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New electron-deficient aminophosphonite–phosphite ligands for asymmetric hydroformylation of styrene

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Abstract

We have developed a new class of electron-deficient aminophosphonite–phosphite ligands based on (1*R*,2*S*)-ephedrine and chlorophosphites. These ligands were tested in rhodium catalysed hydroformylation of styrene and led to reactions with high regio and chemoselectivities into the branched aldehyde but nevertheless with low enantioselectivities. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Hydroformylation; Aminophosphonite–phosphite; Rhodium; Enantioselective catalysis; Styrene; Hydratropaldehyde

1. Introduction

Numerous chiral diphosphines [1–3], diphosphinites [4–9] and diphosphites [10–19] have been synthesised and used as stereodirecting auxiliaries in enantioselective catalysis. For example, diphosphites have been involved in various asymmetric processes including hydrogenation [10–13], hydrocyanation [14–18] and hydrosilylation [19]. Recently, both the breakthrough accomplished by Takaya and co-workers with the BINAPHOS and the ready accessibility to optically pure diols have popularised the synthesis and evaluation of many electron-deficient phosphine–phosphite and diphosphite ligands. The latter can bear stereogenic elements in the chiral backbone along with others in the P/O heterocyclic coordination sites. Such ligands have been investigated mainly for their potential in platinum and rhodium based hydroformylation reactions [20–26]. It should be emphasised that the chiral aldehydes produced are attractive building blocks for organic synthesis, some of which provide an entry to a class of anti-inflammatory drugs.

We have already reported on the synthesis of aminophosphine-phosphinites (AMPP) and aminophosphine-carboxyphosphinites (AMPCP) and their use in rhodium and platinum based enantioselective hydroformylations [27–29]. These ligands are obtained from reaction of natural aminoalcohols and α-aminoacids, respectively with various chlorophosphines. In view of the specific properties expected for diphosphanes featuring enhanced electron deficient coordination sites, we thought to explore the synthesis and evaluation in enantioselective hydroformylation of ligands obtained from aminoalcohols and chlorophosphites. As the ephedrine based AMPPs have shown to be the most efficient of the series for the mentioned catalytic process [30], we devoted our efforts to the synthesis and use in hydroformy-

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lation of ligands derived from (1*R*,2*S*)-ephedrine bearing various diols as terminal groups on the phosphorous atoms. The four diols chosen to synthesise the heterocyclic end groups were the achiral 1,2-ethane-diol, catechol, 2,2′-biphenol and optically pure (+)-(*R*,*R*)-diethyltartrate. We report here on the synthesis and characterisation of the above-mentioned new ligands and on their use in the enantioselective hydroformylation of styrene.

2. Experimental

2.1. General methods and chemicals

All the reactions were carried out under a dry N_2 atmosphere. The ligands are air-sensitive and are best stored in Schlenk tubes under N_2 at low temperature. 2-chloro-1,3,2-dioxaphospholane (**2a**), *o*-phenylenephosphorochloridite (**2b**), phosphorous trichloride, (1*R*,2*S*)-ephedrine (**1**), L-(+)-diethyltartrate were of commercial quality and used as purchased (Acros). 2,2′-biphenol was dried by azeotropic distillation with toluene. Styrene was purified by passage through basic alumina and distilled prior to use. $Rh_4(CO)_{12}$ was obtained from Strem and used as received. Toluene, tetrahydrofuran and diethylether were distilled from Na/benzophenone. Triethylamine was dried over potassium hydroxide and distilled in the presence of 2% phenyl isocyanate. The conversion, chemoselectivity, and regioselectivity of the hydroformylation reactions were determined by GC analysis on a CP Sil 5 CB column mounted on a Chrompack 9001 chromatograph that was equipped with a FID detector. The determination of the optical purity was performed on a 25 m × 0.32 mm Chirasil-Dex column on the alcohols obtained after reduction of the aldehydes.

All NMR spectra were recorded at ambient probe temperature in $CDCl_3$ on a Brüker AM-300 spectrometer operating at 300, 75 and 121 MHz respectively for ^1H, ^{13}C, and ^{31}P. $^1H(\delta)$ was referenced to residual $CHCl_3$ at 7.24; ^{13}C (ppm) to $CDCl_3$ at 77.0; ^{31}P (ppm) to external 85% H_3PO_4 at 0.0 ppm. All coupling constant (J) values are in Hz. For the ^{31}P NMR monitoring of the ligand syntheses, the spectra were recorded on crude samples taken from the reaction mixtures (unlocked at constant field).

2.2. General procedure for the synthesis of the ligands

2.2.1. Synthesis of the chlorophosphites (**2c** and **2d**) [11,31]

2c: A Schlenk tube was charged with 2,2′-biphenol (3.64 g, 14 mmol), toluene (35 ml), and a stir bar and the solution was cooled to 0°C. Then, a mixture containing PCl_3 (2.06 g, 15 mmol), toluene (15 ml), and Et_3N (5 ml) was added slowly via cannula and the medium was allowed to come to ambient temperature. After overnight stirring at ambient temperature, the mixture was filtered through a sintered glass funnel. The precipitate was washed with toluene (50 ml). The solvent and the slight excess of PCl_3 were evaporated from the combined filtrates under reduced pressure. A distillation under vacuum yielded the pure chlorophosphite **2c** in 90% yield. ^{31}P NMR (ppm): 181.5 (s).

2d was synthesised following the procedure and the workup given for **2c** starting from (+)-diethyltartrate (93% yield). The chlorophosphite was used without distillation, as the purity was satisfactory for a direct use in the next step. ^{31}P NMR (ppm): 172 (s).

2.2.2. General synthesis of ligands **4a–d**

In a typical experiment, a Schlenk tube was charged with (1*R*,2*S*)-ephedrine (**1**) (1.98 g, 12 mmol) and THF (30 ml). Then, a solution of the appropriate chlorophosphite in a mixture of THF (20 ml) and Et_3N (6 ml) was added slowly via cannula. The reaction mixture was stirred vigorously at room temperature and monitored by ^{31}P NMR. At the end of the reaction, the excess of chlorophosphite and eventual phosphorous impurities were removed by filtration through basic alumina (1 cm × 3 cm). The solvents were evaporated under reduced pressure affording ligands **4a–d** which were further dried under oil-pump vacuum. Ligand **4c** could not be obtained pure enough for an evaluation in catalysis.

4a: Yield = 85%. $^{31}P\{^1H\}$ NMR (ppm): 135 (s, P(O)), 143 (s, P(N)). ^1H NMR: δ 1.22 (d, $J_{HH} = 7$, 3H, CH_3), 2.4 (d, $J_{HP} = 5$, 3H, CH_3(N)), 3.8–4.2 (m, 9H, 4 CH_2 and CH(N)), 4.9 (dd, $J_{HH} = 9.5$, $J_{HP} = 6$, 1H, CH(O)), 7.25 (m, 5H, Haro). $^{13}C\{^1H\}$ NMR (ppm): 15.1 (d, $J_{CP} = 7$, CH_3), 27 (s, CH_3(N)), 57.6 (d, $J_{CP} = 36$, CH(N)), 64 (d, $J_{CP} = 9$, CH_2), 64.6 (d, $J_{CP} = 9$, CH_2), 78.6 (d, $J_{CP} = 12.5$, CH(O)), 128–140 (Caro). Anal. Calcd. For $C_{14}H_{21}NO_5P_2$: C,

48.70; H, 6.13; N, 4.06. Found: C, 49.05; H, 6.12; N, 3.98.

4b: Yield = 21%. $^{31}P\{^1H\}$ NMR (ppm): 128 (s, P(O)), 148 (s, P(N)). ^1H NMR: δ 1.1 (d, $J_{HH} = 7$, 3H, CH_3), 2.15 (d, $J_{HP} = 5$, 3H, CH_3(N)), 3.5 (m, 1H, CH(N)), 4.7 (m, 1H, CH(O)), 6.7–7.2 (m, 13H, Haro).

4d: Yield = 60%. ^{31}P $\{^1H\}$ NMR (ppm): 144 (s, P(O)), 158 (s, P(N)). ^1H NMR: δ 1.15 (d, $J_{HH} = 7$, 3H, CH_3), 1.27 (t, $J_{HH} = 12$, 12H, CH_3(Et)), 2.23 (d, $J_{HP} = 5$, 3H, CH_3(N)), 4.2 (m, 8H, CH_2(Et)), 4.4–4.5 (m, 4H, *C*H(CO)), 4.8 (dd, $J_{HH} = 8.5$, $J_{HP} = 5.5$, 1H, CH(O)). $^{13}C\{^1H\}$ NMR (ppm): 14 (d, $J_{CP} = 7$, *C*H_3(CH)), 16.3 (s, CH_3(Et)), 26 (s, CH_3(N)), 58 (dd, $J_{CP} = 38$ and 7, CH(N)), 62 (s, CH_2(Et)), 75 (dd, $J_{CP} = 21$ and 8, CH(O)), 85 (d, $J_{CP} = 20$, *C*H(CO), 127–141 (Caro), 169 (s, CO).

2.2.3. Synthesis of ligands ***5a–d***

Ligands **5a–d** have been synthesised following the above procedure starting from (1*R*,2*S*)-Ph-Ephos-NH [32] (4.19 g, 12 mmol). An identical workup led to the desired ligands.

5a: Yield = 44%. $^{31}P\{^1H\}$ NMR (ppm): 111 (s, P(O)), 143 (s, P(N)). ^1H NMR: δ 1.35 (d, $J_{HH} = 7$, 3H, CH_3), 2.3 (d, $J_{HP} = 5$, 3H, CH_3(N)), 3.7–4.2 (m, 5H, 2 CH_2 and CH(N)), 4.72 (dd, $J_{HH} = 7.8$, $J_{HP} = 8.8$, 1H, CH(O)), 7–7.6 (m, 15H, Haro). $^{13}C\{^1H\}$ NMR (ppm): 16.6 (d, $J_{CP} = 6$, CH_3)), 26.5 (s, CH_3(N)), 57.4 (dd, $J_{CP} = 37.5$ and 7.5, CH(N)), 64.4 (d, $J_{CP} = 8.5$, CH_2), 85.7 (d, $J_{CP} = 11$, CH(O)), 130–150 (Caro). Anal. Calcd. For $C_{24}H_{27}NO_3P_2$: C, 65.60; H, 6.19; N, 3.19. Found: C, 65.96; H, 6.15; N, 3.25.

5b: Yield = 68%. $^{31}P\{^1H\}$ NMR (ppm): 112 (s, P(O)), 149 (s, P(N)). ^1H NMR: δ 1.3 (d, $J_{HH} = 7$, 3H, CH_3), 2.15 (d, $J_{HP} = 5$, 3H, CH_3(N)), 3.9 (m, 1H, CH(N)), 4.7 (dd, $J_{HH} = 8.5$, $J_{HP} = 8$, 1H, CH(O)), 7–7.6 (m, 15H, Haro). $^{13}C\{^1H\}$ NMR (ppm): 16.6 (d, $J_{CP} = 7$, CH_3), 26.5 (s, CH_3(N)), 57.4 (d, $J_{CP} = 36$, CH(N)), 85.7 (d, $J_{CP} = 21$, CH(O)), 130–150 (Caro).

5c: Yield = 50%. $^{31}P\{^1H\}$ NMR (ppm): 112 (s, P(O)), 150 (s, P(N)). ^1H NMR: δ 1.46 (d, $J_{HH} = 7$, 3H, CH_3), 2.23 (d, $J_{HP} = 5$, 3H, CH_3(N)), 4.0 (m, 1H, CH(N)), 4.8 (dd, $J_{HH} = 10$, $J_{HP} = 10$, 1H, CH(O)), 7–7.6 (m, 19H, Haro). $^{13}C\{^1H\}$ NMR (ppm): 16 (d, $J_{CP} = 6$, CH_3), 26 (s, CH_3(N)), 56.9 (d, $J_{CP} = 35$, CH(N)), 64.2 (s, CH_2), 85 (s, CH(O)), 132–145 (Caro).

5d: Yield = 75%. $^{31}P\{^1H\}$ NMR (ppm): 111 (s, P(O)), 156 (s, P(N)). ^1H NMR: δ 1.25 (t, $J_{HH} = 12$, 6H, 2 CH_3(Et)), 1.34 (d, $J_{HP} = 7$, 3H, CH_3), 2.35 (d, $J_{HH} = 5$, 3H, CH_3(N)), 3.9 (m, 1H, CH(N)), 4.2 (m, 4H, CH_2(Et)), 4.4 (m, 2H, *C*H(CO)), 4.8 (dd, $J_{HH} = 9$, $J_{HP} = 6$, 1H, CH(O)), 7–7.5 (m, 23H, Haro). $^{13}C\{^1H\}$ NMR (ppm): 15.3 (d, $J_{CP} = 7$, CH_3), 16.6 (s, *C*H_3 (CH)), 26.5 (s, CH_3(N)), 57.4 (d, $J_{CP} = 36$, CH(N)), 64.4 (s, CH_2), 85.7 (d, $J_{CP} = 18$, CH(O)), 127–140 (Caro).

2.3. Synthesis of platinum complexes

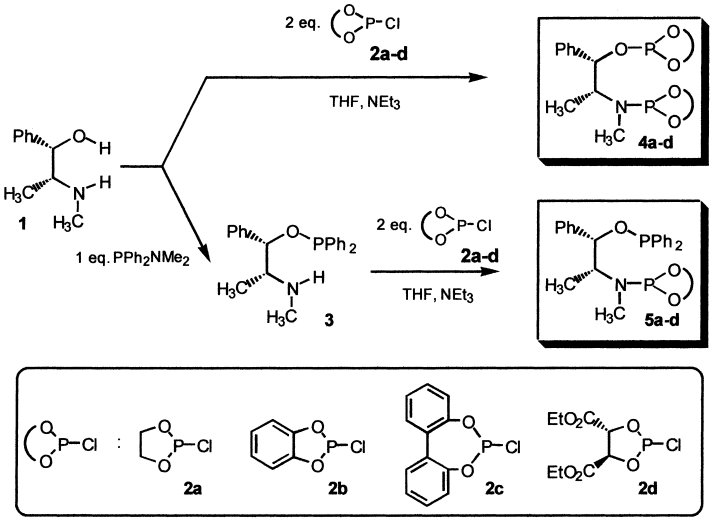
The platinum complexes were obtained by mixing one equivalent of the ligand and one equivalent of Pt(COD)Cl_2 in dichloromethane at room temperature. After 30 min stirring, the solvent was evaporated under reduced pressure. The complexes were obtained quantitatively as orange powders, which were further dried under oil pump vacuum.

2.4. Hydroformylation of styrene

In a typical experiment, a Schlenk tube was charged with a stir bar, the ligand (0.216 mmol), $Rh_4(CO)_{12}$ (26.9 mg, 0.036 mmol), and toluene (10 ml). After 15 min stirring at room temperature, the solution was transferred via cannula to a 50 ml stainless steel double walled autoclave equipped with stir bar. After a N_2 purge, styrene (6 g, 57.6 mmol), diluted in toluene (10 ml) (substrate = 2.16 mol/l), was also added via cannula. The autoclave was pressurised to 12 bar of syn-gas (H_2/CO 1/1) and then heated to the desired temperature. The pressure was kept constant throughout the whole reaction by using a gas reservoir along with a pressure regulator. The evolution of the catalytic reactions was monitored by GC analysis of aliquots of the reaction mixture. At the end of the reaction, the autoclave was cooled to room temperature, depressurised, and the reaction mixture analysed by GC in order to determine the conversion and the chemo-, regio-, and enantioselectivities.

2.5. Determination of the optical purity of the aldehyde

The toluene was removed from the crude catalytic mixture by rotatory evaporation. The crude mixture



Scheme 1.

was then dissolved in diethylether (20 ml) and an excess of $LiAlH_4$ was added. After one hour stirring at room temperature, the medium was cautiously quenched with aqueous HCl (1N) (10 ml). The aqueous layer was extracted two times with diethylether (10 ml). The combined organic phases were washed with water (2 × 20 ml), dried over $MgSO_4$, concentrated by rotatory evaporation, and analysed by GC.

3. Results and discussion

3.1. Synthesis and characterisation of the ligands

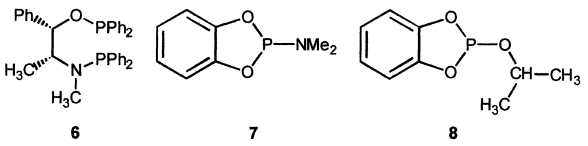
With the aim to prepare new electron deficient ligands for hydroformylation of styrene, we proposed to synthesise ephedrine based diphosphanes having alkoxy moieties at the phosphorous atoms (Scheme 1). Two chlorophosphites (**2a** and **2b**), precursors of the ligands, were commercially available and the two others (**2c** and **2d**) were synthesised according to a slight modification of reported procedures [11,31]. Then, the reaction between ephedrine and the chlorophosphites as well as the workup were carried out following the classical route used for the synthesis of AMPP diphosphanes. The new ligands (**4a–d** and **5a–d**, Scheme 1) were isolated in moderate to good yields (21–85%) and characterised as outlined in the Experimental.

Table 1
^{31}P NMR chemical shifts of the ligands[a]

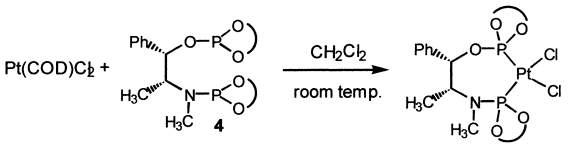
Chemical shift (ppm)	**4a**	**5a**	**4b**	**5b**	**4d**	**5c**	**5d**	**6**
δ P_O	135	111	128	112	144	112	111	113
δ P_N	143	143	148	149	158	150	156	65

[a] $CDCl_3$, 121 MHz.

The ^{31}P NMR properties of the ligands present interesting patterns (Table 1). Indeed, when comparing the chemical shifts of the fully arylated parent ligand Ph,Ph-Ephos **6** (Scheme 2) with those of ligands bearing diol ends, an inversion of the order of the signals generally observed for AMPP ligands is strongly suggested. As a matter of fact, for AMPPs, the PO signals are generally downfield from those of the PN one. As such, ligand **6** is exhibiting P(O) and P(N) signals at respectively 113 and 65 ppm whereas derivative **5a** is providing signals at 111 and 143 ppm. Accordingly, the chemical shift at 143 ppm was attributed to the PN resonance. Note



Scheme 2.



Scheme 3.

that all signals of the PN-diol moieties appeared consistently downfield from those of the PO-diols residues (see Table 1). As a check on the preceding NMR assignments, the ^{1}H-^{31}P COSY have been recorded and their analysis confirmed unambiguously the observed reversal. Moreover, a similar effect was strongly suggested by comparing the chemical shifts of 2-(dimethylamino)-1,3,2-benzodioxaphosphole **7** ($\delta P_N = 146.7$ ppm) and 2-isopropoxy-1,3,2-benzodioxa-phosphole **8** ($\delta P_O = 139$ ppm) (Scheme 2) [33,34].

If **4** turned out to be a mixture of the routinely observed intermediates PO-NH/HO-NP, the ^{1}H-^{13}C COSY study would exhibit a NMR trend identical to that reported above. Thus, in order to unequivocally probe the disubstituted nature of **4**, we have prepared the platinum complex [$PtCl_2$(**4a**)] through reaction of Pt(COD)Cl_2 with **4** in an equimolar amount (Scheme 3). The ^{31}P NMR spectra recorded on the resulting crude reaction mixture was perfectly neat without any by-product nor free ligand and the classical signal pattern of platinum complexes bearing a chelated diphosphine was observed. Indeed for [$PtCl_2$(**4a**)], P(O) and P(N) signals were respectively at 78 and 100 ppm and constituted by two doublets due to the coupling between the two phosphorous atoms ($J_{PP} = 19$ Hz) accompanied by the ^{195}Pt satellites ($J_{PtP(N)} = 5650$ Hz, $J_{PtP(O)} = 5454$ Hz).

3.2. Catalysis

These new ligands were next examined in the enantioselective hydroformylation of styrene (Scheme 4).

Table 2
Asymmetric hydroformylation of styrene catalysed by rhodium complexes[a]

Run	Ligand	Temp. (°C)	Conv. (%)[b]	b/n[b]	Ethyl-benzene	e.e. (%)[c]
1	**6**	40	15	8	0	30
2	**5a**	40	66	11	3	5
3	**4a**	80	100	9	0	8
4	**5b**	40	71	14	–	5
5	**4b**	80	45	1.5	–	5
6	**5c**	40	86	9	–	15
7	**5d**	40	88	9	–	20
8	**4d**	80	94	4	–	8

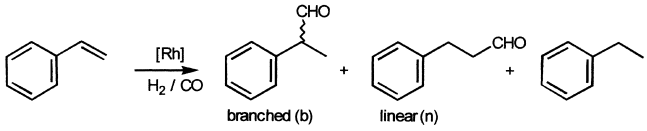
[a] Reactions were carried out in a 50 ml stainless steel autoclave in toluene (20 ml) [Rh] = 5.41×10^{-3} mol/l; P(H_2/CO : 1/1) = 12 bar; Precatalyst = $Rh_4(CO)_{12}$; Ligand/Rh = 1.5; S/Rh = 400; t = 48 h.

[b] Conversion and b/n (branched aldehyde/linear aldehyde) determined by GC analysis.

[c] Determined by GC analysis (Chirasil DEX) on the corresponding alcohol obtained by reduction of the aldehyde with $LiAlH_4$. All runs provided branched aldehyde with *R* configuration.

The results are summarised in Table 2. Generally, high chemoselectivities and good to high regioselectivities were achieved in favour of the branched aldehyde. Ethylbenzene is almost never formed as usually observed using rhodium catalysts [29].

In the case of the earlier reported (1*R*,2*S*)-Ph,Ph-Ephos **6** [27] with two phenyl entities on each phosphorous atom, only 15% conversion has been reached in 48 h with an e.e. value of 30% (run 1). When the aminophosphine group was replaced by an aminophosphonite moiety with an achiral diol at the P centre as in ligands **5a–c**, the activity increased significantly since conversions up to 86% could be achieved when applying identical experimental conditions (runs 2, 4, and 6). The selectivities into the branched aldehyde ranged from 90 to 93.5%. Nevertheless, a decrease in enantioselectivity was observed (5–15%). When ligand **5d** having the chiral diethyltartrate at the aminophosphorous centre was used, the



Scheme 4.

reaction provided the branched aldehyde in 80% yield and 20% e.e. (run 7). Hence, the acidity increase at one end of the ligand led to a faster hydroformylation. The ligands of type **4** with diols at both phosphorous ends were less efficient in terms of activity, regio, and enantioselectivity (runs 3, 5, and 8). Moreover, no reaction occurred at all at 40°C. This might indicate that a steric and electronic asymmetry between the two ends is required for efficient ephedrine based ligands. It was not possible to evaluate an eventual cooperative effect between the chirality of the starting amino alcohol and that of the diol end groups because of the inaccessibility to all diastereomers of the reported ligands.

4. Conclusion

In summary, ligands containing various diols as part of the heterocyclic phosphorous coordination sites can be synthesised in good yields. These ligands induce high chemo and regioselectivities when involved in rhodium based hydroformylation of styrene. Unfortunately, they are poor chiral modifiers for an enantioselective process since a maximum of 20% e.e. into hydratropaldehyde could be obtained. Research is under way to apply these ligands in other enantioselective reactions.

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