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GREEN AND EFFICIENT SYNTHESIS OF DIHYDROPYRIMIDINONE ANALOGUES *VIA* HPA-CLAY CATALYZED BIGINELLI REACTION

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5-Ethoxycarbonyl-4-(4-methoxyphenyl)-6-methyl-3,4-dihydropyrimidin-2(1H)-one ***4f***
M. P. (202°C), ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.21 (3H, t, *J*= 7.12 Hz), 2.34 (3H, s), 3.79(3H, s), 4.09 (2H, q, *J*= 7.16 Hz), 5.06 (1H, s), 6.86 (2H, d, *J*= 8.7 Hz), 7.26 (2H, d, *J*= 8.9 Hz), 7.66 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.18, 18.65, 55.20, 55.28, 59.99, 101.68, 114.02, 127.82, 136.15, 145.93, 153.36, 159.28, 165.71; IR (KBr) ν_{max}cm^{-1}: 3225, 3097, 2928, 1710, 1651, 1514, 1462, 1097; MS at *m/z* 290.13 (M^++Na).

Ethyl4-(benzo[d][1,3]dioxol-6-yl)-1,2,3,4-tetrahydro-6-methyl-2-oxopyrimidine-5-carboxylate ***4m***
^{1}H NMR (200 MHz, $CDCl_3$): δ 1.18 (3H, t, *J*= 7.08 Hz), 2.34 (3H, s), 4.01 (2H, q, *J*= 7.08 Hz), 5.34 (1H, s), 5.66 (1H, s), 5.94 (2H, s), 6.70 (1H, d, *J*= 1.61 Hz), 6.75 (1H, m), 6.80 (1H, d, *J*= 8.71 Hz), 7.92 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$): δ 14.20, 18.55, 55.45, 60.02, 101.12, 101.35, 107.09, 108.18, 120.0, 137.92, 146.33, 147.23, 147.94, 153.65, 165.67; IR (KBr) ν_{max}cm^{-1}: 3225, 3097, 2928, 1710, 1651, 1514, 1462, 1097; MS at *m/z* 304.31 (M^++Na). 752; HRMS (ESI$^+$) $C_{15}H_{16}N_2O_5$; Calcd: 305.1131 [M + H] $^+$; Found: 305.1131 [M + H] $^+$.

Ethyl 4-(4-(6-methoxy-2-oxo-2H-chromen-7-yl) but-2-en-2-yl)-6-methyl-2-oxo-1, 2, 3, 4-tetrahydropyrimidine-5-carboxylate ***5***
Yellowish Solid, (Mp: 144-146°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.6 (1H, d, *J*= 9.5 Hz), 7.3 (1H, d, *J*= 8.6 Hz), 7.2 (1H, s), 6.8 (1H, d, *J*= 8.6 Hz), 6.5 (1H, t, *J*= 7.3 Hz), 6.2 (1H, d, *J*= 9.4 Hz), 4.9 (1H, s), 4.1 (2H, q, *J*= 8 Hz), 3.9 (3H, s), 3.8 (2H, d, *J*= 7.6 Hz), 2.3 (3H, s), 1.9 (3H, s), 1.2 (3H, t, *J*= 8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.4, 161.8, 155.7, 154.6, 151.5, 149.3, 142.8, 134.5, 133.2, 126.1, 119.8, 119.3, 114.7, 106.8, 92.8, 61.5, 56.7, 48.9, 30.3, 18.9, 16.2, 14.6. IR (KBr)ν_{max}cm^{-1}**:** 3340, 3012, 1728, 1681, 1072, 832. HR-ESIMS *m/z*: 413.4424 (calculated for $C_{22}H_{24}N_2O_6$, 412.4358).

Ethyl 1,2,3,4-tetrahydro-6-methyl-2-oxo-4-propylpyrimidine-5-carboxylate ***6***
White solid, (Mp: 156-158°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.2 (1H, m), 4.2 (2H, q, *J*= 6.7 Hz), 2.2 (3H, s), 1.4 (2H, m), 1.28 (4H, m), 0.9 (3H, t, *J*= 6.9 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.8, 156, 149.2, 102.2, 61.1, 51.9, 40.3, 18.4, 18.1, 14.6, 14.2. IR (KBr)ν_{max} cm^{-1}: 1744, 1717, 1637, 1440, 1215. HR-ESIMS *m/z*: 227.1393 (calculated for $C_{11}H_{18}N_2O_3$, 226.1317).

Ethyl 4-butyl-1,2,3,4-tetrahydro-6-methyl-2-oxopyrimidine-5-carboxylate ***7***
White solid, (Mp: 146-149 °C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.2 (1H, m), 4.1 (2H, q, *J*= 7.1 Hz), 2.2 (3H, s), 1.4 (2H, m), 1.2 (7H, m), 0.9 (3H, t, *J*= 7.4Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.4, 154.8, 146.8, 101.5, 59.9, 51.4, 36.8, 31.4, 24, 22.5, 14.3, 14. IR (KBr)ν_{max}cm^{-1}: 1744, 1717, 1705, 1637, 1440, 1107. HR-ESIMS *m/z*: 241.1546 (calculated for $C_{12}H_{20}N_2O_3$, 240.1474).

Ethyl-methyl-2-oxo-4-pentyl-1,2,3,4-tetrahydropyrimidine-5-carboxylate ***8***
White solid, (Mp: 150-152°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.8 (1H, s), 4.1 (1H, m), 4 (2H, q, *J*= 7.3 Hz), 3.2 (1H, s), 2.1 (3H, s), 1.4 (2H, m), 1.9 (6H, m), 0.8 (3H, t, *J*= 7.6 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.4, 154.8, 146.8, 101.5,

59.9, 51.4, 31.4, 37.4, 24, 22.5, 18.5, 14.3, 14. IR (KBr)ν_{max} cm^{-1}: 1744, 1705, 1637, 1617, 1440. HR-ESIMS *m/z*: 255.1703 (calculated for $C_{13}H_{22}N_2O_3$, 254.1630).

Ethyl-4-hexyl-1,2,3,4-tetrahydro-6-methyl-2-oxo-pyrimidine-5-carboxylate ***9***
White solid, (Mp: 153-156°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.6 (1H, s), 4.1 (1H, m), 4 (2H, q, *J*= 7.2 Hz), 3 (1H, s), 2.1 (3H, s), 1.4 (2H, m), 1.2 (8H, m), 0.8 (3H, t, *J*= 7.3 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7, 153.2, 145.8, 100.3, 59.5, 50.4, 32.2, 24.3, 23.1, 19.29, 15.3, 14.2, 13.9. IR (KBr)ν_{max} cm^{-1}: 1744, 1705, 1637, 1617, 1440. HR-ESIMS *m/z*: 269.1853 (calculated for $C_{14}H_{24}N_2O_3$, 268.1787).

Ethyl4-(3-hydroxystyryl)-1,2,3,4-tetrahydro-6-methyl-2-oxopyrimidine-5-carboxylate ***10***
Yellowish powder solid, (Mp: 135-137°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.3 (1H, m), 7.3 (1H, m), 7.2 (1H, m), 7.2 (1H, m), 6.5 (1H, d, *J*= 16 Hz), 6.2 (1H, dd, *J*= 3.9,16 Hz), 4.2 (1H, m), 4.1 (2H, q, *J*= 7.4 Hz), 2.3 (3H, s), 1.3 (3H, t, *J*= 7.3Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.6, 158.4, 154.5, 144.6, 130.6, 130.1, 129.7, 128.8, 127.6, 118.3, 109.7, 101.8, 59.7, 53.7, 18.4, 14.9. IR (KBr)$\nu_{max}$$cm^{-1}$: 3240, 3117, 2960, 1693, 1514, 1462. HR-ESIMS *m/z*: 303.1339 (calculated for $C_{16}H_{18}N_2O_4$, 302.1267).

Ethyl4-(3,4-dihydronaphthalen-2-yl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate ***11***
Pale yellow solid, (Mp: 181-183°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.1-7.2 (4H, m),6.3 (1H, s), 5.3 (1H, s), 5 (1H, s), 4.1 (2H, q, *J*= 7.6 Hz), 2.8 (2H, t, *J*= 7.6 Hz), 2.3 (3H, s), 2.2 (2H, m), 1.2 (3H, t, *J*= 7.5 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.7, 155.7, 149.5, 142.3, 136.1, 135.2, 128.2, 128.4, 127.5, 125, 99.8, 61.1, 29.2, 23.9, 18.3, 14.8. IR (KBr)ν_{max} cm^{-1}: 3240, 3117, 2960, 1693, 1728, 1681, 1608, 1514. HR-ESIMS *m/z*: 313.1546 (calculated for $C_{18}H_{20}N_2O_3$, 312.1474).

Ethyl-4-(1-chloro-3,4-dihydronaphthalen-2-yl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyr imidine-5-carboxylate ***12***
White solid, (Mp: 162-164°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.6 (1H, m), 7.2 (2H, m), 7.1 (1H, m), 5.8 (1H, s), 5.3 (1H, s), 5.2 (1H, s), 4.1 (2H, q, *J*= 6.7 Hz), 2.8 (2H, m), 2.3 (3H, s), 2.4 (2H, m), 1.1 (3H, t, *J*= 7.3Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.5, 154.8, 150.3, 137.7, 137.4, 134.14, 129.5, 128.3, 127.9, 127.4, 126.2, 98.6, 61.4, 29.2, 25.1, 18.9, 15.2. IR (KBr)ν_{max} cm^{-1}: 3235, 2936, 1693, 1700, 1681, 1596. HR-ESIMS *m/z*: 347.1156 (calculated for $C_{18}H_{19}ClN_2O_3$, 346.1084).

Ethyl-4-((Z)-3-(methoxycarbonyl)-1-chloro-1-(3,4-dimethoxyphenyl)prop-1-en-2-yl)-1,2,3,4-tetrahydro-6-methyl-2-oxopyrimidine-5-carboxylate ***13***
Brown powder solid, (Mp: 126-128°C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.1 (1H, d, *J*= 2.7 Hz), 7 (1H, dd, *J*= 2.3&8.3 Hz), 6.9 (1H, d, *J*= 8.3 Hz), 4.5 (1H, s), 4.2 (2H, q, *J*= 7.2 Hz), 3.6 (6H, s), 3.6 (3H, s), 2.7 (2H, s), 2.2 (3H, s), 1.3 (3H, t, *J*= 6.9 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 174, 169.9, 155.7, 151.3, 150.1, 148.5, 143.4, 134.3, 123.1, 121.6, 113.8, 113, 95.1, 61.5, 57.6, 56.7, 51.8, 36.3, 16.3, 14.7. IR (KBr)ν_{max} cm^{-1}: 3195, 2860, 1693, 1514, 1462. HR-ESIMS *m/z*: 453.1423 (calculated for $C_{21}H_{25}ClN_2O_7$, 452.1350).

Ethyl-1,2,3,4-tetrahydro-6-methyl-4-(2,2-dimethylchroman-6-yl)-2-oxo-pyrimidine-5- carboxylate ***14***
Yellowish solid, (Mp: 134-137 °C). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.5 (1H, s), 7 (2H, m), 6.6 (1H, d, *J*= 2 Hz), 5.4 (1H, m), 5.3 (1H, s), 4.1 (2H, q, *J*= 7.6 Hz), 2.7 (2H, t, *J*= 7.5 Hz), 2.3 (3H, s), 1.7 (2H, t, *J*= 7.5 Hz), 1.3 (6H, s), 1.2 (3H, t, *J*= 7.6 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.8, 153.7, 145.9, 135, 127.7, 125.7, 120.9, 117.2, 101.6, 74.3, 59.9, 55.2, 54, 32.6, 30.2, 26.8, 22.5, 18.6, 14.1. IR (KBr) ν_{max} cm^{-1}: 3335, 3156, 1693, 1700, 1608, 1576, 1488. HR-ESIMS *m/z*: 345.1822 (calculated for $C_{19}H_{24}N_2O_4$, 344.1736).

Ethyl-1,2,3,4-tetrahydro-6-methyl-4-((Z)-2-(2,2-dimethylchroman-6-yl)vinyl)-2-oxo pyrimidine-5-carboxylate ***15***
Yellowish solid, (Mp: 120-123 °C) ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.3 (1H, s), 7.2 (2H, m), 6.7 (1H, dd, *J*= 8.5&2.4 Hz), 6.2 (1H, d, *J*= 16 Hz), 5.9 (1H, dd, *J*= 3.2&16.0 Hz), 4.5 (1H, d, *J*= 4.0 Hz), 4.1 (2H, q, *J*= 7.3 Hz), 2.4 (3H, s), 2.7 (2H, t, *J*= 7.0 Hz) 1.7 (2H, t, *J*= 7.0 Hz), 1.2 (3H, t, *J*= 7.1 Hz), 1.2 (6H, s). ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.6, 155.9, 154.6, 152.4, 130.5, 129, 128, 125.8, 122.8, 115.1, 100.7, 78.3, 61.5, 34.4, 27.4, 25, 19.5,14.4. IR (KBr)$\nu_{max}$$cm^{-1}$: 3240, 3117, 2960, 1693, 1514, 1097. HR-ESIMS m/z: 371.11979 (calculated for $C_{21}H_{26}N_2O_4$, 370.1893).

Ethyl-4-(4-methoxystyryl)-6-methyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxyl- ate ***16***
Colourless liquid.^{1}H NMR (400 MHz, $CDCl_3$): δ 7.2 (1H, s), 7 (2H, d, *J*= 7.9 Hz), 6.8 (2H, d, *J*= 8 Hz), 6.2 (1H, d, *J*= 16 Hz), 5.9 (1H, dd, *J*= 3.2, 16 Hz), 5.5 (1H, s), 4.5 (1H, d, *J*= 4 Hz), 4.1 (2H, q, *J*= 7.3 Hz), 3.7 (3H, s), 2.4 (3H, s), 1.2 (3H, t, *J*= 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.6, 154.5, 144.6, 132.9, 129.7, 129.3, 127.4, 127.3, 118.3, 109.7, 101.8, 85.3, 59.7, 55.2, 36.4, 19.5,14.4. IR (KBr)$\nu_{max}$$cm^{-1}$: 3240, 2960, 1693, 1514, 1462, 1097. HR-ESIMS *m/z*: 317.1495 (calculated for $C_{17}H_{20}N_2O_4$, 316.1423).

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Ethyl 4-(2,3-dimethoxystyryl)-1,2,3,4-tetrahydro-6-methyl-2-oxopyrimidine-5-carboxylate ***17***
Yellowish liquid, ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.2 (1H, s), 6.8 (2H, d, *J*= 16 Hz), 6.5 (3H, m),5.9 (1H, dd, *J*= 3.2, 16 Hz), 5.5 (1H, s), 4.5 (1H, d, *J*= 4 Hz), 4.1 (2H, q, *J*= 7.4 Hz), 3.7 (6H, s), 2.4 (3H, s), 1.2 (3H, t, *J*= 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.7, 156.8, 155.7, 151.2, 144.6, 130, 127.1, 122.5, 119.9, 104.5, 101.9, 93.8, 59.7, (2C, 55.3), 36.4, 19.5, 14.3. IR (KBr)ν_{max}cm^{-1}: 3240, 2960, 1693, 1514. HR-ESIMS *m/z*: 347.1601 (calculated for $C_{18}H_{22}N_2O_5$, 346.1529).

Ethyl 4-(3,4,5-trimethoxystyryl)-1,2,3,4-tetrahydro-6-methyl-2-oxopyrimidine-5-carboxylate ***18***
Greyish liquid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.2 (1H, s), 6.5 (2H, d, *J*= 16 Hz), 6.2 (2H, s), 5.9 (1H, dd, *J*= 3.2, 16 Hz), 5.5 (1H, s), 4.5 (1H, d, *J*= 3.4 Hz), 4.1 (2H, q, *J*= 7.6 Hz), 3.7 (9H, s), 2.4 (3H, s), 1.2 (3H, t, *J*= 7.1 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.8, 156.8, 155.7, 150.2, 144.6, 130, 127.1, 122.5, 104.5, 101.9, 59.7, 55.3, 55.3, 55.3, 36.4, 19.5, 14.34. IR (KBr)ν_{max}cm^{-1}: 3240, 2960, 1693, 1514, 1462. HR-ESIMS *m/z*: 377.1822 (calculated for $C_{19}H_{24}N_2O_6$, 376.1634).

Ethyl-4-(2,4-dimethoxystyryl)-1,2,3,4-tetrahydro-6-methyl-2-oxopyrimidine-5-carboxylate ***19***
Yellowish liquid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.2 (1H, s), 7 (1H, d, *J*= 7.8 Hz), 6.8 (2H, d, *J*= 16 Hz), 6.2 (1H, m), 6.2 (1H, d, *J*= 1.8 Hz), 5.96(1H, dd, *J*= 3.2, 16 Hz), 5.5 (1H, s), 4.5 (1H, d, *J*= 4 Hz), 4.1 (2H, q, *J*= 7.3Hz), 3.7 (6H, s), 2.4 (3H, s), 1.2 (3H, t, *J*= 7.1Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.7, 159.8, 156.7, 154.2, 144.6, 130, 127.1, 122.5, 119.9, 104.5, 101.9, 93.8, 59.7, (2C, 55.3), 36.4, 19.5, 14.3. IR (KBr)ν_{max}cm^{-1}: 3240, 2960, 1693, 1514, 1097. HR-ESIMS *m/z*: 347.1611 (calculated for $C_{18}H_{22}N_2O_5$, 346.1529).

Ethyl4-((E)-2-(benzo[d][1,3]dioxol-6-yl)vinyl)-1,2,3,4-tetrahydro-6-methyl-2-oxo pyrimidine-5-carboxylate ***20***
White solid, (Mp: 181-184°C). ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.8 (1H, s), 6.9 (1H, d, *J*= 8.5 Hz), 6.7 (1H, m), 6.6 (1H, d, *J*= 2.2 Hz), 6.3 (1H, d, *J*= 16.0 Hz), 5.9 (2H, s), 5.9 (1H, dd, *J*= 3.4&16.0 Hz), 4.61 (1H, d, *J*= 3.4 Hz), 5.6 (1H, s), 4.1 (2H, q, *J*= 7.2 Hz), 2.3 (3H, s), 1.2 (3H, t, *J*= 7.2 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.7, 154.2, 148.8, 146.9, 145.3, 136.8, 129.2, 124.3, 120.8, 109.1, 107.2, 102, 101.8, 60.2, 55.6, 19.4, 14.5. IR (KBr)ν_{max}cm^{-1}: 3230, 2930, 1710, 1655, 1520, 1107. HR-ESIMS *m/z*: 331.1309 (calculated for $C_{18}H_{22}N_2O_5$, 330.1216).

Ethyl-4-(benzo[d][1,3]dioxol-6-yl)-1,2,3,4-tetrahydro-6-methyl-2-oxopyrimidine-5-carboxylate ***21***
White solid, (Mp: 176-178°C. ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.9 (1H, s), 6.8 (1H, d, *J*= 8.7 Hz), 6.7 (1H, m), 6.7 (1H, d, *J*= 1.6 Hz), 5.9 (2H, s), 5.6 (1H, s), 5.3 (1H, s), 4.0 (2H, q, *J*= 7Hz), 2.3 (3H, s), 1.18 (3H, t, *J*= 7Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.6, 153.6, 147.9, 147.2, 146.3, 137.9, 120, 108.1, 107, 101.3, 101.1, 60, 55.4, 18.5, 14.2. IR (KBr)ν_{max}cm^{-1}: 3225, 2928, 1710, 1651, 1514, 1097. HR-ESIMS *m/z*: 305.1131 (calculated for $C_{18}H_{22}N_2O_5$, 304.1059).

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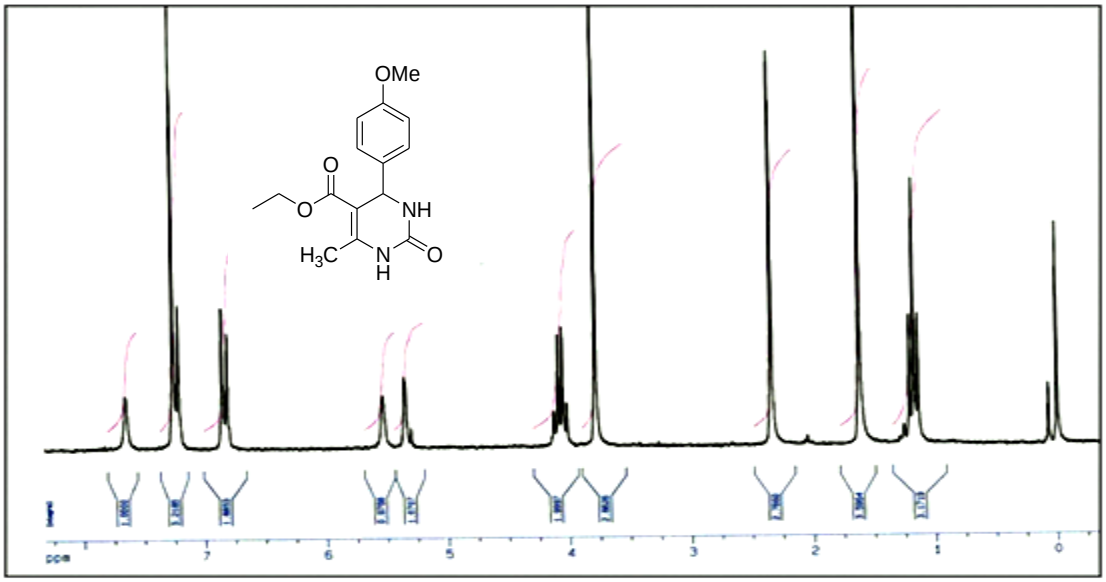

Figure S1. ^{1}H NMR spectrum of compound 4f.

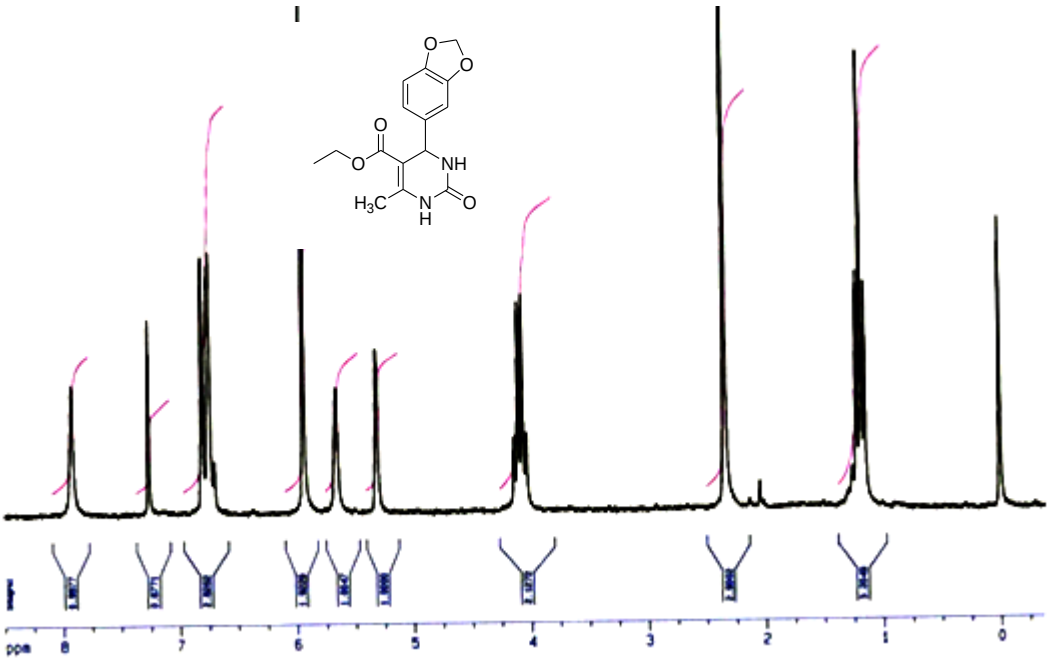

Figure S2. ^{1}H NMR spectrum of compound 4m.

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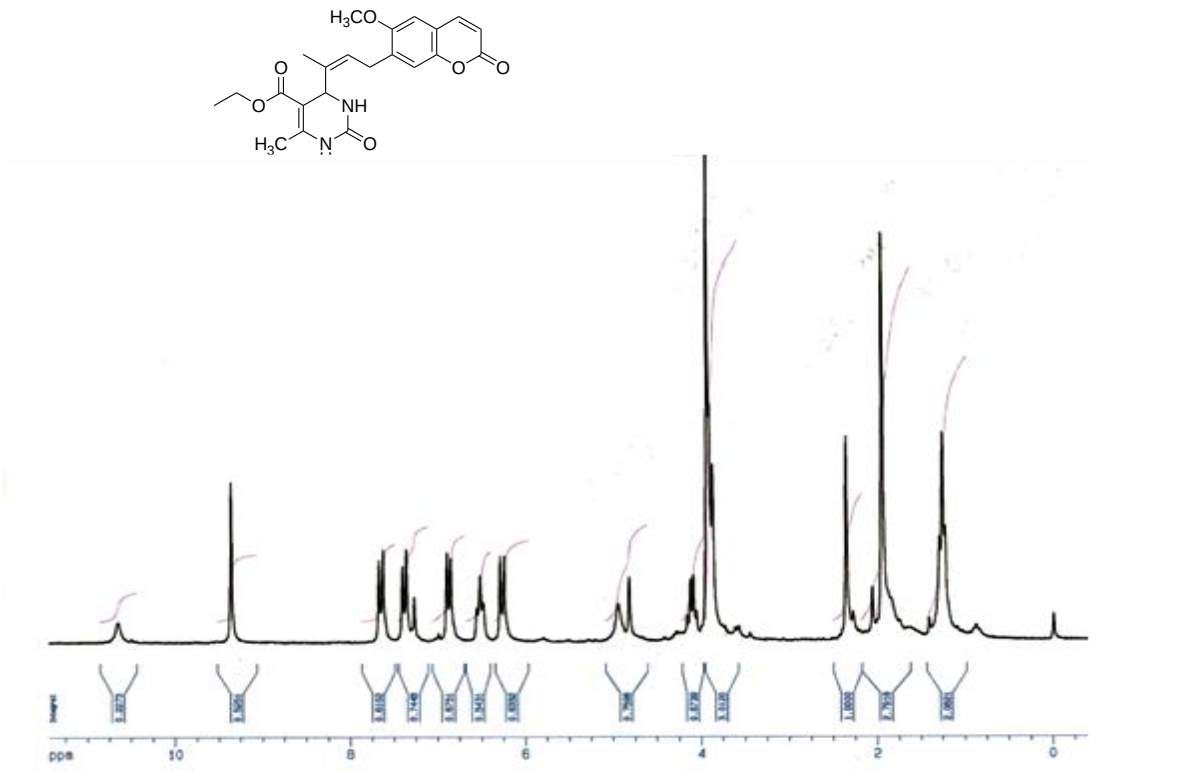

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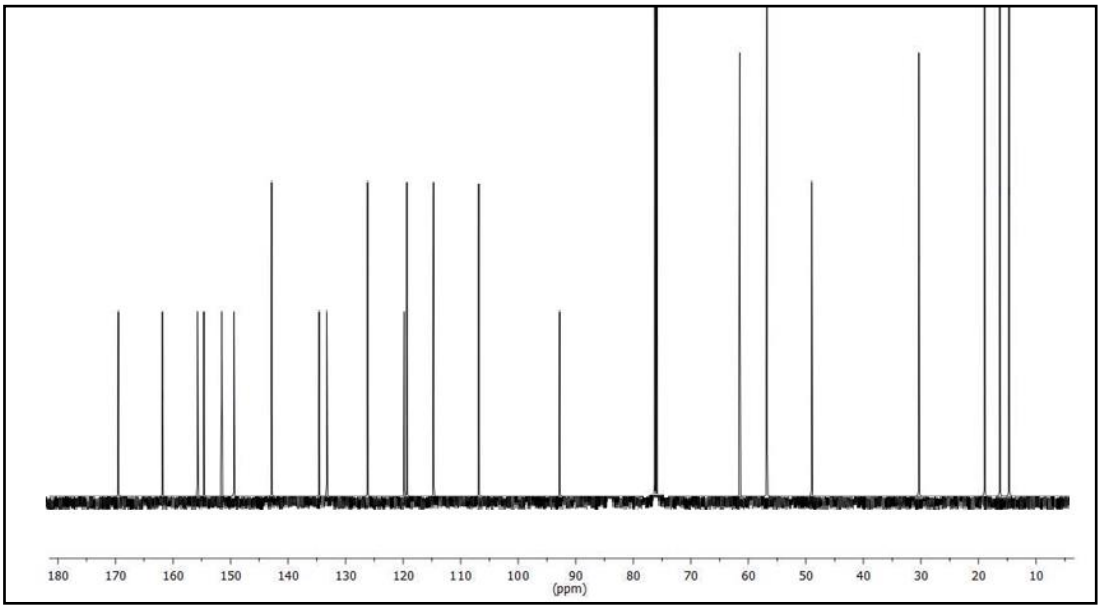

(b)

Figure S3. ^{1}H &^{13}C NMR spectra of compound 5.

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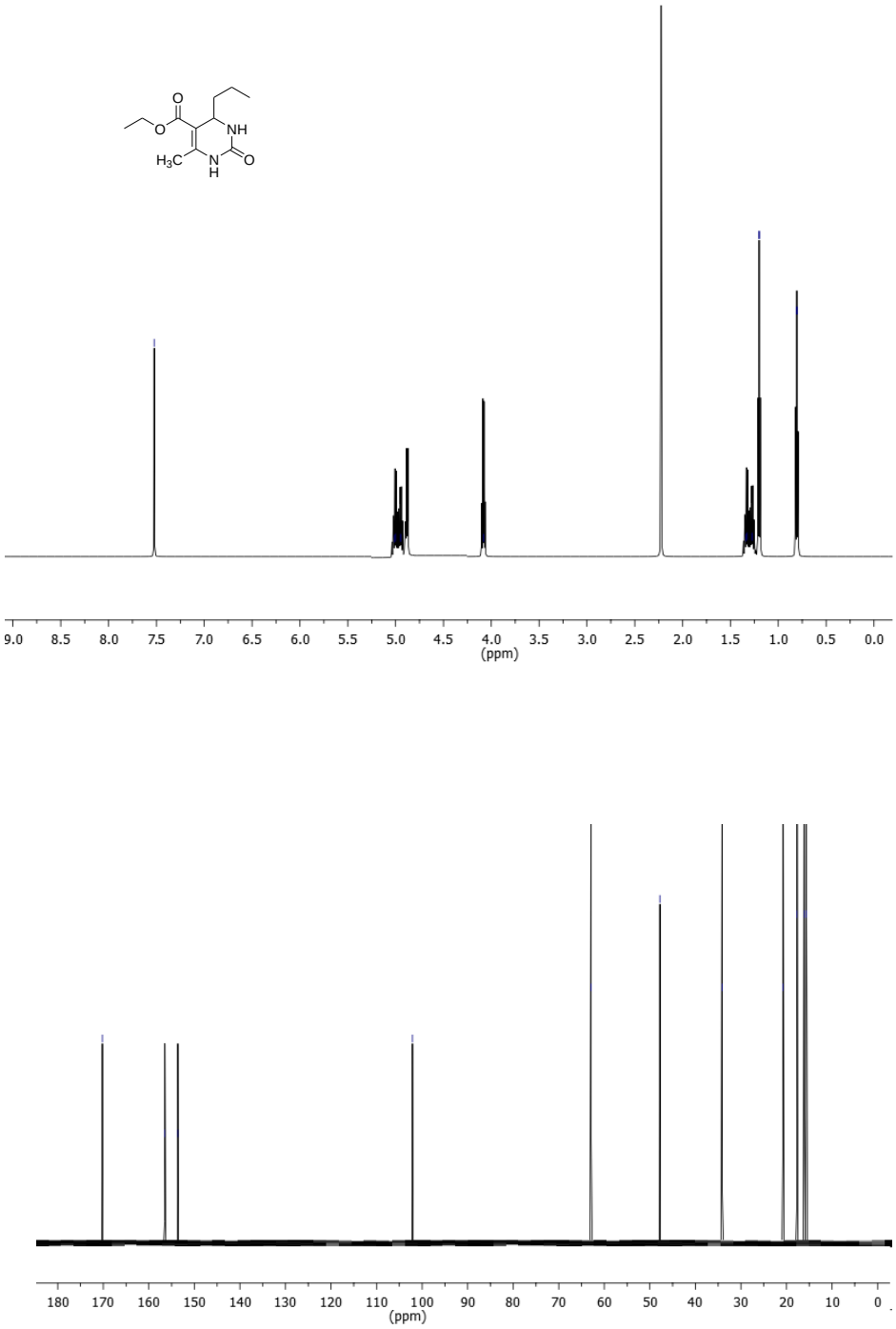

Figure S4. ^{1}H & ^{13}CNMR spectra of compound 6.

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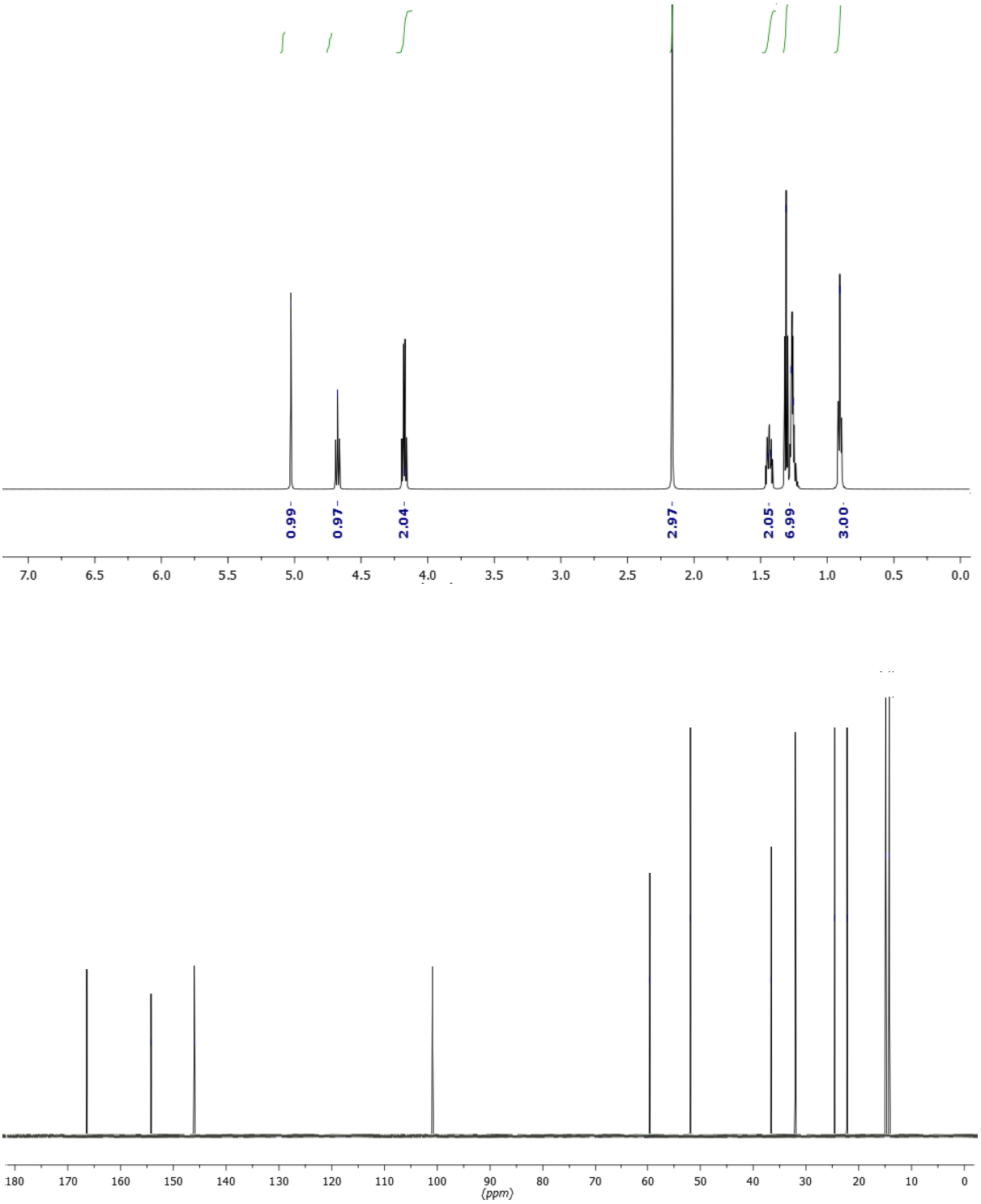

Figure S5. ^{1}H & ^{13}CNMR spectra of compound 7.

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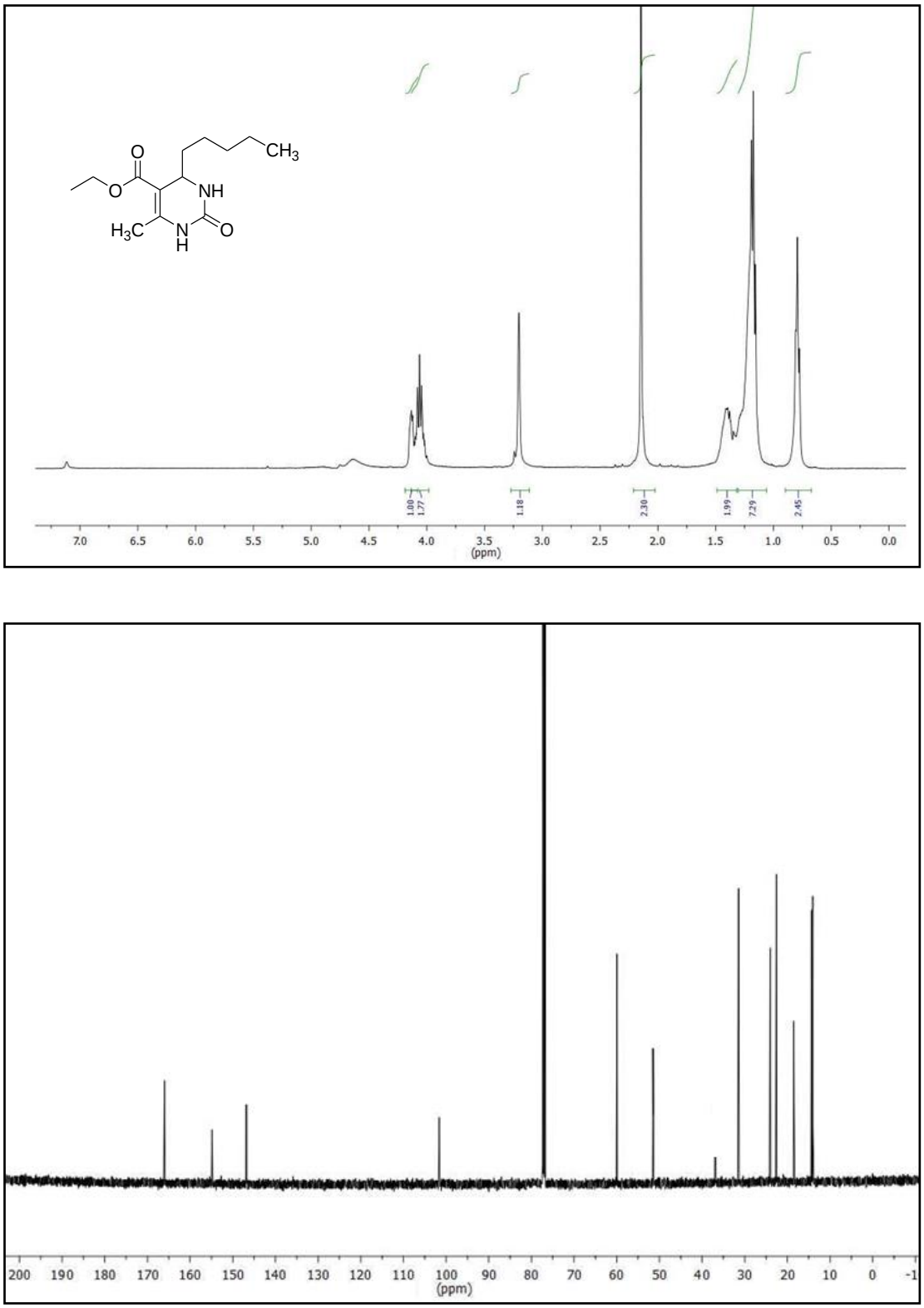

Figure S6. ^{1}H & ^{13}CNMR spectra of compound 8.

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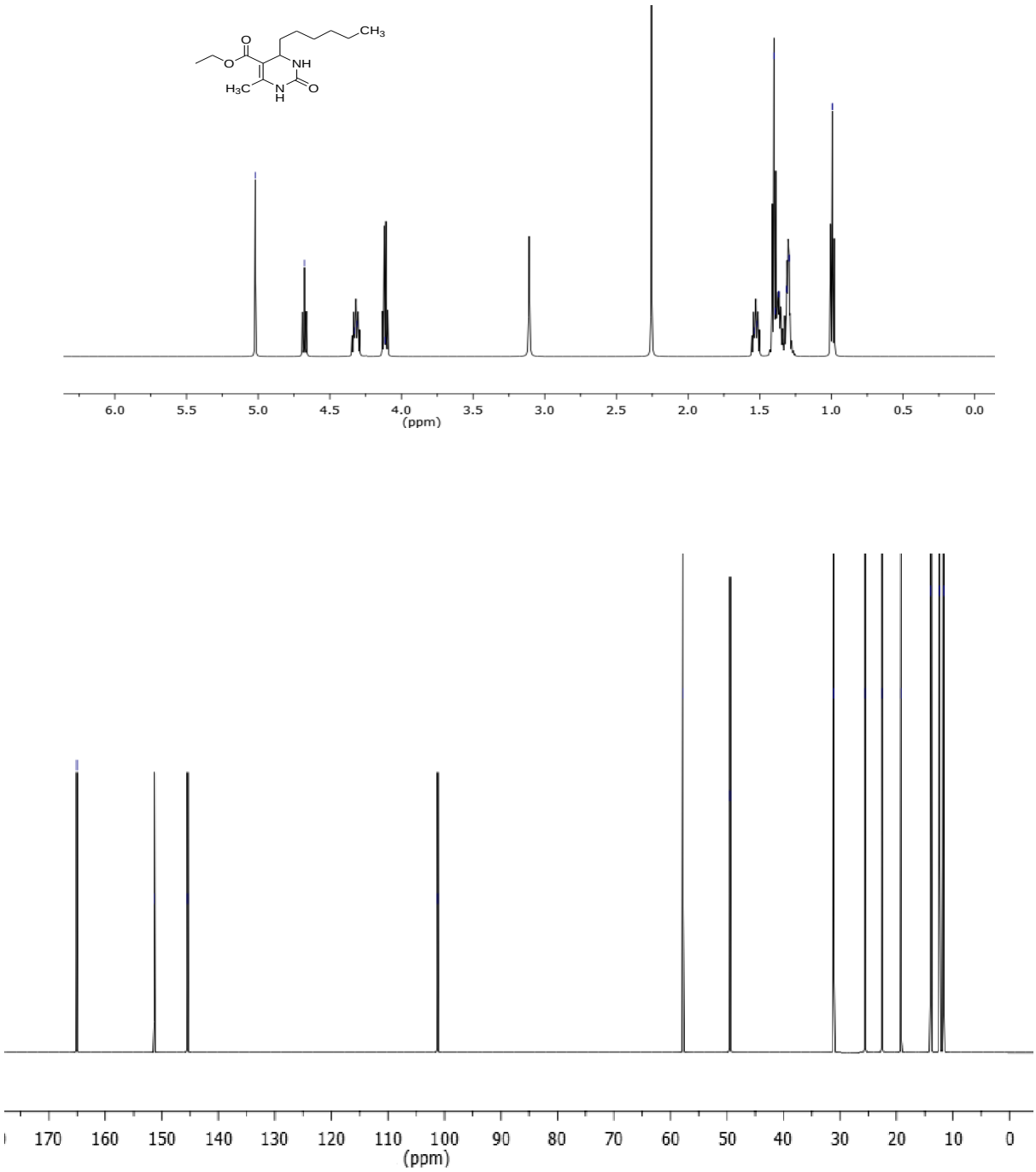

Figure S7. ^{1}H & ^{13}CNMR spectra of compound 9.

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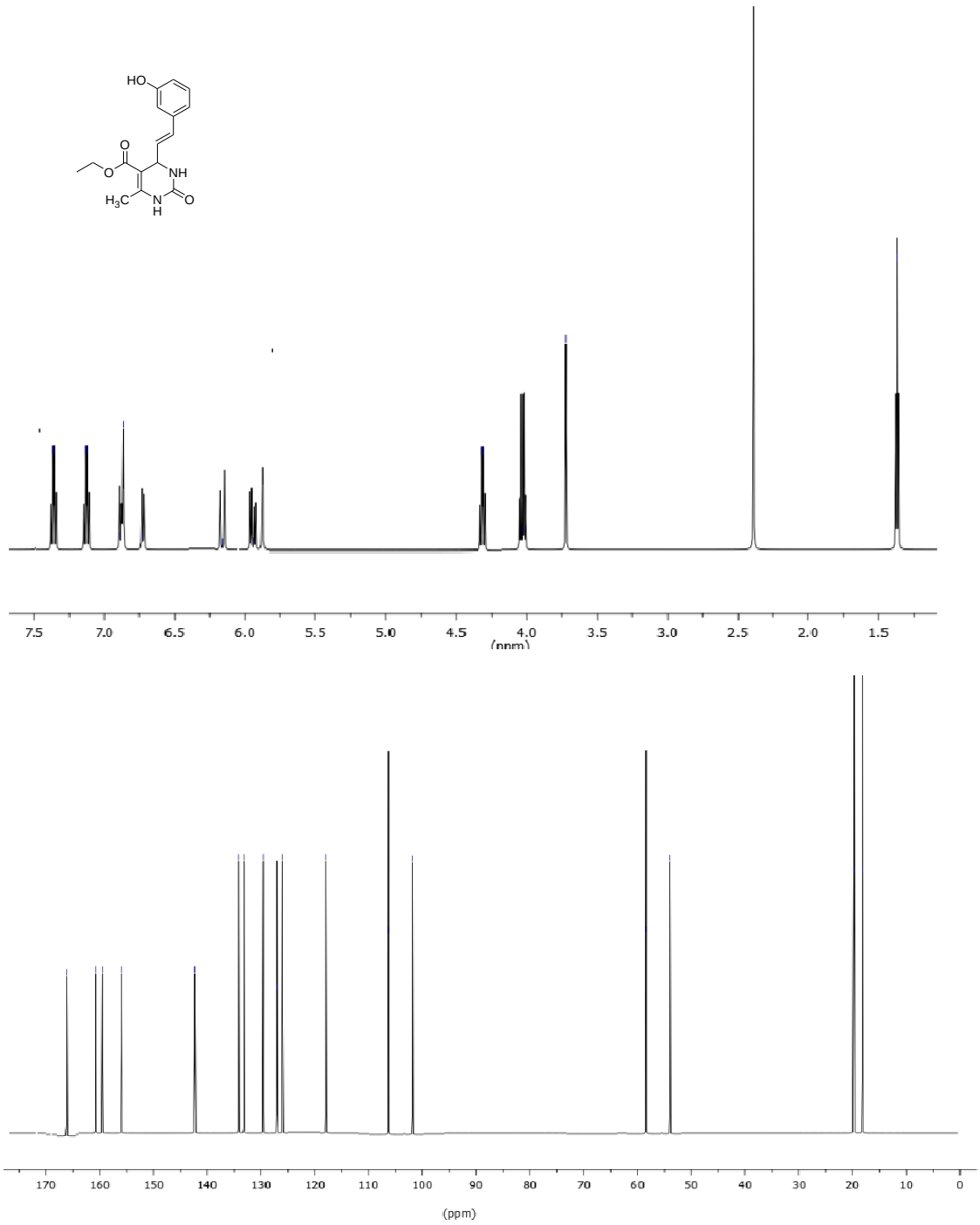

Figure S8. ^{1}H & ^{13}C NMR spectra of compound 10.

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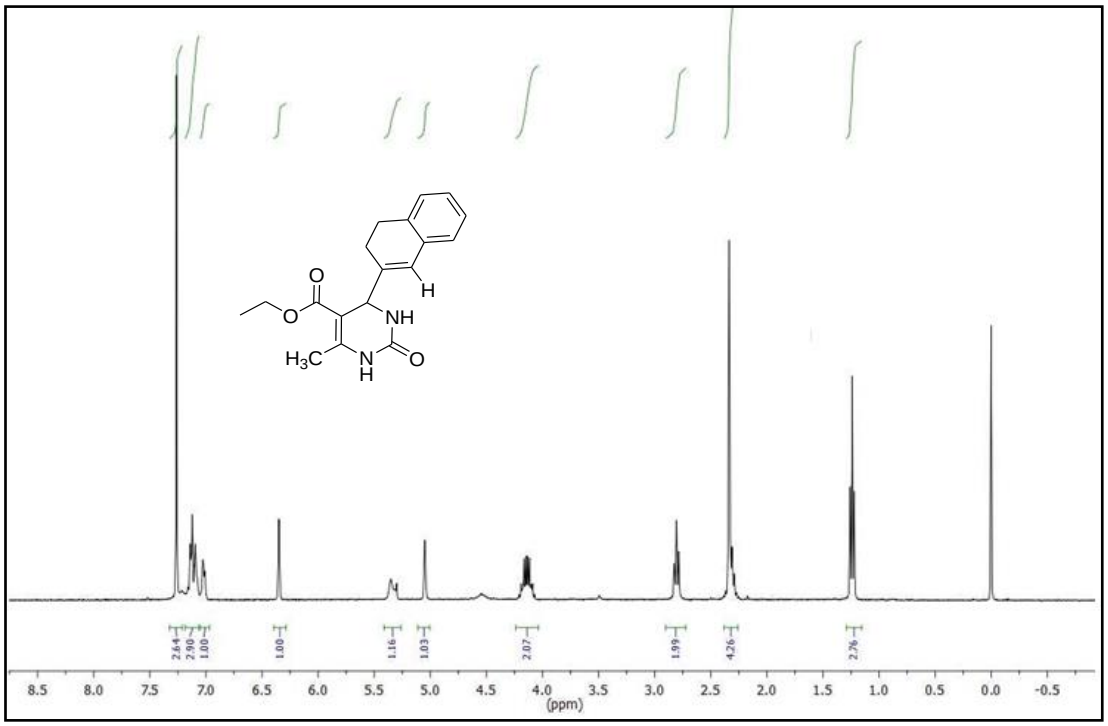

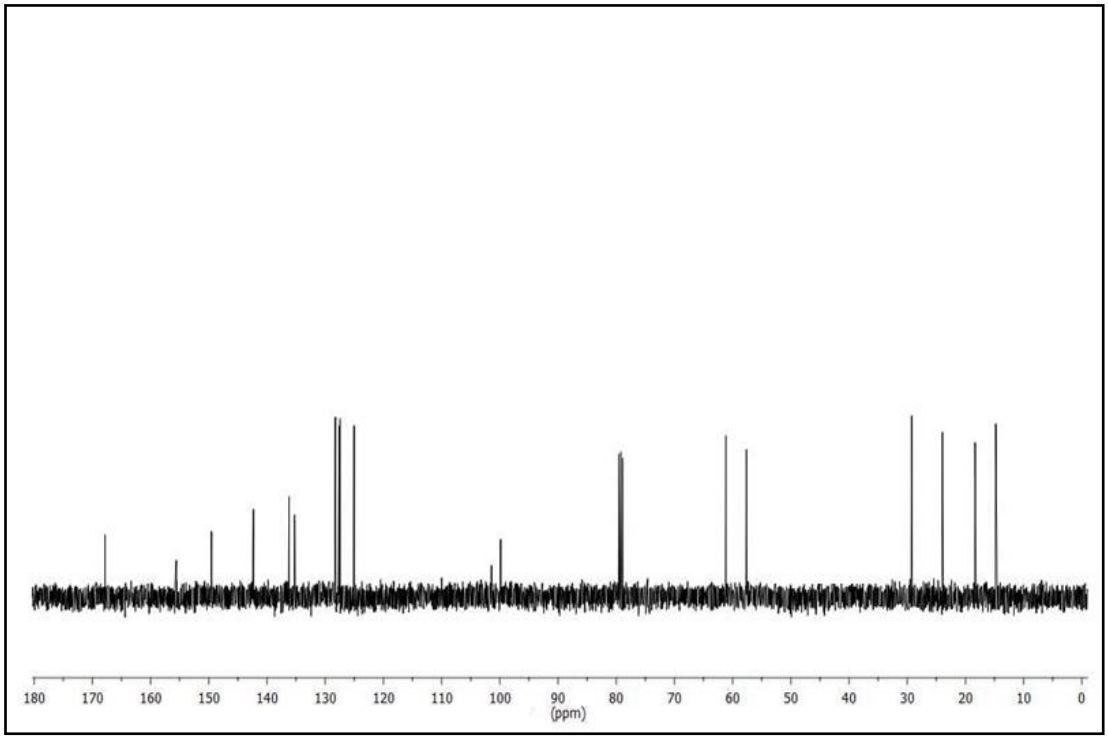

Figure S9. ^{1}H & ^{13}CNMR spectra of compound 11.

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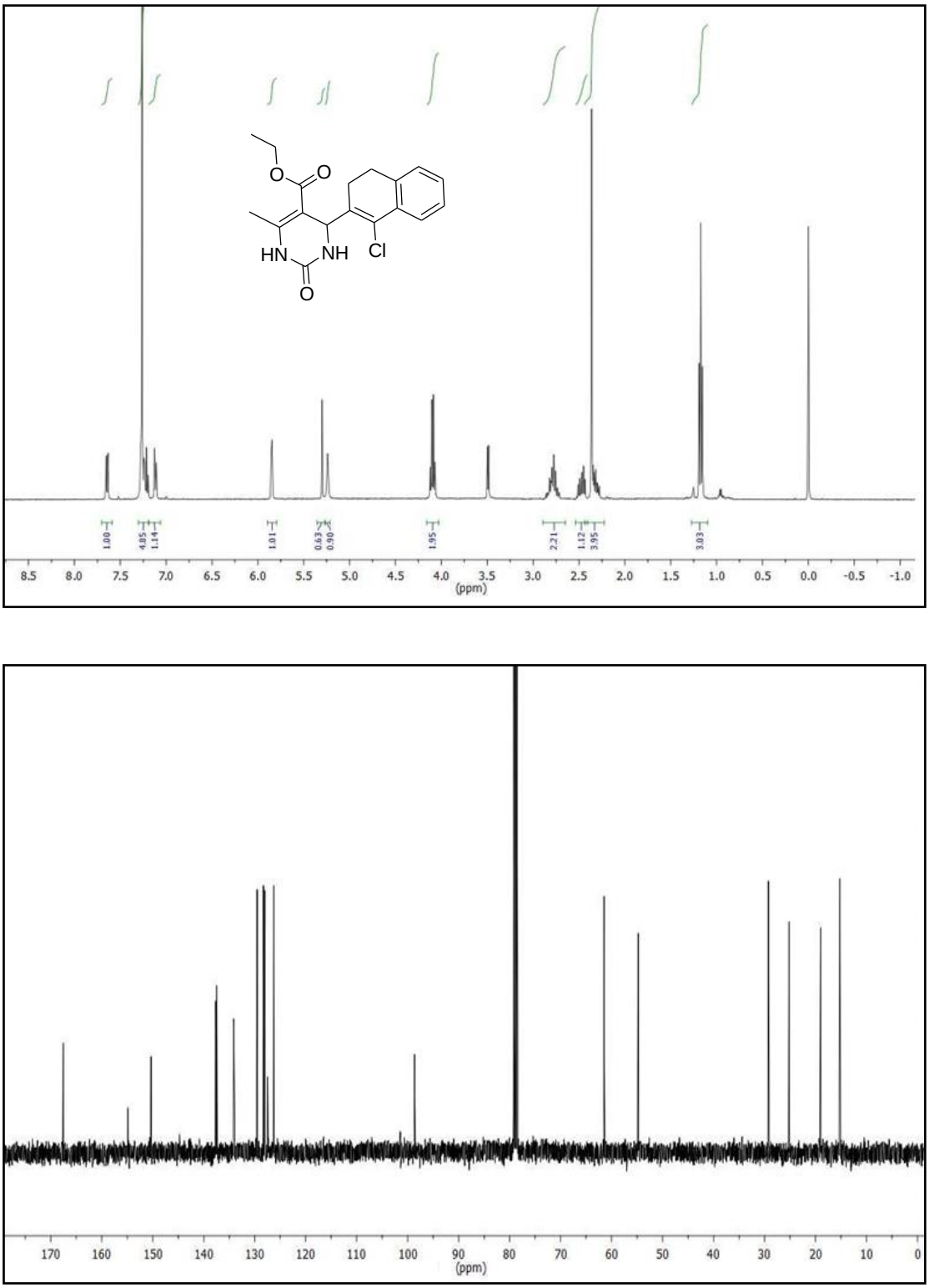

Figure S10. ^{1}H & ^{13}C NMR spectra of compound 12.

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https://doi.org/10.19261/cjm.2024.1135

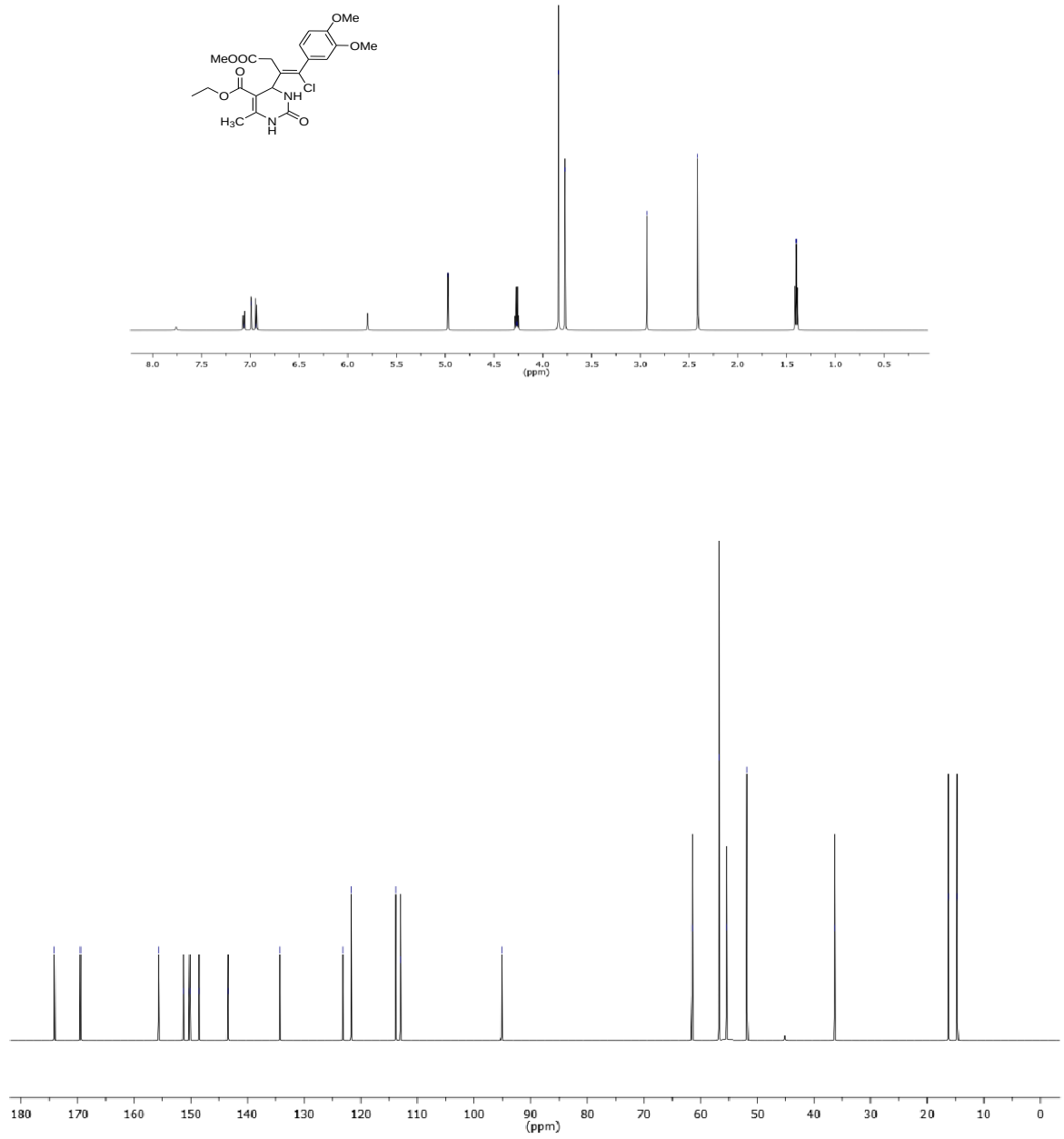

Figure S11. ^{1}H & ^{13}C NMR spectra of compound 13.

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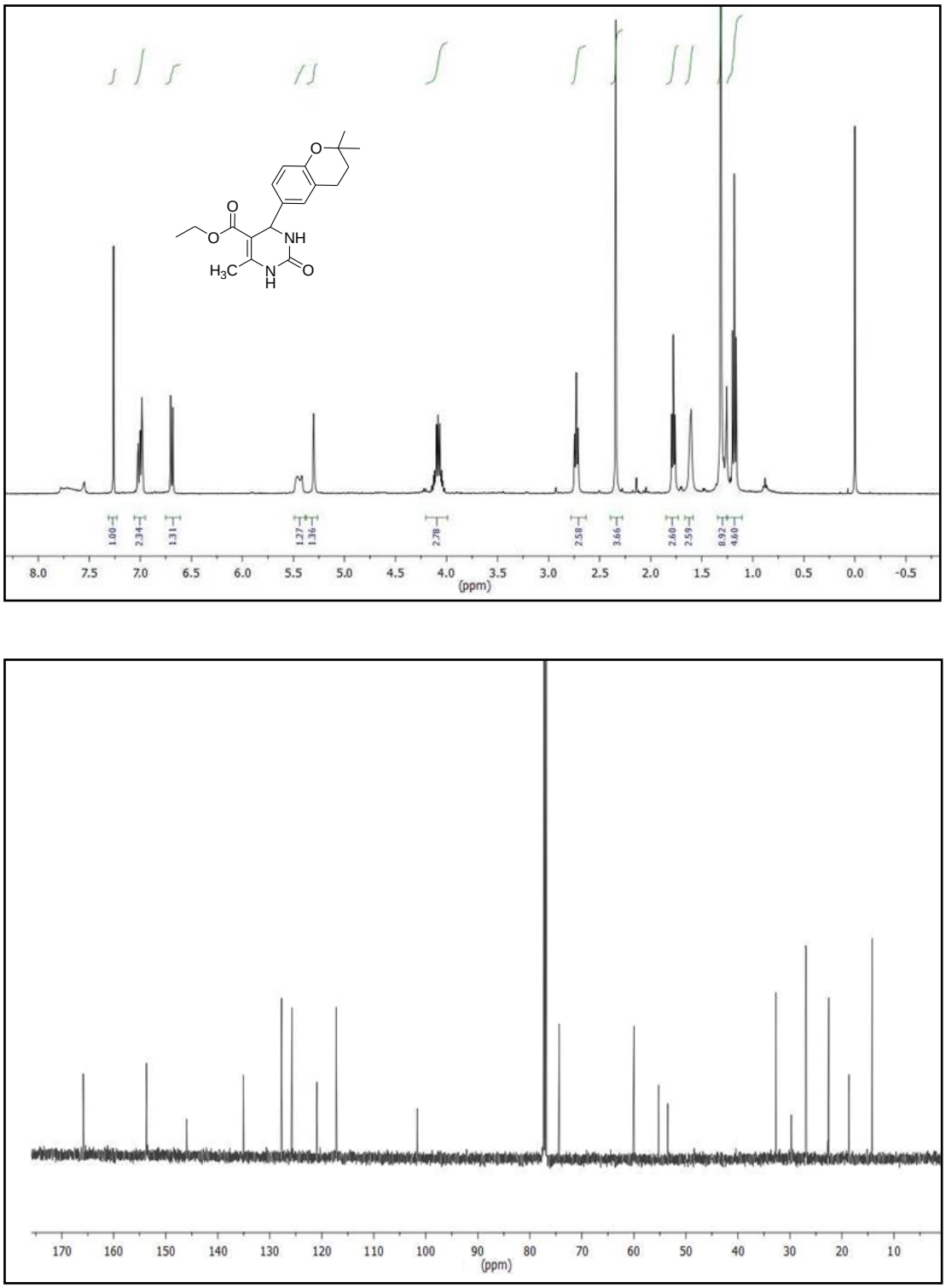

Figure S12. ^{1}H & ^{13}C NMR spectra of compound 14.

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https://doi.org/10.19261/cjm.2024.1135

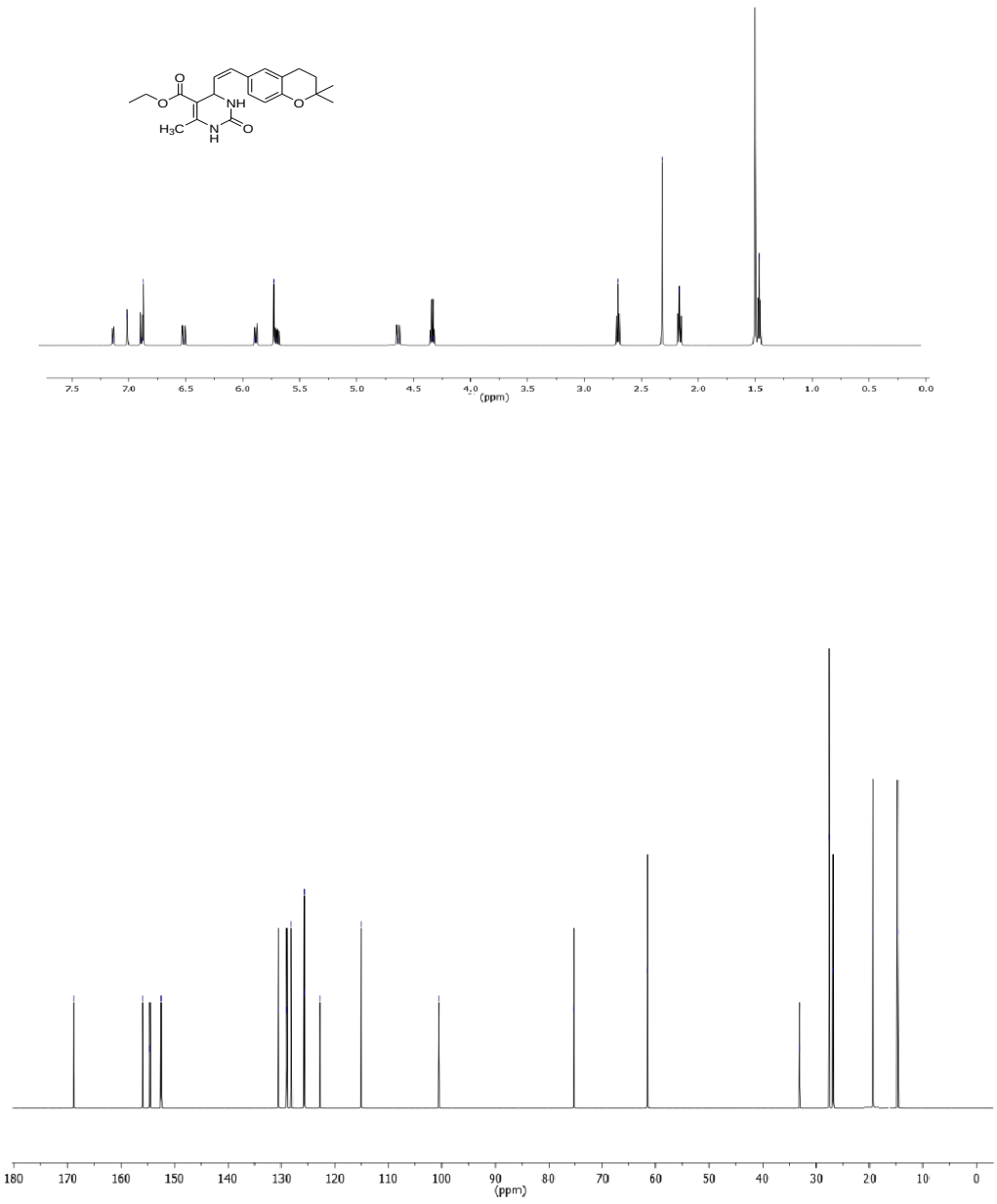

Figure S13. ^{1}H & ^{13}C NMR spectra of compound 15.

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https://doi.org/10.19261/cjm.2024.1135

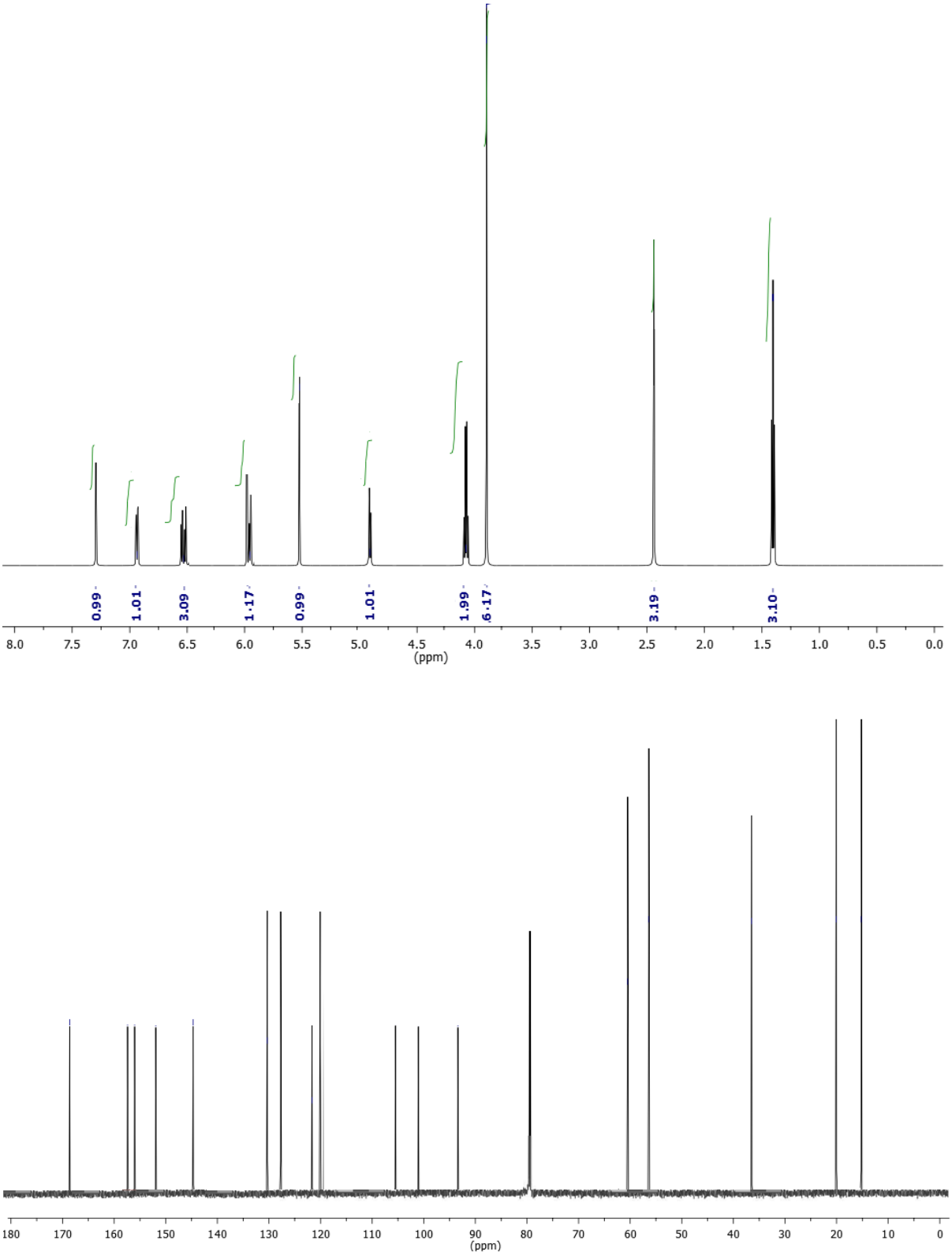

Figure S14. ^{1}H & ^{13}C NMR spectra of compound 17.

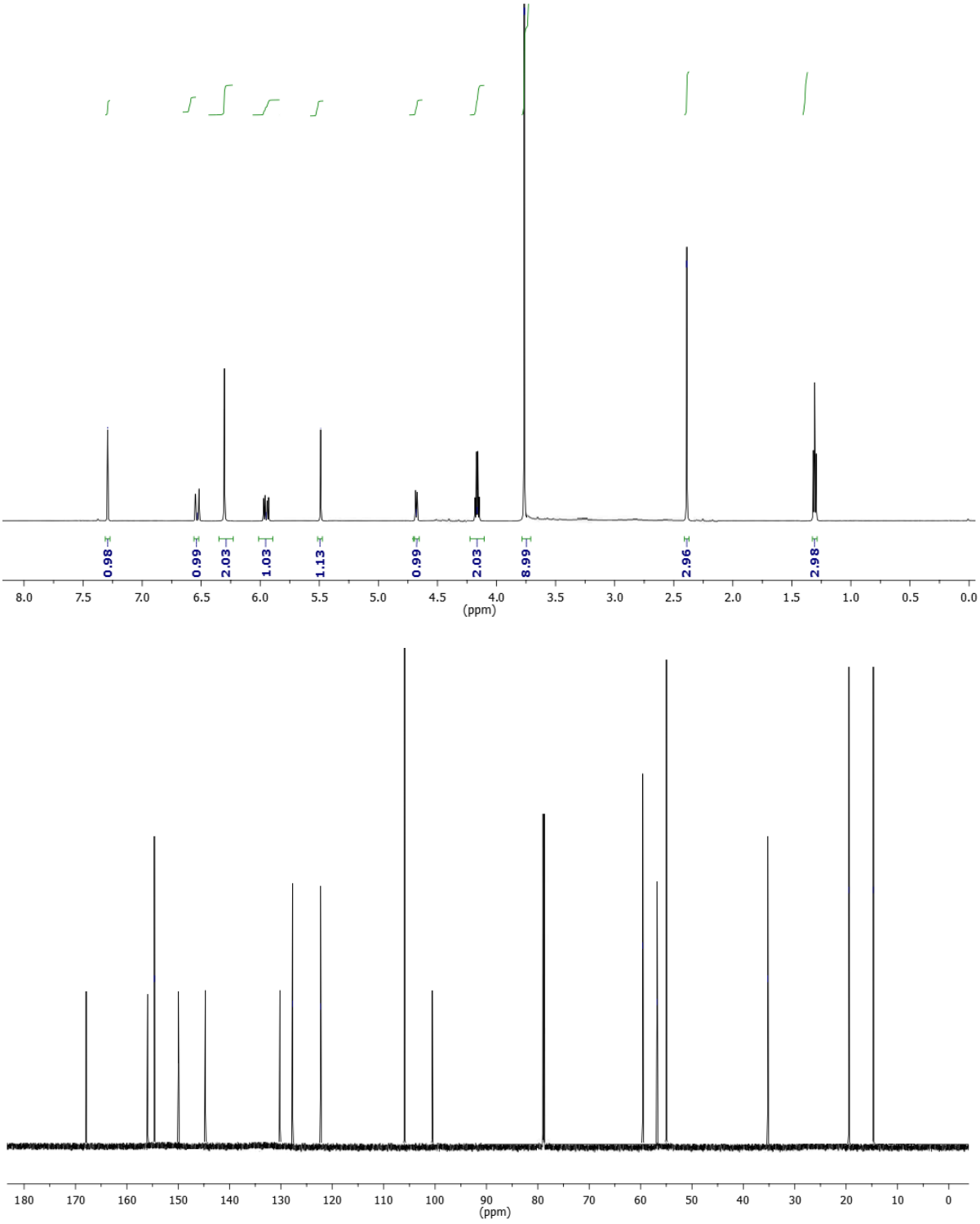

Figure S15. ^{1}H & ^{13}C NMR spectra of compound 18.

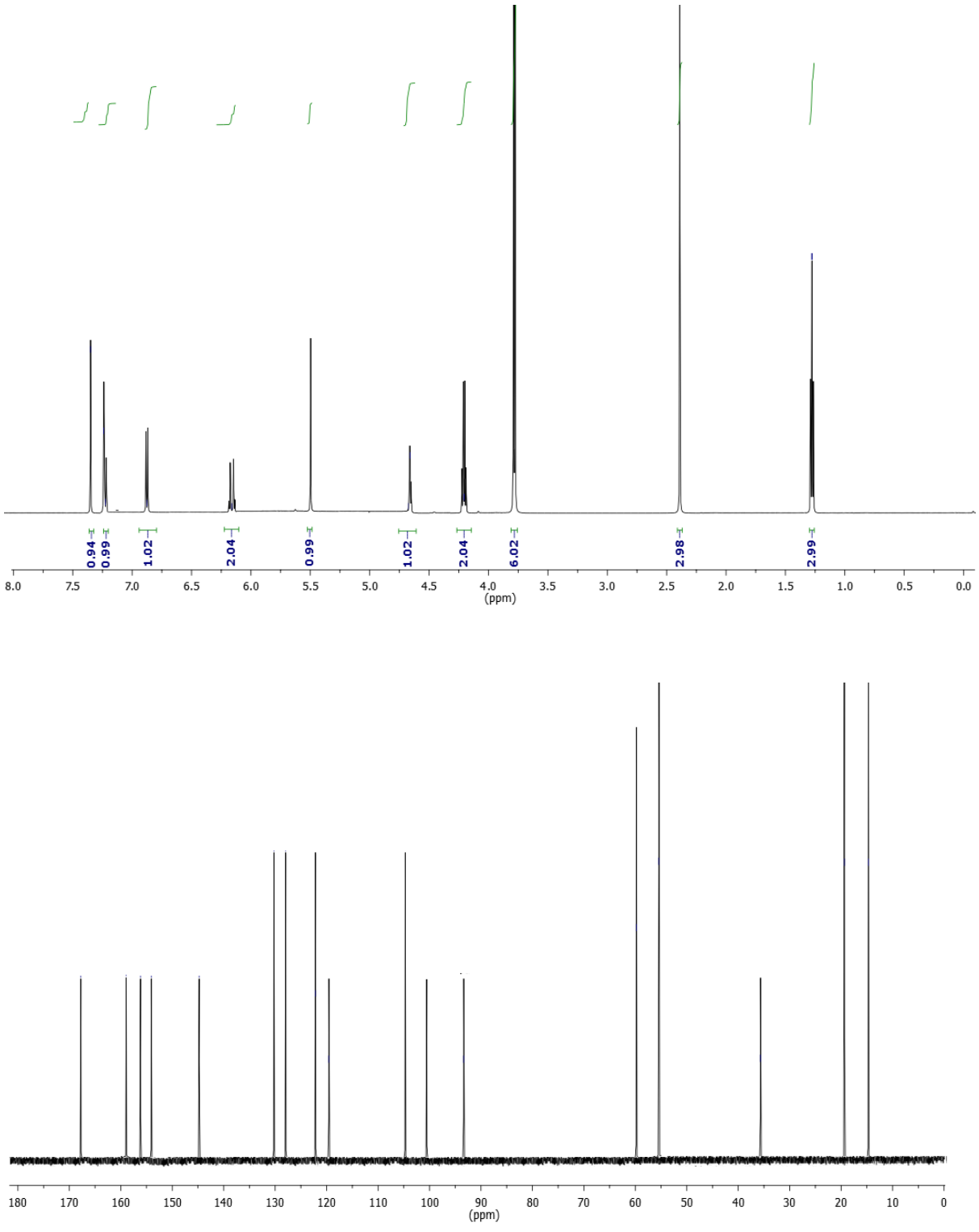

Figure S16. ^{1}H & ^{13}C NMR spectra of compound 19.

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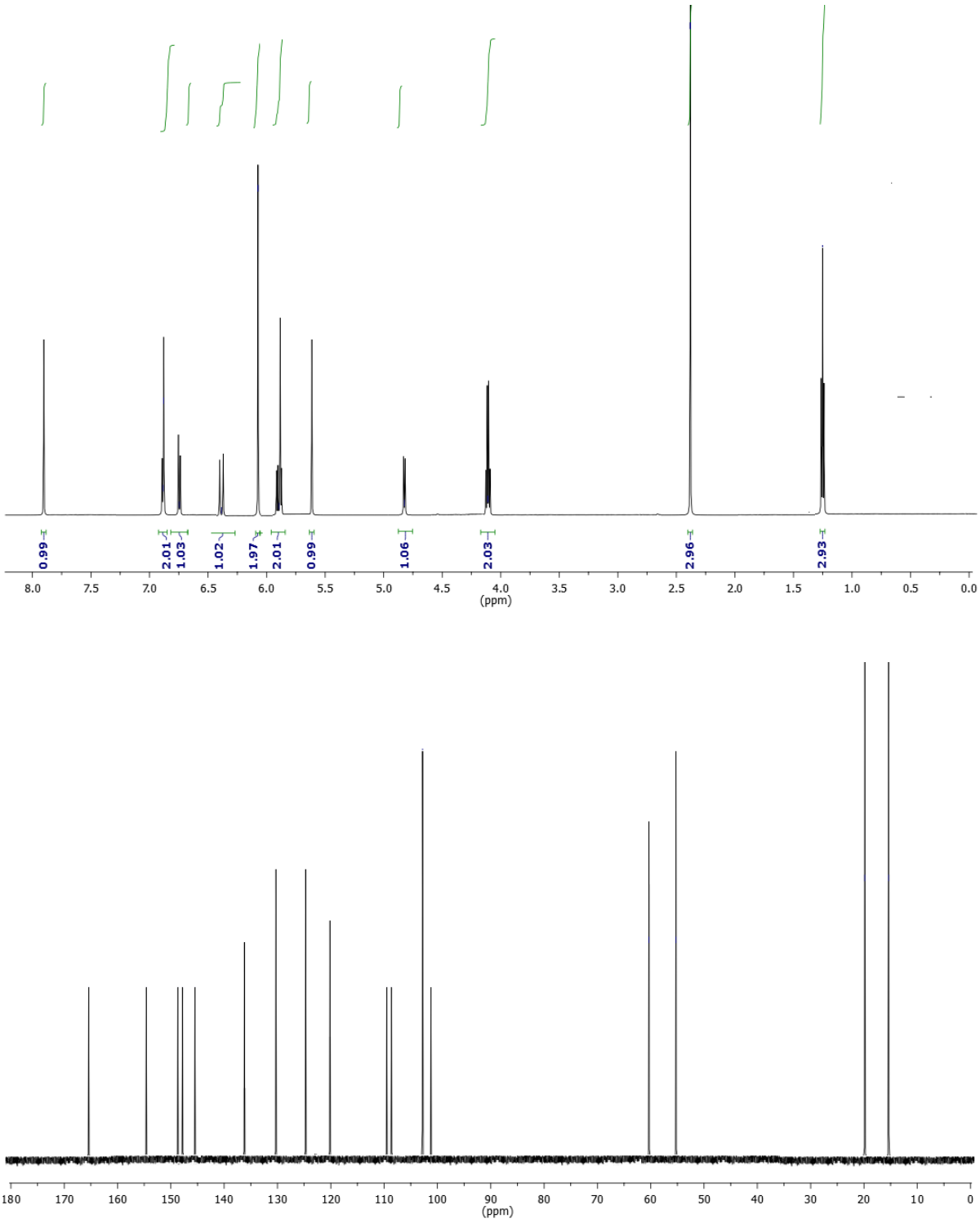

Figure S17. ^{1}H & ^{13}C NMR spectra of compound 20.

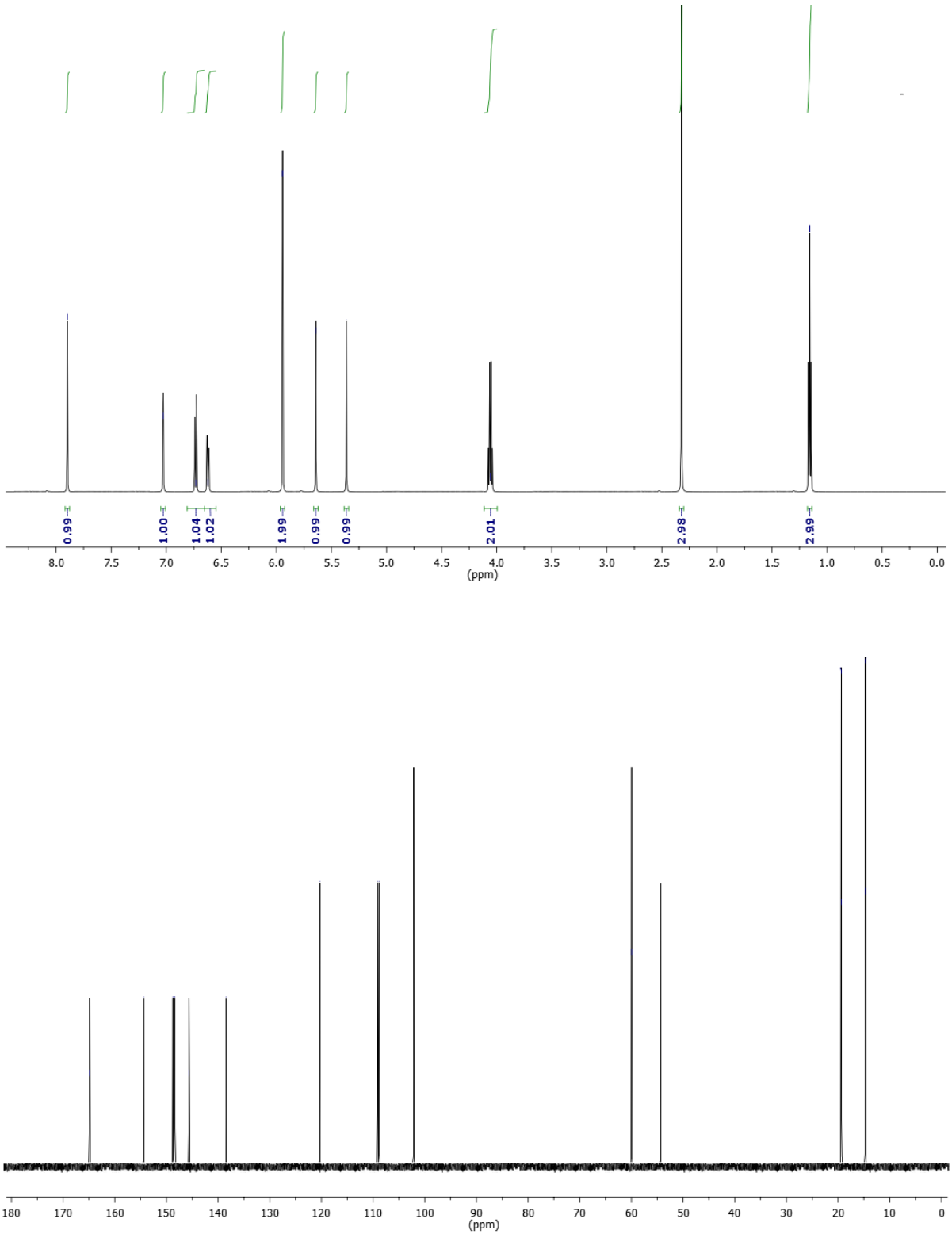

Figure S18. ^{1}H & ^{13}C NMR spectra of compound 21.