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SYNTHETIC LUBRICANT

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1 Claim. (Cl. 252—56)

1

This invention relates to synthetic lubricating compositions. Particularly it relates to synthetic lubricants having utility at both high and low temperatures. More particularly the invention relates to new and improved synthetic lubricants of the ester type which exhibit outstanding characteristics as to pour point and low temperature viscosity.

In the lubricating art, considerable progress has been realized in recent years in the production of lubricants characterized by one or more specific properties and adapted for specific uses. In general, this progress is attributed to two developments, new and improved refining techniques, and addition agents capable of imparting particular properties to available lubricants. Thus, viscosity index improvers and pour point depressors are added to available automotive lubricants to render the lubricants more adaptable to change in temperature conditions. There are, of course, limits upon the range of effectiveness of these addition agents, and certain requirements cannot be met in every instance.

With the development of the "turbo-jet" and "turbo-prop" type aircraft engines, which operate at peak efficiency at extremely high altitudes, there has developed a corresponding need for a lubricant which is efficacious at the extremely low temperatures encountered at high altitudes or in frigid areas. Engine manufacturers and operations personnel dealing with these turbine engines must be supplied with a lubricant possessing exceptionally low viscosities at low temperatures and at the same time having satisfactory lubricating qualities at high temperatures.

The mineral lubricating oils which exhibit satisfactory low temperature viscosities have generally been found to have flash points that are dangerously low and high temperature viscosities that are below those required. In other words, when the mineral oil is thin enough at low temperatures, it is too volatile at higher temperatures, where it is also too thin to possess satisfactory lubricity. It has been found that, generally speaking, additive agents do not satisfactorily furnish the required characteristics.

Recently, in an effort to obtain the superior lubricants needed for these uses, a new field nas been explored, namely, the synthesis of lubricants from various materials. Esters represent one class of materials which have attracted unusual interest as synthetic lubricants. Esters are generally characterized by high viscosity indices and flash points and lower pour points than mineral oils of a corresponding viscosity. It is with a synthetic lubricant of the ester type that this invention is concerned.

2

The new synthetic lubricants of the ester type to which the instant invention is directed are highly branched chain esters of adipic acid having the following general structural formula:

$$R—OOC—(CH_2)_4—COOR$$

R in the above formula are highly branched chain aliphatic radicals containing 7 carbon atoms and corresponds to those of alcohols prepared by interaction of propylene dimer with carbon monoxide and hydrogen according to the oxo process.

The oxo process may be described as being the catalytic reaction of a mono-olefin with carbon monoxide and hydrogen. This interaction occurs at temperatures in the order of 300° F. to 400° F. and at pressures in the order of about 1000 to 3000 p. s. i. In the presence of a suitable catalyst such as a heavy metal carbonyl, cobalt carbonyl, for example, there is formed an aldehyde which is subsequently hydrogenated to form a primary alcohol. This process was first developed in Germany and is described in U. S. Patent No. 2,327,066, issued to Roelen in 1943.

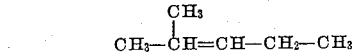
It has been found that diesters of adipic acid formed by reacting said acid with the mixture of alcohols obtained by subjecting a C_6 olefin to the oxo process has outstanding properties as a synthetic lubricant. The C_6 olefin, ordinarily a dimer of propylene, is ordinarily obtained from the subjection of certain refinery streams containing a substantial percentage of propylene to separation processes and polymerizing or dimerizing the propylene component by polymerizing procedures. One such process, known as U. O. P. polymerization, consists of passing the olefin stream in liquid form in contact with an acid catalyst comprising phosphoric acid on kieselguhr. Other acidic catalysts such as liquid phosphoric acids, phosphoric acid or copper phosphate impregnated on silica gel, sulfuric acid, Friedel-Crafts catalysts, activated clays, silica-alumina, pyrophosphate, etc. may also be used. Suitable conditions when employing the phosphoric acid catalysts of the U. O. P. type are temperatures of 300° to 500° F. and pressures of 250 to 5000 p. s. i.

It has been demonstrated that the propylene dimers prepared in accordance with the polymerization processes familiar in the art contain the following two olefins in predominant amounts.

1. 2-methyl-pentene-3

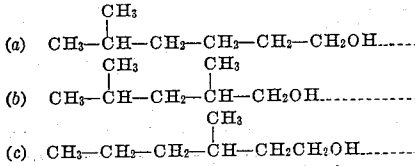
$$CH_3—\underset{\displaystyle CH_3}{\underset{|}{CH}}—CH=CH—CH_3$$

2. 2-methyl-pentene-2



Various other methyl pentenes and dimethyl butene constitute the remainder.

Upon reaction with carbon monoxide and excess hydrogen in the oxo process the mixture of olefins obtained as above result in a series of branched chain alcohols of a great number of structural configurations. For instance, it has been determined that when 2-methyl-pentene-3 is the olefin that is subjected to the oxo process there is formed the following mixture of alcohols:



	Percent
(a)	40
(b)	30
(c)	30

The other olefins present give a proportionate number of alcohols so that the total C_7 oxo alcohol mixture is composed of a great number of variations as to structure, each having one hydroxyl group and 7 carbon atoms.

When a double molecular proportion of this mixture of alcohols is used to esterify a molecular proportion of adipic acid the di-C_7 oxo adipate formed has a molecular configuration which results in properties that make the compound an outstanding synthetic lubricating oil. This will be more completely shown in the following table.

In the table below the properties of seven adipic acid diesters are set out in detail. These esters were all prepared by a procedure familiar to the art of which the following is typical.

Di-C_7 oxo adipate was prepared by mixing 975 g. (8.4 moles) of C_7 oxo alcohol, 584 g. (4 mole) of adipic acid, in the presence of 1 g. of toluenesulfonic acid. This mixture was refluxed and 149 ml. of water removed with the aid of 200 ml. of toluene as water entrainer. The residue was percolated through Attapulgus clay, stripped of toluene and excess alcohol and distilled at 2 mm. pressure. The product distilled sharply at 175° C. and weighed 1200 g. which was 87.5% of the theoretical based on the adipic acid charged.

Table.—Adipic acid esters

Esterifying alcohol	Kinematic Viscosity (C. S.)				Flash Point, ° F.	Pour Point, ° F.	Shell 4-ball E.P. Test (Kg.)	
	210° F.	100° F.	−40° F.	−65° F.			Seiz.	Weld.
C_{10} Oxo Alcohol	3.70	15.20	3,430.0	26,000	450	−75	55	95
C_9 Oxo Alcohol	3.45	12.81	1,955.0	16,600	420	−75		
C_8 Oxo Alcohol	2.80	10.0	1,040.0	6,460	395	−110	50	100
C_7 Oxo Alcohol	2.30	7.4	460.0	2,290	395	−110	50	95
2-ethyl-butyl alcohol	1.77	5.35	465.0	2,310	361	−98		
isohexyl alcohol	1.98	5.68	510.0	Solid	372	−54		
n-amyl alcohol	1.75	4.76	462.0		329	−108		

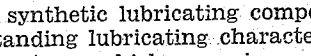
For use in jet engine lubrication it is imperative that the lubricating oil have a combination of high flash point and a low pour point. In addition, the viscosity at 210° F. must be sufficient to provide lubrication at that temperature. A most important feature of a synthetic lubricant for jet engine lubrication is its low temperature viscosity since equipment utilizing jet engines operate at extremely low temperatures at high altitudes. It is essential that the engine lubricant be sufficiently labile at low temperatures to be capable of being circulated in the lubrication system. As is clearly pointed out in the table above, esters of invention have excellent viscosity characteristics at all test temperatures and a flash point and pour point that is outstanding. It will be noted that as the esterifying alcohol chain length decreases the flash point also decreases. One outstanding feature of the C_7 oxo alcohol diester mixture is that it has a flash point as high as the C_8 oxo alcohol esters, a fact which is completely unexpected in the light of the 30°–35° drop in flash points between any other two consecutive esters.

The C_7 oxo ester mixture is also outstanding in its viscosities at −40° F. and −65° F. The viscosity of this mixture at these temperatures is also completely unexpected in the light of the viscosity of the C_8 oxo alcohol ester at these temperatures. It will be noted that the viscosity at −40° F. and −65° F. of those esters below C_7 oxo esters in the table are higher than the C_7 oxo ester mixture. The flash points of these esters make them less desirable for jet lubrication. These flash and viscosity results on the esters have been checked several times on several syntheses.

To summarize briefly, this invention relates to a synthetic lubricating composition having outstanding lubricating characteristics at low temperatures which comprises a mixture of C_7 oxo alcohol esters of adipic acid.

What is claimed is:

A synthetic lubricating oil having outstanding lubricating characteristics at low temperatures which consists essentially of a mixture of diesters of adipic acid, each ester having the formula



wherein R is the combining radical of an alcohol obtained upon reaction of a propylene dimer with carbon monoxide and hydrogen in the presence of a heavy metal carbonyl catalyst at temperatures of about 300 to 400° F. and pressures of from 1,000 to 3,000 atmospheres to form an aldehyde and hydrogenation of said aldehyde to said alcohol.

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