Supporting Information

For

Copper-mediated Electrophilic Imination of Alkenylzirconocenes with *O*-Benzoyl Ketoximes and Aldoximes

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General Considerations

All manipulations were conducted in Schlenk tube and under nitrogen with a slightly positive pressure. Unless otherwise noted, all starting materials were commercially available and were used without further purification. Benzophenone *O*-benzoyloxime (**2a**) was obtained by the reaction of benzophenone oxime with benzoyl chloride,¹ and the benzophenone oxime was obtained by the reaction of hydroxylamine hydrochloride and benzophenone.¹ Other *O*-benzoyloxime **2b**,¹ **2c**,¹ **2d**,¹ **2e**,¹ **2f**,² and **2g**² were synthesized through the corresponding oxime with benzoyl chloride.¹ Tetrahydrofuran (THF) was refluxed and freshly distilled from dark purple solutions of sodium and benzophenone under nitrogen atmosphere. ¹H NMR and ¹³C NMR spectra were recorded on 300MHz NMR spectrometer with TMS as internal standard. GC-MS spectra were recorded on Hewlett Packard GC-MS system. Mass spectra were obtained using a Bruker Esquire ion trap mass spectrometer in positive ion mode.

Experimental Procedures

1. Preparation of alkenylzirconocenes:

Method A: Preparation of alkenylzirconocenes **1a-1d**. To a solution of Cp_2ZrCl_2 (176 mg, 0.6 mmol) in 3 mL of THF, EtMgCl (2 M in THF, 1.2 mmol), was added at -78 °C and the mixture was stirred for 1 h at the same temperature. To this solution, internal alkyne (0.5 mmol) was added and stirred for 2 h at 0 °C. Then, EtOH (0.55 mmol) was added. The solution was warmed to room temperature and stirred for 1 h to afford the corresponding alkenylzirconocene **1a-1d**.

Method B: Preparation of alkenylzirconocene **1e**: To a solution of Cp_2ZrCl_2 (176 mg, 0.6 mmol) in 3 mL of THF, BuLi (1.6 M in hexane, 1.2 mmol), was added at -78 °C and the mixture was stirred for 1 h at the same temperature. To this solution, 7-phenylhept-1-en-6-yne (0.5 mmol) was added and stirred for 2 h at room temperature. Then, EtOH (0.55 mmol) was added and stirred for 1 h to afford the corresponding alkenylzirconocene **1e**.

Method C: Preparation of alkenylzirconocenes **1f**: To a solution of Cp_2ZrCl_2 (176 mg, 0.6 mmol) in 3 mL of THF, BuLi (1.6 M in hexane, 1.2 mmol) was added at -78 °C and the mixture was stirred for 1h at the same temperature. To this solution, phenylethylene (2 mmol) was added and stirred for 2 h at room temperature. 3-hexyne was added to the solution and stirred for 2 h at 0 °C. Then, EtOH (0.55 mmol) was added and stirred for 1 h at room temperature to afford the corresponding alkenylzirconocene **1f**.

Method D: Preparation of alkenylzirconocenes **1g-1h**. To a solution of Cp_2ZrCl_2 (176 mg, 0.6 mmol) in 3 mL of THF, EtMgCl (2 M in THF, 1.2 mmol) was added at -78 °C and the mixture was stirred for 1h at the same temperature. To this solution, internal alkyne (0.5 mmol) was added and stirred for 2 h at 0 °C. Then, diallyl ether (1.0 mmol) was added and stirred at 35 °C for 1h to afford the corresponding alkenylzirconocene **1g-1h**.

2. General procedure for the imination of alkenylzirconocenes with O-benzoyloxime:

To a solution of alkenylzirconocene 1 prepared in situ by method A-D, CuCl

(50 mg, 0.5 mmol) was added at 0 °C for 10-15 min, then, benzophenone *O*-benzoyloxime (226 mg, 0.75 mmol) was added. After stirred for 12 h at 50 °C, the reaction mixture was quenched with water and extracted with dichloromethane (DCM). The solvent was evaporated and residue was purified by flash chromatography (alumina, buffered by Et_3N , 10:1 = PE:EtOAc) to afford the 2-azadiene **3**.

3. General procedure for the imination of alkenylzirconocenes with O-benzoyloxime on 10 mmol.

To a solution of Cp₂ZrCl₂ (3.52g, 12 mmol) in 30 mL of THF, EtMgCl (2 M in THF, 12 mL, 24.0 mmol) was added at -78 °C and the mixture was stirred for 1 h at the same temperature. To this solution, internal alkyne (10 mmol) was added and stirred for 2-4 h at 0 °C. Then, EtOH (0.64 mL, 11 mmol) was added. The solution was warmed to room temperature and stirred for 1 h to afford the corresponding alkenylzirconocene **1a-1c**. To above solution, CuCl (0.99 g, 10 mmol) was added at 0 °C for 20 min. Then, benzophenone *O*-benzoyloxime (4.52 g, 15 mmol) was added. After stirred for 12 h at 50 °C, the reaction mixture was quenched with water and extracted with dichloromethane (DCM). The solvent was evaporated and residue was purified by flash chromatography (alumina, buffered by Et₃N, 10:1 = PE:EtOAc) to afford the 2-azadiene **3aa** (2.44 g, 63%), **3ba** (1.31 g, 45%), and **3ca** (1.29 g, 38%).

4. NMR data for 2-azadienes 3

$$\begin{array}{c} Ph & Ph \\ Ph & \swarrow \\ Ph & Et \\ Ph & 3aa \end{array}$$

Yellow oil (147 mg), 76% yield. ¹H NMR (300MHZ, CDCl₃) δ 0.86 (t, J = 7.5 Hz, 3H), 2.47 (q, J = 7.5 Hz, 2H), 6.71-6.73 (m, 2H), 6.84-6.94 (m, 6H), 7.04-7.06 (m, 3H), 7.17-7.23 (m, 5H), 7.33-7.41 (m, 2H), 7.78-7.80 (m, 2H); ¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 11.9, 26.4, 125.4, 125.8, 126.0, 127.1, 127.7, 127.8, 128.2, 128.5, 129.1, 129.6, 130.1, 130.6, 137.7, 139.4, 140.5, 141.3, 143.8, 168.3. GC-MS found for 387, HRMS (ESI mode) calcd for C₂₉H₂₅N+H⁺ 388.2060, found 388.2062.



Yellow oil (79 mg), 54% yield. ¹H NMR (300MHZ,CDCl₃) δ 0.73-0.83 (m, 6H), 0.93 (t, J = 7.2Hz, 3H), 1.79-1.94 (m, 6H), 7.15-7.16 (m, 2H), 7.27-7.33 (m, 6H), 7.59-7.61 (m, 2H); ¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 12.0, 13.3, 13.4, 22.5, 23.3, 25.1, 121.9, 127.8, 128.0, 128.3, 128.6, 128.9, 129.9, 137.5, 140.1, 142.8, 164.8. GC-MS found for 291, HRMS (ESI mode) calcd for C₂₁H₂₅N+H⁺ 292.2060, found 292.2057.

$$\begin{array}{c} \begin{array}{c} Ph & Et \\ Ph & Et \\ Ph & Et \end{array}$$

Yellow oil (81 mg), 48% yield. ¹H NMR (300MHZ,CDCl₃) δ 0.87 (t, *J* = 7.5 Hz, 3H), 1.04 (t, *J* = 7.5 Hz, 3H), 2.02 (q, *J* = 7.5 Hz, 2H), 2.24 (q, *J* = 7.5 Hz, 2H), 6.81-6.86 (m, 4H), 7.03-7.04 (m, 3H), 7.17-7.27 (m, 3H), 7.33-7.39 (m, 3H), 7.70-7.72 (m, 2H); ¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 12.0, 13.5, 22.9, 23.3, 126.0, 127.2, 127.3, 127.6, 128.0, 128.1, 128.2, 128.9, 130.2, 138.1, 139.7, 140.7, 142.0, 167.3. GC-MS found for 339, HRMS (ESI mode) calcd for C₂₅H₂₅N+H⁺ 340.2060, found 340.2064. Me



Yellow oil (99 mg), 69% yield. ¹H NMR (300MHZ, CDCl₃) δ 0.90 (t, *J* = 7.5 Hz, 3H),1.66 (s, 3H), 1.84 (s, 3H), 2.26 (q, *J* = 7.5 Hz, 2H), 7.23-7.26 (m, 2H), 7.30-7.36 (m, 6H); 7.68-7.70 (m, 2H). ¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 4.31, 12.0, 18.9, 25.5, 89.5, 125.9, 127.7, 128.0, 128.5, 128.8, 129.2, 130.1, 136.3, 137.7, 140.2, 166.4. GC-MS found for 287, HRMS (ESI mode) calcd for C₂₁H₂₁N+H⁺ 288.1747, found 288.1750.



Yellow oil (89 mg), 51% yield. ¹H NMR (300MHZ, CDCl₃) δ 1.14 (d, *J* =5.2 Hz, 3H), 1.33-1.37 (m, 1H), 1.44-1.49 (m, 1H), 1.68-1.74 (m, 1H), 1.79-1.83 (m, 1H), 2.19-2.24 (m, 1H), 2.33-2.41 (m, 1H), 2.91-2.93 (m, 1H), 6.89-6.91 (m, 2H), 6.95-7.03 (m, 5H), 7.11-7.18 (m, 3H), 7.34-7.39 (m, 3H), 7.74-7.76 (m, 2H); ¹³C NMR (75MHz, CDCl₃) δ 19.8, 25.0, 31.7, 34.9, 36.8, 125.7, 127.3, 127.4, 128.0, 128.1, 128.2, 128.9, 130.1, 134.6, 138.3, 139.4, 139.9, 141.5, 166.8. GC-MS found for 351, HRMS (ESI mode) calcd for C₂₆H₂₅N+H⁺ 352.2060, found 352.2054.



Yellow oil (113 mg), 62% yield. ¹H NMR (300MHZ, CDCl₃) δ 0.85 (t, J = 7.5 Hz, 3H), 1.01 (t, J = 7.5 Hz, 3H), 1.92 (d, J = 6.5 Hz, 2H), 2.02 (d, J = 6.5 Hz, 2H), 2.24 (t, J = 7.5 Hz, 2H), 2.64 (t, J = 7.5 Hz, 2H), 7.11-7.20 (m, 7H), 7.33-7.42 (m, 6H), 7.67-7.68 (m, 2H); ¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 13.4, 23.4, 25.3, 32.8, 33.7, 119.8, 125.6, 127.8, 128.0, 128.2, 128.4, 128.5, 128.6, 129.0, 130.0, 137.5, 140.0, 143.1, 144.1, 164.7. GC-MS found for 367; HRMS (ESI mode) calcd for C₂₇H₂₉N+H⁺368.2373, found 368.2376.



Yellow oil (83 mg), 55% yield. ¹H NMR (300MHZ,CDCl₃) δ 0.84 (t, *J* = 7.5 Hz, 3H), 1.01 (t, *J* = 7.5 Hz, 3H), 1.87-2.02 (m, 4H), 2.75 (d, *J* = 6.5 Hz, 2H) 4.91-4.99 (m, 2H), 5.60-5.74 (m, 1H), 7.22-7.24 (m, 2H), 7.33-7.39 (m, 6H); 7.66-7.68 (m, 2H). ¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 13.2, 13.3, 23.1, 25.1, 35.6, 115.1, 118.7, 127.9, 128.0, 128.3, 128.7, 128.9, 130.0, 137.0, 137.5, 140.0, 144.2, 165.0. GC-MS found for 303; HRMS (ESI mode) calcd for C₂₂H₂₅N+H⁺ 304.2060, found 304.2056.



Yellow oil (170 mg), 85% yield. ¹H NMR (300MHZ,CDCl₃) δ 3.27 (d, J = 6.5 Hz, 2H), 4.92-5.04 (m, 2H), 5.68-5.79 (m, 1H), 6.70-6.72 (m, 2H), 6.84-6.97 (m, 7H),

7.01-7.04 (m, 2H), 7.16-7.24 (m, 5H), 7.36-7.42 (m,2H), 7.78-7.80 (m, 2H); 13 C NMR (75MHz, CDCl₃, Me₄Si) δ 38.4, 115.9, 122.2, 125.8, 126.1, 126.3, 127.2, 127.7, 127.8 128.2, 128.3, 128.5, 129.1 129.6, 130.0, 130.6, 135.9, 137.7, 139.3, 140.5, 141.4, 145.1, 168.3. GC-MS found for 399; HRMS (ESI mode) calcd for C₃₀H₂₅N+H⁺ 400.2060, found 400.2065.



Yellow oil (98 mg), 48% yield. ¹H NMR (300MHz, CDCl₃, Me₄Si) δ 0.89 (t, *J* = 7.2 Hz, 3H), 1.03 (t, *J* = 7.2 Hz, 3H), 2.04 (q, *J* = 7.2 Hz, 2H), 2.23 (q, *J* = 7.2 Hz, 2H), 6.74-6.82 (m, 3H), 7.00-7.07 (m, 3H), 7.16-7.37 (m, 5H), 7.60-7.63 (m, 2H); ¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 12.1, 13.4, 23.0, 23.5, 126.2, 127.5, 128.0, 128.4, 128.8, 129.2, 130.0, 133.3, 134.4, 136.0, 136.6, 137.8, 140.4, 141.5, 164.9. GC-MS found for 407; HRMS (ESI mode) calcd for C₂₅H₂₃Cl₂N+H⁺ 408.1280, found 408.1277.



Yellow oil (76 mg), 47% yield. ¹H NMR (300MHz, CDCl₃,) δ 0.91 (t, *J* = 7.5 Hz, 3H), 2.25 (s, 3H), 2.38 (q, *J* = 7.5 Hz, 2H), 7.03-7.21 (m, 9H), 7.42-7.45 (m, 4H), 7.95-7.98 (m, 2H); ¹³C NMR (75MHz, CDCl₃,) δ 12.1, 18.3, 26.2, 124.0, 125.9, 126.5, 127.2, 128.0, 128.4, 128.9 129.6, 130.2, 130.4, 139.3, 139.6, 141.7, 143.8, 165.3. GC-MS found for 325; HRMS (ESI mode) calcd for C₂₄H₂₃N+H⁺ 326.1903, found 326.1904.



Yellow oil (76 mg), 55% yield. ¹H NMR (300MHz, CDCl₃) & 0.88-0.98 (m, 6H), 1.96

(q, J = 7.5 Hz, 2H), 2.09-2.15 (m, 5H), 7.11-7.29 (m, 9H), 7.77-7.81 (m, 1H); ¹³C NMR (75MHz, CDCl₃) δ 12.1, 13.8, 18.0, 22.6, 23.7, 124.0, 126.7, 127.0, 128.0, 128.3, 129.0, 130.0, 139.8, 139.9, 142.1, 164.5. GC-MS found or 277; HRMS (ESI mode) calcd for C₂₀H₂₃N+H⁺ 278.1903, found 278.1900.



Yellow oil (69 mg), 57% yield. ¹H NMR (300MHz, CDCl₃,) δ 1.00-1.06 (m, 6H), 2.11-2.17 (m, 5H), 2.28 (q, *J* = 7.5 Hz, 2H), 2.58 (d, *J* = 7.5 Hz, 2H), 4.89-4.94 (m, 2H), 5.65-5.71 (m, 1H), 7.37-7.39 (m, 3H), 7.87-7.88 (m, 2H); ¹³C NMR (75MHz, CDCl₃) δ 12.9, 13.8, 17.9, 23.3, 25.0, 35.2, 115.0, 115.8, 126.9, 128.3, 130.0, 136.8, 139.8, 144.2, 163.7. GC-MS found for 241; HRMS (ESI mode) calcd for C₁₇H₂₃N+H⁺ 242.1903, found242.1908.



Yellow oil (118 mg), 70% yield. ¹H NMR (300MHz, CDCl₃,) δ 2.20 (s, 3H), 3.16 (d, J = 7.5 Hz, 2H), 4.91-5.02 (m, 2H), 5.74-5.79 (m, 1H), 7.04-7.18 (m, 10H), 7.40-7.43(m, 3H), 7.94-7.96 (m, 2H); ¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 18.9, 38.5, 115.6, 120.1, 126.0, 126.8, 127.2, 127.7, 127.9, 128.4, 129.6, 130.1, 130.5, 136.1, 139.2, 139.5, 141.9, 145.2, 165.9. GC-MS found for 337; HRMS (ESI mode) calcd for C₂₅H₂₃N+H⁺ 338.1903, found338.1909.

$$\begin{array}{c} Ph & Et \\ \hline Ph & Et \\ Ph & 3cg \end{array}$$

Yellow oil (59 mg), 45% yield. ¹H NMR (300MHz, CDCl₃,) δ 0.95 (t, *J* = 7.5 Hz, 3H), 1.18 (t, *J* = 7.5 Hz, 3H), 2.00 (q, *J* = 7.5 Hz, 2H), 2.81 (q, *J* = 7.5 Hz, 2H), 7.11-7.14 (m, 2H), 7.31-7.45 (m, 6H), 7.59 (s, 1H), 7.70-7.74 (m, 2H); ¹³C NMR (75MHz, CDCl₃) δ 13.3, 14.0, 22.8, 25.6, 127.3, 128.2, 128.6, 130.1, 130.3, 132.9, 137.5, 137.7, 143.5, 145.3, 155.0. GC-MS found for 263; HRMS (ESI mode) calcd for C₁₉H₂₁N+H⁺ 264.1747, found 264.1742.



3ah

3ch

Yellow oil (80 mg), 49% yield. ¹H NMR (300MHz, CDCl₃) δ 1.09 (t, *J* = 7.5 Hz, 3H), 2.32 (s, 3H), 3.12 (q, *J* = 7.5 Hz, 2H), 6.98-7.00 (m, 2H), 7.05 (m, 2H), 7.09-7.17 (m, 4H), 7.23-7.38 (m, 4H), 7.65-7.67 (m, 2H), 7.91 (s, 1H); ¹³C NMR (75MHz, CDCl₃) δ 13.6, 21.7, 26.2, 126.0, 127.6, 128.2, 128.6, 129.5, 129.6, 131.2, 131.8, 134.8, 137.6, 141.0, 141.5, 141.9, 145.3, 157.5. GC-MS found for 325; HRMS (ESI mode) calcd for C₂₄H₂₃N+H⁺ 326.1903, found 326.1903.



Yellow oil (69 mg), 50% yield. ¹H NMR (300MHz, CDCl₃, Me₄Si) δ 0.94 (t, *J* = 7.2 Hz, 3H), 1.16 (q, *J* = 7.2 Hz, 3H), 1.98 (q, *J* = 7.2 Hz, 2H), 2.34(s, 3H), 2.80 (q, *J* = 7.2 Hz, 2H), 7.10-7.16 (m, 4H), 7.31-7.42 (m, 2H), 7.56-7.60 (m, 3H); ¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 13.3, 13.9, 21.6, 22.7, 25.5, 127.1, 128.2, 128.4, 129.0, 130.8, 135.1, 137.6, 140.3, 143.5, 144.5, 155.0. GC-MS found for 277; HRMS (ESI mode) calcd for C₂₀H₂₃N+H⁺ 278.1903, found 278.1903.



3hh

Yellow oil (64 mg), 38% yield. ¹H NMR (300MHz, CDCl₃,) δ 2.38 (s, 3H), 3.83 (d, *J* = 7.5 Hz, 2H), 4.98-5.14(m, 2H), 5.91-5.95 (m, 1H), 7.02-7.08(m, 6H), 7.13-7.22 (m, 6H), 7.67-7.69 (m, 2H), 7.93 (s, 1H);¹³C NMR (75MHz, CDCl₃) δ 21.6, 37.9, 115.3, 126.1, 127.0, 127.5, 128.0, 128.6, 129.4, 129.5, 131.0, 134.5, 136.3, 137.3, 137.4, 141.2, 141.8, 146.3, 158.0. GC-MS found for 337; HRMS (ESI mode) calcd for C₂₅H₂₃N+H⁺ 338.1903, found 338.1901.



Yellow oil (90 mg), 52% yield. ¹H NMR (300MHz, CDCl_{3.}) δ 0.98 (t, *J* = 7.5 Hz, 3H), 3.02 (q, *J* = 7.5 Hz, 2H), 6.86-7.04 (m, 6H), 7.17-7.27 (m, 6H), 7.53-7.55 (m, 2H), 7.91 (s, 1H); ¹³C NMR (75MHz, CDCl₃) δ 13.7, 26.2, 126.3, 127.7, 128.2, 128.6, 129.0, 129.5, 129.7, 131.1, 131.7, 135.9, 136.5, 137.2, 141.6, 143.3, 145.0, 155.8. GC-MS found or 345; HRMS (ESI mode) calcd for C₂₃H₂₀ClN+H⁺ 346.1357, found 346.1352.



Yellow oil (96 mg), 65% yield. ¹H NMR (300MHz, CDCl₃, Me₄Si) δ 0.94 (t, *J* = 7.2 Hz, 3H), 1.16 (t, *J* = 7.2 Hz, 3H), 1.99 (q, *J* = 7.2 Hz, 2H), 2.34(s, 3H), 2.80 (q, *J* = 7.2 Hz, 2H), 7.08-7.10 (m, 2H), 7.30-7.42 (m, 4H), 7.51 (s, 1H), 7.62-7.64 (m, 2H); ¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 13.2, 13.9, 22.8, 25.6, 127.3, 128.6, 128.8, 129.0, 129.3, 135.8, 136.2, 137.2, 143.3, 146.1, 153.4. GC-MS found for 297; HRMS (ESI mode) calcd for C₁₉H₂₀ClN+H⁺ 298.1357, found 298.1360.



3hi

Yellow oil (80 mg), 45% yield. ¹H NMR (300MHz, CDCl₃,) δ 3.85 (d, *J* = 7.5 Hz, 2H), 4.99-5.14 (m, 2H), 5.90-5.97 (m, 1H), 7.00-7.09 (m, 6H), 7.18-7.21 (m, 4H), 7.36-7.38 (m, 2H), 7.71-7.73 (m, 2H), 7.89 (s, 1H);¹³C NMR (75MHz, CDCl₃, Me₄Si) δ 37.9, 115.5, 126.3,127.2, 127.5, 128.2, 128.9, 129.4, 129.7, 131.0, 135.6, 136.6, 137.0, 137.1, 138.1, 141.5, 146.0, 156.3. GC-MS found for 357; HRMS (ESI mode) calcd for C₂₄H₂₀ClN+H⁺ 358.1357, found 358.1361.

References:

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NOESY spectra of 3hc



Copies of 1H and 13C NMR Spectra





¹H NMR and ¹³C NMR for compound **3ba**



¹H NMR and ¹³C NMR for compound **3ca**



¹H NMR and ¹³C NMR for compound **3da**



¹H NMR and ¹³C NMR for compound **3ha**



¹H NMR and ¹³C NMR for compound **3ga**



¹H NMR and ¹³C NMR for compound **3ea**



¹H NMR and ¹³C NMR for compound **3fa**



¹H NMR and ¹³C NMR for compound **3ac**



¹H NMR and ¹³C NMR for compound **3gc**

Electronic Supplementary Material (ESI) for Chemical Communications This journal is The Royal Society of Chemistry 2013



¹H NMR and ¹³C NMR for compound **3cc**



¹H NMR and ¹³C NMR for compound **3cg**



¹H NMR and ¹³C NMR for compound **3hc**



¹H NMR and ¹³C NMR for compound **3ch**



¹H NMR and ¹³C NMR for compound **3ah**

Electronic Supplementary Material (ESI) for Chemical Communications This journal is The Royal Society of Chemistry 2013



¹H NMR and ¹³C NMR for compound **3ai**



¹H NMR and ¹³C NMR for compound **3bi**



¹H NMR and ¹³C NMR for compound **3cb**



¹H NMR and ¹³C NMR for compound **3hh**



¹H NMR and ¹³C NMR for compound **3hi**