

An efficient and eco-friendly synthesis of 2-pyridones and functionalized azaxanthone frameworks *via* indium triflate catalyzed domino reaction

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Experimental General:

All chemicals were purchased from Sigma Aldrich. All melting points are uncorrected. ^1H and ^{13}C NMR spectra were recorded in CDCl_3 and $\text{DMSO}-d_6$ using TMS as an internal standard on a Bruker avance spectrometer at 400 Mhz amd 100 MHz and JEOL spectrometer at 500 and 125 Mhz, respectively. Mass spectra were recorded using a JEOL GCMate-II–HR mass spectrometer. Analytical TLC was performed on precoated aluminium sheets of siliga gel G/UV-254 of 0.2 mm thickness (Merck, Germany).

Starting materials

Starting materials **2** and **4** derivatives (Table 1 in manuscript) has been prepared according to the literature procedure.^{13c,d}

General procedure for the preparation of 2-pyridone and chrominopyridine derivatives: A mixture of 3-formylchromone **1** (1 mmol), (Z)-N-methyl-1-(methyl-thio)-2-nitroethenamine/N,N'-dimethyl-2-nitro-ethene-1,1-diamine **2/4** (1 mmol), and indium triflate (2 mol %) in ethanol (3 mL) were charged in a 25 mL round bottomed flask and the mixture was heated at reflux. The resulting solution was stirred for 1-3 hrs. The consumption of the starting material was monitored by TLC. After completion of the reaction, the products (**3a-s**) was filtered and washed with ethanol, dried under vacuum and the products **5a-p** was purified by column chromatography to obtain pure products **3a-s** and **5a-p** in good yields (65-95 %). The identities of products **3a-s** and **5a-p** were confirmed by NMR and EI-HRMS, giving good agreement with the assigned structures.

Synthetic transformation of products 3a:

A mixture of γ -nitro-2-pyridone **3a** (1 mmol) and stannous chloride dihydrate (7 equiv) in ethanol (3 mL) were charged in a 25 mL round bottomed flask and the mixture was heated at reflux. The resulting solution was stirred for 2 hrs. The consumption of the starting material was monitored by TLC. After completion of the reaction, the products was purified by column chromatography to obtain pure products **14** in good yield (82 %). The identities of products **14** was confirmed by NMR and EI-HRMS, giving good agreement with the assigned structures.

Isolated as yellow solid, 82%, mp: 188-190 °C, ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ_{H} 10.20 (1H, s), 7.74 (1 H, d, $J = 1.3$), 7.57 (1H, d, $J = 1.8$), 7.39 (2H, dd, $J = 16.8, 8.0$), 7.03 – 6.93 (2H, m), 3.72 (1H, s), 3.59 (3 H, s), 3.57 (1H, s) ppm. ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ_{C} 192.73, 158.55, 156.44, 137.27, 132.98, 130.50, 130.26, 125.21, 119.55, 117.61, 117.23, 110.72, 38.43 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{13}\text{H}_{12}\text{N}_2\text{O}_3$: 244.0848, Found: 244.0847

Catalyst recovery and reuse during the preparation of 2-pyridone 3a

A mixture of 3-formylchromone **1** (1 equiv), (Z)-N-methyl-1-(methyl-thio)-2-nitroethenamine **2** (1 equiv), and indium triflate (2 mol %) in ethanol were charged in a 25 mL round bottomed flask and the mixture was heated at reflux. The resulting solution was stirred for 1 hr. The consumption of the starting material was monitored by TLC. After completion of the reaction, the products **3a** was filtered and washed with ethanol, dried under vacuum to obtain pure product **3a** as 88 % yield. The filtrate was evaporated to dryness by repeated codistillation with toluene and finally dried under vacuum at 95–100°C. The recovered catalyst is effective in subsequent experiments. It should be noted that the yields in second and even fifth run are comparable to that of the first run (Table 1).

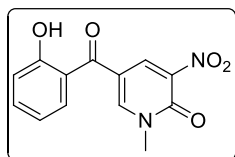
Table 1 Catalyst recovery and reuse during the preparation of 2-pyridone **3a**

Run	In(OTf) ₃ Product (%)	Yield ^{a,b,c} (%)
1 st	3a	88
2 nd	3a	86
3 rd	3a	84
4 th	3a	84
5 th	3a	80

^aThe reaction was performed with 3-formylchromone **1** (1 equiv), NMSM **2** (1 equiv), In(OTf)₃ (2 mol %) and ethanol, at reflux,

^bReaction progress was followed by TLC analysis. ^cYield of isolated products.

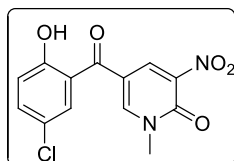
3a:5-(2-hydroxybenzoyl)-1-methyl-3-nitropyridin-2(1H)-one



Isolated as yellow solid, 89%, mp: 219-221°C, ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.34 (s, 1H), 8.74 (s, 1H), 8.52 (d, *J* = 2.3 Hz, 1H), 7.51–7.22 (m, 2H), 7.07–6.67 (m, 2H), 3.62 (s, 3H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 190.19, 155.83, 154.05, 150.44, 137.66, 136.15, 133.39, 130.21, 124.11, 119.45, 116.77, 114.28,

38.62 ppm. EI-HRMS: Anal. Calcd for C₁₃ H₁₀ N₂O₅: 274.0590, Found: 274.0587

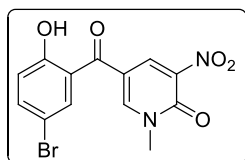
3b: 5-(5-chloro-2-hydroxybenzoyl)-1-methyl-3-nitropyridin-2(1H)-one



Isolated as yellow solid, 88 %, mp: 202-204 °C, ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.61 (s, 1H), 8.78 (d, *J* = 2.5 Hz, 1H), 8.56 (d, *J* = 2.5 Hz, 1H), 7.47 (dd, *J* = 8.8, 2.7 Hz, 1H), 7.38 (d, *J* = 2.7 Hz, 1H), 7.01 (d, *J* = 8.8 Hz, 1H), 3.65 (s, 3H) ppm. ¹³C NMR (100 MHz, DMSO-*d*₆) δ 193.94, 159.51, 159.32, 156.00, 142.56, 141.67, 137.80, 134.30, 131.31, 128.28, 123.82, 119.25, 43.85 ppm. EI-HRMS:

Anal. Calcd for C₁₃H₉ClN₂O₅: 308.0200, Found: 308.0199.

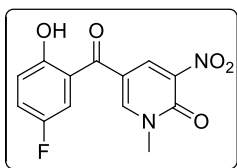
3c:5-(5-bromo-2-hydroxybenzoyl)-1-methyl-3-nitropyridin-2(1H)-one



Isolated as yellow solid, 85 %, mp: 210-212 °C, ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.65 (s, 1H), 8.78 (d, *J* = 2.0 Hz, 1H), 8.57 (d, *J* = 2.1 Hz, 1H), 7.69–7.37 (m,

2H), 6.97 (d, $J = 8.7$ Hz, 1H), 3.66 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ 193.85, 159.92, 159.31, 156.06, 142.60, 141.64, 140.63, 137.14, 131.86, 124.26, 119.25, 115.71, 43.86. EI-HRMS: Anal. Calcd for $\text{C}_{13}\text{H}_9\text{BrN}_2\text{O}_5$: 351.9695, Found: 351.9690.

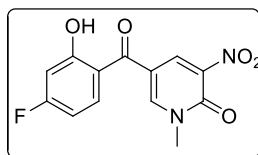
3d:5-(5-fluoro-2-hydroxybenzoyl)-1-methyl-3-nitropyridin-2(1H)-one



Isolated as yellow solid, 83 %, mp: 210-212 °C, ^1H NMR (400 MHz DMSO- d_6) δ 10.32 (s, 1H), 8.77 (d, $J = 2.0$ Hz, 1H), 8.57 (d, $J = 2.4$ Hz, 1H), 7.30 (td, $J = 8.6$, 3.2 Hz, 1H), 7.21 (dd, $J = 8.6$, 3.2 Hz, 1H), 7.00 (dd, $J = 9.0$, 4.4 Hz, 1H), 3.66 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 194.09, 161.62, 159.32-159.27 (d, $J = 4.10$ Hz), 157.06, 156.08, 142.66, 141.65, 130.24-130.17 (d, $J = 6.70$ Hz), 125.16-124.93 (d, $J = 23.75$ Hz), 123.40-123.32 (d, $J = 8.70$ Hz), 121.08-120.84 (d, $J = 24.69$ Hz), 119.21, 43.86.

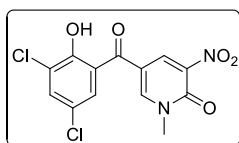
HRMS: Anal. Calcd for $\text{C}_{13}\text{H}_9\text{FN}_2\text{O}_5$: 292.0495, Found: 292.0491.

3e:5-(4-fluoro-2-hydroxybenzoyl)-1-methyl-3-nitropyridin-2(1H)-one



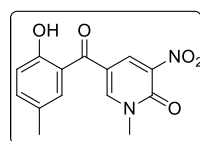
Isolated as yellow solid, 86 %, mp: 260-261 °C, ^1H NMR (400 MHz, DMSO- d_6) δ 8.77 (d, $J = 2.3$ Hz, 1H), 8.58 (d, $J = 2.4$ Hz, 1H), 7.50 (dd, $J = 8.2$, 7.2 Hz, 1H), 6.80 (ddd, $J = 12.8$, 9.5, 2.1 Hz, 2H), 3.66 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 194.35, 171.37, 168.89, 163.37-163.25 (d, $J = 13.62$ Hz), 159.31, 155.74, 142.75-142.69 (d, $J = 5.68$ Hz), 141.56, 137.94-137.82 (d, $J = 11.35$ Hz), 126.34-126.32 (d, $J = 2.25$ Hz), 119.58, 112.15-108.71 (dd, $J = 322.91$, 23.01 Hz), 43.81 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{13}\text{H}_9\text{FN}_2\text{O}_5$: 292.0495, Found: 292.0493.

3f:5-(3,5-dichloro-2-hydroxybenzoyl)-1-methyl-3-nitropyridin-2(1H)-one



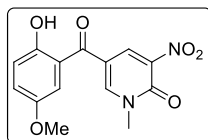
Isolated as yellow solid, 84 %, mp: 209-211 °C, ^1H NMR (400 MHz, DMSO- d_6) δ 8.72 (d, $J = 1.8$ Hz, 1H), 8.56 (d, $J = 2.0$ Hz, 1H), 7.72 (d, $J = 2.2$ Hz, 1H), 7.36 (d, $J = 2.2$ Hz, 1H), 3.60 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 188.11, 154.03, 150.92, 150.50, 136.81, 136.60, 131.89, 128.11, 127.84, 123.54, 123.36, 113.82, 38.62 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{13}\text{H}_8\text{Cl}_2\text{N}_2\text{O}_5$: 341.9810, Found: 341.9810.

3g:5-(2-hydroxy-5-methylbenzoyl)-1-methyl-3-nitropyridin-2(1H)-one



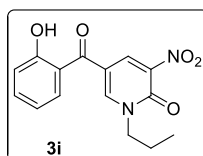
Isolated as yellow solid, 80 %, mp: 216-218 °C, ^1H NMR (400 MHz, DMSO- d_6) δ 10.18 (s, 1H), 8.77 (d, $J = 2.4$ Hz, 1H), 8.56 (d, $J = 2.5$ Hz, 1H), 7.27 (dd, $J = 8.3$, 1.9 Hz, 1H), 7.20 (d, $J = 1.7$ Hz, 1H), 6.91 (d, $J = 8.3$ Hz, 1H), 3.68 (s, 3H), 2.26 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 195.55, 159.29, 158.83, 155.73, 143.10, 141.37, 139.31, 135.36, 133.50, 129.06, 121.92, 119.63, 43.86, 25.05 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_5$: 288.0746, Found: 288.0743.

3h:5-(2-hydroxy-5-methoxybenzoyl)-1-methyl-3-nitropyridin-2(1H)-one



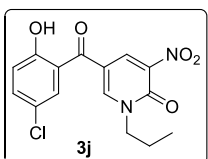
Isolated as yellow solid, 78 %, mp: 215-217 °C, ^1H NMR (400 MHz, DMSO- d_6) δ 9.86 (s, 1H), 8.71 (s, 1H), 8.52 (d, $J = 2.0$ Hz, 1H), 7.02 (dd, $J = 8.9, 2.9$ Hz, 1H), 6.91 – 6.77 (m, 2H), 3.68 (s, 3H), 3.62 (s, 3H). ^{13}C NMR (100 MHz, DMSO- d_6) δ 195.05, 159.34, 157.40, 155.77, 154.79, 142.97, 141.35, 129.46, 125.20, 123.13, 119.46, 118.86, 60.74, 43.80. EI-HRMS: Anal. Calcd for $\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_6$: 304.0695, Found: 304.0689.

3i: 5-(2-hydroxybenzoyl)-3-nitro-1-propylpyridin-2(1H)-one



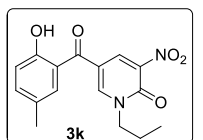
Isolated as yellow solid, 75 %, mp: 170-172°C, ^1H NMR (400 MHz, DMSO- d_6) δ_{H} 10.43 (1H, s), 8.70 (1H, d, $J = 2.5$), 8.55 (1H, d, $J = 2.5$), 7.51–7.36 (2H, m), 7.05 – 6.89 (2H, m), 4.08 (2H, t, $J = 7.2$), 1.70 (2H, dd, $J = 14.6, 7.3$), 0.88 (3H, t, $J = 7.4$) ppm. ^{13}C NMR (100 MHz, DMSO- d_6): δ_{C} 190.68, 156.30, 154.09, 150.36, 137.91, 137.26, 133.99, 130.84, 124.58, 120.00, 117.25, 115.02, 52.49, 22.24, 11.06 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_5$: 302.0903, Found: 302.0900.

3j: 5-(5-chloro-2-hydroxybenzoyl)-3-nitro-1-propylpyridin-2(1H)-one



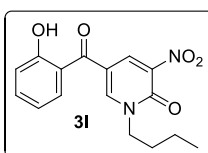
Isolated as yellow solid, 82 %, mp: 192-194°C, ^1H NMR (400 MHz, DMSO- d_6): δ_{H} 10.55 (1H, d, $J = 63.9$), 8.72 (1H, d, $J = 2.5$), 8.56 (1H, d, $J = 2.5$), 7.47 (1H, dd, $J = 8.8, 2.7$), 7.38 (1H, d, $J = 2.7$), 7.01 (1H, d, $J = 8.8$), 4.08 (2H, t, $J = 7.2$), 1.66 (2H, dd, $J = 14.5, 7.3$), 0.88 (3H, t, $J = 7.3$). ^{13}C NMR (100 MHz, DMSO- d_6): δ_{C} 189.13, 154.83, 154.09, 150.69, 137.50, 137.47, 133.15, 129.73, 126.47, 123.55, 119.10, 114.66, 52.44, 22.22, 11.04. EI-HRMS: Anal. Calcd for $\text{C}_{15}\text{H}_{13}\text{ClN}_2\text{O}_5$: 336.0513, Found: 336.0512.

3k: 5-(2-hydroxy-5-methylbenzoyl)-3-nitro-1-propylpyridin-2(1H)-one



Isolated as yellow solid, 79 %, mp: 177-179°C, ^1H NMR (400 MHz, DMSO- d_6): δ_{H} 10.16 (1H, s), 8.69 (1H, d, $J = 2.5$), 8.56 (1H, t, $J = 6.3$), 7.25 (1H, dt, $J = 17.6, 8.8$), 7.20 (1H, s), 6.90 (1H, d, $J = 8.3$), 4.16–3.98 (2H, m), 2.25 (3H, s), 1.77–1.63 (2H, m), 0.88 (3 H, t, $J = 7.6$) ppm. ^{13}C NMR (100 MHz, DMSO- d_6): δ_{C} 190.74, 154.13, 154.09, 150.38, 137.95, 137.28, 134.66, 130.76, 128.75, 124.27, 117.20, 115.05, 52.46, 22.23, 20.30, 11.07 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_5$: 316.1059, Found: 316.1054.

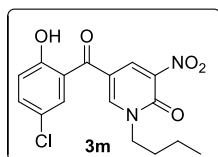
3l: 1-butyl-5-(2-hydroxybenzoyl)-3-nitropyridin-2(1H)-one



Isolated as yellow solid, 83 %, mp: 155-157°C, ^1H NMR (400 MHz, DMSO- d_6): δ_{H} 10.42 (1H, s), 8.70 (1H, d, $J = 2.5$), 8.55 (1H, d, $J = 2.5$), 7.50 – 7.35 (2H, m), 6.99 (2H, dd, $J = 16.0, 7.9$), 4.12 (1H, t, $J = 7.3$), 3.40 (1H, d, $J = 6.3$), 1.74 – 1.50 (2H, m), 1.37 – 1.22 (2 H, m), 0.88 (3 H, td, $J = 7.3, 3.4$) ppm. ^{13}C NMR (100 MHz, DMSO- d_6): δ_{C} 190.66, 156.37, 154.04, 150.30, 137.88, 133.96, 130.83, 124.54, 119.95, 117.25, 115.05, 112.70, 50.81, 30.96, 19.55, 13.87 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_5$: 316.1059, Found: 316.1052.

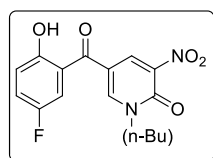
112.70, 50.81, 30.96, 19.55, 13.87 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_5$: 316.1059, Found: 316.1052.

3m:1-butyl-5-(5-chloro-2-hydroxybenzoyl)-3-nitropyridin-2(1H)-one



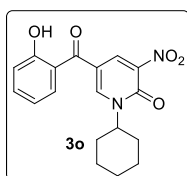
Isolated as yellow solid, 78 %, mp: 160-162°C, ^1H NMR (400 MHz, DMSO- d_6): δ_{H} (400 MHz, DMSO) 10.61 (1H, s), 8.72 (1H, d, $J = 2.5$), 8.56 (1H, d, $J = 2.5$), 7.47 (1H, dd, $J = 8.8, 2.7$), 7.38 (1H, d, $J = 2.7$), 7.02 (1H, d, $J = 8.8$), 4.11 (2H, t, $J = 7.3$), 1.71 – 1.56 (2H, m), 1.29 (2H, m), 0.90 (3 H, t, $J = 7.3$) ppm. ^{13}C NMR (100 MHz, DMSO- d_6): δ_{C} 189.11, 154.86, 154.07, 150.60, 137.51, 137.43, 133.15, 129.73, 126.48, 123.54, 119.11, 114.70, 50.77, 30.94, 19.52, 13.92. ppm. EI-HRMS: Anal. Calcd for $\text{C}_{16}\text{H}_{15}\text{ClN}_2\text{O}_5$: 350.0669, Found: 350.0664.

3n: 1-Butyl-5-(5-fluoro-2-hydroxybenzoyl)-3-nitropyridin-2(1H)-one



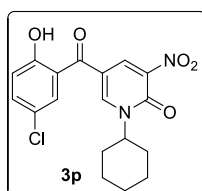
Isolated as yellow solid, 75 %, mp: 144-146 °C, ^1H NMR (400 MHz, CDCl_3): δ_{H} 10.86 (1H, s), 8.68 (1H, d, $J = 2.6$), 8.30 (1H, d, $J = 2.6$), 7.33 (1H, dd, $J = 9.2, 7.7$), 7.21 (1H, dd, $J = 8.4, 3.0$), 7.10 (1H, dd, $J = 9.2, 4.5$), 4.22–4.04 (2 H, m), 1.89–1.77 (2H, m), 1.44 (2H, dd, $J = 15.1, 7.5$), 1.00 (3H, t, $J = 7.4$). ^{13}C NMR (100 MHz, CDCl_3): δ_{C} 192.21, 158.68-158.66 (d, $J = 2.18$ Hz), 156.10, 153.71, 153.65, 147.43, 137.42, 124.73-124.49 (d, $J = 23.84$ Hz), 120.70-120.63 (d, $J = 7.59$ Hz), 117.80-117.74 (d, $J = 7.11$ Hz), 116.13-115.89 (d, $J = 23.69$ Hz), 113.92, 52.04, 31.08, 19.80, 13.50 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{16}\text{H}_{15}\text{FN}_2\text{O}_5$: 334.0965, Found: 334.0961.

3o: cyclohexyl-5-(2-hydroxybenzoyl)-3-nitropyridin-2(1H)-one



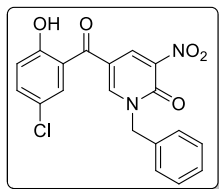
Isolated as yellow solid, 87 %, mp: 206-208°C, ^1H NMR (400 MHz, DMSO- d_6): δ_{H} 10.47 (1H, s), 8.51 (2H, d, $J = 3.7$), 7.56–7.28 (2H, m), 7.12–6.83 (2H, m), 4.72 (1H, dd, $J = 16.2, 7.5$), 1.85 (4H, t, $J = 13.5$), 1.62 (3H, dd, $J = 20.7, 11.8$), 1.41 (2H, q, $J = 12.7$), 1.22 (1H, t, $J = 12.8$) ppm. ^{13}C NMR (100 MHz, DMSO- d_6): δ_{C} 190.51, 156.29, 153.86, 146.42, 137.36, 137.17, 134.15, 131.02, 124.45, 120.09, 117.25, 115.09, 57.43, 31.61, 25.74, 24.90 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_5$: 342.1216, Found: 342.1212.

3p:5-(5-chloro-2-hydroxybenzoyl)-1-cyclohexyl-3-nitropyridin-2(1H)-one



Isolated as yellow solid, 85 %, mp: 210-212°C, ^1H NMR (400 MHz, CDCl_3): δ_{H} 11.05 (1H, s), 8.62 (1H, s), 8.29 (1H, s), 7.52 (H, d, $J = 8.9$), 7.47 (1H, s), 7.09 (1H, d, $J = 8.9$), 5.00 (1H, t, $J = 11.5$), 2.16 – 1.93 (4H, m), 1.82 (1H, d, $J = 13.9$), 1.66 – 1.40 (4H, m), 1.32 – 1.06 (1H, m) ppm. ^{13}C NMR (100 MHz, CDCl_3): δ_{C} 192.38, 161.06, 153.52, 144.21, 137.67, 136.77, 136.41, 130.13, 124.27, 120.85, 118.88, 113.78, 57.05, 32.81, 25.63, 25.02 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{18}\text{H}_{17}\text{ClN}_2\text{O}_5$: 376.0826, Found: 376.0820.

3q: 1-Benzyl-5-(5-chloro-2-hydroxybenzoyl)-3-nitropyridin-2(1H)-one

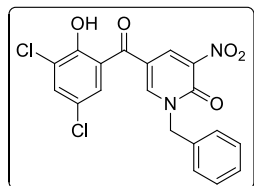


Isolated as yellow solid, 65 %, mp: 186-188 °C, ^1H NMR (400 MHz, CDCl_3): δ_{H} 11.00 (s, 1H), 8.67 (d, $J = 2.6$ Hz, 1H), 8.23 (d, $J = 2.6$ Hz, 1H), 7.46 – 7.40 (m, 5H), 7.32 (d, $J = 2.6$ Hz, 1H), 7.26 (s, 1H), 7.05 (d, $J = 8.9$ Hz, 1H), 5.29 (s, 2H).

^{13}C NMR (100 MHz, CDCl_3): δ_{C} 191.99, 161.00, 153.81, 146.69, 137.31, 136.80, 133.52, 129.95, 129.74, 129.57, 129.06, 127.42, 124.22, 120.81, 118.81, 114.01.

54.11 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{19}\text{H}_{13}\text{ClN}_2\text{O}_5$: 384.0513, Found: 384.0511.

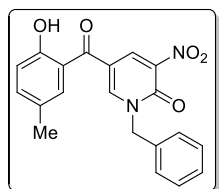
3r:1-Benzyl-5-(3,5-dichloro-2-hydroxybenzoyl)-3-nitropyridin-2(1H)-one



Isolated as yellow solid, 78 %, mp: 180-182 °C, ^1H NMR (400 MHz, CDCl_3): δ_{H} 11.07 (1H, s), 8.66 (1H, d, $J = 2.6$), 8.24 (1H, d, $J = 2.6$), 7.61 (1H, d, $J = 2.4$), 7.48–7.37 (5H, m), 7.26 (1H, d, $J = 2.2$), 5.29 (2H, s). ^{13}C NMR (100 MHz, CDCl_3): δ_{C} 191.44, 160.47, 156.13, 153.78, 147.03, 137.17, 136.03, 133.38, 129.75, 129.62, 129.10, 128.56, 124.77, 124.24, 119.88, 113.70, 54.14 ppm. EI-

HRMS: Anal. Calcd for $\text{C}_{19}\text{H}_{12}\text{Cl}_2\text{N}_2\text{O}_5$: 418.0123, Found: 418.0122.

3s:1-Benzyl-5-(2-hydroxy-5-methylbenzoyl)-3-nitropyridin-2(1H)-one

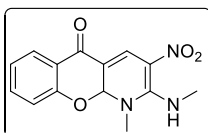


Isolated as yellow solid, 72 %, mp: 184-186°C, ^1H NMR (400 MHz, CDCl_3): δ_{H} 10.96 (1H, s), 8.67 (1H, d, $J = 2.5$), 8.26 (1H, d, $J = 2.5$), 7.46–7.39 (5H, m), 7.34 (1H, dd, $J = 8.5, 2.0$), 7.10 (1H, d, $J = 1.3$), 6.97 (1H, d, $J = 8.5$), 5.30 (2H, s), 2.22 (3 H, s). ^{13}C

NMR (100 MHz, CDCl_3): δ_{C} 192.91, 160.57, 153.93, 146.80, 138.09, 137.92, 137.80, 133.96, 130.73, 129.55, 129.37, 129.02, 128.69, 118.91, 117.83, 114.71, 53.84, 20.49 ppm. EI-

HRMS: Anal. Calcd for $\text{C}_{20}\text{H}_{16}\text{N}_2\text{O}_5$: 364.1059, Found: 364.1056.

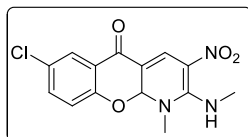
5a:1-methyl-2-(methylamino)-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one



Isolated as yellow solid, 94 %, mp: 200-202°C, ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 10.59 (d, $J = 3.7$ Hz, 1H), 7.90 (s, 1H), 7.81 (dd, $J = 7.8, 1.5$ Hz, 1H), 7.63 – 7.56 (m, 1H), 7.15 (t, $J = 7.2$ Hz, 1H), 7.07 (d, $J = 8.2$ Hz, 1H), 6.40 (d, $J = 16.6$ Hz, 1H), 3.51

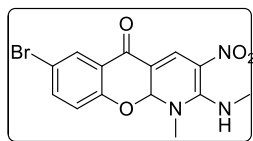
(s, 3H), 3.25 (d, $J = 5.1$ Hz, 3H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$) δ 179.39, 157.41, 156.49, 136.07, 126.85, 126.52, 122.87, 122.47, 118.14, 113.10, 112.97, 90.49, 42.71, 33.89. EI-HRMS: Anal. Calcd for $\text{C}_{14}\text{H}_{13}\text{N}_3\text{O}_4$: 287.0906, Found: 287.0902.

5b:7-chloro-1-methyl-2-(methylamino)-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one

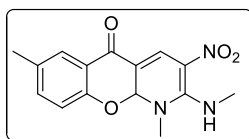


Isolated as yellow solid, 93 %, mp: 219-221°C, ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 10.54 (s, 1H), 7.94 (s, 1H), 7.74 (d, $J = 2.7$ Hz, 1H), 7.65 (dd, $J = 8.8, 2.7$ Hz, 1H), 7.14 (d, $J = 8.8$ Hz, 1H), 6.44 (s, 1H), 3.52 (s, 3H), 3.25 (d, $J = 5.2$ Hz,

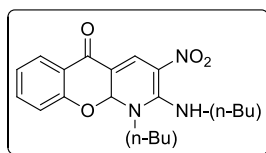
3H). ^{13}C NMR (100 MHz, DMSO) δ 179.42, 157.41, 154.48, 136.86, 131.61, 126.43, 126.36, 122.51, 117.94, 113.32, 112.92, 90.38, 42.68, 33.88 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{14}\text{H}_{12}\text{ClN}_3\text{O}_4$: 321.0516, Found: 321.0513

5c: 7-bromo-1-methyl-2-(methylamino)-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one

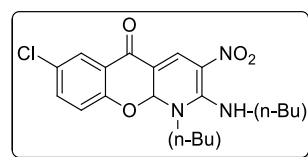
Isolated as yellow solid, 95 %, mp: 220-222°C, ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 10.54 (s, 1H), 7.94-7.81 (m, 2H), 7.74 (dd, $J = 8.7, 2.3$, 1H) 7.08 (d, $J = 8.8$ Hz, 1H), 6.45 (s, 1H), 3.52 (s, 3H), 3.25 (d, $J = 5.3$ Hz, 3H) ppm. ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ_c 178.67, 157.83, 156.03, 138.84, 132.12, 129.31, 124.96, 121.34, 114.69, 113.69, 112.39, 91.36, 43.32, 34.46 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{14}\text{H}_{12}\text{BrN}_3\text{O}_4$: 365.0011, Found: 365.0010

5d: 7-methyl-1-methyl-2-(methylamino)-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one

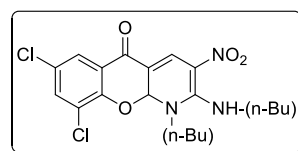
Isolated as yellow solid, 92 %, mp: 192-194°C, ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 10.60 (d, $J = 4.5$ Hz, 1H), 7.90 (s, 1H), 7.60 (d, $J = 1.5$ Hz, 1H), 7.42 (dd, $J = 8.4, 2.1$ Hz, 1H), 6.98 (d, $J = 8.4$ Hz, 1H), 6.33 (s, 1H), 3.51 (s, 3H), 3.26 (d, $J = 5.2$ Hz, 3H), 2.30 (s, 3H). ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 179.42, 157.42, 154.49, 136.86, 131.62, 126.43, 126.36, 122.52, 117.94, 113.32, 112.93, 90.39, 42.66, 33.87, 20.00. EI-HRMS: Anal. Calcd for $\text{C}_{15}\text{H}_{15}\text{N}_3\text{O}_4$: 301.1063, Found: 301.1060

5e: 1-Butyl-2-(butylamino)-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one

Isolated as yellow semi solid, 87 %, ^1H NMR (400 MHz, CDCl_3) δ_H 11.07 (1 H, d, $J = 2.5$), 8.16 (1 H, s), 7.92 (1 H, dd, $J = 7.8, 1.6$), 7.54 – 7.43 (1 H, m), 7.12 – 7.02 (1 H, m), 6.97 (1 H, d, $J = 8.2$), 6.13 (1 H, s), 3.91 – 3.43 (4 H, m), 1.92 – 1.71 (4 H, m), 1.50 (2 H, dd, $J = 15.1, 7.5$), 1.46 – 1.30 (2 H, m), 0.99 (6 H, td, $J = 7.3, 5.7$). ^{13}C NMR (100 MHz, CDCl_3) δ_c 180.44, 157.03, 156.66, 135.87, 127.65, 123.39, 122.66, 117.98, 114.18, 114.12, 89.09, 54.18, 47.48, 32.10, 29.97, 19.86, 19.83, 13.73, 13.52 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{20}\text{H}_{25}\text{N}_3\text{O}_4$: 371.1845, Found: 371.1838

5f: 1-Butyl-2-(butylamino)-7-chloro-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one

Isolated as yellow semi solid, 85 %, ^1H NMR (400 MHz, CDCl_3) δ_H 11.01 (1H, s), 8.19 (1H, s), 7.88 (1H, d, $J = 1.1$), 7.43 (1H, dd, $J = 8.7, 2.8$), 6.93 (1H, d, $J = 8.8$), 6.12 (1H, s), 3.92 – 3.79 (1H, m), 3.78 – 3.64 (1H, m), 3.65 – 3.53 (1H, m), 3.48 (1H, dd, $J = 12.5, 6.3$), 1.79 (4 H, dd, $J = 12.9, 5.7$), 1.50 (2H, dd, $J = 15.0, 7.5$), 1.45 – 1.33 (2H, m), 0.99 (6H, td, $J = 7.3, 5.4$) ppm. ^{13}C NMR (100 MHz, CDCl_3) δ_c 179.26, 157.01, 154.99, 135.60, 128.53, 128.21, 127.07, 124.33, 119.64, 114.36, 112.99, 89.32, 54.31, 47.54, 32.10, 29.95, 19.87, 19.83, 13.72, 13.51 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{20}\text{H}_{24}\text{ClN}_3\text{O}_4$: 405.1455, Found: 405.1454

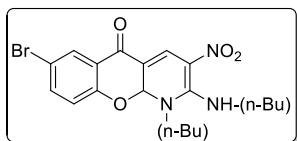
5g: 1-Butyl-2-(butylamino)-7,9-dichloro-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one

Isolated as yellow semi solid, 89 %, ^1H NMR (400 MHz, CDCl_3) δ_H 11.04 (1 H, s), 8.25 (1 H, s), 7.81 (1 H, d, $J = 2.5$), 7.55 (1H, d, $J = 2.5$), 6.19 (1H,

s), 3.78 (2H, t, $J = 7.9$), 3.66–3.40 (2H, m), 2.03 – 1.77 (4H, m), 1.59–1.33 (4 H, m). 1.0 (6H, t, $J = 7.0$). ^{13}C NMR (100 MHz, CDCl_3) δ_{C} 178.41, 156.95, 155.36, 135.17, 129.41, 127.94, 125.82, 125.24, 124.17, 114.39, 111.77, 90.44, 54.63, 47.61, 32.14, 30.40, 19.95, 19.87, 13.71, 13.51 ppm. EI-

HRMS: Anal. Calcd for $\text{C}_{20}\text{H}_{23}\text{Cl}_2\text{N}_3\text{O}_4$: 439. 1066, Found: 439. 1063

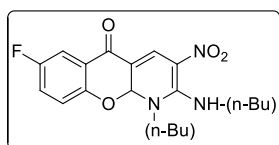
5h: 7-Bromo-1-butyl-2-(butylamino)-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one



Isolated as yellow semi solid, 80 %, ^1H NMR (400 MHz, CDCl_3) δ_{H} 11.01 (1H, d, $J = 2.5$), 8.18 (1H, s), 8.02 (1H, d, $J = 2.5$), 7.56 (1H, dd, $J = 8.7$, 2.5), 6.87 (1H, d, $J = 8.7$), 6.12 (1H, s), 3.97– 3.34 (4H, m), 1.94–1.69 (4H, m), 1.58–1.33 (4H, m), 0.99 (6H, td, $J = 7.3$, 5.1) ppm. ^{13}C NMR (100 MHz,

CDCl_3) δ_{C} 179.14, 157.00, 155.47, 138.41, 130.16, 128.55, 124.75, 119.99, 115.42, 114.36, 112.90, 89.31, 54.32, 47.54, 32.10, 29.96, 19.87, 19.83, 13.73, 13.52 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{20}\text{H}_{24}\text{BrN}_3\text{O}_4$: 449.0950, Found: 449.0950

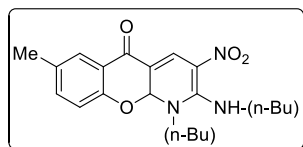
5i: 1-Butyl-2-(butylamino)-7-fluoro-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one



Isolated as yellow semi solid, 84 %, ^1H NMR (400 MHz, CDCl_3) δ_{H} 11.03 (1 H, s), 8.18 (1 H, d, $J = 2.5$), 7.60 – 7.53 (1 H, m), 7.24 – 7.15 (1 H, m), 6.96 (1 H, dd, $J = 9.0$, 4.2), 6.12 (1 H, s), 3.90 – 3.43 (4 H, m), 1.80 (4 H, dd, $J = 14.3$, 7.0), 1.45 (4 H, ddd, $J = 44.7$, 14.8, 7.4), 1.00 (6 H, dt, $J = 11.8$, 6.0) ppm. ^{13}C

NMR (100 MHz, CDCl_3) δ_{C} 179.56, 157.04, 152.69-152.68 (d, $J = 1.96$ Hz), 128.42, 124.27-124.20 (d, $J = 6.47$ Hz), 123.28-123.03 (d, $J = 24.31$ Hz), 119.64-119.56 (d, $J = 7.59$ Hz), 114.34, 113.20, 113.03, 112.79, 89.25, 54.24, 47.53, 32.09, 29.93, 19.86, 19.83, 13.72, 13.51 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{20}\text{H}_{24}\text{FN}_3\text{O}_4$: 389.1751, Found: 389.1748.

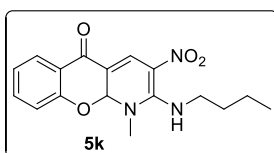
5j: 1-Butyl-2-(butylamino)-7-methyl-3-nitro-1H-chromeno[2,3-b]-pyridin-5(10aH)-one



Isolated as yellow semi solid, 86 %, ^1H NMR (400 MHz, CDCl_3) δ_{H} 11.09 (1H, d, $J = 10.6$), 8.20 (1H, d, $J = 0.4$), 7.76 (1H, d, $J = 1.6$), 7.32 (1H, dd, $J = 8.3$, 2.1), 6.87 (1H, d, $J = 8.4$), 6.08 (1H, s), 3.82 – 3.45 (4H, m), 1.78 (4H, m), 1.53 (2H, m), 1.37 (2H, dd, $J = 14.7$, 7.3), 1.0 (6H, t, $J = 6.9$) ppm. ^{13}C

NMR (100 MHz, CDCl_3) δ_{C} 180.65, 157.12, 154.63, 136.87, 132.35, 127.64, 127.44, 123.07, 117.69, 114.36, 114.23, 89.02, 54.09, 47.50, 32.13, 29.92, 20.50, 19.88, 19.84, 13.73, 13.53 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{21}\text{H}_{27}\text{N}_3\text{O}_4$: 385.2002, Found: 385.2000

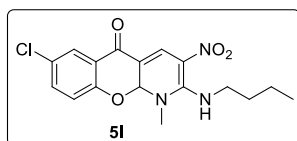
5k: 2-(butylamino)-1-methyl-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one



Isolated as yellow semi solid, 80 %, ^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ_{H} 11.68 (1H, s), 8.94 (1H, s), 8.85 (1H, d, $J = 7.6$), 8.65 (1H, t, $J = 7.6$), 8.20 (1H, t, $J = 7.5$), 8.12 (1H, d, $J = 8.3$), 7.42 (1H, s) 4.81–4.74 (1 H, m), 4.63 (1H, d, $J =$

4.6), 4.55 (3H, s), 2.73–2.61 (2H, m), 2.41 (2H, dd, $J = 14.9, 7.3$), 1.95 (3H, t, $J = 7.3$) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ_{C} 181.03, 158.18, 158.02, 137.75, 128.41, 127.85, 124.34, 124.12, 119.70, 115.08, 114.48, 91.85, 48.25, 44.32, 33.15, 20.70, 14.92 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{17}\text{H}_{19}\text{N}_3\text{O}_4$: 329.1376, Found: 329.1371.

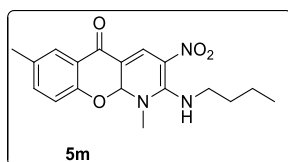
5l:2-(butylamino)-7-chloro-1-methyl-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one



Isolated as yellow semi solid, 84 %, ^1H NMR (400 MHz, DMSO- d_6) δ_{H} 10.60 (1H, s), 7.93 (1H, s), 7.73 (1H, d, $J = 2.7$), 7.64 (1H, dd, $J = 8.8, 2.7$), 7.13 (1H, d, $J = 8.8$), 6.44 (1H, s), 3.84–3.67 (1H, m), 3.67–3.54 (1H, m), 3.51 (3H, s), 1.65 (2H, dt, $J = 14.0, 6.9$), 1.45–1.31 (2H, m), 0.92 (3H, t, $J = 7.4$)

ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ_{C} 178.80, 157.09, 155.63, 136.13, 127.13, 126.28, 124.48, 121.03, 118.86, 113.65, 112.89, 91.13, 47.24, 43.40, 32.09, 19.67, 13.89 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{17}\text{H}_{18}\text{ClN}_3\text{O}_4$: 363.0986, Found: 363.0980.

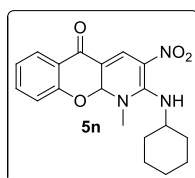
5m:2-(butylamino)-1,7-dimethyl-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one



Isolated as yellow semi solid, 83 %, ^1H NMR (400 MHz, DMSO- d_6) δ_{H} 10.65 (1H, d, $J = 2.5$), 7.88 (1H, s), 7.59 (1H, d, $J = 1.6$), 7.41 (1H, dd, $J = 8.4, 2.1$), 6.96 (1H, d, $J = 8.4$), 6.31 (1H, s), 3.85–3.64 (1H, m), 3.57 (1H, dd, $J = 13.6, 7.1$), 3.50 (3H, s), 2.29 (3H, s), 1.71–1.57 (2H, m), 1.36 (2H, m), 0.91 (3H, t, $J = 7.4$)

ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ_{C} 179.95, 157.14, 154.98, 137.43, 132.20, 126.93, 126.65, 122.97, 118.44, 114.21, 113.40, 90.72, 47.21, 43.20, 32.14, 20.49, 19.68, 13.87. EI-HRMS: Anal. Calcd for $\text{C}_{18}\text{H}_{21}\text{N}_3\text{O}_4$: 343.1532, Found: 343.1529.

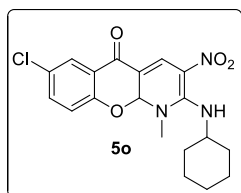
5n:1-benzyl-5-(5-chloro-2-hydroxybenzoyl)-3-nitropyridin-2(1H)-one



Isolated as yellow semi solid, 85 %, ^1H NMR (400 MHz, DMSO- d_6) δ_{H} 10.69 (1H, d, $J = 8.2$), 7.89 (1H, s), 7.81 (1H, dd, $J = 7.8, 1.3$), 7.6–7.51 (1H, m), 7.16 (2H, d, $J = 7.4$), 6.40 (1H, s), 4.02–3.92 (1H, m), 3.50 (3H, s), 2.08 (1H, d, $J = 9.6$), 1.91–1.81 (1H, m), 1.71 (1H, d, $J = 8.8$), 1.60–1.14 (7H, m) ppm. ^{13}C NMR (100 MHz, DMSO- d_6) δ_{C}

179.94, 156.92, 156.37, 136.66, 127.35, 126.64, 123.32, 123.06, 118.68, 114.12, 113.84, 90.94, 55.21, 43.24, 34.12, 33.15, 25.17, 24.07, 23.88 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{19}\text{H}_{21}\text{N}_3\text{O}_4$: 355.1532, Found: 355.1533.

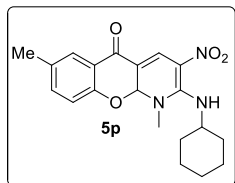
5o:7-chloro-2-(cyclohexylamino)-1-methyl-3-nitro-1H-chromeno[2,3-b]pyridin-5(10aH)-one



Isolated as yellow semi solid, 87 %, ^1H NMR (400 MHz, DMSO- d_6) δ_{H} 10.45 (1H, d, $J = 7.7$), 7.71 (1H, s), 7.51 (1H, d, $J = 2.1$), 7.42 (1H, dd, $J = 8.7, 2.2$), 6.92 (1H, d, $J = 8.8$), 6.23 (1H, s), 3.77 (1H, s), 3.29 (3H, s), 1.87 (1H, s), 1.67 (1H, d,

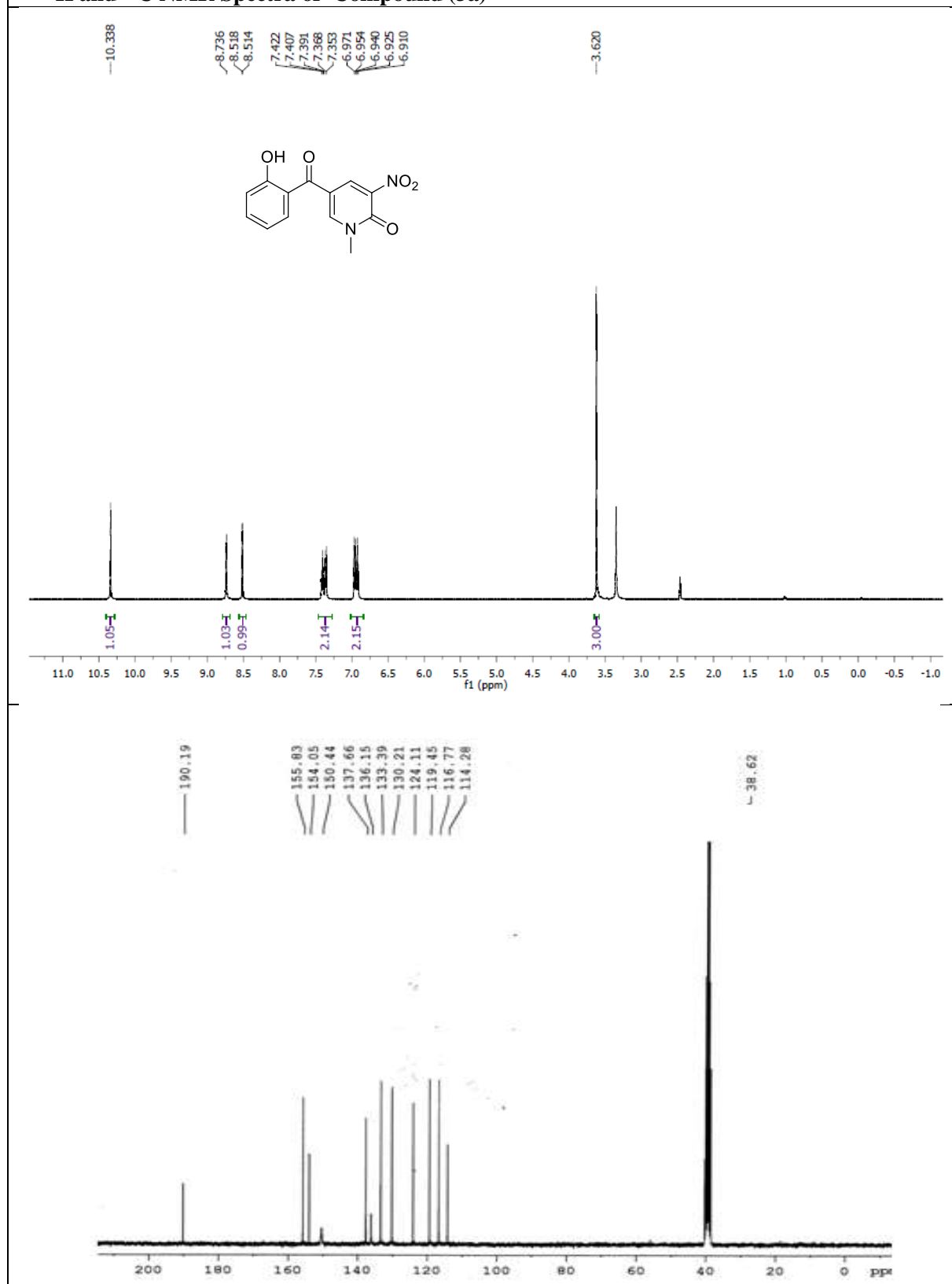
$J = 8.5$), 1.50 (1H, s), 1.44 –1.02 (7H, m) ppm. ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ_{C} 178.87, 156.32, 155.59, 136.19, 127.50, 127.18, 126.28, 124.47, 121.10, 114.02, 113.08, 91.21, 55.27, 43.40, 34.07, 33.11, 25.14, 24.05, 23.89 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{19}\text{H}_{20}\text{ClN}_3\text{O}_4$: 389.1142, Found: 389.1140.

5p:2-(cyclohexylamino)-1,7-dimethyl-3-nitro-1*H*-chromeno[2,3-*b*]pyridin-5(10*aH*)-one

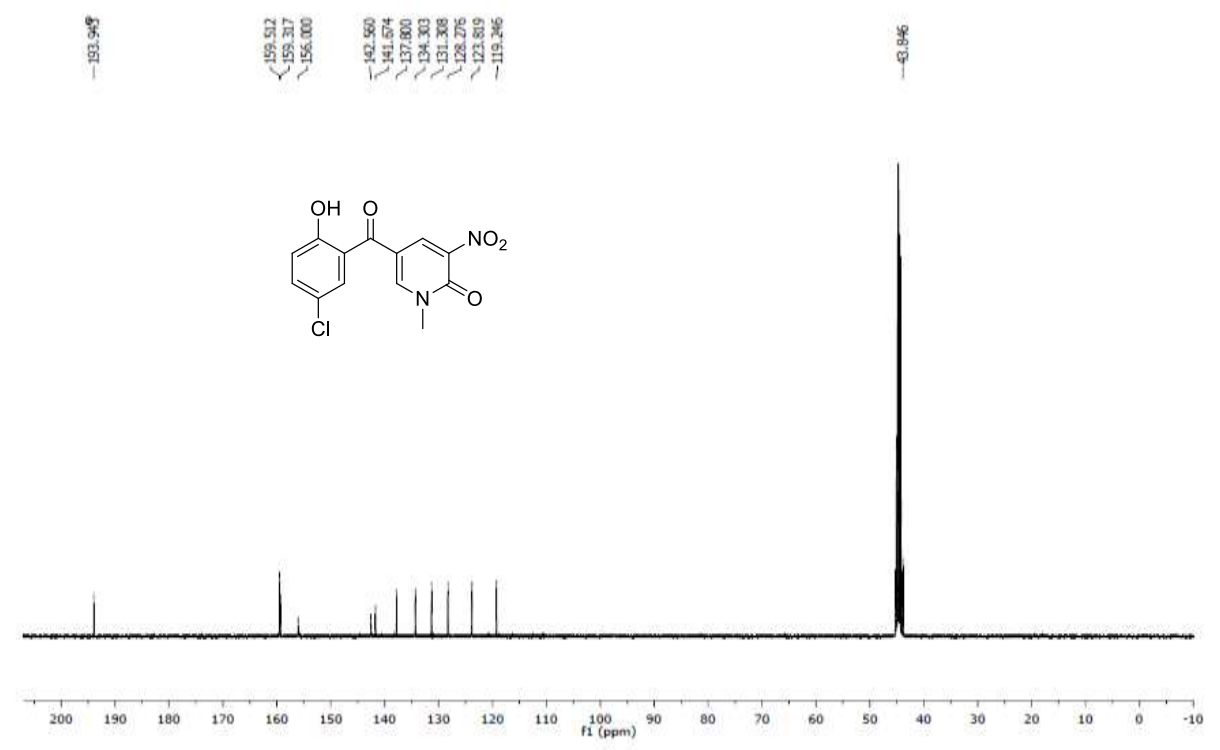
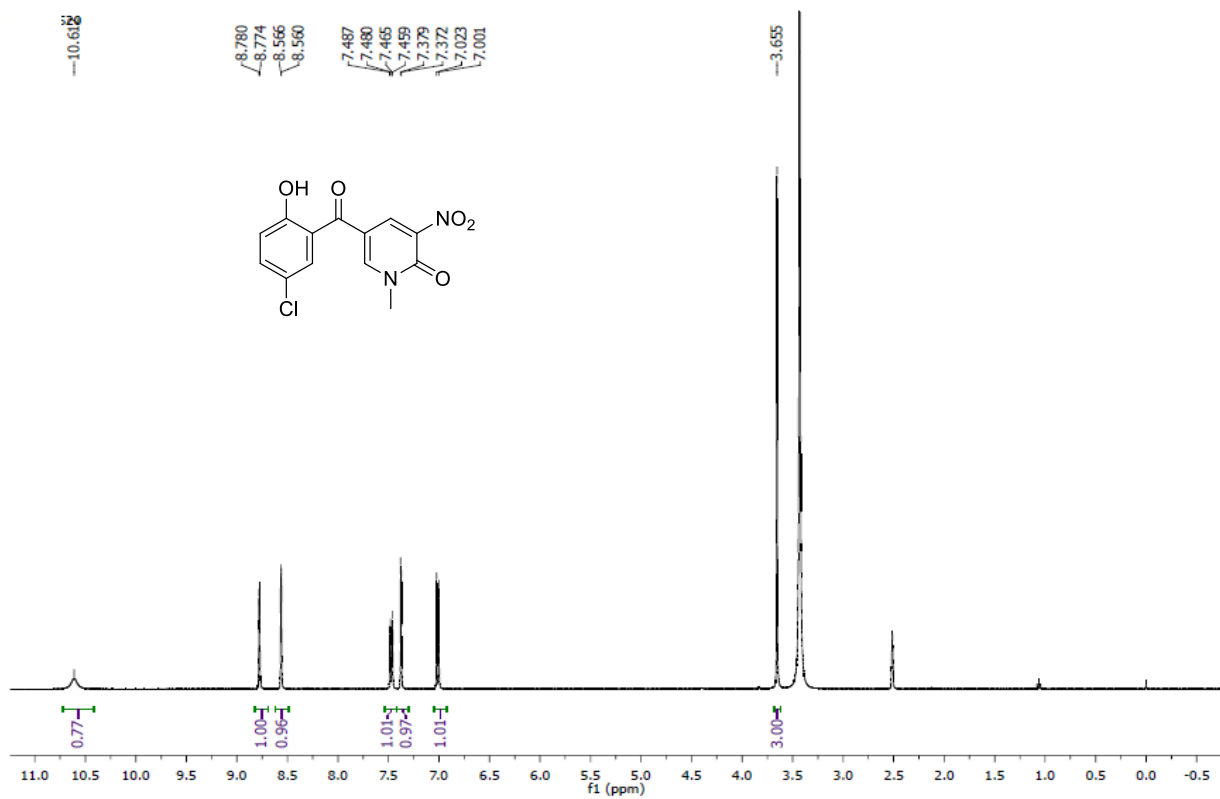


Isolated as yellow semi solid, 86 %, ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ_{H} 10.70 (1 H, d, $J = 8.2$), 7.88 (1H, s), 7.60 (1H, d, $J = 1.3$), 7.42 (1H, dd, $J = 8.4, 2.0$), 6.99 (1H, d, $J = 8.4$), 6.36 (1H, s), 4.01–3.91 (1H, m), 3.49 (3H, s), 2.29 (3H, s), 2.08 (1H, d, $J = 9.9$), 1.95–1.82 (1H, m), 1.72 (1H, dd, $J = 9.4, 3.8$), 1.45 (7H, m) ppm. ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ_{C} 180.04, 156.41, 154.95, 137.52, 132.27, 126.95, 126.49, 122.98, 118.52, 114.40, 113.79, 90.84, 55.20, 43.24, 34.10, 33.14, 25.16, 24.06, 23.87, 20.50 ppm. EI-HRMS: Anal. Calcd for $\text{C}_{20}\text{H}_{23}\text{N}_3\text{O}_4$: 369.1689, Found: 369.1684.

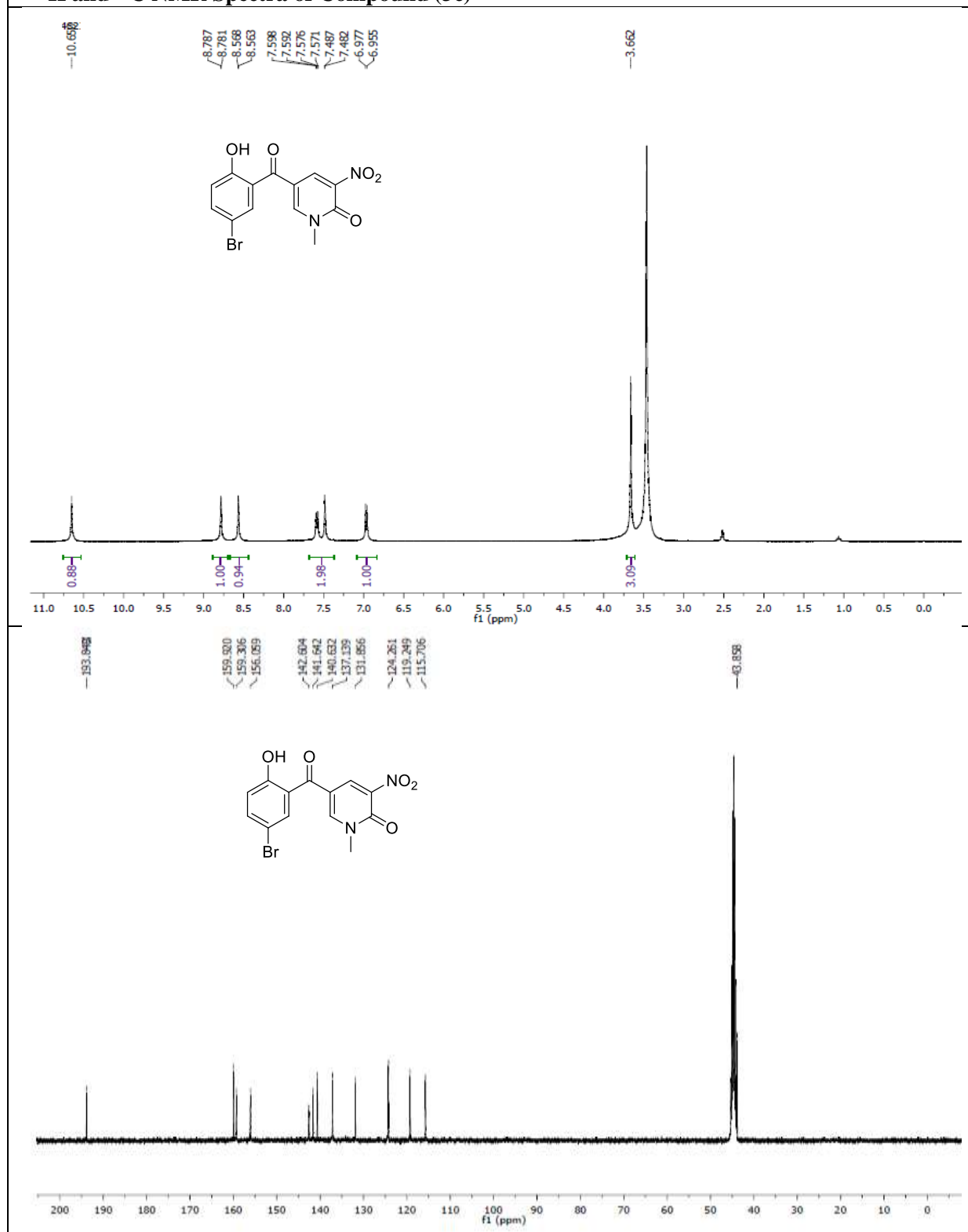
¹H and ¹³C NMR Spectra of Compound (3a)



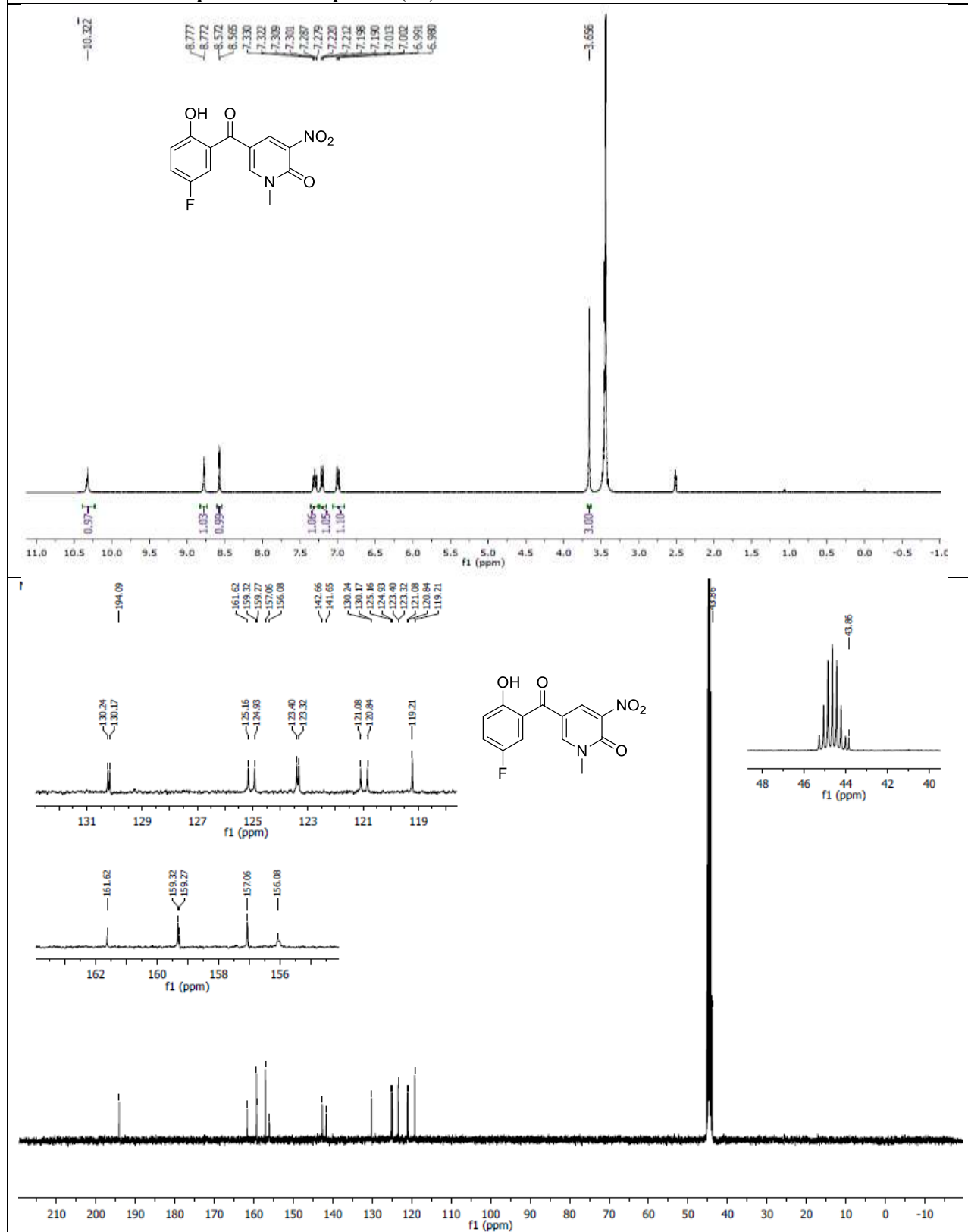
¹H and ¹³C NMR Spectra of Compound (3b)



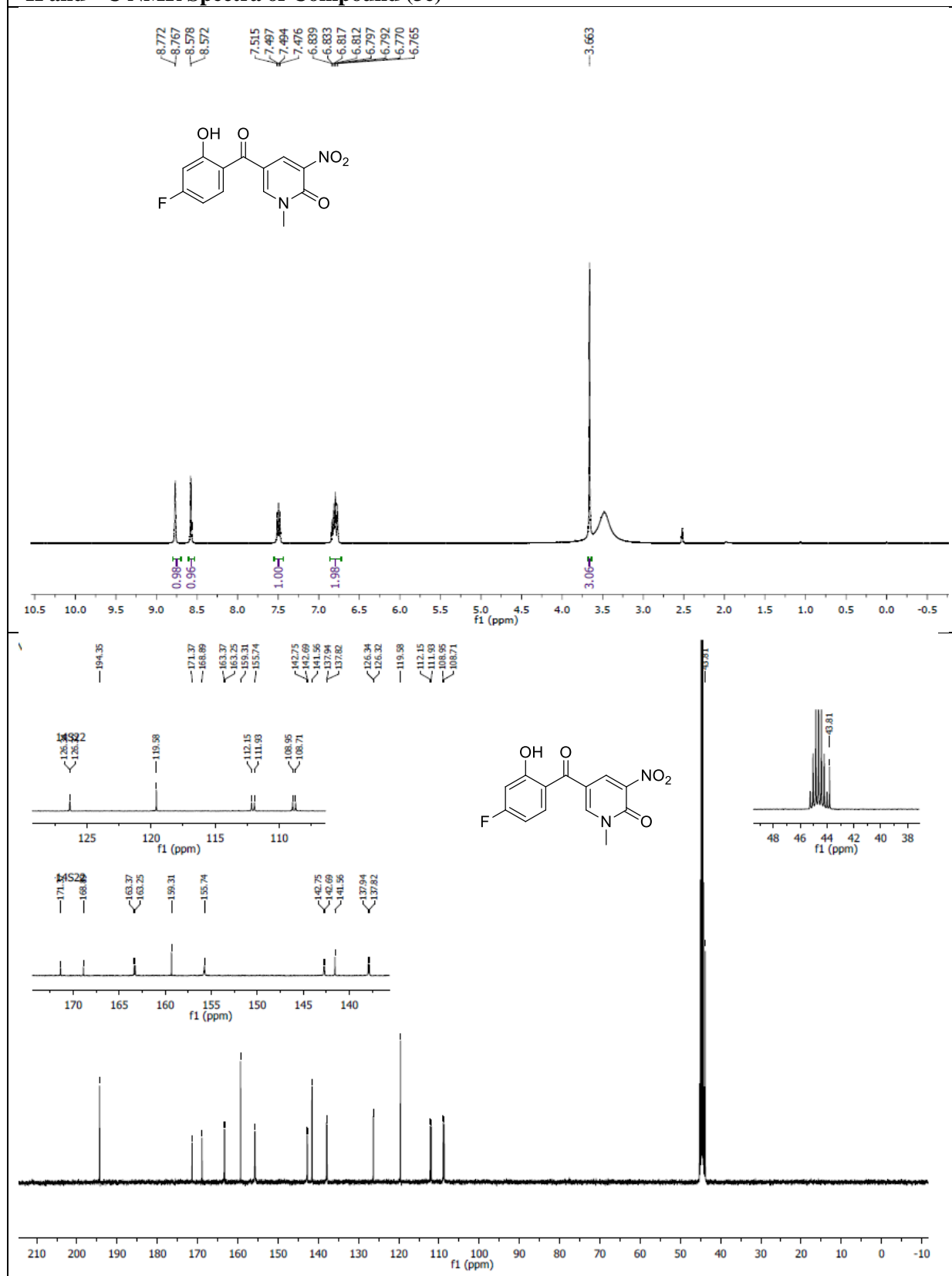
¹H and ¹³C NMR Spectra of Compound (3c)



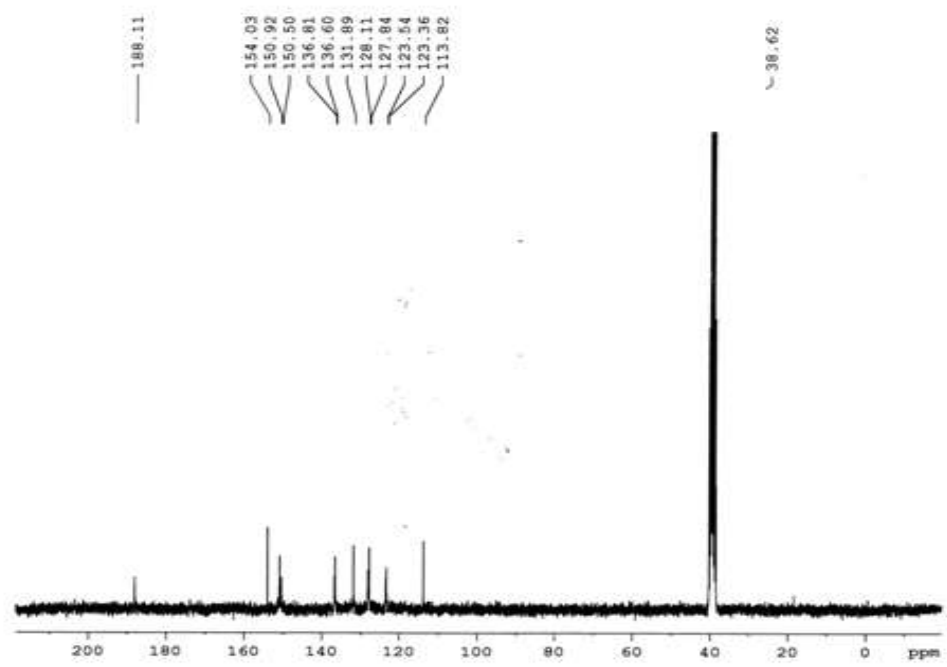
¹H and ¹³C NMR Spectra of Compound (3d)



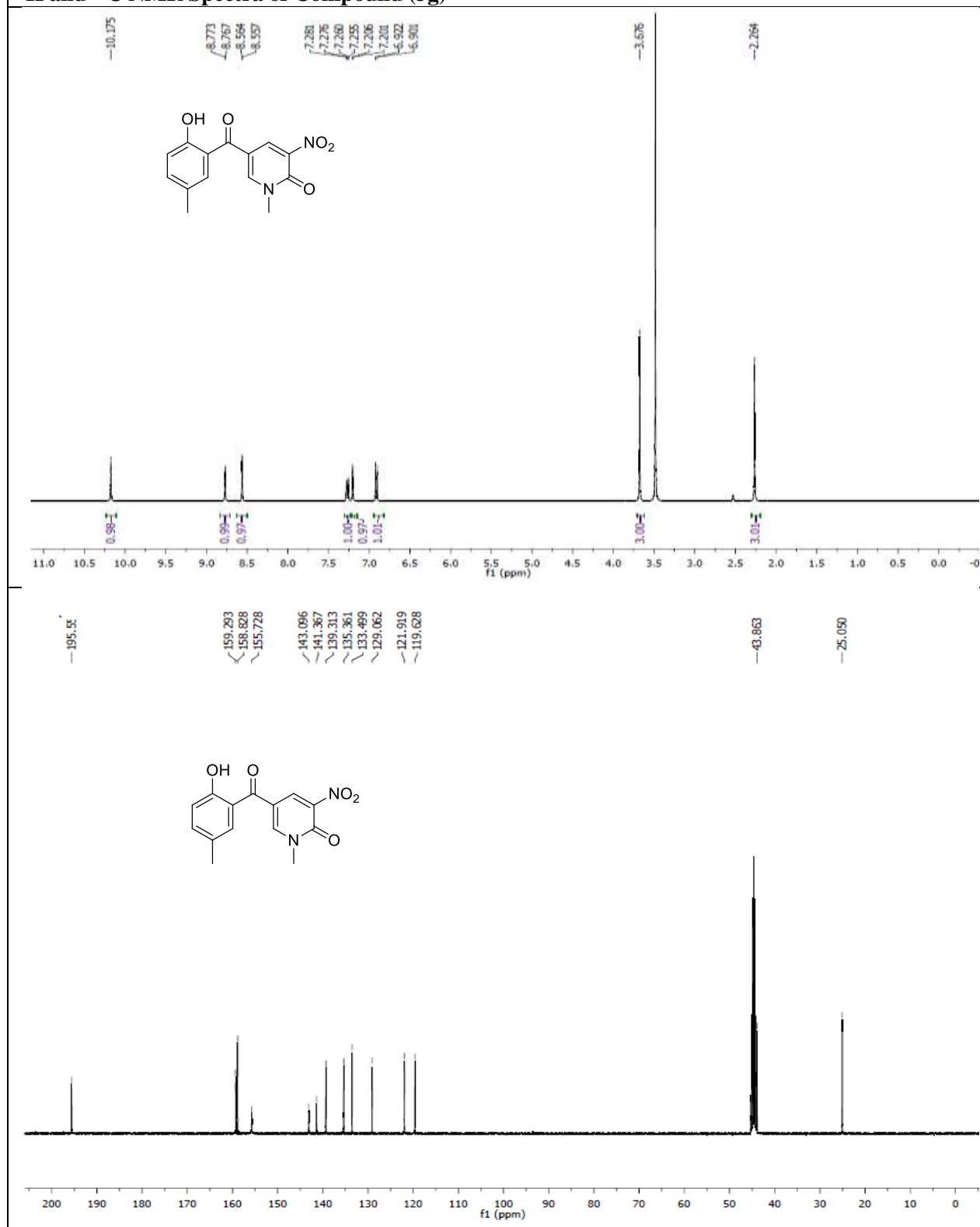
¹H and ¹³C NMR Spectra of Compound (3e)



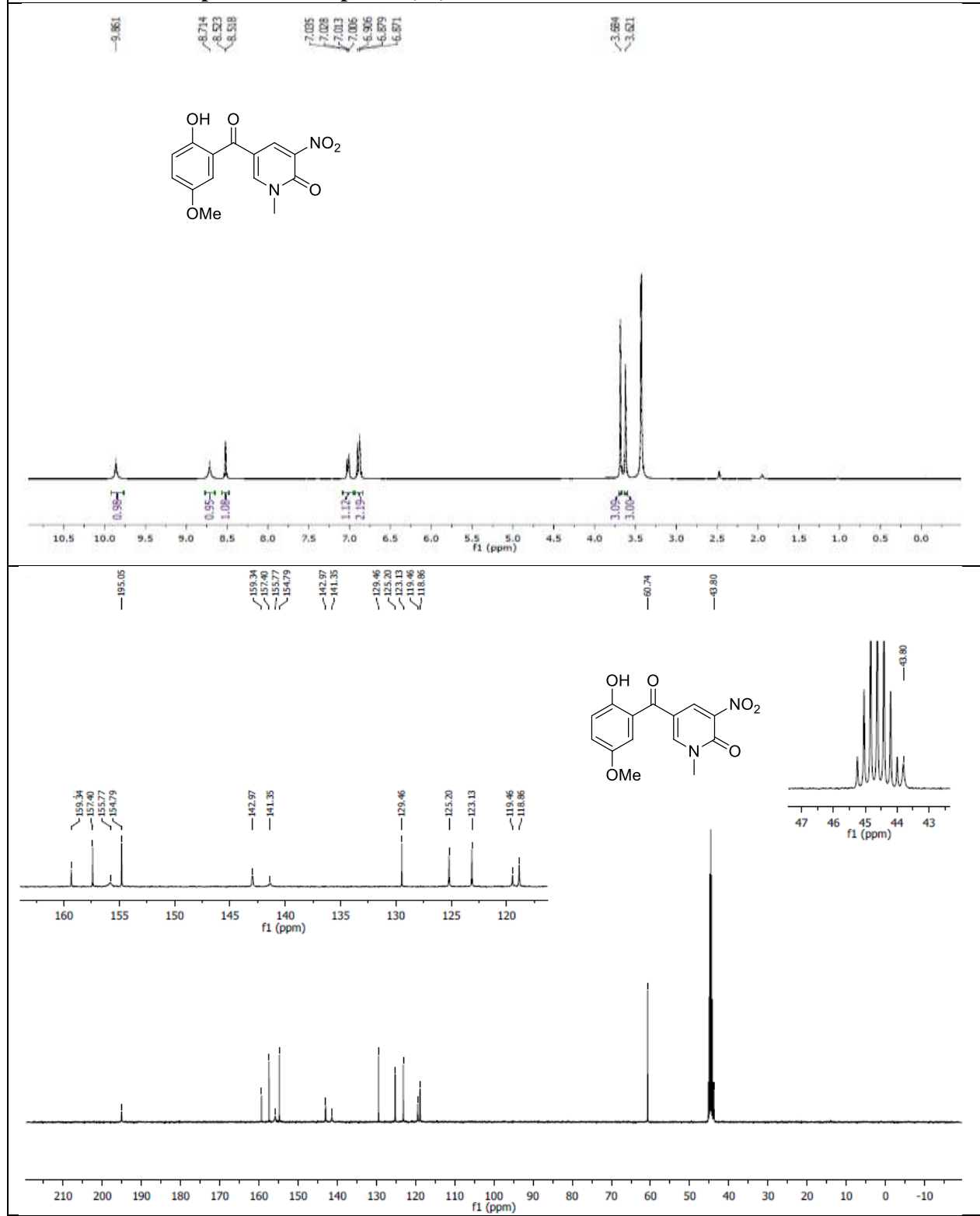
¹H and ¹³C NMR Spectra of Compound (3f)



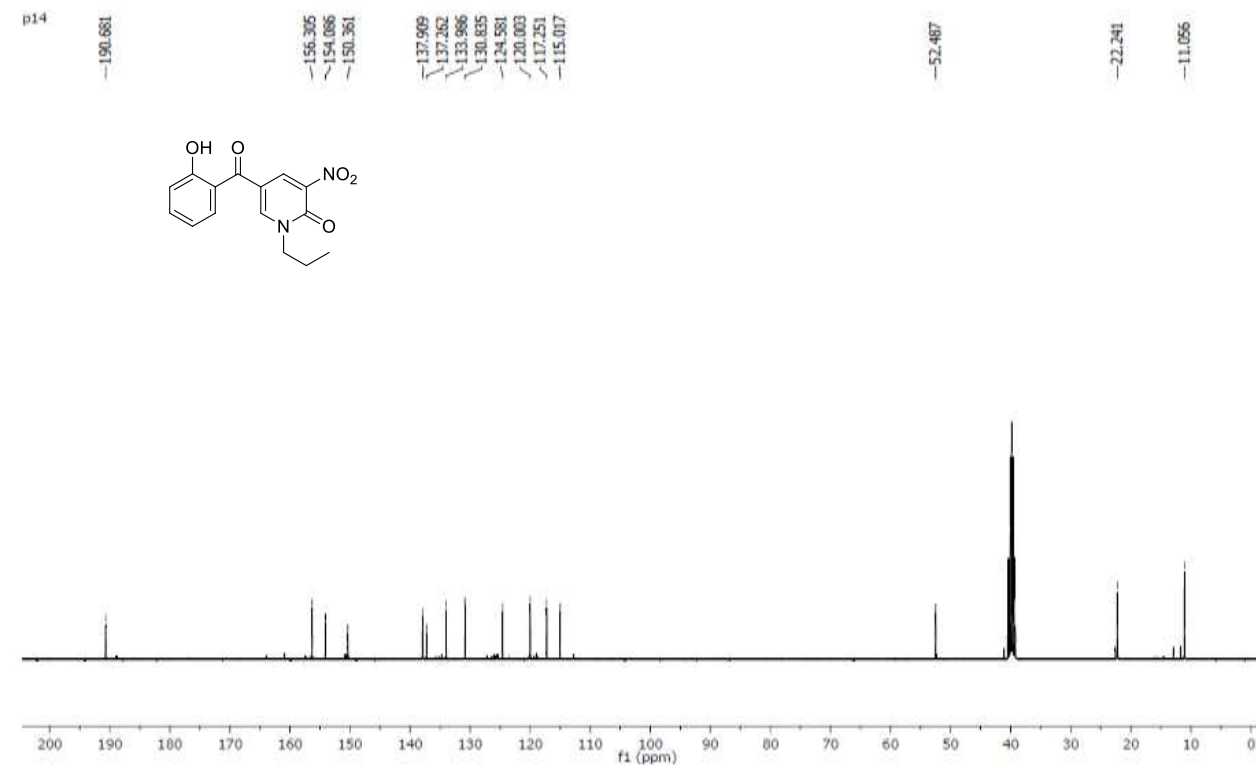
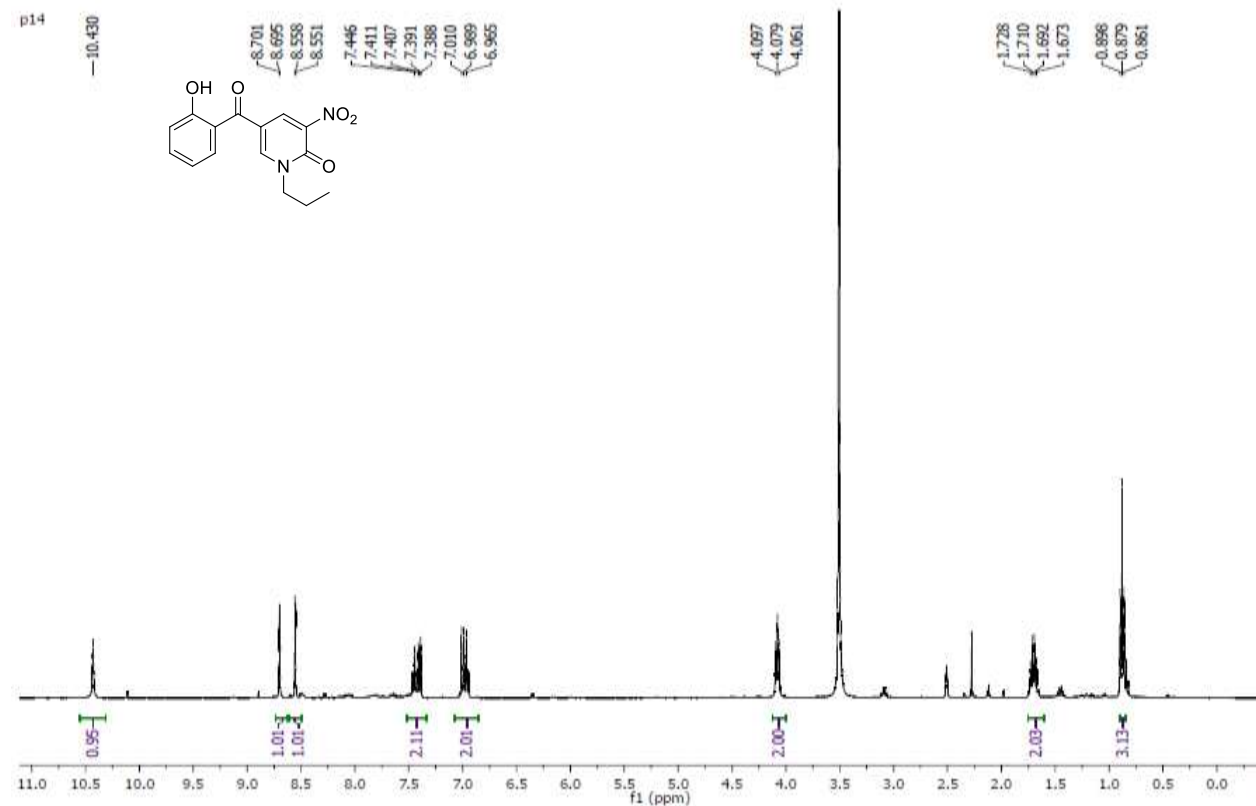
¹H and ¹³C NMR Spectra of Compound (3g)



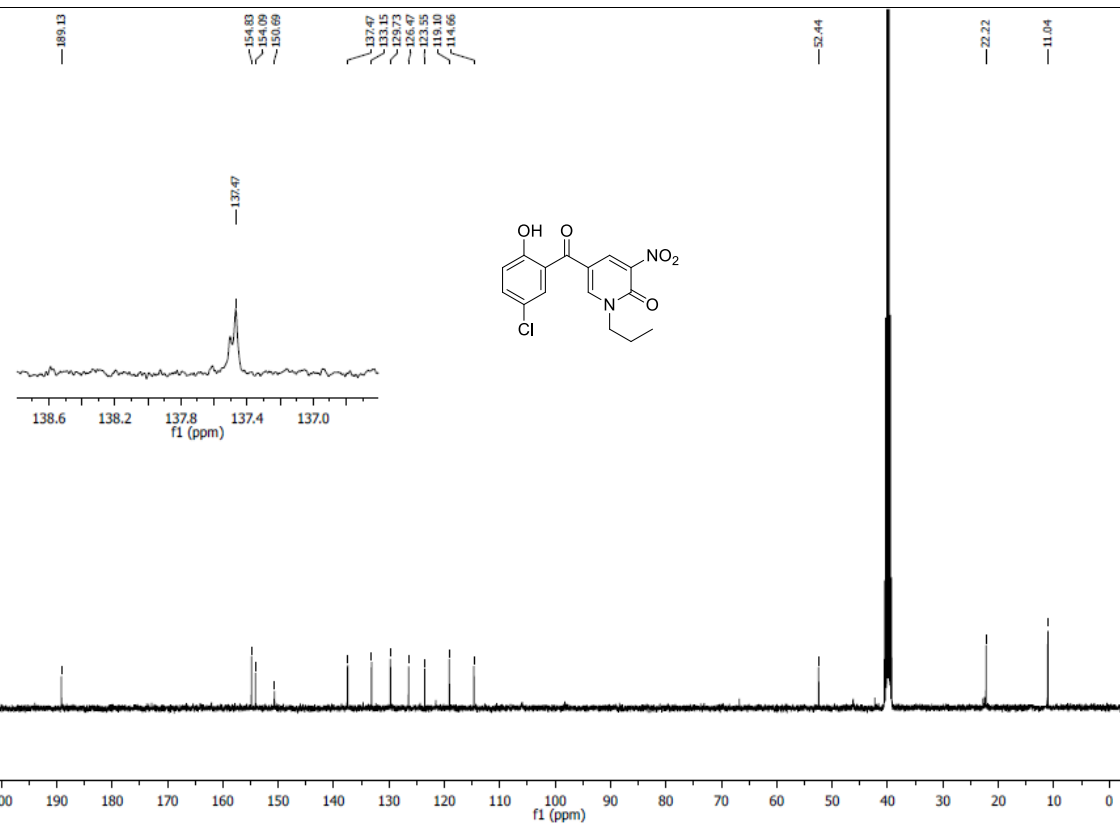
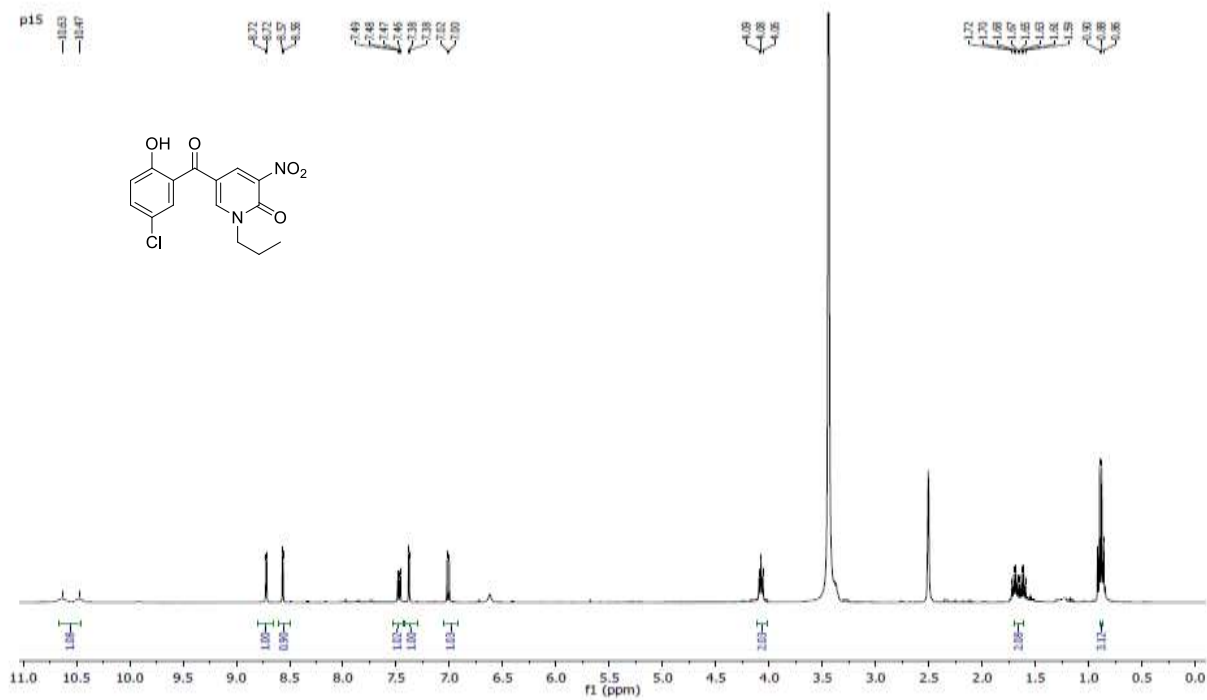
¹H and ¹³C NMR Spectra of Compound (3h)



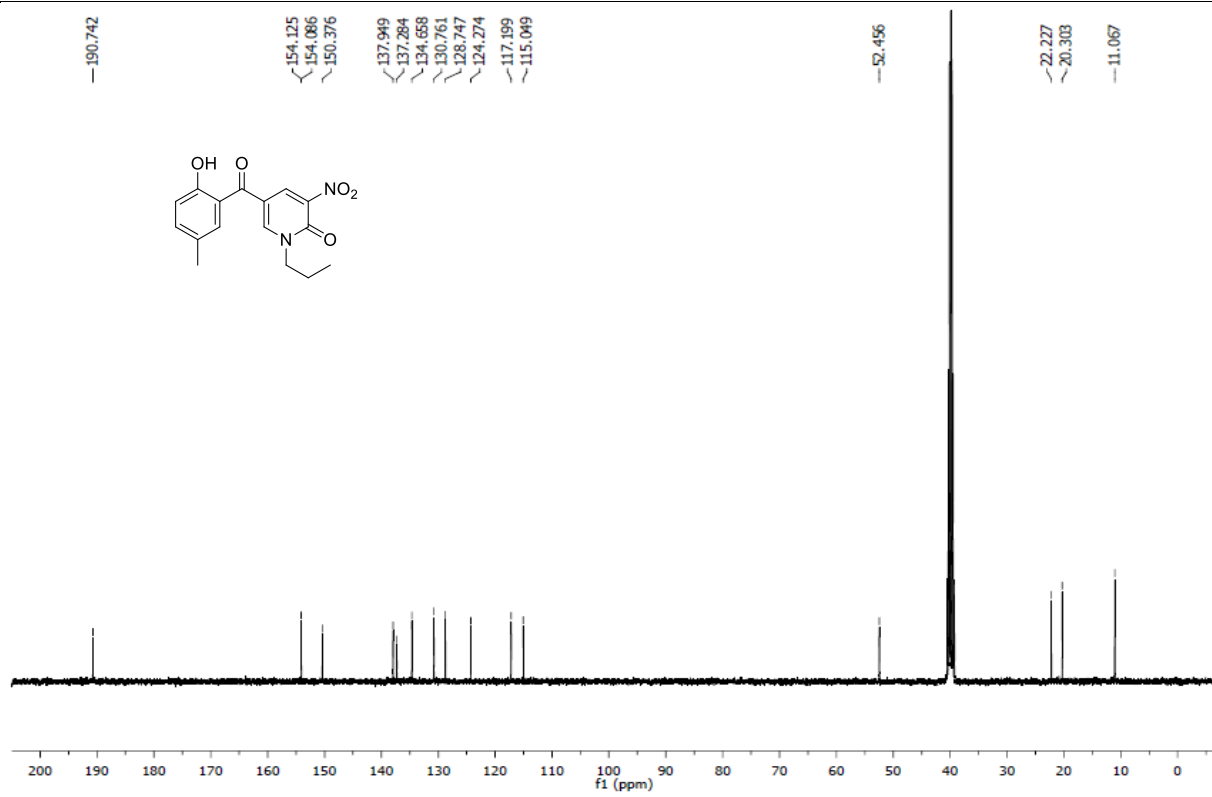
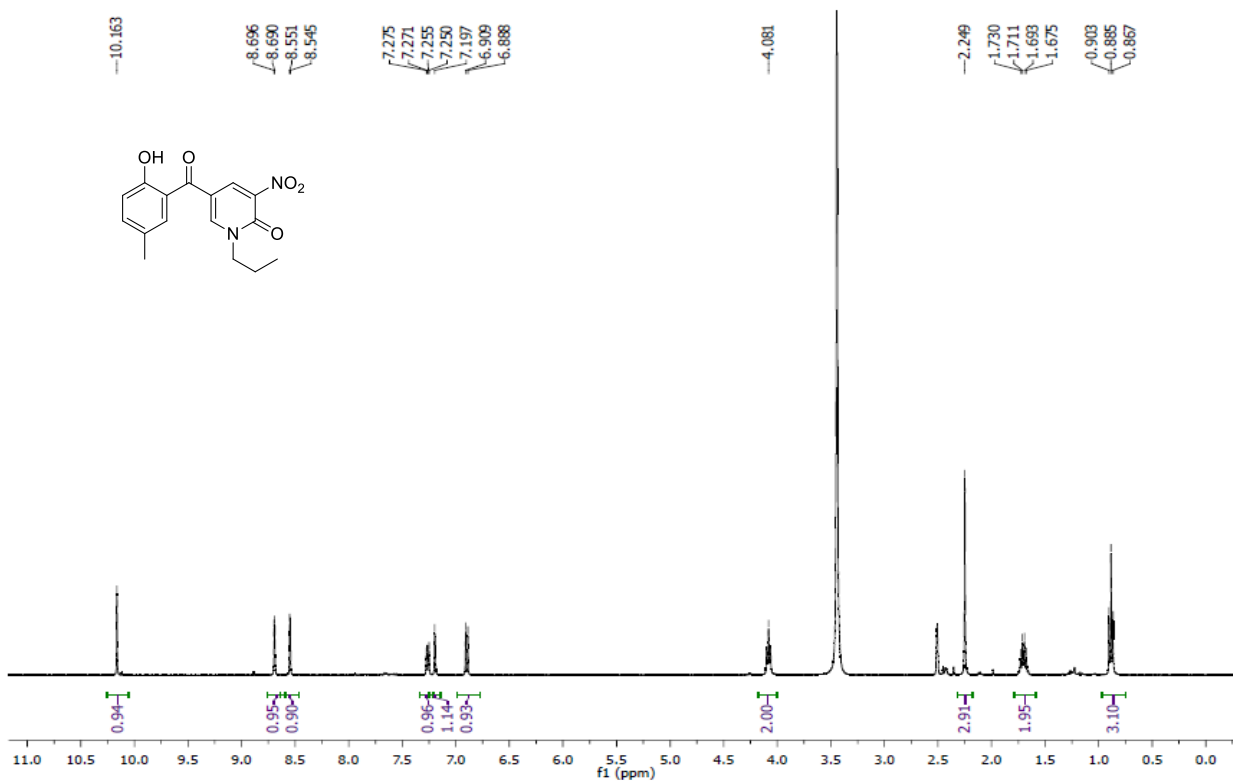
¹H and ¹³C NMR Spectra of Compound (3i)



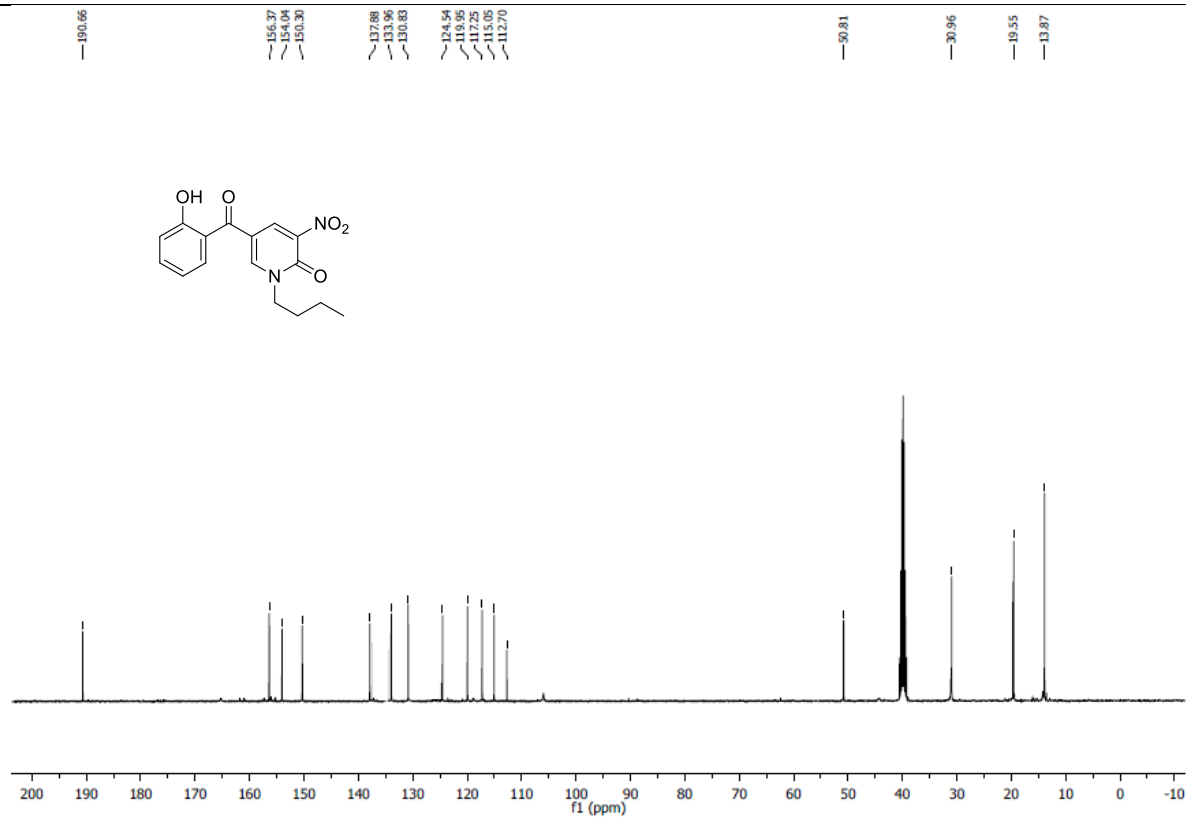
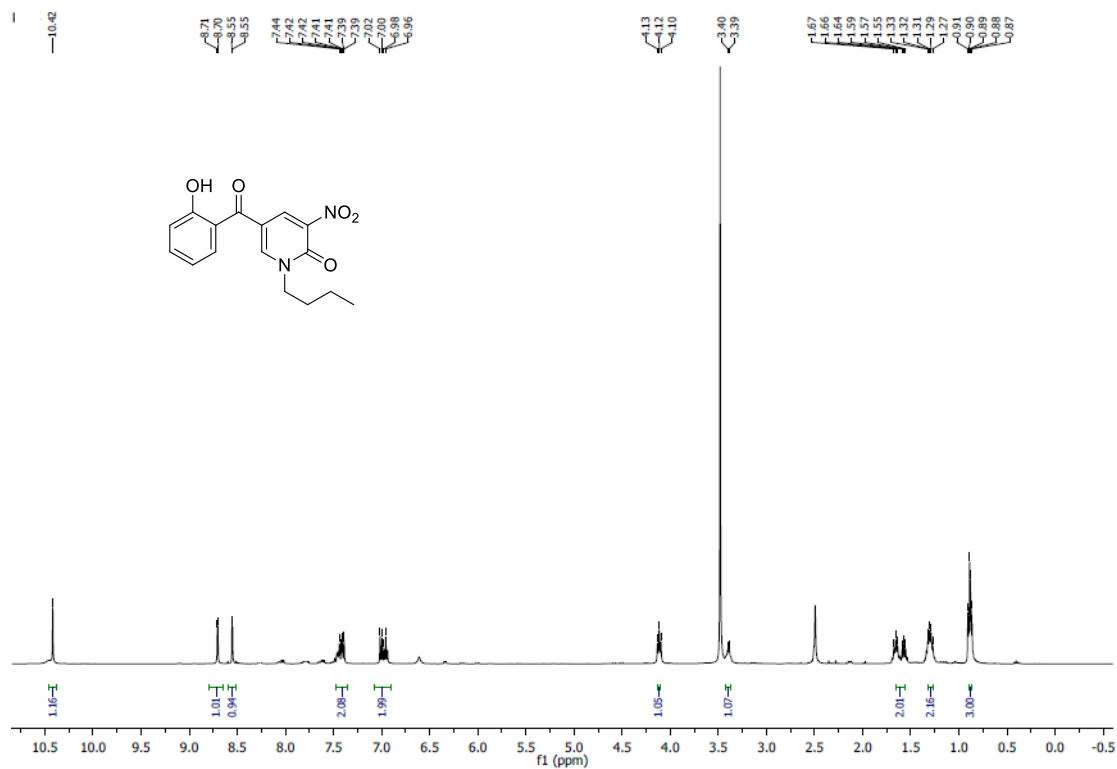
¹H and ¹³C NMR Spectra of Compound (3j)



¹H and ¹³C NMR Spectra of Compound (3k)



¹H and ¹³C NMR Spectra of Compound (31)



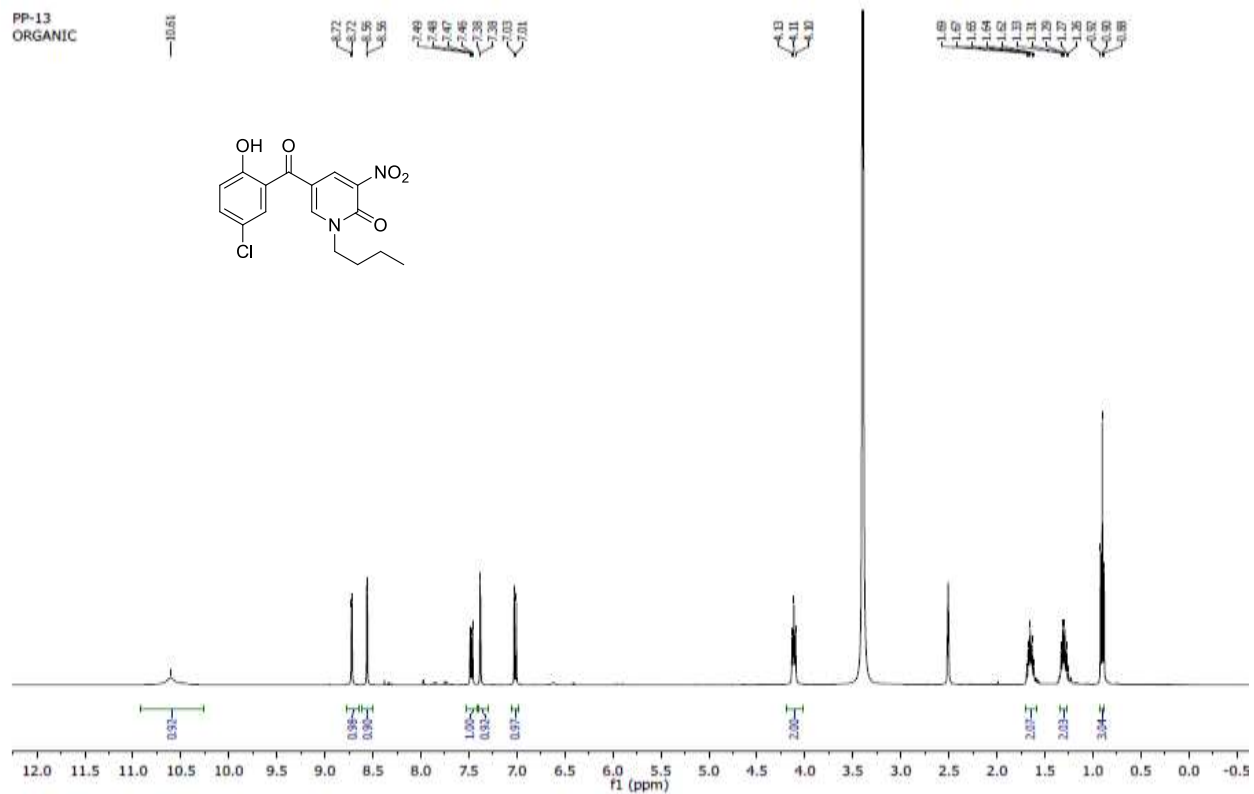
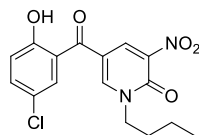
¹H and ¹³C NMR Spectra of Compound (3m)

PP-13
ORGANIC

10.51
7.72
7.72
7.55
7.49
7.48
7.47
7.46
7.38
7.38
7.03
7.01

4.13
4.11
4.10

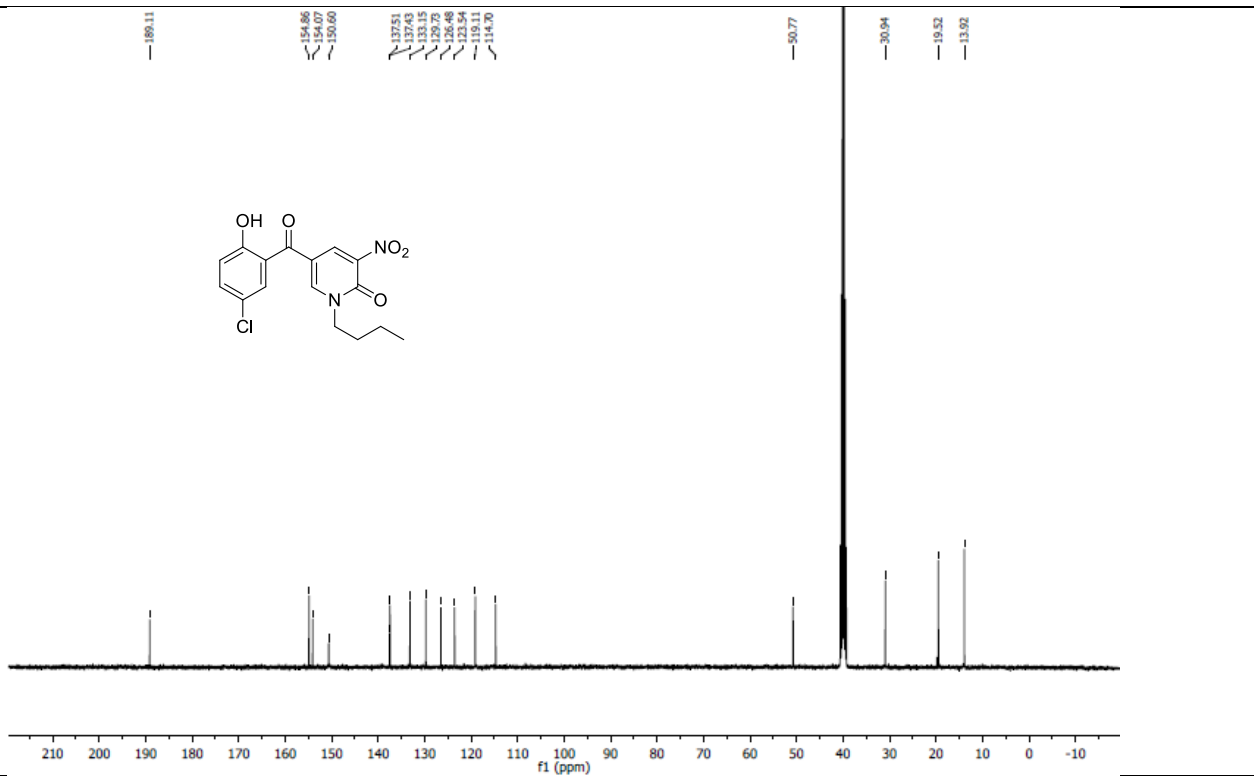
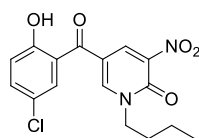
1.69
1.65
1.64
1.62
1.53
1.51
1.29
1.28
1.00
0.98



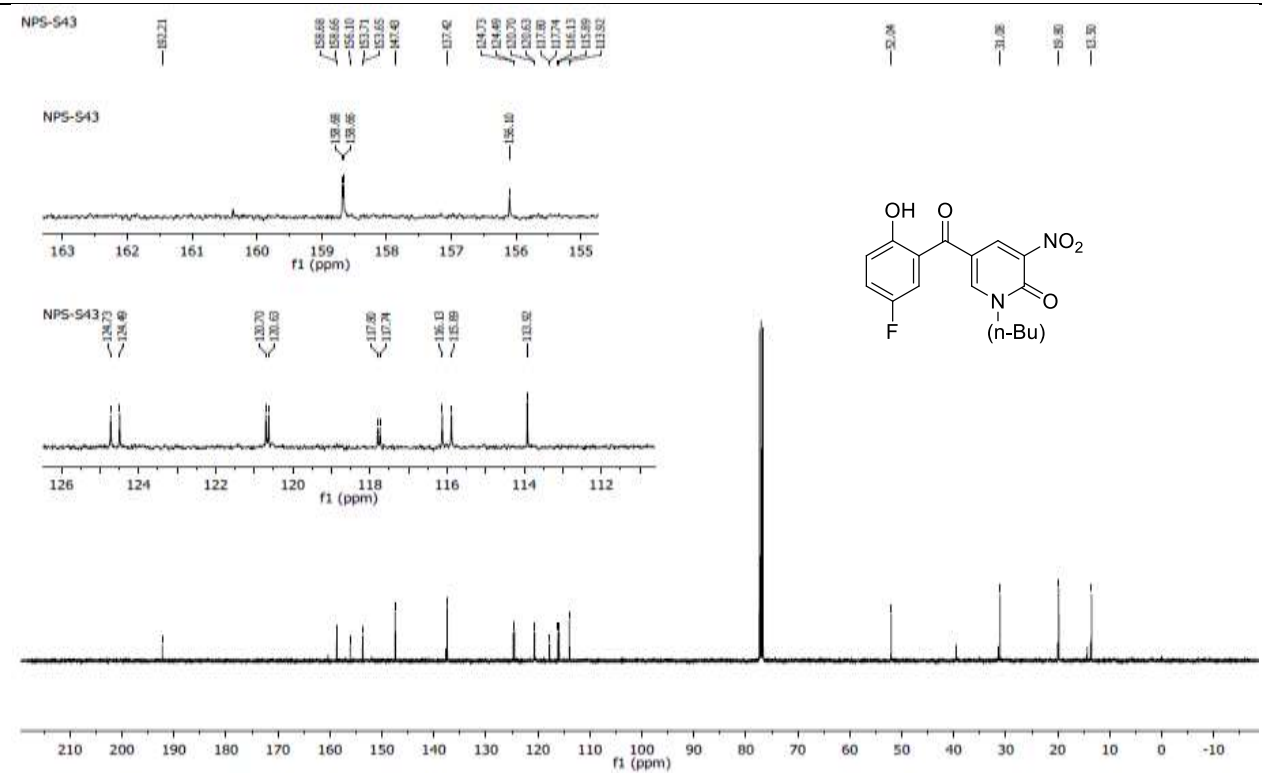
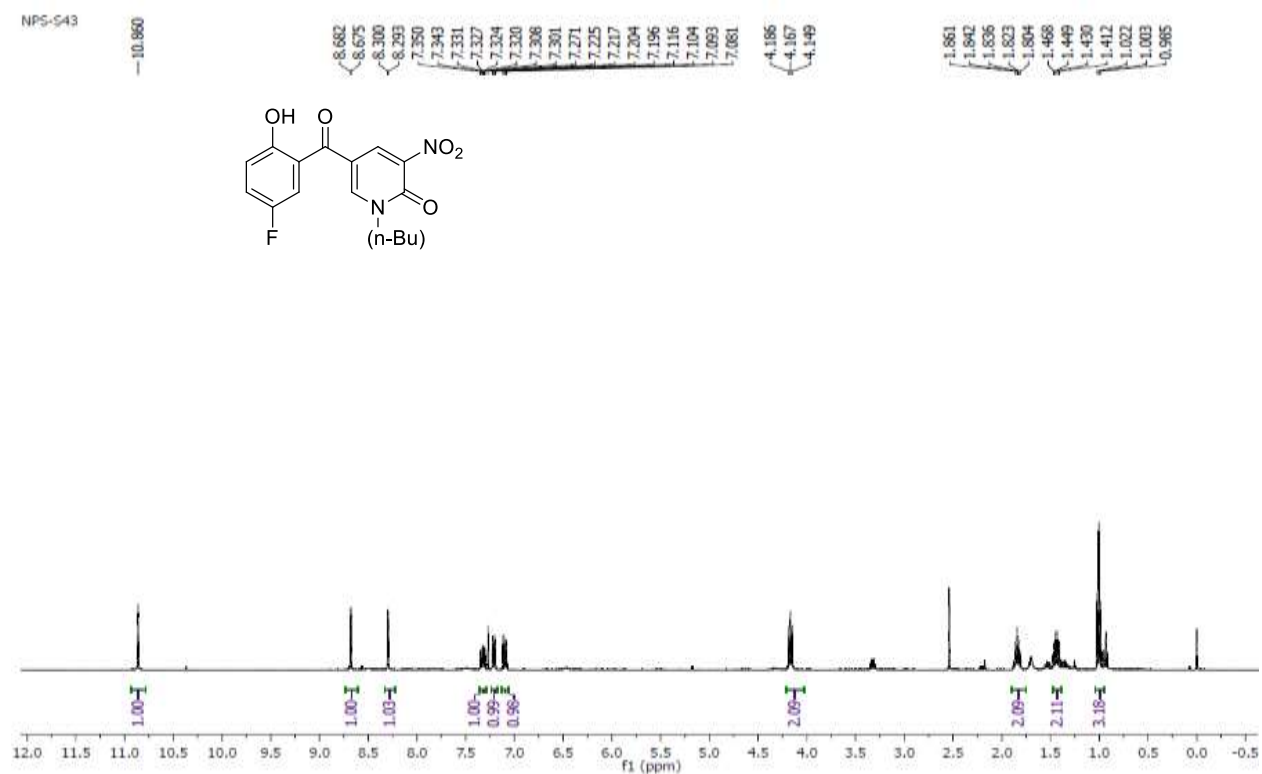
189.11
154.86
154.07
150.60
137.51
137.43
133.15
129.78
129.66
125.94
119.11
114.00

50.77

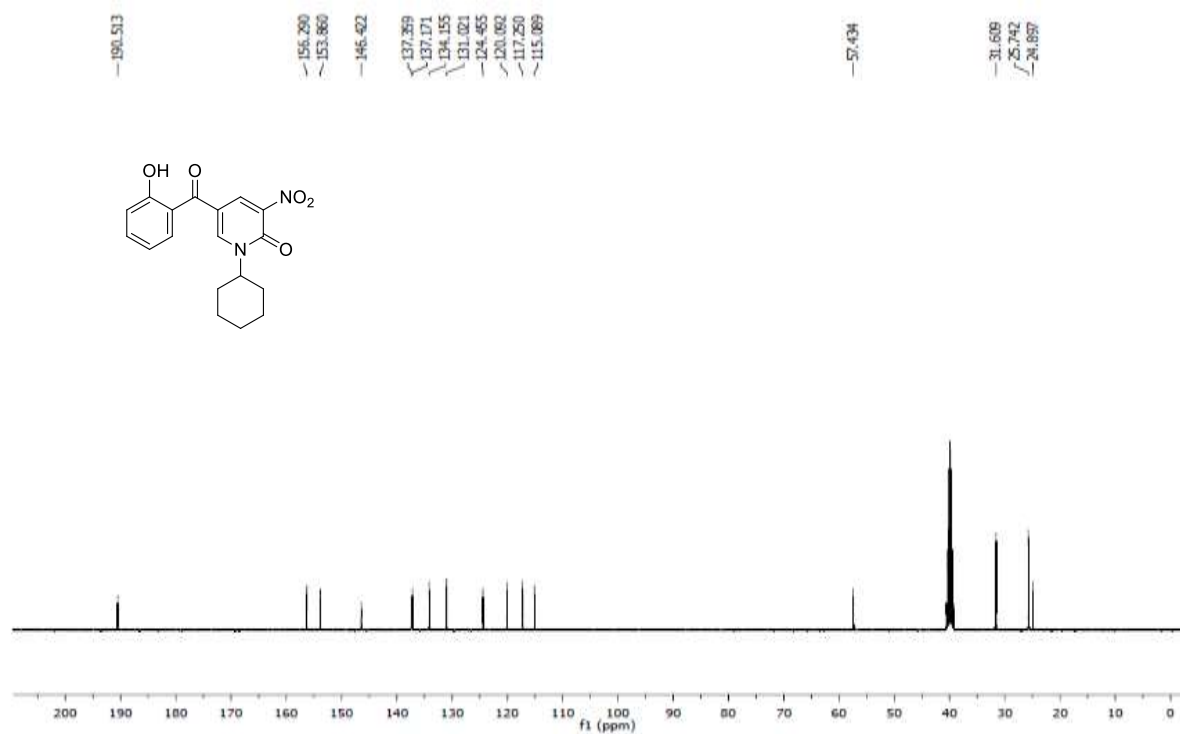
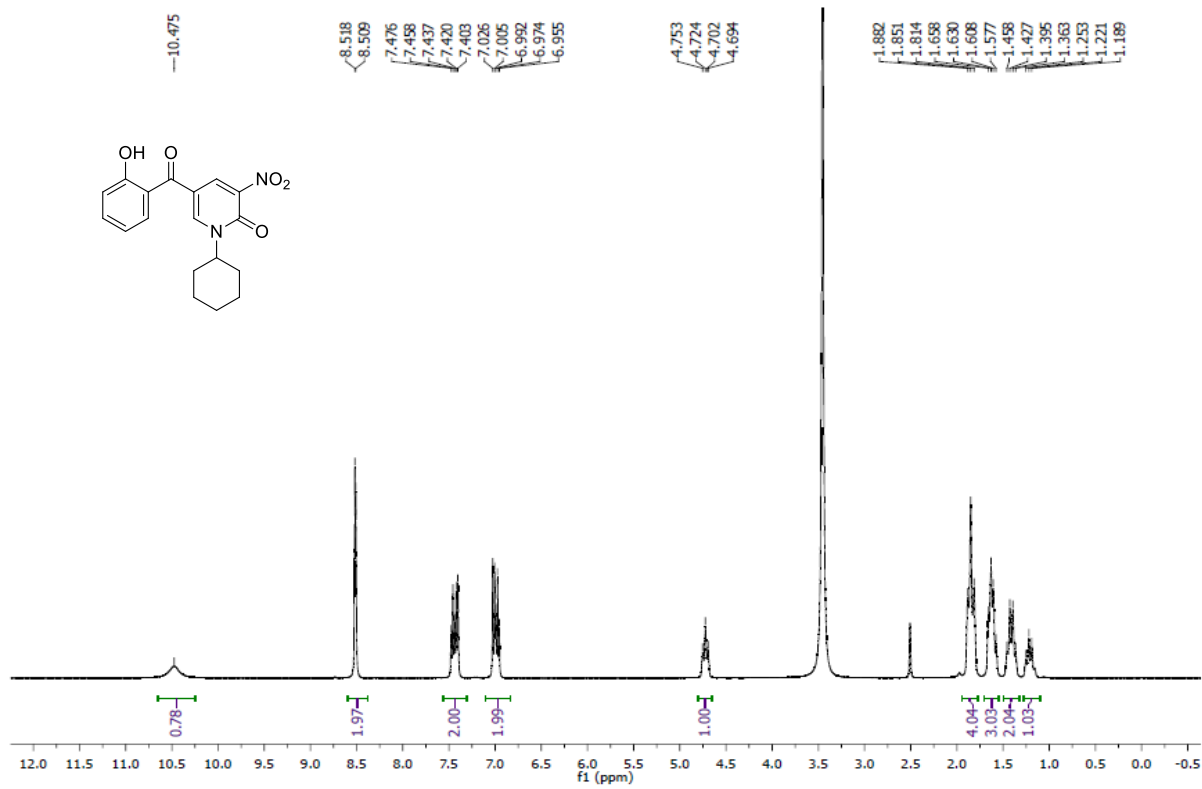
30.94
19.52
13.92



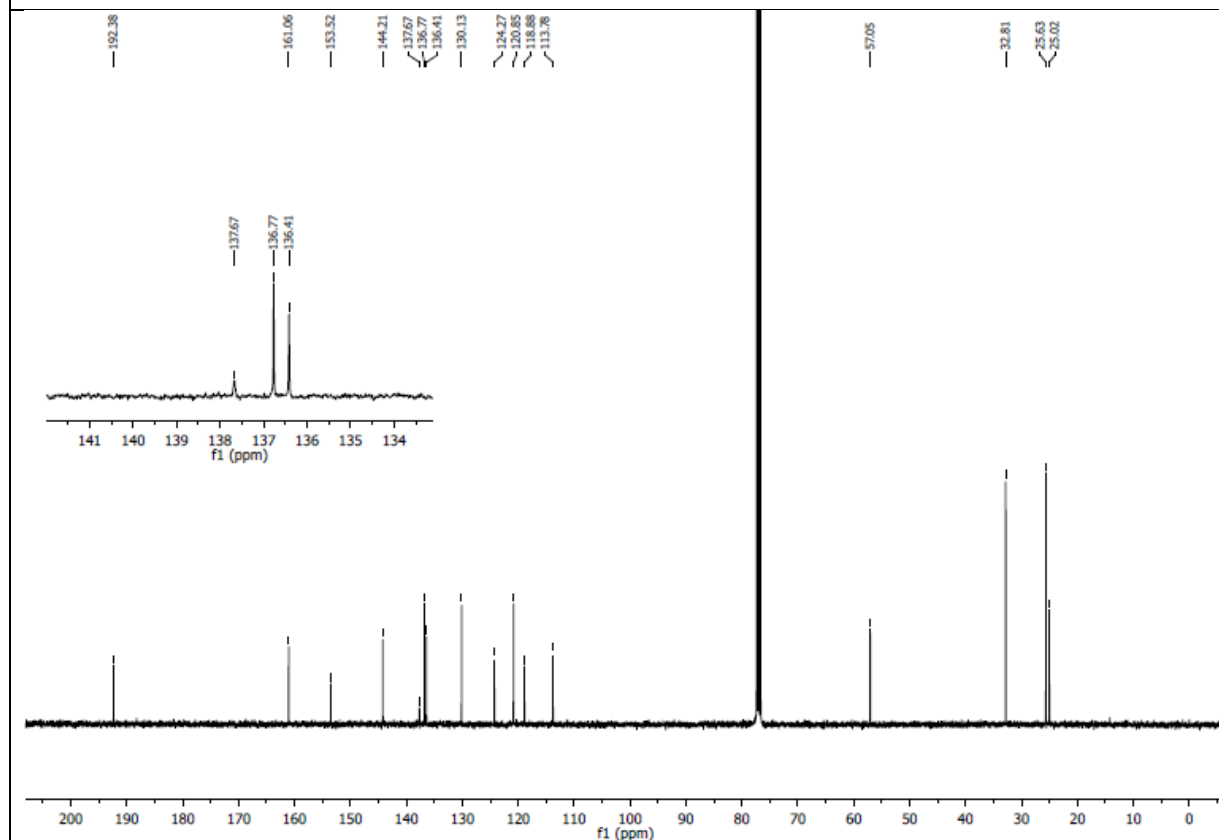
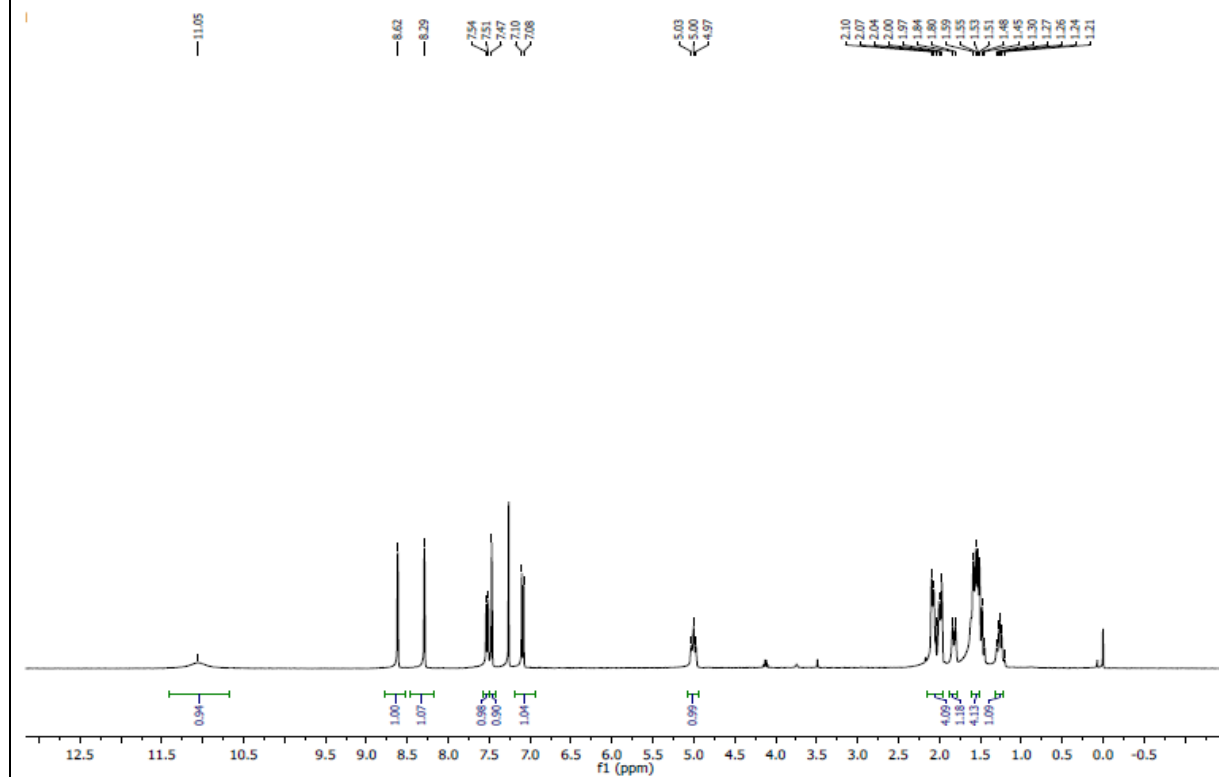
¹H and ¹³C NMR Spectra of Compound (3n)



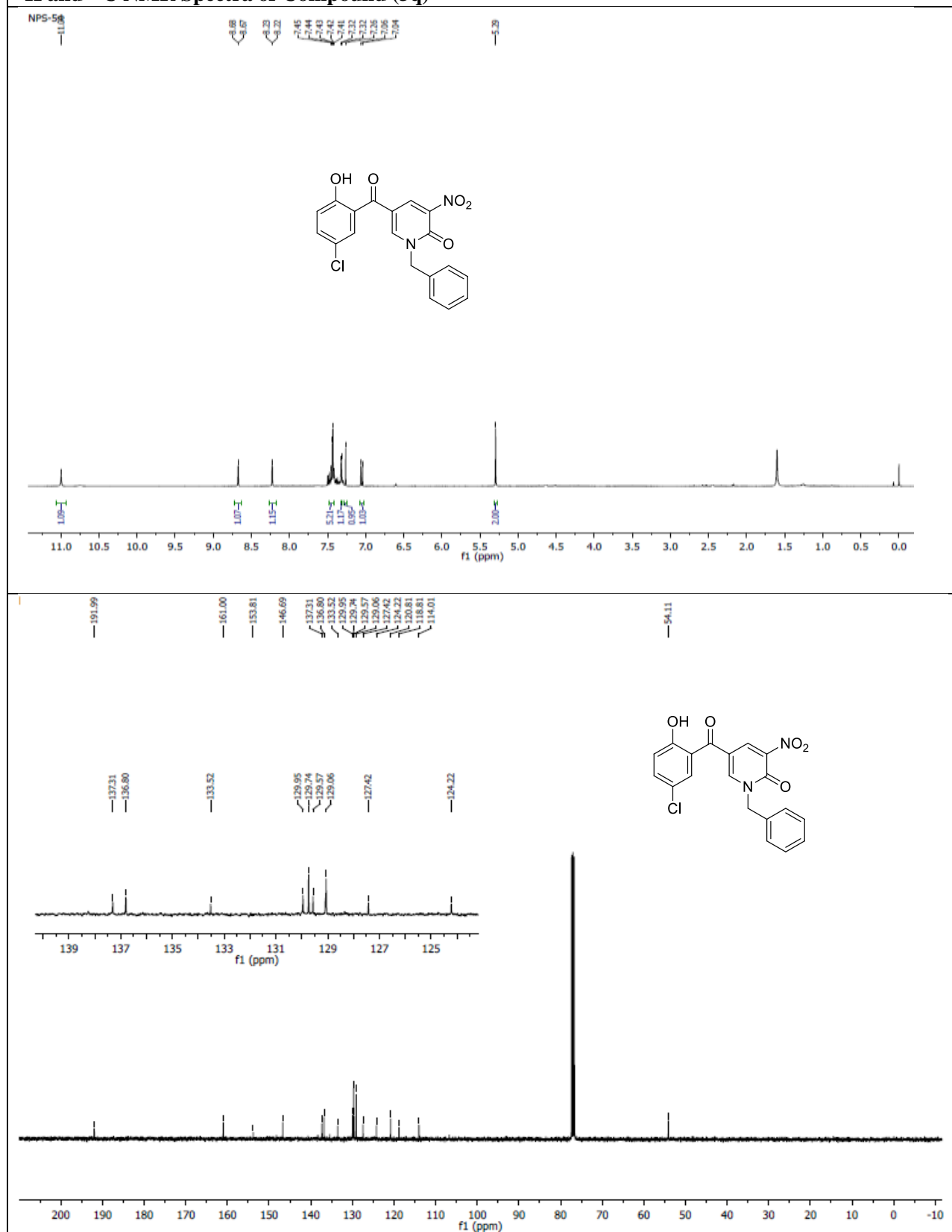
¹H and ¹³C NMR Spectra of Compound (3o)



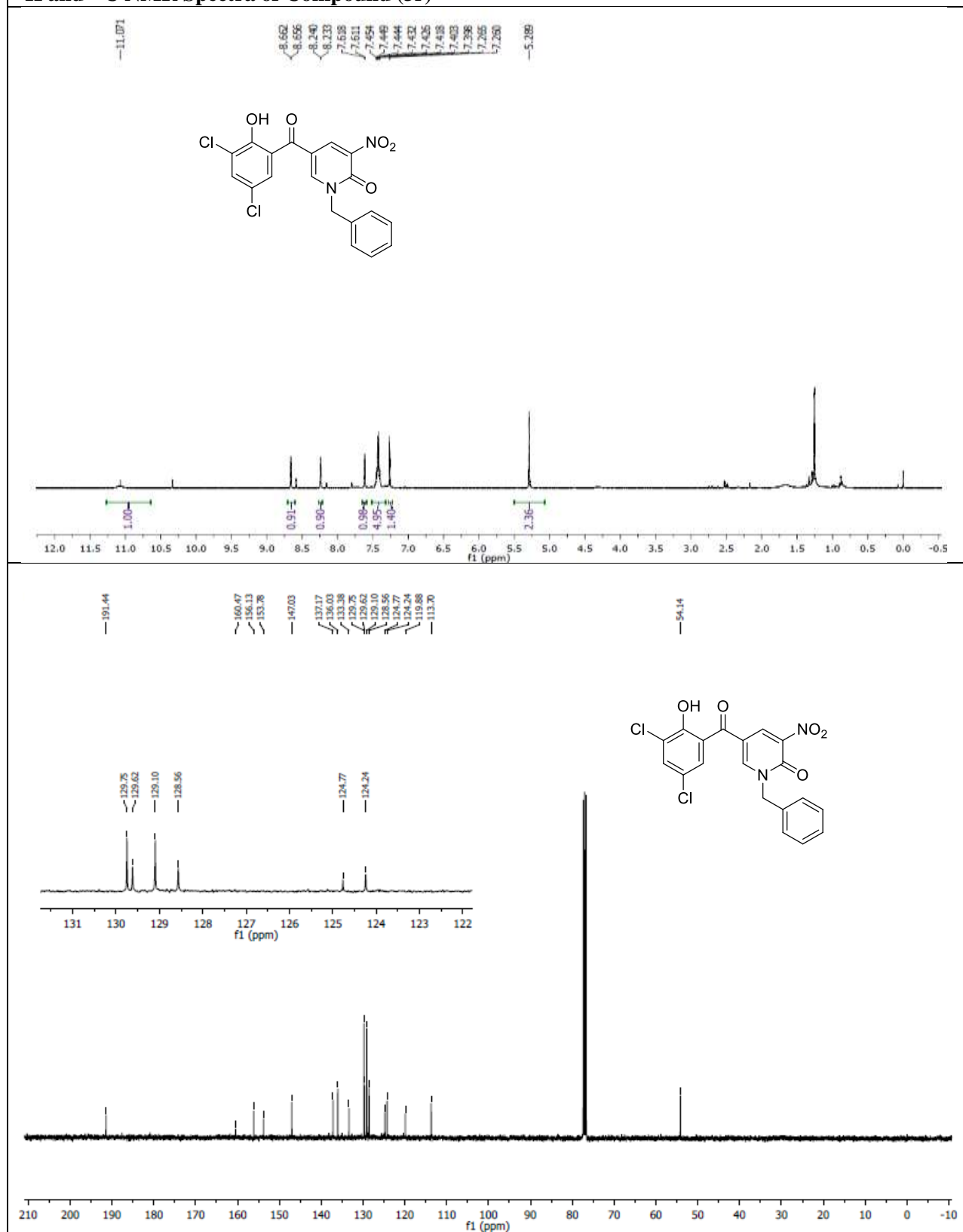
¹H and ¹³C NMR Spectra of Compound (3p)



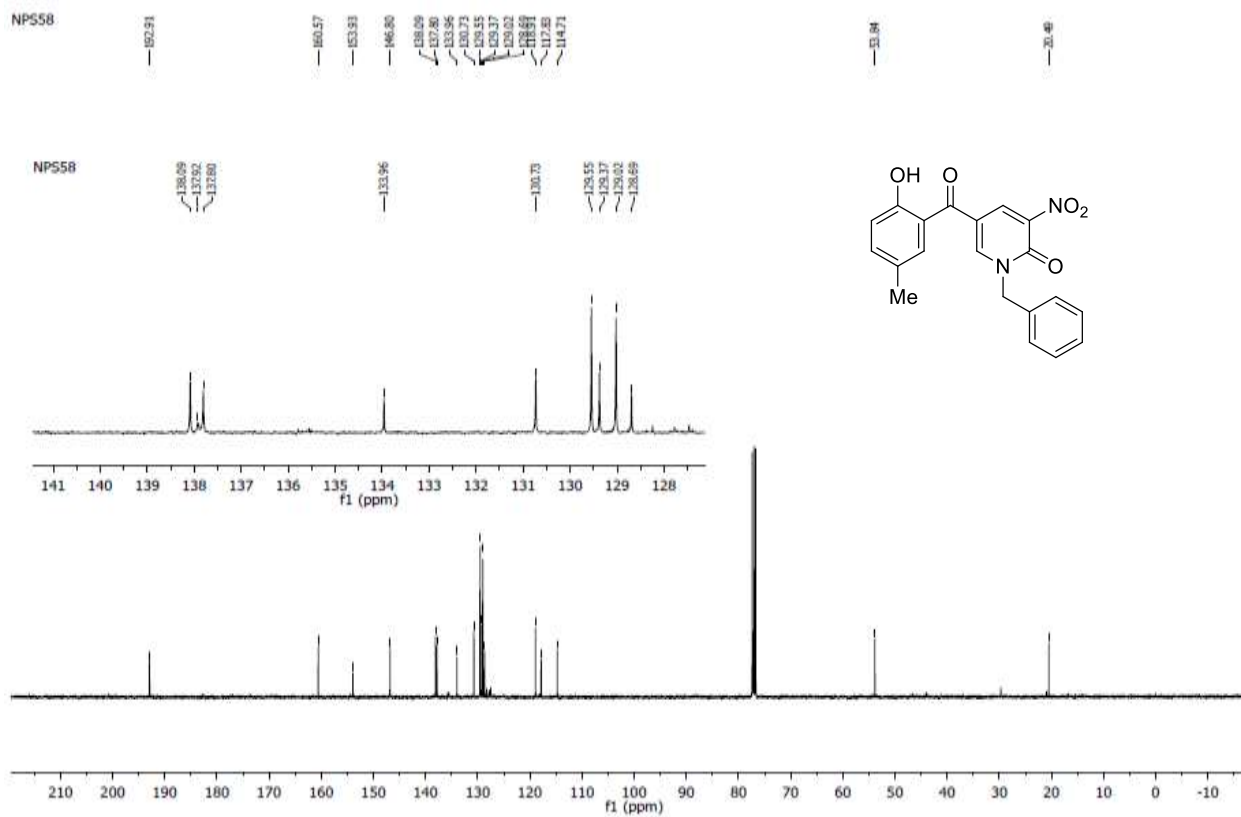
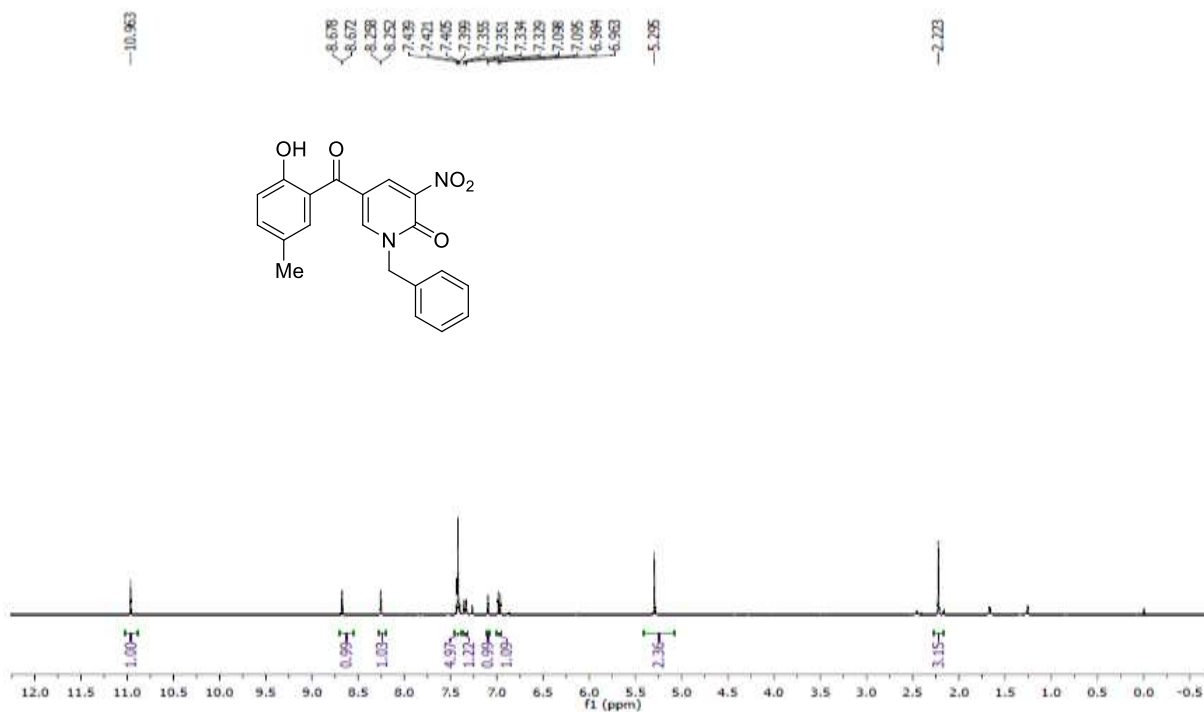
¹H and ¹³C NMR Spectra of Compound (3q)



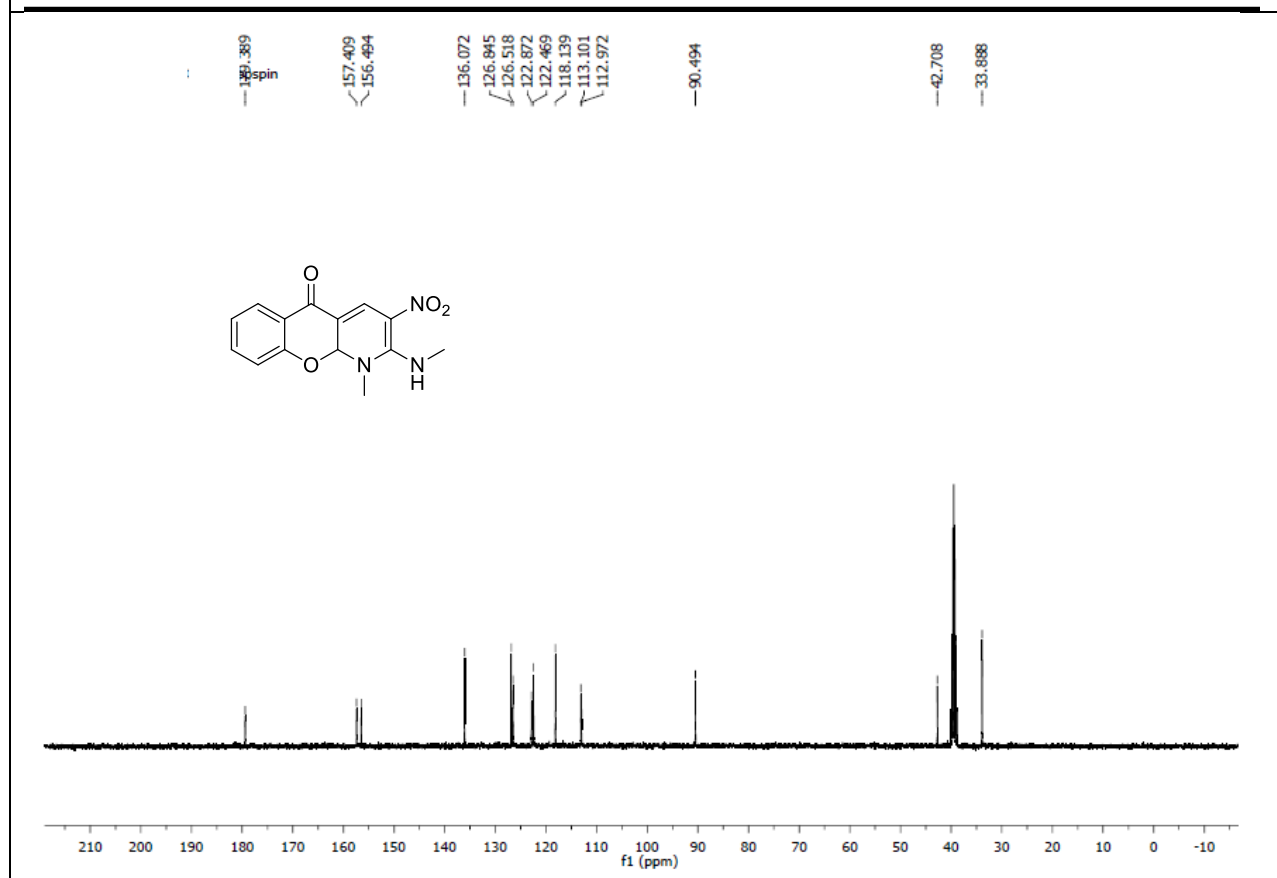
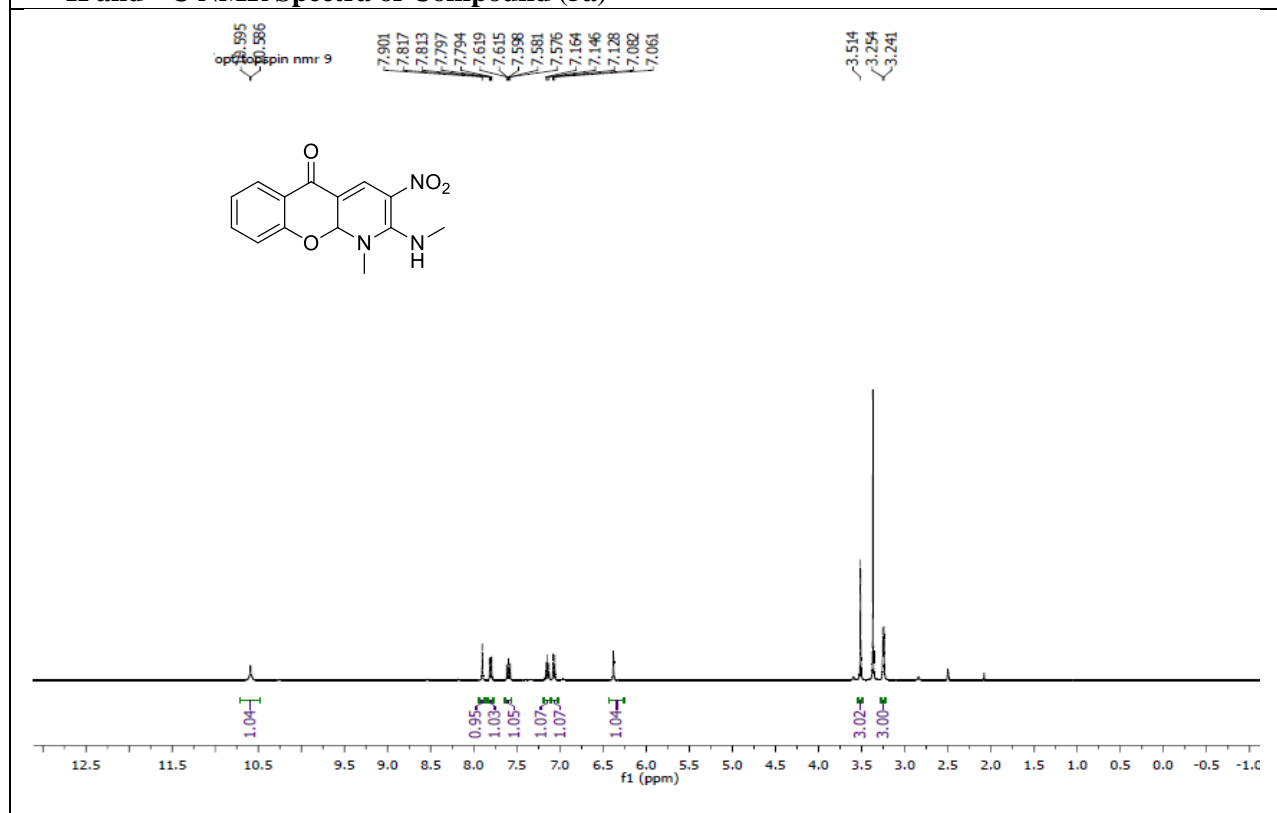
¹H and ¹³C NMR Spectra of Compound (3r)



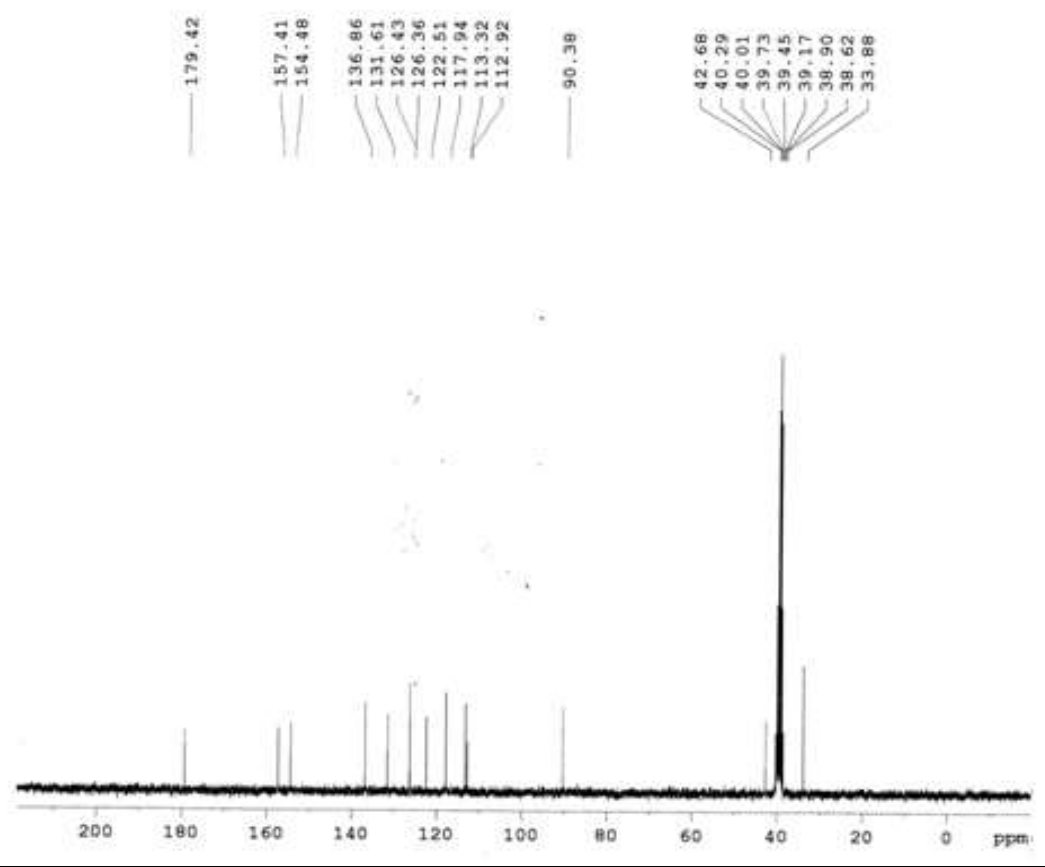
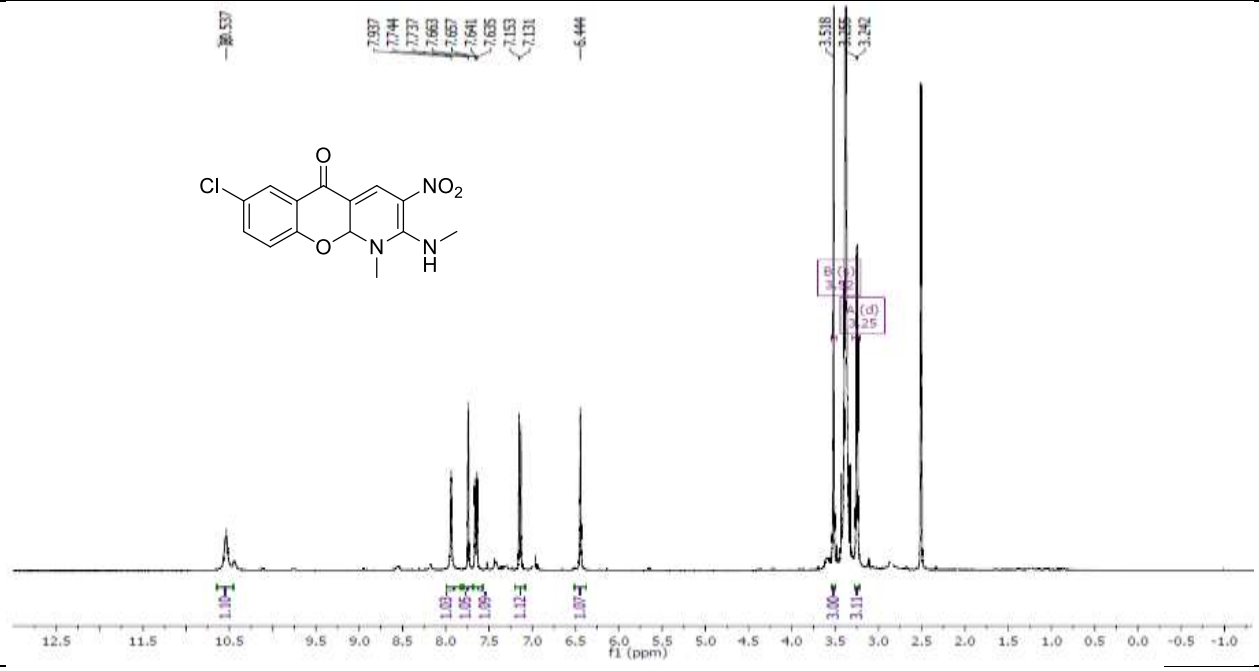
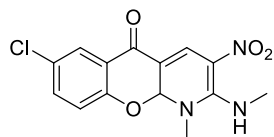
¹H and ¹³C NMR Spectra of Compound (3s)



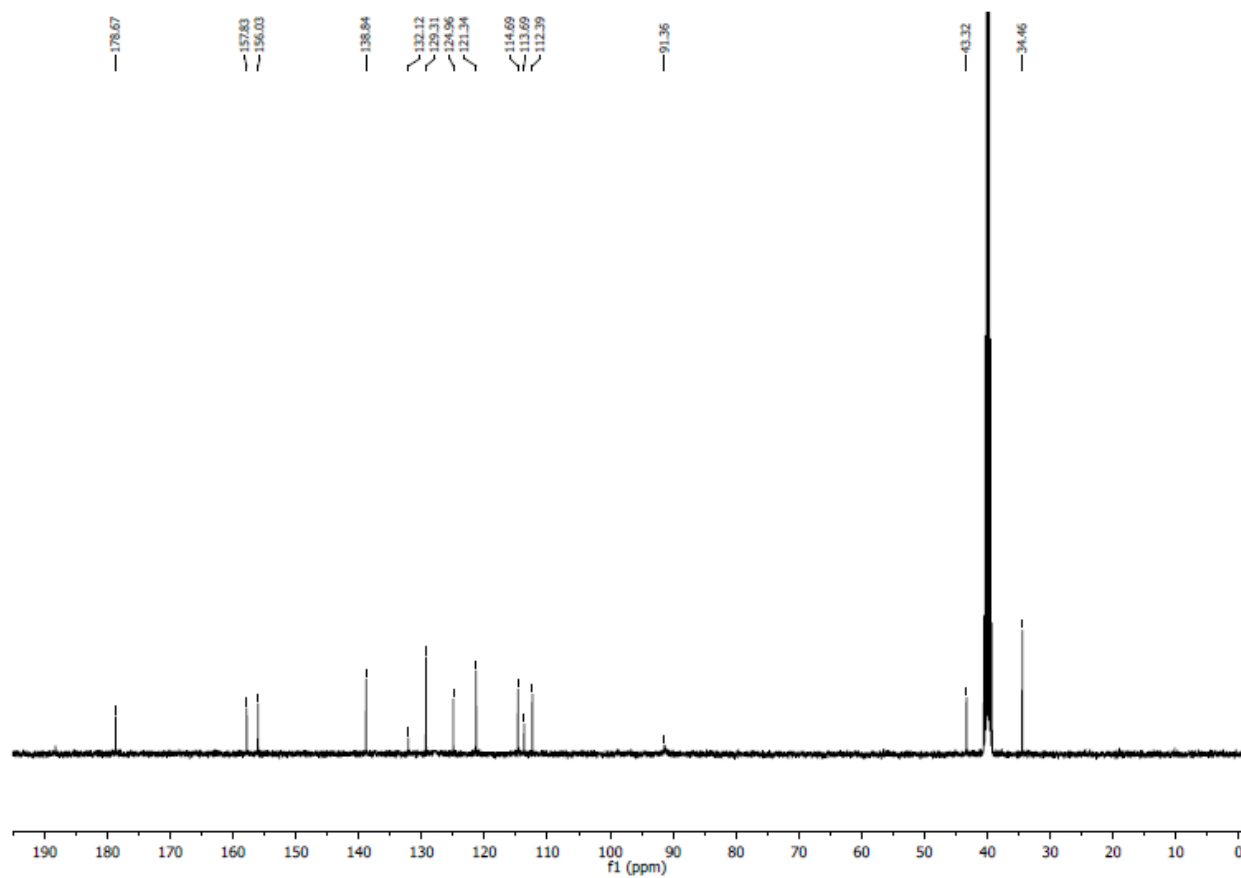
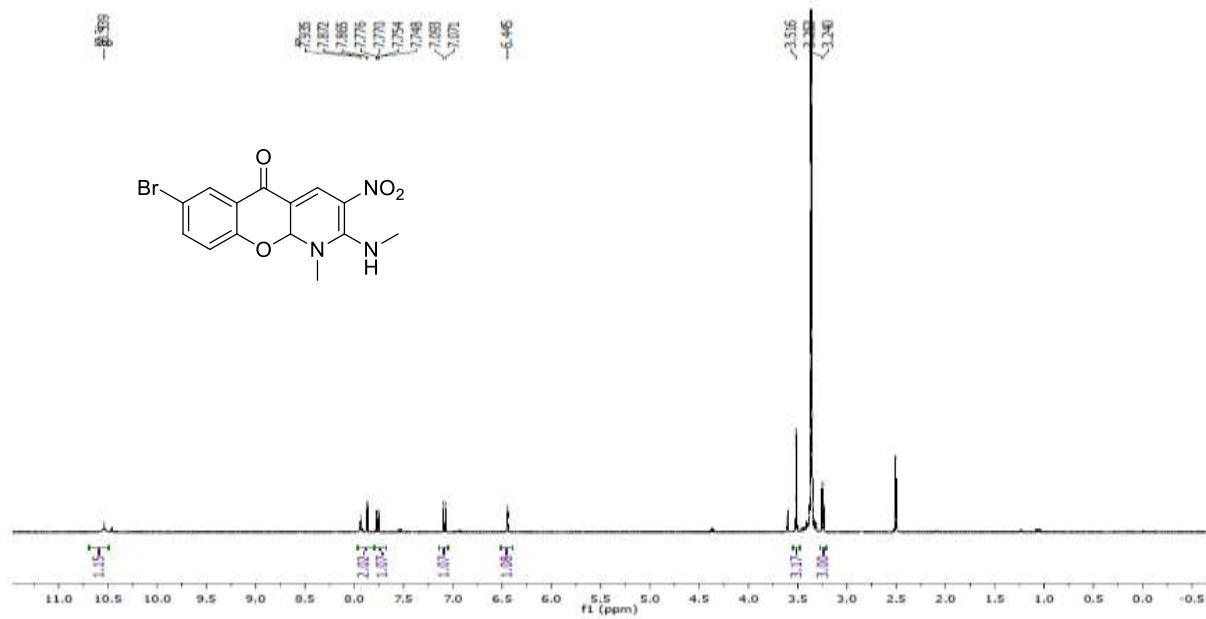
¹H and ¹³C NMR Spectra of Compound (5a)



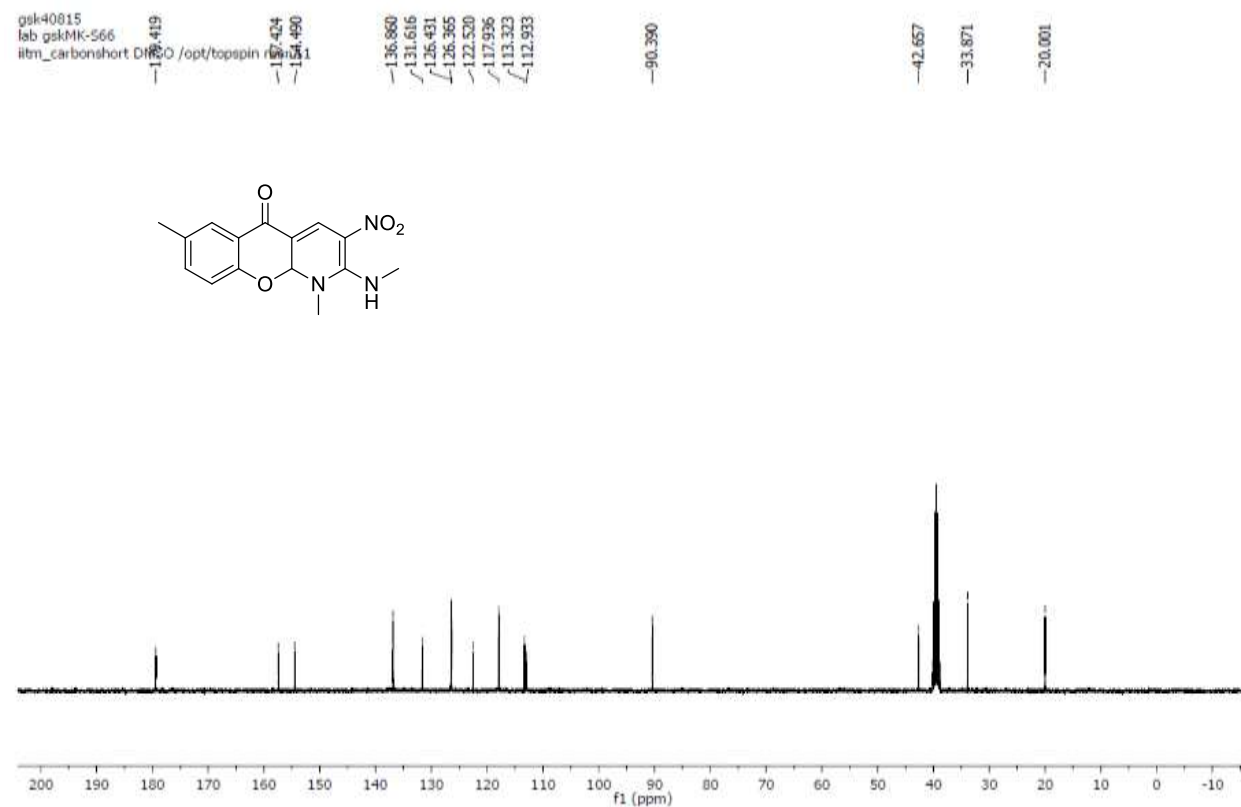
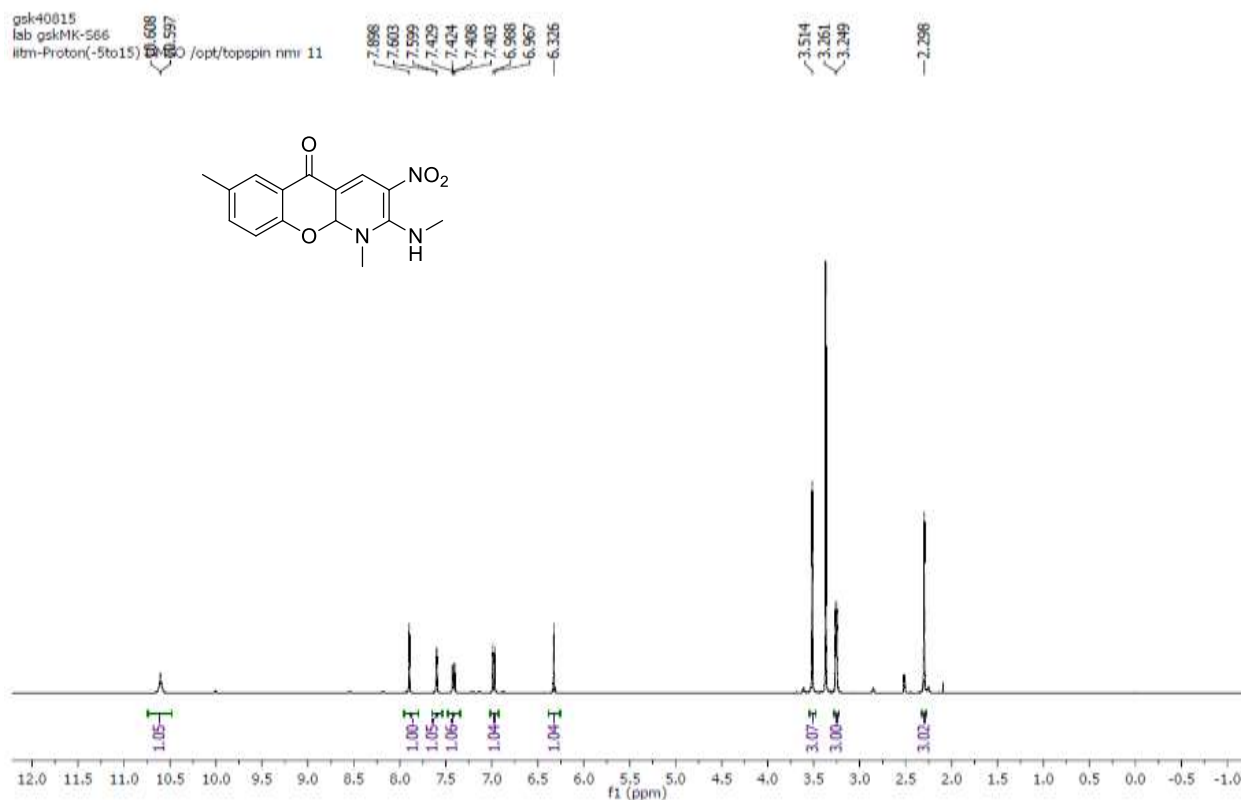
¹H and ¹³C NMR Spectra of Compound (5b)



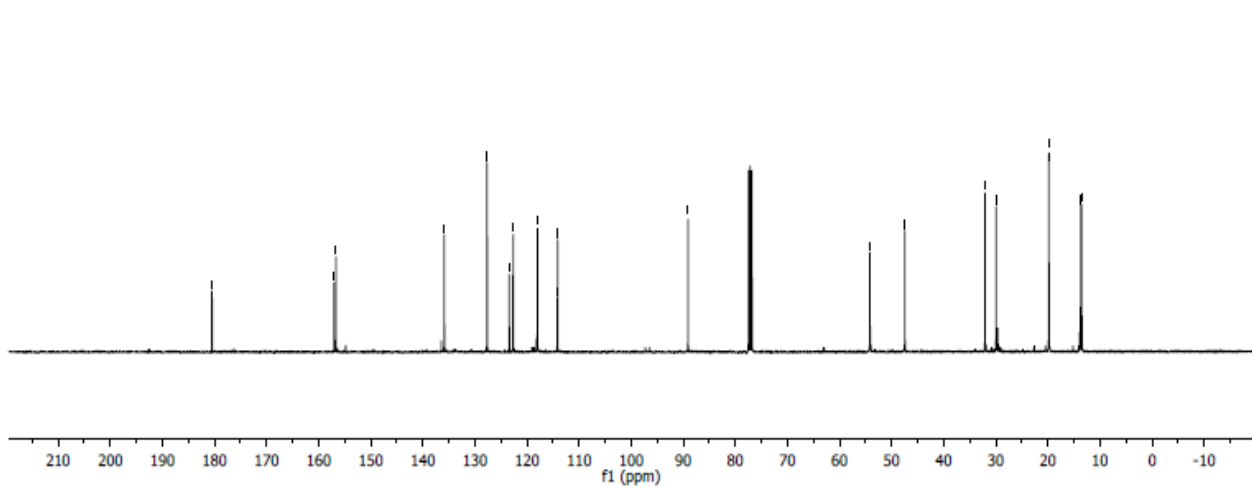
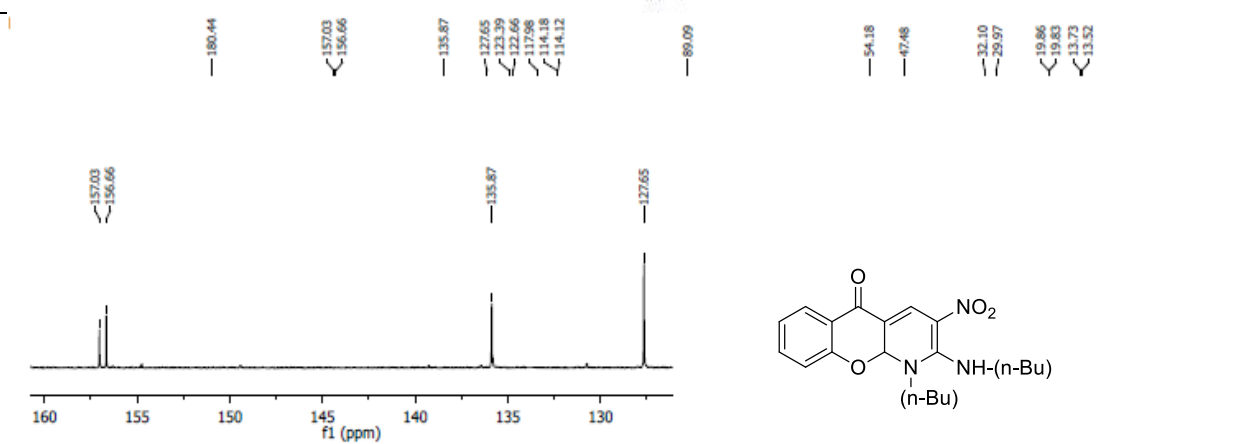
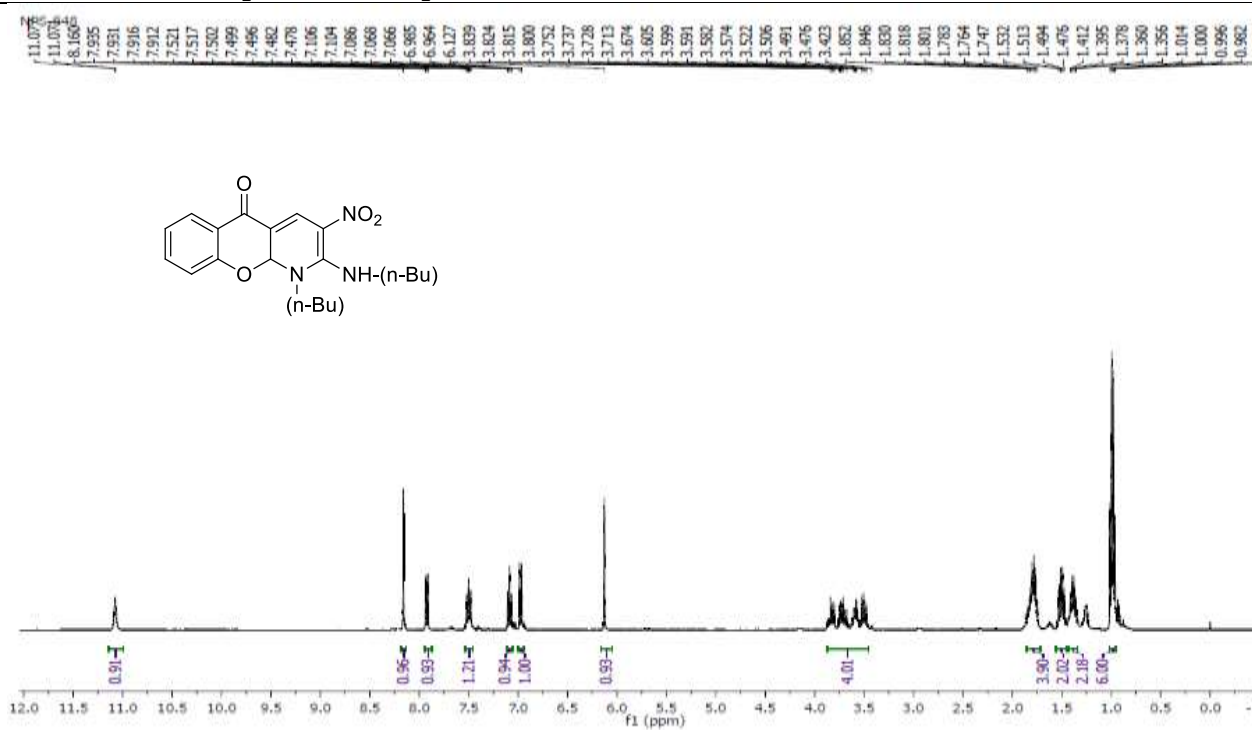
¹H NMR Spectra of Compound (5c)



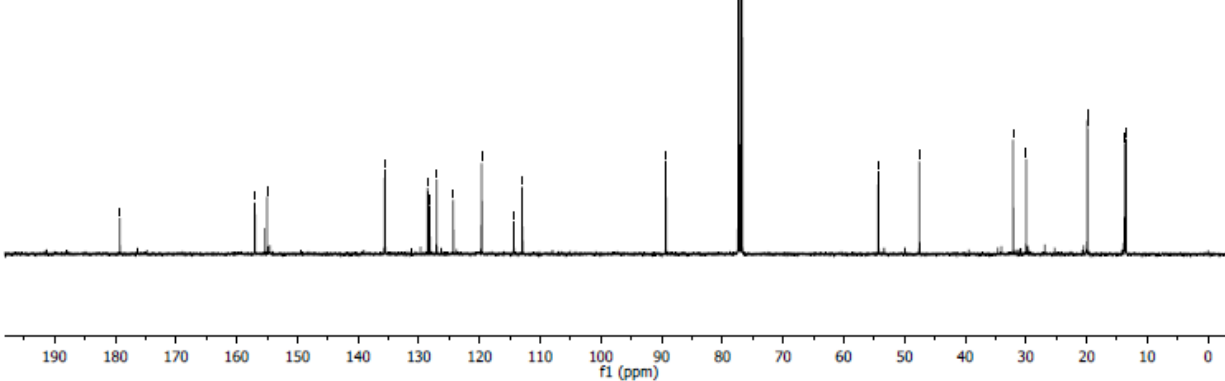
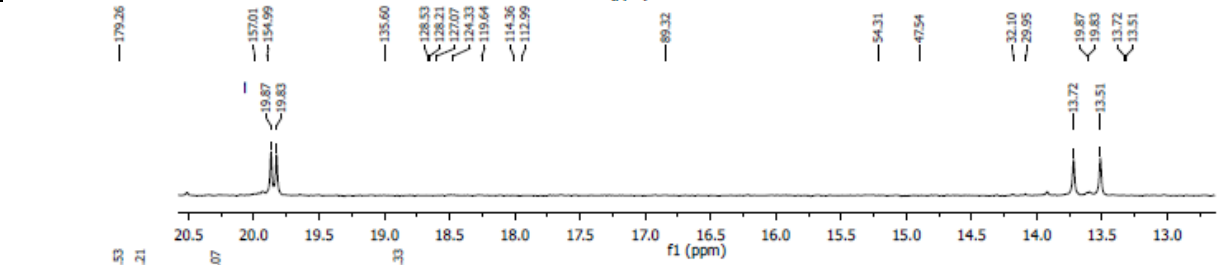
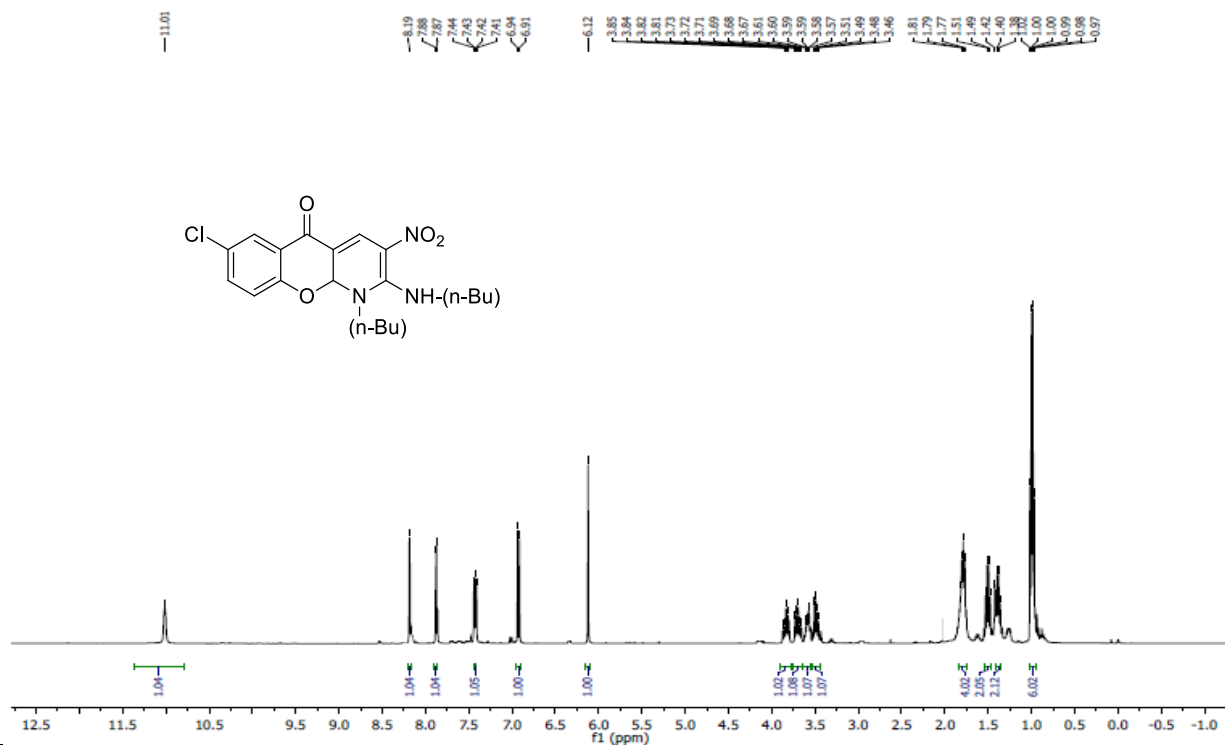
¹H and ¹³C NMR Spectra of Compound (5d)



¹H and ¹³C NMR Spectra of Compound (5e)



¹H and ¹³C NMR Spectra of Compound (5f)



¹H and ¹³C NMR Spectra of Compound (5g)

NPS-553

-11.036

-8.247

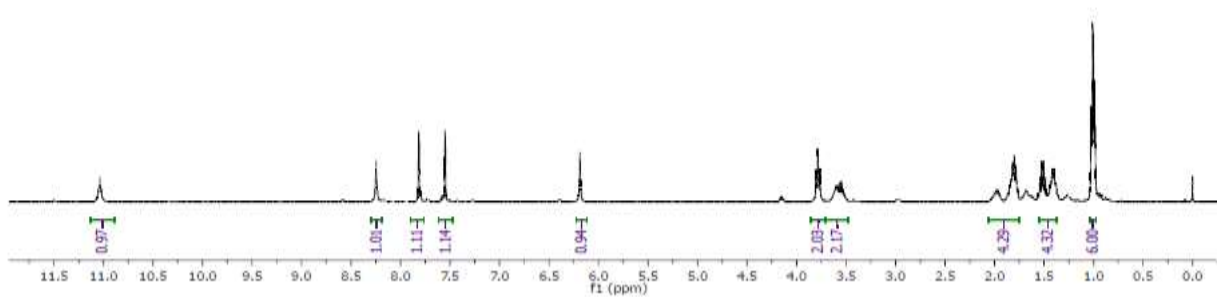
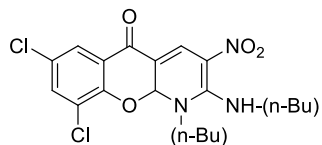
-7.818

-7.812

-7.552

-7.273

-6.187



178.41

156.95

155.36

135.17

129.41

127.94

125.82

125.24

124.17

114.39

111.77

90.44

54.63

47.61

32.14

30.40

19.95

19.87

13.71

13.51

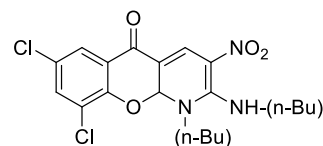
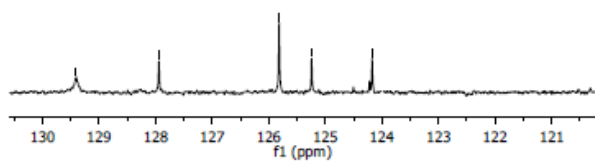
129.41

127.94

125.82

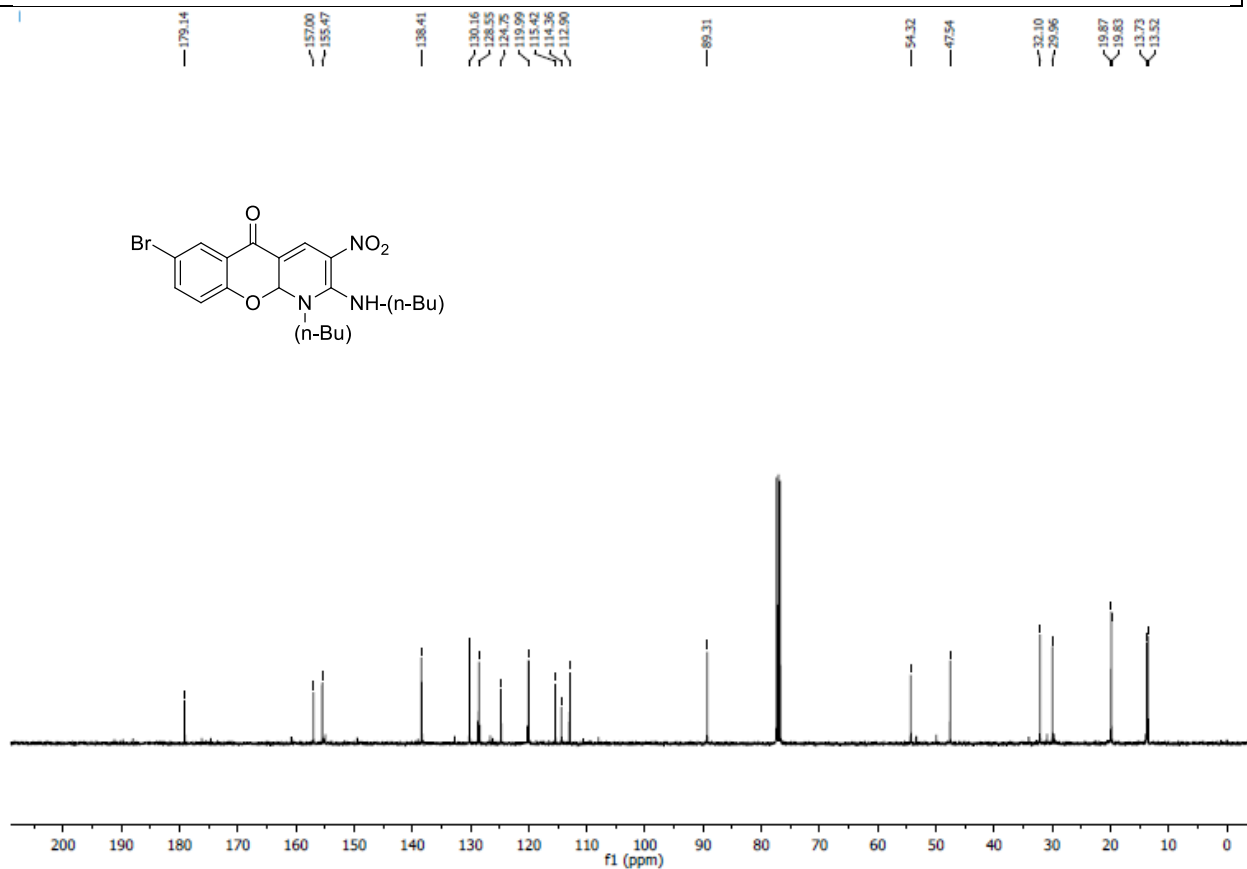
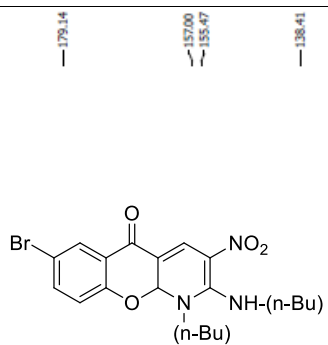
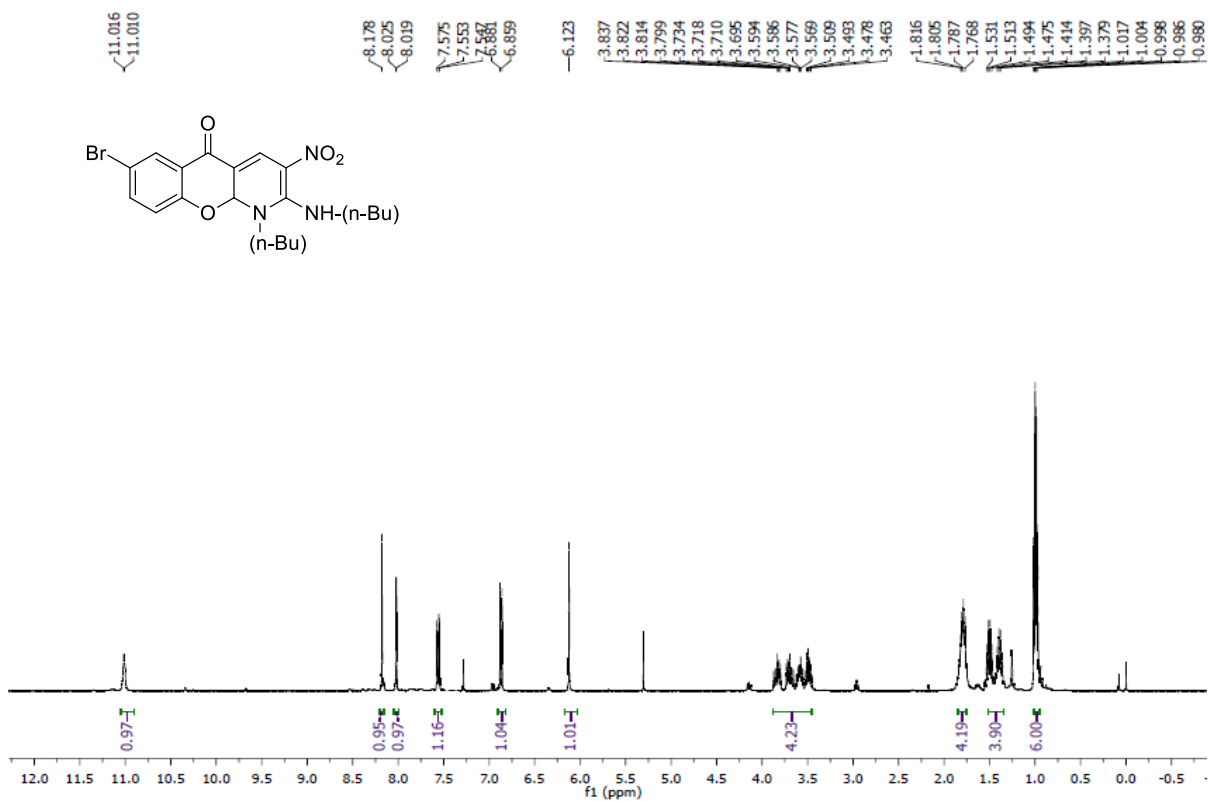
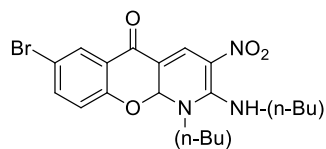
125.24

124.17

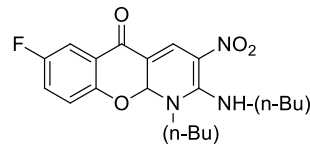
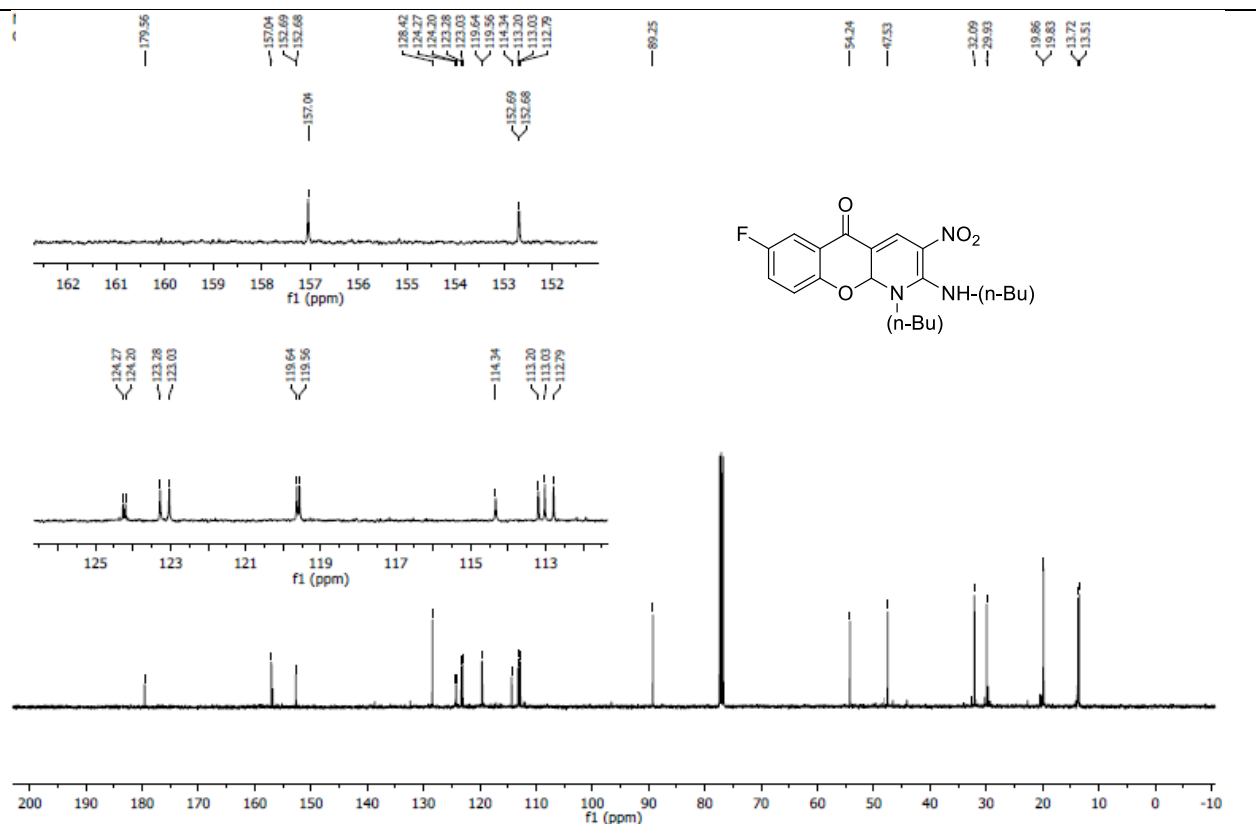
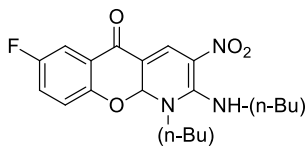
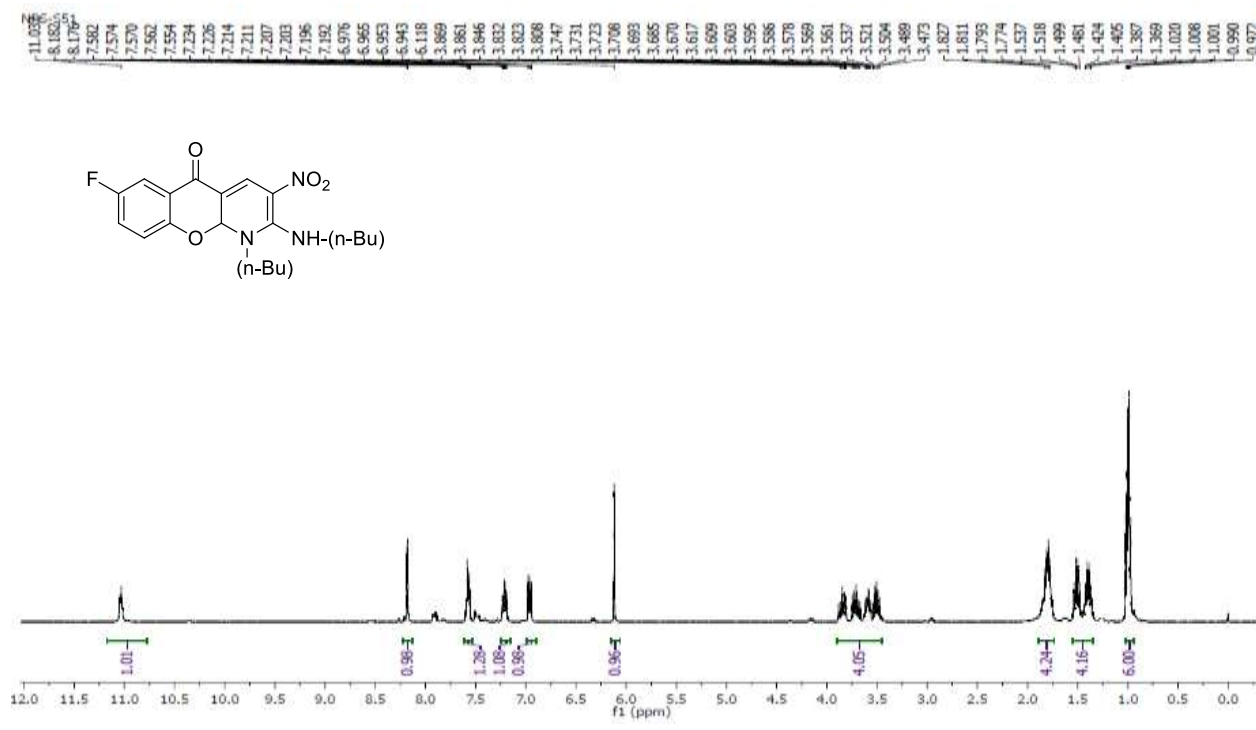


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

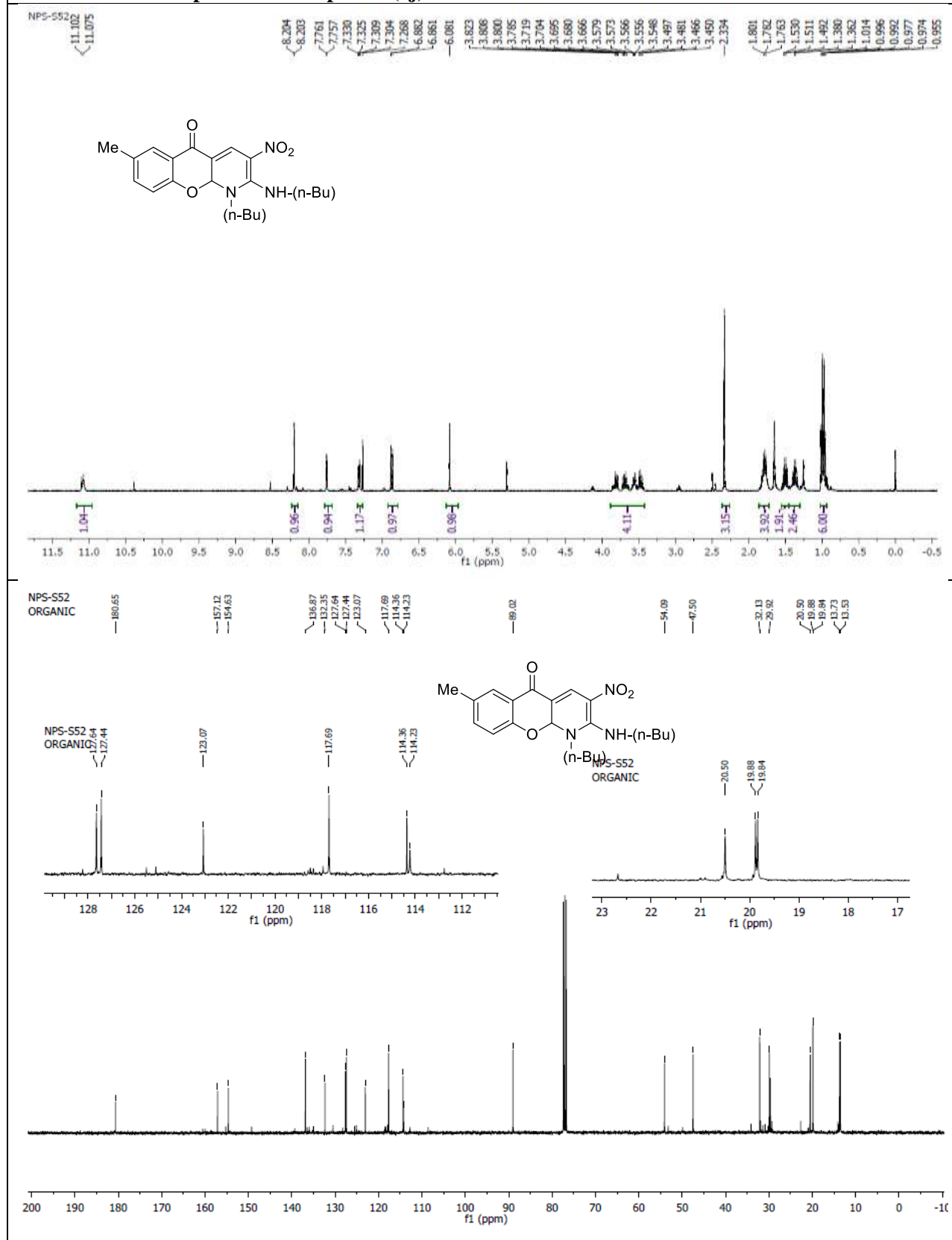
¹H and ¹³C NMR Spectra of Compound (5h)



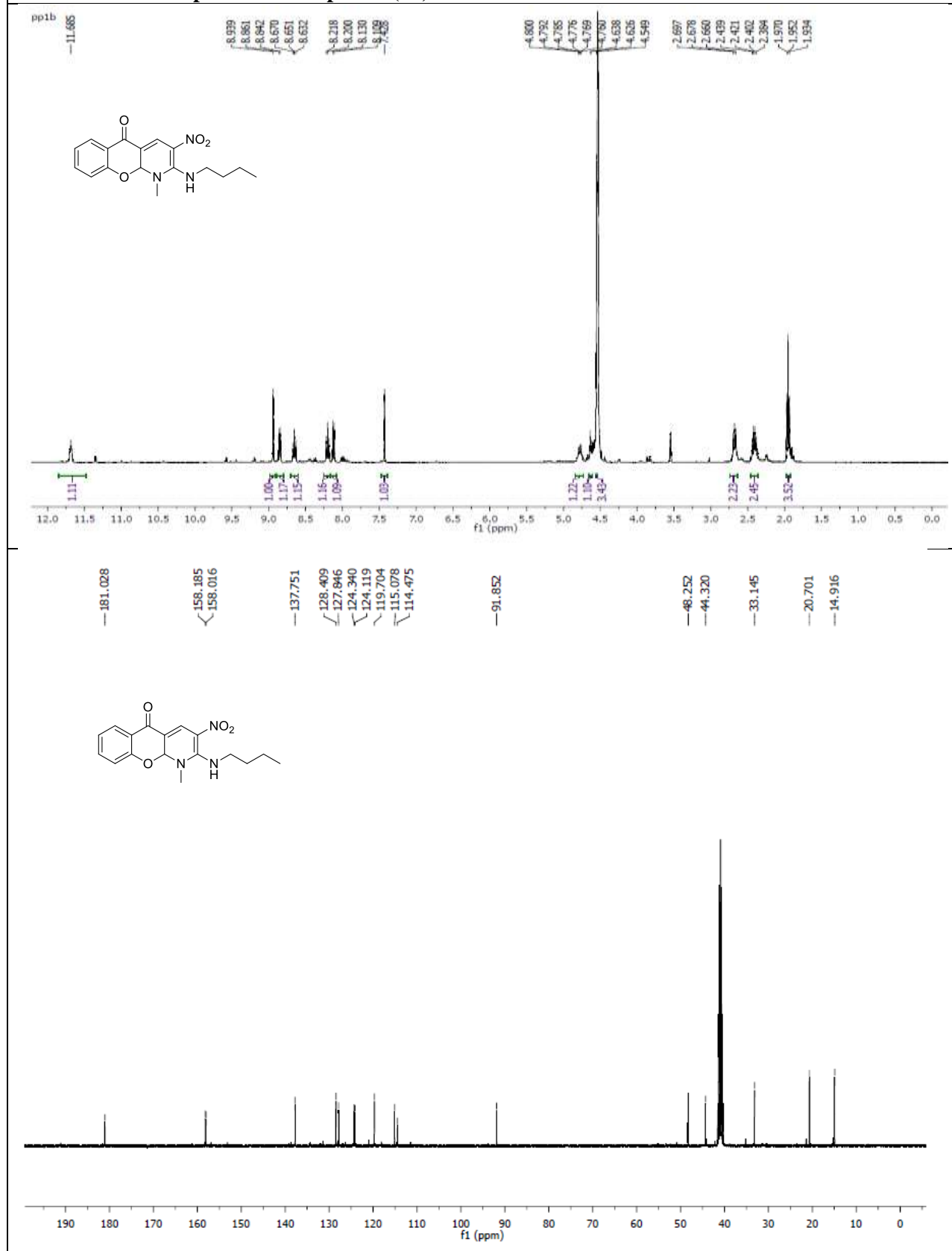
¹H and ¹³C NMR Spectra of Compound (5i)



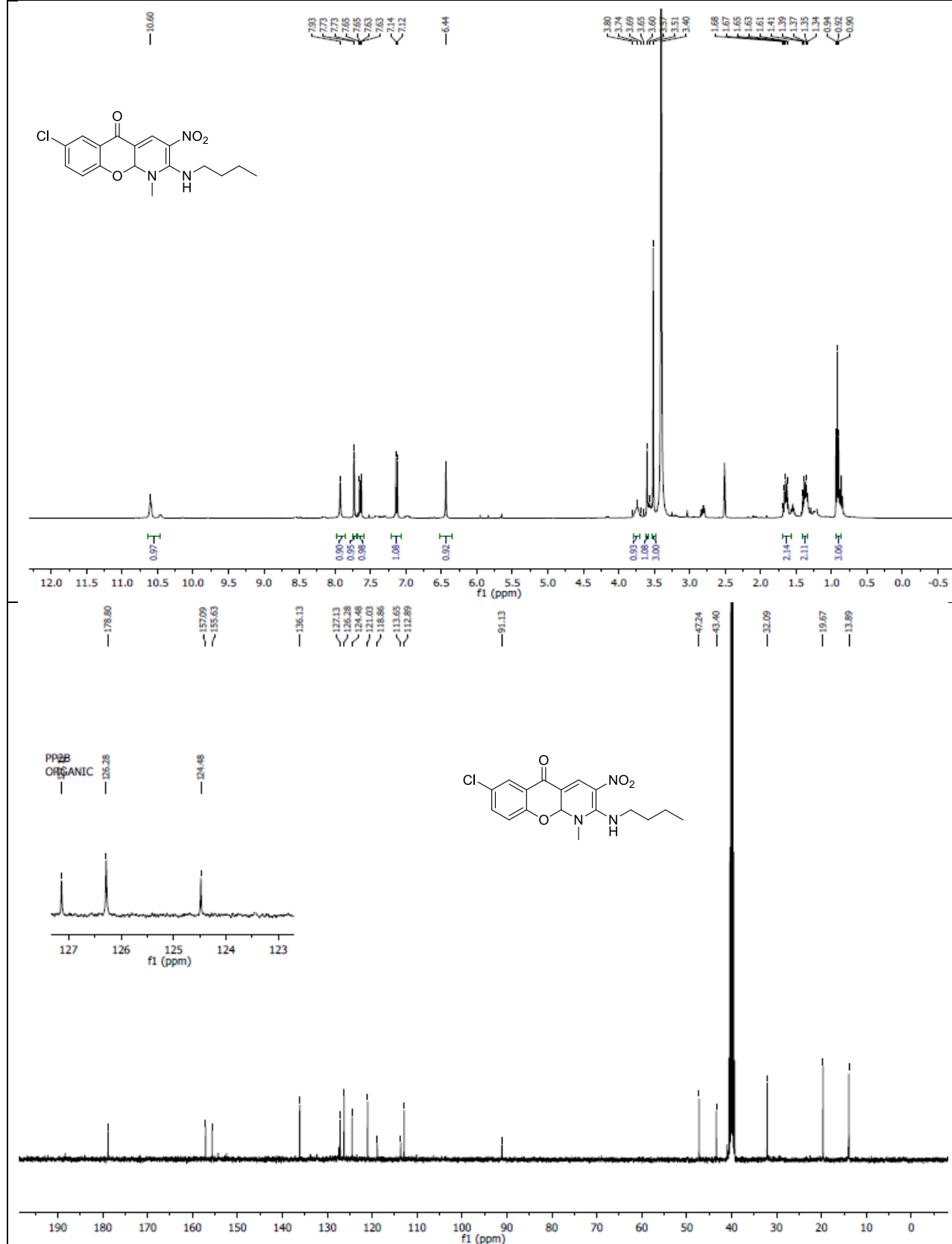
¹H and ¹³C NMR Spectra of Compound (5j)



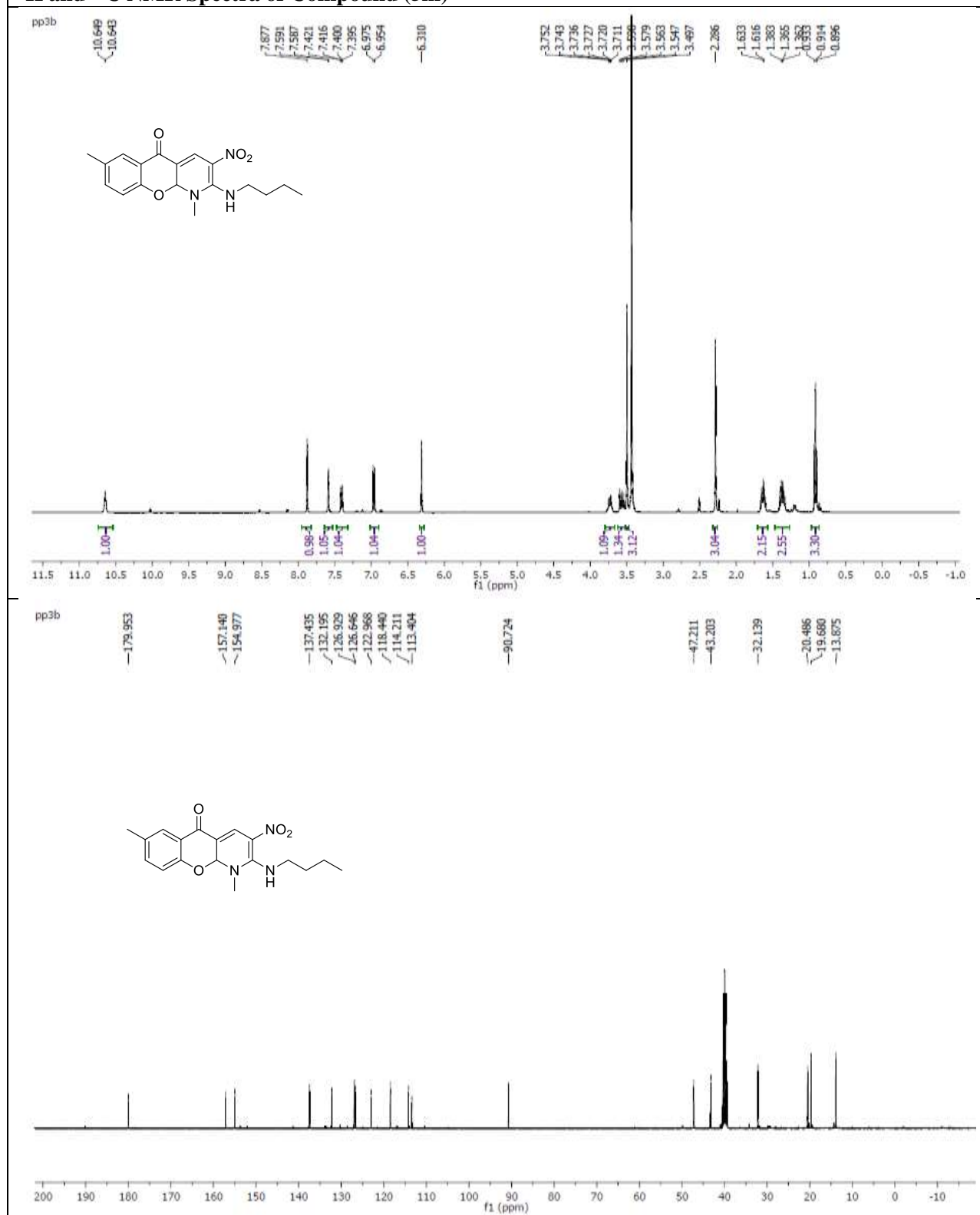
¹H and ¹³C NMR Spectra of Compound (5k)



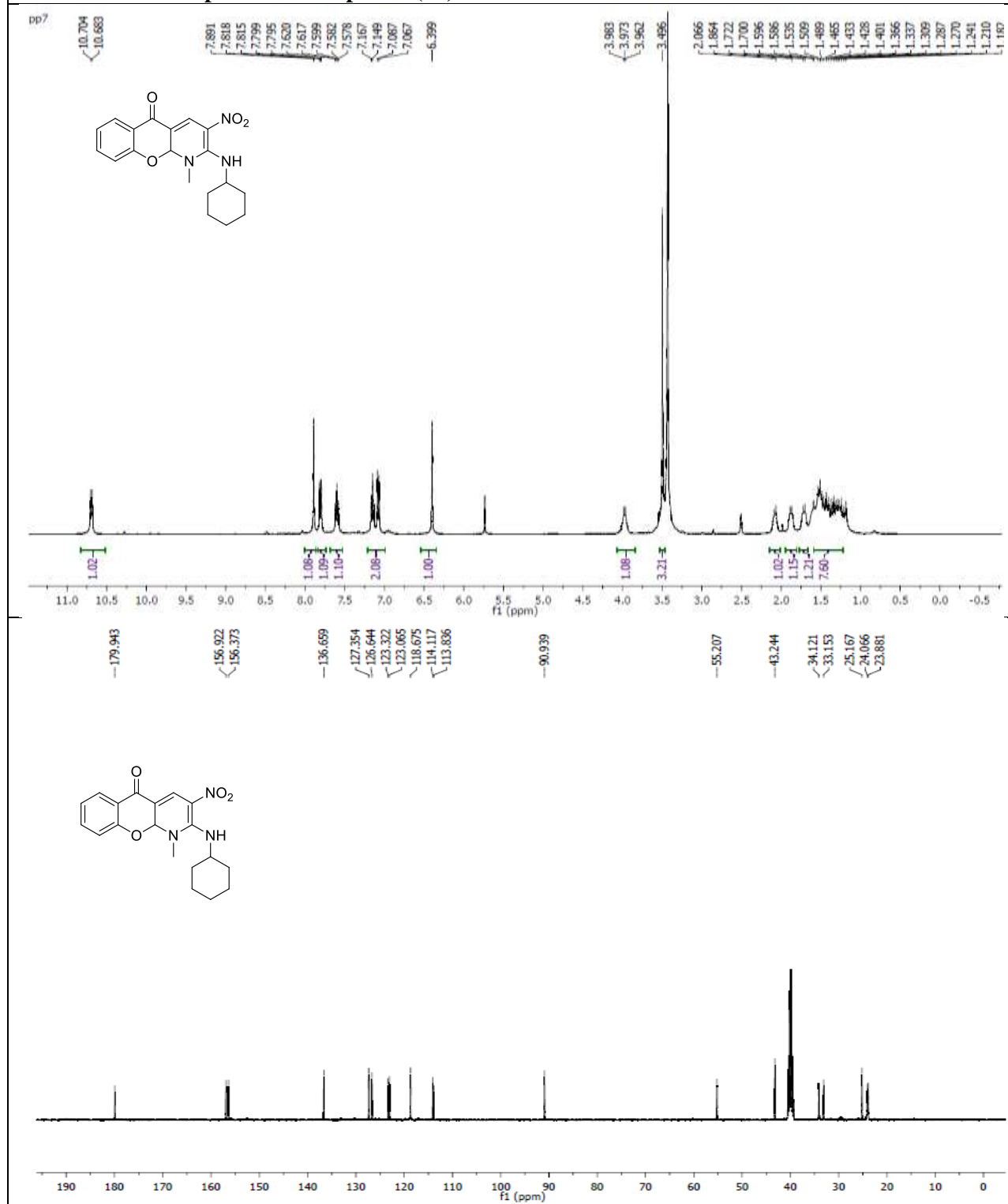
¹H and ¹³C NMR Spectra of Compound (5l)



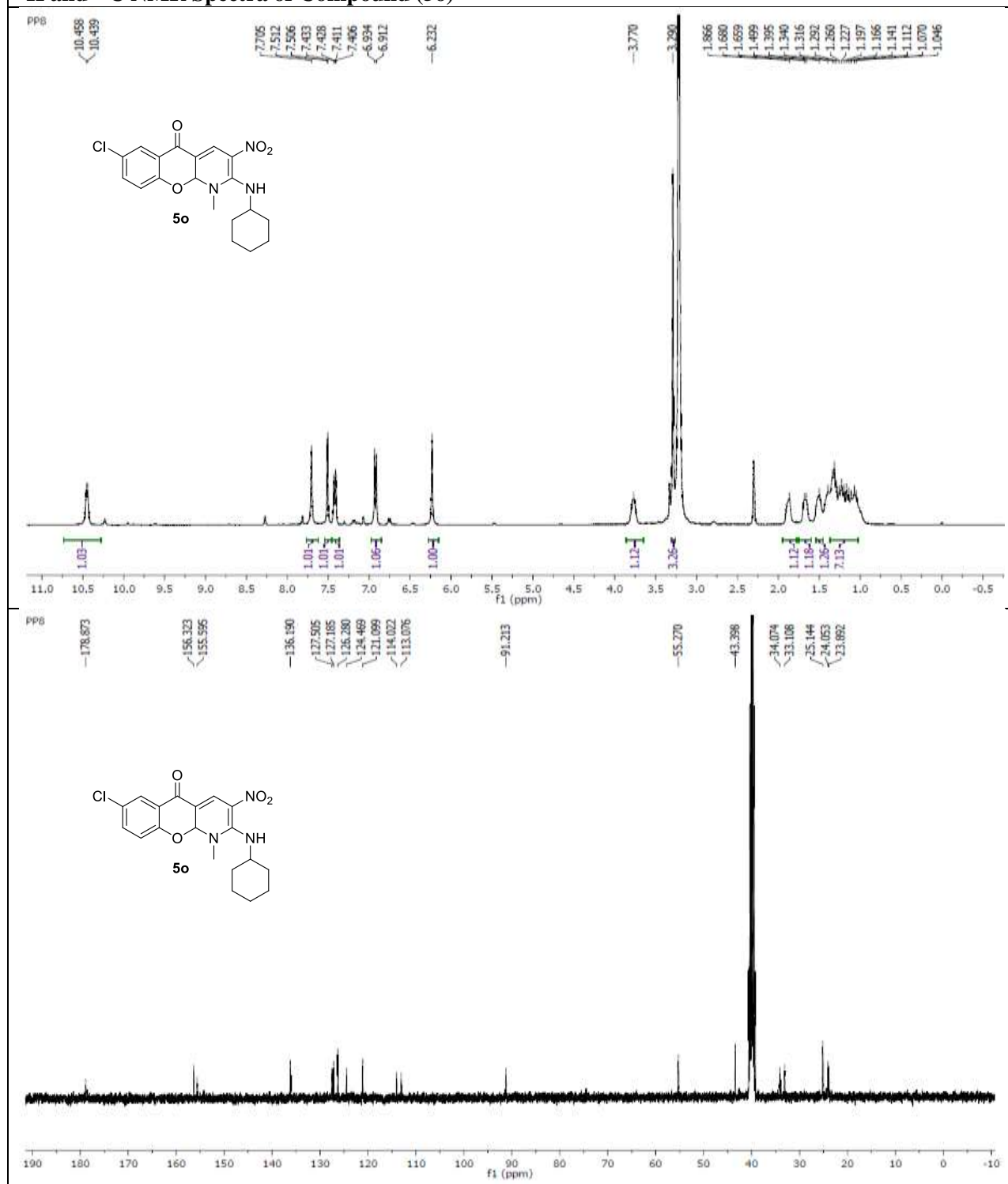
¹H and ¹³C NMR Spectra of Compound (5m)



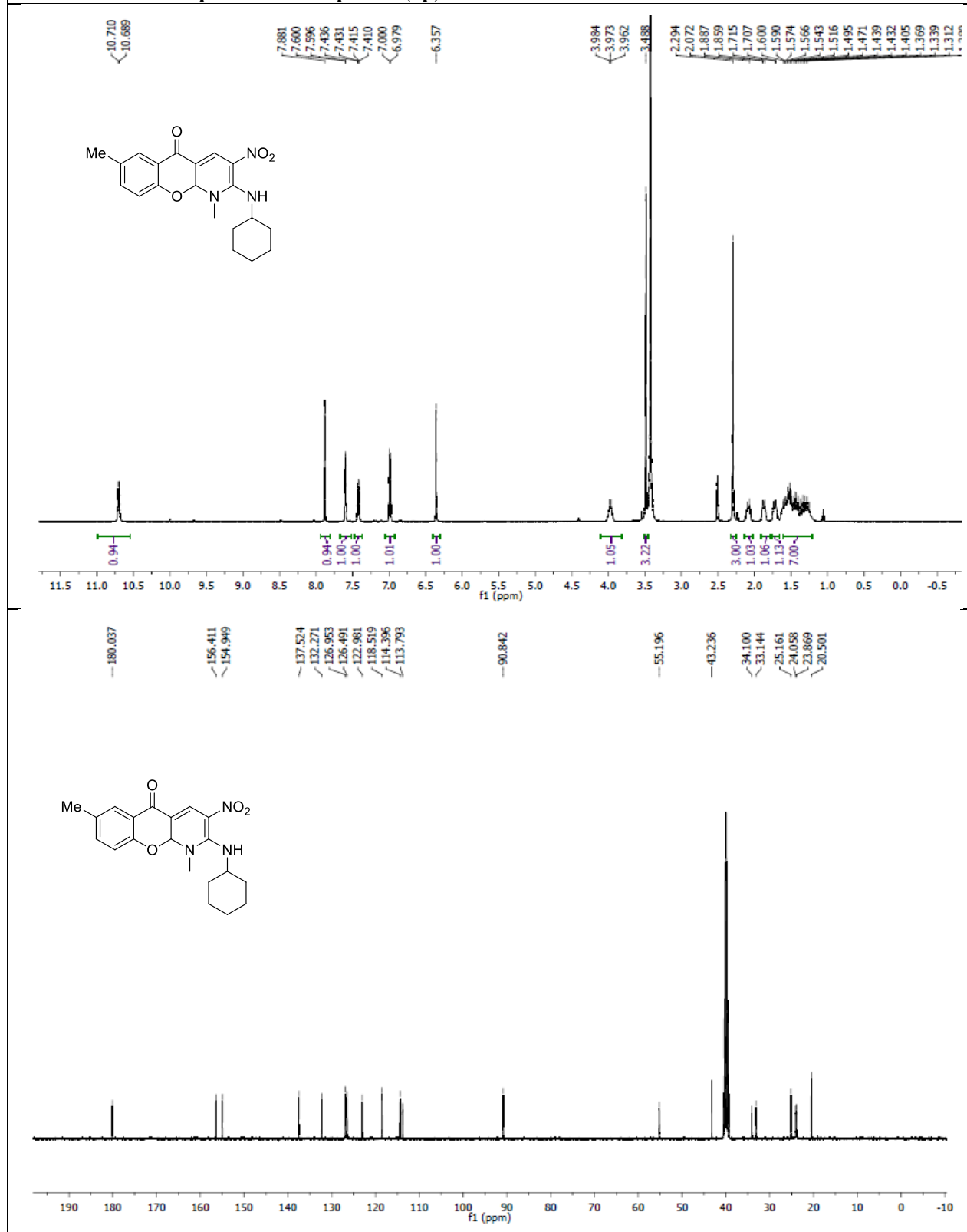
¹H and ¹³C NMR Spectra of Compound (5n)



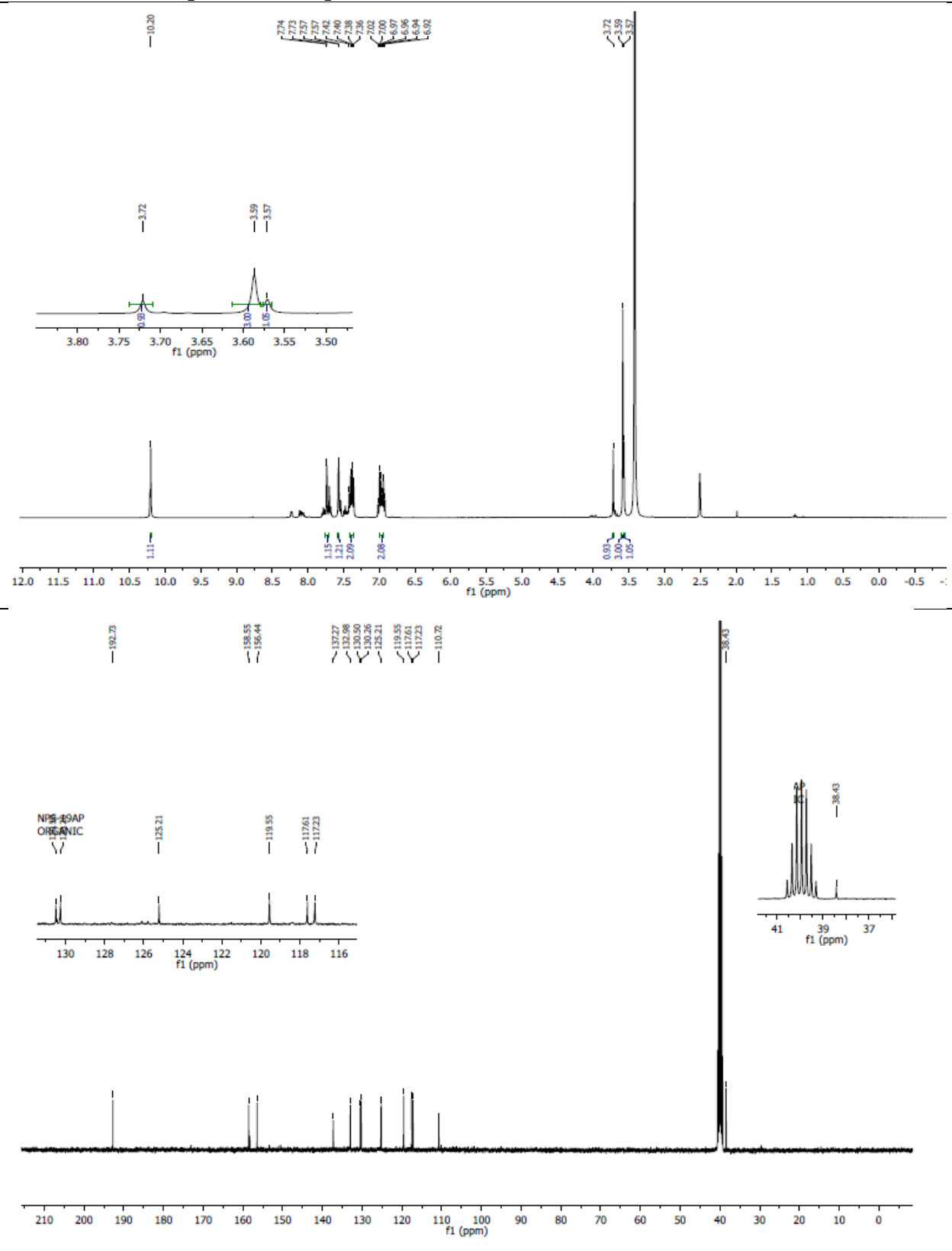
¹H and ¹³C NMR Spectra of Compound (5o)



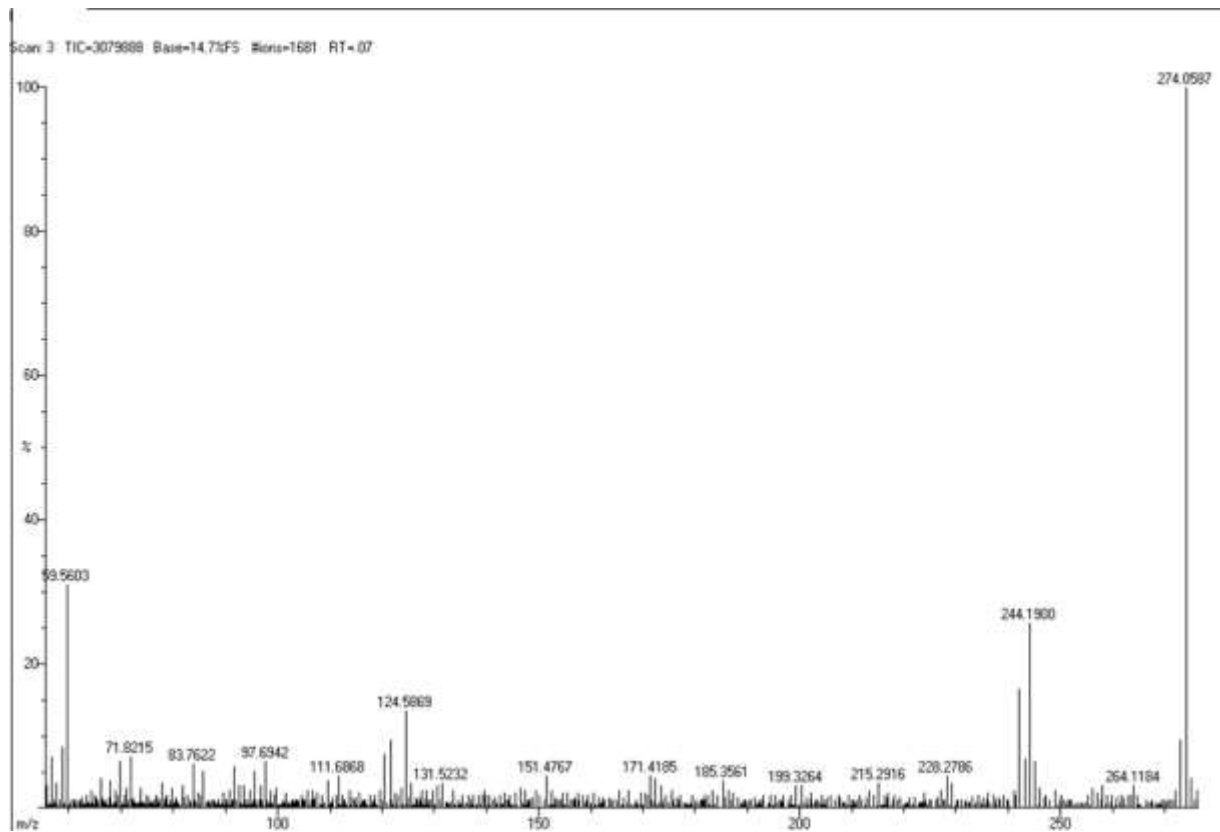
¹H and ¹³C NMR Spectra of Compound (5p)



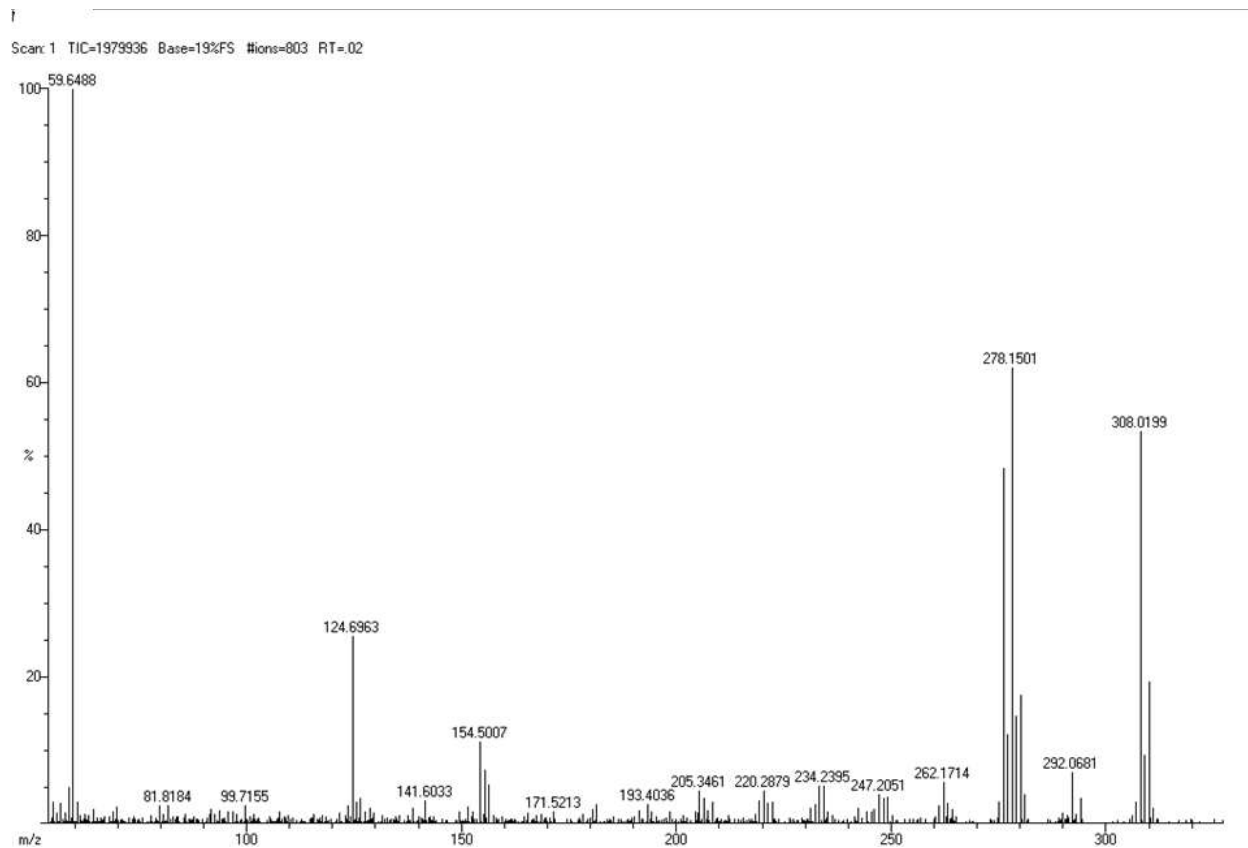
¹H and ¹³C NMR Spectra of Compound (14)



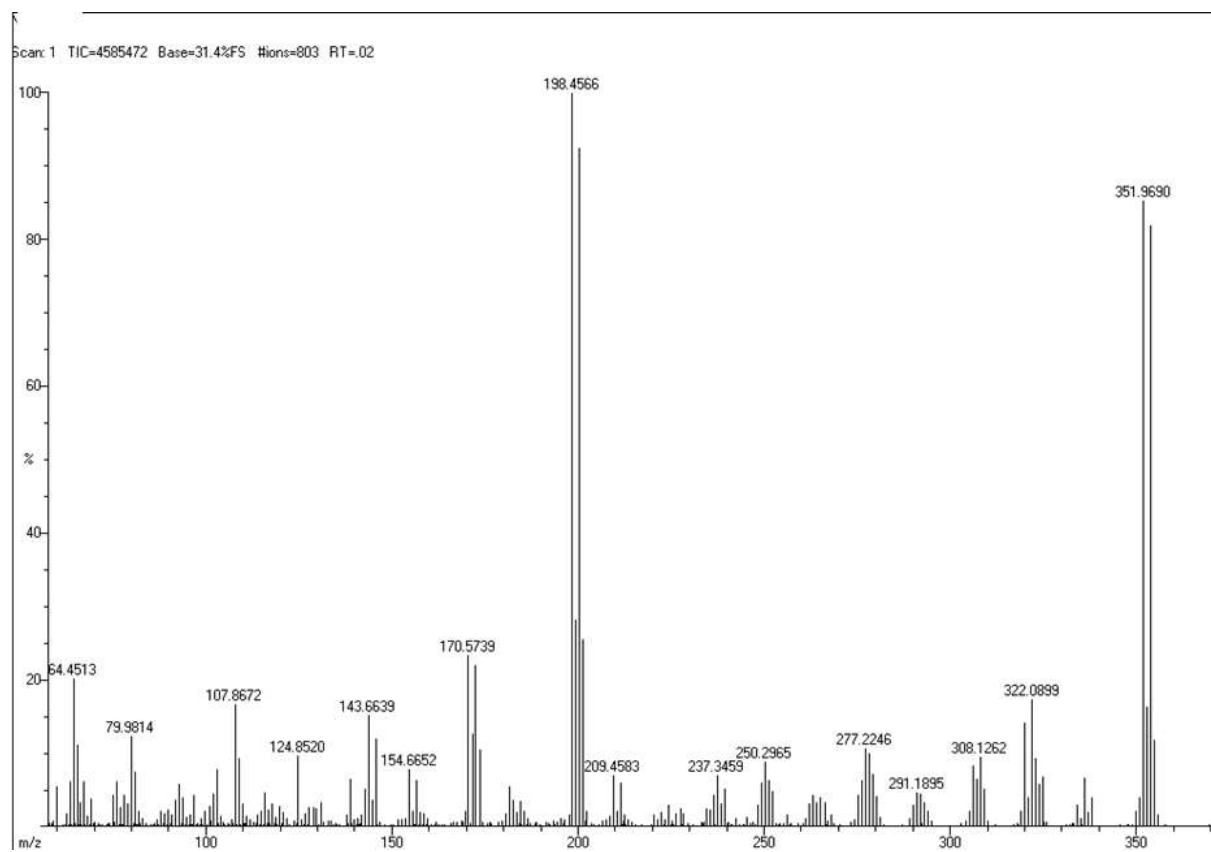
EI-HRMS Spectrum:



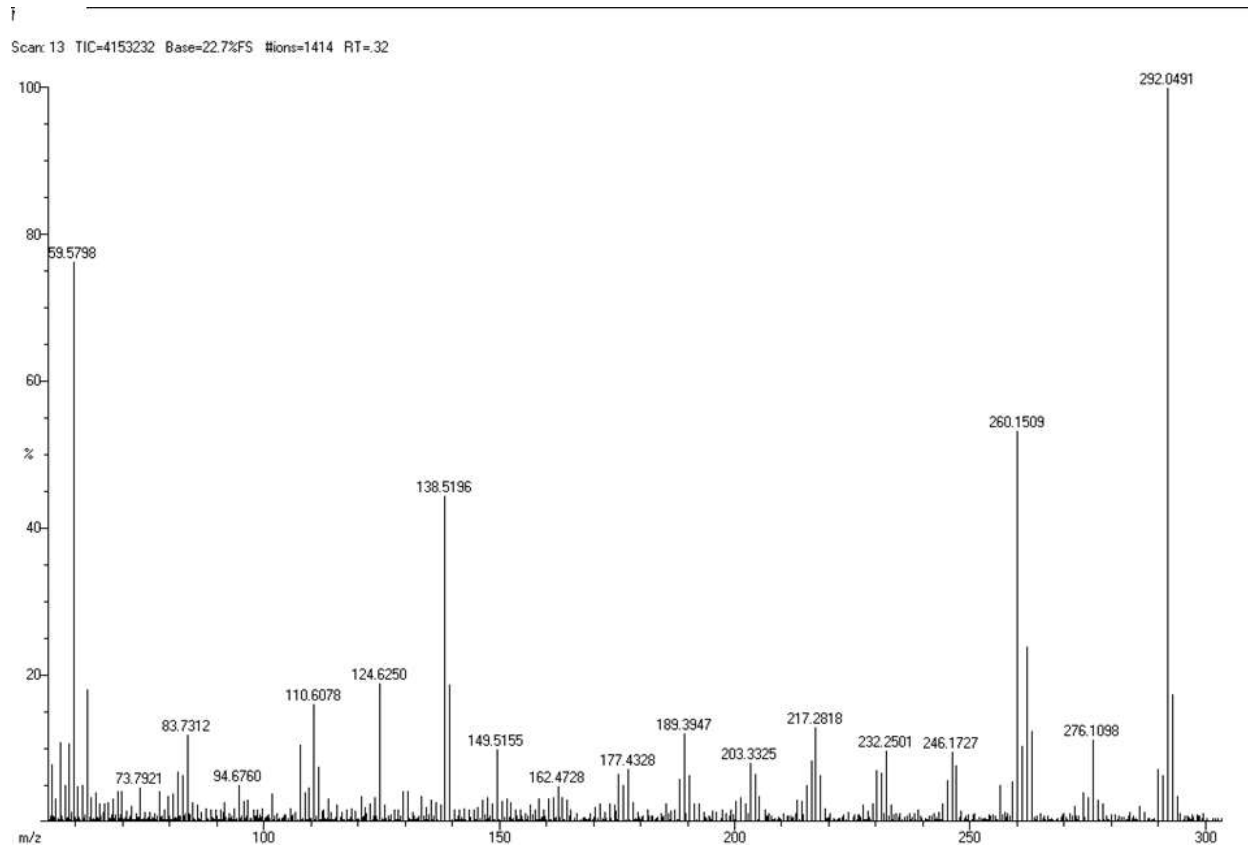
EI-HRMS spectrum of compound 3a



EI-HRMS spectrum of compound 3b

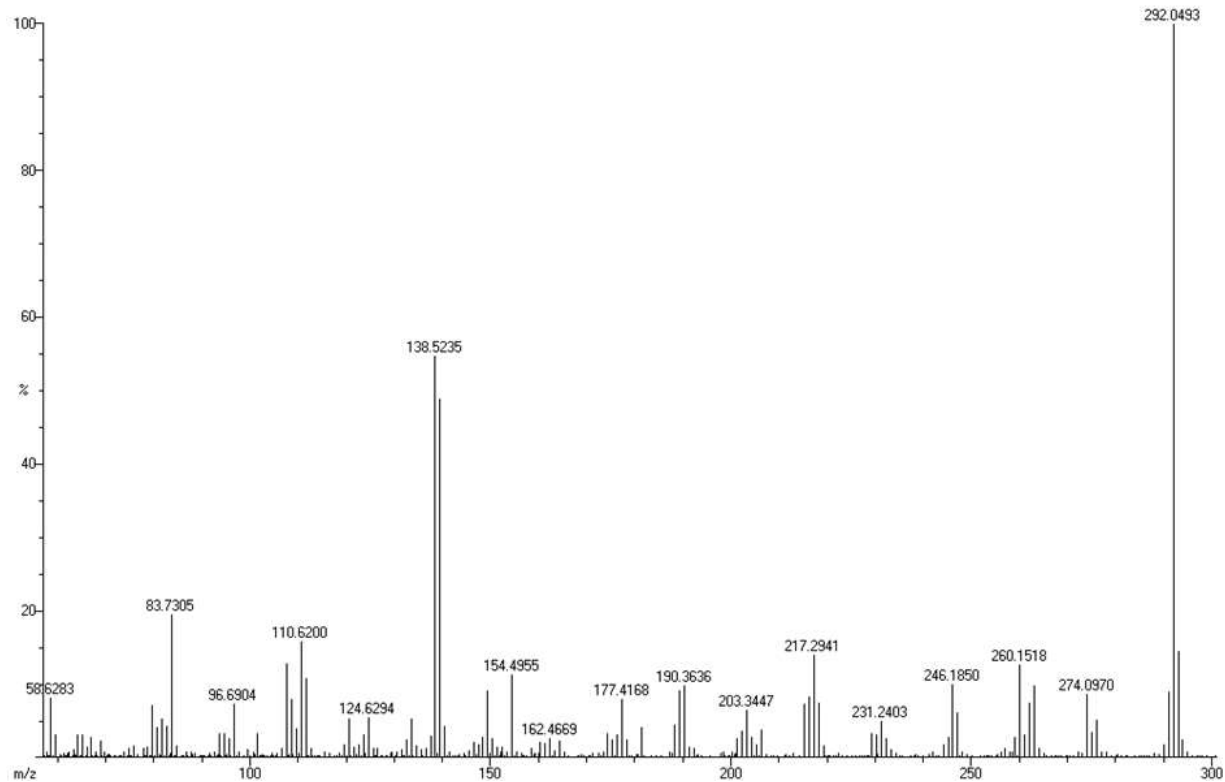


EI-MS spectrum of compound 3c

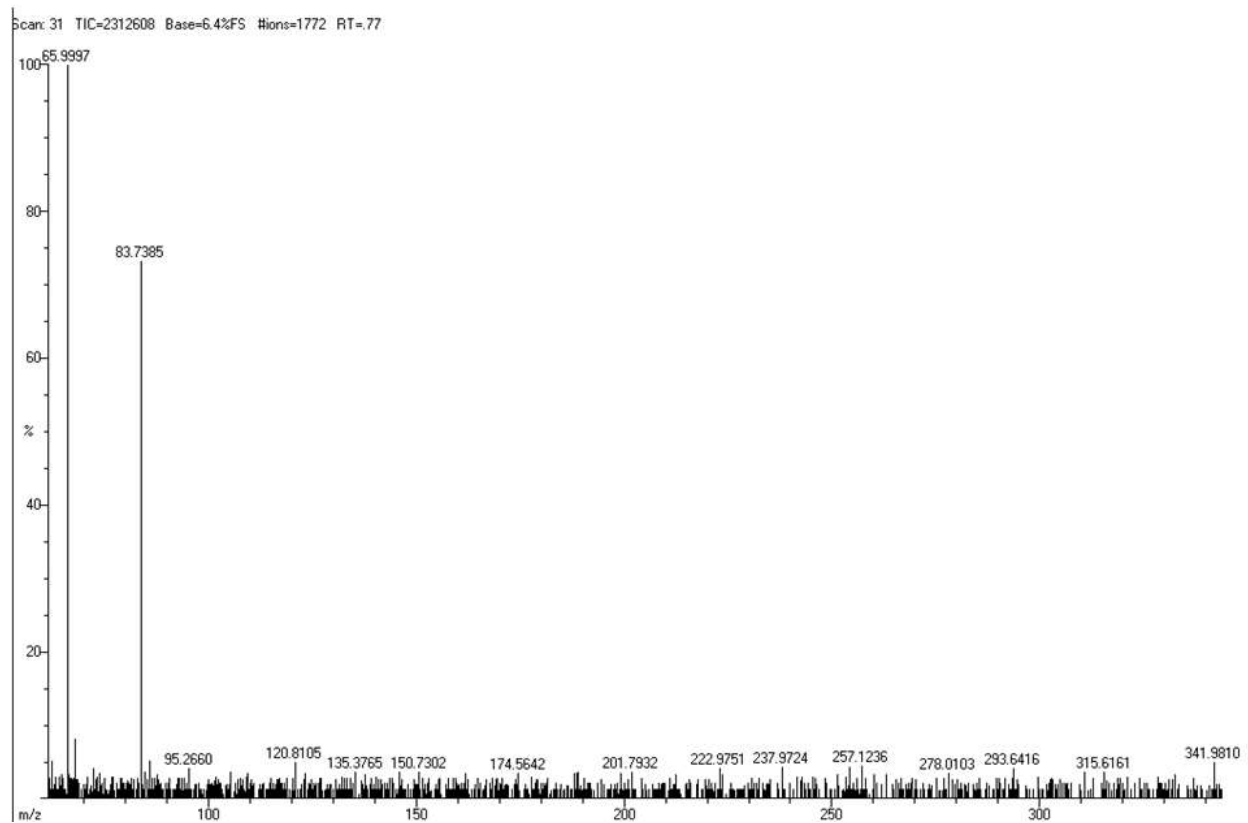


EI-HRMS spectrum of compound 3d

Scan: 2 TIC=4706656 Base=39.3%FS #Ions=1463 RT=.03

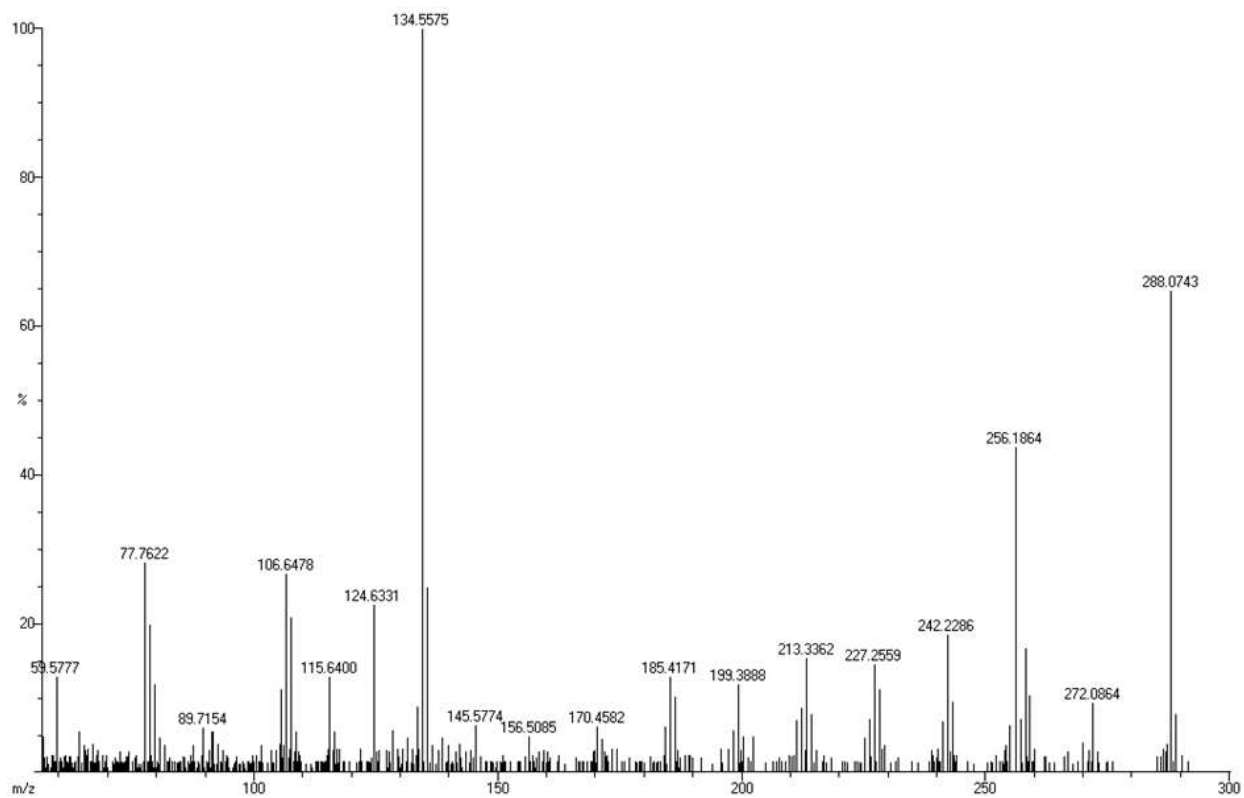


EI-HRMS spectrum of compound 3e

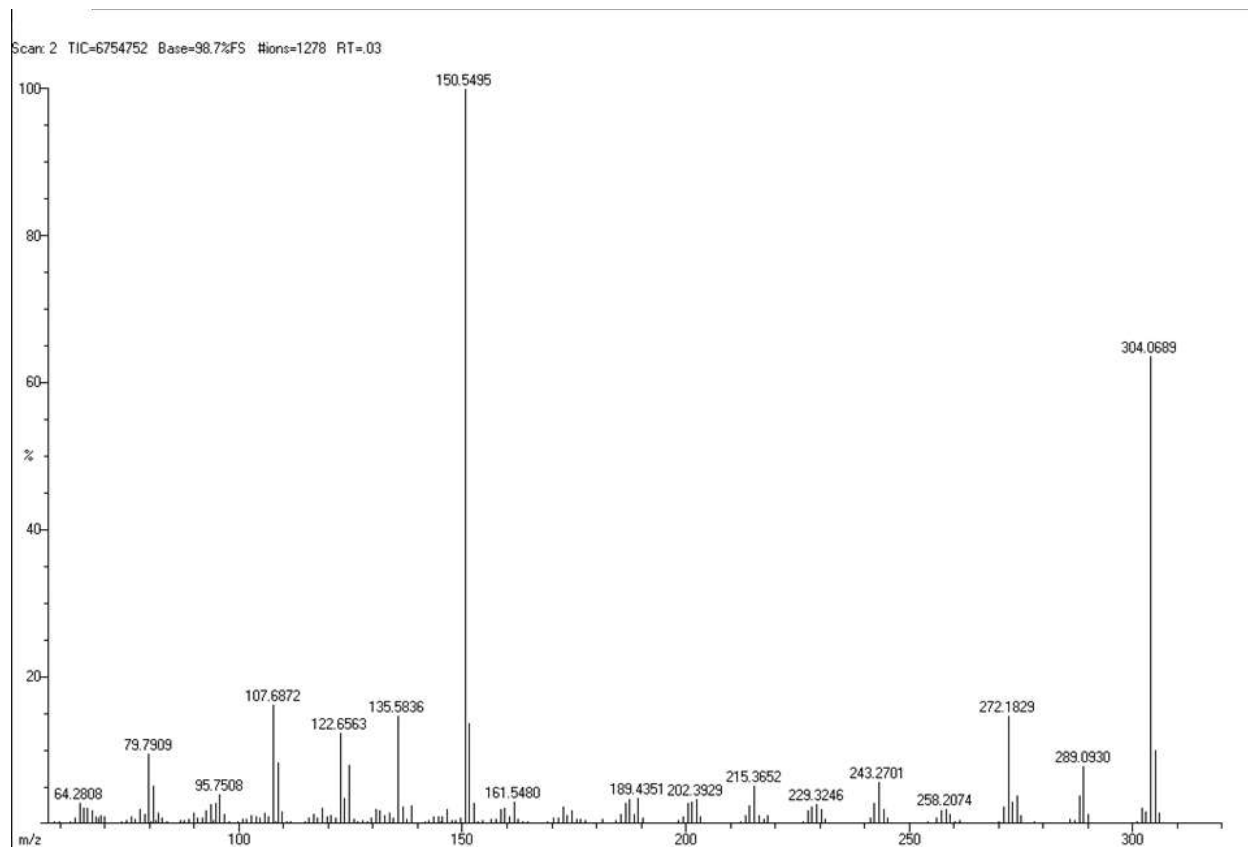


EI-MS spectrum of compound 3f

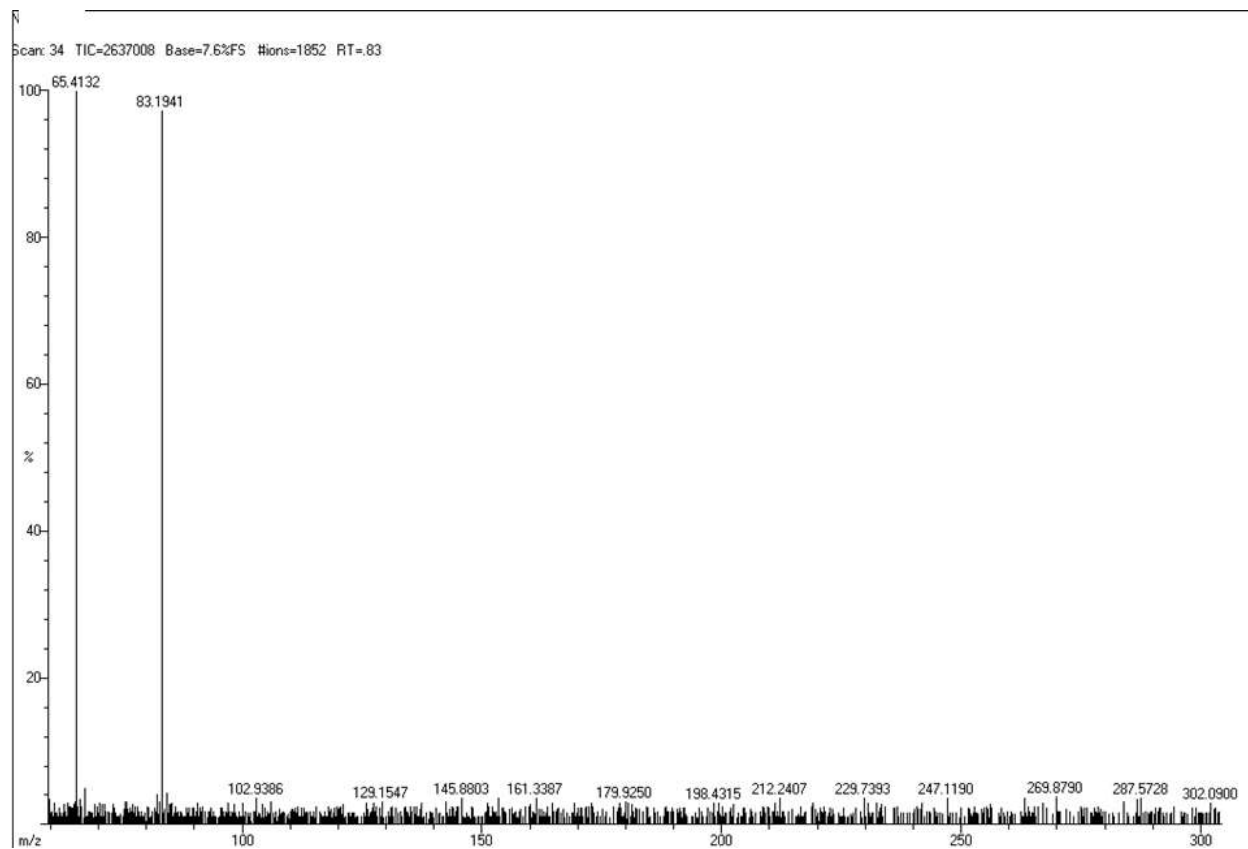
Scan 1 TIC=1098672 Base=6%FS #Ions=645 RT=.02



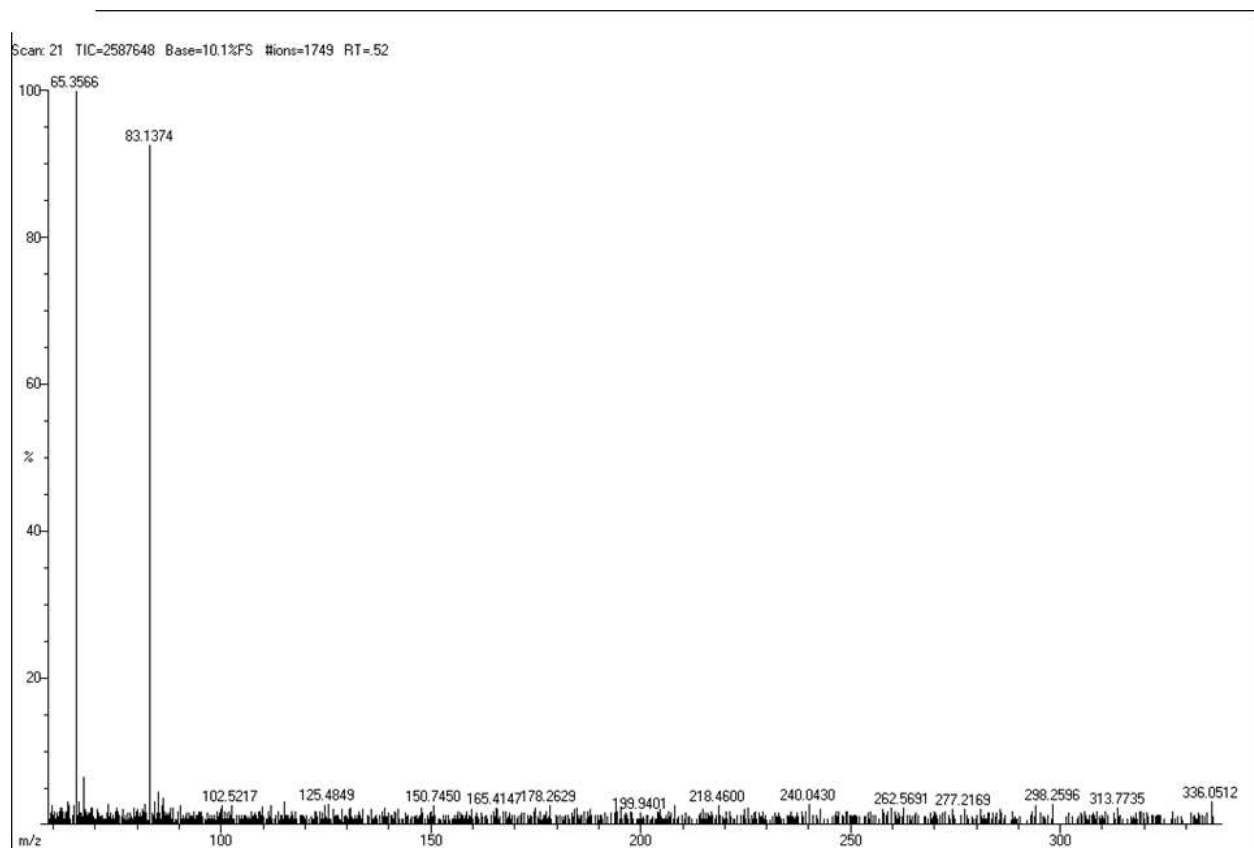
EI-HRMS spectrum of compound 3g



EI-HRMS spectrum of compound 3h

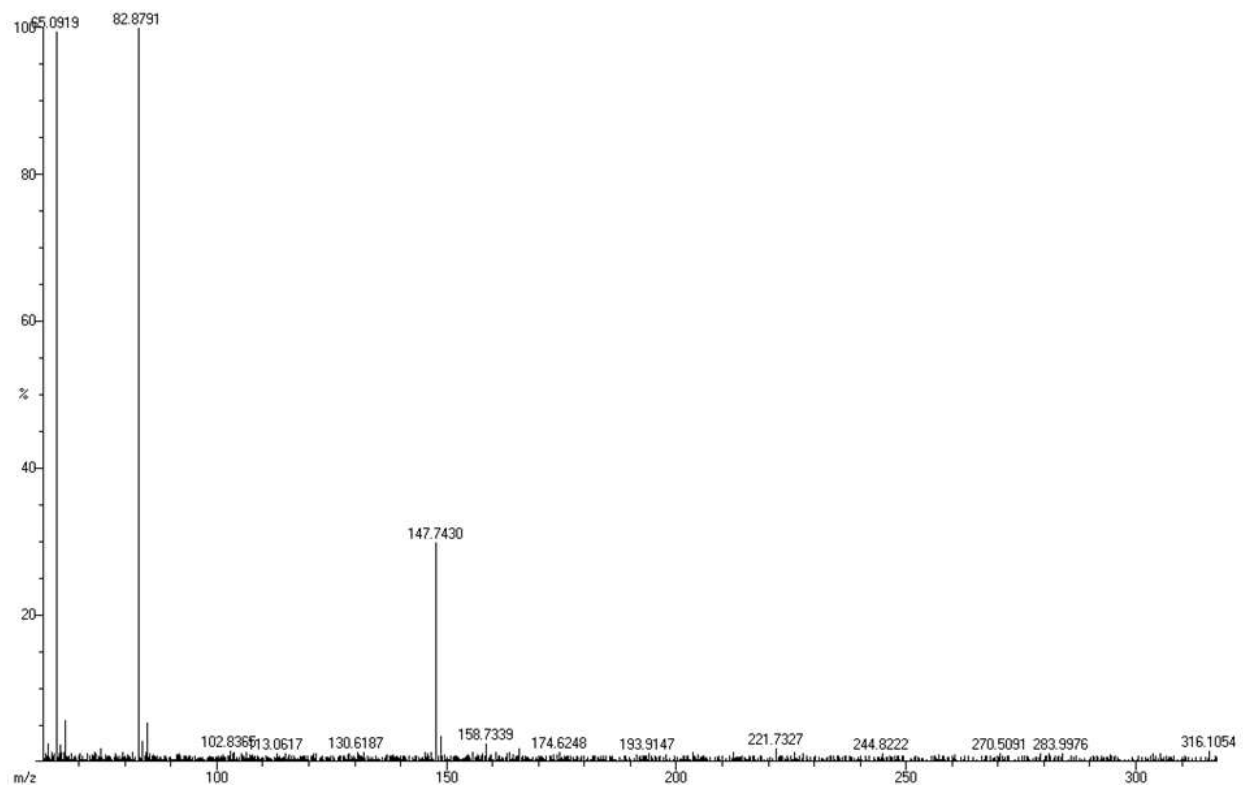


EI-HRMS spectrum of compound 3i

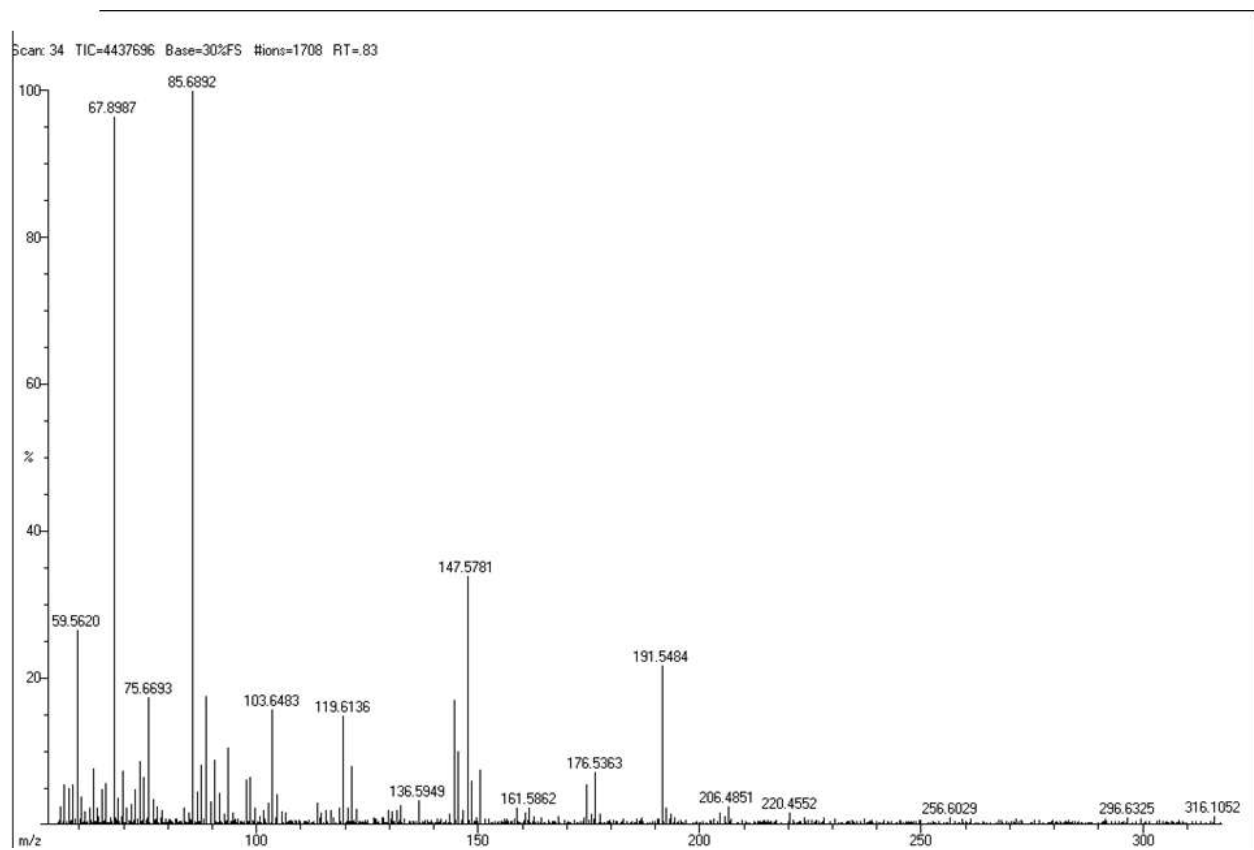


EI-MS spectrum of compound 3j

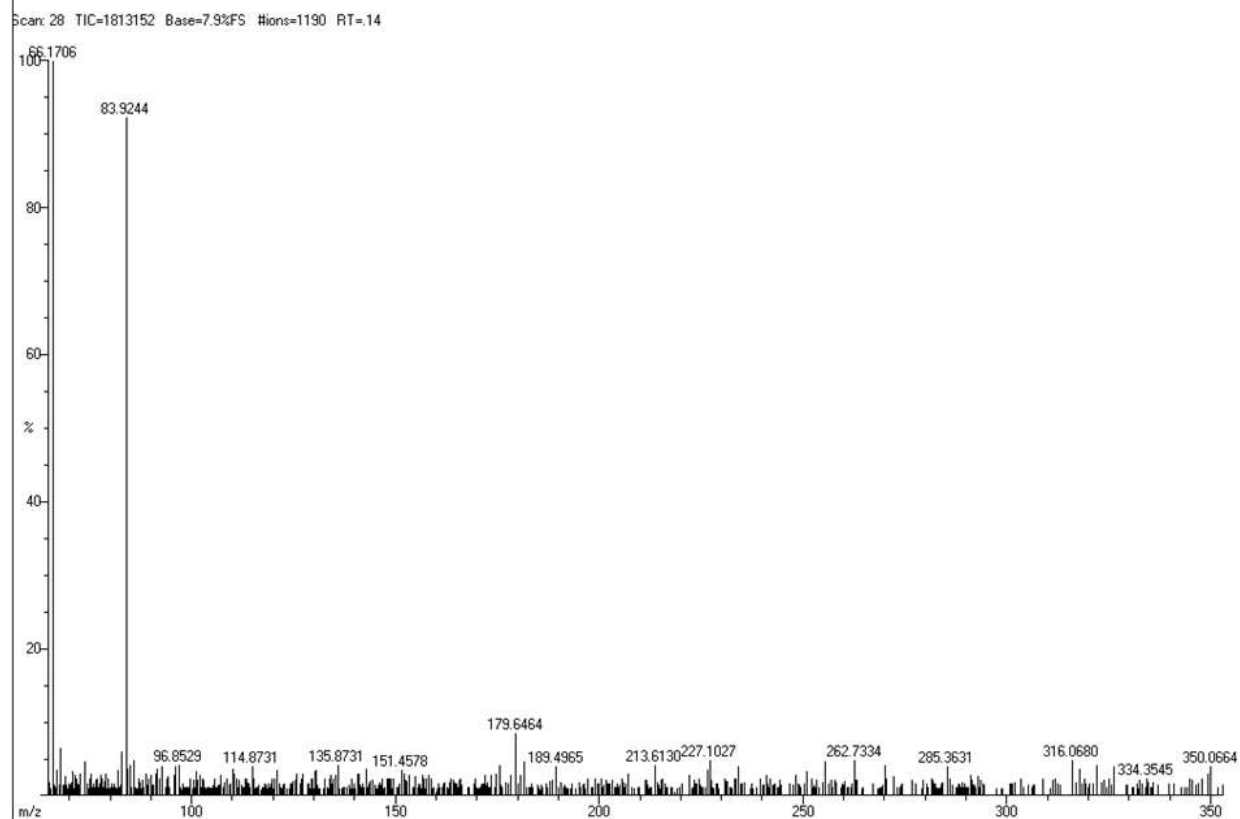
Scan: 22 TIC=3129824 Base=20.2%FS #Ions=1856 RT=.53



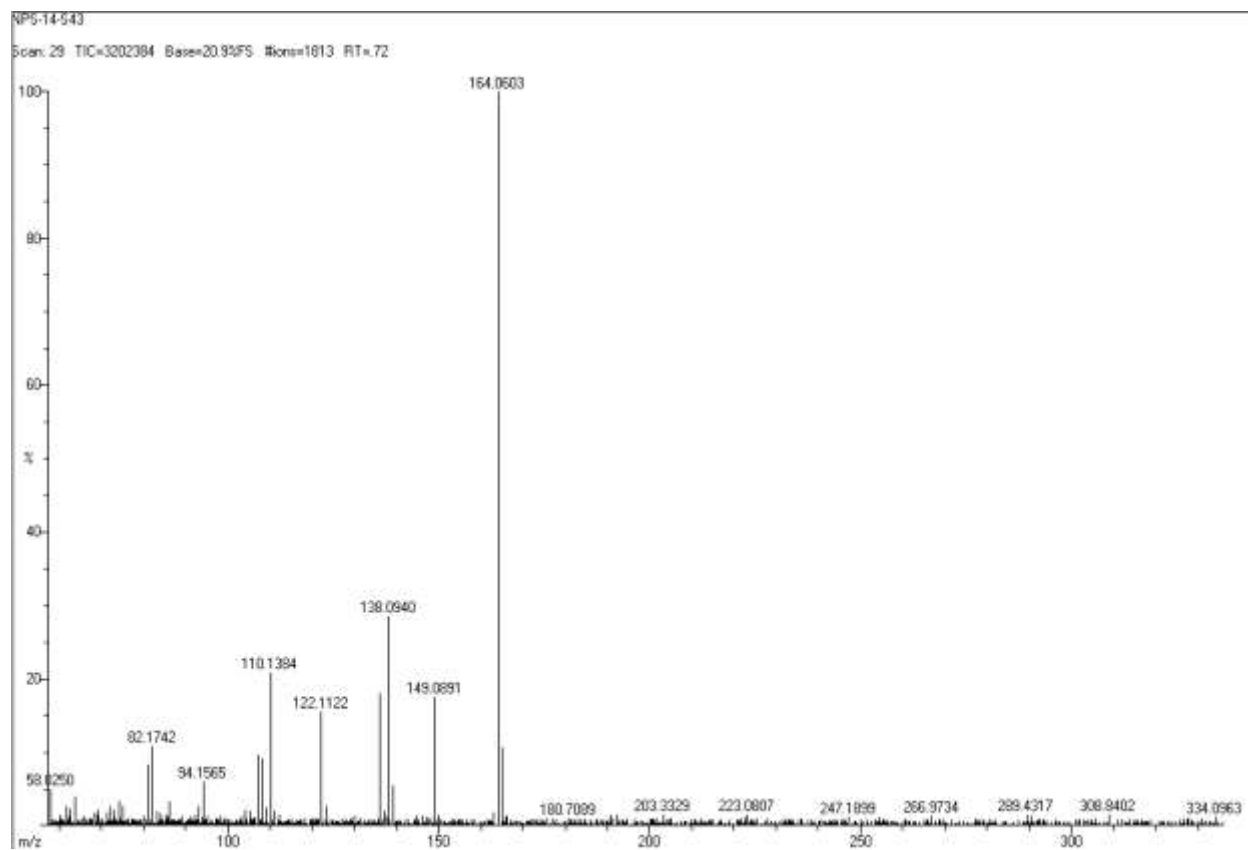
EI-MS spectrum of compound 3k



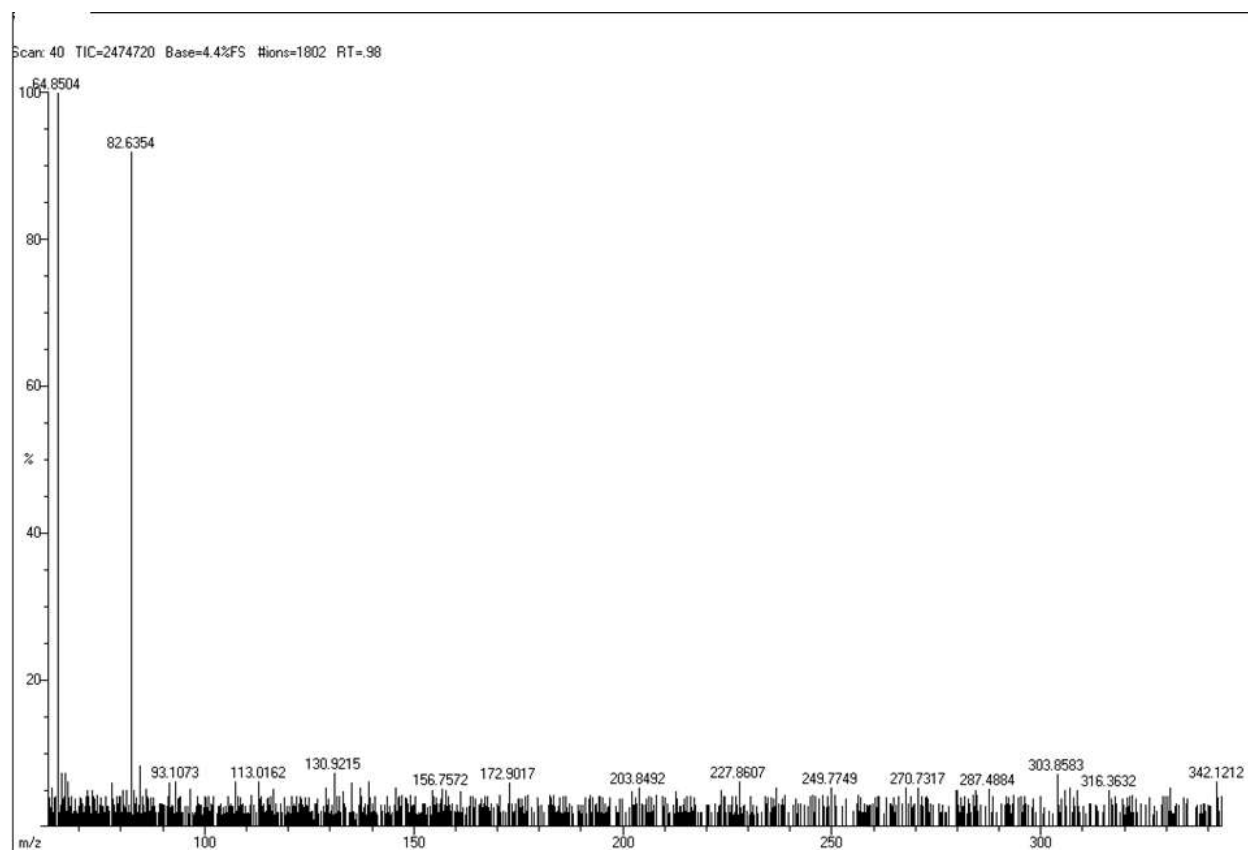
EI-MS spectrum of compound 31



EI-MS spectrum of compound 3m

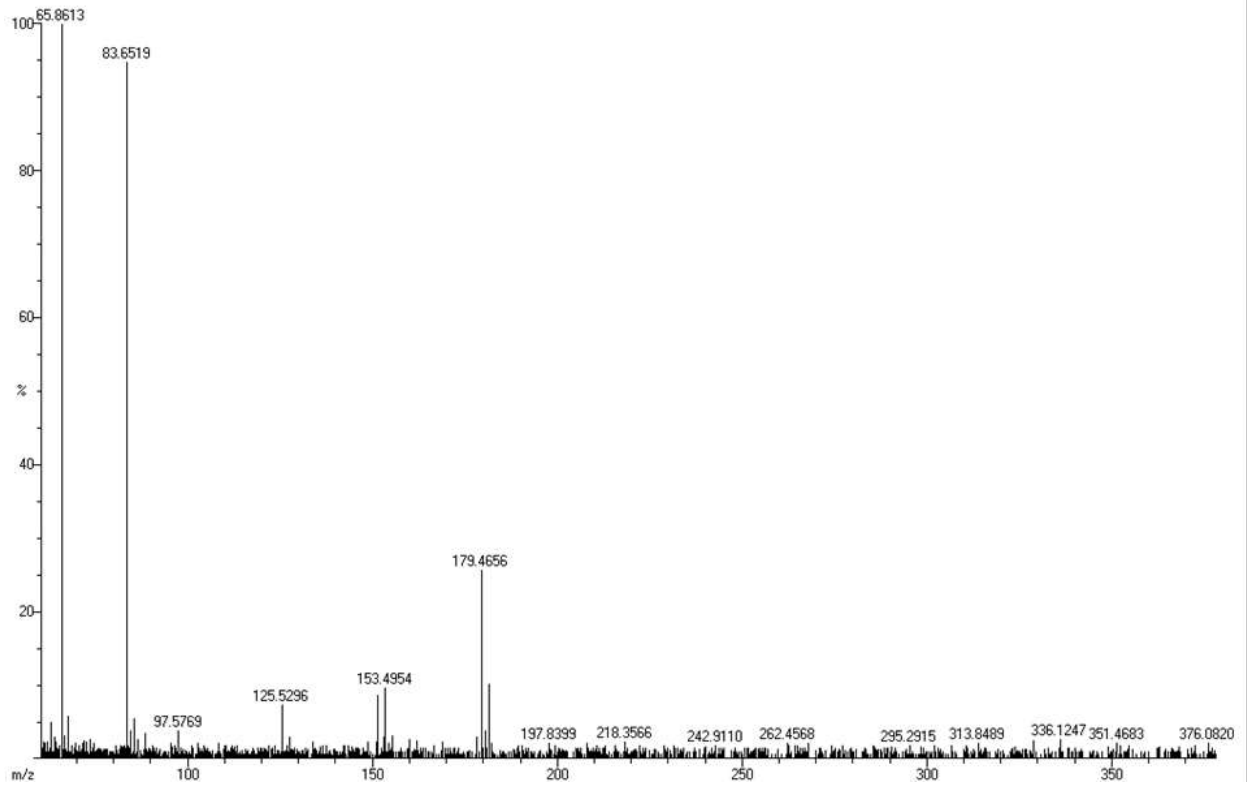


EI-MS spectrum of compound 3n

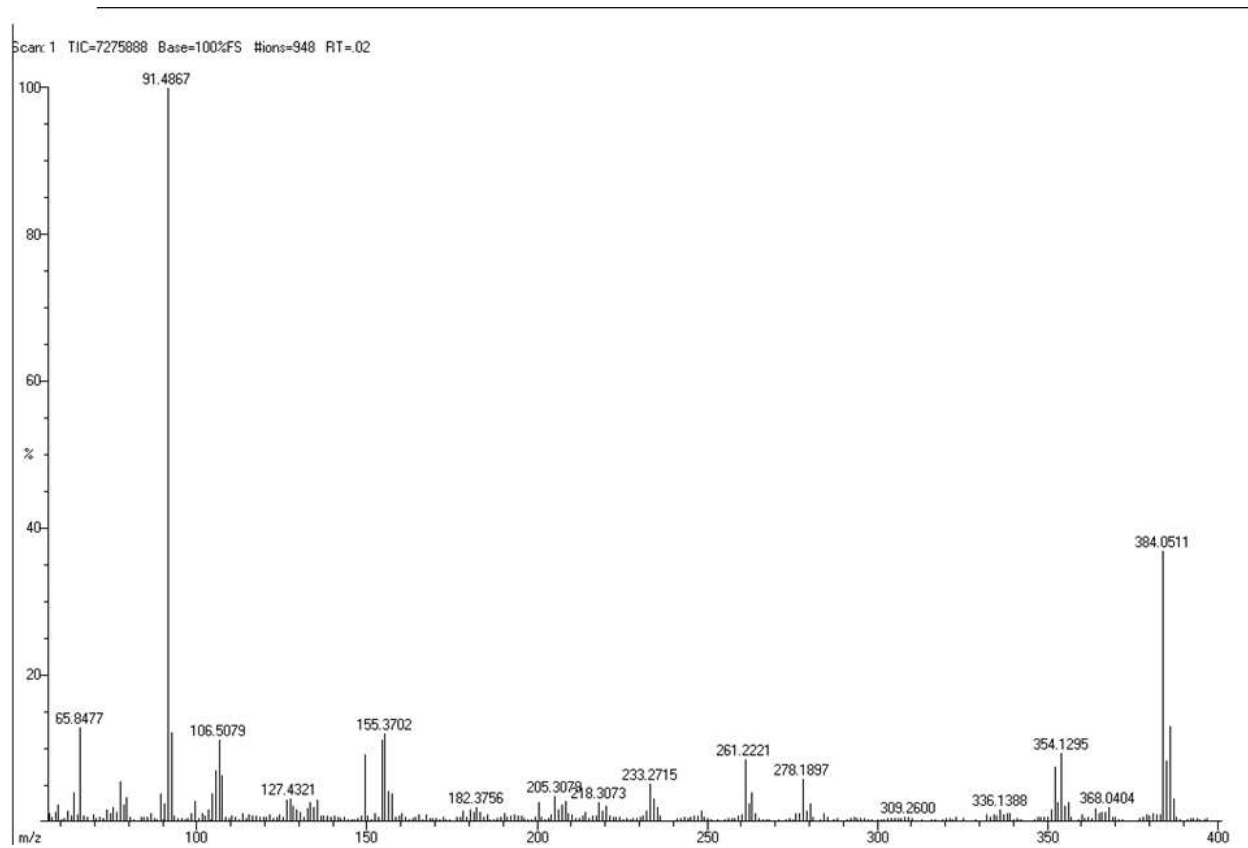


EI-MS spectrum of compound 3o

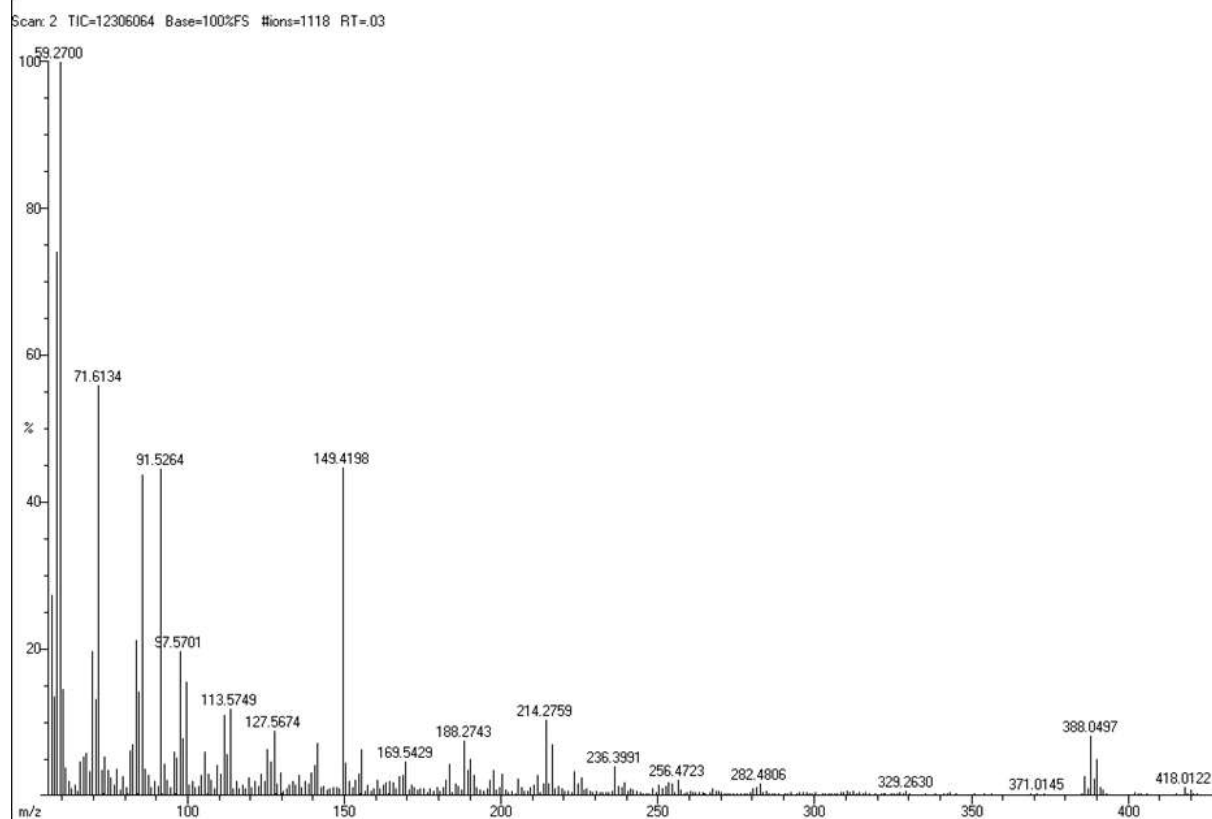
Scan: 32 TIC=2993392 Base=12.2%FS #Ions=1842 RT=.78



EI-HRMS spectrum of compound 3p

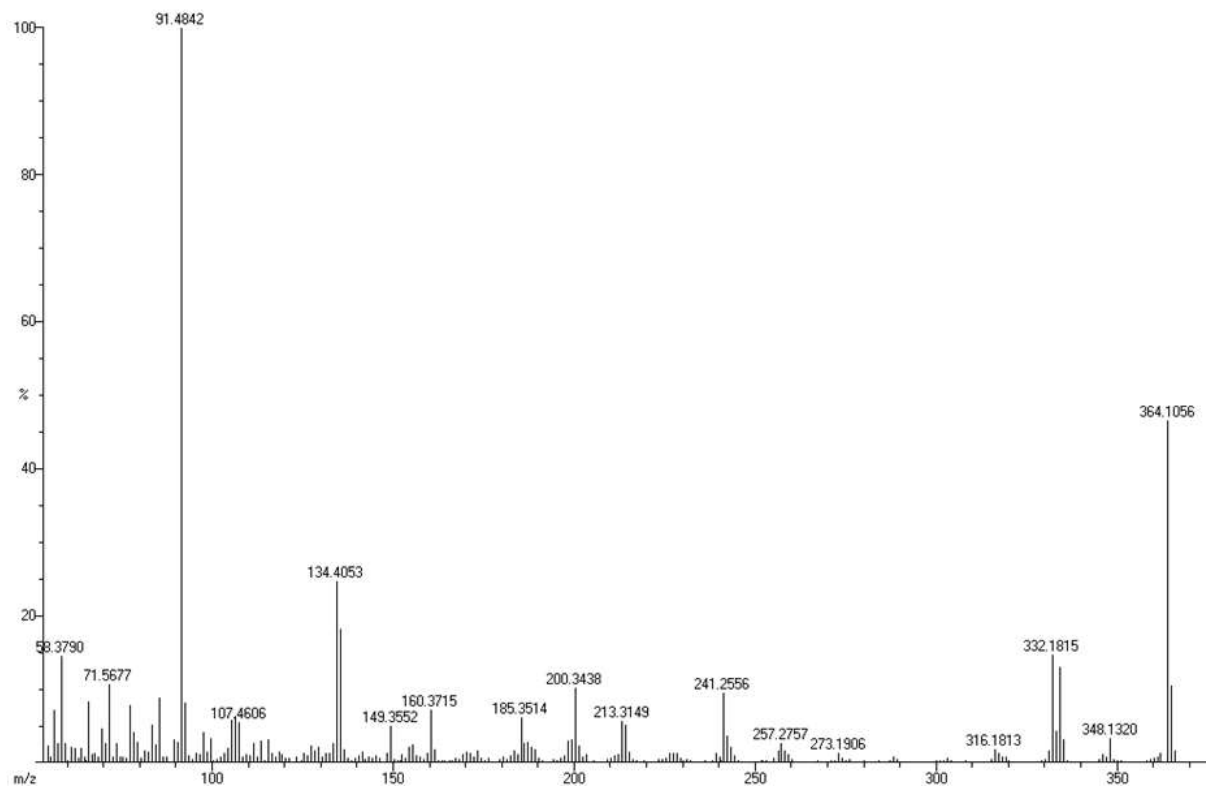


EI-HRMS spectrum of compound 3q



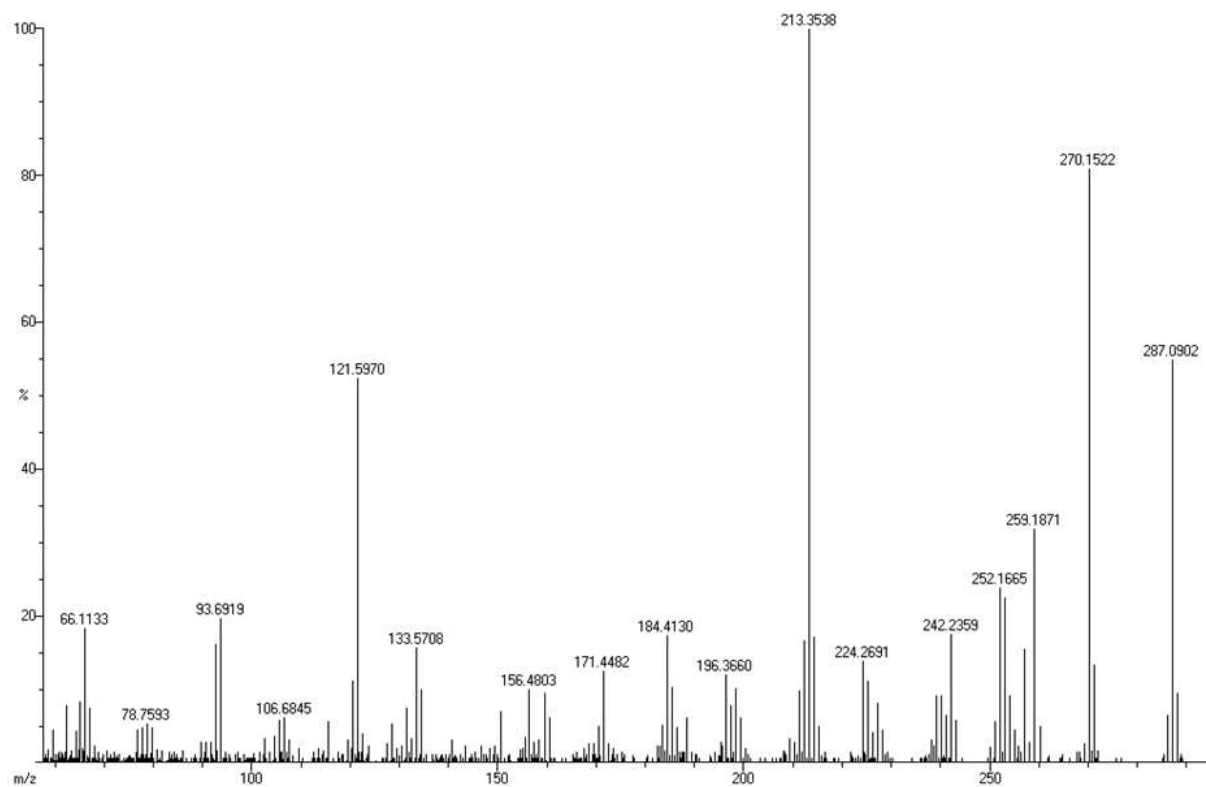
EI-MS spectrum of compound 3r

Scan: 1 TIC=6350400 Base=85.4%FS #Ions=807 RT=.02

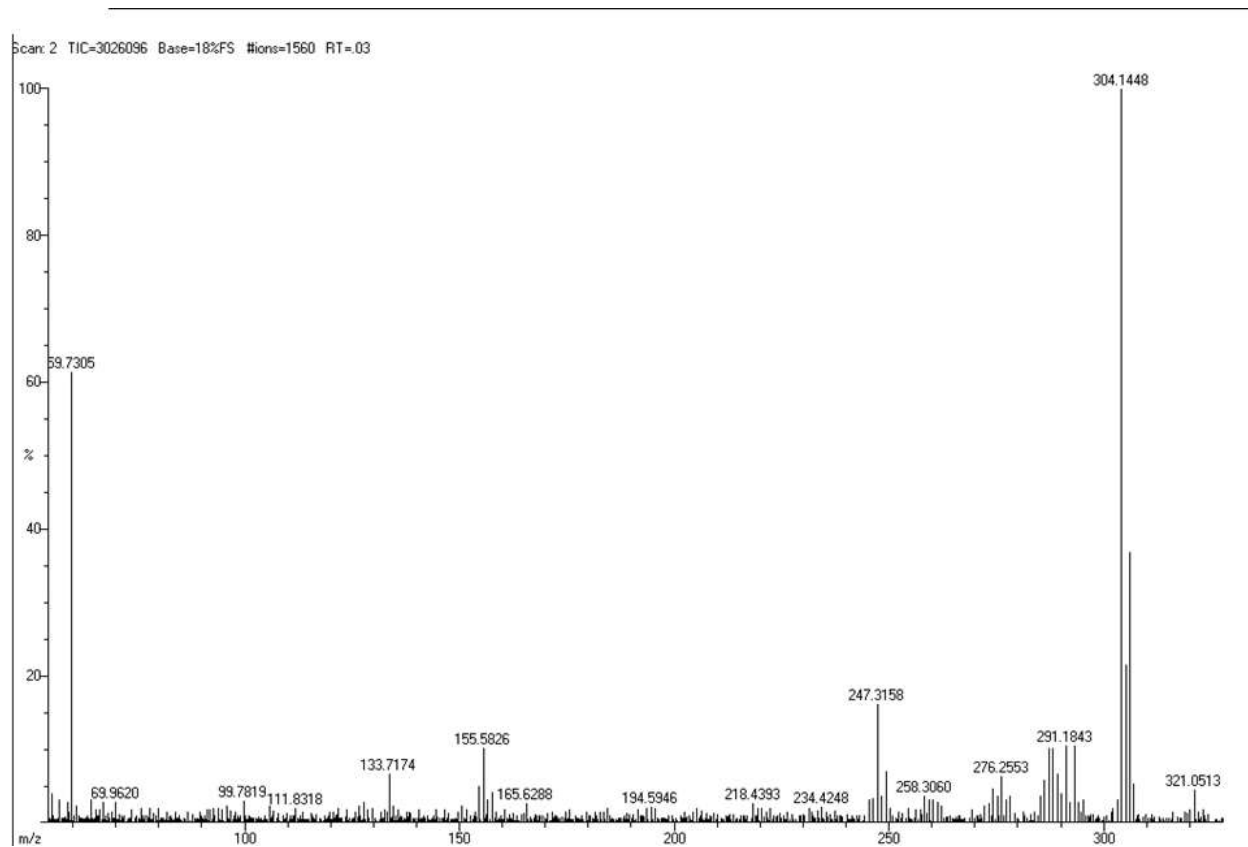


EI-HRMS spectrum of compound 3s

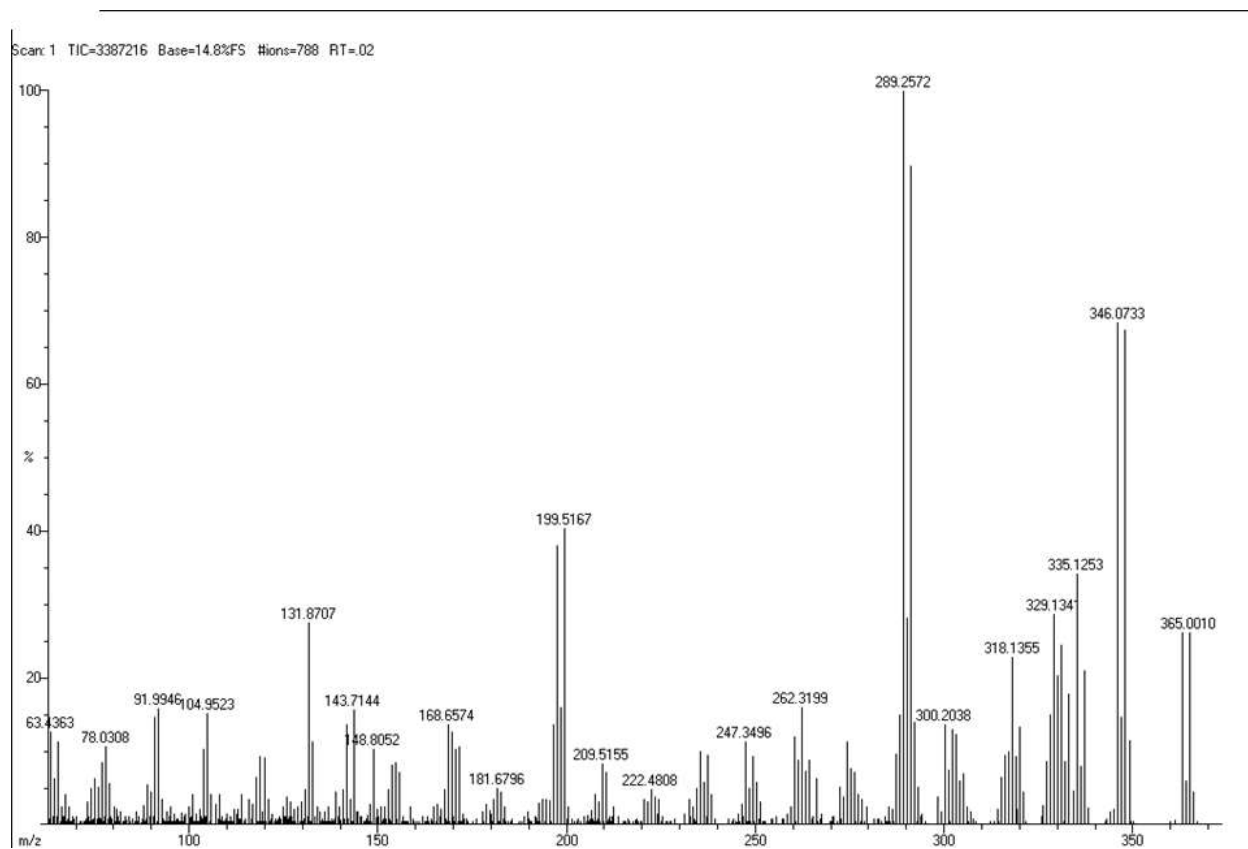
Scan: 1 TIC=1853312 Base=11.6%FS #Ions=685 RT=.02



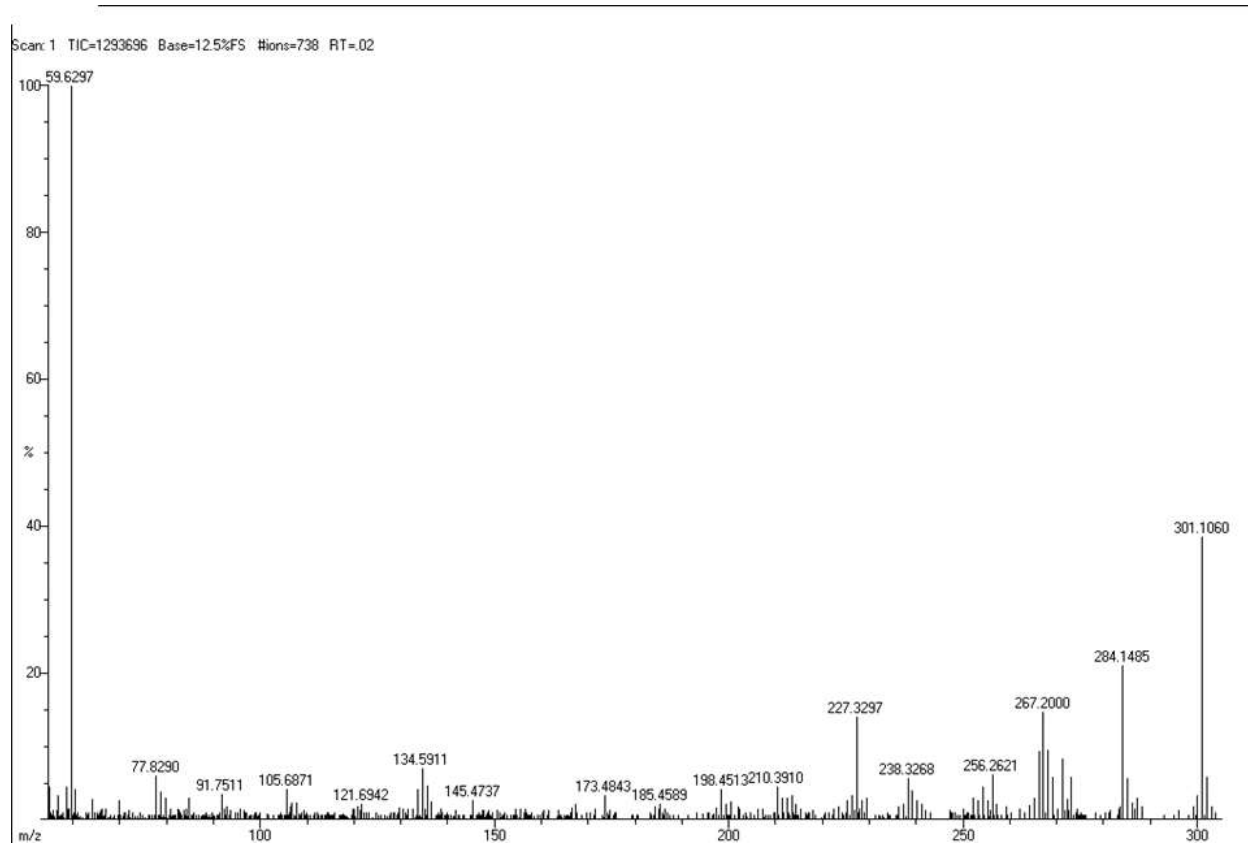
EI-HRMS spectrum of compound 5a



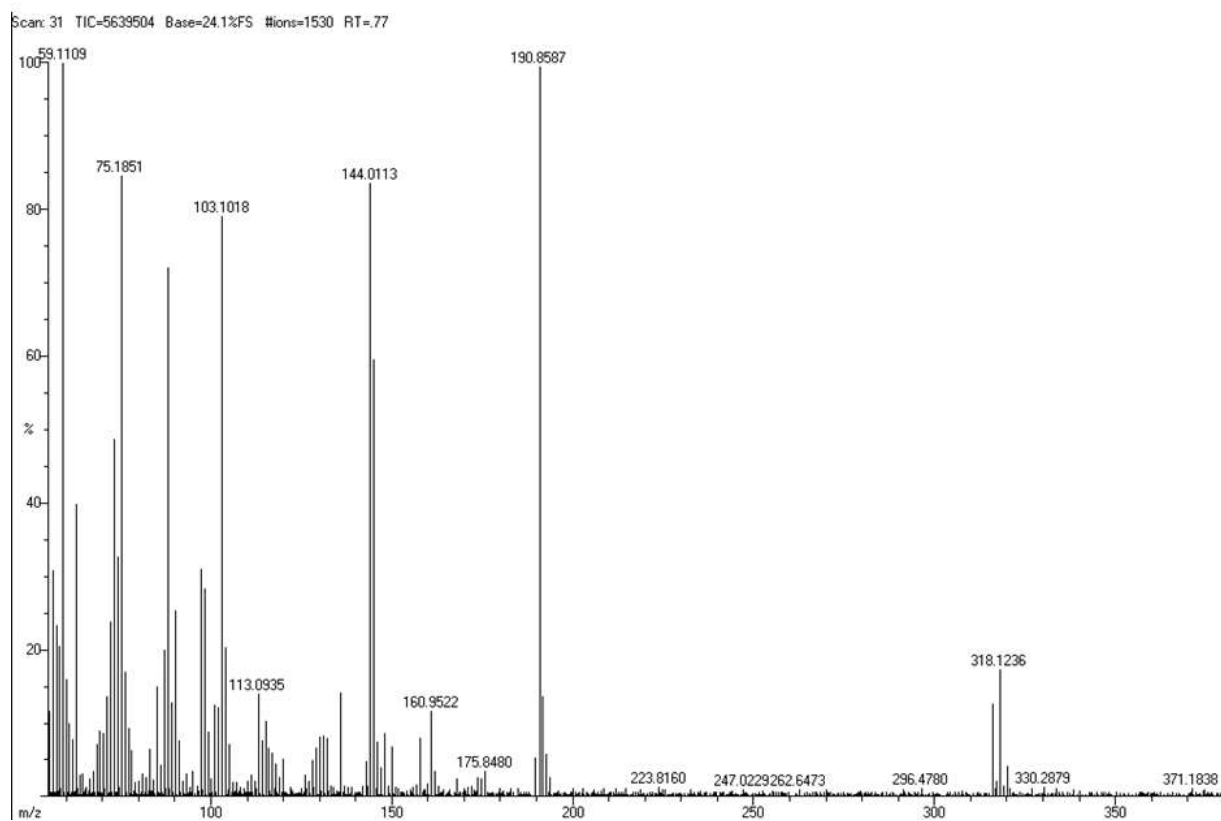
EI-HRMS spectrum of compound 5b



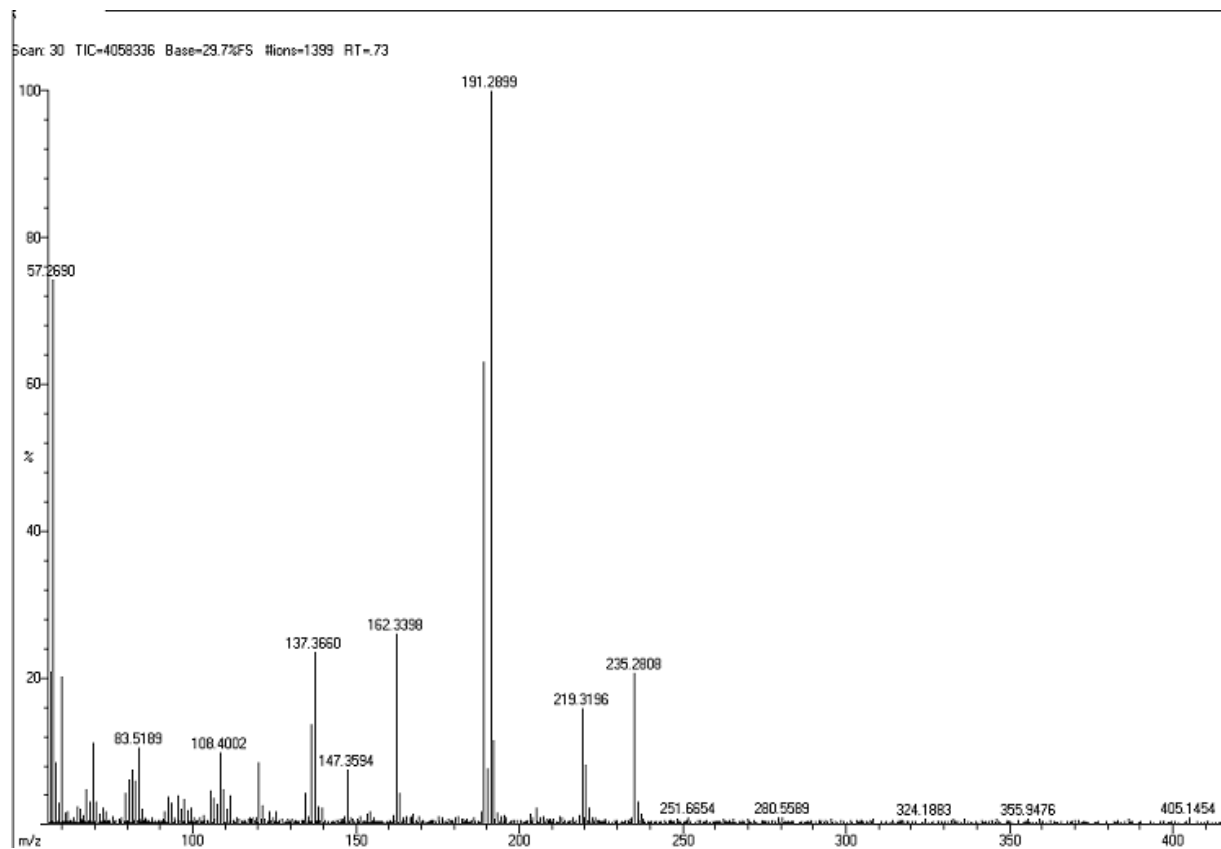
EI-HRMS spectrum of compound 5c



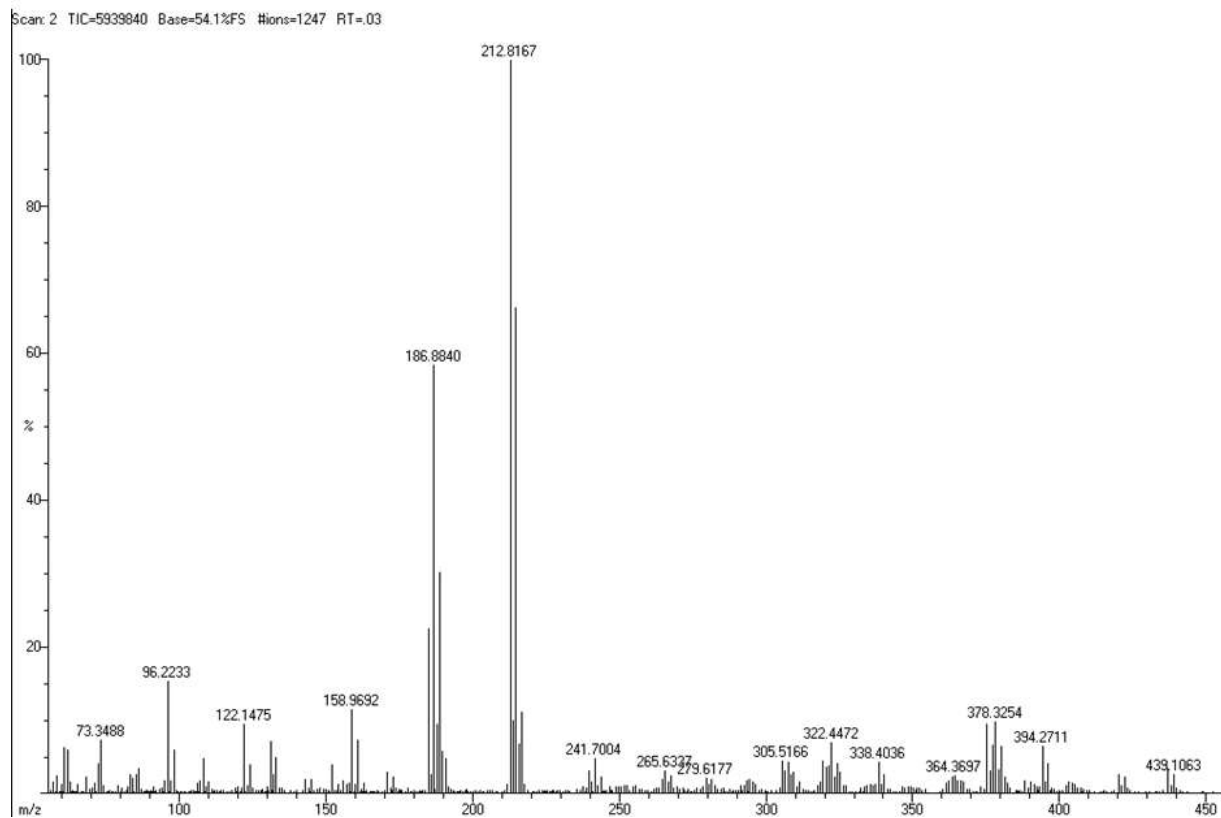
EI-HRMS spectrum of compound 5d



EI-MS spectrum of compound 5e

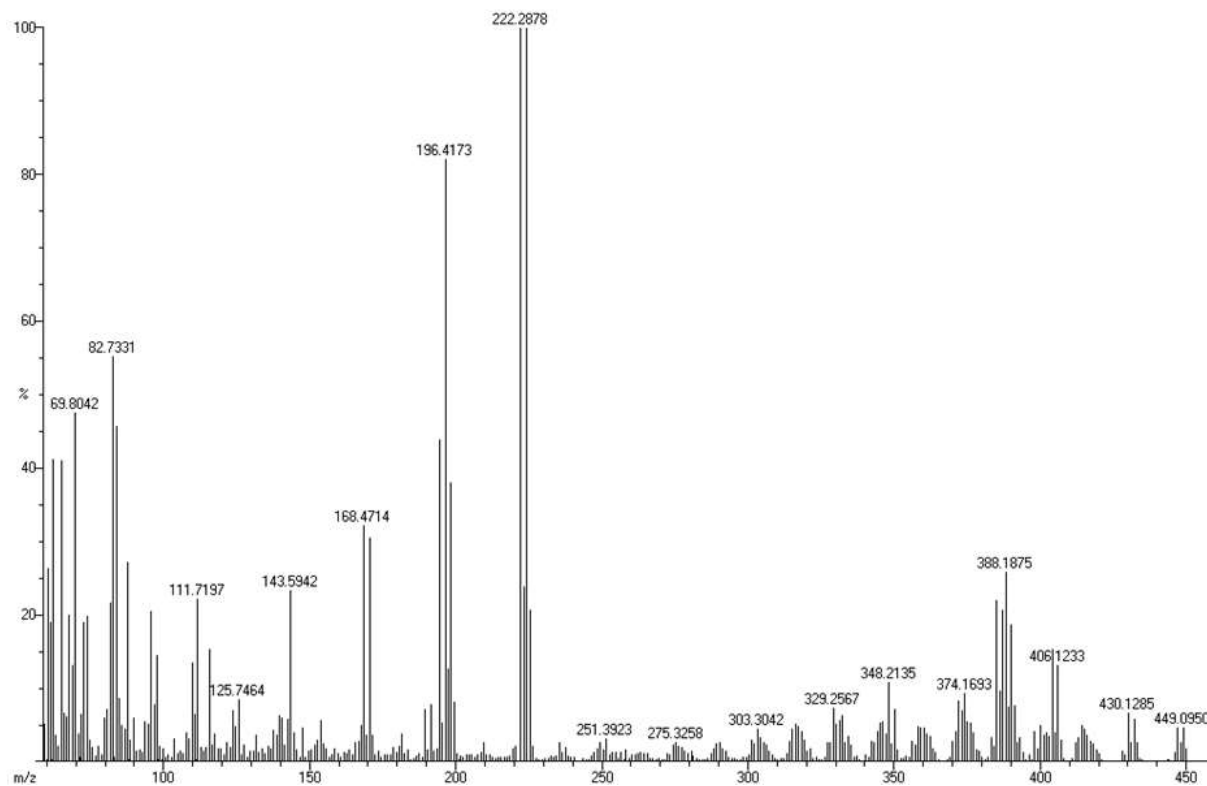


EI-HRMS spectrum of compound 5f

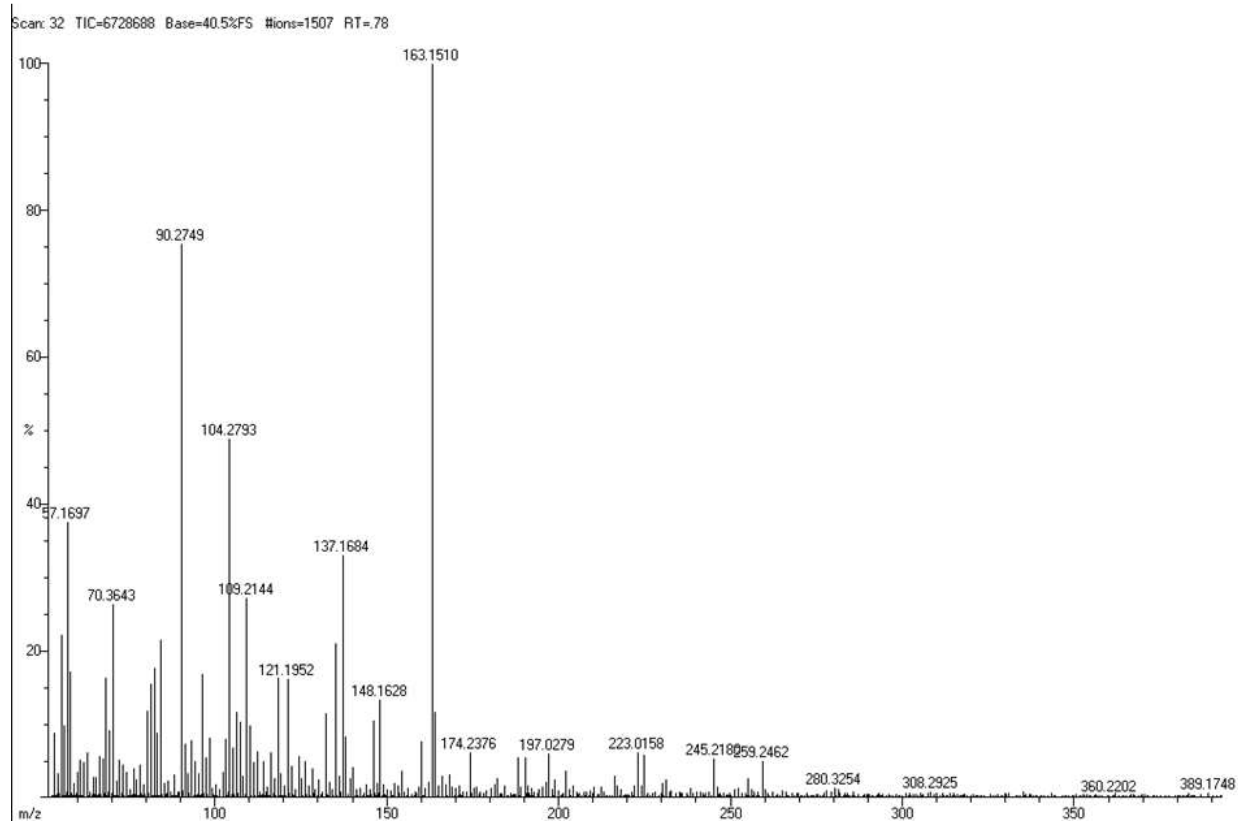


EI-HRMS spectrum of compound 5g

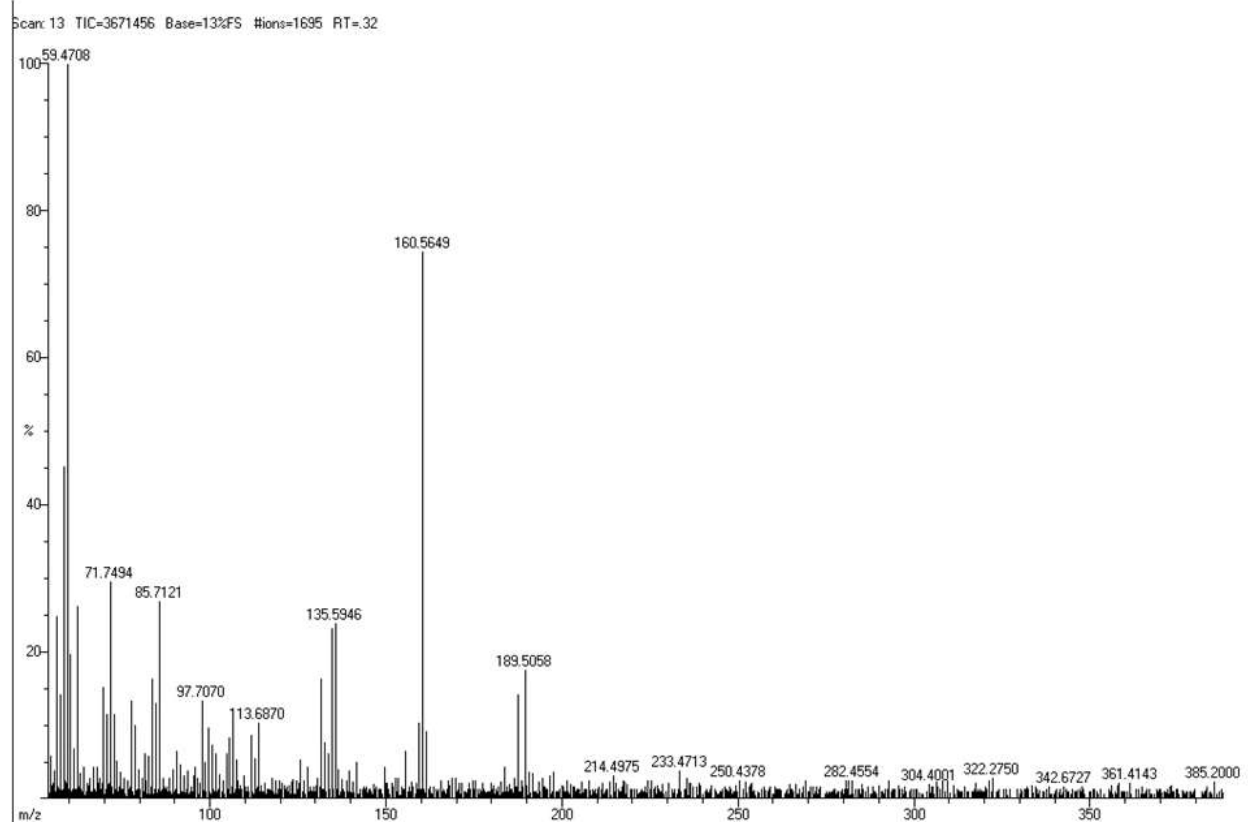
Scan: 1 TIC=23452064 Base=100%FS #Ions=808 RT=.02



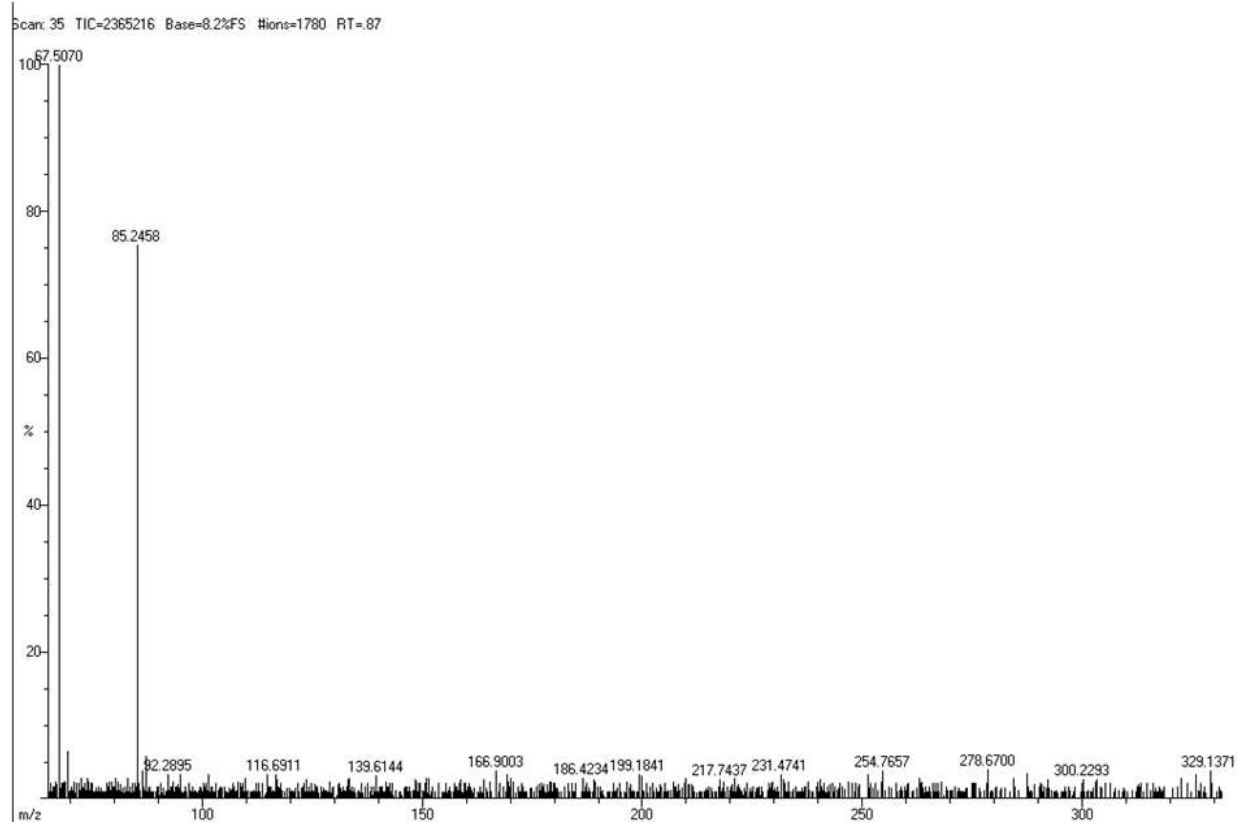
EI-HRMS spectrum of compound 5h



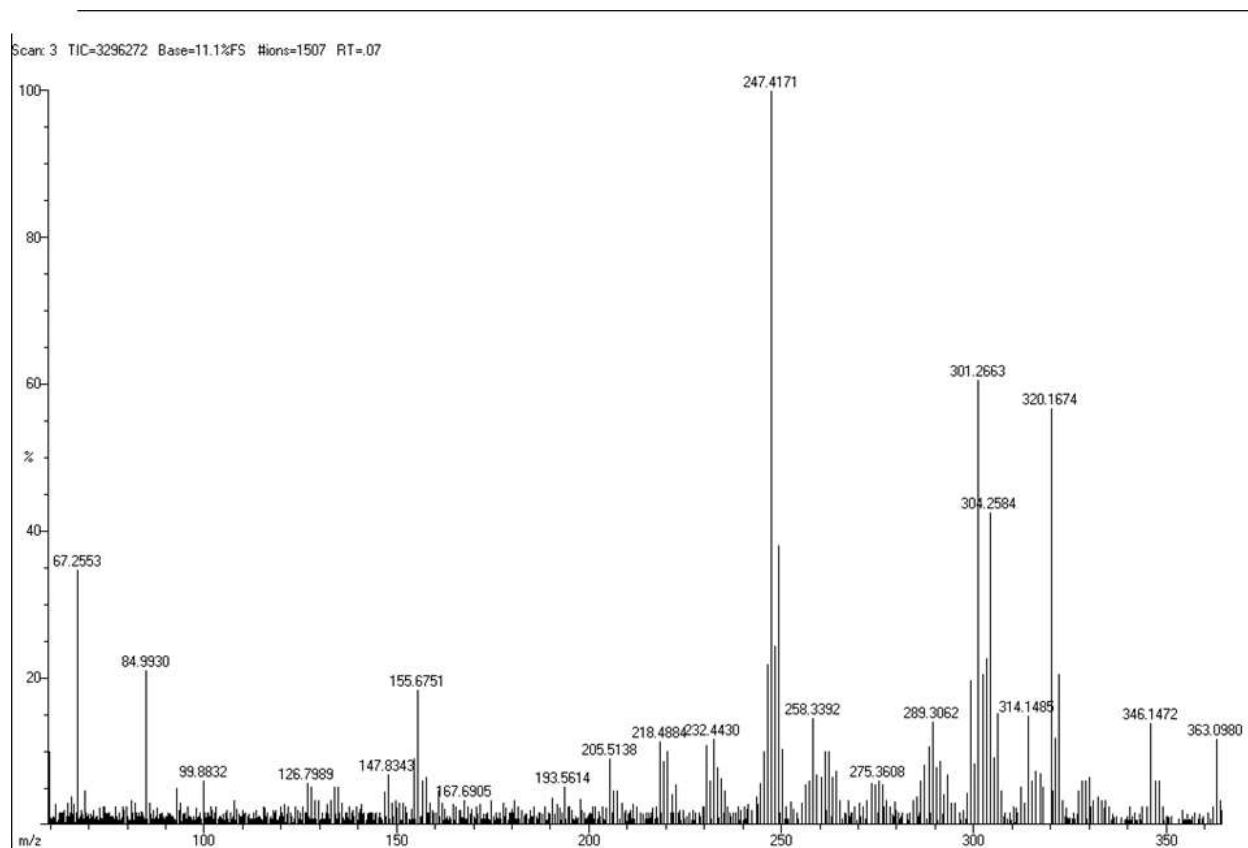
EI-MS spectrum of compound 5i



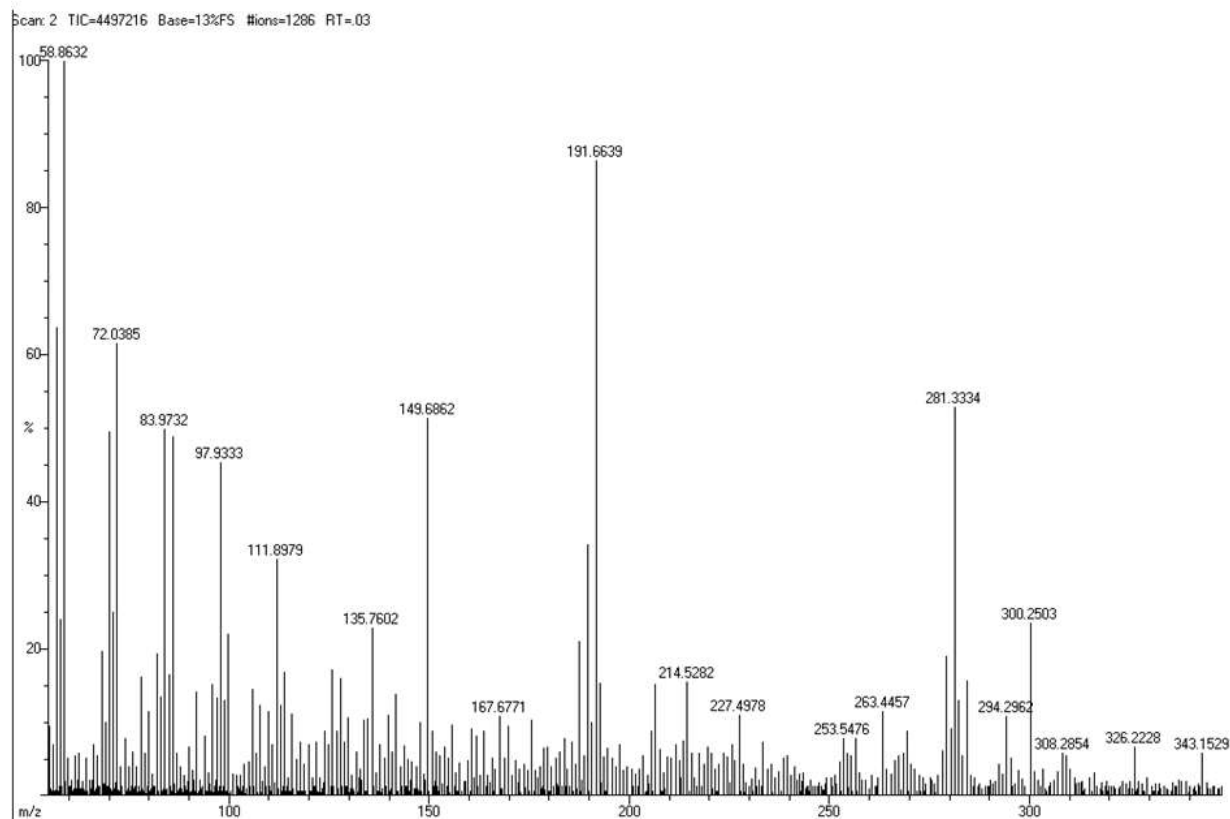
EI-MS spectrum of compound 5j



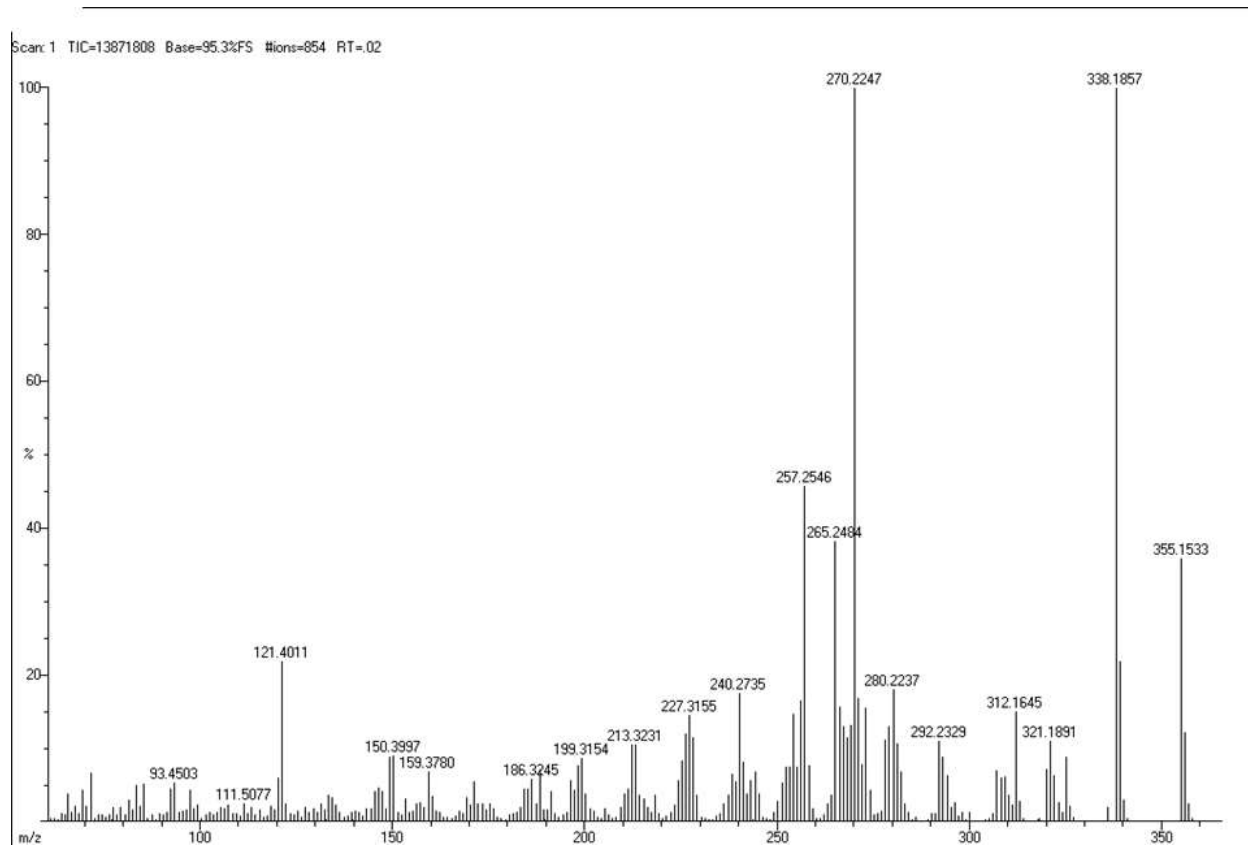
EI-MS spectrum of compound 5k



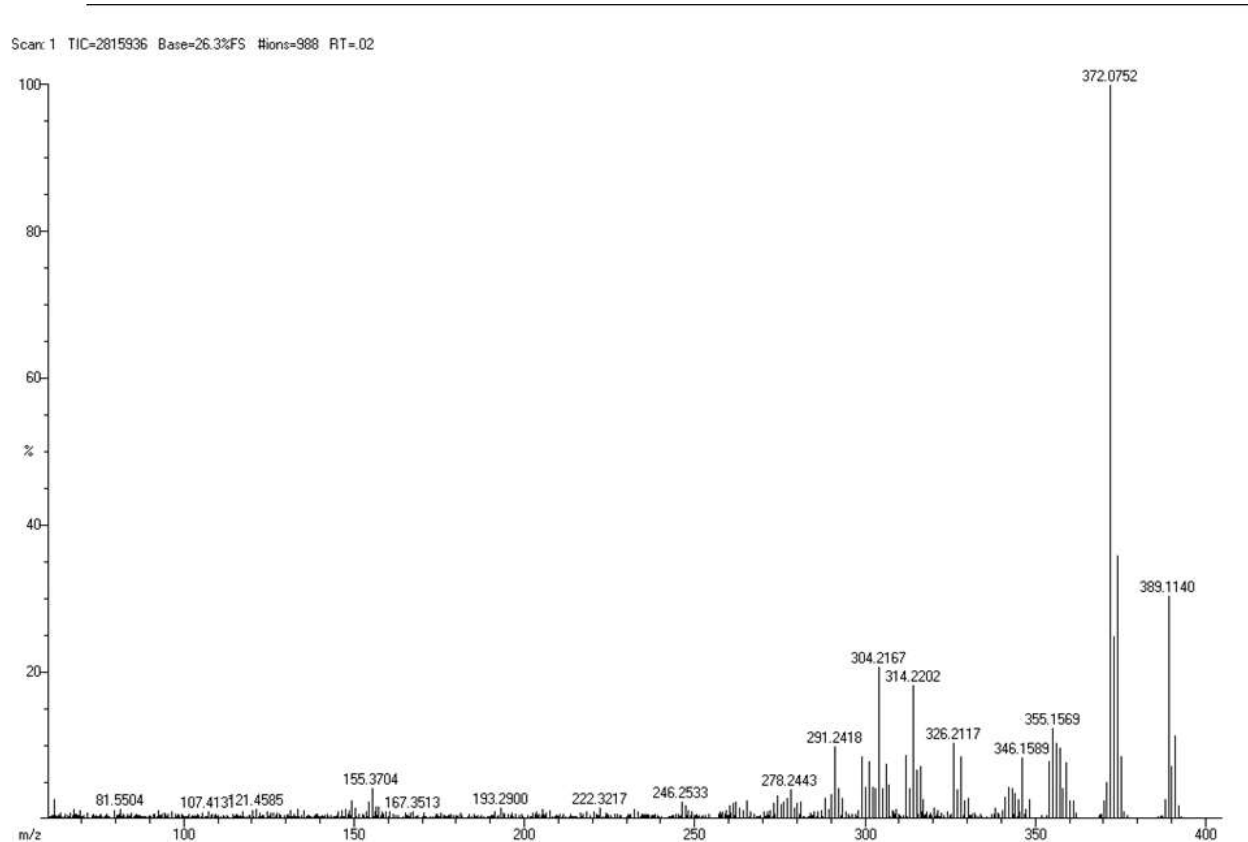
EI-HRMS spectrum of compound 51



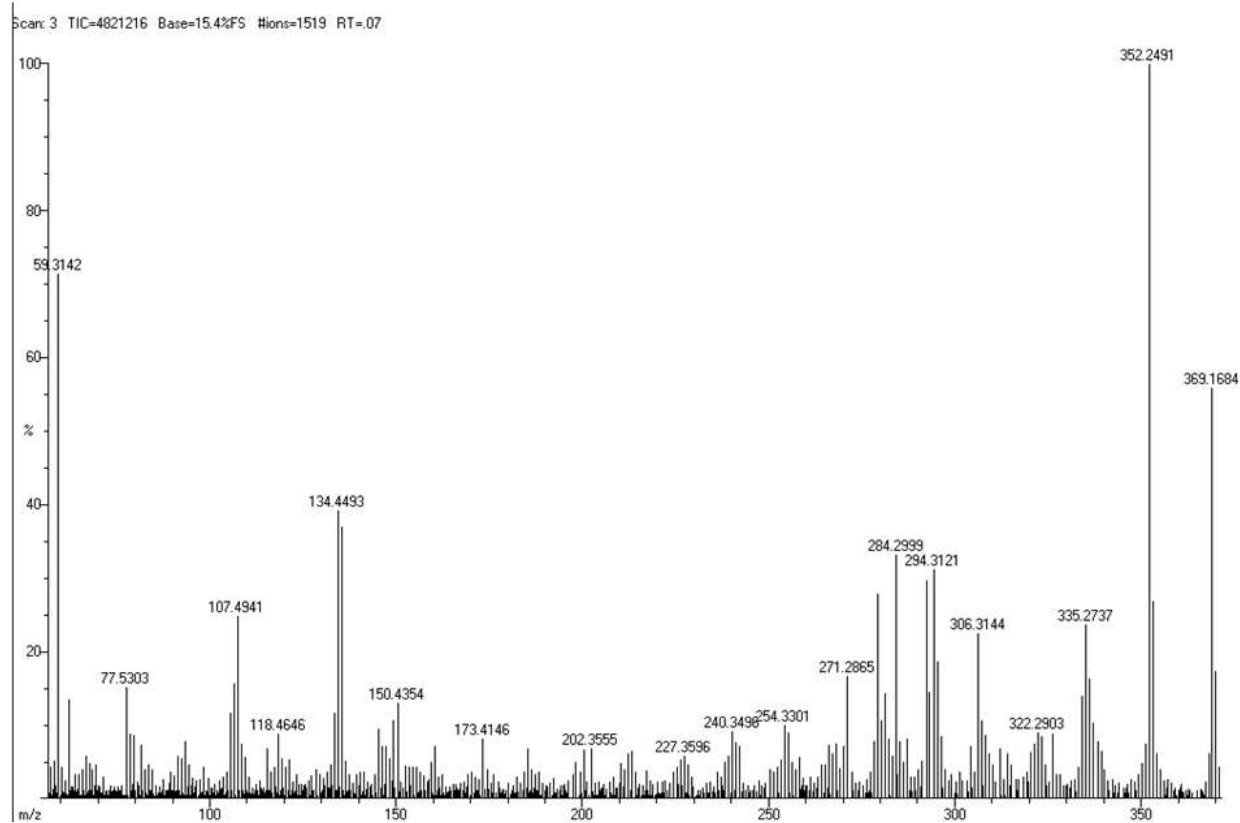
EI-MS spectrum of compound 5m



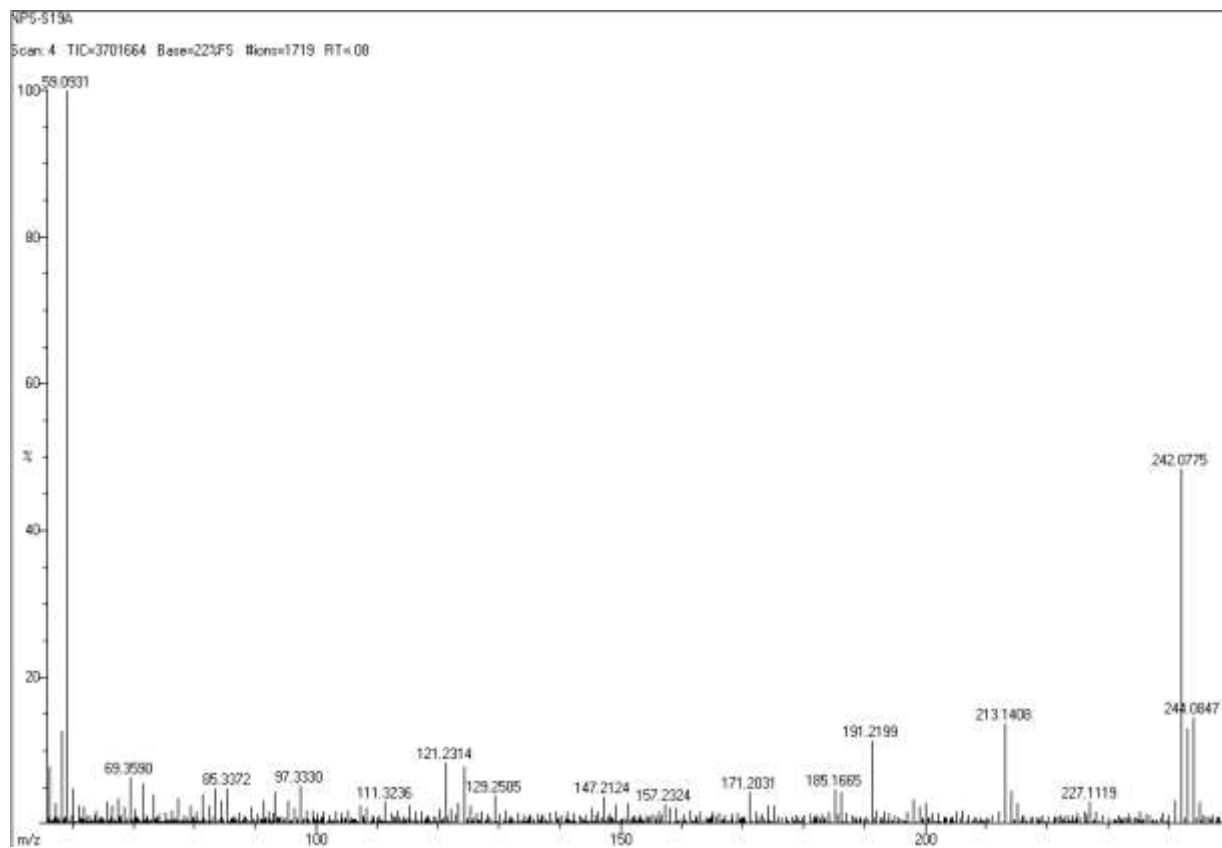
EI-HRMS spectrum of compound 5n



EI-MS spectrum of compound 5o



EI-MS spectrum of compound 5p



EI-MS spectrum of compound 14