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Enantioselective Pd-catalysed allylation with BINOL-derived monodentate phosphite and phosphoramidite ligands

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Abstract

P-monodentate phosphite (**1**) and phosphoramidite (**2**) ligands have been synthesised from (*S*)-BINOL. Complexation of the new ligands with $[Rh(CO)_2Cl]_2$ and $[Pd(allyl)Cl]_2$ has been found to give neutral and cationic complexes $[Rh(CO)Cl(L)]_2$ and $[Pd(allyl)(L)_2]^+BF_4^-$, correspondingly. Applicability of these ligands in asymmetric C*–C, C*–N, C*–S bond formation and alkene hydrogenation reactions has been demonstrated. The phosphoramidite **2** showed higher enantioselectivity than its phosphite analogue **1** and provided good enantioselectivity in the Pd-catalysed allylic substitution of 1,3-diphenylallyl acetate with dimethyl malonate (up to 90% ee), sodium *para*-toluene sulfinate (up to 75% ee), pyrrolidine (up to 65% ee) and sodium diformylamide (up to 68% ee), as well as in the Rh-catalysed hydrogenation of dimethyl itaconate (up to 76% ee).

Keywords: Monodentate ligands; Allylic substitution; Hydrogenation

1. Introduction

Phosphite (type A) and phosphoramidite (type B) derivatives of BINOL (Fig. 1) represent the most efficient group of P-monodentate ligands for asymmetric metal complex catalysis.

They showed excellent results in enantioselective Rh-catalysed hydrogenation, Cu-catalysed addition of organozinc reagents, Ir-catalysed allylic substitution, Pd-catalysed hydrosilylation-oxidation [1–6]. BINOL-based compounds may have either exclusively axial chirality or contain additional C*-stereocentres. The latter group has been well developed during the last years, but axially chiral ligands are easy to prepare and use commercially available chiral BINOL as the only optically active precursor. Besides, due to the lack of additional C*-stereocentres, inside this group can be made a more correct comparison of catalytic efficiency of structural types A and B which differ significantly in their electronic characteristics. Thus, in the Rh-catalysed hydrogenation of prochiral ethers of unsaturated carboxylic acids and enamides ligands of both types A and B demonstrate good results (>90% ee) [7–13]. Phosphoramidites of type B provide higher enantioselectivity in the Ir-catalysed allylic alkylation of dienyl esters [14], Ru-catalysed hydrogenation of ketones [15] and Rh-catalysed cyclisation of aromatic imines [16]. On the contrary, A-type aryl phosphites (R = Ar) perform better in the Ir-catalysed allylic alkylation of esters of cinnamyl alcohol with dimethyl malonate [17,18]. Therefore, testing of both types of ligands is recommended for the optimisation of any new catalytic process.

Surprisingly, in the literature there are no examples of using simple P-monodentate phosphite and phosphoramidite derivatives of BINOL in the Pd-catalysed asymmetric allylation. The only exception is our article [19], where we reported moderate optical yields. Starting the present research we aimed at the increasing of enantioselectivities and broadening the range of applied nucleophiles. Additionally, we made a

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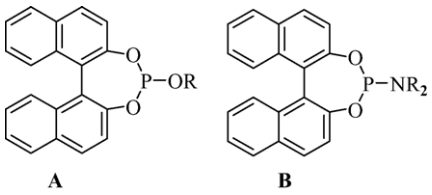


Fig. 1. Phosphite and phosphoramidite derivatives of BINOL.

comparison of ligands of types A and B, which have very different electronic properties (e.g. π-acceptor ability), in the Pd-catalysed asymmetric allylation of 1,3-diphenylallyl acetate, as well as in the Rh-catalysed hydrogenation of dimethyl itaconate and hydrosilylation of acetophenone.

2. Experimental

2.1. General methods

IR spectra were recorded on a Specord M80 or Nicolet 750 instruments. ^{31}P and ^{13}C NMR spectra were recorded on a Bruker AMX-400 instrument at 162.0 and 100.6 MHz, respectively. Chemical shifts (ppm) are given relative to Me_4Si (^{13}C NMR) and 85% H_3PO_4 (^{31}P NMR). ^{19}F NMR spectra were recorded on a Bruker WP-200-SY spectrometer at 188.3 MHz, using CF_3COOH as an external reference. Complete assignment of all the resonances in ^{13}C NMR spectra was achieved by the use of DEPT techniques. Electron impact (EI) mass spectra were recorded on a Varian MAT 311 instrument with direct injection of a sample. Electrospray ionization (ESI) mass spectra were measured on a Finnigan LCQ Advantage mass spectrometer. Elemental analyses were performed at the Laboratory of Microanalysis (Institute of Organoelement Compounds, Moscow).

Conversion of substrate **7** and optical yields of product **8** were determined using HPLC (Daicel Chiralcel OD-H column) according to the literature [20]. Optical yields of product **9** were determined using HPLC ((*R,R*)-WHELK-01 column) according to the literature [21]. Optical yields of compound **10** were determined by HPLC (Daicel Chiralcel OD-H column) as described previously [22]. Optical yields of product **11** were determined using HPLC (Chiralcel OD column) according to the literature [23]. Conversion of substrate **12** and optical yields of product **13** were determined via capillary GC (column GT-A, 30 m × 0.25 mm GT-A, trifluoroacetyl γ-cyclodextrin) according to the literature [24]. Conversion of substrate **14** and optical yields of product **15** were determined by GC (Chiraldex B-DM column) as described previously [25].

All reactions were carried out under a dry argon atmosphere in freshly dried and distilled solvents; Et_3N, Pr_2NH and pyrrolidine were twice distilled over KOH and then over a small amount of $LiAlH_4$ before use. 1,1,1,3,3,3-Hexafluoropropan-2-ol was distilled over Na_2SO_4. Phosphorylating reagent (S_{ax})-2-chloro-dinaphtho[2,1-d:1′,2′-f] [1–3] dioxaphosphepine was synthesized according to the known procedure [26]. Sodium diformylamide was prepared as published [27]. Starting substrate **7** was synthesized as published [28]. (*S*)-BINOL, dimethyl malonate, BSA (*N,O*-bis(trimethylsilyl) acetamide), sodium *para*-toluene sulfinate, diphenylsilane and starting substrates **12** and **14** were commercially available.

Rhodium(I) complexes **3**, **4** were synthesized for the ^{31}P NMR and IR experiments in chloroform analogously to the known procedures [19]. The syntheses of palladium(II) complexes **5**, **6** were performed by techniques similar to that reported [19,25].

Catalytic experiments: allylic alkylation of substrate **7** with dimethyl malonate and allylic sulfonylation with sodium *para*-toluene sulfinate were performed according to appropriate procedures [19]; hydrogenation of substrate **12** and hydrosilylation of substrate **14** were carried out according to published procedures [25,29].

2.2. Synthesis

2.2.1. Synthesis of ligands *1* and *2*. General procedure

A solution of Et_3N (0.2 ml, 1.4 mmol) and the relevant alcohol or amine (1.4 mmol) in toluene (7 ml) was added dropwise at 0 °C during 15 min to a vigorously stirred solution of (S_{ax})-2-chloro-dinaphtho[2,1-d:1′,2′-f] [1–3] dioxaphosphepine (0.5 g, 1.4 mmol) in toluene (7 ml). The mixture was heated up on stirring to be boiled and then cooled down to 20 °C. Solid $Et_3N \times HCl$ was filtered off. Hexane (15 ml) was added to the filtrate. The resulting mixture was filtered and the solvent evaporated at reduced pressure (40 Torr). The product was kept in vacuo (1 Torr) for 2 h.

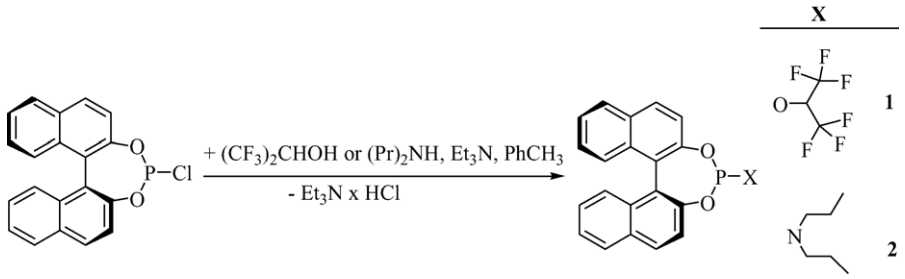
2.2.1.1. (S_{ax})-2-(Hexafluoroisopropyloxy)-dinaphtho [2,1-d:1′,2′-f] [1–3] dioxaphosphepine (1). White solid, 0.655 g (97% yield); mp, 54–55 °C; ^{31}P NMR ($CDCl_3$); δ_P: 148.6. ^{13}C NMR ($CDCl_3$): δ_C, ($J_{C,P}$, Hz): 147.3–120.2 (C_{Ar}), 122.4 (q, $^1J_{C,F}$ = 277, CF_3), 70.5 (qt, ^2J = 3.6, $^2J_{C,F}$ = 33.2, CH). ^{19}F NMR ($CDCl_3$), δ_F: 4.7 (dq), 4.5 (dq) ($^4J_{F,F}$ = 7.4, $^4J_{F,P}$ = 3.4 Hz). MS (EI), *m/z* (*I*, %): 482 (100, [*M*]+), 268 (58); 239 (39). Anal. Calc. for $C_{23}H_{13}F_6O_3P$ (%): C 57.28, H 2.72, P 6.42. Found: C 57.11, H 2.49, P 6.38.

2.2.1.2. (S_{ax})-2-(dipropylamino)-dinaphtho [2,1-d:1′,2′-f] [1–3] dioxaphosphepine (2). White solid, 0.558 g (96% yield); mp, 47–48 °C; ^{31}P NMR ($CDCl_3$); δ_P: 149.7. ^{13}C NMR ($CDCl_3$): δ_C, ($J_{C,P}$, Hz): 150.0–121.8 (C_{Ar}), 45.8 (d, ^2J = 19.5 Hz, NCH_2), 21.3 (CH_2), 11.0 (CH_3). MS (EI), *m/z* (*I*, %): 415 (56, [*M*]+), 386 (60); 315 (100). Anal. Calc. for $C_{26}H_{26}NO_2P$ (%): C 75.16, H 6.31, P 7.46. Found: C 75.44, H 6.49, P 7.37.

2.3. Cationic palladium complexes

2.3.1. [Pd(allyl)(*1*)$_2$]$^+$ BF_4^- (*5*)

White solid, 87% yield; mp, 73 °C; ^{31}P NMR ($CDCl_3$); δ_P: AX system: δ_P 144.3, *d* and δ_P 138.6, d, $^2J_{P,P'}$ 110.6 Hz,



Scheme 1.

79%; AX system: δ_P 145.8, d and δ_P 134.4, d, $^2J_{P,P'}$ 120.8 Hz, 21%.

^{19}F NMR ($CDCl_3$), δ_F: 4.0 (dq), 3.7 (dq) ($^4J_{F,F}$ = 7.5, $^4J_{F,P}$ = 3.3 Hz, CF_3); −74.7 (br.s), −74.8 (br.s) (BF_4^-). MS (ESI), *m*/*z* (*I*, %): 1112 (100, [*M* − BF_4]+), 483 (12); 87 (100, [BF_4]$^-$).

*2.3.2. [Pd(allyl)(**2**)$_2$]$^+$ BF_4^- (**6**)*

White solid, 92% yield; mp, 81 °C; ^{31}P NMR ($CDCl_3$); δ_P: AB system: δ_P 143.7, d and δ_P 142.2, d, $^2J_{P,P'}$ 102.1 Hz, 75%; AX system: δ_P 147.0, d and δ_P 142.8, d, $^2J_{P,P'}$ 115.4 Hz, 25%

MS (ESI), *m*/*z* (*I*, %): 978 (100, [*M* − BF_4]+), 417 (7); 87 (100, [BF_4]$^-$).

2.4. Catalytic experiments

2.4.1. Pd-catalysed allylic amination of 1,3-diphenylallyl acetate with pyrrolidine

A solution of [Pd(allyl)Cl]$_2$ (3.7×10^{-3} g, 1×10^{-5} mol) and appropriate ligand (2×10^{-5} to 4×10^{-5} mol) in 5 ml of appropriate solvent was stirred for 40 min (alternatively, the pre-synthesized complex **5** or **6** (2×10^{-5} mol) was dissolved in appropriate solvent (5 ml)). Then 1,3-diphenylallyl acetate (0.1 ml, 5×10^{-4} mol) was added and solution stirred for 15 min, then freshly distilled pyrrolidine (0.12 ml, 1.5×10^{-3} mol) was added and the reaction mixture stirred for 48 h. After that, a resulting solution was filtered through *Celite*. The solvent was removed in vacuum (40 Torr), and the residue dried in vacuum (10 Torr, 12 h) to obtain ((*E*)-1,3-diphenylallyl)pyrrolidine (**10**) as a cream crystalline solid. All spectroscopic data of compound **10** are in good agreement with published data [22].

2.4.2. Pd-catalysed allylic amination of 1,3-diphenylallyl acetate with NaN(CHO)$_2$

A solution of [Pd(allyl)Cl]$_2$ (3.7×10^{-3} g, 1×10^{-5} mol) and appropriate ligand (2×10^{-5} to 4×10^{-5} mol) in 5 ml of appropriate solvent was stirred for 40 min. Then 1,3-diphenylallyl acetate (0.1 ml, 5×10^{-4} mol) was added and solution stirred for 15 min, then freshly distilled Et_3N (0.06 ml, 4×10^{-4} mol) and NaN(CHO)$_2$ (0.234 g, 2.46×10^{-3} mol) were added and the reaction mixture stirred for 72 h. After that, a resulting solution was filtered, the solvent was removed in vacuum (40 Torr), and the residue was chromatographed on SiO_2 in hexane/ethyl acetate (4/1) to obtain *N*-formyl-*N*-((E)-1,3-diphenylallyl)formamide (**11**) as a colourless oil. All spectroscopic data of compound **11** are in good agreement with published data [23].

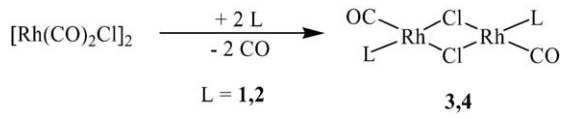
3. Results and discussion

3.1. Synthesis of the BINOL-derived monodentate ligands and their complexation with Rh(I) and Pd(II)

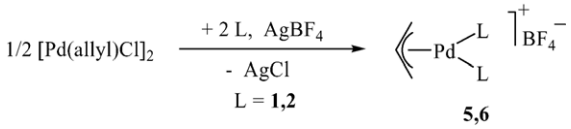
Compounds **1** and **2** were obtained according to the following scheme (Scheme 1).

They represent crystalline compounds, stable on prolonged storage. To estimate the electronic properties of **1** and **2**, their chlorocarbonyl complexes of rhodium (I) were synthesised (Scheme 2). Significantly larger $^1J_{P,Rh}$ coupling constant and ν(CO) frequency for the complex **3** (Table 1) prove higher π-acidity of phosphite **1** in comparison to phosphoramidite **2** [25,30,31].

Cationic Pd(II) complexes were obtained for the use in Pd-catalysed allylation (Scheme 3).



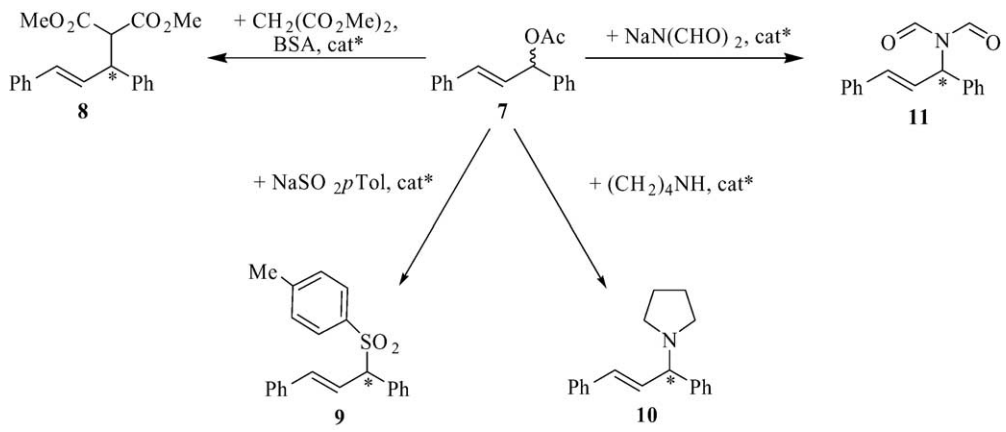
Scheme 2.



Scheme 3.

Table 1
Selected spectroscopic data for compounds **3** and **4** (in $CHCl_3$)

Compound	^{31}P NMR		IR, ν(CO), cm^{-1}
	δ_P	^1J(P,Rh) (Hz)	
3	146.1	295.1	2043
4	143.5	272.1	2008



Scheme 4.

AB and *AX* systems in their ^{31}P NMR spectra (see Experimental tail) indicate non-equivalence of two P-monodentate ligands in the coordination sphere of the palladium atom and disclose the presence of *exo*- and *endo*-isomers of complexes **5** and **6** [19].

3.2. The novel BINOL-derived monodentate ligands in asymmetric catalysis

BINOL-based ligands **1**, **2** and their pre-formed Pd complexes **5**, **6** were tested in asymmetric Pd-catalysed allylic substitution (Scheme 4, Tables 2–5). Phosphite **1** afforded products **8**, **9** and **11** with low ee's, and only for **10** a moderate enantioselectivity was observed (55% ee, Table 4, entry 1). On the contrary, phosphoramidite **2** provided good chemical and optical yields, especially in allylic sulfonylation (up to 75% ee, Table 3) and in allylic alkylation (up to 90% ee, Table 2).

Notably, in the two last mentioned processes the enantioselectivity is almost independent on the L^*/Pd ratio and nature of solvent (Tables 2 and 3). In allylic amination the applied solvent plays an important role (Tables 4 and 5). Better results of phosphoramidite **2** (in comparison to **1**) are likely to be caused by the higher electron-donating ability of the ligand. Of importance are also its higher steric demands, since the Tolman's cone angle of phosphoramidite **2** (140°, calculated by use of semi-empirical quantum mechanical AM1 technique [32]) significantly exceeds the analogous parameter for **1** (111°).

Table 2
Enantioselective allylic alkylation of **7** with dimethyl malonate (BSA, NaOAc, 20 °C, 48 h)

Entry	Catalyst	L^*/Pd	Solvent	Conversion (%)	ee (%)
1	[Pd(allyl)Cl]$_2$/**1**	1/1	THF	7	32 (*S*)
2	[Pd(allyl)Cl]$_2$/**1**	2/1	THF	6	33 (*S*)
3	**5**	1/1	THF	8	13 (*R*)
4	[Pd(allyl)Cl]$_2$/**2**	1/1	THF	68	86 (*R*)
5	[Pd(allyl)Cl]$_2$/**2**	2/1	THF	75	83 (*R*)
6	**6**	1/1	THF	99	90 (*R*)
7	**6**	1/1	CH_2Cl_2	98	79 (*R*)

Table 3
Enantioselective allylic sulfonylation of **7** with $NaSO_2pTol$ (THF, 20 °C, 48 h)

Entry	Catalyst	L^*/[Pd]	Isolated yield (%)	ee (%)
1	[Pd(allyl)Cl]$_2$/**1**	1/1	–	–
2	[Pd(allyl)Cl]$_2$/**1**	2/1	–	–
3	**5**	1/1	24	15 (*S*)
4	[Pd(allyl)Cl]$_2$/**2**	1/1	70	75 (*R*)
5	[Pd(allyl)Cl]$_2$/**2**	2/1	18	71 (*R*)
6	**6**	1/1	67	72 (*S*)

Table 4
Enantioselective allylic amination of **7** with pyrrolidine (20 °C, 48 h)

Entry	Catalyst	L^*/Pd	Solvent	Isolated yield (%)	ee (%)
1	[Pd(allyl)Cl]$_2$/**1**	1/1	THF	60	55 (*R*)
2	[Pd(allyl)Cl]$_2$/**1**	2/1	THF	52	39 (*R*)
3	**5**	1/1	THF	37	7 (*R*)
4	[Pd(allyl)Cl]$_2$/**2**	1/1	THF	69	57 (*S*)
5	[Pd(allyl)Cl]$_2$/**2**	2/1	THF	74	65 (*S*)
6	**6**	1/1	THF	40	58 (*S*)
7	**6**	1/1	CH_2Cl_2	29	1 (*S*)

Interestingly, the depicted on Fig. 2 phosphoramidite with inferior to **2** cone angle ($\theta = 109°$) provides only 60% ee in allylic alkylation and 46% ee in allylic sulfonylation [19], but nevertheless, these results are still better than those demonstrated by electron-accepting phosphite **1** which has almost the same Tolman's cone angle.

Ligands **1** and **2** were also tested in asymmetric hydrogenation of dimethyl itaconate **12** (Scheme 5; $[Rh(COD)_2]^+BF_4^-$/2L^* as the catalyst; CH_2Cl_2, 1.1 bar H_2,

Table 5
Enantioselective allylic amination of **7** with $NaN(CHO)_2$ (Et_3N, 20 °C, 72 h)

Entry	Catalyst	L^*/Pd	Solvent	Isolated yield (%)	ee (%)
1	[Pd(allyl)Cl]$_2$/**1**	1/1	THF	36	13 (*S*)
2	[Pd(allyl)Cl]$_2$/**1**	1/1	CH_3CN	41	28 (*R*)
3	[Pd(allyl)Cl]$_2$/**2**	1/1	THF	–	–
4	[Pd(allyl)Cl]$_2$/**2**	1/1	CH_3CN	44	68 (*R*)

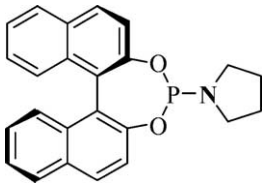
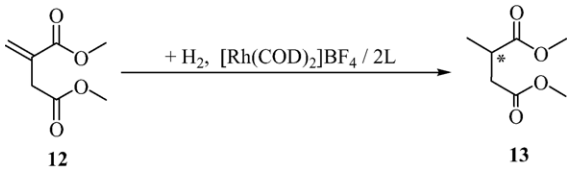


Fig. 2. Phosphoramidite with cone angle 109°.



Scheme 5.

20 °C, 24 h). Phosphoramidite **2** (48% conversion, 76% ee (*S*)) again showed better results than **1** (26% conversion, 56% ee (*S*)).

One can conclude that a pronounced π-acceptor character of phosphite **1** results in decreasing of its enantioselectivity in the described catalytic processes. But further (regarding phosphoramidites) increasing of the electron-donor ability can also lead to depression of enantioselectivity.

Thus, binaphthyldiamine-based diazaphospholidines (type C and D, Fig. 3) gave <30% ee in the Rh-catalysed hydrogenation of dimethyl itaconate **12** [33]. In general, any catalytic process with the participation of P-monodentate axially-chiral phoshite ligands seems to require fine tuning of steric and electronic parameters of the ligands.

Surprisingly, application of the BINOL-based compounds **1** and **2** in Rh and Ir-catalysed hydrosilylation of acetophenone **14** (Scheme 6, toluene, 20 °C, 24 h) resulted in practically racemic product **15** (30–60% conversion, 1–4% ee), despite the fact that rather broad range of catalytic systems was tested: $[Rh(COD)Cl]_2$, $L^*/Rh = 1–3$; $[M(COD)_2]^+BF_4^-/2L^*$ (M = Rh, Ir). Therefore, BINOL-based bidentate ligands seem to be essential to catalyse this reaction. Thus, previously described by us BINOL-derived P,N-bidentate oxazolinophosphite gave up to 58% ee in the reaction [25].

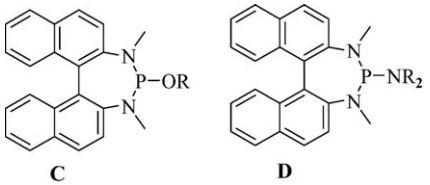
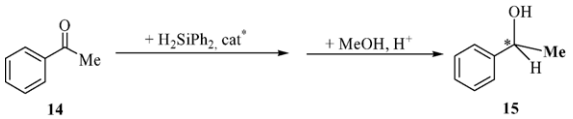


Fig. 3. Binaphthyldiamine-based diazaphospholidines.



Scheme 6.

4. Conclusions

Novel BINOL-based phosphite **1** and phosphoramidite **2** have been synthesised, characterised and successfully tested in Pd-catalysed asymmetric allylic substitution and in the Rh-catalysed asymmetric hydrogenation of dimethyl itaconate. It has been found that the more sterically hindered and better electron-donating phosphoramidite **2** represents a superior stereoselector.

Acknowledgements

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