METHOD OF MAKING METAL-LIGAND COMPLEXES

Field of Disclosure

[0001] Embodiments of the present disclosure are directed towards methods of making metal-ligand complexes, more specifically, making metal-ligand complexes without isolating intermediate compounds.

Background

[0002] Metallocenes can be used in various applications, including as polymerization catalysts, as antiknock additives in gasoline, and as lubricants, as well as potential use in medical applications such as cancer, malaria and bacterial infection treatments. Applications of metallocenes may depend in part on facile methods for making them, e.g., to reduce undesired impurities, at reasonable cost and scale.

Summary

[0003] The present disclosure provides various ~~embodiments~~examples, including:

[0004] A method of making a metal-ligand complex, the method including: reacting a $(L^1)H$ compound and a base in a primary solvent to make a reaction mixture comprising an intermediate anionic L^1 compound in the primary solvent; and reacting the intermediate anionic L^1 compound and a L^2MX_3 complex to make a $L^1L^2MX_2$ complex, wherein L^1 and L^2 independently are cyclopentadienyl-type (Cp-type) ligands, M is zirconium or hafnium, and each X is a halide and wherein the intermediate anionic L^1 compound is not isolated from the reaction mixture.

[0005] The method comprising reacting the $L^1L^2MX_2$ complex with an alkylating agent to make a $L^1L^2MR_2$ complex, wherein the $L^1L^2MX_2$ complex is not isolated, wherein each R is a hydrocarbyl substituent.

[0005a] In one aspect, the present disclosure refers to a method of making a metal-ligand complex, the method comprising:

reacting a $(L^1)H$ compound and a base in a primary solvent to make a reaction mixture comprising an intermediate anionic L^1 compound in the primary solvent; and

reacting the intermediate anionic L^1 compound and a L^2MX_3 complex to make a $L^1L^2MX_2$ complex, wherein L^1 and L^2 independently are cyclopentadienyl-type (Cp-

type) ligands, M is zirconium or hafnium, and each X is a halide and wherein the intermediate anionic L^1 compound is not isolated from the reaction mixture;

wherein the method comprises reacting the $L^1L^2MX_2$ complex with an alkylating agent to make a $L^1L^2MR_2$ complex, wherein the $L^1L^2MX_2$ complex is not isolated from the reaction mixture, wherein each R is a hydrocarbyl substituent; and

wherein each of L^1 and L^2 is independently a substituted cyclopentadienyl or a substituted indenyl.

Detailed Description

[0006] $L^1L^2MX_2$ complexes, which may be referred to as metal-ligand complexes having halide substituents, can be made by reacting a $(L^1)H$ compound, which may be referred to as an unsaturated cyclic hydrocarbon, and a base in a primary solvent to make a reaction mixture comprising an intermediate anionic L^1 compound in the primary solvent, and reacting the intermediate anionic L^1 compound and a L^2MX_3 complex. Surprisingly, it has been found that the $L^1L^2MX_2$ complex can be made

What is claimed is:

1. A method of making a metal-ligand complex, the method comprising:

reacting a $(L^1)H$ compound and a base in a primary solvent to make a reaction mixture comprising an intermediate anionic L^1 compound in the primary solvent; and

reacting the intermediate anionic L^1 compound and a L^2MX_3 complex to make a $L^1L^2MX_2$ complex, wherein L^1 and L^2 independently are cyclopentadienyl-type (Cp-type) ligands, M is zirconium or hafnium, and each X is a halide and wherein the intermediate anionic L^1 compound is not isolated from the reaction mixture;

wherein the method comprises reacting the $L^1L^2MX_2$ complex with an alkylating agent to make a $L^1L^2MR_2$ complex, wherein the $L^1L^2MX_2$ complex is not isolated from the reaction mixture, wherein each R is a hydrocarbyl substituent; and

wherein each of L^1 and L^2 is independently a substituted cyclopentadienyl or a substituted indenyl.

~~2. The method of claim 1, comprising reacting the $L^1L^2MX_2$ complex with an alkylating agent to make a $L^1L^2MR_2$ complex, wherein the $L^1L^2MX_2$ complex is not isolated from the reaction mixture, wherein each R is a hydrocarbyl substituent.~~

~~3~~2. The method of ~~any one of~~ claim~~s~~ 1~~-2~~, wherein the primary solvent is selected from ethers, polyethers, thioethers, and hydrocarbons.

~~4~~3. The method of any one of claims 1-2, wherein the primary solvent is selected from dimethoxyethane, toluene, ether, tetrahydrofuran, and combinations of any two or more thereof.

~~5~~4. The method of any one of claims 1-~~2~~3, wherein each X is Cl.

~~6~~5. The method of any one of claims ~~2~~1-4, wherein the alkylating agent is an alkylmagnesium halide.

~~7~~6. The method of any one of claims 1-~~2~~5, wherein the base is selected from butyllithium, 1-methylpropyllithium, 2-methylpropyllithium, and 1,1-dimethylethyllithium.

~~8. The method of any one of claims 1-2, wherein each of L^1 and L^2 is independently a substituted cyclopentadienyl or a substituted indenyl.~~

~~9~~7. The method of any one of claims 1-~~2~~6, wherein L^1 and L^2 are different.

~~10~~8. The method of any one of claims 1-~~2~~7, wherein the $L^1L^2MX_2$ complex is (methylcyclopentadienyl)(1,5-dimethylindenyl)hafnium dichloride.

~~11~~9. The method of any one of claims ~~2~~1-8, wherein each R is independently a C_1 to C_{20} alkyl or benzyl.

~~12~~10. The method of any one of claims ~~2~~1-9, wherein ~~the~~ the $L^1L^2MR_2$ complex is (methylcyclopentadienyl)(1,5-dimethylindenyl)hafnium dimethyl.

~~13~~11. The method of any one of claims ~~2~~1-10, wherein the reacting the $(L^1)H$ compound and the base to make the intermediate anionic L^1 compound, and the reacting the intermediate anionic L^1 compound and the L^2MX_3 complex to make the $L^1L^2MX_2$ complex occurs in a same reaction vessel without removing the intermediate anionic L^1 compound from the reaction vessel.

~~14~~12. The method of claim ~~13~~11, wherein the reacting the $(L^1)H$ compound and the base to make the intermediate anionic L^1 compound and the reacting the intermediate anionic L^1 compound and the L^2MX_3 complex to make the $L^1L^2MX_2$ complex, and the reacting the $L^1L^2MX_2$ complex and the alkylating agent to make the $L^1L^2MR_2$ complex occurs in the same reaction vessel without removing the intermediate anionic L^1 compound and the $L^1L^2MX_2$ complex from the reaction vessel.

~~15~~13. The method of any one of claims ~~14~~1-12, wherein the reacting the $(L^1)H$ compound and the base, the reacting the intermediate anionic L^1 compound and the L^2MX_3 complex, and the reacting the $L^1L^2MX_2$ complex with the alkylating agent occurs in the primary solvent.