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One-step highly selective borylation/Suzuki cross-coupling of two distinct aryl bromides in pure water⁺

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A Na₂PdCl₄-catalysed B₂(OH)₄-mediated direct cross-coupling of two distinct aryl bromides in pure water is described. This one-step borylation/Suzuki method exhibits an outstanding cross-coupling selectivity and no tendency to produce homocoupling products, therefore leading to biaryls and heterobiaryls in moderate to good yields. Moreover, the reaction has high regio-selectivity and can be scaled-up. Using such a simple, economical and practical protocol, the unsymmetrical diarylation of 3,5-dibromopryidine is achieved.

Biaryls are ubiquitous in natural products, pharmaceuticals and organic materials, and are often used as ligands in transition metal catalysis.¹ Although to date Pd- or Ni-catalysed crosscouplings of arylmetallic reagents (ArM; M = B, Sn, Si, Zn, Mg, Mn, Bi, In, etc.) with aryl halides are some of the most effective methods to form biaryls,²⁻⁹ the need to preform each arylmetallic reagent can be limiting. For example, due to the synthetic challenges and environmental concerns in the preparation and purification of arylboron reagents,^{2a,c,10} the Suzuki crosscoupling is somewhat inefficient.¹¹ In this regard, one-pot multistep borylation/Suzuki cross-coupling (MBSC) strategies have been recently developed for avoiding the isolation of arylboron intermediates.¹²⁻¹⁴ To achieve reactivity and cross-selectivity, for example, the C-X12 or C-H13 MBSCs require expensive ligands and/or boron sources as well as organic solvents for the borylation reaction, and/or double loading of specific catalysts in the Suzuki step; the Sandmeyer-type¹⁴ MBSC is a more complex process involving three consecutive steps of diazotization, borylation and Suzuki coupling in organic co-solvents. On the other hand, although the Pd, Ni or Co-catalysed direct cross-coupling of two aryl halides provides more straightforward access to biaryls,^{15–17} this procedure not only needs to combine with reductive agents, but also requires large excess of one coupling partner for producing the cross-coupling products. In most cases, it also suffers from long reaction time, harsh conditions as well as a narrow scope of the substrates. As such, there is a continued need to develop simpler, more efficient and selective cross-coupling methods for the construction of biaryls.

We envisaged that an ideal strategy for biaryl synthesis could be an optimal combination of the direct procedure and MBSC reaction, *i.e.* the one-step borylation/Suzuki cross-coupling (OBSC) of two different aryl halides. Obviously this method that integrates the C–X borylation and the Suzuki coupling into one step faces a considerably more difficult synthetic challenge, because of the inevitable homo-coupling originating from nonselective and often incomplete borylation. To the best of our knowledge, there have been no reports on such an OBSC reaction.

Water as a solvent can offer unique reactivity and selectivity that cannot be attained in organic solvents;¹⁸ it was therefore anticipated that the use of pure water as a solvent might lead to an effective OBSC reaction with high reactivity and crossselectivity even when two aryl bromides are employed as the substrates, *albeit* different halides with a significant difference in reactivity (*e.g.*, ArI/ArBr or ArBr/ArCl) are normally used for biaryl cross-couplings. Here, we first report the Na₂PdCl₄-catalysed B₂(OH)₄-mediated OBSC of two different aryl bromides in pure water (Scheme 1). Moreover, the application of this protocol for the regioselective C2-arylation of 2,5-dibromopyidine and the unsymmetrical diarylation of 3,5-dibromopyidine is described.

By selecting equimolar amounts of PhBr and 4-BrPhCOCH₃ as model substrates, we began to investigate this OBSC reaction with a slight excess of $B_2(OH)_4$ or B_2pin_2 , using 1–2 mol% Pd catalyst in the presence of several bases, *i.e.* Na₂CO₃, K₃PO₄, NaOAc or NaOH (Table 1). It was found that the reaction did not occur in pure water at 100 °C unless NaOH was used to obtain the product **1a** in an 87% yield (Table 1; entries 1 and 2). In this case sodium dodecyl sulfate (SDS) as an additive improved the yield (99% yield, Table 1, entry 3). The use of PCy₃HBF₄¹⁹ in a 4:1 ratio with Na₂PdCl₄ accelerated the reaction (Table 1, entry 4), even in the presence of only 1 mol% Pd catalyst (Table 1, entry 5). Under the conditions of entry 4, the absence of $B_2(OH)_4$ resulted

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Scheme 1 A one-step method for the borylation/Suzuki cross-coupling of two distinct aryl bromides in pure water.

Table 1 Optimization of the reaction conditions^a

	Br + Br	Pd cat., Base Boron source Solv., additive	Ph	∠COCH₃ 1a
Entry	Pd Cat./ligand	B source	Additive	Yield ^b (%)
1	Na ₂ PdCl ₄	$B_2(OH)_4$		0 ^c
2	Na ₂ PdCl ₄	$B_2(OH)_4$		87
3	Na_2PdCl_4	$B_2(OH)_4$	SDS	99
4	Na ₂ PdCl ₄ /PCy ₃ HBF ₄	$B_2(OH)_4$	SDS	100^d
5	Na ₂ PdCl ₄ /PCy ₃ HBF ₄	$B_2(OH)_4$	SDS	96 ^e
6	Na ₂ PdCl ₄ /PCy ₃ HBF ₄		SDS	0
7	Na ₂ PdCl ₄ /PCy ₃ HBF ₄	$B_2(pin)_2$	SDS	0
8	Pd/C	$B_2(OH)_4$	SDS	0
9	PdCl ₂ /PCy ₃ HBF ₄	$B_2(OH)_4$	SDS	12
10	$Pd(OAc)_2/PCy_3HBF_4$	$B_2(OH)_4$	SDS	8
11 ^f	Na ₂ PdCl ₄ /PCy ₃ HBF ₄	$B_2(OH)_4$	SDS	0^{g}
12^{h}	Na ₂ PdCl ₄ /PCy ₃ HBF ₄	$B_2(OH)_4$	SDS	0^{i}
13	Na ₂ PdCl ₄ /PCy ₃ HBF ₄	$B_2(OH)_4$		5^{j}
14	Na ₂ PdCl ₄ /PCy ₃ HBF ₄	$B_2(OH)_4$		0^k

^{*a*} Reaction conditions: PhBr (1 mmol), 4-BrPhCOCH₃ (1 mmol), boron source (1.2 mmol), NaOH (2.5 mmol), SDS (10 mol%), Pd catalyst (2 mol%), PCy₃HBF₄ (8 mol%). Pd catalyst and PCy₃HBF₄ in 2.5 ml water were preheated at 60 °C for 0.5 h before addition of substrates and another 2.5 ml portion of water. The reaction mixture was refluxed at 100 °C for 12 h. ^{*b*} GC yield. ^{*c*} 2.5 mmol of Na₂CO₃, K₃PO₄ or NaOAc was used as base. ^{*d*} Refluxing for 4 h. ^{*e*} 1 mol% Na₂PdCl₄ and 4 mol% PCy₃HBF₄. ^{*f*} 2 mmol of PhBr as substrate. ^{*g*} Yield of Ph-Ph. ^{*h*} 2 mmol of 4-BrPhCOCH₃ as substrate. ^{*i*} Yield of CH₃COPh-PhCOCH₃. ^{*j*} EtOH as solvent at reflux for 12 h. ^{*k*} DMF: H₂O in 1:1 (v/v) as solvent at 100 °C for 12 h.

in no reaction (Table 1; entry 6), demonstrating that the C-Br borylation to intermediate boronic acid is the key to realizing the OBSC reaction. The attempts to replace $B_2(OH)_4$ by $B_2pin_{24}^{20}$ another active borylating agent in aqueous solution failed (Table 1; entry 7), indicating that $B_2(OH)_4$ plays an irreplaceable role. Several other catalysts *i.e.* Pd/C, $PdCl_2$ and $Pd(OAc)_2$ were also investigated and found to have no catalyst activity (Table 1; entries 8-10). It is particularly noteworthy that, despite the homo-coupling being inevitable in the direct cross-coupling of aryl halides,¹⁵⁻¹⁷ no such product (neither Ph–Ph nor CH₃COPh– PhCOCH₃) was observed in this Na₂PdCl₄-B₂(OH)₄-H₂O system (for further details see ESI[†]) even in the presence of PhBr (or 4-BrPhCOCH₃) alone as a substrate (Table 1; entries 11 or 12). To investigate the contribution of pure water as a solvent to the reactivity and the complete cross-selectivity, the reaction was conducted in conventional solvents as compared to that in pure water. However, EtOH as the solvent led to almost no product of **1a** apart from large amounts of homo-coupling products (Table 1; entry 13); a DMF/H₂O (1:1, v/v) solvent could not make the reaction happen (Table 1; entry 14). These results show that pure water as a solvent favours the reactivity and cross-coupling selectivity.

With the above optimized conditions in hand, we next investigated the substrate scope and selectivity of the OBSC reaction (Table 2). The best results were obtained when an electron-deficient aryl bromide reacted with an electron-rich one; on the contrary, the combination appears to be less favourable when each aryl partner contains an electron-donating group (for example, **1e**). These results reveal that the more electron-rich an aryl bromide is, the more easily it is borylated; the more electron-deficient the other aryl bromide is, the better it works with the boronic acid intermediate.^{2*a*,21} As a result, this method uniquely favours the cross-coupling pathway, and indeed the homo-coupling was observed in no case. *Ortho*-substitution was well tolerated in this study, yielding the mono- or di-*ortho*-substituted product (**1f**, **1g** or **1h**). Moreover, the mono-arylation



^{*a*} Reaction conditions: two bromides (1 mmol each), $B_2(OH)_4$ (1.2 mmol), Na_2PdCl_4 (1 mol%), PCy_3HBF_4 (4 mol%), NaOH (2.5 mmol) and SDS (10 mol%). Na_2PdCl_4 and PCy_3HBF_4 in 2.5 ml water were preheated at 60 °C for 0.5 h before addition of substrates and 2.5 ml water and heated at 100 °C for 12 h. ^{*b*} Isolated yields. ^{*c*} 2-BrPhCN and 4-BrPhCH_3 (20 mmol each), $B_2(OH)_4$ (22 mmol), Na_2PdCl_4 (2 mol%), Pcy_3HBF_4 (8 mol%), NaOH (50 mmol) and SDS (10 mol%) in 80 ml water were heated at 100 °C for 12 h.

of 1,4-dibromobenzene led to 84% yield of 1i, providing a handle for further functionalization. In addition, this protocol proved to be suitable for scale-up. For example, the synthesis of a known intermediate of Santan^{17a} (1g) could be easily performed on a 20 mmol scale, the yield was almost identical to that obtained on a 1 mmol scale.

Although an array of cross-couplings has been developed to access heterobiaryls in water,²² novel methods in an economical and practical manner from accessible heteroaryl halides remain an important and challenging goal. As such, our investigation continued with the OBSC of heteroaryl bromides. As shown in Table 3, a variety of heteroaryl substrates reacted smoothly under the present conditions. For example, 2-bromobenzofuran underwent the OBSC to give **2k** in a 53% yield. Furthermore, N- and S-containing heterocycles such as an indole, pyridines, quino-lines and thiophenes all afforded the desired products (**2a–j**) in good yields, even in the presence of base-sensitive functional groups like –CHO, –COMe, and –COOEt.

This OBSC reaction exhibited high regio-selectivity. As shown in Scheme 2, the mono-arylation of 2,5-dibromopyridine was found to occur selectively at the C2 position. In comparison,



^{*a*} Reaction conditions: two bromides (1 mmol each), $B_2(OH)_4$ (1.2 mmol), Na_2PdCl_4 (2 mol%), PCy_3HBF_4 (8 mol%), NaOH (2.5 mmol) and SDS (10 mol%). Na_2PdCl_4 and PCy_3HBF_4 in 2.5 ml water were preheated at 60 °C for 0.5 h before addition of substrates and 2.5 ml water and heated at 100 °C for 12 h. ^{*b*} Isolated yields. ^{*c*} The reaction was performed for 10 h.





Scheme 3 The unsymmetrical diarylation of 3,5-dibromopyidine in a one-pot manner. Reaction conditions: (1) Na₂PdCl₄ (2 mol%) and PCy₃HBF₄ (8 mol%) in 2.5 ml water were preheated at 60 °C for 0.5 h. Then, 3,5-dibromopyridine, 2-bromotoluene (1 mmol each), B₂(OH)₄ (1.2 mmol), NaOH (2.5 mmol), SDS (10 mol%) and 2.5 ml water were added and heated for 12 h at 100 °C; (2) 3-bromothiophene (1 mmol), B₂(OH)₄ (1.2 mmol), NaOH (2.5 mmol) were added and heated for 12 h at 100 °C.

although 3-bromothiophene as a coupling partner is more reactive than 4-bromotoluene, its coupling yield of **3a** was lower than that of **3b** due to the easier formation of the diarylated product.

Given that the unsymmetrical diarylation of symmetrical dihalo aryls in a one-pot manner has remained poorly addressed to date,²³ especially uncovered in water, we preferred to use this OBSC method to iteratively diarylate 3,5-dibromopyridine in pure water, based on which it was successfully mono-arylated to give the product **2e** in a 77% yield. As described in Scheme 3, such a reaction was achieved by performing two sequential OBSCs in a one-pot manner, which led to isolation of the dicoupled product **4a** in a 54% overall yield.

In summary, we have developed the direct cross-coupling of two different aryl bromides by Na_2PdCl_4 catalysis mediated by $B_2(OH)_4$. Compared with reported Pd-catalysed biaryl crosscouplings, the advantages of this method include (1) the use of pure water as the solvent leading to a greener protocol; (2) being a simpler and more practical one-step procedure which has broad substrate scope and functional group compatibility (including electron-donating and moderate electron-deficient groups); (3) having complete cross-coupling selectivity which is achieved without an excess of either substrate; and (4) favouring the use of an inexpensive and atom-economical boron source and overcoming the use of an expensive and synthetically challenging ligand. These features will render it a useful tool to facilitate and simplify a variety of biaryl systems and more complex natural products.

Conflicts of interest

There are no conflicts to declare.

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