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Domino Ring-Opening/Carboxamidation Reactions of *N*-Tosyl Aziridines and 2-Halophenols/Pyridinol: Efficient Synthesis of 1,4-Benzo- and Pyrido-oxazepinones

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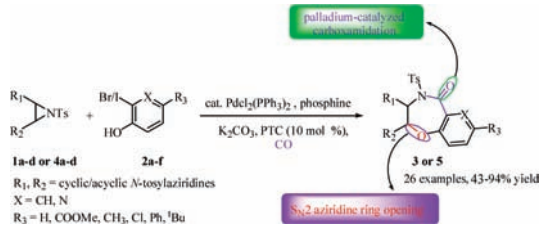
ABSTRACT



A domino process is described for the synthesis of 1,4-benzo- and pyrido-oxazepinones by one-pot sequential ring-opening/carboxamidation reactions of various *N*-tosylaziridines with a range of 2-halophenols/pyridinol under phase-transfer catalysis.

The strained three-membered ring heterocycles, aziridines, are considered to be versatile building blocks for the synthesis of various valuable nitrogen-containing compounds because of their unique ability to function as carbon electrophiles.[1] Aziridines can undergo a ring-opening reaction with various nucleophiles to provide direct access to a range of substituted amines.[2] During the last several decades, domino reactions have attracted significant attention by many organic chemists to prepare complex molecules, as they can enhance the synthetic efficiency, avoid the separation of intermediates, and reduce the amount of waste.[3] Thus, the development of new methods for the simultaneous formation of two or more new bonds (usually C−C and C−N(O) bonds) under the same reaction conditions in a single step is quite advantageous since it allows the rapid buildup of molecular complexity from relatively simple starting materials.[4] Transition-

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metal-catalyzed reactions, particularly palladium-catalyzed reactions, are a valuable synthetic tool for achieving this goal.[5,6]

In the context of our research interest on the chemistry of aziridines[7] and application of Pd-catalyzed domino processes for the preparation of various heterocycles,[8] we report a new domino ring-opening/carboxamidation reaction of *N*-tosylaziridines and 2-halophenols/pyridinol under phase-transfer catalysis to give 1,4-benzo- and pyrido-oxazepinones in fine yields (Figure 1).[9−12] It is well-known that many compounds

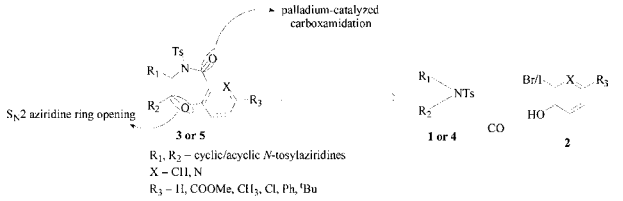
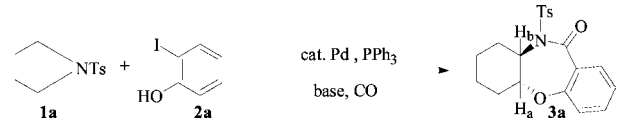


Figure 1. Strategy for the domino ring-opening/carboxamidation reaction.

possessing benzo- and pyrido-oxazepinone scaffolds display biological activity of medicinal interest.[13]

Initially, the domino ring-opening/carboxamidation reactions were examined by reacting the *N*-tosylaziridine of cyclohexene **1a** (1 mmol) with 2-iodophenol **2a** (1.1 mmol) at 300 psi of carbon monoxide in the presence of $Pd(OAc)_2$ (0.03 mmol), triphenylphosphine (PPh_3) (0.03 mmol), and Cs_2CO_3 (3 equiv) in THF at 110 °C for 24 h. The reaction afforded 60% yield of the desired 1,4-benzoxazepinone **3a** (Table 1, entry 1). No significant improvement in the yield of the product **3a** was observed when the nature of the Pd catalyst was changed, and the reaction was carried out at low pressure of carbon monoxide and lower temperature (Table 1, entries 2 and 3). The change of the base from Cs_2CO_3 to K_2CO_3 provided 66% yield of the desired 1,4-benzoxazepinone **3a** (Table 1, entry 4). As solvent can have a significant effect on the reaction, we have further studied this domino reaction in the more polar solvent dioxane. However, change of the solvent proved less efficient and

Table 1. Optimization of the Domino Reaction Conditions for the Reaction of *N*-Tosylaziridine of Cyclohexene **1a** with 2-Iodophenol **2a**[a]



entry	catalyst/phosphine (mol %)	CO (psi)	base	*T* (°C)	**3a** (%)[b]
1	$Pd(OAc)_2$ (3)/PPh_3 (3)	300	Cs_2CO_3	110	60
2	$Pd(OAc)_2$ (3)/PPh_3 (3)	300	Cs_2CO_3	80	63
3	$PdCl_2(PPh_3)_2$ (3)/PPh_3 (3)	200	Cs_2CO_3	80	65
4	$PdCl_2(PPh_3)_2$ (1.5)/PPh_3 (1.5)	200	K_2CO_3	80	66
5	$PdCl_2(PPh_3)_2$ (1.5)/PPh_3 (1.5)	200	K_2CO_3	80	61[c]
6	$PdCl_2(PPh_3)_2$ (1.5)/PPh_3 (1.5)	200	K_2CO_3	80	76[d]

[a] Reaction conditions: **1a** (1 mmol), **2a** (1.1 mmol), THF (5 mL), 24 h. [b] Isolated yield. [c] Reaction was carried out in dioxane. [d] With phase-transfer catalyst benzyltriethylammonium chloride (TEBA, 10 mol %).

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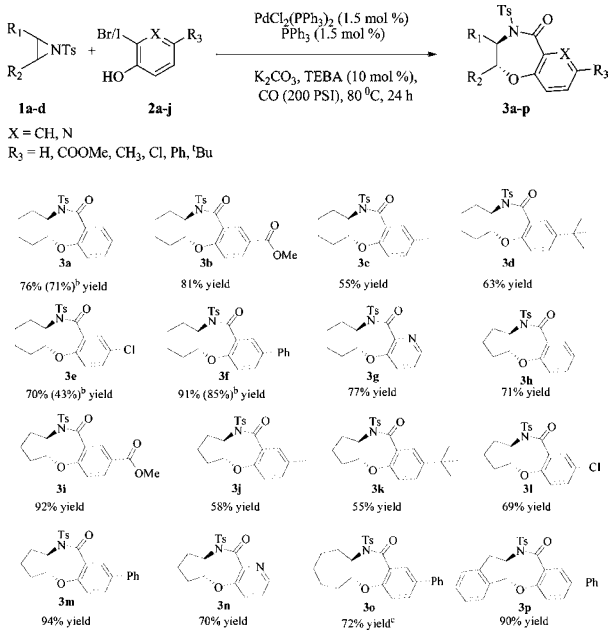
Table 2. Domino Ring-Opening/Carboxamidation Reaction of Cyclic *N*-Tosylaziridines **1a**−**d** with 2-Halophenols/Pyridinol **2a**−**j**[a]



[a] Reaction conditions: **1a**−**d** (1.0 mmol), **2a**−**j** (1.1 mmol), $PdCl_2(PPh_3)_2$ (1.5 mol %), triphenylphosphine (1.5 mol %), TEBA (10 mol %), K_2CO_3 (3 equiv), THF (5 mL), 80 °C, 24 h. [b] Isolated yield with 2-bromophenols. Reaction conditions: *N*-tosylaziridines (1.0 mmol), 2-bromophenols (1.1 mmol), $PdCl_2(PPh_3)_2$ (3 mol %), dppf (6 mol %), 18-crown-6 (10 mol %), K_2CO_3 (3 equiv), DMF (5 mL), 130 °C, 48 h. [c] Reaction was carried out in DMF with 400 psi CO pressure at 110 °C, 24 h.

did not increase the isolated yield of the desired product (Table 1, entry 5). The yield of **3a** increased to 76% when the phase-transfer catalyst, benzyltriethylammonium chloride (TEBA, 10 mol %), was used (Table 1, entry 6).[14]

The stereochemistry of the product was determined by measuring the coupling constant of the methine proton (H_a) and by 1D NOE and the NOESY NMR spectra of the product **3a**. *Trans* stereochemistry was determined by the coupling constant of the proton H_a at 3.71 ppm (ddd, 1H, $J = 11.53$, 11.50, and,3.35 Hz) and further confirmed by 1D NOE and NOESY spectra as no NOE effects were detected between H_a and H_b (see Supporting Information).

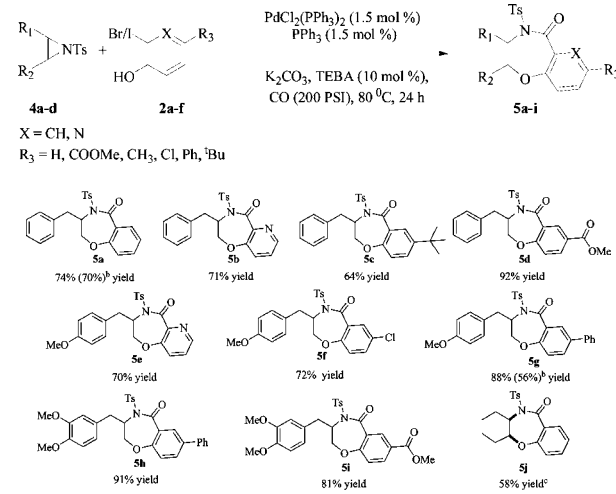
Under the optimized reaction conditions, the scope of this reaction was further examined by treating different *N*-tosylaziridines with a variety of 2-halophenols/pyridinol. The results are summarized in Table 2. The reaction of the *N*-tosylaziridine of cyclohexene **1a** with 2-iodophenol bearing electron-donating (Table 2, products **3c** and **3d**) and electron-withdrawing (Table 2, product **3e**) groups provides lower yields of the desired 1,4-benzoxazepinones. Similar results were obtained with *N*-tosylaziridines of cyclopentene **1b** on reacting with 2-iodophenols **2c**−**e** (Table 2, products **3j**−**l**).

In contrast, 2-iodophenols bearing phenyl and methyl ester groups provide higher yields of the corresponding 1,4-benzoxazepinones on domino reaction with *N*-tosylaziridines **1a** and **2a** (Table 2, products **3b**, **3f**, **3i**, and **3m**). The domino reaction of *N*-tosylaziridines **1a**,**b** with 2-iodopyridinol also afforded the corresponding 1,4-pyridoxazepinones in good yield (Table 2, products **3g** and **3n**).

It is interesting to note that the domino reaction of seven-membered ring aziridines **1c** with 2-iodophenol **2a** does not provide the desired 1,4-benzoxazepinone under the optimized reaction conditions. However, when the reaction was carried out at 110 °C with 400 psi carbon monoxide pressure in DMF for 24 h, the desired product was isolated in 72% yield (Table 2, product **3o**). The domino reaction also works well with indene *N*-tosylaziridine **1d** (Table 2, product **3p**).

Apart from the cyclic *N*-tosylaziridines, various acyclic *N*-tosylaziridines **4a**−**d** were also examined for this domino ring-opening/carboxamidation reaction, and the results are listed in Table 3. A variety of *N*-(*p*-toluenesulfonyl)-2-benzylaziridines **4a**−**c** were reacted under the optimized reaction conditions with different 2-iodophenols/pyridinol to afford the corresponding 1,4-benzo- and pyrido-oxazepinones in 64−91% isolated yields (Table 3). The reaction is completely regioselective and provides only one regioisomer. The domino reaction with *N*-(*p*-toluenesulfonyl)-2,3-diethylaziridine required higher temperature (110 °C), CO pressure (400 psi), and polar solvent DMF to afford 1,4-benzoxazepinone in moderate yield (Table 3, product **5j**).

Table 3. Domino Ring-Opening/Carboxamidation Reaction of Acyclic *N*-Tosylaziridines **4a**−**d** with 2-Halophenols/Pyridinol **2a**−**f**[a]



[a] Reaction conditions: **1a**−**d** (1 mmol), **2a**−**j** (1.1 mmol), $PdCl_2(PPh_3)_2$ (1.5 mol %), triphenylphosphine (1.5 mol %), TEBA (10 mol %), K_2CO_3 (3 equiv), THF (5 mL), 80 °C, 24 h. [b] Isolated yield with 2-bromophenols. Reaction conditions: *N*-tosylaziridines (1 mmol), 2-bromophenols (1.1 mmol), $PdCl_2(PPh_3)_2$ (3 mol %), dppf (6 mol %), 18-crown-6 (10 mol %), K_2CO_3 (3 equiv), DMF (5 mL), 130 °C, 48 h. [c] Reaction was carried out in DMF with 400 psi CO pressure at 110 °C, 24 h.

This domino reaction was next studied with 2-bromophenol. Thus, *N*-tosylaziridine of cyclohexene **1a** was reacted with 2-bromophenol **2h** under the optimized reaction condi-

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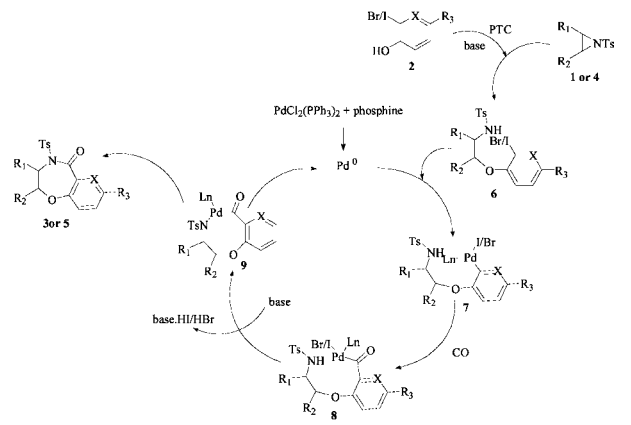

Figure 2. Proposed reaction mechanism.

tions; however, none of the desired product was obtained. Increasing the reaction temperature (110 °C) or CO pressure (300 psi) or using a different phosphine ligand (dppp, P^tBu_3) with toluene as the solvent was also unsuccessful. After some experimentation, we found that this type of domino reactions works well by employing $PdCl_2(PPh_3)_2$ (3 mol %) as the Pd catalyst, bisdiphenylphosphino ferrocene (dppf, 6 mol %), K_2CO_3 (3 equiv) as the base, and 18-crown-6 as the PTC at 400 psi CO pressure in DMF at 130 °C for 48 h.[15,16] Under these conditions, the starting material was converted to 1,4-benzoxazepinone **3a** in 71% isolated yield (Table 2, product **3a**). Several other 2-bromophenols were used for this type of domino reaction with various *N*-tosylaziridines under the above-mentioned optimized reaction conditions to give desired 1,4-benzoxazepinones in good to excellent yields (Table 2, products **3e** and **3f**, Table 3, products **5a** and **5g**).

A possible reaction mechanism for the formation of benzo- and pyrido-oxazepinones **3** or **5** is shown in Figure 2. First, the base-catalyzed ring-opening of *N*-tosylaziridines **1** or **4** with 2-halophenols/pyridinol **2** under phase-transfer catalysis condition generates the amine **6**. The oxidative addition of **6** to the in situ generated palladium(0) species[17] leads to a palladium complex **7**. Insertion of carbon monoxide into the aryl carbon−palladium bond of **7** affords **8**, and nucleophilic attack of the tosyl-amine on an aroylpalladium complex gives intermediate **9**. The latter undergoes reductive elimination affording 1,4-benzo- and pyrido-oxazepinones **3** or **5** with regeneration of palladium(0).

In summary, we have developed a new efficient synthetic method for the synthesis of 1,4-benzo- and pyrido-oxazepinones via a domino process through one-pot ring-opening/carboxamidation reaction sequences of *N*-tosylaziridines with 2-halophenols/pyridinol under phase-transfer conditions. The method works efficiently with a range of *N*-tosylaziridines and 2-halophenols/pyridinol to provide facile access to a variety of 1,4-benzo- and pyrido-oxazepinones.

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Supporting Information Available: Experimental procedures, characterization data for all new compounds, and copies of ^1H NMR and ^{13}C NMR spectra for substrates and products. This material is available free of charge via the Internet at http://pubs.acs.org.

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