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Enantioselective addition of phenylacetylene to aldehydes catalyzed by silica-immobilized titanium(IV) complex of β-hydroxyamide

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Abstract

A chiral β-hydroxyamide was synthesized from L-phenylalanine and successfully grafted onto amorphous silica gel. The silica-immobilized ligand was characterized by FT-IR, solid state NMR and elemental analysis. This is the first example of asymmetric addition of phenylacetylene to aldehydes catalyzed by silica-immobilized titanium(IV) complex of β-hydroxyamide with high yields (up to 95%) and good enantioselectivities (up to 81% ee). The catalyst could be reused up to five times without serious loss of enantioselectivity.

Keywords: Enantioselective addition; Titanium tetraisopropoxide; Silica-immobilized catalyst; Diethylzinc; β-Hydroxyamide

1. Introduction

Optically active propargyl alcohols are important building blocks for the synthesis of many organic compounds [1]. The first effective asymmetric alkynyl addition to aldehydes was demonstrated by Corey and Cimprich [2]. The catalytic enantioselective addition of terminal alkynes to aldehydes has recently generated enormous interest [3–11]. Many chiral ligands, such as *N*-methylephedrine [12–14], BINOL and its derivatives [5,10,15–20] and sulfonamides [21,22] have been used successfully in this reaction. Other chiral ligands, such as amino alcohols [23,24], oxazoline [25,26] and imino alcohol [27], have also been reported to catalyze this reaction.

The heterogeneous asymmetric catalytic systems, which have an inherent advantage of easy handling, separation and facilitation of industrial application, have been used successfully in a lot of reactions [28–30]. However, there have been far fewer reports on asymmetric catalytic reactions [31] using inorganic supported catalysts, which have many advantages over most polymers supported ones for their superior mechanical and thermal stability. In recent years, silica-immobilized ligands have been successfully used for reactions, such as asymmetric addition of diethylzinc to aldehydes [32–36], asymmetric transfer hydrogenation [37], asymmetric Diels–Alder reaction [38–40] and asymmetric Henry reaction [41]. To our knowledge, there is no report of silica-immobilized catalysts for asymmetric alkynylation reactions.

Recently, our group has developed a new β-hydroxyamide chiral ligand **1a**, and successfully introduced it into the asymmetric addition of phenacetylene to aldehydes to obtain excellent enantio-selectivities (up to 97% ee) [42]. With our continuing efforts towards the development of recyclable silica-immobilized ligands [43], we immobilized β-hydroxyamide **1a** on amorphous silica gel to afford silica-immobilized ligand **5**. Enantioselective addition of phenylacetylene to aldehydes

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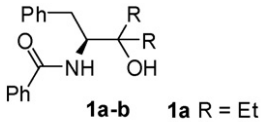
catalyzed by silica-immobilized titanium(IV) complex of β-hydroxyamide **5** was carried out, affording high yields (up to 95%) and good enantioselectivities (up to 81%).



1a-b **1a** R = Et **1b** R = Ph

2. Experimental

2.1. General

Melting points were determined using an X-4 melting point apparatus and were uncorrected. Optical rotations were measured with a Perkin-Elmer 341 polarimeter at 18 °C in CH_2Cl_2. ^1H NMR and ^{13}C NMR spectra were recorded on a Varian Mercury-400 MHz spectrometer with TMS as an internal standard. The solid-state ^{13}C NMR experiment was performed on a Bruker AV400 WB solid-state NMR instrument at 100 MHz. IR spectra were obtained on a Nicollet NEXUS 670 FT-IR spectrometer. HRMS data were measured with ESI techniques (Bruker Apex II). Elemental analyses were performed on an Elementar vario EL appararus. The pore sizes and surface areas were determined on a Micromeritics ASAP 2010 system. Enantiomeric excess values were determined by HPLC with a Chiralcel OD-H column. All catalytic reactions were carried out under nitrogen atmosphere.

2.2. Reagents and solvents

L-Phenylalanine was purchased form Alfa Aesar. 3-Mercaptopropyltrimethoxysilane and phenylacetylene were bought from ABCR GmbH & CoKG and $Ti(O^iPr)_4$ from Acros. Diethylzinc (1 M solution in dichloromethane), trimethylsilylimidazole (TMSIm) and 4-(bromomethyl)benzoyl bromide were synthesized according to literature methods [44–46], respectively. Amorphous silica gel (160–200 mesh) was subjected to heat treatment at 150 °C for 3 h and cooled under nitrogen prior to use. Dichloromethane was freshly distilled from phosphorous pentoxide. Toluene, hexane and diethyl ether were freshly distilled from a deep-blue solution of sodium-benzophenone under nitrogen. $Ti(O^iPr)_4$ was distilled under nitrogen prior to use.

2.3. Synthesis of chiral ligands

Compounds **2** and **4** were synthesized according to literature procedures [47,35], respectively.

2.3.1. 4-(Bromomethyl)-N-[(S)-3-ethyl-3-hydroxy-1-phenylpentan-2-yl]benzamide (*3*)

A solution of 4-(bromomethyl)benzoyl bromide (10 mmol) in CH_2Cl_2 (20 mL) was added to a cold (0 °C) solution of amino alcohol **2** (10 mmol) and *N*,*N*-diisopropylethylamine (DIEA) (10 mmol) in CH_2Cl_2 (20 mL). The reaction mixture was allowed to warm to room temperature and stirred for 12 h. After washing sequentially with 1 M HCl, 5% $NaHCO_3$ aqueous solution and saturated brine, the organic layer was dried over anhydrous $MgSO_4$, concentrated under vacuum and recrystallized from ethyl acetate (EA)/hexane to afford benzamide **3** as white solid (yield 82%). m.p. 149–151 °C. $[\alpha]_D^{18} = -141$ (c 1.00, CH_2Cl_2). ^1H NMR (400 MHz, $CDCl_3$) δ: 0.91 (t, J = 7.6 Hz, 3H, CH_3), 0.97 (t, J = 7.6 Hz, 3H, CH_3), 1.53–1.77 (m, 4H, CH_2), 2.84 (dd, J = 14.2, 10.2 Hz, 1H, $PhCH_2$), 3.14 (dd, J = 14.2, 4.0 Hz, 1H, $PhCH_2$), 4.28–4.34 (m, 1H, CH), 4.45 (s, 2H, $PhCH_2Br$), 6.19 (d, J = 8.8 Hz, 1H, NH), 7.15–7.25 (m, 5H, ArH), 7.36 (d, J = 8.0 Hz, 2H, ArH), 7.47 (d, J = 8.8 Hz, 2H, ArH). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 7.66, 8.00, 27.78, 28.11, 32.27, 35.14, 56.58, 126.33, 126.59, 127.28, 128.37, 129.00, 129.11, 134.60, 138.74, 140.84, 167.35. IR (KBr): 3455, 3355, 2962, 2928, 2876, 1627, 1536, 1450, 1259, 1130, 983, 931, 741, 693 cm^{-1}. HRMS (ESI): $M+Na^+$, 426.1039; found: 426.1039. Anal. calculated for $C_{21}H_{26}BrNO_2$: C, 62.38; H, 6.48; N, 3.46. Found: C, 62.56; H, 6.58; N, 3.12.

2.3.2. Synthesis of silica-immobilized ligand *5*

Compound **4** (7.5 g) was suspended in dry toluene (30 mL) under nitrogen. DIEA (1.51 mL, 15 mmol) and benzamide **3** (6.07 g, 15 mmol) were added and the mixture was stirred at 70 °C for 12 h. After washing with toluene (100 mL) and the mixture of CH_2Cl_2 and CH_3OH (1:1, 100 mL), the solid was suspended in the mixture solvent (CH_2Cl_2:CH_3OH = 1:1, 50 mL) and stirred for 12 h. After filtration and thorough wash with CH_2Cl_2 (50 mL), hexane (50 mL), CH_3OH (50 mL), acetone (50 mL) and CH_2Cl_2 (50 mL), the solid was dried at 110 °C in vacuum for 48 h to give the immobilized ligand **5** as a yellowish powder. IR (KBr): 3436, 2967, 2859, 1630, 1608, 1501, 1449, 1187, 1099, 952, 845, 805, 759, 697, 468 cm^{-1}. Anal. found: C, 12.26; H, 2.09; N, 0.29. Average pore diameter: 6.3 nm. S_{BET}: 282 m^2/g.

2.4. General procedure for the asymmetric addition of phenylacetylene to aldehydes

Method A: Under dry nitrogen, the silica-immobilized ligand (0.04 mmol) and $Ti(O^iPr)_4$ (0.12 mmol) were mixed in solvent (1.0 mL) at room temperature and stirred for 1 h. Then a solution of $ZnEt_2$ (1.0 M in CH_2Cl_2) was added. After the mixture was stirred at room temperature for 2 h, phenylacetylene was added and stirred for another 1 h and the solution was treated with aldehyde (0.2 mmol). After the reaction was completed (TLC), the reaction solution was cooled to 0 °C and quenched by 0.5 M aqueous HCl. The mixture was extracted with diethyl ether (3 × 10 mL), washed with brine (3 × 15 mL), dried with anhydrous Na_2SO_4 and concentrated under vacuum. The crude product was purified by column chromatography (silica gel, petroleum ether (PE:EA = 8:1) to give the propargyl alcohol.

Method B: According to a modified procedure of Pu's [17], phenylacetylene (0.6 mmol) and diethylzinc (0.6 mmol, 1 M in CH_2Cl_2) were added to a flask containing dry toluene (1 mL). The solution was refluxed for 5 h. It was then transferred into the suspension of silica-immobilized ligand **5** (0.04 mmol) in

toluene (0.5 mL). After the mixture was stirred at room temperature for 15 min, $Ti(O^iPr)_4$ (0.12 mmol) was added and the stirring continued for another 1 h. Benzaldehyde (0.2 mmol) was added, and the reaction mixture was stirred for 12 h. The reaction was quenched by 0.5 M aqueous HCl. The mixture was extracted with diethyl ether (3 × 10 mL), washed with brine (3 × 15 mL), dried with anhydrous Na_2SO_4 and concentrated under vacuum. The crude product was purified by column chromatography (silica gel, PE:EA = 8:1) to give the propargyl alcohol.

2.5. General procedure for the restoration of silica-immobilized ligand 5

After one catalytic cycle, the silica-immobilized ligand **5** was washed sequentially with 0.5 M HCl (30 mL), H_2O (15 mL), MeOH (15 mL), the mixture of MeOH and CH_2Cl_2 (1:1, 30 mL) and CH_2Cl_2 (30 mL) and dried under vacuum at 110 °C for 18 h and 60 °C for 50 h. The catalyst was used for the next catalytic cycle.

3. Results and discussion

3.1. Synthesis and characterization of silica-immobilized β-hydroxyamide

The silanol groups on amorphous silica gel are unsuitable for the enantioselective addition of phenylacetylene to aldehydes. It is necessary to "protect" the Si–OH groups. Here we chose Seebach's strategy [35] to immobilize our β-hydroxyamide **1a** onto amorphous silica gel. As shown in Scheme 1, reaction of 4-(bromomethyl)benzoyl bromide [45] with the amino alcohol **2**, which was synthesized from L-phenylalanine according to literature methods [44], in the presence of DIEA afforded β-hydroxyamide **3**. The "silanol protected" supported **4** was synthesized through four steps by literature methods [35]. Finally, chiral compound **3** was allowed to react with **4** in the presence of DIEA at 70 °C for 24 h to give silica-immobilized ligand **5**.

FT-IR was first employed for the characterization of the silica-immobilized ligands. As shown in Fig. 1, the aliphatic C–H bonds (2962, 2846 cm^{-1}) and the absorption bands at 1605, 1497 and 1454 cm^{-1} due to the C=O and C=C stretching vibration of the immobilized β-hydroxyamide could be seen.

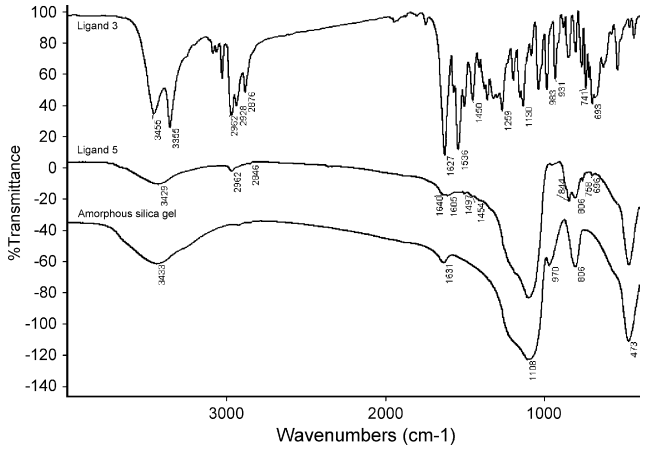


Fig. 1. IR spectra of ligand **3**, ligand **5** and amorphous silica gel.

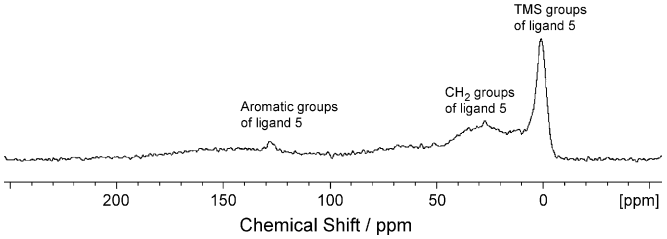
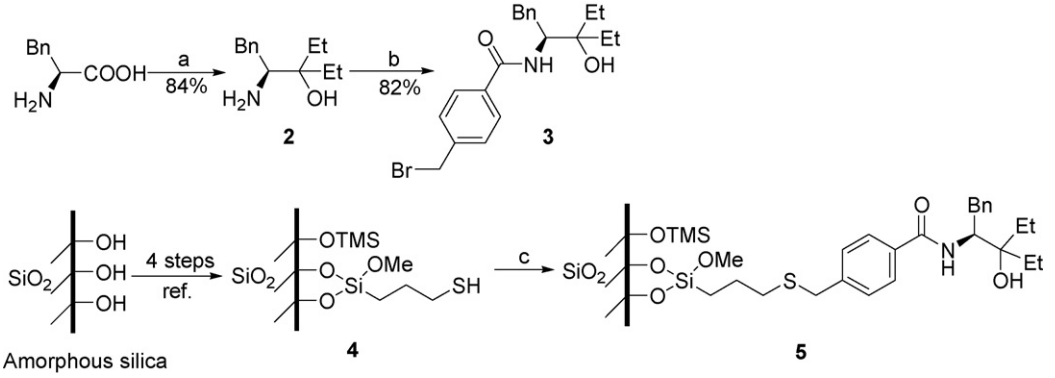


Fig. 2. ^{13}C MAS NMR spectrum of silica-immobilized ligand **5**.

Solid-state NMR is a powerful tool for the characterization of silica-immobilized ligands. As shown in Fig. 2, the peaks in the range of 125–130 ppm are due to the aromatic groups on silica-immobilized ligand **5** and the peaks in the range of 20–40 ppm due to the CH_2 groups bridging the chiral ligand and silica walls. The high peak at 0 ppm demonstrated the TMS groups capped on the silica walls (Fig. 2).

As can be seen from Table 1, silica-immobilized chiral ligand **5** presented a decrease in the surface area (S_{BET}) than the amorphous silica support, which could be attributed to the presence of chiral ligands and TMS groups on the silica walls that partially block the adsorption of nitrogen [32]. The amount of chiral



Scheme 1. Synthesis of silica-immobilized chiral ligand **5**. Reagents and conditions: (a) (i) $SOCl_2$, methanol, 0 °C–reflux, 3 h; (ii) EtMgBr, Et_2O, r.t., 12 h; (b) 4-(bromomethyl)benzoyl bromide, DIEA, CH_2Cl_2, r.t., 12 h; (c) **3**, DIEA, toluene, 70 °C, 24 h.

Table 1
Characterization of amorphous silica gel and silica-immobilized chiral ligand **5**

Compound	Surface area (m^2/g)[a]	Average pore diameter (nm)[a]	Content of **1a** (mmol/g)[b]
Silica gel	412	1.4	–
5	282	6.3	0.204

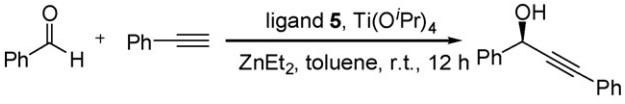
[a] The surface area and the average pore diameter were calculated by the BET method.
[b] Calculated from nitrogen analysis.

ligand grafted onto silica gel was calculated from elemental analysis of nitrogen.

3.2. Enantioselective addition of phenylacetylene to aldehydes

Silica-immobilized chiral ligand **5** was used to catalyze the asymmetric addition of phenylacetylene to benzaldehyde in the presence of $ZnEt_2$ and $Ti(O^iPr)_4$ to afford (*R*)-1,3-diphenylprop-2-yn-1-ol. We first increased the amount of chiral ligand **5** from 15 to 20 mol% and found that the ee value was increased from 66 to 75% (Table 2, entries 1 and 2). Further increase in the amount of chiral ligand did not afford better yield and ee value (Table 2, entry 3). This reaction was strongly influenced by the amount of $Ti(O^iPr)_4$. Either decreasing or increasing the amount of $Ti(O^iPr)_4$ dramatically decreased the enantioselectivities (Table 2, entries 2, 4 and 5). Other solvents, such as hexane, ether and CH_2Cl_2 were also tested in this reaction. The results showed that toluene is the best choice (Table 2, entries 2 and 6–8). In order to further improve the enantioselectivity, we ran the catalytic reaction by a modified procedure of Pu's [17]. Unfortunately, only 62% ee value and 50% yield were obtained (Table 2, entry 9). Decreasing the toluene from 1.5 to 1.0 mL slightly increased the ee value up to 78% (Table 2, entry 10). Compared with the results obtained from the homogenous systems, the ee value was remarkably low with the silica-immobilized ligand. It may be caused by the influence of Si–O–Si bond on the silica-immobilized ligand.

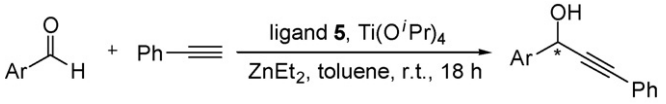
Table 2
Asymmetric addition of phenylacetylene to benzaldehyde catalyzed by ligand **5**[a]



Entry	Ligand (mol%)	$Ti(O^iPr)_4$/Ligand	Solvent (mL)	Yield (%)[b]	ee (%)[c]/config.[d]
1	**5** (15)	3/1	Toluene (1.5)	70	66 (*R*)
2	**5** (20)	3/1	Toluene (1.5)	75	75 (*R*)
3	**5** (25)	3/1	Toluene (1.5)	68	74 (*R*)
4	**5** (20)	2/1	Toluene (1.5)	60	52 (*R*)
5	**5** (20)	4/1	Toluene (1.5)	87	62 (*R*)
6	**5** (20)	3/1	Hexane (1.5)	75	72 (*R*)
7	**5** (20)	3/1	Et_2O (1.5)	73	57 (*R*)
8	**5** (20)	3/1	CH_2Cl_2 (1.5)	84	73 (*R*)
9[e]	**5** (20)	3/1	Toluene (1.5)	50	62 (*R*)
10	**5** (20)	3/1	Toluene (1.0)	93	78 (*R*)
11	**5** (20)	3/1	Toluene (0.75)	87	75 (*R*)

[a] Method A. Reaction temperature: r.t.; reaction time: 12 h.
[b] Isolated yields.
[c] Determined by HPLC with Chiralcel OD-H column.
[d] Absolute configuration was assigned by comparison to literature value.
[e] Method B.

Table 3
Asymmetric addition of phenylacetylene to aldehydes catalyzed by ligand **5**[a]



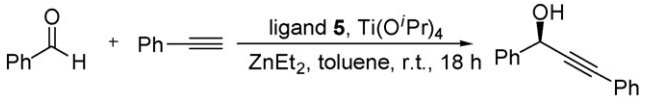
Entry	Aldehydes	Yield (%)[b]	ee (%)[c]
1	Benzaldehyde	95	78
2	2-Chlorobenzaldehyde	87	69
3	3-Chlorobenzaldehyde	86	74
4	4-Chlorobenzaldehyde	84	72
5	4-Tolualdehyde	89	70
6	2-Naphthaldehyde	93	81

[a] Method A. Ligand **5**/$Ti(O^iPr)_4$/$ZnEt_2$/phenylacetylene/ArCHO = 0.2:0.6:3:3:1 mmol; solvent: toluene; reaction temperature: r.t.; reaction time: 18 h.
[b] Isolated yields.
[c] Determined by HPLC with Chiralcel OD-H column.

The optimized conditions were applied to the asymmetric addition of phenylacetylene to aromatic aldehydes. As summarized in Table 3, high isolated yields (84–95%) and good enantioselectivities (69–81%) were achieved by using silica-immobilized chiral ligand **5**. The best enantioselectivity (81% ee) was obtained in the alkynylation of 2-naphthaldehyde (Table 3, entry 6).

Reusability of silica-immobilized ligand **5** was checked by using benzadehyde as representative substrate, because it is one of the advantages of heterogeneous ligands than homogeneous ones. After one catalytic cycle, ligand **5** was recovered and dried for the next catalytic cycle. Only a small loss of enantioselectivity was observed after five runs (Table 4).

Table 4
Reusability of ligand **5** for the asymmetric addition of phenylacetylene to benzaldehyde[a]



Run	Yield (%)[b]	ee (%)[c]/config.[d]
1	95	78 (*R*)
2	91	75 (*R*)
3	89	77 (*R*)
4	88	76 (*R*)
5	86	75 (*R*)

[a] Method A. After the general procedure of asymmetric addition, silica-immobilized ligand **5** was washed sequentially with 0.5 M HCl (30 mL), H_2O (30 mL), MeOH (30 mL), the mixture of MeOH and CH_2Cl_2 (1:1, 30 mL) and CH_2Cl_2 (30 mL), then dried under vacuum 110 °C for 18 h and 60 °C for 50 h before reusability.
[b] Isolated yields.
[c] Determined by HPLC with Chiralcel OD-H column.
[d] Absolute configuration was assigned by comparison to literature value.

4. Conclusions

We have reported the first use of silica-immobilized titanium(IV) complex of β-hydroxyamide as a catalyst for the asymmetric addition of phenylacetylene to aromatic aldehydes. High yields (up to 95%) and good enantioselectivities (up to 81%) were successfully achieved. After the restoring procedure, silica-immobilized ligand **5** can be used in multiple runs (up to 5 times) without serious loss of enantioselectivity. The application of this silica-immobilized chiral ligand to other asymmetric reactions is underway.

Acknowledgements

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