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Rapid Access to Highly Functionalized Alkyl Boronates by NiH-Catalyzed Remote Hydroarylation of Boron-Containing Alkenes

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Abstract: *The direct and selective functionalization of relatively simple and readily accessible precursors to produce highly functionalized alkyl boronates is a synthetically useful process. Herein we report a NiH-catalyzed remote hydroarylation process that can, through a synergistic combination of chain walking and subsequent cross-coupling, introduce an aryl group at the adjacent carbon atom of alkyl boronates under mild conditions. By means of a preliminary experiment with moderate enantioselectivity, it was shown that an asymmetric version could also be realized.*

Alkyl boronates are a privileged scaffold in materials science and drug discovery; they are also valuable and versatile precursors for the construction of structurally complex molecules.[1] Recently, there have been advances in the stereospecific transformation of alkyl boronates to forge C−C, C−O, C−N, and C−X bonds.[2] Accordingly, efficient, selective, and sustainable methods to introduce a boryl moiety have been developed to access these functionalized alkyl boronates (Figure 1 a, left).[3] The introduction of a functional group into a boron-containing substrate by catalytic functional group transformation offers a complementary route to these valuable and densely functionalized boronates (Figure 1 a, right).[4] In contrast, the direct and selective C(sp^3)−H functionalization of easily accessible alkyl boronates remains a significant unexplored challenge.

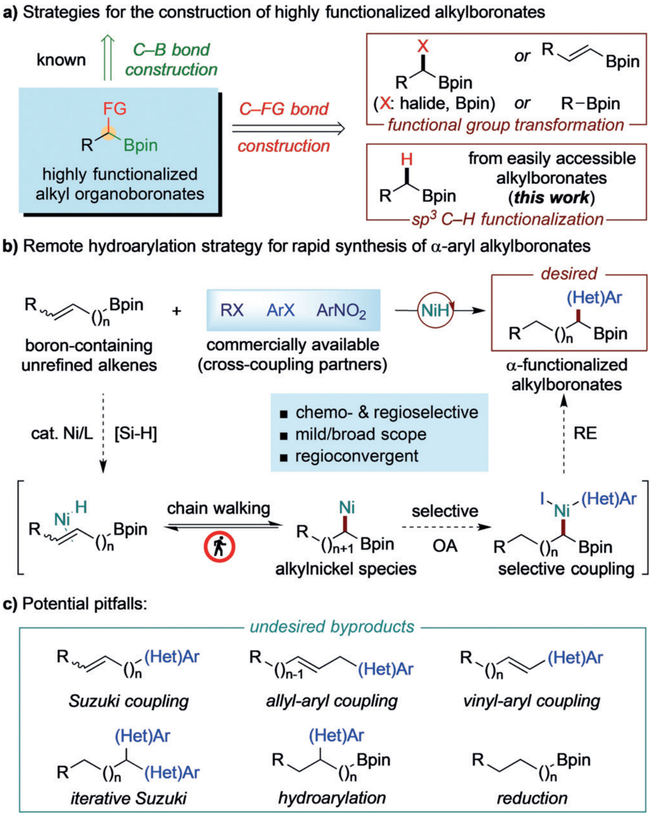

Figure 1. Design plan: Access to highly functionalized alkyl boronates by functionalization of readily accessible precursors.

The recently reported remote functionalization strategy based on the synergistic combination of chain walking and cross-coupling chemistry provides a mild and efficient strategy for the rapid assembly of structurally complex molecules from easily prepared alkenes and a wide variety of commercially available cross-coupling partners.[5–9] Considering the low cost and sustainability of nickel cross-coupling chemistry,[10] migratory cross-couplings catalyzed by nickel hydride[11] have led to the discovery and development of a variety of unique and valuable transformations.[9] In this type of reaction, nickel plays two roles, catalyzing the processes of chain walking and cross-coupling. We recently questioned whether this generic strategy could be used to gain rapid access to a valuable class of α-functionalized alkyl boronates from easily accessible unsaturated alkyl boronates and commercially available cross-coupling partners (Figure 1 b). It was envisioned that the NiH species generated in situ would promote a rapid chain walking process, accessing various alkylnickel species along the alkyl chain prior to a selective cross-coupling with an aryl halide. Ideally, with a suitable ligand, such a migratory arylation process could take place at the adjacent α-carbon atom of the alkyl boronate to deliver the corresponding α-aryl alkyl boronate in a chemo- and regioselective manner from an alkene whose double bond is in an arbitrary position. Herein, we describe the successful execution of this reaction under exceptionally mild conditions.

While the above strategy can be viewed as an attractive approach to highly functionalized alkyl boronates, there are many potential pitfalls (Figure 1 c). First, a Ni-catalyzed Suzuki reaction is possible because both starting materials and products are organoboronates. Second, a chain walking process could potentially lead to the formation of isomeric products due to the similar reactivities of the alkylmetal

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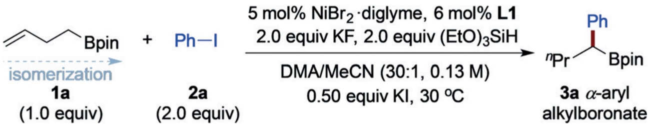
intermediates. Third, the alkenes and aryl iodides could be reduced by nickel hydride. Achieving the requisite chemo- and regioselectivity is a major unexplored challenge.

Being aware of these possible pitfalls, we began our investigation with an examination of the remote hydroarylation[12] of homoallyl boronic acid pinacol ester (**1a**) with iodobenzene (**2a**). After extensive examination of nickel sources, ligands, silanes, bases, solvents, and additives, the desired α-aryl alkyl boronate (**3a**) was obtained at 30 °C in 70 % isolated yield. The reaction manifested excellent regioselectivity (regioisomeric ratio, rr: α-aryl product/all other isomers = 97:3; Table 1, entry 1). Use of other nickel sources such as NiI_2 gave diminished yields (entry 2). An evaluation of other ligands showed that the use of a similar ligand, neocuproine (**L2**), led to a significantly lower yield (entry 3), and replacement of **L1** with the parent bipyridine (bpy) produced no desired arylation product (entry 4). Polymethylhydrosiloxane (PMHS) was shown to be a less effective silane (entry 5), and replacement of KF by CsF led to diminished yield (entry 6). The reactivity could however be improved by the addition of KI as an additive (entry 1 vs. entry 7) and acetonitrile as co-solvent (entry 1 vs. entry 8). Finally, bromobenzene was found to be considerably less reactive than iodobenzene (entry 9).

Table 1: Variation of the reaction parameters.

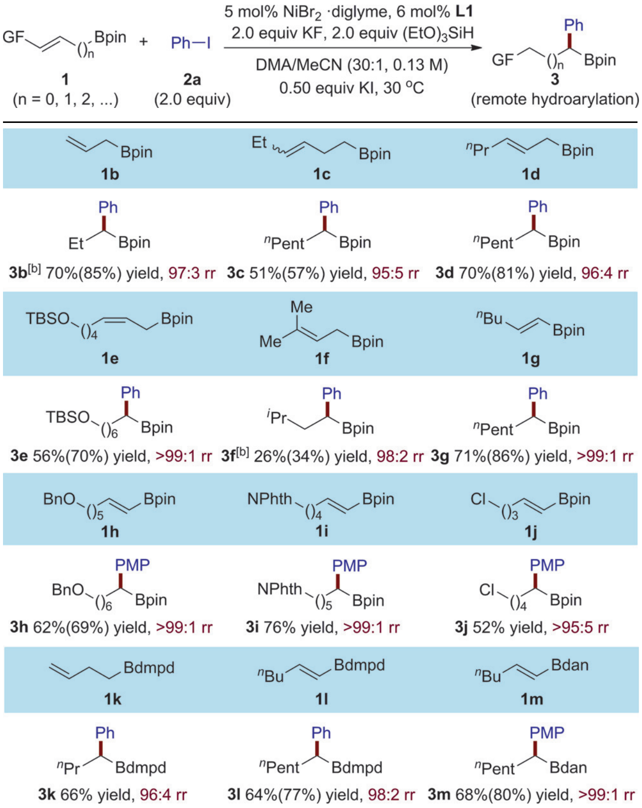


Entry	Variation from the standard conditions	Yield [%][a]	rr[b]
1	none	87 (70)	97:3
2	NiI_2 instead of $NiBr_2$·diglyme	27	98:2
3	**L2** instead of **L1**	25	95:5
4	bpy instead of **L1**	trace	–
5	PMHS instead of $(EtO)_3SiH$	35	93:7
6	CsF instead of KF	47	95:5
7	without KI	78	92:8
8	without MeCN	36	95:5
9	PhBr instead of PhI	39	96:4

[a] Yields determined by GC analysis using *n*-dodecane as the internal standard; the yield in parentheses is the yield of isolated product and is an average of two runs (0.20 mmol scale). [b] Ratio of arylation at the adjacent carbon atom of the boronate to the sum of all other isomers as determined by GC analysis. PMHS = polymethylhydrosiloxane.

Under the optimized reaction conditions, a variety of unactivated terminal (**1a**, **1b**, and **1k**) and internal (**1c**–**1f**) alkenes as well as activated alkenyl boronic esters (**1g**–**1j**, **1l**, and **1m**) successfully underwent the desired migratory arylation, delivering the α-aryl alkyl boronates in good yields and with excellent regioselectivity (Table 2). As expected, both *E* (**1d**, **1g**–**1j**, **1l**, and **1m**) and *Z* (**1e**) alkenes as well as *E*/*Z* mixtures (**1c**) were accommodated well, and high selectivity for arylation at the adjacent carbon position of the alkyl boronate was observed, regardless of the position of the C=C bond in the starting material (compare **1c**, **1d**, and **1g**). The current reaction conditions could also be used with a more sterically hindered trisubstituted alkene (**1f**) to form the migratory product (**3f**), albeit in diminished yield. Notably, even with a heteroatomic substituent at the other terminus of the alkyl chain, including ethers (**1e**, **1h**), a phthaloyl amide (**1i**), and an alkyl chloride (**1j**), arylation at the α-carbon atom of the alkyl boronate was still observed.

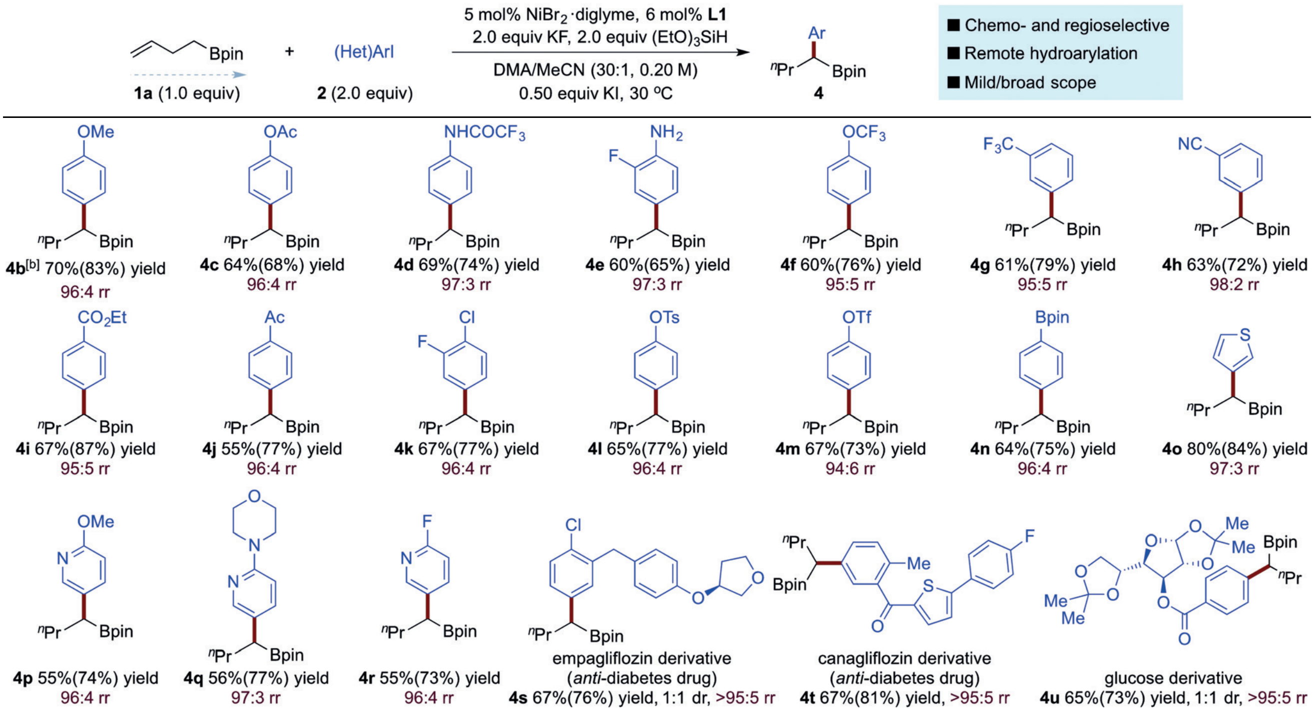
Table 2: Scope of the alkene component.[a]



[a] Yields of isolated products are given (0.20 mmol scale, average of 2 runs); yields in parentheses refer to the crude ^{1}H NMR yield (1,1,2,2-tetrachloroethane as the internal standard). rr represents the ratio of the product of arylation at the adjacent carbon atom of the boronate to the sum of all other isomers as determined by GC analysis; ratios reported as > 95:5 were determined by crude ^{1}H NMR analysis. [b] At 35 °C. Phth = phthaloyl.

A subsequent survey of aryl iodides revealed that a range of aryl and heteroaryl groups could be used. As depicted in Table 3, several electron-rich (**2b**–**2e**) and electron-poor (**2f**–**2n**) aryl iodides were shown to be acceptable substrates. A variety of functional groups were readily accommodated, including ethers (**2b**, **2f**, **2p**, **2q**, **2s**, and **2u**), esters (**2c**, **2i**, and **2u**), an amide (**2d**), anilines (**2e**, **2q**), a nitrile (**2h**), ketones (**2j**, **2t**), and an acetal (**2u**). Of particular interest is that potential coupling motifs, including aryl fluorides (**2e**, **2k**, **2r**, and **2t**), an aryl chloride (**2k**), an aryl tosylate (**2l**), an aryl triflate (**2m**), and a boronic ester (**2n**) remained intact and were available for subsequent chemical modification. A series of heterocycles frequently found in medicinally rele-

Table 3: Scope of the aryl iodide coupling partner.[a]

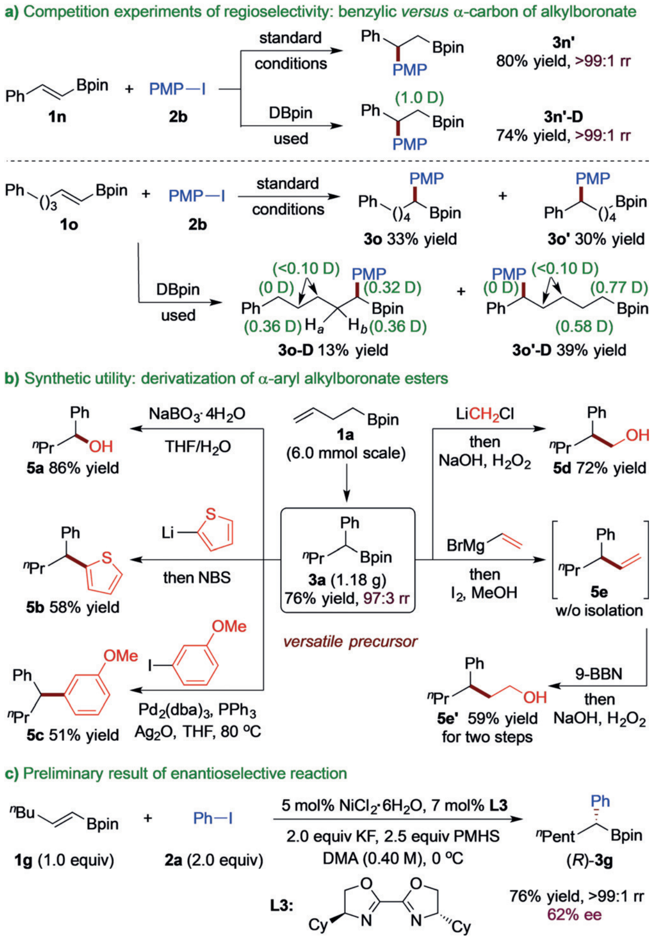


[a] Yield and rr as defined in Table 2. [b] DMA/MeCN (0.13 M).

vant targets, including thiophenes (**2o**, **2t**) and pyridines (**2p**, **2q**, and **2r**), were well tolerated. This valuable transformation could therefore be used for the late-stage functionalization of pharmaceutically relevant and structurally complex intermediates (**2s**–**2u**). Aryl iodides derived from commercially available pharmaceuticals, such as empagliflozin (**2s**) and canagliflozin (**2t**), successfully underwent this migratory cross-coupling. Carbohydrate compounds such as the glucose derivative **2u** also readily underwent the targeted migratory arylation.

The regioselectivity could also be switched to other positions. Competition experiments were carried out to compare the site selectivity of the benzylic position and the adjacent α-carbon atom of the alkyl boronate (Scheme 1a). In general, under the current conditions, arylation takes place at the benzylic position rather than the adjacent α-carbon atom of the boronate. In the case of the β-boronic ester substituted styrene **1n**, arylation at the benzylic position to produce exclusively the β-aryl-substituted boronate product **3n′** was observed. In the case of an alkenyl boronate with a remote aryl group in the alkyl chain (**1o**), products arylated at both the α-carbon atom of the boronate (**3o**) and the benzylic position (**3o′**) were obtained as a 1:1 mixture. In contrast to the highly regiospecific insertion of NiH into a styrenic intermediate, isotope labeling experiments indicated that the insertion of NiH into the β-alkyl-substituted alkenyl boronate is non-regiospecific and reversible.

The robustness and synthetic utility of this catalytic system were further demonstrated by the gram-scale synthesis and subsequent derivatization of the products (Scheme 1b). On gram scale, **3a** was isolated in 76% yield. As noted at the outset, α-functionalized alkyl boronates are versatile syn-



Scheme 1. Competition experiments, synthetic utility, and preliminary results regarding the enantioselective version.

thetic intermediates that can be converted into a wide range of other valuable compounds through facile transformations. As illustrated in Scheme 1 b, a wide array of functional groups could be introduced smoothly into the benzylic position of **3a** in good yields by applying known procedures (**5a**–**5e**).

The current transformation can be realized in an enantioselective fashion by using a suitable chiral ligand. In a preliminary experiment (Scheme 1 c) with the simple chiral (*S*,*S*)-Cy-Biox ligand **L3**, (*R*)-**3g** was obtained with moderate enantioselectivity (62 % *ee*) and isolated in good yield (76 %).

In summary, based on the NiH-catalyzed remote hydrofunctionalization platform, we have established a practical and efficient remote hydroarylation process for the generation of α-functionalized alkyl boronates from simple boron-containing olefins and commercially available cross-coupling partners. Under the mild conditions used, excellent chemo- and regioselectivities were observed for a wide range of both alkene and aryl iodide partners. Further studies on the catalytic asymmetric version of this transformation based on ligand design are in progress.

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Conflict of interest

The authors declare no conflict of interest.

Keywords: arylation · C−H activation · isomerization · nickel · regioselectivity

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