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TREATMENT OF PARAFFINS WITH ALUMINUM BROMIDE

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1

This invention relates to a process for the treatment of certain paraffinic materials, for example, a lubricating oil or a wax, to improve certain of its important characteristics. In one of its aspects, the invention relates to the treatment of a refined lubricating oil or a wax to lower its pour point employing a novel process which presents certain features enabling great improvements in quality of the treated oil product. In another of its aspects the invention relates to the provision of a novel process employing certain critical conditions essential to obtain the said improvements in quality or characteristics of a refined lubricating oil or wax. In still another aspect of the invention it relates to the effecting of certain novel changes within a lubricating oil or wax fraction employing a catalyst which is the sole agent now known which can cause effectuation of said changes. Among the characteristics of such a material which can be significantly improved, pour point mentioned above, and viscosity index especially are to be noted.

Among the objects of the invention is to provide a process for lowering the pour point of a paraffinic material such as a waxy lubricating oil while at the same time materially and significantly increasing its viscosity index.

Other objects and advantages are apparent from this disclosure and its appended claims.

The process of the present invention is preeminently applicable to the treatment of a lubricating oil containing refractory wax constituents to convert said constituents in a manner and to extent such that a substantial lowering of its pour point is accomplished. Accordingly the invention will be described making reference to the treatment of high-melting paraffin-containing stocks such as waxy lubricating oil base stocks of undesirably high pour point.

According to the present invention a paraffinic material of undesirably high pour point, such as a waxy lubricating oil base stock, or paraffin wax, is admixed with a substantial quantity of aluminum bromide to form an homogeneous mass which is then allowed to stand or is agitated for a number of hours depending upon the extent of treatment to be effected.

Preferably, before admixing the aluminum bromide with the oil, it is treated with an alumi-

2

num halide, an acid, or other material to remove undesirable constituents such as aromatic compounds and sludge-forming materials.

By effecting the treatment referred to in the immediately preceding paragraph, not only is the process of the invention rendered more effective to reduce the pour point of the oil, but also there can be observed an important increase in its viscosity index. Thus according to the invention, as an important feature thereof, is the combination of the step of treating the oil to remove undesirable constituents of the nature or character of those just described prior to admixing the aluminum bromide therewith.

To enhance the action of the aluminum bromide, a low-boiling saturated hydrocarbon material can be admixed with the aluminum bromide-oil mass. According to the invention, among the low-boiling hydrocarbon materials which are thus employed are the branched-chain paraffins with from four to about twelve carbon atoms, for example isopentane, the naphthenes with from six to about ten carbon atoms, for example, methylcyclopentane, or mixtures thereof. Usually about ten to about one hundred per cent by weight, of the paraffinic material, of low boiling hydrocarbon will be employed to assist the conversion of the waxy constituents of the oil by the aluminum bromide catalyst.

The homogeneous mass of oil, catalyst and low boiling hydrocarbon is allowed to stand or is agitated for a time sufficient to effect the desired extent of conversion. Usually a period of standing or agitation of from about ten hours to about one hundred hours at a temperature in the approximate range of 10° C. to 100° C. will be sufficient to satisfactorily lower the pour point of the oil. It will be understood, however, that the precise period of time will depend upon the temperature of the treatment, the nature of the oil being treated, the quantity of catalyst employed and even upon the exact nature and quantity of the low boiling hydrocarbon in the mass.

The quantity of the aluminum bromide catalyst employed is to be in the range of about ten to about fifty per cent by weight of the oil treated.

The exact nature of the reaction or reactions occurring during the conversion of the waxy constituents of the oil being treated with the aluminum bromide catalyst cannot be stated. However, it appears from a careful study of the data presented that isomerization of the waxy molecules plays a part in the conversion. Although some disproportionation appears to occur resulting in the increased quantity of the low-boiling

hydrocarbons recovered, it is believed equally clear that the lowering of the pour point and the increases in viscosity index which have been accomplished are very likely, if not actually, due to isomerization of the waxy molecules, as stated. The use of aluminum chloride did not result in a lowering of the pour point of any of the paraffinic materials treated.

The following examples illustrate the invention. Parts are by weight. Pour points were determined by ASTM (D97–39) method.

EXAMPLES

Example I

A mixture was prepared of 100 parts of a hexadecane concentrate having a freezing point (pour point) of 17.3° C., 60 parts of 95 per cent methylcyclopentane and 32 parts of anhydrous aluminum bromide. At the end of 40 hours stirring at 60–70° C., a sludge was separated which contained 2.0 parts hydrocarbon and 7.7 parts aluminum bromide. The remainder was washed free of dissolved aluminum bromide and was vacuum distilled. There was obtained 69.6 parts low-boiling hydrocarbons, including all the added methylcyclopentane, and 85.3 parts of a hexadecane concentrate boiling about 120° C. at 3–5 mm. Its freezing point (pour point) was 10.0° C., a reduction of 7.3° C. from the original.

Example II

A mixture was prepared of 100 parts of a hexadecane concentrate having a freezing point (pour point) of 17.1° C., 41.5 parts of isopentane and 33 parts of anhydrous aluminum bromide. At the end of 27 hours stirring at 25–35° C., a sludge was separated which contained 1.3 parts hydrocarbon and 5.0 parts aluminum bromide. The remainder was washed free of dissolved aluminum bromide and was vacuum distilled. There was obtained 61.6 parts low-boiling hydrocarbons, including all the added isopentane, and 76.4 parts of a hexadecane concentrate boiling about 120° C. at 3–5 mm. Its freezing point (pour point) was 14.9° C., a reduction of 2.2° C. from the original.

Example III

A mixture of 100 parts of a paraffin wax which solidified at 51–55° C. (pour point), and 27 parts of 95 per cent methylcyclopentane was stirred for 48 hours near 60° C. and then for 49 hours near 100° C. with 33 parts anhydrous aluminum bromide which was added in three nearly equal portions at 0, 23, and 30.5 hours. A sludge was separated which contained 7.1 parts of hydrocarbon and 17 parts of aluminum bromide. The remainder was washed free of dissolved aluminum bromide and was vacuum distilled. There was obtained 34.6 parts low-boiling hydrocarbons including the added methylcyclopentane and 75.6 parts of residue boiling about 136° C. at 6 mm. It solidified at 40–53° C. (pour point), an average reduction of 7° C. from the original.

Example IV

An SAE 20 lubricating oil from which aromatics and sludge-forming materials had been removed was treated with aluminum bromide in accordance with this invention to lower the pour point. To 55.1 parts by weight of the oil were added 22.6 parts by weight of isopentane and 22.3 parts by weight of aluminum bromide. The mixture was vigorously agitated at 90–100° F. for 48 hours. The oil was then washed with water to remove dissolved aluminum bromide. The washed oil was dried with anhydrous potassium carbonate. The dried oil was topped at 250° F. and 1 mm. pressure to remove light hydrocarbons. Properties of the oil before and after the aluminum bromide treatment are shown in the following tabulation:

	Before $AlBr_3$ Treatment	After $AlBr_3$ Treatment
Pour Point, ° F.	35	15
Viscosity at 100° F., SUS	251.3	200.0
Viscosity at 210° F., SUS	49.84	46.92
Viscosity index	101	104
Yield, wt. percent		79

It is noteworthy that the treatment of the oil with aluminum bromide and isopentane in accordance with this invention reduced the pour point 20 degrees and increased the viscosity index.

Reasonable variation and modification are possible within the scope of the foregoing disclosure and the appended claims to the invention, the essence of which is that, under certain specific conditions, specifically and critically aluminum bromide can be employed to reduce the pour point of a paraffinic material and at the same time to increase its viscosity index.

I claim:

1. The process for lowering the pour point of a paraffinic material which comprises maintaining together said material and a low-boiling saturated hydrocarbon with a substantial proportion of aluminum bromide at a temperature in the approximate range 10° C.–100° C. for a period of time sufficient to cause a lowering of its pour point.

2. The process for lowering the pour point of a paraffinic lubricating oil which comprises maintaining together said material and a low-boiling saturated hydrocarbon with a substantial proportion of aluminum bromide at a temperature in the approximate range 10° C.–100° C. for a period of time sufficient to cause a lowering of its pour point.

3. The process for lowering the pour point of a paraffinic oil which comprises maintaining together said material and a low-boiling saturated hydrocarbon with ten to fifty weight per cent of aluminum bromide at a temperature in the approximate range 10° C.–100° C. for a period of time sufficient to cause a desired lowering of its pour point.

4. The process for treating a wax-containing paraffinic lubricating oil to reduce its pour point which comprises admixing said oil with about ten to about fifty per cent by weight of aluminum bromide, about ten to about one hundred per cent by weight of a low-boiling saturated hydrocarbon and maintaining the admixture at a temperature in the approximate range 10° C.–100° C. for a period of time in the range of from about ten to about one hundred hours and recovering an oil of lowered pour point.

5. The process of claim 1 wherein the paraffinic material is first treated to remove sludge-forming constituents therefrom.

6. The process of claim 1 wherein the paraffinic material is first treated to remove therefrom undesirable sludge-forming materials such as aromatic compounds.

7. The process of claim 1 wherein the paraffinic

material is first treated with an aluminum halide to remove undesirable sludge-forming constituents therefrom.

8. The process for improving the pour point of a waxy lubricating oil which comprises treating said oil with an agent effective to remove aromatics therefrom; admixing the treated oil with 10–50 parts by weight of aluminum bromide, about ten to about one hundred per cent by weight of a low-boiling saturated hydrocarbon and maintaining the admixture at a temperature in the approximate range 10° C. to 100° C. for a period of 10 to 100 hours and recovering therefrom an oil of lowered pour point.

9. The lowering of the pour point of a hexadecane concentrate which comprises admixing therewith a low-boiling saturated hydrocarbon material and a substantial quantity of anhydrous aluminum bromide, as such, and maintaining the admixture until a desirable lowering of the pour point can be observed.

10. The process of claim 9 wherein the low-boiling saturated hydrocarbon material is methylcyclopentane, the temperature is maintained in the range 60°–70° C., and the oil has an initial pour point of about 17.3° C.

11. The process of claim 9 wherein the low-boiling saturated hydrocarbon material is isopentane, the temperature is maintained in the range 25° C.–35° C. and the oil has an initial pour point of about 17.1° C.

12. The lowering of the pour point of a paraffin wax fraction having a pour point within the approximate range of 51° C.–55° C. which comprises admixing therewith a low-boiling saturated hydrocarbon material and a substantial quantity of anhydrous aluminum bromide, as such, and maintaining the admixture until a desirable lowering of the pour point can be observed.

13. The process of claim 12 wherein the low-boiling saturated hydrocarbon material is methylcyclopentane, the temperature is maintained at about 60° C.

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References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
1,601,406	Moody	Sept. 28, 1926
1,811,243	Osmer	June 23, 1931
2,106,010	Levi et al.	Jan. 18, 1938
2,468,746	Greensfelder et al.	May 3, 1949
2,475,358	Moore et al.	July 5, 1949

OTHER REFERENCES

Anhydrous Aluminum Chloride in Organic Chemistry, by C. A. Thomas, Reinhold Publishing Co., New York, 1941, p. 875.

Chemical Constituents of Petroleum, by A. N. Sachanen, Reinhold Publishing Co., New York, 1945, p. 210.