Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information

N-heterocyclic carbene (NHC) catalyzed amidation of aldehydes with amines via tandem N-hydroxysuccinimide ester formation

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Experimental details

General considerations

^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature using 400 MHz (Jeol) spectrometer and referenced internally to residual solvent resonances (for $CDCl_3$, ^{1}H at δ 7.26, $^{13}C\{1H\}$ at δ 77.16). The data are reported as follows: chemical shift in ppm on the δ scale, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz) and integration. Mass analysis was carried out using Waters Xevo TQD System Liquid chromatography-mass spectrometer. Melting points are reported uncorrected and measured on BUCHI M-565 melting point apparatus. Analytical thin layer chromatography was performed on 0.25 mm extra hard silica gel plates with UV-254 fluorescent indicator. Liquid chromatography was performed on 230-400 mesh silica gel (SiO2). All reactions were carried out under an atmosphere of nitrogen in glassware, which had been oven-dried as per standard procedure. Unless otherwise noted, all reagents were commercially obtained and, where appropriate, purified prior to use. All physical data were in accordance with the data reported in the literature.

General procedure for NHS-ester synthesis

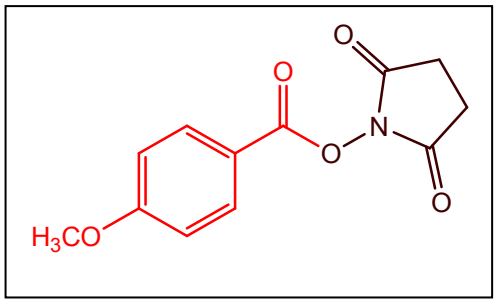
To an oven dried Schlenk tube, base NaH (10mol%) was added to a solution of 1,3-Dimesitylimidazolium chloride (10mol%) in acetonitrile (3ml) under the atmosphere of nitrogen. The resulting mixture was stirred vigorously for about 15 minutes. After that aldehyde (2.5mmol), NHS (2.5mmol) and oxidant *tert*-butyl hydroperoxide (TBHP) (3 equiv) was added to the flask. The reaction mixture was heated to reflux temperature for eight hours in an oil bath. After that reaction was cooled to room temperature and washed with water. Organic portion was extracted by ethylacetate, dried over sodium sulphate and purified by column chromatrography using ethylacetate and hexane as an eluent to yield the desired ester (3a-m).

General procedure for amide synthesis

To an oven dried Schlenk tube, base NaH (10mol%) was added to a solution of 1,3-Dimesitylimidazolium chloride (10mol%) in acetonitrile (3ml) under the atmosphere of nitrogen. The resulting mixture was stirred vigorously for about 15 minutes. After that aldehyde (2.5mmol), NHS (2.5mmol) and oxidant *tert*-butyl

hydroperoxide (TBHP) (3 equiv) was added to the flask. The reaction mixture was heated to reflux temperature for eight hours in an oil bath. After the disappearance of NHS on TLC, the amine (2mmol) was added to the reaction mixture in one portion and was allowed to stir at room temperature for 6h to 8h. After that reaction was washed with water. Organic portion was extracted by ethylacetate, dried over sodium sulphate and purified by column chromatrography using ethylacetate and hexane as an eluent to yield the desired amide 4a-p.

2,5-dioxopyrrolidin-1-yl-4-methoxybenzoate (3a)[1]



White solid, m.p- 1423-145 ˚C yield – 85%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.11 – 8.03 (m, 2H), 6.98 – 6.92 (m, 2H), 3.87 (s, 3H), 2.88 (s, 4H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.67, 165.01, 161.60, 132.99, 117.14, 114.32, 55.72, 25.77, LC-MS : *m/z* calculated $[M+H]^{+}$ = 250.0612, found 250.0615.

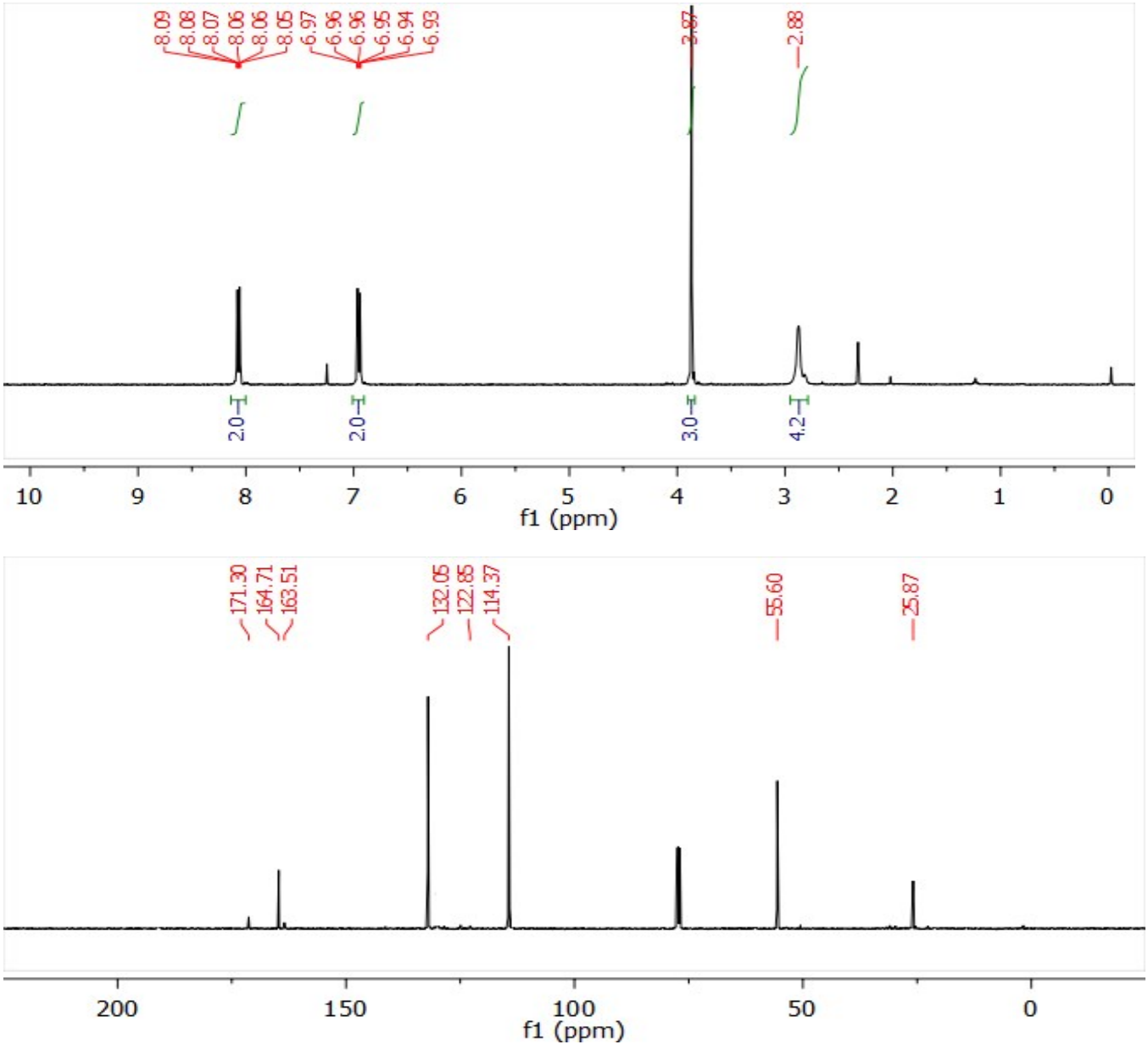
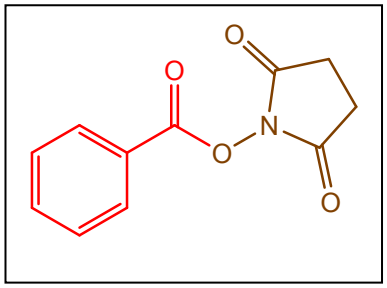


Figure: ^{1}H and ^{13}C spectra of compound 3a.

2,5-dioxopyrrolidin-1-yl-benzoate (3b)[2]



White solid, m.p- 135-139 ˚C yield – 83%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.11 (dd, *J*=8.4, 1.3 Hz, 2H), 7.66 (t, *J*=7.5 Hz, 1H), 7.52 – 7.46 (m, 2H), 2.88 (s, 4H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.48, 162.00, 135.06, 130.67, 128.98, 125.20, 25.78, LC-MS : *m/z* calculated $[M+H]^{+}$ = 220.0522, found 220.0530.

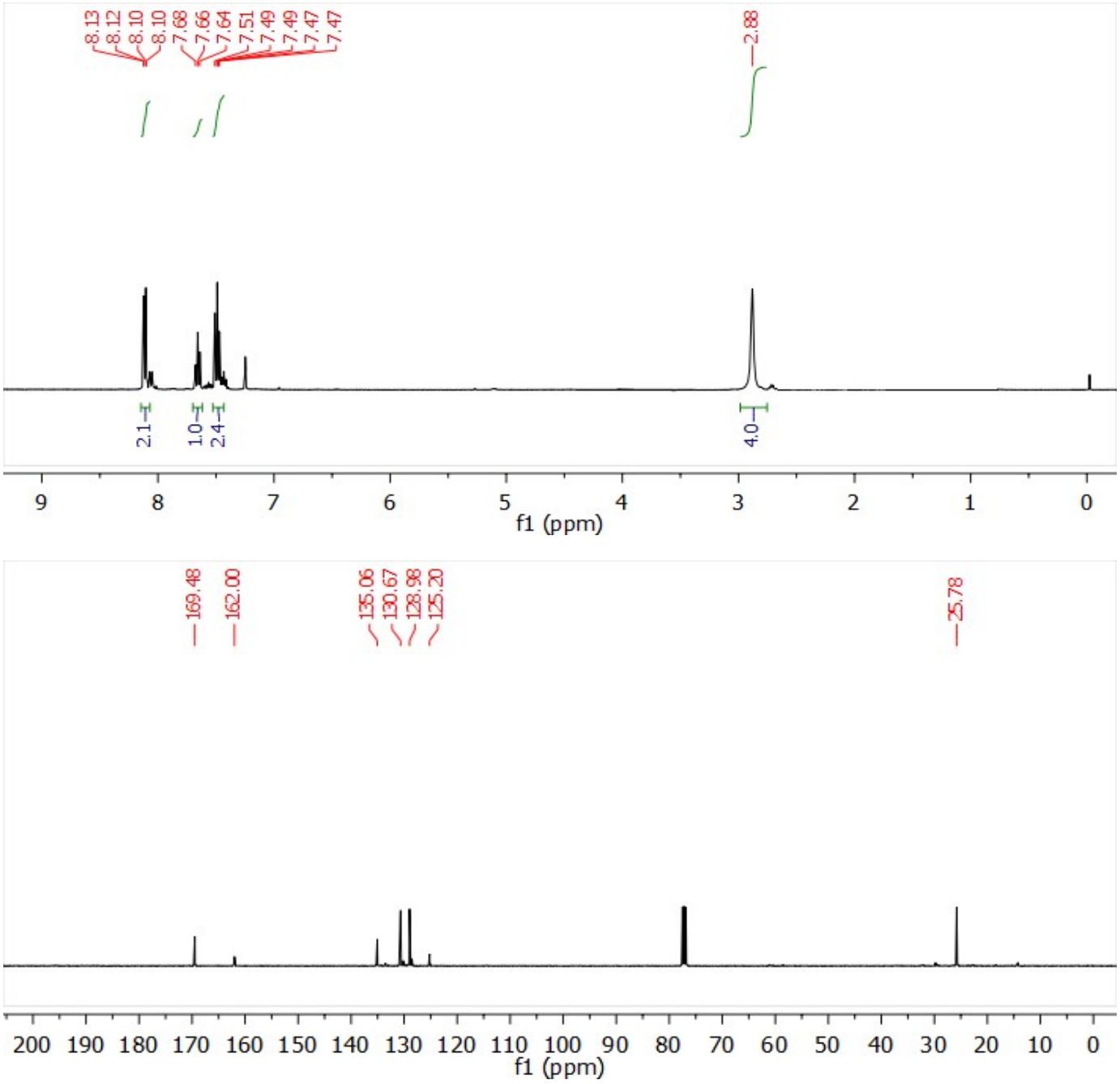
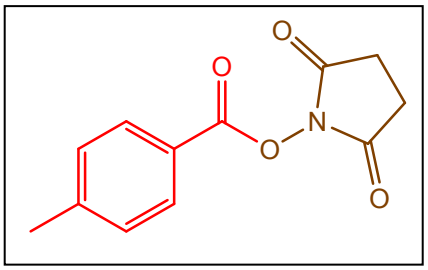


Figure: ^{1}H and ^{13}C spectra of compound 3b.

2,5-dioxopyrrolidin-1-yl-4-methylbenzoate (3c)[3]



White solid, m.p- 180-181 ˚C yield – 84%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.01 (d, *J*=8.2 Hz, 2H), 7.29 (d, *J*=8.0 Hz, 2H), 2.89 (s, 4H), 2.43 (s, 3H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.50, 161.99, 146.23, 130.74, 129.68, 122.34, 25.78, 22.03, LC-MS : *m/z* calculated $[M+H]^{+}$ = 234.0743, found 234.0751.

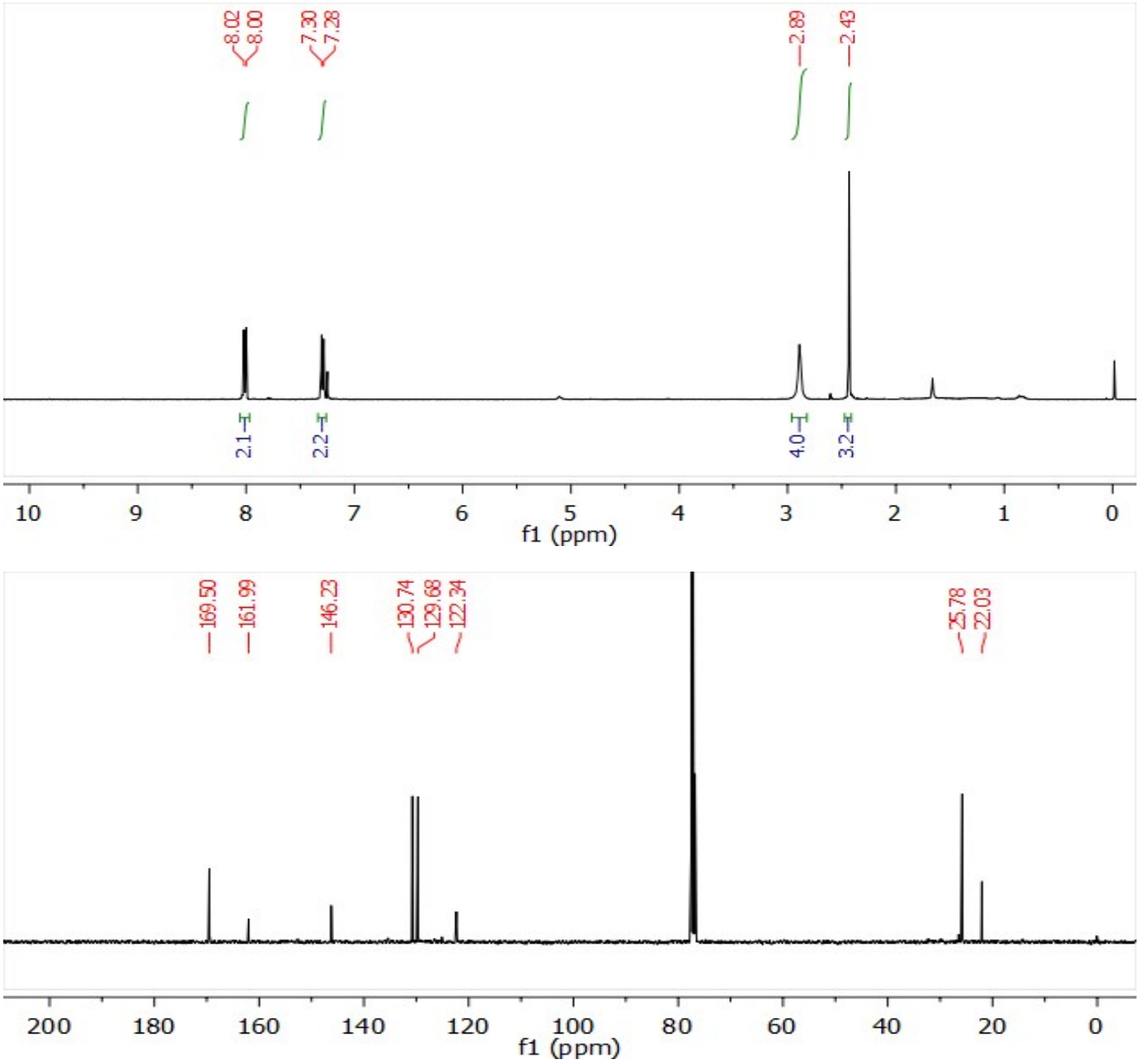
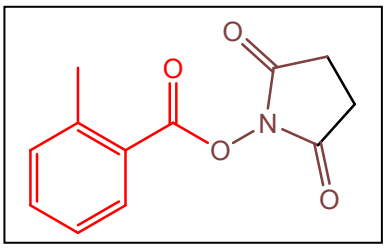


Figure: ^{1}H and ^{13}C spectra of compound 3c.

2,5-dioxopyrrolidin-1-yl-2-methylbenzoate (3d)[4]



White solid, m.p- 135-137 ˚C yield – 82%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.11 (dd, *J*=8.2, 1.3 Hz, 1H), 7.51 (td, *J*=7.6, 1.3 Hz, 1H), 7.30 (t, *J*=7.6 Hz, 2H), 2.90 (s, 4H), 2.61 (s, 3H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.54, 162.19, 142.24, 134.13, 132.09, 131.44, 126.21, 124.32, 25.80, 21.72, LC-MS : *m/z* calculated $[M+H]^{+}$ = 234.0736, found 234.0742.

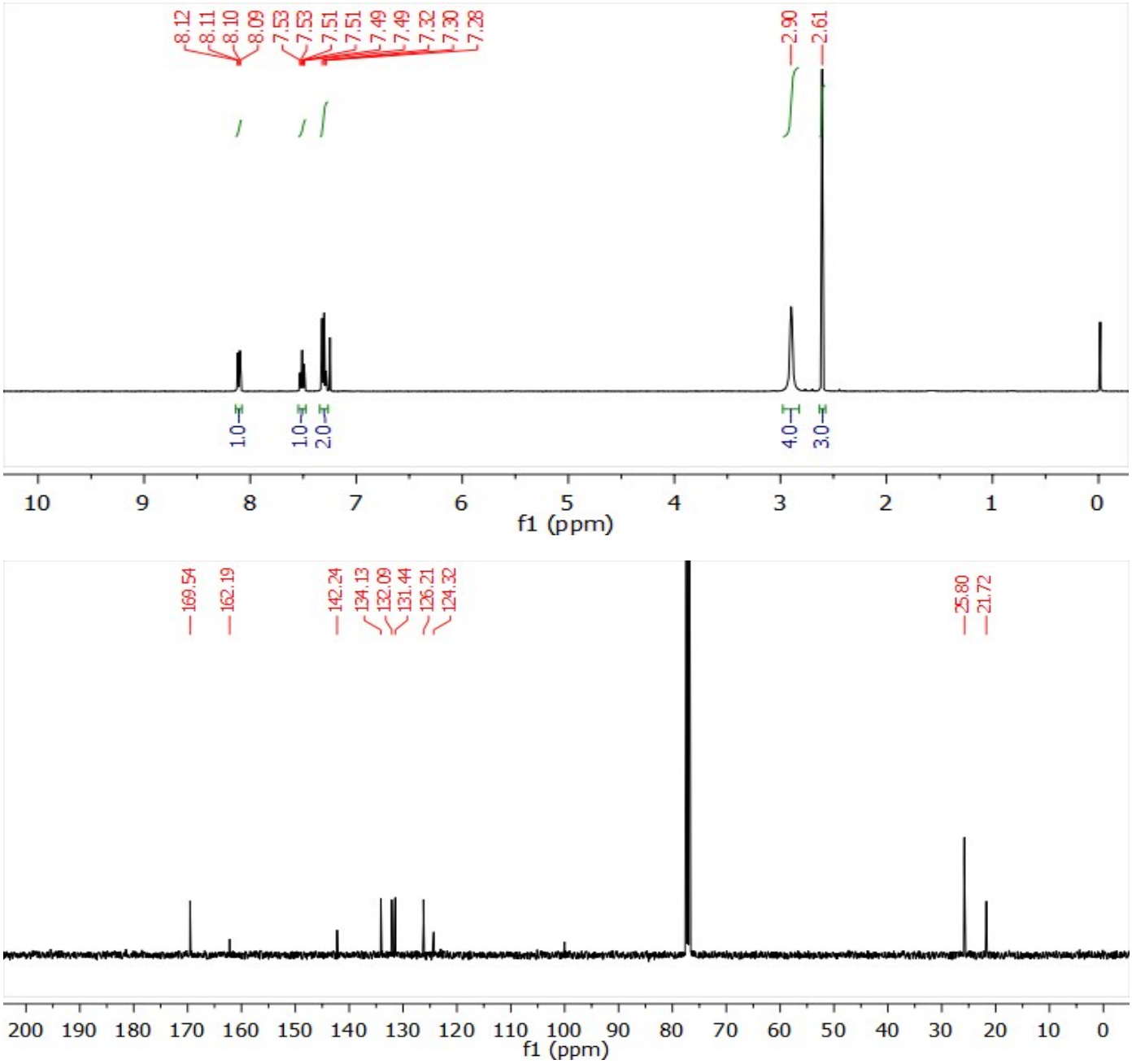
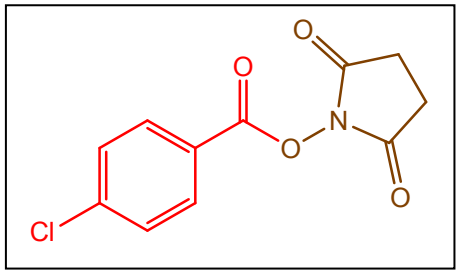


Figure: ^{1}H and ^{13}C spectra of compound 3d.

2,5-dioxopyrrolidin-1-yi-4-chlorobenzoate (3e)[3]



White solid, m.p- 207-208 ˚C yield – 82%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.10 – 8.04 (m, 2H), 7.54 – 7.46 (m, 2H), 2.91 (s, 4H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.23, 161.24, 141.83, 132.02, 129.44, 123.63, 25.76, LC-MS : *m/z* calculated $[M+H]^{+}$ = 254.1076, found 254.1072.

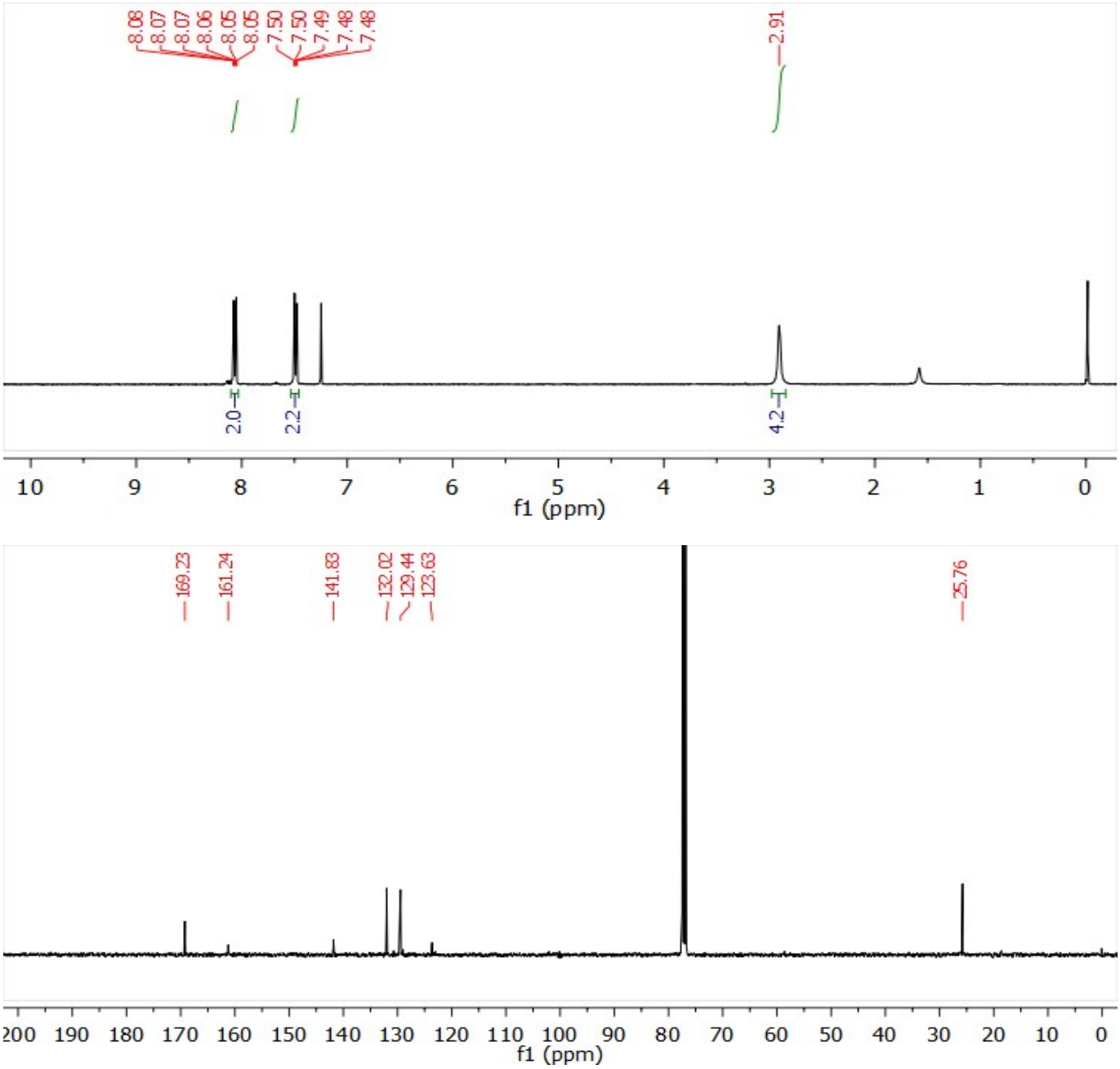
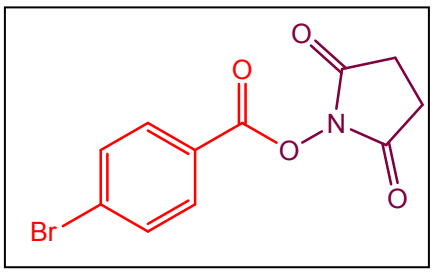


Figure: ^{1}H and ^{13}C spectra of compound 3e.

2,5-dioxopyrrolidin-1-yl-4-bromobenzoate (3f)[4]



White solid, m.p- 141-143 ˚C yield – 80%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.01 – 7.96 (m, 2H), 7.68 – 7.63 (m, 2H), 2.90 (s, 4H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.21, 161.42, 132.44, 132.03, 130.60, 124.09, 25.76, LC-MS : *m/z* calculated $[M+H]^{+}$ = 297.9608, found 297.9612.

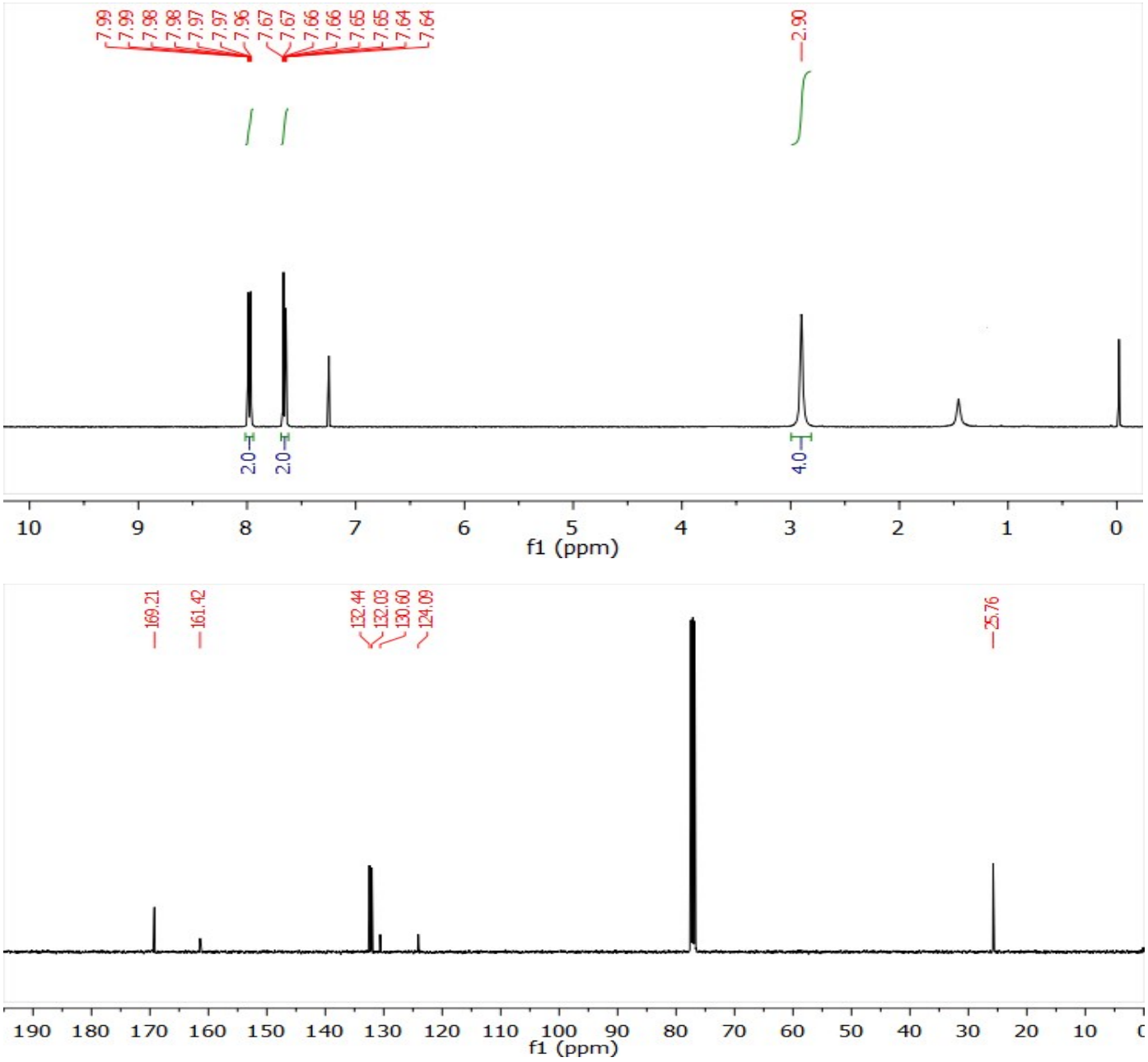
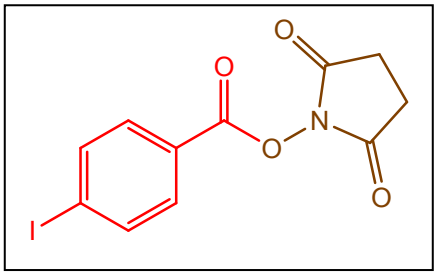


Figure: ^{1}H and ^{13}C spectra of compound 3f.

2,5-dioxopyrrolidin-1-yl-4-iodobenzoate (3g)



White solid, yield – 76%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.91 – 7.79 (m, 4H), 2.90 (s, 4H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.23, 161.71, 138.42, 131.79, 124.62, 100.01, 77.45, 25.77, LC-MS : *m/z* calculated $[M+H]^{+}$ = 345.9581, found 345.9575.

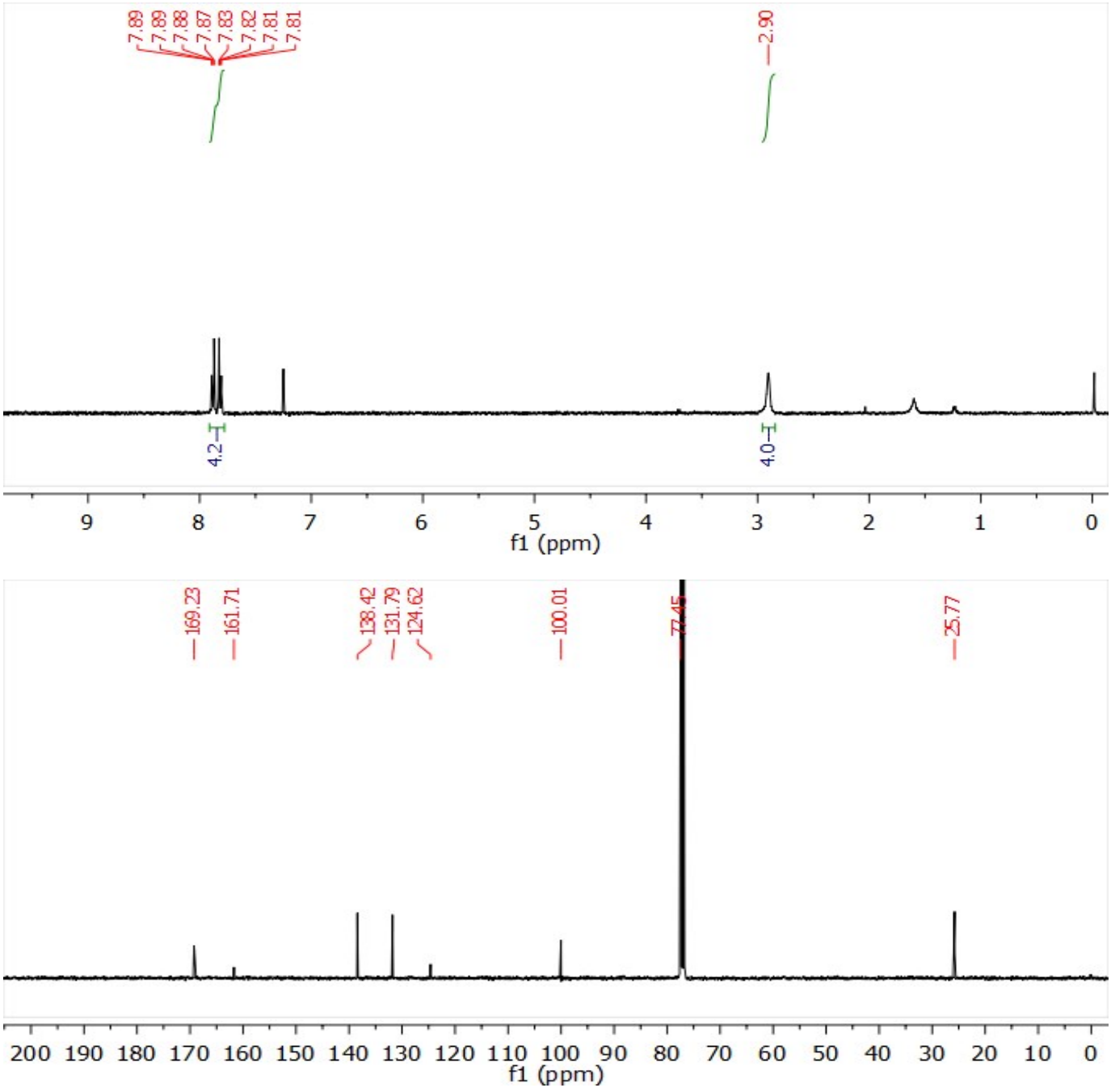
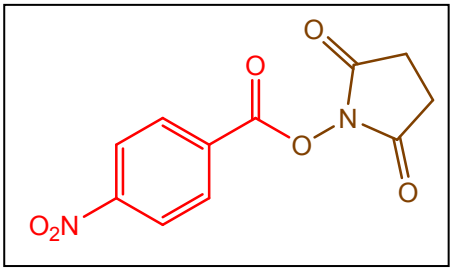


Figure: ^{1}H and ^{13}C spectra of compound 3g.

2,5-dioxopyrrolidin-1-yl 4-nitrobenzoate (3h)



White solid, yield – 71%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.50 – 8.01 (m, 4H), 2.94 (s, 4H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 168.87, 160.42, 151.61, 131.90, 130.78, 124.07, 25.76, LC-MS : *m/z* calculated $[M+H]^{+}$ = 265.1463, found 265.1466.

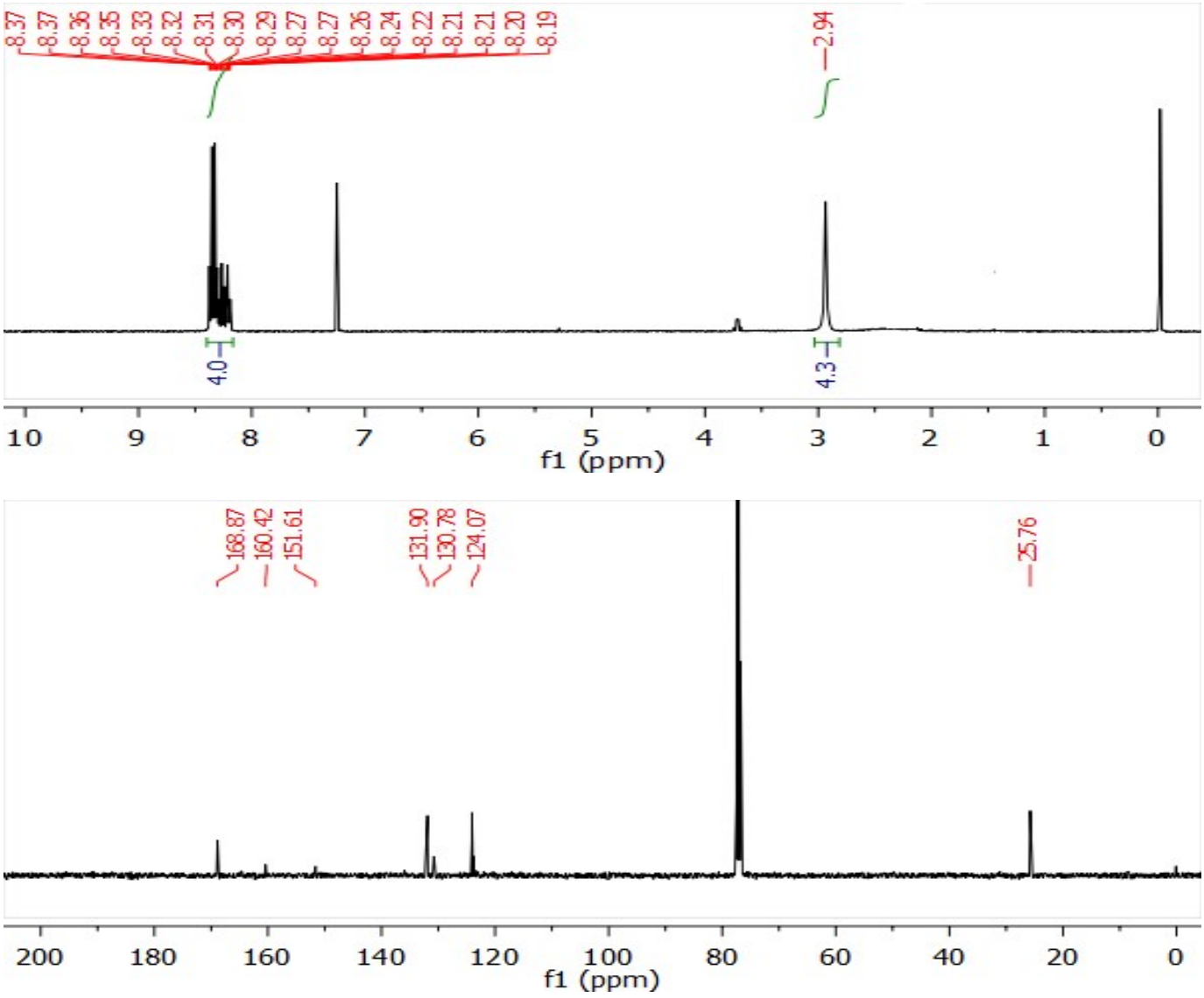
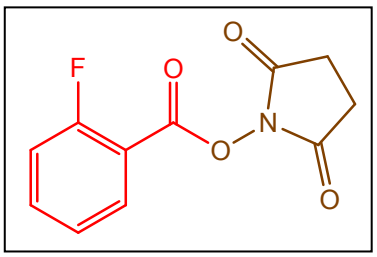


Figure: ^{1}H and ^{13}C spectra of compound 3h.

2,5-dioxopyrrolidin-1-yl-2-fluorobenzoate (3i)[3]



White solid, m.p. – 113-114 ˚C, yield – 74%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.09 – 8.01 (m, 1H), 7.68 – 7.60 (m, 1H), 7.31 – 7.16 (m, 2H), 2.88 (s, 4H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.37, 163.88, 161.25, 137.01, 132.71, 124.61, 117.60, 113.74, 25.76, LC-MS : *m/z* calculated $[M+H]^{+}$ = 238.0452, found 238.0450.

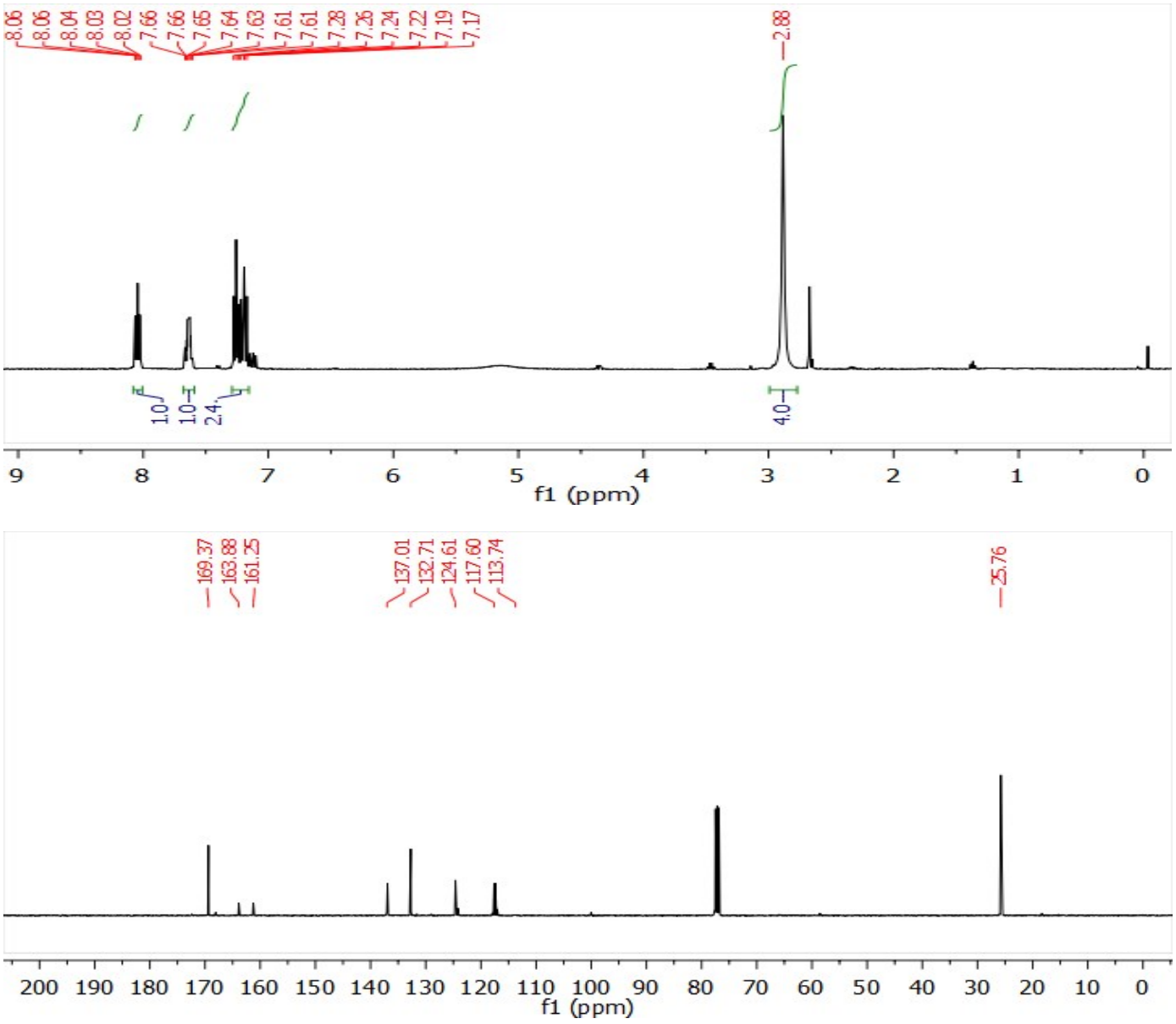
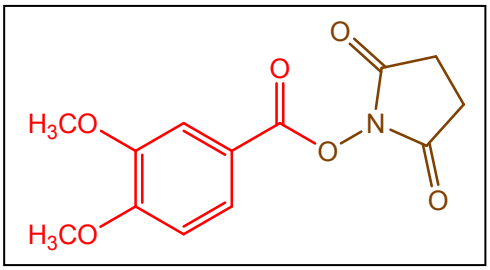


Figure: ^{1}H and ^{13}C spectra of compound 3i.

2,5-dioxopyrrolidin-1-yl-3,4-dimethoxybenzoate (3j)



White solid, m.p. – 126-128˚C, yield – 86%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.76 (dd, *J*=8.4, 2.0 Hz, 1H), 7.58 (d, *J*=1.9 Hz, 1H), 6.90 (d, *J*=8.5 Hz, 1H), 3.93 (s, 6H), 2.72 (s, 4H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 173.32, 171.99, 153.77, 148.72, 124.68, 121.81, 112.33, 110.38, 56.09, 27.43, LC-MS : *m/z* calculated $[M+H]^{+}$ = 280.1705, found 280.1720.

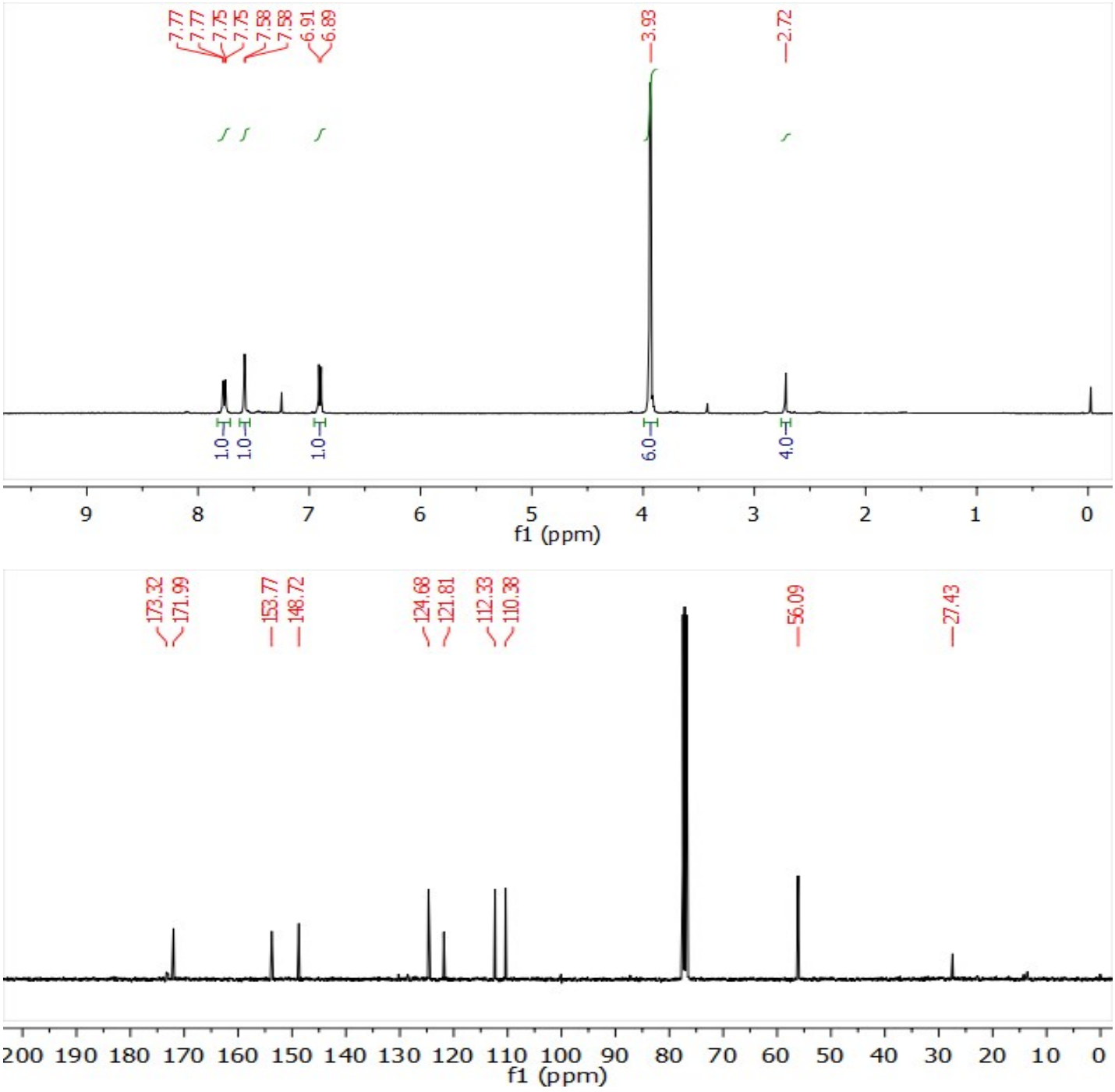
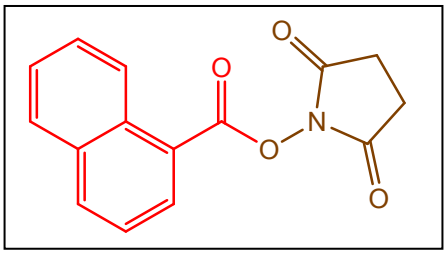


Figure: ^{1}H and ^{13}C spectra of compound 3j.

2,5-dioxopyrrolidin-1-yl-1-naphthooate (3k)[2]



White solid, m.p. – 120-122 ˚C, yield – 81%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.84 – 8.77 (m, 1H), 8.45 (dd, *J*=7.3, 1.2 Hz, 1H), 8.14 (d, *J*=8.2 Hz, 1H), 7.95 – 7.88 (m, 1H), 7.69 – 7.52 (m, 3H), 2.94 (d, *J*=4.6 Hz, 4H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.61, 162.23, 135.72, 133.81, 132.00, 131.56, 128.91, 128.85, 126.94, 125.38, 124.58, 121.60, 25.86, LC-MS : *m/z* calculated $[M+H]^{+}$ = 270.0728, found 270.0736.

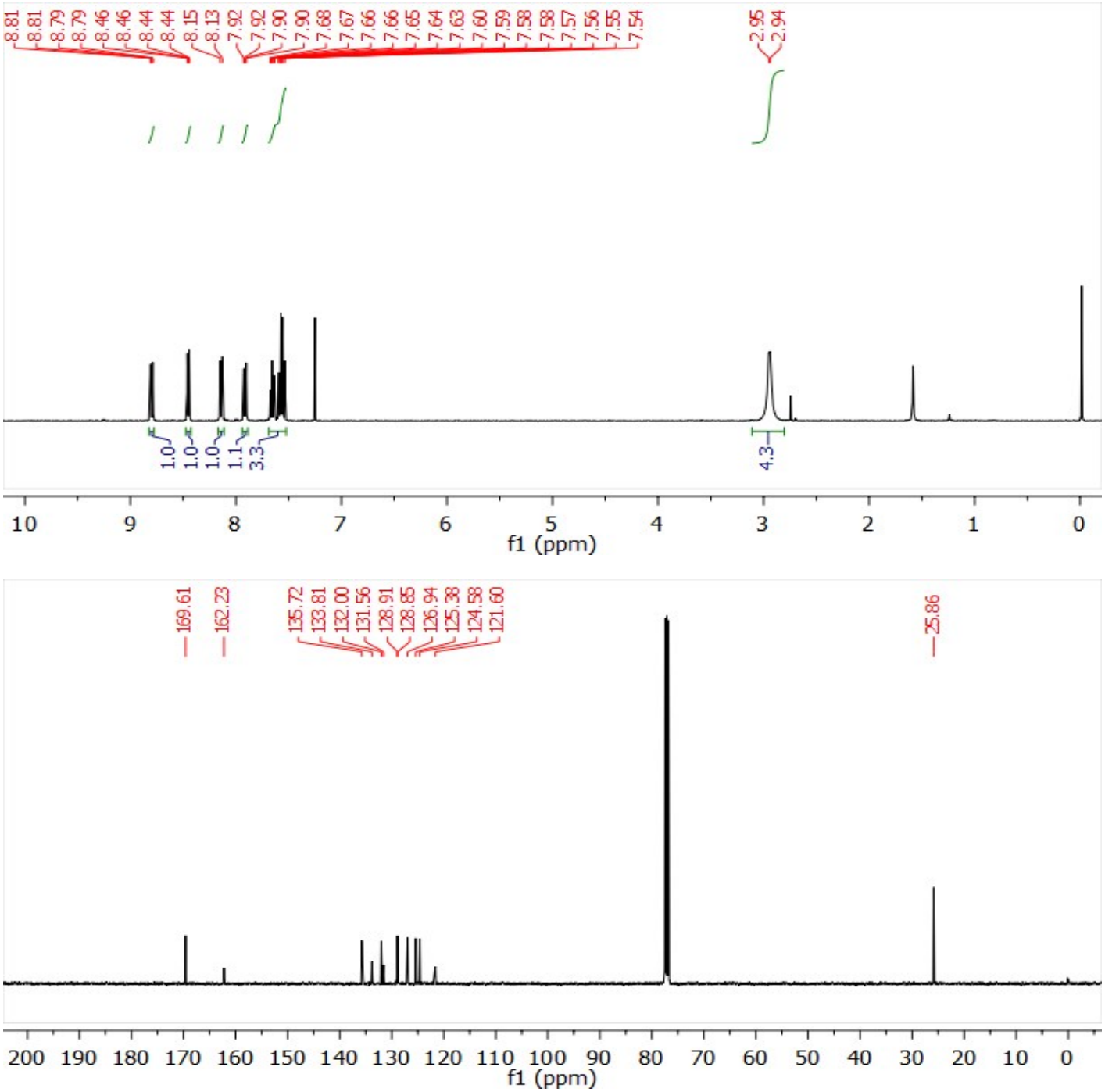
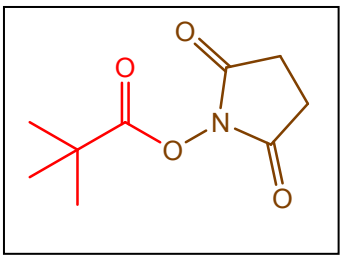


Figure: ^{1}H and ^{13}C spectra of compound 3k.

2,5-dioxopyrrolidin-1-yl-pivalate (3l)



Yellow liquid, yield – 87%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 2.80 (s, 4H), 1.36 (s, 9H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 173.55, 169.43, 38.46, 27.13, 25.71, LC-MS : *m/z* calculated $[M+H]^{+}$ = 200.0814, found 200.0817.

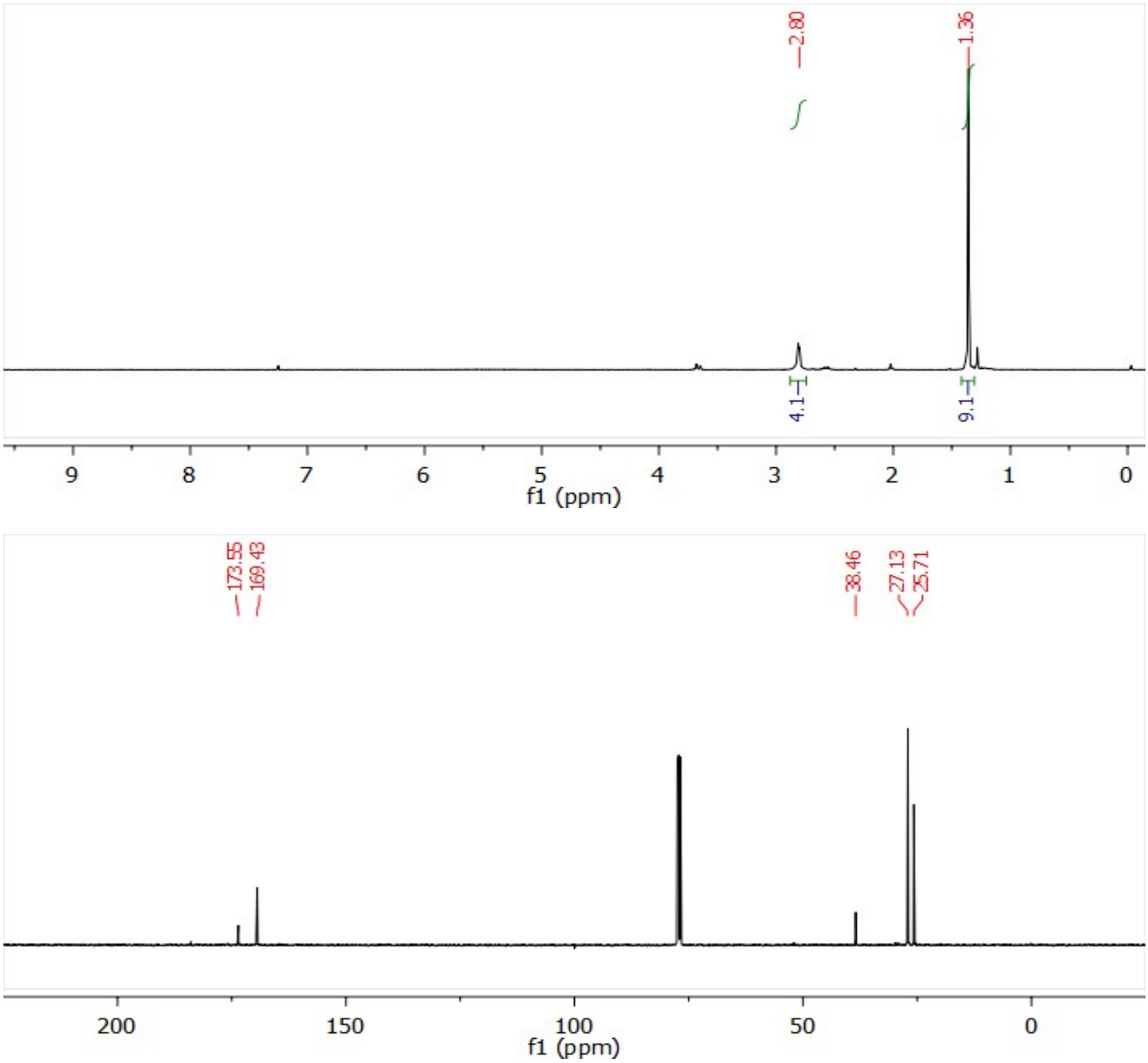
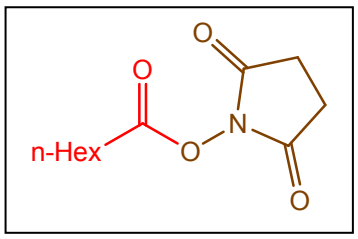


Figure: ^{1}H and ^{13}C spectra of compound 3l.

2,5-dioxopyrrolidin-1-yl-heptanoate (3m)



Yellow liquid, yield – 83%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 2.83 (s, 4H), 2.59 (t, *J*=7.5 Hz, 2H), 1.72 (m, *J*=7.5 Hz, 2H), 1.44 – 1.20 (m, 6H), 0.87 (t, *J*=7.0 Hz, 3H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.38, 168.85, 31.33, 31.03, 28.54, 25.68, 24.61, 22.48, 14.08, LC-MS : *m/z* calculated $[M+H]^{+}$ = 228.1263, found 228.1270.

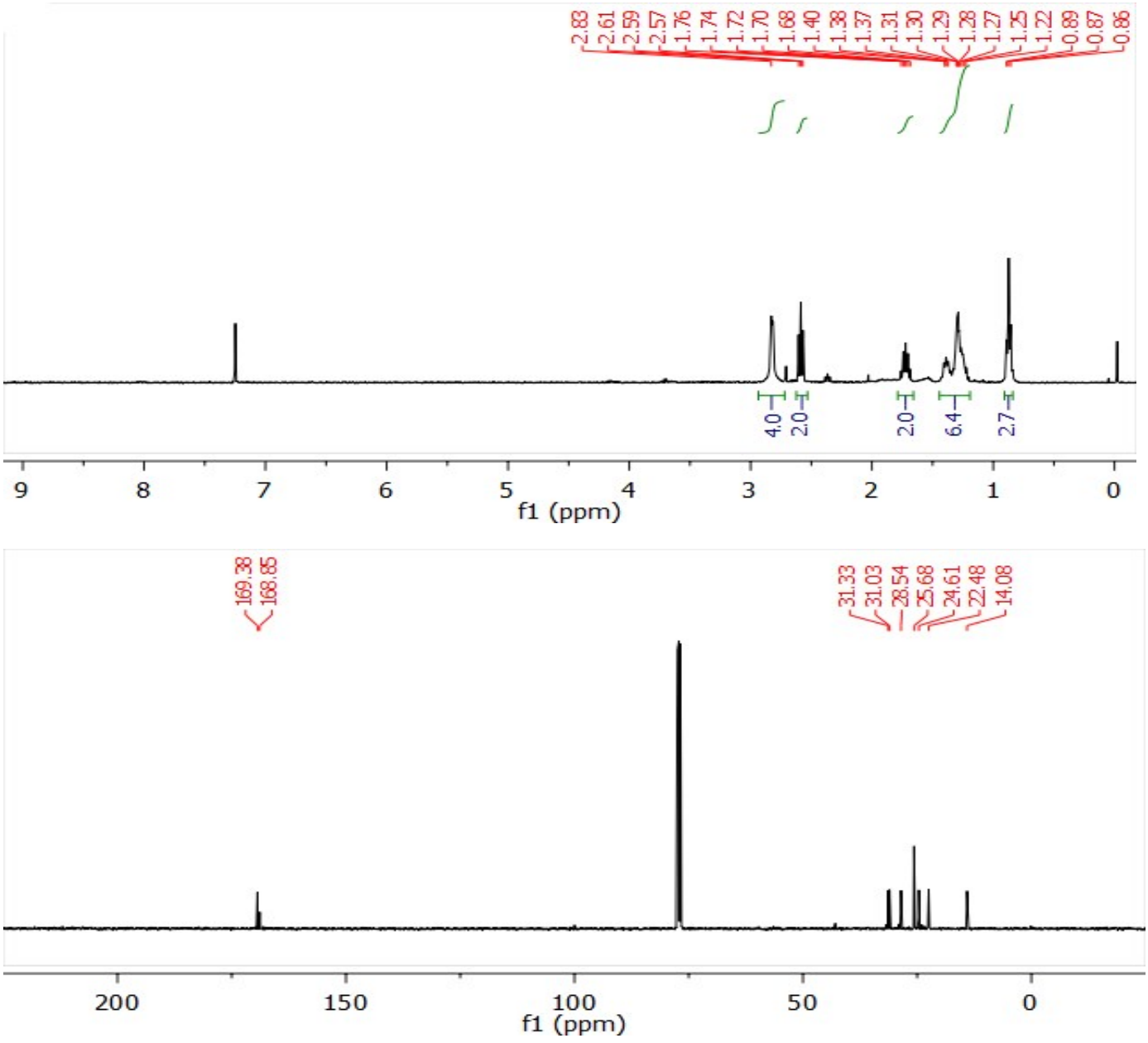
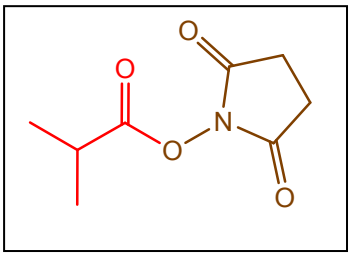


Figure: ^{1}H and ^{13}C spectra of compound 3m.

2,5-dioxopyrrolidin-1-yl-isobutyrate (3n)



White solid, m.p – 46-47˚C, yield – 80%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 2.54 (m, *J*=7.0 Hz, 5H), 1.16 (d, *J*=7.0 Hz, 6H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 183.72, 169.56, 33.92, 28.33, 18.78, LC-MS : *m/z* calculated $[M+H]^{+}$ = 186.0721, found 186.0724.

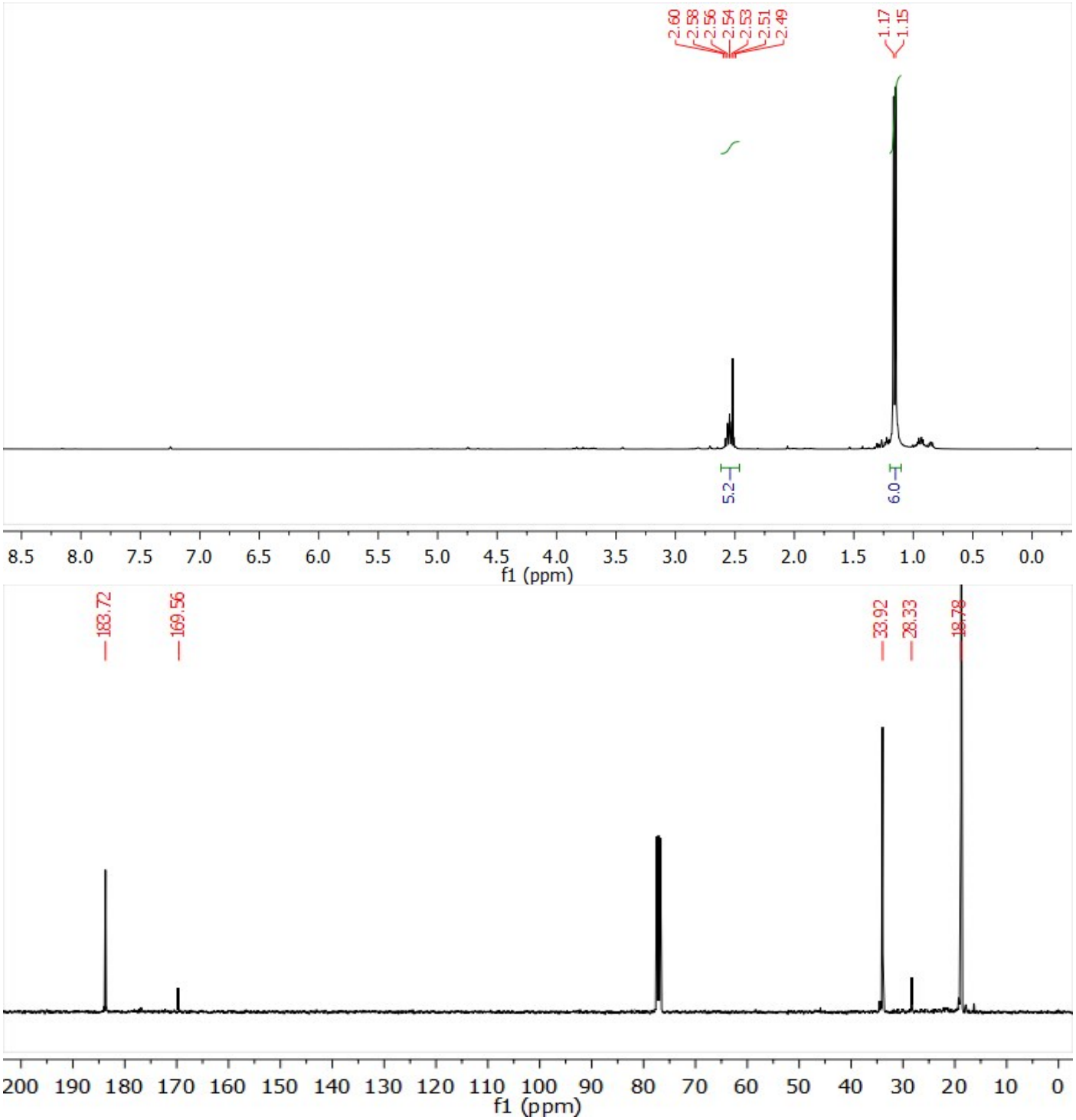
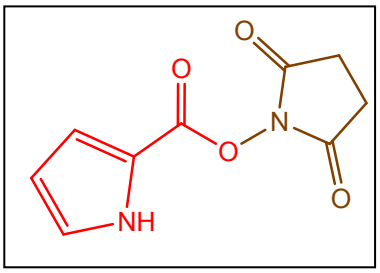


Figure: ^{1}H and ^{13}C spectra of compound 3n.

2,5-dioxopyrrolidin-1-yl 1*H*-pyrrole-2-carboxylate (3o)



Yellow oil, yield – 88%, ^{1}H NMR(DMSO-d_6, 400 MHz): δ (ppm) 7.26 (d, *J*=1.8 Hz, 1H), 7.07 (d, *J*=3.6 Hz, 1H), 6.29 (t, 1H), 2.82 (s, 4H), ^{13}C NMR(DMSO-D_6, 101 MHz): δ (ppm) 168.75, 162.60, 123.12, 122.31, 114.37, 111.50, 25.98, LC-MS : *m/z* calculated $[M+H]^+$ = 209.0511, found 209.0514.

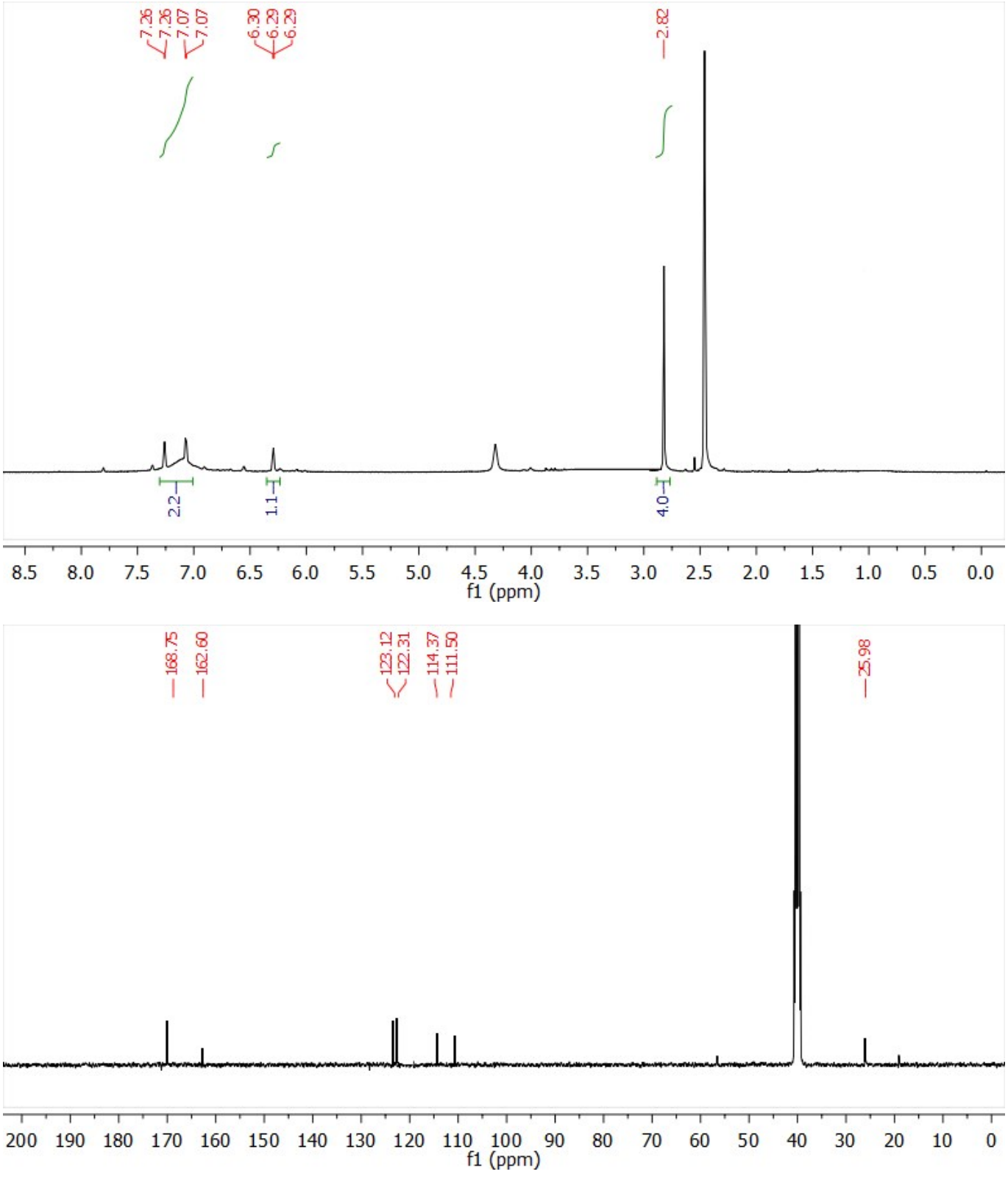
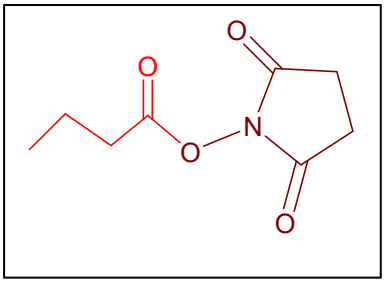


Figure: ^{1}H and ^{13}C spectra of compound 3o.

2,5-dioxopyrrolidin-1-yl-butyrate (3p)



Light yellow oil, yield – 72%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 2.80 (s, 4H), 2.54 (t, *J*=7.3 Hz, 2H), 1.72 (m, *J*=14.8, 7.4 Hz, 2H), 0.99 (t, *J*=7.4 Hz, 3H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.54, 168.67, 32.78, 25.65, 18.26, 13.65, LC-MS : *m/z* calculated $[M+H]^{+}$ = 186.0761, found 186.0764.

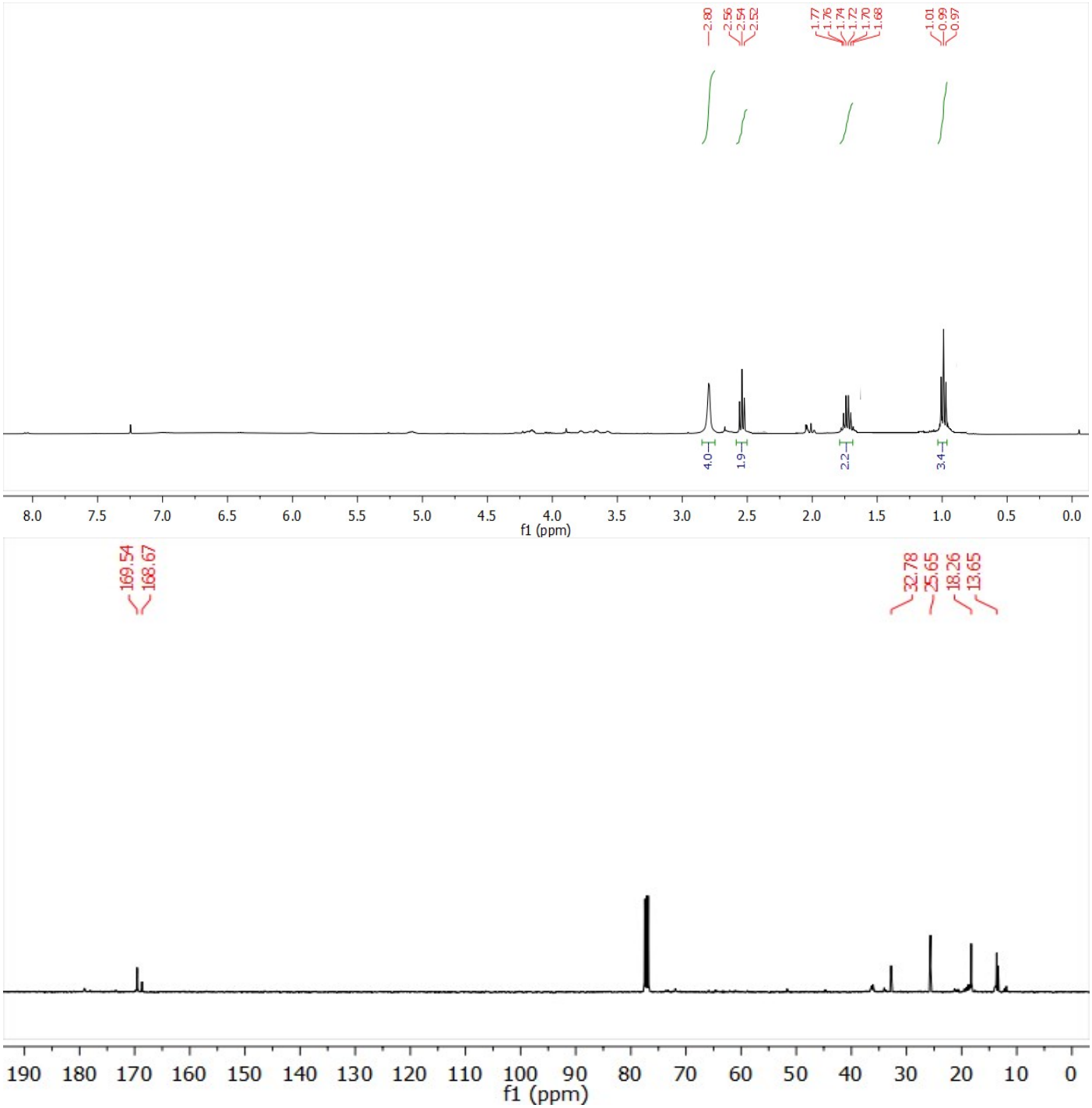
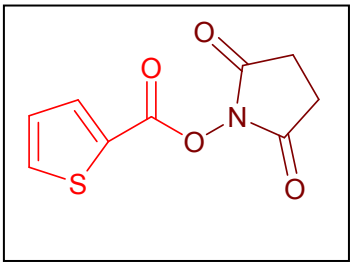


Figure: ^{1}H and ^{13}C spectra of compound 3p.

2,5-dioxopyrrolidin-1,5-yl-thiophen-2-carboxylate (3q)



White solid, m.p -123-125℃, yield – 81%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.99 (dd, *J*=3.8, 1.2 Hz, 1H), 7.75 (dd, *J*=5.0, 1.2 Hz, 1H), 7.20 – 7.14 (m, 1H), 2.86 (s, 4H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.36, 157.46, 136.78, 135.90, 128.55, 126.95, 25.73, LC-MS : *m/z* calculated $[M+H]^{+}$ = 226.0143, found 226.046.

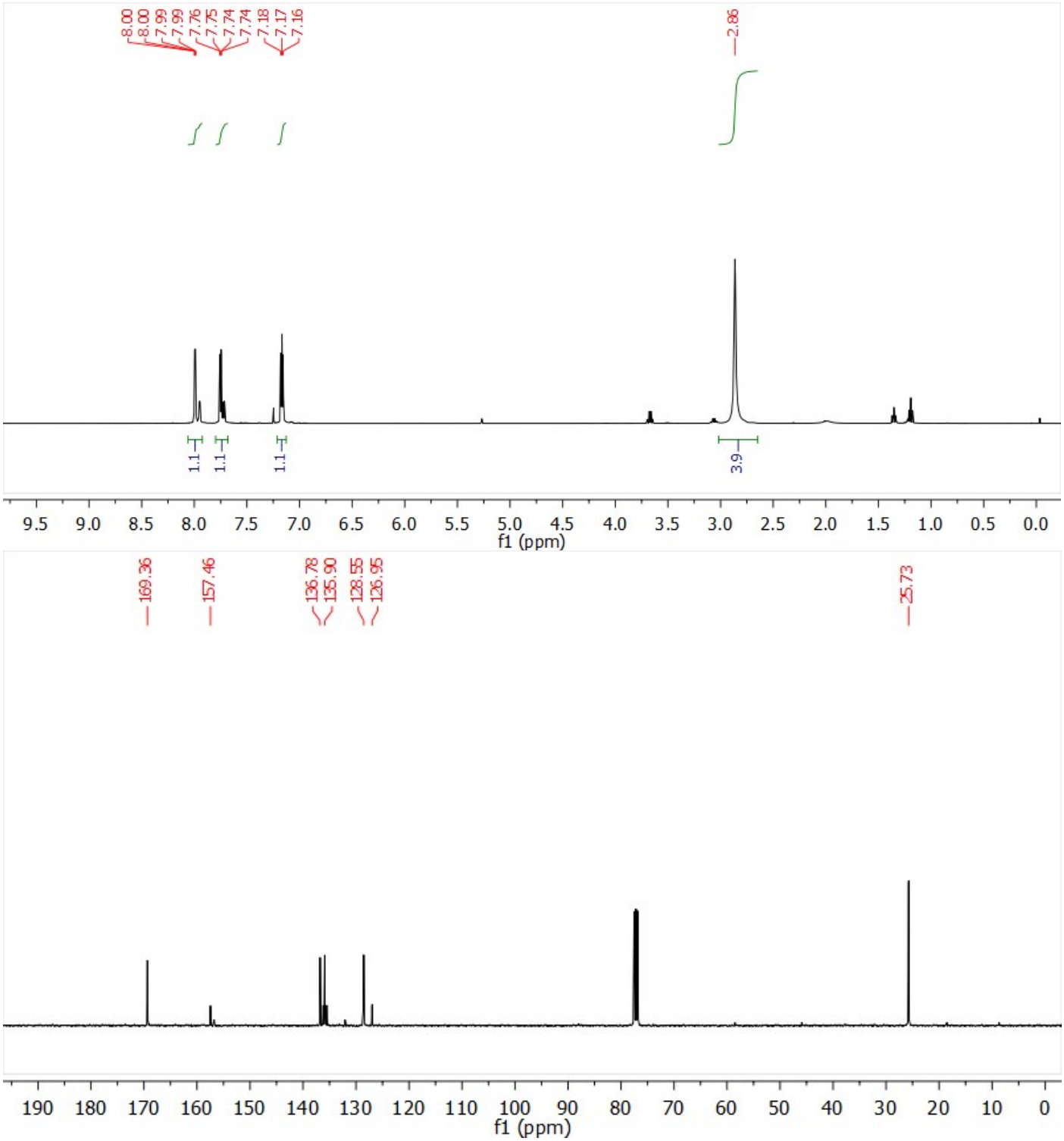
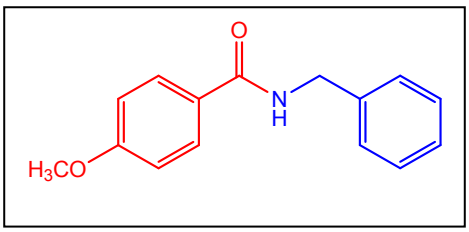


Figure: ^{1}H and ^{13}C spectra of compound 3q.

_N_-benzyl-4-methoxybenzamide (4a)[5]



White solid, m.p - 147-149 °C ; yield - 92%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.78 – 7.72 (m, 2H), 7.38 – 7.25 (m, 5H), 6.95 – 6.87 (m, 2H), 6.34 (brs, 1H), 4.62 (d, *J*=5.6 Hz, 2H), 3.83 (s, 3H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 166.95, 162.33, 138.48, 133.53, 128.86, 128.03, 127.68, 126.71, 113.87, 55.51, 44.18, LC-MS : *m/z* calculated $[M+H]^{+}$ = 242.1881, found = 242.1872.

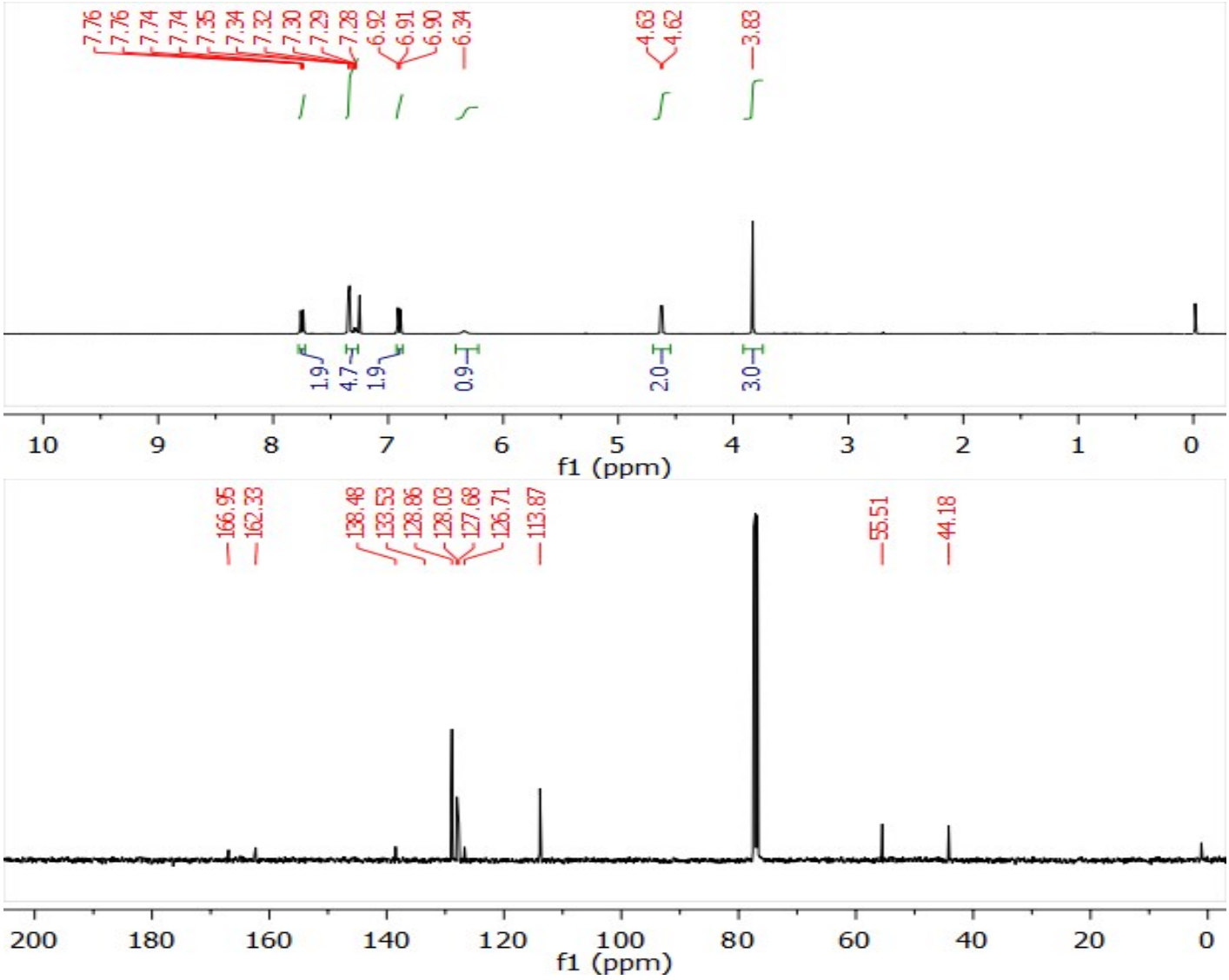
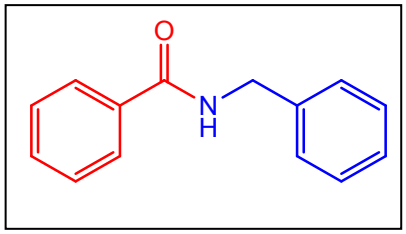


Figure: ^{1}H and ^{13}C spectra of compound 4a.

_N_-benzylbenzamide (4b)[5]



White solid, m.p – 120-122 ˚C; yield - 91%; ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.81 – 7.76 (m, 2H), 7.52 – 7.26 (m, 8H), 6.50 (s, 1H), 4.63 (d, *J*=5.7 Hz, 2H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 167.48, 138.28, 134.46, 131.67, 128.89, 128.70, 128.03, 127.73, 127.07, 44.23, LC-MS : *m/z* calculated $[M+H]^{+}$ =212.1075, found =212.1074.

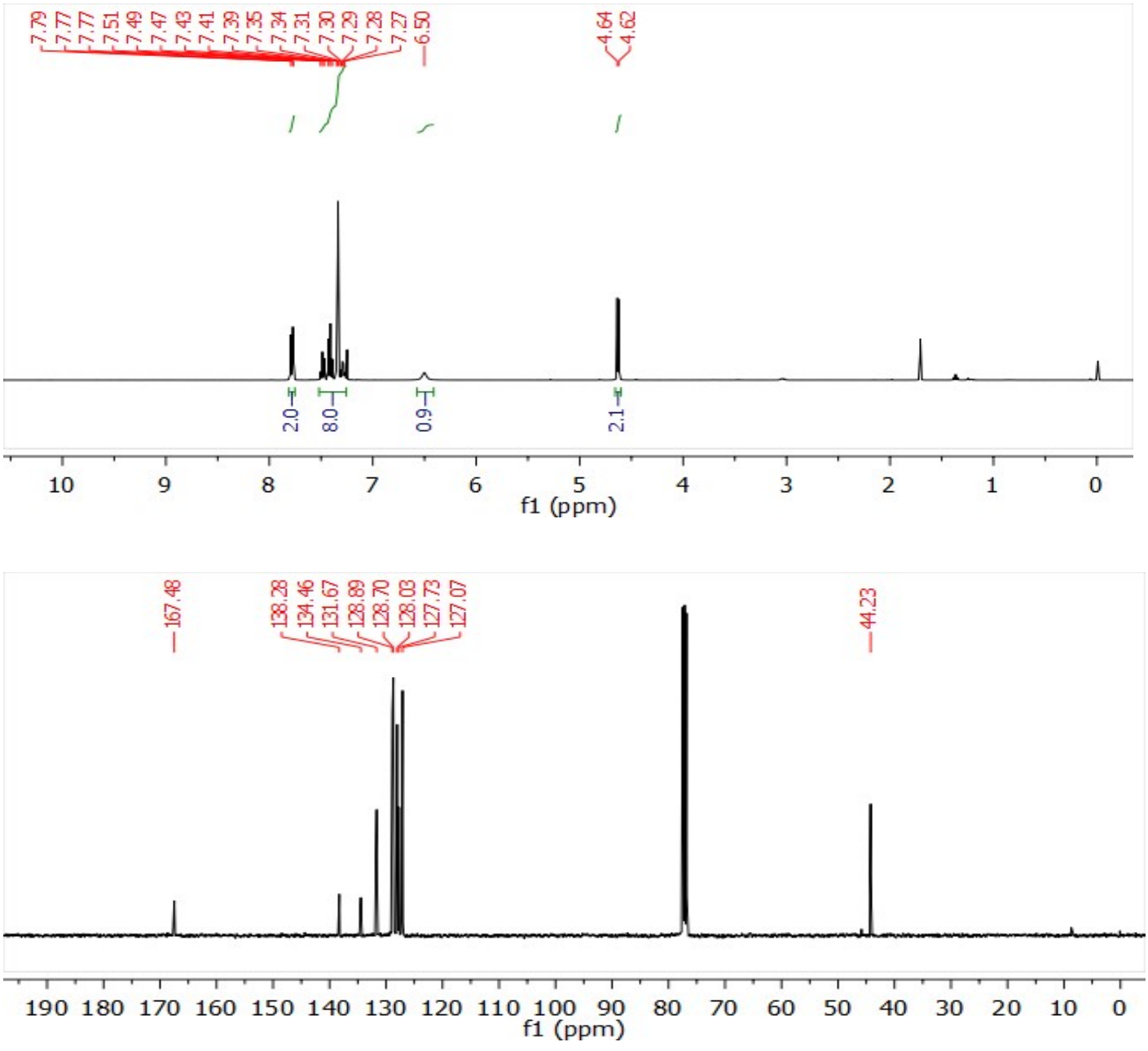
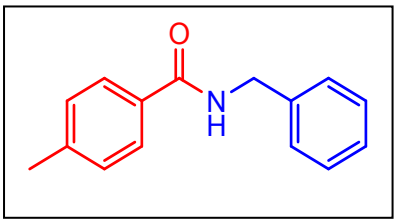


Figure: ^{1}H and ^{13}C spectra of compound 4b.

_N_-benzyl-4-methylbenzamide (4c)[5]



White solid, m.p - 95-97 ^{o}C; yield - 90%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.69 (dd, *J*=8.2 Hz, 2H), 7.36 – 7.18 (m, 7H), 6.44 (brs, 1H), 4.62 (d, *J*=5.7 Hz, 2H), 2.38 (s, 3H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 167.41, 142.08, 138.40, 131.60, 129.34, 128.87, 128.02, 127.68, 127.06, 44.17, 21.54, LC-MS : *m/z* calculated $[M+H]^{+}$ = 226.1701, found = 226.1700.

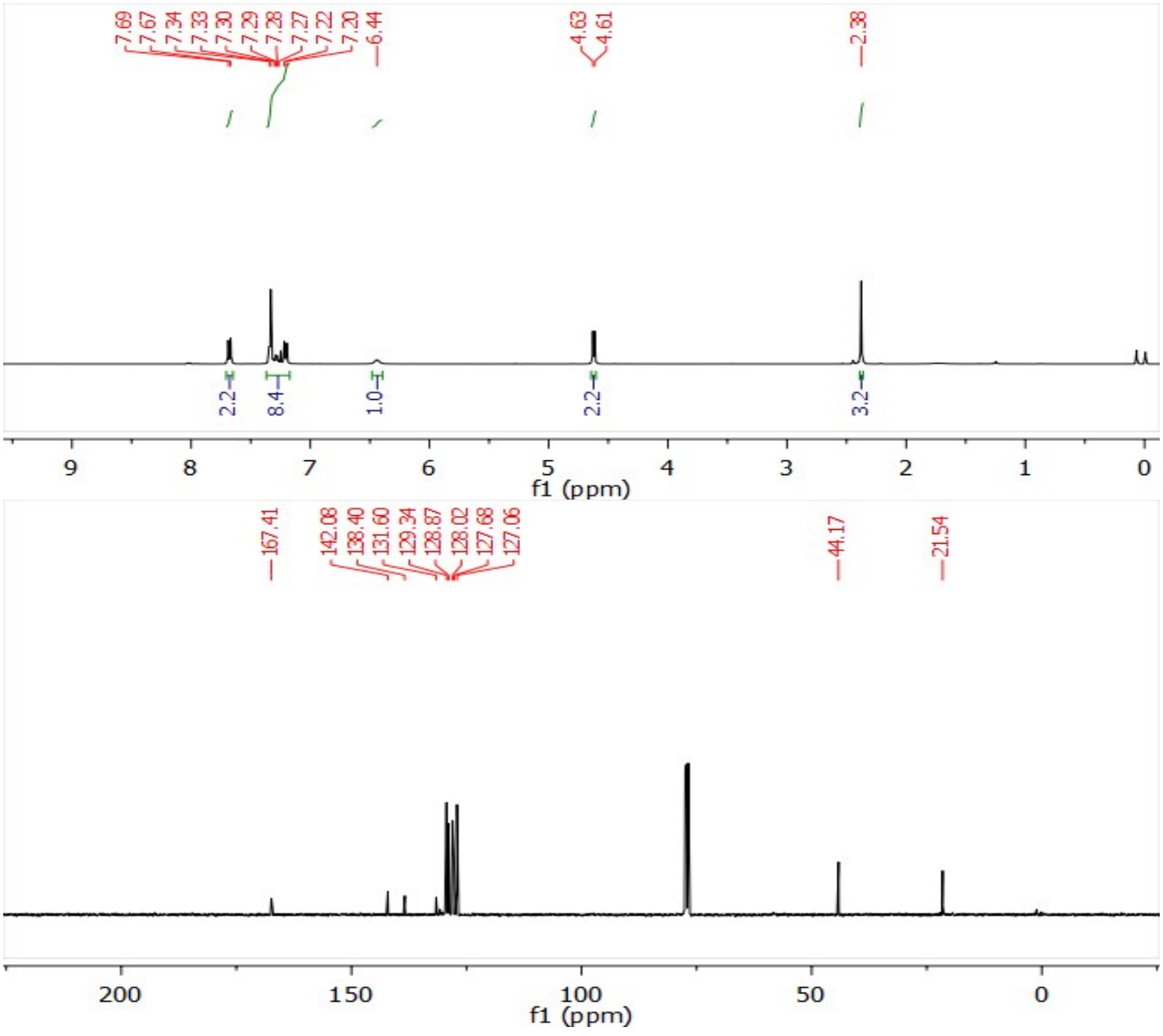
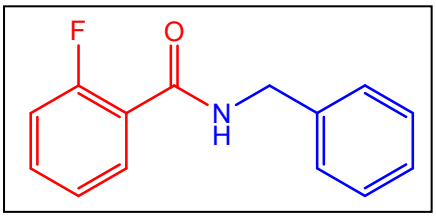


Figure: ^{1}H and ^{13}C spectra of compound 4c.

_N_-benzyl-2-fluorobenzamide (4d)[6]



White solid, mp 67–69 °C; yield- 85%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.00 (m, *J*=27.3, 7.2 Hz, 1H), 7.52 – 6.97 (m, 8H), 4.63 (d, *J*=5.7 Hz, 2H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 167.01, 159.41, 138.13, 133.50, 132.03, 128.83, 127.61, 124.90, 124.07, 121.16, 116.24, 44.13, , LC-MS : *m/z* calculated $[M+H]^{+}$ =230.1510, found =230.1517.

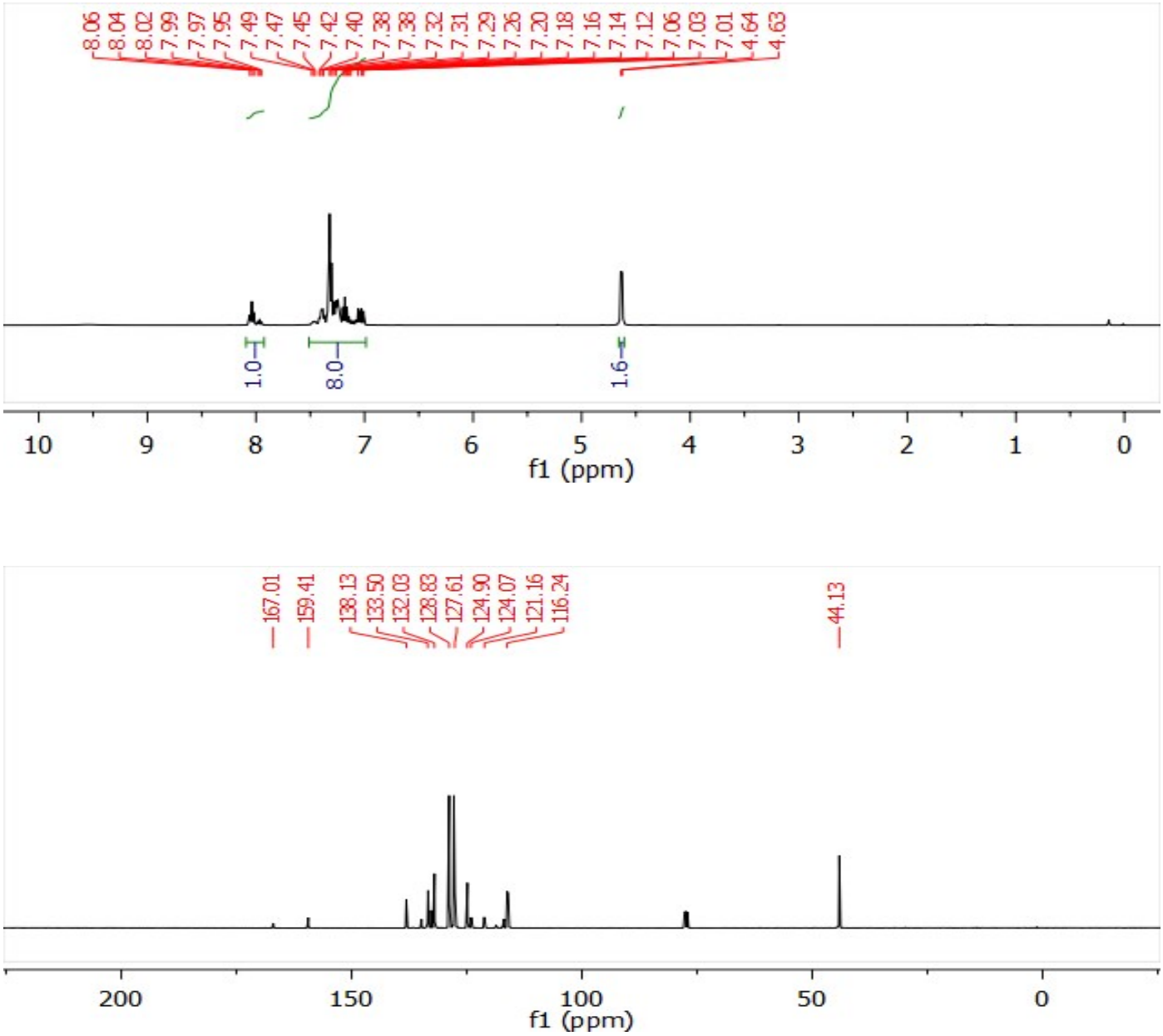
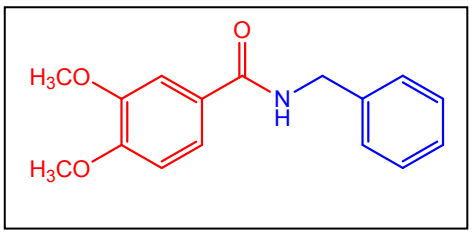


Figure: ^{1}H and ^{13}C spectra of compound 4d.

_N_-benzyl-3,4-dimethoxybenzamide (4e)[7]



White solid, m.p – 140-141 ˚C, yield – 89%; ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.45 – 7.18 (m, 5H), 6.96 – 6.78 (m, 3H), 4.60 (s, 2H), 3.87 (s, 6H),^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 167.10, 152.14, 149.08, 138.52, 128.81, 127.96, 127.03, 126.34, 119.83, 117.59, 110.63, 56.09, 44.16, LC-MS : *m/z* calculated $[M+H]^{+}$ = 302.1324, found = 302.1321.

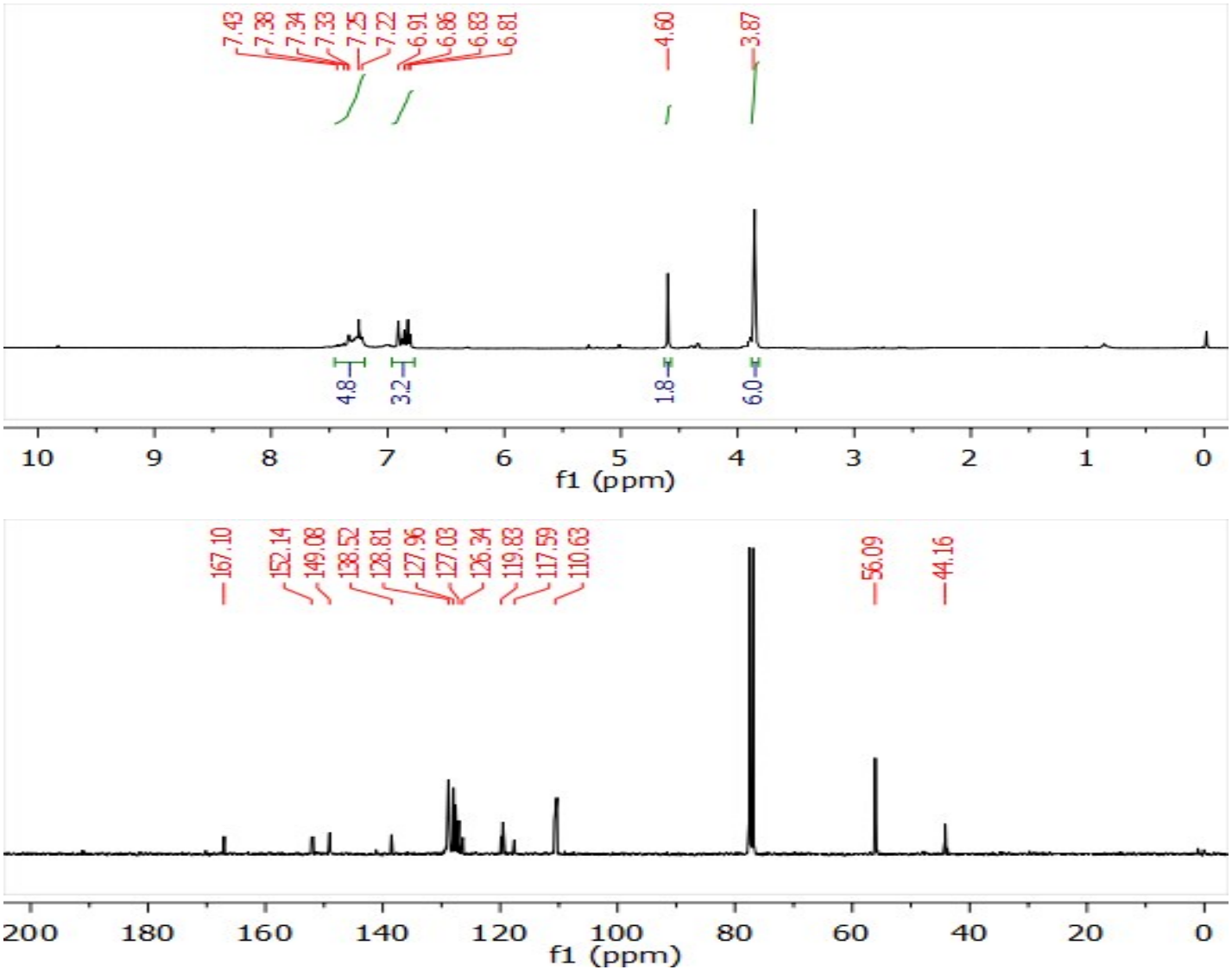
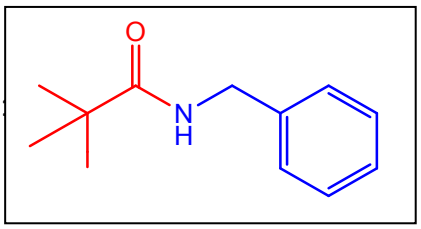


Figure: ^{1}H and ^{13}C spectra of compound 4e.

***N*-benzylpivalamide (4f)[8a]**



White solid, m.p – 82-83 ˚C; yield - 84%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.43 – 7.19 (m, 5H), 5.90 (brs, 1H), 4.43 (d, *J*=5.6 Hz, 2H), 1.22 (s, 9H) 13C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 178.41, 138.70, 128.82, 127.76, 127.55, 43.69, 38.82, 27.72, LC-MS : *m/z* calculated [M+H]+ =192.1790, found =.192.1794.

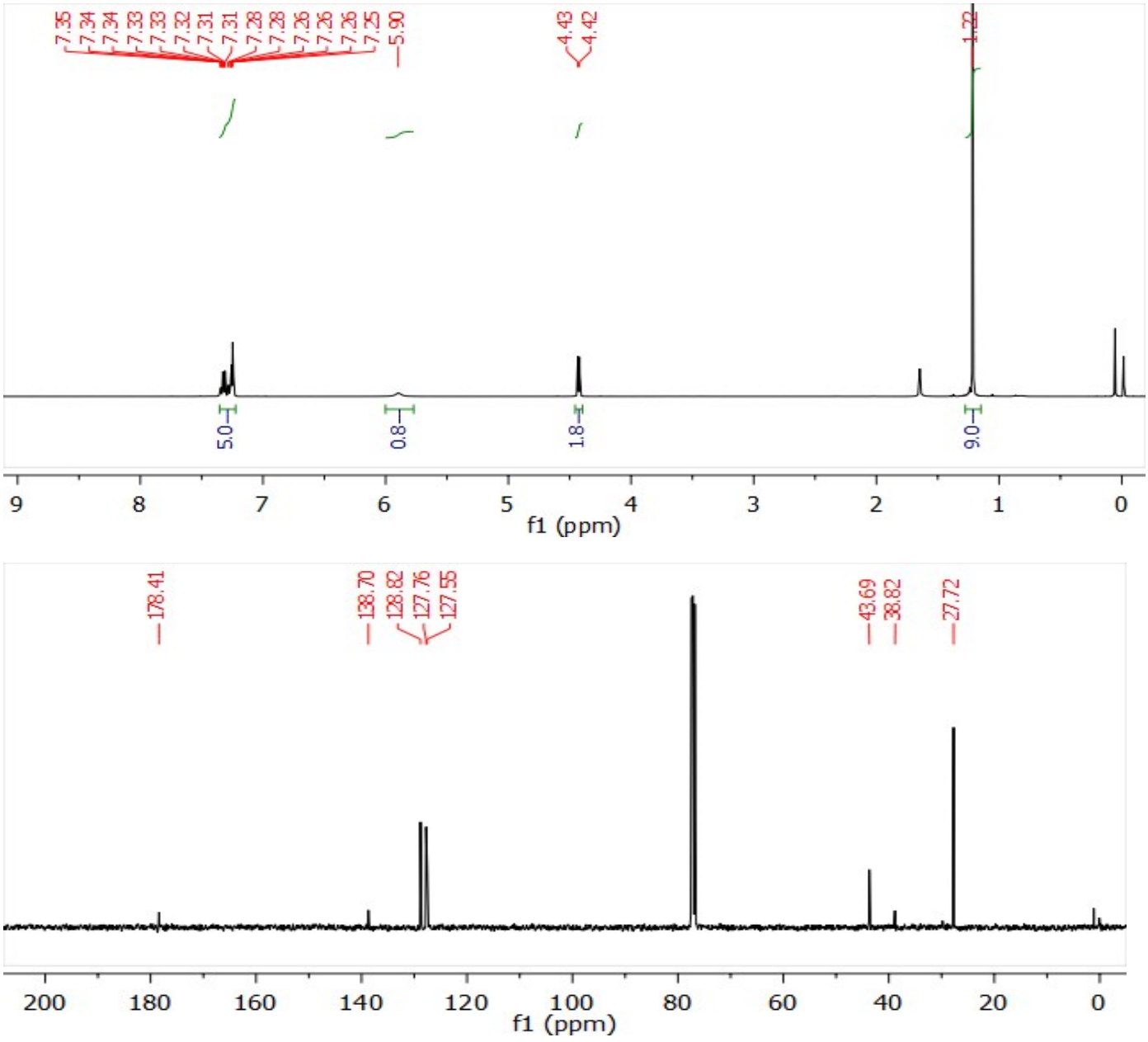
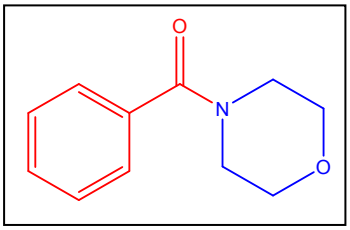


Figure: ^{1}H and ^{13}C spectra of compound 4f.

Morpholino(phenyl)methanone (4g)[7]



White solid, m.p – 48–50 ˚C; yield - 78%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ(ppm) 7.41-7.34 (m, *J*=6.1 Hz, 1H), 4.03– 3.17 (m, 5H), 3.86 – 3.24 (m, 8H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 170.55, 135.39, 129.99, 128.67, 127.18, 67.00, 42.64, LC-MS : *m/z* calculated $[M+H]^{+}$ = 192.1354, found 192.1354.

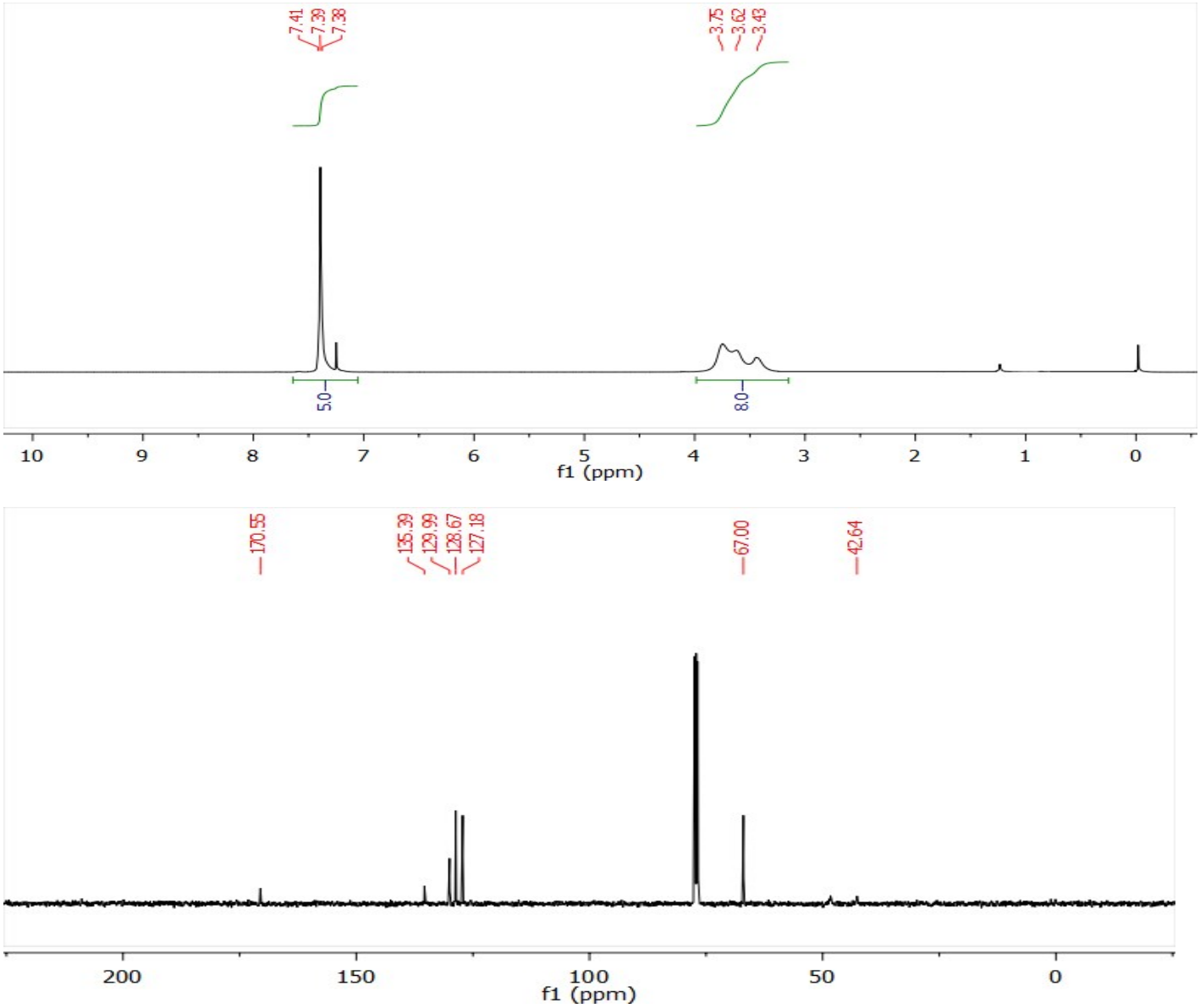
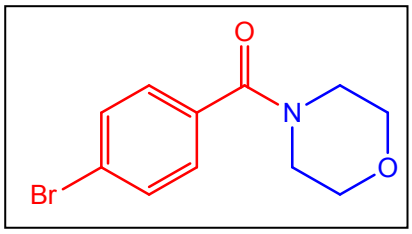


Figure: ^{1}H and ^{13}C spectra of compound 4g.

(4-bromophenyl)(morpholino)methanone (4h)[8b]



White solid, m.p –˚C; yield - 77%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.58 – 7.49 (dd, 2H), 7.26 (dd, *J*=8.0, 1.3 Hz, 2H), 3.87 – 3.50 (m, 8H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.52, 134.12, 131.93, 128.94, 124.38, 66.90, 52.06, LC-MS : *m/z* calculated [M+H]+ =270.0176, found =270.0179.

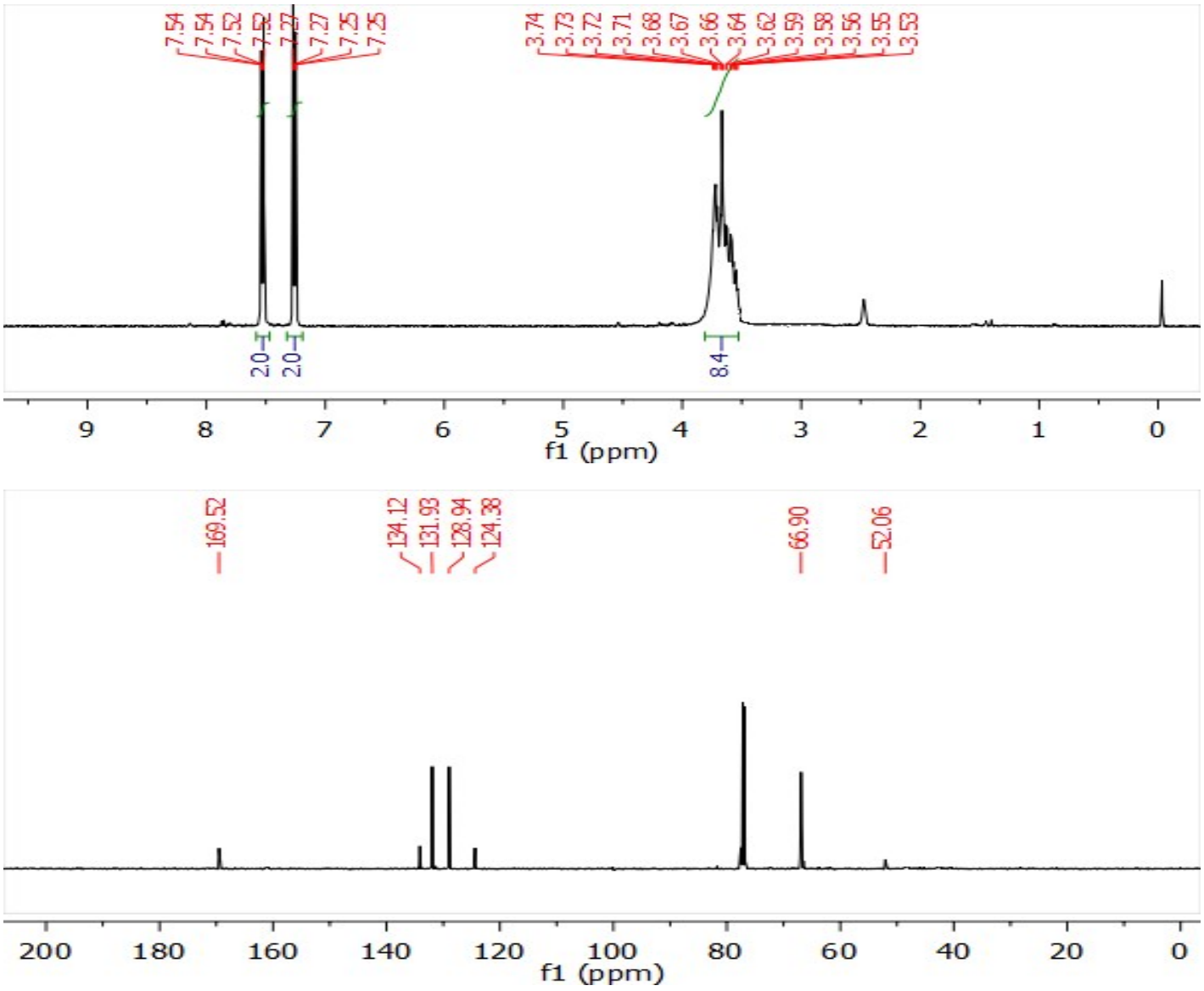
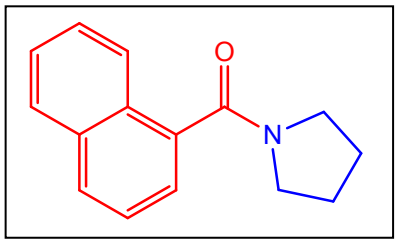


Figure: ^{1}H and ^{13}C spectra of compound 4h.

Napthalen-1-yl(pyrrolidin-1-yl)methanone (4i)[8c]



Yellow oil, yield - 76%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.91 – 7.78 (m, 3H), 7.57 – 7.40 (m, 4H), 3.78 (t, *J*=7.1 Hz, 2H), 3.11 (t, *J*=6.8 Hz, 2H), 1.99 (m, *J*=6.9 Hz, 2H), 1.82 (m, *J*=6.7 Hz, 2H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 169.42, 135.88, 133.65, 129.22, 128.52, 127.08, 126.41, 125.96, 125.32, 124.99, 123.84, 48.65, 26.13, LC-MS : *m/z* calculated [M+H]+ =226.1206, found =226.1209.

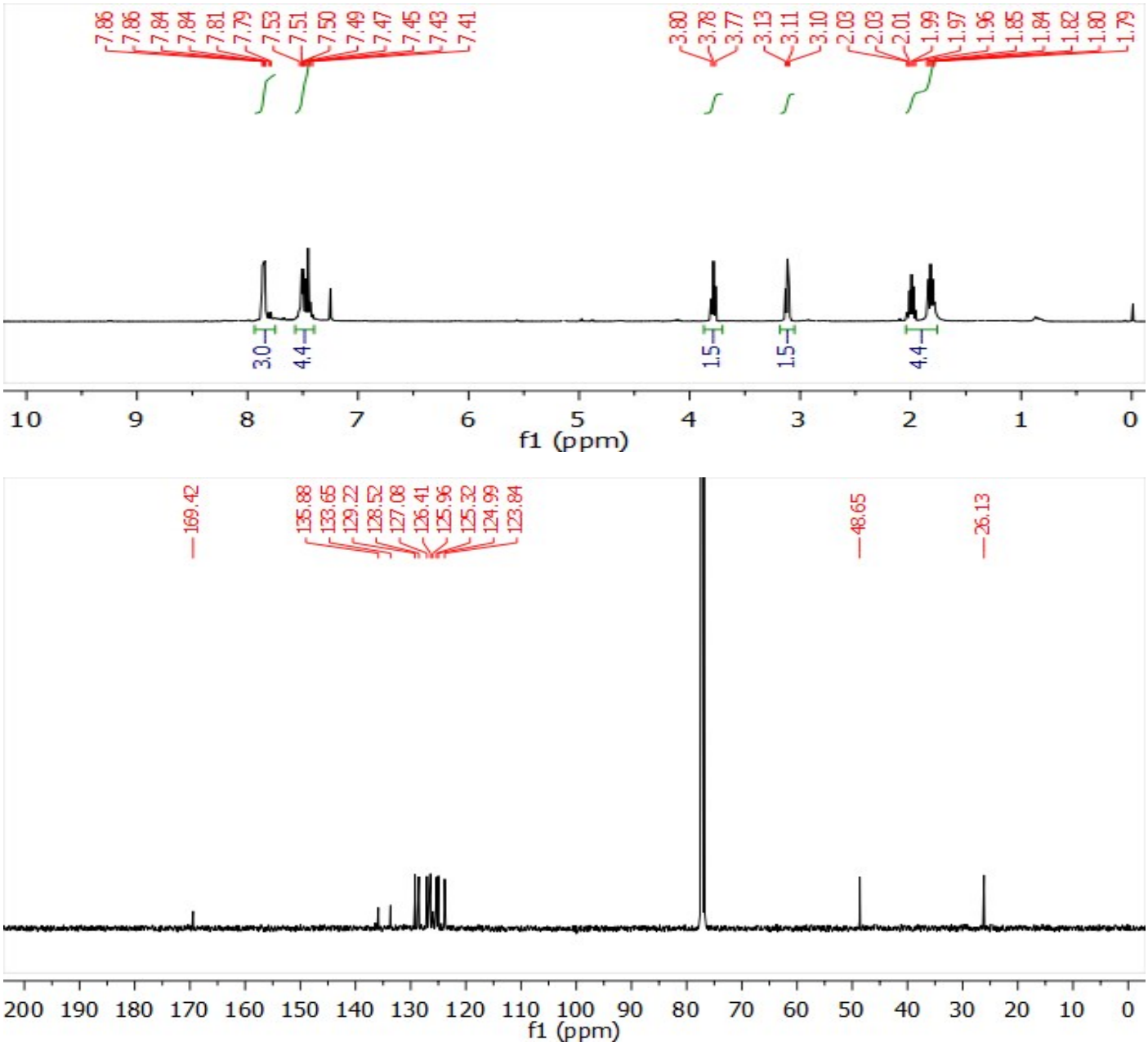
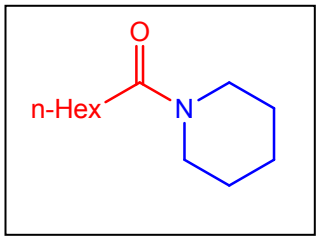


Figure: ^{1}H and ^{13}C spectra of compound 4i.

1-(piperidin-1-yl)heptan-1-ol (4j)[9]



Yellow oil, yield – 81%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 3.56 – 3.50 (t, 2H), 3.40 – 3.35 (t, 2H), 2.33 – 2.25 (t, 2H), 1.56 (m, *J*=26.0, 11.3, 6.4 Hz, 8H), 1.36 – 1.22 (m, 6H), 0.86 (t, *J*=6.8 Hz, 3H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 171.66, 46.82, 42.67, 33.62, 31.74, 29.33, 26.67, 25.68, 24.69, 22.65, 14.16, LC-MS : *m/z* calculated [M+H]+ = 198.2260, found = 198.2264.

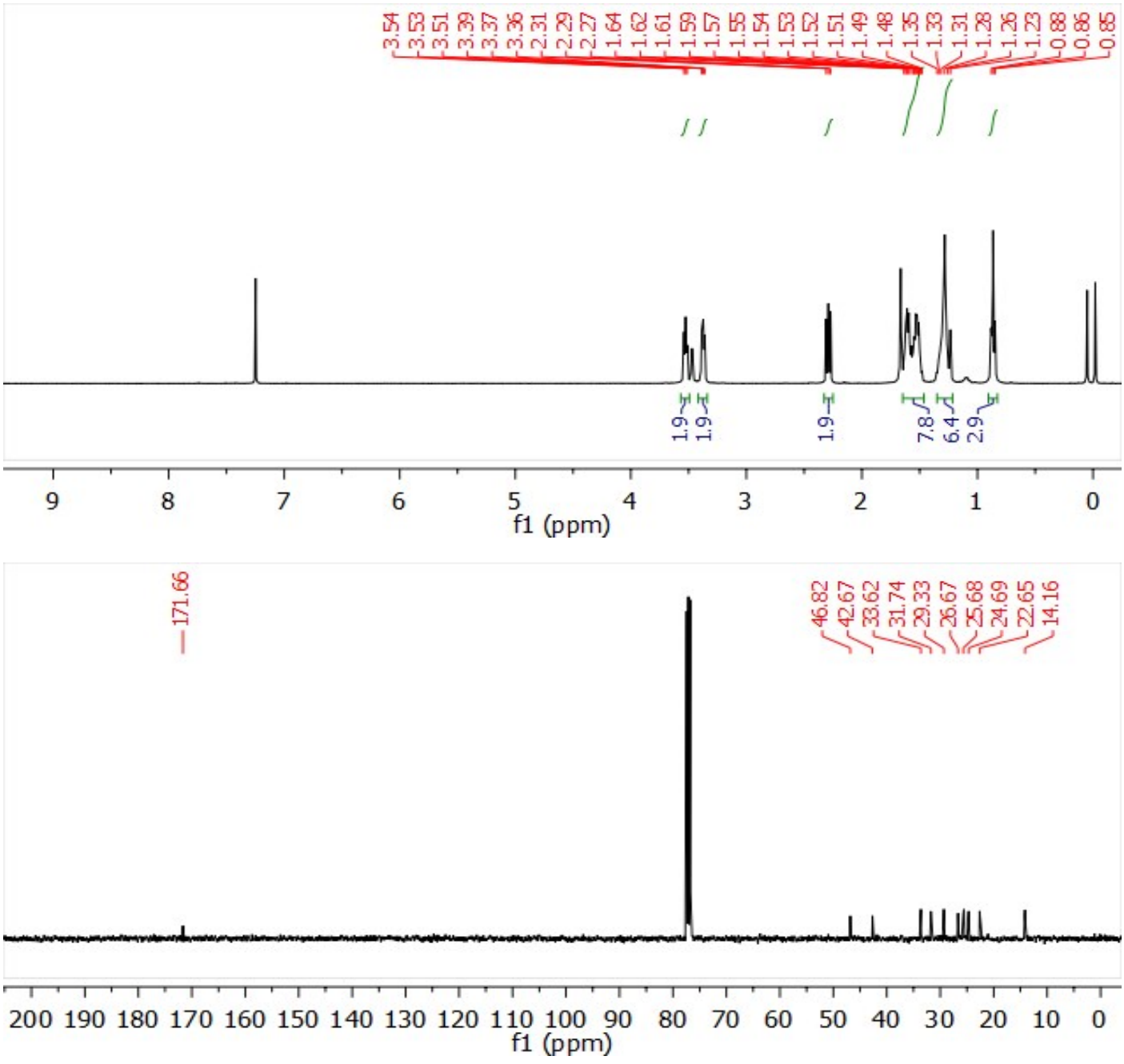
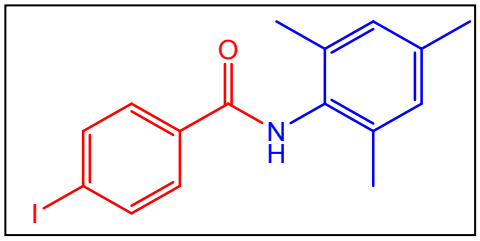


Figure: ^{1}H and ^{13}C spectra of compound 4j.

4-iodo-*N*-mesitylbenzamide (4k)



White solid, m.p - ; yield – 70%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.80 (dd, *J*=8.6, 1.9 Hz, 2H), 7.61 (dd, *J*=8.5 Hz, 2H), 7.40 (s, 1H), 6.91 (s, 2H), 2.28 (s, 3H), 2.19 (s, 6H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 165.48, 138.02, 137.38, 135.34, 133.97, 131.02, 129.15, 128.93, 98.81, 21.09, 18.45, LC-MS : *m/z* calculated [M+H]+ = 366.0341, found = 366.0344.

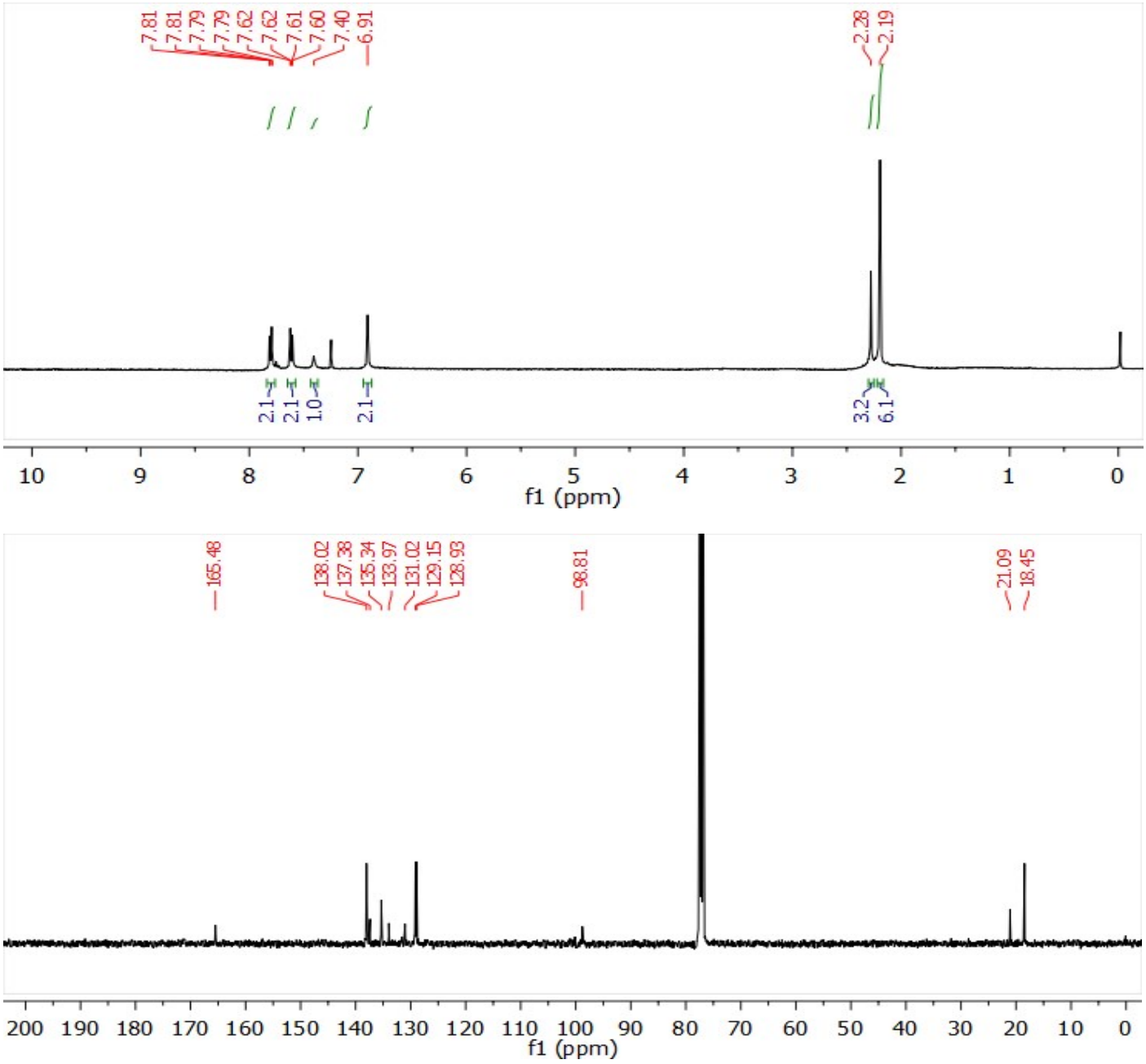
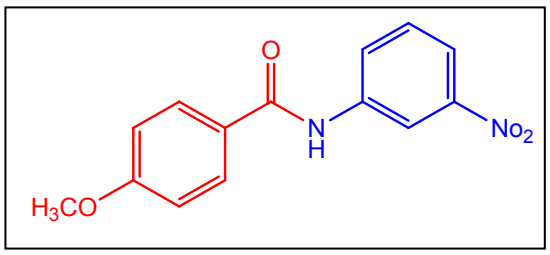


Figure: ^{1}H and ^{13}C spectra of compound 4k.

4-methoxy-*N*-(3-nitrophenyl)benzamide (4l)[9]



white solid, m.p – 177-178 ˚C ; yield - 69%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.95 – 7.86 (m, 4H), 6.85 – 6.78 (m, 4H), 3.75 (s, 3H), 13C NMR(CHLOROFORM-*D*, 101 MHz): δ(ppm) 164.67, 162.41, 134.13, 132.94, 132.40, 131.92, 129.23, 121.34, 114.33, 114.23, 113.83, 55.70, LC-MS : *m/z* calculated [M+H]+ = 273.1619, found = 273.1622.

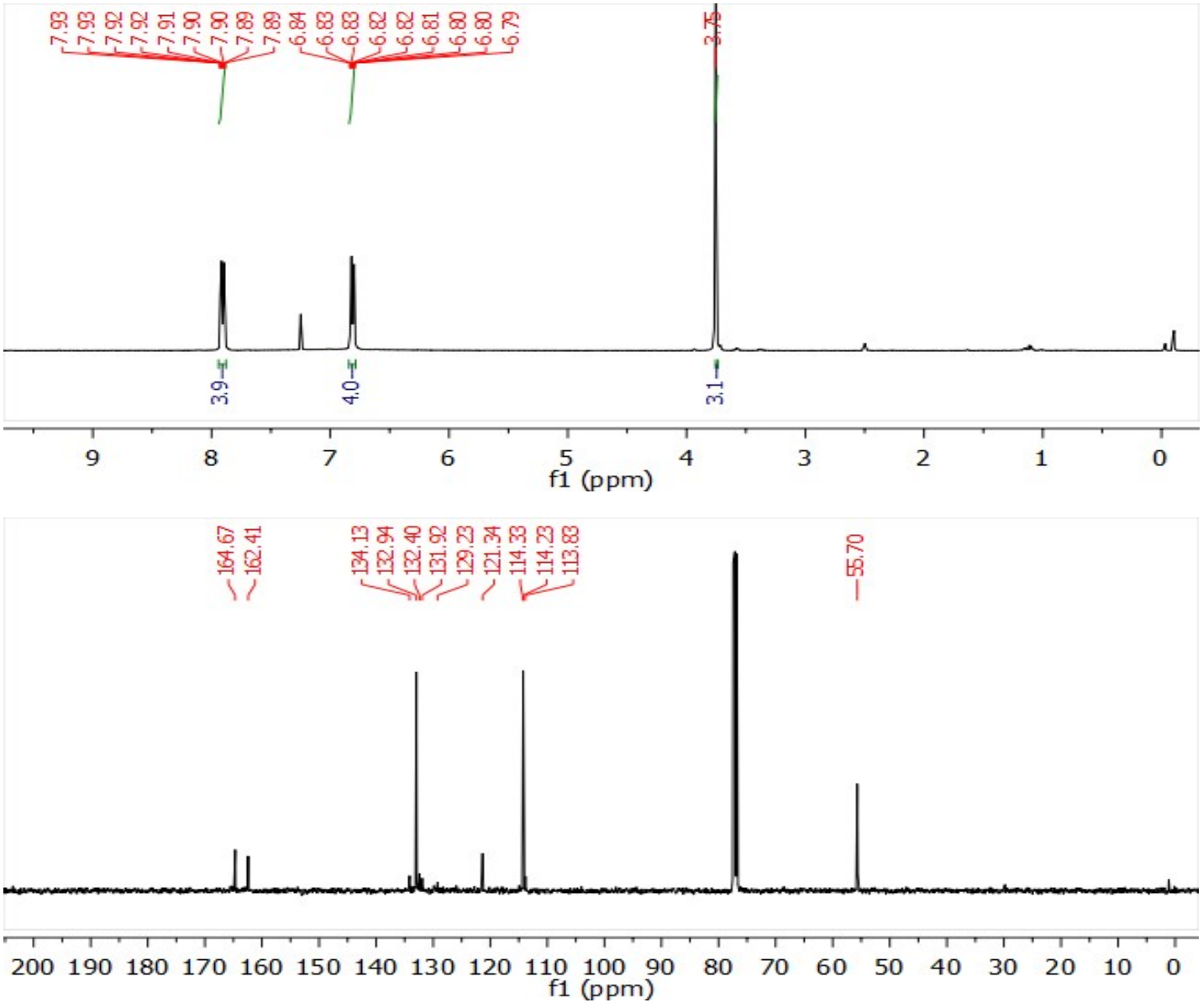
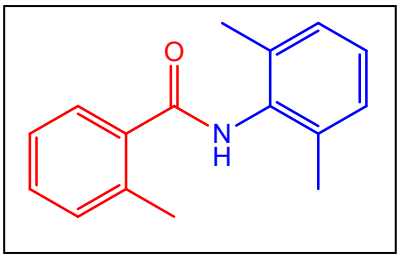


Figure: ^{1}H and ^{13}C spectra of compound 4l.

***N*-(2,6-dimethylphenyl)-2-methylbenzamide (4m)[8d]**



White solid, m.p. 138-140°C, yield - 77%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.41 – 6.95 (m, 7H), 6.38 (brs, 1H), 2.54 (s, 3H), 2.30 (s, 6H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 161.88, 137.62, 134.15, 131.12, 130.07, 129.58, 128.92, 128.16, 127.16, 126.47, 123.73, 18.52, 17.53, LC-MS : *m/z* calculated [M+H]+ = 240.1325, found = 240.1328.

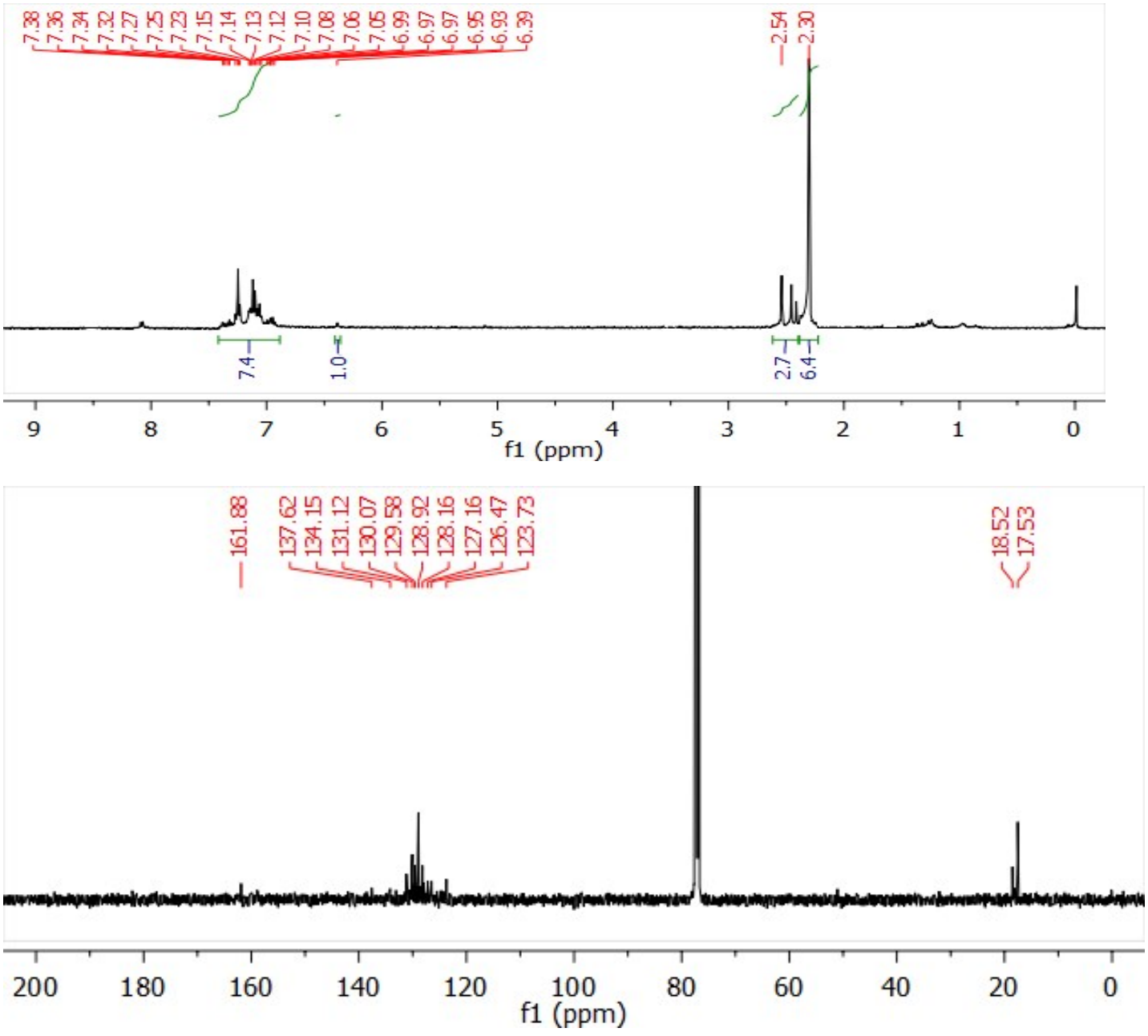
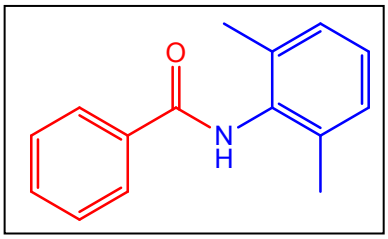


Figure: ^{1}H and ^{13}C spectra of compound 4m.

_N_-(2,6-dimethylphenyl)benzamide (4n)[9]



White solid , m.p – 162-163 ˚C ; yield - 75%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.92 (dd, *J*=7.1 Hz, 2H), 7.53 (m, *J*=27.8, 7.3 Hz, 3H), 7.38 (s, 1H), 7.17 – 7.09 (m, 3H), 2.28 (s, 6H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 166.01, 135.67, 134.59, 133.90, 131.95, 128.91, 128.42, 127.59, 127.31, 18.62, LC-MS (ESI) : *m/z* calculated $[M+H]^{+}$ = 226.1761, found = 226.1767.

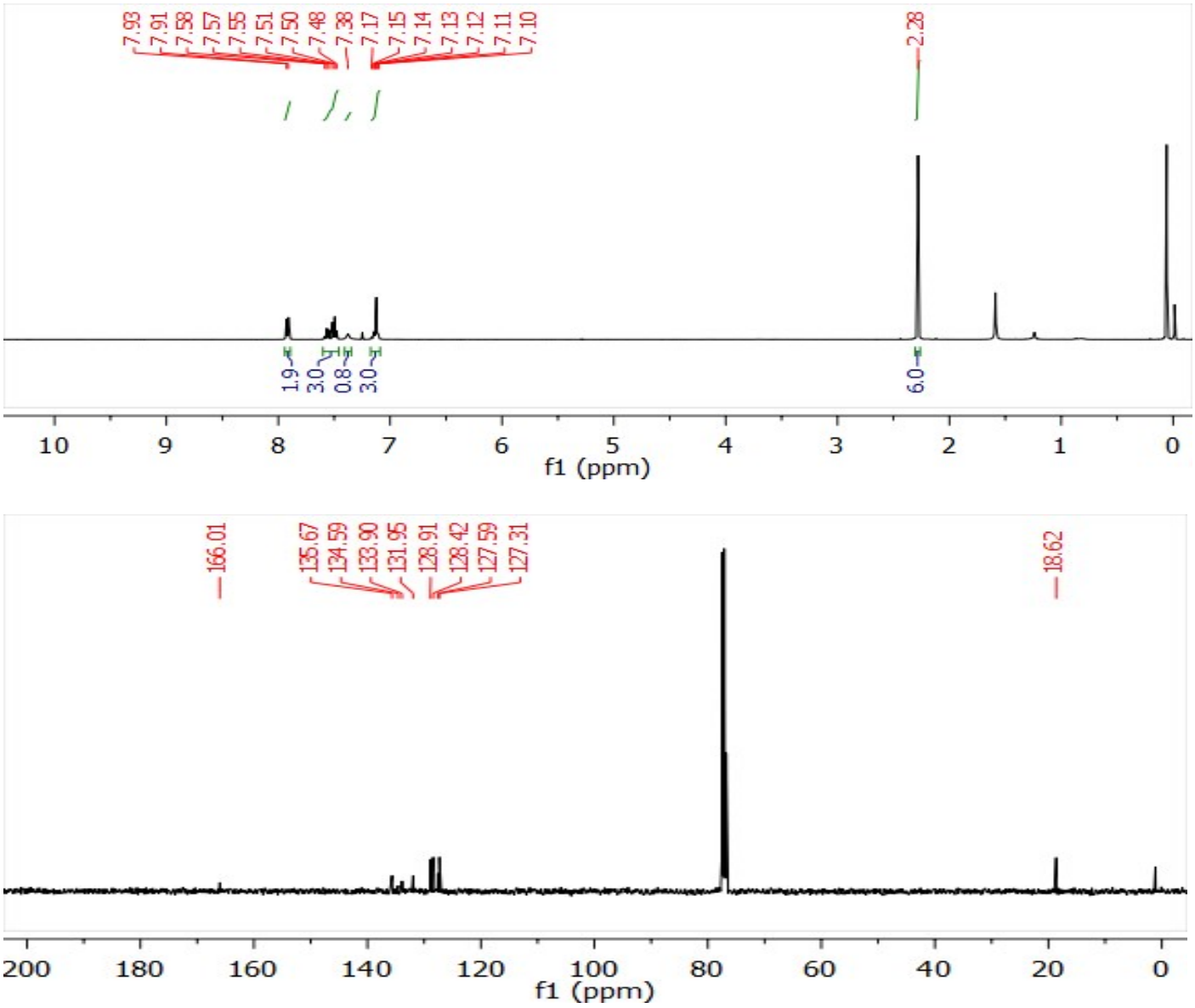
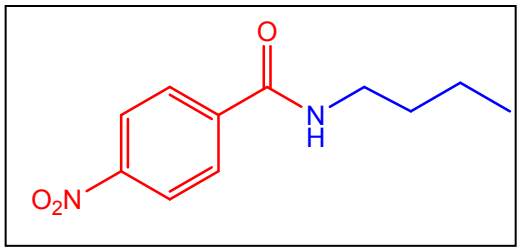


Figure: ^{1}H and ^{13}C spectra of compound 4n.

***N*-butyl-4-nitrobenzamide (4o)[7]**



Yellow solid, m.p - 131-133 °C ; yield - 71%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 8.26 (d, *J*=8.9 Hz, 2H), 7.90 (d, *J*=8.9 Hz, 2H), 6.29 (brs, 1H), 3.46 (q, *J*=7.2, 6.6 Hz, 2H), 1.60 (m, *J*=7.8, 7.4 Hz, 2H), 1.40 (m, *J*=14.6, 7.3 Hz, 2H), 0.95 (t, *J*=7.3 Hz, 3H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 165.60, 149.57, 140.52, 128.16, 123.91, 40.25, 31.66, 20.23, 13.85, LC-MS : *m/z* calculated $[M+H]^{+}$ = 223.1090, found = 223.1104.

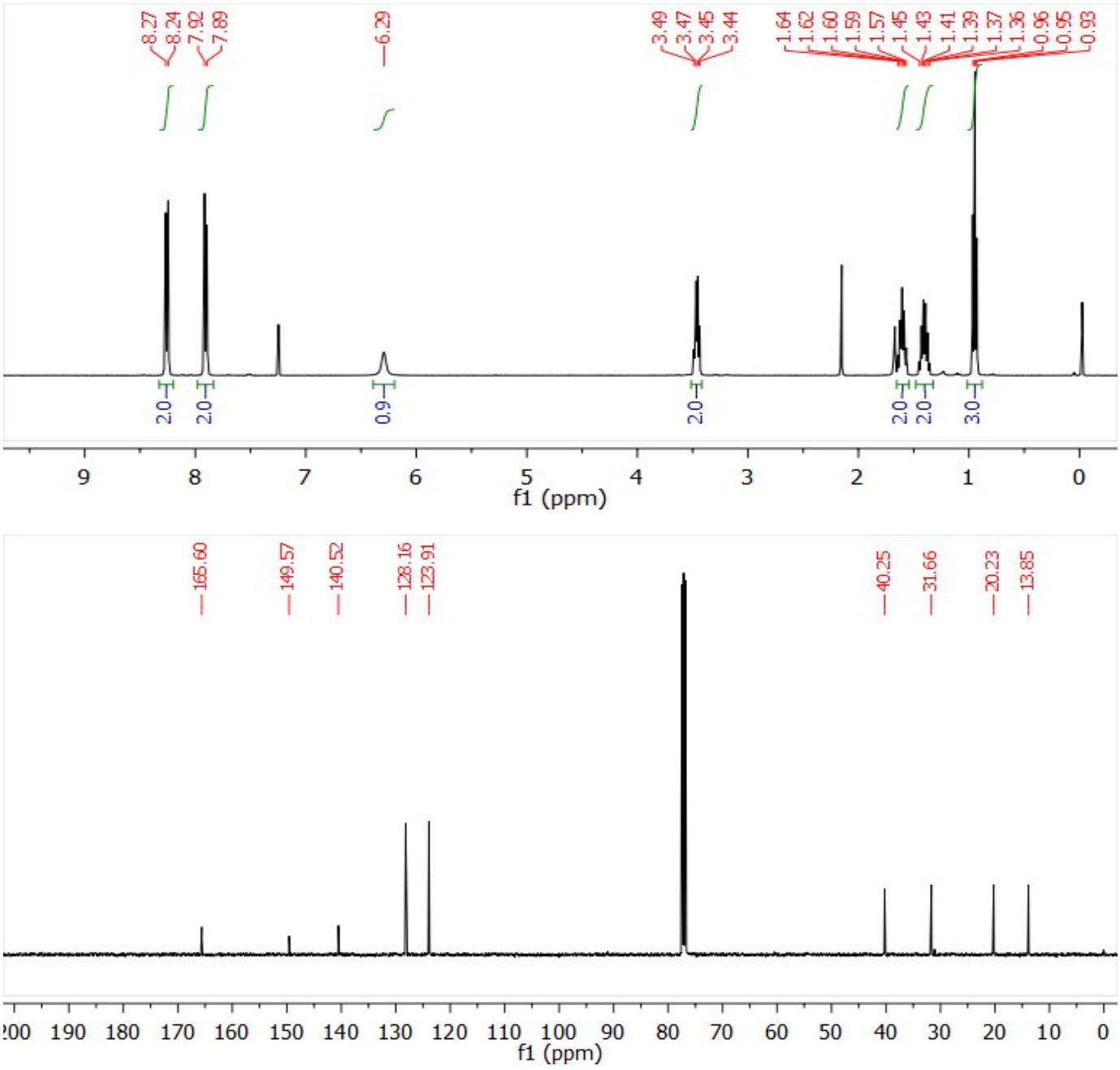
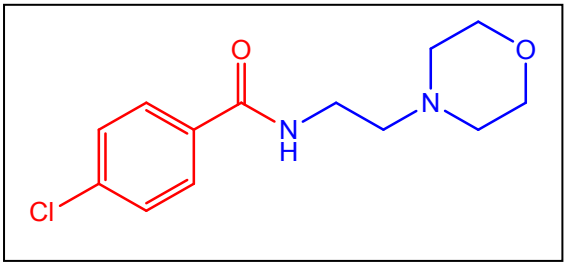


Figure: ^{1}H and ^{13}C spectra of compound 4o.

4-chloro-N-(2-morpholinoethyl)benzamide (4p)[10]



White solid, m.p – 130-132˚C , yield – 87%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.70 (d, *J*=8.6 Hz, 2H), 7.43 – 7.37 (m, 20H), 6.82 (s, 1H), 3.71 (m, *J*=10.4, 4.2 Hz, 4H), 3.54 (q, *J*=5.5 Hz, 2H), 2.63 – 2.43 (m, 6H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 166.47, 137.75, 132.98, 128.93, 128.47, 67.01, 56.91, 53.38, LC-MS : *m/z* calculated $[M+H]^{+}$ = 269.1051, found = 269.1059.

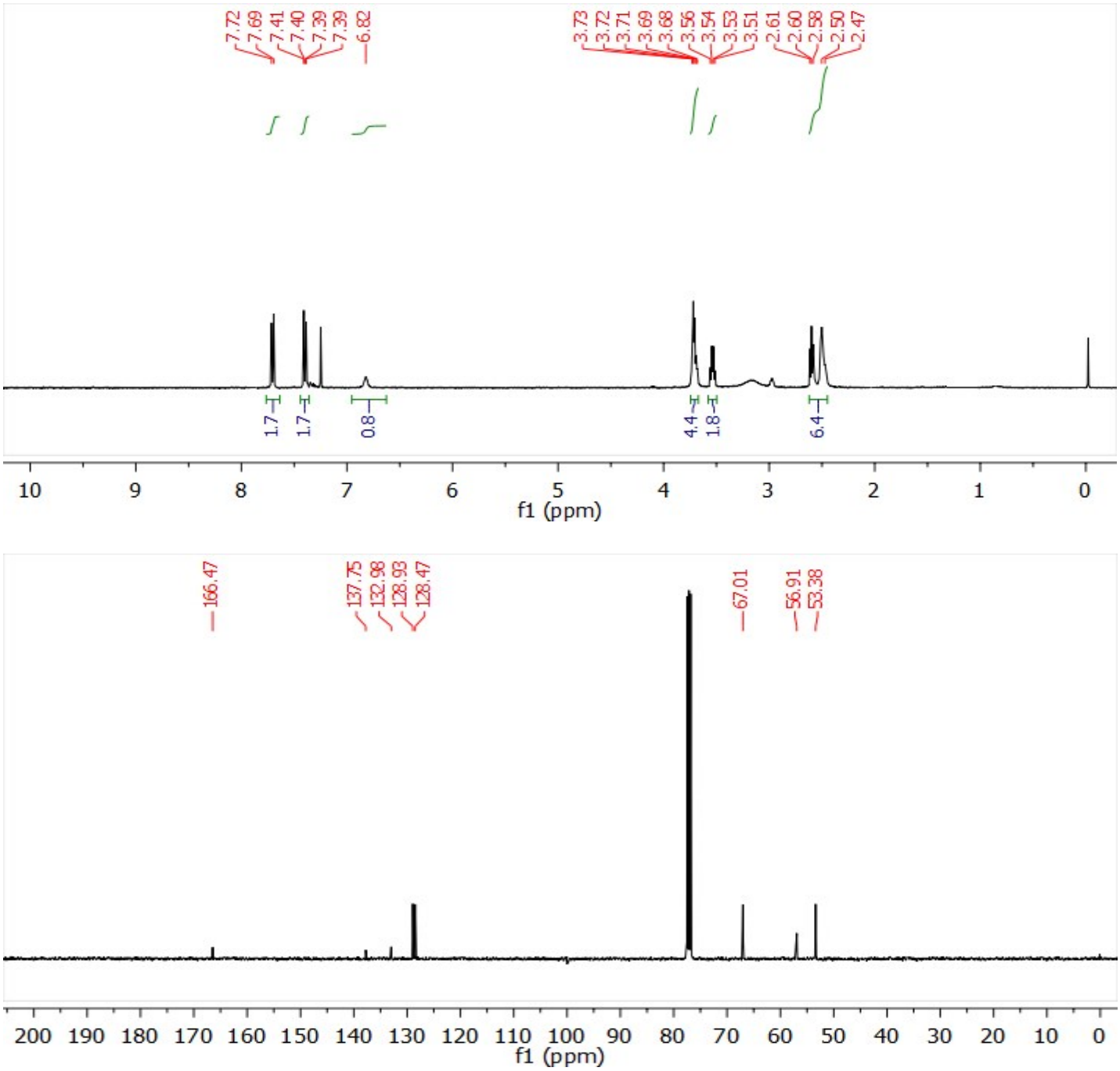
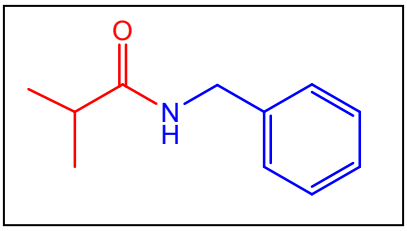


Figure: ^{1}H and ^{13}C spectra of compound 4p.

***N*-benzylisobutyramide (4q)**[11]



White solid, m.p – 83-85˚C, yield – 78%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.31 (m, *J*=2.9 Hz, 5H), 6.64 (brs, 1H), 4.36 (d, *J*=8.1, 0.8 Hz, 2H), 2.47 (m, *J*=6.7 Hz, 1H), 1.10 (d, *J*=6.8 Hz, 6H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 176.73, 137.92, 128.90, 127.09, 126.95, 42.10, 34.53, 19.29, LC-MS : *m/z* calculated $[M+H]^{+}$ = 269.1051, found = 269.1059.

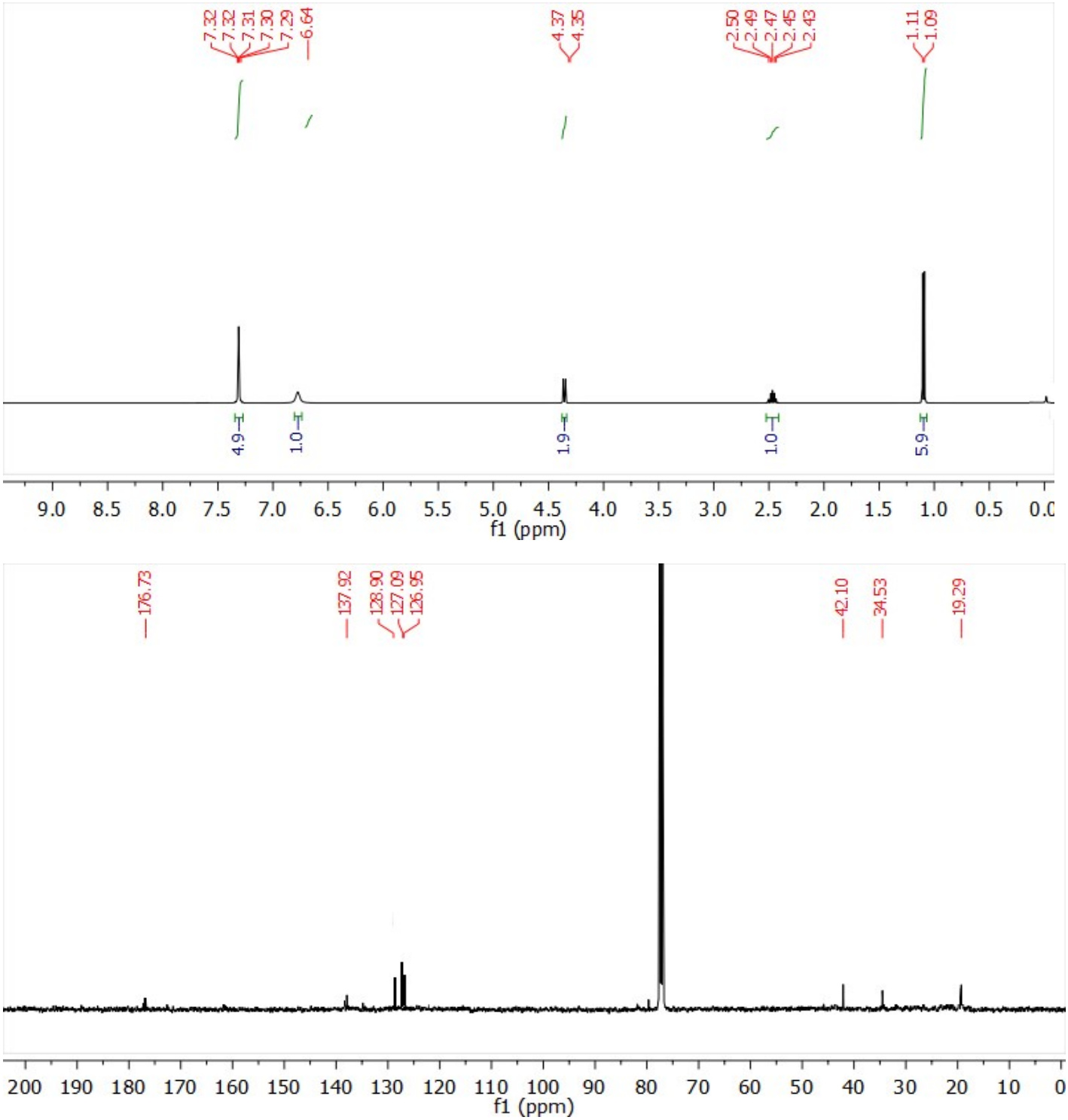
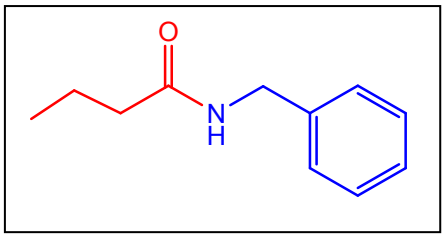


Figure: ^{1}H and ^{13}C spectra of compound 4q.

N-benzylbutyramide (4r)[11]



Off white solid, m.p – 47-48˚C, yield – 69%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.40 – 7.01 (m, 5H), 6.35 (t, *J*=7.5 Hz, 1H), 4.30 (d, *J*=5.8 Hz, 2H), 2.14 – 2.07 (t, 2H), 1.56 (m, *J*=14.7, 7.4 Hz, 2H), 0.83 (t, *J*=7.4 Hz, 3H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 173.43, 138.87, 128.53, 127.70, 127.17, 43.30, 38.46, 19.25, 13.82, LC-MS : *m/z* calculated $[M+H]^{+}$ = 178.1206, found = 178.1209.

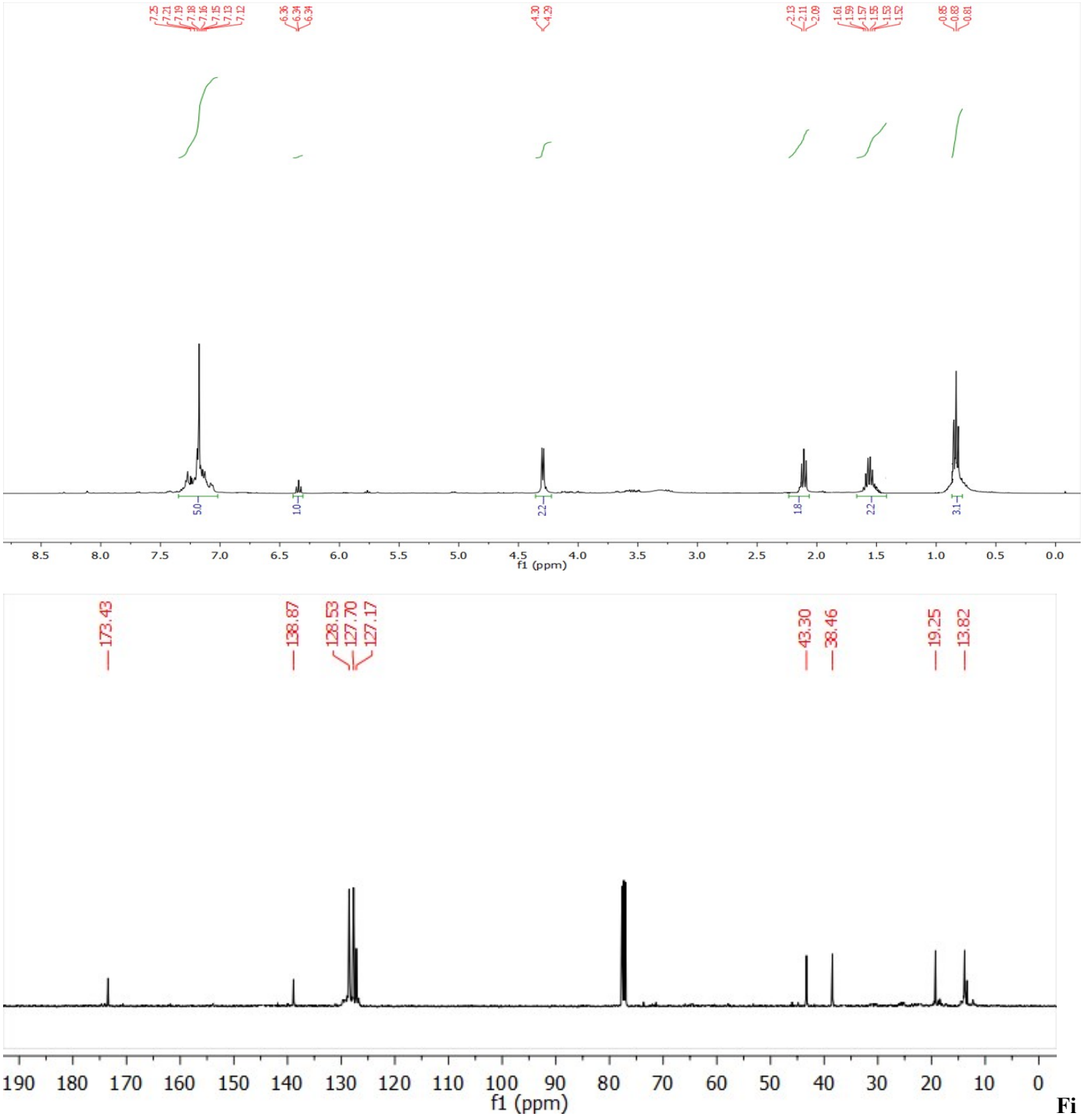
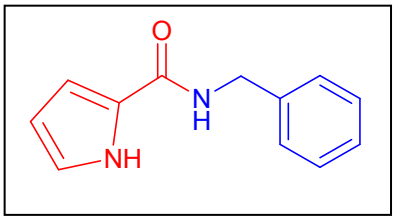


Figure: ^{1}H and ^{13}C spectra of compound 4r.

***N*-benzyl-1-*H*-pyrolle-2-carboxamide (4s)[12]**



Yellow oil, , yield – 85%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.83 – 7.76 (m, 1H), 7.47 – 7.07 (m, 7H), 6.75 (d, *J*=45.4 Hz, 1H), 6.16 (brs, 1H),) 4.51 (d, *J*=5.1 Hz, 1H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 162.36, 136.17, 128.77, 128.16, 127.74, 127.40, 122.18, 110.48, 109.67, 43.31. LC-MS : *m/z* calculated $[M+H]^{+}$ = 201.1045, found = 201.1048.

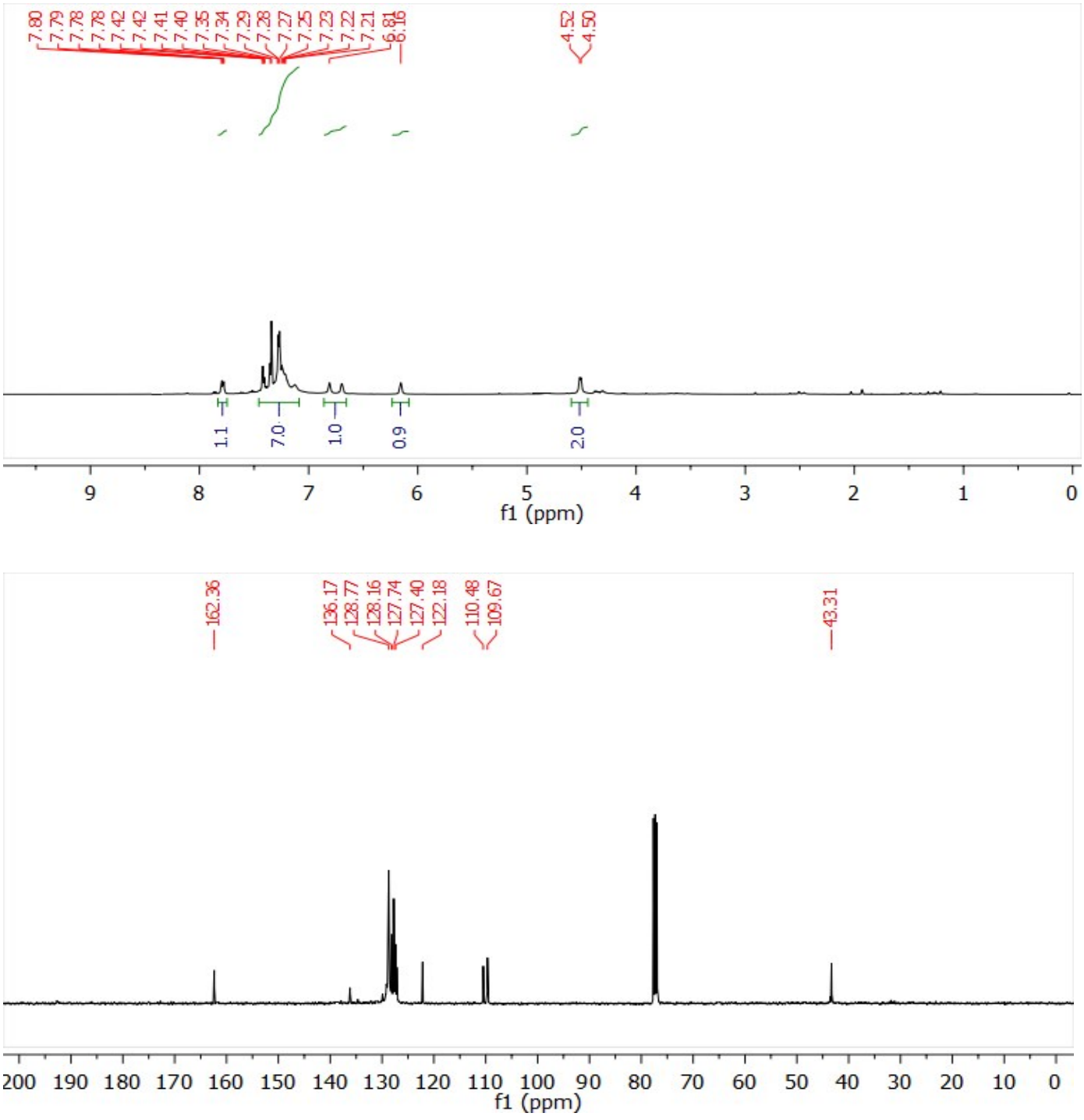
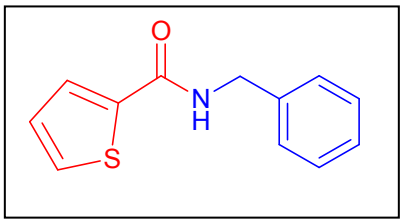
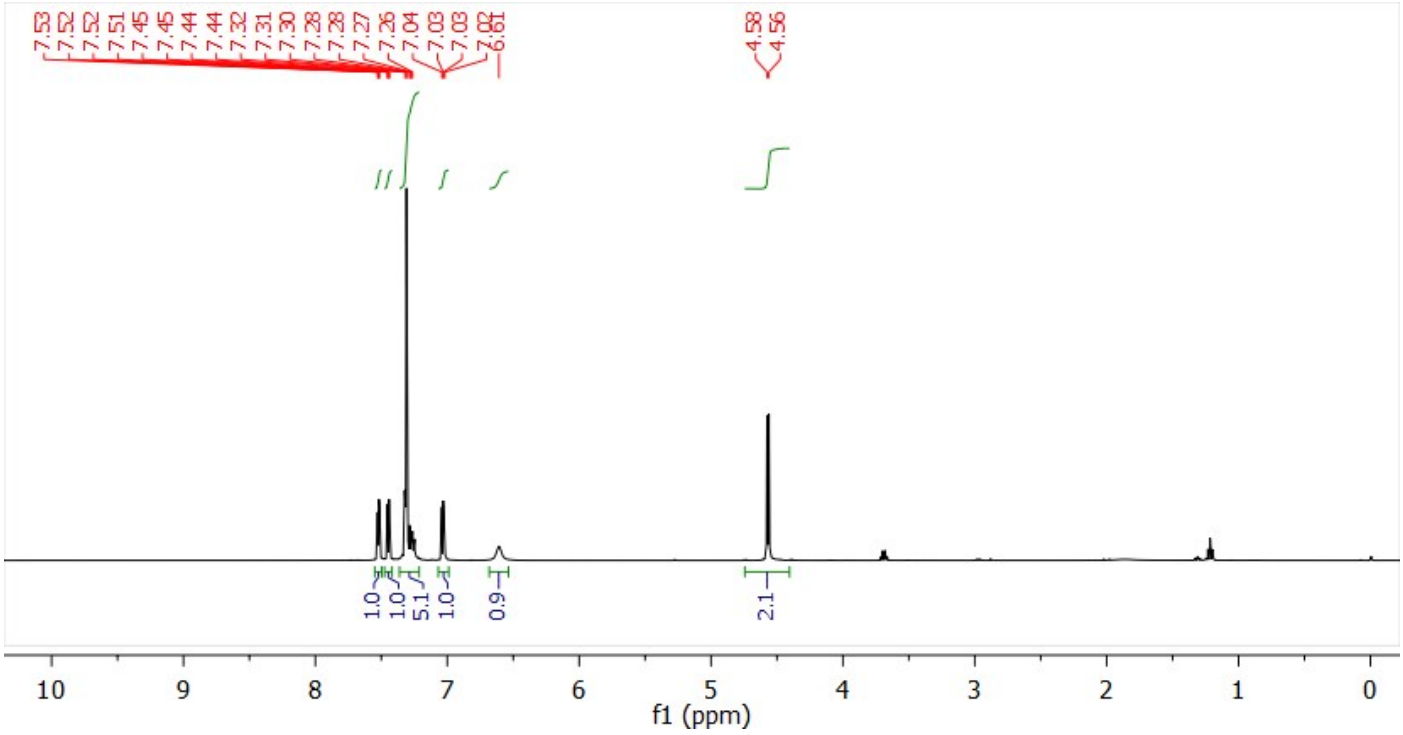


Figure: ^{1}H and ^{13}C spectra of compound 4s.

N-benzylthiophene-2-carboxamide (4t)[12]



White solid, m.p. – 119-120˚C, , yield – 79%, ^{1}H NMR(Chloroform-*d*, 400 MHz): δ (ppm) 7.52 (dd, *J*=3.7, 1.1 Hz, 1H), 7.45 (dd, *J*=5.0, 1.1 Hz, 1H), 7.36 – 7.23 (m, 5H), 7.03 (dd, *J*=5.0, 3.7 Hz, 1H), 6.62 (brs, 1H), 4.57 (d, *J*=5.8 Hz, 2H), ^{13}C NMR(CHLOROFORM-*D*, 101 MHz): δ (ppm) 162.01, 138.92, 138.20, 130.18, 128.84, 128.30, 128.00, 127.76, 127.69, 44.04. LC-MS : *m/z* calculated $[M+H]^{+}$ = 218.1612, found = 218.1615.



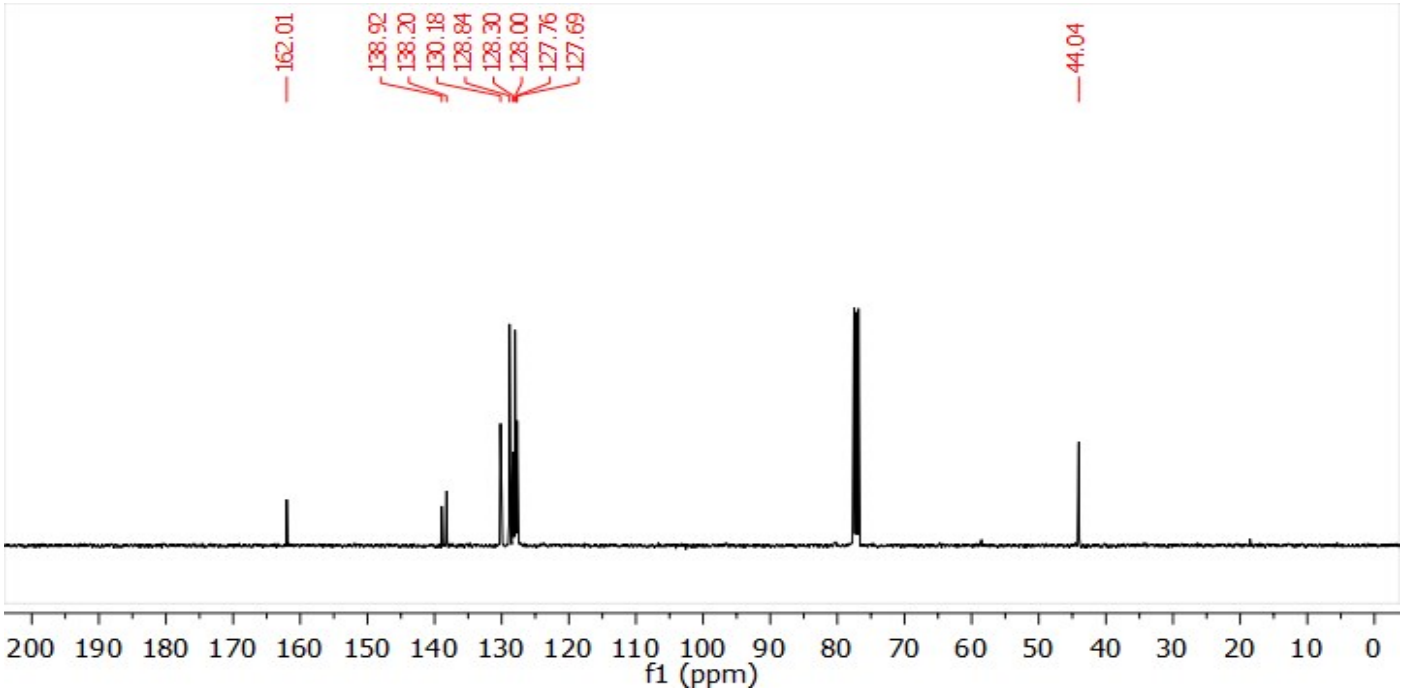


Figure: ^{1}H and ^{13}C spectra of compound 4t.

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