

United States Patent

Giolito

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[54] **STYRENATED TRIARYL PHOSPHATE
FUNCTIONAL FLUIDS**

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[58] Field of Search252/78, 75, 49.8; 260/966

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[57] **ABSTRACT**

The transmission of pressure in a closed hydraulic system is accomplished by using an effective amount of styrenated phenyl bis(isopropylphenyl) phosphate.

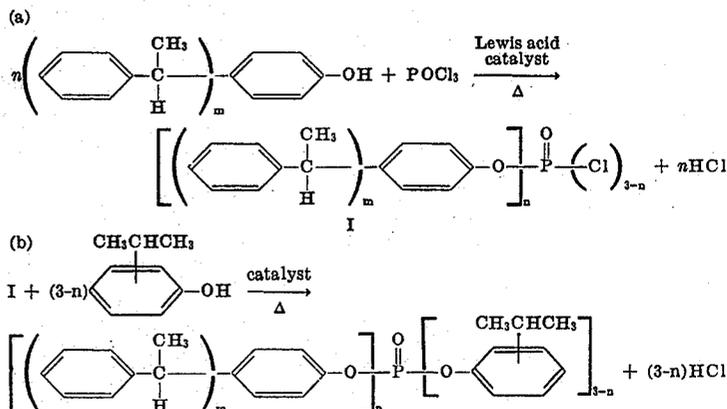
2 Claims, No Drawings

STYRENATED TRIARYL PHOSPHATE FUNCTIONAL FLUIDS

BACKGROUND OF THE INVENTION

Functional fluids suitable for the operation of hydraulic mechanism require a combination of properties which are often unattainable in many presently available materials. The

The compounds of the present invention are prepared by a two step process comprising: (a) reacting phosphorus oxychloride with a styrenated phenol or a styrenated phenol mixture in the presence of a Lewis acid catalyst, and (b) reacting the product of step (a) with an isopropylated phenol or an isopropylated phenol mixture. The process can be shown diagrammatically as follows:



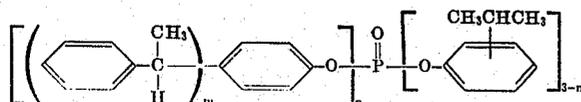
properties which are required for safe and satisfactory low temperature operation include a low pour point which permits a hydraulic fluid to operate at low temperature. Other important properties which are desirable in hydraulic fluids are a high viscosity and a flat viscosity temperature curve, that is, a high viscosity index which allows the fluid to remain operable over a wide range of temperatures. One of the vital properties which is required for hydraulic fluids which are to be utilized in either commercial industry and mining or military use is minimum flammability. Other desirable qualities for such products include a relatively high boiling point, low corrosion characteristics and low oxidation susceptibility.

It is known in the art that the viscosity index of hydraulic fluids normally decrease when they contain an increasing proportion of cyclic structures. (References: Murphy et al., Industrial and Engineering Chemistry, Vol. 42, page 2,415; Bried et al., Industrial and Engineering Chemistry, Vol. 39, page 484 and reference cited therein.) Therefore, highly aromatic compounds are usually poor choices for hydraulic fluids. It is also known that benzyl compounds are normally highly susceptible to free radical attack upon the benzyl moiety by oxygen and other reactive species. Therefore, styrenated compounds should exhibit low oxidative stability and would therefore be considered to be poor choices for hydraulic fluids.

TECHNICAL DISCLOSURE OF THE INVENTION

It has now been discovered that novel styrenated phenyl bis(isopropylphenyl) phosphates are suitable for use as hydraulic fluids having been found, surprisingly, to possess all the above described properties required for the successful utilization of such products. In particular, these phosphates have high viscosities in the range from about 800 to about 1,200 Saybolt Universal Seconds at 100° F. and have relatively high viscosity indices when compared to commercial fluids in this viscosity range. Further, they have pour points below 0° F. and high autoignition temperatures in the range from about 900° to about 1,100° F.

The phosphates employed in the process of the present invention are novel styrenated phenyl bis(isopropylphenyl) phosphates having the structure:



wherein n is a number greater than 0 and less than 1, and m is a number from 1 to 2 inclusive.

wherein n is a number greater than zero but less than 1 and m is a number from 1 to 2 inclusive. Steps (a) and (b) can be carried out sequentially or simultaneously. However, the sequential process is preferred because it allows for better control over the value of n , which value is essential to the hydraulic fluids of the present invention.

The reaction of the present invention is conducted in the presence of a Lewis acid catalyst. The term "Lewis acid catalyst" is meant to designate those inorganic compounds which are strong electron pair acceptors. These compounds are well known in the art and are illustrated by the following: ferric, aluminum, zinc and magnesium chloride, molybdenum pentachloride, stannic tetrachloride and boron trifluoride. The Lewis acid catalyst is employed in amounts from about 0.1 to about 5.0 percent by weight of the entire reaction mixture and preferably in the range from about 0.05 to about 2 percent by weight.

The styrenated phenols employed in making the hydraulic fluids of the present invention are commercially available as mixtures of a major portion of styrenated phenol, and minor portions of phenol and the di-styrenated phenols. They can also be synthesized by reacting styrene with phenol at elevated temperatures in the presence of various catalysts. The isopropylated phenols are also commercially available, normally as mixtures consisting of a major portion of ortho, meta and para isopropyl phenols; together with smaller portions of the 2,6-diisopropyl phenol, 2,4-diisopropyl phenol, triisopropyl phenol and phenol.

The ratio of the isopropylphenyl moiety to the styrenated phenyl moiety must be maintained within certain limits in order to obtain hydraulic fluids having the desired properties mentioned above. This ratio should be greater than 2 to 1, that is, n is a number less than 1, in order to get fluids having viscosities in the desired range of from about 900 to about 1,200 S.U.S. Therefore, less than 1 mole of the styrenated phenol or a styrenated phenol mixture should normally be employed per mole of phosphorus oxyhalide in step (a) and at least 2 moles of isopropyl phenol or of an isopropyl phenol mixture should be employed in step (b).

The reaction temperature should be maintained in the range from about 20° to about 300° C. and preferably in the range from about 100° to about 200° C. The reaction times are normally in the range from about 4 to about 20 hours; the shorter times being obtained when the higher temperatures are employed.

The styrenated phenyl bis(isopropylphenyl) phosphate esters are characterized by having desirable and unusually high viscosities and viscosity indices. This is surprising since

they have a large proportion of cyclic structures per molecule which would normally be expected to lower their viscosity indices as was stated above. Thus, one would expect the triaryl phosphate functional fluids having a base stock of the tricresyl and trixylyl phosphates to have higher viscosity indices than the styrenated phenyl bis(isopropylphenyl) phosphates of the present invention. However, the compounds of the present invention exhibit viscosity indices which are much higher than those of the triaryl phosphates.

The styrenated triaryl phosphates used in the process of the present invention can be used individually or blended with anticorrosion agents, defoamers and various load bearing additives all of which are well described in the literature. For economic reasons, the functional fluids of this invention can be mixed with less costly petroleum oils and/or polychloroaromatic compounds, particularly polychlorobenzenes and polychlorobiphenyls.

The compounds used in the process of the present invention are deployed in a closed hydraulic system such as compressors, hydraulic lifts, deck edge elevators, brake systems, basic oxygen furnaces, die casting equipment, leveling devices or servo control units in such a manner that when pressure is applied to the phosphate at a specific point within the confines of the hydraulic system, the pressure will be transmitted to every other point along the hydraulic system by the phosphate.

EXAMPLE 1

A 2 liter Pyrex 3 neck reactor was fitted with a thermometer, condenser, dropping funnel and a magnetic stirrer. The condenser was connected by means of a Y tube to a nitrogen bleed system and an aqueous HCl trap. The reactor was charged with 163.4 grams of phosphorus oxychloride and 195 grams of a commercially available styrenated phenol which is composed of 21.4 percent phenol, 57.8 percent monostyrenated phenol, 15.3 percent di-styrenated phenol, and 5.3 percent tri-styrenated phenol. The system was bled with nitrogen gas while the contents of the reactor were stirred at a moderate speed. Then 2.0 grams of magnesium chloride was added rapidly to the reactor. The reactor temperature was gradually raised from 25° to 100° C. over a period of about 1 hour and was maintained for approximately 4 hours during which time 1 mole of hydrogen chloride was collected in the aqueous trap. Thereafter, 268 grams of a commercially available isopropylated phenol was added dropwise to the reactor over a 40 minute period at a temperature of 108°-117° C. This phenol mixture had a composition consisting of 85.6 percent of the ortho, meta and para isomers of isopropyl phenol, 3.8 percent of 2,6-diisopropyl phenol, 9.6 percent 2,4-diisopropyl phenol and 1 percent of triisopropyl phenol. The temperature was gradually raised to 160° C. and maintained at that temperature until 1.9 moles of hydrogen chloride was collected in the aqueous trap. The reaction mixture was then transferred to 1 liter distilling flask and stripped of low boiling substituents at 187° C. (0.50 mm) for 1 hour. The crude product was added dropwise to a flask containing 500 milliliters of well agitated 1 percent caustic solution at 60° C. and stirred for 1 hour. The mixture was allowed to phase and then separated by means of a separatory funnel. The caustic wash was repeated 3 more times at 60° and then washed with a 1 percent solution of phosphoric acid. The product was then washed with water and azeotroped with 1 liter of benzene to dryness. The benzene was removed by distilling at 42° C. (200 mm). The cloudy product was filtered through a bed of Fullers Earth at 60°-70° C. The product was then analyzed and the results are contained in Table I.

EXAMPLE 2

The process of Example 1 was repeated and the analysis is contained in Table I.

TABLE I

Property	Example I	Example II
S.U.S. 100° F.	1,029	1,087
NMR moles styraphenyl per mole phosphate	<1.0	<1.0
Acid No. mg. KOH/gm.	0.14	0.56
n_D^{25}	1.5571	1.5580
d_{25}	1.11	1.11

EXAMPLE 3

The process of Example 1 was repeated. The product was shown by NMR to contain approximately 0.7 styraphenyl groups per molecule. It was compared with a commercially available hydraulic fluid in the same viscosity range sold under the name of Fyrquel 1,000 by Stauffer Chemical Company of New York. The results of this comparison are contained in Table II.

TABLE II

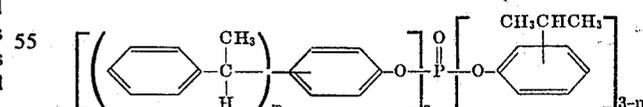
Properties	Styraphenyl bis (isopropylphenyl) phosphate	Fyrquel 1,000
Viscosity at 100° F. (S.U.S.)	1009.9	1,000
Viscosity at 210° F.	61.23	51.8
Viscosity Index	-47	-216
Pour Point, °F.	-5	+30

It is obvious from Table II that the compounds of the present invention exhibit superior viscosity indices and pour points when compared with commercially available hydraulic fluids in the same viscosity range.

The fluids employed in the present invention also perform favorably when tested in a Vickers 104 Vane Pump. During test runs, these fluids exhibit viscosity, color and neutralization characteristics which are equal to or superior to the commercially available hydraulic fluids tested.

WHAT IS CLAIMED IS:

1. A process for the transmission of pressure in a hydraulic system which comprises deploying within said hydraulic system an effective amount of a phosphate ester having the structure corresponding to the formula:



wherein n is a number greater than 0 and less than 1 and m is a number from 1 to 2 inclusive; and applying pressure to said phosphate ester at any point in said system so as to thereby transmit the thus applied pressure throughout said system through the medium of said phosphate.

2. The process of claim 1 wherein n is a number from about 0.5 to about 0.9 inclusive.

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