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Rhodium-Catalyzed Reaction of Sulfoxonium Ylides and Anthranils toward Indoloindolones via a (4 + 1) Annulation

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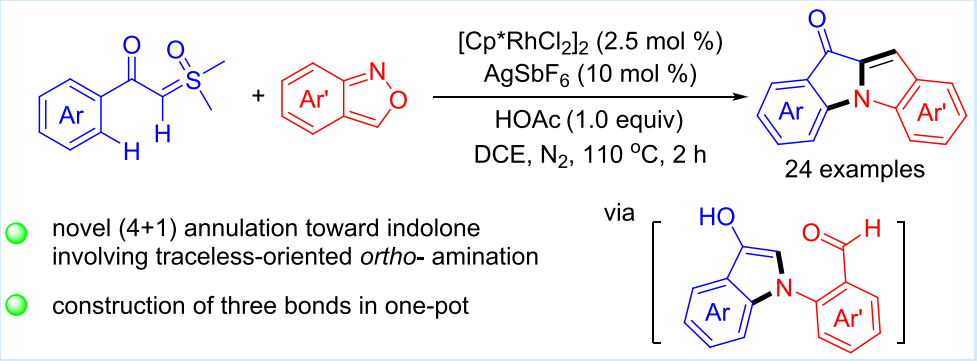
Supporting Information

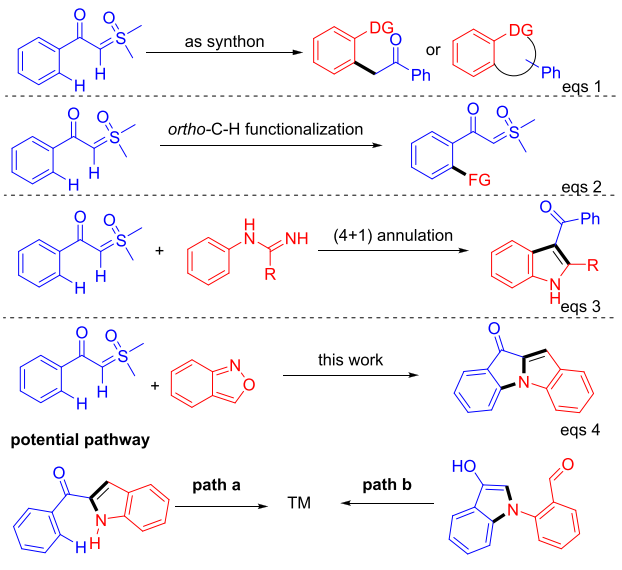


ABSTRACT: A rhodium-catalyzed annulation between aroyl sulfoxonium ylides and anthranils has been developed to synthesize 10*H*-indolo[1,2-*a*]indol-10-one derivatives. This reaction started with an unpredented (4 + 1) annulation toward *N*-(2-formylphenyl) indolones, proceeding with the sequential *ortho*-amination of the C−H bond in aroyl sulfoxonium ylides by anthranils and the insertion of N−H to carbene. Finally, the Aldol condensation constructed the second indole ring. This procedure features the formation of two C−N bonds and one C═C bond in one pot.

Recently, much progress has been made in the field of aroyl sulfoxonium ylides,[1] which served as novel carbene surrogates catalyzed by transition metals in the rapid construction of new C−C,[2] C−N,[3] and C−O[4] bonds.[5] Pioneered by Vaitla's elegant work on the coordination of aroyl sulfoxonium ylides with iridium,[6] Aïssa,[7] Li,[8] Kim,[9] and Ellman[10] independently developed the rhodium−carbon carbene-involved sequential C−H functionalization to access the *ortho*-acylation or cyclization products,[11] such as isoquinolones, isocoumarins, and pyrimidines, etc., where ylides served as either C1 or C2 synthons (Scheme 1, eq 1). Alternatively, the carbonyl in aroyl sulfoxonium ylides could enable the metal-catalyzed chelation-assisted *ortho*-C−H functionalization (Scheme 1, eq 2), developed by Li[12] and Fan,[13] leading to new aroyl sulfoxonium ylides, naphthols, and naphthalenones.

Meanwhile, aroyl sulfoxonium ylides could also undergo the (4 + 1) annulation with amidine toward 3-acylindole (Scheme 1, eq 3).[14] In light of the potential coordination of the N atom in the indole ring, we envisioned to develop the cascade annulation of aroyl sulfoxonium ylides with ideal substrates toward hetero polycycles, where the *in situ* formed potential directing group enabled the *ortho*-C−H functionalization to furnish the second cyclization. With this regard, anthranil was selected as the proper substrate since it served as the surrogate of 2-aminobenzaldehyde nitrene in metal-catalyzed *ortho*-C−H aromatization reactions.[15] Thus, the sequential insertion of

Scheme 1. Reaction of Aroyl Sulfoxonium Ylides



nitrene to the α-C−H in sulfoxonium ylides and Knoevenagel condensation may achieve the (4 + 1) annulation toward 2-

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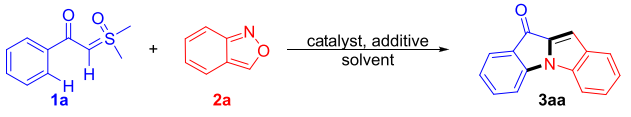
acylindole (path a in Scheme 1, eq 4). Afterward, the *in situ* formed indolyl-assisted intramolecular *ortho*-amination of the C−H bond selectively furnishes the second cyclization toward indoloindolones. Alternatively, the *ortho*-amination of the C−H bond in aroyl sulfoxonium ylides by anthranils followed by the (4 + 1) annulation may also access indoloindolones (path b in Scheme 1, eq 4).[16]

With the development of rhodium-catalyzed C−H activation[17] and our recent achievements,[18] herein, we report our study on the rhodium-catalyzed reaction of aroyl sulfoxonium ylides and anthranils toward 10*H*-indolo[1,2-*a*]indol-10-ones. Compared with the previous reported synthetic strategies,[19] this procedure features not only the simultaneous construction of indole and indolone rings by simple starting materials but also the diversity and complexity of the final compounds. Being a class of bioactive polycyclic heteroarenes combining indole and indolone scaffolds, indolo[1,2-*a*]indols are ubiquitous in the fields of dyes and external fluorescent indicators.[20]

We started our work by exploring the reaction of benzoyl sulfoxonium ylide **1a** (0.1 mmol) and anthranil **2a** (0.11 mmol) in the presence of $[Cp^*RhCl_2]_2$ (2.5 mol %) and $AgSbF_6$ (10 mol %) in toluene under 110 °C (Table 1).

Table 1. Selected Results for Screening the Optimized Reaction Conditions[a]



entry	catalyst	additive	solvent	yield[b] (%)
1	$[Cp^*RhCl_2]_2/AgSbF_6$	-	toluene	trace[c]
2	$[Cp^*RhCl_2]_2/AgSbF_6$	-	DCM	16[c]
3	$[Cp^*RhCl_2]_2/AgSbF_6$	-	CH_3CN	22[c]
4	$[Cp^*RhCl_2]_2/AgSbF_6$	-	THF	13[c]
5	$[Cp^*RhCl_2]_2/AgSbF_6$	-	DCE	35[c], 55
6	$[Cp^*RhCl_2]_2$	-	DCE	n.r.
7	$AgSbF_6$	-	DCE	n.r.
8	$[Cp^*RhCl_2]_2/AgNTf_2$	-	DCE	48
9	$[Cp^*RhCl_2]_2/AgBF_4$	-	DCE	43
10	$[Cp^*RhCl_2]_2/AgSbF_6$	Cs_2CO_3	DCE	n.r.
11	$[Cp^*RhCl_2]_2/AgSbF_6$	HOAc	DCE	81, 65[d] 68[e], 73[f]
12	$[Cp^*RhCl_2]_2/AgSbF_6$	PivOH	DCE	60

[a]Reaction conditions: Benzoyl sulfoxonium ylide **1a** (0.1 mmol), anthranil **2a** (0.11 mmol), $[Cp^*RhCl_2]_2$ (2.5 mol %), $AgSbF_6$ (10 mol %), other indicated additives (0.1 mmol), solvent (3.0 mL), and N_2 (1.0 atm), at 110 °C for 2 h, in a sealed Schlenk tube. [b]Isolated yield. [c]Under air. [d]At 80 °C. [e]At 100 °C. [f]At 120 °C.

However, no reaction took place at all (Table 1, entry 1). To our delight, the annulated product 10*H*-indolo[1,2-*a*]indol-10-one **3aa** was isolated in 16% yield in dichloromethane (DCM). Replacing DCM with THF or MeCN resulted in a similar yield, and the reaction efficiency was further increased in 1,2-dichloroethane (DCE) (entries 2−5). Fortunately, the yield of **3aa** increased to 55% under N_2 (entry 5). No reaction took place in the absence of either $[Cp^*RhCl_2]_2$ or $AgSbF_6$ (entries 6 and 7). Additionally, other Ag(I) species, such as $AgNTf_2$ (48%) and $AgBF_4$ (43%), showed similar reactivity under this transformation (entries 8 and 9). The reaction was totally inhibited in the presence of Cs_2CO_3 as a base (entry 10). On the contrary, the yield of **3aa** dramatically increased to 81% in the presence of HOAc (entry 11). However, pivalic acid (PivOH, 60%) was not so effective (entry 12). The reaction efficiency was not improved under elevated (73%, 120 °C) or lower (65%, 80 °C; 68%, 100 °C) reaction temperatures (entry 11).

With the optimized reaction conditions in hand, the scope and limitation of aroyl sulfoxonium ylides were studied, as shown in Figure 1. As expected, some functional groups, such

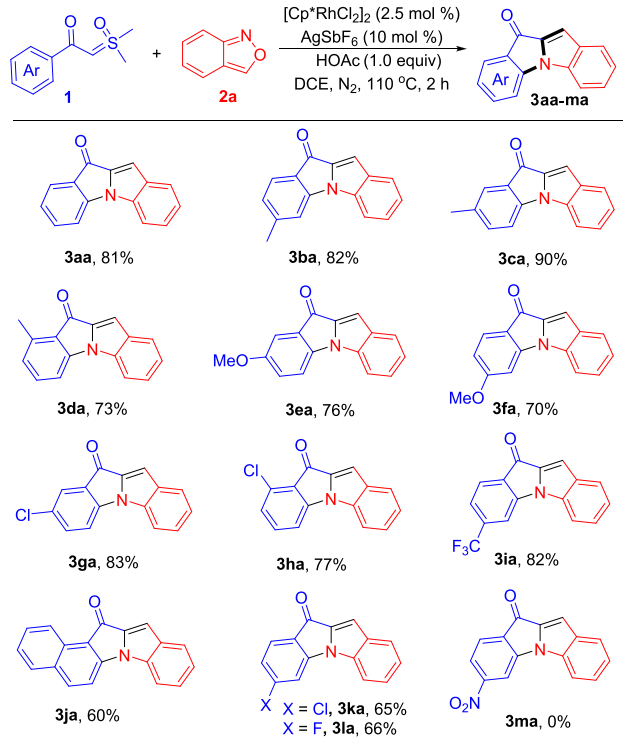


Figure 1. Scope of aroyl sulfoxonium ylides. Reaction conditions: aroyl sulfoxonium ylides **1** (0.1 mmol), anthranil **2a** (0.11 mmol), $[Cp^*RhCl_2]_2$ (2.5 mol %), $AgSbF_6$ (10 mol %), HOAc (0.1 mmol), DCE (3.0 mL), and N_2 (1.0 atm), at 110 °C for 2 h, in a sealed Schlenk tube.

as methyl (**3ba**−**da**, 73−90%), methoxy (**3ea**, 76%; **3fa**, 70%), halogen (**3ga**, **3ha**, **3ka**, and **3la**, 65−83%), and trifluoromethyl (**3ia**, 82%), were well compatible under the reaction conditions with good yields. Unfortunately, strong electron-withdrawing groups such as NO_2 inhibited the reaction. Importantly, various substrates possessing substituents at the *para*- (**3ba**, 82%, **3fa**, 70%; **3ia**, 82%; **3ka**, 65%; **3la**, 66%), *meta*- (**3ca**, 90%, **3ea**, 76%; **3ga**, 83%), and *ortho*- (**3da**, 73%, **3ha**, 77%) positions of the phenyl ring all ran smoothly under the standard procedures. Notably, the naphthyl analogue was also a good reaction partner, providing **3ja** in 60% yield. In a 1.0 mmol scale reaction, **3aa** was isolated in 45% yield.

Next, the scope of substituted anthranils was studied, as shown in Figure 2. Generally, all substrates ran smoothly to afford the 10*H*-indolo[1,2-*a*]indol-10-ones in 65%−88% yields. The procedure tolerated chloro (**3cb**, 85%; **3cf**, 76%), fluoro (**3cc**, 79%; **3cg**, 70%), and bromo (**3ad**, 74%; **3cd**, 86%; **3dd**, 70%) groups well, which provided handles for potentially further functionalization. In addition to halogens, substrates containing electron-donating groups (**3ce**, 88%; **3ch**, 83%) are also suitable reaction partners under the standard procedure. Specially, anthranils with unsaturated functional groups (**3ak**,

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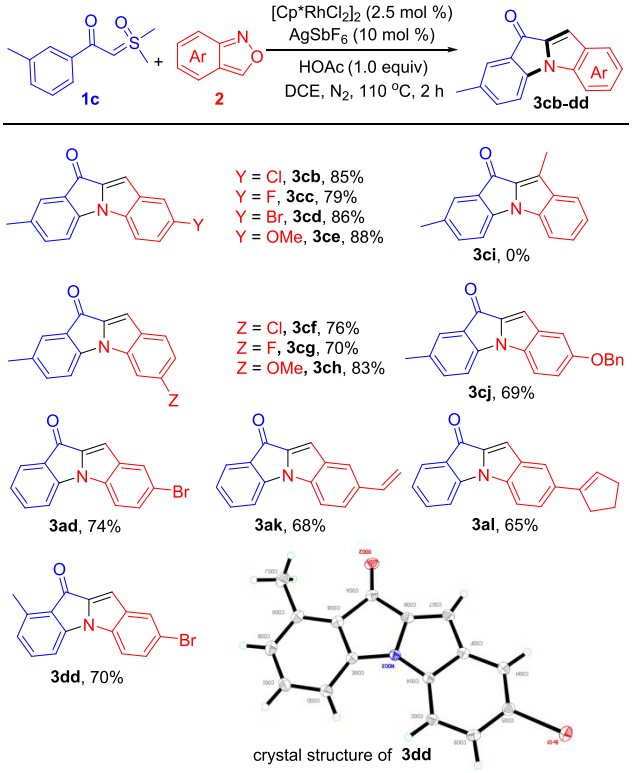
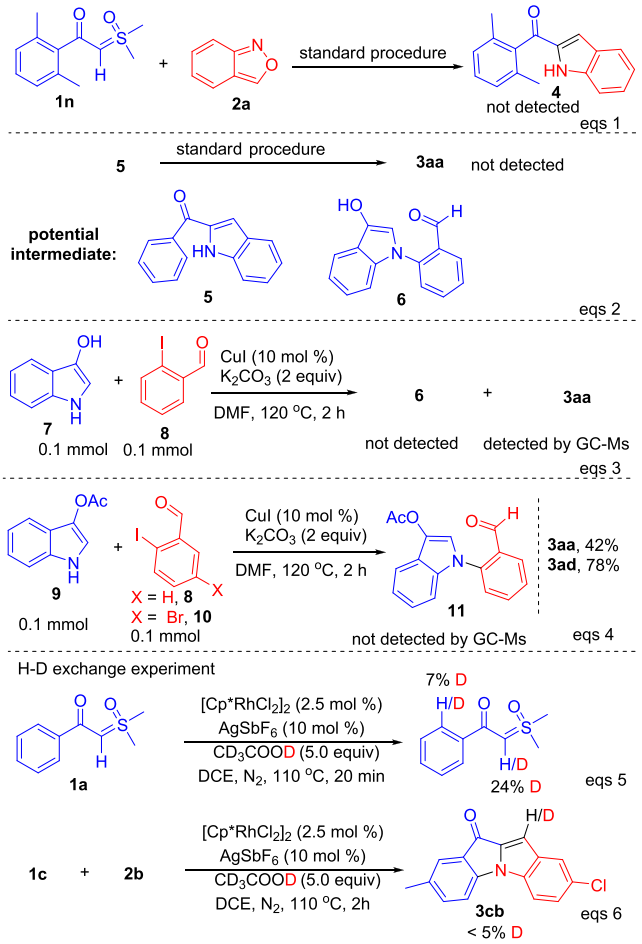


Figure 2. Substrate scope of substituted anthranils. Reaction conditions: benzoyl sulfoxonium ylide **1a** (0.1 mmol), substituted anthranils **2** (0.11 mmol), $[Cp^*RhCl_2]_2$ (2.5 mol %), $AgSbF_6$ (10 mol %), HOAc (0.1 mmol), DCE (3.0 mL), and N_2 (1.0 atm), at 110 °C for 2 h, in a sealed Schlenk tube.

68%; **3al**, 65%) and protected hydroxyl (OBn, **3cj**, 69%) worked well, which further increased the practibility of this procedure. Furthermore, the structure of **3dd** was confirmed by X-ray crystallography (for details, see Supporting Information). This result strongly confirmed that the carbonyl in the final products was derived from aroyl sulfoxonium ylides rather than anthranils.

More control experiments were conducted to get some insights into this reaction. First, the 2,6-dimethylbenzoyl sulfoxonium ylide (**1n**) was subjected to the reaction with anthranil (**2a**) under standard procedures. However, no 2-benzoyl indole (**4**) was detected at all (Scheme 2, eq 1). Second, intermediate **5** did not transform into **3aa** under the standard procedure (Scheme 2, eq 2). These results ruled out the possibility of the involvement of path a depicted in eq 4 of Scheme 1. Third, during the Ullman reaction of indoxyl (**7**) with 2-iodobenzaldehyde (**8**), **3aa** rather than the expected coupling product **6** was detected by GC-MS (Scheme 2, eq 3). Thus, the presumed intermediate **6** may transform into the final products, although it was too reactivated to be detected (path b in eq 4 of Scheme 1). Moreover, the Ullman reaction of indoxyl acetate (**9**) with 2-iodobenzaldehyde (**8**) or 5-bromo-2-iodobenzaldehyde (**10**) provided **3aa** and **3ad** in 42% and 78% isolated yields, where intermediate **11** was not detected by GC-MS at all (Scheme 2, eq 4).[21] The results suggested the *in situ* hydrolysis of **11** to 3-hydroxyl indole, which underwent the intramolecular Aldol condensation immediately toward the final products. Finally, the H/D exchange for benzoyl sulfoxonium ylide **1a** confirmed that the cleavage of the C−H bond in **1a** was reversible (Scheme 2, eq 5).[13] Thus, any attempt to test the inter- and intramolecular KIE for the benzoyl sulfoxonium ylide failed. Replacing CH_3COOH with CD_3COOD under standard conditions, almost no D-incorporation was observed in **3cb** (Scheme 2, eq 6).

Scheme 2. Control Experiments



Based on the control experiments and some relevant literature,[5,12,13] a mechanism for the procedure is proposed in Scheme 3. The catalytic cycle starts with the depolymerization of rhodium catalyst and C−H activition with benzoyl sulfoxonium ylide (**1a**), leading to a rhodacyclic intermediate **A**. Next, the insertion of anthranil (**2a**) produces a five-membered cyclic "Rh=N" nitrene species **C**,[15] which then transforms into a six-membered rhodacycle **D** and **E** through intramolecular migration.[12] After releasing a molecule of DMSO, a new "Rh=C" carbene species **F** is formed. Then, the insertion of N−H in **F** to the carbene takes place to furnish the (4 + 1) annulation toward intermediate **G**. The protonation of intermediate **G** releases Rh(III) catalyst to re-enter the catalytic cycle, along with intermediate **6**. Finally, intermediate **6** transforms to **3aa** via an intramolecular Aldol condensation.

In conclusion, we have developed a Rh(III)-catalyzed *ortho*-C−H annulation between *α*-aroyl sulfoxonium ylides and anthranils toward a series of 10*H*-indolo[1,2-*a*]indol-10-ones in moderate to good yields, proceeding with a (4 + 1) annulation involving sequential *ortho*-amination of the C−H bond in aroyl sulfoxonium ylides and insertion of N−H to carbene. Our procedure features the rapid construction of two

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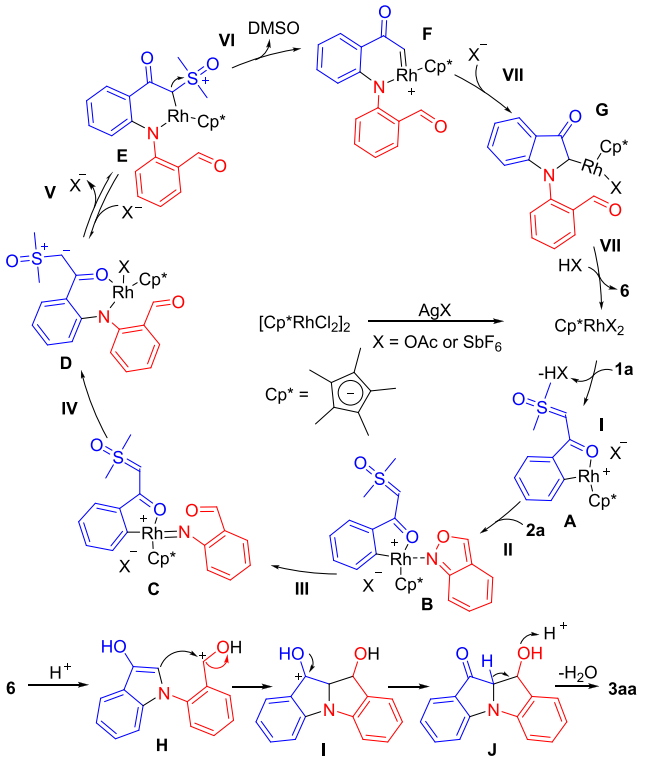
Scheme 3. Tentative Mechanism



C−N bonds and one C═C bond in one pot, which not only provides a facile route to access indole derivatives with diversity and complexity but also broadens the application of aroyl sulfoxonium ylides in transition-metal-catalyzed C−H functionalization.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.9b02249.

Experimental procedures along with copies of spectra (PDF)

Accession Codes

CCDC 1935878 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

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Notes

The authors declare no competing financial interest.

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