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PROCESS FOR PREPARING AN IMPROVED LUBRICATING OIL

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This invention relates to the preparation of multigrade lubricating oils and in particular relates to procedure whereby multigrade lubricating oils can be prepared by the simultaneous treatment of a mixture of a hydrocarbon wax and a liquid lubricating oil stock.

It is well known that paraffin wax can be isomerized into improved lubricating oils. See, for instance, Patents 2,668,790, 2,668,866 and 2,817,693. This does not result in conversion to a multigrade lubricating oil since the viscosity of the isomerized product is entirely too low to serve as a source of multigrade lube oil components; i.e., the viscosity of the product is below that of 10W SAE oil in most cases and below 10W–20 multigrade oil in every case. It is also known that certain liquid lubricating oil stocks can be converted into high quality multigrade lubricating oils by treatment with hydrogen in the presence of certain catalysts at elevated temperature and pressure. See United States Patent 2,960,458, issued November 15, 1960, Beuther et al. The procedure described in this patent has not been considered as satisfactory procedure for conversion of hydrocarbon waxes. As a matter of fact in this application it is indicated that any small amount of wax which might be present is not converted.

This invention has for its object to provide improved procedure for the preparation of multigrade lubricating oils by hydrogen treatment of a mixture of a liquid lubricating oil stock and a relatively large amount of a hydrocarbon wax. Another object of this invention is to provide improved procedure for the simultaneous conversion of a hydrocarbon wax and a liquid lubricating oil stock into multigrade lubricating oils. Other objects will appear hereinafter.

These and other objects of our invention are accomplished by subjecting a hydrocarbon mixture to treatment with hydrogen at a pressure above about 1750 p.s.i. at a temperature between about 715° and 825° F. at a space velocity between about 0.2 and 4.0 in the presence of a sulfide of a metal of group VI of the periodic system mixed with a sulfide of the iron group and supported on a carrier having substantial cracking activity. The hydrocarbon charge stock which is thus treated with hydrogen is a mixture of (1) a deasphalted residuum and/or an unpressable distillate having a viscosity index between about 60 and 100 and a viscosity at 210° F. of between about 70 and 250 S.U.S., and (2) a hydrocarbon wax. The hydrocarbon wax component comprises between about 12 and 80 percent of the hydrocarbon mixture. This treatment results in conversion of both the liquid hydrocarbon and the wax contained in the original charge stock mixture into high quality multigrade lubricating oil components. The reaction product is then subjected to a treatment for separation of multigrade lubricating oil components therefrom.

We will first discuss the properties of the liquid lube oil portion of the charge stock. In order to obtain high yields of multigrade oils of the desired viscosity it is essential to employ a residual or unpressable distillate liquid charge stock. This is because the conditions necessary to give a multigrade lube oil cause considerable ring scission with formation of lower boiling products. The liquid portion of the starting material may be any de-

asphalted residuum obtained by vacuum or like distillation of any crude petroleum or residual fraction thereof or any unpressable distillate which has a V.I. of between about 60 and 100 and a viscosity of 210° F. of between about 70 and 250 S.U.S. Thus for instance the charge stock may be prepared by vacuum distillation of a Pennsylvania, Mid-Continent, West Texas, Kuwait, etc. crude to obtain an unpressable distillate or an undistilled residue, which requires deasphalting. We have found that deasphalting with agents such as sulfuric acid, phenol, sulfur dioxide, etc. results in removal of components which, upon hydrogenation, have desirable properties for a multigrade oil product. Therefore if these materials are employed for asphalt removal, the yield of multigrade oils will be markedly lower. A higher carbon residue will result in undesirable shortening of the life of the catalyst under the relatively severe hydrogen treatment conditions employed to produce the multigrade oil. For this reason we prefer to employ liquid charge stocks having a low carbon residue such as below about 4.0 (Conradson). However, higher carbon residue charge stock may be used if catalyst life is not of great importance or a rugged catalyst is used.

The utilization of a liquid portion of the charge stock which has a V.I. of above about 60 is essential if a reasonable yield of a multigrade oil is to be obtained. A charge having a V.I. between about 60 and 100 is advantageously used. The high V.I. products, necessary for multigrade lubricating oil, are produced in good yield only if a charging material of at least about 60 V.I. is employed. Excessive ring scission to obtain the V.I. required for multigrade oils will drastically lower yields if a lower than 60 V.I. charge stock is used. A viscosity of between about 70 and 250 S.U.S. at 210° F. is necessary so that the multigrade oil product will have the proper viscosity after the hydrogen treatment.

The hydrocarbon wax components of the charge stock mixture may be any form of hydrocarbon wax provided that the wax components have a melting point above about 120° F. The charge stock may be a slack wax which contains varying amounts of hydrocarbon liquid in addition to the wax components. On the other hand the wax portion of the charge stock mixture may be a hard paraffin wax of a refined type. Microcrystalline wax is also satisfactory wax. These waxes may be subjected to various purification treatments such as precipitation from solvents such as methyl-ethyl ketone, filtration, hydrogenation, etc. As a matter of fact one of the preferred embodiments of our invention includes the use of a wax which has been unconverted in our process. In other words, the unconverted hydrocarbon wax or the wax which has been altered by the hydrogen treatment to an extent insufficient to convert it is separated from the reaction products and this wax is returned to the hydrogen treatment process. When practicing such return, the returned or recycled wax is considered as part of the 12 to 80 percent wax in the charge stock. A wax content of between about 15 and 50 percent is preferred. If below about 12 percent wax is present in the charge stock, the conversion of the wax is uneconomically low. If more than about 80 percent of the wax is present in the charge, the wax is converted, under our reaction conditions, into uneconomically large amounts of hydrocarbons having such low boiling points and viscosities that they are unsatisfactory for a multigrade lube oil product. It appears that under our reaction conditions the wax is hydrocracked rather than isomerized and that excessive amounts of wax result in excessive cracking to light products.

It is essentail to employ a catalyst which contains a metal sulfide of group VI left-hand column of the periodic system mixed with an iron group metal sulfide. Thus sulfides of tungsten, molybdenum or chromium mixed

with sulfides of nickel, cobalt or iron may be used. A mixture of tungsten and nickel sulfides is preferred. These catalysts are composited with a porous carrier which has substantial cracking activity, i.e., a cracking activity of above about 12 as determined by the M. W. Kellogg test described below. Examples of satisfactory carriers are any of the well known catalysts conventionally employed for catalytic cracking. Thus the carrier may be a natural or synthetic high silica-low alumina catalyst conventionally used for catalytic cracking. These cracking carriers may contain magnesia, zirconia, etc. in place of alumina. Such catalysts are known to be advantageous cracking catalysts. Recently catlaytic cracking has been improved by utilizing a catalyst which has a relatively high alumina content as compared with previously employed silica-alumina cracking catalysts. It is satisfactory to employ these more recent silica-alumina cracking catalysts which may contain as much as 50 percent alumina. "H–44 alumina," manufactured by Aluminum Company of America, has a cracking activity of 14.1 on the Kellogg scale and is a satisfactory carrier. However, all activated aluminas are not satisfactory. Thus Harshaw activated alumina has an activity of about 5 on the Kellogg scale and yields a much poorer product having insufficiently high V.I. to serve as a source of multigrade oil. The catalyst may contain between about .1 to 5 atoms of iron group metal per atom of group VI metal.

These catalysts may be composited with the carrier utilizing any known catalyst preparation method. Thus, for instance, the two metal sulfide components may be deposited on the carrier by conventional impregnation techniques. Alternatively, they may be deposited on the carrier by precipitation techniques. Both metal components may be simultaneously deposited, or if desired, they may be deposited in sequence. Co-precipitation of the carrier and catalyst components may also be practiced. Ordinarily water soluble salts of the group VI and iron group catalyst components such as nitrates, oxalates, etc. will be employed and after the impregnation has been completed these salts will usually be converted into the corresponding metal oxides by calcining. Formation of the sulfide can be carried out in any well known manner. One satisfactory procedure is to contact the catalyst containing the oxides of the metals with a mixture of hydrogen sulfide and hydrogen at elevated temperature. Complete sulfiding is not necessary. However, sulfiding to above about 15 percent is desirable. All of these procedures are well known in the prior art and details thereon will not be gone into. The catalyst may contain 5 to 40 percent and preferably 10 to 25 percent of total group VI and iron group metals. The catalyst may contain a halogen such as fluorine or chlorine as an activating agent. These catalysts can all be regenerated by combustion in a conventional manner. Regeneration usually will not be required until after the throughput (volumes of charge per volume of catalyst prior to regeneration) of about 5000. After each regeneration the catalyst is presulfided as described above.

The cracking activity of the carrier can be conveniently defined by relating it to the Kellogg cracking activity scale, developed by the M. W. Kellogg Company. This scale defines cracking activity as percent by volume of conversion obtained by passing a standard charge stock through the catalyst under standard test conditions. The Kellogg cracking activity scale is explained in "Physical, Chemical and Catalytic Testing of Diakel Powdered Cracking Catalyst," a technical report of the Petroleum Research Division of the M. W. Kellogg Company, dated June 7, 1943. The carrier to be employed should be one which has an activity for cracking equivalent to a rating of at least 12 percent on the Kellogg scale. A rating of between 12 and 80 is in general satisfactory. A rating above about 45 is advantageous. These values relate to the cracking activity of the carrier in an unpromoted state.

To determine the Kellogg cracking activity of a catalyst, the catalyst is tested as a powder under the following cracking conditions:

Feed	35° A.P.I. Mid-Continent gas oil.
Catalyst temperature	850±5° F.
Pressure	Atmospheric.
Catalyst charge	710 grams.
Oil rate	500±20 cubic centimeters per hour.
Velocity, inlet conditions	Approximately 0.1 foot per second.
Weight of oil per hour per weight of catalyst bed	0.6±0.02.
Length of cracking test	2 hours.
Blowdown nitrogen	3 cubic feet per hour (0.2 foot per second).

The oil feed used in the cracking test is a light Mid-Continent gas oil with the following typical inspections.

Gravity, ° A.P.I.	34.8
A.S.T.M. distillation, ° F.:	
I.B.P.	468
5%	512
10%	521
20%	534
30%	546
40%	562
50%	578
60%	595
70%	618
80%	647
90%	686
95%	720
E.P.	748
Aniline point, ° F.	171
Sulfur, weight percent	0.29

The allowable variations of oil feed inspections are as follows:

Gravity, ° A.P.I.	35±1
A.S.T.M. distillation, ° F.:	
10%	520±10
50%	580±10
90%	690±10
E.P.	750±25

The catalyst to be tested is heat treated at 850° F. for a two-hour period before testing. This heat treatment is accomplished by filling a steel dish with 1100 grams of the catalyst under investigation and inserting it into a circulating air muffle furnace which has been preheated to 850± 5° F. The catalyst should remain in the circulating air muffle furnace for two hours with the air stream flowing. The catalyst is then removed from the furnace.

The powdered catalyst test apparatus consists of a tubular reactor with a preheating coil and filter, a furnace, oil feed tank and pump, condenser, receiver and knock-back trap, gas meter and accessory equipment. In operating this test equipment, the reactor and preheating coil is mounted within the furnace and oil is pumped from the feed tank through transfer valves into the preheater coil. Oil vapors enter the reactor through a small orifice at the bottom of the fluid bed and flow upward. The cracked products leaving the bed pass into an enlarged settling zone, through a filter in the top of the reactor and through a condenser into a receiver situated in an ice water bath. Gases leaving the receiver pass through a knock-back column cooled to −40° F. and then through a gas meter to a product gas holder.

The test reactor consists of a section of 1¼ inch pipe which is 4 feet 9 inches in length, surmounted by a 6-inch section of 2-inch pipe containing a glass wool filter. A preheater coil consisting of 10 feet of ¼ inch O.D. tubing

is wound on the outside of the 1¼ inch pipe and connects with a small orifice in the conical bottom attached to the latter.

In preparing for the test, nitrogen is passed through the preheater coil and the reactor at a rate of 2 cubic feet per hour which is approximately equivalent to the oil vapor rate during the run. The catalyst is then slowly charged into the reactor and the reactor is then secured within the heated furnace. The receiver in the recovery system is held at 32° F. with wet ice and the knock-back traps are held at −40° F., with a 50–50 mixture of ethyl glycol and water cooled with Dry Ice.

A two-hour cracking test is then conducted under the conditions outlined above employing a charge stock as specified. After this test is concluded, a nitrogen blow-down of 3 cubic feet per hour should be continued for 30 minutes. The liquid product is then drawn from the receiver into a chilled bottle, weighed and placed in an ice box. A few minutes should be allowed for any liquid holdup in the knock-back to drain out. The reactor is then removed from the furnace and the catalyst is poured into a container and weighed.

At the completion of the cracking test, three products are available for analysis—total liquid, total gas and spent catalyst. The specific gravity of the liquid product expressed as ° A.P.I. should be taken at 35° to 40° F. according to A.S.T.M. procedure Serial No. D–287–39t. The distillation of the liquid test product should be carried out according to A.S.T.M. method D86–40 appearing in "Distillation of Gasoline, Naphtha, Kerosene and Similar Petroleum Products" (the distillation procedure to be employed for the gas oil charged to the test unit is A.S.T.M. test D158–4 appearing in "A.S.T.M. Standards for Petroleum Products and Lubricants"). The analysis of the gas products from the test unit which consist of carbon dioxide, hydrogen sulfide and air should be carried out accordiing to the Orsat method. A gas density determination should be made by the Edwards balance method. A carbon analysis determination of the spent catalyst is made by burning the sample in a stream of oxygen, absorbing the CO_2 produced and determining the weight of CO_2 absorbed. It may be necessary to extract oil from the catalyst prior to the carbon analysis. This is accomplished by washing with 100 to 150 cubic centimeters alcohol followed by 100 to 150 cubic centimeters of 95 percent carbon tetrachloride. This is followed by drying in an oven at 375° F. to 400° F. overnight. After drying, the carbon content of the extracted catalyst is then determined. The amount of oil extracted is determined by evaporating the extract until no trace of carbon tetrachloride or alcohol is detected. The residue remaining is the oil removed from the catalyst.

A weight balance should be made. One hundred times the total weight of liquid product plus gas product plus carbon divided by the weight of oil feed is the weight balance in percent. For a test unit operation to be acceptable, the weight balance should be between 95 and 100 percent.

The Kellogg activity rating of the catalyst is expressed as volume percent conversion obtained under the standard test conditions. The activity rating can be calculated from the test results as follows:

$$\frac{\text{Total liquid product (grams)}}{\text{Liquid product specific gravity}} = \text{milliliters liquid product}$$

$$\frac{\text{Liquid product (milliliters)} \times \text{volume percent distillate plus loss at 400° F.}}{100} = \text{milliliters gasoline}$$

$$\frac{\text{Total oil feed (grams)}}{\text{Feed specific gravity}} = \text{milliliters oil feed}$$

$$\frac{\text{Milliliters gasoline}}{\text{Milliliters oil feed}} \times 100 = \text{gasoline yield volume percent}$$

$$\text{Milliliters liquid product} - \text{milliliters gasoline} = \text{milliliters cycle oil}$$

$$\frac{\text{Milliliters cycle oil}}{\text{Milliliters oil feed}} \times 100 = \text{cycle oil volume percent}$$

$$100 - \text{volume percent cycle oil} = \text{conversion volume percent} = \text{Kellogg cracking activity in percent}$$

The reaction conditions specified are necessary to obtain the conversion into a multigrade product or to a product containing the multigrade components. If a temperature much below 715° F. is employed, the conversion to multigrade oil will be insufficient to be commercially attractive. On the other hand if a temperature much above 825° F. is employed, the conversion into materials having too low a viscosity for multigrade oil will be excessive. Within the ranges specified the temperature and space velocity can be interchanged to give about the same results. In other words a low temperature with a low space velocity will give about the same yield and quality of multigrade oil as a higher temperature and higher space velocity. Pressures of below about 1750 p.s.i.g. do not result in sufficient conversion nor as selective a conversion and give an undesirably short catalyst life. Much higher pressures such as 5000 or even 10,000 may be employed but are not commercially attractive since they are more costly to produce and maintain and do not result in much if any improvement in yields or product quality. A pressure of between about 2000 and 4000 p.s.i. is preferred since such pressures result in longer catalyst life and are most economical.

Pure hydrogen may, of course, be used. However, hydrogen of lower purity such as reformer hydrogen works very well. If an impure hydrogen is used it is recommended that part of the recycled hydrogen be bled from the recycle stream or that a recycle hydrogen clean-up procedure be used. The hydrogen may be circulated in a ratio of between about 2500 and 10,000 s.c.f. per barrel of charge. Higher or lower amounts of hydrogen can be employed. Water should be maintained at as low a level as possible in the hydrogen or other materials used in the process since it has a deactivating effect on the catalyst.

The product from the hydrogenation step will contain lower boiling reaction products such as gasoline, furnace oil and gases which are unsuitable for multigrade oils. Also any unconverted wax present in the product is undesirable in a lubricating oil. Therefore it is necessary to remove these materials from the product. The gasoline and furnace oil can be removed by stripping or distillation. Removal of wax, if present, is accomplished by any treatment conventionally used for dewaxing ordinary lube oils. This procedure is advantageously carried out to give a pour point of between about −5° and +5° F. An example of satisfactory dewaxing treatment is dissolving the oil in a solvent such as methyl ethyl ketone and/or toluene, propane, etc. and cooling and filtering off the wax. Thereafter the solvent is removed by distillation. If a lower pour point than that specified above is desired, it can be conveniently obtained by addition of a small amount of any conventional pour point depressant such as "Acryloid 618" which is a polymethacrylate or by addition of polyisobutylenes. The unconverted wax which is separated may be recycled as pointed out above.

The hydrogenated and dewaxed (if dewaxing is applied at this stage) product may be distilled to directly separate fractions which have the properties of multigrade oils. Thus the product may be distilled to separate fractions having the maximum and minimum S.A.E. viscosity requirements for the multigrade oil to be produced; for instance for a 10W/20 multigrade oil a maximum vis-

cosity at 0° F. of less than 12,000 S.U.S. and a viscosity at 210° F. of between 45 and 58 S.U.S.; for a 20W/30 multigrade oil a maximum viscosity at 0° F. of less than 48,000 S.U.S. and a viscosity at 210° F. of between about 58 and 70 S.U.S.; for a 20W/40 multigrade oil a maximum viscosity at 0° F. of less than 48,000 S.U.S. and a viscosity at 210° F. of between about 70 and 85 S.U.S.; and for a 10W/30 multigrade oil a maximum viscosity at 0° F. of less than 12,000 S.U.S. and a viscosity at 210° F. of between about 58 and 70 S.U.S. On the other hand it is satisfactory to distill the hydrogenated product to obtain separate fractions for blending to obtain a blended 10W/20 multigrade oil or a blended 10W/30 or 20W/40 etc. multigrade oil which meet the S.A.E. specifications for these multigrade oils. When utilizing this method of separating and recombining portions of fractions of the hydrogenated product, it is probable that some of the lubricating oil fractions in the 20W/30 or 20W/40 S.A.E. range will be added to the lower S.A.E. range oils and vice versa to obtain a blended product having the desired characteristics. The procedure for blending is well known to lubricating oil chemists. Regardless of which distillation method is used, light non-lubricating portions of the product such as gases, gasoline, furnace oil, and hydrocarbon wax, if not previously removed, should be removed or separated from the lube oil fractions. The furnace oil has good burning characteristics and the gasoline is a good stock for hydrogen reforming. The distillation should be conducted so as to avoid thermal decomposition as much as possible. Vacuum and/or distillation with an inert gas such as steam is advantageously employed.

EXAMPLE

In the three test series of this example an Ordovician residuum having a gravity (degrees A.P.I.) of 23.6; a V.I. of 92 and an I_2 number of 13.7 was used as the liquid portion of the charge stock. This Ordovician residuum contained 12 percent by volume of natural wax. The same catalyst was used in all of these tests and was prepared as described below. In the first series of tests this residuum was subjected to treatment with hydrogen under the conditions given in columns 1, 2 and 3 of Table I. The products obtained by this hydrogen treatment of Ordovician residuum were subjected to distillation to remove portions boiling below 725° F. The undistilled portion was then dewaxed at 0° F. using methyl-ethyl ketone. The yields and the inspections on the dewaxed products are given in columns 1, 2 and 3 of Table I.

In the second series of tests 15 percent hydrotreated wax obtained by precipitation from a methyl-ethyl ketone solution of hydrotreated Ordovician residuum was added to the Ordovician residuum described above. Therefore the charge stock was the same as in the first series of tests (columns 1, 2 and 3) except that the charge stock contained 15 percent of added hydrotreated wax to make a total wax content of 27 percent. The results of this series of tests are given in columns 4 and 5 of Table I.

In a third series of tests 40 percent of hydrotreated wax was added to the Ordovician residuum. Therefore the charge stock in the third series of tests was the same as in the first and second series except that the charge stock contained a total of 52 percent wax. The results from the third series of tests are given in columns 6 and 7 of Table I.

Table I

Charge Stock	100% Ordovician Residuum			85% Ordovician Res., 15% Hydrotreated Wax		60% Ordovician Res., 40% Hydrotreated Wax	
Column Number	1	2	3	4	5	6	7
Operating Conditions:							
Temperature, ° F	730	741	750	750	730	730	750
Pressure, p.s.i.g	3,000	3,000	3,000	3,000	3,000	3,000	3,000
Space Velocity, v./v./hr	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Hydrogen Rate: s.c.f./bbl	5,000	5,000	5,000	5,000	5,000	5,000	5,000
Yield of 725° F. Product (Dewaxed):							
Percent by Vol. of total chg				51.9	52.8	49.2	44.6
Percent by Vol. of orig. chg. (residuum w./ natural wax)	69.8	59.5	57.2	61.7	62.8	84.3	76.4
Inspections of 725° F. Product (Dewaxed):							
Gravity, ° A.P.I	30.4	31.5	32.8	34.2	31.5	33.1	35.6
V.I	110	116	120	125	116	121	132
I_2 Number	2.9	2.8	2.8		2.4		
Yield of Wax: Percent Vol. total chg	10.9	9.0	7.9	11.0	24.6	27.3	15.4

In preparing the catalyst used in the above tests, Davison F–1–25 75 percent silica–25 percent alumina cracking catalyst was formed into 3/16-inch tablets to a calcined hardness of 20 pounds using 2 percent "Acrowax C" and 5 percent Du Pont "Elvanol 71–30" as a lubricant and binder, respectively. The tablets were broken to 10–20 mesh, and the granules were then calcined at 1000° F. for 60 hours in an electric muffle furnace to remove the lubricant and binder. One hundred (100) parts by weight of calcined granules were soaked for 15 minutes in 165 parts of a fluorine-aqueous solution containing 2.42 percent by weight fluorine. The wet mass was dried in an oven for 24 hours at 250° F. and then calcined in an electric muffle furnace for 10 hours at 1000° F.

An ammonium meta-tungstate solution was prepared by slurrying 100 parts by weight of C.P. finely divided tungstic acid ($WO_3 \cdot H_2O$) in 167 parts of distilled water at room temperature. To this slurry was added slowly and with mechanical stirring 11.8 parts of ammonia (28 percent NH_3) diluted with 47 parts of distilled water. The mixture was allowed to stand for about two hours at room temperature and then heated to near the boiling temperature and allowed to cool and stand overnight. The resulting slurry was filtered, and the solution was found to have a pH of 5 and contained about 24.5 percent WO_3. The solution contained 71.3 percent of the tungsten charged to the initial slurry. WO_3 content of the solution was increased to 46.5 percent by evaporation. The specific gravity of the final solution was 1.663. The NiW solution was prepared by dissolving 100 parts of

$$Ni(NO_3)_2 \cdot 6H_2O$$

in 342 parts of the 46.5 percent by weight WO_3 solution. The mixture then contained 35.5 percent WO_3 and 5.7 percent NiO, and had a specific gravity of 1.91. In carrying out the impregnation, 100 parts by weight of the fluorine impregnated silica-alumina was placed in a vessel which was evacuated to 6–10 mm. Hg, and then 230 parts of NiW solution added to cover the support. The contents were held under vacuum for five minutes and then at atmospheric pressure for ten minutes. The excess liquid was then filtered from the impregnated granules. The wet material was oven dried at 250° F. for 24 hours and then calcined in electric muffle furnace at 1000° F. for ten hours. The finished catalyst contained 23.6 percent tungsten, 4.3 percent nickel and 1.6 percent fluorine, and had a surface area of 118 m^2/g.

From the above data it will be noted that the presence of added wax materially increased the viscosity index and the yields at any given temperature. It will also be noted from a comparison of runs 4 and 5 that increasing the temperature with 27 percent wax did not reduce the yield, although such reduction in yield did take place when the wax content was 12 percent (runs 1, 2 and 3) and when the wax content was 52 percent (runs 6 and 7). It is for this reason that we prefer to employ a wax content of between about 15 and 50 percent. Thus if the wax content is above about 50 percent, the wax is hydrocracked to an undesirable extent resulting in formation of lower boiling materials which are unsuitable for multigrade lubricating oils. Comparison of column 3 with column 7 shows that the yield of multigrade oil from the original charge stock in column 7 was considerably higher than in column 3, although both processes were carried out at the same temperature. Since this yield of multigrade oil in column 7 was greater, i.e. 76.4 percent as compared with 57.2 percent, it is evident that presence of a large amount of wax causes increased conversion of the liquid portion of the charge stock.

The viscosity indices of all of the products produced in columns 4 to 7 inclusive were well above the minimum required for an SAE multigrade oil. These products could be used as multigrade oils. Alternatively distillation, to obtain blending stocks for producing blended multigrade oils, may be applied to these products.

We claim:

1. The process for preparing a multigrade lubricating oil from a hydrocarbon charge stock comprising essentially a mixture of (1) a high boiling petroleum fraction selected from the group consisting of deasphalted residuum and unpressable distillate having a V.I. between about 60 and 100 and a viscosity at 210° F. of between about 70 and 250 S.U.S. and a (2) hydrocarbon wax, the hydrocarbon wax component having a melting point above about 120° F. and constituting between about 12 and 80 percent of the hydrocarbon mixture which process comprises contacting said charge stock with hydrogen at a pressure above about 1750 p.s.i., at a temperature between about 715° and 825° F., at a space velocity between about 0.2 and 4.0 in the presence of a mixture of a sulfide of a metal of group VI and a sulfide of a metal of the iron group composited with a cracking catalyst having a cracking activity above about 12 on the Kellogg scale, whereby non-wax hydrocarbons contained in the original charge stock and the hydrocarbon wax components are converted into liquid hydrocarbons having the properties of a multigrade lubricating oil, and subjecting the product to a treatment for separation of multigrade lubricating oil components.

2. The process for preparing a multigrade lubricating oil from a hydrocarbon charge stock comprising essentially a mixture of deasphalted residuum having a V.I. between about 60 and 100 and a viscosity at 210° F. of between about 70 and 250 S.U.S. and paraffin wax, the paraffin wax component having a melting point above 120° F. and constituting between about 12 and 80 percent of the hydrocarbon mixture which process comprises contacting said charge stock with hydrogen at a pressure above about 1750 p.s.i., at a temperature between about 715° and 825° F., at a space velocity between about 0.2 and 4.0 in the presence of a mixture of a sulfide of a metal of group VI and a sulfide of a metal of the iron group composited with a cracking catalyst having a cracking activity above about 12 on the Kellogg scale, whereby non-wax hydrocarbons contained in the original charge stock and the paraffin wax components are converted into liquid hydrocarbons having the properties of a multigrade lubricating oil and subjecting the product to a treatment for separation of multigrade lubricating oil components.

3. The process for preparing a multigrade lubricating oil from a hydrocarbon charge stock comprising essentially a mixture of deasphalted residuum having a V.I. between about 60 and 100 and a viscosity at 210° F. of between about 70 and 250 S.U.S. and a hydrocarbon wax, the wax component having a melting point above about 120° F. and constituting between about 12 and 80 percent of the hydrocarbon mixture which process comprises contacting said charge stock with hydrogen at a pressure above about 1750 p.s.i. at a temperature between about 120° and 825° F., at a space velocity between about 0.2 and 4.0 in the presence of a mixture of a sulfide of a metal of group VI and a sulfide of a metal of the iron group composited with a silica-alumina cracking catalyst having a cracking activity above about 45 on the Kellogg scale, whereby non-wax hydrocarbons contained in the original charge stock and the paraffin wax components are converted into liquid hydrocarbons having the properties of a multigrade lubricating oil, subjecting the product to a treatment for separation of multigrade lubricating oil components and for separation of paraffin wax, and recycling the paraffin wax to the hydrogen treatment process.

4. The process for preparing a multigrade lubricating oil from a hydrocarbon charge stock comprising essentially a mixture of deasphalted residuum having a V.I. between about 60 and 100 and a viscosity at 210° F. of between about 70 and 250 S.U.S. and a hydrocarbon wax, the wax component having a melting point above about 120° F. and constituting between about 12 and 80 percent of the hydrocarbon mixture which process comprises contacting said charge stock with hydrogen at a pressure above about 1750 p.s.i., at a temperature between about 715° and 825° F. at a space velocity between about 0.2 and 4.0 in the presence of a mixture of tungsten and nickel sulfides composited with a cracking catalyst having a cracking activity above about 45 on the Kellogg scale, whereby non-wax hydrocarbons contained in the original charge stock and the paraffin wax components are converted into liquid hydrocarbons having the properties of a multigrade lubricating oil, subjecting the product to a treatment for separation of multigrade lubricating oil components and for separation of paraffin wax, and recycling the paraffin wax to the hydrogen treatment process.

5. The process for preparing a multigrade lubricating oil from a hydrocarbon charge stock comprising essentially a mixture of deasphalted residuum having a viscosity index between about 60 and 100 and a viscosity at 210° F. of between about 70 and 250 S.U.S. and a hydrocarbon wax, the wax component having a melting point above about 120° F. and constituting between about 15 and 50 percent of the hydrocarbon mixture which process comprises contacting said charge stock with hydrogen at a pressure between about 2000 and 4000 p.s.i., at a temperature between about 715° and 825° F., at a space velocity between about 0.2 and 4.0 in the presence of a mixture of a sulfide of a metal of group VI and a sulfide of a metal of the iron group composited with a cracking catalyst having a cracking activity above about 45 on the Kellogg scale, whereby both non-wax hydrocarbons and paraffin wax contained in the original charge stock are converted into multigrade lubricating oil components, subjecting the product to a treatment for separation of wax, and for separation of multigrade lubricating oil components having a viscosity between the maximum and minimum viscosity limits of a multigrade lubricating oil, and recycling the separated wax to the hydrogen treatment.

6. The process for preparing a multigrade lubricating oil from a hydrocarbon charge stock comprising essentially a mixture of deasphalted residuum having a viscosity index between about 60 and 100 and a viscosity at 210° F. of between about 70 and 250 S.U.S. and a hydrocarbon wax, the wax component having a melting point above about 120° F. and constituting about 27 percent of the hydrocarbon mixture which process comprises contacting said charge stock with hydrogen at a pressure between about 2000 and 4000 p.s.i., at a temperature between

715° and 825° F., at a space velocity between about 0.2 and 4.0 in the presence of a mixture of a sulfide of a metal of group VI and a sulfide of a metal of the iron group composited with a cracking catalyst having a cracking activity above about 45 on the Kellogg scale, whereby both non-wax hydrocarbons and paraffin wax contained in the original charge stock are converted into multigrade lubricating oil components, subjecting the product to a treatment for separation of wax, and for separation of multigrade lubricating oil components having a viscosity between the maximum and minimum viscosity limits of a multigrade lubricating oil, and recycling the separated wax to the hydrogen treatment.

7. The process for preparing a multigrade lubricating oil from a hydrocarbon charge stock comprising essentially a mixture of deasphalted residuum having a viscosity index between about 60 and 100 and a viscosity at 210° F. of between about 70 and 250 S.U.S. and paraffin wax, the paraffin wax component having a melting point above about 120° F. and constituting between about 15 and 50 percent of the hydrocarbon mixture which process comprises contacting said charge stock with hydrogen at a pressure between about 2000 and 4000 p.s.i., at a temperature between about 715° and 825° F., at a space velocity between about 0.2 and 4.0 in the presence of a mixture of a sulfide of a metal of group VI and a sulfide of a metal of the iron group composited with a silica-alumina cracking catalyst having a cracking activity above about 45 on the Kellogg scale, whereby both non-wax hydrocarbons and hydrocarbon wax contained in the original charge stock are converted into multigrade lubricating oil components, subjecting the product to a treatment for separation of wax, and for separation of multigrade lubricating oil components having a viscosity between the maximum and minimum viscosity limits of a multigrade lubricating oil, and recycling the separated wax to the hydrogen treatment.

8. The process for preparing a multigrade lubricating oil from a hydrocarbon charge stock comprising essentially a mixture of deasphalted residuum having a viscosity index between about 60 and 100 and a viscosity at 210° F. of between about 70 and 250 S.U.S. and paraffin wax, the paraffin wax component having a melting point above about 120° F. and constituting between about 15 and 50 percent of the hydrocarbon mixture which process comprises contacting said charge stock with hydrogen at a pressure between about 2000 and 4000 p.s.i. at a temperature between about 715° and 825° F., at a space velocity between about 0.2 and 4.0 in the presence of a mixture of sulfides of tungsten and nickel deposited upon a silica-alumina cracking catalyst having a cracking activity above about 45 on the Kellogg scale, whereby both non-wax hydrocarbons and paraffin wax contained in the original charge stock are converted into multigrade lubricating oil components, subjecting the product to a treatment for separation of paraffin wax, and for separation of multigrade lubricating oil components having a viscosity between the maximum and minimum viscosity limits of a multigrade lubricating oil, and recycling the separated paraffin wax to the hydrogen treatment.

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