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MINERAL OIL COMPOSITIONS AND PROCESSES OF PREPARING THE SAME

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This invention relates to mineral oil compositions and processes of preparing the same, and it comprises mineral oil compositions, such as lubricating oils and greases, containing small amounts of an acylated and polymerized indene, it further comprises such mineral oil compositions containing small amounts of the product of acylating indene, or one of its polymers, in the presence of a metallic chloride acylating catalyst, hydrolyzing the intermediate metallic chloride complex and recovering the catalyst-free acylated and polymerized indene.

For many uses in the art lubricating oil compositions having low pour points must be used. The pour point of an oil is in part directly related to the amount of paraffin wax in the oil and it is customary to dewax the oil both for the recovery of usable paraffin and for lowering the pour point of the oil. Even after drastic dewaxing operations the final lubricating oil does not always have as low a pour point as the art desires. Paraffin wax itself, however, has lubricating characteristics, and prior workers have sought to keep some of the wax in the oil by so modifying the pour point of the oil that in spite of its wax content the oil can be used for lubricating at low temperatures. This has been accomplished in recent years by adding to the oil substances to which the name "pour point depressors" has been given. Quite a few substances for this purpose have been discovered. Theoretically it is assumed that a pour point depressor inhibits crystallization of the paraffin wax in the oil and thus prevents the oil, when subjected to low temperatures, from forming a kind of sludge or slush of relatively large wax crystals occupying the entire volume of the oil and occluding liquid oil within the network of crystals.

At the present time condensation products between chlorinated paraffins and aromatics are used to a considerable extent as pour point depressors. Products of this nature are much less efficient in their actions than compounds described in this invention. In addition they contribute nothing to the oiliness of the lubricant to which they are added which is markedly different from our products.

In all instances the amount of pour point depressor added is relatively small. The quantities generally average a fraction of a percent based on the quantity of oil.

We have now discovered a class of materials which function particularly well as pour point depressors, and which have the added advantage that they add oiliness to the oil. The substances which we use can be generically defined as acylated indenes wherein the acyl group contains at least twelve carbon atoms. These acylated indenes are new materials in themselves and are the subject matter of the Ralston, Vander Wal and Bauer application Serial No. 228,260 filed September 2, 1938. These compounds are prepared, broadly speaking, by reacting indene, or one of its polymers, with a fatty acid chloride having at least twelve carbon atoms in the presence of an acylating catalyst, such as aluminum chloride. This reaction results in the formation of an intermediate complex containing the catalyst which is thereafter hydrolyzed to free it of catalyst, and the final product recovered. Thus the preparation of these acylated indenes follows closely the acylation of other aromatic hydrocarbons by the Friedel-Crafts synthesis. But during the acylation of the indene the indene polymerizes. Or it is possible that the indene is first acylated and thereafter the acyl derivative polymerizes. In any event, the final product is an acylated and polymerized indene. Or, to put it more definitely, it is a polymerized indene containing acyl groups having twelve or more carbon atoms.

In the preparation of these pour point depressors any fatty acid chloride having twelve or more carbon atoms can be used. These can be either saturated or unsaturated, or they can be fatty acid chlorides made from fatty acids prepared by the oxidation of paraffin wax or other hydrocarbons yielding fatty acids on oxidation. Mixtures of two or more different fatty acid chlorides can be used as the acylating agent. This gives us final products of mixed character and very complex constitution. Indeed the pour point depressor effect of our products may possibly be due to the complex nature of them. As fatty acid chlorides we can use lauryl, myristyl, palmityl, stearyl, oleyl, linoleyl and linolenyl chlorides, these being the more usual ones.

In broad aspects, the fatty acid chloride is added to a suspension of the acylating catalyst, such as aluminum chloride, in an inert solvent, and to this mixture indene dissolved in an inert solvent is added. The proportions of fatty acid chloride to indene can vary over wide limits as we have found by experiment. This probably means that so long as the indene polymer contains at least one acyl group the product has pour point depressor characteristics. But more than one acyl group can be present in the final product.

We shall now give an example of how our pour point depressors are made from fatty acid chlorides of at least twelve carbon atoms and indenes.

Example

Twenty grams of aluminum chloride are suspended in 50 cc. of tetrachloroethane in a three-necked flask equipped with a stirrer, dropping funnel and thermometer. To this is added, rapidly, 30 grams of stearyl chloride, the temperature being held below 25° C. during the addition. 15.7 grams of a 74 percent solution of indene in tetrachloroethane are added over a period of one and one-half hours. The temperature varies between about 25° C. and 30° C. during the addition. The temperature is then kept at about 45° C. to 55° C. for about one hour. The resulting reaction product is then hydrolyzed by admixing with ice and the solvents removed by steam distillation. The product is dissolved in ether, washed well with water and the ether solution dried with sodium sulfate. The ether is then removed under a vacuum.

In precisely the same manner other acylated indenes are prepared as more fully disclosed in the aforesaid application.

The final product is semi-solid and plastic, and resinous in character. It dissolves readily in mineral oils in proportions used for pour point depressing.

We shall now indicate by way of example how our products function when added to lubricating oils.

In the following examples the oils designated as 1, 2 and 3 are three different kinds of lubricating oils in common commercial use. In the following examples where we refer to "indene solution (74%)" we have reference to the solution given in the foregoing example of the characteristic way of making our compounds. The examples also indicate the pour point of the original oil and the pour point of the oil after the addition of our pour point depressors.

Example 1

A compound prepared from 15.7 parts of indene solution (74%) and 30 parts of stearyl chloride is added to the following lubricating oils. 0.5% of the compound is added with the following results:

Oil	Saybolt viscosity at 100° F.	Pour point, °F.	
		Original	Treated
	Seconds		
1	266	+30	−25
2	433	+10	−15
3	366	−5	−15

Example 2

0.5% of a compound prepared from 15.7 parts of indene solution (74%) parts of stearyl chloride is added to the oils with the following results:

Oil	Saybolt viscosity at 100° F.	Pour point, °F.	
		Original	Treated
	Seconds		
1	266	+30	−15
2	433	+10	−15
3	366	−5	−20

Example 3

0.5% of a product prepared by the reaction of 15.7 parts of indene solution (74%) and 30 parts of oleyl chloride is added to the oils with the following results:

Oil	Saybolt viscosity at 100° F.	Pour point, °F.	
		Original	Treated
	Seconds		
1	266	+30	−10
2	433	+10	−15
3	366	−5	−10

Example 4

0.5% of a product prepared by the action of 15.7 parts of indene solution (74%) and 30 parts of linolenyl chloride is added to the oils with the following results:

Oil	Saybolt viscosity at 100° F.	Pour point, °F.	
		Original	Treated
	Seconds		
1	266	+30	−15
2	433	+10	−10
3	360	−5	−10

Thus from the above examples it is apparent that our pour point depressors are markedly effective for the purpose intended, a pour point lowering of as much as 55° F. being obtained. Our pour point depressors are especially effective on oils having rather high pour points, thus indicating the marked ability of our compounds to prevent crystallization of the wax.

Similar results are obtained when other fatty acid chlorides having twelve or more carbon atoms are used. The amount of pour point depressor can vary over wide limits. Noticeable improvement in pour point lowering is obtained when only 0.1% of our products is added. Larger amounts, up to 2% or 3% can also be added but much greater amounts, such as 10% or 15% are not necessary in order to obtain a substantial pour point lowering.

In addition to the property of lowering the pour point of the mineral oils these products also decrease the heat generated during running friction. The oils were tested in an apparatus described by Ralston, Hoffman and Stephens, found in National Petroleum News, dated November 3, 1937. The addition of 0.5% of the product used in Example 1 showed a difference of 24.5° F. in temperature rise after one and one-half hours between an original and a treated oil.

In the appended claims we use the langauge "an indene" to include the indene dimers, trimers or tetramers as well as indene itself since the indene thereof is the essential polymerizing component.

Although we have referred more particularly to the use of our acylated indene materials with lubricating oils it is apparent that they can also be added to lubricating greases where it is desirable to reduce the heat of friction or to prevent the grease from assuming a relatively hard, non-flowing state when subjected to a cold environment. When preparing such greases we can first dissolve the pour point depressor in the lubricating oil and then make a lubricating grease therefrom in the usual way.

Having thus described our invention, what we claim is:

1. A mineral oil containing, as a pour point depressor, an acylated indene, the acyl group having at least twelve carbon atoms.

2. A mineral oil containing, as a pour point depressor, a stearylated indene.

3. A mineral oil containing, as a pour point depressor, an acylated indene polymer wherein the acyl group contains at least twelve carbon atoms.

4. A mineral oil containing, as a pour point depressor, a stearylated indene polymer.

5. A mineral oil containing, as a pour point depressor, the product obtained by reacting indene with a fatty acid chloride having at least twelve carbon atoms in the presence of an acylating catalyst, and recovering a catalyst-free product.

6. A mineral oil containing, as a pour point depressor, the product of reacting an indene with stearyl chloride in the presence of an acylating catalyst and recovering a catalyst-free product.

7. The process of depressing the pour point of a mineral oil which comprises adding to the oil the products resulting from reacting an indene with a fatty acid chloride having at least twelve carbon atoms in the presence of an acylating catalyst and recovering a catalyst-free product.

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