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# Silver–NHC mediated C–C bond activation of alkyl nitriles and catalytic application in oxazoline synthesis

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## **Electronic Supporting Information**

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## 1. Experimental procedures for the synthesis of Ag-NHC complexes 2-5

#### General

All solvents used were purchased from commercial suppliers and used with no further purification. All the commercially available chemicals were used as received. Ag<sub>2</sub>O was stored in a desiccator in the absence of light. The synthesis of Ag complexes was conducted in the absence of light, and all manipulations were performed with no additional precautions for air or moisture sensitivity. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on Varian Innova spectrometers operating at 300, 400 or 500 MHz. Chemical shifts ( $\delta$  in ppm) were referenced to residual solvent resonances. Liquid IR (in CH<sub>2</sub>Cl<sub>2</sub>) and solid state IR spectra were recorded on a Bruker VERTEX 70 IR spectrometer in the 400 – 4000 cm<sup>-1</sup> region at 1 cm<sup>-1</sup> resolution. Elemental analysis of C, H and N were performed by the Microanalytical Laboratory at University College Dublin, Ireland, using an Exeter Analytical CE-440 elemental analyzer. Gas chromatography spectra were obtained using a Shimadzu GC-174 gas chromatograph. Compounds **1a**,<sup>S1</sup> **1b**<sup>S2</sup> and azolium salt precursors of complexes **3**,<sup>S3</sup> **4**,<sup>S4</sup> and **5**<sup>S5</sup> were synthesized according to previously reported procedures.

#### General procedure for the synthesis of Ag-NHC complexes 2-5

The azolium salt (0.45 mmol, 1 equiv), and  $Ag_2O$  (0.79 mmol, 1.75 equiv) were stirred in CH<sub>3</sub>CN at reflux for 3 hours in the absence of light. A white precipitate (AgI) was usually observed by the end of the reaction. The solution was returned to RT, and CH<sub>2</sub>Cl<sub>2</sub> (10-15 mL) was added. The solution was filtered through Celite and solvent removed *in vacuo*. The residue was dissolved in CH<sub>2</sub>Cl<sub>2</sub> and precipitated with diethyl ether/pentane. The precipitate was collected by filtration and washed with excess Et<sub>2</sub>O. Complexes **3**, **4** and **5** were obtained as sticky solids.

#### Complex 2a/2b (mixture)

According to the general method, **1a** (150 mg, 0.37 mmol), Ag<sub>2</sub>O (150 mg, 0.65 mmol) were reacted in CH<sub>3</sub>CN at reflux for 3 h. A mixture of complexes **2a** and **2b** were obtained as an off-white solid; combined yield: 106 mg, 67 % (based on 83% of 2a, 17% of 2b). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.65–7.63 (m, 2H, H<sub>Ph</sub>), 7.57–7.55 (m, 3H, H<sub>Ph</sub>), 7.01 (s, 2H, H<sub>Mes</sub>), 4.23 (s, 3H, NCH<sub>3</sub>), 2.38 (s, 3H, CH<sub>3 Mes</sub>), 2.02 (s, 6H, CH<sub>3 Mes</sub>); <sup>13</sup>C {<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 100 MHz):  $\delta$  149.30 (*C*<sub>trz</sub>-Ph), 140.74, 136.27, 134.04 (3 × C<sub>Mes</sub>), 130.52 (C<sub>Ph</sub>), 129.63 (C<sub>Mes</sub>), 129.52, 129.45, 126.94 (3 × C<sub>Ph</sub>), 37.71 (NCH<sub>3</sub>), 21.31 (CH<sub>3 Mes</sub>), 17.69 (CH<sub>3 Mes</sub>), C<sub>carbene</sub> and C<sub>cyano</sub> not observed;. Anal calcd for 0.83 C<sub>19</sub>H<sub>19</sub>AgN<sub>4</sub> + 0.17 C<sub>18</sub>H<sub>19</sub>AgIN<sub>3</sub>: C, 53.23; H, 4.50; N, 12.70. Found: C, 53.29; H, 4.35; N, 12.47.

# **Complex 2a**

According to the general method, **1b** (150 mg, 0.41 mmol), Ag<sub>2</sub>O (167 mg, 0.72 mmol) and KCN (53 mg, 0.82 mmol) were reacted in refluxing CH<sub>3</sub>CN for 3 h. Complex **2a** was obtained as a white solid; Yield: 125 mg, 74 %. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  7.64–7.62 (m, 2H, H<sub>Ph</sub>), 7.56–7.54 (m, 3H, H<sub>Ph</sub>), 7.00 (s, 2H, H<sub>Mes</sub>), 4.22 (s, 3H, NCH<sub>3</sub>), 2.38 (s, 3H, CH<sub>3Mes</sub>), 2.01 (s, 6H, CH<sub>3Mes</sub>); <sup>13</sup>C{<sup>1</sup>H} (CDCl<sub>3</sub>, 100 MHz):  $\delta$  149.39 (*C*<sub>trz</sub>-Ph), 140.82, 136.27, 134.02 (3 × C<sub>Mes</sub>), 130.58 (C<sub>Ph</sub>), 129.67 (C<sub>Mes</sub>), 129.54, 129.50, 126.93 (3 × C<sub>Ph</sub>), 37.69 (NCH<sub>3</sub>), 21.33 (CH<sub>3 Mes</sub>), 17.67 (CH<sub>3 Mes</sub>), C<sub>carbene</sub> and C<sub>cyano</sub> not observed; IR (CH<sub>2</sub>Cl<sub>2</sub>) 2203.28 (*v*<sub>CN</sub>); ESI-MS (positive):<sup>S6</sup> m/z 660.87, 662.88 ([trz–Ag–trz]<sup>+</sup>; ESI-MS (negative):<sup>S6</sup> m/z 158.45, 160.44 ([NC–Ag–CN]<sup>-</sup>; Anal. Calcd for C<sub>19</sub>H<sub>19</sub>AgN<sub>4</sub> (411.25) × 0.3 CH<sub>2</sub>Cl<sub>2</sub>: C, 53.08; H, 4.52; N, 12.83. Found: C, 52.95; H, 4.26; N, 13.07.

# **Complex 2b**

According to the general method, **1a** (150 mg, 0.37 mmol) and Ag<sub>2</sub>O (150 mg, 0.65 mmol) were reacted in CH<sub>2</sub>Cl<sub>2</sub> at RT for 24 h. Complex **2b** was obtained as a white solid; Yield: 116 mg, 61%. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 300 MHz):  $\delta$  = 7.66–7.63 (m, 2H, H<sub>Ph</sub>), 7.55–7.53 (m, 3H, H<sub>Ph</sub>), 6.99 (s, 2H, H<sub>Mes</sub>), 4.24 (s, 3H, NCH<sub>3</sub>), 2.38 (s, 3H, H<sub>Mes</sub>), 2.01 (s, 6H, H<sub>Mes</sub>); <sup>13</sup>C{<sup>1</sup>H} (CDCl<sub>3</sub>, 125 MHz):  $\delta$  = 149.07 (C<sub>trz</sub>-Ph), 140.61, 136.33, 134.06 (3 × C<sub>Mes</sub>), 130.49 (C<sub>Ph</sub>), 129.58 (C<sub>Mes</sub>), 129.51, 129.46, 127.01(3 × C<sub>Ph</sub>), 37.86 (NCH<sub>3</sub>), 21.36 (CH<sub>3 Mes</sub>), 17.69 (CH<sub>3 Mes</sub>), C<sub>carbene</sub> not observed; ESI-MS (positive):<sup>S6</sup> m/z 660.87, 662.88 ([trz–Ag–trz]<sup>+</sup>; ESI-MS (negative):<sup>S6</sup> m/z 359.85, 361.73 ([I–Ag–I]<sup>-</sup>; Anal. Calcd for C<sub>18</sub>H<sub>19</sub>AgIN<sub>3</sub> (512.14) × 0.25 CH<sub>2</sub>Cl<sub>2</sub>: C, 41.10; H, 3.69; N, 7.88. Found: C, 41.32; H, 3.48; N, 7.77.

# Complex 3

According to the general method, 1,4-dibutyl-3-methyl-1,2,3-triazolium iodide salt (144 mg, 0.62 mmol) and Ag<sub>2</sub>O (300 mg, 1.29 mmol) were reacted in CH<sub>3</sub>CN at reflux for 3 hours. Complex **3** was obtained as a brown oil; Yield 94 mg, 38 % (based on 94% cyano and 6% iodo complex). <sup>1</sup>H NMR data is identical to published data. <sup>S3</sup> <sup>1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta$  = 4.34 (t, <sup>3</sup>*J*<sub>*HH*</sub> = 7.4 Hz, 2H, NC*H*<sub>2</sub>), 4.00 (s, 3H, NC*H*<sub>3</sub>), 2.71 (t, <sup>3</sup>*J*<sub>*HH*</sub> = 7.6 Hz, 2H C<sub>trz</sub>–C*H*<sub>2</sub>), 1.93 (quint, <sup>3</sup>*J*<sub>*HH*</sub> = 7.4 Hz, 2H, NCH<sub>2</sub>C*H*<sub>2</sub>), 1.68 (quint, <sup>3</sup>*J*<sub>*HH*</sub> = 7.6 Hz, 2H, C<sub>trz</sub>–CH<sub>2</sub>C*H*<sub>2</sub>), 1.42–1.36 (m, 2H, C<sub>trz</sub>–CH<sub>2</sub>CH<sub>2</sub>C*H*<sub>2</sub>), 1.35–1.29 (m, 2H, NCH<sub>2</sub>CH<sub>2</sub>C*H*<sub>2</sub>), 0.94 (m, 6H, CH<sub>2</sub>, *CH*<sub>3</sub>); <sup>13</sup>C{<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta$  = 163.42 (C<sub>carbene</sub>), 149.00 (C<sub>trz</sub>), 55.89 (NCH<sub>2</sub>), 35.94 (NCH<sub>3</sub>), 32.80 (NCH<sub>2</sub>CH<sub>2</sub>), 31.63 (C<sub>trz</sub>–CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>), 25.23 (C<sub>trz</sub>–CH<sub>2</sub>), 22.40 (C<sub>trz</sub> – CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>), 19.72 (NCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>), 13.83 (C<sub>trz</sub>–CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>), 13.57 (NCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>3</sub>); ESI-MS (positive): <sup>S6</sup> m/z 497.24, 499.25 ([trz–Ag–trz]<sup>+</sup>; ESI-MS (negative): <sup>S6</sup> m/z 158.46, 160.45 ([NC–Ag–CN]<sup>-</sup>; Anal. Calcd for 0.95 C<sub>12</sub>H<sub>21</sub>AgN<sub>4</sub> + 0.05 C<sub>11</sub>H<sub>21</sub>AgIN<sub>3</sub> × 0.5 MeOH: C, 42.69; H, 6.62; N, 15.80. Found: C, 43.04; H, 6.46, N, 15.72.

#### **Complex 4**

According to the general method, 1,3-dibutyl-1,3-imidazolium iodide salt (100 mg, 0.32 mmol) and Ag<sub>2</sub>O (130 mg, 0.56 mmol) were reacted in CH<sub>3</sub>CN at reflux for 3 hours. Complex **4** was obtained as a brown oil; Yield: 92 mg, 84% (based on 30% cyano and 70% iodo complex). NMR spectral data are identical to the corresponding silver biscarbene analogue.<sup>S7 1</sup>H NMR (CDCl<sub>3</sub>, 500 MHz):  $\delta = 6.95$  (s, 2H, CH<sub>Ar</sub>), 4.09 (t, <sup>3</sup>*J*<sub>HH</sub> = 7.2 Hz, 4H, NCH<sub>2</sub>), 1.77 (quintet, <sup>3</sup>*J*<sub>HH</sub> = 7.4 Hz, 4H, NCH<sub>2</sub>CH<sub>2</sub>), 1.32(sextet, <sup>3</sup>*J*<sub>HH</sub> = 7.4 Hz, 4H, CH<sub>2</sub>CH<sub>3</sub>), 0.95 (t, 6H, <sup>3</sup>*J*<sub>HH</sub> = 7.4 Hz, 6H, CH<sub>2</sub>CH<sub>3</sub>); <sup>13</sup>C {<sup>1</sup>H} NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta = 120.98$  (C<sub>Ar</sub>), 51.95 (NCH<sub>2</sub>), 33.64 (NCH<sub>2</sub>CH<sub>2</sub>), 19.81 (CH<sub>2</sub>CH<sub>3</sub>), 13.73 (CH<sub>3</sub> Anal. Calcd for a 7:3 mixture of iodo and cyano complex, 0.30 C<sub>12</sub>H<sub>20</sub>AgN<sub>3</sub> + 0.70 C<sub>11</sub>H<sub>20</sub>AgIN<sub>2</sub>: C, 35.27; H, 5.24; N, 8.37. Found: C, 35.66; H, 5.14; N, 8.08.

# **Complex 5**

According to the general method, 1,3-dibutyl-1,3-benzimidazolium iodide salt (130 mg, 0.36 mmol) and Ag<sub>2</sub>O ( 146 mg, 0.63 mmol) were reacted in CH<sub>3</sub>CN at reflux for 3 hours. Complex **5** was obtained as an off-white solid and NMR spectra are identical to those of published silver benzimidazolylidene salts.<sup>S8</sup> Yield: 66 mg, 58 % (based on 94% of cyano complex, 6% iodo complex). <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz):  $\delta$  = 7.49–7.47 (m, 2H, Ar), 7.42–7.40 (m,2H, Ar), 4.38–4.34 (t, <sup>3</sup>*J*<sub>HH</sub> = 7.3 Hz, 4H, NCH<sub>2</sub>), 1.92–1.84 (m, 4H, NCH<sub>2</sub>*CH*<sub>2</sub>), 1.45–1.35 (m, 4H, NCH<sub>2</sub>CH<sub>2</sub>*CH*<sub>2</sub>), 0.98 (t, <sup>3</sup>*J*<sub>HH</sub> = 7.3 Hz, 6H, CH<sub>3</sub>); <sup>13</sup>C {<sup>1</sup>H}NMR (CDCl<sub>3</sub>, 125 MHz):  $\delta$  133.75 (C4<sub>Ar quaternary</sub>), 124.28 (C6<sub>Ar</sub>), 111.67 (C5<sub>Ar</sub>) 49.48 (NCH<sub>2</sub>), 32.56 (NCH<sub>2</sub>*C*H<sub>2</sub>), 20.15 (NCH<sub>2</sub>CH<sub>2</sub>*C*H<sub>2</sub>), 13.86 (CH<sub>3</sub>). Anal Calcd for 0.94 C<sub>16</sub>H<sub>22</sub>AgN<sub>3</sub> + 0.06 C<sub>15</sub>H<sub>22</sub>AgIN<sub>2</sub>: C, 51.70; H, 5.99; N, 11.12. Found: C, 52.07; H, 5.91, N, 10.73.

# NMR spectra of complexes 2–5

Complex 2a/2b



Complex 2a/2b

 $^{13}C NMR$ 



**Complex 2a** <sup>1</sup>*H NMR* 



Complex 2a

 $^{13}C NMR$ 



# Complex 2b



Complex 2b

 $^{13}CNMR$ 



**Complex 3** <sup>1</sup>*H NMR* 



Complex 3
<sup>13</sup>C NMR



# Complex 4



**Complex 4** <sup>13</sup>C NMR



# **Complex 5**



Complex 5
<sup>13</sup>C NMR



## 3. Experimental procedure for the catalytic synthesis of oxazolines

In a typical procedure, 1,3,5-trimethyoxybenzene (21 mg, 0.125 mmol, internal NMR standard) and 4-bromobenzaldehydye (50.9 mg, 0.275 mmol) were dissolved in CH<sub>2</sub>Cl<sub>2</sub> (0.75 mL) in a 15 mL snap top vial containing a magnetic stirring bar. Methyl isocyanoacetate (25  $\mu$ L, 0.25 mmol) was then added via syringe. To the vial, a catalyst solution (0.1 mL, 3 x 10<sup>-4</sup> mmol, 0.125 mol% in CH<sub>2</sub>Cl<sub>2</sub>) was added and the vial was sealed. The reaction was run for 6 hours at room temperature. Aliquots were taken at 5, 20, 60, 180 and 360 min, and filtered through a pad of silica. Yields were determined by comparison of the 1,3,5-trimethyoxybenzene/methyl isocyanoacetate integral ratio, and cis/trans ratios were determined by <sup>1</sup>H NMR spectroscopy.



#### 4. Time-conversion profile for oxazoline synthesis

**Figure S1:** Time-conversion profile for the aldol condensation of methyl isocyanoacetate with 4bromobenzaldehyde by complexes 2-5 in CH<sub>2</sub>Cl<sub>2</sub> at room temperature. Yields determined by <sup>1</sup>H NMR spectroscopy (1,3,5-trimethoxybenzene as internal standard).

#### 5. X-ray crystallographic data

The single crystals of mixed complex 2 suitable for X-ray diffraction study were grown from slow diffusion of diethyl ether to a concentration solution on 2 in CHCl<sub>3</sub>. Crystal data was collected using an Agilent Technologies SuperNova A diffractometer fitted with an Atlas

detector at 100(2) K using Mo-K $\alpha$  radiation (0.71073 Å). A complete (**2a/b**) or three times redundant (**2a**) dataset was collected, assuming that the Friedel pairs are not equivalent. The structures were solved by direct methods using SHELXS-97 and refined by full-matrix least-squares on F<sub>2</sub> using SHELXS-97.<sup>S9</sup>

CCDC No	1044757		
Empirical formula	$C_{18.86}H_{19}N_{3.86}AgI_{0.14}$		
Molecular formula	0.86 (C <sub>19</sub> H <sub>19</sub> N <sub>4</sub> Ag) x 0.14 (C <sub>18</sub> H <sub>19</sub> N <sub>3</sub> AgI)		
Formula weight	424.97		
Temperature	100(2) K		
Wavelength	0.71073 Å		
Crystal system	Monoclinic		
Space group	P2 <sub>1</sub> /n (#14)		
Unit cell dimensions	a = 9.6546(2)  Å	$\alpha = 90^{\circ}$	
	b = 10.6457(2) Å	$\beta = 90.385(2)^{\circ}$	
	c = 17.1150(3)  Å	$\gamma = 90^{\circ}$	
Volume	1759.04(6) Å <sup>3</sup>		
Z	4		
Density (calculated)	1.605 Mg/m <sup>3</sup>		
Absorption coefficient	1.391 mm <sup>-1</sup>		
F(000)	853.8		
Crystal size	0.2451 x 0.1972 x 0.1125 mm <sup>3</sup>		
Theta range for data collection	2.85 to 28.29°.		
Index ranges	-12<=h<=12, -14<=k<=14, -22<=l<=22		
Reflections collected	23920		
Independent reflections	4362 [R(int) = 0.0371]		
Completeness to $\theta = 28.29^{\circ}$	99.8 %		
Absorption correction	Analytical		
Max. and min. transmission	0.975 and 0.937		
Refinement method	Full-matrix least-squares on F <sup>2</sup>		
Data / restraints / parameters	4362 / 0 / 231		
Goodness-of-fit on F <sup>2</sup>	1.060		
Final R indices [I>2sigma(I)]	R1 = 0.0275, WR2 = 0.0632		
R indices (all data)	R1 = 0.0336, $wR2 = 0.0675$		
Largest diff. peak and hole	0.671 and -0.495 e.Å <sup>-3</sup>		

 Table S1. Crystal data and structure refinement for 2a/2b co-crystals.

CCDC No.	1044758		
Empirical formula	$C_{19}H_{19}N_4Ag$		
Formula weight	411.25		
Temperature	100(2) K		
Wavelength	0.71073 Å		
Crystal system	Monoclinic		
Space group	P2 <sub>1</sub> /n (#14)		
Unit cell dimensions	$a = 9.59019(8) \text{ Å} \qquad \alpha = 90^{\circ}$		
	b = 10.57651(9) Å $\beta$ = 90.1567(7)°		
	$c = 17.1999(2) \text{ Å} \qquad \gamma = 90^{\circ}$		
Volume	1744.59(3) Å <sup>3</sup>		
Z	4		
Density (calculated)	1.566 Mg/m <sup>3</sup>		
Absorption coefficient	1.162 mm <sup>-1</sup>		
F(000)	832		
Crystal size	0.2190 x 0.1538 x 0.0831 mm <sup>3</sup>		
Theta range for data collection	2.87 to 32.81°.		
Index ranges	-14<=h<=14, -15<=k<=15, -25<=l<=25		
Reflections collected	38715		
Independent reflections	6171 [R(int) = 0.0252]		
Completeness to $\theta = 32.00^{\circ}$	98.9 %		
Absorption correction	Analytical		
Max. and min. transmission	0.930 and 0.844		
Refinement method	Full-matrix least-squares on F <sup>2</sup>		
Data / restraints / parameters	6171 / 0 / 221		
Goodness-of-fit on F <sup>2</sup>	1.043		
Final R indices [I>2sigma(I)]	R1 = 0.0191, $wR2 = 0.0445$		
R indices (all data)	R1 = 0.0226, $wR2 = 0.0464$		
Largest diff. peak and hole	0.456 and -0.309 e.Å <sup>-3</sup>		

Table S2. Crystal data and structure refinement for 2a.

# 5. References

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 $2 \text{ NHC-Ag-X} \longrightarrow [\text{NHC-Ag-NHC}]^+[\text{AgX}_2]^-$ 

either because of the dilute conditions used for MS or during ionization, or both. Both ions were unambiguously identified for complexes **2a** (X = CN) and **2b** (X = I), and **3** (X = CN), readily identified by an intense (100%) M+2 signal due to almost equal <sup>107</sup>Ag and <sup>109</sup>Ag isotope distribution (51.8% and 48.2% natural abundance, both signals are reported).

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