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# (54) De-ashing lubricating oils.

(57) A process for purifying used oil and the like comprising reacting the oil with a treating agent that will form solids from the ash-forming components of the oil, separating the solids from the oil, and subjecting at least a portion of the oil to a two stage hydrotreatment with interstage removal of hydrogen sulfide and/or ammonia.

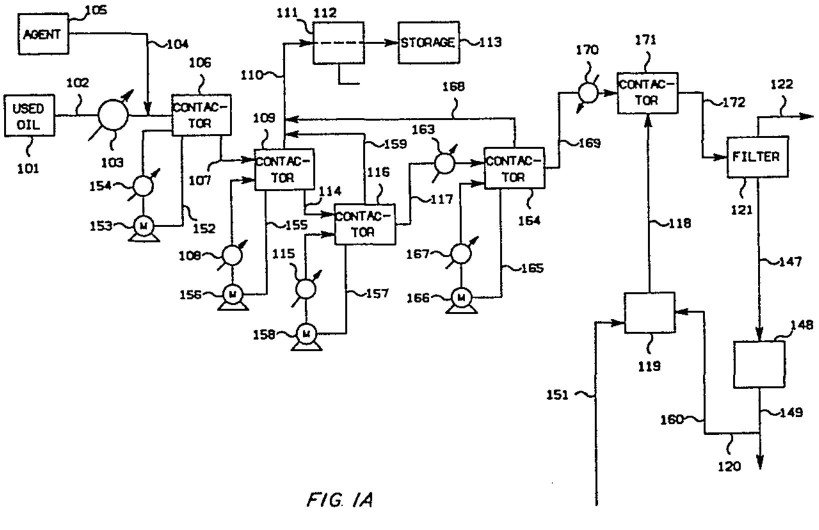

FIG. 1A

EP 0 077 564 A2

Croydon Printing Company Ltd.

# DE-ASHING LUBRICATING OILS

This invention relates to a method for reducing the ash-content of lubricating oil containing ash-forming components. In another aspect this invention relates to a method for the treatment of used lubricating oils to obtain purified oil suitable for use as fuel oil, in grease formulations, or in the preparation of lubricating oil formulations.

Used motor oil has been estimated as being generated in the United States at a rate of about 1.1 billion gallons per year. Some of this used oil has been used as furnace oil and some has been used on rural dirt roads for dust control. Much of the oil has been merely discarded in sewers, dumps, and back alleys. With the ever decreasing petroleum reserves, it becomes more and more essential that this used oil be saved and used as long as possible.

One major obstacle to re-use of used oil in many applications involves the presence of various ash-forming impurities that remain dispersed in the oil due to the very effective dispersant characteristics of the additives in modern day lubricant systems.

Materials contained in a typical used crankcase oil that are considered to contribute to the ash content of the oil include sub-micron size carbon particles, inorganic materials such as atmospheric dust, metal particles, lead and other metal compounds originating from fuel combustion. Besides lead, which is generally present at concentrations of 1.0 to 2.5 weight percent, appreciable amounts of zinc, barium, calcium, phosphorus and iron are also present in the used crankcase oil.

Examination of the used oil under an optical microscope at 600 magnifications reveals the very effective dispersant characteristics of modern day lube oils. The particle size of the particulates is estimated from this microscopic examination to be 0.1-1.0 microns with virtually no occurrence of agglomerates in the oil.

The presence of the ash-forming components in used oil puts limits on the extent to which the material can be used economically without ecological damage. For example, reuse of the used oil as fuel oil can give rise to serious atmospheric pollution when the oil contains in excess of one percent lead. Also, such fuel oil often results in burner and refractory maintenance costs that offset the purchase price differential between used oil and regular furnace oil.

Clearly, it is in the national interest to provide economical ways of removing the impurities from used oil so that it can be reused practically.

Recently, a technique of purifying used oil has been developed in which the used oil is reacted with an aqueous solution of an ammonium salt treating agent, then the water phase is removed, and the resulting oil phase-containing mass is separated by filtration. Such a technique is described in U. S. Patent No. 4,151,072, the disclosure of which is incorporated herein by reference.

An improvement of that process is disclosed in U. S. 4,247,389, the disclosure of which is also incorporated herein by reference. The improvement of that patent involves a special heat soak step that results in improved separation of the ash-forming components from the oil.

In both of the above-mentioned processes, it has been found to often be desirable to subject at least portions of the de-ashed oil to decolorizing and deodorizing step. Typically the decolorizing and deodorizing step has involved a hydrotreatment. The present invention is based upon the applicants' discovery that an improvement in the integrated process is obtained if the hydrotreatment is conducted in two stages with interstage flashing to remove hydrogen sulfide and ammonia present in the effluent from the first stage before said effluent is passed to the second step.

## Summary of the Invention

In accordance with the present invention, there is provided a process for the production of an essentially ash-free oil stock from lubricating oil containing ash-forming components in which the lubricating oil is contacted with an aqueous solution of a treating agent which reacts with ash-forming components to form solids which can be separated from the oil, the major portion of the water is removed from the oil-water mixture, solids are removed from the oil, and at least a portion of the oil is subjected to hydrotreatment wherein the hydrotreatment is conducted in two stages with interstage flashing to remove hydrogen sulfide and ammonia present in the effluent from the first stage before said effluent is passed to the second stage.

## Brief Description of the Drawings

FIGURE 1A is a schematic process flow diagram of a demetallizing process that can be used in conjunction with the present invention.

FIGURE 1B is a schematic process flow diagram of a process for hydrotreating demetallized oil in accordance with the present invention.

## Detailed Description of the Invention

The present invention is applicable to the de-ashing of oil in which ash-forming components can be rendered removable by the treating agent. The invention is particularly applicable to the purification of oils that have been used for internal combustion engine lubrication purposes such as crankcase oils, e.g., in gasoline engines or diesel engines. Other sources of used oils include steam-turbine oils, transmission and gear oils, steam-engine oils, hydraulic oils, heat-transfer oils and the like.

The oils generally used for preparing internal combustion engine lubricants are the refinery lubricating cuts from paraffin-base, mixed-base, or naphthenic crudes. Their viscosities are generally in the range of from about 100 to about 1,800 SUS at 100°F. The oils also contain various additives such as oxidation inhibitors (e.g., barium, calcium and zinc alkyl thiophosphates, di-t-butyl-p-cresol, etc.), antiwear agents (e.g., organic lead compounds such as lead diorganophosphorodithioates, zinc dialkyldithiophosphates, etc.), rust inhibitors (e.g., calcium and sodium sulfonates, etc.), dispersants (e.g., calcium and barium sulfonates and phenoxides, etc.), viscosity index improvers (e.g., polyisobutylenes, poly-(alkylstyrenes), etc.), detergents (e.g., calcium and barium salts of alkyl benzene sulfonic acids) and ashless-type detergents such as alkyl-substituted succinimides, etc.

If desired, water entrained in the untreated used lubricating oil can be removed before use of same in the process of this invention. Such a separation can be readily achieved by removal of the water phase which may occur in the storage tanks for the used lubricating oil.

The ammonium salt treating agents which are useful in the process of the present invention are those selected from the group consisting of ammonium sulfate, ammonium bisulfate, ammonium phosphate, diammonium hydrogen phosphate, ammonium dihydrogen phosphate, ammonium thiosulfate, ammonium polyphosphates such as ammonium metaphosphate, urea sulfate, guanidine sulfate, urea phosphate, and guanidine phosphate, and mixtures thereof. Said treating agents can be formed in situ if desired as, for example, by combining ammonia and/or ammonium hydroxide with sulfuric acid and/or phosphoric acid and/or an ammonium hydrogen sulfate or phosphate, i.e., ammonium bisulfate, diammonium hydrogen phosphate, and/or ammonium dihydrogen phosphate. When the treating agent is formed in situ, the reactants employed can be introduced at the same time, or one after the other.

Although the concentration of treating agent in the aqueous solution of treating agent is not critical and more dilute solutions can be used, the economics of the process are enhanced by the use of relatively concentrated solutions in order that the amount of water to be removed subsequently will not be great. Generally the concentration of treating agent in the aqueous solution will be within the range of about 30 to about 95 weight percent, typically about 80 weight percent, of that in an aqueous solution that is saturated with the treating agent at 25°C. Frequently some water will be found in used oil, and in these instances the concentration of the treating agent can be adjusted accordingly.

In the process of this invention, the treating agent should preferably be employed in an amount at least sufficient to react with essentially all of the metal constituents in the used oil. Although the weight ratio of the treating agent to the oil can vary greatly, depending in part upon the nature and concentration of metal-containing components in the oil and on the particular treating agent employed, generally it will be within the range of about 0.002:1 to about 0.05:1, most often being within the range of about 0.005:1 to about 0.015:1, and typically being about 0.01:1. Although larger amounts of treating agent can be used, in most instances this would be wasteful of treating agent.

Water can be removed from the mixture resulting from the combination of the aqueous solution and the oil by any suitable means. Distillation is the preferred method of removing water. Generally, the distillation is carried out at a temperature in the range of about 100° to about 140°C and a pressure in the range of about 5 to about 25 psig for a period of time sufficient to effect removal of a major portion of the water. Light hydrocarbons contained in the oil that boil under the distillation conditions, e.g., gasoline, will be, of course, separated from the oil along with the water.

Preferably the solids are separated from the oil by filtering. Generally, it is desirable to use a filter aid in the separation process. Filter aids which are useful in the practice of the invention include those selected from the group consisting of diatomaceous earth, perlite, and cellulose fibers. Presently preferred is diatomaceous earth.

The heat soak step described in U. S. 4,247,389 is carried out at a temperature in the range of about 320° to about 420°C for a period of time sufficient to improve the filtration rate of the oil. Preferably the heat soak is carried out at a temperature in the range of about 340°C to about 370°C. Generally, the time that a volume of oil will be exposed to the heat soak will be in the range of about 5 minutes to about an hour, more preferably about 15 minutes to about 30 minutes.

In some cases, especially in handling oils that have unusually high amounts of heavies, it is desirable to employ a distillation between the filtration step and the hydrotreating step. All or only part of the distillation fractions can be subjected to the hydrotreatment. Often some of the lighter fractions will be sufficiently clear that hydrotreatment will not be necessary. Further, in some cases, it may be determined that the end use of the heavy fraction is such that hydrotreatment of the heavy bottoms fraction is not needed.

A further understanding of the present invention and its advantages will be provided by reference to FIGURES 1A and 1B which sets forth a typical process employing the features of the present invention.

In FIGURE 1A, used oil from storage tank 101 is passed via line 102 to heater 103 and contactor 106. Aqueous treating agent such as diammonium hydrogen phosphate from makeup tank 105 is introduced via line 104. If desired, agent precursors ammonia, phosphoric acid, and water can be introduced into the heated oil downstream of heater 103, thereby forming the treating agent in situ in line 102 and contactor 106. The oil from heater 103 is passed in admixture with treating agent into the first agitated contactor 106 wherein the mixture is maintained under agitation for a time sufficient to react with at least a portion of the ash-forming components in the oil. Preferably, a recycle stream is passed through conduit 152 to pump 153 and then through heater 154 before its return to contactor 106, thereby providing heat and agitation to the contents of the contactor. Stirring means also can be employed.

Thereafter the mixture is passed via conduit 107 to second contactor 109, which is maintained at a temperature in the range of about 110° to about 140°C, for a time sufficient to effect distillation of a major portion of the water and at least some of the light hydrocarbons present therein. Thus, while retained in contactor 109, essentially all of the water and at least a portion of the light hydrogen components of the mixture are removed via line 110 and passed to separator 111 wherein a hydrocarbon layer and a water layer are allowed to form. The hydrocarbon phase can then be transferred via line 112 to storage 113. The water layer can be removed and discarded or employed for any desired purpose. Preferably, a recycle stream is passed through conduit 155 to pump 156 and then through heater 108 before its return to contactor 109, thereby providing heat and agitation to the contents of the reactor. Stirring means also can be employed.

The resulting mixture comprising a hot oil phase which is essentially free of water is passed via conduit 114 to a third contactor wherein it is subjected to agitation and a temperature in the range of about 140 to about 200°C to remove additional water and lighter components. Preferably, a recycle stream is passed through conduit 157 to pump 158 and then through heater 115 before its return to contactor 116, thereby providing heat and agitation to the contents of the contactor. Any residual water and light hydrocarbons are removed from contactor 116 via line 159.

If desired, any one or two or all of contactors 106, 109 and 116 can be provided with jackets heated by steam or other source of heat to aid in maintaining the contents of the contactors at the desired temperatures. Any one or two or all of contactors 106, 109 and 116 can be equipped with stirrers to provide additional agitation, in an operable but presently less preferred arrangement, a stirrer in any one or more of the three contactors can be used instead of the recycle system employed with the corresponding one or more of the three contactors, any additional heating being provided by heaters in the line ahead of the contactors and/or by heated jackets around the contactors. Also, if desired, any one or two or all of conduits 102, 107 and 114 can feed into the recycle stream for contactors 106, 109 and 116, respectively, i.e., into conduits 152, 155 and 157, respectively, instead of directly into the respective contactor as shown. In one preferred technique the feed in conduit 102, rather than being passed directly into contactor 106, is passed into conduit 152 at the inlet side of pump 153. In a still more preferred technique, pump 153 is a high-volume pump that will cause the oil to flow in the turbulent flow range so as to promote heat transfer and decrease scaling in the conduit 152.

The heated oil from contactor 116 is passed via conduit 117 through heater 163 to a fourth contactor 164 wherein the mixture is subjected to agitation at a temperature in the range of about 320° to about 420°C for a period of time sufficient to result in a product which when later filtered will contain less ash than it would contain if it had not been so heated. Preferably, a recycle stream is passed through conduit 165 to pump 166 and then through heater 167 before its return to contactor 164, thereby providing heat and agitation to the contents of contactor 164. Any residual water or light components can be removed from contactor 164 via line 168.

Treated oil from contactor 164 is passed through conduit 169 through a cooler 170 wherein the oil is cooled to a temperature in the range of about 150° to about 180°C and then passed into a fifth contactor

171 wherein it is admixed with filter aid provided via conduit 118, preferably as a slurry in light hydrocarbons provided from makeup tank 119. In a presently, preferred embodiment, not illustrated, the oil from contactor 164 is cooled at least in part as a result of passing in indirect heat exchange with the feed passing through line 102 whereby the heat in the oil in line 169 is used to heat the feed oil in line 102.

Following admixture of filter aid, the resulting mixture is passed via line 172 to filter 121, which optionally can be precoated with filter aid. The use of the heat soak step of the present invention can in many cases result in a reduction in the amount of filter aid required for a suitable filtration rate.

Filter cake from filter 121 is removed via line 147 and optionally passed to furnace 148 from which, following burning or calcination, at least a portion of the resulting ash containing filter aid can be passed to waste via line 149 or recycled via conduits 120 and 160 to slurry makeup tank 119 for further use in the system. Fresh filter aid is added through conduit 160. Light hydrocarbons for use in preparing the slurry can be recovered from the integrated process and can be passed to tank 119 via conduit 151.

The filtered oil is passed to a run storage tank 200 via line 122. See FIGURE 1B. If fractionation of the oil is desired at this point in the process, the filtered oil is passed to a distillation column 202. The column can be operated to yield any desired number of fractions. In the drawing only three fractions are shown, a lights fraction, an intermediate fraction, and a bottoms fraction. Typically the bottoms fraction includes substantially all of the components of the filtered oil that have boiling points higher than 1050°F. Each of the fractions are passed from the distillation zone to run storage tanks. In the drawing, the lights fraction is passed to tank 204, the intermediate fraction to tank 205, and the bottoms fraction to tank 206. By means of properly placed valves, the various fractions can each individually be passed to the hydrotreatment zone. Alternatively, one or more of the fractions can be combined and then subjected to hydrotreatment. In another alternative, all or part of individual fractions can be recovered for use without hydrotreatment.

If no distillation is used, then the filtered oil can be passed directly into the hydrotreating section from storage tank 200. The present invention is particularly useful in such operations since it has been discovered by the inventors that the higher boiling components of the oil contain numerous compounds that upon hydrogenation result in amounts of $H_2S$ and/or ammonia that seriously affects the effectiveness of the hydrogenation.

Regardless of whether distillation is employed or whether it is undistilled oil that is passed for hydrotreatment in the illustrated embodiment, the oil that is to be hydrotreated is combined with hydrogen provided via line 208 and the combined stream passed through heater 210. The resulting hot oil containing the added hydrogen is then passed to contactor 212 wherein decomposition is effected of sulfonates contained in the oil.

While it is presently preferred that contactor 212 contain bauxite or an activated carbon adsorbent bed therein, this unit can employ other adsorbents such as those selected from the group consisting of silica gel, clay, activated alumina, combinations thereof, and the like. The adsorbent serves to effect breakdown and decomposition of the ammonium salts of sulfonic acids and the ashless detergents contained in the oil. The adsorbent further serves to collect a small portion of the resulting products and thus precludes passage of such undesirable decomposition products to the hydrotreater. Such adsorbents can be regenerated by conventional means and reused.

Preferably, the adsorbent contains about 0.2 to about 20 weight percent of at least one metal selected from the group consisting of Group VIB and Group VIII metals, this weight percent being based on the total weight of modified adsorbent. This modified adsorbent can be prepared by impregnation of the adsorbent with an aqueous solution of a water-soluble compound of a Group VIB or Group VIII metal, followed by evaporation of water. Water-soluble compounds presently preferred for this use are iron compounds such as ferric ammonium oxalate, ferric ammonium citrate, ferric sulfate, and ferrous ammonium sulfate.

The resulting treated oil is thereafter passed from contactor 212 to the first hydrotreatment reactor 214. If desired additional hydrogen can be added to the oil after its treatment in contactor 212.

In hydrotreater 214, the oil is subjected to hydrogenation conditions suitable for hydrogenating unsaturated materials and to effect decomposition of residual sulfur, oxygen, and nitrogen bodies. Any suitable hydrogenation catalyst can be employed. Examples of suitable catalysts for use in hydrotreater 128 include those selected from the group consisting of Group VIB and Group VIII metals and combinations thereof supported on refractory oxides such as those conventionally used in hydrodesulfurization processes. A typical example of a suitable catalyst is Filtrol RHPC available from the Filtrol Corporation.

After exiting hydrotreater 214, the oil is passed to a phase separator 216 wherein hydrogen sulfide, ammonia, and light hydrocarbons are flashed overhead. The oil from the separator is then passed to heater 218, preferably after being combined with more hydrogen. The heated oil containing hydrogen is then passed to the second hydrotreater reactor 220. The catalyst employed in reactor 220 can be the same or different as that employed in reactor 214.

The oil exiting hydrotreater 220 is passed to phase separator 222 wherein again hydrogen sulfide, ammonia, and light hydrocarbons are flashed overhead.

The overhead streams from separators 216 and 222 are preferably combined and subjected to a scrubber 224 capable of removing HCl, $NH_3$, and $H_2S$ from the gases. Overhead from the scrubber can then be vented or subjected to further processing for recovery of any desired value therefrom.

The oil passing out of separator 222 is then passed to a stripper 226 wherein light hydrocarbons are separated from those of the oil boiling range. Preferably, the stripping is conducted with steam although other gases can be employed.

The overhead from the stripper 226 is passed to a settler 228 where a hydrocarbon phase and a water phase are allowed to form. The hydrocarbon layer is removed via line 230 and stored for further use in the process as needed.

The hot oil from the stripper can then be further processed as desired. Typically if the original oil feed contained significant amounts of heavies and there was no separation of the heavies from the oil prior to the hydrotreatment, then the oil would be subjected to a distillation to obtain a product suitable for reformulation with conventional additives to yield the desired weight and quality of lubricating oil.

The use of two stage hydrotreating with interstage flashing results in an integrated process that is much less apt to be adversely affected by variations in the composition of the used oil feedstock. Thus, by using the present invention, one can operate on feeds having wide variations in the amount of high molecular weight nitrogen compounds present without having to make large variations in the operating conditions of the various stages of the process. The invention further results in improved hydrotreating and increased hydrotreating catalyst life.

As further illustration of the present invention, there is provided below typical and preferred ranges of operating conditions for the various components of a process of the type illustrated in FIGURE 1. Obviously, the conditions will vary depending upon the type of feedstock, the type of treating agent, and the results desired.

| Ref. No. | Unit Description | Typical | Approximate Preferred Ranges |
|---|---|---|---|
| 103 | Heater | Temperature 95°C<br>Pressure 17 psia | 60-120°C<br>Atmospheric-250 psia |
| 104 | Treating Agent | Weight ratio agt:oil 0.01:1 | 0.005:1-0.05:1 |
| 106 | Contactor | Temperature 95°C<br>Pressure 17 psia<br>Time 30 minutes | 60-120°C<br>Atmospheric-50 psia<br>10 minutes-2 hours |
| 109 | Contactor | Temperature 125°C<br>Pressure 16 psia<br>Time 30 minutes | 110-140°C<br>5-25 psia<br>10 minutes-2 hours |
| 116 | Contactor | Temperature 160°C<br>Pressure 16 psia<br>Time 30 minutes | 140-200°C<br>5-25 psia<br>10 minutes-2 hours |
| 111 | Phase Separator | Temperature 40°C<br>Pressure atmospheric | 0-80°C<br>Atmospheric-45 psia |
| 164 | Contactor | Temperature 360°C<br>Pressure atmospheric<br>Time 30 minutes | 320-420°C<br>Atmospheric-45 psia<br>5 minutes-2 hours |

| | | | |
|---|---|---|---|
| 171 | Contactor | Temperature 150°C<br>Pressure atmospheric<br>Time 30 minutes | 100-180°C<br>Atmospheric-25 psia<br>10 minutes-2 hours |
| 121 | Filter | Temperature 115°C<br>Pressure differential plate and frame filter 80 psia<br>Continuous rotary drum filter 10 psia | 60-200°C<br>5-100 psia<br>2-14 psia |
| 148 | Furnace | Temperature 760°C<br>Pressure atmospheric | 650-870°C<br>Substantially atmospheric |
| 118 | Filter Aid | Weight ratio aid:oil 0.01:1 | 0.1-0.15:1 |
| 220 | Fractionator | Temperature 647°F<br>Pressure 1.8-2.0 psia | 300-370°C<br>1.7-4.0 psia |
| 210 | Heater | Temperature 370°C<br>Pressure 735 psia | 200-480°C<br>150-300 psia |
| 212 | Contactor | Temperature 370°C<br>Pressure 760 psia | 200-480°C<br>150-3000 psia |
| 214 | Hydrotreater | Temperature 360°C<br>Pressure 780 psia | 200-430°C<br>150-3000 psia |
| 216 | Separator | Temperature 360°C<br>Pressure 685 psia | 290-400°C<br>600-800 psia |
| 220 | Hydrotreater | Temperature 360°C<br>Pressure 665 psia | 200-430°C<br>150-3000 psia |
| 222 | Separator | Temperature 360°C<br>Pressure 620 psia | 290-400°C<br>600-800 psia |
| 224 | Scrubber | Temperature 90°C<br>Pressure 600 psia | 80-95°C<br>500-700 psia |
| 226 | Stripper | Temperature 370°C<br>Pressure 20 psia | 280-395°C<br>Atmospheric-50 psia |

It is noted that the preceding comments are directed to illustrative embodiments of the present invention. Obviously, there are many variations and modifications that can be made without departing from the spirit and scope of the present invention.

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## Claims

1. A process for the production of an essentially ash-free oil stock from lubricating oil containing ash-forming components wherein the lubricating oil is contacted with an aqueous solution of a treating agent which reacts with the ash-forming components to form solids which can be separated from said oil, the major portion of the water is removed from said oil-water mixture, the solids are removed from the oil and at least a portion of the oil is subjected to hydrotreatment to lighten the color of the oil, characterized by carrying out said hydrotreatment in two stages with interstage flashing to remove hydrogen sulfide and ammonia present in the effluent from the first stage before said effluent is passed to the second stage.

2. A process according to claim 1 characterized in that said treating agent comprises an ammonium salt selected from at

least one of ammonium sulfate, ammonium bisulfate, ammonium phosphate, diammonium hydrogen phosphate, and ammonium dihydrogen phosphate.

3. A process according to claim 2 characterized in that said ammonium salt is diammonium hydrogen phosphate.

4. A process according to any of claims 1 to 3 characterized in that the filtered oil prior to being subjected to hydrotreatment is heated to a temperature in the range of 200°C to 480°C and the heated oil is contacted with at least one adsorbent selected from activated carbon, silica gel, clay, bauxite and alumina.

5. A process according to any of claims 1 to 4 characterized in that the oil that is subjected to the hydrotreatment includes components having boiling points higher than 566°C.

6. A process according to claim 5 characterized in that the oil is subjected to the hydrotreatment includes substantially all of the components of the filtered oil having boiling points higher than 566°C.

7. A process according to claim 6 characterized in that the oil that is subjected to the hydrotreatment includes substantially all of the lubricating oil range hydrocarbons that are present in the filtered oil that oil below 566°C.

8. A process according to any of claims 1 to 4 characterized in that the oil that is subjected to the hydrotreatment consists essentially of components having boiling points below 566°C.

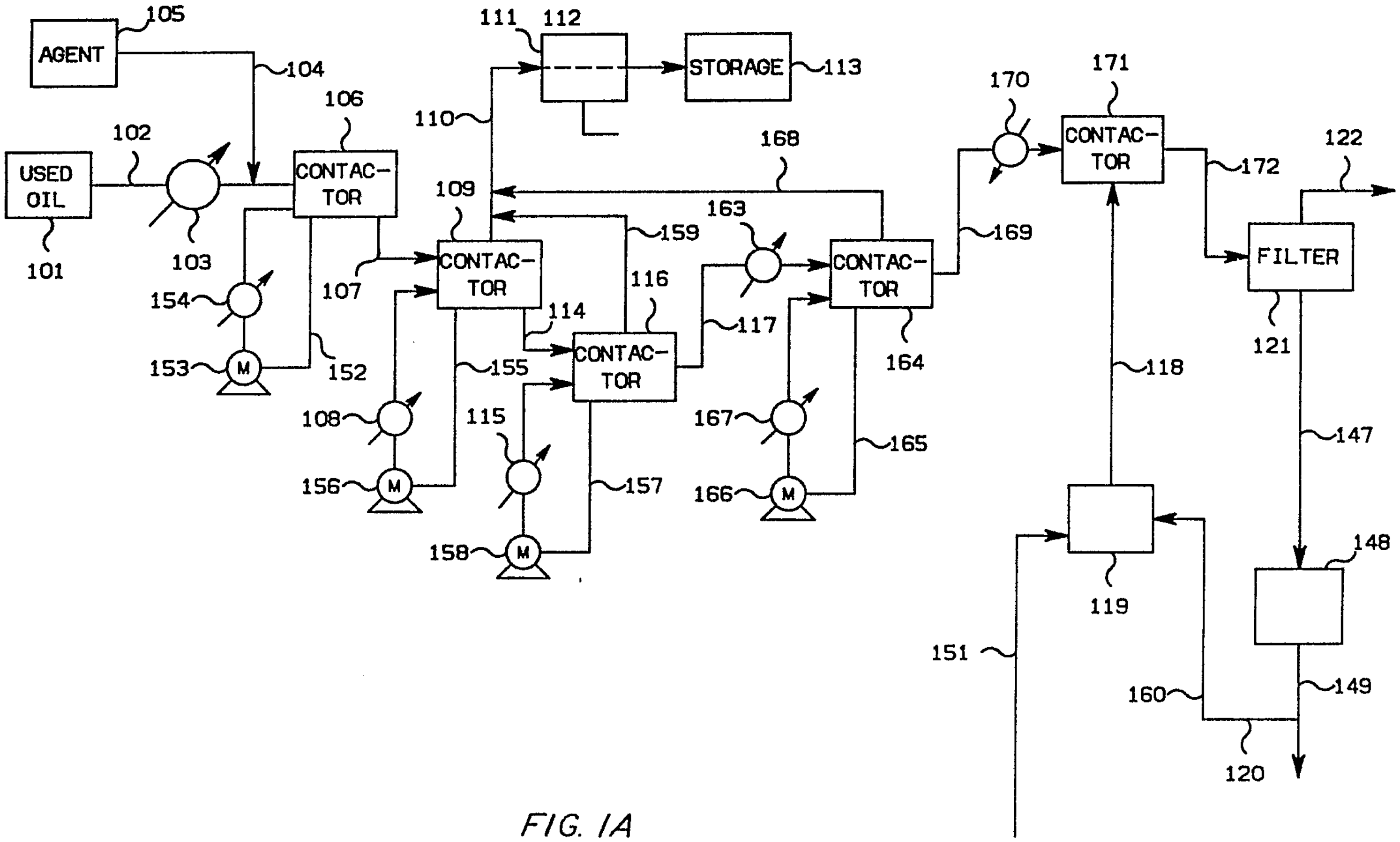
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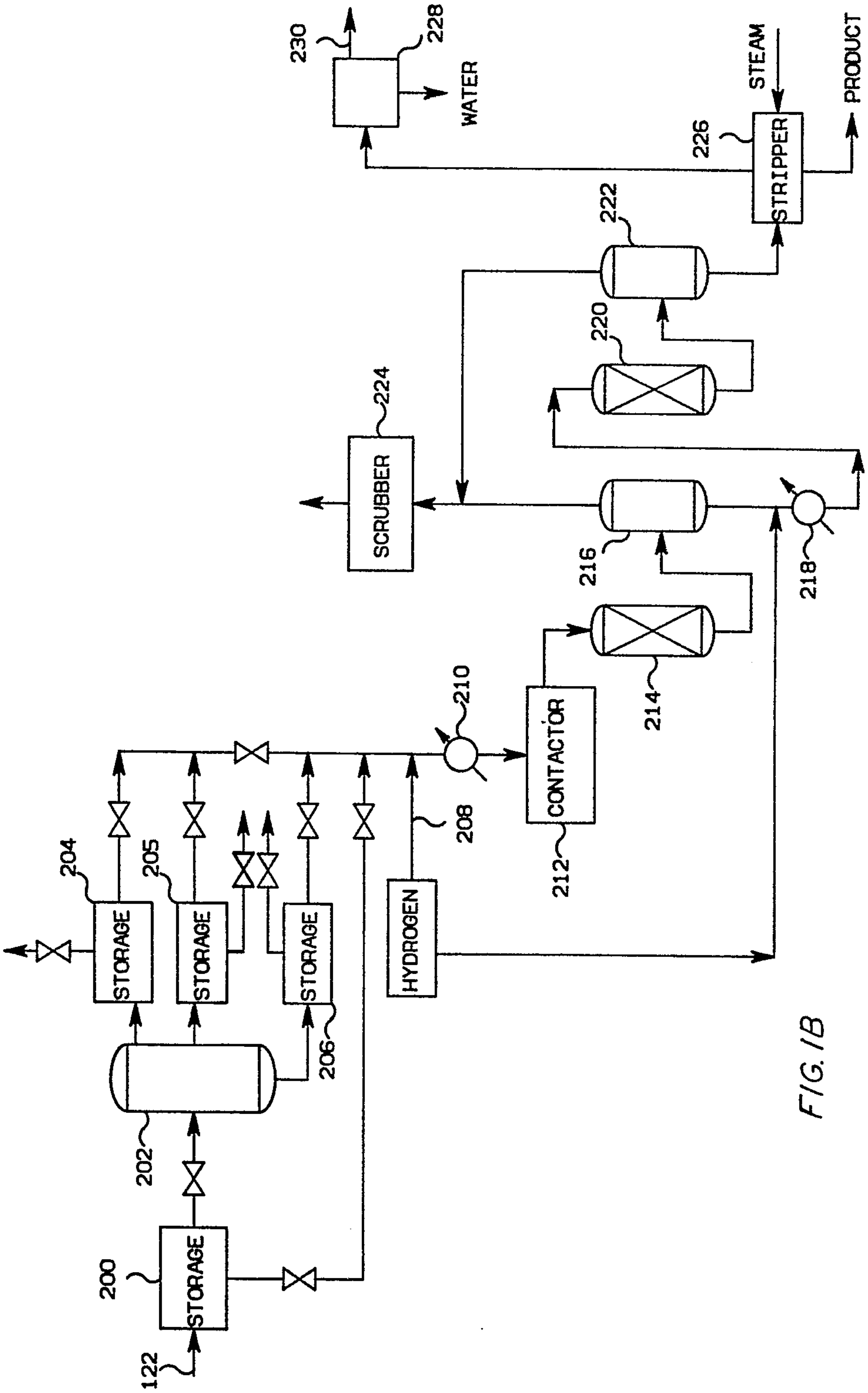
FIG. 1A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