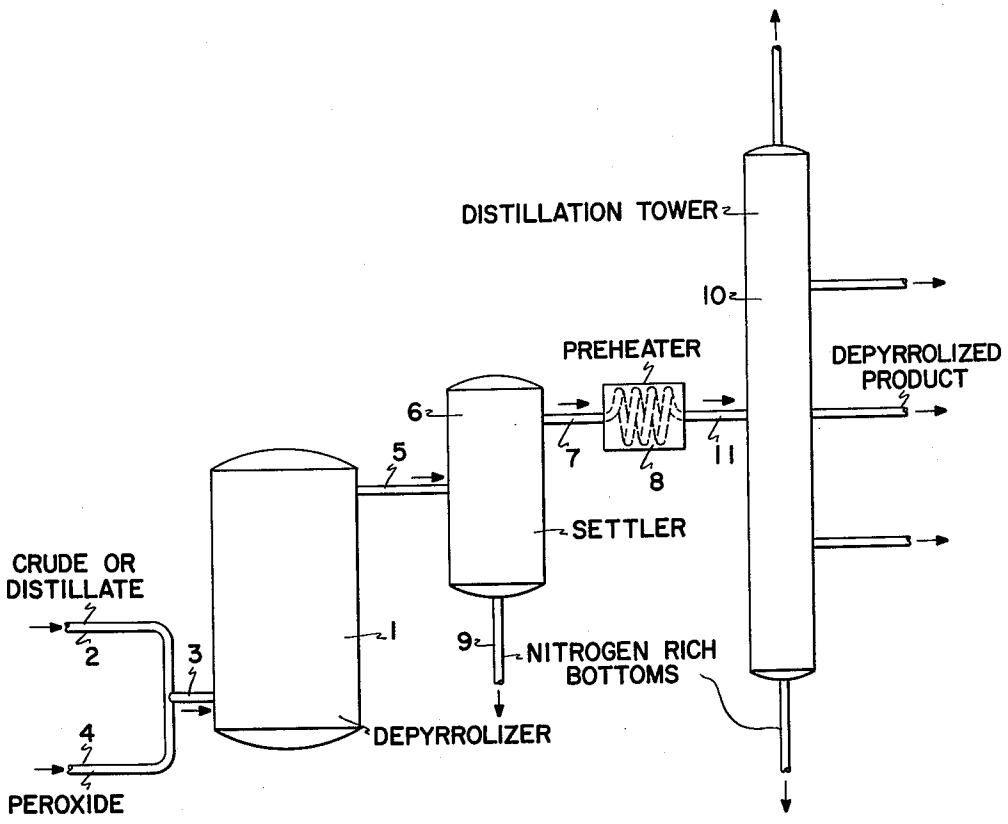
Jan. 9, 1962 A. A. OSWALD ET AL 3,016,349

DENITROGENATION OF PETROLEUM

Filed Aug. 10, 1959



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United States Patent Office 3,016,349 Patented Jan. 9, 1962

3,016,349

DENITROGENATION OF PETROLEUM

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Filed Aug. 10, 1959, Ser. No. 832,734
8 Claims. (Cl. 208—254)

The present invention relates to a new and improved method of refining petroleum crudes and petroleum fractions. More specifically, this invention teaches the removal of nitrogen from said material by treatment with organic peroxides, sulfides and sulfoxides.

The presence of pyrrole-type nitrogen compounds in various petroleum fractions causes color loss and organic sediment formation upon storage for various lengths of time prior to actual use or consumption. These soluble gums and insoluble black organic sediments interfere with the satisfactory performance of these petroleum fractions when they are employed, for example, as middle distillate fuels. Filters and fine orifices, such as burner nozzles and fuel injectors, become plugged rapidly.

In recent years the problem has become more acute. Because of the increasing demand for middle distillate fuels, petroleum refiners have been forced to include higher boiling fractions and catalytically cracked gas oil in heating oils. The addition of these components aggravates the sediment formation.

A further problem of stability has been encountered with jet fuels needed for high performance military aircraft. Jet fuels are often used as a heat exchange medium for cooling the engine lubricating oil. This requires the exposure of the fuel to temperatures in the order of 500° F. for short periods of time. Sediment formation is accelerated at these high temperatures, filters are rapidly plugged, and frequent and costly maintenance of the fuel system is required.

It is well known that alkylated pyrroles and indoles, mainly of the dialkylated type are constituents of petroleum fractions. See Sauer et al., Industrial and Engineering Chemistry, Vol. 44, p. 2606 (1952). It is also well known that these constituents are the main color and sludge formers in many fuels. See Dinneen et al., Industrial and Engineering Chemistry, Vol. 43, p. 1604 (1951); Chenicek et al., Industrial and Engineering Chemistry, Vol. 43, p. 935 (1951). In addition pyrrolic nitrogen compounds act as catalyst poisons during further processing.

In the past, treating methods such as sodafining, acid treating and hydrofining and the incorporation of a variety of additives have been used to control sediment formation. Though these approaches have met with some success, they are often unsatisfactory and erratic.

In accordance with this invention, it has been found that the addition of organic peroxides at temperatures between 50° to 250° F. and the addition of organic sulfides and sulfoxides at temperatures between 60° to 350° F. to petroleum products results in the conversion of nitrogen compounds of the pyrrole type to higher boiling compounds. On subsequent distillation the major proportion of these contaminants remains in the bottoms and a stabilized overhead product is obtained.

To further illustrate the invention, reference is made to the accompanying FIGURE, which shows a preferred process for the removal of pyrrole type compounds from a petroleum crude or distillate. A petroleum crude or distillate is fed to a depyrrolizer **1** through lines **2** and **3**. The distillate may be a narrow cut or may include the whole crude boiling range. Where the feed is crude, water, sediment and salt are usually removed before the addition of the peroxide. Peroxide is injected into the feed stream through line 4. The feed and the pyrrole is heated in depyrrolizer to a temperature from about 50° to 250° F. After the pyrroles react with the peroxide to form sediment and/or high boiling compound, the product is removed through line **5** and passed into settler **6**. The sediment-free overflow from the settler **6** passes through line **7** to preheater **8**. The nitrogen-rich bottoms are removed from the bottom of settler **6** through line **9**. The sediment-free overflow then passes from the preheater **8** to distillation tower **10** through line **11**. From this tower either a depyrrolized distillate of wide boiling range or depyrrolized distillate fractions are obtained ready for use or further process. If the peroxide added to the feed is not wholly consumed in the depyrrolizer, the product from the settler **6** can be washed with aqueous caustic before going to the preheater **8**.

A wide variety of organic peroxides may be used to denitrogenate petroleum hydrocarbons in accordance with the invention. Acyl peroxides, peracids, alkyl and aralkyl hydroperoxides, dialkyl peroxides are examples of the materials that may be utilized. By way of illustration, specific compounds include benzoyl peroxide, 4-chlorobenzoyl peroxide, 2,4-dichlorobenzoyl peroxide, peracetic acid, t-butyl hydroperoxide, cumene hydroperoxide and bis-t-butyl peroxide.

The preferred ranges on the amount of peroxide added are dependent on the concentration of the pyrrolic nitrogen compounds in the oil to be depyrrolized. About 0.1–5 moles of peroxide per mole pyrrole in the oil can be used but it is preferred to use peroxide quantities between 0.5 and 3 moles of peroxide per mole pyrrole. The use of greater than molar quantity of peroxide is sometimes necessary because of side reactions with other fuel components.

Though the above illustration shows the removal of the concentrated nitrogen phase (sediment) from the stabilized petroleum product by distillation, other methods of separation may be employed. For example, the hydrocarbon insoluble, solid products of peroxide-pyrrole reactions can be removed by filtration from the treated petroleum fraction. Any excess of hydroperoxide reagent can be converted to sodium salt by aqueous caustic and extracted as water phase subsequent to filtration. Filtration can be also followed by extraction with a highly polar solvent such as ethylene glycol or phenol.

To further illustrate the advantages of the instant invention the following examples are given. In the first several examples controlled amounts of pyrrole were added to liquid hydrocarbons to simulate the compositions of petroleum fractions.

EXAMPLE 1

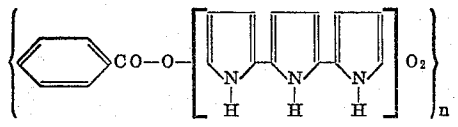
Precipitation of pyrrole nitrogen from benzene solution with benzoyl peroxide

To a benzene solution (50 ml.) containing 3.35 grams (0.05 mole) of pyrrole, 12.1 grams (0.05 mole) of benzoyl peroxide were added. The mixture was stirred for half an hour with a magnetic stirrer, and left standing for 3 days at room temperature. A subsequent filtation recovered a heavy black sediment. This sediment was washed with benzene and naphtha (50 ml. each) and then extracted twice with 500 ml. boiling water for 20 minutes. The remaining insoluble residue weighed 1.5 grams after drying.

An elemental analysis of this residue revealed the following:

	Found
Carbon	64.82
Hydrogen	3.79
Nitrogen	12.1

Such a composition of matter may have the structure of a "pyrrole black" for which the following formula is proposed:



For the proposed composition, $(C_{19}H_{12}O4N_3)n$, the calculated values are:

	Found
Carbon	65.54
Hydrogen	4.01
Nitrogen	12.01

The action of other acyl peroxides (4-chlorobenzoyl peroxide, 2,4-dichlorobenzoyl peroxide) on benzene solutions containing pyrrole was similar to that of benzoyl peroxide.

EXAMPLE 2

Precipitation of pyrrole nitrogen with benzoyl peroxide in the presence of t-dodecyl mercaptan

A solution of 12.1 grams (0.05 mole) of benzoyl peroxide in 50 ml. benzene was added to a mixture of 3.35 grams (0.05 mole) of pyrrole and 3.7 grams (0.017 mole) of t-dodecyl mercaptan. The mixture which became immediately black was treated in the manner described in Example 1. About 2.3 grams of a black powder was obtained, which contained 13.4% nitrogen and 0.28% sulfur.

EXAMPLE 3

Formation of a residue of a high nitrogen content by a reaction between cumene hydroperoxide and pyrrole in benzene solution

To 25 ml. benzene solution of 13.4 grams (0.2 mole) of pyrrole, 30.6 grams (0.2 mole) of cumene hydroperoxide was added and the resulting mixture was refluxed for 2½ hours. Benzene was removed from the resulting orange solution by distillation at 10 mm. pressure. On distilling the residue 8 grams of a yellow liquid containing 3.8% N were obtained between 84° to 86° C. at 2 mm. The residue was a purple solid weighing 4 grams. An analysis revealed that this residue contained 13.8% N.

EXAMPLE 4

The precipitation of pyrrole nitrogen from benzene solution by cumene hydroperoxide

A benzene solution (333 ml.) containing 2.2 grams (0.33 mole) of pyrrole and 15.2 grams (0.1 mole) of butyl hydroperoxide was kept in an open 500 ml. Erlenmeyer flask for 2 weeks at 110° F. At the end of this period, the reaction mixture was filtered and the precipitate formed is shown as milligrams/100 ml. in the following table compared with data for the blanks with only one of the reagents.

Added Compound	Sediment Mg/100 Ml.	N percent
Pyrrole	Nil	
Cumene Hydroperoxide	Nil	
Pyrrole and Cumene Hydroperoxide	487	13.4

EXAMPLE 5

The precipitation of 2,5-dimethylpyrrole by hydroperoxides

To 100 ml. cetane solution containing 2.8 grams (0.03 mole) of 2,5-dimethylpyrrole, equimolar (0.03 mole) quantity of hydroperoxide was added. The mixture was stored in an open 250 ml. Erlenmeyer flask at 110° F. for 24 hours. At the end of this period the insoluble

sediment was determined. The results are shown in the following:

Added Hydroperoxide in Addition to 2,5-Dimethylpyrrole	Sediment Mg/100 Ml.	N percent
Nil	40	8.6
t-Butyl Hydroperoxide	580	9.9
Cumene Hydroperoxide	1,390	10.1

EXAMPLE 6

Removal of nitrogen by peroxide treatment from catalytically cracked untreated gas oil

(a) *Control.*—One liter of an untreated catalytically cracked gas oil was heated up at a rate of 40° C. per hour to 90° C. in an open 2 liter round bottom flask on a steam bath. Then the oil was kept at the same temperature for 6 hours. After this time, the oil was distilled and nitrogen and sulfur concentrations were determined in the 20 to 90% overhead distillate and in the 10% residue.

(b) *t-Butyl hydroperoxide treatment.*—To one liter of the oil used above 13.5 grams (0.15 mole) of butyl hydroperoxide were added. Then the oil underwent the same treatment as the control. The treated oil was washed free of butyl hydroperoxide by 0.1 N aqueous $NaHSO_3$ and distilled in the same manner as the control.

(c) *Diacetyl peroxide treatment.*—To one liter of the above oil 17.7 grams (0.15 mole) of diacetyl peroxide were added. Then the oil was heated up at a rate of 40° C. per hour to 90° C. and kept there for 2 hours. After that period, it was worked up as under (*b*).

The results of these experiments are shown in the following table:

TABLE I

No.	Added Peroxide	Analysis of Distillate Overhead (90%)		Analysis of Residue Bottoms (10%)	
		S, percent	N, percent	S, percent	N, percent
(*a*)	Nil	0.91	0.0050	1.29	0.096
(*b*)	t-Butyl Hydroperoxide	0.90	0.0033	1.27	0.140
(*c*)	Cumene Hydroperoxide	0.98	0.0031	1.22	0.151

EXAMPLE 7

Denitrogenation of catalytically cracked gas oil

0.5 wt. percent of 2,4-dichlorobenzoyl peroxide was added to catalytically cracked gas oil and the solution kept at 200° F. for two hours. Product was distilled to 666° F. final boiling point. 95.3% yield of the product was realized. 50% of nitrogen was left in distillation bottoms. The results are shown below:

TABLE II

	Nitrogen, Wt. Percent	
	No Peroxide Treat	Peroxide Treat
Catalytically Cracked Gas Oil	0.030	
Distillation Overhead, 666° F., FBP	0.028	0.015
Distillation Bottoms (666° F.+)	0.058	0.29
Yield of the Distillate at 666° F., FBP	96.8	95.3

EXAMPLE 8

Two grams (0.03 mole) of pyrrole were dissolved in 100 ml. of benzene. To the solution 9.3 grams (0.03 mole) of (2-hydroperoxy-2-phenyl)-propyl 2-naphthyl sulfide, the hydroperoxide prepared in Example 2 of copending application U.S. Serial No. 777,617, was added and the solution was placed into an oven at 110° F.

Following the addition of the hydroperoxide, the color of the solution changed to brown in 15 minutes. After sixteen hours at 100° F. the following observations were made on the reaction mixture. Control solutions containing benzene and pyrrole and the hydroperoxide in the same amounts and the same concentrations as they appear in the above-described solution were subjected to the same conditions and the corresponding observations made thereon.

TABLE III

Components in Benzene Solution (each in 0.3 m/1cc.)	Observations After 16 hours at 110° F.	
	Color (Tag-Robinson)	Sediment (mg./100 ml.)
Hydroperoxide + Pyrrole	0	582
Hydroperoxide	9	Nil
Pyrrole	25+	Nil

The sediment was produced by the reaction of the hydroperoxide with the pyrrole. This is shown by the results of an analysis made of the sediment which revealed it to contain 7.4% nitrogen and 7.5% sulfur. The solution containing the hydroperoxide alone did not contain any sediment but on further standing precipitated colorless crystals which upon analysis showed them to be without any peroxide character. The solution containing pyrrole alone gave no evidence of precipitation after several hours of standing.

An alternative method for removing nitrogen compounds is by the forming of the peroxide in situ from added reactive hydrocarbons. This can be done by oxidizing suitable added hydrocarbons in the petroleum fraction containing the nitrogen compounds. The addition of aliphatic hydrocarbons containing activating groups is preferable because they are easier to peroxidize. Such activated hydrocarbons are, for example, cumene, styrene, indene and octadecene. Oxidation can be accomplished by using molecular oxygen (air) or active peroxides. Ultraviolet light, peroxides, etc., may be employed as oxidation catalysts.

The following example is illustrative of this modification of the invention.

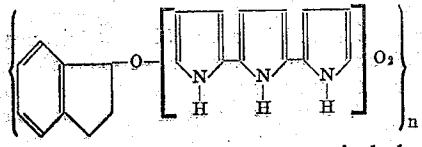
EXAMPLE 9

Formation of a residue of high nitrogen by reacting indene peroxide formed in situ with pyrrole

Oxygen was bubbled into a mixture of 23.2 grams (0.2 mole) of indene and 20.1 grams (0.3 mole) of pyrrole on a steam bath for 2½ hours. After this time, the unreacted compounds were removed by heating the mixture at 2 mm. pressure up to 160° C. A yellow brown residue weighing 1.7 grams remained. The analytical values obtained for this residue are compared below with the values calculated for an assumed indene peroxide-pyrrole reaction product.

	Values Found for the Residue	Values Calculated for $C_{21}H_{20}O_3N_3$ [1]
Carbon	70.76	69.60
Hydrogen	5.77	5.56
Oxygen	13.67 (by difference)	13.24
Nitrogen	9.8	11.60

[1] Assumed structure for $C_{21}H_{20}O_3N_3$:



The previous example illustrates a particularly advantageous feature of this invention, that is, if olefins are present, as is usually the case, in the nitrogen contaminated petroleum fraction it may be unnecessary to add hydroperoxides. These hydroperoxides may be formed in situ by contacting with air or oxygen.

It has been found that the hydrocarbon peroxide reacts much more readily with the pyrroles in the presence of catalytic quantities of metal ions. Among the metal ions, cobalt, copper, iron, manganese and cerium are very effective. Other ions of suitable redox potential with changing valence may also be utilized. Such ions, when in their higher valence forms, react with hydroperoxides to form peroxide radicals. The increased reaction rate may be attributed to the generation of these peroxide radicals and the subsequent reaction of the pyrroles. The following experiments show the effect of metal ions on the reaction between hydroperoxides and pyrroles.

EXAMPLE 10

Two moles per liter of pyrrole and t-butyl hydroperoxide were added to benzene. To part of the sample 0.001 mole per liter of copper oleate was added. The peroxide number, determined according to the ferrous sulfate method (see Wagner et al., Industrial and Engineering Chemistry, Anal. Ed., Vol. 19, p. 976 (1947)), was recorded after 1 hour and 3 days.

The following table shows the data obtained:

	Copper Oleate, m/1.	Peroxide Number after—	
		1 hour	3 days
t-Butyl Hydroperoxide	0	----------	3,410
	0.001	3,367	3,130
Pyrrole and t-Butyl Hydroperoxide	0	2,700	3,780
	0.001	299	0

For purposes of comparison, 2 moles per liter of t-butyl hydroperoxide were added to a benzene solution in the absence of any pyrrole. The above data clearly show that the amount of unreacted peroxide is considerably less when the copper oleate has been added to the solution. This, of course, indicates that the action proceeded with greater rapidity.

EXAMPLE 11

Pyrrole and various derivatives thereof were reacted with t-butyl hydroperoxide in benzene. The general procedure used was to add about 0.1 gram of pyrrole or its derivative to two samples of benzene, each of 5 ml. To one of the samples about 0.05 gram of cupric chloride was added. To both samples 0.4 ml. of t-butyl hydroperoxide was added. The samples were allowed to stand for one hour at room temperature. The following table shows the appearance of the samples.

TABLE IV

Reactant with t-butyl Hydroperoxide	Reaction in the Absence of $CuCl_2$	Reaction in the Presence of $CuCl_2$
Pyrrole	No visible change after 1 hour.	Immediate exothermic reaction occurs and a dark brown precipitate is formed.
2,5-Dimethyl-pyrrole	Solution turns yellow in an hour.	Solution turns dark amber rapidly and a brown precipitate is formed in about 5 min.
2,4-Dimethyl-3-ethyl-pyrrole.	Yellow coloration in an hour.	Solution turns dark brown rapidly and precipitate is formed in 10 min.
-Phenyl-pyrrole	No visible change after 1 hour.	Medium red coloration of solution in an hour.
Indole	Haze formation but no coloration in an hour.	Amber coloration of solution in an hour.
2,3-Dimethyl-indole	Solution turns dark brown in 5 minutes.	Solution turns dark amber rapidly and a gray precipitate is formed.

This table illustrates that the reaction proceeds more rapidly in the presence of cupric chloride. When each of the solutions containing the cupric chloride was diluted with equal amounts of n-heptane, further amounts of precipitate were observed at the end of one hour.

Another method of removing pyrrole-type nitrogen compounds has been discovered in accordance with this invention. It has been found that pyrroles also react with sulfides and sulfoxides to form black polymeric substances. Though pyrrole-type nitrogen compounds are removed, e.g. indoles, carbazoles, the pyridines are not responsive to such treatment. However, pyridines are not a cause of fuel instability. This is shown in the fact that when 40 p.p.m. of pyridine in a xylene solution was heated for 16 hours at 210° F. no sediment was formed and no coloration occurred. The desirability of removing pyrrolic nitrogen compounds has been discussed above. A wide variety of organic sulfides and sulfoxides can be utilized in accordance with this invention. Dialkyl, alkylaryl, diaryl sulfides and sulfoxides are examples of materials which may be utilized. The substituted derivatives of such compounds may be also used. By way of illustration specific compounds include dimethyl sulfoxide, indanyl p-toluyl sulfoxide, diphenyl sulfide. The hydroxy substituted sulfides and sulfoxides such as 2-phenyl-2-hydroxy-ethyl phenyl sulfoxide, 2-(1-hydroxy)-indanyl p-toluyl sulfoxide and bis-hydroxyethyl sulfide, are particularly suitable for use in this invention.

The following table indicates some of the sulfides and sulfoxides which were used for nitrogen removal in the examples of this invention.

TABLE V

No.		Melting Point (° C.)
I	2-Indanyl p-toluyl sulfide	86–86.5
II	2-Indanyl p-toluyl sulfoxide	107–108
III	2-Indanyl p-toluyl sulfone	134.5–135.5
IV	2-(1-hydroxy)-indanyl p-toluyl sulfoxide	134–135
V	(2-Phenyl-2-hydroxy)-ethyl phenyl sulfoxide.	95–105
VI	(2-Hydroxy)-octadecyl phenyl sulfoxide	61–62

The preparation of such materials is shown in co-pending application Serial No. 777,861.

To further illustrate the advantages of this aspect of the invention, the following examples are given. The sulfur compounds added are referred to by Roman numerals which correspond to the compounds of Table V.

EXAMPLE 12

0.003 mole/liter of sulfur compounds I, II, IV, V and VI was added to xylene containing 0.003 mole/liter of pyrrole. The solutions were stored at 210° F. for 16 hours and then the amount of sediment formed and the percent nitrogen in the sediment were determined. The following table indicates the data obtained.

TABLE VI

Sulfur Compound	Sediment, mg/100 ml.	N, percent
I	9	9.7
II	14.5	7.5
IV	34	5.8
V	8	8.3
VI	5.5	4.0

The above data clearly indicate that an appreciable amount of the nitrogen is removed by the addition of the sulfur compounds.

EXAMPLE 13

The sulfur compounds listed in Table V were dissolved in xylene to give solutions of 0.003 mole/liter concentrations. To samples of such solutions 0.003 mole/liter of freshly distilled pyrrole was added. Then 200 ml. solutions containing the sulfur compounds together with pyrrole were stored in open 500 ml. Erlenmeyer flasks at 110° F. one month. After this period the samples were filtered and the filtrates were distilled from a steam bath at 15 mm. The colors of the samples after the storage, the amount of sediments and the nitrogen concentration in the distillates are shown in Table VII.

TABLE VII

S Compound Added	Pyrrole Added (p.p.m. N)	Color of Sample		Sediment mg./100 ml.	N in Distillate (p.p.m.)	Color of Distillate after Two Months
		Appearance	Tag-Robinson			
None	None	Colorless	>25	Nil		Colorless.
None	40	Yellow tint	24	Nil	7.8	Brownish.
I	40	Green	9	3.2	2.9	Light.
II	40	Dark	0.5	2.6	2.7	Light.
III	40	Yellow	24	Nil		
IV	40	Dark Green	2.5	4.3	1.4	Light.
V	40	Brown	5	1	3.8	Light.
VI	40	Brown	10	0.6		

From Table VII it is apparent that sulfides and sulfoxides convert pyrrole to sediment and residue. As a consequence, the distillation of such samples produces a stable hydrocarbon (xylene) with low nitrogen content.

EXAMPLE 14

Reaction of 2-indanyl p-toluyl sulfoxide and pyrrole in nitrogen atmosphere

0.003 mole/liter of both 2-indanyl p-toluyl sulfoxide and pyrrole and the same compounds alone were dissolved in benzene. The solutions were put into vials purged with nitrogen, sealed and stored at 110° F. After one month storage a sediment with a violet tint appeared in the vial containing the sulfoxide and the pyrrole. The control solutions with the pyrrole or the sulfoxide remained clear and colorless.

EXAMPLE 15

Removal of nitrogen compounds from kerosene

(a) *Bis-methyl sulfoxide and air.*—A crude oil mixture of 80% Officina and 20% Gunipa (Venezuelan) was distilled in an atmospheric still with 15 theoretical plates. A cut obtained between 300° to 600° F. by ASTM distillation was treated in the following manner.

9.2 grams dimethyl sulfoxide and 1 liter of oil were placed into a 1200 ml. three-necked round bottomed flask equipped with a thermometer, water condenser and sintered glass air inductor. Air was introduced into the mixture for 6 hours while it was kept between 87° to 92° C. and stirred by a magnetic stirrer. At the end of this period the mixture was left to stand overnight when 0.3 gram of dark brown oil had separated on the bottom of the vessel. This oil had a nitrogen content of 1% while the original nitrogen content of the oil was 20.2 p.p.m.

(b) *Bis-hydroxyethyl sulfide and air.*—12.2 grams (0.1 mole) of bis-hydroxyethyl sulfide was added to 1 liter of the distillate (300° to 600° F. ASTM) used previously. The heterogeneous mixture was aerated in the same manner as under (*a*) for 6 hours. The bis-hydroxyethyl sulfoxide phase contained 58 p.p.m. nitrogen after the treatment.

After the removal of the sulfoxide phase, the oil was distilled to remove a 300° to 520° F. fraction. In (*a*) and (*b*) this fraction contained a nitrogen content of only 7 p.p.m. and 5.9 p.p.m., respectively.

What is claimed is:

1. A process for the denitrogenation of hydrocarbons containing pyrrole-type nitrogen compounds which comprises contacting 0.1 to 5 moles, per mole of said pyrrole-type nitrogen compounds, of an organic compound selected from the class consisting of peroxides, sulfides and sulfoxides with said hydrocarbons, converting said nitrogen compounds by reaction with said compounds whereby a high boiling fraction of high nitrogen concentration is formed, and separating said fraction from said hydrocarbons, said hydrocarbons being of substantially reduced nitrogen content.

2. The process of claim 1 wherein the said organic compound is a sulfide.

3. The process of claim 1 wherein said organic compound is a sulfoxide.

4. The process of claim 1 wherein the said organic compound is a peroxide.

5. The process of claim 4 wherein the fraction of high nitrogen concentration is formed in the presence of metal ions.

6. The process of claim 5 wherein the metal ions are of the class consisting of cobalt, copper, iron, manganese, and cerium.

7. A process for the denitrogenation of hydrocarbons containing pyrrole-type nitrogen and olefins which comprises oxidizing said olefins to form peroxides in situ, reacting said peroxides with said nitrogen compounds and subsequently separating said fraction from said hydrocarbons, said hydrocarbons being of substantially reduced nitrogen content.

8. The process of claim 7 wherein said olefins are oxidized by the introduction of air into said olefins.

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