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An Improved Protocol for Regioselective Ring-Opening of Sulfonyl Aziridines with Iodine Promoted by PPh_3

Jinfeng Zhang, Lingguo Meng,* Chuntao Li, and Guoyuan Xiao

Department of Chemistry, Huaibei Normal University, Huaibei, Anhui 235000, China

A new route to synthesize β-iodo amines from sulfonyl aziridines and iodine was developed in the presence of PPh_3. This ring-opening reaction was an efficient and simple process to give β-iodo amines in excellent yields with high chemoselectivity.

Keywords sulfonyl aziridines, iodine, ring-opening, β-iodo amines, PPh_3

Introduction

Aziridines are one kind of the most important intermediates in organic synthesis and have served as key building blocks in the preparation of various nitrogen-containing molecules.[1] On the other hand, aziridines have covered many interesting reactions, such as ring expansion,[2] cyclization,[3] isomerization,[4] *N*-arylation,[5] *etc*. Apart from above reactions, the ring-opening of aziridines has been also studied intensively and numerous synthetic methods have been developed to realize the opening of aziridine rings.[6] Here, we focused on the preparation of β-iodo amines[7] by using sulfonyl aziridines as starting materials in the presence of PPh_3 and iodine.

To our best knowledge, the usual preparative methods are regioselective ring-opening of aziridines with tetraalkylammonium halides by using the following reagents, such as β-cyclodextrin,[8a] ammonium-12-molybdophosphate[8b] and BF_3•OEt_2,[8c] as catalyst or promoter. On the other hand, the β-iodo amines also could be obtained through ring-opening of aziridines in the presence of $CeCl_3$,[9a] ZnI_2[9b] and ionic liquid.[9c] Except above synthetic methods, Wu also reported thiophenol promoted efficient ring-opening of aziridines with iodine to synthesize β-iodo amines, but thiphenol is one of disagreeable, sick and harmful materials.[10] Herein, we describe an alternative methodology to synthesize β-iodo amines by iodination of aziridines using molecular iodine and PPh_3. It should be noted that better regioselectivity was observed than Wu's methods in some extent.

Results and Discussion

Our studies were initiated by the addition of PPh_3 to a solution of 2-phenyl-1-tosylaziridine (**1a**) and iodine under various reaction conditions, and the results are shown in Table 1. Treatment of **1a** with 0.5 equiv. of iodine and 0.5 equiv. of PPh_3 in CH_2Cl_2 at room temperature in 1 min afforded *N*-(2-iodo-2-phenylethyl)-4-methylbenzenesulfonamide (**2a**) in 96% yield (Table 1, Entry 1). The yield of **2a** could not be further improved by increasing the amount of PPh_3, but decreased sharply when 0.2 equiv. of PPh_3 was added in the reaction (Table 1, Entries 2 and 3). 35% yield of **2a** was obtained when 0.2 equiv. of iodine was used and the yield was failure to further increase when 1.0 equiv. of iodine was used. Solvent screening revealed a significant solvent effect. When shifting the solvent to $CHCl_3$, toluene, dioxane, or THF, the desired product **2a** was obtained in 85%, 81%, 75% or 70% yield, respectively (Table 1, Entries 6—9). With use of CCl_4, CH_3CN or DMF as a solvent, product **2a** was isolated in low yields (Table 1, Entries 10—12). Only a trace amount of the **2a** was detected by TLC when DMSO was used as solvent (Table 1, Entry 13).

With the optimized reaction conditions in hand, a variety of substituted aziridines reacted with iodine smoothly to generate the corresponding ring-opening products in excellent yields with high chemoselectivity and the results were summarized in Table 2. Clearly, aziridines bearing the substitutes on the *para*-positions of benzene rings have no obvious effect on the yields of the reactions. Aziridines with electron-withdrawing groups, such as F, Cl, and Br on the *para*-positions of benzene rings reacted with iodine to give the corresponding products **2b**—**2d** in 95%—97% yields (Table 2, Entries 2—4). However, the reaction became complicated and no desired product was isolated when 2-(4-methoxyphenyl)-1-tosylaziridine was used as substrate, which might be ascribed to the unstable chemical

* E-mail: milig@126.com; Tel.: 0086-0561-3802047
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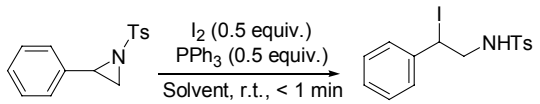
Table 1 Ring-opening of aziridine **1a** with iodine in the presence of PPh_3[a]



1a **2a**

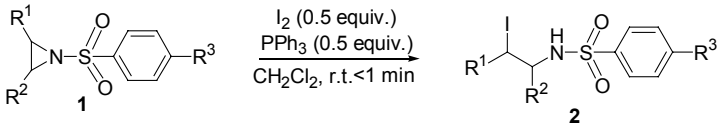
Entry	I_2 (equiv.)	PPh_3 (equiv.)	Solvent	Yield[b]/%
1	1/2	1/2	CH_2Cl_2	96
2	1/2	1/5	CH_2Cl_2	48
3	1/2	1.0	CH_2Cl_2	96
4	1/5	1/2	CH_2Cl_2	35
5	1.0	1/2	CH_2Cl_2	93
6	1/2	1/2	$CHCl_3$	85
7	1/2	1/2	Toluene	81
8	1/2	1/2	Dioxane	75
9	1/2	1/2	THF	70
10	1/2	1/2	CCl_4	35
11	1/2	1/2	CH_3CN	30
12	1/2	1/2	DMF	20
13	1/2	1/2	DMSO	trace

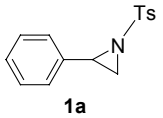
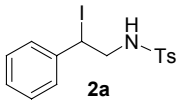
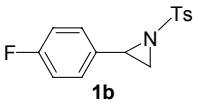
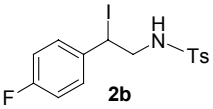
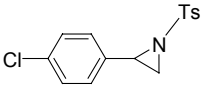
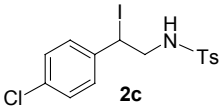
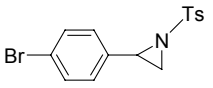
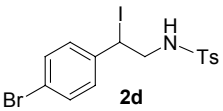
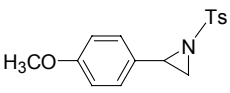
[a] Reaction conditions: **1a** (0.2 mmol), I_2 (amount indicated in Table 1), PPh_3 (amount indicated in Table 1), solvent (2.0 mL), r.t., in air, ＜1 min. [b] Isolated yields.

property of the desired product. The *ortho*-position effect of aziridine was not observed in the reaction of 2-(2-chlorophenyl)-1-tosylaziridine with iodine, which generated the product **2f** in 90% yield. 97% and 94% yields were obtained when 3-chlorophenyl- and 3-bromophenyl-1-tosylaziridine were proceeded in the reaction, respectively. Furthermore, 2-(naphthalen-2-yl)-1-tosylaziridine could be converted to desired products in 96% yield. Aliphatic aziridines, such as 2-hexyl-1-tosylaziridine and 7-tosyl-7-azabicyclo[4.1.0]heptane, also afforded corresponding ring-opening products in excellent yields, and it is important to note that only single ring-opening product was found and better chemoselectivity was observed compared to previous synthetic methods (Table 2, Entry 10).[10] On the other hand, the reactions of 4-bromophenylsulfonyl- and 4-chlorophenylsulfonyl-2-phenylaziridine with iodine also gave corresponding products in 95% and 93% yields, respectively.

To further evaluate the scope of the ring-opening reaction, epoxides were examined under the above optimized reaction conditions. It would be seen from Scheme 1, 7-oxabicyclo[4.1.0]heptane and 2-phenyloxirane reacted with iodine smoothly to generate iodohydrins products in 85% and 95% yields, respectively.

Table 2 PPh_3-mediated ring-opening reaction of aziridines[a]



Entry	Aziridine, **1**	Product, **2**	Yield[b]/%
1	 **1a**	 **2a**	96
2	 **1b**	 **2b**	97
3	 **1c**	 **2c**	96
4	 **1d**	 **2d**	95
5	 **1e**	**2e** was not detected	complicated

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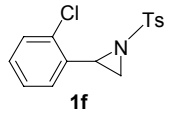
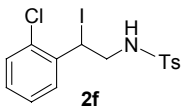
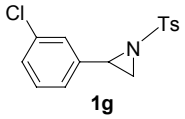
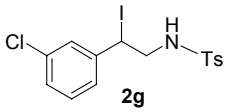
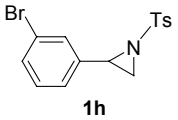
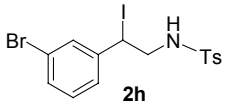
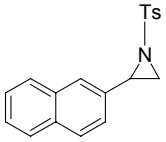
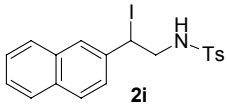
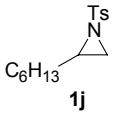
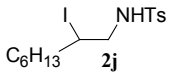
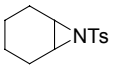
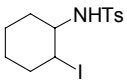
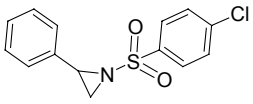
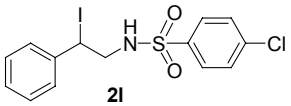
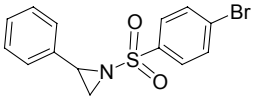
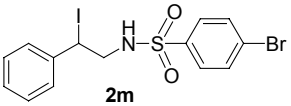
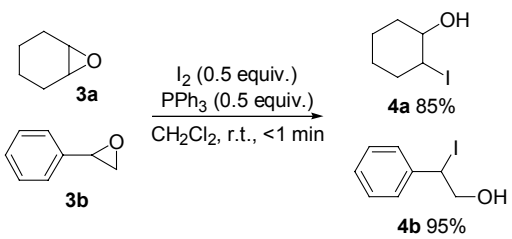
Continued

Entry	Aziridine, **1**	Product, **2**	Yield[b]/%
6	 **1f**	 **2f**	90
7	 **1g**	 **2g**	97
8	 **1h**	 **2h**	94
9	 **1i**	 **2i**	96
10	 **1j**	 **2j**	93
11	 **1k**	 **2k**	91
12	 **1l**	 **2l**	95
13	 **1m**	 **2m**	93

[a] **1a** (0.2 mmol), I_2 (0.1 mmol), PPh_3 (0.1 mmol), CH_2Cl_2 (2.0 mL), r.t., in air, $<$1 min. [b] Isolated yield.

Scheme 1 PPh_3-mediated ring-opening reaction of epoxides



Conclusions

In conclusion, we have described a new route to synthesize β-iodo amines from sulfonyl aziridines and iodine in the presence of PPh_3 and these methods are also suitable for the ring-opening of epoxides. Compared with previous reports, the synthetic method has some merits: (1) easier to manipulate; (2) without any metals; (3) better regioselectivity in some extent than Wu's methods.

Experimental

All reactions were conducted in oven-dried glassware with magnetic stirring. Chromatographic purification was performed on silica gel (100—200 mesh) and analytical thin layer chromatography (TLC) on silica gel 60-F_{254} (Qindao), which was detected by fluorescence. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were measured with a Bruker AC 400 spectrometer with $CDCl_3$ or d_6-DMSO as solvent and recorded relative to internal tetramethylsilane standard. High resolution mass spectra were obtained with a Micromass GCT-

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TOF mass spectrometer. IR spectra were recorded as thin films or as solids in KBr pellets on a Perkin-Elmer FT210 spectrophotometer. Melting points were determined on a digital melting point apparatus and temperatures were uncorrected.

General procedure for the preparation of *β*-iodoamines or iodohydrins

To a solution of aziridines **1** or epoxide **3** (0.20 mmol) and iodine (0.10 mmol) in CH_2Cl_2 (2.0 mL) was added PPh_3 (0.10 mmol), and the reaction mixture was stirred at room temperature in 1 min. Then the mixture was purified directly by flash column chromatography on silica gel to afford the corresponding product.

***N*-(2-Iodo-2-phenylethyl)-4-methylbenzenesulfonamide (2a)**[9a] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.72 (d, J=8.0 Hz, 2H), 7.33 (d, J=8.0 Hz, 2H), 7.26 (s, 5H), 5.04 (t, J=7.6 Hz, 1H), 4.97 (t, J=6.4 Hz, 1H), 3.73−3.66 (m, 1H), 3.55−3.48 (m, 1H), 2.45 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 143.8, 139.9, 137.0, 129.9, 129.0, 128.7, 127.5, 127.0, 51.2, 29.9, 21.6.

***N*-(2-(4-Fluorophenyl)-2-iodoethyl)-4-methylbenzenesulfonamide (2b)** White solid 141−143 ℃; ^{1}H NMR (400 MHz, d_6-DMSO) δ: 8.05 (t, J=5.6 Hz, 1H), 7.65 (d, J=8.4 Hz, 2H), 7.44−7.40 (m, 2H), 7.36 (d, J=8.4 Hz, 2H), 7.13−7.08 (m, 2H), 5.20−5.16 (m, 1H), 3.49−3.36 (m, 2H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 163.1 (d, J=243.8 Hz), 143.2, 138.0, 137.8 (d, J=3.2 Hz), 130.3 (d, J=8.4 Hz), 130.1, 126.8, 115.9 (d, J=21.4 Hz), 51.1, 30.5, 21.4; IR (KBr) v: 3280, 2925 cm^{-1}; HRMS (EI) calcd for $C_{15}H_{15}FNO_2S$ (M^+–I): 292.0808; found 292.0803.

***N*-(2-(4-Chlorophenyl)-2-iodoethyl)-4-methylbenzenesulfonamide (2c)**[9a] ^{1}H NMR (400 MHz, d_6-DMSO) δ: 8.05 (t, J=5.6 Hz, 1H), 7.64 (d, J=8.4 Hz, 2H), 7.40 (d, J=8.4 Hz, 2H), 7.35−7.31 (m, 4H), 5.17−5.13 (m, 1H), 3.47−3.39 (m, 2H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.2, 140.5, 138.0, 132.9, 130.1, 129.0, 126.8, 50.9, 29.9, 21.4.

***N*-(2-(4-Bromophenyl)-2-iodoethyl)-4-methylbenzenesulfonamide (2d)**[8a] ^{1}H NMR (400 MHz, d_6-DMSO) δ: 8.05 (t, J=6.0 Hz, 1H), 7.64 (d, J=8.4 Hz, 2H), 7.47 (d, J=8.4 Hz, 2H), 7.39−7.31 (m, 4H), 5.17−5.13 (m, 1H), 3.50−3.38 (m, 2H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.2, 140.9, 138.0, 131.9, 130.3, 130.1, 126.8, 121.5, 50.8, 29.9, 21.4.

***N*-(2-(2-Chlorophenyl)-2-iodoethyl)-4-methylbenzenesulfonamide (2f)** Colorless oil. ^{1}H NMR (400 MHz, d_6-DMSO) δ: 8.14 (t, J=6.0 Hz, 1H), 7.67 (d, J=8.0 Hz, 2H), 7.60 (dd, J=8.4, 1.2 Hz, 1H), 7.41−7.28 (m, 5H), 5.42−5.38 (m, 1H), 3.55−3.51 (m, 2H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.3, 138.4, 137.9, 132.6, 130.2, 130.1, 129.1, 128.3, 126.8, 49.6, 26.0, 21.4; IR (neat) v: 3285, 2920 cm^{-1}; HRMS (EI) calcd for $C_{15}H_{15}{}^{35}ClNO_2S$ (M–I)$^+$ 308.0512; found 308.0525.

***N*-(2-(3-Chlorophenyl)-2-iodoethyl)-4-methylbenzenesulfonamide (2g)** White solid 124−126 ℃; ^{1}H NMR (400 MHz, d_6-DMSO) δ: 8.09 (t, J=6.0 Hz, 1H), 7.65 (d, J=8.0 Hz, 2H), 7.43 (s, 1H), 7.36−7.16 (m, 5H), 5.16−5.13 (m, 1H), 3.49−3.45 (m, 2H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.8, 143.2, 138.0, 133.5, 130.8, 130.1, 128.4, 128.0, 127.0, 126.8, 50.6, 29.4, 21.4; IR (KBr) v: 3287, 2925 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{16}{}^{35}ClINO_2S$ (M+H)$^+$ 435.9635; found 435.9631.

***N*-(2-(3-Bromophenyl)-2-iodoethyl)-4-methylbenzenesulfonamide (2h)** White solid 122−125 ℃; ^{1}H NMR (400 MHz, d_6-DMSO) δ: 8.09 (t, J=6.0 Hz, 1H), 7.65 (d, J=8.0 Hz, 2H), 7.55 (s, 1H), 7.43−7.34 (m, 4H), 7.24 (d, J=8.0 Hz, 1H), 5.15−5.11 (m, 1H), 3.48−3.45 (m, 2H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 144.1, 143.2, 138.0, 131.3, 131.1, 130.8, 130.1, 127.4, 126.8, 122.0, 50.7, 29.4, 21.4; IR (KBr) v: 3287, 2925 cm^{-1}; HRMS (ESI) calcd for $C_{15}H_{16}{}^{79}BrINO_2S$ (M+H)$^+$ 479.9130; found 479.9126.

***N*-(2-Iodo-2-(naphthalen-2-yl)ethyl)-4-methylbenzenesulfonamide (2i)** White solid 141−142 ℃; ^{1}H NMR (400 MHz, d_6-DMSO) δ: 8.12 (t, J=6.0 Hz, 1H), 7.86−7.84 (m, 4H), 7.66 (d, J=8.0 Hz, 2H), 7.51−7.49 (m, 3H), 7.32 (d, J=8.0 Hz, 2H), 5.38−5.34 (m, 1H), 3.62−3.55 (m, 2H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.2, 138.7, 138.1, 133.0, 130.0, 128.8, 128.3, 127.9, 126.9, 126.9, 126.8, 126.5, 126.3, 50.9, 32.3, 21.4; IR (KBr) v: 3278, 2930 cm^{-1}; HRMS (EI) calcd for $C_{19}H_{18}NO_2S$ (M^+–I) 324.1058; found 324.1054.

***N*-(2-Iodooctyl)-4-methylbenzenesulfonamide (2j)**[11] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.79 (d, J=8.0 Hz, 2H), 7.31 (d, J=8.0 Hz, 2H), 5.00 (d, J=8.0 Hz, 1H), 3.26−3.15 (m, 2H), 2.98−2.94 (m, 2H), 2.42 (s, 3H), 1.49−1.34 (m, 2H), 1.25−1.05 (m, 8H), 0.85 (t, J=7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.5, 137.8, 129.7, 127.0, 52.7, 35.3, 31.5, 28.6, 25.1, 22.4, 21.5, 14.4, 14.0.

***N*-(2-Iodocyclohexyl)-4-methylbenzenesulfonamide (2k)**[9a] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.81 (d, J=8.0 Hz, 2H), 7.32 (d, J=8.0 Hz, 2H), 5.26 (d, J=6.4 Hz, 1H), 4.02−3.97 (m, 1H), 3.31−3.27 (m, 1H), 2.42 (s, 3H), 2.35−2.33 (m, 1H), 2.19−2.16 (m, 1H), 1.99−1.89 (m, 1H), 1.68−1.66 (m, 1H), 1.53−1.50 (m, 1H), 1.41−1.22 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 143.5, 137.4, 129.6, 127.3, 59.1, 34.9, 32.9, 29.6, 26.5, 26.5, 21.5.

4-Chloro-*N*-(2-iodo-2-phenylethyl)benzenesulfonamide (2l) White solid 151−153 ℃; ^{1}H NMR (400 MHz, d_6-DMSO) δ: 8.26 (t, J=6.0 Hz, 1H), 7.78 (d, J=8.8 Hz, 2H), 7.62 (d, J=8.8 Hz, 2H), 7.38 (d, J=7.2 Hz, 2H), 7.30−7.21 (m, 3H), 5.19−5.15 (m, 1H), 3.55−3.40 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.3, 139.8, 137.8, 129.8, 129.0. 128.8, 128.6, 128.1, 50.9, 31.7; IR (KBr) v: 3291, 2923 cm^{-1}; HRMS (EI) calcd for $C_{14}H_{13}{}^{35}ClNO_2S$ (M^+–I) 294.0356; found 294.0354.

4-Bromo-*N*-(2-iodo-2-phenylethyl)benzenesulfonamide (2m) White solid 150−152 ℃; ^{1}H NMR (400

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MHz, d_6-DMSO) δ: 8.26 (t, J=6.0 Hz, 1H), 7.77 (d, J=8.8 Hz, 2H), 7.70 (d, J=8.8 Hz, 2H), 7.38 (d, J=7.2 Hz, 2H), 7.30—7.23 (m, 3H), 5.18—5.14 (m, 1H), 3.55—3.39 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.3, 140.2, 132.7, 129.0, 128.9, 128.6, 128.1, 126.7, 50.9, 31.7; IR (KBr) v: 3290, 2920 cm^{-1}; HRMS (EI) calcd for $C_{14}H_{13}{}^{79}BrNO_2S$ (M^+–I) 337.9850; found 337.9844.

2-Iodocyclohexanol (4a)[10] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 4.06—4.00 (m, 1H), 3.68—3.62 (m, 1H), 2.48—2.42 (m, 2H), 2.12—1.98 (m, 2H), 1.85—1.82 (m, 1H), 1.44—1.20 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 75.8, 43.3, 38.4, 33.5, 27.8, 24.3.

2-Iodo-2-phenylethanol (4b)[10] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 (d, J=7.2 Hz, 2H), 7.32—7.27 (m, 3H), 5.21—5.18 (m, 1H), 4.11—4.06 (m, 1H), 2.21 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 140.0, 128.9, 128.5, 127.9, 68.6, 35.6.

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