Pd-Catalyzed asymmetric allylation involving bis(diamidophosphite) based on the salen-type chiral diamine

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New bis(diamidophosphite) ligand with stereogenic phosphorus atoms in the 1,3,2-diazaphospholidine rings was synthesized based on (1*R*,2*R*)-[*N*,*N*′-bis(3-hydroxybenzylidene)]-1,2-diaminocyclohexane. This ligand provided up to 73% *ee* in Pd-catalyzed asymmetric allylic alkylation of (*E*)-1,3-diphenylallyl acetate with dimethyl malonate and up to 80% *ee* in its amination with pyrrolidine, with the starting substrate conversion being quantitative.

Key words: diimines, diamidophosphites, ligands, catalysts, palladium complexes, allylation, asymmetric synthesis.

Phosphorus-containing ligands play a very important role in coordination chemistry and metal complex catalysis.[1—6] Bis(diamidophosphites) are a relatively small, but very efficient group of phosphite ligands, significantly differing from the more common phosphite and amidophosphite chiral inducers. Thus, the nitrogen atoms with the corresponding substituents are bulkier and more electron-donating groups at the phosphorus atom than oxygen atoms. Varying substituents at the phosphorus and/or nitrogen atoms allows for fine tuning of these chirality inducers in accordance with the requirements of specific catalytic transformations. Known bis(diamidophosphite) ligands are successfully used in asymmetric reactions of Pd-catalyzed allylation and cycloaddition, as well as Rh-catalyzed hydrogenation and hydroformylation.[7—18]

It seems promising to obtain bis(diamidophosphite) derivatives of chiral salen diimines, *i.e.* the Schiff bases from primary diamines and hydroxy-substituted aromatic aldehydes. It is known that salens are efficient asymmetric inducers in a wide range of metal complex catalysis processes.[19,20]

In this work, we describe the synthesis and application of new *P*,*P*′,*N*,*N*′-bis(diamidophosphite) in Pd-catalyzed enantioselective allylic substitution. Note that ligands with 1,3,2-diazaphospholidine rings are stable to oxidation and hydrolysis and also have balanced electronic characteristics.[21,22] The presence of a stereogenic donor phosphorus atom in the structure of such ligands can successfully promote efficient transfer of chirality in the catalytic cycle.[19,23,24] To assess the asymmetrization potential of new bis(diamidophosphite), we selected the model reactions of allylic alkylation and amination, which are commonly used to evaluate the efficiency of new inducers of chirality and which also open access to valuable organic and natural compounds.[23,25—28] The products of alkylation with dimethyl malonate serve as precursors in the synthesis of chiral esters and amides of unsaturated carboxylic acids, while allylic amines are widely used in the construction of molecules of α- and β-amino acids, N-heterocycles, carbohydrate derivatives, alkaloids, and peptides.[29—32]

Results and Discussion

The condensation of 3-hydroxybenzaldehyde (**1**) with (*R*,*R*)-1,2-diaminocyclohexane (**2**) in CH_2Cl_2 in the presence of Na_2SO_4 gave the starting chiral diimine **3** (Scheme 1). The reaction of compound **3** with phosphorylating reagent **4** in toluene in the presence of Et_3N as a hydrogen chloride acceptor gave bis(diamidophosphite) **5** (see Scheme 1).

Ligand **5** is a mixture of epimers at phosphorus stereocenters. This follows from the presence of two singlets at δ_P 123.7 (91%) and 119.1 (9%) in the ^{31}P NMR spectrum of its solution in $CDCl_3$. The main epimer of compound **5** has the (*R*)-configuration of *P** stereocenters, which is confirmed by the observed in the ^{13}C NMR spectrum spin-spin coupling constant value $^2J_{C,P} = 35.2$ Hz between the $N\underline{C}H_2CH_2$ and phosphorus atoms of the phosphabicyclo[3.3.0]octane fragments. This value indicates the *anti*-orientation of the pseudo-equatorial exocyclic substituent X at the phosphorus atom and the $-(CH_2)_3-$

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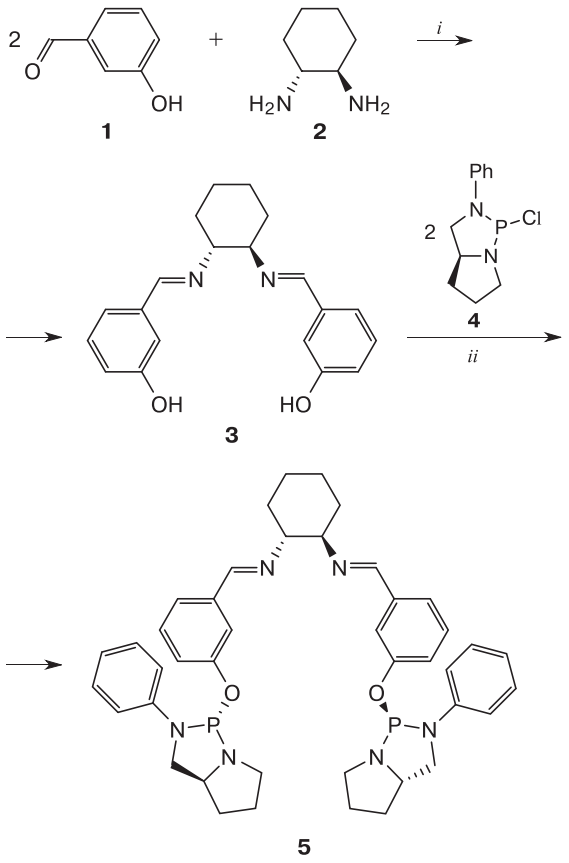
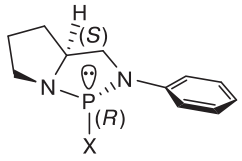
Scheme 1



Reagents and conditions: *i*. CH_2Cl_2, Na_2SO_4, ~40 °C, 4 h; *ii*. Et_3N, toluene, ~20 °C, 24 h.

fragment of the pyrrolidine ring of the phosphabicyclo[3.3.0]octane framework and, therefore, the *syn*-orientation between the lone electron pair of the phosphorus atom and the $N\underline{C}H_2CH_2$ atom.



The asymmetric phosphorus atoms in the minor epimer of ligand **5** have the (*S*)-configuration.[13,16,33—36] The presence of a third diastereomer with the opposite absolute configurations of stereogenic phosphorus atoms in the ligand molecule cannot be excluded, either. A detailed study of the solution of ligand **5** in $CDCl_3$ by 2D NMR spectroscopy techniques ($^1H,^1H$-COSY, $^1H,^1H$-TOCSY, $^1H,^{13}C$-HSQC and $^1H,^{13}C$-HMBC) made it possible to assign all the signals in the ^1H and ^{13}C NMR spectra for its main epimer (see Experimental).

Table 1. The results of Pd-catalyzed alkylation of substrate **6** with dimethyl malonate to give product **7**[a]

Entry	**5** : Pd	Solvent	Conversion[b] (%)	*ee*[b] (%)
1	0.5	THF	72	64 (*S*)
2	1	THF	100	50 (*S*)
3	2	THF	100	54 (*S*)
4	0.5	CH_2Cl_2	100	65 (*S*)
5	1	CH_2Cl_2	100	66 (*S*)
6	2	CH_2Cl_2	100	73 (*S*)

[a] 1 mol.% of $[Pd(All)Cl]_2$, BSA, KOAc, 20 °C, 48 h.
[b] Conversion of substrate **6** and enantiomeric excess of product **7** were determined by HPLC (see Experimental).

Bis(diamidophosphite) **5** is easily purified by flash chromatography, it is sufficiently stable in air, capable of long-term storage in a dry atmosphere, and can be produced on a several gram scale.

To evaluate the catalytic efficiency, ligand **5** (represented, as noted above, by a mixture of diastereomers) was used in Pd-catalyzed reactions of asymmetric allylic alkylation and amination of (*E*)-1,3-diphenylallyl acetate **6** (Scheme 2, Tables 1 and 2), using $[Pd(All)Cl]_2$ as a palladium source. In the allylic alkylation of substrate **6** with dimethyl malonate (C-nucleophile), *P*,*P*′,*N*,*N*′-bis(diamidophosphite) **5** provides the asymmetric induction in the range of 50—73% *ee*, with a predominance of (*S*)-enantiomer of the reaction product **7** (see Table 1). The optimal solvent is CH_2Cl_2. Generally, the molar ratio L : Pd has no unambiguous effect on the enantioselectivity level. The conversion of the starting substrate **6** as a rule is quantitative.

The use of pyrrolidine as the N-nucleophile provided the *ee* value of up to 80% with the quantitative conversion of substrate **6** in almost all experiments. Amine **8** with the (*R*)-configuration was predominantly formed (see Table 2). Significantly larger enantiomeric excesses were observed when the reaction was carried out in THF. Another dif-

Scheme 2

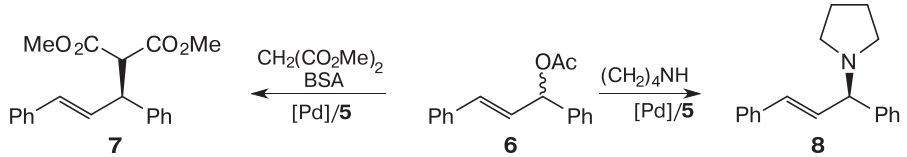


Table 2. The results of Pd-catalyzed amination of substrate **6** with pyrrolidine to give product **8**[a]

Entry	**5** : Pd	Solvent	Conversion[b] (%)	*ee*[b] (%)
1	0.5	THF	94	80 (*R*)
2	1	THF	100	71 (*R*)
3	2	THF	100	65 (*R*)
4	0.5	CH_2Cl_2	100	64 (*R*)
5	1	CH_2Cl_2	100	53 (*R*)
6	2	CH_2Cl_2	100	43 (*R*)

[a] 1 mol.% of $[Pd(All)Cl]_2$, 20 °C, 48 h.
[b] Conversion of substrate **6** and enantiomeric excess of product **8** were determined by HPLC (see Experimental).

ference from the allylic alkylation was that the enantioselectivity increased with the decreasing molar ratio L : Pd.

In conclusion, we accomplished the synthesis of new *P*,*P*′,*N*,*N*′-bis(diamidophosphite) ligand, namely, (1*R*,2*R*)-*N*,*N*′-bis{3-[(2*R*,5*S*)-3-phenyl-1,3-diaza-2-phosphabicyclo[3.3.0]oct-2-yloxy]benzylidene}-1,2-diaminocyclohexane. In the model process of allylic substitution involving (*E*)-1,3-diphenylallyl acetate, the *ee* value was up to 80%. We are investigating further use of ligand **5** as a chirality inducer.

Experimental

^{31}P, ^{1}H, and ^{13}C NMR spectra were recorded on a Varian Inova 500 spectrometer (202.33, 499.8, and 125.69 MHz, respectively) relative to 85% H_3PO_4 in D_2O and Me_4Si. The signals in the ^{1}H and ^{13}C NMR spectra were assigned using APT, ^{1}H,^{1}H-COSY, ^{1}H,^{1}H-TOCSY, ^{1}H,^{13}C-HSQC, and ^{1}H,^{13}C-HMBC techniques and already known data.[7,10,26—28] Optical rotation was measured on an Atago AP-300 polarimeter. Specific rotation is expressed in deg mL g^{-1} dm^{-1}, concentration of solutions in g (100 mL)$^{-1}$. Enantiomeric analysis of products of catalytic reactions was carried out on a Stayer HPLC chromatograph. Elemental analysis was performed on a Carlo Erba EA1108 CHNS-O CHN microanalyzer.

All reactions were carried out under dry argon in anhydrous solvents. (*R*,*R*)-1,2-Diaminocyclohexane (**2**) was obtained by resolution of the racemic mixture using (*R*,*R*)-tartaric acid according to a known procedure.[37,38] The phosphorylating agent (5*S*)-2-chloro-3-phenyl-1,3-diaza-2-phosphabicyclo[3.3.0]-octane (**4**), as well as the substrate (*E*)-1,3-diphenylallyl acetate (**6**) and the complex $[Pd(All)Cl]_2$ were synthesized according to known procedures.[33,39] Catalytic experiments on asymmetric allylation of substrate **6** with dimethyl malonate and pyrrolidine, determination of the conversion of compound **6**, as well as the enantiomeric excesses of products **7** and **8**, were carried out in accordance with published procedures.[16,40]

3-Hydroxybenzaldehyde (**1**), racemic 1,2-diaminocyclohexane, dimethyl malonate, BSA, pyrrolidine, and triethylamine were commercially available from Fluka and Aldrich.

(1*R*,2*R*)-*N*,*N*′-Bis(3-hydroxybenzylidene)-1,2-diaminocyclohexane (3). 3-Hydroxybenzaldehyde (3.01 g, 24.65 mmol) and freshly calcined Na_2SO_4 (3.3 g, 23.24 mmol) were added to a vigorously stirred solution of (1*R*,2*R*)-1,2-diaminocyclohexane (1.33 g, 11.63 mmol) in CH_2Cl_2 (16 mL). The reaction mixture was refluxed for 4 h with stirring, cooled to 20 °C, filtered, and the filtrate was concentrated *in vacuo* (40 Torr). The resulting product was purified by crystallization from CH_2Cl_2/THF (4 : 1). The yield was 2.51 g (67%), light yellow crystals, m.p. 78—80 °C, $[\alpha]^{25}_D$ −239.0 (*c* 1.0, MeOH). Found (%): C, 74.69; H, 6.94; N, 8.53. $C_{20}H_{22}N_2O_2$. Calculated (%): C, 74.51; H, 6.88; N, 8.69. ^{13}C NMR (methanol-d_6), δ: 25.51 (CH_2); 33.86 ($\underline{C}H_2CH$); 74.96 ($CH_2\underline{C}H$); 115.35 (CH(Ar)); 119.12 (CH(Ar)); 120.90 (CH(Ar)); 130.59 (CH(Ar)); 138.48 (C(Ar)); 158.81 (C(Ar)); 164.35 (NCH). ^{1}H NMR (methanol-d_6), δ: 1.46—1.55 (br.m, 2 H, CH_2); 1.73—1.91 (br.m, 4 H, $C\underline{H}_2CH$); 1.81—1.91 (br.m, 2 H, CH_2); 3.36—3.41 (br.m, 2 H, $CH_2C\underline{H}$); 6.80—6.82 (m, 2 H, CH(Ar)); 7.03—7.05 (m, 2 H, CH(Ar)); 7.05 (s, 2 H, CH(Ar)); 7.15 (t, 2 H, CH(Ar), $^{3}J_{H,H}$ = 7.8 Hz); 8.14 (s, 2 H, NCH).

(1*R*,2*R*)-*N*,*N*′-Bis{3-[(2*R*,5*S*)-3-phenyl-1,3-diaza-2-phosphabicyclo[3.3.0]oct-2-yloxy]benzylidene}-1,2-diaminocyclohexane (5). Compound **3** (0.32 g, 1 mmol) was added in one portion to a vigorously stirred solution of phosphorylating agent **4** (0.48 g, 2 mmol) and Et_3N (0.35 mL, 2.5 mmol) in toluene (20 mL) at 20 °C. The resulting mixture was stirred for 24 h and filtered through a short column with SiO_2/Al_2O_3. The filtrate was concentrated *in vacuo* (40 Torr), the resulting product was purified by flash chromatography on Al_2O_3, eluent toluene. The yield was 0.59 g (81%), a white paraffin-like substance. Found (%): C, 69.23; H, 6.68; N, 11.34. $C_{42}H_{48}N_6O_2P_2$. Calculated (%): C, 69.03; H, 6.62; N, 11.50. ^{13}C NMR ($CDCl_3$), δ: 24.48 (s, CH_2 (cyclohexane)); 26.44 (d, $NCH_2\underline{C}H_2$, $^{3}J_{C,P}$ = 4.4 Hz); 32.00 (s, CH_2); 32.90 (s, $\underline{C}H_2CH$ (cyclohexane)); 47.77 (d, $N\underline{C}H_2CH_2$, $^{2}J_{C,P}$ = 35.2 Hz); 53.78 (d, $N\underline{C}H_2CH$, $^{2}J_{C,P}$ = 7.8 Hz); 62.92 (d, $NCH_2\underline{C}H$, $^{2}J_{C,P}$ = 8.9 Hz); 73.67 (s, $CH_2\underline{C}H$ (cyclohexane)); 115.45 (d, *o*-CH_{Ph}, $^{3}J_{C,P}$ = 13.0 Hz); 119.39 (s, *p*-CH_{Ph}); 121.73 (d, CH_{Ar}, $^{4}J_{C,P}$ = 4.8 Hz); 122.25 (s, CH_{Ar}); 123.71 (d, CH_{Ar}, $^{3}J_{C,P}$ = 4.8 Hz); 129.00 (s, CH_{Ar}); 129.20 (s, *m*-CH_{Ph}); 137.78 (s, C_{Ar}); 145.29 (d, *ipso*-C_{Ph}, $^{2}J_{C,P}$ = 15.3 Hz); 154.02 (d, CO_{Ar}, $^{2}J_{C,P}$ = 4.4 Hz); 160.29 (s, NCH). ^{1}H NMR ($CDCl_3$), δ: 1.38—1.45 (m, 2 H, CH_2); 1.45—1.53 (m, 2 H, CH_2 (cyclohexane)); 1.73—1.81 (m, 4 H, $NCH_2C\underline{H}_2$); 1.76—1.84 (m, 4 H, $C\underline{H}_2CH$ (cyclohexane)); 1.78—1.83 (m, 2 H, CH_2); 1.83—1.91 (br.m, 2 H, CH_2 (cyclohexane)); 3.03—3.07 (m, 2 H, $NC\underline{H}_2CH$); 3.18—3.25 (m, 2 H, $NC\underline{H}_2CH_2$); 3.27—3.30 (m, 2 H, $NC\underline{H}_2CH$); 3.32—3.36 (br.m, 2 H, $CH_2C\underline{H}$ (cyclohexane)); 3.43—3.50 (m, 2 H, $NCH_2C\underline{H}$); 3.57—3.66 (m, 2 H, $NC\underline{H}_2CH_2$); 6.82 (d, 2 H, CH_{Ar}, $^{3}J_{H,H}$ = 7.6 Hz); 6.90 (t, 2 H, *p*-CH_{Ph}, $^{3}J_{H,H}$ = 7.1 Hz); 7.04—7.05 (m, 4 H, *o*-CH_{Ph}); 7.08 (s, 2 H, CH_{Ar}); 7.12 (t, 2 H, CH_{Ar}, $^{3}J_{H,H}$ = 7.8 Hz); 7.27 (t, 4 H, *m*-CH_{Ph}, $^{3}J_{H,H}$ = 7.8 Hz); 7.34 (t, 2 H, CH_{Ar}, $^{3}J_{H,H}$ = 7.6 Hz); 8.05 (s, 2 H, NCH).

Asymmetric alkylation of (*E*)-1,3-diphenylallyl acetate (6) with dimethyl malonate. A solution of the complex $[Pd(All)Cl]_2$ (0.0019 g, 0.005 mmol) and ligand **5** (0.0037 g, 0.005 mmol, or 0.0073 g, 0.01 mmol, or 0.0146 g, 0.02 mmol) in the corresponding solvent (5 mL) was stirred for 40 min. (*E*)-1,3-Diphenylallyl acetate (0.1 mL, 0.5 mmol) was added, and the solution was stirred for another 15 min, then dimethyl malonate (0.1 mL, 0.87 mmol), BSA (0.22 mL, 0.87 mmol), and potassium acetate (0.002 g) were added. The reaction mixture was stirred for 48 h, diluted with hexane (5 mL), and filtered through a short layer of SiO_2. The solvents were evaporated under reduced pressure (40 Torr), the residue was vacuum dried (10 Torr). The conversion of substrate **6** and the enantiomeric excess of product **7** were

determined by HPLC on a Daicel Chiralcel OD-H chiral stationary phase (eluent C_6H_{14}—Pr^iOH (99 : 1), 0.3 mL min^{-1}, 254 nm, $t(R)$ = 28.0 min, $t(S)$ = 29.3 min).

Asymmetric amination of (*E*)-1,3-diphenylallyl acetate (6) with pyrrolidine. A solution of $[Pd(All)Cl]_2$ (0.0019 g, 0.005 mmol) and ligand **5** (0.0037 g, 0.005 mmol, or 0.0073 g, 0.01 mmol, or 0.0146 g, 0.02 mmol) in the corresponding solvent (5 mL) was stirred for 40 min. (*E*)-1,3-Diphenylallyl acetate (0.1 mL, 0.5 mmol) was added and the solution was stirred for another 15 min, then freshly distilled pyrrolidine (0.12 mL, 1.5 mmol) was added. The reaction mixture was stirred for 48 h, diluted with hexane (5 mL), and filtered through a short layer of SiO_2. The solvents were evaporated under reduced pressure (40 Torr), the residue was vacuum dried (10 Torr). The conversion of substrate **6** and the enantiomeric excess of product **8** were determined by HPLC on a Daicel Chiralcel OD-H chiral stationary phase (eluent C_6H_{14}—Pr^iOH—$HNEt_2$ (200 : 1 : 0.1), 0.9 mL min^{-1}, 254 nm, $t(R)$ = 5.0 min, $t(S)$ = 6.1 min).

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