Jan. 23, 1945. J. T. DICKINSON ET AL 2,367,671

PROPANE FRACTIONATION OF HEAVY OILS

Filed Nov. 13, 1941 4 Sheets-Sheet 1

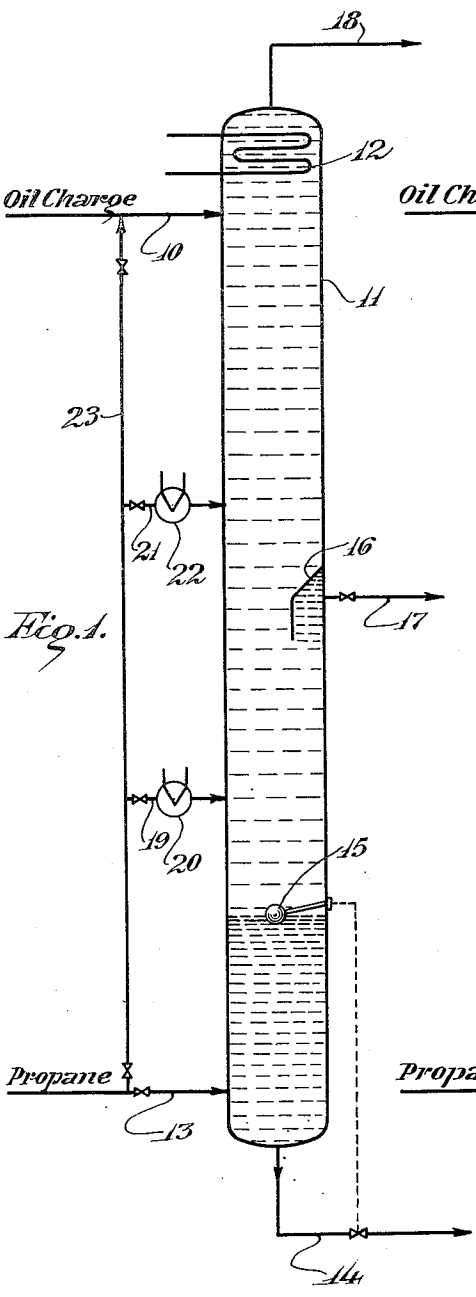


Fig. 1.

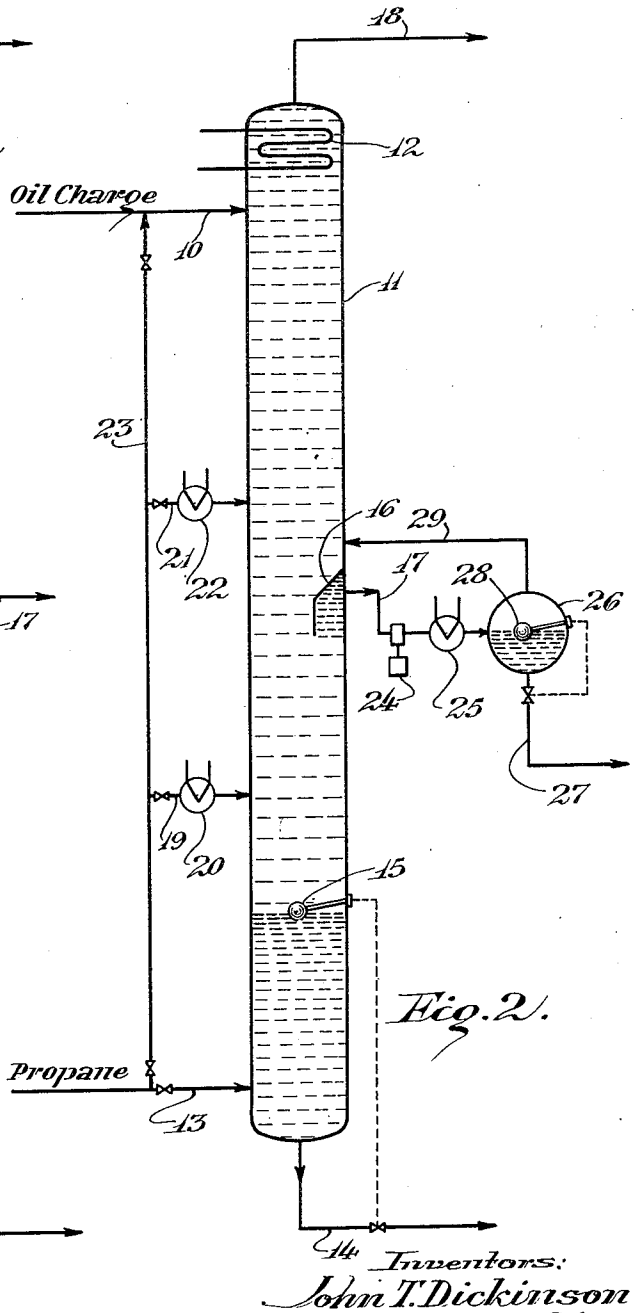


Fig. 2.

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PROPANE FRACTIONATION OF HEAVY OILS

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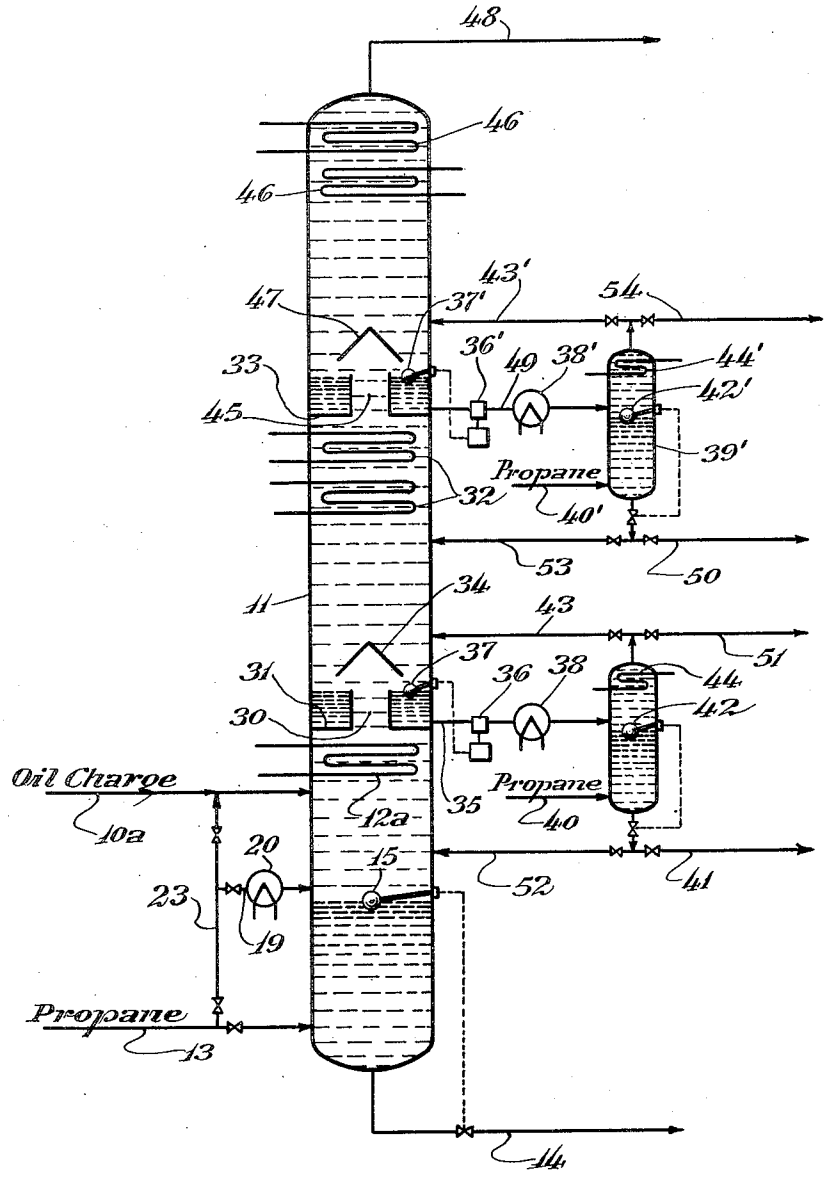


Fig. 3.

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PROPANE FRACTIONATION OF HEAVY OILS

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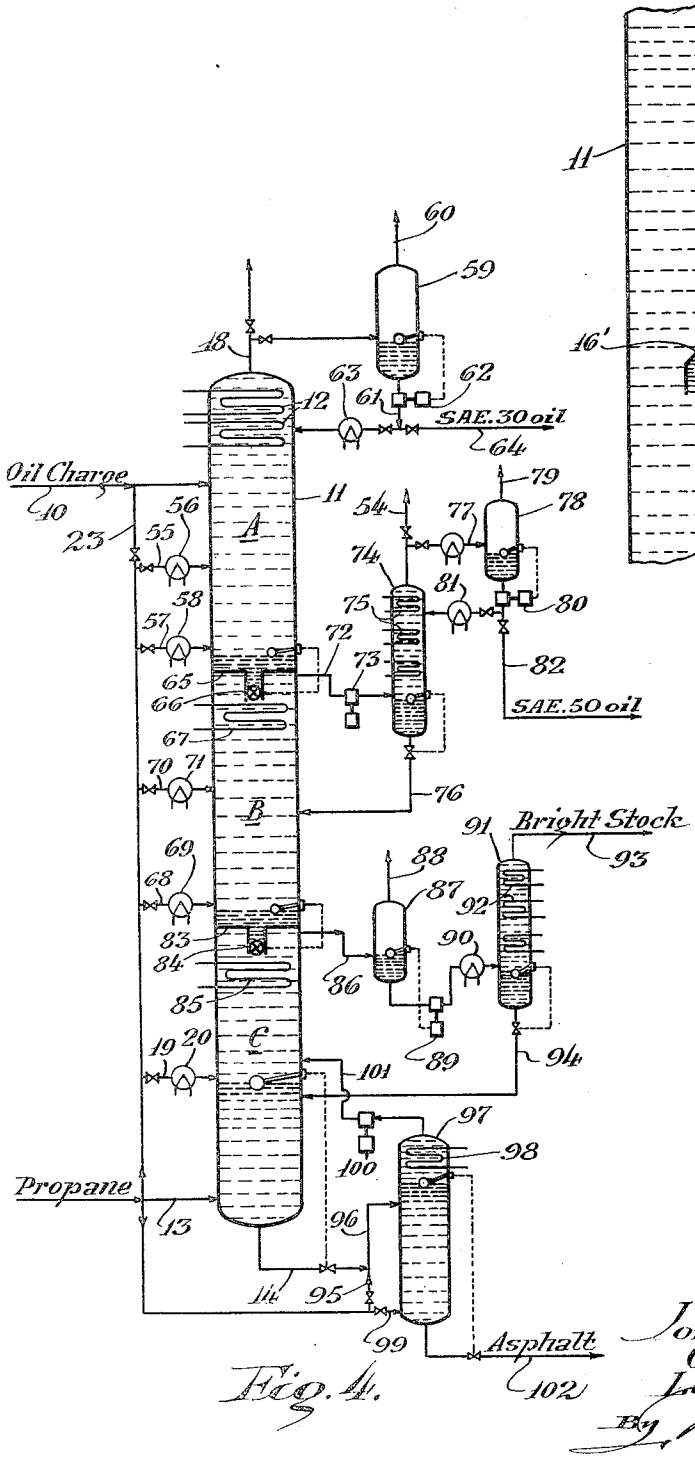


Fig. 4.

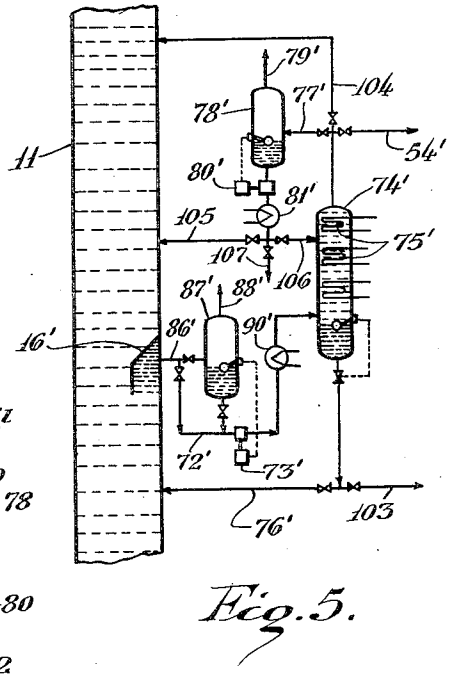


Fig. 5.

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PROPANE FRACTIONATION OF HEAVY OILS

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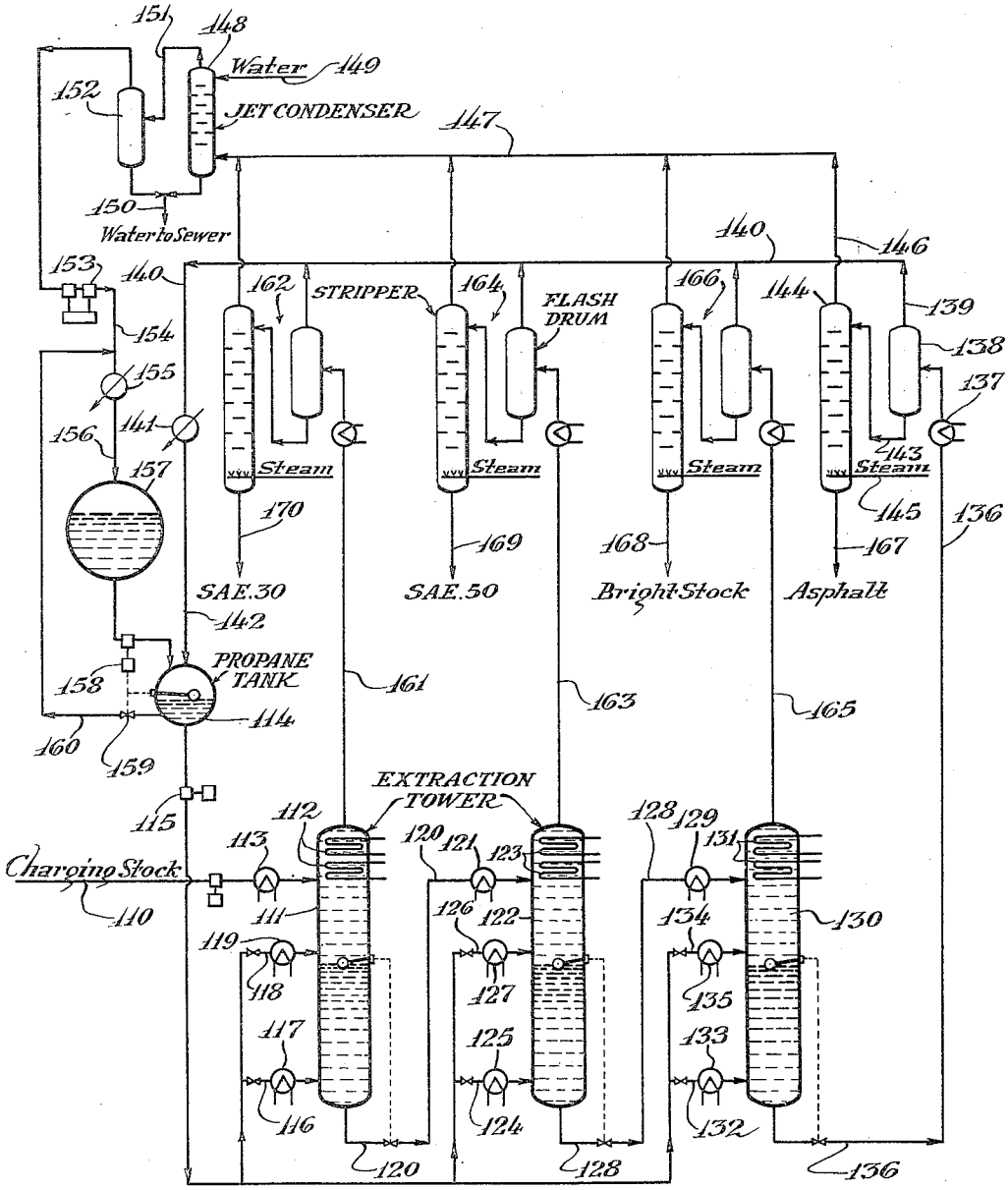


Fig. 6.

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UNITED STATES PATENT OFFICE

2,367,671

PROPANE FRACTIONATION OF HEAVY OILS

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Application November 13, 1941, Serial No. 418,944

14 Claims. (Cl. 196—13)

This invention relates to the propane fractionation of heavy oils and it pertains more particularly to improvements in countercurrent propane fractionation systems.

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, fish and vegetable oils and to the fractionation of resins and resin acids. All of the materials hereinabove named and equivalents thereof have the common property of containing oil, oily, or oil-like components which may be separated from other components by propane and hence may be termed oleiferous materials. The term "oleiferous" as used in the following claims is hereby defined as including all of such materials.

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, dichlorethylether, 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 separating a charging stock into a plurality of fractions of different physical or chemical properties in a single countercurrent system. A further object is to provide improved methods and means for withdrawing intermediate products of closely defined characteristics from a counter-current propane fractionation system at one or more intermediate points thereof. A further object is to provide improved methods and means for separating undesirable components from intermediate streams and for returning said undesirable components to the countercurrent system without adversely affecting the operation thereof.

A further object of our invention is to provide improved methods and means for regulating propane-to-oil ratios, temperatures, flow velocities, etc., at various points in a countercurrent propane fractionation system whereby the sharpest possible fractionation is not only obtained between two components but is also obtained between these two components and one or more additional intermediate components. A further object is to provide improved methods and means for fractionating a long residumm into SAE 30 oil, SAE 50 oil, bright stock and asphalt or into other grades and qualities of lubricating oil fractions. Other objects will be apparent as the detailed description of our process proceeds.

In practicing our invention we may employ a single countercurrent contacting tower with side stream draw-offs or we may employ a series of countercurrent contacting towers. In this tower, or in each tower if a plurality is used, we maintain a relatively low tower bottom temperature, an increasing temperature gradient in the tower and a relatively high tower-top temperature. We have found that the temperature gradient alone, however, is not as effective or as efficient as the system which also has a propane-to-oil gradient, i. e., which provides for the optimum propane-to-oil ratio at all points in the tower and in all side stream fractionation systems when such systems are employed.

Our invention utilizes the unique properties of propane particularly within the approximate temperature range of 100 to 200° F. The fractionating properties of propane change very markedly in this temperature range and the fractionating properties also change very markedly with propane-to-oil ratios. In all cases, of course, sufficient pressure must be employed to maintain the entire system in liquid phase but in each fractionation stage the pressure must be sufficiently low to permit the formation of two liquid phases.

Low propane-to-oil ratios, low temperatures and high pressures generally lead to the production of only a single liquid phase and in order

to obtain fractionation it is generally necessary to increase the propane-to-oil ratio or to increase the temperature. Increases in temperature always tends toward further liquid phase separation. However, when the optimum propane-to-oil ratio has been reached a further increase in propane-to-oil ratio tends toward re-establishment of a single liquid phase rather than toward further phase separation. Invariably one of the liquid phases in a fractionation system will be rich in propane while another is lean in propane. Further fractionation of the propane-rich phase may be obtained by further heating of this phase or by the removal of propane. Further fractionation of a propane-lean phase may be obtained by the further heating of this phase or by the addition of propane thereto.

Generally speaking, the minimum propane-to-oil ratios for fractionating relatively low viscosity stocks must be higher than for fractionating extremely viscous or high molecular weight stocks. At any given temperature there is an optimum propane-to-oil ratio for maximum phase separation and this propane-to-oil ratio is usually fairly high, i. e., upwards of 6 to 1. Increasing the propane-to-oil ratio up to this maximum tends to effect further phase separation at any given temperature. At any given propane-to-oil ratio an increase in temperature invariably effects further phase separation, i. e., tends to precipitate lighter and lighter components of the charge. The effect of temperature increase is not uniform throughout the range of 100 to 200° F. but is more nearly uniform at high propane-to-oil ratios than at low propane-to-oil ratios. With extremely heavy residuums or asphalt stocks there is a gradual increase in propane insoluble material produced at propane-to-oil ratios of about 4:1 as the temperature increases from about 100° to about 200° F. With 4:1 propane-to-oil ratio on ordinary reduced crudes there is a relatively small increase in propane insoluble material produced as the temperature is raised from 100° to some intermediate temperature usually within the approximate range of 140 to 160° F. but at this intermediate temperature there is a sharp increase in propane insoluble material; as this intermediate temperature zone is passed, further increases in temperature produce a more gradual increase in propane soluble material. At high propane-to-oil ratios, i. e., within the approximate range of 6:1 to 10:1, the intermediate temperature zone is not so different in its effect. If close fractionation is desired at the intermediate temperature zone it is, therefore, usually desirable to operate with a propane-to-oil ratio of at least 6:1.

An important feature of our invention is the provision of a countercurrent propane fractionation system wherein one or more intermediate fractions are produced and withdrawn as a side stream. This side stream may be treated at such propane-to-oil ratio and temperature that undesirable components thereof may be separated therefrom and returned to the main countercurrent system. The invention will be more clearly understood from the following detailed description of our preferred embodiments thereof.

In the accompanying drawings which form a part of this specification:

Figure 1 is a schematic diagram illustrating the simplest form of our side stream draw-off;

Figure 2 is a schematic diagram of a similar system illustrating the return to the countercurrent system of propane and oil fractions separated from the side stream;

Figure 3 is a schematic diagram illustrating a countercurrent fractionation system with side streams withdrawn above the point of oil inlet;

Figure 4 is a schematic diagram of a countercurrent tower wherein the charge is introduced near the top, propane is introduced at the bottom and intermediate points and side streams are withdrawn between the points of charge inlet and propane inlet respectively;

Figure 5 is a schematic diagram illustrating side stream draw-offs and rejected components return lines where the side stream constitutes a propane-rich phase; and

Figure 6 is a schematic flow sheet of a commercial system embodying the principles illustrated in Figure 5 but employing a plurality of towers instead of a single tower, the side stream fractionation devices being incorporated in the upper part of each tower.

Referring to the specific example illustrated in Figure 1, a Mid-Continent residuum is charged through line **10** at a point near the top of countercurrent tower **11**. A heating coil **12** at the top of this tower maintains a tower-top temperature of about 197° F. About 6 volumes of propane are introduced at the bottom of the tower through line **13** at such temperature as to maintain a tower bottom of about 144° F. A liquid interface is maintained at a point near the bottom of the tower, the asphalt being withdrawn from the base of the tower through line **14** at such a rate as to maintain a constant interface level indicated by float means **15** or by a gauge glass or by other indicating means. Preferably the liquid level (i. e., liquid interface level) indicator automatically regulates the draw-off through line **14**.

Under baffle **16** at about the middle of the tower a side stream is withdrawn through line **17** and depropanized. The overhead fraction is withdrawn through line **18** and depropanized. The yield and properties of the charging stock and the three products thus produced are substantially as follows:

	Charge	Deasphalted oil	Side stream	Bottom
Yield		66.9	12.6	20.5
Grav., °A. P. I.	23.6	27.0	22.5	13.7
Vis., SSU/210° F.	100.2	60.7	168	
Color, T. R.	2-DD	1½	4¼D	
Car. res., percent	3.4	0.55		
Flash, COC		415	540	
Fire, COC		470	625	

It will be noted that the viscosity of the overhead oil was about 60 seconds while the viscosity of the side stream was almost 170 seconds Saybolt at 210° F. It will also be noted that the color of the overhead fraction was 1½ Tag Robinson while the color of the side stream was 4¼ D Tag Robinson. The flash of the side stream shows that there was very little contamination between deasphalted oil and the side cut.

The above operation is improved by adding propane at one or more intermediate points in the tower. Thus instead of adding 6 volumes of propane at the base of the tower we may add 4 volumes at this point, 2 volumes through line **19** and heat exchanger **20**, 2 more volumes through line **21** and heat exchanger **22**, etc. At the beginning of the operation it may be desired to add propane directly to the charging stock through line **23**.

A still further improvement in this operation may be obtained by a further fractionation of the withdrawn side stream as illustrated in Figure 2.

This stream may be forced by pump 24 through heat exchanger 25 to raise its temperature at least about 10 or 20° F. The heated mixture is then introduced into settling chamber 26 to effect the separation of the two phases which result from the heating step. The heavier phase may be withdrawn through line 27 in amounts regulated by liquid level control indicator 28. The lighter fraction, which is rich in propane, may be returned through line 29 to tower 11 above baffle 16. By this simple expedient the viscosity and flash point of the side stream are both increased and the bulk of the propane is returned to the tower at a high temperature, thus promoting more favorable fractionating conditions in the upper part of the tower.

In Figure 3 we have illustrated another modification of the invention wherein the charging stock is introduced through line 10a below the point or points of side stream draw-off. Above the point of oil inlet we may provide heating coils 12a to knock back any asphaltic material and thus prevent such material from flowing upward through opening 30 of liquid trap 31. Thus in the lower part of the tower we may introduce about 4 volumes of propane through line 13 at such temperature as to maintain the tower bottom at 120° F. 2 additional volumes of propane may be added through line 19 and heater 20 to maintain a tower temperature of about 135° at this point. Heating coils 12a may maintain a tower temperature just below the first liquid trap at about 160° F.

The solution which passes upward through the opening 30 in trap 31 is further heated by heating coils 32 so that by the time it reaches liquid trap 33 it is at a temperature of about 180° F. Between the two liquid traps there is substantial phase separation due to the increase in temperature and a propane-lean phase separates out and drops downwardly. This phase is deflected by baffle 34 into liquid trap 31 from which it is withdrawn through line 35 by pump 36 controlled by liquid level indicator 37. The withdrawn phase may be heated by heater 38 to a temperature of at least about 170° F. to effect further phase separation and the heated liquids are then introduced into side stream fractionator 39 at the base of which additional propane is introduced through line 40. Liquids are withdrawn from the base of fractionator 39 through line 41 at rates controlled by liquid level indicator 42. The propane which is introduced through line 40 tends to wash out the lighter components of the side stream and the added propane together with the higher temperature results in the formation of an upper propane-rich phase which is withdrawn through line 43 and introduced into tower 11 above liquid trap 31. A heating coil 44 may be employed in the top of fractionator 39 for knocking back any heavy oil components and may take the place of heater 38 or be employed in addition thereto.

The hot oil solution which passes upwardly through the opening 45 in liquid trap 33 is still further heated by heating coils 46 which maintain the tower-top temperature of about 200° F. At this high temperature further phase separation occurs and the heavier components drop downwardly as a propane-lean phase into liquid trap 33, baffle 47 serving to deflect this propane-lean phase into the trap. The final propane-rich phase is withdrawn from the top of the tower through line 48 to suitable depropanizers. The propane-lean fraction from trap 33 is withdrawn as a side stream 49 and further fractionated in the same manner as side stream 35 is fractionated, the final propane-lean fraction being withdrawn through line 50 to suitable depropanizers. Thus in the system illustrated by Figure 3, a light oil fraction corresponding to SAE 20 or SAE 30 lubricating oil may be obtained from the stream from line 48, and SAE 40 or SAE 50 stock may be obtained from the stream from line 50, a high quality bright stock may be obtained from the stream from line 41 and a high melting point asphalt substantially free from oil is obtained from the stream from line 14.

In some cases it may be important to remove the heavier components instead of the lighter components from the withdrawn side streams thus instead of returning the overhead from fractionator 39 to tower 11 this overhead may be withdrawn through line 51 to a depropanizer and instead of withdrawing the propane-lean phase from the bottom of fractionator 39 to a depropanizer this material may be returned through line 52 to the fractionating tower at a point below the side stream draw-off or even below the liquid interface level. Similarly, we may return the propane-lean fraction from the base of the upper fractionator 39' through line 53 to a point between the liquid trap-out plates 31 and 33 and we may withdraw the overhead from fractionator 39' to a depropanizer through line 54.

In Figure 4 we have illustrated another modification of our invention wherein the charging stock is introduced at a point near the top of the tower (as in Figure 1) and wherein propane is introduced at the bottom and a plurality of spaced points along the side of the tower. In this modification the heavy oil fractions gradually move downward in the tower as distinguished from the modification in Figure 3 wherein the lighter oil fractions move upwardly in the tower. In Figure 3 the propane solution passes upwardly from a lower to an upper section of the tower while in Figure 4 a propane-lean fraction passes downwardly from an upper to a lower section of the tower. This difference in operation requires different side stream fractionation methods. One volume of a 22.1° A. P. I. gravity residuum is introduced through line 10 at a point near the top of tower 11 to obtain a tower-top temperature of about 195° F. Preferably the tower temperature at the point of oil inlet is about 190° and the tower-top is maintained at about 200° by means of heating coils 12. About 2 volumes of propane may be added to the charging stock through line 23 or introduced at an intermediate point in the upper tower section through line 55 and heater 56 to maintain a tower temperature at this point of about 185° F. About 4 volumes of propane are introduced to line 57 and heater 58 to maintain a tower temperature at the bottom of this bottom zone A of about 175° F.

The high tower-top temperature knocks back all of the asphaltic materials and high viscosity materials so that only low viscosity propane soluble materials are taken overhead through line 18. Part or all of the overhead stream may be introduced into a depropanizing chamber 59 from which a substantial amount of propane may be withdrawn through line 60. The partially depropanized products may be withdrawn through line 61 by means of pump 62 at such a rate as to maintain a constant liquid level in chamber 59. This depropanized material may be returned through heater 63 to the top of tower 11 or may be withdrawn through line 64 for further de-

propanizing and stripping. By returning a portion of the depropanized overhead stream to the top of the tower we effectively reduce the propane-to-oil ratio in the top of the tower. When the charging stock contains a large proportion of low viscosity components it may be undesirable to so reduce the propane-to-oil ratio at the top of the tower. On the other hand, if only about 10% of the charging stock is of sufficiently low viscosity to be taken overhead then the propane concentration in the top of the tower will be too high for maximum phase separation at this point and the reduction in the effective propane-to-oil ratio in the tower top is highly advantageous.

The asphalt and viscous oil components from the propane-lean phase in the upper tower section pass downwardly through the upper section of tower 11 to form a pool at the base of this upper section and above partition plate 65. This propane-lean phase flows downwardly through the partition plate at rates controlled by valve 66 which is so regulated as to maintain the desired interface level in the upper section of the tower. A heating coil 67 maintains a tower temperature beneath plate 65 at about 175 to 180° F. About 3½ to 4 volumes of propane based on original stock (or about 7 to 8 volumes of propane based on the amount of oil passing through valve 66) may be introduced through line 68 and heater 69 at such temperature as to maintain a tower temperature at the bottom of the intermediate zone of about 150° F. Instead of introducing all of the propane into this zone at the bottom thereof we may introduce about one-third to one-half of the propane through line 70 and heater 71 at an intermediate point of the intermediate zone so that the tower temperature at this point will be within the approximate range of 160° to 165° F.

The high temperature immediately below plate 65 knocks back all of the asphalt and very viscous oil components so that a propane-rich fraction of non-asphaltic oil substantially free from bright stock may be withdrawn through line 72 and introduced by pump 73 into side stream fractionator 74 which is provided with heating coils 75. These heating coils provide a temperature gradient of at least 10 or 20° F. in the side stream fractionator and this temperature gradient rejects any asphalt or bright stock components which might be present in the side stream. The asphalt and bright stock components are returned through line 76 to an intermediate point of the intermediate section in such amounts as to maintain a constant interface level in the side stream fractionator.

Where a very small amount of oil is present in this side stream increased phase separation may be effected by decreasing the propane concentration in the upper part of side stream fractionator 74. Thus the overhead from this fractionator may either be withdrawn directly through line 54 for oil recovery or it may be passed through line 77 to depropanizer 78 from which a substantial portion of the propane is removed through line 79. Depropanized oil is removed from the base of this depropanizer by means of pump 80 to maintain a constant liquid level in chamber 78. Such depropanized material may be passed through heater 81 back to the top of side stream fractionator 74 or may be withdrawn through line 82 for oil recovery.

The propane-lean phase containing asphalt and bright stock accumulates as a pool above partition plate 83 and it flows downwardly through valve 84 at such a rate as to maintain a constant interface level above plate 83. A heating coil 85 below plate 83 maintains a tower temperature at about 165° F. About 2½ to 3 volumes of propane (based on original charge) may be introduced at the base of the tower through line 13 at such temperature as to maintain a tower bottom of about 130° F. This amount of propane will make the propane-to-oil ratio in the section of the tower below plate 83 about 6:1 to 8:1. Instead of introducing all of the propane at the base of the tower we may introduce about one-third or one-half of the propane through line 19 and heater 20 to maintain a tower temperature of about 145° F. at this point.

The high temperature below plate 83 tends to knock back all of the asphaltic components so that a propane bright stock solution may be withdrawn through line 86 to flash chamber 87 from which a substantial amount of the propane may be withdrawn through line 88. The partially depropanized oil may then be charged by pump 89 through heater 90 to side stream fractionator 91, pump 89 maintaining a constant liquid level in chamber 87. A temperature gradient of at least 10 to 20° F. is maintained in fractionator 91 by means of heating coils 92. This temperature gradient insures the removal of asphaltic components from the bright stock solution which is withdrawn to a depropanizing system through line 93. The asphalt phase is withdrawn from the base of fractionator 91 through line 94 at such a rate as to maintain a substantially constant interface level in the fractionator. This propane-lean asphalt solution is returned to an intermediate point in the lower part of the tower, preferably below the interface level therein.

To the asphalt removed from the base of the tower through line 14 further amounts of propane may be added through line 95. This causes further phase separation and the resulting liquids may be introduced by line 96 into fractionator 97. The top temperature of this fractionator may be held at about 150° F. by means of heating coil 98. Additional propane may be introduced at the base of fractionator 97 through line 99 to maintain a bottom fractionator temperature of about 120° F. The propane-rich phase from the top of this fractionator may be returned by pump 100 through line 101 to an intermediate point of the lower tower section, preferably above the interface. The final asphalt solution may be withdrawn through line 102 in such amounts as to maintain a constant interface level in fractionator 97. By operating in the manner described in connection with Figure 4, the 22.1° A. P. I. residuum is fractionated to give about 50% of an SAE 30 stock having an A. P. I. gravity upwards of 27 and a viscosity of about 67 seconds Saybolt at 210° F., about 13% of an SAE 50 stock having an A. P. I. gravity of about 24 and a Saybolt viscosity of about 110 seconds at 210° F., about 9% of a bright stock having an A. P. I. gravity of about 22, a viscosity of about 190 seconds at 210° F. and about 28% of an asphalt having an A. P. I. gravity of about 11 or 12° and a furol viscosity at 210° F. upwards of 460 seconds. This method of fractionating a long residuum into lubricating oils of various grades has distinct advantages over the distillation methods heretofore employed for this purpose because it avoids all thermal decomposition and results in better qualities, better colors, better Conradson carbons at given viscosity, etc. The specifications of the

withdrawn side streams may be held within very close limits by the side stream fractionation and the materials rejected by side stream fractionation are returned to the main countercurrent contacting system without in any way disrupting the operation thereof.

The side stream fractionation may be effected in a large variety of different ways. Whenever the side stream is a propane-rich fraction, i. e., a fraction withdrawn from the upwardly moving phase in a countercurrent tower it is generally desirable to use propane separation and further heating for effecting further phase separation. Thus in Figure 5 whenever a downwardly extending baffle 16' is employed at the side stream draw-off point we may either withdraw the stream through line 86' to a propane vaporizer 87' or we may pass a side stream directly through line 72' by means of pump 73', through heater 90' to fractionating tower 74'. Since heating coils 75' are generally employed in such side stream fractionation means the heater 90' is not always necessary. When the object is to remove heavier components from the withdrawn side stream these heavier components may be returned through line 76' to the tower below the point of side stream draw-off and the desired side stream may be withdrawn to a depropanizer through line 54'. When it is desired to remove lighter components from the side stream the desired fraction is withdrawn to the depropanizer through line 103 and the overhead may be returned to the tower above the point of side stream withdrawal through line 104. In cases where it is undesirable to increase the propane concentration in the upper part of the tower the overhead from fractionator 74' may be passed through line 77' to flash chamber 78' for removal of propane through line 79' and the depropanized overhead may be returned by pump 80' through heater 81' and line 105 to a point in the tower above the side stream draw-off. A portion of this depropanized overhead may also be returned through line 106 to the upper part of the side stream fractionator for decreasing the propane concentration therein. The portion of this overhead not returned to fractionator 74' or tower 11 may be withdrawn to a depropanizing system through line 107.

When the side stream is a propane-lean phase the side stream fractionation system will be along the lines illustrated in Figure 3 or along the lines illustrated by fractionator 97 in Figure 4. In this case further phase separation may be effected by the addition of propane or by heating or by release of pressure. Either the light phase or the heavy phase may be returned to the tower depending upon the characteristics of the side stream.

In Figure 6 we have illustrated a specific example of our invention embodying certain features of the system shown in Figure 4 but employing a plurality of towers wherein the "side stream" refluxing is obtained in the top of each tower instead of in a separate fractionator. This system is designed for fractionating about 2200 barrels per day of a Mid-Continent long residuum into SAE 30 oil, SAE 50 oil, bright stock and asphalt. The charging stock in this case has the following characteristics:

A. P. I. gravity	degrees	22.1
Viscosity SSU at 210° F	seconds	156.8
Color Tag Robinson	DD	1½
Carbon residue	per cent	3.9
Flash	°F	500
Viscosity gravity constant		.842

This stock is charged through line 110 to an upper part of tower 111 which tower may be about 10 feet in diameter by about 40 feet high. Heating coils 112 are employed at the top of this tower for maintaining a tower-top temperature of about 200° F., the tower operating under a pressure of about 550 to 600 pounds per square inch.

The tower temperature may be maintained at about 190° F. at the point of oil inlet, the charging stock being heated in heat exchanger 113 to the extent necessary for obtaining such temperature.

About 4 volumes of propane (based on charging stock) may be pumped from storage tank 114 by pump 115 through line 116 and heat exchanger 117 to the base of tower 111 at such temperature as to maintain a tower bottom at about 170° F. About 2 volumes of propane are introduced through line 118 and heat exchanger 119 at an intermediate point in said tower at such temperature to maintain a tower temperature at this point of about 180° F. The propane precipitated material is withdrawn from the base of this tower through line 120 at such a rate as to maintain a constant interface level in the tower which is preferably spaced from the bottom of said tower but below the intermediate point of propane inlet. The withdrawn propane-lean material is introduced through heat exchanger 121 at an upper point of tower 122 the supplied heat being sufficient to maintain a tower temperature at this point of about 175° F. Heating coils 123 maintain a tower-top temperature of about 185° F. About 2 volumes of propane based on original charge are introduced through line 124 and heat exchanger 125 into the base of tower 122, the added heat being sufficient to mainttain a tower bottom temperature of about 155° F. About 1 volume of propane based on original charge is passed through line 126 and heat exchanger 127 into an intermediate point of tower 122 for maintaining the tower temperature at this point of about 165° F.

The precipitated material from tower 122 is withdrawn from the base thereof through line 128 in such amounts as to maintain a substantially constant interface level in the tower. This precipitated material is then passed through heat exchanger 129 and introduced at an upper point in tower 130 at such temperature as to maintain a tower temperature at this point of about 160° F. Heating coils 131 maintain a tower-top temperature of about 170° F. About 1 volume of propane based on original charge is introduced through line 132 and heat exchanger 133 to the base of tower 130 in order to maintain a tower bottom temperature of about 140° F. An additional volume of propane based on original charge is passed through line 134 and heat exchanger 135 to an intermediate point of tower 130 at such temperature that the tower temperature at this point will be about 150° F.

The precipitated material from tower 130 is withdrawn through line 136 at such a rate as to maintain a substantially constant interface level in the tower. The withdrawn stream is passed through a heat exchanger or pipe still 137 wherein it is heated to a temperature of about 450° F. and then introduced into a propane flash drum 138 which operates under such pressure that the vaporized propane which leaves this flash drum through lines 139 and 140 may be condensed in cooler 141 and returned by line 142 to storage tank 114 without the use of compressors. Pro-

pane storage tank 114 is preferably maintained at a temperature of about 130° F.

Depropanized oil from the base of flash drum 138 passes through line 143 to the upper part of low pressure stripper 144 wherein it is stripped with steam introduced through line 145. The propane vapors and steam are taken overhead through line 146 to jet condenser 148 wherein the steam is condensed by water introduced through line 149. The water and condensed steam is withdrawn through line 150 to the sewer. Propane from the jet condenser is conducted by line 151 to liquid trap 152 from which any additional water may be withdrawn through line 150. Propane vapors from the top of trap 152 are compressed by compressor 153 and passed by line 154 to condenser 155 the condensed propane being introduced by line 156 into propane storage tank 157 which is maintained at a temperature of about 100° F. Tank 157 is the main storage tank and propane from this tank is pumped into tank 114 by means of pump 158. When the liquid level in tank 114 gets too low additional propane is introduced by means of pump 158. When the liquid level in this tank gets too high, propane is vented therefrom through valve 159 and line 160 back to tank 157.

The propane solution from the top of tower 111 is passed by line 161 to depropanizing system 162. The stream from the top of tower 122 is passed by line 163 to the top of depropanizing system 164. The stream from the top of tower 130 passes by line 165 to depropanizing system 166. Since each of these depropanizing systems is similar to the depropanizing employed for the asphalt stream, further detailed description is unnecessary.

About 660 barrels per day of asphalt is withdrawn through line 167. This asphalt has an A. P. I. gravity of about 11 or 12 and a furol viscosity upwards of 460 seconds at 210° F.

About 220 barrels per day of bright stock is withdrawn from the system through line 168. This bright stock has an A. P. I. gravity of about 22°, a viscosity of about 190 seconds Saybolt at 210° F., a color or about 7D or 8D Tag Robinson, a carbon residue slightly over 1%, a flash of about 565° F., and a viscosity gravity constant of about .837.

About 330 barrels per day of SAE 50 stock is removed from this system through line 169. This stock has an A. P. I. gravity of about 24°, a viscosity at 210° F. of about 110 seconds, a Tag Robinson color of about 5D, a carbon residue of about .7% and a viscosity gravity constant of about .83.

About 990 barrels per day of SAE 30 oil is removed from the system through line 170. This oil has an A. P. I. gravity of about 27 or 28, a viscosity at 210° F. of about 60 to 65, a Tag Robinson color of about 4, a carbon residue of about .4 and a viscosity gravity constant of about .82.

While we have described a number of specific examples of our invention it should be understood that our invention is not limited to these particular examples nor to the particular charging stocks employed nor to the use of pure propane. The word "propane" as used in the above specification and in the following claims is intended to mean 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 lower pressures may be employed if butane is present in appreciable amounts. Generally speaking, we prefer to employ a "propane" which is substantially free from olefins.

The precise temperatures and temperature gradients employed in our countercurrent system, side stream fractionation means, etc., will depend, of course, upon the nature of the stock which is being fractionated and particularly on the relative amounts of various molecular weight components in the charge. The approximate propane-to-oil ratios and temperatures required for separating various charging stocks are already known to those skilled in the art or may be readily ascertained by simple preliminary experiments. Generally speaking, we prefer to employ propane-to-oil ratios within the approximate range of 6:1 to 10:1 or more and we prefer to operate within the approximate temperature range of about 120 to 200° F. We prefer to employ a temperature gradient at the top of each tower or fractionator within the approximate range of 10 to 30° F.

From the above description it will be noted that the upward flow rate at the top of tower 111 was within the approximate range of 1 foot per minute. Because of the lesser quantity of materials and the greater graivty differentials in towers 122 and 130 these towers may have diameters of only about 8 feet and 7 feet respectively. In order to obtain equilibrium conditions and good separation the vertical flow rates throughout the system should not substantially exceed about 1 foot per minute. The specific side stream fractionation conditions will, of course, be dependent upon the specific characteristics desired in the withdrawn side stream and those skilled in the art will be enabled from the above description to select or determine the proper conditions in any specific case.

We claim;

1. The method of fractionating an oleiferous charging stock which method comprises continuously countercurrently contacting said charging stock with propane in a unitary fractionation zone, continuously withdrawing a side stream of intermediate characteristics from an intermediate point in said fractionation zone, fractionating the withdrawn side stream of intermediate characteristcs, returning one fraction thereof in liquid phase to said countercurrent fractionation zone, continuously withdrawing a propane-lean fraction from one end of said zone and continuously withdrawing a propane-rich fraction from the other end of said zone.

2. The method of claim 1 wherein the propane-lean fraction is withdrawn at a relatively low temperature, the propane-rich fraction is withdrawn at a relatively high temperature and the intermediate fraction is withdrawn at an intermediate temperature from said fractionation zone.

3. The method of claim 1 wherein the intermediate fraction is withdrawn below the point of charging stock inlet.

4. The method of claim 1 wherein the intermediate fraction is withdrawn from a point above the charging stock inlet.

5. The method of claim 1 wherein propane is introduced into the fractionation zone at a plurality of spaced points between the point of charging stock inlet and the point of propane-lean product withdrawal.

6. The method of operating a countercurrent propane fractionation system for the production

of at least three different products which method comprises maintaining a low temperature at one end of a unitary fractionation zone, maintaining a high temperature at the other end of said zone, maintaining an intermediate temperature at an intermediate point in said zone, withdrawing a side stream from an intermediate point in said zone, heating said withdrawn side stream to effect further liquid phase separation, and returning one of said separated phases to said fractionation zone.

7. The method of operating a countercurrent propane fractionation system for obtaining at least three different products therefrom which method comprises countercurrently contacting a charging stock with propane in a fractionation zone, maintaining a low temperature and low propane-to-oil ratio at one end of said zone, maintaining a high temperature and a high propane-to-oil ratio in the other end of said zone, maintaining an intermediate temperature at an intermediate point in said zone, withdrawing a side stream from an intermediate point in said zone, altering the propane-to-oil ratio in said side stream for effecting further liquid phase separation and returning one of said separated phases to said contacting zone.

8. The method of claim 7 wherein the withdrawn side stream is rich in propane and wherein propane is removed from the withdrawn side stream to effect further phase separation.

9. The method of claim 7 wherein the removed side stream is lean in propane and wherein propane is added thereto to effect further phase separation.

10. The method of claim 7 which includes the further step of heating the withdrawn side stream to effect further phase separation.

11. In a propane fractionation system comprising a vertical tower and at least one liquid trap at an intermediate point in said tower, the method of fractionating a charging stock to produce at least three separate fractions which method comprises introducing propane at the base of said tower, introducing a charging stock at a point in said tower spaced from the base whereby a countercurrent flow is effected in the base of said tower, heating the top of said tower to cause phase separation and a downward flow of propane insoluble material from the top toward the bottom of the tower, withdrawing one product from the top of said tower, withdrawing another product from the bottom of said tower, withdrawing a third product stream from a point adjacent said trap at the intermediate point in said tower, separating a liquid propane-rich oil fraction from the withdrawn third product stream and returning said liquid propane-rich oil fraction to said tower at an intermediate point thereof.

12. The method of claim 11 wherein the third product is withdrawn from said tower at a point above the point of charging stock inlet.

13. The method of claim 11 wherein the third product is withdrawn from the tower at a point below the charging stock inlet.

14. The method of fractionating a long residuum into at least three separate fractions in a countercurrent propane fractionation system which method comprises maintaining one end of said fractionation system at a temperature within the approximate range of 180 to 200° F., maintaining the other end of said fractionation system within the approximate temperature range of 120 to 140° F., maintaining an intermediate point in said fractionation system at a temperature within the approximate range of 150 to 170° F., introducing the residuum into said fractionation system at a point wherein the temperature is at least about 150° F., maintaining countercurrent flow in said system so that propane with dissolved lower viscosity oil components moves toward the high temperature end of said system and wherein precipitated high viscosity components diluted with propane move from the high temperature to the low temperature end of said system, withdrawing a stream from said system at an intermediate point as well as from the top and bottom thereof, effecting a further phase separation of said withdrawn stream and returning one of said separated phases to said countercurrent system.

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