The Suzuki-Miyaura Reaction Performed Using a Palladium-N-Heterocyclic Carbene Catalyst and a Weak Inorganic Base

Frédéric Izquierdo,[a] Martin Corpet[a] and Steven P. Nolan [a]*

Abstract: N-Heterocyclic carbenes (NHCs) have been shown to be useful ligands for the Suzuki-Miyaura cross-coupling at low catalyst loadings. We now report that the commercially available and air-stable [Pd(IPr)(cin)Cl] pre-catalyst permits the formation of various functionalized biaryls from aryl chlorides and boronic acids (37 examples) under very mild conditions using a mixture of ethanol/water as solvent and an inorganic base.

INTRODUCTION

Palladium-catalyzed cross-coupling reactions are a popular method of forming C-C bonds and have become a standard synthetic assembly tool in organic and organometallic chemistry.[1] Amongst these, the Suzuki-Miyaura reaction represents a preferred methodology. In an effort to reduce the generation of hazardous substances, variations of the Suzuki-Miyaura cross-couplings have been developed using *green* solvents,[2] particularly water.[3] These variations are especially attractive as water is nontoxic, non-flammable, inexpensive, and can be easily separated from organic products. Amongst the different approaches developed to reach *greener* reaction conditions, the most often used procedures are based on mixtures of organic solvents and water,[3b, 3e, 4] as well as modifications of the ligand, including the addition of functional groups which secure the water-solubility of the catalyst (*e.g.*, sulfonate) or use of phase-transfer reagents.[4b-d, 4f, 4g, 5] However, these solutions often require tedious synthetic procedures to obtain the desired pre-catalysts.

While most of the early catalytic protocols involve phosphine ligands,[3e, 3i, 6] N-heterocyclic carbenes (NHCs) complexes have shown remarkable catalytic performance,[7] including in the Suzuki-Miyaura reaction.[4k, 8] Numerous complexes of the [Pd(NHC)(cin)Cl] (cin = cinnamyl) family are now commercially available, and these complexes have proven effective in the Suzuki-Miyaura coupling, but usually require the use of a strong base (*e.g.*, KO^{t}Bu) to generate the catalytically active species which limits the functional group tolerance and the choice of solvent.[4k, 8a, 9] Colacot and co-workers have shown that their Pd-(allyl) system could be activated using to K_2CO_3 in a toluene/water mixture.[10] Recently, Hazari and co-workers have described the preparation of the dinuclear Pd(I) complex [Pd_2(IPr)$_2$(η^3-cin)Cl] (IPr: 1,3-*bis*(2,6-di*iso*propylphenyl)imidazol-2-ylidene) *via* the reduction of [Pd(IPr)(cin)Cl] in an EtOH/K_2CO_3 system. In this report, it was proposed that this complex is in equilibrium with the catalytically active Pd^0-NHC intermediate *via* a disproportionation reaction. Based on these studies the Hazari group proposed the mechanism depicted in Scheme 1 (A).[11] Similarly, Minakata and co-workers showed that the [Pd(SIPr)(cin)Cl] (SIPr: 1,3-bis(2,6-di*iso*propylphenyl)imidazolin-2-ylidene) pre-catalyst could also be activated using a mild base (Na_2CO_3) and boronic acid as a reductant in a toluene:water mixture (Scheme 1, B).[12]

[a] EaStCHEM School of Chemistry, University of St Andrews, St Andrews, KY16 9ST, UK.
Fax: (+44) 1334 463 808; Tel: (+44) 1334 463 763;
E-mail: snolan@st-andrews.ac.uk.

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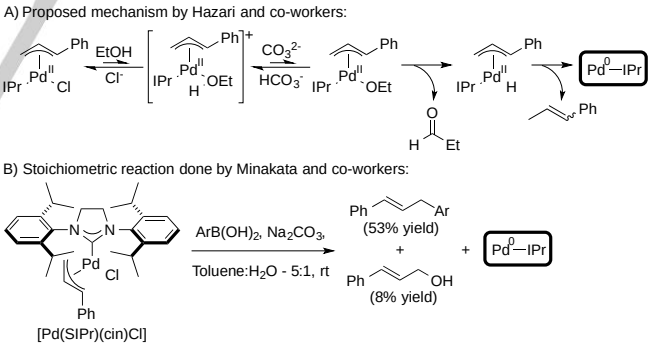
RESULTS AND DISCUSSION

With these interesting reports in mind, we decided to further investigate the use of alcohol solvents in the Suzuki-Miyaura cross-coupling reaction using commercially available Pd-NHC pre-catalysts in the presence of mild inorganic bases. We began our study examining the reaction of phenylboronic acid and *p*-chlorotoluene in ethanol at 40 °C, using the commercially available [Pd(NHC)(cin)Cl] pre-catalysts (NHC = IPr (**A**), SIPr (**B**), IPr* (**C**): 1,3-bis(2,6-bis(diphenylmethyl)-4-methylphenyl)imidazol-2-ylidene).[13] Among these catalysts, the IPr and SIPr-based pre-catalysts showed good conversions with 65% and 64% respectively after 1 h. The pre-catalyst bearing the bulkiest NHC, IPr*, gave low conversion (9%). The latter required longer reaction time to afford full conversion. This is possibly due to a slower activation of the pre-catalyst due to steric congestion of the metal center.[14] At 0.5 mol% catalyst loading, the conversion remained nearly the same if the temperature was increased to 80 °C. Different solvent/base combinations were also studied. Lower conversions were observed with other inorganic bases such as Na_2CO_3 or K_3PO_4. Poorer conversions were also obtained using other alcohols.[13]

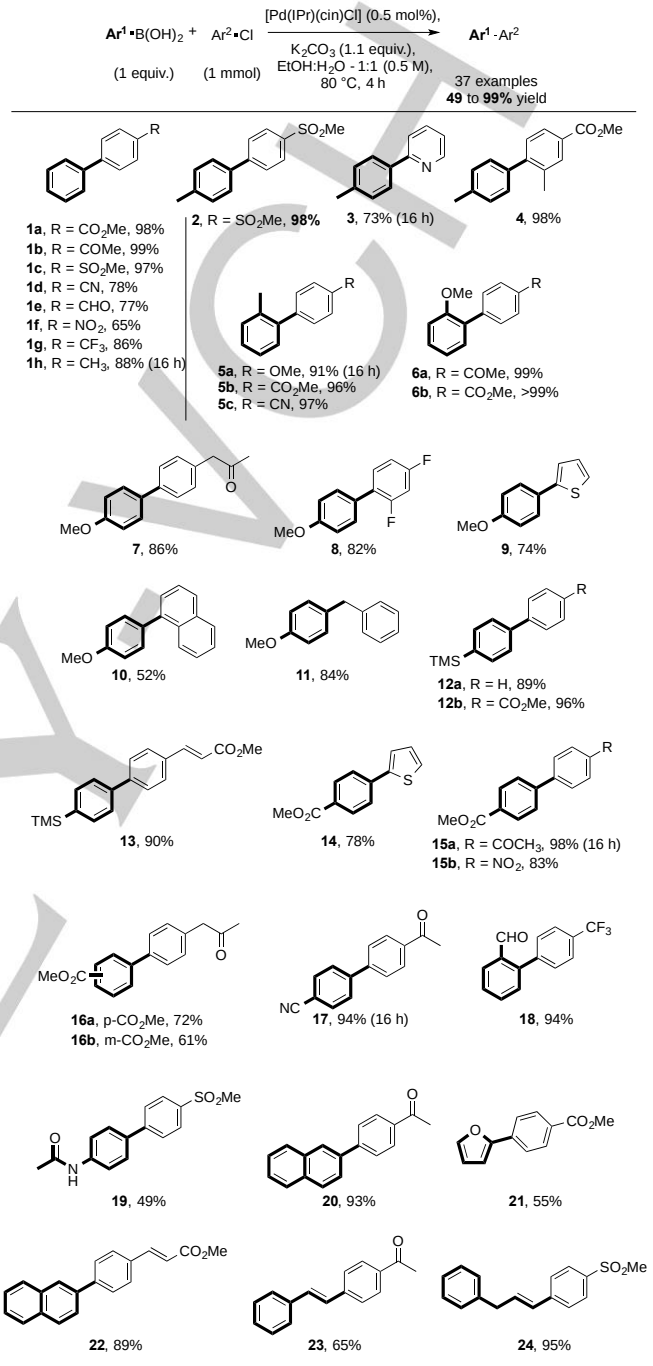


Scheme 1. Activation pathways of [Pd(NHC)(cin)Cl] pre-catalyst.

Further optimization was performed on a more activated substrate, methyl 4-chlorobenzoate, to gauge the viability of the inorganic base with more reactive substrates.[13] Under the previously described conditions, full conversion of starting material was observed with this substrate. However, trans-esterification with the solvent proved to be problematic leading to a 63:37 ratio of methyl to ethyl ester. Decreasing the amount of boronic acid was found to be detrimental affording an unfavorable ratio (Me:Et = 23:77). As the formation of the trans-esterification product can be associated with the excess base used, we were pleased to observe almost no by-product using 1.1 equivalent of base (Me:Et = 93:7). In order to develop *greener* conditions and to possibly increase further the reactivity

of the boronic acid different ethanol:water ratios were studied. Finally, a ratio of ethanol:water of 1:1, gave full conversion with no observed transesterification, and thus was chosen to evaluate the scope of the methodology. The optimized conditions are then 0.5 mol% of **A**, 1 equivalent of boronic acid and 1.1 equivalents of K_2CO_3 in a 1:1 ethanol:water mixture at 80 °C in air. Under these conditions, clean and complete conversion could be achieved in 4 h for methyl 4-chlorobenzoate, and in 16 h for *p*-chlorotoluene. Other commercially available ethanol:water mixtures were tested and resulted in similar high yields.[12],[15]

With the optimized reaction conditions in hand, the scope and limitations of the transformation were studied (Scheme 2). Various aryl chlorides were coupled with phenylboronic acid. The methodology allowed the coupling of a wide range of aryl chlorides bearing different functional groups in the *para*- position with very good to excellent yields (**1a-1h**, 65 to 99% isolated yield), nonetheless coupling reaction involving alcohols or carboxylic acids could not be achieved. In many cases, the final product was obtained with a purity higher than 95% without the need for further purification (**1a-c**, **2-4**, **5b-c**, **6a-b**, **8**, **15a** and **18**).[13] With a non-activating group such as a methyl group, the desired product was obtained in very good yield but required a longer reaction time (**1h**, 88%). A broad range of boronic acids with varying electronic and steric properties were next investigated. Following the same trend as its phenyl derivative, the *p*-tolylboronic acid reacted cleanly with a sulfone-functionalized chloride (**2**, 98%). The same boronic acid was coupled with 2-chloropyridine to afford the desired compound (**3**) after 16 h in 73% isolated yield as well as with methyl 4-chloro-3-methylbenzoate giving the corresponding product in excellent yield (**4,** 98%). The more hindered *o*-tolylboronic acid reacted efficiently with *p*-chloroanisole (**5a**, 91%) after 16 h while the reaction with activated substrates gave the desired product also in very high yields (**5b**, **5c**) after the standard 4 h reaction time. Furthermore, using activated boronic acid such as *o*-methoxyphenylboronic acid gave excellent yields (**6a-b**). Its *p*-methoxy- derivative allowed the coupling with many aryl partners (**7-11**), even with the less common *p*-chlorophenylacetone (**7**, 86%). The intermediate of diflunisal[16] (**8**) was achieved in very good yield (82%), without the need for any additional purification steps. On the other hand, the reaction of *p*-methoxyphenylboronic acid was less efficient with 2-chlorothiophene and 2-chloronaphthalene, affording the products in good to moderate yields (**9** and **10**, 74% and 52%), presumably due to steric hindrance in the latter case. The same boronic acid reacted very efficiently with benzyl chloride to afford the expected compound (**11**) in 84% isolated yield. The *p*-trimethylsilylphenylboronic acid coupled very well with both unactivated and activated aryl chlorides (**12a-b**, 89 and 96%) as well as with methyl *p*-chlorocinnamate (**13**, 90%). Unactivated boronic acids were then investigated (**14**-**19**). Boronic acid bearing a *p*-carboxymethyl group reacted efficiently with 2-chlorothiophene (**14**, 78%) and with functionalized aryl chlorides (**15a-b** and **16a-b**, 61 to 98%). Another unactivated boronic acid, functionalized with a cyano group in the *para*- position, reacted smoothly with *p*-chloroacetophenone to afford the desired product after 16 h (**17**, 94%). The cross-coupling reaction between *o*-formylphenylboronic acid and *p*-chlorobenzotrifluoride led to the Xenalipin intermediate[17] (**18**) in excellent yield without further purification. The more challenging *p*-acetamidophenylboronic acid was reacted with *p*-chlorophenylmethyl sulfone and yielded **19** in a moderate 49% isolated yield. 2-Naphtylboronic acid also reacted efficiently with activated aryl chlorides (**20** and **22**).



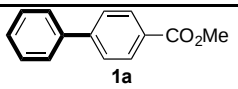
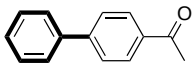
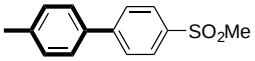
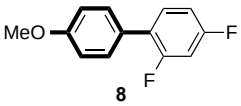
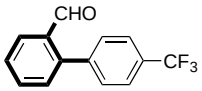
Scheme 2. Scope and limitations of the Suzuki-Miyaura coupling with [Pd(IPr)(cin)(Cl].

In comparison with previous work, Nechaev describes a similar method using a six-membered ring derivative of IPr with a phase-transfer agent in water at reflux. This method appears limited to unfunctionalized heteroaromatic compounds.[18] The present method appears much more versatile and is straightforward to launch.

Finally, the robustness of the methodology was tested on a multi-gram scale for a selection of products (Table 1). We were

pleased to see that **1a** (Table 1, entry 1) was obtained in excellent yield after workup using only 0.25 mol% of catalyst. Compound **1b** was isolated in 90% yield after column chromatography. The reaction afforded **1c** (Entry 3) in excellent yield after recrystallisation from hot ethylacetate. Finally, the Diflusinal (Table 1, entry 4) and Xenalipin intermediates (Entry 5) were isolated in good yields after extraction, without the need of chromatography.

Table 1. Scale up of the Suzuki-Miyaura cross-coupling using [Pd(IPr)(cin)Cl].[a]

Entry	Product	Yield (%)[b]
1	 **1a**	>99[c] (10.6 g)[d]
2	 **1b**	90 (1.8 g)[d]
3	 **1c**	96 (11.8 g)[d]
4	 **8**	75 (5.8 g)[d]
5	 **18**	78 (6.5 g)[d]

[a] Reaction conditions: In a flask fitted with a septum and a stirring bar, were added successively all the reagents, the pre-catalyst and the solvents. The mixture was heated at 80 °C for 4 h. [b] Isolated yield. [c] Reaction conducted with 0.25 mol% of catalyst. [d] After purification, providing gram quantities of isolated products.

CONCLUSION

In conclusion, we have described a very user-friendly methodology for the Suzuki-Miyaura reaction under non-inert conditions, using a commercially available Pd-(NHC) complex as pre-catalyst at low catalyst loading. The reactions were optimized in a 1:1 water and ethanol mixture in the presence of a mild and inexpensive inorganic base that permits to extend the scope of the Suzuki-Miyaura reaction using Pd-NHC as pre-catalysts. We have shown that this system can couple a wide range of aryl chlorides and boronic acids in good to excellent yields and have exemplified this with a number of examples conducted on a multigram scale.

Experimental Section

General procedure for the Suzuki-Miyaura reaction in ethanol:water. Under normal atmosphere, in a vial equipped with a stirring bar and sealed with a screw cap fitted with a septum, were added the boronic acid (0.5 mmol, 1 equiv.), K_2CO_3 (76 mg, 0.55 mmol, 1.1 equiv.) and the aryl-, heteroarylchloride (0.5 mmol). Then, were added successively distilled water (0.5 mL) and [Pd(IPr)(cin)(Cl] (1.62 mg, $2.5.10^{-3}$ mmol, 0.5 mol%) in solution in EtOH (0.5 mL). After reaction at 80°C, the mixture was allowed to cool to room temperature. An equal volume of water and CH_2Cl_2 (10 mL) was added and the aqueous phase was extracted with CH_2Cl_2 (3x10 mL). The combined organic phases were dried over $MgSO_4$ and the solvent was removed under vacuum. As a function of the substrate, the desired product may or may not require further purification but usually does not.

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Keywords: Catalysis • Cross-coupling • NHC • Palladium • Suzuki-Miyaura

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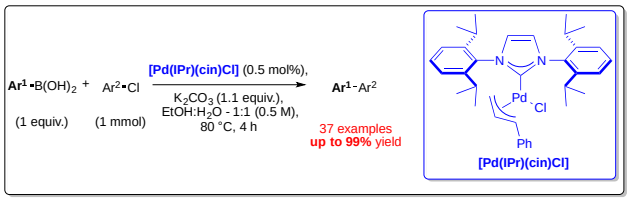
SHORT COMMUNICATION



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