



US 20080083648A1

(19) **United States**

(12) **Patent Application Publication**
Bishop et al.

(10) **Pub. No.: US 2008/0083648 A1**

(43) **Pub. Date: Apr. 10, 2008**

(54) **HEAVY LUBE OIL FROM
FISCHER-TROPSCH WAX**

(76) Inventors: **Adeana Richelle Bishop**, Vienna, VA
(US); **William Berlin Genetti**, Fairfax,
VA (US); **Nancy Marie Page**, Baton
Rouge, LA (US); **Loren Leon Ansell**,
Baton Rouge, LA (US); **Jack Wayne
Johnson**, Clinton, NJ (US)

Correspondence Address:
ExxonMobil Research & Engineering Company
P.O. Box 900
1545 Route 22 East
Annandale, NJ 08801-0900 (US)

(21) Appl. No.: **11/974,080**

(22) Filed: **Oct. 11, 2007**

Related U.S. Application Data

(63) Continuation-in-part of application No. 10/266,391,
filed on Oct. 8, 2002, now abandoned.

Publication Classification

(51) **Int. Cl.**
C10G 73/02 (2006.01)

(52) **U.S. Cl.** **208/28**

(57) **ABSTRACT**

A heavy lubricant base stock is made by (a) producing a synthesis gas from natural gas, (b) reacting the H₂ and CO in the gas in the presence of a cobalt Fischer-Tropsch catalyst, at reaction conditions effective to synthesize waxy hydrocarbons boiling in the heavy lubricant oil range, which are hydrodewaxed it at least two stages, with interstage separation and removal of the lighter material.

HEAVY LUBE OIL FROM FISCHER-TROPSCH WAX

[0001] This application is a Continuation-In-Part of U.S. Ser. No. 10/266,391 filed Oct. 8, 2002.

BACKGROUND OF THE DISCLOSURE

[0002] 1. Field of the Invention

[0003] The invention relates to a multi-stage process for producing heavy lube oil from Fischer-Tropsch wax. More particularly the invention relates to producing a heavy lubricant base stock from wax synthesized by reacting H₂ and CO produced from natural gas in the presence of a cobalt Fischer-Tropsch catalyst, by hydrodewaxing the wax in multiple stages, with interstage separation and removal of the lighter material.

[0004] 2. Background of the Invention

[0005] The relatively pure waxy and paraffinic hydrocarbons synthesized by the Fischer-Tropsch process, particularly over a cobalt catalyst which maximizes higher molecular weight hydrocarbon production, are excellent sources of premium lubricant oils, including heavy lubricant oil. The sulfur, nitrogen and aromatics content of the waxy hydrocarbons is essentially nil and the raw hydrocarbons can therefore be passed to upgrading operations, without prior hydrogenation treatment. In a Fischer-Tropsch process, the H₂ and CO in the synthesis gas feed react in the presence of the hydrocarbon synthesis catalyst to form waxy hydrocarbons which, when produced over a cobalt catalyst, contain substantial amounts of waxy, normal paraffinic hydrocarbons boiling in the lubricant oil range, including the heavy lubricant range of 850° F.+ (454° C.+). Fischer-Tropsch wax refers to the waxy hydrocarbon fraction typically removed from the synthesis reactor as liquid, but which is solid at ambient room temperature and pressure conditions. The heavy lubricant fraction requires deep dewaxing to produce a heavy lubricant base stock having acceptable cloud and pour points. Since it is solid wax at ambient conditions, solvent dewaxing processes cannot be used.

[0006] Various processes have been disclosed for catalytically dewaxing these and other waxy hydrocarbons. Many, such as those employing a ZSM-5 catalyst, dewax by hydrocracking the waxy hydrocarbons to products boiling below the lubricant oil range. Others require hydroprocessing to remove heteroatoms and aromatics. Illustrative, but nonlimiting examples of various catalytic dewaxing processes are disclosed in, for example, U.S. Pat. Nos. 6,179,994; 6,080,301; 6,090,989; 6,051,129; 5,689,031; 5,075,269 and EP 0 668 342 B1. The problem of severe hydroprocessing and low product yield is exacerbated when dewaxing a heavy lubricant oil fraction to an acceptable cloud point. There is a need for a process that will increase the yield of a dewaxed, heavy lubricant base stock produced from Fischer-Tropsch wax.

SUMMARY OF THE INVENTION

[0007] The invention relates to a process for producing a heavy lubricant base stock, by hydrodewaxing Fischer-Tropsch wax having heavy hydrocarbons boiling in the heavy lubricant oil range in multiple (e.g., at least two) stages, with interstage separation and removal of the lighter material. In another embodiment the invention relates to (a)

producing a synthesis gas from natural gas, (b) reacting the H₂ and CO in the gas in the presence of a cobalt Fischer-Tropsch catalyst, at reaction conditions effective to synthesize waxy hydrocarbons, including a fraction boiling in the heavy lubricant oil range, and (c) passing at least the heavy fraction to an upgrading facility in which the waxy, heavy lubricant oil hydrocarbon fraction is hydrodewaxed in at least two stages, with interstage separation and removal of the lighter material, to produce a heavy lubricant base stock. A process in which natural gas is converted to synthesis gas which, in turn, is converted to hydrocarbons, is referred to as a gas conversion process. Thus, this embodiment relates to a gas conversion process plus product upgrading by hydrodewaxing. The hydrodewaxing process comprises (i) hydrodewaxing the wax or waxy feed, to produce an isomeric fraction comprising a partially dewaxed heavy lubricant oil fraction and a lower boiling hydrocarbon fraction, (ii) separating these two fractions, and (iii) further hydrodewaxing the partially hydrodewaxed heavy lubricant oil fraction in at least one additional stage, to produce a heavy lubricant base stock. If more than two stages are used to produce the heavy lubricant base stock, lower boiling hydrocarbons formed in a stage are removed from the heavy fraction, before it is passed into the next stage. The waxy feed passed into the first hydrodewaxing stage may comprise hydrocarbons boiling in both fuel and lubricant oil ranges, only the lubricant oil range or only the heavy lubricant oil range. It is preferred in the practice of the invention, that the waxy feed passed into the first stage comprise hydrocarbons boiling below the lubricant oil boiling range (e.g., 600-650° F.—(316-343° C.—)). The dewaxed hydrocarbons comprising the heavy lubricant base stock have other uses, such as for example, a heavy white oil, a pharmaceutical oil, as a carrier or base for medicinal formulations, in chemical and pharmaceutical manufacturing, and the like.

[0008] By hydrodewaxing is meant the waxy feed and partially dewaxed heavy lubricant oil fraction are contacted with hydrogen and a hydrodewaxing catalyst that dewaxes mostly by hydroisomerization, and not by hydrocracking. This excludes dewaxing catalysts such as ZSM-5, which dewax mostly by hydrocracking the waxy molecules, particularly a heavy lubricant fraction, to hydrocarbons boiling below the desired product range. A hydrodewaxing catalyst comprising a ZSM-48 zeolite (ZSM-48 zeolites herein include EU-2, EU-11 and ZBM-30 which are structurally equivalent) component and a hydrogenation component, has been found to be particularly useful in the process of the invention and is used in at least one, and preferably all of the hydrodewaxing reaction stages. When a hydrodewaxing catalyst comprising a ZSM-48 zeolite is used in all the hydrodewaxing reaction stages, the yield of the liquid product is 5% greater, preferably 7% greater, still more preferably 10% greater, than when using other catalyst systems. By heavy lubricant fraction is meant hydrocarbons having an initial boiling point in the range of from about 850-950° F. (454-510° C.) and an end point above 1,000° F. (538° C.). A heavy lubricant base stock has an initial boiling point of at least about 850° F. (454° C.), an end point above 1,000° F. (538° C.), preferably above 1050° F. (566° C.), and cloud and pour points below those possessed by the raw, unprocessed, heavy fraction in the Fischer-Tropsch wax feed entering the first stage, and any other stages between the first and last hydrodewaxing stages, of the multistage hydrodewaxing process of the invention. The initial and end boiling point

values referred to herein are nominal and refer to the T5 and T95 cut points obtained by gas chromatograph distillation (GCD), using the method set forth below. By partially dewaxed heavy lubricant fraction is meant that the heavy lubricant fraction has been hydrodewaxed to lower its pour point below that which it had prior to being partially dewaxed, but not as low as the desired pour point, which is achieved by further dewaxing the partially dewaxed heavy fraction in the next one or more successive hydrodewaxing reaction stages.

[0009] The hydrogenating component of the hydrodewaxing catalyst will comprise at least one Group VIII metal and preferably at least one noble metal, as in platinum and palladium. The use of at least two reaction stages and a hydrodewaxing catalyst that dewaxes mostly by isomerization (e.g., a ZSM-48 component and a noble metal component), has been found to produce heavy lubricant base stocks, having acceptably low cloud and pour points, with relatively high product yield (e.g., with relatively low feed conversion to hydrocarbons boiling below the heavy lubricating oil range). Using only one hydroisomerization reaction stage, even with a hydrodewaxing catalyst comprising a ZSM-48 zeolite component and a noble metal hydrogenation component, has been found to convert substantially more of the heavy fraction, to lower boiling hydrocarbons and thereby produces less of the desirable heavy lubricant base stock oil. The multiple stage hydroisomerization process of the invention also permits each stage to operate at a lower temperature than that required for a single stage. This results in longer catalyst life and the production of a lubricant base stock containing less aromatics and other unsaturates, than is possible with a single stage. This means that at most only very mild hydrorefining of the base stock is required and this is another advantage of the process of the invention. The process of the invention is particularly useful for producing heavy lubricant base stock oils with low cloud and pour points. The use of a hydrodewaxing catalyst comprising a ZSM-48 zeolite component and a hydrogenation component, eliminates the need for hydrotreating or hydrorefining the raw, untreated Fischer-Tropsch wax feed to remove oxygenates prior to the dewaxing. The process of the invention eliminates the need for one or more separate hydrocracking, hydroisomerization, and catalytic or solvent dewaxing steps prior to hydrodewaxing, which are taught in the prior art and which substantially reduce product yield, particularly of the desired heavy lubricant base stock.

DETAILED DESCRIPTION

[0010] The hydrodewaxing process of the invention is described above as comprising at least two hydrodewaxing stages. The raw Fischer-Tropsch wax feed produced by a cobalt catalyst and preferably a non-shifting cobalt catalyst, need not be treated to remove aromatics, unsaturates or heteroatoms (including oxygenates), before it is passed into the first hydrodewaxing stage. This wax or waxy, Fischer-Tropsch synthesized hydrocarbons (these terms are used synonymously herein) is hydrodewaxed in a first stage, to produce an isomerate effluent comprising a partially dewaxed heavy lubricant oil fraction, and a lower boiling hydrocarbon fraction. This partially dewaxed heavy lubricant oil, which now has a cloud and pour point lower than the heavy lubricant fraction in the wax feed, is separated from the lower boiling hydrocarbons and passed into a second stage, in which it is further hydrodewaxed. The

second hydrodewaxing stage produces an isomerate effluent comprising (i) a heavy lubricant oil fraction having cloud and pour points lower than that produced in the first stage and (ii) lower boiling hydrocarbons. The heavy lubricant fraction is separated from the lower boiling hydrocarbons. In an embodiment in which only two stages are used, the heavy lubricant isomerate produced in the second stage comprises a heavy lubricant base stock having the desired cloud and pour points. If three stages are used, the separated second stage heavy lubricant isomerate is passed into a third stage for more hydrodewaxing, to produce the heavy lubricant base stock, and so forth. Thus, the wax feed is passed only into the first stage. The partially hydrodewaxed heavy lubricant oil isomerate fraction produced in the first stage sequentially passes into the second and any successive stages, with interstage separation of the heavy and lower boiling fractions, to produce a heavy lubricant base stock that has been hydrodewaxed to the desired pour and/or cloud points. By stage is meant one or more hydrodewaxing reaction zones, with no interzone separation of reaction products and typically, but not necessarily, refers to a separate hydrodewaxing reactor. A heavy lubricant base stock produced by this process is typically de hazed and/or hydrofinished at mild conditions, to improve color and stability, to form a finished lubricant base stock. As is known, haze is cloudiness or a lack of clarity, and is an appearance factor. Dehazing is typically achieved by either catalytic or absorptive methods to remove those constituents that result in haziness. Hydrofinishing is a very mild, relatively cold hydrogenating process, which employs a catalyst, hydrogen and mild reaction conditions to remove trace amounts of heteroatom compounds, aromatics and olefins, to improve oxidation stability and color. Hydrofinishing reaction conditions include a temperature of from 302 to 662° F. (150 to 350° C.) and preferably from 302 to 550° F. (150 to 288° C.), a total pressure of from 400 to 3000 psig. (2859 to 20786 kPa), a liquid hourly space velocity ranging from 0.1 to 5 LHSV (hr^{-1}) and preferably 0.5 to 3 hr^{-1} . The hydrogen treat gas rate will range from 250 to 10000 scf/B (44.5 to 1780 m^3/m^3). The catalyst will comprise a support component and one or more catalytic metal components of metal from Groups VIB (Mo, W, Cr) and/or iron group (Ni, Co) and/or noble metals (Pt, Pd) of Group VIII. The Groups VIB and VIII referred to herein, refers to Groups VIB and VIII as found in the Sargent-Welch Periodic Table of the Elements copyrighted in 1968 by the Sargent-Welch Scientific Company. The metal or metals may be present from as little as 0.1 wt. % for noble metals, to as high as 30 wt. % of the catalyst composition for non-noble metals. Preferred support materials are low in acid and include, for example, amorphous or crystalline metal oxides such as alumina, silica, silica alumina and ultra large pore crystalline materials known as mesoporous crystalline materials, of which MCM-41 is a preferred support component. The preparation and use of MCM-41 is disclosed, for example, in U.S. Pat. Nos. 5,098,684, 5,227,353 and 5,573,657.

[0011] In the practice of the invention, the interstage separation during hydrodewaxing may not be exact and, as a consequence, the separated heavy fraction passed into the next hydrodewaxing stage may contain minor (e.g., less than 50, preferably less than 25 and more preferably less than 5 wt. %) amounts of lower boiling hydrocarbons. It is preferred that, after the first stage, the amount of lower boiling hydrocarbons remaining in the separated heavy fraction

passed into the second any successive stages be as low as possible. This will be determined by the type of separation used (e.g., flashing or fractionation) and the requirements of the process. In each stage, a portion of the heavy lubricant fraction is lost due to conversion into hydrocarbons boiling below the heavy lubricant boiling range. However, and as shown in the examples below, the combination of the multi-stage hydrodewaxing process of the invention and the use of a dewaxing catalyst comprising a ZSM-48 zeolite component and a preferably noble metal hydrogenating component, minimizes this valuable lubricant loss, as compared to the use of only a single stage.

[0012] In the hydrodewaxing reaction, the pour and cloud points are lowered, primarily by isomerizing the higher molecular weight hydrocarbon molecules. Although the isomerization is conducted in presence of hydrogen, there is not net hydrogen consumption in a purely isomerization reaction. Interstage and final separation of the heavy lubricant hydrocarbons from the lower boiling hydrocarbons is achieved by flashing or fractionation. Hydrodewaxing reaction conditions employed in the process of the invention include a respective temperature, hydrogen partial pressure and space velocity broadly ranging from 450-750° F. (232-399° C.), 10-2,000 psig. (69-13790 kPa), and 0.1-5.0 LHSV. These conditions will more generally range from 500-700° F. (260-371° C.), 100-1000 psig. (690-6895 kPa) and 0.5-3.0 LHSV, with a temperature of 570-660° F. (299-349° C.) and a pressure of from 200-700 (1379-4827 kPa) more typical.

[0013] A heavy lubricant base stock of the invention comprises a dewaxed oil having a kinematic viscosity at 100° C. of at least 8 cSt (centistokes), preferably at least 10 cSt, and more preferably at least 12 cSt, an initial boiling point in the range of at least about 850-950° F. (454-510° C.), with an end boiling point greater than 1,000° F. (538° C.) and typically greater than 1050° F. (566° C.). It has low temperature properties able to meet target specifications or requirements. Its pour and cloud points are lower than those of the heavy lubricant oil fraction in the wax feed and in any hydrodewaxing stages upstream of the last stage. The pour point is lower than the cloud point. A heavy lubricant base stock will typically be a clear and bright oily liquid at room temperature and pressure conditions of 75° F. (24° C.) and one atmosphere (101 kPa) pressure. However, in some cases the cloud point may be higher than 75° F. (24° C.). A heavy lubricant base stock having an end boiling point above 1,250° F. (677° C.), with respective cloud and pour points of 1° C. and -31° C., has been made according to the invention. Low temperature property requirements of both the heavy lubricant base stock and a finished lubricant will vary and can depend on both the application for which it is intended and the geographical location in which the lubricant will be used. A lubricant or finished lubricant product (these two terms are used herein synonymously) is prepared by forming a mixture of the heavy lubricant base stock described herein and an effective amount of at least one additive or, more typically, an additive package containing more than one additive. Illustrative, but non-limiting examples of such additives include one or more of a detergent, a dispersant, an antioxidant, an antiwear additive, an extreme pressure additive, a pour point depressant, a VI improver, a friction modifier, a demulsifier, an antioxidant, an antifoamant, a corrosion inhibitor, and a seal swell control additive. The heavy lubricant base stock used in forming the mixture is

typically one that has been mildly hydrofinished and optionally dehazed after hydrodewaxing, to improve its color, appearance and stability.

[0014] The waxy feed or wax fed into the first hydrodewaxing stage comprises all or a portion of the waxy hydrocarbon fraction produced in a Fischer-Tropsch hydrocarbon synthesis reactor, which is liquid at the reaction conditions. It must contain hydrocarbons boiling above 1000° F. (538° C.) to produce the heavy lubricant base stock composition of the invention. It is known that in a Fischer-Tropsch hydrocarbon synthesis process, liquid and gaseous hydrocarbon products are formed by contacting a synthesis gas comprising a mixture of H₂ and CO with a Fischer-Tropsch catalyst, in which the H₂ and CO react to form hydrocarbons under shifting or non-shifting conditions and, in the process of the invention, under non-shifting conditions in which little or no, and preferably no water gas shift reaction occurs, particularly when the catalytic metal comprises Co. The synthesis gas typically contains less than 0.1 vppm and preferably less than 50 vppb of sulfur or nitrogen in the form of one or more sulfur and nitrogen-bearing compounds. Methods for removing nitrogen and sulfur from synthesis gas down to these very low levels are known and disclosed in, for example, U.S. Pat. Nos. 6,284,807; 6,168,768; 6,107,353 and 5,882,614. In the process of the invention, the cobalt Fischer-Tropsch catalyst comprises a catalytically effective amount of Co and optionally one or more of Re, Ru, Ni, Th, Zr, Hf, U, Mg and La on a suitable inorganic support material, preferably one which comprises one or more refractory metal oxides. Preferred supports for Co containing catalysts comprise titania, particularly when employing a slurry hydrocarbon synthesis process in which higher molecular weight, mostly paraffinic liquid hydrocarbon products are desired. Useful catalysts and their preparation are known and illustrative, but nonlimiting examples may be found, for example, in U.S. Pat. Nos. 4,568,663; 4,663,305; 4,542,122; 4,621,072 and 5,545,674. Fixed bed, fluid bed and slurry hydrocarbon synthesis processes are well known and documented in the literature. In all of these processes the synthesis gas is reacted in the presence of a suitable Fischer-Tropsch type of hydrocarbon synthesis catalyst, at reaction conditions effective to form hydrocarbons. Some of these hydrocarbons will be liquid, some solid (e.g., wax) and some gas at standard room temperature conditions of temperature and pressure of 25° C. and one atmosphere (101 kPa) pressure. Slurry Fischer-Tropsch hydrocarbon synthesis processes are often preferred, because when a cobalt catalyst, and preferably a non-shifting cobalt catalyst is used, they are able to produce more of the relatively high molecular weight, paraffinic hydrocarbons useful for lubricant and heavy lubricant base stocks. In order to achieve this, the synthesis reactor is operated under conditions to produce at least 14 pounds of 700° F.+ (371° C.) hydrocarbons per 100 pounds of CO converted to hydrocarbons and preferably at least 20 pounds of 700° F.+ (371° C.) hydrocarbons for every 100 pounds of CO converted to hydrocarbons. Preferably less than 10 pounds of methane are formed for every 100 pounds of CO converted. Increasing the amount of 700° F.+ (371° C.) hydrocarbons produced in the synthesis reactor is achieved by one or more of (a) decreasing the H₂:CO ratio in the synthesis feed gas, (b) decreasing the reaction temperature, and (c) increasing the reaction pressure. These high 700° F.+ (371° C.) hydrocarbon production levels have been achieved in a slurry hydrocarbon synthesis reactor,

using a catalyst having a rhenium promoted cobalt component and a titania support component. Increasing the amount of 700° F.+ (371° C.) hydrocarbons produced in the synthesis reactor is achieved by one or more of (a) decreasing the H₂:CO mole ratio in the synthesis feed gas, (b) decreasing the reaction temperature, and (c) increasing the reaction pressure. By non-shifting is meant that less than 5 wt. % and preferably less than 1 wt. % of the carbon in the feed CO is converted to CO₂.

[0015] In a hydrocarbon synthesis process conducted under nonshifting conditions with a cobalt catalyst, the mole ratio of the H₂ to CO in the synthesis gas is preferably the stoichiometric consumption mole ratio, which is typically about 2.1/1. The synthesis gas comprising a mixture of H₂ and CO is passed into the reactor (injected or bubbled up into the bottom of the slurry body in a slurry synthesis reactor), in which the H₂ and CO react in the presence of the Fischer-Tropsch hydrocarbon synthesis catalyst, at conditions effective to form hydrocarbons, a portion of which are liquid at the reaction conditions (and which comprise the hydrocarbon slurry liquid in a slurry reactor). In a slurry reactor, the synthesized hydrocarbon liquid is separated from the catalyst particles as filtrate by means such as simple filtration, although other separation means can be used. Some of the synthesized hydrocarbons are vapor and pass out of the hydrocarbon synthesis reactor as overhead gas, along with unreacted synthesis gas and gaseous reaction products. Some of these overhead hydrocarbon vapors are typically condensed to liquid and combined with the hydrocarbon liquid filtrate. Thus, the initial boiling point of the synthesized hydrocarbons removed from the reactor as liquid will vary depending on whether or not some of the condensed hydrocarbon vapors have been combined with it. Hydrocarbon synthesis process conditions vary somewhat depending on the catalyst, reactor and desired products. Typical conditions effective to form hydrocarbons comprising mostly C₅₊ paraffins, (e.g., C₅₊-C₂₀₀) and preferably C₁₀₊ paraffins, in a fixed bed, fluidized bed or slurry hydrocarbon synthesis process employing a catalyst comprising a supported cobalt component include, for example, temperatures, pressures and hourly gas space velocities in the range of from about 320-600° F., 80-600 psi and 100-40,000 V/hr/NV, expressed as standard volumes of the gaseous CO and H₂ mixture (60° F., 1 atm) per hour per volume of catalyst, respectively. In the practice of the invention, the waxy hydrocarbons or wax feed may be produced in a slurry, fixed or fluidized bed Fischer-Tropsch reactor.

[0016] A portion of the hydrocarbons synthesized in the Fischer-Tropsch hydrocarbon synthesis reactor that are normally gas or vapor at the reaction conditions are typically entrained in the liquid effluent. The vaporous effluent from the Fischer-Tropsch hydrocarbon synthesis reactor may be cooled to condense and recover some of the synthesized hydrocarbons that are vapor at the reaction conditions, and all or a portion of this condensate may be combined with the liquid effluent. Thus, the initial boiling point of the wax will vary, depending on the reactor, catalyst, conditions, amount of condensate combined with the liquid effluent, and the desired product slate. This will also result in some variations in composition. The entire wax fraction removed from the synthesis reactor as liquid (e.g., 400-450° F.+ (204-232°+)) may be fed into the first hydrodewaxing stage. If desired, some of the hydrocarbons may be removed from the wax, before it is fed into the first hydrodewaxing stage. Therefore,

in the process of the invention, the wax fed into the first hydrodewaxing reactor, may or may not boil continuously from its initial boiling point, up to its end boiling point. Still further, if desired, all, most or only a portion of the lower boiling material may be removed from the wax prior to hydrodewaxing. This means that the initial boiling point of the wax feed may range from about 400-450° F. up to 800° F. (427° C.) or more.

[0017] In the illustrative, but nonlimiting examples below, the wax was produced in a slurry Fischer-Tropsch reactor, containing a rhenium promoted cobalt catalyst having a titania support component, had an initial boiling point of between about 430-450° F. (221-232° C.). Lower boiling naphtha hydrocarbons (C₅₊ up to 430 or 450° F.) produced by the synthesis reaction, were not combined with the higher boiling, liquid reactor effluent. This wax comprised more than 90 wt. % paraffins, with from 2-4 wt. % oxygenates and 2-5 wt. % olefins, which vary depending on the reaction conditions. Aromatics were not detectable by NMR analysis. The wax contained less than 1 wppm sulfur and less than 1 wppm nitrogen. The wt. % oxygen from oxygenates is measured by neutron activation in combination with high-resolution ¹H-NMR. The total oxygen content may be placed on a water-free basis by measuring water content using calcium carbide (to form acetylene) followed by GC-MS if the water content is less than about 200 wppm. For greater than 200 wppm water content, the Karl-Fischer method in ASTM standard D-4928 is used. Aromatics are determined by X-Ray Fluorescence (XRF), as described in ASTM Standard D-2622. Sulfur is measured by XRF as per ASTM standard D-2622 and nitrogen by syringe/inlet oxidative combustion with chemiluminescence detection per ASTM standard D-4629.

[0018] A Fischer-Tropsch wax feed and at least two hydrodewaxing reaction stages, with interstage removal of hydrocarbons boiling below the heavy lubricant oil range, are essential features of the invention. A hydrodewaxing catalyst comprising a hydrogenation component, a solid acid component and a binder (zeolite catalyst), preferably the hydrogen form, is used in at least one of the reaction stages.

[0019] Illustrative, but nonlimiting examples of suitable catalyst components useful for hydrodewaxing include, for example, ZSM-23, ZSM-35, ZSM-48, ZSM-57, ZSM-22 also known as theta one or TON, and the silica aluminophosphates known as SAPO's (e.g., SAPO-11, 31 and 41), SSZ-32, zeolite beta, mordenite and rare earth ion exchanged ferrierite. Also useful are alumina and amorphous silica aluminas.

[0020] As in the case of many other zeolite catalysts, it may be desired to incorporate the solid acid component with a matrix material also known as a binder, which is resistant to the temperatures and other conditions employed in the dewaxing process herein. Such matrix materials include active and inactive materials and synthetic or naturally occurring zeolites as well as inorganic materials such as clays, silica and/or metal oxides e.g., alumina. The latter may be either naturally occurring or in the form of gelatinous precipitates, sols or gels including mixtures of silica and metal oxides. Use of a material in conjunction with the solid acid component, i.e., combined therewith, which is active, may enhance the conversion and/or selectivity of the catalyst herein. Inactive materials suitably serve as diluents

to control the amount of conversion in a given process so that products can be obtained economically and orderly without employing other means for controlling the rate or reaction. Frequently, crystalline silicate materials have been incorporated into naturally occurring clays, e.g., bentonite and kaolin. These materials, i.e., clays, oxides, etc., function, in part, as binders for the catalyst. It is desirable to provide a catalyst having good crush strength since in a petroleum refinery the catalyst is often subject to rough handling which tends to break the catalyst down into powder-like materials which cause problems in processing.

[0021] Naturally occurring clays which can be composited with the solid acid component include the montmorillonite and kaolin families which include the sub-bentonites, and the kaolins commonly known as Dixie, McNamee, Georgia and Florida clays, or others in which the main mineral constituent is halloysite, kaolinite, dickite, nacrite or anauxite. Such clays can be used in the raw state as originally mined or initially subjected to calcination, acid treatment or chemical modification.

[0022] In addition to the foregoing materials, the solid acid component can be composited with a porous matrix material such as silica-alumina, silica-magnesia, silica-zirconia, silica-thoria, silica-beryllia, silica-titania, as well as ternary compositions such as silica-alumina-thoria, silica-alumina-zirconia, silica-alumina-magnesia and silica-magnesia-zirconia. The matrix can be in the form of a cogel. Mixtures of these components can also be used. The relative proportions of finely divided solid acid component and inorganic oxide gel matrix vary widely with the crystalline silicate content ranging from about 1 to about 90 percent by weight, and more usually in the range of about 2 to about 80 percent by weight, of the composite.

[0023] It is preferred to use a hydrodewaxing catalyst comprising a ZSM-48 zeolite component and a hydrogenation component (ZSM-48 catalyst) in at least one stage. This could be any one of the two or more stages. In a more preferred embodiment, the ZSM-48 catalyst is used in all of the stages. When a ZSM-48 catalyst is used in all the stages, the yield of liquid product is 5% greater, preferably 7% greater, still more preferably 10% greater, than when using other catalyst systems. In this embodiment, the ZSM-48 catalyst is used to hydrodewax the waxy feed which contains the heavy lubricant fraction and also the heavy lubricant isomerate produced in the one or more subsequent stages. The hydrogenation component will comprise at least one Group VIII metal component and preferably a noble Group VIII metal component, as in Pt and Pd. Noble metal concentrations will range from about 0.1-5 wt % of the metal, and more typically from about 0.2-1 wt. %, based on the total catalyst weight, including the ZSM-48 zeolite component and any binder used in the catalyst composite. The Group VIII referred to herein refers to Group VIII as found in the Sargent-Welch Periodic Table of the Elements copyrighted in 1968 by the Sargent-Welch Scientific Company. Hydrodewaxing experiments conducted with Fischer-Tropsch wax have revealed that the ZSM-48 catalyst is superior to others, including, for example, rare earth ion exchanged ferrierite, mordenite, zeolite beta, SAPO-11, TON and ZSM-23, all using a Pt hydrogenating component. It is also superior to Pd/amorphous silica alumina (20% silica). In these experiments, the ZSM-48 catalyst was more selective to lubricant oil production, including heavy lubricant frac-

tion production, which means less conversion to hydrocarbons boiling below the lubricant oil range (e.g., -650-750° F.- (343-399° C.-)) and less conversion of the heavy hydrocarbons to hydrocarbons boiling below the heavy lubricant range (e.g., 900-1,000° F.+ (482-538° C.+)). Conversion is calculated according to the following arithmetic relation, using 700° F.+ (371° C.+ conversion as a specific example.

$$700^{\circ} \text{ F.+conversion} = [1 - (\text{wt. \% } 700^{\circ} \text{ F.+fraction in product}) / (\text{wt. \% } 700^{\circ} \text{ F.+in feed})] \times 100$$

[0024] The preparation of ZSM-48 is well known and is disclosed, for example, in U.S. Pat. Nos. 4,397,827; 4,585,747 and 5,075,269, and EP 0 142 317, the disclosures of which are incorporated herein by reference. Other hydrodewaxing catalysts useful in the practice of the invention, include any of the well known catalysts that dewax mostly by isomerization and not by cracking or hydrocracking. Zeolites comprising ten and twelve membered ring structures are useful as dewaxing catalysts, particularly when combined with a catalytic metal hydrogenating component. Illustrative, but nonlimiting examples of suitable zeolites and other catalyst components useful for hydrodewaxing include, for example, rare earth ion exchanged ferrierite, mordenite, beta alumina, zeolite beta, ZSM-23, ZSM-35, ZSM-57, ZSM-22 also known as theta one or TON, and the silica aluminophosphates known as SAPO's (e.g., SAPO-11, 31 and 41), and SSZ-32. Also useful are alumina and amorphous silica aluminas. However, in experiments using a hydrodewaxing catalyst comprising Pt on zeolite beta and a catalyst comprising Pt on amorphous silica alumina, about 50 wt. % of the desirable 950° F.+ (510° C.+ heavy lubricant fraction was converted to hydrocarbons boiling in the fuels range.

[0025] In the context of the invention, the terms "hydrogen" and "hydrogen treat gas" are synonymous and may be either pure hydrogen or a hydrogen-containing treat gas which is a treat gas stream containing hydrogen in an amount at least sufficient for the intended reactions, plus other gas or gasses (e.g., nitrogen and light hydrocarbons such as methane) which will not adversely interfere with or affect either the reactions or the products. The treat gas stream introduced into a reaction stage will preferably contain at least about 50 vol. %, more preferably at least about 80 vol. % hydrogen.

[0026] In the integrated process embodiment which includes synthesis gas production, the synthesis gas is produced from natural gas and contacted with a cobalt Fischer-Tropsch catalyst to produce the waxy hydrocarbons, which are dewaxed by the multi-stage hydrodewaxing process. It is not unusual for natural gas to comprise as much as 92+ mole % methane, with the remainder primarily C₂₊ hydrocarbons, nitrogen and CO₂. Thus, it is an ideal and relatively clean fuel for synthesis gas production. The methane has a 2:1 H₂:C ratio and is ideal for producing a synthesis gas having an H₂:CO mole ratio of nominally 2.1:1 by a combination of partial oxidation and steam reforming. This is the stoichiometric mole ratio used with a non-shifting cobalt catalyst for hydrocarbon synthesis. Thus, natural gas is ideal for producing synthesis gas having the desired stoichiometric 2.1:1 H₂:C mole ratio required when using a cobalt Fischer-Tropsch hydrocarbon synthesis and preferably one that is non-shifting. Sulfur and heteroatom compounds are removed from the natural gas, and in some cases also

nitrogen and CO₂. The remaining methane-rich gas, along with oxygen or air and steam, is passed into a synthesis gas generator. Oxygen is preferred to air, because it does not introduce nitrogen into the synthesis gas generator (reactor). During the synthesis gas reaction nitrogen can form HCN and NH₃, both of which are poisons to a cobalt Fischer-Tropsch catalyst and must therefore be removed down to levels below 1 ppm. If nitrogen is not removed from the natural gas, and/or if air is used as the source of oxygen, before converting it into synthesis gas, HCN and NH₃ must be removed from the synthesis gas, before it is passed into the one or more hydrocarbon synthesis reactors. In a synthesis gas generator, the natural gas reacts with oxygen and/or steam to form synthesis gas, which then serves as the feed for the hydrocarbon synthesis. Known processes for synthesis gas production include partial oxidation, catalytic steam reforming, water gas shift reaction and combinations thereof. These processes include gas phase partial oxidation (GPOX), autothermal reforming (ATR), fluid bed synthesis gas generation (FBSG), partial oxidation (POX), catalytic partial oxidation (CPO), and steam reforming. ATR and FBSG employ oxygen and form the synthesis gas by partial oxidation and catalytic steam reforming. ATR and FBSG are preferred for producing synthesis gas in the practice of the invention. A review of these processes and their relative merits may be found, for example, in U.S. Pat. No. 5,883,138. The invention will be further understood with reference to the examples below.

EXAMPLES

[0027] In the examples below, the Fischer-Tropsch wax feed comprised either the (i) 430° F.+ (232° C.+) waxy fraction obtained from a slurry Fischer-Tropsch reactor, in which the H₂ and CO were reacted in the presence of a titania supported cobalt rhenium catalyst to form hydrocarbons, most of which were liquid at the reaction conditions or the (ii) 1000° F.+ (538° C.+) fraction of the 430° F.+ (232° C.+) waxy fraction. The synthesis reactor was operating at conditions to produce at least 14 pounds (6.4 kg) of 700° F.+ (371° C.+) hydrocarbons per 100 pounds (45.4 kg) of CO converted to hydrocarbons. The raw (untreated) wax was fed into the first stage of the two and three stage runs below. The interstage separation cut point was 950° F. (510° C.). This means that only the partially hydrodewaxed 950° F.+ (510° C.) isomerate recovered from the proceeding stage was passed into the second stage of the two stage process and into the second and third stages of the three stage process. The boiling point distribution for the waxy feed used in the two and three stage runs is given in the table below.

Wt. % Boiling Point Distribution of Fischer Tropsch Reactor Waxy Feed	
430° F.+ (232° C.+)	98
700° F. (371° C.+)	71.5
1000° F.+ (538° C.+)	26.2

[0028] The hydrogen treat gas used in all the examples was pure hydrogen. A ZSM-48 catalyst was used for hydrodewaxing the waxy feed in all the examples. It comprised 0.6 wt. % Pt as the hydrogenating component, on a composite of the hydrogen form of the ZSM-48 zeolite and an alumina binder. The hydrogen form of the ZSM-48

zeolite was prepared according to the procedure in U.S. Pat. No. 5,075,269, the disclosure of which is incorporated herein by reference. The Pt component was added by impregnation, followed by calcining and reduction, using known procedures. Gas chromatograph distillations (GCD) were conducted using a high temperature GCD method modification of ASTM D-5307. The column consisted of a single capillary column with a thin liquid phase, less than 0.2 microns. External standards were used, consisting of a boiling point calibrant ranging from 5 to 100 carbons. A temperature programmed injector was used and, prior to injection, the samples were gently warmed using hot water. Boiling ranges were determined using this method and the T5 and T95 GCD results. Cloud point values were measured using ASTM D-5773 for Phase Tec Instruments, under the lubricant procedure method. Pour point was measured according to ASTM D-5950 for ISL Auto Pour Point measurement. Cloud and pour points in the Tables below are given in ° C. Viscosity and viscosity index were measured according to the ASTM protocols D-445 and D-2270, respectively.

Comparative Example 1

[0029] The 1000° F.+ (538° C.+) fraction of the 430° F.+ (232° C.+) wax feed described above was hydrodewaxed in a single stage, by reacting with hydrogen in the presence of the ZSM-48 catalyst described above. The reaction conditions were 635° F. (336° C.), 250 psig. (1724 kPa), and 2300 SCF/B of H₂ and a LHSV of 1. These reaction conditions were used to achieve a target cloud point of 5° C. for the 1000° F.+ (538° C.+) heavy lubricant base stock. The results of this run are shown in Table 1 below.

Example 1

[0030] In this example, the 430° F.+ (232° C.+) waxy feed was hydrodewaxed by reacting it with hydrogen over the ZSM-48 catalyst described above, to the same 1000° F.+ (538° C.+) heavy lubricant base stock cloud point of 5° C. in two stages, according to the practice of the invention. The waxy feed was hydrodewaxed in the first stage at 587° F. (308° C.), 250 psig. (1724 kPa) of hydrogen at a treat rate of 2300 SCF/B of H₂ and a waxy feed LHSV of 1. The first stage isomerate was fractionated to separate the 700° F.– (371° C.–) fraction, to determine the extent of the 700° F.+ (371° C.+) feed conversion to lower boiling material and then further fractionated to separate and recover a 950° F.+ (510° C.+) heavy oil fraction. The 950° F.+ (510° C.) heavy oil fraction was then hydrodewaxed in a second stage at 614° F. (323° C.), 250 psig. (1724 kPa) H₂ at a treat rate of 2500 SCF/b and a heavy oil LHSV of 1. The second stage isomerate was fractionated to recover the final 1000° F.+ (538° C.+) heavy lubricant base stock. The results of this run are also shown in Table 1.

TABLE 1

	700° F.+ (371° C.+)	1000° F.+ (538° C.+)	Temp. ° F./	1000° F.+ (538° C.+)		
	Conversion	Conversion	° C.	Pour	Cloud	VI
Single Stage	39	61	635/335	-48	5	153

TABLE 1-continued

	700° F.+ (371° C.+)	1000° F.+ (538° C.+)	Temp. ° F./ ° C.	1000° F.+ (538° C.+)		
	Conversion	Conversion	° C.	Pour	Cloud	VI
Multiple Stage	29	52				
Stage One	11	19	587/308	26	—	
Stage Two	20	41	614/323	-27	5	155

[0031] These results clearly demonstrate the benefits of the multi-stage process, in that at the same 5° C. target cloud point, a significant increase in the amount of the 1000° F.+ (538° C.+) heavy lubricant base stock was produced using two stages, as opposed to just a single stage. Using a single stage resulted in a conversion of 39 wt. % to lower boiling, 700° F.– (371° C.–) hydrocarbons, while a total of only 29% occurred in the multi-stage process of the invention. Since a lubricant stock cut point is typically about 700° F. (e.g., ~700° F./371° C.+ material), this means that more lighter lubricant boiling hydrocarbons (750-950° F. (399-610° C.) boiling range) were also produced by the two stage process of the invention. Similarly, the total 1000° F.+ (538° C.+) fraction conversion to lower boiling, 1000° F.– (538° C.–) hydrocarbons for the single stage process was 61%, as opposed to only 52% using the two stage process of the invention. Thus, a greater yield of the heavy lubricant base stock at the same target cloud point was achieved using the process of the invention, with no loss in VI and with a lower pour/cloud point spread.

Comparative Example 2

[0032] This run was identical to that of Comparative Example 1, except that the reactor was run at the slightly lower temperature of 630° F. (322° C.), to achieve a 1000° F.+ (538° C.+) cloud point of 11° C. The results are shown below in Table 2.

Example 2

[0033] This run was identical to that of Example 1, except that the second stage reaction was conducted at a lower temperature of 610° F. (321° C.), to reach the 11° C. cloud point for the 1000° F.+ (538° C.+) product. These results are also shown in Table 2, as a comparison to the results for the Comparative Example 2, single stage run.

TABLE 2

	700° F.+ (371° C.+)	1000° F.+ (538° C.+)	Temp. ° F./ ° C.	1000° F.+ (538° C.+)		
	Conversion	Conversion	° C.	Pour	Cloud	VI
Single Stage	30	58	630/332	-28	11	148
Multiple Stage	26	48				
Stage One	11	19	587/308	26		
Stage Two	17	35	610/321	-19	11	159

[0034] As was the case for Example 1, in these runs the extent of feed conversion to 700° F.– (371° C.–) and 1000° F.– (538° C.–) boiling hydrocarbons was much lower using the multiple stage process of the invention, than with only a single stage.

Comparative Example 3

[0035] This run was also identical to that of Comparative Example 1, except that the reactor was run at a higher temperature of 640° F. (338° C.), to achieve a 1000° F.+ (538° C.+) cloud point of -2° C. The results are shown in Table 3.

Example 3

[0036] This run was identical to that of Example 1, except that the second stage reaction was conducted at a higher temperature of 620° F. (327° C.), to reach the same -2° C. cloud point for the 1000° F.+ (538° C.+) heavy lubricant base stock product, as for the Comparative Example 3 cloud point. These results are also shown in Table 3, as a comparison to the results for the Comparative Example 3, single stage run.

TABLE 3

	700° F.+ (371° C.+)	1000° F.+ (538° C.+)	Temp. ° F./ ° C.	1000° F.+ (538° C.+)		
	Conversion	Conversion	° C.	Pour	Cloud	VI
Single Stage	45	72	640/338	-49	-2	142
Multiple Stage	39	63				
Stage One	11	19	587/308	26		
Stage Two	31	54	620/327	-50	-6	148

[0037] As was the case for the previous two examples, in this run the extent of feed conversion to 700° F.– (371° C.–) and 1000° F.– (538° C.–) boiling hydrocarbons was much lower using the multiple stage process of the invention, than with only a single stage. The target cloud point of -2° C. was overrun by -4° C. with the two stage process of the invention, resulting in more conversion to the lower cloud point of -6° C. Nevertheless, despite the greater conversion with the two stage process overshoot, both the 700° F.+ (371° C.+) and 1000° F.+ (538° C.+) conversions to lower boiling hydrocarbons are still substantially less with the multiple stage process of the invention than with the single stage process.

Example 4

[0038] The same 430° F.+ (232° C.+) waxy feed and catalyst used in the examples above, were used in this example. However, in this example the feed was hydrodewaxed to a 1000° F.+ (538° C.+) heavy fraction cloud point of 5° C. in three stages, according to the practice of the invention. The wax feed was hydrodewaxed in the first stage at 587° F. (308° C.), 259 psig. (1786 kPa) of hydrogen at a treat rate of 2300 SCF/b H₂ and a waxy feed LHSV of 1. The treat gas, treat gas rate and space velocity of the feed were the same in all three stages. The first stage isomerate was fractionated to separate and remove the 950° F.– (610° C.–)

fraction, with the remaining 950° F.+ (610° C.-) fraction then passed into the second stage. In the second stage, the temperature was 607° F. (321° C.). After removal of the 950° F.- (610° C.-) hydrocarbons from the second stage isomerate, the remaining, further partially dewaxed 950° F.+ (610° C.+) fraction was passed into the third stage, in which it was further hydrodewaxed at a temperature of 600° F. (316° C.). The third stage isomerate was fractionated to separate and recover the 1000° F.+ (538° C.+) heavy lubricant base stock. The results of this run are shown in Table 4, with the results from the single stage hydroisomerization of Comparative Example 1 shown for comparison.

TABLE 4

	700° F.+ (371° C.+)	1000° F.+ (538° C.+)	Temp. ° F./	1000° F.+ (538° C.+)		
	Conversion	Conversion	° C.	Pour	Cloud	VI
Single Stage	39	61	635/335	-48	5	153
Multiple Stage	28	48				
One Stage	11	19	587/308			
Two Stage	13	26	607/319			
Three Stage	7	14	600/316	-24	5.2	

[0039] The amount of the desired 1000° F.+ (538° C.+) heavy lubricant base stock product was slightly higher using three stages, than that obtained using two stages (compare to Example 1 in which the same 1000° F.+ (538° C.+) heavy lubricant base stock had the same cloud point of 5° C.). The amount of feed conversion to 700° F.- (371° C.-) boiling hydrocarbons using three stages, was about the same as that resulting from using two stages, with both substantially less than that using only one stage. This further demonstrates the greater amount of heavy lubricant base stock yields obtained using the multiple stage hydrodewaxing process of the invention, because the waxy feed to the first stage contained substantial amounts of 950° F.- (610° C.-) boiling hydrocarbons. Yet, a total of only 28% of the 430° F.+ (232° C.+) feed hydrocarbons were converted to 700° F.- (371° C.-) boiling hydrocarbons using three stages, compared to the 39% of the 1000° F.+ (538° C.+) hydrocarbons which were converted to 700° F.- (371° C.-) boiling hydrocarbons, using only one stage. Further, the amount of the valuable 1000° F.+ (538° C.+) heavy lubricant base stock lost by conversion to lower boiling hydrocarbons was 61, 29 and 28 wt. % respectively, for the single, two and three stage processes.

Example 5

[0040] The same 430° F.+ (232° C.+) waxy feed and catalyst used in the examples above, were also used in this example. However, in this example the feed was hydrodewaxed to a 1000° F.+ (538° C.+) heavy fraction cloud point of -3° C. in three stages. The 430° F.+ (232° C.+) waxy feed was hydrodewaxed in the first stage at 587° F. (308° C.), 259 psig. (1786 kPa) of hydrogen at a treat rate of 2300 SCF/b H₂ and a waxy feed LHSV of 1. The treat gas, treat gas rate and space velocity of the feed were the same in all stages. The first stage isomerate was fractionated to separate and

remove the 950° F.- (610° C.-) fraction, with the remaining, partially dewaxed, heavy 950° F.+ (610° C.+) fraction then passed into the second stage. In the second stage, the temperature was 607° F. (319° C.). After removal of the 950° F.- (610° C.-) hydrocarbons from the second stage isomerate, the remaining 950° F.+ (610° C.+) fraction was passed into the third stage, in which it was further hydrodewaxed at a temperature of 610° F. (321° C.). The third stage isomerate was fractionated to separate and recover the 1000° F.+ (538° C.+) heavy lubricant base stock. The results of this run are shown in Table 5, with the results from the Comparative Example 3 single stage hydrodewaxing shown for comparison.

TABLE 5

	700° F.+ (371° C.+)	1000° F.+ (538° C.+)	Temp.	1000° F.+ (538° C.+)		
	Conversion	Conversion	° F.	Pour	Cloud	VI
Single Stage	45	72	640/338	-49	-2	153
Multiple Stage	33	55				
One Stage	11	19	587/308			
Two Stage	13	26	607/319			
Three Stage	13	25	610/321	-42	-3	

[0041] As was the case for the case for Example 4, the amount of the desired 1000° F.+ (538° C.+) heavy lubricant base stock product for this run, as reflected by the overall or total conversion to hydrocarbons boiling below 1000° F. (538° C.), was much lower using three stages, than that obtained using only one stage. The amount of feed conversion to 700° F.- (371° C.-) boiling hydrocarbons using three stages, was also substantially less than that which resulted from using only one stage.

Example 6

[0042] A 430° F.+ (232° C.+) waxy feed was contacted with a ZSM-48 catalyst under hydrodewaxing conditions in a first hydrodewaxing stage to produce an 850° F.+ (454° C.) hydroisomerate. The hydroisomerate was then hydrodewaxed in a second hydrodewaxing stage under the conditions set forth in Table 6.

TABLE 6

Property	ZSM-48 Catalyst	ZSM-23 Catalyst	Units
Temperature	309	315	° Centigrade
Pressure	32.6	33	bar
LHSV	1	1	hr-1
Hydrogen rate	112	112	ml/min
Hydrogenolysis	0.21%	0.39%	Methane yield
Light gases	2.4%	7.7%	C ₂ -C ₄ gases
1000 F.+ Raffinate	19.63%	17.81%	
1000 F.+ Viscosity	17.26	16.16	cSt
Conversion	25	25	%
Liquid Yield	97.4%	91.9%	C ₅₊ product/feed

[0043] As will be noted, a higher yield of liquid product is obtained when a ZSM-48 catalyst is used in all stages of a

multistage process. The yield of liquid product is increased by about 6% demonstrating that the combination of the multi-stage hydrodewaxing process of the invention and the use of a dewaxing catalyst comprising a ZSM-48 zeolite component and a preferably noble metal hydrogenating component, minimizes valuable lubricant loss, as compared to the use of only a single stage or a multistage process with a different catalyst system. Not only was the yield of liquid product higher but the gas make using a ZSM-48 catalyst was significantly lower.

What is claimed is:

1. A multistage process for producing a heavy lubricant base stock from Fischer-Tropsch wax comprises hydrodewaxing said wax in a first hydrodewaxing stage to produce an isomerate comprising a partially dewaxed heavy lubricant oil fraction, followed by hydrodewaxing said heavy lubricant oil fraction in one or more successive hydrodewaxing stages, with interstage removal of hydrocarbons boiling below said heavy lubricant oil fraction, to form said heavy lubricant base stock having a kinematic viscosity at 100° C. of at least about 8 cSt and wherein said hydrodewaxing is achieved in the presence of hydrogen and a hydrodewaxing catalyst that dewaxes by isomerization, said hydrodewaxing catalyst comprising a hydrogenating component, a ZSM-48 zeolite component, and a binder.

2. A process according to claim 1 wherein said heavy lubricant base stock has cloud and pour points lower than that of said heavy fraction in said wax.

3. A process according to claim 2 wherein said heavy lubricant base stock has an initial boiling point in the range of 850-1000° F. (454-538° C.).

4. A process according to claim 3 wherein said partially dewaxed heavy lubricant oil fraction hydrodewaxed in said one or more successive hydrodewaxing stages comprises less than 25 wt. % lower boiling hydrocarbons.

5. A process according to claim 4 wherein said heavy lubricant base stock has been hydrofinished and optionally de hazed.

6. A process according to claim 5 wherein said heavy lubricant base stock is combined with one or more lubricant additives to form a lubricant.

7. A process according to claim 6 wherein said heavy lubricant base stock has an end boiling point above 1050° F. (566° C.) and a cloud point no greater than 75° F. (24° C.).

8. The process of claim 1 wherein said hydrogenation component comprises at least one Group VIII metal component.

9. The process of claim 8 wherein said binder is selected from the group consisting of zeolites, clays, silica, alumina, metal oxides, silica-alumina, silica-magnesia, silica-zirconia, silica-thoria, silica-beryllia, silica-titania, silica-alu-

mina-thoria, silica-alumina, zirconia, silica-alumina-magnesia, silica-magnesia-zirconia and mixtures thereof.

10. A process according to claim 8 wherein said at least one Group VIII metal component is a noble metal.

11. A process according to claim 10 wherein said noble metal is at least one of Pt and Pd.

12. A process comprising (a) producing synthesis gas comprising a mixture of H₂ and CO from natural gas, (b) contacting said synthesis gas with a cobalt Fischer-Tropsch hydrocarbon synthesis catalyst at reaction conditions effective for said H₂ and CO to react and form waxy hydrocarbons, including hydrocarbons boiling in the heavy lubricant oil range, (c) passing at least a portion of said waxy hydrocarbons to a hydrodewaxing upgrading facility in which said waxy hydrocarbons are (i) hydrodewaxed in the presence of a hydrodewaxing catalyst and hydrogen in a first hydrodewaxing stage, to produce an isomerate comprising a partially dewaxed heavy lubricant oil fraction and lower boiling hydrocarbons, (ii) separating said partially dewaxed heavy lubricant oil fraction and lower boiling hydrocarbons, and (ii) hydrodewaxing said separated heavy lubricant oil fraction in one or more successive hydrodewaxing stages, with interstage removal of hydrocarbons boiling below said heavy lubricant oil fraction, to form heavy lubricant base stock having a kinematic viscosity at 100° C. of at least about 8 cSt, wherein a hydrodewaxing catalyst comprising a ZSM-48 zeolite component and a noble metal hydrogenating component is used.

13. A process according to claim 12 wherein said waxy hydrocarbons formed in (b) are not hydroprocessed prior to being passed to said hydrodewaxing upgrading facility.

14. A process according to claim 13 wherein said heavy lubricant base stock has cloud and pour points lower than that of said heavy fraction in said wax.

15. A process according to claim 12 wherein said noble comprises at least one of Pt and Pd.

16. A process according to claim 15 wherein said base stock has been hydrofinished and optionally de hazed.

17. A process according to claim 15 wherein at least one of said Pt and Pd noble metal hydrogenating components is used in said first hydrodewaxing stage and at least one of said successive hydrodewaxing stages used to further hydrodewax said heavy lubricant oil fraction.

18. A process according to claim 17 wherein said base stock is combined with one or more lubricant additives to form a lubricant.

19. A process according to claim 18 wherein said heavy lubricant base stock has an end boiling point above 1050° F. (566° C.) and a cloud point no greater than 75° F. (24° C.).

* * * * *