

PROCESS FOR THE PRODUCTION OF WHITE OILS**FIELD OF THE INVENTION**

5 The invention relates to a process for the production of white oils having a very low aromatic content.

BACKGROUND ART

10 White oils are known for the skilled person and correspond to highly refined mineral oils and thus are of high purity. White oils generally fall into two classes, technical grade and medicinal grade. Medicinal grade white oils are typically chemically inert and substantially without color, odor, or taste. The technical grade white oils are generally used in textile lubrication, sealants, adhesives or bases for insecticides. The more highly refined medicinal grade white oils are those suitable for use in drug compositions, cosmetics, foods, and for the
15 lubrication of food handling machinery.

 White oils have high stability properties, in particular high thermal stability, are chemically inert, without odor and without color. White oils are notably defined in the Code of Federal Regulation of the FDA for example in sections 21 C.F.R. § 172.878 regarding direct food additives, 21 C.F.R. § 178.3620 (a) regarding indirect food additives, 21 C.F.R. § 573.680
20 regarding animal food additives and H1 food processing lubricant standards, 21 C.F.R. § 178.3620 (b) regarding indirect food additives and 21 C.F.R. § 573.680 regarding animal food additives. White oils are also defined in the French and European Pharmacopoeia.

 White oils are generally produced by refining an appropriate petroleum feedstock to remove oxygen, nitrogen, and sulfur compounds, reactive hydrocarbons such as aromatics,
25 and any other impurity which would prevent use of the resulting white oil in the pharmaceutical or food industry.

 EP 1 171 549 discloses a hydrofining process of a hydrocarbon feedstock having a substantial amount of sulphur at temperatures ranging from 200 to 400°C in order to produce white oils.

30 The inventors surprisingly discovered that the processes of the prior art leads to by-products, notably cracking products, and thus to a loss of yield of the process.

 There is thus a need for a process for producing white oils with higher yields, with a process easy to implement and with a reduced cost, without involving high amounts of sulphur.

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SUMMARY OF THE INVENTION

 The invention provides a process for producing a white oil having an initial boiling point of at least 250°C, the process comprising a step of catalytically hydrogenating a base oil

feedstock at a temperature of from 120 to 210°C, at a pressure of from 30 to 160 bars and a liquid hourly space velocity of 0.2 to 5 hr⁻¹, the base oil feedstock comprising less than 5 ppm by weight of sulphur.

According to an embodiment, the base oil feedstock comprises less than 3 ppm by weight, preferably less than 1 ppm by weight of sulphur.

According to an embodiment, the base oil feedstock has an initial boiling point ranging from 250 to 350°C and a final boiling point ranging from 350 to 600°C.

According to an embodiment, the base oil feedstock has a viscosity at 40°C of at least 6 cSt, preferably at least 7 cSt, more preferably at least 7,5 cSt.

According to an embodiment, the base oil feedstock is selected from Groups II, III, IV and mixture thereof, preferably from Groups II and III, more preferably from Group III, of the API classification.

According to an embodiment, the base oil feedstock is selected from oils issued from a hydrocracking process and from oils issued from a deep desulphurization process.

According to an embodiment, the hydrogenating step is performed at a temperature ranging from 150 to 200°C, preferably from 150 to 190°C.

According to an embodiment, the hydrogenating step is performed at a pressure ranging from 50 to 150 bars, preferably from 50 to 130 bars.

According to an embodiment, the hydrogenating step is performed at a liquid hourly space velocity ranging from 0.4 to 3 hr⁻¹, preferably from 0.5 to 1.5 hr⁻¹.

According to an embodiment, the catalyst is a nickel catalyst, preferably a supported nickel catalyst.

According to an embodiment, the catalyst is not in a sulfided form when the hydrogenation step is started.

According to an embodiment, the hydrogenating step is performed in a unit comprising at least 2 reactors, preferably in series.

According to an embodiment, the process further comprises a fractionating step, preferably performed after the hydrogenating step.

According to an embodiment, the white oil has an aromatic content below 1000 ppm by weight, preferably below 500 ppm by weight, more preferably below 300 ppm by weight, even more preferably below 200 ppm by weight.

White oils produced thanks to the process of the invention reply to the purity criterion of the European Pharmacopoeia (monography on liquid paraffins of pharmacopoeia EuPh 6.0 01/2008), of the US Pharmacopoeia (US Pharmacopoeia Light Mineral Oil, USP32 – NF 27), and of the Japanese Pharmacopoeia (Japanese Pharmacopoeia Light liquid Paraffin).

BRIEF DESCRIPTION OF THE FIGURES

Fig. 1 represents the evolution of the amount of monoaromatic compounds during the different phases of the example 2.

Fig. 2 represents the amount of compounds in function of their boiling temperature,
5 for different hydrogenation temperatures.

DESCRIPTION OF EMBODIMENTS OF THE INVENTION

The present invention relates to a process for producing a white oil having an initial boiling point of at least 250°C, the process comprising a step of catalytically hydrogenating a
10 base oil feedstock at a temperature of from 120 to 210°C, at a pressure of from 30 to 160 bars and a liquid hourly space velocity of 0.2 to 5 hr⁻¹, the base oil feedstock comprising less than 5 ppm by weight of sulphur.

Within the meaning of the present invention, the IBP is different from the FBP of a
15 product, this applies for example for the feedstock and for the white oil.

Feedstock (also named "feed"):

The base oil feed typically comprises less than 5 ppm by weight of sulphur, preferably less than 3 ppm by weight, more preferably less than 1 ppm by weight. The sulphur content
20 can be measured according to ASTM D2622 standard using X-ray Fluorescence.

The base oil feed typically has an initial boiling point (IBP) ranging from 250 to 350°C and a final boiling point (FBP) ranging from 350 to 600°C. The IBP and FBP can be measured according to ASTM D86 standard and/or ASTM D1160 standard. ASTM D1160 standard is used to measure boiling points above 400°C. The boiling range, i.e. the difference between the
25 IBP and the FBP, is preferably lower than 200°C, more preferably lower than 150°C, even more preferably lower than 100°C.

According to an embodiment, the base oil feed has a boiling range within the range of 250-400°C, preferably of 270-380°C, i.e. the initial and the final boiling point are preferably within the range of 250-400°C or 270-380°C.

30 The base oil feed typically has a viscosity at 40°C of at least 6.0 mm²/s, preferably at least 7.0 mm²/s, more preferably at least 7.5 mm²/s. The viscosity can be measured according to ASTM D445 standard.

According to an embodiment of the invention, the feedstock has an aromatic content ranging from 5 ppm to 1% by weight, preferably from 50 ppm to 500 ppm by weight, based
35 on the total weight of the feedstock. The aromatic content can be measured by UV spectrometry.

According to an embodiment of the invention, the base oil feedstock has a density at 15°C ranging from 0.8100 to 0.8700 g/mL, preferably from 0.8200 to 0.8600 g/mL. The density at 15°C can be measured according to ISO 12185 standard.

5 The process of the invention can typically comprise a step of providing a base oil feed as defined in the present invention for the hydrogenating step, for example a base oil comprising less than 5 ppm by weight of sulphur, having an initially boiling point (IBP) ranging from 250 to 350°C and a final boiling point (FBP) ranging from 350 to 600°C and having a viscosity at 40°C of at least 6.0 mm²/s.

10 According to an embodiment, the base oil feedstock is selected from oils issued from a hydrocracking process and from oils issued from a deep desulphurization process, preferably from a hydrocracking process.

According to this embodiment, the hydrocracking process can be performed on a hydrocarbon feedstock and the heaviest fraction issued from the hydrocracking process may be used as the base oil feed of the process of the present invention.

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The base oil feedstock can be defined as specified in the American Petroleum Institute (API) Base Oil Interchangeability Guidelines. There are five base oil groups that are as given in the table that follows.

	Saturates	Sulfur	Viscosity index (VI)
Group I mineral oils	< 90 %	> 0.03 %	80 ≤ VI < 120
Group II hydrocracked oils	≥ 90 %	≤ 0.03 %	80 ≤ VI < 120
Group III hydrocracked oils or hydroisomerized oils	≥ 90 %	≤ 0.03 %	≥ 120
Group IV	Polyalphaolefins (PAO)		
Group V	Esters and other bases not included in Groups I to IV		

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According to an embodiment of the invention, the base oil feedstock is selected from Groups II, III or IV or mixtures thereof, more preferably from Groups II or III, even more preferably from a base oil of Group III.

25 Hydrogenation step.

The feedstock is hydrogenated. The feedstock can optionally be pre-fractionated.

Hydrogen that is used in the hydrogenation unit is typically a high purity hydrogen, e.g. with a purity of more than 99%, albeit other grades can be used.

30 Hydrogenation takes place in one or more reactors. The reactor can comprise one or more catalytic beds. Catalytic beds are usually fixed beds.

Hydrogenation takes place using a catalyst. Typical hydrogenation catalysts include but are not limited to: nickel, platinum, palladium, rhenium, rhodium, nickel tungstate, nickel molybdenum, molybdenum, cobalt molybdenate, nickel molybdenate on silica and/or alumina carriers or zeolites. A preferred catalyst is Ni-based and is supported on an alumina carrier, having preferably a specific surface area varying between 100 and 200 m²/g of catalyst. According to a particular embodiment, the catalyst consists in nickel as metallic compound.

The hydrogenation conditions are typically the following:

- Pressure: 30 to 160 bars, preferably 40 to 150 bars, and most preferably 50 to 130 bars;
- Temperature: 120 to 210°C, preferably 125 to 200°C and most preferably 130 to 190°C;
- Liquid hourly space velocity (LHSV) : 0.2 to 5 hr⁻¹, preferably 0.4 to 3, and most preferably 0.5 to 1.5;
- Hydrogen treat rate: adapted to the above conditions, which can be up to 200 Nm³/ton of feed.

The temperature in the reactors can be typically about 120-190°C and the pressure can be typically from 50 to 100 bars while the liquid hourly space velocity can be typically about 1h⁻¹ and the treat rate is adapted, depending on the feed quality and the first process parameters.

The hydrogenation process of the invention can be carried out in several stages. There can be two or three stages, preferably three stages, preferably in three separate reactors. The first stage will operate the sulphur trapping, hydrogenation of substantially all unsaturated compounds, and up to about 90% of hydrogenation of aromatics. The flow exiting from the first reactor contains substantially no sulphur. In the second stage the hydrogenation of the aromatics continues, and up to 99% of aromatics are hydrogenated. The third stage is a finishing stage, allowing an aromatic content as low as 1000ppm by weight or even less such as below 500ppm, more preferably less than 200ppm, even for high boiling products.

The catalysts can be present in varying or substantially equal amounts in each reactor, e.g. for three reactors according to weight amounts of 0.05-0.5/0.10-0.70/0.25-0.85, preferably 0.07-0.25/0.15-0.35/0.4-0.78 and most preferably 0.10-0.20/0.20-0.32/0.48-0.70.

It is also possible to have one or two hydrogenation reactors instead of three.

It is also possible that the first reactor be made of twin reactors operated alternatively in a swing mode. This may be useful for catalyst charging and discharging: since the first reactor comprises the catalyst that is poisoned first (substantially all the sulphur is trapped in and/or on the catalyst) it should be changed often.

One reactor can be used, in which two, three or more catalytic beds are installed.

It may be necessary to insert quenches on the recycle to cool effluents between the reactors or catalytic beds to control reaction temperatures and consequently hydrothermal equilibrium of the hydrogenation reaction. In a preferred embodiment, there is no such intermediate cooling or quenching.

5 In case the process makes use of 2 or 3 reactors, the first reactor will act as a sulphur trap. This first reactor will thus trap substantially all the sulphur. The catalyst will thus be saturated quickly and may be renewed from time to time. When regeneration or rejuvenation is not possible for such saturated catalyst the first reactor is considered as a sacrificial reactor which size and catalyst content both depend on the catalyst renewal frequency.

10 In an embodiment the resulting product and/or separated gas is/are at least partly recycled to the inlet of the hydrogenation stages. This dilution helps, if this were to be needed, maintaining the exothermicity of the reaction within controlled limits, especially at the first stage. Recycling also allows heat-exchange before the reaction and also a better control of the temperature.

15 The stream exiting the hydrogenation unit contains the hydrogenated product and hydrogen. Flash separators are used to separate effluents into gas, mainly remaining hydrogen, and liquids, mainly hydrogenated hydrocarbons. The process can be carried out using three flash separators, one of high pressure, one of medium pressure, and one of low pressure, very close to atmospheric pressure.

20 The hydrogen gas that is collected on top of the flash separators can be recycled to the inlet of the hydrogenation unit or at different levels in the hydrogenation units between the reactors.

Because the final separated product is at about atmospheric pressure, it is possible to feed directly the optional fractionation stage, which is preferably carried out under vacuum
25 pressure that is at about between 10 to 50 mbars, preferably about 30 mbars.

The optional fractionation stage can be operated such that various hydrocarbon fluids can be withdrawn simultaneously from the fractionation column, and the boiling range of which can be predetermined.

Therefore, fractionation can take place before hydrogenation, after hydrogenation, or
30 both.

The hydrogenation reactors, the separators and the fractionation unit can thus be connected directly, without having to use intermediate tanks. By adapting the feed, especially the initial and final boiling points of the feed, it is possible to produce directly, without intermediate storage tanks, the final products with the desired initial and final boiling points.
35 Moreover, this integration of hydrogenation and fractionation allows an optimized thermal integration with reduced number of equipment and energy savings.

White oils

The invention thus discloses a white oil cut that can be obtained by the process of the invention. The white oil cut typically has an initial boiling point higher than 250°C and an aromatic content of less than 1000 ppm by weight. The aromatic content can be measured by UV spectrometry.

5 According to a preferred embodiment, the aromatic content of the white oil is less than 200 ppm by weight, preferably less than 100 ppm by weight, more preferably less than 80 ppm by weight.

According to a preferred embodiment, the white oil obtained in the invention has boiling points within the range of from 300 to 420°C, preferably from 310 to 410°C.

10 According to an embodiment, the white oil obtained in the invention has one or several of the following features:

- a density at 15°C ranging from 0.8100 to 0.8700 g/mL, preferably from 0.8200 to 0.8600 g/mL, and/or
- a Saybolt colour higher than or equal to +30, measured according to NF M 07003 standard, and/or
- 15 - a Cleveland Flash Point ranging from 150 to 250°C, preferably from 175 to 225°C, more preferably from 190 to 200°C, measured according to ASTM D92 standard, and/or
- a viscosity at 40°C ranging from 6 to 25 mm²/s, preferably from 7.5 to 21 mm²/s, and/or
- 20 - an aniline point of at least 70°C, preferably at least 90°C, measured according to ISO 2977 standard, and/or
- a pour point ranging from -40°C to +10°C, preferably from -30°C to -5°C, measured according to ISO 3016 standard, and/or
- 25 - a refractive index at 20°C according to ASTM D1218 ranging from 1.4500 to 1.4850, preferably from 1.4600 to 1.4800.

The following example illustrates the invention without limiting it.

30 EXAMPLES

The unit that was used in the examples is a unit comprising two reactors in series.

Example 1: Production of white oil from a base oil A

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Example 1a: Description of the feed and preparation of the unit

A base oil A having the features detailed in table 1 below has been submitted to a catalytic hydrogenation.

Table 1: base oil A characterization analysis

Characteristics and methods	values	units
density @ 15°C	0.8304	g/mL
Distillation ASTM D86 T°C @ IBP	282.2	°C
T°C @ 5% vol	299.8	°C
T°C @ 10% vol	306.8	°C
T°C @ 20% vol	316.2	°C
T°C @ 30% vol	324.2	°C
T°C @ 40% vol	331.8	°C
T°C @ 50% vol	338.3	°C
T°C @ 60% vol	344.7	°C
T°C @ 70% vol	351.2	°C
T°C @ 80% vol	358.5	°C
T°C @ 90% vol	368.3	°C
T°C @ 95% vol	376.4	°C
T°C @ FBP	377.4	°C
Flash point Pensky Martens ASTM D 93	147.6	°C
Kinematic Viscosity ASTM D445 @ 40°C	7.684	mm ² /s
Aromatics by UV method	200	ppm
Sulphur by ASTM D4294	0.1	ppm

The catalyst used was a Nickel supported on alumina catalyst. The catalyst has been reduced in situ with hydrogen before introducing the feed, for example with 80 Nl/h of hydrogen for 1 hour.

Before introducing the base oil feed A, the catalytic system has been first subjected to a stabilization phase using a standard gas oil feed, at 150°C, LHSV of 1.5 h⁻¹ and a hydrogen pressure of 100 bars. After 60 hours on stream, a stable monoaromatic content of 8 ppm by weight was reached.

Example 1b: Process of the invention

Then, after the stabilization phase, a catalytic dehydrogenation has been performed on the base oil feed A detailed in table 1 with the following conditions: a temperature of 130°C, a LHSV of 1 h⁻¹ and a pressure of 100 bars.

The unit of the catalytic hydrogenation stabilized to a monoaromatic content outlet of 80 ppm by weight.

The sample was then distilled in two fractions. The heaviest fraction (315°C +) has a monoaromatic content of 95 ppm by weight.

5 This heaviest fraction satisfies the specifications of a white oil, in particular the purity criterion of the according to European Pharmacopeia of the monography of the liquid paraffins (EuPh 6.0 01/2008), of the US Pharmacopoeia Light Mineral Oil, USP32 – NF 27, and of the Japanese Pharmacopoeia Light liquid Paraffin.

Example 1c: Control of the deactivation of the catalyst

10 Finally, after 100 hours of test (stabilization of example 1a and example 1b), the unit was set to the same conditions as of the stabilization phase (with the standard gas oil feed) and maintained for about 100 more hours, a stable monoaromatic content of 7 ppm by weight was reached, which indicates that no catalyst deactivation occurred.

During all the experiment, the mass balance was > 99 %, calculated according to the following formula:

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$$mass\ balance = 1 - \left(\frac{IN - OUT}{IN} \right)$$

wherein IN represents the total mass of liquid and gas at the inlet of the reactor and OUT represents the total mass of liquid and gas at the outlet of the reactor.

20 Example 2: Production of white oil from a base oil B

Example 2a: Description of the feed and preparation of the unit

A base oil B having the features detailed in table 2 below has been submitted to a catalytic hydrogenation.

Table 2: base oil B characterization analysis

Characteristics and methods	values	units
Density @ 15°C	0.8311	g/mL
Distillation ASTM D86 T°C @ IBP	339.9	°C
T°C @ 5% vol	352.1	°C
T°C @ 10% vol	354.3	°C
T°C @ 20% vol	355.2	°C
T°C @ 30% vol	356.4	°C
T°C @ 40% vol	357.3	°C
T°C @ 50% vol	360.3	°C
T°C @ 60% vol	361.7	°C
T°C @ 70% vol	364.9	°C
T°C @ 80% vol	368.3	°C
T°C @ 90% vol	373.9	°C
T°C @ 95% vol	379.1	°C
T°C @ FBP	379.1	°C
% Recovered @ 350°C (*)	2.8	% vol
% Recovered @ 370°C (*)	83.1	% vol
Recovered %vol	97.5	% vol
Residue %vol	2.5	% vol
Loss %vol	0	% vol
(*) % Loss included Yes / No	y	-
Flash point Pensky Martens ASTM D 93	206	°C
Pour point ASTM D5950 (rep. D97)	<-30	°C
Kinematic Viscosity ASTM D445 @ 20°C	24.27	mm ² /s
Kinematic Viscosity ASTM D445 @ 40°C	11.46	mm ² /s
Total Nitrogen by Chemiluminescence	10.6	ppm
Sulphur by UVF ppm	2.8	ppm
Monoaromatics by UV method	301.8	mg/kg

The catalyst used was a Nickel supported on alumina catalyst. The catalyst has been reduced in situ with hydrogen before introducing the feed, for example with 80 NI/h of hydrogen for 1 hour.

Before introducing the base oil feed A, the catalytic system has been first subjected to a stabilization phase using a standard gas oil feed, at 150°C, LHSV of 1.5 h⁻¹ and a hydrogen pressure of 100 bars. After 60 hours on stream, a stable monoaromatic content of 8 ppm by weight was reached.

Example 2b: Process of the invention

After stabilization phase, temperature was decreased to 130°C (ramp, 20 °C/h) and pressure reduced to 50 bar. Base oil feed B was introduced. The test continued according to the conditions detailed in table 3 below.

Example 2c: Control of the deactivation of the catalyst

Finally, after 530 hours of test, the unit was set to the same conditions as of the stabilization phase and maintained for about 100 more hours, a stable monoaromatic content of 8 ppm by weight was reached (see Fig. 1), which indicates no catalyst deactivation occurred.

Fig. 1 presents the monoaromatic content in ppm by weight of the outlet stream during the phase I which is the stabilization phase, the phase II comprising the five conditions detailed in table 3, and the phase III corresponding to example 2c performed in order to evaluate the deactivation of the catalyst.

At the end of each condition of example 2b (cond. 1 to 5), the sample that has been taken up satisfies the specifications for a white oil (purity criterion of pharmacopeia).

Table 3: Description of the phases of the example 2

	Stabilization phase (example 2a)	Example 2b Test (According to the invention)					Example 2c
		Conditions					
		Cond.1	Cond.2	Cond.3	Cond.4	Cond.5	
	Fresh feed	Fresh feed			Diluted feed**		Fresh feed
Pressure (barg)	100	50	100	100	50	100	100
LHSV (h ⁻¹)	1.5	1	1	1	1	1	1.5
Feed rate (mL/h)	168	112	112	112	112	112	168
H ₂ /feed (NL/L)	100	54	54	54	54	54	100
Hydrogen flow (NL/h)	16.8	6	6	6	6	6	16.8
WABT* (°C) (Reactor 1)	150	130	130	150	130	130	150
WABT* (°C) (Reactor 2)	150	130	130	150	130	130	150
Duration of condition (h)	120	50	190	150	50	90	100

*WABT: Weight Average Bed Temperature

**43%wt of fresh feed and 57%wt of recycled product from the outlet (exit) of the reactor

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During all the experiment, the mass balance was > 99 %, calculated according to the following formula:

$$\text{mass balance} = 1 - \left(\frac{IN - OUT}{IN} \right)$$

- 10 wherein IN represents the total mass of liquid and gas at the inlet of the reactor and OUT represents the total mass of liquid and gas at the outlet of the reactor.

Example 3: Evaluation of the effect of the temperature

- 15 Example 3a: Description of the feed and preparation of the unit

A base oil C having the features detailed in table 4 below has been submitted to a catalytic hydrogenation.

Table 4: base oil C characterization analysis

Characteristics and methods	values	units
Density @ 15°C	0.8298	g/mL
Distillation ASTM D86 T°C @ IBP	322.7	°C
T°C @ 5% vol	345.2	°C
T°C @ 10% vol	350.1	°C
T°C @ 20% vol	352.5	°C
T°C @ 30% vol	355.0	°C
T°C @ 40% vol	357.7	°C
T°C @ 50% vol	360.3	°C
T°C @ 60% vol	363.3	°C
T°C @ 70% vol	366.5	°C
T°C @ 80% vol	370.8	°C
T°C @ 90% vol	376.8	°C
T°C @ 95% vol	381.8	°C
T°C @ FBP	381.9	°C
Flash point Pensky Martens ASTM D 93	206	°C
Pour point ASTM D5950 (rep. D97)	-36	°C
Aniline Point ASTM D611	107.2	°C
Kinematic Viscosity ASTM D445 @ 40°C	11.37	mm ² /s
Sulphur by UVF ppm	0.1	ppm
Monoaromatics by UV method	522	mg/kg

- 5 The catalyst used was a Nickel supported on alumina catalyst. The catalyst has been reduced in situ with hydrogen before introducing the feed, for example with 80 NI/h of hydrogen for 1 hour.

- 10 Before introducing the base oil feed A, the catalytic system has been first subjected to a stabilization phase using a standard gas oil feed, at 150°C, LHSV of 1.5 h⁻¹ and a hydrogen pressure of 100 bars. After 60 hours on stream, a stable monoaromatic content of 8 ppm by weight was reached.

Example 3b: Process of the invention

- 15 Then, after the stabilization phase, a catalytic dehydrogenation has been performed on the base oil feed A detailed in table 4 with the following conditions: a temperature of 130°C, a LHSV of 1 h⁻¹ and a pressure of 100 bars.

The test continued by performing temperature steps of 20°C, up to 210°C.

Fig. 2 presents the results obtained during the test, i.e. the proportions of compounds in function of the boiling point of the compounds.

It can be clearly observed in Fig. 2 that with increasing temperature some cracking
5 reactions occur, producing some lighter components.

Example 4: Production of white oil from a base oil D

Example 4a: Description of the feed and preparation of the unit

10 A base oil D having the features detailed in table 5 below has been submitted to a catalytic hydrogenation.

Table 5: base oil D characterization analysis

Characteristics and methods	Values	Units
Density @ 15°C	0.8521	g/mL
Distillation ASTM D86 T°C @ IBP	323.6	°C
T°C @ 5% vol	341.2	°C
T°C @ 10% vol	346.7	°C
T°C @ 20% vol	351.3	°C
T°C @ 30% vol	356.0	°C
T°C @ 40% vol	360.6	°C
T°C @ 50% vol	366.1	°C
T°C @ 60% vol	371.2	°C
T°C @ 70% vol	377.7	°C
T°C @ 80% vol	385.7	°C
T°C @ 90% vol	398.1	°C
T°C @ 95% vol	410.6	°C
T°C @ FBP	414.0	°C
Flash point Pensky Martens ASTM D 93	185.5	°C
Pour point ASTM D5950 (rep. D97)	-22	°C
Aniline Point ASTM D611	103.8	°C
Kinematic Viscosity ASTM D445 @ 20°C	35.79	mm ² /s
Kinematic Viscosity ASTM D445 @ 40°C	15.58	mm ² /s
Total Nitrogen by Chemiluminescence	1.6	ppm
Aromatic content by UV method	<100	ppm
Sulphur by UVF ppm	2.16	ppm

The catalyst used was a Nickel supported on alumina catalyst. The catalyst has been reduced in situ with hydrogen before introducing the feed, for example with 80 Nl/h of hydrogen for 1 hour.

5 Before introducing the base oil feed D, the catalytic system has been first subjected to a stabilization phase using a gas oil feed, at 150°C, LHSV of 1.5 h⁻¹ and a hydrogen pressure of 100 bars. After 60 hours on stream, a stable monoaromatic content of 8 ppm by weight was reached.

10 Example 4b: Process of the invention

After stabilization, the unit was set to the following conditions (Cond.1) : T = 150°C, LHSV = 1h⁻¹, P = 50 bars. Base oil feed D was introduced. The test continued by increasing pressure to 100 bars for a second conditions (Cond.2). Table 6 presents the results obtained during the test for the 2 testing conditions.

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Table 6: product characterization analysis (condition 1 and 2).

Analyze	Method	Unit	Cond.1	Cond.2
Density at 15°C	EN ISO 12185	kg/m ³	851.9	852.0
Appearance	VISUAL		Clear & Bright	Clear & Bright
Saybolt colour	NF M 07003		>+30	>+30
Cleveland Flash Point	ASTM D92	°C	192	194
Viscosity at 40°C	EN ISO 3104	mm ² /s	15.625	15.659
Viscosity at 100°C	EN ISO 3104	mm ² /s	3.482	3.488
Aniline Point	ISO 2977	°C	104.1	104.4
Pour point	ISO 3016	°C	-24	-27
Aromatic content	UV method	ppm	46	32
Sulfur content	NF M 07059	ppm	<1	<1
Refractive index			1.4674	1.4674
Distillation D86		Unit		
Initial Point	EN ISO 3405	°C	321.6	317.8
2% Point	EN ISO 3405	°C	335.0	336.7
5% Point	EN ISO 3405	°C	341.8	344.5
10% Point	EN ISO 3405	°C	346.4	348.3
20% Point	EN ISO 3405	°C	351.3	352.7
30% Point	EN ISO 3405	°C	356.1	357.1
40% Point	EN ISO 3405	°C	361.2	361.7
50% Point	EN ISO 3405	°C	366.0	366.3
60% Point	EN ISO 3405	°C	370.8	371.1
65% Point	EN ISO 3405	°C	372.9	373.5
70% Point	EN ISO 3405	°C	376.2	376.7
80% Point	EN ISO 3405	°C	383.5	384.1
90% Point	EN ISO 3405	°C	394.0	395.5
Dry Point	EN ISO 3405	°C	401.0	402.6
European Pharmacopeia evaluation (purity)			Conform	Conform

Example 5: Production of white oil from a base oil E5 Example 5a: Description of the feed and preparation of the unit

A base oil E having the features detailed in table 7 below has been submitted to a catalytic hydrogenation.

Table 7: base oil E characterization analysis

Characteristics and methods	Values	Units
Density @ 15°C	0.8366	g/mL
*Distillation ASTM D2887 T°C @ IBP	317.0	°C
T°C @ 5%wt	361.0	°C
T°C @ 10%wt	367.0	°C
T°C @ 20%wt	393.0	°C
T°C @ 30%wt	405.0	°C
T°C @ 40%wt	416.0	°C
T°C @ 50%wt	426.0	°C
T°C @ 60%wt	437.0	°C
T°C @ 70%wt	450.0	°C
T°C @ 80%wt	466.0	°C
T°C @ 90%wt	489.0	°C
T°C @ 95%wt	510	°C
T°C @ FBP	561	°C
Flash point Pensky Martens ASTM D 93	204.0	°C
Pour point ASTM D5950 (rep. D97)	36	°C
Aniline Point ASTM D611	103.8	°C
Kinematic Viscosity ASTM D445 @ 20°C	4.181	mm ² /s
Kinematic Viscosity ASTM D445 @ 40°C	18.07	mm ² /s
Total Nitrogen by Chemiluminescence	1.3	ppm
Total Aromatic content by HLPC IP391	5.1	%wt
Sulphur by UVF ppm	4.8	ppm

*ASTM D86 distillation was not carried out due to the high distillation points

The catalyst used was a Nickel supported on alumina catalyst. The catalyst has been
5 reduced in situ with hydrogen before introducing the feed, for example with 80 Nl/h of
hydrogen for 1 hour.

Before introducing the base oil feed E, the catalytic system has been first subjected to
a stabilization phase using a gas oil feed, at 150°C, LHSV of 1.5 h⁻¹ and a hydrogen pressure
10 of 100 bars. After 60 hours on stream, a stable monoaromatic content of 8 ppm by weight
was reached.

Example 5b: Process of the invention

After stabilization, the unit was set to the following conditions (Cond.1): T = 150°C, LHSV = 1h⁻¹, P = 130 bars. Base oil feed E was introduced. The test continued by increasing the temperature, pressure, and dilution ratio (hydrogenated Base oil feed E/fresh Base oil feed E. Table 8 presents the detailed operating conditions.

5

Table 8: Description of the phases of the example 5

	Cond.1	Cond.2	Cond.3	Cond.4
	Fresh feed		Diluted feed	
Pressure (barg)	130	130	150	150
LHSV (h ⁻¹)	1	1	0.5	0.2
Feed rate (mL/h)	112	112	56	22.4
H ₂ /feed (NL/L)	150	150	130	130
Hydrogen flow (NL/h)	16.8	16.8	7.3	2.9
WABT* (°C) (Reactor 1)	150	190	190	210
Dilution ratio (hydrogenated/fresh feed)	-	-	1.5	2.3

*WABT: Weight Average Bed Temperature

Table 9 presents the results obtained during the test for each of the testing conditions.

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Table 9: product characterization analysis (condition 1, 2, 3 and 4).

Analyze	Method	Unit	Cond.1	Cond.2	Cond.3	Cond.4
Density at 15°C	EN ISO 12185	kg/m ³	834.8	834.8	834.5	835.2
Aromatic content	UV method	ppm	1106	592	276	49
Sulfur content	NF M 07059	ppm	<0.6	<0.6	<0.6	<0.6

CLAIMS

1. A process for producing a white oil having an initial boiling point of at least 250°C, the process comprising a step of catalytically hydrogenating a base oil feedstock at a temperature of from 120 to 210°C, at a pressure of from 30 to 160 bars and a liquid hourly space velocity of 0.2 to 5 hr⁻¹, the base oil feedstock comprising less than 5 ppm by weight of sulphur.
2. The process according to claim 1, wherein the base oil feedstock comprises less than 3 ppm by weight, preferably less than 1 ppm by weight of sulphur.
3. The process according to claim 1 or 2, wherein the base oil feedstock has an initial boiling point ranging from 250 to 350°C and a final boiling point ranging from 350 to 600°C.
4. The process according to any one of claim 1 to 3, wherein the base oil feedstock has a viscosity at 40°C of at least 6 cSt, preferably at least 7 cSt, more preferably at least 7,5 cSt.
5. The process according to any one of claims 1 to 4, wherein the base oil feedstock is selected from Groups II, III, IV and mixture thereof, preferably from Groups II and III, more preferably from Group III, of the API classification.
6. The process according to any one of claims 1 to 5, wherein the base oil feedstock is selected from oils issued from a hydrocracking process and from oils issued from a deep desulphurization process.
7. The process according to any one of claims 1 to 6, wherein hydrogenating step is performed at a temperature ranging from 150 to 200°C, preferably from 150 to 190°C.
8. The process according to any one of claims 1 to 7, wherein the hydrogenating step is performed at a pressure ranging from 50 to 150 bars, preferably from 50 to 130 bars.
9. The process according to any one of claims 1 to 8, wherein the hydrogenating step is performed at a liquid hourly space velocity ranging from 0.4 to 3 hr⁻¹, preferably from 0.5 to 1.5 hr⁻¹.
10. The process according to any one of claims 1 to 9, wherein the catalyst is a nickel catalyst, preferably a supported nickel catalyst.

11. The process according to any one of claims 1 to 10, wherein the catalyst is not in a sulfided form when the hydrogenation step is started.
- 5 12. The process according to any one of claims 1 to 11, wherein the hydrogenating step is performed in a unit comprising at least 2 reactors, preferably in series.
13. The process according to any one of claims 1 to 12, further comprising a fractionating step, preferably performed after the hydrogenating step.
- 10 14. The process according to any one of claims 1 to 13, wherein the white oil has an aromatic content below 1000 ppm by weight, preferably below 500 ppm by weight, more preferably below 300 ppm by weight, even more preferably below 200 ppm by weight.