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

Gurdeep S. Nandra,† Pui Shan Pang,† Michael J. Porter,*† and Jason M. Elliott.§

†Department of Chemistry, University College London, Christopher Ingold Building, 20 Gordon Street, London, WC1H 0AJ, U.K.;
§Merck Sharp & Dohme Research Laboratories, The Neuroscience Research Centre, Terlings Park, Eastwick Road, Harlow, Essex, CM20 2QR, U.K.

Supporting information

Ethyl (Z)-3-(8-benzyl-2,8-diazabicyclo[3.3.0]octan-2-yl)-2-(triethylicsilyloxy) 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 µmol) 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.

ν max/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, q, J 7.7 Hz); δC (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 (CH2), 55.8 (CH2), 51.3 (CH2), 48.4 (CH2), 40.6 (CH), 32.6 (CH2), 30.2 (CH2), 14.6 (CH3), 7.0 (CH3), 5.5 (CH2); m/z (FAB) 431 (MH+, 100%), 338 (55), 172 (40). HRMS calculated for C24H39N2O3Si (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. νmax/cm⁻¹ (film) 2880, 1674; δH (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); δC (75 MHz; CDCl 3) 160.8 (CH), 107.2 (C), 64.6 (CH2), 43.8 (CH2), 37.5 (CH2), 35.7 (CH2), 34.3 (CH2); m/z (Cl+) 172 (MH+, 100%), 121 (28). HRMS calculated for C9H13NO3 (MH+) 172.0974, observed 172.0980.

**1-Formyl-4-benzhydrylpiperazine (7f)**

Pink solid; δH (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); δC (75 MHz; CDCl 3) 160.8 (CH), 142.0 (C), 128.7 (CH), 127.8 (CH), 127.3 (CH), 75.9 (CH), 52.3 (CH2), 51.1 (CH2), 45.9 (CH2), 40.2 (CH2).
1-Formyl-4-(3-chlorophenyl)piperazine (7g)
Pale brown oil; \( \nu_{\text{max}}/\text{cm}^{-1} \) (film) 2829, 1649; \( \delta_H \) (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); \( \delta_C \) (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\(_2\)); \text{m/z} \ (\text{CI}+) 225 \ (\text{MH}^+ \ 100\%), 197 \ (44), 166 \ (31). HRMS calculated for C\(_{11}\)H\(_{13}\)ClN\(_2\)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 \((Z)-3-(pyrrolidin-1-yl)-2-(triethylsilyloxy)acrylate (8a)\)**
\( N \)-Formylypyrrolidine (7a, 0.10 g, 1.0 mmol) and rhodium(II) acetate (2 mg, 4 \( \mu \)mol) 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 \textit{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; \( \nu_{\text{max}} / \text{cm}^{-1} \) (film) 2953, 2876, 1693, 1639; \( \delta_H \) (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); \( \delta_C \) (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\(_2\)); \text{m/z} \ (\text{Cl}+) 300 \ (\text{MH}^+ \ 36\%), 270 \ (100); HRMS calculated for C\(_{15}\)H\(_{30}\)NO\(_3\)Si (MH\(^+\)) 300.1995, observed 300.1984; Analysis calc. for C\(_{15}\)H\(_{29}\)NO\(_3\)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; $v_{\text{max}}$ / cm$^{-1}$ (film) 2937, 2876, 1693, 1639; $\delta_{\text{H}}$ (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); $\delta_{\text{C}}$ (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 C$_{16}$H$_{32}$NO$_3$Si (MH$^+$) 314.2151, observed 314.2137; Analysis calc. for C$_{16}$H$_{31}$NO$_3$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-dioxa-8-azaspiro[4.5]dec-8-yl)-2-(triethylsilyloxy)acrylate (8c)**

Colourless oil; $v_{\text{max}}$ / cm$^{-1}$ (film) 2953, 2876, 1693, 1639; $\delta_{\text{H}}$ (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); $\delta_{\text{C}}$ (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 C$_{18}$H$_{34}$NO$_5$Si (MH$^+$) 372.2206, observed 372.2194; Analysis calc. for C$_{18}$H$_{33}$NO$_5$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; $v_{\text{max}}$ / cm$^{-1}$ (film) 2957, 2876, 1699, 1643; $\delta_{\text{H}}$ (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); $\delta_{\text{C}}$ (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$^+$4, 86%), 286 (100), 88 (25); HRMS calculated for C$_{18}$H$_{30}$NO$_4$Si (MH$^+$) 316.1944, observed 316.1940.
**Ethyl (Z)-3-(4-methylpiperazin-1-yl)-2-(triethylsilyloxy)acrylate (8e)**

Colourless oil; $v_{\text{max}}$ / cm$^{-1}$ (film) 2953, 2876, 1693, 1643; $\delta_H$ (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); $\delta_C$ (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 C$_{16}$H$_{33}$N$_2$O$_3$Si (MH$^+$) 329.2260, observed 329.2249.

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

White solid; m.p. 64–66 ºC; $v_{\text{max}}$ / cm$^{-1}$ (KBr disc) 2953, 1688, 1632; $\delta_H$ (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); $\delta_C$ (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 C$_{28}$H$_{41}$N$_2$O$_3$Si (MH$^+$) 481.2886, observed 481.2898; Analysis calc. for C$_{28}$H$_{40}$N$_2$O$_3$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; $v_{\text{max}}$ / cm$^{-1}$ (film) 2954, 2876, 1692, 1676, 1643, 1593; $\delta_H$ (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); $\delta_C$ (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 C$_{21}$H$_{34}$ClN$_2$O$_3$Si (MH$^+$) 425.2027, observed 425.2045.
Ethyl (Z)-3-dimethylamino-2-(triethylsilyloxy)acrylate (8h)

Colourless oil; \( \nu_{\text{max}} / \text{cm}^{-1} \) (film) 2953, 2876, 1699, 1643; \( \delta_{\text{H}} \) (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); \( \delta_{\text{C}} \) (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 C\(_{13}\)H\(_{28}\)NO\(_3\)Si (MH\(^+\)) 274.1842, observed 274.1842.

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

Colourless oil; \( \nu_{\text{max}} / \text{cm}^{-1} \) (film) 2955, 2876, 1693, 1636; \( \delta_{\text{H}} \) (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); \( \delta_{\text{C}} \) (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 C\(_{17}\)H\(_{32}\)NO\(_3\)Si (MH\(^+\)) 326.2151, observed 326.2141.

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

Colourless oil; \( \nu_{\text{max}} / \text{cm}^{-1} \) (film) 2953, 2876, 1699, 1634; \( \delta_{\text{H}} \) (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); \( \delta_{\text{C}} \) (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 C\(_{21}\)H\(_{34}\)NO\(_3\)Si (MH\(^+\)) 376.2308, observed 376.2302.

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

Colourless oil; \( \nu_{\text{max}} / \text{cm}^{-1} \) (film) 2955, 2876, 1749, 1697, 1651; \( \delta_{\text{H}} \) (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, 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); \( \delta_{\text{C}} \) (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 C\(_{13}\)H\(_{28}\)NO\(_3\)Si (MH\(^+\)) 274.1842, observed 274.1842.
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 (Cl+) 346 (MH$^+$, 49%), 316 (100), 121 (25); HRMS calculated for C$_{16}$H$_{32}$NO$_5$Si (MH$^+$) 346.2050, observed 346.2032; Analysis calc. for C$_{16}$H$_{31}$NO$_5$Si: 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 (8l)**

Colourless oil; $\nu_{max}$ / 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 (Cl+) 412 (MH$^+$, 63%), 392 (100), 91 (39); HRMS calculated for C$_{34}$H$_{34}$NO$_3$Si (MH$^+$) 412.2308, observed 412.2309.

$^1$H NMR spectrum of 6 (500 MHz; CDCl$_3$)
$^{13}$C NMR spectrum of 6 (125 MHz; CDCl$_3$)
$^{1}\text{H NMR spectrum of } \text{8a (300 MHz, CDCl}_3\text{)}$
$^{13}$C NMR spectrum of 8a (75 MHz; CDCl$_3$)
$^1$H NMR spectrum of 8b (300 MHz; CDCl$_3$)

\[ \text{Diagram of chemical structure} \]
$^{13}$C NMR spectrum of 8b (75 MHz, CDCl$_3$)
$^{1}$H NMR spectrum of 8c (300 MHz; CDCl$_3$)
$^{13}$C NMR spectrum of 8c (75 MHz; CDCl$_3$)
$^1$H NMR spectrum of 8d (300 MHz; CDCl₃)
$^{13}$C NMR spectrum of 8d (75 MHz; CDCl$_3$)
$^1$H NMR spectrum of 8e (300 MHz; CDCl$_3$)
$^1$C NMR spectrum of 8e (75 MHz; CDCl$_3$)
$^1$H NMR spectrum of 8f (300 MHz; CDCl$_3$)
$^{13}$C NMR spectrum of 8f (75 MHz, CDCl$_3$)
$^1$H NMR spectrum of 8g (300 MHz; CDCl$_3$)
$^{13}\text{C NMR spectrum of 8g (75 MHz; CDCl}_3\)$
$^1$H NMR spectrum of 8h (300 MHz, CDCl$_3$)

- CO$_2$EtN
- OSiEt$_3$
$^{13}$C NMR spectrum of 8h (75 MHz; CDCl$_3$)
$^1$H NMR spectrum of 8i (400 MHz, CDCl$_3$)
$^1$C NMR spectrum of 8 (75 MHz, CDCl$_3$)

\[ \text{OSiEt}_3 \text{CO}_2\text{Et} \]
$^1$H NMR spectrum of 8j (400 MHz, CDCl$_3$)
$^{13}$C NMR spectrum of 8j (75 MHz; CDCl₃)
$^1$H NMR spectrum of 8k (300 MHz; CDCl$_3$)
$^{13}$C NMR spectrum of 8k (75 MHz; CDCl$_3$)
$^1$H NMR spectrum of 8l (300 MHz, CDCl$_3$)

Ph

$\text{OSiEt}_3$

Ph

$\text{N}^+\text{CO}_2\text{Et}$
$^{13}$C NMR spectrum of 8 (75 MHz; CDCl$_3$)

Ph
\[ \text{OSiEt}_3 \]
\[ \text{Ph} \]
\[ \text{N} \]
\[ \text{CO}_2\text{Et} \]