Synthesis of Highly Substituted 2-Imidazolines through a Three-Component Coupling Reaction

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Received 7 September 2007; revised 5 October 2007
Advanced online publication: 07.12.2007

Abstract: A simple strategy for the synthesis of highly substituted 2-imidazolines starting from terminal alkynes, sulfonyl azides, and N-unsubstituted aziridines via two steps with high regioselectivity is described.

Key words: 2-imidazolines, sulfonyl azides, N-unsubstituted aziridines, three-component coupling, isomerization

2-Imidazolines have fascinated organic chemists worldwide for a longtime, not only because they possess good biological and pharmacological activities but also because these heterocycles are useful synthetic intermediates and function as chiral auxiliaries, chiral catalysts, and ligands for asymmetric catalysis. However, only a few examples for the synthesis of highly substituted 2-imidazolines have been reported. Some of these methods involve disadvantages such as the difficult availability of precursors and the use of complex reagents. For these reasons, simple and atom economical synthesis of highly substituted imidazolines, which could be manipulated to include a variety of substituent groups from easily available precursors, would be of interest. Herein we describe the preparation of several N-unsubstituted aziridines and their conversion into 2-imidazolines.

Aziridines are versatile building blocks for the syntheses of many nitrogen-containing biologically active molecules. Due to their significant position in synthetic organic chemistry, diverse synthetic methodologies have been reported for the preparation of aziridines. They react with various nucleophiles to yield regioselective ring-opened products. Isomerization of appropriate aziridine derivatives provides useful synthetic pathways leading to heterocyclic systems. N-Unsubstituted aziridines show behavior characteristic of secondary amines, caused by the nucleophilicity of the nitrogen atom. Recently, Chang et al. reported several copper-catalyzed multicomponent reactions of sulfonyl azides, terminal alkynes, and various nucleophiles such as amines, water, alcohols, imines, and salicylaldehydes. Inspired by their findings, we have prepared functionalized aziridines 2 by a three-component reaction between terminal alkynes, sulfonyl azides, and N-unsubstituted aziridines in the presence of CuI and base under very mild conditions. The aziridines 2 can undergo isomerization leading to highly substituted 2-imidazolines 3 in the presence of sodium iodide in acetone. The synthetic route is depicted in Scheme 1.

Our investigations began with the reaction of p-toluenesulfonyl azide with phenylacetylene and trans-phenyl(3-phenylaziridin-2-yl)methanone catalyzed by CuI in CHCl₃. Some other copper catalysts were also investigated in this reaction. CuI was found to be superior to other copper sources examined including CuBr, CuCl, and Cu(PPh₃)₃Br. Successful formation of substituted aziridine was observed in chloroform when 10 mol% CuI and a slight excess of triethylamine (1.2 equiv) were added at room temperature. The use of other bases such as pyridine, K₂CO₃, and (t-Pr)₂NET resulted in reduced yields. The use of THF or CH₂Cl₂ as a solvent can shorten the reaction time, but some by-products were formed. However, no conversion was observed in the absence of Cu catalysts or bases.

The generality of the functionalized aziridine synthesis was next investigated with a range of terminal alkynes, p-toluenesulfonyl azide, and trans-N-unsubstituted aziridines under the optimized reaction conditions (Table 1). Due to the steric hindrance of N-unsubstituted aziridines long reaction times (24–36 h) were required. Electronic variation in the N-unsubstituted aziridine derivatives altered greatly the efficiency of the reaction (Table 1, entries 1–10). When Ar¹ is 2-ClC₆H₄ or 2-BrC₆H₄, a higher yield of the product was obtained (Table 1, entries 3, 5).

Not only aromatic acetylenes but also aliphatic alkynes can react smoothly under these conditions to afford the corresponding aziridines in good yields (Table 1, entries...
Additionally, when electron-deficient aziridines or substituted terminal alkynes were employed, no product was detected (Table 1, entries 14–16). The structure and relative stereochemistry of \(2b\) were established by X-ray crystallography (Figure 1)\(^{13}\) and the trans relationship between \(\text{Ar}_1\) and \(\text{COAr}_2\) subunits was confirmed.

Next, we investigated the isomerization of trans-substituted aziridine \(2\) to 2-imidazoline catalyzed by sodium iodide (Table 2). The isomerization may take place by the addition of the iodide to the benzimidoyl carbon to give the intermediate \(\text{I}\), which subsequently cyclizes to the 2-imidazoline.\(^{9a,c}\) A 1:3 mixture of cis-\(3\) and trans-\(3\) was obtained when \(2\) was treated with NaI in refluxing acetone for two hours. Some of the trans-imidazolines trans-\(3\) were isomerized into cis-\(3\) in refluxing acetone until the thermodynamics equilibrium is reached.\(^{2b}\) It is interesting that the formation of only trans-\(3\) was observed when \(2\) was treated with NaI in acetone at room temperature for 6–8 hours. Based on this observation, various trans-substituted aziridines were efficiently isomerized to the corresponding trans-2-imidazolines in good yields at room temperature. X-ray crystal structure data of \(3\) (Figure 2)\(^{14}\) unambiguously proved the trans relationship between \(\text{Ar}_1\) and \(\text{COAr}_2\) subunits.

During our investigations, we were pleased to find that 2-imidazoline can be obtained directly via multicomponent reaction of \(p\)-toluenesulfonyl azide, phenylacetylene, and N-unsubstituted aziridine. When \(\text{Ar}_1\) is 4-MeC\(_6\)H\(_4\), reaction with either CuI or CuCl results in good yield of the product (Scheme 2).

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**Table 1  Aziridines 2 Prepared\(^a\)**

<table>
<thead>
<tr>
<th>Entry</th>
<th>(\text{Ar}_1)</th>
<th>(\text{Ar}_2)</th>
<th>R</th>
<th>2</th>
<th>Yield (%)(^b)</th>
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<tbody>
<tr>
<td>1</td>
<td>Ph</td>
<td>Ph</td>
<td>Ph</td>
<td>(2a)</td>
<td>81</td>
</tr>
<tr>
<td>2</td>
<td>4-ClC(_6)H(_4)</td>
<td>Ph</td>
<td>Ph</td>
<td>(2b)</td>
<td>57</td>
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<tr>
<td>3</td>
<td>2-ClC(_6)H(_4)</td>
<td>Ph</td>
<td>Ph</td>
<td>(2c)</td>
<td>91</td>
</tr>
<tr>
<td>4</td>
<td>4-BrC(_6)H(_4)</td>
<td>Ph</td>
<td>Ph</td>
<td>(2d)</td>
<td>46</td>
</tr>
<tr>
<td>5</td>
<td>2-BrC(_6)H(_4)</td>
<td>Ph</td>
<td>Ph</td>
<td>(2e)</td>
<td>88</td>
</tr>
<tr>
<td>6</td>
<td>3-BrC(_6)H(_4)</td>
<td>Ph</td>
<td>Ph</td>
<td>(2f)</td>
<td>54</td>
</tr>
<tr>
<td>7</td>
<td>Ph</td>
<td>4-BrC(_6)H(_4)</td>
<td>Ph</td>
<td>(2g)</td>
<td>38</td>
</tr>
<tr>
<td>8</td>
<td>Ph</td>
<td>4-FC(_6)H(_4)</td>
<td>Ph</td>
<td>(2h)</td>
<td>53</td>
</tr>
<tr>
<td>9</td>
<td>Ph</td>
<td>3-BrC(_6)H(_4)</td>
<td>Ph</td>
<td>(2i)</td>
<td>71</td>
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<tr>
<td>10</td>
<td>Ph</td>
<td>4-MeOC(_6)H(_4)</td>
<td>Ph</td>
<td>(2j)</td>
<td>58</td>
</tr>
<tr>
<td>11</td>
<td>Ph</td>
<td>Ph</td>
<td>C(_6)H(_4)</td>
<td>(2k)</td>
<td>77</td>
</tr>
<tr>
<td>12</td>
<td>2-ClC(_6)H(_4)</td>
<td>Ph</td>
<td>C(_6)H(_4)</td>
<td>(2l)</td>
<td>69</td>
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<tr>
<td>13</td>
<td>2-BrC(_6)H(_4)</td>
<td>Ph</td>
<td>C(_6)H(_4)</td>
<td>(2m)</td>
<td>65</td>
</tr>
<tr>
<td>14</td>
<td>4-NO(_2)C(_6)H(_4)</td>
<td>Ph</td>
<td>Ph</td>
<td>(2n)</td>
<td>NR</td>
</tr>
<tr>
<td>15</td>
<td>Ph</td>
<td>Ph</td>
<td>4-BrC(_6)H(_4)</td>
<td>(2o)</td>
<td>NR</td>
</tr>
<tr>
<td>16</td>
<td>Ph</td>
<td>Ph</td>
<td>4-NO(_2)C(_6)H(_4)</td>
<td>(2p)</td>
<td>NR</td>
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\(^a\) The reactions were carried out with N-unsubstituted aziridines (0.5 equiv), terminal alkynes (0.6 equiv), TsN\(_3\) (0.6 equiv), Et\(_3\)N (0.6 equiv), and CuI (10 mol%) in CHCl\(_3\) at r.t. for 24–36 h.

\(^b\) Isolated yields; NR = no reaction.
In summary, we have reported here a two-step sequence for the synthesis of 2-imidazolines via CuI catalyzed multicomponent reactions. The process has several advantages including, (1) simple and easily available starting materials, (2) special, highly substituted product structure and providing a wide range of aryl and alkyl 2-imidazolines, and (3) high regioselectivity and mild reaction conditions. Our further studies will focus on the development of related transformations and the application of this method to the preparation of chiral 2-imidazolines containing natural products.

All reagents were used directly as obtained commercially unless otherwise noted. Melting points were determined on a microscopic apparatus and are uncorrected. Column chromatography was carried out on silica gel. 1H NMR spectra were recorded at 300 MHz in CDCl₃ and 13C NMR spectra were recorded at 75 MHz in CDCl₃ using TMS as internal standard. A Nicolet AVATAR 360 FT-IR spectrometer was used for IR spectra.

**trans-**\(N^1\)-\(\{1-(2\)-benzoyl-3-phenylaziridin-1-yl\}-2-phenylethylidene\)-4-methylbenzenesulfonamide (2a); Typical Procedure

To a stirred mixture of \(p\)-toluenesulfonyl azide (118.3 mg, 0.6 mmol), phenylacetylene (61.2 mg, 0.6 mmol), and CuI (9.5 mg, 0.05 mmol) in CHCl₃ (5 mL) was added the aziridine nucleophile (0.5 mmol) at r.t. Et₃N (0.6 mmol) was added prior to the addition of nucleophiles. After completion of the reaction, which was monitored by TLC, the mixture was diluted with CH₂Cl₂ (3 mL) and aq NH₄Cl (3 mL). The mixture was stirred for an additional 30 min and the two layers were separated. The aqueous layer was extracted with CH₂Cl₂ (3 × 3 mL). The combined organic layers were dried (MgSO₄), filtered, and concentrated in vacuo. The crude residue was purified by flash column chromatography to give the desired product 2a; yield: 200 mg (81%); white solid; mp 116–118 °C (EtOAc–hexane) (Table 1).

**Isomerization of Aziridines 2 Catalyzed by NaI**

<table>
<thead>
<tr>
<th>Entry</th>
<th>Aziridine 2</th>
<th>Product</th>
<th>Yield (%)b</th>
</tr>
</thead>
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<tr>
<td>1</td>
<td>2a</td>
<td>3a</td>
<td>75</td>
</tr>
<tr>
<td>2</td>
<td>2b</td>
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<tr>
<td>3</td>
<td>2c</td>
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<td>88</td>
</tr>
<tr>
<td>4</td>
<td>2d</td>
<td>3d</td>
<td>70</td>
</tr>
<tr>
<td>5</td>
<td>2e</td>
<td>3e</td>
<td>68</td>
</tr>
<tr>
<td>6</td>
<td>2f</td>
<td>3f</td>
<td>37</td>
</tr>
<tr>
<td>7</td>
<td>2g</td>
<td>3g</td>
<td>77</td>
</tr>
<tr>
<td>8</td>
<td>2h</td>
<td>3h</td>
<td>47</td>
</tr>
<tr>
<td>9</td>
<td>2i</td>
<td>3i</td>
<td>50</td>
</tr>
<tr>
<td>10</td>
<td>2j</td>
<td>3j</td>
<td>68</td>
</tr>
<tr>
<td>11</td>
<td>2k</td>
<td>3k</td>
<td>48</td>
</tr>
<tr>
<td>12</td>
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<td>3l</td>
<td>60</td>
</tr>
<tr>
<td>13</td>
<td>2m</td>
<td>3m</td>
<td>76</td>
</tr>
</tbody>
</table>

* The reactions were carried out with substituted aziridine 2 (0.25 equiv) and NaI (1.0 equiv) in acetone at r.t. for 6–8 h.

a Isolated yields.

**trans-**\(N^1\)-\(\{1-(2\)-benzoyl-3-(4-chlorophenyl)aziridin-1-yl\}-2-phenylethylidene\)-4-methylbenzenesulfonamide (2b) White solid; mp 122–123 °C (EtOAc–hexane) (Table 2).

**Table 2** Isomerization of Aziridines 2 Catalyzed by NaI

<table>
<thead>
<tr>
<th>Entry</th>
<th>Aziridine 2</th>
<th>Product</th>
<th>Yield (%)b</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2a</td>
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<tr>
<td>8</td>
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<tr>
<td>9</td>
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<tr>
<td>10</td>
<td>2j</td>
<td>3j</td>
<td>68</td>
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<tr>
<td>11</td>
<td>2k</td>
<td>3k</td>
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</tr>
<tr>
<td>12</td>
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<td>60</td>
</tr>
<tr>
<td>13</td>
<td>2m</td>
<td>3m</td>
<td>76</td>
</tr>
</tbody>
</table>

1H NMR (300 MHz, CDCl₃): \(\delta = 7.89–7.60\) (m, 4 H), 7.60–7.51 (m, 1 H), 7.51–7.34 (m, 2 H), 7.34–7.23 (m, 4 H), 7.23–7.09 (m, 3 H), 7.09–7.00 (m, 5 H), 4.80–4.40 (br, 1 H), 4.40–3.95 (br, 1 H), 3.75 (s, 2 H), 2.28 (s, 3 H).

13C NMR (75 MHz, CDCl₃): \(\delta = 192.9, 160.6, 143.0, 139.6, 136.2, 134.9, 134.3, 130.3, 129.8, 129.4, 129.1, 129.0, 128.9, 128.7, 127.4, 127.0, 126.7, 126.5, 49.0, 41.7, 31.4, 21.8.

1H NMR (300 MHz, CDCl₃): δ = 7.73–7.61 (m, 4 H), 7.61–7.58 (m, 1 H), 7.42–7.37 (m, 3 H), 7.35–7.10 (m, 9 H), 7.01–6.99 (m, 1 H), 4.45–4.38 (br, 1 H), 4.38–4.01 (br, 1 H), 3.69 (s, 2 H), 2.37 (s, 3 H).

1C NMR (75 MHz, CDCl₃): δ = 191.9, 160.5, 141.0, 138.6, 136.2, 134.9, 131.6, 130.0, 129.1, 128.8, 128.4, 128.1, 127.5, 127.4, 127.0, 126.7, 126.2, 121.8, 49.5, 41.5, 31.4, 21.5.

Anal. Calcld for C₁₉H₁₈BrN₂O₃S: C, 68.23; H, 4.39; N, 4.88. Found: C, 68.25; H, 4.30; N, 4.84.

trans-N-[1-[2-(4-bromobenzoyl)-3-phenylaziridin-1-yl]-2-phenylethylidene]-4-methylbenzenesulfonamide (2g)
White solid; mp 133–135 °C (EtOAc–hexane).

IR (KBr): 3435, 1679, 1562, 1303, 1153, 1085 cm⁻¹.

1H NMR (300 MHz, CDCl₃): δ = 7.69–7.60 (m, 2 H), 7.60–7.40 (m, 3 H), 7.40–7.27 (m, 3 H), 7.27–7.17 (m, 5 H), 7.17–7.00 (m, 5 H), 4.78–4.38 (br, 1 H), 4.35–4.00 (br, 1 H), 3.76 (s, 2 H), 2.37 (s, 3 H).

1C NMR (75 MHz, CDCl₃): δ = 212.1, 164.6, 143.0, 139.6, 136.2, 134.6, 133.8, 132.4, 129.8, 129.4, 129.1, 128.9, 128.0, 126.4, 125.8, 125.3, 123.5, 122.7, 53.6, 42.7, 36.6, 25.1.

Anal. Calcld for C₁₉H₁₈BrN₂O₃S: C, 68.23; H, 4.39; N, 4.88. Found: C, 68.28; H, 4.36; N, 4.85.

trans-N-[1-[2-(4-fluorobenzoyl)-3-phenylaziridin-1-yl]-2-phenylethylidene]-4-methylbenzenesulfonamide (2h)
White solid; mp 131–133 °C (EtOAc–hexane).

IR (KBr): 3436, 1682, 1565, 1153, 1085 cm⁻¹.

1H NMR (300 MHz, CDCl₃): δ = 7.95–7.90 (m, 4 H), 7.28–7.23 (m, 4 H), 7.23–7.17 (m, 5 H), 7.17–7.11 (m, 5 H), 4.80–4.40 (br, 1 H), 4.16–4.03 (br, 1 H), 3.75 (s, 2 H), 2.38 (s, 3 H).

1C NMR (75 MHz, CDCl₃): δ = 194.9, 169.7, 141.0, 138.7, 136.2, 134.1, 134.0, 130.1, 129.8, 129.4, 129.0, 128.7, 127.4, 127.0, 126.7, 126.5, 49.7, 41.8, 32.4, 21.8.

Anal. Calcld for C₁₉H₁₈FN₂O₃S: C, 70.29; H, 4.92; N, 5.47. Found: C, 70.38; H, 4.90; N, 5.50.

trans-N-[1-[2-(3-bromobenzoyl)-3-phenylaziridin-1-yl]-2-phenylethylidene]-4-methylbenzenesulfonamide (2i)
White solid; mp 136–137 °C (EtOAc–hexane).

IR (KBr): 3430, 1685, 1566, 1150, 693 cm⁻¹.

1H NMR (300 MHz, CDCl₃): δ = 7.71–7.60 (m, 4 H), 7.30–7.19 (m, 4 H), 7.19–7.09 (m, 10 H), 4.60–4.40 (br, 1 H), 4.60–4.01 (br, 1 H), 3.74 (s, 2 H), 2.39 (s, 3 H).

1C NMR (75 MHz, CDCl₃): δ = 195.9, 164.6, 143.2, 138.6, 136.1, 134.5, 134.3, 130.3, 129.8, 129.2, 129.1, 129.0, 128.7, 127.4, 127.3, 126.4, 125.8, 49.7, 41.5, 38.1, 21.8.

Anal. Calcld for C₁₉H₁₈BrN₂O₃S: C, 68.23; H, 4.39; N, 4.88. Found: C, 68.28; H, 4.42; N, 4.85.

trans-N-[1-[2-(4-methoxybenzoyl)-3-phenylaziridin-1-yl]-2-phenylethylidene]-4-methylbenzenesulfonamide (2j)
Yellow solid; mp 116–118 °C (EtOAc–hexane).

IR (KBr): 3439, 1732, 1600, 1561, 1240, 1145, 667 cm⁻¹.

1H NMR (300 MHz, CDCl₃): δ = 7.75–7.72 (m, 4 H), 7.26–7.20 (m, 3 H), 7.20–7.10 (m, 9 H), 6.88–6.86 (m, 2 H), 4.60–4.40 (br, 1 H), 4.19–4.00 (br, 1 H), 3.90 (s, 3 H), 3.75 (s, 2 H), 2.36 (s, 3 H).

1C NMR (75 MHz, CDCl₃): δ = 192.8, 167.2, 164.6, 142.3, 139.0, 134.5, 133.8, 130.7, 129.8, 129.1, 128.8, 128.3, 128.1, 126.7, 126.4, 125.9, 113.7, 60.0, 55.3, 47.9, 21.2, 13.9.

Anal. Calcld for C₁₉H₁₈O₂N₂S: C, 70.97; H, 5.38; N, 5.34. Found: C, 70.95; H, 5.46; N, 5.37.
trans-N-[1-(2-Benzoyl-3-phenylaziridin-1-yl)heptylidene]-4-methylbenzenesulfonamide (2k)

Oil.

IR (neat): 3346, 1678, 1571, 1300, 1152, 685 cm⁻¹.

1H NMR (300 MHz, CDCl₃): δ = 8.24–7.85 (m, 2 H), 7.85–7.59 (m, 2 H), 7.59–7.46 (m, 1 H), 7.46–7.24 (m, 7 H), 7.24–6.98 (m, 2 H), 4.80–4.30 (br, 1 H), 4.00–3.85 (br, 1 H), 2.80–2.55 (m, 2 H), 2.38 (s, 3 H), 1.95–1.50 (m, 2 H), 1.43–0.60 (m, 6 H), 0.98–0.81 (s, 3 H).

13C NMR (75 MHz, CDCl₃): δ = 141.93, 162.73, 143.00, 139.68, 136.29, 134.95, 134.35, 131.70, 129.18, 129.87, 128.18, 128.85, 128.16, 124.87, 49.81, 31.09, 28.46, 23.21, 21.17. 14.1.

Anal. Calcd for C₃₆H₃₃N₃O₂S: C, 71.28; H, 6.60; N, 5.73. Found: C, 71.31; H, 6.58; N, 5.79.

trans-N-[1-(2-Benzoyl-3-(2-chlorophenyl)aziridin-1-yl)heptylidene]-4-methylbenzenesulfonamide (2l)

Oil.

IR (neat): 3428, 1676, 1580, 1438, 1152, 685 cm⁻¹.

1H NMR (300 MHz, CDCl₃): δ = 8.10–7.82 (m, 2 H), 7.82–7.63 (m, 2 H), 7.63–7.51 (m, 1 H), 7.51–7.40 (m, 2 H), 7.40–7.36 (m, 2 H), 7.36–7.23 (m, 2 H), 7.23–7.05 (m, 4 H), 4.65–4.35 (br, 1 H), 4.25–4.02 (br, 1 H), 2.85–2.50 (m, 2 H), 2.34 (s, 3 H), 1.96–1.60 (m, 2 H), 1.48–1.00 (m, 6 H), 0.88–0.67 (s, 3 H).

13C NMR (75 MHz, CDCl₃): δ = 190.66, 164.45, 142.42, 139.90, 135.67, 133.73, 132.60, 129.71, 129.28, 128.85, 128.14, 127.14, 49.79, 47.35, 31.08, 28.58, 27.25, 21.22, 21.11, 13.7.


trans-N-[1-(2-Benzoyl-3-(2-bromophenyl)aziridin-1-yl)heptylidene]-4-methylbenzenesulfonamide (2m)

Oil.

IR (neat): 3426, 1676, 1581, 1437, 1153, 679 cm⁻¹.

1H NMR (300 MHz, CDCl₃): δ = 8.10–7.80 (m, 2 H), 7.80–7.59 (m, 2 H), 7.59–7.46 (m, 2 H), 7.46–7.40 (m, 2 H), 7.40–7.36 (m, 2 H), 7.36–7.23 (m, 2 H), 7.23–7.05 (m, 4 H), 4.65–4.35 (br, 1 H), 4.25–4.02 (br, 1 H), 2.85–2.50 (m, 2 H), 2.34 (s, 3 H), 1.97–1.60 (m, 2 H), 1.43–1.03 (m, 6 H), 0.98–0.83 (s, 3 H).

13C NMR (75 MHz, CDCl₃): δ = 190.66, 164.55, 142.42, 139.90, 135.67, 134.32, 133.73, 132.39, 129.71, 129.28, 128.48, 127.64, 21.52, 24.84, 47.97, 47.35, 31.08, 29.44, 28.24, 21.22, 13.17.


trans-(2-Benzyl-5-phenyl-1-tosyl-4,5-dihydro-1H-imidazol-4-yl)(phenyl)methanone (3a)

Typical Procedure

A mixture of 2a (124 mg, 0.25 mmol) and anhyd NaI (150 mg, 1 mmol) in acetone (15 mL) was allowed to stir for 6 h. The solvent was removed in vacuo and the residue taken up in CH₂Cl₂ (15 mL). The organic layer was washed with H₂O (3 × 10 mL) and dried (Na₂SO₄). Evaporation of the solvent in vacuo and purification of the residue by flash chromatography on silica column gave 3a; yield: 94 mg (75%); white solid; mp 146–149 °C (Table 2).

IR (KBr): 2924, 1685, 1376, 1162 cm⁻¹.

1H NMR (300 MHz, CDCl₃): δ = 8.04–8.01 (m, 2 H), 7.56–7.43 (m, 1 H), 7.43–7.38 (m, 6 H), 7.38–7.33 (m, 3 H), 7.33–7.20 (m, 3 H), 7.20–7.09 (m, 4 H), 5.77–5.73 (d, J = 4.8 Hz, 2 H), 5.22–5.20 (d, J = 4.8 Hz, 2 H), 4.31–4.26 (d, J = 14.4 Hz, 1 H), 4.04–3.99 (d, J = 14.4 Hz, 1 H), 2.37 (s, 3 H).

13C NMR (75 MHz, CDCl₃): δ = 193.35, 159.44, 144.34, 141.12, 135.51, 135.34, 136.69, 129.69, 129.31, 128.85, 128.16, 128.00, 127.45, 126.9, 126.3, 79.3, 64.1, 36.1, 21.5.
(trans)-2-Benzyl-5-(3-bromophenyl)-1-tosyl-4,5-dihydro-1H-imidazol-4-yl phenylmethanone (3i)

Yellow solid; mp 121–123 °C.


(trans)-2-Benzyl-5-phenyl-1-tosyl-4,5-dihydro-1H-imidazol-4-yl(phenyl)methanone (3f)

White solid; mp 135–136 °C.

Anal. Calcd for C31H28N2O4S: C, 70.97; H, 5.38; N, 5.34. Found: C, 70.90; H, 5.42; N, 5.30.

(trans)-2-Benzyl-5-phenyl-1-tosyl-4,5-dihydro-1H-imidazol-4-yl(phenyl)methanone (3g)

Oil.


(trans)-5-(2-Chlorophenyl)-2-hexyl-1-tosyl-4,5-dihydro-1H-imidazol-4-yl(phenyl)methanone (3k)

Yellow solid; mp 119–120 °C.

Anal. Calcd for C31H28N2O4S: C, 71.28; H, 6.60; N, 5.73. Found: C, 71.30; H, 6.53; N, 5.76.

(trans)-5-(2-Chlorophenyl)-2-hexyl-1-tosyl-4,5-dihydro-1H-imidazol-4-yl(phenyl)methanone (3m)

White solid; mp 119–120 °C.

IR (KBr): 3437, 1689, 1361, 1164 cm⁻¹.

1H NMR (300 MHz, CDCl₃): δ = 8.10–8.01 (m, 2 H), 7.69–7.61 (m, 2 H), 7.61–7.52 (m, 1 H), 7.52–7.20 (m, 9 H), 5.84–5.83 (d, J = 5.1 Hz, 2 H), 5.22–5.19 (d, J = 5.1 Hz, 2 H), 2.78–2.75 (m, 1 H), 2.59–2.46 (m, 1 H), 2.40 (s, 3 H), 1.77–1.66 (m, 6 H), 1.35–1.13 (m, 6 H), 0.89–0.75 (s, 3 H).

13C NMR (75 MHz, CDCl₃): δ = 191.9, 163.9, 159.1, 152.5, 144.3, 141.4, 135.5, 131.9, 129.6, 128.9, 128.4, 127.9, 127.4, 126.3, 126.1, 113.7, 79.5, 64.1, 55.4, 35.9, 21.5.

Anal. Calcd for C31H28N2O4S: C, 71.28; H, 6.60; N, 5.73. Found: C, 71.30; H, 6.53; N, 5.76.
6.23–6.21 (d, J = 4.8 Hz, 2 H), 5.14–5.12 (d, J = 4.8 Hz, 2 H), 2.91–2.81 (m, 1 H), 2.53–2.49 (m, 1 H), 2.47 (s, 3 H), 1.88–1.60 (m, 2 H), 1.36–1.26 (m, 6 H), 0.83–0.82 (s, 3 H).

$^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ = 192.9, 160.6, 144.7, 135.7, 134.6, 133.5, 133.1, 129.9, 129.4, 129.2, 128.5, 127.9, 127.7, 121.3, 63.9, 31.3, 30.1, 28.8, 26.8, 22.4, 21.6, 13.9.

Anal. Caled for C$_2$H$_3$BrN$_2$O$_3$S: C, 61.37; H, 5.51; N, 4.94. Found: C, 61.42; H, 5.50; N, 4.90.

trans-[(2-Benzyl-5-p-toly-1-tosyl-4,5-dihydro-1H-imidazol-4-yl)(phenyl)methanone (3n)]

Acknowledgment

We would like to thank the NSF-20621091 and the ‘Hundred Scientist Program’ from the Chinese Academy of Sciences for the financial support of this work.

References


