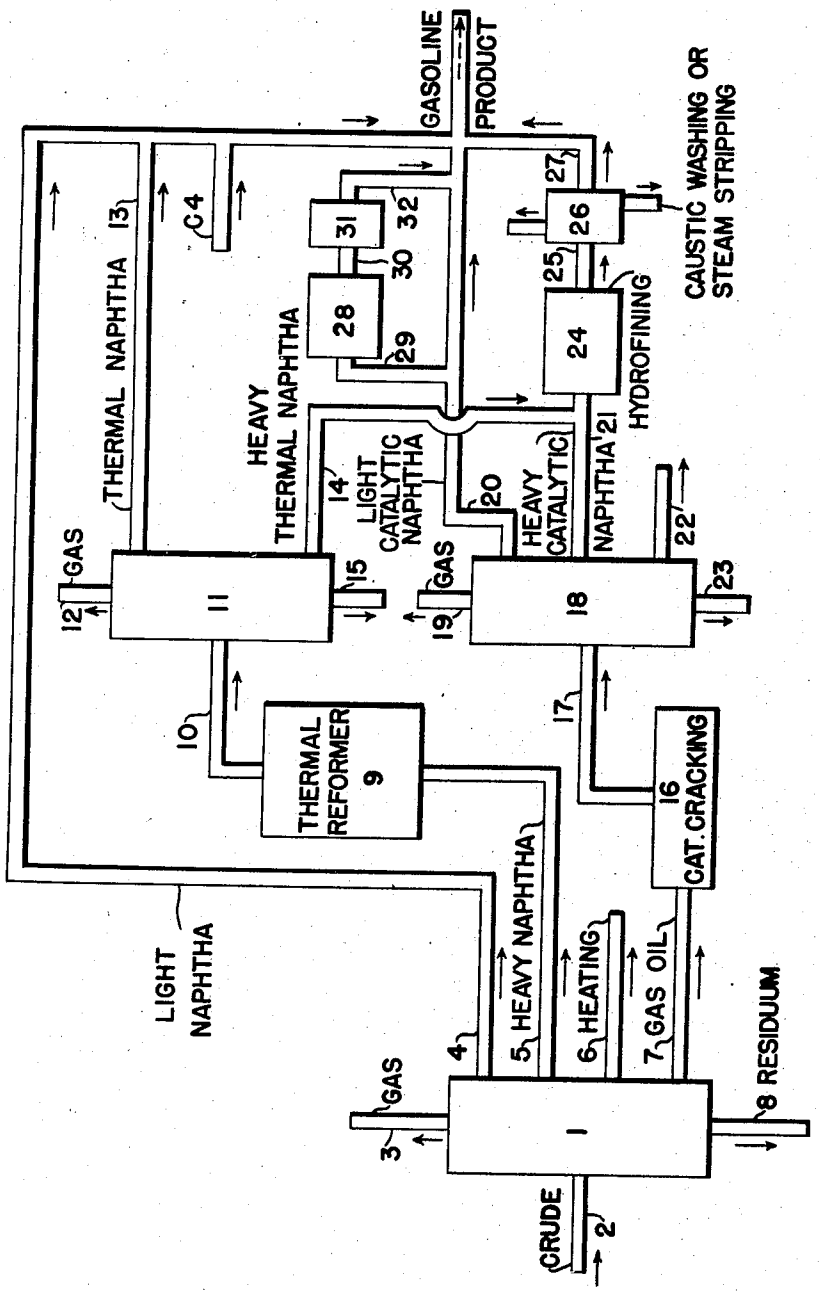
March 10, 1959 R. C. MORBECK ET AL 2,877,172

COMBINED THERMAL REFORMING, CATALYTIC CRACKING AND HYDROFINING PROCESS TO IMPROVE ENGINE CLEANLINESS

Filed July 14, 1952



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United States Patent Office 2,877,172
Patented Mar. 10, 1959

2,877,172

COMBINED THERMAL REFORMING, CATALYTIC CRACKING AND HYDROFINING PROCESS TO IMPROVE ENGINE CLEANLINESS

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Application July 14, 1952, Serial No. 298,656

7 Claims. (Cl. 208—79)

The present invention is concerned with the production of high quality petroleum oil products. The invention is more particularly concerned with the treatment of cracked petroleum oil fractions boiling below about 700° F. by a controlled, mild hydrofining operation. In accordance with the present invention thermally or catalytically cracked petroleum hydrocarbons, especially those boiling below about 430° F. are hydrofined whereby the resulting product comprises a fuel having improved engine, cleanliness. The hydrofining operation is conducted so as to give a reduction in bromine number not greater than about 20%.

It is well known in the art to refine petroleum fractions for the production of high quality gasolines, kerosenes, and diesel fuels by various procedures. For example, it is known to treat petroleum fractions boiling in the motor fuel boiling range with sulfuric acid and with various other reagents. While the use of sulfuric acid produces a satisfactory product, in many operations, particularly when the feed oil comprises a cracked stock, a relatively high polymerization loss results. Furthermore, when the concentration of undesirable constituents is relatively high, it is necessary to use rather large quantities of acid which necessitates further refining of the acid treated oil after removal of the treating acid. In addition, the acid must be recovered and reconcentrated or otherwise restored. It has now been discovered that providing the cracked constituents of a motor fuel blend are hydrofined in a controlled manner, the quality of the fuel particularly with respect to engine cleanliness characteristics is materially improved. By the present process wherein cracked petroleum hydrocarbons boiling in the motor fuel boiling range are hydrofined, the same are substantially completely freed of undesirable constituents which cause corrosion, varnish and valve deposits in an internal combustion engine. Sludge deposits in the lubricating oil are also avoided.

The process of the present invention may be more fully understood by reference to the drawing illustrating an embodiment of the same.

Referring specifically to the drawing, a crude oil feed stock is introduced into distillation zone **1** by means of feed line **2**. Temperature and pressure conditions in zone **1** are adapted to remove overhead by means of line **3** normally gaseous hydrocarbons. Constituents boiling in the light naphtha boiling range are removed by means of line **4**, while hydrocarbons boiling in the heavy naphtha boiling range are removed by means of line **5**. A heating oil fraction is removed by means of line **6**, while a gas oil fraction is removed by means of line **7**. A residuum fraction is removed by means of line **8**.

In accordance with the present invention the heavy naphtha fraction removed by means of line **5** is passed to a thermal cracking or reforming zone **9**. In this zone the oil is raised to a temperature in the range from about 850° to 1050° F. and held at a pressure in the range from about 250 to 1000 lbs. p. s. i. g. The operating conditions are adapted, that is, time, temperature and

pressure, to crack the feed and to produce a higher octane number product. The cracked or reformed product is removed from thermal cracking zone **9** by means of line **10** and passed into a distillation zone **11**. Temperature and pressure conditions in zone **11** are adapted to segregate a gas by means of line **12**, a light thermal naphtha by means of line **13**, a heavy thermal naphtha by means of line **14** and a higher boiling fraction which is removed from zone **11** by means of line **15**.

In accordance with the present invention the gas oil fraction removed by means of line **7** is passed into a catalytic cracking zone **16**, wherein the same is subjected to a catalytic cracking conditions, in the presence of a suitable catalyst, as, for example, alumina or magnesia. Temperatures in the catalytic cracking zone are in the range of about 800° F. to 1100° F., while pressures are in the range from about 5 to 150 lbs. p. s. i. g. The catalytically cracked product is removed from zone **16** by means of line **17** and passed into a fractionating zone **18**. Temperature and pressure conditions in zone **18** are adapted to segregate a gas which is removed by means of line **19**, a catalytically cracked light naphtha which is removed by means of line **20**, a catalytic heavy naphtha which is removed by means of line **21**, and a heating oil fraction segregated by means of line **22**. A higher boiling fraction is removed by means of line **23**.

In accordance with the present invention the heavy thermal naphtha segregated by means of line **14** and the catalytically cracked heavy naphtha segregated by means of line **21** are passed to hydrofining zone **24**, wherein the same is mildly hydrofined. The hydrofined product is removed from zone **24** by means of line **25** and passed into a caustic washing or steam stripping zone **26**.

The hydrofined product is removed from zone **26** by means of line **27**, and preferably combined with the other segregated naphtha fractions.

It is also within the concept of the present invention to hydrofine under certain conditions the light catalytic naphtha segregated by means of line **20**. If this mode of operation be utilized, the light catalytic naphtha is passed into hydrofining zone **28** by means of line **29**. The hydrofined product is removed from zone **28** by means of line **30** and passed into a caustic washing or steam stripping zone **31**. The finished product is withdrawn from zone **31** by means of line **32** and preferably blended with other naphtha streams.

It is essential, in practicing the present invention that the hydrofining operation conducted on the cracked oil be a "mild hydrofining operation." This is to be distinguished from conventional hydrofining operations heretofore practiced in the art. Such hydrofining operations have been employed at pressures from 200 to 500 lbs. p. s. i. g., at feed rates of .5 to 2.0 volumes of feed per volume of catalyst per hour. Relatively high rates of hydrogen recycle have been employed as for example, 2,000 to 4,000 standard cu. ft. per barrel in order to prevent carbonization of the catalyst. Likewise, very active catalysts have been used which are effective for desulfurization. Under these conditions hydrogen consumption has generally been in the range of 150 to 600 standard cu. ft. per barrel of feed. This relatively high consumption of hydrogen in the past has made the process expensive to operate, so that its application in the past has been limited to the treatment of relatively high sulfur stocks which could not be desulfurized by any other available treating operation. The catalyst heretofore employed and as used in the present process is cobalt molybdate supported on a carrier, as for example alumina.

Due to the fact that the sulfur content of cracked naphthas is relatively low, since the cracking operation desulfurizes to a great extent, conventional hydrofining

operations have not generally been necessary in the processing of cracked oils.

The "mild hydrofining" conditions of the present invention may be secured by lowering the temperature, increasing the feed rate per volume of catalyst or by using a less active catalyst. In accordance with the present invention the temperatures used are in the range from about 400 to 700° F., preferably in the range from about 500 to 650° F. Pressures employed are in the range from 50 to 250 lbs. p. s. i., preferably in the range from about 100 to 200 lbs. p. s. i. The feed rates, in accordance with the present process, are in the range from about 1–20 volumes of liquid per volume of catalyst per hour. Preferred feed rates are in the range from 6–16 v./v./hr. The hydrogen in the gas to the hydrofining unit may vary from 50 to 100%. This means that, for example, dilute hydrogen from a hydroformer can be used in the hydrofining process. A particularly desirable method of hydrofining in accordance with the present process is to recycle appreciable quantities of hydrogen to the hydrofining unit in order to completely prevent carbonization of the catalyst.

The catalyst utilized in the present operation may comprise known hydrofining catalyst, as for example, cobalt molybdate on a carrier as for example, alumina, providing other operating conditions are adjusted to secure a "mild hydrofining" process. The amount of cobalt molybdate employed is about 5 to 13% by weight based upon the weight of the alumina.

The catalyst is prepared by known methods, such as by impregnation of the alumina with an ammoniacal solution of cobalt and molybdenum salts. The catalyst is dried and decomposed to convert the cobalt and molybdenum salts to the oxides.

Other catalysts may be employed, as for example, a mixture of tungsten sulfide and nickel sulfide.

It is to be understood that the mild hydrofining conditions of the present invention are secured by the adjustment of the above named operating conditions. For instance, if a relatively high liquid feed rate is used as compared to the amount of catalyst present, the higher temperature range may be employed. On the other hand, if a very active catalyst is used, it is desirable to use a relatively high feed rate or to use a relatively low temperature. The mild hydrofining conditions of the present invention are measured by the amount of hydrogen consumption per barrel of oil feed. As pointed out heretofore in the art, conventional hydrofining operations utilized for the desulfurization of certain stocks are conducted under conditions whereby the hydrogen consumption ranges from 150 to 600 standard cu. ft. of hydrogen per barrel of oil. These operations used heretofore in the art secured a substantial sulfur reduction (50 to 90%). In accordance with the present process, operating conditions are adjusted so that the hydrogen consumption in standard cu. ft. per barrel does not exceed 60 and is preferably less than 40.

A very desirable method of measuring the hydrofining conditions is to so control the variable factors so that the reduction in bromine number is not greater than about 20%. As pointed out above in the operation of gasoline engines on fuels containing cracked components, there is a decided tendency for varnish to deposit on the piston and for sludge to be deposited in the lubricating oil. This tendency for sludge to deposit is particularly pronounced in stop and go driving such as is encountered in the operation of delivery trucks and local busses. This deposition of sludge in the oil tends to clog oil lines and streams and thus impair the operation of the lubricating system in the engine.

The sludge-forming tendency of the cracked naphthas is dependent to a considerable extent on the boiling range; the heavy cracked naphthas being much worse offenders than the light naphthas, that is, cracked naphthas boiling in the range from about 250° F. to 430° F. Also, as pointed out heretofore, acid treating is one method which has been found to be effective in reducing the sludge-forming tendency of cracked naphthas used in gasolines. For example, treats of two to twelve pounds of 75–90% acid per barrel of naphtha, have proved effective in reducing sludge forming tendencies. However, acid treating is an expensive operation involving batch treats with treating losses of about 2 to 10%. Furthermore, recovery and regeneration of the acid is an expensive operation and frequently contributes to atmospheric pollution.

The present invention is concerned with the use of mild hydrofining as a new means of improving the engine cleanliness characteristics of cracked naphthas. The effectiveness of mild hydrofining for this purpose has been demonstrated by engine tests.

The process of the present invention will be more fully understood by the following example illustrating the same:

EXAMPLE I

A naphtha obtained by thermal reforming a virgin naphth and having a boiling range of 275–428° F. was hydrofined over cobalt molybdate catalyst supported on alumina. The temperature was 565° F., the feed rate 8.5 v./v./hr., and the pressure 200 p. s. i. g. The hydrogen rate including recycle was 1000 cu. ft. per barrel of feed. Inspections on the feed and on the product after caustic washing for hydrogen sulfide removal are shown in the following tabulation:

Table I

	Feed	Hydrofined Product
Bromine Number[1]	18	15
Sulfur—Wt. Percent	0.05	0.04
Phenol Number[2]	390	33
Diene Number[3]	1.32	0.08
Ultraviolet Absorptivity at 235 mμ	2.65	0.65
Diazonium Fluoborate Deposit Factor[4]	30	1
CFRR Octane No. with 2 cc. TEL	[5] 84.1	[5] 84.1

[1] Centigrams of bromine per gram.
[2] Equivalent milligrams paratertiary amyl phenol per 100 cc.
[3] Milligrams bromine per gram.
[4] The diazonium fluoborate deposit factor is an index of the engine cleanliness characteristics of a motor fuel. This test utilizes the coupling reaction which takes place between diazotised p-nitraniline fluoborate and certain unsaturated compounds (which cause engine fouling) to form a colored compound. Intensity of color formed after a given time interval is taken as an index of the deposit forming compounds present. In running the test the fraction of the sample boiling over 275° F. and purified by treatment with dilute acid and alkali and steam distillation to 3% bottoms is used. A known quantity of this fraction is added to a solution of p-nitraniline diazonium fluoborate ($NO_2C_6H_4N{=}NBF_4$) in acetone and the optical density measured at intervals in a Hellige-Diller photometer. The optical density is plotted against time and the value after 20 minutes read from the curve. The optical density multiplied by the ratio of the fraction boiling over 275° F. to the whole sample gives the diazonium fluoborate deposit factor.
[5] Differential octane ratings.

The mildness of the hydrofining conditions used is evident from the fact that the bromine number was reduced only from 18 to 15 and there was no loss in the leaded octane number of the treated product. The phenol number, the diene number, the ultraviolet absorptivity and the diazonium fluoborate deposit factor indicated that the fuel had substantially improved engine cleanliness characteristics. The above example illustrates that mild hydrofining is effective in improving engine cleanliness characteristics. It is very important that the hydrofining conditions be maintained mild in order to prevent loss in octane number on the product.

The only positive test available to demonstrate engine cleanliness is an engine test itself. The preferred test for determining sludge forming tendency is designed to simulate stop-and-go driving. In this test, the engine is operated intermittently at high and low speeds. It is impracticable to run the test on heavy naphthas alone. Such naphthas are generally blended with virgin naphthas and butane, which have excellent engine cleanliness characteristics, in order to obtain a blend of suitable volatility for testing. Both the raw and the hydrofined thermal re-

formates were tested in blends containing 46% reformate, 4% butane and 50% of light virgin naphtha.

The results of the engine test are expressed on the basis of demerit rating, the higher the demerit rating, the dirtier the gasoline. The rating is based on visual appearance of various parts of the engine. The demerit rating on the blend containing the raw thermal reformate was 1.6, compared to 0.6 on the blend containing the hydrofined reformate. These values are representative of very dirty and very clean gasolines, indicating that mild hydrofining of the thermal reformate had produced a marked effect on engine cleanliness characteristics.

Other operations were conducted wherein hydrofining tests of different severity were conducted. The results of these operations are as follows:

Table II

HYDROFINING THERMAL REFORMATE (250° F. TO 430° F.)

[Cobalt molybdate catalyst, 200 p. s. i. g.]

Column	A	B	C	D	E	F	G	H	I	J
Catalyst Sample	Feed	A						B		
Operating Conditions:										
Temperature, ° F		600	600	600	600	500	700	595	595	565
Feed Rate, v./v./hr		1	4	8	16	1	8	5.9	7.8	8.5
Fresh H_2, S. C. F. B		200	200	200	200	200	200	580	180	170
H_2 Purity, Percent		70	70	70	70	70	70	100	100	100
Total H_2, S. C. F. B		1,000	1,000	1,000	1,000	1,000	1,000	1,400	810	750
Product Inspections:										
Bromine No. cgs./g	18	4	12	15	16	11	11	8	10	15
Diene No	1.32	0	0.10	0.13	0.56	0.17	0.17			0.08
Diazonium Fluoborate—										
Optical Density	0.33	0	0	0.02	0.07	0	0			
Deposit Factor	30	0	0	2.0	6.9	0	0			1
Phenol No	390	3	15	42	98	9	26			33
Sulfur, Wt. Percent	0.050	0.016	0.019	0.030	0.038	0.051	0.042			0.04
CFRR Octane No.:										
Clear	75.6	69.4	73.9	75.0	75.3	72.4	73.7	71.9	73.1	75.0
2 cc. TEL	84.5	81.7	84.3	84.4	84.5	82.7	84.5	83.2	83.9	84.1

In practicing the present invention it is desirable that the hydrofining conditions are such that little or no loss of octane number results. In the hydrofining operation olefins are converted to paraffins and this tends to reduce octane number. On the other hand, sulfur is removed from the product so that the lead susceptibility is increased, and this tends to raise the leaded octane number of the product. It is important to so adjust the conditions that the loss of octane number by conversion of olefins to paraffins is substantially balanced by the improved lead susceptibility of the hydrofined product. Referring to column B of Table II, it is apparent that when thermal reformate was hydrofined at 600° F., a feed rate of 1 v./v./hr. bromine number was reduced from 18 to 4, and sulfur from .05 to .016 weight percent. The overall result of this change was that the leaded octane number was reduced from 84.5 to 81.7. In columns D and E it is apparent that when the feed rate was increased to 8 or 16 v./v./hr., the bromine number was reduced only to 15 to 16 and the sulfur to .030 to .038. The leaded octane number of the products from these operations was identical of that of the feed showing that the loss in octane number from conversion of olefins to paraffins had been substantially counterbalanced by the increased lead susceptibility resulting from sulfur reduction. It is this balance of conditions with which the invention is primarily concerned. The operating conditions illustrated in column C are intermediate between those shown in columns B and D and the results as illustrated by bromine number, sulfur removal and leaded octane number are likewise intermediate. Comparison of columns B and F shows that by operating at 500° F. in place of 600° F., a substantially higher bromine number is obtained. Comparison of columns G and D shows that by increasing the temperature to 700° F. by maintaining feed rate constant, the bromine number of the product is reduced illustrating that temperature can be used to increase the severity of the hydrofining.

For the operations described in columns H, I, and J, a new sample of catalyst was used which had the same composition as the catalyst used in columns B through G, but had a slightly higher activity. When the thermal reformate feed stock was treated with this catalyst at 595° and 5.9 v./v./hr., the bromine number of the product was 8, indicating that excessive saturation of olefins had taken place. In an attempt to alleviate this situation, the feed rate was increased to 7.8 v./v./hr., but this raised the bromine number to a value of 10 which was still lower than desirable. A further change in operating conditions was made to reduce the temperature from 595° to 565° F., and to increase the feed rate to 8.5 v./v./hr. At these operating conditions the bromine number was brought to 15 which is considered a satisfactory level for this operation. These changes illustrate the variables which can be changed to achieve the desired mild hydrofining conditions. In general, it is desirable to adjust the hydrofining conditions so that the bromine number reduction is limited to about 33%, and preferably to about 20% to assure minimum loss in leaded octane number. In Table II the feed stock used was a heavy thermal reformate obtained by thermal reforming of a heavy virgin naphtha. The invention may also be practiced using a heavy catalytic naphtha, for example, one produced by fluid catalytic cracking of a heavy gas oil. Hydrofining results obtained with a feed of this type are shown in Table III.

Table III

HYDROFINING HEAVY CATALYTIC NAPHTHA

[194° F. to 437° F.]

Feed Rate, v./v./hr	Feed	1	4	8
Bromine Number	23	6	13	17
Sulfur, wt. percent	0.24	.02	.05	.07
General Motor Gum, mg./100 ml.[1]	3	1	1	1
DFDF	16	0	0	3
CFRR Octane Number:				
Clear	92.5	87.0	89.0	90.4
2 cc. of TEL	96.1	93.6	95.0	95.4

[1] 2 lb. UOP #5 inhibitor/5,000 gal.

As the feed rate was increased from 1 to 8 v./v./hr., bromine number was increased from 6 to 17, and sulfur from .02 to .07 weight percent. For the operation of 8 v./v./hr., the leaded octane number of the product was 95.4 compared to 96.1 for the feed, showing that the

mildest type of hydrofining showed the smallest loss in octane rating.

While the operation has been described with respect to heavy cracked naphthas, it is also very desirable for the treatment of light naphthas secured from a catalytic cracking operation boiling in the range from about 130° F. to 330° F. It has been discovered that if this particular naphtha is hydrofined as described, the combustion chamber deposits are materially reduced.

EXAMPLE II

A number of operations were carried out wherein a catalytically cracked light naphtha was hydrofined as described. The results of these operations are as follows:

Table IV

HYDROFINING CATALYTIC LIGHT NAPHTHA (130° F.–330° F.) OVER $C_oM_oO_4$ CATALYST

Operation	1	2	3
Catalyst Age, Hours		128	134
Operating Conditions:			
Temperature, ° F		600	600
Feed Rate, v./Hr./v.	[1] Feed	4.0	15.7
H_2 Rate, S. C. F./B.	1,000 (Nominal)	1,000 (Nominal)	1,000 (Nominal)
Pressure, p. s. i. g.	200	200	200
Product Inspections:			
Bromine Number, cgs./gm.	91	55	80
Sulfur, wt. percent	0.14	0.049	0.110
CFRR Octane Number:			
Clear	93.5	88.6	92.1
+2 cc. TEL	97.7	95.3	97.0

[1] IBP—118° F., 10% at 154° F., 50% at 219° F., 90% at 315° F., 363° FEP.

The results shown in column 2 of Table IV show that when hydrofining at a feed rate of 4 v./v./hr., bromine number was reduced from 91 to 55 and sulfur from .14 to 0.49. Under these conditions the leaded octane number of the product was 2.4 units lower than that of the feed. As shown in column 3 when the feed rate was increased to 15.7 v./v./hr., the bromine number was reduced only to 80 and sulfur to .110 weight percent. The product obtained under these conditions had a leaded octane number only 0.7 unit less than that of the feed. These data illustrate that mild hydrofining can be applied to light cracked naphthas as well as to heavy cracked naphthas with substantially no loss in octane number provided conditions are carefully regulated to minimize conversion of olefins to paraffins as measured by loss in bromine number.

What is claimed is:

1. Process for the production of a high quality motor fuel which comprises distilling a crude oil to segregate a hydrocarbon fraction boiling in the range from about 100° F. to 330° F., a hydrocarbon fraction boiling in the range of about 250° F. to 430° F., and a hydrocarbon fraction boiling in the gas oil boiling range, thermally reforming said hydrocarbon fraction boiling in the range from about 250° F. to 430° F., distilling the product from said thermal reforming operation to segregate a light thermal naphtha fraction and a heavy thermal naphtha fraction, hydrofining said heavy thermal naphtha fraction under conditions to secure a bromine number reduction of less than 20%, catalytically cracking said gas oil fraction to produce a catalytically cracked naphtha, thereafter blending said naphtha fraction boiling in the range from 100° F. to 330° F., said light thermal naphtha, said hydrofined heavy thermal naphtha, and said catalytic naphtha, whereby a high quality gasoline blend having improved engine cleanliness characteristics is secured.

2. Process for the production of hydrocarbon constituents boiling in the motor fuel boiling range having improved engine cleanliness which comprises distilling a crude oil to segregate a hydrocarbon fraction boiling in the heavy naphtha boiling range, reforming said fraction under conditions to raise the octane number thereof, thermally distilling said fraction to secure a light thermal naphtha and a heavy thermal naphtha, thereafter hydrofining said heavy thermal naphtha in the presence of extraneously generated hydrogen, at a temperature in the range of 400° to 700° F., at a pressure in the range of 50 to 250 pounds per square inch, at a throughput of about 1 to 20 v./v.hr., employing a catalyst selected from the group consisting of cobalt molybdate on a carrier, cobalt oxide, molybdenum oxide, tungsten sulfide and nickel sulfide, under conditions to secure a positive consumption of hydrogen not in excess of 60 standard cubic feet per barrel securing a bromine number reduction of less than 20%.

3. Process as defined by claim 2 wherein said heavy thermal naphtha boils in the range from about 250° F. to 430° F.

4. Process for the production of hydrocarbon constituents boiling in the motor fuel boiling range having improved engine cleanliness which comprises distilling a crude oil to segregate a hydrocarbon fraction boiling in the gas oil boiling range, catalytically cracking said fraction under conditions to secure hydrocarbon constituents boiling in the motor fuel boiling range, distilling said latter fraction to secure a light catalytic naphtha and a heavy catalytic naphtha, thereafter hydrofining said heavy catalytic naphtha in the presence of extraneously generated hydrogen, at a temperature in the range of 400° to 700° F., at a pressure in the range of 50 to 250 pounds per square inch, at a throughput of about 1 to 20 v./v./hr., employing a catalyst selected from the group consisting of cobalt molybdate on a carrier, cobalt oxide, molybdenum oxide, tungsten sulfide and nickel sulfide, under such conditions to secure a positive consumption of hydrogen not in excess of 60 standard cubic feet per barrel securing a bromine number reduction of less than 20%.

5. Process as defined by claim 4 wherein said heavy catalytic naphtha boils in the range from about 250° to 430° F.

6. Process for the production of hydrocarbon constituents boiling in the motor fuel boiling range having improved combustion chamber characteristics which comprises distilling a crude oil to segregate a hydrocarbon fraction boiling in the gas oil boiling range, thereafter catalytically cracking said gas oil to produce hydrocarbon constituents boiling in the motor fuel boiling range, segregating a light hydrocarbon fraction from said constituents, thereafter hydrofining said light fraction in the presence of extraneously generated hydrogen, at a temperature in the range of 400° to 700° F., at a pressure in the range of 50 to 250 pounds per square inch, at a throughput of about 1 to 20 v./v.hr., employing a catalyst selected from the group consisting of cobalt molybdate on a carrier, cobalt oxide, molybdenum oxide, tungsten sulfide and nickel sulfide, under conditions to secure a positive consumption of hydrogen not in excess of 60 standard cubic feet per barrel securing a bromine number reduction of less than 20%.

7. Process as defined by claim 6 wherein said light fraction boils in the range from about 100° to 330° F.

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