

June 28, 1960

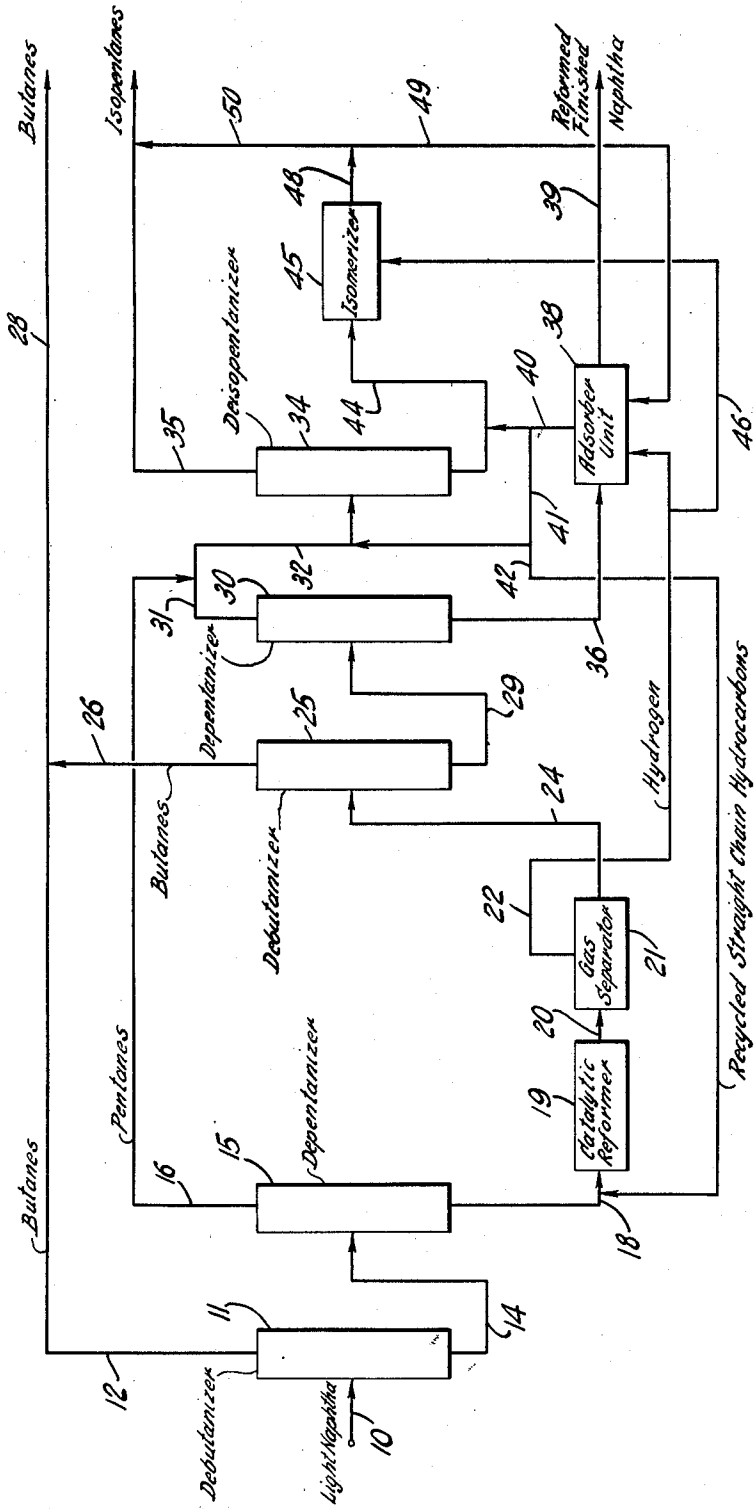
R. A. WOODLE ET AL
NAPHTHA TREATING PROCESS

2,943,037

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2 Sheets-Sheet 1

FIG. 1.



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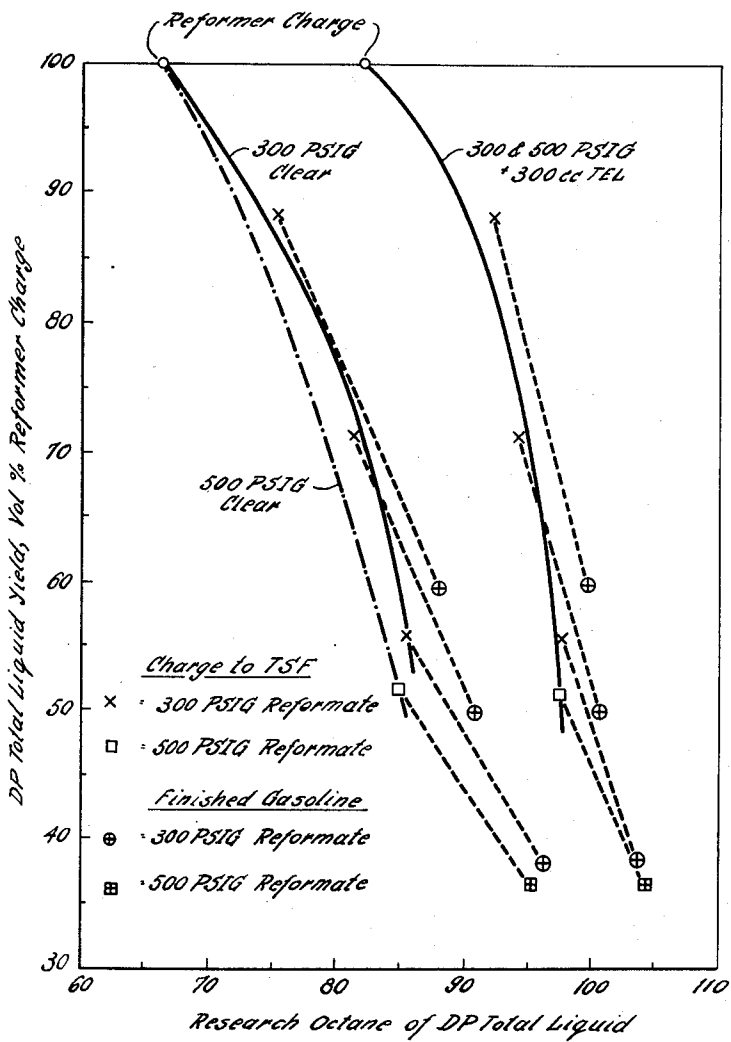
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T-19.2.

Yield Octane Relationship - Reforming & Finishing



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2,943,037

NAPHTHA TREATING PROCESS

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3 Claims. (Cl. 208—79)

This invention relates to a process for the treatment of light petroleum fractions. More particularly, this invention relates to the treatment of light naphthas boiling in the range 40–250° F., more or less, and containing straight chain hydrocarbons and non-straight chain hydrocarbons. Light naphthas which are advantageously treated in accordance with the practice of this invention include such naphthas as a light straight run naphtha, a light catalytically reformed naphtha, a light fluid catalytic cracked naphtha and a light thermally cracked naphtha and natural gasoline.

Light naphthas are generally characterized by a relatively low octane number. This is due to the relatively high proportion of low octane normal paraffins, such as n-pentane, n-hexane, n-heptane, n-octane and the like in such naphthas. Because of their relatively low octane numbers light naphthas are not particularly well suited for blending in present day high octane motor fuels wherein an octane number of 100 or greater is desired. However, since the light naphtha fraction usually comprises a substantial portion of the crude oil and in order to obtain a satisfactory yield of high octane motor fuel from a given amount of crude oil, upgrading of the light naphtha fraction therefrom must be accomplished.

A principal object of this invention is to provide an improved light naphtha conversion process.

Another object of this invention is to provide a process for the conversion of light petroleum fractions, particularly light petroleum naphthas containing substantial amounts of straight chain hydrocarbons, in a plurality of relatively high octane streams suitable for blending in a high octane motor fuel.

Still another object of this invention is to provide a process for upgrading light naphthas having a boiling range in the range of 40–250° F. and containing a substantial amount of straight chain hydrocarbons, at least about 5% by volume, to yield a plurality of relatively high octane hydrocarbon streams.

How these and other objects of this invention may be accomplished will become apparent from the accompanying detailed description and drawings which schematically illustrate an embodiment of the practice of this invention and wherein Fig. 1 is a flow diagram of a naphtha treating process in accordance with one embodiment of the practice of this invention and wherein Fig. 2 graphically illustrates the advantages obtainable in the practice of this invention.

In accordance with our invention a light naphtha fraction, such as a light straight run naphtha, a light catalytically reformed naphtha, a light fluid catalytic cracked naphtha, a light thermal cracked naphtha, natural gasoline, or mixtures thereof, is fractionated to separate therefrom a relatively high octane hydrocarbon stream, such as a stream comprised essentially or predominantly of isopentane. The remaining naphtha fraction, substantially free of pentanes, particularly isopentane, is then sub-

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jected to catalytic reforming to produce a reformer effluent having a relatively high octane number. This reformer effluent made up of hydrogen, butanes, pentanes and higher molecular weight hydrocarbons is fractionated and there are separately recovered therefrom a hydrogen stream, a C₄ or butane hydrocarbon stream, a C₅ or pentane hydrocarbon stream and a remaining reformate stream. These streams are then suitably treated and blended in accordance with the practice of this invention to yield a plurality of hydrocarbon streams having a relatively improved octane number. For example, the remaining reformate stream is fractionated by selective adsorption to separate the straight chain hydrocarbons therefrom, thereby yielding a finished reformate having a higher octane number. The separated straight chain hydrocarbons may then be recovered and suitably converted, as by isomerization, into a relatively higher octane number stream.

A petroleum naphtha fraction suitable for use in the practice of this invention might have an initial boiling point in the range 40–85° F. and an end point in the range 175–250° F. Further, such a light petroleum fraction must contain both straight chain hydrocarbons and non-straight chain hydrocarbons and might have the following compositions.

Hydrocarbon type:	Percent by volume
Naphthenes -----	0–60
Aromatics -----	0–40
Saturates (including normal paraffins and isoparaffins) -----	20–95
Unsaturates (including normal olefins and isoolefins) -----	0–50

One treating step, as indicated above, employed in a combination treating process of our invention includes a selective adsorption operation wherein a relatively high boiling portion of a catalytic reformate derived from the treatment of a light naphtha fraction in accordance with the practice of this invention is treated or contacted with a solid selective adsorbent which selectively adsorbs straight chain hydrocarbons to the substantial exclusion of non-straight chain hydrocarbons. By straight chain hydrocarbons is meant normal paraffins, normal olefins, normal diolefins and the like such as n-hexane, n-heptane, n-octane and the corresponding olefins, diolefins or the corresponding normal polyolefins and acetylenic hydrocarbons. Non-straight chain hydrocarbons which are substantially unaffected or unadsorbed by the special selective adsorbent employed in the practice of this invention include the isoparaffins and the isoolefins such as isohexane, isoheptane, isooctane, isohexene, isoheptene, isooctene and the like as well as the naphthenes and aromatic hydrocarbons.

Any solid adsorbent which selectively adsorbs straight chain hydrocarbons to the substantial exclusion of non-straight chain hydrocarbons can be employed in the practice of this invention. It is preferred, however, to employ as the adsorbent certain natural or synthetic zeolites or aluminosilicates, such as a calcium aluminosilicate, which exhibit the property of a molecular sieve, that is, adsorbents made up of porous matter or crystals wherein the pores are of molecular diameter and are of uniform size. A particularly suitable solid adsorbent for the adsorption of straight chain hydrocarbons to the substantial exclusion of non-straight chain hydrocarbons is a calcium aluminosilicate manufactured by Linde Air Products Company and designated type 5A molecular sieve. The crystals of this particular calcium aluminosilicate, apparently actually a sodium calcium aluminosilicate, have a pore size or diameter of about 5 Angstrom units,

a pore size sufficient to admit straight chain hydrocarbons, such as the n-paraffins, to the substantial exclusion of the non-straight chain hydrocarbons, such as the naphthenic, aromatic, isoolefinic and isoparaffinic hydrocarbons, e.g., isobutane and higher. This particular selective adsorbent is available in various sizes, such as $\frac{1}{16}$ " and $\frac{1}{8}$ " diameter pellets as well as in a finely divided powder form, suitable for employment in a fixed bed, a moving bed or a fluidized bed adsorption process.

Other selective adsorbents may be employed in the practice of this invention. For example, it is contemplated that adsorbents having the property of selectively adsorbing straight chain hydrocarbons to the substantial exclusion of non-straight chain hydrocarbons in the manner of a molecular sieve may be obtained by suitable treatment of various oxide gels, especially metal oxide gels of the polyvalent amphoteric metal oxides.

Other suitable selective adsorbents are known and include the synthetic and natural zeolites which, when dehydrated, may be described as crystalline zeolites having a rigid three dimensional anionic network and having interstitial dimensions sufficiently large to adsorb straight chain hydrocarbons but sufficiently small to exclude the non-straight chain hydrocarbons. The naturally occurring zeolite, chabazite, exhibits such desirable properties. Another suitable naturally occurring zeolite is analcite which, when dehydrated and when all or part of the sodium is replaced by an alkaline earth metal, such as calcium, yields a material which may be represented by the formula $(Ca, Na)Al_2Si_4O_{12} \cdot 2H_2O$ and which, after suitable conditioning, will adsorb straight chain hydrocarbons to the substantial exclusion of non-straight chain hydrocarbons. Naturally occurring, or synthetically prepared phacellicite, gmelinite, harmotome and the like or suitable modifications of these products by base exchange are also applicable in the practice of this invention.

Other solid adsorbents which adsorb straight chain hydrocarbons such as n-paraffins and n-olefins to the substantial exclusion of non-straight chain hydrocarbons, including the aromatic and naphthenic hydrocarbons, are known.

Referring now to the drawing and in greater detail to Fig. 1 thereof there is schematically illustrated a flow diagram of one embodiment of the practice of this invention. A light straight run naphtha boiling in the range 60–225° F. is introduced via line 10 into debutanizer 11 wherein the butanes or C₄ hydrocarbons are removed overhead via line 12. The remaining bottoms portion is recovered via line 14 and introduced into depentanizer 15 wherein the pentane or C₅ fraction comprised predominantly of n-pentane and isopentane is recovered overhead via line 16. The bottoms from depentanizer 15 comprising the relatively high molecular weight hydrocarbons, paraffinic and non-paraffinic, as well as straight chain and non-straight chain hydrocarbons, initially present in the light straight run naphtha is recovered via line 18 and introduced into catalytic reformer 19 where they are subjected to catalytic reforming in contact with a suitable catalyst such as a platinum-containing catalyst, e.g., platforming catalyst. Catalytic reformer 19 is operated under such conditions of temperature so as to effect the isomerization and/or dehydrocyclization of the straight chain paraffinic hydrocarbons and the non-straight chain paraffinic hydrocarbons introduced thereto via line 18, as well as aromatization of the naphthenes.

There is recovered from catalytic reformer 19 via line 20 a total reformer effluent comprising hydrogen, together with a small amount of normally gaseous hydrocarbons such as methane and ethane, as well as a C₄ hydrocarbon fraction comprising butane and isobutane, a C₅ hydrocarbon fraction comprising n-pentane and isopentane together with relatively high molecular weight reformed hydrocarbons such as non-straight chain C₆, C₇ and C₈ and higher hydrocarbons, such as isohexane, isooheptane,

isopentane, benzene, toluene, xylenes and the like. The reformer effluent from catalytic reformer 19 is carried via line 20 into gas separator 21 wherein the normally gaseous portion thereof comprised substantially entirely of hydrogen is removed overhead via line 22. The remaining liquid reformer effluent passes from separator 21 via line 24 into debutanizer 25 wherein there is recovered overhead via line 26 the C₄ hydrocarbon fraction comprising butane and isobutane which is blended or otherwise admixed with the C₄ hydrocarbon fraction obtained initially from the light straight run naphtha via debutanizer 11 and line 12. The resulting admixture of the butanes may be separately recovered for blending purposes via line 28.

The bottoms from debutanizer 25 is removed via line 29 and introduced into depentanizer 30 wherein the pentane or C₅ hydrocarbon fraction thereof is recovered and then preferably admixed with the C₅ fraction recovered overhead from depentanizer 15 via line 16 and introduced via line 32 into deisopentanizer 34 from which isopentane or a portion thereof is recovered overhead via line 35. The remaining bottoms reformer effluent recovered from depentanizer 30 via line 36 is introduced into adsorber unit or adsorption zone 38. The remaining reformer effluent introduced into adsorption zone 38 comprises reformed relatively high molecular weight hydrocarbons, including non-straight chain paraffinic hydrocarbons such as isohexane, isooheptane, isooctane and the like as well as the cyclic aromatic hydrocarbons such as benzene, toluene, xylenes together with some unreacted or unconverted straight chain hydrocarbons such as n-hexane, n-heptane, n-octane and the like.

There is provided within adsorption zone 38 a selective adsorbent such as Linde type 5A molecular sieve which selectively adsorbs straight chain hydrocarbons to the substantial exclusion of non-straight chain hydrocarbons. The selective adsorption unit or adsorption zone 38 may comprise a fixed bed, a fluidized bed or a moving bed of selective adsorbent maintained at a suitable temperature, such as a temperature in the range 200–750° F., to effect selective adsorption of the straight chain hydrocarbons from the remaining reformer effluent introduced thereto. The adsorption operation is carried out at any convenient pressure, such as a pressure in the range 0–500 p.s.i.g. Desirably the temperature and pressure within adsorption zone 38 is adjusted so that the reformer effluent undergoing fractionation therein is maintained in the vapor or gaseous phase. Liquid phase adsorption, although operable, is less preferred.

Following the selective adsorption operation carried out within selective adsorption unit 38 there is recovered therefrom via line 39 a finished reformat or reformed naphtha comprised substantially only of non-straight chain hydrocarbons and having an increased octane number as compared with the light straight run naphtha introduced into the process via line 10. The finished reformed naphtha recovered from the process via line 39 may be used directly as a motor fuel or as a high octane blending component of a high octane motor fuel. Advantageously, this finished reformed naphtha may also be blended with the butanes recovered from debutanizer 11 and debutanizer 25 via lines 12 and 26, respectively.

The straight chain hydrocarbons adsorbed within the selective adsorbent employed within adsorber unit 38 are desorbed therefrom. In accordance with one embodiment of the practice of this invention the desorption of the adsorbed straight chain hydrocarbons is effected by contacting the selective adsorbent with the gaseous fraction recovered from gas separator 21 via line 22. As indicated hereinbefore, this gaseous fraction is composed predominantly of hydrogen. The desorption operation is carried out at a relatively elevated temperature, usually in the range 400° F.–800° F., preferably at a temperature 50–250 degrees Fahrenheit greater than the adsorption temperature. Like the selective adsorption operation

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the desorption operation is carried out at a suitable temperature and pressure so that the resulting desorbed straight chain hydrocarbons are desorbed or recovered in the gaseous phase. A suitable desorption temperature is in the range 300–800° F. and a pressure in the range 0–500 p.s.i.g. If desired, isothermal and/or isobaric adsorption and desorption operations may be employed.

Various materials besides hydrogen or the normally gaseous portion of the reformer effluent gas may be employed to effect desorption of the adsorbed hydrocarbons from the adsorbent. Suitable gaseous desorbing agents include methane, ethane, propane, butanes such as n-butane, isobutane or mixtures thereof, nitrogen, flue gas, carbon dioxide, air, or suitable mixtures thereof and the like. In general, any gaseous material which is compatible with or otherwise chemically inert with respect to the straight chain hydrocarbons to be desorbed and chemically inert with respect to the adsorbent employed may be utilized. It is preferred, however, as previously indicated to employ the reformer hydrogen effluent recovered from gas separator 21 via line 22 or another material such as a C₄ hydrocarbon (butane and/or isobutane) readily separable by distillation from the resulting straight chain hydrocarbon desorbate.

The desorbed straight chain hydrocarbons are recovered from adsorber unit 38 via line 40. If desired, the total desorption effluent comprising the desorbed straight chain hydrocarbons and gaseous desorbing medium (hydrogen) is returned via lines 41, 42 and 18 to catalytic reformer 19. When the practice of this invention is carried out in accordance with this embodiment the straight chain hydrocarbons originally present in the light straight run naphtha introduced into the process via line 10 are substantially completely converted to non-straight chain hydrocarbons, either the corresponding non-straight chain isoparaffinic hydrocarbons and/or the related cyclic aromatic hydrocarbons.

If desired, the desorption effluent recovered from adsorber unit 38 via line 40 may be introduced via line 44 together with the bottoms from deisopentimizer 34 comprised predominantly of n-pentane into isomerizer 45. As indicated in Fig. 1, isomerizer 45, if desired, may be supplied with gaseous hydrogen recovered from gas separator 21 via lines 22 and 46.

Isomerizer 45 is operated under suitable conditions of temperature and pressure, preferably employing an isomerizing platinum-containing catalyst such as UOP R-5 platforming catalyst, to effect isomerization or conversion of the straight chain hydrocarbons into the corresponding non-straight chain isoparaffinic hydrocarbons. The isomerization of the pentane fraction recovered as bottoms from deisopentimizer 34 via line 44 and the isomerization of the desorbed straight chain hydrocarbons recovered from adsorber unit 38 via line 40, as indicated in Fig. 1, may be carried out advantageously within isomerizer 45. Desirably, however, separate isomerizers (not illustrated) are provided to effect separate isomerization of the n-pentanes and separate isomerization of the desorbed straight chain hydrocarbons emanating from adsorber unit 38. The isomate or isomerizer effluent issuing from isomerizer 45 via line 48 may be reintroduced into adsorber unit 38 via line 49 to effect separation of the non-straight chain and the straight chain hydrocarbons therein. Further, if desired, the resulting isomerizer effluent may be admixed or otherwise blended via line 50 with the isopentanes recovered from deisopentimizer 34 via line 35.

Blending of the isomerizer effluent with the isopentane in line 35 is particularly desirable or suitable when the isomerizer effluent is comprised substantially only of C₅ hydrocarbons such as would be the case when a separate isomerization facility is provided for the n-pentane recovered from deisopentimizer 34.

The following example is illustrative of the practice of this invention.

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EXAMPLE

A light straight run gasoline was debutanized and depentanized to yield a reformer charge stock having an initial boiling point of 150° F. and an end point of 206° F. The API gravity of the charge stock was 71.8 and a hydrocarbon type analysis of the charge stock showed that it contained 3.4% by weight aromatics, 1% by weight olefins, 72.3% by weight paraffins and 23.3% by weight naphthenes. The charge stock had an ASTM research octane No., clear, of 65.6, +3 cc. TEL of 82.0.

The charge stock was subjected to catalytic reforming (platforming) by contact under reforming conditions of temperature and pressure with a platinum-containing catalyst (UOP R-5 platforming catalyst). The resulting reformate was depentanized to yield a reformate having an ASTM research octane No., clear, of 75.4, +3 cc. TEL of 92.2.

The depentanized reformate was then subjected to a selective adsorptive separation operation employing a sodium calcium aluminosilicate molecular sieve type adsorbent (Linde type 5A molecular sieve) to yield a finished reformate having an ASTM research octane No., clear, of 88.2, +3 cc. TEL of 99.8.

The straight chain hydrocarbons separated from the reformate by selective adsorption are advantageously separately treated as by isomerization to improve the octane number thereof. For example, a mixture of straight chain hydrocarbons comparable to the straight chain hydrocarbons separated from the reformate during the aforesaid selective adsorption operation having a composition of about 23% by volume n-pentane, 56% by volume n-hexane and 21% by volume n-heptane and having an ASTM research octane No., clear, of 39 was contacted with a platinum isomerization catalyst at a temperature of about 850° F., at a pressure of 550 p.s.i.g. and a space velocity of 1 v./hr./v. employing a hydrogen recycle rate of about 4,000 s.c.f. per barrel of charge. The resulting isomate consisted of a mixture of straight chain and non-straight chain hydrocarbons and had an ASTM research octane No., clear, of about 74.

The resulting isomate, in accordance with one embodiment of the practice of this invention, was then vaporized and contacted with a solid selective adsorbent for the removal of the straight chain hydrocarbons therefrom under conditions similar to those employed in the treating of the depentanized catalytic reformate. The thus-finished isomate exhibited an ASTM research octane No., clear, of about 82.0. Blending of the finished catalytic reformate and the finished isomate could then be carried out to yield a naphtha particularly suitable as a motor fuel.

Another mixture of straight chain hydrocarbons comparable to the hydrocarbons desorbed from the solid adsorbent and having an ASTM research clear octane number of about 39 was contacted with an aromatization or dehydrogenation catalyst consisting of chromic oxide, magnesium oxide and aluminum oxide. The mixture of straight chain hydrocarbons was contacted at a pressure of 40 p.s.i.g. and a space velocity of 0.4 v./hr./v. in the presence of hydrogen at a recycle rate of 1200 s.c.f./bbl. of charge. Contacting temperatures and the results obtained are given in Table I below:

Table I

Temperature, ° F.	920	943
Wt. Percent Recovery (Liquid)	93.8	81.3
Bromine No.	20	22
Vol. Percent Aromatics	15	13
ASTM Research Octane No. (Clear)	60.6	57.6
+3 cc. TEL/gal.	80.4	80.2

Other mixtures of straight chain hydrocarbons comparable to the straight chain hydrocarbons recovered during the desorption operation having a composition of about 23% by volume n-pentane, 56% by volume

n-hexane and 21% by volume n-heptane and exhibiting an ASTM research octane number, clear, of 39 were contacted with various catalysts under isomerizing conditions of temperature and at a pressure of about 500 p.s.i.g., a space velocity of 1.0 v./hr./v. and a hydrogen recycle rate of 4000 s.c.f./bbl. The catalysts, reaction temperatures and ASTM research octane No., clear, of the converted products and of the finished converted products, i.e., converted products after having been contacted with adsorbent for the removal of straight chain hydrocarbons, are set out in Table II below:

Table II

Temp., ° F.	800			850			900		
	Plat.	Baker	Ultra	Plat.	Baker	Ultra	Plat.	Baker	Ultra
Catalyst.....									
Liq. Rec., Wt. Percent.....	95.9	99.3	90.1	95.0	99.0	88.2	67.6	70.1	72.7
Converted Product, Octane No.....	60.0	51.0	54.0	75.4	74.0	77.1	89.6	70.0	78.9
Product Finished by Selective Adsorption, Octane No.....	78.0	61.0	72.0	84.0	82.0	82.6	-----	80.3	-----

Plat.—UOP platforming catalyst.

Baker—Sinclair—Baker RD 150 catalyst.

Ultra—Standard of Indiana ultraforming catalyst.

The foregoing examples illustrate how a light naphtha fraction advantageously is treated to produce a plurality of upgraded stocks particularly suitable for blending to produce a high octane motor fuel.

Referring now to Fig. 2 of the invention which graphically illustrates the advantages of the practice of this invention there is illustrated the yield-octane relationships of naphtha fractions treated in accordance with the practice of this invention. As illustrated in Fig. 2 the leaded octane (+3 cc. TEL) of the finished gasoline range from 99.8 to 104.4. A line correlating the finished gasoline leaded octane yield data intersected 100 octane (+3 cc. TEL) at about 55 volume percent depentanized gasoline yield basis fresh feed to the catalytic reformer (platformer).

From the data presented hereinabove it is apparent that a depentanized light straight run gasoline can be upgraded to above 100 octane (the research +3 cc. TEL) by catalytic reforming (platforming) and subsequent finishing or removal of the straight chain hydrocarbons from the depentanized reformate.

It has been shown that from 100 barrels of depentanized light straight run gasoline the following quantities of liquid materials would be produced

Finished gasoline, ASTM Research Octane No. + 3 cc. TEL	100	104
Finished gasoline, bbls.....	55	36.4
Isopentane, bbls.....	2.1	6.3
N-pentane, bbls. (which can be upgraded to isopentane).....	1.2	5.1
Desorbate, bbls. (which can be upgraded by isomerization or aromatization to high octane hydrocarbons).....	25	15.3
Butane, bbls.....	5.5	16.8

Obviously many modifications and changes may be made in the practice of this invention without departing from the spirit or scope thereof.

We claim:

1. A process for upgrading a light naphtha fraction into a plurality of relatively high octane hydrocarbon streams which comprises fractionating a light naphtha petroleum fraction having a boiling range in the range

40–250° F. and containing pentanes and higher molecular weight paraffinic hydrocarbons to separate said pentanes from said high molecular weight paraffinic hydrocarbons, fractionating said pentanes to separate isopentane from n-pentane, recovering the resulting separated isopentane, subjecting the separated n-pentane to isomerization to yield an isomerized effluent comprising isopentane, catalytically reforming the higher molecular weight paraffinic hydrocarbons to yield a reformer effluent comprising hydrogen, C₄ hydrocarbons, pentanes and higher molecular weight reformed hydrocarbons, sepa-

rately separating said hydrogen, said C₄ hydrocarbons and said pentane from said reformer effluent, separately recovering the resulting separated C₄ hydrocarbons, subjecting the resulting separated pentanes to fractionation to separate isopentane from n-pentane, blending the thus-separated isopentane with the aforesaid previously separated isopentane and blending the thus-separated n-pentane with the aforesaid separated n-pentane prior to isomerization, subjecting the remaining reformer effluent to contact with a solid molecular sieve alumina-silicate selective adsorbent which selectively adsorbs straight chain hydrocarbons to the substantial exclusion of non-straight chain hydrocarbons, said contacting operation being carried out at a temperature in the range 200–750° F. and at a pressure in the range of 0–500 p.s.i.g. and under conditions such that said reformer effluent is in the gaseous phase, recovering from the aforesaid selective adsorption operation a first hydrocarbon stream substantially free of straight chain hydrocarbons and desorbing the adsorbed straight chain hydrocarbons from said selective adsorbent by contacting said adsorbent with gaseous aforesaid separated C₄ hydrocarbons at a temperature in the range 400–800° F. and at a pressure in the range 0–500 p.s.i.g., said desorption temperature being 50–250 degrees Fahrenheit greater than the aforesaid adsorption temperature and such that the resulting desorbed straight chain hydrocarbons are in the gaseous phase.

2. A process in accordance with claim 1 wherein said straight chain hydrocarbons desorbed from said selective adsorbent by contact with said C₄ hydrocarbon are recycled to the aforesaid catalytic reforming operation.

3. A process in accordance with claim 1 wherein said straight chain hydrocarbons desorbed from said selective adsorbent are isomerized in the aforesaid isomerization operation.

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