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3,194,814
HIGH MOLECULAR WEIGHT ALKENYL-N-ALLYL SUCCINIMIDE

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This invention relates to oil-soluble nitrogen-containing compositions and to the process of preparing the same. The compositions of this invention are useful as dispersing agents in lubricants, especially lubricants intended for use in the crankcase of internal combustion engines, gears, and power transmitting units.

One of the principal problems associated with present day crankcase lubricants is that posed by the inevitable presence in the lubricant of foreign particles such as dirt, soot, water and decomposition products resulting from breakdown of the lubricating oil. Even if there were none of this latter contaminant present the very nature of the design of the modern internal combustion engine is such that a significant amount of foreign matter will accumulate in the crankcase. Perhaps the most important of these contaminants is water because it seems to be responsible for the deposition of a mayonnaise-like sludge. It appears that if there were no water present the solid components of the mayonnaise-like sludge would circulate with the oil and be removed by the oil filter. It will be readily appreciated that the deposition of the sludge presents a serious problem with respect to the efficient operation of the engine and that it is desirable to prevent such deposition of sludge-like material.

The presence of water and the precursors of sludge in a lubricating oil is dependent largely upon the operating temperature of the oil. If the oil is operated at a high temperature the water, of course, will be eliminated by evaporation about as fast as it accumulates. In the absence of water as stated above the other foreign particles will be removed by the filter. At low oil temperatures, on the other hand, water will accumulate and so consequently will sludge. It is apparent that the environment in which a crankcase lubricant is maintained will determine to a large extent the ultimate performance of that lubricant.

High operating temperatures are characteristic of a lubricant in an engine that is run at relatively constant high speed. Thus, in an engine that is run at 60 miles per hour for a long period of time it is very unlikely that there will be any accumulation of water and it is similarly unlikely that there will be any formation and deposition of sludge, but in ordinary stop-and-go driving, the crankcase lubricant will be alternately hot and cold, an ideal environment for the accumulation of water. In such cases the formation of sludge is a serious problem. This problem has been with the automotive industry for many years and its solution has been approached by the use of known detergents such as metal phenates and sulfonates but without notable success. Although such known detergents are very effective in solving the detergency problems associated with motor oils at high temperatures they have not been particularly effective in solving the problems associated with low temperature operation, or to put it better, those problems which are associated with crankcase lubricants in engines which are operated at alternating high and low temperatures.

It is accordingly a principal object of this invention to provide novel compositions of matter.

It is also an object of this invention to provide compositions which are adapted for use as additives in hydrocarbon oils.

It is also an object of this invention to provide compositions which are effective as detergents in lubricating compositions.

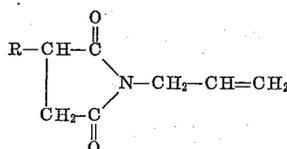
It is another object of this invention to provide a novel process for the preparation of products which are effective as dispersants in lubricant compositions.

It is another object of this invention to provide novel compositions which are effective dispersants in lubricant compositions intended for use in engines operated at alternating high and low temperatures.

It is another object of this invention to provide improved hydrocarbon oil compositions.

It is another object of this invention to provide improved lubricating compositions.

These and other objects are achieved in accordance with this invention by providing a succinimide having the structural formula

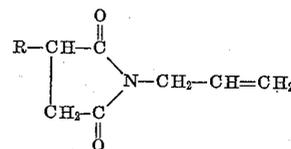
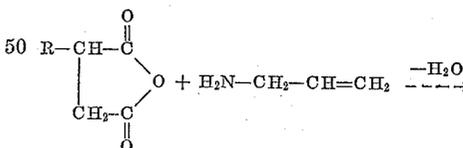


wherein R is a polyisobutene radical having from about 50 to about 200 carbon atoms.

A critical aspect of this invention is that the polyisobutene radical contains at least about 50 carbon atoms. The criticality is predicated upon the discovery that a similar succinimide having a smaller R radical in its molecular structure is not useful for the purposes of this invention.

The sources of the R radical include polyisobutenes having molecular weights within the range from about 700 to about 3000. Such polymers are well known in the art. They can be obtained for instance, by contacting isobutene, preferably in a solvent such as hexane, butane, or naphtha, with a Friedel-Crafts catalyst such as aluminum chloride, boron trifluoride, zinc chloride, or titanium tetrachloride at a temperature between about -50° C. and 25° C.

The polyisobutene-substituted succinimide of this invention is obtained by the reaction of approximately equimolar mixtures of an appropriate polyisobutene-substituted succinic anhydride or acid with allyl amine. The reaction between an anhydride and the diamine is illustrated by the following equation:



The reaction should be carried out at a temperature which is sufficiently high to cause the removal of water from the reaction mixture. Ordinarily the temperature is between about 100° C. and 250° C., preferably between about 150° C. and 200° C. The presence of a solvent during the reaction is often advantageous to facilitate mixing of the reactants and control of the reaction temperature. Solvents useful herein include mineral oil, xylene, diphenyl oxide, and dodecane.

The polyisobutene substituted succinic acids or anhydrides which are contemplated as a reactant in the

above reaction are readily available from the reaction of maleic anhydride with a polyisobutene having the appropriate molecular weight. The reaction involves merely heating the two reactants at a temperature about 100°–200° C. The product from such a reaction is an alkenyl succinic anhydride, the alkenyl group may be hydrogenated to an alkyl group. The anhydride may be hydrolyzed by treatment with water or steam to the corresponding acid.

The following example illustrates the preparation of the succinic imides in this invention: (parts are by weight)

Example 1

A polyisobutene-substituted succinic anhydride having an acid number of 97 and an equivalent weight of 577 is prepared by the reaction of a chlorinated polyisobutene (having an average molecular weight of 1000 and a chlorine content of 4.5%) and maleic anhydride. To a mixture of 57 parts (1 equivalent or 1 mole) of allyl amine and 778 parts of mineral oil there is added 1130 parts (2 equivalents or 1 mole) of the polyisobutene substituted succinic anhydride at 60°–80° C. within a period of 1 hour. The mixture is heated at 150°–155° C. for 5 hours while nitrogen is bubbled through the mixture. The residue is an oil solution containing 40% of the polyisobutene-substituted succinimide of allyl amine. The solution is found to have a nitrogen content of 0.71% (the theoretical nitrogen content of the imide is 0.72%).

The principal utility of the polyisobutene-substituted succinimides of this invention is as detergent additives in lubricating oils. They also are useful in other compositions, especially hydrocarbon compositions such as gasolines, burner fuel oils, cutting oils, transformer oils, hydraulic fluids, etc. The concentration of the succinimides as additives in such compositions may range from about 0.1% to about 10% by weight. For example, lubricating compositions for use in gasoline internal combustion engines may contain from about 0.5% to about 5% of the additive, lubricating compositions for use in gears and diesel engines may contain as much as 10% or even more of the additive, and gasolines and burner fuel oils may contain as little as 0.001% or even less of the additive.

The lubricating oils in which the additive of this invention are useful may be of synthetic, animal, vegetable, or mineral origin. Ordinarily mineral lubricating oils are preferred by reason of their availability, general excellence, and low cost. For certain applications oils belonging to one of the other three groups may be preferred. For instance, synthetic polyester oils such as didodecyl adipate and di-2-ethylhexyl sebacate are often preferred as jet engine lubricants. Normally the oils preferred will be fluid oils ranging in viscosity from about 40 Saybolt Universal seconds at 100° F to about 200 Saybolt Universal seconds at 210° F.

To prepare the final oil compositions the additive of this invention may be added to the oil at the appropriate concentration. Alternatively, a concentrate of the additive may be prepared by dissolving the additive in a limited amount of the oil and the concentrate may then be diluted with additional amounts of the oil to prepare the final oil compositions.

The lubricating compositions in which the additives of this invention are present may contain other additives such as supplemental ashless detergents, metal containing detergents, corrosion inhibitors, rust inhibitors, oxidation inhibitors, load-carrying additives, anti-foam additives, pour point depressants, viscosity index improving agents, additives to improve the frictional characteristics, etc. The concentrations of these additives in the lubricating com-

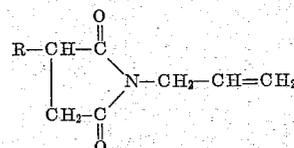
positions may range from about 0.001% to 20% by weight.

A specific example of a lubricating composition useful in the crankcase of an automobile is a SAE 30 mineral lubricating oil containing 1.5% (by weight) of the product of Example 1. Other examples of lubricating compositions include: SAE 90 mineral lubricating oil containing 3% of the product of Example 1 and 1% of zinc dioctylphosphorodithioate; SAE 20 mineral lubricating oil containing 0.5% of the product of Example 2, 1% of barium didodecyl benzene sulfonate, and 2% of 2,6-di-tert-butyl-4-methylphenol; and SAE 30 mineral lubricating oil containing 3% of the product of Example 3, 1.5% of bis(octylphenyl) sulfide and 1% of dibutyl tetrasulfide.

The effectiveness of the succinimides of this invention as additives in lubricating compositions to prevent the formation of harmful deposits is shown by an oxidation-dispersancy test which is useful as a screening test for determining the effectiveness of the additive under light duty service conditions. In this test a 350-cc. sample of a lubricating oil containing the additive is placed in a 2" x 15" borosilicate tube. A 1 3/8" x 5 7/8" SAE 1020 steel panel is immersed in the oil. The sample then is heated at 300° F. for 48 hours while air is bubbled through the oil at the rate of 10 liters per hour. The oxidized sample is cooled to 120° F., homogenized with 0.5% by volume of water, allowed to stand at room temperature for 24 hours and then filtered through two layers of No. 1 Whatman filter paper at 20 mm. mercury pressure. The weight of the precipitate, washed with naphtha and dried, is taken as a measure of the effectiveness of the additive, i.e., the greater the weight of the precipitate the less effective the dispersant additive. By this test a Mid-Continent, conventionally refined petroleum oil having a viscosity of 200 Saybolt Universal seconds at 100° F. and containing 0.001% by weight of iron naphthenate (to promote oxidation) is found to give 900–1000 milligrams of sludge per 100 cc. of the oil sample whereas the same oil containing additionally 1.5% by weight of the polyisobutene-substituted succinimide of allyl amine gives only 255 milligrams of sludge per 100 cc. of the oil sample.

What is claimed is:

1. A succinimide having the structural formula



wherein R is a polyisobutene radical having from about 50 to about 200 carbon atoms.

2. The succinimide of claim 1 wherein R is a polyisobutene radical having about 75 carbon atoms.

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