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3,097,158

TREATING HYDROCARBON DISTILLATES

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No Drawing. Filed Sept. 12, 1962, Ser. No. 223,260

6 Claims. (Cl. 203—204)

This invention relates to the treatment of sour hydrocarbon distillates in order to produce a distillate of reduced mercaptan content and of acceptable color.

Sour hydrocarbon distillates are subjected to sweetening in order to reduce the mercaptan content of the distillate. In the sweetening reaction, the mercaptans are oxidized to disulfides. The disulfides are soluble in the hydrocarbon distillate and remain in the distillate. However, when certain kerosenes are subjected to sweetening, the color of the kerosene depreciates. This is objectionable because consumers prefer a good color kerosene and difficulty is encountered in marketing the off-color kerosene. The present invention provides a novel process for improving the color of the kerosene after sweetening.

The exact mechanism which causes discoloration of the hydrocarbon distillate during sweetening has not been elucidated. Apparently some of the constituents of the hydrocarbon distillate undergo reaction to form color bodies. For example, it is believed that polyalkylphenols contained in the hydrocarbon distillate are oxidized to quinones and that the quinones cause discoloration of the hydrocarbon distillate. Accordingly, in order to improve the color of the hydrocarbon distillate after sweetening, it is important to act upon these color bodies but not to adversely affect the other constituents of the other hydrocarbon distillate. It is a particular advantage of the novel process of the present invention that the further treatment of the sweetened kerosene apparently reacts with the color bodies but does not react with the other constituents. For example, the disulfides entrained in the sweetened kerosene should not be converted back to mercaptans. As will be shown in an example, appended to the present specifications, treatment of the sweetened kerosene in the manner herein set forth caused substantially no increase in the mercaptan content of the finally treated kerosenes.

In one embodiment the present invention relates to a combination process for treating sour hydrocarbon distillate to produce a distillate of reduced mercaptan content and of acceptable color, which comprises oxidizing mercaptans contained in said distillate to disulfides and thereafter treating the distillate with a hydrocarbon soluble acyl hydrazide.

In a specific embodiment the present invention relates to a process for treating sour kerosene which comprises reacting said mercaptans contained in said kerosene with air in the presence of a phthalocyanine catalyst and thereafter treating said kerosene with p-toluene sulfonyl hydrazide.

The novel process of the present invention is used for the treatment of any hydrocarbon fraction. While the process may be used for the treatment of normally gaseous hydrocarbons, gasoline, naphtha, etc., it is particularly useful for the treatment of hydrocarbon distillates heavier than gasoline, including kerosene, jet fuel, solvent, stove oil, range oil, burner oil, gas oil, fuel oil, etc. In general, the kerosene will have an initial boiling point of from about 300° to about 450° F. and an end boiling point of from about 475° to about 550° F. Solvents and stove oil, for example, usually have initial boiling points within the range of from about 350° to about 500° F. and end boiling points of from about 525° to about 600° F.

Any suitable sweetening process may be employed. One process comprises the conventional doctor sweet-

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ening process in which the hydrocarbon distillate is treated with doctor solution (sodium plumbite) and sulfur to oxidize the mercaptans to disulfides. Another conventional process is the copper treating process in which the mercaptans are reacted with copper chloride, either as a slurry or as a fixed bed, to oxidize the mercaptans to disulfides. Other sweetening processes include the Hypochlorite process, and various modifications of the processes set forth above.

Another process for oxidizing mercaptans to disulfides utilizes a phthalocyanine catalyst. Any suitable phthalocyanine catalyst may be used and preferably comprises a metal phthalocyanine. Particularly preferred metal phthalocyanines include cobalt phthalocyanine and vanadium phthalocyanine. Other metal phthalocyanines include iron phthalocyanine, copper phthalocyanine, nickel phthalocyanine, chromium phthalocyanine, etc. The metal phthalocyanine, in general, is not readily soluble in aqueous solvents and, therefore, when used in an aqueous alkaline solution or for ease of compositing with a solid carrier, a derivative of the phthalocyanine is preferred. A particularly preferred derivative is the sulfonated derivative. Thus, an especially preferred phthalocyanine catalyst is cobalt phthalocyanine sulfonate. Such a catalyst comprises cobalt phthalocyanine disulfonate and also contains cobalt phthalocyanine monosulfonate. Another preferred catalyst comprises vanadium phthalocyanine sulfonate. These compounds may be obtained from any source or prepared in any suitable manner as, for example, by reacting cobalt or vanadium phthalocyanine with 25–50% fuming sulfuric acid. While the sulfonic acid derivatives are preferred, it is understood that other suitable derivatives may be employed. Other derivatives include particularly the carboxylated derivative which may be prepared, for example, by the action of trichloroacetic acid on the metal phthalocyanine or by the action of phosgene and aluminum chloride. In the latter reaction the acid chloride is formed and may be converted to the desired carboxylated derivative by conventional hydrolysis.

Treatment of the hydrocarbon distillate in the presence of the phthalocyanine catalyst preferably is effected in the presence of an alkaline reagent. Any suitable alkaline reagent may be employed. A preferred reagent comprises an aqueous solution of an alkali metal hydroxide such as sodium hydroxide (caustic), potassium hydroxide, etc. Other alkaline solutions include aqueous solutions of lithium hydroxide, rubidium hydroxide, cesium hydroxide, etc. although, in general, these hydroxides are more expensive and, therefore, generally are not preferred for commercial use. Preferred alkaline solutions are aqueous solutions of from about 1% to about 50% and more preferably 5% to 25% by weight concentration of sodium hydroxide or potassium hydroxide. While water is the preferred solvent, it is understood that other suitable solvents may be used including, for example, an aqueous solution of alcohol, ketone, etc.

When using the phthalocyanine catalyst, air, oxygen or other suitable oxidizing agent is introduced into the reaction zone. In some cases, the hydrocarbon distillate may contain entrained air in a sufficient amount to effect the desired oxidation, but usually it is desirable to introduce extraneous air in order to be sure that sufficient air is present for the desired purpose.

Treating of the hydrocarbon distillate with the phthalocyanine catalyst is effected at any suitable temperature, which may range from ambient to 210° F. when operating at atmospheric pressure, or up to about 400° F. or more when operating at superatmospheric pressure. In general, it is preferred to utilize a slightly elevated temperature which may range from about 100° F. to about 175° F. Atmospheric pressure or superatmospheric

pressure, which may range up to 1000 pounds or more, may be used.

Treatment of the hydrocarbon distillate with the phthalocyanine catalyst is effected in any suitable manner and may be either batch or continuous type of operation. Regardless of which method is used, the phthalocyanine catalyst is employed either as a solution or as a fixed bed. When the catalyst is employed in solution, the amount of catalyst may range from 5 to 1000 parts per million or more and preferably from about 20 to about 500 parts per million by weight of the alkaline reagent solution. In one embodiment the catalyst previously is prepared as a solution in a suitable solvent including ammoniated water, aqueous sodium hydroxide, etc., and then is introduced in this manner to the oxidation zone. In another embodiment the catalyst is added as such to the oxidation zone, to become dissolved in the alkaline reagent solution therein.

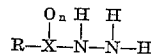
When the catalyst is employed as a fixed bed in the oxidation zone, the catalyst is prepared as a composite with a solid support. Any suitable support may be employed and preferably comprises activated charcoal, coke or other suitable forms of carbon. In some cases the support may comprise silica, alumina, magnesia, etc. or mixtures thereof. The solid catalyst is prepared in any suitable manner. In one method, preformed particles of the solid support are soaked in a solution containing the catalyst, after which excess solution is drained off and the catalyst is used as such or is subjected to a drying treatment, mild heating, blowing with air, hydrogen, nitrogen, etc., or successive treatments using two or more of these treatments prior to use. In other methods of preparing the solid composite, a solution of the phthalocyanine catalyst may be sprayed or poured over the particles of the solid support, or such particles may be dipped, suspended, immersed or otherwise contacted with the catalyst solution. The concentration of phthalocyanine catalyst in the composite may range from 0.1% to 10% by weight or more of the composite.

In a batch type operation, the sour hydrocarbon distillate, alkaline reagent solution and catalyst are disposed in a reaction zone, and air is bubbled therethrough until the desired oxidation is completed. In a continuous type operation, the sour hydrocarbon distillate, alkaline reagent solution and catalyst, when the latter is employed in dissolved form, are supplied to the oxidation zone, preferably at a lower portion thereof. It is understood that the catalyst and alkaline reagent solution may be introduced to the reaction zone either separately or in admixture and either commingled with or separate from the sour hydrocarbon distillate. In a fixed bed continuous process, the catalyst is disposed as a fixed bed in a reaction zone, and the sour hydrocarbon distillate, air and alkaline solution, when desired, are passed into the reaction zone, in upward or downward flow, and either together or separately.

Regardless of the particular operation employed, the products are separated to recover treated hydrocarbon distillate of reduced mercaptan content and to separate alkaline reagent solution for reuse in the process. When the soluble catalyst is employed, the catalyst is recovered in admixture with the alkaline reagent solution and is recycled therewith for further use in the process. When desired, additional quantities of phthalocyanine catalyst may be added continuously or intermittently during the treatment of the sour hydrocarbon distillate.

As hereinbefore set forth, the hydrocarbon distillate, following the treatment with phthalocyanine catalyst, in many cases, will be of poor color. As will be shown in an example appended to the present specifications, such treatment of kerosene having an initial Saybolt color of 30 resulted in a treated kerosene having a Saybolt color of about 0. In accordance with the present invention, the kerosene now is treated with a hydrocarbon soluble acyl hydrazide.

Any suitable hydrocarbon soluble acyl hydrazide is used in accordance with the present invention. The acyl hydrazide is illustrated by the following general formula:



where R is a hydrocarbon radical, n is an integer of from 1 to 2, and X is selected from the group consisting of sulfur and carbon. R in the above general formula preferably is selected from aliphatic, aryl, aliphatic aryl, aryl aliphatic and cycloaliphatic groups. The acyl hydrazide is hydrocarbon soluble and, therefore, R contains at least 4 and preferably at least 6 and up to 30 or more carbon atoms. Illustrative examples of different R groups will be set forth hereinafter.

Where X in the above general formula is sulfur, a particularly preferred acyl hydrazide is p-toluene sulfonyl hydrazide (also named p-toluene sulfonyl hydrazine). p-Toluene sulfonyl hydrazide is available commercially or it may be prepared in any suitable manner. One method of preparing p-toluene sulfonyl hydrazide is by the reaction of toluene with chlorosulfonic acid to form p-toluene sulfonyl chloride and then reacting the p-toluene sulfonyl chloride with hydrazine.

Other aromatic sulfonyl hydrazides include benzene sulfonyl hydrazide, xylene sulfonyl hydrazide, ethylbenzene sulfonyl hydrazide, diethylbenzene sulfonyl hydrazide, propylbenzene sulfonyl hydrazide, dipropylbenzene sulfonyl hydrazide, butylbenzene sulfonyl hydrazide, dibutylbenzene sulfonyl hydrazide, pentylbenzene sulfonyl hydrazide, diethylbenzene sulfonyl hydrazide, hexylbenzene sulfonyl hydrazide, dihexylbenzene sulfonyl hydrazide, heptylbenzene sulfonyl hydrazide, dodecylbenzene sulfonyl hydrazide, octylbenzene sulfonyl hydrazide, dioctylbenzene sulfonyl hydrazide, nonylbenzene sulfonyl hydrazide, dinonylbenzene sulfonyl hydrazide, decylbenzene sulfonyl hydrazide, didecylbenzene sulfonyl hydrazide, undecylbenzene sulfonyl hydrazide, tridecylbenzene sulfonyl hydrazide, tetradecylbenzene sulfonyl hydrazide, pentadecylbenzene sulfonyl hydrazide, hexadecylbenzene sulfonyl hydrazide, heptadecylbenzene sulfonyl hydrazide, octadecylbenzene sulfonyl hydrazide, nonadecylbenzene sulfonyl hydrazide, eicosylbenzene sulfonyl hydrazide, etc., and correspondingly substituted naphthalene sulfonyl hydrazides, anthracene sulfonyl hydrazides, etc. The alkaryl sulfonyl hydrazides contain alkyl groups which preferably have from 1 to about 20 carbon atoms and include the specific compounds hereinbefore set forth. The alkyl group preferably is in a position para to the sulfonyl group.

Illustrative examples of hydrocarbon sodium aralkyl sulfonyl hydrazides include benzyl sulfonyl hydrazide, phenylethyl sulfonyl hydrazide, phenylpropyl sulfonyl hydrazide, phenylbutyl sulfonyl hydrazide, phenylpentyl sulfonyl hydrazide, phenylhexyl sulfonyl hydrazide, etc. Cycloalkyl sulfonyl hydrazides include cyclopentyl sulfonyl hydrazide, cyclohexyl sulfonyl hydrazide, etc. and corresponding alkylcycloalkyl sulfonyl hydrazides.

Where X in the above general formula is sulfur and R is an aliphatic radical, illustrative compounds include butyl sulfonyl hydrazide, amyl sulfonyl hydrazides, hexyl sulfonyl hydrazide, heptyl sulfonyl hydrazide, octyl sulfonyl hydrazide, nonyl sulfonyl hydrazide, decyl sulfonyl hydrazide, undecyl sulfonyl hydrazide, dodecyl sulfonyl hydrazide, tridecyl sulfonyl hydrazide, tetradecyl sulfonyl hydrazide, pentadecyl sulfonyl hydrazide, hexadecyl sulfonyl hydrazide, etc. as illustrative of the saturated derivatives, and butenyl sulfonyl hydrazide, pentenyl sulfonyl hydrazide, hexenyl sulfonyl hydrazide, heptenyl sulfonyl hydrazide, octenyl sulfonyl hydrazide, nonenyl sulfonyl hydrazide, decenyl sulfonyl hydrazide, undecenyl sulfonyl hydrazide, dodecenyl sulfonyl hydrazide, tridecenyl sulfonyl hydrazide, tetradecenyl sulfonyl hydrazide, pentadecenyl sulfonyl hydrazide, hexadecenyl sulfonyl hydrazide, heptadecenyl sulfonyl hydrazide, oc-

tadecenyl sulfonyl hydrazide, nonadecenyl sulfonyl hydrazide, eicosenyl sulfonyl hydrazide, etc. as illustrative of the unsaturated derivatives.

Where X in the above general formula is carbon, illustrative acyl hydrazides include butyryl hydrazide, valeryl hydrazide, caproyl hydrazide, caprylyl hydrazide, capryl hydrazide, lauryl hydrazide, myristyl hydrazide, palmityl hydrazide, stearyl hydrazide, arachidyl hydrazide, behenyl hydrazide, lignoceryl hydrazide, cerotyl hydrazide, etc. as illustrative of those derived from saturated fatty acids, and decylenyl hydrazide, stillingyl hydrazide, dodecylinyl hydrazide, palmiloleyl hydrazide, oleyl hydrazide, ricinoleyl hydrazide, petrocelyl hydrazide, vaccenyl hydrazide, linoleyl hydrazide, eleostearyl hydrazide, licanyl hydrazide, parinaryl hydrazide, gadoleyl hydrazide, arachidonyl hydrazide, cetoleyl hydrazide, erucyl hydrazide, selacholeyl hydrazide, etc. as illustrative of those derived from unsaturated fatty acids. The fatty acid hydrazides are prepared in any suitable manner as, for example, by the reaction of a fatty acid ester with hydrazine. Thus, valeryl hydrazide is prepared by the reaction of methyl valerate or ethyl valerate with hydrazine. In another method, the acyl hydrazide is prepared by the reaction of the aliphatic acid chloride with hydrazine.

Illustrative examples of other acid hydrazides include benzoyl hydrazide, toluyl hydrazide, salicyoyl hydrazide, anisoyl hydrazide, etc., naphthoyl hydrazide, cyclopentyl formyl hydrazide, cyclopentyl acetyl hydrazide, cyclopentyl propionyl hydrazide, cyclopentyl butyryl hydrazide, etc., cyclohexyl formyl hydrazide, cyclohexyl acetyl hydrazide, cyclohexyl propionyl hydrazide, cyclohexyl butyryl hydrazide, etc.

From the above description, it will be seen that a number of different acyl hydrazides may be used in accordance with the present invention. However, it is understood that all of the acyl hydrazides are not necessarily of equivalent activity in improving the color of the hydrocarbon distillate.

Treatment of the hydrocarbon distillate with the acyl hydrazide is effected in any suitable manner. In a preferred embodiment, the treatment is effected in the presence of an alkaline reagent which conveniently is an aqueous solution of an alkali metal hydroxide. The alkali metal hydroxide is selected from those hereinbefore specifically set forth in connection with the oxidation of the hydrocarbon distillate in the presence of the phthalocyanine catalyst. In another embodiment the alkaline reagent may comprise ammonium hydroxide, alkylamines, etc.

In one embodiment treatment of the hydrocarbon distillate with the acyl hydrazide is effected in a batch type operation. In this method the hydrocarbon distillate, alkali metal hydroxide solution and acyl hydrazide are supplied to a reaction vessel equipped with mixing blades or other suitable stirring mechanism to provide intimate mixing of the reactants. In a continuous type operation the hydrocarbon distillate is passed, either countercurrently or concurrently, into contact with the acyl hydrazide and alkali metal hydroxide, when employed, in a suitable vessel. The vessel preferably is provided with suitable contacting means including side to side pans, bubble decks, bubble trays, perforated plates, etc. and/or a fixed bed of inert packing material. Following the contacting, the reaction mixture is processed to recover the sweetened hydrocarbon distillate of improved color. When an aqueous solution of alkaline reagent is employed, the reaction mixture is allowed to separate into a hydrocarbon phase and an aqueous phase in either the zone of contacting or in a different zone, and each phase is separately withdrawn. Following separation and removal of the hydrocarbon distillate, the hydrocarbon distillate preferably is water washed in order to remove alkaline solution and other water soluble materials from the hydrocarbon distillate.

The treatment of the hydrocarbon distillate with the

acyl hydrazide preferably is effected at a mild temperature which may range from ambient and generally will not exceed about 200° F. The pressure will vary with the particular type of operation and may range from atmospheric to 1000 pounds or more, generally being in the range from atmospheric to about 200 pounds per square inch.

For economic reasons it is preferred to use the acyl hydrazide in as small a concentration as satisfactory for the purpose. Because it is believed that the acyl hydrazide decomposes during use, provision should be made in a continuous type process for the addition of acyl hydrazide as required, said addition being intermittently or continuously. The concentration of acyl hydrazide generally will be within the range of from about 0.0001% to about 1% by weight of the hydrocarbon distillate.

While the combination process of the present invention is particularly applicable to the treatment of sour hydrocarbon distillates, it is understood that it may be used for the treatment of mercaptan-containing fractions from other sources as, for example, alcohols. In still another application, the novel features of the present invention are employed for the oxidation of mercaptans synthetically prepared or recovered as a special fraction containing the mercaptan as a substantial or major portion thereof. In such cases, the mercaptan is oxidized to the corresponding disulfide and color depreciation is minimized.

The following examples are introduced to illustrate further the novelty and utility of the present invention but not with the intention of unduly limiting the same.

Example I

A commercial kerosene having an initial boiling point of 370° F., an end boiling point of 520° F., a mercaptan content of 0.239% by weight and a Saybolt color of 30 was treated at 122° F. with air and an equal volume of an aqueous 10° Baumé sodium hydroxide solution containing 250 parts per million of cobalt phthalocyanine disulfonate catalyst. The treatment was effected in a batch type operation for 60 minutes.

The treated kerosene was separately recovered and now had a mercaptan sulfur content of 0.0004% by weight, but the Saybolt color of the kerosene had depreciated to about 0.

500 g. of the kerosene treated in the above manner were mixed with 50 g. of 10% sodium hydroxide solution and 0.1 g. of p-toluene sulfonyl hydrazide and stirred for one hour at an average temperature of about 158° F. Following the reaction, the mixture was allowed to settle into an upper kerosene phase and a lower aqueous caustic phase. The kerosene was separately removed and was washed three times with 200 mls. of water. The kerosene now had a Saybolt color of 14 and a mercaptan content of 0.0005% by weight.

From the above data, it will be seen that the treatment of the kerosene with the acid hydrazide improved the Saybolt color from 0 to 14 and had no substantial effect on the mercaptan content. As hereinbefore set forth, it is important that this treatment does not convert the disulfides contained in the kerosene back to mercaptans.

Example II

An aromatic solvent having an initial boiling point of 360° F. and an end boiling point of 580° F. is treated with air in the presence of vanadium phthalocyanine sulfonate catalyst at 100° F. and 50 pounds per square inch pressure in a batch type operation. The sweetened but discolored solvent oil then is treated at 120° F. with stearyl hydrazine in a batch type operation. The stearyl hydrazide is used in a concentration of 0.2% by weight of the solvent oil. The treating is effected in the presence of 20° Baumé potassium hydroxide solution in a concentration of 5% by weight of the solvent oil. After the treating is completed, the solvent is separated as an upper layer from a lower aqueous potassium hydroxide layer.

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The solvent then is washed with water to remove any entrained potassium hydroxide and other water-soluble materials. The finally treated solvent is reduced in mercaptan content and is of acceptable color.

Example III

A sour West Texas kerosene having a mercaptan sulfur content of 0.15% by weight is subjected to sweetening at 110° F. and 75 pounds per square inch pressure in contact with air and cobalt phthalocyanine sulfonate catalyst. The kerosene, in admixture with an aqueous 5° Baumé caustic solution and air, is passed upwardly through a zone containing the phthalocyanine catalyst as a fixed bed in a reaction zone, and the effluent products are passed into a settling zone where excess air is vented. The kerosene separates from the sodium hydroxide solution and is separately withdrawn from the process. The kerosene now has a mercaptan sulfur content of about 0.002% by weight but is off-color. The kerosene then is treated with benzyl sulfonyl hydrazide, used in a concentration of 0.05% by weight of the kerosene, at a temperature of 140° F. in the presence of 5° Baumé sodium hydroxide solution. The kerosene is washed to remove entrained sodium hydroxide and other water-soluble materials. The finally treated kerosene is reduced in mercaptans and is of acceptable color.

Example IV

Sour jet fuel is sweetened in a conventional doctor treating process in which the jet fuel is mixed with fresh sodium plumbite solution at ambient temperature, after which free sulfur is added and the mixing is continued. The reaction mixture then is allowed to settle and the doctor solution is separated from the treated jet fuel. The jet fuel, now reduced in mercaptans, is mixed with valeryl hydrazide and 5° Baumé potassium hydroxide solution. The mixture is stirred at 135° F. for 45 minutes and then is allowed to settle into an upper jet fuel phase and a lower aqueous phase. Each phase is separately withdrawn and the jet fuel then is water washed to remove entrained caustic and other water-soluble materials. The jet fuel now is of reduced mercaptan content and of acceptable color.

I claim as my invention:

1. A combination process for treating a sour hydro-

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carbon fraction to produce a product of reduced mercaptan content and of acceptable color, which comprises oxidizing mercaptans contained in said fraction to disulfides and thereafter treating said fraction with a hydrocarbon soluble acyl hydrazide.

2. A combination process for treating sour hydrocarbon distillate to produce a distillate of reduced mercaptan content and of acceptable color, which comprises oxidizing mercaptans contained in said distillate to disulfides and thereafter treating the distillate with a hydrocarbon soluble aromatic sulfonyl hydrazide.

3. A combination process for treating sour hydrocarbon distillate to produce a distillate of reduced mercaptan content and of acceptable color, which comprises oxidizing mercaptans contained in said distillate to disulfides and thereafter treating the distillate with a hydrocarbon soluble aliphatic sulfonyl hydrazide.

4. A combination process for treating sour hydrocarbon distillate to produce a distillate of reduced mercaptan content and of acceptable color, which comprises reacting mercaptans contained in said distillate with an oxidizing agent in the presence of a phthalocyanine catalyst to form disulfides and thereafter treating the distillate with a hydrocarbon soluble acyl hydrazide.

5. A combination process for treating sour kerosene to produce a kerosene of reduced mercaptan content and of acceptable color, which comprises reacting mercaptans contained in said kerosene with air in the presence of cobalt phthalocyanine sulfonate catalyst and alkaline solution to oxidize said mercaptans to disulfides, separating treated kerosene, thereafter further treating the kerosene with p-toluene sulfonyl hydrazide and alkaline solution, and water washing the thus treated kerosene.

6. A combination process for treating sour kerosene to produce a kerosene of reduced mercaptan content and of acceptable color, which comprises reacting mercaptans contained in said kerosene with air in the presence of vanadium phthalocyanine sulfonate catalyst and alkaline solution to oxidize said mercaptans to disulfides, separating treated kerosene, thereafter further treating the kerosene with p-toluene sulfonyl hydrazide and alkaline solution, and water washing the thus treated kerosene.

No references cited.