Available online at www.sciencedirect.com
SCIENCE DIRECT®
Tetrahedron: Asymmetry
Tetrahedron: *Asymmetry* 16 (2005) 3183–3187

Influence on the enantioselectivity in allylic alkylation of the spacer between the amido group of D-glucosamine and the diphenylphosphino group

Anzhelika Konovets,[a] Katarzyna Glegoła,[a,c] Alexandra Penciu,[a] Eric Framery,[a,*] Philippe Jubault,[b] Catherine Goux-Henry,[a] K. Michał Pietrusiewicz,[c] Jean-Charles Quirion[b] and Denis Sinou[a]

[a]*Laboratoire de Synthèse Asymétrique, UMR UCBL/CNRS 5181, ESCPE Lyon, Université Claude Bernard Lyon 1, 43 boulevard du 11 novembre 1918, F-69622 Villeurbanne Cedex, France*
[b]*Laboratoire d'Hétérochimie Organique, UMR INSA/CNRS 6014, Université de Rouen, 1 rue Tesnière, F-76821 Mont-Saint-Aignan Cedex, France*
[c]*Department of Organic Chemistry, Maria Curie-Skłodowska University, ul. Gliniana 33, 20-614 Lublin, Poland*

Received 7 July 2005; revised 23 August 2005; accepted 25 August 2005

Abstract—The synthesis of a new generation of chiral phosphine-amides derived from D-glucosamine is described. The palladium-catalyzed asymmetric allylic alkylation of racemic 1,3-diphenyl-2-propenyl acetate with dimethyl malonate has been investigated. The results obtained from these new ligands showed the importance of the rigidity of the complex and of the D-glucosamine skeleton on the enantioselectivity of the reaction.

1. Introduction

Over the last 15 years, palladium-catalyzed allylic substitution has been one of the most studied catalytic reactions. The carbon–carbon and carbon–heteroatom bond formation reactions have found many applications in asymmetric synthesis. Since the first example described by Trost and Strege in 1977,[1] many asymmetric catalytic systems have been described. Amongst the best ones are Trost's *P–P* ligand[2] and Pfaltz's *P–N* ligand[3] with enantioselectivities up to 95% being obtained using these ligands. In the main, the optimization of several experimental parameters is often necessary in order to obtain both the high activity and the high enantioselectivity, and the ligand structure is one of the main factors.

In order to obtain chiral ligands, one methodology is the use of chiral natural precursors, which can easily be functionalized, and carbohydrates offer many advantages. During the last decade, many ligands derived from carbohydrates have been used in a large number of catalytic asymmetric reactions.[4] With respect to the palladium-catalyzed allylic substitution, several ligands based on carbohydrates have already been described in the literature. In addition to *P–P*, *S–S*, and *S–X* ($X = P$ or N) ligands derived from ferrocenylglucose,[5] bis-thioglycosides[6] and oxazoline-thioglucose,[7] the *P–N* ligands are amongst the most frequently described. The *P–N* ligands based on oxazoline have been the first efficient ones, with enantioselectivities up to 98% and 96% ee being obtained using a phosphinoaryloxazoline ligand derived from a carbohydrate[8] and a phosphinite-oxazoline derived from D-glucosamine.[9] A *P–N* ligand derived from a carbohydrate has also been described by Ruffo and co-workers,[10] but gave very low enantioselectivity. Finally, a *P–N* ligand derived from the glucopyranose oxime-ether[11] has also been prepared, but gave no selectivity in the model allylic substitution reactions. Two years ago, we reported on the potential of the phosphine-amide ligand **1** derived from D-glucosamine (Fig. 1).[12] With this ligand, enantioselectivities up to 97% were obtained in the palladium-catalyzed allylic alkylation of 1,3-diphenyl-2-propenyl acetate with various nucleophiles. We observed that

* Corresponding author. Tel.: +33 472 446 263; fax: +33 472 448 160; e-mail: framery@univ-lyon1.fr

0957-4166/$ - see front matter © 2005 Elsevier Ltd. All rights reserved.
doi:10.1016/j.tetasy.2005.08.035

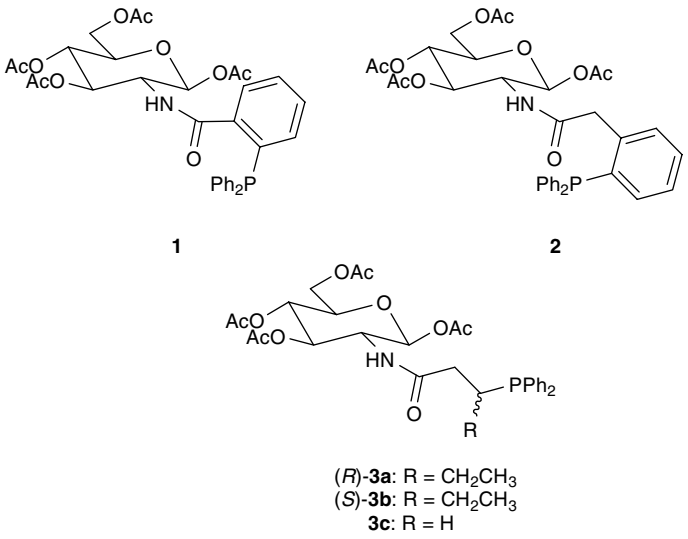


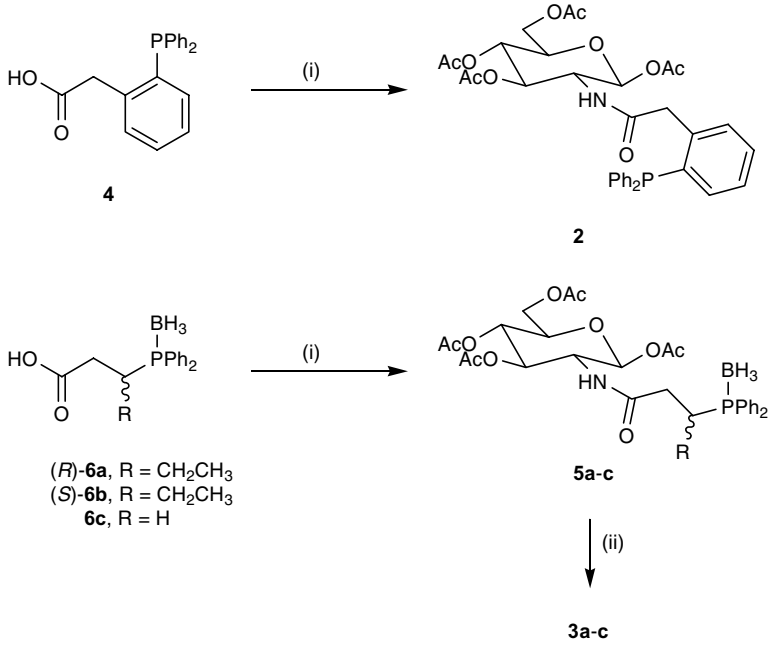
Figure 1. Phosphine-amide ligands **1**–**3** derived from D-glucosamine.

the enantioselectivity was higher with a ratio ligand/palladium of 1/1 ligand/palladium ratio, than with a ratio of 2/1. This behavior was explained by two different modes of chelation, *P*,*O*-chelation versus *P*,*P*-chelation, respectively. The *P*,*O*-chelation gave higher enantioselectivity, due to the rigidity of the formed complex, since in the case of *P*,*P*-chelation, there is more freedom in the complex.

Herein, we report on the influence of the flexibility of the spacer between the amido group of D-glucosamine and the diphenylphosphino group in palladium-catalyzed asymmetric allylic alkylations. We used the catalyst prepared from palladium and phosphine-amide ligand **1** already described,[12] and the catalyst prepared also from the new phosphine-amide ligands **2** and **3a**–**c** based on D-glucosamine (Fig. 1).

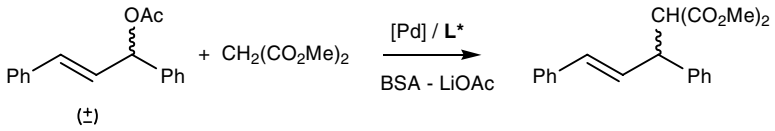
2. Results and discussion

The syntheses of D-glucosamine based diphenylarylphosphine **2** and diphenylalkylphosphines **3a**–**c** are shown in Scheme 1. The phosphine-amide ligand **2** was obtained in 62% yield by the coupling of [2-(diphenylphosphino)phenyl]acetic acid **4**, prepared as described in the literature,[13] with 2-amino-1,3,4,6-tetra-*O*-acetyl-2-deoxy-β-D-glucopyranose in CH_2Cl_2/



Scheme 1. Synthesis of the D-glucosamine-based diphenylarylphosphine **2** and diphenylalkylphosphine **3a**–**c**. Reagents and conditions: (i) 2-amino-1,3,4,6-tetra-*O*-acetyl-2-deoxy-D-glucopyranose EDC, HOBT, CH_2Cl_2/THF, rt, 24 h; (ii) DABCO, toluene, rt, 48 h.

Table 1. Palladium-catalyzed asymmetric allylic alkylation of racemic 1,3-diphenylprop-2-enyl acetate with dimethyl malonate[a]



Entry	Ligand L*	Conv. (%)[b]	Yield (%)[c]	ee (%)[d] (config.)[e]
1	**1**[f]	100	98	83 (*R*)
2	**2**	100	92	29 (*R*)
3	**3a**	100	95	76 (*R*)
4	**3b**	100	96	86 (*R*)
5	**3c**	100	99	66 (*R*)

[a] [Substrate]:[nucleophile]:[BSA]:[LiOAc]:[Pd]:[Ligand] = 25:75:75:1:1:1.
[b] Conversion determined by GC analysis.
[c] Isolated pure product.
[d] Determined by HPLC analysis (column Chiralpak AD 0.46 × 25 cm).
[e] Determined by comparison with an authentic sample.
[f] See Ref. 12.

THF in the presence of 1-[3-dimethylaminopropyl]-3-ethylcarbodiimide (EDC) and 1-hydroxybenzotriazole (HOBT). The borane–phosphine-amide complexes **5a–c** were obtained following the same procedure from carboxylic acids **6a–c**[14] in 74%, 75%, and 61% yields, respectively. The addition of 1,4-diazabicyclo[2.2.2]octane (DABCO) to a solution of **5a–c** in toluene gave the corresponding D-glucosamine-based diphenylalkylphosphine **3a–c** in almost quantitative yields.

Ligands **2** and **3a–c** were examined in the palladium-catalyzed asymmetric allylic alkylation of racemic 1,3-diphenyl-2-propenyl acetate with dimethyl malonate and were compared to the results obtained using ligand **1** (Table 1). The conditions of this palladium-catalyzed allylic substitution are the same as those described previously.[12] The reaction was performed in THF (0.125 M) using 2 mol % of $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$, 4 mol % of ligand **2** or **3a–c**, 3 equiv of dimethyl malonate and a mixture of *N*,*O*-bis(trimethylsilyl)acetamide (BSA) (3 equiv) and LiOAc (2 mol %) as base. The conversion was quantitative for each ligand after 24 h at rt. The results are summarized in Table 1.

The obtained enantioselectivities using ligands **3a–b** bearing a stereogenic center on the spacer are quite similar to, or lower than, those previously reported for amido-phosphine **1** (83% ee). Ligand **3b** bearing an (*S*)-stereogenic center gave a ee higher than ligand **1** (86% ee), while ligand **3a**, bearing an (*R*)-stereogenic center gave a lower ee (76% ee), with the same configuration being obtained in all cases. Ligands **2** and **3a–c** gave the alkylation product with the same (*R*)-configuration. Ligand **3c** with no stereogenic center in the α-position to the diphenylphosphino group gave ees up 66%. Finally, ligand **2** bearing a $CH_2C_6H_4PPh_2$ function instead of a $C_6H_4PPh_2$ gave the lowest enantioselectivity, with ees up to 29%.

In the case of the *P*,*O*-chelated complex obtained from diamidocyclohexane-phosphine,[15] the lengths of the Pd–C bonds have been measured. The length of the Pd–C bond *trans* to *P* is 2.20 Å, and the Pd–C bond *trans* to *O* is 2.07 Å, inducing in a *P*,*O*-chelated complex the nucleophilic attack predominantly at the allyl terminus *trans* to the Pd–P bond. Since the (*R*) alkylated product was obtained as the major enantiomer in all cases, the reaction most likely proceeds through complex **A** rather than complex **B** as shown in Figure 2. The difference in enantioselectivity between ligand **1** and ligand **2** could be due to the rigidity of the complex of Pd with ligand **1** bearing a phenyl ring between the amido group and the diphenylphosphino group. Effective chelation of palladium with the oxygen and phosphorus atoms gave a quite rigid six-member chelate affording higher enantioselectivity (Fig. 2). In the case of ligand **2**, there is one methylene group between the amido function and the phenyl ring thus affording a more flexible seven-member chelate with palladium. The conformation of this chelate is not favorable, and ligand **2** gave a lower ee. Concerning ligands **3a–c**, the amido function and the diphenylphosphino group are separated by two methylene units, affording a flexible six-member chelate with palladium (Fig. 2). Ligand **3c** gave ees up to 66%, ligands **3a** and **3b** gave ees up to 76% and 86%, respectively, with the same (*R*)-configuration being obtained. The model showed effectively that for both ligands **3a** and **3b** (Fig. 2), the chelate is mainly stabilized by the relative disposition of the carbohydrate moiety and the ethyl group (R_2 in Fig. 2). According to the obtained results, the most important factor is the asymmetric induction due to the carbohydrate moiety, which directed the attack of the nucleophile. The position of the ethyl group in the space [(*R*)- or (*S*)-configuration] modified slightly the steric environment, and consequently the ee.

3. Conclusion

In conclusion, new chiral phosphine-amides derived from D-glucosamine were easily prepared, and used in the palladium-catalyzed asymmetric allylic alkylation with ees

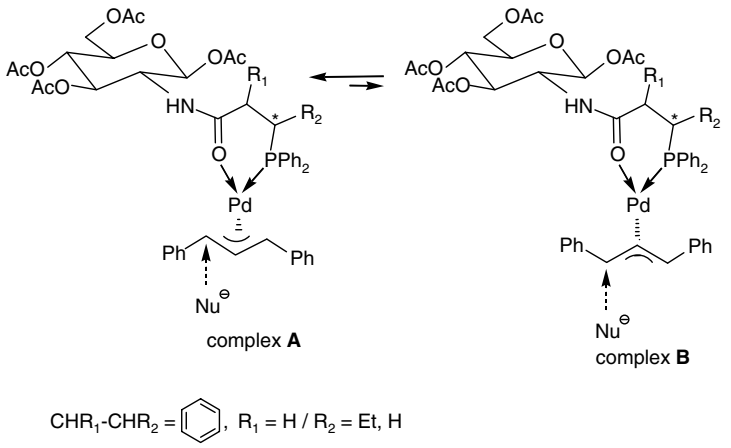


Figure 2. A plausible scheme in the allylic alkylation using six-member chelates **1** and **3a**–**c**.

up to 86%. The influence of the D-glucosamine moiety seems to be the most important factor in this asymmetric allylic alkylation. The synthesis of new carbohydrate-based ligands is currently in progress, in order to have a deeper insight on the factors influencing this asymmetric-catalyzed reaction.

4. Experimental

4.1. General

Solvents were purified by standard methods and dried if necessary. All commercially available reagents were used as received. All reactions were monitored by TLC (TLC plates GF_{254} Merck). Air and moisture sensitive reactions were performed under the usual inert atmosphere techniques. Reactions involving organometallic catalysis were carried out in a Schlenk tube under an inert atmosphere. Column chromatography was performed on silica gel 60 (230–240 mesh, Merck). Optical rotations were recorded using a Perkin–Elmer 241 polarimeter. NMR spectra were recorded with a Bruker AMX 300 spectrometer and referenced as follows: ^{1}H (300 MHz), internal $SiMe_4$ at δ 0.00 ppm and ^{31}P (121 MHz), external 85% H_3PO_4 at δ 0.00 ppm. Conversion was determined by GC using a Quadrex OV1 column (30 m × 0.25 mm), and enantiomeric excess was determined by HPLC with a Chiralpak^{AD} column (25 cm × 4.6 mm) using a ratio of hexane/*i*-propanol as eluent.

4.2. Synthesis of 1,3,4,6-tetra-*O*-acetyl-2-deoxy-2-({[2-diphenylphosphino)phenyl]acetyl}amino-β-D-glucopyranose, 2

2-Amino-1,3,4,6-tetra-*O*-acetyl-2-deoxy-β-D-glucopyranose (500 mg, 1.44 mmol), carboxylic acid **4** (484 mg, 1.51 mmol), EDC (326 mg, 1.70 mmol), and HOBT (230 mg, 1.70 mmol) were dissolved in a mixture of CH_2Cl_2 (10 mL) and THF (20 mL) in a Schlenk tube. After being stirred for 48 h at rt, the solvents were evaporated. The residue was purified by chromatography on silica gel to give phosphine-amide **2** (yield 62%). $R_f = 0.60$ (petroleum ether/ethyl acetate 1/2); $[\alpha]_D^{25} = +25.3$ (*c* 0.6, $CHCl_3$); ^{1}H NMR ($CDCl_3$): δ 7.63–7.52 (m, 1H, H_{arom}), 7.45–7.26 (m, 11H, H_{arom}), 7.24–7.16 (m, 1H, H_{arom}), 6.92–6.85 (m, 1H, H_{arom}), 5.68 (br d $J = 8.7$ Hz, 1H, NH), 5.50 (d, $J = 8.9$ Hz, 1H, H-1), 5.07 (dd, $J = 9.4$, 9.2 Hz, 1H, H-3), 5.03 (dd, $J = 9.6$, 9.4 Hz, 1H, H-4), 4.28 (dd, $J = 12.4$, 4.5 Hz, 1H, H-6), 4.20 (dd, $J = 8.9$, 9.2 Hz, 1H, H-2), 4.10 (dd, $J = 12.4$, 2.2 Hz, 1H, H-6′), 3.75 (ddd, $J = 9.6$, 4.5, 2.2 Hz, 1H, H-5), 3.70 (br s, 2H, CH_2), 2.10 (s, 3H, CH_3), 2.06 (s, 3H, CH_3), 1.94 (s, 3H, CH_3), 1.86 (s, 3H, CH_3); ^{31}P NMR ($CDCl_3$): δ −12.9; HRMS $[M+H]^+$ $C_{34}H_{36}NO_{10}P$: calcd 650.2155, found 650.2152.

4.3. General procedure for the synthesis of 3a–c

2-Amino-1,3,4,6-tetra-*O*-acetyl-2-deoxy-β-D-glucopyranose (500 mg, 1.44 mmol), carboxylic acid **6a**–**c** (1.51 mmol), EDC (326 mg, 1.70 mmol), and HOBT (230 mg, 1.70 mmol) were dissolved in a mixture of CH_2Cl_2 (10 mL) and THF (20 mL) in a Schlenk tube. After being stirred for 48 h at rt, the solvents were evaporated. The residue was purified by chromatography on silica gel to give borane–phosphine-amide complexes **5a**–**c**. Complexes **5a**–**c** and DABCO (4.53 mmol) were dissolved in toluene (10 mL) in a Schlenk tube under argon. After being stirred for 48 h at rt, the solvent was evaporated. The crude powder was purified by flash-chromatography on silica gel using petroleum ether/ethyl acetate (10/15) as the eluent to give ligand **3a**–**c**.

4.3.1. 1,3,4,6-Tetra-*O*-acetyl-2-deoxy-2-{[(3*R*)-3-(diphenylphosphino)pentanoyl]amino}-β-D-glucopyranose, 3a. $R_f = 0.60$ (petroleum ether/ethyl acetate 10/15); $[\alpha]_D^{25} = +8.5$ (*c* 0.2, $CHCl_3$); ^{1}H NMR ($CDCl_3$): δ 7.92–7.82 (m, 2H, H_{arom}), 7.80–7.69 (m, 2H, H_{arom}), 7.52–7.32 (m, 6H, H_{arom}), 5.80 (br d $J = 8.7$ Hz, 1H, NH), 5.69 (d, $J = 8.9$ Hz, 1H, H-1), 5.30 (dd, $J = 9.4$, 9.2 Hz, 1H, H-3), 5.10 (dd, $J = 9.8$, 9.4 Hz, 1H, H-4), 4.29 (dd, $J = 12.4$, 4.5 Hz, 1H, H-6), 4.26 (dd, $J = 9.2$, 8.9 Hz, 1H, H-2), 4.11 (dd, $J = 12.4$, 1.9 Hz, 1H, H-6′), 3.88 (ddd, $J = 9.8$, 4.5, 1.9 Hz, 1H, H-5), 3.39–3.24 (m, 1H, CH), 2.40–2.20 (m, 2H, CH_2CO), 2.09 (s, 3H,

$COCH_3$), 2.04 (s, 3H, $COCH_3$), 2.00 (s, 3H, $COCH_3$), 1.93 (s, 3H, $COCH_3$), 1.58–1.39 (m, 2H, CH_2CH_3), 0.78 (t, $J = 7.2$ Hz, 3H, CH_2CH_3); ^{31}P NMR ($CDCl_3$): δ −4.5; HRMS $[M+H]^+$ $C_{31}H_{39}NO_{10}P$: calcd 616.2312, found 616.2317.

4.3.2. 1,3,4,6-Tetra-*O*-acetyl-2-deoxy-2-{[(3*S*)-3-(diphenylphosphino)pentanoyl]amino}-β-D-glucopyranose, 3b. $R_f = 0.60$ (petroleum ether/ethyl acetate 10/15); $[\alpha]_D^{25} = +17.2$ (*c* 0.25, $CHCl_3$); ^1H NMR ($CDCl_3$): δ 7.92–7.83 (m, 2H, H_{arom}), 7.82–7.73 (m, 2H, H_{arom}), 7.52–7.32 (m, 6H, H_{arom}), 5.73 (d, $J = 8.8$ Hz, 1H, H-1), 5.68 (br d $J = 8.7$ Hz, 1H, NH), 5.16 (dd, $J = 9.4$, 9.2 Hz, 1H, H-3), 5.11 (dd, $J = 9.4$, 9.4 Hz, 1H, H-4), 4.30 (dd, $J = 12.5$, 4.5 Hz, 1H, H-6), 4.25 (dd, $J = 9.4$, 8.8 Hz, 1H, H-2), 4.13 (dd, $J = 12.5$, 2.2 Hz, 1H, H-6′), 3.83 (ddd, $J = 9.4$, 4.5, 2.2Hz, 1H, H-5), 3.39–3.24 (m, 1H, CH), 2.40–2.20 (m, 2H, CH_2CO), 2.10 (s, 3H, $COCH_3$), 2.06 (s, 3H, $COCH_3$), 2.04 (s, 3H, $COCH_3$), 1.83 (s, 3H, $COCH_3$), 1.60–1.41 (m, 2H, CH_2CH_3), 0.80 (t, $J = 7.2$ Hz, 3H, CH_2CH_3); ^{31}P NMR ($CDCl_3$): δ −4.5; HRMS $[M+H]^+$ $C_{31}H_{39}NO_{10}P$: calcd 616.2312, found 616.2316.

4.3.3. 1,3,4,6-Tetra-*O*-acetyl-2-deoxy-2-({[2-(diphenylphosphino)phenyl]acetyl}amino)-β-D-glucopyranose, 3c. $R_f = 0.55$ (petroleum ether/ethyl acetate 10/15); $[\alpha]_D^{25} = +14.5$ (*c* 0.2, $CHCl_3$); ^1H NMR ($CDCl_3$): δ 7.80–7.60 (m, 4H, H_{arom}), 7.52–7.38 (m, 6H, H_{arom}), 6.03 (br d $J = 8.1$ Hz, 1H, NH), 5.75 (d, $J = 8.7$ Hz, 1H, H-1), 5.24 (dd, $J = 9.8$, 9.4 Hz, 1H, H-3), 5.10 (dd, $J = 9.8$, 9.4 Hz, 1H, H-4), 4.28 (dd, $J = 12.4$, 4.2 Hz, 1H, H-6), 4.21 (dd, $J = 9.4$, 8.7 Hz, 1H, H-2), 4.12 (dd, $J = 12.4$, 1.9 Hz, 1H, H-6′), 3.86 (ddd, $J = 9.8$, 4.2, 1.9 Hz, 1H, H-5), 2.58–2.49 (m, 2H, CH_2), 2.36–2.30 (m, 2H, CH_2CO), 2.08 (s, 3H, $COCH_3$), 2.07 (s, 3H, $COCH_3$), 2.04 (s, 3H, $COCH_3$), 1.98 (s, 3H, $COCH_3$); ^{31}P NMR ($CDCl_3$): δ −15.2; HRMS $[M+Na]^+$ $C_{29}H_{34}NO_{10}NaP$: calcd 610.1818, found 610.1815.

4.4. General procedure for the allylic alkylation

In a Schlenk tube, $[Pd(\eta^3\text{-}C_3H_5)Cl]_2$ (8.8 mg, 24 μmol) and the ligand (48 μmol) were dissolved in THF (1 mL). After being stirred for 1 h at rt, a solution of racemic 1,3-diphenyl-2-propenyl acetate (302 mg, 1.2 mmol) in THF (1 mL) was added. After 30 min, this solution was transferred to a Schlenk tube containing dimethyl malonate (475 mg, 3.6 mmol), BSA (732 mg, 3.6 mmol), and LiOAc (2.5 mg, 24 μmol) in THF (2 mL). The reaction mixture was stirred at the desired temperature for 24 h. The conversion was determined by GC analysis. The mixture was then diluted with diethyl ether (15 mL) and water (5 mL). The organic phase was washed with brine and dried over $MgSO_4$. Evaporation of the solvent gave a residue, which was purified by chromatography (petroleum ether/ethyl acetate 10/1). The enantiomeric excesses were determined by HPLC analysis.

Acknowledgements

A.K. thanks the CNRS, and A.P. and K.G. thank the MENSR for fellowships.

References

1. Trost, B. M.; Strege, P. E. *J. Am. Chem. Soc.* **1977**, *99*, 1649–1651.
2. Trost, B. M.; Van Vranken, D. L.; Bingel, C. *J. Am. Chem. Soc.* **1992**, *114*, 9327–9343.
3. (a) Von Matt, P.; Pfaltz, A. *Angew. Chem., Int. Ed. Engl.* **1993**, *32*, 566–568; (b) Williams, J. M. J. *Synlett* **1996**, 705–710; (c) Helmchen, G. *J. Organomet. Chem.* **1999**, *576*, 203–214.
4. (a) Dieguez, M.; Pamies, O.; Claver, C. *Chem. Rev.* **2004**, *104*, 3189–3215; (b) Dieguez, M.; Pamies, O.; Ruiz, A.; Diaz, Y.; Castillon, S.; Claver, C. *Coord. Chem. Rev.* **2004**, *248*, 2165–2192.
5. Nazarov, A. A.; Hartinger, C. G.; Arion, V. B.; Giester, G.; Keppler, B. K. *Tetrahedron* **2002**, *58*, 8489–8492.
6. Khiar, N.; Araujo, C. S.; Alvarez, E.; Fernandez, I. *Tetrahedron Lett.* **2003**, *44*, 3401–3404.
7. (a) Hong-Wick, K.; Pregosin, P. S.; Trabesinger, G. *Organometallics* **1998**, *17*, 3254–3264; (b) Barbaro, P.; Currao, A.; Hermann, J.; Nesper, R.; Pregosin, P. S.; Salzmann, R. *Organometallics* **1996**, *15*, 1879–1888.
8. Glaser, B.; Kunz, H. *Synlett* **1998**, 53–55.
9. (a) Yonehara, K.; Hashizume, T.; Mori, K.; Ohe, K.; Uemura, S. *Chem. Commun.* **1999**, 415–416; (b) Yonehara, K.; Hashizume, T.; Mori, K.; Ohe, K.; Uemura, S. *J. Org. Chem.* **1999**, *64*, 9374–9380; (c) Hashizume, T.; Yonehara, K.; Ohe, K.; Uemura, S. *J. Org. Chem.* **2000**, *65*, 5197–5201.
10. Borriello, C.; Cucciolito, M. E.; Panunzi, A.; Ruffo, F. *Inorg. Chim. Acta* **2003**, *353*, 238–244.
11. Brunner, H.; Schönherr, M.; Zabel, M. *Tetrahedron: Asymmetry* **2003**, *14*, 1115–1122.
12. Tollabi, M.; Framery, E.; Goux-Henry, C.; Sinou, D. *Tetrahedron: Asymmetry* **2003**, *14*, 3329–3333.
13. Hou, X.-L.; Dong, D. X.; Yuan, K. *Tetrahedron: Asymmetry* **2004**, *15*, 2189–2191.
14. Léautey, M.; Jubault, P.; Pannecoucke, X.; Quirion, J.-C. *Eur. J. Org. Chem.* **2003**, 3761–3768.
15. (a) Butts, C. P.; Crosby, J.; Lloyds-Jones, G. C.; Stephen, S. C. *Chem. Commun.* **1999**, 1707–1708; (b) Fairlamb, I. J. S.; Lloyd-Jones, G. C. *Chem. Commun.* **1999**, 2447–2448; (c) Kim, Y. K.; Lee, S. J.; Ahn, K. H. *J. Org. Chem.* **2000**, *65*, 7807–7813.