Cycloheptatrienyl Cycloheptatriene Metal Compounds and Process of Making the Same


No Drawing. Filed Dec. 1, 1959, Ser. No. 856,348

Claims (10 Claims).

This invention relates to new organometallic compounds and a method for their preparation. More specifically, this invention relates to organometallic compounds in which metallo carbonyl moieties are bonded to both of the cycloheptatrienyl rings present in a 1-cycloheptatrienyl cycloheptatriene compound.

An object of this invention is to provide new organometallic compounds and a method for their preparation. A more specific object is to provide compounds in which metal atoms as to their electron configuration are satisfied. R1 and R2 are carbon atoms which may be mono- or divalent. Typical of such chemical equivalents thereof are those of the formula

\[
(\text{CO})_2 M_1 \text{M} \text{R} \text{R} \text{M} \text{(CO)}_2
\]

in which \( M_1 \), \( M_2 \), and \( R_2 \) are molybdenum, chromium, and tungsten. The group VIB metals are manganese, technetium, and rhenium, and the group VIII metals are iron, ruthenium, osmium, and nickel. The metals of groups I-B and VIB are preferred in forming the compounds of my invention. Further preferred are my compounds in which \( M_1 \) and \( M_2 \) are molybdenum since these compounds are generally more stable than other of my compounds. "b" and "c" are integers ranging from zero to six and may be the same or different while "d" and "f" are integers ranging from one to three and may be the same or different. "c" is an integer ranging from zero to one.

In the above formula, the metal atoms \( M_1 \) and \( M_2 \) have an electron configuration ranging from two less than up to and including the electron configuration of the next higher rare gas. Preferred compounds are those in which each of the metal atoms has achieved the electron configuration of the next higher rare gas. As an example, in the compound 1-cycloheptatrienyl cycloheptatriene bis(molybdenum tricarbonyl) both molybdenum atoms have the electron configuration of xenon. This is accomplished by donation of 12 electrons to each of the metal atoms \( M_1 \) and \( M_2 \). In this case, the 1-cycloheptatrienyl cycloheptatriene compound contributes 12 electrons, six of which go to \( M_1 \) and six of which go to \( M_2 \). All 12 electrons from the 1-cycloheptatrienyl cycloheptatriene compounds come from their double bonds with each double bond contributing two electrons. Each carbonyl group, which of there are three for each of the two molybdenum atoms, contributes two electrons to the molybdenum atom. This gives a total of six electrons donated by the three carbonyl groups to each molybdenum atom. As exemplified in 1-cycloheptatrienyl cycloheptatriene bis(molybdenum tricarbonyl), the integers "d" and "f" are such that the above enumerated requirements of the metal atoms as to their electron configuration are satisfied. \( R_1 \) and \( R_2 \) are monovalent hydrocarbon groups which may be the same or different. They are substituents on the two cycloheptatrienyl rings comprising the 1-cycloheptatrienyl cycloheptatriene compound. They may contain from one to twenty carbon atoms and may be such radicals as, for example, alkyl, aryl, alkyl, and arylalkyl.

Typical of the compounds of my invention are: 1-methyl-7-(2-methyl-2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(chromium tricarbonyl), 2-ethyl-7-(2-methyl-2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(tungsten tricarbonyl), 7-(2-tert-buty1-2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(nickel carbonyl), 7-(2-ethyl-2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(molybdenum tricarbonyl), 7,3-oxo-2-propyl-2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(coke baryon), 7-(3-isopropyl-2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene molybdenum tricarbonyl chromium tricarbonyl (1:1:1 complex) in which the chromium tricarbonyl moiety is bonded to the isopropyl substituted cycloheptatrienyl ring, 3-ethyl-7-(3-ethyl-2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(iron dicarbonyl), 3-ethyl-7-(3-phenyl-2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(manganese dicarbonyl).

As described above, my invention involves compounds in which metallo carbonyl moieties are bonded to each of the cycloheptatrienyl rings in a 1-cycloheptatrienyl cycloheptatriene compound. My invention is not limited to such compounds, however, and is intended to include all chemical equivalents thereof. Typical of such chemically equivalent compounds are those of the formula

\[
\text{X} = \text{M}_1 \text{M}_2 \text{R}_1 \text{R}_2 \text{X} \text{M}_1 \text{M}_2 \text{R}_1 \text{R}_2
\]

in which \( M_1 \), \( M_2 \), \( R_1 \), \( R_2 \) "b" and "c" have the meaning as set forth above. \( Z_1 \) and \( Z_2 \) are electron donating groups which may be the same or different and are each capable of donating from one to six electrons to the metal atoms \( M_1 \) and \( M_2 \). "p" and "q" are integers ranging from one to six and may be the same or different. The metal atoms \( M_1 \) and \( M_2 \) have, as previously defined, an electron configuration ranging from two less than up to and including the electron configuration of the next higher rare gas.

"X" in the above formula is a bridging group between the two cycloheptatrienyl moieties forming the molecule, which molecule is defined generally as a ditroplyl molecule. It may be any group which contains two open bonds so as to form either a direct link between the two cycloheptatrienyl groups or to form one link in a chain between the cycloheptatrienyl radicals. Typical examples of such groups are as follows:

\[
\begin{align*}
\text{--} & \quad \text{--} \\
\text{R} & \quad \text{R} \\
\text{R} & \quad \text{R} \\
\text{R} & \quad \text{R} \\
\text{0} & \quad \text{0} \\
\text{--} & \quad \text{--}
\end{align*}
\]

Various combinations of the above groups can also be used as the bridging groups "X." Thus, "X" can be:

\[
\begin{align*}
\text{--} & \quad \text{--} \\
\text{O} & \quad \text{O} \\
\text{CH}_2 & \quad \text{CH}_2 \quad \text{O}
\end{align*}
\]

or

\[
\begin{align*}
\text{--} & \quad \text{--} \\
\text{O} & \quad \text{O} \\
\text{--} & \quad \text{--}
\end{align*}
\]

The above examples of groups meeting the requirement of "X" are solely by way of illustration and are not intended to limit the scope of the term "X" in the above formula.
"R," in the above examples of bridging groups, denotes a hydrocarbon radical which may be alkyl, aryl, alkaryl or aralkyl, and can also be hydrogen. Various Z groups capable of sharing a single electron with the metal atom include monovalent inorganic groups such as hydrogen, the halogens and the cyanide group, CN. Such a "Z" group may also be a monovalent organic radical containing up to about 16 carbon atoms. These radicals include alkyl, aryl, alkaryl, and the like. Also included are acyclic unsaturated radicals such as the alkenyl and alkynyl radicals.

"Z" groups capable of sharing two electrons with the metal atom include ammonia, primary-, secondary-, and tertiary-amine, cyclic nitrogen compounds in which the nitrogen is in the trivalent state, monoenoic molecules, organophosphine compounds, phosphine halides, arsines, stibines, and bismuthines, iodonitrides, and the like. The nitrosyl group, NO, is an example of an electron donor capable of donating three electrons to either of the metal atoms, M3 or M2, as in the above formula. Examples of electron donating groups capable of donating six electrons to the metal atom are benzene and substituted benzenes. These groups may contain from six to 18 carbon atoms. Examples of typical donating groups are benzene, mesitylene, tolune, biphenyl, tetralin, mesitylenes and the like.

Typical of those chemical compounds which are equivalent to my compounds as defined above are: 1-ethyl-1-[p - (6-ethyl - 2-methyl - 2,4,6-cycloheptatrienyl - 1 - yl oxy) phenyl] - 6 - methyl - 2 - n propyl - 1,3,5-cycloheptatriene manganese dicarbonyl molybdenum tricarbonyl in which the manganese atom is coordinated with the di substituted cycloheptatrienyl ring and the molybdenum atom is coordinated with the tri substituted ring, 7 - 2,4,6-cycloheptatrienyl - 1 - ycarbonyl - 1,3,5-cycloheptatriene cobalt carbonyl manganese dicarbonyl (1:1:1) bis(2,4,6-cycloheptatrienyl-1-y) methane bis(manganese dicarbonyl) cyanide, 1 - methyl - 7 - ((2-ethyl - 2,4,6-cycloheptatrienyl - 1 - yl) thioc 1,3,5-cycloheptatriene bis(chromium tricarbonyl)1,1-methyl-7-[(4-methyl - 2,4,6-cycloheptatrienyl - 1 - yl) methyl) - 1,3,5-cycloheptatriene bis(chromium tricarbonyl)1,1,3,5,4,5,6-hexamethyl - 7 - (2,4,6-cycloheptatrienyl - 1 - yl - ycarbonyl) - 1,3,5-cycloheptatriene bis(molybdenum tricarbonyl), methyl bis(2,2,4,6-cycloheptatrienyl-1-y) amine chromium tricarbonyl molybdenum tricarbonyl (1:1:1 complex) 2-methyl-7-(dimethyl)-(3-ethyl)-2,4,6-cycloheptatrienyl - 1 - one - 5-propyl - 1,3,5-cycloheptatriene bis(tungsten tricarbonyl), methyl bis(2,4,6-cycloheptatrienyl) phosphate bis(manganese dicarbonyl), 7 - [(2,4,6-cycloheptatrienyl - 1 - yl) - diethylsilyl] methyl - diethylsilyl]-2-p-toly-1,3,5-cycloheptatriene bis(nickel carbonyl), and bis(2,4,6-cycloheptatrienyl-1-y) aluminium bis(cobalt carbonyl).

My compounds are prepared by reacting a 1-cycloheptatrienyl cycloheptatriene compound having the formula with R1, R2, b, c and f having the meanings previously defined

![Diagram](https://via.placeholder.com/150)

with a metal carbonyl compound or a mixture of metal carbonyl compounds in which the metal or metals are chosen from groups VIB-VIIB of the periodic table. In this reaction the 1-cycloheptatrienyl cycloheptatriene compound displaces either two or three carbonyl groups from each molecule of the metal carbonyl reactant so that a metallo carbonyl moiety containing either two or three less carbonyl groups than the original reactant is bonded to each of the cycloheptatrienyl rings of the product. In the formation of a mixed metal compound, this reaction may be carried out stepwise to form an intermediate compound in which a metallo carbonyl moiety is bonded to only one of the cycloheptatrienyl rings. This intermediate is then reacted with an additional quantity of a metal carbonyl compound to bond a metallo carbonyl moiety to the remaining cycloheptatrienyl ring.

In general, the process may be carried out at temperatures between about 75 to about 200° C. Preferably, however, temperatures in the range from about 100 to about 150° C. are employed since, within this range, rela-

tively higher yields are obtained with a minimum of undesirable side reactions. The pressure under which the process is carried out is not critical. Preferably, however, the process is conducted at atmospheric pressure or slightly higher although higher pressures, up to 500 atmospheres, can be employed if desired.

The process is generally conducted under a blanket of inert gas such as nitrogen, helium, argon and the like.

The process is preferably conducted in the presence of a non-reactive solvent. Typical of reaction solvents which may be employed in my process are high boiling satur-

tated hydrocarbons such as n-octane, n-decane, and other paraffinic hydrocarbons having up to about 20 carbon atoms such as eicosane, pentadecane, and the like. Typical ether solvents are ethyl ether, ethyl methyl ether, diethylene glycol, ethylene glycol, diethyl ether, diethylene glycol dibutyl ether, ethylene glycol di-

ethyl ether, ethylene glycol diethyl ether, trioxane, tetra-

dihydrofuran, ethylene glycol dibutyl ether and the like.

Ester solvents which may be employed include pentyi butanoate, ethyl decanoate, ethyl hexanoate, and the like. Silicone oils such as the dimethyl polysiloxanes, bis(cyclo-


perature at which separation of the product has been affected through crystallization. Obviously, if the solvent freezes before the solid product precipitates, it will be impossible to make a separation through crystallization. The above criteria, as to physical properties of the solvent, are not unique to this process. In any chemical process, it is necessary to pick a solvent whose physical properties make it readily separable from the product being formed. It is deemed, therefore, within the skill of the art to select the most suitable solvent... use in any particular embodiment of the process of the invention.

The process is preferably conducted with agitation of the reaction mixture. Although agitation is not critical to the success or failure of the process, its use is preferred since it accomplishes a smooth and even reaction rate.

The time required for the process varies depending on the other reaction variables. In general, however, a time period from about two to about 40 hours is sufficient.

In some cases, the process is advantageously carried out in the presence of an ultraviolet light source. This tends in some cases to decrease the reaction time and give a higher yield of product.

It is desirable to use an excess quantity of the metal carbonyl reactant in order to insure formation of the bis(metalloccarbonyl) 1-cycloheptatrienyl cycloheptatriene compound as opposed to formation of a mono metalloccarbonyl 1-cycloheptatrienyl cycloheptatriene compound. Generally, a total quantity of from about two to about 10 moles of the metal carbonyl reactant are employed for each mole of the 1-cycloheptatrienyl cycloheptatriene reactant. Within this range, it is found that the bis(metalloccarbonyl) 1-cycloheptatrienyl cycloheptatriene compound is formed in relatively good yield.

In some cases, hydroquinone or other free radical reaction inhibitors can be employed in the reaction to prevent any polymerization of the 1-cycloheptatrienyl cycloheptatriene reactant. Their presence is not critical, however, to the success of the reaction. Typical of other applicable free radical inhibitors are p-t-butyl catelol, p-hydroxy anisole, 4-aminonaphth, chloranil, 2,4-di-nitrochlorobenzene, diisocarbamate and the like.

To further illustrate the compounds of the invention and their mode of preparation, there are presented the following examples in which all parts and percentages are by weight unless otherwise indicated.

**Example I**

Nineteen parts of 1-cycloheptatrienyl cycloheptatriene and 56 parts of molybdenum hexacarbonyl were refluxed for 36 hours under nitrogen at a temperature of 100-120°C, in approximately 3,500 parts of petroleum ether. The ether was then removed by heating the reaction mixture at 20°C and a pressure of 0.5 millimeter. Excess molybdenum carbonyl and 1-cycloheptatrienyl cycloheptatriene were sublimed from the residue at 60°C/0.1 mm. The remaining solid was washed with light petrol to remove the 1-cycloheptatrienyl cycloheptatriene bis(molybdenum tricarbonyl) formed in the reaction. The light-brown residue was extracted with hot chloroform. The extracts were combined and concentrated and on cooling to −20°C gave three parts of 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(molybdenum tricarbonyl). The theoretical analysis for this compound is: C, 44.3 percent; H, 2.6 percent; O, 17.7 percent, and Mo, 35.5 percent. Found: C, 43.5 percent; H, 3.1 percent; O, 17.7 percent; Mo, 35.5 percent. When Example I is repeated under slight pressure at a temperature of 150°C, good yields of product are obtained.

**Example II**

79 parts of 7-(2,4,6-cycloheptatrien-1-yl)oxy)-1,3,5-cycloheptatriene and 210 parts of molybdenum hexacarbonyl were heated at reflux for 15 hours under nitrogen at a temperature ranging between 100 and 120°C. The reflux was carried out using approximately 3,500 parts of petroleum ether as a solvent. The solvent was then removed by heating at 20°C/0.5 mm. The unreacted molybdenum hexacarbonyl and 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene were sublimed from the residue by heating at 75°C/0.1 mm. The residue was then washed with light petrol and extracted with hot chloroform. The chloroform extracts were concentrated by means of evaporation and cooled to −20°C. At this point, 7-(2,4,6-cycloheptatrien-1-yl)oxy)-1,3,5-cycloheptatriene bis(molybdenum tricarbonyl) crystallized from the solution. A theoretical analysis of this compound is as follows: C, 42.9 percent; H, 2.5 percent; O, 20.1 percent, and Mo, 34.4 percent. Found: C, 44.3 percent; H, 2.4 percent; O, 20.4 percent, and Mo, 34.2 percent.

**Example III**

A solution comprising 0.50 mole of chromium hexacarbonyl and 0.05 mole of 1-methyl-7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene in n-nonane is heated at reflux under nitrogen for two hours. The reaction product is then discharged from the reaction vessel, filtered, and the excess solvent and unreacted starting materials are removed by heating under vacuum. The residue is dissolved in low-boiling petroleum ether and chromatographed on alumina. The eluate is heated in vacuo to remove the petroleum ether solvent and give a good yield of 1-methyl-7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(chromium tricarbonyl).

**Example IV**

A solution comprising 0.30 mole of tungsten hexacarbonyl, 0.10 mole of 1-ethyl-7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene in petroleum ether is heated under nitrogen for 10 hours at reflux. The reaction product is then filtered, and solvent and unreacted starting materials are removed from the filtrate by heating in vacuo. The residue is dissolved in low-boiling petroleum ether and chromatographed on alumina. On removing the solvent from the eluate by heating under vacuum, there is obtained 1-ethyl-7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(tungsten tricarbonyl).

**Example V**

A solution comprising 0.03 mole of 1-benzyl-7-(2,4,6-cycloheptatrien-1-yl)oxy)-1,3,5-cycloheptatriene and 0.15 mole of manganese carbonyl in diethylene glycol dimethylether solvent is heated at reflux for 40 hours under nitrogen. The reaction mixture is then filtered, and solvent and unreacted starting materials are removed by heating in vacuo. The residue is dissolved in low-boiling petroleum ether and chromatographed on alumina. The product band is heated in vacuo to give a good yield of 1-benzyl-7-(2,4,6-cycloheptatrien-1-yl)oxy)-1,3,5-cycloheptatriene bis(manganese dicarbonyl).

**Example VI**

A solution is formed by dissolving 0.20 mole of iron pentacarbonyl and 0.01 mole of 1-p-tolyl-7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene in tetrahydrofuran. The resulting solution is heated at reflux for 20 hours under nitrogen after which the reaction mixture is filtered, and solvent and unreacted starting materials are removed by heating in vacuo. There is obtained from the residue, by means of chromatographic separation as in the previous examples, a good yield of 1-p-tolyl-7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(iron tricarbonyl).

**Example VII**

One-tenth mole of molybdenum hexacarbonyl and 0.10 mole of 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene are dissolved in n-nonane and the resulting solution is heated at reflux for two hours under nitrogen. The reaction mixture is discharged from the reaction vessel, filtered and heated under vacuum to remove solvent and unreacted starting materials. The residue is dissolved in low-boiling petroleum ether and chromatographed on alumina to yield an eluate which when heated in vacuum
gives 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene molybdenum tricarbonyl (1:1 complex). This compound is dissolved in n-nonane along with 0.10 mole of chromium hexacarbonyl and the resulting solution is heated at reflux for two hours under nitrogen. The reaction product is discharged, filtered and solvent and unreacted starting materials are removed by heating the filtrate under vacuum. The residue is triturated with low-boiling petroleum ether and chromatographed on alumina to yield an eluate which when heated in vacuum gives a good yield of 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene molybdenum tricarbonyl chromiumtricarbonyl 1:1:1 complex.

Example VIII

A solution comprising 0.20 mole of nickel carbonyl and 0.02 mole of 1-ethyl-7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene and 0.04 mole of 3-ethyl-7-(3-ethyl-6-methyl-2,4,6-cycloheptatrien-1-yl)-5,6-dimethyl -1,3,5-cycloheptatriene in di-(2-ethylhexyl) adipate is heated with an ultraviolet light source for five hours at reflux under nitrogen. The reaction product is discharged, filtered, and the filtrate is heated under vacuum to remove unreacted starting materials and solvent. The residue is dissolved in low-boiling petroleum ether and chromatographed on alumina to give a good yield of 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(nickel carbonyl).

Example IX

A solution comprising 0.30 mole of dicobalt octacarbonyl, 0.04 mole of 3-ethyl-7-(3-ethyl-6-methyl-2,4,6-cycloheptatrien-1-yl)-5,6-dimethyl -1,3,5-cycloheptatriene in ethylcyclohexane is heated for 15 hours at reflux under nitrogen. The reaction product is discharged, filtered, and the filtrate is heated under vacuum to remove solvent and unreacted starting materials. The residue is dissolved in low-boiling petroleum ether and chromatographed on alumina to give an eluate which, when heated in vacuum, gives a good yield of 3-ethyl-7-(3-ethyl-6-methyl-2,4,6-cycloheptatrien-1-yl)-5,6-dimethyl -1,3,5-cycloheptatriene as a mixture of two isomers.

As illustrated by the foregoing examples, my process involves reaction between a 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene compound and a group VII-VIII metal carbonyl. Chemically equivalent processes involve reaction between a compound having the structural formula

![Chemical structure](attachment:image)

and a metal-ligand compound containing a group VII-VIII metal and a ligand Z to form a compound

![Compound](attachment:image)

The compounds of my invention can be used in forming metal mirrors comprising a layer or coating of a particular metal selected from groups VII-VIII of the periodic table. These mirrors are formed by thermally decomposing one of the compounds of my invention at a temperature above 400°C. On the decomposition of the compound, the metal deposits on adjacent surfaces to form therein a metallic mirror. These mirrors have useful and desirable properties of electrical conductance and further serve to protect the base material against corrosion. Also, they can be used to decorate the base material as by applying the mirror to a base material that is covered by a stencil. The compounds of the present invention can be deposited on glass, glass mirrors, metal reflector molecules, and other insulating supports. The resultant metal-coated material can then be used as a conductor or as an insulating tape for electrical applications. When the metals are deposited, thermal decomposition on the support, a so-called "printed" electrical circuit can be obtained. It is preferrred that inert gases, e.g., argon, be used to protect the base material from oxidation during the mirror-forming operation.

Deposition on glass cloth illustrates one form of the applied processes. A glass cloth band weighing one gram is dried for one hour in an oven at 150°C. Then together with 0.5 gram of 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(molybdenum tricarbonyl), it is enclosed in a glass tube devoid of air and heated at 400°C for one hour, after which the tube is cooled and opened. The cloth has a uniform metallic appearance and exhibits a gain in weight of about 0.02 gram. The cloth has a greatly decreased resistivity. Each individual fibre proves to be a conductor. As would be expected, the application of a current to the cloth causes an increase in temperature. Thus, a conducting cloth has been prepared. This cloth can be used to reduce static electricity, for decoration, for thermal insulation by reflection, and as a heating element.

The compounds of my invention have further utility as additives to jet fuels, home heater fuels and diesel fuels to reduce smoke and soot. Further, they are excellent antiknock agents when used in fuels and are lubricity improvers when used in lubricants. The compounds of my invention are particularly preferred in which manganese or iron are present in the molecule but none of the compounds of my invention are particularly preferred in which manganese or iron are present in the molecule. My compounds in which molybdenum is present in the molecule are particularly preferred antistatic additives. My compounds may be used alone or in combination with other additives such as scavengers, deposit-modifying agents containing phosphorus or boron and with antiknock agents such as tetraethylen. They may be used in fuels containing from about 0.01 to about 13.8 grams of lead antiknock per gallon.

When introduced in liquid hydrocarbon fuel used in a spark ignition internal combustion engine, my compounds may be used in a concentration range from about 0.015 to about 10 grams per gallon based on the weight of metal. A preferred concentration range is from about 0.03 to about 6 grams of metal per gallon of fuel.

My compounds can be added directly to the hydrocarbon fuels or lubricating oils after which the mixture is agitated until a homogeneous fluid results. Also my compounds may be first blended into concentrated fluids containing solvents such as kerosene, antioxidants and other antiknock agents such as tetraethylen. The concentrated fluid can then be blended with a hydrocarbon fuel to form a fuel particularly adapted for use in a spark ignition internal combustion engine. When my compounds are employed in a concentrated fluid in combination with lead, my compounds are present in an amount so that for each gram of lead present there is a sufficient quantity of my compound to give about 0.01 to about 10 grams of a group VII-VIII metal. A preferred range comprises from about 0.1 to about 6 grams of a group VII-VIII metal as a compound of the instant invention for each gram of lead as an organolead compound.

The scavengers employed in combination with my compounds are either phosphorus compounds or halohydrocarbons. The halohydrocarbons scavengers can be either aliphatic or aromatic with the halogen atoms being attached to carbon atoms either in the aliphatic or aromatic portion of the molecule. The scavenger compounds may be inorganic alcohols or halogenated inorganic compounds. For example, halosiloxyl ethers, halohydrazines, haloesters, haloamides, and the like. When used in forming an antiknock fluid, the atom ratio of metal to halogen ranges from about 50:1 to about 1:1. The halohydrocarbon scavengers normally contain from about two to about 10 carbon atoms in the molecule.

When a phosphorus scavenger is employed with my compounds in formulating an antiknock fluid, it can be present in an amount between about 0.01 to about 1.5 times the amount of phosphorus. A theory of scavenger is that amount of scavenger which will react completely with the...
metal present in the antiknock mixture. Reaction between a halide scavenger and lead gives the lead dihalide. Thus, a theory of halogen scavenger represents, in the case of lead, two atoms of halogen for each atom of lead. A phosphorus scavenger reacts with lead to form lead orthophosphate, $\text{Pb}_2(\text{PO}_4)\text{.}_2$. Thus, a theory of phosphorus represents, in the case of lead, an atom ratio of two atoms of phosphorus to three atoms of lead. Theories of phosphorus or halohydrocarbon scavengers of other metals are computed in the same manner by stoichiometric calculations.

I claim:

1. New organometallic compounds selected from the class consisting of 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(molybdenum tricarbonyl) and 7-(2,4,6-cycloheptatrien-1-yl-oxy)-1,3,5-cycloheptatriene bis(molybdenum tricarbonyl).

2. 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene bis(molybdenum tricarbonyl).

3. 7-(2,4,6-cycloheptatrien-1-yl-oxy)-1,3,5-cycloheptatriene bis(molybdenum tricarbonyl).

4. Process for the preparation of the compounds of claim 1 comprising reacting a compound selected from the class consisting of 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene and 7-(2,4,6-cycloheptatrien-1-yl-oxy)-1,3,5-cycloheptatriene, with molybdenum hexacarbonyl.

5. The process of claim 4 wherein the reaction is carried out in the presence of a non-reactive solvent.

6. The process of claim 5 wherein the process is carried out at temperatures ranging between about 75 to 200° C.

7. The process of claim 5 wherein the reaction temperature ranges from about 100 to about 150° C.

8. The process of claim 7 wherein the process is conducted under a blanketing atmosphere of an inert gas.

9. Process of claim 8 wherein the compound reacted with molybdenum hexacarbonyl is 7-(2,4,6-cycloheptatrien-1-yl)-1,3,5-cycloheptatriene.

10. Process of claim 8 wherein the compound reacted with molybdenum hexacarbonyl is 7-(2,4,6-cycloheptatrien-1-yl-oxy)-1,3,5-cycloheptatriene.

References

Cited in the file of this patent

UNITED STATES PATENTS

2,409,167 Veltman ----------------- Oct. 8, 1946

FOREIGN PATENTS

218,942 Australia ------------- Nov. 26, 1958

OTHER REFERENCES
