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A Novel Bifunctional Ti(IV) Complex Catalyzed Asymmetric Silylcyanation of Aldehydes

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ABSTRACT: *A novel Lewis acid and Lewis base bifunctional Ti(IV) complex was formed in situ upon the treatment of ligand (R)-3,3′-bis(diphenylphosphinoyl)-BINOL with Ti(PrO-i)$_4$, which is proven to be an efficient catalyst in the asymmetric trimethylsilylcyanation of aldehydes. The corresponding cyanohydrins were obtained in high to excellent chemical yield (83–93%) albeit with moderate enantioselectivities (up to 51% enantiomeric excess).* © 2010 Wiley Periodicals, Inc. Heteroatom Chem 22:31–35, 2011; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20652

INTRODUCTION

Optically active cyanohydrins are important intermediates in organic synthesis for the synthesis of a variety of valuable classes of chiral compounds, such as α-amino acids, α-hydroxy carboxylic acids, β-amino alcohols, vicinal diols, and α-hydroxy ketones. Many efficient approaches have been reported for their preparation by biochemical and chemical methods [1–8]. In the latter case, the most important one is the asymmetric silylcyanation of aldehydes with trimethylsilyl cyanide catalyzed by a Lewis acid center (Ti^{V}, Al^{III}, V^{V}, La^{III}, Mg^{II}, and Li^{I}), in the presence of a chiral ligand, such as Schiff bases [9–18], dihydroxy compounds [19–21], bis-oxazolines [22,23], amides [24–31], and phosphorus compounds [32–36]. Lately, chiral Lewis bases coordinating to and activating silylated nucleophiles have been introduced into this field [32,37]. Therefore, it is rational to design a new bifunctional asymmetric catalyst consisting of Lewis acid and Lewis base moieties, which activate both electrophiles and nucleophiles at defined positions simultaneously. Using this concept, we report here the titanium complex formed in situ upon the treatment of ligand (*R*)-3,3′-bis(diphenylphosphinoyl)-BINOL **(L1)** as a novel bifunctional catalyst in asymmetric trimethylsilylcyanation of aldehydes. We envisioned that the titanium would work as a Lewis acid to activate the carbonyl group, and the oxygen atom of the phosphine oxide would function as a Lewis base to activate the silylated nucleophiles.

RESULTS AND DISCUSSION

Following Ishihara's procedure [38], (*R*)-3,3′-bis(diphenylphosphinoyl)-BINOL (**L1**) was prepared from commercially available (*R*)-BINOL in two steps (Scheme 1). (*R*)-BINOL in THF was treated with NaH (2.2 equiv) followed by the dropwise addition of diphenylphosphinic chloride (2.2 equiv), to give the corresponding phosphinate (*R*)-1 quantitatively without further purification [39]. The rearrangement of (*R*)-1 upon treatment with lithium diisopropylamide (10 equiv) in THF at –78°C gave **L1** as a colorless crystal after being purified by column chromatography (200–300 mesh, gradient eluted with dichloromethane/ethyl acetate) [40].

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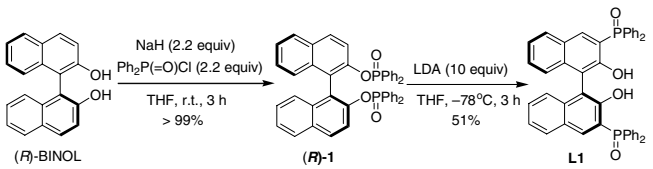
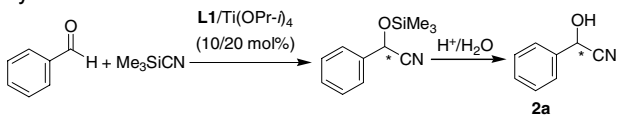


SCHEME 1 Preparation of **L1**.

With ligand **L1** in hand, the influence of solvents and temperature on the reaction was systematically investigated first by using the reaction of benzaldehyde and trimethylsilylcyanide as a model in the presence of 10 mol% **L1** and 20 mol% Ti(PrO-*i*)$_4$. The results are presented in Table 1.

As shown in Table 1, the Ti(IV)/**L1** catalytic system exhibited high catalytic activity to the trimethylsilylcyanation of benzaldehyde. The corresponding cyanohydrin **2a** was obtained in good to excellent yields for all the solvents tested (entries 1–4, 87–93%). However, the solvents had a notable effect on the enantioselectivity of the reaction. For example, (*R*)-mandelonitrile was attained with an enantioselectivity of 51% enantiomeric excess (ee) when the reaction was performed in THF (entry 4), whereas conducting the reaction in chloroform led to (*S*)-mandelonitrile as the major isomer almost with the same level of stereoselectivity (entry 3). Either employment of methylene chloride or DMF as the solvent resulted in total loss of stereocontrol to afford cyanohydrin **2a** in racemic form (entries 1 and 2). So we chose THF as the best solvent for this reaction in terms of yield and enantioselectivity. By

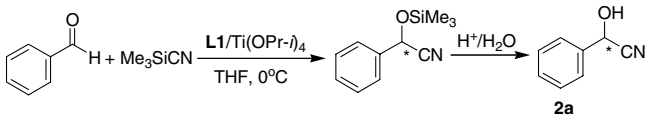
TABLE 1 Influence of Solvents and the Reaction Temperature on the Asymmetric Trimethylsilylcyanation of Benzaldehyde



Entry	*Solvent*	*Temperature (°C)*	*Yield (%)*[a]	*Enantiomeric Excess (%)*[b]	*Con.*[c]
1	CH_2Cl_2	0	93	4	*S*
2	DMF	0	87	0	–
3	$CHCl_3$	0	90	50	*S*
4	THF	0	91	51	*R*
5	THF	−40	88	0	–
6	THF	−20	90	32	*R*
7	THF	20	92	50	R

Con. = Configuration.
[a]Isolated yield.
[b]Determined by GC analysis of the acetate of the cyanohydrin on a chiral column.
[c]Determined by comparison of the sign of specific rotation value with the literature [12,16,41].

TABLE 2 Screening the Loading of Catalysts in the Asymmetric Trimethylsilylcyanation of Benzaldehyde Catalyzed by **L1** /Ti(IV)



Entry	***L1*** *(mol%)*	*Ti(IV) (mol%)*	*Yield (%)*[a]	*Enantiomeric Excess (%)*[b]	*Con.*[c]
1	10	20	91	51	*R*
2	5	10	87	27	*R*
3	20	40	89	42	*R*
4	30	60	90	35	*R*
5	10	10	83	14	*R*
6	10	40	86	27	*R*

Con. = Configuration.
[a]Isolated yield.
[b]Determined by GC analysis of acetate of the cyanohydrin on a chiral column.
[c]Determined by comparison of the sign of specific rotation value with the literature [12,16,41].

comparison of the parallel reactions carried out at different temperatures (20, 0, –20, –40°C) (entries 4–7), it is demonstrated that 0°C was the optimal reaction temperature for the reaction, which gave the highest enantioselectivity (51% ee, entry 4).

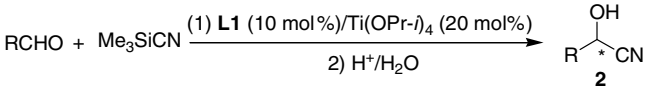
Other factors, such as catalyst loading and the mole ratio of ligand **L1** to Ti(OPr-*i*)$_4$ influencing the reaction, were also preliminarily investigated employing the reaction of benzaldehyde and trimethylsilylcyanide as the model; the results are presented in Table 2.

As shown in Table 2, although the catalyst loading demonstrates a slight influence on the yield of the cyanohydrin **2a**, either increasing or lowering the catalyst loading resulted in an obvious decrease in the enantioselectivity of the reaction. In addition, the mole ratio of Ligand **L1** to Ti(OPr-*i*)$_4$ was also found to be an essential factor for this reaction. By adjusting the mole ratio of **L1** to Ti(OPr-*i*)$_4$ from 1:2 to 1:4 or 1:1, both led to a dramatic decrease in enantiomeric excess of values of the product (entries 1, 5, and 6).

Based on the above-mentioned results, the reaction was best conducted in THF at 0°C in the presence of the Ti(IV) complex in situ formed from **L1** (10 mol%) and Ti(OPr-*i*)$_4$ (20 mol%). Under these optimal reaction conditions, the asymmetric trimethylsilylcyanation of a set of aldehydes was performed to examine the substrate generality of this reaction. The results are summarized in Table 3.

As shown in Table 3, the novel Ti(IV)/**L1** catalytic system demonstrated high efficacy in the transformation of aldehydes to cyanohydrins. In all cases,

TABLE 3 Asymmetric Trimethylsilylcyanation of Aldehydes Catalyzed by **L1**/Ti(OPr-*i*)$_4$



Entry	*R*	*Yield (%)*[a]	*Enantiomeric Excess (%)*[b]	*Con.*[c]
1 (**2a**)	C_6H_5	89	51	*R*
2 (**2b**)	4-ClC_6H_4	88	22	*R*
3 (**2c**)	2-$MeOC_6H_4$	93	11	*R*
4 (**2d**)	4-$MeOC_6H_4$	85	19	*R*
5 (**2e**)	2-MeC_6H_4	90	27	*R*
6 (**2f**)	3-MeC_6H_4	91	35	*R*
7 (**2g**)	2-Naphthyl	88	21	*R*
8 (**2h**)	$PhCH_2CH_2$	85	5	*R*
9 (**2i**)	2-Furyl	76	9	*R*

Con. = Configuration.
[a]Isolated yield.
[b]Determined by GC analysis of the acetate of the cyanohydrin on a β-DEX120 column.
[c]Absolute configuration was assigned by comparison of the sign of specific rotation value with the literature [12,16,41].

the corresponding products were obtained in good to excellent yield (76–93%). Whereas, the introduction of either electron-withdrawing (entry 2) or electron-donating substituents (entries 3–6) on the benzene ring disfavored the stereocontrol of the reaction and the ee values decreased markedly. The best result (51% ee, **2a**) was observed in the case of nonsubstituted benzaldehyde. Aliphatic aldehyde, 3-phenylpropanal, and heteroaromatic aldehydes, 2-furaldehyde, also worked well in this transformation to provide cyanohydrins **2h** and **2i** in good yield albeit with quite poor enantioselectivities (Table 3, entries 8 and 9).

CONCLUSION

In summary, asymmetric trimethylsilylcyanation of aldehydes was realized in the presence of a novel Lewis acid and Lewis base bifunctional Ti(IV) complex formed in situ upon the treatment of ligand (*R*)-3,3′ bis(diphenylphosphinoyl)-BINOL (**L1**) with Ti(PrO-*i*)$_4$. The corresponding cyanohydrins were obtained in high yield (76–93%) with moderate enantioselectivities (up to 51% ee). Although the results were unsatisfactory, this concept could provide a guide for designing new asymmetric catalysts for the reaction of a variety of nucleophiles, including silylated ones, with carbonyl compounds.

EXPERIMENTAL

General Methods

All reagents and solvents were commercial grade and purified prior to use when necessary. ^{1}H and ^{13}C NMR were determined with the help of either a Bruker AMX-300 or a Varian 400 MHz instrument. Chemical shifts were measured relative to residual solvent peaks as an internal standard set to δ = 7.26 and δ = 77.1 ppm ($CDCl_3$) for ^{1}H and ^{13}C, respectively. Specific rotations were measured with a Perkin–Elmer 341MC polarimeter. Enantiomeric excesses were determined with the help of an Agilent 6890 instrument (β- DEX120 column). Elemental analyses were conducted with a Yanaco CHN Corder MT-3 automatic analyzer. Melting points were determined with the help of a T-4 melting point apparatus and are uncorrected.

Following Ishihara's procedure [38], (*R*)-3,3′-Bis(diphenylphosphinoyl)-BINOL (**L1**) was prepared from commercially available (*R*)-BINOL in two steps.

(R)-1,1′-Binaphthalene-2,2′-bis(diphenylphosphinate)((R)-**1**) [39].^{31}P NMR (δ, $CDCl_3$) 39.89 ppm.

(R)-3,3′-Bis(diphenylphosphinoyl)-BINOL (**L1**) was purified by column chromatography (200–300 mesh, gradient eluted with dichloromethane/ethyl acetate) [40]. Yield: 51%. mp > 280°C, $[\alpha]_D^{20}$ +93.2° (c = 1.31, $CHCl_3$). ^{1}H NMR (δ, CDCl3): 7.22–7.31 (m, 8H, Ar-H), 7.51–7.62 (m, 11H, Ar-H), 7.72–7.84 (m, 11H, Ar-H), 10.58 (broad, 2H, OH). ^{31}P NMR (δ, $CDCl_3$) 40.69 ppm.

General Procedure for **L1***/Ti(OPr-i)$_4$-Catalyzed Asymmetric Silylcyanation of Aldehydes*

A solution of **L1** (689 mg, 10 mmol%) in THF (3 mL) was stirred in a Pyrex Schlenk tube at room temperature under a nitrogen atmosphere. To the solution, Ti(OPr-*i*)$_4$ (60 μL, 0.4 mmol) was added with a syringe pump after the complete dissolution of **L1**. This solution was stirred for 2 h at room temperature then cooled to 0°C, aldehyde (2 mmol) and trimethylsilyl cyanide (400 mg, 4 mmol) were added. After stirring for 24 h at this temperature, the mixture was poured into a mixture of HCl (1 N, 15 mL) and ethyl acetate (30 mL) and was stirred for 4 h at room temperature. The organic layer was washed with distilled water and saturated $NaHCO_3$ (each 10 mL) and dried with anhydrous sodium sulfate. The solution was concentrated and purified by column chromatography (200–300 mesh, gradient eluted with petroleum ether/ethyl acetate) to yield the expected cyanohydrin. The pure cyanohydrin was converted directly into the corresponding acetates by treatment with acetic acid anhydride (2 equiv) and pyridine (2 equiv) in CH_2Cl_2 (20 mL) at room temperature for 12 h. The separated organic layer was washed with 5% H_2SO_4, distilled water, and saturated $NaHCO_3$ (each 10 mL), then dried with

anhydrous sodium sulfate and concentrated. The crude acetate was purified by column chromatography (200–300 mesh; petroleum ether/ethyl acetate, 5:1) to provide the acetylated cyanohydrin, which was used for further chiral GC analysis.

(R)-(+)-Mandelonitrile (**2a**) [41]. Colorless oil. Yield: 118 mg, 89%. $[\alpha]_D^{20} = +20.6$ ($c = 1.15$, $CHCl_3$), 51% ee. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 3.19$ (br s, 1H, OH), 5.53 (s, 1H, CH), 7.43–7.45 (m, 3 Harom), 7.51–7.53 (m, 2 Harom) ppm. GC (corresponding acetate, β-DEX120 column): tR = 23.47 (minor), 22.35 (major) min.

(R)-(+)-α-Hydroxy-4-chlorophenylacetonitrile (**2b**) [41]. Colorless oil. Yield: 147 mg, 88%. $[\alpha]_D^{20} = +8.9$ ($c = 1.00$, $CHCl_3$), 22% ee. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 4.28$ (br s, 1H, OH), 5.52 (s, 1H, OH), 7.43 (d, $J = 8.8$ Hz, 2 Harom), 7.47 (s, $J = 8.8$ Hz, 2 Harom) ppm. GC (corresponding acetate, β-DEX120 column): tR = 66.28 (minor), 63.46 (major) min.

(R)-(+)-α-Hydroxy-2-methoxyphenylacetonitrile (**2c**) [41]. Colorless oil. Yield: 151 mg, 93%. $[\alpha]_D^{20} = +3.4$ ($c = 1.03$, $CHCl_3$), 11% ee. ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 3.45$ (d, $J = 9.0$ Hz, 1H, OH), 3.96 (s, 3H, CH_3), 5.56 (d, $J = 9.0$ Hz, 1H, CH), 7.00–7.05 (m, 2 Harom), 7.38–7.44 (m, 2 Harom) ppm. GC (corresponding acetate, β-DEX120 column); tR = 67.78 (major), 68.07 (minor) min.

(R)-(+)-α-Hydroxy-4-methoxyphenylacetonitrile (**2d**) [41]. Colorless oil. Yield: 138 mg, 85%. $[\alpha]_D^{20} = +9.8$ ($c = 1.22$, $CHCl_3$), 19% ee. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 2.78$ (br s, 1H, OH), 3.84 (s, 3H, CH_3), 5.49 (s, 1H, CH), 6.96–7.46 (m, 4 Harom) ppm. GC (corresponding acetate, β-DEX120 column): tR = 75.30 (minor), 72.97 (major) min.

(R)-(+)-α-Hydroxy-2-methylphenylacetonitrile (**2e**) [41]. Colorless oil. Yield: 132 mg, 90%. $[\alpha]_D^{20} = +15.1$ ($c = 1.02$, $CHCl_3$), 27% ee. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 2.46$ (br s, 3H, CH_3), 5.68 (s, 1H, CH), 7.26–7.43 (m, 4 Harom) ppm. GC (corresponding acetate, β-DEX120 column): tR = 51.16 (minor), 50.27 (major) min.

(R)-(+)-α-Hydroxy-3-methylphenylacetonitrile (**2f**) [41]. Colorless oil. Yield: 133 mg, 91%. $[\alpha]_D^{20} = +15.7$ ($c = 1.00$, $CHCl_3$), 35% ee. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 2.39$ (s, 3H, CH_3), 3.30 (br s, 1H, OH), 5.47 (s, 1H, CH), 7.23–7.33 (m, 4 Harom) ppm. GC (corresponding acetate, β-DEX120 column): tR = 53.70 (minor), 51.30 (major) min.

(R)-(+)-2-Hydroxy-2-(1-naphthyl)acetonitrile (**2g**) [12]. Colorless oil. Yield: 161 mg, 88%. $[\alpha]_D^{20} = +13.7$ ($c = 1.05$, $CHCl_3$), 21% ee. ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 3.43$ (d, $J = 6.9$ Hz, 1H, OH), 6.17 (d, $J = 6.9$ Hz, 1H, CH), 7.42–8.22 (m, 7 Harom) ppm. GC (corresponding acetate, β-DEX120 column): tR = 155.49 (major), 151.58 (minor) min.

(R)-(–)-2-Hydroxy-4-phenylbutanenitrile (**2h**) [41]. Colorless oil. Yield: 143 mg, 89%. $[\alpha]_D^{20} = -0.58$ ($c = 1.13$, $CHCl_3$), 5% ee. ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 2.15$–2.21 (m, 2H, CH_2), 2.61 (br s, 1H, OH), 2.83–2.88 (m, 2H, CH2), 4.43 (t, $J = 6.9$ Hz, 1H, CH), 7.21–7.24 (m, 3 Harom), 7.30–7.34 (m, 2 Harom) ppm. GC (corresponding acetate, β-DEX120 column): tR = 67.38 (major), 65.59 (minor) min.

(R)-(–)-2-(Furan-2-yl)-2-hydroxyacetonitrile (**2i**) [41]. Colorless oil. Yield: 111 mg, 90%. $[\alpha]_D^{20} = -3.91$ ($c = 1.10$, $CHCl_3$), 9% ee. ^{1}H NMR (400 MHz, $CDCl_3$): $\delta = 4.31$ (br s, 1H, OH), 5.57 (s, 1H, CH), 7.27–7.71 (m, 3 Harom) ppm. GC (corresponding acetate, β-DEX120 column): tR = 26.13 (major), 28.53 (minor) min.

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