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Pd-catalysed asymmetric Suzuki–Miyaura reactions using chiral mono- and bidentate phosphorus ligands

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ABSTRACT

A series of monodentate and bidentate chiral phosphorus based ligands including diphosphites and diphosphonites derived from carbohydrates were tested in the asymmetric Suzuki coupling of aryl halides with 2-substituted 1-naphthylboronic acids. Good activities and selectivities were achieved, although modest ee's were obtained (up to 37%). The X-ray structure of $PtCl_2$(**12**) bearing the C_2-symmetry diphosphite ligand **12** derived from carbohydrate, is also reported.

1. Introduction

The Suzuki–Miyaura reaction is a powerful process for the synthesis of biaryl compounds [1–4], with many inherent advantages over other methods such as the tolerance to a broad range of functional groups, non-toxic by-products, and the stability of the organoboronic partners that are inert to air and water [5,6]. Axially chiral biaryls are important structural motifs in biologically active molecules [7] and ligands for catalysis [8–10]. Two examples are shown in Fig. 1: the vancomycin, which is a clinically used glycopeptide antibiotic [11] and the steganacin, which is a cytotoxic tubulin-binding dibenzocyclooctadiene lignin [12]. The chirality of these compounds arises from the restricted rotation around the aryl–aryl bond [13]. Binaphthalenes are among the most representative compounds exhibiting axial chirality and over the last years, the synthesis of optically active 1,1-binaphthalenes through asymmetric Suzuki–Miyaura coupling has received much attention [14–23]. The first reports on the synthesis of axially chiral biaryls by enantioselective Suzuki–Miyaura reaction were published independently in 2000 by Buchwald [14] and Cammidge [15]. Later, Johannsen and Jensen reported the Pd-catalysed asymmetric coupling reaction using Pd catalysts with the dialkyl arylferrocenylphosphines ligands with moderate ee's (up to 54%) [16]. Mikami et al. reported that excellent conversion and enantioselectivity could be achieved in short reaction times using cationic Pd/BINAP complexes [17]. Espinet and co-workers also improved the reaction times required to achieve high conversions and ee's in this asymmetric process using microwave technology [18].

In terms of ligands, phosphines are the most used ligands in Pd-catalysed asymmetric Suzuki–Miyaura coupling (Fig. 2) [19]. For instance, Buchwald and co-workers reported the enantioselective synthesis of axially chiral biaryls for a large substrate scope employing the catalytic system $Pd(OAc)_2$/KenPhos (B, Fig. 2) with ee's in the range 88–94% ee [20]. However, recently, other types of ligands were successfully employed in this asymmetric process. Phosphonite ligands (D, Fig. 2) were shown to be efficient in the asymmetric cross coupling of aryl chlorides using low catalyst loading and short reaction times (ee up to 78%) [21]. Pd/phosphoramidite–oxazoline systems (ligand E, Fig. 2) were used by Guiry in the asymmetric Suzuki–Miyaura coupling between 2-methylnaphthylboronic acid and 1-bromonaphthalene, leading to up to 46% ee at room temperature [22]. Recently, Song and Gong reported the use of chiral PCN pincer Pd complexes (I, Fig. 2) in the

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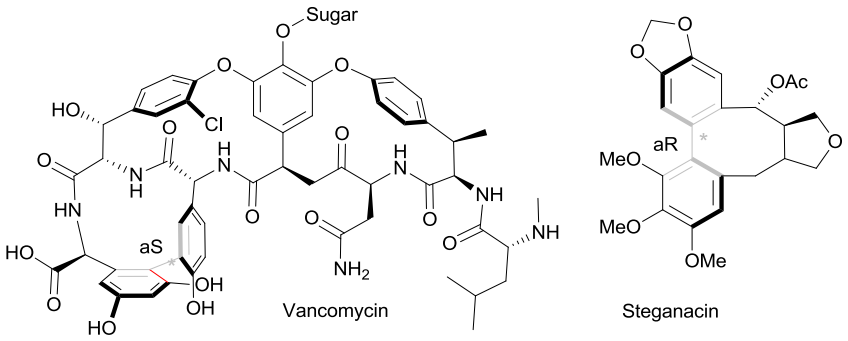


Fig. 1. Examples of natural products containing a chiral biaryl unit.

asymmetric Suzuki–Miyaura reaction with ee's up to 26% [23]. Furthermore, ligands such as chiral dienes (H, Fig. 2) were shown to be efficient in Pd-catalysed asymmetric Suzuki–Miyaura coupling reaction with ee's up to 90% [24]. Lassaletta and co-workers reported the use of C_2-symmetric nitrogen donor ligands (F, Fig. 2) with excellent ee's (up to 98%) [25]. Later, the same authors reported the use of phosphinohydrazone ligands (G, Fig. 2) in this reaction with high activity and good ee's [26].

A recyclable catalytic system was also reported by Uozumi and co-workers, who developed a polymer-supported ligand (K, Fig. 2) for the Pd-catalysed asymmetric Suzuki–Miyaura coupling of several naphthyl halide derivatives with excellent ee's [27].

Here, we present our results obtained in the coupling of napthyl bromides and iodides using a series of monodentate P-donor ligands including the commercial phosphine **1** and other ligands bearing a chiral naphthyl moiety and bidentate ligands such as chiral diphosphites and diphosphinites based on carbohydrate [28].

2. Results and discussion

2.1. Pd-catalysed asymmetric Suzuki–Miyaura coupling using Pd catalysts bearing monodentate chiral ligands *1*–*5*

First, the catalytic conditions were optimised using the commercial ligand **1** (Scheme 1) in the Pd-catalysed asymmetric Suzuki–Miyaura coupling of 2-ethoxy-1-naphthylboronic acid and 1-bromonaphthalene in the presence of various Pd/L ratios, palladium precursors, bases and solvents (see Supplementary

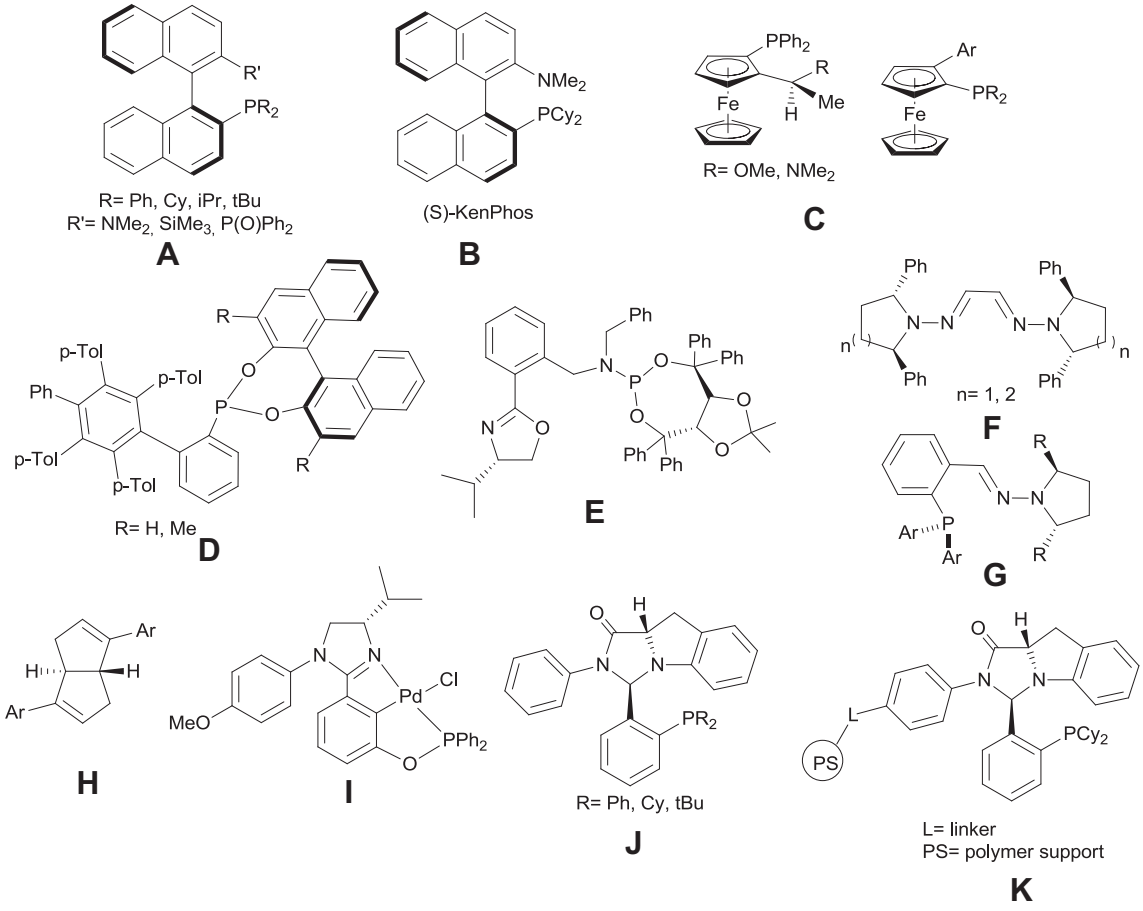
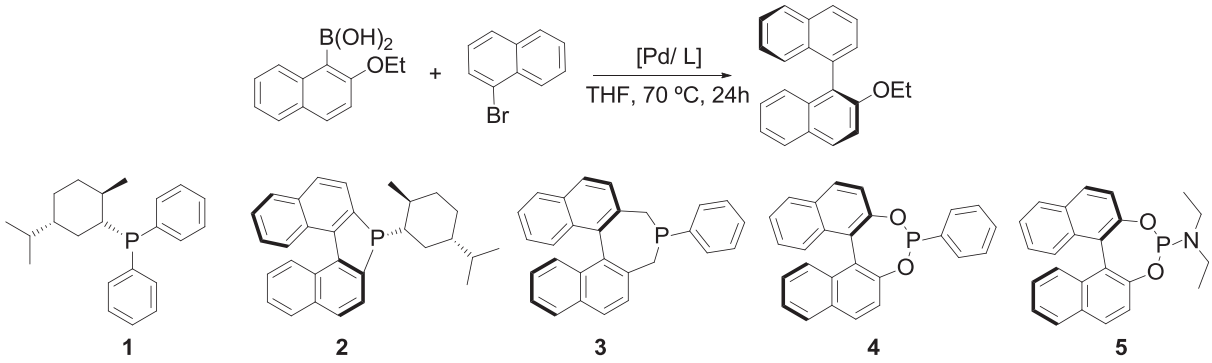


Fig. 2. Selected ligands reported in asymmetric Suzuki-coupling reactions.



Scheme 1. Chiral monodentate ligands used in this study.

information). The best results were obtained [$PdCl_2(NCPh)_2$] as Pd source, CsF as a base at 70 °C in THF during 4 h.

The catalytic systems with the ligands **1**–**5** were then tested under these conditions in the Pd-catalysed asymmetric Suzuki–Miyaura reaction of bromonaphthalene with naphthylboronic acids bearing methoxy-, ethoxy- and benzyloxy substituents in 2-position (Table 1). In all cases, excellent selectivities to the cross coupling products were obtained (>95%).

The system Pd/**1** afforded 76, 56 and 80% of conversion but only 10, 14 and 1% of ee respectively (Entries 1–3). Similar results were obtained with the catalytic system bearing the ligand **2** with 76 and 49% of conversion and 10 and 9% ee, respectively (entries 4–5). However, using the phosphine ligand **3**, lower conversions and up to 24% ee were achieved (entry 6–8).

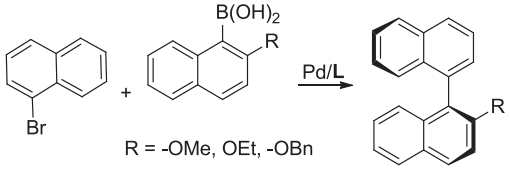
The catalytic system containing the ligand **4** afforded low conversion and ee up to 17% (entries 9–11). Finally, using the phosphoramidite ligand **5**, the reaction between 1-bromonaphthalene and 2-methoxynaphthalene-1-boronic acid afforded 23% of conversion and 10% of ee (entry 12). When 2-ethoxynaphthalene-1-boronic acid was used 33% of conversion and 12% of ee was obtained (entry 13). With 2-benzyloxynaphthalene-1-boronic acid, 50% conversion was achieved but without enantioselectivity (Entry 14).

To summarise, the monodentate ligands **1**–**5** were used in the Pd-catalysed asymmetric Suzuki–Miyaura coupling reactions of bromonaphthalene and 2-substituted naphthyl boronic acids and afforded conversions up to 80% but with only moderate to low ee's.

Next, these catalytic systems were evaluated in the Pd-catalysed asymmetric Suzuki–Miyaura reactions of iodonaphthalene with the same 2-alkoxy naphthylboronic acids and in the presence of CsF as a base at 70 °C during 4 h (Table 2). As expected, higher conversions were achieved with this substrate, but low enantioselectivity was again obtained in all cases.

Other substrates were also tested using the Pd/**1** catalytic system (Table 3). The reactions of 2-methoxy-1-bromonaphthalene with 2-OEt and 2-Me-1-naphthylboronic acids (Entries 1–2) led to fair conversions but selectivity and ee were moderate to poor. Similar results were obtained when 2-Me-1-bromonaphthalene was the substrate, independently of the naphthyl boronic acid used (Entries 3–5). No significant change was observed when 2-bromotoluene was the substrate (Entry 6).

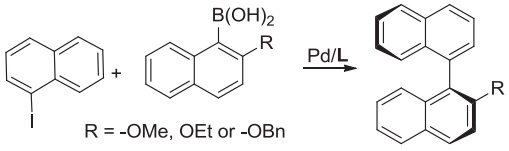
Table 1
Pd-catalysed asymmetric Suzuki–Miyaura coupling of 1-bromonaphthalene with 2-substituted 1-naphthylboronic acids using ligands **1**–**5**.[a]



Entry	L	R	Conv[b] (%)	Sel[b] (%)	ee[c] (%)
1	**1**	–OMe	76	98	10
2		–OEt	56	98	14
3		–OBn	80	99	<1
4	**2**	–OMe	76	99	10
5		–OEt	49	99	9
6	**3**	–OMe	15	94	24
7		–OEt	21	98	9
8		–OBn	11	99	<1
9	**4**	–OMe	19	98	7
10		–OEt	15	99	17
11		–OBn	3	–	<1
12	**5**	–OMe	23	97	10
13		–OEt	33	98	12
14		–OBn	50	99	<1

[a] Reaction conditions: 1.0 mmol bromonaphthalene, 2.0 mmol naphthylboronic acid, 5 mmol CsF, 1 mol % Pd precursor, 1.5 mol% ligand, 3 mL of THF, $T = 70$ °C, $t = 4$ h.
[b] Determined by GC.
[c] Determined by HPLC.

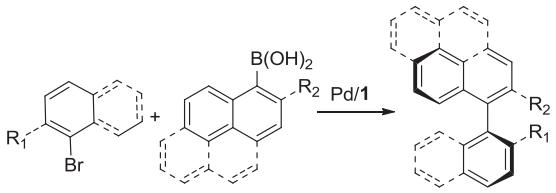
Table 2
Pd-catalysed asymmetric Suzuki–Miyaura coupling of 1-iodonaphthalene with 2-substituted 1-naphthylboronic acids using ligands **1**–**5**.[a]

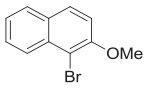
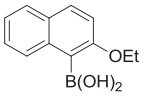
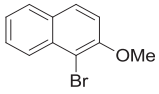
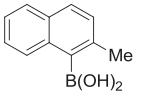
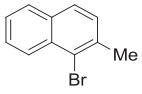
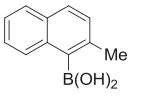
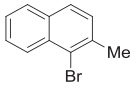
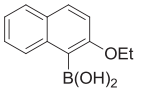
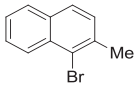
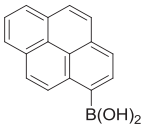
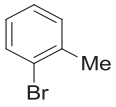
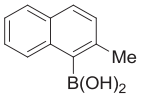


Entry	L	R	Conv (%)[b]	Sel (%)[b]	ee (%)[c]
1	**1**	–OMe	80	98	22
2	**1**	–OEt	66	99	14
3	**1**	–OBn	83	99	1
4	**2**	–OMe	73	93	15
5	**2**	–OEt	60	96	7
6	**3**	–OMe	63	98	12
7	**3**	–OEt	17	99	9
8	**3**	–OBn	73	99	4
9	**4**	–OMe	3	–	–
10	**4**	–OEt	67	98	14
11	**4**	–OBn	73	99	3
12	**5**	–OMe	64	98	16
13	**5**	–OEt	54	99	7
14	**5**	–OBn	40	99	2

[a] Reaction conditions: 1.0 mmol iodonaphthalene, 2.0 mmol naphthylboronic acid, 5 mmol base, 1 mol % Pd precursor, 1.5 mol% ligand, 3 mL of THF, $T = 70$ °C, $t = 4$ h.
[b] Determined by GC.
[c] Determined by HPLC.

Table 3
Pd-catalysed asymmetric Suzuki–Miyaura coupling of various substrates using Pd catalyst bearing monodentate chiral ligand **1**.[a]



Entry	Aryl halide	Boronic acid	Conv (%)[b]	Sel (%)[b,c]	ee (%)[d]
1			57	89	35
2			65	50	12
3			53	89	24
4			48	80	28
5			67	99	26
6			65	87	10

[a] Reaction conditions: 1.0 mmol bromonaphthalene, 2.0 mmol naphthylboronic acid, 5 mmol CsF, 1 mol % $[PdCl_2(PhCN)_2]$, 1.5 mol% ligand **1**, 3 mL of THF, $t = 24$ h, $T = 70$ °C.
[b] Determined by GC.
[c] Selectivity to the cross-coupling product.
[d] Determined by HPLC.

When the reaction described in entry 1 (Table 3) was repeated at a larger scale (5 mmol of substrate), the conversion, selectivity and ee were slightly lower (50, 75 and 20%, respectively). The enantiomeric mixture was isolated in 25% yield.

To summarise, the Pd system bearing the ligand **1** catalyses the asymmetric Suzuki–Miyaura coupling reaction of several bromide substrates and substituted 2-naphthylboronic acids with moderate conversions (up to 67%) and low to moderate ee's (up to 35%).

In view of the poor enantioinduction achieved with monodentate ligands, we decided to screen in the asymmetric Suzuki–Miyaura coupling a range of bidentate ligands including chiral diphosphines, diphosphinites and diphosphites.

2.2. Pd-catalysed asymmetric Suzuki–Miyaura coupling using Pd catalysts bearing bidentate chiral ligands

The bidentate ligands used in this study are presented in Fig. 3.

The diphosphinite ligand **8**, which was reported in the enantioselective Rh-and Ir-catalysed asymmetric hydrogenation of unsaturated substrates [29], features the same backbone than the ligand **12**, which is very efficient in allylic alkylation and amination reactions [30].

Although crystals of Pd complexes bearing the ligand **12** could not be obtained, suitable colourless crystals for X-ray diffraction were obtained for the platinum derivative $[PtCl_2(\mathbf{12})]$ by slow diffusion of Et_2O into a CH_2Cl_2 solution of the complex. The X-ray analysis shows the presence of large voids (20.5%) in the unit cell, but only one diethyl ether molecule per complex was successfully refined. The molecular structure of the complex is shown in Fig. 4 and the corresponding selected bond distances and angles are summarised in Table 4. The complex is located on a crystallographic two-fold axis that, passing through the metal and the tetrahydrofuranyl oxygen O4, relates the two chlorine atoms and the phosphite moieties.

The metal presents a square planar geometry with coplanar donor atoms. The Pt–P bond lengths (2.201(4) Å) are slightly shorter than the Pt–Cl ones (2.335(4) Å). It is worth noting the difference between the Cl(1)–Pt–Cl(1′) and the chelating P(1′)–Pt–P(1) bond angle (89.9(2) vs 101.91(18)°), likely induced by steric constraints and by the geometry of the chelating ligand. The rings of the biphenyl moiety are tilted by *ca.* 50°.

After optimisation of the reaction conditions using ligand **12**, the optimum Pd/L ratio was determined to be 1/1.5 (see Supplementary information). The catalytic asymmetric Pd-catalysed coupling of iodonaphthalene with substituted naphthyl boronic acids were carried out using the ligands **6**–**13**. All the catalysts showed high chemoselectivity towards the desired cross coupling products (>97%). The results are summarised in Table 5.

With the diphosphine ligands **6**–**7**, moderate conversions (*ca.* 30–50%) with very low ee's (up to 17%) were achieved (Entries 1–4). With the C_2-symmetry diphosphinite ligand **8**, similar conversions and ee were obtained (Entries 5–6). Slightly higher ee's (*ca.* 30%) but large difference in conversions were observed for both boronic acids when the C_1-symmetry diphosphinite **9** was used (Entries 7–8). The diphosphite **10**, featuring the same chiral backbone than **9**, gave an excellent conversion and a slight increase in ee to 37% (Entry 9). Finally, with the diphosphite ligands **11**–**13**, moderate conversion (*ca.* 50%) and low ee's were obtained (Entries 10–15). To the best of our knowledge, this is the first time that chiral diphosphite ligands are applied in the asymmetric Suzuki–Miyaura reaction.

These results thus indicate that diphosphite ligands are efficient ligands in this asymmetric process, especially in terms of activity and selectivity, although structural modification of the ligands are required for the enantioselectivity of the process to be improved.

3. Conclusions

A range of Pd catalytic systems with mono- and bidentate ligands, including diphosphonites and diphosphites derived from carbohydrates, were screened for the first time in the asymmetric Suzuki–Miyaura coupling of iodo- and bromonaphthalenes and substituted naphthylboronic acids. As to monodentate ligands, the neomenthyl phosphine ligand **1** provided the highest activity with an excellent selectivity to the cross coupling product (up to >99%) and moderate enantioselectivity (up to *ca.* 35%). The results obtained with the bidentate ligands **6**–**13** indicate that, even if the ee's obtained in this work are far from being exciting, π-acidic ligands such as diphosphites hold the promise to be efficient in the Pd-catalysed asymmetric Suzuki coupling of these substrates.

4. Experimental part

4.1. General methods

All air- or water-sensitive reactions were performed using standard Schlenk techniques under a nitrogen atmosphere.

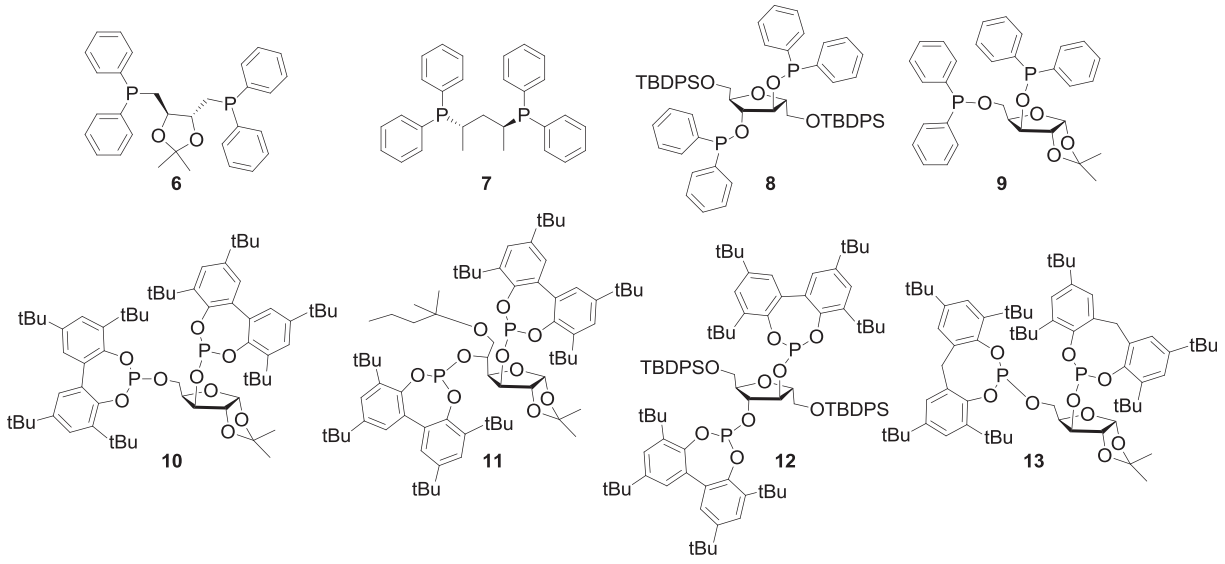


Fig. 3. Chiral bidentate ligands used in Pd-catalysed asymmetric Suzuki–Miyaura coupling reactions.

Chemicals were purchased from Aldrich Chemical Co and Fluka. All solvents were distilled over drying reagents and were deoxygenated before use. The precursors [$PdCl_2$(COD)] [31] (COD = 1,5-cyclooctadiene) and [$PdCl_2(PhCN)_2$] [32] were prepared following previously described methods. The boronic acids used in this study were either purchased from Aldrich Chemical or synthesised according to literature procedures [33]. The syntheses of the ligands **2**–**5** [34] and **8**–**13** were performed according to previously reported procedures [28b,35].

The deuterated solvents for NMR measurements were dried over molecular sieves prior to use. NMR spectra were acquired on a Varian Mercury 400 MHz spectrometer.

Merck silica gel 60 (0.040–0.063 mm) was employed for flash chromatography. The conversion of the reaction was measured by GC on a Hewlett–Packard HP 5890 A instrument (split/splitless injector, J&W scientific, HP5, 25 m column, internal diameter 0.25 mm, film thickness 0.33 mm, carrier gas: 150 kPa He, F.I.D. detector) equipped with a Hewlett–Packard eHP3396 series II integrator. The ee values were determined by High Performance Liquid Chromatography using a Daicel AD-H column.

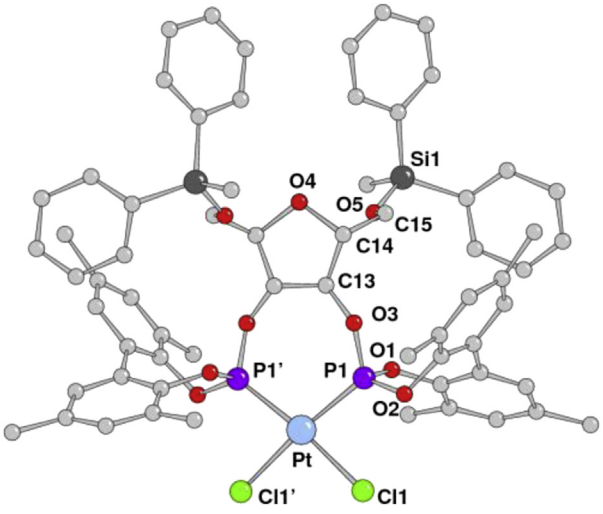


Fig. 4. Molecular structure of the complex [$PtCl_2$(**12**)] (methyl groups of ^{t}Bu not shown for clarity).

4.2. General procedure for asymmetric Suzuki–Miyaura coupling reaction

A Schlenk tube was charged with 0.25 mmol of naphthyl halide, 0.0025 mmol of Pd precursor, 0.00375 mmol of the appropriate chiral ligand, 0.5 mmol of boronic acid, and 1.25 mmol of base. Anhydrous solvent was added, the flask was sealed and the mixture was stirred and heated at the corresponding temperature. The reaction mixture was treated with 10 mL of distilled water, extracted with 3 × 10 mL of CH_2Cl_2, dried over $MgSO_4$, and purified by flash chromatography to obtain the corresponding products. The conversion and selectivity was monitored by gas chromatography. The ee values were determined by High Performance Liquid Chromatography.

4.3. Crystallographic measurements

Data collection was carried out on a Cu rotating anode equipped with a Bruker CCD2000 detector (λ = 1.54178 Å). Cell refinement, indexing and scaling of the data set were performed using programs Denzo and Scalepack [36]. The structure was solved by direct methods, subsequent Fourier analyses [37] and refined by the full-matrix least-squares method based on F^2 with all observed reflections [37]. The asymmetric unit of the compound contains half crystallographic independent OEt_2 molecule. Since the crystal presents a large void of 1137.7 Å^3 correspondent to 20.5% of the unit cell, the program Squeeze [38] was applied to the data set indicating 130 electrons per unit cell. The calculations were performed using the WinGX System, Ver 1.80.05 [39].

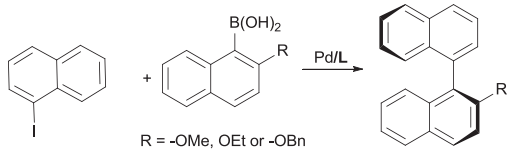
Crystal data: $C_{94}H_{126}Cl_2O_9P_2PtSi_2.C_4H_{10}O$, M = 1858.18, monoclinic, space group *C*2, a = 21.970(2), b = 19.724(2), c = 13.19 80(4) Å, β = 104.120(11)°, V = 5546.4(10) Å^3, Z = 2, D_c = 1.113 g/cm^3,

Table 4
Selected bond lengths (Å) and angles (°) for complex [$PtCl_2$(**12**)].

Bond lengths (Å)		Bond angles (°)	
Pt–P(1)	2.201(4)	Cl(1)–Pt–Cl(1′)	89.9(2)
Pt–Cl(1)	2.335(4)	P(1)–Pt–P(1′)	101.91(18)
P(1)–O(1)	1.577(8)	P(1′)–Pt–Cl(1)	174.00(17)
P(1)–O(2)	1.576(7)	P(1)–Pt–Cl(1)	84.09(11)
P(1)–O(3)	1.580(7)	C(13)–O(3)–P(1)	125.9(5)

Primed atoms at $-x+1, y, -z+1$.

Table 5
Pd-catalysed asymmetric Suzuki–Miyaura coupling using Pd catalyst with bidentate chiral ligands **6**–**13**.[a]



Entry	Ligand	−B(OH)$_2$	Conv (%)[b]	Sel (%)[b]	ee (%)[c]
1	**6**	−OMe	32	99	3
2	**6**	−OEt	40	99	4
3	**7**	−OMe	50	97	17
4	**7**	−OEt	31	98	3
5	**8**	−OMe	36	99	22
6	**8**	−OEt	56	97	17
7	**9**	−OMe	14	98	30
8	**9**	−OEt	73	99	21
9	**10**	−OEt	96	99	37
10	**11**	−OMe	50	98	4
11	**11**	−OEt	49	99	19
12	**12**	−OMe	65	98	2
13	**12**	−OEt	56	98	11
14	**13**	−OMe	58	99	8
15	**13**	−OEt	43	98	12

[a] Reaction conditions: 1.0 mmol iodonaphthalene, 2.0 mmol naphthylboronic acid, 5 mmol CsF, 1.0 mol% Pd precursor, 1.0 mol% ligand, 3 mL of THF, $t = 4$ h, $T = 70$ °C.
[b] Determined by GC.
[c] Determined by HPLC.

μ(Cu-Kα) = 3.661 mm^{-1}, $F(000) = 1948$, θ range = 3.05–46.16°. Final $R1 = 0.0454$, $wR2 = 0.1130$, $S = 1.072$ for 521 parameters, 11,510 reflections, 4217 unique (R(int) = 0.0440), residuals in ΔF map 0.954, −0.505 e. Å^{-3}.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jorganchem.2013.06.022.

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