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(54) Title
PROCESS FOR THE SYNTHESIS OF MONOMETHYLMETALLOCENES AND DIMETHYLMETALLOCENES AND THEIR SOLUTIONS SPECIFICALLY FOR USE IN THE POLYMERIZATION OF OLEFINS

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(56) Prior Art Documents
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(57) Claim

1. Process for preparing methyl-transition metal compounds of the general formula (1)

$$(Q)_m(CpR_a)(Cp'R'_{a'})M(CH_3)_nX_{3-n} \qquad (1)$$

where

Cp is a cyclopentadienyl, an indenyl, a fluorenyl radical

R, R′ are alkyl, phosphine, amine, alkyl ether or aryl ether groups with $0 \leq a \leq 5$, $0 \leq a' \leq 5$

Cp′ is one of the groups Cp or

Cp′ is N when R′ = alkyl or aryl radical and a′ = 1 and

Q is a single-membered or multi-membered bridge

$$(R^1-\overset{|}{\underset{|}{Z}}-R^2)_b$$

.../2

between Cp and Cp', where R^1 and R^2 are identical or different and are a hydrogen atom, a C_1-C_{10}-alkyl group, a C_6-C_{10}-aryl group and Z is carbon, silicon or germanium with b = 1, 2 or 3

M is a transition metal of the groups III to VI, in particular Ti, Zr, Hf

X is halogen, in particular Cl, Br and

n can be 1 or 2

m can be 0 or 1,

characterised in that halogen-transition metal compounds of the general formula (2

$$Q_m(CpR_n)(Cp'R'_{n'})MX_2 \qquad (2)$$

where

Cp is a cyclopentadienyl, an indenyl, a fluorenyl radical

R, R' are alkyl, phosphine, amine, alkyl ether or aryl ether groups with $0 \le a \le 5$ $0 \le a' \le 5$

Cp' is one of the groups Cp or

Cp' is N with R' = alkyl or aryl radical with a=1 and

Q is a single-membered or multi-membered bridge

$$(R^1\text{-}\overset{|}{\underset{|}{Z}}\text{-}R^2)_b$$

between Cp and Cp', where R^1 and R^2 are identical or different and are a hydrogen atom, a C_1-C_{10}-alkyl group, a C_1-C_{10}-alkyl group, a C_6-C_{10}-aryl group and Z is carbon, silicon or germanium with b=1, 2 or 3

M is a transition metal of the groups III to VI, in particular Ti, Zr, Hf

X is halogen, in particular Cl, Br and

m can be 0 or 1

are initally charged in a hydrocarbon together with trimethylaluminum and are reacted, if desired at elevated temperature, and the reaction solution is then admixed with an alkali metal and/or alkaline earth metal fluoride and the aluminum fluoride complexes formed are subsequently separated off.

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ORIGINAL
COMPLETE SPECIFICATION
STANDARD PATENT

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Invention Title:	Process for the synthesis of monomethylmetallocenes and dimethylmetallocenes and their solutions specifically for use in the polymerization of olefins

The following statement is a full description of this invention, including the best method of performing it known to me:-

5020

The present invention relates to an improved process for preparing alkyl-transition metal complexes, in particular monoalkylmetallocenes and dialkylmetallocenes and to a process for preparing solutions of such compounds which are directly suitable for use in the polymerization of olefins.

Monoalkylmetallocene and particularly dialkylmetallocene compounds can in combination with specific cocatalysts, such as, for example, aluminoxanes or, in particular, triphenylboron derivatives, tetraphenylborate derivatives and alkylaluminum fluorides, form highly active catalyst systems (J. Organomet. Chem. 1992, 434, C1 - C5, Organometallics 1991, 10, 3910; J. Am. Chem. Soc. 1989, 111, 2728; EP-A-0 522 581; EP-A-0 468 537).

Processes hitherto known in the literature for preparing monoalkylmetallocene and dialkylmetallocene compounds (in particular dimethyl compounds) in which lithium alkyls or alkyl Grignard compounds are used, have a series of disadvantages.

As a result of the use of methyl Grignard or methyllithium, the syntheses are tied to polar solvents such as, typically, diethyl ether or tetrahydrofuran. However, these solvents are catalyst poisons for the application in olefin polymerization, so that after the reaction of the metallocenes with the corresponding alkylating agent, the solvent has to be completely

removed and the compound has to be isolated in pure form.

Furthermore, depending on the metallocene, the yields in their reactions to give the corresponding alkyl derivatives are very variable. (Chemistry of Organo-Zirconium and -Hafnium Compounds", D.J. Cardin, M.F. Lappert, C.L. Raston, 1986, Ellis Horwood Limited, 145-180; "Organometallic Chemistry of Titanium, Zirconium and Hafnium", P.C. Wailes, R.S.P. Coutts and H. Wiegold, 1974, Academic Press, Ins., 92-97, 150-151, 185-187; Gmelins Handbook of Inorganic Chemistry, Volume 10, Organo-Zirconium Compounds, 1973, Verlag Chemie - Weinheim/Bergstraße, p. 54-58; Gmelins Handbook of Inorganic Chemistry, Volume 11, Organo-Hafnium Compounds, 1973, Verlag Chemie - Weinheim/Bergstraße, p. 12).

It is therefore an object of the present invention to develop a process which overcomes these disadvantages of the prior art and by means of which either the pure alkyl-transition metal compounds can be prepared directly in high yields or even can be obtained without additional work-up steps directly in the solutions of the alkyl-transition metal compounds which are free of catalyst poisons and can be used in olefin polymerization.

This object is achieved by reaction of halogen-transition metal compounds with aluminum alkyls and inorganic salts in hydrocarbons according to the general scheme

$$Cp_2MX_2 \xrightarrow{nR_3Al} \cdot Cp_2MR_2 \cdot nR_2AlX \cdot \xrightarrow[n \cdot R_3AlXX' \cdot M'^+]{nM'X'} Cp_2MR_nX_{2-n}$$

The invention accordingly provides a process for preparing compounds of the general formula (1)

$$(Q)_m(CpR_a)(Cp'R'_{a'})M(CH_3)_nX_{2-n} \qquad (1)$$

where

Cp is a cyclopentadienyl, an indenyl, a fluorenyl radical

R, R' are alkyl, phosphine, amine, alkyl ether or aryl ether groups with $0 \leq a \leq 5$, $0 \leq a' \leq 5$

Cp' is one of the groups Cp or

Cp' is N with R' = alkyl or aryl radical with a = 1 and

Q is a single-membered or multi-membered bridge

$$(R^1\text{-}\underset{|}{\overset{|}{Z}}\text{-}R^2)_b$$

between Cp and Cp', where R^1 and R^2 are identical or different and are a hydrogen atom, a C_1-C_{10}-alkyl group, a C_6-C_{10}-aryl group and Z is carbon, silicon or germanium with b = 1, 2 or 3

M is a transition metal of the groups III to VI, in particular Ti, Zr, Hf

X i halogen, in particular Cl, Br and

n can be 1 or 2

m can be 0 or 1.

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The halogen-transition metal compounds used according to the invention correspond to the general formula (2)

$$(Q)_m(CpR_a)(Cp'R'_{a'})MX_2 \qquad (2)$$

where

Cp is a cyclopentadienyl, an indenyl, a fluorenyl radical

R,R' are alkyl, phosphine, amine, alkyl ether or aryl ether groups with $0 \leq a \leq 5$, $0 \leq a' \leq 5$

Cp' is one of the groups Cp or

Cp' is N with R' = alkyl or aryl radical with a = 1 and

Q is a single-membered or multi-membered bridge

$$(R^1-\overset{|}{\underset{|}{Z}}-R^2)_b$$

between Cp and Cp', where R^1 and R^2 are identical or different and are a hydrogen atom, a C_1-C_{10}-alkyl group, a C_6-C_{10}-aryl group and Z is carbon, silicon or germanium with b = 1, 2 or 3

M is a transition metal of the groups III to VI, in particular Ti, Zr, Hf

X is halogen, in particular Cl, Br and

m can be 0 or 1.

These compounds belong to the known prior art and are described, for example, in "Chemistry of Organo-Zirconium and -Hafnium Compounds", D.J. Cordin [sic];

M.F. Lappert; C.L. Raston, 1986, Ellis Horwood Ltd, 145-180; EP-A-O 576 970, EP-A-0 549 900, EP-A-0 522 581, EP-A-O-519 237, EP-A-0-468 537, EP-A-0-420 436, EP-A-0 416 815, EP-A-0-302 424.

These compounds are initially charged in an inert solvent, preferably aliphatic and/or aromatic hydrocarbons.

The hydrocarbons preferably have boiling points between 50 and 150°C, in particular between 70-120°C, such as hexane, heptane, octane, decane, toluene, xylene.

Trimethylaluminum (TMA) is metered into this mixture while stirring vigorously at room or elevated temperature.

Depending on the desired degree of substitution (monomethyl or dimethyl compound), the molar ratio of halogen-transition metal compound : TMA is from 1:1.1 to 1:2.4. In the case of the dimethyl compounds, the excesses are not critical, but, with regard to process economy, should be kept as small as possible.

The mixture of halogen-transition metal compound and TMA is reacted with vigorous stirring at temperatures of 20-120°C, preferably 70-100°C. At these temperatures, reaction times of 0.5-1.5 hours are sufficient.

The reaction mixture is, if desired after cooling to room temperature, admixed with an alkali metal and/or alkaline earth metal fluoride, preferably NaF or KF. Preference is here given to the ratio of F^- to TMA = 1:1.

For the quantitative formation of the methyl-transition metal compound, the reaction is advantageously

carried out to completion for 0.5-1.5 hours at 70-100°C.

The insoluble aluminum fluoride complexes formed are separated off by conventional methods such as decantation, centrifugation, filtration.

The filtrate which contains the methyl-transition metal compounds in high purity and yield can, without further work-up processes, be directly used in the polymerization of olefins.

If desired, the methyl-transition metal compounds can also be isolated by conventional methods.

This is to be illustrated by the following examples. All reactions were carried out with exclusion of moisture and O_2 in an inert gas atmosphere.

Examples

Example 1

a) Reaction of indenyl$_2$ZrCl$_2$ with TMA/KF in toluene

20 g of indenyl$_2$ZrCl$_2$ (51 mmol) were initially charged in 250 ml of toluene and admixed with 7.35 g of trimethylaluminum (102 mmol) and heated to 80°C.

After 30 minutes, 5.92 g of KF (102 mmol) were added and the mixture was refluxed for 1 hour.

The reaction solution was then filtered hot and the filtrate cooled to -20°C.

14.2 g (79%) of indenyl$_2$ZrMe$_2$ were able to be isolated by means of filtration.

^{1}H-NMR ($CDCl_3$):

7.5-7.4 (m, 4H, aromatic H); 7.15-7.05 (m, 4H, aromatic H); 6.08 (d, 4H, C_5H_2);

5.95 (t, 2H, C_5H); -1.15 (s, 6H, CH_3)

Zr: found: 24.8 (calc.: 25.9%);

Hydrolysis gas: CH_4: found: 127 standard ml/g (calc.: 127.4 standard ml/g)

b) Reaction of indenyl$_2$ZrCl_2 with KF/TMA in heptane

The procedure was similar to 1 a), but heptane was used in place of toluene. 14.75 (83%) of clean product were obtained.

Zr: found: 25.1% (calc.: 25.9%);

Hydrolysis gas: CH_4: found: 127 standard ml/g (calc.: 127.4 standard ml/g)

[^{1}H-NMR identical with that in 1 a)]

Example 2:

Reaction of ethyleneindenyl$_2$ZrCl_2 with KF/TMA

19.2 g of rac-ethyleneindenyl$_2$ZrCl_2 (35.9 mmol) in 200 ml of heptane were initially charged and admixed with 7.35 g of trimethylaluminum (102 mmol).

After refluxing for 1/2 hour, 5.92 g of KF (102 mmol) were added and the mixture was refluxed for a

further 1 1/2 hours.

After hot filtration, the filtrate was evaporated to 50 ml and cooled to -20°C.

15 g of pure ethylene-indenyl$_2$ZrMe$_2$ (86.5% of theory) were able to be finally isolated by means of filtration.

Zr: found: 23.5% (calc.: 24.2%);

Hydrolysis gas: CH_4: found: 115 standard ml/g (calc.: 118.6 standard ml/g)

^{1}H-NMR ($CDCl_3$):

7.5-7.0 (m, 8H, C_6H_4); 6.55 (d, 2H, C_5H); 6.0 (d, 2H, C_5H); 3.4-3.1 (m, 4H, $-CH_2CH_2-$); -1.4 (s, 6H, CH_3)

Example 3

Reaction of Cp_2ZrCl_2 with KF/TMA

1.94 g of Cp_2ZrCl_2 (6.64 mmol) were suspended in 10 ml of heptane and admixed with 0.96 g of trimethylaluminum (13.8 mmol) and refluxed for 1/2 hour. After addition of 0.77 g of KF, the mixture was refluxed for a further 60 minutes.

Subsequently, ^{1}H-NMR was able to detect only the desired compound Cp_2ZrMe_2 as metallocene in the solution.

^{1}H-NMR ($CDCl_3$):

6.1 (s, 10H, C_5H_5), -0.4 (s, 6H, CH_3)

Example 4

Reaction of n-butylCp_2ZrCl_2 with KF/TMA

1.94 g of n-butyl Cp_2ZrCl_2 (4.8 mmol) were added to 10 ml of heptane, admixed with 0.7 g of trimethylaluminum (9.6 mmol) and stirred for 1/2 hour at 80°C.

0.56 g of KF (9.6 mmol) was then added and again refluxed for 1 hour. ^{1}H-NMR spectroscopy was then able to detect only the desired compound n-butylCp_2ZrMe_2 as metallocene in the solution.

Neither the starting material n-butylCp_2ZrCl_2 nor the intermediate stage n-butyl$Cp_2ZrCl_2(CH_3)$ [sic] were detectable.

^{1}H-NMR ($CDCl_3$):

5.95-5.9 (m, 4H, C_5H_2), 5.83-5.78 (m, 4H, C_5H_2)

2.45 (t, 4H, $-CH_2-$); 1.6-1.25 (m, 8H, $-CH_2-CH_2$); 0.95 (t, 6H, CH_3);

-0.5 (s, 6H, CH_3)

Example 5

Reaction of 1,3-butylmethylCp_2ZrCl_2

The procedure was similar to that in 4., but using 2.1 g (4.8 mmol) of 1,3-butylmethylCp_2ZrCl_2.

Again, only the desired 1,3-n-butylmethylCp_2ZrMe_2 was able to be detected as metallocene compound.

^{1}H-NMR ($CDCl_3$):

5.78 (t, 2H, C_5H); 5.52 (d, 4H, C_5H_2), 2.4-2.15 (m, 4H, $-CH_2-$)

2.05 (s, 6H, CH_3); 1.6-1.3 (m, 8H, $-CH_2CH_2-$); 0.9 (t, 6H, CH_3)

-0.53 (s, 6H, CH_3)

Example 6

Reaction of $Me_2Si[(^tBuN)(Me_4Cp)]TiCl_2$ with KF/TMA

2.38 g (6.4 mmol) of $Me_2Si[(Me_4Cp)(N^tBu)]TiCl_2$ were initially charged in 10 ml of heptane and admixed at room temperature with 1 g of trimethylaluminum (13.8 mmol).

The mixture was refluxed for 30 minutes and then admixed with 0.84 g of KF (13.8 mmol) and again refluxed for 30 minutes.

Subsequently, ^{1}H-NMR was able to detect only the compound $Me_2Si[(Me_4Cp)(N^tBu)]TiMe_2$ in the reaction solution.

^{1}H-NMR ($CDCl_3$):

2.18 (s, 6H, Me_2Cp); 1.92 (s, 6H, Me_2Cp); 1.58 (s, 9H, t-butylN);

0.48 (s, 6H, $(H_2C)_2Si$); 0.18 (s, 6H, $(H_3C)_2Ti$)

Example 7

Reaction of n-butylCp$_2$HfCl$_2$ with KF/TMA

2.21 g of bis(n-butylcyclopentadienyl)hafnium dichloride (4.49 mmol) were initially charged in 30 ml of heptane

and admixed at room temperature with 1.76 ml of trimethylaluminum. The mixture was subsequently stirred for 30 minutes at 90°C.

1.04 g of potassium fluoride (17.98 mmol) were then added and the mixture stirred for a further 30 minutes at 90°C.

^{1}H-NMR spectroscopy showed only the desired product bis(n-butylCp)$HfMe_2$ and no longer any starting material.

^{1}H-NMR ($CDCl_3$):
5.85 (m, 4H, H_2Cp); 5.75 (m, 4H, H_2Cp); 2.45 (t, 4H, $-CH_2-$);
1.65-1.2 (m, 8H, $-CH_2CH_2-$); 0.95 (t, 6H, $-CH_3$); -0.62 (s, 6H, H_3C-Ti)

Example 8

Attempt at the reaction of indenyl$_2ZrCl_2$ with LiCl/TMA

25 g of indenyl$_2ZrCl_2$ (64 mmol) were initially charged in 200 ml of heptane, admixed with 25 ml of trimethylaluminum (255 mmol) and refluxed for 1 hour.

10.81 g of LiCl (255 mmol) were then added and the mixture again refluxed for 1 hour.

^{1}H-NMR was able to detect no formation of the desired dimethyl derivative.

Example 9

Attempt at the reaction of indenyl$_2ZrCl_2$ with $ZnCl_2$

2.7 g of indenyl$_2$ZrCl$_2$ (7.5 mmol) were initially charged in 30 ml of heptane, admixed with 2.7 ml of trimethylaluminum (28 mmol) and refluxed for 1 hour.

4.24 g of zinc chloride (28 mmol) and the mixture was again refluxed for 1 hour.

No formation of the desired dimethyl derivative was able to be observed by means of ^{1}H-NMR.

Example 10

Attempt at the reaction of indenyl$_2$ZrCl$_2$ with KCl/TMA

2.92 indenyl$_2$ZrCl$_2$ (7.5 mmol) were initially charged in 30 ml of heptane together with 2.92 g of trimethylaluminum (30 mmol) and refluxed for 1 hour.

2.22 g of KCl (30 mmol) were then added, and the mixture was refluxed for a further 2 hours.

^{1}H-NMR was able to detect no formation of indenyl$_2$ZrMe$_2$.

Example 11

Reaction of indenyl$_2$ZrCl$_2$ with NaF/TMA

40 g of indenyl$_2$ZrCl$_2$ (102 mmol) were initially charged in 400 ml of heptane and admixed with 40 ml of trimethylaluminum (408 mmol). The mixture was refluxed for 1 hour.

17.13 g of NaF (408 mmol) were then added, and the mixture was refluxed for a further 2 hours.

The reaction solution was filtered hot and cooled to -20°C.

9.91 g (27.5% of theory) of pure indenyl$_2$ZrCl(CH_3) were able to be isolated.

^{1}H-NMR:

7.6-7.15 (m, 8H, C_6H_4); 6.2-6.05 (m, 6H, C_5H_3); -0.55 (s, 6H, CH_3)

Example 12

Reaction of indenyl$_2$ZrCl$_2$ with KF/TMA (1:1)

50 g (128 mmol) of indenyl$_2$ZrCl$_2$ were initially charged in 500 ml of heptane and admixed at room temperature with 12.6 ml (128 mmol) of trimethylaluminum. The mixture was refluxed for 1 hour.

7.95 g (128 mmol) of KF were then added, and the mixture was refluxed for a further 2 hours.

After hot filtration to remove the insoluble salts, the filtrate was cooled to -20°C.

31.8 g (71% of theory) of pure indenyl$_2$ZrCl(CH_3) were able to be isolated by means of filtration.

^{1}H-NMR (identical with that in 11.)

Comparative Example

a) Reaction of $Me_2Si[Me_4Cp)(N^tBu)]TiCl_2$ with TMA without addition of KF.

2.4 g (6.5 mmol) of $Me_2Si[Me_4Cp)(N^tBu)]TiCl_2$ were initially charged in 10 ml of heptane and admixed with 1.92 g of

trimethylaluminum. The mixture was then refluxed for 1 hour.

Subsequently, ^{1}H-NMR was able to detect the monomethylated compound in small amounts (14%). However, the solution comprised 86% of the starting material, the corresponding titanium dichloride complex. (However, if 1.55 g of KF were then added and the mixture allowed to react for a further 1 hour at 80°C, quantitative formation of the dimethyltitanocene resulted.)

b) Separate reaction of KF and trimethylaluminum prior to use for the methylation of $Me_2Si[Me_4Cp)(N^tBu)]TiCl_2$.

0.46 g of KF (8 mmol) and 0.8 ml (8 mmol) of trimethylaluminum were stirred in 10 ml of heptane for 1/2 hour at 90°C. 1.48 g of $Me_2Si[Me_4Cp)(N^tBu)]TiCl_2$ were then added at room temperature, and the mixture was stirred for a further 3 hours at 80°C.

^{1}H-NMR was able to detect, besides the starting compound (82%), only the monomethylated compound (18%).

c) Addition of KF to $Me_2Si[Me_4Cp)(N^tBu)]TiCl_2$.

0.96 g of KF (16.5 mmol) and 2.04 g of the titanium dichloride compound were initially charged in 10 ml of heptane and stirred for 1 hour at 80°C.

No reaction (replacement of Cl by F) could be detected.

The Claims defining the invention are as follows:

1. Process for preparing methyl-transition metal compounds of the general formula (1)

$$(Q)_m(CpR_a)(Cp'R'_{a'})M(CH_3)_nX_{2-n} \qquad (1)$$

where

Cp is a cyclopentadienyl, an indenyl, a fluorenyl radical

R, R' are alkyl, phosphine, amine, alkyl ether or aryl ether groups with $0 \leq a \leq 5$, $0 \leq a' \leq 5$

Cp' is one of the groups Cp or

Cp' is N when R' = alkyl or aryl radical and a' = 1 and

Q is a single-membered or multi-membered bridge

$$(R^1-\overset{|}{\underset{|}{Z}}-R^2)_b$$

between Cp and Cp', where R^1 and R^2 are identical or different and are a hydrogen atom, a C_1-C_{10}-alkyl group, a C_6-C_{10}-aryl group and Z is carbon, silicon or germanium with b = 1, 2 or 3

M is a transition metal of the groups III to VI, in particular Ti, Zr, Hf

X is halogen, in particular Cl, Br and

n can be 1 or 2

m can be 0 or 1,

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characterised in that halogen-transition metal compounds of the general formula (2)

$$(Q)_m(CpR_a)(Cp'R'_{a'})MX_2 \qquad (2)$$

where

Cp is a cyclopentadienyl, an indenyl, a fluorenyl radical

R, R' are alkyl, phosphine, amine, alkyl ether or aryl either groups with $0 \leq a \leq 5$, $0 \leq a' \leq 5$

Cp' is one of the groups Cp or

Cp' is N with R' = alkyl or aryl radical with a-1 and

Q is a single-membered or multi-membered bridge

$$(R^1-\overset{|}{\underset{|}{Z}}-R^2)_b$$

between Cp and Cp', where R^1 and R^2 are identical or different and are a hydrogen atom, a C_1-C_{10}-alkyl group, a C_6-C_{10}-alkyl group, a C_6-C_{10}-aryl group and Z is carbon, silicon or germanium with b=1, 2 or 3

M is a transition metal of the groups III to VI, in particular Ti, Zr, Hf

X is halogen, in particular Cl, Br and

m can be 0 or 1

are initally charged in a hydrocarbon together with trimethylaluminum and are reacted, if desired at

elevated temperature, and the reaction solution is then admixed with an alkali metal and/or alkaline earth metal fluoride and the aluminum fluoride complexes formed are subsequently separated off.

2. Process according to Claim 1, characterized in that the reaction is carried out at temperatures of 20-120°C.

3. Process according to Claim 1, characterized in that the reaction is carried out in substituted or unsubstituted aliphatic hydrocarbons and substituted or unsubstituted aromatic hydrocarbons having boiling points between 50-150°C.

4. Process according to Claim 1, characterized in that the fluorides used are NaF or KF.

5. Process according to Claim 1, characterized in that the molar ratio of halogen-transition metal compound: trimethylaluminum:fluoride = 1:1:1 in the case of the monomethyl-transition metal compound and 1:2:2 in the case of the dimethyl-transition metal compound.

6. A process for the production of methyl-transition metal compounds of the general formula (1) substantially as hereinbefore described with reference to the Examples.

DATED this 6th day of February 1995,

WITCO GMBH
By Its Patent Attorney
DAVIES COLLISON CAVE

Abstract:

Process for the synthesis of monomethylmetallocenes and dimethylmetallocenes and their solutions specifically for use in polymerization of olefins

The invention provides a process for preparing methyl-transition metal compounds in which the metallocene to be alkylated is initially charged in a hydrocarbon desired for the subsequent application, admixed with an aluminum alkyl and subsequently converted by addition of an inorganic salt into the end product:

$$Cp_2MX_2 \xrightarrow{2\ R_3Al} \xrightarrow[2\ [R_2AlXX']^-M'^+]{2\ M'X'} Cp_2MR_2$$

The reaction solution thus obtained can be freed of precipitated salts by simple filtration and directly used for polymerization, since the dialkylmetallocene is obtained in high purity and yield. If desired, it can also be isolated without problems.