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Palladium(II)-Catalyzed *ortho*-Arylation of Aromatic Alcohols with a Readily Attachable and Cleavable Molecular Scaffold

Qiankun Li,[a] Brian J. Knight,[a] and Eric M. Ferreira*[a]

Abstract: A palladium(II)-catalyzed C–H arylation process of alcohols has been developed. The strategy utilizes a novel quinoline-based hemiacetal scaffold that can direct the selective C–H bond functionalization. This reaction provides a useful method to construct biaryl compounds of benzyl alcohols in good to excellent yields. The new molecular scaffold can be readily attached, removed, and recovered.

Transition metal (TM)-catalyzed selective C–H functionalization represents a versatile approach to construct the core structures in both natural products and pharmaceuticals.[1] Arylation via a C–H functionalization strategy can present a complementary approach to Pd^0-catalyzed cross couplings for biaryl synthesis.[2] A commonly employed strategy to achieve highly selective activations is to use substrates possessing a Lewis basic functional group that can coordinate the metal and direct the precise reaction of the otherwise unreactive C–H bond. Representative example substrate classes include pyridines, oxazolines, and amides,[1c,3] among several others. Easily attached and removed functional groups that can bestow the necessary Lewis basicity may allow this approach to be adapted to less commonly employed substrate classes. Alcohols are one such substrate family; these have been comparatively infrequent in C–H functionalization chemistry, likely due to their susceptibility toward oxidation and their relatively weaker coordination ability.[4] In 2010, Yu and co-workers disclosed a series of reports on Pd^{II}-catalyzed hydroxyl-directed C–H functionalization;[5] tertiary alcohols were necessary to achieve high yields, however. In contrast, primary and secondary alcohols tended to afford products in notably diminished yields.[6]

An alternative approach relating to those cases involves alcohol "scaffolding," using species that can be directly attached to and later removed from the alcohol functional group. Transformations reported by Hartwig,[7] Yu,[8] Tan,[9] Dong,[10] Zhao,[11] and us[12] demonstrated the feasibility of this concept in C–H functionalization. Specifically, a recent report from Zhao and coworkers[11b] demonstrated the Pd-catalyzed *ortho*-selective arylation to construct biaryls, using oximes as the surrogate species. As part of a program directed toward designing novel molecular directing groups for C–H functionalization,[12,13] we recently described the development of a practical scaffold for catalytic C–H functionalization (PyA - **Py**ridyl **A**cetal, Figure 1), which is readily attachable to alcohols, cleavable, and recoverable in excellent yields.[12] We had demonstrated the capacity of this scaffold to induce oxidative olefinations of benzylic, homo-, and bishomobenzylic alcohols. Herein, we disclose the expansion of this strategic concept toward arylation, requiring the development of a new molecular scaffold based on a quinoline-incorporated acetal (QuA - **Qu**inolinyl **A**cetal, Figure 1). We demonstrate its utility in *ortho*-selective C–H arylations of benzylic and homobenzylic alcohols. Of note, we highlight this scaffold has similar features to our earlier report in terms of attachability and cleavability, as well as show that this method can incorporate *ortho*-substituted arene coupling partners, illustrating complementarity to the earlier report.[11b]

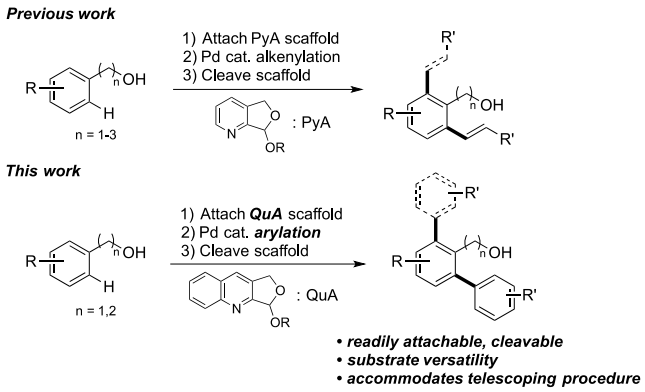

Figure 1. Pd(II)-catalyzed PyA/QuA-assisted C–H functionalization.

In preliminary attempts to expand our scaffold-directed chemistry to arylation, we first investigated the PyA scaffold-attached benzylic alcohol (**3**) as the substrate. The scaffold was readily attached from the parent alcohol (**1**) using hemiacetal **2** under dehydrative conditions (Scheme 1).[12] When the acetal was treated under ligand-accelerated Pd-catalyzed C–H arylation conditions developed by Yu and coworkers [PhBPin (3 equiv), $Pd(OAc)_2$ (10 mol %), Ac-Gly-OH (20 mol %), Ag_2CO_3 (1 equiv), CsF (2 equiv) in HFIP],[14] the desired product was observed in 11% yield (NMR), with 83% yield of unreacted starting material. Although we were encouraged by the proof-of-principle reactivity, the low yield demanded further attention. After a series of optimizations (see the Supporting Information for full details), 45% yield of arylated product could be obtained, as a 42:3 mixture of the *ortho*-monoarylated species (**5**) and a further diarylated species (**6**). This latter species presumably arises via a subsequent directed arylation of product **5** at the ortho position of the initially installed arene. A 38% yield of the recovered starting material was also observed. We hypothesized that this overall arylation process was somewhat hampered by the pairing of boronate species with the strongly Lewis basic pyridyl scaffold. Thus, we believed we needed to

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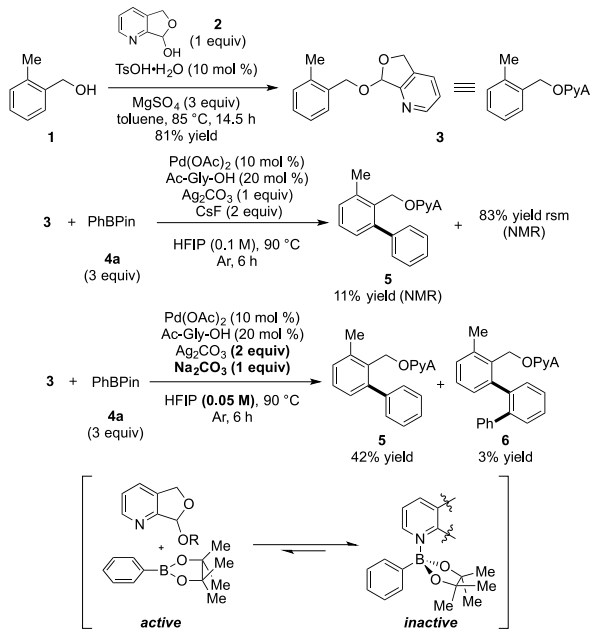
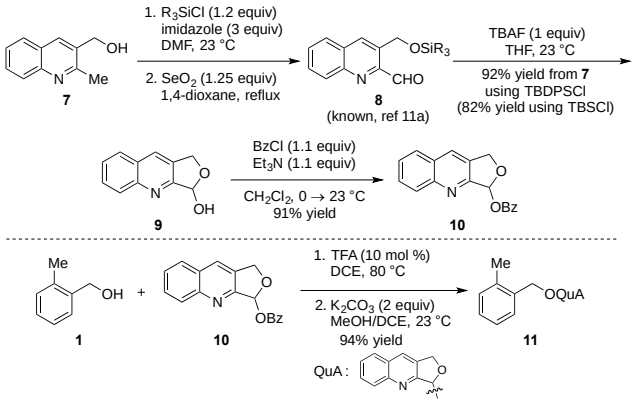
explore structural changes to our scaffold system in order to improve reactivity.



Scheme 1. Preliminary C–H arylation based on pyridyl acetal (PyA).

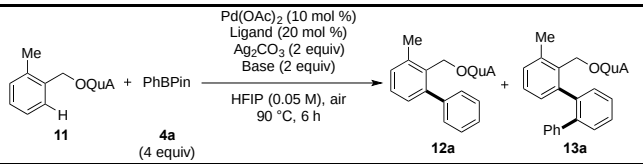
Toward this idea, we reasoned that a more tempered coordinating group may lead to improved reactivity. Quinolines have been widely used as directing groups for TM-catalyzed C–H functionalizations,[15] so we pursued an analogous scaffold based on this heterocycle. The synthesis of the scaffold is illustrated in Scheme 2. From known quinoline alcohol **7**,[16] a silylation/oxidation/desilylation sequence afforded hemiacetal **9** in excellent yield. TBSCl could be used instead of TBDPSCl for this sequence, although the yield of hemiacetal **9** in that case was slightly diminished.[17] Although we had used the hemiacetal functional group to attach the PyA moiety (e.g., Scheme 1), for the quinoline-based compound we observed an acetal dimerization that complicated purification. Ultimately, we found that benzoylation of hemiacetal **9** proceeded smoothly, and benzoate **10** could be readily attached to *o*-methyl benzyl alcohol under acidic conditions (TFA/DCE) to afford the **Qu**inolinyl-**A**cetal (QuA)-attached alcohol in 94% yield.[18]



Scheme 2. **Qu**inolinyl-**A**cetal (QuA) synthesis and scaffold attachment.

To our delight, when the QuA-attached benzyl alcohol **11** was subjected to the reaction, a significantly improved arylation (69/4% mono/di) was achieved (Table 1, entry 1), with an additional 24% of recovered starting material (rsm). Using 4 equiv of PhBPin gave a slightly improved reaction (entry 2). After reevaluating bases, 2 equiv of K_2CO_3 gave the best yield (75/14% mono/di with 6% rsm, entry 5). Aryltrifluoroborates were also reactive, albeit with lower activity than PhBPin (entry 8). Using benzoquinone or water has been shown in specific cases to facilitate transmetalation and/or reductive elimination;[19] unfortunately these additives did not improve the yield for this transformation (entries 9, 10). When amino acid based ligands were analyzed, *N*-acetylisoleucine (Ac-Ile-OH) was optimal, with a much improved mono- to diarylation ratio (entry 14).[20] The ligand Ac-*t*Leu-OH afforded an even better mono/di ratio, but lower yields overall (entry 15). Presumably, more bulky ligands can suppress the formation of the diarylated product, but they also lead to lower yields. Ultimately we chose entry 14 in Table 1 as the optimized conditions for further studies, providing the optimal balance of high reactivity and monoarylation selectivity.

Table 1. C–H arylation optimization with QuA-attached alcohol **11**.



Entry[j]	Base	Ligand	% Yield[a] (**12a** / **13a** / rsm)
1[b]	Na_2CO_3	Ac-Gly-OH	69 / 4 / 24
2	Na_2CO_3	Ac-Gly-OH	71 / 6 / 17
3	Na_3PO_4	Ac-Gly-OH	71 / 8 / 17
4	$KHCO_3$	Ac-Gly-OH	75 / 11 / 7
5	K_2CO_3	Ac-Gly-OH	75 / 14 / 6
6	K_3PO_4	Ac-Gly-OH	50 / 2 / 24
7	Cs_2CO_3	Ac-Gly-OH	75 / 12 / 9

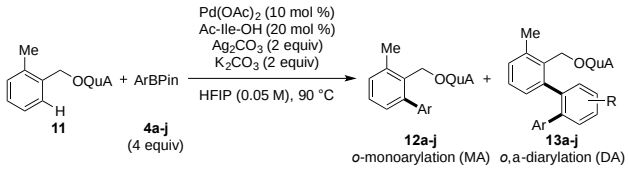
8[c]	K_2CO_3	Ac-Gly-OH	57 / 2 / 39
9[d]	K_2CO_3	Ac-Gly-OH	18 / 0 / 76
10[e]	K_2CO_3	Ac-Gly-OH	74 / 12 / 9
11	K_2CO_3	Ac-Val-OH	77 / 8 / 8
12	K_2CO_3	Ac-Ala-OH	70 / 6 / 16
13	K_2CO_3	Ac-Leu-OH	75 / 8 / 10
14	K_2CO_3	Ac-Ile-OH	84 / 5 / 7
15	K_2CO_3	Ac-*t*Leu-OH	76 / 1 / 17

[a] NMR yield with 1-octene as the standard. [b] 3 equiv of PhBPin used. [c] $PhBF_3K$ used instead of PhBPin. [d] 0.5 equiv benzoquinone was added. [e] 2 equiv H_2O was added.

Table 1. C–H arylation optimization with QuA-attached alcohol **11**.

The substrate scope was next explored (Table 2). We first tested the scope of arylboronic pinacol esters. Variously substituted Ar-BPins (with both electron-donating and electron-withdrawing substituted groups, such as OMe, CF_3, CO_2Me) were tolerated, affording the corresponding products in good to excellent yields (entries 2-7). Moreover, 2-naphthylboronic acid pinacol ester was also tolerated, giving the corresponding monoarylated product in 76% yield (entry 8). Most notably, arylboronic pinacol esters that were *ortho*-substituted were participatory in this reaction manifold, and the corresponding arylated products were isolated in good yields (entries 9, 10). *Ortho*-substituted aryl species were reported to be unreactive in Zhao's oxime-based system,[11b] demonstrating that our method has some complementarity to this prior art.

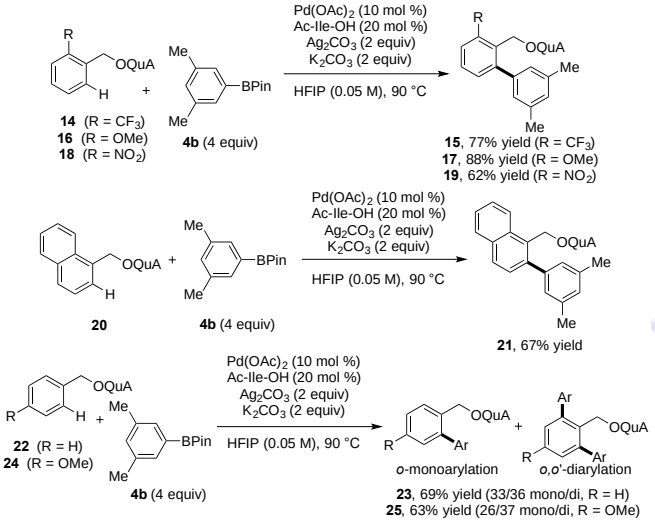
Table 2. C–H arylation - Variation of aryl boronate.



Entry	ArBPin	Yield (%)[a] MA	DA	rsm	Entry	ArBPin	Yield (%)[a] MA	DA	rsm
1	**4a**	82	7	6	6	**4f**	80	(4)	(16)
2	**4b**	84	0	(3)	7	**4g**	71	7	(9)
3	**4c**	76	(9)	(5)	8	**4h**	76	0	(8)
4	**4d**	72	(4)	6	9	**4i**	69	0	(4)
5	**4e**	71	6	(5)	10	**4j**	63	0	(14)

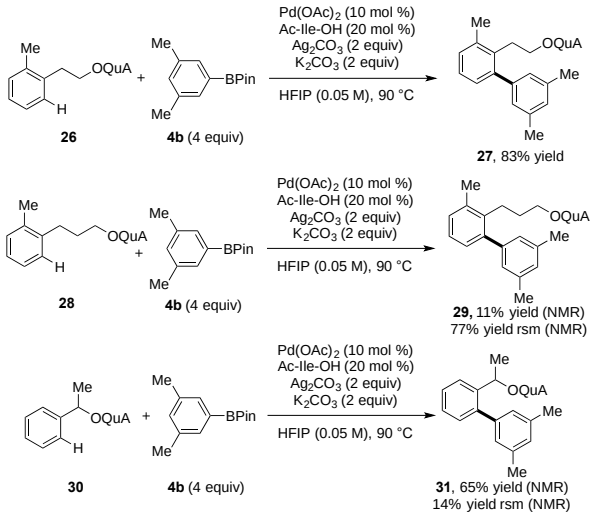
[a] Yields are as isolated mixtures of MA/DA/rsm components. Yields in parentheses are NMR yields based on integration of the crude product mixture. These components were removed in purification.

A range of scaffold-attached benzylic alcohol substrates were also evaluated (Scheme 3). The QuA scaffold was attached to these alcohols and those in Scheme 4 in 77-96% yield using the same conditions as described in Scheme 4. For *ortho*-substituted arenes, good yields of the biaryls were formed. For cases that were unsubstituted at the ortho position, both mono- and *ortho*-diarylated products were observed.



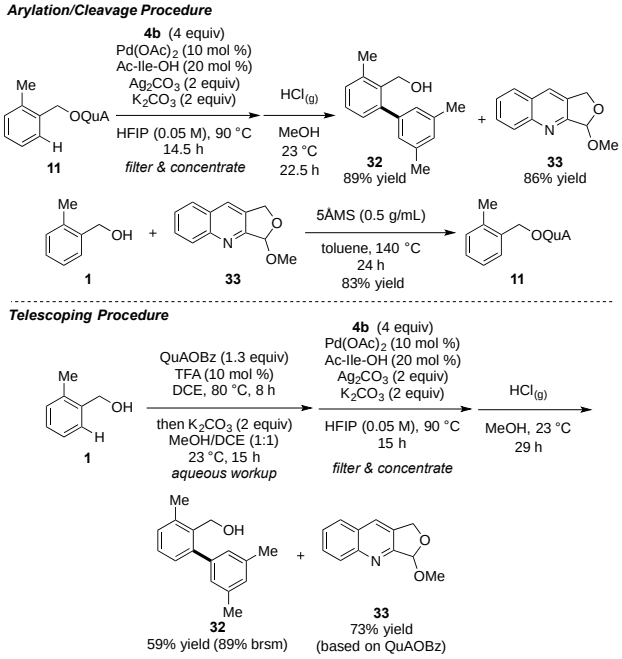
Scheme 3. Examination of benzylic alcohol scope.

In the aforementioned alkenylation process, we observed that our scaffold could be successfully extended to systems beyond primary benzylic alcohols. For arylation, this extension was also achievable, although the results in these cases were somewhat more modest (Scheme 4). A substrate derived from a homobenzylic alcohol could be coupled in this transformation, as product **27** was afforded in 83% yield.[21] A compound originating from a bishomobenzylic alcohol, however, showed low reactivity, with only 11% yield of the arylated species produced, and 77% yield of the recovered starting material. A secondary alcohol-based substrate was also reactive, with only mono-arylation observed.



Scheme 4. Arylation of non-primary benzylic alcohol-based substrates.

A significant attribute of our acetal-based scaffold is the ease of both attachment and cleavage,. A sequential procedure, with arylation followed by immediate direct scaffold cleavage was performed (Scheme 5). The two steps, with only one purification, afforded the biaryl alcohol in 89% yield. Furthermore, the methyl acetal-derived scaffold (**33**) was isolated in 86% yield, highlighting its recoverability. Although the benzoate derived precursor was generally preferable for attachment, this recovered acetal compound (**33**) could be readily reused (Scheme 5). Much like in our olefination studies, a telescoping procedure using our quinolinyl scaffold could be executed. Benzylic alcohol **1** was converted to the arylated alcohol (**32**) in 59% yield (89% yield brsm) without any intermediate purifications. The iterative procedure was higher yielding than the telescoping process (84% vs. 59%); the initial attachment afforded small amounts of quinoline-based impurities that hampered the arylation reactivity when telescoped. Nevertheless, the overall telescoping procedure can indeed be executed, and arylated alcohol **32** was obtained in reasonable yield.



Scheme 5. Sequential procedure, recovery, and telescoping.

In conclusion, we have developed a modified molecular scaffold that enables a practical strategy for the C-H arylation of benzylic and homobenzylic alcohols. The derivatization of the scaffold to incorporate a quinolinyl moiety proved pivotal to temper the Lewis basicity and enable boronic ester compatibility. The new scaffold is readily synthesized in just two steps from known starting materials.[22] The scaffold is readily attached and removed, and the biaryl compounds can be accessed in good to excellent yields. A range of both alcohol and boronic ester coupling partners could be tolerated, and an overall telescoping procedure is feasible with this technology. Further investigations using our scaffolding strategy in C–H functionalization are currently underway and will be reported in due course.

Experimental Section

Typical C–H arylation procedure: A suspension of acetal **11** (29.6 mg, 0.102 mmol), PhBPin (**4a**, 81.0 mg, 0.397 mmol), $Pd(OAc)_2$ (2.3 mg, 0.0103 mmol), *N*-acetylisoleucine (3.4 mg, 0.0197 mmol), Ag_2CO_3 (57.0 mg, 0.207 mmol), and K_2CO_3 (29.0 mg, 0.210 mmol) in hexafluoroisopropanol (2.00 mL) in a 2-dram vial with a PTFE-lined cap was heated at 90 °C and stirred for 6 h. The reaction was cooled to ambient temperature and filtered through a short pad of silica gel, eluting with diethyl ether (50 mL). The filtrate was concentrated by rotary evaporation, and the resulting residue was purified by flash column chromatography (10:1:1 hexanes/EtOAc/CH_2Cl_2 eluent) to afford arylation products **12a/13a** (35.6 mg, 89% yield (82:7 mono/di) + 6% recovered **11**, R_f = 0.40 in 5:1:1 hexanes/EtOAc/CH_2Cl_2) as a light yellow oil. **12a**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, *J* = 8.5 Hz, 1 H), 8.03 (s, 1 H), 7.83 (d, *J* = 8.2 Hz, 1 H), 7.76-7.68 (m, 1 H), 7.61-7.53 (m, 1 H), 7.52-7.41 (m, 4 H), 7.41-7.34 (m, 1 H), 7.25-7.17 (m, 2 H), 7.14 (dd, *J* = 7.2, 1.3 Hz, 1 H), 6.22 (s, 1 H), 5.34 (d, *J* = 13.2 Hz, 1 H), 5.20 (d, *J* = 13.2 Hz, 1 H), 4.90 (d, *J* = 9.8 Hz, 1 H), 4.68 (d, *J* = 9.8 Hz, 1 H), 2.59 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.7, 148.8, 143.9, 141.6, 139.5, 132.7, 130.7, 130.1, 129.8, 129.5, 128.9, 128.3, 128.1, 128.07, 128.04, 127.98, 127.2, 104.1, 70.2, 66.0, 19.9; IR (film) 1503, 1067, 1007, 910, 760 cm^{-1}; HRMS (ESI+) m/z calc'd for (M +H)$^+$ [$C_{25}H_{21}NO_2$+ H]$^+$: 368.1645, found 368.1646.

Acknowledgements

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Keywords: C–H functionalization • Arylation • C-C coupling • Palladium • Quinoline acetal

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[17] See the Supporting Information for details.

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[21] Biaryl compound **27** could be isolated in only ~92% purity, together with an unidentified byproduct.

[22] The aldehyde product from step 2 in the sequence in Scheme 2 (**8**, SiR_3 = TBDPS) is known. See ref 11a.

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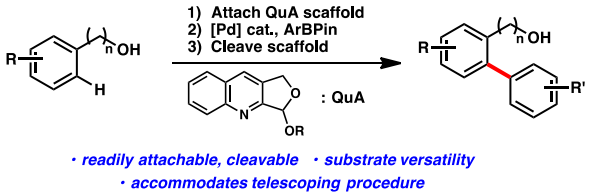
COMMUNICATION



Catalytic (QuA)rylation. A Pd^{II}-catalyzed C–H arylation process of alcohols has been developed. The strategy utilizes a novel quinoline-based hemiacetal scaffold that can direct the selective C–H bond functionalization. This reaction provides a useful method to construct biaryl compounds of benzyl alcohols in good to excellent yields. The new molecular scaffold can be readily attached, removed, and recovered.

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