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Iodobenzene Diacetate/Tetrabutylammonium Iodide-Induced Aziridination of *N*-Tosylimines with Activated Methylene Compounds under Mild Conditions

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Abstract: Aziridination of *N*-tosylimines with activated methylene compounds induced by iodobenzene diacetate [$PhI(OAc)_2$] and tetrabutylammonium bromide [Bu_4NBr] afforded the corresponding 2,2-difunctionalized aziridines in good yields with the aid of a catalytic amount of base. The reaction is hypothesized to proceed *via* a tandem nucleophilic addition-oxidative cyclization pathway.

Keywords: cycloaddition; hypervalent compounds; imines; methylene compounds; nitrogen heterocycles; oxidation

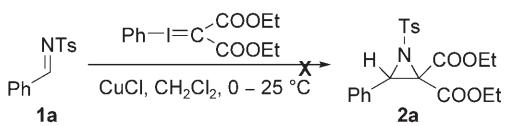
Introduction

Aziridines are versatile intermediates in organic synthesis. They not only appear as a three-membered heterocyclic substructure in many biologically important natural products, but also can undergo ring opening reaction to afford other building blocks in synthesis.[1,2] The addition of a carbon to a C=N bond is the most attractive approach in ring formation. The reaction between imines and ylide,[3,4] along with the aza-Darzens reaction,[5] is well-documented to synthesize functionalized aziridines. Compared to the extensive studies on the synthesis of 2-monofunctionalized aziridines,[6] the preparation of 2,2-difunctionalized aziridines has, however, received much less scrutiny. Aziridine-2,2-dicarboxylates, for example, have recently been identified as protease inhibitors.[7] As a consequence, they are considered as potentially attractive starting materials for biologically active substances.[8] Cardillo et al. reported the synthesis of aziridine-2,2-dicarboxylates *via* a two-step reaction involving the conjugated addition of hydroxylamino derivatives (TMS-NH-OTMS) to alkylidene malonates, followed by cyclization under basic conditions.[9] Pellacani et al. demonstrated that aziridine-2,2-dicarboxylates could be prepared by the aziridination of electron-deficient C=C bonds with nosyloxy carbamates (NsO-NH-COOR) in the presence of CaO.[10] This procedure, however, cannot be extended to 2-benzylidenemalonates. The synthesis of 2,2-difunctionalized aziridines by the direct cycloaddition of imines with methylene compounds, if applicable, would be desirable in many applications. As a part of our development of the synthetic application of sulfonamides in the presence of hypervalent iodine,[11] we report herein the results regarding the aziridination of *N*-tosylimines with methylene compounds activated by the combination of iodobenzene diacetate [$PhI(OAc)_2$] and tetrabutylammonium bromide (Bu_4NBr).

Results and Discussion

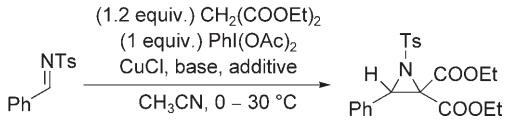
Although the synthetic application of β-dicarbonyl iodonium ylides in the transition metal-catalyzed cyclopropanation of C=C bond has been well developed,[12,13] the aziridination of C=N bond with β-dicarbonyl iodonium ylides remains to be investigated. An intermolecular aziridination of *N*-tosylimine **1a** with phenyliodonium ylide was initially tested, but no aziridine ring was formed under the typical cyclopropanation conditions (Scheme 1).

To avoid the decomposition of the phenyliodonium ylide,[14] a one-pot reaction was tested but failed (Table 1, entry 1). When Bu_4NI was added as a phase-



Scheme 1.

Table 1. Exploration of the one-pot aziridination of *N*-toysl-imine **1a** with diethyl malonate.



Entry	CuCl [equiv.]	Base [equiv.]	Additive [equiv.]	**2a** [%][a]
1	0.1	KOH (2)		0
2	0.1	KOH (2)	Bu_4NI (1)	2
3	0.1	KOH (2)	18-crown-6 (1)	0
4	0.1	KOH (2)	Bu_4NBr (1)	8
5	0.1	KOH (2)	Bu_4NCl (1)	0
6	0.1	*t*-BuOK (2)	Bu_4NBr (1)	15
7	0	*t*-BuOK (2)	Bu_4NBr (1)	17

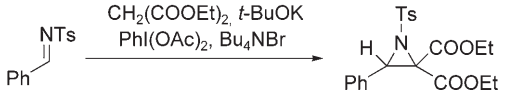
[a] Isolated yield based on imine **1a**.

transfer catalyst aziridination occurred and product **2a** was isolated in 2% yield (entry 2). As a comparison, the control experiment with 18-crown-6 as phase-transfer catalyst failed to achieve aziridination (entry 3). The yield of **2a** increased to 8% using Bu_4NBr (entry 4), while no aziridination product was detected in the presence of Bu_4NCl (entry 5). Because a ring opening product of **2a** attacked by a hydroxy group was isolated as one of the by-products, KOH was replaced by the less nucleophilic base *t*-BuOK. As a result a higher yield of **2a** was obtained (entry 6). It was, however, surprising to find that aziridination still occurred even in the absence of a metal catalyst (entry 7).

Further optimization of the reaction conditions is shown in Table 2. The best ratio of imine **1a**, $CH_2(COOEt)_2$, $PhI(OAc)_2$, Bu_4NBr and *t*-BuOK was 1:1.2:2:2:0.5, with which the yield of **2a** increased to 83% (entry 7). It was noteworthy that a catalytic amount of base could drastically promote the aziridination. The reaction still proceeded smoothly even using 0.25 equiv. of *t*-BuOK (entry 8). Control experiment showed that no aziridine was formed in the absence of *t*-BuOK (entry 9). In processes involving, e.g., the reaction between ylides and imines and the aza-Darzens reaction, a stoichiometric amount of base is normally required. The aziridination of *N*-tosylimine with malonate could proceed with varied efficiency in various solvents except water, in which no aziridine was isolated (entries 10–17). The major by-product of the reaction was diethyl 2-benzylidenemalonate. This was assumed to be generated from the Knoevenagel condensation of imine **1a** with diethyl malonate.[11c]

The generality of the present $PhI(OAc)_2/Bu_4NBr$-induced aziridination of *N*-tosylimines with activated methylene compounds was further explored under optimized conditions (Table 3). For the reactions of aromatic imines with diethyl malonate, most of them proceeded well to afford the corresponding aziridine-2,2-dicarboxylates in good yields (entries 1–10). The electron-rich imines **1k** and **1l** were found to be poor substrates for the aziridination. Their reactions were sluggish, and the expected products were not stable enough to be purified, hence the formation of aziridine was detected by ^1H NMR spectroscopy of the crude product (entries 11 and 12). Dimethyl malonate showed the same reactivity as diethyl malonate (entry 13). The reaction of sterically hindered di-*tert*-butyl malonate required a higher temperature and a longer time (entry 14). Moreover, the reactions with ethyl acetoacetate and 2,4-pentanedione also gave the corresponding aziridines in good yields with an extra requirement of lower reaction temperature (entries 15 and 16). No corresponding aziridine was formed when ethyl 4-chloro-3-oxobutanoate, malononitrile, ethyl 2-cyanoacetate, and ethyl 2-nitroacetate were employed, and the corresponding Knoevenagel condensation products were isolated (entries 17–20). When alkanesulfonylimines were used as substrates, only moderate yields were obtained (entries 21–23).

Table 2. Optimization of reaction conditions.

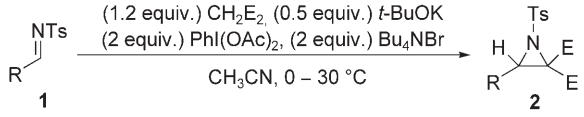


Entry	$PhI(OAc)_2$ [equiv.]	Bu_4NBr [equiv.]	*t*-BuOK [equiv.]	Solvent	**2a** [%][a]
1	1.0	1.0	2	CH_3CN	17
2	2.0	1.0	2	CH_3CN	25
3	3.0	1.0	2	CH_3CN	24
4	2.0	2.0	2	CH_3CN	52
5	2.0	3.0	2	CH_3CN	51
6	2.0	2.0	1	CH_3CN	62
7	2.0	2.0	0.5	CH_3CN	83
8	2.0	2.0	0.25	CH_3CN	73
9	2.0	2.0	0	CH_3CN	0
10	2.0	2.0	0.5	toluene	73
11	2.0	2.0	0.5	$ClCH_2CH_2Cl$	62
12	2.0	2.0	0.5	THF	56
13	2.0	2.0	0.5	EtOAc	38
14	2.0	2.0	0.5	*t*-BuOH	37
15	2.0	2.0	0.5	DMSO	29
16	2.0	2.0	0.5	DMF	25
17	2.0	2.0	0.5	H_2O	0

[a] Isolated yield based on imine **1a**.

A nucleophilic addition product of imine with malonate **3a** was detected during the reaction, and disappeared after the reaction. A reaction pathway was initially hypothesized as shown in Figure 1. After the generation of **3a**, the enolate anion of **3a** attacks $PhI(OAc)_2$ to yield an intermediate **A**. After an intramolecular nucleophilic replacement and a reductive elimination of PhI, an aziridine ring is formed.[12]

Table 3. $PhI(OAc)_2/Bu_4NBr$-induced, one-pot aziridination of *N*-tosylimines with activated methylene compounds.

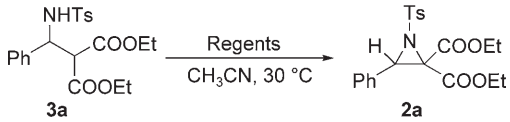


1 2

Entry	R	CH_2E_2	**2** [%][a]
1	Ph	$CH_2(COOEt)_2$	**2a** (83)
2	*p*-$CH_3C_6H_4$	$CH_2(COOEt)_2$	**2b** (77)
	m-$CH_3C_6H_4$	$CH_2(COOEt)_2$	**2c** (78)
4	*p*-ClC_6H_4	$CH_2(COOEt)_2$	**2d** (74)
5	*o*-ClC_6H_4	$CH_2(COOEt)_2$	**2e** (73)
6	*o*-BrC_6H_4	$CH_2(COOEt)_2$	**2f** (78)
7	*p*-CNC_6H_4	$CH_2(COOEt)_2$	**2g** (82)[b]
8	*p*-FC_6H_4	$CH_2(COOEt)_2$	**2h** (68)[b]
9	*m*-$NO_2C_6H_4$	$CH_2(COOEt)_2$	**2i** (89)[b]
10	1-Naphthyl	$CH_2(COOEt)_2$	**2j** (73)
11	*p*-$CH_3OC_6H_4$	$CH_2(COOEt)_2$	**2k**[c]
12	*p*-$(CH_3)_2NC_6H_4$	$CH_2(COOEt)_2$	**2l**[c]
13	Ph	$CH_2(COOCH_3)_2$	**2m** (83)
14	Ph	$CH_2(COOBu\text{-}t)_2$	**2n** (75)[d]
15	Ph	CH_3COCH_2COOEt	**2o** (81)[e,f]
16	Ph	$CH_3COCH_2COCH_3$	**2p** (77)[f]
17	Ph	$ClCH_2COCH_2COOEt$	–[g]
18	Ph	$CH_2(CN)_2$	–[g]
19	Ph	$NCCH_2COOEt$	–[g]
20	Ph	O_2NCH_2COOEt	–[g]
21	*i*-Bu	$CH_2(COOEt)_2$	**2q** (35)
22	*t*-Bu	$CH_2(COOEt)_2$	**2r** (43)
23	*n*-C_7H_{15}	$CH_2(COOEt)_2$	**2s** (37)

[a] Isolated yield.
[b] 0.25 equivalent of *t*-BuOK was used.
[c] The formation of aziridines was detected by the ^1H NMR of the crude product.
[d] The reaction was carried out at 0–40 °C.
[e] The ratio of *trans:cis* is 50:50 as determined by ^1H NMR.
[f] The reaction was carried out at 0–15 °C.
[g] The Knoevenagel condensation product was isolated.

However, the experiments indicated that **3a** could not be converted into aziridine **2a** in the presence of $PhI(OAc)_2$ and *t*-BuOK (Scheme 2). The ring cyclization occurred with the addition of Bu_4NBr, and aziridine **2a** was obtained in 52% yield. More notably, the reaction proceeded better in the absence of *t*-BuOK, with an increased yield of aziridine (88%). We have reported a $PhI(OAc)_2/Bu_4NBr$-induced intramolecular oxidative cyclization of homoallylic sulfonamides.[11b] Acetyl hypobromite (AcOBr) was supposed to be the reaction intermediate. Further investigation showed that aziridine **2a** could be formed but in a lower yield using a mixture of Br_2/AcONa, and no reaction occurred if only Br_2 was used.



Reagents (equiv.)	Yield (%)
$PhI(OAc)_2$ (2), *t*-BuOK (1)	0
$PhI(OAc)_2$ (2), *t*-BuOK (1), Bu_4NBr (2)	52
$PhI(OAc)_2$ (2), Bu_4NBr (2)	88
Br_2 (2)	0
Br_2 (2), AcONa (2)	11
Br_2 (2), AcONa (2), Bu_4NBr (2)	25

Scheme 2.

A tentative mechanism for the aziridination is shown in Figure 2. The reaction might proceed *via* a tandem nucleophilic addition-oxidative cyclization pathway. After the nucleophilic addition of malonate with imine to yield **3a**, the enolate anion of **3a** reacts with AcOBr, which is generated from the ligand exchange between $PhI(OAc)_2$ and Bu_4NBr and their subsequent reductive elimination of PhI, to form an intermediate **B**. Intermediate **B** undergoes an intramolecular nucleophilic attack by the α-amide, and the further elimination of a bromine anion provides the aziridine.

The nucleophilic addition of alkyl *N*-tosylimines with diethyl malonate in the presence of 1 equiv. *t*-BuOK proceeded more sluggishly, and the corresponding addition products **3q**, **3r** and **3s** were isolated in low yields (35-45%). However, **3q**, **3r** and **3s** are good substrates for the ring cyclization in the pres-

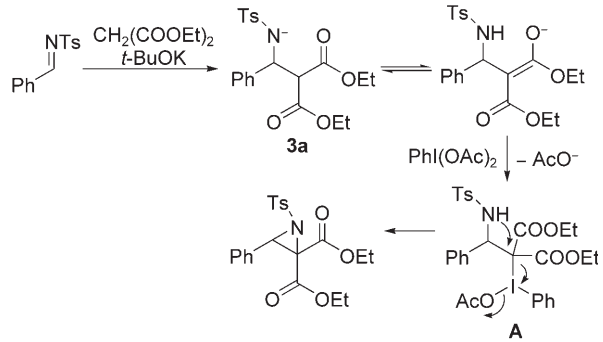


Figure 1.

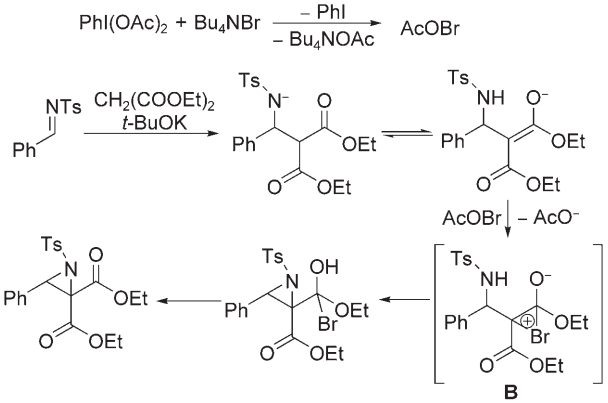
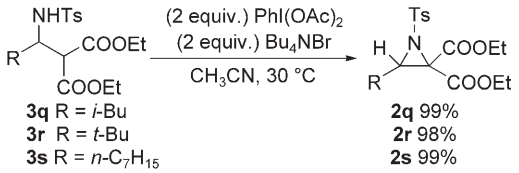


Figure 2.



Scheme 3.

ence of $PhI(OAc)_2$/Bu_4NBr. The corresponding aziridines were obtained in the near quantitative yields (Scheme 3). This indicates that the lower yields obtained in the one-pot aziridination of alkyl *N*-Ts imines were due to their instability and low reactivity in the nucleophilic addition step.

Conclusions

In summary, we have developed an efficient method for the synthesis of 2,2-difunctionalized aziridines by the aziridination of *N*-tosylimines with activated methylene compounds induced by $PhI(OAc)_2$ and Bu_4NBr in the presence of a catalytic amount of base. The potential of this reaction system has been evaluated for its simple procedure, mild reaction conditions, and adaptability to a wide variety of substrates. The further development of the asymmetric reactions is ongoing and will be reported in due course.

Experimental Section

General Experimental Procedure

A solution of imine (0.25 mmol) and CH_2E_2 (0.3 mmol) in anhydrous CH_3CN was cooled to 0 °C, and treated with $PhI(OAc)_2$ (161 mg, 0.5 mmol), Bu_4NBr (161 mg, 0.5 mmol) and *t*-BuOK (14 mg, 0.125 mmol). The resultant mixture was warmed up and stirred at 30 °C. After the imine had disappeared (determined by TLC), the mixture was concentrated, and directly purified by flash column chromatography (10–20% ethyl acetate in hexane) to provide the corresponding aziridine. The characterization data are available in the Supporting Information.

Acknowledgements

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