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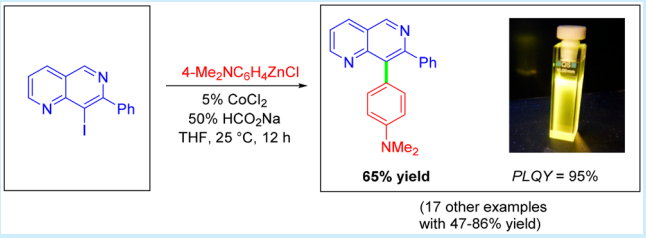
Preparation of Polyfunctional Naphthyridines by Cobalt-Catalyzed Cross-Couplings of Halogenated Naphthyridines with Magnesium and Zinc Organometallics

Robert Greiner,[†] Dorothée S. Ziegler,[†] Denise Cibu,[†] Andreas C. Jakowetz,[†] Florian Auras,[‡] Thomas Bein,[†] and Paul Knochel*[,†]

[†]Department of Chemistry, Ludwig-Maximilians-Universität, Butenandtstrasse 5-13, 81377 Munich, Germany
[‡]Cavendish Laboratory, University of Cambridge, Cambridge CB3 0HE, United Kingdom

Supporting Information

ABSTRACT: $CoCl_2$ (5%) catalyzes cross-couplings of various halogenated naphthyridines with alkyl- and arylmagnesium halides. Also, arylzinc halides undergo smooth cross-couplings with various naphthyridines in the presence of $CoCl_2 \cdot 2LiCl$ (5%) and sodium formate (50%), leading to polyfunctional arylated naphthyridines. Two of these arylated naphthyridines are highly fluorescent, with quantum efficiencies reaching 95% and long excited-state lifetimes of up to 12 ns.



N-Heterocyclic scaffolds are ubiquitous building blocks for pharmaceuticals, agrochemicals, and materials science.[1] There is a need for new N-heterocyclic structures since novel ring systems may display original biological or physical properties. Recently, the naphthyridine scaffold has attracted increased attention.[2] Its functionalization was found to be especially difficult, and only a few methods are available.[3] Although Fe-catalyzed cross-couplings have been used to functionalize chloronaphthyridines, the scope of such cross-couplings is quite limited.[4] Interestingly, Co-catalyzed cross-couplings generally display a broader reaction scope and have proved to be very useful for the functionalization of electron-deficient N-heterocycles.[5] We recently showed that the addition of appropriate ligands (e.g., sodium formate or pivalate) considerably extends the scope of these cross-couplings.[6] Herein we report that Co-catalyzed cross-couplings allow efficient functionalization of various halogenated naphthyridines.

In preliminary experiments, we found that chloronaphthyridines **1a**–**c** are easily alkylated using 5% $CoCl_2$ in THF (Table 1). Thus, the reaction of 3,6-dichloro-1,8-dimethyl-2,7-naphthyridine (**1a**) with 2-phenylethylmagnesium bromide (**2a**) at 25 °C (30 min) provided monoalkylated naphthyridine **3a** in 80% yield (Table 1, entry 1). Similarly, alkylmagnesium reagent **2a** allowed the conversion of 1-chloro-2,7-naphthyridine (**1b**) to the expected 1-phenethyl-2,7-naphthyridine (**3b**) in 82% yield (entry 2). Treatment of **1b** with MeMgCl (**2b**) gave the corresponding 1-methyl-2,7-naphthyridine (**3c**) in 98% yield (entry 3). Furthermore, *sec*-BuMgCl (**2c**) underwent cross-coupling with **1b** to afford 2,7-naphthyridine **3d** in 54% yield (entry 4). Co-catalyzed alkylation of 5-chloro-1,6-naphthyridine **1c** with BuMgCl (**2d**) provided 1,6-naphthyridine **3e** in 69% yield (entry 5). Additionally, the reaction of 5-chloro-1,6-naphthyridine **1c** with cyclopropylmagnesium bromide (**2e**) gave 1,6-naphthyridine **3f** in 52% yield (entry 6).

Furthermore, we have found that 3,6-dichloronaphthyridine **1a** was easily bisarylated in the presence of 5% $CoCl_2$ using arylmagnesium reagents[7] such as **4a**–**d** (Scheme 1). Thus, the reaction of **1a** with 4-trimethylsilylphenylmagnesium bromide (**4a**) (3.0 equiv) at −40 °C provided bisarylated 2,7-naphthyridine **5a** in 62% yield within 4 h. Similarly, 4-*N*,*N*-dimethylaminophenylmagnesium bromide (**4b**) and 4-anisylmagnesium bromide (**4c**) underwent Co-catalyzed cross-couplings (−40 °C, 4−12 h) with **1a**, leading to 3,6-substituted naphthyridines **5b** and **5c**, respectively, in 60−73% yield. Sterically hindered mesitylmagnesium bromide (**4d**), however, reacted with **1a** at 25 °C within 4 h, furnishing 2,7-naphthyridine **5d** in 93% yield.

We noticed that $C(sp^2)-C(sp^2)$ cross-couplings of naphthyridines **1b** and **1c** with PhMgCl using 5% $CoCl_2$ led to low yields (<30%). This problem could be solved by replacing arylmagnesium halides with the corresponding arylzinc reagents and using HCO_2Na as a ligand.[6a] Thus, **1b** reacted smoothly with PhZnCl (**6a**) or [1,1′-biphenyl]-4-ylzinc chloride (**6b**) within 12 h at 25 °C, furnishing the corresponding arylated naphthyridines **7a** and **7b** in 80−82% yield (Table 2, entries 1 and 2). Furthermore, a range of arylzinc reagents **6c**–**f** bearing various functional groups underwent such Co-catalyzed Negishi[8] cross-couplings with **1b**, providing the expected products **7c**–**f** in 69−79% yield (entries 3−6). Heteroaryl−heteroaryl cross-couplings are utmost challenging because of catalyst deactivation when Pd or Ni catalysts are used.[9] However, in the presence of THF-soluble $CoCl_2 \cdot 2LiCl$ (5%) and HCO_2Na (50%), the cross-

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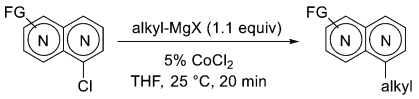
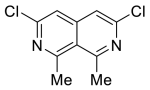
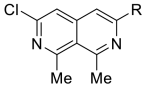
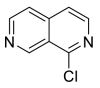
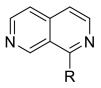
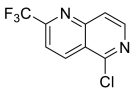
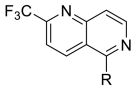
Table 1. Co-Catalyzed Alkylation of Chloronaphthyridines 1a–c with Alkylmagnesium Reagents 2a–e

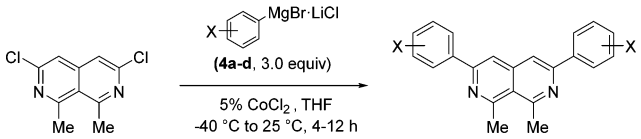


1a-c FG = H, Cl, CF_3, Me; X = Cl, Br **3a-f**

entry	naphthyridine	RMgX	product[a], yield (%)
			
1	**1a**	**2a**; R = $Ph(CH_2)_2$	**3a**, 80
			
2	**1b**	**2a**; R = $Ph(CH_2)_2$	**3b**, 82
3	**1b**	**2b**; R = Me	**3c**, 98
4	**1b**	**2c**; R = *s*-Bu	**3d**, 54
			
5	**1c**	**2d**; R = Bu	**3e**, 69
6	**1c**	**2e**; R = *c*-Pr	**3f**, 52

[a]Isolated yields of analytically pure products.

Scheme 1. Co-Catalyzed Bisarylation of Naphthyridine 1a with Various Arylmagnesium Bromides (4a–d)

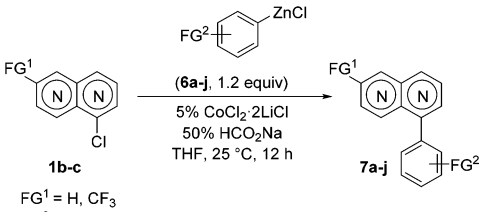


1a

5a: X = 4-$SiMe_3$; 62%
5b: X = 4-NMe_2; 73%
5c: X = 4-OMe; 60%
5d: X = 2,4,6-Me; 93%

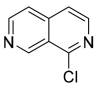
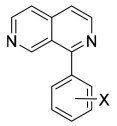
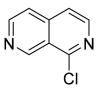
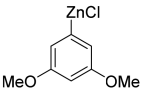
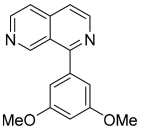
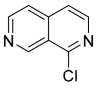
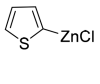
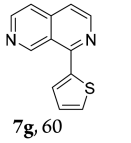
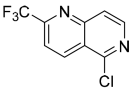
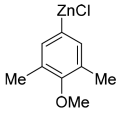
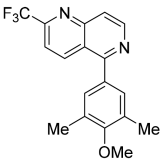
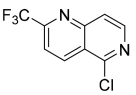
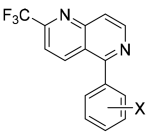
coupling of 1-chloronaphthyridine **1b** with 2-thienylzinc chloride (**6g**) afforded 2,7-naphthyridine **7g** in 60% yield (entry 7). We further have shown that naphthyridine **1c** was easily arylated under these conditions. Thus, the reaction of **1c** with arylzinc chlorides **6h** and **6i** provided 1,6-naphthyridines **7h** and **7i**, respectively, in 74–83% yield (entries 8 and 9). Interestingly, the Co-catalyzed arylation of **1c** with 2-(triisopropylsilyloxy)-phenylzinc chloride (**6j**) succeeded at only elevated temperature (60 °C, 12 h) to give naphthyridyl alcohol derivative **7j** in 61% yield (entry 10). Also, iodo-substituted naphthyridines were excellent substrates for such Co-catalyzed cross-couplings. Thus, the reaction of electron-rich arylzinc reagents **6k** and **6l** with 4-iodo-1,5-naphthyridine (**1d**) afforded 1,5-naphthyridines **7k** and **7l**, respectively, in 78–80% yield (Table 3, entries 1 and 2). Similarly, the reaction of electron-deficient *p*-NCC_6H_4ZnCl (**6m**) with **1d** led to naphthyridine **7m** in 83% yield (entry 3). Furthermore, heteroarylzinc reagent **6n** reacted smoothly with **1d** to afford naphthyridine **7n** in 73% yield (entry 4). Remarkably, sterically demanding naphthylzinc reagent **6o** was also converted with **1d** to 1,5-naphthyridine **7o** in 47% yield (entry 5).

Table 2. Co-Catalyzed Arylations of Arylzinc Reagents 6a–j with Chloronaphthyridines 1b and 1c



1b-c **7a-j**

FG^1 = H, CF_3
FG^2 = H, OMe, OTBS, OTIPS, OBn, CN, F, CF_3, SMe, Ph, Me

entry	naphthyridine	zinc reagent	product[a], yield (%)
			
1	**1b**	**6a**; X = H	**7a**, 80
2	**1b**	**6b**; X = *p*-Ph	**7b**, 82
3	**1b**	**6c**; X = *p*-CF_3	**7c**, 69
4	**1b**	**6d**; X = *p*-OTBS	**7d**, 72
5	**1b**	**6e**; X = *p*-CN, *m*-F	**7e**, 74
			
6	**1b**	**6f**	**7f**, 79
			
7	**1b**	**6g**	**7g**, 60
			
8	**1c**	**6h**	**7h**, 74
			
9	**1c**	**6i**; X = *p*-OBn	**7i**, 83
10	**1c**	**6j**; X = *o*-OTIPS	**7j**, 61[b]

[a]Isolated yields of analytically pure products. [b]The cross-coupling reaction proceeded at 60 °C for 12 h.

This mild method also allows the coupling of sensitive iodonaphthyridines. Thus, the coupling of zinc reagent **6p** with 4-iodo-1,5-naphthyridine **1e** provided the corresponding 4,8-functionalized 1,5-naphthyridine **7p** in 86% yield (entry 6). Similarly to the bisarylation of dichloronaphthyridine **1a** (Scheme 1), we also examined the Co-catalyzed reaction of 2,4-diiodo-1,5-naphthyridine (**1f**) with *p*-$MeOC_6H_4ZnCl$ (**6l**) and obtained the corresponding bis(anisyl)naphthyridine **7q** in 75% yield (entry 7). Finally, Co-catalyzed cross-coupling of

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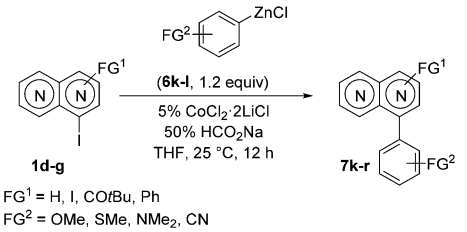
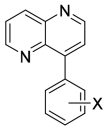
Table 3. Co-Catalyzed Arylations of Arylzinc Reagents 6k and 6l with Iodonaphthyridines 1d–g



entry	naphthyridine	zinc reagent	product[a], yield (%)
			
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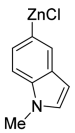
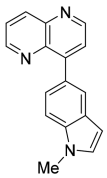
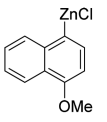
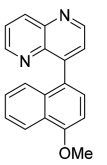
2	**1d**	**6l**; X = *p*-OMe	**7l**, 78
3	**1d**	**6m**; X = *p*-CN	**7m**, 83
			
4	**1d**	**6n**	**7n**, 73
			
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	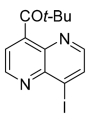	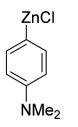	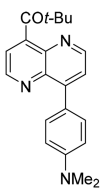
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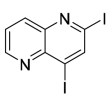
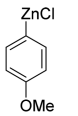
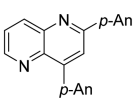
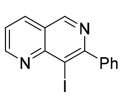
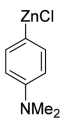
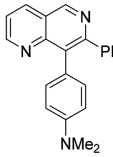
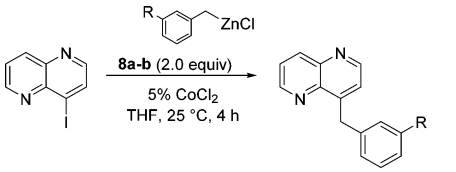
			
7	**1f**	**6l**	**7q**[b], 75
			
8	**1g**	**6p**	**7r**[c], 65

[a]Isolated yields of analytically pure products. [b]2.4 equiv of zinc reagent was used. *p*-An = *p*-MeOC$_6$H$_4$. [c]3.0 equiv of the arylzinc reagent was necessary for complete conversion.

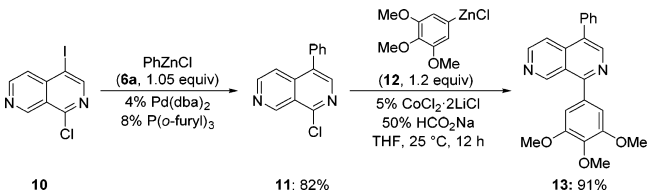
sterically hindered 8-iodo-1,6-naphthyridine (**1g**)[10] with *p*-Me$_2$NC$_6$H$_4$ZnCl (**6p**) furnished naphthyridine **7r** in 65% yield (entry 8). We also found that **1d** smoothly reacts with benzylic zinc reagents.[11] Thus, the couplings of **1d** with benzylzinc chlorides **8a** and **8b** provided 1,5-naphthyridines **9a** and **9b** in 62–75% yield (Scheme 2).

Scheme 2. Co-Catalyzed Cross-Coupling of 4-Iodo-1,5-naphthyridine (1d) with Benzylic Zinc Reagents 8a and 8b



By using mixed halogenated naphthyridines, we observed that 1-chloro-4-iodo-2,7-naphthyridine (**10**) was regioselectively functionalized by stepwise cross-coupling utilizing successive Pd and Co catalyses (Scheme 3). Thus, Pd-catalyzed Negishi

Scheme 3. Regioselective Pd/Co-Catalyzed Cross-Coupling



cross-coupling of **10** with PhZnCl (**6a**) selectively furnished 2,7-naphthyridine **11** in 82% yield. Subsequent Co-catalyzed cross-coupling with arylzinc chloride **12** gave the mixed bisarylated naphthyridine **13** in 91% yield.

Naphthyridine derivatives have previously been applied as fluorescent probes[12] or as ligands for fluorescent complexes.[13] The newly prepared naphthyridines **5b** and **7r** are highly fluorescent in various organic solvents and display strong solvatochromism (Figure 1a,b and Supporting Information (SI) Figures 1–4). We suggest that these phenomena are based on strong interactions between the electron donor NMe$_2$ and the electron-poor naphthyridine moiety as an electron acceptor. While the absolute photoluminescence quantum efficiencies (PLQEs) of **5b** in various solvents are about 20%, the PLQE of **7r** is almost quantitative in nonpolar solvents (toluene, 95 ± 5%; cyclohexane, 93 ± 5%) and drops only slightly in more polar solvents (CHCl$_3$, 81 ± 5%; 1,4-dioxane, 80 ± 5%; THF, 71 ± 5%; see SI Figures 5 and 6 for details). These high emission efficiencies are accompanied by very long excited lifetimes of 3.8 and 12.0 ns for **5b** and **7r**, respectively (Figure 1c,d; see the SI for details).

In summary, we have prepared various novel polyfunctionalized naphthyridines by Co-catalyzed cross-couplings. Alkyl- and arylmagnesium reagents reacted smoothly with chloro-2,7-naphthyridines using CoCl$_2$ (5%). The addition of sodium formate allowed an extension of the range of functionalized N-heterocycles by mild cross-coupling of chloro- and iodonaphthyridines with arylzinc reagents. Two of the new naphthyridines are highly fluorescent (PLQE = 20–95%) with tunable emission from blue to yellow and long excited-state lifetimes from 3.8 to 12.0 ns. Further extensions of organometallic naphthyridine functionalizations are currently underway in our laboratories.

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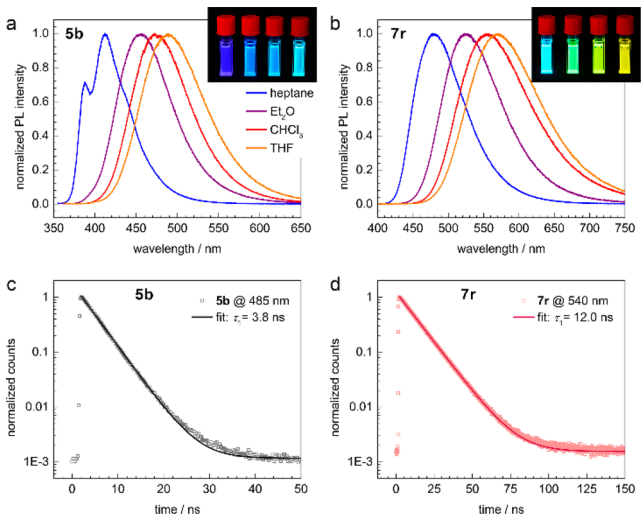

Figure 1. (a, b) PL spectra of compounds **5b** and **7r**, respectively, dissolved in heptane (blue), Et_2O (purple), $CHCl_3$ (red), and THF (orange). The excitation wavelengths were 360 and 390 nm, respectively. The insets show photographs of the solutions under UV illumination. (c, d) Time-correlated single-photon counting (TCSPC) traces of **5b** and **7r** in $CHCl_3$, measured at the peaks of the PL spectra (open symbols). The solid lines are monoexponential fits.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.7b03242.

Experimental details, GC data, melting points, and IR, ^1H and ^{13}C NMR, and mass spectra (PDF)

■ AUTHOR INFORMATION

Corresponding Author

*E-mail: paul.knochel@cup.uni-muenchen.de.

ORCID

Florian Auras: 0000-0003-1709-4384

Paul Knochel: 0000-0001-7913-4332

Notes

The authors declare no competing financial interest.

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