BEILSTEIN JOURNAL OF ORGANIC CHEMISTRY

Supporting Information

for

Robust perfluorophenylboronic acid-catalyzed stereoselective synthesis of 2,3-unsaturated *O*-, *C*-, *N*- and *S*-linked glycosides

Madhu Babu Tatina, Xia Mengxin, Rao Peilin and Zaher M. A. Judeh

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Experimental data and copies of ^{1}H and ^{13}C NMR spectra of glycosides 3a–u, 5a–d and 7a–h are provided

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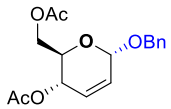
1. Materials and methods

Chemical reagents were purchased from Sigma-Aldrich or Alfa Aesar and were used as received without further purification. ^{1}H NMR spectra were recorded at 300 MHz on a Bruker Avance DPX 300. ^{13}C NMR spectra were recorded at 75.47 MHz on a Bruker Avance DPX 300. Unless stated otherwise, data refer to solutions in $CDCl_3$ with TMS as an internal reference. HRMS were recorded on a Qstar XL MS/MS system. Analytical TLC was performed using Merck 60 F_{254} precoated silica gel plates (0.2 mm thickness) and visualized using UV radiation (254 nm) or stained using ceric ammonium nitrate in 30% H_2SO_4 solution. Flash chromatography was performed using Merck silica gel 60 (60–120 mesh).

2. General procedure for the synthesis of compounds 3a-3u, 5a-5d and 7a-7h

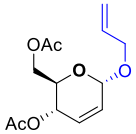
To a stirred solution of 3,4,6-tri-*O*-acetyl-D-glucal (**1a**, 136 mg, 0.5 mmol) or 2,3,4,6-tetra-*O*-acetyl-D-glucal (**4a**, 165 mg, 0.5 mmol) or 3,4-di-*O*-acetyl-L-rhamnal **6a** (107 mg, 0.5 mmol) in anhydrous nitromethane (3 mL) was added the acceptor (0.55 mmol) and perfluorophenylboronic acid (0.1 mmol) at room temperature. In the case of **1a** and **4a**, the resulting solution was stirred at 40 °C for 6 h while in the case of **6a**, it was stirred at room temperature for 2 h (monitored by TLC). The reaction mixture was evaporated under reduced pressure, and the residue was purified using silica gel column chromatography (EtOAc/hexane).

2.1. Benzyl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3a)[1]



Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and benzyl alcohol (0.55 mmol, 58 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **3a** as white solid (147 mg, 92%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 (m, 5H), 5.89 (m, 2H), 5.36 (d, *J* = 9.5, 1H), 5.16 (s, 1H), 4.83 (d, *J* = 11.7 Hz, 1H), 4.62 (d, *J* = 11.7 Hz, 1H), 4.31 – 4.22 (m, 1H), 4.21 – 4.08 (m, 1H), 2.12 (s, 3H), 2.10 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.3, 137.6, 129.3, 128.5, 128.0, 127.9, 127.7, 126.9, 93.6, 70.3, 67.1, 65.3, 62.9, 20.9, 20.8.

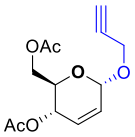
2.2 Allyl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3b)[1]



Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and allyl alcohol (0.55 mmol, 38 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **3b** as white solid (114 mg, 85%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.98 – 5.72 (m, 3H), 5.29 – 5.19 (m, 2H), 5.14 (dd, *J* = 10.3, 1.5 Hz, 1H), 5.01 (s, 1H), 4.26 – 4.11 (m, 3H), 4.12 – 3.96 (m,

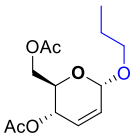
2H), 2.04 (s, 3H), 2.02 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.8, 169.3, 133.1, 128.2, 126.7, 116.5, 92.6, 68.3, 65.9, 64.3, 61.9, 19.9, 19.8.

2.3 Propargyl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3c)[1]



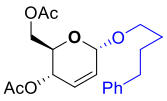
Prepared by the general procedure **1a** (0.5 mmol, 136 mg) and propargyl alcohol (0.55 mmol, 32 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **3c** as white solid (117 mg, 88%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.98 – 5.81 (m, 2H), 5.35 (dd, *J* = 9.5, 1.3 Hz, 1H), 5.25 (s, 1H), 4.40 – 4.15 (m, 2H), 4.11 (m, 1H), 2.48 (t, *J* = 2.4 Hz, 1H), 2.12 (s, 2H), 2.10 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.7, 170.2, 129.8, 127.2, 92.7, 79.1, 74.8, 67.2, 65.1, 62.7, 55.0, 20.9, 20.8.

2.4 *n*-propyl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3d)[1]



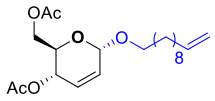
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and *n*-propanol (0.55 mmol, 41 µl). Column chromatography purification using EtOAc/hexane (2:8) gave **3d** as colourless liquid (121 mg, 89%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.88 – 5.76 (m, 2H), 5.28 (dd, *J* = 9.6, 1.3 Hz, 1H), 4.98 (s, 1H), 4.25 – 4.15 (m, 2H), 4.15 – 4.06 (m, 1H), 3.69 (dd, *J* = 11.5, 4.7 Hz, 1H), 3.46 (dt, *J* = 9.5, 6.5 Hz, 1H), 1.59 (m, 2H), 0.91 (t, *J* = 7.4 Hz, 3H).

2.5 Benzenebutyl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3e)[2]



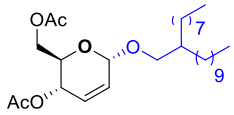
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and 4-Phenyl-1-butanol (0.55 mmol, 84 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **3e** as colourless liquid (115 mg, 90%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 5.7, 1.4 Hz, 3H), 7.23 – 7.15 (m, 2H), 5.96 – 5.77 (m, 2H), 5.33 (dd, *J* = 9.6, 1.4 Hz, 1H), 5.03 (s, 1H), 4.30 – 4.22 (m, 1H), 4.20 (d, *J* = 2.4 Hz, 1H), 4.17 – 4.07 (m, 1H), 3.87 – 3.77 (m, 1H), 3.54 (dt, *J* = 9.6, 6.1 Hz, 1H) 2.67 (t, *J* = 7.3 Hz, 2H), 2.10 (s, 3H), 2.09 (s, 3H), 1.76 – 1.64 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.3, 142.2, 129.0, 128.4, 128.33, 128.30, 127.9, 125.7, 94.4, 68.7, 66.9, 65.3, 63.0, 35.6, 29.3, 28.1, 20.9, 20.7.

2.6 10-Undecen-1-yl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3f)



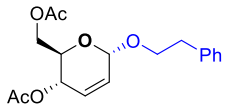
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and 10-undecen-1-ol (0.55 mmol, 110 µL). Column chromatography purification using EtOAc/hexane (1:9) gave **3f** as colourless liquid (169 mg, 89%). $[\alpha]_D^{20}$ +63.617 (c, 1.4, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.92 – 5.75 (m, 3H), 5.33 (dd, *J* = 9.6, 1.3 Hz, 1H), 5.03 (s, 1H), 5.00 – 4.90 (m, 2H), 4.32 – 4.19 (m, 2H), 4.19 – 4.00 (m, 1H), 3.78 (dt, *J* = 9.5, 6.7 Hz, 1H), 3.51 (dt, *J* = 9.5, 6.6 Hz, 1H), 2.11 (s, 3H), 2.10 (s, 3H),2.08 – 2.00 (m, 2H) 1.61 (m, 3H), 1.30 (s, 9H), 1.00 – 0.74 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.3, 139.1, 128.9, 127.9, 114.1, 94.4, 69.0, 66.8, 65.3, 63.0, 33.7, 29.7, 29.5, 29.4, 29.3, 29.2, 28.9, 26.2, 20.9, 20.8. HRMS (ESI+): m/z $[M + Na]^+$ calcd for $C_{21}H_{34}O_6Na$: 405.2253; found: 405.2268.

2.7 2-Octyl-dodecan-1-yl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3g)



Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and 2-Octyl-1-dodecanol (0.55 mmol, 195 µL). Column chromatography purification using EtOAc/hexane (0.5:9.5) gave **3g** as colourless liquid (219 mg, 86%). $[\alpha]_D^{20}$ +25.984 (c, 1.9, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.97 – 5.80 (m, 2H), 5.33 (dd, *J* = 9.7, 1.2 Hz, 1H), 5.01 (s, 1H), 4.28 (dd, = 11.9, 5.4 Hz, 1H), 4.18 (dd, *J* = 12.1, 2.3 Hz, 1H), 4.14 – 3.98 (m, 1H), 3.70 (dd, *J* = 9.4, 6.2 Hz, 1H), 3.37 (dd, *J* = 9.3, 5.6 Hz, 1H), 2.12 (s, 3H), 2.10 (s, 3H), 1.65 (s, 1H), 1.28 (s, 32H), 0.90 (t, *J* = 6.7 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.3, 128.8, 128.0, 94.6, 72.0, 66.9, 65.6, 65.3, 63.0, 40.5, 38.1, 31.91, 31.9, 30.9,30.0, 29.64, 29.61, 29.3, 26.8,26.7, 22.6, 20.9, 20.7, 14.0. HRMS (ESI+): m/z $[M + Na]^+$ calcd for $C_{30}H_{54}O_6Na$: 533.3818; found: 533.3812

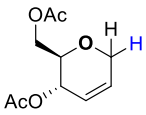
2.8 Benzeneethyl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3h)[2]



Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and 1-phenylethanol (0.55 mmol, 66 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **3h** as colourless liquid (146 mg, 88%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 – 7.27 (m, 3H), 7.24 (d, *J* = 4.5 Hz, 2H), 5.96 – 5.78 (m, 2H), 5.31 (dd, *J* = 9.7, 1.4 Hz, 1H), 5.04 (s, 1H), 4.21 (dd, *J* = 12.1, 5.2 Hz, 1H), 4.09 (d, *J* = 2.3 Hz, 1H), 4.06 – 3.95 (m, 2H), 3.78 (dt, *J* = 9.7, 6.9 Hz, 1H), 2.95 (t, *J* = 7.0 Hz, 2H), 2.10 (s, 3H), 2.09 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$)

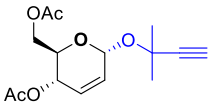
δ 170.8, 170.3, 138.7, 129.1, 128.9, 128.3, 127.7, 126.3, 94.4, 69.5, 66.9, 65.2, 62.9, 36.35, 20.9, 20.7.

2.9 ((2R,3S)-3-Acetoxy-3,6-dihydro-2*H*-pyran-2-yl) methyl acetate (3i)[3]



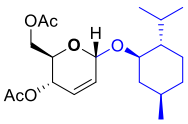
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and triethylsilane (0.55 mmol, 88 µL). Column chromatography purification using EtOAc/hexane (2:8) gave 3i as colourless liquid (79 mg, 74%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.95−5.92 (m, 1H), 5.78-5.75 (ddd, *J* = 9.4 Hz, 3.6 Hz, 1.8 Hz, 1H), 5.27−5.24 (1H, m), 4.24−4.15 (m, 4H), 3.75-3.70 (m, 1H), 2.10 (3H, s), 2.08 (3H, s). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 170.3, 129.4, 124.2, 73.8, 65.2, 65.0, 63.2, 21.0, 20.8

2.10 2-Methyl-3-butyn-2-yl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3j)



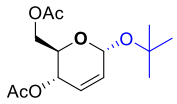
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and 2-methyl-3-butyn-2-ol (0.55 mmol, 53 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **3j** as colourless liquid (118 mg, 80%). $[\alpha]_D^{20}$ +113.627 (c, 0.07, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.90 (d, *J* = 10.3 Hz, 1H), 5.86 – 5.74 (m, 1H), 5.67 (s, 1H), 5.29 (m, 1H), 4.27 – 4.22 (m, 1H), 4.17 (m, 2H), 2.54 (s, 1H), 2.10 (s, 6H), 1.63 (s, 3H), 1.61 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.3, 128.9, 128.5, 90.9, 85.3, 73.3, 71.6, 67.0, 65.2, 63.1, 30.54, 29.88, 21.0, 20.8. HRMS (ESI^+): m/z $[M + Na]^+$ calcd for $C_{15}H_{20}O_6Na$: 319.1158; found: 319.1138.

2.11 L-Menthyl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythrohex-2-enopyranoside (3k)[4]



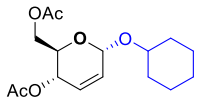
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and L-menthol (0.55 mmol, 86 mg). Column chromatography purification using EtOAc/hexane (2:8) gave **3k** as colourless liquid (165 mg, 90%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.87 (s, 2H), 5.29 (d, *J* = 8.7 Hz, 1H), 5.11 (s, 1H), 4.30 – 4.13 (m, 3H), 3.53 – 3.33 (m, 1H), 2.19 (m, 1H), 2.12 (s, 3H), 2.08 (s, 3H), 1.66 (m, 2H), 1.51 – 1.36 (m, 1H), 1.33 – 1.19 (m, 2H), 1.15 – 0.99 (m, 2H), 0.98 – 0.85 (m, 6H), 0.82 (d, *J* = 6.9 Hz, 1H), 0.78 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.3, 128.5, 128.0, 96.1, 81.0, 66.7, 65.3, 63.3, 48.8, 43.3, 34.2, 31.8, 25.6, 23.1, 22.4, 21.1, 20.9, 20.8, 16.2.

2.12 *tert*-butyl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3l)[5]



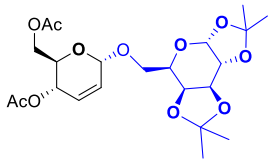
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and *tert*-butanol (0.55 mmol, 53 µL). Column chromatography purification using EtOAc/hexane (2:8) gave 3l as colourless liquid (111 mg, 78%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.86 (d, *J* = 10.2 Hz, 1H), 5.81 – 5.72 (m, 1H), 5.36 – 5.33 (m, 1H), 5.32 – 5.25 (m, 1H), 4.32 – 4.12 (m, 3H), 2.10 (s, 3H), 2.09 (s, 3H), 1.31 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.4, 129.5, 128.1, 88.8, 75.1, 66.6, 65.3, 63.2, 28.4, 21.0, 20.8.

2.13 Cyclohexyl 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranoside (3m)[1]



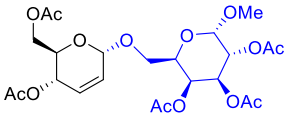
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and cyclohexanol (0.55 mmol, 57 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **3m** as colourless liquid (143 mg, 92%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.03 – 5.72 (m, 2H), 5.31 (dd, *J* = 9.2, 1.3 Hz, 1H), 5.18 (s, 1H), 4.34 – 4.09 (m, 3H), 3.77 – 3.55 (m, 1H), 2.1 (s, 3H), 2.09 (s, 3H), 1.92 (m, 2H) 1.71 (m, 3H), 1.57 (m, 1H), 1.30 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.3, 128.7, 128.5, 92.7, 76.7, 66.7, 65.4, 63.1, 33.7, 32.1, 25.5, 24.4, 24.1, 20.9, 20.7.

2.14 4,6-Di-*O*-acetyl-2,3-dideoxy-D-threo-hex-2-enopyranoside-(α1-6)-1,2:3,4-di-*O*-isopropylidene-D-galactopyranoside (3n)[6a]



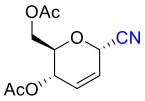
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and 1,2:3,4-di-*O*-isopropylidene-α-D-galactopyranose (0.55 mmol, 143 mg). Column chromatography purification using EtOAc/hexane (2:8) gave **3n** as white solid (132 mg, 56%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.93 – 5.79 (m, 2H), 5.54 (d, *J* = 5.0 Hz, 1H), 5.54 (d, *J* = 5.0 Hz, 1H), 5.11 (s, 1H), 4.64 (dd, *J* = 7.9, 2.4 Hz, 1H), 4.37 – 4.31 (m, 1H), 4.31 – 4.23 (m, 2H), 4.21 – 4.09 (m, 2H), 4.09 – 3.96 (m, 1H), 3.89 (dd, *J* = 10.2, 6.3 Hz, 1H), 3.77 (dd, *J* = 10.1, 7.0 Hz, 1H), 2.12 (s, 3H), 2.10 (s, 3H), 1.55 (s, 3H), 1.46 (s, 3H), 1.36 (s, 3H), 1.35 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.9, 170.3, 129.2, 127.7, 109.3, 108.6, 96.3, 94.6, 70.9, 70.6, 70.5, 67.0, 66.9, 66.2, 65.2, 62.9, 26.05, 26.00, 24.9, 24.5, 20.9, 20.8.

2.15 Methyl-α-D-[2,3,4-Tri-*O*-acetyl-6-*O*-(4',6'-di-*O*-acetyl-2',3'-dideoxy-a-D-hex-2'enopyranosyl)]glucopyranoside (3o)[6b]



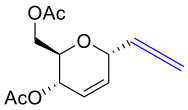
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and 2,3,4-tri-O-acetyl-α-D-methylglucoopyranoside (0.55 mmol, 176 mg). Column chromatography purification using EtOAc:hexane (3:7) solvent system gave 3o as white solid (159 mg, 60% yield). ^{1}H NMR (300 MHz, CDCl3): δ = 5.89 (m, 2H), 5.50 (t, *J* = 9.3 Hz, 1H), 5.36 (dd, *J* = 9.7, 1.4 Hz, 1H), 5.23 – 5.13 (m, 1H), 5.07 (s, 1H), 4.97 (d, *J* = 3.6 Hz, 1H), 4.91 (dd, *J* = 10.2, 3.6 Hz, 1H), 4.30 (dd, *J* = 12.4, 4.5 Hz, 1H), 4.17 – 4.04 (m, 2H), 3.95 (dt, *J* = 10.0, 3.4 Hz, 1H), 3.87 (dd, *J* = 11.1, 3.9 Hz, 1H), 3.65 (dd, *J* = 11.1, 2.7 Hz, 1H), 3.43 (s, 3H), 2.12 (s, 3H), 2.11 (s, 3H), 2.10 (s, 3H), 2.06 (s, 3H), 2.03 (s, 3H).

2.16 4,6-Di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranosyl cyanide (3p)[8]



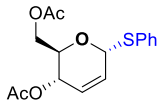
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and Trimethylsilyl cyanide (1.0 mmol, 125 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **3p** as colourless liquid (86 mg, 72%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.05 (dt, *J* = 10.2, 1.9 Hz, 1H), 5.90 (ddd, *J* = 10.2, 3.5, 1.9 Hz, 1H), 5.35 (dd, *J* = 9.1, 2.0 Hz, 1H), 5.15 – 5.06 (m, 1H), 4.27 (d, *J* = 3.9 Hz, 2H), 4.08 – 4.01 (m, 1H), 2.13 (s, 3H), 2.12 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.6, 170.0, 129.7, 123.5, 115.5, 72.0, 62.7, 62.6, 62.2, 20.8, 20.7

2.17 4,6-di-*O*-acetyl-2,3-dideoxy-α-D-*erythro*-hex-2-enopyranoside propadiene (3q)[9]



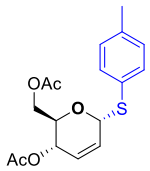
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and Trimethyl(propargyl)silane (0.55 mmol, 82 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **3q** as colourless liquid (88 mg, 70%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.93 (ddd, *J* = 10.3, 2.8, 1.7 Hz, 1H), 5.87 – 5.74 (m, 1H), 5.36 – 5.22 (m, 2H), 4.92 – 4.89 (m, 2H), 4.34 (s, 1H), 4.22 (dd, *J* = 4.2, 2.3 Hz, 2H), 3.93 (ddd, *J* = 8.4, 5.0, 3.3 Hz, 1H), 2.12 (s, 3H), 2.10 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 209.1, 171.0, 170.4, 130.6, 125.0, 89.3, 77.2, 70.5, 68.8, 65.0, 63.1, 21.0, 20.8.

2.18 Phenyl 4,6-di-*O*-acetyl-2,3-dideoxy-1-thio-α-D-*erythro*-hex-2-enopyranoside (3r)[7]



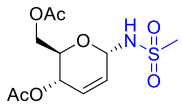
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and thiophenol (0.55 mmol, 56 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **3r** as colourless liquid (151 mg, 94%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 – 7.40 (m, 2H), 7.29 – 7.12 (m, 3H), 5.99 (ddd, *J* = 10.1, 3.1, 1.9 Hz, 1H), 5.79 (dt, *J* = 10.1, 1.7 Hz, 1H), 5.69 (bs, 1H), 5.31 (dd, *J* = 9.5, 1.9 Hz, 1H), 4.46 – 4.31 (m, 1H), 4.30 – 4.09 (m, 2H), 2.04 (s, 3H), 2.00 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.3, 134.7, 131.8, 128.9, 128.5, 127.6, 127.6, 83.7, 67.3, 65.1, 63.1, 21.0, 20.8.

2.19 4-methylPhenyl 4,6-di-*O*-acetyl-2,3-dideoxy-1-thio-α-D-*erythro*-hex-2-enopyranoside (3s)[7]



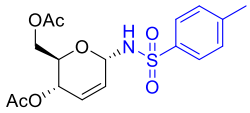
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and 4-Methylbenzenethiol (0.55 mmol, 68 mg). Column chromatography purification using EtOAc/hexane (2:8) gave **3s** as light yellow liquid (151 mg, 90%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.1 Hz, 2H), 7.14 (d, *J* = 8.0 Hz, 2H), 6.08 (ddd, *J* = 10.1, 3.1, 1.9 Hz, 1H), 5.93 – 5.80 (m, 1H), 5.70 (d, *J* = 1.3 Hz, 1H), 5.39 (dd, *J* = 9.5, 1.9 Hz, 1H), 4.56 – 4.44 (m, 1H), 4.27 (dd, *J* = 6.1, 4.3 Hz, 1H), 2.36 (s, 3H), 2.13 (s, 3H), 2.11 (s, 3H).

2.20 4,6-Di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranosyl-4-methanesulfonamide (3t)[10]



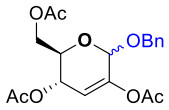
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and Methanesulfonamide (0.55 mmol, 52 mg). Column chromatography purification using EtOAc:Hexane (3:7) solvent system gave **3t** as colourless liquid (99 mg, 65%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.01 (d, *J* = 10.2 Hz, 1H), 5.88 (d, *J* = 12.3 Hz, 1H), 5.57 (q, *J* = 9.2 Hz, 2H), 5.26 (d, *J* = 9.0 Hz, 1H), 4.39 – 4.06 (m, 3H), 4.03 – 3.83 (m, 1H), 3.16 (s, 3H), 2.13 (s, 3H), 2.08 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5, 170.1, 130.3, 126.6, 76.7, 67.4, 64.7, 63.2, 43.0, 20.9, 20.7.

2.21 4,6-Di-*O*-acetyl-2,3-dideoxy-α-D-erythro-hex-2-enopyranosyl-4 methylbenzenesulfonamide (3u)[10]



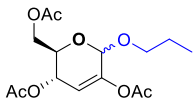
Prepared by the general procedure using **1a** (0.5 mmol, 136 mg) and p-toluenesulfonamide (0.55 mmol, 94 mg). Column chromatography purification using EtOAc:Hexane (3:7) solvent system gave **3u** as *colourless* liquid (124 mg, 65%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.3 Hz, 2H), 7.37 – 7.26 (m, 2H), 6.03 (d, *J* = 8.8 Hz, 1H), 5.92 (d, *J* = 10.1 Hz, 1H), 5.81 (ddd, *J* = 10.1, 3.0, 1.9 Hz, 1H), 5.26 (dd, *J* = 9.3, 1.8 Hz, 1H), 3.89 (dd, *J* = 12.2, 3.4 Hz, 1H), 3.56 (dt, *J* = 9.2, 3.1 Hz, 1H), 3.36 (dd, *J* = 12.2, 2.7 Hz, 1H), 2.44 (s, 1H), 2.03 (s, 3H), 2.02 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.6, 170.0, 143.8, 138.6, 130.4, 129.5, 127.1, 126.7, 66.7, 64.2, 61.8, 21.5, 20.8, 20.7.

2.22 Benzyl 2,4,6-tri-*O*-acetyl-3-deoxy-α/β-D-erythro-hex-2-enopyranoside (5a)[11]



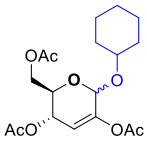
Prepared by the general procedure using **4a** (0.5 mmol, 165 mg) and benzyl alcohol (0.55 mmol, 57 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **5a** as colourless liquid (128 mg, 68%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 (m, 7H), 5.77 (d, *J* = 2.1 Hz, 1H), 5.50 (dd, *J* = 9.3, 1.5 Hz, 1H), 5.35 – 5.23 (m, 2H), 5.15 (s, 1H), 4.83 (d, *J* = 11.8 Hz, 1H), 4.63 (m, 1H), 4.35 (t, *J* = 5.9 Hz, 1H), 4.30 – 4.05 (m, 3H), 2.14 (s, 3H), 2.12 (s, 3H), 2.09 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.8, 170.1, 168.2, 146.3, 140.9, 137.3, 128.5, 127.9, 127.6, 115.4, 112.2, 111.3, 93.06, 92.24, 72.6, 70.5, 69.9, 67.3, 63.2, 62.4, 20.93. 20.9, 20.8.

2.23 *n*-Propyl 2,4,6-tri-*O*-acetyl-3-deoxy-α/β-D-erythro-hex-2-enopyranoside (5b)[11]



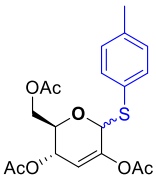
Prepared by the general procedure using **4a** (0.5 mmol, 165 mg) and *n*-propanol (0.55 mmol, 41 µL). Column chromatography purification using EtOAc/hexane (2:8) gave **5b** as colourless liquid (102 mg, 62%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.74 (d, *J* = 2.1 Hz, 1H), 5.47 (dd, *J* = 9.4, 1.3 Hz, 1H), 5.08 (s, 1H), 4.29 (dd, *J* = 6.0, 2.9 Hz, 1H), 4.27 – 4.20 (m, 2H), 4.17 – 4.09 (m, 1H), 3.84 – 3.68 (m, 1H), 3.57 – 3.43 (m, 3H), 2.19 (s, 4H), 2.12 (s, 3H), 2.09 (s, 4H), 1.70 – 1.59 (m, 4H), 0.96 (t, *J* = 7.6 Hz, 5H).

2.24 Cyclohexyl 2,4,6-tri-*O*-acetyl-3-deoxy-α/β-D-erythro-hex-2-enopyranoside (5c)[11]



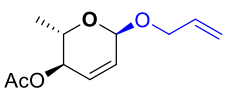
Prepared by the general procedure using **4a** (0.5 mmol, 165 mg) and cyclohexanol (0.55 mmol, 57 µL). Column chromatography purification using EtOAc/hexane (2:8) gave 5c as colourless liquid (129 mg, 70%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.73 (s, 1H), 5.43 (m, 1H), 5.21 (s, 1H), 4.36 – 4.04 (m, 3H), 3.64 (dt, *J* = 9.1, 4.3 Hz, 1H), 2.16 (s, 3H), 2.10 (s, 3H), 2.08 (s, 3H), 1.97 – 1.68 (m, 4H), 1.58 – 1.38 (m, 2H), 1.38 – 1.19 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.7, 170.1, 168.2, 146.9, 114.95, 92.51, 67.0, 65.4, 62.7, 33.5, 31.9, 25.4, 24.1, 23.8, 20.9, 20.8, 20.7.

2.25 4-Methylphenyl 2,4,6-tri-*O*-acetyl-3-deoxy-α/β-1-thio-D-erythro-hex-2-enopyranoside (5d)[11]



Prepared by the general procedure using **4a** (0.5 mmol, 165 mg) and 4-Methyl benzenethiol (0.55 mmol, 68 mg). Column chromatography purification using EtOAc/hexane (2:8) gave **5d** as light yellow colour liquid (105 mg, 78%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.1 Hz, 2H), 7.14 (d, *J* = 8.2 Hz, 2H), 5.78 – 5.62 (m, 2H), 5.53 – 5.46 (m, 1H), 4.60 – 4.48 (m, 1H), 4.27 (dd, *J* = 8.6, 4.8 Hz, 2H), 2.35 (s, 3H), 2.22 (s, 3H), 2.12 (s, 3H), 2.11 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.7, 170.1, 168.0, 146.7, 138.2, 134.1, 132.7, 130.1, 129.8, 129.5, 115.1, 83.9,81.6, 74.6, 67.5, 65.1, 64.9, 62.7, 21.1, 20.9, 20.7.

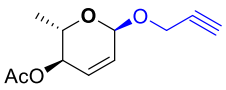
2.26 Allyl 4-*O*-acetyl-2,3,6-trideoxy-α-D-erythro-hex-2-enopyranoside (7a)[12]



Prepared by the general procedure using **6a** (0.5 mmol, 107 mg) and allyl alcohol (0.55 mmol, 38 µL). Column chromatography purification using EtOAc/hexane (1:9) gave **7a** as colourless liquid (94 mg, 89%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.04 – 5.76 (m, 3H), 5.32 (dd, *J* = 17.2, 1.6 Hz, 1H), 5.26 – 5.15 (m, 1H), 5.07 (dd, *J* = 9.2, 1.4 Hz, 1H), 5.03 (s, 1H), 4.27 (ddt, *J* = 6.6, 5.2, 1.4 Hz, 1H), 4.13 – 3.96 (m, 2H), 2.10 (s, 3H), 1.24 (d, *J* = 6.3

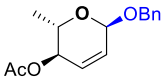
Hz,3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.54, 129.4, 127.9, 94.3, 70.9, 70.3, 64.6, 23.0, 21.0, 17.9, 10.6.

2.27 Propargyl 4-*O*-acetyl-2,3,6-trideoxy-α-D-erythro-hex-2-enopyranoside (7b)[12]



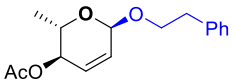
Prepared by the general procedure using **6a** (0.5 mmol, 107 mg) and propargyl alcohol(0.55 mmol, 32 µL). Column chromatography purification using EtOAc/hexane (1:9) gave **7b** as colourless liquid (81 mg, 78%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.02 – 5.73 (m, 2H), 5.19 (s, 1H), 5.09 (dd, *J* = 9.2, 1.5 Hz, 1H), 4.32 (d, *J* = 2.4 Hz, 2H), 4.05 – 3.89 (m, 1H), 2.46 (t, *J* = 2.4 Hz, 1H), 2.10 (s, 3H), 1.25 (d, *J* = 6.3 Hz, 3H).

2.28 Benzyl 4-*O*-acetyl-2,3,6-trideoxy-α-D-erythro-hex-2-enopyranoside (7c)[12]



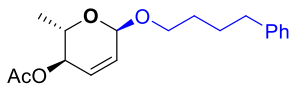
Prepared by the general procedure using **6a** (0.5 mmol, 107 mg) and Benzyl alcohol (0.55 mmol, 57 µL). Column chromatography purification using EtOAc/hexane (1:9) gave **7c** as colourless liquid (111 mg, 85%). 1H NMR (300 MHz, $CDCl_3$) δ 7.44 – 7.23 (m, 5H), 5.98 – 5.74 (m, 2H), 5.16 – 5.00 (m, 2H), 4.81 (d, *J* = 11.9 Hz, 1H), 4.63 (d, *J* = 11.9 Hz, 1H), 4.14 – 3.95 (m, 1H), 2.10 (s, 3H), 1.22 (d, *J* = 6.3 Hz, 3H). 13C NMR (75 MHz, $CDCl_3$) δ 170.5, 137.9, 129.8, 128.4, 127.9, 127.7, 127.7, 93.7, 93.7, 70.9, 70.1, 64.9, 21.1, 17.9.

2.29 Benzeneethyl 4-*O*-acetyl-2,3,6-trideoxy-α-D-erythro-hex-2-enopyranoside (7d)[12]



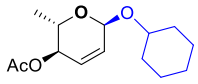
Prepared by the general procedure using **6a** (0.5 mmol, 107 mg) and 2-phenylethanol (0.55 mmol, 66 µL). Column chromatography purification using EtOAc/hexane (1:9) gave **7d** as colourless liquid (110 mg, 80%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 – 7.19 (m, 5H), 5.95 – 5.75 (m, 2H), 5.05 (dd, *J* = 9.2, 1.5 Hz, 1H), 4.97 (s, 1H), 4.01 (dt, *J* = 9.7, 7.2 Hz, 1H), 3.89 (dd, *J* = 9.2, 6.3 Hz, 1H), 3.76 (dt, *J* = 9.7, 7.0 Hz, 1H), 2.95 (t, *J* = 7.1 Hz, 2H), 2.10 (s, 3H), 1.18 (d, *J* = 6.3 Hz, 3H).

2.30 Benzenebutyl 4-*O*-acetyl-2,3,6-trideoxy-α-D-erythro-hex-2-enopyranoside (7e)



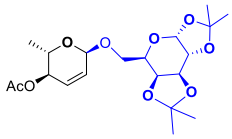
Prepared by the general procedure using **6a** (0.5 mmol, 107 mg) and 4-phenylbutanol (0.55 mmol, 84 µL). Column chromatography purification using EtOAc/hexane (1:9) gave **7e** as colourless liquid (120 mg, 79%). $[\alpha]_D^{20}$ -66.564 (c, 1.2, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 6.0, 0.9 Hz, 2H), 7.20 (dd, *J* = 5.2, 2.4 Hz, 3H), 5.90 – 5.73 (m, 2H), 5.05 (d, *J* = 1.4 Hz, 1H), 4.96 (s, 1H), 4.07 – 3.90 (m, 1H), 3.88 – 3.72 (m, 1H), 3.60 – 3.45 (m, 1H), 2.75 – 2.56 (m, 2H), 2.10 (s, 3H), 1.70 (m, 4H), 1.23 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5, 162.6, 142.3, 129.5, 128.4, 128.3, 127.9, 125.7, 94.4, 70.9, 68.5, 64.7, 35.6, 29.4, 28.0, 21.0, 17.9

2.31 Cyclohexyl 4-*O*-acetyl-2,3,6-trideoxy-α-D-erythro-hex-2-enopyranoside (7f)[13]



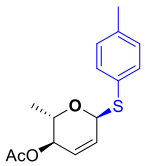
Prepared by the general procedure using **6a** (0.5 mmol, 107 mg) and cyclohexanol (0.55 mmol, 57 µL). Column chromatography purification using EtOAc/hexane (1:9) gave **7f** as colourless liquid (101 mg, 80%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.89 – 5.72 (m, 2H), 5.10 (bs, 1H), 5.05 (dd, *J* = 6.7, 2.6 Hz, 1H), 4.03 (dd, *J* = 9.2, 6.3 Hz, 1H), 3.70 – 3.55 (m, 1H), 2.08 (s, 3H), 1.91 (m, 2H), 1.81 – 1.69 (m, 2H), 1.59 – 1.49 (m, 2H), 1.47 – 1.30 (m, 4H), 1.22 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.59, 152.93, 129.2, 128.5, 92.6, 76.4, 71.0, 64.5, 33.9, 32.2, 25.6, 24.3, 24.1, 21.0, 17.9.

2.32 4-*O*-acetyl-2,3,6-trideoxy-D-threo-hex-2-enopyranoside-(α1-6)-1,2:3,4-di-*O*-isopropylidene-D-galactopyranoside (7g)[6]



Prepared by the general procedure using **6a** (0.5 mmol, 107 mg) and 1,2:3,4-di-*O*-isopropylidene-α-D-galactopyranose (0.55 mmol, 143 mg). Column chromatography purification using EtOAc/hexane (1:9) gave **7g** as colourless liquid (107 mg, 52%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.85 (s, 2H), 5.56 (d, *J* = 5.0 Hz, 1H), 5.07 (d, *J* = 7.8 Hz, 2H), 4.62 (dd, *J* = 7.9, 2.3 Hz, 1H), 4.31 (ddd, *J* = 9.4, 6.5, 1.9 Hz, 2H), 4.06 – 3.92 (m, 2H), 3.77 – 3.62 (m, 1H), 2.10 (s, 3H), 1.56 (s, 3H), 1.47 (s, 3H), 1.36 (s, 6H), 1.24 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5, 129.5, 127.9, 109.2, 108.5, 96.3, 94.2, 71.13, 71.0, 70.6, 70.5, 67.1, 66.5, 64.8, 26.1, 26.0, 24.9, 24.4, 21.1, 17.8.

2.33 4-Methyl phenyl 4-*O*-acetyl-2,3,6-trideoxy-1-thio-α-D-*erythro*-hex-2-enopyranoside (7h)[14]



Prepared by the general procedure using **6a** (0.5 mmol, 107 mg) and 4-methylbenzenethiol (0.55 mmol, 68 mg). Column chromatography purification using EtOAc/hexane (1:9) gave **7h** as colourless liquid (122 mg, 88%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.1 Hz, 2H), 7.15 (d, *J* = 8.1 Hz, 2H), 6.05 (ddd, *J* = 10.1, 2.8, 2.0 Hz, 1H), 5.84 (dd, *J* = 10.1, 1.5 Hz, 1H), 5.66 (s, 1H), 5.17 (ddd, *J* = 9.0, 3.7, 1.8 Hz, 1H), 4.40 – 4.19 (m, 1H), 2.36 (s, 3H), 2.13 (s, 3H), 1.30 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5, 137.5, 132.0, 129.7, 128.6, 127.9, 84.0, 70.7, 65.35, 21.1, 18.0.

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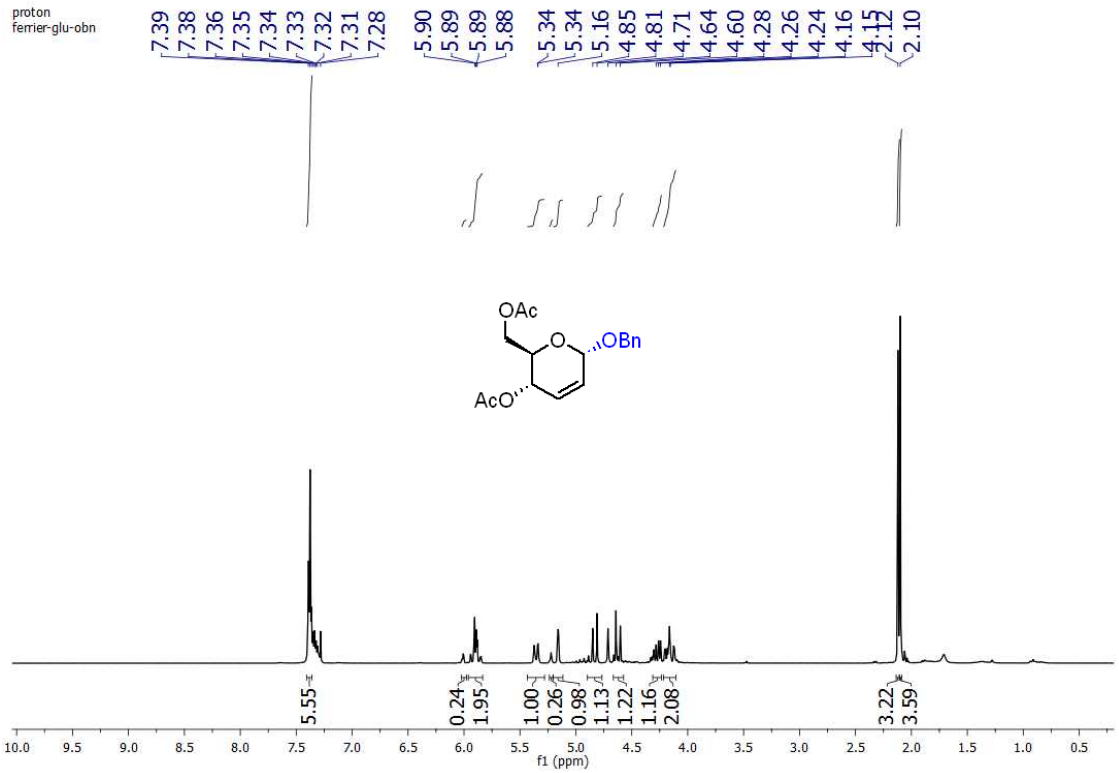
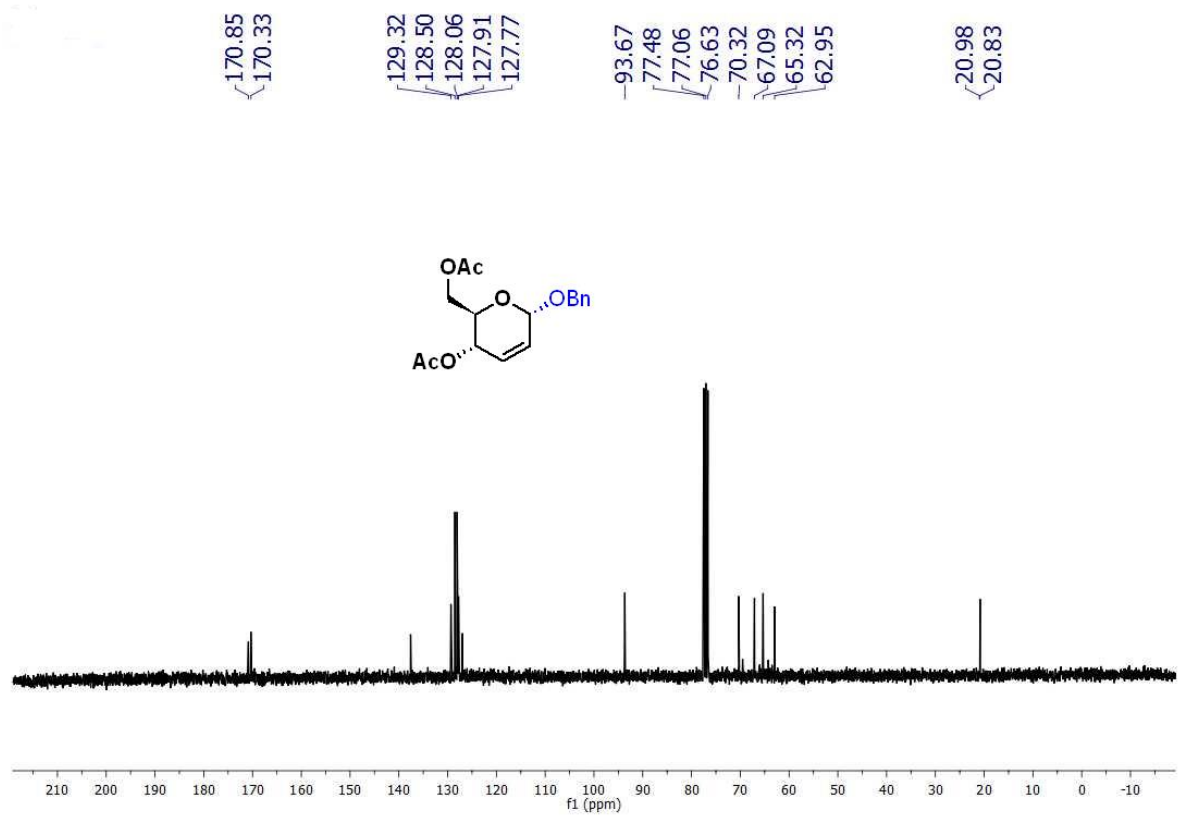
3. Spectral images of compounds 3a–u, 5a–d and 7a–h

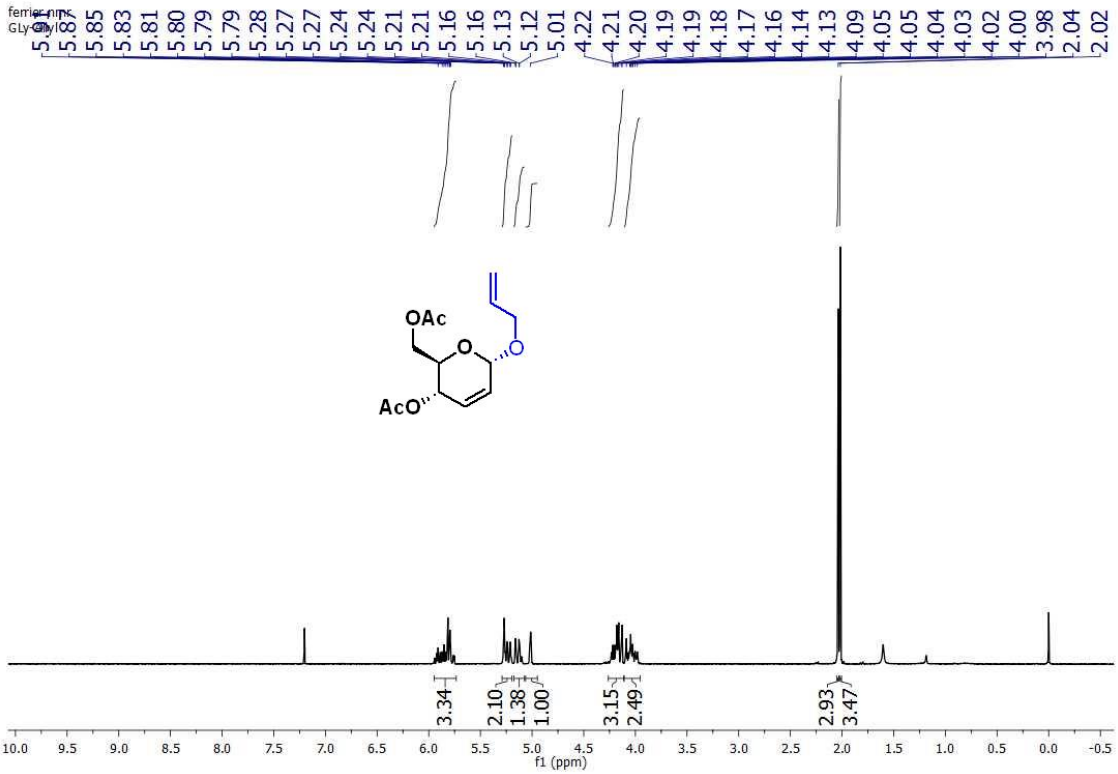
^{1}H NMR of compound 3a



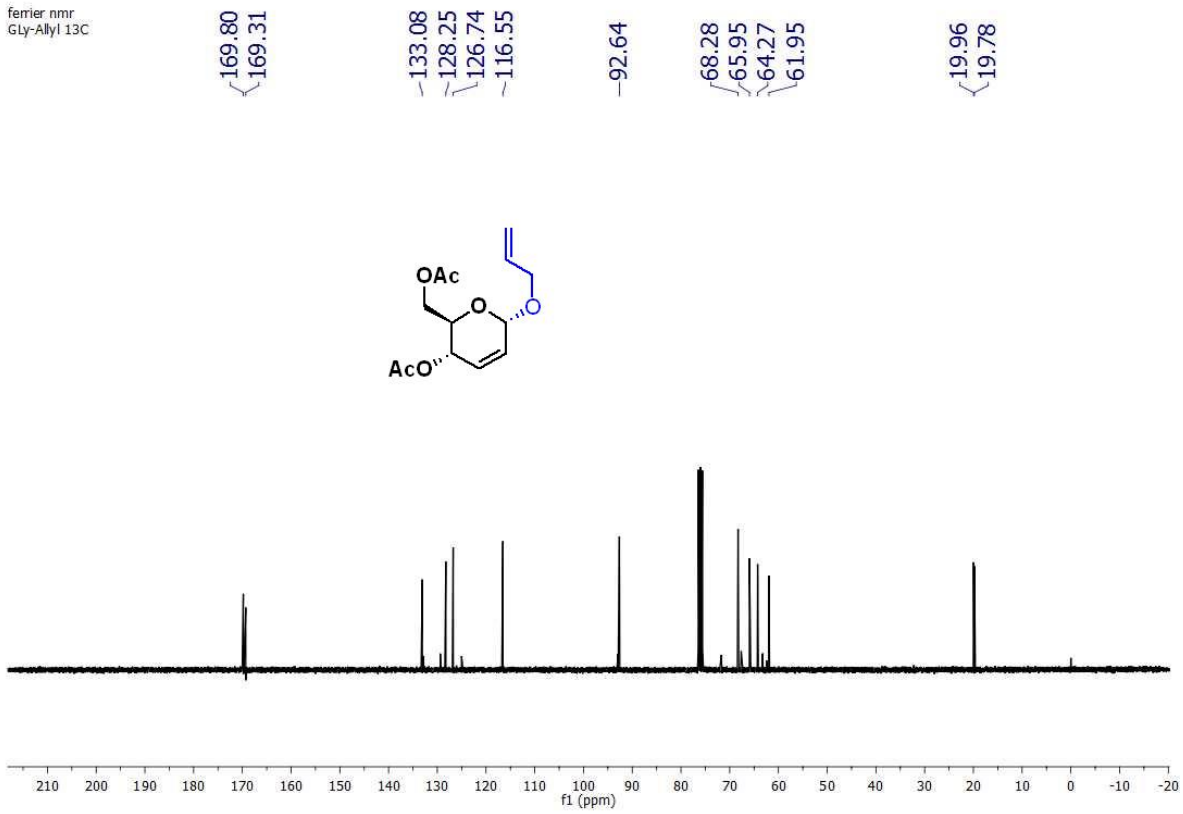
^{13}C NMR of compound 3a



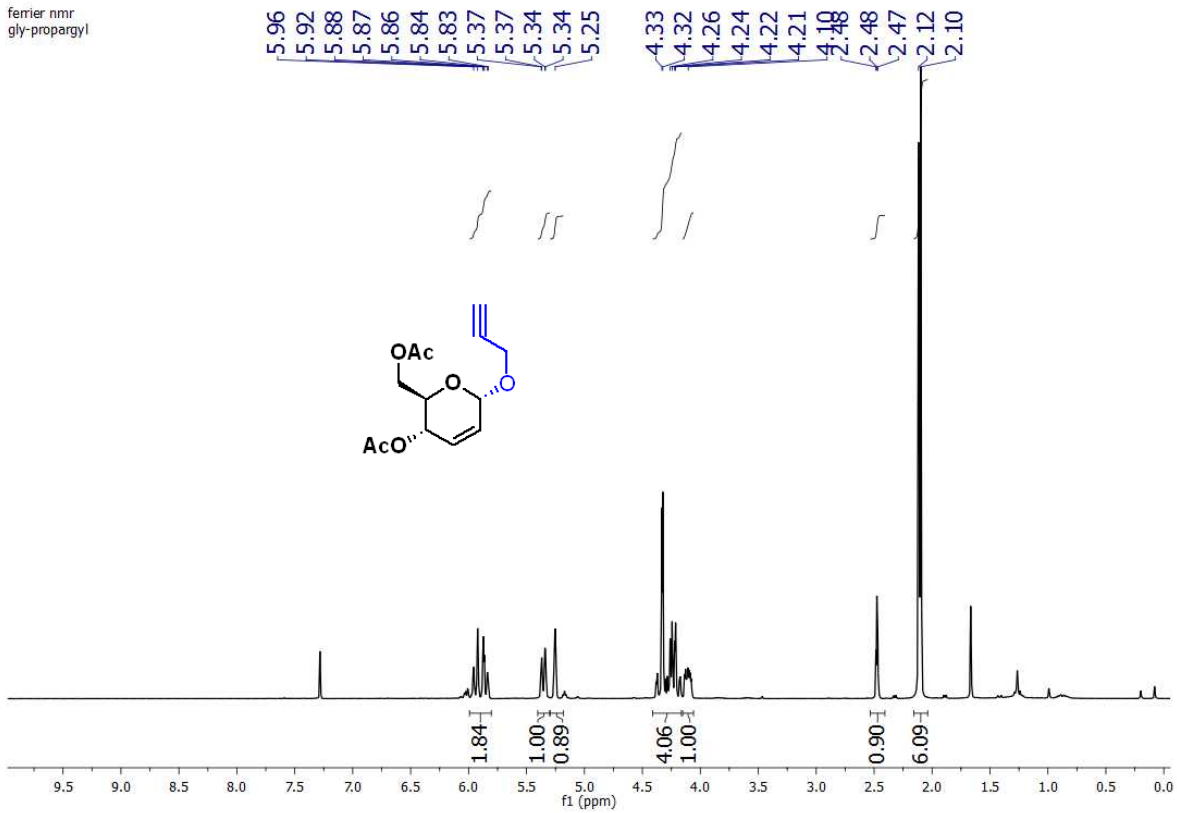
^{1}H NMR of compound 3b



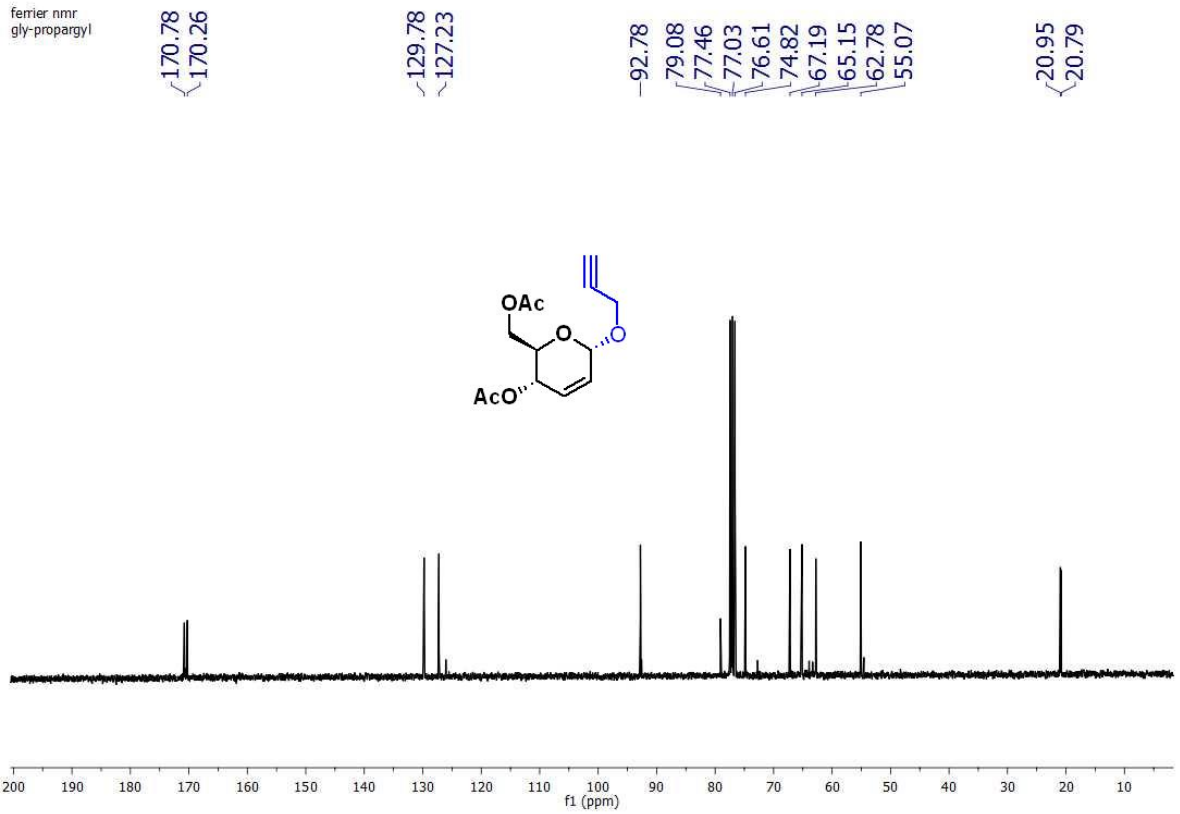
^{13}C NMR of compound 3b



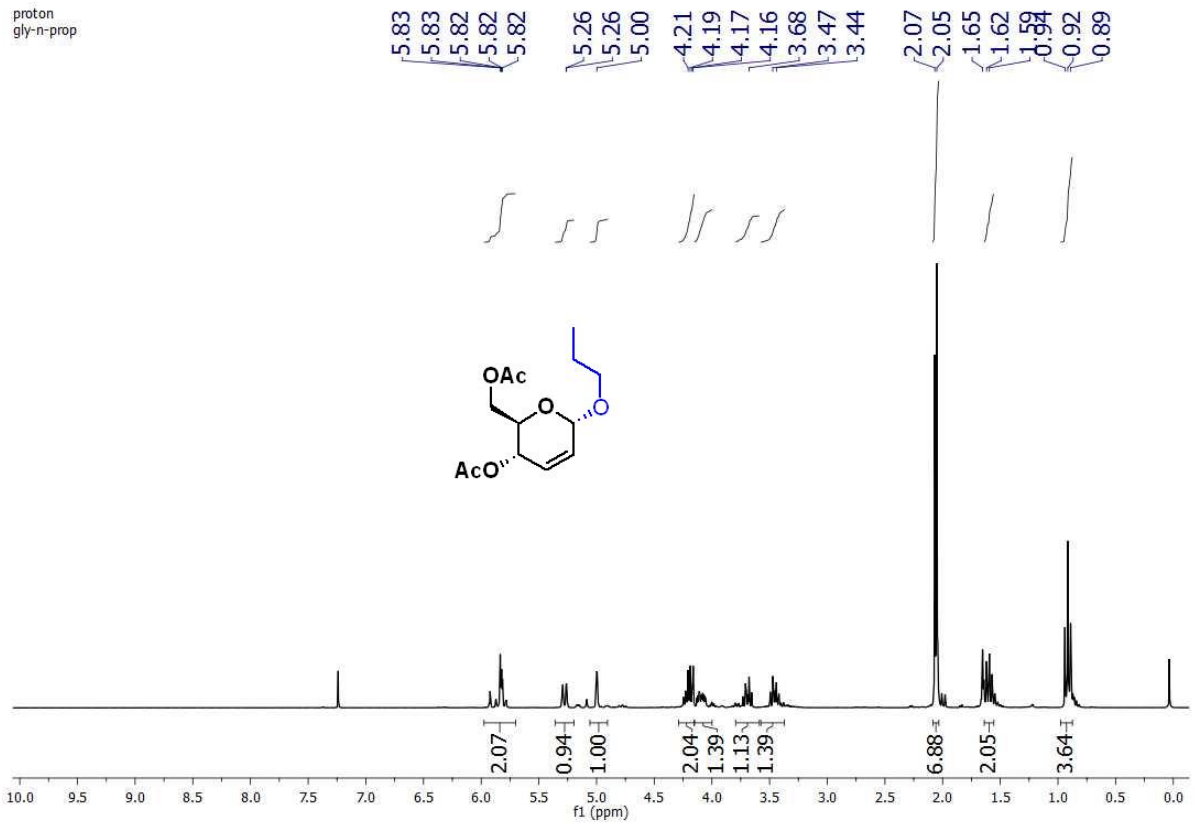
^{1}H NMR of compound 3c



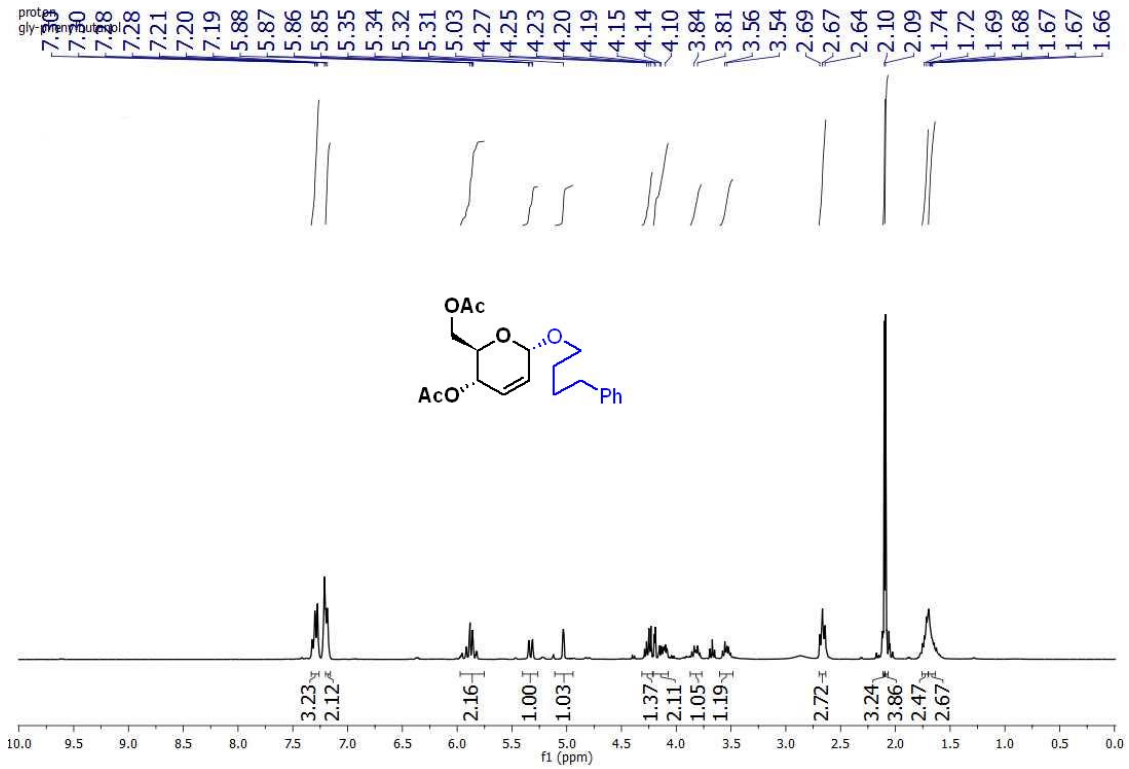
^{13}C NMR of compound 3c



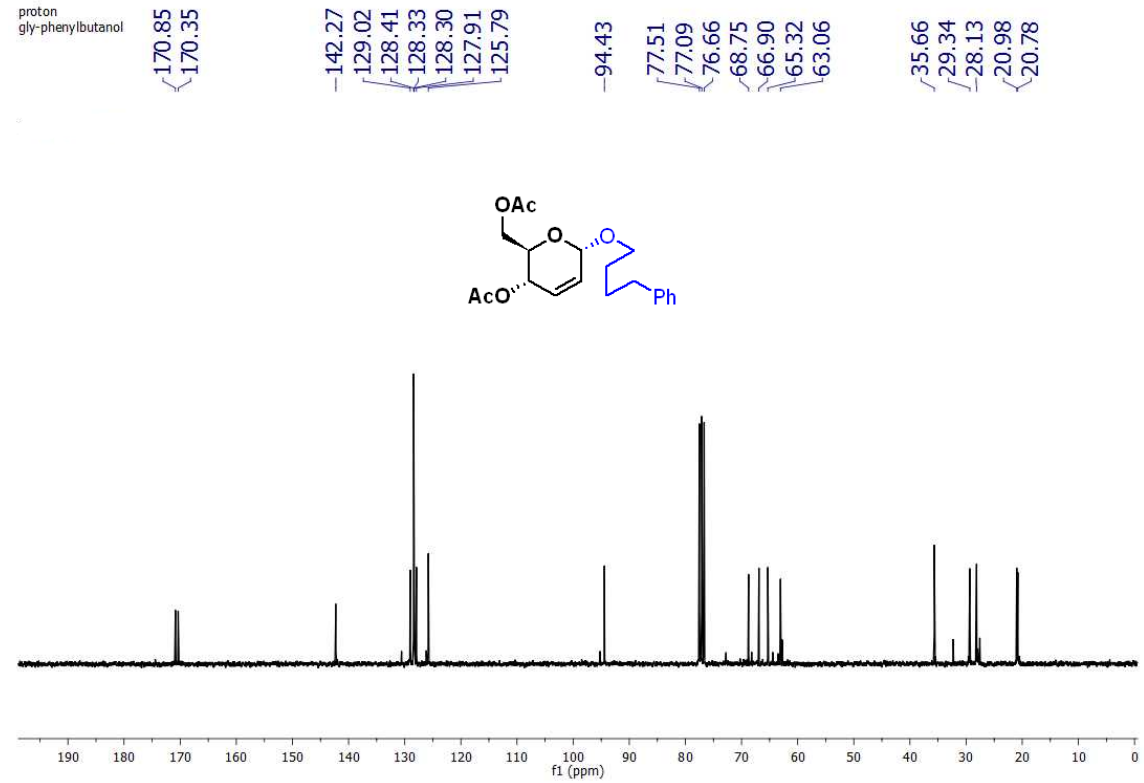
^{1}H NMR of compound 3d



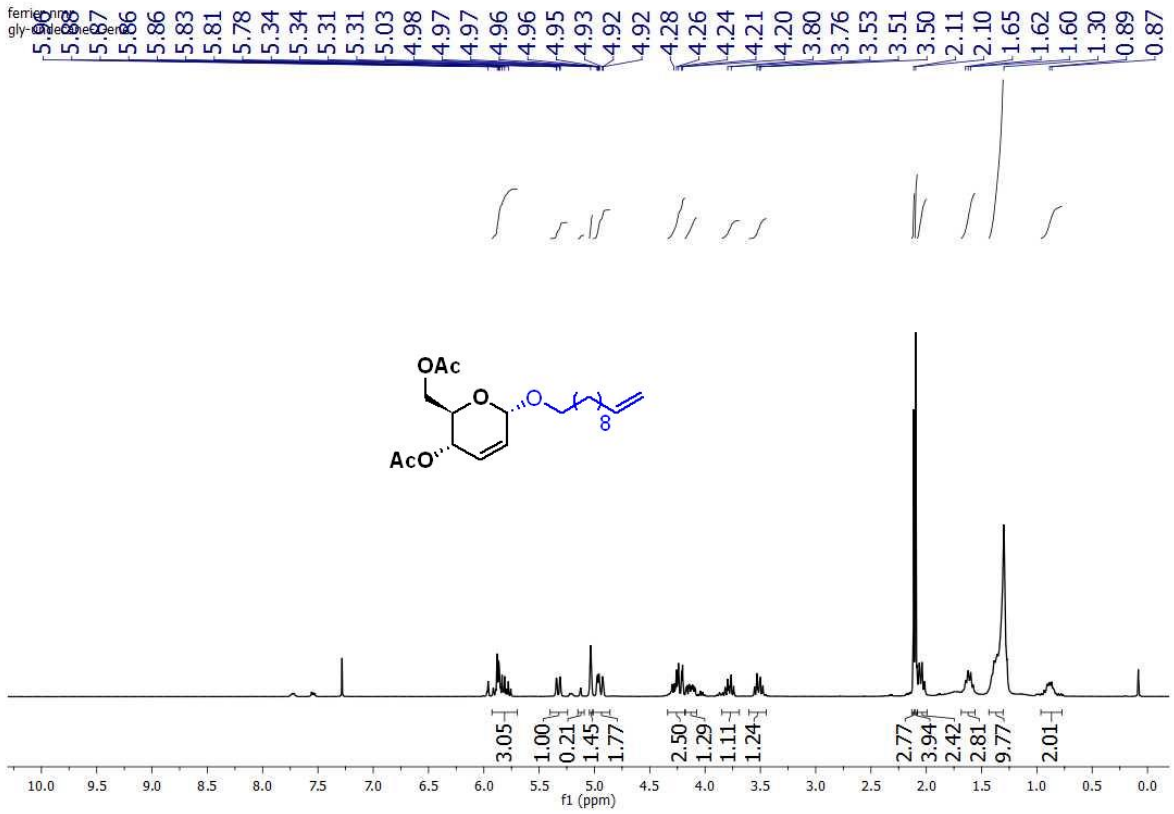
^{1}H NMR of compound 3e



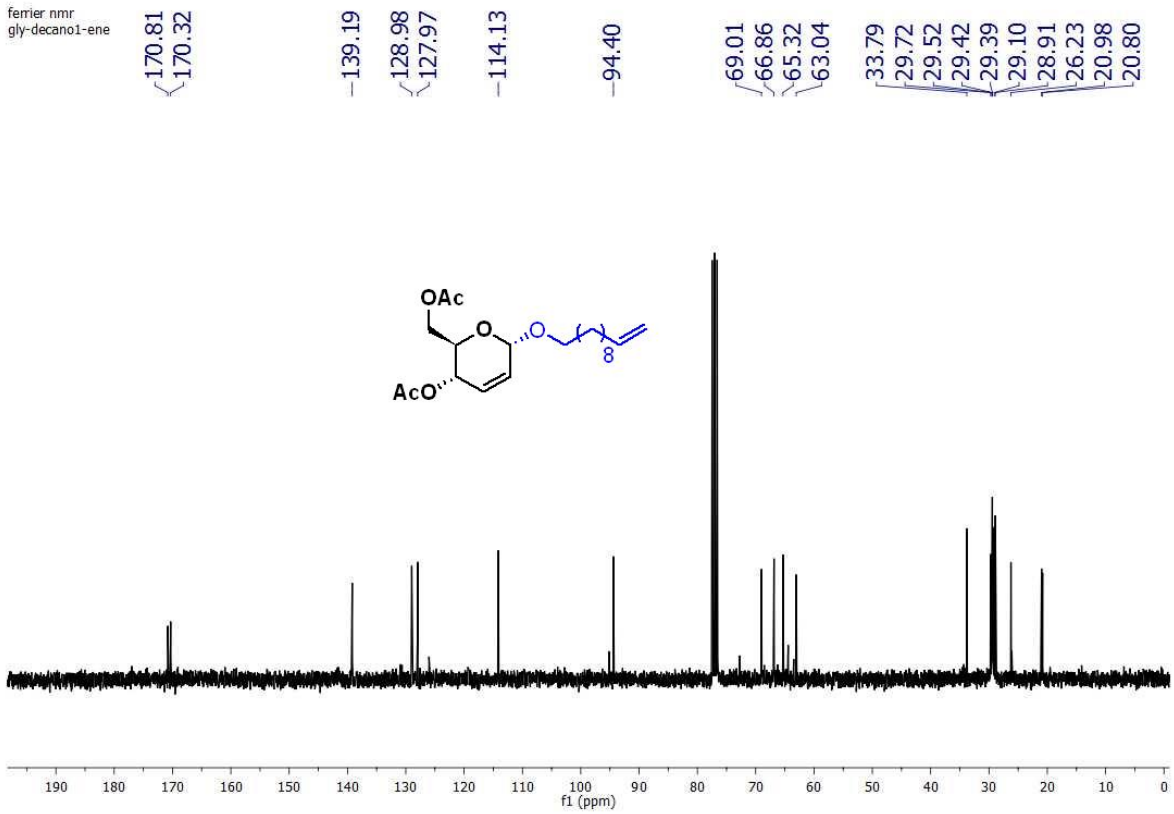
^{13}C NMR of compound 3e



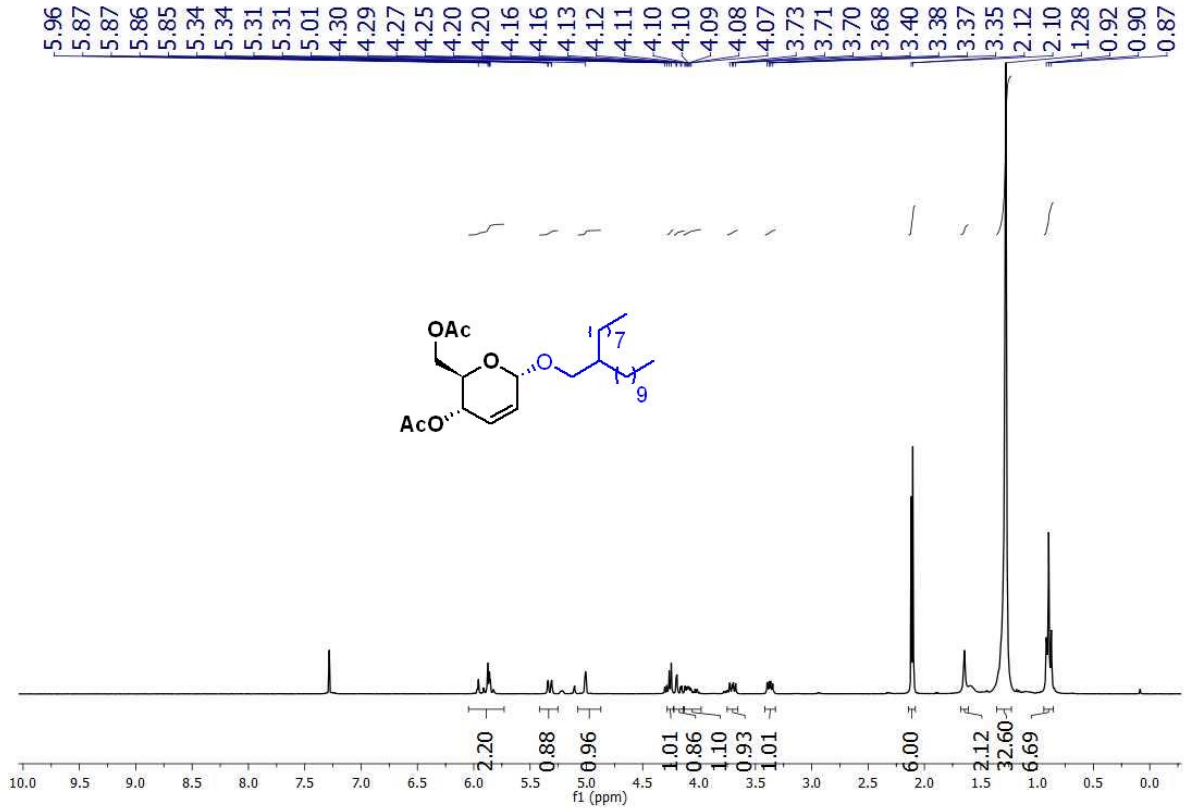
^{1}H NMR of compound 3f



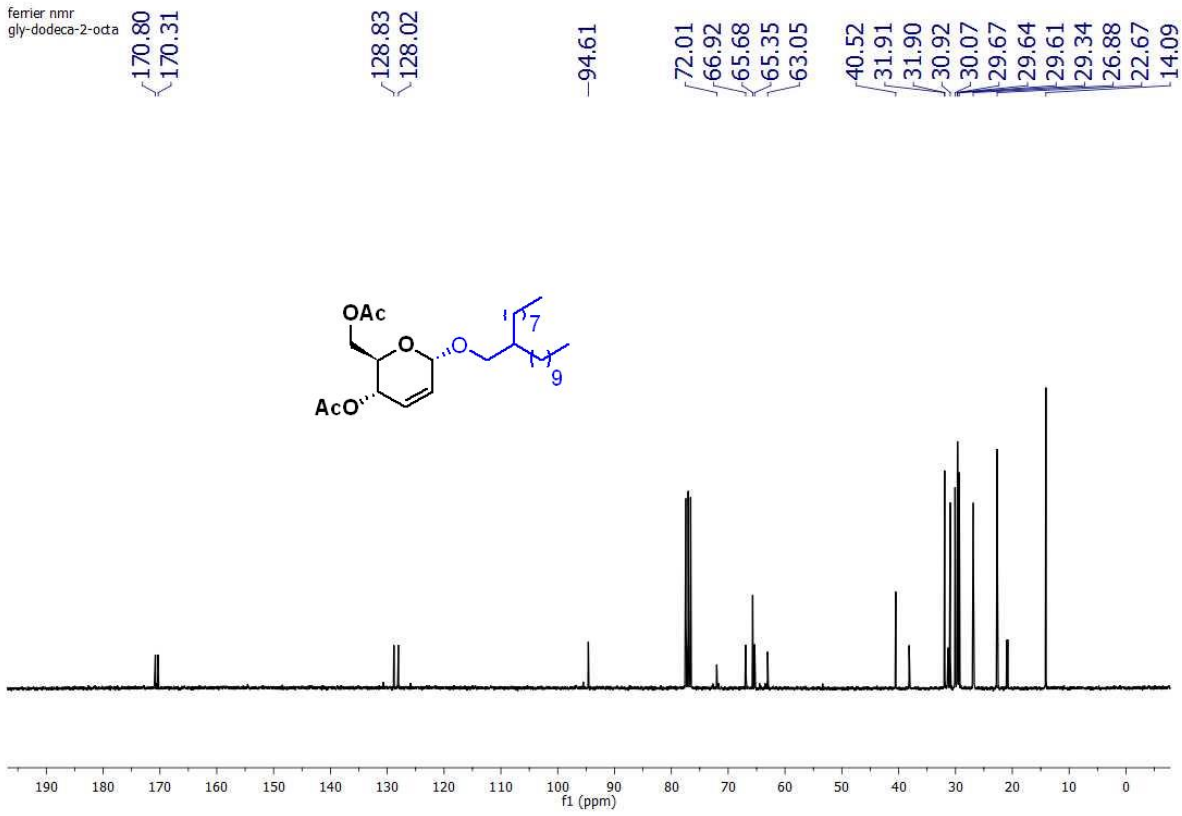
^{13}C NMR of compound 3f



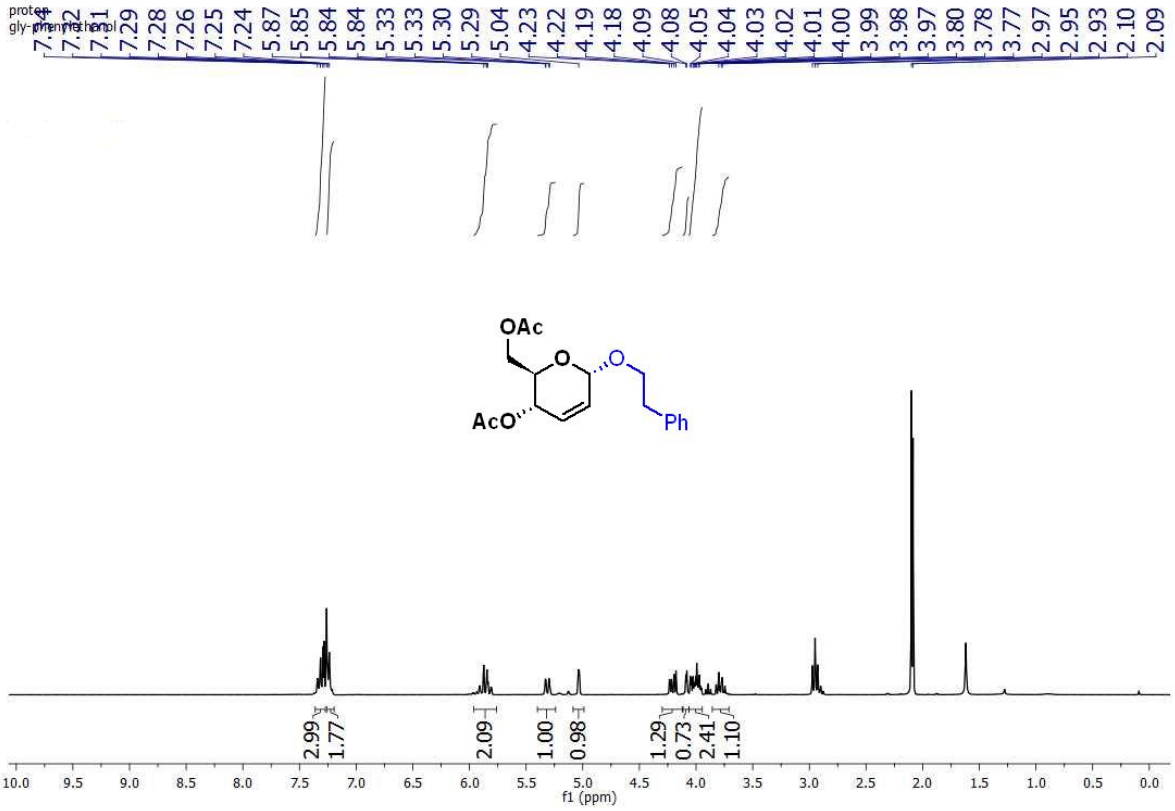
^{1}H NMR of compound 3g



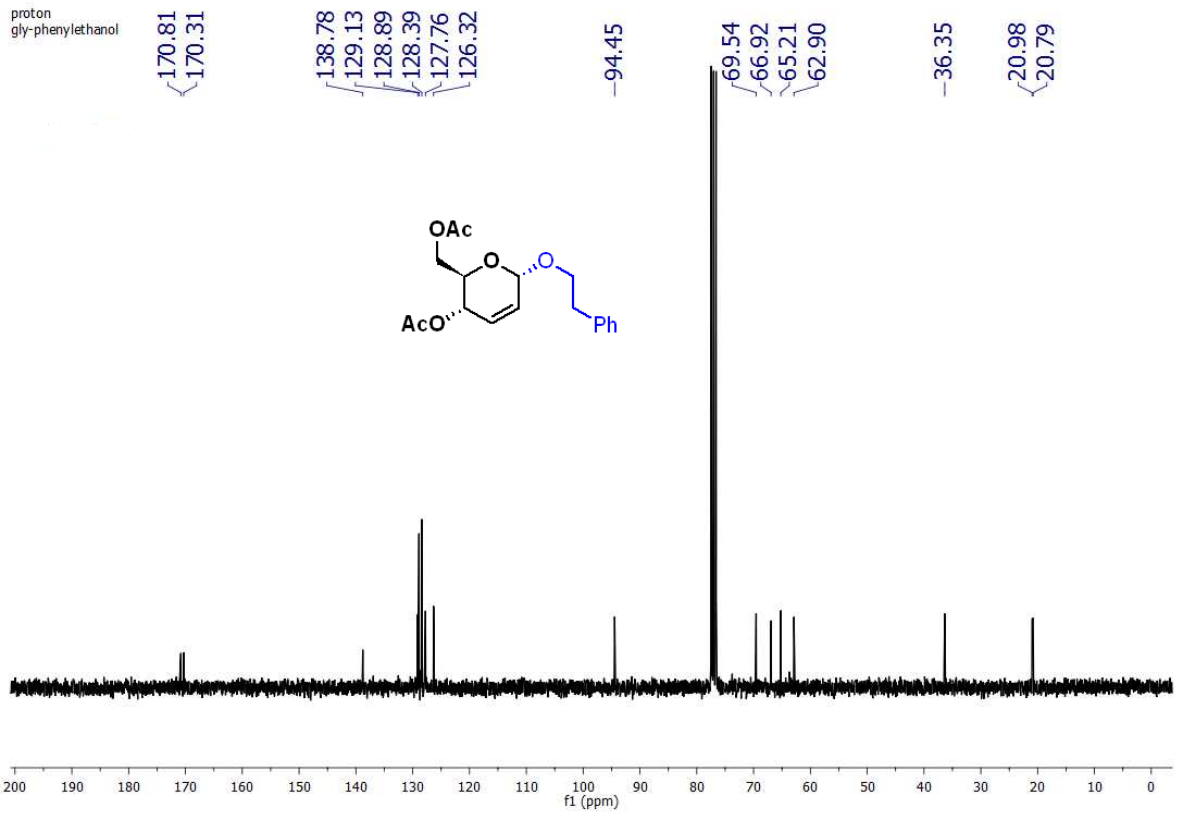
^{13}C NMR of compound 3g



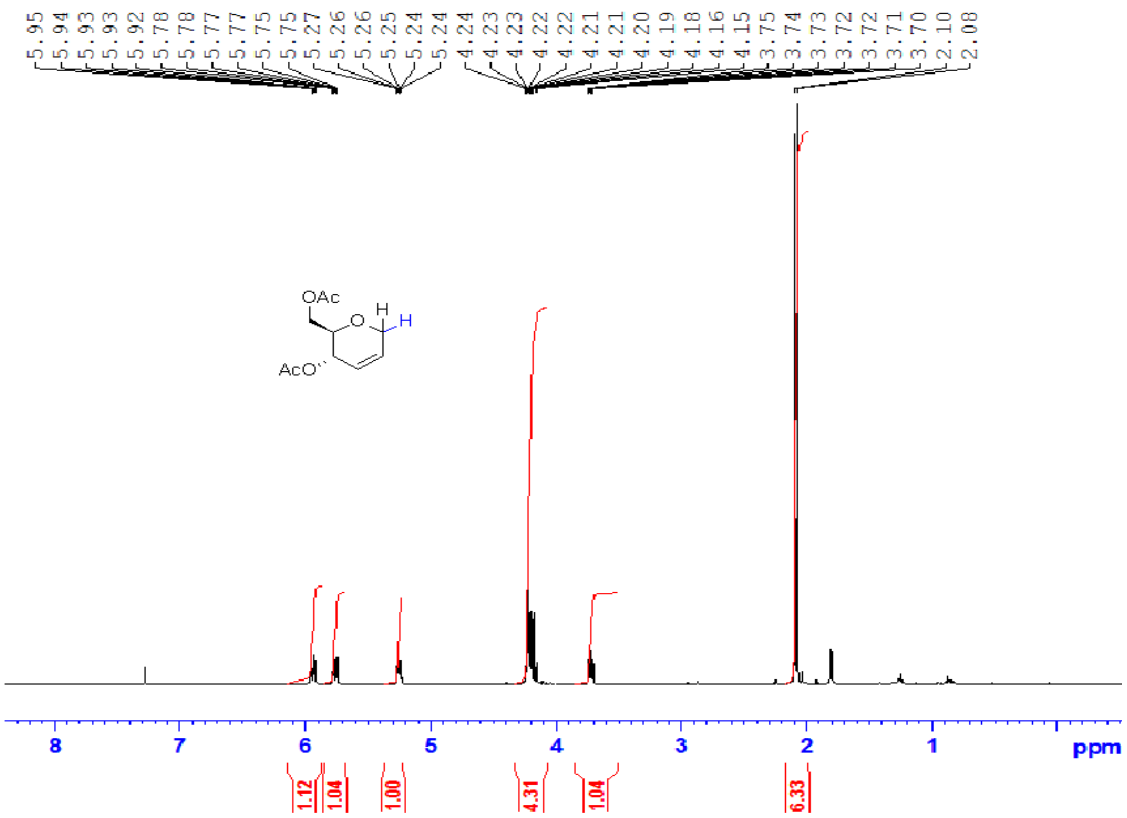
^{1}H NMR of compound 3h



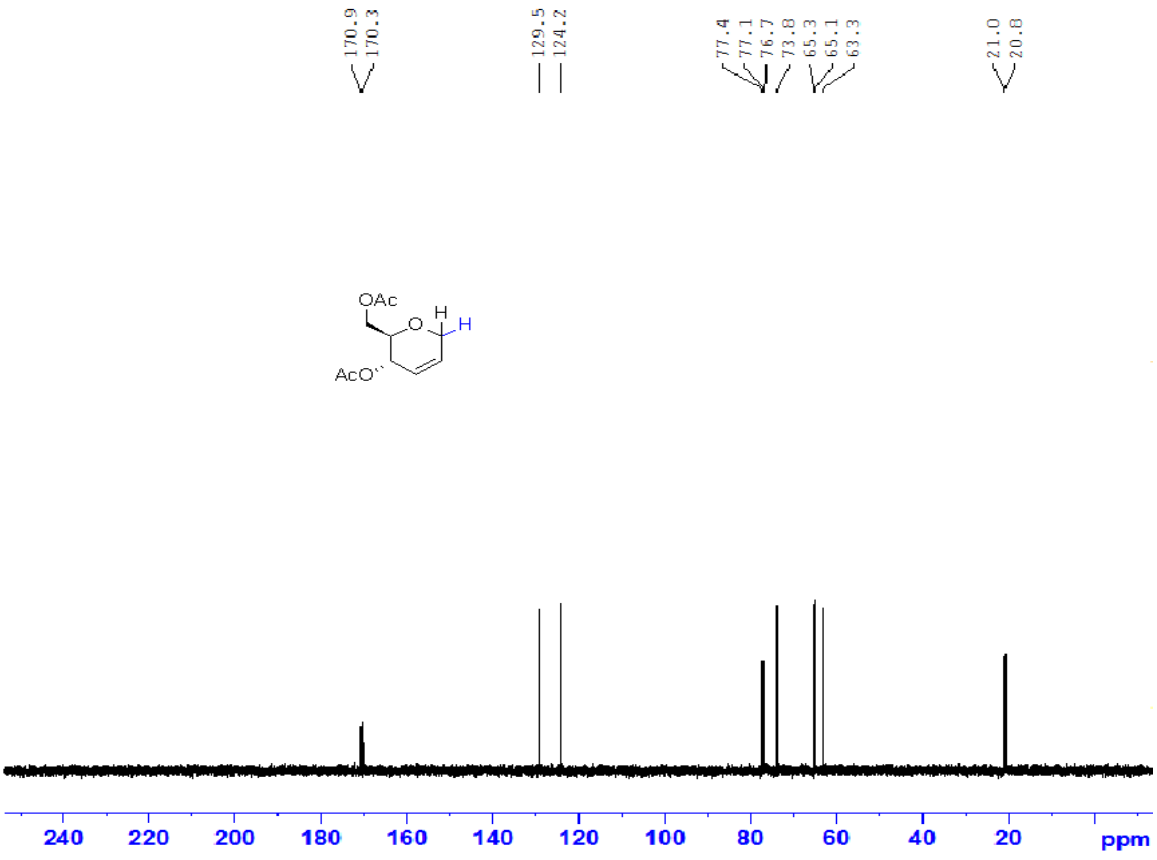
^{13}C NMR of compound 3h



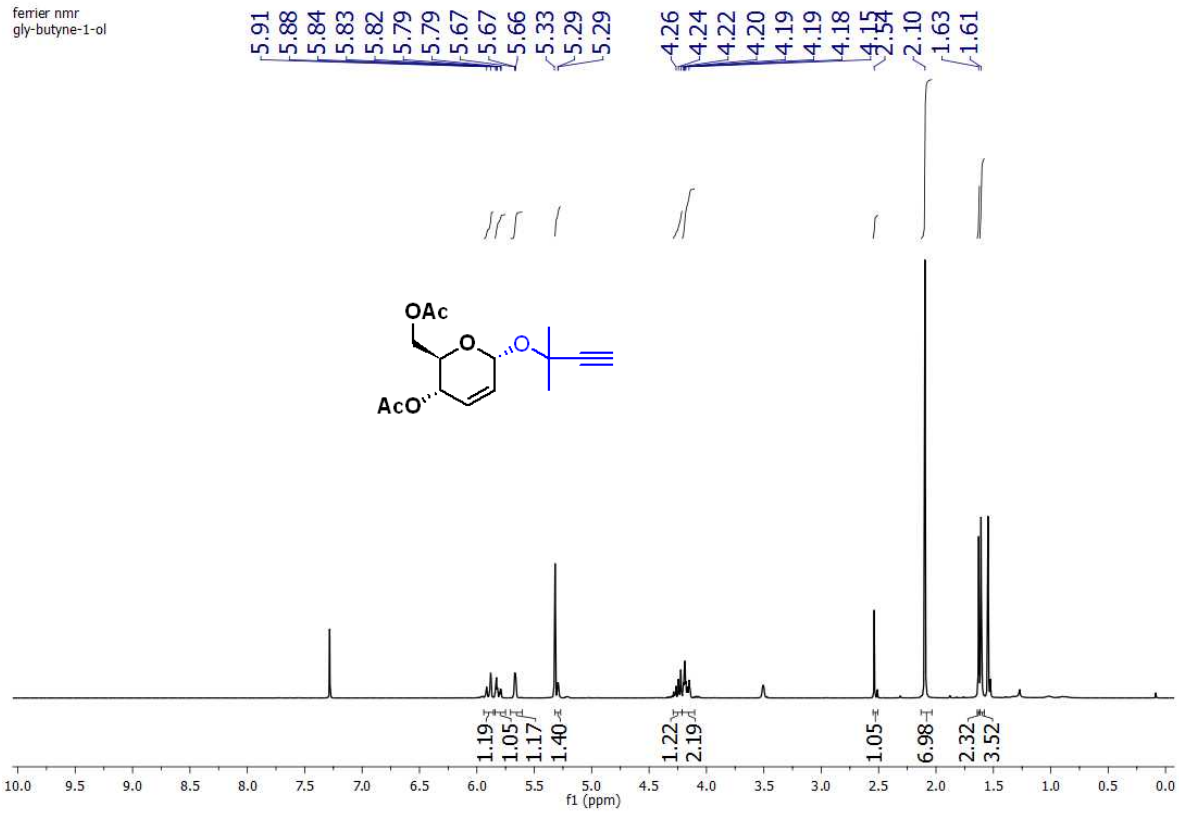
^{1}H NMR of compound 3i



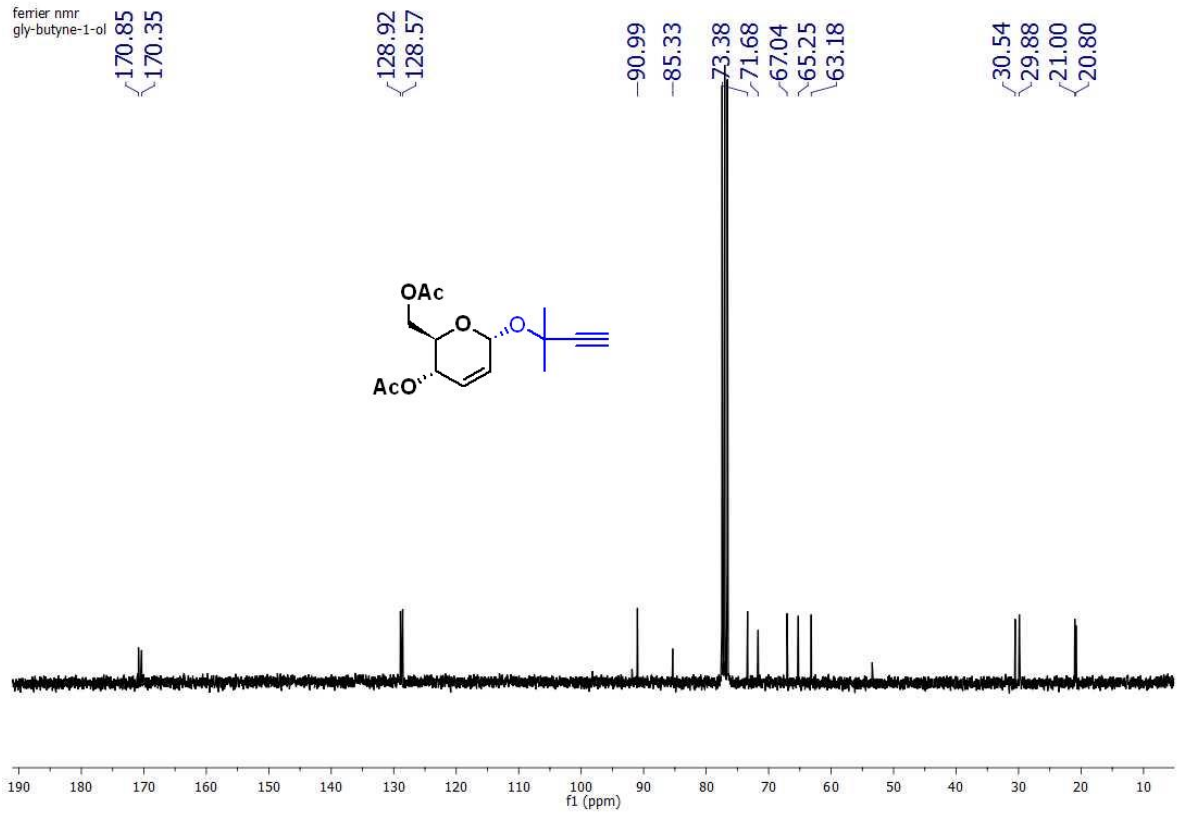
^{13}C NMR of compound 3i



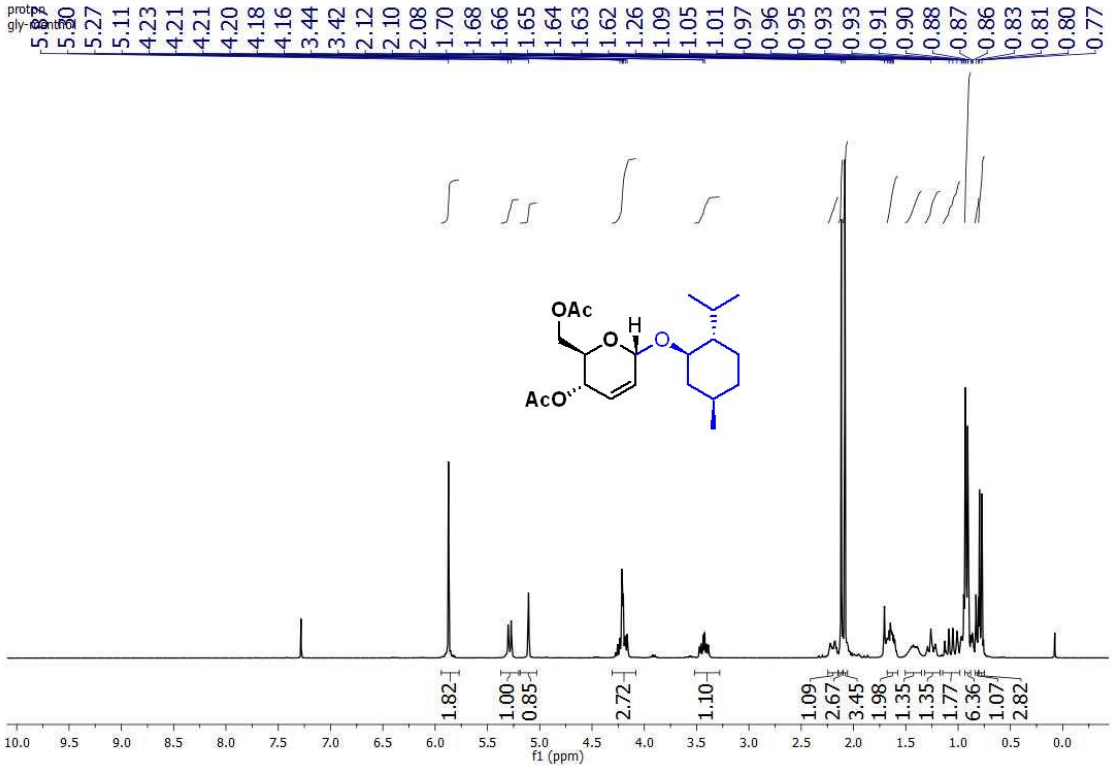
[1]H NMR of compound 3j



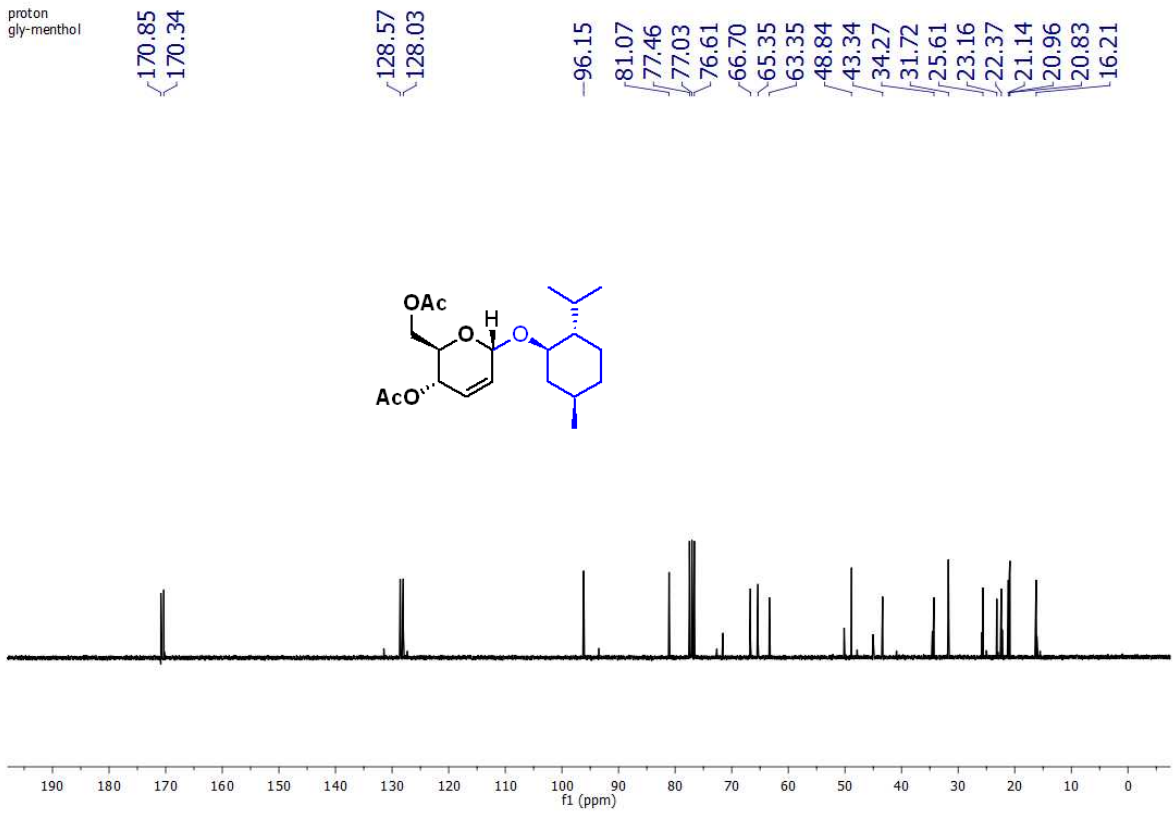
[13]C NMR of compound 3j



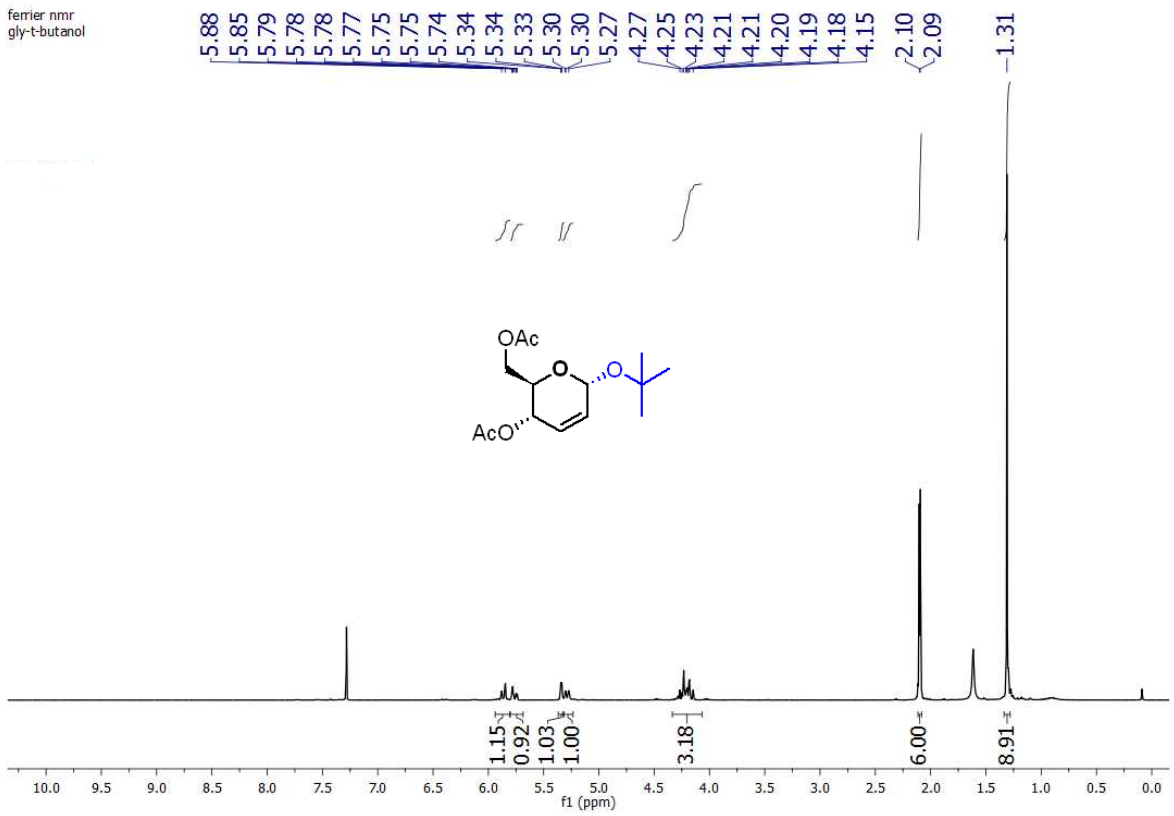
^{1}H NMR of compound 3k



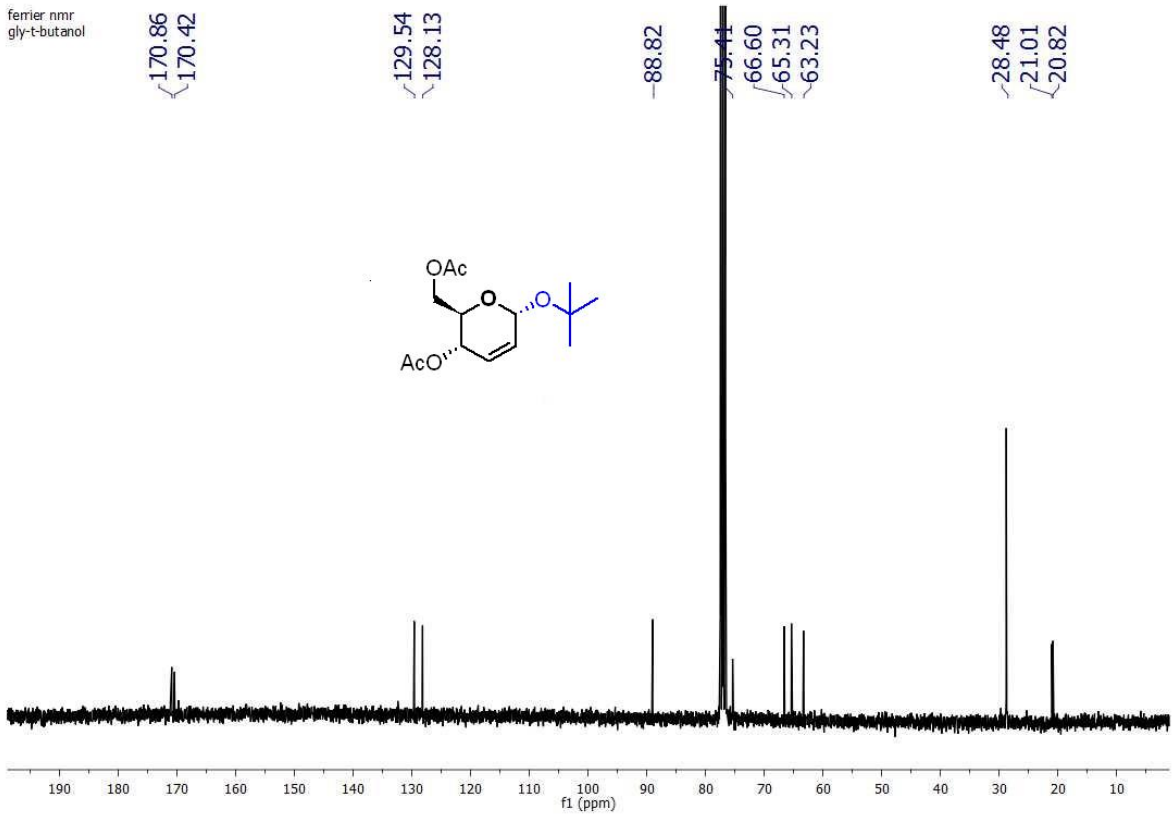
^{13}C NMR of compound 3k



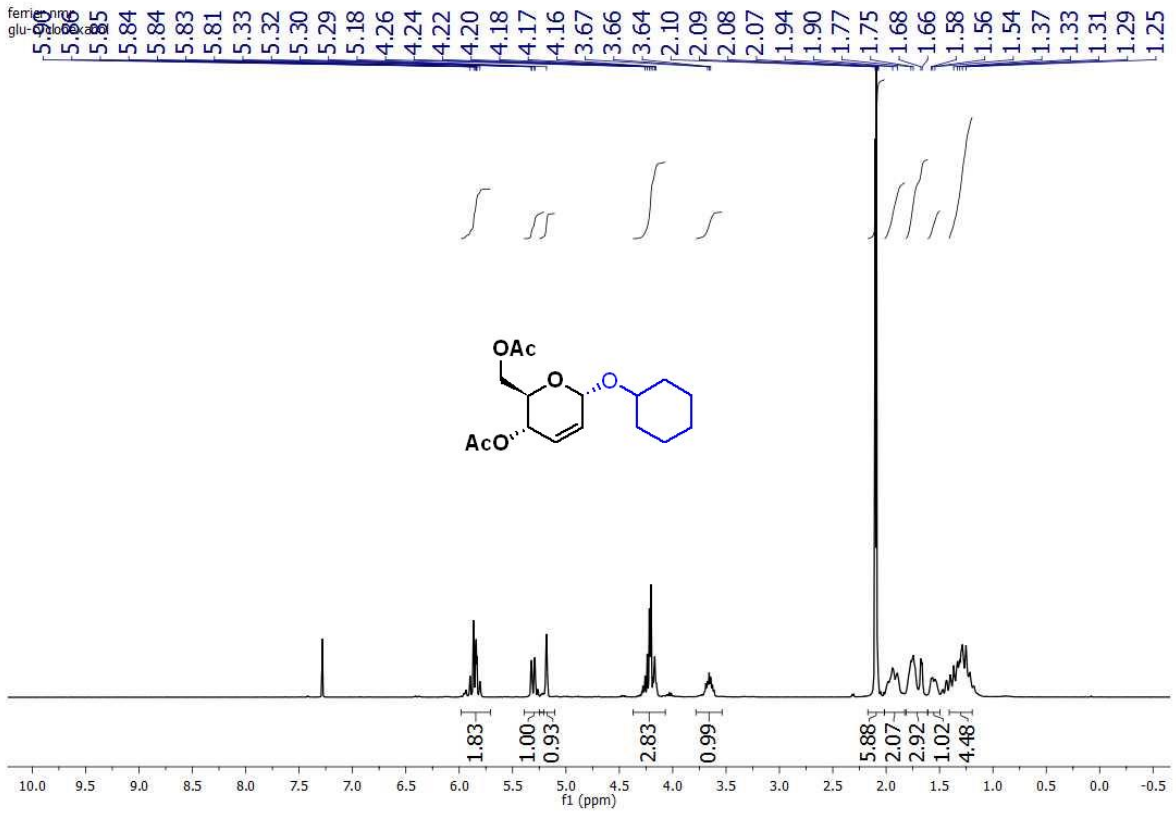
^{1}H NMR of compound 3l



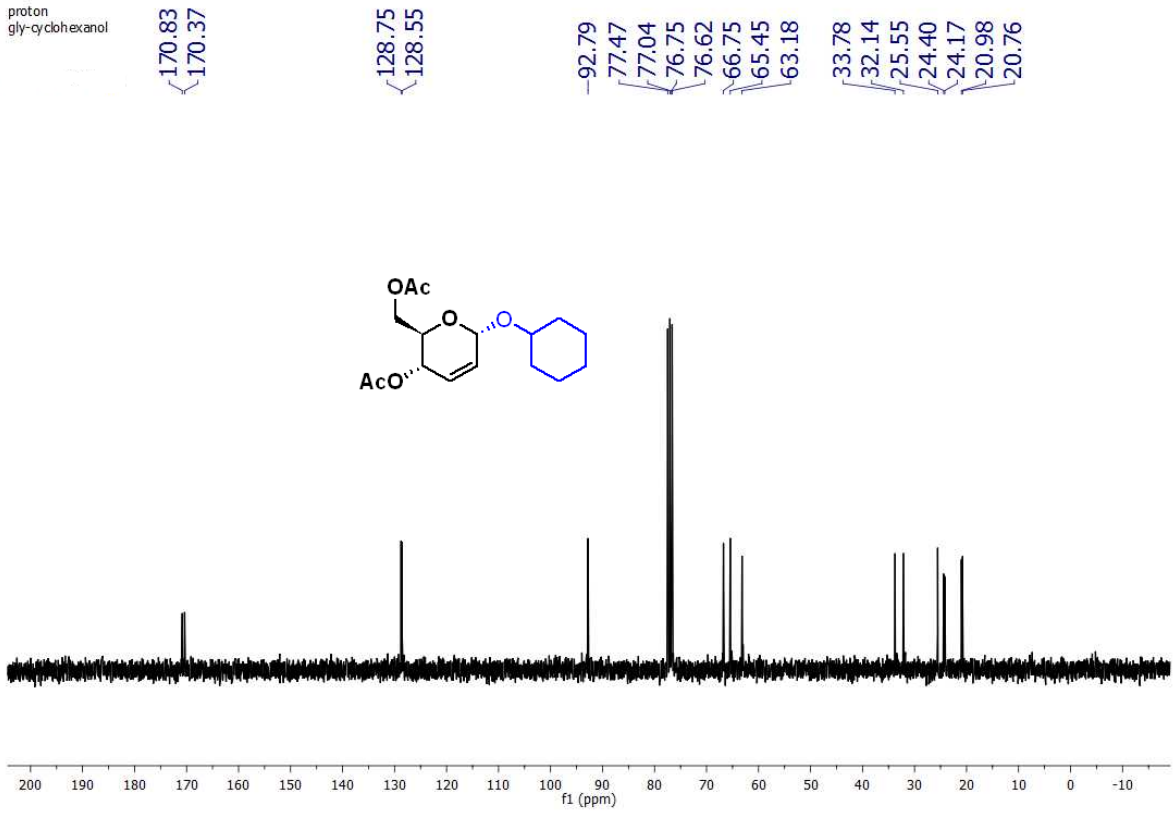
^{13}C NMR of compound 3l



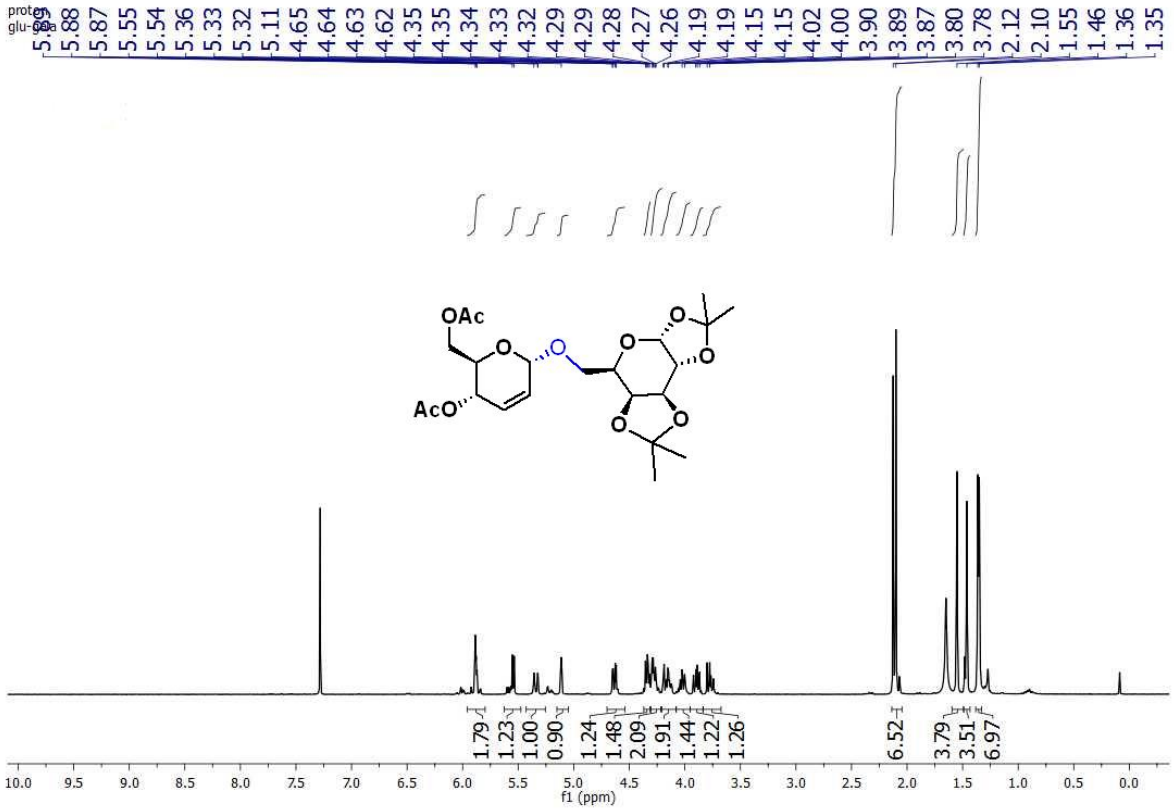
^{1}H NMR of compound 3m



^{13}C NMR of compound 3m



^{1}H NMR of compound 3n



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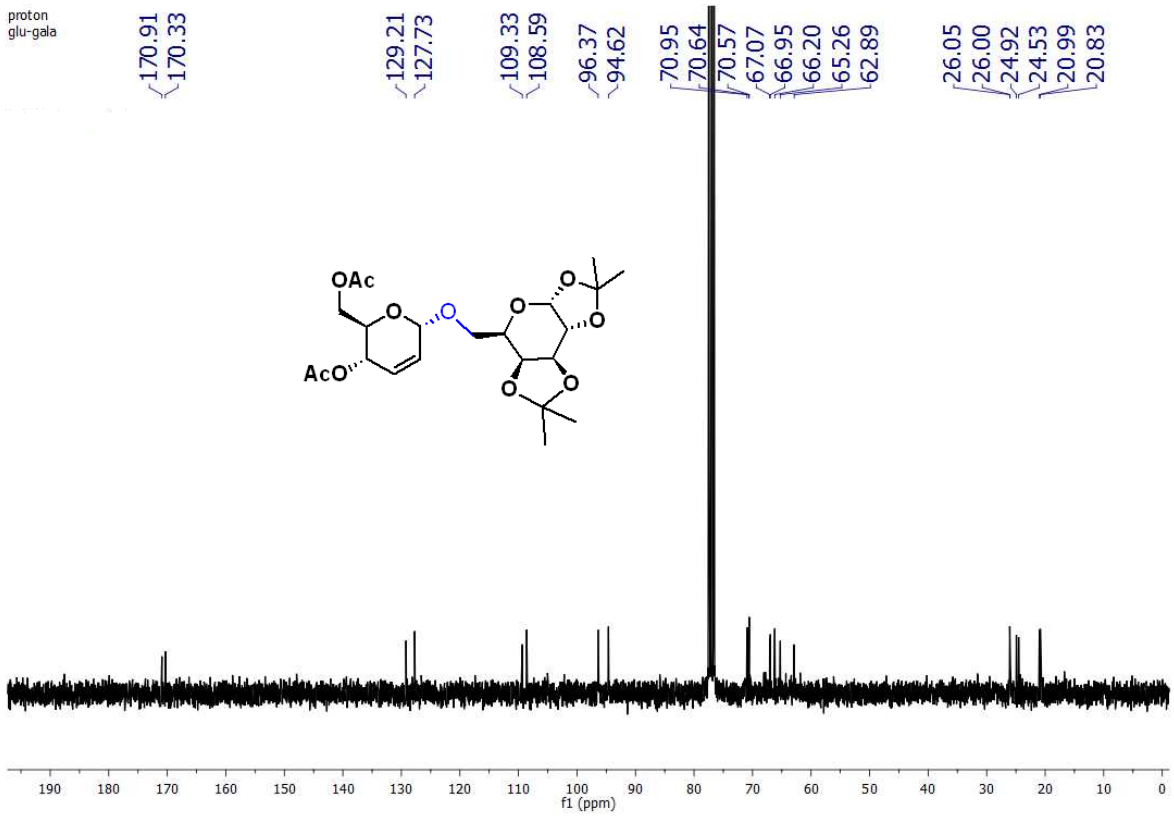
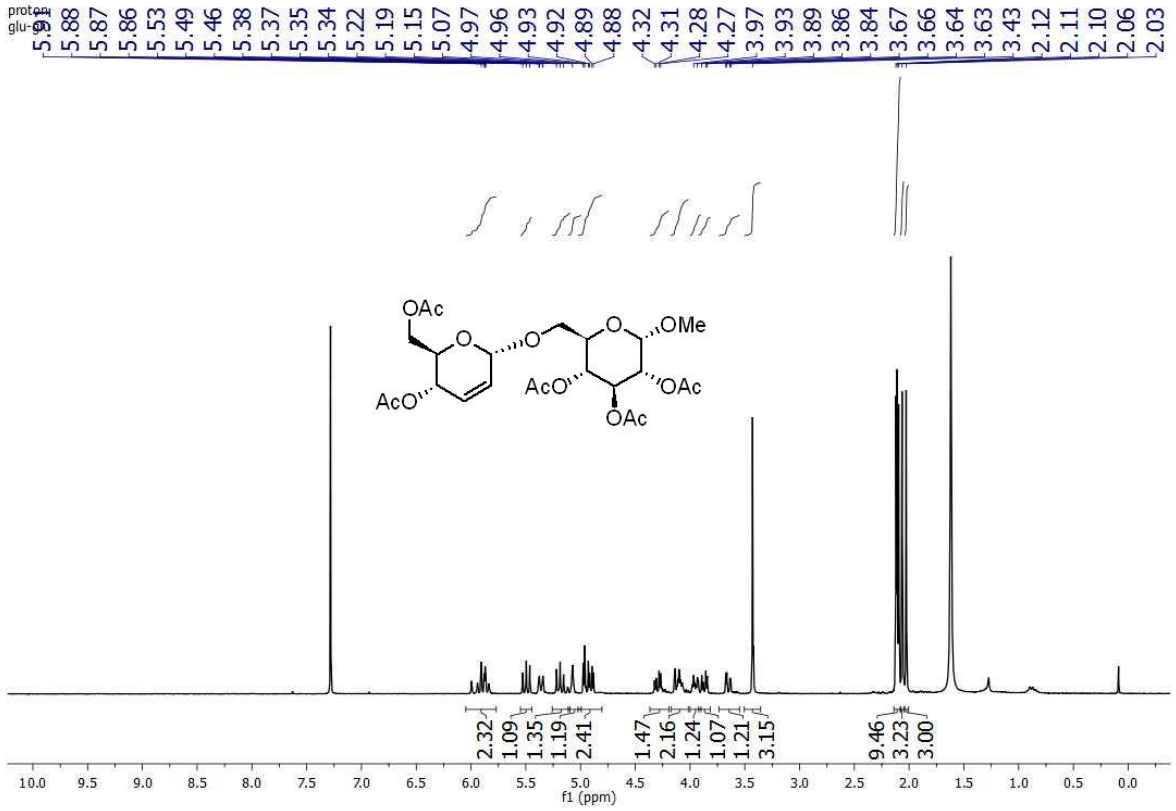
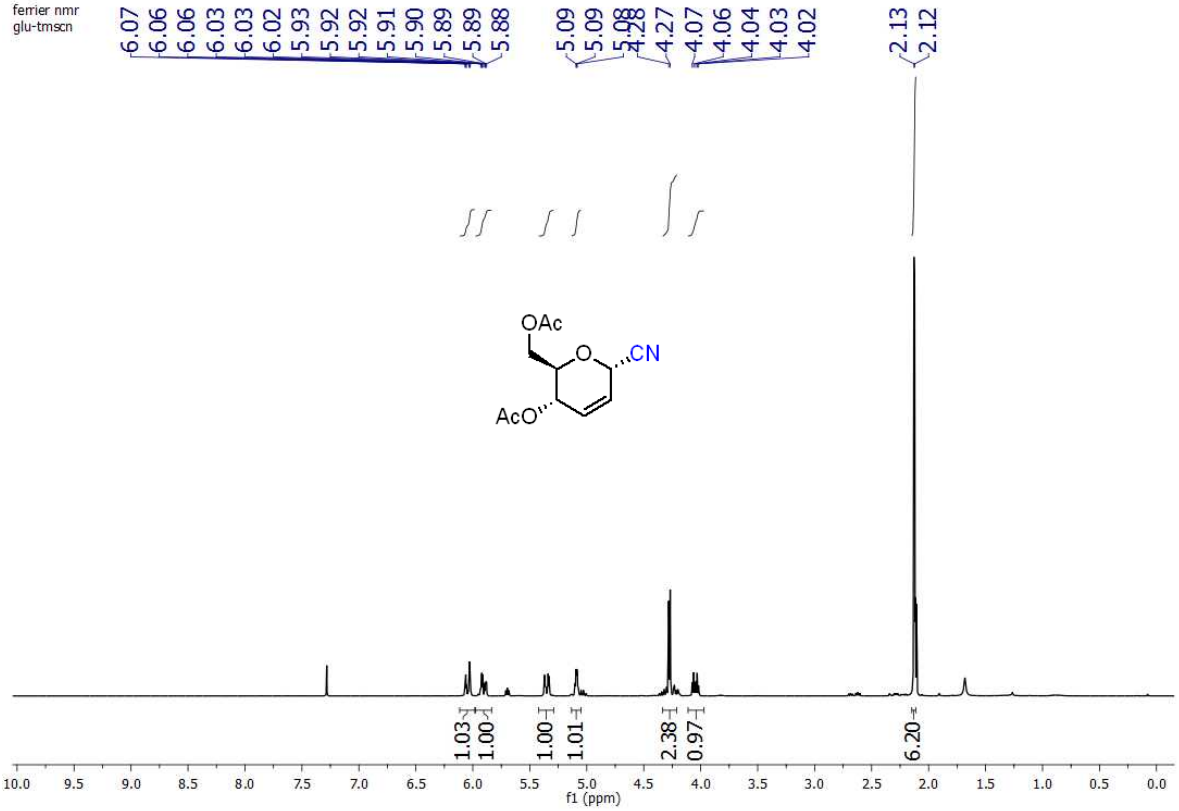


^{1}H NMR of compound 3o



^{1}H NMR of compound 3p



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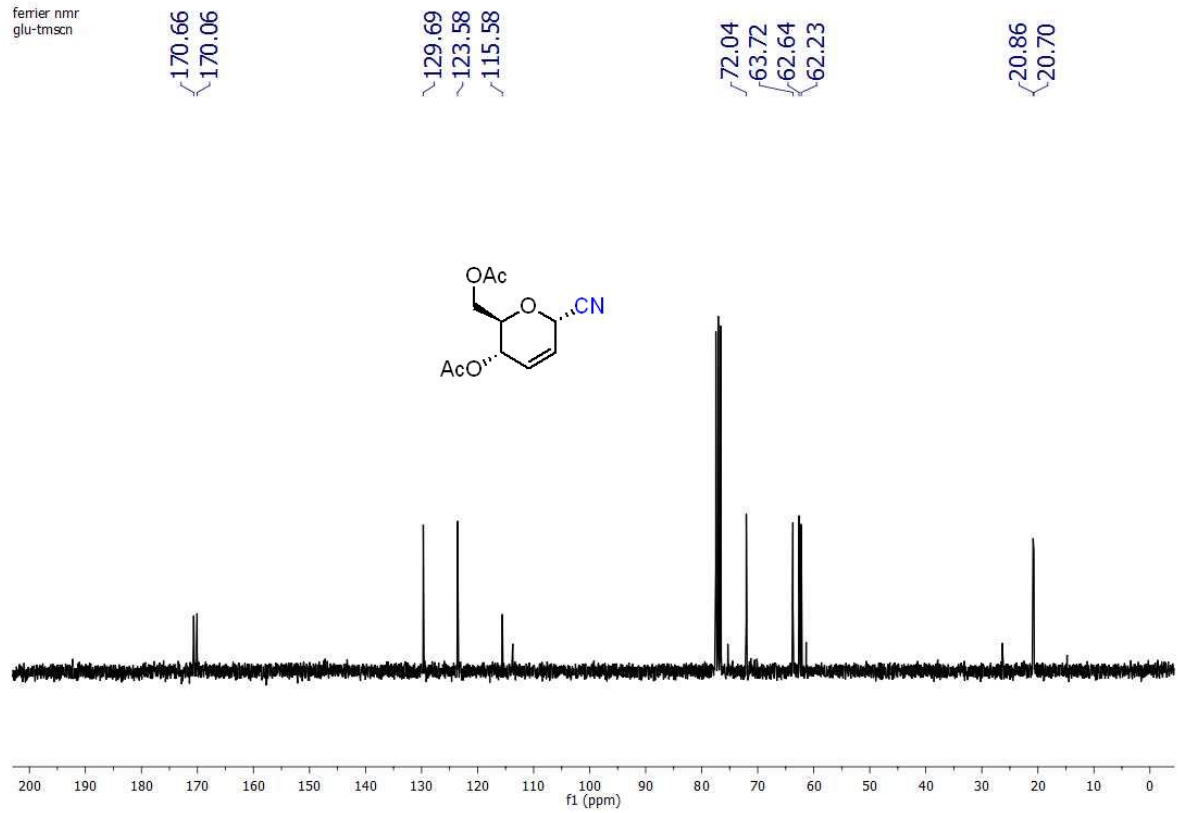
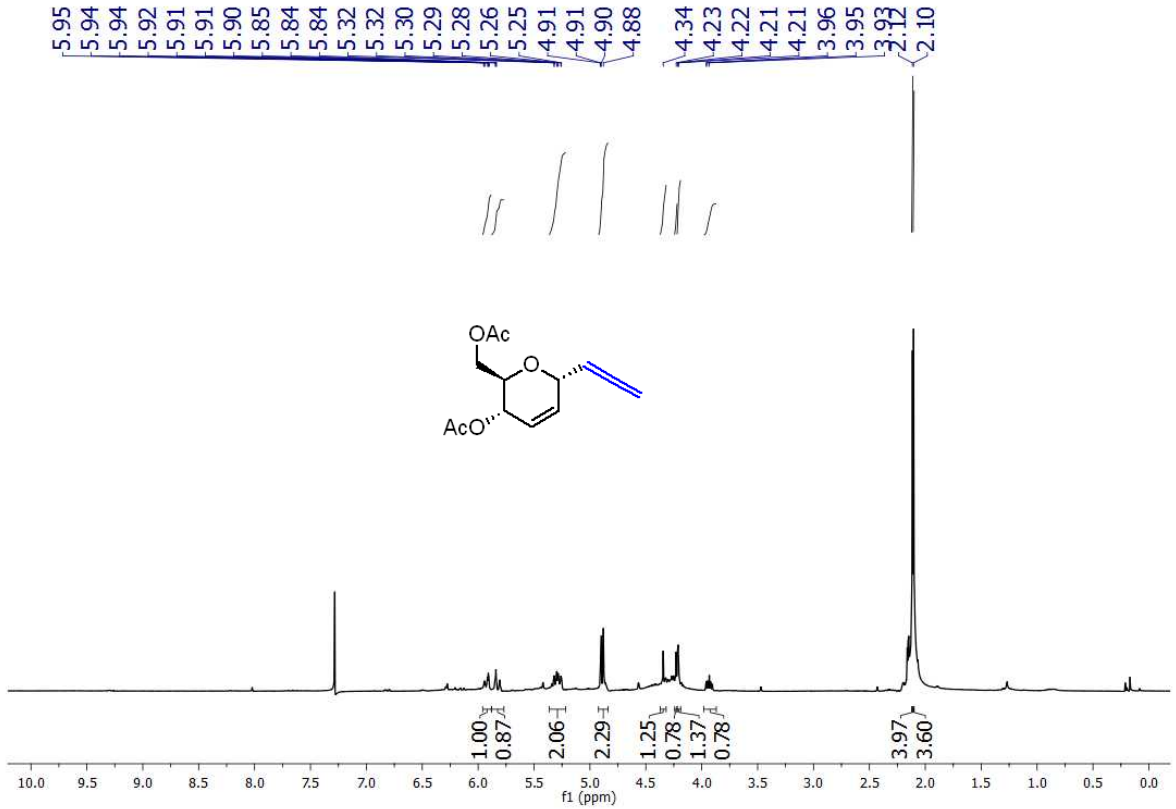
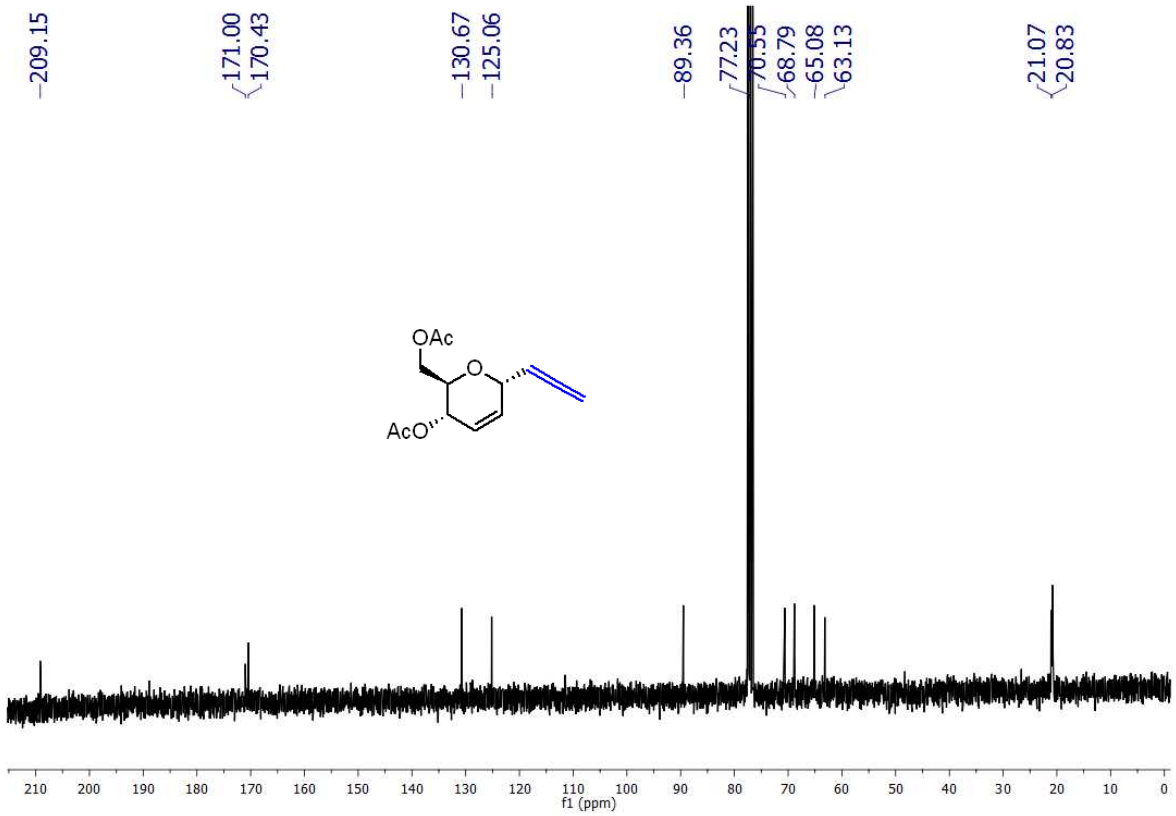


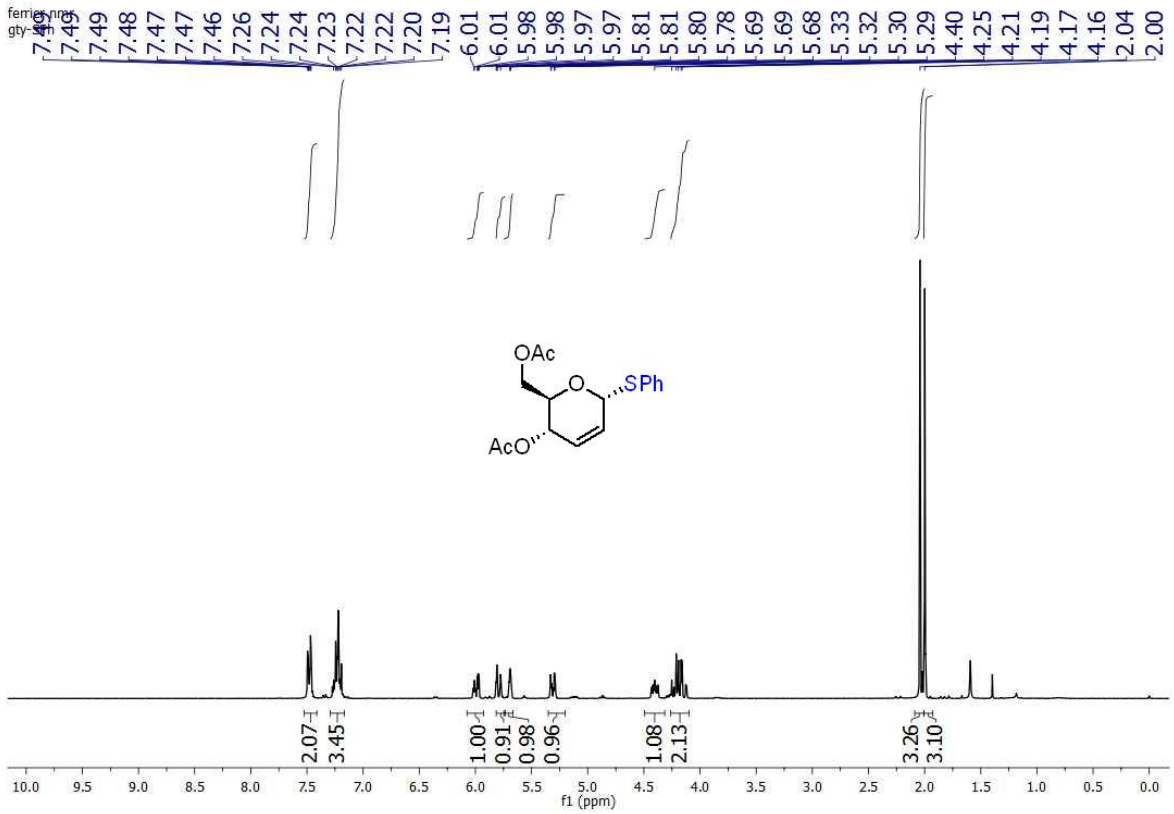
^{1}H NMR of compound 3q



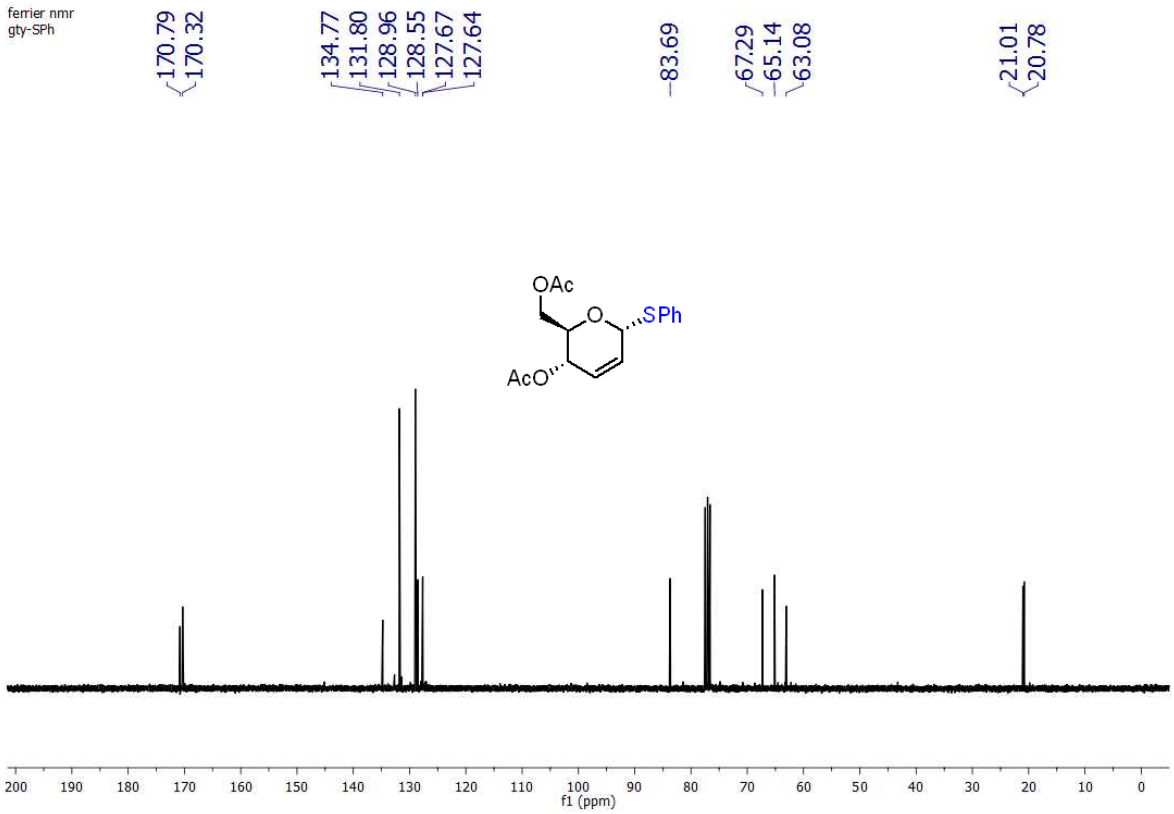
^{13}C NMR of compound 3q



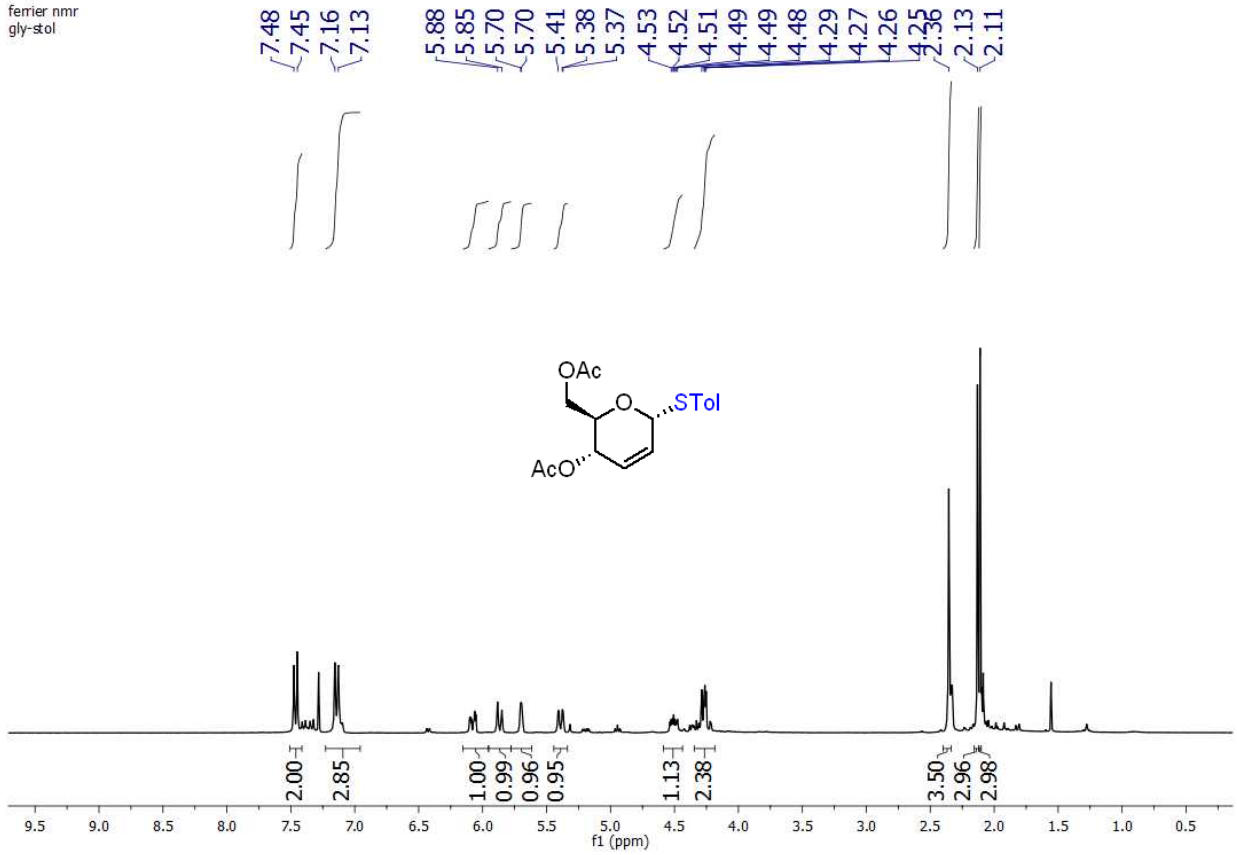
^{1}H NMR of compound 3r



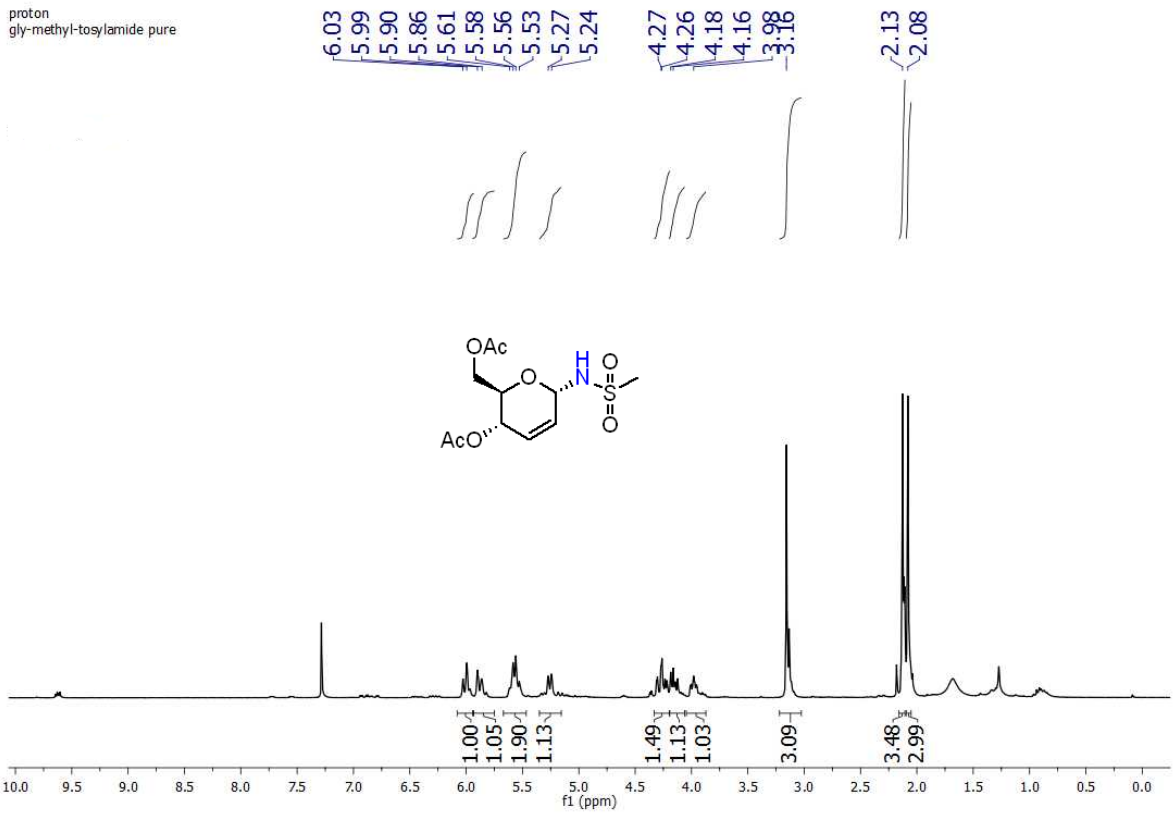
^{13}C NMR of compound 3r



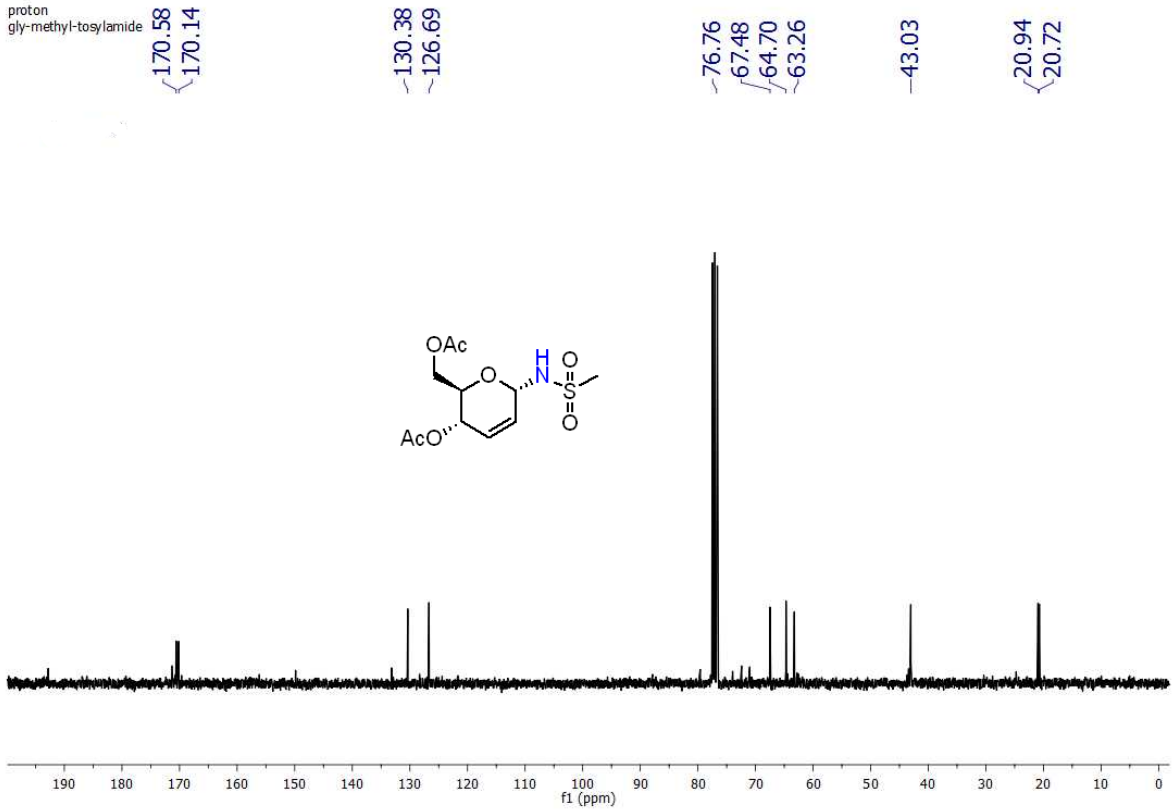
^{1}H NMR of compound 3s



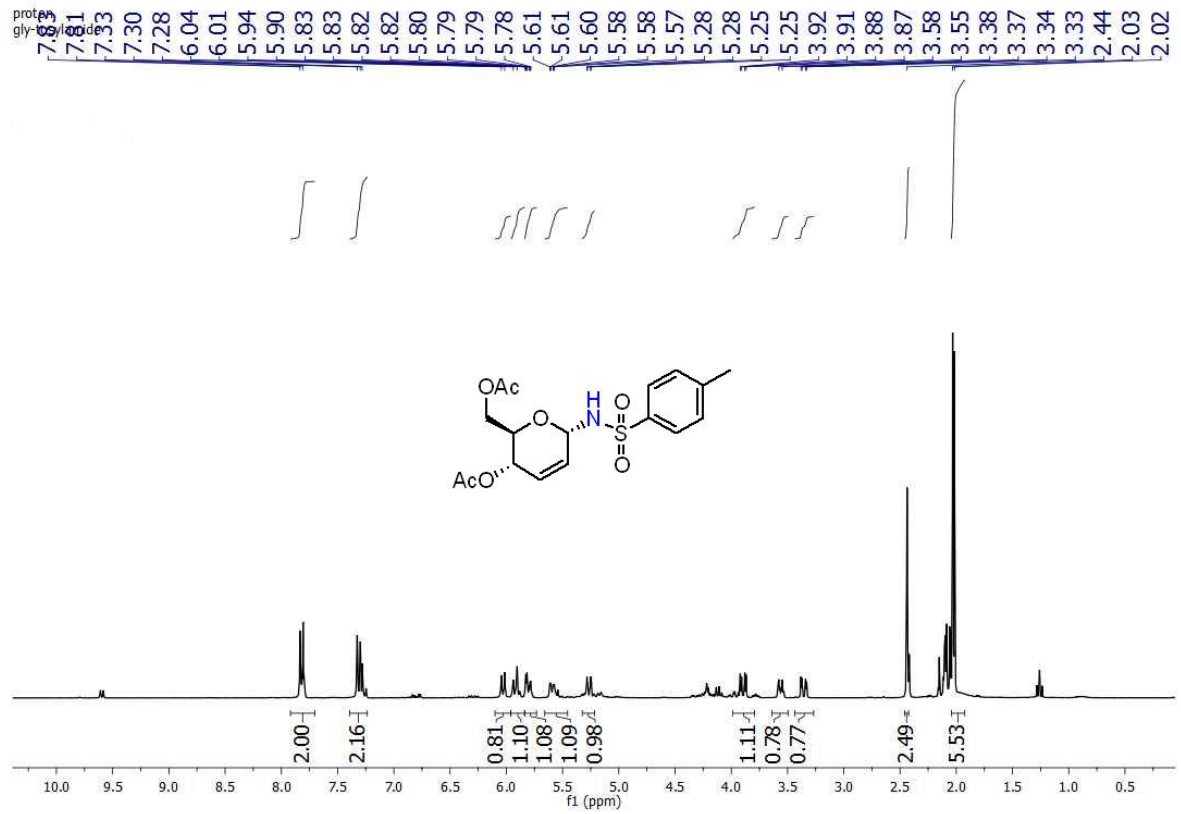
^{1}H NMR of compound 3t



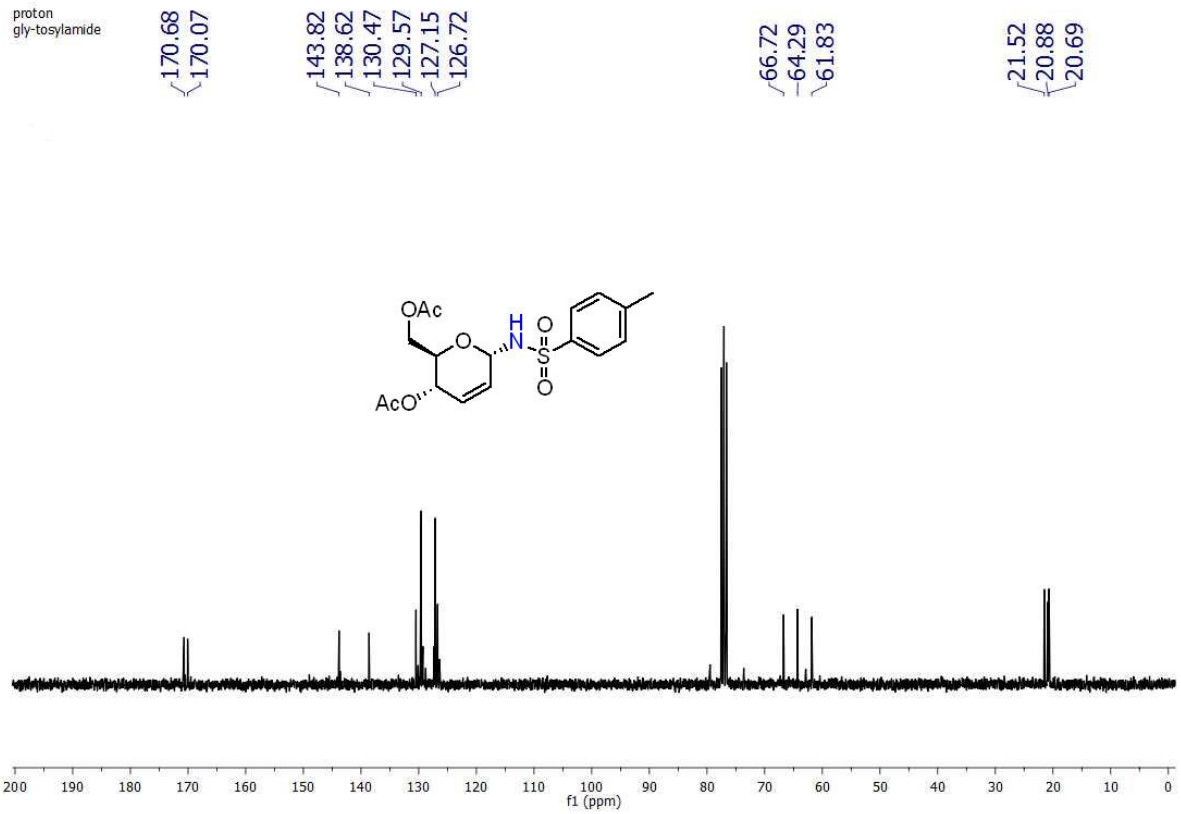
^{13}C NMR of compound 3t



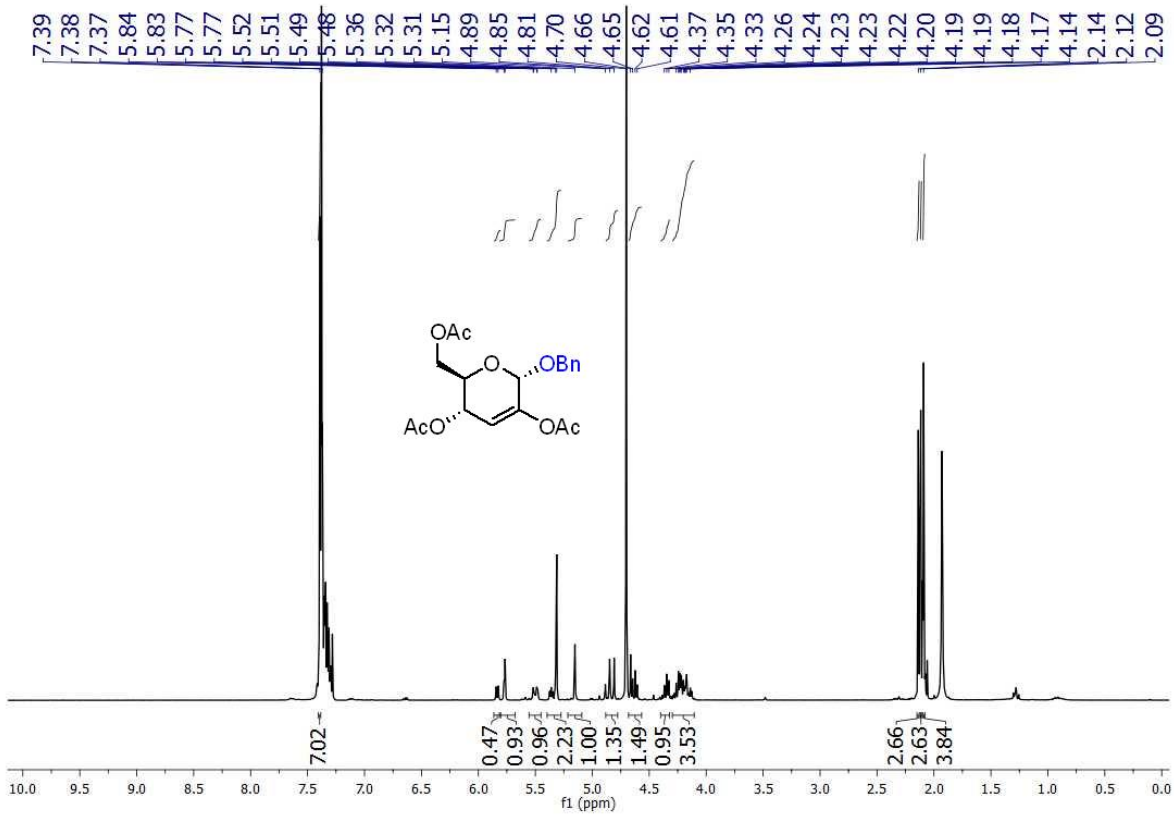
^{1}H NMR of compound 3u



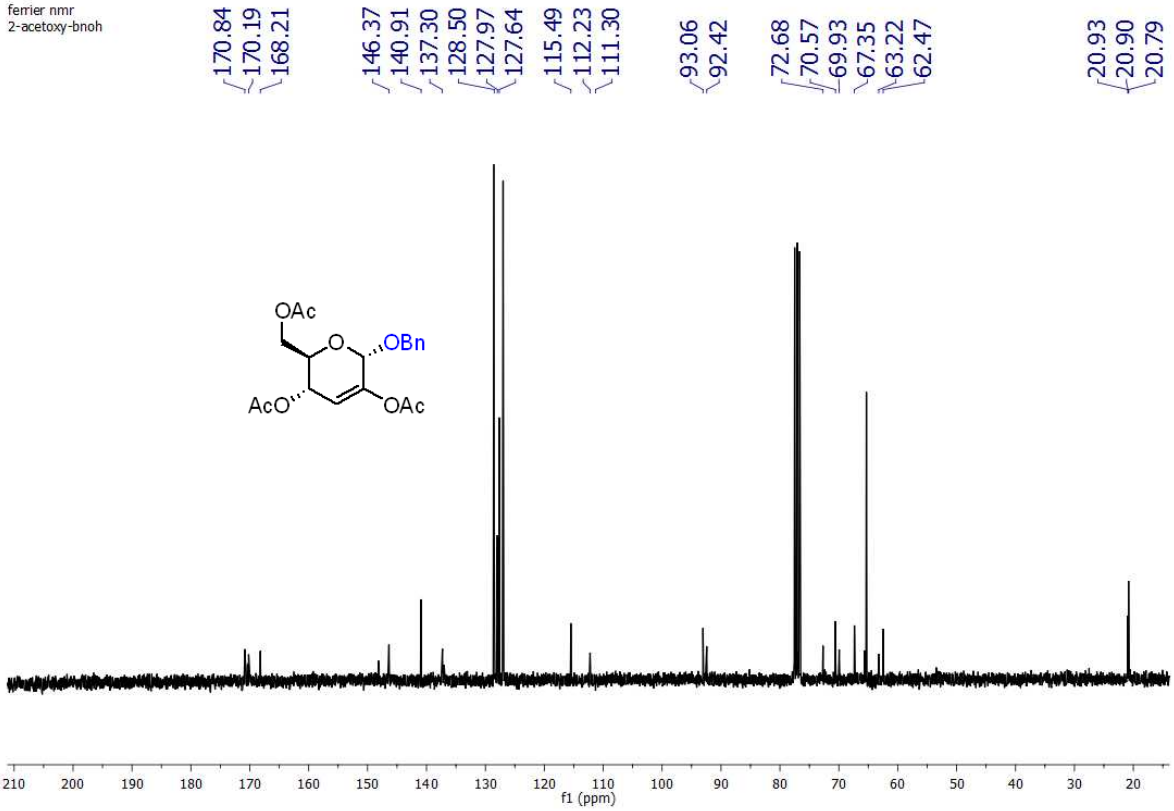
^{13}C NMR of compound 3u



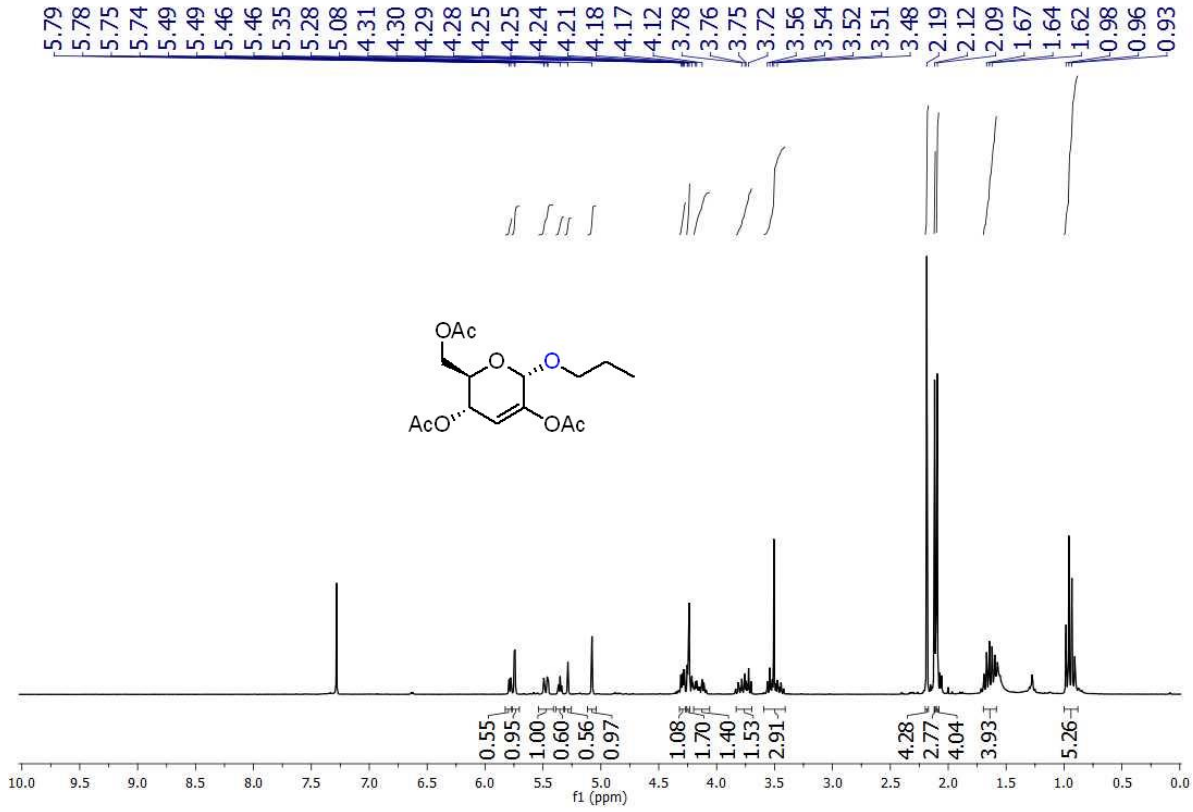
^{1}H NMR of compound 5a



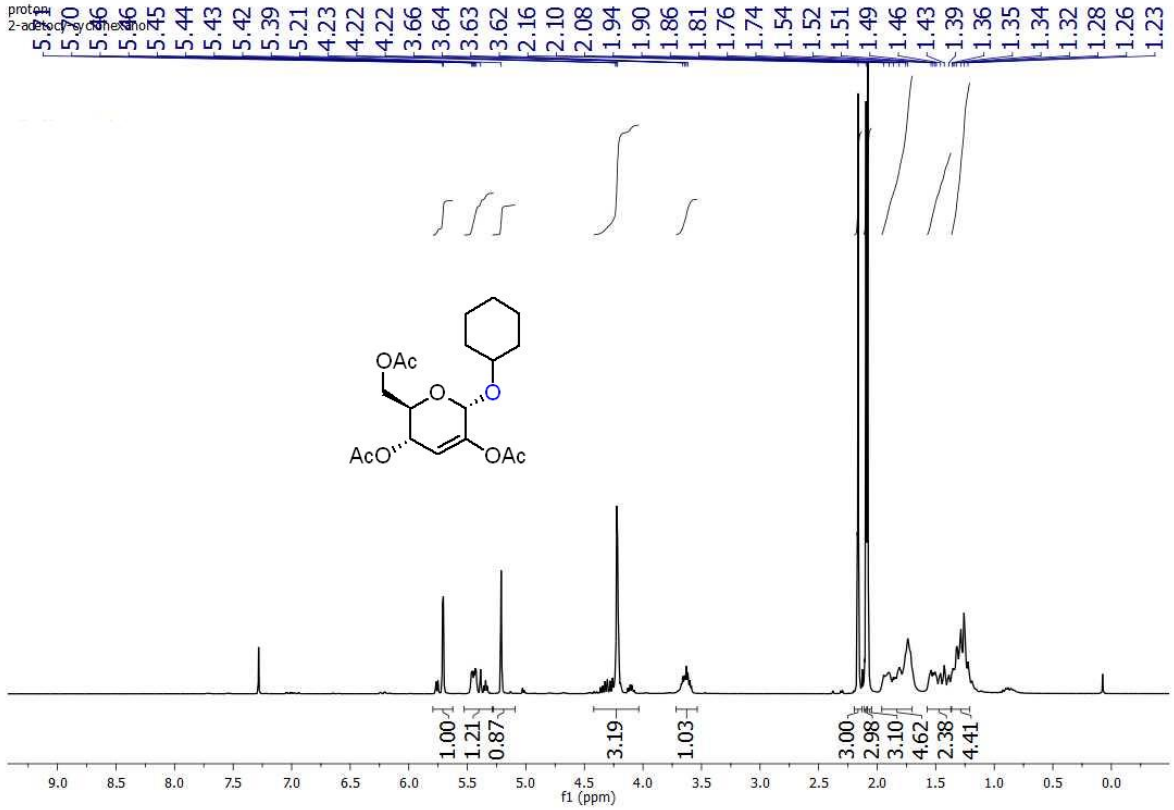
^{13}C NMR of compound 5a



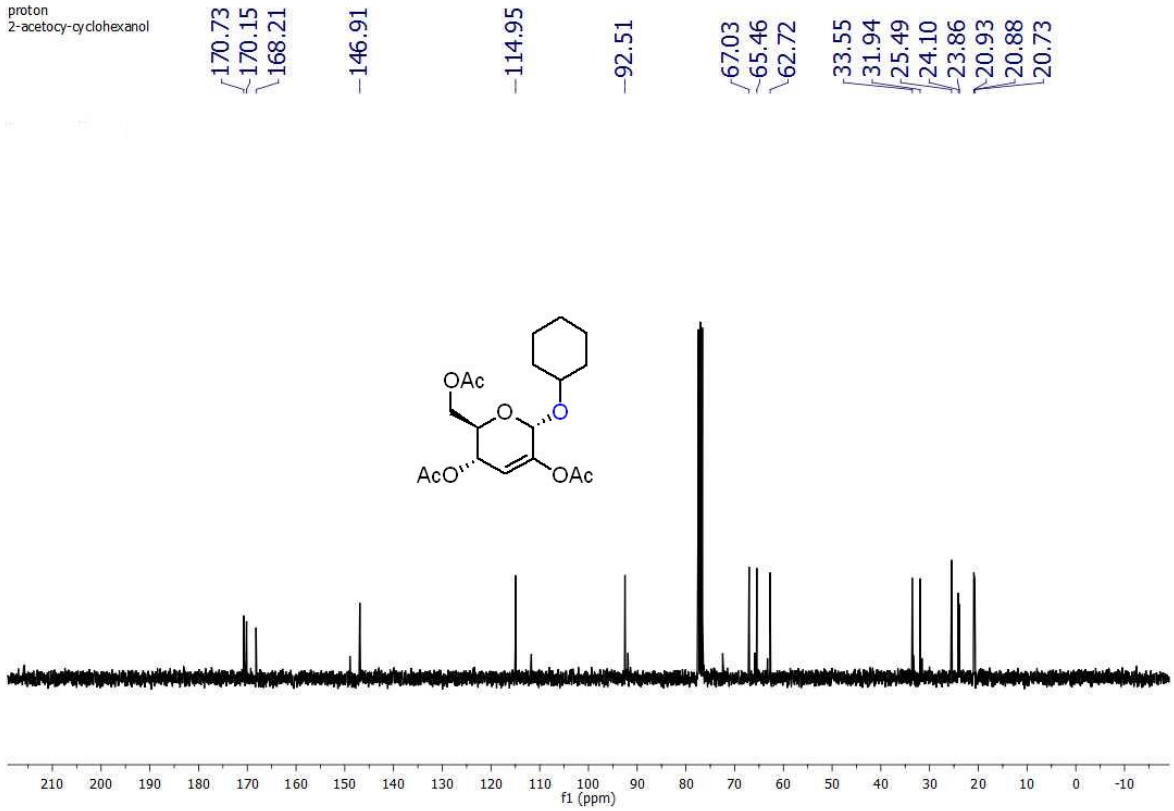
^{1}H NMR of compound 5b



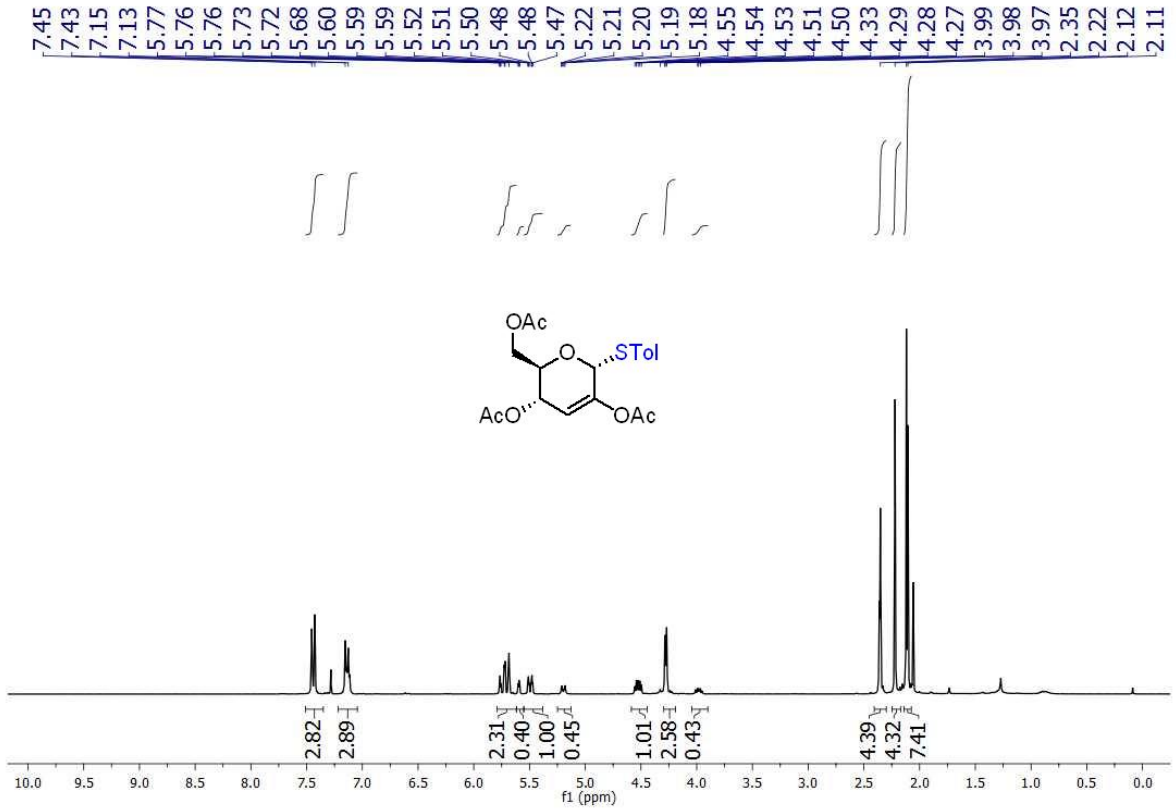
^{1}H NMR of compound 5c



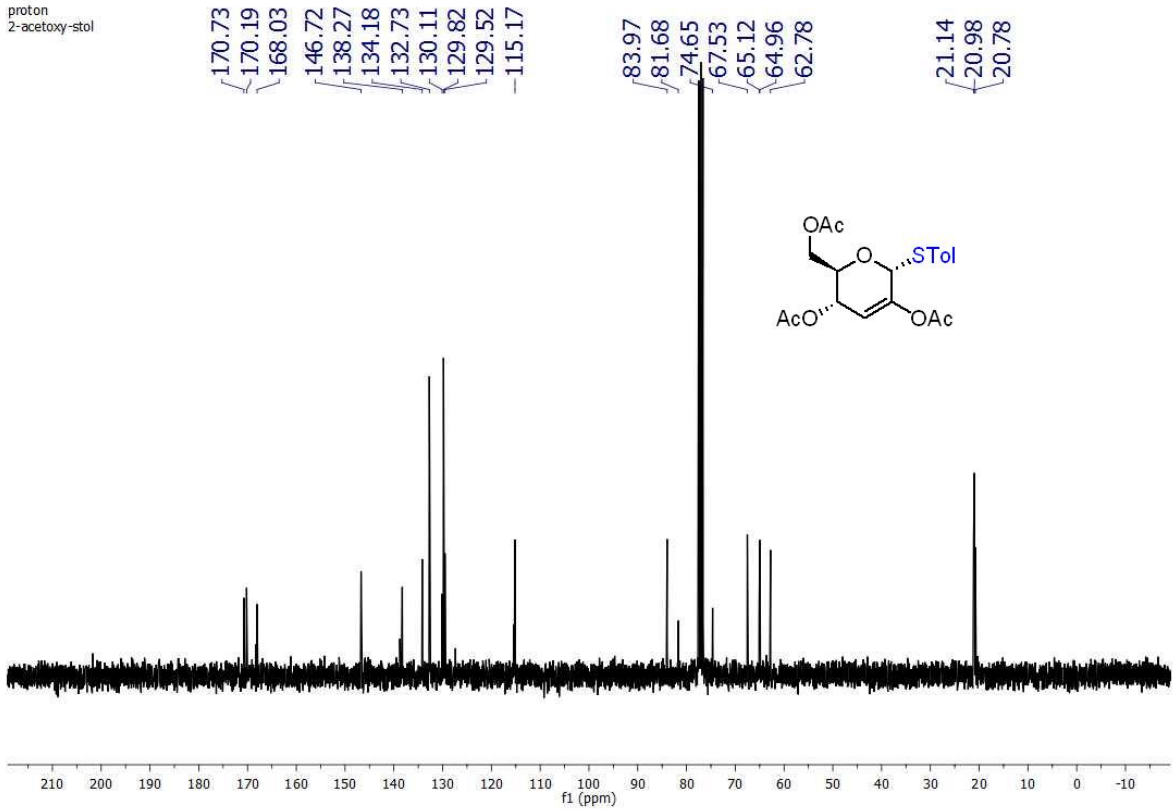
^{13}C NMR of compound 5c



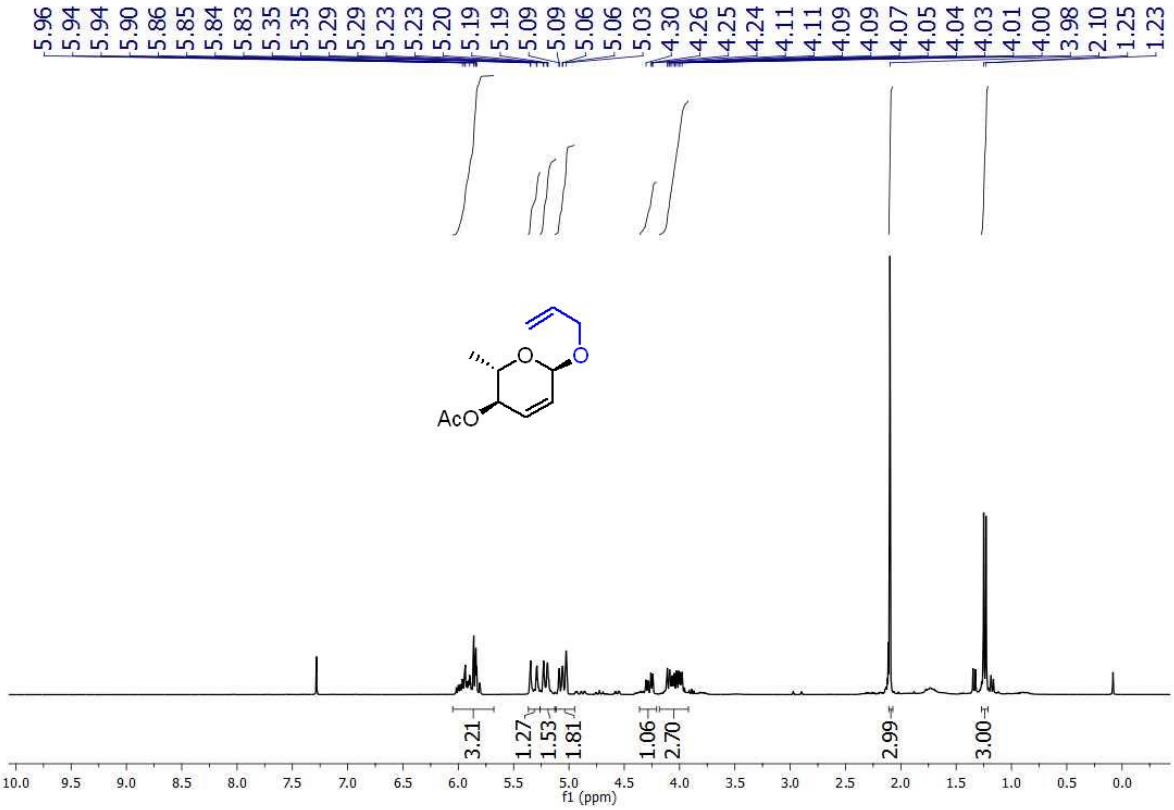
^{1}H NMR of compound 5d



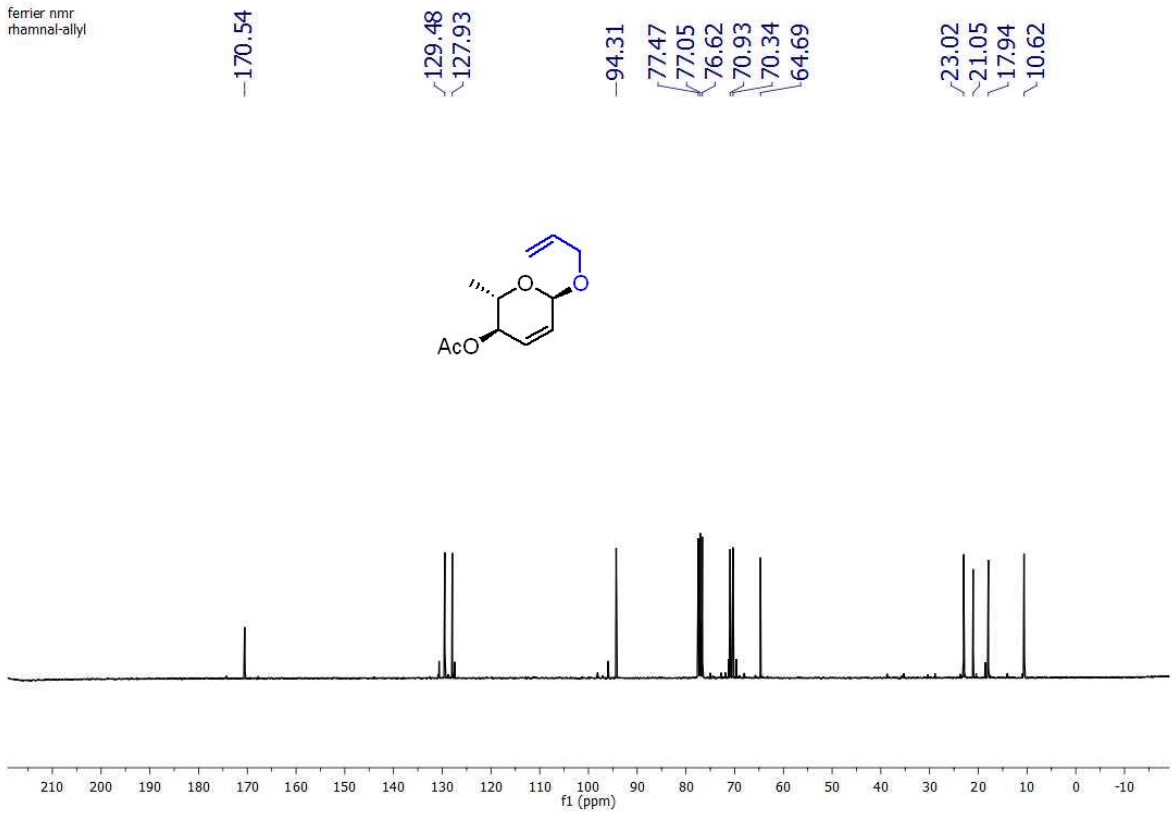
^{13}C NMR of compound 5d



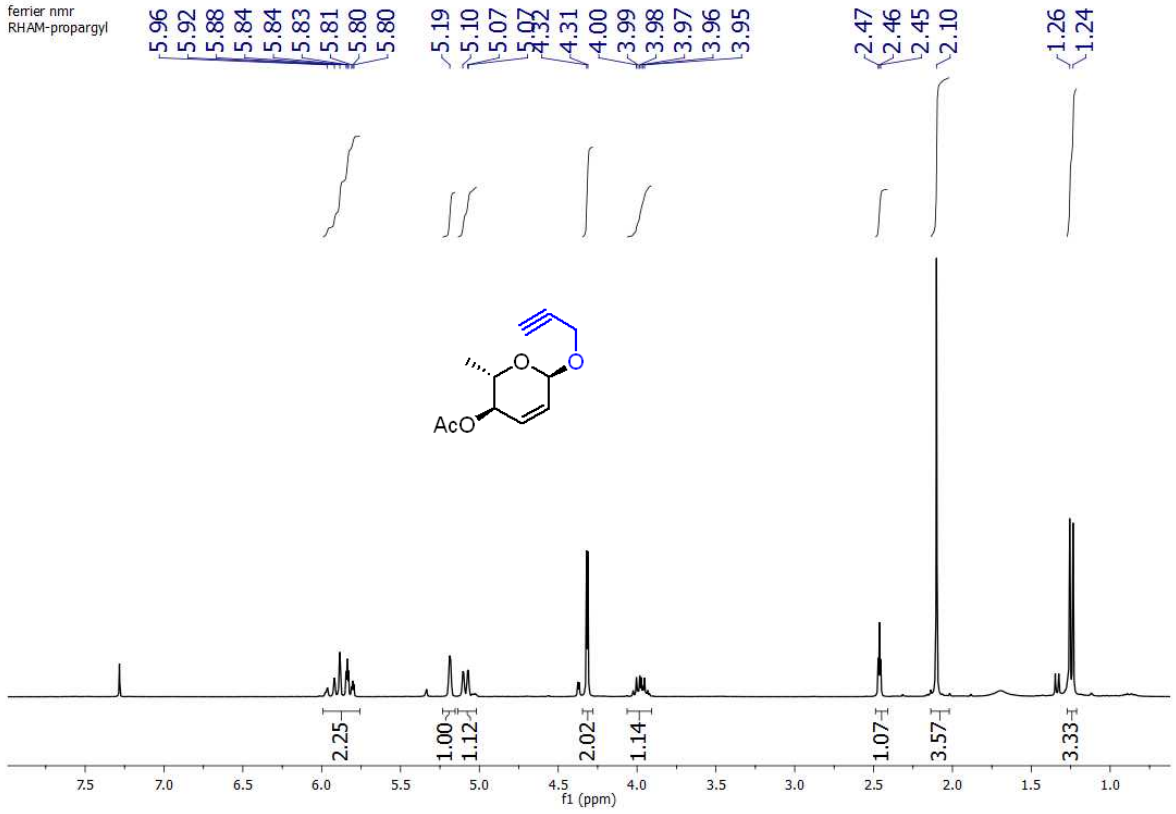
^{1}H NMR of compound 7a



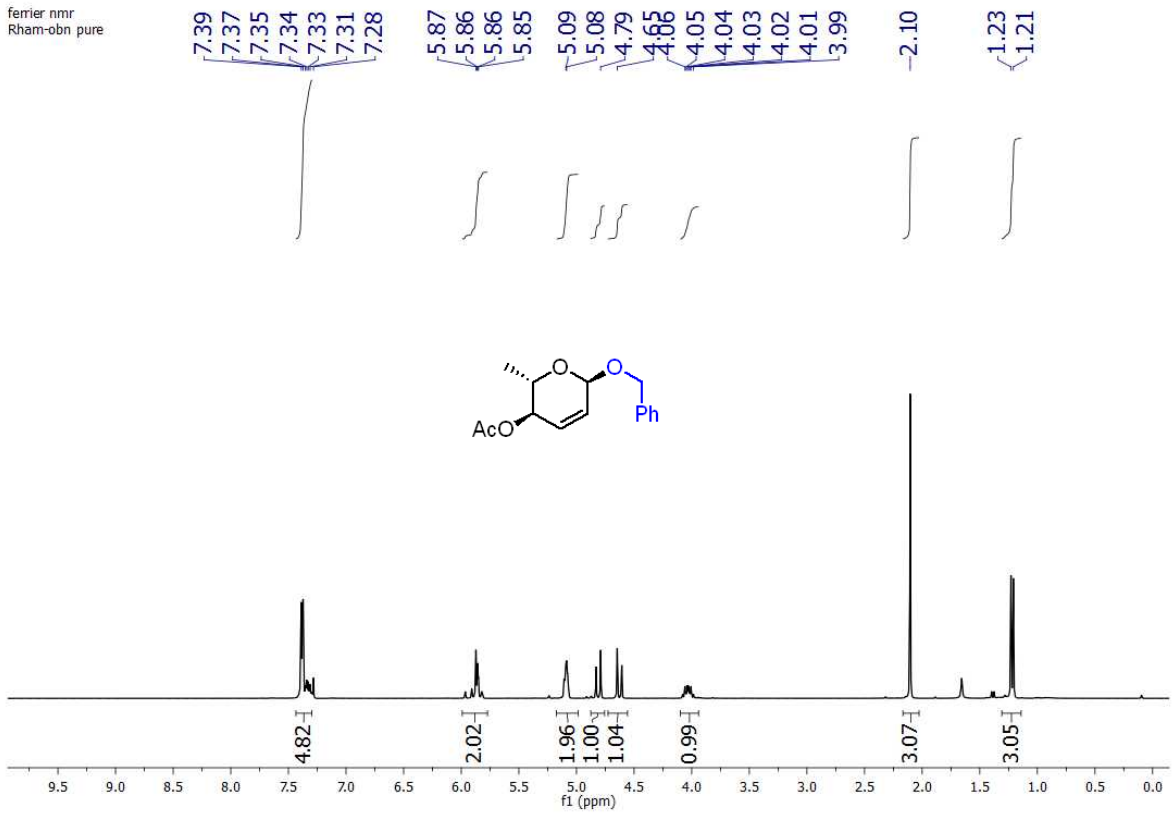
^{13}C NMR of compound 7a



^{1}H NMR of compound 7b



^{1}H NMR of compound 7c



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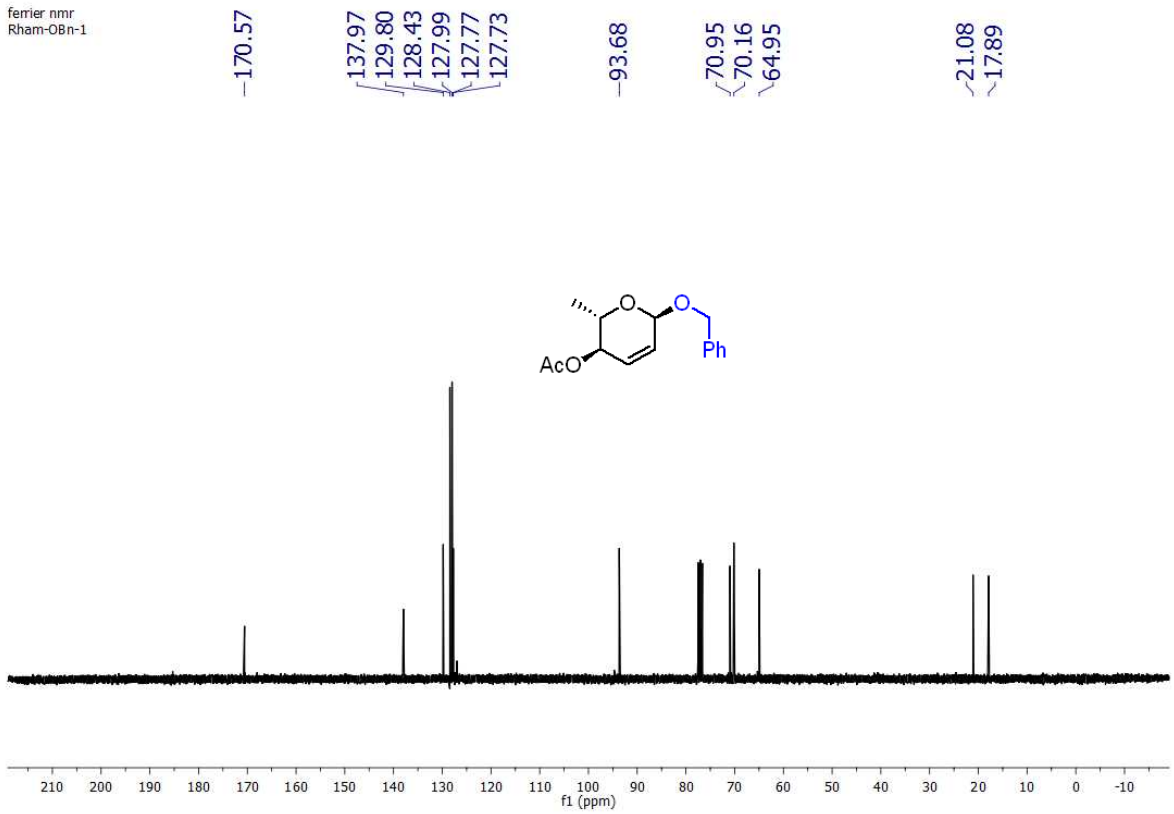
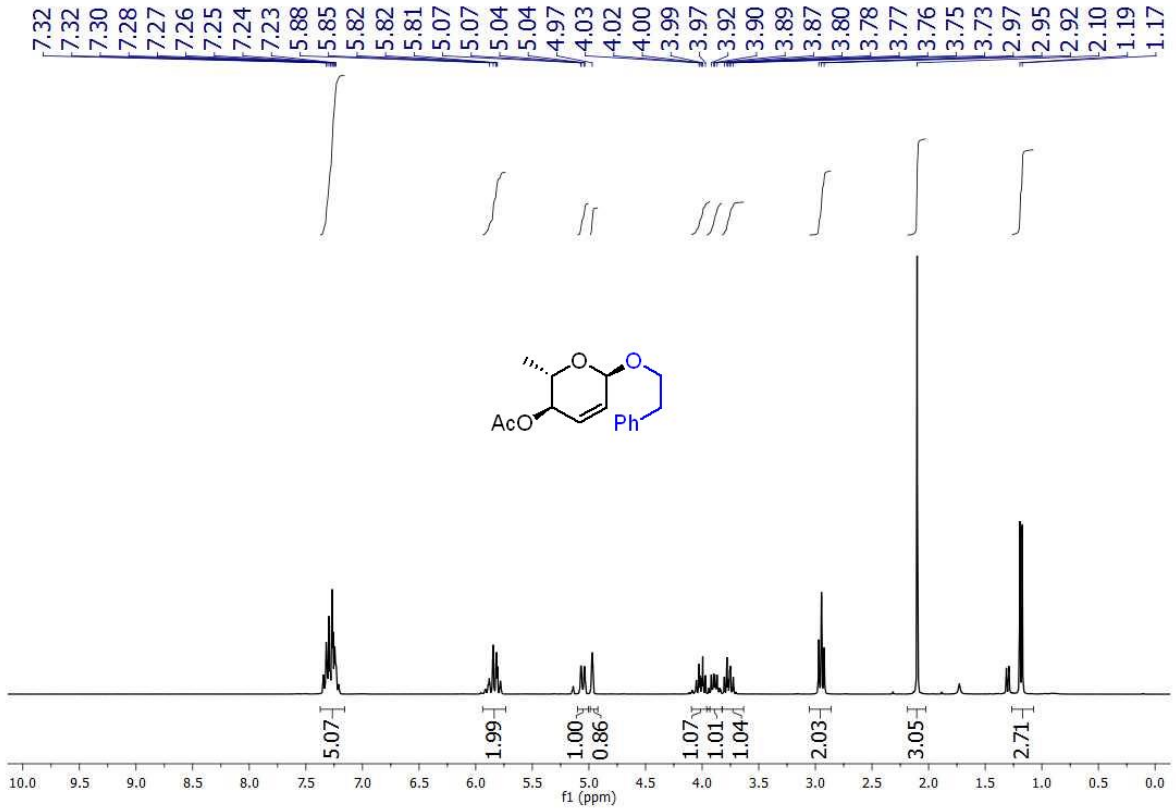
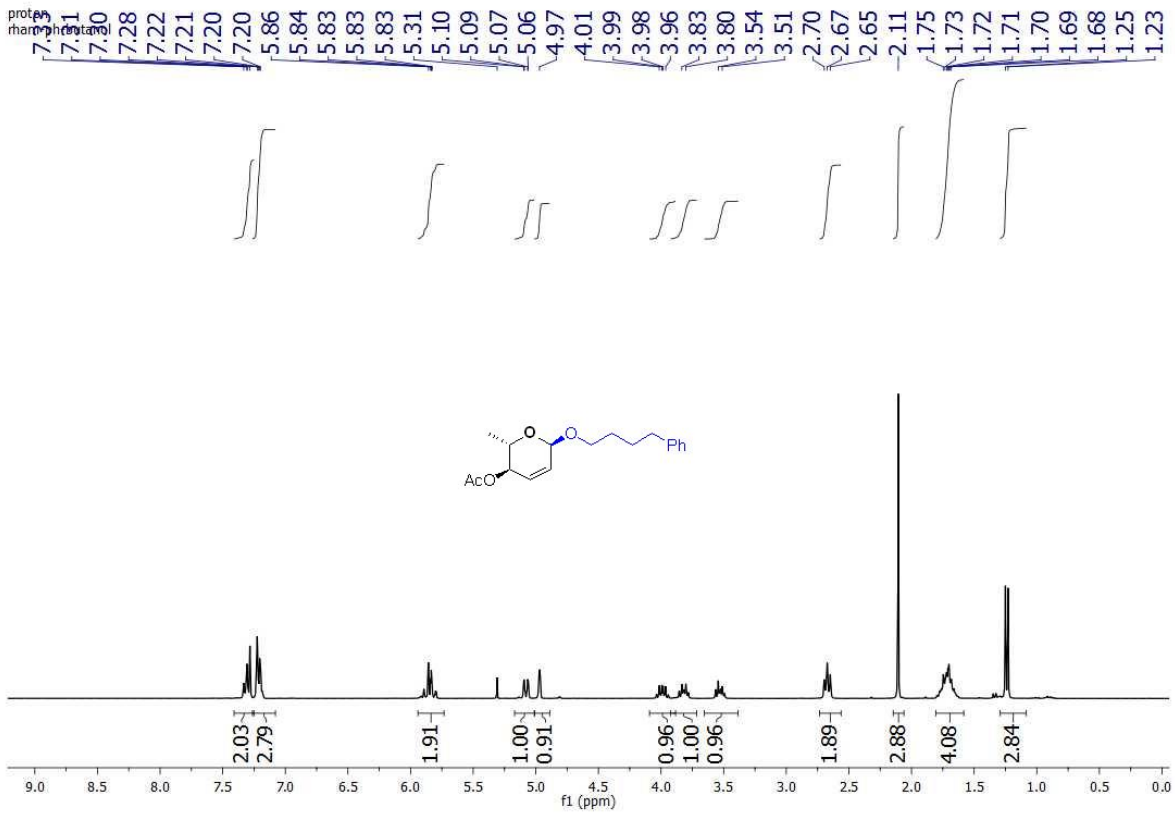


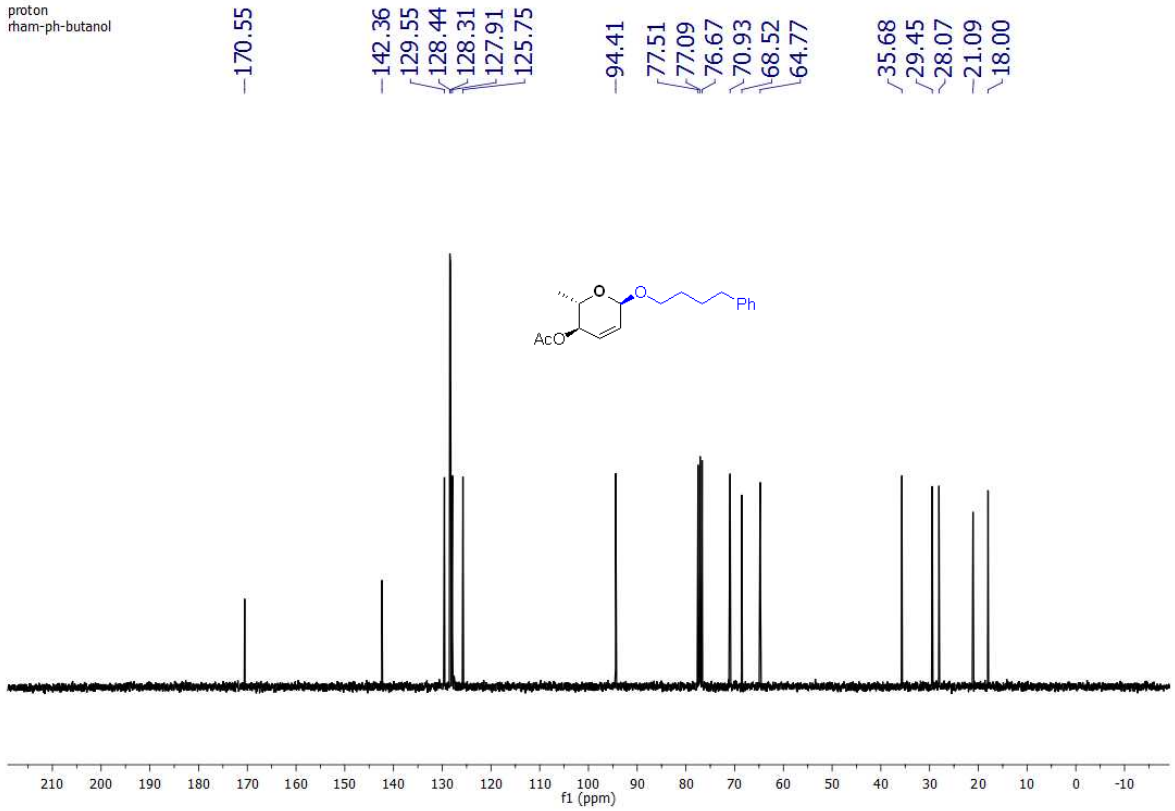
^{1}H NMR of compound 7d



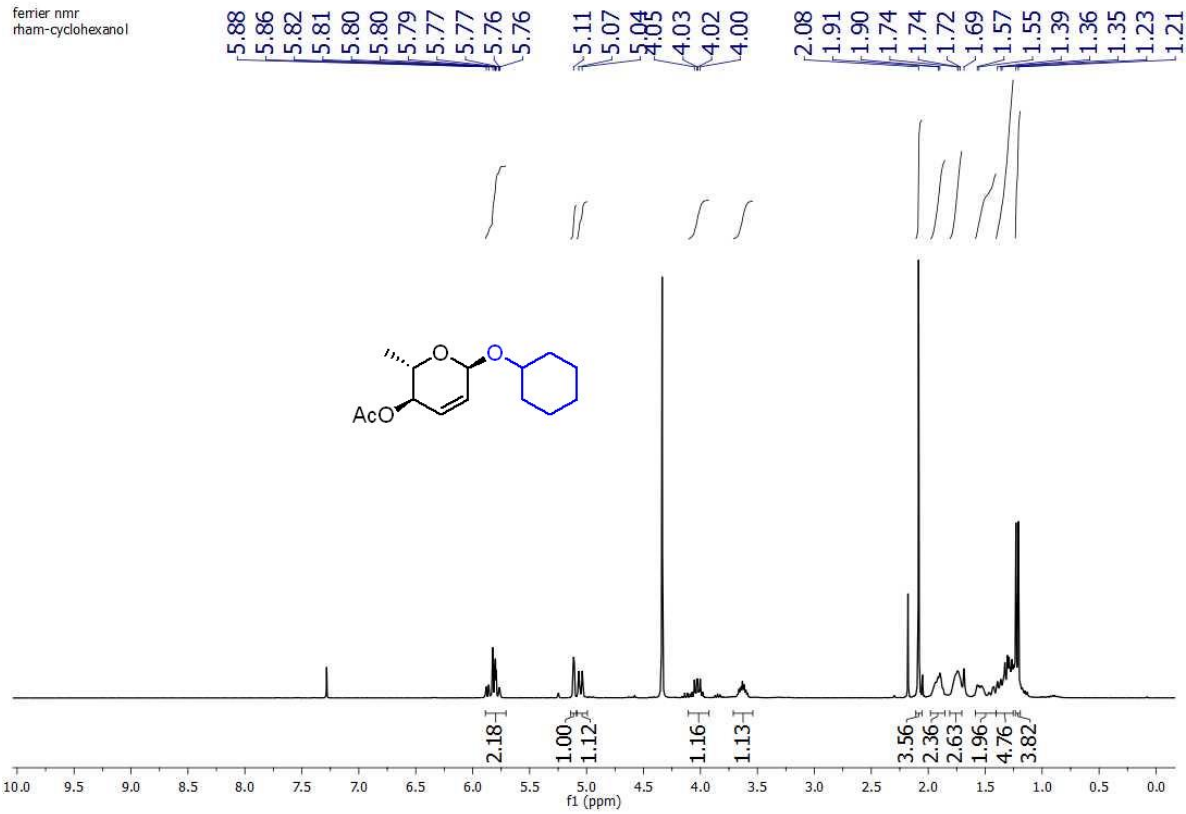
^{1}H NMR of compound 7e



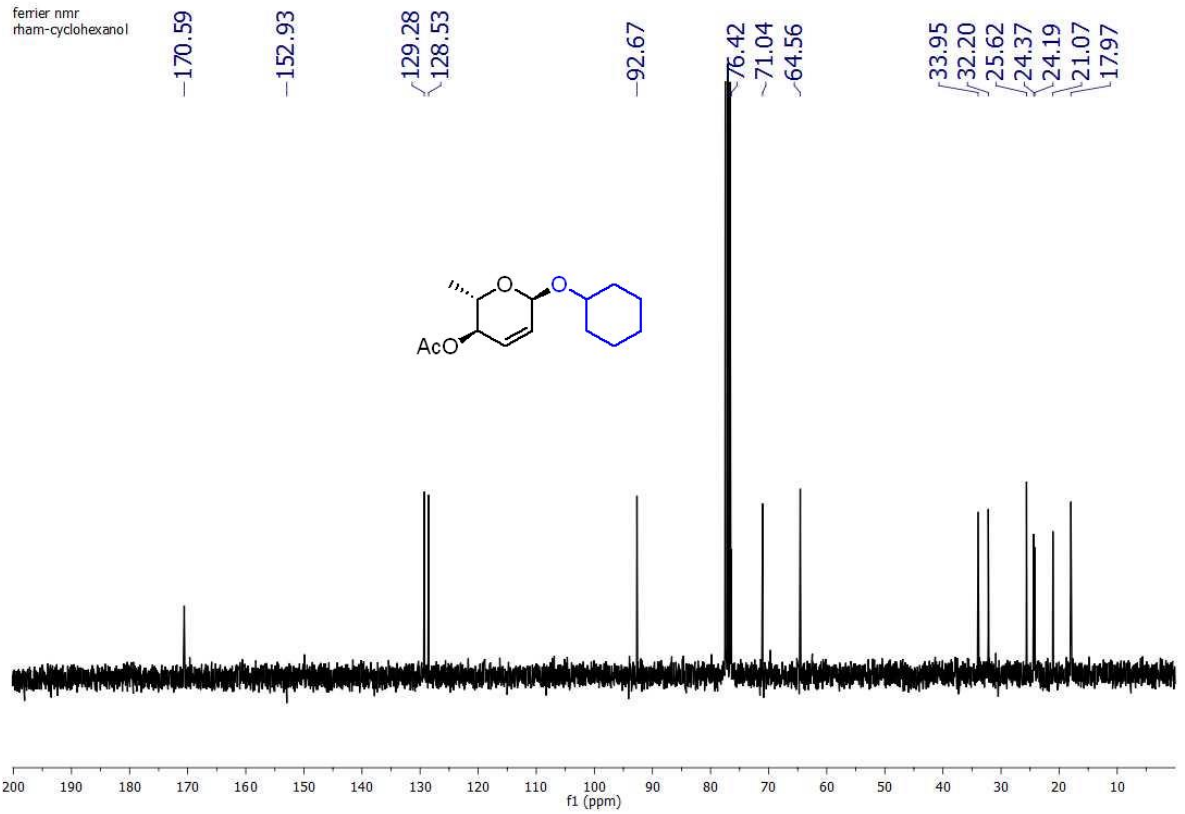
^{13}C NMR of compound 7e



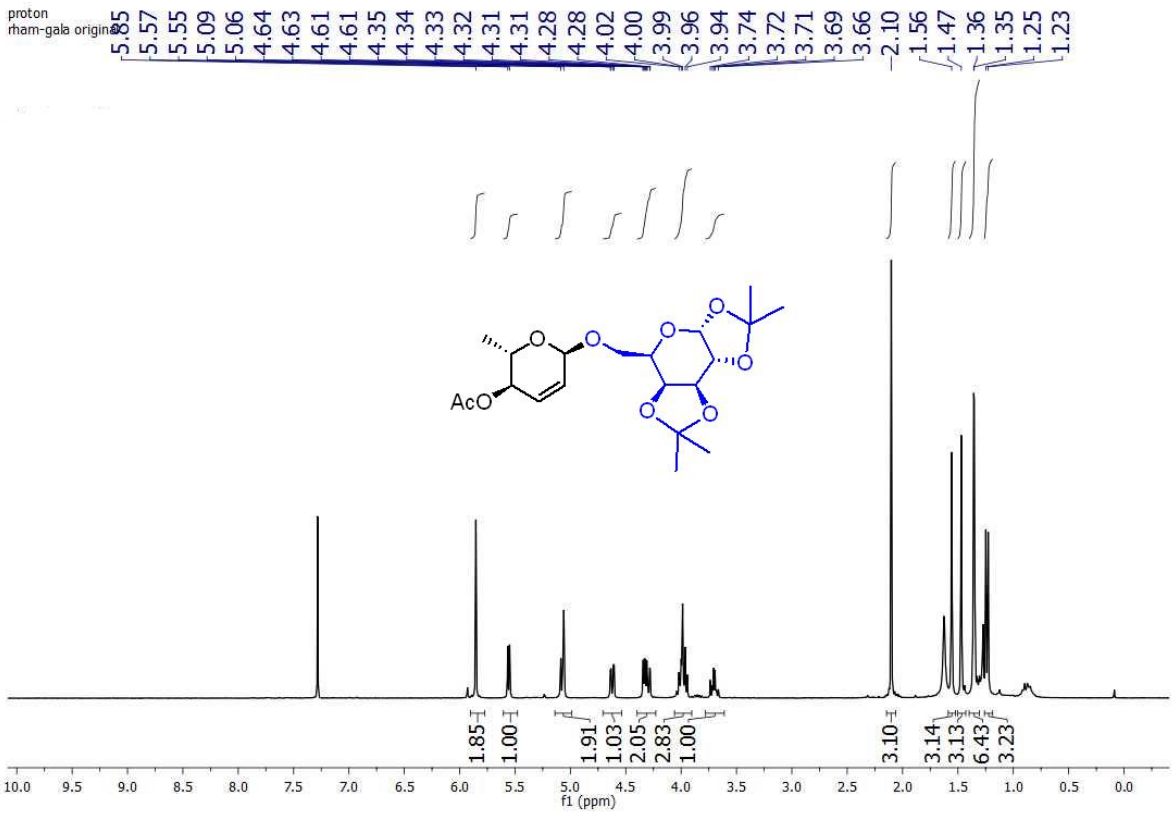
^{1}H NMR of compound 7f



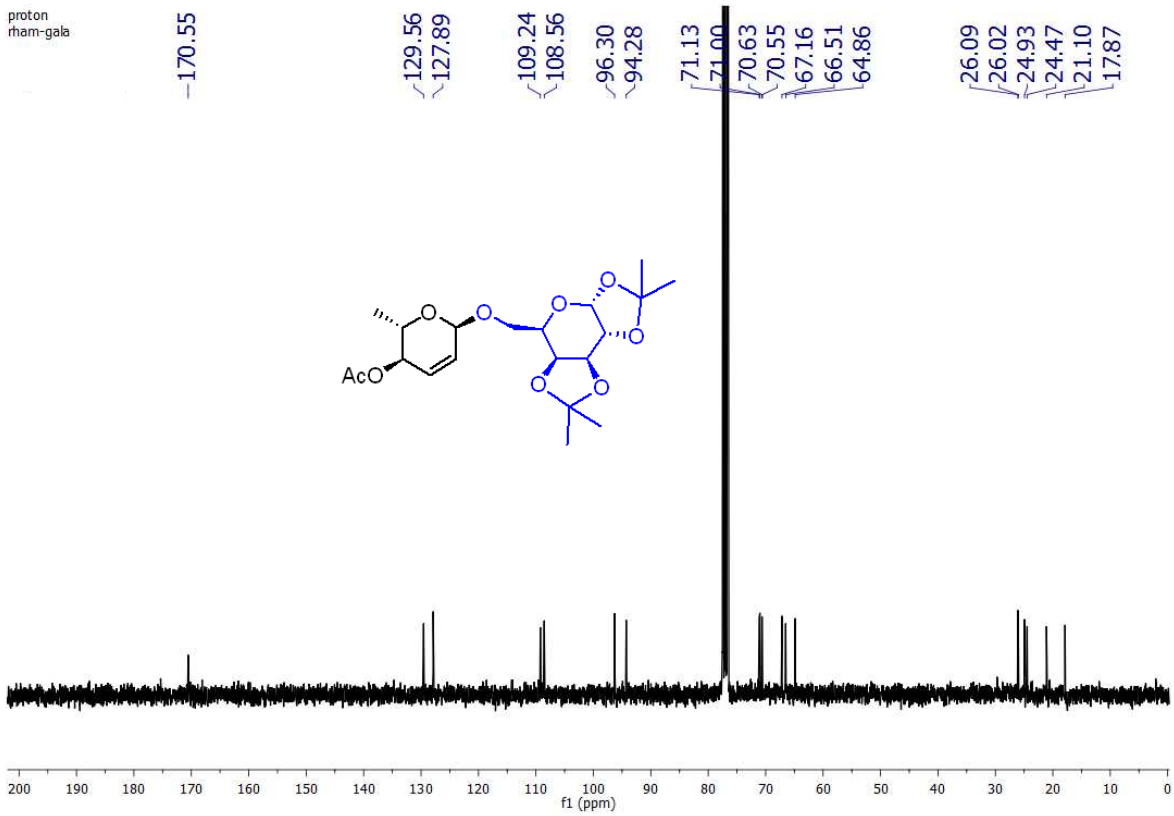
^{13}C NMR of compound 7f



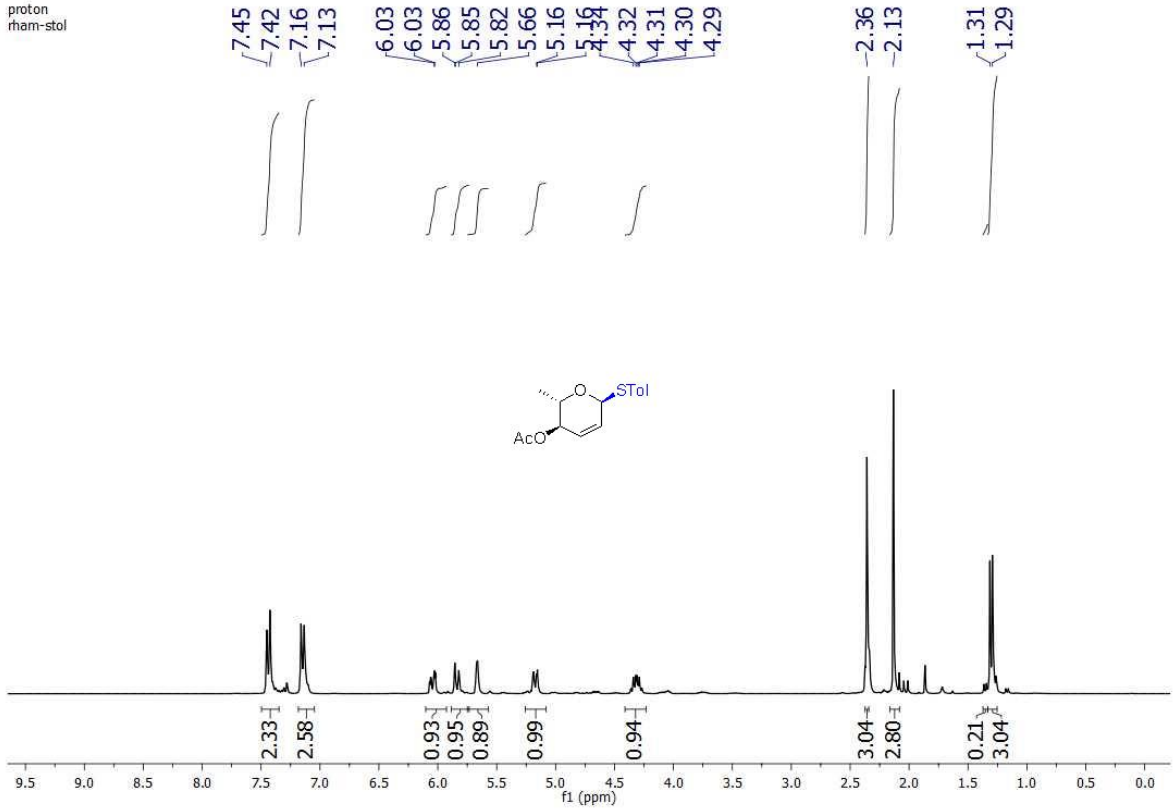
^{1}H NMR of compound 7g



^{13}C NMR of compound 7g



^{1}H NMR of compound 7h



^{13}C NMR of compound 7h