OCH_2R^6 is an alkoxide radical composed of at least 3 and a maximum of 40 C atoms having a branch in the 2-position, relative to the O function; i.e., $-R^6$ $-CHR^8R^9$ where R^8R^9 independently stand for alkyl radicals $C_1 - C_{18}$; R^7 is an alkyl radical containing 2-15 C atoms which is either linear or has a branch at the 3- or higher position (relative to the O function); and the sum of x and y is a number between 0.01 and 0.8, preferably between 0.02 and 0.3, and especially preferably between 0.03 and 0.2.

The product solutions prepared by this method have relatively high concentrations of alkaline earth alkoxide compounds (i.e., cMg > 0.5 mol/kg), and yet the viscosities, at typically > 1,000 cP, remain unsatisfactorily high (see also the comparative example in the present document).

A simple method is sought which, starting with an inexpensive source of magnesium and with a high space-time yield, results in less viscous, concentrated (i.e., ≥ 0.5, preferably ≥ 1.0 mol/kg) solutions of a magnesium alcoholate in aprotic solvents, preferably aliphatic or aromatic hydrocarbons. A further aim is for the desired products to have the lowest possible content of interfering impurities such as iodine, and protic materials such as alcohols and water, so that the products are suitable for preparing Ziegler-Natta catalysts.

Summary

In one aspect, the present invention refers to solutions of mixed alkaline earth alkoxide compounds $M(OCH_2R^6)_{2-a-b}(OR^7)a[O(CHR^8)_nOR^9]_b$in a mixture with an aluminum compound $Al(OCH_2R^6)_{3-c-d}(OR^7)_c[O(CHR^8)_nOR^9]_d$ in aprotic solvents, wherein

- M is an alkaline earth metal selected from Mg, Ca, Ba, and Sr;
- OCH_2R^6 is an alkoxide radical composed of at least 3 and a maximum of 40 C atoms having a branch in the 2-position, relative to the O function; i.e., $R^6 = -CHR^{10}R^{11}$ where R^{10}, R^{11} independently stand for alkyl radicals $C_1 - C_{18}$;
- R^7 is an alkyl radical containing 2-15 C atoms which is either linear or has a branch at the 3- or higher position (relative to the O function);
- R^8 is an alkyl radical containing 1-6 C atoms which is either linear or has a branch at the 3- or higher position (relative to the O function);
- R^9 is an alkyl radical containing 2-15 C atoms which is either linear or has a branch;
- n = an integer between 1 and 4; and
- a + b ≤ 2, c + d ≤ 3, a and c can take any value from 0.01 to 0.8, and b and d can take any value from 0.1 to 1.99, wherein the content of dissolved aluminum relative to the dissolved alkaline earth metal is in the range between 0.2 and about 20 mol%.

In another aspect, the present invention refers to a method for preparing a solution according to the present invention ~~mixed alkaline earth alkoxide compounds $M(OCH_2R^6)_{2-x}(OR^7)_x$ in aprotic solvents~~, characterized in that an alkaline earth metal activated with alkylaluminum compounds is reacted in an aprotic solvent with an alcohol containing an alkoxy function $HO(CHR^8)_nOR^9$, a primary alcohol that is unbranched or has a branch at the 3- or higher position and contains 2-15 C atoms HOR^7, and optionally a primary alcohol that is branched at the 2-position. In yet another aspect, the present invention refers to use of a solution as described herein for the preparation of polymerization catalysts, in particular heterogenized polyolefin catalysts of the Ziegler-Natta type. In a further aspect, the present invention refers to use of a solution as described herein in organic synthesis, for example as a base.

Description

The objective is achieved by providing mixed alkaline earth alkoxide compounds $M(OCH_2R^6)_{2-a-b}(OR^7)_a[O(CHR^8)_nOR^9]_b$ in a mixture with an aluminum compound $Al(OCH_2R^6)_{3-c-d}(OR^7)_c[O(CHR^8)_nOR^9]_d$ in aprotic solvents, wherein
M is an alkaline earth metal selected from Mg, Ca, Ba, and Sr;

Claims

1. Solutions of mixed alkaline earth alkoxide compounds $M(OCH_2R^6)_{2-a-b}(OR^7)_a[O(CHR^8)_nOR^9]_b$ in a mixture with an aluminum compound $Al(OCH_2R^6)_{3-c-d}(OR^7)_c[O(CHR^8)_nOR^9]_d$ in aprotic solvents, wherein
   - M is an alkaline earth metal selected from Mg, Ca, Ba, and Sr;
   - OCH_2R^6 is an alkoxide radical composed of at least 3 and a maximum of 40 C atoms having a branch in the 2-position, relative to the O function; i.e., $R^6 = -CHR^{10}R^{11}$ where R^{10}, R^{11} independently stand for alkyl radicals $C_1 - C_{18}$;
   - R^7 is an alkyl radical containing 2-15 C atoms which is either linear or has a branch at the 3- or higher position (relative to the O function);
   - R^8 is an alkyl radical containing 1-6 C atoms which is either linear or has a branch at the 3- or higher position (relative to the O function);
   - R^9 is an alkyl radical containing 2-15 C atoms which is either linear or has a branch;
   - n = an integer between 1 and 4; and
   - $a + b \leq 2$, $c + d \leq 3$, a and c can take any value from 0.01 to 0.8, and b and d can take any value from 0.1 to 1.99, wherein the content of dissolved aluminum relative to the dissolved alkaline earth metal is in the range between 0.2 and about 20 mol%.

2. The solutions according to claim 1, characterized in that the alkaline earth metal concentration is in the range of 0.4 to 1.6 mmol/g, preferably between 0.7 and 1.4 mmol/g.

3. The solutions according to claims 1 to 2, characterized in that the contents of protic impurities relative to the dissolved alkaline earth element are generally between 0.1 and 40 mol%, preferably 1 and 30 mol%.

4. The solutions according to claims 1 to 3, characterized in that the content of dissolved aluminum relative to the dissolved alkaline earth metal is in the range between 1 and 8 mol%.

5. The solutions according to claims 1 to 4, characterized in that preferably a C_2-C_4 glycol monoether – e.g., 2-ethoxyethanol, 3-ethoxy-1-propanol, 3-ethoxy-1-butanol, 2-(2-ethylhexoxy) ethanol, 2-butoxyethanol, 2-hexyloxyethanol, and 1,3-propylene glycol monobutyl ether or any given mixture of at least two of the listed alcohols – is used as the alcohol containing an alkoxy function $HO(CHR^8)_nOR^9$.

6. The solutions according to claims 1 to 5, characterized by containing 0.1 to 80 mol%, especially preferably 1 to 40 mol% free alcohol, relative to the alkaline earth metal present in the solution.

7. A method for preparing a solution according to claim 1, characterized in that an alkaline earth metal activated with alkylaluminum compounds is reacted in an aprotic solvent with an alcohol containing an alkoxy function $HO(CHR^8)_nOR^9$, a primary alcohol that is unbranched or has a branch at the 3- or higher position and contains 2-15 C atoms HOR^7, and optionally a primary alcohol that is branched at the 2-position.

8. The method according to claim 7, characterized in that trialkyl, alkyl-alkoxy, and/or alkyl-halide compounds are used as an alkylaluminum compound.

9. The method according to claim 7 or 8, characterized in that hydrocarbons are used as a solvent, wherein either aliphatic solvents selected from the group consisting of cyclohexane, methylcyclohexane, hexane, heptane, octane, nonane, decane, dodecane, decalin, and commercially available boiling cuts (benzene fractions) or aromatic solvents selected from the group consisting of benzene, toluene, ethylbenzene, xylenes, and cumene are used.

10. The method according to claims 7 to 9, characterized in that the reaction takes place at temperatures between about 0 and 180°C, preferably between about 40 and 140°C, particularly at the boiling temperature of the solvent.

11. The method according to claims 7 to 10, characterized in that a C_2-C_4 glycol monoether – e.g., 2-ethoxyethanol, 3-ethoxy-1-propanol, 3-ethoxy-1-butanol, 2-

(2-ethylhexoxy) ethanol, 2-butoxyethanol, 2-hexyloxyethanol, and 1,3-propylene glycol monobutyl ether or any given mixture of at least two of the listed alcohols – is preferably used as an alcohol containing an alkoxy function $HO(CHR^8)_nOR^9$; isobutanol, 2-methyl-1-pentanol, 2-ethyl-1-butanol, 2-ethyl-1-pentanol, 2-ethyl-4-methyl-1-pentanol, 2-propyl-1-heptanol, 2-methyl-1-hexanol, 2-ethylhexanol, and 2-ethyl-5-methyl-1-octanol, or any given mixture of at least two of the listed alcohols is used as an alcohol branched at the 2-position ($HOCH_2R^6$); and ethanol, propanol, butanol, pentanol, hexanol, octanol, decanol, dodecanol, 3-methylbutan-1-ol, or any given mixture of at least two of the listed alcohols is used as a primary alcohol which is unbranched or which has a branch at the 3- or higher position and contains 2-15 C atoms (HOR^7).

12. The method according to claims 7 to 11, characterized in that first the alcohol (HOR^7) is added, and only then the alcohol containing an alkoxy function $HO(CHR^8)_nOR^9$ and the alcohol that is branched at the 2-position ($HOCH_2R^6$) are added.

13. Use of a solution according to any one of claims 1 to 6 for the preparation of polymerization catalysts, in particular heterogenized polyolefin catalysts of the Ziegler–Natta type.

14. Use of a solution according to any one of claims 1 to 6 in organic synthesis, for example as a base.

Abstract

The invention relates to concentrated low-viscosity solutions of mixed alkaline earth alkoxide compounds

$M(OCH_2R^6)_{2-a-b}(OR^7)_a[O(CHR^8)_nOR^9]_b$ in a mixture with an aluminum compound $Al(OCH_2R^6)_{3-c-d}(OR^7)_c[O(CHR^8)_nOR^9]_d$ in aprotic solvents, wherein

- M is an alkaline earth metal selected from Mg, Ca, Ba, and Sr;
- OCH_2R^6 is an alkoxide radical composed of at least 3 and a maximum of 40 C atoms having a branch in the 2-position, relative to the O function; i.e., $R^6 = -CHR^{10}R^{11}$ where R^{10}, R^{11} independently stand for alkyl radicals $C_1 - C_{18}$;
- R^7 is an alkyl radical containing 2-15 C atoms which is either linear or has a branch at the 3- or higher position (relative to the O function;
- R^8 is an alkyl radical containing 1-6 C atoms which is either linear or has a branch at the 3- or higher position (relative to the O function);
- R^9 is an alkyl radical containing 2-15 C atoms which is either linear or has a branch;
- n = an integer between 1 and 4; and
- $a + b \leq 2$, $c + d \leq 3$, a and c can take any value from 0.01 to 0.8, and b and d can take any value from 0.1 to 1.99.

Claims

1. Solutions of mixed alkaline earth alkoxide compounds $M(OCH_2R^6)_{2-a-b}(OR^7)_a[O(CHR^8)_nOR^9]_b$ in a mixture with an aluminum compound $Al(OCH_2R^6)_{3-c-d}(OR^7)_c[O(CHR^8)_nOR^9]_d$ in aprotic solvents, wherein
   - M is an alkaline earth metal selected from Mg, Ca, Ba, and Sr;
   - OCH_2R^6 is an alkoxide radical composed of at least 3 and a maximum of 40 C atoms having a branch in the 2-position, relative to the O function; i.e., $R^6 = -CHR^{10}R^{11}$ where R^{10}, R^{11} independently stand for alkyl radicals $C_1 - C_{18}$;
   - R^7 is an alkyl radical containing 2-15 C atoms which is either linear or has a branch at the 3- or higher position (relative to the O function);
   - R^8 is an alkyl radical containing 1-6 C atoms which is either linear or has a branch at the 3- or higher position (relative to the O function);
   - R^9 is an alkyl radical containing 2-15 C atoms which is either linear or has a branch;
   - n = an integer between 1 and 4; and
   - $a + b \leq 2$, $c + d \leq 3$, a and c can take any value from 0.01 to 0.8, and b and d can take any value from 0.1 to 1.99.

2. The solutions according to claim 1, characterized in that the alkaline earth metal concentration is preferably in the range of 0.4 to 1.6 mmol/g, especially preferably between 0.7 and 1.4 mmol/g.

3. The solutions according to claim 1 or 2, characterized in that the room temperature viscosities are ≤ 300 cP, preferably ≤ 200 cP, especially preferably ≤ 100 cP.

4. The solutions according to claims 1 to 3, characterized in that the contents of protic impurities relative to the dissolved alkaline earth element are generally between 0.1 and 40 mol%, preferably 1 and 30 mol%.

5. The solutions according to claims 1 to 4, characterized in that the content of dissolved aluminum relative to the dissolved alkaline earth metal is in the range between 0.2 and about 20 mol%, preferably between 1 and 8 mol%.

6. The solutions according to claims 1 to 5, characterized in that preferably a C_2-C_4 glycol monoether – e.g., 2-ethoxyethanol, 3-ethoxy-1-propanol, 3-ethoxy-1-butanol, 2-(2-ethylhexoxy) ethanol, 2-butoxyethanol, 2-hexyloxyethanol, and 1,3-propylene glycol monobutyl ether or any given mixture of at least two of the listed alcohols – is used as the alcohol containing an alkoxy function $HO(CHR^8)_nOR^9$.

7. The solutions according to claims 1 to 6, characterized by containing 0.1 to 80 mol%, especially preferably 1 to 40 mol% free alcohol, relative to the alkaline earth metal present in the solution.

8. A method for preparing mixed alkaline earth alkoxide compounds $M(OCH_2R^6)_{2-x}(OR^7)_x$ in aprotic solvents, characterized in that an alkaline earth metal activated with alkylaluminum compounds is reacted in an aprotic solvent with an alcohol containing an alkoxy function $HO(CHR^8)_nOR^9$, a primary alcohol that is unbranched or has a branch at the 3- or higher position and contains 2-

15 C atoms HOR^7, and optionally a primary alcohol that is branched at the 2-position.

9. The method according to claim 8, characterized in that trialkyl, alkyl-alkoxy, and/or alkyl-halide compounds are used as an alkylaluminum compound.

10. The method according to claim 8 or 9, characterized in that hydrocarbons are used as a solvent, wherein either aliphatic solvents selected from the group consisting of cyclohexane, methylcyclohexane, hexane, heptane, octane, nonane, decane, dodecane, decalin, and commercially available boiling cuts (benzene fractions) or aromatic solvents selected from the group consisting of benzene, toluene, ethylbenzene, xylenes, and cumene are used.

11. The method according to claims 8 to 10, characterized in that the reaction takes place at temperatures between about 0 and 180°C, preferably between about 40 and 140°C, particularly at the boiling temperature of the solvent.

12. The method according to claims 8 to 11, characterized in that a C_2-C_4 glycol monoether – e.g., 2-ethoxyethanol, 3-ethoxy-1-propanol, 3-ethoxy-1-butanol, 2-(2-ethylhexoxy) ethanol, 2-butoxyethanol, 2-hexyloxyethanol, and 1,3-propylene glycol monobutyl ether or any given mixture of at least two of the listed alcohols – is preferably used as an alcohol containing an alkoxy function $HO(CHR^8)_nOR^9$; isobutanol, 2-methyl-1-pentanol, 2-ethyl-1-butanol, 2-ethyl-1-pentanol, 2-ethyl-4-methyl-1-pentanol, 2-propyl-1-heptanol, 2-methyl-1-hexanol, 2-ethylhexanol, and 2-ethyl-5-methyl-1-octanol, or any given mixture of at least two of the listed alcohols is used as an alcohol branched at the 2-position ($HOCH_2R^6$); and ethanol, propanol, butanol, pentanol, hexanol, octanol, decanol, dodecanol, 3-methylbutan-1-ol, or any given mixture of at least two of the listed alcohols is used as a primary alcohol which is unbranched or which has a branch at the 3- or higher position and contains 2-15 C atoms (HOR^7).

13. The method according to claims 8 to 12, characterized in that first the alcohol (HOR^7) is added, and only then the alcohol containing an alkoxy function $HO(CHR^8)_nOR^9$ and the alcohol that is branched at the 2-position ($HOCH_2R^6$) are added.

14. Use of a solution according to any one of claims 1 to 7 for the preparation of polymerization catalysts, in particular heterogenized polyolefin catalysts of the Ziegler–Natta type.

15. Use of a solution according to any one of claims 1 to 7 in organic synthesis, for example as a base.

Abstract

The invention relates to concentrated low-viscosity solutions of mixed alkaline earth alkoxide compounds

$M(OCH_2R^6)_{2-a-b}(OR^7)_a[O(CHR^8)_nOR^9]_b$ in a mixture with an aluminum compound $Al(OCH_2R^6)_{3-c-d}(OR^7)_c[O(CHR^8)_nOR^9]_d$ in aprotic solvents, wherein

- M is an alkaline earth metal selected from Mg, Ca, Ba, and Sr;
- OCH_2R^6 is an alkoxide radical composed of at least 3 and a maximum of 40 C atoms having a branch in the 2-position, relative to the O function; i.e., $R^6 = -CHR^{10}R^{11}$ where R^{10}, R^{11} independently stand for alkyl radicals $C_1 - C_{18}$;
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- R^8 is an alkyl radical containing 1-6 C atoms which is either linear or has a branch at the 3- or higher position (relative to the O function);
- R^9 is an alkyl radical containing 2-15 C atoms which is either linear or has a branch;
- n = an integer between 1 and 4; and
- $a + b \leq 2$, $c + d \leq 3$, a and c can take any value from 0.01 to 0.8, and b and d can take any value from 0.1 to 1.99.