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Direct C-2 Arylation of 7-Azaindoles: Chemoselective Access to Multiarylated Derivatives

Prakash Kannaboina,[†,‡] K. Anilkumar,[†,‡] S. Aravinda,[‡] Ram A. Vishwakarma,[‡] and Parthasarathi Das*[,†,‡]

Academy of Scientific and Innovative Research (AcSIR) and Medicinal Chemistry Division, Indian Institute of Integrative Medicine(CSIR), Jammu 180001, India

partha@iiim.ac.in

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ABSTRACT

Pd-catalyzed direct C−H arylation of *N*-methyl-7-azaindole at the C-2 position by diverse arylboronic acids was achieved at room temperature. The method is general and was applied in chemoselective synthesis of multiarylated 7-azaindole derivatives bearing three different aryl groups at the 2, 3, and 5 positions.

The azaindole ring system is a key structural unit present in a number of therapeutically important molecules.[1,2] Owing to the presence of an additional nitrogen atom in the 7-azaindole ring system, when compared to indole, this structural moiety possesses an advantage to act as a hydrogen-bond donor as well as an acceptor framework, a property that has been successfully utilized in drug discovery.[3−5] Marketed anticancer drug vemurafenib (PLX-4032)[6] and many other kinase inhibitors (Figure 1) contain a *C*-arylated 7-azaindole moiety. A structure−activity relationship (SAR) study reveals that *C*-arylation in both the azole and azine ring plays a significant role in kinase inhibition.[7] Therefore, selective *C*-arylations of the 7-azaindole nucleus in a controlled manner will provide an

[†] Academy of Scientific and Innovative Research (AcSIR).
[‡] Medicinal Chemistry Division.

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efficient synthetic tool to modulate selective or sequential arylations at various positions of 7-azaindole that could be interesting from the point of view of medicinal chemistry.

Direct *C*-arylation of heteroarenes by C−H bond activation with the aid of transition metals is a practical alternative to conventional cross-coupling reactions.[8] In this context, direct C-2 arylation of indole has been studied where aryl halides,[9] arylboronic acids,[10] aryltrifluoroborate salt,[11] and [Ph-I-Ph]BF_4[12] were used as coupling partners, but selective C-2 arylation of 7-azaindole has been less explored.[13,14]

Sames[9a] and Fagnou[13] reported independently the Pd-catalyzed C-2 arylation of 7-azaindole by using aryl iodides as coupling partners at elevated temperature.

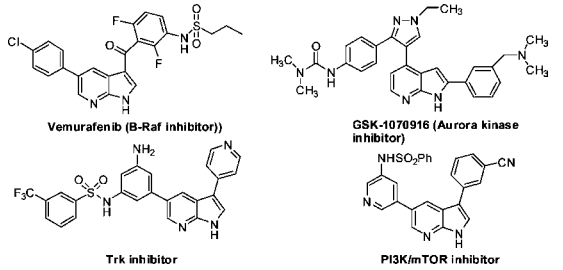
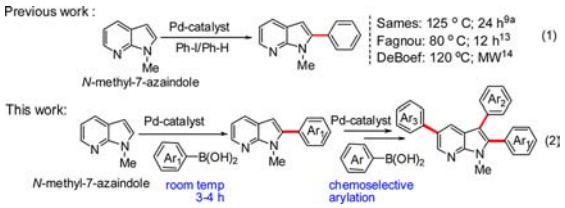


Figure 1. *C*-Arylated 7-azaindoles as biological target.

DeBoef has reported Pd-catalyzed oxidative arylation of 1-SEM-7-azaindole in an excess of benzene under microwave irradiation at 120 °C, which gave a mixture of 2-phenyl-7-azaindole and 3-phenyl-7-azaindole in an 8:1 ratio.[14] Therefore, a mild methodology for the direct arylation at C-2 position is highly desirable (Scheme 1, eq 1). Herein, we wish to report a Pd-catalyzed direct C-2 arylation of *N*-methyl-7-azaindole that uses boronic acids as coupling partners at room temperature and $Na_2S_2O_8$ as the terminal oxidant (Scheme 1, eq 2). Further, we demonstrate its utility in the synthesis of multiarylated 7-azaindole derivatives.[15]

Our initial efforts toward direct coupling of the (sp^2) C−H bond of *N*-methyl-7-azaindole (**1a**) at the C-2 position with phenylboronic acid used various terminal oxidants (Table 1, entries 1−11). We first examined the oxidant molecular oxygen, as described by Shi and co-workers in the case of indole.[10a] We observed formation of the desired product in 30% yield when AcOH was used as a solvent and $Pd(OAc)_2$ (5 mol %) as a catalyst (Table 1, entry 1). Use of air or $Cu(OAc)_2$ as the oxidant did not provide any improvement in yield (Table 1, entries 2 and 3). A poor yield (30%) was observed in the case of *tert*-butyl hydroperoxide (TBHP) (Table 1 entry 4), while $PhI(OAc)_2$, 1,4-benzoquinone (BQ), *m*-CPBA, Ag_2O, and H_2O_2 (Table 1, entries 5−9) were ineffective. Among the peroxydisulfate salts, $Na_2S_2O_8$ showed an obvious effect on the coupling reaction and **2a** was synthesized in good isolated yield (60%) (Table 1, entry 11). A change of solvent from AcOH to PivOH or TFA resulted in poor yield (Table 1, entries 12 and 13). An examination of different Pd sources was also carried out. We found that $Pd(TFA)_2$, $Pd_2(dba)_3$, and $PdCl_2$ failed to promote the cross-coupling reaction (Table 1, entries 14−16). In order to optimize the reaction conditions further, we performed the cross-coupling in the presence of a ligand (see the Supporting Information). Among several phosphine ligands examined with $Pd(OAc)_2$, it was found that a protocol which employed PPh_3 (10 mol %) provided the coupled product in 75% isolated yield (Table 1, entry 17). However, the reaction was sluggish under an N_2 atmosphere (Table 1, entry 20).

Scheme 1. Pd-Catalyzed C-2-Selective Arylation



We have also investigated the scope of *N*-substitution on the azaindole nucleus using the optimized procedure as depicted in Table 1. Only *N*-alkyl-substituted azaindoles proved to be suitable substrates, *N*-phenyl, *N*-sulfonyl or *N*-acetyl azaindoles remained inert under these cross-coupling conditions (see the Supporting Information).

With the optimized reaction conditions in hand, the scope of the reaction with respect to boronic acids was investigated and the results are summarized in Scheme 2. The reaction can be performed with a wide range of boronic acids. For example, with *N*-methyl-7-azaindole, C-2 arylated products were obtained in good yields (70−85%) in the case of both electron-rich (**2b**−**d**,**g**−**h**) and electron-deficient (**2e**−**f**) phenylboronic acids. The efficiency of arylation of *N*-methyl-7-azaindole was not affected by steric hindrance, as *ortho*-substituted phenylboronic acid afforded the desired product (**2h**) in good yield (70%). Disubstituted boronic acids were also tested under these conditions and the desired products were obtained in good yield (**2i**,**j**, 70%). Bicyclic 2-napthylboronic acid also readily participated in direct arylation to furnish the expected product in 80% isolated yield (**2k**). To enhance the substrate scope, we carried out the

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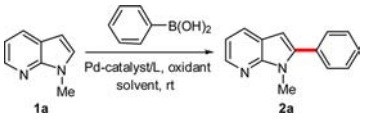
Table 1. Optimization Studies on C-2 Arylation of *N*-Methyl-7-azaindole[a]



entry	catalyst mol (5%)	oxidant	solvent	time (h)	yield[b] (%)
1	$Pd(OAc)_2$	O_2	AcOH	12	30
2	$Pd(OAc)_2$	air	AcOH	12	30
3	$Pd(OAc)_2$	$Cu(OAc)_2$	AcOH	12	40
4	$Pd(OAc)_2$	TBHP	AcOH	12	30
5	$Pd(OAc)_2$	$PhI(OAc)_2$	AcOH	12	10
6	$Pd(OAc)_2$	BQ	AcOH	12	10
7	$Pd(OAc)_2$	*m*-CPBA	AcOH	12	n.r.
8	$Pd(OAc)_2$	Ag_2O	AcOH	12	15
9	$Pd(OAc)_2$	H_2O_2	AcOH	12	n.r.
10	$Pd(OAc)_2$	$K_2S_2O_8$	AcOH	8	40
11	$Pd(OAc)_2$	$Na_2S_2O_8$	AcOH	6	70 (60)[c]
12	$Pd(OAc)_2$	$Na_2S_2O_8$	PivOH	6	10
13	$Pd(OAc)_2$	$Na_2S_2O_8$	TFA	6	10
14	$Pd(TFA)_2$	$Na_2S_2O_8$	AcOH	12	trace
15	Pd_2dba_3	$Na_2S_2O_8$	AcOH	12	n.r.
16	$PdCl_2$	$Na_2S_2O_8$	AcOH	12	n.r.
17[d]	**$Pd(OAc)_2$**	**$Na_2S_2O_8$**	**AcOH**	**4**	**85 (75)**[c]
18[e]	$Pd(OAc)_2$	$Na_2S_2O_8$	AcOH	6	40
19[f]	$Pd(OAc)_2$	$Na_2S_2O_8$	AcOH	6	45
20[g]	$Pd(OAc)_2$	$Na_2S_2O_8$	AcOH	24	10

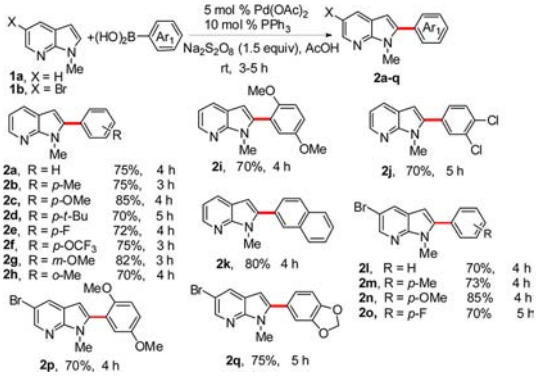
[a] Reaction conditions: *N*-methyl-7-azaindole (1.0 equiv), phenylboronic acid (1.0 equiv), $Pd(OAc)_2$ (5 mol %), $Na_2S_2O_8$ (1.5 equiv), AcOH (1 mL), rt, air. [b] GC yield. [c] isolated yield. [d] 10 mol % of PPh_3 was used. [e] 10 mol % of $(p\text{-Tol})_3P$ was used. [f] 10 mol % of X-phos was used. [g] Reaction performed under N_2 atmosphere. n.r. = no reaction.

cross-coupling process with 5-bromo-substituted *N*-methyl-7-azaindole, and to our delight, bromo derivatives were tolerated under these catalytic conditions (**2l**–**q**). Here also the electronic nature of the boronic acid did not affect the course of the reaction, as the *C*-arylated products were isolated in high yield (70–85%). Bicyclic boronic acid benzo[*d*][1,3]dioxol-5-ylboronic acid also gave the cross-coupled product (**2q**) in 75% yield.

Having optimized the C-2 arylations, we focused on exploring conditions that would enable us to effect multi arylation of 7-azaindole. Very few reports are available for cross-coupling reaction at the C-3 postion of 7-azaindole.[7c,e] Surprisingly, C-3 arylation has never been explored in the presence of the C-2 arylated 7-azaindole system. Therefore, finding generalized cross-coupling conditions for C-3 arylation would be very useful from a synthetic standpoint. Our initial efforts were unsuccessful. Therefore, we performed the NIS-mediated iodination on *N*-methyl-2-aryl-7-azaindoles, and the 3-iodo derivatives were isolated in high yields (80–85%) (Scheme 3).

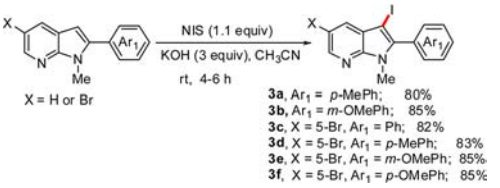
We used 3-iodo-*N*-methyl-2-*p*-tolyl-7-azaindole (**3a**) as a test substrate for further work in the presence of $Pd(OAc)_2$ (5 mol %), and Na_2CO_3 as a base in 1,4-dioxane/H_2O (3:1) at 100 °C. No product was observed even after 12 h (Table 2, entry 1). When we performed the reaction in

Scheme 2. C-2 Arylation of *N*-Methyl 7-Azaindoles[a]



[a] Reaction conditions: *N*-methyl-7-azaindole (1 equiv), boronic acid (1.2 equiv), $Pd(OAc)_2$ (5 mol %), PPh_3 (10 mol %), $Na_2S_2O_8$ (1.5 equiv), AcOH (2 mL), rt, air, 3–5 h.

Scheme 3. C-3 Iodination of *N*-Methyl-2-aryl-7-azaindoles[a]



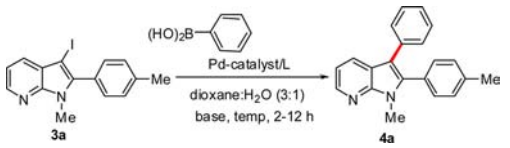
[a] Reaction conditions: *N*-methyl-2-aryl-7-azaindole (1 equiv), NIS (1.1 equiv), KOH (3 equiv), CH_3CN (1 mL), rt.

combination with dppf (5 mol %) as a ligand, **4a** was isolated in 30% yield (Table 2, entry 2). Several bases were examined along with variations in time and temperature (Table 2, entries 3–6). As depicted in Table 2 when Cs_2CO_3 was used with $Pd(OAc)_2$ at 60 °C in 1,4-dioxane/H_2O (3:1) the isolated yield was 85% (Table 2, entry 5). Further screening of different ligand systems such as PPh_3 (Table 2, entry 7) and X-phos (Table 2, entry 8) showed these to be less effective. Use of different Pd-sources (Table 2, entries 9 and 10) resulted in poor yield. Lower yields were obtained when we changed the solvent system to DMF/H_2O (Table 2, entry 11). No product formation was observed when 1,4-dioxane was used as the only solvent (Table 2, entry 12). This indicated that the presence of water as solvent was crucial for coupling.

With the optimized protocol in hand the scope of the cross-coupling reaction was explored by using various arylboronic acids with different 3-iodo-1-methyl-2-aryl-7-azaindoles (Scheme 4). For example electron-donating and electron–withdrawing boronic acids all afforded 3-arylated derivatives **4c** and **4b**–**d**, respectively, in good yields (75–82%).

In addition to substituted phenylboronic acids, benzo[*d*][1,3]dioxol-5-ylboronic acid was also tested under these conditions (**4e**). Interestingly, the presence aryl substitution at C-2 position did not play any significant role in cross-coupling at the C-3 position. In order to synthesize

Table 2. Optimization Studies for C-3 Arylation[a]



entry	catalyst	L	base	temp (° C)	time (h)	yield[b] (%)
1	$Pd(OAc)_2$		Na_2CO_3	100	12	n.r.
2	$Pd(OAc)_2$	dppf	Na_2CO_3	100	6	30
3	$Pd(OAc)_2$	dppf	K_2CO_3	100	6	50
4	$Pd(OAc)_2$	dppf	Cs_2CO_3	100	2	75
5	**$Pd(OAc)_2$**	**dppf**	**Cs_2CO_3**	**60**	**2**	**85**
6	$Pd(OAc)_2$	dppf	K_3PO_4	60	6	40
7	$Pd(OAc)_2$	PPh_3	Cs_2CO_3	60	6	10
8	$Pd(OAc)_2$	X-phos	Cs_2CO_3	60	6	35
9	$Pd(PPh_3)_4$	dppf	Cs_2CO_3	60	12	10
10	$Pd(dppf)Cl_2$	dppf	Cs_2CO_3	60	12	20
11[d]	$Pd(OAc)_2$	dppf	Cs_2CO_3	60	6	45
12[e]	$Pd(OAc)_2$	dppf	Cs_2CO_3	60	6	n.r.

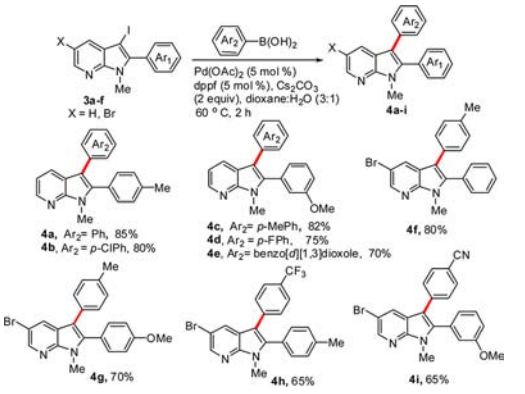
[a] Reaction conditions: **3a** (1.0 equiv), phenylboronic acid (1.0 equiv) $Pd(OAc)_2$ (5 mol %), dppf (5 mol %), Cs_2CO_3 (2 equiv), 1,4-dioxane/H_2O (2 mL; 3:1 mixture), 60 °C, air. [b] Isolated yields. [d] Reaction performed in DMF/H_2O (3:1). [e] Only 1,4-dioxane was used as solvent. n.r. = no reaction.

the precursor for C-5 cross-coupling, we performed C-3 arylation of the 5-bromo derivatives of 3-iodo-*N*-methyl-2-aryl-7-azaindole under these optimized conditions, and to our delight, the reaction was completely chemoselective. *p*-Tolylboronic acid gave higher yields (70−80%) (**4f**,**g**) compared to electron-poor phenylboronic acids (65%) (**4h**,**i**).

Having accomplished C-2 and C-3 arylation, we then focused our attention on optimizing the cross-coupling for the C-5 position. Experimentation with the combination of $Pd(OAc)_2$ and dppf showed that the reaction with 5 mol % of $Pd(OAc)_2$, 10 mol % of dppf, and 3.0 equiv of Cs_2CO_3 in 1,4-dioxane and water (3:1) at 80 °C for 6−8 h afforded the C-5-arylated product in the best yields. Using these conditions, we evaluated the scope of C-5 arylation with various boronic acids, and the results are summarized in Scheme 5. Good yields (60−72%) were observed with the electronic nature of the boronic acids not playing much of a role in the arylation process (**5a**−**f**). Bicyclicboronic acids were also investigated, in case of 2-napthylboronic acid the yield is 63% (**5c**), whereas benzo[*d*][1,3]dioxol-5-ylboronic acid resulted in 60% (**5e**) yield. In this context, X-ray crystal structure analysis of 5-(2,4-difluorophenyl)-1-methyl-2-phenyl-3-*p*-tolyl-1*H*-pyrrolo[2,3-*b*]pyridine (**5b**) confirmed its structure (see the Supporting Information).

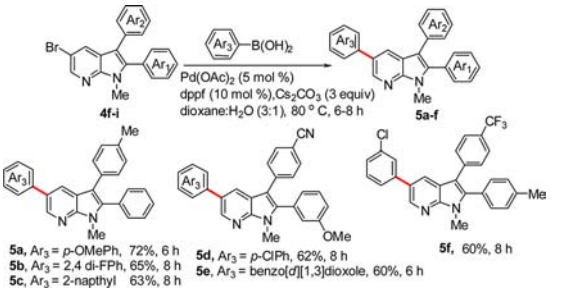
In summary, we have developed a practical Pd-catalyzed method for the C-2 arylation of *N*-methyl-7-azaindole at room temperature, where PPh_3 was used as a ligand and $Na_2S_2O_8$ as an oxidant in an acidic medium. Gereral methods for the chemoselective *C*-arylation, at C-3 and C-5 postions have also been discovered. This Pd-catalyzed

Scheme 4. Pd-Catalyzed C-3 Arylation of 3-Iodo-*N*-methyl-2-aryl-7-azaindoles[a]



[a] Reaction conditions: 3-iodo-*N*-methyl-2-aryl-7-azaindole (1 equiv), boronic acid (1.1 equiv), $Pd(OAc)_2$ (5 mol %), dppf (5 mol %), Cs_2CO_3 (2 equiv), 1,4-dioxane/H_2O (1 mL, 3:1 mixture), 60 °C, 2 h.

Scheme 5. Pd-Catalyzed C-5 Arylation of 5-Bromo-*N*-methyl-2,3-diaryl-7-azaindoles[a]



[a] Reaction conditions: 5-bromo-*N*-methyl-2,3-diaryl-7-azaindole (1 equiv), boronic acid (1.1 equiv), $Pd(OAc)_2$ (5 mol %), dppf (10 mol %), Cs_2CO_3 (3 equiv), 1,4-dioxane/H_2O (1 mL, 3:1 mixture), 80 °C, 6−8 h.

cross-coupling condition is ligand, base, and solvent specific. These selective cross-coupling conditions have provided the first examples of 7-azaindole derivatives bearing different aryl units at the 2, 3, and 5 positions. These reaction conditions are generally versatile. Thus they can be further exploited in the synthesis of library compounds for drug discovery and for materials chemistry applications.

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Supporting Information Available. Experimental details and all spectral data. This material is available free of charge via Internet at http://pubs.acs.org

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