

- [54] **UPGRADING CRUDE OIL BY COMBINATION PROCESSING** 3,671,419 6/1972 Ireland et al..... 208/57
- 3,714,023 1/1973 Stine..... 208/66
- 3,748,251 7/1973 Demmel et al. .... 208/120
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[63] Continuation of Ser. No. 363,705, May 24, 1973, abandoned.

[52] **U.S. Cl.** ..... 208/93; 208/66; 208/80; 208/120

[51] **Int. Cl.<sup>2</sup>** ..... **C10G 37/10**

[58] **Field of Search** ..... 208/66, 120, 93, 153, 80

[56] **References Cited**

**UNITED STATES PATENTS**

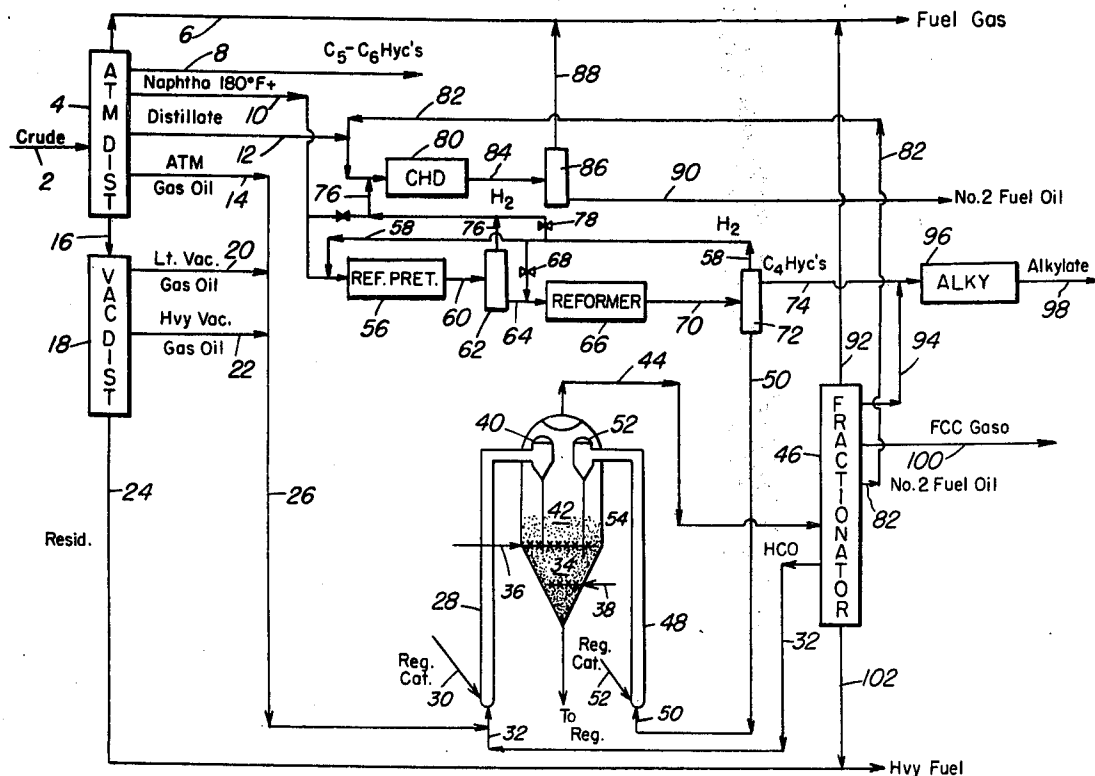
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[57] **ABSTRACT**

The combination processing concept described involves using a low severity reforming operation primarily as a source of hydrogen for CHD processing and other treating operations in the production of low octane reformate (90–95 R+O) which is then upgraded by cracking over a crystalline aluminosilicate catalyst to produce a higher octane gasoline boiling product. Alkylation of reformer and FCC light ends contributes to gasoline boiling product yields.

**8 Claims, 1 Drawing Figure**





## UPGRADING CRUDE OIL BY COMBINATION PROCESSING

This application is a continuation of application Ser. No. 363,705, filed May 24, 1973 now abandoned.

### BACKGROUND OF THE INVENTION

The upgrading of crude oil to more desirable products including gasoline by combination processing is known in the art. In many of these processes the combination of alkylation, catalytic cracking, reforming, hydroprocessing, coking, hydrocracking, isomerization and polymerization have all been used in more than one different arrangement in an attempt to improve upon the yields and products obtained from a given crude charge. Patents typical of the above processing combinations are those of Schreiner Jr. et al U.S. Pat. Nos. 3,019,180; Gould et al 3,409,540; and Ireland et al 3,671,419. The processing combination of the present invention encompasses some of the individual steps of these prior art patents in a new arrangement directed to producing an overall refinery product slate including a high octane gasoline product obtained by catalytic cracking of a low severity reformat product.

### SUMMARY OF THE INVENTION

The present invention is concerned with a process combination for upgrading a crude oil charge to fuel oils and gasoline product. In one aspect, the present invention relies upon a combination of distillation steps for the production of hydrocarbon charge materials suitable for CHD treating, reforming, alkylation and fluid catalytic cracking in which combination the reforming operation is relied upon primarily as a source of high purity hydrogen and low severity reformat product. The reformat product is upgraded by a crystalline zeolite fluid cracking catalyst in a riser cracking unit in parallel arrangement with a gas oil riser cracking unit sharing a common regenerator. Light virgin naphtha hydrocarbons boiling in the range of C<sub>5</sub> and C<sub>6</sub> hydrocarbons may also be upgraded to more desirable products in a separate riser fluid cracking unit or this light naphtha material may be combined with a gas oil charge material converted in a fluid catalyst riser cracking unit. For example, the light naphtha comprising C<sub>5</sub> and C<sub>6</sub> hydrocarbons may initially contact regenerated catalyst in a bottom portion of the riser to form a suspension at an elevated temperature of at least 1000°F. and thereafter in a downstream portion of the riser the gas oil charge is brought in contact with the suspension at elevated temperature cracking conditions of at least 1000°F. However, under no circumstances should the light virgin naphtha (C<sub>5</sub> - C<sub>6</sub> hydrocarbons) be combined with the low severity reformat converted in a separate riser cracking unit employing a crystalline aluminosilicate (CAS) conversion catalyst. On the other hand, a portion of the gasoline product of cracking may be re-cracked in the presence of the reformat charge to improve it further in boiling range and octane rating. The catalyst used in the fluid cracking steps of the process combination may be a Y type of crystalline zeolite cracking catalyst alone or in admixture with a ZSM-5 type of crystalline aluminosilicate.

The processing concepts of the present invention are particularly amenable to modern-day low coke producing crystalline aluminosilicate catalyst compositions and such catalyst may be used to advantage in both the hydrocarbon conversion operation of the process and

the catalyst regeneration operation by developing a greater accumulation of carbonaceous deposits on the total mass of catalyst than heretofore obtained before regeneration thereof. The reasons for this observed phenomenon is not readily explained and most unexpected. Furthermore, it has been found that the concepts going to the very essence of the present invention are applicable to other zeolite catalyst mixtures and particularly those comprising a mixture of a crystalline faujasite cracking component with a ZSM-5 type of crystalline material.

The catalyst mixture and/or compositions suitable for use in this invention comprise a mixture of small pore and large pore crystalline aluminosilicate in combination with one another as separate discrete particles and these may be composited from substantially any high activity large pore crystalline zeolite cracking component in admixture with, for example, a ZSM-5 type of catalyst composition. The ZSM-5 type catalyst composition is a relatively small average pore diameter material smaller than, for example, a rare earth exchanged X or Y crystalline zeolite.

The large and small pore crystalline zeolites above discussed may be dispersed within a separate or a common matrix material suitable for encountering relatively high temperatures contemplated in the fluid cracking operation of this invention with its attendant catalyst regeneration operation. The catalyst mixture or composition contemplated for use in this invention will catalyze the conversion of the various components comprising the hydrocarbon feed including normal paraffins to produce for example gasoline as well as LPG types of gaseous materials. Thus the catalysts suitable for this invention have activity for cracking several different kinds and types of hydrocarbons found in gas oil boiling range materials in combination with a very selective cracking of normal paraffins and singly branched hydrocarbons which are restructured and/or upgraded to desired higher boiling components.

The novel process combination of this invention using a catalyst system comprising a mixture of separate catalyst particles or a homogeneous composition of one or more crystalline zeolite components dispersed in an amorphous matrix material wherein the zeolite component or components acts substantially independently as herein defined upon given hydrocarbon components and each catalyst component is relied upon substantially to support the function of the other. Thus it is contemplated employing in the catalyst system of this invention, a large pore crystalline aluminosilicate having a pore size in excess of about 9 Angstroms as a major component with the minor component being a small pore crystalline component having a maximum pore size not exceeding about 9 Angstroms and preferably being less than about 7 Angstroms. On the other hand, the large and small pore zeolites may be used in substantially equal amounts or the smaller pore crystalline zeolite may be in a minor or major proportion. On the other hand, either crystalline zeolite component may be used alone and dispersed in a suitable matrix material as herein defined. The small pore crystalline zeolite is preferably a ZSM-5 type of crystalline material such as that described in U.S. Pat. 3,702,886, issued Nov. 14, 1972 or copending application Ser. No. 865,418 filed Oct. 10, 1969, now abandoned. The large pore crystalline zeolite may be any of the now known crystalline aluminosilicates which are suitable for cracking hydrocarbons and providing a pore size in

excess of 8 Angstroms. Such a composition has the structure and capability to act upon substantially all the components usually found in a gas oil feed boiling in the range of 500°F. up to 950° or 1100°F. Large pore zeolites of this type are well known and include materials or synthetic faujasite of both the X and Y type as well as zeolite L. Of these materials zeolite Y is particularly preferred.

The crystalline zeolites above identified may be exchanged, combined, dispersed or otherwise intimately admixed with a porous matrix. By porous matrix it is intended to include inorganic and organic compositions with which the crystalline aluminosilicates may be affixed. The matrix may be active or substantially inactive to the hydrocarbon conversion reactions encountered. The preferred porous matrix may be selected from the group comprising inorganic oxides such as clay, acid treated clay, silica-alumina etc. A more complete description of a catalyst composition comprising ZSM-5 type materials which may be used with advantage in this invention and their method of preparation may be found in the application and patent above identified.

Hydrocarbon charge stocks which may be converted by the combination and method of this invention comprise petroleum fractions having an initial boiling point of at least 400°F. and an end point of at least 600°F. and as high as 950° to 1100°F. The present invention also contemplates the cracking of naphtha boiling hydrocarbons such as a virgin naphtha boiling in the range of C<sub>5</sub> hydrocarbons up to about 400°F. end point. A preferred naphtha charge for the combination of the present invention is one having an initial boiling point of about 180°F. and an end point as high as about 440°F. to improve its octane rating in combination with producing significant quantities of LPG type material and/or feed material suitable for alkylation reaction. Hydrocarbons boiling above about 400°F. include gas oils, residual oils, cycle stocks, whole topped crudes and heavy hydrocarbon fractions derived by destructive hydrogenation processes. These feeds may be used alone or in combination with one another as herein provided.

The present invention is directed particularly to a combination operation for the production of a relatively high octane gasoline product which particularly eliminates the need for high cost, high severity reforming operations and their related operating problems. Thus the present invention is particularly concerned with eliminating reforming operations providing a product in excess of about 90 or 98 R+O gasoline boiling material. Accordingly a third reactor stage of a reforming sequence wherein primarily hydrocracking reactions occur may be eliminated by this invention. In addition the processing combination of this invention permits relatively large savings in light ends processing units and offsite facilities by integrating the reformer and a fluid catalytic cracking (FCC) unit light ends recovery in the production of a high quality naphtha product. Furthermore, a low severity reforming operation substantially improves hydrogen product purity which can be used with particular advantage and reformers thus operated also become much cheaper in the recycle gas recovery system since the hydrogen purity is already very high. A further significant advantage which may be realized by the present invention is in the use of the least expensive reforming catalyst for the low severity, primarily dehydrogenation reforming

operation in which one or two reactors may be used. That is, only a maximum of two reactor stages need to be employed generally to accomplish dehydrogenation of naphthenes in the feed under relatively low severity operating conditions such as those required to provide a low octane reformat in the range of (90-95 R+O). On the other hand, it is contemplated employing only one reactor stage in the reforming operation to effect dehydrogenation of naphthenes.

#### BRIEF DESCRIPTION OF THE DRAWING

The drawing shows a combination of refinery processing steps arranged to provide a low cost operation by integrating a low severity reforming operation with a high severity fluid catalytic cracking operation, a catalytic hydrodesulfurization operation and an alkylation operation connected through appropriate interrelated distillation steps.

#### DISCUSSION OF SPECIFIC EMBODIMENTS

The drawing depicting the processing combination of the present invention shows the production of three gasoline streams for use in gasoline blending facilities not shown. These gasoline streams include a light virgin naphtha, (FCC) fluid catalytic cracking gasoline product of gas oil cracking and FCC upgraded naphtha reformat in combination with alkylate product. It is contemplated subjecting the light virgin naphtha stream comprising primarily C<sub>5</sub> and C<sub>6</sub> hydrocarbons to a high temperature contact with the zeolite cracking catalyst in a separate riser reactor or with the gas oil feed of the fluid cracking unit. Such conversion steps are particularly attractive when employing a catalyst mixture comprising a Y faujasite crystalline aluminosilicate in combination with a ZSM-5 type of crystalline aluminosilicate described above. In the processing arrangement of the present invention, the distillation steps, product octane incremental improvement, chemical makeup and volumetric production can be largely controlled by adjusting operating variables available in particularly the fluid cracking operation thereby greatly reducing refinery tankage, processing vessels and interconnecting piping requirements.

Referring now to the drawing, crude oil boiling in the range of about 100° to about 1400°F. is introduced by conduit 2 to an atmospheric distillation step 4 of the processing combination of this invention. In atmospheric distillation step 4, the crude oil is separated to recover fuel gas from an upper portion by conduit 6, a light virgin naphtha comprising primarily C<sub>5</sub> and C<sub>6</sub> hydrocarbons by conduit 8, a virgin naphtha fraction provided with an initial boiling point of about 180°F. being withdrawn by conduit 10, a distillate fraction boiling in the range of from about 400°F. up to about 650°F. being withdrawn by conduit 12, and an atmospheric gas oil boiling above about 600°F. being withdrawn by conduit 14. Residual material boiling above about 600°F. is withdrawn from the bottom by conduit 16 and passed to a vacuum distillation step 18. In vacuum distillation step 18, a light and a heavy vacuum gas oil are separately recovered by conduits 20 and 22 respectively as shown. Residual material is withdrawn from the bottom by conduit 24 for use as heavy fuel material.

The gas oil fractions obtained from distillation steps 4 and 18 by conduits 14, 20 and 22 are combined and conveyed by conduit 26 to the inlet of riser reactor 28 of a fluid catalyst cracking unit. Hot regenerated cata-

lyst is introduced to the lower portion of riser 28 by conduit 30 in an amount sufficient to form a catalyst/oil suspension with the gas oil feed providing a cracking temperature of at least about 1000°F. and more usually 1050° or 1100°F. A heavy recycle oil product of catalytic cracking recovered from the product fractionator may be introduced by conduit 32 to a bottom portion or an upper portion of riser 28 wherein it is subjected to re cracking.

Although not specifically shown, the light virgin naphtha comprising C<sub>5</sub> and C<sub>6</sub> hydrocarbons in conduit 8 may be introduced separately to the bottom of the riser to effect a very high temperature conversion at high catalyst/oil ratio but with short contact time before combined with the hydrocarbon feed comprising gas oil passed to riser 28. The light virgin naphtha may be combined with the gas oil feed or it may be passed to a separate riser reactor. On the other hand, the light virgin naphtha may be converted in a bed of catalyst 34 separated from the riser reactors of the fluid cracking unit and introduced by conduit 36 above the stripping gas inlet conduit 38.

The catalyst-hydrocarbon suspension formed and passed upwardly through riser reactor 28 is retained in the riser to provide a gas oil hydrocarbon residence time within the range of 1 to about 8 seconds before separation of catalyst particles from hydrocarbon vapors by cyclonic means represented by separator 40. Separated catalyst particles at an elevated temperature usually in excess of about 900°F. and as high as about 1000°F. under some operating conditions are conveyed by the cyclone dipleg 42 to the catalyst bed 34. In the event higher temperatures are desired in catalyst bed 34, hot regenerated catalyst may be added thereto. Separated hydrocarbon vapors are removed from separator 40 by confined passageways communicating with product withdrawal conduit 44. Conduit 44 conveys the hydrocarbon products of fluid cracking to a product fractionation step 46.

In a parallel riser reactor 48, a low severity reformat product obtained as herein described in a restricted reforming operation is upgraded by contact with the fluid cracking crystalline aluminosilicate conversion catalyst suspended therein at a temperature of at least 1000°F. under conditions providing a hydrocarbon residence time in the range of 1 to about 8 seconds. Hydrocarbon residence time in the range of 2 to 4 seconds may be particularly employed at higher temperature conversion conditions of about 1100°F. The low severity reformat feed of about 90-95 R+O octane rating alone or in combination with gasoline product of gas oil cracking and obtained as herein defined is introduced to the bottom portion of riser 48 by conduit 50. Hot regenerated catalyst is supplied to the bottom portion of riser 48 by conduit 52 in an amount sufficient to provide a desired catalyst/oil ratio suspension at a temperature of at least 1000°F. for passage upwardly through riser 48. The suspension is separated by cyclonic means 52 at the end of riser 48 into a hydrocarbon phase and a catalyst phase. The catalyst phase is passed by dipleg 54 to the bed of catalyst 34 with the hydrocarbon phase removed by confined passageways communicating with withdrawal conduit 44.

The naphtha fraction withdrawn by conduit 10 from distillation step 4 is conveyed to a naphtha hydrogenation or pretreating operation 56 wherein the naphtha is contacted with recycled high purity hydrogen product of the low severity reforming operation and recycled by

conduit 58. In the naphtha pretreater 56 sulfur and nitrogen compounds are converted so that upon subsequent separation they may be removed overhead with gaseous material comprising hydrogen not consumed during the naphtha pretreat operation. The pretreated naphtha reduced to a sulfur level of at least 20 ppm and a nitrogen level of at least 10 ppm but more usually to less than 5 ppm is conveyed by conduit 60 to a separation step represented by separator 62. The pretreated naphtha separated from sulfur and nitrogen compounds as by separation step 62 is conveyed by conduit 64 to a platinum catalyst reforming operation 66. Hydrogen product of reforming separated from reformat material in a separation step represented by separator 72 is withdrawn by conduit 58 for recycle by interconnecting conduit 68 to the reformer 66. The product effluent of low severity reforming conveyed by conduit 70 to the separation step represented by separator 72 also provides for separating and recovery of a C<sub>4</sub> hydrocarbon rich stream withdrawn by conduit 74 and low severity reformat of about 90-95 R+O octane rating withdrawn by conduit 50.

Hydrogen containing gases separated from the product effluent of the naphtha pretreating step as by separation equipment represented by 62 is conveyed by conduit 76 with or without high purity make up hydrogen in conduit 78 to a catalytic hydrodesulfurization step 80. In the hydrodesulfurization step 80, the distillate fraction in conduit 12 combined with a No. 2 fuel oil product of catalytic cracking are subjected to catalytic hydrodesulfurizing conditions in the presence of hydrogen product gas obtained from the low severity reforming operation. Thus the high purity hydrogen rich gas product of reforming is effectively utilized by cascading the hydrogen first through the naphtha pretreating operation 56 and then to the desulfurizing step 80. The No. 2 fuel oil product of gas oil cracking and separated in fractionation step 46 is withdrawn by conduit 82 and conveyed to the desulfurizing step (CHD). The product effluent of the CHD step 80 is then passed by conduit 84 to separation equipment represented by 86 wherein a fuel gas product is separated and withdrawn by conduit 88 for admixture with fuel gas in conduit 6. Desulfurized No. 2 fuel oil product is withdrawn from zone 86 by conduit 90.

In fractionation equipment represented by fractionator 46 provided downstream of the fluid cracking unit a fuel gas is separated and withdrawn by conduit 92 for admixture with the fuel gas in conduit 6 recovered from the atmosphere distillation step 4. Alkylation feed material is withdrawn from fractionation step 46 by conduit 94 and passed to an alkylation unit 96 along with C<sub>4</sub> hydrocarbons separated from reformat in conduit 74. Alkylate product is withdrawn from alkylation unit 96 by conduit 98. Gasoline product of the fluid cracking operation is separated and recovered from the fractionation step 46 by conduit 100. A portion of this gasoline product may be re cracked as herein provided to reduce its end boiling point. Heavy fuel oil is withdrawn from the bottom of the fractionation step by conduit 102 and combined with the heavy fuel in conduit 24.

The operating conditions relied upon in the catalytic hydrodesulfurization step 80 to upgrade the atmospheric distillate fraction alone or in combination with a No. 2 fuel oil product of catalytic cracking includes a temperature selected from within the range of about 550°F. to about 750°F. at a pressure generally within

the range of 150 to 800 psig. Catalysts suitable for this purpose may be any one of those known in the prior art but preferably the catalyst is either a cobalt molybdenum or a nickel tungsten type of desulfurizing catalyst suitable for the purpose.

The reforming operating conditions are those selected to provide particularly a low severity reforming operation and one particularly directed to accomplish dehydrogenation of naphthenes in one or more sequence of contact steps. Thus since hydrocracking conditions within the reforming operation are to be particularly avoided it is contemplated employing either one or two reactor reforming operations as required to effect the desired dehydrogenation of naphthenes at temperature conditions selected from within the range of 750°F. to 950°F. the lower temperatures being preferred at a pressure particularly promoting the dehydrogenation reaction in the range of 150 to 400 psig., the lower pressure being preferred at a space velocity in the range of 2 to 20 and preferably above 8 v/v/hr. and a hydrogen to hydrocarbon ratio preferably in the range of 2 to 6 to 1.

As mentioned above, the reforming catalyst is preferably one of low cost which accomplishes the desired dehydrogenation of naphthenes under the fairly restricted low severity reforming conditions and particularly those promoting the economies of the combination operation. Thus any of the well known low cost reforming catalyst of the platinum type may be employed with success. This does not mean to say however that other more costly reforming catalyst cannot be employed particularly when the desired dehydrogenation of naphthenes may be accomplished with such a catalyst in a single reforming zone. It is clear therefore that any dehydrogenation catalyst suitable for the purpose may be employed.

The catalyst used in the low severity reforming operation of this invention may be a composite catalyst containing a metal of the platinum series. Typically such a metal is platinum deposited on a porous support such as alumina or combinations thereof with other oxides such as silica, zirconia, titania or germania. The composite may contain a halogen such as chlorine and/or fluorine. On the other hand, the more sophisticated and selective multi metallic component catalysts containing in addition to platinum, one or more other metal components, may be effectively employed in the present process. A representative catalyst of such multi-metallic composites is the combination of platinum with rhemium containing halogen deposited on an alumina support. Other combinations include platinum and gold, platinum and iridium and platinum-gold-iridium with the gold and iridium component being in minor proportions, all of which are deposited on a porous matrix or support material identified above and preferably deposited on an alumina support of the eta, gamma, chi or mixed eta, gamma variety.

The alkylation operation of the present invention may be either sulfuric acid or a hydrogen fluoride type of alkylation operation. On the other hand, a new family of catalyst compositions known as ZSM-5 type of CAS may be employed to upgrade C<sub>4</sub> - C<sub>5</sub> type hydrocarbons by the combination of cracking and cyclization to form aromatics.

Pretreatment of the virgin naphtha fraction to be reformed to remove sulfur and nitrogen compounds therefrom is essential because of the sensitivity of platinum type reforming catalysts to poisoning with sulfur

and nitrogen compounds. Desulfurizing and denitrogenating the naphtha charge is usually accomplished by contact in the presence of hydrogen with a catalyst whereby the contaminants are converted to hydrogen sulfide and ammonia easily separated from the hydrogenated charge. A preferred catalyst for this operation is cobalt molybdate on a support such as alumina. Other desulfurizing catalyst known in the art may also be used in the method of this invention. The operating conditions relied upon for hydrogenation of the naphtha feed include a pressure selected from within the range of 50 to 1000 psig at a temperature selected from within the range of 500° to 900°F. A space velocity in the range of 1 to 10 v/v/hr may be employed at a hydrogen concentration of between 200 and 2000 cubic feet per barrel of hydrocarbon feed. It is desirable to restrict the level of either nitrogen or sulfur in the reforming naphtha charge not to exceed about 5 ppm.

Having thus generally discussed the combination process of the present invention and specifically described a method and combination of operating step for practicing the invention, it is to be understood that no undue restrictions are to be imposed except as defined by the claims.

I claim:

1. In a combination process for upgrading crude oils by catalytic cracking, reforming, desulfurization and alkylation, the improvement which comprises, separating a crude oil to recover gas oils, light and heavy naphtha boiling hydrocarbons and a distillate fraction boiling in the range of about 400°F. to about 650°F.,
  - pretreating the heavy naphtha fraction to remove sulfur and nitrogen, reforming the pretreated heavy naphtha under conditions primarily restricted to dehydrogenation of naphthenes thereby forming a hydrogen rich gas stream, a C<sub>4</sub> hydrocarbon rich stream and a low octane liquid reformat stream, catalytically cracking said low octane liquid reformat in a first riser conversion zone in the presence of a crystalline zeolite conversion catalyst at an elevated temperature of at least 1000°F. and a hydrocarbon residence time within the range of 1 to 8 seconds,
  - catalytically cracking said gas oils with a crystalline zeolite conversion catalyst in a second conversion zone at an elevated temperature of at least 1000°F., recovering from said cracking operations a low boiling olefin rich fraction, a gasoline product fraction and a No. 2 fuel oil fraction,
  - combining the No. 2 fuel oil fraction with said distillate fraction and subjecting the combined fractions to catalytic hydrodesulfurization in the presence of hydrogen rich gas recovered from said reforming operation,
  - alkylating said low boiling olefins with said C<sub>4</sub> hydrocarbons and collecting the gasoline boiling products of said combination operation for gasoline blending.
2. The process of claim 1 wherein light naphtha separated from the crude is combined with the gas oil passed to the second conversion zone.
3. The process of claim 1 wherein the light naphtha separated from the crude oil is initially combined with hot catalyst in a bottom portion of the second riser conversion zone to form a suspension and gas oil is added to the suspension in a downstream portion of the conversion zone.

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4. The process of claim 1 wherein a heavy gasoline product fraction of catalytic cracking is recovered from a lower boiling gasoline fraction and the heavy gasoline product fraction is re-cracked in said first conversion zone in admixture with said low octane liquid reformate.

5. The process of claim 1 wherein the crystalline zeolite cracking catalyst comprises ZSM-5.

6. The process of claim 5 wherein catalyst is recovered from each of said conversion zones as a dense fluid bed of catalyst and C<sub>5</sub> and C<sub>6</sub> hydrocarbons com-

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prising the light virgin naphtha are converted with the ZSM-5 catalyst component.

7. The process of claim 1 wherein hydrogen rich gas product of reforming is employed to desulfurize the naphtha feed passed to reforming and in desulfurization of said distillate fraction.

8. The process of claim 1 wherein hydrogen rich product gas of the reforming operation is passed sequentially through the naphtha pretreating step and then the distillate desulfurization step.

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