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Synthesis of some new chiral cyclic *o*-hydroxylarylphosphonodiamides as ligands for asymmetric silylcyanation of aromatic aldehydes

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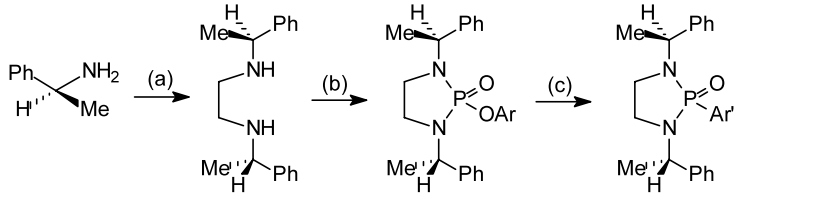
Abstract—Some new chiral cyclic *o*-hydroxylarylphosphonodiamides were synthesized from (−)-α-phenylethylamine and found to be efficient ligands for the Ti(OPr-*i*)$_4$ catalyzed asymmetric trimethylsilylcyanation of aromatic aldehydes. Corresponding cyanohydrins were obtained in good chemical yield and enantiomeric excesses up to 90%.

1. 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-hydroxyl carboxylic acids, β-amino alcohols, vicinal diols, α-hydroxyketones, etc. Many efficient approaches have been reported for obtaining them by biological and chemical methods.[1] In the latter, the most important one was the asymmetric silylcyanation of aldehydes with trimethylsilylcyanide catalyzed by a Lewis acid, such as Ti(OPr-*i*)$_4$, $TiCl_4$, $AlCl_3$, R_2AlCl, $SmCl_3$ etc. in the presence of a chiral ligand. In this reaction, a wide range of chiral ligands have been elaborated, such as Schiff bases,[2] diols,[3]diamides,[4] phosphorus compounds[5] etc. As shown in literature, many effective chiral ligands have a free hydroxyl group or an amino group bearing at least a N–H bond which is favorable to form the Lewis acid center of the catalyst by coordinating conveniently with the metal atom in Lewis acid. Moreover, if a phosphoryl group (P=O) is existed at an appropriate position in the ligand molecule, the unshared electron pair on oxygen atom should act as a Lewis base. In the catalyst, it contains both a Lewis acid center and a Lewis base center, namely, LALB catalyst. It is a new type of chiral bifunctional catalyst.[3d] In this paper, new cyclic *o*-hydroxylarylphosphonodiamides **3** were synthesized starting from (−)-α-phenylethylamine and the catalytic effect of the bifunctional catalyst formed in situ from **3** and Ti(OPr-*i*)$_4$ in asymmetric silylcyanation of aromatic aldehydes were investigated.



1

2a: Ar = Ph
2b: Ar = 4-MeC_6H_4
2c: Ar = 1-Naphthyl

3a: Ar' = 2-HOC_6H_4
3b: Ar' = 2-OH-5-MeC_6H_4
3c: Ar' = 1-OH-2-Naphthyl

(a) $BrCH_2CH_2Br$, Et_3N, 110°C; (b) $ArOP(O)Cl_2$, Et_3N/CH_2Cl_2, r.t.; (c) *n*-BuLi/THF, -78°C.

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2. Results and discussion

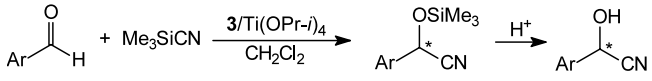
According to literature method,[6] the reaction of (−)-α-phenylethylamine and 1,2-dibromoethane in the presence of an acid binding agent readily led to (−)-*N*,*N*′-di-α-phenylethyl ethylenediamine **1**. Further cyclization of **1** with *O*-aryl phosphorodichloridates resulted in cyclic *O*-aryl phosphorodiamidates **2**. The stereoselective P–O to P–C rearrangement of **2** in the presence of *n*-BuLi gave the title compounds cyclic *o*-hydroxylarylphosphonodiamides **3**. The similar rearrangement has also been reported by Buono et al.[7]

The catalytic effect of the chiral titanium complexes prepared in situ from compound **3** and Ti(OPr-*i*)$_4$ in the asymmetric silylcyanation of aromatic aldehydes were investigated. The results were listed in Table 1.

As shown in Table 1, in all cases, the corresponding cyanohydrins was obtained in high chemical yields varying from 80 to 98%. Furthermore, the (*R*)-cyanohydrin was formed as the major enantiomer whatever substrate was employed.[5f,9]

The molar ratio of ligand **3** to Ti(OPr-*i*)$_4$ was observed to be an essential factor to the enantioselectivity. It was found that the optimal molar ratio of ligand **3** to Ti(OPr-*i*)$_4$ is 4:1, which led to the corresponding cyanohydrin in high chemical yield and relatively good enantioselectivity (entries 2 and 1, 3, 4; 5 and 7, 8). Moreover, the amount of ligand had a marked influence on the enantioselectivity of the reaction. For example, the use of 40 mol% of ligand **3c** led to the desired cyanohydrin in high yield with ee value of 90% (entry 5), while the use of 20 mol% and 10 mol% of **3c** only gave moderate and low enantioselectivity (entry 7, 62% ee; entry 8, 29% ee), respectively. Additionally, the reaction temperature also had an obvious influence on enantioselectivity. For example, satisfactory result (entry 5, 90% ee) was obtained when the reaction was carried out at 0°C, whereas the enantioselectivity decreased when the reaction was run at 20°C (entry 6, 73% ee). Therefore, in most cases, the reaction was run at 0°C since this reaction is extremely sluggish when carried out below 0°C.

Table 1. Trimethylsilylcyanation of aromatic aldehydes catalyzed by **3**/Ti(OPr-*i*)$_4$



Entry	Ar	**3** (mol%)	Ti(OPr-*i*)$_4$ (mol%)	Temp. (°C)	Yield (%)[a]	$[\alpha]_D$ (*c* 1, $CHCl_3$)	Ee (%)
1	Ph	**3c** (20)	10	0	90	+17.0	38[c]
2	Ph	**3c** (40)	10	0	98	+19.1	43[c], 42.7[b]
3	Ph	**3c** (40)	20	0	90	+18.6	41[c]
4	Ph	**3c** (40)	40	0	98	+14.0	35[c]
5	2-$MeOC_6H_4$	**3c** (40)	10	0	92	+24.6	90[d]
6	2-$MeOC_6H_4$	**3c** (40)	10	20	86	+19.8	73[d]
7	2-$MeOC_6H_4$	**3c** (20)	10	0	86	+16.8	62[d]
8	2-$MeOC_6H_4$	**3c** (10)	10	0	86	+8.0	29[d]
9	4-$MeOC_6H_4$	**3c** (40)	10	0	92	+24.3	50[e]
10	2-MeC_6H_4	**3c** (40)	10	0	80	+30.6	72[f]
11	4-MeC_6H_4	**3c** (40)	10	0	95	+20.9	41[g]
12	4-ClC_6H_4	**3c** (40)	10	10	96	+6.1	15[h]
13	3-ClC_6H_4	**3c** (40)	10	10	90	+2.0	4[i]
14	2-$NO_2C_6H_4$	**3c** (40)	10	10	96	+9.2	8[j]
15	3-$NO_2C_6H_4$	**3c** (40)	10	10	96	+1.2	5[k]
16	2-Naphthyl	**3c** (40)	10	0	90	+11.2 (EtOH)	76[l]
17	Ph	**3b** (40)	10	0	90	+9.5	21[c], 20.6[b]
18	2-$MeOC_6H_4$	**3b** (40)	10	0	98	+11.8	43[d]
19	Ph	**3a** (40)	10	0	90	+14.4	32[c], 31.7[b]
20	2-$MeOC_6H_4$	**3a** (40)	10	0	98	+11.3	41[d]

[a] Isolated yield.
[b] Determined by GC with chiral column after derivation with acetic anhydride.
[c] Determined by the comparison of specific rotation values $[\alpha]_D^{20}$ +45.0 ($CHCl_3$).[8]
[d] $[\alpha]_D^{20}$ −21.0 ($CHCl_3$) with 77% ee.[1a]
[e] $[\alpha]_D^{25}$ +49.0 ($CHCl_3$).[8]
[f] $[\alpha]_D^{25}$ +21.3 ($CHCl_3$) with 51% ee.[9]
[g] $[\alpha]_D^{25}$ +47.4 ($CHCl_3$) with 92% ee.[9]
[h] $[\alpha]_D^{25}$ +27.2 ($CHCl_3$) with 66% ee.[9]
[i] $[\alpha]_D^{25}$ +32.0 ($CHCl_3$) with 57% ee.[9]
[j] $[\alpha]_D^{25}$ +56.0 ($CHCl_3$) with 50% ee.[9]
[k] $[\alpha]_D^{25}$ +12.2 ($CHCl_3$) with 29% ee.[9]
[l] $[\alpha]_D^{25}$ +10.9 (EtOH) with 73% ee.[2c]

A series of aromatic aldehydes were used as substrate in **3c**/Ti(OPr-*i*)$_4$ catalyzed asymmetric silylcyanation under the same condition. A significant variation in enantiomeric excesses was observed depending on the nature of the aldehydes. When there exists electron withdrawing substituent (such as NO_2, Cl) on benzene ring, the enantioselectivity is quite low although the chemical yield is excellent (entries 12–15); When electron donating group (such as MeO, Me) substituted benzaldehydes and β-naphthyl aldehyde were employed as substrate, moderate to satisfactory enantioselectivity could be obtained. Furthermore, the enantioselectivity was also influenced by the postion of the substituent on benzene ring. The best result (90% ee) was obtained with *o*-methoxybenzaldehyde (entry 5), while the enantiomeric excesses of *p*-methoxybenzaldehyde was much lower (entry 9, 50% ee). The same phenomena appeared between *o*-methylbenzaldehyde and *p*-methylbenzaldehyde (entry 10, 11).

It was also found the nature of the ligand **3** had an obvious influence on enantioselectivity of the reaction. The ee values of the ligand in which Ar′ was an (un)substituted hydroxyphenyl group (such as **3a** and **3b**) were less than those of ligand **3c** in which Ar′ was a hydroxynaphthyl (entries 17, 19 and 2; 18, 20 and 5). The introduction of a methyl group on the hydroxyphenyl group had no obvious influence on the stereoselectivity (entries 17 and 19; 18 and 20). It seems that the more bulky naphthyl group has a decisive influence on the enantioselectivity of the reaction.

3. Conclusion

In conclusion, a new type of chiral cyclic *o*-hydroxylarylphosphonodiamides **3** has been synthesized starting from (−)-α-phenylethylamine. The corresponding cyanohydrins were obtained in high chemical yield in titanium complex formed in situ from compound **3** and Ti(OPr-*i*)$_4$ catalyzed silylcyanation of aromatic aldehydes. The enantioselectivity of the reaction was determined by the nature of substrate and ligand employed, the molar ratio of the ligand to Ti(OPr-*i*)$_4$ as well as the amount of the catalyst. The improvement of this type of ligand for enhancing the ee value and decreasing the amount of catalyst and the application of them for other asymmetric reaction are continuing in our laboratory.

4. Experimental

^{1}H and ^{31}P NMR were recorded in $CDCl_3$ on a Bruker AC-P200 instrument using TMS as an internal standard for ^{1}H NMR and 85% H_3PO_4 as an external standard for ^{31}P NMR. Optical rotations were measured on a Perkin-Elmer 241MC polarimeter. Elemental analyses were conducted on a Yanaco CHN Corder MT-3 automatic analyzer. Melting points were determined on a T-3 melting point apparatus. All temperatures were uncorrected. All of the solvent was used after dried and redistillation. Ti(OPr-*i*)$_4$ was purchased from Fluka. Me_3SiCN was prepared according to the literature procedure.[10]

4.1. Preparation of (−)-*N*,*N*′-di-α-phenylethyl ethylenediamine

To a mixture of 48.4 g (0.43 mol) of (−)-α-phenylethylamine and 20.2 g (0.2 mol) of triethylamine was added dropwise 18.8 g (0.1 mol) of 1,2-dibromoethane at 110°C under a nitrogen atmosphere. The resulting mixture was stirred for another 22 h at 110–130°C. After cooling to 60°C 150 mL of saturated KOH solution was added carefully. The organic layer was separated and the water layer was extracted with methylene chloride (3×100 mL). The combined organic phase was washed successively with water, saturated brine and dried over anhydrous sodium sulfate. After recovery of excess (−)-α-phenylethylamine (25.0 g) the residue was distilled under reduced pressure to afford 20.7 g expected product, yield 77%, bp 162–163°C/133 Pa $[\alpha]_D = -65.9$ (*c* 1, $CHCl_3$) [Lit.[6] $[\alpha]_D = -69.2$ (*c* 1, $CHCl_3$)].

4.2. Preparation of *O*-aryl phosphorodiamidates 2 (general procedure)

To a stirring mixture of (−)-*N*,*N*′-di-α-phenylethyl ethylenediamine 1 (2.70 g, 10 mmol), Et_3N (2.40 g, 24 mmol) and CH_2Cl_2 (40 mL) was added dropwise *O*-aryl phosphorodichloridate (10 mmol) at 0°C. The resulting mixture was stirred for 24 h at room temperature, then adjusted to pH 7 with 2N aqueous NaOH. The organic layer was separated and washed successively with distilled water and brine and dried over anhydrous magnesium sulfate. After removal of solvent the residue was purified by column chromatography on silica gel (200–300 mesh, 5:1 petroleum ether/ethyl acetate as eluent) to afford the phosphorodiamidate **2**.

4.2.1. (−)-*O*-Phenyl phosphorodiamidate 2a. Thick liquid, yield: 86% $[a]_D^{20} = -3.5$ (*c* 1, $CHCl_3$). Anal. calcd. for $C_{24}H_{27}N_2O_2P$: C, 70.92; H, 6.69; N, 6.89. Found: C, 70.85; H, 6.45; N, 6.70. ^{1}H NMR (δ, $CDCl_3$): 1.54 (d, 3H, CH_3), 1.71 (d, 3H, CH_3), 2.86 (m, 4H, $2CH_2$), 4.40 (q, 2H, 2CH), 7.30 (m, 15H arom); ^{31}P NMR (δ, $CDCl_3$): 17.86.

4.2.2. (−)-*O*-4-Methylphenyl phosphorodiamidate 2b. Thick liquid, yield: 76% $[a]_D^{20} = -3.0$ (*c*1, $CHCl_3$). Anal. calcd. for $C_{25}H_{29}N_2O_2P$: C, 71.41; H, 6.95; N, 6.66. Found: C, 71.11; H, 6.97; N, 6.79. ^{1}H NMR (δ, $CDCl_3$): 1.52 (d, 3H, CH_3), 1.68 (d, 3H, CH_3), 2.31 (s, 3H, CH_3), 2.82 (m, 4H, $2CH_2$), 4.48 (m, 2H, 2CH), 7.23 (m, 14H arom); ^{31}P NMR (δ, $CDCl_3$): 17.95.

4.2.3. (+)-*O*-1-Naphthyl phosphorodiamidate 2c. Thick liquid, yield: 84% $[\alpha]_D^{20} = +11.8$ (*c* 1, $CHCl_3$). Anal. calcd. for $C_{29}H_{29}N_2O_2P$: C, 73.67; H, 6.40; N, 6.14. Found: C, 73.66; H, 6.44; N, 5.82. ^{1}H NMR (δ, $CDCl_3$): 1.26 (d, 3H, CH_3), 1.68 (d, 3H, CH_3), 2.95 (m, 4H, $2CH_2$), 4.50 (m, 2H, 2CH), 7.40 (m, 15H arom), 7.91 (m, 1H arom), 8.32 (m, 1H arom); ^{31}P NMR (δ, $CDCl_3$): 18.20.

4.3. Preparation of *o*-hydroxylarylphosphonodiamides 3 (general procedure)

To a stirring solution of compound **2** (10 mmol) in THF (60 mL) was added dropwise a solution of *n*-BuLi (20 mmol, 1 M in hexane) at −78°C under a nitrogen atmosphere. After 15 min the cold bath was removed and the reaction mixture was poured into a mixture of CH_2Cl_2 (150 mL) and saturated aqueous NH_4Cl (30 mL). The organic layer was separated and dried over anhydrous sodium sulfate. After removal of solvent the crude product was purified by column chromatography on silica gel (200–300 mesh, 5:1 petroleum ether/ethyl acetate as eluent) to give compound **3**.

4.3.1. (−)-2-Hydroxyphenylphosphonodiamide 3a. Thick liquid, yield: 41% $[\alpha]_D^{20}=-32.3$ (*c*1, $CHCl_3$). Anal. calcd. for $C_{24}H_{27}N_2O_2P$: C, 70.92; H, 6.69; N, 6.89. Found: C, 71.10; H, 6.64; N, 6.84. ^{1}H NMR (δ, $CDCl_3$): 1.40 (d, 3H, CH_3), 1.51 (d, 3H, CH_3), 2.98 (m, 4H, $2CH_2$), 4.31 (q, 2H, 2CH), 7.10 (m, 14H arom); ^{31}P NMR (δ, $CDCl_3$): 33.55.

4.3.2. (−)-2-Hydroxy-5-methylphenylphosphonodiamide 3b. Thick liquid, yield: 36% $[\alpha]_D^{20}=-13.6$ (*c*1, $CHCl_3$). Anal. calcd. for $C_{25}H_{29}N_2O_2P$: C, 71.41; H, 6.95; N, 6.66. Found: C, 71.42; H, 6.98; N, 6.60. ^{1}H NMR (δ, $CDCl_3$): 1.36 (d, 3H, CH_3), 1.51 (d, 3H, CH_3), 2.23 (s, 3H, CH_3), 3.12 (m, 4H, $2CH_2$), 4.32 (dq, 2H, 2CH), 7.93 (m, 13H arom); ^{31}P NMR (δ, $CDCl_3$): 33.88.

4.3.3. (−)-2-Hydroxy-1-naphthylphosphonodiamide 3c. Thick liquid, yield: 60% $[\alpha]_D^{20}=-83.3$ (*c*1, $CHCl_3$). Anal. calcd. for $C_{29}H_{29}N_2O_2P$: C, 73.67; H, 6.40; N, 6.14. Found: C, 73.73; H, 6.43; N, 6.00. ^{1}H NMR (δ, $CDCl_3$): 1.37 (d, 3H, CH_3), 1.5 3 (d, 3H, CH_3), 3.18 (m, 4H, $2CH_2$), 4.42 (dq, 2H, 2CH), 7.28 (m, 14H arom), 7.82 (m, 1Harom), 8.34 (m, 1H arom); ^{31}P NMR (δ, $CDCl_3$): 34.60.

4.4. The asymmetric silylcyanation of benzaldehyde (typical procedure, entry 1)

To a solution of **3c** (0.182 g, 0.4 mmol) in 5 mL of methylene chloride was added Ti(OPr-*i*)$_4$ (0.0286 g, 0.1 mmol) under a nitrogen atmosphere at room temperature and the resulting mixture was stirred for 1 h. Then freshly distilled benzaldehyde (0.106 g, 1 mmol) and trimethylsilyl cyanide (0.2 g, 2 mmol) were added to it at 0°C and the whole stirred for 24 h at the same temperature. The mixture was poured into a mixture of 1N hydrochloric acid (30 mL) and ethyl acetate (30 mL) and stirred vigorously for 4 h at room temperature. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (2×20 mL). The combined organic phase was washed with brine and dried over anhydrous sodium sulfate. After removal of solvent the crude product was purified by column chromatography on silica gel (200–300 mesh, 5:1 petroleum ether/ethyl acetate as eluent) to afford 0.13 g of the corresponding cyanohydrin.

After measuring the optical rotation, the cyanohydrin was converted into the corresponding acetate by reacting with two equivalents of acetic anhydride in methylene chloride (20 mL) in the presence of pyridine at room temperature for 12 h. The mixture was washed sequentially with 5% H_2SO_4, distilled water and saturated aqueous $NaHCO_3$, and dried over anhydrous sodium sulfate. After removal of solvent the crude product was purified by column chromatography on silica gel (200–300 mesh, 5:1 petroleum ether/ethyl acetate as eluent) to give the acetylated cyanohydrin which was analyzed by GC with chiral column for determining ee value.

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