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Rh(III)-Catalyzed Oxidative Coupling of 1,2-Disubstituted Arylhydrazines and Olefins: A New Strategy for 2,3-Dihydro-1*H*-Indazoles

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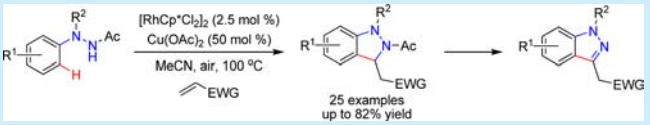
Supporting Information

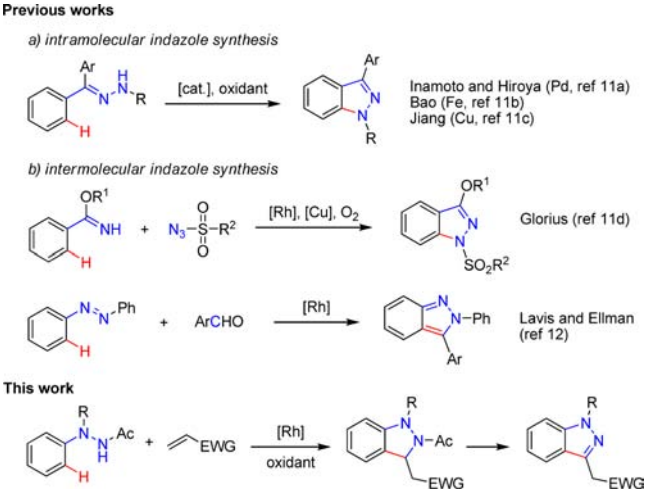
ABSTRACT: A rhodium(III)-catalyzed oxidative olefination of 1,2-disubstituted arylhydrazines with alkenes via sp^2 C−H bond activation followed by an intramolecular aza-Michael reaction is described. This strategy allows the direct and efficient construction of highly substituted 2,3-dihydro-1*H*-indazole scaffolds.



The indazole nucleus is a ubiquitous structural motif found in heterocyclic compounds with a broad spectrum of biological and medicinal applications.[1] In fact, compounds containing the indazole motif are known to exhibit a variety of biological activities, such as high binding affinity for estrogen receptors,[2] inhibition of protein kinases,[3] serotonin 5-HT_2 receptor agonists,[4] HIV protease inhibition,[5] and antitumor activity.[6] The prevalence of indazoles in bioactive molecules has led to the development of many useful methods for their preparation.[7] Surprisingly, however, the 2,3-dihydro-1*H*-indazole scaffold remains virtually unknown and untested, despite its obvious structural resemblance to the indazole ring system.[8] Thus, the 2,3-dihydro-1*H*-indazole class can be a new candidate for the next generation of pharmaceutical molecules.

Transition-metal-catalyzed C−H bond functionalization has attracted much attention owing to its remarkable potential for atom economy and environmental sustainability.[9] In particular, a great deal of effort has been devoted to the formation of various heterocycles via transition-metal-catalyzed C−H functionalization events.[10] In this context, there has been recent progress in the area of indazole synthesis via oxidative annulation protocols (Scheme 1).[11] For example, Inamoto and Hiroya reported the Pd-catalyzed C−H activation of tosylhydrazones followed by intramolecular amination to afford 3-aryl or 3-alkyl indazoles.[11a] Bao[11b] and Jiang,[11c] respectively, demonstrated the Fe- and Cu-catalyzed aerobic C−N bond formation of hydrazones for the formation of indazoles. Glorius described the tandem Rh-catalyzed C−N bond formation and Cu-catalyzed N−N bond formation between arylimidates and organo azides to provide 1*H*-indazoles.[11d] Recently, Lavis and Ellman disclosed an efficient method for the preparation of *N*-aryl-2*H*-indazoles via Rh(III)-catalyzed C−H bond addition of azobenzenes to aldehydes without external oxidants.[12] The past years have witnessed considerable progress in the field of rhodium-catalyzed C−H functionalization reactions.[13] Notably, Rh(III)-catalyzed C−H activation followed by an annulation reaction with carbon−carbon π-bonds has been frequently used as a powerful tool to construct various heterocycles.[14]

Scheme 1. Indazole Synthesis via C−H Functionalization



Recently, we described tandem rhodium-catalyzed oxidative C−C bond formation followed by intramolecular cyclization of acetanilides[15] and *N*-benzyltriflamides[16] with olefins to afford the corresponding indole and isoindoline heterocycles. Our continued efforts in catalytic C−H bond functionalization[17] prompted us to explore the direct coupling reaction of arylhydrazines and olefins. Herein, we present the tandem Rh(III)-catalyzed oxidative olefination and intramolecular aza-Michael reaction of 1,2-disubstituted arylhydrazines with alkenes via sp^2 C−H bond activation to give highly substituted 2,3-dihydro-1*H*-indazoles. To the best of our knowledge, this is

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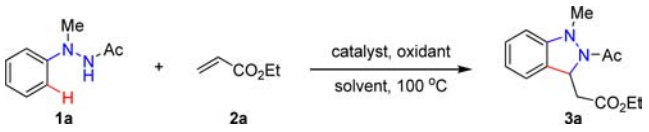
a first report of the direct and catalytic formation of 2,3-dihydro-1*H*-indazoles.

Our investigation was commenced by examining the conversion of *N*′-methyl-*N*′-phenylacetohydrazide (**1a**) to 2,3-dihydro-1*H*-indazole **3a** using a combination of a rhodium catalyst and copper salt as an oxidant (Table 1). To our delight,

Table 1. Selected Optimization for Reaction Conditions[a]



entry	catalyst	oxidant (equiv)	solvent	yield (%)[b]
1	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2·H_2O$ (2)	*t*-AmOH	55
2	$[Ru(p\text{-cymene})Cl_2]_2$	$Cu(OAc)_2·H_2O$ (2)	*t*-AmOH	N.R.
3	$Pd(OAc)_2$	$Cu(OAc)_2·H_2O$ (2)	*t*-AmOH	N.R.
4[c]	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2·H_2O$ (2)	*t*-AmOH	39
5	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2·H_2O$ (2)	DMF	12
6	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2·H_2O$ (2)	DCE	47
7	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2·H_2O$ (2)	THF	65
8	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2·H_2O$ (2)	MeCN	68
9	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (2)	MeCN	73
10	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (1)	MeCN	80
11	**$[RhCp^*Cl_2]_2$**	**$Cu(OAc)_2$ (0.5)**	**MeCN**	**75**
12	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (0.25)	MeCN	57
13[d]	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (0.5)	MeCN	70
14[e]	$[RhCp^*Cl_2]_2$	$Cu(OAc)_2$ (0.5)	MeCN	43

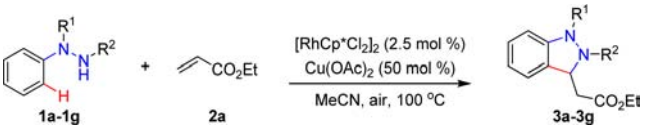
[a]Reaction conditions: **1a** (0.3 mmol), **2a** (0.6 mmol), catalyst (2.5 mol %), oxidant (quantity noted), solvent (1 mL) under air at 100 °C for 20 h in pressure tubes. [b]Isolated yield by flash column chromatography. [c]$AgSbF_6$ (10 mol %) was added as an additive. [d]O_2 gas (1 atm) was used. [e]N_2 gas (1 atm) was used.

the coupling reaction of **1a** and **2a** in the presence of 2.5 mol % of $[RhCp^*Cl_2]_2$ and 2 equiv of $Cu(OAc)_2·H_2O$ in *tert*-amyl alcohol (*t*-AmOH) at 100 °C for 20 h provided the desired product **3a** in 55% yield (Table 1, entry 1). However, other catalysts such as $[Ru(p\text{-cymene})Cl_2]_2$ and $Pd(OAc)_2$ were found to be ineffective in this coupling reaction (Table 1, entries 2 and 3). In addition, the cationic rhodium complex, derived from $[Cp^*RhCl_2]_2$ and $AgSbF_6$, was found to be less effective in the current reaction system (Table 1, entry 4).

After a screening of solvents under otherwise identical conditions, MeCN and THF were found to be highly effective solvents in this coupling reaction, whereas other solvents such as DMF and DCE failed to facilitate high levels of conversion (Table 1, entries 5−8). Further study showed that the $Cu(OAc)_2$ oxidant displayed increased catalytic activity to afford **3a** in 73% yield (Table 1, entry 9). Interestingly, the use of a decreased amount (100 mol %) of $Cu(OAc)_2$ afforded the increased formation of our desired product **3a** in high yield (80%), as shown in entry 10. A further decrease in the amount of oxidant to 50 mol % also provided a comparable yield (75%) (Table 1, entry 11), but a considerable lowering in the formation of product was observed when a catalytic amount (25 mol %) of oxidant was used (Table 1, entry 12). The reaction was then performed under an oxygen and nitrogen atmosphere affording **3a** in 70% and 43% yields, respectively (Table 1, entries 13 and 14), while no formation of **3a** was observed under an air or oxygen atmosphere without $Cu(OAc)_2$. These results indicate that oxygen in the air may play a role as a terminal oxidant in the reaction catalyzed by Cu(II).

Next we examined the influence of *N*,*N*′-protection groups under optimal reaction conditions, and the selected results are outlined in Table 2. 1-Methyl-1-phenylhydrazine (**1b**) did not

Table 2. Screening of *N*,*N*′-Protection Groups[a]



entry	R^1	R^2	product	yield (%)[b]
1	Me	Ac	**3a**	75
2	Me	H	**3b**	N.R.
3	Me	pivaloyl	**3c**	N.R.
4	Me	Bz	**3d**	N.R.
5	Et	Ac	**3e**	66
6	Bn	Ac	**3f**	27
7[c]	Ph	Ac	**3g**	23

[a]Reaction conditions: **1a−1g** (0.3 mmol), **2a** (0.6 mmol), $[RhCp^*Cl_2]_2$ (2.5 mol %), $Cu(OAc)_2$ (50 mol %), MeCN (1 mL) under air at 100 °C for 20 h in pressure tubes. [b]Isolated yield by flash column chromatography. [c]C2-Olefinated compound **3gg** on a phenyl moiety at R^1 position was also obtained in 18% yield (see Supporting Information for details).

yield the desired product **3b** (Table 2, entry 2). *N*′-Methyl-*N*′-phenylhydrazides **1c** and **1d** with a pivaloyl or benzoyl group at the R^2 position were found to be unreactive (Table 2, entries 3 and 4). 2-Ethyl-substituted 2-phenylacetohydrazide **1e** was compatible with the reaction conditions (Table 2, entry 5), whereas benzyl- or phenyl-substituted hydrazides **1f** and **1g** were far more ineffective in this coupling reaction (Table 2, entries 6 and 7).

With the optimized reaction conditions in hand, the scope and limitation of *N*′-methyl-*N*′-arylacetohydrazides were examined, as shown in Scheme 2. The coupling of ethyl acrylate (**2a**) and *N*′-methyl-*N*′-arylacetohydrazides **1h−1l** with electron-donating and -withdrawing groups at the *para*-position on the aromatic ring was found to be favored in the oxidative olefination and subsequent intramolecular cyclization to afford the corresponding products **3h−3l** in moderate to good yields. Particularly noteworthy were the monoselectivity of olefination at two *ortho*-C−H bonds and the tolerance of the reaction conditions to the bromo and chloro moieties, which provides a versatile synthetic handle for further functionalization of the products. This reaction was also compatible with *meta*-substituted arylacetohydrazides **1m−1o**, and all reactions preferentially occurred at the less hindered position furnishing the corresponding products **3m−3o** as a single regioisomer. In addition, *ortho*-substituted compounds also participated in the oxidative coupling to furnish **3p** and **3q** with slightly decreased reactivity.

To further explore the substrate scope and limitation of this process, a broad range of olefins **2b−2k** were screened to couple with **1a**, as shown in Scheme 3. To our pleasure, acrylates **2b−2f** and acrylamide **2g** proved to be good substrates for this transformation affording the corresponding products **4b−4g** in high yields. In contrast, acrylonitrile (**2h**) and but-3-en-2-one (**2i**) were coupled with **1a** with significantly decreased reactivity under the optimal reaction conditions. After further optimization, we found that an increased loading

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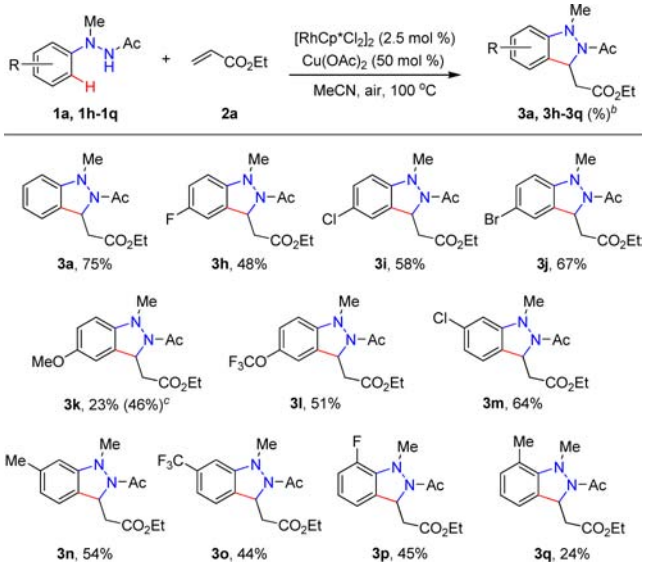
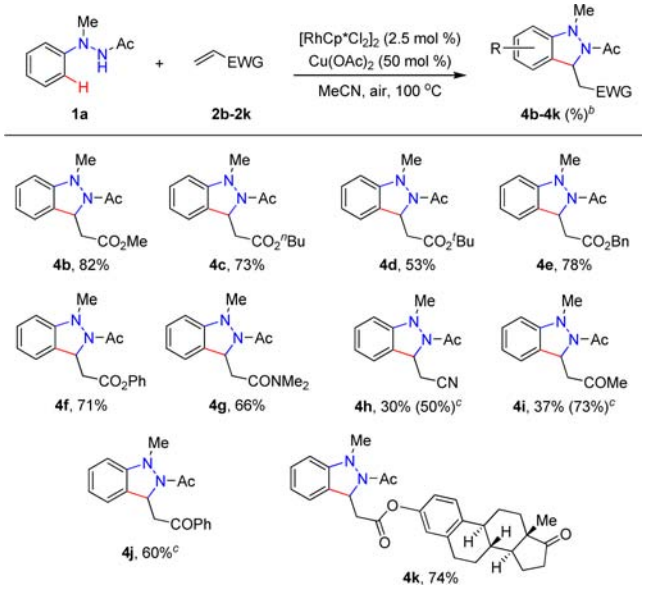
Scheme 2. Scope of Hydrazides[a]



[a]Reaction conditions: **1a** and **1h−1q** (0.3 mmol), **2a** (0.6 mmol), $[RhCp^*Cl_2]_2$ (2.5 mol %), $Cu(OAc)_2$ (50 mol %), MeCN (1 mL) under air at 100 °C for 20 h in pressure tubes. [b] Isolated yield by flash column chromatography. [c] THF was used as a solvent.

Scheme 3. Scope of Olefins[a]

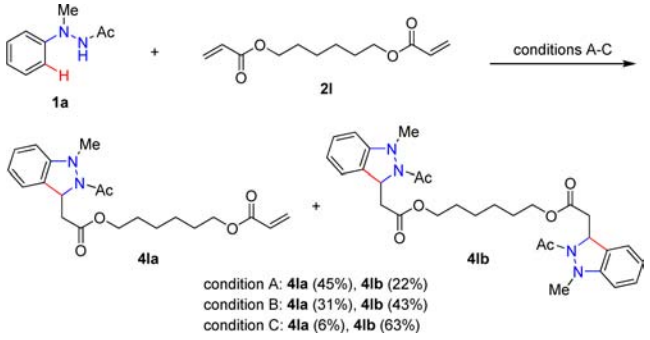


[a]Reaction conditions: **1a** (0.3 mmol), **2b−2k** (0.6 mmol), $[RhCp^*Cl_2]_2$ (2.5 mol %), $Cu(OAc)_2$ (50 mol %), MeCN (1 mL) under air at 100 °C for 20 h in pressure tubes. [b] Isolated yield by flash column chromatography. [c] Olefin (0.9 mmol), $Cu(OAc)_2$ (100 mol %), THF (1 mL).

of $Cu(OAc)_2$ in THF solvent provided our desired products **4h−4j** in good to high yields. Finally, this protocol offers the possibility of late-stage functionalization of biologically active compounds that contain the olefin functional groups. For example, acrylate **2k** with an estrogen scaffold was smoothly converted to **4k** in 74% yield.

To further evaluate the scope of this process, the coupling of diacrylate **2l** with **1a** was examined, as shown in Scheme 4. As expected, diacrylate **2l** was converted to monofunctionalized

Scheme 4. Oxidative Coupling of Diacrylate[a]

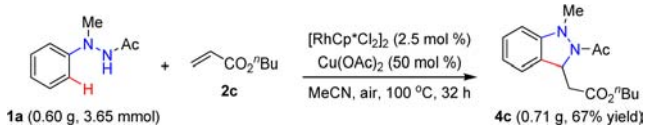


[a]Condition A: **1a** (0.75 mmol), **2l** (0.3 mmol), $[RhCp^*Cl_2]_2$ (2.5 mol %), $Cu(OAc)_2$ (50 mol %), MeCN (1 mL) under air at 100 °C for 20 h in pressure tubes. Condition B: **1a** (0.75 mmol), **2l** (0.3 mmol), $[RhCp^*Cl_2]_2$ (5 mol %), $Cu(OAc)_2$ (100 mol %), MeCN (1 mL) under air at 100 °C for 20 h in pressure tubes. Condition C: **1a** (0.9 mmol), **2l** (0.3 mmol), $[RhCp^*Cl_2]_2$ (5 mol %), $Cu(OAc)_2$ (300 mol %), MeCN (1.5 mL) under air at 100 °C for 40 h in pressure tubes.

compound **4la** and bis-functionalized compound **4lb** in 45% and 22% yields, respectively, under the standard reaction conditions. In addition, upon use of increased amounts of **1a**, rhodium catalyst, and copper salt, the bis-functionalized 2,3-dihydro-1*H*-indazole **4lb** was obtained in 63% yield.

To highlight the robustness and practicality of the preparation of 2,3-dihydro-1*H*-indazole, we successfully scaled the reaction to 3.65 mmol and obtained 0.71 g of **4c** after 32 h in 67% isolated yield (Scheme 5). In addition, we performed an

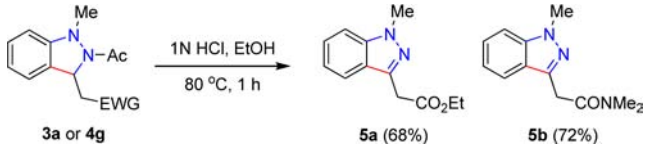
Scheme 5. Scale-up Experiment



intermolecular competition reaction between acrylate **2a** and acrylamide **2g** to afford the corresponding products with a 1.5:1 ratio in 64% combined yield (see Supporting Information for details).

Finally, we were pleased to find that an acetyl protecting group was efficiently removed. Thus, acid-mediated hydrolysis was successfully applied to compounds **3a** and **4g**, providing access to 1*H*-indazoles **5a** and **5b** in high yields (Scheme 6).

Scheme 6. Removal of *N*-Protection Group



In conclusion, we have disclosed a highly efficient method for the preparation of 2,3-dihydro-1*H*-indazoles via the rhodium-catalyzed oxidative alkenylation of 1,2-disubstituted arylhydrazines with olefins and subsequent intramolecular cyclization. These transformations have been applied to a wide range of substrates and typically proceed with excellent levels of chemoselectivity as well as with high functional group tolerance. Furthermore, this protocol allows the generation of

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an array of 1*H*-indazoles, which are known as biologically active scaffolds.

■ ASSOCIATED CONTENT

Supporting Information

Experimental procedures, characterization data, and ^{1}H and ^{13}C NMR spectra for all compounds. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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