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Nickel(II)-catalyzed diastereoselective [3 + 2] cycloaddition of *N*-tosyl-aziridines and aldehydes *via* selective carbon–carbon bond cleavage†

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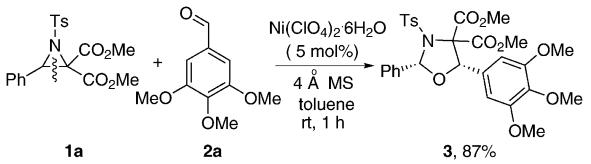
An efficient and mild $Ni(ClO_4)_2$-catalyzed [3 + 2] cycloaddition of *N*-tosylaziridines and aldehydes *via* C–C bond cleavage was developed. The cycloaddition reaction proceeds with high diastereoselectivity and regioselectivity leading to highly substituted 1,3-oxazolidines. Notably, this novel reaction can be easily expanded to gram level scale and the thermal conditions cannot achieve the same transformation.

Among the charming panorama of ylides, azomethine ylides (AMY) represent a particular case of useful intermediates,[1] providing general and direct access to five-membered heterocycles. As a highly strained cyclic amine, aziridines can easily undergo C–N bond cleavage to work as a masked $1_C,3_N$-ylide, which has been extensively exploited in organic synthesis.[2] However, aziridines are also known to react by thermal or photochemical electrocyclic ring-opening at the carbon–carbon bond to give azomethine ylides, as useful reactive species for a wide range of synthetic applications.[3] For example, Schirmeister[3k] has confirmed the formation of ylide species of the aziridine-2,2-dicarboxylates by the photochromism and reactions with dipolarophiles (alkenes or aldehydes) under thermal conditions. A few cases of Lewis acids favoring the C–C bond heterolysis of donor–acceptor aziridines under mild conditions, leading to the corresponding *N*-arylated azomethine ylides were reported in the past years.[4] Engels[5] has investigated the C–N *vs.* C–C bond breakages of the aziridine derivates through computation, and yet recently Johnson[4b] has achieved the first productive [3 + 2] or [4 + 2] cycloadditions of *N*-phenyl aziridine dicarboxylates with electron-rich olefins mediated by $ZnCl_2$ (1.2 equiv). We have been engaged in the selective carbon–carbon bond cleavage of heterocycle motifs for a while,[6] and very recently, we have expanded this conversion to related *N*-tosylaziridines with their facile conversion to highly substituted pyrrolidines by reaction with electron-rich alkenes.[6c] Inspired by this, we wish to document a novel and efficient synthesis of 1,3-oxazolidines with highly diastereoselectivity by the formal [3 + 2] cycloaddition of aldehydes with AMYs[7] generated from *N*-tosylaziridine dicarboxylates *via* C–C bond cleavage under mild conditions.[8] 1,3-oxazolidines are common structural units in many natural products,[9a] designed medicinal agents,[9b] chiral auxiliaries,[9c–e] and precursors of α-amino-β-hydroxyl compounds in organic synthesis.[3o,7i,9f,g]

Our studies in this area began by choosing *N*-tosylaziridine **1a**[10] and 3,4,5-trimethoxybenzaldehyde **2a** as the model substrates to test the possibility of formal [3 + 2] cycloaddition under the catalysis of a Lewis acid. A representative selection of Lewis acids including $Sc(OTf)_3$, $Mg(OTf)_2$, MgI_2, $Fe(OTf)_3$, $Cu(OTf)_2$, $In(OTf)_3$, $Sn(OTf)_2$, $Yb(OTf)_3$, and $Ni(ClO_4)_2{\cdot}6H_2O$ in various solvents at room temperature were tested (see Supporting Information†). To our delight, the reaction worked very well in toluene at room temperature with 4 Å molecular sieves as an additive by using 1.5 equiv of **2a** in the presence of 5 mol% of $Ni(ClO_4)_2{\cdot}6H_2O$ catalyst, which provides the highly substituted oxazolidine **3** in 87% isolated yield as a single *cis*-isomer [eqn (1)]. Without 4 Å molecular sieves, the starting material easily undergoes decomposition by reacting with trace amount of water in the system under the reaction conditions. $Yb(OTf)_3$ is another effective and potential catalyst to give **3** in 84% yield. Other tested catalysts or solvents failed to improve the yields. The structure and relative stereochemistry of **3** (racemic) were established by X-ray crystallography analysis.†[11] Surprisingly, when the reaction was run at 90 °C in toluene without the catalyst, no desired product was observed, but instead, an unexpected oxazole **4**[12] was isolated in 57% yield [eqn (2)], indicating the reaction proceeds through a different reaction pathway under thermal conditions with those under the catalysis of Lewis acid.



1a **2a** **3**, 87%

(1)

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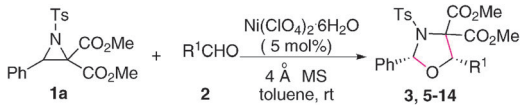
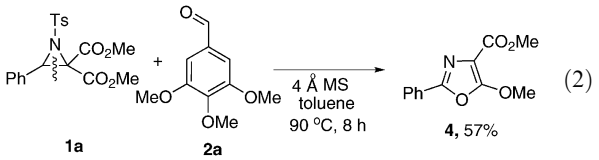
Table 1 Study of the reaction scope by variation of aldehyde component[a,c]



Entry	R^1CHO **2**	Time (h)	Isolated yield (%)
1	PhCHO (**2b**)	2	**5** (67)
2	3-MeC_6H_4CHO (**2c**)	1	**6** (64)
3	4-^{i}PrC_6H_4CHO (**2d**)	1	**7** (72)
4	4-$MeOC_6H_4CHO$ (**2e**)	1	**8** (92)
5	2-$MeOC_6H_4CHO$ (**2f**)	1	**9** (55)
6[b]	4-ClC_6H_4CHO (**2g**)	3	**10** (50)
7[b]	4-BrC_6H_4CHO (**2h**)	3	**11** (55)
8	Furan-2-carbaldehyde (**2i**)	1	**12** (80)
9[b]	1-Naphthaldehyde (**2j**)	2	**13** (70)
10	*E*-Styryl (**2k**)	1	**14** (87)
11[d]	3,4,5-$(MeO)C_6H_2CHO$ (**2a**)	8	**3** (89)

[a] Standard conditions: **1a** (0.4 mmol), **2** (1.5 equiv.), 5 mol% catalyst, and 200 mg of activated 4 Å MS in 4 mL of toluene at room temperature. [b] 3 equiv. of aldehydes were used. [c] All the reactions afforded the *cis*-product as the single isomer. [d] **1a** (10 mmol), 1.5 equiv. of **2a**, and 1 mol% of catalyst were used.

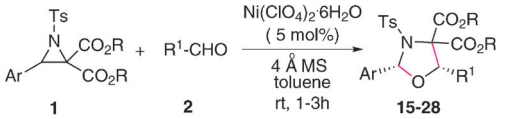


(2)

With the optimal reaction conditions in hand, the scope of the Ni(II)-catalyzed formal [3 + 2] cycloaddition reaction was explored by variation of aldehyde component **2**, and the results are summarized in Table 1. In general, the cycloadditions of aziridine **1a** with electron-rich aromatic aldehydes work better than those with electron-deficient aromatic aldehydes. The reactions of aziridine **1a** with benzaldehyde **2b**, 3-methylbenzaldehyde **2c** and 4-isopropylbenzaldehyde **2d** proceed smoothly to afford the corresponding cycloadducts in 64–72% yields under standard conditions (Table 1, entries 1–3). 92% yield of the cycloadduct **8** can be achieved by using 4-methoxybenzaldehyde **2e** as dipolarophile, while the reaction of 2-methoxybenzaldehyde **2f** with **1a** only gives moderate yield, which may be caused by the steric interactions (Table 1, entries 4–5). The reactions of 4-chlorobenzaldehyde **2g** and 4-bromobenzaldehyde **2h** require longer time to give the corresponding products **10–11** in moderate yields (Table 1, entries 6–7) with further convertible functional groups (Br or Cl). Gratifyingly, furan-2-carbaldehyde **2i**, 1-naphthaldehyde **2j**, and even cinnamaldehyde **2k** can be used as the dipolarophiles to give the corresponding products **12–14** in high yields (Table 1, entries 8–10). It should be noteworthy that all the reactions afford exclusively the *cis*-isomer.

We next turned to study the scope of this reaction by variation of the aziridine component (Table 2). In general, the desired products can be obtained in excellent yields with excellent diastereoselectivity. Both electron-withdrawing and electron-donating groups can be introduced to the aryl moiety of the aziridine **1** (Table 2, entries 1–7) and it seems that there is no substituent effect on the reactivity. The reactions of *N*-tosyl-aziridines **1i** and **1j** also work very well under standard conditions to give the corresponding products **22** and **23** in 82%

Table 2 Study of the reaction scope by variation of aziridine component[a]



	Aziridine **1**			
Entry	Ar	R	R^1CHO **2**	Isolated yield (%)
1	4-$NO_2C_6H_4$	Me (**1b**)	**2a**	**15** (99)
2	4-ClC_6H_4	Me (**1c**)	**2a**	**16** (83)
3	4-BrC_6H_4	Me (**1d**)	**2a**	**17** (97)
4	4-MeC_6H_4	Me (**1e**)	**2a**	**18** (84)
5	4-^{i}PrC_6H_4	Me (**1f**)	**2a**	**19** (84)
6	3-MeC_6H_4	Me (**1g**)	**2a**	**20** (88)
7	2-BrC_6H_4	Me (**1h**)	**2a**	**21** (90)
8	Ph	Et (**1i**)	**2a**	**22** (82)
9	Ph	^{i}Pr (**1j**)	**2a**	**23** (90)
10	4-^{i}PrC_6H_4	^{i}Pr (**1k**)	**2a**	**24** (90)
11	4-$NO_2C_6H_4$	Me (**1b**)	**2b**	**25** (84)
12	4-$NO_2C_6H_4$	Me (**1b**)	**2e**	**26** (96)
13[b]	4-$NO_2C_6H_4$	Me (**1b**)	**2g**	**27** (70)
14[c]	Ph	Me (**1l**)	**2a**	**28** (82)

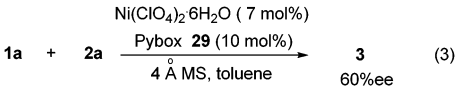
[a] Standard conditions: **1** (0.4 mmol), **2** (1.5 equiv.), 5 mol% catalyst, and 200 mg of activated 4 Å MS in 4 mL of toluene at room temperature. [b] 3 equiv. of aldehyde was used. [c] The *N*-Ts group was replaced by *N*-Ns group.

and 90% isolated yields, respectively, indicating the ester group does not affect the reaction (Table 2, entries 8–9). The results of **1b** with aldehydes **2b**, **2e** and **2g** further indicate that electron-rich aryl aldehydes work better than those electron-deficient ones (Table 2, entries 11–13). It is noteworthy that the replacement of tosyl by the nosyl group in **1a** (**1l**) did not alter the reactivity of the corresponding aziridine to produce the desired *N*-Ns oxazolidine **28** in 82% yield (Table 2, entry 14).

Furthermore, this novel reaction can be easily scaled up to 10 mmol with only 1 mol% catalyst loading in a slightly higher yield, demonstrating the synthetically utility of this protocol (Table 1, entry 11). Gratifyingly, a preliminary experiment showed that an enantioselective variant of this [3 + 2] cycloaddition can be achieved by the application of commercially available Pybox **29**[13] as the chiral ligand. The reaction of **1a** and **2a** proceeds well at room temperature to give the *cis*-1,3-oxazolidine in 60% ee with a good yield [eqn (3), Scheme 1]. Though the ee value is only moderate at present stage, it still demonstrates that an asymmetric reaction is feasible using this approach.

The plausible reaction pathways leading to oxazolidines and oxazole **4** are proposed (see Supporting Information†). By coordination of $Ni(ClO_4)_2$ to the dicarboxylates, the aziridines are liable to give AMYs, which would be trapped by the aldehydes subsequently.[6a,c] However, when heated, the unavoidable proximity of the generated azomethine ylide to the C=O bond of either of the two ester groups may lead to a rapid 1,5-dipolar electrocyclization, thus gives a different product.[2q]

In summary, we have discovered a novel and efficient $Ni(ClO_4)_2$-catalyzed carbon-carbon bond cleavage of *N*-tosylaziridines,



(3)

Scheme 1 Catalytic enantioselective variant.

which provides a mild, highly diastereo- and regioselective method for synthesis of highly substituted oxazolidines through [3 + 2] cycloaddition of the derived *N*-tosyl metal coordinated ylides and aldehydes. The reaction undergoes a different reaction pathway under the classical thermal conditions. Moderate enantioselectivity can be achieved by application of Pybox as the chiral ligand, which may be improved by further modification. Furthermore, this protocol is very useful in organic synthesis, considering the high efficiency of the catalyst, as well as the convenient preparation of the substrates.[10] Further studies including the reaction scope, synthetic application and examination of other asymmetric catalysis are ongoing and will be reported in due course.

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11 CCDC 795474 (**3**) 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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