

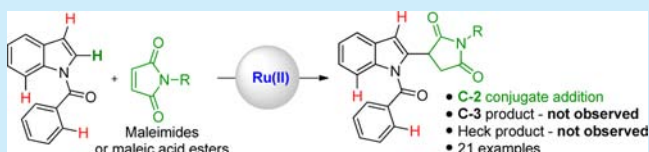
# Site-Selective Addition of Maleimide to Indole at the C-2 Position: Ru(II)-Catalyzed C–H Activation

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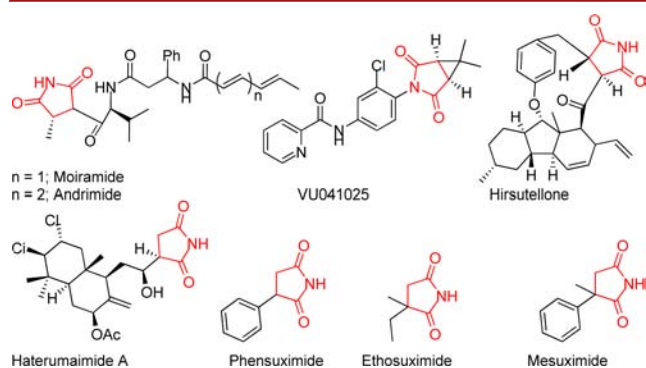
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**S** Supporting Information

**ABSTRACT:** Synthesis of 3-(indol-2-yl)succinimide derivatives is presented using a directing group strategy. Selective functionalization of C-2 in the presence of highly reactive C-3 in indole derivatives has been achieved. A conjugate addition product instead of Heck-type product has been brought about by careful selection of the alkene partner (maleimides and maleate esters) such that a  $\beta$ -hydride elimination is avoided.



The succinimide class of organic molecules are important intermediates in large molecule synthesis.<sup>1</sup> Derivatives of succinimide rings are structural units found in many natural products and clinical drug candidates, indicating that molecules of this class possess a wide range of biological and pharmaceutical activities (see Figure 1).<sup>2</sup> Moreover, succinimides can be easily

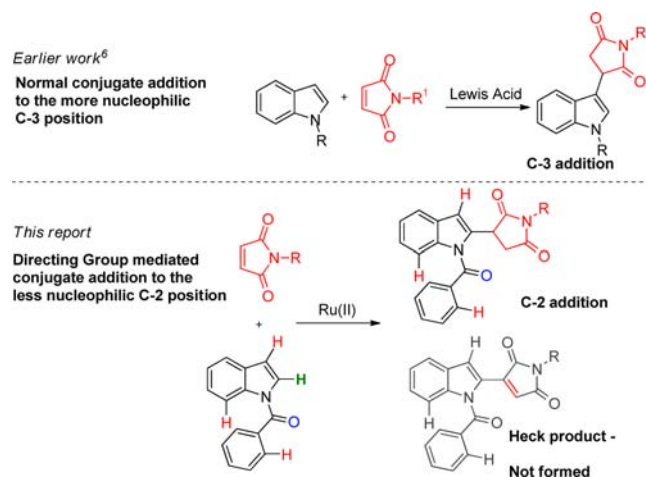


**Figure 1.** Succinimide rings in various natural products, drug candidates, and pharmaceutically active compounds.

reduced into 5-membered pyrrolidine rings,  $\gamma$ -lactams and lactams, which by themselves are useful natural product motifs.<sup>3</sup> The indole ring is abundant among natural products, pharmacologically active compounds, and many commercially marketed drugs.<sup>4</sup> The importance of developing methods to synthesize indole derivatives can never be overestimated, as indicated by the large number of reports appearing consistently in journals from the past several decades.<sup>5</sup>

Synthesis of succinimides, via conjugate addition using the high reactivity of C-3, is known in the literature.<sup>6</sup> However, such conjugate addition at the C-2 position of indole in the presence of an unblocked C-3 position is difficult to achieve. To overcome this problem, we envisaged the use of C–H activation methods.<sup>7,8</sup> Such selective functionalization is possible by using a directing group strategy with transition-metal catalysts (Scheme 1).<sup>9</sup>

## Scheme 1. Directing Group Mediated Conjugate Addition



Many examples of directing groups and various catalytic systems to effect such transformations are well-known in the literature;<sup>9</sup> however, applications toward selective functionalization of indole moieties remain rare.<sup>10</sup> When used as coupling partners with Ru(II) catalysts, alkenes typically result in Heck-type product formation by a  $\beta$ -hydride elimination.<sup>11</sup> We hypothesized that alkenes that do not furnish a “syn planar”  $\beta$ -hydrogen to the metal would not have the ability to undergo  $\beta$ -hydride elimination and, therefore, would lead to conjugate addition products.<sup>12</sup> In continuation of our previous work<sup>13</sup> on the selective functionalization of indole, herein we report the C-2 succinimidation of *N*-benzoylindole derivatives using [Ru(*p*-cymene)Cl<sub>2</sub>]<sub>2</sub> catalyst.

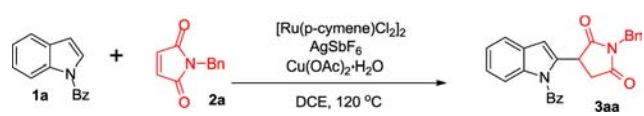
The optimization studies began with *N*-benzoylindole (**1a**) and *N*-benzylmaleimide (**2a**) as the model substrates. The first reaction between **1a** (0.2 mmol) and **2a** (2 equiv) was performed in the presence of 5 mol % of [Ru(*p*-cymene)Cl<sub>2</sub>]<sub>2</sub> catalyst, 20

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mol % of  $\text{AgSbF}_6$ , and 1.0 equiv of  $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$  in DCE (2 mL) at 80 °C to yield the desired product **3aa** in 44% NMR yield (entry 1, Table 1). By decreasing the amount of **2a** to 1.5 equiv,

Table 1. Optimization Studies<sup>a</sup>



entry	additive (equiv)	solvent/temp (°C)	yield (%) <sup>b</sup>
1	none	DCE/80	44
2 <sup>c</sup>	none	DCE/80	40
3	none	DCE/100	62
4	none	DCE/120	65
5	PivOH (10)	DCE/120	40
6	AcOH (10)	DCE/120	80
7	AcOH (5)	DCE/120	60
8	none	AcOH/DCE /120	56
9	H <sub>2</sub> O (10)	DCE/120	62
10 <sup>d</sup>	AcOH (10)	DCE/120	70
<b>11<sup>e</sup></b>	<b>AcOH (10)</b>	<b>DCE/120</b>	<b>84</b>
Deviation from above standard conditions			
12	CH <sub>3</sub> CN or DMF as solvent instead of DCE		nd
13	DCM as solvent at 80 °C		50
14	CHCl <sub>3</sub> as solvent		46
15	No $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$ and no AcOH		nd
16	No $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$		15
17	50 mol % $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$		30
18	$[\text{Rh}(\text{C}_5\text{Me}_5)_2\text{Cl}_2]_2$ instead of $[\text{Ru}(\text{p-cymene})\text{Cl}_2]_2$		65

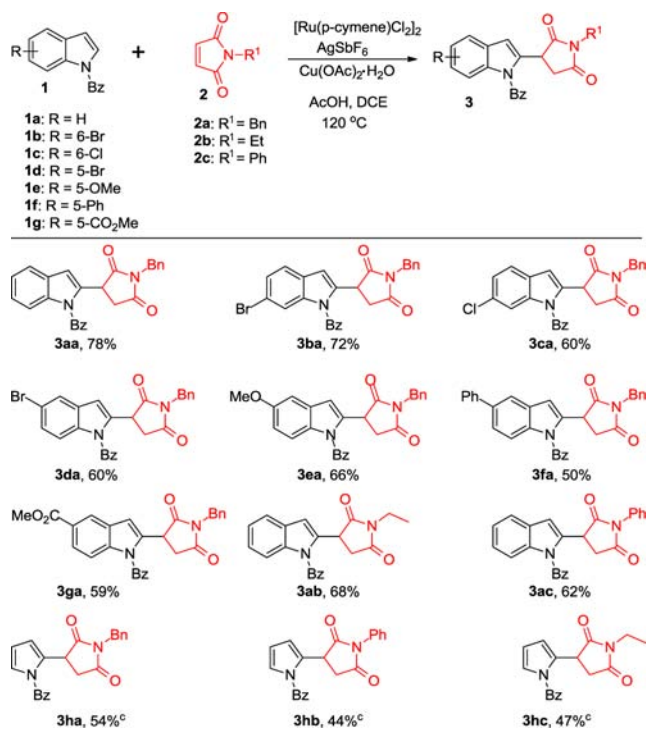
<sup>a</sup>Reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol),  $[\text{Ru}(\text{p-cymene})\text{Cl}_2]_2$  (5 mol %),  $\text{AgSbF}_6$  (20 mol %),  $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$  (1 equiv), DCE (2 mL), 24 h. <sup>b</sup>NMR yields using terephthalaldehyde as internal standard. <sup>c</sup>1.5 equiv of **2a**. <sup>d</sup>10 mol % of Ru. <sup>e</sup>Under argon atmosphere for 24 h. nd, not detected.

we found that the yield of **3aa** dropped to 40%. Increasing the temperature of the reaction to 100 °C led to a yield of 62% (entry 3, Table 1). Further increase of the temperature to 120 °C resulted in a marginal increase in the yield to 65% (entry 4). In order to improve the yield of the product **3aa**, the effect of various additives was examined. Use of pivalic acid (10 equiv) led to a decrease in the yield (40%, entry 5) of the product, whereas use of acetic acid (10 equiv) led to an increase in the yield to 80% (entry 6). A decrease in the amount of AcOH to 5 equiv also led to a decrease of the yield of **3aa** (60%, entry 7). Using AcOH as a cosolvent was detrimental to the outcome of the reaction (56%, entry 8), whereas water as an additive in place of AcOH resulted in decreased yields (62%, entry 9). Increasing the catalyst loading to 10 mol % decreased the yield to 70% (entry 10). The reaction conditions of entry 6 were found to give an inconsistent yield. This problem was circumvented by performing the reaction under inert conditions and refraining from opening the reaction vial until 24 h had passed (84%, entry 11). Screening a variety of solvents such as CH<sub>3</sub>CN, DMF, DCM, or CHCl<sub>3</sub> did not result in any improvement (entries 12–14). A few control studies (entries 15–17) indicate that  $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$  is essential for this redox-neutral reaction. However, its role is uncertain, and we believe it is a source of acetate ion and also activates the electrophile by Lewis acid catalysis.

On the basis of these screening studies, the standard reaction condition was established (entry 11, Table 1), and an exploration of substrate scope was undertaken with a variety of indole

derivatives (**1**) as well as maleimide derivatives (**2**) (Scheme 2). Halogenated derivatives of *N*-benzoyl indole (**1b**, **1c**, and **1d**)

Scheme 2. Substrate Scope with Maleimides<sup>a,b</sup>



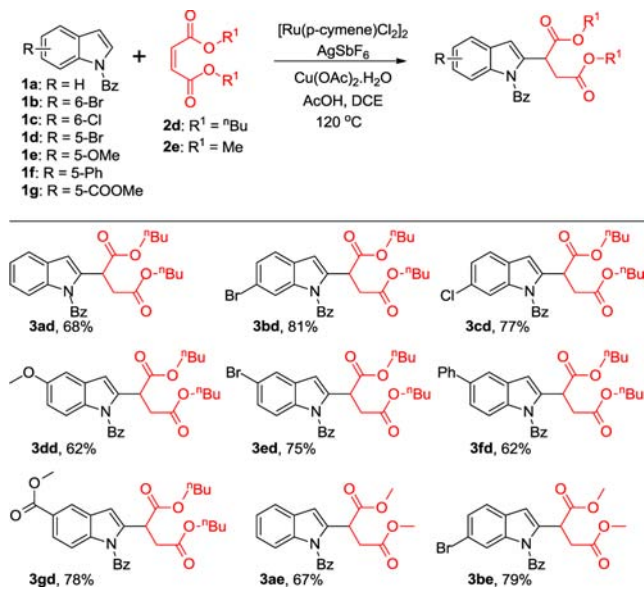
<sup>a</sup>Reaction conditions: *N*-benzoylindole derivative (**1** equiv), maleimide (**2** equiv),  $[\text{Ru}(\text{p-cymene})\text{Cl}_2]_2$  (5 mol %),  $\text{AgSbF}_6$  (20 mol %),  $\text{Cu}(\text{OAc})_2 \cdot \text{H}_2\text{O}$  (1 equiv), AcOH (10 equiv), DCE (2 mL), 120 °C, 24 h. <sup>b</sup>Isolated yields. <sup>c</sup> $[\text{Rh}(\text{C}_5\text{Me}_5)_2\text{Cl}_2]_2$  was used instead of  $[\text{Ru}(\text{p-cymene})\text{Cl}_2]_2$ .

when reacted with *N*-benzylmaleimide (**2a**) furnished the requisite products (**3ba**, **3ca**, and **3da**) in good yields without any loss of the halogen atom. Electron-rich 5-methoxy derivative **1e** underwent the reaction with **2a** in a smooth manner to furnish the desired product **3ea** in 66% yield. The electron-neutral derivative (5-phenyl) of *N*-benzoylindole, **1f**, also reacted with **2a** to yield the corresponding product **3fa** in moderate yield (50%). The electron-deficient (5-methoxycarbonyl) derivative of *N*-benzoylindole (**1g**) also underwent a smooth reaction to form the corresponding product (**3ga**) in good yields without any trace of product obtained by the competing reaction with the ester as directing group. Changing the maleimide derivative from *N*-benzyl to *N*-ethyl (**2b**) and *N*-phenyl (**2c**) also furnished the corresponding products **3ab** and **3ac** in good yields (68% and 62%, respectively). However, the 1,4-addition reaction of maleimide derivatives (**2a**, **2b**, and **2c**) with *N*-benzoylpyrrole derivatives resulted in poor yields under the optimal conditions. This problem was overcome by modifying the optimal conditions, wherein  $[\text{Rh}(\text{C}_5\text{Me}_5)_2\text{Cl}_2]_2$  was used instead of  $[\text{Ru}(\text{p-cymene})\text{Cl}_2]_2$ , and the corresponding C-2-substituted products (**3ha**, **3hb**, and **3hc**) were obtained in moderate yields (54%, 44%, and 47%, respectively). In cases of low yields, we found extensive decomposition of the starting material under the reaction conditions.

The scope of this reaction was further extended by performing a reaction of a variety of indole derivatives with *n*-butyl and methyl esters of maleic acid (**2d**, **2e**) under the optimal

conditions (Scheme 3). In these reactions, we were pleased to find the corresponding adducts were obtained in good yields.

### Scheme 3. Substrate Scope with Maleate Esters<sup>a,b</sup>

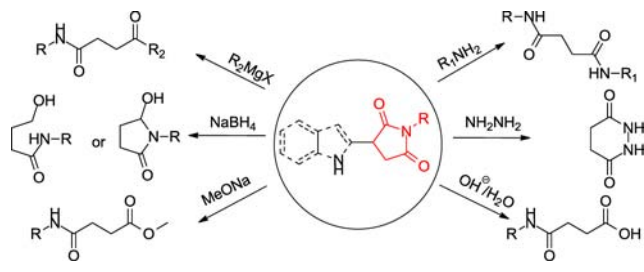


<sup>a</sup>Reaction conditions: *N*-benzoylindole derivative (1 equiv), maleate ester (2 equiv), [Ru(*p*-cymene)Cl<sub>2</sub>]<sub>2</sub> (5 mol %), AgSbF<sub>6</sub> (20 mol %), Cu(OAc)<sub>2</sub>·H<sub>2</sub>O (1 equiv), AcOH (10 equiv), DCE (2 mL), 120 °C, 24 h. <sup>b</sup>Isolated yields.

Halogenated, electron-rich, electron-neutral, as well as electron-deficient *N*-benzoylindole derivatives reacted in a smooth manner with dibutylmaleate (2d) to yield the corresponding diester adducts (3ad, 3bd, 3cd, 3dd, 3ed, 3fd, and 3gd) in good to excellent yields (68%, 81%, 77%, 62%, 75%, 62%, and 78%, respectively). The reaction was found to be equally efficient when 2d was replaced with dimethylmaleate (2e), leading to the formation of the products 3ae and 3be in good yields (67% and 79%, respectively).

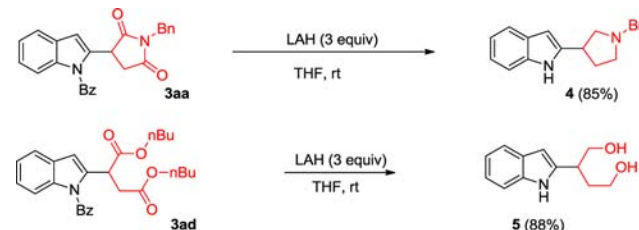
Succinimide rings can be cleaved by using selective reagents to generate a wide variety of useful functional groups like dicarboxylic acids, diamides, diols, hydrazides, etc. (Scheme 4).<sup>14</sup> They can be reduced to form useful pyrrolidinyl rings using

### Scheme 4. Opening of Succinimide Ring<sup>14</sup>



strong reducing agents such as LAH. Since such applications have not been reported with the indole moiety attached at third position of the succinimide ring, we have demonstrated the formation of 2-(1-benzylpyrrolidin-3-yl)-1*H*-indole (4), which was obtained in very high yields (Scheme 5, 85%). Similarly, the product 3ad was also subjected to reduction to form 1,4-diol with indole ring attached at the second position (Scheme 5, 2-(1*H*-indol-2-yl)butane-1,4-diol (5)). In both the cases, it was

### Scheme 5. Synthetic Applications<sup>a</sup>

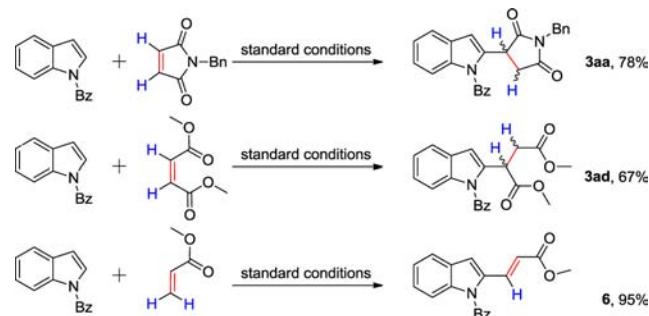


<sup>a</sup>Conditions: 3aa/3ad (0.37 mmol), LAH (1.1 mmol), THF (2 mL), room temperature. Values in parentheses indicate isolated yields.

observed that the benzoyl group was cleaved in situ by reduction with LAH, thereby saving a further deprotection step.

In order to gain some understanding of the mechanism, methyl acrylate was used instead of maleimide or maleate under the optimal conditions, and it furnished the Heck coupled product (6, Scheme 6). The terminal step of a Heck reaction is  $\beta$ -

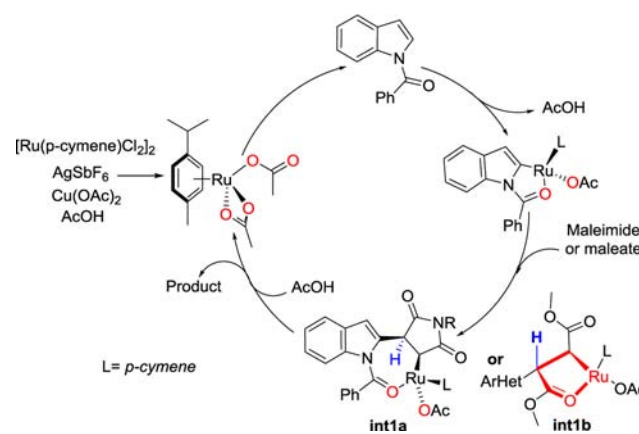
### Scheme 6. Maleimide and Maleate vs Acrylate



hydride elimination leading to the formation of an alkene. Moreover, the  $\beta$ -hydrogen has to be in a *syn*-periplanar orientation with the metal center. Therefore, we propose that maleimide and maleate do not provide this  $\beta$ -hydrogen atom in a *syn*-periplanar fashion. Although this is easy to visualize in the case of maleimide (rigid 5-membered ring, **int1a**)<sup>15</sup> it is counterintuitive in maleate ester, which is expected to possess conformational flexibility. However, a proposed formation of **int1b** leads to the formation of a conformationally rigid ring wherein the  $\beta$ -hydrogen is not *syn*-periplanar to the Ru(II) center (Scheme 7).

In conclusion, we have herein presented a novel regioselective addition reaction of the indole C-2 position to maleimide leading

### Scheme 7. Proposed Mechanism





to the formation of 3-substituted succinimide derivatives. This distinction of regioselectivity has been achieved by employing Ru(II) catalyst and a directing group strategy. Succinimides and indole derivatives are useful moieties in the field of organic chemistry, and many of them are synthetic drugs or natural products.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: [10.1021/acs.orglett.5b01809](https://doi.org/10.1021/acs.orglett.5b01809).

Experimental procedures; characterization data for all new compounds (PDF)

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### Notes

The authors declare no competing financial interest.

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