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Cobalt-Catalyzed Annulation of Benzimidates or NH-Benzaldimines with Ynamides: Synthesis of 1-Alkoxy- and 1-Alkyl-3-Aminoisoquinolines

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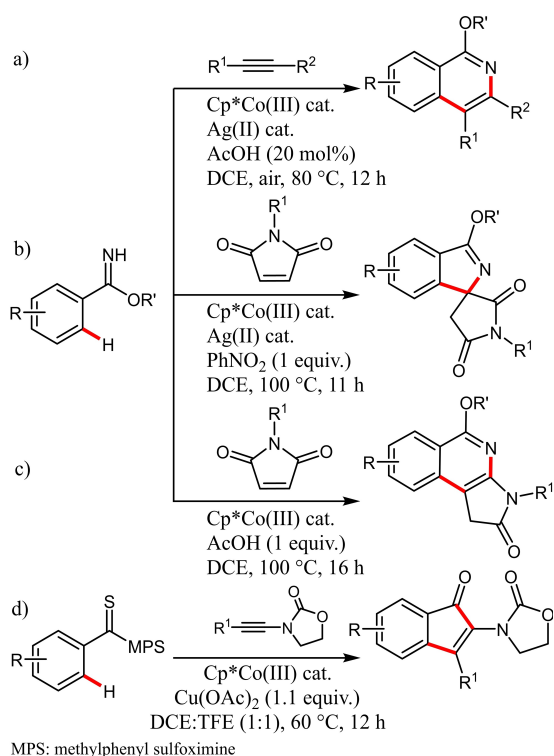
Abstract: Inexpensive cobalt-catalyzed, pivalic acid-assisted oxidative C–H functionalization of benzimidates or NH-benzaldimines with ynamides is presented, providing an access to 3-aminoisoquinoline derivatives. The procedure is compatible with various substrates featuring alkyl, alkoxy, acyl or halogen as substituents, heteroaryl structures, and diverse ynamides. The reaction has been scaled up, with subsequent transformation of the yielded products. Control experiments and mechanistic investigations corroborate the regioselectivity of this conversion, positioning the amine group proximal to the nitrogen atom in isoquinolines.

Keywords: Ynamide; Cobalt catalysis; 3-Aminoisoquinoline; C–H Activation/Annulation Reaction; Benzimidate

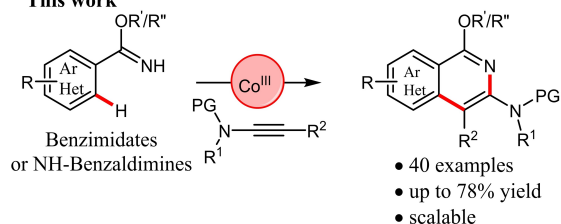
The development of new methodologies for selectively forming bonds is a prominent focus in contemporary synthetic chemistry. Within this realm, the transition metal-catalyzed olefination of inert C–H bonds with readily available unsymmetrical alkyne derivatives has emerged as a potent approach for incorporating an alkenyl substituent onto (hetero)arenes in a step-efficient and atom-economical manner.^[1,2] Furthermore, the iso-

quinoline motif is frequently encountered in numerous functional molecules, notably in several biologically significant compounds.^[3,4] Recently, various transition metal-catalyzed C–H activation/annulation methodologies employing rare and noble third-row transition metals, such as rhodium,^[5–9] ruthenium^[10–12] and palladium^[13–14] complexes, have been documented as offering elegant pathways to access this motif.^[15] This strategy involves the use of a directing group capable of chelating the metal, thereby assisting the oxidative addition of the metal center to the C–H bond. To fulfil the requirements of green chemistry and sustainable development, the exploitation of earth-abundant transition metals as catalysts in unreactive chemical bond activations has become more attractive, owing to their distinct benefits of affordability and minimal to no toxicity.

Recently, high-valent cobalt complexes have shown their singular nature in aromatic C–H activation, particularly for the synthesis of biologically and pharmaceutically active heterocycles.^[16] Various nitrogen-based directing groups have been used in Co(III)-catalyzed C–H activation. Aryl imidates, readily available compounds, have been investigated for C–H functionalization due to their good reactivity. The C–H alkenylation/annulation of benzimidates with alkynes using a Cp*Co(III) catalyst under air was reported by Ding and Sun, leading to a series of substituted isoquinolines (Scheme 1a).^[17] A cobalt-catalyzed spiro



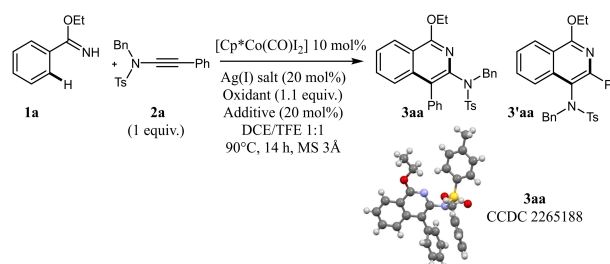
MPS: methylphenyl sulfoximine

This work**Scheme 1.** Context of the investigation and our current work.

annulation of benzimidates with maleimides, providing a rapid route to five-membered nitrogen containing spirocyclic molecules was reported by Zhang (Scheme 1b).^[18] Unlike previously known cobalt-catalyzed oxidative C–H functionalization reactions, this transformation occurs efficiently in the absence of an oxidant but in the presence of nitrobenzene as a promoter. Lv and Chen later advantageously utilized a similar strategy for the synthesis of fused-ring heterocycles (Scheme 1c).^[19] While examples of metalcatalyzed benzimidate annulation reactions with alkynes or alkenes are well documented, the use of ynamide, an *N*-containing polarized unsymmetrical alkyne, remains rare. Recently, Sahoo reported a cobalt-catalyzed thioamide-directed C(arene)–H annulation with ynamide, leading to 2-amidoindenones (Scheme 1d).^[20] In this context and in light of our continuous interest in ynamide chemistry,^[21–23] we herein present a cobalt-catalyzed hydroarylation and annulation sequence of ynamides with benzimidates to yield original 3-aminoisoquinolines. This reaction features a broad substrate

scope and good functional group tolerance. Notably, the reaction was scaled up, and the resulting products were further transformed.

We started our investigations by using benzimidate **1a** and ynamide **2a**^[23] as model substrates to optimize the reaction conditions (Table 1). Gratifyingly, we observed a yield of 28% of the desired 3-aminoisoquinoline **3aa** as the sole compound in the presence of the Co(III) complex [Cp*Co(CO)₂] (10 mol%), Ag₂CO₃ (10 mol%) in TFE/DCE (1:1) at 90 °C for 14 h (entry 1). Due to the likely chelate-assisted regioselective insertion of ynamide (cf **B**, Scheme 5), no trace of

Table 1. Optimization of the reaction conditions.^[a]

Entry	Ag(I) salt	Additive	Oxidant	Yield ^[f] 3aa (%)
1	Ag ₂ CO ₃	–	Ce(SO ₄) ₂	28
2	Ag ₂ CO ₃	KOAc	Ce(SO ₄) ₂	55
3	Ag ₂ CO ₃	NaOAc	Ce(SO ₄) ₂	50
4	Ag ₂ CO ₃	CsOAc	Ce(SO ₄) ₂	63
5	Ag ₂ CO ₃	KOPiv	Ce(SO ₄) ₂	49
6	Ag₂CO₃	PivOH	Ce(SO₄)₂	73(69)^[g]
7	Ag ₂ CO ₃	AcOH	Ce(SO ₄) ₂	4
8	Ag ₂ CO ₃	PhCO ₂ H	Ce(SO ₄) ₂	43
9 ^[a]	Ag ₂ CO ₃	PivOH	Ce(SO ₄) ₂	59
10 ^[b]	Ag ₂ CO ₃	PivOH	Ce(SO ₄) ₂	0
11 ^[c]	Ag ₂ CO ₃	PivOH	Ce(SO ₄) ₂	11
12	–	PivOH	Ce(SO ₄) ₂	39
13	AgNTf ₂	PivOH	Ce(SO ₄) ₂	32
14	AgOTf	PivOH	Ce(SO ₄) ₂	33
15	AgSbF ₆	PivOH	Ce(SO ₄) ₂	20
16	Ag ₂ CO ₃	PivOH	Cu(OAc) ₂	22
17	Ag ₂ CO ₃	PivOH	Cu(OAc) ₂ ·H ₂ O	12
18	Ag ₂ CO ₃	PivOH	CuO	73
19	Ag ₂ CO ₃	PivOH	Ag(OAc) ₂	20
20	Ag ₂ CO ₃	PivOH	Mn(OAc) ₂	54
21 ^[d]	Ag ₂ CO ₃	PivOH	–	31
22 ^[e]	Ag ₂ CO ₃	PivOH	Ce(SO ₄) ₂	46

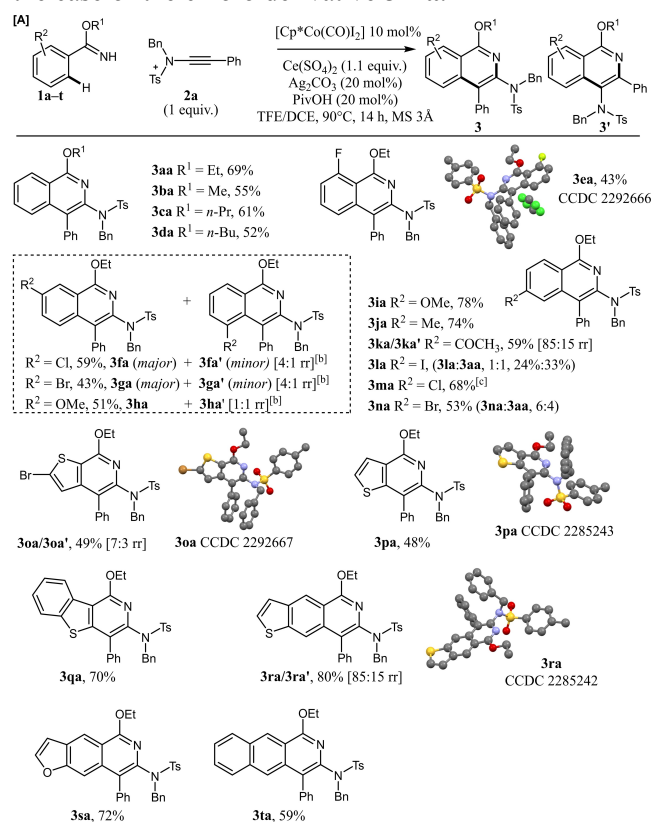
^[a] 0.4 equiv. of PivOH was used.^[b] Without Co catalyst or using CoBr₂ or Co(OAc)₂.^[c] Using [Cp*Co(MeCN)₃](SbF₆)₂.^[d] Using O₂ as the oxidant, a yield of 51% was observed.^[e] Without MS 3 Å.^[f] Yields were determined by ¹H NMR analysis of the crude reaction mixtures using mesitylene as an internal standard.^[g] isolated yield. DCE: 1,2-Dichloroethane. TFE: 2,2,2-Trifluoroethanol.

the regioisomeric product **3'aa** was detected. The structure of **3aa** was unambiguously determined by X-ray crystallography. While the reaction yields increased to 49–63% when adding a base as an additive (entries 2–5), the presence of PivOH showed a clear improvement, and afforded **3aa** in 69% isolated yield (entry 6). PivOH turned out to be beneficial and necessary, likely facilitating C–H activation and cyclization processes. Motivated by this outcome, we screened acid additives, including AcOH or PhCOOH, albeit without improvement (entries 6–8). It is worth noting that increasing the amount of PivOH to 0.4 equiv. resulted in a decrease in the yield to 59% and the formation of a side compound **7**, resulting from the regio- and diastereoselective hydroacyloxylation of ynamide **2a** (entry 9 and see also SI).^[24] No reaction was observed in the absence of cobalt catalyst, or in the presence of other cobalt complexes such as CoBr₂ or Co(OAc)₂, underscoring the indispensability of the Co(III) complex for the reaction (entry 10). Employing [Cp*Co(MeCN)₃](SbF₆)₂ as a catalyst gave lower yields (entry 11). Silver(I) salt is also essential for this transformation (entry 12), with Ag₂CO₃ providing optimal results. The diminished yields observed with other silver salts likely stemmed from ynamide degradation (entries 13–15).

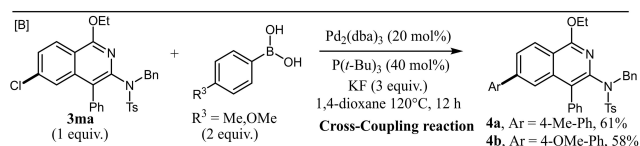
Subsequently, various oxidants were assessed (entries 16–20), with Ce(SO₄)₂ and CuO yielding the most favorable outcomes. Due to producing cleaner reaction crudes, Ce(SO₄)₂ was ultimately selected as the oxidizing agent. Exclusion of Ce(SO₄)₂ led to a pronouncedly lower yield (entry 21), suggesting a substantial co-oxidant effect; conducting the reaction in air, with O₂ serving as the co-oxidant, failed to produce satisfactory yields. The presence of 3 Å molecular sieves, trapping traces of water, proved crucial to the reaction (entry 22), preventing the conversion of ynamide into the corresponding amide. Subsequent exploration of solvent effects revealed that a 1:1 mixture of TFE/DCE was the optimal choice (cf Supporting Information).^[25] Moreover, reducing the reaction temperature to 60 °C or room temperature resulted in yields of 28% or traces of the product, respectively. It is worth noting that attempts to use diphenylacetylene as the coupling partner gave only a trace amount of the expected compound (not shown), underscoring the unique reactivity of ynamide in this reaction.

With the optimized reaction conditions in hand, we next examined the substrate scope of various benzimidates **1a–t** (Scheme 2A). The different nature of alkyl benzimidates **1a–d** was first studied, leading to the corresponding 1-alkoxy-3-aminoisoquinolines **3aa–3da** with good yields. A moderate yield (**3ea**, 43%) was observed from ethyl 2-fluorobenzimidate. Upon utilizing ethyl 3-chloro-, 3-bromo-, or 3-methoxybenzimidate, substrates possessing unsymmetrical sites for C–H activation, the respective isoquinolines

were isolated with moderate yields as an inseparable mixture of isomers **3fa** and **3fa'** (59%, 4:1 rr), **3ga** and **3ga'** (43%, 4:1 rr), or **3ha** and **3ha'** (51%, 1:1 rr). In this case, two acidic protons favorable to C–H bond activation can indeed be considered, and according to the literature, the major regioisomer undergoes C–H activation at the less sterically congested site.^[26] However, the formation of regioisomers at the ynamide site cannot be ruled out. Arylimidates bearing both electron-donating (Me, OMe) and -withdrawing (halogen, ketone) groups at the *para* position of the phenyl ring (**1i–n**) all coupled smoothly with ynamide **2a**, affording the annulated products **3ia–na** in good to excellent yields. Products **3ka** and **3ka'** having an electron-poor (*p*-CO₂Me) aryl substituent were isolated as an inseparable mixture of ynamide regioisomers. Both chelation of the ynamide tosyl group and π -interaction of its aryl terminal group with Co species can contribute to the formation of regioisomers.^[20] It should be pointed out that in the cases of the iodo- and bromo-derivatives **3la** and **3na**, a side product was observed, resulting from a reduction step leading to **3aa** (with R² = H).^[27] This side reaction was not observed in the case of the chloro derivative **3ma**.



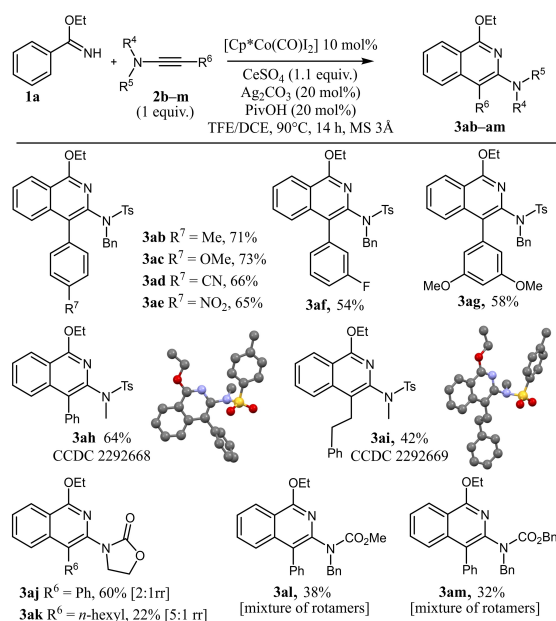
Aiming to attain access to molecular diversity and key scaffolds for novel pharmaceuticals, we succeeded in synthesizing polyheterocyclic compounds featuring a thiophene, benzothiophene or benzofuran scaffold (**3oa–sa**) with moderate to good yields. It is worth noting that products **3oa/3oa'** and **3ra/3ra'** were



Scheme 2. The scope of benzimidates [A] and product derivatizations [B]. [a] All reactions were run on a 0.3 mmol scale. Hydrogen atoms have been omitted from the X-ray structure for clarity. Regioisomeric ratios (π) were determined by ^1H NMR analysis of the crude reaction mixtures. [b] Regioisomeric ratio was determined from isolated yields. [c] The reaction was carried out on a 2.0 mmol scale: 1.084 g, 59% yield.

isolated as a mixture of ynamide regioisomers. The structures of **3oa**, **3pa** and **3ra** were unambiguously determined by X-ray crystallography. The naphthalene derivative **3ta** was also isolated in a yield of 59%. The robust nature of the mild cobalt catalysis was reflected by the gram-scale synthesis of the chloro derivative **3ma**. The synthetic utility of our approach was mirrored by cross-coupling reactions leading to **4a** or **4b** with good yields (Scheme 2B).

To ascertain the scope of this reaction, various ynamides **2b–m** bearing different terminal substituents (aryl or alkyl) and/or protecting groups on the nitrogen atom were studied (Scheme 3). Remarkably, a total regioselectivity was generally observed, with results indicating the tolerance of diverse substituents and substitution patterns on the terminal aryl ring. The structures of **3ah** and **3ai** were unambiguously determined by X-ray crystallography. Furthermore, the

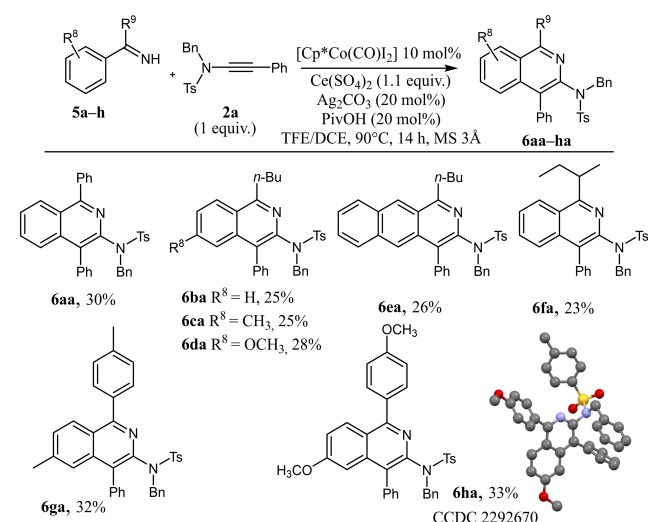


Scheme 3. Scope of ynamides. Reactions run on a 0.3 mmol scale, isolated yields. Hydrogen atoms have been omitted from the X-ray structure for clarity.

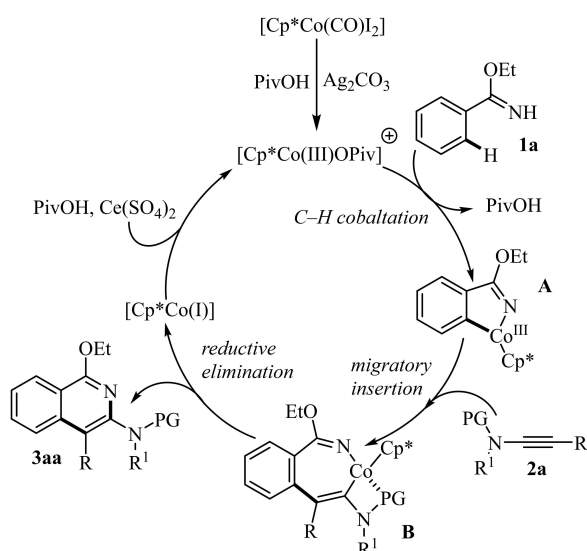
reaction demonstrated compatibility with ynamides containing an oxazolidine-2-one ring, yielding **3aj–ak**, albeit with low selectivities, since a weakly coordinating directing group may disfavor the stabilization of the ortho-alkenyl cobalt intermediate (cf. **B** in Scheme 5). It is worth noting that alkyl-substituted ynamides are more prone to degradation, likely accounting for the poor yield observed for **3ak**.^[28] A mixture of rotamers was detected for carbamates **3al** and **3am**. Furthermore, the reaction turned out to be compatible with a variety of functional groups, which were amenable to further useful transformations.

Interestingly, we also demonstrated that the reaction is not confined solely to benzimidates; NH benzaldimines **5a–h** underwent the same high regioselectivity, albeit more sluggishly (Scheme 4). The 1-alkyl or 1-aryl-3-aminoisoquinolines **6aa–ha** were isolated in low yields, imines being less reactive towards organocobalt than benzimidates. Despite the meager yields observed, to the best of our knowledge, this C–H activation/annulation reaction between NH benzaldimine and ynamide is unprecedented.

Furthermore, a study on the kinetic isotope effect (KIE) was undertaken (cf. Supporting Information). A primary KIE value of 2.44 was observed in the parallel experiments, suggesting that the C–H bond cleavage is involved in the turnover-limiting step. Building upon previously reported studies,^[29–31] we propose a plausible catalytic cycle, as depicted in Scheme 5. Initially, the exchange of ligands involving $[\text{Cp}^*\text{Co}(\text{III})(\text{CO})\text{I}_2]$, Ag_2CO_3 and PivOH provides the cationic $[\text{Cp}^*\text{CoOPiv}]^+$ active species. Subsequently, imidate “N” coordination of **1a** with the *in situ* generated high-valent Co(III) species followed by the activation of the *o*-C(arene)–H bond, in close proximity to the directing



Scheme 4. Reactions of NH benzaldimines. Reactions run on a 0.3 mmol scale, isolated yields. Hydrogen atoms have been omitted from the X-ray structure for clarity.



Scheme 5. Plausible Catalytic Cycle.

group, forms a five-membered metalacycle **A**; the *ortho* C–H activation proceeds through a CMD process. The chelate-assisted regioselective insertion of ynamide leads to intermediate **B**. Finally, the reductive elimination delivers the desired 3-aminoisoquinoline product **3aa** and $[\text{Cp}^*\text{Co}(\text{I})]$ which is then re-oxidized in the presence of the oxidant $\text{Ce}(\text{SO}_4)_2$ to regenerate the active $\text{Co}(\text{III})$ complex.

In conclusion, we have devised a novel method for synthesizing 1-alkoxy- and 1-alkyl-3-aminoisoquinolines through the cobalt-catalyzed annulation of benzenimidates or NH-benzaldimines with ynamides. This strategy unveils C–H activation/annulation as a complementary method for crafting the targeted cyclic framework, a significant pharmacophore structure. The process exhibits tolerance towards diverse substituents, facilitating further functionalization, and displays scalability when conducted on a gram scale.

Experimental Section

General Procedure for the Cobalt-Catalyzed Annulation of benzenecarboximidates **1a–t** or benzaldimines **5a–h** and Ynamides **2a–m**

An oven-dried reaction vial equipped with a stir bar was charged with benzenecarboximidate **1a–t** (0.3 mmol, 1.5 equiv.) or benzaldimine **5a–h** (0.3 mmol, 1.5 equiv.), ynamide **2a–m** (0.2 mmol, 1.0 equiv.), $[\text{Cp}^*\text{Co}(\text{CO})\text{I}_2]$ complex (9.5 mg, 0.02 mmol, 10 mol%), Ag_2CO_3 (10.8 mg, 0.04 mmol, 20 mol%), PivOH (4.0 mg, 0.04 mmol, 20 mol%), $\text{Ce}(\text{SO}_4)_2$ (73.0 mg, 0.22 mmol, 1.1 equiv.), 3 Å molecular sieves (50.0 mg), TFE/DCE 1:1, v/v (1.0 mL). The mixture was then degassed three times, using the freeze-pump-thaw technique, and the vial was filled back with argon and sealed. The reaction mixture was heated at 90 °C for 14 h. The reaction mixture was then cooled-down to room temperature (ca. 20 °C), the vial unsealed, and

CH_2Cl_2 (10 mL) was added. The suspension was then filtered through Celite® washing with CH_2Cl_2 . The solvents were evaporated under vacuum and the crude product was purified by SiO_2 -column chromatography (petroleum ether/EtOAc 98:2 to 96:4, v/v) to afford the expected 1-alkoxy-3-aminoisoquinolines **3'** and/or **3** or the 1-alkyl-3-aminoisoquinolines **6**.

General Procedure for the Synthesis of Compounds **4a–b** via Suzuki Cross Coupling

An oven-dried reaction vial equipped with a stir bar was charged with compound **3ma** (54 mg, 0.1 mmol), KF (17.4 mg, 0.3 mmol), the appropriate 4-substituted phenylboronic acid reagent (0.2 mmol, 2 equiv.), $\text{Pd}_2(\text{dba})_3$ (18.3 mg, 0.02 mmol, 20 mol%), $\text{P}(t\text{-Bu})_3$ (40 μL, 0.04 mmol, 40 mol%), and freshly distilled and degassed 1,4-dioxane (1.0 mL). The vial was sealed under an argon atmosphere and the mixture was stirred at 120 °C for 12 h. The reaction mixture was then cooled-down to room temperature (ca. 20 °C), the vial unsealed, and the mixture was filtered through celite® washing with EtOAc. The solvents were then removed under vacuum, and the crude product was purified by SiO_2 -column chromatography (petroleum ether/EtOAc 95:5, v/v). Compounds **4a** and **4b** were isolated in 61% and 58% yields, respectively.

Crystallographic Data

CCDC-2265188 (**3aa**), CCDC-2292666 (**3ea**), CCDC-2292667 (**3oa**), CCDC-2285243 (**3pa**), CCDC-2285242 (**3ra**), CCDC-2292668 (**3ah**), CCDC-2292669 (**3ai**), and CCDC-2292670 (**6ha**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/structures.

Acknowledgements

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