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Silver Triflate Catalyzed Tandem Heterocyclization/Alkynylation of 1-((2-Tosylamino)aryl)but-2-yne-1,4-diols to 2-Alkynyl Indoles

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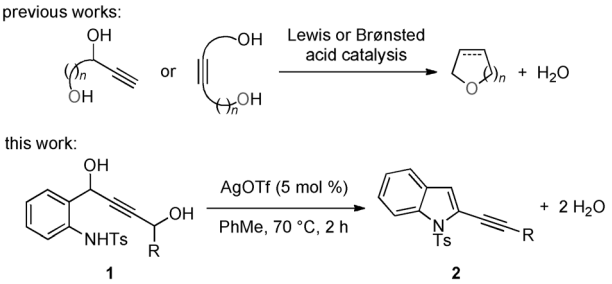
Indoles are a key structural component in many natural and pharmaceutical products as well as functional materials.[1–3] Because of this, and their ability to serve as a versatile building block in organic synthesis, a myriad of impressive methods for the construction of indole derivatives have been developed over the years. Recently, this has hitherto included transition-metal-catalyzed cross-coupling of an indole with an alkyne, either preformed or generated in situ, to access synthetically valuable 2-alkynyl indole derivatives.[3] However, the reactions were shown to require stoichiometric or excess amounts of various reagents, which can lead to equimolar or more amounts of waste products. Added to this is the need to introduce structural elements to direct the C–C bond-forming process to occur regioselectively at the C2 position of the indole ring. For this reason, establishing synthetic methods to this immensely important nitrogen heterocycle in an efficient manner and with control of substitution patterns from readily accessible substrates continues to be actively pursued.

Lewis and Brønsted acid-catalyzed reactions of unsaturated alcohols have emerged over the years as efficient and convenient synthetic strategies for C–C and C–X (X = N, O, S) bond formation.[4–6] For example, we recently reported a method for the synthesis of indenyl-fused and 2,3-disubstituted indoles that relied on the cycloisomerization of 2-tosylaminophenylprop-1-yn-3-ols in the presence of a gold(I) catalyst.[2b] We subsequently demonstrated that the synthetic method could be fine-tuned to provide 1*H*-indole-2-carbaldehydes and (*E*)-2-(iodomethylene)indolin-3-ols by introducing *N*-iodosuccinimide into the reaction conditions.[2a] Further exploration of this field led us to investigate the potential Lewis acid-catalyzed reactivity of propargylic diols. Thus far, the Lewis and Brønsted acid-mediated chemistry of this class of compounds has been reported to give only the oxygen heterocycle and an equimolar amount of H_2O (Scheme 1).[6] In contrast, a process involving a Lewis acid-triggered C–OH bond activation of a propargylic diol, which results in the formation of an N-heterocycle with the liberation of two molecules of H_2O as potentially the only by-product is not known. As part of ongoing efforts to develop this type of reaction, our discovery that inexpensive, ecologically benign, and readily available simple Ag^I salts can effect tandem heterocyclization/alkynylation of propargylic 1,4-diols of the type **1** with an appropriately placed aniline moiety is reported herein (Scheme 1). This provides a convenient route to 2-alkynyl indoles **2** that assembles both the indole ring and alkyne moiety in one step for a wide range of substrates. Achieved under mild conditions, it also represents the first synthetic method for the preparation of this N-heterocycle that does not rely on a cross-coupling strategy.



Scheme 1. Lewis and Brønsted acid-catalyzed reactivities of propargylic diols.

The 1-((2-tosylamino)aryl)but-2-yne-1,4-diols studied in this work were prepared from the reaction of the corresponding aldehyde and substituted *N*-tosyl-1-(2-aminophenyl)prop-2-yn-1-ol pretreated with LDA following literature procedures.[7] By using *N*-tosyl-1-(2-aminophenyl)-1,3-diphenyl-prop-2-yn-1-ol **1a** as the probe substrate, we began by examining a variety of Lewis and Brønsted acid catalysts to establish the reaction conditions (Table 1). This study initially revealed treating a solution of **1a** in toluene with AgOTf (5 mol%) at room temperature for 7 h gave 3-phenyl-2-(phenylethynyl)-1-tosyl-1*H*-indole **2a** and 3-phenyl-2-(2-phenylvinylidene)-1-tosyl indolin-3-ol **3a** in 45 and 30% yields, respectively (Table 1, entry 1). The structure of the 2-alkynyl indole product was determined by

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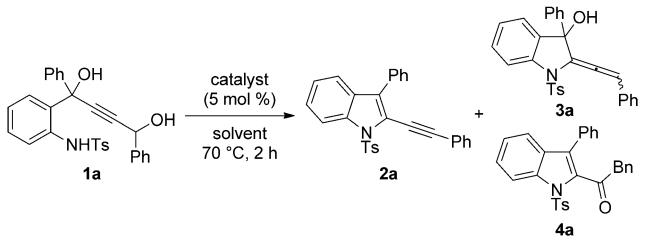
Table 1. Optimization of the reaction conditions.[a]



Entry	Catalyst	Solvent	Yield [%][b]		
			2a	**3a**	**4a**
1[c]	AgOTf	PhMe	45	30	–
2	AgOTf	PhMe	88	–	–
3[d]	AgOTf	PhMe	78	–	–
4[e]	AgOTf	PhMe	69	–	–
5[f]	AgOTf	PhMe	86	–	–
6[g]	AgOTf	PhMe	85	–	–
7[h]	AgOTf	PhMe	68	–	–
8	AgOTf	$MeNO_2$	78	–	–
9	AgOTf	1,4-dioxane	79	–	–
10	AgOTf	$(CH_2Cl)_2$	65	–	–
11	AgOTf	THF	–[i]	–	–
12	AgOTf	MeCN	–[i]	–	–
13	$AgNTf_2$	PhMe	38	–	–
14	$AgPF_6$	PhMe	30	–	18
15	$AgSbF_6$	PhMe	20	–	55
16	$AgBF_4$	PhMe	35	–	15
17	AgOAc	PhMe	–[i]	–	–
18	$Cu(OTf)_2$	PhMe	68	–	–
19	$Yb(OTf)_3$	PhMe	50	–	–
20	*p*-TsOH·H_2O	PhMe	–[j]	–	–
21	TFA	PhMe	20	–	–
22	TfOH	PhMe	32	–	–
23	Tf_2NH	PhMe	35	–	–

[a] All reactions were performed at the 0.1 mmol scale with catalyst/**1a** ratio = 1:20 in 4 mL of solvent at 70 °C for 2 h. [b] Isolated product yield. [c] Reaction carried out at room temperature for 7 h. [d] Reaction carried out in the presence of 5 mol % of Et_3N. [e] Reaction carried out in the presence of 10 mol % of Et_3N. [f] Reaction carried out in the presence of 5 mol % of K_2CO_3. [g] Reaction carried out in the presence of 10 mol % of K_2CO_3. [h] Reaction carried out in the presence of 1 equiv of K_2CO_3. [i] No reaction based on TLC and ^1H NMR analysis of the crude reaction mixture. [j] Decomposition products obtained based on TLC and ^1H NMR analysis of the crude reaction mixture.

^1H NMR spectroscopy and X-ray crystallography (Figure 1).[8] Our studies subsequently showed that formation of the 2-vinylidene indolin-3-ol byproduct could be suppressed to give **2a** as the only product in 88 % yield by increasing the reaction temperature to 70 °C for 2 h (Table 1, entry 2). Slightly lower product yields were obtained when the reaction was repeated in the presence of 5 or 10 mol % of Et_3N or K_2CO_3 as well as 1 equiv of the latter inorganic base (Table 1, entries 3–7). Likewise, changing the solvent from toluene to $MeNO_2$, 1,4-dioxane or 1,2-dichloroethane gave slightly lower product yields of 65–79 % (Table 1, entries 8–10). In contrast, replacing toluene with THF or MeCN as the solvent was found to result in recovery of the substrate in near quantitative yield (Table 1, entries 11 and 12). Similarly, a survey of other low cost silver(I) salts and Lewis acids did not provide any improvements (Table 1, entries 13–19). Moreover, in reactions where $AgPF_6$, $AgSbF_6$ or $AgBF_4$ was employed as the catalyst, the Meyer–Schuster rearrangement adduct **4a** was also afforded as a side product in 15–55 % yield (Table 1, entries 14–16).[9] The analogous AgOAc-mediated reaction was the only instance in which the substrate was recovered in near quantitative yield (Table 1, entry 17). Low product yields of 20–35 % were additionally afforded in control experiments with the Brønsted acid catalysts TFA, TfOH and Tf_2NH, whereas *p*-TsOH·H_2O led to decomposition of the substrate (Table 1, entries 20–23). A similar outcome was found when the Brønsted-acid-mediated reactions were re-examined in a variety of solvents and at various catalyst loadings and temperatures.[7] Under these various conditions, the 2-alkynyl indole product was furnished in 25–48 % yield and/or with recovery of **1a** in up to 68 % yield and/or substrate decomposition. Along with the above results of reaction in the presence of a base, the possibility of a hidden Brønsted acid catalyst was shown to be unlikely based on further control experiments with AgOTf at 1 and 5 mol % heated to reflux in 1,2-dichloroethane prior to use or 5 mol % of AgOTf in the presence of 10 mol % of *t*BuCl, which furnished **2a** in low yields of 13–38 %.[7,10] On the basis of the above results, the reaction of **1a** in the presence of AgOTf (5 mol %) in toluene at 70 °C for 2 h provided the optimal conditions.

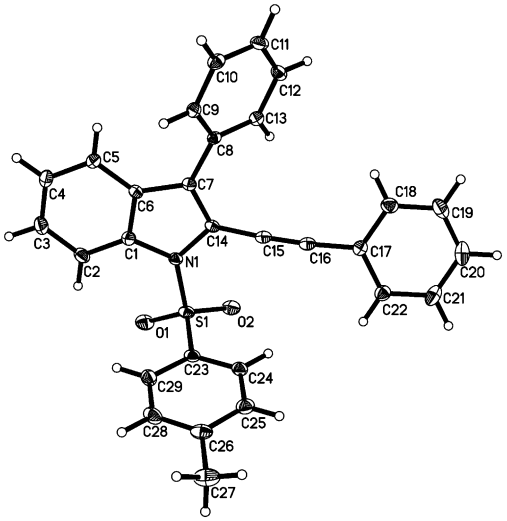
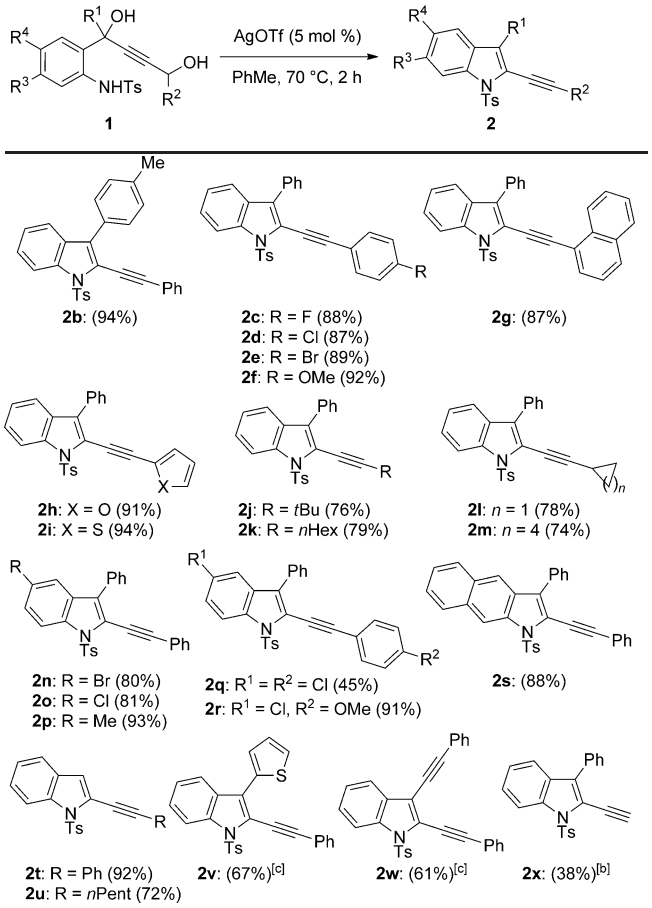


Figure 1. ORTEP drawing of **2a** with thermal ellipsoids at 50 % probability levels.[8]

With the optimized conditions in hand, we next turned to evaluating their generality for a series of propargylic 1,4-diols and the results are summarized in Table 2. These reactions demonstrated that by using AgOTf as catalyst, the conditions proved to be broad and a variety of 2-alkynyl indoles could be afforded in good to excellent yields from the corresponding substrates **1b–x**. Starting alcohols with a pendant phenyl moiety and their derivatives with electron-withdrawing or electron-donating groups in the *para* position at R^1 or R^2 were found to react well, affording **2b–f** in excellent yields of 87–94 %. Likewise, 2-alkynyl indoles **2g–m**,

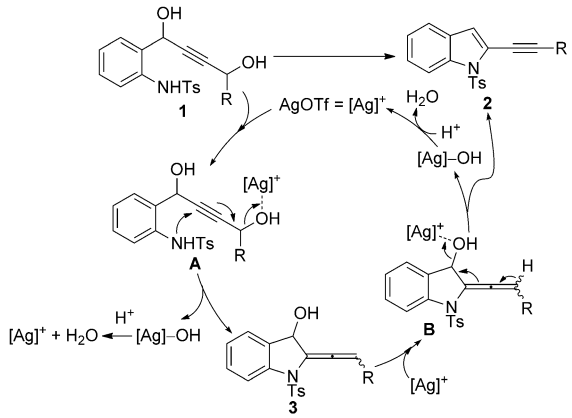
Table 2. Tandem heterocyclization/alkynylation of **1b–x** catalyzed by AgOTf.[a]



[a] All reactions were performed at the 0.1 mmol scale with AgOTf/**1** ratio = 1:20 in toluene (4 mL) at 70 °C for 2 h. Values in parenthesis denote isolated product yields. [b] Reaction performed at 40 °C for 0.5 h. [c] Reaction performed for 0.5 h.

containing a 1-napthyl, heteroaryl, alkyl, or cycloalkane substituent on the alkyne side chain, were obtained in excellent yields of 74–94% from the corresponding alcoholic substrates **1g–m**. The presence of an electron-withdrawing or electron-donating group or benzofused ring on the aniline moiety was found to have no influence on the course of the reaction with **2n–p** and **2r–s** obtained in 80–93% yield. Additionally, substrates where both the carbinol carbon centers are secondary alcohols, as in **1t** and **1u**, were found to proceed well and provide **2t** and **2u** in 92 and 72% yields, respectively. This is noteworthy as these adducts cannot be prepared following a cross-coupling approach due to the need for the C3 position of the indole ring to be occupied by a functional group so that the C–C bond-forming process can only occur at the C2 position of the N-heterocyclic substrate.[3] Starting 1,4-diols **2v** and **2w**, with a pendant thiophene or alkyne moiety at R^1, were also found to be well tolerated under the reaction conditions, giving the corresponding 2-alkynyl indoles in respective yields of 67 and 61%. Under the standard conditions, reaction of **1q** in which $R^2 = pClC_6H_4$ and $R^4 = Cl$ and **1x** where $R^2 = H$, were the only examples found to give the corresponding 2-alkynyl indoles **2q** and **2x** in lower yields of 45 and 38%, respectively.

A tentative mechanism for the present Ag^I-catalyzed 2-alkynyl indole forming reaction is outlined in Scheme 2. This could initially involve activation of **1** through coordination



Scheme 2. Proposed reaction pathway for the formation of 2-alkynyl indoles.

of the metal catalyst with the sterically less hindered secondary alcohol moiety of the substrate to give the silver(I)-coordinated intermediate **A**. It is possible that this could subsequently trigger 5-*exo-dig* cyclization of the pendant aniline group to the alkyne moiety and formation of 2-vinylidene indolin-3-ol **3**. Further coordination of this newly formed adduct to AgOTf, which is re-generated from [Ag]–OH by protonolysis and also affords a molecule of H_2O, gives Ag^I-activated allene species **B**. A second C–OH bond activation step that initiates deprotonation of the allene moiety followed by elimination of [Ag]–OH,[11] which releases the metal catalyst once again by protonolysis, would then provide **2** and another molecule of water.

While fortuitous, the competitive formation of **3a** for the cyclization of **1a** at room temperature under the conditions mentioned earlier in entry 1, Table 1 argues in favor of the mechanism put forward in Scheme 2. This argument was further corroborated by the observation that when a solution of **3a** in toluene was treated with 5 mol% of AgOTf under the conditions shown in Scheme 3, the expected 2-alkynyl indole **2a** was obtained as the sole product in 92% yield. The role of the silver catalyst in facilitating the two C–OH bond activation steps could also be shown by repeating the

Scheme 3. Dehydrative alkynylation of **3a** catalyzed by AgOTf.

reactions of **1a** and **3a** under similar conditions but in the absence of the catalyst. In both instances, this test led to the recovery of the respective starting alcohols in near quantitative yield.

In summary, we have demonstrated for the first time that the silver(I)-mediated C–OH bond activation of 1,4-propargylic diols is an effective and chemoselective strategy for the construction of 2-alkynyl indoles. The reaction was shown to tolerate a diverse set of starting alcohols and afford the N-heterocycle for applications in natural product synthesis and medicinal and materials chemistry. Previous methods to this immensely important member of the indole family of compounds have mainly relied on synthetic strategies that require a cross-coupling step and structural elements to regioselectively direct alkynylation to occur at the C2 position of the nitrogen ring. Our approach is rapid, forming the indole ring and alkyne side chain of the N-heterocycle sequentially from a wide variety of starting materials and a catalytic system that are low cost, readily available, and ecologically benign.

Experimental Section

General Procedure: A solution of propargylic 1,4-diol **1a** (0.1 mmol) in toluene (2 mL) was added dropwise to a solution of AgOTf (5 μmol) in anhydrous toluene (2 mL) at room temperature. The resulting mixture was stirred at 70 °C for 2 h and monitored by TLC analysis. On completion, the reaction mixture was brought to room temperature and concentrated under reduced pressure. Purification by flash column chromatography on silica gel (eluent: *n*-hexane/EtOAc = 9:1) gave the 2-akynyl indole compound.

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Keywords: alcohols · heterocyclization/alkynylation · homogeneous catalysis · indoles · silver

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[7] Please refer to the Supporting Information for further details.

[8] CCDC-844472 (**2a**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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