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(54) **SYNTHESE DE COMPOSES SILYLES ET N - SILYLES**

(54) **SILYLATED AND N-SILYLATED COMPOUND SYNTHESIS**

(57) La présente invention concerne de nouveaux silanes de formule $(RSO_3)_2$-Si-Q_2.0.5HX. Elle a trait à la synthèse de ligands bis-cyclopentadiényle silylés et de mono (cyclopentadiényle) N-silylés et de ligands indényle analogues au moyen des nouveaux silanes.

(57) Novel silanes having the formula $(RSO_3)_2$-Si-Q_2.0.5HX are described. Synthesis of silylated bis-cyclopentadienyl and N-silylated mono(cyclopentadienyl) ligands and similar indenyl ligands utilizing the novel silanes is described.

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(54) **Title:** SILYLATED AND N–SILYLATED COMPOUND SYNTHESIS

(57) **Abstract**

Novel silanes having the formula $(RSO_3)_2$–Si–Q_2.0.5HX are described. Synthesis of silylated bis–cyclopentadienyl and N–silylated mono(cyclopentadienyl) ligands and similar indenyl ligands utilizing the novel silanes is described.

SILYLATED AND N-SILYLATED COMPOUND SYNTHESIS

This application is a continuation of United States application Serial No. 09/016,641 filed 30 January 1998.

FIELD OF INVENTION

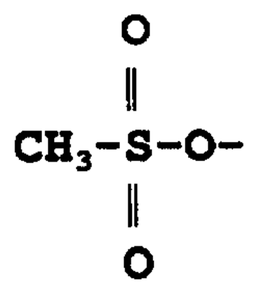
This invention relates to certain novel silanes and to the synthesis of silylated and N-silylated organic compounds therewith.

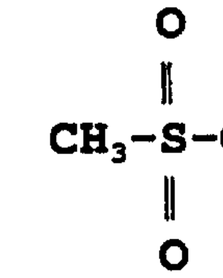
BACKGROUND OF THE INVENTION

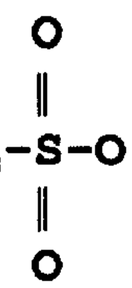
Typical procedures for the synthesis of silylated and N-silylated bis and mono(cyclopentadienyl) and indenyl ligands involve the addition of $Cl_2Si(CH_3)_2$ during synthesis of monocyclopentadienyl compounds to the lithiated ligand precursor. These procedures are not cost effective due to a requirement for excess $Cl_2Si(CH_3)_2$, the production of undesirable by-products, and a consequent need for expensive purification procedures.

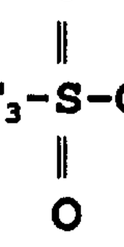
DEFINITIONS

In this specification, the following expressions have the meanings set forth:

1. MsO means CH_3O_3S or 

2. MsOH means CH_4O_3S or 

3. TfO means CF_3O_3S or 

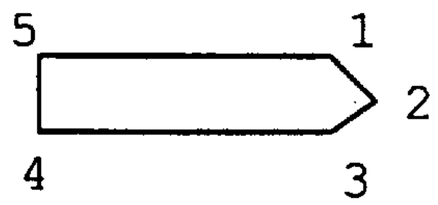
4. TfOH means CHF_3O_3S or 

5. Monocyclopentadienyl ligand means any ligand having the formulae $C_5H_xR_y$, wherein:

X = 0-5

y = 0-5

R = any alkyl or aromatic group or combination thereof, and H or R can occupy any one or more of the positions 1 to 5 of the formula



For example, R may be an alkyl group having one to eight carbon atoms including but not limited to methyl, ethyl, propyl, isopropyl, butyl, tertbutyl, hexyl or octyl. Methyl is the preferred alkyl group. R, when an aromatic group, may be phenyl, xylyl, mesityl, naphthyl or fluorenyl.

6. Silylated monocyclopentadienyl ligand means any ligand having the formula $(R_3Si)_zC_5H_xR_y$, wherein $C_5H_xR_y$ is as defined in definition 5, Z=1-5 and R and R_y are identical or different alkyl or aromatic groups.

7. N-silylated monocyclopentadienyl ligand means any ligand having the formula $RNH(SiR_2)C_5H_xR_y$, wherein $C_5H_xR_y$ is as defined in definition 5, and R and R_y are identical or different alkyl or aromatic groups.

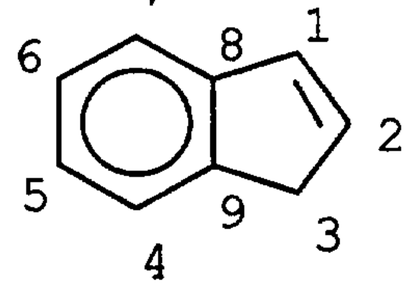
8. Silylated biscyclopentadienyl ligand means any ligand having the formula $(C_5H_xR_y)_2SiR_2$, wherein $C_5H_xR_y$ and R_y are as defined by definitions 6 and 7.

9. Silylated monoindenyl ligand means any ligand having the formula $(R_3Si)(C_9H_xR_y)$ wherein

X = 0-7

y = 0-7

H or R can occupy any positions 1 to 7 and R_3Si can occupy only position 3 of the formula



wherein R and R_y are as defined by definitions 6 and 7.

10. N-silylated monoindenyl ligand means any ligand having the formula $RNH(SiR_2)C_9H_xR_y$, wherein R and R_y are as defined by definitions 6 and 7. $(C_9H_xR_y)$ is as defined in definition 9 and wherein X = 0-7 and y = 0-7.

11. Silylated bisindenyl ligand means any ligand having the formula $(R_2Si)C_9H_xR_y$ wherein R and R_y are as defined in definitions 6 and 7; X = 0-7 and y = 0-7.

SUMMARY OF THE INVENTION

One aspect of the invention includes novel silanes having the Formula (I):

$$(CX_3SO_3)_2SiR_2 \qquad (I)$$

or the Formula (II):

$$(CX_3SO_3)_2SiR \qquad (II)$$
$$|$$
$$NHR^1$$

in which X is H or F, each R in formula (I) may be the same or a different alkyl or aromatic group as defined by definition 5 with the proviso that when X is F in formula (I), R is not methyl, and R^1 is an alkyl or aromatic group which may be the same or different from R.

Another aspect of the invention is a method for the synthesis of silylated and N-silylated compounds having the Formula (III)

$$Y_2Si(R)_2 \qquad (III)$$

or the Formula (IV)

$$YSi(R_2) \qquad (IV)$$
$$|$$
$$NHR^1$$

in which Y is any organic group and in which R and R^1 are the same or different organic groups, preferably substituted or unsubstituted aliphatic or aryl groups as defined by definition 5.

The invention includes methods for reacting organic alkali metallides having the formula YM, in which Y is any organic group and M is any alkali metal with a silane having the Formula (I) or Formula (II) wherein the product is a compound having the Formula (III) or Formula (IV).

A first step of such methods includes preparation of an organic alkali metallide. Methods for the preparation of such compounds are known. For example, any compound having a -CH group, preferably acidic, is reacted with an alkali metal alkyl having the formula R^3M, in which R^3 may be any hydrocarbyl group and M may be lithium, potassium or sodium. M may also be a magnesium halide. N-butyl lithium or tert-butyl lithium are preferred RM compounds. The reaction is conducted in a non-interfering solvent, preferably diethyl ether or tetrahydrofuran, which may also include or be combined or mixed with a hydrocarbon such as toluene. The reaction mixture contains a desired alkali metallide.

In a second step, the alkali metallide product of the first step is optionally but not necessarily isolated from the first step reaction mixture and reacted with a silane having the Formula (I) or the Formula (II). Methods for such isolating such compounds are known.

In one aspect of the invention, the compound having -CH group is a C_5-ring containing compound useful as an olefin polymerization catalyst ligand or as a precursor of such a ligand. Such ligands include but are not limited to substituted, unsubstituted, mono-, or bis- cyclopentadienyl, indenyl, naphthenyl and antracenyl ligands. These ligands may be hydrogenated. For example, such ligands include cyclopentadienes, bis-cyclopentadienes, indenes, bis-indenes, mono- and poly- alkyl, preferably methyl, substituted cyclopentadienes and indenes, such as tetraethyl cyclopentadiene and 2-methyl indene, 2-methyl-benzo(indene), bis-2-methyl-benzo(indene), dimethyl silane, substituted,

unsubstituted and bis-phenanthrene, and cyclopentaphenanthrene which may be but need not be hydrogenated.

Another aspect of the invention may include a method which comprises combining a compound having the formula $Q^1-(Z)-Q^2Li_2$ and a compound having the formula $R^1_3SiO_3R^2$ in a non-interfering solvent wherein said compound having the formula $Q^1-(Z)-Q^2Li$ reacts with the compound having the formula $R^1_3SiO_3R^2$ to produce a compound having the formula

$$R^1_3SiQ^1(Z)_xQ^2SiR^1_3$$

or

$R^1_3SiQ^1$ where Q^1=indene

or

$R^1_3SiQ^2$ where Q^2=cyclopentadiene

wherein R^1 and Q^1 and Q^2 each have 1 to 10 carbon atoms are the same or different aryl, preferably phenyl, and R^2 are identical or different alkyl groups.

Specifically, Q^1 and Q^2

(i) may be the same or different;

(ii) are preferably unsubstituted;

(iii) may be substituted at any position not occupied by linkage to $(Z)_x$ or to lithium and

(iv) Z is a linking group, preferably $(CH_2)_y$ in which y is 1 to 6 or $Si(R^2)$ wherein R^2 is a 1 to 6 carbon atom alkyl group.

Useful Q^1 and Q^2 substituents include one to six carbon atom alkyl, preferably methyl, groups; halogens, preferably

chorine, fluorine or bromine, and substituents which form rings with two Q^1 or Q^2 carbon atoms.

Compounds having the formula $Q^1(Z)$ Q^2Li are prepared in known manner by reacting a compound of formula $Q^1(Z)_x$ Q^2 with an alkyl lithium compound, preferably n-butyl or *t*-butyl lithium in a non-interfering solvent, preferably ether or tetrahydrofuran. The lithiation reaction is appropriately conducted at a temperature of from about -80°C to about 40°C.

The reaction mixture which contains lithiated $Q^1-(X)_x$ Q^2 may be combined directly with $R^1_3SiO_3SR^2$ to yield $R^1_3SiQ^1(X)_x$ Q^2SiR_3. The reaction proceeds to substantial completion in about thirty minutes at room temperature. See Example 8. Alternatively, the lithium salt may be isolated prior to reaction with $R^1_3SiO_3SR^2$.

DETAILED DESCRIPTION OF THE INVENTION

The Formula (I) silanes may be prepared by reacting a compound of the formula R^1SO_3H, in which R^1 is any straight or branched chain alkyl group preferably having one to eight carbon atoms, with a compound of the formula X_2SiQ_2, in which X and Q are as defined.

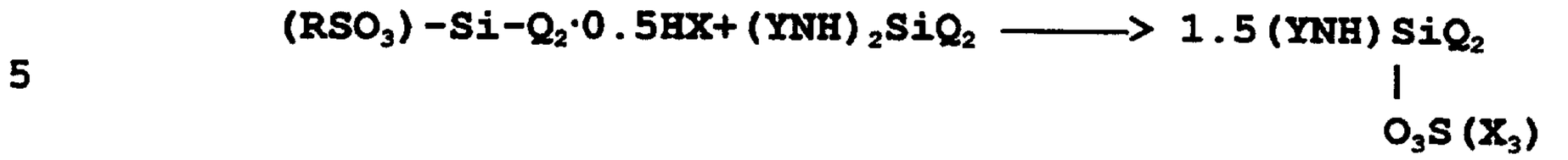
The synthesis of one Formula (I) silane is illustrated by Equation 1:

$$2RSO_3H + X_2SiQ_2 \xrightarrow[\text{Heat}]{RT} (R^1SO_3)_2\text{-Si-}Q_2\cdot 0.5HX + 1.5HX \qquad (1)$$

RT = Room Temperature.

The novel Formula (II) silanes are synthesized by reacting RSO_3H with a compound having the formula $(YNH)_2SiQ_2$,

in which R and Q are as defined, and Y is an alkyl group which may be the same as or different from Q. See Equation 2:

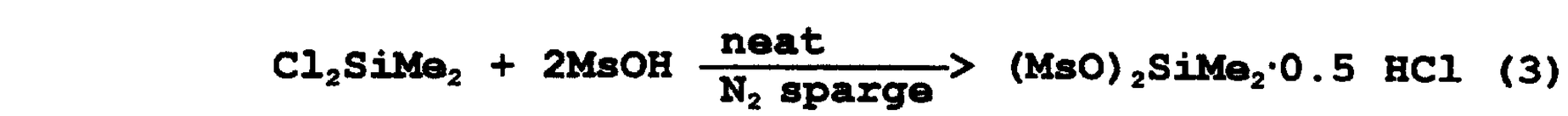


$$(RSO_3)\text{-}Si\text{-}Q_2\cdot 0.5HX + (YNH)_2SiQ_2 \longrightarrow 1.5\,(YNH)SiQ_2 \quad (2)$$
$$\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\quad |$$
$$\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\qquad\quad O_3S(X_3)$$

EXAMPLE 1

Preparation of Formula I Silane - $(MSO)_2SiMe_2\cdot 0.5$ HCl

$[(CH_3O_3S)_2Si(CH_3)_2\cdot 0.5HCl]$. To a 500 mL flask containing neat Cl_2SiMe_2 (64 g., 0.50 mol) was added MsOH (97 g., 1.01 mol); the immiscible solution rapidly evolved HCl that was scrubbed with NaOH (250 g., 50 wt% solution) or with iced water. After the solution was stirred overnight, the homogeneous oil was sparged with N_2 gas an additional day. This synthesis is illustrated by Equation 3:



$$Cl_2SiMe_2 + 2MsOH \xrightarrow[N_2\ \text{sparge}]{\text{neat}} (MsO)_2SiMe_2\cdot 0.5\ HCl \quad (3)$$

^1H NMR analysis of the product showed that one equivalent of HCl was present with two equivalents $(MSO)_2SiMe_2$; yield is quantitative.

EXAMPLE 2

Preparation of a Formula (II) Silane - $MsOSiMe_2$ | $NH(t\text{-butyl})$

(a) Preparation of $(t\text{-BuNH})_2SiMe_2$ (equation 4).

A 12 L flask equipped with an additional funnel and reflux condenser was charged with $t\text{-BuNH}_2$ (11 mol, 805 g) and THF (7 L). The solution was slowly treated with Cl_2SiMe_2

(5 mol, 645 g) that resulted in an endothermic reaction. After the temperature had dropped to 40°C, the white slurry was filtered, the *t*-$BuNH_3Cl$ was washed with THF (1 L), and the filtrate was reduced to an oil that contained 97% pure $(t\text{-BuNH})_2SiMe2$ (^1H NMR). Yield was quantitative (1 Kg). See equation 4.

$$Cl_2SiMe_2 + 4\ t\text{-}BuNH_2 \xrightarrow{THF} (t\text{-BuNH})_2SiMe_2 + 2\ t\text{-}BuNH_3Cl \quad \text{(quantitative)} \qquad (4)$$

(b) Preparation of $MsOSi(CH_3)_2$ | $NH(t\text{-butyl})$

The $(t\text{-BuNH})_2SiMe_2$ prepared as described in Example 2(a) was added to one equivalent of neat $(MsO)_2SiMe_2 \cdot 0.5HCl$ at room temperature, resulting in a 50-60°C exotherm. The resulting oil which contained insoluble solids was filtered through a glass frit to give >98% pure $(t\text{-BuNH})(MsO)SiMe_2$ (^{1}NMR). See equation 5.

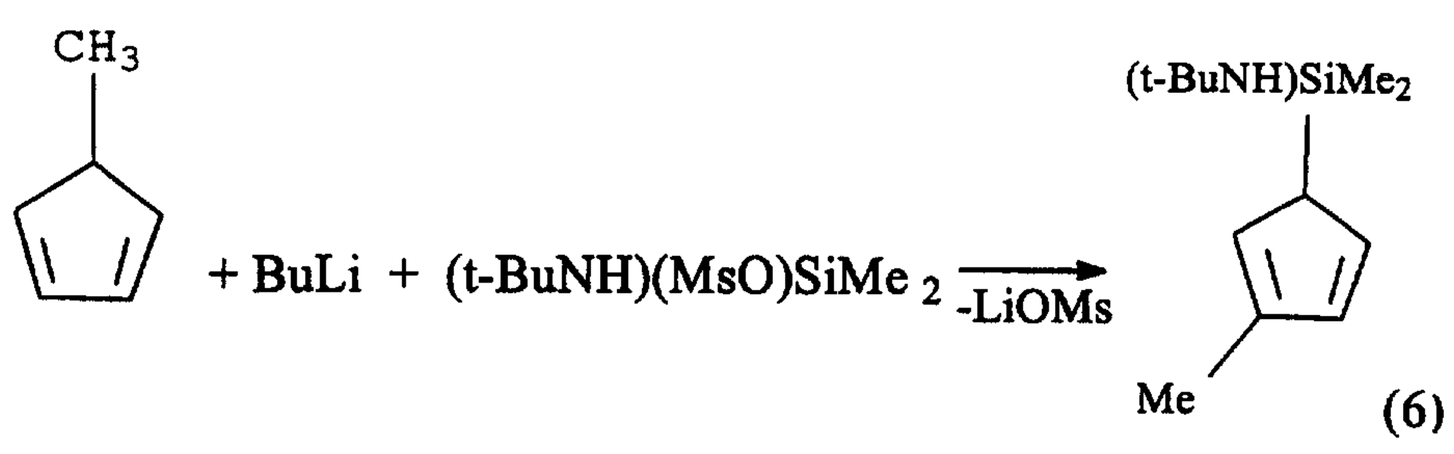
$$(MsO)_2SiMe_2 \cdot 0.5HCl + (t\text{-BuNH})_2SiMe_2 \xrightarrow{neat} 1.5\,(MsO)Si(Me)_2 \mid NH(t\text{-butyl}) \qquad (5)$$

EXAMPLE 3

Formula (II) Silane - $CF_3O_3S\text{-}Si(Me_3)_2$ | $NH(t\text{-butyl})$ was prepared as described in Example 2 (Equation 5) except that $(TfO)_2Si(Me)_2$ replaces $(MsO)_2SiMe_2 \cdot 0.5$ HCl.

EXAMPLE 4

Preparation of 2-Methylcyclopentadienyl(*t*-Butylamido) Dimethylsilane (Equation 6). A 1 L flask was charged with 2-methylcyclopentadiene (16 g, 200 mmol) and THF (160 g). The solution was cooled (-10°C) and treated with n-BuLi (1.6 M, 125 mL, 200 mmol). After the resulting white heterogeneous solution was stirred at room temperature for thirty minutes, the solution was treated with (*t*-BuNH)(MsO)$SiMe_2$ (47 g, 190 mmol) and the solution was stirred overnight. The solution was filtered through Celite, the residual LiOMs was washed with ether (500 mL), and the filtrate was reduced to a light-green oil. No further purification was necessary. Yield was quantitative.



(6)

In this example, 2-methylcyclopentadiene may be replaced by cyclopentadiene to provide a quantitative yield of cyclopentadienyl (*t*-butyl amido) dimethylsilane.

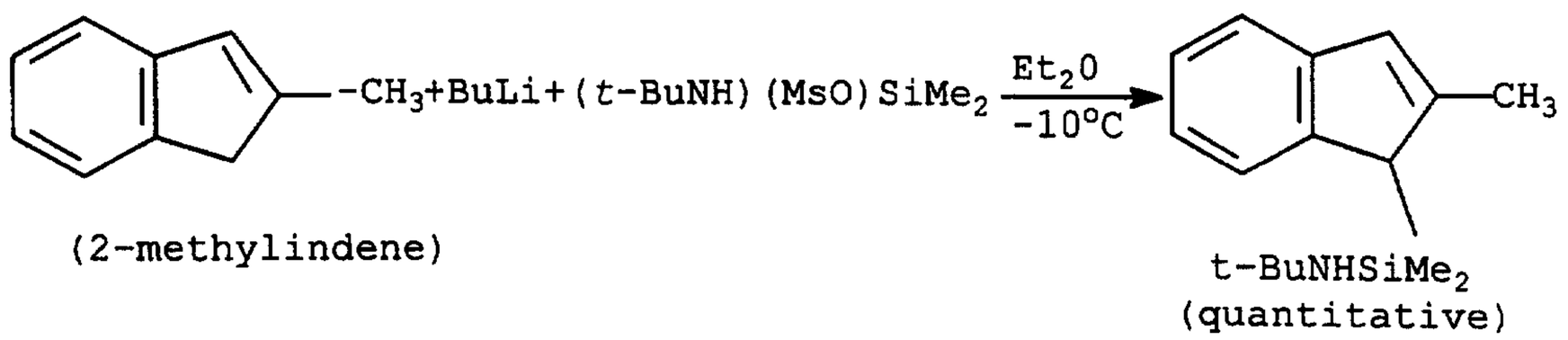
Also in this example, 2-methylcyclopentadiene may be replaced by 3-methyl-2-ethyl-cyclopentadiene to provide a quantitative yield of 3-methyl-2-ethyl-cyclopentadienyl (*t*-butyl amido) dimethylsilane.

Also in this example, *t*-BuNHTfOSi(Me)$_2$ may be used with similar results.

This example illustrates a method in which a type II silane is added directly to the reaction mixture in which an alkali metalide is formed. Alternatively, the alkali metalide, here lithium-2-methylcyclopentadiene, may be isolated from the reaction mixture in known manner and thereafter reacted with either a type I or type II silane.

EXAMPLE 5

Preparation of 2-Methylindenyl(*t*-Butylamido) Dimethylsilane (Equation 7). A 5 L flask was charged with 2-methylindene (1.67 mol, 217 g) and ether (1.5 L). The solution was cooled (-10°C) and treated with BuLi (1.67 mol, 1.04L). After the solution was stirred for one hour at room temperature, the solution was cooled (-10°C) and $Me_2Si(MsO)NH(t\text{-}Bu)$ (a type II silane) was added in one portion, resulting in a 20°C exotherm. After one hour at room temperature, the solution was filtered through Celite, the residual solid LiOMs was washed with ether (1.5 L), and the filtrate was reduced to a yellow oil that contained >98% pure 2-methylindenyl(*t*-butylamido) dimethylsilane (^{1}H NMR) in quantitative yield.



(7)

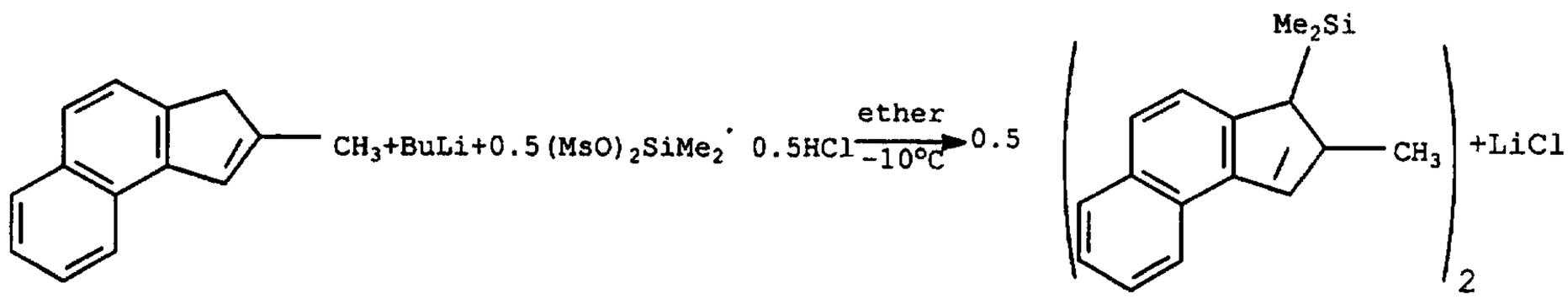
In this example, (*t*-buNH)TfOSiMe$_2$ may be used instead of *t*-BuNH(MsO)SiMe$_2$.

Also, in this example, 2-methylindene may be replaced with fluorine to provide a quantitative yield of 9-fluorenyl-*t*-butyl amido dimethyl silane.

Also, in this example, 2-methylindene may be replaced with bromobenzene to obtain a quantitative yield of the expected phenyl-*t*-butylamido dimethyl silane.

EXAMPLE 6

Preparation of bis(2-methyl-benzoindenyl) dimethylsilane (equation 8). A 2L flask charged with 2-methyl-benzo(indene) (73 g, 405 mmol) and ether (500 mL) was cooled to -10°C and treated with n-BuLi (1.6 M, 255 mL, 405 mmol). The solution was allowed to warm to room temperature for 30 minutes, cooled to about -10°C, and then treated with a neat Formula I silane (MsO)$_2$SiMe$_2$-0.5 HCl (54g, 203 mmol) resulting in a 10-15°C exotherm. After one hour at room temperature, the white slurry was treated with CH_2Cl_2 (500 mL), and the solution was filtered through Celite into a 5L flask. The solids were washed with CH_2Cl_2 (500 mL), and the filtrate was evacuated to dryness. The white solid residue was treated with ether (200 mL), and the solvent was evacuated so that most of the residual CH_2Cl_2 was removed. The solid was then treated with ether (1 L) and triturated for thirty minutes before filtering and washing the white solid with ether (200 mL). Yields vary from 20-50%. The 2-methyl-benzo(indene) was recovered by treatment of the filtrate with NaOH (20 wt%) in THF.



2-methylbenzo(indene)

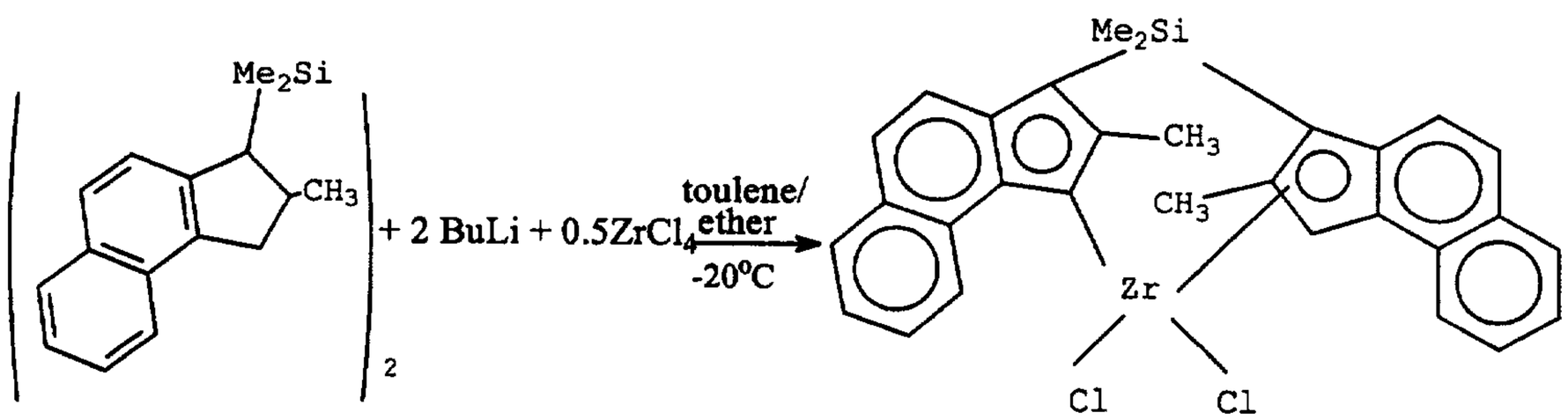
(8)

The above procedure was repeated, except that $(MSO)_2SiMe_2 \cdot 0.5HCl$ was replaced with $TfO_2Si(Me)_2$. The yield of bis(2-methyl-benzoindenyl) dimethylsilane was 60-65% of the bis(2-methylbenzoindenyl) dimethylsilane.

EXAMPLE 7

Preparation of Metallocene Catalyst from the Example 5 Product (Equation 9). A 1 L flask was charged with bis(2-methyl-benzoindenyl) dimethylsilane (48 g, 115 mmol), toluene (480 mL), and ether (20 g, 270 mmol). The solution was cooled (-10°C) and then treated with BuLi (1.6 M, 145 mL, 230 mmol). After the tanned-colored heterogeneous solution was stirred at room temperature for two hours, the solution was cooled (-20°C) and treated with $ZrCl_4$ (27 g, 115 mmol). By the time the solution had warmed to -10°C, a bright yellow solution had resulted. After the yellow solution was stirred at room temperature for 2 hours, the solution was filtered, and the yellow solid was washed with toluene until the filtrate was pale yellow. The yellow filter cake was treated with an equal mass of Celite, the solids were slurried in dry CH_2Cl_2, and the product was extracted with CH_2Cl_2 through a layer of Celite into a 12 L

flask that contained toluene (1 L); the extraction was stopped when the yellow color of the filtrate had turned translucent. The CH_2Cl_2 solvent was evaporated to give a toluene-slurry of yellow crystals. The solution was filtered, the yellow crystals were washed with toluene (1 L), and the yellow solid was slurried in toluene (5 L) for four hours. The solution was filtered to give 28 grams of diastereomerically pure metallocene (^1H NMR; yield - 38%).

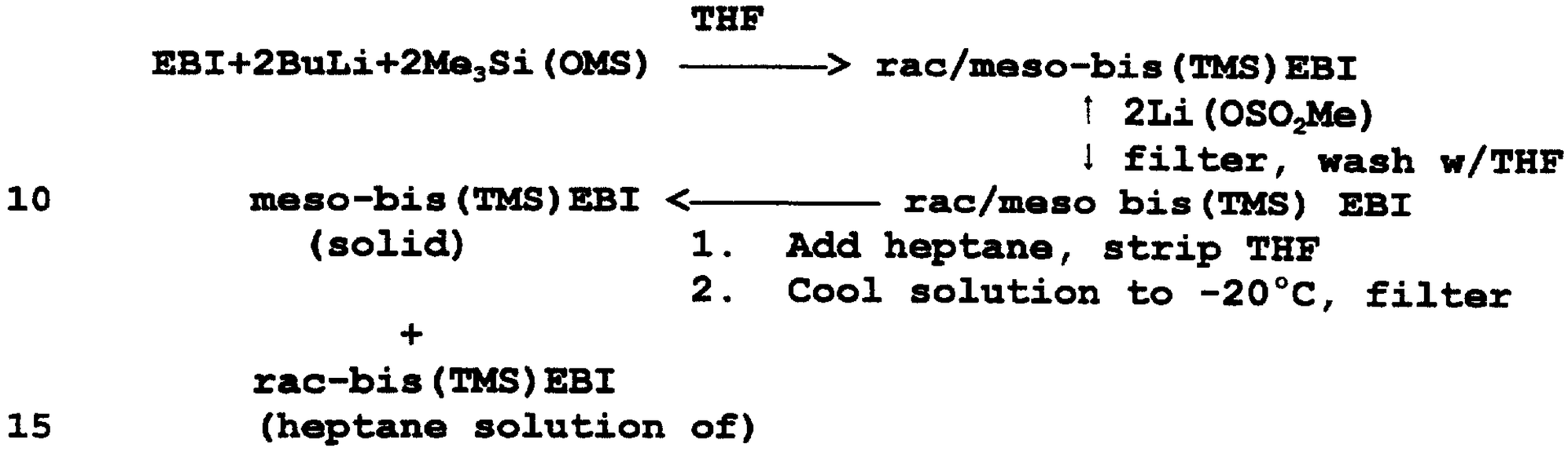


EXAMPLE 8

Preparation of Bis(3-Trimethylsilyl (TMS) indenyl) ethane (Equation 10). A 1L flask was charged with ethylene bis-indene (EBI) (0.100 mol, 26g) and THF (260g). The solution was cooled (-10°C) and treated with BuLi (0.200 mol. 125 mL). After one hour at RT, the solution was cooled (-10°C) and treated with Me_3Si(OMS) (0.200 mol., 34 g) in one portion. After thirty minutes at RT, the solution was filtered through Celite, the solids containing rac/meso bis(TMS) EBI were washed with THF (130 g), and the filtrate

was reduced giving a solid that contained 98% rac-meso product in >98% yield. The product was extracted with heptane to separate the rac and meso isomers.

This procedure is illustrated by the following equation 10:

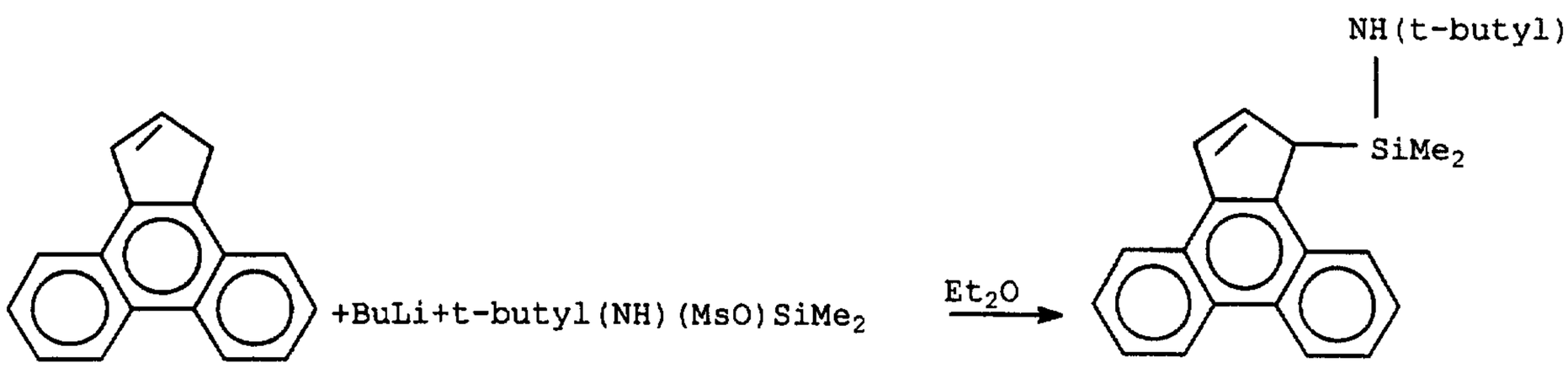


The above procedure was repeated with several analogs of EBI with similar results. Specific analogs of EBI were bis(2-methylindenyl) ethane, bis(4,7-dimethylindenyl) ethane, cyclopentadiene and methylcyclopentadiene. In this example, $Me_3Si(OTf)$ may be used instead of $Me_3Si(OMs)$.

EXAMPLE 9

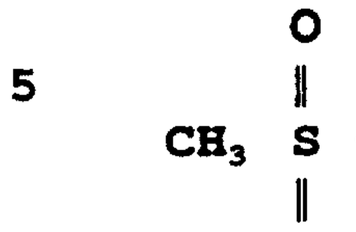
Preparation of N-Silylated Cyclopentaphenanthrene.

This procedure is illustrated by equation 11:



FW	816	1.6M	223	345
	32.1g	92.9ml	33.2g	Quantitative
	0.1486	0.1486	0.1486	1 HNMR

Cyclopentaphenanthrene is mostly dissolved in diethyl ether (800 mL), n-BuLi is added, and the reaction mixture was stirred overnight.



was added neat, followed by stirring for one-half hour. The reaction mixture was filtered. Ether was removed. Yield - quantitative. In this reaction, any compound of Formula (II), page 5, may be used instead of *t*-butyl $NHMsOSiMe_2$. Compounds obtaining the corresponding R groups instead of *t*-butyl are produced.

In this example, $CF_3SO_3Si(CH_3)_2NH$ *t*-butyl may be used instead of $CH_3SO_3SI(CH_3)_2NH$ *t*-butyl.

I CLAIM:

1. A compound having the formula I

$(CX_3SO_3)_2SiQ_2$

or the formula II

$(CX_3SO_3)_2SiQ$
|
NHZ

in which X is H or F, each Q in formula (I) may be the same or a different alkyl or aryl hydrocarbyl group with the proviso that when X is F in formula (I), Q is not methyl, and Z is an alkyl or aryl hydrocarbyl group which may be the same or different from Q.

2. A claim 1 compound in which each Q is an alkyl group having one to eight carbon atoms.

3. A claim 1 compound in which X is H and Z is a tertiary butyl group.

4. The compound $(CH_3SO_3)_2Si(CH_3)_2 \cdot 0.5HCl$.

5. The compound $(CF_3SO_3)_2Si(CH_3)_2$
|
NH(*t*-butyl)

6. The compound $(CH_3SO_3)_2Si(CH_3)_2$
|
NH(*t*-butyl).

7. A method for preparing a silylated or N-silylated mono- or bis- cyclopentadienyl or indenyl compound which comprises reacting a lithiated mono or bis cylopentadienyl or indenyl compound with a claim 1 compound of Formula I or Formula II.

8. A method for preparing an N-silylated mono- or bis- cyclopentadienyl or indenyl compound which comprises

reacting a lithiated mono- or bis- cyclopentadienyl or indenyl compound with a claim 6 compound.

9. A method which comprises:

(i) reacting a compound including a -CH group with a compound having the formula RM in which R is any hydrocarbyl group and M is an alkali metal in a non-interfering solvent

wherein a reaction mixture containing a compound having a -CM group in said solvent is produced;

(ii) reacting a metallocene ligand with said compound having the formula -CM produced in step (i)

wherein a reaction mixture containing an alkali metallide of said metallocene ligand is produced; and

(iii) reacting said alkali metallide of said metallocene ligand with a compound of Formula (I) or Formula (II).

10. The claim 9 method wherein said step (i) compound RM is an alkyl lithium compound.

11. The claim 9 or claim 10 method wherein said step (iii) is performed by adding said formula (I) or formula (II) compound to said step (ii) reaction mixture.

12. The claim 9 or claim 10 method further comprising a step (ii)(a), wherein said alkali metallide is separated from said step (ii) reaction mixture prior to step (iii).

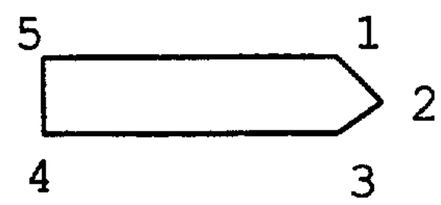
13. The claim 9 or claim 10 method wherein said step (ii) metallocene ligand is

(i) a monocyclopentadienyl ligand having the formulae $C_5H_xR_y$, wherein:

x = 0-5

y = 0-5

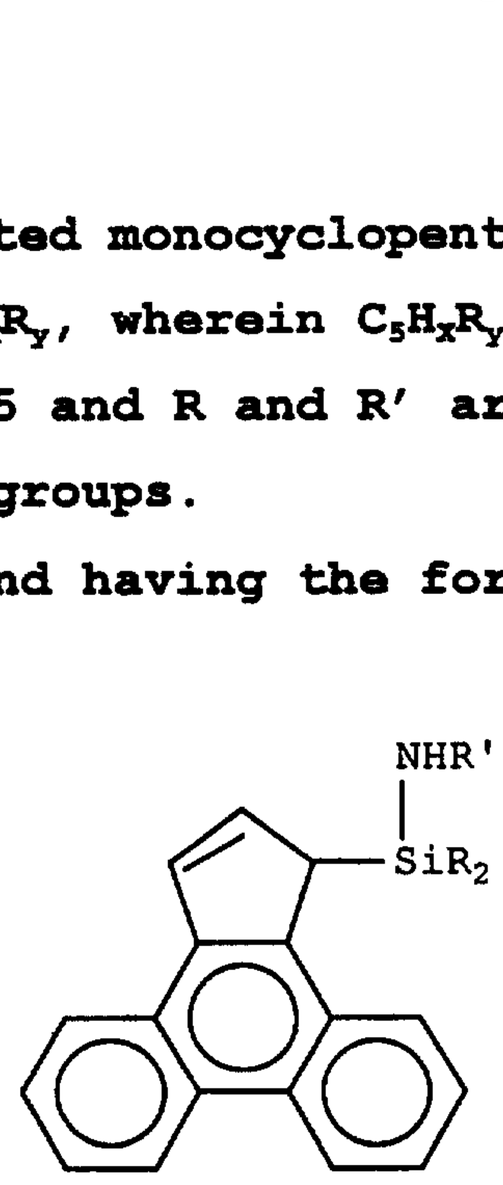
R = any aryl or aromatic group and H or R can occupy any of the positions 1 to 5 of the formula



or

(ii) a silylated monocyclopentadienyl ligand having the formulae $(R_3Si)_zC_5H_xR_y$, wherein $C_5H_xR_y$ is as defined in definition 5, Z=1-5 and R and R′ are identical or different alkyl or aromatic groups.

14. A compound having the formula



in which R and R^1 are the same or different alkyl or aromatic groups.

15. A claim 14 compound in which R^1 is a t-butyl group.