Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Divergent synthesis of various iminocyclitols from D-ribose

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Supplementary Information

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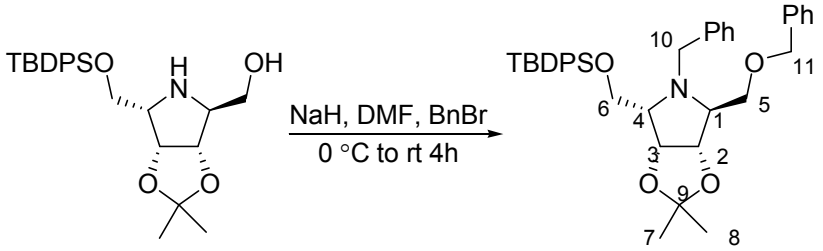
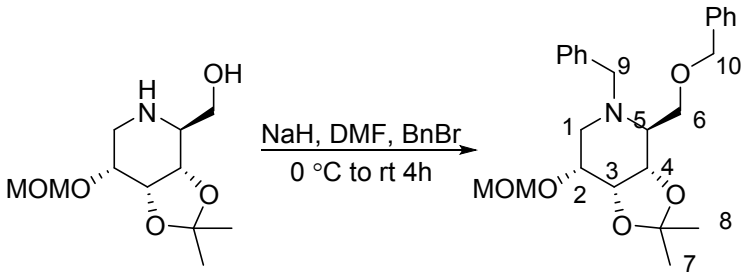
Procedure for Benzyl Protection:

Compound**11a**(0.8 g, 2 mmol, 1 equiv) in DMF (10 mL) was added to a stirred suspension of sodium hydride (0.14 g, 6 mmol, 3 equiv) in DMF (5 mL) at 0 ºC under nitrogen atmosphere and stirring was continued for 20 min. Benzyl bromide (0.7 mL, 6 mmol, 3 equiv) was added to the reaction mixture and stirred at room temperature for 4 h. After complete disappearance of starting material on TLC, the reaction was quenched with water (30 mL). Organic layer was extracted with diethyl ether (2×20 mL), and the combined organic phases were washed with brine (1×20 mL), dried over anhydrous Na_2SO_4, filtered, concentrated, and purified by column chromatography.



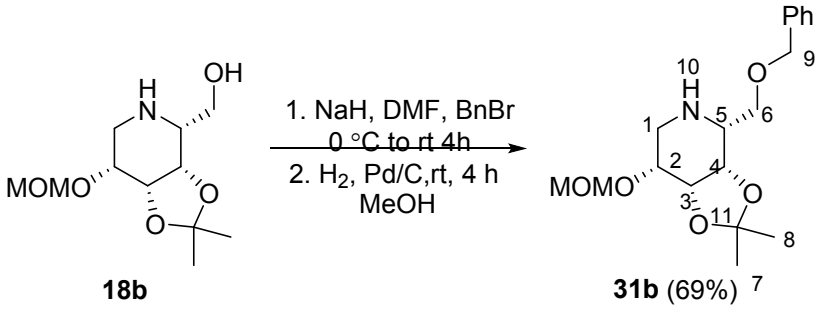
10a **30a** (89%)

Compound **(30)**: Column chromatography (petroleum ether/EtOAc, 9:1); oily liquid (0.21 g, 0.33 mmol, 89%); $[\alpha]_D^{25}$ = +12.8 (*c* 0.8, $CHCl_3$); IR ν_{max} (thin film):3068, 1561 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.74-7.68 (m, 4H, Ar-H), 7.45-7.17 (m, 16H, Ar-H), 4.73-4.71 (m, 1H, C_3H), 4.66 (m, 3H, $C_{11}H$,$C_{10}H$), 4.56 (d, *J* = 6.3 Hz, 1H, C_2H), 4.11 (d, *J* = 13.7 Hz, 1H, $C_{10}H$), 4.07-4.04 (m, 1H, C_5H), 3.97-3.92 (m, 2H, C_6H), 3.55-3.52 (m, 1H, C_5H), 3.43-3.39 (m, 1H, C_4H), 3.19-3.16 (m, 1H, C_1H), 1.50 (s, 3H, C_7H), 1.31 (s, 3H, C_8H), 1.08 (s, 9H, $(CH_3)_3$C-Si) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 141.0(C-Ar), 135.8(C-Ar), 135.7(C-Ar), 133.7(C-Ar), 129.7(C-Ar), 128.6(C-Ar), 128.5(C-Ar), 128.4(C-Ar), 127.7(C-Ar), 127.0(C-Ar), 115.5(C-9), 82.1(C-11), 81.1(C-10), 66.6(C-3), 66.5(C-2), 65.3(C-5), 62.6(C-6), 59.3(C-1), 52.5(C-4), 27.0 ($(CH_3)_3$C-Si), 26.5(C-8), 24.8(C-7), 19.3(*C*-Si) ppm HRMS (ESI-TOF) *m/z* $[M+H]^+$ calcd for $C_{39}H_{48}NO_4Si$ 622.3353; found 622.3354.



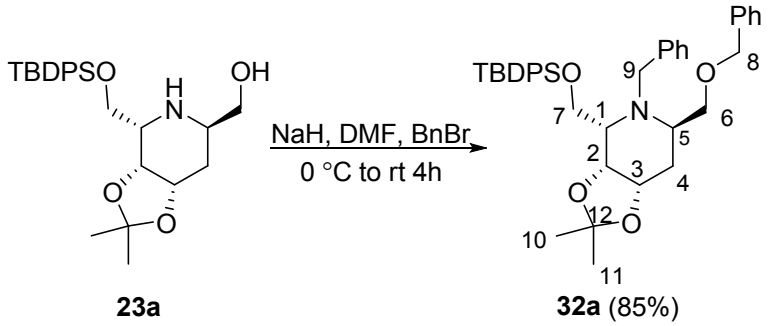
18a **31a** (85%)

*Compound***(31a)**: In a similar manner as described for compound **30a**, Column chromatography (petroleum ether/EtOAc, 9:1); oily liquid (0.17 g, 0.39mmol, 85%);$[\alpha]_D^{25}$ = -9.3 (*c* 0.2, $CHCl_3$); IR ν_{max} (thin film): 3028, 1495 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.32-7.23 (m, 10H, Ar-H), 4.64 (d,*J* = 6.7 Hz, 1H, -O-CH_2-O-), 4.61 (d,*J*= 7.3Hz, 1H, -O-CH_2-O-), 4.56 (d, *J* = 11.6 Hz, 1H, $C_{10}H$), 4.48-4.44 (m, 2H, C_6H, $C_{10}H$), 4.16-4.11 (m, 2H, C_6H, C_9H), 3.88-3.84 (m, 1H, C_2H), 3.81 (dd,*J* = 10.7, 2.1 Hz, 1H, C_3H), 3.59 (dd,*J* = 11.0, 4.2 Hz, 1H, C_4H), 3.30 (s, 3H, -OCH_3), 3.26 (d,*J* = 13.6Hz, 1H, C_9H), 2.77 (dd,*J* = 11.0, 5.5 Hz, 1H, C_1H), 2.51-2.48 (m, 1H, C_5H), 2.36-2.33 (t, *J* = 11.0 Hz, 1H, C_1H), 1.54 (s, 3H, C_7H), 1.39 (s, 3H, C_8H) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 139.0(C-Ar), 138.1(C-Ar), 128.9(C-Ar), 128.4(C-Ar), 128.3(C-Ar), 127.9(C-Ar), 127.7(C-Ar), 127.0(C-Ar), 109.4(C-11), 95.9(-O-CH$_2$-O-), 74.9(C-10), 74.5(C-9), 73.4(C-2), 71.7(C-3), 68.7(C-4), 63.6(C-6), 57.1(-OCH$_3$), 55.5(C-5), 50.5(C-1), 28.2(C-8), 26.3(C-7) ppm; HRMS (ESI-TOF) *m/z* $[M+H]^+$ calcd for $C_{25}H_{34}NO_5$ 428.2437; found 428.2431.



18b **31b** (69%)

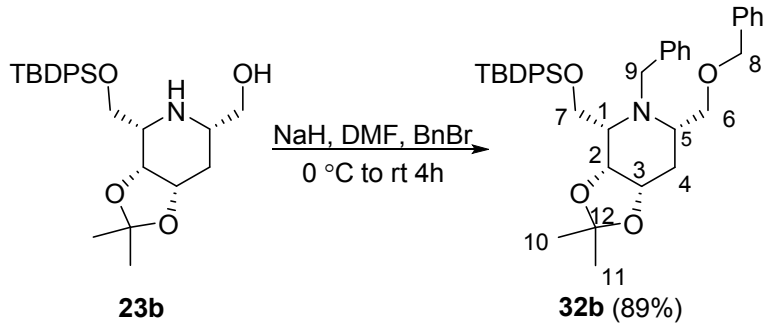
Compound(**31b**):Column chromatography (petroleum ether/EtOAc, 1:1); oily liquid (0.11 g, 0.32mmol, 69%);$[\alpha]_D^{25}$ = +21.2 (*c* 0.8, $CHCl_3$); IRν_{max} (thin film): 3018, 1495 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.30-7.32 (m, 4H, Ar-H), 7.28-7.26 (m,1H, Ar-H), 4.74 (d, *J* = 6.8 Hz, 1H, -O-CH_2-O-), 4.66 (d, *J* = 6.8 Hz, 1H, -O-CH_2-O-), 4.57 (d, *J* = 12.0 Hz, 1H, C_9H), 4.51 (d, *J* = 12.0 Hz, 1H, C_9H), 4.35 (dd,*J* = 6.8, 4.0 Hz, 1H, C_3H), 4.24 (dd, *J* = 7.4, 2.8 Hz, 1H, C_4H), 3.82-3.80 (m, 1H, C_2H), 3.55-3.49 (m, 2H, C_6H), 3.37 (s, 3H–OCH_3), 3.09 (dd, *J* = 13.1, 6.8 Hz, 1H, C_1H), 3.03 (dt, *J* = 6.8, 2.3 Hz, 1H, C_5H), 2.88 (dd, *J* = 13.1, 4.6 Hz, 1H, C_1H), 2.1 (bs, 1H, $N_{10}H$)1.42 (s, 3H, C_7H), 1.38 (s, 3H, C_8H)ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 138.2(C-Ar), 128.4(C-Ar), 127.8(C-Ar), 127.7(C-Ar), 109.4(C-11), 96.1(-O-CH$_2$-O-), 73.8(C-9), 73.4(C-2), 73.0(C-3), 70.0(C-4), 69.8(C-6), 55.6(-OCH_3), 54.1(C-5), 44.9(C-1), 26.0(C-8), 25.0(C-7) ppm; HRMS (ESI-TOF) *m/z* $[M+H]^+$ calcd for $C_{18}H_{28}NO_5$ 338.1967; found 338.1969.



23a **32a** (85%)

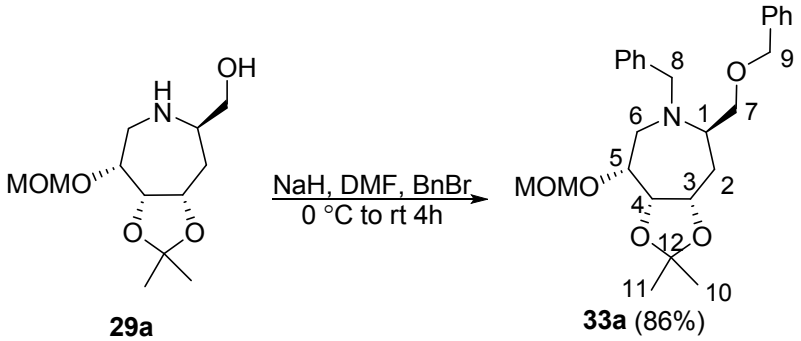
Compound(**32a**):Column chromatography (petroleum ether/EtOAc, 9:1); oily liquid (0.15 g, 0.23mmol 85%); $[\alpha]_D^{25}$ = -30.64 (*c* 0.12, $CHCl_3$); IR ν_{max} (thin film): 3060, 1536 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.68-7.64 (m, 4H, Ar-H), 7.42-7.14 (m, 16H, Ar-H), 4.46-4.35 (m, 3H, C_8H, C_3H), 4.31 (d, *J* = 2.8 Hz, 2H, C_2H), 4.02-3.97 (m, 2H, C_9H), 3.90 (dd, *J* = 5.5, 9.5 Hz, 1H, C_5H), 3.79 (d, *J* = 14.9 Hz, 1H, C_9H), 3.29-3.23 (m, 3HC_1H, C_6H), 1.97-1.91 (m, 1H, C_4H), 1.81-1.75 (m, 1H, C_4H), 1.55 (s, 3H, $C_{10}H$), 1.35 (s, 3H, $C_{11}H$), 1.05 (s, 9H, $(CH_3)_3$C-Si) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 141.9(C-Ar), 138.6(C-Ar), 135.7(C-Ar), 135.6(C-Ar), 134.9(C-Ar), 133.7(C-Ar), 129.7(C-Ar), 129.6(C-Ar), 128.7(C-Ar), 128.6(C-Ar), 128.5(C-Ar), 128.4(C-Ar), 128.2(C-Ar), 128.0(C-Ar), 127.7(C-Ar), 127.6(C-Ar), 127.5(C-Ar), 126.5(C-Ar), 108.0(C-12), 73.0(C-8), 73.00(C-6), 72.8(C-9), 71.6(C-7), 63.3(C-2), 58.0(C-3), 55.9(C-1),

55.2(C-5), 27.5(C-4), 27.5(C-10), 26.9(CH_3)$_3$C-Si, 24.8(C-11), 19.3(*C*-Si) ppm; HRMS (ESI-TOF) *m/z* $[M+H]^+$ calcd for $C_{40}H_{50}NO_4Si$ 636.3509; found 636.3509.



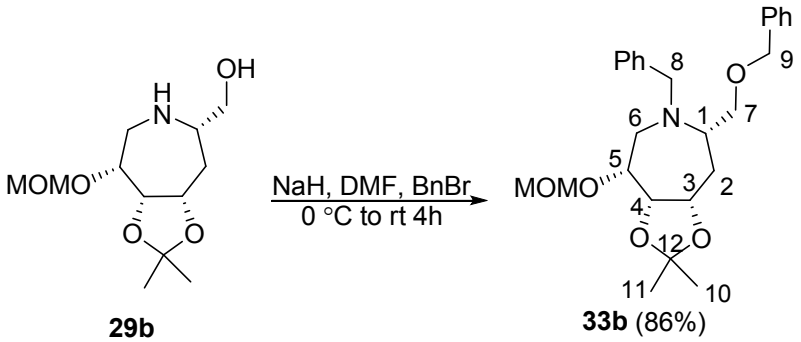
23b **32b** (89%)

Compound(**32b**):Column chromatography (petroleum ether/EtOAc, 9:1); oily liquid (0.2 g, 0.31mmol 89%); $[\alpha]_D^{25}$ = -19.1 (*c* 0.8, $CHCl_3$); IR ν_{max} (thin film):3059, 1589 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.62-7.54 (m, 4HAr-H), 7.40-7.12 (m, 16H, Ar-H), 4.38-4.35 (m, 1H, C_2H), 4.33-4.34 (m, 2H, C_8H, C_3H), 4.28-4.20 (m, 1H, C_8H), 3.90-3.84 (m, 2H, C_6H), 3.77-3.71 (m, 2H, C_9H), 3.44-3.40 (m, 1H, C_7H), 3.32-3.28 (m, 1H, C_7H), 3.17-3.12 (m, 1H, C_1H) 2.94-2.88 (m, 1H, C_5H), 1.92-1.86 (m, 1H, C_4H), 1.76-1.68 (m, 1H, C_4H), 1.50 (s, 3H, $C_{11}H$), 1.33 (s, 3H, $C_{10}H$), 0.99 (s, 9H, (CH_3)$_3$C-Si)) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.9(C-Ar), 138.3(C-Ar), 135.69(C-Ar), 135.61(C-Ar), 133.7(C-Ar), 133.6(C-Ar), 129.5(C-Ar), 128.3(C-Ar), 128.0(C-Ar), 127.8(C-Ar), 127.6(C-Ar), 127.5(C-Ar), 126.3(C-Ar), 108.2(C-12), 73.2(C-8), 73.1(C-3), 72.9(C-2), 72.4(C-6), 64.0(C-7), 62.6(C-9), 58.6(C-1), 53.0(C-5), 28.4(C-4), 28.1(C-10), 26.8(CH_3)$_3$C-Si, 25.6(C-11), 19.2(*C*-Si) ppm HRMS (ESI-TOF) *m/z*$[(M+H)-(HCl)]^+$ calcd for $C_{40}H_{50}NO_4Si$ 636.3509; found 636.3509.



29a **33a** (86%)

Compound(**33a**):Column chromatography (petether/Ethyl acetate 8:2); oily liquid (0.3 g, 0.67 mmol 86%); $[\alpha]_D^{25}$ = +18.6 (*c* 0.22, $CHCl_3$); IR ν_{max} (thin film): 2947, 1436 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.38-7.21 (m, 10 H, Ar-H), 4.56-4.41 (m, 4H, -O-*CH*$_2$-O-, C_4H, C_9H), 4.49-4.46 (m, 2H, C_9H, C_3H), 4.00 (d, *J* = 9.7 Hz, 1H, C_5H), 3.78 (d, *J* = 14.3 Hz, 1H, C_8H), 3.60 (d, *J* = 14.3 Hz, 1H, C_8H), 3.56 (dd, *J* = 9.7, 7.1 Hz, 1H, C_7H), 3.47 (dd, *J* = 9.7, 5.7 Hz, 1H, C_7H), 3.44-3.40 (m, 1H, C_1H), 3.29 (s, 3H, -O*CH*$_3$), 3.20 (dd, *J* = 14.3, 10.0 Hz, 1H, C_6H), 2.65 (d, *J* = 14.9 Hz, 1H, C_6H), 1.90-1.83 (m, 2H, C_2H), 1.54 (s, 3H, $C_{11}H$), 1.38 (s, 3H, $C_{10}H$) ppm; ^{13}C NMR (125 MHz, $CDCl_3$): δ 140.0 (C-Ar), 138.3 (C-Ar), 128.8 (C-Ar), 128.4 (C-Ar), 128.3 (C-Ar), 127.9 (C-Ar), 127.6 (C-Ar), 127.0 (C-Ar), 107.7 (C-12), 94.9 (-O-*C*H_2-O-), 79.2 (C-9), 73.4 (C-4), 73.1 (C-3), 72.7 (C-5), 69.8 (C-8), 56.0 (C-7), 55.6 (-O*C*H_3), 50.6 (C-1), 50.1 (C-6), 36.2 (C-2), 29.7 (C-10), 25.9 (C-11) ppm; HRMS (ESI-TOF) *m/z* $[M+H]^+$ calcd for $C_{26}H_{36}NO_5$ 442.2593; found 442.2591.



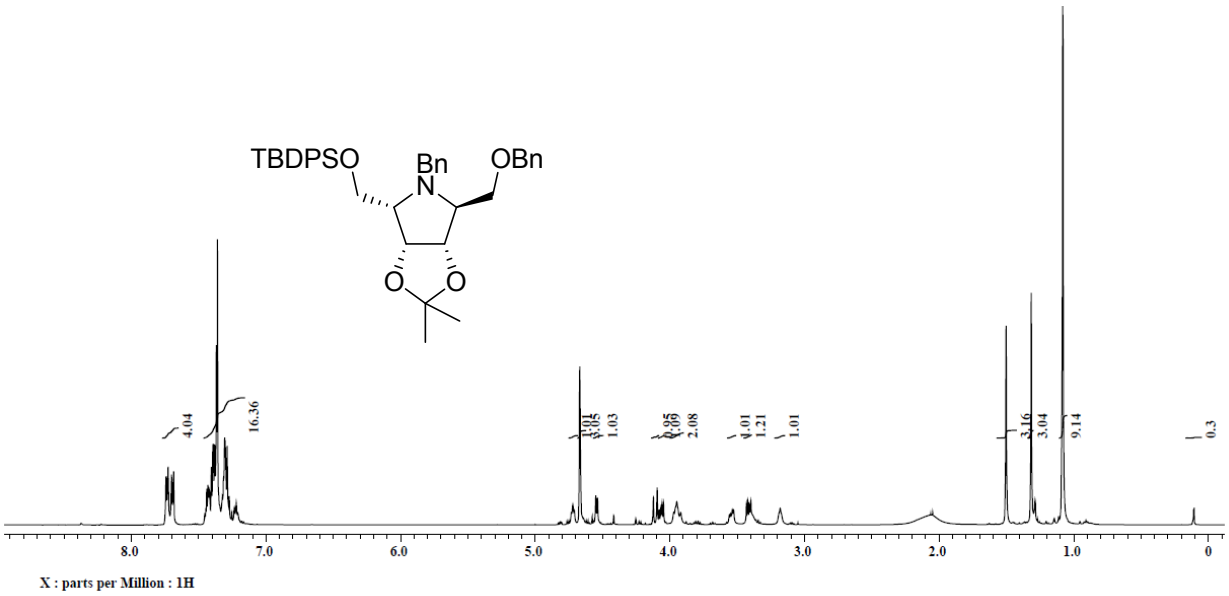
29b **33b** (86%)

Compound(**33b**):Column chromatography (petether/Ethyl acetate 8:2); oily liquid (0.25 g, 0.56 mmol, 87%); $[\alpha]_D^{25}$ = +13.3 (*c* 1.6, $CHCl_3$); IR ν_{max} (thin film): 2947, 1436 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.36-7.22 (m, 10 H, Ar-H), 4.62 (d, *J* = 8.6 Hz, 1H, -O-*CH*$_2$-O-), 4.55-4.39 (m, 5H, -O-*CH*$_2$-O-, C_4H, C_9H, C_3H), 3.93-3.90 (m, 2H, C_5H, C_8H), 3.79 (dd, *J* = 12.6, 8.6 Hz, 1H, C_7H), 3.74 (d, *J* = 17.7 Hz, 1H, C_8H), 3.47 (dd, *J* = 12.0, 6.3 Hz, 1H, C_7H), 3.28 (s, 3H, -O*CH*$_3$), 3.19-3.14 (m, 2H, C_6H, C_1H), 2.70 (dd, *J* = 18.9, 6.9 Hz, 1H, C_6H), 2.37-2.28 (m, 1H, C_2H), 2.10-2.06 (m, 1H, C_2H), 1.47 (s, 3H, $C_{11}H$), 1.36 (s, 3H, $C_{10}H$) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.3 (C-Ar), 138.5 (C-Ar), 128.5 (C-Ar), 128.4 (C-Ar), 128.3 (C-Ar), 127.6 (C-Ar), 127.0 (C-Ar), 107.4 (C-12), 96.3 (-O-*C*H_2-O-), 78.3 (C-9), 75.5 (C-4), 73.9

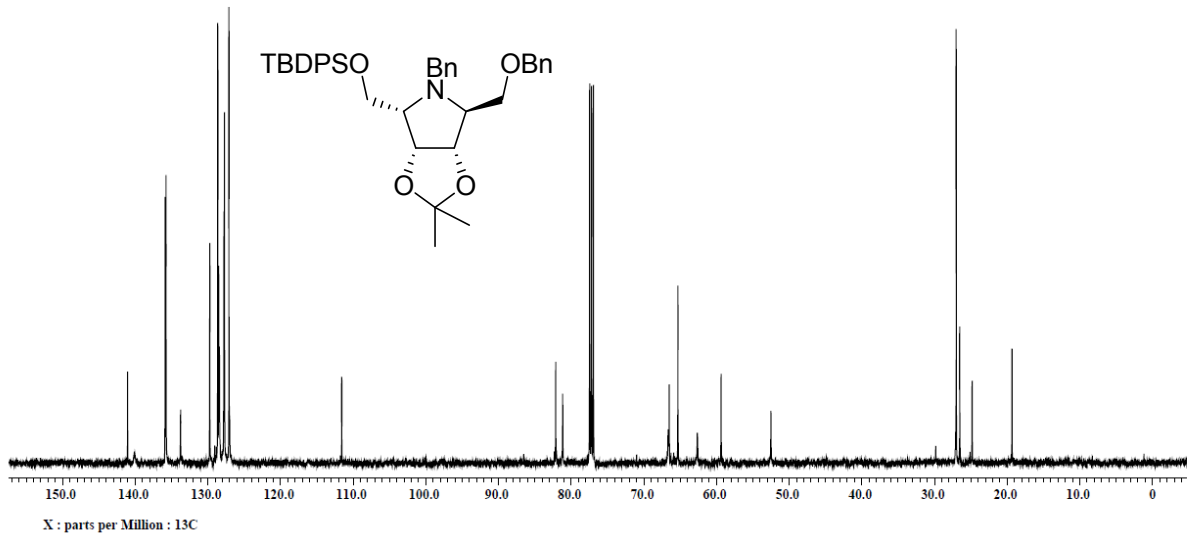
(C-3), 73.3 (C-5), 70.4 (C-8), 59.4 (C-7), 58.2 (-O*C*H_3), 55.4 (C-1), 48.3 (C-6), 32.4 (C-2), 26.8 (C-10), 24.3 (C-11) ppm; HRMS (ESI-TOF) *m/z* $[M+H]^+$ calcd for $C_{26}H_{36}NO_5$ 442.2593; found 442.2596.

NMR Correlation data for benzyl derivatives

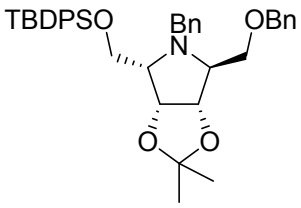
Compound 30

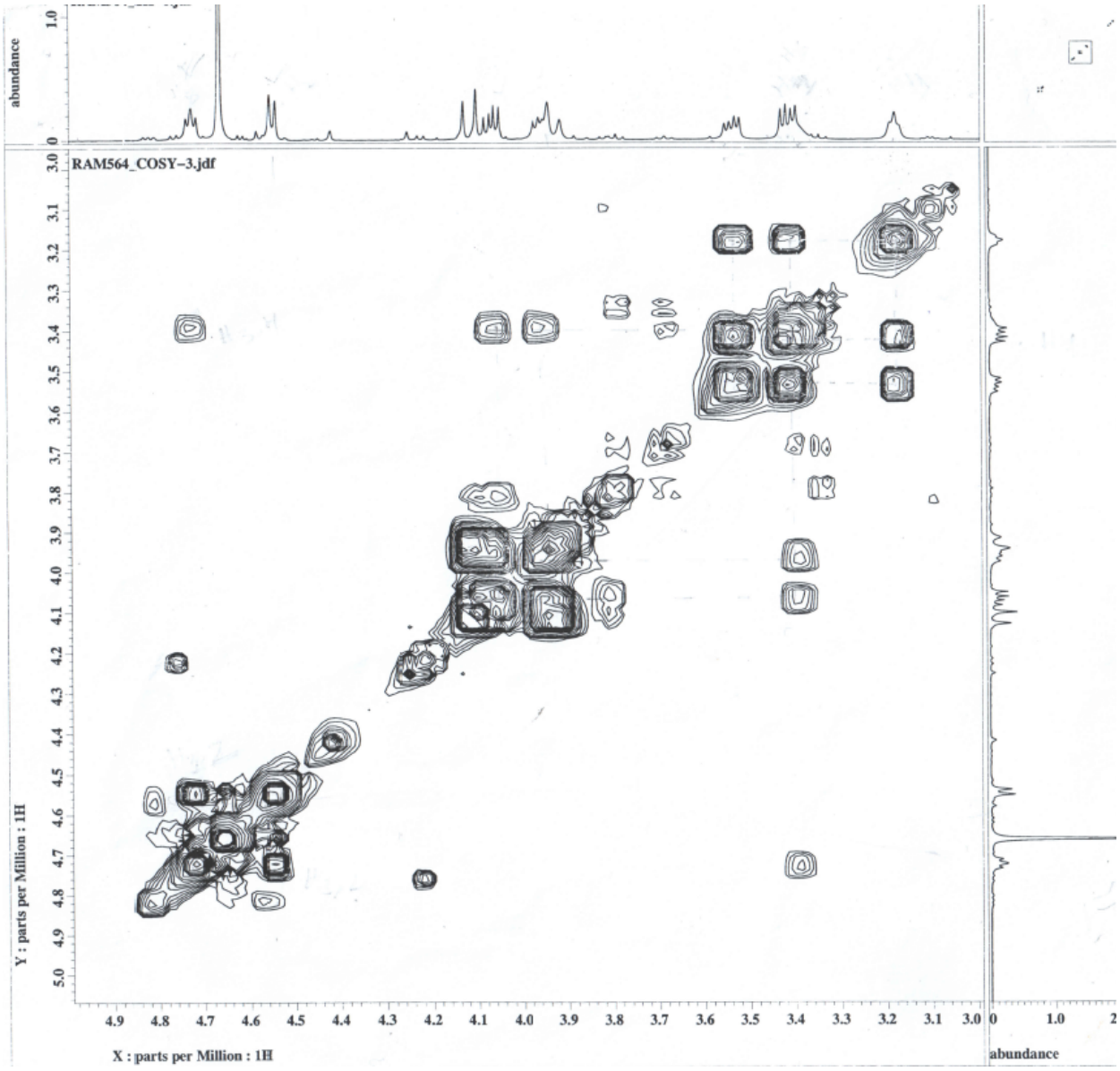


^{1}H NMR (500 MHz, $CDCl_3$)

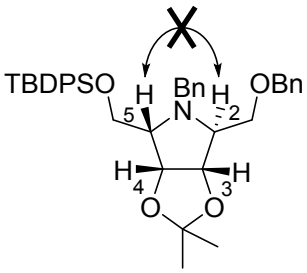


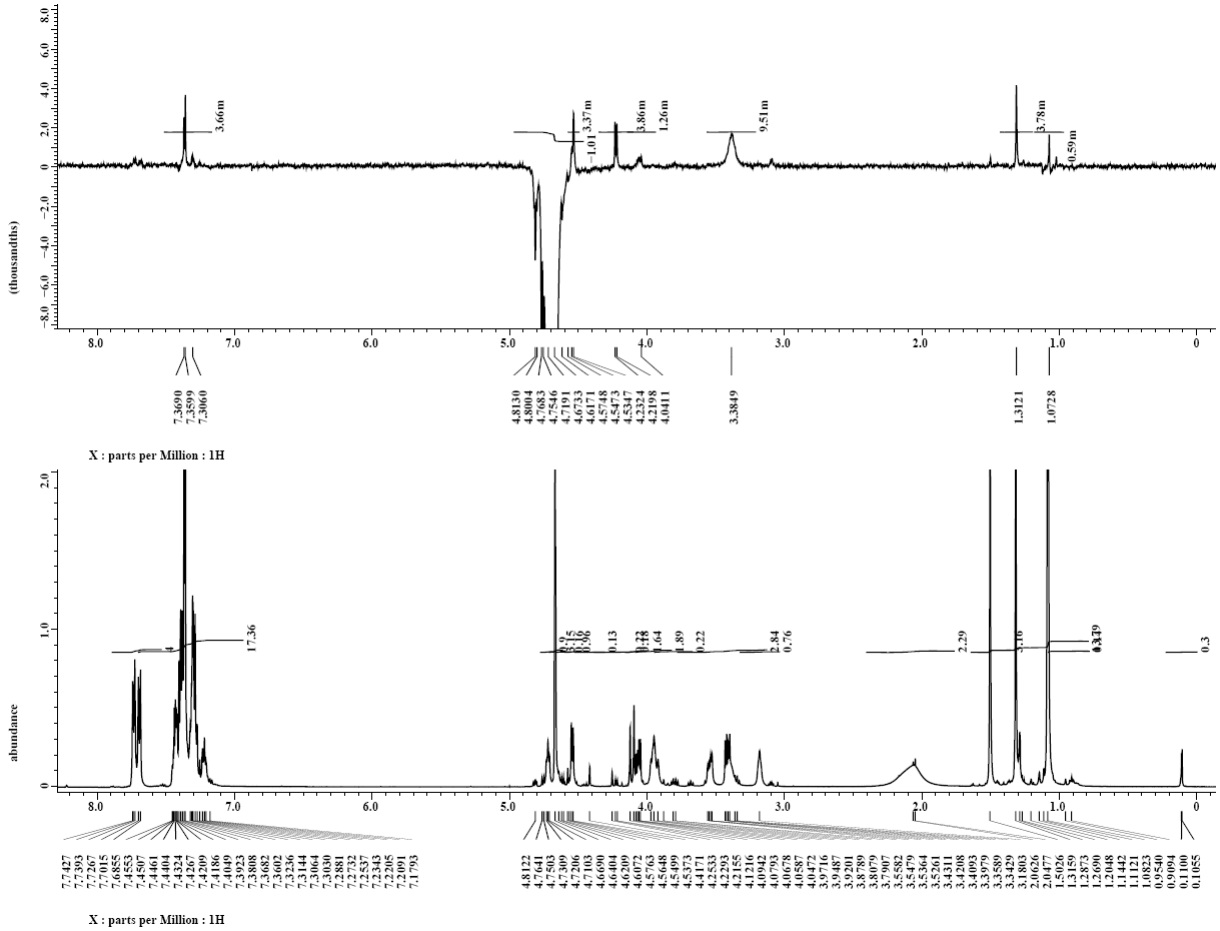
^{13}C NMR (125 MHz, $CDCl_3$)





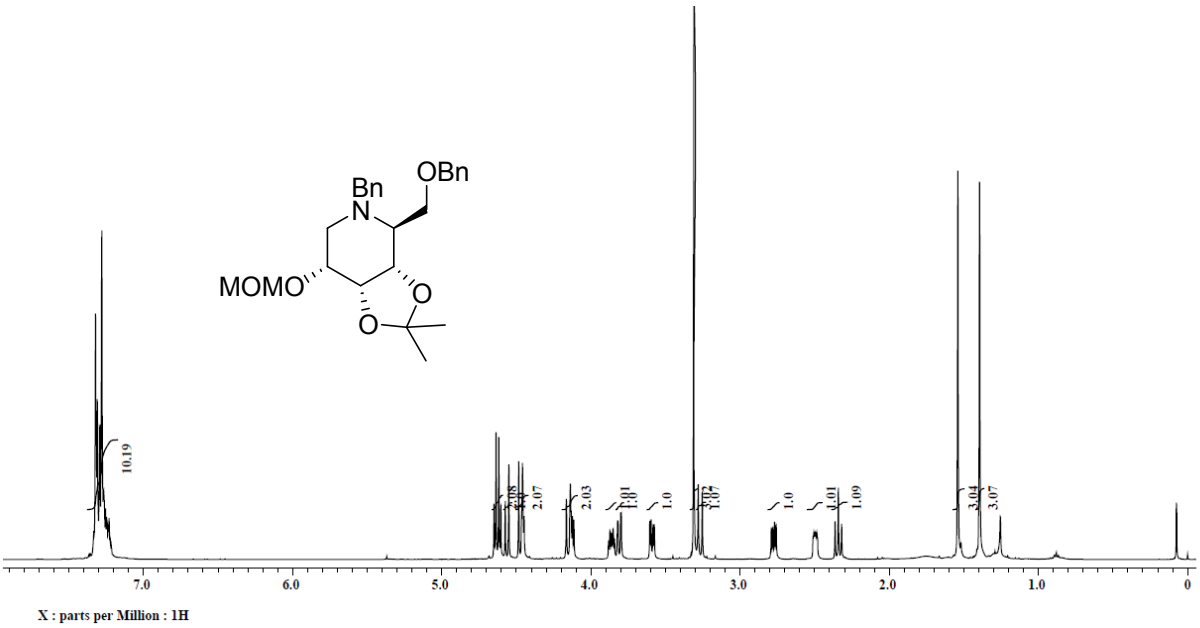
^{1}H-^{1}H COSY (500 MHz)



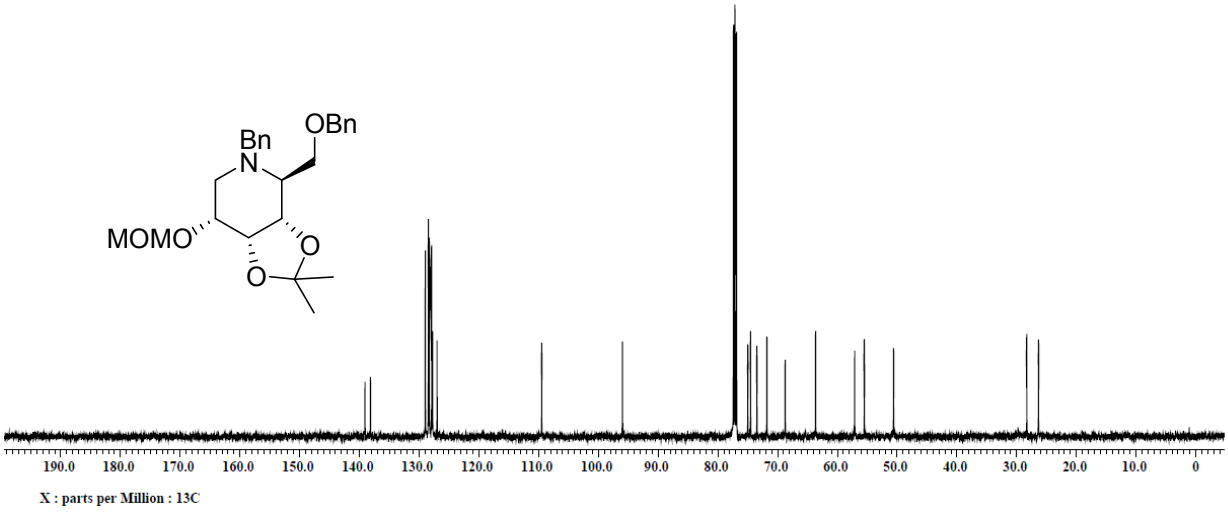


nOe spectrum (irradiation of H-4)

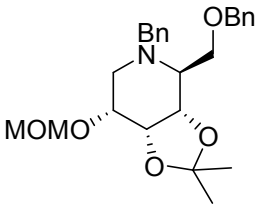
Compound 31a

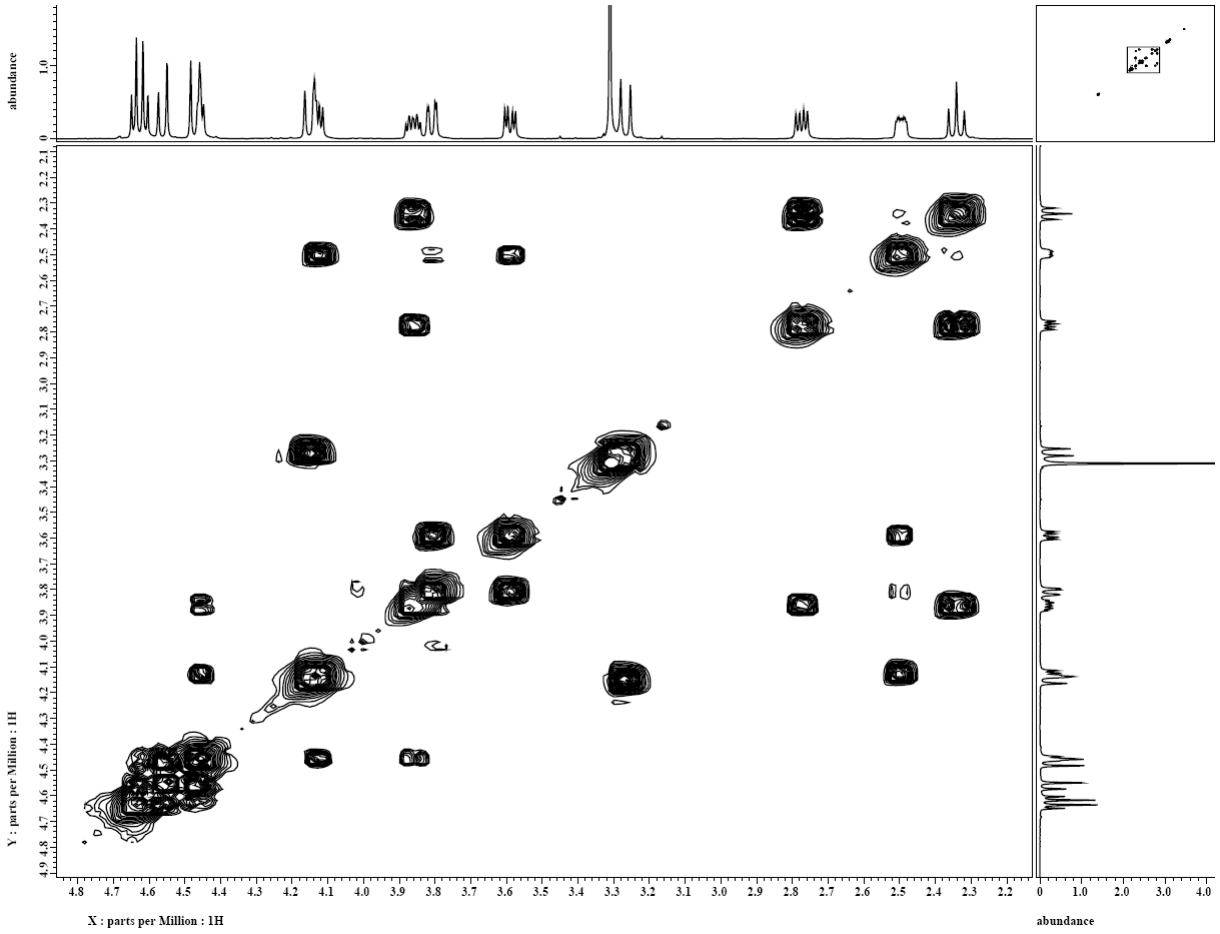


^{1}H NMR (500 MHz, $CDCl_3$)

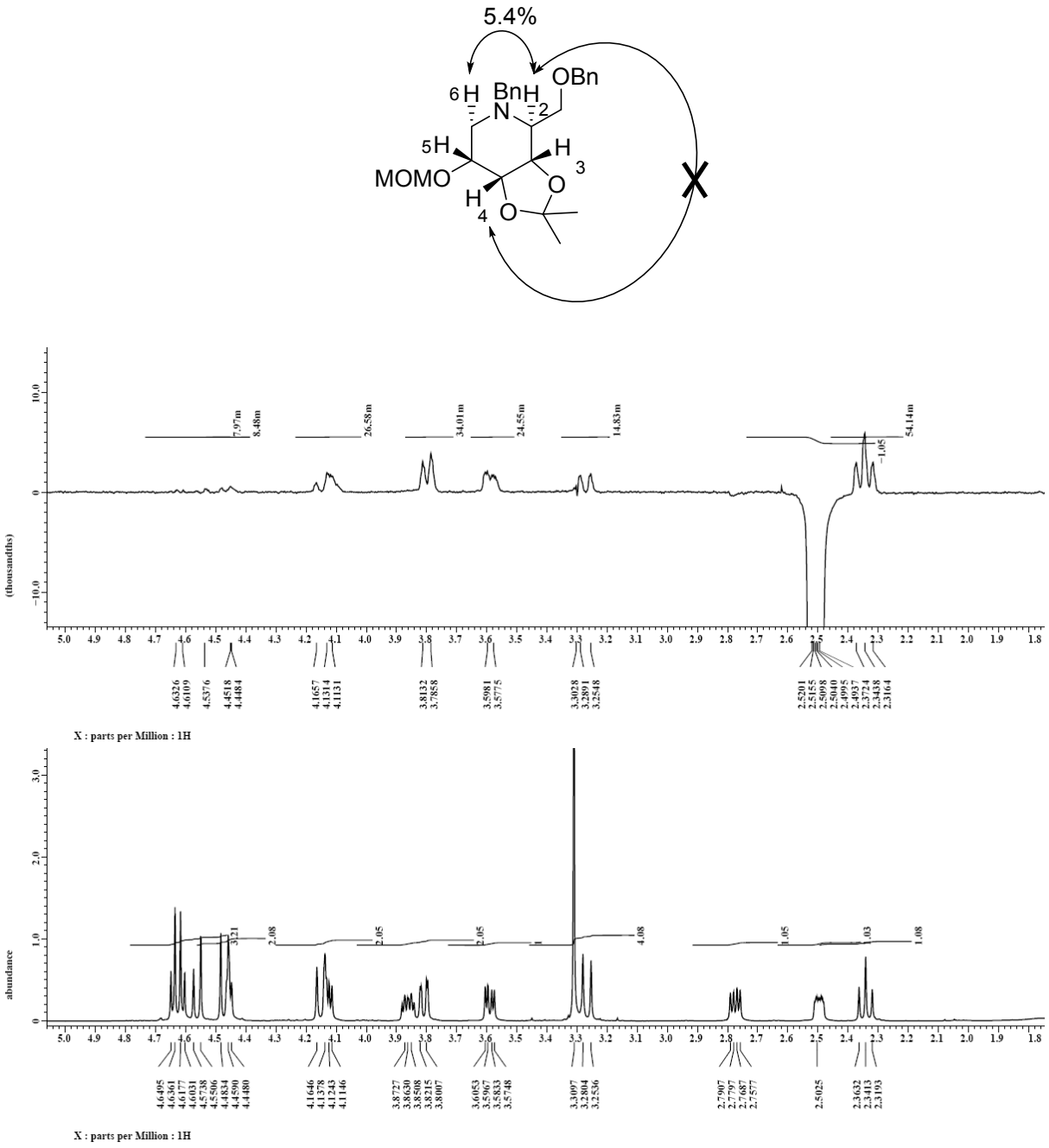


^{13}C NMR (125 MHz, $CDCl_3$)



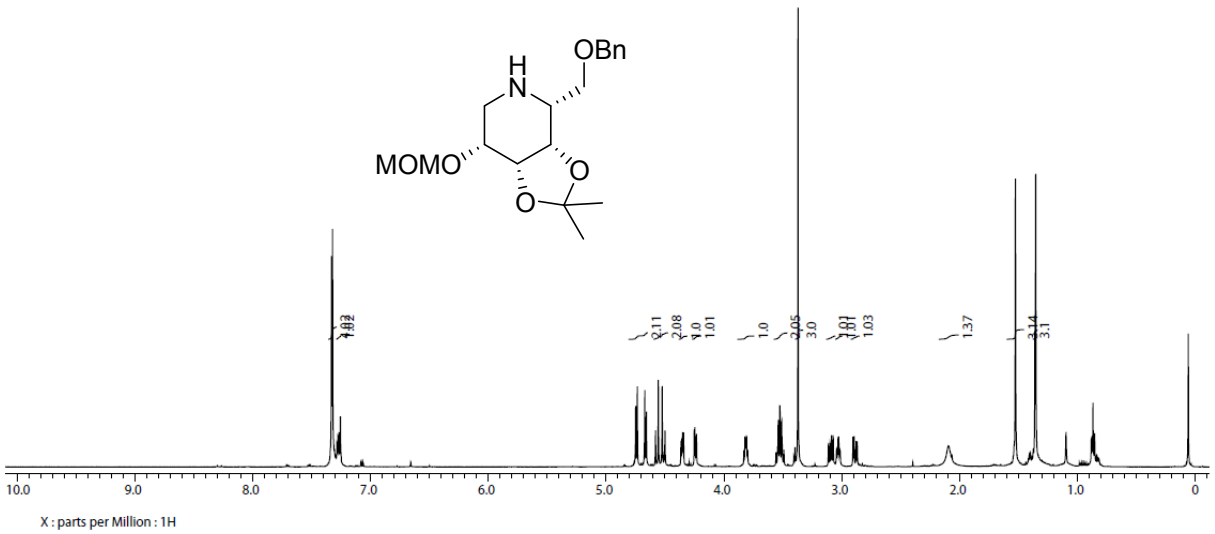


^{1}H-^{1}H COSY (500 MHz)

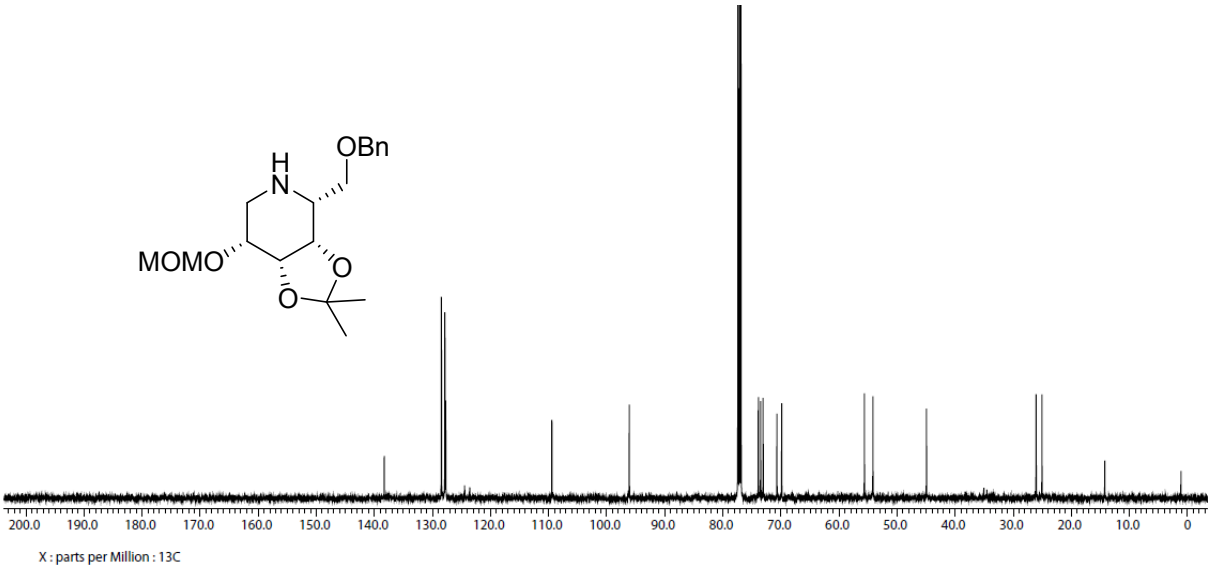


nOe spectrum (irradiation of H-2)

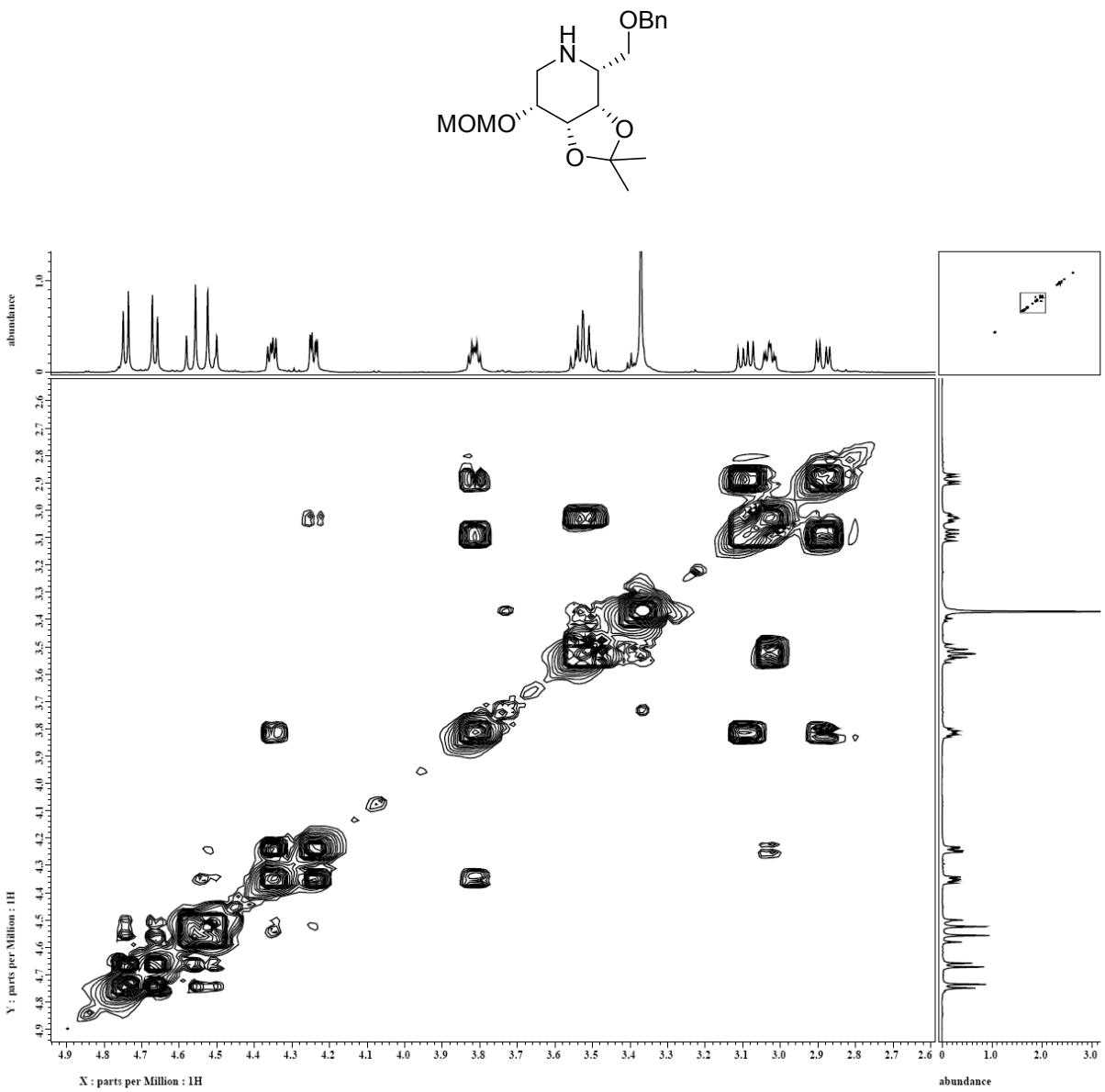
Compound 31b



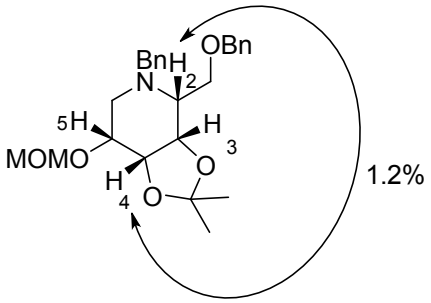
^{1}H NMR (500 MHz, $CDCl_3$) spectrum of

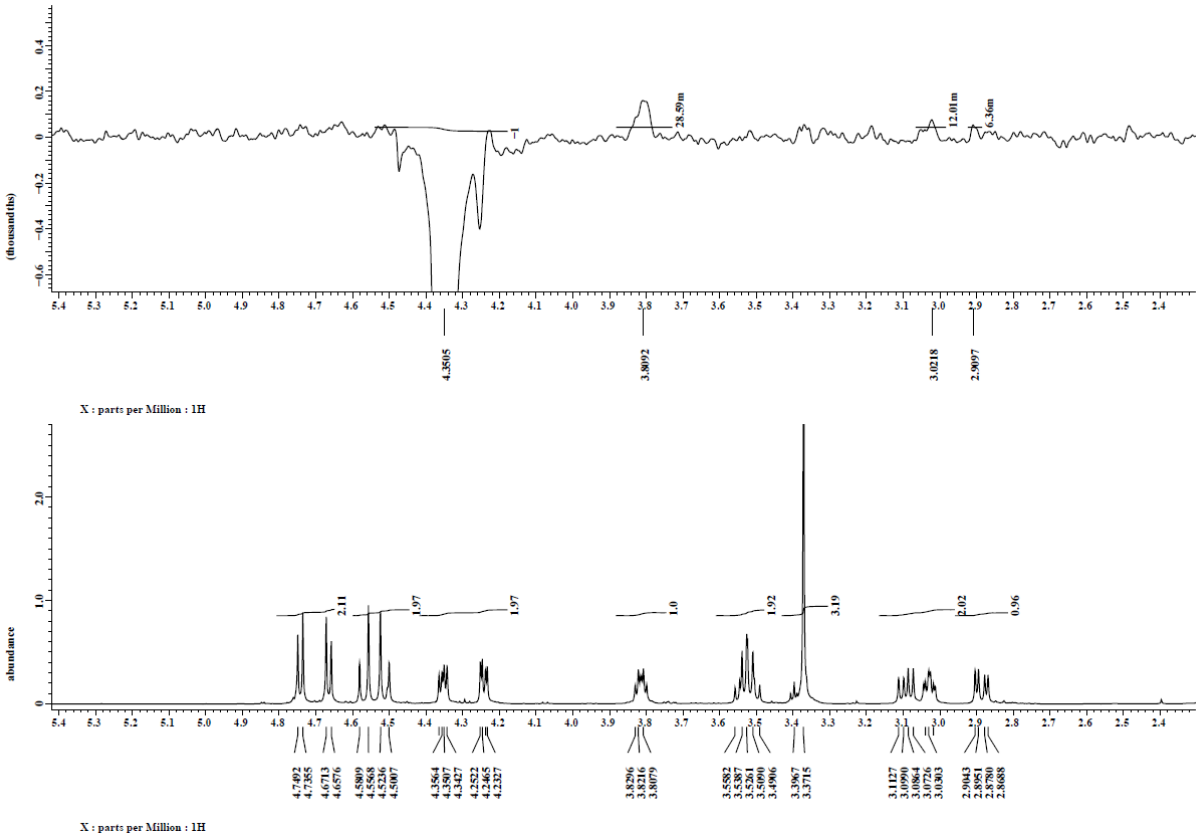


^{13}C NMR (125 MHz, $CDCl_3$)



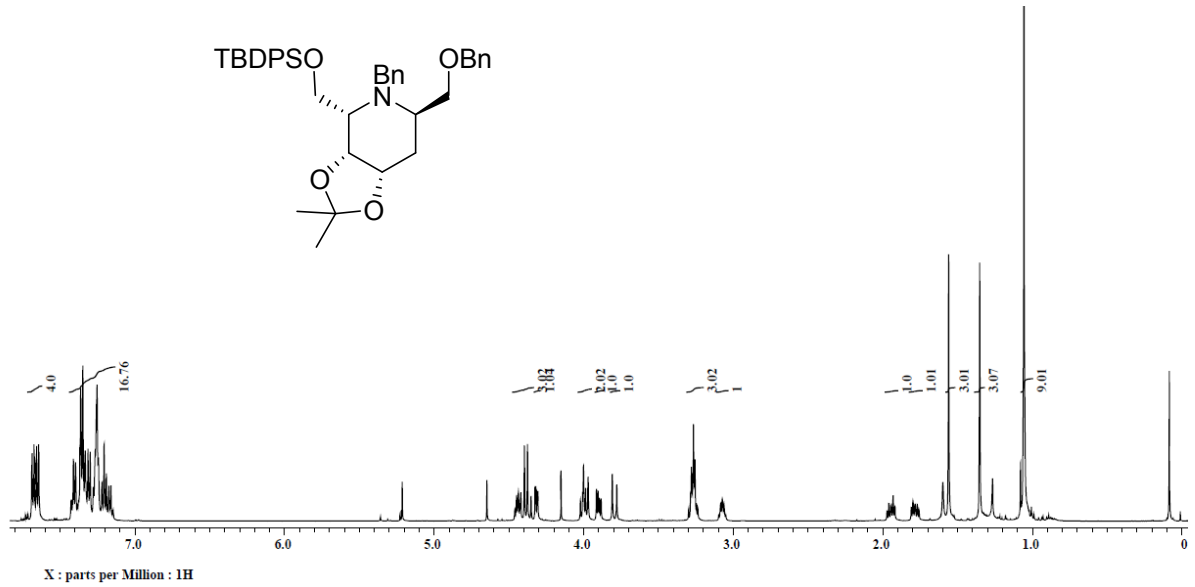
^{1}H-^{1}H COSY (500 MHz)



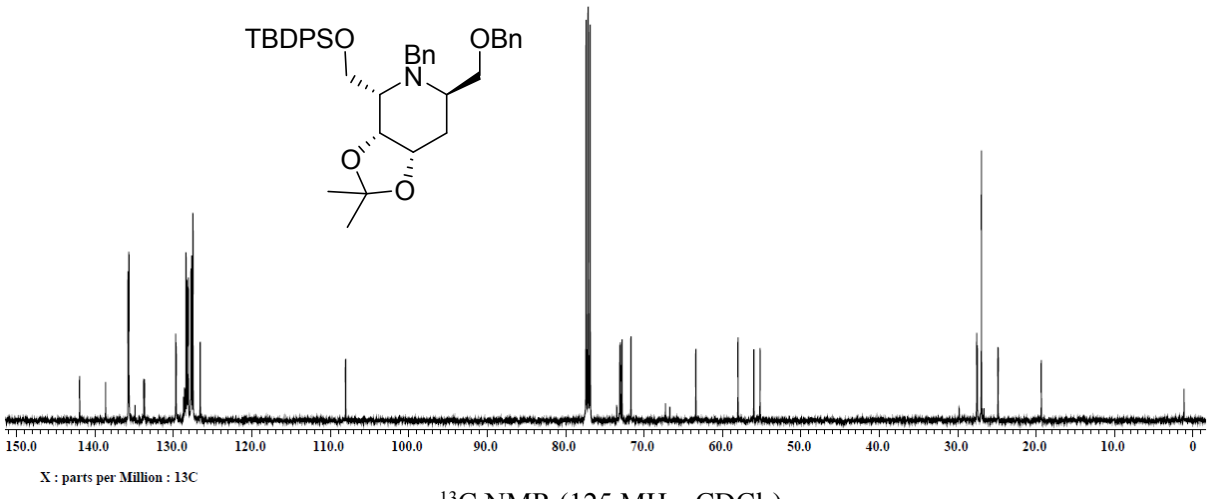


nOe spectrum (irradiation of H-4)

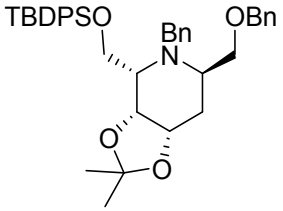
Compound32a



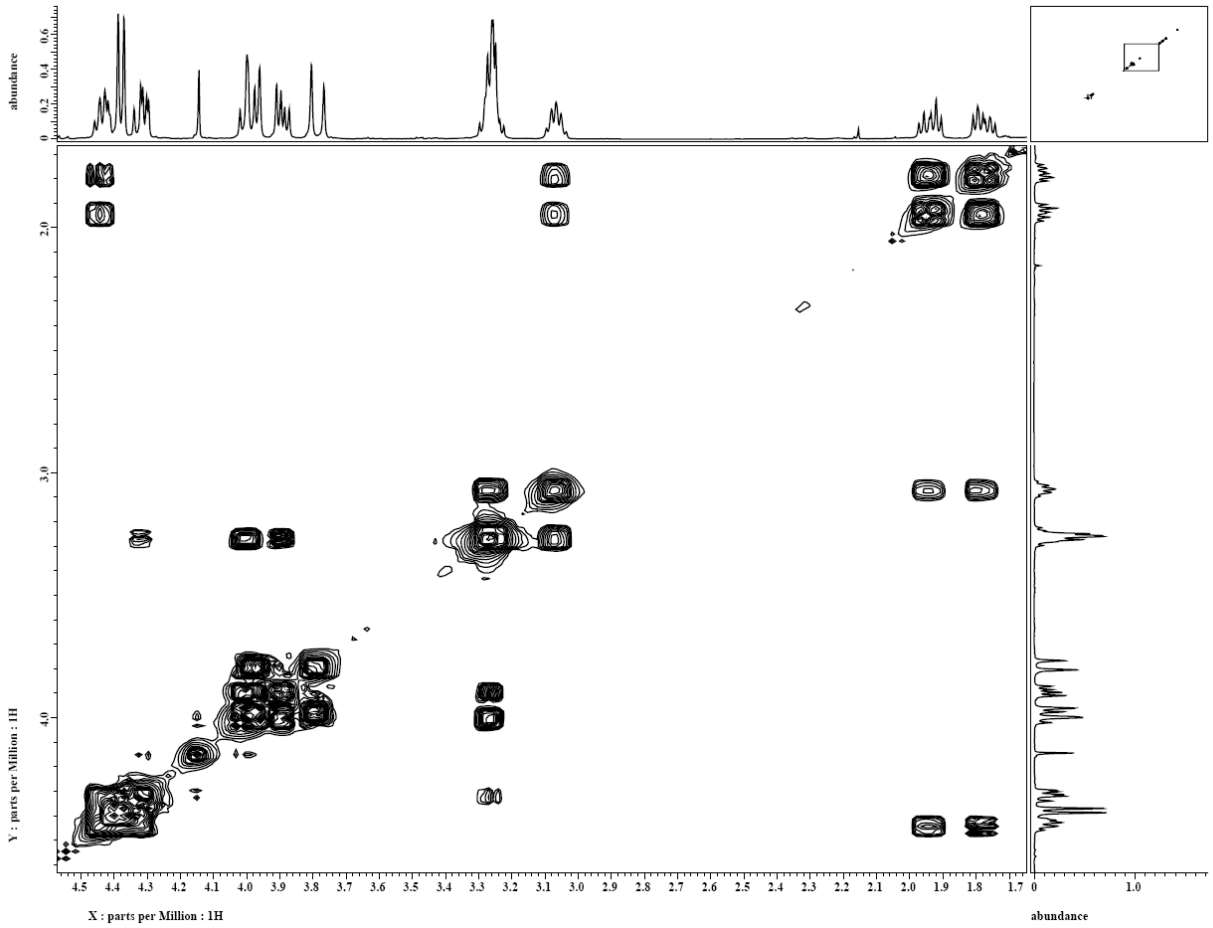
^{1}H NMR (500 MHz, $CDCl_3$)



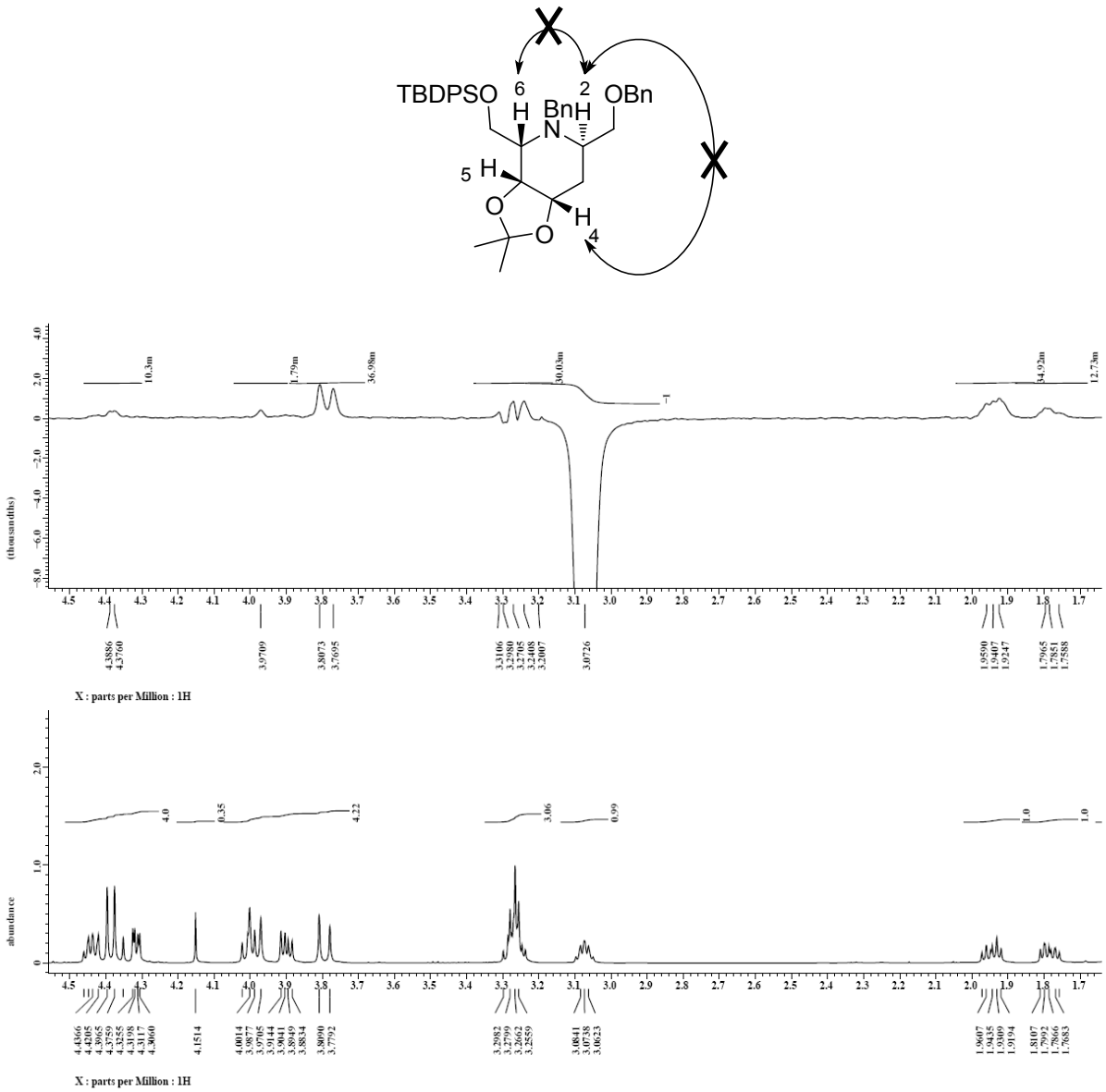
^{13}C NMR (125 MHz, $CDCl_3$)



-1.-_

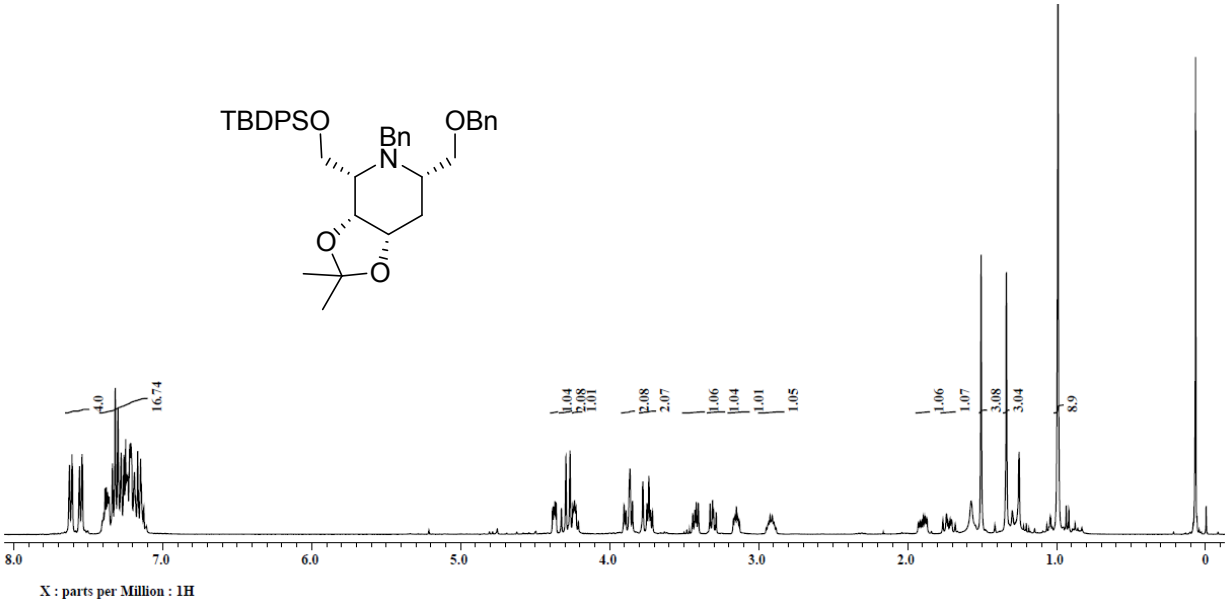


^{1}H-^{1}H COSY (500 MHz)

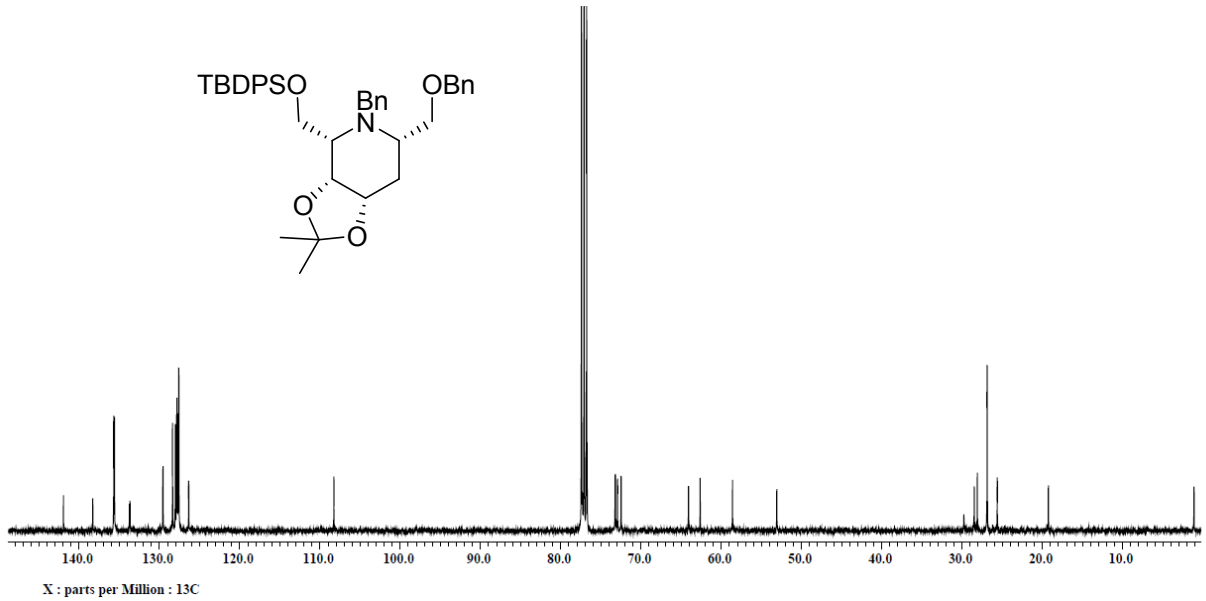


nOe spectrum (irradiation of H-2)

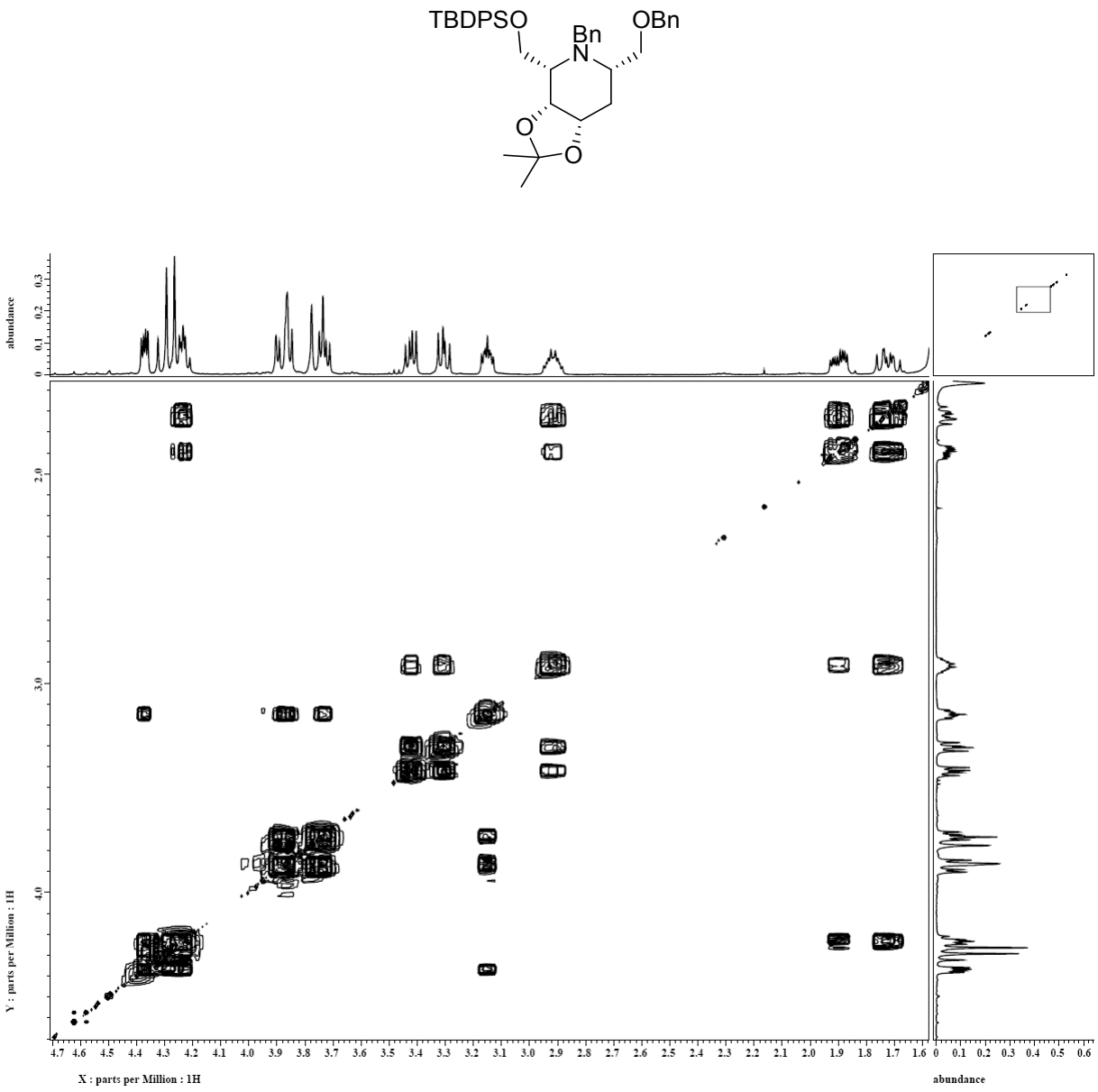
Compound 32b



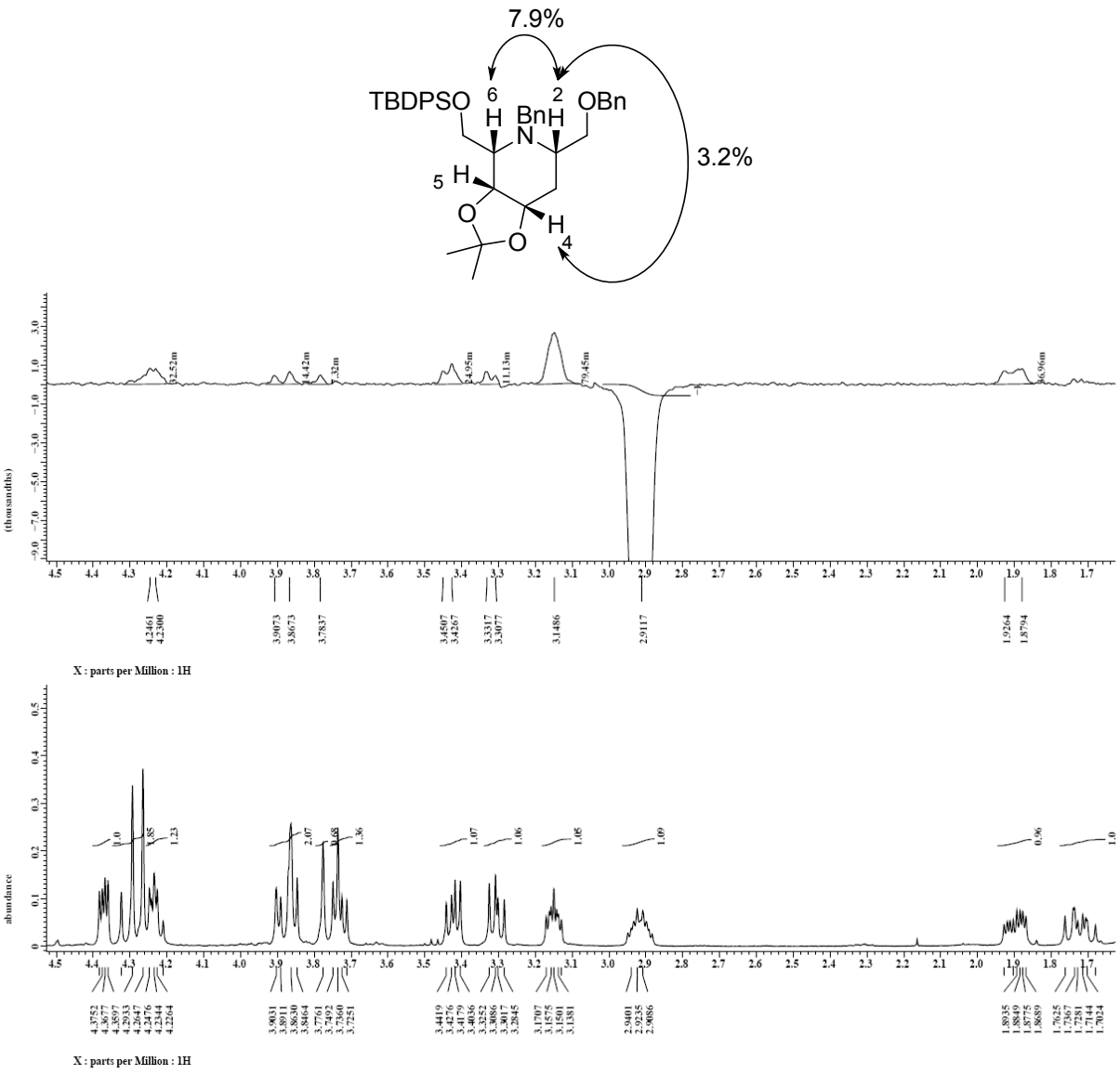
^{1}H NMR (500 MHz, $CDCl_3$)



^{13}C NMR (125 MHz, $CDCl_3$)

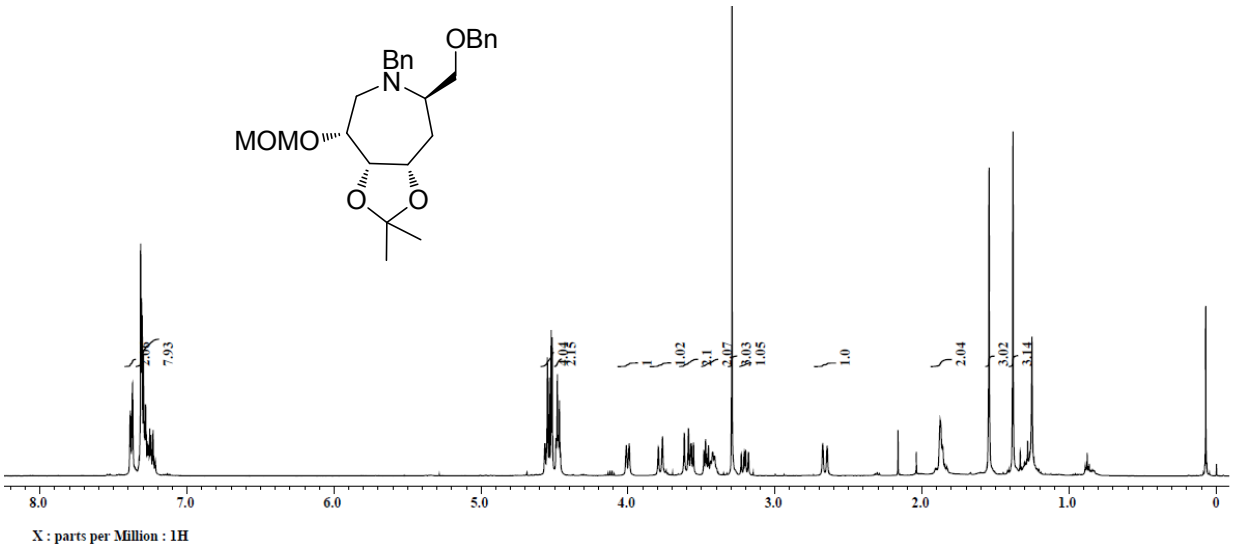


^{1}H-^{1}H COSY (500 MHz)

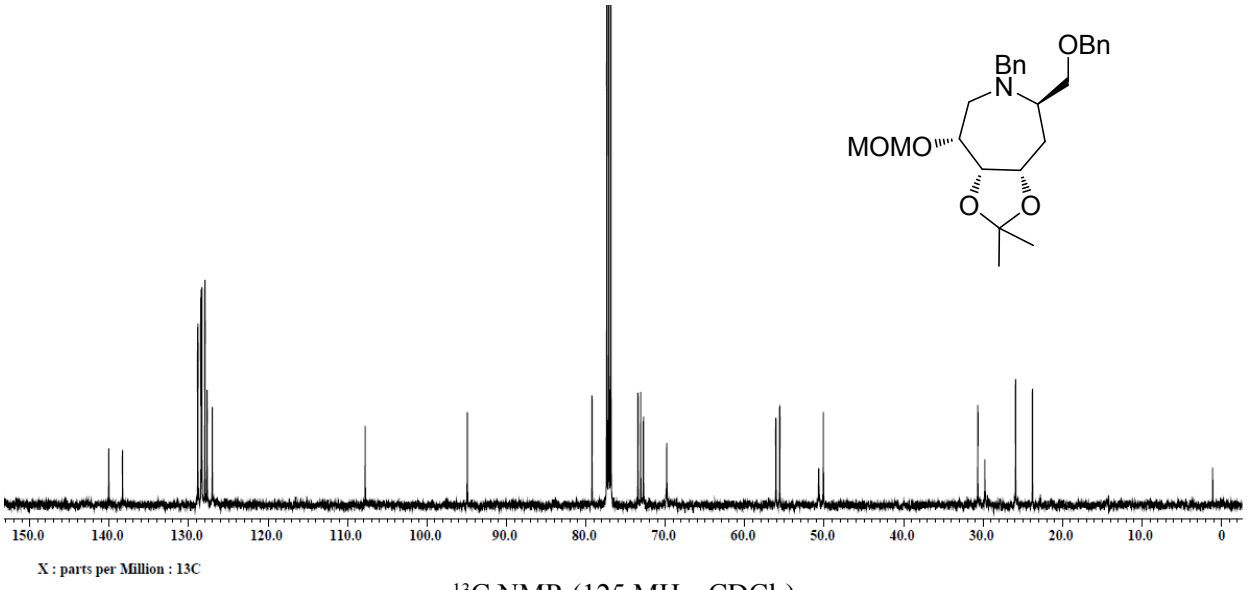


nOe spectrum (irradiation of H-2)

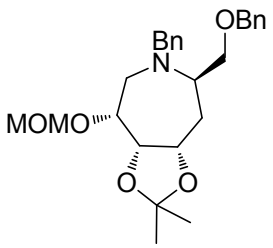
Compound 33a

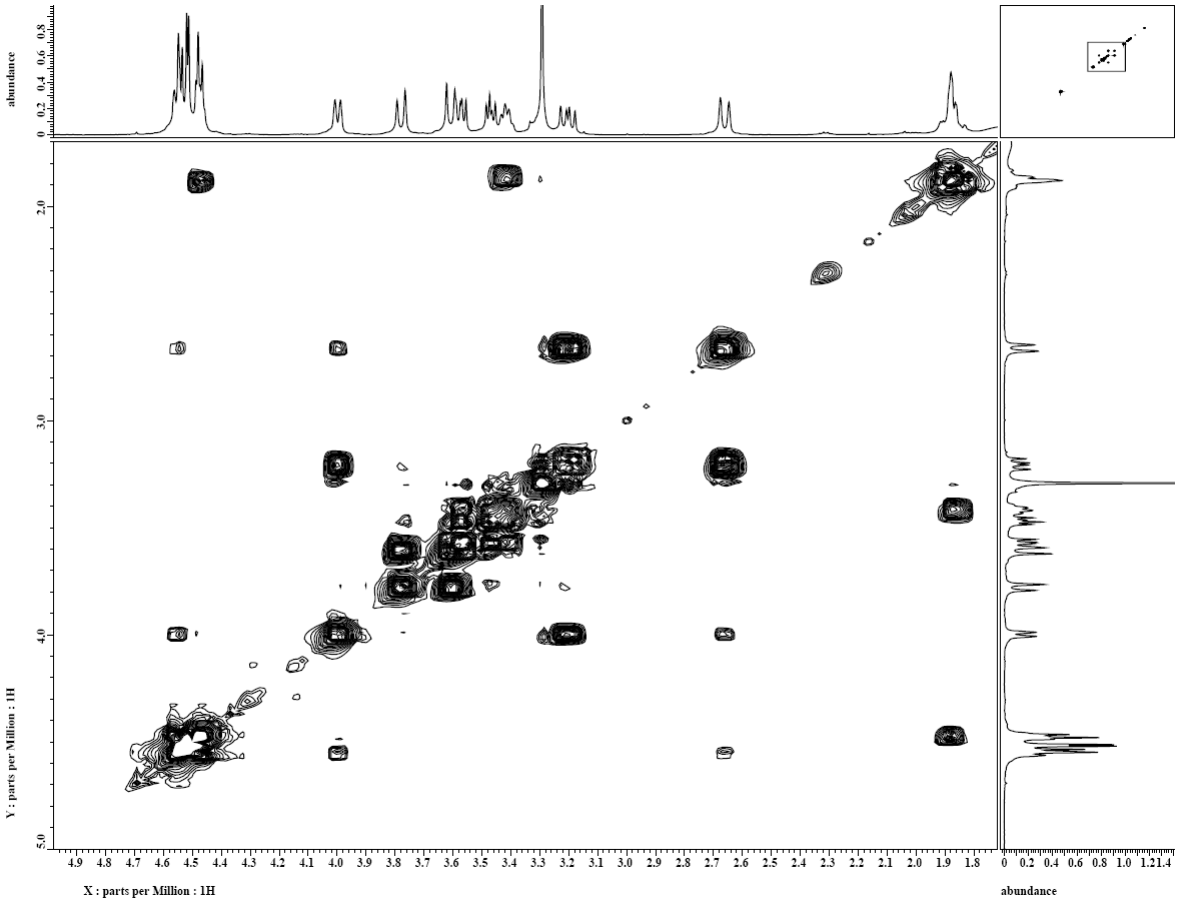


^{1}H NMR (500 MHz, $CDCl_3$)

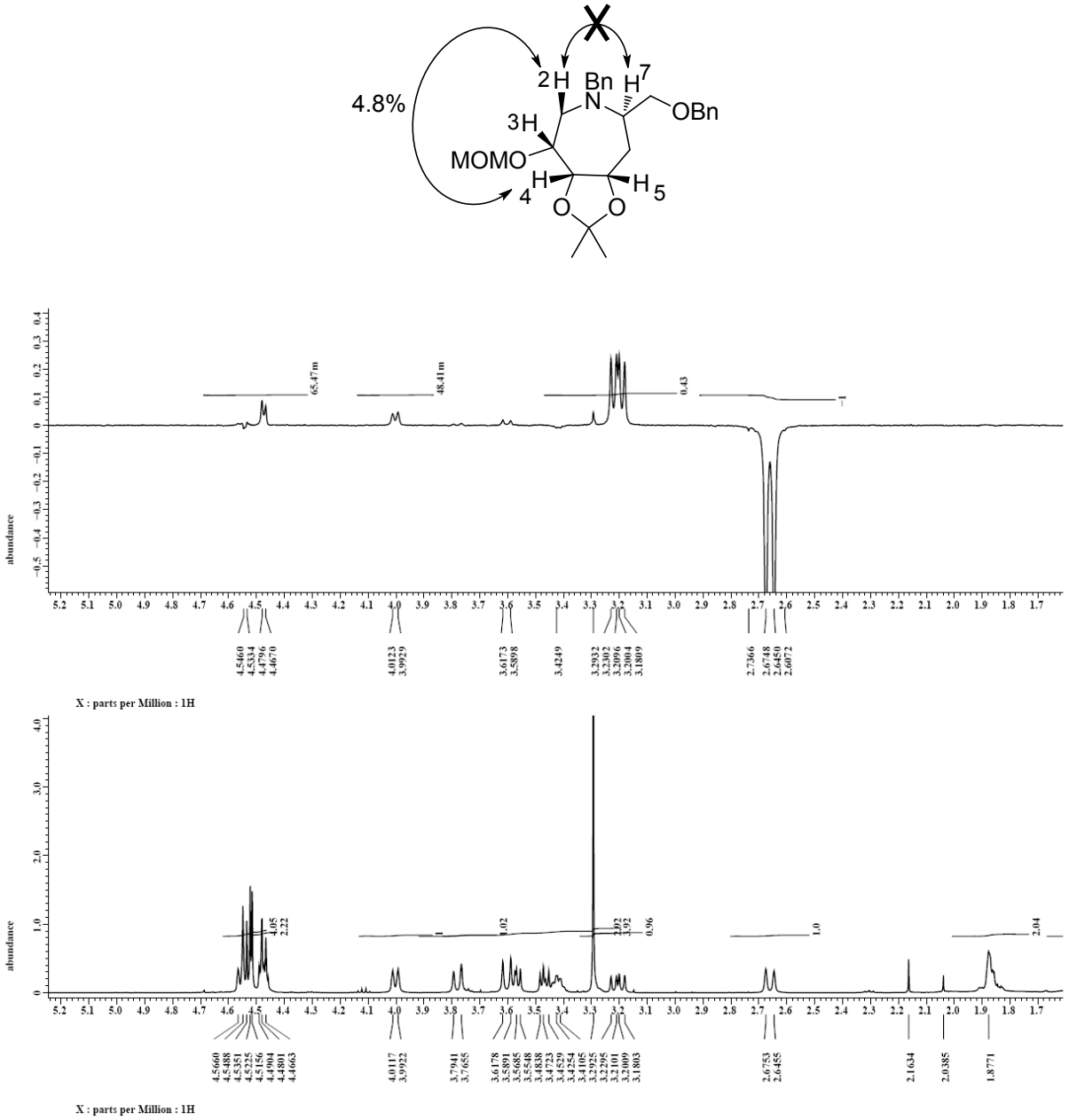


^{13}C NMR (125 MHz, $CDCl_3$)



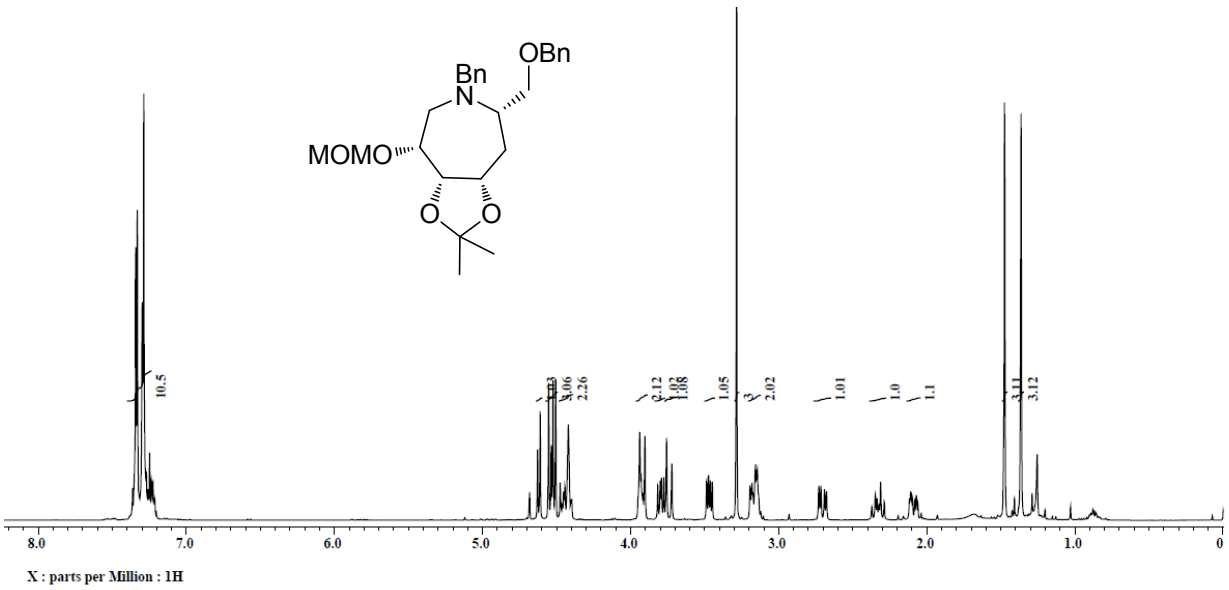


^{1}H-^{1}H COSY (500 MHz)

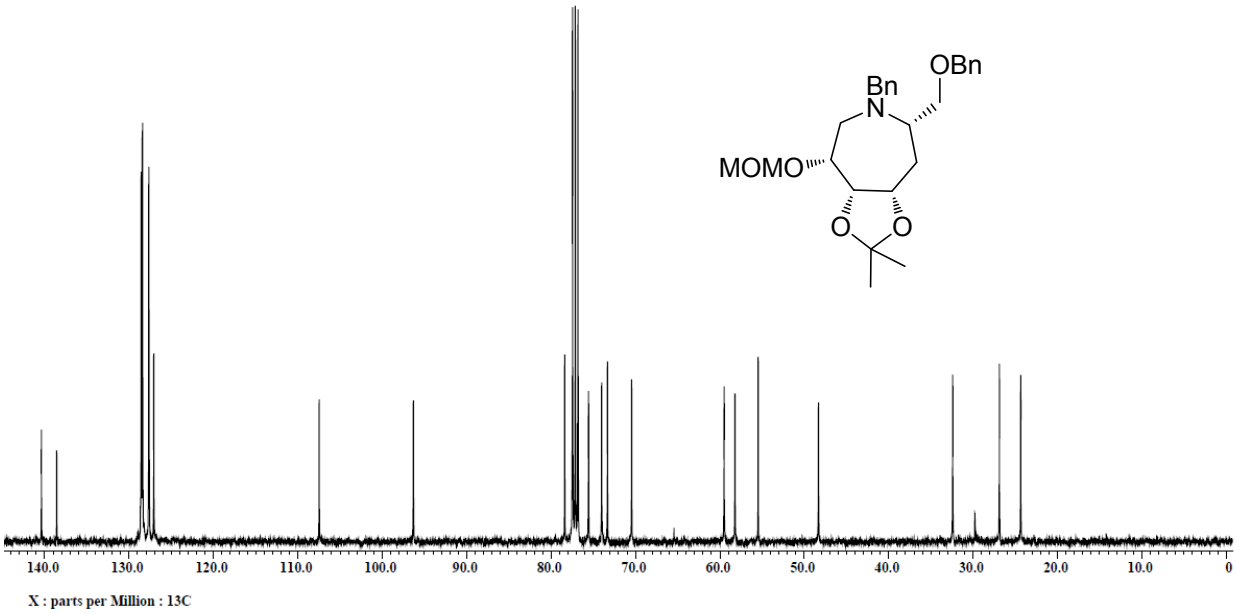


nOe spectrum (irradiation of H-2)

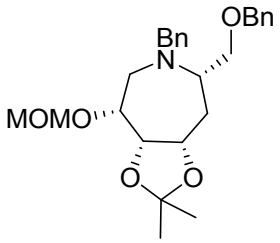
Compound 33b

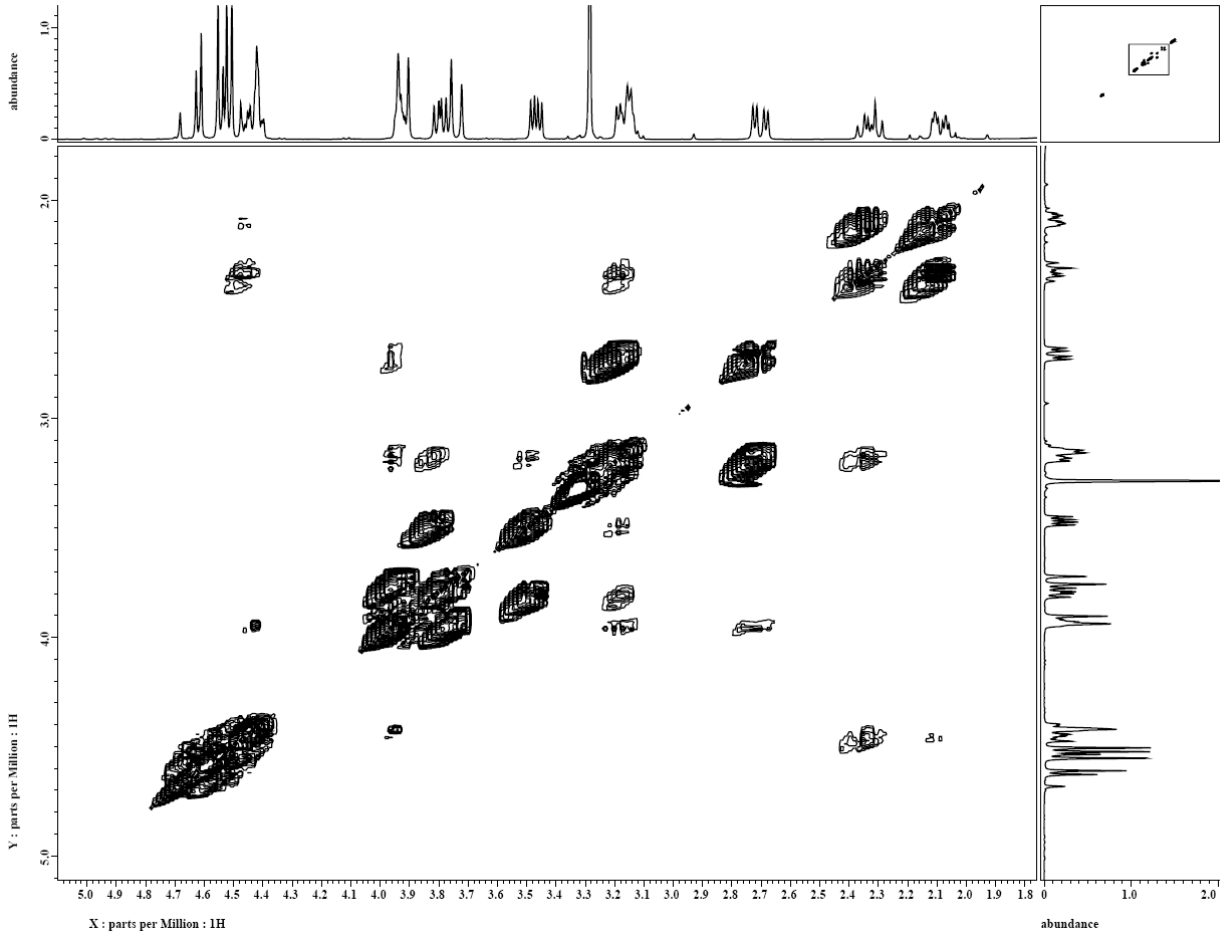


^{1}H NMR (500 MHz, $CDCl_3$)

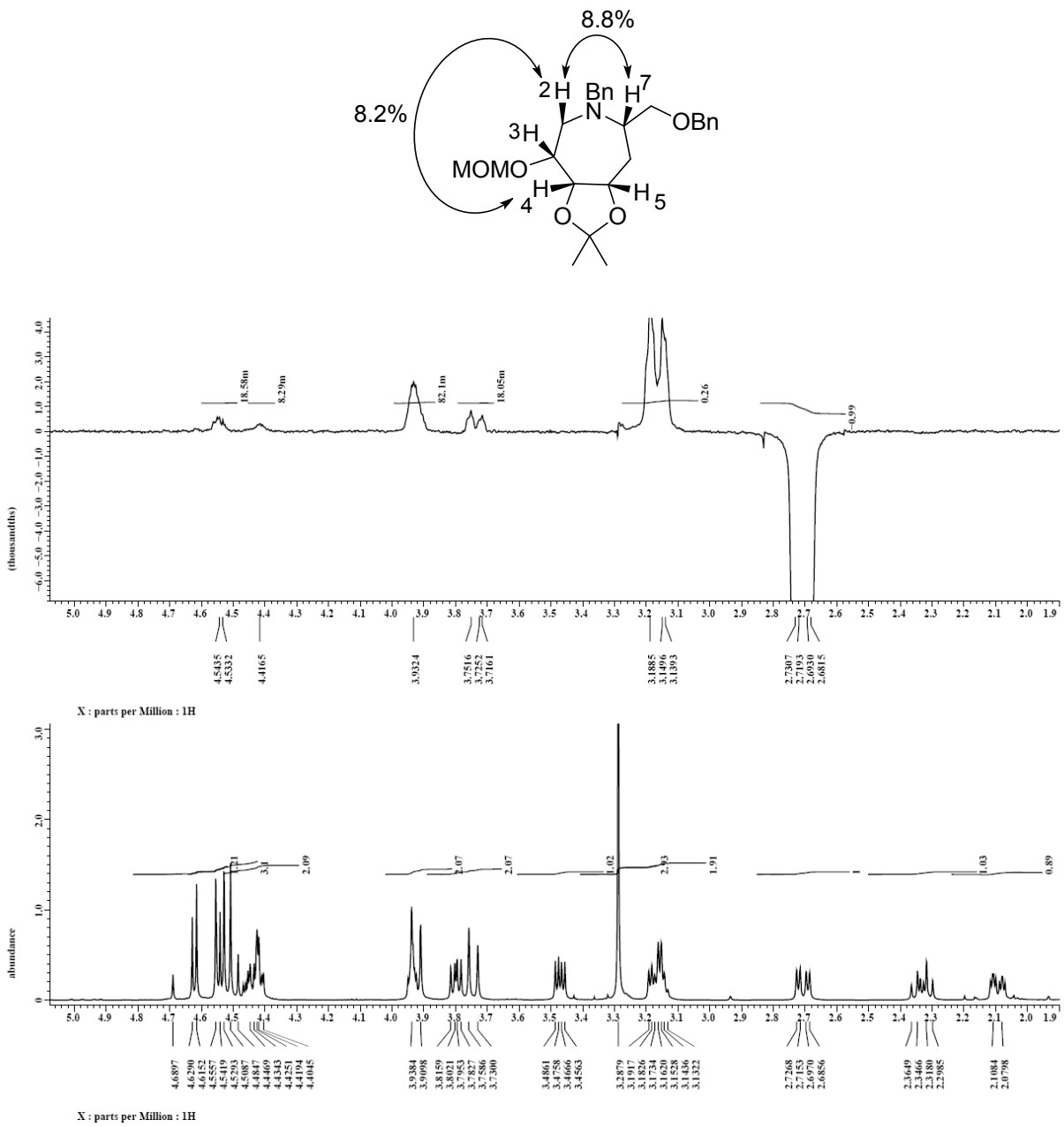


^{13}C NMR (125 MHz, $CDCl_3$)



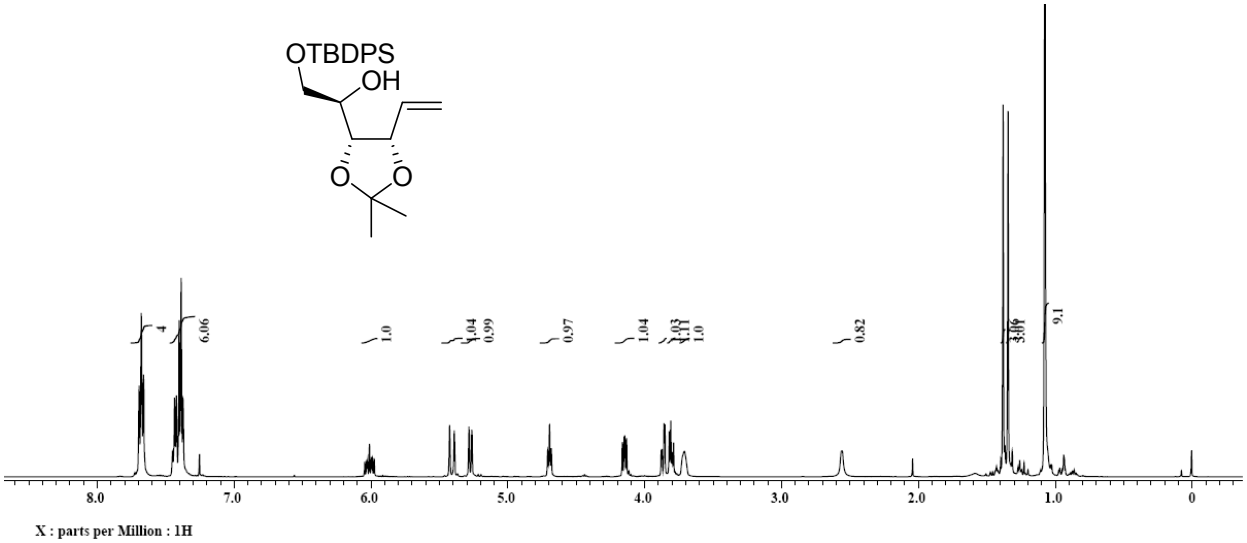


^{1}H-^{1}H COSY (500 MHz)

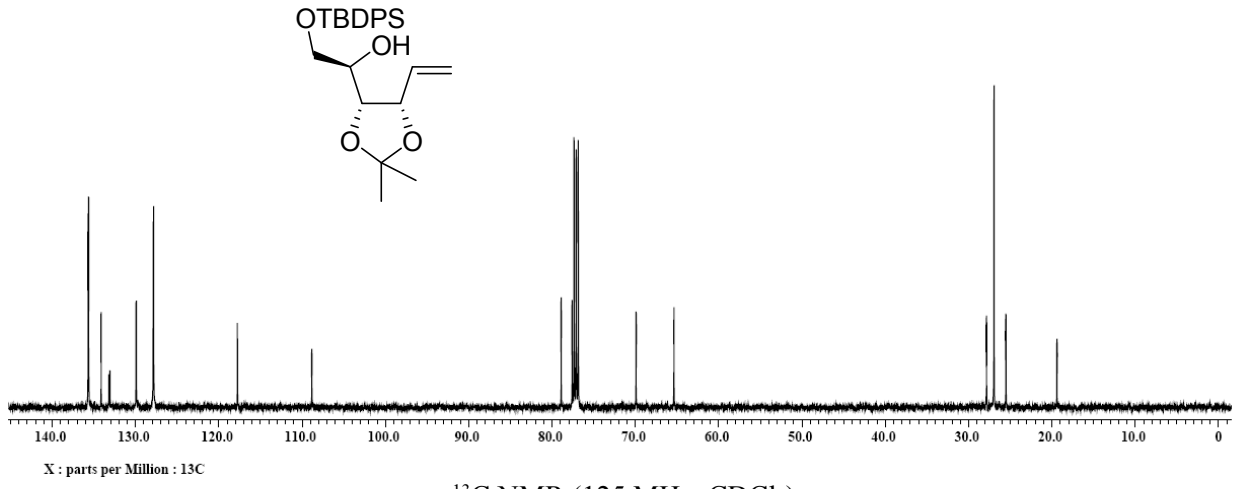


nOe spectrum (irradiation of H-2)

Compound 6

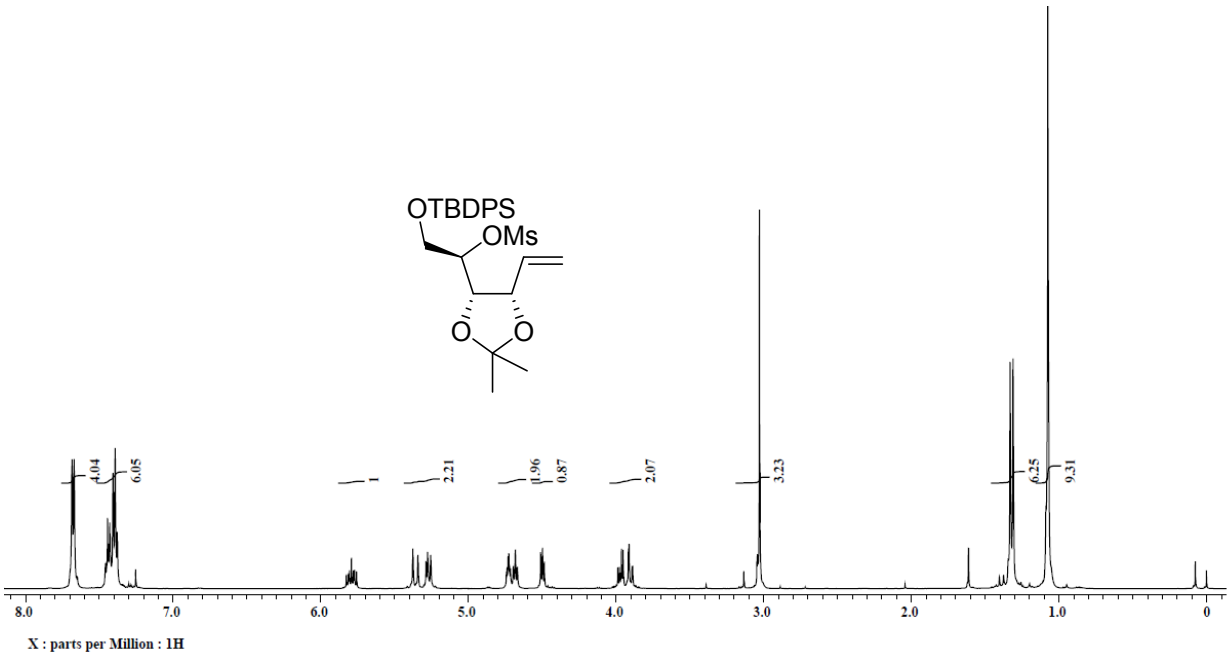


^{1}H NMR (500 MHz, $CDCl_3$)

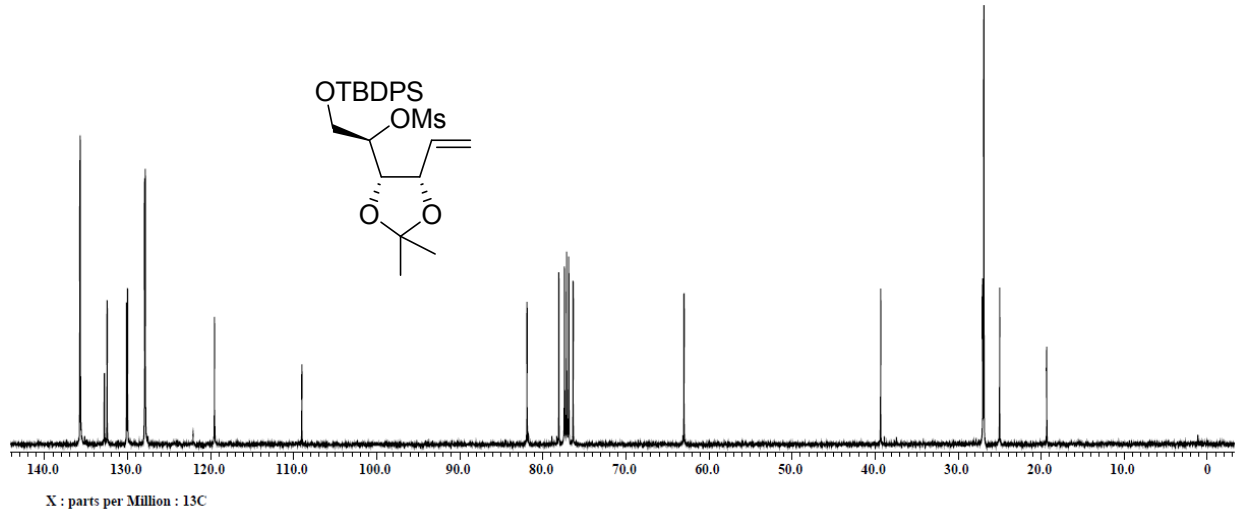


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 7

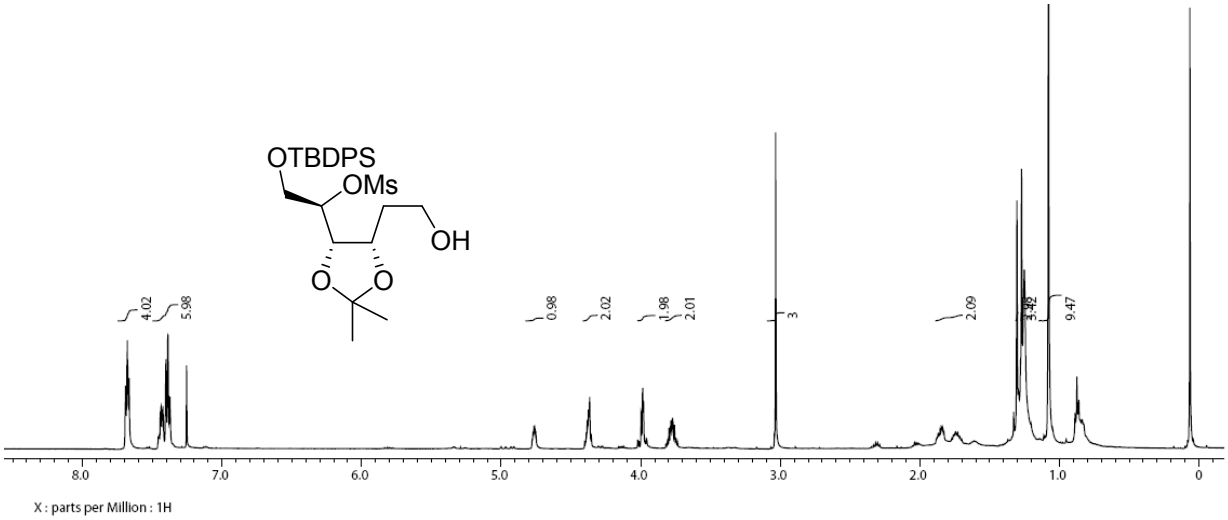


^{1}H NMR (500 MHz, $CDCl_3$)

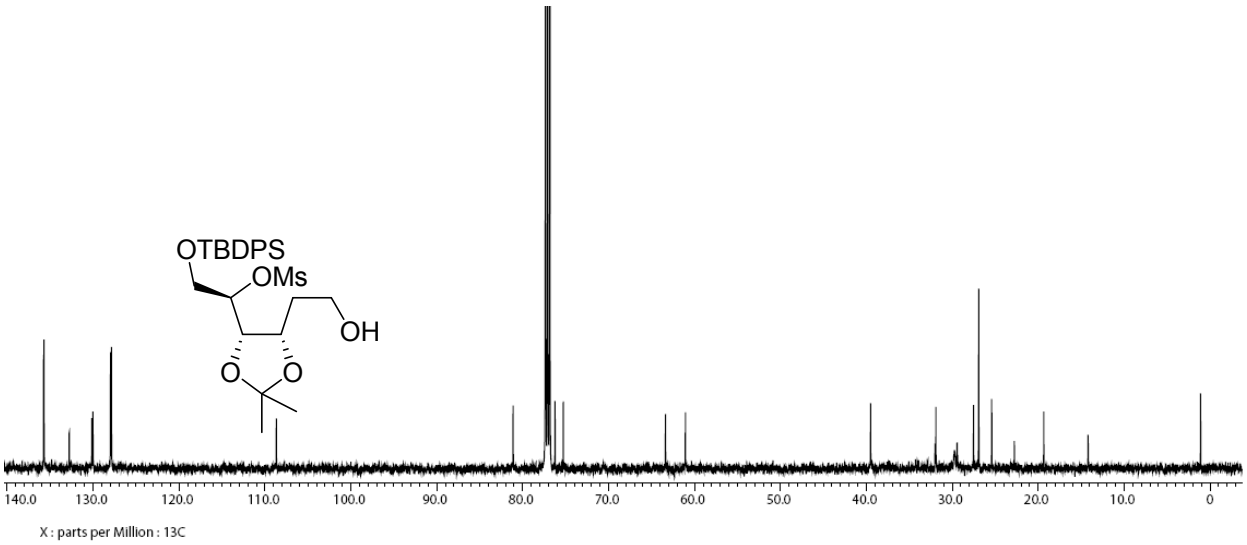


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 8

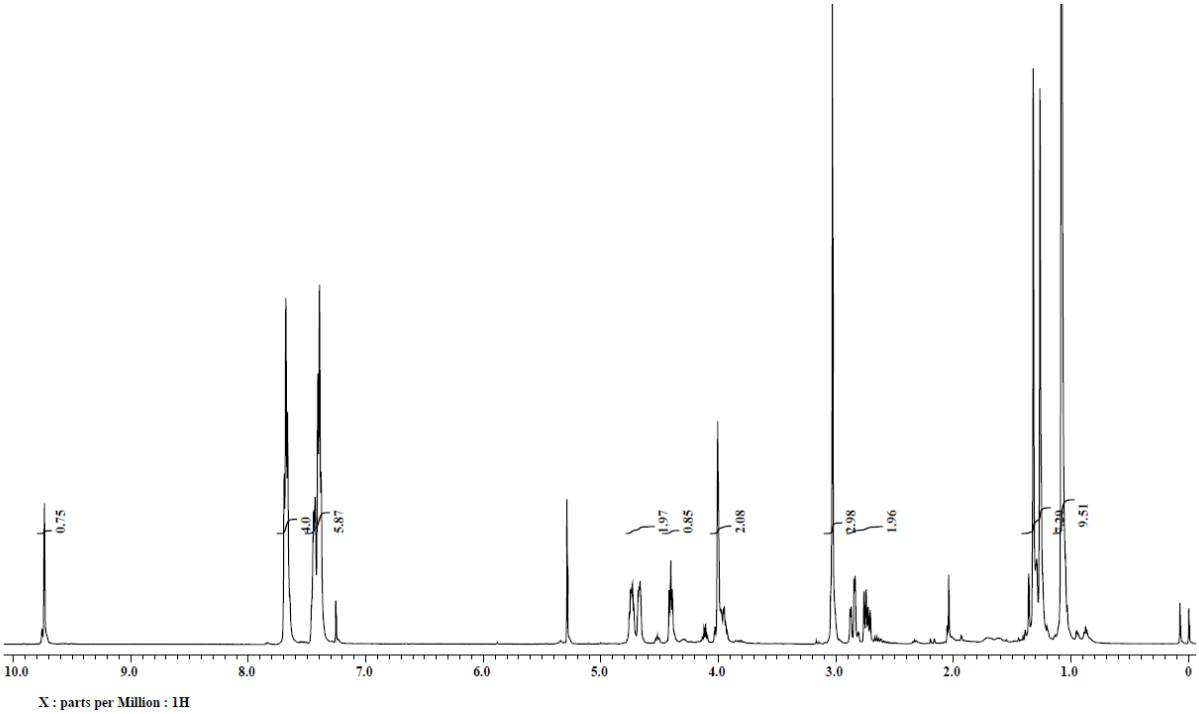


^{1}H NMR (500 MHz, $CDCl_3$)

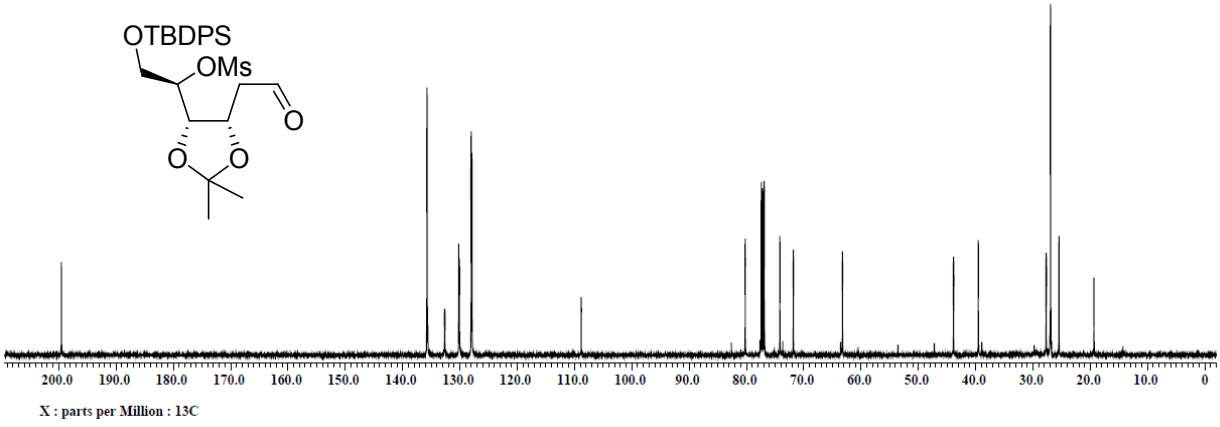


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 9

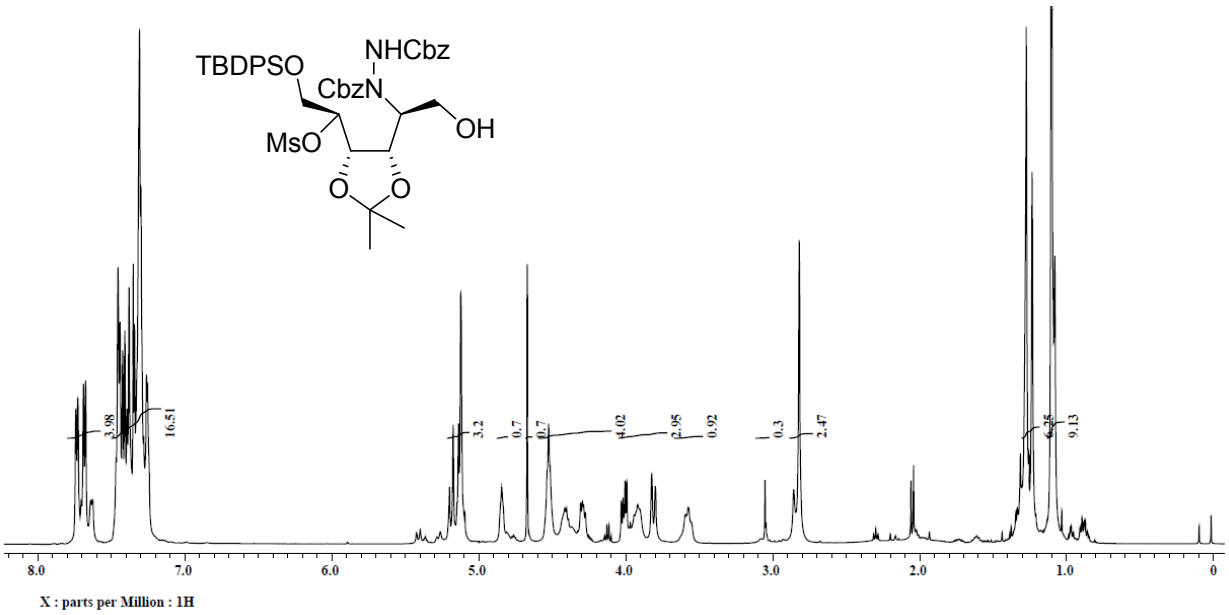


^{1}H NMR (500 MHz, $CDCl_3$)

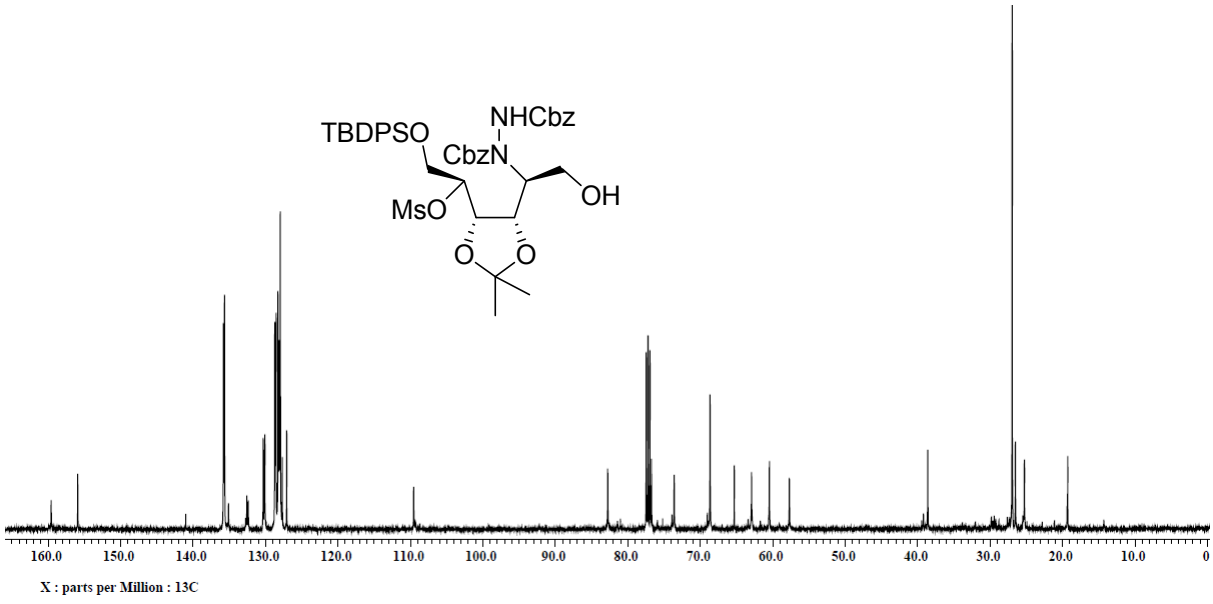


^{13}C NMR (125 MHz, $CDCl_3$)

Compound10a

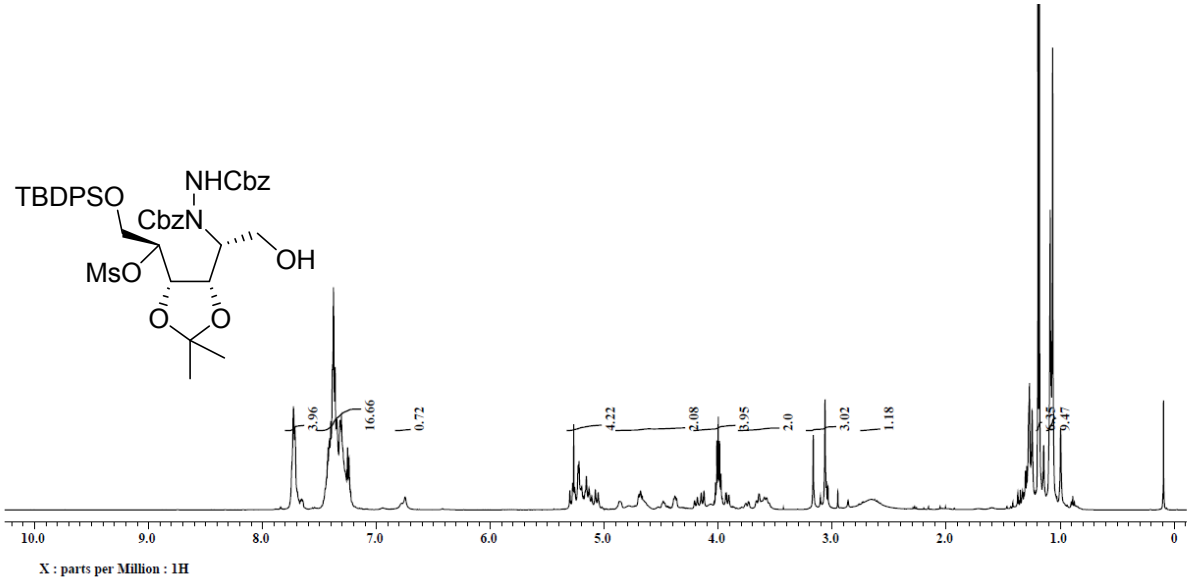


^{1}H NMR (500 MHz, $CDCl_3$)

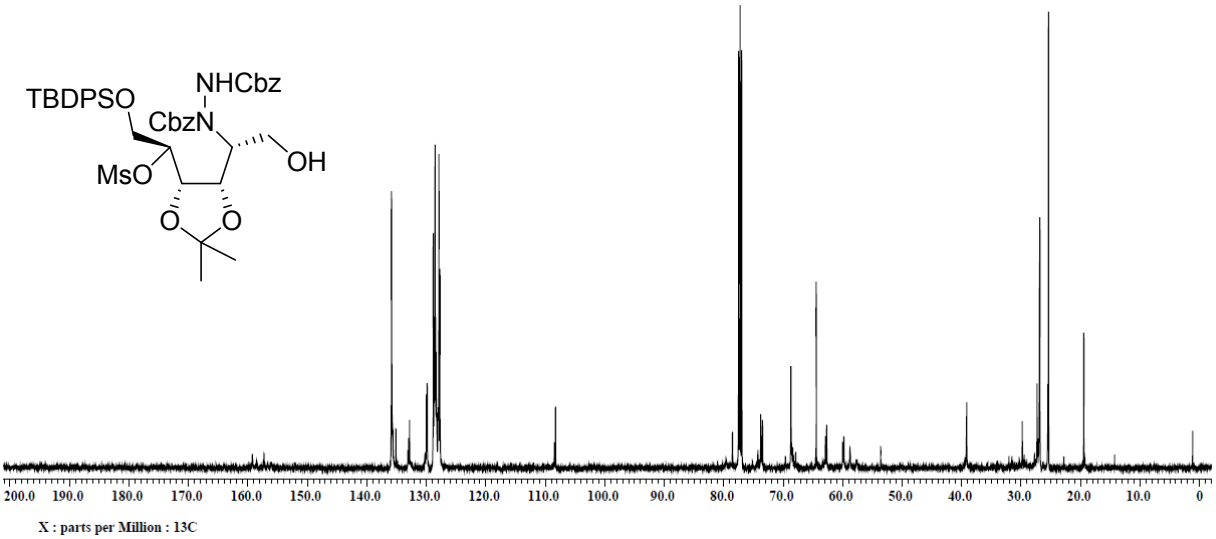


^{13}C NMR (125 MHz, $CDCl_3$)

Compound10b

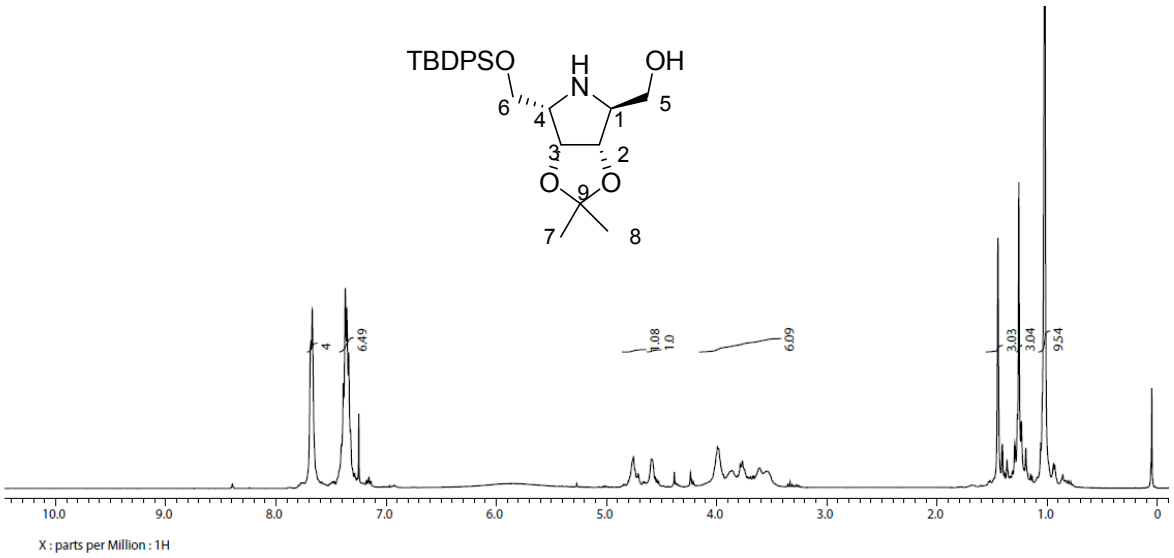


^{1}H NMR (500 MHz, $CDCl_3$)

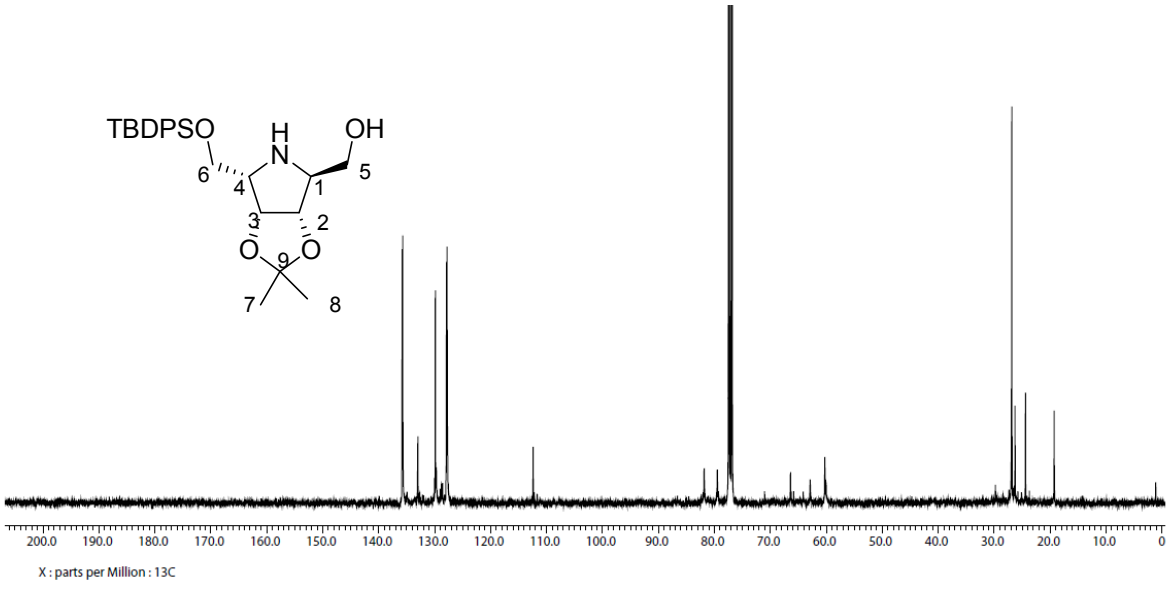


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 11a

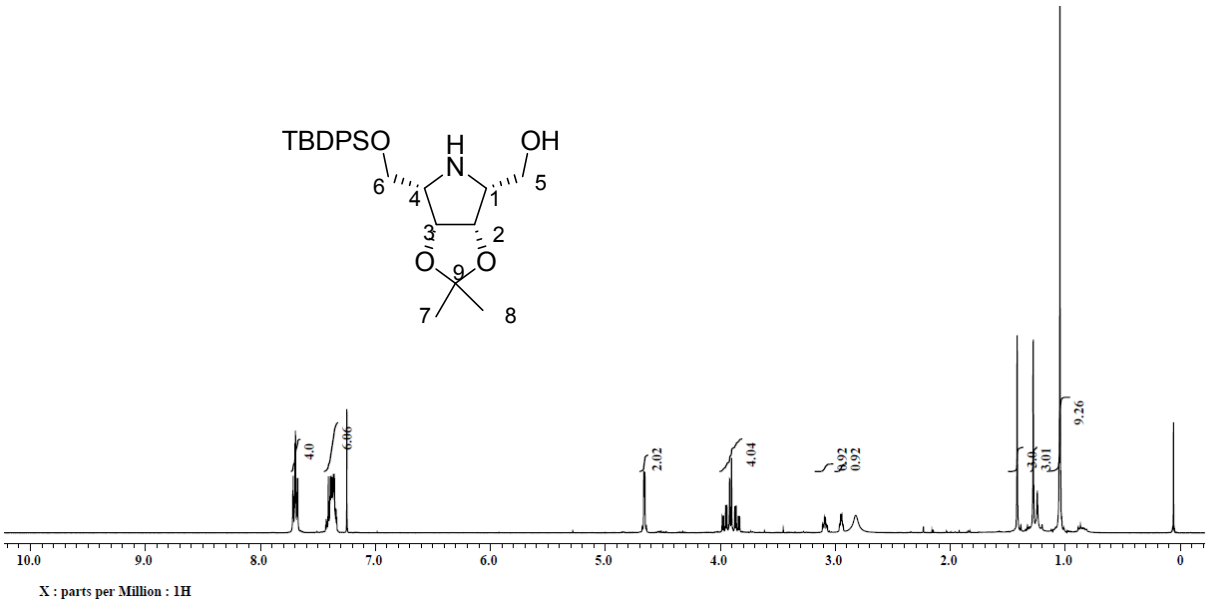


^{1}H NMR (500 MHz, $CDCl_3$)

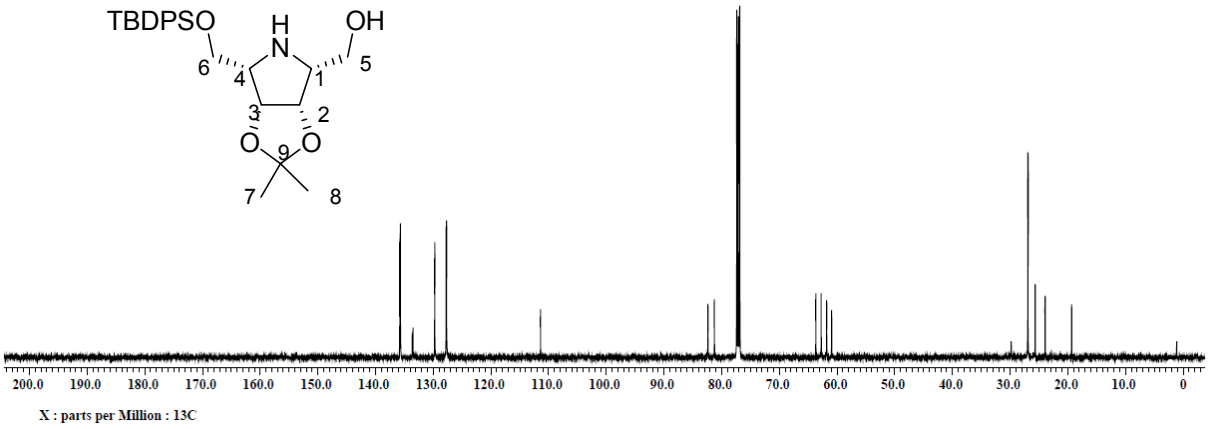


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 11b

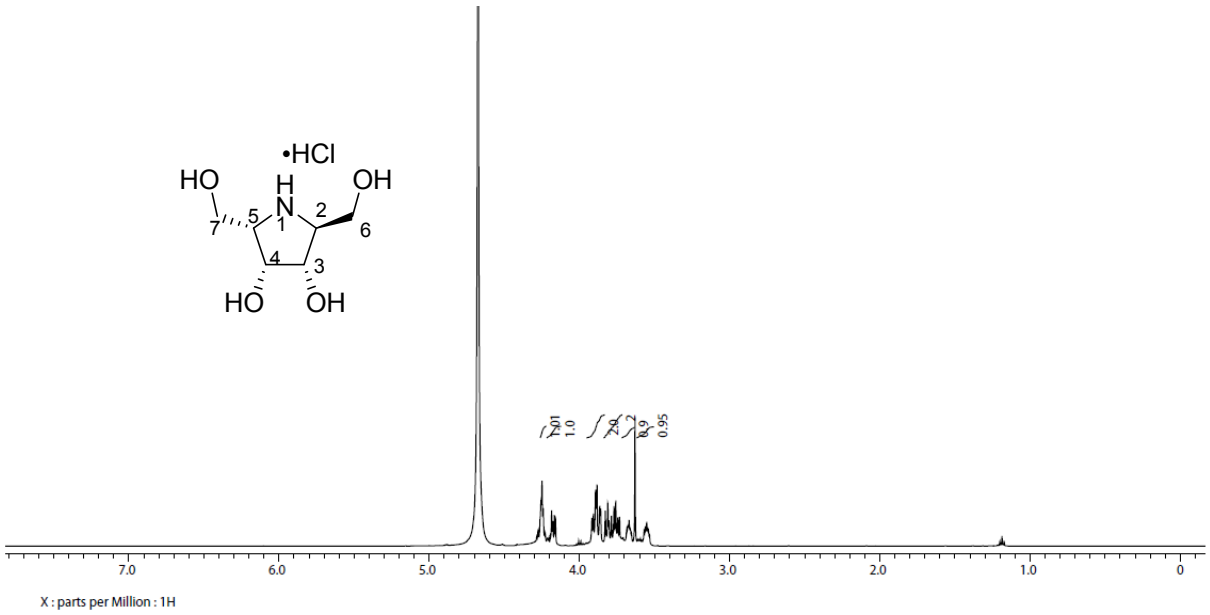


^{1}H NMR (500 MHz, $CDCl_3$)

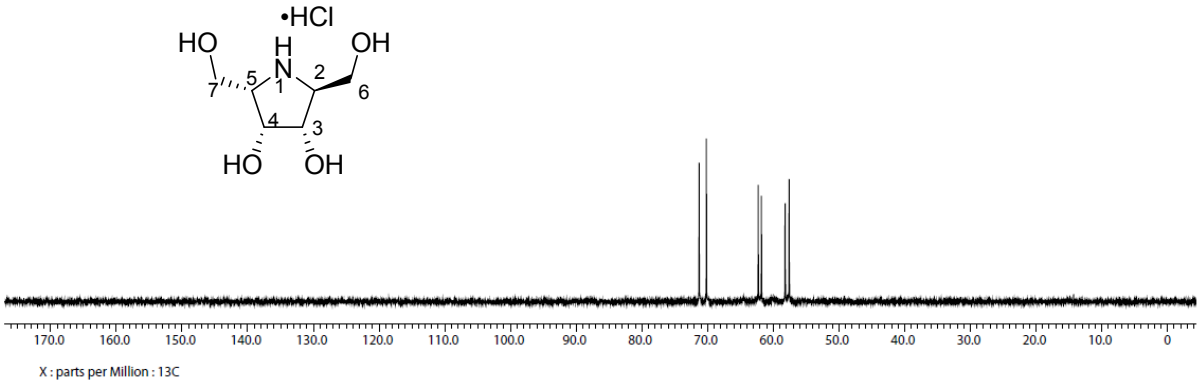


^{13}C NMR (125 MHz, $CDCl_3$)

Compound1a

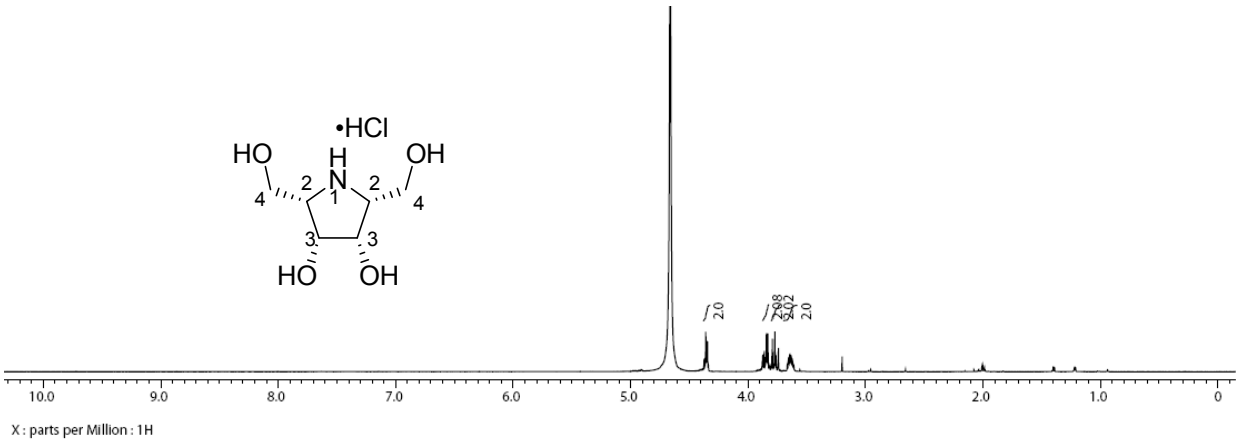


^{1}H NMR (500 MHz, D_2O) spectrum of

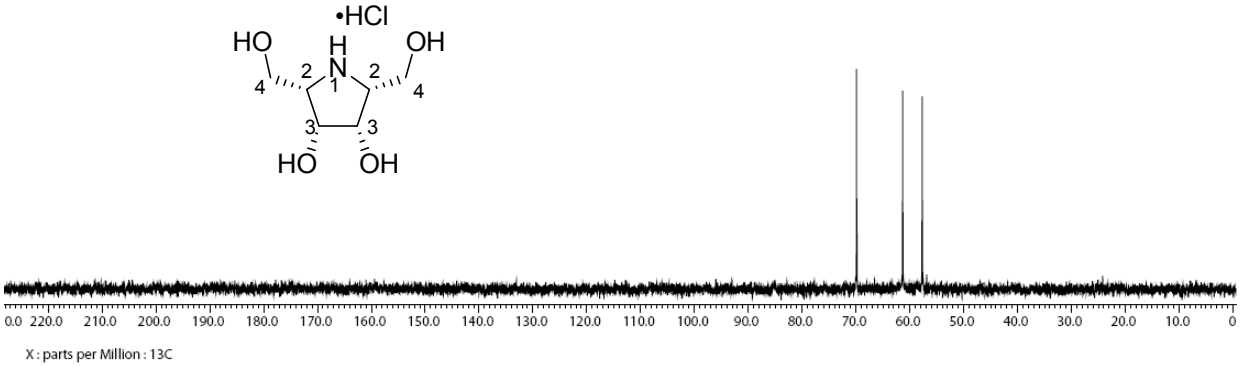


^{13}C NMR (125 MHz, D_2O) spectrum of compound **1a**

Compound1b

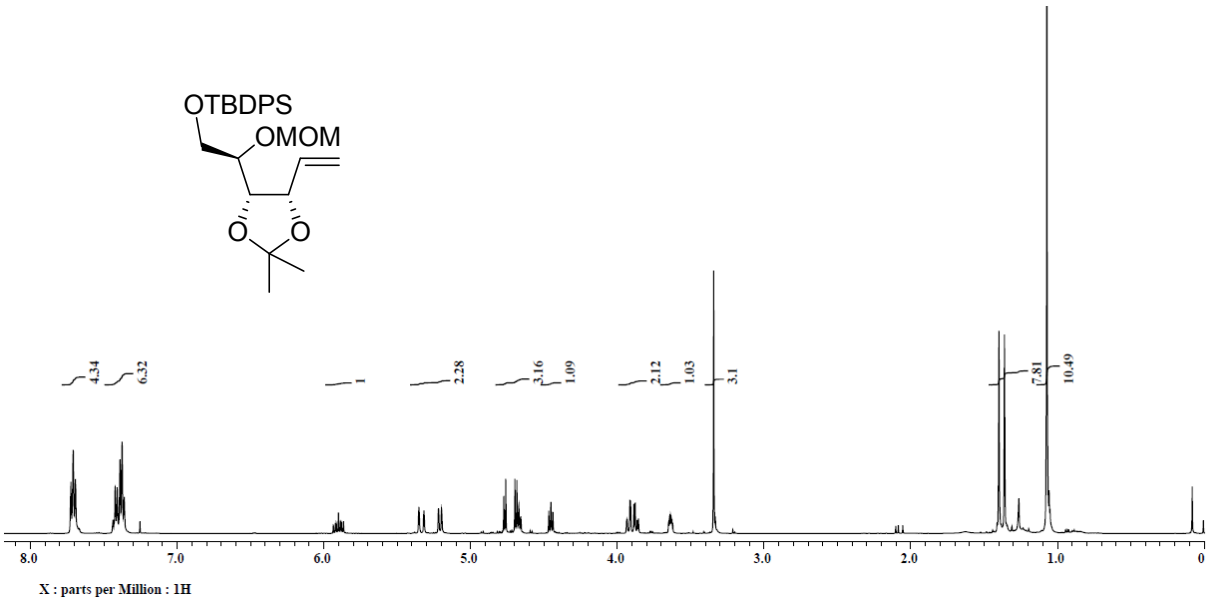


^{1}H NMR (400 MHz, D_2O)

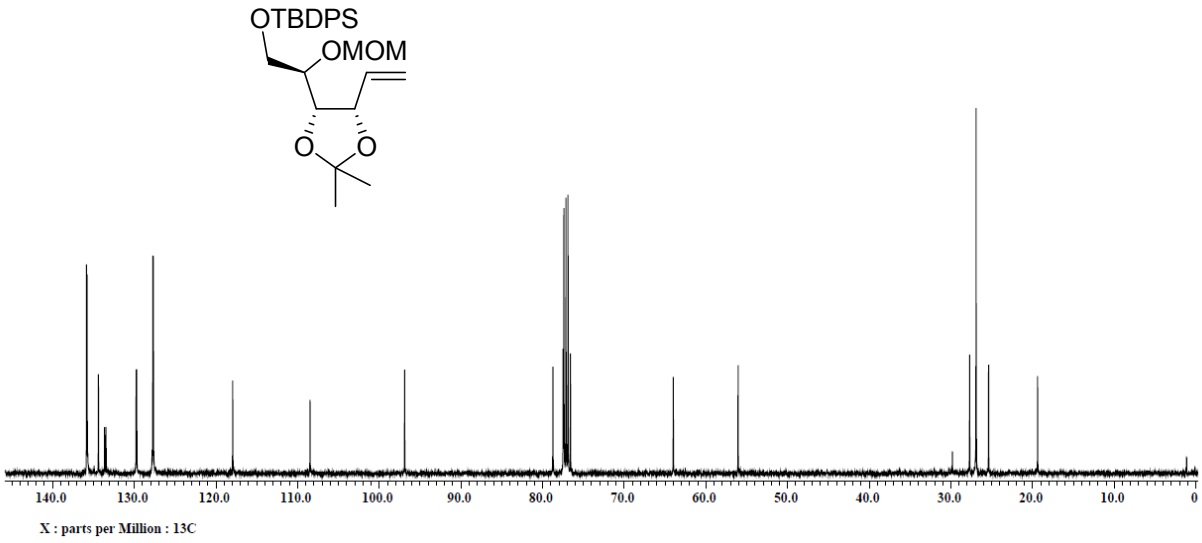


^{13}C NMR (100 MHz, D_2O)

Compound12

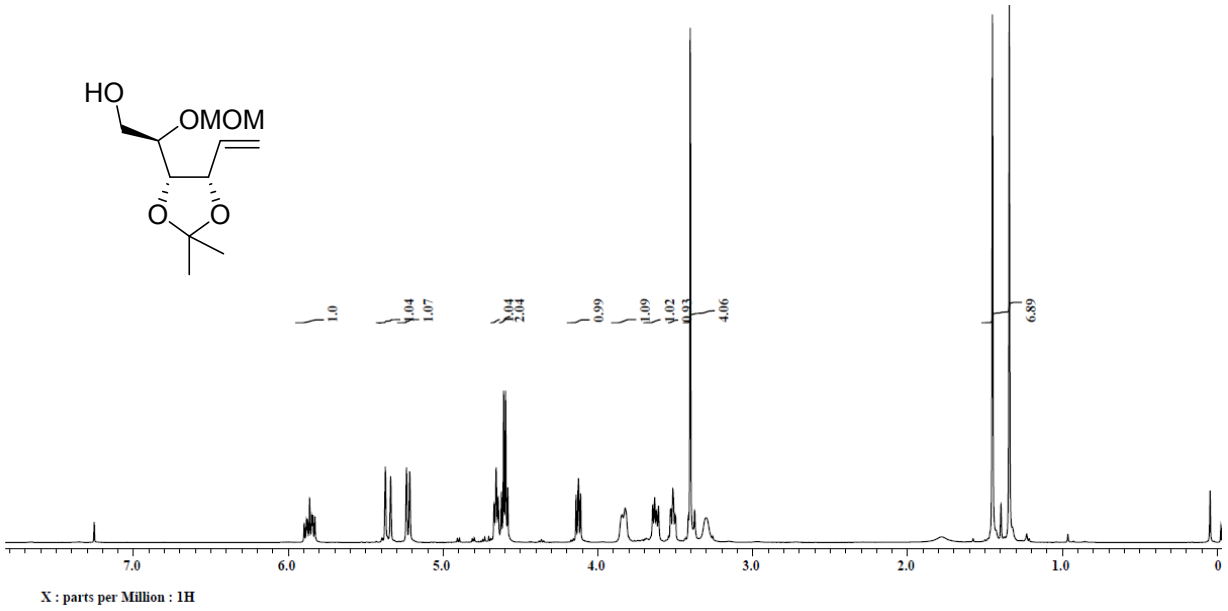


^{1}H NMR (500 MHz, $CDCl_3$)

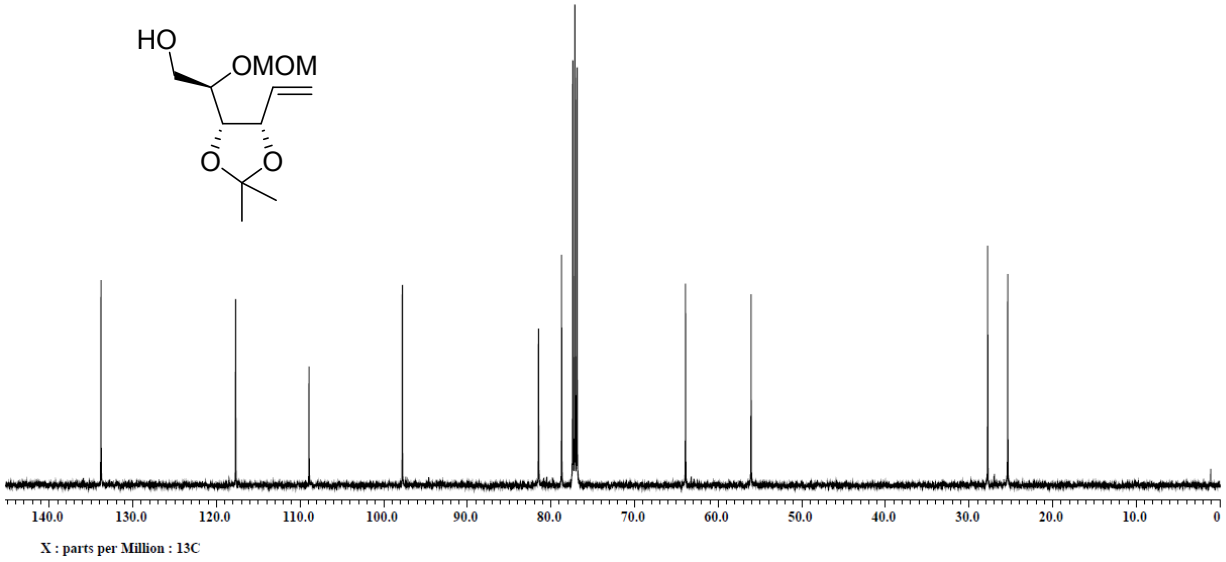


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 13

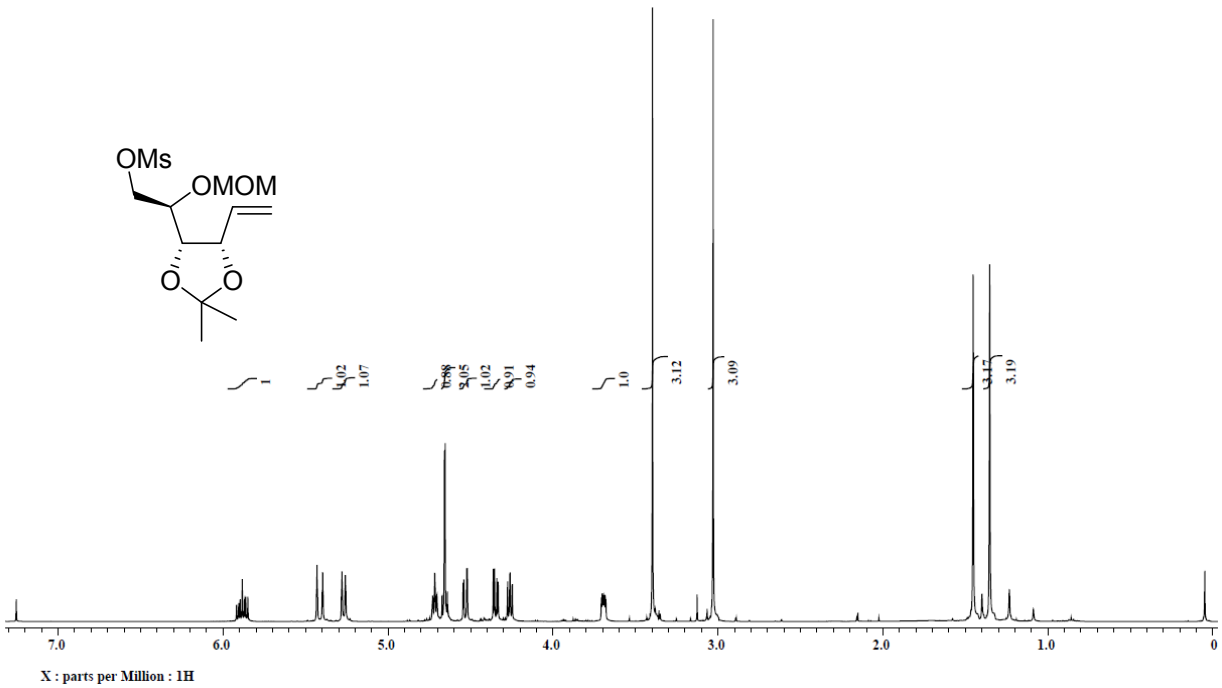


^{1}H NMR (500 MHz, $CDCl_3$)

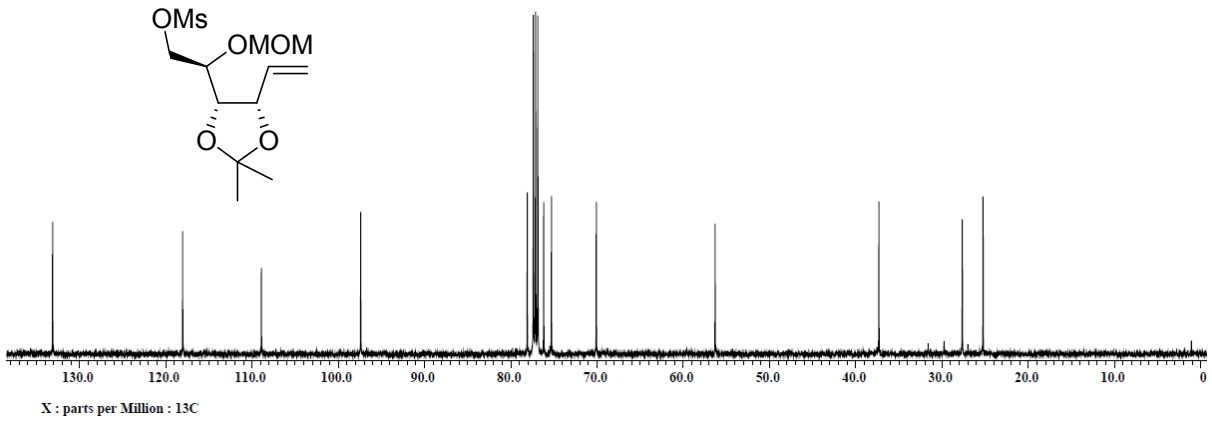


^{13}C NMR (125 MHz, $CDCl_3$

Compound 14

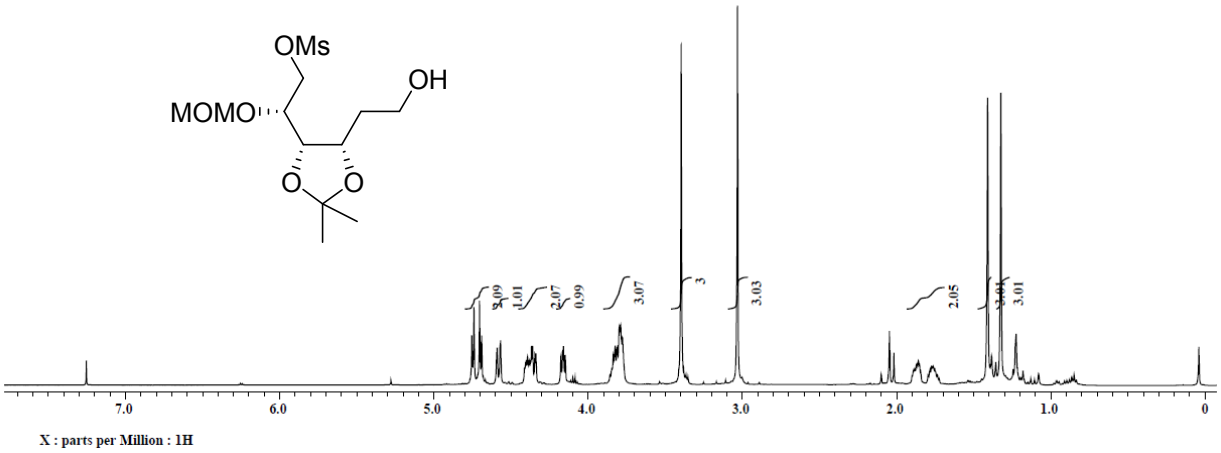


^{1}H NMR (500 MHz, $CDCl_3$)

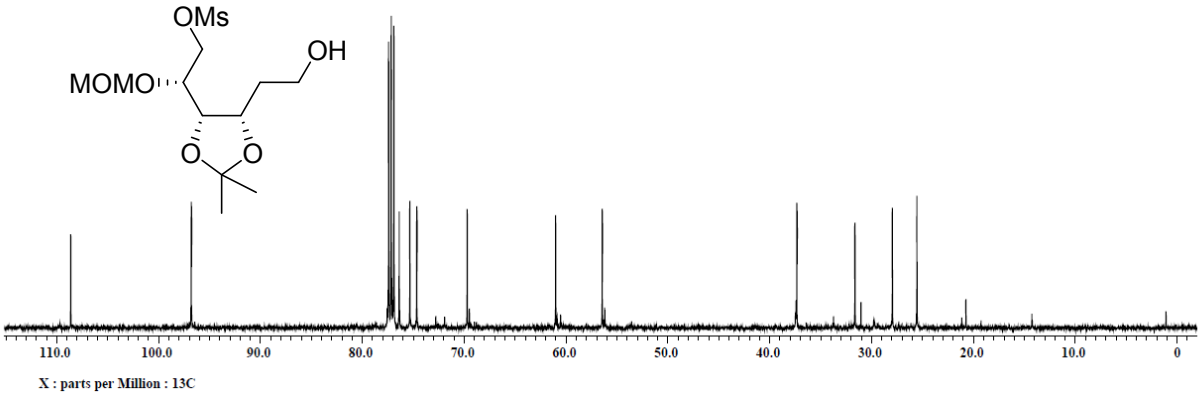


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 15

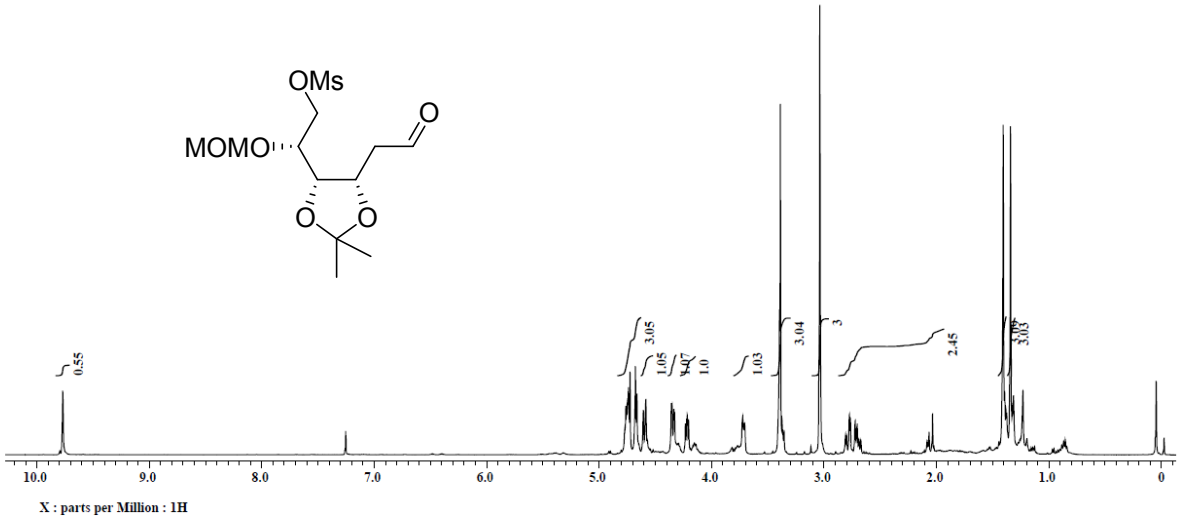


^{1}H NMR (500 MHz, $CDCl_3$)

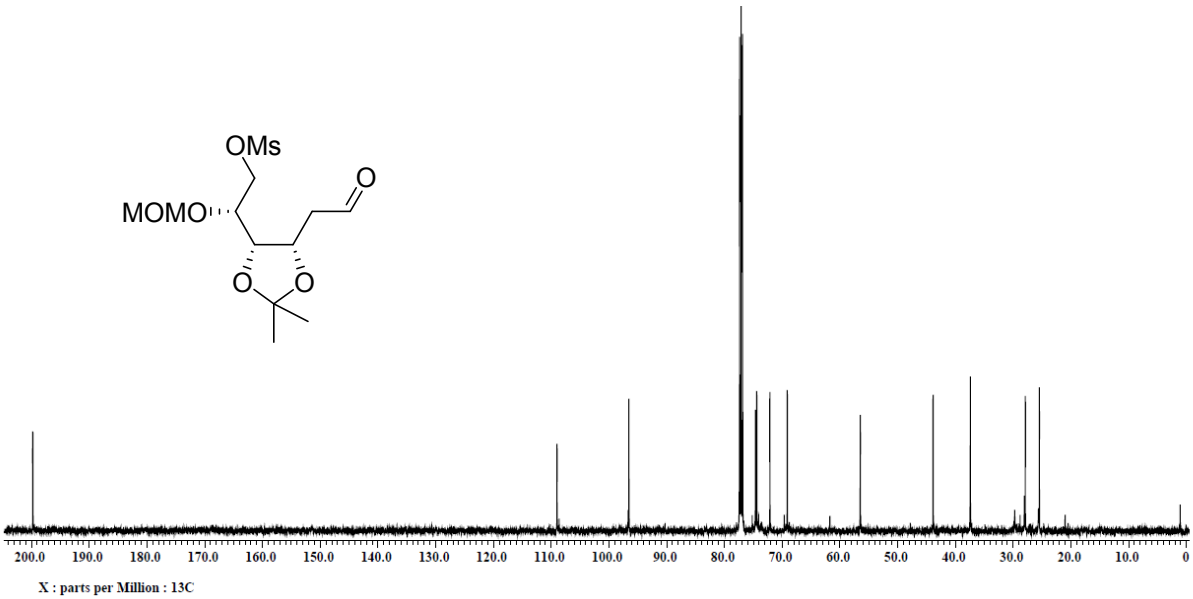


^{13}C NMR (125 MHz, $CDCl_3$)

Compound16

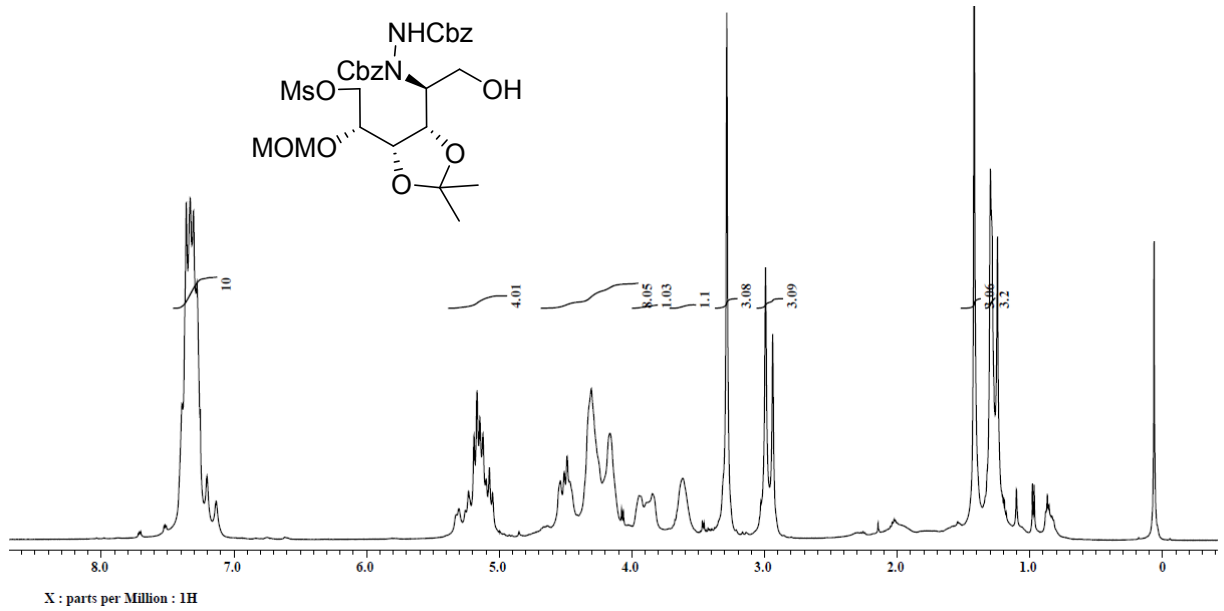


^{1}H NMR (500 MHz, $CDCl_3$)

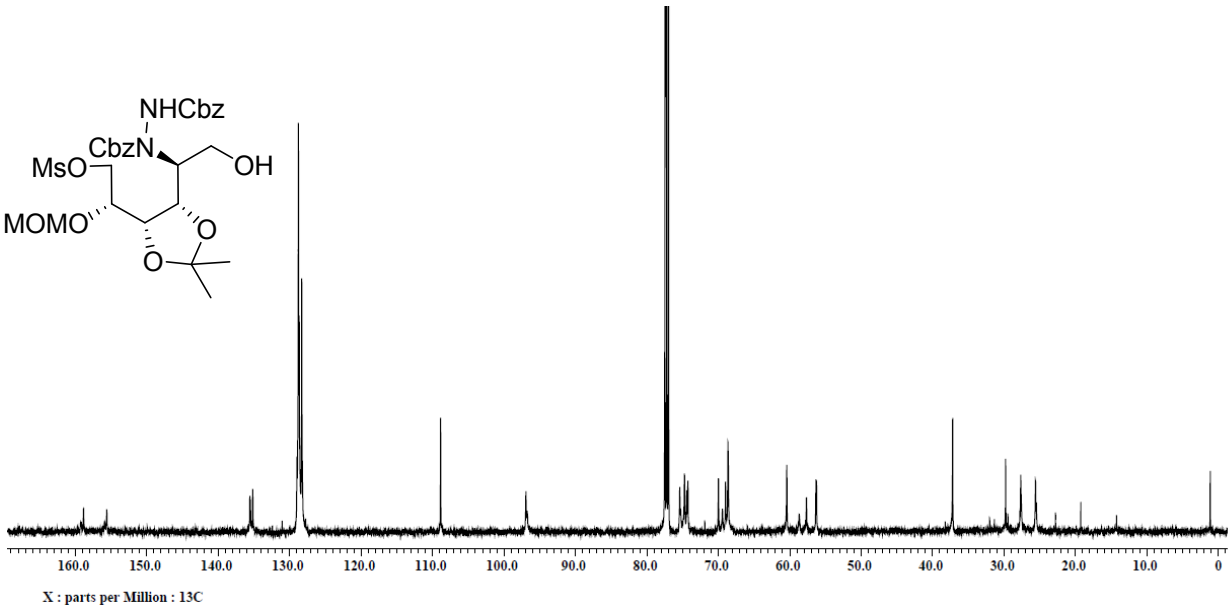


^{13}C NMR (125 MHz, $CDCl_3$)

Compound17a

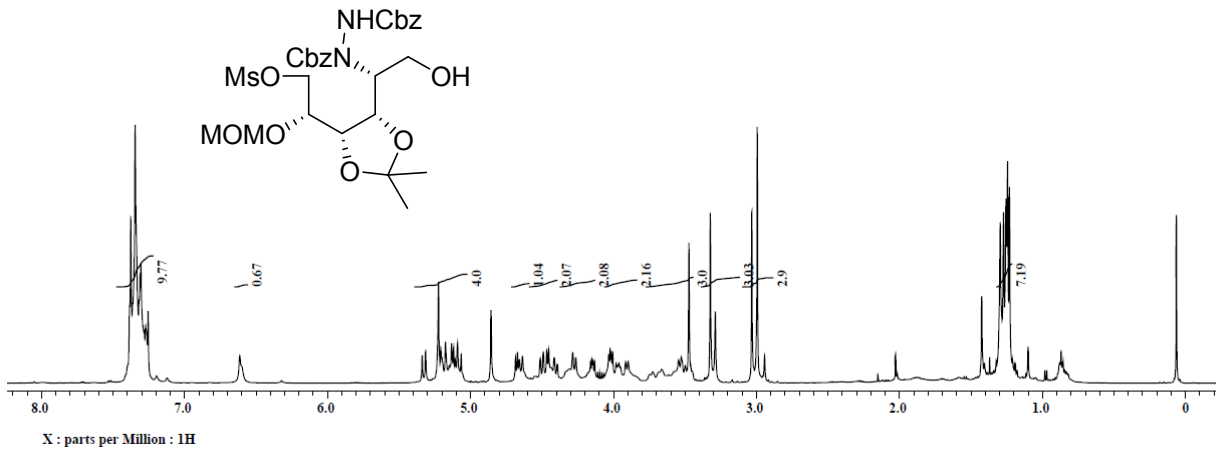


^{1}H NMR (500 MHz, $CDCl_3$)

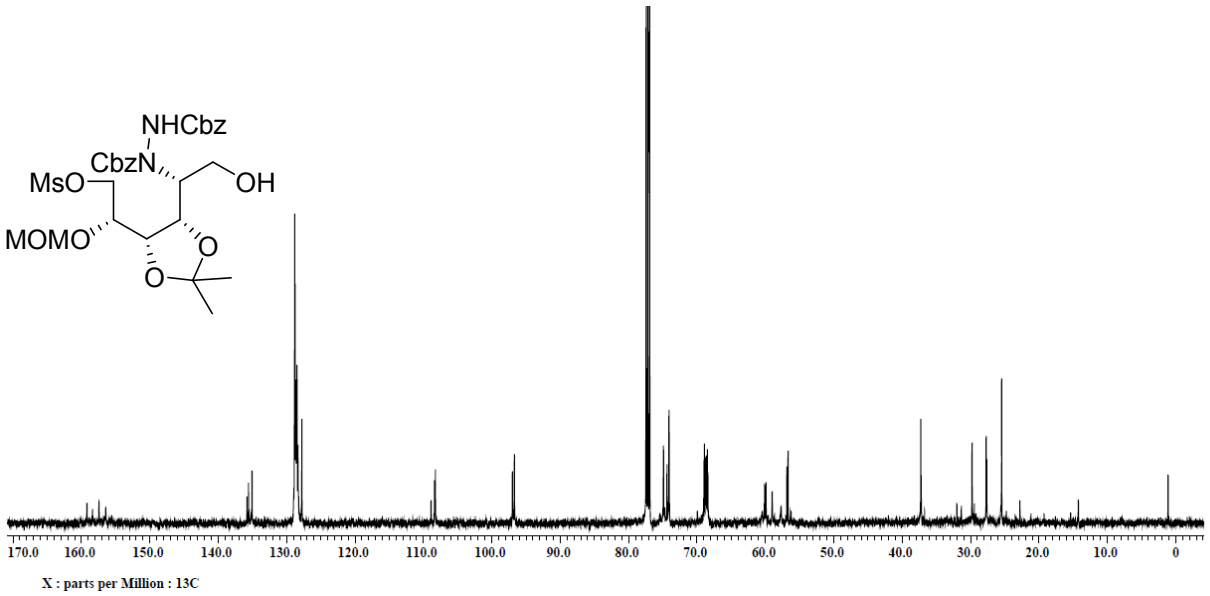


^{13}C NMR (125 MHz, $CDCl_3$)

Compound7b

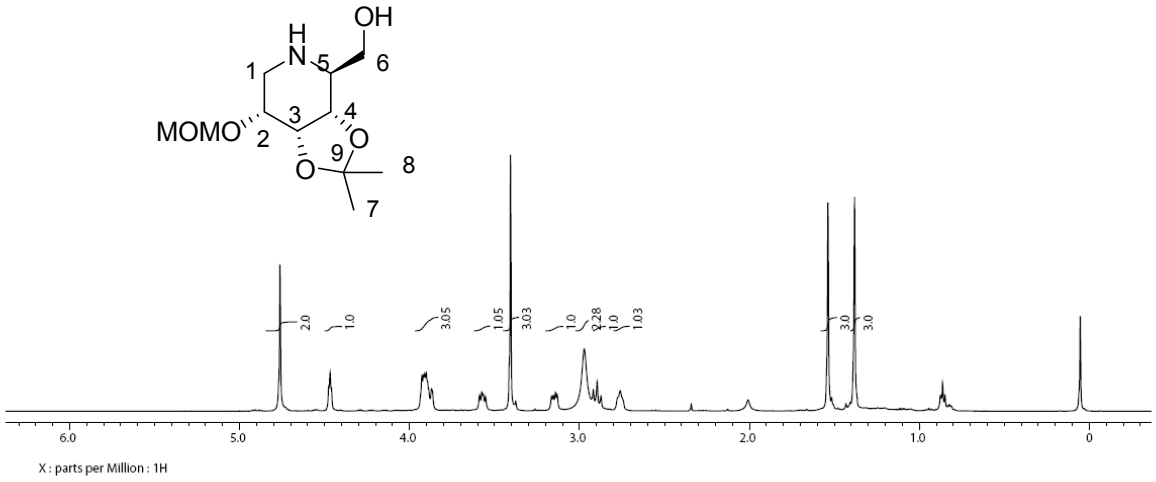


^{1}H NMR (500 MHz, $CDCl_3$)

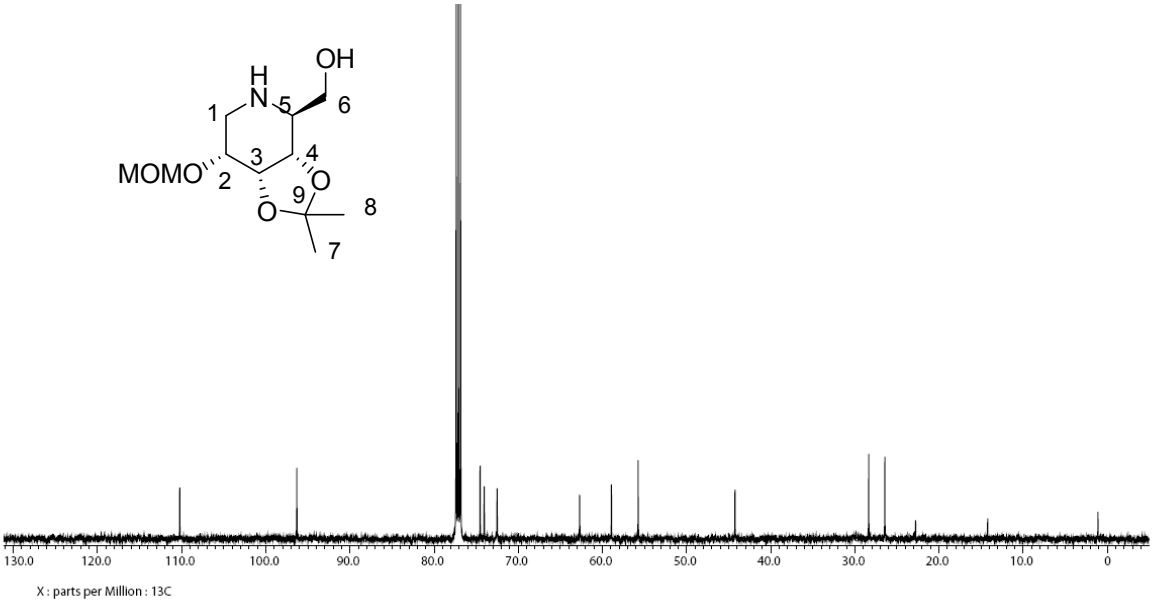


^{13}C NMR (125 MHz, $CDCl_3$)

Compound18a

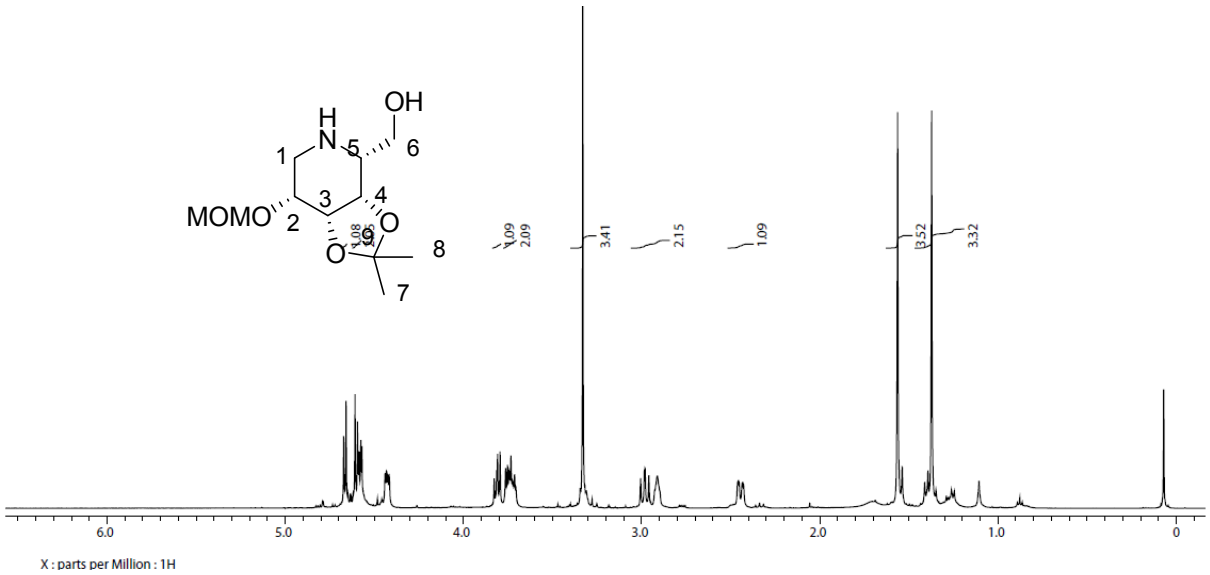


^{1}H NMR (500 MHz, $CDCl_3$)

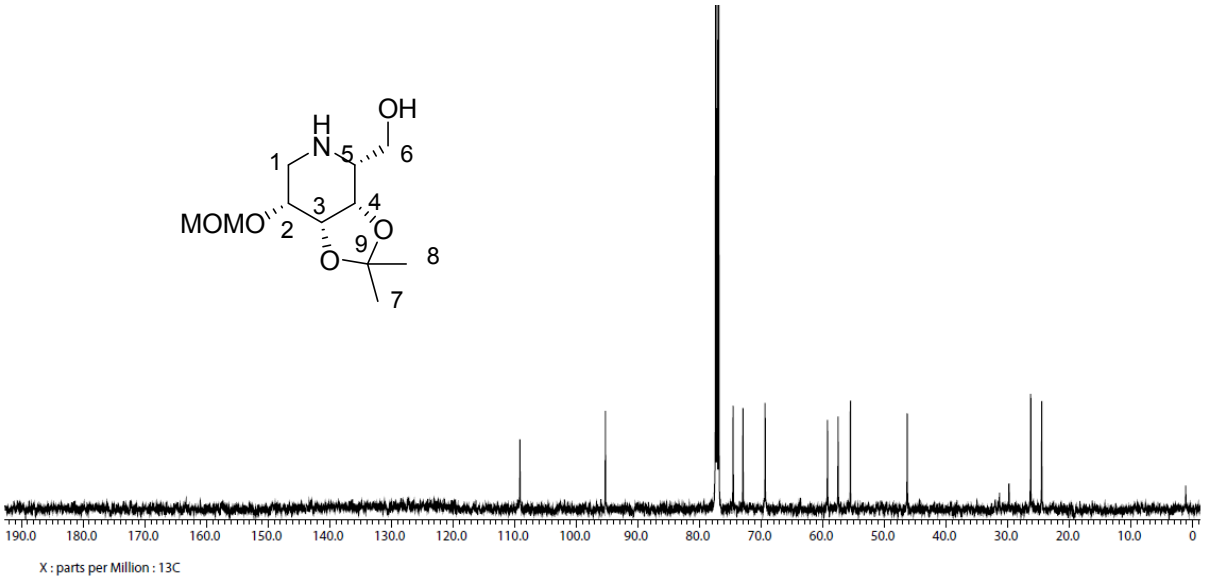


^{13}C NMR (125 MHz, $CDCl_3$)

Compound18b

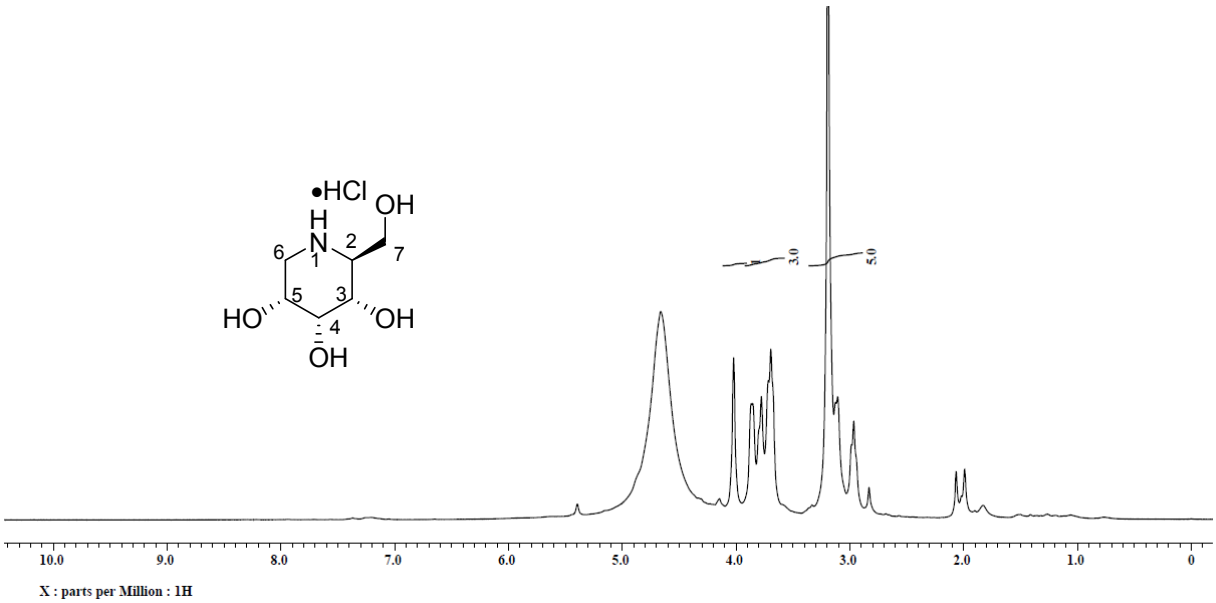


^{1}H NMR (500 MHz, $CDCl_3$)

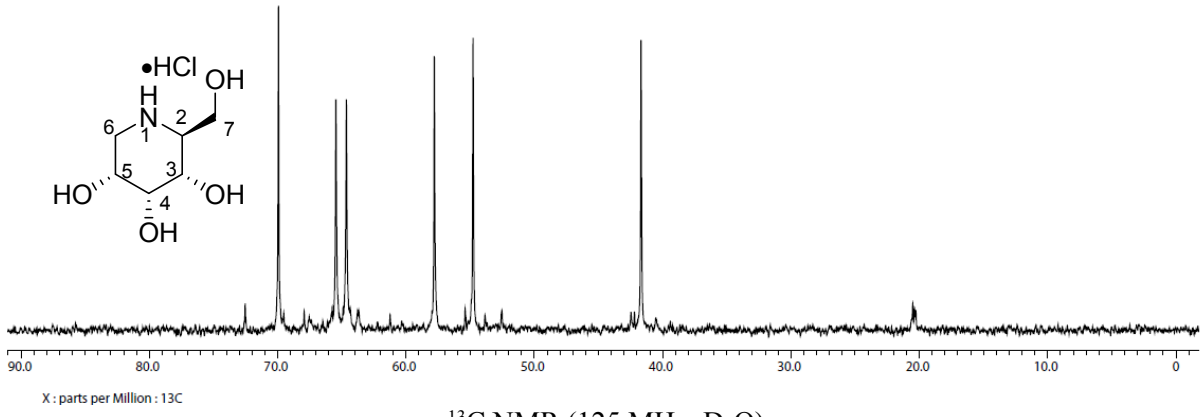


^{13}C NMR (125 MHz, $CDCl_3$)

Compound2a

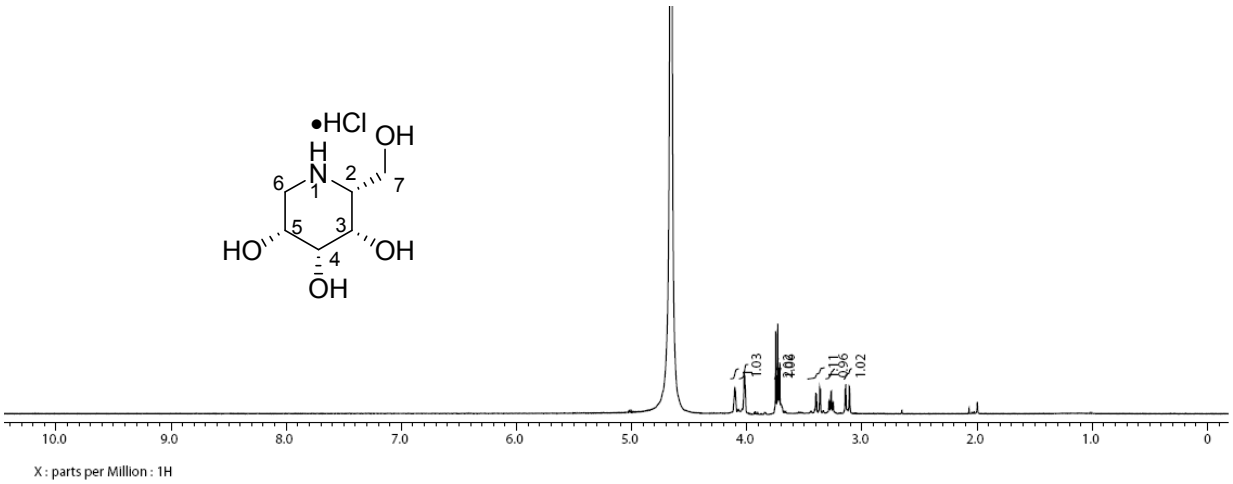


^{1}H NMR (500 MHz, D_2O)

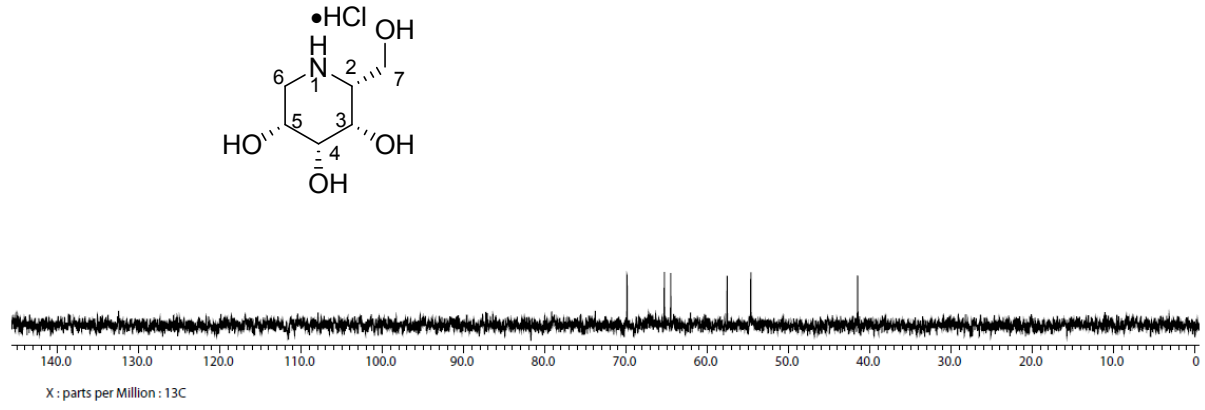


^{13}C NMR (125 MHz, D_2O)

Compound 2b

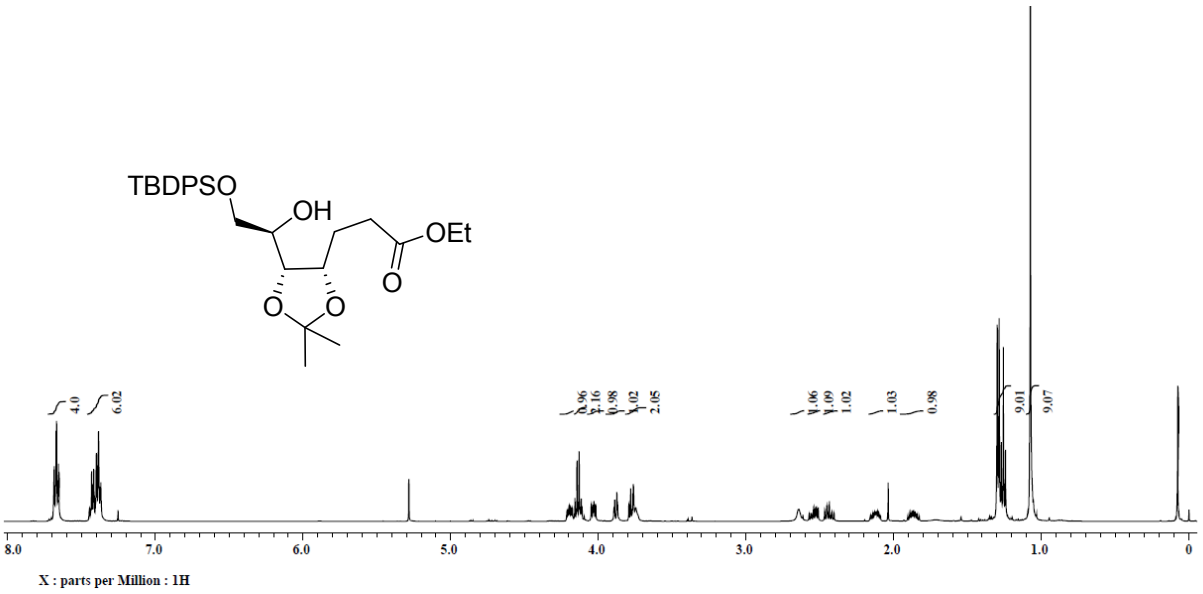


^{1}H NMR (500 MHz, D_2O)

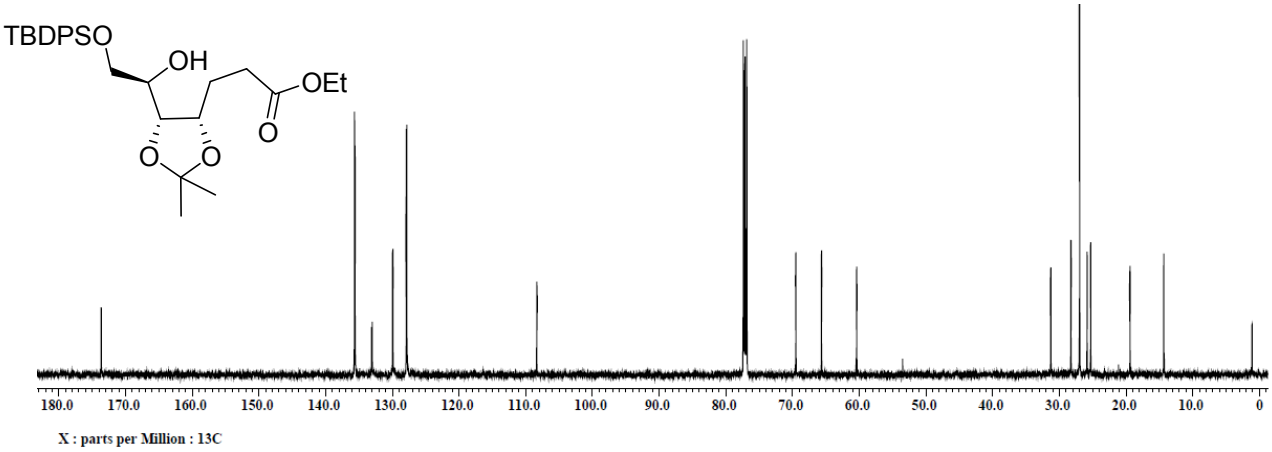


^{13}C NMR (100 MHz, D_2O)

Compound 19

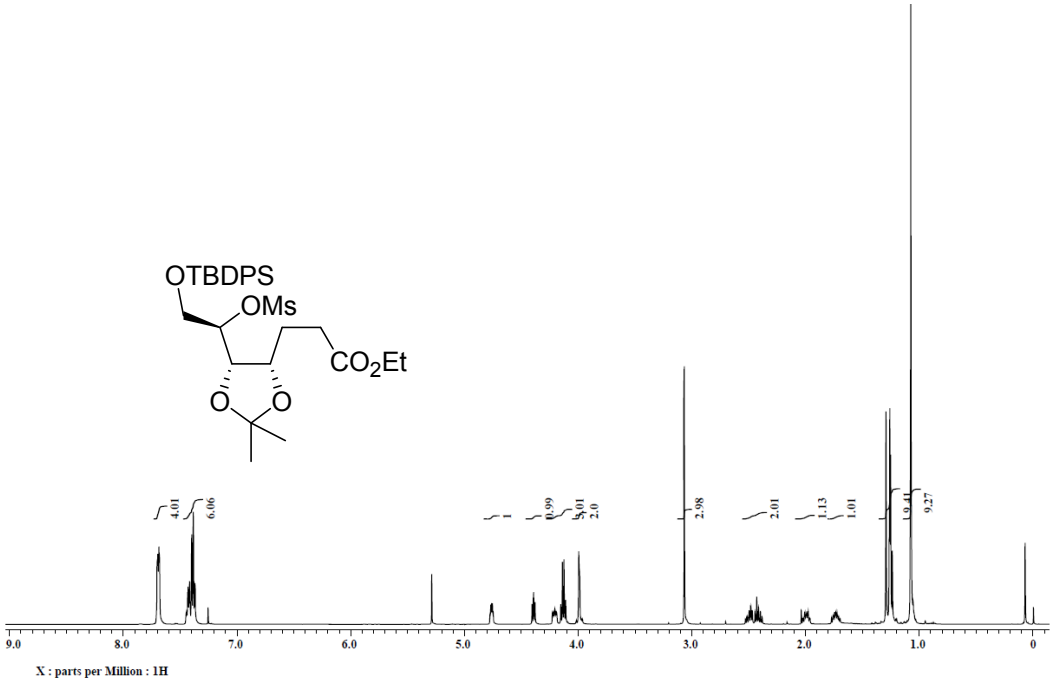


^{1}H NMR (500 MHz, $CDCl_3$)

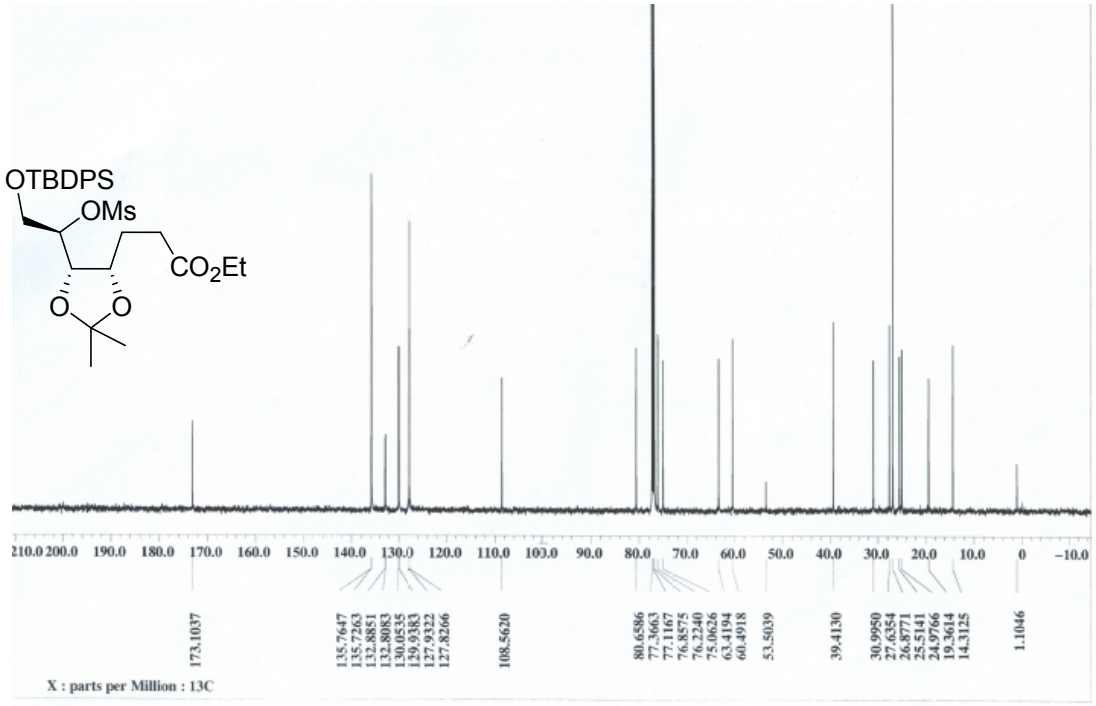


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 20

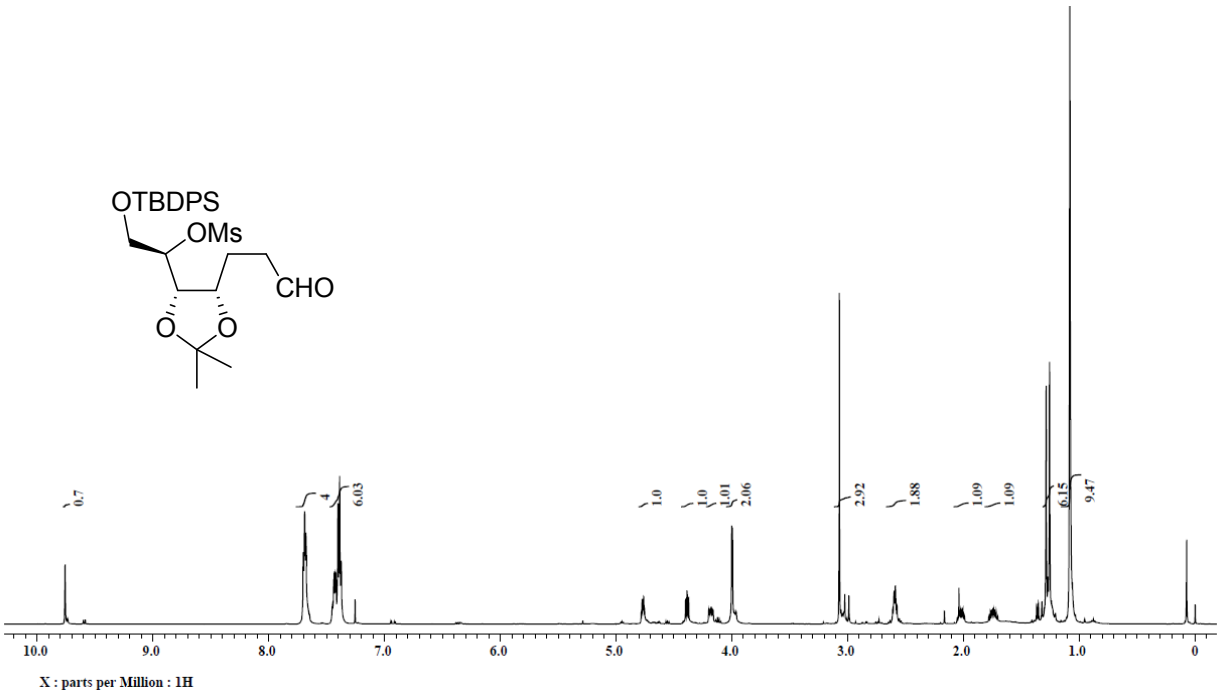


^{1}H NMR (500 MHz, $CDCl_3$)

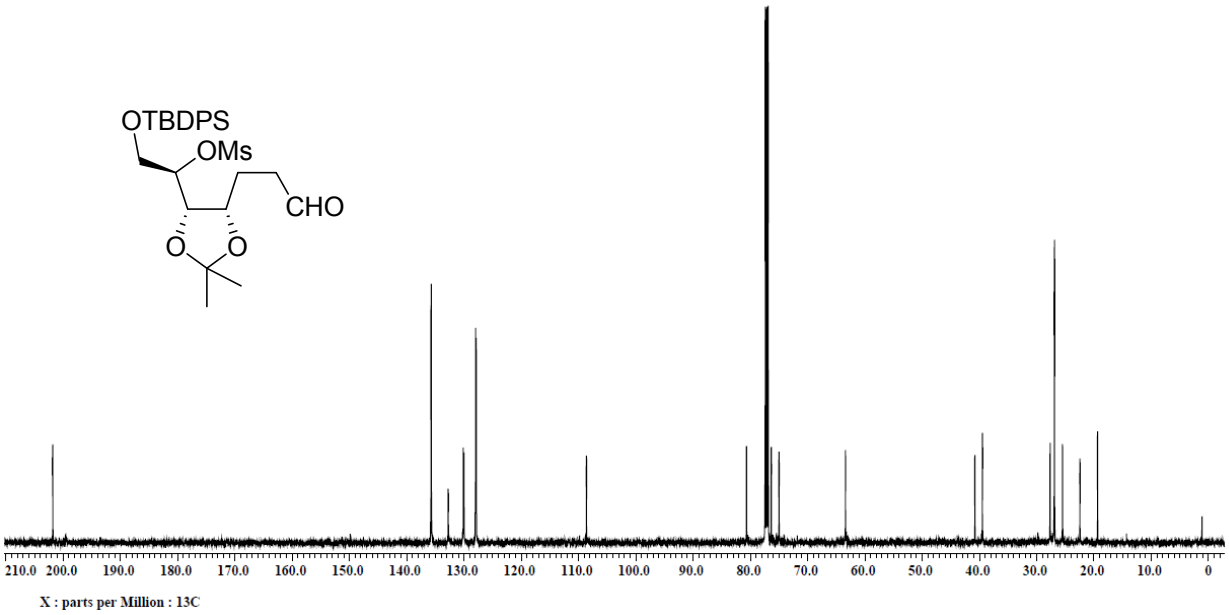


^{13}C NMR (125 MHz, $CDCl_3$)

Compound21

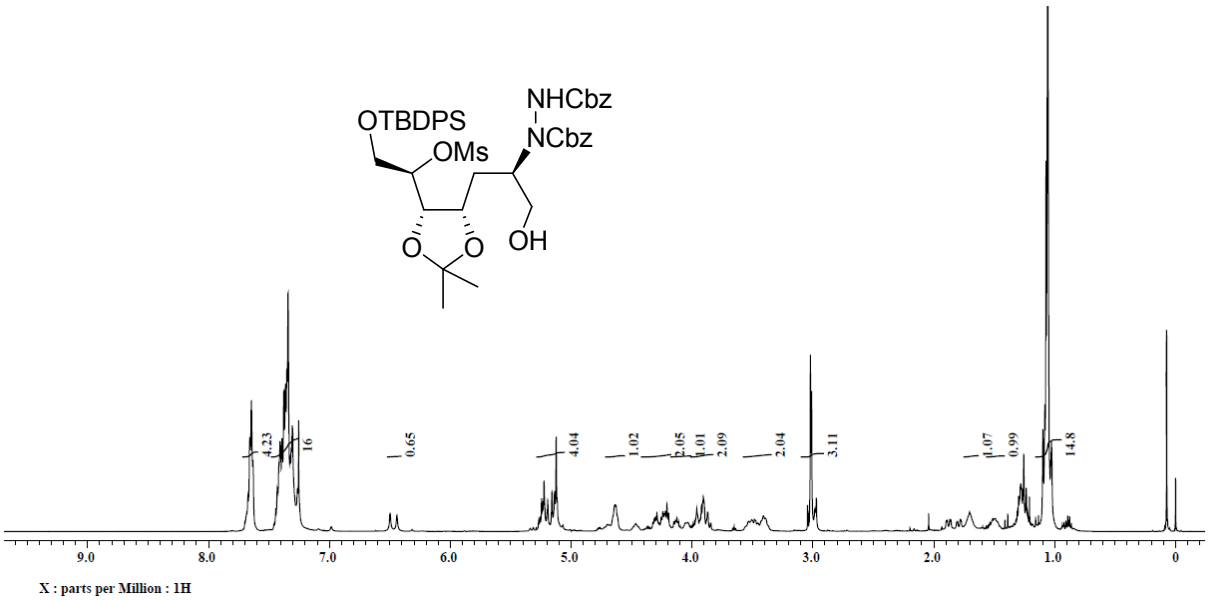


^{1}H NMR (500 MHz, $CDCl_3$)

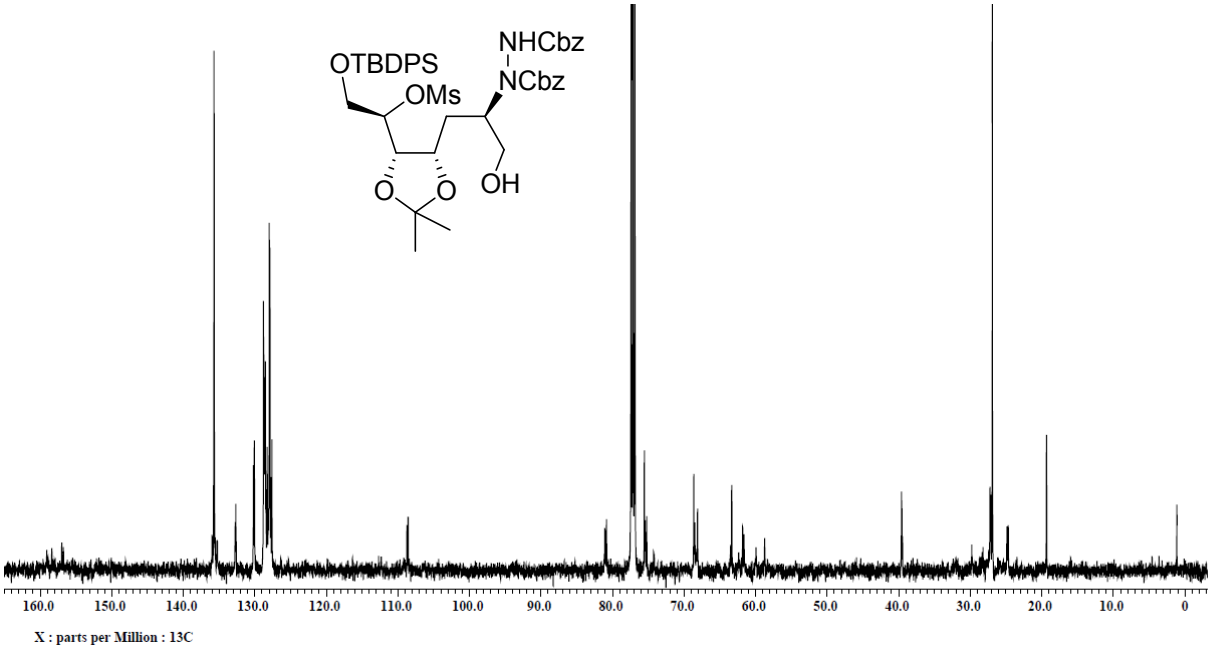


^{13}C NMR (125 MHz, $CDCl_3$)

Compound2a

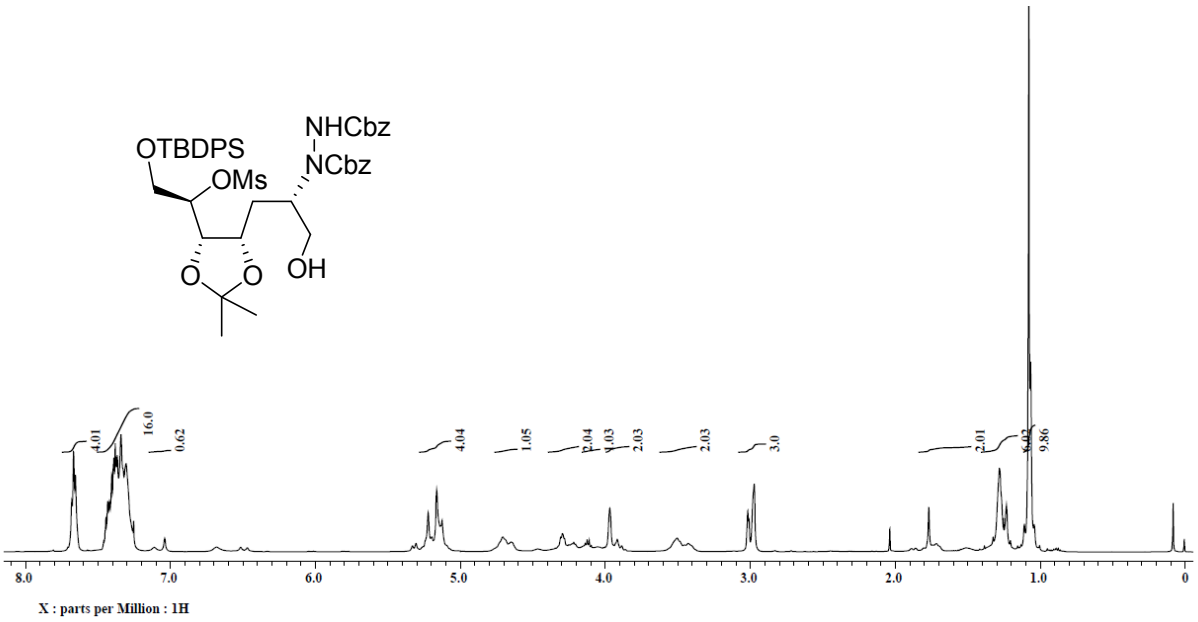


^{1}H NMR (500 MHz, $CDCl_3$)

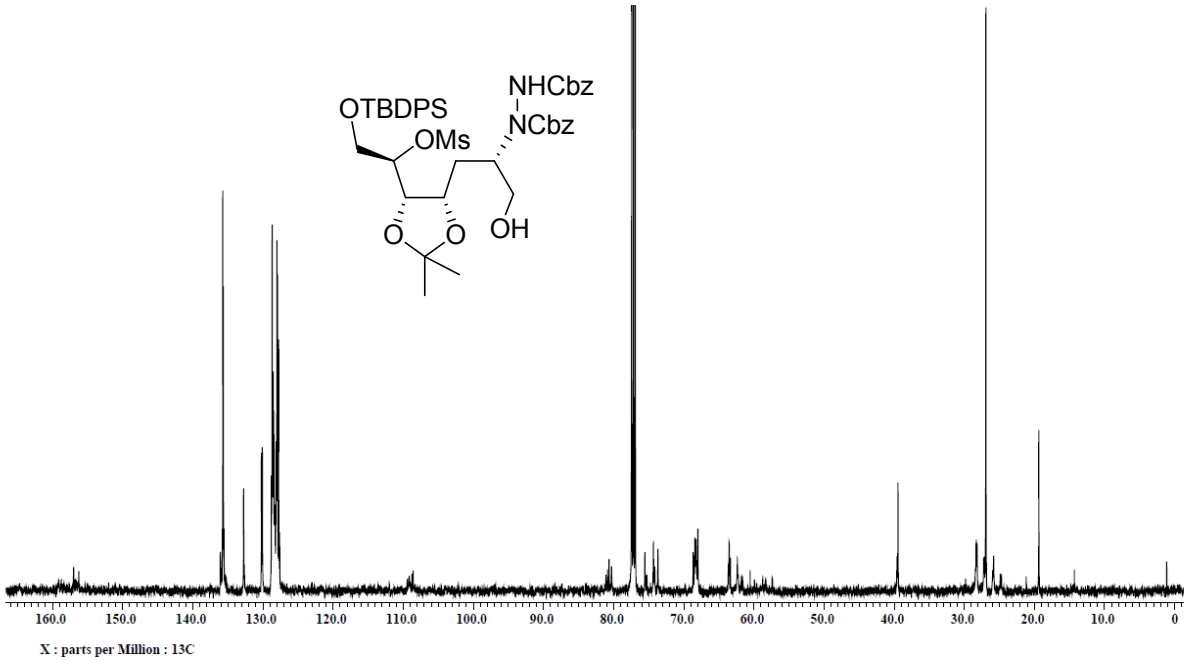


^{13}C NMR (125 MHz, $CDCl_3$) spectrum of compound **22a**

Compound 22b

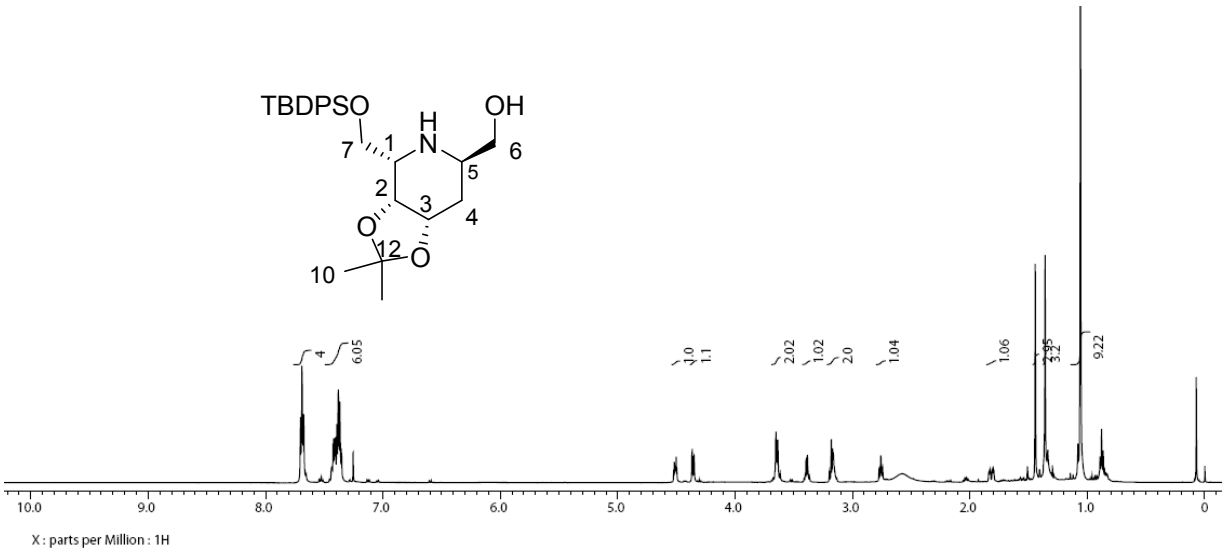


^{1}H NMR (500 MHz, $CDCl_3$)

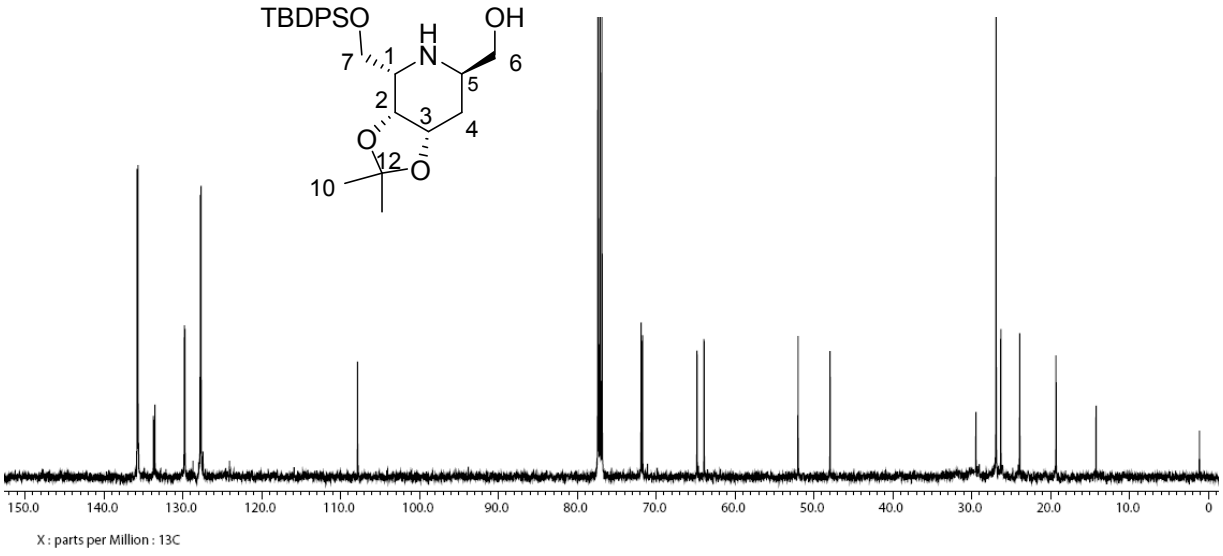


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 23a

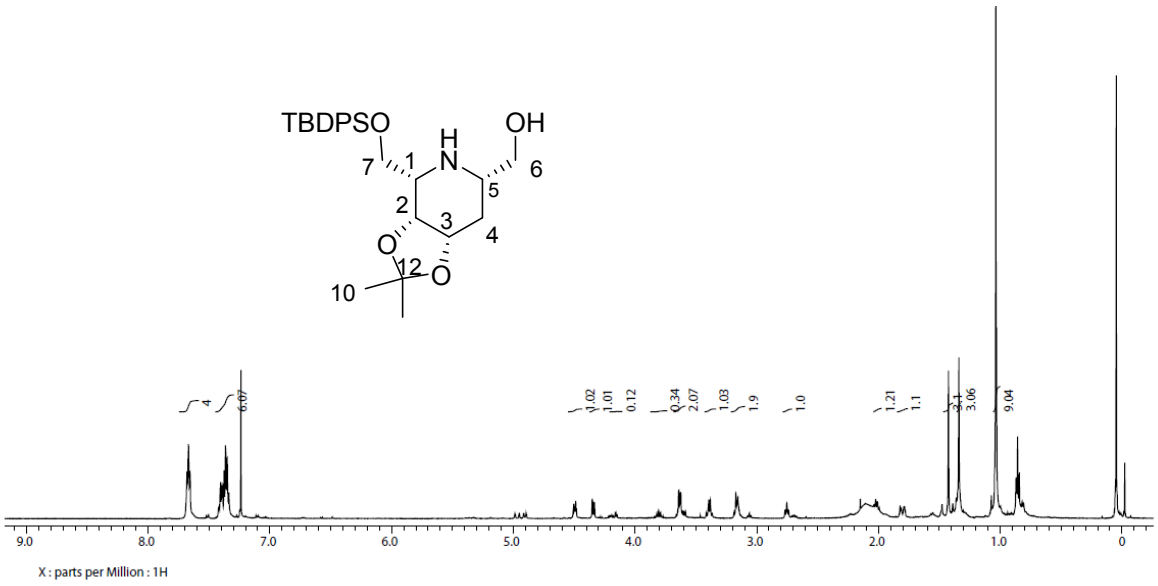


^{1}H NMR (500 MHz, $CDCl_3$)

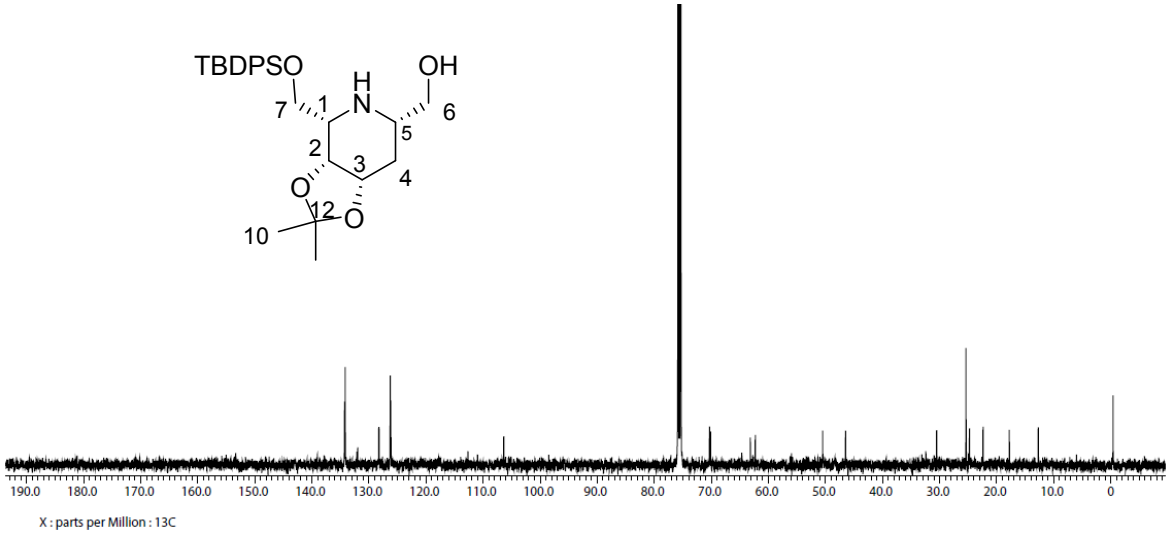


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 23b

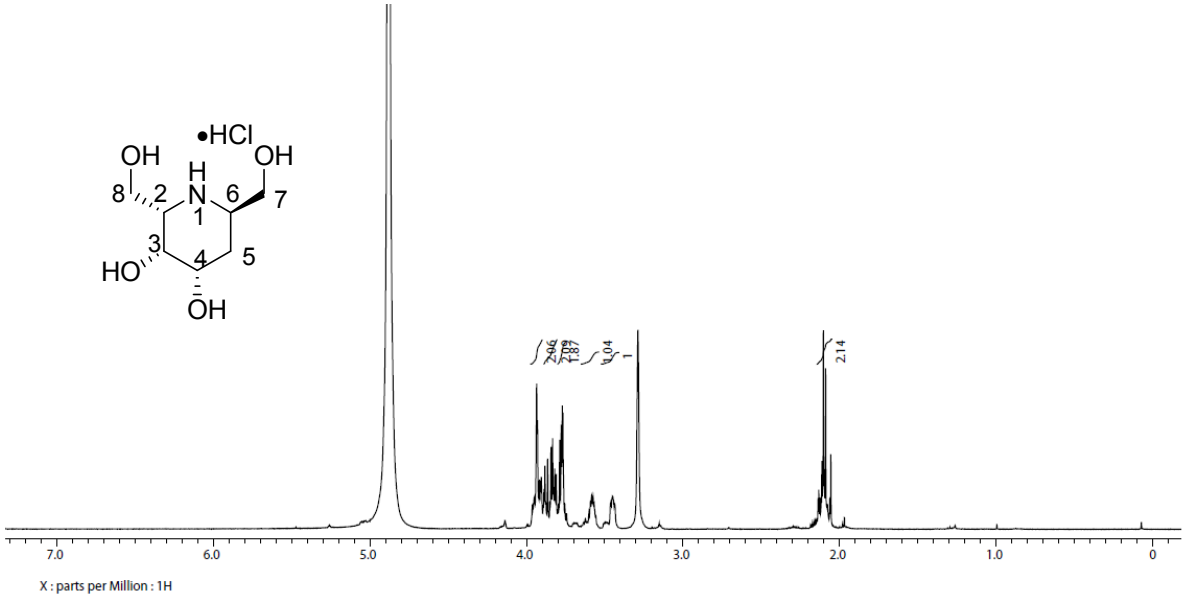


^{1}H NMR (500 MHz, $CDCl_3$)

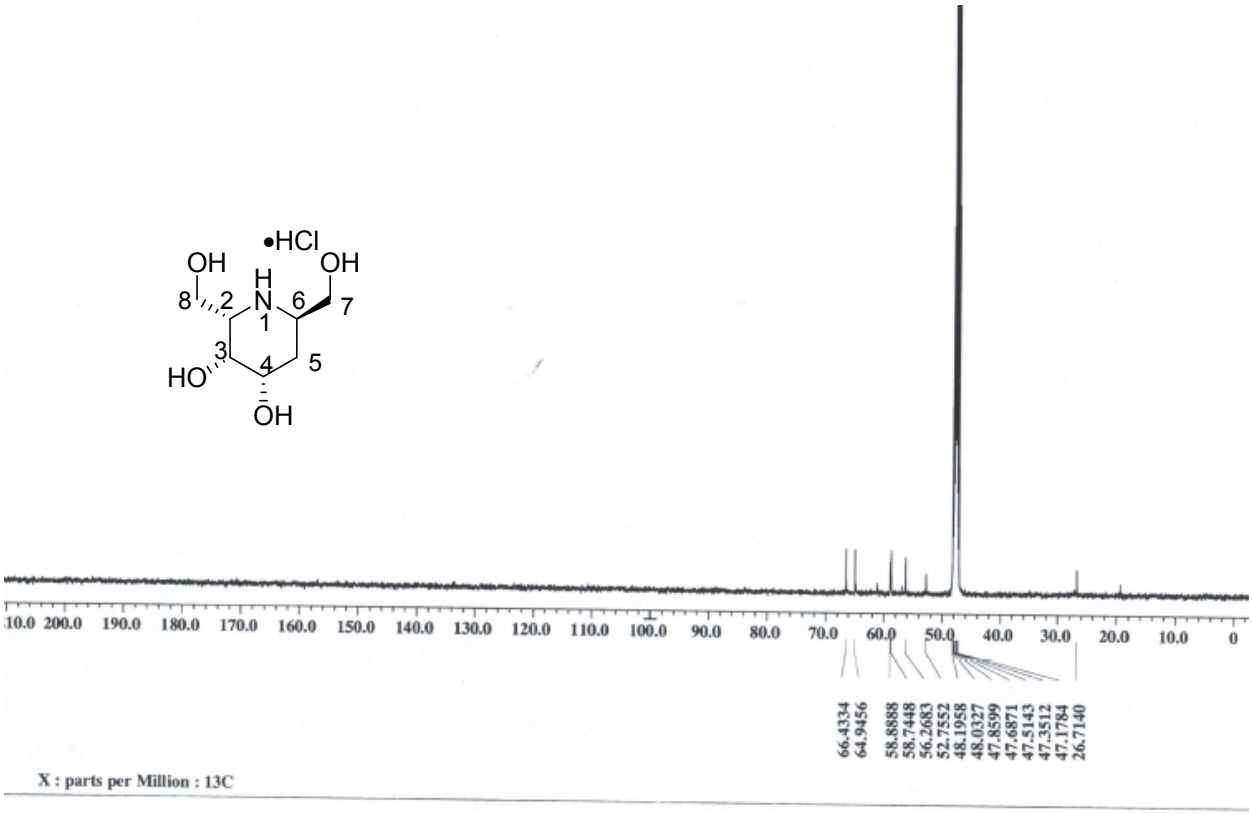


^{13}C NMR (125 MHz, $CDCl_3$)

Compound 3a

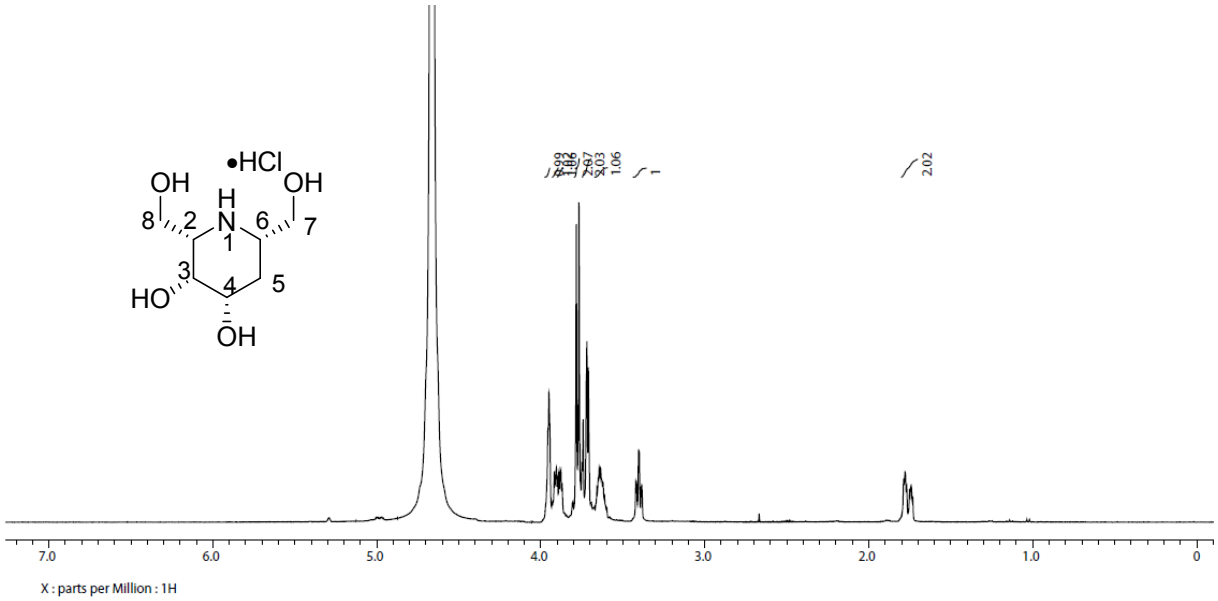


^{1}H NMR (500 MHz, CD_3OD)

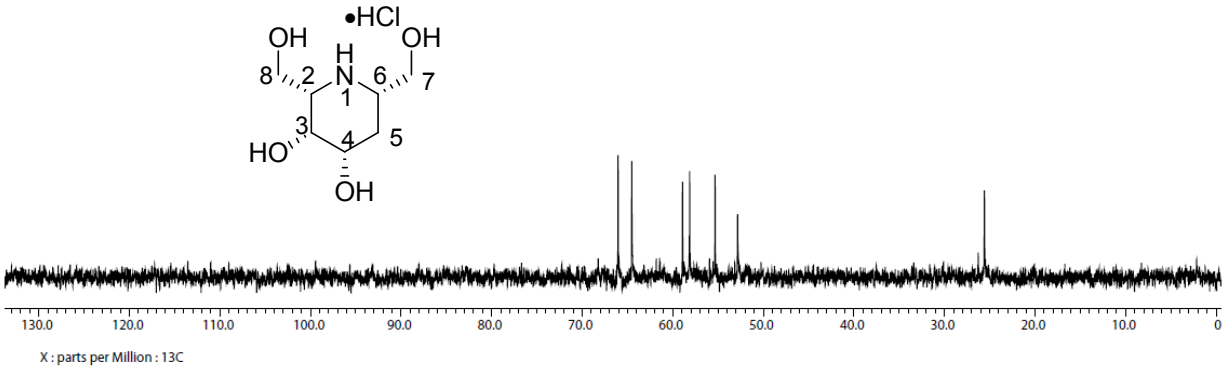


^{13}C NMR (125 MHz, CD_3OD)

Compound 3b

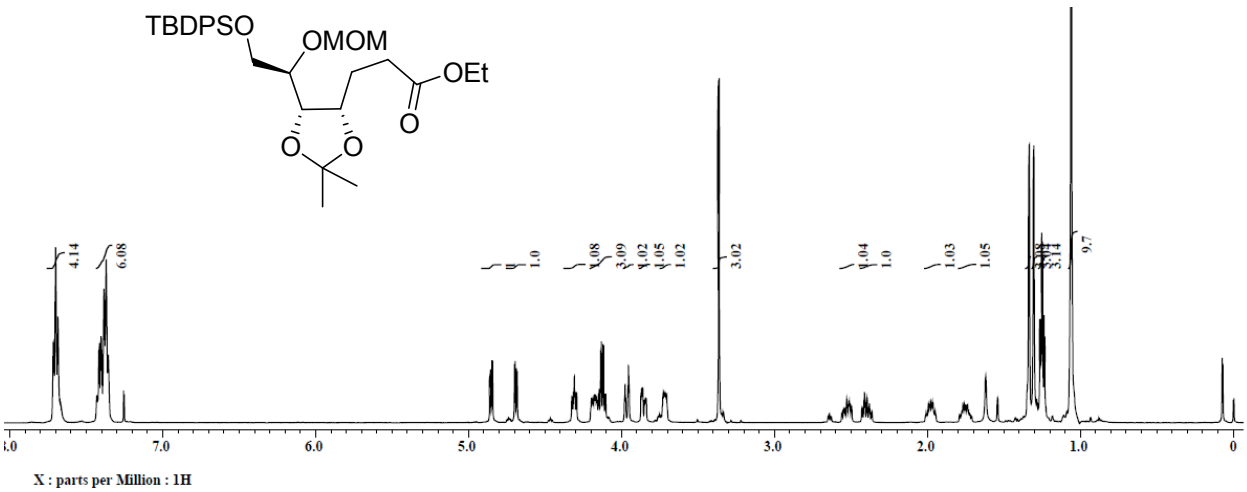


^{1}H NMR (500 MHz, D_2O) spectrum of

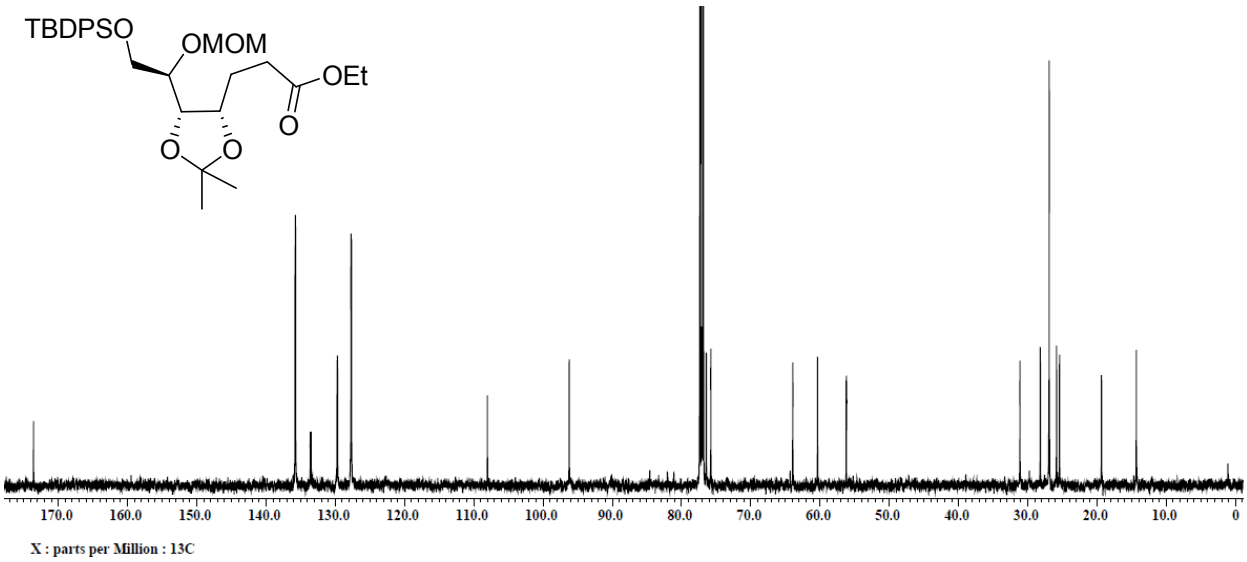


^{13}C NMR (125 MHz, D_2O)

Compound24

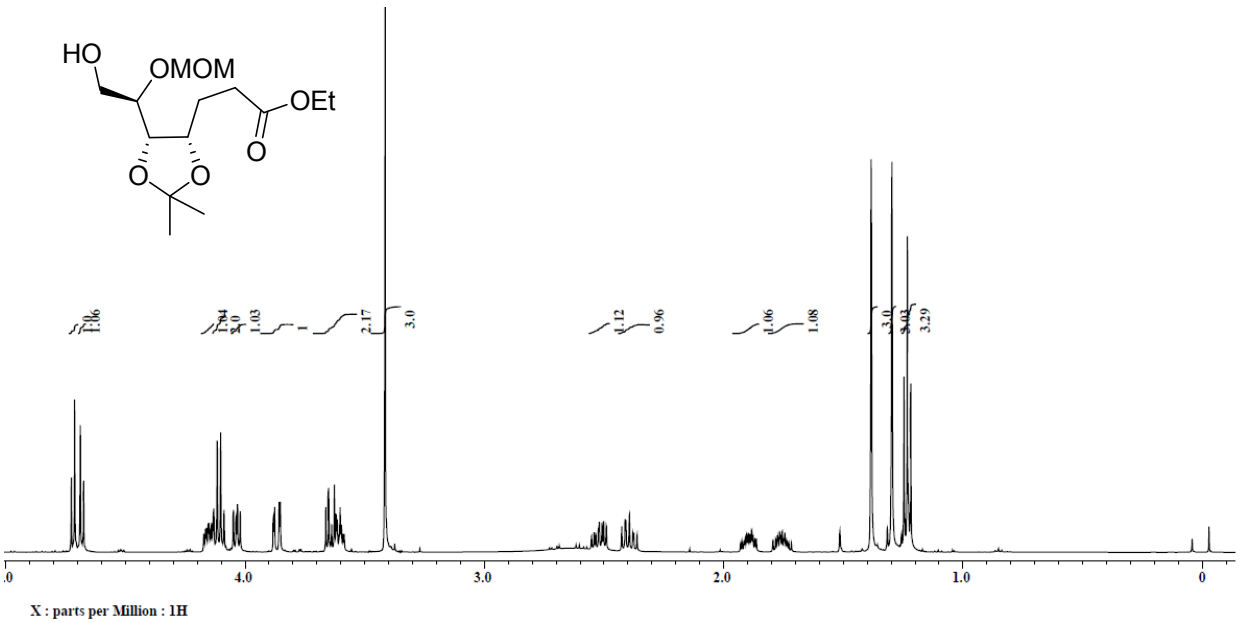


^{1}H NMR (500 MHz, $CDCl_3$)

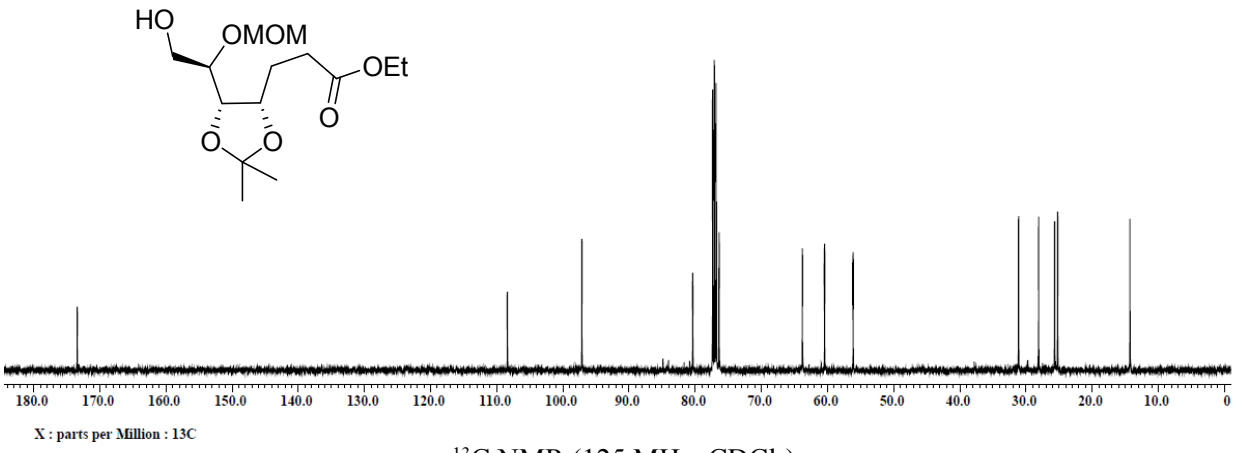


^{13}C NMR (125 MHz, $CDCl_3$)

Compound25

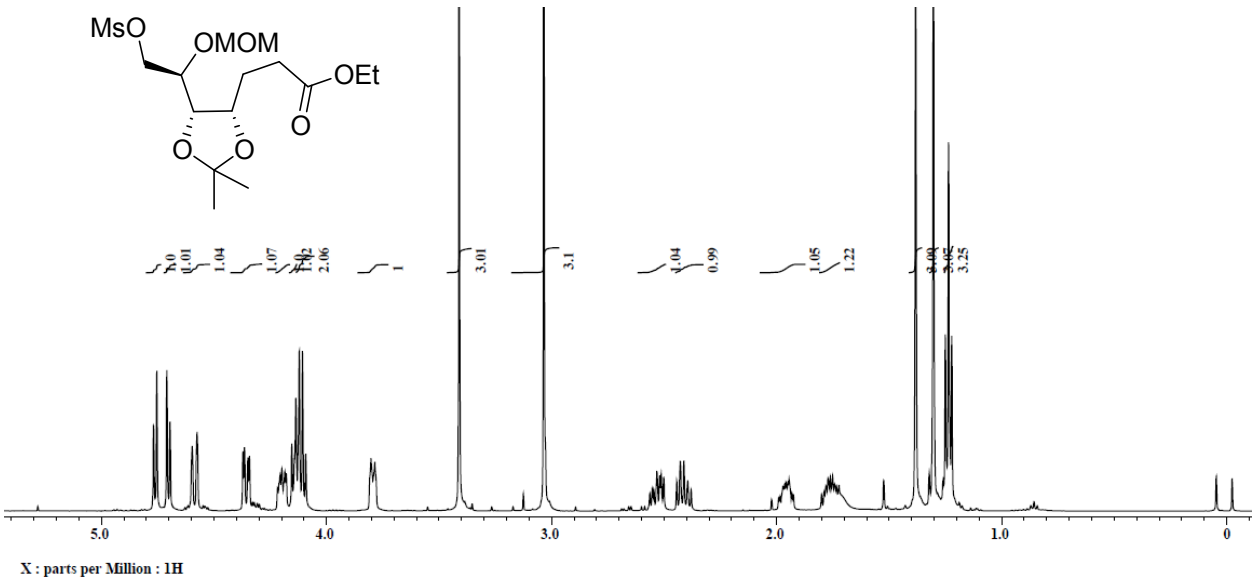


^{1}H NMR (500 MHz, $CDCl_3$)

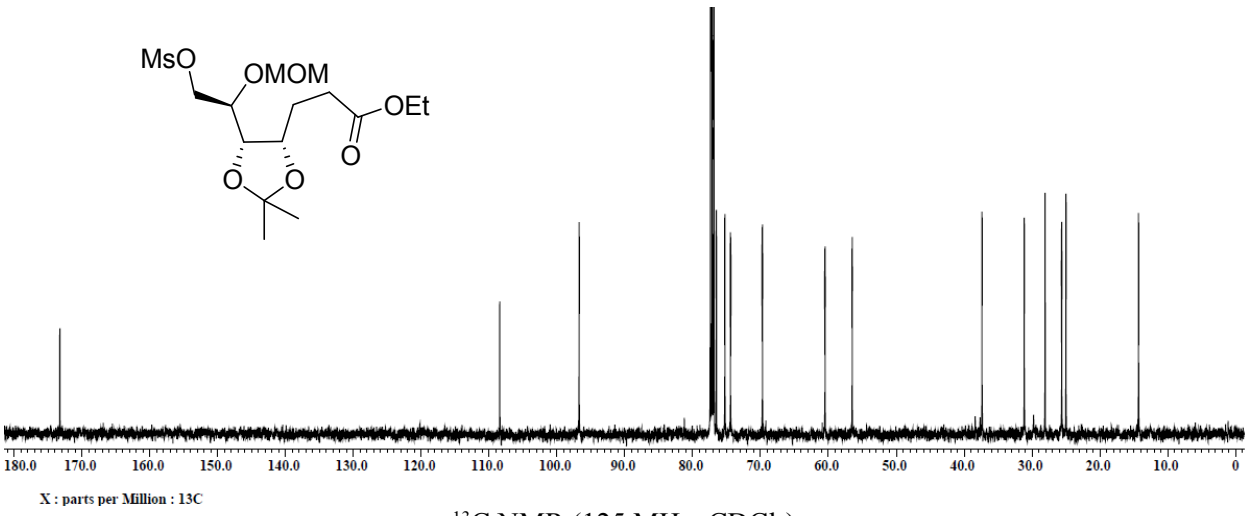


^{13}C NMR (125 MHz, $CDCl_3$)

Compound26

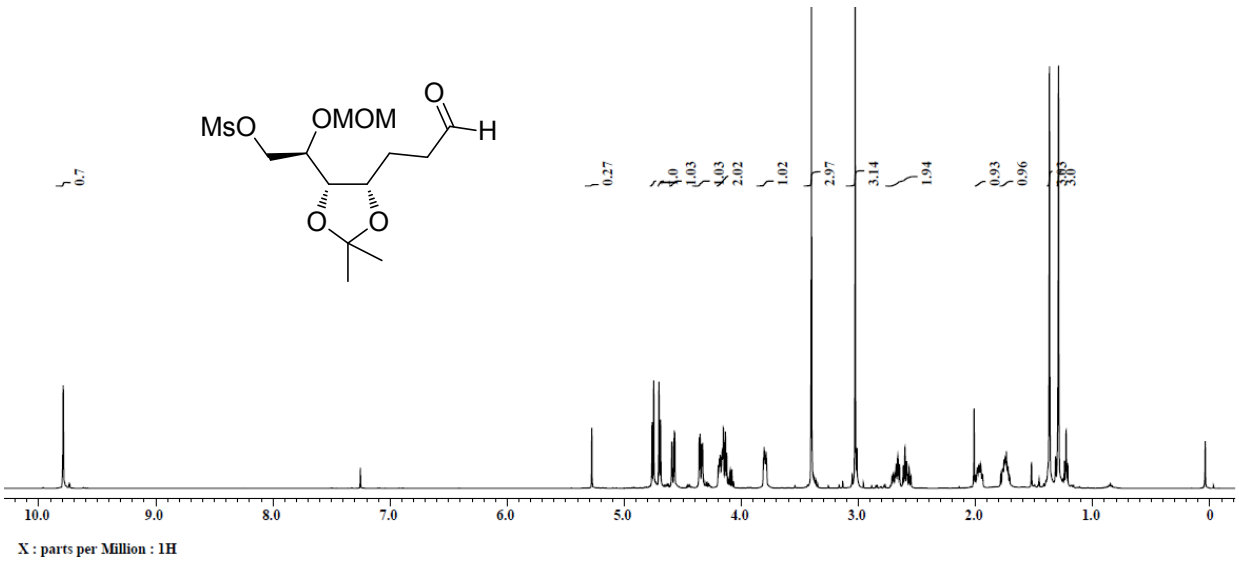


^{1}H NMR (500 MHz, $CDCl_3$)

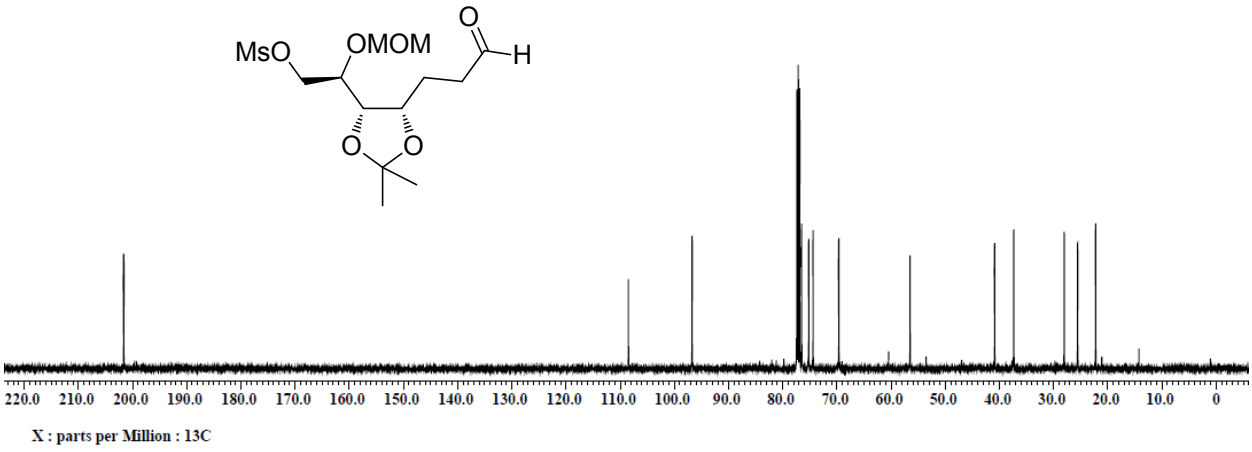


^{13}C NMR (125 MHz, $CDCl_3$)

Compound27

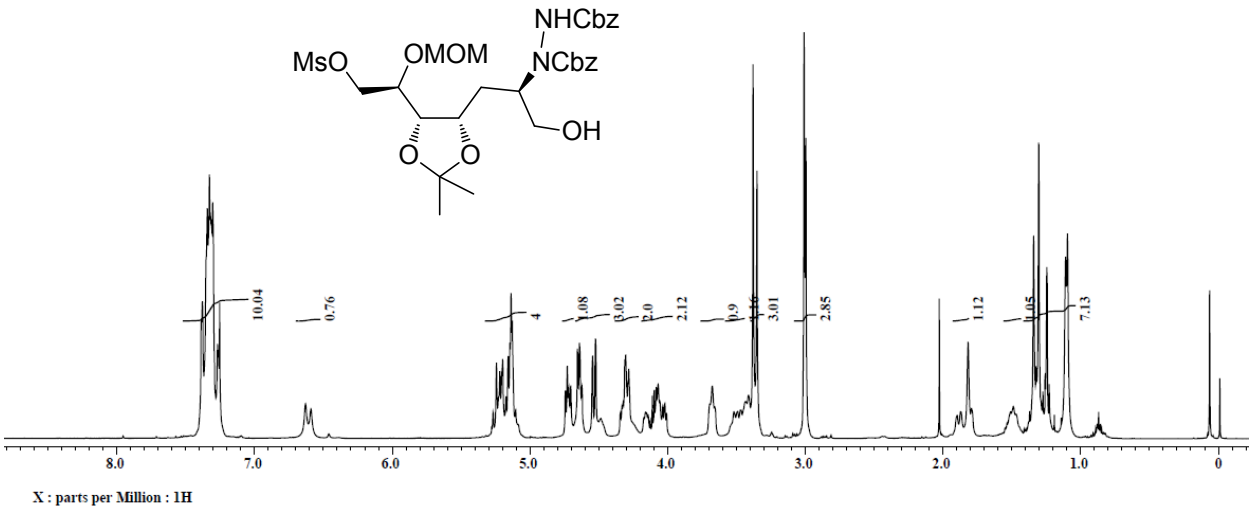


^{1}H NMR (500 MHz, $CDCl_3$)

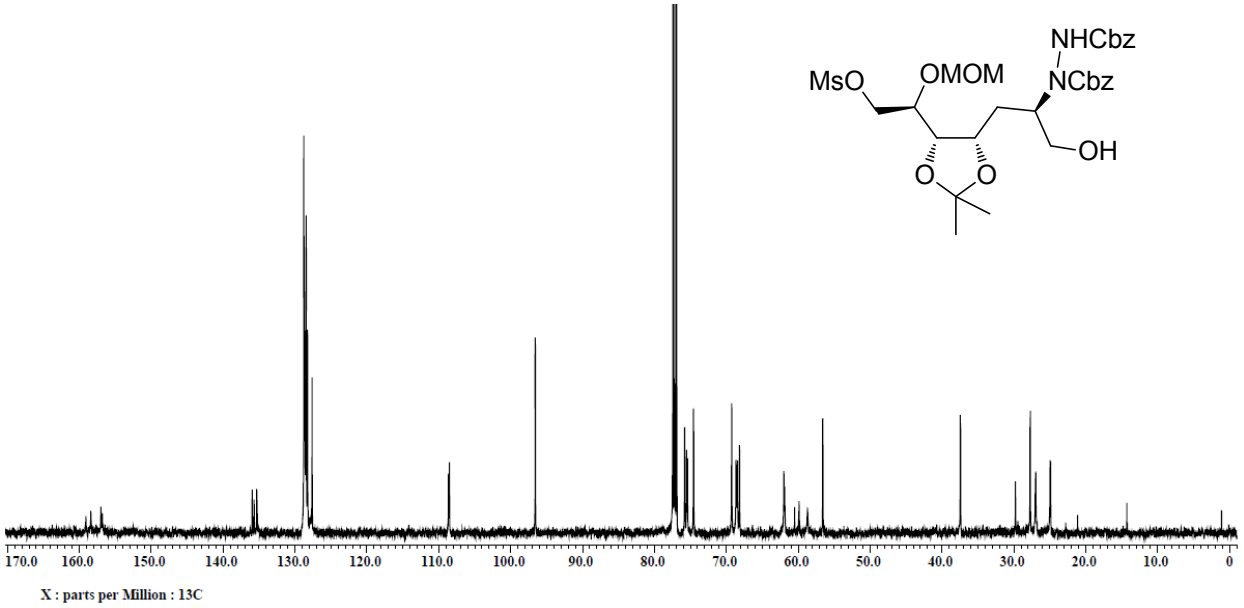


^{13}C NMR (125 MHz, $CDCl_3$)

Compound28a

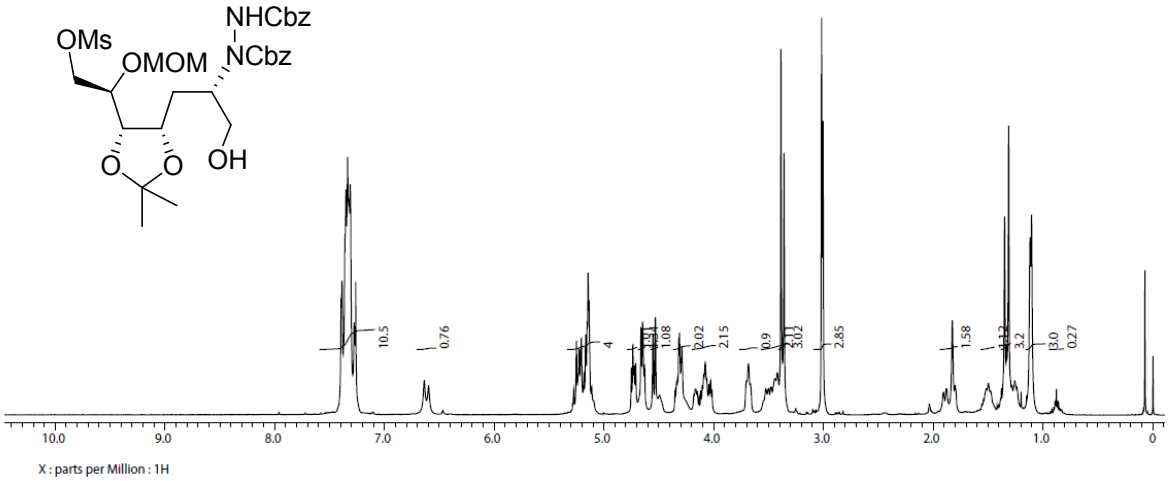


^{1}H NMR (500 MHz, $CDCl_3$)

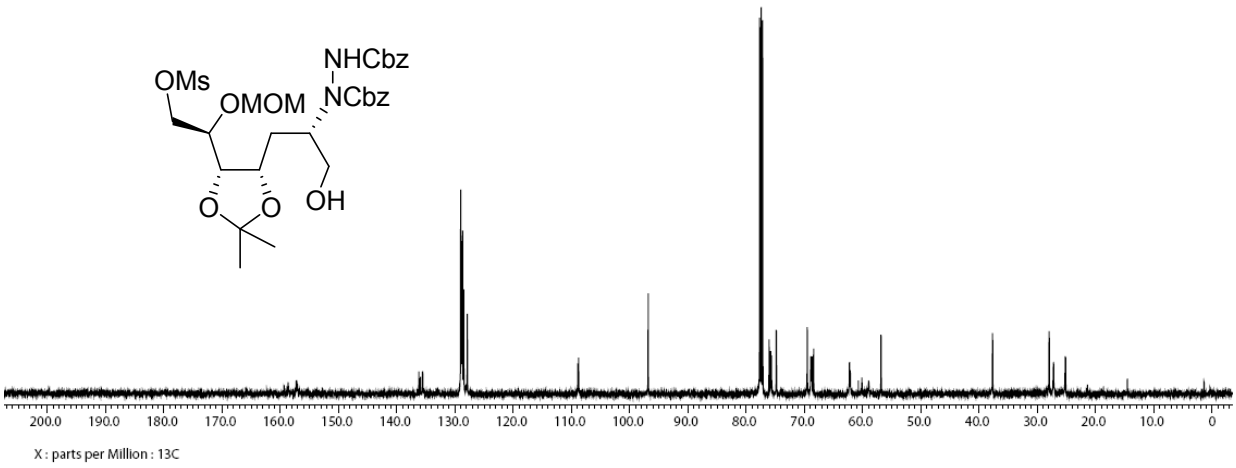


^{13}C NMR (125 MHz, $CDCl_3$)

Compound28b

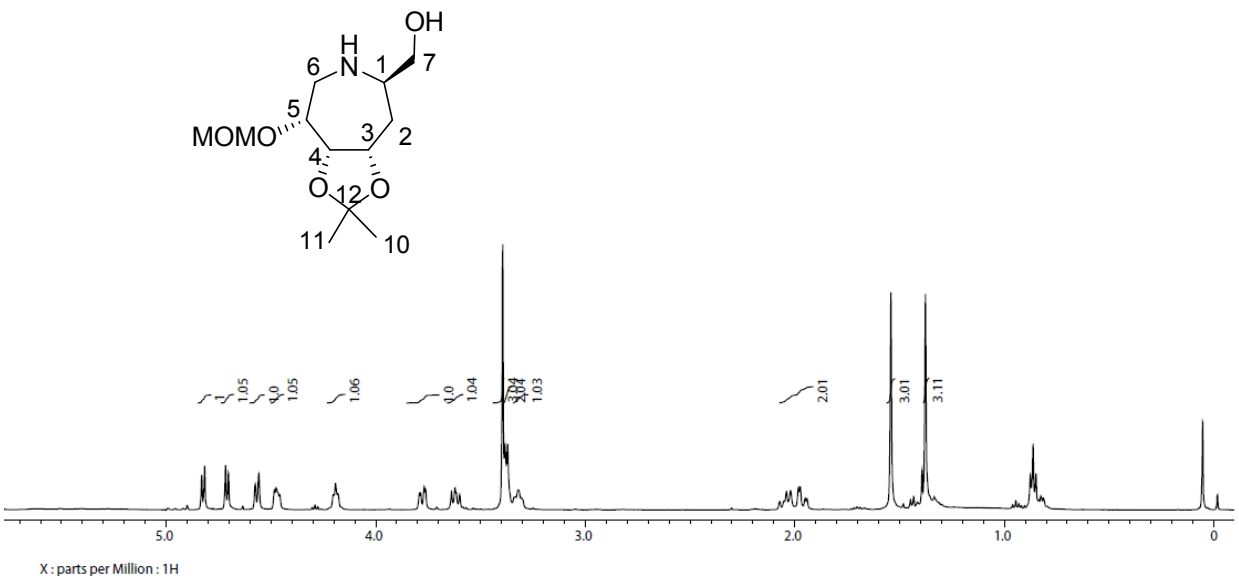


^{1}H NMR (500 MHz, $CDCl_3$)

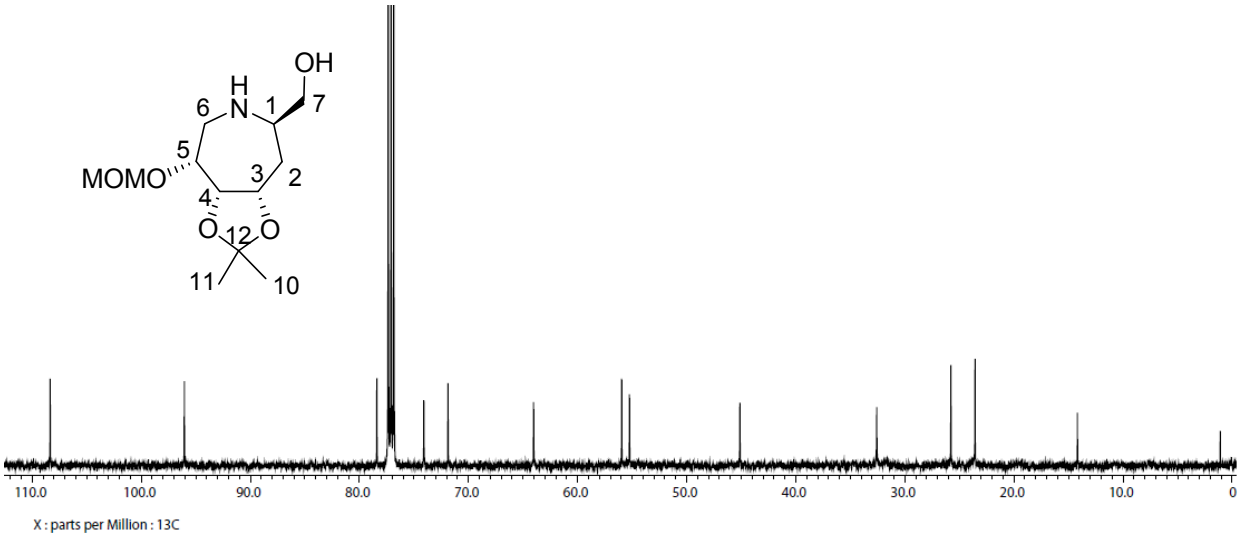


^{13}C NMR (125 MHz, $CDCl_3$)

Compound29a

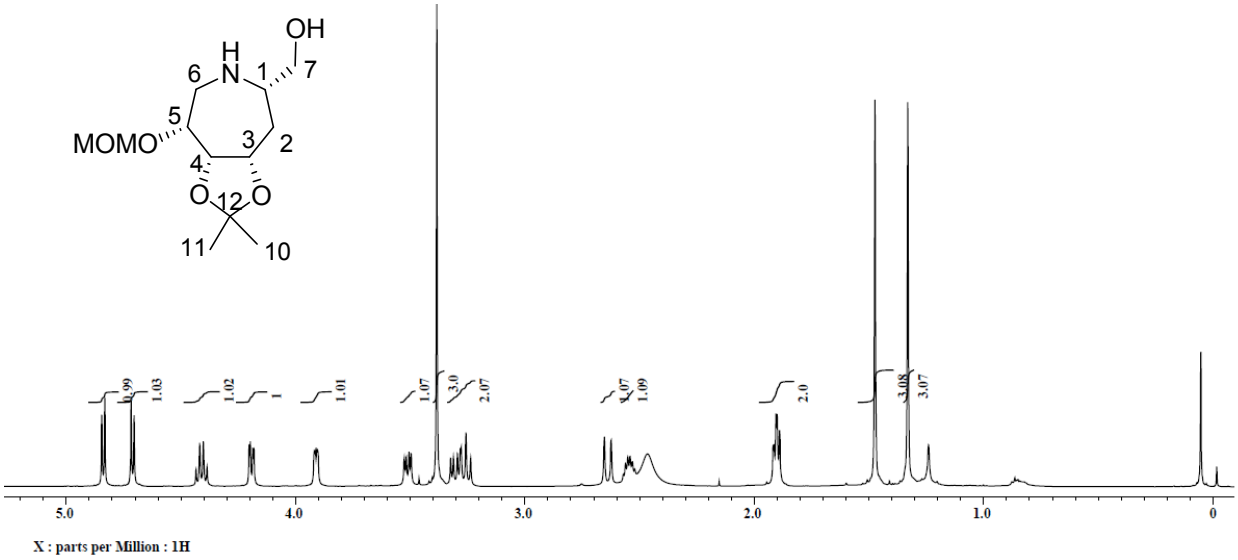


^{1}H NMR (500 MHz, $CDCl_3$)

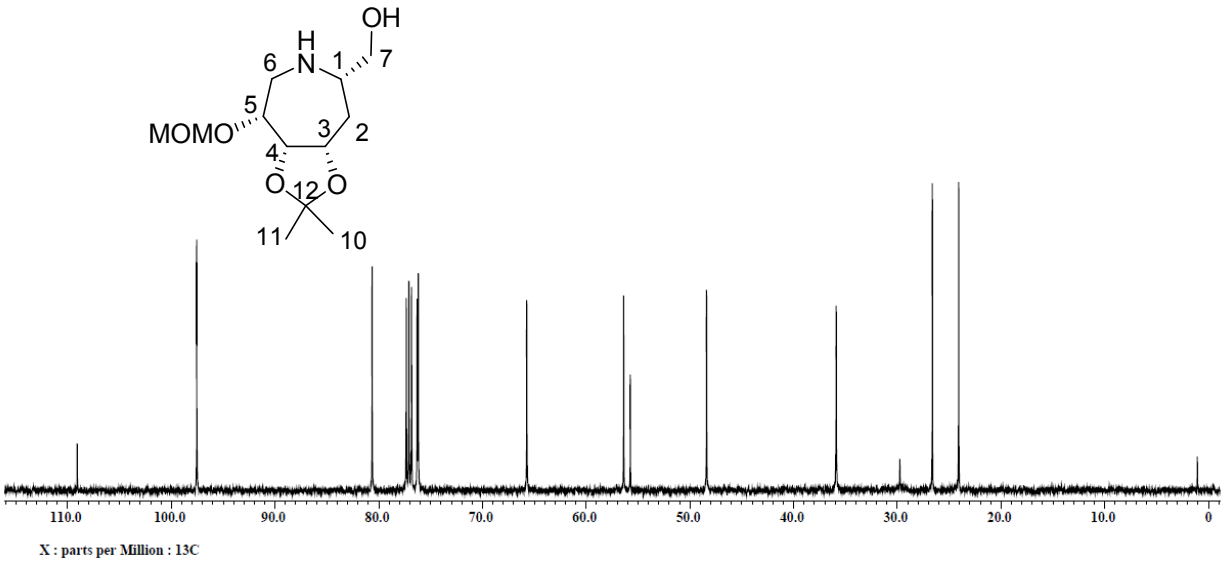


^{13}C NMR (125 MHz, $CDCl_3$)

Compound29b

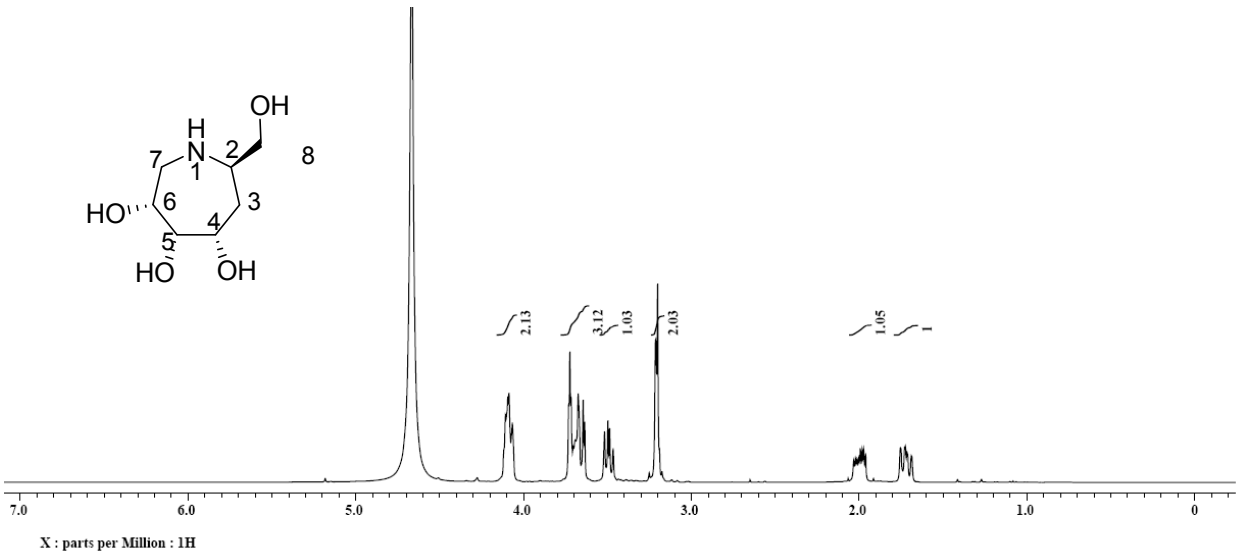


^{1}H NMR (500 MHz, $CDCl_3$)

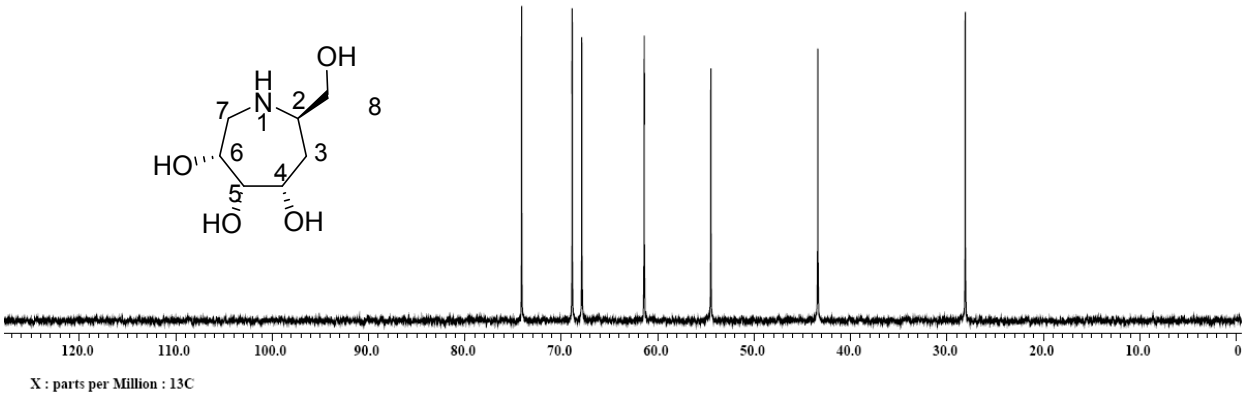


^{13}C NMR (125 MHz, $CDCl_3$)

Compound4a

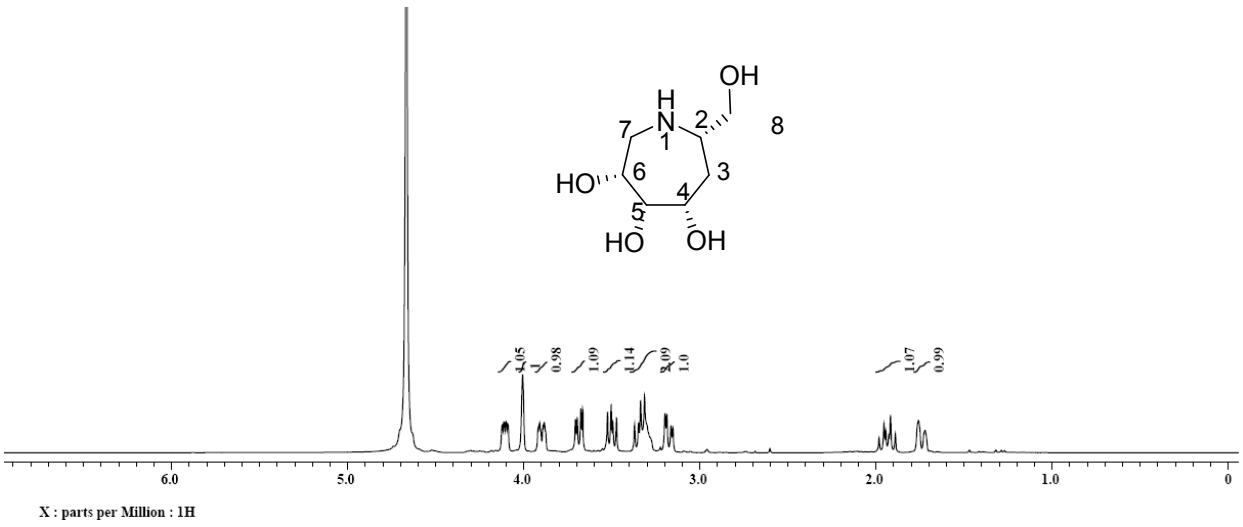


^{1}H NMR (400 MHz, D_2O) spectrum of

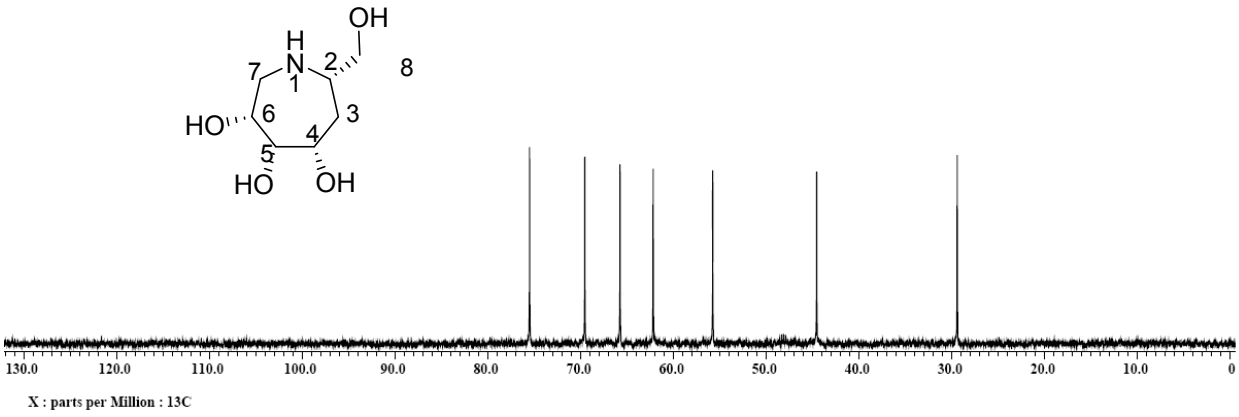


^{13}C NMR (100 MHz, D_2O) spectrum of compound**4a**

Compound4b



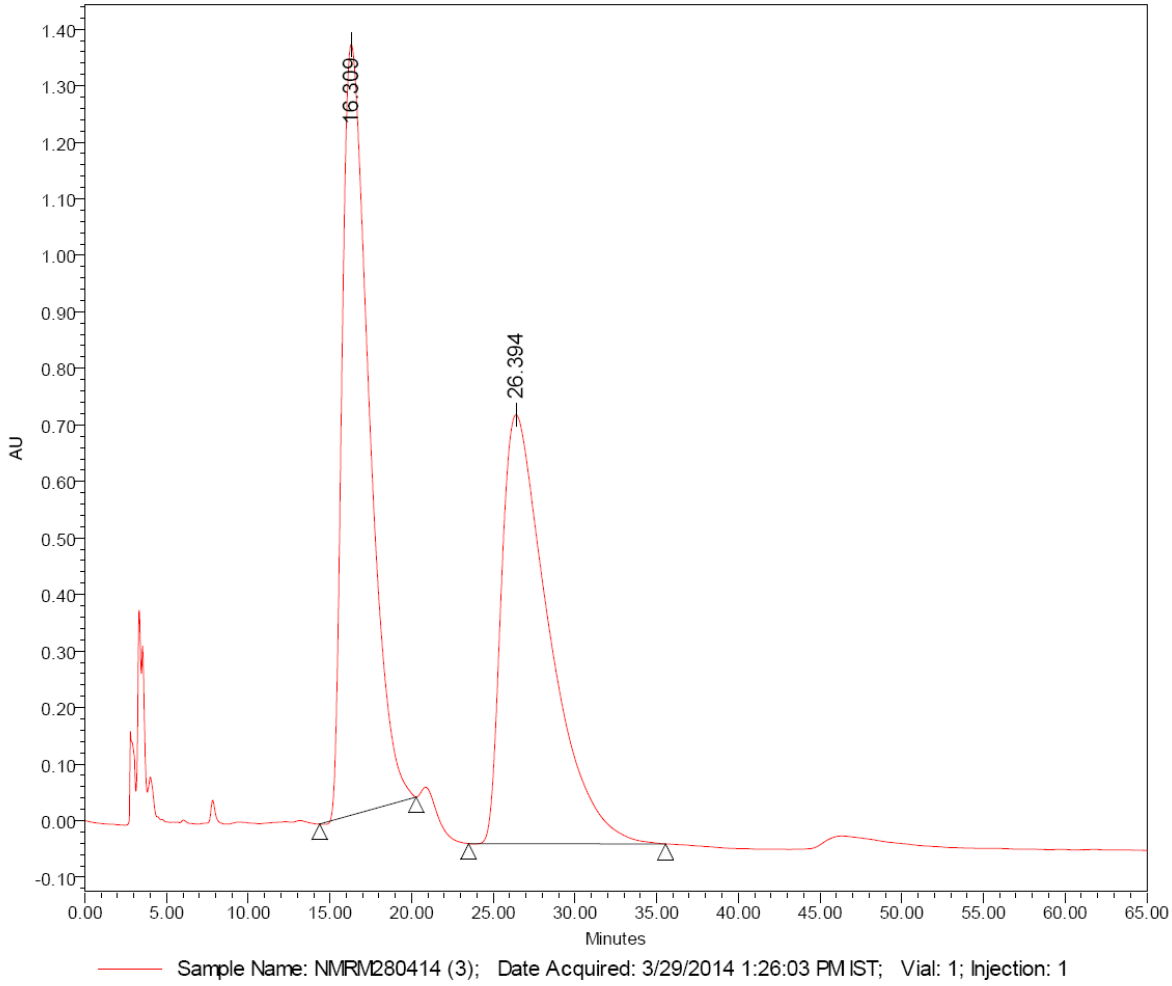
^{1}H NMR (400 MHz, D_2O)



^{13}C NMR (100 MHz, D_2O)

HPLC DATA

HPLC data of compound **10** (racemic)



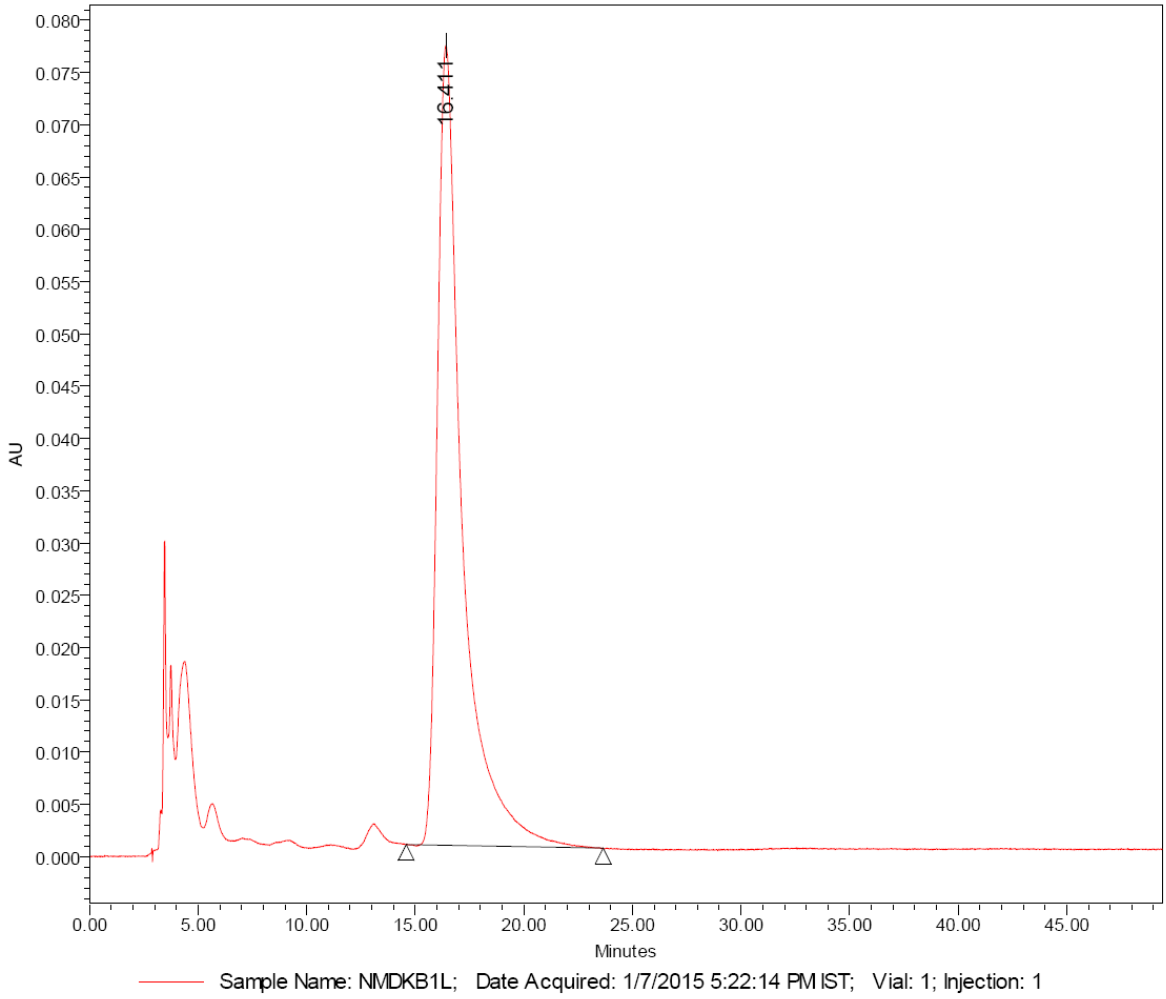
Sample Name: NMRM280414 (3); Date Acquired: 3/29/2014 1:26:03 PM IST; Vial: 1; Injection: 1

Peak Summary with Statistics

Name:

	Sample Name	Vial	Inj	Retention Time (min)	Area	% Area	Height
1	NMRM280414 (3)	1	1	16.309	156739236	50.54	1363075
2	NMRM280414 (3)	1	1	26.394	153402386	49.46	758672
Mean				21.351			
Std. Dev.				7.131			

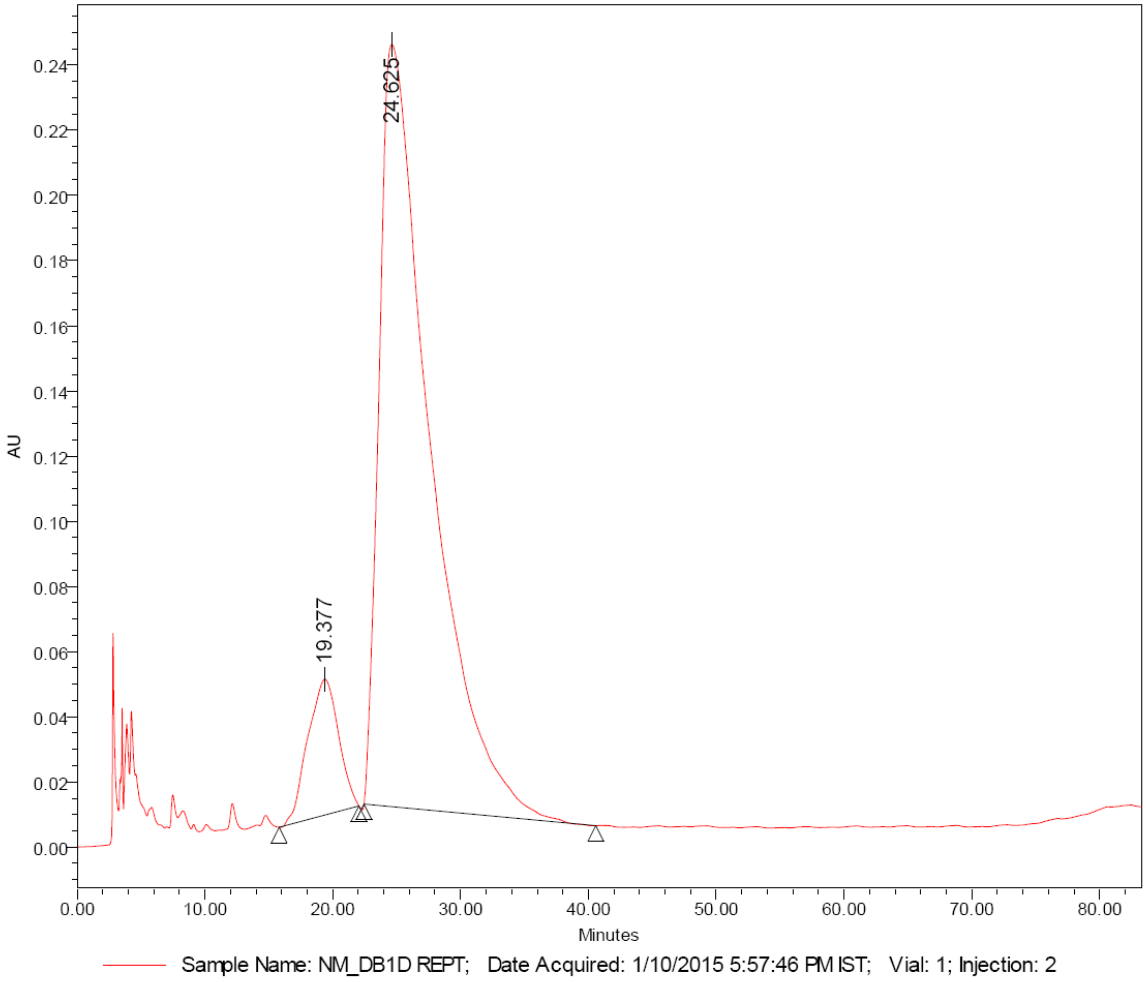
HPLC data of compound **10a**



Peak Summary with Statistics
Name:

	Sample Name	Vial	Inj	Retention Time (min)	Area	% Area	Height
1	NMDKB1L	1	1	16.411	6098992	100.00	76505
Mean				16.411			
Std. Dev.							
% RSD							

HPLC data of compound **10b**



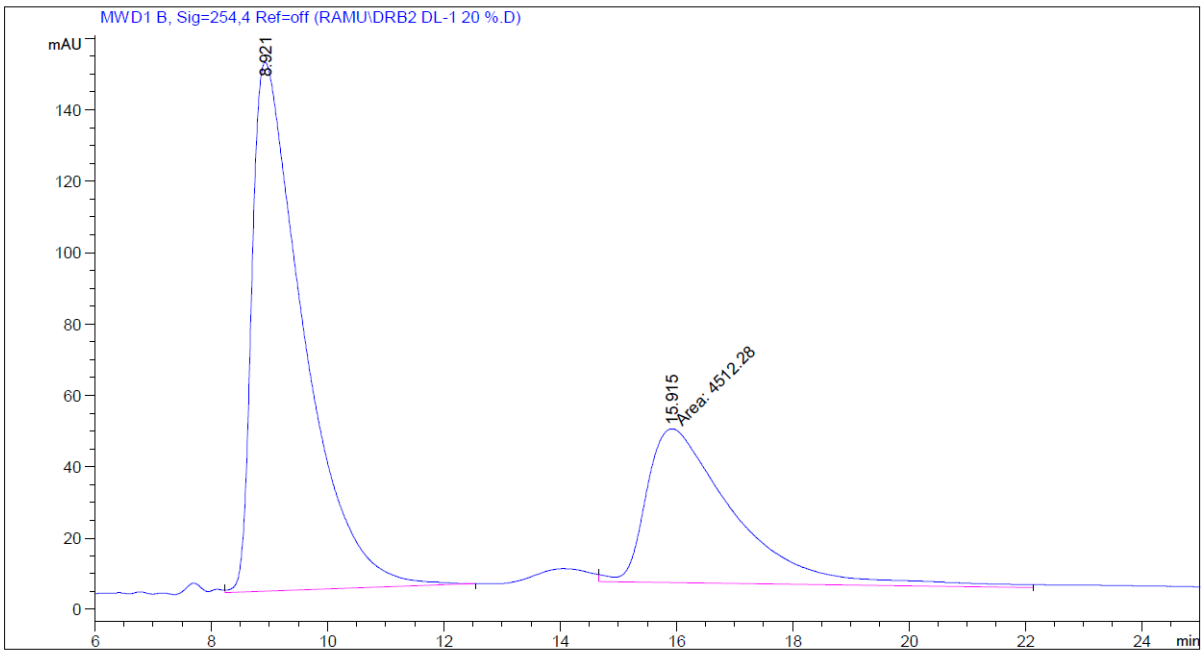
Sample Name: NM_DB1D REPT; Date Acquired: 1/10/2015 5:57:46 PM IST; Vial: 1; Injection: 2

Peak Summary with Statistics
Name:

	Sample Name	Vial	Inj	Retention Time (min)	Area	% Area	Height
1	NM_DB1D REPT	1	2	24.625	64944502	90.28	233910
2	NM_DB1D REPT	1	2	19.377	6990878	9.72	41712
Mean				22.001			
Std. Dev.				3.711			

HPLC data of compound **17** (racemic)

Additional Info : Peak(s) manually integrated

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=====================================================================
                        Area Percent Report
=====================================================================

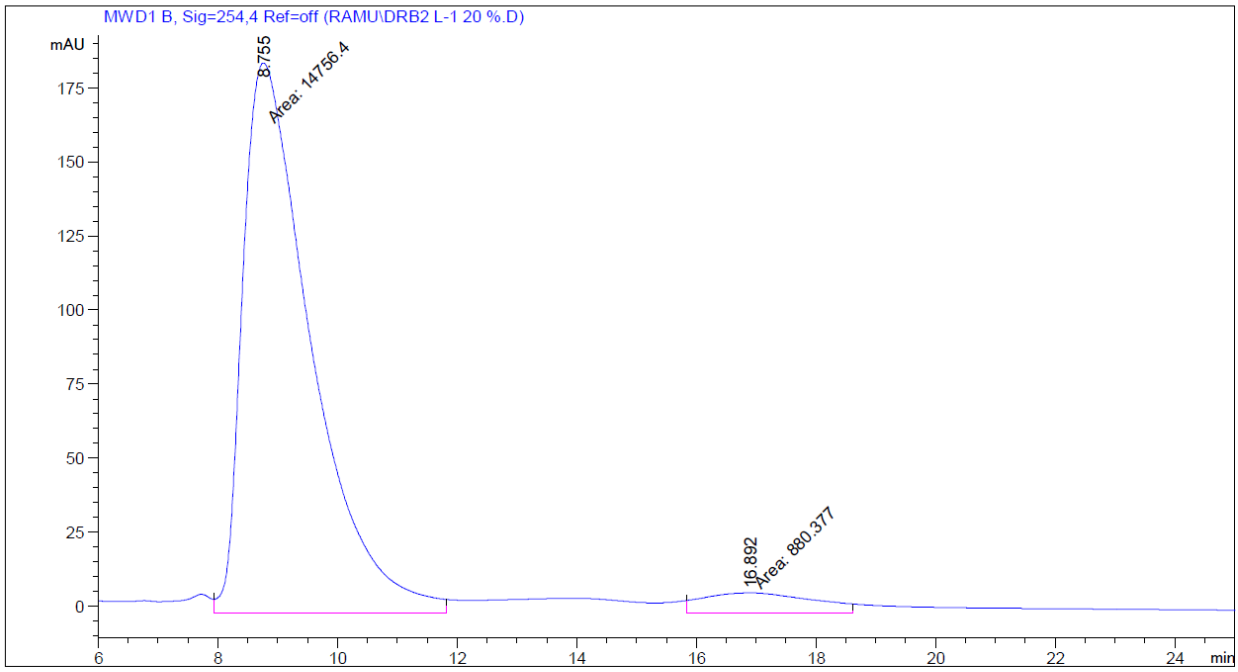
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MWD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.921 VB    0.8731 9110.36133  148.22237  66.8766
   2  15.915 MM    1.7441 4512.27734   43.12032  33.1234
```

HPLC data of compound **17a**

Additional Info : Peak(s) manually integrated



==========

Area Percent Report

==========

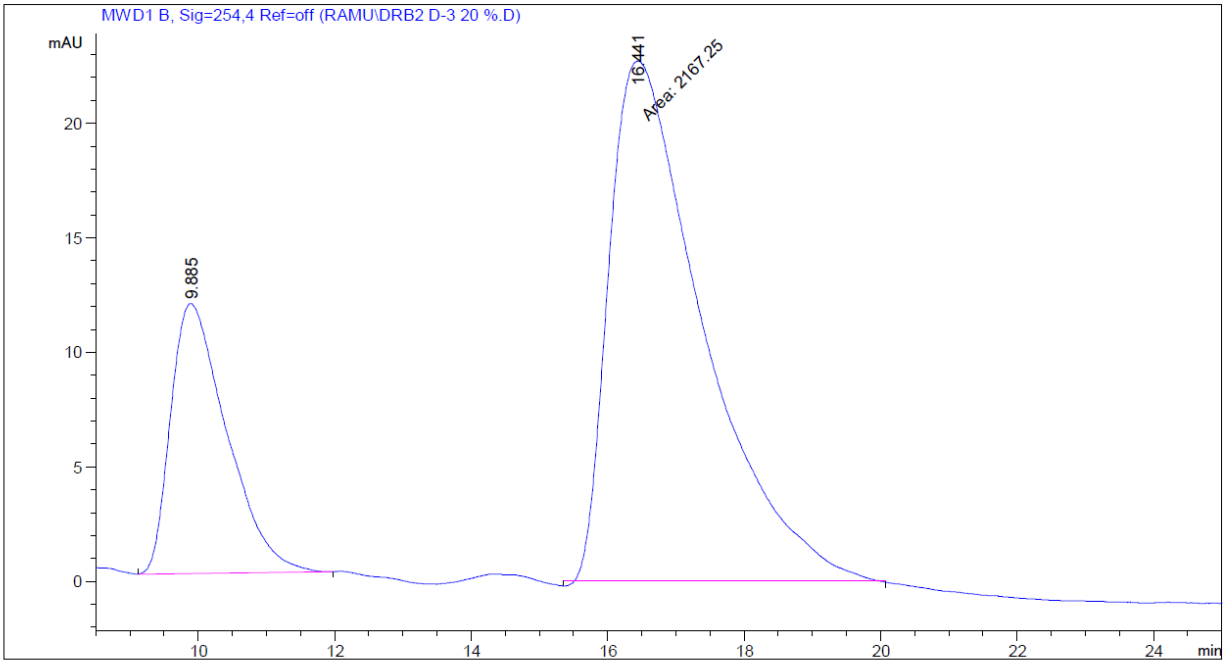
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.755	MM	1.3240	1.47564e4	185.76009	94.3698
2	16.892	MM	2.1541	880.37659	6.81151	5.6302

HPLC data of compound **17b**

Additional Info : Peak(s) manually integrated



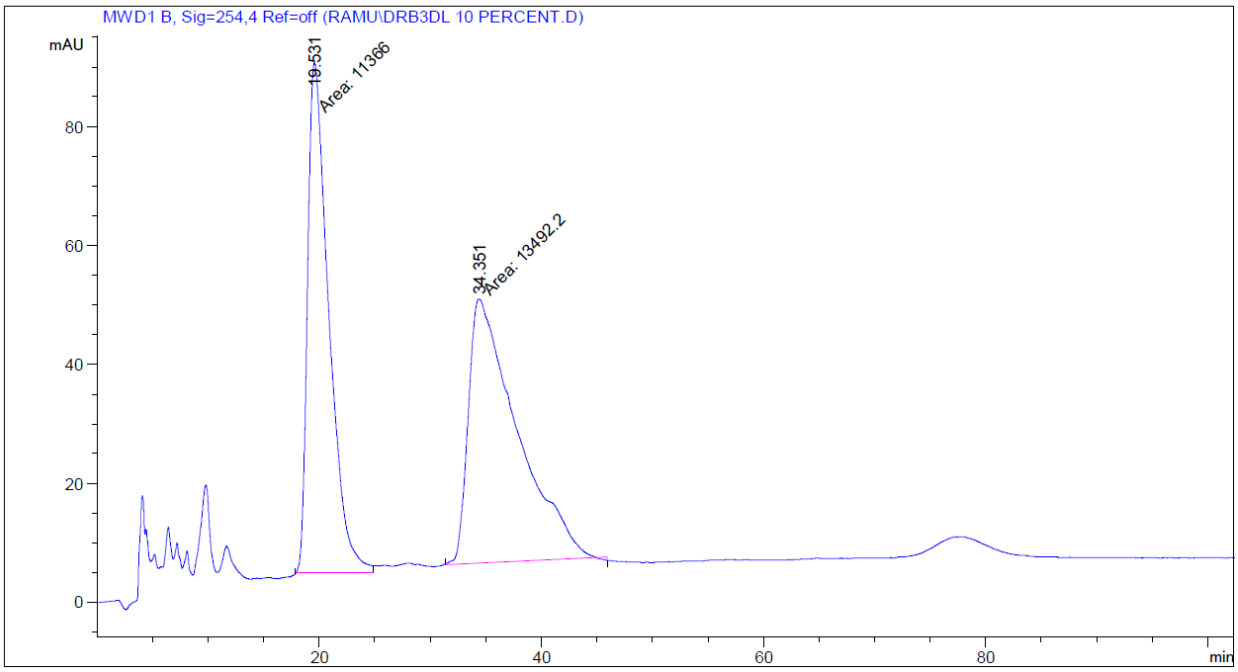

```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MWD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.885 BB    0.8388  673.37836   11.78235  23.7052
   2  16.441 MM    1.5926 2167.25366   22.68087  76.2948
```

HPLC data of compound **22** (racemic)



Area Percent Report

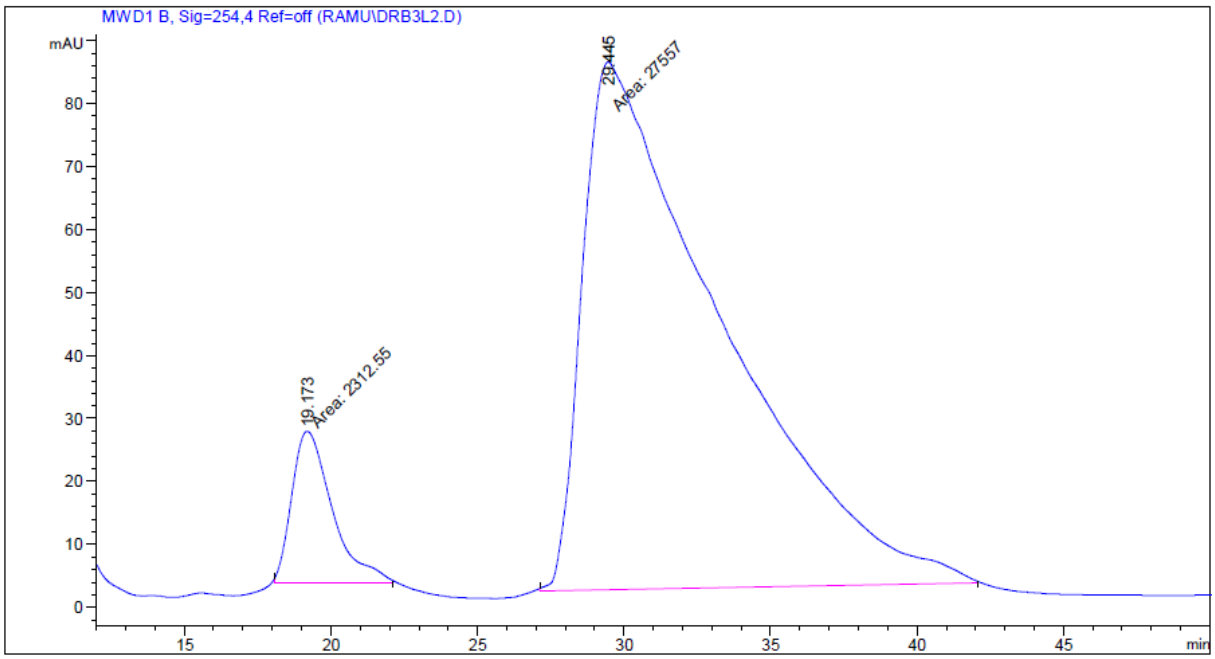
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: MWD1 B, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.531	MM	2.2082	1.13660e4	85.78431	45.7234
2	34.351	MM	5.0616	1.34922e4	44.42665	54.2766
Totals :				2.48582e4	130.21096	

HPLC data of compound **22a**

Additional Info : Peak(s) manually integrated




```
=====================================================================
                        Area Percent Report
=====================================================================

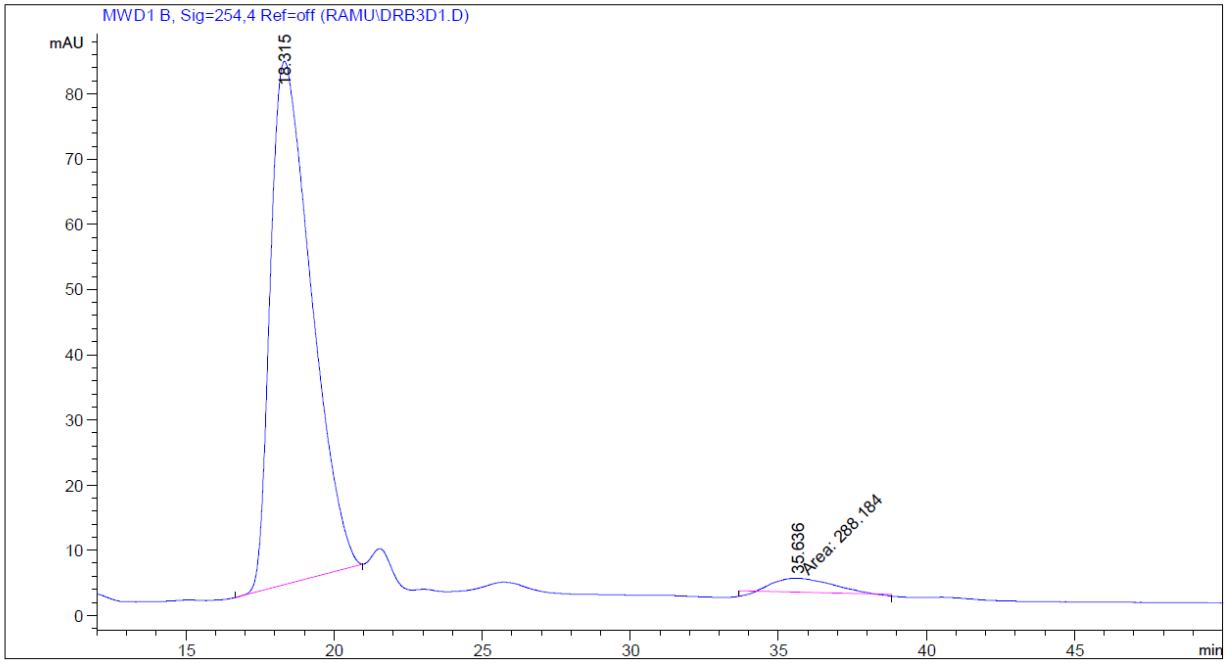
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MWD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  19.173 MM    1.6014 2312.55200   24.06782   7.7422
   2  29.445 MM    5.4805 2.75570e4    83.80362  92.2578
```

HPLC data of compound **22b**

Additional Info : Peak(s) manually integrated




```
=====================================================================
                        Area Percent Report
=====================================================================

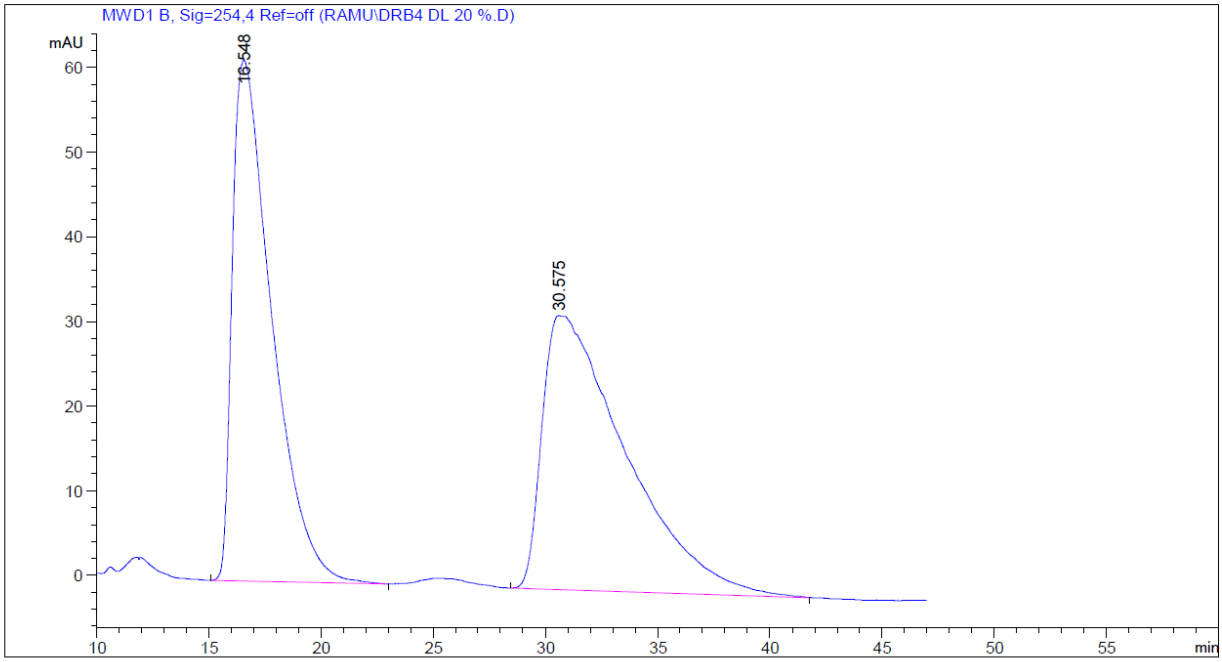
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MWD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  18.315 BBA   1.4865 7699.76758   80.31213  96.3923
   2  35.636 MM    2.2767  288.18362    2.10969   3.6077
```

HPLC data of compound **28** (racemic)

Additional Info : Peak(s) manually integrated




```
=====================================================================
                      Area Percent Report
=====================================================================

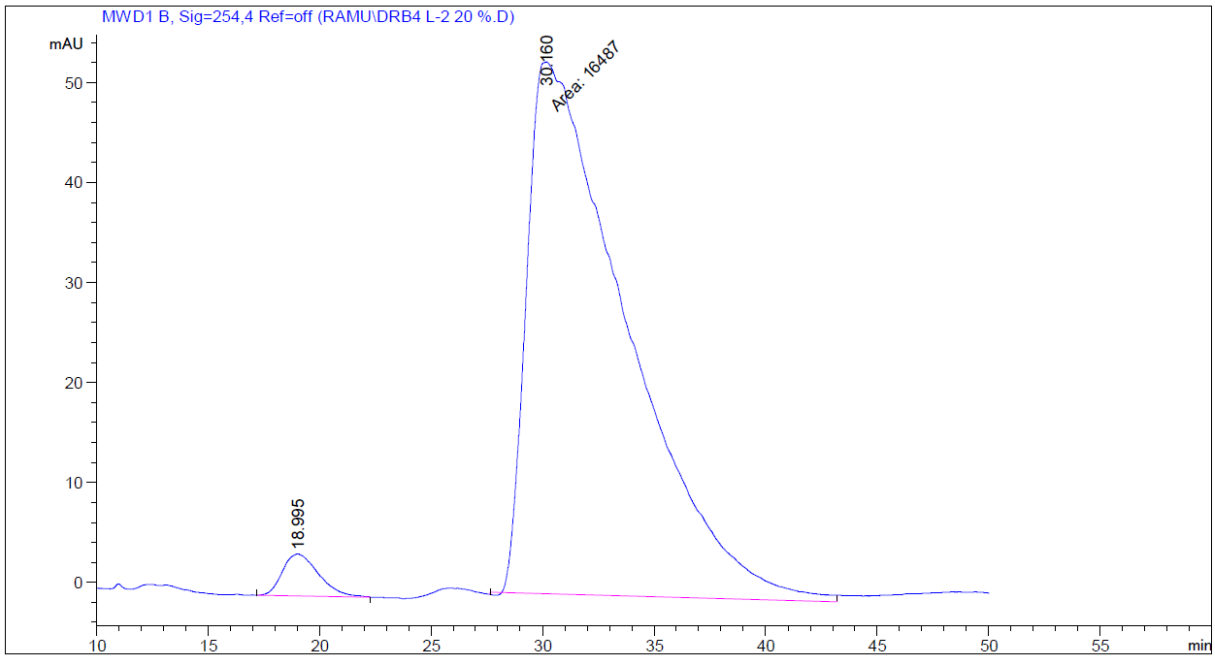
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MWD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.548 BB    1.7085 7544.90381   61.53383  47.4229
   2  30.575 BB    3.2352 8364.93066   32.36626  52.5771
```

HPLC data of compound **28a**

Additional Info : Peak(s) manually integrated




```
=====================================================================
                        Area Percent Report
=====================================================================

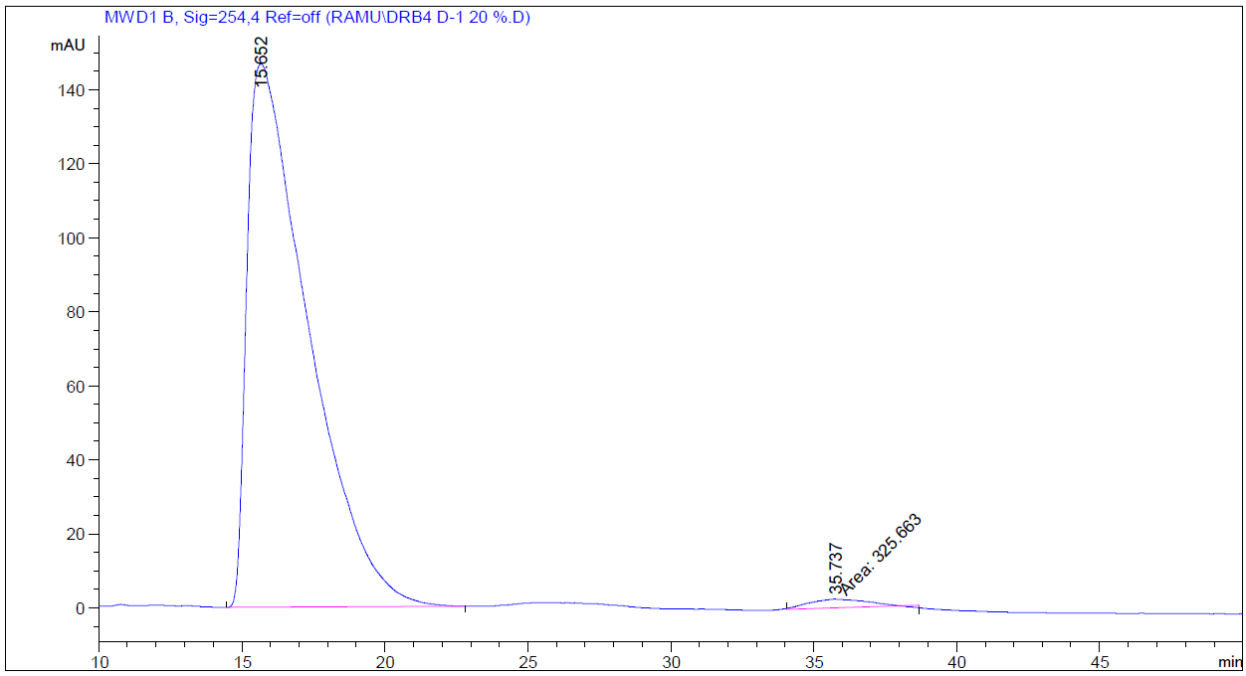
Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MWD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  18.995 BB    1.3243  473.77185    4.19906   2.7933
   2  30.160 MM    5.1674 1.64870e4    53.17634  97.2067
```

HPLC data of compound **28b**

Additional Info : Peak(s) manually integrated




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier:                   :      1.0000
Dilution:                     :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: MWD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.652 BB    1.9720 2.18578e4   146.89430  98.5320
   2  35.737 MM    2.3142  325.66333    2.34541   1.4680
```