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Study on the electronic effects on stereoconservativity of Suzuki coupling in chiral groove of binaphthyl

Michal Juríček, Henrich Brath, Peter Kasák [1], Martin Putala *

Department of Organic Chemistry, Faculty of Natural Sciences, Comenius University in Bratislava, Mlynská dolina, 842 15 Bratislava, Slovak Republic

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Abstract

Suzuki diarylation of enantiopure 2,2′-diiodo-1,1′-binaphthyl catalyzed by triphenylphosphane palladium complex is accompanied by almost complete racemization of binaphthyl moiety (7% e.e.). Based on formerly proposed mechanism, secondary oxidative addition of Pd(II) to Pd(IV)-complex, competitive to transmetallation, is expected to be responsible for racemization. In accordance with it, racemization pathway can be suppressed in the favour of stereoconservative route by electronic factors, which control the rate of oxidative addition. Among the electronic factors, decreasing donating ability of the tested phosphane ligands resulted in increase of e.e. of the diarylated product up to 65%, using triindol-1-ylphosphane. However, this factor slows down also the rate of the primary oxidative addition that lowers the yield of the diarylated product. Further decrease in donating ability of the ligand makes palladium complex almost inactive in this cross-coupling reaction. Effect of the leaving group of binaphthyl 2,2′-dielectrophile (as a matter of the reactivity of C–X bond towards oxidative addition) was found to be even more dramatic: almost stereoconservative route in case of 2,2′-dibromo-1,1′-binaphthyl (95% e.e.), but no reaction in case of corresponding ditriflate.

Keywords: Binaphthyl; Mechanism; P ligands; Palladium complexes; Stereoconservative; Suzuki cross-coupling

1. Introduction

Configurationally stable, enantiopure 2,2′-substituted 1,1′-binaphthyl derivatives [1,2] represent important group of chiral compounds with applications reported either in asymmetric catalysis as ligands [3], in supramolecular chemistry as building blocks [4] or in materials science as compounds with interesting optical or/and electronic properties [5]. Preparation of binaphthyl derivatives bearing carbon groups at positions 2 and 2′ requires use of stereoconservative Negishi [6,7] or Kumada [6] cross-couplings from enantiopure ditriflate **1** (just in the case of methylation [6]) or more reactive diiodide **2** [6,7]. We showed that Suzuki diarylation of the diiodide **2** proceeds with almost complete racemization of binaphthyl moiety [8,9]. However, stereoconservativity of Suzuki diarylation at positions 2 and 2′ can be achieved by umpolung, using 1,1′-binaphthyl-2,2′-diboronic acid as binaphthyl precursor [9].

As an explanation of racemization during Suzuki arylation of diiodide **2** we proposed following mechanism based on configurationally unstable pallada(IV) cycle **3** (Scheme 1) [8]:

(1) The first step – an oxidative addition of **2** to the palladium complex (insertion of palladium to C–I bond of a **2** in position 2) gives palladium(II) complex **4**, analogous to usual intermediates in cross-coupling reaction from aryl halides [10].
(2) If the organometallic reagent (organozinc or organomagnesium halide) is sufficiently reactive, transmetallation can take place and the first C–C bond is formed. A C–C bond at position 2′ is formed analogously. In such a mechanism there is no reason for

* Corresponding author. Tel.: +421 2 60696 323; fax: +421 2 60696 690.
E-mail address: putala@fns.uniba.sk (M. Putala).
[1] Present address: Polymer Institute of the Slovak Academy of Sciences, Dúbravská cesta 9, 842 36 Bratislava, Slovak Republic.

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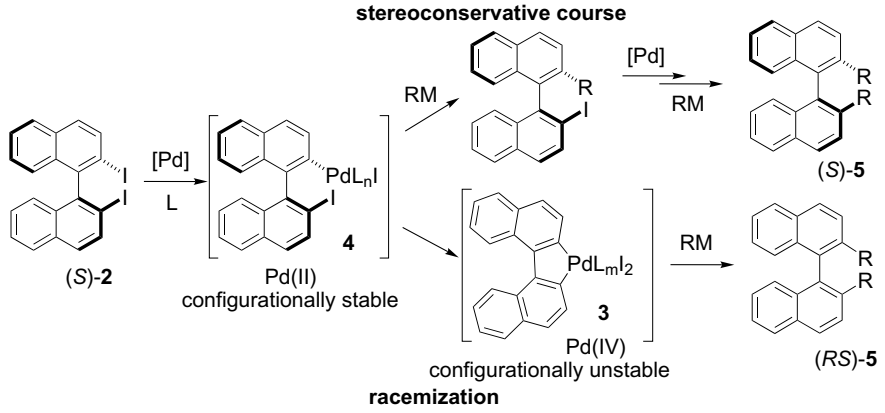


Scheme 1. Proposed pathways for cross-coupling reaction of the diiodide **2**.

racemization, and product **5** is obtained enantiopure – with complete conservation of stereogenic information from substrate.

(3) However, in the case of less reactive organometallics (boronate or stannane), transmetallation is expected to be slower than insertion of palladium in **4** to C–I bond at the position 2′ to form a palladacyclic complex **3** containing palladium(IV). It is known that 1,1′-binapthalene derivatives having positions 2 and 2′ bridged with one-atom linker have a low racemization barrier (between 45 and 65 kJ mol^{-1} [11]). These derivatives lack non-bonding interactions at positions 2 and 2′ (ring flipping of fused unsaturated five-membered ring requires only low activation energy) and also non-bonding interactions at positions 8 and 8′ are decreased (the outer benzene rings of naphthalene units are pulled apart). Thus, the binaphthyl moiety is easily racemized in this step. The palladacyclic complex **3** is not necessary an intermediate, this can be also considered as an activated complex, through which either reversible migration of palladium from position 2 to 2′ accompanied by racemization can occur or the reactivity of binaphthyl-palladium species towards transmetallation can be increased. Then, after transmetallation and formation of C–C bonds at the both 2 and 2′ positions, racemic product **5** is formed.

Our mechanistic proposal was supported by the ^{31}P NMR spectra of intermediate Pd complexes prepared by the reaction of equimolar amount of the diiodide **2** and palladium(0) complex [8] (two signals from C_1-symmetric complex **4**, single signal from C_2-symmetric complex **3**). Although Pd(IV)-complexes are not accepted as general intermediates in cross-coupling reactions, we presume that the diiodide **2** is specific substrate offering to proximate C–I bonds.

Pd(IV)-complexes become more often to be considered as likely intermediates in some Heck and cross-coupling reactions [12]. For instance, 1,4-migration of Pd in Pd(II)-complex from the position 2 to the position 2′ on 1,1′-biphenyl derivatives via Pd(IV)-intermediate is expected to be more probable than to take place via concerted mechanism pathway [13]. Pt(IV)-complexes containing a biphenyl moiety similar to that in complex **3**, were characterized [14]. Also, structurally relative Pd,Pd-dibromopallada(IV)cyclopentadiene species stabilized with an *N*,*N*-ligand were observed as intermediates [15].

Here we report results of our effort to control the racemization of binaphthyl moiety during Suzuki reaction of binaphthyl 2,2′-dielectrophiles by control of the second oxidative addition either by use of less electron rich phosphane ligands or by use of less reactive binaphthyl 2,2′-dielectrophiles, which undergo oxidative addition less readily.

2. Experimental

2.1. Methods

Flash column chromatography was performed on Merck Silica Gel (60H). Merck Silica Gel F254 plates were used for thin layer chromatography and visualization was effected with UV-light (254 nm). Melting points were measured on a Electrothermal-IA9200 instrument and the values are uncorrected. Specific optical rotations were measured on a Perkin–Elmer 241 polarimeter and are given in deg cm^2 g^{-1} dm^{-1}. HPLC analysis was done on Chiralcel Daicel OD-H column using a UV–vis detector LCD 5000. UV–vis spectra were measured on a Hewlett–Packard Diode Array 8245 spectrophotometer. IR data were recorded on SPECORD M 80 spectrophotometer. ^1H, ^{13}C and ^{31}P NMR were recorded on VARIAN GEMINI 300 instrument at 298 K. Chemical shifts are reported in ppm downfield to internal standard TMS (0.00 ppm) and the solvent was used as a reference. Working frequency was 300 MHz for ^1H NMR, 75.5 MHz for ^{13}C NMR and 121.5 MHz for ^{31}P NMR. Coupling constants are given in Hz. GC–MS spectra (70 eV, 150 μA, EI) were recorded on Voyager GC/MS Finnigan instrument. Elemental analyses were determined with Erba Science 1106 instrument.

2.2. Materials

All chemicals were used as purchased if not stated otherwise. All solvents for coupling reactions were degassed with thaw freeze pump in three cycles and these reactions were performed under argon atmosphere using Schlenk technique. Ditriflate (*RS*)-, and (*S*)-**1** [16], diiodide (*RS*)-, and (*S*)-**2** [7], dibromide (*RS*)- and (*S*)-**6** [17], triindol-1-ylphosphane [18], and tris[3-(trifluoromethyl)phenyl]phosphane [19] were prepared according to the literature procedures.

2.3. Synthesis

2.3.1. General procedure for investigation of electronic ligand effect on the Suzuki coupling from (*S*)-2 (Table 1)

A Schlenk flask was charged with (*S*)-**2** (99% e.e., 100 mg, 0.2 mmol), *p*-tolylboronic acid (**8a**, 109 mg, 0.8 mmol, 2 equiv. to C_{AR}–I), $Ba(OH)_2 \cdot 8H_2O$ (760 mg, 2.4 mmol), $Pd(dba)_2$ (12 mg, 0.02 mmol, 5 mol% to C_{AR}–I) and ligand (0.08 mmol, 4 equiv. to Pd). The flask was evacuated and filled with argon in three cycles. 3 mL water and 3 mL THF were successively injected into the flask and the mixture was heated to reflux being stirred for 36 h. After cooling, the reaction mixture was poured into 5% aq HCl and extracted thrice with CH_2Cl_2 (10 mL portions). Combined organic fractions were washed twice with water and brine and dried over Na_2SO_4. After filtration and evaporation the solvent, the residue was chromatographed on silica gel (see Table 1).

2.3.2. General procedure for investigation of leaving group effect on the Suzuki coupling from (*S*)-1, 2, and 6 (Table 2)

A Schlenk flask was charged with binaphthyl dielectrophile (*S*)-**1**, **2** or **6** (0.2 mmol), boronic acid **7a** or **7b** (0.8 mmol, 2 equiv. to C_{AR}–X), $Ba(OH)_2 \cdot 8H_2O$ (760 mg, 2.4 mmol) and $Pd(PPh_3)_4$ (23 mg, 0.02 mmol, 5 mol% to C_{AR}–X). The flask was evacuated and filled with argon in three cycles. 3 mL water and 3 mL THF were successively injected into the flask and the mixture was heated to reflux being stirred for 36 h. After cooling, the reaction mixture was poured into 5% aq HCl and extracted thrice with CH_2Cl_2 (10 mL portions). Combined organic fractions were washed twice with water and brine and dried over Na_2SO_4. After filtration and evaporation the solvent, the residue was chromatographed on silica gel (see Table 2).

(S)-2,2′-bis(4-methylphenyl)-1,1′-binaphthyl (**5a**). White solid. M.p. 179–183 °C. $[\alpha]_D^{22} = -169.8$ ($c = 1.01$, $CHCl_3$). $R_f = 0.45$ (hexanes/ethyl acetate = 10/1). Spectral data were in agreement with the literature values [9]. HPLC: Chiralcel Daicel OD-H column, eluent: *n*-hexane with 2% DME, temperature: 21 °C, flow: 0.75 ml/min, pressure: 26 bar, detector (UV): $\lambda = 280$ nm; k': 2.10 (*S*)-**5a**, 2.56 (*R*)-**5a**.

(S)-2,2′-diphenyl-1,1′-binaphthyl (**5b**). White solid. M.p. 125–128 °C. $[\alpha]_D^{22} = -128.5$ ($c = 1$, $CHCl_3$). $R_f = 0.42$ (hexanes/ethyl acetate = 10/1). Spectral data were in agreement with the literature values [9]. HPLC: Chiralcel Daicel OD-H column, eluent: *n*-heptane with 1% DME, temperature: 16 °C, flow: 1.0 ml/min, pressure: 31 bar, detector (UV): $\lambda = 280$ nm; k': 6.52 (*S*)-**5b**, 7.35 (*R*)-**5b**.

(R)-2-(4-methylphenyl)-1,1′-binaphthyl (**8a**). White solid. M.p. 110–111 °C. $[\alpha]_D^{22} = -133$ ($c = 1$, $CHCl_3$). $R_f = 0.52$ (hexanes/chloroform = 4/1). Spectral data were in agreement with literature values [20]. HPLC: Chiralcel Daicel OD-H column, eluent: *n*-hexane with 2% DME, temperature: 21 °C, flow: 0.75 ml/min, pressure: 26 bar, detector (UV): $\lambda = 254$ nm; k': 2.29 (*R*)-**8a**, 2.92 (*S*)-**8a**.

(R)-2-phenyl-1,1′-binaphthyl (**8b**). White solid. M.p. 131–133 °C (lit. [19] 133 °C). $R_f = 0.52$ (hexanes/chloroform = 4/1). Spectral data were in agreement with the literature values [20]. HPLC: Chiralcel Daicel OD-H column, eluent: *n*-hexane with 2% DME, temperature: 20 °C, flow: 0.75 ml/min, pressure: 26 bar, detector (UV): $\lambda = 254$ nm; k': 2.46 (*R*)-**8b**, 3.22 (*S*)-**8b**.

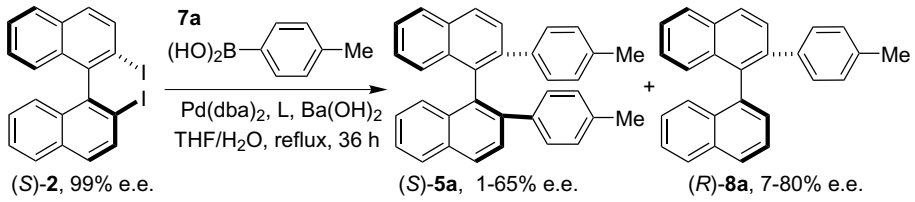
3. Results and discussion

3.1. Electronic ligand effect

In accordance with proposed mechanism [8], we attempted to verify hypothesis that phosphane ligands with reduced electron density should decrease the rate of oxidative addition, especially the second one, from Pd(II) to Pd(IV) intermediate (although the oxidation state on palladium is formal, since Pd–I and Pd–C bonds are only slightly polar based on the electronegativity difference) and therefore they should favour stereoconservative reaction pathway via stepwise arylation (proceeding just through Pd(II) intermediate **4** and then through analogous 2-arylated one at position 2′) over racemization route via double oxidative addition including configurationally unstable Pd(IV) intermediate **3**. Although the electronic effect of phosphane ligand affects also the rate of consecutive reductive elimination (proceeding via both Pd(II) and Pd(IV) intermediates), this reaction step is supposed not to be the rate determining one. Quantitative analysis of ligand effect (QALE) [21] uses two basic parameters to express electronic properties of phosphane ligands: χ_d for σ-donating properties (lower value means stronger donating properties) and π_p for π-acidity (value increases with increasing π back-bonding character of the ligand).

We examined electronic effect of several phosphane ligands. Choice of the ligands was made based on their commercial or synthetic availability, stability at reaction conditions (heating in alkaline medium), but mostly in order to have a set of ligands with different electronic properties. Reactions were performed with *para*-tolylboronic acid (**7a**) as model substrate at optimized conditions found for this particular cross-coupling reaction [9]. The results are shown in Table 1. In all cases, ditolylated product **5a** was found to be the main product. Also, hydro-dehalogenated monotolylated product **8a** was isolated in low yields.

Table 1
Electronic ligand effect on Suzuki arylation of diiodide **2**[a]



(*S*)-**2**, 99% e.e. (*S*)-**5a**, 1-65% e.e. (*R*)-**8a**, 7-80% e.e.

Entry	Ligand	χ_d [21]	π_p [21]	**5a**		**8a**	
				Yield[b] (%)	e.e. (%)	Yield[b] (%)	e.e. (%)
1	$FcPCy_2$	(>1.4[c])	(0[c])	68	1	15	55
2	Ph_3P	13.3	0	52	7	10	80
3	$(3\text{-}CF_3C_6H_4)_3P$	(<20.5[d])	(0[d])	29	9	20	56
4	$(MeO)_3P$	17.9	2.8	34	49	<5	n.d.
5	$(Indolyl)_3P$	(<31.9[e])	(<1.9[e])	24	65	19	15
6	$(Pyrrolyl)_3P$	31.9	1.9	11	4	15	77
7	$(C_6F_5)_3P$	(>31.1[f])	–	7	3	3	28
8	dba	–	–	7	6	36	7

[a] 0.2 mmol (*S*)-**2**, 0.8 mmol **7a**, 2.4 mmol $Ba(OH)_2 \cdot 8H_2O$, 0.02 mmol $Pd(dba)_2$, 0.08 mmol L, 3 mL THF, 3 mL H_2O, reflux for 36 h; acidic work-up followed by flash chromatography.
[b] Isolated yield.
[c] Values for Cy_3P.
[d] Values for $(4\text{-}CF_3C_6H_4)_3P$.
[e] Values for $(pyrrolidino)_3P$.
[f] Value for $R[OP(C_6F_5)_2]_2$, R = *trans*-cyclopentan-1,2-diyl [23].

Enantiomeric excess of the both products was determined by HPLC using Chiralcel Daicel OD-H column.

Indeed, we observed increase in the e.e. of ditolylated product **5a** with decreasing σ-donating properties of the ligand (increasing χ_d value) up to certain extent; from 1% e.e. for dicyclohexyl(ferrocenyl)phosphane (Table 1, entry 1) up to 65% e.e. for triindol-1-ylphosphane (Table 1, entry 5). However, higher e.e. was observed with ligands exhibiting π-acidity (non-zero π_p value). The yield of the product **5a** decreases with increasing e.e. since the electronic effect of the ligand slows down not only the secondary oxidative addition from Pd(II) to Pd(IV), but also the primary oxidative addition to form Pd(II) intermediate **4**. This leads to decreased synthetic efficiency of the catalytic system. When using ligands with weaker σ-donating properties than triindol-1-ylphosphane (Table 1, entries 6 and 7) we observed significant drop in both the yield and e.e. of the product **5a**. We expect these ligands (tripyrrol-1-ylphosphane and perfluorotriphenylphosphane) to be already so electron poor that they most probably deactivate palladium complex towards oxidative addition, and both yield and e.e. of **5a** are comparable with ligandless reaction (performed just with palladium source $Pd(dba)_2$; Table 1, entry 8).

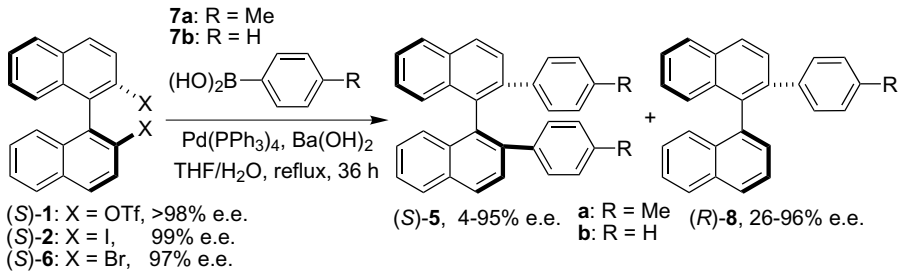
The formation of monotolylated product **8a** is also partially stereoconservative (conserving spatial arrangement of the binaphthyl moiety) although the stereochemical descriptor has changed [22] (applying CIP system rules). Results observed for monotolylated product **8a** (yield, e.e.) do not follow any clear dependence on electronic properties of the ligand used. Most probably, steric effects should play an important role, since the highest e.e. for **8a** were found using less hindered ligands: triphenylphosphane (80% e.e., Table 1, entry 2) and tripyrrol-1-ylphosphane (77% e.e., Table 1, entry 6).

3.2. Leaving group effect

The reactivity of aryl electrophiles in cross-coupling reactions (i.e. rate of oxidative addition) is known to depend also on leaving group and can be considered as partially of electronic nature. In the case of palladium complexes catalyzed cross-coupling reactions, reactivity of relative substrates decreases in general in the following order: iodide > bromide > triflate. We examined whether also this factor can affect reaction pathway and hence stereoconservativity of the arylation of binaphthyl 2,2′-dielectrophiles (Table 2), using standard reaction conditions and tetrakis(triphenylphospane)palladium as a catalyst.

We found that this factor has significant effect on the yield of diarylated product **5**: 6% drop in the yield from diiodide **2** to dibromide **6** (Table 2, entries 1 versus 3 and 2 versus 4) and no reaction in the case of ditriflate **1** (Table 2, entries 5 and 6). It has to be noted that binaphthyl 2,2′-dielectrophiles are highly sterically hindered substrates since they possess large group (2-substituted 1-naphthyl) at the adjacent position (position 1) to the place where the first oxidative addition takes place (position 2). Stereoconservativity of the coupling, i.e. the second oxidative addition, is even dramatically affected by leaving group: almost complete racemization in the case of diiodide **2** (Table 2, entries 1 and 2) versus almost stereoconservative course in the case of dibromide **6** (Table 2, entries 3 and 4).

Table 2
Leaving group effect on Suzuki arylation of diiodide **2**[a]



Entry	Binaphthyl substrate (X)	Boronic acid (R)	**5**		**8**	
			Yield[b] (%)	e.e. (%)	Yield[b] (%)	e.e. (%)
1	**2**(I)	**7b**(H)	44	4	19	34
2	**2**(I)	**7a**(Me)	52	7	10	80
3	**7**(Br)	**7b**(H)	38	84	11	26
4	**7**(Br)	**7a**(Me)	46	95	32	96
5	**1**(OTf)	**7b**(H)	0	–	<3	n.d.
6	**1**(OTf)	**7a**(Me)	0	–	<3	n.d.

[a] 0.2 mmol (*S*)-**1**, **2**, or **6**, 0.8 mmol **7**, 2.4 mmol $Ba(OH)_2 \cdot 8H_2O$, 0.02 mmol $Pd(PPh_3)_4$, 3 mL THF, 3 mL H_2O, reflux for 36 h; acidic work-up followed by flash chromatography.
[b] Isolated yield.

Found e.e. of diphenylated product **5b** (Table 2, entries 1 and 3) are slightly lower than those of ditolylated **5a** (entries 2 and 4). This is most probably due to lower efficiency of the less electron rich phenylboronic acid (**7b**) in transmetallation, which allows competitive oxidative addition from Pd(II) to Pd(IV) to proceed in higher extent.

Results concerning stereoconservativity in formation of monoarylated product **8** do not exhibit strong dependence on leaving group. The monotolylated product **8a** was obtained significantly more enantioenriched compared to monophenylated **8b** (Table 2, entries 1 versus 2 and 3 versus 4) (most probably for the abovementioned lower reactivity of phenylboronic acid, **7b**).

4. Conclusions

In our study we showed that, in accordance with formerly proposed mechanism, racemization pathway of the palladium complex catalyzed Suzuki diarylation of enantiopure diiodide **2** can be suppressed in the favour of stereoconservative route by electronic factors, which control the rate of oxidative addition. Investigated factors were: decreasing electron donating properties of the phosphane ligand (resulting in increase of e.e. of the product **5a** from 1% up to 65%) and leaving group of binaphthyl 2,2′-dielectrophile (from 7% e.e. up to 95% e.e.). However, the yield of the diarylated product **5** decreases in this order. Slowing down the oxidative addition by using either very electron poor ligands or ditriflate **1** as a substrate caused failure of the reaction. Providing direct evidence of proposed mechanism and exploitation of racemization for the development of enantioselective diarylation of binaphthyl are subject of intensive study in our laboratory.

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