

SUPPORTING INFORMATION

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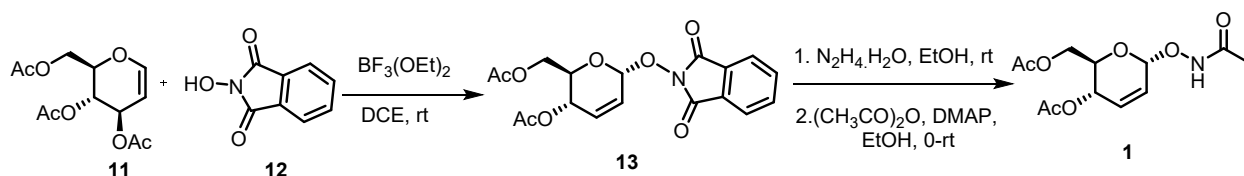
1. General Information

All compounds were characterized by spectroscopic data. The ^1H and ^{13}C NMR spectra were obtained using 400 and 500 MHz spectrometers with TMS as internal standard. Chemical shift (δ) is expressed in ppm, J values are given in Hz and deuterated CDCl_3 was used as solvent. All the reactions were monitored by thin layer chromatography (TLC). Column chromatography was performed on silica gel (60-120 mesh). All the chemicals used in experiments were purchased from commercial source mostly from sigma Aldrich and were used without further purification.

2. Experimental

2.1. General procedures

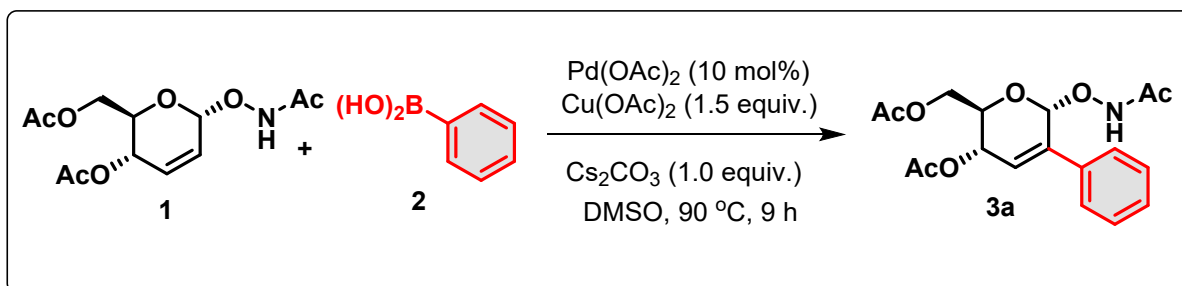
2.1.1 General procedure for the synthesis of N-(glycosyloxy)acetamide **1**.



Tri-*O*-acetyl-D-glucal **11** (6.0 g, 22 mmol, 1.0 equiv.) and N-hydroxyphthalimide **12** (3.60 g, 22 mmol, 1.0 equiv.) dissolved in DCE (40 ml) in a round bottom flask at rt. Afterwards $\text{BF}_3(\text{OEt})_2$ (1.56 g, 11 mmol, 0.5 equiv.) was added slowly with syringe to the mixture and stirred for 20-30 minutes at room temperature. After the completion of reaction monitored by TLC, the reaction mixture was quenched by the addition of a saturated solution of NaHCO_3 . The solution was transferred into a separatory funnel and washed with ethyl acetate. The organic phase was dried over anhydrous MgSO_4 and the filtrate was concentrated under reduced pressure. The residue left was purified by column chromatography over silica gel (60-120 mesh) using petroleum ether and hexane to acquire the pure product **13** as a white crystal solid (75% yield, 6.2 gm).¹ Then in a round bottom flask compound **13** (5 g, 13.33 mmol, 1.0 equiv.), was dissolved in 40 ml of ethanol and $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ (800.1mg, 16.0 mmol, 1.2 equiv.), was added slowly at room temperature via syringe. The resulting mixture was allowed to stir for 0.5 h until the reaction was completed (monitored by TLC). After the complete conversion of starting material, the reaction mixture was cooled at 0 °C in an icebath and DMAP (325.7 mg, 2.7 mmol, 0.2 equiv.), acetic anhydride (5.4 g, 53.3 mmol, 4.0 equiv.), were added slowly into the mixture. The reaction mixture was removed

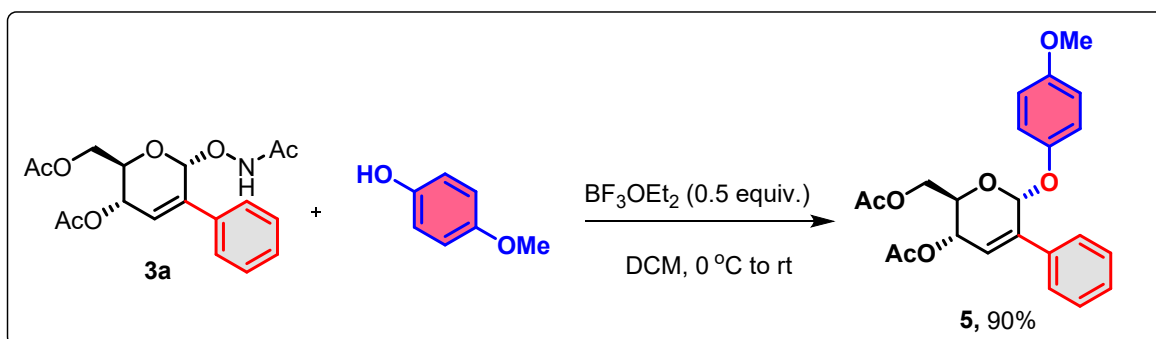
from icebath after 10 minutes and was stirred at room temperature until the reaction completes (monitored by TLC). After the complete conversion, the reaction mixture was diluted with ethyl acetate and washed with brine. The organic layer was separated, dried over MgSO_4 and evaporated. The residue left was purified by column chromatography over silica gel (60-120 mesh) using petroleum ether and hexane to get the pure product **1** as a colorless viscous (94% yield, 3.6 g).²

2.1.2 General procedure for the synthesis of compound 3a-3p.



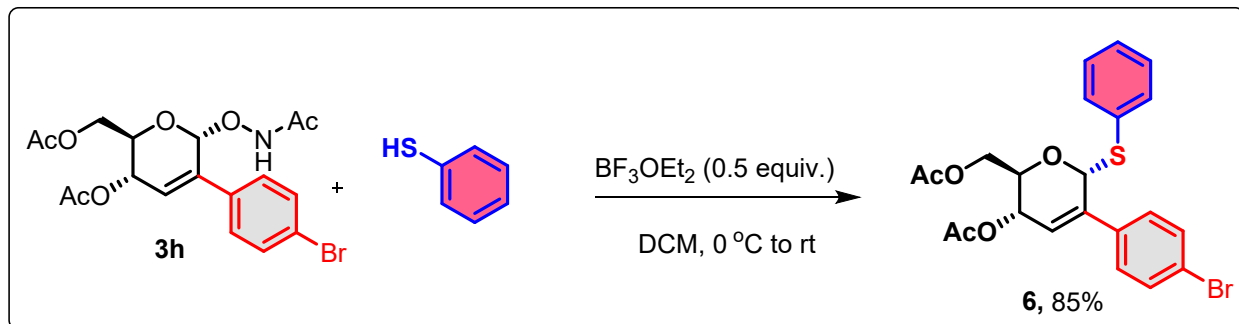
A mixture of **1** (30 mg, 0.10 mmol, 1.0 equiv.), Phenyl boronic acid (25.5 mg, 0.21 mmol, 2.0 equiv.), $\text{Pd}(\text{OAc})_2$ (2.3 mg, 0.01 mmol, 10 mol %), $\text{Cu}(\text{OAc})_2$ (28.5 mg, 0.16 mmol, 1.5 equiv.) and Cs_2CO_3 (34.0 mg, 0.10 mmol, 1.0 equiv.) were loaded into a Schlenk tube with magnetic bead. Then DMSO (3ml) was added to the mixture and the resulting mixture was stirred for 9 h at 90 °C in an oil bath. After completion of reaction, the reaction mixture was cooled and filtered through a small bed of celite-545. The filtrate was washed with ethyl acetate and water. The organic layer was dried over MgSO_4 , filtered and evaporated in vacuo. The residue left was purified by column chromatography over silica gel (60-120 mesh) using petroleum ether and hexane to acquire a pure product **3a** as a colorless viscous (82% yield, 31 mg).

2.1.3 General procedure for the synthesis of compound 5.



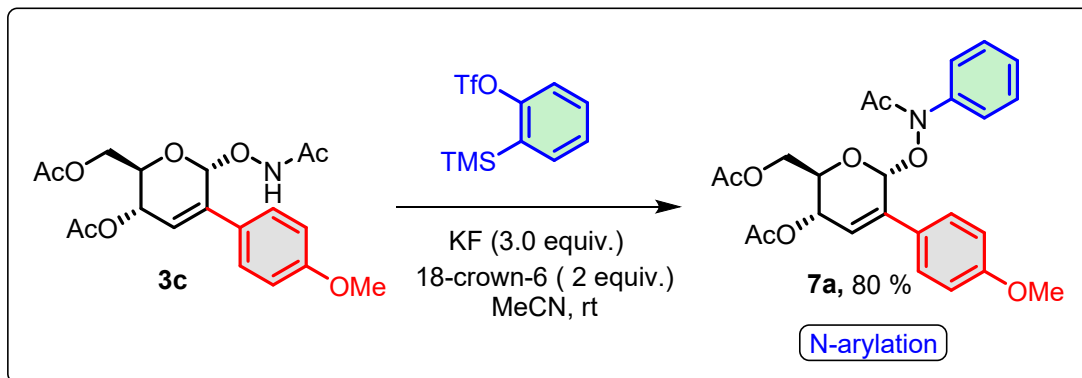
In a round bottom flask compound **3a** (30 mg, 0.083 mmol, 1.0 equiv.) and para methoxy phenol (10.25 mg, 0.083 mmol, 1.0 equiv.) was dissolved in 4 ml of DCM. The reaction mixture was cooled at 0 °C in an ice bath and BF_3OEt_2 (5.86 mg, 0.041 mmol, 0.5 equiv.) were added slowly into the mixture via syringe. The reaction mixture was removed from the ice bath after 5 minutes and was stirred at room temperature until the reaction completes (monitored by TLC). After the complete conversion, the reaction mixture was diluted with DCM and washed with brine. The organic layer was separated, dried over MgSO_4 , and evaporated. The residue left was purified by column chromatography over silica gel (60-120 mesh) using petroleum ether and hexane to acquire a pure product **5** as colourless viscous (90% yield, 30.5 mg).

2.1.4 General procedure for the synthesis of compound 6.



In a round bottom flask compound **3h** (30 mg, 0.083 mmol, 1.0 equiv.) and thiophenol (9.1 mg, 0.083 mmol, 1.0 equiv.) was dissolved in 4 ml of DCM. The reaction mixture was cooled at 0 °C in an ice bath and BF_3OEt_2 (5.86 mg, 0.041 mmol, 0.5 equiv.) were added slowly into the mixture via syringe. The reaction mixture was removed from the ice bath after 5 minutes and was stirred at room temperature until the reaction completes (monitored by TLC). After the complete conversion, the reaction mixture was diluted with DCM and washed with brine. The organic layer was separated, dried over MgSO_4 , and evaporated. The residue left was purified by column chromatography over silica gel (60-120 mesh) using petroleum ether and hexane to acquire a pure product **6** as colourless viscous (85% yield, 28.0 mg).

2.1.5 General procedure for the synthesis of compound 7a-b.

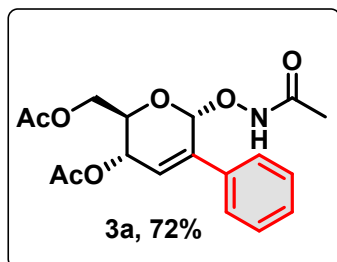


In an oven dried single neck round bottom flask charged with magnetic bead and flashed three times with N₂, a solution of compound **3c** (30 mg, 0.076 mmol, 1 equiv.) in 2 mL of MeCN was added. In the same solution aryne precursor (34.5 mg, 0.1 mmol, 1.5 equiv), KF (13.3 mg, 0.23 mmol, 3.0 equiv.) and 18-crown-6 (2.0 equiv) were also added and the reaction mixture was stirred at rt under N₂ atmosphere until complete consumption of starting material was observed by TLC analysis. Then the reaction mixture was diluted with ethyl acetate and washed with brine. The organic layer was dried over sodium sulphate and evaporated in vacuo. The residue left was purified by column chromatography over silica gel (60-120 mesh) using pet ether/ ethyl acetate as eluent to acquire a pure product **7a** as colourless viscous (80% yield, 28.5 mg).

3. Characterization Data

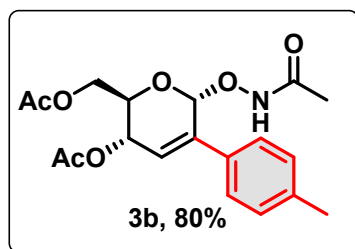
3.1. Characterization Data

6-(acetamidooxy)-2-(acetoxymethyl)-5-phenyl-3,6-dihydro-2H-pyran-3-yl acetate (3a)



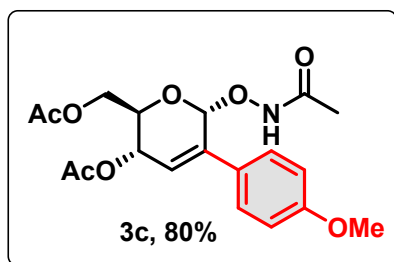
The compound **3a** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (72% yield, 27.5 mg). **¹H NMR (400 MHz, CDCl₃)** δ 8.86 (s, 1H), 7.65 (s, 2H), 7.43 – 7.30 (m, 3H), 6.28 (s, 1H), 5.70 (s, 1H), 5.57 (d, *J* = 8.9 Hz, 1H), 4.52 (s, 1H), 4.30 (t, *J* = 9.3 Hz, 2H), 2.13 (s, 6H), 1.94 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 171.0, 170.5, 168.3, 136.1, 128.8, 128.7, 126.3, 99.7, 67.6, 65.6, 62.8, 21.0, 20.9, 19.9. **HRMS (ESI)**, *m/z* calcd. for C₁₈H₂₁NO₇Na [M+Na]⁺ 386.1216, found 386.1210.

6-(acetamidooxy)-2-(acetoxymethyl)-5-(p-tolyl)-3,6-dihydro-2H-pyran-3-yl acetate (3b)



The compound **3b** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (80% yield, 31.5 mg). **¹H NMR (400 MHz, CDCl₃)** δ 8.88 (s, 1H), 7.53 (s, 2H), 7.17 (d, *J* = 8.1 Hz, 2H), 6.23 (s, 1H), 5.69 (s, 1H), 5.55 (d, *J* = 9.3 Hz, 1H), 4.49 (s, 1H), 4.31 (d, *J* = 3.2 Hz, 2H), 2.34 (s, 3H), 2.12 (s, 6H), 1.94 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 171.0, 170.5, 168.3, 138.7, 135.9, 132.3, 129.4, 126.2, 99.7, 67.6, 65.7, 62.8, 21.2, 21.0, 20.9, 19.9. **HRMS (ESI)**, *m/z* calcd. for C₁₉H₂₃NO₇Na [M+Na]⁺ 400.1372, found 400.1370.

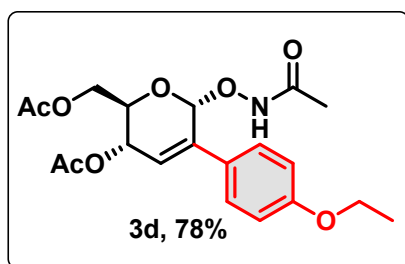
6-(acetamidooxy)-2-(acetoxymethyl)-5-(4-methoxyphenyl)-3,6-dihydro-2H-pyran-3-yl acetate (3c)



The compound **3c** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (80% yield, 33.0 mg). **¹H NMR (400 MHz, CDCl₃)** δ 8.72 (s, 1H), 7.64 (s, 2H),

6.94 – 6.84 (m, 2H), 6.19 (s, 1H), 5.68 (s, 1H), 5.55 (d, $J = 9.0$ Hz, 1H), 4.45 (s, 1H), 4.31 (d, $J = 3.2$ Hz, 2H), 3.80 (s, 3H), 2.13 (d, $J = 2.4$ Hz, 6H), 1.96 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 171.0, 170.5, 168.2, 160.0, 135.4, 127.6, 124.6, 114.0, 99.8, 67.7, 65.8, 62.9, 55.3, 21.0, 20.9, 20.0. HRMS (ESI), m/z calcd. for $\text{C}_{19}\text{H}_{23}\text{NO}_8\text{Na}$ $[\text{M}+\text{Na}]^+$ 416.1321, found 416.1321.

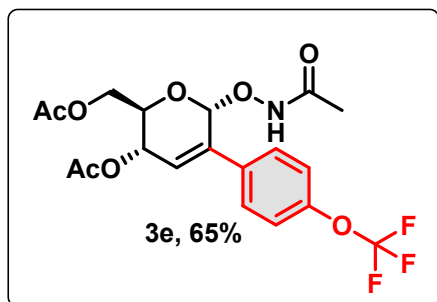
6-(acetamidooxy)-2-(acetoxymethyl)-5-(4-ethoxyphenyl)-3,6-dihydro-2H-pyran-3-yl acetate (3d)



The compound **3d** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (78% yield, 33.0 mg). ^1H NMR (400 MHz, CDCl_3) δ 8.82 (s, 1H), 7.63 (s, 2H),

6.88 (d, $J = 8.8$ Hz, 2H), 6.19 (s, 1H), 5.68 (s, 1H), 5.55 (d, $J = 9.0$ Hz, 1H), 4.46 (s, 1H), 4.31 (d, $J = 3.3$ Hz, 2H), 4.03 (q, $J = 7.0$ Hz, 2H), 2.12 (d, $J = 2.8$ Hz, 6H), 1.96 (s, 3H), 1.40 (t, $J = 7.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.98, 170.46, 168.25, 159.40, 135.44, 128.51, 127.53, 126.20, 124.39, 114.54, 99.75, 67.65, 65.82, 63.47, 62.88, 21.03, 20.86, 19.96, 14.77. HRMS (ESI), m/z calcd. for $\text{C}_{20}\text{H}_{25}\text{NO}_8\text{Na}$ $[\text{M}+\text{Na}]^+$ 430.1478, found 430.1477.

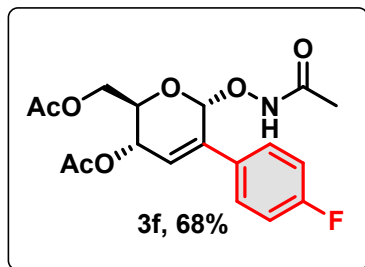
6-(acetamidooxy)-2-(acetoxymethyl)-5-(4-(trifluoromethoxy)phenyl)-3,6-dihydro-2H-pyran-3-yl acetate (3e)



The compound **3e** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (65% yield, 30.5 mg). ^1H NMR (400 MHz, CDCl_3) δ 8.43 (s, 1H), 7.78 (dd, $J = 46.0, 6.7$ Hz, 2H), 7.16 (d, $J = 8.4$ Hz, 2H), 6.22 (s,

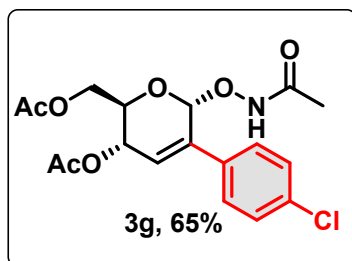
1H), 5.59 (s, 1H), 5.48 (d, $J = 8.7$ Hz, 1H), 4.36 (s, 1H), 4.25 (s, 2H), 2.07 (s, 6H), 1.91 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.9, 170.3, 168.6, 149.5, 135.0, 134.8, 128.0, 124.1, 121.7, 121.0, 99.5, 67.7, 65.6, 62.9, 21.0, 20.9, 20.0. HRMS (ESI), m/z calcd. for $\text{C}_{19}\text{H}_{20}\text{F}_3\text{NO}_8\text{Na}$ $[\text{M}+\text{Na}]^+$ 470.1039, found 470.1030.

6-(acetamidooxy)-2-(acetoxymethyl)-5-(4-fluorophenyl)-3,6-dihydro-2H-pyran-3-yl acetate (3f)



The compound **3f** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtain colorless viscous (68% yield, 27.0 mg). **¹H NMR (400 MHz, CDCl₃)** δ 7.71 (s, 2H), 7.08 (t, *J* = 8.5 Hz, 2H), 6.25 (s, 1H), 5.67 (s, 1H), 5.56 (d, *J* = 9.3 Hz, 1H), 4.46 (s, 1H), 4.33 (s, 2H), 2.15 (d, *J* = 0.9 Hz, 6H), 1.98 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 171.0, 170.4, 164.3, 161.8, 135.2, 131.1, 128.3, 128.2, 126.5, 115.7, 115.5, 99.6, 67.6, 65.7, 62.8, 21.0, 20.9, 19.9. **HRMS (ESI)**, *m/z* calcd. for C₁₈H₂₀NO₇FNa [M+Na]⁺ 404.1121, found 404.1121.

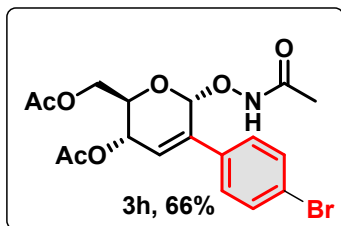
6-(acetamidooxy)-2-(acetoxymethyl)-5-(4-chlorophenyl)-3,6-dihydro-2H-pyran-3-yl acetate (3g)



The compound **3g** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtain colorless viscous (65% yield, 27 mg). **¹H NMR (400 MHz, CDCl₃)** δ 8.93 (s, 1H), 7.57 (s, 2H), 7.26 (d, *J* = 8.3 Hz, 2H), 6.21 (s, 1H), 5.58 (s, 1H), 5.48 (d, *J* = 9.5 Hz, 1H), 4.38 (s, 1H), 4.24 (d, *J* = 3.1 Hz, 2H), 2.06 (s, 6H), 1.89 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 171.0, 170.4, 168.3, 135.1, 134.7, 133.5, 128.82, 127.7, 127.0, 99.4, 67.5, 65.6, 62.7, 31.0, 21.0, 20.9, 20.0. **HRMS (ESI)**, *m/z* calcd. for C₁₈H₂₀NO₇ClNa [M+Na]⁺ 420.0826, found 420.0822.

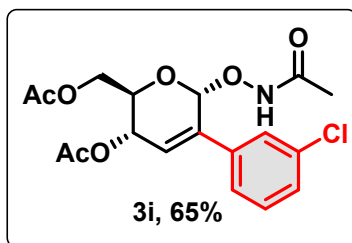
6-(acetamidooxy)-2-(acetoxymethyl)-5-(4-bromophenyl)-3,6-dihydro-2H-pyran-3-yl acetate (3h)

The compound **3h** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtain colorless viscous (66% yield, 30.5 mg). **¹H NMR (400 MHz, CDCl₃)** δ 8.93 (s, 1H), 7.61 – 7.44 (m, 4H), 6.28 (s, 1H), 5.65 (s, 1H), 5.54 (d, *J* = 9.5 Hz, 1H), 4.45 (s, 1H), 4.31 (d,



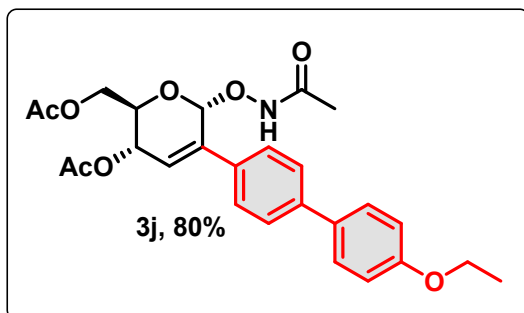
$J = 3.1$ Hz, 2H), 2.13 (s, 6H), 1.96 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 171.02, 170.41, 168.40, 135.16, 132.31, 131.80, 128.01, 123.03, 117.30, 99.41, 67.56, 65.58, 62.74, 20.99, 20.86, 19.94. HRMS (ESI), m/z calcd. for $\text{C}_{18}\text{H}_{20}\text{NO}_7\text{BrNa}$ $[\text{M}+\text{Na}]^+$ 464.0321, found 464.0317.

6-(acetamidooxy)-2-(acetoxymethyl)-5-(3-chlorophenyl)-3,6-dihydro-2H-pyran-3-yl acetate (3i)



The compound **3i** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained white colorless viscous (65% yield, 30.0 mg). ^1H NMR (400 MHz, CDCl_3) δ 8.71 (s, 1H), 7.66 (d, $J = 52.8$ Hz, 2H), 7.31 (d, $J = 3.8$ Hz, 2H), 6.31 (s, 1H), 5.64 (s, 1H), 5.56 (d, $J = 9.5$ Hz, 1H), 4.48 (s, 1H), 4.32 (s, 2H), 2.14 (s, 6H), 1.97 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 171.0, 170.4, 168.4, 136.9, 135.0, 134.7, 129.9, 128.8, 127.9, 126.6, 124.6, 99.5, 67.6, 65.5, 62.7, 21.0, 20.9, 20.0. HRMS (ESI), m/z calcd. for $\text{C}_{18}\text{H}_{20}\text{NO}_7\text{ClNa}$ $[\text{M}+\text{Na}]^+$ 420.0826, found 420.0826.

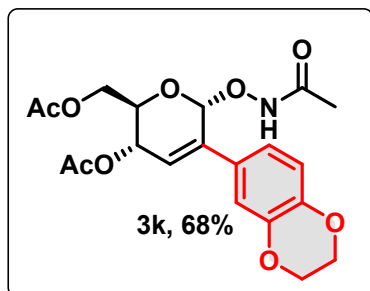
6-(acetamidooxy)-2-(acetoxymethyl)-5-(4'-ethoxy-[1,1'-biphenyl]-4-yl)-3,6-dihydro-2H-pyran-3-yl acetate



The compound **3j** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (80% yield, 40.0 mg). ^1H NMR (400 MHz, CDCl_3) δ 7.64 (s, 1H), 7.52 – 7.40 (m, 5H), 6.87 (d, $J = 8.5$ Hz, 2H), 6.24 (s, 1H), 5.67 (s, 1H), 5.50 (d, $J = 8.9$ Hz, 1H), 4.43 (s, 1H), 4.25 (s, 2H), 3.99 (q, $J = 6.9$ Hz, 2H), 2.06 (s, 6H), 1.90 (s, 3H), 1.36 (t, $J = 7.0$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 171.0, 170.4, 168.3, 158.8, 141.1, 135.7, 133.2, 132.7, 128.0, 126.8, 126.7, 126.2, 114.9,

99.7, 67.7, 65.7, 63.5, 62.9, 21.0, 20.9, 14.9. **HRMS (ESI)**, m/z calcd. for C₂₆H₂₉NO₈Na [M+Na]⁺ 506.1791, found 506.1782.

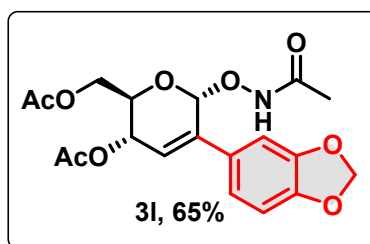
6-(acetamidooxy)-2-(acetoxymethyl)-5-(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)-3,6-dihydro-2H-pyran-3-yl acetate (3k)



The compound **3k** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (68% yield, 30.0 mg). ¹H NMR (400 MHz, CDCl₃) δ 9.08 (s, 1H), 7.31 – 7.15 (m, 1H), 7.12 – 7.01 (m, 1H), 6.75 (d, *J* = 8.4 Hz, 1H), 6.11 (s, 1H), 5.55 (s, 1H),

5.47 (d, *J* = 9.4 Hz, 1H), 4.41 (s, 1H), 4.22 (d, *J* = 3.1 Hz, 2H), 4.17 (s, 4H), 2.04 (s, 6H), 1.88 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 171.0, 170.5, 168.4, 144.1, 143.5, 135.2, 129.0, 128.5, 128.2, 125.3, 125.0, 119.3, 117.4, 115.5, 99.7, 67.6, 65.7, 64.5, 64.3, 62.8, 21.0, 20.8, 19.9. **HRMS (ESI)**, m/z calcd. for C₂₀H₂₃NO₉Na [M+Na]⁺ 444.1271, found 444.1265.

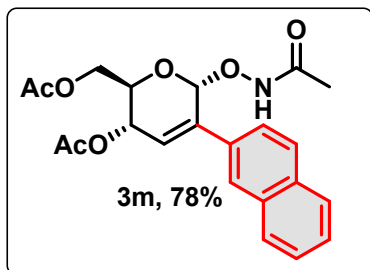
6-(acetamidooxy)-2-(acetoxymethyl)-5-(benzo[d][1,3]dioxol-5-yl)-3,6-dihydro-2H-pyran-3-yl acetate (3l)



The compound **3l** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (65% yield, 28.0 mg). ¹H NMR (400 MHz, CDCl₃) δ 7.17 (d, *J* = 22.7 Hz, 2H), 6.73 (d, *J*

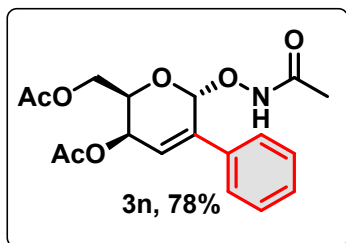
= 8.0 Hz, 1H), 6.09 (s, 1H), 5.89 (s, 2H), 5.55 (s, 1H), 5.47 (d, *J* = 9.3 Hz, 1H), 4.38 (s, 1H), 4.23 (d, *J* = 3.2 Hz, 2H), 2.06 (s, 6H), 1.89 (s, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 171.0, 170.5, 148.0, 135.6, 125.3, 120.4, 108.4, 106.8, 101.3, 100.0, 67.6, 65.7, 62.8, 29.7, 21.0, 20.9, 19.9. **HRMS (ESI)**, m/z calcd. for C₁₉H₂₁NO₉Na [M+Na]⁺ 430.1114, found 430.1110.

6-(acetamidooxy)-2-(acetoxymethyl)-5-(naphthalen-2-yl)-3,6-dihydro-2H-pyran-3-yl acetate (3m)



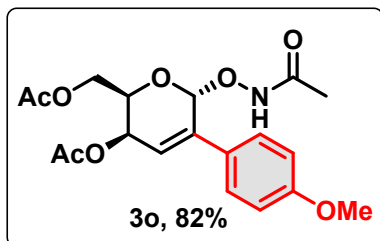
The compound **3m** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (78% yield, 33.5mg). **¹H NMR (400 MHz, CDCl₃)** δ 9.04 (s, 1H), 8.41 (s, 1H), 7.94 – 7.64 (m, 4H), 7.53 – 7.41 (m, 2H), 6.43 (s, 1H), 5.82 (s, 1H), 5.62 (d, *J* = 9.3 Hz, 1H), 4.52 (s, 1H), 4.34 (d, *J* = 2.7 Hz, 2H), 2.13 (s, 6H), 1.96 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 171.0, 171.0, 168.4, 135.9, 133.4, 133.2, 132.0, 128.7, 128.2, 127.5, 126.6, 126.4, 126.2, 123.5, 99.8, 67.7, 65.8, 62.8, 21.0, 20.9, 20.0. **HRMS (ESI)**, *m/z* calcd. for C₂₂H₂₃NO₇Na [M+Na]⁺ 436.1372, found 436.1373.

6-(acetamidooxy)-2-(acetoxymethyl)-5-phenyl-3,6-dihydro-2H-pyran-3-yl acetate (**3n**)



The compound **3n** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (78% yield, 28.5 mg). **¹H NMR (400 MHz, CDCl₃)** δ 8.38 (s, 1H), 7.67 (s, 1H), 7.43 – 7.32 (m, 4H), 6.46 (s, 1H), 5.82 (s, 1H), 5.29 (dd, *J* = 5.6, 2.6 Hz, 1H), 4.66 (s, 1H), 4.33 (d, *J* = 6.2 Hz, 2H), 2.11 (d, *J* = 6.4 Hz, 6H), 1.96 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 170.8, 170.4, 129.1, 128.8, 126.5, 67.4, 63.4, 62.7. **HRMS (ESI)**, *m/z* calcd. for C₁₈H₂₁NO₇Na [M+Na]⁺ 386.1216, found 386.1211.

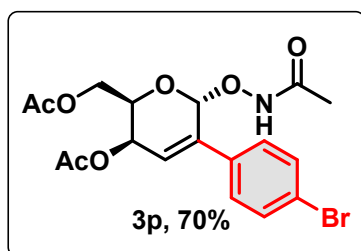
6-(acetamidooxy)-2-(acetoxymethyl)-5-(4-methoxyphenyl)-3,6-dihydro-2H-pyran-3-yl acetate (**3o**)



The compound **3o** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (82% yield, 34.0 mg). **¹H NMR (400 MHz, CDCl₃)** δ 8.45 (s, 1H), 7.67 (s, 2H), 6.95 – 6.89 (m, 2H), 6.39 (s, 1H), 5.81 (s, 1H), 5.27 (dd, *J* = 5.7, 2.7 Hz, 1H), 4.60 (s, 1H), 4.32 (d, *J* = 6.4 Hz, 2H), 3.82 (s, 3H), 2.12 (s, 3H), 2.10 (s, 3H), 1.97 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 170.8,

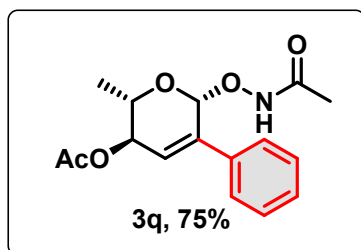
170.4, 160.3, 127.7, 120.5, 114.1, 99.0, 67.4, 63.5, 62.8, 55.3, 20.9, 20.0. **HRMS (ESI)**, m/z calcd. for $C_{19}H_{23}NO_8Na$ $[M+Na]^+$ 416.1321, found 416.1323.

6-(acetamidooxy)-2-(acetoxymethyl)-5-(4-bromophenyl)-3,6-dihydro-2H-pyran-3-yl acetate (3p)



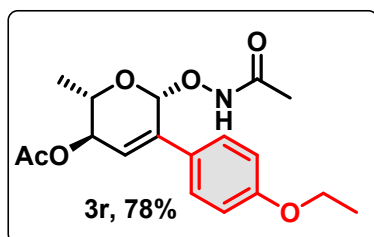
The compound **3p** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (70% yield, 32.0 mg). **1H NMR (400 MHz, $CDCl_3$)** δ 8.72 (s, 1H), 7.54 (t, $J = 18.1$ Hz, 4H), 6.47 (d, $J = 4.1$ Hz, 1H), 5.76 (s, 1H), 5.26 (dd, $J = 5.7, 2.7$ Hz, 1H), 4.60 (s, 1H), 4.32 (dd, $J = 6.2, 2.9$ Hz, 2H), 2.11 (d, $J = 1.9$ Hz, 6H), 1.96 (s, 3H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 170.9, 170.4, 168.3, 137.3, 131.9, 128.1, 123.4, 98.7, 67.2, 63.1, 62.5, 20.9, 20.8, 20.0. **HRMS (ESI)**, m/z calcd. for $C_{18}H_{20}NO_7BrNa$ $[M+Na]^+$ 464.0321, found 464.0320.

6-(acetamidooxy)-2-methyl-5-phenyl-3,6-dihydro-2H-pyran-3-yl acetate (3q)



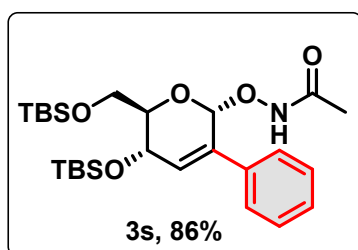
The compound **3q** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (75% yield, 36.0 mg). **1H NMR (400 MHz, $CDCl_3$)** δ 8.70 (s, 1H), 7.61 (s, 2H), 7.27 (dt, $J = 14.1, 7.0$ Hz, 4H), 6.18 (s, 1H), 5.57 (s, 1H), 5.20 (d, $J = 9.4$ Hz, 1H), 4.27 (s, 1H), 2.05 (s, 4H), 1.86 (s, 4H), 1.25 (d, $J = 6.2$ Hz, 4H). **^{13}C NMR (101 MHz, $CDCl_3$)** δ 170.67, 168.19, 136.00, 128.60, 126.37, 99.71, 71.22, 65.65, 36.65, 21.11, 17.78. **HRMS (ESI)**, m/z calcd. for $C_{16}H_{19}NO_5Na$ $[M+Na]^+$ 328.1161, found 328.1166.

6-(acetamidooxy)-5-(4-ethoxyphenyl)-2-methyl-3,6-dihydro-2H-pyran-3-yl acetate (3r)



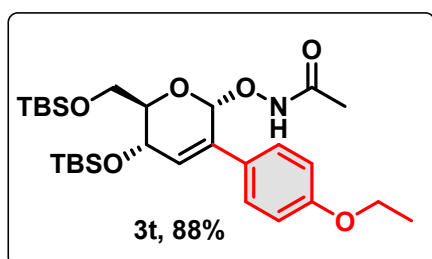
The compound **3r** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (78% yield, 37.0 mg). ^1H NMR (400 MHz, CDCl_3) δ 8.64 (d, $J = 27.8$ Hz, 1H), 7.58 (s, 2H), 6.80 (d, $J = 8.7$ Hz, 2H), 6.09 (s, 1H), 5.56 (s, 1H), 5.19 (d, $J = 9.4$ Hz, 1H), 4.23 (s, 1H), 3.95 (q, $J = 6.9$ Hz, 2H), 2.05 (s, 3H), 1.88 (s, 3H), 1.32 (dd, $J = 8.2, 5.3$ Hz, 3H), 1.24 (d, $J = 6.2$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.7, 168.1, 159.3, 135.3, 127.6, 125.0, 114.5, 99.8, 71.4, 65.7, 63.5, 36.6, 21.1, 17.8, 14.8. HRMS (ESI), m/z calcd. for $\text{C}_{18}\text{H}_{23}\text{NO}_6\text{Na}$ $[\text{M}+\text{Na}]^+$ 349.1525, found 349.1531.

N-((5-((tert-butyldimethylsilyl)oxy)-6-(((tert-butyldimethylsilyl)oxy)methyl)-3-phenyl-5,6-dihydro-2H-pyran-2-yl)oxy)acetamide (3s)



The compound **3s** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (86% yield, 39.5 mg). ^1H NMR (400 MHz, CDCl_3) δ 8.36 (s, 1H), 7.64 (s, 2H), 7.22 (dt, $J = 14.3, 7.4$ Hz, 3H), 6.13 (s, 1H), 5.56 (s, 1H), 4.18 (d, $J = 8.0$ Hz, 1H), 3.85 (d, $J = 10.9$ Hz, 2H), 3.64 (d, $J = 7.1$ Hz, 1H), 1.81 (s, 3H), 0.81 (d, $J = 8.8$ Hz, 18H), 0.01 (d, $J = 12.2$ Hz, 12H). ^{13}C NMR (101 MHz, CDCl_3) δ 167.4, 133.9, 131.6, 128.6, 128.3, 126.2, 99.0, 73.6, 65.1, 62.8, 26.0, 25.7, 20.0, 18.5, 18.0, -4.0, -4.7, -5.2, -5.3. HRMS (ESI), m/z calcd. for $\text{C}_{26}\text{H}_{45}\text{NO}_5\text{Si}_2\text{Na}$ $[\text{M}+\text{Na}]^+$ 530.2734, found 530.2736.

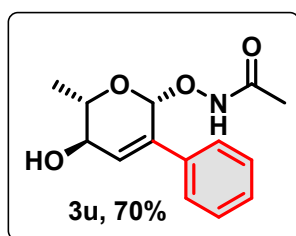
N-((5-((tert-butyldimethylsilyl)oxy)-6-(((tert-butyldimethylsilyl)oxy)methyl)-3-(4-ethoxyphenyl)-5,6-dihydro-2H-pyran-2-yl)oxy)acetamide (3t)



The compound **3t** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate

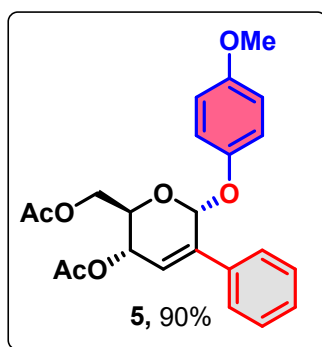
(80:20) as eluent to obtained colorless viscous (88% yield, 40.0 mg). **¹H NMR (400 MHz, CDCl₃)** δ 8.52 (s, 1H), 7.61 (d, *J* = 7.2 Hz, 2H), 6.78 (d, *J* = 8.6 Hz, 2H), 6.03 (s, 1H), 5.54 (s, 1H), 4.16 (d, *J* = 8.3 Hz, 1H), 3.89 (dt, *J* = 17.2, 8.3 Hz, 4H), 3.68 – 3.57 (m, 1H), 1.82 (s, 3H), 1.29 (t, *J* = 6.9 Hz, 3H), 0.81 (d, *J* = 10.0 Hz, 18H), 0.07 – -0.03 (m, 12H). **¹³C NMR (101 MHz, CDCl₃)** δ 167.4, 159.1, 133.2, 129.3, 127.6, 127.4, 114.5, 99.0, 73.7, 65.2, 63.4, 62.9, 26.0, 25.7, 20.0, 18.5, 18.0, 14.8, -4.0, -4.7, -5.2, -5.3. **HRMS (ESI)**, *m/z* calcd. for C₂₈H₄₉NO₆Si₂Na [M+Na]⁺ 574.2996, found 574.2998.

N-((5-hydroxy-6-methyl-3-phenyl-5,6-dihydro-2H-pyran-2-yl)oxy)acetamide (**3u**)



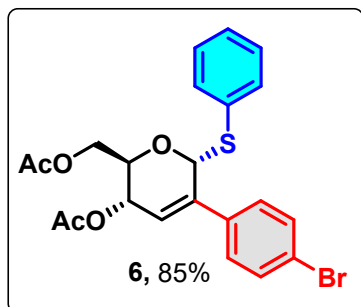
The compound **3u** was synthesized according to the general procedure (2.1.2) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (70% yield, 31.0 mg). **¹H NMR (400 MHz, CDCl₃)** δ 8.96 (s, 1H), 7.62 (s, 2H), 7.37 – 7.26 (m, 3H), 6.31 (s, 1H), 5.62 (s, 1H), 4.04 (t, *J* = 18.2 Hz, 2H), 1.91 (s, 3H), 1.36 (d, *J* = 4.9 Hz, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 168.54, 134.32, 131.35, 128.60, 128.35, 126.18, 99.37, 69.84, 68.85, 19.97, 17.81. **HRMS (ESI)**, *m/z* calcd. for C₁₄H₁₇NO₄Na [M+Na]⁺ 286.1055, found 286.1059.

3-acetoxy-6-(4-methoxyphenoxy)-5-phenyl-3,6-dihydro-2H-pyran-2-yl)methyl acetate (**5**)



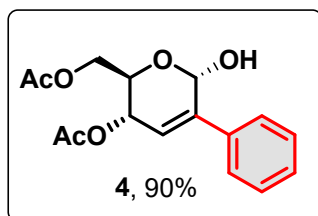
The compound **5** was synthesized according to the general procedure (2.1.3) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (90% yield, 30.5 mg). **¹H NMR (400 MHz, CDCl₃)** δ 7.38 (dd, *J* = 7.9, 1.7 Hz, 2H), 7.29 – 7.25 (m, 3H), 7.05 – 7.01 (m, 2H), 6.81 – 6.76 (m, 2H), 6.20 (d, *J* = 2.3 Hz, 1H), 5.83 (s, 1H), 5.49 (dd, *J* = 9.8, 1.5 Hz, 1H), 4.41 – 4.35 (m, 1H), 4.25 (dd, *J* = 12.1, 5.7 Hz, 1H), 4.17 (dd, *J* = 12.1, 2.5 Hz, 1H), 3.72 (s, 3H), 2.07 (s, 3H), 1.97 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 170.8, 170.5, 155.4, 151.1, 138.4, 135.8, 128.7, 128.6, 126.0, 124.7, 118.6, 114.6, 95.6, 67.3, 65.9, 62.9, 55.7, 21.1, 20.8. **HRMS (ESI)**, *m/z* calcd. for C₂₃H₂₄O₇Na [M+Na]⁺ 435.1420, found 435.1415.

3-acetoxy-5-(4-bromophenyl)-6-(phenylthio)-3,6-dihydro-2H-pyran-2-yl)methyl acetate (6)



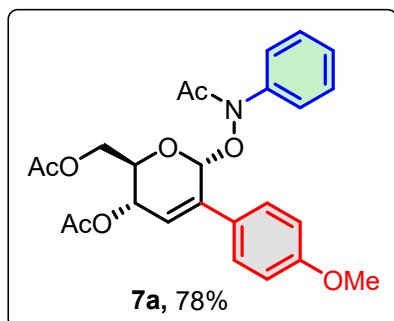
The compound **6** was synthesized according to the general procedure (2.1.4) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (85% yield, 28.0 mg). **¹H NMR (400 MHz, CDCl₃)** δ 7.48 – 7.41 (m, 4H), 7.28 – 7.21 (m, 5H), 6.03 (dd, *J* = 3.2, 1.9 Hz, 2H), 5.48 – 5.42 (m, 1H), 4.63 – 4.56 (m, 1H), 4.27 (dd, *J* = 12.2, 5.8 Hz, 1H), 4.21 (dd, *J* = 12.2, 2.6 Hz, 1H), 2.09 (s, 3H), 2.01 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 170.8, 170.5, 139.2, 134.7, 134.7, 131.9, 131.8, 129.1, 127.8, 127.8, 124.4, 122.9, 85.2, 67.1, 65.2, 63.1, 21.1, 20.8. **HRMS (ESI)**, *m/z* calcd. for C₂₂H₂₁BrO₅SNa [M+Na]⁺ 499.0191, found 491.0189.

3-acetoxy-6-hydroxy-5-phenyl-3,6-dihydro-2H-pyran-2-yl)methyl acetate (4)



The compound **4** was synthesized according to the general procedure (2.1.3) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (90% yield, 29.5 mg). **¹H NMR (400 MHz, CDCl₃)** δ 7.36 – 7.26 (m, 5H), 5.96 (d, *J* = 2.0 Hz, 1H), 5.92 (s, 1H), 5.40 (d, *J* = 9.8 Hz, 1H), 4.07 (dd, *J* = 12.3, 4.0 Hz, 1H), 3.92 (dd, *J* = 12.3, 2.2 Hz, 1H), 3.63 (d, *J* = 9.7 Hz, 1H), 2.01 (s, 3H), 1.99 (s, 3H). **¹³C NMR (101 MHz, CDCl₃)** δ 170.8, 170.3, 138.5, 136.2, 128.5, 126.4, 125.4, 92.5, 66.8, 65.1, 62.3, 21.0, 20.8. **HRMS (ESI)**, *m/z* calcd. for C₁₆H₁₈O₆Na [M+Na]⁺ 329.1001, found 329.0998.

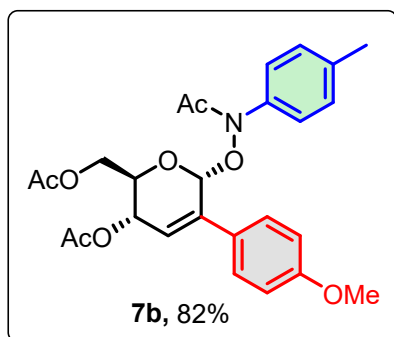
(3-acetoxy-5-(4-methoxyphenyl)-6-((N-phenylacetamido)oxy)-3,6-dihydro-2H-pyran-2-yl)methyl acetate (7a)



The compound **7a** was synthesized according to the general procedure (2.1.5) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (78% yield, 27.5 mg). **¹H NMR (400 MHz, CDCl₃)** δ 7.42 – 7.21 (m, 7H), 6.72 (d, *J* = 8.8 Hz, 2H), 6.06 (s, 1H), 5.82 (s, 1H), 5.46 (d, *J* = 9.6

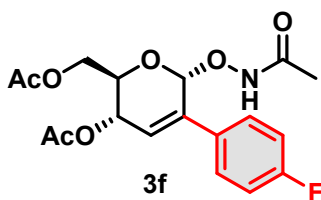
Hz, 1H), 4.04 (dd, $J = 18.6, 6.9$ Hz, 2H), 3.73 (d, $J = 7.3$ Hz, 3H), 3.62 (d, $J = 11.1$ Hz, 1H), 2.02 (d, $J = 4.4$ Hz, 6H), 1.95 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 170.4, 159.9, 140.6, 135.8, 129.3, 128.7, 127.7, 127.2, 113.8, 98.9, 67.8, 65.3, 61.7, 55.3, 22.1, 21.0, 20.8. HRMS (ESI), m/z calcd. for $\text{C}_{25}\text{H}_{27}\text{NO}_8\text{Na}$ $[\text{M}+\text{Na}]^+$ 492.1634, found 492.1630.

3-acetoxy-5-(4-methoxycyclohexa-1,3-dien-1-yl)-6-((N-(p-tolyl)acetamido)oxy)-3,6-dihydro-2H-pyran-2-yl)methyl acetate (7b)



The compound **7b** was synthesized according to the general procedure (2.1.5) and purified using column chromatography over silica gel (60-120 mesh) using pet ether/ethyl acetate (80:20) as eluent to obtained colorless viscous (82% yield, 28.5 mg). ^1H NMR (400 MHz, CDCl_3) δ 7.23 (ddd, $J = 32.1, 16.6, 8.6$ Hz, 7H), 6.81 – 6.74 (m, 2H), 6.14 (s, 1H), 5.87 (d, $J = 7.0$ Hz, 1H), 5.54 (d, $J = 9.9$ Hz, 1H), 4.18 (d, $J = 10.1$ Hz, 1H), 4.13 (dt, $J = 12.3, 3.4$ Hz, 1H), 3.82 – 3.77 (m, 4H), 2.38 (d, $J = 18.4$ Hz, 3H), 2.10 (d, $J = 2.9$ Hz, 6H), 2.01 (d, $J = 12.4$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 170.8, 170.4, 159.8, 139.4, 135.8, 129.9, 129.5, 129.0, 127.9, 127.7, 127.4, 124.4, 113.8, 98.6, 67.8, 65.3, 61.8, 55.3, 21.2, 21.0, 20.8, 14.1. HRMS (ESI), m/z calcd. for $\text{C}_{26}\text{H}_{31}\text{NO}_8\text{Na}$ $[\text{M}+\text{Na}]^+$ 508.1947, found 508.1940.

4. Single Crystal X-Ray Data and ORTEP Representation for compound 3f



Single crystals of (**3f**) were obtained in a dichloromethane: hexane (1:2) solution. A suitable crystal was selected and mounted on a Bruker D8 venture diffractometer. The crystal was kept at 270.0 K during data collection. The structure was solved and refined using Apex 4.

Compound	3f
Empirical formula	C _{2.4} H _{2.67} F _{0.13} N _{0.13} O _{0.93}
Formula weight	50.85
Temperature/K	100(2)
Crystal system	orthorhombic
Space group	P2 ₁ 2 ₁ 2 ₁
a/Å	5.1122(10)
b/Å	13.527(3)
c/Å	26.011(6)
α/°	90
β/°	90
γ/°	90
Volume/Å³	1798.7(7)
Z	30
ρ_{calc}/cm³	1.408
μ/mm⁻¹	0.115
F(000)	800.0
Crystal size/mm³	? × ? × ?
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	4.344 to 50.232
Index ranges	-6 ≤ h ≤ 6, -16 ≤ k ≤ 16, -31 ≤ l ≤ 31
Reflections collected	42680
Independent reflections	3191 [R _{int} = 0.0690, R _{sigma} = 0.0264]
Data/restraints/parameters	3191/0/250
Goodness-of-fit on F²	1.068
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0283, wR ₂ = 0.0731
Final R indexes [all data]	R ₁ = 0.0291, wR ₂ = 0.0735
Largest diff. peak/hole / e Å⁻³	0.18/-0.20
Flack parameter	0.1(3)

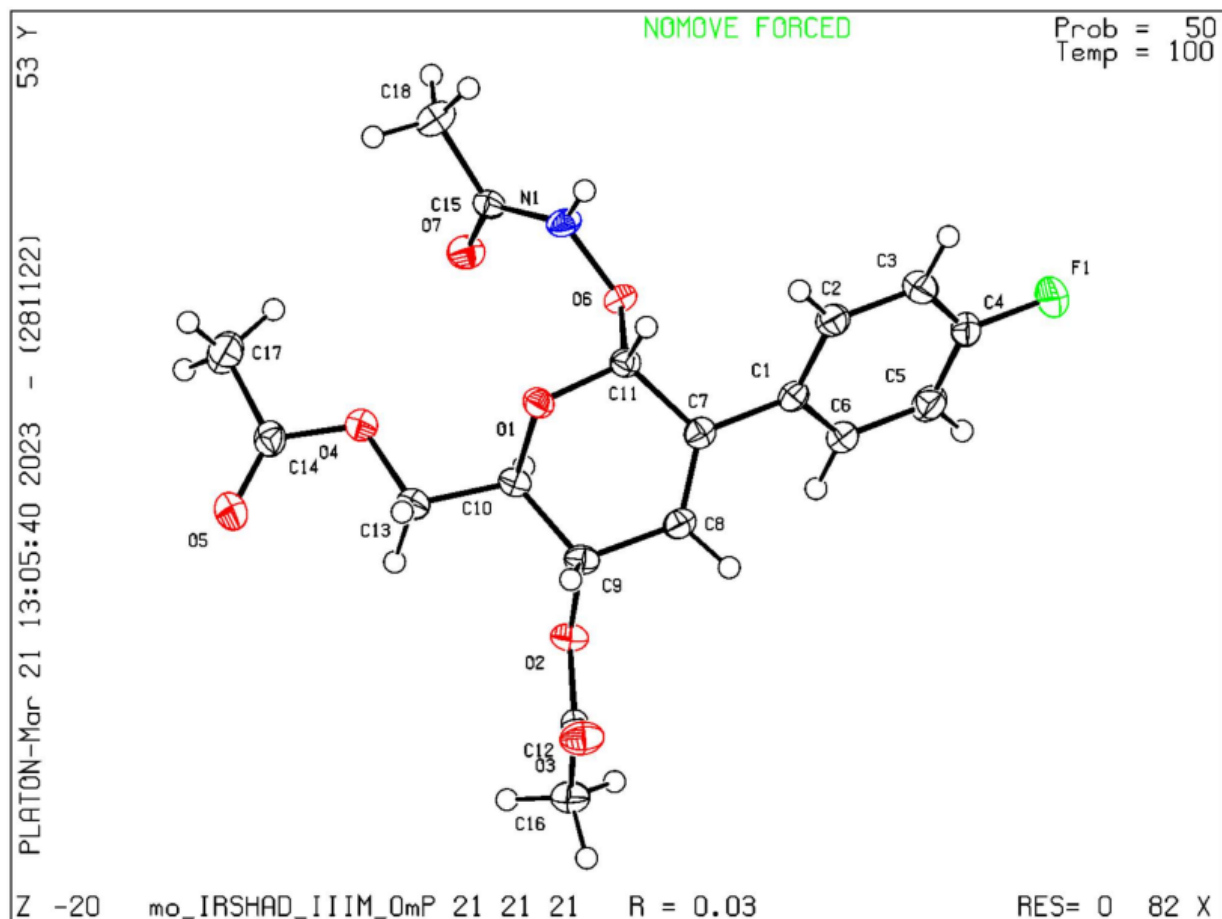


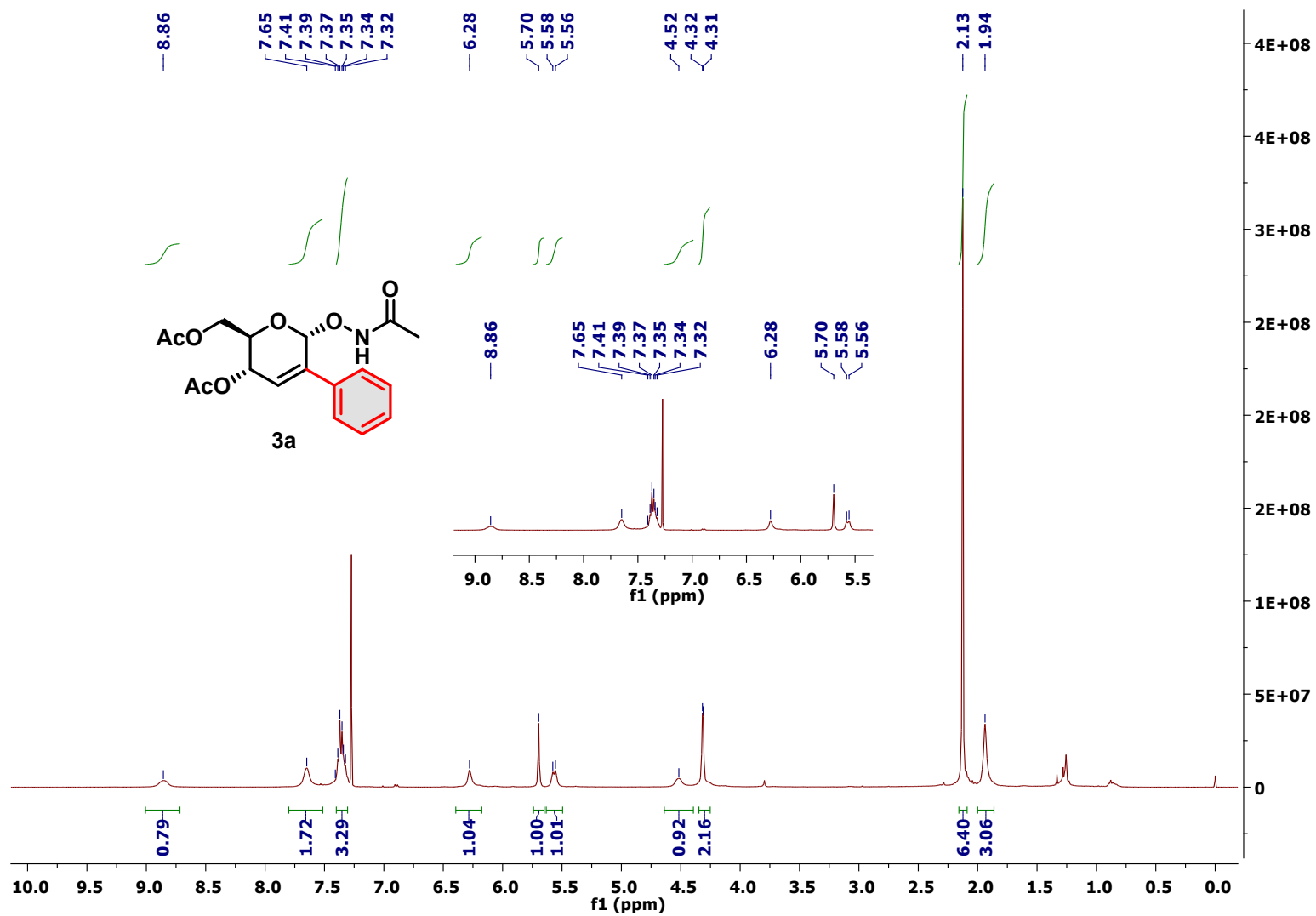
Figure 1: ORTEP representation of the crystal structure of compound **3f** (CCDC 2378048). Thermal ellipsoids are drawn at 50% probability level.

5. References

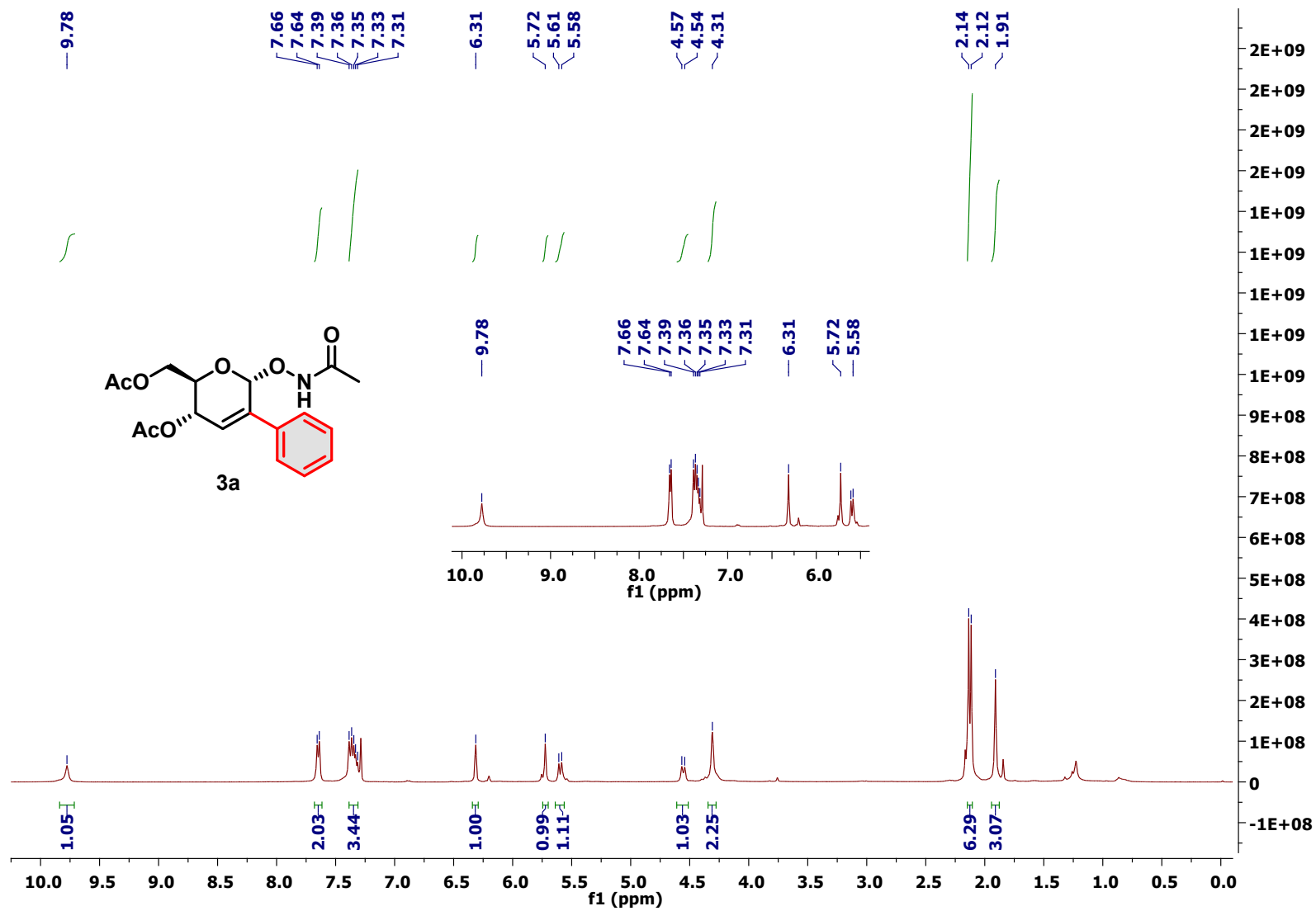
- 1) Reddy, C. R.; Rao, Y.S.; Kumar, T.P.; Reddy, K. V.; Chandrasekhar. *S. Synthesis* **2008**,1, 122–126.
- 2) Liu, M.; Li, B. -H.;Xiong, D. -C.;Ye, X. S. *J. Org. Chem.* **2018**, 83, 8292–8303.

6. NMR Spectra

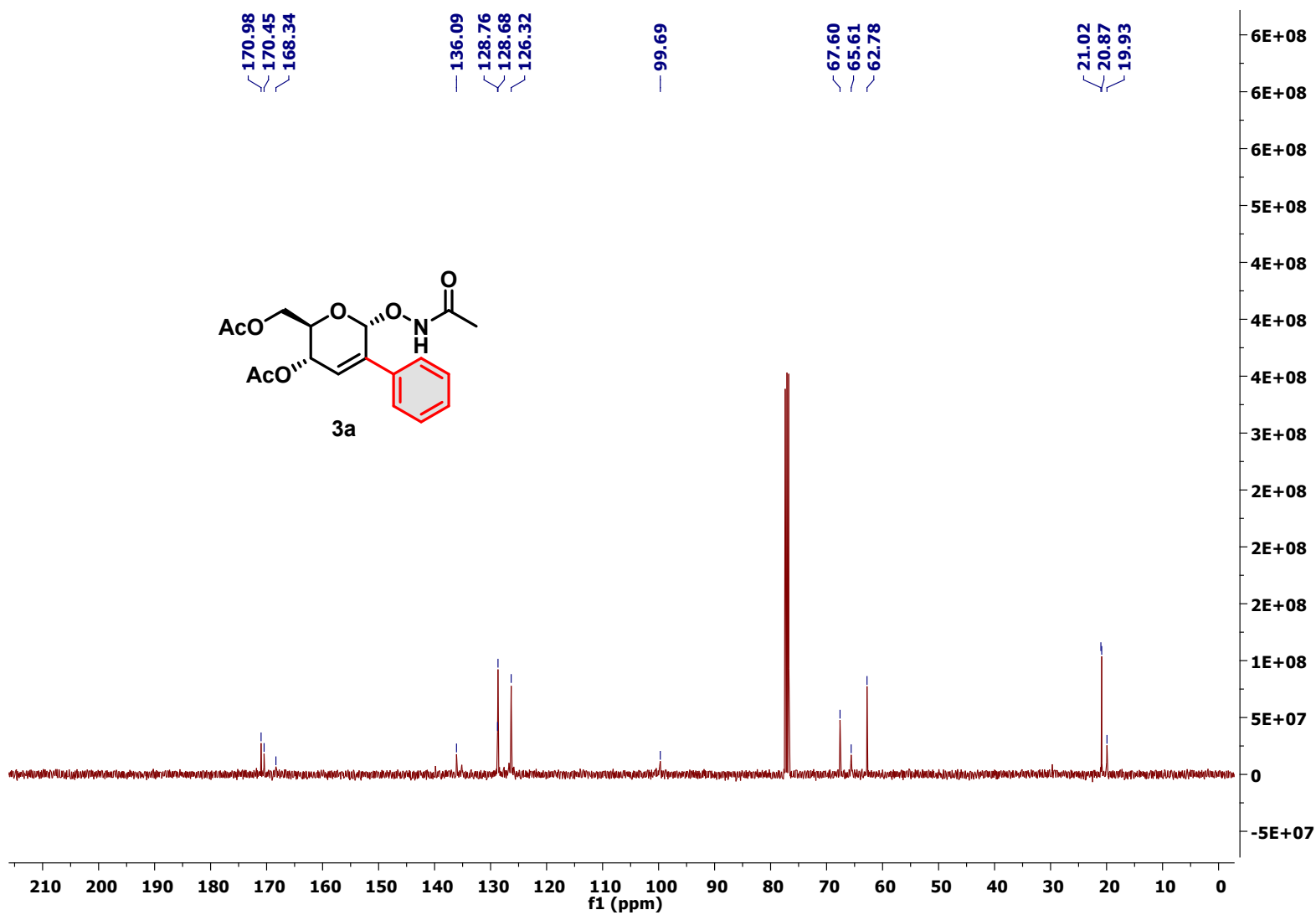
^1H NMR (400 MHz) of **3a** in CDCl_3 (room temperature)



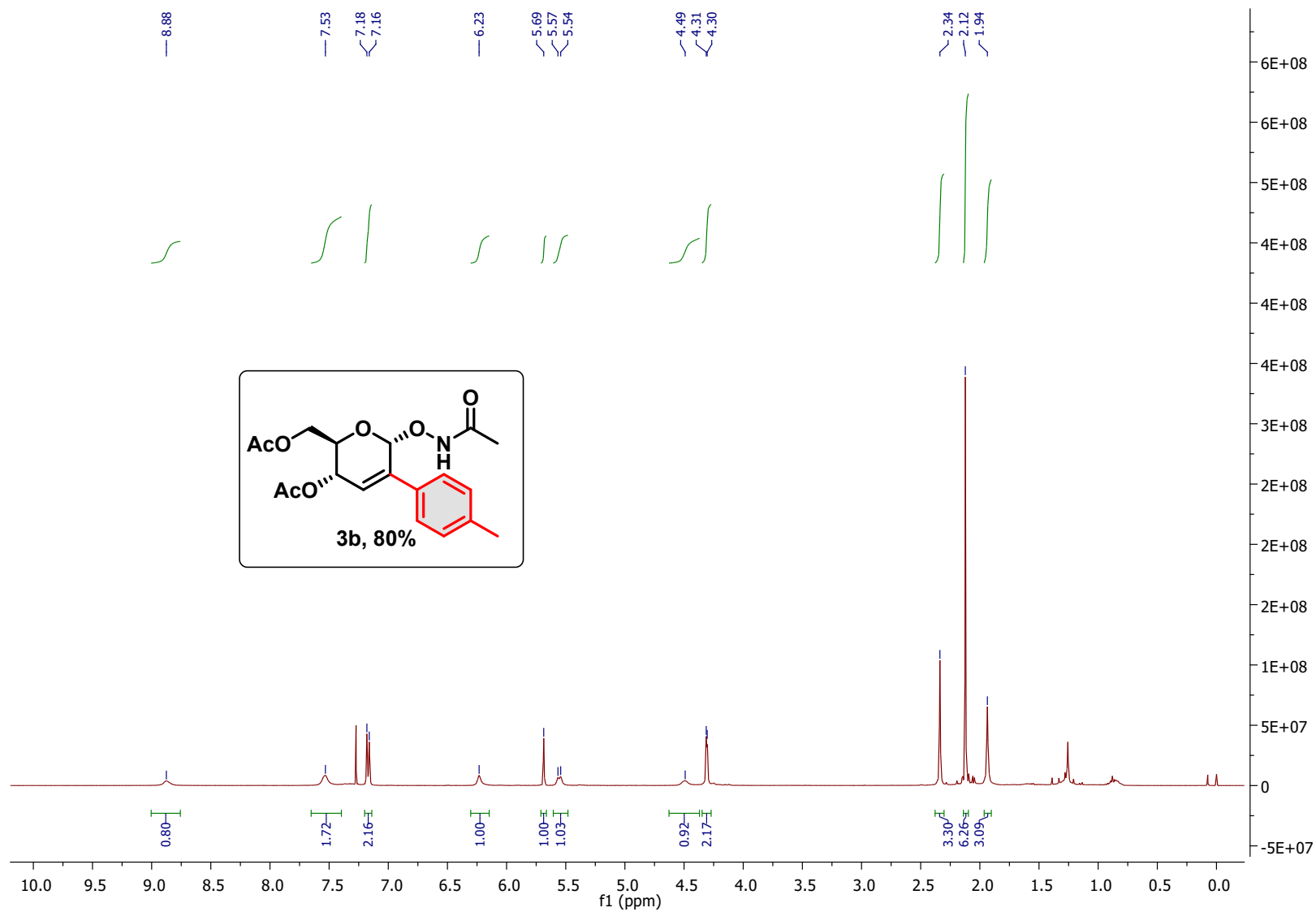
¹H NMR (400 MHz) of **3a** in CDCl₃ (-10 °C)



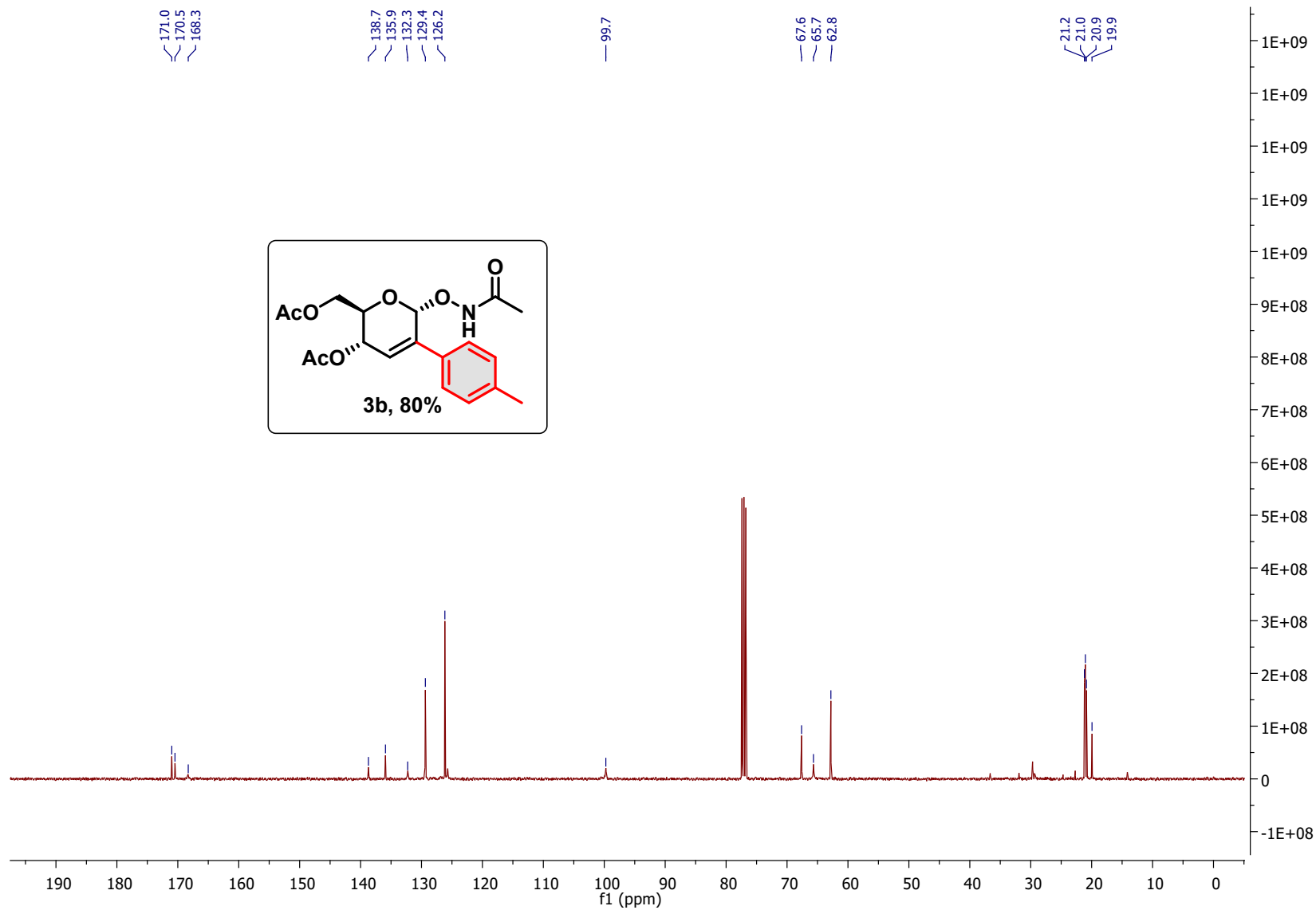
^{13}C NMR (101 MHz) of **3a** in CDCl_3



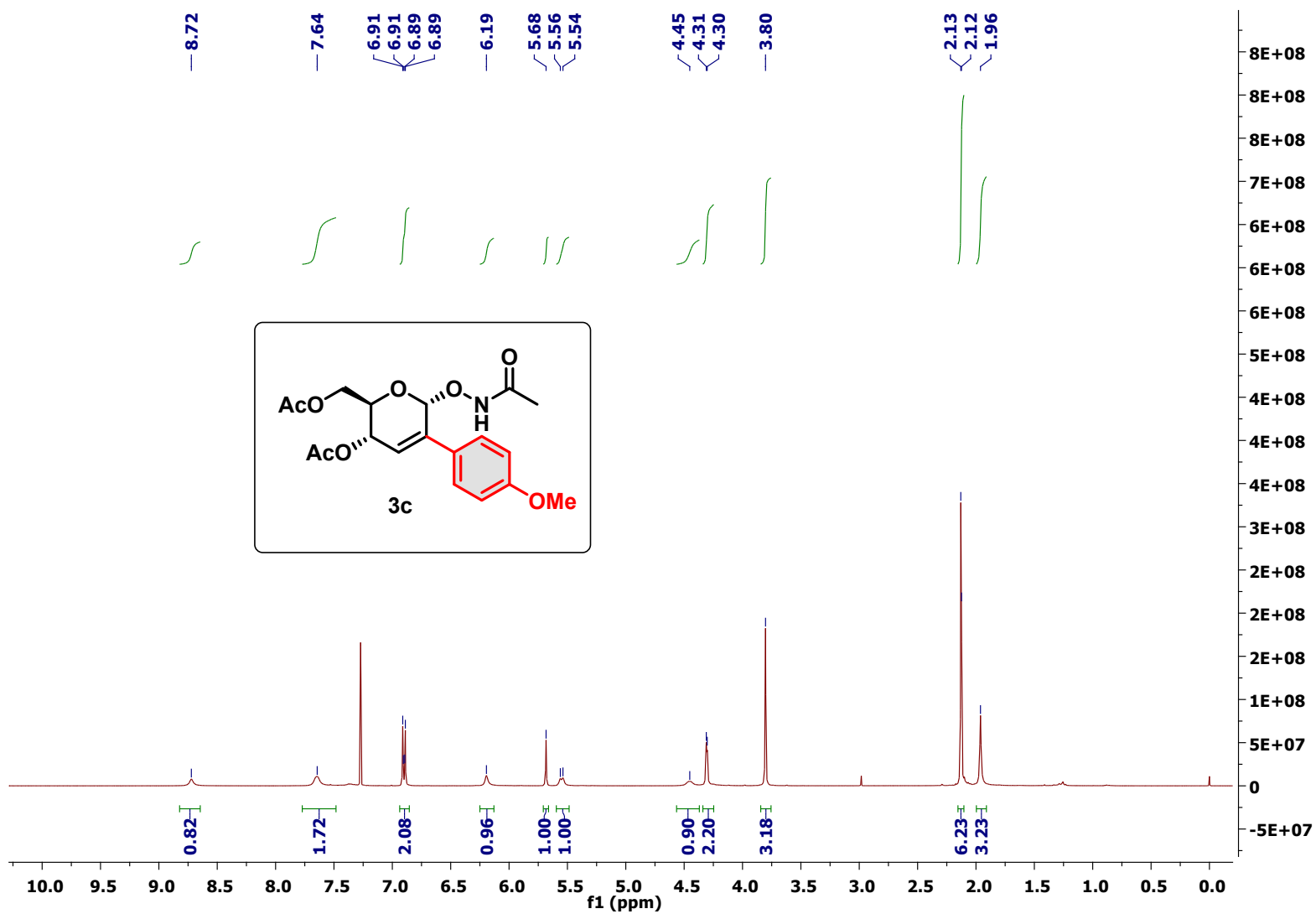
^1H NMR (400 MHz) of **3b** in CDCl_3



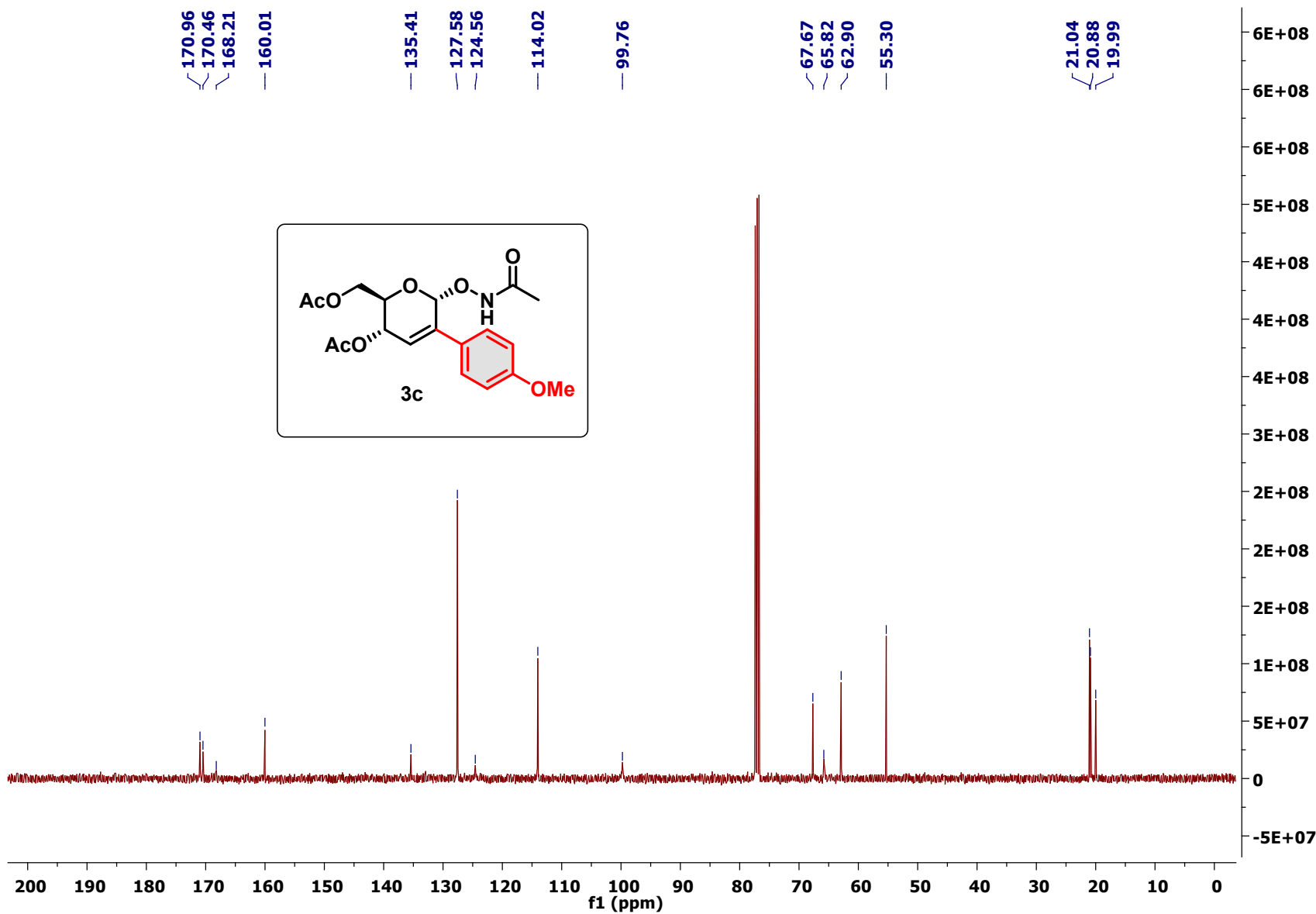
^{13}C NMR (101 MHz) of **3b** in CDCl_3



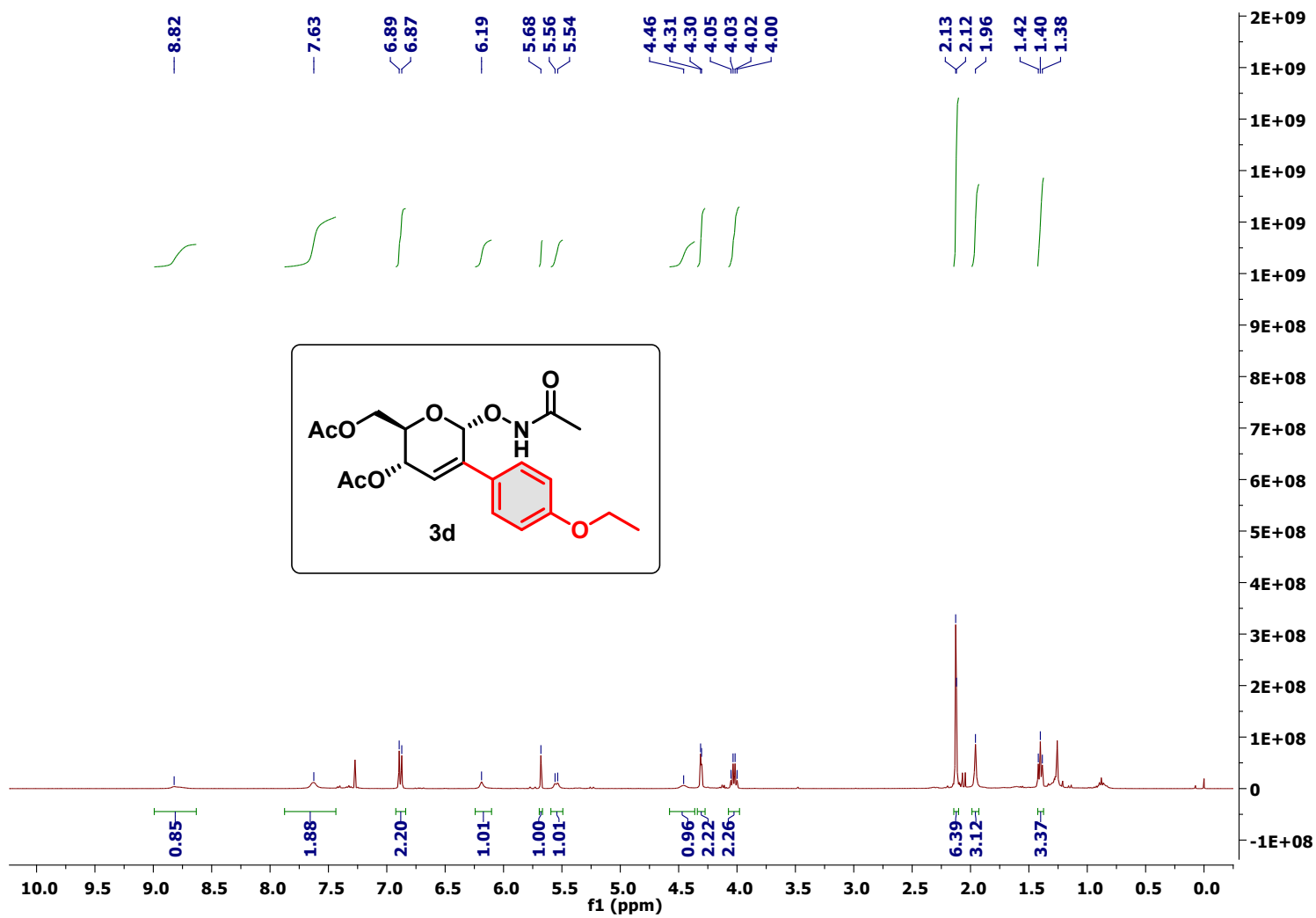
¹H NMR (400 MHz) of **3c** in CDCl₃



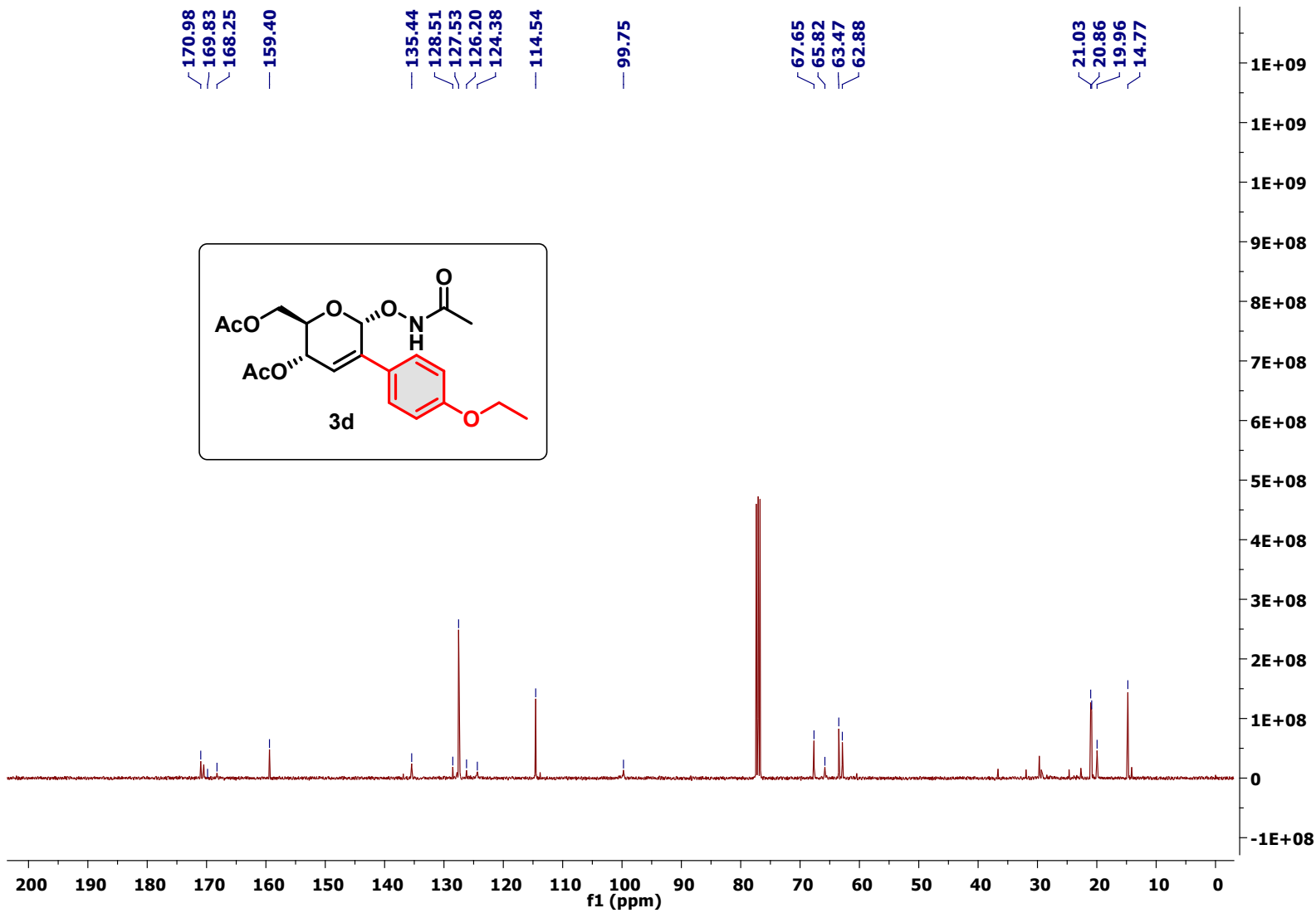
^{13}C NMR (101 MHz) of **3c** in CDCl_3



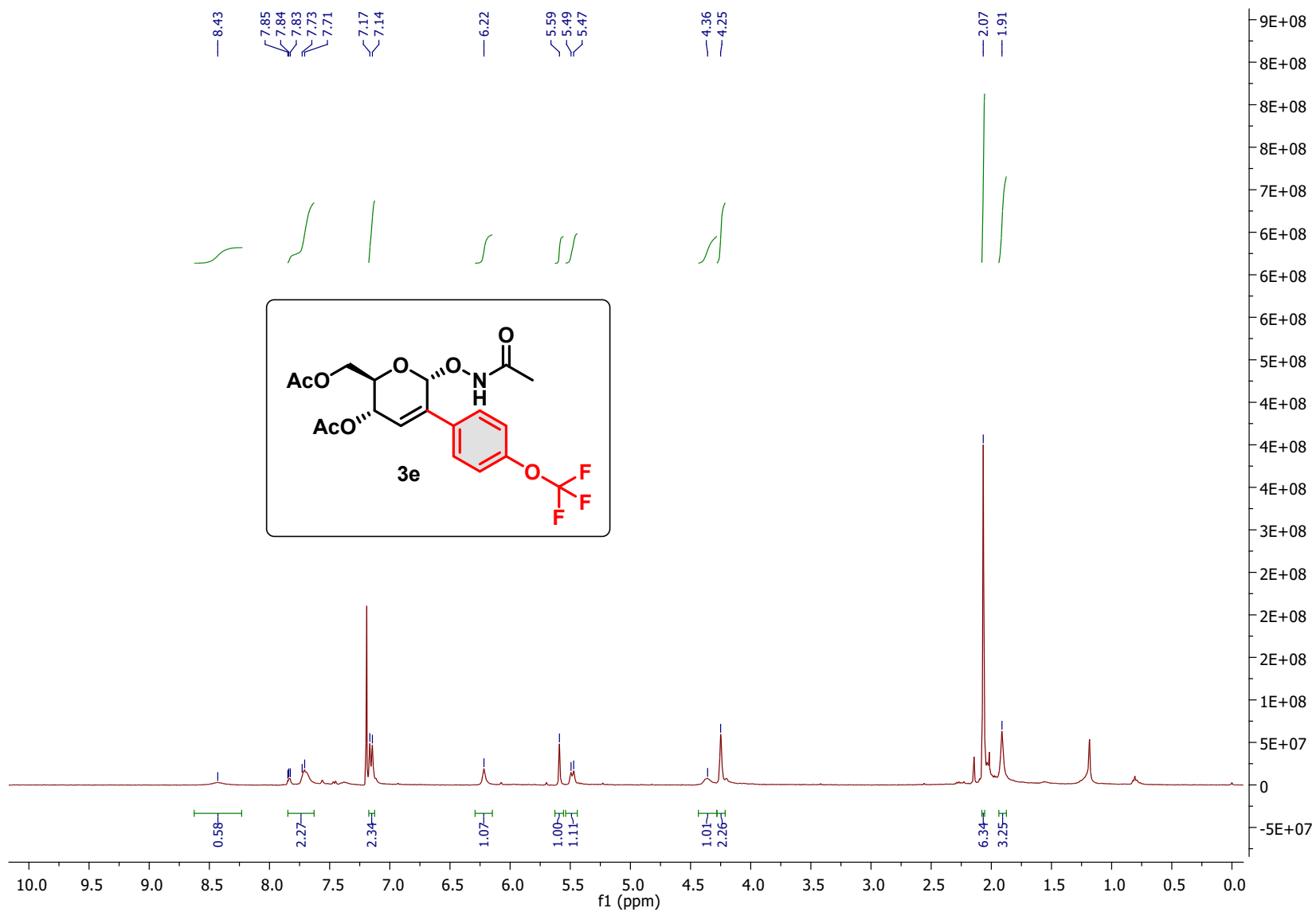
^1H NMR (400 MHz) of **3d** in CDCl_3



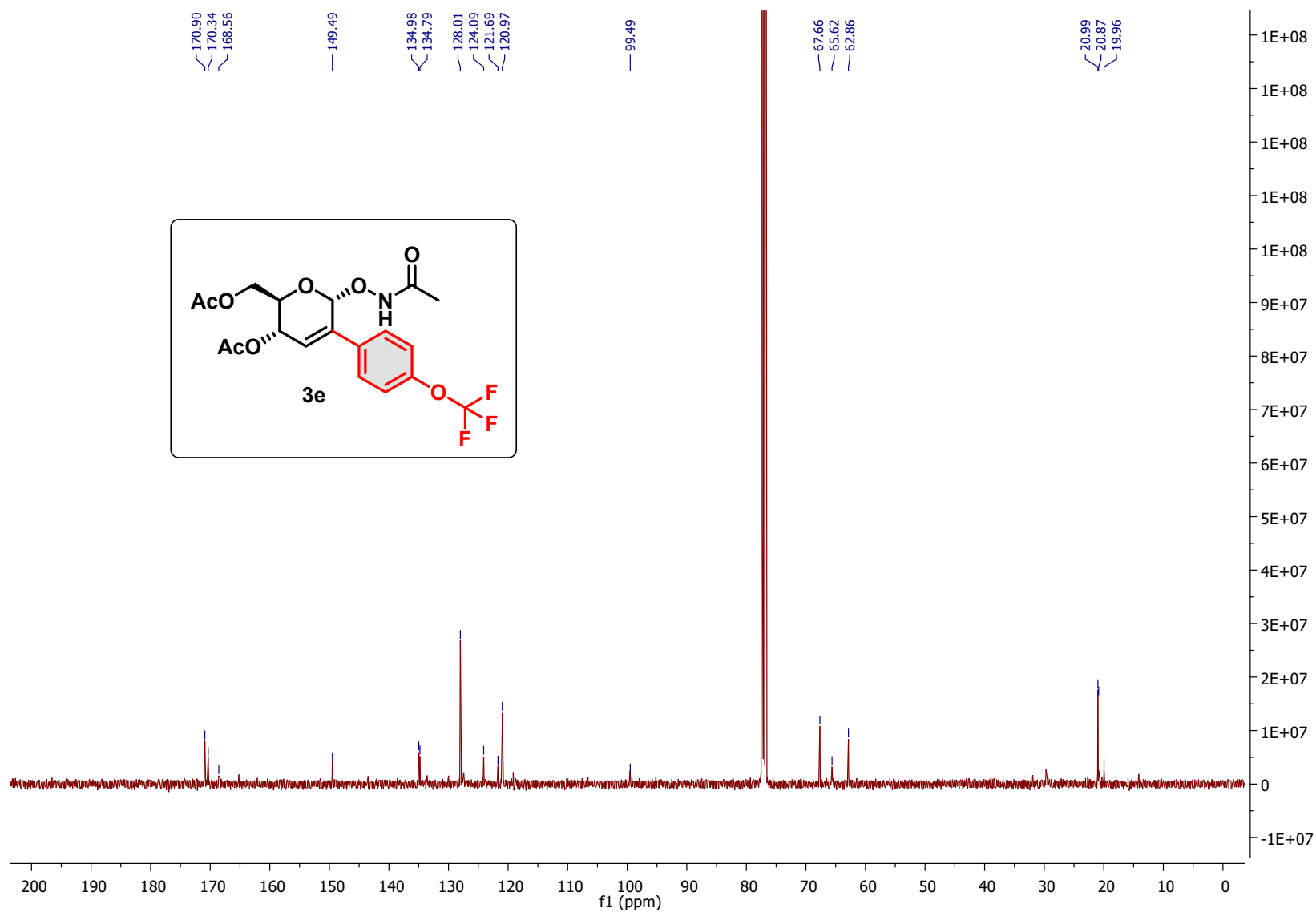
^{13}C NMR (101 MHz) of **3d** in CDCl_3



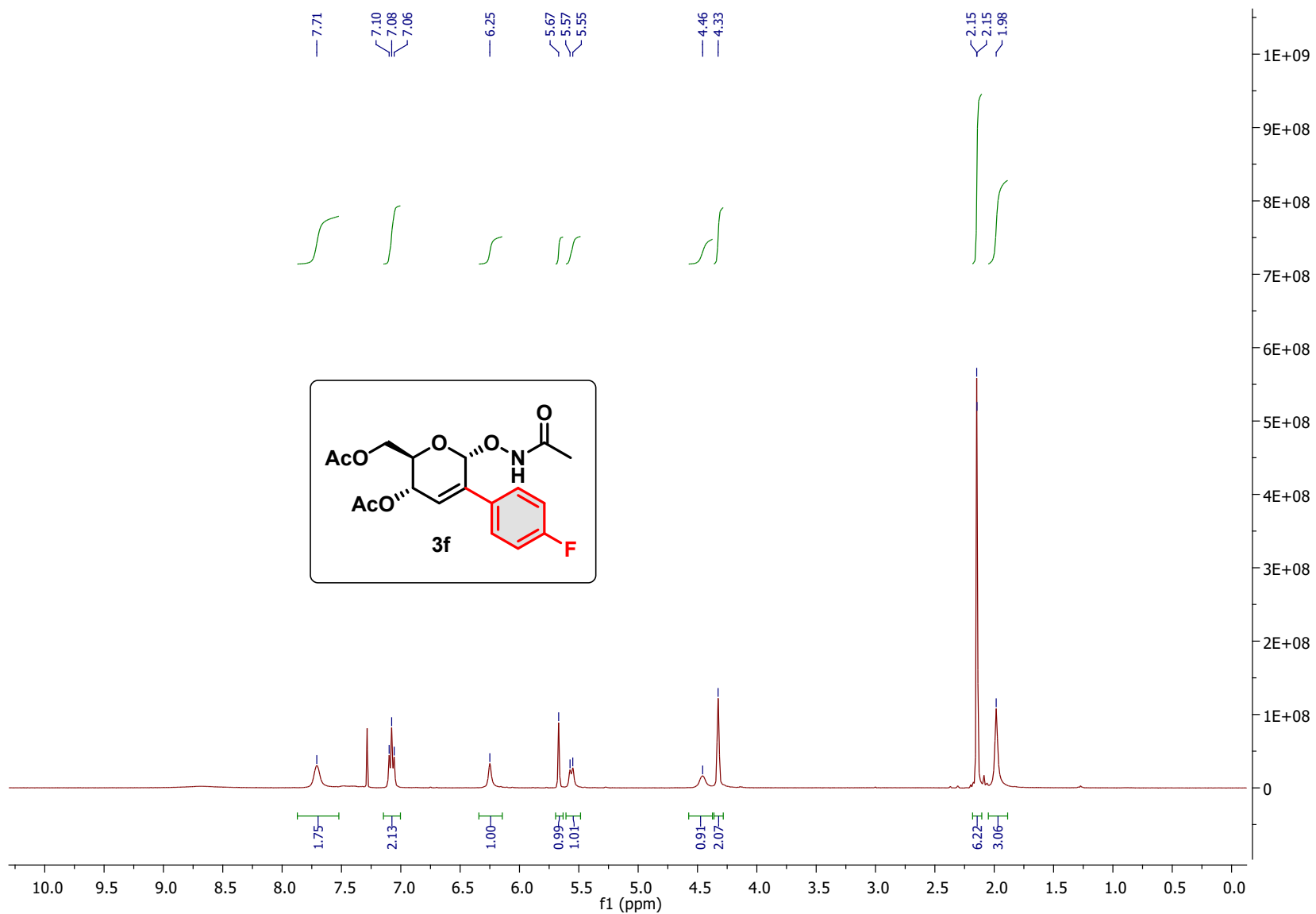
^1H NMR (400 MHz) of **3e** in CDCl_3



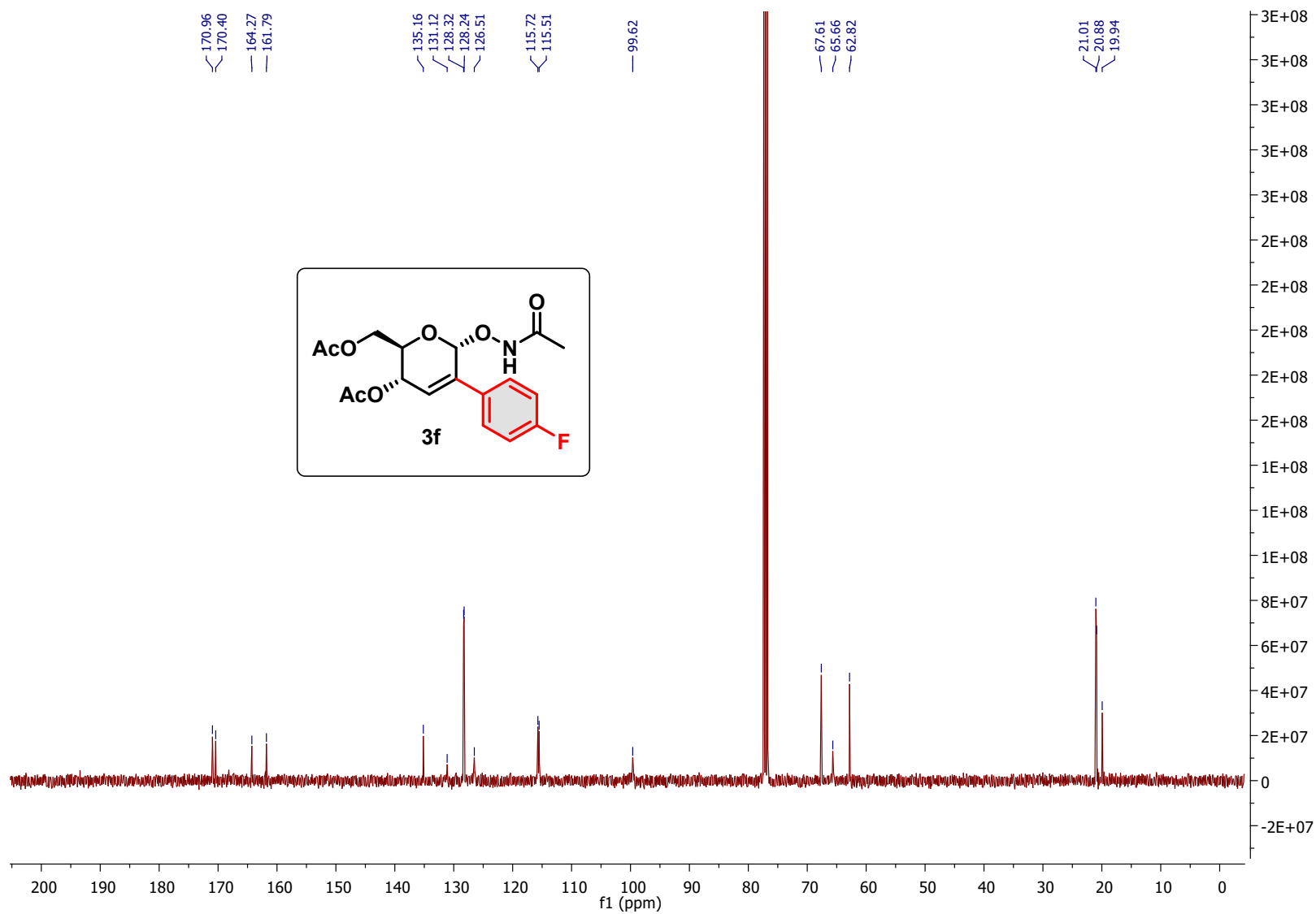
¹³C NMR (101 MHz) of **3e** in CDCl₃



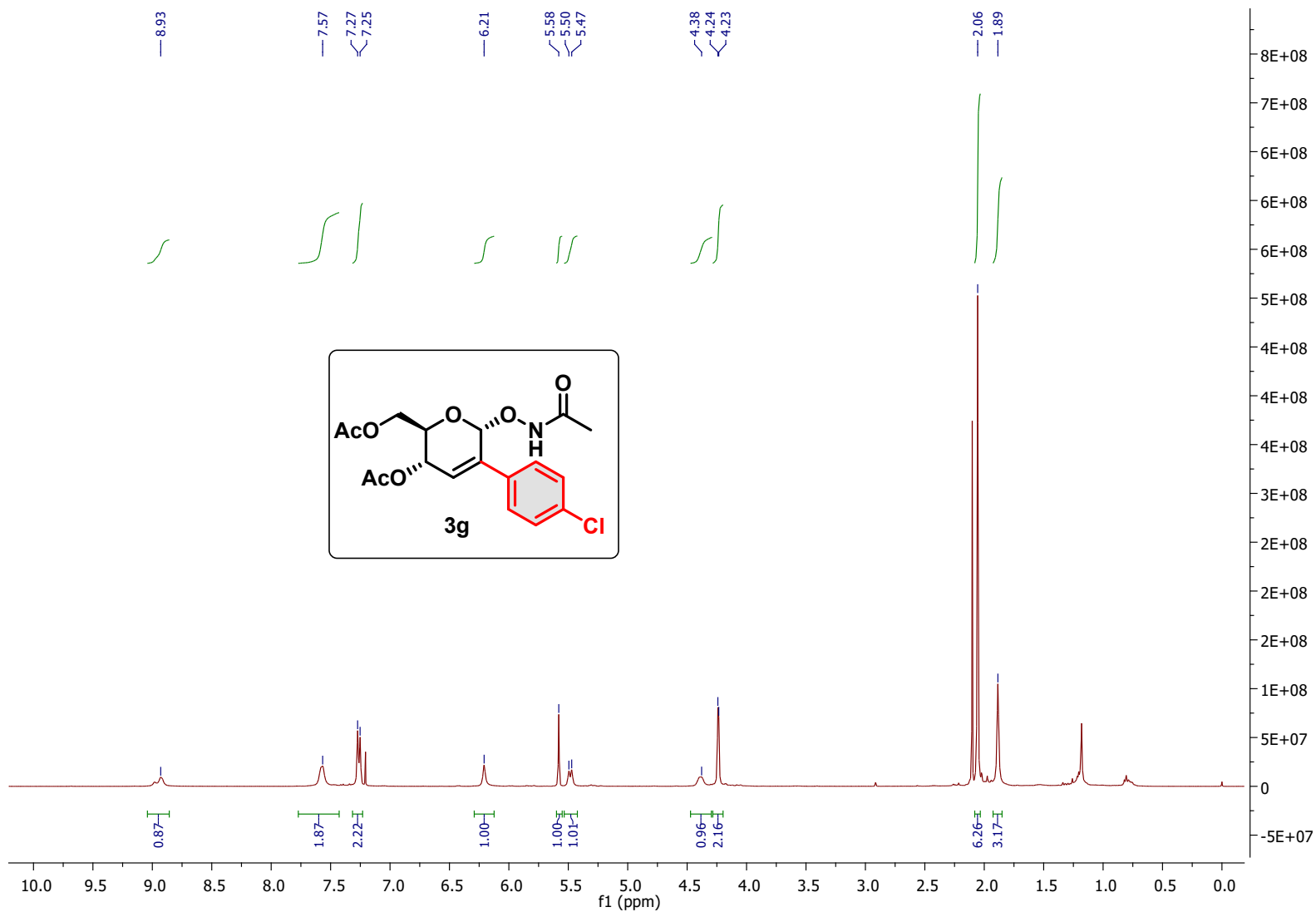
^1H NMR (400 MHz) of **3f** in CDCl_3



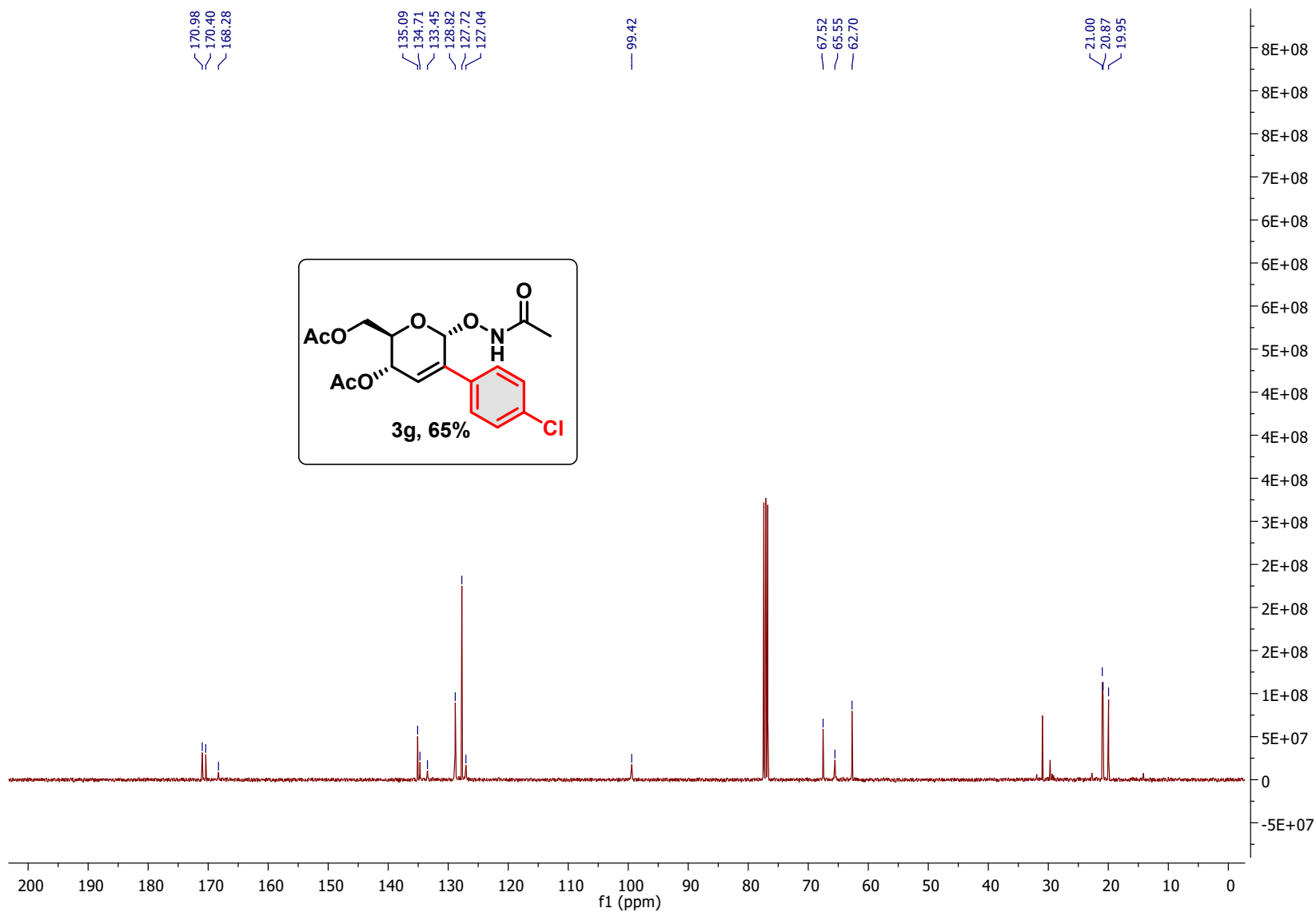
^{13}C NMR (101 MHz) of **3f** in CDCl_3



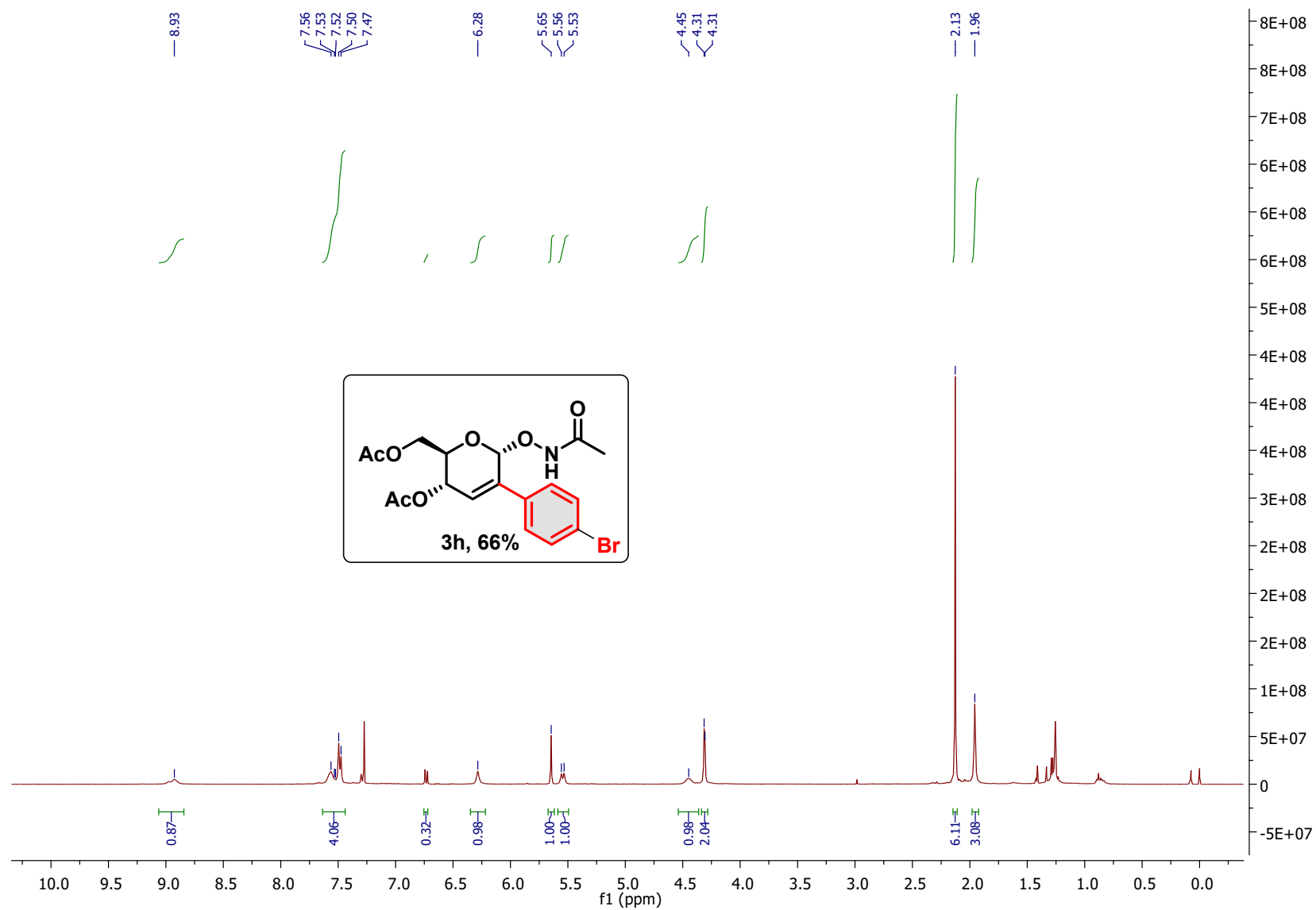
^1H NMR (400 MHz) of **3g** in CDCl_3



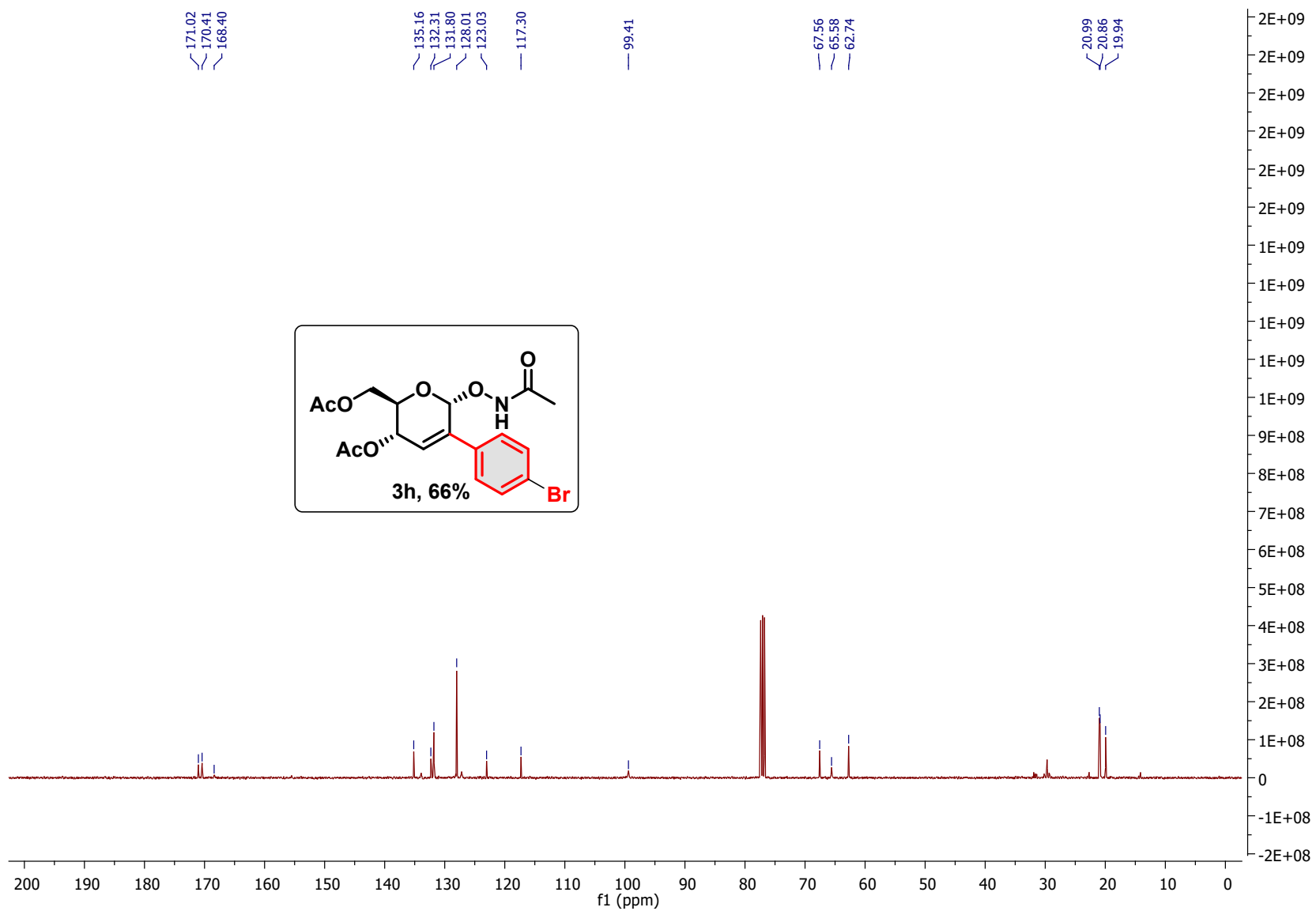
^{13}C NMR (101 MHz) of **3g** in CDCl_3



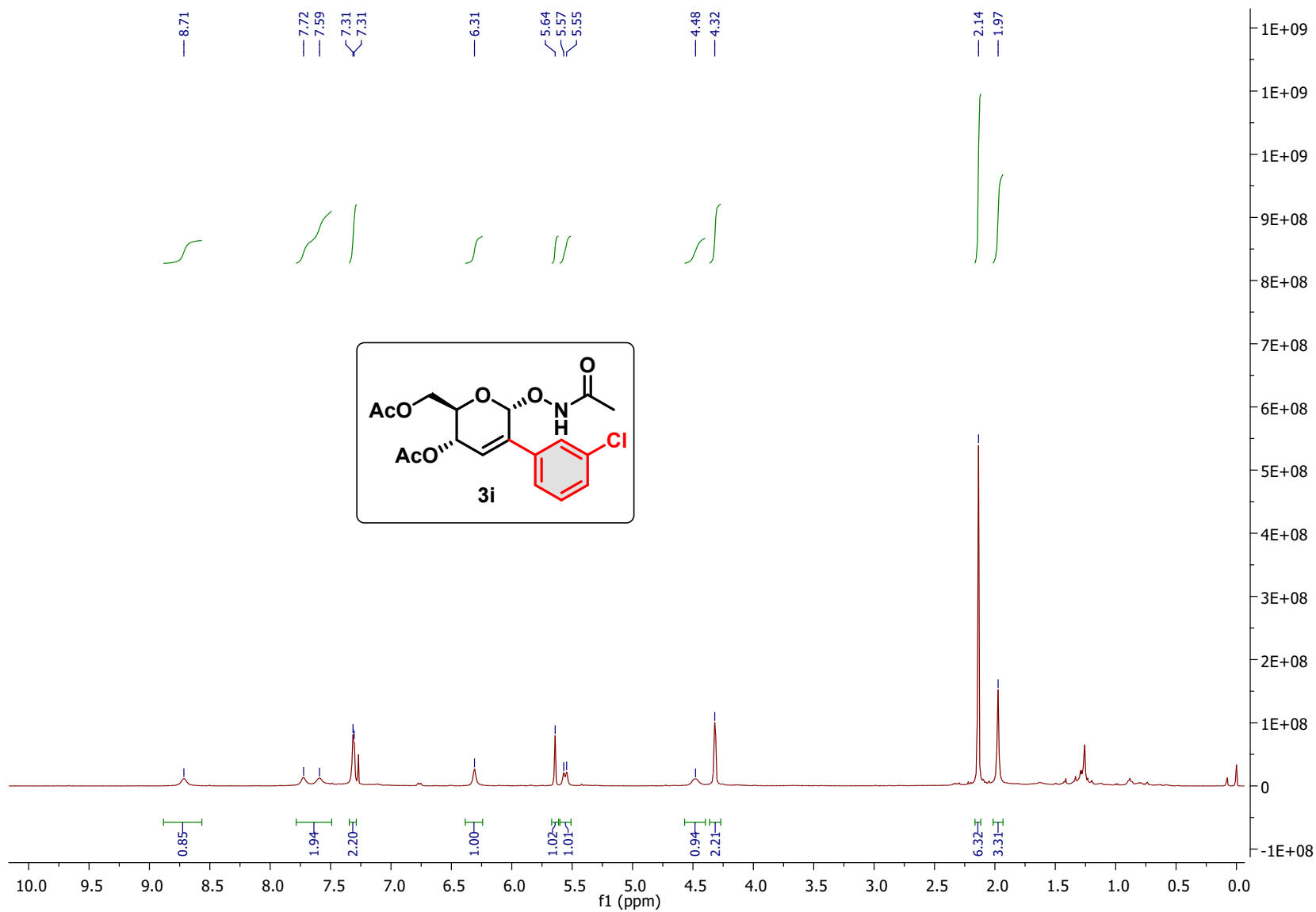
^1H NMR (400 MHz) of **3h** in CDCl_3



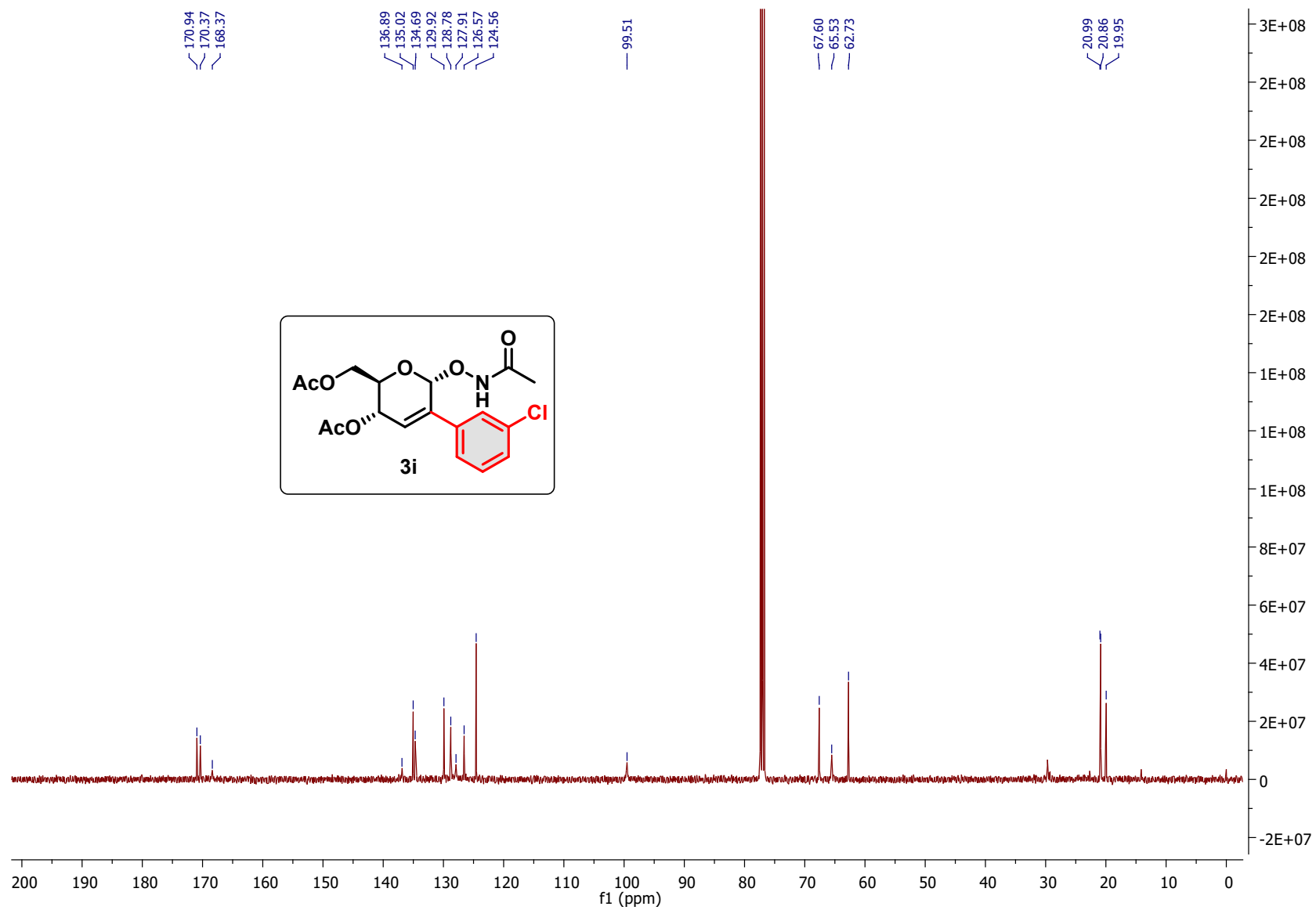
¹³C NMR (101 MHz) of **3h** in CDCl₃



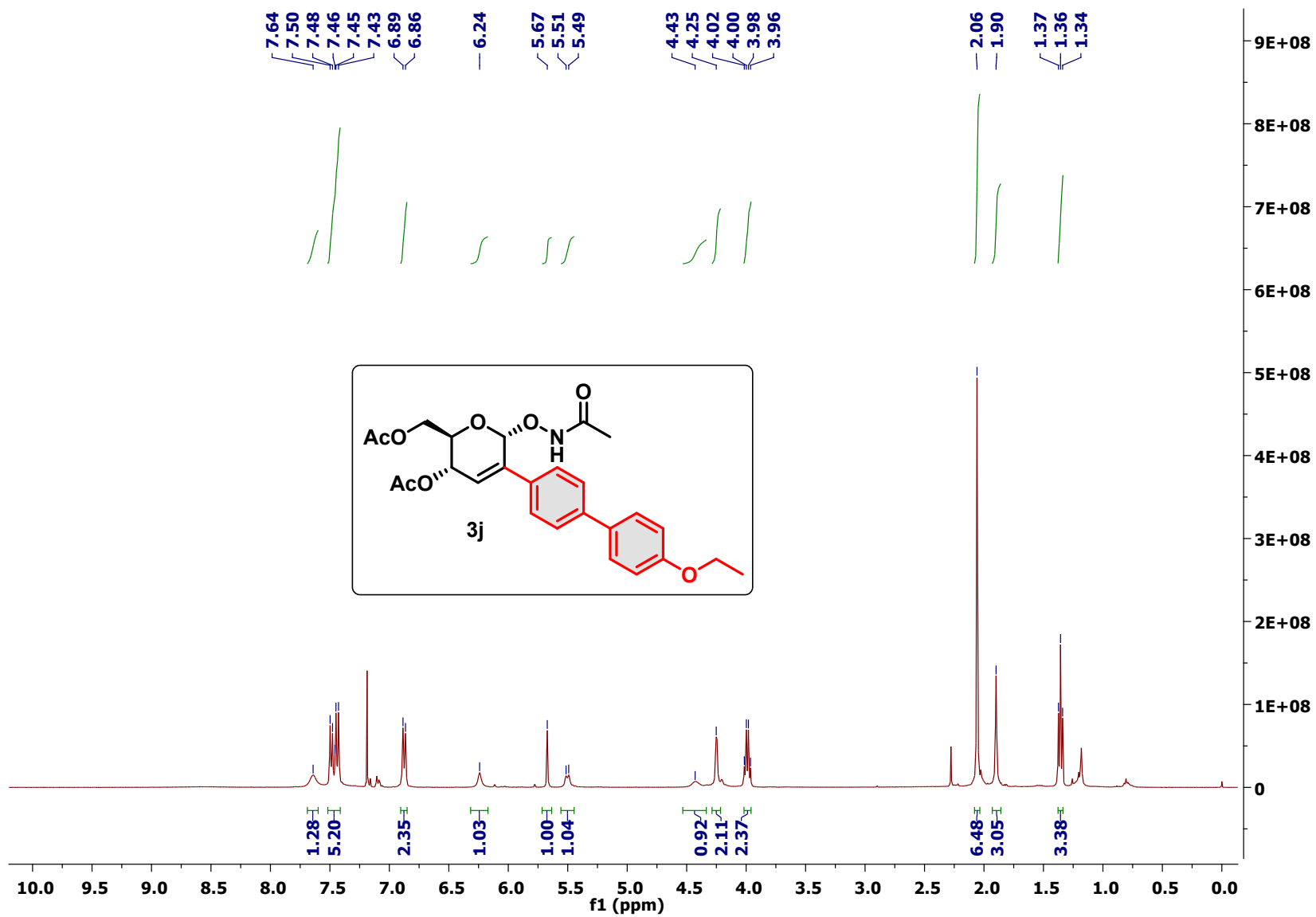
¹H NMR (400 MHz) of **3i** in CDCl₃



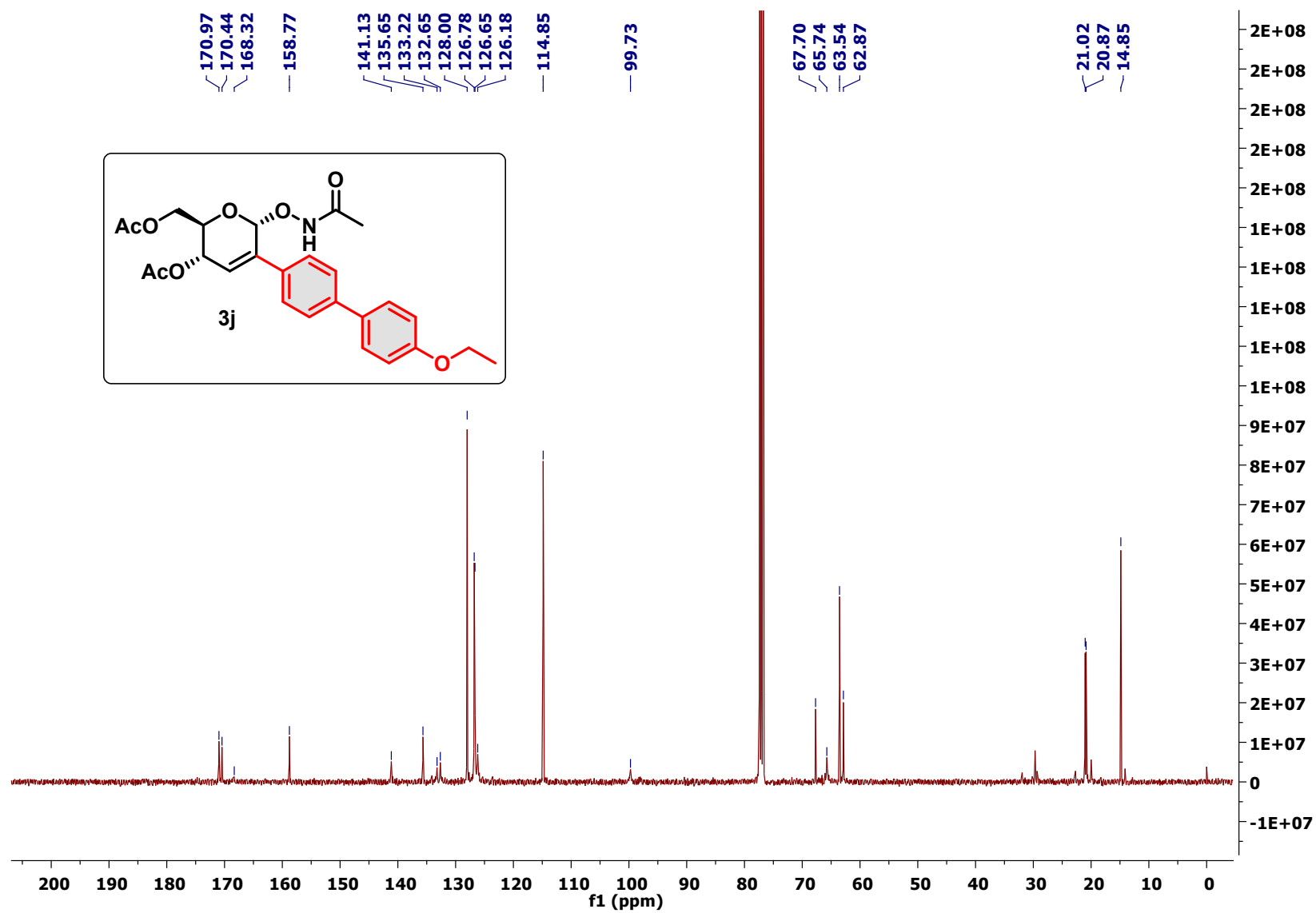
^{13}C NMR (101 MHz) of **3i** in CDCl_3



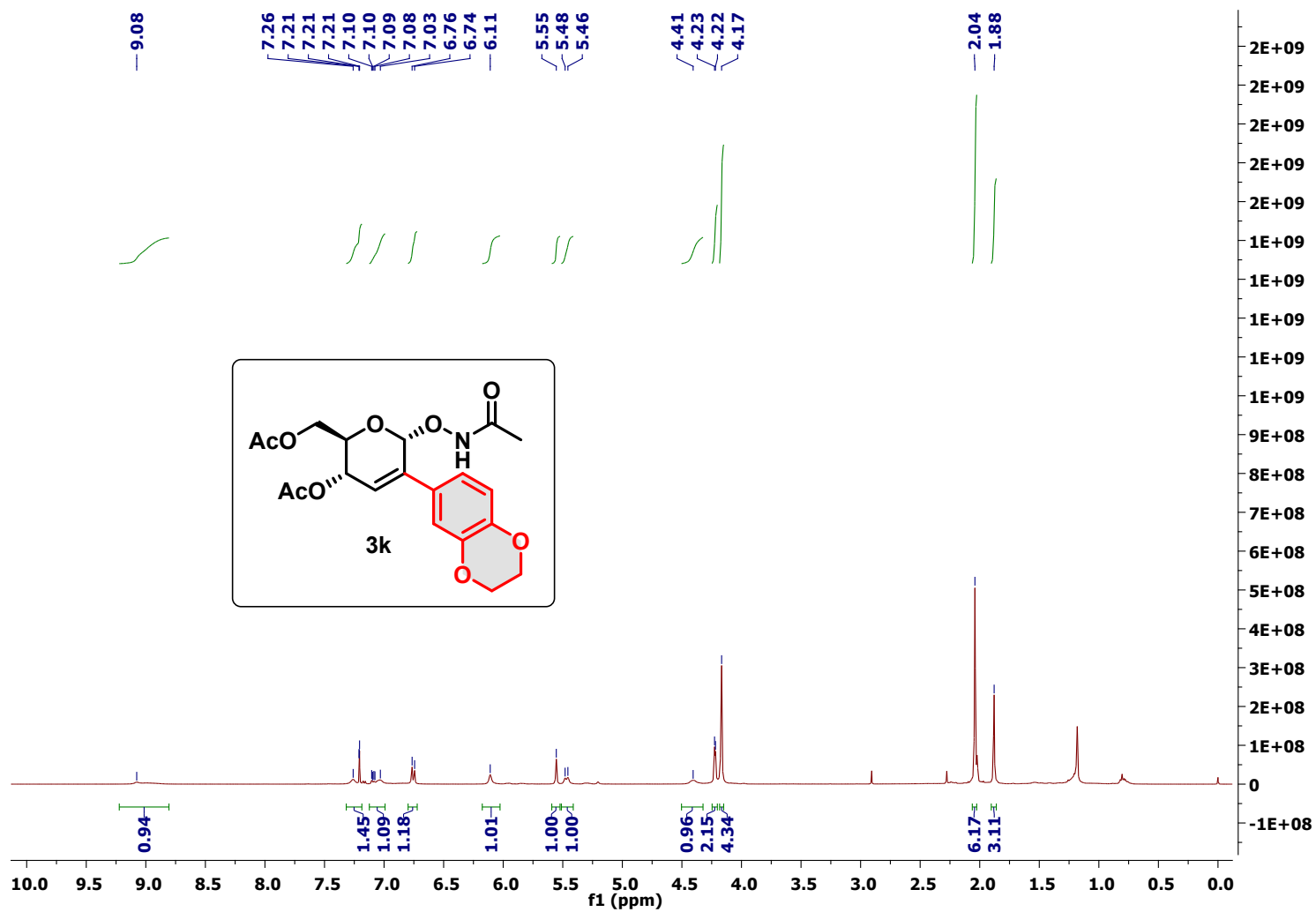
¹H NMR (400 MHz) of **3j** in CDCl₃



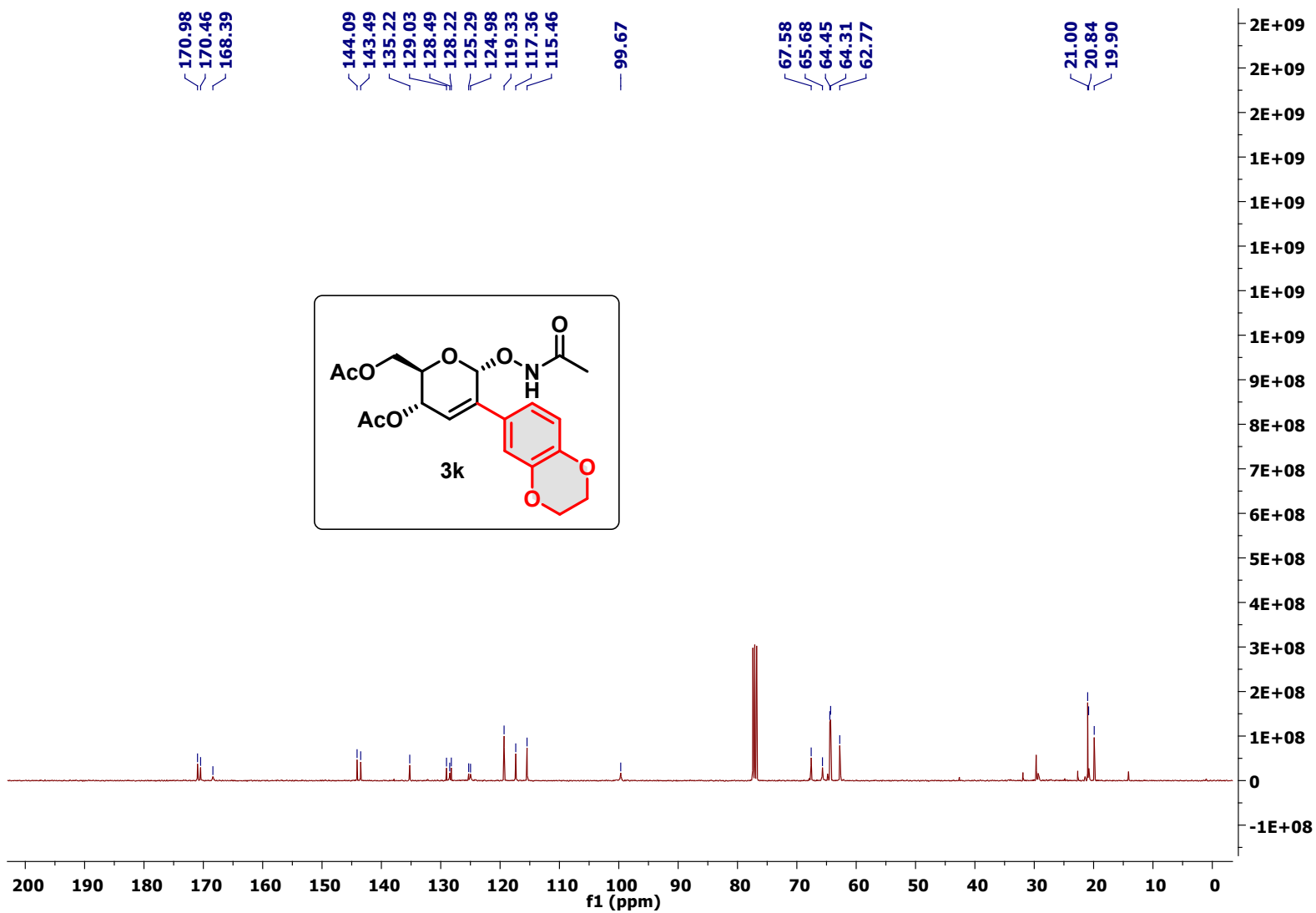
^{13}C NMR (101 MHz) of **3j** in CDCl_3



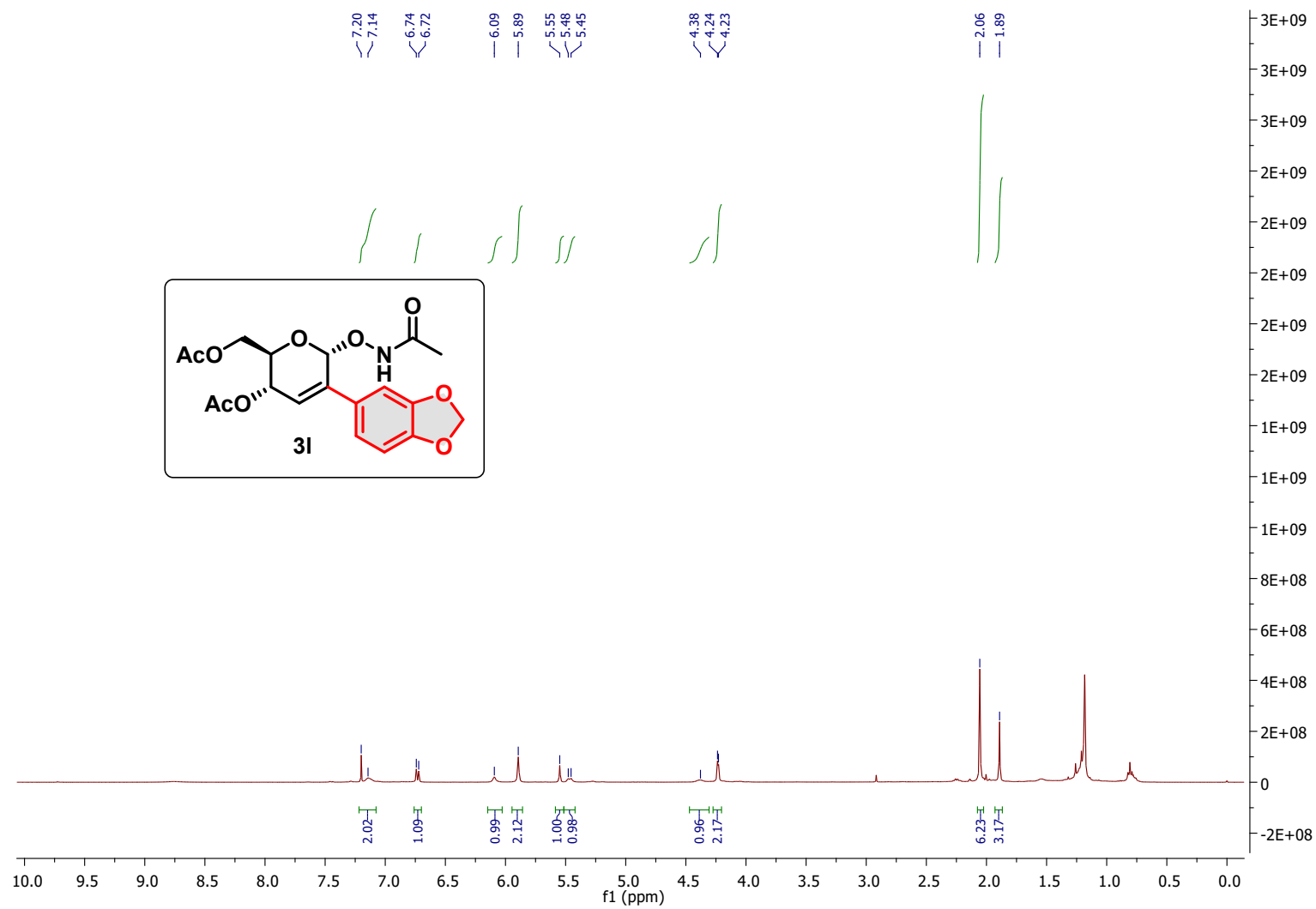
¹H NMR (400 MHz) of **3k** in CDCl₃



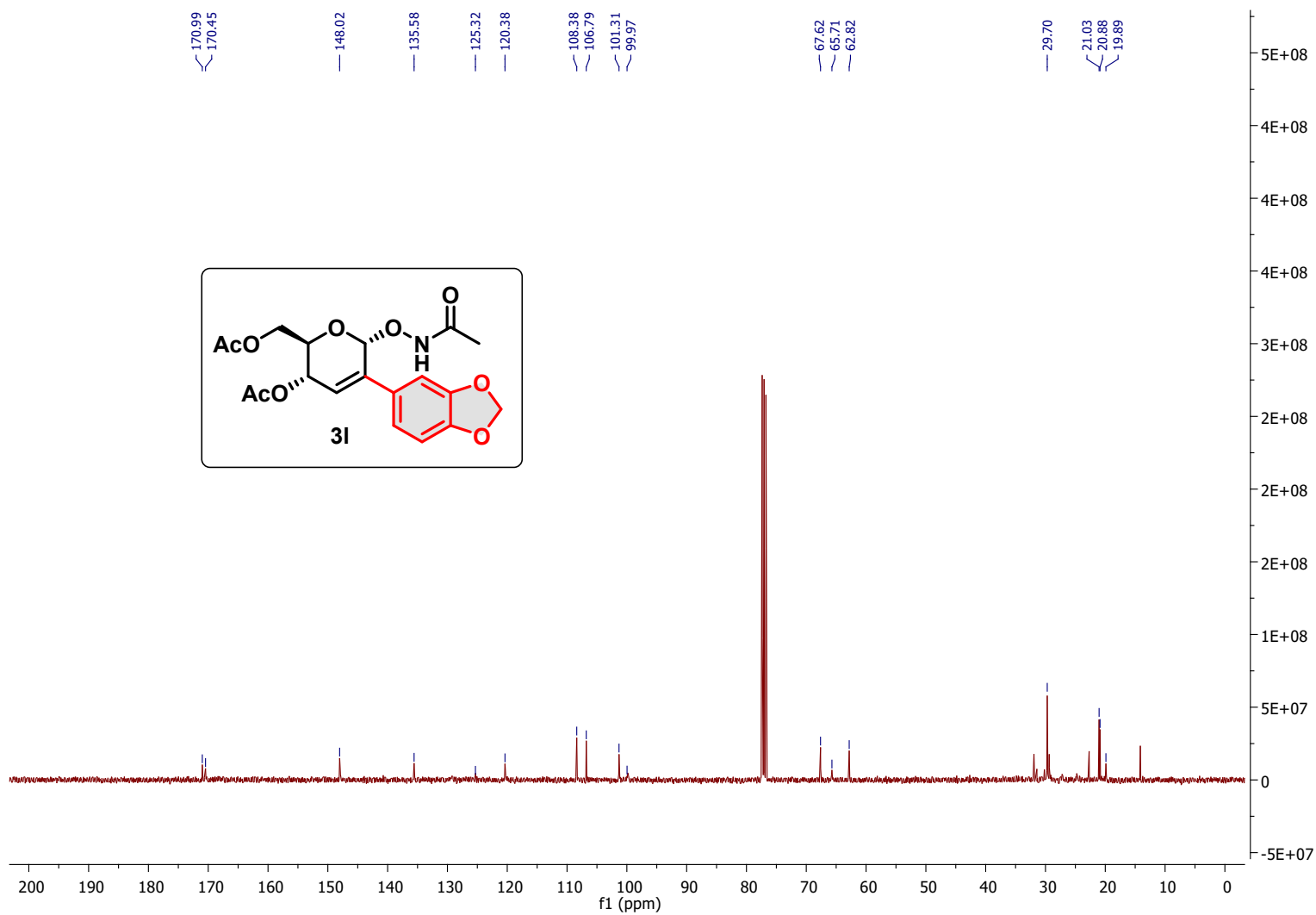
^{13}C NMR (101 MHz) of **3k** in CDCl_3



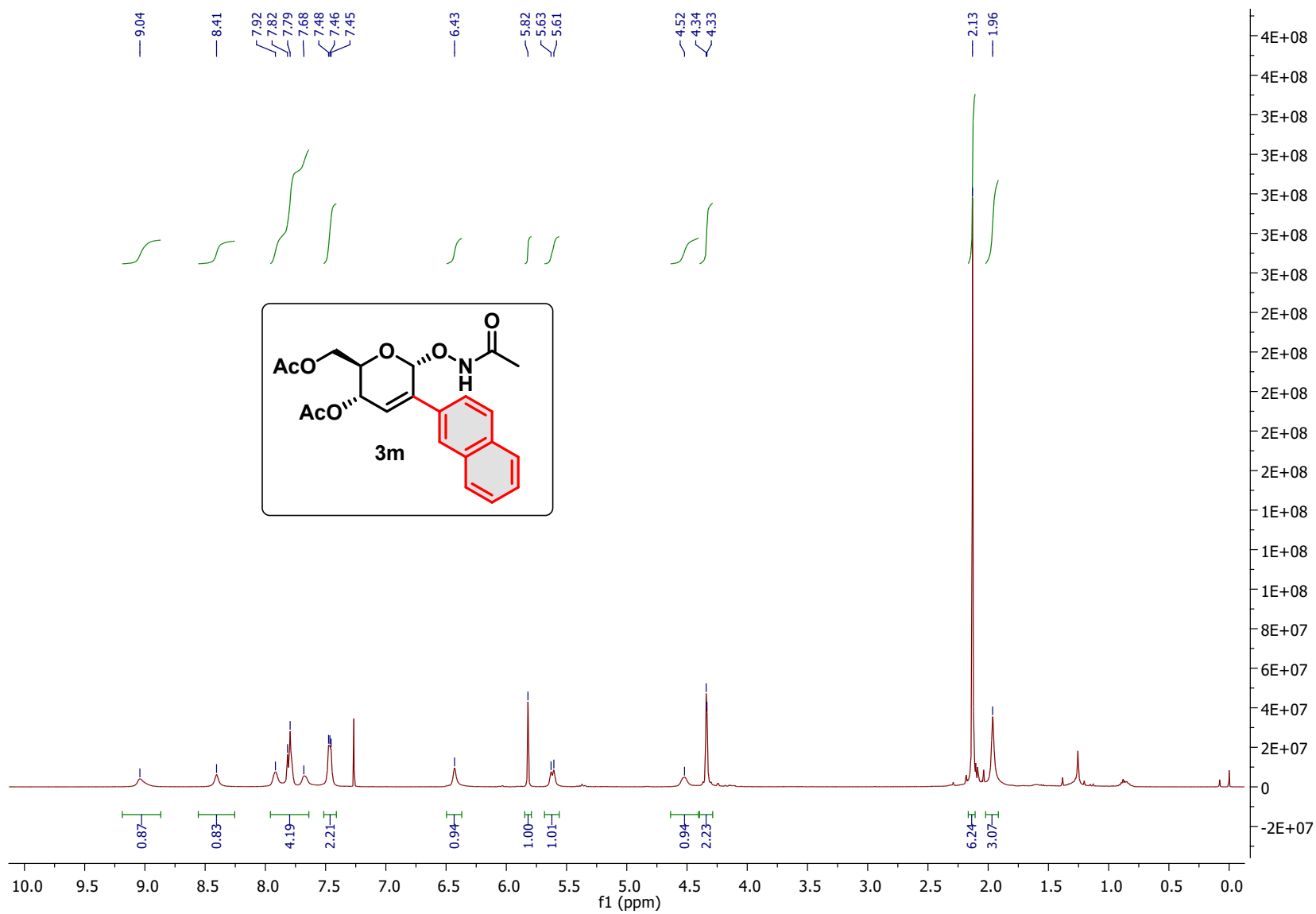
^1H NMR (400 MHz) of **31** in CDCl_3



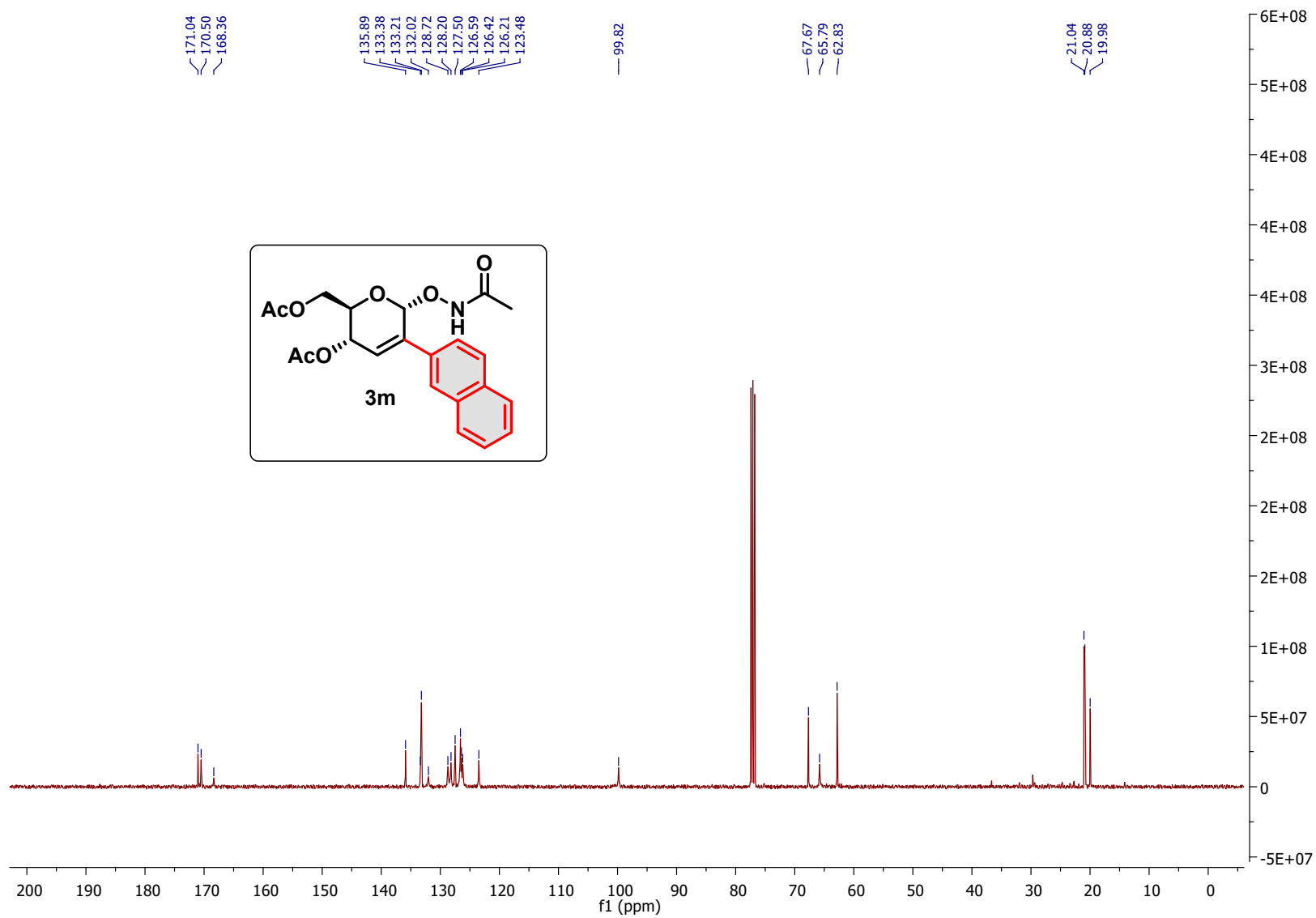
^{13}C NMR (101 MHz) of **31** in CDCl_3



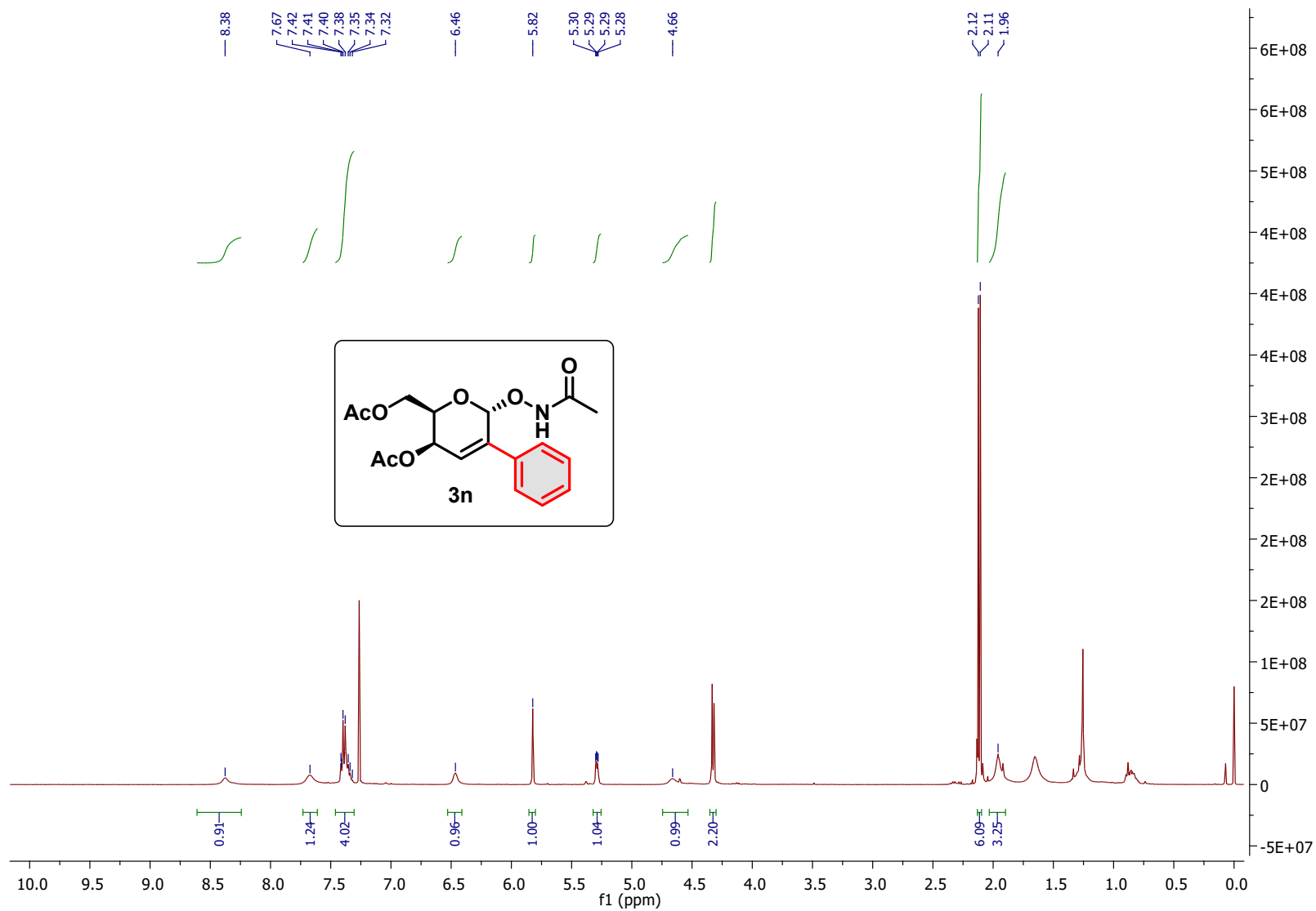
¹H NMR (400 MHz) of **3m** in CDCl₃



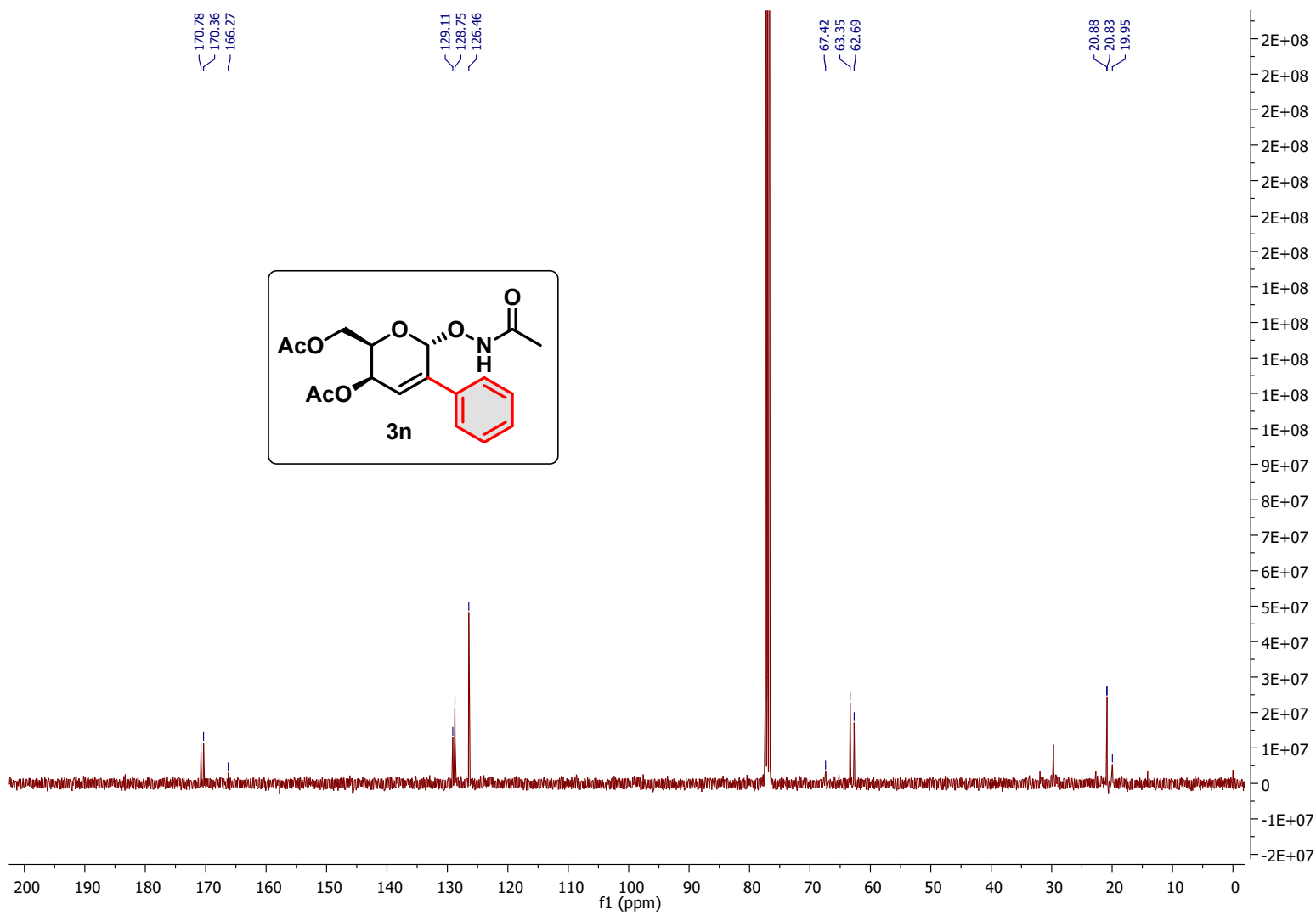
^{13}C NMR (101 MHz) of **3m** in CDCl_3



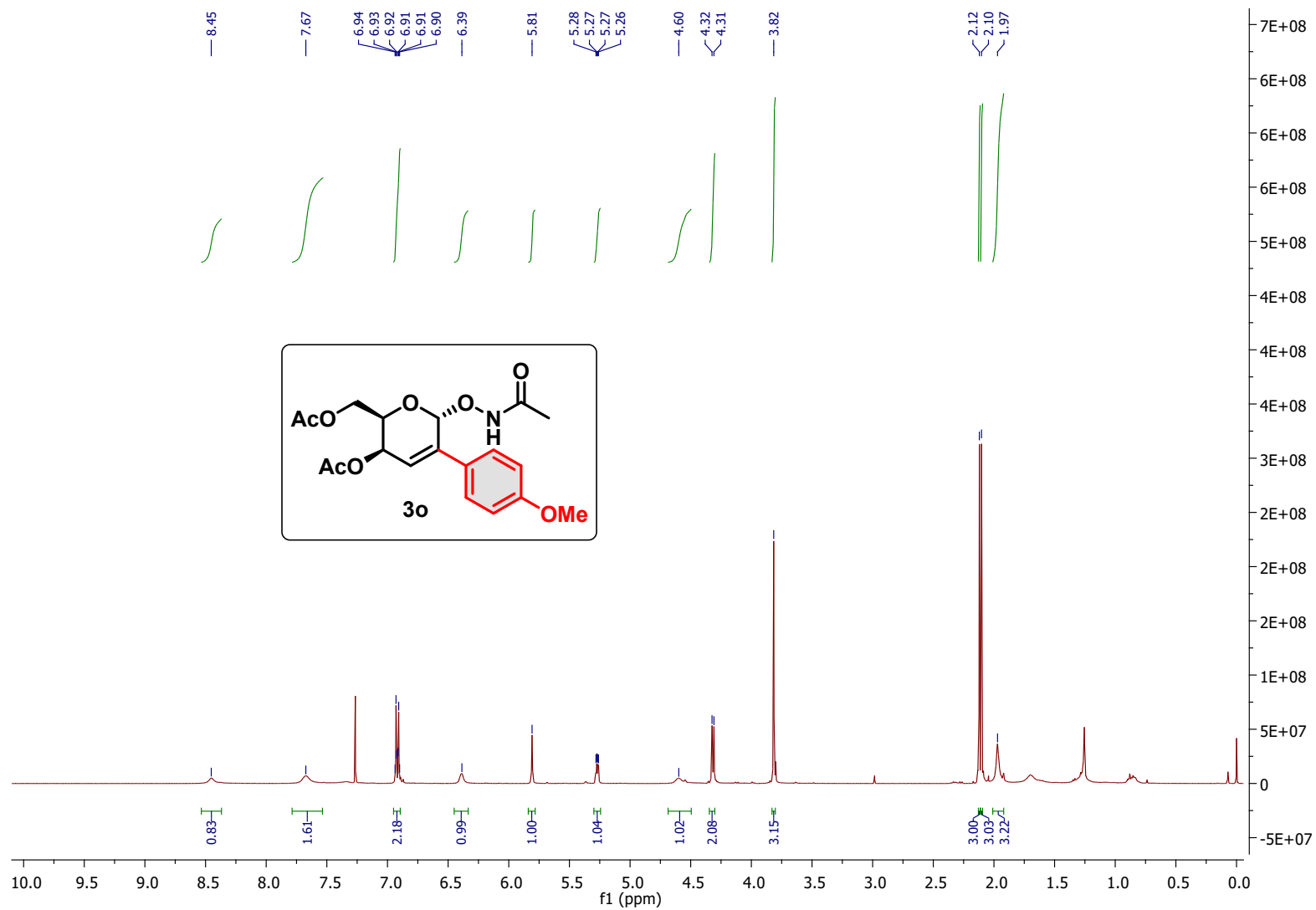
¹H NMR (400 MHz) of **3n** in CDCl₃



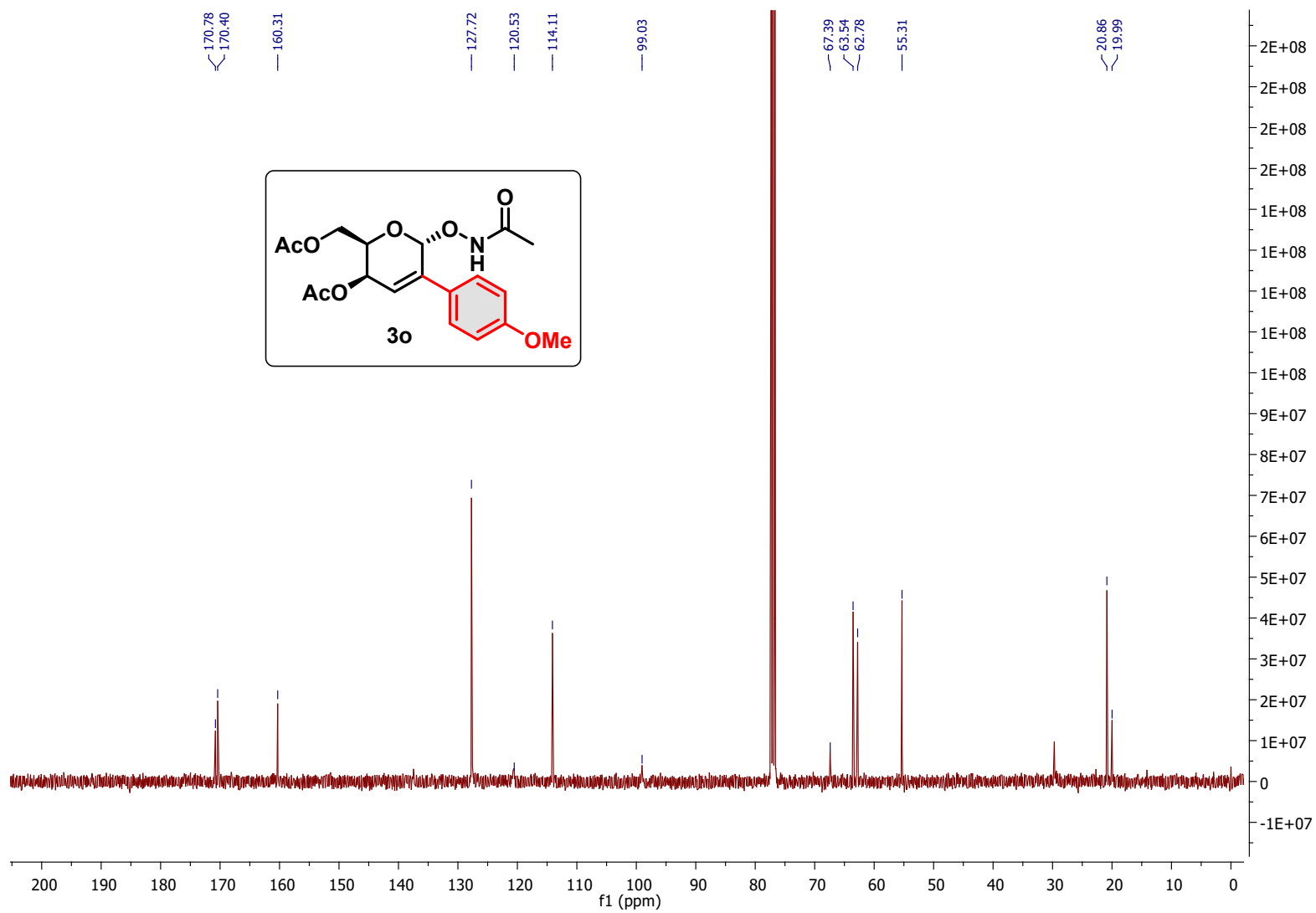
^{13}C NMR (101 MHz) of **3n** in CDCl_3



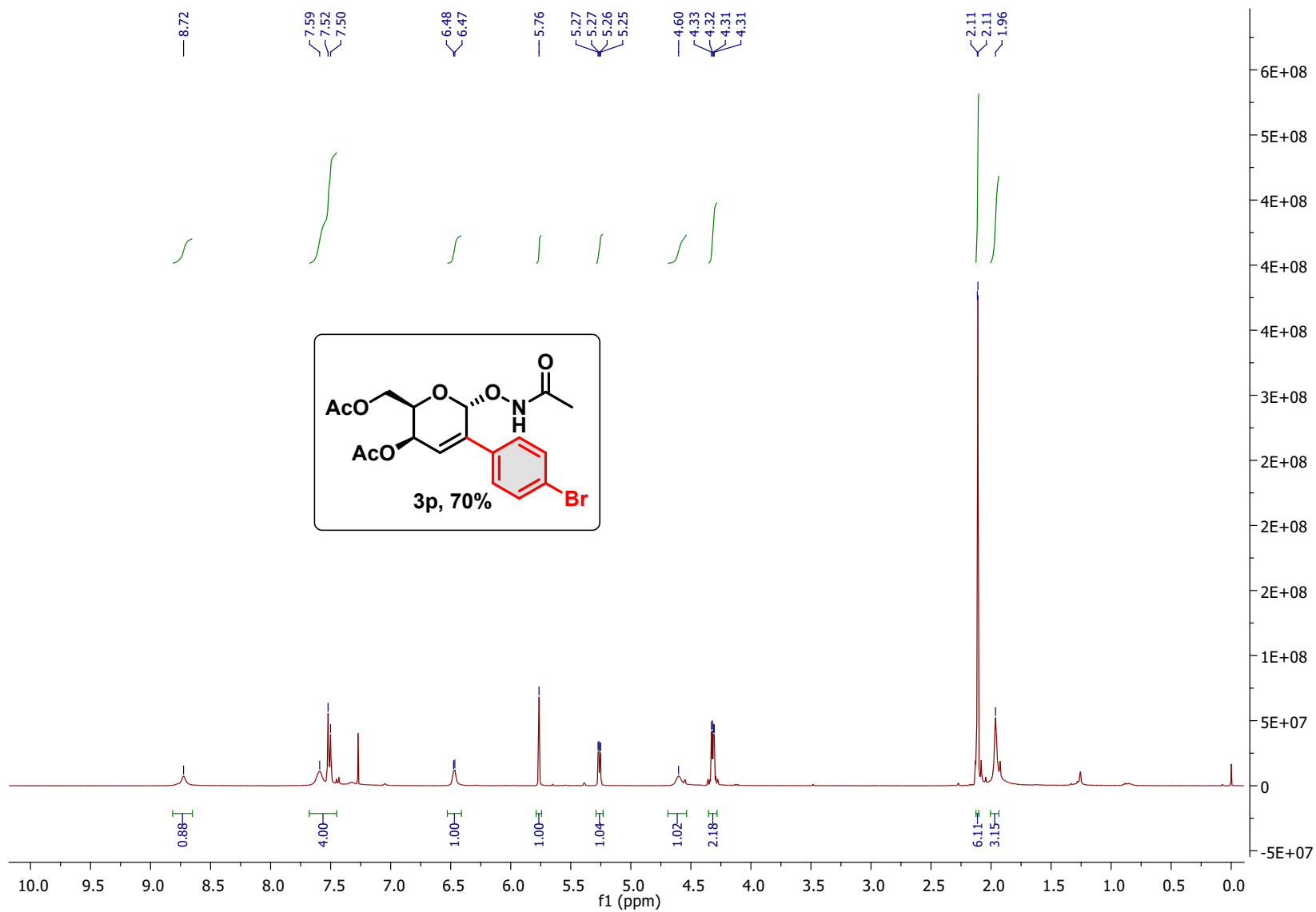
¹H NMR (400 MHz) of **3o** in CDCl₃



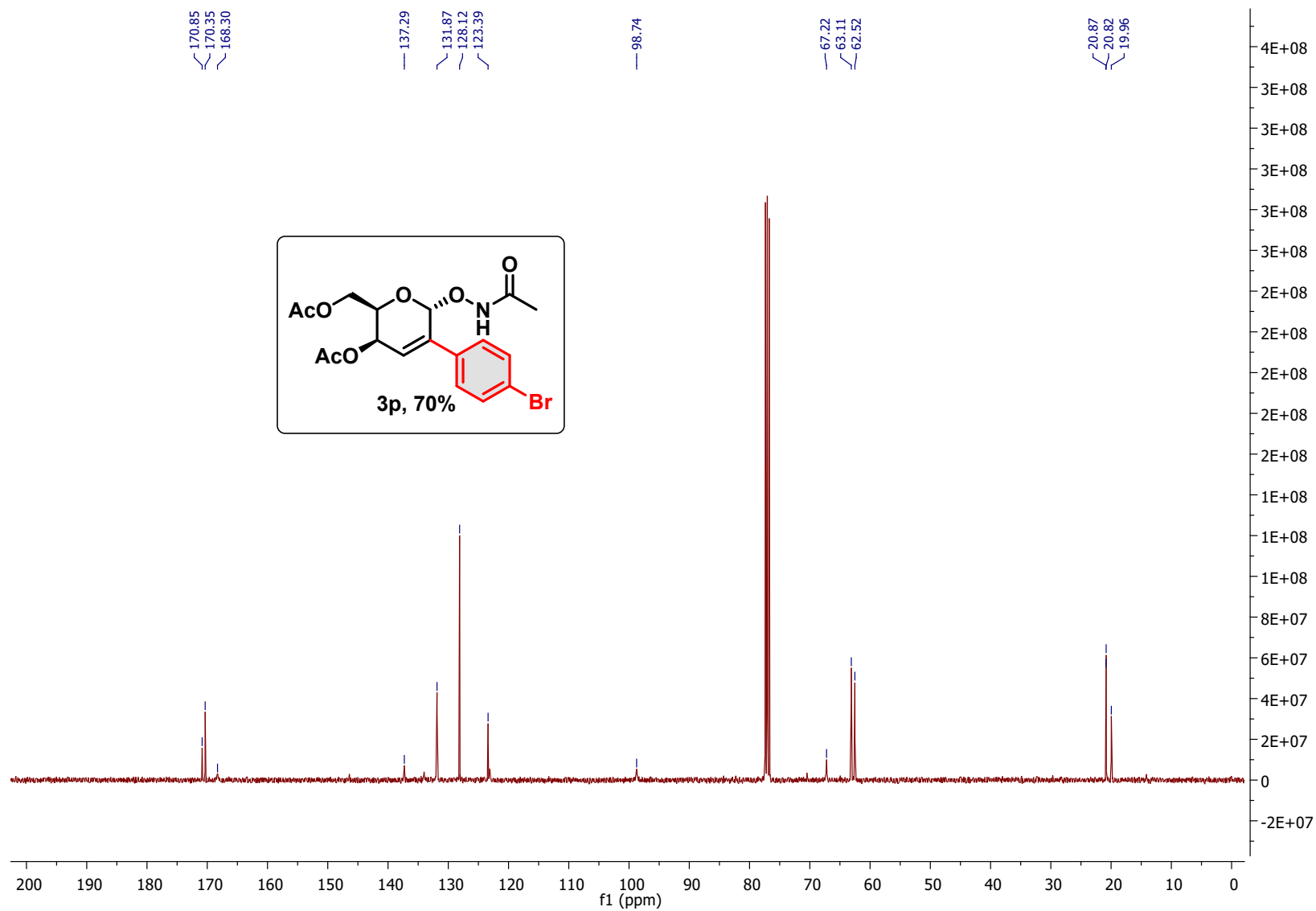
^{13}C NMR (101 MHz) of **3o** in CDCl_3



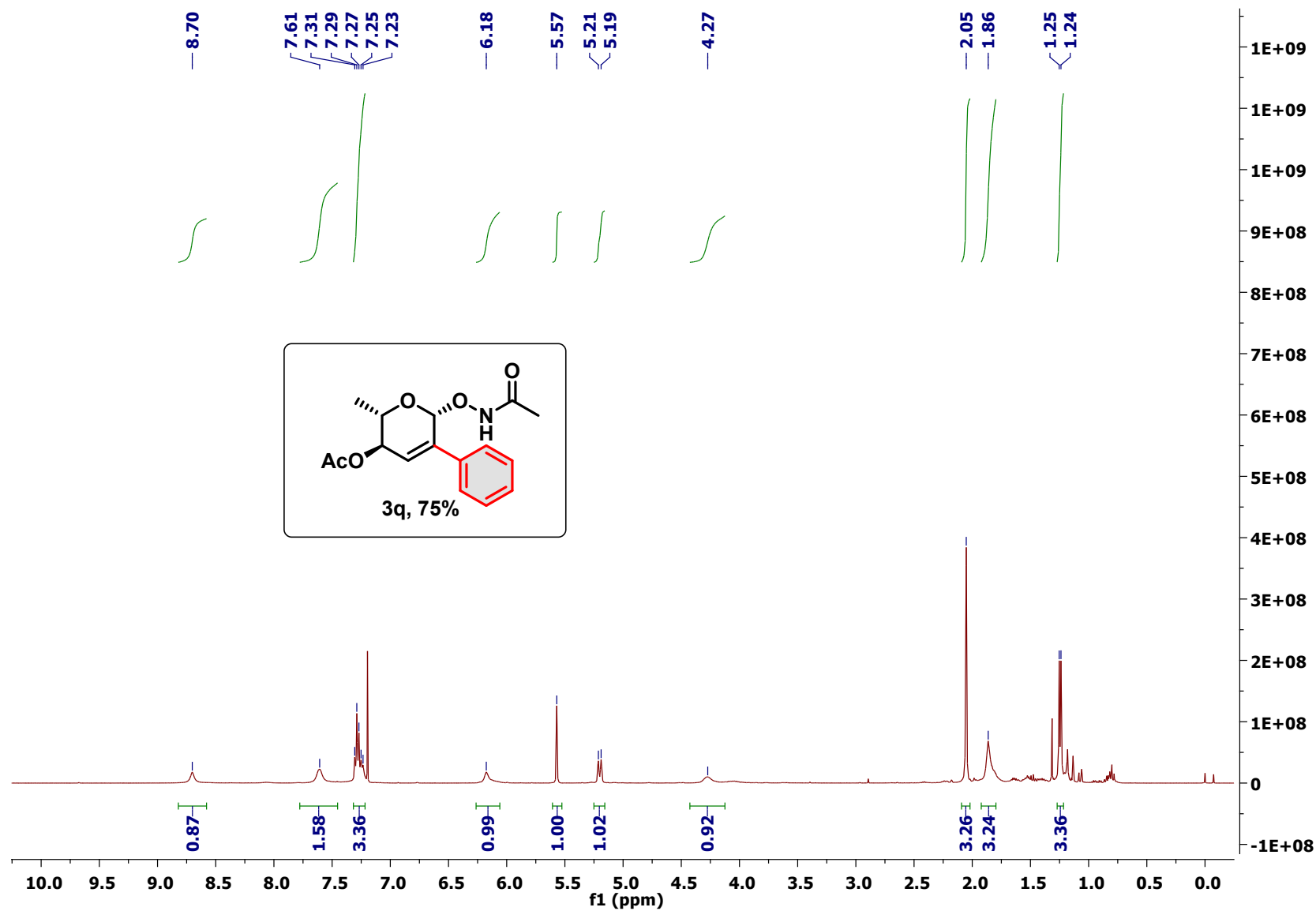
^1H NMR (400 MHz) of **3p** in CDCl_3



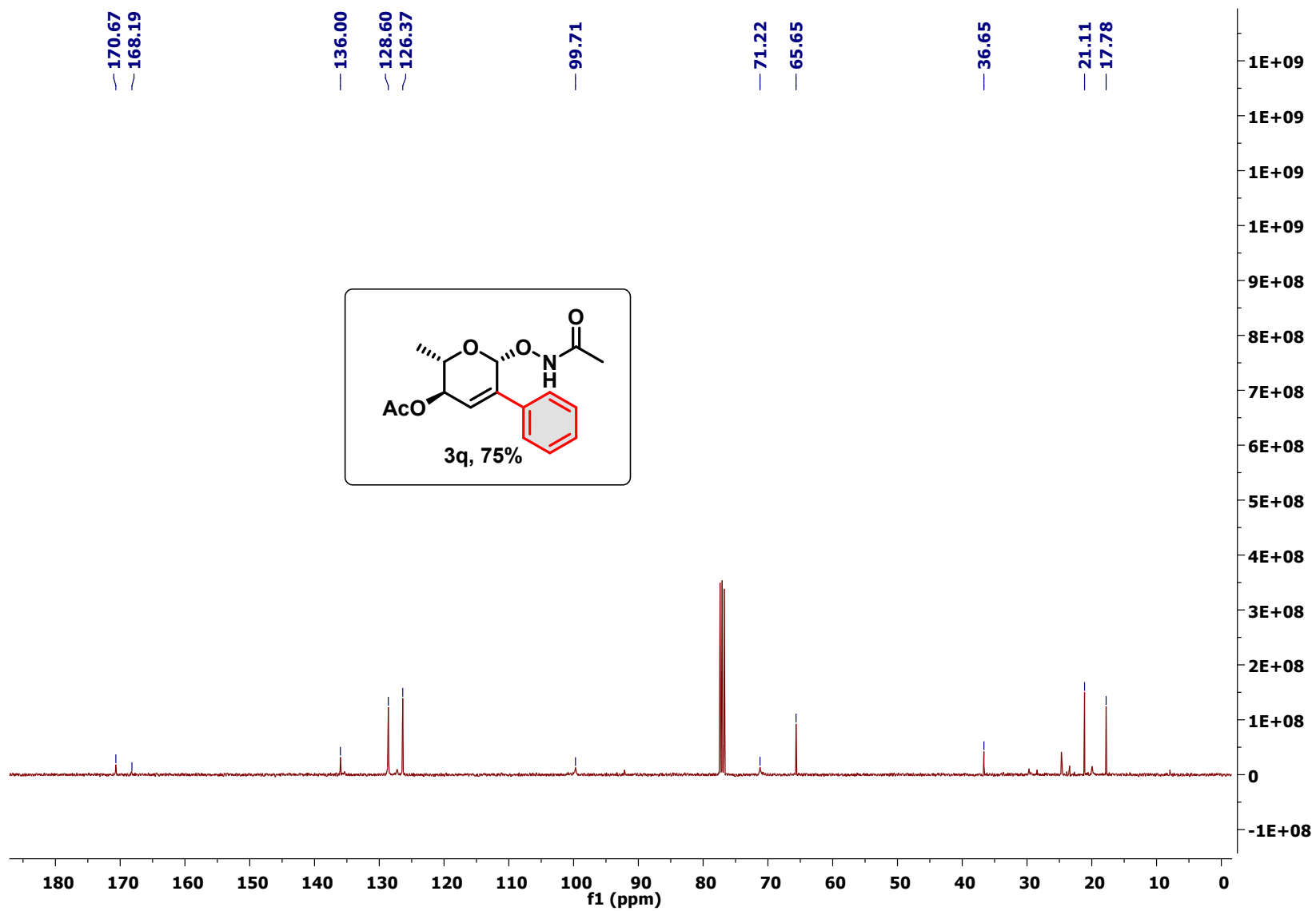
^{13}C NMR (101 MHz) of **3p** in CDCl_3



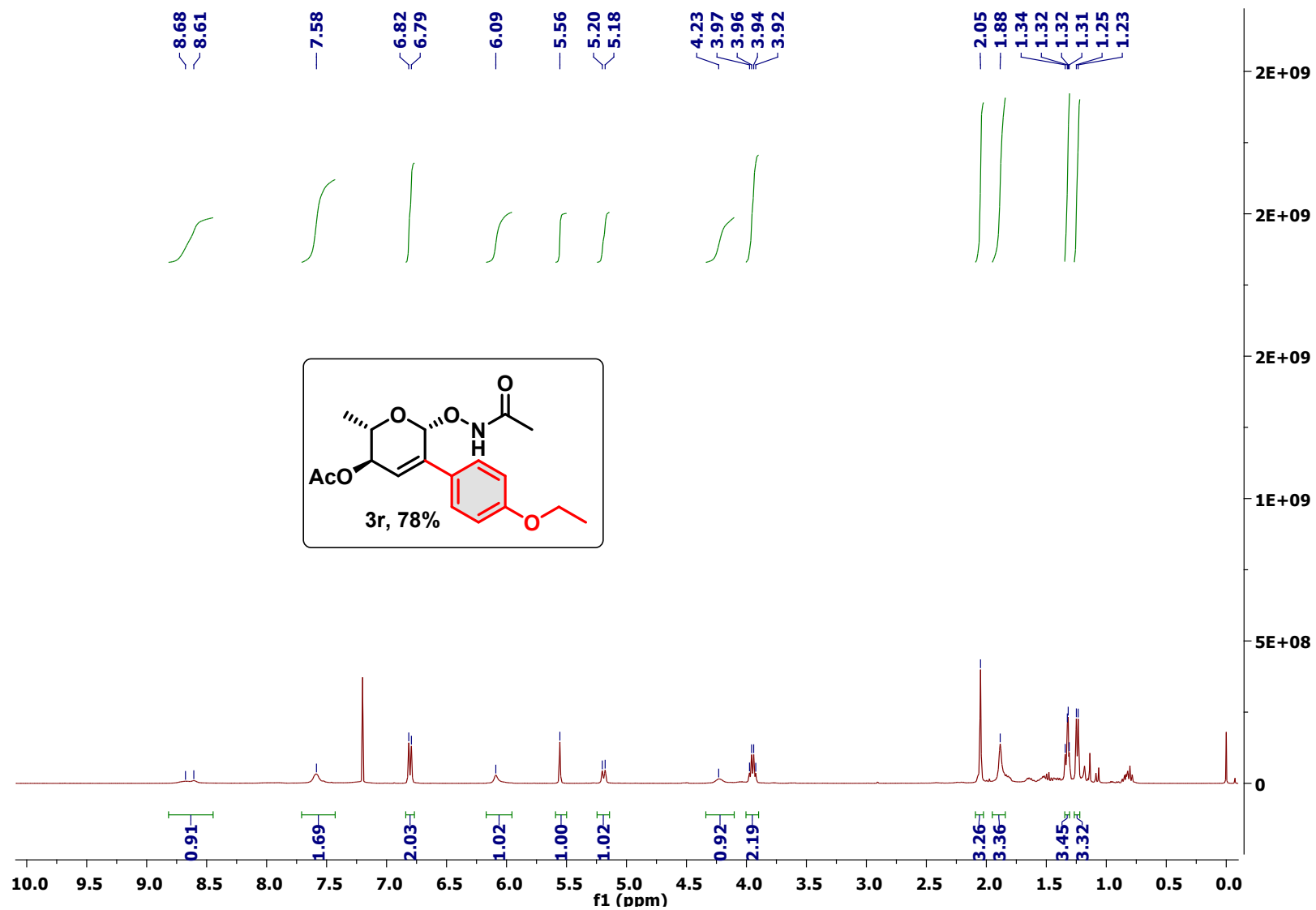
^1H NMR (400 MHz) of **3q** in CDCl_3



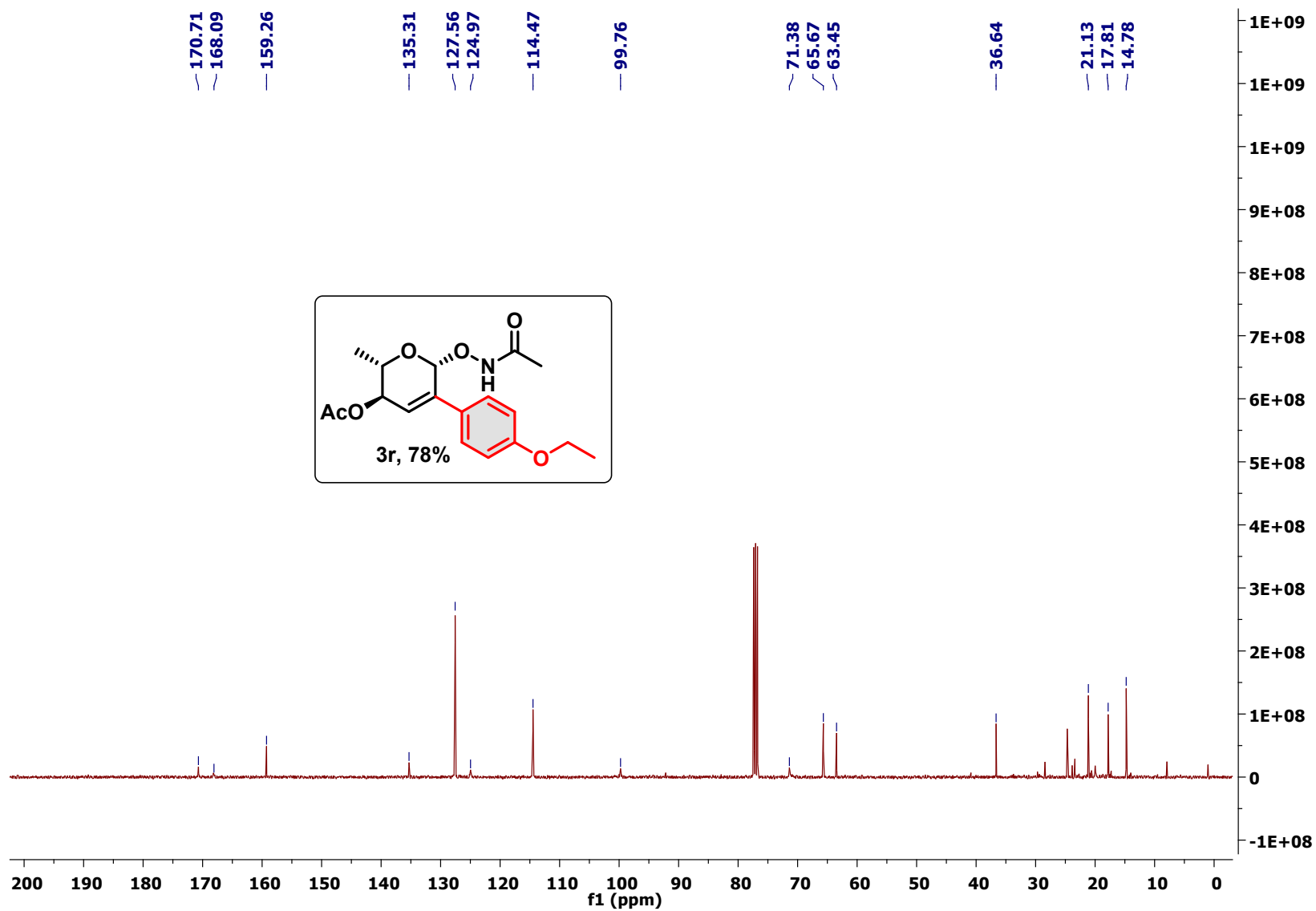
^{13}C NMR (101 MHz) of **3q** in CDCl_3



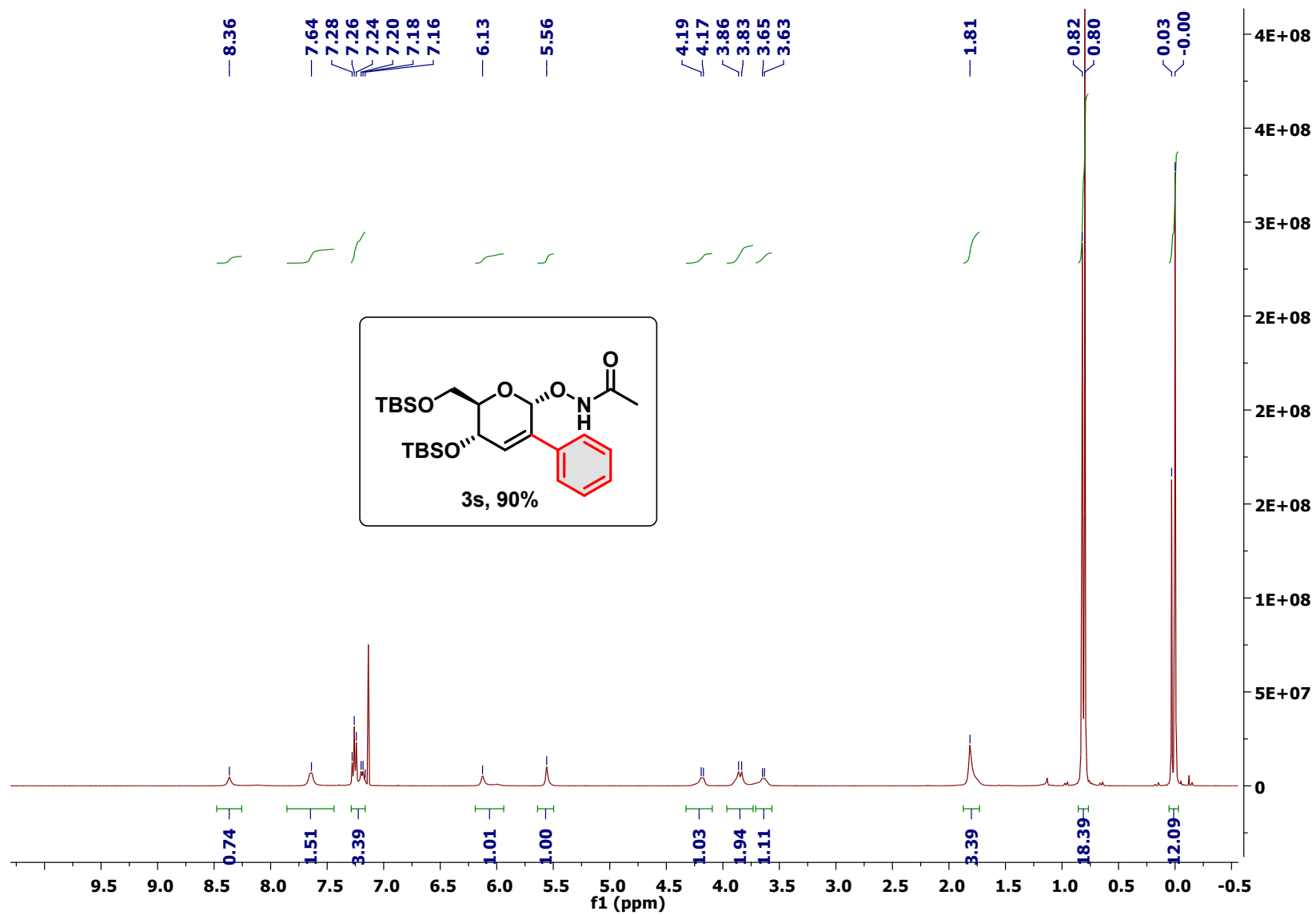
^1H NMR (400 MHz) of **3r** in CDCl_3



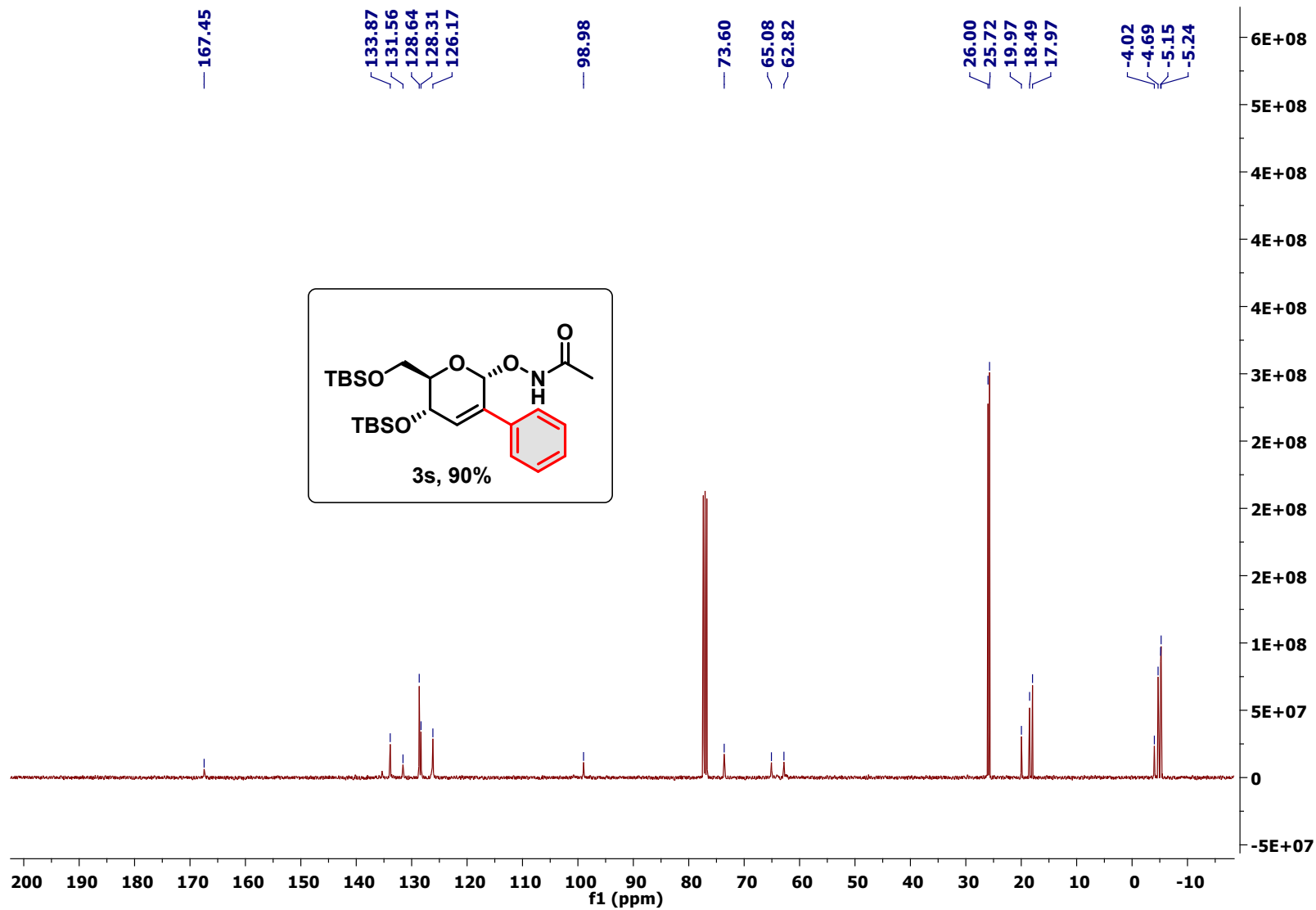
¹³C NMR (101 MHz) of **3r** in CDCl₃



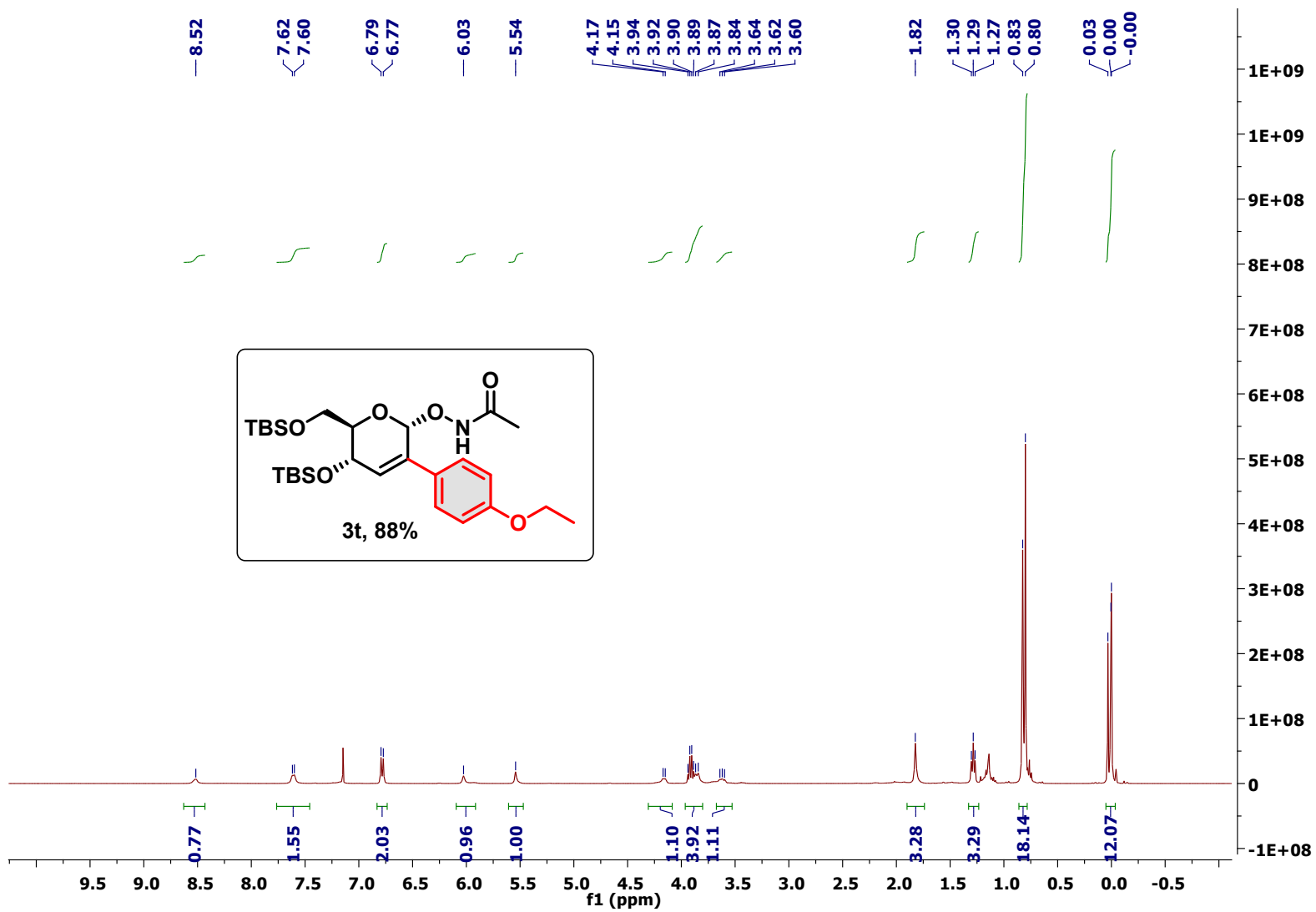
¹H NMR (400 MHz) of **3s** in CDCl₃



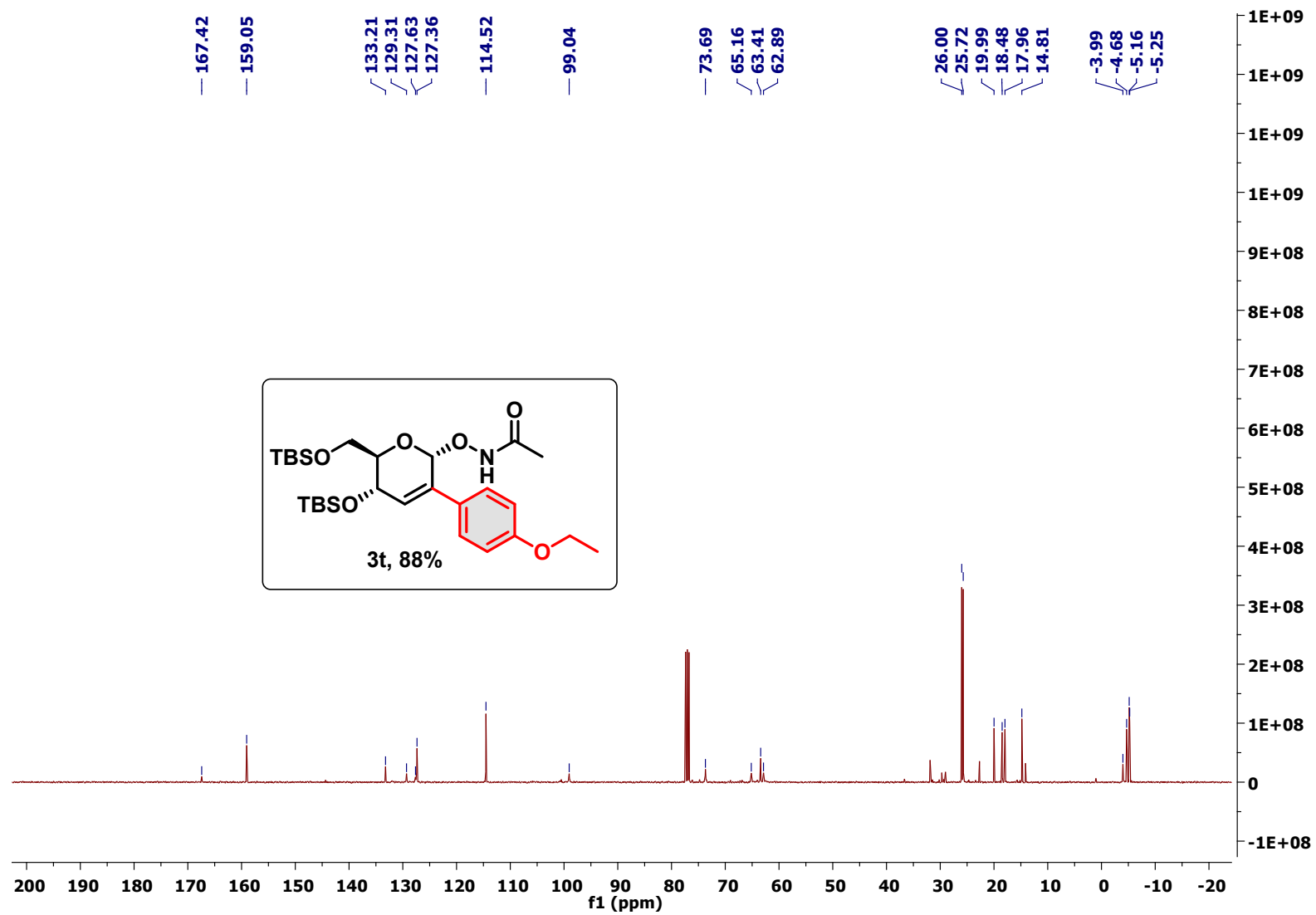
^{13}C NMR (101 MHz) of **3s** in CDCl_3



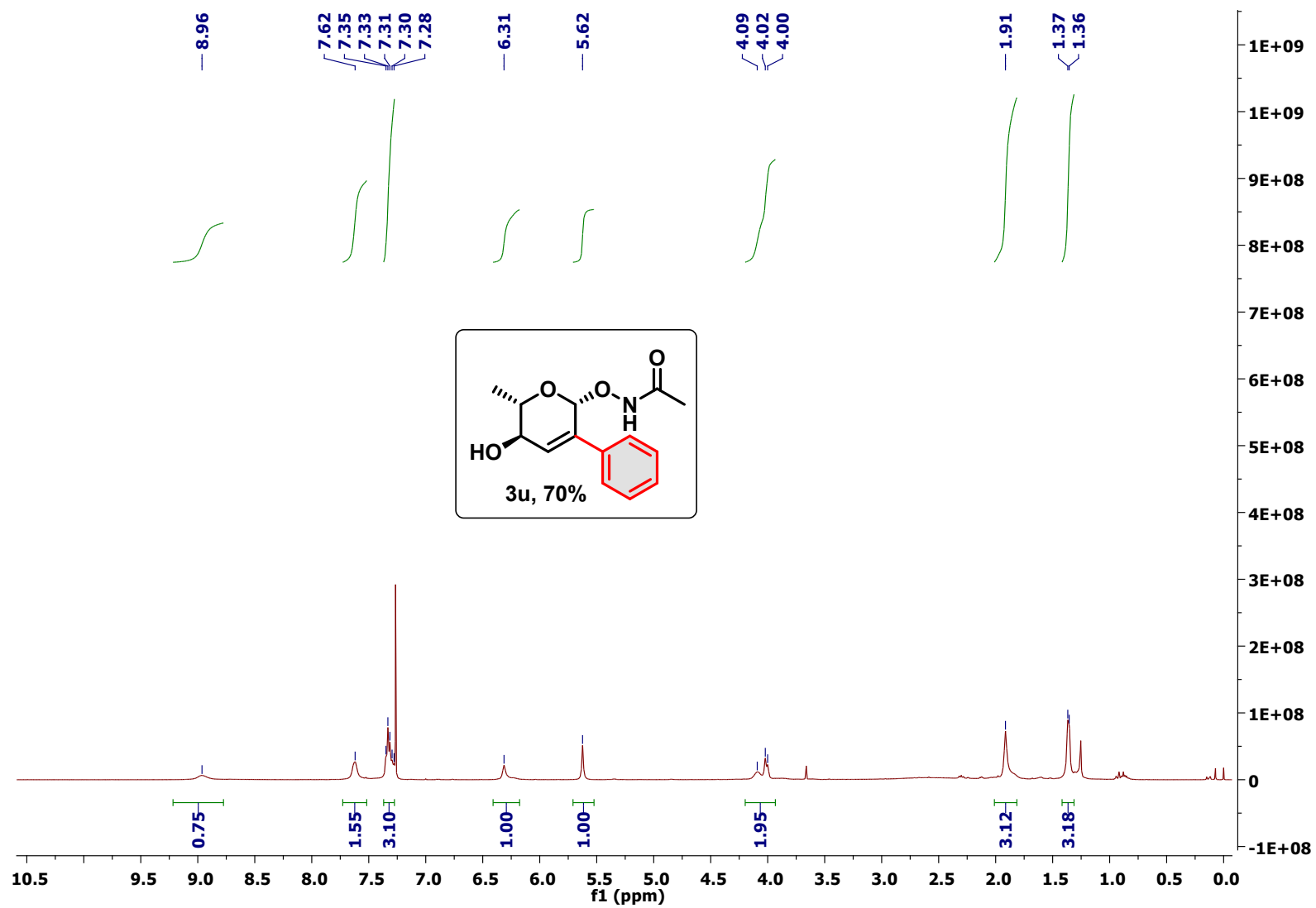
¹H NMR (400 MHz) of **3t** in CDCl₃



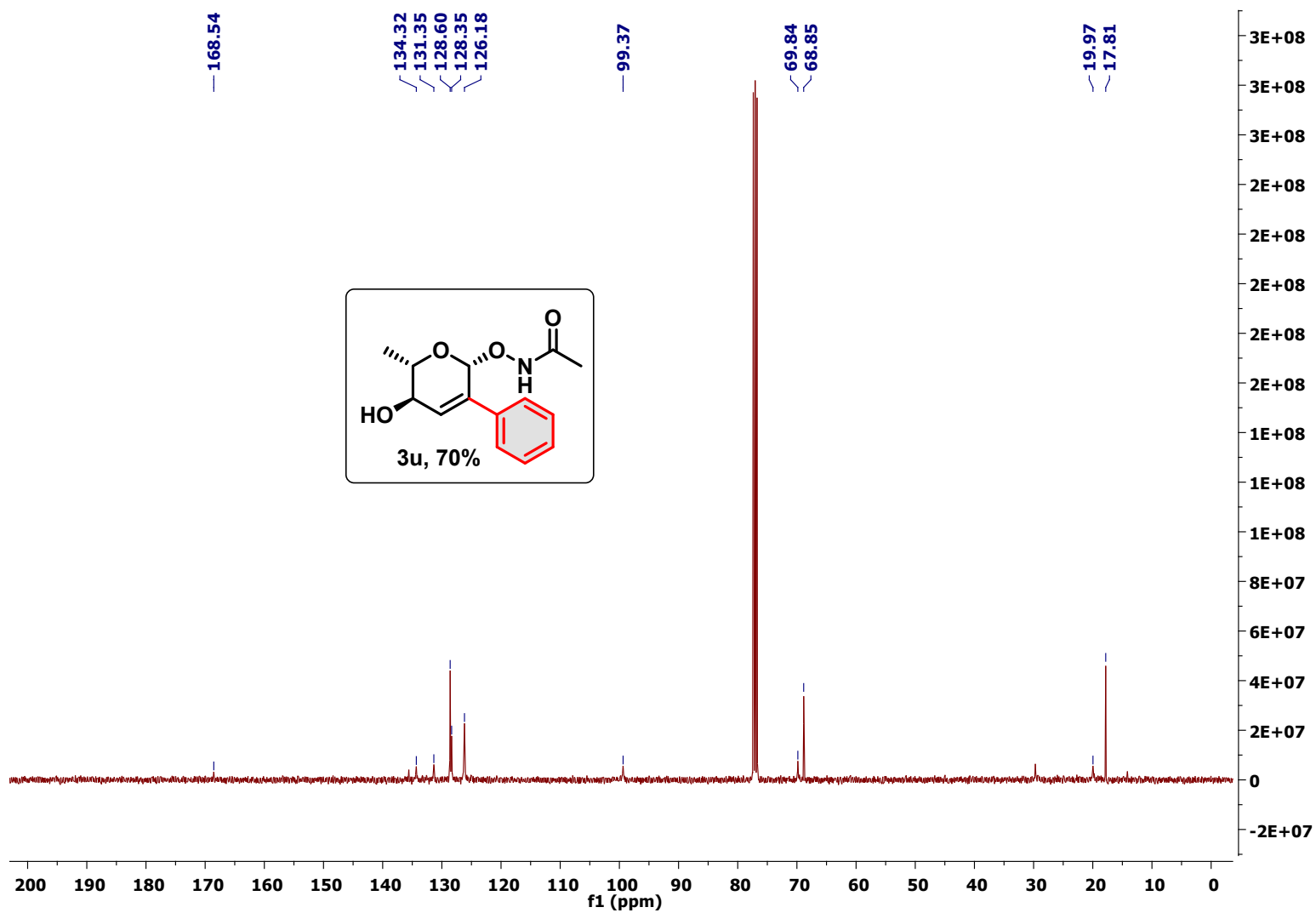
^{13}C NMR (101 MHz) of **3t** in CDCl_3



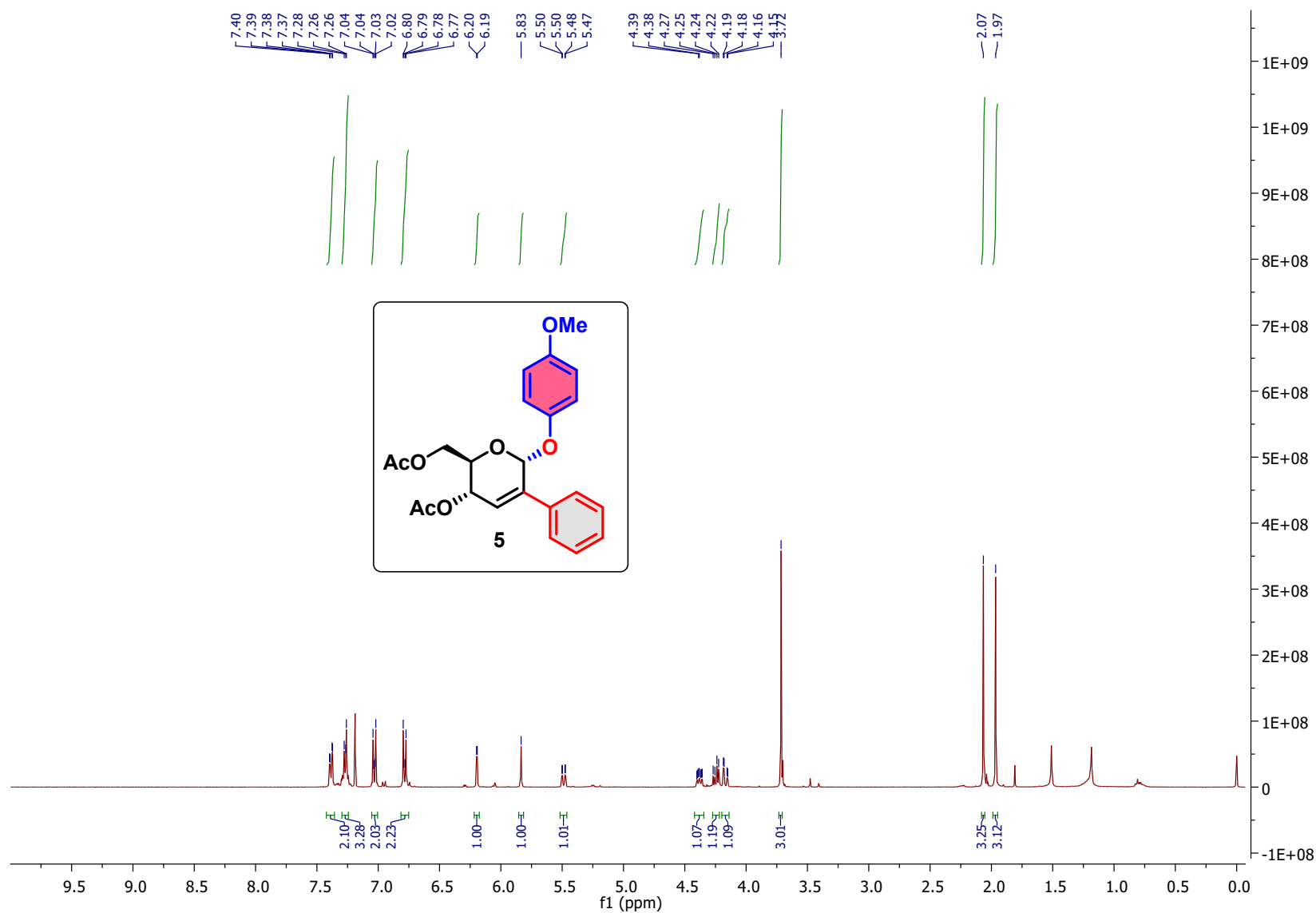
^1H NMR (400 MHz) of **3u** in CDCl_3



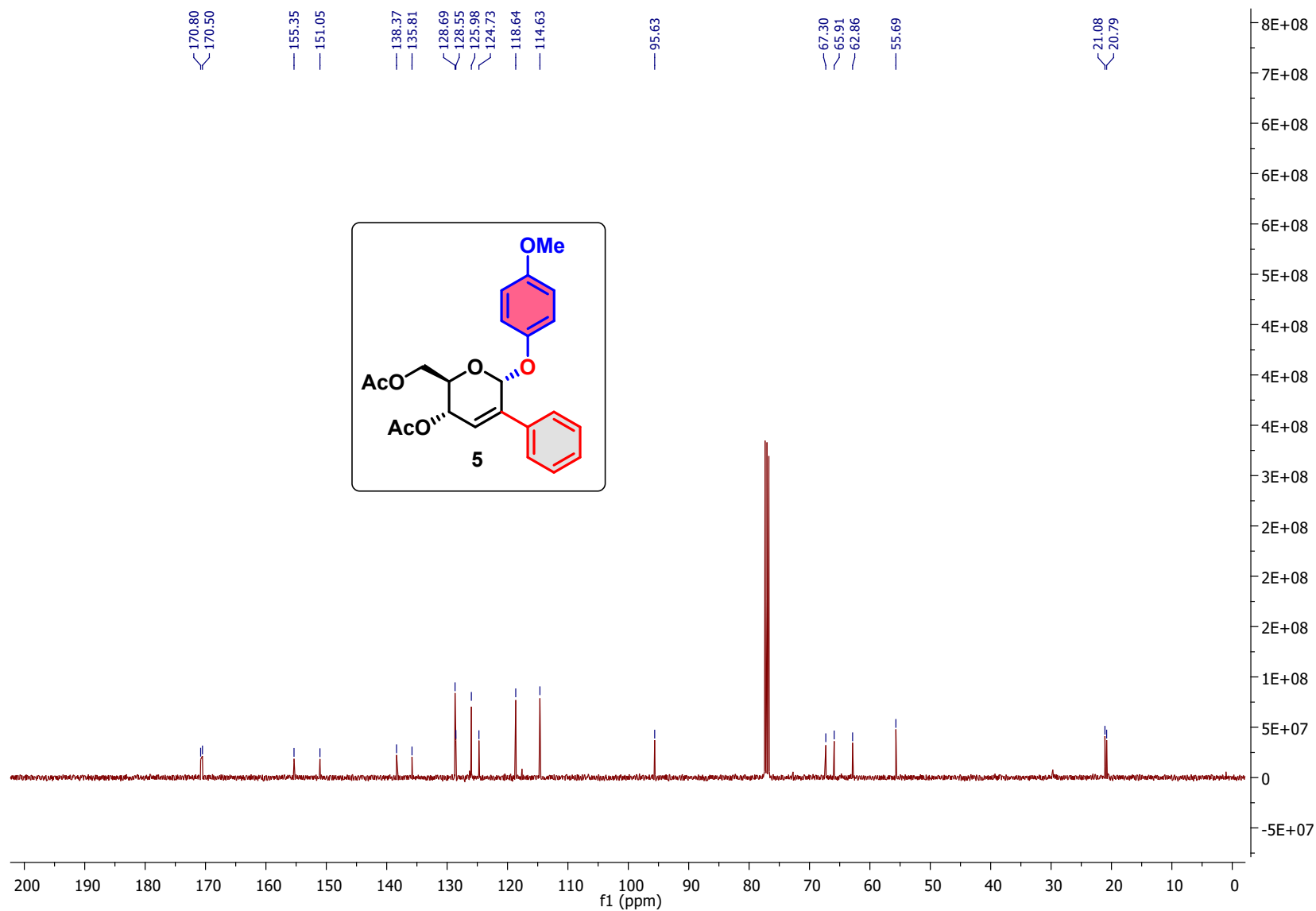
^{13}C NMR (101 MHz) of **3u** in CDCl_3



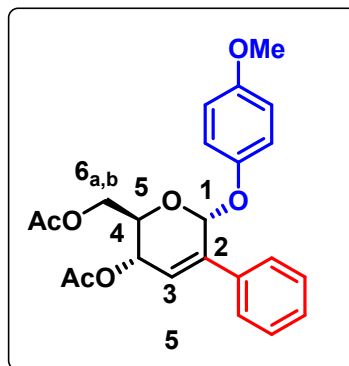
^1H NMR (400 MHz) of **5** in CDCl_3



^{13}C NMR (101 MHz) of **5** in CDCl_3



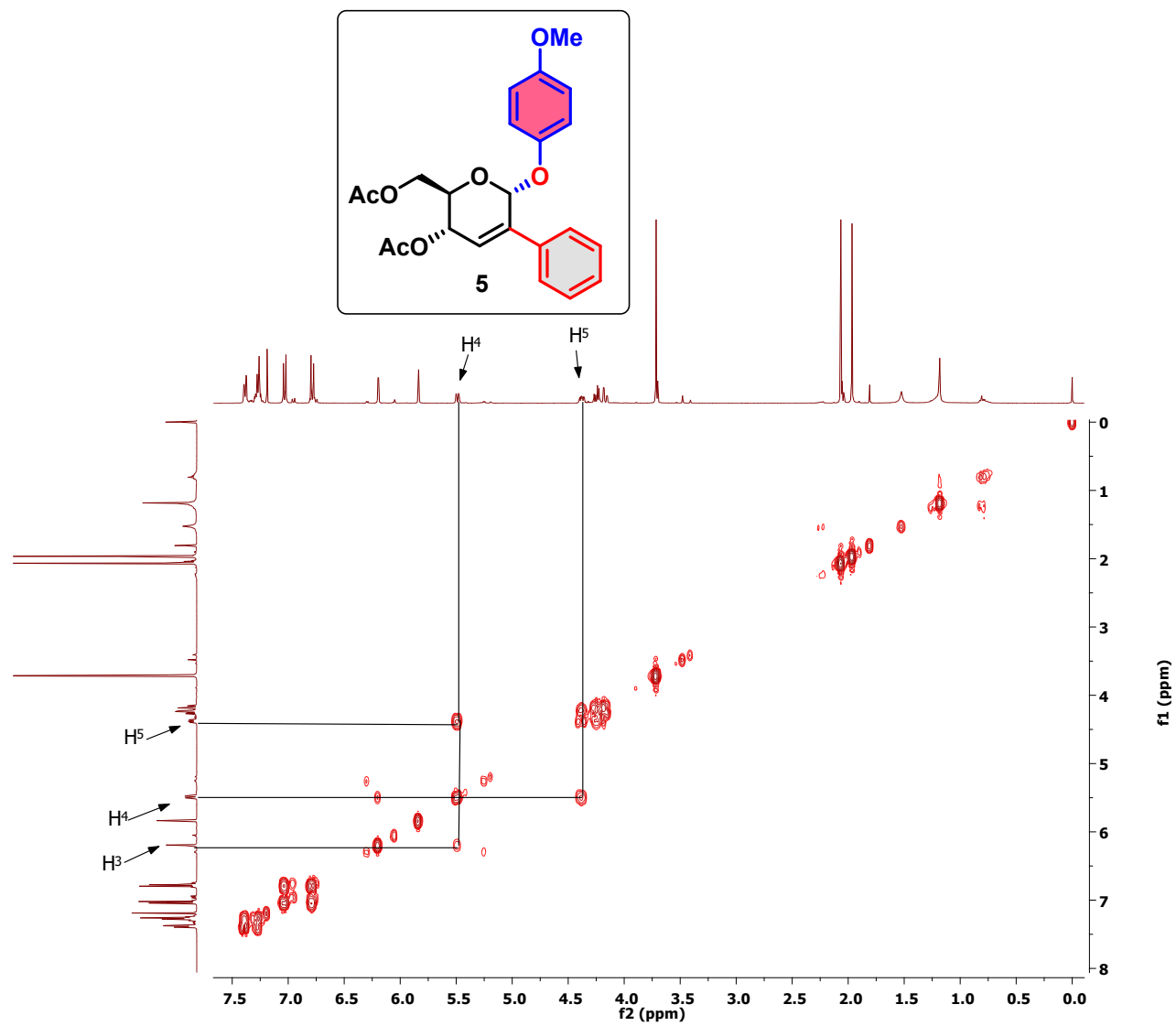
HSQC of compound 5



HSQC Table of compound 5

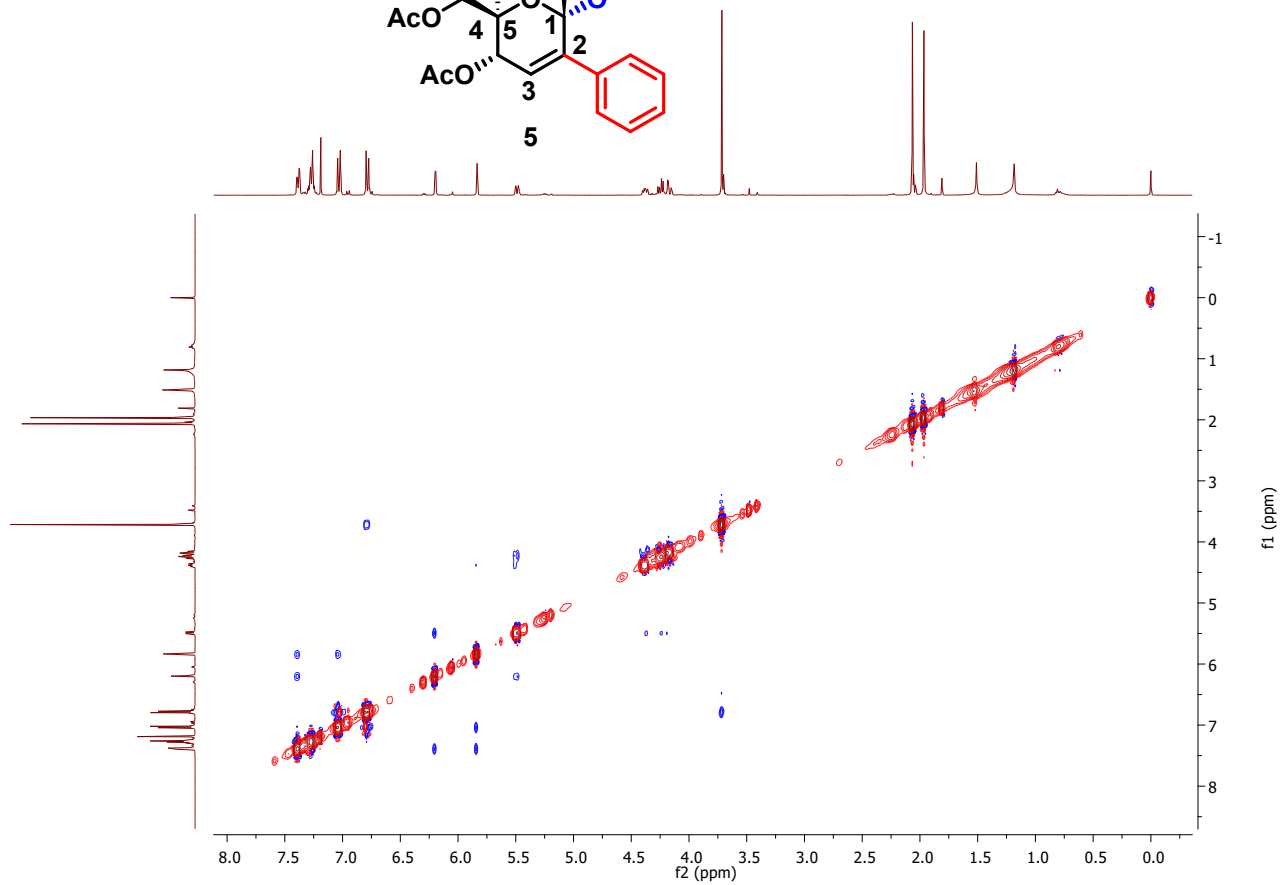
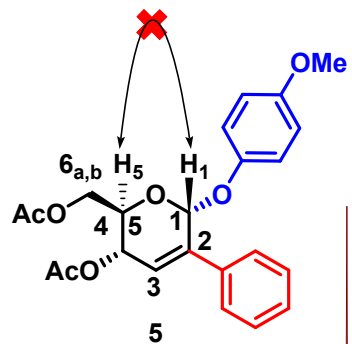
	¹ H	¹³ C
1.	H₁ = 5.83 (s, 1H)	95.6
2.	--	
3.	H₃ = 6.20 (d, <i>J</i> = 2.3 Hz, 1H)	124.7
4.	H₄ = 5.49 (dd, <i>J</i> = 9.8, 1.5 Hz, 1H)	65.9
5.	H₅ =4.38 (m, 1H)	67.3
6.	H_{6a} =4.25 (dd, <i>J</i> = 12.1, 5.7 Hz, 1H)	62.9
7.	H_{6b} = 4.17 (dd, <i>J</i> = 12.1, 2.5 Hz, 1H)	62.9

COSY of compound 5

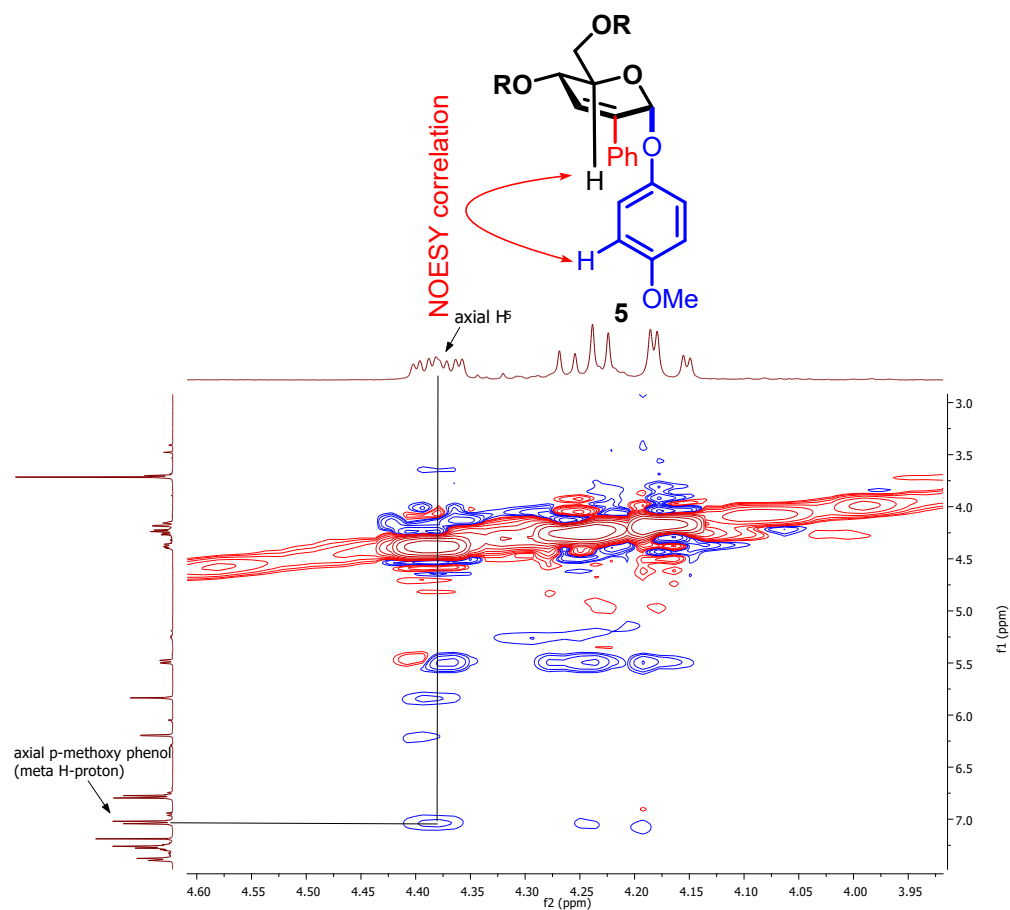


NOESY of compound 5

No NOESY correlation

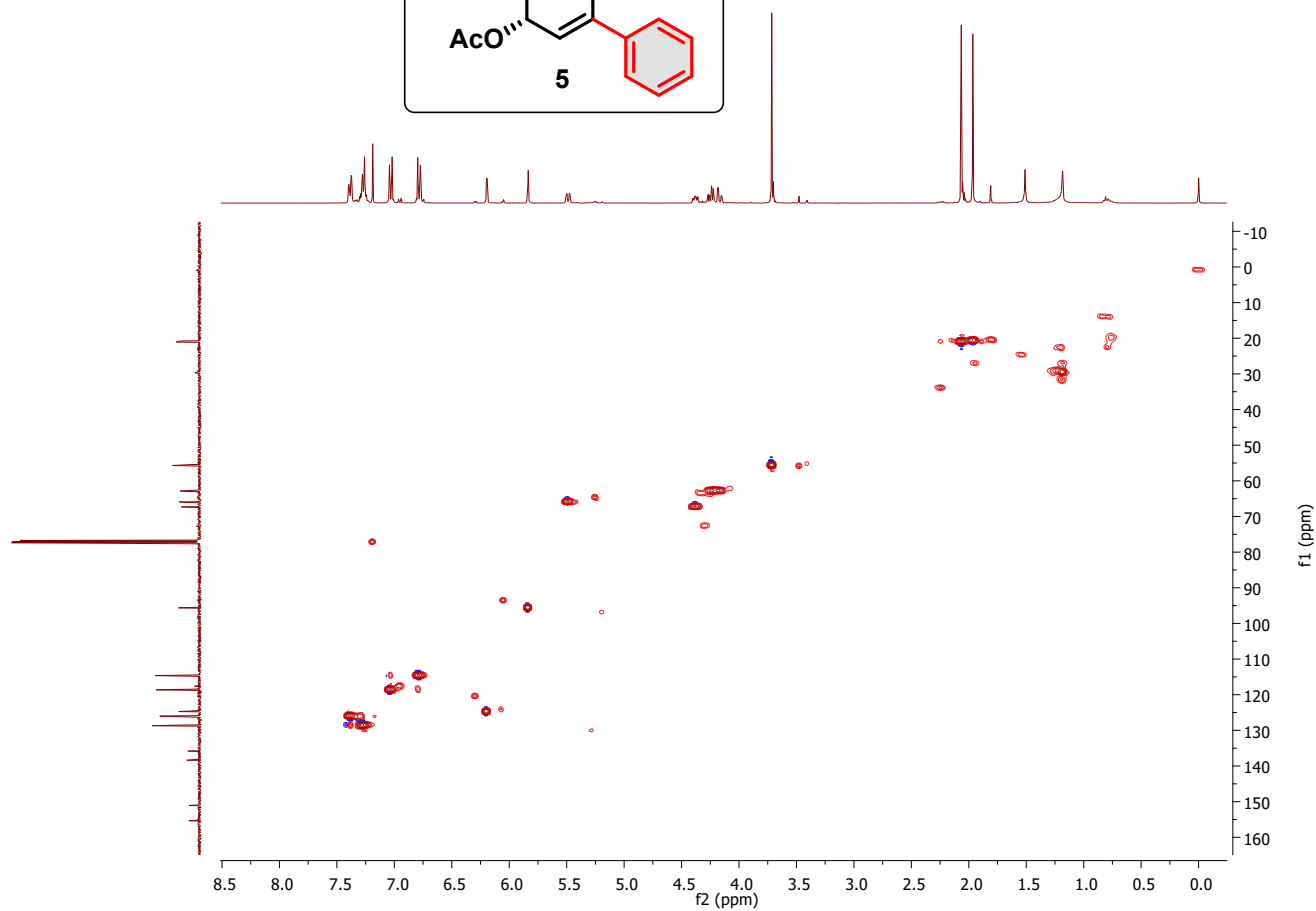
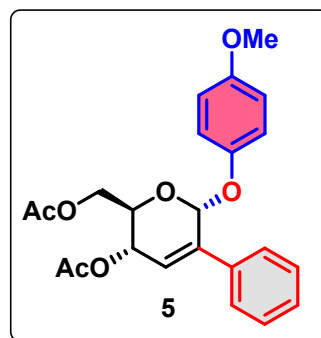


NOESY of compound 5

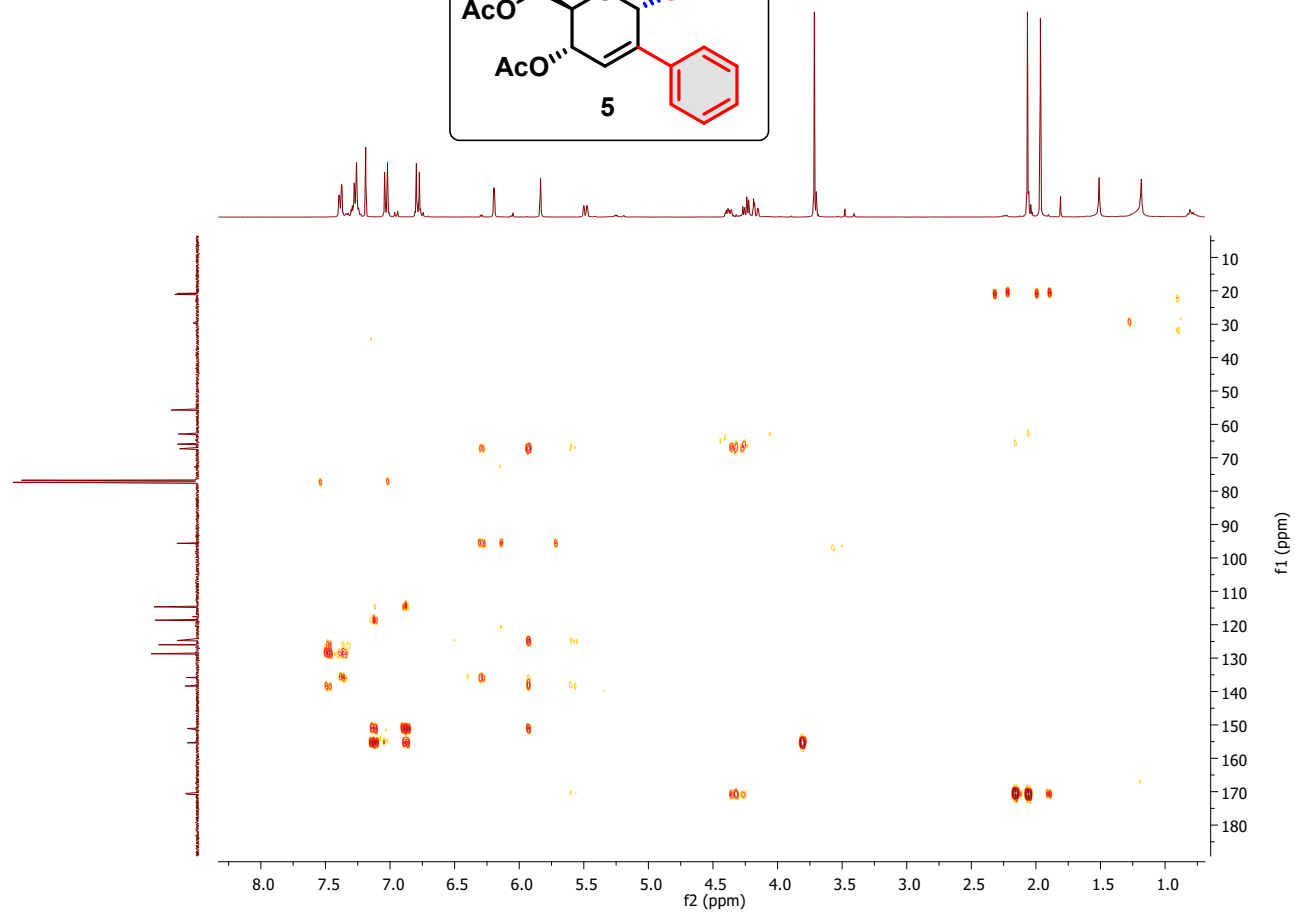
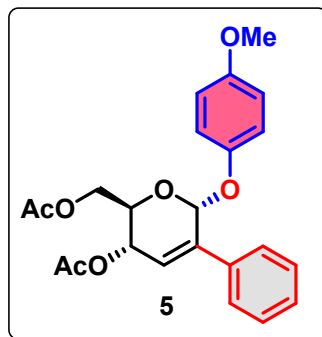


In the NOESY experiment, we observed a correlation between the axial H₅ and the axially oriented meta-H proton of the p-methoxy phenol attached to the anomeric carbon but no correlation was detected between H₁ and H₅, confirming the formation of the α -glycosylated product **5**.

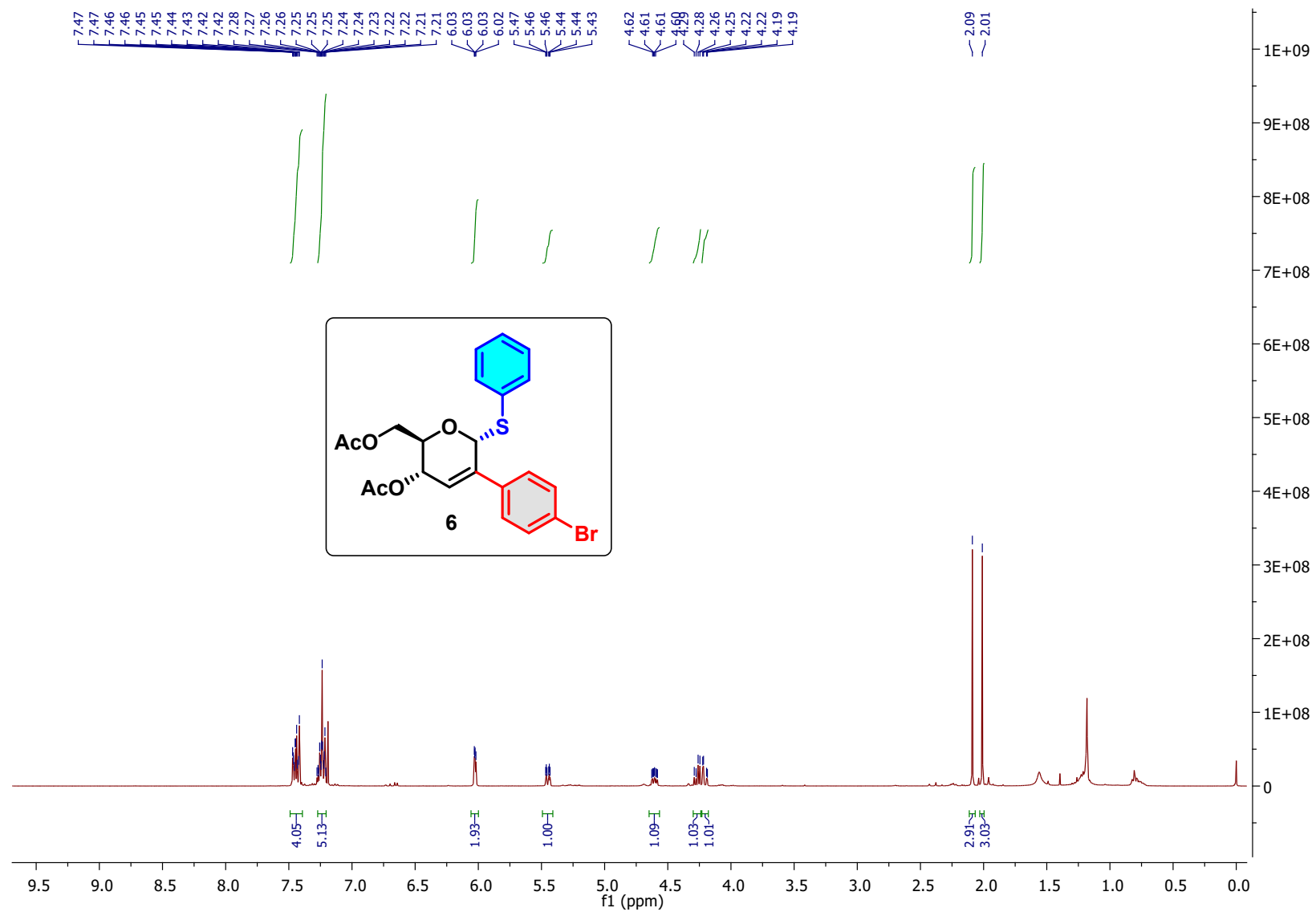
HSQC of compound 5



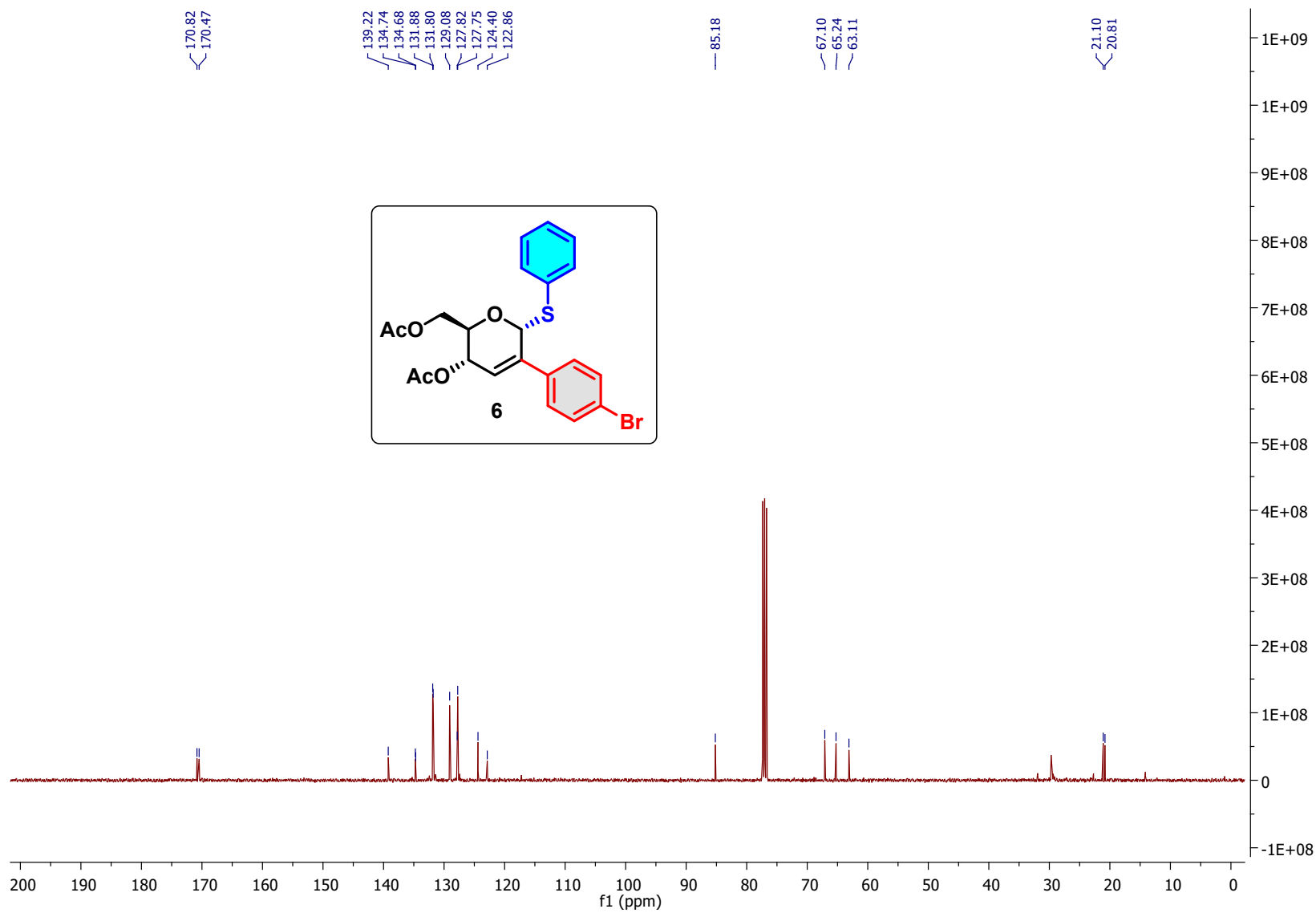
HMBC of compound 5



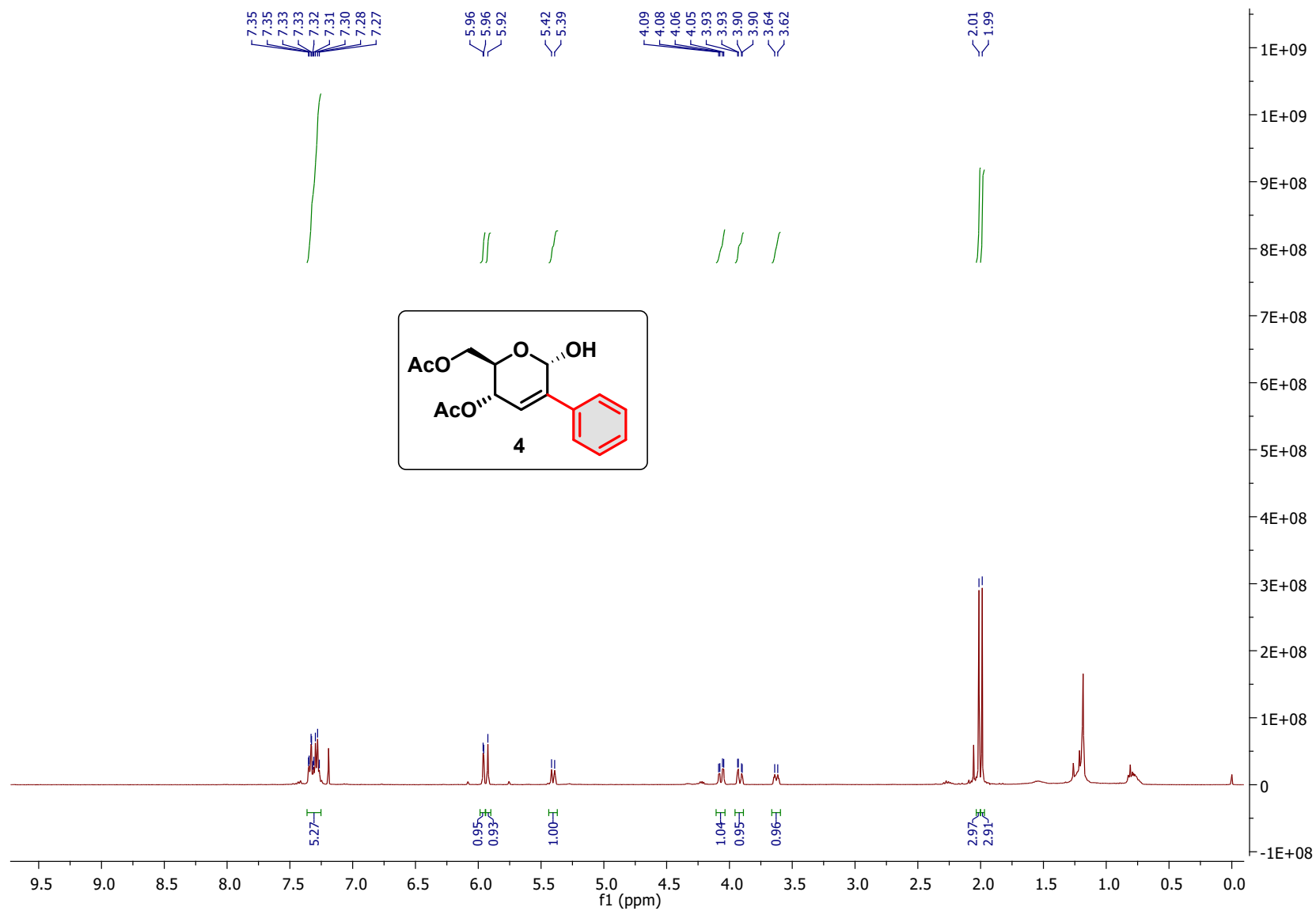
^1H NMR (400 MHz) of **6** in CDCl_3



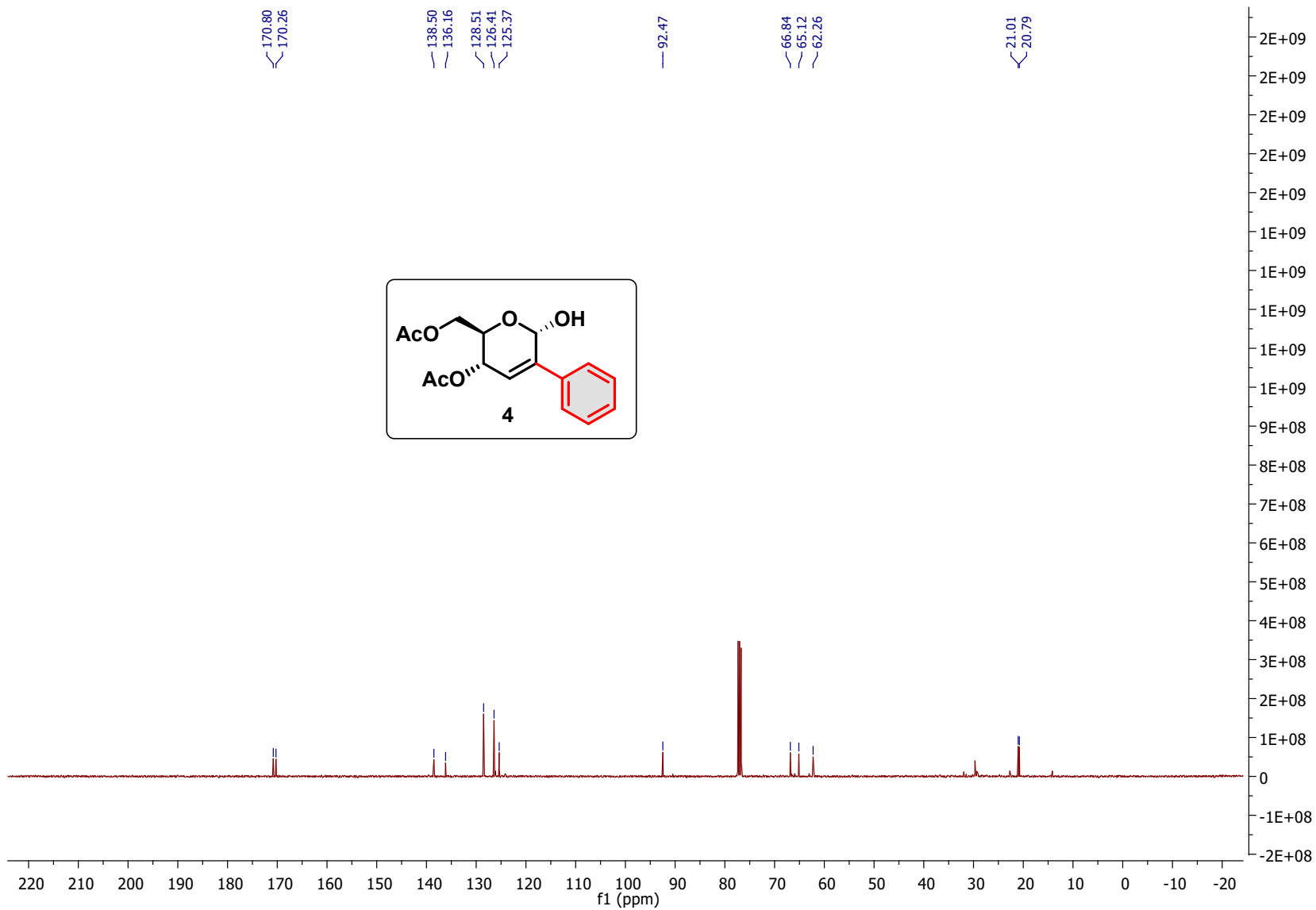
^{13}C NMR (101 MHz) of **6** in CDCl_3



^1H NMR (400 MHz) of **4** in CDCl_3

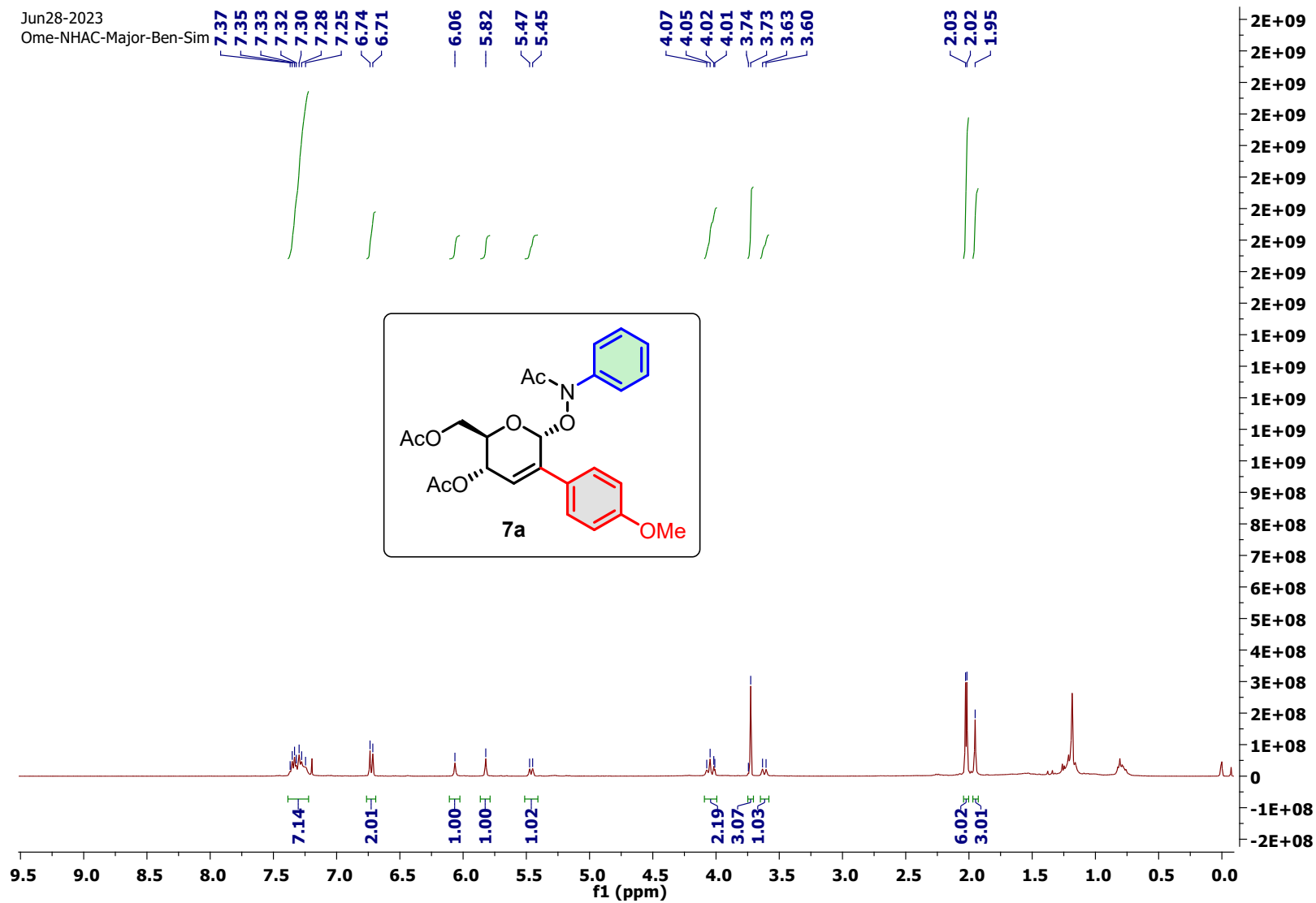


¹³C NMR (101 MHz) of **4** in CDCl₃

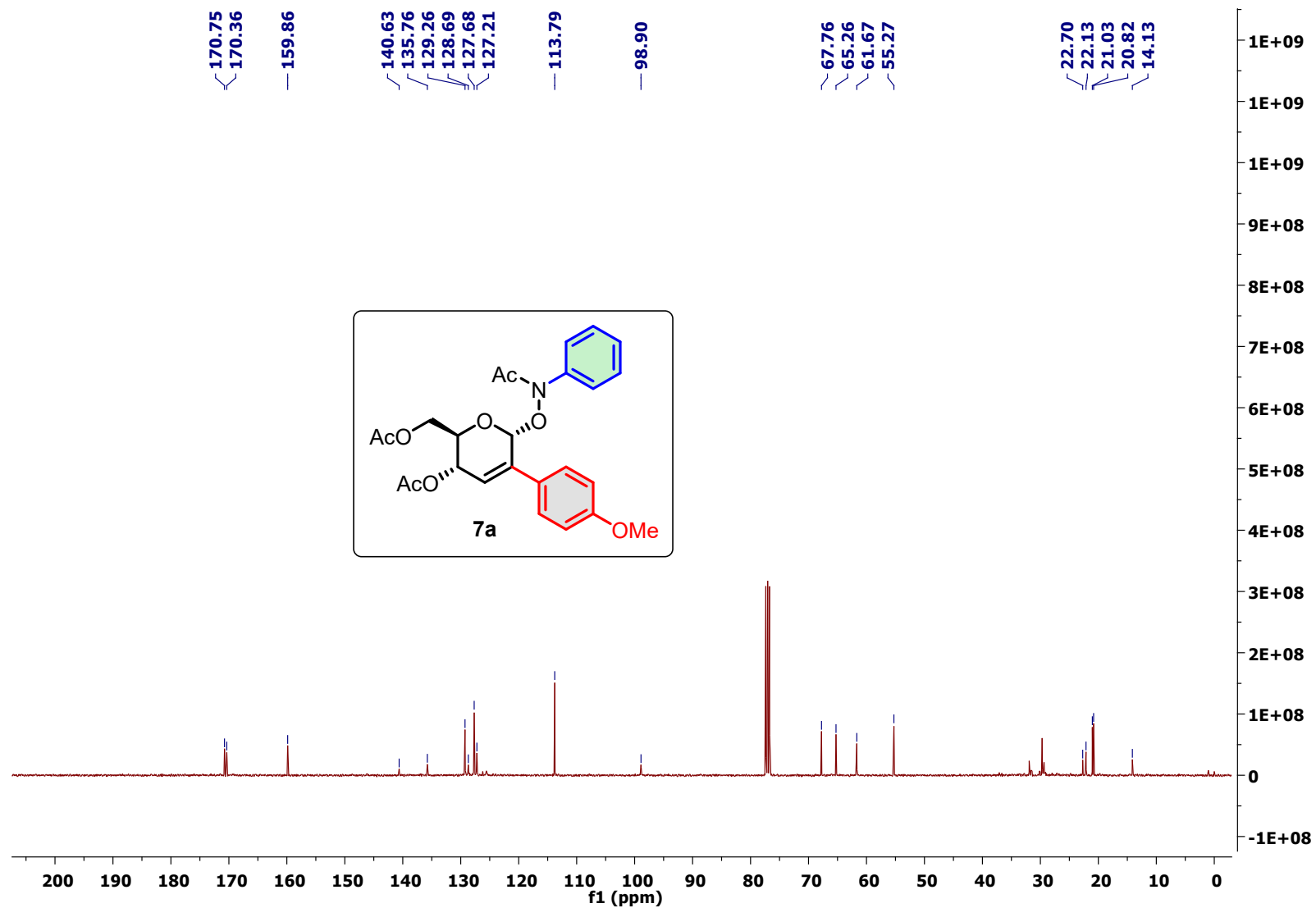


^1H NMR (400 MHz) of **7a** in CDCl_3

Jun28-2023
Ome-NHAC-Major-Ben-Sim

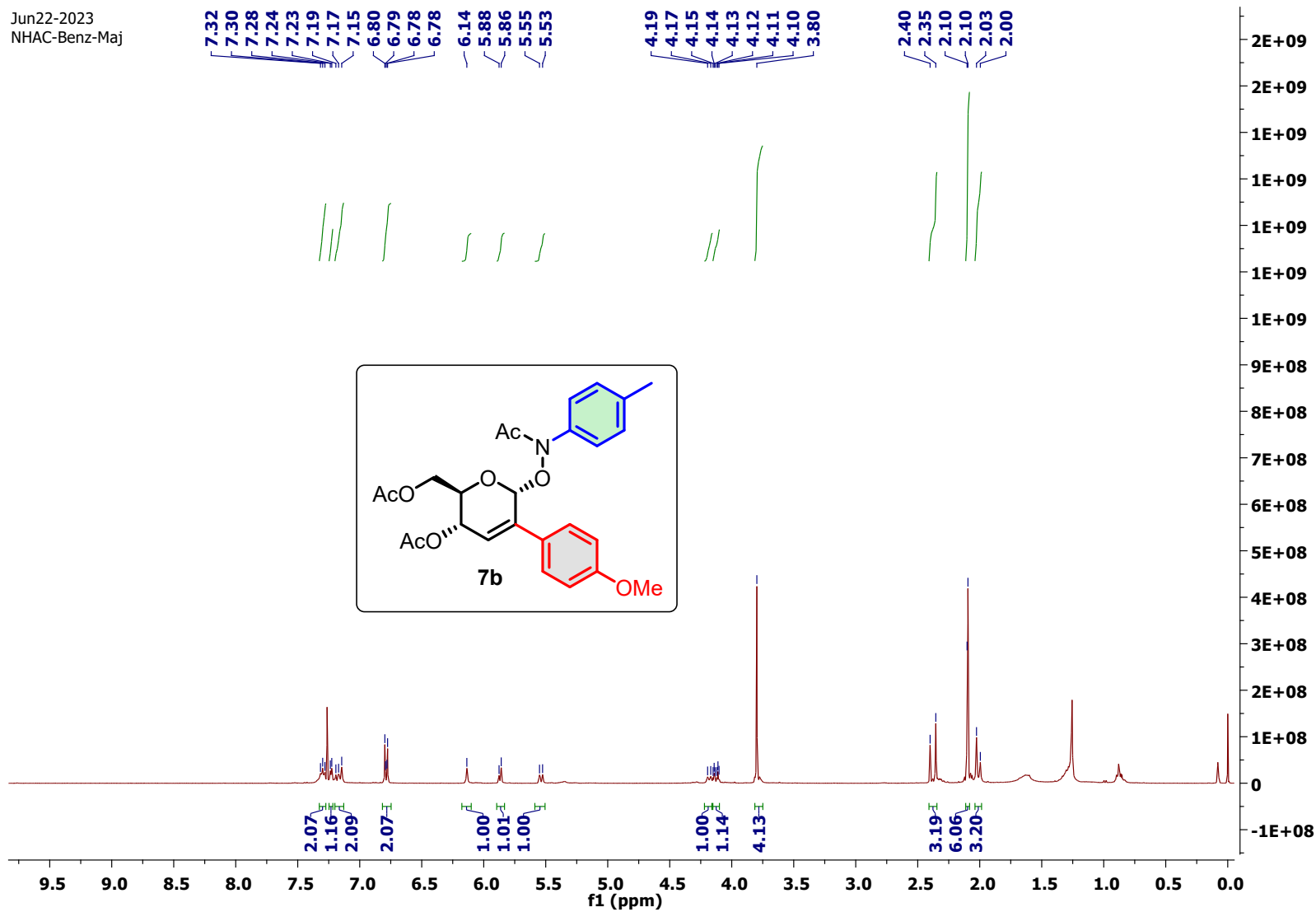


^{13}C NMR (101 MHz) of **7a** in CDCl_3



^1H NMR (400 MHz) of **7b** in CDCl_3

Jun22-2023
NHAC-Benz-Maj



^{13}C NMR (101 MHz) of **7b** in CDCl_3

