Chiral phosphite-type ligands based on ((4*R*,5*R*)-2,2-dimethyl-1,3-dioxolane-4,5-diyl)bis(diphenylmethanol) ((*R*,*R*)-TADDOL) with peripheral arylamino groups

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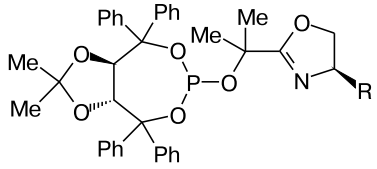
New phosphite and amidophosphite inductors of chirality were obtained based on ((4*R*,5*R*)-2,2-dimethyl-1,3-dioxolane-4,5-diyl)bis(diphenylmethanol) ((*R*,*R*)-TADDOL) containing arylamino groups in the exocyclic substituents. Their use in the palladium-catalyzed enantioselective alkylation of (*E*)-1,3-diphenylallyl acetate with dimethyl malonate gave the *ee* value up to 98%.

Key words: chiral phosphites, chiral amidophosphites, *P,N*-ligands, asymmetric alkylation, palladium catalysts.

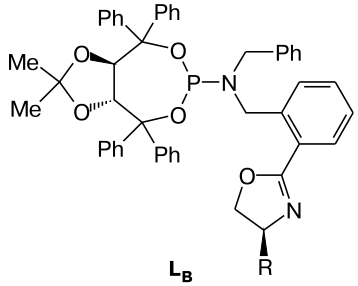
((4*R*,5*R*)-2,2-Dimethyl-1,3-dioxolane-4,5-diyl)bis(diphenylmethanol) ((*R*,*R*)-TADDOL) (**1**) belongs to the most available enantiomerically pure diols and is widely used as highly efficient asymmetric inductor.[1—4] Chiral phosphorus-containing ligands on its basis showed good properties in enantioselective metal complex catalysis.[3,5] In particular, the corresponding phosphite-type *P*,*N*-ligands are used in the Cu-catalyzed processes of conjugate addition and allylic substitution, Rh-catalyzed hydrosilylation, Ir-catalyzed hydroboration, as well as in the Pd-catalyzed reactions of methoxycarbonylation and cyclization.[6—15] *P*,*N*-Phosphites $\mathbf{L_A}$ and amidophosphites $\mathbf{L_B}$ were used in the Pd-catalyzed asymmetric allylic alkylation, first of all, of (*E*)-1,3-diphenylallyl acetate with dimethyl malonate.[16—18]

This model reaction is widely used for the evaluation of the efficiency of new inductors of chirality.[19—23] Apart from that, the products of alkylation with dimethyl malonate are convenient precursors in the synthesis of esters and amides of chiral unsaturated carboxylic acids.[24]

Earlier, we have reported the synthesis and involvement in the palladium- and copper-catalyzed asymmetric conversions of phosphite-type *P*,*N*-ligands $\mathbf{L_C}$ and $\mathbf{L_D}$ based on (R_a)- and (S_a)-1,1′-binaphthalene-2,2′-diol ((R_a)- and (S_a)-BINOL) bearing the moieties of (R_a)- and (S_a)-2′-benzylamino-1,1′-binaphthalen-2-ol ((R_a)- and (S_a)-*N*-Bn-NOBIN) or (*S*)-2-(phenylaminomethyl)pyrrolidine ((*S*)-PAMPY) as exocyclic substituents.[25,26]



$\mathbf{L_A}$



$\mathbf{L_B}$

In the present work, we describe the synthesis and application in enantioselective catalysis of phosphite- and amidophosphite-type *P*,*N*-ligands **2** and **3** with the same exocyclic substituents and the fragment (*R*,*R*)-TADDOL in the dioxaphosphepine ring. A Pd-catalyzed reaction of asymmetric allylic alkylation was chosen as a catalytic process for their testing. We also compared the enantidiscriminating activity of the ligands **2** and **3** between each other and with their known analogs $\mathbf{L_A}$—$\mathbf{L_D}$.

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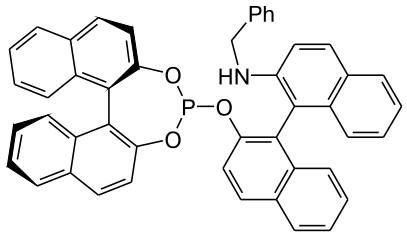
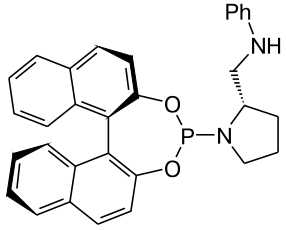


L_C



L_D

Table 1. Pd-Catalyzed alkylation of (*E*)-1,3-diphenylallyl acetate (**4**) with dimethyl malonate[a]

Entry	Ligand (L)	L : Pd	Solvent	Conversion (%)	*ee* (%)[b]
1	**1**	1	THF	10	80 (*R*)
2	**1**	2	THF	15	75 (*R*)
3	**1**	1	CH_2Cl_2	55	98 (*R*)
4	**1**	2	CH_2Cl_2	44	97 (*R*)
5	**2**	1	THF	15	76 (*S*)
6	**2**	2	THF	13	76 (*S*)
7	**2**	1	CH_2Cl_2	100	87 (*S*)
8	**2**	2	CH_2Cl_2	100	79 (*S*)

[a] Reaction conditions: 2 mol. % of $[Pd(allyl)Cl]_2$, 20 °C, 48 h.
[b] Conversion of substrate **4** and enantiomeric excess of product **5** were determined by HPLC (Daicel Chiralcel OD—H, C_6H_{14}—Pr^iOH (99 : 1), 0.3 mL min^{-1}, 254 nm, $t(R)$ = 28.0 min, $t(S)$ = 29.3 min).

Results and Discussion

The new *P*,*N*-ligands **2** and **3** were synthesized by the condensation of (*R*,*R*)-TADDOL (**1**) with PCl_3 catalyzed by *N*-methylpyrrolidone (NMP) and the subsequent reaction with (S_a)-*N*-Bn-NOBIN or (*S*)-PAMPY in toluene in the presence of an excess of Et_3N as a base (Scheme 1).

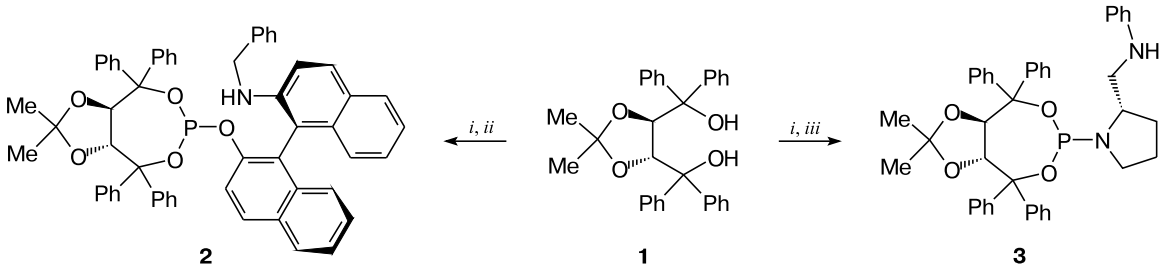
The latter reactants were obtained from commercial (S_a)-BINOL[27—30] and available (*S*)-pyroglutamic acid anilide,[31,32] respectively. It is important that inexpensive natural (*R*,*R*)-tartaric and (*S*)-glutamine acids were used in the synthesis of (*R*,*R*)-TADDOL and (*S*)-PAMPY. Like in the case of the synthesis of ligands $\mathbf{L_C}$ and $\mathbf{L_D}$, the phosphorylation of amino phenol (S_a)-*N*-Bn-NOBIN exclusively involves the hydroxyl group and the pyrrolidine amino group is involved in the diamine (*S*)-PAMPY.

Compounds **2** and **3** are well soluble in common organic solvents, they can be purified by flash-chromatography, are stable enough in air, and can be stored under dry atmosphere for a long time. Their structure was confirmed by ^1H, ^{13}C, and ^{31}P NMR spectroscopy and elemental analysis. Ligands **2** and **3** are characterized by high steric requirement, that follows from the high values of their Tolman cone angles (θ) (215° and 197°, respectively) calculated using the semiempirical quantum chemical method AM1 with full optimization of geometrical parameters.[33,34]

Chiral inductors **2** and **3** were studied in the Pd-catalyzed asymmetric allylic alkylation of (*E*)-1,3-diphenylallyl acetate (**4**) with dimethylmalonate, using $[Pd(allyl)Cl]_2$ as a palladium source (Scheme 2, Table 1).

As it follows from the data given in Table 1, the values of conversion and asymmetric induction strongly depend on the ligand and the solvent nature, while the molar ratio L : Pd does not exert significant influence on the catalytic efficiency. Phosphite **2** shows an excellent enantioselectivity (up to 98% *ee*) and a predominant formation of (*R*)-enantiomer of the reaction product **5** (see Table 1, entries *1*—*4*). Considerably higher conversion and asymmetric induction are observed in CH_2Cl_2. A similar tendency is also characteristic of the catalytic experiments involving amidophosphite **3** (Table 1, entries *5*—*8*). Carrying out the reaction in CH_2Cl_2 can provide up to 87% *ee* with a quantitative conversion of the starting sub-

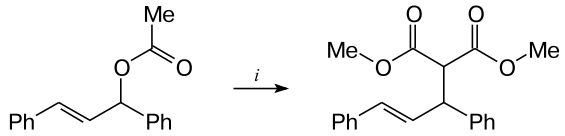
Scheme 1



2 **1** **3**

Reagents and conditions: *i*. PCl_3, NMP; *ii*. (S_a)-*N*-Bn-NOBIN, Et_3N, toluene; *iii*. (*S*)-PAMPY, Et_3N, toluene.

Scheme 2



4 **5**

Reagents and conditions: *i*. $CH_2(CO_2Me)_2$, Pd-cat, BSA, AcOK, solvent.

strate. In all the cases, the (*S*)-enantiomer of product **5** was predominant.

In conclusion, we have obtained new phosphite-type *P*,*N*-ligands **2** and **3**, successfully used them in the asymmetric Pd-catalyzed allylic alkylation of (*E*)-1,3-diphenylallyl acetate (**4**) with dimethylmalonate. The bulkier ligand **2** provides higher enantioselectivity, but lower conversion. Palladium catalysts based on the known *P*,*N*-ligands $\mathbf{L_A}$—$\mathbf{L_D}$ give 56—88% *ee* in this reaction. Therefore, amidophosphite **3** demonstrated efficiency comparable with that of these analogs, while phosphite **2** is a considerably better inductor of chirality. We plan to continue our studies of the processes of asymmetric metal complex catalysis involving ligands **2** and **3**.

Experimental

^{31}P, ^{1}H, and ^{13}C NMR spectra were recorded on Bruker Avance 400 (161.98, 400.13, and 100.61 MHz) and Varian Inova 500 (202.33, 499.8, and 125.69 MHz) relative to 85% H_3PO_4 in D_2O and Me_4Si, respectively. The signals in the ^{1}H and ^{13}C NMR spectra were assigned using the APT, ^{1}H—^{1}H COSY, ^{1}H—^{1}H NOESY, ^{1}H—^{13}C HSQC, ^{1}H—^{13}C HMBC experiments and taking into account the data in the works.[25,26,35,36] Enantiomeric analysis of the catalytic reaction products was carried out on a Stayer HPL chromatograph. Elemental analysis was carried out on a Carlo Erba EA1108 CHNS-O microanalyzer.

All the reactions were carried out under dry argon in anhydrous solvents. (*R*,*R*)-TADDOL (**1**) was synthesized following a known procedure.[36] (S_a)-*N*-Bn-NOBIN and (*S*)-PAMPY were obtained from (S_a)-BINOL (Aldrich) and available (*S*)-pyroglutamic acid anilide (obtained, in turn, from (*S*)-glutamic acid (Aldrich) and aniline (Aldrich)) according to the procedures described earlier.[27—32] The reagents (*E*)-1,3-diphenylallyl acetate (**4**) and $[Pd(allyl)Cl]_2$ were obtained according to the known procedures.[37] The catalytic experiments on asymmetric alkylation of substrate **4** with dimethyl malonate, the determination of compound **4** conversion and enantiomeric excess of product **5** were carried out being guided by the procedure published earlier.[38]

Phosphorus trichloride, *N*-methylpyrrolidone (NMP), dimethyl malonate, bis(trimethylsilyl)acetamide (BSA), and triethylamine are commercially available agents from Fluka and Aldrich.

Synthesis of ligands 2 and 3 (general procedure). *N*-Methylpyrrolidone (0.01 g, 0.1 mmol) was added to a vigorously stirred suspension of (*R*,*R*)-TADDOL (0.93 g, 2 mmol) in PCl_3 (4 mL, 45.5 mmol), and the mixture was refluxed for 5 min until it became homogeneous. Then, an excess of PCl_3 was removed *in vacuo* (40 Torr), the residue was dried *in vacuo* (30 min, 1 Torr) to remove traces of PCl_3 and dissolved in toluene (12 mL). Triethylamine (0.56 mL, 4 mmol) and (S_a)-*N*-Bn-NOBIN or (*S*)-PAMPY (2 mmol) were added to the resulting solution with vigorous stirring at 20 °C. The reaction mixture was stirred for 24 h at 20 °C, then heated to 40 °C and stirred at this temperature for 1 h. After cooling to 20 °C, the mixture was filtered through a short column with SiO_2/Al_2O_3. The filtrate was concentrated *in vacuo* (40 Torr), the products **2** and **3** were purified by column chromatographiy on silica gel (eluent toluene—hexane (2 : 1) and toluene—heptane (1 : 2), respectively).

(3a*R*,8a*R*)-6-[(*S*$_a$)-(2′-Benzylamino-1,1′-binaphthalen-2-yl)oxy]-2,2-dimethyl-4,4,8,8-tetraphenyltetrahydro[1,3]dioxolo[4,5-*e*][1,3,2]dioxaphosphepine (2). The yield was 1.29 g (74%), a white powder, m.p. 107—108 °C. Found (%): C, 80.32; H, 5.62; N, 1.44. $C_{58}H_{48}NO_5P$. Calculated (%): C, 80.07; H, 5.56; N, 1.61. ^{31}P NMR ($CDCl_3$), δ: 134.2 (s). ^{13}C NMR ($CDCl_3$), δ: 26.0 (s, Me); 26.9 (s, Me); 47.6 (s, CH_2N); 79.6 (d, CHO, $^{3}J_{C,P}$ = 5.0 Hz); 82.0 (d, CHO, $^{3}J_{C,P}$ = 11.5 Hz); 83.5 (d, $C(Ph_2)$, $^{2}J_{C,P}$ = 4.3 Hz); 84.1 (s, $C(Ph_2)$); 113.7 (s, $C(Me_2)$); 113.9 (s, CH_{Ar}); 121.4 (s, CH_{Ar}); 122.3 (d, CH_{Ar}, $^{3}J_{C,P}$ = 6.1 Hz); 123.9 (d, C_{Ar}, $^{3}J_{C,P}$ = 3.8 Hz); 124.3 (s, CH_{Ar}); 125.0 (s, CH_{Ar}); 126.0 (s, CH_{Ar}); 126.1 (s, CH_{Ar}); 126.5 (s, CH_{Ar}); 126.6 (s, CH_{Ar}); 126.7 (s, CH_{Ar}); 126.8 (s, CH_{Ar}); 126.9 (s, CH_{Ar}); 127.0 (s, CH_{Ar}); 127.1 (s, CH_{Ar}); 127.2 (s, C_{Ar}); 127.3 (s, CH_{Ar}); 127.5 (s, CH_{Ar}); 127.7 (s, CH_{Ar}); 127.8 (s, CH_{Ar}); 127.9 (s, CH_{Ar}); 128.0 (s, CH_{Ar}); 128.1 (s, C_{Ar}); 128.3 (s, CH_{Ar}); 128.4 (s, CH_{Ar}); 128.8 (s, CH_{Ar}); 128.9 (s, C_{Ar}); 129.4 (s, CH_{Ar}); 131.2 (s, C_{Ar}); 133.6 (s, C_{Ar}); 134.1 (s, C_{Ar}); 139.9 (s, C_{Ar}); 140.6 (s, C_{Ar}); 141.3 (s, C_{Ar}); 143.7 (s, C_{Ar}); 145.2 (d, C_{Ar}, $^{2}J_{C,P}$ = 4.2 Hz). ^{1}H NMR ($CDCl_3$), δ: 0.45 (s, 3 H, Me); 0.78 (s, 3 H, Me); 3.94 (br.s, 1 H, NH); 4.18 (d, 1 H, CH_2N, ^{2}J = 16.0 Hz); 4.30 (d, 1 H, CH_2N, ^{2}J = 16.0 Hz); 5.00 (d, 1 H, CHO, ^{3}J = 8.0 Hz); 5.38 (d, 1 H, CHO, ^{3}J = 8.0 Hz); 6.89 (d, 1 H, CH_{Ar}, ^{3}J = 8.6 Hz); 7.05—7.10 (m, 3 H, CH_{Ar}); 7.11—7.16 (m, 6 H, CH_{Ar}); 7.16—7.23 (m, 15 H, CH_{Ar}); 7.24—7.30 (m, 5 H, CH_{Ar}); 7.40—7.45 (m, 3 H, CH_{Ar}); 7.67 (d, 1 H, CH_{Ar}, ^{3}J = 8.1 Hz); 7.73 (d, 1 H, CH_{Ar}, ^{3}J = 8.9 Hz); 7.85 (d, 1 H, CH_{Ar}, ^{3}J = 8.9 Hz); 7.91 (d, 1 H, CH_{Ar}, ^{3}J = 8.1 Hz).

(3a*R*,8a*R*)-2,2-Dimethyl-4,4,8,8-tetraphenyltetrahydro-6-[(*S*)-2-(phenylaminomethyl)pyrrolidn-1-yl]-[1,3]dioxolo[4,5-*e*]-[1,3,2]dioxaphosphepine (3). The yield was 1.10 g (82%), a white powder, m.p. 112—113 °C. Found (%): C, 75.40; H, 6.54; N, 4.29. $C_{42}H_{43}N_2O_4P$. Calculated (%): C, 75.20; H, 6.46; N, 4.18. ^{31}P NMR ($CDCl_3$), δ: 139.6 (s). ^{13}C NMR ($CDCl_3$), δ: 25.3 (s, Me); 25.4 (s, CH_2); 27.7 (s, Me); 30.36 (s, CH_2); 45.2 (d, CH_2N, $^{2}J_{C,P}$ = 15.0 Hz); 48.6 (d, $CH_2N(Ph)$, $^{3}J_{C,P}$ = = 4.6 Hz); 56.9 (d, CHN, $^{2}J_{C,P}$ = 13.9 Hz); 81.9 (d, $C(Ph_2)$, $^{2}J_{C,P}$ = 9.3 Hz); 82.1 (s, $C(Ph_2)$); 82.3 (d, CHO, $^{3}J_{C,P}$ = 20.8 Hz); 82.6 (d, CHO, $^{3}J_{C,P}$ = 3.8 Hz); 111.6 (s, $C(Me_2)$); 112.8 (s, CH_{NPh}); 116.9 (s, CH_{NPh}); 127.0 (s, CH_{Ph}); 127.1 (s, CH_{Ph}); 127.2 (s, CH_{Ph}); 127.3 (s, CH_{Ph}); 127.4 (s, CH_{Ph}); 127.5 (s, CH_{Ph}); 127.7 (s, CH_{Ph}); 128.2 (s, CH_{Ph}); 128.3 (s, CH_{Ph}); 128.7 (s, CH_{Ph}); 128.8 (s, CH_{Ph}); 129.0 (s, CH_{NPh}); 129.1 (s, CH_{Ph}); 141.5 (s, C_{Ph}); 142.3 (s, C_{Ph}); 146.6 (s, C_{Ph}); 146.9 (s, C_{Ph}); 148.6 (s, C_{NPh}). ^{1}H NMR ($CDCl_3$), δ: 0.33 (s, 3 H, Me); 1.36 (s, 3 H, Me); 1.72—1.80 (m, 1 H, CH_2); 1.87—1.99 (m, 2 H, CH_2); 2.05—2.13 (m, 1 H, CH_2); 3.08—3.16 (m, 2 H,

$CH_2N(Ph)$); 3.21—3.28 (m, 1 H, CH_2N); 3.72—3.79 (m, 1 H, CH_2N); 4.13—4.20 (m, 1 H, CHN); 4.32 (br.t, 1 H, NH, ^3J = = 5.6 Hz); 4.87 (d, 1 H, CHO, ^3J = 8.6 Hz); 5.27 (dd, 1 H, CHO, ^3J = 8.6 Hz, ^4J = 3.6 Hz); 6.44 (d, 2 H, CH_{NPh}, ^3J = 7.6 Hz); 6.66 (t, 1 H, CH_{NPh}, ^3J = 7.6 Hz); 7.09 (t, 2 H, CH_{NPh}, ^3J = = 7.6 Hz); 7.18—7.28 (m, 5 H, CH_{Ph}); 7.29—7.38 (m, 7 H, CH_{Ph}); 7.47—7.52 (m, 4 H, CH_{Ph}); 7.66 (d, 2 H, CH_{Ph}, ^3J = 7.4 Hz); 7.79 (d, 2 H, CH_{Ph}, ^3J = 6.7 Hz).

Asymmetric alkylation of (*E*)-1,3-diphenylallyl acetate (4) with dimethyl malonate. A solution of $[Pd(allyl)Cl]_2$ (0.0037 g, 0.01 mmol) and the corresponding ligand (0.02 mmol or 0.04 mmol) in the corresponding solvent (5 mL) was stirred for 40 min. After addition of (*E*)-1,3-diphenylallyl acetate **4** (0.1 mL, 0.5 mmol), the solution was stirred for another 15 min, followed by the addition of dimethyl malonate (0.1 mL, 0.87 mmol), BSA (0.22 mL, 0.87 mmol), and potassium acetate (0.002 g). The reaction mixture was stirred for 48 h, diluted with hexane (5 mL) and filtered through Celite. The solvents were removed at reduced pressure (40 Torr), the residue was dried *in vacuo* (10 Torr). Conversion of substrate **4** and enantiomeric excess of product **5** 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).

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