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Synthesis of 1,4-Benzoxazepine Derivatives *via* a Novel Domino Aziridine Ring-Opening and Isocyanide-Insertion Reaction

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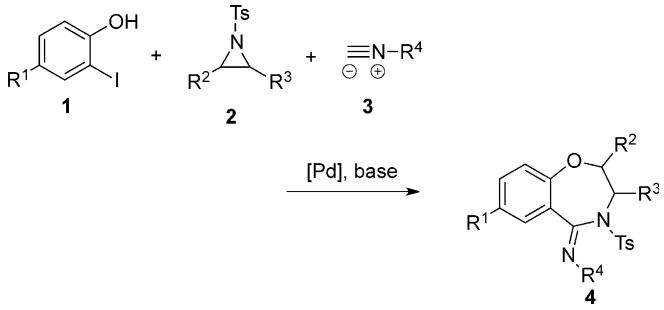
Abstract: A novel and efficient domino process has been developed for the synthesis of 1,4-benzoxazepine derivatives from a range of readily accessible *N*-tosylaziridines, 2-iodophenols and isocyanides. This process involves the aziridine ring-opening reaction with 2-iodophenol, followed by a palladium-catalyzed isocyanide-insertion reaction. This regioselective and high-yielding transformation could be extended to its application for the synthesis of natural products and biologically interesting heterocycles.

Keywords: aziridine ring-opening; 1,4-benzoxazepines; domino process; isocyanide-insertion; regioselectivity

The importance of N-heterocyclic scaffolds in nature products, pharmaceuticals and synthetic materials is well known,[1] thus significant efforts have been devoted to the discovery of methodologies for the preparation of N-heterocyclic compounds in organic chemistry. Among them, 1,4-benzoxazepine derivatives are a very important class of seven-membered rings and such structural units could widely exist in numerous medicinal heterocyclic molecules with promising biological and pharmaceutical activities.[2] These biological characteristics have stimulated organic researchers to explore synthetic methods for 1,4-benzoxazepines and their structural analogues.[3] However, the major drawback of the reported methods is their multistep nature,[3a] which prevents them from generating a large number of structural derivatives efficiently. Over the past decades, domino reactions as one of the most powerful tools for generating complex molecules have attracted more and more attention.[4] This strategy leads to the rapid formation of two or more new bonds under the same conditions, which avoids the separation of the intermediates and minimizes the amount of chemical waste, energy, and time. Thus, the development of a novel domino reaction for the synthesis of 1,4-benzoxazepine derivatives is highly desired.

The aziridines as strained three-membered ring heterocycles are considered to be important building blocks for the synthesis of various N-heterocyclic compounds.[5] They could undergo ring-opening reactions with various nucleophilic reagents to provide a direct access to a range of substituted amines,[6] which are important precursors and have been explored in the synthesis of biological heterocycles.[7] Recently, the Alper group reported a new domino aziridine ring-opening/carboxamidation reaction of *N*-tosylaziridine and 2-halophenols for the synthesis of 1,4-benzoxazepine derivatives.[7a] However, the use of toxic carbon monoxide and the harsh reaction conditions limited the further use and exploration of these reactions.[8] Isocyanides, a kind of unsaturated molecules similar to carbon monoxide, have emerged as versatile building blocks in the construction of medicinally important molecules and natural products.[9] Thus, transition metal-catalyzed reactions involving isocyanides have been studied intensively.[10] This methodology has several advantages such as simple handling, mild conditions and wide substrate scope (variable group at nitrogen) over carbon monoxide.[8] As a result, the transition metal-catalyzed isocyanide-insertion reactions could offer more opportunities for the synthesis of N-heterocyclic compounds. In this context, we designed a palladium-catalyzed domino reaction involving 2-iodophenols **1**, *N*-tosylaziridines **2**, and isocyanides **3**, which would allow an efficient access to 1,4-benzoxazepines **4** by the aziridine ring-opening and isocyanide-insertion events (Scheme 1). To the best of our knowledge, this novel domino strategy based on isocyanides and aziridines has not been reported up to now.



Scheme 1. Approaches towards the 1,4-benzoxazepine structure.

To test the hypothesis, we commenced the investigation with 2-iodophenol **1a** (0.55 mmol), *N*-tosylaziridine of cyclohexene **2a** (0.50 mmol) and *tert*-butyl isocyanide **3a** (0.75 mmol) as model substrates catalyzed by $Pd(PPh_3)_2Cl_2$ (5 mol%) and Cs_2CO_3 (2 equiv.) in toluene under reflux for 24 h. The desired 1,4-benzoxazepine **4a** was obtained in 65% yield (Table 1, entry 1). To our delight, the isolated yield of **4a** was enhanced further to 78% when the amount of base was increased (Table 1, entry 2). However, further raising the amount of base to 4 equiv. could not improve the yield significantly (Table 1, entry 3). When K_2CO_3 and DIPEA were employed as bases instead of Cs_2CO_3, the desired product **4a** was isolated in 62% and 23% yields, respectively (Table 1, entries 4 and 5). Next, several Pd catalysts were screened: it was found that $Pd(PPh_3)_2Cl_2$ was the best choice (Table 1, entries 2, and 6–9). Moreover, no obvious improvement in the yield was observed when the solvent was switched to DMF, MeCN, THF and 1,4-dioxane (Table 1, entries 2, and 10–13).

The structure of the novel 1,4-benzoxazepine **4a** was fully characterized by mass spectrometry, ^1H NMR, ^{13}C NMR spectroscopy and elemental analysis. The *trans* stereochemistry of the product **4a** was confirmed by single-crystal X-ray analysis (Figure 1). According to this single-crystal structure analysis of **4a**, the effect of the steric hindrance may lead to the *Z* configuration of the imino bond (see the Supporting Information).

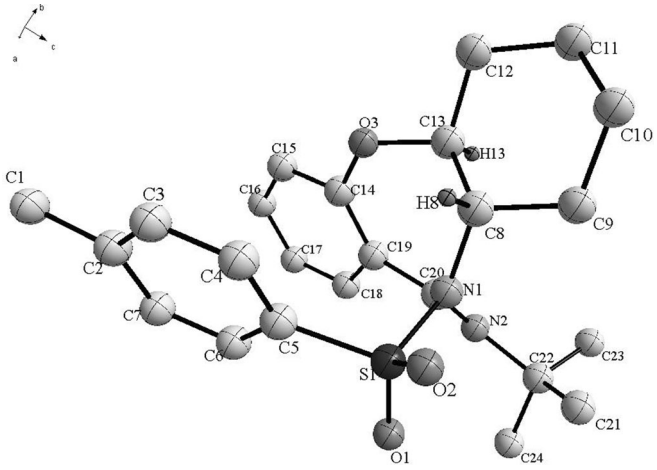
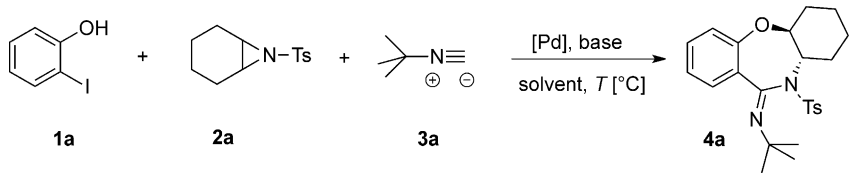


Figure 1. Single-crystal X-ray analysis of **4a**.

Table 1. Optimization of the domino aziridine ring-opening/isocyanide-insertion reaction.[a]

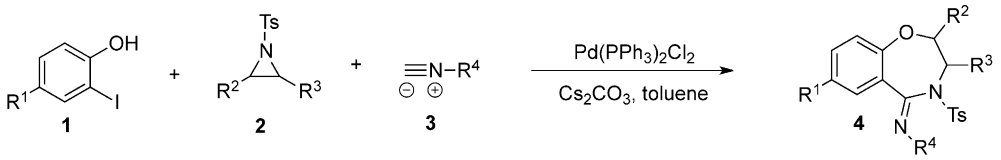


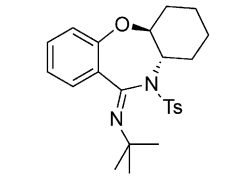
Entry	Catalyst	Base (equiv.)	Solvent	*T* [°C]	Yield [%][b]
1	$Pd(PPh_3)_2Cl_2$	Cs_2CO_3 (2.0)	toluene	110	65
2	$Pd(PPh_3)_2Cl_2$	Cs_2CO_3 (3.0)	toluene	110	78
3	$Pd(PPh_3)_2Cl_2$	Cs_2CO_3 (4.0)	toluene	110	75
4	$Pd(PPh_3)_2Cl_2$	DIPEA (3.0)	toluene	110	23
5	$Pd(PPh_3)_2Cl_2$	K_2CO_3 (3.0)	toluene	110	62
6	$Pd(OAc)_2$	Cs_2CO_3 (3.0)	toluene	110	45
7	$Pd(dppf)Cl_2$	Cs_2CO_3 (3.0)	toluene	110	66
8	$Pd(amphos)_2Cl_2$	Cs_2CO_3 (3.0)	toluene	110	75
9	$PdCl_2$/Xantphos (1:2)	Cs_2CO_3 (3.0)	toluene	110	32
10	$Pd(PPh_3)_2Cl_2$	Cs_2CO_3 (3.0)	DMF	110	72
11	$Pd(PPh_3)_2Cl_2$	Cs_2CO_3 (3.0)	THF	66	67
12	$Pd(PPh_3)_2Cl_2$	Cs_2CO_3 (3.0)	MeCN	81	75
13	$Pd(PPh_3)_2Cl_2$	Cs_2CO_3 (3.0)	1,4-dioxane	100	65

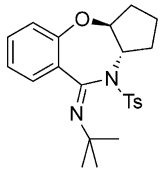
[a] *Reaction conditions:* 2-iodophenol **1a** (0.55 mmol), *N*-tosylaziridine of cyclohexene **2a** (0.50 mmol), *tert*-butyl isocyanide **3a** (0.75 mmol), 5 mol% Pd catalyst, base, solvent (4 mL), 24 h.

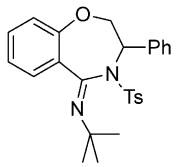
[b] Isolated yield based on **2a**.

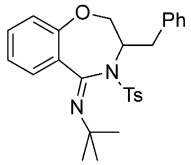
Table 2. Synthesis of 1,4-benzoxazepine derivatives.[a,b]

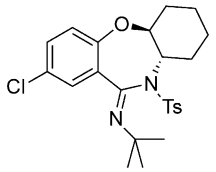


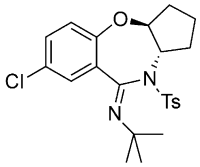

4a 78% (52%)[c]

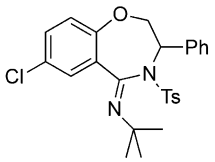

4b 72%

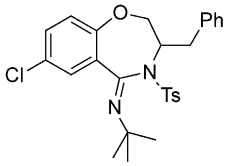

4c 67%

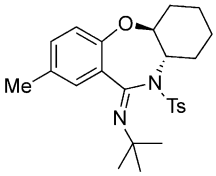

4d 76%

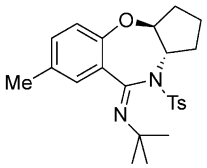

4e 82%

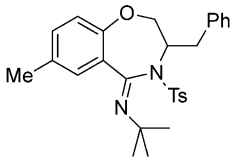

4f 80%

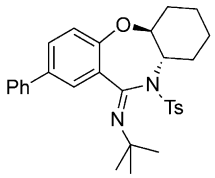

4g 79%

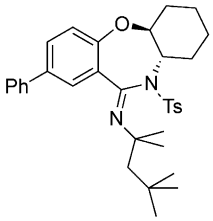

4h 85%

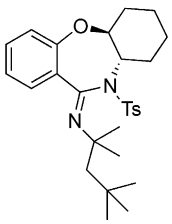

4i 62%

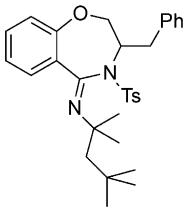

4j 65%

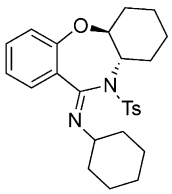

4k 70%


4l 56%


4m 72%


4n 80%


4o 83%

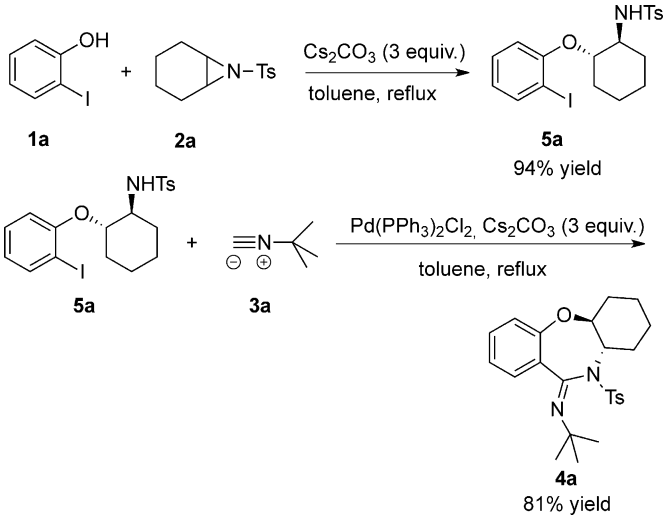

4p 42%

[a] *Reaction conditions:* 2-iodophenols **1** (0.55 mmol), *N*-tosylaziridines **2** (0.50 mmol), isocyanides **3** (0.75 mmol), $Pd(PPh_3)_2Cl_2$ (5 mol%), Cs_2CO_3 (3 equiv.), toluene (4 mL), reflux, 24 h.

[b] Isolated yield based on **2**.

[c] *Reation conditions:* 2-bromophenol (0.55 mmol), *N*-tosylaziridine of cyclohexene (0.50 mmol), *tert*-butyl isocyanide (0.75 mmol), $Pd(PPh_3)_2Cl_2$ (5 mol%), Cs_2CO_3 (3 equiv.), toluene (4 mL), reflux, 24 h.

With these optimized reaction conditions in hand, we turned our attention to the scope of the domino aziridine ring-opening/isocyanide-insertion reaction. Different *N*-tosylaziridines, 2-iodophenols and isocyanides were examined and the results are summarized in Table 2. Various *N*-tosylaziridines, including cyclic and acyclic ones, reacted with *tert*-butyl isocyanide and 2-iodophenol, leading to the desired 1,4-benzoxazepines in high yields (Table 2, **4a–4d**). As for acyclic *N*-tosylaziridines, the reaction was completely regioselective and provided only one regioisomer (Table 2, **4c** and **4d**). The reactions employing various substituted 2-iodophenols bearing a 4-chloro, 4-methyl or 4-phenyl group also worked well to furnish the corresponding 1,4-benzoxazepines (Table 2, **4e**, **4i** and **4l**). Unfortunately, no reaction occurred when 2-iodo-4-nitrophenol was employed in this transformation. We reasoned that the introduction of the electron-withdrawing nitro group decreased the nucleophilic activity of 2-iodo-4-nitrophenol. Next, different isocyanides were also applied to probe the scope of the reaction. Generally, when *tert*-butyl isocyanide and 1,1,3,3-tet-

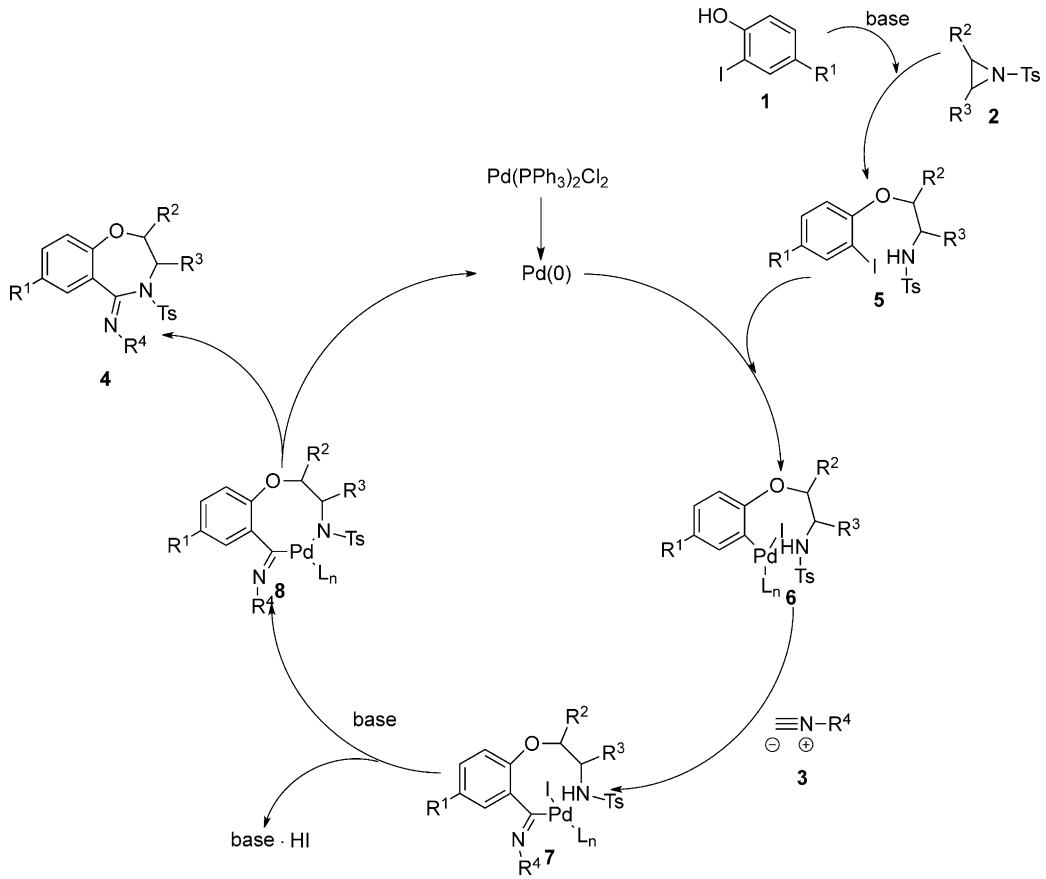


Scheme 2. Investigation of the reaction mechanism.

ramethylbutyl isocyanide were used, the reaction gave the corresponding products in high yields (Table 2, **4a** and **4n**). However, when cyclohexyl isocyanide was applied in this process under the optimized conditions, the desired product was only obtained in moderate yield (Table 2, **4p**). Finally, the treatment of 2-bromophenol, *N*-tosylaziridine of cyclohexene and *tert*-butyl isocyanide afforded the corresponding 1,4-benzoxazepine **4a** in only 52% yield. The lower rate of oxidative addition of the Ar-Br moiety to the *in situ* formed palladium(0) species may lead to the difference in behaviour compared to that of 2-iodophenol.

To gain insights of the reaction mechanism, several control experiments were conducted as shown in Scheme 2. When 2-iodophenol **1a** and *N*-tosylaziridine **2a** were treated in toluene using Cs_2CO_3 (3 equiv.) as a base, the ring-opening product **5a** was obtained in up to 94% yield. Next, amide **5a** was reacted with *tert*-butyl isocyanide **3a** under the optimized conditions, leading to the desired product **4a** in 81% yield. The experimental results revealed that this domino reaction may proceed through the base-catalyzed ring-opening of *N*-tosylaziridines with 2-iodophenols, and then the palladium-catalyzed isocyanide-insertion reaction.

On the basis of these preliminary results and the control experiments conducted, a possible catalytic cycle involved in the present process was outlined as shown in Scheme 3.[7a,8] Initially, the base-catalyzed ring-opening reaction of *N*-tosylaziridine **2** with 2-iodophenol **1** generates the amide **5**. The oxidative ad-



Scheme 3. Possible mechanism of the domino aziridine ring-opening/isocyanide-insertion reaction.

dition of **5** to the *in situ* generated palladium(0) species leads to the palladium complex **6**. This could then undergo the isocyanide-insertion process to give the intermediate **7**. Next, with the aid of the base, hydroiodic acid is extruded out of **7** to generate the eight-membered product **8**. Finally, reductive elimination affords **4**, regenerating the palladium (0) catalyst.

In conclusion, we have demonstrated a novel and efficient procedure for the synthesis of a new class of substituted 1,4-benzoxazepine derivatives *via* a palladium-catalyzed three-component domino reaction of *N*-tosylaziridines, 2-iodophenols and isocyanides. This process involved the aziridine ring-opening reaction with 2-iodophenol, followed by palladium-catalyzed isocyanide-insertion reaction. This domino reaction was successful with a range of *N*-tosylaziridines, 2-iodophenols and isocyanides, and could afford the desired products in moderate to high yields. More importantly, this methodology may be instrumental to the rapid construction of important 1,4-benzoxazepine derivatives with promising biological and pharmaceutical activities.

Experimental Section

General Procedure for 1,4-Benzoxazepine Derivatives

A mixture of 2-iodophenol (0.55 mmol), *N*-tosylaziridine (0.50 mmol), isocyanide (0.75 mmol), $Pd(PPh_3)_2Cl_2$ (18 mg) and Cs_2CO_3 (489 mg) was stirred in toluene (4 mL) under reflux for 24 h. Upon completion of the reaction, the mixture was concentrated under vacuum and the resulting residue was purified by column chromatography.

Characterization Data of a Representative 1,4-Benzoxazepine (4a)

White solid; 144–146 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ = 1.14–1.15 (m, 1H), 1.32–1.47 (m, 2H), 1.59 (s, 9H), 1.50–1.60 (m, 1H), 1.77–1.84 (m, 2H), 2.06–2.08 (m, 1H), 2.31 (s, 3H), 2.44–2.47 (m, 1H), 3.67–3.72 (m, 1H), 3.92–3.98 (m, 1H), 6.23 (d, J = 8.0 Hz, 1H), 6.91–6.98 (m, 3H), 7.07–7.12 (m, 3H), 7.63–7.65 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ = 20.47, 22.79, 23.51, 29.85, 32.03, 32.85, 57.72, 64.65, 79.38, 117.35, 120.62, 124.75, 126.80, 127.65, 129.26, 132.12, 134.13, 137.72, 141.74, 156.32; MS (ESI): m/z = 427 [M+H]; anal. calcd. for $C_{24}H_{30}N_2O_3S$: C 67.58, H 7.09, N 6.57; found: C 67.67, H 7.1, N 6.55.

Crystallographic data for 1,4-benzoxazepine **4a** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 937174. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgements

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