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Suzuki coupling of aroyl-MIDA boronate esters – A preliminary report on scope and limitations



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ABSTRACT

Recent methodological reports for synthesizing acyl-MIDA boronate esters compel an investigation of their potential use as substrates in a standard Suzuki-Miyaura cross-coupling reaction. Here we report the production of benzophenones by C--C cross coupling between a benzoyl-MIDA boronate ester and a multitude of aryl bromide substrates in adequate yields following optimization under ambient conditions outside of a glove box. Under these standard conditions, none of several acyl-MIDA boronate esters (in an alkyl series) serves as a competent coupling partner. The substrate scope is also limited by the finding that the corresponding trifluoroborates of both acyl- and aroyltrifluroborates are not suitable substrates. For reasons of availability and synthetic difficulty in procuring other aroyl-MIDA boronates, this preliminary study examines the reactivity of benzoyl-MIDA boronate with several aryl bromide substrates.

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Introduction

Organoboronates are key substrates in metal-catalyzed cross coupling reactions, most notably the Suzuki-Miyaura reaction [1]. Since its inception in 1979, numerous innovations in Suzuki-Miyaura cross couplings [2,3] have expanded the applications of this now-classic reaction to include a range of catalytic metals, ligands and reaction sub-classes [4]. Over the past 4 decades, an extensive array of organoboronates that include alkyl, aryl, heteroaryl, vinyl, and alkynyl boronates has been investigated as substrates in various cross coupling reactions where the coupling partner is typically an organobromide/iodide, but which may now include sulfonate esters, nitrated organics, diazonium salts [5], N-sulfonyl-aziridines, N-alkylpyridinium salts, and cyclopropanes [6].

Within this reaction manifold, ketones can be synthesized in coupling reactions wherein organoboronates are caused to react with electrophiles that include aroyl halides [7–10], aroyl- and acyl anhydrides [11-16], and aroyl esters [17] as reviewed [18]. The substrate scope of acyl donors now extends to acylimides [19,20], thioesters, [21–23] and amides [24–29].

In contrast to conventional methods such as Friedel-Crafts acylation or the reaction of various acyl donors with standard organometallic substrates e.g. Grignards and organo-zinc reagents

* Corresponding author. E-mail address: dperrin@chem.ubc.ca (D.M. Perrin). [30-32], the aforementioned couplings have the advantage of catalytic action by transition metals (e.g. Pd, Ni, In) under milder conditions that in turn improve functional group tolerance. In these novel reactions, catalytic transition metals may play different roles. Although typically, metals oxidatively insert into the acyl donor to give an acyl-metal species, then transmetalate with the boronic acid, and ultimately cross-couple via reductive elimination, such does not necessarily hold in all cases; as a counter-example, in a Cu-catalyzed ketonization of thioesters, Cu promotes desulfitative coupling via Lewis-acid coordination, but without the standard redox cycle of oxidative insertion or reductive elimination [21,22]. Irrespective of the precise role of the catalytic metal, these couplings all involve an electrophilic carbonyl attached to a leaving group that is necessarily more electronegative than the carbon of the acyl donor. In these cases, the organoboronate partner may serve as a masked carbon nucleophile (Scheme 1a-g).

In stark contrast to all other acyl/aroyl donors, where the leaving group e.g. NHR, OR, SR, imide, halogen etc., is more electronegative than carbon, acyl- and aroyl boronates, arguably are "umpoled" as they present reversed electronegativity. In the case of nucleophilic acyl substitution, the boronate/borate is not expected to depart either as a boryl anion or a borane (upon protonation), and such a departure would be kinetically if not also thermodynamically unfavorable as it would generally defy conventional notions of nucleofugacity in nucleophilic acyl substitution reactions. Nevertheless, in a metal-mediated acylation reaction, one could appreciate that acyl boronates might still undergo

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Scheme 1. Previous reported synthetic methods for ketone synthesis (top) and this work (bottom).



Scheme 2. Hypothetical mechanism for aroyl-MIDA-boronate ester coupling based simply on standard Suzuki coupling.

transmetalation, which could then undergo reductive elimination resulting in cross-coupling as shown putatively in Scheme 2.

As far as organoboronates are concerned, acyl- and aroyl boronates are relative newcomers to the realm of organoboronates. First discovered by Nozaki [33,34] and made practicable by Molander [35,36], several recent reports have increased synthetic accessibility to the MIDA-acyl- and MIDA-aroyl boronate esters as well as the corresponding trifluoroborate salts [37–51] and expanded scope as recently reviewed [52]. Yet to date, the reactivity of acyl/aroyl-boronates in a Suzuki-Miyaura type coupling reaction remains altogether unknown, a void that this preliminary report seeks to fill as we document the attempted Suzuki-Miyaura coupling of MIDA boronates to various organohalides. In this first such report of its kind, we identify major limitations in substrate scope and briefly discuss the potential challenges with this reaction. This reported success in certain cases and associated limitations are worthy of note in guiding progress in this field.

Results and discussion

As MIDA-esters show greater solubility in organic solvents than the corresponding trifluoroborates, and by certain measures are considered to be more amenable to Suzuki-Miyaura coupling [53,54], we focused on MIDA esters. In addition, in our hands the corresponding aroyl/acyltrifluoroborates did not couple under any conditions, which discouraged pursuit of otherwise commercially available aroyltrifluoroborates that are easily synthesized from aryl-Mg salts that routes that typically deliver the MIDA esters [39] (see discussion). Hence, to begin, we explored conditions starting with benzoyl-MIDA boronate **1** that we had previously prepared from phenylvinylboronate MIDA ester and report yields under conditions noted in Table 1.

Initially, we hypothesized that benzoyl MIDA boronate ester would react in accordance with standard Suzuki coupling procedures starting with Pd(II) and a bulky phosphine ligand (XPhos) (entry 2). We favored bulky phosphine ligands because of enhanced rate of elimination and stabilization of Pd-arene interaction (entry 3). Additionally, these types of ligands minimize adventitious oxidation by O₂. We found that our target molecule **3a** was formed in 14% isolated yield from a Pd(OAc)₂/XPhos-catalyzed reaction (entry 2) starting with 1 and 4-bromobenzene. Although low-yielding, this provided a benchmark for screening a multitude of conditions to improve yields. Hence, we surveyed different types of palladium sources (10 mol%) in the model reaction between benzoyl-MIDA boronate ester and 4-bromobenzene in the presence of different bases (3-6 eq.) under open air conditions and in a multitude of solvent conditions. From our results, Pd(0) sources did not support coupling and only Pd(II) precatalysts afforded the desired product. In our hands, the best results were obtained with Pd(dppf)*CH₂Cl₂ (20 mol%) in the absence of any bulky phosphine ligand. Yields increased with higher catalyst loading with a 10% increase in yield observed when another 10 mol% of catalyst was added. Of the commercially available palladium catalysts, PdCl₂(dppf)*CH₂Cl₂ gave the most promising results.

A screen of common bases showed that only potassium carbonate (K_2CO_3) improved yields, while the others were less than optimal. Organic bases such as triethylamine and DBU did not yield any desired product (**entry 9–10**). Use of a biphasic mixture of 5:1 DCM/H₂O also improved yields. Our rationale for this observation could be due to the fact that formation of the benzoylboronic acid depends on aqueous solvolysis of the MIDA ester while the actual coupling may take place in the organic phase.

Use of higher-boiling solvents e.g. 1,2-dichloroethane or chloroform gave little to no product in comparison to CH_2Cl_2 for reasons that are not readily intuited. In our control studies, we show that

Table 1

Optimization of reaction conditions.



Entry	Cat. (x mol%)/ligand	Base	Additive	Solvent	Temperature (°C)	Yield (%)
1	Pd (10)	K ₂ CO ₃	-	acetone-water	90	_
2	Pd(OAc) ₂ (10)/XPhos	K ₂ CO ₃	-	toluene-water	90	14
3	$Pd(OAc)_2$ (10)/PCy ₃	K ₂ CO ₃	-	toluene-water	90	2
4	Pd(OAc) ₂ (10)/TTBP	K ₂ CO ₃	-	toluene-water	90	5
5	$PdCl_2(dppf)^*CH_2Cl_2(10)$	K ₂ CO ₃	-	THF-water	RT	32
6	$PdCl_2(dppf)^*CH_2Cl_2(10)$	Na ₂ CO ₃	-	THF-water	RT	-
7	$PdCl_2(dppf)^*CH_2Cl_2(10)$	Cs ₂ CO ₃	-	THF-water	RT	-
8	PdCl ₂ (dppf)*CH ₂ Cl ₂ (10)	CuCO ₃ *Cu(OH) ₂	-	THF-water	RT	-
9	$PdCl_2(dppf)^*CH_2Cl_2(10)$	NEt ₃	-	THF-water	90	-
10	$PdCl_2(dppf)^*CH_2Cl_2(10)$	DBU	-	THF-water	90	-
11	$PdCl_2(dppf)^*CH_2Cl_2(20)$	K ₂ CO ₃	-	THF-water	60	36
12	PdCl ₂ (dppf)*CH ₂ Cl ₂ (20)	K ₂ CO ₃	-	DCM-water	60	68
13	$PdCl_2(dppf)^*CH_2Cl_2$ (10)	K ₂ CO ₃	-	DCM-water	60	52
14	$PdCl_2(dppf)^*CH_2Cl_2(20)$	K ₂ CO ₃	KF ^a	DCM-water	60	24
15	$PdCl_2(dppf)^*CH_2Cl_2(20)$	K ₂ CO ₃	Cu(OAc)2 ^b	DCM-water	60	25
16	-	K ₂ CO ₃	-	DCM-water	60	-
17	$PdCl_2(dppf)^*CH_2Cl_2$ (20)	-	-	DCM-water	60	-

All reactions were carried out with 0.1 mmol of 1 (1 equiv), 0.11 mmol of 2a (1.1 equiv), Base (6 equiv) 10–20% catalyst loading and 10% ligand loading as indicated above with the indicated solvent systems (1 mL) for 2–3 h. Reported yields are isolated yields.

^a Added 0.2 mmol (2 equiv).

^b Added 0.2 mmol (2 equiv).

both base and metal catalyst are required as removal of either one resulted in no product (**entry 16–17**). Yields were concentration dependent; as the concentration of the reactants was decreased (0.5 M), more biphenyl (byproduct) was produced (presumably due to adventitious reaction with O_2 from air) which significantly lowered the yield of the desired benzophenone due to substrate depletion. Nevertheless, sparging the solvents with argon prior to reaction did not significantly improve the yield of the desired benzophenone.

Following this optimization study, we found that the benzoyl-MIDA boronate ester **1** reacts well with different aryl bromides **2b-o** (see Scheme 3). The majority of the aryl bromides are *para* substituted groups **3c-n**, but by comparing **3b** and **3d**, there is no significant change in yield when the substrate is substituted at the *ortho* position. A noteworthy observation is that coupling is relatively rapid; TLC analysis showed total consumption of **1** within 2–3 h for all the substrates. This is compelling information as most Suzuki-Miyaura reactions require hours, if not overnight, with a sustained high temperature.

From the isolated yields, we observe that the presence of electron-withdrawing groups (**2h-l**, **o**), including a heteroaromatic bromopyrimidine gave moderate to good yields while the presence of electron-donating groups gave mixed results; **2b-g** gave reasonable yields however bromo-phenol **2m** and bromo-aniline **2n** gave no detectable ketone. Although reasons for this are not immediately intuited, we speculate that the aniline could condense with the aroyl-MIDA ester to form an imine, which has been observed for **3n** and this may serve to reduce yields.

In light of this modest success, several acyl-MIDA esters (alkyl series) were subjected to the same reaction conditions above. Unfortunately, none of these underwent Suzuki-Miyaura cross coupling (Scheme 4).

While the basis for this failure remains entirely unclear, we posit that upon MIDA solvolysis, the resulting acylboronic acid, which is to date an elusive species that has never been isolated, could undergo uncontrolled deborylation at rates that may compete with either metal coordination or transmetalation. While such is also conceivable with the benzoyl boronic acid, it is thus indeed puzzling as to why the benzoyl species underwent successful coupling. Further studies would be required to address the solvolysis



Scheme 3. Preparation of biaryl ketones from coupling of benzoyl-MIDA boronate ester and aryl bromides. **1** (1 eq.) and **2b-o** (1.1 eq.) were used in the presence of PdCl₂(dppf)*CH₂Cl₂ (20 mol%) and 3.33 M K₂CO₃ (6 equiv) and mixing in DCM (0.1 M). The reaction is heated to 60 °C. Isolated yields are reported. Yields are an average of two experiments.



Scheme 4. Acyl-MIDA boronate esters with 4-bromobenzonitrile as possible Suzuki-Miyaura cross coupling partners.

rates of these substrates and to provide greater mechanistic insight into this apparent discrepancy which we highlight here. It is noteworthy that that majority of acylative coupling reports cited herein have focused on aroyl donors and most note much higher yields for these compared to aliphatic substrates. It is possible that in the case of aliphatic substrates, β -hydride elimination may erode yields compared to the aroyl substrates.

Of note, unlike other conventional Suzuki-Miyaura approaches, this study was conducted under air- and water-tolerant conditions, which we sought in terms of convenience. We recognize that the use of highly reactive palladium catalyst species in conjunction with inert glove-box conditions may further enhance yields and may enable coupling to (non-aryl) acyl MIDA boronates. However, given the utility of the Suzuki-Miyaura reaction outside of a glove box, we constrained our focus to applications that are userfriendly, scalable, and readily practicable.

We should also note that in our hands the benzoyltrifluoroborate did not couple. The notable indolence of the corresponding acyl/aroyltrifluoroborates to coupling is rationalized by recognizing the considerable kinetic stability of the acyltrifluoroborates to hydrolysis [55], which is widely accepted to be required for coupling [3,56–58]. While we appreciate that certain aroyl-trifluoroborates can be converted to the corresponding MIDA boronate esters by treatment with MIDA-TMS diesters in the presence of BF₃-OEt₂ [42], when we treated *p*-cyanobenzoyl trifluoroborate or the *p*-anisoyl trifluoroborate, we observed no reaction in contrast to the moderate yields (54–65%) that had previously been reported for a very limited number of alkyl-substituted aroyl trifluoroborates [42].

Since in our hands, the conversion of two commercially available aroyltrifluoroborates to the corresponding aroyl MIDA boro-



Scheme 5. Suzuki-Miyaura cross coupling of substituted benzoyl-MIDA boronate esters with aryl bromides.

nate esters was not readily achieved, we assert that aroyl MIDA boronate would need to be procured as previously reported starting with the aryl acetylene precursors followed by hydroboration, BPin-BMIDA exchange, and then either ozonolysis or Upjohn dihydroxylation and Lemieux-Johnson oxidative cleavage [43,44]. By applying these methodologies, we synthesized two different substituted benzoyl MIDA boronate esters, 6a-4-fluoro and 6b-4-tert butyl (Scheme 5). Substrate 6a proved difficult in terms of solubilization, which necessitated modification of the solvent system to facilitate SMCC. By comparing substrates 7aa, 7ab and 7ac, there is no significant change in yield with the differing aryl bromides. Electronically, the anisole and benzonitrile groups did not influence the coupling capabilities when compared to bromobenzene. For substrates 7ba, 7bb, and 7bc, there is some discernable difference between each other. Substrate 7ba displays has the same vield **3c** (Scheme 2) but for **7bb** and **7bc**, the electronics from the arvl bromide may have made a difference. SMCC looks to heavily favor electron-withdrawing aryl bromides more than the electron-donating ones.

Given the general robustness of Suzuki-Miyaura coupling, we speculate that this reaction is likely to tolerate electron-releasing groups on the aryl ring as well, particularly since these substituents would be considered remote from the site of metal coordination or insertion.

Such considerations notwithstanding, even if one were to increase yields and expand the substrate scope further, the elaboration of aryl acetylenes to aroyl-MIDA-boronates or accessing them by converting the trifluoroborates in the presence of the bis-TMS MIDA diester is likely to make this approach to ketone synthesis considerably less efficient than if one were to simply start with the corresponding aroyl ester or amide and couple to a standard nucleophile.

In conclusion, we report the first example of a coupling an aroyl-MIDA boronate ester. In so doing, we synthesized a variety of asymmetric biaryl ketones via Pd-catalyzed cross coupling reactions of benzoyl MIDA boronate ester and aryl bromides. Reactions with non-aryl acylboronate MIDA esters are attempted as well but yielded less than stellar results and further research must be done to incorporate them into future experiments. More importantly, this work expands the Suzuki-Miyaura cross coupling reaction to include acylboronate as substrates for coupling, with current limitations as noted herein.

Note added in proof

In the revision of this work, it became evident that Trofimova et al. *Org. Lett.* 2021, 23, 9, 3294–3299 have published a similar study that is consistent with the findings reported herein.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2021.153147.

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