

Sept. 5, 1961

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2,999,062

SCRUBBING FLUID COKING EFFLUENT

Filed Sept. 12, 1958

2 Sheets-Sheet 1

Fig. 2

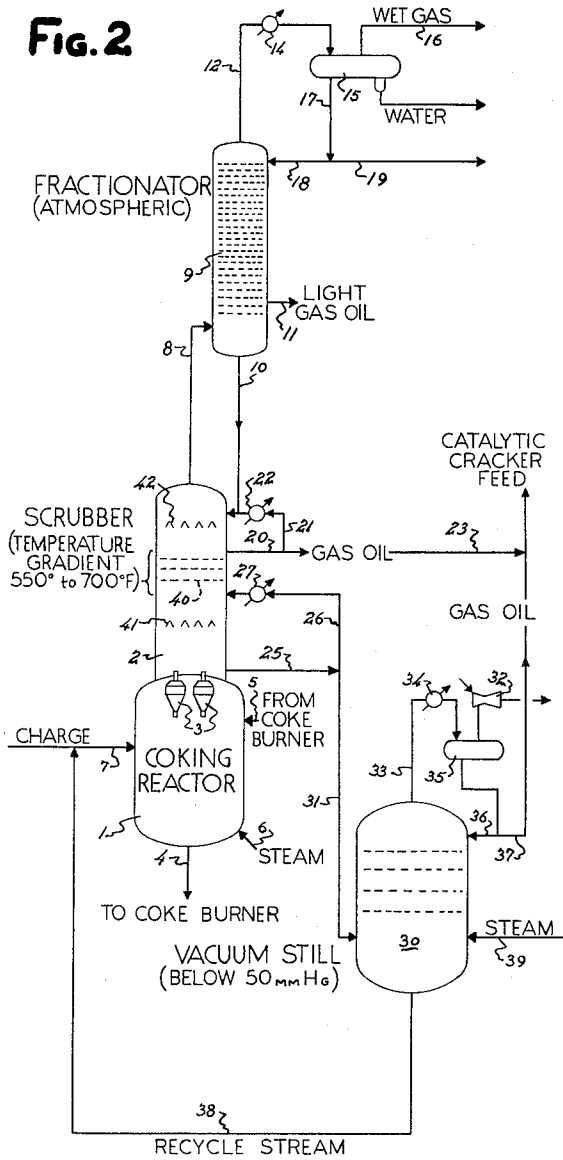
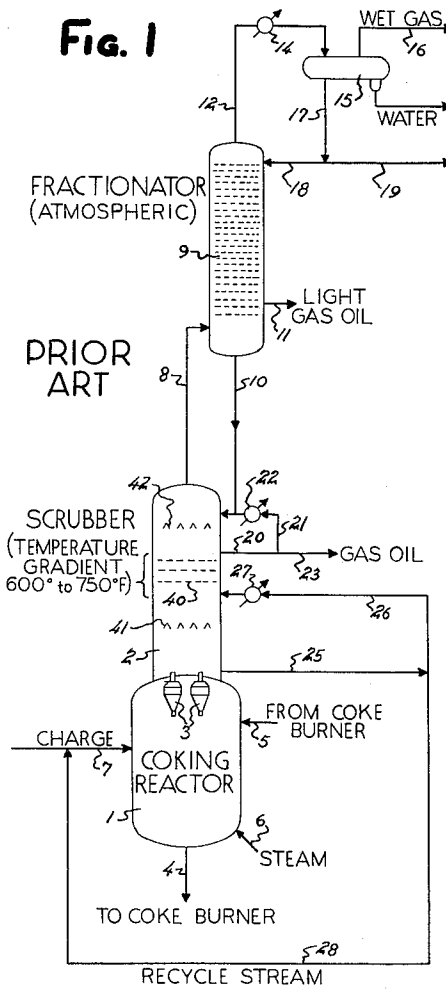


Fig. 1



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2 Sheets-Sheet 2

Fig. 3

RELATIONSHIP BETWEEN RESIDUUM IN HEAVY GAS OIL PRODUCT AND GAS OIL IN RECYCLE STREAM

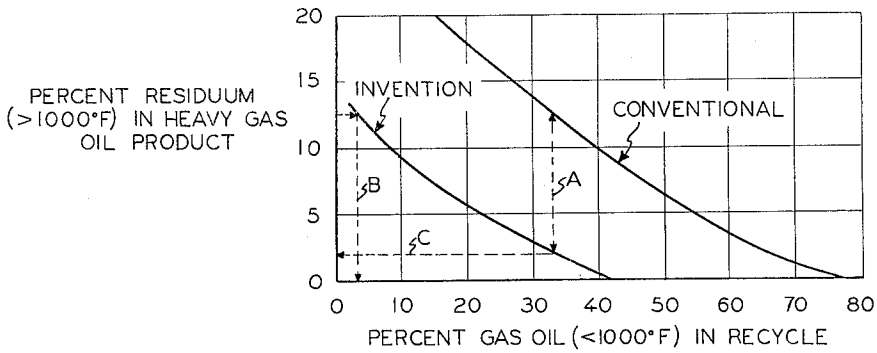
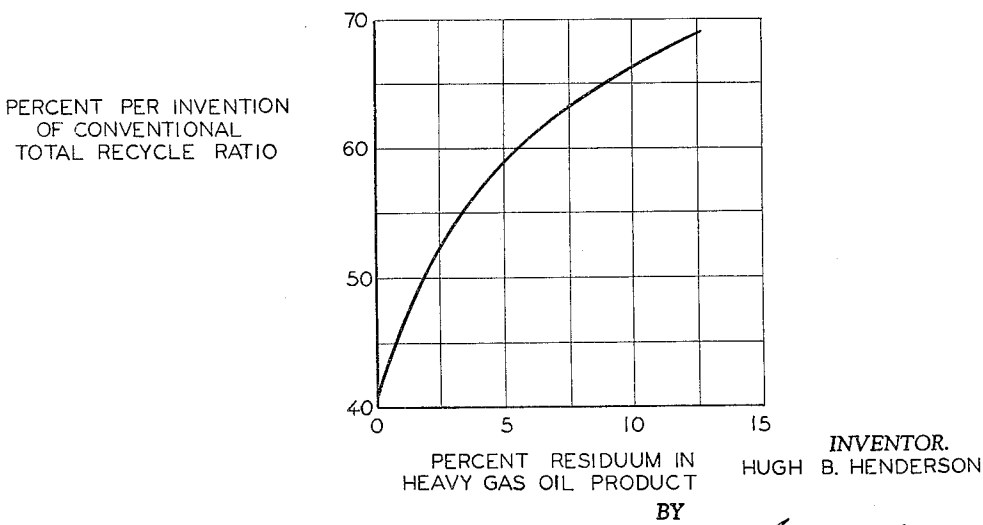


Fig. 4

RELATIONSHIP BETWEEN PER INVENTION TO CONVENTIONAL TOTAL REFLUX RATE AS FUNCTION OF RESIDUUM CONTENT OF HEAVY GAS OIL PRODUCT



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SCRUBBING FLUID COKING EFFLUENT

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Moreover, the conventional process sends a considerable proportion of understroyed residuum into the gas oil stream instead of recycling it to extinction. Residuum has very detrimental effects on catalytic cracking, for it is laden with coke-forming components and a significant amount of heavy metals. The coke-forming components form coke or carbon in the catalytic cracker, reducing the conversion, or gasoline yield of the catalytic cracking plant, since the yield is a function of the carbon that can be burned off the catalyst in a regenerating operation. The heavy metals permanently reduce the value of the catalyst, for they cannot be burned off. Some of them act as dehydrogenation catalysts and affect the distribution of cracked products. They cause the catalytic cracker to make more gas and more coke and less gasoline, and so substantially reduce the capacity of the catalytic cracking plant.

Because of the difficulties caused by the presence of large amounts of residuum in the gas oil stream from the reactor effluent, some refineries have been unable (as a practical matter) to charge this gas oil directly to the catalytic cracker, as was initially the prime object of the fluidized coking process. Instead, the gas oil has had to be processed to eliminate residuum, such as by charging it first to a thermal cracker and then to a fractionator before charging to the catalytic cracker. Such procedure has resulted in a rise in costs.

Attempts to remove residuum from the gas oil have heretofore proven unsatisfactory. When the scrubber was cooled, the amount of residuum in the gas oil stream was reduced somewhat, but as the same time the amount of gas oil in the steam was reduced even more; furthermore, a larger amount of gas oil went back into the recycle stream and was degraded or consumed in the coker. When the scrubber was cooled enough to eliminate substantially all the residuum from the gas oil (i.e., to reduce the residuum content in the gas oil stream below 5%), the gas oil stream itself was reduced by an even greater percentage, and the degradation resulting from recycle of the gas oil was just as harmful as the residuum formerly was.

By the present invention, the recycle rate required to totally destroy the residuum fed to the coker, may be substantially decreased. Simultaneously, the heavy coker gas oil is upgraded and becomes a better charge stock for catalytic cracking units. To do this, the invention calls for (1) removing substantially all of the residuum from the gas oil stream by lowering the temperature in the scrubber and (2) shifting the liquid vapor equilibrium by lowering the hydrocarbon partial pressure, thereby increasing the relative volatility between the gas oil components and the residuum components. This shift may be accomplished by vacuum distillation of all those streams from the reactor effluent containing significant amounts of key components of gas oil and residuum. The distillations is carried out at total pressures below fifty millimeters of mercury absolute pressure and preferably in the range of 10-50 mm. Hg.

In summary then, the present invention removes practically all of the residuum from the gas oil stream by lowering the temperature of the scrubber, even though this means that the gas oil stream obtained directly from the scrubber is reduced. It then subjects the recycle stream to a special vacuum distillation at a pressure below 50 mm. Hg, to produce an overhead and a bottoms stream. The vacuum tower overhead stream contains the heavy gas oils that are desirable as catalytic cracking charge stock, and this overhead stream is not recycled to the reactor and so is saved from degradation; instead it is then added to the direct gas oil stream. The vacuum tower bottoms containing practically all the remaining residuum, are returned to the reactor as recycle stock, so that the

This invention relates to the coking of heavy mineral oils and pitches to produce lighter oils and coke. More particularly, it relates to a novel treatment of residuum found in the effluent resulting from the process of fluidized coking. Still more particularly, it relates to a fluid coking process for decreasing the recycle rate required to totally destroy the residuum charged to the coking process, with a simultaneous upgrading of heavy coker gas oil as a catalytic cracking charge stock; conversely, the recycle rate may be held constant, and an increase in the yield of catalytic cracking charge stock obtained, with a simultaneous upgrading of this stock.

In the so-called fluidized coking process, heavy mineral oils from the refinery (usually containing large amounts of "residuum," which term for the purposes of this specification and the appended claims will be used to denote heavy mineral oil constituents having boiling points above 1000° F.) is sprayed upon heated particles of coke that are maintained in a fluidized condition in a reactor by the passage of steam therethrough. On contact with the heated coke, the residuum is thermally decomposed, or "cracked" (commonly referred to as "coked"), into lighter oils and additional coke, the latter depositing largely as a layer on the original coke particles. A stream of the coke particles is continuously withdrawn from the reaction zone and passed to a burning zone, where some of it is burned to heat the remainder, and heated coke is continuously recirculated to the reactor.

As conventionally practiced, the vapors from the coking zone, consisting of hydrocarbons and steam, are passed to a scrubber wherein the heavier constituents are condensed. The liquid vapor equilibrium conditions in the scrubber are in the range of 4 to 8 p.s.i.a. hydrocarbon partial pressure, with corresponding equilibrium temperatures. From this scrubber, the heaviest constituents are withdrawn and recycle to the coking reactor for further treatment, since they contain a considerable amount of residuum, which it is desired to process to total extinction. A gas oil is also withdrawn from the scrubber, being normally intended for use as charge stock to a catalytic cracker. Still lighter constituents pass through the scrubber as a vapor and enter a fractionator where they are subsequently condensed and removed as liquid and gaseous products, some of the liquid also being suitable as charge stock for the catalytic cracker.

The present invention relates to processing the constituents removed as liquid in the scrubber, particularly those heretofore sent to the recycle stream.

Conventionally, the liquid stream returned to the reactor as recycle or spillback has included a considerable amount of gas oil that would be suitable as charge stock for a catalytic cracking unit. Returning this gas oil to the reactor, along with the residuum, has resulted in its degradation as a catalytic cracking charge stock, since additional residence time at high temperature results in dehydrogenation, thermal cracking, and other reactions considered unfavorable as compared to catalytic cracking. Bearing in mind the fact that one of the prime objects of coking is to obtain large amounts of suitable catalytic cracking charge stock, it will be seen that conventional recycle practice lowers the quality of the catalytic cracker charge stock by recycling this gas oil, and that some of the recycled gas oil is consumed in the coker on recycle, resulting in a loss of high quality charge stock.

residuum can be almost totally destroyed, ending up either as cracked gas oil, gas, or coke.

By processing in accordance with the invention, the following advantages are obtained:

(1) A superior catalytic cracking charge stock is produced, for the gas oil stream (a) contains a very low percentage, if any, of high-boiling (residuum) constituents, tending to form coke on the catalyst and so reducing the gasoline yield of the catalytic cracker; (b) contains a lower percentage of heavy metals, tending to permanently affect the distribution of cracked products from the cracker; (c) contains much less gas oil that is degraded by recycling it to the coker and its high-temperature conditions.

(2) There is less gas oil in the recycle stream; so the recycle rate for destroying a given percentage of residuum is lowered.

(3) No separate heat source (requiring other fuel) is needed for the vacuum distillation of this invention, for the heat required may be furnished in the coking reactor by the by-product coke and transferred as sensible heat in the feed to the vacuum tower.

The invention may be more readily understood by reference to the drawing wherein:

FIGURE 1 is a diagrammatic flow sheet of a portion of prior-art, conventional, fluid coker.

FIGURE 2 is a diagrammatic flow sheet of a corresponding portion of a fluid coker operated in accordance with the invention.

FIGURE 3 is a graph showing the relationship between the percent of residuum in the gas oil product stream and the percent of gas oil in the recycle stream, in both the conventional process and the process of this invention.

FIGURE 4 is a graph showing the relationship in the present invention between the percentage of the conventional recycle rate used and the percentage of the residuum in the heavy gas oil product.

Referring to FIGURE 1, a coking reactor 1 is connected to a scrubber 2 by cyclones 3 in the conventional manner. Conduits 4 and 5 respectively lead to and from a coke burner (not shown). The coke is maintained in a fluidized condition by steam 6 being introduced at various locations. Residuum is charged to the reactor 1 through line 7. In the reactor 1 it contacts the hot fluidized coke and, as a result, some of it is converted to coke, while much more of it is cracked and passes with the effluent through the cyclones 3 into the scrubber 2.

In the scrubber 2, preliminary fractionation takes place at a temperature gradient of about 600°-750° F., with the following effects:

(a) Uncondensed vapors pass up through line 8 into a fractionator 9, from which various fractions are withdrawn at atmospheric pressure, at lines 10, 11 and 12, for example. The lightest stream, line 12, is cooled by a heat-exchanger 14 and passes through a reflux accumulator 15. There the remaining stream (wet gas) passes through line 16, while raw distillate leaves through line 17, part being used as reflux through line 18, the remainder being taken off as product through line 19. A heavier fraction may be passed to the catalytic cracker through line 11, while a still heavier fraction is returned through line 10 to serve as scrubbing oil.

(b) Heavy gas oil passes out from the scrubber 2 through line 20. Conventionally, some of this material is withdrawn into a sidestream 21, cooled by a heat exchange 22 and used (together with cool bottoms from the fractionator 9 through line 10) as scrubbing oil. The remainder of the gas oil is sent out through a line 23 for use as desired, the ideal being to send it to a catalytic cracking unit (not shown), if the gas oil is really suitable for catalytic cracking. Usually, however, it is not suitable without further processing.

(c) Recycle stock, the heaviest of all, passes out from the scrubber 2 through line 25. Some of this is passed by line 26 and cooling heat exchanger 27 back to the scrubber

2 for use as a quenching oil; the remainder is recycled by line 28 to the input line 7 of the coking reactor 1.

The purpose of recycling through line 28 is to crack the residuum to extinction. Corresponding to the physical equilibrium conditions, this recycled residuum contains a proportionate amount of gas oil. This concentration of gas oil is a function of the liquid-to-vapor ratio, the number of equilibrium contacts, and the equilibrium temperature and pressure. The return of this gas oil to the reactor 1 results in its degradation, as previously outlined. If the recycle rate is increased by lowering the end point of the gas oil, in order to decrease the amount of residuum in the gas oil stream 23, the concentration of gas oil in the recycle line 28 increases at a rate corresponding to the adjusted equilibrium temperature. The result is that the recycle rate of actual residuum increases asymptotically as compared to the total recycle rate. The corresponding increase in recycle of gas oil, which is exponential in magnitude as compared to the total recycle rate, causes degradation of this stock as a catalytic cracking charge stock, since the recycle rate of gas oil contained in the total recycle stream 28 increases faster than the recycle rate of the residuum.

The present invention, illustrated in FIGURE 2, provides a vacuum tower or still 30 fed from a stream 31 containing the bottoms withdrawn from the scrubber 2 through the line 25. Substantially all the residuum material is forced into line 25 by adjustment of the scrubber temperature gradient to about 550° to 700° F. by regulation of the amount of reflux in the streams 21 and 27. By this means, the amount of residuum remaining in the gas oil stream 23 is reduced to between 0% and 5%, normally to between 1% and 3%.

A steam jet ejector 32 may be used to provide the necessary vacuum conditions (i.e., a pressure below 50 mm. of Hg) in the tower 30, which contains conventional liquid-vapor contacting devices. The overhead from the tower 30 thus passes through line 33 and overhead condenser 34 to a reflux accumulator 35. Steam is introduced to the vacuum tower 30 through line 39 to reduce hydrocarbon partial pressure and lower temperatures, resulting in more favorable relative volatilities of the components to be separated. Reflux from accumulator 35 is re-introduced to vacuum tower 30 through line 36 to obtain more favorable separation. After withdrawal of the reflux portion through the line 36, overhead gas oil is passed through line 37. This gas oil may be introduced into the line 23 for use as charging stock for the catalytic cracker (not shown). The bottoms from the tower 30 are recycled to the coking reactor 1 through lines 38 and 7.

Thus, in this invention, the approximately 50° F. drop in temperature in the scrubber 2 substantially reduces the volume of the gas oil stream 20 to force substantially all of the residuum therein into the line 25. This means that much additional gas oil is forced into the line 25, but (after withdrawal of quenching oil as usual through the line 26), the stream of gas oil and residuum is sent by the line 31 to the vacuum still 30. The heat needed in the still 30 is carried there by the stream itself from the scrubber 2 through lines 25 and 31. Initially, this heat is produced in the coker burner (not shown) by a very small increase in the amount of coke burned therein, is transmitted to the reactor 1 by the line 5, and is then transferred to the effluent. So there is little cost for this heat and no separate heating installation is used.

Within the still 30, the vacuum conditions, usually are 10-50 mm. Hg. The gas oil is fractionated off as vapor through the line 33, while the bottoms and recycle line 38 contains nearly all the residuum. The reflux of some of the gas oil into the still 30 by line 36 does not harm it, and maintains better conditions in the still 30.

As a result, the gas oil passing through line 37 is almost 100% residuum-free, and the net residuum in line 23 after the stream from line 32 enters it, is reduced

below 5%, usually between 1% and 3%, but depending in part upon the composition of the original residuum of line 7. Coke and heavy metals are no longer a substantial problem in the catalytic cracking unit, and the gas oil in lines 23 and 37 may be charged directly to the catalytic cracker without further treatment to reduce the residuum content. Furthermore, the residuum can be cracked (coked) to extinction, while the quality of the gas oil in lines 23 and 37 is upgraded so that it can be directly charged to a catalytic cracker.

Two specific examples are presented, one in which the recycle rate is raised to destroy substantially all the residuum and one which the conventional recycle rate is used.

EXAMPLE 1

The feed stock or residuum having the properties shown in Table I is introduced at line 7 into the coking reactor 1.

Table I.—Properties of feed stock residuum charged to coking reactor

API gravity	6.9
Conradson carbon, wt. percent	17.5
Sulfur, wt. percent	3.95
A.S.T.M. distillation (D-1160), ° F.:	
IBP	529
5%	828
10%	909
20%	975
30%	1019
40%	1061

The conditions in the coking reactor 1 are shown in Table II.

Table II.—Operating conditions in coking reactor

A. CONVENTIONAL

Reactor temperature:	
Dense phase, ° F	955
Dilute phase, ° F	980
Cyclones, ° F	1010
Total reactor steam, wt. percent of fresh feed	10.1
Scrubber temperature gradient, ° F	600-750

B. PER INVENTION

All factors same as above, except:
Scrubber temperature gradient, ° F 550-700

To be more specific, at the very bottom of the scrubber 2 the vapor enters from the cyclone 3 at about 1000° F. Vapor leaves the scrubber 2 through line 8 at about 550° F. The present invention is concerned with contacting apparatus 40, which may be trays, as shown, or shed decks, in the central part of the scrubber 2, i.e., lying between the gas oil line 20 and the quenching oil reflux line 26. The apparatus 40 may be termed the effective fractionating section or zone of the scrubber 2. The lower contacting trays or sheds 41 are primarily for the purpose of heat exchange between the quench oil and the hot vapors from the cyclones 3. Similarly, the upper contacting sheds or trays 42 are primarily for the purpose of heat exchange between the scrubbing oil and the vapors arising from the effective fractionating zone 40.

When the scrubber temperature gradient is spoken of in either the specification or claims, the gradient in the effective fractionating zone 40 is meant. The conventional scrubber temperature gradient is 600° F. to 750° F. In other words, in the conventional process, the temperature at the bottom of the zone 40 is about 750° F., and the temperature at the top of the zone 40 is about 600° F.

In the present example, as shown in Table II, part B, the temperature gradient is 550° F. to 700° F., which means that it is about 700° F. at the bottom of the zone 40 and about 550° F. at the top. This forces much more material into the line 25, including practically all the residuum, as well as more gas oil than usual. However, the use of the vacuum still 30 achieves further separation of gas oil from residuum. The still 30 in this example is operated at about 25 mm. Hg.

Table III contrasts conventional yields against those of this example of the present invention:

Table III.—Fluid coking yields

	Conventional		Per Invention	
	B.p.s.d.	Vol. percent	B.p.s.d.	Vol. percent
5				
10				
Fresh Feed (line 7):				
650-1,000° F.	6,348	27.0	6,348	27.0
above 1,000° F.	21,252	73.0	21,252	73.0
Total charge	27,600	100.0	27,600	100.0
Recycle (line 38):				
650-1,000° F.	3,520	33.1	6,132	40.5
above 1,000° F.	7,120	66.9	8,973	59.5
Total	10,640	100.0	15,105	100.0
15				
Heavy Gas Oil Product (line 23):				
650-1,000° F.	9,243	87.2	7,900	99.6
above 1,000° F. (Residuum)	1,360	12.8	32	0.4
Total	10,603	100.0	7,932	100.0
20				
Heavy Gas Oil Product, 90% ASTM D-1160:				
Distillation Temperature, ° F	980		875	
25				

While there appears to be a reduction in the heavy gas oil product, it should be noted that it contains only 0.4% residuum, whereas the conventional gas oil contained 12.8% residuum and could not economically be charged directly to the catalytic cracking unit. The product of this invention makes an excellent charge stock. In fact, it is better than the bare table indicates because more of the gas oil near 1000° F. is sent to recycle and less of that near 650° F. is sent there. But the most important fact may well be that there is no need to give any special treatment to the 7,932 barrels per day of heavy gas oil product before sending it directly to the catalytic cracker, whereas, some treatment other than, or prior to, catalytic cracking must be given to the 10,603 barrels per day of conventional heavy gas oil product.

The residuum content of the heavy gas oil product has been reduced by 1,328 barrels out of 1,360 or about 97.6%.

EXAMPLE 2

With the feed and the conditions in the reactor 1 and still 30 the same as in Example 1, but with the temperature gradient of the zone 40 changed to about 560° F. to 710° F. in order to provide a recycle rate in line 38 the same as in line 28 of FIG. 1 (as shown in Table III) the present invention still obtains substantial reduction of the amount of residuum in the gas oil line. Table IV shows the results.

Table IV.—Fluid coking yields where recycle rate equals conventional recycle rate

	B.p.s.d.	Vol. Percent
60		
Fresh Feed (Line 7):		
650-1,000° F.	6,348	27.0
above 1,000° F.	21,252	73.0
Total	27,600	100.0
65		
Recycle (Line 38):		
650-1,000° F.	2,766	26.0
above 1,000° F.	7,374	74.0
Total	10,640	100.0
70		
Heavy Gas Oil Product (Lines 23 and 37):		
650-1,000° F.	9,507	95.5
above 1,000° F.	448	4.0
Total	9,955	100.0
Heavy Gas Oil Product, 90% ASTM D-1160:		
Distillation Temperature, ° F	910	
75		

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Here, the residuum content in the gas oil has been reduced to 4.0%, well below the 5% limit above which it becomes impractical to charge the gas oil directly to a catalytic cracking unit.

Moreover, it will be noted that the 10,640 barrels per day of recycle (the same total amount as in the conventional process) is 74% residuum and only 26% gas oil, whereas in the conventional process, 33.1% of the recycle is gas oil. In actual barrels, this example shows that by following the present process of reducing the temperature in the scrubber and then vacuum distilling the normally-recycled product, the invention sends 754 more barrels of residuum and that much less of gas oil to recycle.

So far as the heavy gas oil product is concerned, it contains 912 barrels less of residuum, a reduction of about 67%. This reduction lowers the content below the critical limit, so that the product can economically be charged to the catalytic cracker.

EXAMPLE 3

Substantially the same results as those given in Example 1 are obtained by leaving the temperature gradient as in Table IIA (Conventional) and raising the hydrocarbon partial pressure. This may be done in various ways. One way is to raise the total pressure in the scrubber. In this example, the scrubber pressure is raised from 5 p.s.i.g. to 12 p.s.i.g. The hydrocarbon partial pressure of about 8 p.s.i.a. in Example 1 is thereby raised to about 12 p.s.i.a.

The results of this example are substantially identical with those of Example 1.

EXAMPLE 4

Another way of raising the hydrocarbon partial pressure is to change the steam content of the effluent. Results substantially identical to those of Example 1 are obtained by lowering the steam content to 5%, expressed as weight percent of fresh feed. The temperature and pressure conditions otherwise remain as in Table IIA.

FIGS. 3 and 4 may help in understanding the significance of this invention. FIG. 3 shows two curves, one resulting from practicing the conventional process of FIG. 1, the other from practicing the invention as in FIG. 2. In both curves the percent of the residuum in the gas oil product is plotted against the percent of the gas oil in the recycle stream. Note that the conventional recycle stream would contain 75% gas oil if the residuum content of the heavy gas oil product were to be reduced to the same level as that attained by this invention in Example 1, where the recycle stream contains only 40.5% gas oil.

Since the total recycle rate is proportional to

$$100$$

$$(100 - \text{percent of gas oil in the recycle stream})$$

It is readily seen that the total recycle rate obtained by this invention is less than half the conventional recycle rate. This fact is illustrated in FIG. 4, where the recycle rate is plotted against the amount of residuum in the heavy gas oil product.

Conversely, the percentage of gas oil in the recycle stream, when using the present invention to produce a heavy gas oil product containing the same amount of residuum as that in the conventional process, drops from 33% to 3%, as shown in FIG. 3, lines A and B. This is equivalent to a 69% reduction in the total recycle rate. Moreover, as FIG. 4 shows, the lower the residuum content of the heavy gas oil product desired, the greater the contrast between the results of the present invention and those of the conventional process.

Similarly, by referring to lines A and C, one can see the

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importance of the invention. The drop in residuum is huge, while the gas oil recycled remains the same.

To those skilled in the art to which this invention relates, many changes in constructions and widely differing embodiments and applications of the invention will suggest themselves without departing from the spirit and scope of the invention. The disclosures and the description herein are purely illustrative and are not intended to be in any sense limiting.

As stated hereinbefore, the term "residuum" is used herein and in the claims to denote those constituents of hydrocarbon oils which have boiling points above 1000° F. at atmosphere pressure. These constituents are generally of an asphaltic or resinous nature and, in ordinary distillation, will crack before they distill. Under the partial pressure conditions obtained in a fluid coker, however, a considerable portion of these constituents vaporize and appear as components of the vapor effluent.

I claim:

1. In a fluid coking process wherein a charge of high-boiling constituents is coked and the effluent is scrubbed to separate out a gas-oil stream normally containing objectionable amounts of residuum, and a heavy stream, normally recycled, the improvement comprising introducing the effluent into the lower end of a scrubbing zone having a temperature gradient lying between temperatures low enough to substantially free said gas-oil stream from residuum, said gas-oil stream leaving from above said zone, and to drive some gas-oil and substantially all of said residuum down into said heavy stream, which leaves from below said zone; distilling said heavy stream in a distillation zone at substantially its withdrawn temperature by means of its heat content when withdrawn and at a pressure below 50 mm. Hg while simultaneously introducing steam in a quantity sufficient to further reduce the hydrocarbon partial pressure and lower the temperature gradient in the distillation zone, thus making additional sensible heat available, using said additional heat to effect an adequate reflux of overhead distillate, thereby obtaining a substantially residuum free gas-oil stream as overhead product and a bottoms stream containing a minimum of gas-oil; and charging said bottoms stream back as part of said coking charge.

2. In a fluid coking process wherein a charge of high-boiling constituents is coked and the effluent is introduced directly into the bottom of a scrubbing zone and scrubbed to separate out a gas-oil stream containing objectionable amounts of residuum and a heavy stream, normally recycled, the improvement comprising scrubbing at a temperature gradient of about 700° F. in the bottom of the scrubbing zone to about 550° F. at the top of said zone, to provide a hydrocarbon partial pressure sufficiently high to substantially free said gas-oil stream from residuum and to drive some of said gas-oil and substantially all of said residuum into said heavy stream, distilling said heavy stream at substantially its withdrawn temperature and in the substantial absence of added heat, utilizing its own heat content at a pressure below 50 mm. Hg to obtain a substantially residuum-free gas-oil stream as overhead, and a bottoms stream; and charging said bottoms stream back to said coking reactor.

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