# Metal-Free Aziridination of Styrene Derivatives with Iminoiodinane Catalyzed by a Combination of Iodine and Ammonium Iodide

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#### **Supporting Information**

General. New compounds were characterized by <sup>1</sup>H, <sup>13</sup>C, IR, MS, HRMS, and elemental analysis. Melting points were determined on a Stanford Research Systems MPA100 OptiMelt Automated Melting Point System. Infrared spectra were recorded on a SHIMADZU IRAffinity-1 FT-IR Spectrometer. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a JEOL FT-NMR spectrometer (<sup>1</sup>H NMR, 400 MHz; <sup>13</sup>C NMR, 100 MHz) using tetramethylsilane as an internal standard. Mass spectra were obtained on a JEOL JMS-DX303HF mass spectrometer. High-resolution mass spectra were obtained on a JEOL JMS-DX303HF mass spectrometer. All reactions were carried out under nitrogen. Products were purified by chromatography on silica gel BW-300 (Fuji Silysia Chemical Ltd.) or aluminum oxide (Merck, 90 active stage I, 0.063–0.200 mm). Analytical thin-layer chromatography (TLC) was performed on pre-coated silica gel glass plates (Merck silica gel 60 F254, 0.25 mm thickness). Compounds were visualized with UV lamp or treatment with an ethanolic solution of phosphomolybdic acid followed by heating.

**Materials.** Dehydrated acetonitrile was purchased and used as obtained. o-Dichlorobenzene was purchased from commercial sources and used after distillation. Alkenes except for **1g** were purchased from commercial sources. Alkene **1g** was prepared by known method. N-tosyliminophenyliodinane (PhI=NTs) was prepared by known method. All other reagents were commercially available.

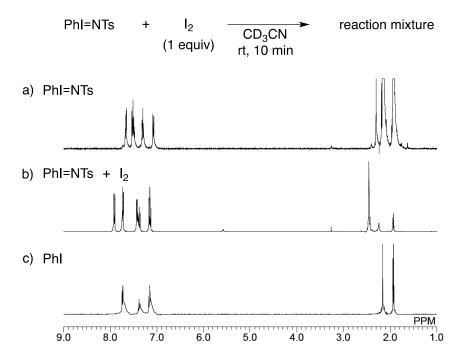
**Typical procedure for the aziridination:** To a solution of Bu<sub>4</sub>NI (0.025 mmol) and I<sub>2</sub> (0.05 mmol) in acetonitrile (2 mL), PhI=NTs (0.5 mmol) and styrenes (0.5 mmol) were added. The reaction mixture was stirred for 3 h at room temperature, and then quenched by  $Na_2S_2O_3$  aq (1 M, 5 mL). The mixture was extracted with diethyl ether (3 x 10 mL). The collected organic layers were dried ( $Na_2SO_4$ ), and evaporation of volatiles gave the

crude product, which was analyzed by <sup>1</sup>H NMR spectroscopy using 1,1,2,2-tetrachloroethane as an internal standard. The crude product was purified by silica gel column chromatography.

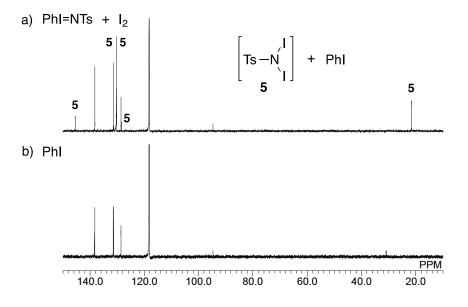
Experimental procedure for the reaction of *trans*-β-methylstyrene (*trans*-1s) with  $I_2/TBAI$  catalyst in acetonitrile: The mixture of  $I_2$  (2.3 mg, 0.0090 mmol), TBAI (1.7 mg, 0.0046 mmol), and *trans*-β-methylstyrene (*trans*-1s) (11.6 mg, 0.098 mmol) was prepared in CD<sub>3</sub>CN (0.5 mL). After mixing at room temperature for 3 h, the mixture was transferred into NMR tube and analyzed by  $^1H$  NMR spectroscopy. No isomerization of starting material (*trans*-1s) was observed.

Experimental procedure for the reaction of  $(2R^*,3R^*)$ -N-(p-toluenesulfonyl)-2-methyl-3-phenylaziridine (trans-2s) with I<sub>2</sub>/TBAI catalyst in acetonitrile: The mixture of I<sub>2</sub> (2.6 mg, 0.010 mmol), TBAI (1.9 mg, 0.0051 mmol), and  $(2R^*,3R^*)$ -N-[(4-methylphenyl)sulfonyl]-2-methyl-3-phenylaziridine (trans-2s)<sup>3</sup> (28 mg, 0.099 mmol) was prepared in CD<sub>3</sub>CN (0.5 mL). After mixing at room temperature for 3 h, the mixture was transferred into NMR tube and analyzed by <sup>1</sup>H NMR spectroscopy. No isomerization of starting material (trans-2s) was observed.

Experimental procedure for NMR study of the reaction of PhI=NTs with I<sub>2</sub>: The mixture of PhI=NTs (37.3 mg, 0.1 mmol) and I<sub>2</sub> (25.4 mg, 0.1 mmol) was prepared in CD<sub>3</sub>CN (0.5 mL). After mixing at room temperature for 10 min, the mixture was transferred into NMR tube. The resulting <sup>1</sup>H and <sup>13</sup>C NMR spectra are shown in Figure S1 and S2, respectively.



**Figure S1.** <sup>1</sup>H NMR spectra in CD<sub>3</sub>CN: a) PhI=NTs. b) The reaction mixture of PhI=NTs and I<sub>2</sub> (1 equiv). c) iodobenzene.



**Figure S2.** <sup>13</sup>C NMR spectra in CD<sub>3</sub>CN: a) The reaction mixture of PhI=NTs and I<sub>2</sub> (1 equiv). b) iodobenzene.

#### **Product data**

#### *N*-(*p*-Toluenesulfonyl)-2-phenylaziridine (2a)



According to the typical procedure,  $Bu_4NI$  (9.3 mg, 0.025 mmol),  $I_2$  (0.0126 g, 0.0496 mmol), PhI=NTs (0.187 g, 0.5 mmol), and styrene (0.0525 g, 0.504 mmol) in acetonitrile gave the product as a white solid (0.128 g, 93% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>3</sup>

#### *N*-(*p*-Toluenesulfonyl)-2-(4-methoxyphenyl)aziridine (2b)

According to the typical procedure, Bu<sub>4</sub>NI (9.2 mg, 0.025 mmol), I<sub>2</sub> (0.0126 g, 0.050 mmol), PhI=NTs (0.187 g, 0.50 mmol), and 4-methoxystyrene (0.0656 g, 0.49 mmol) in acetonitrile gave the product as a white solid (0.076 g, 51% yield). mp: 85.3-86.8; IR: (neat) 1251 (C-O-C), 1319, 1157 (SO<sub>2</sub>) cm<sup>-1</sup>; <sup>1</sup>H NMR: (400 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.86 (d, J = 8.4 Hz, 2H), 7.33 (d, J = 8.4 Hz, 2H), 7.13 (d, J = 8.8 Hz, 2H), 6.82 (d, J = 8.8, 2H), 3.77 (s, 3H), 3.74 (dd, J = 7.2, 4.4 Hz, 1H), 2.96 (d, J = 7.2 Hz, 1H), 2.43 (s, 3H), 2.38, (d, J = 4.4 Hz, 1H); <sup>13</sup>C NMR: (100 MHz, CDCl<sub>3</sub>)  $\delta$  = 159.6, 144.5, 135.0, 129.7, 127.84, 127.76, 126.9, 113.9, 55.2, 40.9, 35.7, 21.6; MS: (CI, 200 eV) m/z 304 (M + 1, 100); HRMS: (CI, 200 eV) calcd for (C<sub>16</sub>H<sub>18</sub>NO<sub>3</sub>S) 304.1007 (M + 1) found m/z 304.1008

#### *N*-(*p*-Toluenesulfonyl)-2-(4-methylphenyl)aziridine (2c)

According to the typical procedure, Bu<sub>4</sub>NI (9.3 mg, 0.025 mmol), I<sub>2</sub> (0.0128 g, 0.050 mmol), PhI=NTs (0.186 g, 0.50 mmol), and 4-methylstyrene (0.0581 g, 0.49 mmol) in acetonitrile gave the product as a white solid (0.127 g, 90% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>3</sup>

S4

#### *N*-(*p*-Toluenesulfonyl)-2-(4-acetoxyphenyl)aziridine (2d)

According to the typical procedure,  $Bu_4NI$  (9.2 mg, 0.025 mmol),  $I_2$  (0.0127 g, 0.050 mmol), PhI=NTs (0.187 g, 0.50 mmol), and 4-acetoxystyrene (0.0833 g, 0.51 mmol) in acetonitrile gave the product as a yellow viscous liquid (0.147 g, 89% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>4</sup>

#### *N*-(*p*-Toluenesulfonyl)-2-(4-chlorophenyl)aziridine (2e)

According to the typical procedure,  $Bu_4NI$  (9.4 mg, 0.025 mmol),  $I_2$  (0.0127 g, 0.050 mmol), PhI=NTs (0.186 g, 0.50 mmol), and 4-chlorostyrene (0.0656 g, 0.47 mmol) in acetonitrile gave the product as a pale yellow solid (0.126 g, 86% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>3</sup>

#### N-(p-Toluenesulfonyl)-2-(4-bromophenyl)aziridine (2f)

According to the typical procedure, Bu<sub>4</sub>NI (9.4 mg, 0.025 mmol), I<sub>2</sub> (0.0127 g, 0.050 mmol), PhI=NTs (0.186 g, 0.50 mmol), and 4-bromostyrene (0.0913 g, 0.50 mmol) in acetonitrile gave the product as a colorless viscous liquid (0.141 g, 75% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>4</sup>

#### *N*-(*p*-Toluenesulfonyl)-2-(4-carbomethoxyphenyl)aziridine (2g)

According to the typical procedure, Bu<sub>4</sub>NI (9.1 mg, 0.025 mmol), I<sub>2</sub> (0.0128 g, 0.050

mmol), PhI=NTs (0.187 g, 0.50 mmol), and 4-carbomethoxystyrene (0.0815 g, 0.50 mmol) in acetonitrile gave the product as a yellow viscous liquid (0.135 g, 81% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>1</sup>

#### *N*-(*p*-Toluenesulfonyl)-2-(4-cyanophenyl)aziridine (2h)

According to the typical procedure, Bu<sub>4</sub>NI (9.2 mg, 0.025 mmol), I<sub>2</sub> (0.0127 g, 0.050 mmol), PhI=NTs (0.186 g, 0.50 mmol), and 4-cyanostyrene (0.0645 g, 0.50 mmol) in acetonitrile gave the product as a white solid (0.119 g, 80% yield). mp: 113.8-114.3; IR: (neat) 2229 (CN), 1317, 1161 (SO<sub>2</sub>) cm<sup>-1</sup>; <sup>1</sup>H NMR: (400 MHz, CDCl<sub>3</sub>)  $\delta$  = 7.86 (d, J = 8.4 Hz, 2H), 7.59 (d, J = 8.4 Hz, 2H), 7.36-7.34 (m, 4H), 3.80 (dd, J = 7.2, 4.4 Hz, 1H), 3.02 (d, J = 7.2 Hz, 1H), 2.45 (s, 3H), 2.36, (d, J = 4.4 Hz, 1H); <sup>13</sup>C NMR: (100 MHz, CDCl<sub>3</sub>)  $\delta$  = 145.0, 140.5, 134.3, 132.3, 129.8, 127.9, 127.2, 118.4, 112.0, 39.8, 36.4, 21.6; MS: (CI, 200 eV) m/z 299 (M + 1, 100); HRMS: (CI, 200 eV) calcd for (C<sub>16</sub>H<sub>15</sub>N<sub>2</sub>O<sub>2</sub>S) 299.0854 (M + 1) found m/z 299.0851

#### N-(p-Toluenesulfonyl)-2-(4-trifluoromethylphenyl)aziridine (2i)

$$F_3C$$

According to the typical procedure,  $Bu_4NI$  (9.3 mg, 0.025 mmol),  $I_2$  (0.0127 g, 0.050 mmol), PhI=NTs (0.187 g, 0.50 mmol), and 4-trifluoromethylstyrene (0.0846 g, 0.49 mmol) in acetonitrile gave the product as a pale yellow solid (0.124 g, 74% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>4</sup>

#### *N*-(*p*-Toluenesulfonyl)-2-(4-nitrophenyl)aziridine (2j)

According to the typical procedure, Bu<sub>4</sub>NI (9.2 mg, 0.025 mmol), I<sub>2</sub> (0.0129 g, 0.051

mmol), PhI=NTs (0.186 g, 0.50 mmol), and 4-nitrostyrene (0.0735 g, 0.49 mmol) in acetonitrile gave the product as a pale yellow solid (0.095 g, 60% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>3</sup>

#### *N*-(*p*-Toluenesulfonyl)-2-(2-methylphenyl)aziridine (2k)

According to the typical procedure,  $Bu_4NI$  (9.3 mg, 0.025 mmol),  $I_2$  (0.0128 g, 0.050 mmol), PhI=NTs (0.187 g, 0.50 mmol), and 2-methylstyrene (0.0588 g, 0.50 mmol) in acetonitrile gave the product as a pale yellow viscous liquid (0.132 g, 92% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>1</sup>

#### N-(p-Toluenesulfonyl)-2-(3-methylphenyl)aziridine (2l)

According to the typical procedure, Bu<sub>4</sub>NI (9.4 mg, 0.025 mmol), I<sub>2</sub> (0.0127 g, 0.050 mmol), PhI=NTs (0.187 g, 0.50 mmol), and 3-methylstyrene (0.0575 g, 0.49 mmol) in acetonitrile gave the product as a pale yellow viscous liquid (0.118 g, 85% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>4</sup>

#### *N*-(*p*-Toluenesulfonyl)-2-methyl-2-phenylaziridine (2m)

According to the typical procedure, Bu<sub>4</sub>NI (9.3 mg, 0.025 mmol),  $I_2$  (0.0126 g, 0.050 mmol), PhI=NTs (0.187 g, 0.50 mmol), and  $\alpha$ -methylstyrene (0.0574 g, 0.49 mmol) in acetonitrile gave the product as a white solid (0.081 g, 58% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>5</sup>

#### N-(p-Toluenesulfonyl)-1,1a,6.6a-tetrahydroindeno[1,2-b]aziridine (2n)

According to the typical procedure,  $Bu_4NI$  (18.2 mg, 0.049 mmol),  $I_2$  (0.0254 g, 0.10 mmol), PhI=NTs (0.187 g, 0.50 mmol), and indene (0.0569 g, 0.49 mmol) in acetonitrile gave the product as a pale yellow solid (0.090 g, 64% yield). The analytical data for this compound were in excellent agreement with the reported data.

### N-(p-Toluenesulfonyl)-1a,2,3,7b-tetrahydro-1H-naphtho[1,2-b]aziridine (20)

According to the typical procedure,  $Bu_4NI$  (18.6 mg, 0.050 mmol),  $I_2$  (0.0254 g, 0.10 mmol), PhI=NTs (0.187 g, 0.50 mmol), and 1,2-dihydronaphthalene (0.0630 g, 0.48 mmol) in acetonitrile gave the product as a pale yellow solid (0.066 g, 46% yield). The analytical data for this compound were in excellent agreement with the reported data.<sup>7</sup>

#### Cis- or trans-N-(p-toluenesulfonyl)-2-methyl-3-phenylaziridine (2p)

**Reaction of trans-β-methylstyrene:** According to the typical procedure, Bu<sub>4</sub>NI (9.3 mg, 0.025 mmol), I<sub>2</sub> (0.0127 g, 0.050 mmol), PhI=NTs (0.186 g, 0.50 mmol), and *trans*-β-methylstyrene (0.0601 g, 0.51 mmol) in acetonitrile gave the crude product in a *cis/trans* ratio of 39/61 determined by <sup>1</sup>H NMR spectroscopy. Purification by silica gel column chromatography gave the product as a colorless viscous liquid (0.128 g, 89% yield (combined yield of *trans* and *cis* isomers)). The analytical data for this compound were in excellent agreement with the reported data.<sup>3</sup>

**Reaction of cis-β-methylstyrene:** According to the typical procedure, Bu<sub>4</sub>NI (9.4 mg, 0.025 mmol), I<sub>2</sub> (0.0126 g, 0.050 mmol), PhI=NTs (0.187 g, 0.50 mmol), and *cis*-β-methylstyrene (0.0601 g, 0.51 mmol) in acetonitrile gave the crude product in a *cis/trans* ratio of 36/64 determined by  $^{1}$ H NMR spectroscopy. Purification by silica gel column chromatography gave the product as a colorless viscous liquid (0.129 g, 92% yield (combined yield of *trans* and *cis* isomers)).

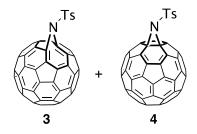
#### Cis- or trans-N-(p-toluenesulfonyl)-2,3-diphenylaziridine (2q)



**Reaction of trans-stilbene:** According to the typical procedure, Bu<sub>4</sub>NI (9.4 mg, 0.026 mmol), I<sub>2</sub> (0.0127 g, 0.050 mmol), PhI=NTs (0.187 g, 0.50 mmol), and *trans*-stilbene (0.0903 g, 0.50 mmol) in acetonitrile gave the crude product in a *cis/trans* ratio of 90/10 determined by <sup>1</sup>H NMR spectroscopy. Purification by silica gel column chromatography gave the product as a white solid (0.137 g, 78% yield (combined yield of *trans* and *cis* isomers)). The analytical data for this compound were in excellent agreement with the reported data.<sup>3</sup>

**Reaction of cis-stilbene:** According to the typical procedure, Bu<sub>4</sub>NI (9.3 mg, 0.025 mmol), I<sub>2</sub> (0.0128 g, 0.050 mmol), PhI=NTs (0.187 g, 0.50 mmol), and *cis*-stilbene (0.0905 g, 0.50 mmol) in acetonitrile gave the crude product in a *cis/trans* ratio of 92/8 determined by <sup>1</sup>H NMR spectroscopy. Purification by silica gel column chromatography gave the product as a white solid (0.117 g, 67% yield (combined yield of *trans* and *cis* isomers)).

# N-(p-Toluenesulfonyl)azafulleroid (3) and N-(p-Toluenesulfonyl)aziridinofullerene (4)



**Reaction with TBAI:** To a solution of Bu<sub>4</sub>NI (3.8 mg, 0.010 mmol), I<sub>2</sub> (5.2 mg, 0.021 mmol), and fullerene (0.072 g, 0.10 mmol) in o-dichlorobenzene (10 mL), PhI=NTs (0.037 g, 0.10 mmol) was added. The reaction mixture was stirred for 1 h at room temperature, and then the mixture was passed through a short column on a silica gel pad (10 g), and the solvent was evaporated under reduced pressure. The residue was purified by silica gel column chromatography to give the product as a brown solid (0.033 g, 37% yield (combined yield of **3** and **4**)), and unreacted starting fullerene was recovered (0.033 g, 46%). The ratio of **3** and **4** was analyzed by  $^{1}$ H NMR spectroscopy (3/4 =

84/16). The analytical data for this compound were in excellent agreement with the reported data.<sup>8</sup>

**Reaction without TBAI:** According to the procedure described above,  $I_2$  (5.0 mg, 0.020 mmol), fullerene (0.072 g, 0.10 mmol), PhI=NTs (0.037 g, 0.10 mmol) in o-dichlorobenzene gave the product as a brown solid (0.035 g, 40% yield (combined yield of 3 and 4)), and unreacted starting fullerene was recovered (0.032 g, 45%). The ratio of 3 and 4 was analyzed by  $^1$ H NMR spectroscopy (3/4 = 85/15).

#### References

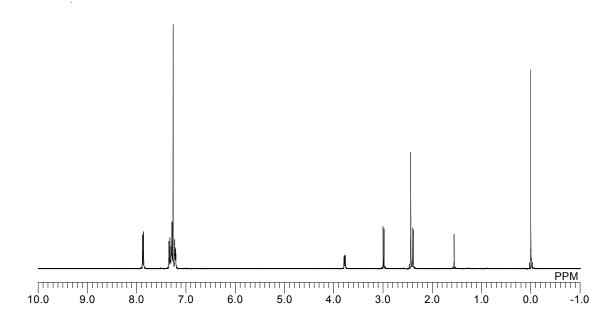
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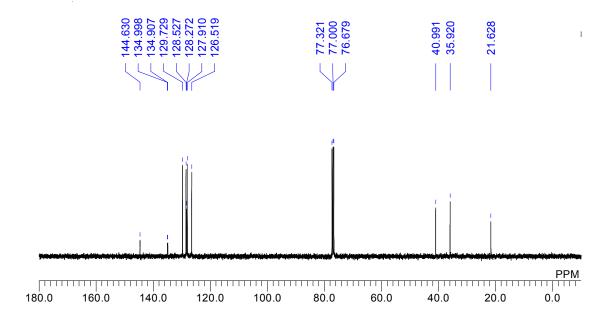
### NMR spectra

### *N*-(*p*-Toluenesulfonyl)-2-phenylaziridine (2a)



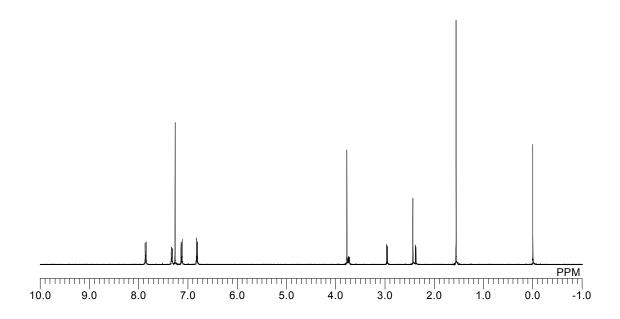
<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

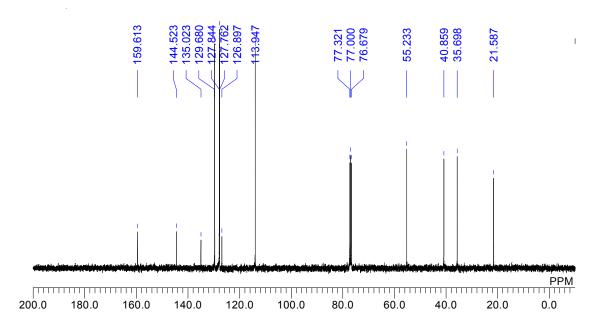




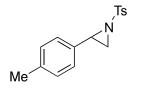
## N-(p-Toluenesulfonyl)-2-(4-methoxyphenyl)aziridine (2b)

<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

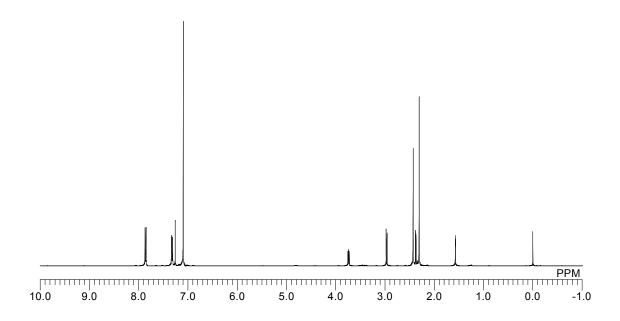


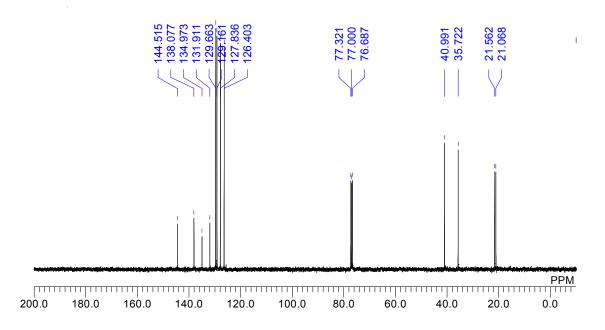


## N-(p-Toluenesulfonyl)-2-(4-methylphenyl)aziridine (2c)



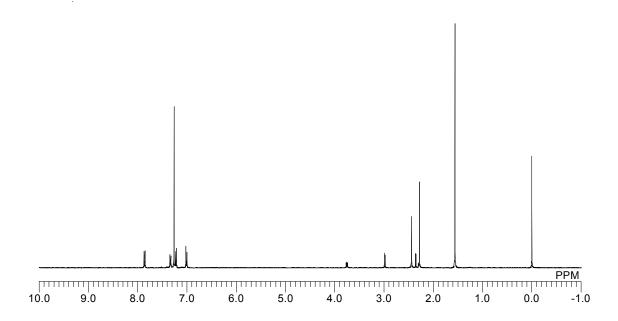
<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

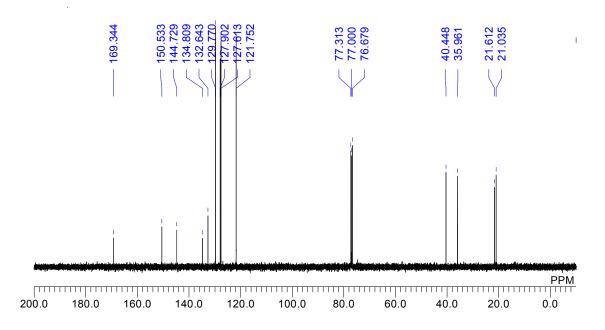




## N-(p-Toluenesulfonyl)-2-(4-acetoxyphenyl)aziridine (2d)

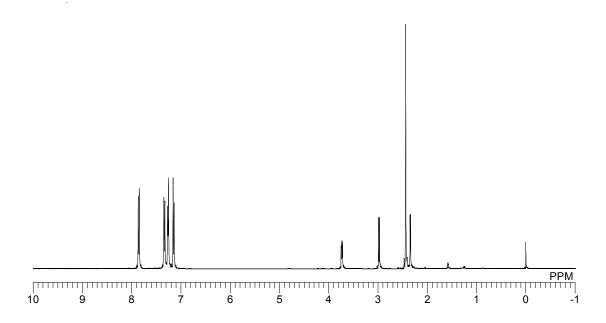
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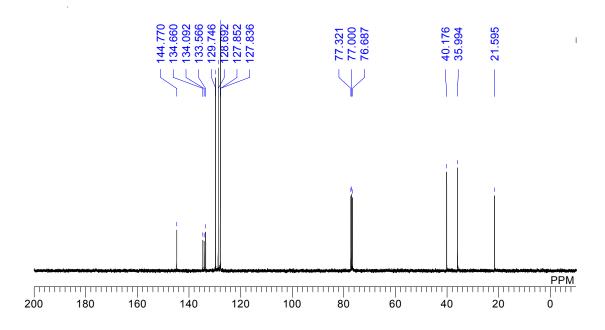




## N-(p-Toluenesulfonyl)-2-(4-chlorophenyl)aziridine (2e)

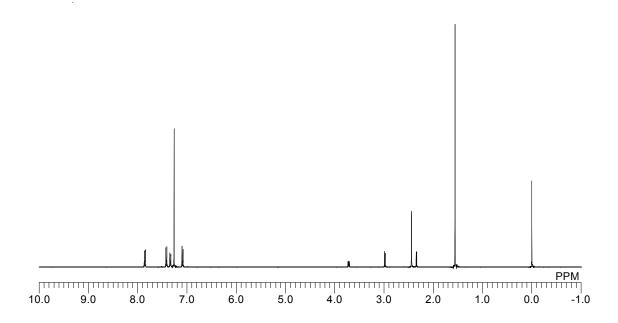
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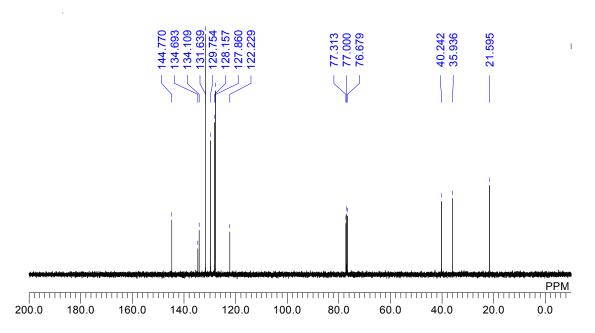




## N-(p-Toluenesulfonyl)-2-(4-bromophenyl)aziridine (2f)

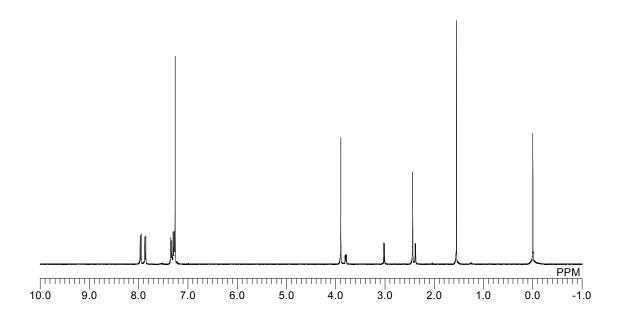
<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

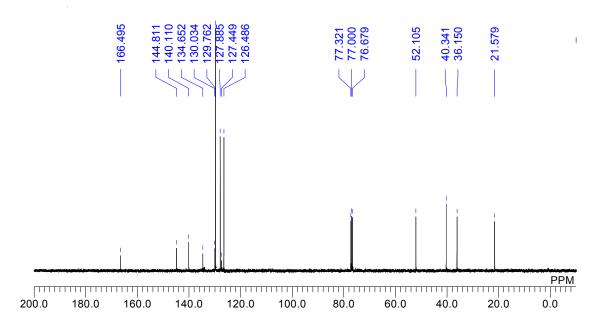




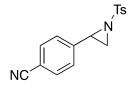
## N-(p-Toluenesulfonyl)-2-(4-carbomethoxyphenyl)aziridine (2g)

<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

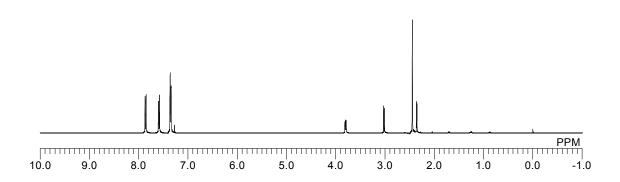


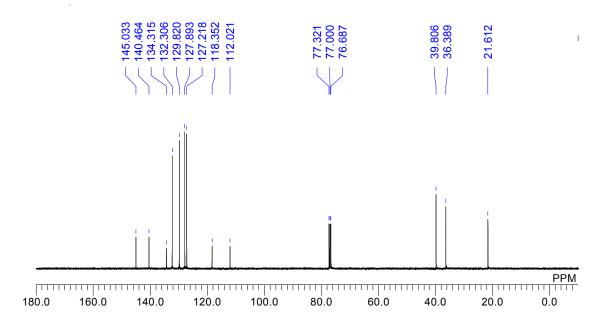


## N-(p-Toluenesulfonyl)-2-(4-cyanophenyl)aziridine (2h)



<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

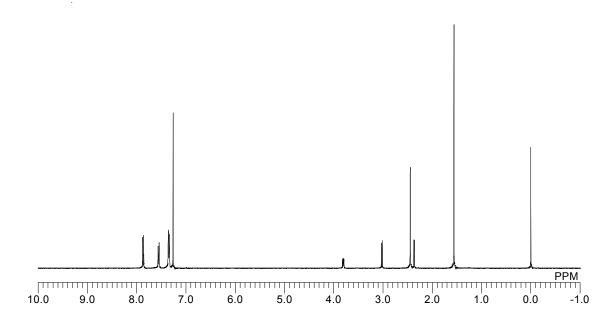


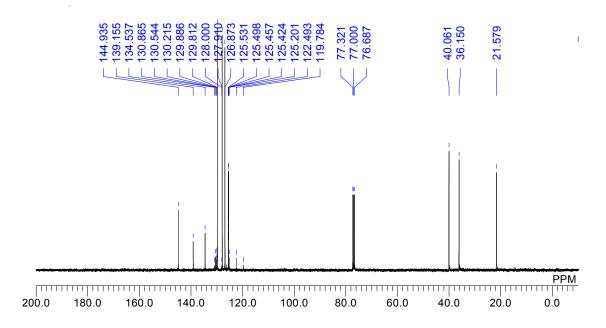


## N-(p-Toluenesulfonyl)-2-(4-trifluoromethylphenyl)aziridine (2i)

$$F_3C$$

<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

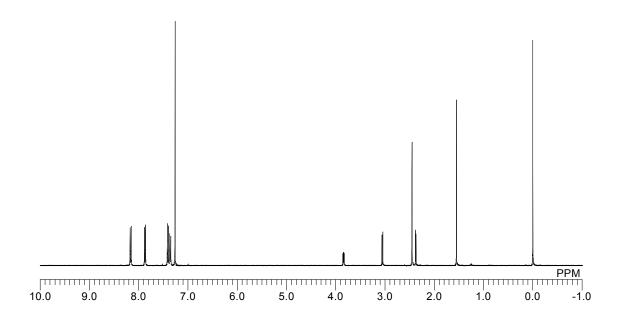


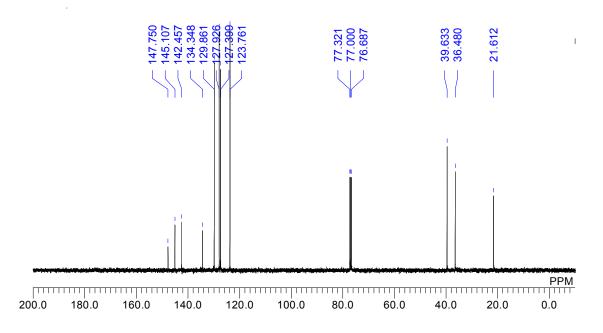


## N-(p-Toluenesulfonyl)-2-(4-nitrophenyl)aziridine (2j)

$$O_2N$$

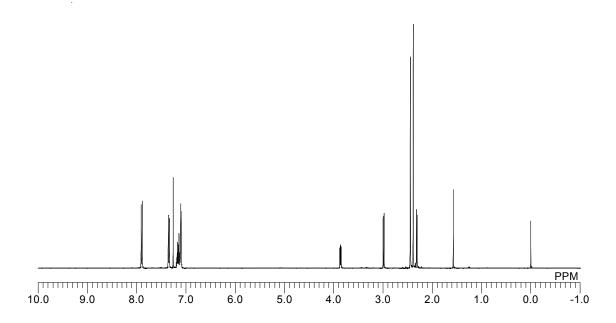
<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

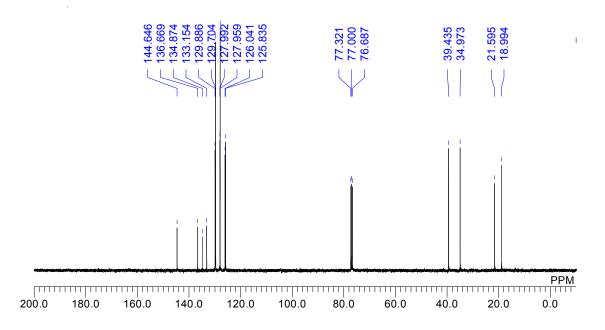




## N-(p-Toluenesulfonyl)-2-(2-methylphenyl)aziridine (2k)

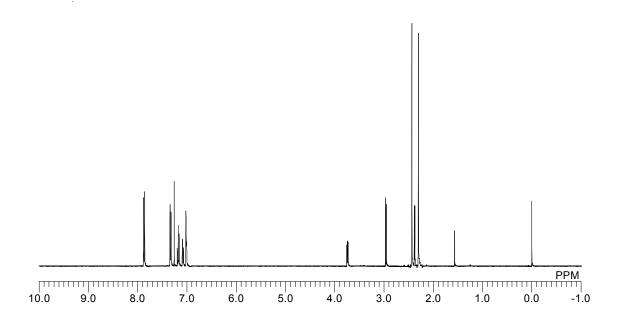
<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

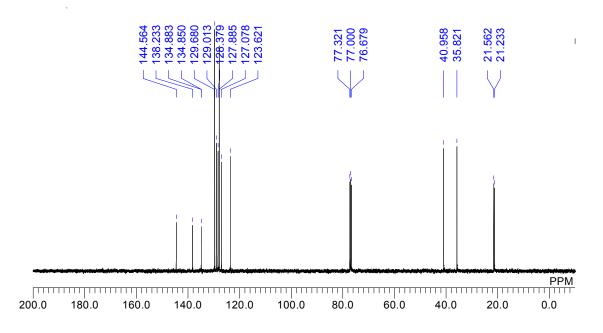




## N-(p-Toluenesulfonyl)-2-(3-methylphenyl)aziridine (2l)

<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

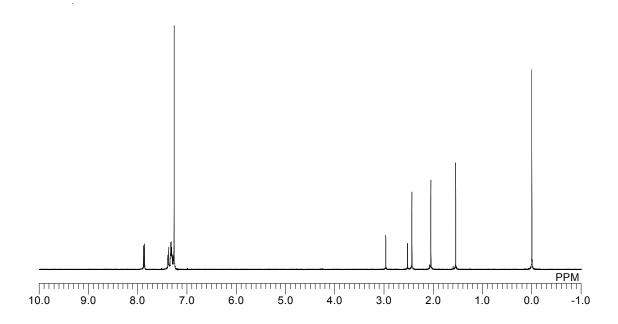


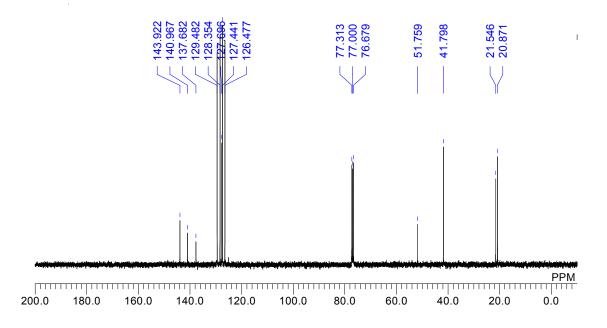


## N-(p-Toluenesulfonyl)-2-methyl-2-phenylaziridine (2m)



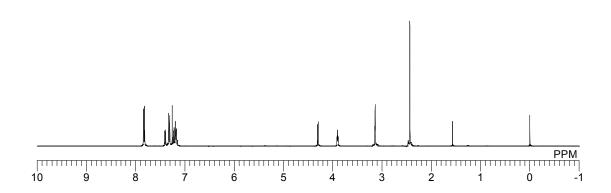
<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

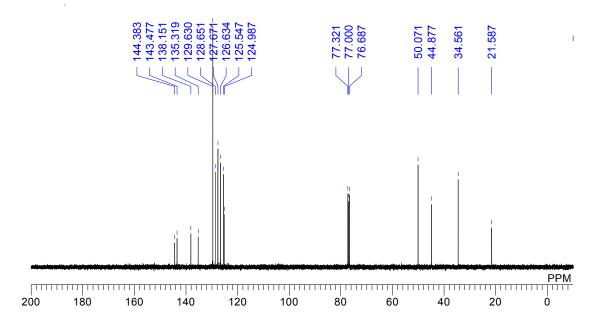




## $N\hbox{-}(p\hbox{-} {\bf Toluene sulfonyl})\hbox{-} {\bf 1,1} \hbox{a,6.6} \hbox{a-tetrahydroindeno} \hbox{[1,2-$b$]} \hbox{aziridine (2n)}$

<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

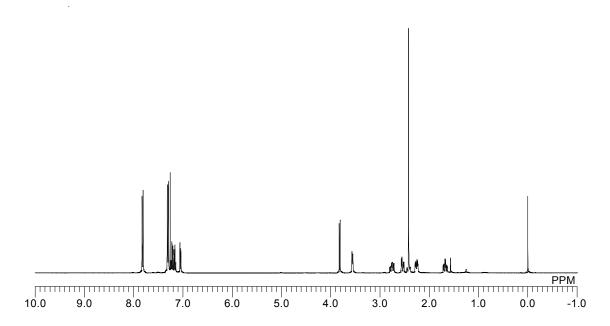


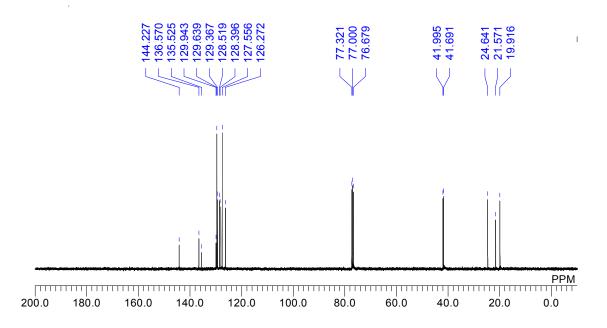


## N-(p-Toluenesulfonyl)-1a,2,3,7b-tetrahydro-1H-naphtho[1,2-b]aziridine (2o)



<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

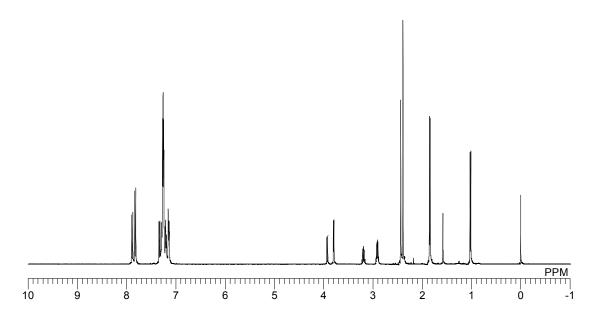


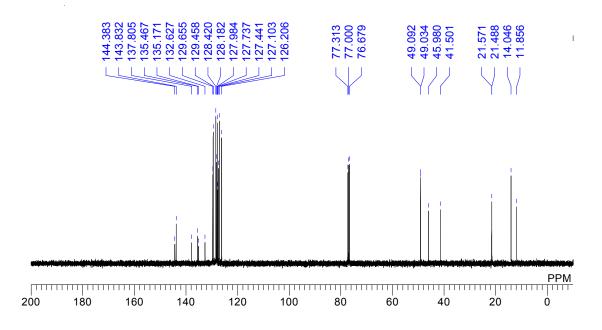


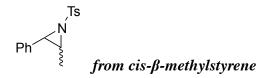
## Cis- or trans-N-(p-toluenesulfonyl)-2-methyl-3-phenylaziridine (2p)



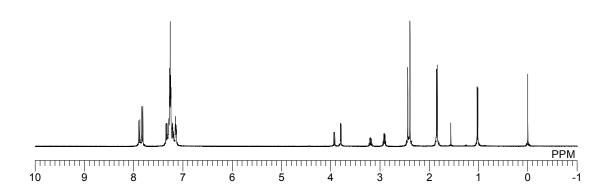
<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

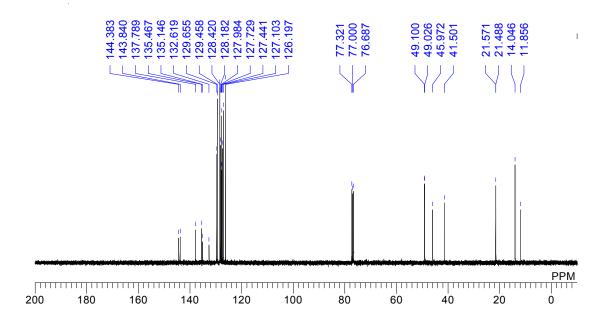






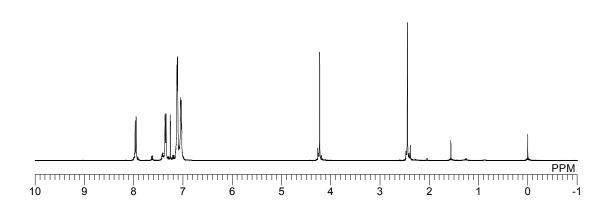
<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

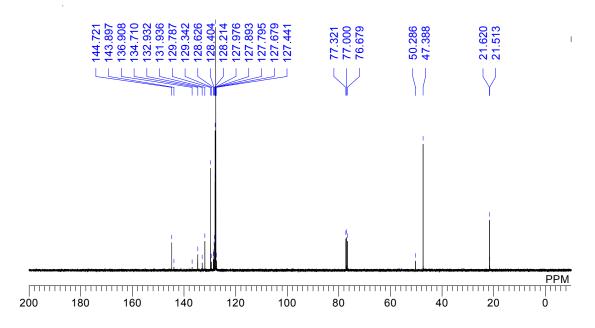


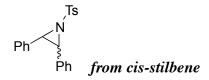


## Cis- or rans-N-(p-toluenesulfonyl)-2,3-diphenylaziridine (2q)

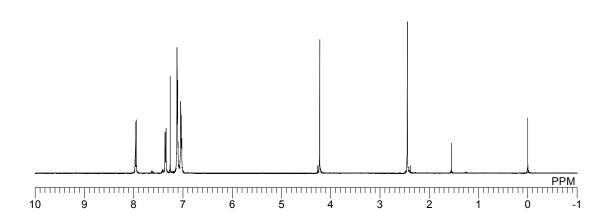
<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)

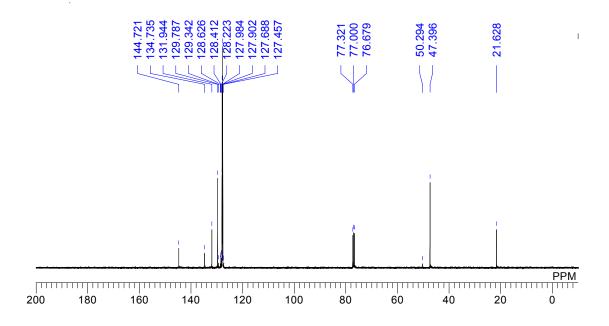






<sup>1</sup>H NMR:(400 MHz, CDCl<sub>3</sub>)





# N-(p-Toluenesulfonyl)azafulleroid (3) and N-(p-Toluenesulfonyl)aziridinofullerene (4)

