

ETHYLENE COPOLYMERS FOR PHOTOVOLTAIC CELLSFIELD

[0001] This application claims the benefit of U.S. Provisional Patent Application
5 63/612,454 filed 20 December 2023 entitled "ETHYLENE COPOLYMERS FOR
PHOTOVOLTAIC CELLS," the entirety of which is incorporated by reference herein.

[0002] Embodiments of the present invention generally relate to ethylene copolymers and
electronic device modules embodying such copolymers. More particularly, embodiments
provided herein relate to ethylene copolymers suitable for making photovoltaic cell
10 applications.

BACKGROUND

[0003] Polyolefin plastomers, primarily copolymers of ethylene with butene or octene are
finding increasing use as polymer encapsulant in photovoltaic (PV) cell applications. These
polymers are replacing ethylene vinyl acetate (EVA) copolymers, where studies have shown
15 that plastomer based encapsulant materials have increased power generation over a service life
of 30 years compared to EVA. Plastomer films used as encapsulant materials have a high
barrier to potential induced degradation (PID) and lower power degradation compared to EVA
film, both of which contribute to lower power loss. Evolution of acetic acid in EVA resins from
service use and discoloration due to yellowing leads to increased power loss for EVA film
20 based encapsulant.

[0004] Polymer film encapsulant for PV cell application needs to satisfy several functional
attributes. Electrical properties denoted by high volume resistivity is useful for lower power
loss. Good optical properties, often measured by high light transmission in the wavelength of
280 to 1100 nm, enhanced barrier to moisture represented by low water vapor transmission
25 rates (WVTR), high cross-link density to provide creep resistance and good mechanical
properties represented by tensile strength, flexural modulus and tear strength are considered
important. The problem that needs to be addressed is how to obtain all these functional
attributes in a single polyolefin polymer.

[0005] There are several patents that disclose the use of plastomer resins as encapsulant
30 material in PV Cell. For example, US 9,349,895B2 and its counterpart CN 103189996B
describe an ethylene alpha-olefin copolymer suitable as a PV cell encapsulant with a density
in the range of 0.865 to 0.884 g/cc, MI (190°C) in the range of 2 to 10 and Shore A Hardness
in the range of 60 to 85. US 8581094B2 and its counterpart CN101563786B describe a PV
cell device with a polyolefin copolymer encapsulant having a density less than 0.9 g/cc, melting

point less than 95°C, alpha-olefin content ranging from 15 to 50 wt%, a SCBDI of at least 50 and optionally a free radical initiator and a co-agent. KR 101191126B1 describes an encapsulant sheet for a solar cell, where the sheet comprises an ultra-low density ethylene alpha olefin copolymer (0.850 to 0.890 g/cc), a low density ethylene alpha olefin copolymer (0.890 to 0.920 g/cc) and a silane graft modified ultra-low density copolymer. KR 101723708 B1 describes a polyolefin resin ter-polymer used as encapsulant material, where the polyolefin has a first crystallization temperature in the range of 45°C to 60°C and a second crystallization temperature lower than the first crystallization temperature of the resin.

[0006] US 8,329,848 B2 describes an ethylene butene copolymer with vinyl groups in the range of 0.06 to 1 per 1,000 C atoms, density in the range of 0.850 to 0.910 g/cc, MIR (I10/I2.16) < 7.7, MI in the range of 0.1 to 25 dg/min and ethylene content in the range of 80 to 95 mole %. US 10,774,205B2 describes polymers with multi-modal composition distribution each having a distinct crystallization peak in TREF in the range of 40°C to 110°C.

[0007] However, there is still a need for new ethylene-based copolymers capable of producing a film that has excellent optical properties at wavelengths of 200 to 900 nm, good processability, moisture resistance, creep resistance, tensile strength, flexural modulus and tear strength. Such films would be particularly suitable for solving PV cell application needs.

SUMMARY

[0008] Ethylene copolymers, electronic device modules and methods for making both are provided herein. The ethylene copolymers include at least 50 wt% ethylene derived units and at least 20 wt% of at least one C3 to C20 comonomer. The ethylene copolymer has a melt index of 0.5 g/10 min to about 50 g/10 min, as measured according to ASTM D1238 (190°C/2.16 kg) and a density of about 0.856 g/cc to 0.890 g/cc, as measured according to ASTM D792; volume resistivity at 23°C of at least $5 \times 10^{15} \Omega\text{cm}$; and 0.01 to 4.0 ppm by weight of aluminum. In certain embodiments, the copolymer has a first long chain branching index ($g'(M_z)$) of 0.80 to 0.93, a second long chain branching index ($g'(M_z+1)$) of 0.80 to 0.93, and less than 0.7 vinyl/total unsaturation. In certain embodiments, the unsaturation level of trisubstituted olefins is 50 to 500. Such ethylene copolymers can be made using metallocene and post metallocene catalysts in solution polymerization processes, as further provided herein.

[0009] In at least one embodiment, the electronic device module has at least one electronic device, and an ethylene copolymer film in direct contact with at least one surface of the electronic device. The ethylene copolymer comprises at least 50 wt% ethylene derived units; and at least 20 wt% of at least one C3 to C20 comonomer, wherein the copolymer has: a melt index of 0.5 g/10 min to about 50 g/10 min, as measured according to ASTM D1238

(190°C/2.16 kg); density of about 0.856 g/cc to 0.890g/cc, as measured according to ASTM D792; volume resistivity at 23°C of at least $5 \times 10^{15} \Omega\text{cm}$; and 0.01 to 4.0 ppm by weight of aluminum.

[0010] In at least one embodiment, the method for making the electronic device module includes providing at least one electronic device, and laminating an ethylene copolymer film onto at least one surface of the electronic device. The ethylene copolymer comprises at least 50 wt% ethylene derived units; and at least 20 wt% of at least one C3 to C20 comonomer, wherein the copolymer has a melt index of 0.5 g/10 min to about 50 g/10 min, as measured according to ASTM D1238 (190°C/2.16 kg); a density of about 0.856 g/cc to 0.890 g/cc, as measured according to ASTM D792; a volume resistivity at 23°C of at least $5 \times 10^{15} \Omega\text{cm}$; and 0.01 to 4.0 ppm by weight of aluminum.

[0011] In at least one other embodiment, the method includes polymerizing ethylene derived units and at least one C3 to C20 comonomer in the presence of a catalyst system, and obtaining an ethylene based copolymer polyolefin comprising at least 50 wt% ethylene derived units; and at least 20 wt% of at least one C3 to C20 comonomer, wherein the copolymer has a melt index of 0.5 g/10 min to about 50 g/10 min, as measured according to ASTM D1238 (190°C/2.16 kg); a density of about 0.857 g/cc to 0.890 g/cc, as measured according to ASTM D792; a volume resistivity at 23°C of at least $5 \times 10^{15} \Omega\text{cm}$; and 0.01 to 4.0 ppm by weight of aluminum

[0012] It has been surprisingly found that these ethylene based copolymers have significantly improved processability properties, low aluminum residue, and high volume resistivity ($>5 \times 10^{15} \text{ Ohm} \cdot \text{cm}$), and are capable of producing films having excellent optical properties at wavelengths of 200 to 900 nm, moisture resistance, creep resistance, making these copolymers particularly suitable for electronic device modules such as PV cell applications. It has also been surprisingly discovered that these ethylene based copolymers have less than 4 ppm or less than 2 ppm, or or less than 0.1 ppm or no aluminum residue, making these ethylene based copolymers particularly suitable for use in PV cells and/or modules.

BRIEF DESCRIPTION OF THE DRAWINGS

[0013] So that the manner in which the above recited features of the present invention can be understood in detail, a more particular description of the invention, briefly summarized above, may be had by reference to embodiments, some of which are illustrated in the appended drawings. It is to be noted, however, that the appended drawings illustrate only typical embodiments of this invention and are therefore not to be considered limiting of its scope, for the invention may admit to other equally effective embodiments.

[0014] The Figure shows the volume resistivity (1 mm thick film, 500 V, 5 minutes charging time) of the molded plaques plotted against the aluminum residue (ppmw) in resins 1-4, according to one or more embodiments provided herein.

DETAILED DESCRIPTION

5 [0015] Ethylene copolymers capable of producing films with excellent optical properties at wavelengths of 280 to 1,100 nm, including moisture resistance, creep resistance, tensile strength, and tear strength are provided. The ethylene copolymers have branching indexes $g'(M_z)$ and $g'(M_z+1)$ measured from GPC-4D coupled with trisubstituted olefins and reactivity ratio (r_1r_2) determined using NMR that are significantly different than other ethylene
10 copolymers of similar densities. It has been surprisingly found that these ethylene copolymers provided herein also have significantly improved processability properties and high volume resistivity ($>10^{15}$ Ohm*cm). It has also been surprisingly discovered that these ethylene based copolymers have less than 4 ppm or less than 2 ppm, or or less than 0.1 ppm or no aluminum residue, making these ethylene based copolymers particularly suitable for electronic device
15 modules such as PV cell applications.

[0016] Not wishing to be bound by theory, it has been surprisingly discovered that these ethylene based copolymers having little to no aluminum residue can be made using large, highly branched activators that are soluble in aliphatic solvent(s). Preferably, the desirable activators have formula weights greater than 1310 g/mol, greater than 1350 g/mol, greater than
20 1380 g/mol, greater than 1400 g/mol, greater than 1450 g/mol, greater than 1500 g/mol, greater than 1550 g/mol, greater than 1600 g/mol, or greater than 1650 g/mol. In certain embodiments, the formula weight of the activator can range from a low of about 1310, 1250, or 1400 to a high of about 1600, 1700, or 1950 g/mol.

[0017] It is to be understood that the disclosure provided herein provides several exemplary
25 embodiments for implementing different features, structures, and/or functions of the invention. Exemplary embodiments of components, arrangements, and configurations are described to simplify the present disclosure; however, these exemplary embodiments are provided merely as examples and are not intended to limit the scope of the invention. Additionally, the present disclosure may repeat reference numerals and/or letters in the various exemplary embodiments
30 and across the Figures provided herein. This repetition is for the purpose of simplicity and clarity and does not in itself dictate a relationship between the various exemplary embodiments and/or configurations discussed in the Figures. Moreover, the exemplary embodiments presented herein can be combined in any combination of ways, i.e., any element from one

exemplary embodiment can be used in any other exemplary embodiment, without departing from the scope of the disclosure.

[0018] Additionally, certain terms are used throughout the following description and claims to refer to particular components. As one skilled in the art will appreciate, various entities can refer to the same component by different names, and as such, the naming convention for the elements described herein is not intended to limit the scope of the invention, unless otherwise specifically defined herein. Further, the naming convention used herein is not intended to distinguish between components that differ in name but not function.

[0019] In the following discussion and in the claims, the terms “including” and “comprising” are meant to be open-ended, and thus should be interpreted to mean “including, but not limited to.” The phrase “consisting essentially of” means that the described/claimed composition does not include any other components that will materially alter its properties by any more than 5% of that property, and in any case does not include any other component to a level greater than 3 mass%.

[0020] The term “or” is intended to encompass both exclusive and inclusive cases, i.e., “A or B” is intended to be synonymous with “at least one of A and B,” unless otherwise expressly specified herein.

[0021] The indefinite articles “a” and “an” refer to both singular forms (i.e., “one”) and plural referents (i.e., one or more) unless the context clearly dictates otherwise. For example, embodiments using “an olefin” include embodiments where one, two, or more olefins are used, unless specified to the contrary or the context clearly indicates that only one olefin is used.

[0022] The term “wt%” means percentage by weight, “vol%” means percentage by volume, “mol%” means percentage by mole, “ppm” means parts per million, and “ppm wt” and “wppm” are used interchangeably and mean parts per million on a weight basis. All concentrations herein, unless otherwise stated, are expressed on the basis of the total amount of the composition in question.

[0023] The term “polymer” refers to any two or more of the same or different repeating units/mer units or units. The term “homopolymer” refers to a polymer having units that are the same. The term “copolymer” refers to a polymer having two or more units that are different from each other, and includes terpolymers and the like. The term “terpolymer” refers to a polymer having three units that are different from each other. The term “different” as it refers to units indicates that the units differ from each other by at least one atom or are different isomerically. Likewise, the definition of polymer, as used herein, includes homopolymers, copolymers, and the like. By way of example, when a copolymer is said to have a “propylene”

content of 10 wt% to 30 wt%, it is understood that the repeating unit/mer unit or simply unit in the copolymer is derived from propylene in the polymerization reaction and the derived units are present at 10 wt% to 30 wt%, based on a weight of the copolymer.

[0024] As used herein, "Mn" refers to the number average molecular weight of the different polymers in a polymeric material, "Mw" refers to the weight average molecular weight of the different polymers in a polymeric material, and "Mz" refers to the z average molecular weight of the different polymers in a polymeric material. The terms "molecular weight distribution" (MWD) and "polydispersity index" (PDI) are used interchangeably to refer to the ratio of Mw to Mn. Unless otherwise noted, all molecular weights (e.g., Mw, Mn, Mz) are reported in units of g/mol.

[0025] Nomenclature of elements and groups thereof used herein are pursuant to the Periodic Table used by the International Union of Pure and Applied Chemistry after 1988. An example of the Periodic Table is shown in the inner page of the front cover of Advanced Inorganic Chemistry, 6th Edition, by F. Albert Cotton et al. (John Wiley & Sons, Inc., 1999).

[0026] The ethylene copolymers contain ethylene and at least one other C3-C20 comonomer. Preferred ethylene copolymers are ethylene-butene and ethylene-octene plastomers. The ethylene content of the lower ethylene content fraction can range from a low of 55 wt% to a high of 76 wt%. The ethylene content of the higher ethylene content fraction can range from a low of 60 wt% to a high of 90 wt%. The ethylene content of the overall polymer can range from a low of 60 wt% to a high of 85 wt%.

[0027] The ethylene copolymers can have a melt index of 0.5 g/10 min to about 50 g/10 min, as measured according to ASTM D1238 (190°C/2.16 kg). The melt index can also range from a low of about 0.5, 1.0 or 2.0 to a high of about 30, 40, or 50 g/10 min. The melt index can also range from a low of about 0.5, 3.0 or 5.0 to a high of about 20, 35, or 45 g/10 min.

[0028] The ethylene copolymers can have a density of from 0.850 g/cc to 0.920 g/cc, as measured according to ASTM D792, which indicates that they can serve as plastomers having the combined qualities of elastomers and polymers. The ethylene copolymers can also have a density of about 0.860 g/cc to 0.880 g/cc. The density can range from a low of about 0.850, 0.855, 0.860, 0.865, or 0.870 to a high of about 0.874, 0.876, 0.880, 0.900, or 0.920 g/cc.

[0029] The ethylene copolymers can have a volume resistivity at 23°C of $5 \times 10^{15} \Omega\text{cm}$ or more.

[0030] The ethylene copolymer can have a $g'M_z+1$ to g' -avg ratio of 0.9 to 1.0. This ratio can also range from a low of 0.91, 0.92 or 0.93 to a high of 0.97, 0.98, or 0.99.

[0031] The ethylene copolymers can have less than 0.7 vinyl/total unsaturation as estimated by H-NMR. The vinyl/total unsaturation can range from a low of about 0.01, 0.02, or 0.03 to a high of about 0.5, 0.6, or 0.7. The vinyl/total unsaturation can also range from a low of about 0.1, 0.2, or 0.3 to a high of about 0.5, 0.6, or 0.7.

5 [0032] The ethylene copolymer can have an unsaturation level of trisubstituted olefins of 50 to 500, as determined by H-NMR. The unsaturation level of trisubstituted olefins can also range from about 50, 80 or 100 to a high of about 300, 400, or 500. The unsaturation level of trisubstituted olefins can also range from about 60 to 480; 80 to 420; or 100 to 300.

[0033] The ethylene copolymers can have a reactivity ratio of 0.8 or less. The reactivity ratio
10 can also range from 0.2 to 0.8. The reactivity ratio can also range from a low of 0.2, 0.3, or 0.35 to a high of 0.5, 0.65, or 0.8. The reactivity ratio can also be 0.2, 0.3, 0.4, 0.5, 0.6, 0.7 or 0.8.

Polymerization Process

[0034] The ethylene copolymers can be made using a solution polymerization process.
15 Preferably, the solution polymerization process is a bulk polymerization process, which refers to a polymerization process in which the monomers and/or comonomers being polymerized are used as a solvent or diluent using little or no inert solvent as a liquid or diluent. A small fraction of inert solvent might be used as a carrier for a catalyst and a scavenger.

[0035] The term "solution polymerization" refers to a polymerization process in which the
20 polymer is dissolved in a liquid polymerization medium, such as an inert solvent, monomer(s), or blends thereof. A solution polymerization is typically homogeneous, which refers to a polymerization process where the polymer product is dissolved in the polymerization medium. Such systems are preferably not turbid as described in J. Vladimir Oliveira, C. Dariva, and J. C. Pinto, Ind. Eng. Chem. Res., 29, 2000, 4627. A homogeneous polymerization process is
25 typically a process where at least 90 wt% of the product is soluble in the reaction media.

[0036] A suitable solution polymerization process for preparing the polymer blend compositions disclosed herein is generally described in more detail in U.S. Patent Nos. 9,359,535, 7,470,118, 7,226,553; and 7,033,152, which are incorporated by reference herein in their entirety. WO 2017/058385A1 describes a solution polymerization process using single
30 or multiple spiral heat exchanger systems for continuous polymerization of C2 to C40 olefins, which can also be used and is also incorporated by reference herein in its entirety.

[0037] The ethylene copolymers can exhibit low levels of long chain branching (LCB). In particular, the ethylene copolymers can have a first long chain branching index ($g'(M_z)$) ranging from 0.30 to 1.00, preferably from 0.70 to 0.97. The first long chain branching index

($g'(M_z)$) can also range from 0.80 to 0.93. The first long chain branching index ($g'(M_z)$) can also range from a low of 0.80, 0.82, or 0.85 to high of 0.90, 0.92, or 0.93. The first long chain branching index ($g'(M_z)$) can also be 0.80, 0.81, 0.82, 0.83, 0.84, 0.85, 0.86, 0.87, 0.88, 0.89, 0.90, 0.91, 0.92, or 0.93.

5 **[0038]** The ethylene copolymers can have a second long chain branching index ($g'(M_{z+1})$) of from 0.30 to 1.00, preferably from 0.70 to 0.97. The second long chain branching index ($g'(M_{z+1})$) can also range from 0.80 to 0.93. The second long chain branching index ($g'(M_{z+1})$) can also range from a low of 0.80, 0.82, or 0.85 to high of 0.90, 0.92, or 0.93. The second long chain branching index ($g'(M_{z+1})$) can also be 0.80, 0.81, 0.82, 0.83, 0.84, 0.85, 10 0.86, 0.87, 0.88, 0.89, 0.90, 0.91, 0.92, or 0.93.

[0039] The ethylene copolymers provided herein can be used in a variety of end-use applications. The ethylene copolymers provided herein are also particularly suitable for use in making solar cells (also known as photovoltaic cells), photovoltaic (PV) modules and other low-current electronic devices or modules such as liquid crystal panels, electro-luminescent 15 devices and plasma display units. A solar cell module typically has one or more cells made from silicon, gallium-arsenic and copper-iridium-selenium with a top transparent protective material and a bottom protective substrate material, with the solar cell and the protective materials fixed by use of an encapsulating material. The ethylene copolymers provided herein can be used as the top protective material, the bottom protective material or both. The ethylene 20 copolymers can provide a film having excellent flexibility, transparency and heat resistance, making the film particularly suitable for use in PV modules.

[0040] Such PV modules often utilize an electronic device in combination with one or more substrates that provide protection and or support for their manufacture, transportation, and use. For example, these types of devices are frequently positioned behind one or more glass cover 25 sheets and/or between two substrates in which one or both of the substrates are made of glass, metal, plastic, rubber or other material. In these cases, the ethylene copolymer can be used as an encapsulant or sealant for the device within the module or, depending upon the design of the module, directly as a covering or skin layer of the module, e.g., a backskin in a solar cell module.

30 **[0041]** The ethylene copolymers can have a unique combination of any two or more of the following attributes:

- a. a volume resistivity at 23°C $>5 \times 10^{15} \Omega\text{cm}$;
- b. $g'M_{z+1}/g'_{\text{avg}} < 1$ for $g'M_{z+1}(\text{branching}) < 0.93$;

- c. significantly higher trisubstituted olefins for $g'M_z$ (branching) <0.93 and $g'M_z+1$ (branching) <0.93 ;
- d. <0.2 vinyl/total unsaturation for $g'M_z < 0.94$;
- e. reactivity ratios ≤ 0.7 for any $g'M_z < 0.94$; and/or
- 5 f. lower r_1r_2 values than comparative copolymers (most >1 and up to ~ 1.5)

[0042] Another unique aspect of the ethylene copolymers provided herein is the aluminum residue attributable to the activator and scavenger used. It has been surprisingly discovered that ethylene copolymers with the foregoing combination of attributes can have less than 4 ppm or less than 2 ppm, or less than 1 ppm, or less than 0.5 ppm, or less than 0.2 ppm, or less than 10 0.1 ppm, or less than 0.01 ppm or no aluminum residue, making these ethylene copolymers particularly suitable for use in PV cells and/or modules. These unique attributes distinguish the ethylene copolymers from other comparative ethylene copolymers.

Comonomer

[0043] The at least one other comonomer can include any one or more C4 to C20 olefins. 15 The C4 to C20 comonomers can be linear, branched, or cyclic. Suitable C4 to C20 cyclic olefins can be strained or unstrained, monocyclic or polycyclic, and can optionally include heteroatoms and/or one or more functional groups. The reactor C2 concentration can range from 0.1 to 40.0 wt% while the reactor comonomer concentration can range from 0.1 to 40.0 wt%.

20 [0044] Specific examples of comonomers include butene, pentene, hexene, heptene, octene, nonene, decene, undecene, dodecene, norbornene, norbornadiene, dicyclopentadiene, cyclopentene, cycloheptene, cyclooctene, cyclooctadiene, cyclododecene, 7-oxanorbornene, 7-oxanorbornadiene, substituted derivatives thereof, and isomers thereof, preferably hexene, heptene, octene, nonene, decene, dodecene, cyclooctene, 1,5 -cyclooctadiene, 1-hydroxy-4- 25 cyclooctene, 1-acetoxy-4-cyclooctene, 5-methylcyclopentene, cyclopentene, dicyclopentadiene, norbornene, norbornadiene, and their respective homologs and derivatives, preferably norbornene, norbornadiene, and dicyclopentadiene.

Catalyst System

[0045] For purposes of the present disclosure, a "catalyst system" is a combination of at least 30 one catalyst compound, an activator, and an optional support material. The catalyst system can further include one or more additional catalyst compounds. For the purposes of the present disclosure, when catalyst systems are described as comprising neutral stable forms of the components, it is well understood by one of ordinary skill in the art, that the ionic form of the component is the form that reacts with the monomers to produce polymers. Catalysts and

activators of the presented disclosure are intended to embrace ionic forms in addition to the neutral forms of the compounds.

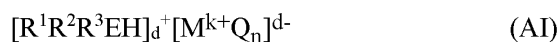
- [0046]** A suitable catalyst system for making the ethylene copolymers provided herein can include one or more bridged metallocene compounds represented by the formula:
- 5 CpA(T)CpBM'X'ⁿ, wherein each CpA and CpB is independently selected from cyclopentadienyl ligands (for example, Cp, Ind, or Flu) and ligands isolobal to cyclopentadienyl, where one or both CpA and CpB may contain heteroatoms, and one or both CpA and CpB may be substituted by one or more R" groups; M' is selected from Groups 3 through 12 atoms and lanthanide Group atoms, preferably Group 4; X' is an anionic leaving
- 10 group; n is 0 or an integer from 1 to 4; (T) is a bridging group selected from divalent alkyl, divalent substituted alkyl, divalent heteroalkyl, divalent alkenyl, divalent substituted alkenyl, divalent heteroalkenyl, divalent alkynyl, divalent substituted alkynyl, divalent heteroalkynyl, divalent alkoxy, divalent aryloxy, divalent alkylthio, divalent arylthio, divalent aryl, divalent substituted aryl, divalent heteroaryl, divalent aralkyl, divalent aralkylene, divalent alkaryl,
- 15 divalent alkarylene, divalent haloalkyl, divalent haloalkenyl, divalent haloalkynyl, divalent heteroalkyl, divalent heterocycle, divalent heteroaryl, a divalent heteroatom-containing group, divalent hydrocarbyl, divalent substituted hydrocarbyl, divalent heterohydrocarbyl, divalent silyl, divalent boryl, divalent phosphino, divalent phosphine, divalent amino, divalent amine, divalent ether, divalent thioether. R" is selected from alkyl, substituted alkyl, heteroalkyl,
- 20 alkenyl, substituted alkenyl, heteroalkenyl, alkynyl, substituted alkynyl, heteroalkynyl, alkoxy, aryloxy, alkylthio, arylthio, aryl, substituted aryl, heteroaryl, aralkyl, aralkylene, alkaryl, alkarylene, haloalkyl, haloalkenyl, haloalkynyl, heteroalkyl, heterocycle, heteroaryl, a heteroatom-containing group, hydrocarbyl, substituted hydrocarbyl, heterohydrocarbyl, silyl, boryl, phosphino, phosphine, amino, amine, germanium, ether, and thioether.
- 25 **[0047]** In at least one embodiment, each of CpA and CpB is independently selected from cyclopentadienyl, indenyl, fluorenyl, cyclopentaphenanthrenyl, benzindenyl, fluorenyl, octahydrofluorenyl, cyclooctatetraenyl, cyclopentacyclododecene, phenanthrindenyl, 3,4-benzofluorenyl, 9-phenylfluorenyl, 8-H-cyclopent[a]acenaphthylenyl, 7-H-dibenzofluorenyl, indeno[1,2-9]anthrene, thiophenoindenyl, thiophenofluorenyl, hydrogenated,
- 30 and substituted versions thereof, preferably cyclopentadienyl, n-propylcyclopentadienyl, indenyl, pentamethylcyclopentadienyl, tetramethylcyclopentadienyl, and n-butylcyclopentadienyl. Each CpA and CpB may independently be indacenyl or tetrahydroindenyl. Particularly suitable cyclopentadienyl-based complexes are described in WO2000/024793, which is incorporated by reference herein.

Activator

[0048] The bridged metallocene compounds can be activated for polymerization catalysis in any manner sufficient to allow coordination or cationic polymerization. This can be achieved for coordination polymerization when one ligand can be abstracted and another will either
 5 allow insertion of the unsaturated monomers or will be similarly abstractable for replacement with a ligand that allows insertion of the unsaturated monomer (labile ligands), e.g., alkyl, silyl, or hydride. Suitable activators for use herein include ammonium or phosphonium groups with long-chain aliphatic hydrocarbyl groups for improved solubility of the activator in aliphatic solvents, as compared to conventional activator compounds. Suitable activators for use herein
 10 can further provide polyolefins having a weight average molecular weight (Mw) of about 100,000 g/mol or greater and a melt temperature (Tm) of about 110°C or greater. Further, activators having a cation having at least one methyl group, and optionally at least one C10 to C50 linear alkyl group can provide enhanced activity for polymer production.

[0049] The present disclosure provides activators, such as ammonium or phosphonium
 15 metallate or metalloid activator compounds, comprising ammonium or phosphonium groups with long-chain aliphatic hydrocarbyl groups combined with metallate or metalloid anions, such as borates or aluminates. When an activator of the present disclosure is used with a catalyst compound (such as a group 4 metallocene compound) in an olefin polymerization, a polymer can be formed having a higher molecular weight and melt temperature than polymers
 20 formed using comparative activators. Likewise, when an activator of the present disclosure where R1 is methyl is used with a group 4 metallocene catalyst in an olefin polymerization, the catalyst system activity is substantially better than comparative activators and can form polymers having a higher molecular weight and/or melt temperature vs. polymers formed using comparative activators.

25 [0050] This invention further relates to activator compounds represented by Formula (AI):



wherein: E is nitrogen or phosphorous, preferably nitrogen;

d is 1, 2 or 3; k is 1, 2, or 3 (preferably 3); n is 1, 2, 3, 4, 5, or 6 (preferably 4, 5 or 6);

n - k = d (preferably d is 1, 2 or 3; k is 3; n is 4, 5, or 6, preferably when M is B, n is 4);

30 R¹ is an optionally substituted C₁-C₂₀ (or C₁ to C₁₀, or C₁-C₆, or C₁-C₄, or C₁-C₂, or C₁) linear alkyl group;

each of R² and R³ is independently an optionally substituted C₁-C₄₀ linear alkyl group (such as a C₆ to C₄₀ linear alkyl group, or a C₁₀ to C₃₀ linear alkyl group) or a meta- and/or para-substituted phenyl group, where the meta and para substituents are, independently, an

optionally substituted C₁ to C₄₀ hydrocarbyl group (such as a C₆ to C₄₀ aryl group or linear alkyl group, a C₁₂ to C₃₀ aryl group or linear alkyl group, or a C₁₀ to C₂₀ aryl group or linear alkyl group), an optionally substituted alkoxy group, an optionally substituted silyl group, a halogen (Br, Cl, I, F, etc.), or a halogen containing group (such as bromoalkyl or bromoaryl),

wherein R¹, R², and R³ together comprise 15 or more carbon atoms (such as 18 or more carbon atoms, such as 20 or more carbon atoms, such as 22 or more carbon atoms, such as 25 or more carbon atoms, such as 30 or more carbon atoms, such as 35 or more carbon atoms, such as 38 or more carbon atoms, such as 40 or more carbon atoms, such as 15 to 100 carbon atoms, such as 25 to 75 carbon atoms);

M is an element selected from group 13 of the Periodic Table of the Elements, preferably boron or aluminum; and

each Q is independently a hydride, bridged or unbridged dialkylamido, halide, alkoxide, aryloxy, hydrocarbyl, substituted hydrocarbyl, halocarbyl, substituted halocarbyl, or halosubstituted-hydrocarbyl radical, provided that when Q is a fluorophenyl group, then R² is not a C₁-C₄₀ linear alkyl group, preferably R² is not an optionally substituted C₁-C₄₀ linear alkyl group (alternately when Q is a substituted phenyl group, then R² is not a C₁-C₄₀ linear alkyl group, preferably R² is not an optionally substituted C₁-C₄₀ linear alkyl group). Preferably, when Q is a fluorophenyl group (alternately when Q is a substituted phenyl group), then R² is a meta- and/or para-substituted phenyl group, where the meta and para substituents are, independently, an optionally substituted C₁ to C₄₀ hydrocarbyl group (such as a C₆ to C₄₀ aryl group or linear alkyl group, a C₁₂ to C₃₀ aryl group or linear alkyl group, or a C₁₀ to C₂₀ aryl group or linear alkyl group), an optionally substituted alkoxy group, or an optionally substituted silyl group. Preferably, each Q is a fluorinated hydrocarbyl group having 1 to 30 carbon atoms, more preferably each Q is a fluorinated aryl (such as phenyl or naphthyl) group, and most preferably each Q is a perfluorinated aryl (such as phenyl or naphthyl) group. Examples of suitable [M^{k+}Q_n]^{d-} also include diboron compounds as disclosed in US Patent No. 5,447,895, which is fully incorporated herein by reference. Preferably at least one Q is not substituted phenyl, preferably all Q are not substituted phenyl. Preferably at least one Q is not perfluorophenyl, preferably all Q are not perfluorophenyl.

[0051] In some embodiments of the invention, R₁ is not methyl, R₂ is not C₁₈ alkyl and R₃ is not C₁₈ alkyl, alternately R₁ is not methyl, R₂ is not C₁₈ alkyl and R₃ is not C₁₈ alkyl and at least one Q is not substituted phenyl, preferably all Q are not substituted phenyl.

[0052] In embodiments, the meta and para substituents are, independently, an optionally substituted linear alkyl group (such as n-hexyl, n-heptyl, n-octyl, n-nonyl, n-decyl, n-undecyl,

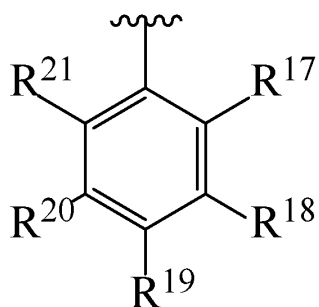
n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, n-icosyl, n-henicosyl, n-docosyl, n-tricosyl, n-tetracosyl, n-pentacosyl, n-hexacosyl, n-heptacosyl, n-octacosyl, n-nonacosyl, or n-tricontyl), an optionally substituted silyl group, such as a trialkylsilyl group, where each alkyl is independently an optionally substituted C₁ to C₂₀ alkyl (such as trimethylsilyl, triethylsilyl, tripropylsilyl, tributylsilyl, trihexylsilyl, triheptylsilyl, trioctylsilyl, trinonylsilyl, tridecylsilyl, triundecylsilyl, tridodecylsilyl, tri-tridecylsilyl, tri-tetradecylsilyl, tri-pentadecylsilyl, tri-hexadecylsilyl, tri-heptadecylsilyl, tri-octadecylsilyl, tri-nonadecylsilyl, tri-icosylsilyl), or an optionally substituted alkoxy group (such as -OR*, where R* is an optionally substituted C₁ to C₂₀ alkyl or aryl (such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, nonadecyl, icosyl, phenyl, phenyl alkyl (such as methyl phenyl, propyl phenyl, etc.), naphthyl, or anthracenyl), a halogen (such as Br or Cl) or a halogen containing group (such as bromomethyl, bromophenyl, and the like).

[0053] In certain embodiments, the meta-substituted phenyl is methylphenyl, ethylphenyl, n-propylphenyl, n-butylphenyl, n-pentylphenyl, n-hexylphenyl, n-heptylphenyl, n-octylphenyl, n-nonylphenyl, n-decylphenyl, n-undecylphenyl, n-dodecylphenyl, n-tridecylphenyl, n-tetradecylphenyl, n-pentadecylphenyl, n-hexadecylphenyl, n-heptadecylphenyl, n-octadecylphenyl, n-nonadecylphenyl, n-icosylphenyl, n-henicosylphenyl, n-docosylphenyl, n-tricosylphenyl, n-tetracosylphenyl, n-pentacosylphenyl, n-hexacosylphenyl, n-heptacosylphenyl, n-octacosylphenyl, n-nonacosylphenyl, n-tricontylphenyl, dimethylphenyl, diethylphenyl, di-n-propylphenyl, di-n-butylphenyl, di-n-pentylphenyl, di-n-hexylphenyl, di-n-heptylphenyl, di-n-octylphenyl, di-n-nonylphenyl, di-n-decylphenyl, di-n-undecylphenyl, di-n-dodecylphenyl, di-n-tridecylphenyl, di-n-tetradecylphenyl, di-n-pentadecylphenyl, di-n-hexadecylphenyl, di-n-heptadecylphenyl, di-n-octadecylphenyl, di-n-nonadecylphenyl, di-n-icosylphenyl, di-n-henicosylphenyl, di-n-docosylphenyl, di-n-tricosylphenyl, di-n-tetracosylphenyl, di-n-pentacosylphenyl, di-n-hexacosylphenyl, di-n-heptacosylphenyl, di-n-octacosylphenyl, di-n-nonacosylphenyl, and di-n-tricontylphenyl. The two meta substituents can be the same or different.

[0054] In embodiments, the para-substituted phenyl is methylphenyl, ethylphenyl, n-propylphenyl, n-butylphenyl, n-pentylphenyl, n-hexylphenyl, n-heptylphenyl, n-octylphenyl, n-nonylphenyl, n-decylphenyl, n-undecylphenyl, n-dodecylphenyl, n-tridecylphenyl, n-tetradecylphenyl, n-pentadecylphenyl, n-hexadecylphenyl, n-heptadecylphenyl, n-octadecylphenyl, n-nonadecylphenyl, n-icosylphenyl, n-henicosylphenyl, n-docosylphenyl, n-

tricosylphenyl, n-tetracosylphenyl, n-pentacosylphenyl, n-hexacosylphenyl, n-heptacosylphenyl, n-octacosylphenyl, n-nonacosylphenyl, or n-tricontylphenyl.

[0055] In embodiments, the meta- and/or para-substituted phenyl group is represented by the formula:



5

where R^{17} and R^{21} are hydrogen, and each of R^{18} , R^{19} , and R^{20} is independently selected from hydrogen, C_1 - C_{40} hydrocarbyl or C_1 - C_{40} substituted hydrocarbyl, halogen, or a halogen-containing group (provided that at least one of R^{18} , R^{19} , and R^{20} is not H, alternately at least two of R^{18} , R^{19} , and R^{20} are not H, alternately all three of R^{18} , R^{19} , and R^{20} are not H), or a combination thereof.

10

[0056] Preferably, R^{17} and R^{21} are hydrogen, and each of R^{18} , R^{19} , and R^{20} is selected from the group consisting of hydrogen, C_1 to C_{40} linear alkyl or C_1 - C_{40} substituted linear alkyl (provided that at least one of R^{18} , R^{19} , and R^{20} is not H, alternately at least two of R^{18} , R^{19} , and R^{20} are not H, alternately all three of R^{18} , R^{19} , and R^{20} are not H).

15

[0057] Preferably, R^{17} and R^{21} are H, and one, two, or three of R^{18} , R^{19} , and R^{20} are selected from the group consisting of H, a linear alkyl group (such as n-hexyl, n-heptyl, n-octyl, n-nonyl, n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, n-icosyl, n-henicosyl, n-docosyl, n-tricosyl, n-tetracosyl, n-pentacosyl, n-hexacosyl, n-heptacosyl, n-octacosyl, n-nonacosyl, or n-tricontyl), an optionally substituted trialkylsilyl group, where each alkyl is independently a C_1 to C_{20} optionally substituted alkyl (such as trimethylsilyl, triethylsilyl, tripropylsilyl, tributylsilyl, trihexylsilyl, triheptylsilyl, trioctylsilyl, trinonylsilyl, tridecylsilyl, triundecylsilyl, tridodecylsilyl, tri-tridecylsilyl, tri-tetradecylsilyl, tri-pentadecylsilyl, tri-hexadecylsilyl, tri-heptadecylsilyl, tri-octadecylsilyl, tri-nonadecylsilyl, tri-icosylsilyl), a halogen (such as Br, Cl, or F), or an optionally substituted alkoxy group (such as $-OR^*$, where R^* is a C_1 to C_{20} optionally substituted alkyl or aryl (such as methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, nonadecyl, icosyl, phenyl, phenyl alkyl (such as methyl phenyl, propyl phenyl, etc.)),

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naphthyl, or anthracene), provided that at least one of R¹⁸, R¹⁹, and R²⁰ is not H, alternately at least two of R¹⁸, R¹⁹, and R²⁰ are not H, alternately all three of R¹⁸, R¹⁹, and R²⁰ are not H).

[0058] In embodiments, R¹ is methyl, R² is a C₁ to C₄₀ linear alkyl group (such as a C₆ to C₄₀ linear alkyl, or C₁₀ to C₃₀ linear alkyl), and R³ is a para-substituted phenyl group, wherein the para substituent is, independently, an optionally substituted C₁ to C₄₀ hydrocarbonyl group (such as a C₆ to C₄₀ aryl group or linear alkyl group, a C₁₂ to C₃₀ aryl group or linear alkyl group, or a C₁₀ to C₂₀ aryl group or linear alkyl group), an optionally substituted alkoxy group, an optionally substituted silyl group, a halogen, or a halogen containing group.

[0059] In embodiments, R¹ is methyl, R² is n-hexyl, n-heptyl, n-octyl, n-nonyl, n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, or n-icosyl, and R³ is methylphenyl, ethylphenyl, n-propylphenyl, n-butylphenyl, n-pentylphenyl, n-hexylphenyl, n-heptylphenyl, n-octylphenyl, n-nonylphenyl, n-decylphenyl, n-undecylphenyl, n-dodecylphenyl, n-tridecylphenyl, n-tetradecylphenyl, n-pentadecylphenyl, n-hexadecylphenyl, n-heptadecylphenyl, n-octadecylphenyl, n-nonadecylphenyl, or n-icosylphenyl.

[0060] In embodiments of any activator formula herein, each R² and/or R³ can be independently optionally substituted with at least one of halide, C₁-C₅₀ alkyl, C₅-C₅₀ aryl, C₆-C₃₅ arylalkyl, or C₆-C₃₅ alkylaryl, provided that substituted R² and R³ groups are not branched alkyl groups (as defined above).

[0061] In at least one embodiment of the invention, an activator is an ionic ammonium or phosphonium borate represented by Formula (I):



wherein:

E is nitrogen or phosphorous;

R¹ is a C₁-C₄₀ linear alkyl,

each of R² and R³ is independently C₁-C₄₀ linear alkyl, C₅-C₂₂-aryl, C₅ to C₅₀ arylalkyl where the alkyl has from 1 to 30 (or 1 to 10) carbon atoms and the aryl has from 6 to 20 carbon atoms, or five-, six- or seven-membered heterocyclyl comprising at least one atom selected from N, P, O and S, wherein each of R¹, R², and R³ is optionally substituted by halogen, wherein R² optionally bonds with R⁵ to independently form a five-, six- or seven-membered ring, preferably wherein, R¹, R², and R³ together comprise 15 or more carbon atoms, such as 18 or more carbon atoms, such as 20 or more carbon atoms, such as 22 or more carbon atoms, such as 25 or more carbon atoms, such as 30 or more carbon atoms, such as 35 or more carbon atoms, such as 40 or more carbon atoms and each of R⁴, R⁵, R⁶, and R⁷ is independently each

a fluorinated hydrocarbyl group having 1 to 30 carbon atoms, more preferably each of R⁴, R⁵, R⁶, and R⁷ is independently is a fluorinated aryl (such as phenyl or naphthyl) group, and most preferably each of R⁴, R⁵, R⁶, and R⁷ is independently is a perfluorinated aryl (such as phenyl or naphthyl) group, wherein at least one of R⁴, R⁵, R⁶, and R⁷ is substituted with from one to seven fluorine atoms, preferably at least one R⁴, R⁵, R⁶, and R⁷ is not substituted phenyl, preferably all of R⁴, R⁵, R⁶, and R⁷ are not substituted phenyl.

[0062] In a preferred embodiment, R¹ is not methyl, R² is not C18 and R³ is not C18. In a preferred embodiment, R¹ is not methyl, R² is not C18 and R³ is not C18 and at least one R⁴, R⁵, R⁶, and R⁷ is not substituted phenyl, preferably all of R⁴, R⁵, R⁶, and R⁷ are not substituted phenyl.

[0063] This invention also relates to activator compounds represented by Formula (I):



wherein: E is nitrogen or phosphorous;

each of R¹, R², and R³ is independently C₁-C₄₀ linear alkyl, C₅-C₅₀-aryl, wherein each of R¹, R², and R³ is independently unsubstituted or substituted with at least one of halide, C₁-C₅₀ alkyl, C₅-C₅₀ aryl, C₆-C₃₅ arylalkyl, or C₆-C₃₅ alkylaryl, wherein R¹, R², and R³ together comprise 15 or more carbon atoms, such as 18 or more carbon atoms, such as 20 or more carbon atoms, such as 22 or more carbon atoms, such as 25 or more carbon atoms, such as 30 or more carbon atoms, such as 35 or more carbon atoms, such as 40 or more carbon atoms, and each of R⁴, R⁵, R⁶, and R⁷ is naphthyl, wherein at least one of R⁴, R⁵, R⁶, and R⁷ is substituted with from one to seven fluorine atoms.

[0064] In a preferred aspect, the activator is an ionic ammonium borate represented by Formula (I):



wherein:

E is nitrogen or phosphorous;

R¹ is a methyl group;

R² is C₆-C₅₀ aryl which is optionally substituted with at least one of halide, C₁-C₃₅ alkyl, C₅-C₁₅ aryl, C₆-C₃₅ arylalkyl, and C₆-C₃₅ alkylaryl;

R³ is C₁-C₄₀ linear alkyl or C₅-C₄₂-aryl which is optionally substituted with at least one of halide, C₁-C₃₅ alkyl, C₅-C₁₅ aryl, C₆-C₃₅ arylalkyl, and C₆-C₃₅ alkylaryl, wherein R² optionally bonds with R³ to independently form a five-, six- or seven-membered ring, and R² and R³ together comprise 20 or more carbon atoms, such as 21 or more carbon atoms, such as 22 or

more carbon atoms, such as 25 or more carbon atoms, such as 30 or more carbon atoms, such as 35 or more carbon atoms, such as 40 or more carbon atoms, and

each of R⁴, R⁵, R⁶, and R⁷ is independently naphthyl or substituted naphthyl, wherein at least one of R⁴, R⁵, R⁶, and R⁷ is naphthyl substituted with from one to seven fluorine atoms.

- 5 [0065] In any embodiment of formula (I) or (AI), R² is unsubstituted phenyl or substituted phenyl. In at least one embodiment, R² is phenyl, methyl phenyl, n-butyl phenyl, n-octadecyl-phenyl, or an isomer thereof, preferably R² is meta or para substituted phenyl, such as meta- or para- substituted alkyl substituted phenyl. In at least one embodiment, R³ is independently selected from C₁ to C₃₀ linear alkyl, such as methyl, ethyl, n-propyl, n-butyl, n-pentyl, n-hexyl, 10 n-heptyl, n-octyl, n-nonyl, n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, and n-icosyl.

[0066] In any embodiment of formula (I) or (AI), each of R⁴, R⁵, R⁶, and R⁷ is independently naphthyl, wherein at least one of R⁴, R⁵, R⁶, and R⁷ is naphthyl substituted with one, two, three, four, five, six or seven fluorine atoms.

- 15 [0067] In any embodiment of formula (I) or (AI), preferably at least one R⁴, R⁵, R⁶, and R⁷ is not substituted phenyl, preferably all of R⁴, R⁵, R⁶, and R⁷ are not substituted phenyl. In a preferred embodiment, R¹ is not methyl, R² is not C18 and R³ is not C18.

[0068] In any embodiment of formula (I) or (AI), preferably all Q or all of R⁴, R⁵, R⁶, and R⁷ are not perfluoroaryl, such as perfluorophenyl.

- 20 [0069] In any embodiment of formula (I) or (AI), all of R⁴, R⁵, R⁶, and R⁷ are naphthyl, wherein at least one, two, three, or four of R⁴, R⁵, R⁶, and R⁷ is/are substituted with one, two, three, four, five, six or seven fluorine atoms.

- [0070] In any embodiment described herein, preferably each of R⁴, R⁵, R⁶, and R⁷ is independently a naphthyl comprising one fluorine atom, two fluorine atoms, three fluorine atoms, four fluorine atoms, five fluorine atoms, six fluorine atoms, or seven fluorine atoms, preferably seven fluorine atoms.

[0071] In at least one embodiment, R⁴ is independently naphthyl comprising one fluorine atom, two fluorine atoms, three fluorine atoms, four fluorine atoms, five fluorine atoms, six fluorine atoms, or seven fluorine atoms.

- 30 [0072] In any embodiment described herein, each of R⁴, R⁵, R⁶, and R⁷ is independently each a fluorinated hydrocarbyl group having 1 to 30 carbon atoms, more preferably each of R⁴, R⁵, R⁶, and R⁷ is independently is a fluorinated aryl (such as phenyl, biphenyl, [(C₆H₃(C₆H₅)₂)₄B], or naphthyl) group, and most preferably each of R⁴, R⁵, R⁶, and R⁷ is independently is a

perfluorinated aryl (such as bi-phenyl, $[(C_6H_3(C_6H_5)_2)_4B]$, or naphthyl) group, preferably at least one R^4 , R^5 , R^6 , and R^7 is not perfluorophenyl.

[0073] In any embodiment of this invention, when Q is a fluorophenyl group, then R^2 is not an optionally substituted C_1 - C_{20} linear alkyl group.

5 **[0074]** In at least one embodiment, an activator is an ionic ammonium borate represented by Formula (I):



wherein:

E is nitrogen or phosphorous;

10 each of R^1 , R^2 , and R^3 is independently C_1 - C_{40} linear alkyl, C_5 - C_{22} -aryl, arylalkyl where the alkyl has from 1 to 10 carbon atoms and the aryl has from 6 to 20 carbon atoms, or five-, six- or seven-membered heterocyclyl comprising at least one atom selected from N, P, O and S, wherein each of R^1 , R^2 , and R^3 is optionally substituted by halogen, $-NR'^2$, $-OR'$ or $-SiR'^3$ (where R' is independently hydrogen or C_1 - C_{20} hydrocarbyl), wherein R^2 optionally bonds with R^5 to
15 independently form a five-, six- or seven-membered ring. R^1 , R^2 , and R^3 together comprise 15 or more carbon atoms, such as 18 or more carbon atoms, such as 20 or more carbon atoms, such as 22 or more carbon atoms, such as 25 or more carbon atoms, such as 30 or more carbon atoms, such as 35 or more carbon atoms, such as 40 or more carbon atoms. In at least one embodiment, R^1 and R^2 are independently C_1 - C_{22} -alkyl, substituted C_1 - C_{22} -alkyl, unsubstituted
20 phenyl, or substituted phenyl. In at least one embodiment, each of R^1 , R^2 and R^3 is independently selected from methyl, ethyl, n-propyl, n-butyl, n-pentyl, n-hexyl, n-heptyl, n-octyl, n-nonyl, n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-butadecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, and n-icosyl.

[0075] In at least one embodiment, each of R^4 , R^5 , R^6 , and R^7 is independently aryl- or naphthyl, wherein at least one of R^4 , R^5 , R^6 , and R^7 is naphthyl substituted with from one to seven fluorine atoms. In at least one embodiment, each of R^4 , R^5 , R^6 , and R^7 is naphthyl, wherein at least one of R^4 , R^5 , R^6 , and R^7 is substituted with from one to seven fluorine atoms.

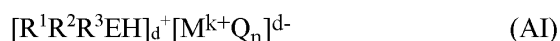
[0076] In at least one embodiment, each of R^4 , R^5 , R^6 , and R^7 is independently a naphthyl comprising one fluorine atom, two fluorine atoms, three fluorine atoms, four fluorine atoms,
30 five fluorine atoms, six fluorine atoms, or seven fluorine atoms.

[0077] In at least one embodiment, R^4 is independently naphthyl comprising one fluorine atom, two fluorine atoms, three fluorine atoms, four fluorine atoms, five fluorine atoms, six fluorine atoms, or seven fluorine atoms, and each of R^5 , R^6 , and R^7 is independently phenyl comprising one fluorine atom, two fluorine atoms, three fluorine atoms, four fluorine atoms,

or five fluorine atoms or naphthyl comprising one fluorine atom, two fluorine atoms, three fluorine atoms, four fluorine atoms, five fluorine atoms, six fluorine atoms, or seven fluorine atoms.

[0078] In at least one embodiment of the invention, the activator is represented by Formula

5 (I) or (AI):



wherein:

M is a group 13 atom, preferably B or Al;

10 d is 1, 2 or 3; k is 1, 2, or 3; n is 1, 2, 3, 4, 5, or 6 (preferably 1, 2, 3, or 4), n - k = d (preferably d is 1, 2 or 3; k is 3; n is 4, 5, or 6, preferably when M is B, n is 4);

E is nitrogen or phosphorous, preferably nitrogen;

each of R¹, R², and R³ is independently a C₁-C₄₀ linear alkyl, a C₅-C₂₂-aryl, a C₇ to C₃₀ arylalkyl (where the alkyl has from 1 to 10 carbon atoms and the aryl has from 6 to 20 carbon atoms), or

15 a five-, six- or seven-membered heterocyclyl comprising at least one atom selected from N, P, O and S, wherein each of R¹, R², and R³ is optionally substituted by halogen, wherein R² optionally bonds with R⁵ to independently form a five-, six- or seven-membered ring, preferably wherein, R¹, R², and R³ together comprise 15 or more carbon atoms, such as 18 or more carbon atoms, such as 20 or more carbon atoms, such as 22 or more carbon atoms, such as 25 or more carbon atoms, such as 30 or more carbon atoms, such as 35 or more carbon atoms, such as 38 or more carbon atoms, such as 40 or more carbon atoms. In at least one embodiment, R¹ and R² are independently C₁-C₂₂-alkyl, substituted C₁-C₂₂-alkyl, unsubstituted phenyl, or substituted phenyl (in at least one embodiment, each of R¹, R² and R³ is independently selected from methyl, ethyl, n-propyl, n-butyl, n-pentyl, n-hexyl, n-heptyl, n-octyl, n-nonyl, n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, and n-icosyl);

each of R⁴, R⁵, R⁶, and R⁷ is independently naphthyl, wherein at least one of R⁴, R⁵, R⁶, and R⁷ is naphthyl substituted with from one to seven fluorine atoms, preferably at least one R⁴, R⁵, R⁶, and R⁷ is not substituted phenyl, preferably all of R⁴, R⁵, R⁶, and R⁷ are not substituted

30 phenyl; and

each Q is independently a hydride, bridged or unbridged dialkylamido, halide, alkoxide, aryloxy, hydrocarbyl, substituted hydrocarbyl, halocarbyl, substituted halocarbyl, or halosubstituted-hydrocarbyl radical. Preferably, each Q is a fluorinated hydrocarbyl group having 1 to 30 carbon atoms, more preferably each Q is a fluorinated aryl (such as phenyl or

naphthyl) group, and most preferably each Q is a perfluorinated aryl (such as phenyl or naphthyl) group. In preferred embodiments of the invention, at least one Q is not substituted phenyl, such as perfluorophenyl, preferably all Q are not substituted phenyl, such as perfluorophenyl.

5 [0079] The terms “cocatalyst” and “activator” are used herein interchangeably and are defined to be any compound which can activate any one of the catalyst compounds of the present disclosure by converting the neutral catalyst compound to a catalytically active catalyst compound cation.

[0080] Catalyst systems of the present disclosure can be formed by combining the catalysts
10 with activators in any suitable manner, including by supporting them for use in slurry or gas phase polymerization. The catalyst systems may also be added to or generated in solution polymerization or bulk polymerization (in the monomer, i.e., little or no solvent).

[0081] Both the cation part of formulas (AI) and (I) as well as the anion part thereof, which is an NCA, will be further illustrated below. Any combinations of cations and NCAs disclosed
15 herein are suitable to be used in the processes of the present disclosure and are thus incorporated herein.

Cation Component

[0082] The cation component of the activators described herein (such as those of formulas (AI) and (I) above), is a protonated Lewis base that can be capable of protonating a moiety,
20 such as an alkyl or aryl, from the transition metal compound. Thus, upon release of a neutral leaving group (e.g. an alkane resulting from the combination of a proton donated from the cationic component of the activator and an alkyl substituent of the transition metal compound) transition metal cation results, which is the catalytically active species.

[0083] In at least one embodiment of formula (I) or (AI), where the cation is $[R^1R^2R^3EH]^+$,
25 E is nitrogen or phosphorous, and each of R^1 , R^2 , and R^3 is independently C_1 - C_{40} linear alkyl, C_5 - C_{22} -aryl, arylalkyl (where the alkyl has from 1 to 10 carbon atoms and the aryl has from 6 to 20 carbon atoms) or five-, six- or seven-membered heterocyclyl comprising at least one atom selected from N, P, O and S, wherein each of R^1 , R^2 , and R^3 is optionally substituted by halogen, $-NR'_2$, $-OR'$ or $-SiR'_3$ (where each R' is independently hydrogen or C_1 - C_{20} hydrocarbyl),
30 wherein R^2 optionally bonds with R^5 to independently form a five-, six- or seven-membered ring. R^1 , R^2 , and R^3 together comprise 15 or more carbon atoms, such as 18 or more carbon atoms, such as 20 or more carbon atoms, such as 22 or more carbon atoms, such as 25 or more carbon atoms, such as 30 or more carbon atoms, such as 35 or more carbon atoms, such as 37 or more carbon atoms, such as 40 or more carbon atoms, such as 45 or more carbon atoms. In

at least one embodiment, R^1 , R^2 , and R^3 are independently substituted or unsubstituted C_1 - C_{22} linear alkyl, or substituted or unsubstituted phenyl. In at least one embodiment, each of R^1 , R^2 and R^3 is independently selected from methyl, ethyl, n-propyl, n-butyl, n-pentyl, n-hexyl, n-heptyl, n-octyl, n-nonyl, n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, and n-icosyl. In at least one embodiment, each of R^2 and R^3 is independently selected from methylphenyl, ethylphenyl, n-propylphenyl, n-butylphenyl, n-pentylphenyl, n-hexylphenyl, n-heptylphenyl, n-octylphenyl, n-nonylphenyl, n-decylphenyl, n-undecylphenyl, n-dodecylphenyl, n-tridecylphenyl, n-tetradecylphenyl, n-pentadecylphenyl, n-hexadecylphenyl, n-heptadecylphenyl, n-octadecylphenyl, n-nonadecylphenyl, and n-icosylphenyl.

[0084] In a preferred embodiment, R^1 is methyl, R^2 is substituted phenyl, R^3 is C_{10} to C_{30} linear alkyl. Preferably R^2 is not meta substituted phenyl.

[0085] In a preferred embodiment, R^1 is methyl, R^2 is C_1 to C_{35} alkyl substituted phenyl (preferably ortho- or meta- substituted), R^3 is C_{10} to C_{30} linear alkyl.

[0086] In a preferred embodiment, R^1 is methyl, R^2 is C_1 to C_{35} alkyl substituted phenyl (preferably para substituted), R^3 is C_{10} to C_{30} linear alkyl.

[0087] In a preferred embodiment, R^1 is methyl; R^2 is C_1 to C_{35} alkyl substituted phenyl, such as methylphenyl, ethylphenyl, n-propylphenyl, n-butylphenyl, n-pentylphenyl, n-hexylphenyl, n-heptylphenyl, n-octylphenyl, n-nonylphenyl, n-decylphenyl, n-undecylphenyl, n-dodecylphenyl, n-tridecylphenyl, n-tetradecylphenyl, n-pentadecylphenyl, n-hexadecylphenyl, n-heptadecylphenyl, n-octadecylphenyl, n-nonadecylphenyl, and n-icosylphenyl, n-henicosylphenyl, n-docosylphenyl, n-tricosylphenyl, n-tetracosylphenyl, n-pentacosylphenyl, n-hexacosylphenyl, n-heptacosylphenyl, n-octacosylphenyl, n-nonacosylphenyl, n-triacontylphenyl; and R^3 is C_{10} to C_{30} linear alkyl, such as n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, n-icosyl, n-henicosyl, n-docosyl, n-tricosyl, n-tetracosyl, n-pentacosyl, n-hexacosyl, n-heptacosyl, n-octacosyl, n-nonacosyl, n-triacontyl.

[0088] In a preferred embodiment, R^2 is C_1 to C_{35} alkyl substituted phenyl, such as methylphenyl, ethylphenyl, n-propylphenyl, n-butylphenyl, n-pentylphenyl, n-hexylphenyl, n-heptylphenyl, n-octylphenyl, n-nonylphenyl, n-decylphenyl, n-undecylphenyl, n-dodecylphenyl, n-tridecylphenyl, n-tetradecylphenyl, n-pentadecylphenyl, n-hexadecylphenyl, n-heptadecylphenyl, n-octadecylphenyl, n-nonadecylphenyl, and n-icosylphenyl, n-henicosylphenyl, n-docosylphenyl, n-tricosylphenyl, n-tetracosylphenyl, n-pentacosylphenyl, n-hexacosylphenyl, n-heptacosylphenyl, n-octacosylphenyl, n-nonacosylphenyl, n-

triacontylphenyl; and R³ is C₁₀ to C₃₀ linear alkyl, such as n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, n-icosyl, n-henicosyl, n-docosyl, n-tricosyl; n-tetracosyl, n-pentacosyl; n-hexacosyl; n-heptacosyl, n-octacosyl, n-nonacosyl, n-triacontyl.

5 [0089] In a preferred embodiment of formula (I), R¹ is methyl, R² is substituted phenyl, R³ is C₁₀ to C₃₀ linear alkyl, and R⁴, R⁵, R⁶, R⁷ are perfluoronaphthyl.

[0090] In a preferred embodiment of formula (AI), R¹ is methyl, R² is substituted phenyl, R³ is C₁₀ to C₃₀ linear alkyl, E is nitrogen, and each Q is perfluoronaphthyl.

[0091] In a preferred embodiment, R¹ is methyl; R² is C₁ to C₃₅ alkyl substituted phenyl,
10 such as as methylphenyl, ethylphenyl, n-propylphenyl, n-butylphenyl, n-pentylphenyl, n-hexylphenyl, n-heptylphenyl, n-octylphenyl, n-nonylphenyl, n-decylphenyl, n-undecyl,phenyl
n-dodecylphenyl, n-tridecylphenyl, n-tetradecylphenyl, n-pentadecylphenyl, n-hexadecylphenyl, n-heptadecylphenyl, n-octadecylphenyl, n-nonadecylphenyl, and n-
icosylphenyl, n-henicosylphenyl, n-docosylphenyl, n-tricosylphenyl, n-tetracosylphenyl, n-
15 pentacosylphenyl, n-hexacosylphenyl, n-heptacosylphenyl, n-octacosylphenyl, n-nonacosylphenyl, n-triacontylphenyl; R³ is C₁₀ to C₃₀ linear alkyl, such as n-decyl, n-undecyl,
n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, n-icosyl, n-henicosyl, n-docosyl, n-tricosyl; n-tetracosyl, n-pentacosyl; n-hexacosyl; n-heptacosyl, n-octacosyl, n-nonacosyl, n-triacontyl; and each Q or each of R⁴, R⁵,
20 R⁶, R⁷ are perfluoronaphthyl.

[0092] In a preferred embodiment of the invention, R¹ is o-MePh, R² and R³ are n-octadecyl.

[0093] In a preferred embodiment of the invention, R¹ is m-MePh, R² and R³ are n-octadecyl.

[0094] In a preferred embodiment of the invention, R¹ is not para-alkylphenyl, such as
25 p-MePh.

[0095] In a preferred embodiment of the invention, R¹ is Me, R² is n-octadecylaryl, and R³ is n-octadecyl.

[0096] In a preferred embodiment of the invention, R¹ is Me, R² is n-octadecylphenyl, and R³ is n-octadecyl.

30 [0097] In a preferred embodiment of the invention, R¹ is Me, R² is n-butylaryl, and R³ is n-octadecyl.

[0098] In a preferred embodiment of the invention, R¹ is Me, R² is n-butylphenyl, and R³ is n-octadecyl.

[0099] In a preferred embodiment of the invention, R¹ is n-decyl, R² is n-butylaryl, and R³ is n-decyl.

[0100] In a preferred embodiment of the invention, R¹ is n-decyl, R² is n-butylphenyl, and R³ is n-decyl.

5 [0101] In a preferred embodiment of the invention, R¹ is n-propyl, R² is p-methylphenyl, and R³ is n-octadecyl.

[0102] In a preferred embodiment of the invention, R¹, R² and R³ together comprise 20 or more carbon atoms, such as 21 or more carbon atoms, such as 22 or more carbon atoms, such as 25 or more carbon atoms, such as 30 or more carbon atoms, such as 35 or more carbon atoms, such as 37 or more carbon atoms, such as 40 or more carbon atoms, such as 45 or more carbon atoms, such as 15 to 100 carbon atoms, such as 25 to 75 carbon atoms, such as 38 to 70 carbon atoms.

[0103] In at least one embodiment, the cation is selected from the group consisting of:

1		2		3	
4		5		6	
7		8		9	
10		11		12	
13		14		15	

16		17		18	
19		20		21	
22		23		24	
25		26		27	
29		28		30	
31		32		33	

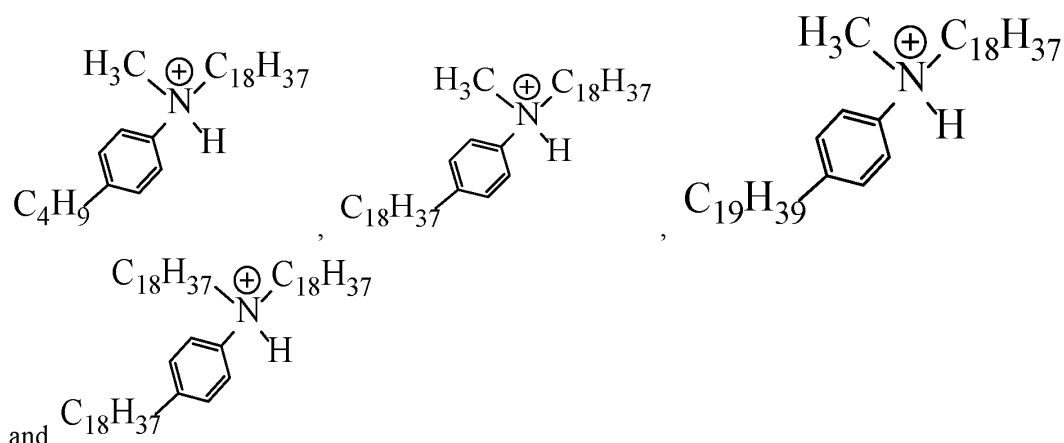
[0104] In at least one embodiment of formulas (AI) and (I), E is nitrogen or phosphorous, R¹ is a methyl group; R² is C₆-C₄₀ aryl (such as substituted phenyl) and R³ is independently C₁-C₃₅ linear alkyl, C₅-C₄₀-aryl, wherein each of R² and R³ is independently unsubstituted or substituted with at least one of C₁-C₃₅ alkyl, C₅-C₃₀ aryl, C₆-C₃₀₅ arylalkyl, C₆-C₃₀ alkylaryl, halogen, wherein R² optionally bonds with R³ to independently form a five-, six- or seven-membered ring, wherein R², and R³ together comprise 20 or more carbon atoms; and optionally R¹, R², and R³ together comprise 21 or more carbon atoms, such as 22 or more carbon atoms, such as 25 or more carbon atoms, such as 30 or more carbon atoms, such as 35 or more carbon atoms, such as 40 or more carbon atoms. In at least one embodiment, R² is independently substituted C₁-C₂₂-alkyl, unsubstituted phenyl, or substituted phenyl. In at least one embodiment, R³ is independently selected from methyl, ethyl, n-propyl, n-butyl, n-pentyl, n-

hexyl, n-heptyl, n-octyl, n-nonyl, n-decyl, n-undecyl, n-dodecyl, n-tridecyl, n-tetradecyl, n-pentadecyl, n-hexadecyl, n-heptadecyl, n-octadecyl, n-nonadecyl, and n-icosyl.

[0105] Preferably, the cation is selected from the group consisting of:

16		17		18	
19		20		21	
22		23		24	
25		26		27	
29		28		30	
31		32		33	

- 5 **[0106]** Preferably the compound represented by formulas (AI) and (I) comprises a cation selected from the group consisting of:



Anion Component

- 5 **[0107]** The anion component of the activators described herein includes those represented by the formula $[\text{M}^{k+}\text{Q}_n]^-$ wherein k is 1, 2, or 3; n is 1, 2, 3, 4, 5, or 6 (preferably 1, 2, 3, or 4), (preferably k is 3; n is 4, 5, or 6, preferably when M is B, n is 4); M is an element selected from Group 13 of the Periodic Table of the Elements, preferably boron or aluminum, and Q is independently a hydride, bridged or unbridged dialkylamido, halide, alkoxide, aryloxy,
- 10 hydrocarbyl, substituted hydrocarbyl, halocarbyl, substituted halocarbyl, and halosubstituted-hydrocarbyl radicals, said Q having up to 20 carbon atoms with the proviso that in not more than 1 occurrence is Q a halide. Preferably, each Q is a fluorinated hydrocarbyl group, optionally having 1 to 20 carbon atoms, more preferably each Q is a fluorinated aryl group, and most preferably each Q is a perfluorinated aryl group. Preferably at least one Q is not
- 15 substituted phenyl, such as perfluorophenyl, preferably all Q are not substituted phenyl, such as perfluorophenyl.

[0108] In a preferred embodiment of any embodiment of Formula (AI), when R^1 is methyl, R^2 is C18 and R^3 is C18, then each Q is not perfluorophenyl.

- [0109]** In at least one embodiment, for the borate moiety ($[\text{BR}^4\text{R}^5\text{R}^6\text{R}^7]^-$) of the activator represented by formula (I), each of R^4 , R^5 , R^6 , and R^7 is independently aryl (such as naphthyl), wherein at least one of R^4 , R^5 , R^6 , and R^7 is substituted with from one to seven fluorine atoms. In at least one embodiment, each of R^4 , R^5 , R^6 , and R^7 is naphthyl, wherein at least one of R^4 , R^5 , R^6 , and R^7 is substituted with from one to seven fluorine atoms.

- [0110]** In at least one embodiment, each of R^4 , R^5 , R^6 , and R^7 is independently naphthyl comprising one fluorine atom, two fluorine atoms, three fluorine atoms, four fluorine atoms,
- 25 five fluorine atoms, six fluorine atoms, or seven fluorine atoms.

[0111] In a preferred embodiment of any embodiment of Formula (I), when R¹ is methyl, R² is C₁₈ and R³ is C₁₈, then each of R⁴, R⁵, R⁶, and R⁷ is not perfluorophenyl.

[0112] In at least one embodiment, R⁴ is independently naphthyl comprising one fluorine atom, two fluorine atoms, three fluorine atoms, four fluorine atoms, five fluorine atoms, six fluorine atoms, or seven fluorine atoms, and each of R⁵, R⁶, and R⁷ is independently phenyl comprising one fluorine atom, two fluorine atoms, three fluorine atoms, four fluorine atoms, or five fluorine atoms or naphthyl comprising one fluorine atom, two fluorine atoms, three fluorine atoms, four fluorine atoms, five fluorine atoms, six fluorine atoms, or seven fluorine atoms.

[0113] In one embodiment, the borate activator comprises tetrakis(heptafluoronaphth-2-yl)borate.

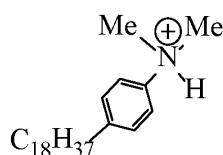
[0114] In at least one preferred embodiment, the activator is represented by Formula (B):



where M is an element selected from group 13 of the Periodic Table of the Elements; each Q is independently a hydride, bridged or unbridged dialkylamido, halide, alkoxide, aryloxy, hydrocarbyl, substituted hydrocarbyl, halocarbyl, substituted halocarbyl, or halosubstituted hydrocarbyl radical; and the [cation]⁺ is selected from the group consisting of:

1		2		3	
4		5		6	
7		8		9	

[0115] In at least one preferred embodiment, [MQ₄]⁻ is perfluoroaryl. In at least one other preferred embodiment, the [cation]⁺ is



[0116] The activators can be added to a polymerization in the form of an ion pair using, for example, [M2HTH]⁺ [NCA]⁻ in which the di(hydrogenated tallow)methylamine (“M2HTH”) cation reacts with a basic leaving group on the transition metal complex to form a transition metal complex cation and [NCA]⁻. Alternatively, the transition metal complex can be reacted with a neutral NCA precursor, such as B(C₁₀F₇)₃, which abstracts an anionic group from the complex to form an activated species. Useful activators include di(hydrogenated tallow)methylamine(perfluoronaphthyl)borate (i.e., [M2HTH]B(C₁₀F₇)₄) and di(octadecyl)tolylamine (perfluoronaphthyl)borate (i.e., [DOdTH]B(C₁₀C₇)₄).

[0117] In at least one embodiment, the activators obtained in their salt form used for a borate activator compound are: Lithium tetrakis(heptafluoronaphthalen-2-yl)borate etherate (Li-BF28), N,N-Dimethylanilinium tetrakis(heptafluoronaphthalen-2-yl)borate (DMAH-BF28), Sodium tetrakis(heptafluoronaphthalen-2-yl)borate (Na-BF28) and N,N-dimethylanilinium tetrakis(heptafluoronaphthalen-2-yl)borate (DMAH-BF28).

[0118] In at least one embodiment, an activator of the present disclosure, when combined with a group 4 metallocene catalyst compound to form an active olefin polymerization catalyst, produces a higher molecular weight polymer (e.g., Mw) than comparative activators that use other borate anions.

[0119] In at least one embodiment, an activator of the present disclosure where R¹ is methyl, when combined with a group 4 metallocene to form an active olefin polymerization catalyst, produces a higher molecular weight polymer (e.g., Mw) than comparative activators that use other borate anions.

[0120] The typical activator-to-catalyst ratio, e.g., all NCA activators-to-catalyst ratio is about a 1:1 molar ratio. Alternate preferred ranges include from 0.1:1 to 100:1, alternately from 0.5:1 to 200:1, alternately from 1:1 to 500:1 alternately from 1:1 to 1000:1. A particularly useful range is from 0.5:1 to 10:1, preferably 1:1 to 5:1. The molar ratio of activator to catalyst can also vary from 0.5 to 2.0.

[0121] It is also within the scope of the present disclosure that the catalyst compounds can be combined with combinations of alumoxanes and the activators described herein.

Synthesis

[0122] In at least one embodiment, the general synthesis of the activators can be performed using a two-step process. In the first step, an amine or phosphine is dissolved in a solvent (e.g. hexane, cyclohexane, methylcyclohexane, ether, dichloromethane, toluene) and an excess (e.g., 1.2 molar equivalents) of hydrogen chloride is added to form a chloride salt. This salt is typically isolated by filtration from the reaction medium and dried under reduced pressure. The isolated chloride is then heated to reflux with about one molar equivalent of an alkali metal metallate or metalloid (such as a borate or aluminate) in a solvent (e.g. cyclohexane, dichloromethane, methylcyclohexane) to form the desired borate or aluminate along with byproduct alkali metal chloride, the latter of which can typically be removed by filtration.

[0123] The activators of the present disclosure are soluble in aliphatic solvent. Aromatic solvents, such as toluene, are absent (e.g. present at zero mol%, alternately present at less than 1 mol%, preferably the catalyst system, the polymerization reaction and/or the polymer produced are free of “detectable aromatic hydrocarbon solvent,” such as toluene. For purposes of the present disclosure, “detectable aromatic hydrocarbon solvent” means 0.1 mg/m² or more as determined by gas phase chromatography. For purposes of the present disclosure, “detectable toluene” means 0.1 mg/m² or more as determined by gas phase chromatography. The polyolefins produced herein preferably contain 0 ppm (alternately less than 1 ppm) of aromatic hydrocarbon. Preferably, the polyolefins produced herein contain 0 ppm (alternately less than 1 ppm) of toluene. The catalyst systems used herein preferably contain 0 ppm (alternately less than 1 ppm) of aromatic hydrocarbon. Preferably, the catalyst systems used herein contain 0 ppm (alternately less than 1 ppm) of toluene.

[0124] In at least one embodiment, the general synthesis of the activators can be performed using a two-step process. In the first step, an amine is dissolved in a solvent. In one or more embodiments, a 20 wt% mixture of the compound in n-hexane, isohexane, cyclohexane, methylcyclohexane, or a combination thereof, forms a clear homogeneous solution at 25°C, preferably a 30 wt% mixture of the compound in n-hexane, isohexane, cyclohexane, methylcyclohexane, or a combination thereof, forms a clear homogeneous solution at 25°C.

[0125] The activators can have a solubility of more than 10 mM (or more than 20 mM, or more than 50 mM) at 25°C (stirred 2 hours) in methylcyclohexane. The activators can have a solubility of more than 1 mM (or more than 10 mM, or more than 20 mM) at 25°C (stirred 2 hours) in isohexane. The activators can have a solubility of more than 10 mM (or more than 20 mM, or more than 50 mM) at 25°C (stirred 2 hours) in methylcyclohexane and a solubility of more than 1 mM (or more than 10 mM, or more than 20 mM) at 25°C (stirred 2 hours) in isohexane.

[0126] After the activator is added to the solvent, an excess (e.g., 1.2 molar equivalents) of hydrogen chloride is added to form an ammonium chloride salt. This salt is typically isolated by filtration from the reaction medium and dried under reduced pressure. The isolated ammonium chloride is then heated to reflux with about one molar equivalent of an alkali metal borate in a solvent (e.g. cyclohexane, dichloromethane, methylcyclohexane) to form the ammonium borate along with byproduct alkali metal chloride, the latter of which can typically be removed by filtration.

[0127] In at least one embodiment, an activator of the present disclosure is soluble in an aliphatic solvent at a concentration of about 10 mM or greater, such as about 20 mM or greater, such as about 30 mM or greater, such as about 50 mM or greater, such as about 75 mM or greater, such as about 100 mM or greater, such as about 200 mM or greater, such as about 300 mM or greater. In at least one embodiment, an activator of the present disclosure dissolves in isohexane or methylcyclohexane at 25°C to form a homogeneous solution of at least 10 mM concentration.

[0128] In at least one embodiment, the solubility of the borate or aluminate activators of the present disclosure in aliphatic hydrocarbon solvents increases with the number of aliphatic carbons in the cation group (i.e., the ammonium or the phosphonium). In at least one embodiment, a solubility of at least 10 mM is achieved with an activator having an ammonium or phosphonium group of about 21 aliphatic carbon atoms or more, such as about 25 aliphatic carbons atoms or more, such as about 35 carbon atoms or more.

[0129] In at least one embodiment, the solubility of the ammonium borate activators of the present disclosure in aliphatic hydrocarbon solvents increases with the number of aliphatic carbons in the ammonium group. In at least one embodiment, a solubility of at least 10 mM is achieved with an activator having an ammonium group of about 21 aliphatic carbon atoms or more, such as about 25 aliphatic carbons atoms or more, such as about 35 carbon atoms or more.

[0130] Useful aliphatic hydrocarbon solvent can be isobutane, butane, pentane, isopentane, hexanes, isohexane, heptane, octane, dodecane, and mixtures thereof; cyclic and alicyclic hydrocarbons, such as cyclohexane, cycloheptane, methylcyclohexane, methylcycloheptane, and mixtures thereof. In at least one embodiment, aromatics are present in the solvent at less than 1 wt%, such as less than 0.5 wt%, such as at 0 wt% based upon the weight of the solvents. The activators of the present disclosure can be dissolved in one or more additional solvents. Additional solvent includes ethereal, halogenated and *N,N*-dimethylformamide solvents. The aliphatic solvent is preferably isohexane and/or methylcyclohexane.

Optional Scavengers or Co-Activators

[0131] In addition to these activator, scavengers or co-activators can be used. Aluminum alkyl or organoaluminum compounds which can be utilized as scavengers or co-activators include, for example, trimethylaluminum, triethylaluminum, triisobutylaluminum, tri-n-hexylaluminum, tri-n-octylaluminum (TNOA), and diethyl zinc.

[0132] In at least one embodiment, little or no (i.e zero mol%) scavenger is used in the process to produce the ethylene polymer. In at least one embodiment, one or more scavengers are added to the process at a molar ratio of scavenger metal to transition metal of less than 100:1, such as less than 50:1, such as less than 15:1, such as less than 10:1.

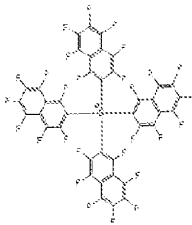
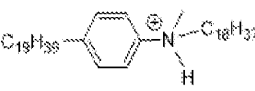
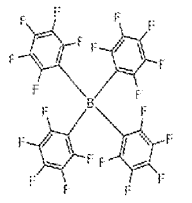
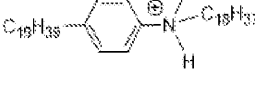
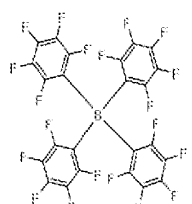
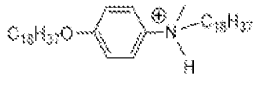
[0133] Additional details of making suitable catalyst system using the bridged metallocene(s), activator(s) and support material(s) can be found in USP 11,011,031 and 11,414,436.

Examples:

[0134] The foregoing discussion can be further described with reference to the following non-limiting examples. Five (Resins 1-5) ethylene-butene copolymers examples (C2/C4) were prepared in a pilot sized solution reactor. The catalyst was di(para-triethylsilylphenyl) methylene (2,7-di-tertbutyl-fluorenyl) (cyclopentadienyl) hafnium dimethyl, and the activator was N-methyl-4-nonadecyl-N-octadecylanilinium dimethylaniliniumtetrakis (heptafluoro naphthyl) borate as the activator/co-catalyst, both available from ExxonMobil Chemical Company.

[0135] Two ethylene-butene copolymers (C2/C4) comparative examples were also prepared. The comparative resin samples (CE1 and CE2) were produced in a pilot sized solution reactor using the same catalyst as Resins 1-5 (i.e. di(para-triethylsilylphenyl) methylene (2,7-di-tertbutyl-fluorenyl) (cyclopentadienyl) hafnium dimethyl), but activated with a different activator, as shown below in Table 1.

[0136] Table 1 shows the activators used to make the inventive resins 1-5 and the comparative resin samples CE1 and CE2.

Activator	Formula Weight (FW) (g/mol)	Structure Anion	Structure Cation
Activator 1: N-methyl-4-nonadecyl-N-octadecylanilinium dimethylaniliniumtetrakis (heptafluoro naphthyl) borate	1650.4		
Activator 2: N-methyl-4-nonadecyl-N-octadecylanilinium tetrakis pentafluorophenyl) borate	1306.2		
Activator 3: N-methyl-N-octadecyl-4-(octadecyloxy) anilinium tetrakis(pentafluorophenyl) borate	1308.2		

[0137] The activators were diluted with aliphatic hydrocarbons to make activator solutions before being fed to pilot sized solution polymerization reactors. The activator solution and the catalyst solution were separately feed to the reactor and mixed within the reactor to make activated catalyst for polymerization. Tri-n-octylaluminum (TNOA), a scavenger, was fed as a diluted solution of 3 wt% neat TNOA and 97 wt% process solvent. This solution was metered by a mass flowmeter and continuously fed to the reactor after mixed with the rest of the monomers. Table 2 summarizes the copolymer properties, aluminum residue and volume resistivity, and Table 3 summarizes key process conditions.

[0138] Table 2: Copolymer properties for Resins 1-5 and comparative examples CE1, CE2.

	s1	s2	s3	s4	s5	CE1	CE2
Comonomer	butene	butene	butene	butene	butene	butene	butene
C2%	76.2	76.5	74.4	74.0	69.0	76.9	74.3
Density	0.878	0.877	0.872	0.873	0.862	0.875	0.875
MI	14.9	17.7	13.2	16.1	1.5	3.8	3.4
Activator	1	1	1	1	1	2	3
Catalyst / Activator molar ratio	1.0	1.0	1.0	1.0	1.0	1.0	1.0
Catalyst efficiency (kg/g)	83	80	127	151	400		
Al residue (wppm)	1.80		0.20	0.20			
g'avg		0.94		0.931		0.99	0.99
g'Mz		0.93		0.91		0.98	0.979
g'Mz+1		0.89		0.88		0.96	0.974
g'Mz+1 / g'avg		0.95		0.94		0.97	0.98
r1r2				0.31			
Volume resistivity (Ωcm)	5.9E+15		7.3E+15	5.3E+15		4.1E+14	4.8E+14

[0139] Table 3: Key process conditions

	s1	s2	s3	s4	s5	CE1	CE2
Catalyst Flow (cc/min)	4.3	4.4	2.8	2.4	0.6	1.10	0.90
Activator Flow (cc/min)	4.3	4.4	2.8	2.4	0.6	1.5	1.2
TNOA flow (g/hr)	0.30	0.30	0.06	0.06	0.30	0.00	0.00
Reactor Inlet Temp. (°C)	25	25	25	25	18	35	35
Reactor Outlet Temp. (°C)	145	145	145	145	130	136	136
C6 Feed (kg/hr)	76	76	74	74	64.3	24	24
C4 Feed (kg/hr)	4.4	4.4	5.1	5.0	6	2.1	2.1
C2 Feed (kg/hr)	9.3	9.3	9.2	9.2	7.6	4.8	4.8
H2 Feed (sccm)	163	160	62	80	245	164	164
Cement Conc. (wt%)	10.3%	10.3%	10.3%	11.0%	10.9%	19	19
H2/C2 Feed (wppm)	94.9	93.1	36.5	47.1	17	170	170
C2 Conversion (%)	85	85	85	85	67	92	90

[0140] Table 4 provides similar information for comparative commercially available copolymers.

	Dow Engage PV8660	Dow Engage PV8669	LG LC575	LG LC675
Comonomer	Octene	Octene	Butene	Butene
Density	0.872	0.873	0.877	0.877
MI	4.8	14.0	5.0	14.0
Al residue (wppm) tested by ICP-OES	4.09	4.32	37.24	35.37
g'avg	0.93	0.93	0.97	0.96
g'Mz	0.94	0.94	0.97	0.96
g'Mz+1	0.95	0.97	0.98	0.96
g'Mz+1 / g'avg	1.03	1.04	1.01	1.00
rI _{r2}	1.4	1.0	1.4	1.2
Volume resistivity (Ωcm)	4.29E+17	3.33E+17	3.03E+17	1.14E+17

5

[0141] The copolymers were formed into molded plaques and tested for volume resistivity (1 mm thick film, 500 V, 5 minutes charging time) and aluminum residue. The Figure shows the volume resistivity of the molded plaques plotted against aluminum residue (ppmw). Resins 1-5 all had volume resistivities >10¹⁵ Ohm*cm with less than 2.0 ppmw aluminum residue, and several at or less than 0.1 ppm, even though all the copolymers had similar densities, MI, and branching indexes.

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Test Procedures

[0142] In the foregoing Examples, the following test methods and procedures were used:

[0143] Densities were measured according to ASTM D792, and the MI values were measured according to ASTM D1238 (190°C/2.16 kg).

15

[0144] The distribution and the moments of molecular weight (M_w, M_n, M_z, M_w/M_n, M_z/M_n, etc.), and the long chain branching indices (g') were determined using a high temperature Gel Permeation Chromatography (Polymer Char GPC-IR) equipped with a multiple-channel band-filter based Infrared detector IR5, an 18-angle light scattering detector and a viscometer. Three Agilent Plgel 10μm Mixed-B LS columns are used to provide polymer separation. Detailed analytical principles and methods for molecular weight determinations are described in paragraphs [0044] – [0051] of International Publication No. WO/2019/246069A1, which is herein incorporated herein by reference (noting that the equation for c referenced in Paragraph [0044] therein for concentration I at each point in the

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chromatogram, is $c = \beta I$, where β is mass constant and I is the baseline-subtracted IR5 broadband signal intensity (I). Unless specifically mentioned, all the molecular weight moments used or mentioned in the present disclosure are determined according to the conventional molecular weight (IR molecular weight) determination methods (e.g., as
5 referenced in Paragraphs [0044] – [0045] of the just-noted publication), noting that for the equation in such Paragraph [0044], $a = 0.695$ and $K = 0.000579(1-0.75Wt)$ are used, where Wt is the weight fraction for hexane comonomer, and further noting that comonomer composition is determined by the ratio of the IR5 detector intensity corresponding to CH2 and CH3 channel calibrated with a series of PE and PP homo/copolymer standards whose nominal values are
10 predetermined by NMR or FTIR (providing methyls per 1000 total carbons (CH3/1000 TC)) as noted in Paragraph [0045] of the just-noted International Publication).

[0145] The long chain branching indices (g') was measured using GPC-4D. A typical GPC-4D profile has Log M vs. g' and is used to estimate a g' average, based on the average across the molecular weight. The branching index g' avg values range from 1 to 0 with 1 being linear
15 (no branching) and 0 being fully branched. The g' avg does not clearly differentiate branching changes at low levels (0.85 to 1), so $g'(M_z)$ and $g'(M_z+1)$ were estimated at higher molecular weight moments (M_z , M_z+1). The branching index $g'(M_z)$ is the g' from GPC-4D profiles estimated at the z-average (third moment) molecular weight average. This calculation is performed by curve fitting the g' vs molecular weight data to a nth order polynomial using the
20 MATLAB program. The value of n is typically between 3 and 4. The M_z value obtained from GPC-IR measurements are inserted into the curve fit to calculate the g' associated with that molecular weight.

¹³C NMR for r1r2.

[0146] Samples were dissolved in deuterated 1,1,2,2-tetrachloroethane-d2 (tce-d2) at a
25 concentration of 67mg/mL at 140°C. Spectra were recorded at 120°C using a Bruker NMR spectrometer of at least 600MHz with a 10mm cryoprobe. A 90° pulse, 10s delay, 512 transients, and gated decoupling were used for measuring the ¹³C NMR. Polymer resonance peaks are referenced to Polyethylene main peak at 29.98 ppm.

[0147] Chemical shift assignments for the ethylene-octene copolymers are described by
30 Randall in “A Review Of High Resolution Liquid Carbon Nuclear Magnetic Resonance Characterization of Ethylene-Based Polymers”, Polymer Reviews, 29:2,201-5 317 (1989). The copolymer content, mole and weight %, triad sequencing, and diad calculations are also calculated and described in the method established by Randall in this paper. Calculations for

reactivity ratio ($r_1 r_2$) were based on the equation $r_1 r_2 = 4[EE][OO]/[EO]^2$; where $[EE]$, $[EO]$, $[OO]$ are the diad molar concentrations; E is ethylene, O is octene.

[0148] The reactivity ratio of product $r_1 r_2$ is described more fully in Textbook of Polymer Chemistry, F. W. Billmeyer, Jr., Interscience Publishers, New York, p.221 et seq. (1957). The reactivity ratio product $r_1 r_2$, where r_1 is the reactivity of ethylene and r_2 is the reactivity of propylene, can be calculated from the measured diad distribution (OO, EE, EO and OE in this nomenclature) by the application of the following formulae:

$$r_1 r_2 = 4(EE)(OO)/(EO)^2$$

$$r_1 = K_{11} / K_{12} = [2(EE)/EO]X$$

$$r_2 = K_{22} / K_{21} = [2(OO)/(EO)]X$$

$$O = (OO) + (EO/2)$$

$$E = (EE) + (EO/2)$$

where Mol % $E = [(E)/(E+O)] \times 100$ and $X = E/O$ in reactor;

K_{11} and K_{12} are kinetic insertion constants for ethylene; and

K_{21} and K_{22} are kinetic insertion constants for propylene.

[0149] As is known to those skilled in the art, a reactivity ratio product $r_1 r_2$ of zero (0) can define an “alternating” copolymer, and a reactivity ratio product of one (1) is said to define a “statistically random” copolymer. In other words, a copolymer having a reactivity ratio product $r_1 r_2$ of between 0.6 and 1.5 is generally said to be random (in strict theoretical terms, generally only a copolymer having a reactivity ratio product $r_1 r_2$ greater than 1.5 contains relatively long homopolymer sequences and is said to be “blocky”).

[0150] Aluminum residue in polymer was tested by ICP-OES with burning-ashing sample preparation method. Accurately weigh ± 20 g of sample on an analytical balance in a clean platina crucible. Place the crucibles into the muffle oven and start the temperature program from room temperature to 550°C for 5 hours. An empty clean crucible was placed in the oven with each batch of samples as blank reference. Let the muffle oven cool down close to room temperature, transfer the crucibles into the acid hood. Add 1 ml of nitric acid and carefully swirl the crucible in such way that the nitric acid is wetting all ashes. Add 0.5 ml hydrofluoric acid and carefully swirl the crucible again. Let react for at least 30 minutes. If necessary, add another 0.5 ml of hydrofluoric acid to bring all ash in solution. Add ± 5 ml milli-Q water and 1 ml hydrochloric acid and carefully swirl the crucible. With the help of a clean disposable plastic pipette, quantitatively transfer the mixture into a clean 50 ml volumetric PFA flask. Rinse the crucible at least 3 times with milli-Q water, transfer quantitatively into the flask each time. Add milli-Q water up to the mark. Using multi-element standards set up a calibration line

prior to measuring the samples, and then use ICP-OES to test element. Aluminum residue as weight ppm was reported.

[0151] Volume resistivity (VR) was tested according to an ExxonMobil method, which is based on ASTM D257. The measurement is done with a Keithley 6517B electrometer and
5 keithley 8009 test fixture. The leakage current is directly tested with the instrument and volume resistivity is calculated with following equation,

$$\rho = \frac{VA}{It}$$

where ρ is volume resistivity ($\Omega \cdot \text{cm}$), V is the applied voltage (volts), A is electrode contact area (cm^2), I is the leakage current (amps) and t is the average thickness of the samples. The
10 volume resistivity test was conducted at 500 Volts & 300s charging time at room temperature, three 1mm thickness molded plaques were tested to get the average for each sample.

[0152] Certain embodiments and features have been described using a set of numerical upper limits and a set of numerical lower limits. It should be appreciated that ranges including the combination of any two values, e.g., the combination of any lower value with any upper value,
15 the combination of any two lower values, and/or the combination of any two upper values are contemplated unless otherwise indicated. Certain lower limits, upper limits and ranges appear in one or more claims below. All numerical values are "about" or "approximately" the indicated value, and take into account experimental error and variations that would be expected by a person having ordinary skill in the art.

[0153] Various terms have been defined above. To the extent a term used in a claim is not defined above, it should be given the broadest definition persons in the pertinent art have given that term as reflected in at least one printed publication or issued patent. Furthermore, all patents, test procedures, and other documents cited in this application are fully incorporated by reference to the extent such disclosure is not inconsistent with this application and for all
20 jurisdictions in which such incorporation is permitted.

[0154] While the foregoing is directed to embodiments of the present invention, other and further embodiments of the invention may be devised without departing from the basic scope thereof, and the scope thereof is determined by the claims that follow.

CLAIMS:

What is claimed is:

1. An ethylene copolymer, comprising:
at least 50 wt% ethylene derived units; and
5 at least 20 wt% of at least one C3 to C20 comonomer, wherein the copolymer has:
a melt index of 0.5 g/10 min to about 50 g/10 min, as measured according to
ASTM D1238 (190°C/2.16 kg);
density of about 0.856 g/cc to 0.890 g/cc, as measured according to ASTM
D792;
10 volume resistivity at 23°C of at least $5 \times 10^{15} \Omega \text{cm}$; and
0.01 to 4.0 ppm by weight of aluminum.
2. The ethylene copolymer of claim 1, further comprising a first long chain branching
index ($g'(M_z)$) of 0.80 to 0.93.
- 15 3. The ethylene copolymer of claim 1, further comprising a second long chain branching
index ($g'(M_{z+1})$) of 0.80 to 0.93.
4. The ethylene copolymer of claim 1, further comprising an average ratio of $g'M_{z+1}$ to
20 $g'(M_z)$ of 0.9 to 1.0.
5. The ethylene copolymer of claim 1, further comprising less than 0.7 vinyl/total
unsaturation and an unsaturation level of trisubstituted olefins of 50 to 500.
- 25 6. The ethylene copolymer of claim 1, further comprising a r_1r_2 reactivity ratio of 0.2 to
0.8.
7. The ethylene copolymer of claim 1, wherein the at least one C3 to C20 comonomer is
butene or octene or a combination thereof.
- 30 8. An electronic device module comprising:
at least one electronic device, and
an ethylene copolymer film in direct contact with at least one surface of the electronic
device, the ethylene copolymer comprising:

at least 50 wt% ethylene derived units; and
at least 20 wt% of at least one C3 to C20 comonomer, wherein the copolymer
has:

a melt index of 0.5 g/10 min to about 50 g/10 min, as measured
according to ASTM D1238 (190°C/2.16 kg);

density of about 0.856 g/cc to 0.890g/cc, as measured according to
ASTM D792;

volume resistivity at 23°C of at least $5 \times 10^{15} \Omega\text{cm}$; and
0.01 to 4.0 ppm by weight of aluminum.

9. The electronic device module of claim 8, wherein the copolymer further comprises a
first long chain branching index ($g'(M_z)$) of 0.80 to 0.93.

10. The electronic device module of claim 8, wherein the copolymer further comprises a
second long chain branching index ($g'(M_{z+1})$) of 0.80 to 0.93.

11. The electronic device module of claim 8, wherein the copolymer further comprises an
average ratio of $g'M_{z+1}$ to $g'(M_z)$ of 0.9 to 1.0.

12. The electronic device module of claim 8, wherein the copolymer further comprises less
than 0.7 vinyl/total unsaturation and an unsaturation level of trisubstituted olefins of 50 to 500.

13. The electronic device module of claim 8, wherein the copolymer further comprises a
 r_1r_2 reactivity ratio of 0.2 to 0.8.

14. The electronic device module of claim 8, wherein the at least one C3 to C20
comonomer is butene or octene or a combination thereof.

15. A method for making an electronic device module comprising:
providing at least one electronic device, and
laminating an ethylene copolymer film onto at least one surface of the electronic
device, the ethylene copolymer comprising:
at least 50 wt% ethylene derived units; and
at least 20 wt% of at least one C3 to C20 comonomer, wherein the copolymer has:

a melt index of 0.5 g/10 min to about 50 g/10 min, as measured according to ASTM D1238 (190°C/2.16 kg);

a density of about 0.856 g/cc to 0.890 g/cc, as measured according to ASTM D792;

a volume resistivity at 23°C of at least $5 \times 10^{15} \Omega \text{cm}$; and

5 0.01 to 4.0 ppm by weight of aluminum.

16. The electronic device module of claim 15, wherein the copolymer further comprises a first long chain branching index ($g'(M_z)$) of 0.80 to 0.93.

10 17. The electronic device module of claim 15, wherein the copolymer further comprises a second long chain branching index ($g'(M_{z+1})$) of 0.80 to 0.93.

18. The electronic device module of claim 15, wherein the copolymer further comprises an average ratio of $g'M_{z+1}$ to $g'(M_z)$ of 0.9 to 1.0.

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19. The electronic device module of claim 15, wherein the copolymer further comprises less than 0.7 vinyl/total unsaturation and an unsaturation level of trisubstituted olefins of 50 to 500.

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20. The electronic device module of claim 15, wherein the copolymer further comprises a r_1r_2 reactivity ratio of 0.2 to 0.8.

21. The electronic device module of claim 15, wherein the C3 to C20 comonomer is butene or octene or a combination thereof.

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22. A method for making an ethylene based copolymer, comprising polymerizing ethylene derived units and at least one C3 to C20 comonomer in the presence of a catalyst system, and obtaining an ethylene based copolymer polyolefin comprising at least 50 wt% ethylene derived units; and at least 20 wt% of at least one C3 to C20 comonomer, wherein the copolymer has:

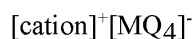
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a melt index of 0.5 g/10 min to about 50 g/10 min, as measured according to ASTM D1238 (190°C/2.16 kg);

a density of about 0.857 g/cc to 0.890 g/cc, as measured according to ASTM D792;

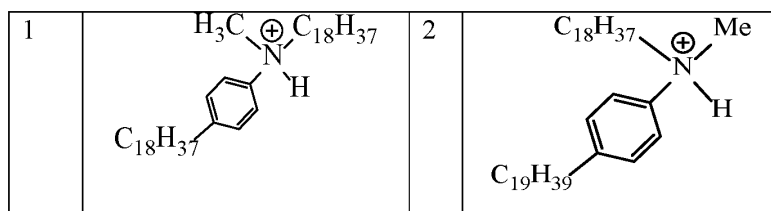
a volume resistivity at 23°C of at least $5 \times 10^{15} \Omega \text{cm}$; and
0.01 to 4.0 ppm by weight of aluminum.

23. The method of claim 22, wherein the catalyst system comprises at least one bridged
5 metallocene and at least one ionic ammonium borate or phosphonium borate activator
represented by

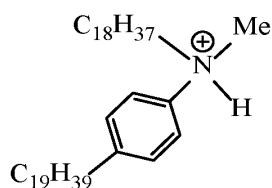


wherein the $[\text{MQ}_4]^-$ is tetrakis (heptafluoro naphthyl) borate and the $[\text{cation}]^+$ is selected from
the group consisting of:

10



24. The method of claim 22, wherein the $[\text{cation}]^+$ is



- 15 25. The method of claim 22, wherein the at least one C3 to C20 comonomer is butene or
octene or a combination thereof