

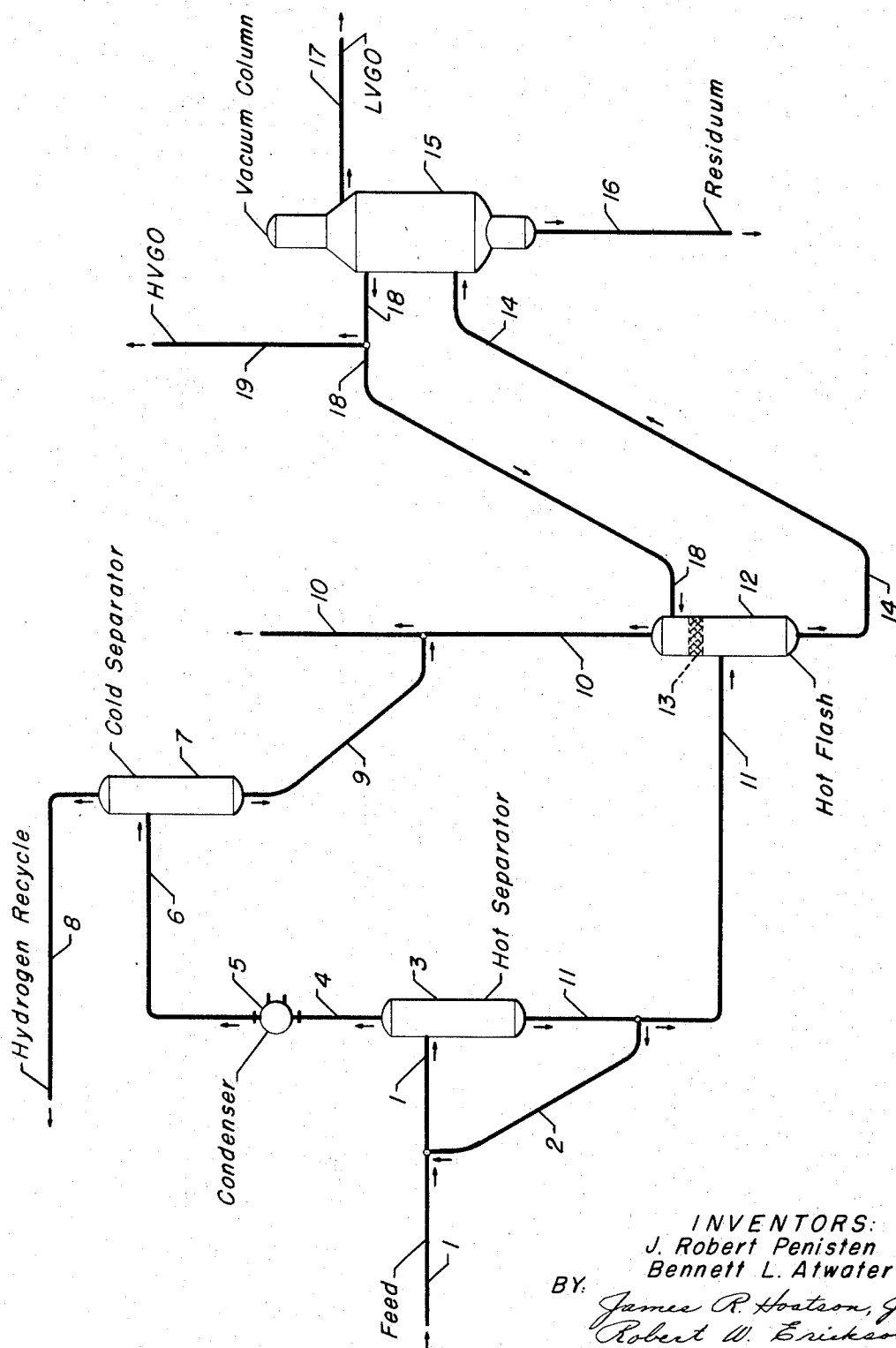
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BLACK OIL CONVERSION PRODUCT SEPARATION PROCESS

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INVENTORS:
J. Robert Penisten
Bennett L. Atwater
BY: *James R. Hoatson, Jr.*
Robert W. Erickson
ATTORNEYS

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BLACK OIL CONVERSION PRODUCT SEPARATION PROCESS

J. Robert Penisten, Palatine, and Bennett L. Atwater, Elk Grove Township, Ill., assignors to Universal Oil Products Company, Des Plaines, Ill., a corporation of Delaware

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The present invention involves a process for effecting the separation of a conversion product effluent, and particularly encompasses the separation of a mixed phase product effluent. More specifically, the inventive concept described herein is directed toward a scheme for separating the hydrocarbonaceous product effluent resulting from the conversion of a charge stock containing high molecular weight hydrocarbons boiling above a temperature of about 1050° F. This conversion product effluent is in mixed phase, and generally comprises hydrogen, and acid gas, ammonia, normally gaseous hydrocarbons and normally liquid hydrocarbons.

Although the separation process is applicable to a hydrocarbon conversion process which may be broadly classified as hydrogen-consuming, it is most advantageously adaptable to a black oil conversion process. This conversion process employs hydrocracking/hydrorefining techniques for the principal purposes of (1) reducing the concentration of various contaminating influences (sulfur and nitrogen-containing compounds as well as metallic complexes), and (2) to convert the heavy hydrocarbonaceous material into lower boiling hydrocarbon products reduced in the concentration of the foregoing contaminants. Illustrative of hydrocarbonaceous material classified as "black oil" are atmospheric tower bottoms products, vacuum tower bottoms products, crude oil residuum, "topped" crude oils, oils extracted from tar sands, etc.

Black oils, particularly those extracted from tar sands, atmospheric and vacuum residuum, contain high molecular weight sulfurous compounds, large quantities of nitrogenous compounds, high molecular weight organo-metallic complexes, principally comprising nickel and vanadium, and a considerable quantity of asphaltic material insoluble in lower boiling hydrocarbons such as pentane and/or heptane. An abundant supply of such hydrocarbonaceous material currently exists, most of which has a gravity ° API at 60° F., less than 20.0, and a significant proportion of which has a gravity less than 10.0° API. Black oils are further characterized in that the boiling range thereof indicates that 10.0% by volume, or more, boils above a temperature of 1050° F. Although not known accurately, a large amount of black oil is currently available, and is characterized in that more than 50.0% by volume boils above a temperature of about 1050° F. The conversion of at least a portion of such material into distillable hydrocarbons—i.e. those boiling at temperatures below about 1050° F.—has hitherto been economically nonfeasible.

Specific examples of the black oils, a process for the conversion of which advantageously utilizes the separation process of our invention, include a vacuum tower bottoms product having a gravity of 7.0° API at 60° F., and containing 4.1% by weight of sulfur and 23.7% by weight of asphaltics; a Middle-East crude oil having a gravity of 11.0° API at 60° F., containing 10.1% by weight of asphaltics and about 5.2% by weight of sulfur; and a vacuum residuum having a gravity of 8.8° API at 60° F., and containing 3.0% by weight of sulfur and 4300 p.p.m. of nitrogen, and having a 20.0% volumetric distillation temperature of 1055° F. In general, the asphaltics are found to be colloiddally dispersed in the black oil, and, when subjected to elevated temperature,

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have the tendency of flocculate and polymerize, whereby the conversion thereof to more valuable oil-soluble products becomes extremely difficult. Thus, for example, the heavy bottoms from a crude oil vacuum distillation column indicates a Conradson Carbon Residue factor of 16.0% by weight.

A principal object of the present invention is to provide an improved process for the separation of a conversion product effluent containing hydrocarbonaceous material boiling above a temperature of about 1050° F. A corollary objective is to separate a conversion product effluent containing hydrogen, an acid gas, normally gaseous hydrocarbons and normally liquid hydrocarbons at least some of which boil above a temperature of about 1050° F.

Another object of this invention is to afford a mixed-phase conversion product effluent separation process in which the recovered normally liquid hydrocarbons boiling below a temperature of 1050° F. are substantially completely free from those non-distillable hydrocarbons boiling above about 1050° F.

Still another object of the present invention is to provide a separation process which produces an internally recycled wash oil used within the process to remove non-distillable asphaltics from equipment employed therein.

These and other objects are achieved by the present invention as more completely described hereinbelow, and especially with reference to the accompanying drawing which is a simplified representation of one embodiment.

As hereinbefore stated, the conversion product effluent is in mixed-phase, and contains hydrogen, hydrogen sulfide, normally gaseous hydrocarbons including methane, ethane and propane, normally liquid gasoline boiling range hydrocarbons including pentane, hexane and hydrocarbons boiling up to about 400° F., middle-distillate hydrocarbons, gas oil boiling range hydrocarbons, and hydrocarbonaceous material boiling above 1050° F. In the present specification and appended claims, butanes will be considered in the classification of normally liquid hydrocarbons since they are generally recovered as a desired product due to their blending value with respect to motor fuel. Also, the term "non-distillables" is intended to connote those hydrocarbons having normal boiling points above about 1050° F. From this type of product effluent, it is generally intended to recover or produce, at least the following as product streams: (1) a 650° F.-plus hydrocarbon fraction suitable for use as fuel oil; (2) a 400° F.-650° F. middle-distillate fraction for use either as fuel oil, or as the charge stock to a hydrocracking unit designed for maximum LPG (liquefied petroleum gas) production; (3) a gasoline boiling range fraction which may or may not contain butanes and pentanes; (4) a butane-pentane concentrate for use as a motor fuel blending component, or as the charge stock to an isomerization unit; (5) a hydrogen-rich gaseous phase to be recycled to the conversion zone to supply a portion of the hydrogen consumed therein; and, (6) a fuel gas waste product comprising methane and ethane, and being substantially free from the more valuable heavier hydrocarbons.

Usually, the mixed-phase conversion zone effluent is introduced, after substantial cooling, into a high-pressure, low temperature separator in order to recover unreacted hydrogen for use as an internal recycle stream within the process, followed by subsequent separation and recovery of distillable hydrocarbon products through the use of distillation and/or fractionation means. Such a scheme suffers from at least two drawbacks when the conversion product effluent results from the hydrocracking/hydrorefining of black oils. Large quantities of hydrogen and normally gaseous hydrocarbons are in the product effluent, and the normally liquid hydrocarbon por-

tion thereof has a high absorption capacity for such material. The conversion product effluent also contains the unreacted non-distillables (1050° F.-plus material), generally referred to as residuum, which material has the tendency to cause plugging of various lines and equipment. The present invention is primarily concerned with the latter problem and with the solution of the processing difficulties which stem therefrom.

Therefore, in a broad embodiment, the present invention relates to a process for separating a mixed-phase hydrocarbonaceous conversion product effluent containing hydrocarbons boiling above about 1050° F. (asphaltic non-distillables), which process comprises the steps of: (a) separating said product effluent in a first separation zone at a temperature above about 700° F. and a pressure above about 1000 p.s.i.g., to provide a first vapor phase and a first liquid phase containing asphaltics; (b) condensing said first vapor phase and separating the condensed vapors in a second separation zone at a temperature below about 140° F. and a pressure substantially the same as said first separation zone, to provide a hydrogen-rich second vapor phase and a second liquid phase principally comprising propane and heavier normally liquid hydrocarbons; (c) introducing at least a portion of said first liquid phase into a third separation zone at a point below a mesh blanket disposed within said third separation zone, at a temperature substantially the same as said first separation zone and a pressure less than about 200 p.s.i.g., to provide a third vapor phase and a third liquid phase containing hydrocarbons boiling above 1050° F.; (d) further separating said third liquid phase in a fourth separation zone at a temperature above about 700° F. and at a subatmospheric pressure to provide a residuum fraction containing those hydrocarbons boiling above about 1050° F. and a heavy gas oil fraction boiling below about 1050° F.; and, (e) introducing at least a portion of said heavy gas oil fraction into said third separation zone at a point above said mesh blanket.

Other embodiments of our invention reside in particular operating conditions and in the use of various internal recycle streams. The quantity of heavy gas oil being introduced into the third separation zone, herein referred to as the hot flash zone, is within the range of from 1.0% to about 10.0% by volume of the total heavy gas oil produced. An intermediate and generally preferred range is from about 3.0% to about 8.0%. Also, a portion of the first liquid phase is recycled to combine with the conversion product effluent, prior to separation in the first separation zone which is herein referred to as a hot separator. The hot separator is maintained at essentially the same pressure as the conversion product effluent, and this pressure is nominally in the range of from about 1000 p.s.i.g. to about 4000 p.s.i.g. The preferred temperature of the product effluent entering the hot separator is below about 750° F. At temperatures above 750° F., heavier normally liquid hydrocarbons tend to be carried over into the first vapor phase, while at temperatures below about 700° F., ammonium salts, from the conversion of nitrogenous compounds, tend to fall into the liquid phase. Therefore, that portion of the first liquid phase being recycled to combine with the product effluent, is first utilized as a heat-exchange medium to the extent necessary to lower its temperature to a level such that the combined charge to the hot separator is at a temperature generally in the range of from 700° F. to about 750° F.

The second separation zone, herein referred to as a cold separator. Although functioning at essentially the same pressure as the product effluent and hot separator, is maintained at a temperature of from about 60° F. to about 140° F. The pressure levels maintained upon the third and fourth separation zones are substantially reduced from the pressure under which the hot and cold separators are maintained. The maximum pressure on the third separation zone will be about 200 p.s.i.g., and this zone will function at an elevated temperature somewhat less than the temperature of the first liquid phase emanat-

ing from the hot separator. Preferably, the fourth separation zone is operated as a vacuum column at a subatmospheric pressure less than about 100 mm. of Hg. The third liquid phase, intended to be introduced into the vacuum column may be at a temperature less than about 700° F., and is, therefore, heated to a temperature above 700° F. with an upper limit of about 900° F., prior to being introduced into the vacuum column.

Prior to discussing the present process in detail, and especially in conjunction with the accompanying drawing, several definitions of various terms and phrases are believed necessary for a clear and complete understanding. References to boiling points and temperature ranges of various hydrocarbons, and mixtures of hydrocarbons, are those obtained through the use of ASTM Standard Distillation Methods. Likewise, the phrase "hexane-400° F." is intended to mean a normally liquid stream boiling below a temperature of 400° F. and including hexanes. Similarly, the term "650° F.-plus" connotes a liquid stream containing those hydrocarbons boiling at 650° F. and above.

The phrase "gasoline boiling range hydrocarbons," is intended to include normally liquid hydrocarbons boiling up to about 400° F. It is understood that the upper temperature limit, or end boiling point, of "gasoline" varies from locale to locale, and is, therefore, considered to be 425° F. or 450° F. For present purposes, however, "normally liquid gasoline hydrocarbons" is intended to include butanes and have a nominal end boiling point of 400° F.

A "pressure substantially the same as," is intended to indicate that the pressure imposed upon a particular vessel is that pressure imposed upon an upstream vessel, allowing only for the normal pressure loss due to fluid flow through the system from one vessel to the other. Also, a "temperature substantially the same as," is used to show that any reduction in temperature stems from normally experienced radiation losses due to the flow of material or from the conversion of sensible to latent heat by "flashing" where a pressure drop occurs.

The process can be further characterized in that the hot separator is employed primarily to provide a first vapor phase substantially completely free from 1050° F.-plus material, concentrating the latter in a first liquid phase. The cold separator, operating at substantially the same pressure as the hot separator, but at a lower temperature in the range of 60° F. to 140° F., serves to concentrate the hydrogen in the condensed first vapor phase. As hereinafter indicated, a hydrogen-rich second vapor phase, comprising about 82.5 mol percent hydrogen, and only about 2.3 mol percent propane and heavier hydrocarbons, is made available for use as a recycle stream to be combined with the fresh black oil charge stock. The liquid phase from the cold separator comprises about 66.7% by volume of butanes and heavier hydrocarbons, and only about 1.9 volume percent boiling in the range of 650° F. to 1050° F.

The hot flash zone functions at a temperature substantially the same as the hot separator, but at a significantly reduced pressure less than 200 p.s.i.g. This vessel serves primarily to concentrate the 400° F.-plus hydrocarbons in a third liquid phase, while also producing a third vaporous phase substantially free from the 1050° F.-plus material and containing only a minor amount of 650° F.-1050° F. hydrocarbons. In order to ensure that none of the unreacted asphaltics present in the conversion product effluent "contaminate" the material of the third vapor phase, with the consequence that various downstream fractionation facilities become plugged the hot flash zone is supplied with a mesh blanket, or de-mister screen, below which the first principally liquid phase, from the hot separator is introduced. Since the hot flash zone is operated at a significantly reduced pressure—i.e. from about 2600 p.s.i.g. down to 65 p.s.i.g.—and at a temperature slightly less than about 750° F., the material

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entering the vessel is flashed with the result that asphaltics have the tendency to be carried overhead with the third principally vaporous phase. Since subsequent separation and fractionation facilities will be maintained at significantly lower temperatures, the continual flashing of these asphaltics ultimately results in plugged lines and heat-exchange equipment, etc. To counteract this, a mesh blanket is inserted, or disposed, within the hot flash zone at a locus above the point where the first liquid phase is introduced. The flashed asphaltics are unable to pass through the screen, and thus do not "contaminate" the material boiling up to about 1050° F., thereby avoiding misoperation of subsequent facilities employed to separate the product into the desired fractions.

The third liquid phase, containing more than 95.0 mol percent 400° F.-plus material, is further separated to concentrate the asphaltics in a residuum fraction and to prepare a wash oil employed to continuously remove the trapped asphaltics from the mesh blanket disposed in the hot flash zone. As hereinafter indicated in describing the drawing, this is perhaps best accomplished through the use of a vacuum column operating at a subatmospheric pressure of 100 mm. of Hg, or less. In this manner, the asphaltic residuum is recovered as a separate bottoms stream substantially free from distillable hydrocarbons. More importantly, the proper kind or type of wash oil is readily prepared. Since the hot flash zone is functioning at a temperature of from 700° F. to somewhat less than 750° F. due to heat loss in transferring material from the hot separator, the wash oil itself should be one which is normally liquid at this temperature. That is, it should be of the character which may be condensed in the upper region of the hot flash zone (above the mesh blanket) so that, as a heavy liquid stream, it will tend to pass down through the mesh blanket and be removed with the liquid phase. In so doing, the wash oil, which has a high capacity for the asphaltics, removes them from the mesh blanket, and carries them into the vacuum column wherein they are removed from the process as the residuum. The wash oil prepared by the present process may be characterized as a heavy gas oil, or, since the use of a vacuum column as the fourth separation zone is preferred, as a heavy vacuum gas oil (HVGO). The amount employed by way of recycle to the hot flash zone, at a point above the mesh blanket, is from 1.0% to about 10.0% by volume of the total heavy gas oil produced. By way of definition, a heavy vacuum gas oil is the higher boiling 70.0% to about 80.0% by volume portion of the total gas oil, the latter including that portion considered in the art as a light vacuum gas oil (LVGO). A commonly referred to boiling range for gas oil is an initial boiling point of 650° F. and an end boiling point of about 1050° F. The higher boiling 70.0% to 80.0% thereof, the heavy gas oil, characteristically is considered as having an initial boiling point of about 750° F. It is, of course, recognized that a "light vacuum gas oil" can have an initial boiling point as low as 500° F. and an end boiling point as high as about 800° F. Similarly, the "heavy vacuum gas oil" can have an initial boiling point as low as 700° F.

Thus, the separation process herein described, and as illustrated in the accompanying drawing, results in five different, principal product streams. A first product stream is a principally gaseous phase containing more than about 80.0% by volume of hydrogen; it is, therefore, advantageously utilized as a recycle stream to supply a portion of the hydrogen required in the conversion zone. A second, principally liquid product stream is obtained by combining the second liquid phase from the cold separator and the third vapor phase (preferably after being suitably cooled) from the hot flash zone, and comprises more than about 60.0% butanes and heavier hydrocarbons. As hereinafter indicated, this stream can be conveniently treated and/or fractionated in order to recover various component fractions thereof.

A third product stream is the heavy vacuum gas oil, a

portion of which is recycled as wash oil to the hot flash zone. The remainder may be, where desired, combined with the fourth product stream which is the light vacuum gas oil fraction. Obviously, the fifth product stream is the asphaltic residuum.

From the foregoing brief description, it will be readily ascertained by those possessing skill in the art of petroleum processing techniques, that the present invention comprises a series of integrated steps for the separation of a mixed-phase reaction product effluent, resulting from a black oil conversion process, in an easy and economical manner. The conversion of black oils is generally intended to accomplish primarily two objects: first, to desulfurize the black oil to the extent dictated by the desired end result, whether maximizing fuel oil, or gasoline boiling range hydrocarbons; secondly, it is intended to produce "distillable hydrocarbons," being those normally liquid hydrocarbons including pentanes, and for present purposes butanes, having boiling points below about 1050° F. The conversion conditions are intended to include temperatures above about 600° F., with an upper limit of about 800° F., as measured at the inlet to the fixed-bed of catalyst disposed within the reaction zone. Since the bulk of the reactions being effected are exothermic, the reaction zone effluent will be at a higher temperature. In order that catalyst stability be preserved, it is generally preferred to control the inlet temperature at a level such that the temperature of the reaction product effluent does not exceed 950° F. Hydrogen is admixed with the black oil charge stock, by means of compressive recycle, in an amount usually less than about 10,000 s.c.f./bbl., at the selected operating pressure; the hydrogen is present in the recycle gaseous phase preferably in an amount of about 80.0% or more. A preferred range of the quantity of hydrogen being admixed with the fresh black oil charge stock is from about 3000 to about 8000 s.c.f./bbl. The conversion reaction zone will be maintained at a pressure greater than about 1000 p.s.i.g., generally with an upper limit of about 4000 p.s.i.g. The black oil passes through the catalyst at a liquid hourly space velocity (defined as volumes of liquid hydrocarbon charge per hour, as measured at 60° F., per volume of catalyst disposed within the reaction zone) of from about 0.25 to about 2.0.

As hereinbefore set forth, hydrogen is employed in admixture with the charge stock, and preferably in an amount of from about 3000 to about 8000 s.c.f./bbl. The hydrogen-containing gaseous phase, herein sometimes designated as "recycle hydrogen" since it is conveniently recycled externally of the conversion zone, fulfills a number of various functions; it serves as a hydrogenating agent, a heat carrier, and particularly a means for stripping converted material from the catalytically active sites for the incoming, unconverted hydrocarbon charge stock. In view of the fact that some hydrogenation will be effected, there will be a net consumption of hydrogen; to supplement this, hydrogen must be added to the system from a suitable external source. The catalytic composite disposed within the reaction zone can be characterized as comprising a metallic component possessing hydrogenation activity, which component is composited with a refractory inorganic oxide carrier material which may be of either synthetic or natural origin. The precise composition and method of manufacturing the catalytic composite is not considered to be an essential element of the present process.

Other conditions and preferred operating techniques will be given in conjunction with the following description of one embodiment incorporating the separation process of the present invention. In further describing this process, reference will be made to the accompanying figure which is presented for the sole purpose of illustration. In the drawing, the embodiment is presented by means of a simplified flow diagram in which such details as pumps, instrumentation and controls, heat-exchange and heat-recovery circuits, valving, start-up lines and similar hardware have been omitted as being non-essential to an understand-

ing of the techniques involved. The use of such miscellaneous appurtenances, to modify the illustrated process flow, are well within the purview of those skilled in the art.

For the purpose of demonstrating the illustrated embodiment, and the utilization therein of the process of the present invention, the drawing will be described in connection with the conversion of a vacuum residuum having a gravity of 8.8° API at 60° F., and an ASTM 20.0% volumetric distillation temperature of 1055° F. In addition, the description will be directed toward a commercially-scaled unit having a capacity of about 10,000 bbl./day of fresh charge. It is to be understood that the charge stock, stream compositions, operating conditions, design of fractionators, separators and the like are exemplary only, and may be varied widely without departure from the spirit of my invention, the scope of which is defined by the appended claims. Other charge stock properties are presented in the following Table I:

TABLE I.—VACUUM RESIDUUM PROPERTIES

Gravity, °API at 60° F.	8.8
Distillation, D1160, °F.:	
Initial boiling point	690
2%	860
5%	950
10%	1000
20%	1055
Sulfur, wt. percent	3.0
Nitrogen, total p.p.m.	4300
Heptane insolubles, wt. percent	6.5

This vacuum residuum is intended to be converted into 80.0% by weight of hydrocarbon products recoverable by standard distillation in commonly used fractionation facilities. Furthermore, it is intended that this object be accomplished with minimal production of methane and ethane, and minimal loss of propane as a gaseous waste product. Obviously, a corollary object is to maximize the recovery of normally liquid hydrocarbons, inclusive of butanes. The vacuum residuum is processed in a fixed-bed catalytic conversion zone in admixture with 5000 s.c.f./bbl. of hydrogen, at an inlet pressure of about 2700 p.s.i.g. and an inlet temperature of about 800° F. The liquid hourly space velocity, based upon fresh liquid feed only, is 0.5, and the combined feed ratio with respect to total liquid feed is 2.0.

With reference now to the drawing, the black oil conversion product effluent, in mixed phase and containing about 9.4% by weight of asphaltic residuum, is introduced into hot separator 3 via line 1, and after being admixed with hot separator recycle in line 2, the source of the latter hereafter set forth. The conditions of the product effluent, at the outlet of the conversion zone are a temperature of about 875° F. and a pressure of about 2550 p.s.i.g. Prior to entering hot separator 3, the 313,000 lbs./hr. of conversion zone effluent is employed as a heat-exchange medium in order to lower its temperature, and is then combined with 142,000 lbs./hr. of hot separator recycle. The latter, after use as a heat-exchange medium, exists at a temperature of about 400° F. The temperature, therefore, of the total material entering separator 3 is about 750° F., the pressure being about 2535 p.s.i.g. A principally vaporous phase, in an amount of about 60,500 lbs./hr. is removed via line 4 into condenser 5, while a total of about 134,000 lbs./hr. is recycled, while hot (about 875° F.), to combine with the fresh charge stock to the conversion zone. However, since this does not constitute an essential element of our separation process, this recycle system is not illustrated in the drawing. As previously set forth, 142,000 lbs./hr. is diverted through line 2, and after use as a heat-exchange medium, whereby its temperature is lowered to about 400° F., continues therethrough to combine with the conversion product effluent in line 1.

In the following Table II, the analyses of the various streams involved in the function and operation of hot

separator 3 are presented. For convenience, the quantities are given in mols/hr. for the conversion for the product effluent entering the hot separator (line 1), for the first vapor phase (line 4) and for the net liquid phase to the hot flash zone 12 (line 11).

TABLE II.—HOT SEPARATOR STREAM ANALYSES

Line No.	1	4	11
Component, mols/hr.:			
Ammonia	17	17	-----
Nitrogen	31	29	1.0
Hydrogen Sulfide	247	217	13.7
Hydrogen	4,125	3,834	160.0
Methane	594	539	26.4
Ethane	129	108	9.8
Propane	110	93	8.5
Butane	68	55	6.2
Pentane	31	23	3.4
Hexane, 400° F.	148	93	26.5
400° F.-650° F.	215	53	75.8
650° F.-1,050° F.	308	5	142.4
1,050° F. plus	96	-----	45.2
Totals	6,169	5,066	518.9

No analysis is given for the hot separator recycle stream in line 2; this material remains substantially unchanged, the actual amount being determined on the basis of the temperature of the total material entering hot separator 3.

The vaporous phase in line 4, 60,500 lbs./hr., is condensed in condenser 5, and passes through line 6 into the cold separator 7. In the instant illustration, the material removed as the sour water stream is in the amount of about 370 lbs./hr., exclusive of the wash water.

A hydrogen-rich gaseous phase, in an amount of 27,900 lbs./hr., is withdrawn from separator 7 through line 8, via compressive means not shown, and is recycled thereby to the conversion reaction zone. A net liquid phase, in the amount of 32,230 lbs./hr., is withdrawn through line 9. In the following Table III, component stream analyses are given for the hydrogen-rich gaseous phase (line 8) and the second principally liquid phase (line 9).

TABLE III.—COLD SEPARATOR STEAM ANALYSES

Line No.	8	9
Component, mols/hr.:		
Ammonia	-----	-----
Nitrogen	27	2
Hydrogen Sulfide	136	81
Hydrogen	3,700	134
Methane	452	87
Ethane	74	34
Propane	58	35
Butane	30	25
Pentane	9	14
Hexane, 400° F.	7	86
400° F.-650° F.	-----	53
650° F.-1,050° F.	-----	5
1,050° F. plus	-----	-----
Totals	4,493	556

As indicated in the table, cold separator 7 produces a gaseous phase comprising about 82.5 mol percent hydrogen. It should be noted too that this stream contains only about 2.3% by volume of propane and heavier hydrocarbons. Analysis further indicates that the end boiling point of the hexane-400° F. material is 300° F.

The first liquid phase in line 11 continues therethrough into hot flash zone 12 having disposed therein mesh blanket 13. The material, at a temperature of about 735° F. is introduced at a point below mesh blanket 13. Also introduced into hot flash zone 12, at a point above mesh blanket 13, is 2,135 lbs./hr. of a heavy vacuum gas oil in line 18, the source of which is hereafter described. A third principally vaporous phase is withdrawn via line 10, in the amount of 18,400 lbs./hr., and is admixed with the liquid phase in line 9 as one of the product streams of the present process. A third liquid phase, in the amount of 102,135 lbs./hr., containing the unreacted asphaltics, is removed via line 14, and passes therethrough into vacuum column 15. In the following Table IV, component stream analyses

are given for the vaporous phase from the hot flash zone (line 10) and the liquid phase (line 14) which passes into vacuum column 15. Included in the latter is the 4.4 mols/hr. (2,135 lbs./hr.) of HVGO which is introduced above mesh blanket 13. For convenience, the analysis of the mixture of the second liquid stream (line 9) and the vapor phase (line 10) is indicated in the table as line 9-10.

TABLE IV.—HOT FLASH ZONE STREAM ANALYSES

Line No.	10	14	9-10
Component, mols/hr.:			
Ammonia	1.0	-----	3.0
Nitrogen	13.5	0.2	94.5
Hydrogen Sulfide	158.4	1.6	292.4
Hydrogen	26.1	0.3	113.1
Methane	9.5	0.3	43.5
Ethane	8.2	0.3	43.2
Propane	5.9	0.3	30.9
Butane	3.2	0.2	17.2
Pentane	23.0	3.5	109.0
Hexane, 400° F.	40.4	35.4	93.4
400° F.-650° F.	11.0	135.8	16.0
650° F.-1050° F.	-----	45.2	-----
1050° F. plus	-----	-----	-----
Totals	300.2	223.1	856.2

It should be noted, from the data presented in Table IV, that the product stream combined as lines 9 and 10 is free from hydrocarbonaceous material boiling above about 1050° F. Also, the liquid stream in line 14 comprises only 1.4 mol percent of propane and lighter gaseous constituents. With respect to the product stream combined as lines 9 and 10, it will be recognized that this product may be further separated, probably after being condensed, to further concentrate the normally liquid hydrocarbons such that they may be fractionated without the difficulties attendant vapor loading of the columns.

The principally normally liquid stream in line 14 is heated to a temperature of about 825° F., and is introduced into vacuum column 15 maintained under a sub-atmospheric pressure of about 50.0 mm. of Hg, absolute. A heavy vacuum gas oil, in an amount of 57,635 lbs./hr., or 118.5 mols/hr. is withdrawn via line 18. Of this amount, 4.4 mols/hr. (2,135 lbs./hr.) or about 3.7% by volume thereof, continues through line 18, being introduced thereby into hot flash zone 12 at a point above mesh blanket 13. The remaining portion, 55,500 lbs./hr., is diverted through line 19 as the HVGO product. The asphaltic material, in an amount of about 29,400 lbs./hr., is removed via line 16. A light vacuum gas oil, in an amount of about 15,000 lbs./hr. is removed via line 17. The vacuum column gas to the jets, not illustrated in the drawing, amounts to about 100 lbs./hr., and comprises the pentane and lighter portion of the material in line 14. The following Table V indicates the component stream analyses for the HVGO recovered as a product (line 19) and the LVGO product (line 17).

TABLE V.—VACUUM COLUMN STREAM ANALYSES

Line No.	17	19
Component, mols/hr.:		
Hexanes, 400° F.	3.5	-----
400° F.-650° F.	35.4	-----
650° F.-1,050° F.	17.3	114.1
1,050° F. plus	-----	-----
Totals	56.2	114.1

Of the 114.1 mols/hr. of HVGO withdrawn via line 19, 12.5% by volume boils in the range of 650° F. to 750° F., 29.0% in the range of 750° F. to 850° F., 30.3% from 850° F. to 950° F., and about 28.2% from 950° F. to 1050° F. Similarly, the 56.2 mols/hr. of LVGO in line 17 comprises 0.5% by volume boiling from hexane to 250° F., 2.3% from 250° F. to 350° F., 3.4% from 350° F. to 450° F., 26.9% from 450° F. to 650° F., 36.1%

from 650° F. to 750° F. and 30.8% from 750° F. to 850° F.

The 3.7% by volume of HVGO which continues through line 18 as wash oil for the mesh blanket 13 has the same component composition given above for the HVGO product. The successful application thereof in keeping the mesh blanket clean is indicated by the fact that the 650° F.-1050° F. portion of the third vaporous phase in line 10 (11.0 mols/hr.) comprises 69.0% by volume of hydrocarbons boiling from 650° F. to 750° F., 25.4% boiling from 750° F. to 850° F., 4.6% from 850° F. to 950° F. and about 1.0% from 950° F. to 1050° F. As indicated in the foregoing Table IV, this stream does not contain hydrocarbons boiling about 1050° F.

The foregoing specification, and particularly the description of the accompanying drawing, indicates the separation process of the present invention, as adapted to black oil processing, and clearly shows the method by which the internally recycled wash oil is prepared.

We claim as our invention:

1. A process for separating a mixed-phase hydrocarbonaceous conversion product effluent containing hydrocarbons boiling above about 1050° F., which process comprises the steps of:

- (a) separating said product effluent in a first separation zone at a temperature above about 700° F. and a pressure above about 1000 p.s.i.g., to provide a first vapor phase and a first liquid phase;
- (b) condensing said first vapor phase and separating the condensed vapors in a second separation zone at a temperature below about 140° F. and a pressure substantially the same as said first separation zone, to provide a hydrogen-rich second vapor phase and a second liquid phase principally comprising propane and heavier normally liquid hydrocarbons;
- (c) introducing at least a portion of said first liquid phase into a third separation zone at a point below a mesh blanket disposed within said third separation zone, at a temperature substantially the same as said first separation zone and at a pressure less than about 200 p.s.i.g., to provide a third vaporous phase and a third liquid phase containing hydrocarbons boiling above 1050° F.;
- (d) further separating said third liquid phase in a fourth separation zone at a temperature above about 700° F. and at a subatmospheric pressure to provide a residuum fraction containing those hydrocarbons boiling above about 1050° F. and a heavy gas oil fraction boiling below about 1050° F.; and,
- (e) introducing at least a portion of said heavy gas oil fraction into said third separation zone at a point above said mesh blanket.

2. The process of claim 1 further characterized in that the portion of said heavy gas oil introduced into said third separation zone is in an amount of from about 1.0% to about 10.0% by volume of said heavy gas oil fraction.

3. The process of claim 1 further characterized in that the temperature of said first separation zone is within the range of from about 700° F. to about 750° F., and the pressure is within the range of from about 1000 p.s.i.g. to about 4000 p.s.i.g.

4. The process of claim 1 further characterized in that said second separation zone functions at a temperature of from about 60° F. to about 140° F., and a pressure of from about 1000 p.s.i.g. to about 4000 p.s.i.g.

5. The process of claim 1 further characterized in that said fourth separation zone functions at a subatmospheric pressure less than 100 mm. of Hg.

No references cited.

HERBERT LEVINE, Primary Examiner.