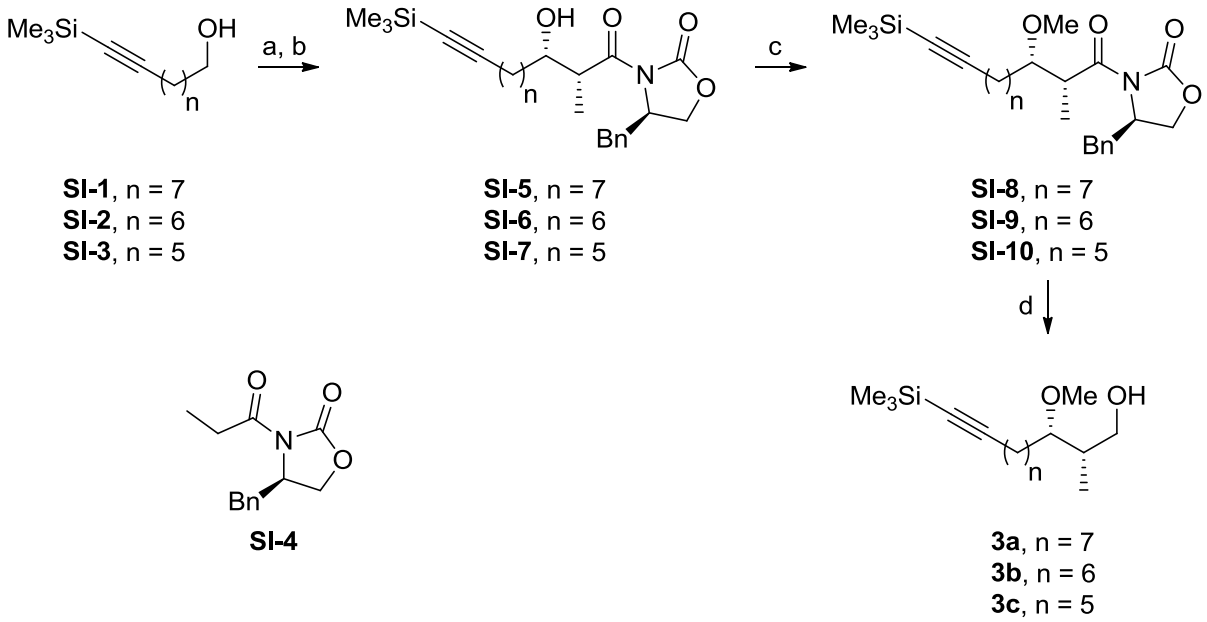
SUPPORTING INFORMATION

Daniel J. Tetlow, Steve J. Winder and Christophe Aïssa
aissa@liverpool.ac.uk
s.winder@sheffield.ac.uk

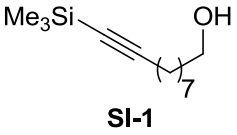
General. Otherwise noted, all reactions were carried out in flame-dried glassware under dry nitrogen atmosphere. The solvents were purified with the solvent purification system Pure Solv MD-6 (THF, Et_2O, CH_2Cl_2, benzene, toluene, hexane). Dry acetone was purchased from VWR. Flash chromatography: Merck silica gel 60 (230-400 mesh). NMR: Spectra were recorded on a Bruker DRX 500 and a Bruker DPX 400 spectrometers in $CDCl_3$; chemical shifts (δ) are given in ppm. The solvent signals were used as references and the chemical shifts converted to the TMS scale ($CDCl_3$: δ_C = 77.0 ppm; residual $CHCl_3$ in $CDCl_3$: δ_H = 7.26 ppm); apparent splitting patterns are designated using the following abbreviations: s (singlet), d (doublet), t (triplet), q (quartet), quint. (quintuplet), m (multiplet), br (broad), and the appropriate combinations. IR: PerkinElmer Spectrum 100 FT-IR spectrometer, wavenumbers ($\tilde{\nu}$) in cm^{-1}. HRMS determined at the University of Liverpool on micromass LCT mass spectrometer (ES+) and Trio-1000 or Agilent QTOF 7200 mass spectrometers (CI). Melting points: Griffin melting point apparatus (not corrected). Elemental analyses: University of Liverpool. Optical rotations were measured on a PerkinElmer Model 343 plus polarimeter with a sodium lamp (D line, 589 nm) at ambient temperature (indicated in ºC as superscript) using a 1 mL quartz cell of 100 mm length; solution concentration (c) are given in g/100 mL. All commercially available compounds were used as received.

Preparation of compounds 3a–3c



[a] $(COCl)_2$, DMSO, Et_3N, CH_2Cl_2. [b] i) **SI-4**, Et_3N, Bu_2BOTf, CH_2Cl_2; ii) **SI-1**–**SI-3**; iii) pH 7-buffer, H_2O_2, MeOH. [c] $Me_3O{\bullet}BF_4$, proton sponge, CH_2Cl_2. [d] $LiAlH_4$, Et_2O.

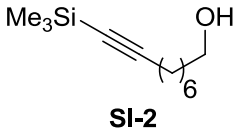
Compound SI-1. This compound was prepared according to a literature procedure.[1] To ethylenediamine (50 mL) at 0 °C under N_2 was added NaH (2.07 g, 51.9 mmol, 60% in oil) portionwise. The reaction mixture was stirred at 0 °C for 15 minutes then at room temperature for 1 hour. The purple/brown reaction mixture was heated at 60 °C for 1 hour, cooled to 40 °C and 3-decyn-1-ol (2 g, 13.0 mmol) was added. The reaction was stirred at 40 °C for 1.5 hours then at 65 °C for 2 hours. The reaction mixture was cooled to 0 °C and quenched by addition of H_2O (50 mL) and 1M HCl (50 mL). The reaction was extracted with Et_2O (50 mL) and EtOAc (50 mL), the combined organics were washed with brine, dried (Na_2SO_4) and the solvent removed under reduced pressure. Purification by flash chromatography (SiO_2, petroleum ether/Et_2O, 4:1 to 2:1) afforded 9-decyn-1-ol as a pale yellow oil (1.42 g, 71%). Some of this material (1.3 g, 8.44 mmol) was diluted in THF (42 mL) under N_2 at 0 °C and MeMgBr (7.8 mL, 23.6 mmol, 3M in Et_2O) was added dropwise. The reaction was warmed to ambient temperature and stirred for 16 hours. TMSCl (3.0 mL, 23.6 mmol) was added and the reaction stirred for 8 hours. 1M HCl (50 mL) was added carefully and the reaction stirred for 15 minutes. The mixture was extracted with Et_2O (2 x 100 mL), the combined organics dried (Na_2SO_4) and the solvent removed under reduced pressure. Purification by flash column chromatography (SiO_2, petroleum ether:Et_2O, 4:1) yielded **SI-1** as a pale yellow oil (960 mg, 50%). ^{1}H-NMR ($CDCl_3$, 500 MHz) δ 3.63 (t, *J* = 6.6 Hz, 2H), 2.18 (td, *J* = 7.2 and 2.7 Hz, 2H), 1.94 (t, *J* = 2.7 Hz, 1H), 1.59-1.49 (m, 4H) and 1.43-1.29 (m, 8H), 0.17 (s, 9H), in agreement with previously published data.[2]



SI-1

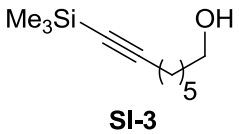
[1] C. Melander. *Org. Biomol. Chem.*, 2011**, 9**, 3041
[2] J. Robertson and J. N. Burrows, *Synthesis*, 1998, 63.

Compound SI-2. 8-nonyl-1-ol was prepared analogously from 3-nonyl-1-ol and **SI-2** (pale yellow oil, 55% over 2 steps) was then obtained following the same procedure as the one used for the preparation of **SI-1**. ^{1}H-NMR ($CDCl_3$, 500 MHz) δ 3.66-3.60 (m, 2H), 2.21 (t, J = 7.1 Hz, 2H), 1.59-1.48 (m, 4H), 1.42-1.30 (m, 6H) and 0.13 (s, 9H), in agreement with previously published data. [3]



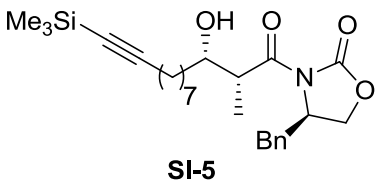
SI-2

Compound SI-3. 7-octyn-1-ol (964 mg, 71%) was prepared analogously from 3-octyn-1-ol and **SI-3** (pale yellow oil, 85%) was then obtained following the same procedure as the one used for the preparation of **SI-1**.^{1}H-NMR ($CDCl_3$, 500 MHz) δ 3.67 (q, J = 6.8 Hz, 2H), 2.25 (t, J = 7.1 Hz, 2H), 1.63-1.52 (m, 4H), 1.47-1.37 (m, 4H), 1.27-1.23 (m, 1H), 0.17 (s, 9H) in agreement with previously published data. [4]



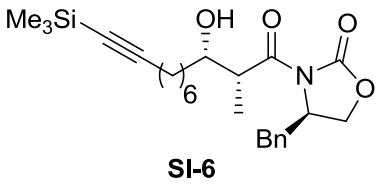
SI-3

Compound SI-5. This compound was prepared from **SI-1** (344 mg, 1.29 mmol) using the same procedure as described for the preparation of **SI-7**. Colourless oil (442 mg, 75% over 2 steps). ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 7.38-7.34 (m, 2H), 7.33-7.30 (m, 1H), 7.24-7.21 (m, 2H), 4.73 (ddt, J = 9.4, 7.8 and 2.9 Hz, 1H), 4.25 (dd, J = 9.1 and 7.7 Hz, 1H), 4.21 (dd, J = 9.1 and 2.9 Hz, 1H), 3.99-3.95 (m, 1H), 3.77 (dq, J = 7.1 and 2.5 Hz, 1H), 3.27 (dd, J = 13.4 and 3.1 Hz, 1H), 2.89 (d, J = 2.3 Hz, 1H(OH)), 2.81 (dd, J = 13.3 and 9.5 Hz, 1H), 2.23 (t, J = 2.3 Hz, 2H), 1.60-1.47 (m, 4H), 1.46-1.30 (m, 8H), 1.28 (d, J = 7.3 Hz, 3H), 0.17 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 177.6, 153.0, 134.9, 129.4 (2C), 129.0 (2C), 127.4, 107.7, 84.2, 71.4, 66.2, 55.1, 42.1, 37.8, 33.8, 29.4, 29.0, 28.7, 28.6, 25.9, 19.8, 10.3, 0.2 (3C); IR (neat): $\tilde{\nu}$ = 3517 (br), 3029, 2931, 2856, 2172, 1778, 1696, 1604, 1497, 1481, 1455, 1383, 1350, 1289, 1247, 1208, 1109, 1075, 1048, 1014, 970, 920, 760, 701 cm^{-1}; HRMS (ES$^+$) calcd for $C_{26}H_{39}NO_4SiNa$ 480.2546, found 480.2545; $[\alpha]_D^{20} = -27.1$ (c = 0.9, $CHCl_3$).



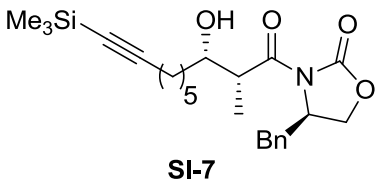
SI-5

Compound SI-6. This compound was prepared from **SI-2** (495 mg, 2.33 mmol) using the same procedure as described for the preparation of **SI-7**. Colourless oil (438 mg, 49% over two steps). ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 7.36-7.32 (m, 2H), 7.30-7.27 (m, 1H), 7.22-7.19 (m, 2H), 4.74-4.68 (m, 1H), 4.23 (dd, J = 9.0 and 7.7 Hz, 1H), 4.19 (dd, J = 9.0 and 2.9 Hz, 1H), 3.97-3.92 (m, 1H), 3.76 (qd, J = 7.0 and 2.5 Hz, 1H), 3.25 (dd, J = 13.4 and 3.2 Hz, 1H), 2.88 (br s, 1H(OH)), 2.79 (dd, J = 13.4 and 9.4 Hz, 1H), 2.21 (t, J = 7.0 Hz, 2H), 1.59-1.47 (m, 4H), 1.44-1.29 (m, 6H), 1.25 (d, J = 7.0 Hz, 3H), 0.15 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 177.5, 153.0, 135.0, 129.4 (2C), 128.9 (2C), 127.4, 107.6, 84.2, 71.4, 66.1, 55.0, 42.1, 37.8, 33.7, 29.0, 28.7, 28.5, 25.8, 19.8, 10.3, 0.1 (3C); IR (neat): $\tilde{\nu}$ = 3528 (br), 2932, 2857, 2172, 1779, 1695, 1497, 1455, 1384, 1350, 1289, 1247, 1209, 1107, 1048, 1013, 970, 842, 760, 701 cm^{-1}; HRMS (ES$^+$) calcd for $C_{25}H_{37}NO_4SiNa$ 466.2390, found 466.2387; $[\alpha]_D^{20} = -25.9$ (c = 1.25, $CHCl_3$).



SI-6

Compound SI-7. Under N_2 atmosphere, DMSO (0.96 mL, 13.47 mmol) was added via syringe to oxalylchloride (0.700 mL, 8.09 mmol) in CH_2Cl_2 (50 mL) at -78 °C. The reaction was stirred for 10 minutes and **SI-3** (1.06 g, 5.39 mmol) in CH_2Cl_2 (3 mL) was added dropwise. The reaction was stirred for 1 hour, Et_3N (3.74 mL, 26.9 mmol) was added and the reaction warmed to room temperature. Saturated aqueous NH_4Cl (30 mL) was added and the reaction mixture was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were dried (Na_2SO_4) and the solvent removed under reduced pressure. The crude aldehyde was used without further purification in the next step. Under N_2 atmosphere, Bu_2OTf (4.35 mL, 4.35

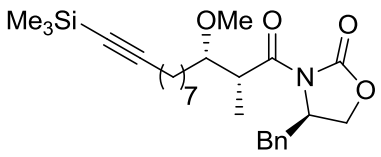


SI-7

[3] M. G. Organ and H. Ghasemi, *J. Org. Chem.*, 2004, **69**, 695.
[4] M.-J.Wu, C.-L. Lee, Y.-C. Wu and C.-P. Chen, *Eur. J. Org. Chem.* **2008**, 854

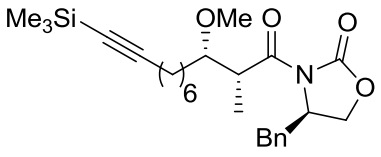
mmol, 1M in CH_2Cl_2) was added dropwise to **SI-4** (930 mg, 3.99 mmol) in CH_2Cl_2 (36 mL) at 0 °C. The reaction was stirred for 10 minutes and Et_3N (0.655 mL, 4.71 mmol) was added. The mixture was cooled to -78 °C and a solution of the aldehyde previously obtained (0.711, 3.63 mmol) in CH_2Cl_2 (2 mL) was added and the mixture was then stirred at -78 °C for 90 minutes. The reaction mixture was warmed to 0 °C and stirred for 90 minutes. A pH7 buffer (10 mL) in MeOH (30 mL) and H_2O_2 (2 mL) in MeOH (10 mL) were added and the reaction mixture stirred at 0 °C for 1 hour. The volatiles were removed under reduced pressure and the reaction extracted with Et_2O (3 × 50 mL). The combined organics were washed with saturated aqueous $NaHCO_3$ (50 mL) and brine (50 mL), dried (Na_2SO_4) and the solvent removed under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 9:1 to 4:1 to 1:1) gave **SI-7** as a colourless oil (790 mg, 51%). ^{1}H-NMR ($CDCl_3$, 500 MHz) δ 7.36-7.31 (m, 2H), 7.30-7.26 (m, 1H), 7.22-7.18 (m, 2H), 4.72-4.68 (m, 1H), 4.22 (dd, *J* = 9.1 and 7.7 Hz, 1H), 4.19 (dd, *J* = 9.1 and 3.0 Hz, 1H), 3.97-3.92 (m, 1H), 3.75 (qd, *J* = 7.1 and 2.6 Hz, 1H), 3.24 (dd, *J* = 13.4 and 3.3 Hz, 1H), 2.86 (br s, 1H(OH)), 2.79 (dd, *J* = 13.4 and 9.5 Hz, 1H), 2.21 (t, *J* = 7.1 Hz, 2H), 1.59-1.30 (m, 8H), 1.25 (d, *J* = 7.1 Hz, 3H) and 0.14 (s, 9H); ^{13}C-NMR ($CDCl_3$, 125 MHz) δ 177.6, 153.0, 135.0, 129.4 (2C), 129.0 (2C), 127.4, 107.5, 84.4, 71.4, 66.2, 55.1, 42.1, 37.8, 33.7, 28.7, 28.5, 25.6, 19.8, 10.4, 0.2 (3C); IR (neat): $\tilde{\nu}$ = 3517 (br), 3029, 2937, 2860, 2172, 1777, 1697, 1604, 1497, 1481, 1455, 1383, 1350, 1289, 1247, 1209, 1109, 1075, 1049, 1011, 977, 921, 840, 760, 700 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 452 (100 %, [M+Na]$^+$); HRMS calcd for $C_{24}H_{35}O_4NSiNa$ 452.2233, found 452.2249; $[\alpha]_D^{20} = -36.4$ (*c* = 1, $CHCl_3$).

Compound SI-8. This compound was prepared from **SI-5** (442 mg, 0.97 mmol) using the same procedure as described for the preparation of **SI-10**. Colourless oil (295 mg, 65%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 7.37-7.33 (m, 2H), 7.31-7.28 (m, 1H), 7.25-7.22 (m, 2H), 4.67 (ddt, *J* = 9.9, 6.9 and 2.8 Hz, 1H), 4.21 (dd, *J* = 9.2 and 7.5 Hz, 1H), 4.18 (dd, *J* = 9.2 and 2.9 Hz, 1H), 4.10-4.02 (m, 1H), 3.43-3.39 (m, 1H), 3.38 (s, 3H), 3.32 (dd, *J* = 13.4 and 3.0 Hz, 1H), 2.78 (dd, *J* = 13.4 and 10.1 Hz, 1H), 2.22 (t, *J* =7.2 Hz, 2H), 1.55-1.35 (m, 5H), 1.35-1.28 (m, 7H), 1.25 (d, *J* = 6.7 Hz, 3H), 0.16 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 175.4, 153.1, 135.3, 129.4 (2C), 128.9 (2C), 127.3, 107.7, 84.2, 82.7, 66.0, 58.4, 55.8, 41.0, 37.8, 31.9, 29.6, 29.0, 28.7, 28.6, 25.8, 19.8, 12.6, 0.2 (3C); IR (neat): $\tilde{\nu}$ = 2931, 2856, 2172, 1779, 1696, 1497, 1481, 1379, 1349, 1289, 1247, 1208, 1194, 1107, 1050, 1013, 966, 921, 839, 759, 700 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 494 (100 %, [M+Na]$^+$); HRMS (ES$^+$) calcd for $C_{27}H_{41}O_4NSiNa$ 494.2703, found 494.2702; $[\alpha]_D^{20} = -55.6$ (*c* = 1.3, $CHCl_3$).



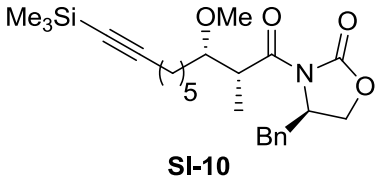
SI-8

Compound SI-9. This compound was prepared from **SI-6** (295 mg, 0.66 mmol) using the same procedure as described for the preparation of **SI-10**. Colourless oil (195 mg, 65 %). ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 7.35-7.31 (m, 2H), 7.30-7.27 (m, 1H,), 7.23-7.21 (m, 2H), 4.65 (ddt, *J* = 9.9, 7.0 and 2.9 Hz, 1H), 4.21-4.15 (m, 2H), 4.10-4.02 (m, 1H), 3.42-3.38 (m, 1H), 3.37 (s, 3H), 3.31 (dd, *J* = 13.0 and 3.1 Hz, 1H), 2.77 (dd, *J* = 13.0 and 9.8 Hz, 1H), 2.21 (t, *J* = 7.0 Hz, 2H), 1.55-1.28 (m, 10H), 1.24 (d, *J* = 7.0 Hz, 3H), 0.14 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 175.4, 153.1, 135.3, 129.4 (2C), 128.9 (2C), 127.3, 107.6, 84.3, 82.6, 65.9, 58.4, 55.7, 41.0, 37.8, 31.9, 29.2, 28.7, 28.5, 25.7, 19.8, 12.6, 0.1 (3C); IR (neat): $\tilde{\nu}$ = 2933, 2858, 2172, 1779, 1696, 1604, 1497, 1481, 1455, 1379, 1349, 1288, 1247, 1208, 1194, 1106, 1050, 1014, 966, 924, 841, 760, 701 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 480 (100 %, [M+Na]$^+$); HRMS calcd for $C_{26}H_{39}NO_4SiNa$ 480.2546, found 480.2557; $[\alpha]_D^{20} = -49.5$ (*c* = 0.85, $CHCl_3$).



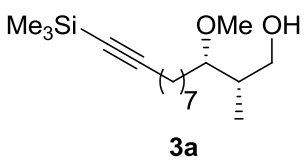
SI-9

Compound SI-10. Under N_2 atmosphere, proton sponge (850 mg, 6.96 mmol) and $Me_3O{\bullet}BF_4$ (1.02 g, 6.96 mmol) were added to **SI-7** (750 mg, 1.74 mmol) in CH_2Cl_2 (17.4 mL, 0.1M) in a flask covered with aluminium foil. The reaction was stirred in the dark at room temperature for 40 hours. The reaction mixture was diluted with CH_2Cl_2 (30 mL), washed with saturated aqueous NH_4Cl (20 mL) and brine (20 mL), dried (Na_2SO_4) and the solvent removed under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 20:1) afforded **SI-10** as a pale yellow oil (632 mg, 82%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 7.35-7.30 (m, 2H), 7.28-7.25 (m, 1H), 7.22-7.19 (m, 2H), 4.65 (ddt, *J* = 9.9, 7.1 and 2.9 Hz, 1H), 4.21-4.15 (m, 2H), 4.10-4.02 (m, 1H), 3.41 (q, *J* = 5.9 Hz, 1H), 3.39 (s, 3H), 3.30 (dd, *J* = 13.4 and 3.2 Hz, 1H), 2.76 (dd, *J* = 13.4 and 9.8 Hz, 1H), 2.21 (t, *J* = 7.2 Hz, 2H), 1.55-1.28 (m, 8H), 1.24 (d, *J* = 7.0 Hz, 3H), 0.13 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 175.4, 153.1, 135.3, 129.4 (2C), 128.9 (2C), 127.3, 107.5, 84.3, 82.7, 66.0, 58.4, 55.7, 41.0, 37.8, 31.8, 28.8, 28.6, 25.3, 19.8, 12.7, 0.2 (3C); IR (neat): $\tilde{\nu}$ = 3029, 2936, 2860, 2172, 1778, 1696, 1604, 1497, 1454, 1378, 1349, 1289, 1247, 1209, 1194, 1106, 1049, 1012, 967, 920, 840, 760, 701 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 444 (100 %, [M+H]$^+$); HRMS calcd for $C_{25}H_{37}O_4NSi$ 444.2565, found 444.2583.



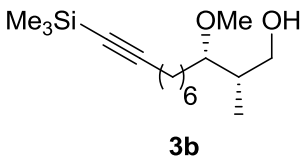
SI-10

Compound 3a. This compound was prepared from **SI-8** (215 mg, 0.456 mmol) using the same procedure as described for the preparation of **3c**. Colourless oil (109 mg, 80%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 3.69 (ddd, *J* = 10.6, 7.5 and 3.4 Hz, 1H), 3.58 (dt, *J* = 10.6 and 4.8 Hz, 1H), 3.40 (s, 3H), 3.27-3.24 (m, 1H), 3.27-3.23 (m, 1H), 2.76-2.72 (m, 1H(OH)), 2.22 (t, *J* = 7.2 Hz, 2H), 2.10-2.00 (m, 1H), 1.60-1.49 (m, 3H), 1.46-1.24 (m, 9H), 0.87 (d, *J* = 7.3 Hz, 3H), 0.16 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 107.6, 85.0, 84.2, 66.4, 57.6, 36.3, 29.6 (2C), 29.0, 28.7, 28.6, 26.2, 19.8, 11.6, 0.1 (3C); IR (neat): $\tilde{\nu}$ = 3407 (br), 2930, 2856, 2173, 1460, 1378, 1248, 1091, 1028, 841, 759, 698 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 321 (100 %, [M+Na]$^+$); HRMS (ES$^+$) calcd for $C_{17}H_{34}O_2SiNa$ 321.2226, found 321.2226; $[\alpha]_D^{20}$ = +10.2 (*c* = 0.85, $CHCl_3$).



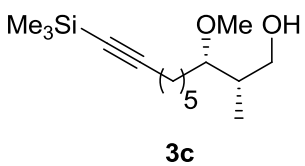
3a

Compound 3b. This compound was prepared from **SI-9** (150 mg, 0.33 mmol) using the same procedure as described for the preparation of **3c**. Colourless oil (65 mg, 69%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 3.69 (dd, *J* = 10.5 and 7.6 Hz, 1H), 3.58 (dd, *J* = 10.8 and 4.7 Hz, 1H), 3.39 (s, 3H), 3.27-3.23 (m, 1H), 2.64 (brs, 1H (OH)), 2.22 (t, *J* = 7.2 Hz, 2H), 2.07-2.00 (m, 1H), 1.59-1.48 (m, 3H), 1.46-1.36 (m, 4H), 1.36-1.24 (m, 3H), 0.86 (d, *J* = 7.0 Hz, 3H), 0.15 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 107.4, 84.9, 84.4, 66.3, 57.7, 36.4, 29.5, 28.9, 28.5, 28.3, 25.7, 19.8, 11.6, 0.1 (3C); IR (neat): $\tilde{\nu}$ = 3400 (br), 2933, 2858, 2174, 1461, 1379, 1248, 1194, 1157, 1091, 1029, 924, 839, 758, 694 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 307 (100 %, [M+Na]$^+$); HRMS calcd for $C_{16}H_{32}O_2SiNa$ 307.2069, found 307.2076; $[\alpha]_D^{20}$ = +8.0 (*c* = 0.15, $CHCl_3$).



3b

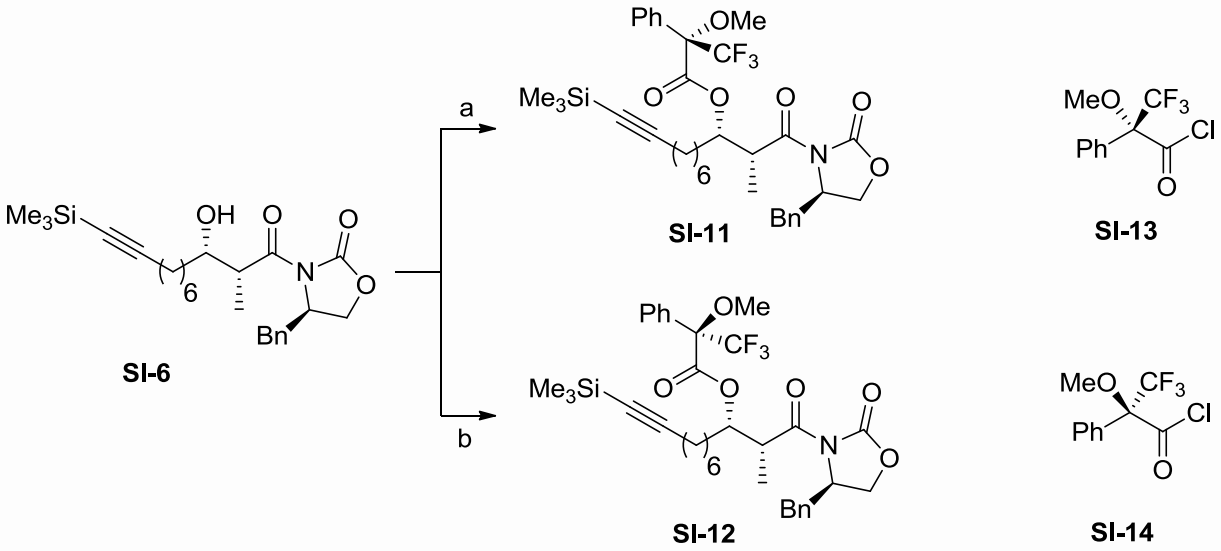
Compound 3c. Under N_2 atmosphere, **SI-10** (602 mg, 1.35 mmol) in Et_2O (1 mL) was added dropwise via cannula to a suspension of $LiAlH_4$ (26 mg, 0.68 mmol) in Et_2O (14 mL) at 0 °C. The reaction was stirred for 30 minutes at 0 °C and a second portion of $LiAlH_4$ (26 mg, 0.68 mmol) was added. The reaction was warmed slowly to room temperature and stirred for 2 hours. The reaction was diluted with Et_2O (10 mL) and saturated aqueous Na_2SO_4 was added dropwise (slowly) until a white precipitate had formed. The reaction mixture was filtered through Celite and the solvent removed under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 10:1 to 2:1) afforded **3** as a pale yellow oil (235 mg, 64%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ = 3.69 (ddd, *J* = 10.8, 7.6 and 4.7 Hz, 1H), 3.58 (ddd, *J* = 10.7, 5.7 and 4.7 Hz, 1H), 3.40 (s, 3H), 3.28-3.23 (m, 1H), 2.65-2.61 (m, 1H(OH)), 2.23 (t, *J* = 7.1 Hz, 2H), 2.09-2.01 (m, 1H), 1.60-1.50 (m, 3H), 1.47-1.37 (m, 4H), 1.35-1.27 (m, 1H), 0.86 (d, *J* = 7.2 Hz, 3H), 0.15 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 107.4, 84.9,



3c

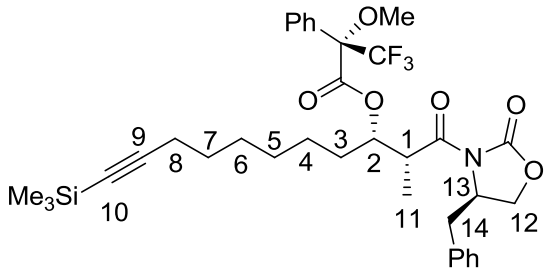
84.4, 66.3, 57.7, 36.4, 29.6, 28.9, 28.5, 25.7, 19.8, 11.6, 0.1 (3C); IR (neat): $\tilde{\nu}$ = 3391 (br), 2934, 2173, 1459, 1379, 1248, 1193, 1161, 1091, 1032, 841, 759, 697 cm^{-1}; MS (ES^+) *m/z* (rel. intensity) 293 (100 %, $[M+Na]^+$); HRMS calcd for $C_{15}H_{30}O_2SiNa$ 293.1913, found 293.1913; $[\alpha]_D^{20} = +8.2$ (*c* = 1.1, $CHCl_3$).

A Mosher ester analysis was performed with one derivative to ascertain the stereochemistry of the products.



[a] **SI-13**, pyridine, CH_2Cl_2. [b] **SI-14**, pyridine, CH_2Cl_2.

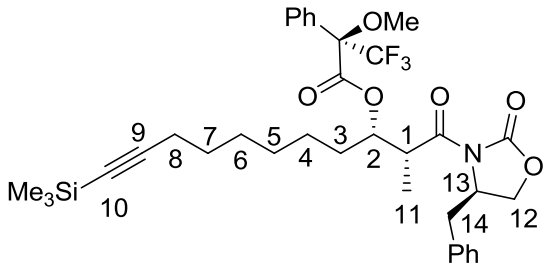
Compound SI-11. A solution of **SI-6** (35 mg, 0.072 mmol), **SI-13** (24 µL, 0.13 mmol) and pyridine (17 µL, 0.22 mmol) in CH_2Cl_2 (0.35 mL) was stirred for 12h at room temperature under N_2. The mixture was quenched with water and extracted with Et_2O (3 × 5 mL). The combined organic layers were dried (Na_2SO_4) and the solvent removed under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 20:1 to 10:1) afforded **SI-11** as a colourless oil (21 mg, 44%).[5] ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.59-7.54 (m, 2H, ArH), 7.42-7.39 (m, 3H, ArH), 7.36-7.32 (m, 2H, ArH), 7.30-7.27 (m, 1H, ArH), 7.21-7.19 (m, 2H, ArH), 5.43 (ddd, *J* = 9.0, 4.0 and 2.9 Hz, 1H, H-2), 4.57-4.52 (m, 1H, H-13), 4.33 (t, *J* = 8.2 Hz, 1H, H-14^A), 4.17 (dd, *J* = 8.9 and 2.2 Hz, 1H, H-14^B), 4.02 (qd, *J* = 7.0 and 3.4 Hz, 1H, H-1), 3.64 (s, 3H, OMe), 3.29 (dd, *J* = 13.3 and 3.4 Hz, 1H, H-12^A), 2.79 (dd, *J* = 13.4 and 9.6 Hz, 1H, H-12^B), 2.17 (t, *J* = 7.1 Hz, 2H, H-8), 1.73-1.64 (m, 1H, H-3^A), 1.59-1.50 (m, 1H, H-3^B), 1.45-1.38 (m, 2H, H-7), 1.35-1.20 (m, 5H), 1.24 (d, *J* = 7.0 Hz, 3H, H-11), 1.20-1.04 (m, 1H), 0.15 (s, 9H, $SiMe_3$).



SI-11

[5] T. R. Hoye, C. S. Jeffrey and F. Shao, *Nature Protocols.*, 2007**, 2**, 2451.

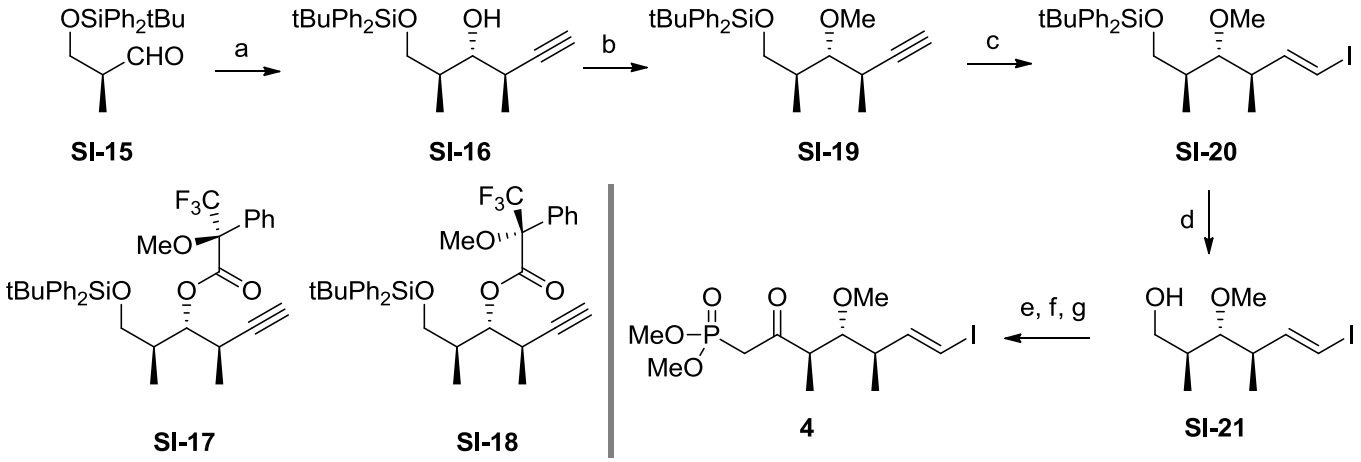
Compound SI-12. This compound was prepared from **SI-6** (25 mg, 0.050 mmol) and **SI-14** (17 µL, 0.095 mmol) using the same procedure as described for the preparation of **SI-11**. Colourless oil (16 mg, 48%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.58-7.54 (m, 2H, ArH), 7.43-7.39 (m, 3H, ArH), 7.36-7.31 (m, 2H, ArH), 7.30-7.27 (m, 1H, ArH), 7.20-7.17 (m, 2H, ArH), 5.45-5.40 (m, 1H, H-2), 4.60-4.54 (m, 1H, H-13), 4.28 (t, *J* =8.3 Hz, 1H, H-14^A), 4.17 (dd, *J* = 9.1 and 2.3 Hz, 1H, H-14^B), 4.00 (qd, *J* = 7.0 and 3.4 Hz, 1H, H-1), 3.55 (s, 3H, OMe), 3.28 (dd, *J* = 13.4 and 3.4 Hz, 1H, H-12^A), 2.76 (dd, *J* = 13.4 and 9.8 Hz, 1H, H-12^B), 2.21 (t, *J* = 7.2 Hz, 2H, H-8), 1.84-1.75 (m, 1H, H-3^A), 1.70-1.62 (m, 1H, H-3^B), 1.52-1.45 (m, 2H, H-7), 1.40-1.26 (m, 6H), 1.17 (d, *J* = 7.0 Hz, 3H, H-11), 0.15 (s, 9H, $SiMe_3$).



SI-12

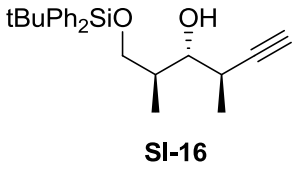
			$\Delta\delta^{SR}$ (=$\delta_{SI-11} - \delta_{SI-12}$)	
	SI-11 δ (ppm)	**SI-12** δ (ppm)	ppm	Hz
OMe	3.64	3.55	+0.09	+45
H-11	1.23	1.17	+0.06	+30
H-14^A	4.33	4.28	+0.044	+22
H-1	4.03	3.99	+0.028	+14
H-12^A	3.30	3.28	+0.018	+9
H-14^B	4.18	4.17	+0.008	+4
H-2	5.43	5.43	+0.007	+3.5
$SiMe_3$	0.15	0.15	+0.005	+2.5
H-12^B	2.79	2.79	-0.003	-1.5
H-13	4.55	4.57	-0.027	-13.5
H-8	2.17	2.21	-0.034	-17
H-7	1.42	1.48	-0.06	-30
H-3^A	1.69	1.79	-0.102	-51
H-3^B	1.54	1.67	-0.133	-66.5

Preparation of intermediate 4



[(a)] $Pd(OAc)_2$, PPh_3, (*S*)-but-3-yn-2-yl methanesulfonate, Et_2Zn, THF. [(b)] NaH, MeI, THF. [(c)] i) Cp_2ZrHCl, THF; ii) N-iodosuccinimide. [(d)] Tetrbutylammonium fluoride, THF. [(e)] Dess-Martin periodinane, $NaHCO_3$, CH_2Cl_2. [(f)] dimethyl methylphosphonate, nBuLi, THF. [(g)] Dess-Martin periodinane, $NaHCO_3$, CH_2Cl_2.

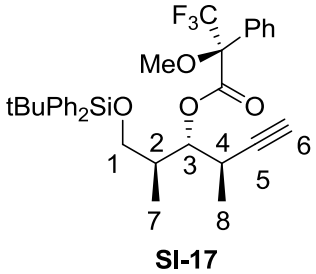
Compound SI-16. Under N_2, $Pd(OAc)_2$ (97 mg, 0.435 mmol, 5 mol%) was dissolved in degassed THF (90 mL). The reaction was cooled to -78 °C, then PPh_3 (113 mg, 0.435 mmol, 5 mol%), **SI-21**[6] (2.84 g, 8.7 mmol) in THF (5 mL) and (*S*)-but-3-yn-2-yl methanesulfonate[7] (1.54 g, 10.4 mmol) were added. Et_2Zn (26.1 mL, 26.1 mmol, 1M in hexane) was then added dropwise to the reaction mixture which was stirred for ten minutes at -78 °C before warming to -20 °C. The reaction was stirred at this temperature for 16 hours. The reaction mixture was poured into a stirred mixture of NH_4Cl (100 mL) and Et_2O (100 mL) and stirred for 30 minutes. The layers were separated and the aqueous phase washed with Et_2O (100 mL). The combined organics were washed with brine (100 mL), dried (Na_2SO_4), stirred with charcoal, and filtered through silica on a Celite plug. The solvent was removed under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 20:1 to 10:1) afforded **SI-16** as a colourless oil (2.74 g, 7.23 mmol, 83%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.73-7.70 (m, 4H), 7.49-7.40 (m, 6H), 3.80 (dd, *J* = 10.2 and 4.6 Hz, 1H), 3.72 (dd, *J* = 10.2 and 7.4 Hz, 1H), 3.53 (d, *J* = 4.6 Hz, 1H), 3.46-3.42 (m, 1H), 2.75-2.70 (m, 1H), 2.12 (d, *J* = 2.3 Hz, 1H), 2.11-2.04 (m, 2H), 1.34 (d, *J* = 7.3 Hz, 3H), 1.08 (s, 9H), 0.87 (d, *J* = 6.9 Hz, 3H), in agreement with literature data.[8] $[\alpha]_D^{20} = +20.1$ (*c* = 1.0, $CHCl_3$).[8]



SI-16

Mosher esters **SI-17** and **SI-18** of **SI-16** were prepared for confirmation of its stereochemistry.

Compound SI-17. This compound was prepared from **SI-16** (21 mg, 0.056 mmol) and **SI-13** (20 µL, 0.107 mmol) using the same procedure as described for the preparation of **SI-11**. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 50:1 to 20:1) afforded **SI-17** as a colourless oil (10.4 mg, 31%).^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.65-7.58 (m, 6H, ArH), 7.47-7.33 (m, 7H, ArH), 7.30-7.26 (m, 2H, ArH), 5.19 (dd, *J* = 7.0 and 4.8 Hz, 1H, H^3), 3.61 (dd, *J* = 10.3 and 5.2 Hz, 1H, H^{1A}), 3.51 (s, 3H, OMe), 3.45 (dd, *J* = 10.2 and 6.4 Hz, 1H, H^{1B}), 2.99 (qdd, *J* = 7.2, 4.8 and 2.5 Hz, 1H, H^4), 2.25-2.18 (m, 1H, H^2), 2.07 (d, *J* = 2.5 Hz, 1H, H^6), 1.22 (d, *J* = 7.2 Hz, 3H, H^8), 1.07 (s, 9H, ^{t}Bu),

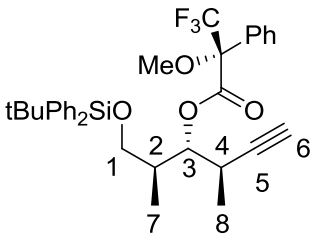


SI-17

[6] F. Arikan, J. Li and D. Menche, *Org. Lett.*, 2008, **10**, 3521.
[7] C. J. Elsevier, P. Vermeer, A Gedanken and W. Runge, *J. Org. Chem.*, 1985, **50**, 364.
[8] $[\alpha]_D^{20} = -17.0$ (*c* = 1.5, $CHCl_3$) was reported for the enantiomer of **SI-22**, see J. A. Marshall and B. A. Johns, *J. Org. Chem.*, 2000, **65**, 1501.

0.98 (d, J = 7.0 Hz, 3H, H^7).

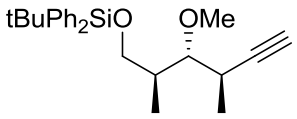
Compound SI-18. This compound was prepared from **SI-16** (17 mg, 0.045 mmol) and **SI-14** (16 µL, 0.085 mmol) using the same procedure as described for the preparation of **SI-11**. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 50:1 to 20:1) afforded **SI-18** as a colourless oil (10 mg, 37%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.66-7.63 (m, 4H, ArH), 7.57 (d, J = 7.4 Hz, 2H, ArH), 7.46-7.31 (m, 9H, ArH), 5.18 (dd, J = 6.9 and 4.7 Hz, 1H, H^3), 3.67 (dd, J = 10.4 and 5.0 Hz, 1H, H^{1A}), 3.50 (dd, J = 10.4 and 6.4 Hz, 1H, H^{1B}), 3.43 (s, 3H, OMe), 2.94 (qdd, J = 7.2, 4.7 and 2.4 Hz, 1H, H^4), 2.28-2.20 (m, 1H, H^2), 1.97 (d, J = 2.4 Hz, 1H, H^6), 1.14 (d, J = 7.2 Hz, 3H, H^8), 1.07 (s, 9H, ^{t}Bu), 1.04 (d, J = 7.0 Hz, 3H, H^7).



SI-18

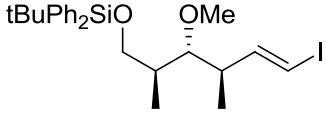
	SI-17	**SI-18**	$\Delta\delta^{SR}$ (=$\delta_{\mathbf{SI-17}}$–$\delta_{\mathbf{SI-18}}$)	
	δ (ppm)	δ (ppm)	ppm	Hz
H^6	2.07	1.97	+0.1	+47
OMe	3.51	3.43	+0.08	+40
H^8	1.22	1.14	+0.08	+36
H^4	2.99	2.94	+0.05	+26
H^3	5.19	5.18	+0.01	+9
^{t}Bu	1.07	1.07	0	0
H^2	2.22	2.24	-0.02	-13
H^{1B}	3.45	3.50	-0.05	-30
H^{1A}	3.61	3.67	-0.06	-32
H^7	0.98	1.04	-0.06	-32

Compound SI-19. Sodium hydride (560 mg, 14.0 mmol, 60% dispersion in mineral oil) was added to a solution of **SI-16** (2.65 g, 7.0 mmol) and iodomethane (5.9 mL, 42.0 mmol) in THF (70 ml) at 0 °C under N_2. The reaction was slowly warmed to room temperature and stirred for 4 hours. The reaction was carefully quenched with saturated aqueous NH_4Cl (50 mL) and extracted with EtOAc (3 × 100 mL). The combined organic layers were dried (Na_2SO_4) and stirred over charcoal before filtration and removal of the solvent under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 20:1) afforded **SI-19** as a pale yellow oil (2.40 g, 87%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.72-7.67 (m, 4H), 7.47-7.43 (m, 2H), 7.42-7.38 (m, 4H), 3.86 (dd, J = 9.9 and 4.8 Hz, 1H), 3.70 (dd, J = 9.9 and 3.6 Hz, 1H), 3.47 (s, 3H), 3.10 (dd, J = 8.5 and 3.6 Hz, 1H), 2.80-2.75 (m, 1H), 2.08 (d, J = 2.4 Hz, 1H), 2.07-1.99 (m, 2H), 1.31 (d, J = 7.0 Hz, 3H), 1.10 (m, 9H), 1.03 (d, J = 7.0 Hz, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 135.7 (2C), 135.6 (2C), 133.8, 133.7, 129.6 (2C), 127.56 (2C), 127.55 (2C), 85.7, 85.0, 69.7, 65.3, 61.0, 39.1, 29.0, 26.9 (3C), 19.3, 18.2, 14.4; IR (neat): $\tilde{\nu}$ = 3309, 3071, 2960, 2931, 2857, 1589, 1472, 1427, 1389, 1391, 1191, 1111, 1089, 1007, 936, 823, 739, 702 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 417 (100 %, $[M+Na]^+$); HRMS calcd for $C_{25}H_{34}O_2SiNa$ 417.2226, found 417.2242; $[\alpha]_D^{20} = -11.3$ (c = 1.1, $CHCl_3$).



SI-19

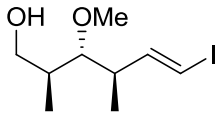
Compound SI-20. A solution of **SI-19** (2.40 g, 6.10 mmol) in THF (5 mL) was added dropwise to a stirred suspension of Cp_2ZrHCl (2.04 g, 7.94 mmol) in THF (60 mL) at 0 °C in the dark and under N_2. The reaction was stirred for 90 minutes at 0 °C. *N*-iodosuccinimide (2.73 g, 12.2 mmol) was then added in one portion and the mixture was stirred for 30 minutes. Saturated aqueous $Na_2S_2O_3$ solution (50 mL) was added and after 15 minutes stirring at room temperature, the reaction mixture was extracted with EtOAc (3 × 70 mL). The combined organic layers were washed with brine (50 mL), dried (Na_2SO_4) and the solvent removed under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 100:0 to 30:1) afforded **SI-20** as a pale yellow gum (2.29 g, 75%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 7.66-7.62 (m, 4H), 7.42-7.34 (m, 6H), 6.50 (dd, J = 14.4



SI-20

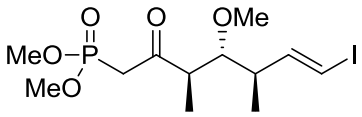
and 8.6 Hz, 1H), 5.94 (d, *J* = 14.4 Hz, 1H), 3.71 (dd, *J* = 9.9 and 5.3 Hz, 1H), 3.63 (dd, *J* = 9.9 and 4.0 Hz, 1H), 3.35 (s, 3H), 2.97 (dd, *J* = 7.8 and 3.5 Hz, 1H), 2.47-2.39 (m, 1H), 1.77-1.68 (m, 1H), 1.05-1.04 (m, 12H), 0.89 (d, *J* = 6.9 Hz, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 148.2, 135.70 (2C), 135.65 (2C), 133.8, 133.7, 129.6, 129.5, 127.6 (4C), 86.1, 74.7, 65.3, 61.1, 43.1, 38.7, 26.9 (3C), 19.3, 17.7, 14.2; IR (neat): $\tilde{\nu}$ = 3070, 3048, 2960, 2929, 2894, 2857, 1600, 1589, 1471, 1460, 1427, 1389, 1361, 1268, 1240, 1180, 1138, 1103, 1092, 1066, 1008, 971, 952, 907, 863, 822, 791, 738, 698 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 545 (100 %, $[M+Na]^+$); HRMS calcd for $C_{25}H_{35}IO_2SiNa$ 545.1349, found 545.1364; $[\alpha]_D^{20} = -1.9$ (*c* = 0.65, $CHCl_3$).

Compound SI-21. TBAF (2.0 mL, 2.04 mmol, 1M in THF) was added to **SI-20** (510 mg, 1.02 mmol) in THF (5 mL) at 0 ºC under N_2. The reaction was stirred at room temperature for 16 hours. The volatiles were removed under reduced pressure and the crude residue was purified by flash column chromatography on silica gel (petroleum ether/Et_2O, 10:1 to 4:1 to 1:1) to yield **SI-21** as a colourless oil (223 mg, 77%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.56 (dd, *J* = 14.5 and 8.4 Hz, 1H), 6.07 (d, *J* = 14.5 Hz, 1H), 3.70-3.59 (m, 2H), 3.48 (s, 3H), 2.95 (dd, *J* = 7.5 and 4.4 Hz, 1H), 2.57 (dd, *J* = 6.5 and 4.7 Hz, 1H(OH)), 2.54-2.47 (m, 1H), 1.85-1.76 (m, 1H), 1.10 (d, *J* = 7.1 Hz, 3H), 0.93 (d, *J* = 7.0 Hz, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 147.9, 89.9, 75.3, 66.1, 61.4, 43.6, 37.7, 16.9, 14.8; IR (neat): $\tilde{\nu}$ = 3397 (br), 3047, 2963, 2929, 2828, 1600, 1455, 1427, 1372, 1268, 1178, 1149, 1060, 1031, 969, 952, 905, 859, 820, 741, 702 cm^{-1}; MS (CI(NH_3)) *m/z* (rel. intensity) 285 (100 %, $[M+H]^+$); HRMS calcd for $C_9H_{17}IO_2$ 285.0346, found 285.0349; $[\alpha]_D^{20} = -11.2$ (*c* = 0.9, $CHCl_3$).



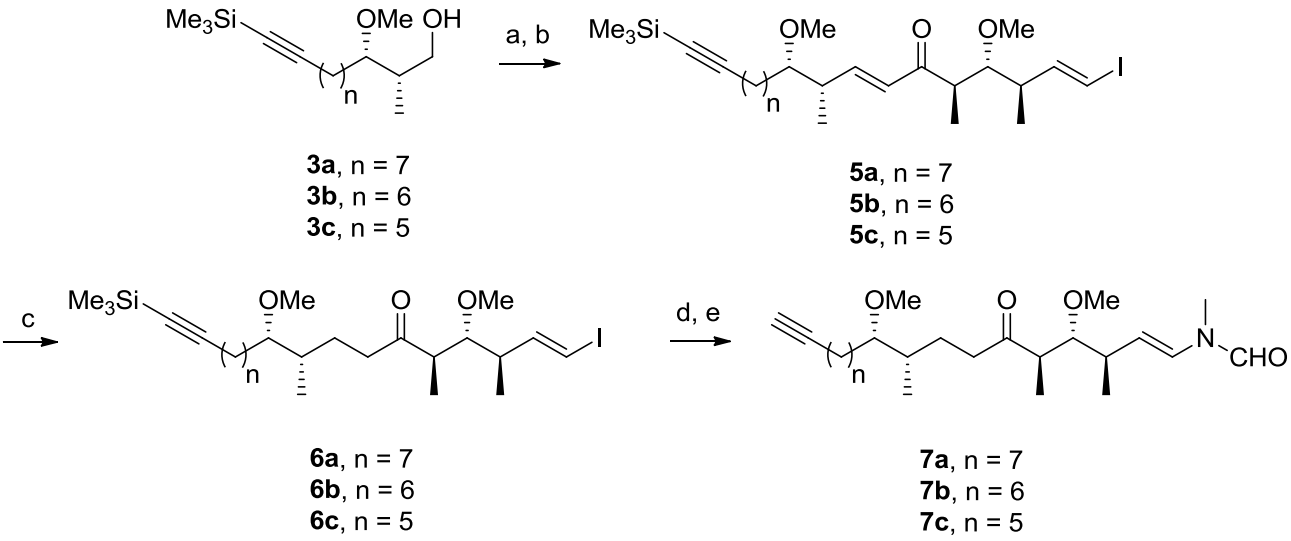
SI-21

Compound 4. Dess-Martin periodinane (958 mg, 2.94 mmol) was added in one portion to **SI-21** (642 mg, 2.26 mmol) and $NaHCO_3$ (379 mg, 4.52 mmol) in CH_2Cl_2 (23 mL, 0.1M) at 0 °C. The reaction was stirred at room temperature for 2 hours. Saturated aqueous $NaHCO_3$ (10 mL) and saturated aqueous $Na_2S_2O_3$ (10 mL) were added. After stirring for 15 minutes, the mixture was diluted with CH_2Cl_2 (30 mL), and the aqueous layer washed with CH_2Cl_2 (2 × 50 mL). The combined organic phases were dried (Na_2SO_4) and concentrated under reduced pressure. The crude aldehyde was used without further purification in the next step. A solution of ^{n}BuLi (2.4 mL, 6.10 mmol, 2.5M in hexanes) was added dropwise to freshly distilled dimethyl methylphosphonate (0.60 mL, 5.65 mmol) in THF (22 mL) at -78 °C under N_2. The cloudy solution was stirred at this temperature for 30 minutes and a solution of the aldehyde obtained previously in THF (2 mL) was added dropwise to give a yellow solution. The reaction was stirred at -78 °C for 1 hour and quenched by addition of saturated aqueous NH_4Cl (50 mL). The reaction was warmed to room temperature and extracted with EtOAc (3 × 70 mL). The combined organic layers were dried (Na_2SO_4) and the solvent removed under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 2:1 to 0:1) afforded a secondary alcohol (0.832 g, 73 % over two steps, pale yellow oil, 1:1 mixture of diastereoisomers). Dess-Martin periodinane (869 mg, 2.05 mmol) was added in one portion to a fraction of this material (800 mg, 1.58 mmol) and $NaHCO_3$ (265 mg, 3.16 mmol) in CH_2Cl_2 (16 mL) at 0 °C, as described above. After the same aqueous work-up as described above, purification by flash chromatography on silica gel (petroleum ether/EtOAc, 4:1 to EtOAc only) afforded **4** as colourless oil (701 mg, 87%). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.53 (dd, *J* = 14.5 and 9.0 Hz, 1H), 6.03 (d, *J* = 14.5 Hz, 1H), 3.80 (d, J_P = 9.0 Hz, 3H), 3.78 (d, J_P = 9.0 Hz, 3H), 3.28 (s, 3H), 3.27 (dd, *J* = 22.0 and 14.5 Hz, 1H), 3.13 (dd, *J* = 9.5 and 9.0 Hz, 1H), 3.07 (dd, *J* = 22.0 and 14.5 Hz, 1H), 2.88 (dq, *J* = 9.0 and 7.0 Hz, 1H), 2.39 (ddq, *J* = 14.3, 7.0 and 2.9 Hz, 1H), 1.08 (d, *J* = 7.0 Hz, 3H), 0.94 (d, *J* = 7.0 Hz, 3H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 205.7 (d, J_P = 7.5 Hz), 146.8, 87.5, 75.8, 61.2, 53.0 (d, J_P = 23.8 Hz), 52.9 (d, J_P = 23.1 Hz), 49.5, 43.1, 42.8 (d, J_P = 128.5 Hz), 17.2, 13.3; IR (neat): $\tilde{\nu}$ = 2929, 2850, 1712, 1601, 1456, 1374, 1351, 1255, 1179, 1088, 971, 954, 925, 880, 808, 738, 705 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 427 (100 %, $[M+Na]^+$); HRMS calcd for $C_{12}H_{22}O_5IPNa$ 427.0147, found 427.0161; $[\alpha]_D^{20} = -99.1$ (*c* = 1.14, $CHCl_3$).



4

Preparation of compounds 5a–5c, 6a–6c and 7a–7c



3a, n = 7
3b, n = 6
3c, n = 5

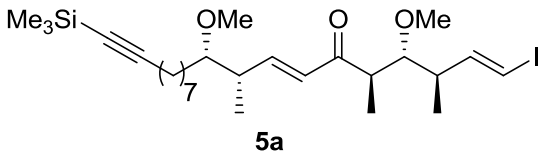
5a, n = 7
5b, n = 6
5c, n = 5

6a, n = 7
6b, n = 6
6c, n = 5

7a, n = 7
7b, n = 6
7c, n = 5

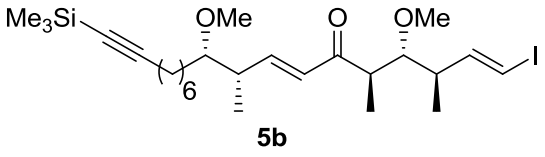
[a] Dess-Martin periodinane, $NaHCO_3$, CH_2Cl_2. [b] i) **4**, $Ba(OH)_2$, THF; ii) aldehydes, THF/water (40:1), rt. [c] $[CuHPPh_3]_6$, toluene, water. [d] CuI (0.2 equiv), 1,2-*trans*-cyclohexyldiamine (0.4 equiv), K_3PO_4 (2 equiv), MeNCHO (10 equiv), 1,4-dioxane, 80 °C. [e] $AgNO_3$ (4 equiv), THF/EtOH/water/2,6-lutidine (1:1:1:0.1), rt, 48h.

Compound 5a. This compound was prepared from **3a** (108 mg, 0.36 mmol) using the same procedure as described for the preparation of **5c**. Colourless oil (121 mg, 59% over two steps). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.88 (dd, *J* = 16.0 and 7.5 Hz, 1H), 6.60 (dd, *J* = 14.6 and 9.2 Hz, 1H), 6.16 (dd, *J* = 16.0 and 0.9 Hz, 1H), 6.07 (d, *J* = 14.6 Hz, 1H), 3.37 (s, 3H), 3.31-3.27 (m, 4H (MeO + 1H)), 3.11-3.07 (m, 1H), 2.96 (dq, *J* = 9.5 and 7.0 Hz, 1H), 2.63-2.55 (m, 1H), 2.48-2.40 (m, 1H), 2.20 (t, *J* = 7.2 Hz, 2H), 1.53-1.46 (m, 2H), 1.45-1.21 (m, 10H), 1.12 (d, *J* = 7.6 Hz, 3H), 1.07 (d, *J* = 7.1 Hz, 3H), 0.94 (d, *J* = 7.1 Hz, 3H), 0.14 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 203.2, 149.9, 147.1, 129.8, 107.6, 86.6, 84.4, 84.2, 75.4, 61.1, 57.8, 46.4, 43.1, 39.6, 31.3, 29.6, 29.0, 28.7, 28.6, 25.6, 19.8, 17.6, 14.7, 13.8, 0.1 (3C); IR (neat): $\tilde{\nu}$ = 2931, 2856, 2173, 1693, 1668, 1624, 1456, 1373, 1248, 1179, 1093, 971, 953, 841, 759, 698 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 597 (100 %, $[M+Na]^+$); HRMS (ES$^+$) calcd for $C_{27}H_{47}IO_3SiNa$ 597.2237, found 597.2235; $[\alpha]_D^{20} = -92.3$ (*c* = 1.02, $CHCl_3$).



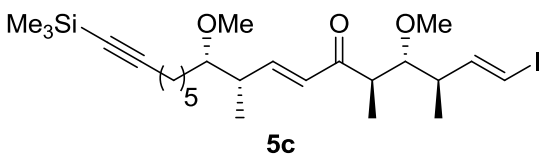
5a

Compound 5b. This compound was prepared from **3b** (65 mg, 0.23 mmol) using the same procedure as described for the preparation of **5c**. Colourless oil (79 mg, 46% over two steps). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.86 (dd, *J* = 15.9 and 7.8 Hz, 1H), 6.60 (dd, *J* = 14.7 and 9.5 Hz, 1H), 6.17 (dd, *J* = 15.9 and 1.2 Hz, 1H), 6.07 (d, *J* = 14.7 Hz, 1H), 3.37 (s, 3H), 3.30 (s, 3H), 3.30-3.27 (m, 1H), 3.12-3.07 (m, 1H), 2.96 (dq, *J* = 9.9 and 6.8 Hz, 1H), 2.63-2.56 (m, 1H), 2.45 (dqd, *J* = 9.0, 7.0 and 2.6 Hz, 1H), 2.20 (t, *J* = 7.0 Hz, 2H), 1.58-1.47 (m, 2H), 1.45-1.34 (m, 6H), 1.33-1.26 (m, 2H), 1.12 (d, *J* = 6.7 Hz, 3H), 1.08 (d, *J* = 6.7 Hz, 3H), 0.95 (d, *J* = 7.0 Hz, 3H), 0.14 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 203.1, 149.8, 147.2, 129.8, 107.6, 86.7, 84.4, 84.3, 75.4, 61.2, 57.9, 46.4, 43.2, 39.6, 31.3, 29.2, 28.7, 28.6, 25.6, 19.8, 17.6, 14.7, 13.8, 0.2 (3C); IR (neat): $\tilde{\nu}$ = 2932, 2857, 2172, 1692, 1667, 1623, 1456, 1373, 1354, 1248, 1179, 1093, 971, 953, 842, 760, 733, 697 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 583 (100 %, $[M+Na]^+$); HRMS calcd for $C_{26}H_{45}IO_3SiNa$ 583.2080, found 583.2079; $[\alpha]_D^{20} = -76.6$ (*c* = 1.33, $CHCl_3$).



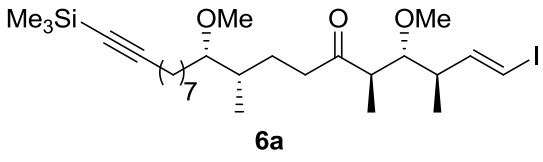
5b

Compound 5c. Dess-Martin periodinane (369 mg, 0.87 mmol) was added in one portion to **3c** (127 mg, 0.47 mmol) and $NaHCO_3$ (79 mg, 0.94 mmol) in CH_2Cl_2 (5 mL) at 0 °C. The reaction was stirred at room temperature for 2 hours. Saturated aqueous $NaHCO_3$ (10 mL) and saturated aqueous $Na_2S_2O_3$ (10 mL) were added. After stirring for 15 minutes, the mixture was diluted with CH_2Cl_2 (30 mL), and the aqueous layer washed with CH_2Cl_2 (2 × 30 mL). The combined organic phases were dried (Na_2SO_4) and concentrated under reduced pressure. The crude aldehyde thus obtained was passed through a short silica plug (eluting with 10:1 petroleum ether/Et_2O) to remove residual impurities and was used without further purification (100 mg, 0.37 mmol) in the next step. Under N_2, $Ba(OH)_2$ (70 mg, 0.41 mmol)[9] was added to **4** (150 mg, 0.37 mmol) in THF (3 mL). The mixture was stirred for 30 minutes at room temperature, before adding the aldehyde prepared above (100 mg, 0.37 mmol) as a THF/H_2O (40:1, 1 mL) solution. After stirring for 1 hour, the reaction was diluted with CH_2Cl_2 (10 mL) and saturated aqueous $NaHCO_3$ (10 mL) was added and the mixture was stirred for 15 minutes. The mixture was separated and the aqueous layer washed with CH_2Cl_2 (2 × 20 mL). The combined organic layers were dried (Na_2SO_4) and the solvent removed under reduced pressure. Purification by flash column chromatography (SiO_2, petroleum ether/Et_2O, 4:1) yielded **5c** as a colourless oil (142 mg, 55% over two steps). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.85 (dd, *J* = 15.9 and 7.6 Hz, 1H), 6.59 (dd, *J* = 14.6 and 9.3 Hz, 1H), 6.16 (dd, *J* = 15.9 and 1.0 Hz, 1H), 6.06 (d, *J* = 14.6 Hz, 1H), 3.37 (s, 3H), 3.30 (s, 3H), 3.30-3.26 (m, 1H), 3.12-3.07 (m, 1H), 2.95 (dq, *J* = 9.5 and 7.0 Hz, 1H), 2.64-2.56 (m, 1H), 2.44 (dqd, *J* = 9.3, 6.8 and 2.0 Hz, 1H), 2.20 (t, *J* = 7.2 Hz, 2H), 1.51 (quint, *J* = 7.2 Hz, 2H), 1.46-1.26 (m, 6H), 1.12 (d, *J* =7.0 Hz, 3H), 1.01 (d, *J* = 6.9 Hz, 3H), 0.94 (d, *J* = 7.0 Hz, 3H), 0.13 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 203.1, 149.7, 147.2, 129.9, 107.5, 86.6, 84.4, 84.3, 75.4, 61.2, 57.9, 46.4, 43.2, 39.6, 31.3, 28.9, 28.5, 25.2, 19.8, 17.6, 14.7, 13.8, 0.2 (3C); IR (neat): $\tilde{\nu}$ = 2934, 2830, 2172, 1692, 1667, 1623, 1456, 1373, 1353, 1248, 1179, 1093, 971, 908, 842, 759, 733, 698 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 569 (100 %, $[M+Na]^+$); HRMS calcd for $C_{25}H_{43}IO_3SiNa$ 569.1924, found 569.1938; $[\alpha]_D^{20} = -83.3$ (*c* = 1.0, $CHCl_3$).



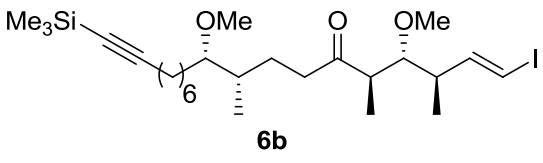
5c

Compound 6a. This compound was prepared from **5a** (97 mg, 0.17 mmol) using the same procedure as described for the preparation of **6c**. Colourless oil (68 mg, 70 %). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.57 (dd, *J* = 14.6 and 9.1 Hz, 1H), 6.04 (d, *J* = 14.6 Hz, 1H), 3.34 (s, 3H), 3.30 (s, 3H), 3.25 (dd, *J* = 9.6 and 2.4 Hz, 1H), 2.99-2.94 (m, 1H), 2.68 (dq, *J* = 9.6 and 7.0 Hz, 1H), 2.54-2.48 (m, 1H), 2.45-2.37 (m, 1H), 2.20 (t, *J* = 7.2 Hz, 2H), 1.78-1.68 (m, 1H), 1.68-1.60 (m, 1H), 1.55-1.46 (m, 2H), 1.46-1.33 (m, 6H), 1.32-1.23 (m, 6H), 1.11 (d, *J* = 6.9 Hz 3H), 0.90 (d, *J* = 7.0 Hz, 3H), 0.85 (d, *J* = 6.8 Hz, 3H), 0.14 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 214.2, 147.1, 107.8, 86.6, 85.3, 84.3, 75.4, 61.3, 57.9, 48.9, 43.1, 42.3, 34.8, 30.4, 29.8, 29.1, 28.8, 28.7, 26.1, 26.0, 19.9, 17.5, 14.7, 13.5, 0.2 (3C); IR (neat): $\tilde{\nu}$ = 2930, 2855, 2173, 1713, 1601, 1457, 1407, 1373, 1248, 1179, 1153, 1091, 969, 954, 922, 841, 759, 698 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 599 (100 %, $[M+Na]^+$); HRMS calcd for $C_{27}H_{49}IO_3SiNa$ 599.2393, found 599.2384; $[\alpha]_D^{20} = -53.1$ (*c* = 1.0, $CHCl_3$).



6a

Compound 6b. This compound was prepared from **5b** (55 mg, 0.098 mmol) using the same procedure as described for the preparation of **6c**. The material thus obtained contained approximately 12% of desilylated analogue and 10% of PPh_3 and was not fully characterised before being used in the following step (colourless oil, 47 mg, yield = 71% after correction). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.57 (dd, *J* = 14.6 and 9.1 Hz, 1H), 6.05 (d, *J* = 14.6 Hz, 1H), 3.35 (s, 3H), 3.31 (s, 3H), 3.25 (dd, *J* = 9.6

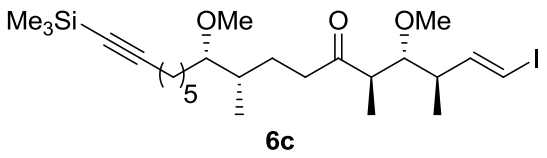


6b

[9] $Ba(OH)_2$•$8H_2O$ was dried at 120 ºC under vacuum for 2h prior to use, see I. Paterson, K.-S. Yeung and J. B. Smaill, *Synlett*, 1993, 774

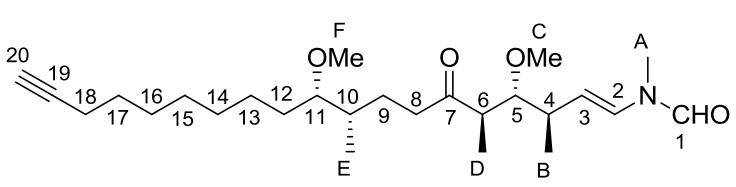
and 2.4 Hz, 1H), 3.01-2.96 (m, 1H), 2.69 (dq, *J* = 9.6 and 7.0 Hz, 1H), 2.55-2.49 (m, 1H), 2.47-2.38 (m, 1H), 2.21 (t, *J* = 7.2 Hz, 2H), 1.79-1.70 (m, 1H), 1.69-1.61 (m, 1H), 1.56-1.49 (m, 2H), 1.48 (m, 9H), 1.33-1.23 (m, 2H), 1.12 (d, *J* = 7.0 Hz, 3H), 0.91 (d, *J* = 7.2 H, 3H), 0.86 (d, *J* = 6.8 Hz, 3H), 0.14 (s, 9H); HRMS (ES^+) calcd for $C_{26}H_{47}IO_3SiNa$ 585.2237, found 585.2236.

Compound 6c. $[CuHPPh_3]_6$ (123 mg, 0.063 mmol) was added to a solution of **5c** (117 mg, 0.21 mmol) in toluene (4.3 mL, 0.05M) followed by deionised H_2O (38 µL, 2.10 mmol) was added. After stirring at room temperature for 2 hours, the reaction was filtered through deactivated neutral alumina and the solvent removed under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 100:1 to 50:1 to 40:1) afforded 6 as a colourless oil (83 mg, contains 12% of desilylated analogue; yield = 64% after correction). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.57 (dd, *J* = 14.6 and 9.1 Hz, 1H), 6.05 (d, *J* = 14.6 Hz, 1H), 3.36 (s, 3H), 3.31 (s, 3H), 3.25 (dd, *J* = 9.8 and 2.5 Hz, 1H), 3.00-2.96 (m, 1H), 2.69 (dq, *J* = 9.6 and 7.0 Hz, 1H), 2.55-2.50 (m, 1H), 2.46-2.39 (m, 1H), 2.22 (t, *J* = 6.7 Hz, 2H), 1.78-1.70 (m, 1H), 1.69-1.62 (m, 1H), 1.56-1.49 (m, 2H), 1.48 (m, 7H), 1.33-1.23 (m, 2H), 1.12 (d, *J* = 7.0 Hz, 3H), 0.91 (d, *J* = 7.2 H, 3H), 0.86 (d, *J* = 7.0 Hz, 3H), 0.15 (s, 9H); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 214.1, 147.1, 107.6, 86.6, 85.2, 84.3, 75.4, 61.2, 57.8, 48.8, 43.0, 42.3, 34.8, 30.3, 29.0, 28.6, 25.9, 25.6, 19.8, 17.4, 14.7, 13.5, 0.2 (3C); IR (neat): $\tilde{\nu}$ = 2933, 2173, 1713, 1601, 1457, 1407, 1373, 1248, 1180, 1155, 1090, 970, 954, 922, 840, 759, 697 cm^{-1}; MS (ES^+) *m/z* (rel. intensity) 571 (100 %, $[M+Na]^+$); HRMS calcd for $C_{25}H_{45}IO_3SiNa$ 571.2080, found 571.2065; $[\alpha]_D^{20} = -63.9$ (*c* = 0.9, $CHCl_3$).



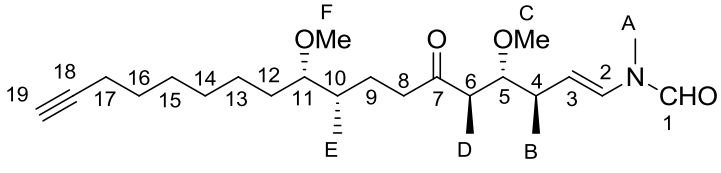
6c

Compound 7a.[10] This compound was prepared from **6a** (65 mg, 0.11 mmol) using the same procedure as described for the preparation of **7c**. Colourless oil (29 mg, 60% over 2 steps, d.r. = 90:10). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 8.27 (s, 0.65H, H^1), 8.06 (s, 0.35H, H^1), 7.12 (d, *J* = 14.7 Hz, 0.35H, H^2), 6.44 (d, *J* = 14.4 Hz, 0.65H, H^2), 5.14-5.06 (m, 1H, H^3), 3.34 (s, 6H, H^C + H^F), 3.35-3.29 (m, 1H, H^5), 3.06 (s, 1.05H, H^A), 3.03 (s, 1.95H, H^A), 2.99-2.94 (m, 1H, H^{11}), 2.68 (dq, *J* = 9.7 and 7.1 Hz, 0.65H^6), 2.65 (dq, *J* = 9.8 and 7.0 Hz, 0.35H^6), 2.56-2.46 (m, 2H, H^8), 2.45-2.34 (m, 1H, H^4), 2.16 (td, *J* = 7.0 and 2.6 Hz, 2H, H^{18}), 1.92 (t, *J* = 2.6 Hz, 1H, H^{20}), 1.79-1.70 (m, 1H, $H^{9\alpha}$), 1.69-1.61 (m, 1H, H^{10}), 1.57-1.50 (m, 2H, H^{17}), 1.47-1.25 (m, 11H, $H^{9\beta}$ + H^{12} + H^{13} + H^{14} + H^{15} + H^{16}), 1.15 (d, *J* = 6.9 Hz, 3H, H^B), 0.91 (d, *J* = 7.0 Hz, 1.8H, H^D), 0.90 (d, *J* = 7.0 Hz, 1.2H, H^D), 0.85 (d, *J* = 7.0 Hz, 1.8H, H^E), 0.84 (d, *J* = 7.0 Hz, 1.2H, H^E); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 214.3 (C^{7min}), 214.2 (C^{7maj}), 162.1 (C^{1maj}), 160.8 (C^{1min}), 128.7 (C^{2maj}), 124.7 (C^{2min}), 113.1 (C^{3min}), 111.3 (C^{3maj}), 87.4 (C^{5min}), 87.3 (C^{5maj}), 85.3 (C^{11}), 84.7 (C^{19}), 68.0 (C^{20}), 61.4 (C^{Cmin}), 61.3 (C^{Cmaj}), 57.7 (C^F), 49.1 (C^{6min}), 49.0 (C^{6maj}), 42.3 (C^{8maj}), 42.2 (C^{8min}), 37.6 (C^{4min}), 37.4 (C^{4maj}), 35.0 (C^{10min}), 34.77 (C^{10maj}), 33.1 (C^{Amin}), 30.4 (C^{12min}), 30.3 (C^{12maj}), 29.7 (C^{14}), 29.0 (C^{15}), 28.7 (C^{16}), 28.4 (C^{17}), 27.6 (C^{Amaj}), 26.06 (C^{13}), 26.03 (C^{9min}), 26.00 (C^{9maj}), 19.4 (C^{Bmaj}), 19.3 (C^{Bmin}), 18.2 (C^{18}), 14.7 (C^{Emaj}), 14.6 (C^{Emin}), 13.6 (C^{Dmin}), 13.5 (C^{Dmaj}); IR (neat): $\tilde{\nu}$ = 3265, 2930, 2856, 1692, 1653, 1457, 1371, 1318, 1274, 1194, 1155, 1090, 1069, 939, 877, 725, 686 cm^{-1}; MS (ES^+) *m/z* (rel. intensity) 458 (100 %, $[M+Na]^+$); HRMS calcd for $C_{26}H_{45}NO_4Na$ 458.3246, found 428.3239; $[\alpha]_D^{20} = -63.3$ (*c* = 1.35, $CHCl_3$).



7a

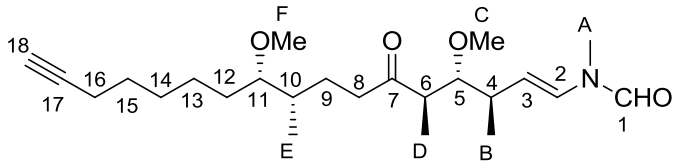
Compound 7b.[10] This compound was prepared from **6b** (46 mg, 0.082 mmol) using the same procedure as described for the preparation of **7a**. Colourless oil (19 mg, 55% over 2 steps, d.r. = 92:8). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 8.21 (s, 0.65H, H^1), 8.06 (s, 0.35H, H^1), 7.12 (d, *J* = 14.6 Hz, 0.35H, H^2), 6.44 (d, *J* = 14.3 Hz,



7b

0.65H, H^2), 5.15-5.06 (m, 1H, H^3), 3.33 (s, 6H, H^C + H^F), 3.33-3.29 (m, 1H, H^5), 3.06 (s, 1.05H, H^A), 3.03 (s, 1.95H, H^A), 3.00-2.93 (m, 1H, H^{11}), 2.68 (dq, *J* = 9.6 and 7.0 Hz, 0.65H^6), 2.65 (dq, *J* = 9.7 and 7.0 Hz, 0.35H^6), 2.56-2.47 (m, 2H, H^8), 2.47-2.34 (m, 1H, H^4), 2.18 (td, *J* = 7.0 and 2.6 Hz, 2H, H^{17}), 1.94 (t, *J* = 2.6 Hz, 1H, H^{19}), 1.80-1.70 (m, 1H, $H^{9\alpha}$), 1.69-1.61 (m, 1H, H^{10}), 1.58-1.50 (m, 2H, H^{16}), 1.47-1.25 (m, 9H, $H^{9\beta}$ + H^{12} + H^{13} + H^{14} + H^{15}), 1.15 (d, *J* = 7.0 Hz, 3H, H^B), 0.92 (d, *J* = 7.0 Hz, 1.8H, H^D), 0.91 (d, *J* = 7.0 Hz, 1.2H, H^D), 0.85 (d, *J* = 7.0 Hz, 1.8H, H^E), 0.84 (d, *J* = 7.0 Hz, 1.2H, H^E); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 214.31 (C^{7min}), 214.25 (C^{7maj}), 162.1 (C^{1maj}), 160.8 (C^{1min}), 128.7 (C^{2maj}), 124.7 (C^{2min}), 113.1 (C^{3min}), 111.3 (C^{3maj}), 87.4 (C^{5min}), 87.3 (C^{5maj}), 85.3 (C^{11}), 84.7 (C^{18}), 68.1 (C^{19}), 61.41 (C^{Cmin}), 61.38 (C^{Cmaj}), 57.8 (C^F), 49.1 (C^{6min}), 49.0 (C^{6maj}), 42.3 (C^{8maj}), 42.2 (C^{8min}), 37.6 (C^{4min}), 37.4 (C^{4maj}), 34.77 (C^{10min}), 34.74 (C^{10maj}), 33.1 (C^{Amin}), 30.33 (C^{12min}), 30.29 (C^{12maj}), 29.3 (C^{14}), 28.7 (C^{15}), 28.4 (C^{16}), 27.6 (C^{Amaj}), 26.0 (C^{9min} + C^{13}), 25.97 (C^{9maj}), 19.40 (C^{Bmaj}), 19.37 (C^{Bmin}), 18.4 (C^{17}), 14.73 (C^{Emaj}), 14.67 (C^{Emin}), 13.6 (C^{Dmin}), 13.5 (C^{Dmaj}); IR (neat): $\tilde{\nu}$ = 3262, 2931, 2858, 1693, 1655, 1457, 1371, 1319, 1274, 1194, 1156, 1092, 1070, 938, 726, 686 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 444 (100 %, $[M+Na]^+$); HRMS calcd for $C_{25}H_{43}O_4Na$ 444.3090, found 444.3082; $[\alpha]_D^{20} = -102$ (*c* = 0.43, $CHCl_3$).

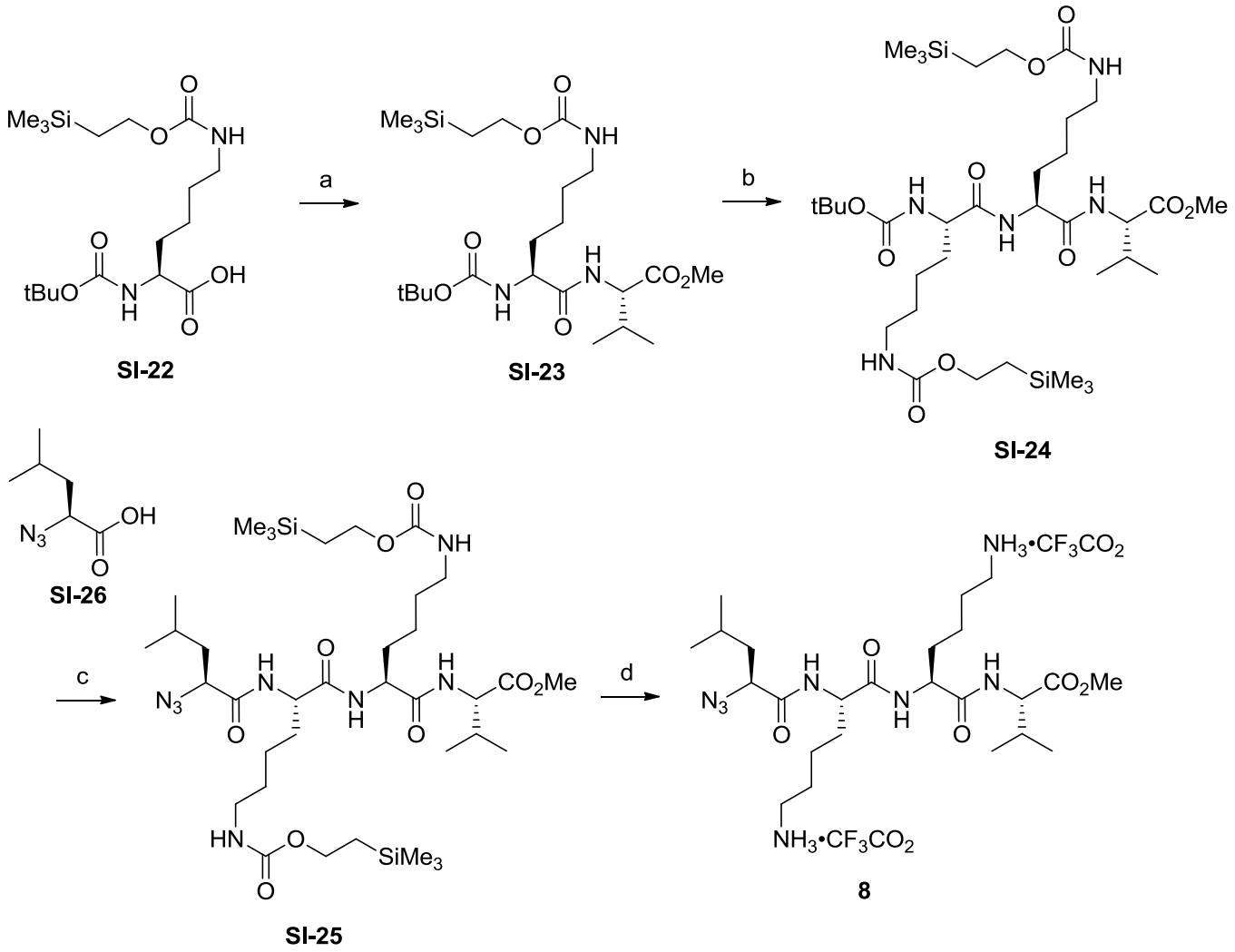
Compound 7c.[10] CuI (2.5 mg, 0.013 mmol), K_3PO_4 (28 mg, 0.13 mmol), 1,2-trans-cyclohexyldiamine (2.9 mg, 0.025 mmol) and MeNCHO (38 mg, 0.65 mmol) were added to a dried resealable J-Young Schlenk tube in a glovebox. The tube was removed from the glovebox and **6c** (36 mg, 0.065 mmol) in dioxane (2 mL) was added. The tube was then sealed and heated at 80 °C for 16 hours. The reaction was cooled to room temperature, diluted with EtOAc and filtered through a short pad of Celite. The solvent was removed under reduced pressure and the crude residue was diluted in THF:EtOH:H_2O:2,6-lutidine (2 mL, 1:1:1:0.1) before adding added $AgNO_3$ (44 mg, 0.26 mmol). The reaction mixture was stirred at room temperature in the dark for 48 hours. The reaction mixture was then diluted and filtered through a pad of Celite. The solvent was removed under reduced pressure. Purification by flash column chromatography on silica gel (petroleum ether/Et_2O, 2:1 to 1:2) gave **7c** as a colourless oil (11.3 mg, 43% over 2 steps, d.r. = 88:12). ^{1}H-NMR (500 MHz, $CDCl_3$) δ 8.29 (s, 0.6H, H^1), 8.08 (s, 0.4H, H^1), 7.13 (d, *J* = 14.9 Hz, 0.4H, H^2), 6.46 (d, *J* = 14.3 Hz, 0.6H, H^2), 5.17-5.07 (m, 1H, H^3), 3.35 (s, 6H, H^C + H^F), 3.33-3.29 (m, 1H, H^5), 3.08 (s, 1.2H, H^A), 3.04 (s, 1.8H, H^A), 3.00-2.95 (m, 1H, H^{11}), 2.68 (dq, *J* = 9.5 and 7.0 Hz, 0.6H^6), 2.65 (dq, *J* = 9.8 and 7.1 Hz, 0.4H^6), 2.56-2.47 (m, 2H, H^8), 2.47-2.34 (m, 1H, H^4), 2.19 (td, *J* = 7.1 and 2.7 Hz, 2H, H^{16}), 1.94 (t, *J* = 2.7 Hz, 1H, H^{18}), 1.80-1.70 (m, 1H, $H^{9\alpha}$), 1.69-1.61 (m, 1H, H^{10}), 1.58-1.50 (m, 2H, H^{15}), 1.48-1.30 (m, 7H, $H^{9\beta}$ + H^{12} + H^{13} + H^{14}), 1.16 (d, *J* = 7.0 Hz, 3H, H^B), 0.93 (d, *J* = 7.0 Hz, 1.8H, H^D), 0.92 (d, *J* = 7.0 Hz, 1.2H, H^D), 0.86 (d, *J* = 7.0 Hz, 1.8H, H^E), 0.85 (d, *J* = 7.0 Hz, 1.2H, H^E); ^{13}C-NMR (125 MHz, $CDCl_3$) δ = 214.3 (C^{7min}), 214.2 (C^{7maj}), 162.1 (C^{1maj}), 160.8 (C^{1min}), 128.7 (C^{2maj}), 124.7 (C^{2min}), 113.1 (C^{3min}), 111.3 (C^{3maj}), 87.4 (C^{5min}), 87.3 (C^{5maj}), 85.2 (C^{11}), 84.6 (C^{17}), 68.1 (C^{18}), 61.4 (C^{Cmin}), 61.3 (C^{Cmaj}), 57.8 (C^F), 49.1 (C^{6min}), 48.9 (C^{6maj}), 42.3 (C^{8maj}), 42.2 (C^{8min}), 37.6 (C^{4min}), 37.4 (C^{4maj}), 34.77 (C^{10min}), 34.75 (C^{10maj}), 33.1 (C^{Amin}), 30.24 (C^{12min}), 30.20 (C^{12maj}), 28.9 (C^{14}), 28.4 (C^{15}), 27.6 (C^{Amaj}), 26.0 (C^{9min}), 25.9 (C^{9maj}), 25.6 (C^{13}), 19.4 (C^{Bmaj}), 19.3 (C^{Bmin}), 18.3 (C^{16}), 14.7 (C^{Emaj}), 14.6 (C^{Emin}), 13.6 (C^{Dmin}), 13.5 (C^{Dmaj}); IR (neat): $\tilde{\nu}$ = 3262, 2930, 1693, 1655, 1456, 1372, 1274, 1193, 1092, 1070, 969, 726, 686 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 430 (100%, $[M+Na]^+$); HRMS calcd for $C_{24}H_{41}NO_4Na$ 430.2933, found 430.2932; $[\alpha]_D^{20} = -66.2$ (*c* = 1.1, $CHCl_3$).



7c

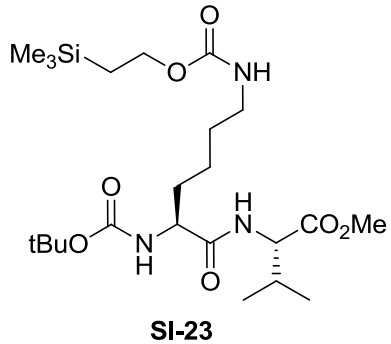
[10] Two rotamers were observed at room temperature. In the description of the ^{13}C NMR spectrum, the signals specific to each rotamer are indicated with the terms maj and min for the major and minor rotamer, respectively.

Preparation of compounds 8 and 9

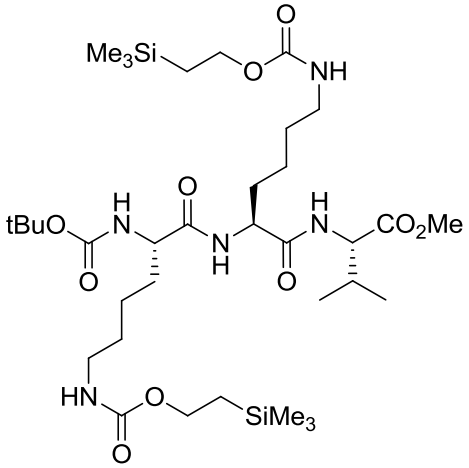


[(a)] HCl•H_2NValOMe, hydroxybenzotriazole, 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride, Et_3N, CH_2Cl_2. [(b)] i) pTsOH, Et_2O; ii) **SI-22**, hydroxybenzotriazole, 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride, Et_3N, CH_2Cl_2. [(c)] i) pTsOH, Et_2O; ii) **SI-26**, hydroxybenzotriazole, 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride, Et_3N, CH_2Cl_2. [(d)] CH_2Cl_2, trifluoroacetic acid.

Compound SI-23. Compound **SI-22** was suspended in CH_2Cl_2 (15 mL) with the hydrochloride salt of H_2NValOMe (227 mg, 1.66 mmol), hydroxybenzotriazole (224 mg, 1.66 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (317 mg, 1.66 mmol), before adding Et_3N (0.42 mL, 3.02 mmol). The reaction mixture was stirred at room temperature for 16 hours. The reaction was diluted with EtOAc (50 mL), washed sequentially with citric acid (0.5M, 50 mL) and saturated aqueous $NaHCO_3$ (50 mL). The organic phase was dried (Na_2SO_4) and the solvent removed under reduced pressure to give the title compound as a colourless oil (737 mg, 1.47 mmol, 97%), which was used without further purification in the following step. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.67-6.49 (m, 1H(NH)), 5.17-4.94 (m, 1H(NH)), 4.82-4.66 (m, 1H(NH)), 4.52 (dd, J = 8.8 and 4.9 Hz, 1H), 4.19-4.03 (m, 3H), 3.73 (s, 3H), 3.21-3.07 (m, 2H), 2.22-2.12 (m, 1H), 1.88-1.71 (m, 1H), 1.67-1.57 (m, 1H), 1.56-1.47 (m, 2H), 1.44 (s, 9H), 1.43-1.33 (m, 2H), 1.01-0.91 (m, 2H), 0.92 (d, J = 6.7 Hz, 3H), 0.89 (d, J = 6.9 Hz, 3H), 0.02 (s, 9H); IR (neat): $\tilde{\nu}$ = 3320 (br), 2954, 1742, 1691, 1662, 1521, 1456, 1438, 1392, 1366, 1337, 1248, 1212, 1171, 1045, 1022, 937, 859, 837, 778, 733, 694 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 526 (100 %, $[M+Na]^+$); HRMS calcd for $C_{23}H_{45}N_3O_7SiNa$ 526.2924, found 526.2924.

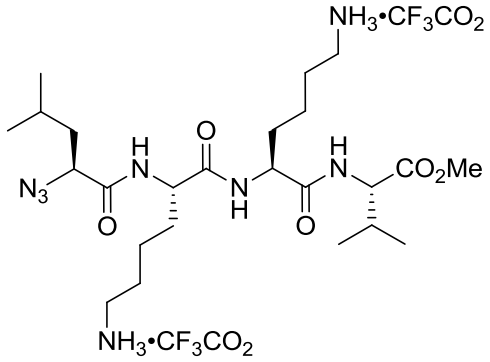


Compound SI-24. *p*-toluenesulfonic acid (293 g, 1.54 mmol) was added to a solution of **SI-23** (706 mg, 1.40 mmol) in Et_2O (50 mL). The reaction was held at 30 °C for 30 minutes. The solvent was removed under reduced pressure at 30 °C and held at this temperature under vacuum for 1 hour. The crude product was kept under high vacuum for 16 hours to give a glass residue (MS (ES^+) *m/z* (rel. intensity) 404 (100 %, $[M+H]^+$), 426 (50 %, $[M+Na]^+$); HRMS calcd for $C_{18}H_{38}N_3O_5Si$ 404.2581, found 404.2578) which was suspended in CH_2Cl_2 (14 mL) with **SI-22** (491 mg, 1.26 mmol), hydroxybenzotriazole (207 mg, 1.54 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (294 mg, 1.54 mmol) before adding Et_3N (0.39 mL, 2.8 mmol). The same procedure as described for the preparation of **SI-23** was then applied to give crude **SI-24** as a colourless oil (926 mg, 85%), which was used without further purification in the following step. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 4.52 (dd, *J* = 8.8 and 4.9 Hz, 1H), 4.46-4.38 (m, 1H), 4.22-4.03 (m, 5H), 3.73 (s, 3H), 3.24-3.03 (m, 4H), 2.22-2.12 (m, 1H), 1.92-1.73 (m, 2H), 1.73-1.58 (m, 2H), 1.58-1.46 (m, 4H), 1.43 (s, 9H), 1.42-1.31 (m, 4H), 1.05-0.90 (m, 4H), 0.91 (d, *J* = 6.7 Hz, 3H), 0.89 (d, *J* = 6.9 Hz, 3H), 0.03 (s, 18H);[11] IR (neat): $\tilde{\nu}$ = 3311 (br), 2953, 1693, 1648, 1524, 1457, 1438, 1392, 1366, 1337, 1249, 1172, 1059, 912, 859, 836, 778, 731, 694 cm^{-1}; MS (ES^+) *m/z* (rel. intensity) 798 (100 %, $[M+Na]^+$); HRMS calcd for $C_{35}H_{69}N_5O_{10}Si_2Na$ 798.4481, found 798.4501.



SI-24

Compound 8. *p*-toluenesulfonic acid (224 mg, 1.28 mmol) was added to a solution of **SI-24** (914 mg, 1.17 mmol) in Et_2O (40 mL). The reaction was held at 30 °C for 30 minutes. The solvent was removed under reduced pressure at 30 °C and held at this temperature under vacuum for 1 hour. The crude product was kept under high vacuum for 16 hours to give a glass residue (MS (ES^+) *m/z* (rel. intensity) 698 (100 %, $[M+Na]^+$); HRMS calcd for $C_{30}H_{61}N_5O_8Si_2Na$ 698.3964, found 698.3956) which was suspended in CH_2Cl_2 (12 mL) with **SI-26**[12] (239 mg, 1.52 mmol), hydroxybenzotriazole (197 mg, 1.29 mmol) and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (244 mg, 1.29 mmol), before adding Et_3N (0.30 mL, 2.34 mmol). The reaction mixture was stirred at room temperature for 16 hours. The reaction was diluted with EtOAc (40 mL), washed sequentially with citric acid (0.5M, 40 mL) and saturated aqueous $NaHCO_3$ (40 mL). The organic phase was dried (Na_2SO_4) and the solvent removed under reduced pressure to give **SI-25** as a colourless oil (**MS** (ES^+) *m/z* (rel. intensity) 837 (100 %, $[M+Na]^+$); **HRMS** calcd for $C_{36}H_{70}N_8O_9Si_2Na$ 837.4702, found 837.4711). This crude material was then diluted in CH_2Cl_2/TFA (18 mL, 4:1, 0.1M) at 0 ºC. After stirring at room temperature for 2 hours, the solvent was removed under reduced pressure. Purification by flash chromatography on deactivated silica gel[13] (CH_2Cl_2 then CH_2Cl_2/MeOH, 9:1) afforded **8** as a pale orange glass residue (441 mg, 50%). ^{1}H-NMR (500 MHz, CD_3OD) δ 4.45-4.39 (m, 1H), 4.35-4.27 (m, 2H), 3.85 (dd, *J* = 8.5 and 6.0 Hz, 1H), 3.71 (s, 3H), 3.01–2.90 (m, 4H), 2.18-2.11 (m,



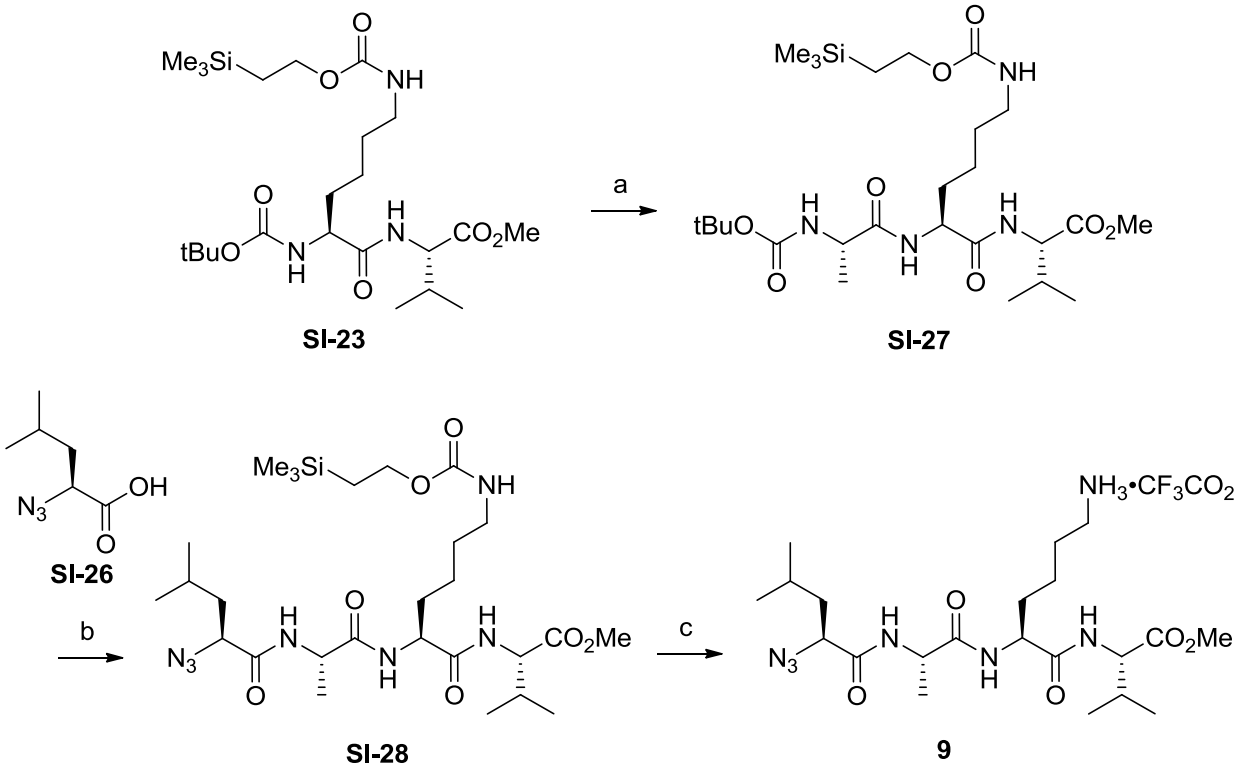
8

[11] Not all the rapidly exchangeable protons are visible in $CDCl_3$. However, several poorly resolved signals were visible between 6.89 and 4.83 ppm: 6.89-4.83 (m, 0.7H), 6.73-6.60 (m, 0.7H), 5.30-5.18 (m, 0.8H), 5.08-4.85 (m, 1.7H).

[12] J. T. Lundquist IV and J. C. Pelletier, *Org. Lett.*, 2001, **3**, 781

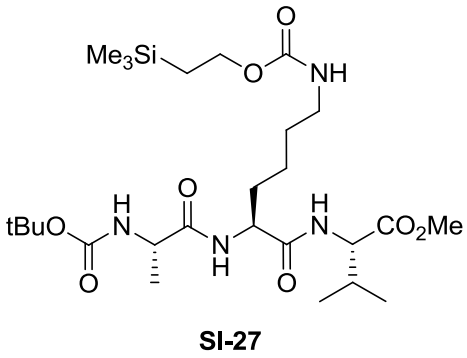
[13] The silica was treated with $EtSiCl_3$ prior to the chromatography. See: P. Panne and M. J. Fox, *J. Am. Chem. Soc.*, 2007, **129**, 22

1H), 1.89-1.78 (m, 2H), 1.76-1.61 (m, 8H), 1.57-1.39 (m, 5H), 0.98-0.92 (m, 12H);[14] ^{13}C-NMR (125 MHz, CD_3OD) δ = 175.0, 174.9, 174.4, 173.3, 63.2, 60.1, 55.4, 55.2, 53.3, 42.1, 41.4, 41.3, 33.2, 33.1, 32.6, 28.8 (2C), 27.0, 24.5 (2C), 24.1, 22.9, 20.2, 19.4; IR (neat): $\tilde{\nu}$ = 3288 (br), 2961, 2873, 2407, 2111, 1735, 1640, 1541, 1436, 1390, 1372, 1314, 1267, 1200, 1179, 1132, 1011, 977, 836, 799, 721 cm^{-1}; MS (ES^+) *m/z* (rel. intensity) 599 (100 %, $[M+Na]^+$); MS (ES^+) *m/z* (rel. intensity) 527 (100 %, $[M+H]^+$); HRMS calcd for $C_{24}H_{47}N_8O_5$ 527.3669, found 527.3672.



[(a)] i) pTsOH, Et_2O; ii) BocAlaOH, hydroxybenzotriazole, 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride, Et_3N, CH_2Cl_2. [(b)] i) pTsOH, Et_2O; ii) **SI-26**, hydroxybenzotriazole, 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride, Et_3N, CH_2Cl_2. [(c)] CH_2Cl_2, trifluoroacetic acid.

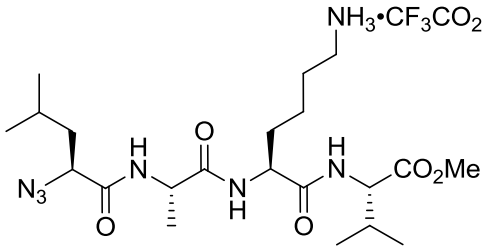
Compound SI-27. This compound was prepared from **SI-23** (116 mg, 0.23 mmol) and BocAlaOH (34 mg, 0.21 mmol) using the same procedure as described for the preparation of **SI-24**. The crude material thus obtained was a colourless oil (92 mg, 76%) and it was used without further purification in the following step. ^{1}H-NMR (500 MHz, $CDCl_3$) δ 6.83-6.69 (m, 1H(NH)), 6.67-6.50 (m, 1H(NH)), 5.22-4.99 (m, 1H(NH)), 4.92-4.75 (m, 1H(NH)), 4.52 (dd, *J* = 8.8 and 4.9 Hz, 1H), 4.46-4.37 (m, 1H), 4.22-4.03 (m, 3H), 3.74 (s, 3H), 3.23-3.07 (m, 2H), 2.22-2.12 (m, 1H), 1.94-1.81 (m, 1H), 1.75-1.63 (m, 1H), 1.58-1.45 (m, 2H), 1.43 (s, 9H), 1.42-1.31 (m, 2H), 1.36 (d, *J* = 7.2 Hz, 3H), 1.01-0.91 (m, 2H), 0.92 (d, *J* = 6.9 Hz, 3H), 0.89 (d, *J* = 7.4 Hz, 3H), 0.03 (s, 9H).



SI-27

[14] Not all the rapidly exchangeable protons are visible in CD_3OD: 8.38 (d, *J* = 7.5 Hz, 0.2H), 8.30 (d, *J* = 7.5 Hz, 0.2H), 8.28 (d, *J* = 7.5 Hz, 0.4H).

Compound 9. This compound was prepared from **SI-27** (92 mg, 0.016 mmol) following the same sequence of steps as described for the preparation of **8**. Pale orange glass (40 mg, 53%) ^{1}H-NMR (400 MHz, CD_3OD, 313 K) δ 4.50-4.42 (m, 1H), 4.40-4.31 (m, 2H), 3.91-3.85 (m, 1H), 3.74 (s, 3H), 3.00-2.93 (m, 2H), 2.17 (sextet, *J* = 6.0 Hz, 1H), 1.93-1.83 (m, 1H), 1.82-1.63 (m, 6H), 1.59-1.45 (m, 2H), 1.40 (d, *J* = 6.7 Hz, 3H), 1.02-0.94 (m, 12H);[15] ^{13}C-NMR (100 MHz, CD_3OD, 313 K) δ = 174.8, 174.2, 173.7, 172.9, 62.4, 59.4, 54.2, 52.6, 50.8, 41.5, 40.8, 32.5, 31.9, 28.1, 26.3, 23.7, 23.4, 22.3, 19.5, 18.6, 18.0; IR (neat): $\tilde{\nu}$ = 3288 (br), 2961, 2419, 2112, 1743, 1644, 1543, 1460, 1371, 1204, 1019, 722 cm^{-1}; MS (ES^+) *m/z* (rel. intensity) 470 (100 %, $[M+H]^+$); HRMS calcd for $C_{21}H_{40}N_7O_5$ 470.3078, found 470.3078.

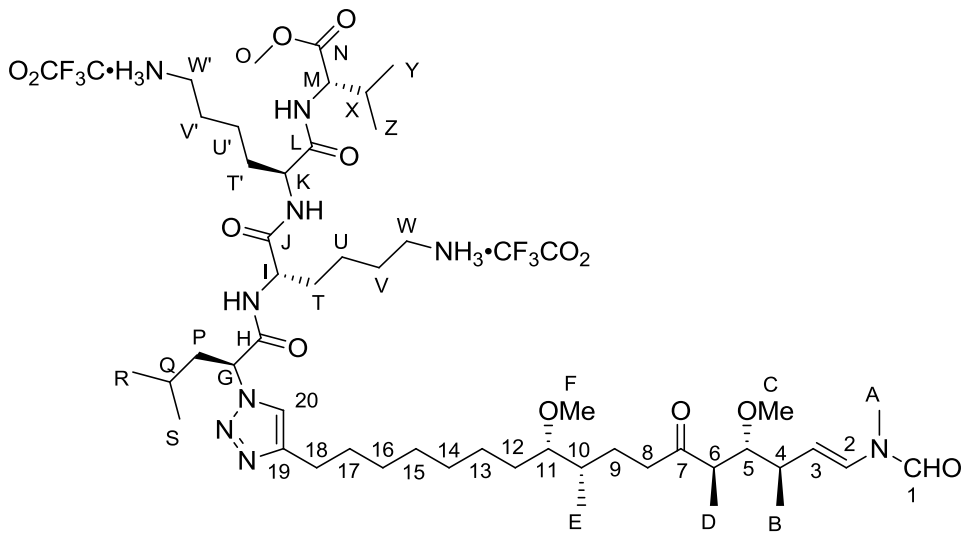


9

[15] Not all the rapidly exchangeable protons are visible in CD_3OD: 8.31 (d, *J* = 6.0 Hz, 0.3H), 8.15-8.11 (m, 0.4H), 8.03 (d, *J* = 8.1 Hz, 0.7H).

Preparation of compounds 10 and 11

Compound 10.[16] Compound **7c** (7.4 mg, 0.017 mmol), **8** (12.8 mg, 0.017 mmol), $CuSO_4$ (0.017 mL, 0.1M in water, 10 mol%) and sodium-L-ascorbate (0.6 mg, 0.0034 mmol, 40 mol%) were stirred in ^{t}BuOH (0.17 mL, 0.1M) for 48 hours at 35 °C. The reaction mixture was diluted with ethanol and filtered though a short pad of celite. The solvent was removed under reduced pressure and the crude residue purified by flash column chromatography on silylated silica gel[12] (EtOAc/EtOH, 20:1 to 1:1) to give **10** as a gum (8.0 mg, 39%, d.r. = 87:13).

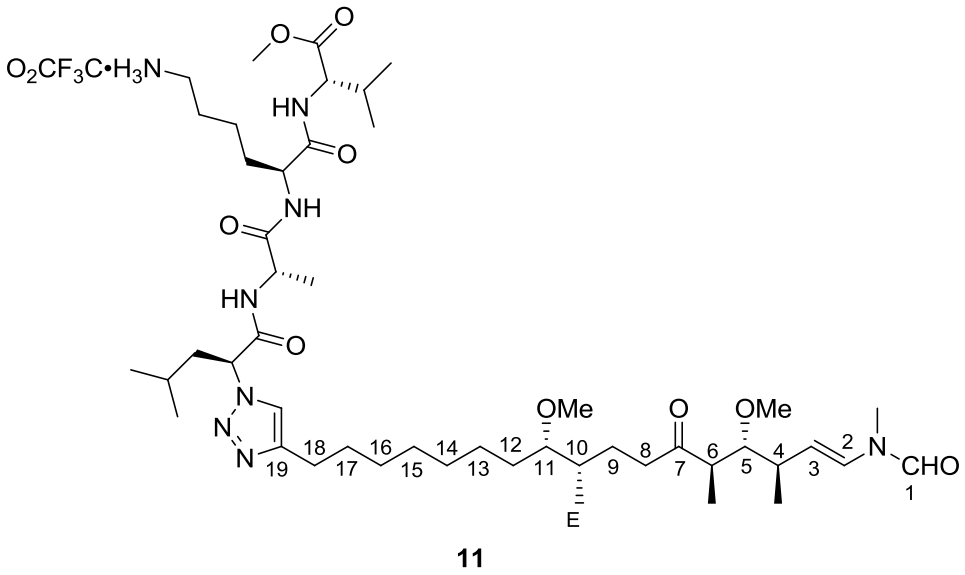


10

^{1}H-NMR (500 MHz, CD_3OD) δ 8.32 (s, 0.65H, H^1), 8.06 (s, 0.35H, H^1), 7.93-7.85 (m, 1H, H^{20}), 7.10 (d, *J* = 14.8 Hz, 0.35H, H^2), 6.71 (d, *J* = 14.2 Hz, 0.65H, H^2), 5.50-5.40 (m, 1H, H^G), 5.26 (dd, *J* = 14.7 and 9.4 Hz, 0.35H, H^3), 5.19 (dd, *J* = 14.1 and 9.2 Hz, 0.65H, H^3), 4.43 (dd, *J* = 8.8 and 5.6 Hz, 1H, H^I/H^K), 4.36-4.27 (m, 2H, H^M + H^I/H^K), 3.72 (s, 3H, H^O), 3.36 (s, 6H, H^C + H^F), 3.11 (s, 1.05H, H^A), 3.06-2.99 (m, 1H, H^5), 3.03 (s, 1.95H^A), 2.98-2.93 (m, 2H, H^W/$H^{W'}$), 2.92-2.87 (m, 2H, H^W/$H^{W'}$), 2.80-2.74 (m, 1H, H^6), 2.74-2.66 (m, 2H, H^{18}), 2.59-2.53 (m, 2H, H^8), 2.51-2.43 (m, 1H, H^4), 2.20-2.12 (m, 1H, H^X), 2.12-2.02 (m, 1H, H^P), 2.02-1.94 (m, 1H, H^P), 1.89-1.78 (m, 2H, $H^{T\alpha}$ + $H^{T\alpha'}$), 1.77-1.57 (m, 10H, $H^{9\alpha}$ + H^{10} + H^{17} + H^V + $H^{V'}$ + $H^{T\beta}$ + $H^{T\beta'}$), 1.55-1.25 (m, 19H, $H^{9\beta}$ + H^{12} + H^{13} + H^{14} + H^{15} + H^{16} + H^Q + H^U + $H^{U'}$), 1.17 (d, *J* = 7.0 Hz, 3H, H^B), 1.01-0.88 (m, 15H, H^D + H^R + H^S + H^Y + H^Z), 0.85 (d, *J* = 6.8 Hz, 3H, H^E); MS (ES^+) *m/z* (rel. intensity) 962 (100 %, $[M+H]^+$); IR (neat): $\tilde{\nu}$ = 3306 (br), 3068, 2934, 2870, 1740, 1652, 1547, 1459, 1438, 1373, 1275, 1202, 1180, 1136, 1094, 972, 835, 799, 722 cm^{-1}; HRMS calcd for $C_{50}H_{92}N_9O_9$ 962.7018, found 962.7047.

[16] Two rotamers were visible in the ^{1}H NMR spectrum. The signal for H^{11} is masked by CD_3OD. The rapidly exchangeable protons were mostly invisible in CD_3OD and are therefore not listed in the description.

Compound 11.[15] This compound was obtained from **7c** (5.0 mg, 0.011 mmol) and **9** (6.7 mg, 0.011 mmol) using the same procedure as described for the preparation of **10**. Gum (7.1 mg, 63%, d.r. = 87:13). ^{1}H-NMR (500 MHz, CD_3OD) δ 8.32 (s, 0.65H, H^1), 8.09 (s, 0.35H, H^1), 7.90-7.82 (m, 1H, H^{20}), 7.10 (d, *J* = 14.6 Hz, 0.35H, H^2), 6.70 (d, *J* = 14.2 Hz, 0.65H, H^2), 5.45-5.38 (m, 1H, H^G), 5.26 (dd, *J* = 14.6 and 9.0 Hz, 0.35H, H^3), 5.19 (dd, *J* = 14.2 and 9.3 Hz, 0.65H, H^3), 4.44 (dd, *J* = 8.4 and 5.4 Hz, 1H, H^K), 4.32 (d, *J* = 7.0 Hz, 1H, H^M), 4.26 (q, *J* = 6.9 Hz, H^I), 3.72 (s, 3H, H^O), 3.36 (s, 6H, H^C + H^F), 3.11 (s, 1.05H, H^A), 3.06-2.99 (m, 1H, H^5), 3.03 (s, 1.95H^A), 2.98-2.93 (m, 2H, H^W), 2.80-2.74 (m, 1H, H^6), 2.74-2.66 (m, 2H, H^{18}), 2.58-2.52 (m, 2H, H^8), 2.51-2.42 (m, 1H, H^4), 2.20-2.12 (m, 1H, H^X), 2.12-2.02 (m, 1H, H^P), 2.02-1.94 (m, 1H, H^P), 1.89-1.78 (m, 1H, $H^{T\alpha}$), 1.76-1.62 (m, 7H, $H^{9\alpha}$ + H^{10} + H^{17} + H^V + $H^{T\beta}$), 1.55-1.25 (m, 18H, $H^{9\beta}$ + H^{12} + H^{13} + H^{14} + H^{15} + H^{16} + H^Q + H^U + $H^{Z'}$), 1.17 (d, *J* = 7.0 Hz, 3H, H^B), 1.01-0.88 (m, 15H, H^D + H^R + H^S + H^Y + H^Z), 0.85 (d, *J* = 6.8 Hz, 3H, H^E); IR (neat): $\tilde{\nu}$ = 3306 (br), 3302 (br), 2930, 2414, 1793, 1740, 1651, 1547, 1457, 1371, 1272, 1202, 1148, 1092, 723 cm^{-1}; MS (ES$^+$) *m/z* (rel. intensity) 905 (100 %, $[M+H]^+$); HRMS calcd for $C_{47}H_{85}N_8O_9$ 905.6440, found 905.6470.



11

Bioassays conducted on 7a–7c, 10 and 11

Rabbit muscle actin was prepared as described in Winder and Walsh 1990.[17] For G-actin interaction assays, actin was labelled with acrylodan according to a previously published method.[18] Compounds were incubated with 2µM prodan-actin and following excitation at 380nm any interaction was monitored by scanning the emission between 400 and 650nm using a Varian Cary Eclipse fluorescence spectrophotometer.

The effect of compounds on 6µM F-actin in vitro was determined following a high speed centrifugation pelleting assay and SDS-PAGE analysis of resultant supernatant (G-actin) and pellet (F-actin) fractions as described previously. SDS-gels were scanned with a Biorad ChemiDoc XRS+ and quantified with Image Lab software using volume integration and local background subtraction.[19] Representative results are depicted in Figure S1.

Actin polymerisation assays were conducted with varying concentrations of compounds using 5µM pyrenyl G-actin in a Varian fluorescence spectrophotometer according to previously described procedures.[20] Representative results are depicted in Figure S2.

REF52 rat embryo fibroblasts were grown in DMEM with 10% FCS as described previously.[21] Cells were seeded in flat bottomed 96 well tissue culture plates and incubated with various concentrations of the compounds for either 2 or 24 hours. Following incubation, cells were fixed with 3.7% formaldehyde in PBS (10 min) permeabilised in 0.1% Triton X-100 in PBS (1 min) and incubated with 1µg/ml rhodamine phalloidin for one hour. Following 3 washes in PBS cells were mounted directly in the 96 well plate by addition of 20µl of Hydromount containing DAPI. Cells were visualised on a Leica DMIRE2 microscope and images captured using Leica Q-fluoro software.

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[17] S. J. Winder and M. P. Walsh, *J Biol Chem.*, 1990, **265**, 10148.
[18] G. Marriott, K. Zechel K and T. M. Jovin, *Biochem.*, 1988, **27**, 6214.
[19] S. J. Winder, L. Hemmings, S. K. Maciver, S. J. Bolton, J. M. Tinsley, K. E. Davies, D. R. Critchley and J. Kenderick-Jones, *J Cell Sci.*, 1995, **108**, 63.
[20] M. Pfuhl, S. J. Winder and A. Pastore, *EMBO J.*, 1994, **13**, 1782.
[21] Y.-J. Chen, H. J. Spence, J. M. Cameron, T. Jess, J. L. Ilsley and S. J. Winder, *Biochem J.*, 2003, **375**, 329.

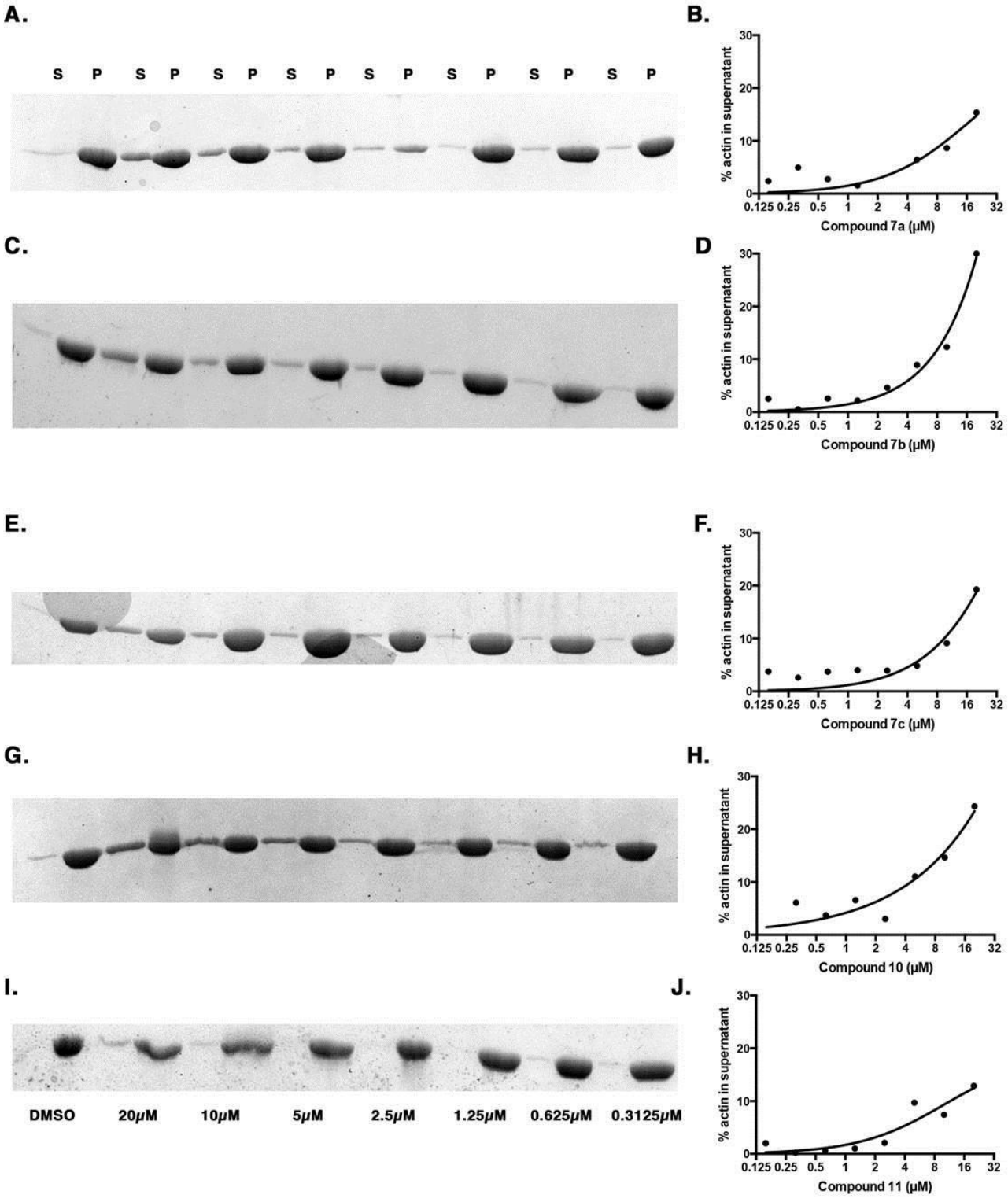


Figure S1. Coomassie blue stained SDS gel of equivalent volumes of supernatant (S) and pellet (P) fractions following incubation of 6μM F-actin with the indicated concentrations of compounds and separation by high speed centrifugation {**7a** (A)**, 7b** (C), **7c** (E), **10** (G) and **11** (I)}. At higher concentrations of **10**, there is a clear shift of actin from pellet to supernatant fraction indicative of depolymerisation of F-actin. Quantification of the amount of actin in the supernatant was perfomed for **7a-c, 10, 11** and DMSO control. In the presence of DMSO only, between 1 and 2% of actin in is the supernatant fraction. A compound was deemed to have a significant effect at a concentration that resulted in 5% of actin in the supernatant fraction. Quantification of the gels in A, C, E, G and I is shown in B, D, F, H and J, respectively.

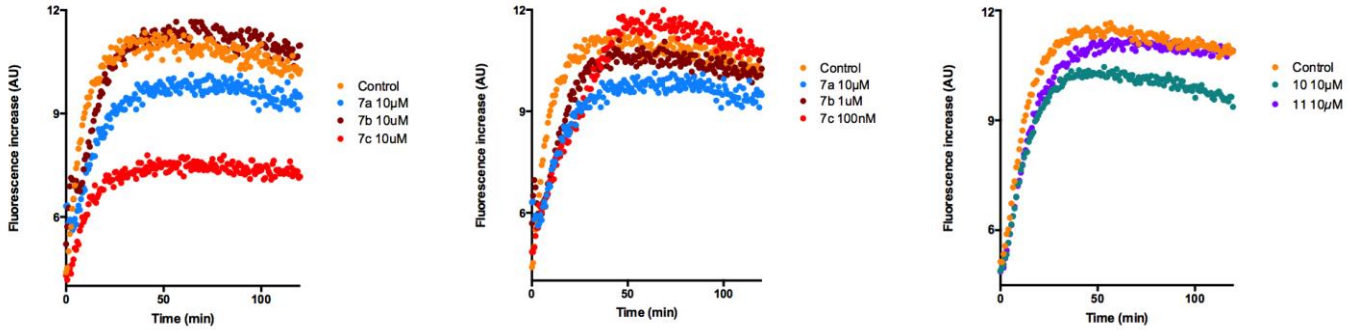
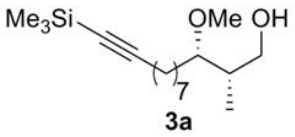


Figure S2. Effect of compounds **7a-c, 10** and **11** on the polymerisation rate of 2μM pyrenyl actin as monitored by the increase in pyrenyl fluorescence at 384nm on polymerisation. Reactions were monitored for 120 minutes and recorded as arbitrary fluorescence units. The initial rate of polymerisation is the most reliable parameter to determine an effect of a compound on simple actin polymerisation. Rates were calculated for compounds in the initial linear phase of polymerisation up to 20 minutes. The minimum concentration that reduced the polymerisation rate by 10% compared to DMSO control was deemed significant and is indicated in Table 1 of the manuscript.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3a

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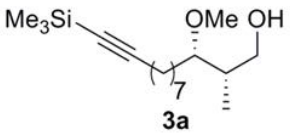
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3a

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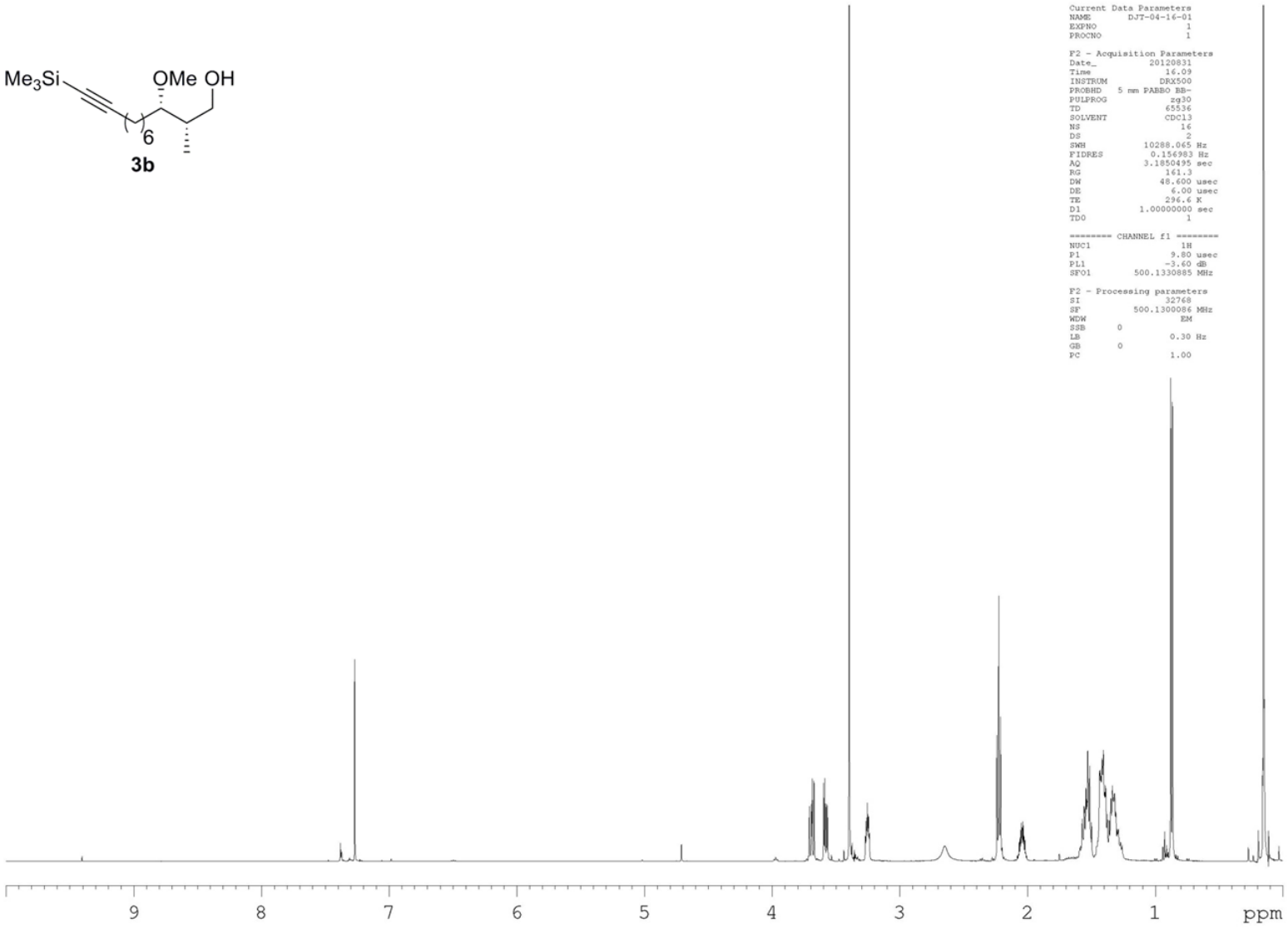
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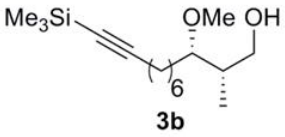
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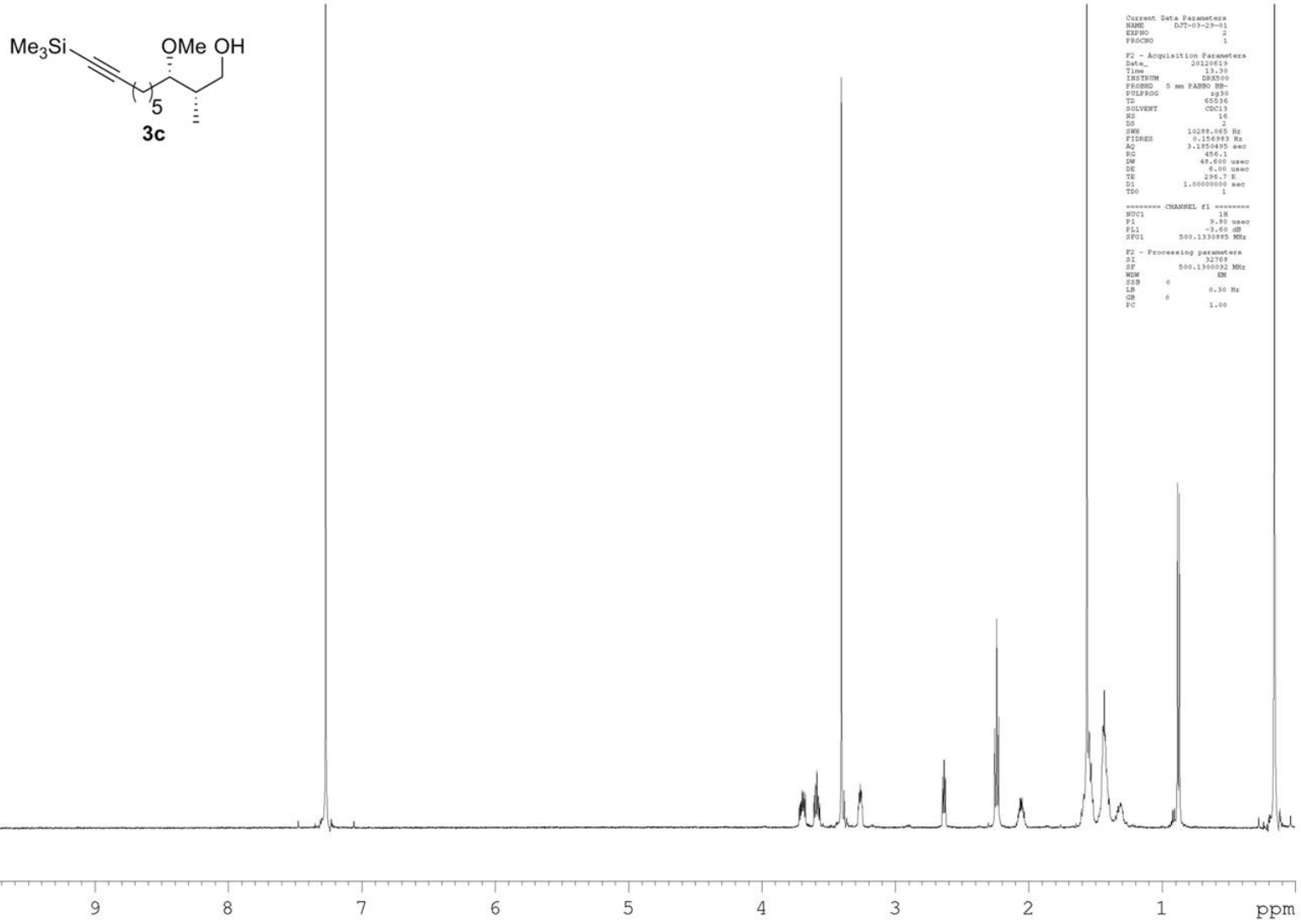
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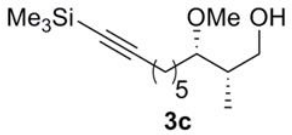
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3c

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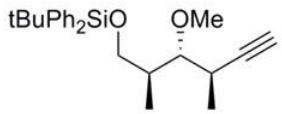
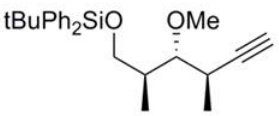


SI-19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SI-19

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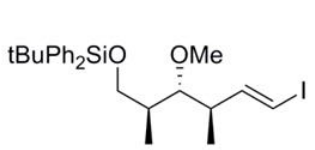
ppm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SI-20

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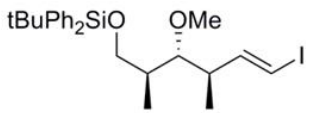
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SI-20

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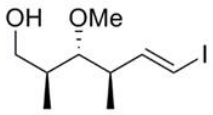
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SI-21

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TE 295.7 K
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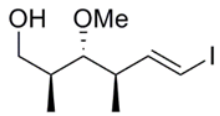
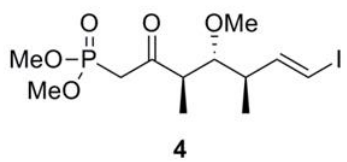
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -3.60 dB
SFO1 500.1330445 MHz
F2 - Processing parameters
SI 32768
SF 500.1300060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm


OH
OMe
I

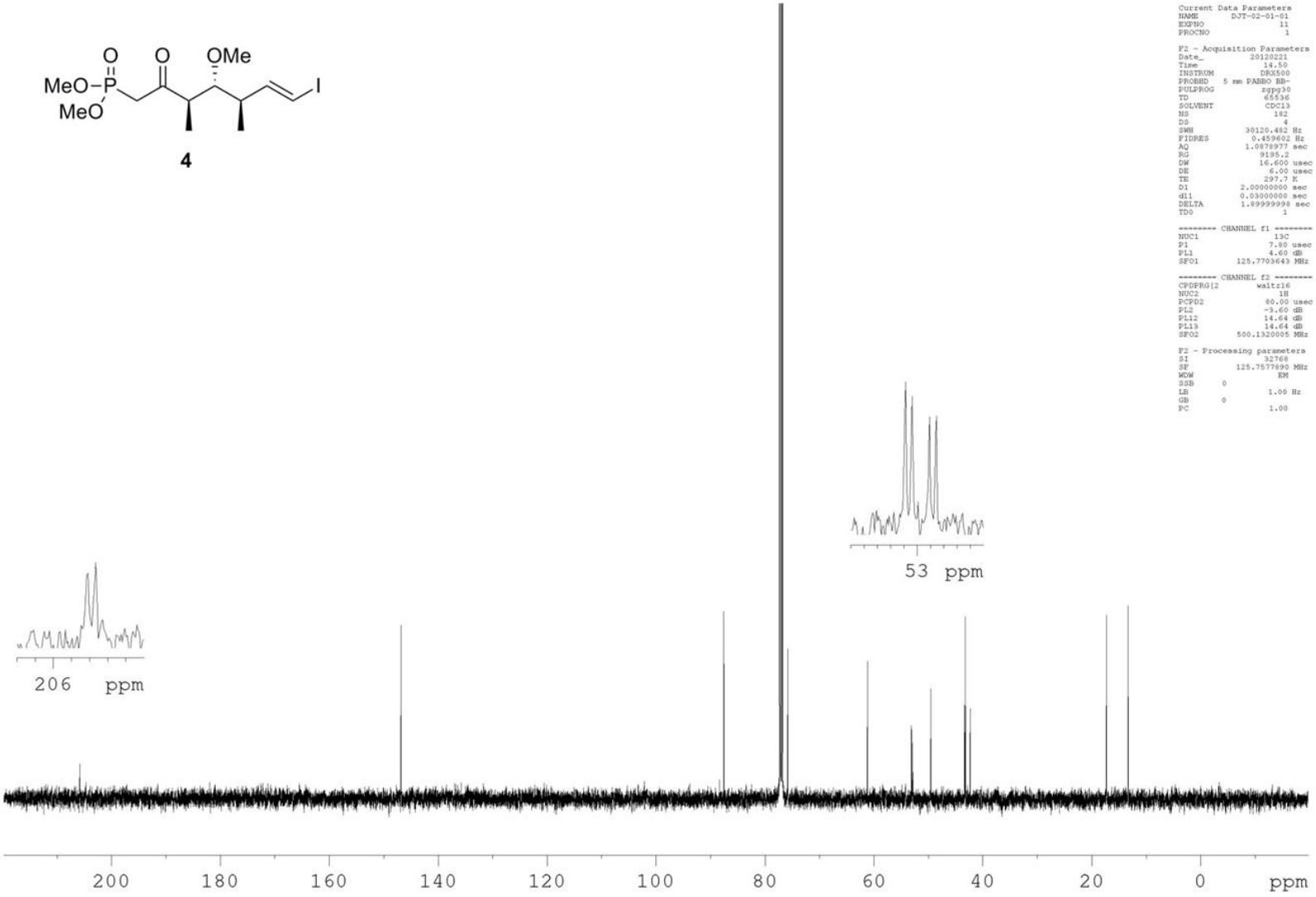
SI-21

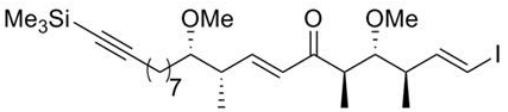
Current Data Parameters
NAME DJT-01-98-01
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120217
Time 17.43
INSTROM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 123
DS 4
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0878977 sec
RG 10321.3
DW 16.600 usec
DE 6.00 usec
TE 297.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
PL1 4.60 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.60 dB
PL12 14.64 dB
PL13 14.64 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


O
O
OMe
MeO–P
MeO
I

4

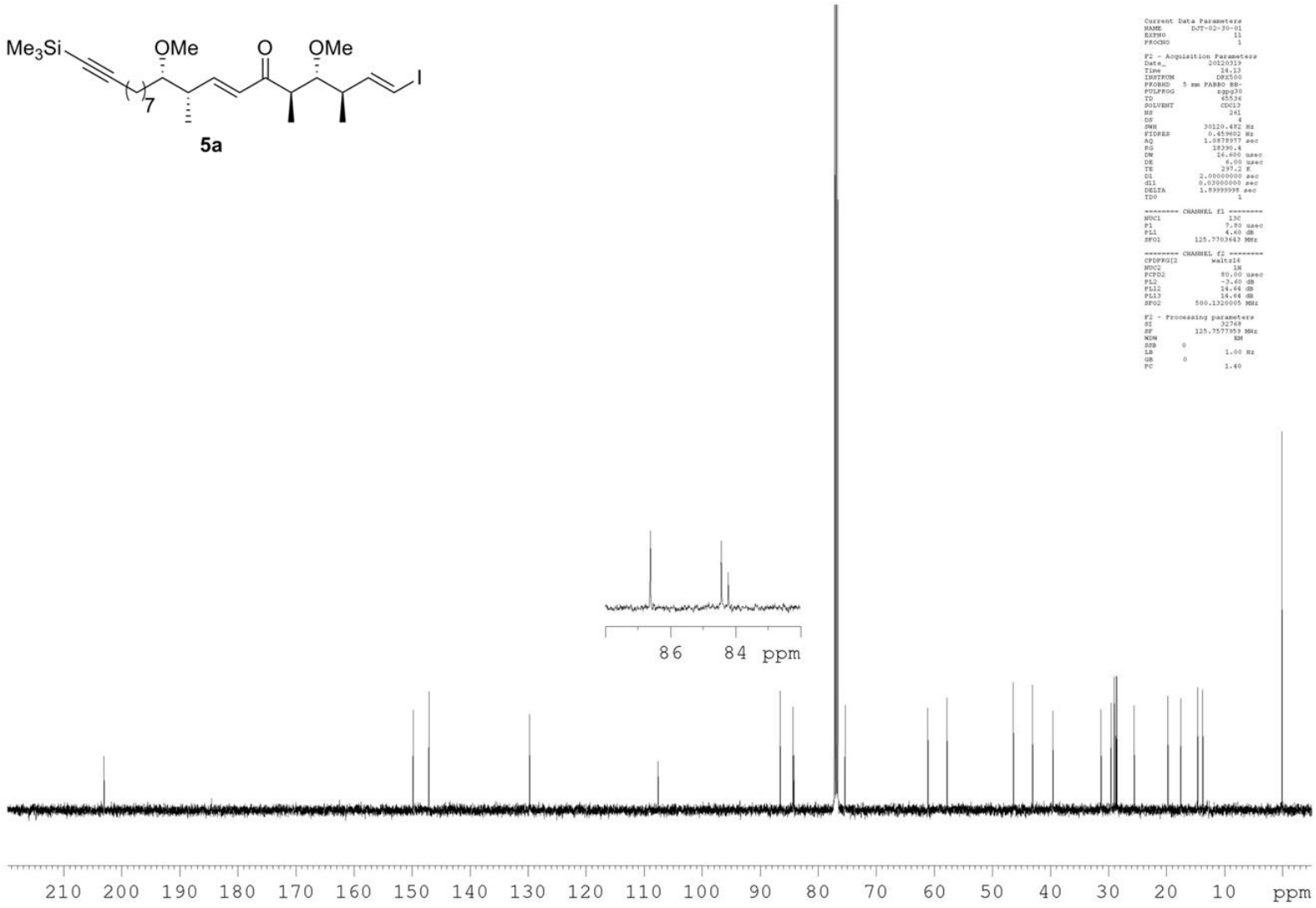
Current Data Parameters
NAME SAT-02-19-01
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120308
Time 17.26
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10299.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 128
DW 48.600 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -3.60 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.3
3.2
3.1
ppm
2.9
ppm
9
8
7
6
5
4
3
2
1
ppm

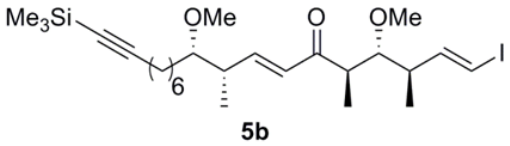

MeO–P
MeO
O
O
OMe
I
4
Current Data Parameters
NAME DJT-02-01-01
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120221
Time 14.50
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 182
DS 4
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0878977 sec
RG 9195.2
DW 16.600 usec
DE 6.00 usec
TE 297.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
PL1 4.60 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.60 dB
PL12 14.64 dB
PL13 14.64 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
206 ppm
53 ppm
200
180
160
140
120
100
80
60
40
20
0
ppm


Me3Si
OMe
O
OMe
I
7

5a

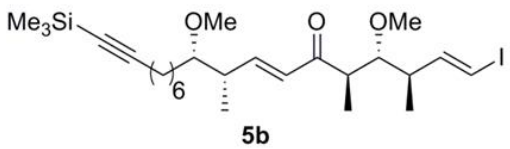
Current Data Parameters
NAME DJT-02-30-01
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120319
Time 13.55
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 90.5
DW 48.600 usec
DE 6.00 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -3.60 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm


Me3Si
OMe
O
OMe
I
7
5a
86
84 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Me3Si
OMe
O
OMe
I
6

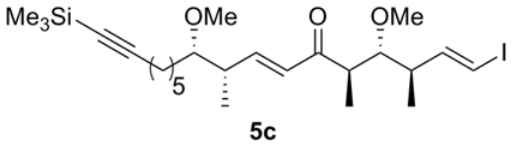
5b

Current Data Parameters
NAME DJT-04-20-01
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120905
Time 17.52
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 114
DW 48.600 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -3.60 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm


Me3Si
OMe
O
OMe
I
6

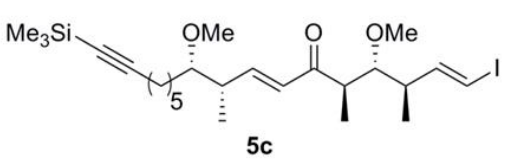
5b

Current Data Parameters
NAME DJT-04-20-01
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120905
Time 17.58
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 91
DS 4
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0878977 sec
RG 14596.5
DW 16.600 use
DE 6.00 use
TE 299.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 =======
NUC1 13C
P1 7.80 use
PL1 6.40 dB
SF01 125.7703643 MHz
======== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -3.40 dB
PL12 14.64 dB
PL13 14.64 dB
SF02 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577925 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Me3Si
OMe
O
OMe
I
5

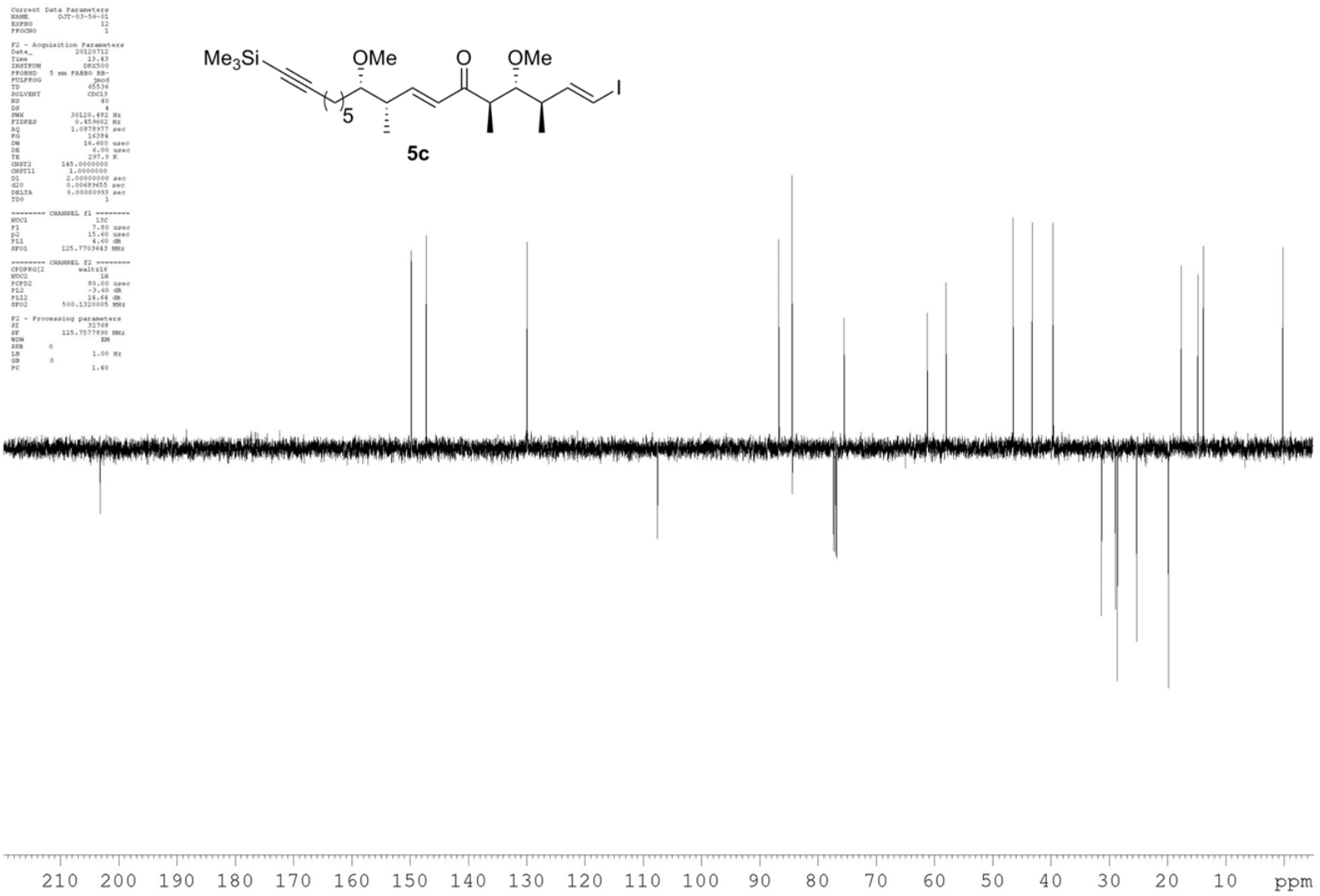
5c

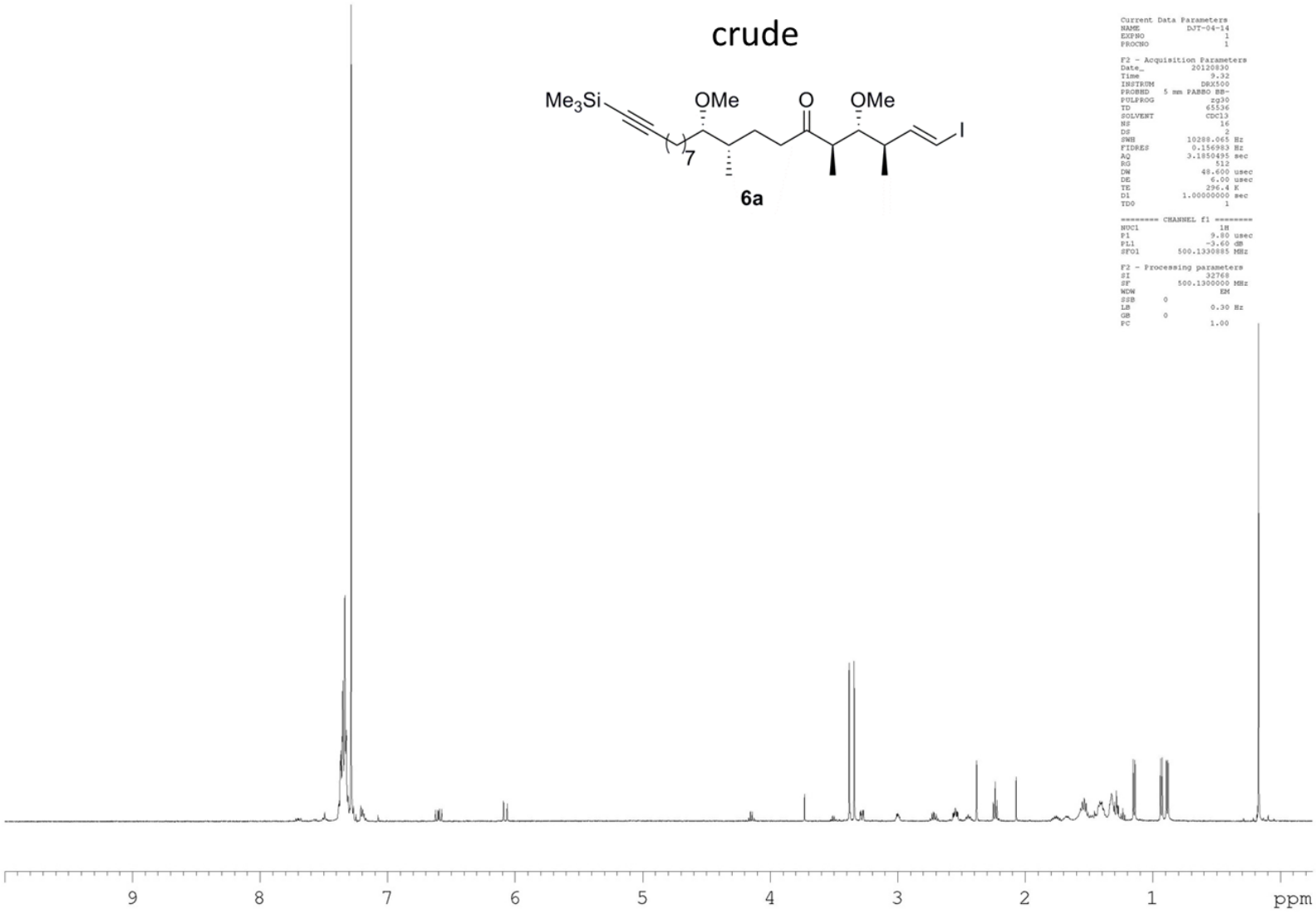
Current Data Parameters
NAME DJT-03-56-01
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120712
Time 13.33
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 71.8
DW 48.600 usec
DE 6.00 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -3.60 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 ppm
9 8 7 6 5 4 3 2 1 0 ppm

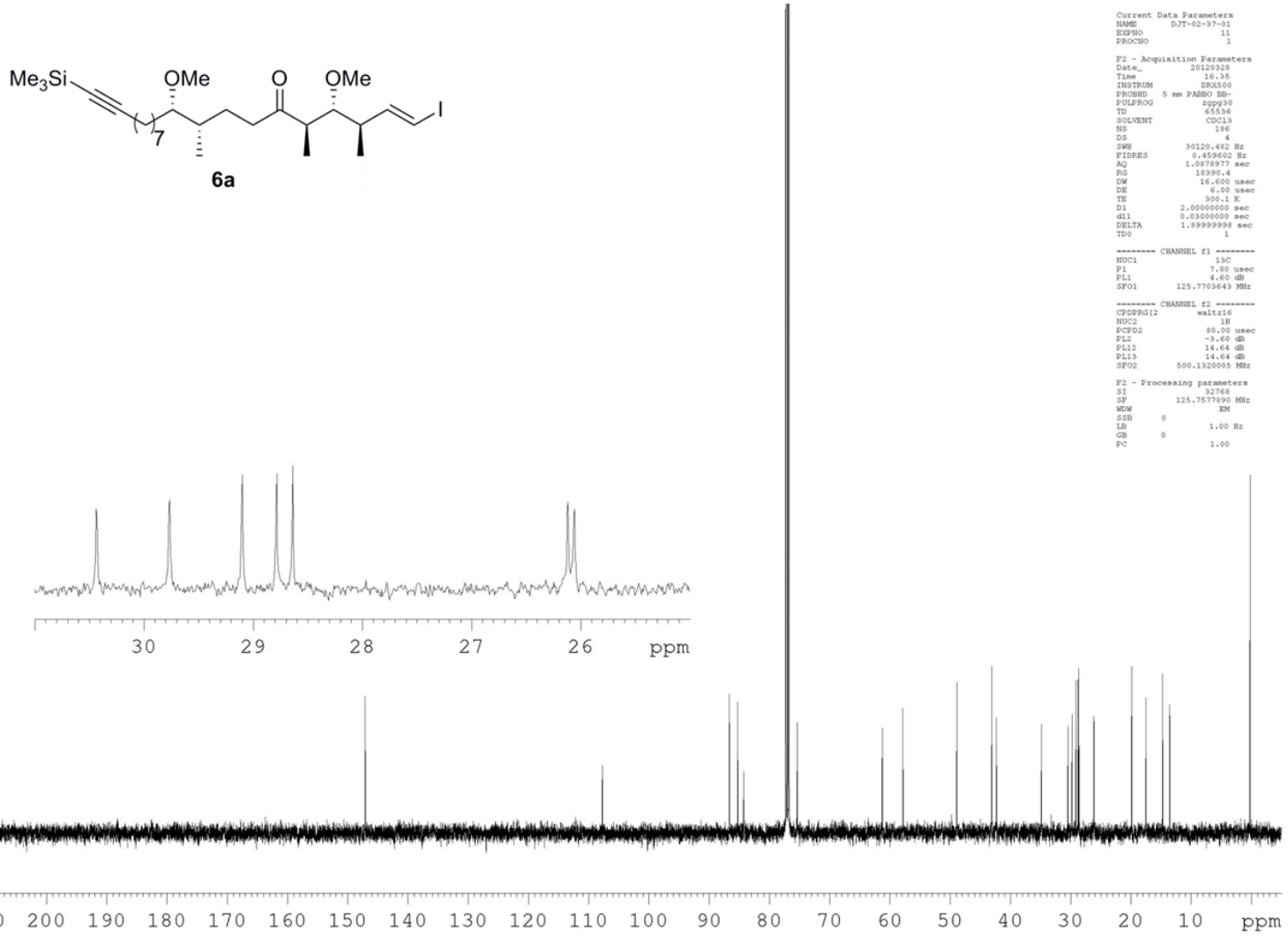

Me3Si
OMe
O
OMe
I
5

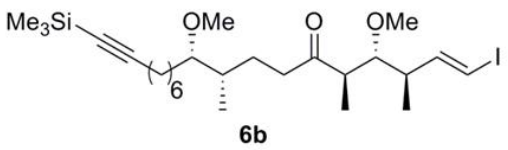
5c

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Current Data Parameters
NAME DJT-03-54-01
EXPNO 12
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120712
Time 17.43
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 40
DS 4
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0878977 sec
RG 16384
DW 16.600 usec
DE 6.00 usec
TE 297.9 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
d20 0.00689655 sec
DELTA 0.00000993 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
p2 15.60 usec
PL1 4.60 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.60 dB
PL12 14.66 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Me3Si
OMe
O
OMe
I
5
5c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

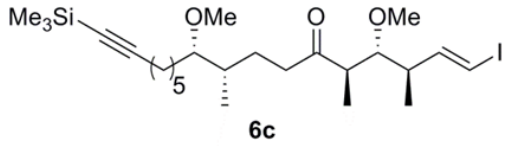

crude
Me3Si
OMe
O
OMe
I
7
6a
Current Data Parameters
NAME DJT-04-14
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120830
Time 9.32
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 512
DW 48.600 usec
DE 6.00 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -3.60 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm


Me3Si
OMe
O
OMe
I
7
6a
Current Data Parameters
NAME DJT-02-37-01
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120328
Time 16.35
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 186
DS 4
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0878977 sec
RG 18390.4
DW 16.600 usec
DE 6.00 usec
TE 300.1 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 7.80 usec
PL1 4.60 dB
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.60 dB
PL12 14.64 dB
PL13 14.64 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
30 29 28 27 26 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


Me3Si
OMe
O
OMe
I
6

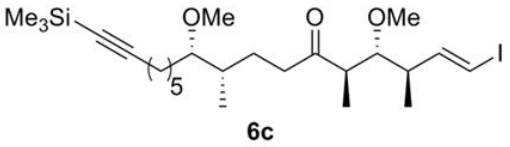
6b

Current Data Parameters
NAME DJT-04-23-01
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120910
Time 12.56
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 287.4
DW 48.600 usec
DE 6.00 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 1H
P1 9.80 usec
PL1 -3.60 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm


Me3Si
OMe
O
OMe
I
5

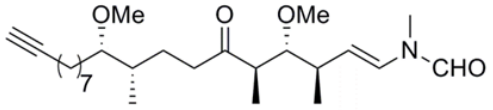
6c

Current Data Parameters
NAME DJT-03-73-01
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120730
Time 14.07
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 143.7
DW 48.600 usec
DE 6.00 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -3.60 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300077 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm


Me3Si
OMe
O
OMe
I
5

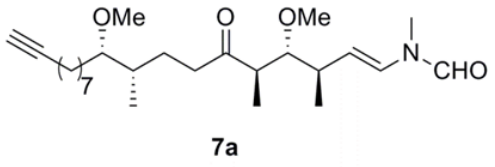
6c

Current Data Parameters
NAME DJT-03-73-01
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120730
Time 14.17
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 140
DS 4
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0878977 sec
RG 11585.2
DW 16.600 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
PL1 4.60 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.60 dB
PL12 14.64 dB
PL13 14.64 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577940 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


OMe
O
OMe
N
CHO
7

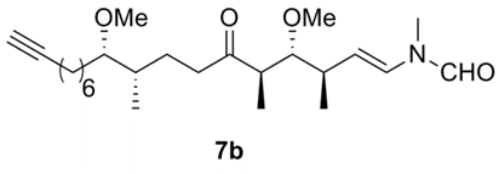
7a

Current Data Parameters
NAME DJT-04-21B-01
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120910
Time 15.04
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 114
DW 48.600 usec
DE 6.00 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.80 usec
PL1 -3.60 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm


OMe
O
OMe
N
CHO
7

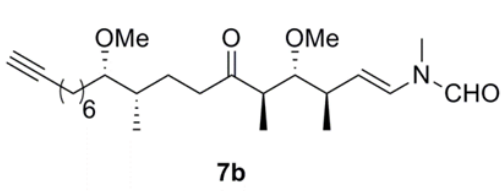
7a

Current Data Parameters
NAME DJT-04-21B-01
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120910
Time 15.16
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 427
DS 4
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0878977 sec
RG 9195.2
DW 16.600 usec
DE 6.00 usec
TE 299.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.80 usec
PL1 4.60 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.60 dB
PL12 14.64 dB
PL13 14.64 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577942 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


OMe
O
OMe
N
CHO
6

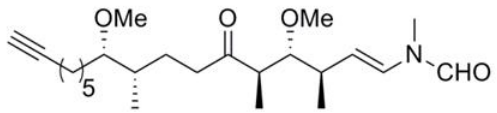
7b

Current Data Parameters
NAME DJT-04-24B-01TEST
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130422
Time 14.34
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10288.065 Hz
FIDRES 0.156983 Hz
AQ 3.1850495 sec
RG 143.7
DW 48.600 usec
DE 6.00 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.65 usec
PL1 -3.70 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300138 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm


OMe
O
OMe
N
CHO
6

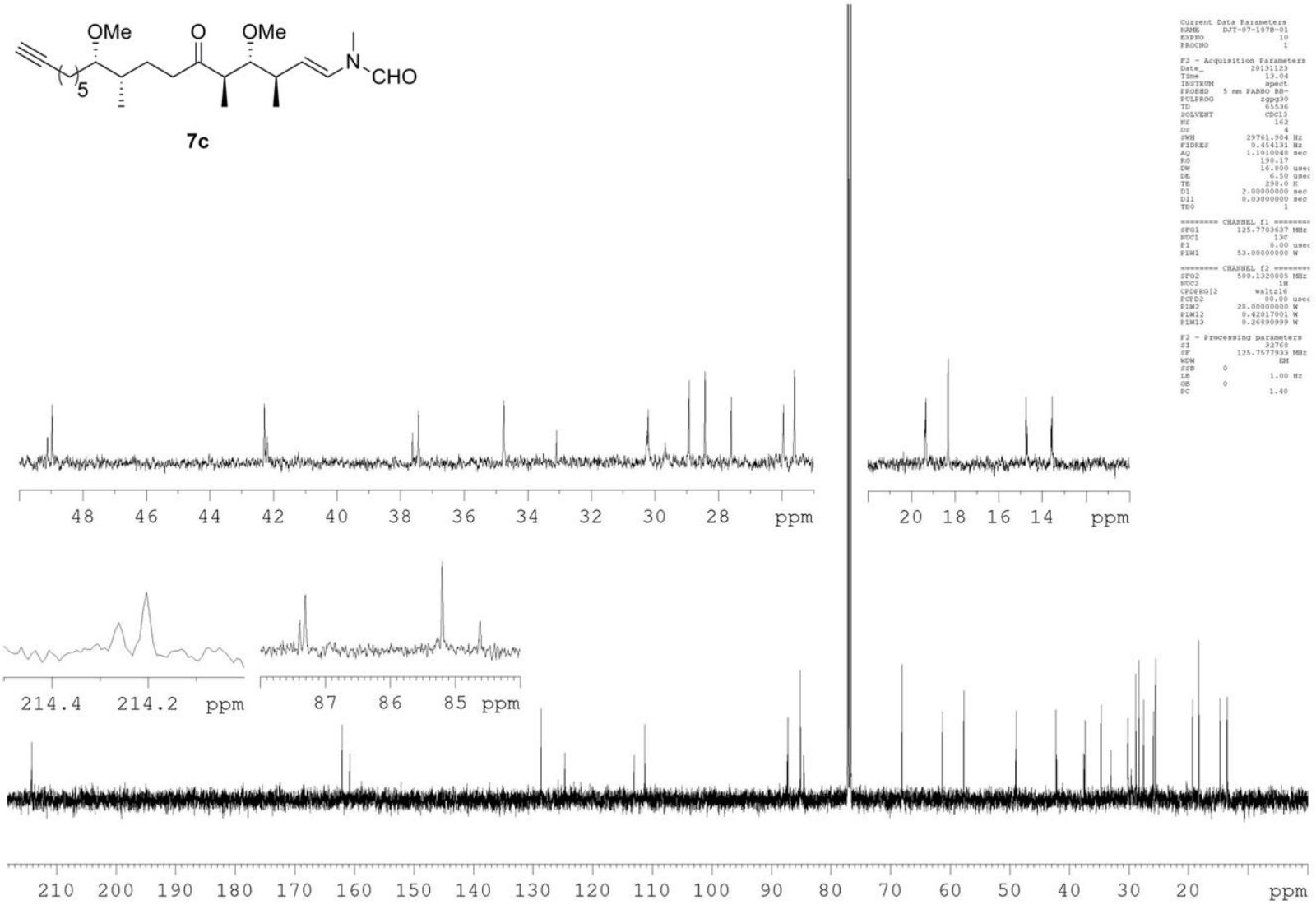
7b

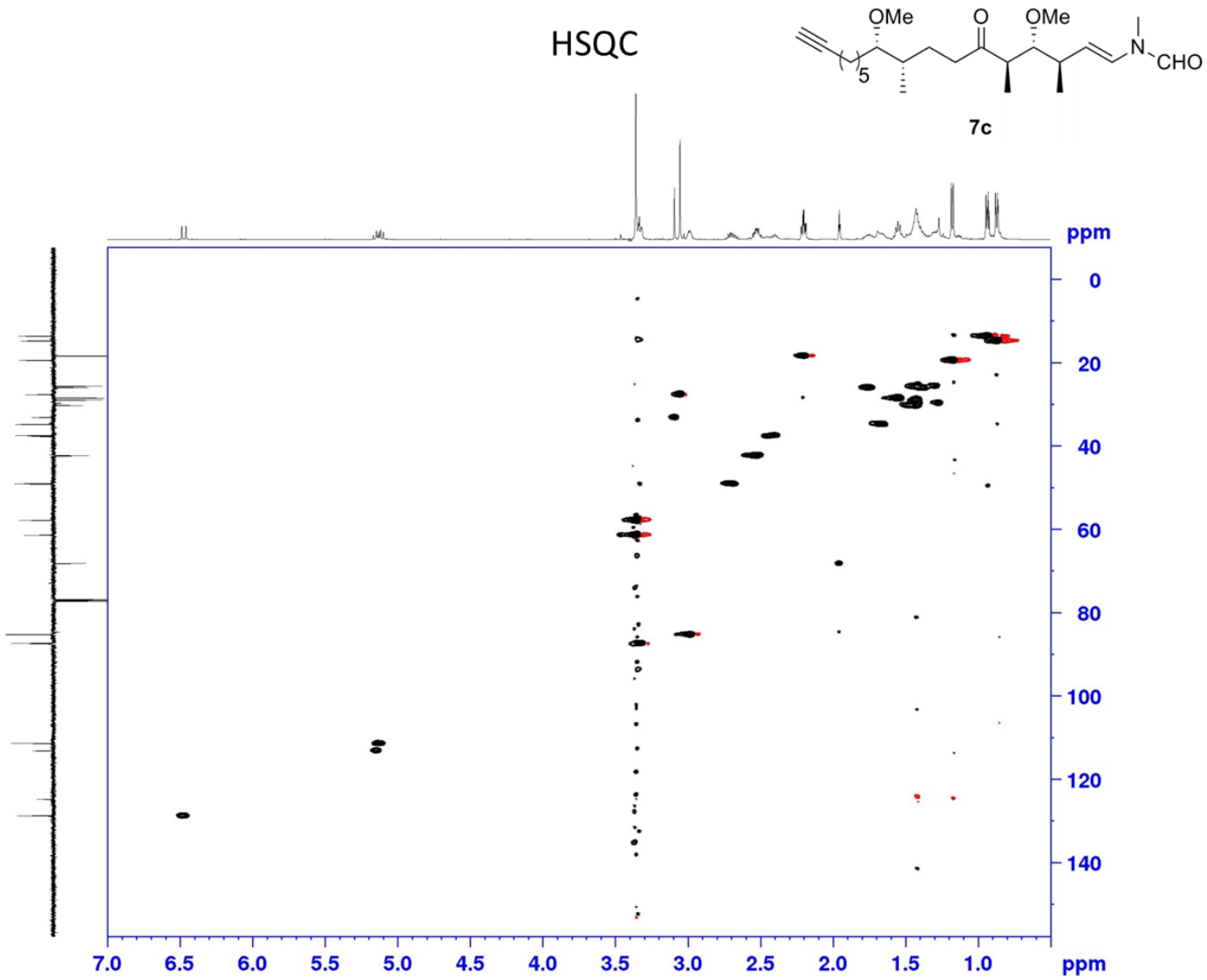
Current Data Parameters
NAME DJT-04-24B-01TEST
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130422
Time 14.51
INSTRUM DRX500
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 506
DS 4
SWH 30120.482 Hz
FIDRES 0.459602 Hz
AQ 1.0878977 sec
RG 5792.6
DW 16.600 usec
DE 6.00 usec
TE 296.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.25 usec
PL1 4.20 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG[2 waltz16
NUC2 1H
PCPD2 88.00 usec
PL2 -2.70 dB
PL12 14.65 dB
PL13 14.65 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


OMe
O
OMe
N
CHO
5

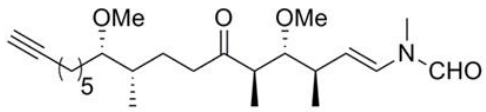
7c

Current Data Parameters
NAME DJT-07-107B-01
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131125
Time 10.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 106.33
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.00 usec
PLW1 26.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm

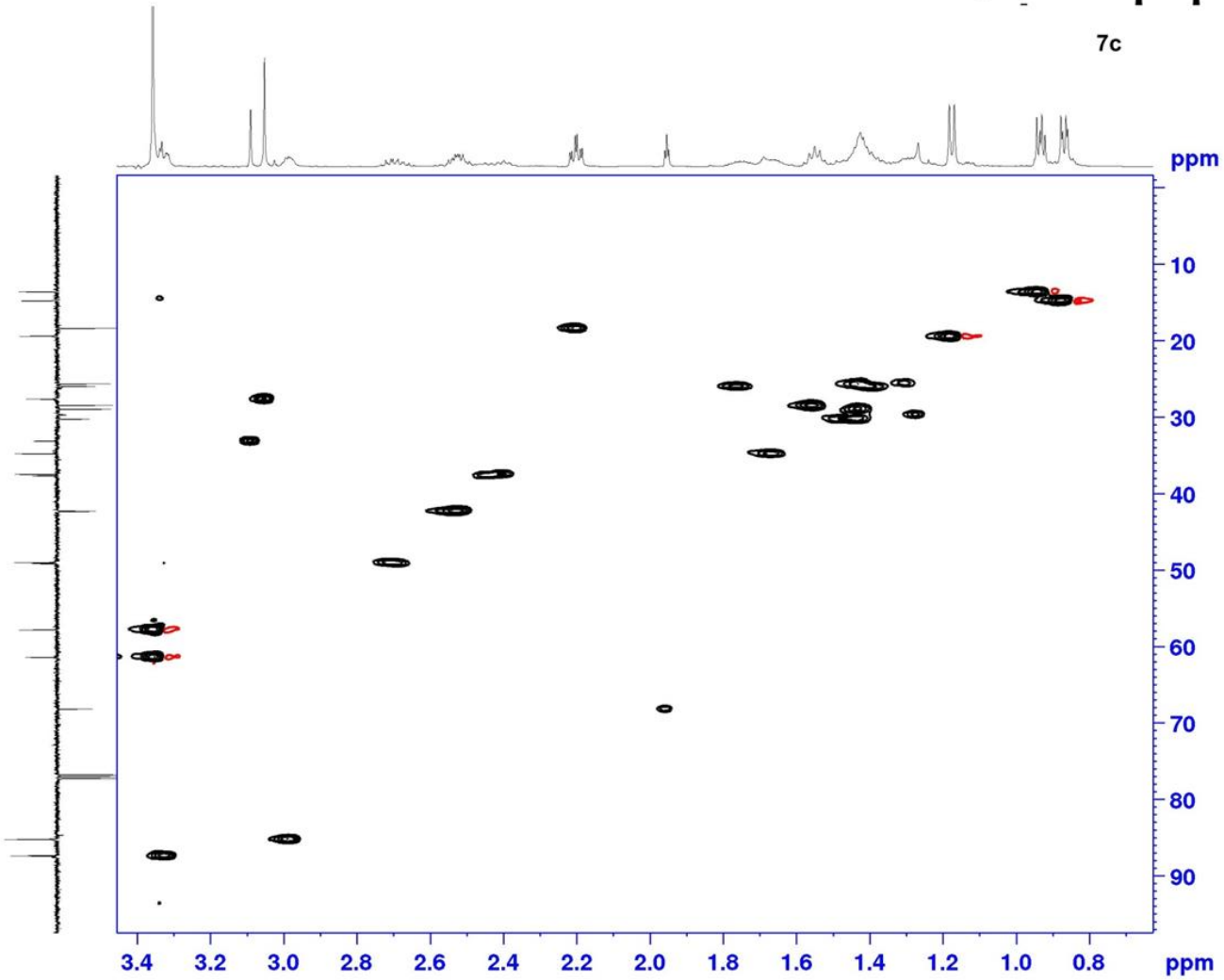

OMe
O
OMe
N
CHO
5
7c
Current Data Parameters
NAME DJT-07-107B-01
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131123
Time 13.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 162
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010048 sec
RG 198.17
DW 16.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 125.7703637 MHz
NUC1 13C
P1 8.00 usec
PLW1 53.00000000 W
======== CHANNEL f2 ========
SFO2 500.1320005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 80.00 usec
PLW2 28.00000000 W
PLW12 0.42017001 W
PLW13 0.26890999 W
F2 - Processing parameters
SI 32768
SF 125.7577933 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
48 46 44 42 40 38 36 34 32 30 28 ppm
20 18 16 14 ppm
214.4 214.2 ppm
87 86 85 ppm
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


HSQC
OMe
O
OMe
N
CHO
5
7c
ppm
0
20
40
60
80
100
120
140
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

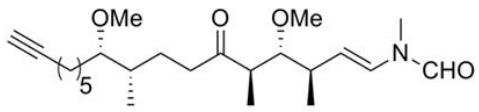
HSQC


OMe
O
OMe
N
CHO
5

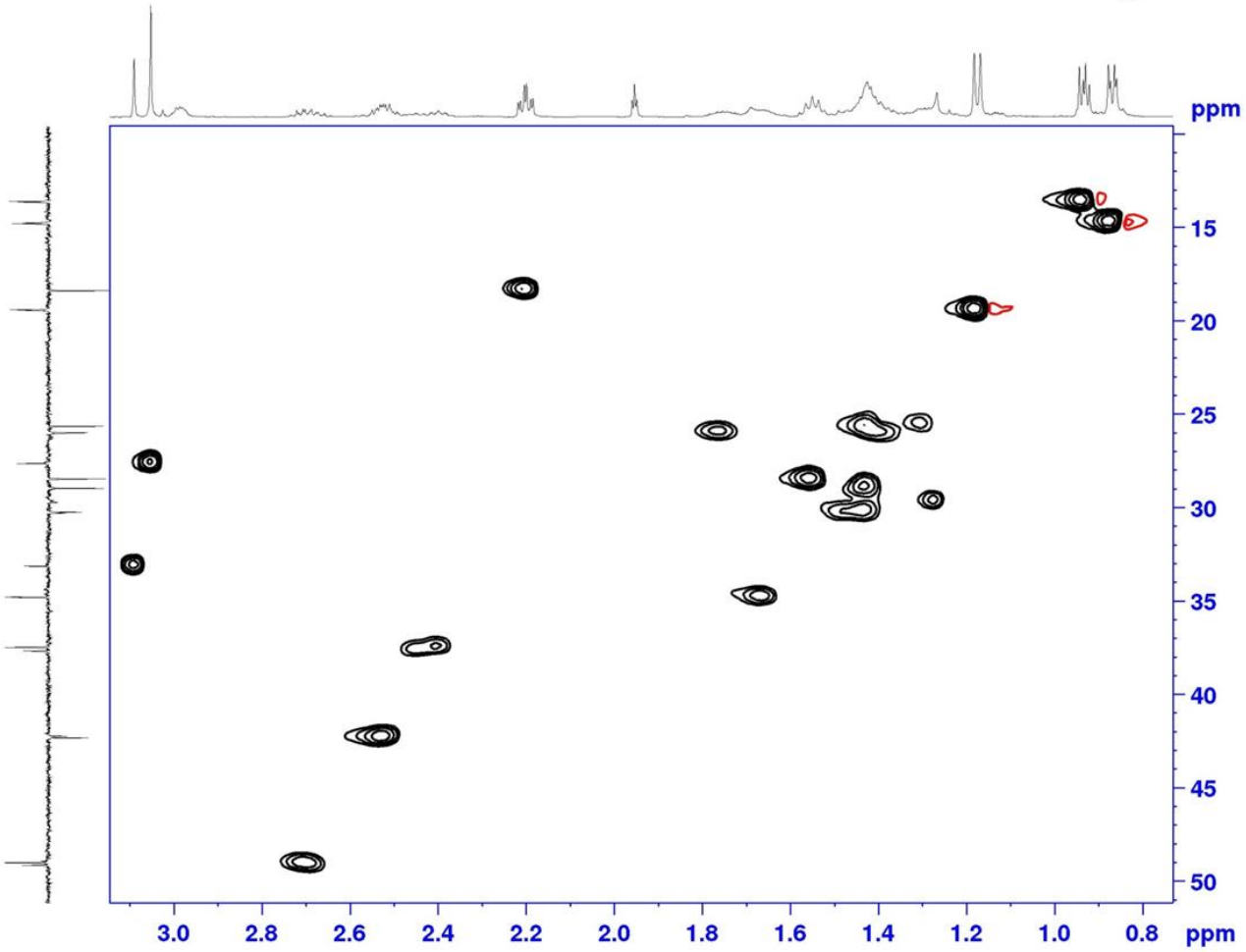
7c

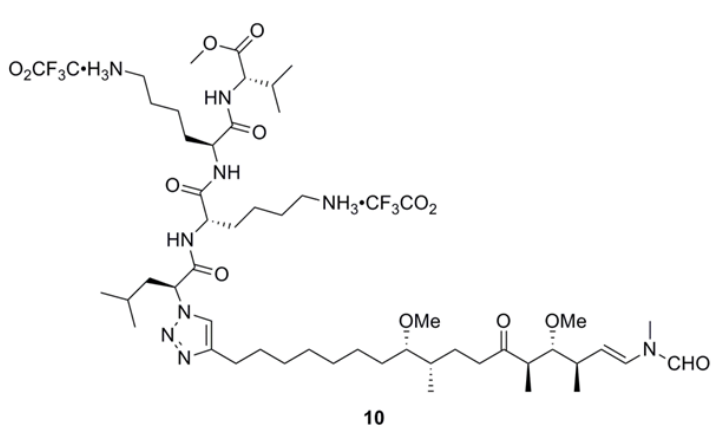

ppm
10
20
30
40
50
60
70
80
90
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
ppm

HSQC


OMe
O
OMe
N
CHO
5

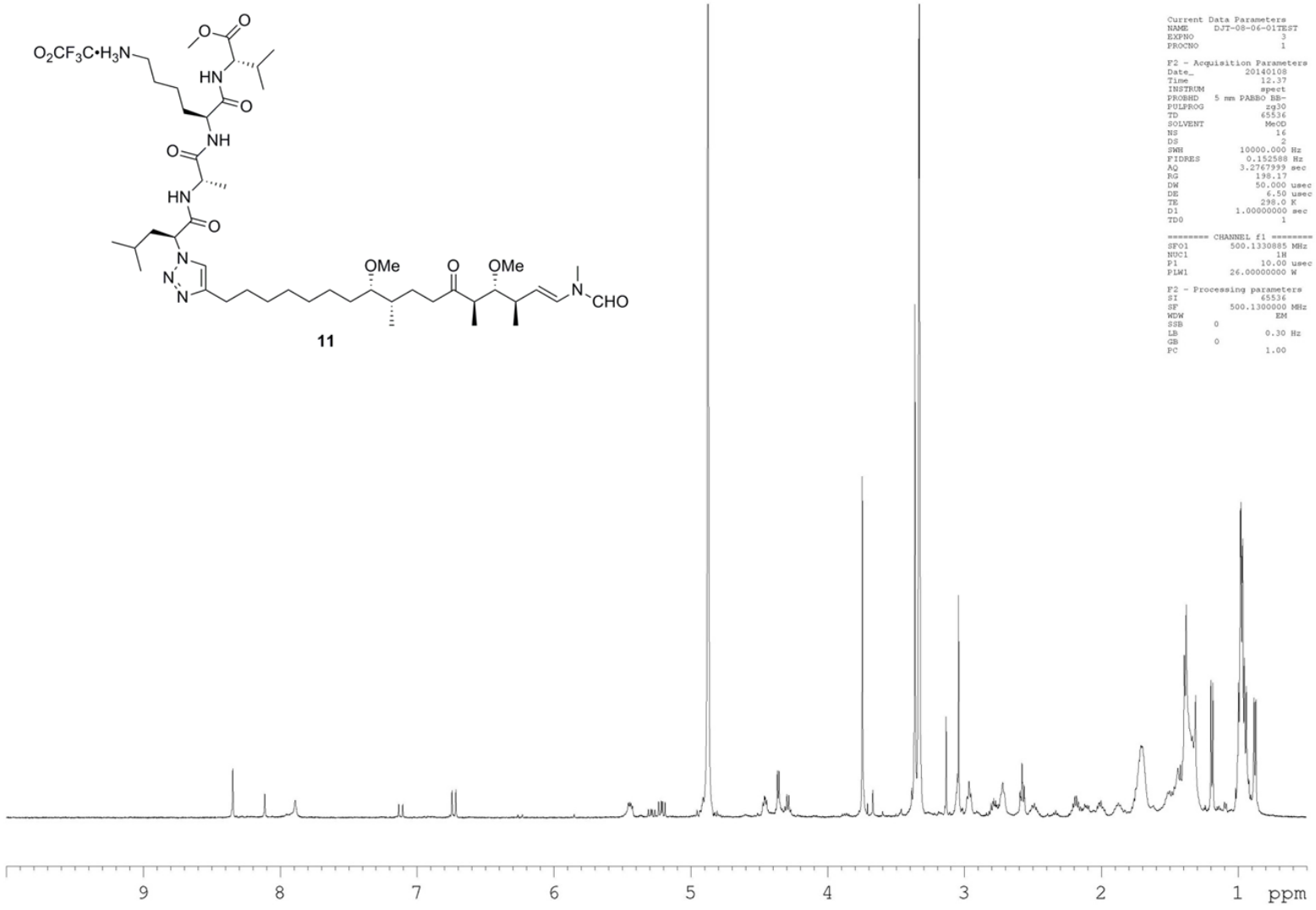
7c


ppm
15
20
25
30
35
40
45
50
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
ppm


O2CF3C•H3N
HN
O
NH
NH3•CF3CO2
OMe
OMe
N
CHO

10

Current Data Parameters
NAME DJT-08-02-01TEST
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131220
Time 17.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 198.17
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.00 usec
PLW1 26.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm


O2CF3C•H3N
HN
O
NH
HN
N
N
N
OMe
O
OMe
N
CHO
11
Current Data Parameters
NAME DJT-08-06-01TEST
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140108
Time 12.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2767999 sec
RG 198.17
DW 50.000 usec
DE 6.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 500.1330885 MHz
NUC1 1H
P1 10.00 usec
PLW1 26.00000000 W
F2 - Processing parameters
SI 65536
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
ppm