Complexing and catalytic properties of easily available chiral iminophosphite based on biphenyl-2,2′-diol

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A chiral iminophosphite ligand based on biphenyl-2,2′-diol and its chelates with rhodium(I) and palladium(II) were synthesized for the first time. Successful use of these compounds in asymmetric allylic substitution was demonstrated. The enantioselectivity of palladium-catalyzed alkylation of methyl pent-3-en-2-yl carbonate with dimethyl malonate reached 70%, that in the alkylation of 1,3-diphenylallyl acetate with dimethyl malonate was 88%. In the rhodium-catalyzed sulfonylation of 1,3-diphenylallyl acetate with sodium *p*-toluenesulfinite, the enantioselectivity was 56%.

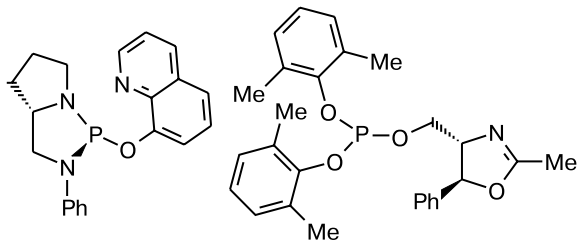
Keywords: P,N-bidentate ligands, chiral iminophosphites, rhodium, palladium, asymmetric allylation.

The last five years have seen extensive involvement of chiral ligands with three P—O and/or P—N bonds in the asymmetric metal complex catalysis.[1—3] This is due to their high π-acidity and stability against oxidation as well as synthetic availability and low cost. Indeed, the cost of monophosphite derivatives of 1,1′-bis-2-naphthol (BINOL), which are highly efficient in asymmetric hydrogenation, is only 2% of the cost of a known diphosphine ligand, 2,2′-bis(diphenylphosphino)-1,1′-binaphthyl (BINAP).[4] Heterobifunctional chiral phosphites, especially P,N-bidentate ones, possess especially broad spectrum of action. These compounds have shown themselves as excellent stereoselectors in the Cu-catalyzed conjugate addition, Ir-catalyzed hydrogenation, and Rh-catalyzed hydrosilylation.[1—3,5,6]

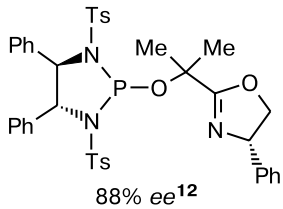
The use of these ligands in the classical Pd-catalyzed allylic substitution, *i.e.*, alkylation of 1,3-diphenylallyl acetate with dimethyl malonate, has also been reported; however, most often, the optical yields were moderate (not more than 43%).[7—10] The exceptions are listed below.

Notably, the good enantioselectivity observed in this case is due to the presence of either P* stereo center or highly effective oxazoline fragment.

Meanwhile, the use of phosphites with a peripheral imino group as catalysts has been successfully initiated.[11,13—15] Like phosphitooxazolines, they contain a chiral block with an sp^2-hybridized N donor atom, but they can be prepared starting from imino alcohols, which are much more diverse and readily available synthons than hydroxyoxazolines. The catalytic efficiency of iminophosphites can be quite comparable with that observed for other types of P,N-bidentate ligands. The present communication outlines the results of successful use of a new readily available iminophosphite ligand in the Pd-catalyzed allylation.



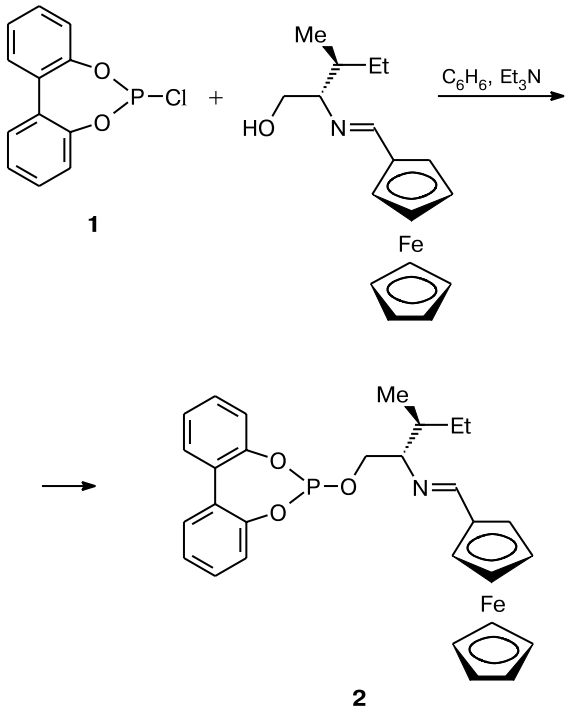
85% *ee*[9] 85% *ee*[11]



88% *ee*[12]

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 4, pp. 778—782, April, 2004.

Scheme 1



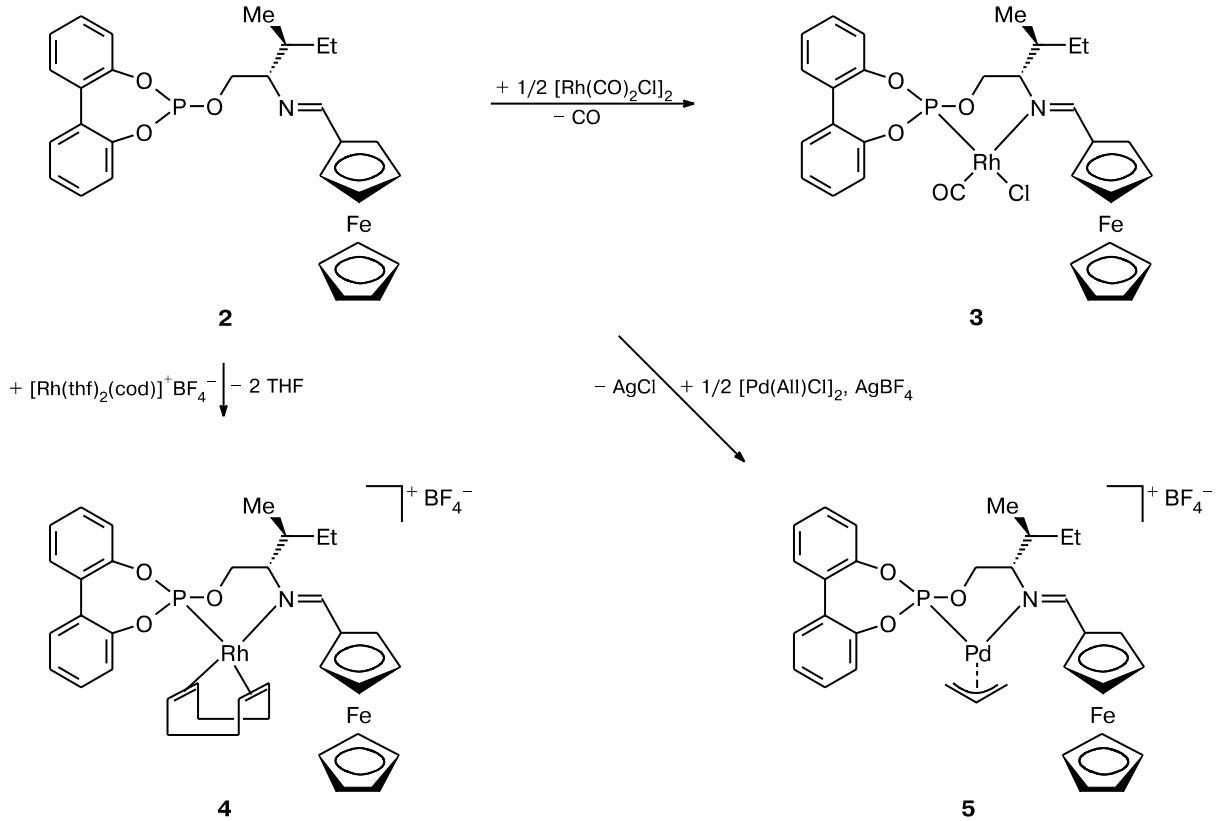
Results and Discussion

The new P,N-bidentate ligand was prepared by direct phosphorylation of the corresponding imino alcohol on treatment with reagent **1** (Scheme 1).

Note that phosphorochloridite **1** can be prepared from biphenyl-2,2′-diol and PCl_3 by two protocols, namely, in THF in the presence of Et_3N[16] and without a solvent.[17] We improved the latter protocol by using a catalytic amount of *N*-methylpyrrolidone, which had been used previously to prepare a similar phosphorochloridite on the basis of BINOL.[18] This shortened the reaction time more than 10-fold and decreased substantially the reaction temperature, giving rise to a quick procedure for the synthesis of convenient and cheap phosphorylating agent **1**.

Ligand **2** is stable during long-term storage in a dry atmosphere and is readily soluble in most organic solvents. In terms of the complexation mode, it is a typical chelating reagent. Thus it reacts with $[Rh(CO)_2Cl]_2$, $[Rh(thf)_2(cod)]^+BF_4^-$ (cod is cycloocta-1,5-diene), and $[Pd(All)Cl]_2$ (in the presence of $AgBF_4$) to give neutral and cationic metal chelates with *cis*-arrangement of the P and N atoms (Scheme 2).

Scheme 2



This is indicated, in particular, by ^{31}P NMR spectroscopy data for compounds **2**—**5** (in $CHCl_3$), which are presented below.

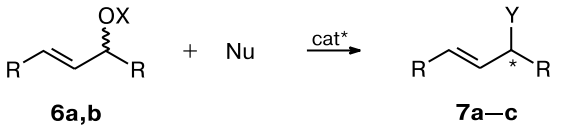
Compound	δ_P (J/Hz)
2	141.9
3	145.7 ($^1J_{P,Rh}$ = 274.7)
4	131.5 ($^1J_{P,Rh}$ = 265.8)
5	138.1, 137.9

The presence of two singlets in the ^{31}P NMR spectrum of complex **5** is due to the existence of both *exo*- and *endo*-isomers in solution.[19] The mass spectra (electrospray ionization, ESI) were found to exhibit characteristic peaks with m/z (I_{rel} (%)) 738 $[M - BF_4]^+$ (100), 630 $[M - cod]^+$, 527 $[L]^+$ (for **4**) and 674 $[M - BF_4]^+$ (100), 528 $[L + H]^+$ (for **5**). Noteworthy is also the large coordination shift $\Delta\delta_C = \delta_C(\text{complex}) - \delta_C(\text{ligand}) = 12.6$ of the signal of the azomethine C atom in the ^{13}C NMR spectrum of complex **4** (see Experimental).

Parameters of the ^{31}P NMR and IR spectra of compound **3** ($^1J_{P,Rh}$ = 274.7 Hz and ν(CO) = 2022 cm^{-1} ($CHCl_3$)) attest to pronounced π-acceptor properties of iminophosphite **2** (see Ref. 14 and references cited therein).

Ligand **2** and its metal complexes have been used in catalytic asymmetric allylic substitution (Scheme 3).

Scheme 3



6a,b **7a—c**

6	R	X
a	Me	CO_2Me
b	Ph	COMe

7	R	Y
a	Me	$CH(CO_2Me)_2$
b	Ph	$CH(CO_2Me)_2$
c	Ph	*p*-$TolSO_2$

cat* is catalyst

The results are summarized in Tables 1—3. Note that an optical yield of 70% *ee* attained in the alkylation of methyl pent-3-en-2-yl carbonate (**6a**) with dimethyl malonate (see Table 1, run *2*) is rather high for this difficult-to-optimize substrate.[20]

Table 1. Data on the catalytic allylic alkylation of **6a** (Nu = $CH_2(CO_2Me)_2$) catalyzed by Pd complexes of ligand **2**

Run	Catalyst	Solvent	Yield of **7a**	*ee*
			%	
1	**5**	CH_2Cl_2	80	54 (*R*)
2	$[Pd(All)Cl]_2$/2L	CH_2Cl_2	76	70 (*R*)
3	$[Pd(All)Cl]_2$/2L	THF	90	46 (*R*)

Table 2. Results of catalytic allylic alkylation of **6b** (Nu = $CH_2(CO_2Me)_2$) catalyzed by palladium and rhodium complexes with ligand **2**

Run	Catalyst	Solvent	Conversion of **6b**	*ee*
			%	
1	**5**	CH_2Cl_2	95	88 (*R*)
2	**5**	THF	26	70 (*R*)
3	$[Pd(All)Cl]_2$/2L	CH_2Cl_2	89	73 (*R*)
4	$[Pd(All)Cl]_2$/4L	CH_2Cl_2	16	1 (*S*)
5	$[Pd_2(dba)_3]\cdot CHCl_3$/2L*	CH_2Cl_2	2	7 (*R*)
6	**4**	CH_2Cl_2	3	41 (*R*)
7	**4**	THF	0	—

* dba is dibenzylideneacetone.

Table 3. Data of catalytic allylic sulfonylation of **6b** (Nu = *p*-$TolSO_2Na$, THF as the solvent) catalyzed by palladium and rhodium complexes with ligand **2**

Run	Catalyst	Yield of **7c**	*ee*
		%	
1	**5**	11	25 (*S*)
2	$[Pd(All)Cl]_2$/2L	13	29 (*S*)
3	$[Pd(All)Cl]_2$/4L	17	41 (*S*)
4	**4**	13	56 (*S*)

Good enantioselectivity (up 88% *ee*, see Table 2, run *1*) has also been observed in the alkylation of a more sterically hindered substrate, 1,3-diphenylallyl acetate (**6b**). Attention is attracted by the dependence of the obtained results on the nature of the solvent (see Table 2, runs *1* and *2*) and the catalyst. The notable increase in the asymmetric induction on passing from the $[Pd(All)Cl]_2$/2L catalyst system to complex **5** (see Table 2, runs *1* and *3*) can be explained by favorable influence of the replacement of the outer-sphere Cl^- anion by BF_4^-. Of equal interest is the dramatic decrease in the degree of conversion and enantioseletivity (with inversion of the absolute configuration of product **7b**) upon an increase in the L/Pd molar ratio (see Table 2, runs *3* and *4*). In our opinion, this may be due to the destruction of the catalytically active chelate, $[Pd(All)(P^{\frown}N)]^+Cl^-$ (the character "⌒" designates the residue of the ligand except the P and N atoms) upon the action of excess ligand. It is also noteworthy that the use of additional catalyst systems results in a substantial decrease in the degree of conversion and the optical yield (see Table 2, runs *5*—*7*).

The catalytic reactions involving ligand **2** are rather sensitive also to the nature of the nucleophile. In particular, in the allylic sulfonylation of 1,3-diphenylallyl acetate (**6b**), somewhat lower values of the enantiomeric excess were obtained (*cf.* data of Tables 2 and 3). Note that the use of Rh complex **4**, instead of Pd catalysts,

leads to a substantial enhancement of the asymmetric induction (see Table 3, run *4*). To the best of our knowledge, this is the first example of Rh-catalyzed allylic sulfonylation.

Our attempts to perform allylic amination of substrates **6a,b** (Nu = $PhCH_2NH_2$) failed even on refluxing the reaction mixtures (cat* is $[Pd(All)Cl]_2$/2L or complex **5**) in THF for 48 h. Generally, imino phosphite **2** can be considered as a specific ligand for enantioselective catalytic processes involving C-nucleophiles. Indeed, the level of enantioselectivity equal to 88%, attained with this ligand in the Pd-catalyzed alkylation of 1,3-diphenylallyl acetate with dimethyl malonate, is quite comparable with that obtained for the above-mentioned best P,N-bidentate phosphite-type ligands (85—88%).

Experimental

^{31}P and ^{13}C NMR spectra were recorded on a Bruker AMX-400 spectrometer (161.98 and 100.61 MHz) relative to 85% H_3PO_4 in D_2O and $CDCl_3$ (δ_C 76.91), respectively. The ^{13}C NMR signals were assigned using the DEPT procedure; in the assignment of signals of the coordinated cod ligand in the spectrum of complex **4**, data from a previous publication were also used.[21] The EI mass spectra (70 eV) were recorded on a Varian MAT-311 instrument, while the electrospray ionization (ESI) mass spectra were run on a Micromass Bio II-ZS instrument. The IR spectra of solutions in $CHCl_3$ were recorded on a Specord M-80 spectrophotometer in a polyethylene cell. The optical yields of compounds **7a** and **7c** were determined by GC (a DP-TFA-γ-CD chiral column) and HPLC (a (*R,R*)-WHELK-01 chiral column), respectively, according to recommendations from published studies.[13,19] The degree of conversion of substrate **6b** and the optical yield of product **7b** were found by HPLC (a Daicel Chiralcel OD-H chiral column), as described previously.[22]

All reactions were carried out under dry argon in anhydrous solvents; PCl_3 was distilled directly prior to use. Biphenyl-2,2′-diol and (2*S*,3*S*)-2-(ferrocenylideneamino)-3-methylpentan-1-ol[14] were dried *in vacuo* (1 h, 1 Torr) before being used in the reaction; Et_3N was distilled over KOH and then, immediately prior to use, over $LiAlH_4$. The initial complexes, $[Rh(CO)_2Cl]_2$, $[Rh(thf)_2(cod)]^+BF_4^-$, $[Pd(All)Cl]_2$, and $[Pd_2(dba)_3]\cdot CHCl_3$ were prepared by known procedures.[23—26] Rhodium complex **3** was prepared and characterized by spectroscopy in a solution in $CHCl_3$ without isolation, as described previously.[27] Complexes **4** and **5** were synthesized according to the corresponding protocols.[19,28]

Catalytic experiments on allylic alkylation of compound **6a** with dimethyl malonate and allylic sulfonylation of **6b** with sodium *p*-toluenesulfinite was carried out by reported procedures.[13,19] The initial substrate **6b** (1,3-diphenylallyl acetate) was synthesized by a known protocol.[25] Bis(trimethylsilyl)acetamide (BSA) and dimethyl malonate (Acros Organics) were used as received.

2-Chlorodibenzo[*d,f*][1,3,2]dioxaphosphepine (1). *N*-Methylpyrrolidone (0.01 g, 0.1 mmol) was added to a suspension of biphenyl-2,2′-diol (2 g, 10.7 mmol) in PCl_3 (3.3 mL, 37.5 mmol), and the mixture was refluxed for 15 min to become completely homogeneous. The excess of PCl_3 was removed *in vacuo* (40 Torr), the residue was dissolved in 10 mL of toluene, and the solution was concentrated *in vacuo* (40 Torr) to remove traces of PCl_3. The product was distilled *in vacuo* (b.p. 125—126 °C, 1 Torr). Yield 2.29 g (85%). The spectroscopic and physicochemical characteristics of product **1** fully correspond to published data.[16,17]

(2′*S*,3′*S*)-2-{2′-[(Ferrocenylidene)amino]-3′-methylpentyloxy}dibenzo[*d,f*][1,3,2]dioxaphosphepine (2). Reagent **1** (1 g, 4 mmol) and Et_3N (0.56 mL, 4 mmol) were dissolved in 12 mL of toluene, and (2*S*,3*S*)-3-methyl-2-(ferrocenylidenamino)pentan-1-ol (1.252 g, 4 mmol) was added with vigorous stirring and cooling to 0 °C. The resulting solution was stirred for 10 min, heated to boiling, and cooled to 20 °C, and $Et_3N\cdot HCl$ was filtered off. Hexane (15 mL) was added to the filtrate, the precipitate was filtered off, and the filtrate was concentrated *in vacuo* (40 Torr). The resulting solution was evacuated (1 h, 1 Torr) to give compound **2** in 1.935 g (92%) yield as a red resin. Found (%): C, 66.05; H, 5.73; N, 2.66. $C_{29}H_{30}FeNO_3P$. Calculated (%): C, 66.21; H, 5.62; N, 2.54. ^{13}C NMR ($CDCl_3$), δ: 10.8, 15.5 (both s, Me); 25.4 (s, CH_2); 35.9 (s, CH); 66.2 (s, CH_2O); 68.2, 68.4, 68.9, 70.1 (all s, C_{Fc}); 76.1 (s, CHN); 80.4 (s, $C_{Fc\ (ipso)}$); 121.6, 121.9, 124.7, 124.8, 129.0, 129.6 (all s, CH_{Ph}); 130.6 (d, C_{Ph}, ^3J = 2.7 Hz); 130.8 (d, C_{Ph}, ^3J = 3.4 Hz); 149.6 (d, C_{Ph}, ^3J = 4.9 Hz); 149.8 (d, C_{Ph}, ^3J = 5.3 Hz); 161.5 (s, CH=). MS (EI, 70 eV), *m/z* (I_{rel} (%)): 527 $[M]^+$ (3), 443 (92), 377 $[M - C_6H_4—C_6H_4 + 2\ H]^+$ (100).

(2′*S*,3′*S*)-2-{2′-[(Ferrocenylidene)amino]-3′-methylpentyloxy}dibenzo[*d,f*][1,3,2]dioxaphosphepine-*P,N*)(η⁴-cycloocta-1,5-diene)rhodium(1+) tetrafluoroborate (4). Yield 88%. Red-orange powder, m.p. 147—149 °C (with dec.). Found (%): C, 53.85; H, 5.13; N, 1.70. $C_{37}H_{42}BF_4FeN_3OPRh$. Calculated (%): C, 54.03; H, 5.27; N, 1.58. ^{13}C NMR ($CDCl_3$), δ: 10.7, 14.2 (both s, Me); 25.3 (s, CH_2); 26.1, 29.2, 29.6, 34.1 (all s, CH_2(cod)); 38.4 (s, CH); 67.5 (s, CH_2O); 69.4, 70.1, 70.5, 72.1 (all s, C_{Fc}); 77.5 (s, CHN); 79.0 (s, $C_{Fc\ (ipso)}$); 74.8 (d, $^1J_{C,Rh}$ = 10.4 Hz) and 80.1 (d, $^1J_{C,Rh}$ = 10.8 Hz) (CH= (cod) *trans*-N); 113.4 (dd, $^1J_{C,Rh}$ = 13.8 Hz, $^2J_{C,P}$ = 3.0 Hz) and 114.0 (dd, $^1J_{C,Rh}$ = 11.2 Hz, $^2J_{C,P}$ = 4.2 Hz) (CH= (cod) *trans*-P); 120.7—148.1 (C_{Ph}); 174.1 (s, CH=).

(2′*S*,3′*S*)-2-{2′-[(Ferrocenylidene)amino]-3′-methylpentyloxy}dibenzo[*d,f*][1,3,2]dioxaphosphepine-*P,N*)(π-allyl)palladium(2+) tetrafluoroborate (5). Yield 85%. Red powder, m.p. 167—169 °C (with dec.). Found (%): C, 50.46; H, 4.63; N, 1.84. $C_{32}H_{35}BF_4FeN_3OPPd$. Calculated (%): C, 50.62; H, 4.81; N, 2.02.

Palladium- and rhodium-catalyzed allylic alkylation of 1,3-diphenylallyl acetate with dimethyl malonate (general procedure). A solution of $[Pd(All)Cl]_2$ (3.7 mg, 0.01 mmol) or $[Pd_2(dba)_3]\cdot CHCl_3$ (10.3 mg, 0.01 mmol) and ligand **2** (0.02—0.04 mmol) in 5 mL of the corresponding solvent was stirred for 40 min (alternatively, ready complex **4** or **5** (0.02 mmol) was dissolved). 1,3-Diphenylallyl acetate (**6b**) (0.1 mL, 0.5 mmol) was added and the mixture was stirred for 15 min. Dimethyl malonate (0.1 mL, 0.87 mmol), BSA (0.22 mL, 0.87 mmol) and NaOAc (2 mg) were added. The reaction mixture was stirred for 48 h, diluted with 5 mL of the same solvent, and filtered through a column with Celite. The filtrate was concentrated *in vacuo* (40 Torr) and the residue was dried *in vacuo* (12 h, 10 Torr). The product was a colorless oil that solidified on storage.

The authors are sincerely grateful to S. E. Lyubimov (S. A. Esenin Ryazan State Pedagogical University) for the help in the preparation of ligand **2**. The authors wish to thank the Regis Technologies company (USA), which kindly provided the (*R,R*)-WHELK-01 chiral column and the Daicel Chemical Industries Ltd. company (Japan) for providing the Daicel Chiralcel OD-H chiral column.

This study was financially supported by the Russian Foundation for Basic Research (Project No. 03-03-32181) and the Council for the Grants for Young Russian Doctors of Science (Project No. MD 21.2003.03).

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Received September 29, 2003;
in revised form November 14, 2003