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Synthesis of Pyrido[2,3-*b*]indole Derivatives via Rhodium-Catalyzed Cyclization of Indoles and 1-Sulfonyl-1,2,3-triazoles

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Abstract: Acyloxy-substituted α,β-unsaturated imines generated *in situ* from triazoles can act as aza-[4C] synthons and be trapped by indoles in a stepwise [4 + 2] cycloaddition reaction, thus providing rapid access to valuable pyrido[2,3-*b*]indoles in high yields. Attractive features of this reaction system include operational simplicity, readily available substrates, construction of sterically demanding quaternary centers, and convenient derivatization using triflate.

Keywords: Pyrido[2,3-*b*]indole; [4 + 2] Annulation; Aza-[4C] synthon; Carbenes; 1,2,3-Triazole

Indole alkaloids containing a pyrido[2,3-*b*]indole motif are present in a variety of natural products, such as communesin F,[1] chaetominine,[2] and neoxaline[3] (Figure 1). Because these compounds and their derivatives are biologically active,[1–3] many strategies for the construction of the pyrido[2,3-*b*]indole scaffold have been developed in the past few decades.[4–8] With the goal of preparing the well-known natural product communesin F, many groups have developed methods to synthesize tetracyclic indolo[2,3-*b*]quinolines.[4] Similarly, many approaches to synthesize the 6,5,6,5-tetracyclic skeleton have also been established in the synthesis of chaetominine and its analogs.[5] However, few synthetic methods for the preparation of tricyclic pyrido[2,3-*b*]indoles that do not bear other rings have been reported,[6–8] and the formation of a C–N bond on the piperidine ring is the most common method.[6] However, these methods[6a–d] are usually complicated and inefficient because of their multistep nature. Garg developed a strategy to construct pyrido[2,3-*b*]indoles using an interrupted Fisher indolization sequence,[7] and Ghorai reported a [4 + 2] cyclization reaction to construct pyrido[2,3-*b*]indoles using indoles and azetidines.[8] Nevertheless, these authors only reported one example, and, thus, the applicability of these methods is not clear. Herein, we report an efficient and practical method for the synthesis of pyrido[2,3-*b*]indoles involving the direct cyclization of the indoles and aza-[4C] units.

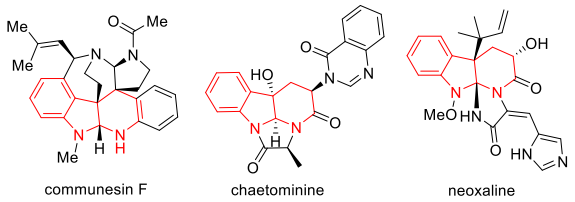

Figure 1. Representative natural compounds.

In 2008, Fokin and Gevorgyan demonstrated that Dimroth-type equilibrium between 1-sulfonyl-1,2,3-triazoles and α-diazo imines can be interrupted by a rhodium(II) catalyst, leading to the efficient production of α-imino rhodium carbenes (Scheme 1a).[9] Because of the easy access to triazoles,[10] the simple reaction procedure, and the unique reactivity of the α-imino carbenes in the synthesis of nitrogen-containing compounds, numerous studies have been conducted in this area in recent years.[11,12] In general, 1-sulfonyl-1,2,3-triazoles serve as aza-[3C] units in the reaction with indoles (Scheme 1b).[13] For example, in 2013, Davies[13a] reported a reaction to synthesize pyrroloindoline via a catalytic enantioselective formal [3 + 2] cycloaddition of C3-substituted indoles and 1-sulfonyl-1,2,3-triazoles. Subsequently, Shi[13b] reported an intramolecular version of a similar formal [3 + 2] cycloaddition. In 2015, Murakami,[13d] Anbarasan,[13e] and Chen[13f] almost simultaneously reported a method to synthesize tryptamine derivatives employing C3-nonsubstituted indoles and 1-sulfonyl-1,2,3-triazoles. In addition to this, Shi,[14] Anbarasan,[15] and our group[12k,12m,12o] found that 1-sulfonyl-1,2,3-triazoles can also serve as aza-[4C] units in some special cases (Scheme 1c). In these cases, the 1,2-sulfur (or OAc) migration of an α-imino rhodium carbene delivers an α,β-unsaturated imine, which can participate in the ring formation reaction as an aza-[4C] synthon. Thus,

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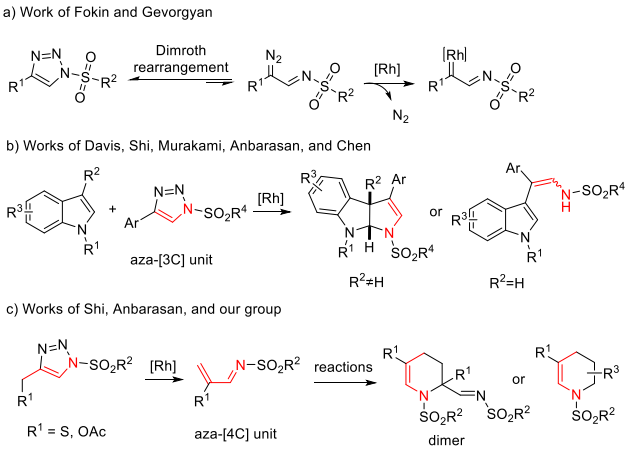
we envisaged that a compound containing a pyrido[2,3-*b*]indole core could be easily obtained when employing indoles as a [2C] synthon and the *in situ* generated α,β-unsaturated imines as an aza-[4C] synthon.

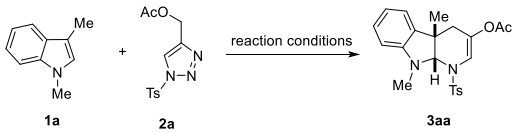


Scheme 1. Previously reported reactions.

We initiated our study by the treatment of readily accessible triazole **2a** (0.2 mmol) and 1,3-dimethyl-1*H*-indole (**1a**, 0.2 mmol) with 2 mol% $Rh_2(OAc)_4$ in 1,2-dichloroethane (1,2-DCE, 2 mL) at 80 °C for 4 h. Fortunately, the expected product (**3aa**) bearing a *cis*-pyrido[2,3-*b*]indole skeleton was obtained in 42% yield (Table 1, entry 1), and the structure was unambiguously confirmed by X-ray crystallographic analysis.[16] Encouraged by this exciting result, further experiments were conducted to improve the reaction efficiency. By monitoring the reaction process, we found that the α,β-unsaturated imine intermediate was generated quickly through denitrogenation and acetoxy migration of triazole **2a**. In contrast, the indole and α,β-unsaturated imine reacted more slowly. Furthermore, the dimer (Scheme 1c, aza-Diels–Alder product, R^1 = OAc, R^2 = tolyl) of the α,β-unsaturated imine also appeared when the reaction time was increased. To inhibit this side reaction, the quantity of triazole **2a** was increased to two equivalents, and the reaction time was controlled at 4 h (entry 2). Solvent screening revealed that 1,2-DCE was the best solvent (entries 2−5). The reaction temperature was also evaluated, and 60 °C was found to be optimal. Crucially, higher or lower temperatures resulted in reduced yields (entries 2, 6−9). To our surprise, the yield of **3aa** increased to 81% when we reduced the amount of solvent (entries 8, 10, and 11). In addition, the yield further increased to 85% when using 1 mol% $Rh_2(OAc)_4$ (entry 12). Various rhodium(II) catalysts were tested, but $Rh_2(OAc)_4$ gave the best yield (entries 13−15).

Using the optimized reaction conditions (Table 1, entry 12), the substrate scope of triazole **2** was investigated (Scheme 2). In addition to triazole acetate (**2a**), the benzoate (**2b**) and phenylacetate (**2c**) also reacted with 1,3-dimethyl-1*H*-indole (**1a**) in the presence of $Rh_2(OAc)_4$, and the products **3ab** and **3ac** were obtained in 85% and 78% yields, respectively. Remarkably, the sterically hindered trityl group had no obvious impact on the reaction, and compound **3ad** was obtained in 87% yield. Moreover, other functional groups such as chloride, *t*-butyloxycarbonyl (Boc), and morpholine were all well tolerated in this reaction (**3ae–ag**). When changing the protecting group on the nitrogen to methylsulfonyl, product **3ah** was obtained in 73% yield. Notably, sulfur-tethered triazole **2i** could also be used in this reaction, affording the corresponding **3ai** in 68% yield.

Table 1. Optimization of Reaction Conditions.[a]



Entry	Catalyst	Solvent	*T*/°C	Yield/%[b]
1[c]	$Rh_2(OAc)_4$	1,2-DCE	80	42
2	$Rh_2(OAc)_4$	1,2-DCE	80	62
3	$Rh_2(OAc)_4$	toluene	80	54
4	$Rh_2(OAc)_4$	1,1,2-TCE	80	37
5[d]	$Rh_2(OAc)_4$	DCM	80	30
6	$Rh_2(OAc)_4$	1,2-DCE	90	42
7	$Rh_2(OAc)_4$	1,2-DCE	70	62
8	$Rh_2(OAc)_4$	1,2-DCE	60	63
9	$Rh_2(OAc)_4$	1,2-DCE	50	29
10[e]	$Rh_2(OAc)_4$	1,2-DCE	60	81
11[f]	$Rh_2(OAc)_4$	1,2-DCE	60	80
12[e,g]	$Rh_2(OAc)_4$	1,2-DCE	60	85
13[e,g]	$Rh_2(piv)_4$	1,2-DCE	60	82
14[e,g]	$Rh_2(oct)_4$	1,2-DCE	60	74
15[e,g]	$Rh_2(adc)_4$	1,2-DCE	60	76

[a] General reaction conditions: **1a** (0.2 mmol), **2a** (0.4 mmol), [Rh] (2 mol%), solvent (2 mL), N_2 atmosphere, 4 h.
[b] Isolated yield.
[c] **2a** (0.2 mmol).
[d] Carried out in a sealed tube.
[e] 1,2-DCE (1 mL).
[f] 1,2-DCE (0.67 mL).
[g] [Rh] (1 mol%).
1,1,2-TCE = 1,1,2-trichloroethane, DCM = dichloromethane, piv = pivalate, oct = octanoate, adc = 1-adamantanecarboxylate.

Subsequently, the reaction scope in relation to indole **1** was also investigated under the optimized conditions (Scheme 3). Initially, the effect of the protecting group on the indole nitrogen was examined. Electron-donating substituents on the nitrogen were tolerated in the reactions, giving related products **3ba–da** in moderate yields. In contrast, substrates with electron-withdrawing groups such as Boc and phenyl or without protecting groups on the nitrogen did not afford the expected products. A methyl group at the 4-, 5-, or 6-position of the indole was well-

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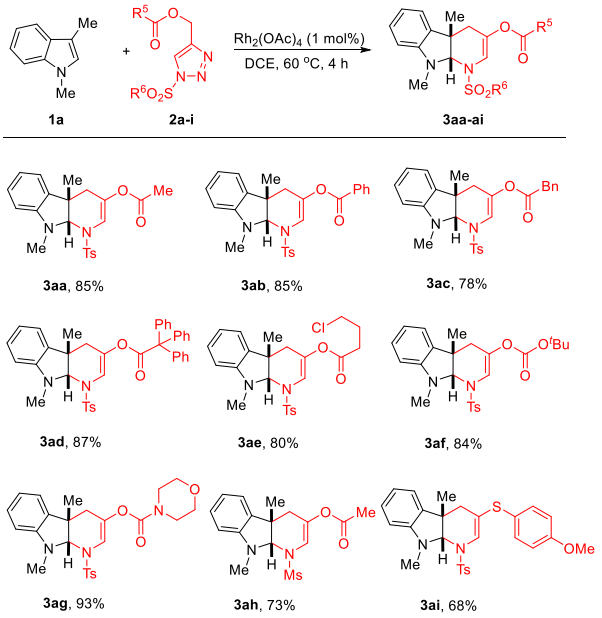
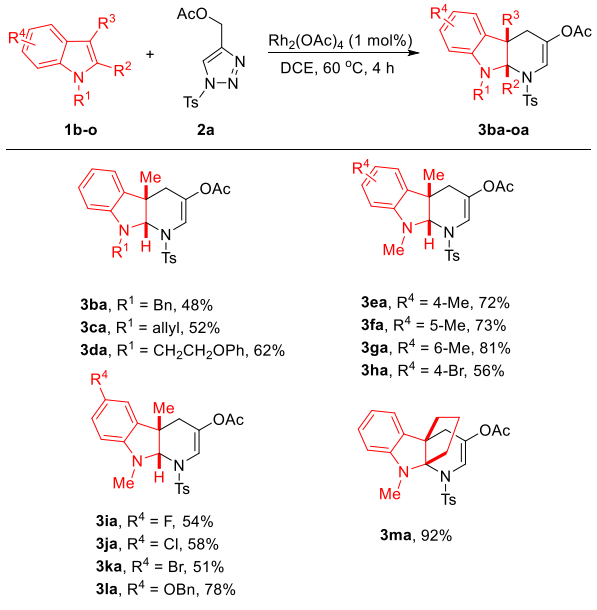
tolerated, and the resulting cyclization products were produced in 72−81% yield (**3ea–ga**). In addition, several products containing valuable halogens and ethers were synthesized conveniently (**3ha–la**, 51−78%). Excitingly, compound **3ma**, which has a complicated tetracyclic skeleton and two adjacent quaternary carbons, was obtained in 92% yield.



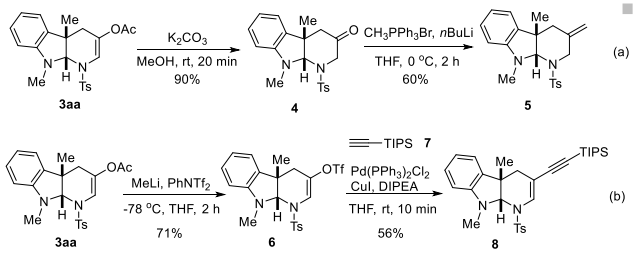
Scheme 2. Substrate Scope of Triazole **2**.



Scheme 3. Substrate Scope of Indole **1**.

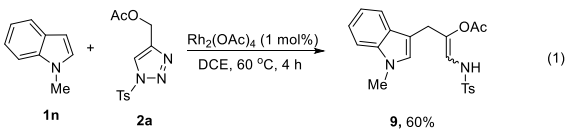
In addition to indoles, other dienophiles such as 3-methylbenzofuran, 5-methoxy-3-methylbenzofuran, 3-methylbenzo[*b*]thiophene, 1*H*-indene, and 1,2-dihydronaphthalene were also reacted with the triazole **1a** under the optimized reaction conditions. Unfortunately the corresponding products could not be obtained, and the starting material dienophiles were recovered. The above results indicate that the electron density of the dienophile has a significant effect on the success of the [4 + 2] cyclization.

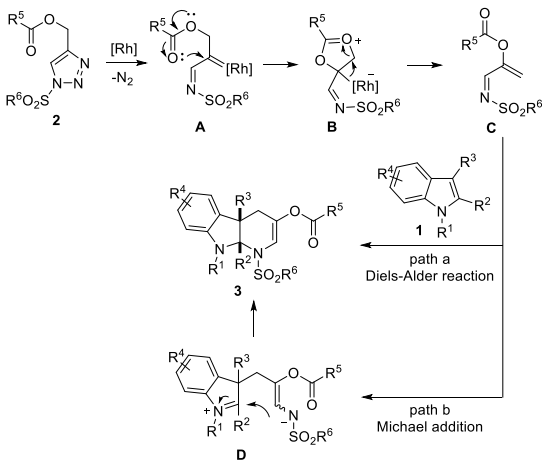
Subsequently, several derivatization reactions were performed to illustrate the utility of the product. Ketone **4** was obtained when **3aa** was hydrolyzed under weakly basic conditions, and this ketone was converted into compound **5** when treated with a phosphonium ylide (Scheme 4a). Additionally, in the presence of MeLi and $PhNTf_2$, the OAc group of **3aa** could be easily transformed into OTf, which is one of the most useful functional groups in metal-catalyzed cross-coupling reactions. For example, compound **8** was obtained in 56% yield when triflate **6** was reacted with (triisopropylsilyl)acetylene under Sonogashira coupling reaction conditions (Scheme 4b).



Scheme 4. Synthetic transformation of **3aa**.

A mechanism for this reaction is proposed in Scheme 5 on the basis of the present results and previous reports.[12,14,15] In the presence of the rhodium(II) catalyst, the denitrogenation of triazole **2** leads to the formation of α-imino rhodium carbene **A**. Subsequently, the carbonyl oxygen attacks the electrophilic carbene carbon, and the subsequent elimination of the rhodium catalyst delivers α,β-unsaturated imine **C** rapidly. In path a, compound **3** is obtained directly through the aza-Diels–Alder reaction of indole **1** and intermediate **C**. Alternatively, the C3 of indole could attack the α,β-unsaturated imine, delivering Michael addition intermediate **D**. The subsequent intramolecular nucleophilic addition produces the final product: **3** (path b). Notably, when indole **1n** was employed in the transformation, no pyrido[2,3-*b*]indole structure was formed, and, instead, compound **9** was obtained in 60% yield [Eq. (1)]. We speculate that when the R^3 group of intermediate **D** is hydrogen, the intramolecular proton transfer is faster than the cyclization. Thus, the stepwise pathway may be the most likely route.





Scheme 5. Proposed Mechanism.

In conclusion, we have developed a rhodium-catalyzed denitrogenation reaction of triazoles with indoles that provides rapid access to pyrido[2,3-*b*]indoles in high yields. The triazole acts as an aza-[4C] synthon in this reaction rather than an aza-[3C] synthon, as used in previous reports. This transformation is particularly attractive because of its operational simplicity, readily available substrates, construction of sterically demanding quaternary centers, and convenient derivatization using a triflate derivative. Further studies of the construction of complex polycyclic compounds using triazoles and their applications in natural product synthesis are under investigation.

Experimental Section

General procedure for the synthesis of 3

To a mixture of indole **1** (0.20 mmol) and triazole **2** (0.40 mmol) in anhydrous DCE (1 mL) under N_2, $Rh_2(OAc)_4$ (0.002 mmol) was added. The reaction mixture was stirred at 60 °C for 4 h. The mixture was cooled to room temperature and the solvent was removed. The crude product was purified using a silica gel column (basified with NaOH, pH 8.0–9.0) to afford product **3**.

Acknowledgements

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COMMUNICATION

Synthesis of Pyrido[2,3-*b*]indole Derivatives via Rhodium-Catalyzed Cyclization of Indoles and 1-Sulfonyl-1,2,3-Triazoles

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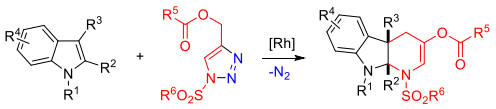

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