propane deasphalting tower at a point spaced from the top thereof and wherein a heating coil is provided in the top of the tower, we have found that the viscosity of the deasphalted oil depends not only on the temperature at the top of the tower but also on

the temperature of the tower at the point of oil inlet. The effect of this latter temperature on deasphalted oil viscosity presupposes, of course, that the point of oil introduction is at such distance from the top of the tower and that such flow rates are employed as to provide adequate time and space for refluxing.

In Figure 1 we have plotted the results obtainable by tests made on a 15% East Texas residuum in a countercurrent tower employing about 6 volumes of propane per volume of oil treated. Points *a*, *b* and *c* represent deasphalted oil viscosities obtained where the tower temperature at the oil inlet is the same as the tower-top temperature so that a uniform temperature prevailed throughout the refluxing zone. Line *a*, *b*, *c* thus defines the viscosity of deasphalted oil for reflux zone temperatures from about 135 to 165° F. for this particular stock and propane-to-oil ratio.

Line *a*, *b*, *c* shows that with a uniform refluxing zone temperature of 165° F., the deasphalted oil will have a viscosity of about 104 seconds (all viscosities being in seconds Saybolt Universal at 210° F.). If the tower top is maintained at 165° F. but the tower temperature at the oil inlet point is lowered to about 156° F., as shown by points *d*, the deasphalted oil viscosity (113 seconds) will be about the same as with a uniform reflux zone at a temperature of about 159° F., i. e., the oil inlet temperature will have a greater effect than the tower-top temperature on the deasphalted oil viscosity. With a tower-top temperature of 165° F. and the tower temperature at the point of oil inlet of 151° F., as indicated by points *e*, the deasphalted oil viscosity (115.5 seconds) will be the same as with the uniform reflux zone temperature of about 158° F.; in this case the tower-top temperature and the temperature at the point of oil inlet has substantially an equal effect on the deasphalted oil viscosity. With a tower-top temperature of 165° F. and a tower temperature at the point of oil inlet at 140° F., as indicated by points *f*, the deasphalted oil viscosity (116 seconds) will be about the same as with a uniform reflux zone temperature of about 157° F.; in this case the tower-top temperature has a much greater effect on the deasphalted oil viscosity than the temperature at the point of oil inlet.

In going from a reflux zone temperature gradient of about 14° F. as represented by points *e*, to a temperature gradient of about 25° F., as represented by points *f*, there was only a slight change in deasphalted oil viscosity. In practical commercial operations the oil inlet temperature has no substantial effect on the viscosity of the deasphalted oil if the temperature differential in the refluxing zone exceeds about 30° F. Figure 1 clearly brings out this point and it also brings out the fact that a deasphalted oil of given viscosity may be obtained either by (1) maintaining a uniform temperature in the refluxing zone or, (2) by employing a temperature gradient above the point of oil inlet with a "tower-top temperature" which is higher and a "tower temperature at the point of oil inlet" which is lower than the uniform temperature which would otherwise be employed.

The next and perhaps most important consideration in connection with the use of a temperature gradient above the point of oil inlet is the effect of such temperature gradient on deasphalted oil yield, color and quality. We have discovered that by using a temperature gradient of at least 5 and preferably more than 10° F. above the point of oil inlet the deasphalted oil yield

gradually increases with an increase in temperature gradient. The magnitude of this increase in yield of a given viscosity oil is shown by the lower graph in Figure 2. No noticeable increase in yield is obtained with temperature gradients up to about 5 or 10° F. but for each 5 degrees of temperature differential above 10° F. we obtain an increase in yield of roughly about 1%. Thus the first advantage of the temperature gradient above the point of oil inlet is to increase the yield of deasphalted oil without increasing the deasphalted oil viscosity.

The middle graph in Figure 2 shows that the color of the deasphalted oil shows no noticeable improvement with temperature gradients up to 5° F. but with each 5 degrees of temperature differential thereafter there is a marked improvement in the color of the deasphalted oil. Thus the color with a 5 degree temperature differential is about 3½ D, with a 10 degree temperature differential the color of the deasphalted oil is about 5½ D, with a 20 degree temperature differential the color of the deasphalted oil is about 1½ (all colors on the Tag Robinson scale).

Oil quality may be indicated by the quotient of viscosity at 210° F. divided by carbon residue or Conradson carbon. From the top chart in Figure 2 it will be noted that with a 10 degree temperature differential above the point of oil inlet the deasphalted oil has a quality of about 84. With a 20 degree temperature differential above the point of oil inlet the deasphalted oil quality is increased to 90. With a 30 degree temperature differential above the point of oil inlet the deasphalted oil has a quality of about 96.

Thus from Figure 2 it will be seen that the yield, color and quality of deasphalted oil are all improved by employing a temperature differential or temperature gradient in the deasphalting tower above the point of oil inlet provided that this temperature gradient is at least 5° F. and is preferably more than 10° F. From Figure 1 it will be seen that an oil of desired viscosity may be obtained not only by regulating the tower top temperature but also by regulating the oil inlet temperature, provided that the temperature gradient does not exceed about 30° F. In other words, the phenomenon illustrated by Figure 1 provides the means for obtaining a deasphalted oil of given viscosity by the use of a temperature gradient above the point of oil inlet and the use of this temperature gradient accomplishes the remarkable and unexpected increase in yield, improvement in color and increase in quality illustrated in Figure 2.

While the data illustrated in Figures 1 and 2 were obtained on East Texas residuum with about 6 volumes of propane, this data is illustrative of results obtainable on any reduced crude or equivalent charging stocks throughout a wide range of propane-to-oil ratios and other operating conditions. For the deasphalting of reduced crudes we prefer to employ propane-to-oil ratios within the approximate range of 6:1 to 10:1, the first commercial installation being designed for a ratio of about 8:1 by volume. The 8:1 ratio has been found to be more selective than the 6:1 ratio and it has likewise been found to permit higher charge rates even though the upward vertical flow in the tower is somewhat increased.

A tower diameter of about 10 feet has been found to be satisfactory for a commercial plant designed to charge 2000 to 2200 barrels per day of reduced crude with an overall propane-to-oil ratio of 8:1. Thus in the upper part of the

tower the flow rate will be within the approximate range of 12 to 15 inches per minute. In the lower part of the tower the upward flow rate will be within the approximate range of 5 to 8 inches per minute. The downward flow rate in the bottom of the tower will be within the approximate range of 2 to 3 inches per minute. These flow rates may vary, of course, with different charging stocks, propane-to-oil ratios, etc. In any case the flow rate should be sufficiently low to permit the desired precipitation and removal of propane insoluble material.

A commercial plant embodying the features of our invention is illustrated in Figure 3. Charging stock from source 10 is pumped by pump 11 through heat exchanger 12 to a point about one-fourth of the distance from the top of fractionating or deasphalting tower 13. The specifications of this stock are approximately as follows:

API gravity	degrees..	15.6
Viscosity (S. S. U. at 210° F.).....	seconds..	485
Color		¹ Black
Carbon residue.....	per cent..	11.1
Flash point	° F..	550
Viscosity gravity constant.....		878

¹ Color is 52500 by the optical density method (in Ind. Eng. Chem., Vol 6, page 23).

In this particular plant the oil is charged at the rate of about 2100 barrels per day and the tower is about 10 feet in diameter by about 40 feet high. The tower is provided with slat baffles 14 up to about the 30-foot level, these baffles occupying about 50% of the total cross-sectional area. We have found that packing material of the type ordinarily employed in solvent extraction towers is not so desirable in our propane fractionation system and in some instances may be positively detrimental. In a tower which is not provided with baffles of some sort, however, there may be a tendency toward channelling and a lack of intimate contact that is required for efficient operation. Staggered baffles ranging from about 30% to about 70% of the cross-sectional area make possible the avoidance of possible channelling and accomplish the desired contact.

Propane is stored in storage tank 15 at a temperature of about 120° F. and a gauge pressure of about 250 pounds per square inch or more. Propane is withdrawn from the tank through line 16 by means of pump 17 at the rate of about 17,000 barrels per day, i. e., about 8 volumes of propane for each volume of stock charged to the system. About half of this propane, i. e., about 4 volumes, is introduced directly through line 18 at the base of tower 14. The other half of the propane is passed through heat exchanger 19 and heated to a temperature of about 160° F. About half of this hot propane (about 2 volumes) is introduced at the middle of tower 14 through line 20 and the other half of the hot propane (2 volumes) is introduced to the tower about 10 feet from the bottom thereof through line 21. The charging stock is introduced to the tower at a temperature of about 200° F. Heating coils 22 are provided in the top of the tower and sufficient heat is supplied to maintain a tower-top temperature of 165° F. Under the conditions above set forth the tower temperature at the point of oil inlet will be about 150° F., the tower temperature at its middle point will be about 143° F., at its 10-foot level the tower temperature will be about 136° F. and at its bottom the tower temperature will be about 120° F. In order to

prevent vaporization of propane in the tower it is maintained at a gauge pressure of about 450 to 500 pounds per square inch.

As above pointed out, an important feature of our invention is the use of a temperature gradient in the top or reflux section of tower 14. In order that this gradient may be substantially uniform we prefer to employ a plurality of spaced heating coils in this reflux section. These coils may occupy about 30% of the cross-sectional area and may serve as baffles. Our invention, however, is not limited to any specific arrangement of coils in this reflux section.

The position of the liquid interface level in the tower is preferably about one quarter of the way up although it may vary from a point near the bottom of the tower to a point near the middle thereof. In this particular example the tower is provided with a liquid level indicator 23 and the discharge of asphalt from the base of the tower may be automatically regulated by the position of this liquid level indicator. In the bottom 10 feet of the tower the precipitated asphalt forms a continuous phase and valuable oil components are efficiently recovered therefrom by the upward flow of propane at the relatively low temperature and relatively low flow rate. In the upper part of the tower the higher temperatures and increased propane-to-oil ratios effect substantially complete removal of the undesirable asphaltic constituents. The use of the temperature gradient above the point of oil inlet serves to remove even further amounts of undesirable color bodies, carbon forming constituents and other undesirable components without appreciably decreasing the viscosity or yield of the deasphalted oil. It has always been possible to improve color, carbon residue, etc. by producing oil of lower viscosity and by sacrificing yield but by the use of our temperature gradient above the point of oil inlet we maintain the viscosity, increase the yield and very greatly improve the color and carbon residue.

The deasphalted oil together with most of the propane is withdrawn from the top of the tower through line 24, passed through pressure reducing valve 25 and introduced through line 26 to vaporizer 27. The stream entering this vaporizer is at a temperature of about 140° F. and at a gauge pressure of about 275 pounds per square inch. This stream contains about 19,400 pounds per hour of deasphalted oil, 95,750 pounds per hour of liquid propane and about 26,200 pounds per hour of propane vapor.

The solution in vaporizer 27 is heated by low pressure steam in coils 28 to a temperature of about 180° F. which effects further vaporization of propane so that about 116,500 pounds per hour of propane vapors may be returned through lines 29, 30, 31 and 32 directly to condenser 33 and thence through line 34 to storage tank 15.

Oil and unvaporized propane from vaporizer 27 is passed through line 35 in amounts controlled by liquid level control means 36 into a second propane vaporizer 37 wherein it is heated by high pressure steam in coils 38 to a temperature of about 250° F. About 3,600 pounds per hour of propane is thus vaporized and returned to the propane storage tank via lines 39, 30, 31, 32 and 34.

The hot oil from the second vaporizer is passed through line 40 in amounts regulated by liquid level control means 41 to low pressure stripper 42 which may operate at a gauge pressure of

about 5 or 6 pounds per square inch. Steam is introduced at the base of this stripper through line 43. The 600 or 700 pounds per hour of stripping steam carries the remaining 1,800 or 1,900 pounds per hour of propane from the top of the stripper at a temperature of about 235° F. through lines 44 and 45 to jet condenser 46.

The deasphalted oil is withdrawn from the base of the stripper by pump 47 in amounts regulated by liquid level controls means 48 at a temperature of about 235° F. It is then cooled in exchanger 49 to about 150° F. and withdrawn to storage. The yield of the deasphalted oil is about 70%, i. e., about 1450 barrels per day and its properties are approximately as follows:

API gravity	degrees	23
Viscosity (S. S. U. at 210° F.)	seconds	125
Color (Tag Robinson)		1½
Carbon residue		1.4
Flash	°F.	540
Viscosity gravity constant		.84

The precipitated asphalt diluted with an approximately equal volume of propane is withdrawn from the base of tower 14 through line 50 and heated in pipe still furnace or heat exchanger 51 to a temperature of about 400 to 450° F. The hot asphalt propane solution is then introduced through line 52 and to vaporizer 53 at a gauge pressure of about 260 or 270 pounds per square inch. About 4800 pounds per hour of propane vapors are withdrawn through line 54 at a temperature of about 400° F. and returned via lines 31, 32 and 34 to storage tank 15.

Asphalt is withdrawn from the bottom of vaporizer 53 through line 55 in amounts regulated by liquid level control means 56 and the withdrawn asphalt is then introduced at the top of asphalt stripper 57 which operates at a gauge pressure of about 5 or 6 pounds per square inch. About 180 pounds per hour of high pressure steam is introduced at the base of the stripper through line 58 and this steam together with about 200 pounds per hour of propane vapors are withdrawn through lines 59 and 45 to jet condenser 46. The depropanized asphalt is withdrawn from the base of the stripper by pump 60 in amounts regulated by liquid level control means 61. About 680 barrels per day of asphalt is thus produced. This asphalt has a specific gravity of about 1.06 and a melting point of about 165° F.

Foaming and entrainment difficulties are avoided in vaporizer 53 and stripper 57 by maintaining a temperature in these zones of about 400° F.

Steam is condensed from propane vapors in jet condenser 46 by means of water introduced through line 62. This water together with condensed steam is withdrawn to the sewer through line 63. The propane leaves the top of the jet condenser at about 100° F. and it passes through line 64 to compressor trap 65 wherein additional water may be removed through line 66. The propane vapors are then passed through line 67 to compressor 68 wherein the vapors are compressed to a gauge pressure of about 265 to 275 pounds per square inch. The compressed vapors then pass through line 69, line 32, condenser 33 and line 34 to the propane storage tank.

The propane storage tank is provided with a purge gas cooler 70. Gases leaving the storage tank through line 71 pass through this cooler. Uncondensed gases may be withdrawn from the system through line 72 and condensed propane may be returned to the storage tank through

line 73. Make-up propane may be added to storage tank 15 as required.

Our invention is not limited to the specific charging stocks, products or operating conditions hereinabove described but is applicable to a wide variety of charging stocks and to a wide variety of operating conditions. From the detailed description of the specific example of our invention as applied to the deasphalting of a 15% East Texas residuum those skilled in the art may readily determine the operating conditions required for fractionating other oils and for the production of any desired products. In some instances, particularly in the fractionation of oils to obtain a low viscosity product, the tower-top temperatures may be 200° F. or even higher but for the deasphalting of reduced crudes we prefer to employ tower-top temperatures within the approximate range of about 160° F. to 180° F. and to employ a temperature gradient within the approximate range of 10 to 30° F. For deasphalting, the tower bottom temperatures are usually within the approximate range of about 120 to 140° F.

For the fractionation of lubricating oils into separate lubricating oil fractions of different viscosities the propane-to-oil ratios, tower temperatures and other operating conditions may differ substantially from the preferred conditions for deasphalting a reduced crude. Since these propane-to-oil ratios, temperatures, etc. are already known to those skilled in the art or may be readily ascertained from published data, calculations or simple preliminary experiments, it is unnecessary to point out such conditions in any further detail. In all cases, however, improved effectiveness and efficiency can be obtained at the proper propane-to-charging stock ratio by (1) introducing the propane not only at the bottom of the tower but also at one or more spaced points in the tower and at higher temperatures so that a proper propane-to-oil ratio may be maintained at various points and so that heating coils in the tower may be dispensed with, (2) by increasing the temperature uniformly or at spaced points from the bottom to the top of the countercurrent contacting zone in the tower, (3) by employing an increasing temperature gradient at the top of the tower above the point of oil inlet (and in the case of reduced crude by limiting this temperature gradient to within the approximate range of about 10 to 30° F.), and (4) by properly positioning the liquid interface in the tower.

The position of the interface may depend somewhat upon the nature of the charging stock. For viscous asphalts and the like it may be desirable to operate with an interface level at the bottom of the tower. If the depropanized precipitated material is of greater volume than the depropanized propane soluble material and is not too viscous, this liquid interface should be nearer the top than the bottom of the countercurrent contacting zone. The position of this interface for non-viscous oil should roughly correspond to the split which is to be made in the charging stock. When the charging stock contains about 25% of non-viscous insoluble material the interface should be about one-fourth of the way up in the tower. If the charging stock contains 50% of non-viscous insoluble material, the interface should be maintained at about the middle of the tower. If the charging stock contains 75% of non-viscous insoluble material, the interface should be about three-fourths of the way up in

the tower. Improved results may be obtained in any case by the use of the temperature gradient in the refluxing zone above the point of oil inlet, and this reflux zone is of particular importance in the case of operations where the interface level is near the charging stock inlet.

The word "propane" as used in the above specification and in the following claims should not be limited to pure propane but is intended to cover commercial propane and equivalent normally gaseous precipitating agents. Lower temperatures and higher pressures will be required if ethane is present in appreciable amounts. Higher temperatures and low pressures may be employed if butane is present in appreciable amounts. Olefin hydrocarbons are less desirable than paraffin hydrocarbons in our fractionation process and we, therefore, prefer to employ a propane which is substantially free from olefins.

It is not essential that our countercurrent process be carried out in a single tower since the invention may also be practiced in a plurality of towers or fractionation vessels. Other modifications, alternatives, operating conditions, etc. will be apparent to those skilled in the art from the above detailed description.

We claim:

1. The method of increasing the efficiency of a countercurrent propane fractionation system wherein charging stock is introduced into one end of a fractionation zone at a relatively high temperature and propane is introduced at the other end of said zone at a relatively low temperature, which method comprises introducing additional amounts of propane at at least one intermediate point in said zone, maintaining an intermediate point in said zone at a substantially higher temperature than the temperature at the point of propane inlet and at a substantially lower temperature than the temperature at the point of charging stock inlet, and employing a rate of upward flow in the upper part of the tower approximately twice as high as the rate of upward flow in the lower part of the tower.

2. The method of increasing the efficiency of a countercurrent propane fractionation system wherein propane is introduced at the base of a tower for countercurrent contact with a charging stock introduced at the top of the tower and wherein the top of the tower is maintained at a higher temperature than the bottom of the tower, which method comprises introducing propane at an intermediate point in the tower at a temperature higher than the tower temperature at the point of introduction whereby the temperature, the propane-to-charging stock ratio and the flow velocities are higher in the upper part of the tower than in the lower part of the tower, and introducing an amount of propane at the intermediate point sufficient to give approximately twice as high an upward flow rate in the upper part of the tower as is maintained in the lower part of the tower.

3. The method of improving the effectiveness and efficiency of a countercurrent propane fractionation system wherein a charging stock is introduced at an upper point in the tower, which method comprises introducing propane into the tower at a plurality of vertically spaced points below the point of charging stock introduction and in such amounts that the upward flow rate in the lower part of the tower would be in the general vicinity of 5 to 8 inches per minute while

the upward flow rate in the top of the tower will be in the general vicinity of about 12 to 15 inches per minute.

4. The method of fractionating a reduced crude for the production of maximum yields of high quality lubricating oil, which method comprises introducing one volume of reduced crude together with an amount of propane ranging from about 6 volumes to about 10 volumes into a contacting zone, withdrawing propane precipitated material from the base of said contacting zone, withdrawing a propane lubricating oil solution from the top of said contacting zone and introducing it into a reflux zone having an increasing temperature gradient of about 10° to 30° F., returning material precipitated in the reflux zone to the contacting zone, withdrawing a propane-lubricating oil solution from the top of said reflux zone, and employing an upward flow rate at the top of the contacting zone at least about twice as large as the upward flow rate at the base of said zone.

5. The method of fractionating a petroleum residual stock which method comprises introducing said stock into a propane fractionation tower at a point nearer the top than the bottom of the tower but spaced from the tower top, introducing propane at the bottom of said tower, maintaining a temperature at the bottom of the tower within the approximate range of 120 to 140° F., maintaining the temperature at the top of the tower within the approximate range of 160 to 180° F., maintaining the temperature of the tower at the point of residual stock inlet at a temperature within the approximate range of 10 to 25 degrees lower than the temperature of the tower top, and maintaining a rate of upward flow in the upper part of the tower about twice as high as the rate of upward flow in the lower part of the tower.

6. The method of claim 5 which includes the further step of maintaining a liquid interface in the lower part of said tower but above the point of propane inlet.

7. The method of claim 5 which includes the further step of introducing additional amounts of propane into the tower at at least one intermediate point therein.

8. The method of operating a countercurrent propane deasphalting system which comprises introducing a charging stock at an upper point in the tower which is spaced from the tower top, introducing propane at the bottom and at at least one intermediate point in the tower, maintaining a liquid interface in the lower part of the tower, maintaining an upward flow rate in the upper part of the tower which is about twice as great as the upward flow rate in the lower part of the tower, maintaining a temperature in the bottom of the tower of about 120° F., maintaining a temperature in the tower at the point of oil inlet at about 150° F. and maintaining a tower temperature between the bottom and the point of oil inlet of about 140° F.

9. The method of claim 8 which includes the further step of maintaining a tower-top temperature of about 165° F. whereby a reflux zone is established between the tower top and the point of charging stock inlet and whereby the temperature gradient in the reflux zone is about 15° F.

10. The method of deasphalting a reduced crude charging stock in a countercurrent tower which method comprises introducing one volume of oil at a point about one-fourth of the way down

in said tower, introducing about 4 volumes of propane at the bottom of said tower, introducing about 2 volumes of propane about one-fourth of the way up in the tower, introducing about 2 volumes of propane at a point about half way up in the tower, maintaining a tower-bottom temperature of about 120° F., a tower temperature about one-fourth way up of about 136° F., a temperature about half way up of about 143° F. and a temperature at the point of oil inlet at about 150° F., maintaining a tower-top temperature of about 165° F., maintaining a liquid interface in the lower part of the tower, maintaining an upward flow rate in the top of the tower of about 12 to 15 inches per minute, maintaining an upward flow rate in the lower part of the tower at about 5 to 8 inches per minute, withdrawing propane precipitated material from the base of said tower and removing propane from the withdrawn precipitated material, removing a propane oil solution from the top of said tower and removing propane from said solution.

11. The method of deasphalting a petroleum residual stock which method comprises intro-

5 ducing said stock at a point spaced from the upper end of a deasphalting tower at such temperature as to maintain a tower temperature at the point of introduction of about 150° F., introducing propane at the base of said tower at such temperature as to provide a tower bottom temperature of about 120° F., introducing propane at an intermediate point in the tower at such temperature as to maintain a tower middle temperature of about 140° F. and in such amounts that the rate of upward flow in the upper part of the tower will be about twice as high as the rate of upward flow in the lower part of the tower, supplying heat at the top of the tower in order to maintain a tower-top temperature of about 165° F., withdrawing precipitated material from the bottom of the tower at such a rate as to maintain a substantially constant liquid level in the lower part of said tower and withdrawing a propane oil solution from the top of said tower.

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