Synthesis of Vinylogous Carbamates by Rhodium(II)-Catalysed Olefination of Tertiary Formamides with a Silylated Diazoester

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Supporting information

Ethyl (Z)-3-(8-benzyl-2,8-diazabicyclo[3.3.0]octan-2-yl)-2-(triethylsilyloxy)acrylate (6)

8-Benzyl-2,8-diazabicyclo[3.3.0]octane-2-carbaldehyde¹ (**1**, 94 mg, 0.4 mmol) and rhodium(II) acetate dimer (18 mg, 41 μ mol) were dissolved in benzene (1 mL) and heated to reflux. A solution of ethyl diazo(triethylsilyl)acetate² (**2**, 112 mg, 0.5 mmol) in benzene (1 mL) was added dropwise and the resulting solution was stirred under reflux for 1 hour.

The solution was cooled to room temperature and concentrated *in vacuo*. Flash chromatography (Al₂O₃; ethyl acetate: petroleum ether 40-60 1:32) gave alkene **6** (69 mg, 39%) as a colourless oil.

$\nu_{\text{max}}/\text{cm}^{-1}$ (CHCl₃ cast) 3391br, 3063w, 3028w, 2953s, 2910m, 2874s, 2806w, 1693s, 1635s, 1366s, 1273, 1117; δ_{H} (500 MHz; CDCl₃) 7.33-7.22 (5H, m), 6.87 (1H, s), 4.50 (1H,

d, J 6.6 Hz), 4.14 (2H, q, J 7.2 Hz), 3.95 (1H, ddd, J 11.3, 8.1, 1.9 Hz), 3.88 (1H, d, J 13.4 Hz), 3.71 (1H, d, J 13.3 Hz), 3.47 (1H, ddd, J 11.3, 10.6, 6.3 Hz), 2.84-2.77 (1H, m), 2.64 (1H, dt, J 9.1, 7.0 Hz), 2.54 (1H, ddd, J 9.2, 7.3, 5.4 Hz), 2.05-2.01 (1H, m), 1.88 (1H, ddt, J 12.4, 10.8, 8.2 Hz), 1.67 (1H, ddt, J 12.5, 6.2, 1.7 Hz), 1.50 (1H, ddt, J 12.6, 7.1, 5.3 Hz), 1.26 (3H, t, J 7.1 Hz), 1.01 (9H, t, J 7.8 Hz), 0.76 (6H, q, J 7.7 Hz); δ_{C} (125 MHz; CDCl_3) 167.2 (C), 139.0 (C), 132.0 (CH), 128.6 (CH), 128.2 (CH), 126.9 (CH), 118.0 (C), 87.3 (CH), 59.6 (CH_2), 55.8 (CH_2), 51.3 (CH_2), 48.4 (CH_2), 40.6 (CH), 32.6 (CH_2), 30.2 (CH_2), 14.6 (CH_3), 7.0 (CH_3), 5.5 (CH_2); m/z (FAB) 431 (MH^+ , 100%), 338 (55), 172 (40). HRMS calculated for $\text{C}_{24}\text{H}_{39}\text{N}_2\text{O}_3\text{Si}$ (MH^+) 431.2730, observed 431.2723.

General procedure for preparation of formamides: 1,4-Dioxa-8-azaspiro[4.5]decane-8-carbaldehyde (7c)

Formamides which were not available from commercial sources (**7c**, **7f**, **7g**, **7j**, **7k** and **7l**) were prepared by a modification of the method of Moffat.³

1,4-Dioxa-8-azaspiro[4.5]decane (0.50 g, 3.5 mmol) and ethyl formate (5.0 mL, 62 mmol) were stirred under reflux overnight. Excess ethyl formate and ethanol were removed *in vacuo* to afford formamide **7c** as a pale brown oil (0.59 g, 99%) which was used without further purification. $\nu_{\text{max}}/\text{cm}^{-1}$ (film) 2880, 1674; δ_{H} (300 MHz; CDCl_3) 8.03 (1H, s), 3.98 (4H, s), 3.64-3.60 (2H, m), 3.47-3.43 (2H, m), 1.74-1.66 (4H, m); δ_{C} (75 MHz; CDCl_3) 160.8 (CH), 107.2 (C), 64.6 (CH_2), 43.8 (CH_2), 37.5 (CH_2), 35.7 (CH_2), 34.3 (CH_2); m/z (CI+) 172 (MH^+ , 100%), 121 (28). HRMS calculated for $\text{C}_8\text{H}_{13}\text{NO}_3$ (MH^+) 172.0974, observed 172.0980.

1-Formyl-4-benzhydrylpiperazine (7f)

Pink solid; δ_{H} (300 MHz; CDCl_3) 7.99 (1H, s), 7.43-7.40 (4H, m), 7.31-7.26 (4H, m), 7.23-7.17 (2H, m), 4.27 (1H, s), 3.57-3.54 (2H, m), 3.37-3.34 (2H, m), 2.41-2.36 (4H, m); δ_{C} (75 MHz; CDCl_3) 160.8 (CH), 142.0 (C), 128.7 (CH), 127.8 (CH), 127.3 (CH), 75.9 (CH), 52.3 (CH_2), 51.1 (CH_2), 45.9 (CH_2), 40.2 (CH_2).

1-Formyl-4-(3-chlorophenyl)piperazine (7g)

Pale brown oil; $\nu_{\max}/\text{cm}^{-1}$ (film) 2829, 1649; δ_{H} (300 MHz; CDCl_3) 8.10 (1H, s), 7.22-7.16 (1H, m), 6.89-6.86 (2H, m), 6.81-6.78 (1H, m), 3.71-3.68 (2H, m), 3.54-3.51 (2H, m), 3.21-3.11 (4H, m); δ_{C} (75 MHz; CDCl_3) 160.8 (CH), 152.0 (C), 135.1 (C), 130.2 (CH), 120.6 (CH), 116.9 (CH), 115.0 (CH), 50.0 (CH_2), 49.0 (CH_2), 45.4 (CH_2), 39.8 (CH_2); m/z (CI^+) 225 (MH^+ , 100%), 197 (44), 166 (31). HRMS calculated for $\text{C}_{11}\text{H}_{13}\text{ClN}_2\text{O}$ (MH^+) 225.0795, observed 225.0796.

The spectroscopic properties of the following formamides corresponded with those previously reported: *N*-allyl-*N*-benzylformamide (**7j**);⁴ *N*-formylsarcosine ethyl ester (**7k**);⁵ *N*-benzyl-*N*-phenylformamide (**7l**).⁶

General procedure for olefination of formamides: Ethyl (Z)-3-(pyrrolidin-1-yl)-2-(triethylsilyloxy)acrylate (8a)

N-Formylpyrrolidine (**7a**, 0.10 g, 1.0 mmol) and rhodium(II) acetate (2 mg, 4 μmol) were dissolved in benzene (3 mL) and heated to reflux. A solution of ethyl diazo(triethylsilyl)acetate (0.29 g, 1.25 mmol) in benzene (3 mL) was added and the mixture stirred under reflux for a further 90 minutes. After cooling to room temperature, concentration of the reaction mixture *in vacuo* and column chromatography on alumina (ethyl acetate:petroleum ether 0:100–3:97) afforded ethyl (Z)-3-(pyrrolidin-1-yl)-2-(triethylsilyloxy)acrylate (**8a**, 0.24 g, 80%) as a colourless oil; $\nu_{\max}/\text{cm}^{-1}$ (film) 2953, 2876, 1693, 1639; δ_{H} (300 MHz; CDCl_3) 6.86 (1H, s), 4.13 (2H, q, J 7.1 Hz), 3.48-3.43 (4H, m), 1.84-1.80 (4H, m), 1.26 (3H, t, J 7.1 Hz), 0.96 (9H, t, J 7.8 Hz), 0.71 (6H, q, J 7.8 Hz); δ_{C} (75 MHz; CDCl_3) 167.2 (C), 131.7 (CH), 117.8 (C), 59.5 (CH_2), 50.6 (CH_2), 25.4 (CH_2), 14.6 (CH_3), 7.0 (CH_3), 5.3 (CH_2); m/z (CI^+) 300 (MH^+ , 36%), 270 (100); HRMS calculated for $\text{C}_{15}\text{H}_{30}\text{NO}_3\text{Si}$ (MH^+) 300.1995, observed 300.1984; Analysis calc. for $\text{C}_{15}\text{H}_{29}\text{NO}_3\text{Si}$: C 60.2, H 9.8, N 4.7; found: C 60.0, H 9.9, N 5.0%.

Ethyl (Z)-3-(piperidin-1-yl)-2-(triethylsilyloxy)acrylate (8b)

Colourless oil; ν_{max} / cm^{-1} (film) 2937, 2876, 1693, 1639; δ_{H} (300 MHz; CDCl_3) 6.61 (1H, s), 4.14 (2H, q, J 7.1 Hz), 3.41-3.40 (4H, m), 1.62-1.58 (6H, m), 1.25 (3H, t, J 7.1 Hz), 0.97 (9H, t, J 7.8 Hz), 0.71 (6H, q, J 7.8 Hz); δ_{C} (75 MHz; CDCl_3) 167.4 (C), 133.3 (CH), 117.3 (C), 59.7 (CH_2), 51.1 (CH_2), 26.2 (CH_2), 24.4 (CH_2), 14.6 (CH_3), 6.9 (CH_3), 5.3 (CH_2); m/z (CI^+) 314 (MH^+ , 84%), 284 (100), 268 (50), 256 (27); HRMS calculated for $\text{C}_{16}\text{H}_{32}\text{NO}_3\text{Si}$ (MH^+) 314.2151, observed 314.2137; Analysis calc. for $\text{C}_{16}\text{H}_{31}\text{NO}_3\text{Si}$: C 61.3, H 10.0, N 4.5; found: C 61.2, H 10.2, N 4.1%.

Ethyl (Z)-3-(1,4-dioxo-8-azaspiro[4.5]dec-8-yl)-2-(triethylsilyloxy)acrylate (8c)

Colourless oil; ν_{max} / cm^{-1} (film) 2953, 2876, 1693, 1639; δ_{H} (300 MHz; CDCl_3) 6.60 (1H, s), 4.14 (2H, q, J 7.1 Hz), 3.97 (4H, s), 3.54-3.50 (4H, m), 1.72-1.69 (4H, m), 1.25 (3H, t, J 7.1 Hz), 0.96 (9H, t, J 7.9 Hz), 0.70 (6H, q, J 7.9 Hz); δ_{C} (75 MHz; CDCl_3) 167.1 (C), 132.3 (CH), 118.4 (C), 106.8 (C), 64.4 (CH_2), 59.8 (CH_2), 48.1 (CH_2), 35.3 (CH_2), 14.6 (CH_3), 6.9 (CH_3), 5.3 (CH_2); m/z (CI^+) 372 (MH^+ , 33%), 342 (41), 172 (100), 144 (39); HRMS calculated for $\text{C}_{18}\text{H}_{34}\text{NO}_5\text{Si}$ (MH^+) 372.2206, observed 372.2194; Analysis calc. for $\text{C}_{18}\text{H}_{33}\text{NO}_5\text{Si}$: C 58.2, H 9.0, N 3.8; found: C 57.9, H 9.1, N 3.7%.

Ethyl (Z)-3-(morpholin-4-yl)-2-(triethylsilyloxy)acrylate (8d)

Colourless oil; ν_{max} / cm^{-1} (film) 2957, 2876, 1699, 1643; δ_{H} (300 MHz; CDCl_3) 6.54 (1H, s), 4.15 (2H, q, J 7.1 Hz), 3.69-3.66 (4H, m), 3.43-3.40 (4H, m), 1.27 (3H, t, J 7.1 Hz), 0.96 (9H, t, J 7.9 Hz), 0.71 (6H, q, J 7.9 Hz); δ_{C} (75 MHz; CDCl_3) 166.9 (C), 132.2 (CH), 119.6 (C), 66.8 (CH_2), 60.0 (CH_2), 50.2 (CH_2), 14.6 (CH_3), 6.9 (CH_3), 5.3 (CH_2); m/z (CI^+) 316 (MH^+ , 86%), 286 (100), 88 (25); HRMS calculated for $\text{C}_{15}\text{H}_{30}\text{NO}_4\text{Si}$ (MH^+) 316.1944, observed 316.1940.

Ethyl (Z)-3-(4-methylpiperazin-1-yl)-2-(triethylsilyloxy)acrylate (8e)

Colourless oil; $\nu_{\max}$ / cm^{-1} (film) 2953, 2876, 1693, 1643; δ_{H} (300 MHz; CDCl_3) 6.57 (1H, s), 4.13 (2H, q, J 7.1 Hz), 3.45 (4H, t, J 5.0 Hz), 2.39 (4H, t, J 5.0 Hz), 2.28 (3H, s), 1.26 (3H, t, J 7.1 Hz), 0.96 (9H, t, J 7.9 Hz), 0.70 (6H, q, J 7.9 Hz); δ_{C} (75 MHz; CDCl_3) 167.1 (C), 132.4 (CH), 118.7 (C), 59.9 (CH_2), 55.0 (CH_2), 50.6 (CH_2), 46.2 (CH_3), 14.6 (CH_3), 6.9 (CH_3), 5.3 (CH_2); m/z (CI^+) 329 (MH^+ , 100%), 299 (100), 283 (34), 103 (24); HRMS calculated for $\text{C}_{16}\text{H}_{33}\text{N}_2\text{O}_3\text{Si}$ (MH^+) 329.2260, observed 329.2249.

Ethyl (Z)-3-(4-benzhydrylpiperazin-1-yl)-2-(triethylsilyloxy)acrylate (8f)

White solid; m.p. 64–66 °C; $\nu_{\max}$ / cm^{-1} (KBr disc) 2953, 1688, 1632; δ_{H} (300 MHz; CDCl_3) 7.41 (4H, d, J 7.1 Hz), 7.30–7.26 (4H, m), 7.21–7.16 (2H, m), 6.57 (1H, s), 4.22 (1H, s), 4.14 (2H, q, J 7.1 Hz), 3.46–3.43 (4H, m), 2.40–2.37 (4H, m), 1.26 (3H, t, J 7.1 Hz), 0.94 (9H, t, J 8.0 Hz), 0.67 (6H, q, J 8.0 Hz); δ_{C} (75 MHz; CDCl_3) 167.1 (C), 142.4 (C), 132.6 (CH), 128.6 (CH), 127.9 (CH), 127.1 (CH), 118.5 (C), 77.5 (CH), 59.8 (CH_2), 52.0 (CH_2), 50.1 (CH_2), 14.6 (CH_3), 6.9 (CH_3), 5.2 (CH_2); m/z (CI^+) 481 (MH^+ , 68%), 451 (100), 167 (64); HRMS calculated for $\text{C}_{28}\text{H}_{41}\text{N}_2\text{O}_3\text{Si}$ (MH^+) 481.2886, observed 481.2898; Analysis calc. for $\text{C}_{28}\text{H}_{40}\text{N}_2\text{O}_3\text{Si}$: C 70.0, H 8.4, N 5.8; found: C 69.8, H 8.6, N 5.5%.

Ethyl (Z)-3-[4-(3-chlorophenyl)piperazin-1-yl]-2-(triethylsilyloxy)acrylate (8g)

Colourless oil; $\nu_{\max}$ / cm^{-1} (film) 2954, 2876, 1692, 1676, 1643, 1593; δ_{H} (300 MHz; CDCl_3) 7.17 (1H, t, J 8.1 Hz), 6.91–6.77 (3H, m), 6.61 (1H, s), 4.16 (2H, q, J 7.1 Hz), 3.59–3.56 (4H, m), 3.18–3.15 (4H, m), 1.28 (3H, t, J 7.1 Hz), 0.99 (9H, t, J 7.9 Hz), 0.74 (6H, q, J 7.9 Hz); δ_{C} (75 MHz; CDCl_3) 166.9 (C), 152.3 (C), 135.0 (C), 131.9 (CH), 130.1 (CH), 120.0 (CH), 120.0 (C), 116.3 (CH), 114.4 (CH), 60.0 (CH_2), 49.6 (CH_2), 49.1 (CH_2), 14.6 (CH_3), 6.9 (CH_3), 5.3 (CH_2); m/z (CI^+) 425/427 (MH^+ , 85/39%), 395 (100), 345 (52), 225 (29), 197 (39), 161 (31), 115 (45), 87 (51); HRMS calculated for $\text{C}_{21}\text{H}_{34}\text{ClN}_2\text{O}_3\text{Si}$ (MH^+) 425.2027, observed 425.2045.

Ethyl (Z)-3-dimethylamino-2-(triethylsilyloxy)acrylate (8h)

Colourless oil; $\nu_{\max}$ / cm^{-1} (film) 2953, 2876, 1699, 1643; δ_{H} (300 MHz; CDCl_3) 6.61 (1H, s), 4.13 (2H, q, J 7.1 Hz), 2.97 (6H, s), 1.25 (3H, t, J 7.1 Hz), 0.96 (9H, t, J 7.8 Hz), 0.70 (6H, q, J 7.8 Hz); δ_{C} (75 MHz; CDCl_3) 167.3 (C), 134.3 (CH), 117.7 (C), 59.7 (CH_2), 42.0 (CH_3), 14.6 (CH_3), 6.9 (CH_3), 5.3 (CH_2); m/z (CI^+) 274 (MH^+ , 82%), 244 (100), 228 (67), 119 (21), 91 (43), 74 (23); HRMS calculated for $\text{C}_{13}\text{H}_{28}\text{NO}_3\text{Si}$ (MH^+) 274.1842, observed 274.1842.

Ethyl (Z)-3-(diallylamino)-2-(triethylsilyloxy)acrylate (8i)

Colourless oil; $\nu_{\max}$ / cm^{-1} (film) 2955, 2876, 1693, 1636; δ_{H} (400 MHz; CDCl_3) 6.66 (1H, s), 5.77 (2H, ddt, J 16.6, 10.7, 5.9 Hz), 5.17-5.12 (4H, m), 4.14 (2H, q, J 7.1 Hz), 3.86 (4H, d, J 5.8 Hz), 1.27 (3H, t, J 7.1 Hz), 0.95 (9H, t, J 7.9 Hz), 0.71 (6H, q, J 7.9 Hz); δ_{C} (75 MHz; CDCl_3) 167.2 (C), 134.3 (CH), 132.1 (CH), 118.0 (C), 117.2 (CH_2), 59.8 (CH_2), 53.7 (CH_2), 14.6 (CH_3), 6.9 (CH_3), 5.4 (CH_2); m/z (CI^+) 326 (MH^+ , 42%), 296 (100); HRMS calculated for $\text{C}_{17}\text{H}_{32}\text{NO}_3\text{Si}$ (MH^+) 326.2151, observed 326.2141.

Ethyl (Z)-3-(N-allyl-N-benzylamino)-2-(triethylsilyloxy)acrylate (8j)

Colourless oil; $\nu_{\max}$ / cm^{-1} (film) 2953, 2876, 1699, 1634; δ_{H} (400 MHz; CDCl_3) 7.35-7.23 (5H, m), 6.80 (1H, s), 5.78 (1H, ddt, J 16.8, 10.5, 6.0 Hz), 5.17 (1H, dq, J 10.2, 1.4 Hz), 5.13 (1H, dq, J 17.1, 1.5 Hz), 4.51 (2H, s), 4.16 (2H, q, J 7.1 Hz), 3.79 (2H, d, J 5.8 Hz), 1.28 (3H, t, J 7.1 Hz), 0.93 (9H, t, J 7.9 Hz), 0.68 (6H, q, J 7.9 Hz); δ_{C} (75 MHz; CDCl_3) 167.2 (C), 138.1 (C), 134.0 (CH), 132.5 (CH), 128.5 (CH), 127.8 (CH), 127.3 (CH), 117.9 (C), 117.6 (CH_2), 59.8 (CH_2), 54.2 (CH_2), 53.7 (CH_2), 14.6 (CH_3), 6.9 (CH_3), 5.4 (CH_2); m/z (CI^+) 376 (MH^+ , 57%), 346 (57), 330 (34), 91 (63), 59 (35), 41 (100); HRMS calculated for $\text{C}_{21}\text{H}_{34}\text{NO}_3\text{Si}$ (MH^+) 376.2308, observed 376.2302.

Diethyl (Z)-4-aza-4-methyl-2-(triethylsilyloxy)hex-2-enedioate (8k)

Colourless oil; $\nu_{\max}$ / cm^{-1} (film) 2955, 2876, 1749, 1697, 1651; δ_{H} (300 MHz; CDCl_3) 6.64 (1H, s), 4.22-4.09 (6H, m), 3.00 (3H, s), 1.29-1.23 (6H, m), 0.95 (9H, t, J 7.9 Hz), 0.70 (6H,

q, J 7.9 Hz); δ_C (75 MHz; $CDCl_3$) 169.8 (C), 166.9 (C), 132.5 (CH), 119.2 (C), 61.0 (CH_2), 60.0 (CH_2), 54.4 (CH_2), 42.1 (CH_3), 14.6 (CH_3), 14.2 (CH_3), 6.9 (CH_3), 5.3 (CH_2); m/z (CI+) 346 (MH^+ , 49%), 316 (100), 121 (25); HRMS calculated for $C_{16}H_{32}NO_5Si$ (MH^+) 346.2050, observed 346.2032; Analysis calc. for $C_{16}H_{31}NO_5Si$: C 55.7, H 9.0, N 4.1; found: C 55.8, H 9.2, N 4.1%.

Ethyl (Z)-3-(benzylphenylamino)-2-(triethylsilyloxy)acrylate (8I)

Colourless oil; ν_{max} / cm^{-1} (film) 2953, 2876, 1703, 1643; δ_H (300 MHz; $CDCl_3$) 7.33-7.20 (8H, m), 6.98-6.93 (3H, m), 5.18 (2H, s), 4.22 (2H, q, J 7.1 Hz), 1.32 (3H, t, J 7.1 Hz), 0.87 (9H, t, J 7.9 Hz), 0.60 (6H, q, J 7.9 Hz); δ_C (75 MHz; $CDCl_3$) 166.5 (C), 146.3 (C), 138.6 (CH), 128.9 (CH), 128.6 (CH), 126.9 (CH), 126.3 (CH), 126.2 (CH), 125.2 (C), 121.7 (CH), 118.0 (CH), 60.5 (CH_2), 52.9 (CH_2), 14.5 (CH_3), 6.8 (CH_3), 5.3 (CH_2); m/z (CI+) 412 (MH^+ , 63%), 392 (100), 91 (39); HRMS calculated for $C_{34}H_{34}NO_3Si$ (MH^+) 412.2308, observed 412.2309.

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¹H NMR spectrum of **6** (500 MHz; CDCl₃)

¹³C NMR spectrum of **6** (125 MHz; CDCl₃)

¹H NMR spectrum of **8a** (300 MHz; CDCl₃)

¹³C NMR spectrum of **8a** (75 MHz; CDCl₃)

¹H NMR spectrum of **8b** (300 MHz; CDCl₃)

¹³C NMR spectrum of **8b** (75 MHz; CDCl₃)

¹H NMR spectrum of **8c** (300 MHz; CDCl₃)

¹³C NMR spectrum of **8c** (75 MHz; CDCl₃)

¹H NMR spectrum of **8d** (300 MHz; CDCl₃)

¹³C NMR spectrum of **8d** (75 MHz; CDCl₃)

¹H NMR spectrum of **8e** (300 MHz; CDCl₃)

¹³C NMR spectrum of **8e** (75 MHz; CDCl₃)

¹H NMR spectrum of **8f** (300 MHz; CDCl₃)

¹³C NMR spectrum of **8f** (75 MHz; CDCl₃)

¹H NMR spectrum of **8g** (300 MHz; CDCl₃)

¹³C NMR spectrum of **8g** (75 MHz; CDCl₃)

CC(C)=C(C(=O)OCC)OSi(C)(C)CC

¹³C NMR spectrum of **8h** (75 MHz; CDCl₃)

¹H NMR spectrum of **8i** (400 MHz; CDCl₃)

¹³C NMR spectrum of **8i** (75 MHz; CDCl₃)

¹H NMR spectrum of **8j** (400 MHz; CDCl₃)

¹³C NMR spectrum of **8j** (75 MHz; CDCl₃)

¹H NMR spectrum of **8k** (300 MHz; CDCl₃)

CCOC(=O)CN(C)C=C(OSi(C)(C)C)C(=O)OCC

¹H NMR spectrum of **8l** (300 MHz; CDCl₃)

¹³C NMR spectrum of **8l** (75 MHz; CDCl₃)