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Rhodium-Catalyzed C-H Functionalization of Indoles with Diazo Compounds: Synthesis of Structurally Diverse 2,3-Fused Indoles

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Abstract. A Rhodium-catalyzed C2-H functionalization of indoles with diazo compounds, followed by intramolecular nucleophilic addition to C=O or C=C bonds, is reported for divergent synthesis of 2,3-fused indoles. Besides acceptor/acceptor diazo compounds, donor/acceptor diazo compounds are broadly tolerated, giving various 2,3-fused indoles with perfect diastereocontrol. Notably, a selective C-H dialkylation reaction at C2 and C7 position of indoles has also been developed by simply changing the reaction conditions. This environmentally benign transformation proceeds under mild conditions and gives dinitrogen as the only by-product.

Keywords: Cp*Rh(III) catalyst; C-H activation; Diazo compounds; 2,3-fused indoles

The indole motif is prevalent in a variety of bioactive natural products, pharmaceuticals and agrochemicals.[1] Among them, 2,3-Fused indoles have received considerable attention due to their interesting biological activities (Figure 1).[2] Although several methods have been developed for the synthesis of this popular scaffold, [3] the development of new approaches that allow rapid establishment of these scaffolds in simple operation from readily available precursors is still highly desirable.

Over the past several years, the diazo compounds have been widely used as powerful cross-coupling partners for transition-metal-catalyzed carbene transformations. [4] However, intermolecular aromatic C(sp^2)−H insertion into diazo compounds has very limited precedent. Until recently, a significant breakthrough has been made by Yu, [5] who reported the first intermolecular insertion of aromatic C(sp^2)−H bonds into α-diazocarbonyl compounds via a Rh(III)-catalyzed C-H activation (Scheme 1a). [6] Since then, other groups[7] and our group[8] have displayed the successful exploration of Rh(III)-catalyzed C-H functionalizations with diazo compounds.

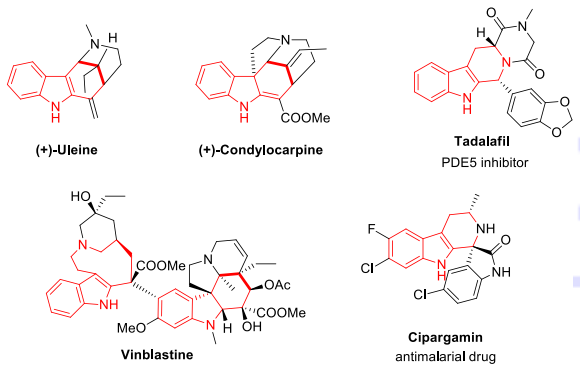

Figure 1. Representative biologically active 2,3-fused indoles.

Very recently, our group reported a Rh(III)-catalyzed cyclization reaction of nitrones with diazo compounds, where the C(sp^3)-Rh intermediate is not protonated and undergoes an intramolecular addition to the polarized C=N bond, thus producing various N-hydroxyindolines (Scheme 1b). [8c] In this context, with our continuing interest in the sustainable organic synthesis of fused indoles, [9] we herein report a Rh(III)-catalyzed C2-H functionalization of indoles with diazo compounds, where the resulting C(sp^3)-Rh intermediate undergoes an intramolecular addition reaction to the C=O or C=C bonds, providing various 2,3-fused indoles (Scheme 1c). This reaction proceeds under mild conditions, giving benign N_2 as the only byproduct.

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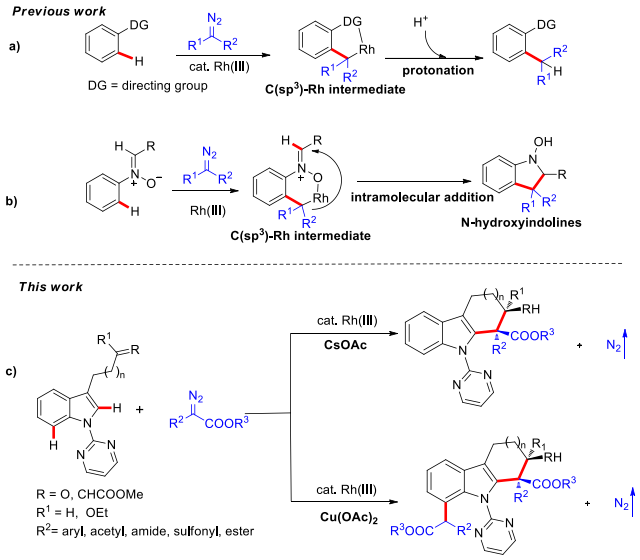
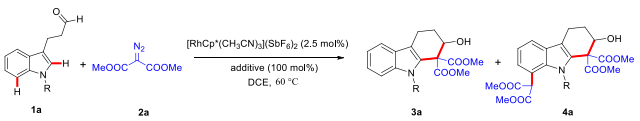


Scheme 1. Rh(III)-catalyzed C-H functionalization with diazo compounds.

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



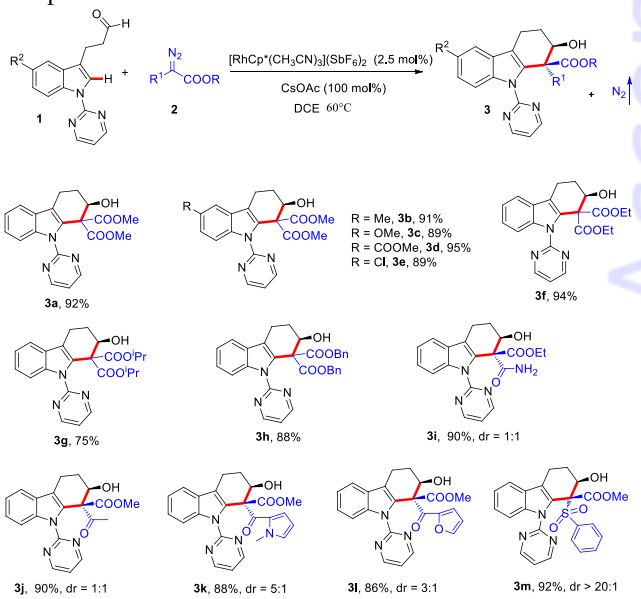
entry	R	additive	yield of **3a** (%)	yield of **4a** (%)
1	$(CH_3)_2NCO$	-	0	0
2	Ac	-	0	0
3	2-pyrimidyl	-	43	12
4	2-pyrimidyl	PivOH	< 10	16
5	2-pyrimidyl	AgOAc	75	< 10
6	2-pyrimidyl	CsOAc	92	<2
7[b]	2-pyrimidyl	-	< 5	42
8[c]	2-pyrimidyl	$Cu(OAc)_2$	<2	82

[a] Reaction conditions: 0.2 mmol of **1a**, **2a** (1.5 equiv.), catalyst (2.5 mol %), additive (100 mol %) and DCE (2 mL) at 60°C for 8 h in a sealed tube. Yield isolated by column chromatography. [b] Using $[Cp^*RhCl_2]_2$ (2.5 mol %) and $AgSbF_6$ (10 mol %) as catalyst [c] 0.2 mmol of **1a**, **2a** (4 equiv.), $[RhCp^*Cl_2]_2$ (2.5 mol %), $AgSbF_6$ (10 mol %), $Cu(OAc)_2$ (20 mol %) and DCE (2 mL) at 80°C for 16 h in a sealed tube.

The initial experiments were performed with a variety of *N*-substituted indoles (**1a**) and diazomalonate (**2a**) in the presence of 2.5 mol% $[Cp^*Rh(MeCN)_3](SbF_6)_2$ in 1,2-dichloroethane (Table 1, entry 1). The choice of indole protecting group was found to be crucial (Table 1, entries 1-3), with 2-pyrimidyl being optimal (Table 1, entry 3). To our delight, the coupling reaction occurred smoothly to provide the desired product **3a** and di-alkylation product **4a** with a ratio of ca. 3:1 (entry 3). Encouraged by this result, various additives were next screened (entries 4-6). The addition of acid decreased the yield of product **3a** (entry 4). Pleasingly, the situation changed significantly when adding some acetate additives (entries 5-6), with CsOAc being optimal to afford **3a** as the sole product in 92% yield, as shown in entry 6. Interestingly, when employing $[Cp^*RhCl_2]_2$ (2.5 mol %) and $AgSbF_6$ (10 mol %) as catalyst, **4a** could be obtained as the main product in 42% yield (entry 7). To our delight, introduction of $Cu(OAc)_2$ as the additive further improved the yield of **4a** to 82% (entry 8).

With the optimized reaction conditions in hand, we embarked on the investigation of the scope of this annulation reaction. As shown in scheme 2, indoles containing both electron-donating (**3b**, **3c**) and -withdrawing (**3d**, **3e**) groups were readily coupled diazomalonate (**2a**) to provide the corresponding products in good to excellent yields. Some important functional groups such as halogen and ester, were remarkably compatible, offering the opportunity for further synthetic transformations. The scope of diazo coupling partners has also been established and various α-diazomalonates underwent this coupling reaction smoothly to provide products **3f**-**3h** in satisfactory yields. In addition to α-diazomalonates, other acceptor-acceptor diazo compounds with one ester group replaced by another electron-withdrawing group such as amide (**3i**), acetyl (**3j**-**3l**), sulfonyl (**3m**), were also found to be favorable in current transformation, giving the corresponding products in excellent yields. It is noteworthy that aryl substituted α-diazoacetylacetates (**3k**, **3l**) and sulfonylacetate (**3m**) exhibited good diastereoselectivity (dr up to >20:1).

Scheme 2. Scope of indoles and acceptor/acceptor diazo compounds.

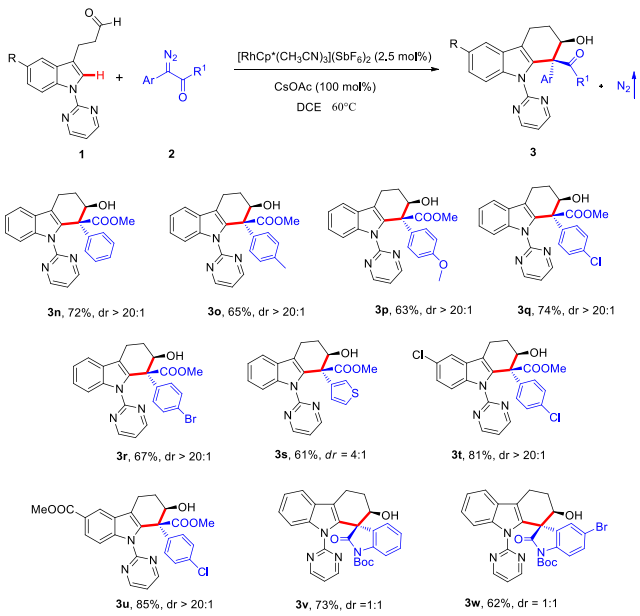


Reaction conditions: 0.2 mmol of **1**, **2** (1.5 equiv), $[RhCp^*(CH_3CN)_3](SbF_6)_2$ (2.5 mol %), CsOAc (100

mol %) and DCE (2 mL) at 60°C for 8 h. Yield isolated by column chromatography.

Encouraged by these results, we further examined donor/acceptor diazo compounds which were often less reactive (Scheme 3).[10] Pleasingly, donor/acceptor diazo compounds were broadly tolerated with reasonable scope (**3n-3w**), irrespective of the electronic nature of the substituents on the phenyl ring (**3o-3u**). Cyclic diazo substrates also delivered the desired spiro compounds in high yields under the reaction conditions (**3v**, **3w**). Notably, all the products showed perfect diastereocontrol (dr > 20:1), except **3s**, **3v** and **3w** (dr = 1:1). The configuration was verified via NOESY spectra (see supporting information for detail). The good diastereocontrol may be understood by a favored six-membered cyclic transition state **AA** when the O-Rh intermediate undergoes the intramolecular aldol reaction.[11]

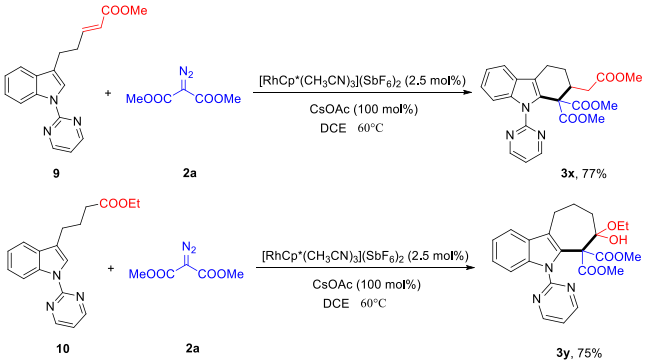
Scheme 3. Scope of donor/acceptor diazo-compounds.[a]



[a] Reaction conditions: 0.2 mmol of **1**, 0.3 mmol of **2**, $[RhCp^*(CH_3CN)_3](SbF_6)_2$ (2.5 mol %), CsOAc (100 mol %) and DCE (2 mL) at 60°C for 8 h. Yield isolated by column chromatography.

To further expand this coupling/annulation reaction with diazo compounds, we wondered whether a intramolecular Michael addition of the C(sp^3)–Rh intermediate to acrylate might be possible (Scheme 4). To our delight, the indole with acrylate skeleton (**9**) delivered the 2,3-fused indole **3x** in 77% yield under the standard reaction conditions. More significantly, the C(sp^3)-Rh intermediate could undergo an intramolecular Grignard-type addition reaction to ester, providing the seven-membered 2,3-fused indole **3y** in good yield.

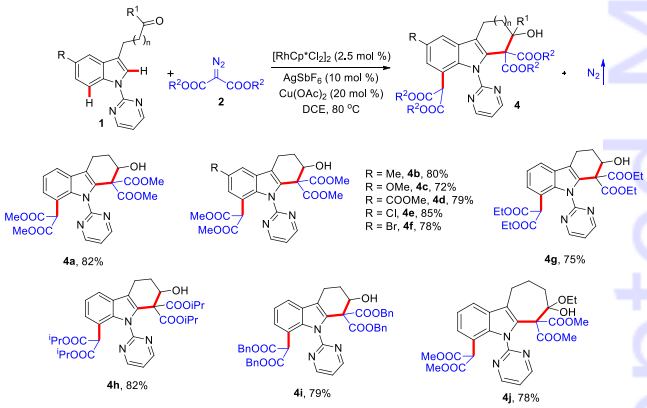
Scheme 4. Synthesis of 2,3-fused indoles.[a]



[a] Reaction conditions: 0.2 mmol of **9** or **10**, **2a** (1.5 equiv), $[RhCp^*(CH_3CN)_3](SbF_6)_2$ (2.5 mol %), CsOAc (100 mol %) and DCE (2 mL) at 60°C for 8 h.

Finally, the scope of the double C-H activation/alkylation was also examined (Scheme 5). Various substrates containing both electron-donating (**4b**, 4c) and electron-withdrawing groups (**4d-4f**) at C5 position of the indole ring were successfully coupled with dimethyl diazomelonate, providing C2, C7-dialkylation products **4b-f** in good to excellent yields. Besides dimethyl diazomelonate, ethyl, isopropyl and benzyl diazomelonates also worked well to provide the corresponding dialkylation products **4g-i** in good yields.

Scheme 5. Rh-catalyzed C2, C7-dialkylation of indole C-H bonds.[a]



[a] Reaction conditions: 0.2 mmol of **1**, 0.8 mmol of **2**, $[RhCp^*Cl_2]_2$ (2.5 mol %), $AgSbF_6$ (10 mol %), $Cu(OAc)_2$ (20 mol %) and DCE (2 mL) at 80°C for 16 h.

To illustrate the synthetic utility of this reaction, treatment of **3f** with DIPEA resulted in decarbonylation to afford **5f** in 72% yield (Scheme 6a). In addition, **3f** could undergo a decarbonylation/elimination of hydroxyl group/oxidation to give carbazole derivative **6f** in 67 yield when treating **3f** with LiOH in EtOH and H_2O at 60 °C (Scheme 6b). Finally, the pyrimidyl group could be successfully removed by the treatment of NaOEt in DMSO at 100 °C for 3 h to give **7f** in 75% yield.

Scheme 6. Transformation of product **3f**.

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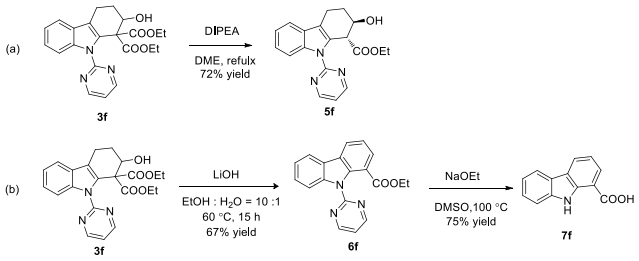
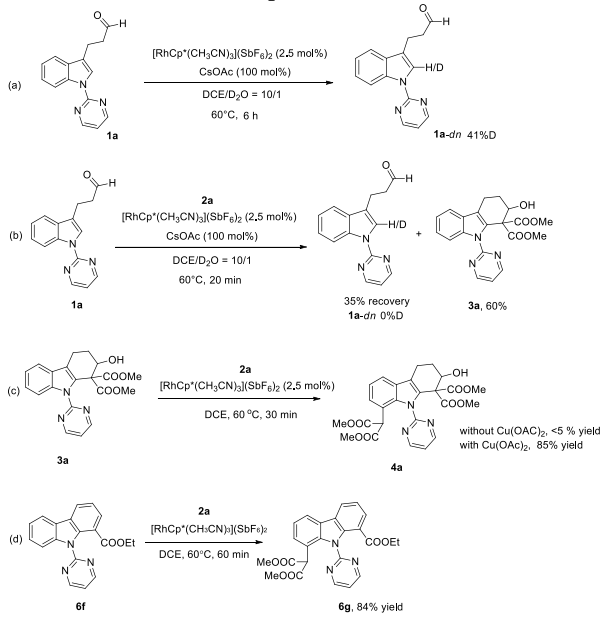


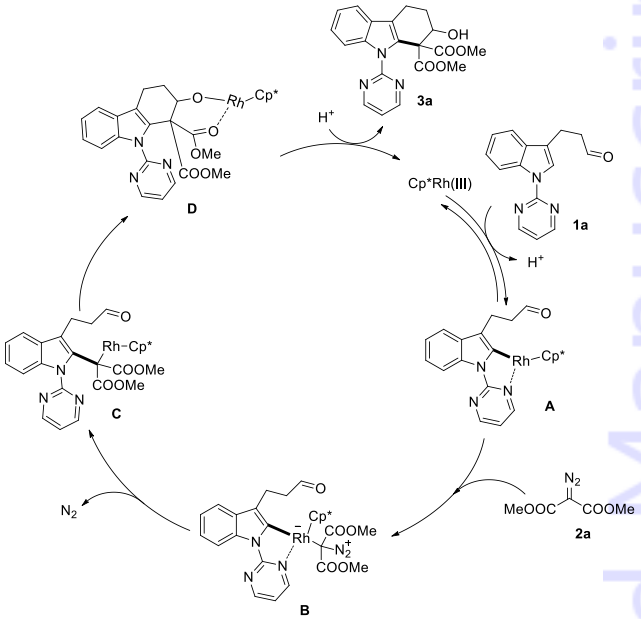
Several experiments were carried out to obtain a more mechanistic insight (scheme 7). First, a H/D scrambling was observed at the C2-position of indole when **1a** was reacted with the $[RhCp^*(CH_3CN_3)](SbF_6)_2$ catalyst in DCE and D_2O in the absence of **2a**, indicating the C–H bond cleavage is reversible (Scheme 7a). However, in the presence of **2a**, no H/D scrambling was observed in the recovered **1a**, indicating that C–H functionalization is much faster than the deuteration (Scheme 7b). To explain why $Cu(OAc)_2$ facilitate the second C7-H functionalization to form the dialkylation product **4a**, several control experiments were carried out. First, treatment of **3a** with diazomalonate in the presence of $[Cp^*Rh(MeCN)_3](SbF_6)_2$ at 60 °C for 30 min gave only a trace amout of **4a** (<5% yield) and addition of $Cu(OAc)_2$ as an additive gave **4a** in 85% yield (Scheme 7c). However, **6f** without a hydroxyl group can undergo a Rh(III)-catalyzed C7-H alkylation with diazomalonate smoothly to give **6g** in good yield in the absence of $Cu(OAc)_2$ (Scheme 7d). Based on the above data, we speculated that the hydroxyl group of **3a** may react with Rh(III) catalyst to form stable O-Rh species **D** (See Scheme 8 for structure), thus inhibiting the second C7-H functionalization to form **4a** and additon of $Cu(OAc)_2$ may lead to a transmetalation of the intermidiate **D** with the copper salt, thus facilitating to release the active Rh(III) species to catalyze the C7-H alkylation.[12]

Scheme 7. Control experiments.



Based on the literature precedents,[5-8] we tentatively propose the following reaction mechanism (Scheme 8). First, a five-membered cyclometalated Rh(III) complex A is formed via a Rh(III)-catalyzed $C(sp^2)$–H bond cleavage. Coordination of the diazo compound with A gives the intermediate B which undergoes an intramolecular 1,2-migratory insertion of the aryl group to afford the intermediate C. Subsequent intramolecular nucleophilic addition to aldehyde afford the rhodium alkoxide D which undergoes a protonation to yield the product **3a** and regenerate the rhodium catalyst.

Scheme 8. Proposed mechanism.



In summary, an efficient Rh(III)-catalyzed C2-alkylation of indoles with diazo compounds, followed by intramolecular addition reaction, has been developed for the synthesis of various 2,3-fused indoles. Besides acceptor/acceptor diazo compounds, donor/acceptor diazo compounds are broadly tolerated, providing various 2,3-fused indoles with perfect diastereocontrol. Notably, a C-H dialkylation reaction at C2 and C7 position of indoles has also been developed by simply changing the reaction conditions. This transformation proceeds under mild conditions, obviates the need for oxidants, and releases N_2 as the single byproduct, thus providing an environmentally benign method of 2,3-fused indole synthesis.

Experimental Section

General procedure for the synthesis of compound 3: To a dram screw-cap vial mixture of 0.2 mmol of **1**, $[RhCp^*(MeCN)_3](SbF_6)_2$ (2.5 mol%), CsOAc (100 mol %), DCE (2 mL) and 0.3 mmol of diazomalonate (**2a**) at 60 °C under Ar for 8 h. Then the reaction is cooled to room temperature, the solvent was removed and the residue was

purified by flash chromatography on silica gel (petroleum ether/EtOAc = 3:1) to give the desired product **3a** in yield 92%.

General procedure for the synthesis of compound 4: To a dram screw-cap vial mixture of 0.2 mmol of **1**, $[RhCp^*Cl_2]_2$ (2.5 mol %), $AgSbF_6$ (10 mol %), $Cu(OAc)_2$ (20 mol %), DCE (2 mL) and 0.8 mmol of diazomalonate (**2a**) at 80 °C under Ar for 16 h. Then the reaction is cooled to room temperature, the solvent was removed and the residue was purified by flash chromatography on silica gel (petroleum ether/EtOAc = 1:1) to give the desired product **4a** in yield 82%.

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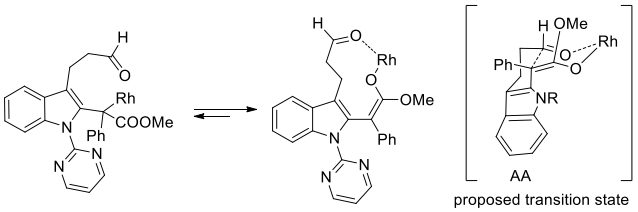
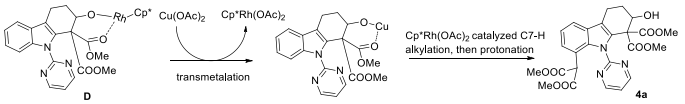
10 Davies has defined diazo compounds as donor, acceptor, or both. See: D. Morten, H. M. L. Davies, *Chem. Soc. Rev.* **2011**, *40*, 1857.

11



12



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COMMUNICATION

Rhodium-Catalyzed C-H Functionalization of Indoles with Diazo Compounds: Synthesis of Structurally Diverse 2,3-Fused Indoles

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