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(54) **Method of producing food grade quality white mineral oil.**

(57) The production of food grade quality white mineral oils from predominantly naphthenic or cycloparaffinic crude distillates heretofore have required acid treating using sulfuric acid followed by neutralization, water wash and possibly finishing step. Herein, however, three stages of hydroprocessing without any solvent extraction or acid treatment prior step are employed to produce the desired food grade quality white mineral oil having a trace of aromatic constituents therewithin. Specific steps are defined in the application in terms of the severity of the hydrogenation in the hydrotreating operation at each respective step; as well as the steps of separating gaseous constituents of the hydroprocessing product.

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Field of the Invention

This invention relates to the method of producing food grade quality white mineral oil. More particularly, this invention relates to a method of producing from a naphthenic distillate as a feedstock, a food grade white mineral oil with only a trace of aromatics therewithin.

Background of the Invention

The prior art is replete with various referrals to methods of treating hydrocarbons. These range from the technology during the depression to modern methods of treating hydrocarbons.

There are almost as many references to employing hydrogen in hydrogenation and hydrotreating aspects. Included are a couple of textbooks published right after World War II including "The Textbook of Organic Chemistry" by E. Wertheim, Second Edition, Blakiston Company, Philadelphia, Pennsylvania, 1947, and "Unit Processes in Organic Synthesis", Groggins, Editor, 3rd Edition, McGraw Hill, New York, New York, 1947. As pointed out in these texts, careful control of hydrogenation can give careful results. This application envisions employing such careful control.

The prior art has seen many ways of trying to achieve a food grade quality of white mineral oil but they have always been expensive and employed acid treatment, neutralization and an adsorption tower or the like for removing of undesired constituents to give the final product.

Specifically, the prior art has failed to provide an economical method of achieving a food grade quality white mineral oil without expensive and labor intensive steps such as acid treating, neutralization and adsorbing of undesired constituents from the product.

Summary of the Invention

Accordingly it is an object of this invention to provide an economical method of achieving a food grade white mineral oil without the labor intensity of processes of the prior art.

It is a specific object of this invention to provide an economical continuous flow process of providing a food grade white mineral oil without the labor intensive processes of the prior art. These and other objects will become apparent when taken with the descriptive matter hereinafter, particularly when taken in conjunction with the appended drawings.

In accordance with one aspect of this invention, there is provided a method of producing a food grade quality of white mineral oil by subjecting a naphthenic or cycloparaffinic feedstock to three

stages of hydroprocessing without any solvent extraction or acid treatment prior to the treatment to give the final desired quality with only a trace of the aromatic hydrocarbons, or aromatic carbons, therewithin.

In another aspect of this invention, there is provided a method of producing a food grade quality in which a naphthenic feedstock is first hydrogenated followed by a step of separating gaseous constituents produced during the hydrogenation reactions, followed by a second stage of hydroprocessing, or hydrogenation, followed by separation of the gaseous constituents produced by this second stage of hydrogenation, followed by a third and less severe hydrotreating step to produce the desired food grade white mineral oil.

Specific reaction conditions for the respective steps are discussed hereinafter.

Brief Description of the Drawings.

Fig. 1 is a schematic flow diagram of a prior art process for producing food grade white mineral oil.

Fig. 2 is a schematic flow diagram of the process of this invention for producing food grade white mineral oil.

Description of Preferred Embodiments

Fig. 1 illustrates a prior art method of preparing a food grade white mineral oil. Therein, a naphthenic distillate is extracted with a solvent such as a phenol or N-methyl pyrrolidine to produce a hydrocarbon oil containing only about 4 to 7 percent aromatic carbons which is subjected to an acid treatment. The bottoms fraction recovered from the acid treatment then emerges as acid sludge whereas from the first step of solvent extraction, a solvent extract containing a high level of aromatic compounds is recovered as a bottom product. After the acid treatment, only a trace of aromatic carbons, or aromatic hydrocarbons are in line 15 leading from the acid treater. A finishing step consisting of an adsorption tower 17 employing clay or a hydrotreater tower 17 using hydrogen is employed to lower the remaining trace quantity of aromatics and produce a satisfactory food grade white mineral oil in the effluent line 19.

In contrast, the approach of this invention involves subjecting a naphthenic distillate containing a concentration of aromatic carbons in the range of 15 - 25 percent by weight to hydrogenation to produce a hydrogenated product in which there is a reduction of about 50 to 70 percent of the aromatic hydrocarbons to yield an aromatic carbon content of 7 - 10 percent by weight. This is shown in Fig. 2, at stage 1, also labeled tower 21. The term "naphthenic distillates" is synonymous with

cycloparaffinic distillates. Normally these distillates contain about 15 - 25 percent by weight of aromatic carbons. These naphthenic distillates come in via line 23, Fig. 2. Hydrogen is fed through line 25 in Fig. 2. Both feed constituents are admixed prior to entering stage 1 where hydrogenation is carried out in the presence of a hydrogenation catalyst containing metal components from the Group VIIIA class, preferably Nickel and from the Group VIA class, preferably Molybdenum at a temperature of 550 degrees Fahrenheit to 750 degrees Fahrenheit, preferably about 650 degrees Fahrenheit - 700 degrees Fahrenheit with a partial pressure of hydrogen in the range of 1200 - 2000 pounds per square inch gauge (PSIG), preferably about 1500 - 1800 pounds per square inch gauge. The hydrogenated product then exits the tower 21 through the line 27.

As the next step, the gaseous constituents of the hydrogenated product in line 27 are separated from liquid constituents and flow out through the overhead line 29. The overhead line 29 carries from the stripper 31 hydrogen sulfide and ammonia, inter alia, as the gaseous products of the hydrogenation reactions carried out in the hydrogenation tower of stage 1, labelled 21. After this process, the aromatic carbon content of the liquid constituents will have been reduced to about 7 - 10 percent aromatic carbons as in the liquid bottom draw from the stripper, line 33. These liquid bottoms containing only about half as much aromatic carbons as the initial feedstock in line 23, or less, are then sent through line 35 to a second hydrogenation tower 37. The liquid bottoms, or hydrogenated product from the first stage, in line 35 is admixed with hydrogen by way of line 39. A second hydrogenation is carried out at rather severe conditions in the presence of a hydrogenation catalyst containing metal components from the Group VIIIA class, preferably Nickel and from the Group VIA class, preferably Molybdenum, with the hydrogen partial pressure in the range of 2500 - 3000 PSIG, preferably 2750 - 3000 PSIG and a temperature in the range of 575 - 750 degrees Fahrenheit, preferably 625 - 700 degrees Fahrenheit. The entire reactor effluent then exits by a line 41 to stripper 43 and again the gaseous constituents of the second hydrogenation stage reaction product are separated from liquid constituents and exit line 45 from the second stripper 47. These gaseous constituents include hydrogen sulfide and ammonia, inter alia. The resulting liquid bottoms from the stripper 43, in line 49, contain only about 1 percent of aromatic carbons and they are sent, as by line 51 to be admixed with hydrogen in line 53 and the hydrogenation as a final step is carried out in stage 3, or the final, less severe hydrogenation of stage 3 in the hydrogenation tower 55.

In the final step, less severe hydrogenation of stage three in the third hydrogenation tower, or hydrotreating tower 55, is carried out in the presence of a hydrogenation catalyst containing a metal component of Group VIIIA class such as platinum, palladium or Nickel, preferably platinum in the form usually utilized in reforming reactions with hydrogen partial pressure in the range of 2000 - 3000 PSIG, preferably 2500 - 3000 PSIG and temperature of only about 375 degrees Fahrenheit to 600 degrees Fahrenheit, preferably 450 degrees Fahrenheit - 550 degrees Fahrenheit.

It is noteworthy that in all these reactions, the use of a relatively high partial pressure of hydrogen and relatively lower temperature facilitates carrying out the hydrogenation to give the desired reaction product in reducing the aromatic constituents of the liquid stream without excessive cracking of the stream to undesired lower boiling range material.

In the illustrated embodiment, the liquid bottoms draw in the line 57 will have only about 0.3 percent or less by weight of aromatic constituents and this trace of aromatics is satisfactory as a food grade white mineral oil. Specifically, the polynuclear aromatics will comprise less than 30 parts per million (PPM) of the final food grade white mineral oil.

In operation, the naphthenic distillate comprising the initial feedstock is fed into and admixed with the hydrogen at the desired partial pressure in the incoming stream and hydrogenation is carried out in stage 1. Similarly, in a stripper, the gaseous constituents are allowed to separate from the liquid constituents such that the gases pass out the overhead stream in line 29 and the bottoms pass out the liquid stream 33 and are then fed through the line 35, Fig. 2, to the second stage, or hydrotreating tower, 37. Again, the admixture of hydrogen at its high partial pressure with the liquid constituents effects a direct reaction at elevated temperatures over suitable catalyst to produce the reduction in the aromatic carbons in line 41 such that after the gaseous constituents are separated and go to the overhead line 45, the liquid bottom draw 49 can be fed, low as it is in aromatic carbons, to the third hydrogenation stage 55. At the entrance to the third stage, it is admixed with high pressure hydrogen at the desired high partial pressure and the hydrogenation reactions carried out in the third hydrotreating tower 55. The result is that the final product comes out the bottom effluent line 57.

From the foregoing it can be seen that the desired food grade quality white mineral oil is produced in the line 57 by a process that differs substantially from the prior art technology for preparing food grade white mineral oil.

Although this invention has been described with a certain degree of particularity, it is under-

stood that the present disclosure is made only by way of example and that combination and arrangement of parts may be resorted to without departing from the spirit and the scope of the invention, reference being had for the latter purpose to the appended claims.

Claims

1. A method of producing food grade quality white mineral oil comprising the steps of subjecting a naphthenic feedstock continuously and progressively to three stages of hydroprocessing without any solvent extraction or acid treatment.
2. A method of producing food grade quality white mineral oil comprising the steps of:
 - a. subjecting a naphthenic feed stock to a first stage of hydroprocessing without any solvent extraction or acid treatment to reduce the aromatic carbon content by about fifty percent;
 - b. separating the gaseous constituents from the hydrogenated liquid product from step a;
 - c. subjecting the product from step a from which the gaseous constituents have been separated to a second hydrogenation step to reduce the aromatic carbon content to about one percent;
 - d. separating the gaseous product constituents of the hydrogenation product of step c; and
 - e. finally, subjecting the liquid from which the gaseous constituents have been separated to a final, less severe hydrotreating step to produce a food grade quality white mineral oil with only a trace of aromatics therewithin.
3. A method of producing food grade quality white mineral oil comprising the steps of:
 - a. subjecting a naphthenic, or cycloparaffinic, distillate containing a concentration of aromatic carbons in the range of 15 - 25 percent by weight to hydrogenation to produce a hydrogenated product in which the aromatic carbons concentration is reduced by about 50 percent;
 - b. separating the gaseous constituents of the hydrogenated product as overhead to prepare a liquid bottoms draw containing only about one-half the concentration of aromatic carbons based on a weight percent concentration;
 - c. subjecting said liquid bottoms draw after said gaseous constituents have been separated therefrom to a second step of hydrogenation to produce a second hydrogenated product in which said concentration of aromatic carbons is reduced to about one percent of the liquid component;
 - d. separating the gaseous constituents of said second hydrogenated product as overhead to produce a second liquid bottoms stream containing only about one percent aromatic carbons; and
 - e. finally subjecting said second liquid bottoms stream to a final, less severe hydrotreating step to produce a food grade quality white mineral oil with only a trace of aromatics therewithin.
4. The method of claim 3 wherein said first hydrogenation is carried out in a first hydrotreating tower with a nicked molybdenum catalyst and said gaseous constituents of said hydrotreated product comprise hydrogen sulfide and ammonia which are separated by sending said hydrogenated product effluent from said first tower to a stripper tower means.
5. The method of claim 3 or claim 4 wherein said first hydrogenation is carried out with a partial pressure of hydrogen in the range of 1500 - 1800 PSIG, pounds per square inch gauge; and a temperature in a range of 650 - 700 degrees Fahrenheit.
6. The method of claim 3, claim 4 or claim 5 wherein said second hydrogenation is carried out in a second hydrotreating tower with a nickel molybdenum catalyst and said gas constituents of said second hydrotreated product comprise hydrogen sulfide and ammonia which are separated by sending said second hydrogenation effluent from said second hydrotreating tower to a second stripper tower means.
7. The method of claim 6 wherein said partial pressures of said hydrogen is in the range of 2750 - 3000 PSIG and wherein said temperature is in the range of 625 degrees Fahrenheit - 700 degrees Fahrenheit.
8. The method of any one of claims 3 to 7 wherein said final, less severe hydrogenation is carried out in a third hydrotreating tower in the presence of a platinum reforming catalyst.
9. The method of claim 8 wherein said final, less severe hydrogenation is carried out with a partial pressure of hydrogen being in the range of 2500 - 3000 PSIG and said temperature being

in the range of 450 degrees Fahrenheit - 550 degrees Fahrenheit.

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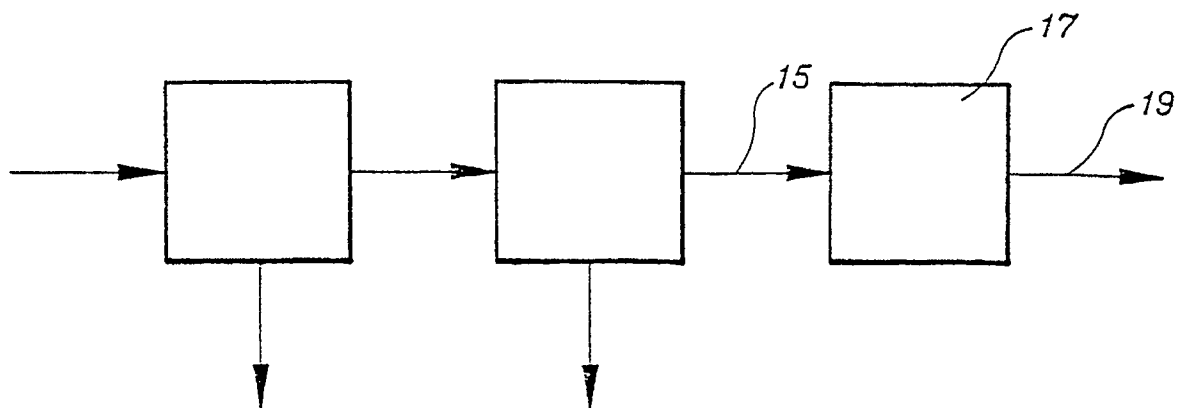


FIG. 1.

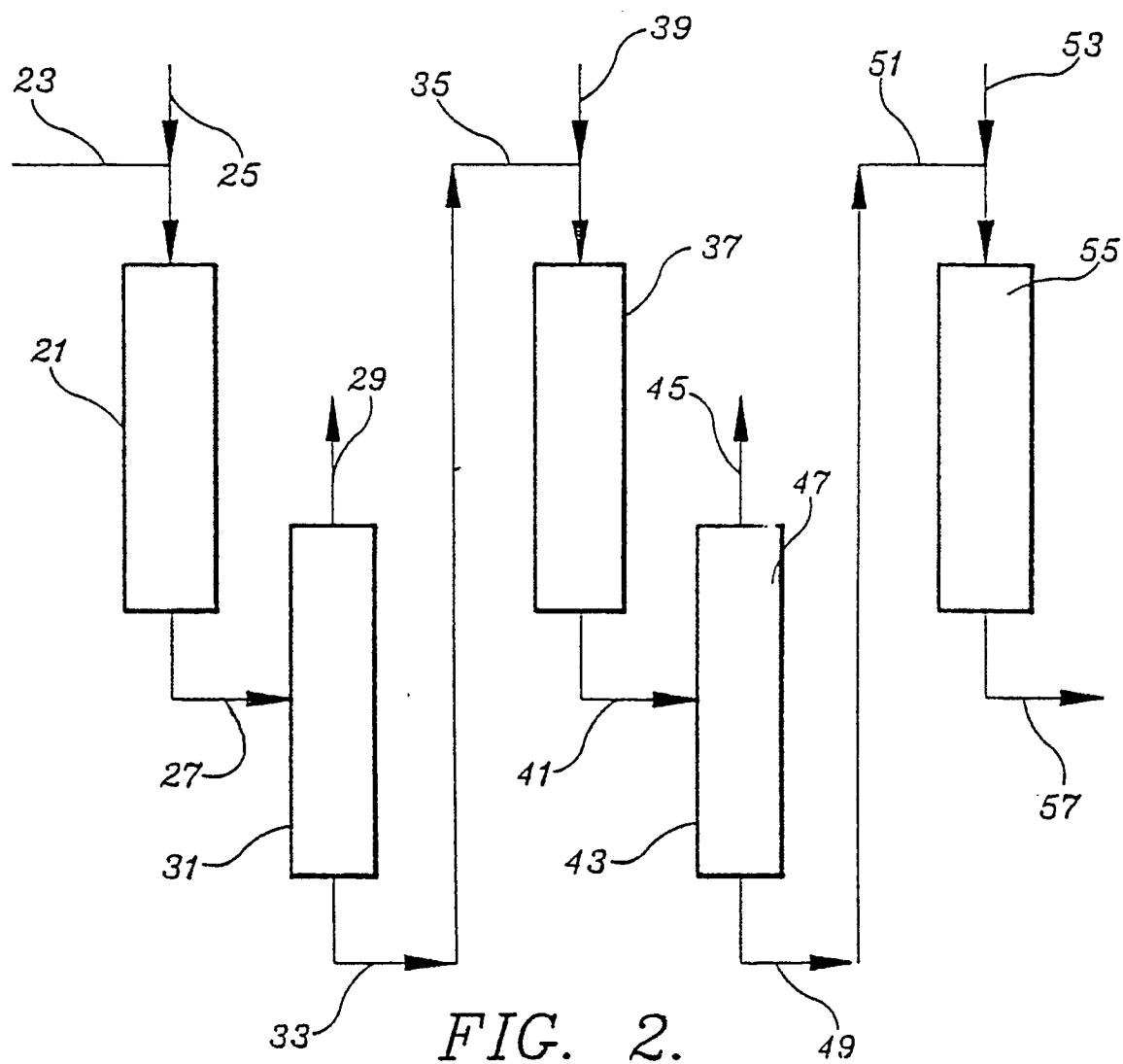


FIG. 2.

EP 91 30 1780

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	US-A-4 325 804 (EVERETT et al.) * Claims;; abstract * - - -	1-9	C 10 G 65/08
A	GB-A-2 001 339 (SHELL) * Claims * - - - - -	1-9	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C 10 G
The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of search 29 May 91	Examiner MICHIELS P.
CATEGORY OF CITED DOCUMENTS X: particularly relevant if taken alone Y: particularly relevant if combined with another document of the same category A: technological background O: non-written disclosure P: intermediate document T: theory or principle underlying the invention		E: earlier patent document, but published on, or after the filing date D: document cited in the application L: document cited for other reasons ----- &: member of the same patent family, corresponding document	