Supplementary Materials: Passerini Reactions on Biocatalytically Derived Chiral Azetidines

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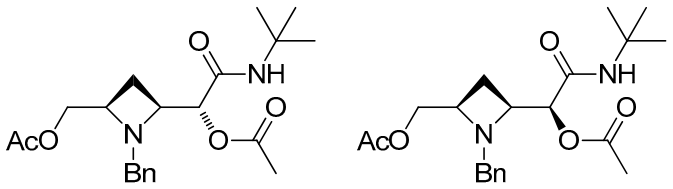
Experimental Procedures and Spectroscopic Characterization of New Compounds

1. General Remarks

NMR spectra were taken in $CDCl_3$ at 300 MHz or 400 MHz (1H), and 75 MHz or 100 MHz (^{13}C), using, as internal standard, TMS (^{1}H-NMR in $CDCl_3$; 0.000 ppm) or the central peak of $CDCl_3$ (^{13}C in $CDCl_3$; 77.02 ppm). Chemical shifts are reported in ppm (δ scale), coupling constants are reported in Hertz. Peak assignments were also made with the aid of gCOSY and gHSQC experiments. In an ABX system, the proton A is considered upfield and the B proton is considered downfield. IR spectra were recorded directly on solid, oil, or foamy samples, with the ATR (attenuated total reflectance) technique. TLC analyses were carried out on silica gel plates, viewed at UV (λ = 254 nm) and developed with Hanessian stain (dipping into a solution of $(NH_4)_4MoO_4{\cdot}4H_2O$ (21 g) and $Ce(SO_4)_2{\cdot}4H_2O$ (1 g) in H_2SO_4 (31 mL) and H_2O (469 mL) and warming), or with Iodine stain (leaving the TLC in a chamber containing solid iodine and leaving it for a few minutes until spots appear) or Dragendorff stain (dipping into a solution derived from mixing solution A of bismuth oxynitrate (1.7 g), acetic acid (40 mL) and water (80 mL) with solution B of potassium iodide (36 g) and water (100 mL); solution A:solution B mix is 1:1). R_f values were measured after an elution of 7–9 cm. HRMS were performed employing an ESI+ ionization method with a Thermo Fisher Scientific Orbitrap Elite (ion trap mode—high resolution) Velos Pro (ionisation) Ultimate 3000 (UPLC System for injection).

2. Characterization of All New Compounds



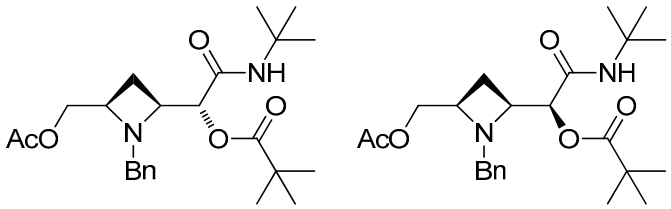
4a **5a**

(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-(tert-butylamino)-2-oxoethyl acetates **4a** *and* **5a**

4a: R_f = 0.51, *n*Hex/Et_2O 1:4 developed with Dragendorff stain; $[\alpha]_D$ = −69.3 (*c* = 0.45, $CHCl_3$); ^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.36–7.24 (m, 5H; H arom), 6.74 (broad s, 1H; NH), 4.79 (d, *J* = 4.3 Hz, 1H; C*H*OAc), 3.92 and 3.89 (AB part of ABX syst., J_{AB} = 11.2, J_{AX} = 5.4, J_{BX} = 6.5 Hz, 2H; CH_2OAc), 3.77 and 3.66 (AB syst., J_{AB} = 13.2 Hz, 2H; CH_2 of Bn), 3.57 (td, *J* = 8.1, 4.6 Hz, 1H; H-2), 3.36 (tt, *J* = 8.1, 6.3 Hz, 1H; H-4), 2.21 and 1.74 (AB part of ABX syst., J_{AB} = 10.4, J_{AX} = 7.9, J_{BX} = 7.9 Hz, 2H; CH_2-3), 2.10 (s, 3H; OAc), 1.92 (s, 3H; OAc), 1.35 (s, 9H; *t*Bu). ^{13}C-NMR (75 MHz, $CDCl_3$): δ 170.7 (C=O), 169.8 (C=O),

166.3(C=O), 137.6 (Cq), 129.1(2 CH arom), 128.5 (2 CH arom), 127.6 (CH arom), 73.5 (*C*HOAc), 67.4 (*C*H$_2$OAc), 61.7 (CH-2), 61.6 (CH$_2$ of Bn), 60.2 (CH-4), 51.4 (Cq), 28.7 (CH$_3$ of *t*Bu), 23.3 (CH$_2$-3), 20.8 (OAc), 20.7 (OAc). HRMS (ESI) calcd for $C_{21}H_{31}N_2O_5$ $[M + H]^+$ 391.2227; found 39.2230.

5a: R_f = 0.59, *n*Hex/Et$_2$O 1:4 developed with Dragendorff stain; $[\alpha]_D$ = −77.1 (*c* = 0.5, $CHCl_3$); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.32–7.20 (m, 5H; H arom), 5.98 (broad s, 1H; NH), 5.11 (d, *J* = 3.6 Hz, 1H; C*H*OAc), 3.81 and 3.50 (AB syst., J_{AB} = 12.9 Hz, 2H, CH$_2$ of Bn), 3.74 and 3.66 (AB part of ABX syst., J_{AB} = 11.3, J_{AX} = 5.7, J_{BX} = 5.7 Hz, 2H; C*H*$_2$OAc), 3.64 (dt, *J* = 8.1, 3.6 Hz, 1H; H-2), 3.27 (tt, *J* = 8.1, 6.0 Hz, 1H; H-4), 2.18 (s, 3H, OAc), 2.13–2.07 (m, 1H; H-3a), 1.88–1.79 (m, 1H; H-3b), 1.86 (s, 3H; OAc), 1.35 (s, 9H; *t*Bu). ^{13}C-NMR (75 MHz, $CDCl_3$) δ 170.8 (C=O), 169.3 (C=O), 166.7(C=O), 138.1 (Cq), 129.1(2 CH arom), 128.2 (2 CH arom), 127.3 (CH arom), 73.7 (*C*HOAc), 67.7 (*C*H$_2$OAc), 62.1 (CH-2), 60.9 (CH$_2$ of Bn), 59.7 (CH-4), 51.4 (Cq), 28.7 (CH$_3$ of *t*Bu), 21.6 (CH$_2$-3), 21.2 (OAc), 20.7 (OAc). IR: υ (cm^{-1}) 2967, 2874, 2803, 2159, 2025, 1969, 1738, 1673, 1523, 1480, 1454, 1366, 1216, 1088, 1032, 975, 916, 750, 699, 666. HRMS (ESI) calcd for $C_{21}H_{31}N_2O_5$ $[M + H]^+$ 391.2227; found 39.2229.

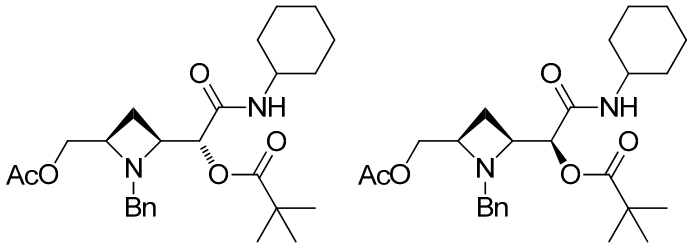


4b **5b**

(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-(tert-butylamino)-2-oxoethyl 2,2-dimethylpropanoates **4b** *and* **5b**

4b: R_f = 0.28, *n*Hex/Et$_2$O 5:4 developed with I$_2$; $[\alpha]_D$ = −3.85 (*c* = 1.9, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.40–7.19 (m, 5H; H arom), 6.40 (broad s, 1H; NH), 4.89 (d, *J* = 4.0 Hz, 1H; C*H*O(C=O)), 3.90–3.77 (m, 3H; CH$_2$OAc and 1 H of CH$_2$Ph), 3.71 (td, *J* = 8.2, 4.1 Hz, 1H; H-2), 3.59 (d, *J* = 13.6 Hz, 1H; 1 H of CH$_2$Ph), 3.32 (tt, *J* = 7.6, 6.4 Hz, 1H; H-4), 2.19 (dt, *J* = 11.0, 7.9 Hz, 1H; H-3a), 1.84 (s, 3H; OAc), 1.74 (dt, *J* = 11.0, 8.4 Hz, 1H; H-3b), 1.30 (s, 18H; 2 *t*Bu); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 177.1 (C=O), 170.7 (C=O), 166.7 (C=O), 137.7 (Cq), 129.0 (2 CH arom), 128.4 (2 CH arom), 127.4 (CH arom), 73.9 (*C*HO(C=O)), 67.6 (*C*H$_2$OAc), 61.31 (H-2), 61.29 (CH$_2$ of Bn), 60.2 (H-4), 51.3 (Cq), 38.9 (Cq), 28.6 (*t*Bu), 27.2 (*t*Bu), 23.0 (CH$_2$-3), 20.6 (OAc). IR: υ (cm^{-1}) 2967, 2934, 2873, 2161, 2025, 1737, 1683, 1515, 1495, 1480, 1455, 1394, 1365, 1227, 1138, 1034, 896, 752, 699, 666. HRMS (ESI) calcd for $C_{24}H_{37}N_2O_5$ $[M + H]^+$ 433.2697; found 433.2692.

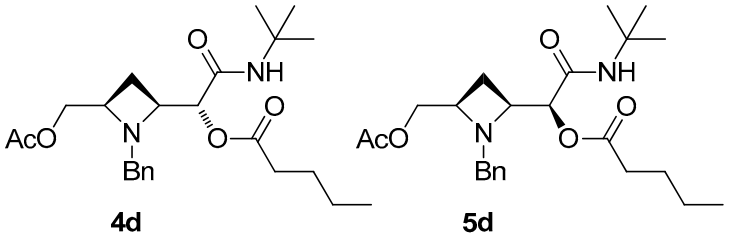
5b: R_f = 0.37, *n*Hex/Et$_2$O 5:4 developed with I$_2$; $[\alpha]_D$ = −39.7 (*c* = 1.6, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.35–7.16 (m, 5H; H arom), 5.98 (broad s, 1H; NH), 5.10 (d, *J* = 3.5 Hz, 1H; C*H*O(C=O)), 3.87 and 3.48 (AB syst., J_{AB} = 13.0 Hz, 2H; CH$_2$ of Bn), 3.76 and 3.64 (AB part of ABX syst., J_{AB} = 11.2, J_{AX} = 5.9, J_{BX} = 5.8 Hz, 2H; C*H*$_2$OAc), 3.67 (dt, *J* = 8.2, 3.5 Hz, 1H; H-2), 3.25 (tt, *J* = 7.7, 6.1 Hz, 1H; H-4), 2.10 (dt, *J* = 10.5, 7.7 Hz, 1H; H-3a), 1.89–1.80 (m, 1H; H-3b), 1.84 (s, 3H; OAc), 1.34 (s, 9H; *t*Bu), 1.31 (s, 9H; *t*Bu); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 176.5 (C=O), 170.9 (C=O), 167.1 (C=O), 138.3 (Cq), 129.3 (2 CH arom), 128.4 (2 CH arom), 127.4 (CH arom), 73.0 (CHO(C=O)), 67.8 (*C*H$_2$OAc), 62.4 (CH-2), 61.1 (CH$_2$ of Bn), 60.4 (CH-4), 51.4 (Cq), 39.0 (Cq), 28.8 (*t*Bu), 27.5 (*t*Bu), 22.3 (CH$_2$-3), 20.8 (OAc). IR: υ (cm^{-1}) 2967, 2934, 2873, 2161, 2025, 1737, 1683, 1515, 1495, 1480, 1455, 1394, 1365, 1227, 1138, 1034, 896, 752, 699, 666. HRMS (ESI) calcd for $C_{24}H_{37}N_2O_5$ $[M + H]^+$ 433.2697; found 433.2696.



(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-(cyclohexylamino)-2-oxoethyl 2,2-dimethylpropanoates **4c** *and* **5c**

4c: R_f = 0.35, *n*Hex/Et$_2$O 1:4 developed with Dragendorff stain; $[\alpha]_D$ = −9.6 (*c* = 2.2, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.39–7.18 (m, 5H; H arom of Bn), 6.48 (broad d, *J* = 7.6 Hz, 1H; NH), 4.94 (d, *J* = 4.1 Hz, 1H; CHO(C=O)), 3.91–3.78 (m, 2H; CH_2OAc), 3.82 and 3.60 (AB syst., J_{AB} = 13.6 Hz, 2H; CH_2 of Bn), 3.78–3.66 (m, 2H; H-2 and CH of Cy), 3.33 (centre of m, 1H; H-4), 2.18 (dt, *J* = 10.6, 7.9 Hz, 1H; H-3a), 1.96–1.55 (m, 5H; H-3b and 4H of Cy), 1.87 (s, 3H; OAc), 1.43–0.99 (m, 6H; Cy), 1.29 (s, 9H; *t*Bu); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 177.1 (C=O), 170.6 (C=O), 166.7 (C=O), 137.6 (Cq), 129.1 (2 CH arom), 128.4 (2 CH arom), 127.4 (CH arom), 73.6 (CHO(C=O)), 67.4 (CH_2OAc), 61.2 (CH-2), 60.0 (CH-4), 48.0 (CH of Cy), 38.9 (CH_2 of Bn), 33.1 (CH_2 of Cy), 32.8 (CH_2 of Cy), 27.3 (Cq), 27.2 (*t*Bu), 25.4 (CH_2 of Cy), 24.7 (CH_2 of Cy), 24.7 (CH_2 of Cy), 23.1 (CH_2-3), 20.6 (OAc). HRMS (ESI) calcd for $C_{26}H_{39}N_2O_5$ $[M + H]^+$ 459.2853; found 459.2851.

5c: R_f = 0.42, *n*Hex/Et$_2$O 1:4 developed with Dragendorff stain; $[\alpha]_D$ = −50.1 (*c* = 1.5, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.33–7.19 (m, 5H; H arom of Bn), 6.04 (broad d, *J* = 7.7 Hz, 1H; NH), 5.20 (d, *J* = 3.6 Hz, 1H; C*H*O(C=O)), 3.89 and 3.49 (AB syst., J_{AB} = 13.0 Hz, 2H; CH_2 of Bn), 3.84–3.73 (m, 1H; CH of Cy), 3.76 and 3.63 (AB part of ABX syst., J_{AB} = 11.3, J_{AX} = 6.0, J_{BX} = 5.9 Hz, 2H, CH_2OAc), 3.69 (td, *J* = 8.2, 3.6 Hz, 1H; H-2), 3.26 (tt, *J* = 7.6, 6.1 Hz, 1H; H-4), 2.10 (dt, *J* = 10.5, 7.7 Hz, 1H; H-3a), 1.96–1.80 (m, 3H; H-3b and 2H of Cy), 1.84 (s, 3H; OAc), 1.74–1.53 (m, 3H; Cy), 1.44–1.05 (m, 5H; Cy), 1.32 (s, 9H; *t*Bu); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 176.4 (C=O), 170.7 (C=O), 166.8 (C=O), 137.9 (Cq), 129.2 (2 CH arom), 128.2 (2 CH arom), 127.3 (CH arom), 72.6 (CHO(C=O)), 67.5 (CH_2OAc), 62.2 (CH-2), 60.8 (CH_2 of Bn), 60.3 (CH-4), 47.7 (CH of Cy), 38.9 (Cq), 33.1 (CH_2 of Cy), 32.8 (CH_2 of Cy), 27.3 (*t*Bu), 25.4 (CH_2 of Cy), 24.6 (CH_2 of Cy), 22.1 (CH_2 of Cy), 20.6 (OAc). IR: υ (cm^{-1}) 3437, 3329, 2932, 2855, 2364, 2160, 2024, 1971, 1732, 1666, 1519, 1479, 1452, 1396, 1366, 1232, 1135, 1032, 892, 751, 699, 666. HRMS (ESI) calcd for $C_{26}H_{39}N_2O_5$ $[M + H]^+$ 459.2853; found 459.2852.

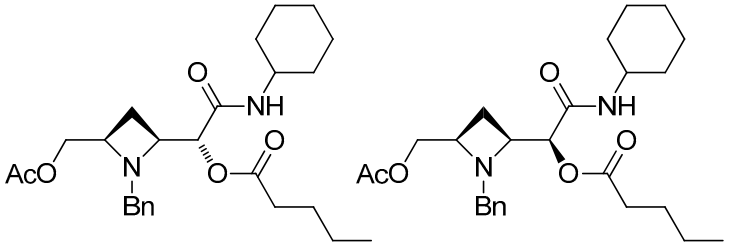


(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-(tert-butylamino)-2-oxoethyl pentanoates **4d** *and* **5d**

4d: R_f = 0.31, *n*Hex/Et$_2$O 1:1 developed with Dragendorff stain; $[\alpha]_D$ = −81.1 (*c* = 0.33, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.39–7.28 (m, 5H; H arom), 6.80 (broad s, 1H; NH), 4.95 (d, *J* = 4.2 Hz, 1H; C*H*O(C=O)), 3.99 (d, *J* = 5.8 Hz, 2H; CH_2OAc), 3.91and 3.73 (AB syst., J_{AB} = 13.5 Hz, 2H; CH_2 of Bn), 3.86–3.77 (m, 1H; H-2), 3.54–3.45 (m, 1H; H-4), 2.51–2.31 (m, 2H; CH_2 of *n*Bu), 2.21 (dt, *J* = 11.3, 8.1 Hz, 1H; H-3a), 1.94 (s, 1H; OAc), 1.92–1.81 (m, 1H; H-3b), 1.71–1.59 (m, 2H; CH_2 of *n*Bu), 1.46–1.29 (m, 2H; CH_2 of *n*Bu), 1.35 (s, 9H; *t*Bu), 0.92 (t, *J* = 7.4 Hz, 3H; CH_3 of *n*Bu.^{13}C-NMR (100 MHz, $CDCl_3$) δ 172.6

(C=O), 170.6 (C=O), 166.2 (C=O), 133.4 (Cq), 129.8 (2 CH arom), 128.7 (2 CH arom), 128.1 (CH arom), 73.3 (CHO(C=O)), 66.4 (CH_2OAc), 61.4 (CH), 60.4 (CH_2), 59.7 (CH), 51.6 (Cq), 33.7 (CH_2), 28.7 (*t*Bu), 26.9 (CH_2), 23.1 (CH_2), 22.2 (CH_2), 20.6 (CH_3), 13.7 (CH_3). IR: υ (cm^{-1}) 2962, 2933, 2873, 2159, 2024, 1969, 1739, 1681, 1523, 1454, 1392, 1365, 1226, 1166, 1108, 1094, 1036, 977, 907, 750, 699, 666. HRMS (ESI) calcd for $C_{24}H_{37}N_2O_5$ $[M + H]^+$ 433.2697; found 433.2694.

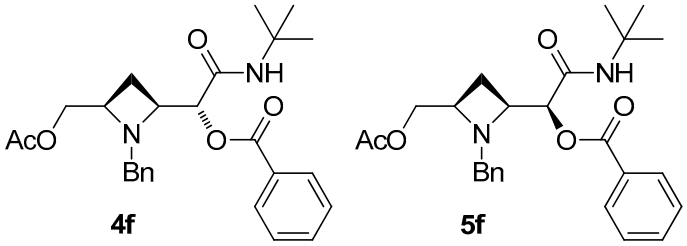
5d: R_f = 0.37, *n*Hex/Et_2O 1:1 developed with Dragendorff stain; $[\alpha]_D$ = −30.0 (*c* = 0.6, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.33–7.23 (m, 5H; CH arom), 6.04 (broad s, 1H; NH), 5.14 (d, *J* = 3.8 Hz, 1H; C*H*O(C=O)), 3.85 and 3.54 (AB syst., J_{AB} = 13.0 Hz, 2H; CH_2 of Bn), 3.78 (dd, *J* = 11.3, 5.7 Hz, 1H; 1 H of C*H*$_2$OAc), 3.74–3.64 (m, 2H; H-2 and 1 H of C*H*$_2$OAc), 3.37–3.26 (m, 1H; H-4), 2.42 (t, *J* = 7.5 Hz, 2H; CH_2 of *n*Bu), 2.13 (dt, *J* = 10.8, 7.9 Hz, 1H; H-3a), 1.93–1.82 (m, 1H; H-3b), 1.87 (s, 3H; OAc), 1.71–1.63 (m, 2H; CH_2 of *n*Bu), 1.46–1.36 (m, 2H; CH_2 of *n*Bu), 1.34 (s, 9H; *t*Bu), 0.95 (t, *J* = 7.3 Hz, 3H; CH_3 of *n*Bu); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 172.0 (C=O), 170.7 (C=O), 166.8 (C=O), 137.6 (Cq), 129.3 (2 CH arom), 128.3 (2 CH arom), 127.4 (CH arom), 73.3 (*C*HO(C=O)), 67.4 (*C*H$_2$OAc), 62.1 (H-2), 60.7 (CH_2 of Bn), 59.9 (H-4), 51.4 (Cq), 34.0 (CH_2 of *n*Bu), 28.7 (*t*Bu), 27.0 (CH_2 of *n*Bu), 22.3 (CH_2 of *n*Bu), 21.9 (CH_2-3), 20.7 (OAc), 13.7 (CH_3 of *n*Bu). HRMS (ESI) calcd for $C_{24}H_{37}N_2O_5$ $[M + H]^+$ 433.2697; found 433.2694.



(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-(cyclohexylamino)-2-oxoethyl pentanoates **4e** *and* **5e**

4e: R_f = 0.31, *n*Hex/Et_2O 1:1 developed with Dragendorff stain; $[\alpha]_D$ = −9.7 (*c* = 1.0, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.36–7.23 (m, 5H; H arom of Bn), 6.57 (broad d, *J* = 7.7 Hz, 1H; NH), 4.90 (d, *J* = 4.6 Hz, 1H; CHO(C=O)), 3.91 and 3.86 (AB part of ABX syst., J_{AB} = 11.4, J_{AX} = 5.5, J_{BX} = 6.4 Hz, 2H; CH_2OAc), 3.76 and 3.65 (AB syst., J_{AB} = 13.2 Hz, 2H; CH_2 of Bn), 3.77–3.69 (m, 1H), 3.66–3.59 (m, 1H), 3.34 (tt, *J* = 7.9, 6.2 Hz, 1H; H-4), 2.43–2.24 (m, 2H; CH_2 of *n*Bu), 2.20 (dt, *J* = 11.0, 7.8 Hz, 1H; H-3a), 1.92 (s, 3H; OAc), 1.98–1.81 (m, 2H; CH_2 of Cy), 1.75–1.55 (m, 6H; H-3b, CH_2 of *n*Bu, 3 H of Cy), 1.44–1.25 (m, 4H; CH_2 of *n*Bu and 2 H of Cy), 1.24–1.06 (m, 3H; 3 H of Cy), 0.91 (t, *J* = 7.3 Hz, 3H; CH_3 of *nBu*); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 172.7 (C=O), 170.7 (C=O), 166.5 (C=O), 137.6 (Cq), 129.1 (2 CH arom), 128.4 (2 CH arom), 127.5 (CH arom), 73.8 (CHO(C=O)), 67.4 (CH_2OAc), 61.6 (CH-2), 61.4 (CH_2 of Bn), 60.0 (CH-4), 48.2 (CH of Cy), 33.8 (CH_2), 33.2 (CH_2), 32.9 (CH_2), 27.0 (CH_2), 25.5 (CH_2), 24.8 (CH_2), 23.2 (CH_2), 22.2 (2 CH_2), 20.7 (OAc), 13.7 (CH_3 of *n*Bu). IR: υ (cm^{-1}) 3301, 2931, 2855, 2363, 2159, 2025, 1738, 1657, 1530, 1452, 1367, 1231, 1164, 1093, 1033, 970, 891, 751, 699, 666. HRMS (ESI) calcd for $C_{26}H_{39}N_2O_5$ $[M + H]^+$ 459.2853; found 459.2856.

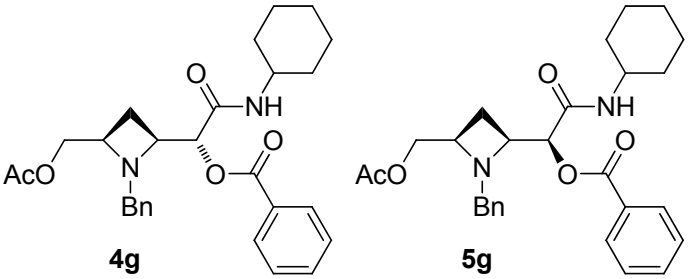
5e: R_f = 0.37, *n*Hex/Et_2O 1:1 developed with Dragendorff stain; $[\alpha]_D$ = −33.9 (*c* = 0.8, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.32–7.18 (m, 5H; H arom of Bn), 6.01 (broad d, *J* = 8.0 Hz, 1H; NH), 5.23 (d, *J* = 3.5 Hz, 1H; CHO(C=O)), 3.83 and 3.50 (AB syst., J_{AB} = 12.9 Hz, 2H; CH_2 of Bn), 3.81–3.70 (m, 2H), 3.65 (m, 2H), 3.33–3.19 (m, 1H; H-4), 2.43 (t, *J* = 7.4 Hz, 2H; CH_2 of *n*Bu), 2.10 (dt, *J* = 10.7, 7.9 Hz, 1H; H-3a), 1.96–1.79 (m, 3H; H-3b, 2 H of Cy), 1.86 (s, 3H; OAc), 1.76–1.56 (m, 5H; CH_2 of *n*Bu, 3 H of Cy), 1.48–1.30 (m, 4H; CH_2 of *n*Bu, 2 H of Cy), 1.25–1.04 (m, 3H; Cy), 0.95 (t, *J* = 7.3 Hz, 3H; CH_3 of *n*Bu); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 172.0 (C=O), 170.8 (C=O), 166.7 (C=O), 138.2 (Cq), 129.2 (2 CH arom), 128.2 (2 CH arom), 127.2 (CH arom), 73.1 (CHO(C=O)), 67.6 (CH_2OAc), 62.2 (CH-2), 60.9 (CH_2 of Bn), 59.8 (CH-4), 47.9 (CH of Cy), 34.0 (CH_2), 33.1 (CH_2), 32.9 (CH_2), 27.0 (CH_2), 25.5 (CH_2), 24.7 (CH_2), 22.3 (2 CH_2), 21.7 (CH_2), 20.6 (OAc), 13.7 (CH_3). HRMS (ESI) calcd for $C_{26}H_{39}N_2O_5$ $[M + H]^+$ 459.2853; found 459.2857.



(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-(tert-butylamino)-2-oxoethyl benzoates **4f** *and* **5f**

4f: R_f = 0.41, *n*Hex/Et_2O 1:1 developed with Dragendorff stain; $[\alpha]_D$ = −23.3 (*c* = 1.7, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 7.4 Hz, 2H; H arom), 7.59 (t, *J* = 7.4 Hz, 1H; H arom), 7.47 (t, *J* = 7.4 Hz, 2H; H arom), 7.35–7.20 (m, 5H; H arom), 6.7 (broad s, 1H; NH), 5.08 (d, *J* = 4.1 Hz, 1H; CHOBz), 3.89 (d, *J* = 5.9 Hz, 2H; CH_2OAc), 3.84 and 3.68 (AB syst., J_{AB} = 13.2 Hz, 2H; CH_2 of Bn), 3.78 (td, *J* = 8.1, 4.1 Hz, 1H; H-2), 3.38 (tt, *J* = 7.9, 6.0 Hz, 1H; H-4), 2.27 (dt, *J* = 11.1, 7.9 Hz, 1H; H-3a), 1.91–1.80 (m, 3H; H-3b), 1.87 (s, 3H; OAc), 1.34 (s, 9H; *t*Bu); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 170.7 (C=O), 166.4 (C=O), 165.4 (C=O), 137.7 (Cq), 133.4 (CH arom), 129.8 (2 CH arom), 129.4 (Cq), 129.1 (2 CH arom), 128.6 (2 CH arom), 128.5 (2 CH arom), 127.6 (CH arom), 74.1 (CHOBz), 67.4 (CH_2OAc), 61.7 (CH-2), 61.6 (CH_2 of *n*Bu), 60.3 (CH-4), 51.4 (Cq), 28.7 (*t*Bu), 23.2 (CH_2-3), 20.6 (OAc). HRMS (ESI) calcd for $C_{26}H_{33}N_2O_5$ $[M + H]^+$ 453.2384; found 433.2381.

5f: R_f = 0.49, *n*Hex/Et_2O 1:1 developed with Dragendorff stain; $[\alpha]_D$ = −40.9 (*c* = 1.7, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.2 Hz, 2H; H arom), 7.63 (t, *J* = 7.4 Hz, 1H; H arom), 7.51 (t, *J* = 7.7 Hz, 2H; H arom), 7.24–7.18 (m, 5H; H arom of Bn), 6.11 (broad s, 1H; NH), 5.35 (d, *J* = 3.6 Hz, 1H; CHOBz), 3.89 and 3.53 (AB syst., J_{AB} = 13.1 Hz, 2H; CH_2 of Bn), 3.79 (td, *J* = 8.1, 3.6 Hz, 1H; H-2), 3.73 and 3.56 (AB part of ABX syst., J_{AB} = 11.3, J_{AX} = 5.7, J_{BX} = 5.9 Hz, 2H, CH_2OAc), 3.28 (tt, *J* = 7.8, 5.8 Hz, 1H; H-4), 2.19 (dt, *J* = 10.6, 7.8 Hz, 1H; H-3a), 2.00 (dt, *J* = 10.6, 8.3 Hz, 1H; H-3b), 1.77 (s, 3H; OAc), 1.36 (s, 9H; *t*Bu); ^{13}C-NMR (75 MHz, $CDCl_3$) δ 170.7 (C=O), 166.8 (C=O), 164.9 (C=O), 138.1 (Cq), 133.5 (CH arom), 129.7 (Cq), 129.7 (2 CH arom), 129.0 (2 CH arom), 128.7 (2 CH arom), 128.2 (2 CH arom), 127.2 (CH arom), 74.1 (CHOBz), 67.5 (CH_2OAc), 62.3 (CH-2), 60.8 (CH_2 of Bn), 60.0 (CH-4), 51.4 (Cq), 28.7 (*t*Bu), 22.0 (CH_2-3), 20.6 (OAc). IR: υ (cm^{-1}) 3436, 3342, 3063, 2967, 2874, 2362, 2159, 2025, 1723, 1674, 1520, 1452, 1393, 1365, 1226, 1176, 1095, 1069, 1026, 976, 907, 753, 709, 666. HRMS (ESI) calcd for $C_{26}H_{33}N_2O_5$ $[M + H]^+$ 453.2384; found 433.2383.

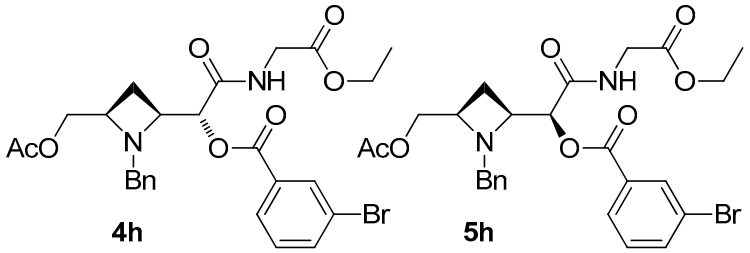


(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-(cyclohexylamino)-2-oxoethyl benzoates **4g** *and* **5g**

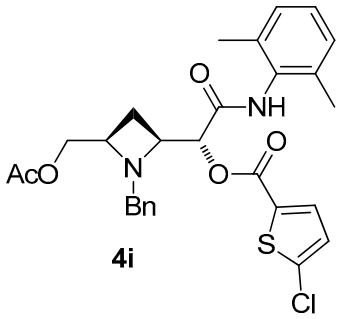
4g: R_f = 0.32, *n*Hex/Et_2O 1:4 developed with Dragendorff stain; $[\alpha]_D$ = −17.2 (*c* = 0.5, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 7.4 Hz, 2H; H arom of Bz), 7.60 (t, *J* = 7.4 Hz, 1H; H arom of Bz), 7.47 (t, *J* = 7.4 Hz, 2H; H arom of Bz), 7.33–7.18 (m, 5H; H arom of Bn), 6.57 (broad d, *J* = 7.7 Hz, 1H; NH), 5.16 (d, *J* = 4.3 Hz, 1H; CHOBz), 3.82 and 3.67 (AB syst., *J* = 13.3 Hz, 2H; CH_2 of Bn), 3.92–3.71 (m, 4H; CH_2OAc, H-2 and CH of Cy), 3.42–3.32 (m, 1H; H-4), 2.26 (dt, *J* = 11.1, 7.9 Hz, 1H; H-3a), 1.98–1.80 (m, 2H; H of Cy), 1.88 (s, 3H; OAc), 1.74–1.54 (m, 4H; H of Cy), 1.40–1.10 (m, 4H; H of Cy); ^{13}C-NMR

(100 MHz, $CDCl_3$) δ 170.7 (C=O), 166.5 (C=O), 165.5 (C=O), 137.5 (Cq), 133.5 (CH arom), 129.8 (2 CH arom), 129.4 (Cq), 129.2 (2 CH arom), 128.6 (2 CH arom), 128.5 (2 CH arom), 127.5 (CH arom), 74.4 (CHOAc), 67.4 (CH_2OBz), 61.6 (CH), 61.5 (CH_2 of Bn), 60.1 (CH), 48.3 (CH-4), 33.1 (CH_2), 32.9 (CH_2), 29.7 (CH_2), 25.5 (CH_2), 24.8 (CH_2), 23.2 (CH_2), 20.7 (OAc). IR: υ (cm^{-1}) 3305, 2931, 2854, 2362, 1722, 1658, 1602, 1531, 1451, 1366, 1316, 1240, 1176, 1096, 1069, 1027, 966, 891, 844, 803, 750, 708, 666. HRMS (ESI) calcd for $C_{28}H_{35}N_2O_5$ $[M + H]^+$ 479.2540; found 479.2537.

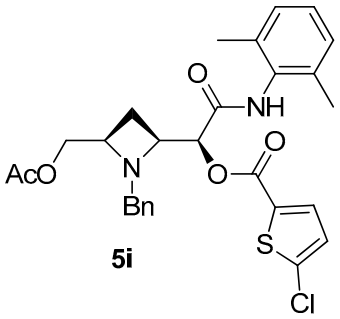
5g: R_f = 0.38, *n*Hex/Et_2O 1:4 developed with Dragendorff stain; $[\alpha]_D$ = −12.7 (*c* = 0.4, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.13–8.06 (m, 2H, H arom of Bz), 7.64 (t, *J* = 7.4 Hz, 1H; H arom of Bz), 7.51 (t, *J* = 7.7 Hz, 2H; H arom of Bz), 7.21 (broad s, 5H; H arom of Bn), 6.14 (broad d, *J* = 7.1 Hz, 1H; NH), 5.45 (d, *J* = 3.3 Hz, 1H; CHOBz), 3.90 and 3.54 (AB syst., J_{AB} = 13.0 Hz, 2H; CH_2 of Bn), 3.86–3.76 (m, 2H), 3.72 (dd, *J* = 11.3, 5.6 Hz, 1H), 3.57–3.48 (m, 1H); 3.33–3.20 (m, 1H, H-4), 2.18 (dt, *J* = 10.4, 7.8 Hz, 1H; H-3a), 2.06–1.82 (m, 3H; H-3b and 2 H of Cy), 1.77 (s, 3H; OAc), 1.75–1.51 (m, 2H; H of Cy), 1.47–1.02 (m, 6H; H of Cy); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 170.7 (C=O), 166.6 (C=O), 165.0 (C=O), 138.0 (Cq), 133.6 (CH arom), 129.7 (2 CH arom), 129.7 (Cq), 129.1 (2 CH arom), 128.7 (2 CH arom), 128.2 (2 CH arom), 127.2 (CH arom), 73.1 (CHOBz), 67.4 (CH_2OAc), 62.3 (CH-2), 60.7 (CH_2 of Bn), 60.0 (CH-4), 48.1 (CH of Cy), 33.2 (CH_2), 32.9 (CH_2), 25.5 (CH_2), 24.8 (CH_2), 24.8 (CH_2), 22.0 (CH_2), 20.6 (OAc). HRMS (ESI) calcd for $C_{28}H_{35}N_2O_5$ $[M + H]^+$ 479.2540; found 479.2539.



(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-((2-ethoxy-2-oxoethyl)amino)-2-oxoethyl 3-bromobenzoates **4h** and **5h**: R_f = 0.44, PE/Et_2O 1:4 developed with I_2 (in this case the two isomers were not separated; characterizations have been made on the mixture); ^{1}H-NMR (300 MHz, $CDCl_3$) (Selected data of mixture of isomers **4h**(M) + **5h**(m)) δ 8.25 (t, *J* = 1.7 Hz, 1H; H arom (m)), 8.16 (t, *J* = 1.7 Hz, 1H; H arom (M)), 8.07–8.02 (m, 1H; H arom (m)), 8.01–7.95 (m, 1H; H arom (M)), 7.88 (broad t, *J* = 5.2 Hz, 1H; NH (M)), 7.76 (ddd, *J* = 8.1, 1.9, 1.1 Hz, 1H; H arom (m)), 7.69 (ddd, *J* = 8.1, 1.9, 1.1 Hz, 1H; H arom (M)), 7.40 (t, *J* = 7.9 Hz, 1H; H arom (m)), 6.87 (broad t, *J* = 5.1 Hz, 1H; NH (m)), 5.47 (d, *J* = 3.4 Hz, 1H; CHO(C=O) (m)), 5.04 (d, *J* = 4.4 Hz, 1H; CHO(C=O) (M)), 1.96 (s, 3H; OAc (M)), 1.80 (s, 3H; OAc (m)), 1.28 (t, *J* = 7.1 Hz, 6H; CH_3 of Et (M + m)); ^{13}C-NMR (75 MHz, $CDCl_3$, mixture of isomers M + m) δ 170.9 (C=O (M)), 170.7 (C=O (m)), 169.5 (C=O (M)), 169.3 (C=O (m)), 167.6 (C=O (m)), 167.4 (C=O (M)), 164.0 (C=O (M)), 163.7 (C=O (m)), 137.7 (Cq (m)), 137.1 (Cq (M)), 136.6 (CH (m)), 136.3 (CH (M)), 132.8 (CH (M)), 132.7 (CH (m)), 131.3 (Cq (m)), 131.1 (Cq (M)), 130.3 (CH (m)), 130.0 (CH (M)), 129.1 (2 CH (M)), 129.0 (2 CH (m)), 128.6 (2 CH (M)), 128.4 (2 CH (m)), 128.24 (CH (m)), 128.22 (CH (M)), 127.7 (CH (M)), 127.3 (CH (m)), 122.8 (Cq (m)), 122.5 (Cq (M)), 73.9 (CHO(C=O) (m)), 73.4 (CHO(C=O) (M)), 67.2 (CH_2OAc (m)), 66.9 (CH_2OAc (M)), 62.1 (CH-2 (m)), 61.8 (CH_2 of Bn (M)), 61.7 (CH_2 of Et (m)), 61.5 (CH_2 of Et (M)), 61.5 (CH-2 (M)), 60.6 (CH_2 of Bn (m)), 60.4 (CH-4 (M)), 59.8 (CH-4 (m)), 41.2 (CH_2NH (M)), 41.0 (CH_2NH (m)), 22.9 (CH_2-3 (M)), 21.8 (CH_2-3 (m)), 20.8 (OAc (M)), 20.6 (OAc (m)), 14.1 (CH_3 of Et (M + m)). IR: υ (cm^{-1}) 3309, 2959, 1731, 1663, 1570, 1537, 1494, 1454, 1438, 1422, 1374, 1334, 1305, 1288, 1254, 1236, 1207, 1169, 1137, 1126, 1083, 1068, 1034, 1000, 974, 913, 898, 887, 863, 841, 807, 770, 747, 728, 702, 672, 637, 615, 606. HRMS (ESI) calcd for $C_{26}H_{30}BrN_2O_7$ $[M + H]^+$ 561.1236; found 561.1244.



4i

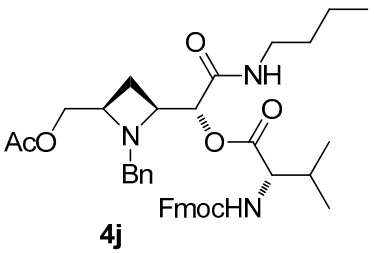


5i

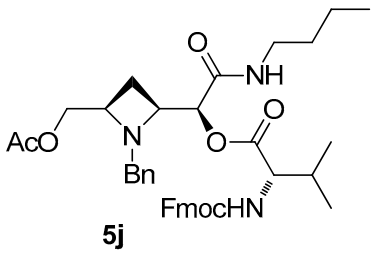
(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-((2,6-dimethylphenyl)amino)-2-oxoethyl 5-chlorothiophene-2-carboxylates **4i** *and* **5i**

4i: R_f = 0.25, PE/Et_2O 1:1 developed with Hanessian stain; $[\alpha]_D$ = +6.6 (*c* = 0.87, $CHCl_3$); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.77 (broad s, 1H; NH), 7.63 (d, *J* = 4.1 Hz, 1H; CH thiophene), 7.34–7.17 (m, 5H; H arom), 7.13–7.00 (m, 3H; H arom), 6.94 (d, *J* = 4.1 Hz, 1H; CH thiophene), 5.32 (d, *J* = 6.2 Hz, 1H; CHO(C=O)), 3.94 and 3.87 (AB part of ABX syst., J_{AB} = 11.5, J_{AX} = 5.0, J_{BX} = 5.8 Hz, 2H; CH_2OAc), 3.90 and 3.75 (AB syst., J_{AB} = 13.0 Hz, 2H; CH_2 of Bn), 3.85–3.78 (m, 1H; H-2), 3.41 (tt, *J* = 8.1, 5.4 Hz, 1H; H-4), 2.33 (dt, *J* = 11.0, 7.8 Hz, 1H; H-3a), 2.18 (s, 6H; 2 CH_3), 2.08 (dt, *J* = 11.0, 8.1 Hz, 1H; H-3b), 1.86 (s, 3H; OAc). ^{13}C-NMR (75 MHz, $CDCl_3$) δ 170.8 (C=O), 165.4 (C=O), 160.1 (C=O), 138.5 (Cq), 137.4 (Cq), 135.2 (2 Cq), 134.1 (CH arom), 132.9 (Cq), 130.3 (Cq), 129.1 (2 CH arom), 128.5 (2 CH arom), 128.3 (2 CH arom), 127.6 (CH arom), 127.5 (CH arom), 127.49 (CH arom), 76.5 (CHO(C=O)), 67.0 (CH_2OAc), 61.6 (CH_2 of Bn), 61.4 (CH-2), 60.2 (CH-4), 23.5 (CH_2-3), 20.6 (OAc), 18.6 (2 CH_3). IR: υ (cm^{-1}) 3270, 3028, 2958, 2870, 1715, 1663, 1629, 1532, 1470, 1454, 1423, 1366, 1335, 1237, 1172, 1088, 1060, 1035, 962, 918, 840, 811, 791, 741. HRMS (ESI) calcd for $C_{28}H_{30}ClN_2O_5S$ $[M + H]^+$ 541.1564; found 541.1551.

5i: R_f = 0.36, PE/Et_2O 1:1 developed with Hanessian stain; $[\alpha]_D$ = −42.6 (*c* = 1.0, $CHCl_3$); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.78 (d, *J* = 4.1 Hz, 1H; CH thiophene), 7.54 (broad s, 1H; NH), 7.32–7.20 (m, 5H; H arom), 7.16–7.06 (m, 3H; H arom), 7.05 (d, *J* = 4.1 Hz, 1H; CH thiophene), 5.58 (d, *J* = 2.9 Hz, 1H; CHO(CO)), 3.93 and 3.58 (AB syst., J_{AB} = 13.0 Hz, 2H; CH_2 of Bn), 3.90 (td, *J* = 8.1, 2.9 Hz, 1H; H-2), 3.76 and 3.60 (AB part of ABX syst., J_{AB} = 11.3, J_{AX} = 5.8, J_{BX} = 5.7 Hz, 2H; CH_2OAc), 3.32 (tt, *J* = 7.8, 5.7 Hz, 1H; H-4), 2.30–2.17 (m, 7H; H-3a, 2 CH_3), 2.15–2.04 (m, 1H; H-3b), 1.81 (s, 3H: OAc). ^{13}C-NMR (75 MHz, $CDCl_3$) δ 170.7 (C=O), 165.5 (C=O), 159.4 (C=O), 138.6 (Cq), 137.6 (Cq), 135.2 (2 Cq), 134.1 (CH), 132.6 (Cq), 130.6 (Cq), 129.2 (2 CH), 128.3 (2 CH), 128.3 (2 CH), 127.9 (CH), 127.7 (CH), 127.4 (CH), 73.8 (CHO(C=O)), 67.5 (CH_2OAc), 62.1 (CH-2), 60.4 (CH_2 of Bn), 59.7 (CH-4), 21.9 (CH_2-3), 20.5 (OAc), 18.5 (2 CH_3). HRMS (ESI) calcd for $C_{28}H_{30}ClN_2O_5S$ $[M + H]^+$ 541.1564; found 541.1557.



4j



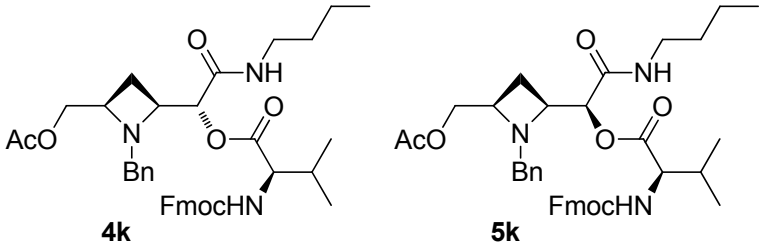
5j

(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-(butylamino)-2-oxoethyl (2s)-2-((((9h-fluoren-9-yl)methoxy)carbonyl)amino)-3-methylpropanoates **4j** *and* **5j**

4j: R_f = 0.39, PE/Et_2O 3:7 developed with Hanessian stain; $[\alpha]_D$ = −13.6 (*c* = 1.0, $CHCl_3$); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.77 (dd, *J* = 7.5, 0.6 Hz, 2H; H arom), 7.57 (dd, *J* = 6.8, 3.0 Hz, 2H; H arom), 7.41 (t, *J* = 7.4 Hz, 2H; H arom), 7.36–7.22 (m, 7H; H arom), 7.07 (broad t, *J* = 5.3 Hz, 1H; NH*n*Bu), 5.36 (broad d, *J* = 8.0 Hz, 1H; N*H*CH), 4.94 (d, *J* = 4.3 Hz, 1H; CHO(C=O)), 4.43 and 4.33 (AB part of ABX syst., J_{AB} = 10.5, J_{AX} = 7.1, J_{BX} = 7.1 Hz, 2H; CH_2 of Fmoc), 4.22 (m, 2H; C*H*NH, CH of Fmoc), 3.78 and 3.58

(AB syst., J_{AB} = 13.4 Hz, 2H; CH_2 of Bn), 3.92–3.66 (m, 3H; CH_2OAc, H-2), 3.38–3.02 (m, 3H; CH_2 of *n*Bu, H-4), 2.24–2.11 (m, 2H; CH of *i*Pr, H-3a), 1.89 (s, 3H; OAc), 1.87–1.74 (m, 1H; H-3b), 1.50–1.38 (m, 2H; CH_2 of *n*Bu), 1.36–1.20 (m, 2H; CH_2 of *n*Bu), 1.02 (t, *J* = 6.4 Hz, 6H; 2 CH_3 of *i*Pr), 0.83 (t, *J* = 7.2 Hz, 3H; CH_3 of *n*Bu). ^{13}C-NMR (75 MHz, $CDCl_3$) δ 171.7 (C=O), 170.7 (C=O), 167.0 (C=O), 156.6 (C=O), 143.6 (2 Cq), 141.3 (Cq), 141.2 (Cq), 137.5 (Cq), 129.2 (2 CH arom), 128.4 (2 CH arom), 127.8 (2 CH arom), 127.5 (CH arom), 127.1 (CH arom), 127.1 (CH arom), 125.0 (CH arom), 124.9 (CH arom), 120.0 (2 CH arom), 75.2 (CHO(C=O)), 67.3 (CH2 of Fmoc), 67.3 (CH_2OAc), 61.3 (CH_2 of Bn), 60.9 (CH-2), 60.0 (CH of Fmoc), 59.8 (CH-4), 47.1 (CHNH), 39.1 (CH_2 of *n*Bu), 31.3 (CH_2 of *n*Bu), 30.5 (CH of *i*Pr), 23.1 (CH_2-3), 20.7 (OAc), 20.1 (CH_2 of *n*Bu), 19.4 (CH_3 of *i*Pr), 18.2 (CH_3 of *i*Pr), 13.7 (CH_3 of *n*Bu). IR: υ (cm^{-1}) 3344, 2961, 2933, 2873, 1742, 1706, 1657, 1533, 1496, 1478, 1466, 1451, 1373, 1347, 1286, 1234, 1183, 1155, 1094, 1029, 909, 787, 758, 739. HRMS (ESI) calcd for $C_{39}H_{48}N_3O_7$ $[M + H]^+$ 670.3492; found 670.3502.

5j: R_f = 0.50, PE/Et_2O 3:7 developed with Hanessian stain; $[\alpha]_D$ = −41.9 (*c* = 0.76, $CHCl_3$); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5 Hz, 2H; H arom), 7.58 (d, *J* = 7.4 Hz, 2H; H arom), 7.40 (t, *J* = 7.2 Hz, 2H; H arom), 7.35–7.19 (m, 7H; H arom), 6.67 (broad t, *J* = 5.3 Hz, 1H; NH*n*Bu), 5.55 (broad d, *J* = 7.9 Hz, 1H; N*H*CH), 5.12 (d, *J* = 3.0 Hz, 1H; CHO(C=O)), 4.49 (dd, *J* = 10.3, 6.9 Hz, 1H; 1 H of CH_2 of Fmoc), 4.36–4.25 (m, 2H; 1 H of CH_2 of Fmoc, C*H*NH), 4.21 (t, *J* = 6.9 Hz, 1H; CH of Fmoc), 3.87–3.68 (m, 3H; CH_2OAc, H-2), 3.77 and 3.57 (AB syst., J_{AB} = 12.9 Hz, 2H; CH_2 of Bn), 3.28 (qd, *J* = 7.8, 6.2 Hz, 1H; H-4), 3.18 (dd, *J* = 12.9, 7.0 Hz, 2H; CH_2 of *n*Bu), 2.46–2.30 (m, 1H; CH of *i*Pr), 2.12 (dt, *J* = 10.9, 8.1 Hz, 1H; H-3a), 1.84 (s, 3H; OAc), 1.78 (dt, *J* = 10.9, 7.9 Hz, 1H; H-3b), 1.48–1.36 (m, 2H; CH_2 of *n*Bu), 1.34–1.22 (m, 2H; CH_2 of *n*Bu), 1.09 (d, *J* = 6.9 Hz, 3H; CH_3 of *i*Pr), 1.02 (d, *J* = 6.9 Hz, 3H; CH_3 of *i*Pr), 0.85 (t, *J* = 7.4 Hz, 3H; CH_3 of *n*Bu). IR: υ (cm^{-1}) 3329, 2960, 2933, 2873, 1739, 1718, 1657, 1532, 1478, 1466, 1451, 1366, 1317, 1271, 1231, 1186, 1169, 1092, 1031, 915, 758, 739. HRMS (ESI) calcd for $C_{39}H_{48}N_3O_7$ $[M + H]^+$ 670.3492; found 670.3498.

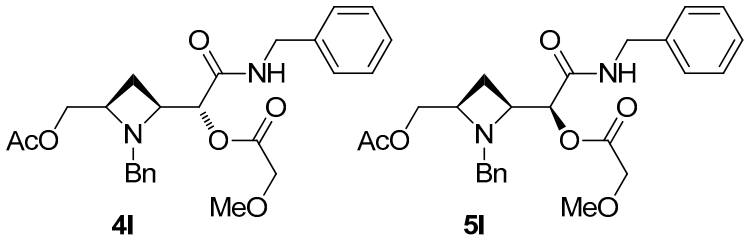


(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-(butylamino)-2-oxoethyl (2r)-2-((((9h-fluoren-9-yl)methoxy)carbonyl)amino)-3-methylpropanoates **4k** *and* **5k**

4k: R_f = 0.31, PE/Et_2O 3:7 developed with Hanessian stain; $[\alpha]_D$ = +1.87 (*c* = 1.0, $CHCl_3$); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.78 (dd, *J* = 7.4, 4.1 Hz, 2H; H arom), 7.60 (dd, *J* = 7.4, 3.5 Hz, 2H; H arom), 7.49–7.27 (m, 5H; H arom), 7.24–7.13 (m, 5H; 4 H arom and NHDragendorff), 5.54 (d, *J* = 8.3 Hz, 1H; N*H*CH), 4.88 (d, *J* = 4.1 Hz, 1H; CHO(C=O)), 4.57–4.49 (m, 1H; 1 H of CH_2 of Fmoc), 4.34–4.19 (m, 3H; 1 H of CH_2 of Fmoc, C*H*NH and CH of Fmoc), 4.02 (dd, *J* = 11.2, 6.1 Hz, 1H; 1 H of CH_2OAc), 3.69 and 3.58 (AB syst., J_{AB} = 13.0 Hz, 2H; CH_2 of Bn), 3.70–3.59 (m, 2H; H-2 and 1 H of CH_2OAc), 3.37–3.18 (m, 3H; H-4 and C*H*$_2$NH), 2.42–2.32 (m, 1H; CH of *i*Pr), 2.25 (dt, *J* = 11.3, 8.2 Hz, 1H; H-3a), 1.92 (s, 3H; OAc), 1.75 (dt, *J* = 11.4, 8.0 Hz, 1H; H-3b), 1.57–1.45 (m, 2H; CH_2 of *n*Bu), 1.41–1.25 (m, 2H; CH_2 of *n*Bu), 1.06 (d, *J* = 6.7 Hz, 3H; CH_3 of *i*Pr), 0.98 (d, *J* = 6.9 Hz, 3H; CH_3 of *i*Pr), 0.90 (t, *J* = 7.4 Hz, 3H; CH_3 of *n*Bu). ^{13}C-NMR (75 MHz, $CDCl_3$) δ 170.85 (C=O), 170.82 (C=O), 167.0 (C=O), 157.1 (C=O), 143.7 (Cq), 143.7 (Cq), 141.3 (Cq), 141.2 (Cq), 137.6 (Cq), 128.9 (2 CH arom), 128.4 (CH arom), 127.80 (CH arom), 127.76 (CH arom), 127.72 (CH arom), 127.5 (CH arom), 127.1 (CH arom), 127.0 (CH arom), 125.1 (CH arom), 125.0 (CH arom), 120.00 (CH arom), 119.96 (CH arom), 74.3 (CHO(C=O)), 67.2 (CH_2 of Fmoc), 66.9 (CH_2OAc), 61.7 (CH_2 of Bn), 61.4 (CH-2), 59.8 (CH-4), 59.4 (CH of Fmoc), 47.1 (CHNH), 39.1 (CH_2 of *n*Bu), 31.4 (CH_2 of *n*Bu), 30.4 (CH of *i*Pr), 22.6 (CH_2-3), 20.7 (OAc), 20.1 (CH_2 of *n*Bu), 19.2 (CH_3 of *i*Pr), 17.3 (CH_3 of *i*Pr), 13.7 (CH_3 of *n*Bu). IR: υ (cm^{-1}) 3329, 2960, 2932, 2873, 1739, 1704, 1656, 1533, 1478,

1466, 1451, 1373, 1318, 1233, 1191, 1134, 1096, 1030, 909, 786, 759, 738. HRMS (ESI) calcd for $C_{39}H_{48}N_3O_7$ $[M + H]^+$ 670.3492; found 670.3487.

5k: R_f = 0.40, PE /Et_2O 3:7 developed with Hanessian stain; $[\alpha]_D$ = −21.8 (*c* = 0.4, $CHCl_3$); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.77 (d, *J* = 7.5 Hz, 2H; H arom), 7.56 (dd, *J* = 7.4, 4.3 Hz, 2H; H arom), 7.41 (t, *J* = 7.5 Hz, 2H; H arom), 7.37–7.17 (m, 7H; H arom), 6.74 (broad t, *J* = 5.5 Hz, 1H; NH*n*Bu), 5.28 (d, *J* = 7.5 Hz, 1H; N*H*CH), 5.12 (d, *J* = 2.5 Hz, 1H; CHO(C=O)), 4.42 and 4.34 (AB part of ABX syst., J_{AB} = 10.5, J_{Ax} = 7.1, J_{BX} = 7.2 Hz, 2H; CH_2 of Fmoc), 4.21 (t, *J* = 7.0 Hz, 1H; CH of Fmoc), 4.10 (t, *J* = 7.4 Hz, 1H; NHC*H*), 3.86–3.67 (m, 3H; H-2, CH_2OAc), 3.78 and 3.58 (AB syst., J_{AB} = 12.8 Hz, 2H; CH_2 of Bn), 3.33–3.15 (m, 2H; H-4, 1 H of C*H*$_2$NH), 3.15–3.00 (m, 1H; 1 H of C*H*$_2$NH), 2.22–2.03 (m, 2H; CH of *i*Pr, H-3a), 1.97–1.81 (m, 1H; H-3b), 1.89 (s, 3H; OAc), 1.49–1.36 (m, 2H; CH_2 of *n*Bu), 1.33–1.21 (m, 2H; CH_2 of *n*Bu), 1.13 (d, *J* = 6.8 Hz, 3H; CH_3 of *i*Bu), 1.09 (d, *J* = 6.8 Hz, 3H; CH_3 of *i*Bu), 0.82 (t, *J* = 7.2 Hz, 3H; CH_3 of *n*Bu). ^{13}C-NMR (75 MHz, $CDCl_3$) δ 171.7 (C=O), 170.8 (C=O), 167.0 (C=O), 156.7 (C=O), 143.6 (Cq), 143.5 (Cq), 141.3 (Cq), 141.2 (Cq), 137.8 (Cq), 129.3 (2 CH arom), 128.2 (2 CH arom), 127.8 (2 CH arom), 127.3 (CH arom), 127.2 (CH arom), 127.1 (CH arom), 124.9 (CH arom), 124.9 (CH arom), 120.1 (CH arom), 120.0 (CH arom), 73.7 (CHO(C=O)), 67.7 (CH_2OAc), 67.3 (CH_2 of Fmoc), 62.2 (CH-2), 61.2 (CH_2 of Bn), 60.4 (CHNH), 59.8 (CH-4), 47.1 (CH of Fmoc), 39.1 (CH_2 of *n*Bu), 31.3 (CH_2 of *n*Bu), 30.3 (CH of *i*Bu), 21.7 (CH_2-3), 20.7 (OAc), 20.0 (CH_2 of *n*Bu), 19.5 (CH_3 of *i*Bu), 18.6 (CH_3 of *i*Bu), 13.7 (CH_3 of *n*Bu). IR: υ (cm^{-1}) 3354, 2961, 2933, 2874, 1743, 1705, 1656, 1535, 1496, 1478, 1466, 1451, 1373, 1286, 1236, 1183, 1155, 1085, 1030, 918, 790, 757, 739. HRMS (ESI) calcd for $C_{39}H_{48}N_3O_7$ $[M + H]^+$ 670.3492; found 670.3490.

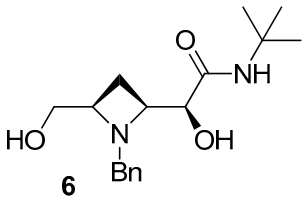


(1R) and (1S) 1-((2S,4R)-4-(Acetoxymethyl)-1-benzylazetidin-2-yl)-2-(benzylamino)-2-oxoethyl 2-methoxyacetate **4l** *and* **5l**

4l: R_f = 0.26, PE/Et_2O 1:5 developed with Hanessian stain; $[\alpha]_D$ = −23.1 (*c* = 1.7, $CHCl_3$); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.39–7.11 (m, 11H; 10H arom and NH), 4.92 (d, *J* = 4.7 Hz, 1H; CHO(C=O)), 4.52–4.34 (m, 2H; CH_2NH), 4.07 and 3.97 (AB syst., J_{AB} = 16.6 Hz, 2H; CH_2 of Bn), 3.80 and 3.74 (AB part of ABX syst., J_{AB} = 11.1, J_{AX} = 5.0, J_{BX} = 6.1 Hz, 2H; CH_2OAc), 3.65 (s, 2H; C*H*$_2$OMe), 3.64–3.54 (m, 1H; H-2), 3.42 (s, 3H; OMe), 3.40–3.28 (m, 1H; H-4), 2.23 (dt, *J* = 11.2, 7.9 Hz, 1H; H-3a), 1.92 (s, 3H; OAc), 1.76 (dt, *J* = 11.2, 8.4 Hz, 1H; H-3b). ^{13}C-NMR (75 MHz, $CDCl_3$) δ 170.6 (C=O), 169.3 (C=O), 166.8 (C=O), 137.7 (Cq), 137.3 (Cq), 128.9 (2 CH arom), 128.7 (2 CH arom), 128.6 (2 CH arom), 128.0 (2 CH arom), 127.6 (2 CH arom), 73.5 (CHO(C=O)), 69.4 (CH_2 of Bn), 66.9 (CH_2OAc), 61.6 (CH_2OMe), 61.6 (CH-2), 60.3 (CH-4), 59.4 (OMe), 43.4 (CH_2NH), 23.0 (CH_2-3), 20.7 (OAc). IR: υ (cm^{-1}) 3315, 3064, 3029, 3004, 2935, 2883, 2829, 1739, 1666, 1605, 1533, 1496, 1454, 1366, 1333, 1233, 1183, 1125, 1029, 976, 932, 910, 820, 750, 732. HRMS (ESI) calcd for $C_{25}H_{31}N_2O_6$ $[M + H]^+$ 455.2182; found 455.2171.

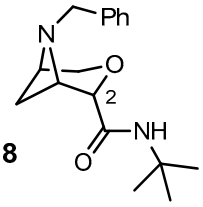
5l: R_f = 0.34, PE/Et_2O 1:5 developed with Hanessian stain; $[\alpha]_D$ = −28.8 (*c* = 0.6, $CHCl_3$); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.39–7.16 (m, 10H; H arom), 6.51 (broad t, *J* = 5.7 Hz, 1H; NH), 5.37 (d, *J* = 3.4 Hz, 1H; CHO(C=O)), 4.51 and 4.40 (AB part of ABX syst., J_{AB} = 14.8, J_{AX} = 6.0, J_{BX} = 5.7 Hz, 2H; C*H*$_2$NH), 4.10 (s, 2H; C*H*$_2$OMe), 3.79 and 3.52 (AB syst., J_{AB} = 12.9 Hz, 2H; CH_2 of Bn), 3.75–3.63 (m, 3H; CH_2OAc and H-2), 3.46 (s, 3H; OMe), 3.30 (tt, *J* = 7.9, 5.6 Hz, 1H; H-4), 2.14 (dt, *J* = 10.8, 7.9 Hz, 1H; H-3a), 1.87 (s, 3H; OAc), 1.87 (dt, *J* = 10.9, 8.3 Hz, 1H; H-3b). ^{13}C-NMR (75 MHz, $CDCl_3$) δ 170.7 (C=O), 168.8 (C=O), 167.1 (C=O), 137.8 (Cq), 137.6 (Cq), 129.2 (2 CH arom), 128.8 (2 CH arom), 128.3 (2 CH arom), 127.8 (2 CH arom), 127.7 (CH arom), 127.4 (CH arom), 73.6 (CHO(C=O)), 69.8 (*C*H$_2$OMe), 67.4 (CH_2OAc),

62.1 (CH-2), 60.8 (CH_2 of Bn), 59.7 (CH-4), 59.5 (CH_3O), 43.2 (CH_2NH), 21.60 (CH_2-3), 20.7 (OAc). IR: υ (cm^{-1}) 3339, 3063, 3029, 2936, 2883, 2828, 1737, 1658, 1605, 1530, 1496, 1454, 1366, 1333, 1235, 1179, 1124, 1086, 1029,976, 927,910, 849, 733. HRMS (ESI) calcd for $C_{25}H_{31}N_2O_6$ $[M + H]^+$ 455.2182; found 455.2174.



6

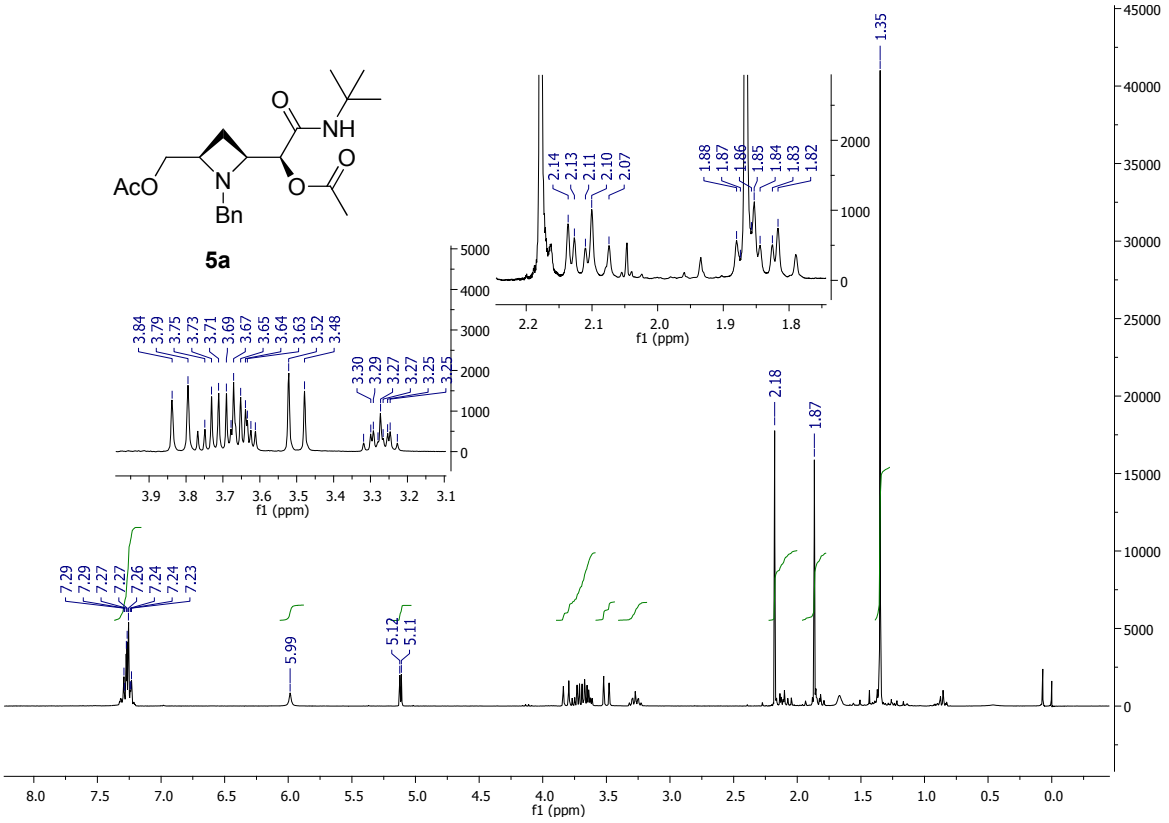
(1S)-2-((2S,4R)-1-Benzyl-4-(hydroxymethyl)azetidin-2-yl)-N-(tert-butyl)-2-hydroxyacetamide **6**: R_f = 0.36, *n*Hex/Et_2O 1:6 developed with Dragendorff stain; m.p. = 123.0–128.8 °C (AcOEt); $[\alpha]_D$ = −74.8 (*c* = 1.1, $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.32–7.26 (m, 5H; CH arom), 6.58 (s, 1H; NH), 4.01 (s, 1H; OH), 3.76–3.69 (m, 3H; CH-2 and CH_2 of Bn), 3.58 (d, *J* = 3.7 Hz, 1H; CHCON), 3.42–3.35 (m, 2H; CH_2OH), 3.32 (tt, *J* = 7.8, 3.8 Hz, 1H; CH-4), 1.98–1.80 (m, 3H, CH_2-3 and OH), 1.31 (s, 9H; *t*Bu); ^{13}C-NMR (100 MHz, $CDCl_3$): δ 169.6 (C=O), 137.4 (Cq), 129.0 (CH arom), 128.7 (CH arom), 127.8 (CH arom), 70.1 (CHOH), 64.2 (CH_2OH), 63.1 (CH-2), 62.7 (CH-4), 60.3 (CH_2 of Bn), 50.8 (Cq), 28.7 (*t*Bu), 17.9 (CH_2-3). IR: υ (cm-^{1}) 3483, 3457, 3383, 2990, 2963, 2940, 2901, 2874, 2832, 1654, 1536, 1496, 1478, 1453, 1393, 1382, 1371, 1339, 1328, 1302, 1274, 1252, 1226, 1195, 1172, 1153, 1134, 1114, 1094, 1044, 1028, 995, 958, 931, 906, 893, 850, 793, 756, 732. HRMS (ESI) calcd for $C_{17}H_{27}N_2O_3$ $[M + H]^+$ 307.2022; found 307.2031.

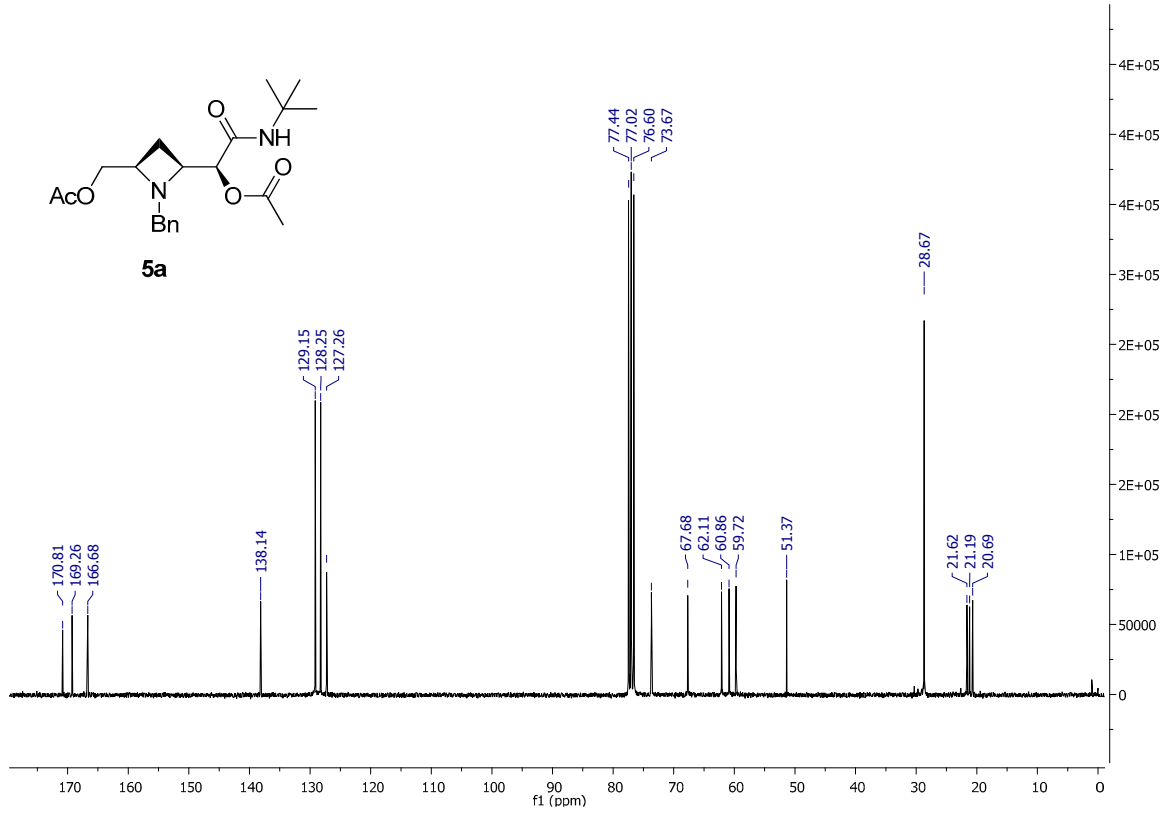


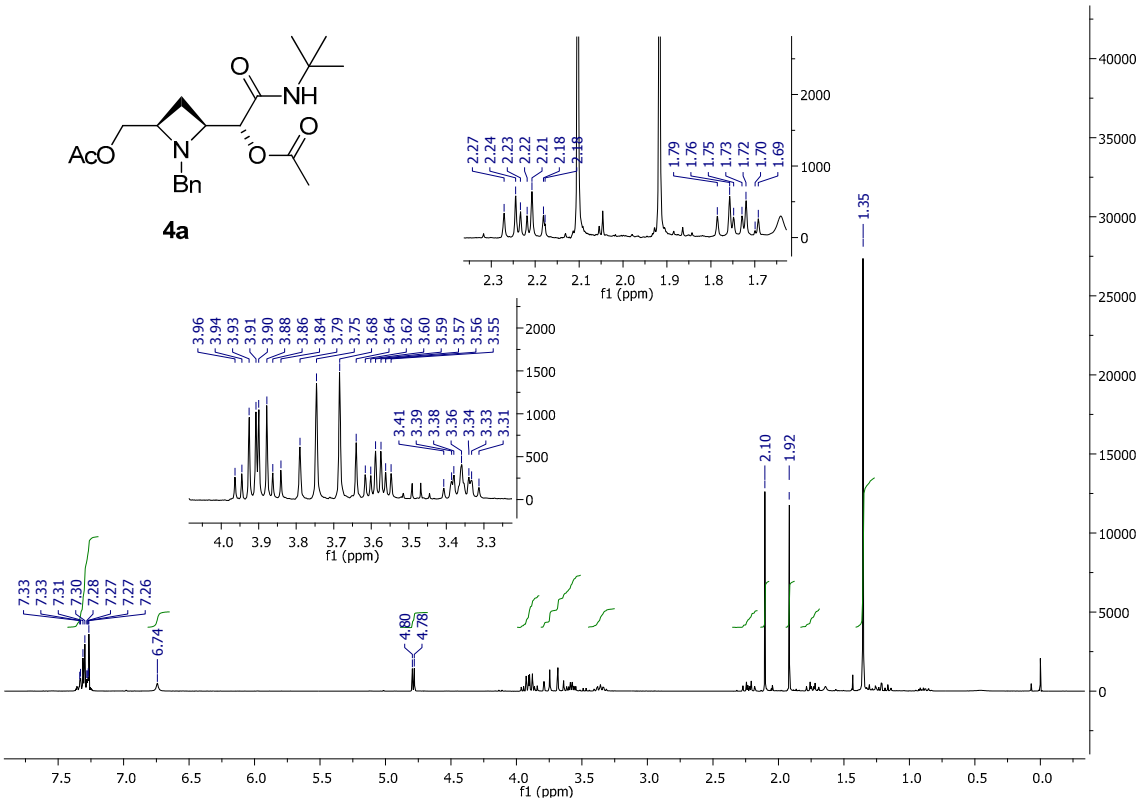
8

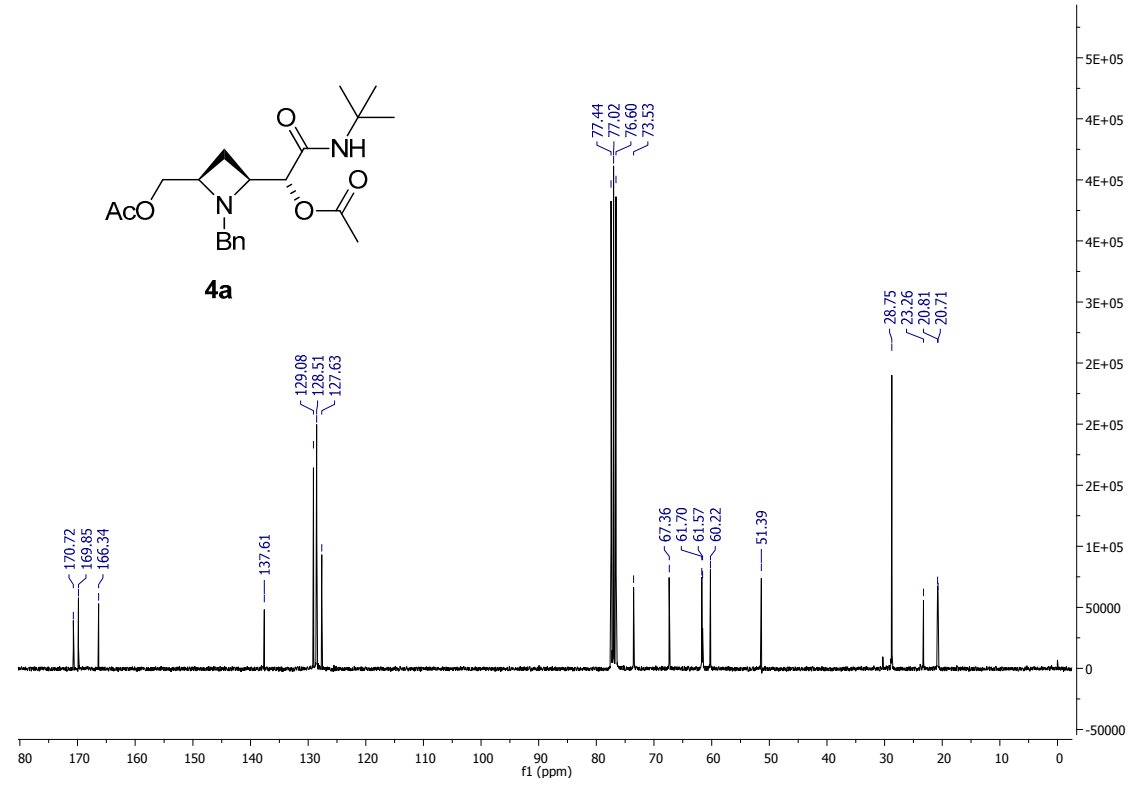
(1S,2S,5R)-6-Benzyl-N-(tert-butyl)-3-oxa-6-azabicyclo[3.1.1]heptane-2-carboxamide **8**: R_f = 0.28 (ETP/Et_2O 1:3); ^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.46–7.14 (m, 5H; H arom), 6.71 (broad s, 1H; NH), 4.54 (d, *J* = 1.5 Hz, 1H; H-2), 4.30 and 3.82 (AB part of ABX syst., J_{AB} = 10.6, J_{AX} = 1.1, J_{BX} = 1.8 Hz, 2H; CH_2-6), 3.89 (ddd, *J* = 5.6, 3.5, 1.7 Hz, 1H; H-3), 3.84 (s, 2H; CH_2 of Bn), 3.55–3.44 (m, 1H; H-5), 2.60 (dt, *J* = 8.5, 6.2 Hz, 1H; H-4a), 1.62 (d, *J* = 8.8 Hz, 1H; H-4b), 1.37 (s, 9H; *t*Bu). ^{13}C-NMR (100 MHz, $CDCl_3$): δ 170.2 (C=O), 137.7 (Cq), 128.5 (2 CH arom), 128.3 (2 CH arom), 127.0 (CH arom), 71.1 (CH-2), 63.2 (CH_2-6), 62.5 (H-3), 59.7 (H-4), 50.8 (Cq), 49.9 (CH_2 of Bn), 28.8 (*t*Bu), 26.1 (CH_2-4).

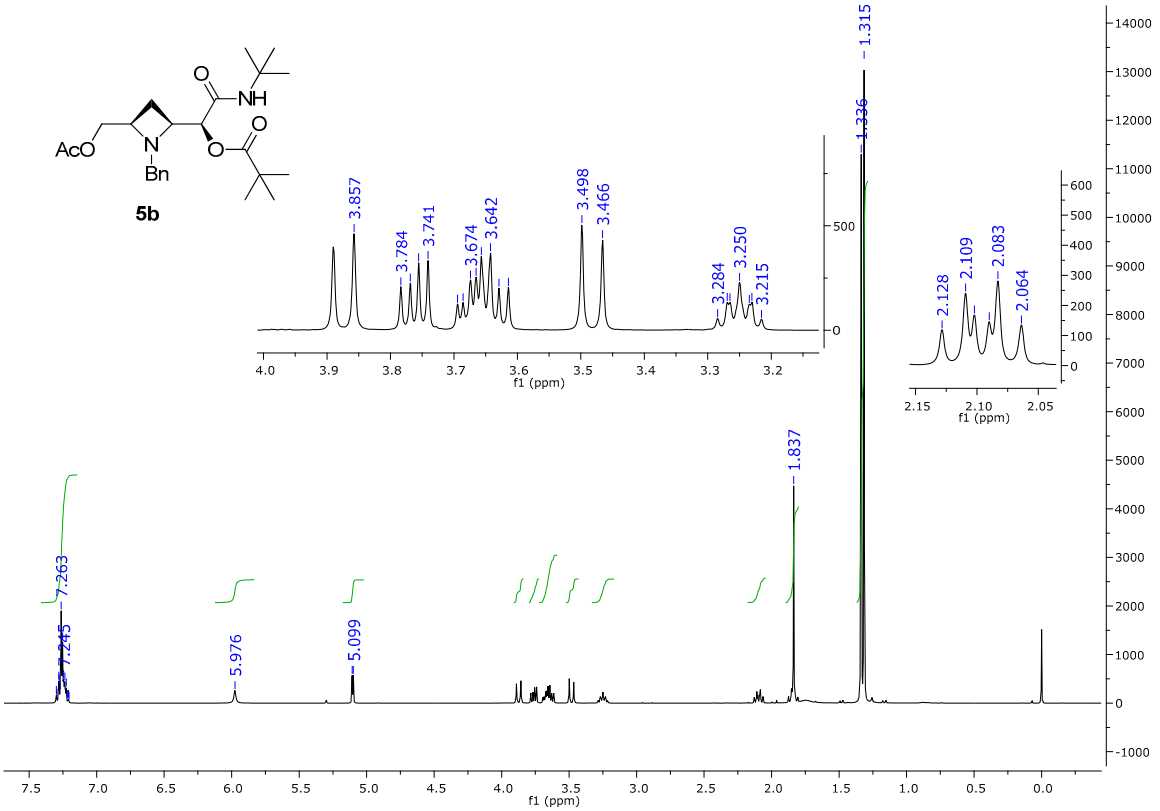
3. Copies of ^{1}H- and ^{13}C-NMR Spectra of Compounds






AcO
N
Bn
O
NH
O
O
4a
f1 (ppm)


AcO
N
Bn
O
NH
O
O
4a
f1 (ppm)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N
Bn
O
NH
O
O
5b
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5.976
5.099
1.837
1.336
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3.857
3.784
3.741
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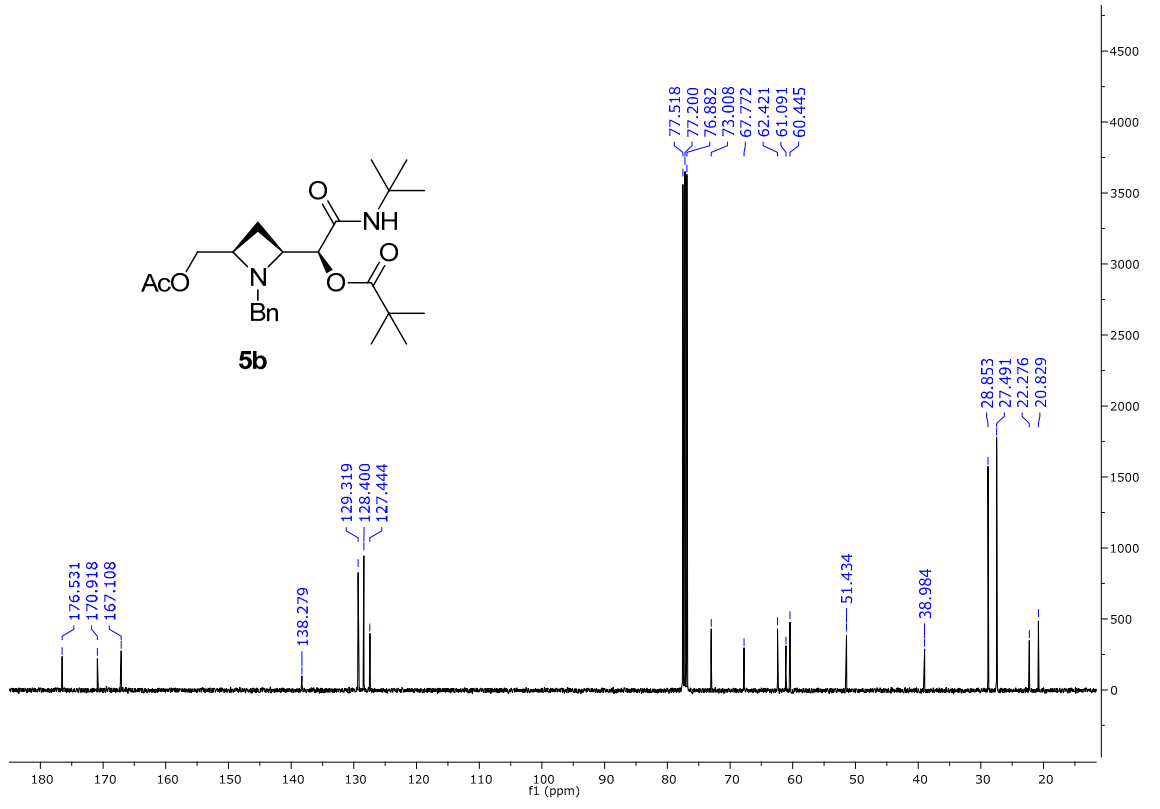
2.083
2.064
f1 (ppm)


AcO
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Bn
O
NH
O
O
5b
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73.008
67.772
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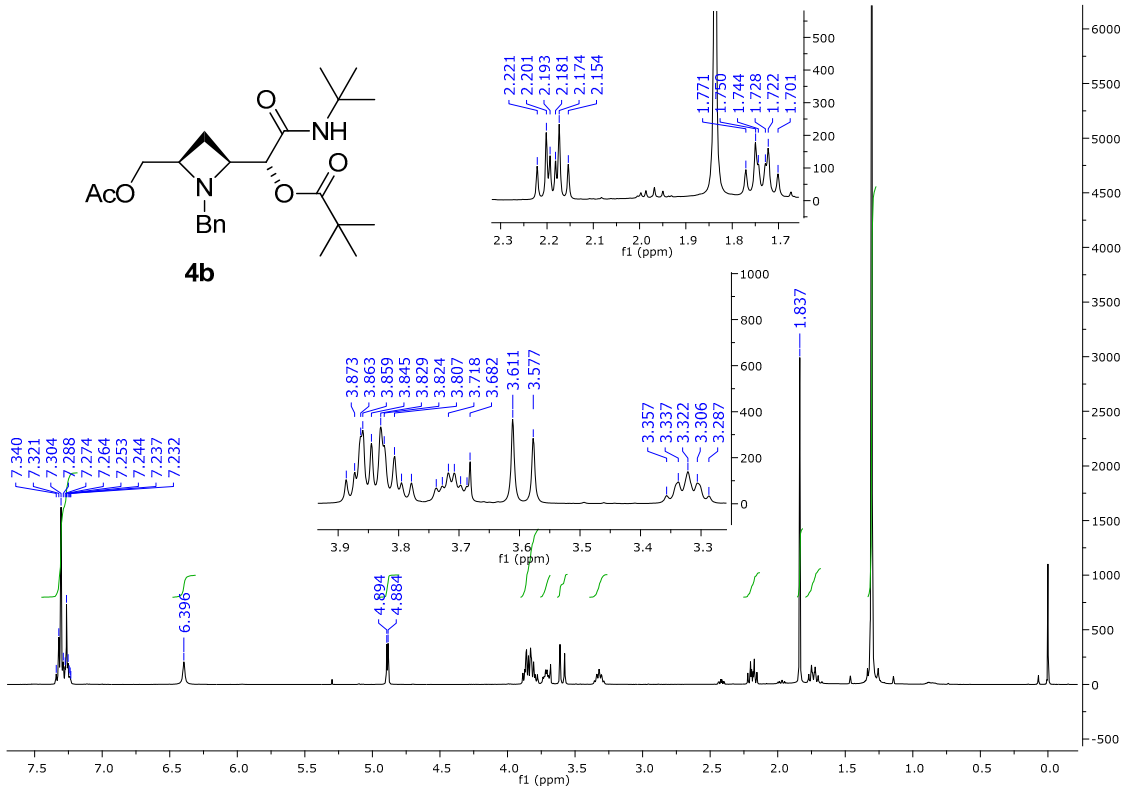
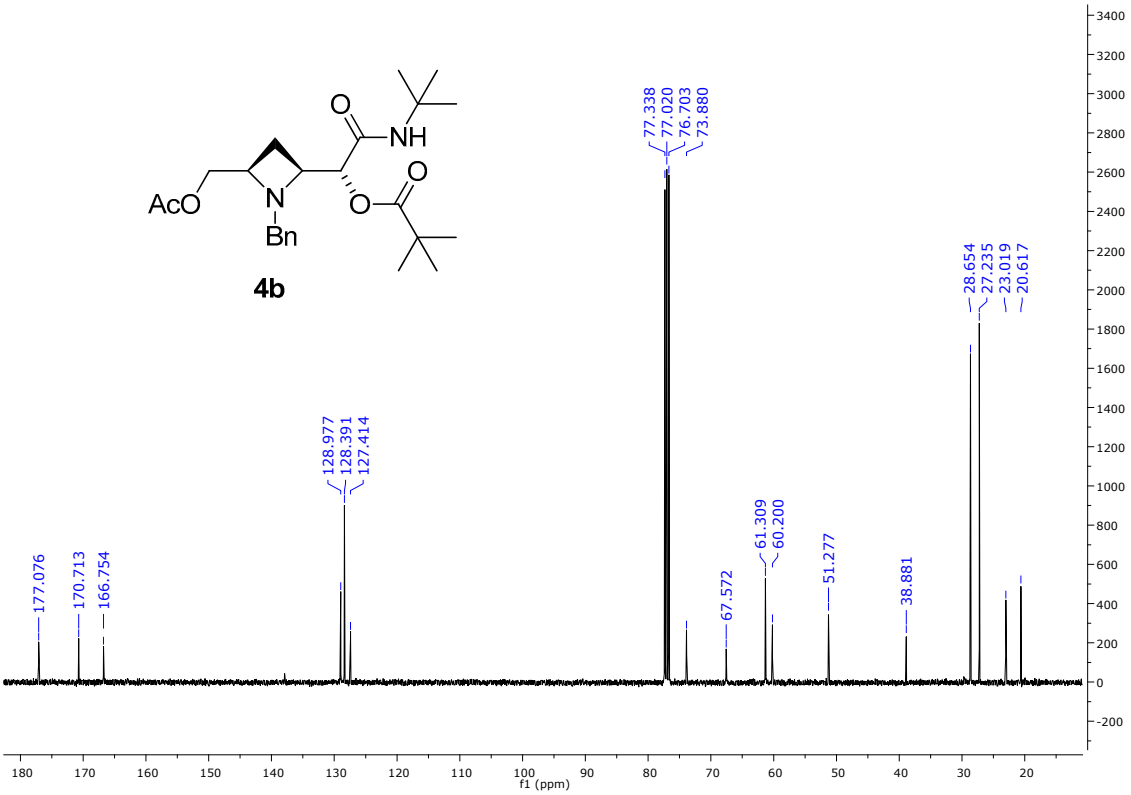
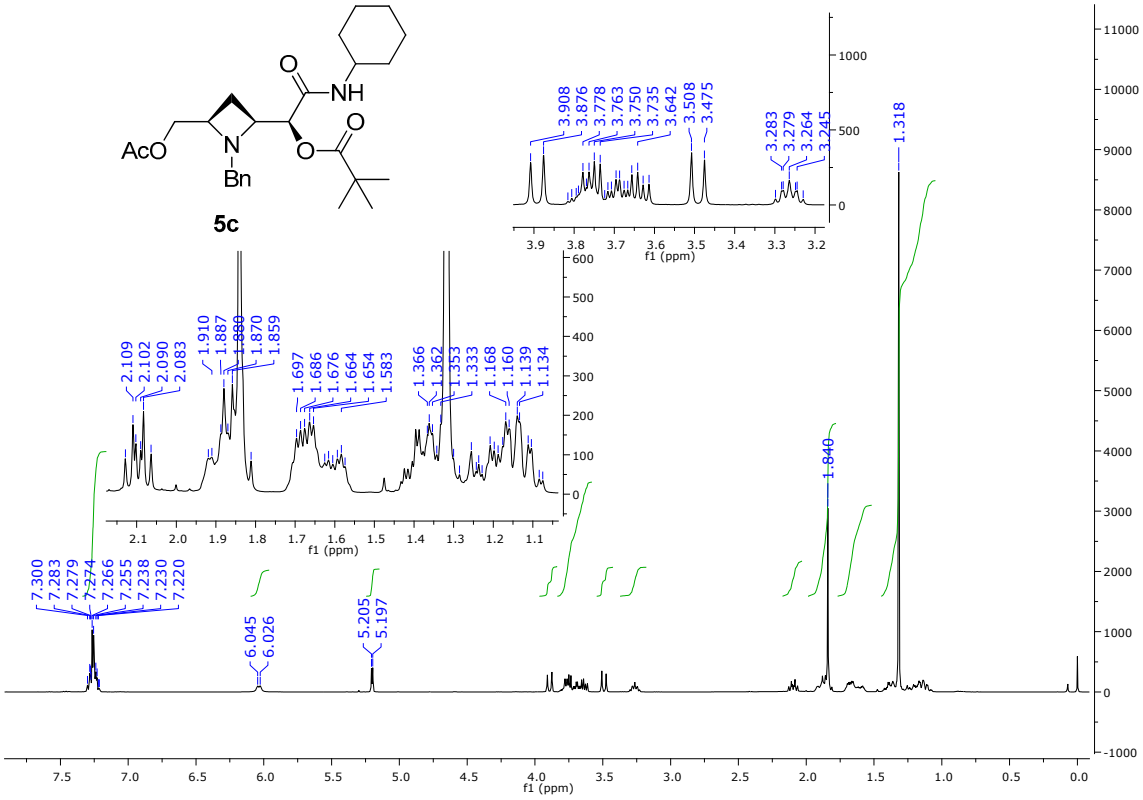
60.445
176.531
170.918
167.108
138.279
129.319
128.400
127.444
51.434
38.984
28.853
27.491
22.276
20.829
f1 (ppm)


4b
7.340
7.321
7.304
7.288
7.274
7.264
7.253
7.244
7.237
7.232
6.396
4.894
4.884
3.873
3.863
3.859
3.845
3.829
3.824
3.807
3.718
3.682
3.611
3.577
3.357
3.337
3.322
3.306
3.287
1.837
2.221
2.201
2.193
2.181
2.174
2.154
1.771
1.750
1.744
1.728
1.722
1.701
f1 (ppm)


4b
177.076
170.713
166.754
128.977
128.391
127.414
77.338
77.020
76.703
73.880
67.572
61.309
60.200
51.277
38.881
28.654
27.235
23.019
20.617
f1 (ppm)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N
Bn
O
NH
O
O
5c
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7.283
7.279
7.274
7.266
7.255
7.238
7.230
7.220
6.045
6.026
5.205
5.197
3.908
3.876
3.778
3.763
3.750
3.735
3.642
3.508
3.475
3.283
3.279
3.264
3.245
2.109
2.102
2.090
2.083
1.910
1.887
1.870
1.859
1.697
1.686
1.676
1.664
1.654
1.583
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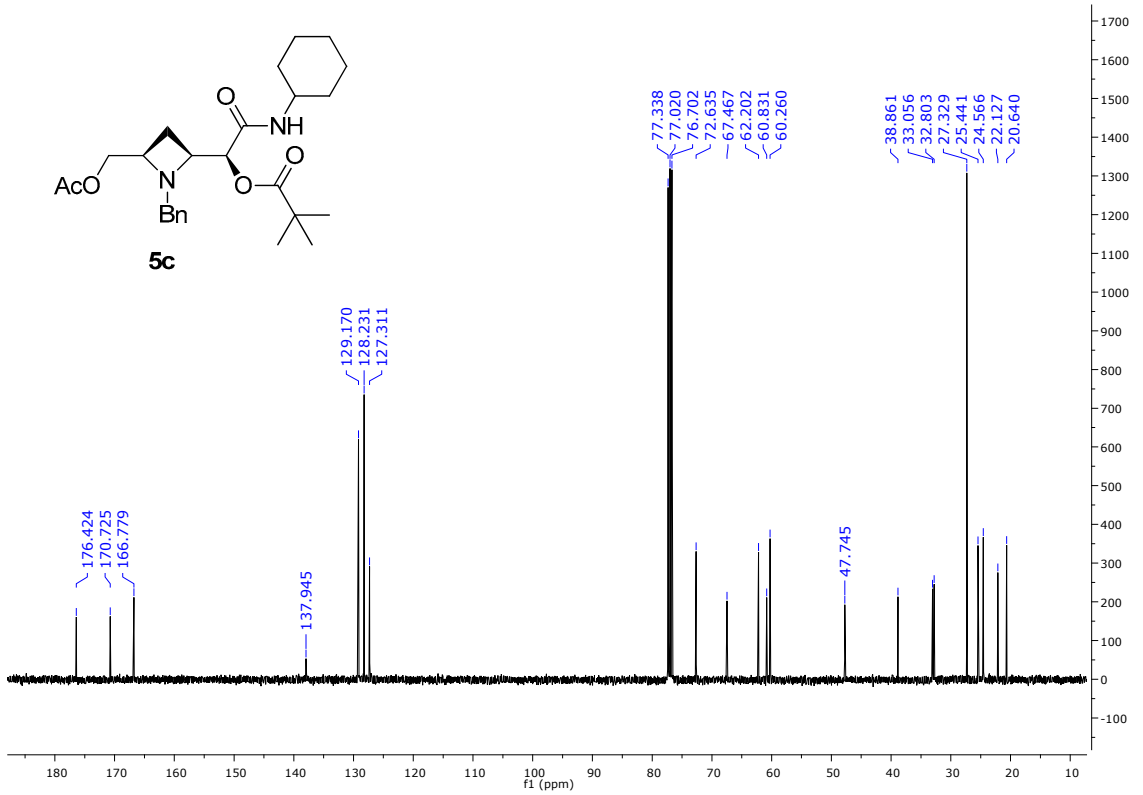
1.362
1.353
1.333
1.168
1.160
1.139
1.134
1.840
1.318
f1 (ppm)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Bn
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NH
O
O
5c
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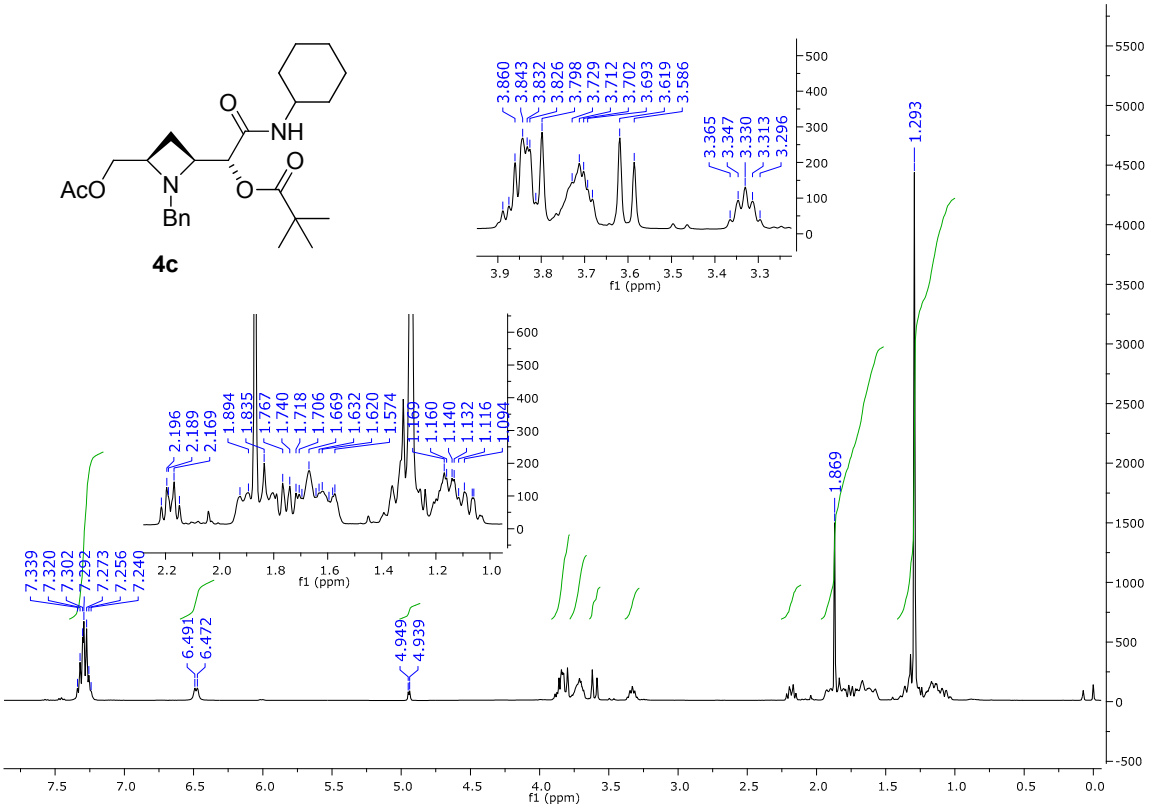
170.725
166.779
137.945
129.170
128.231
127.311
77.338
77.020
76.702
72.635
67.467
62.202
60.831
60.260
47.745
38.861
33.056
32.803
27.329
25.441
24.566
22.127
20.640
f1 (ppm)


AcO
N
Bn
O
NH
O
O
4c
3.860
3.843
3.832
3.826
3.798
3.729
3.712
3.702
3.693
3.619
3.586
3.365
3.347
3.330
3.313
3.296
1.293
2.196
2.189
2.169
1.894
1.835
1.767
1.740
1.718
1.706
1.669
1.632
1.620
1.574
1.160
1.140
1.132
1.116
1.094
1.869
7.339
7.320
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7.292
7.273
7.256
7.240
6.491
6.472
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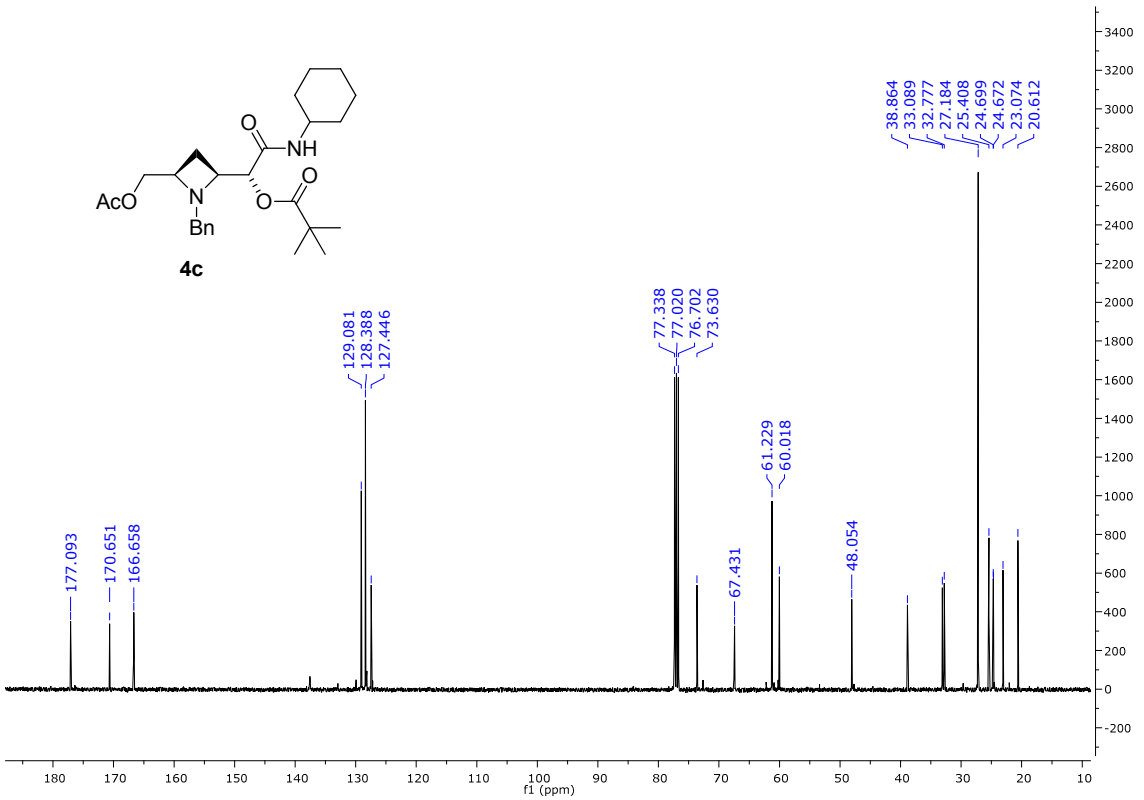
4.939
f1 (ppm)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N
Bn
O
NH
O
O
4c
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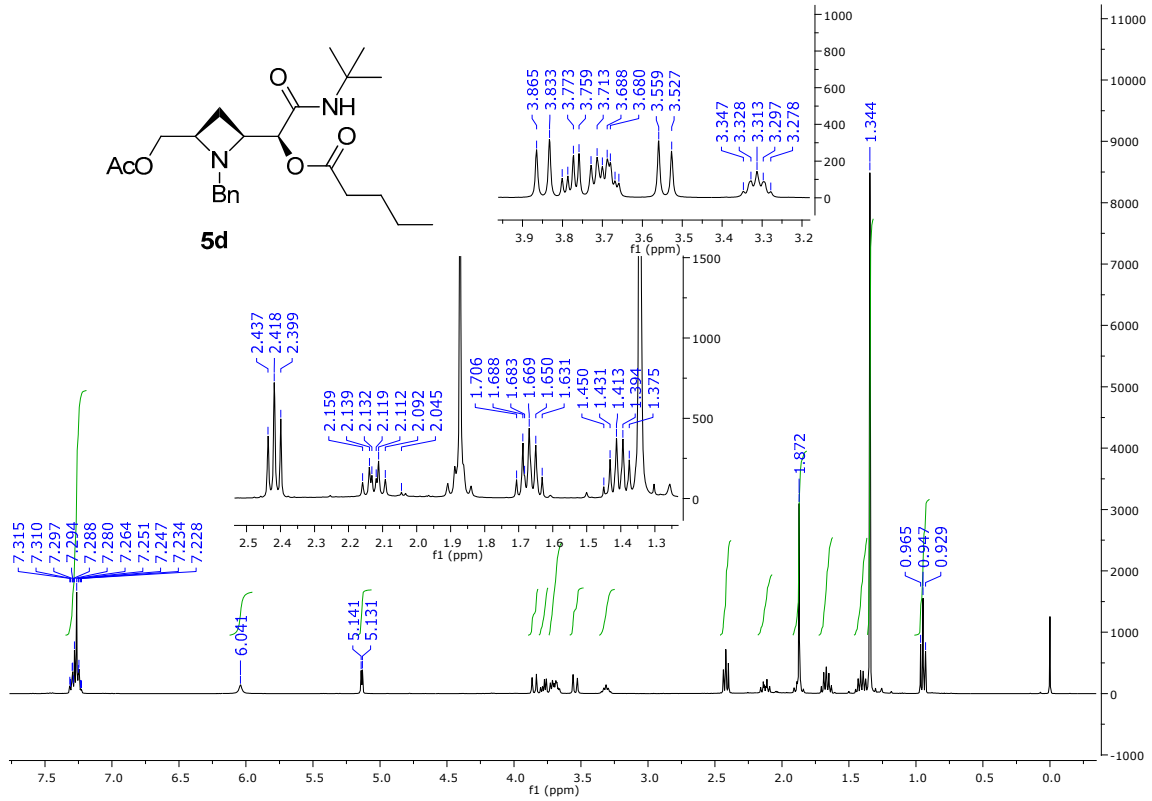
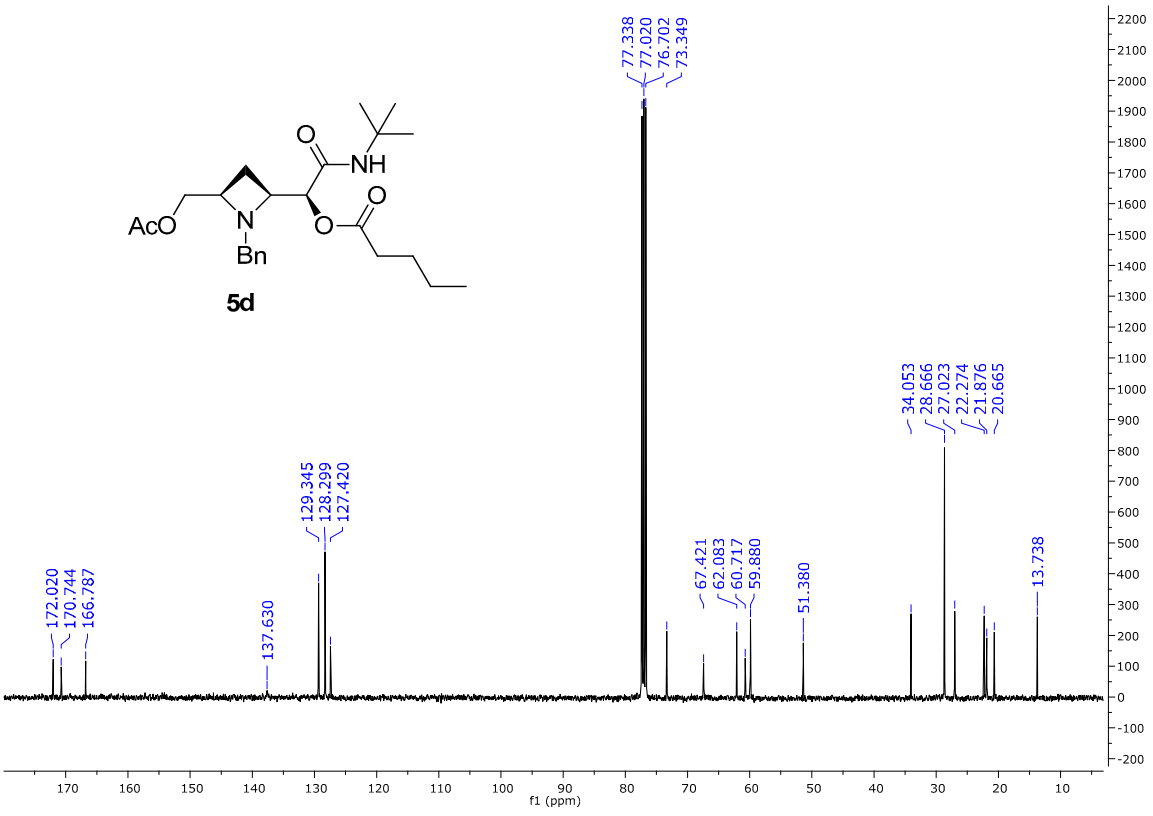
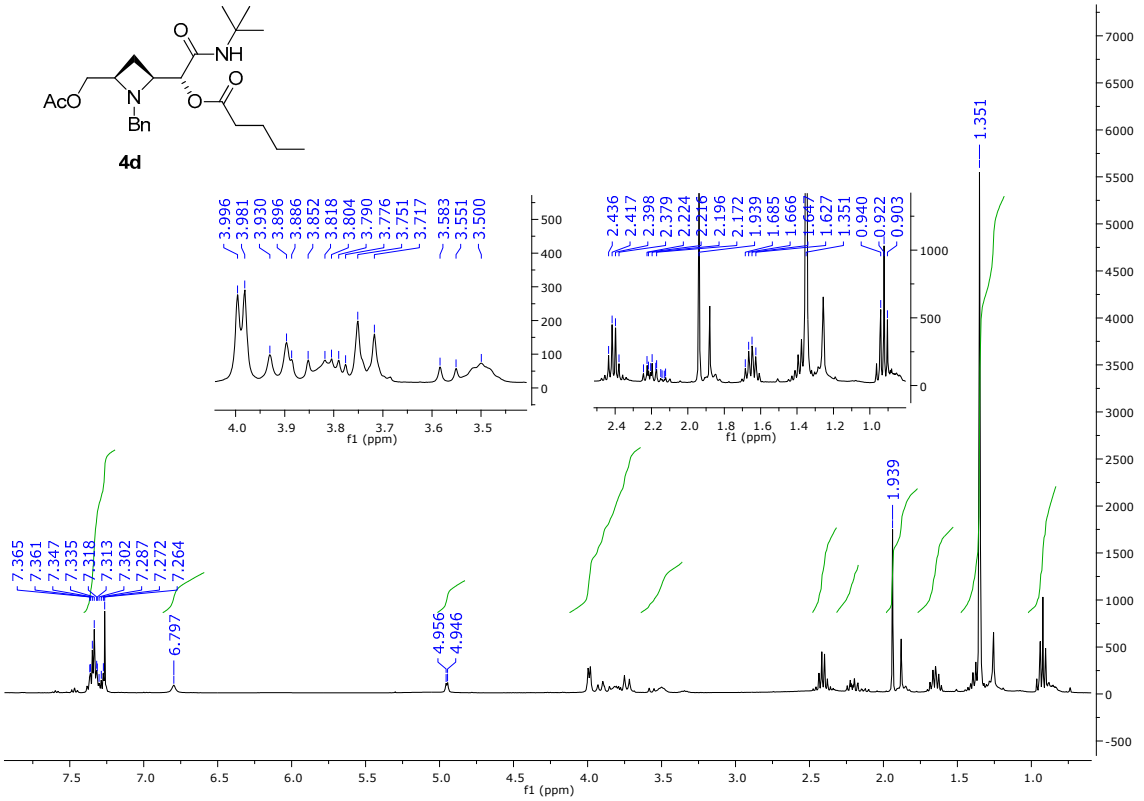
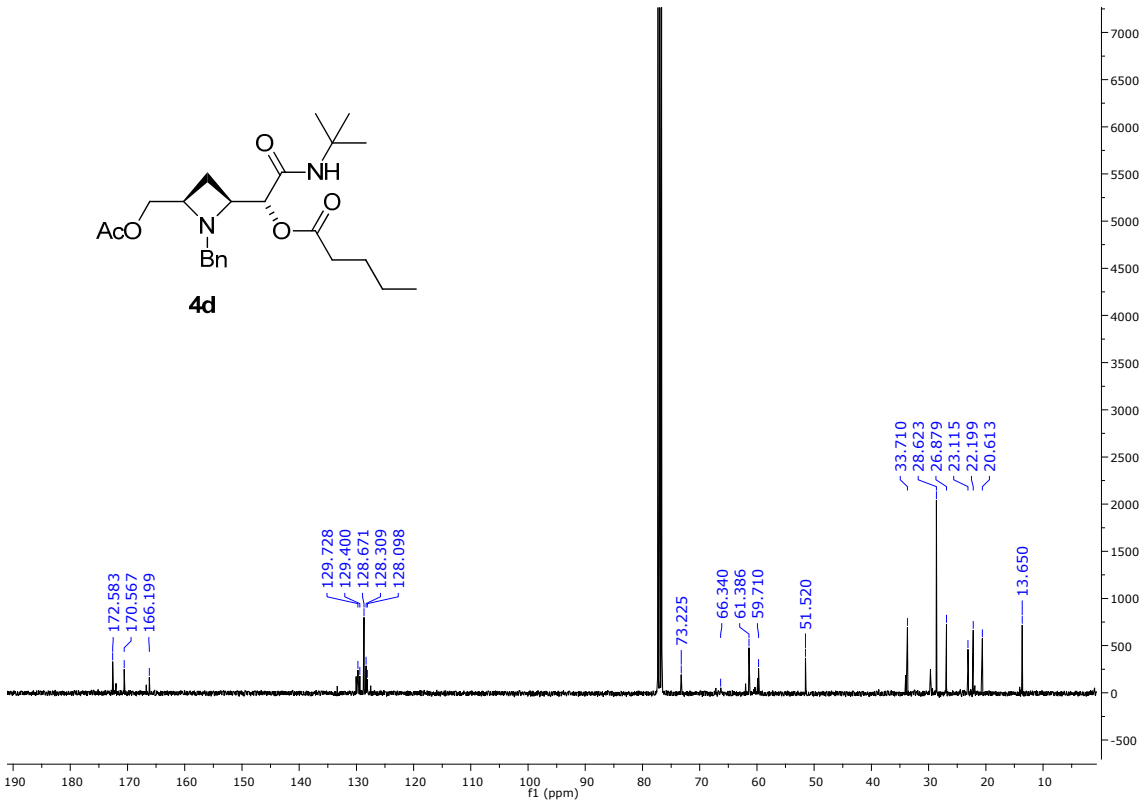
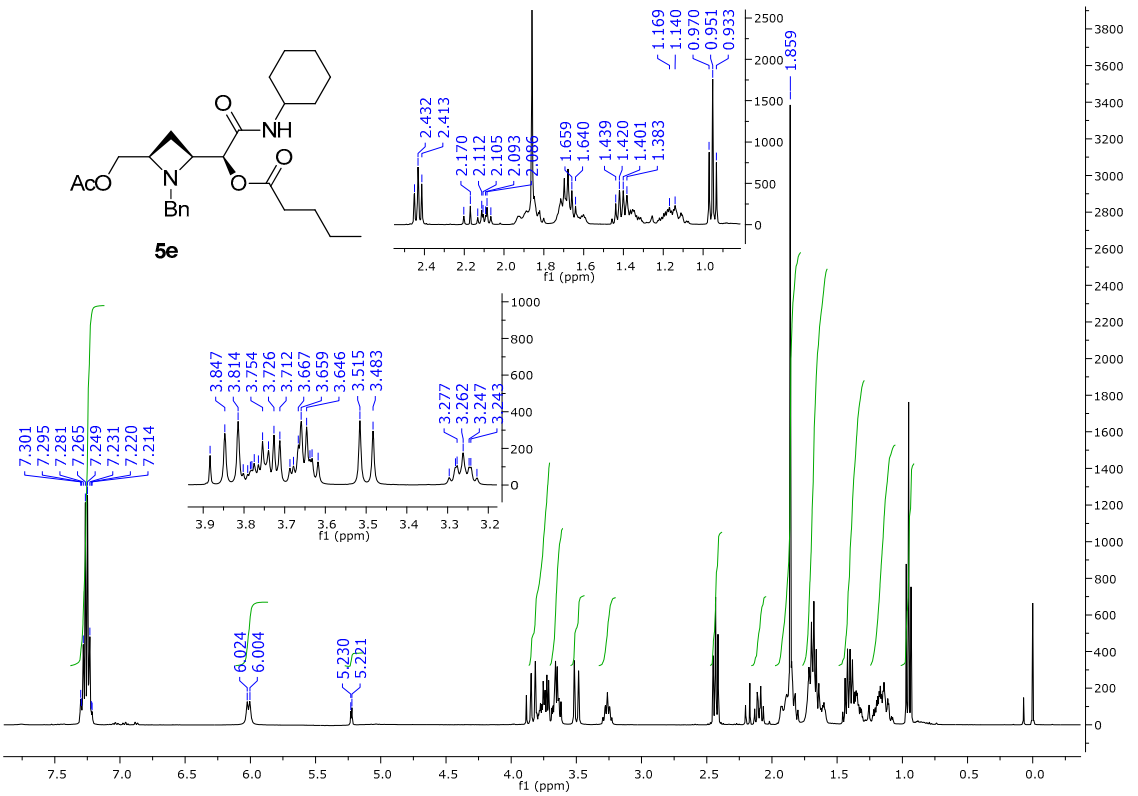
33.089
32.777
27.184
25.408
24.699
24.672
23.074
20.612
129.081
128.388
127.446
77.338
77.020
76.702
73.630
61.229
60.018
67.431
48.054
177.093
170.651
166.658
f1 (ppm)

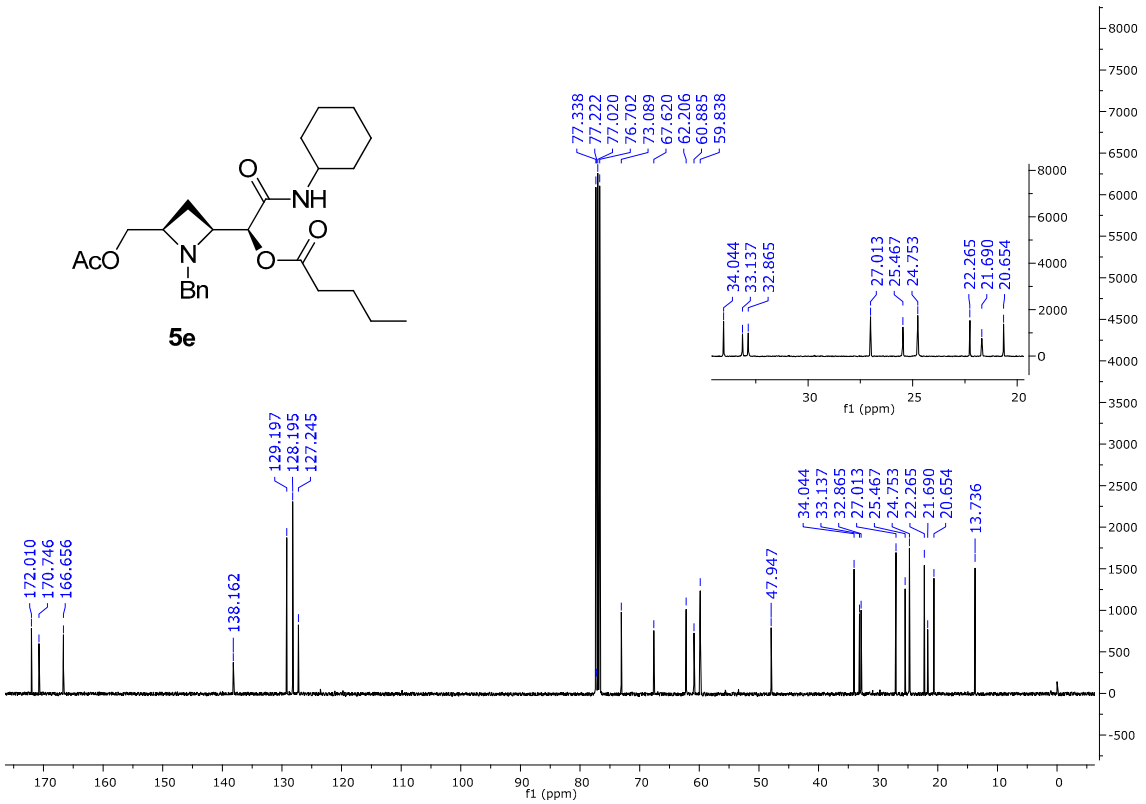

AcO
N
Bn
O
NH
O
O
5d
7.315
7.310
7.297
7.294
7.288
7.280
7.264
7.251
7.247
7.234
7.228
6.041
5.141
5.131
2.437
2.418
2.399
2.159
2.139
2.132
2.119
2.112
2.092
2.045
1.706
1.688
1.683
1.669
1.650
1.631
1.450
1.431
1.413
1.394
1.375
1.872
1.344
0.965
0.947
0.929
3.865
3.833
3.773
3.759
3.713
3.688
3.680
3.559
3.527
3.347
3.328
3.313
3.297
3.278
f1 (ppm)

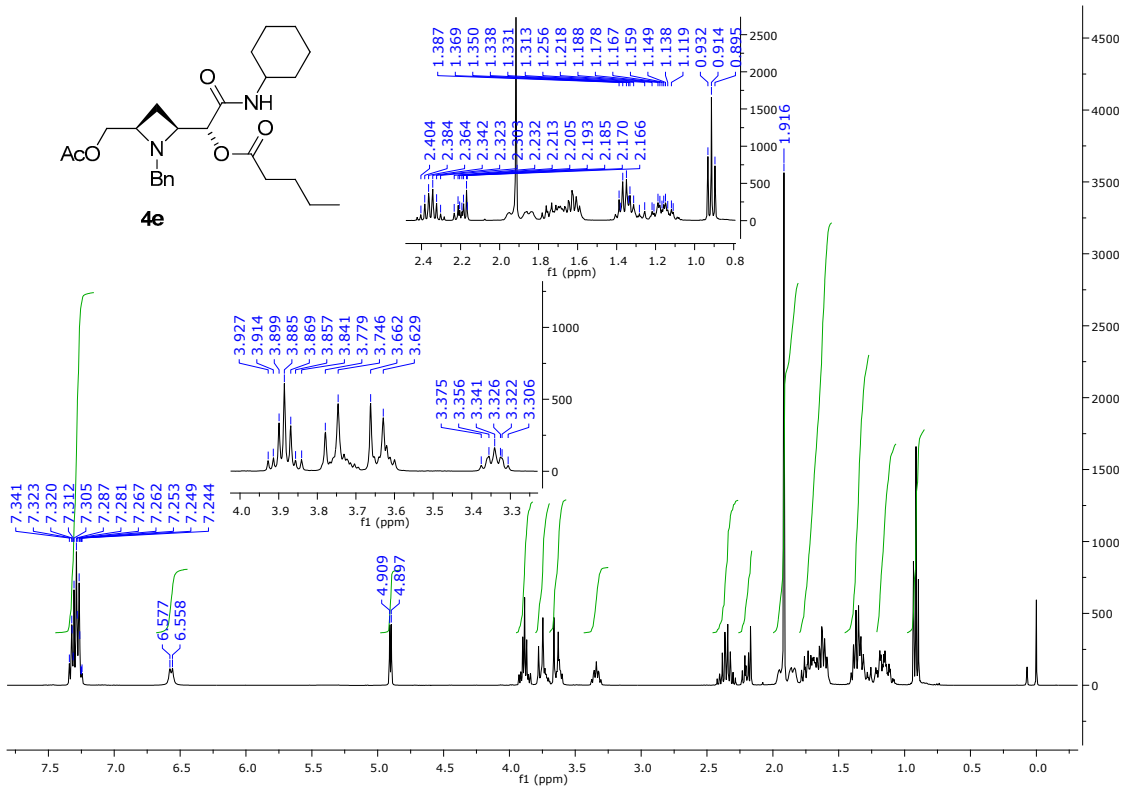

AcO
N
Bn
O
NH
O
O
5d
77.338
77.020
76.702
73.349
172.020
170.744
166.787
137.630
129.345
128.299
127.420
67.421
62.083
60.717
59.880
51.380
34.053
28.666
27.023
22.274
21.876
20.665
13.738
f1 (ppm)

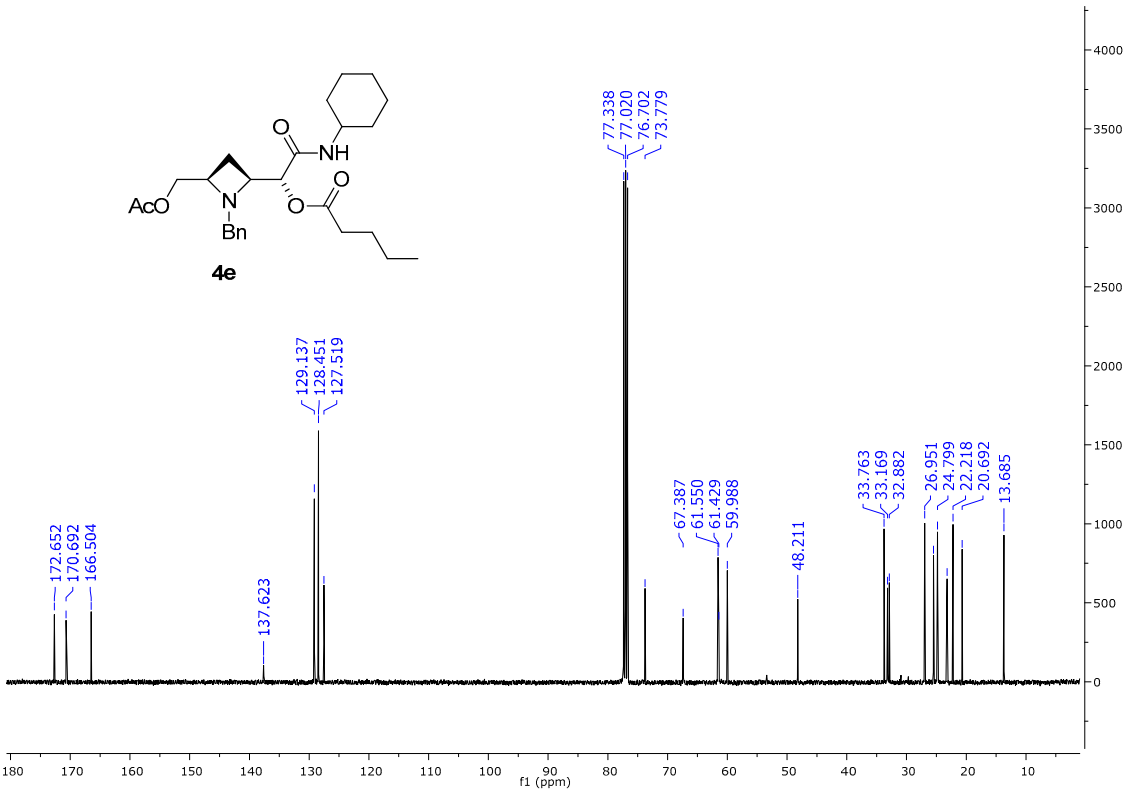

AcO
N
Bn
O
NH
O
O
4d
3.996
3.981
3.930
3.896
3.886
3.852
3.818
3.804
3.790
3.776
3.751
3.717
3.583
3.551
3.500
2.436
2.417
2.398
2.379
2.224
2.216
2.196
2.172
1.939
1.685
1.666
1.647
1.627
1.351
0.940
0.922
0.903
1.351
1.939
7.365
7.361
7.347
7.335
7.318
7.313
7.302
7.287
7.272
7.264
6.797
4.956
4.946
f1 (ppm)

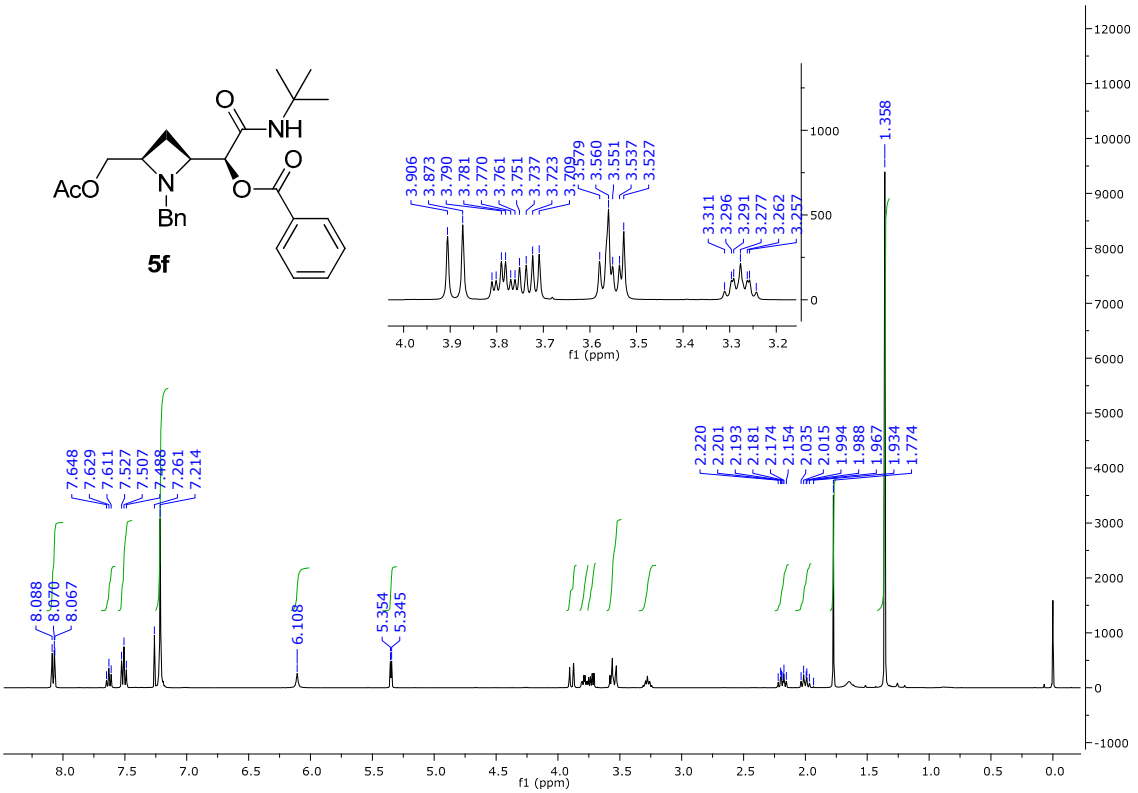

AcO
N
Bn
O
NH
O
O
4d
172.583
170.567
166.199
129.728
129.400
128.671
128.309
128.098
73.225
66.340
61.386
59.710
51.520
33.710
28.623
26.879
23.115
22.199
20.613
13.650
f1 (ppm)


AcO
N
Bn
O
NH
O
O
5e
7.301
7.295
7.281
7.265
7.249
7.231
7.220
7.214
6.024
6.004
5.230
5.221
3.847
3.814
3.754
3.726
3.712
3.667
3.659
3.646
3.515
3.483
3.277
3.262
3.247
3.243
2.432
2.413
2.170
2.112
2.105
2.093
2.086
1.659
1.640
1.439
1.420
1.401
1.383
1.169
1.140
0.970
0.951
0.933
1.859
f1 (ppm)


AcO
N
Bn
O
NH
O
O
5e
77.338
77.222
77.020
76.702
73.089
67.620
62.206
60.885
59.838
34.044
33.137
32.865
27.013
25.467
24.753
22.265
21.690
20.654
129.197
128.195
127.245
172.010
170.746
166.656
138.162
47.947
13.736
f1 (ppm)


AcO
N
Bn
O
NH
O
O
4e
f1 (ppm)


AcO
N
Bn
O
NH
O
O
4e
f1 (ppm)


AcO
N
Bn
O
NH
O
O
5f
3.906
3.873
3.790
3.781
3.770
3.761
3.751
3.737
3.723
3.709
3.579
3.560
3.551
3.537
3.527
3.311
3.296
3.291
3.277
3.262
3.257
1.358
f1 (ppm)
7.648
7.629
7.611
7.527
7.507
7.488
7.261
7.214
8.088
8.070
8.067
6.108
5.354
5.345
2.220
2.201
2.193
2.181
2.174
2.154
2.035
2.015
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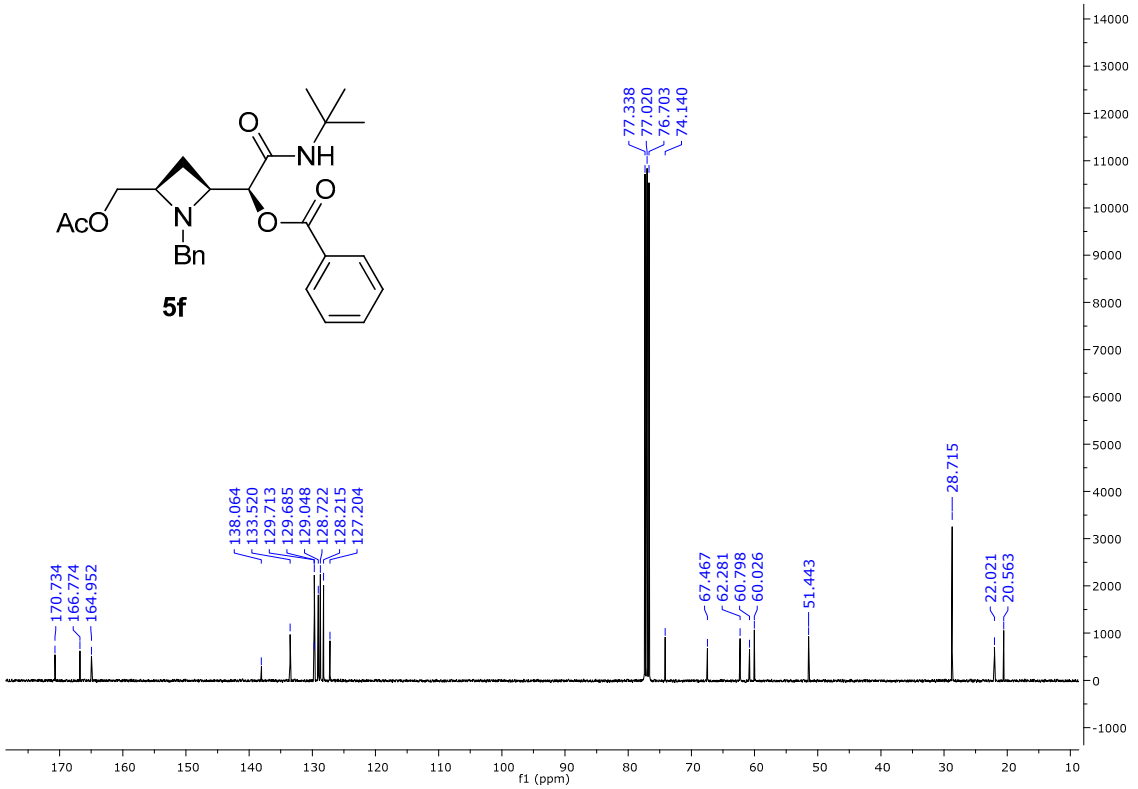
1.988
1.967
1.934
1.774
f1 (ppm)


AcO
N
Bn
O
NH
O
O
5f
77.338
77.020
76.703
74.140
138.064
133.520
129.713
129.685
129.048
128.722
128.215
127.204
170.734
166.774
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67.467
62.281
60.798
60.026
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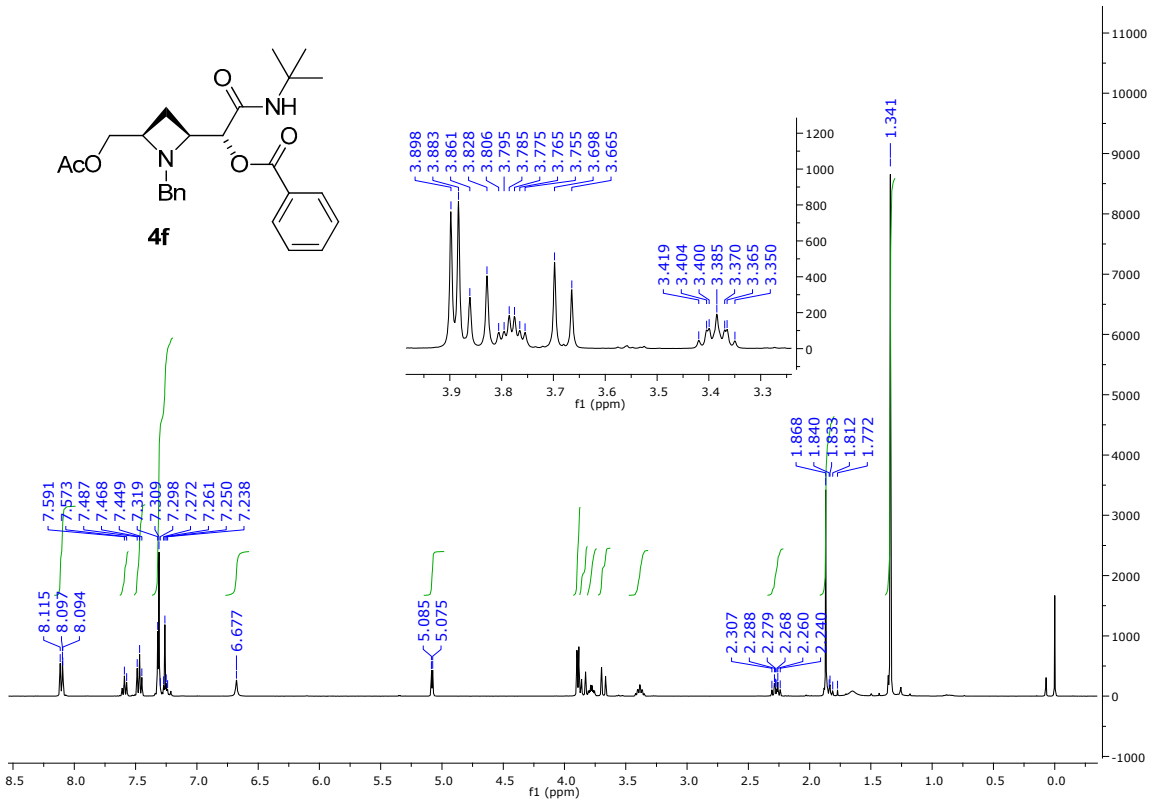
28.715
22.021
20.563
f1 (ppm)


AcO
N
Bn
O
NH
O
O
4f
3.898
3.883
3.861
3.828
3.806
3.795
3.785
3.775
3.765
3.755
3.698
3.665
3.419
3.404
3.400
3.385
3.370
3.365
3.350
1.341
1.868
1.840
1.833
1.812
1.772
7.591
7.573
7.487
7.468
7.449
7.319
7.309
7.298
7.272
7.261
7.250
7.238
8.115
8.097
8.094
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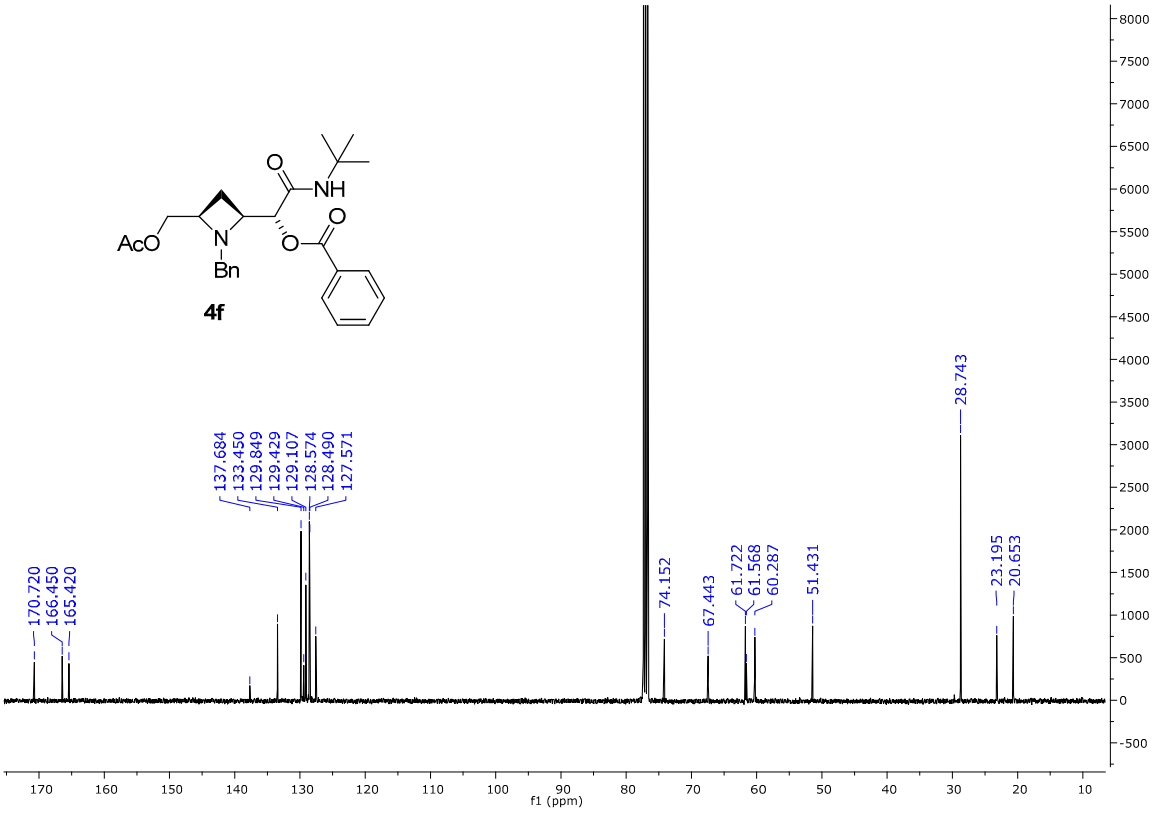
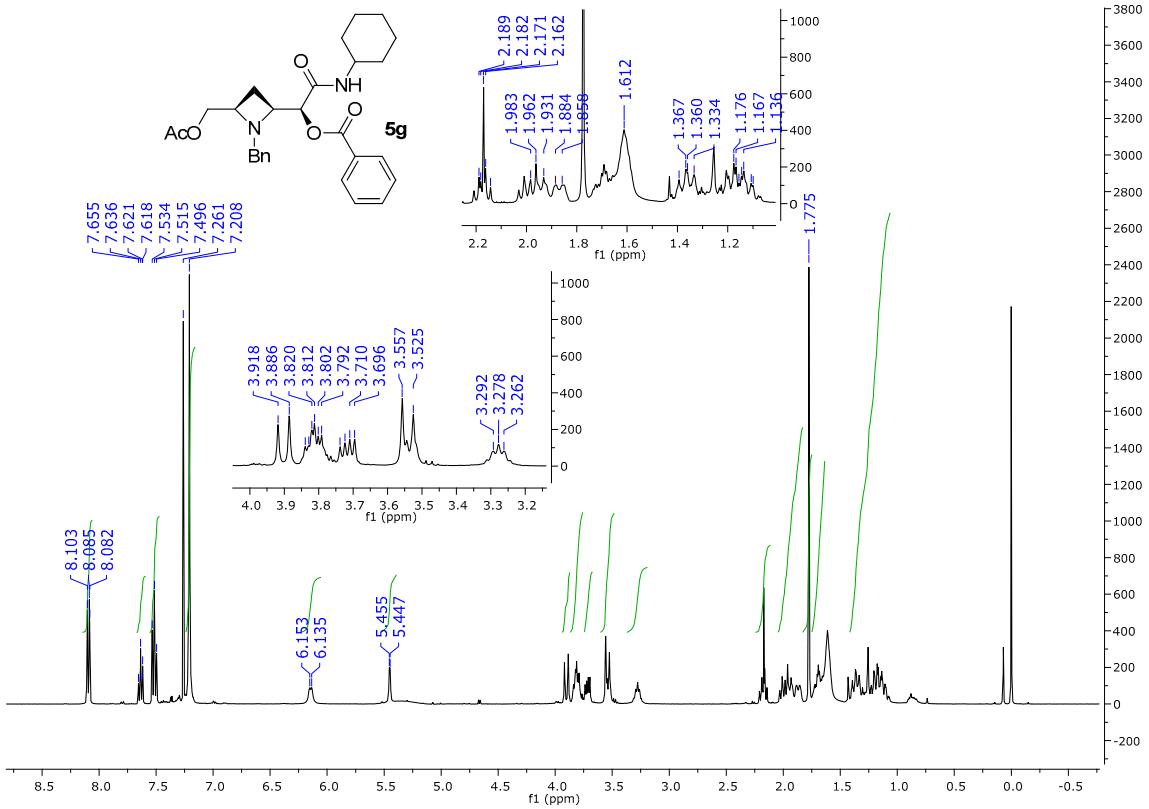
5.075
2.307
2.288
2.279
2.268
2.260
2.240
f1 (ppm)


AcO
N
Bn
O
NH
O
O
4f
137.684
133.450
129.849
129.429
129.107
128.574
128.490
127.571
170.720
166.450
165.420
74.152
67.443
61.722
61.568
60.287
51.431
28.743
23.195
20.653
f1 (ppm)


AcO
N
Bn
O
NH
O
O
5g
7.655
7.636
7.621
7.618
7.534
7.515
7.496
7.261
7.208
8.103
8.085
8.082
6.153
6.135
5.455
5.447
3.918
3.886
3.820
3.812
3.802
3.792
3.710
3.696
3.557
3.525
3.292
3.278
3.262
2.189
2.182
2.171
2.162
1.983
1.962
1.931
1.884
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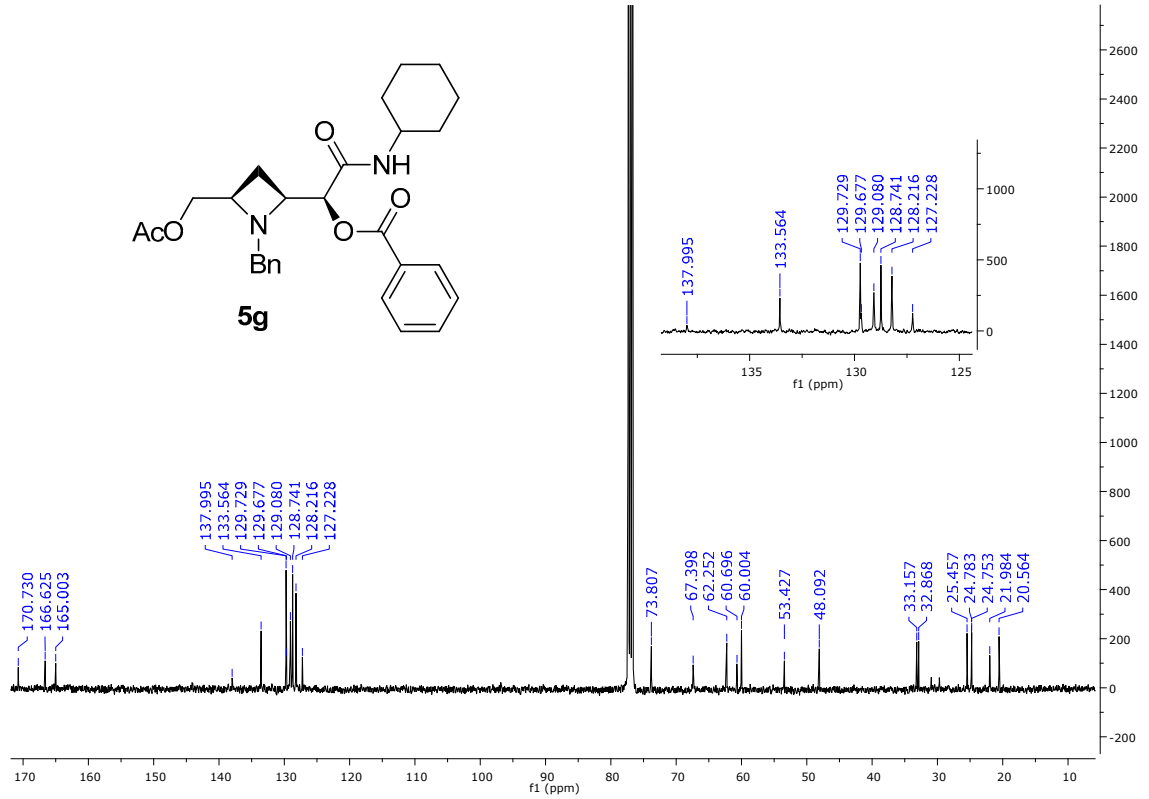
1.612
1.367
1.360
1.334
1.176
1.167
1.136
1.775
f1 (ppm)


AcO
N
Bn
O
NH
O
O
5g
137.995
133.564
129.729
129.677
129.080
128.741
128.216
127.228
170.730
166.625
165.003
73.807
67.398
62.252
60.696
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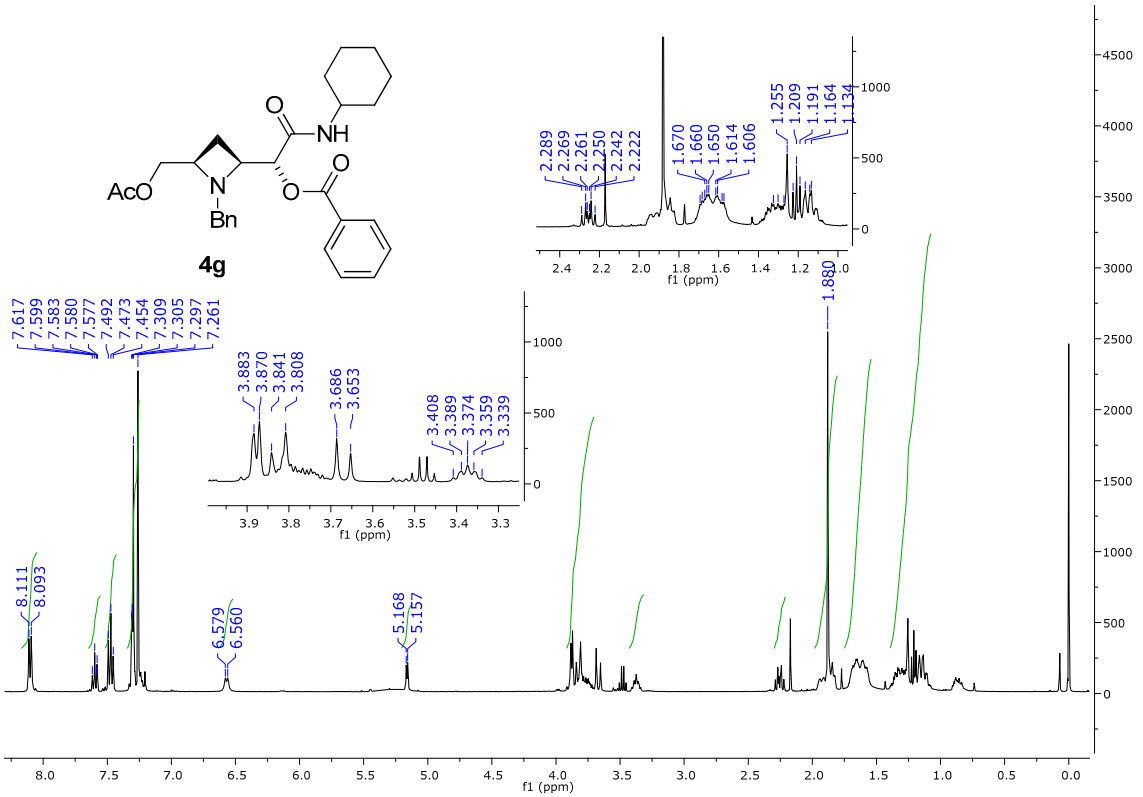
48.092
33.157
32.868
25.457
24.783
24.753
21.984
20.564
f1 (ppm)


4g
7.617
7.599
7.583
7.580
7.577
7.492
7.473
7.454
7.309
7.305
7.297
7.261
8.111
8.093
6.579
6.560
5.168
5.157
3.883
3.870
3.841
3.808
3.686
3.653
3.408
3.389
3.374
3.359
3.339
2.289
2.269
2.261
2.250
2.242
2.222
1.670
1.660
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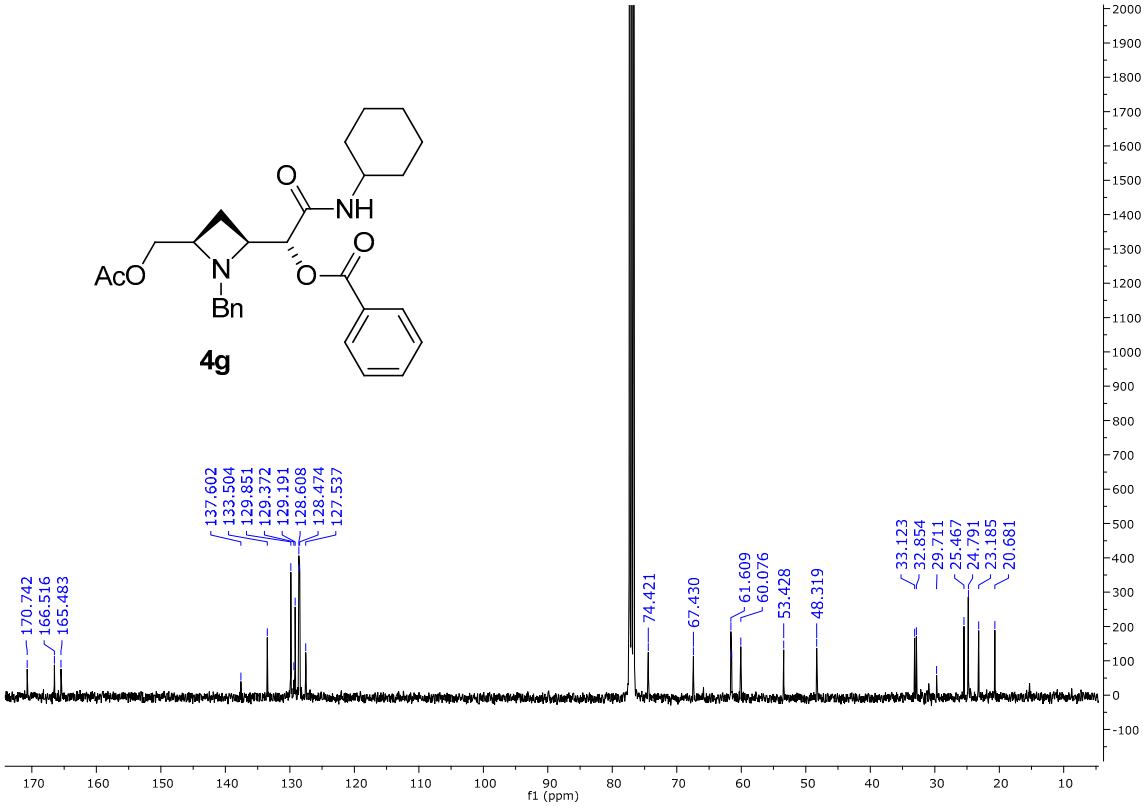
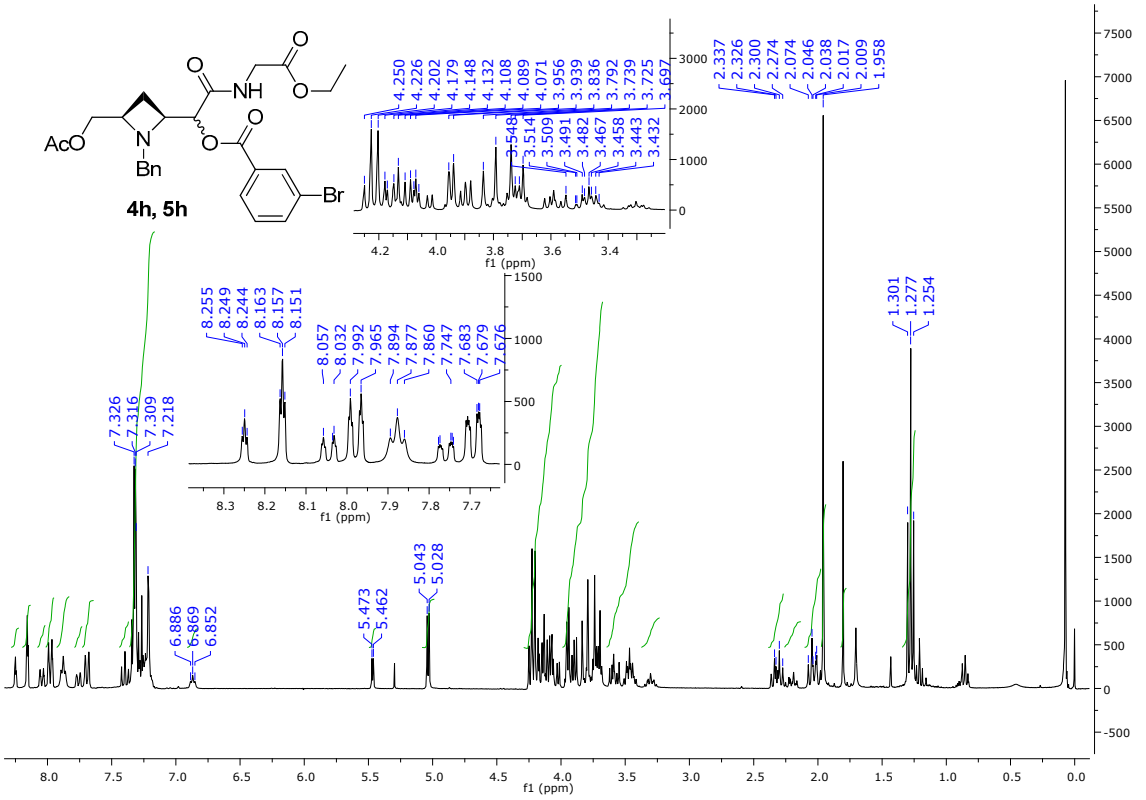
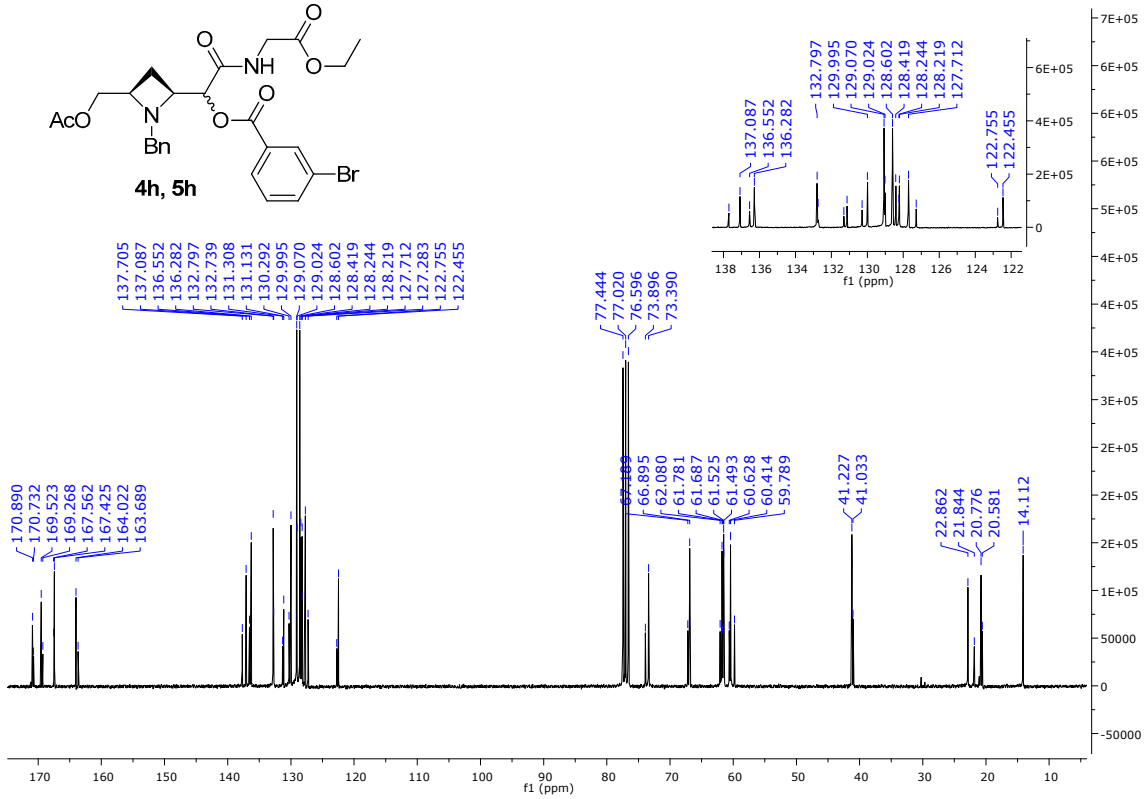
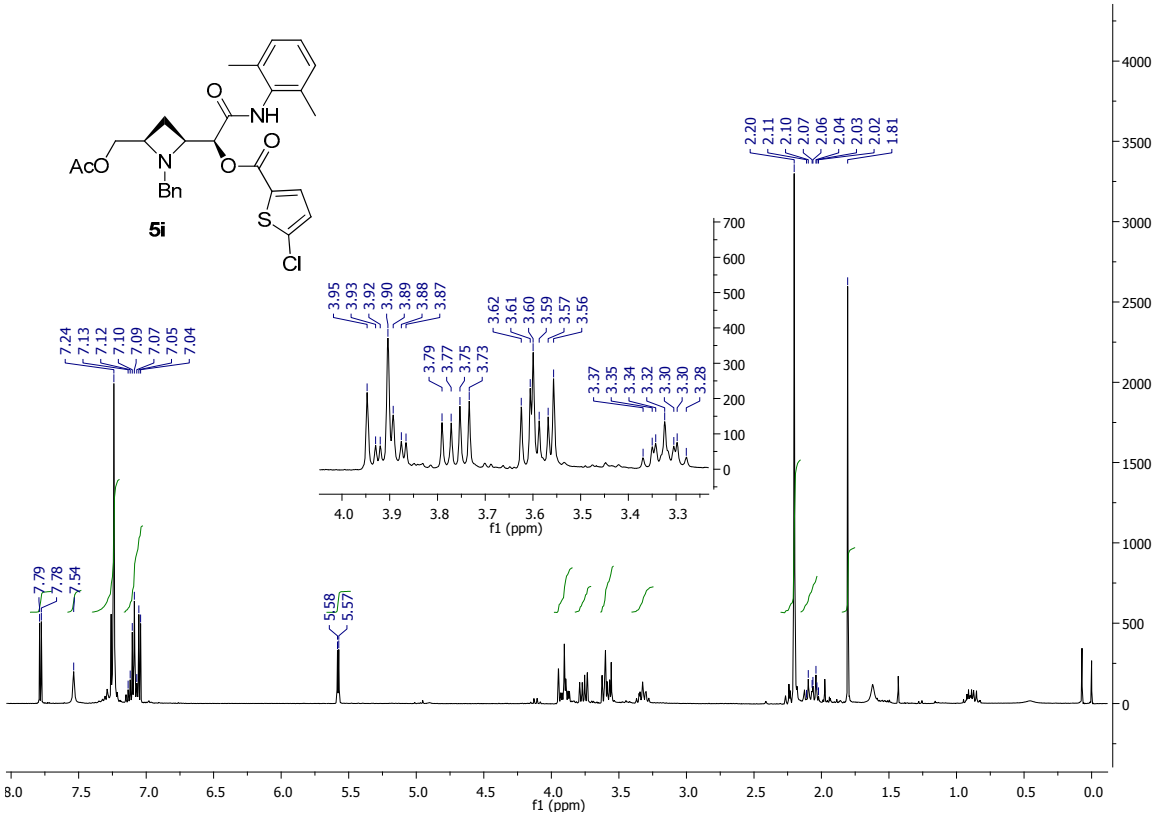
1.606
1.255
1.209
1.191
1.164
1.134
1.880
f1 (ppm)


4g
170.742
166.516
165.483
137.602
133.504
129.851
129.372
129.191
128.608
128.474
127.537
74.421
67.430
61.609
60.076
53.428
48.319
33.123
32.854
29.711
25.467
24.791
23.185
20.681
f1 (ppm)


AcO
N
Bn
NH
O
O
O
O
Br
4h, 5h
f1 (ppm)


AcO
N
Bn
NH
O
O
O
O
Br
4h, 5h
f1 (ppm)


AcO
N
Bn
O
NH
O
O
S
Cl
5i
7.79
7.78
7.54
7.24
7.13
7.12
7.10
7.09
7.07
7.05
7.04
5.58
5.57
3.95
3.93
3.92
3.90
3.89
3.88
3.87
3.79
3.77
3.75
3.73
3.62
3.61
3.60
3.59
3.57
3.56
3.37
3.35
3.34
3.32
3.30
3.30
3.28
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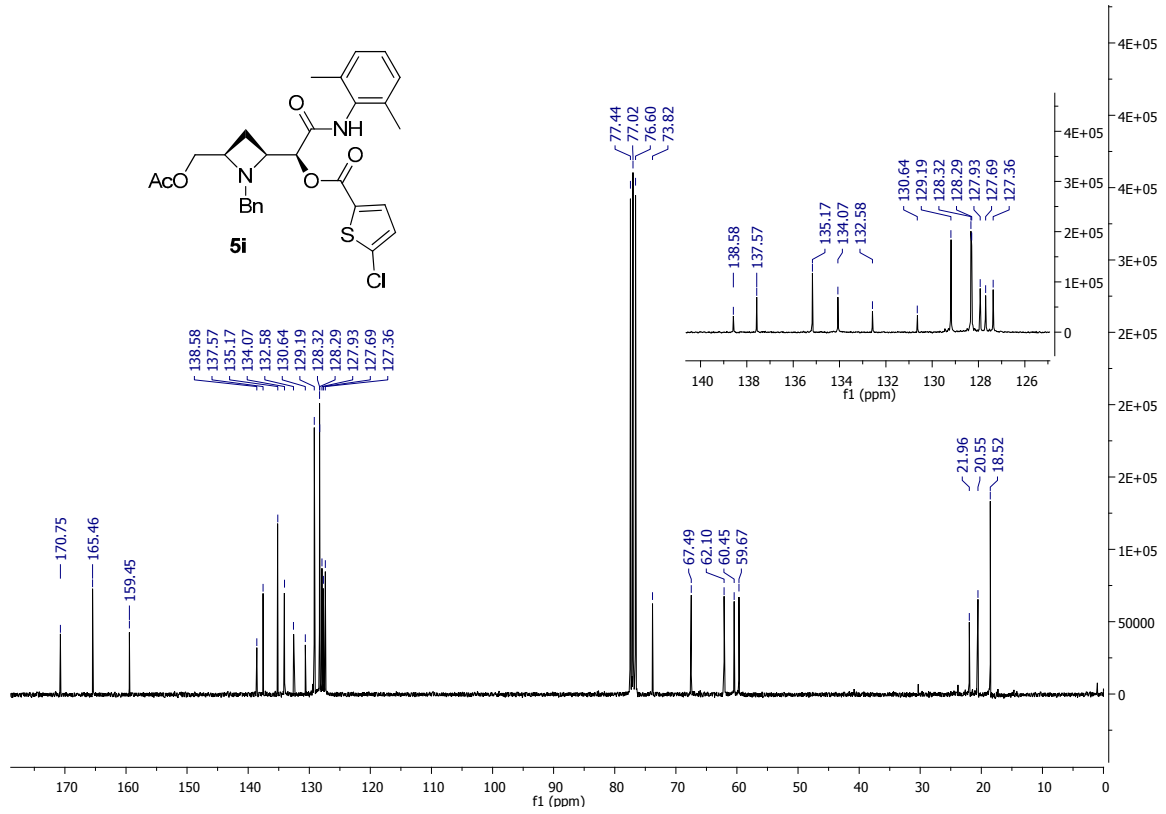
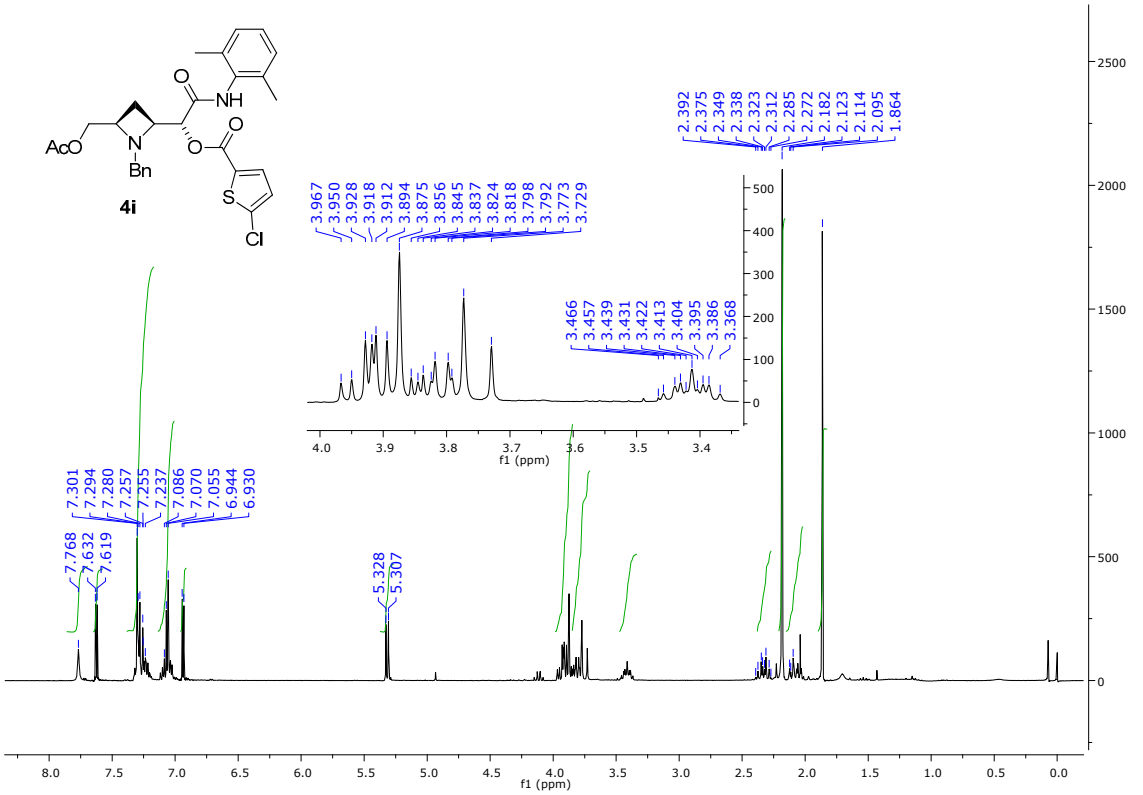
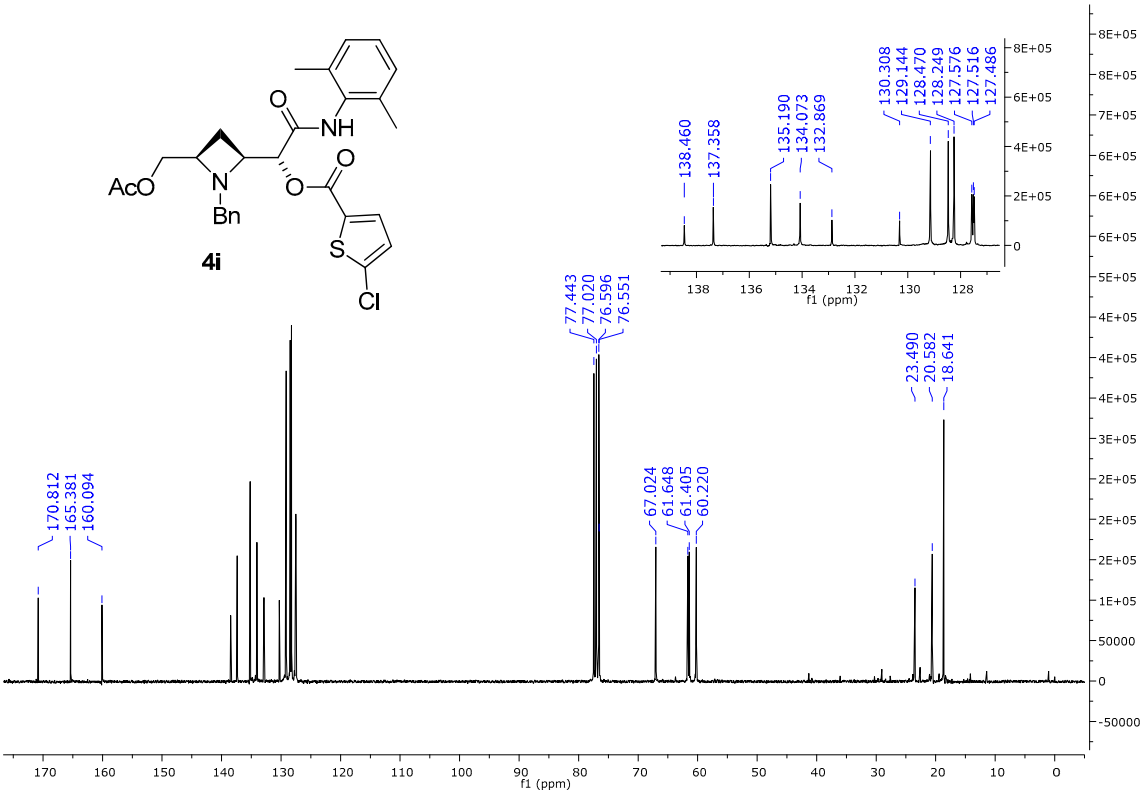
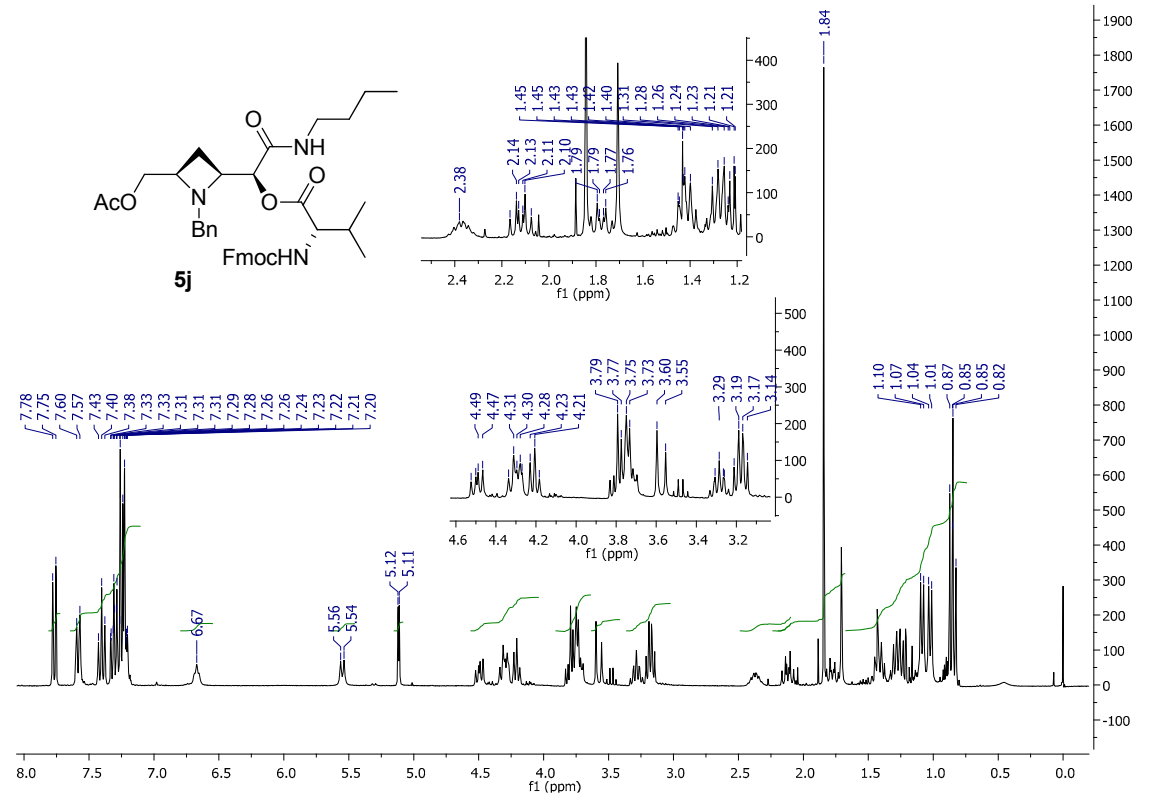
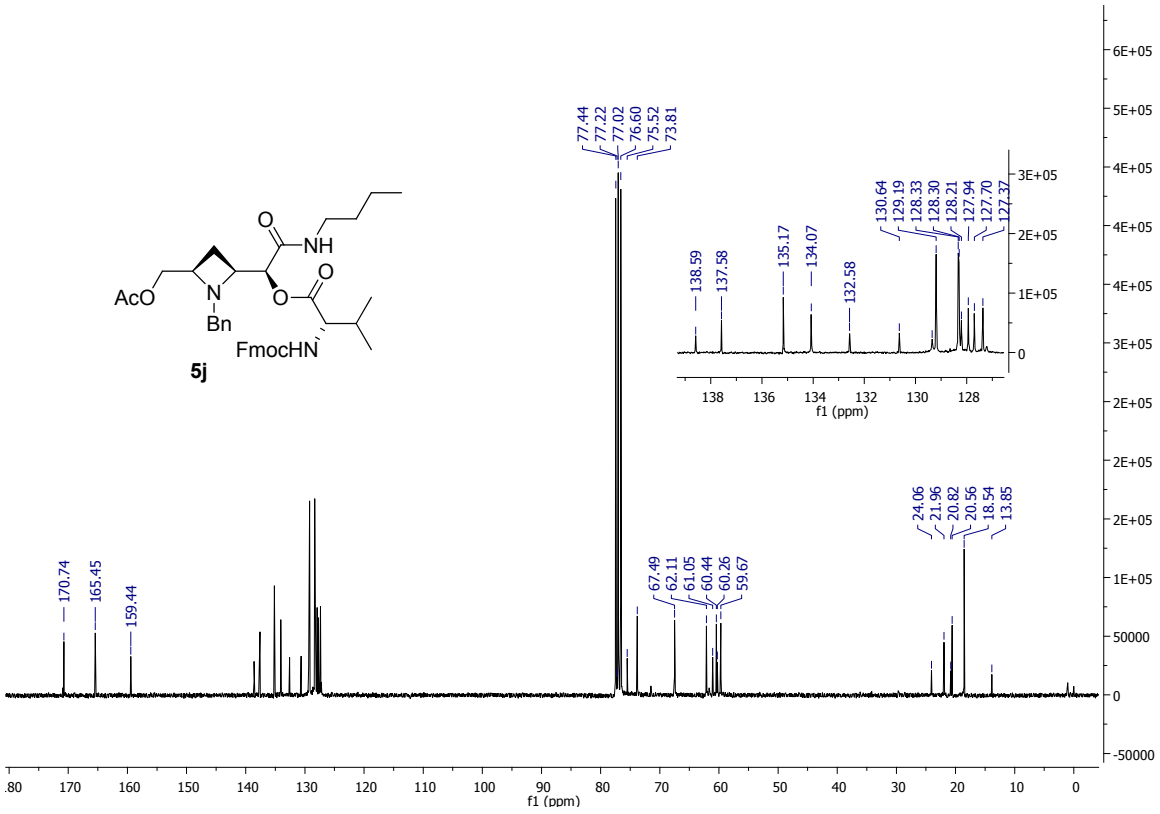
2.11
2.10
2.07
2.06
2.04
2.03
2.02
1.81
f1 (ppm)

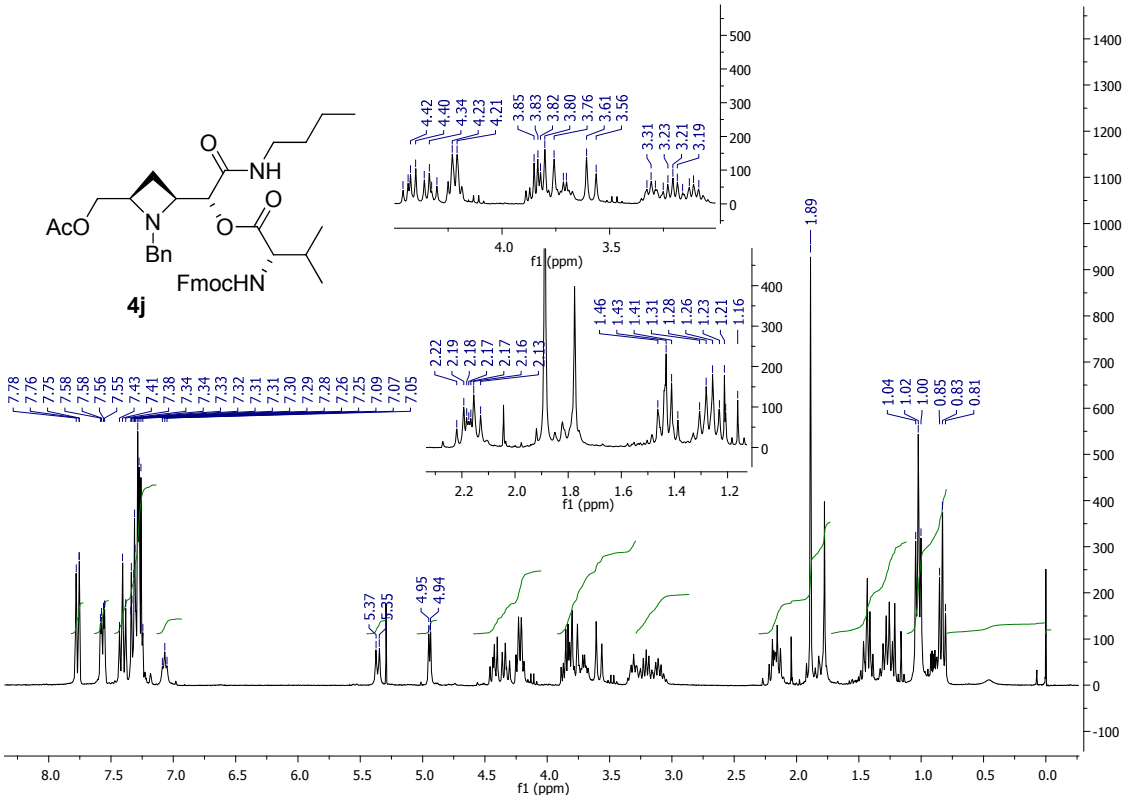

AcO
N
Bn
O
NH
O
O
S
Cl
5i
77.44
77.02
76.60
73.82
138.58
137.57
135.17
134.07
132.58
130.64
129.19
128.32
128.29
127.93
127.69
127.36
170.75
165.46
159.45
67.49
62.10
60.45
59.67
21.96
20.55
18.52
f1 (ppm)

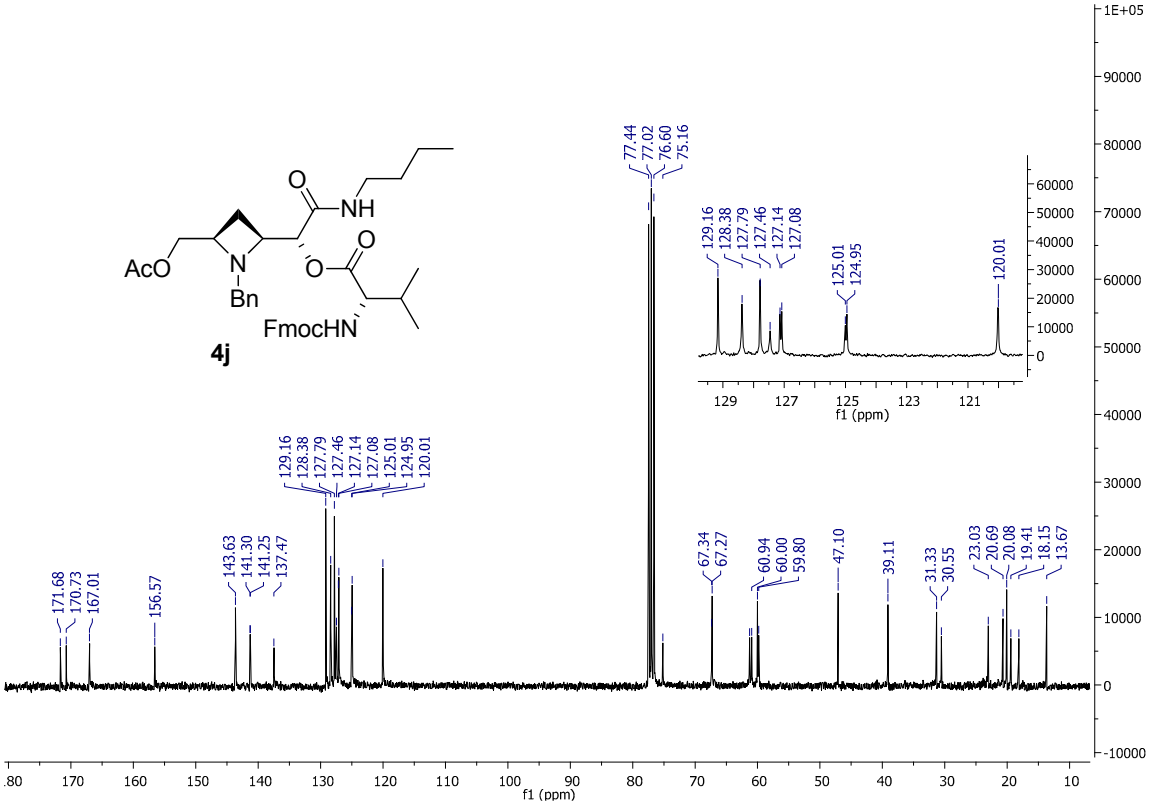

4i
7.768
7.632
7.619
7.301
7.294
7.280
7.257
7.255
7.237
7.086
7.070
7.055
6.944
6.930
5.328
5.307
3.967
3.950
3.928
3.918
3.912
3.894
3.875
3.856
3.845
3.837
3.824
3.818
3.798
3.792
3.773
3.729
3.466
3.457
3.439
3.431
3.422
3.413
3.404
3.395
3.386
3.368
2.392
2.375
2.349
2.338
2.323
2.312
2.285
2.272
2.182
2.123
2.114
2.095
1.864
f1 (ppm)

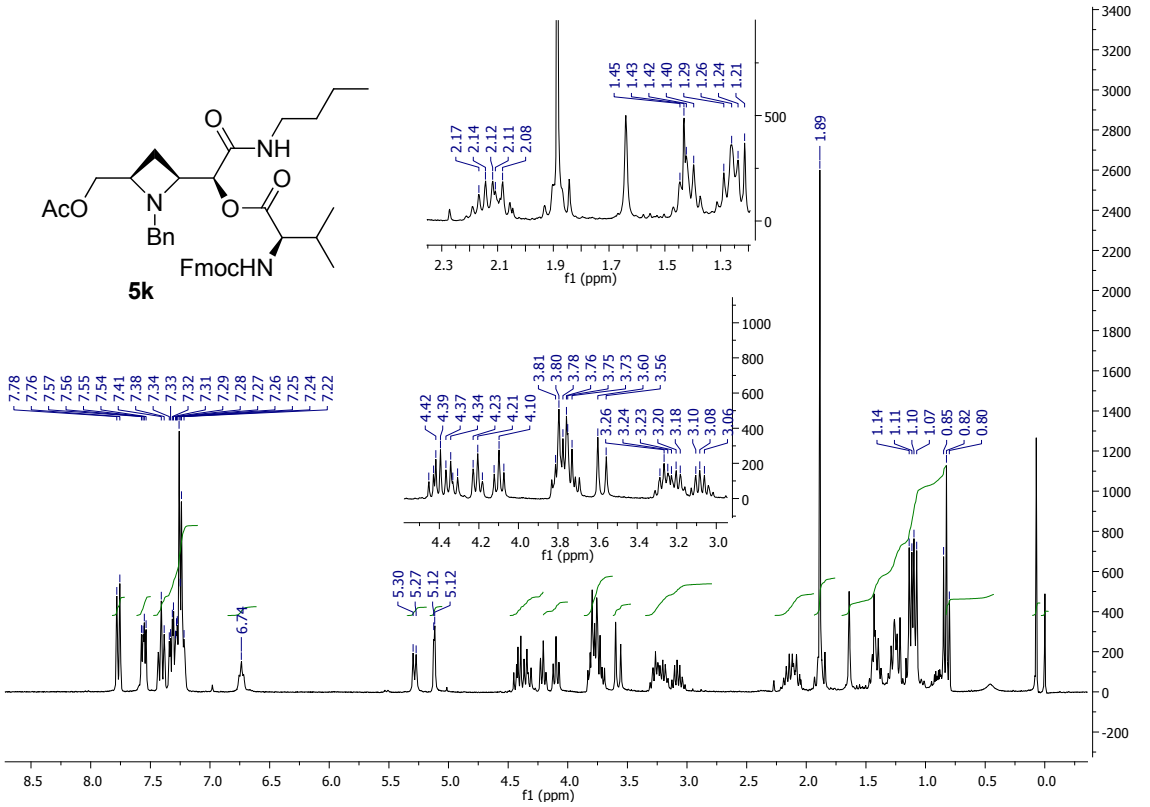

4i
138.460
137.358
135.190
134.073
132.869
130.308
129.144
128.470
128.249
127.576
127.516
127.486
77.443
77.020
76.596
76.551
67.024
61.648
61.405
60.220
23.490
20.582
18.641
170.812
165.381
160.094
f1 (ppm)

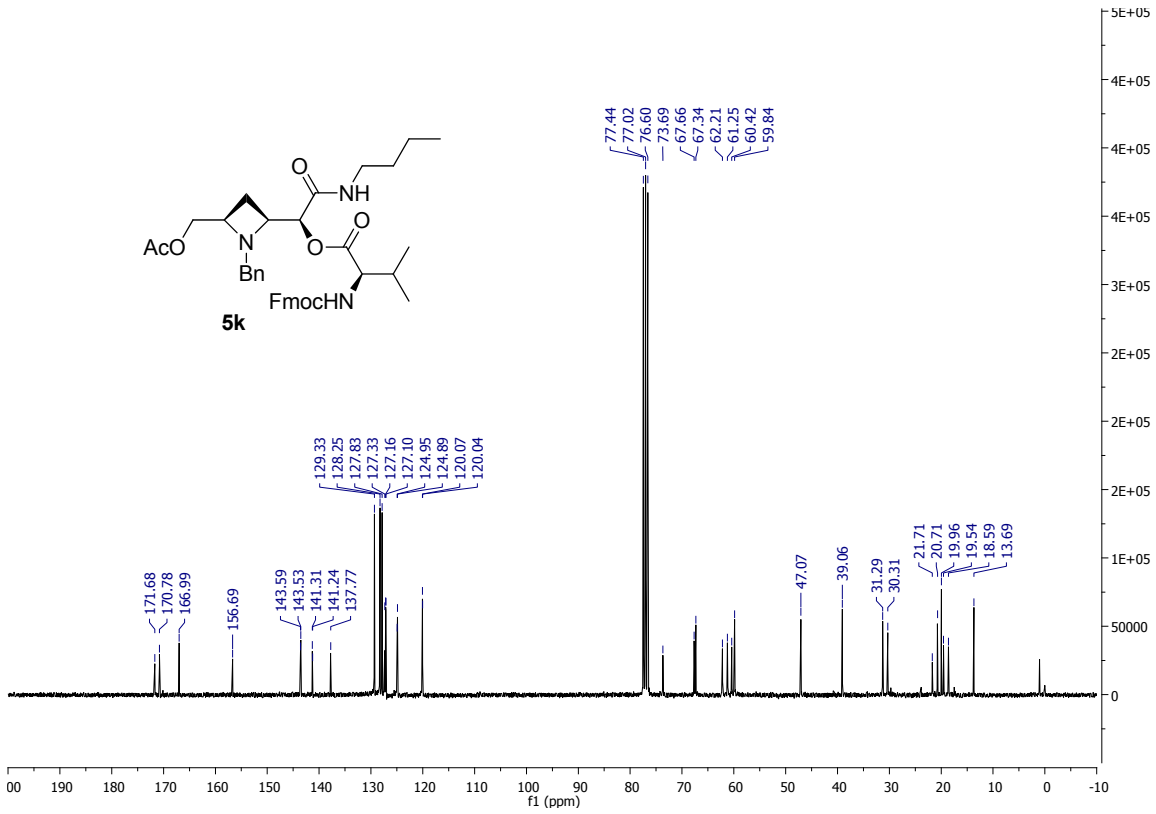

AcO
N
Bn
O
NH
O
O
FmocHN
5j
f1 (ppm)

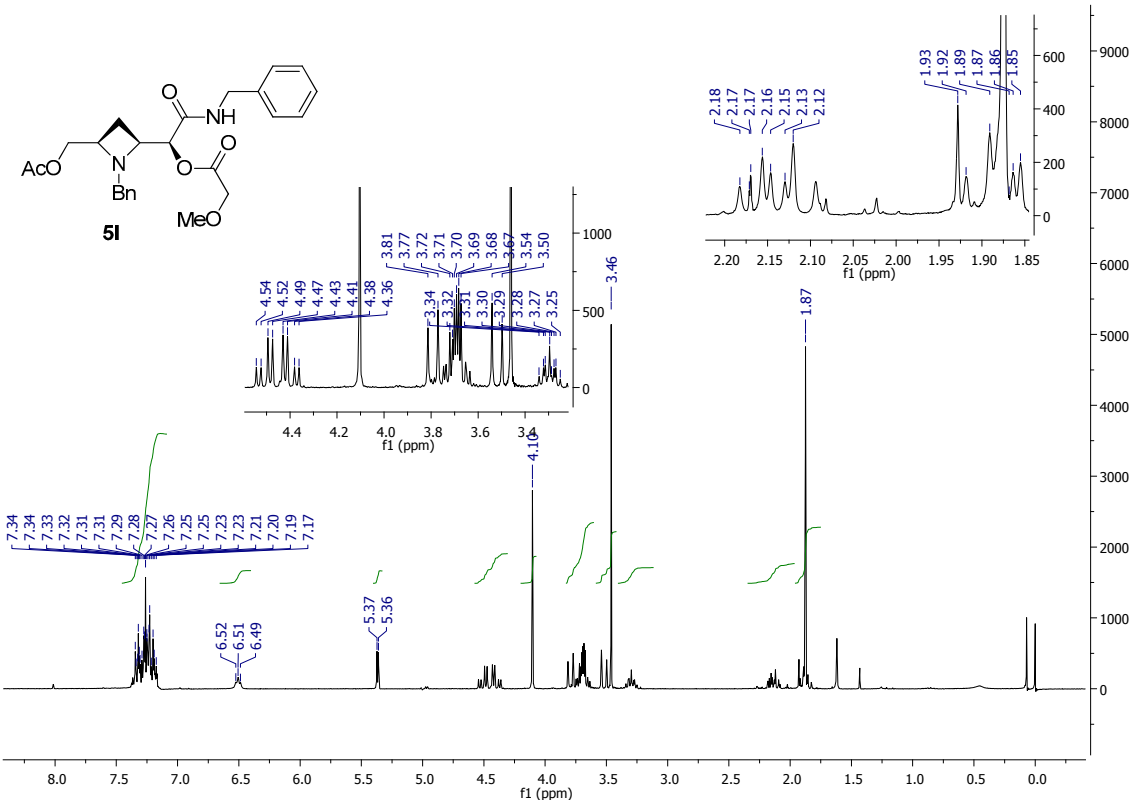

AcO
N
Bn
O
NH
O
O
FmocHN
5j
f1 (ppm)

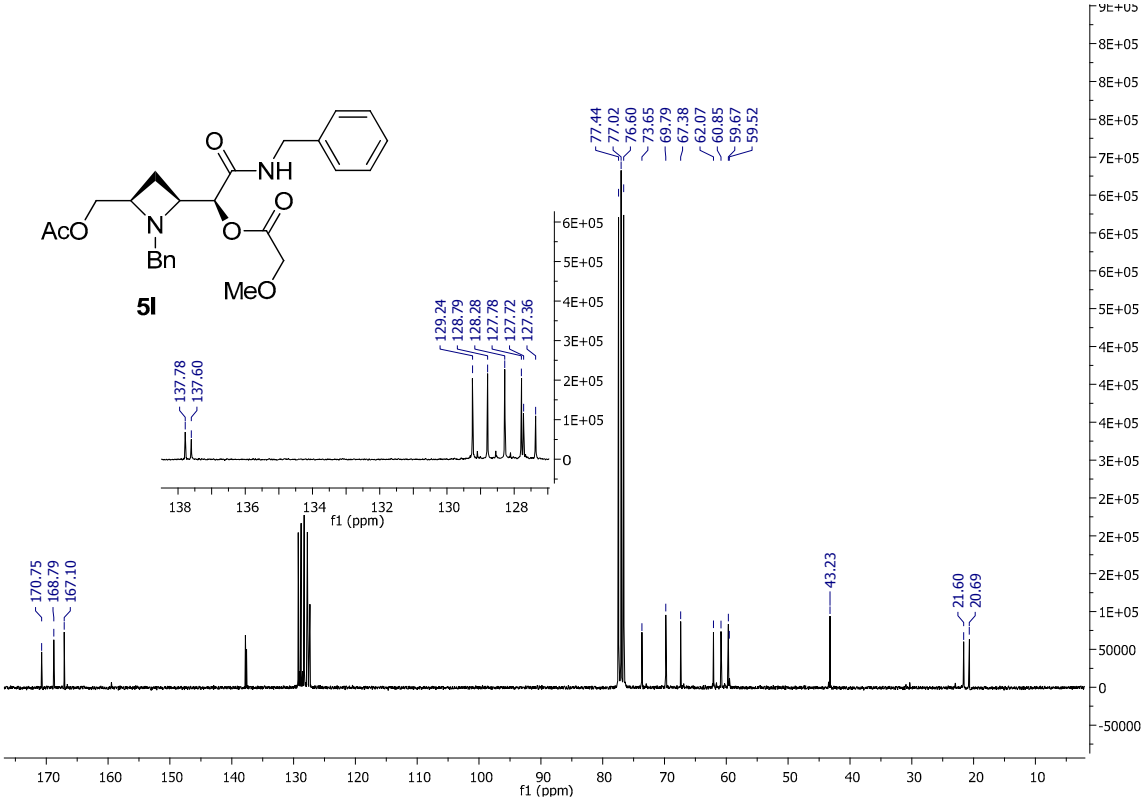

AcO
N
Bn
O
NH
O
O
FmocHN
4j
f1 (ppm)

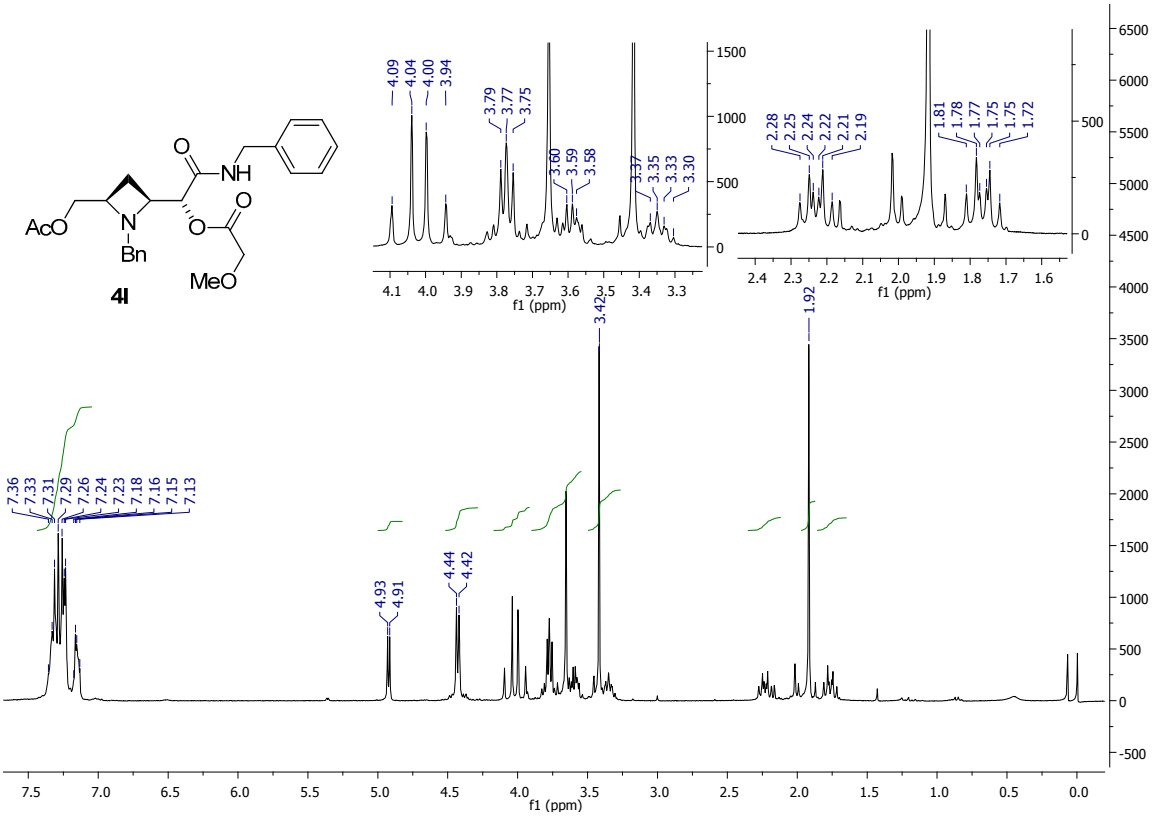

AcO
N
Bn
O
NH
O
O
FmocHN
4j
f1 (ppm)

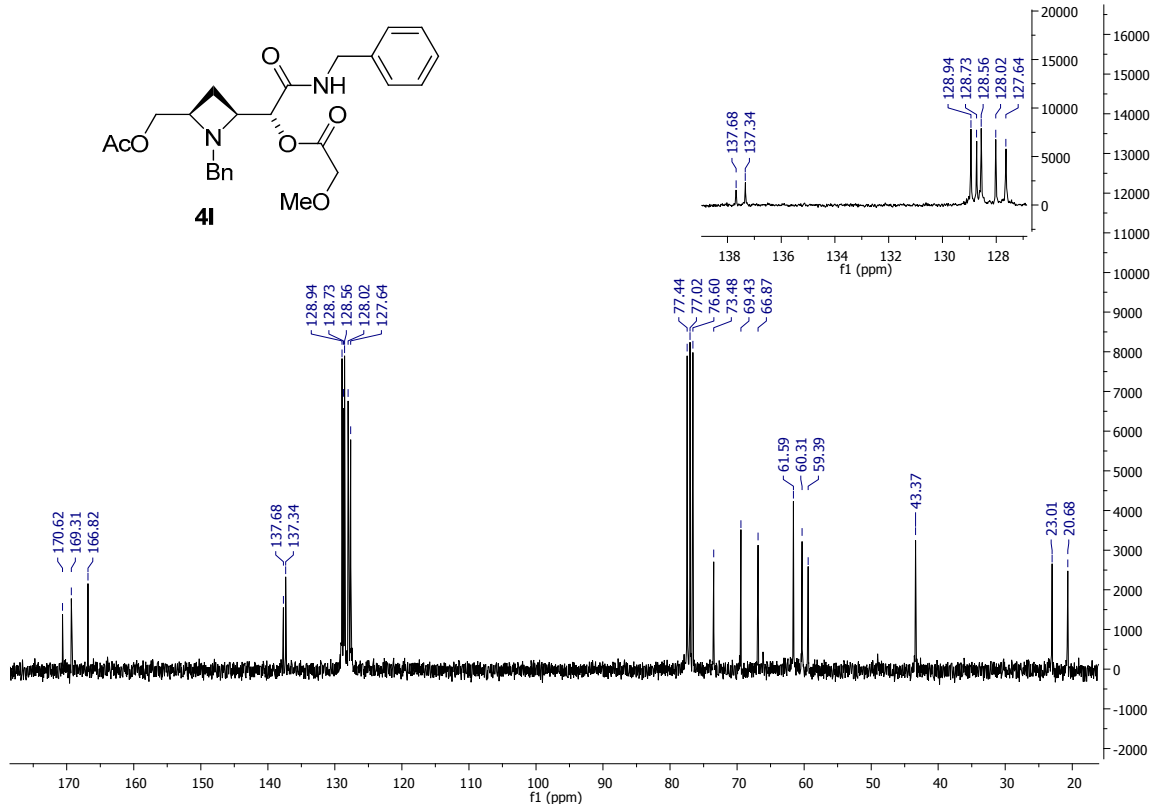

AcO
N
Bn
O
NH
O
O
FmocHN
5k
f1 (ppm)

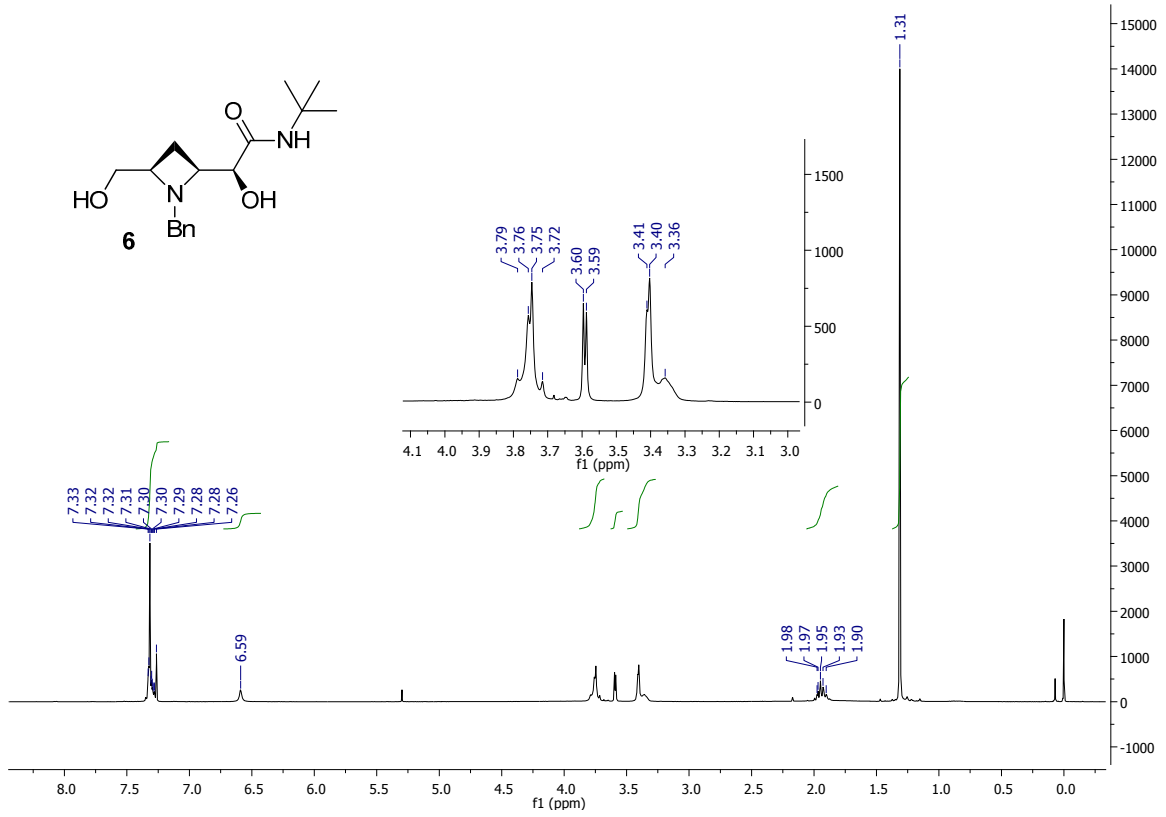

AcO
N
Bn
O
NH
O
O
FmocHN
5k
f1 (ppm)

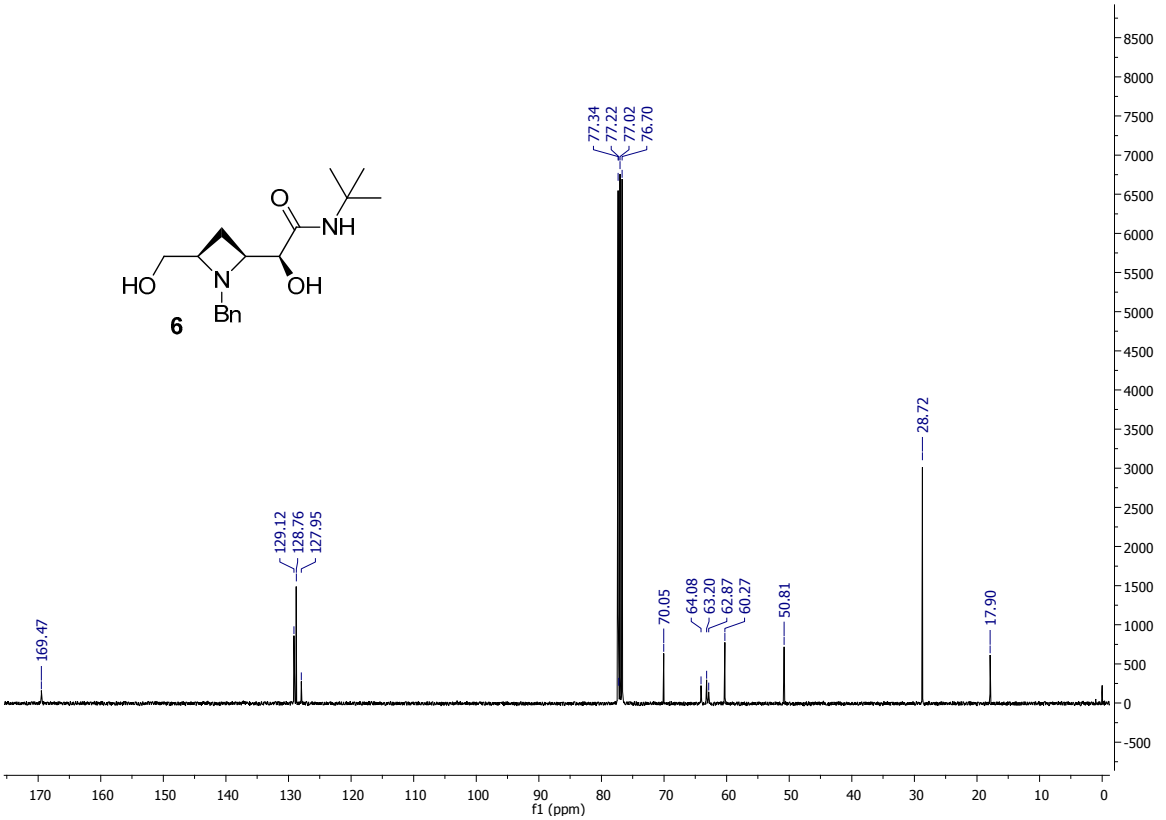

AcO
N
Bn
NH
O
O
O
MeO
5l
f1 (ppm)

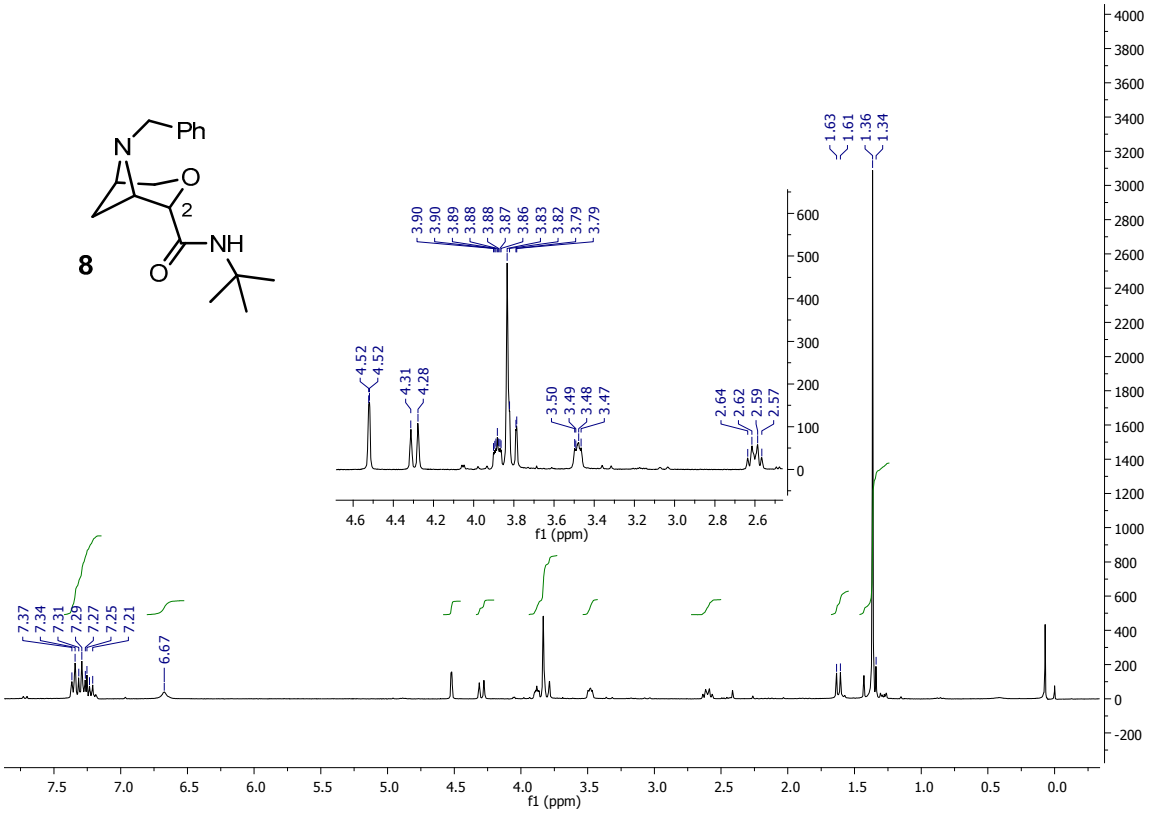

AcO
N
Bn
NH
O
O
O
MeO
5l
f1 (ppm)

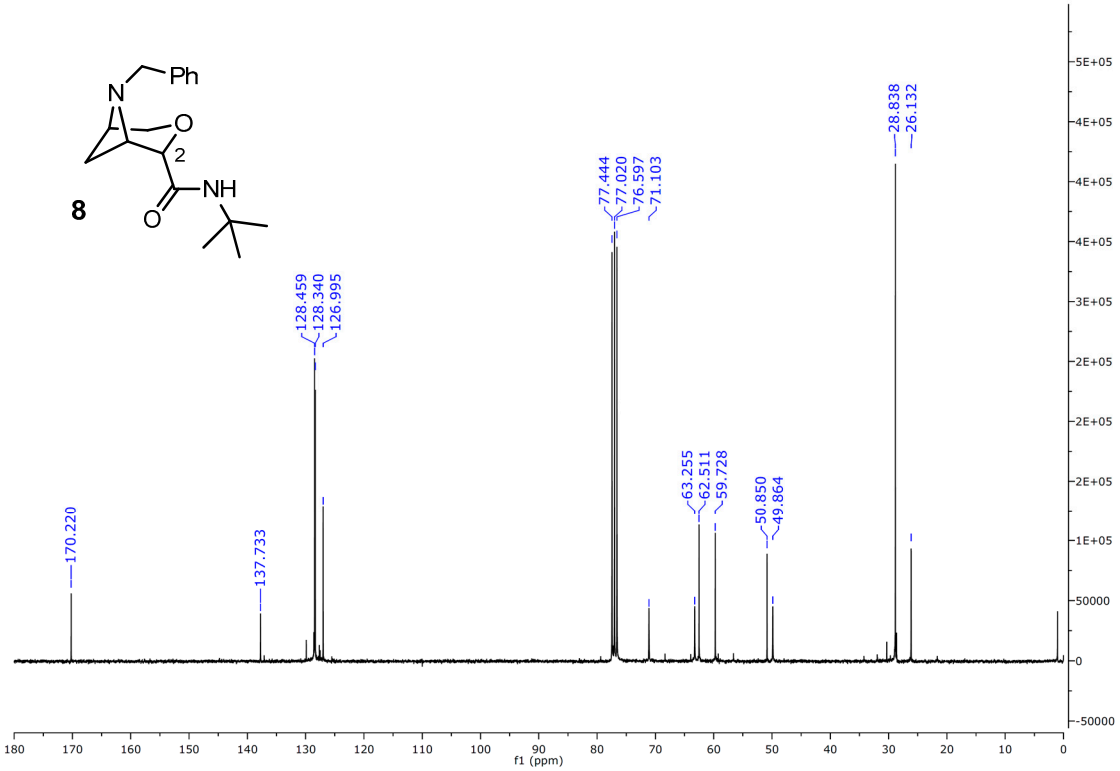

AcO
N
Bn
O
NH
O
O
MeO
4l
4.09
4.04
4.00
3.94
3.79
3.77
3.75
3.60
3.59
3.58
3.37
3.35
3.33
3.30
2.28
2.25
2.24
2.22
2.21
2.19
1.81
1.78
1.77
1.75
1.75
1.72
f1 (ppm)
7.36
7.33
7.31
7.29
7.26
7.24
7.23
7.18
7.16
7.15
7.13
4.93
4.91
4.44
4.42
3.42
1.92
f1 (ppm)


AcO
N
Bn
O
NH
O
O
MeO
4l
137.68
137.34
128.94
128.73
128.56
128.02
127.64
f1 (ppm)
170.62
169.31
166.82
137.68
137.34
128.94
128.73
128.56
128.02
127.64
77.44
77.02
76.60
73.48
69.43
66.87
61.59
60.31
59.39
43.37
23.01
20.68
f1 (ppm)


HO
N
Bn
O
NH
OH
6
1.31
3.79
3.76
3.75
3.72
3.60
3.59
3.41
3.40
3.36
7.33
7.32
7.32
7.31
7.30
7.30
7.29
7.28
7.28
7.26
6.59
1.98
1.97
1.95
1.93
1.90
f1 (ppm)


HO
N
Bn
O
NH
OH
6
77.34
77.22
77.02
76.70
169.47
129.12
128.76
127.95
70.05
64.08
63.20
62.87
60.27
50.81
28.72
17.90
f1 (ppm)


Ph
N
O
2
NH
O
8
1.63
1.61
1.36
1.34
3.90
3.90
3.89
3.88
3.88
3.87
3.86
3.83
3.82
3.79
3.79
4.52
4.52
4.31
4.28
3.50
3.49
3.48
3.47
2.64
2.62
2.59
2.57
f1 (ppm)
7.37
7.34
7.31
7.29
7.27
7.25
7.21
6.67
f1 (ppm)


Ph
N
O
2
NH
O
8
28.838
26.132
77.444
77.020
76.597
71.103
128.459
128.340
126.995
63.255
62.511
59.728
50.850
49.864
170.220
137.733
f1 (ppm)