Supporting Information

One-pot three-component synthesis of 1-amidoalkyl naphthols and polyhydroquinolines using deep eutectic solvent: A green method and mechanistic insight

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Section S1. Chemicals, Supplies, and instruments

Chemicals, supplies

Choline chloride (assay 98%), cyclohexanecarboxaldehyde (assay 97%), 2-chlorobenzaldehyde (assay 99%), 4-chlorobenzaldehyde (assay 97%), 4-fluorobenzaldehyde (assay 98%), 4-*tert*-butylbenzaldehyde (assay 97%), 2-hydroxy-5-nitrobenzaldehyde (assay 98%), 2-carboxybenzaldehyde (assay 97%), 4-(dimethylamino)benzaldehyde (grade ACS reagent, assay 99%), benzo[*d*][1,3]dioxole-5-carbaldehyde (piperonal, assay 99%), furfural (assay 99%), 5-bromo-2-furaldehyde (assay 97%), 5-chloro-2-furaldehyde (assay 98%), 5-(4-chlorophenyl)furfural (assay 95%), 2-benzofurancarboxaldehyde (assay 97%), ethyl acetoacetate (grade ReagentPlus®, assay 99%), 4-bromobenzaldehyde (ReagentPlus®, assay 99%), 2-hydroxybenzaldehyde (for synthesis), and 2-chloroacetamide (assay 98%) were obtained from Sigma-Aldrich. 5,5-Dimethyl-1,3-cyclohexanedione (dimedone) (99%) was obtained from Across. 2-Naphthol (for synthesis), benzamide (for synthesis), zinc chloride ($ZnCl_2$) (for analysis EMSURE® ACS,ISO,Reag. Ph Eur), benzaldehyde (for synthesis), 4-methylbenzaldehyde (for synthesis), 4-methoxybenzaldehyde (for synthesis), 5-hydroxymethyl-2-furancarbaldehyde (5-(hydroxyme-thyl)furfural) (for synthesis), 4-nitrobenzaldehyde (for synthesis), butyraldehyde (for synthesis), ammonium acetate (EMPLURA®), TLC (silica gel 60 F_{254}), and silica gel 230–400 mesh (for column chromatography) were obtained from Merck. Ethyl acetate (purity ≥ 99.5%), *n*-hexane (purity ≥ 99.5%), and chloroform (purity ≥ 99%) were obtained from Xilong Chemical Co., Ltd (China).

Analytical techniques

The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Advance 500 instrument using $CDCl_3$ as solvent and solvent peaks or TMS as internal standards. HRMS (ESI) data were collected using Bruker micrOTOF-QII MS at 80 eV. FT-IR spectra were recorded in the form of KBr pellets by a Bruker Vertex 70. GC-MS analyses were performed on an Agilent GC system 7890 equipped with a mass selective detector Agilent 5973N and a capillary DB-5MS column (30m x 250 μm x 0.25 μm). Thermal gravimetric analysis (TGA) was obtained using a TA Q500 thermal

analysis system with the sample held in a platinum pan in a continuous airflow. Ultrasonic irradiation were performed on an Elma sonic S30H at the frequency of 37 kHz. Raman spectra were recorded on a Horiba Xplora One using a 532 nm argon-ion laser. ICP-MS was recorded on a PerkinElmer 350X. Scanning electron microscope (SEM) was performed on an S4800 Hitachi, Japan. The electron diffraction spectroscopy (EDS) was conducted on a Horiba H7593. ICP-OES was recorded on a PerkinElmer 350X.

Section S2. General procedure

Synthesis and characterization of [CholineCl][$ZnCl_2$]$_3$

General procedure of [CholineCl][$ZnCl_2$]$_3$[1-5]

[CholineCl][$ZnCl_2$]$_3$ was synthesized from choline chloride (5 mmol, 0.698 g) and zinc chloride (15 mmol, 2.044 g), which was heated until the colorless homogeneous liquid at 100 ^{o}C. The purity and structure of [CholineCl][$ZnCl_2$]$_3$ were determined by ^{1}H, ^{13}C NMR, FT-IR, Raman, and TGA.

Characterization of [CholineCl][$ZnCl_2$]$_3$

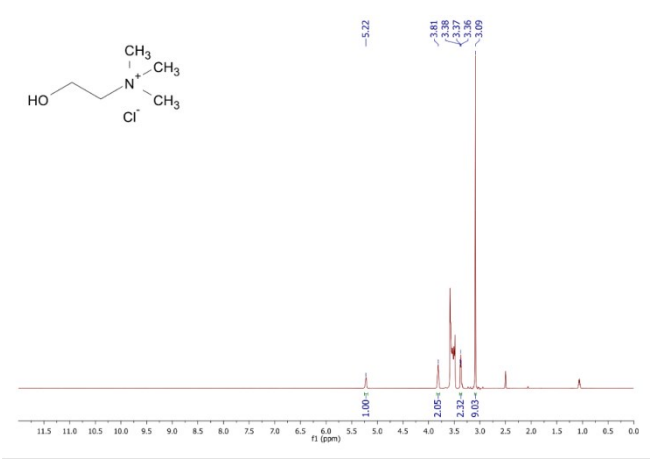


Fig. S1. ^{1}H NMR spectrum of [CholineCl][$ZnCl_2$]$_3$

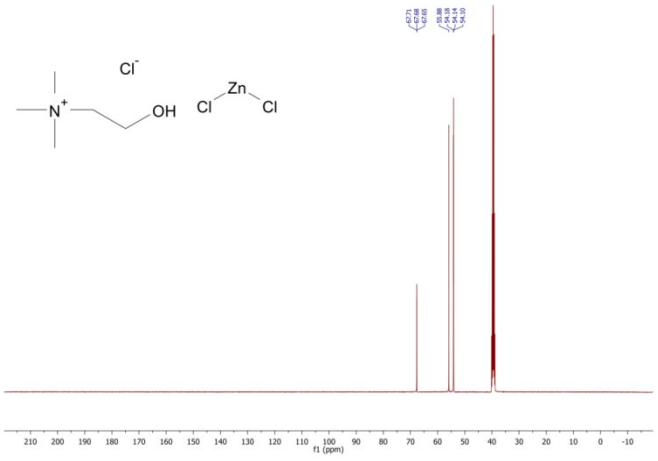


Fig. S2. ^{13}C NMR spectrum of [CholineCl][$ZnCl_2$]$_3$

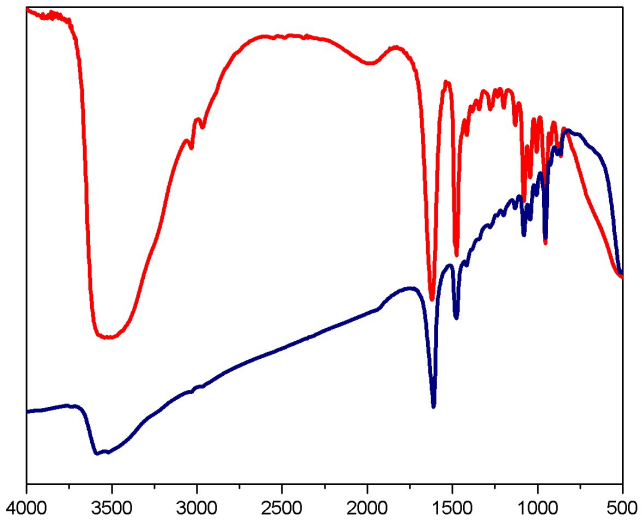


Fig. S3. FT-IR spectra of [CholineCl][$ZnCl_2$]$_3$ catalyst (red), and recycled [CholineCl][$ZnCl_2$]$_3$ after three cycles (blue)

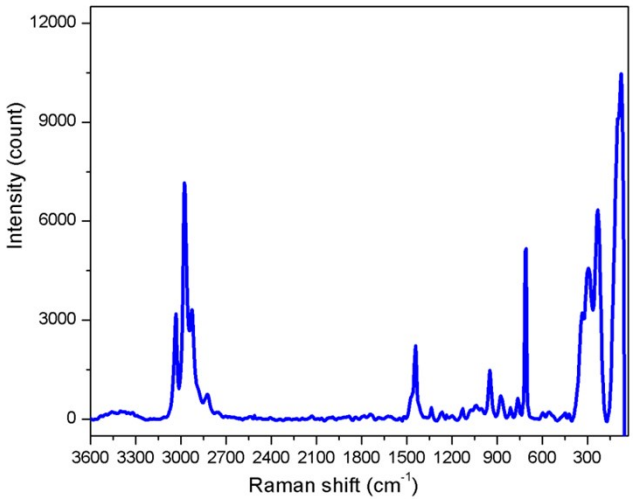


Fig. S4. The Raman spectra of [CholineCl][$ZnCl_2$]$_3$

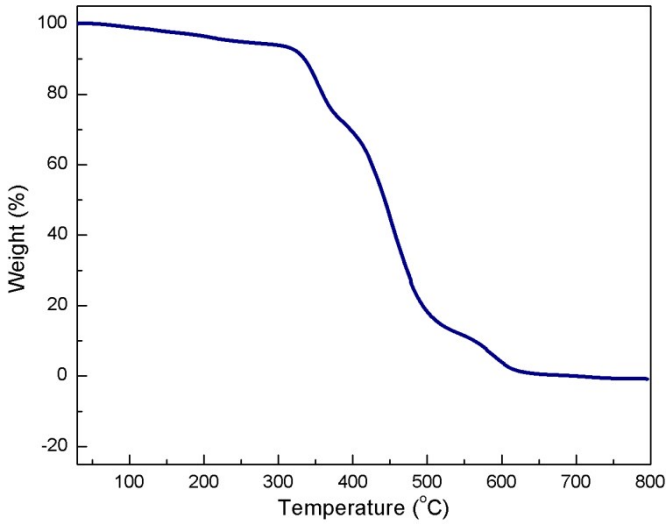


Fig. S5. TGA curve of the [CholineCl][$ZnCl_2$]$_3$

Recycling of [CholineCl][$ZnCl_2$]$_3$

After completion of the reaction, ethyl acetate was added to the reaction mixture as many times as necessary to extract both the starting materials and products completely. Then, [CholineCl][$ZnCl_2$]$_3$ was dried in a vacuum at 80 ˚C for 60 min. DES was used for three consecutive runs, and it is worth noting that the isolated yield of the product decreased slightly after each run.

Section S3. Optimization of the reaction condition

Table S1. Optimization of reaction condition in the synthesis of 1-amidoalkyl-2-naphthols.[a]

Entry	Temperature (°C)	Time (min)	[CholineCl][$ZnCl_2$]$_3$ (mol%)	Isolated yield %
1	RT	40	20	10
2	60	40	20	94
3	80	40	20	95
4	100	40	20	78
5	120	40	20	84
6	60	10	20	80
7	60	20	20	82
8	60	30	20	88
9	60	50	20	86
10	60	60	20	87
11	60	40	2	9
12	60	40	5	29
13	60	40	10	55
14	60	40	30	99
15	US[b]	40	20	No reaction

[a] Reaction conditions: 2-Naphthol (1.0 mmol), benzaldehyde (1.0 mmol), and benzamide (1.0 mmol).

[b] Ultrasound activation.

Table S2. Optimization of reaction condition in the synthesis of polyhydroquinolines

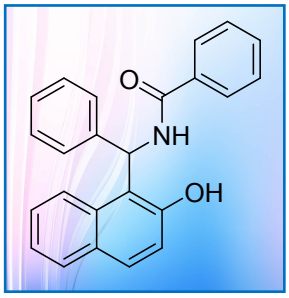
Entry	Temperature	Time (min)	[CholineCl][$ZnCl_2$]$_3$	Isolated

	(°C)		(mol%)	yield %
1	RT	20	0.5	36
2	60	20	0.5	38
3	80	20	0.5	42
4	100	20	0.5	87
5	120	20	0.5	68
6	100	5	0.5	33
7	100	10	0.5	46
8	100	15	0.5	41
9	100	30	0.5	48
10	100	40	0.5	47
11	100	20	0	26
12	100	20	1	67
13	100	20	2	58
14	100	20	5	73
15	100	20	10	53

Reaction conditions: Benzaldehyde (1.0 mmol), dimedone (1.0 mmol), ethyl acetoacetate (1.0 mmol), and ammonium acetate (1.0 mmol).

Section S4. Spectral data

N-((2-Hydroxynaphthalen-1-yl)(phenyl)methyl)benzamide[6-8] (1)



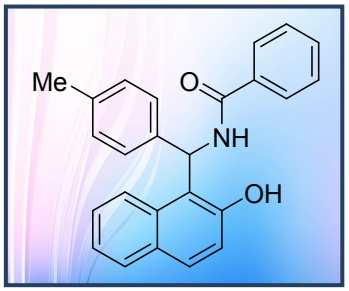
White solid, mp = 233 – 235 °C

IR (KBr, cm^{-1}): 3418, 3061, 3021, 1629, 1570, 1435, 821, 751, 613

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.34 (s, 1H), 9.02 (d, J = 8.5 Hz, 1H), 8.09 (d, J = 8.5 Hz, 1H), 7.89 – 7.78 (m, 4H), 7.55 (t, J = 7.5 Hz, 1H), 7.47 (dd, J = 15.0, 7.5 Hz, 3H), 7.34 – 7.23 (m, 7H), 7.22 – 7.17 (m, 1H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 166.2, 153.7, 142.5, 134.8, 132.8, 131.9, 129.9, 129.1, 129.0, 128.9, 128.7, 127.6, 127.2, 127.0, 126.9, 123.2, 119.2, 118.8, 49.7.

***N*-((2-hydroxynaphthalen-1-yl)(p-tolyl)methyl)benzamide[6-8] (2)**



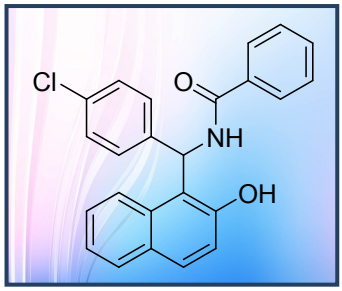
White solid, mp = 208 – 211 ^{o}C

IR (KBr, cm^{-1}): 3418, 3028, 2951, 1629, 1530, 1381, 1346, 870, 748, 687

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.33 (s, 1H), 9.03 (d, J = 8.5 Hz, 1H), 8.07 (d, J = 8.5 Hz, 1H), 7.84 (t, J = 7.5 Hz, 3H), 7.79 (d, J = 9.0 Hz, 1H), 7.54 (t, J = 7.5 Hz, 1H), 7.51 – 7.43 (m, 3H), 7.31 (t, J = 7.5 Hz, 1H), 7.23 (dd, J = 19.0, 8.5 Hz, 4H), 6.84 (d, J = 9.0 Hz, 2H), 3.68 (s, 3H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 166.0, 158.5, 153.5, 134.9, 134.4, 132.7, 131.9, 129.7, 129.1, 129.0, 128.9, 128.2, 127.5, 127.2, 123.1, 119.2, 118.9, 114.1, 55.5, 49.4.

***N*-((4-Chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide[6, 7, 9] (3)**



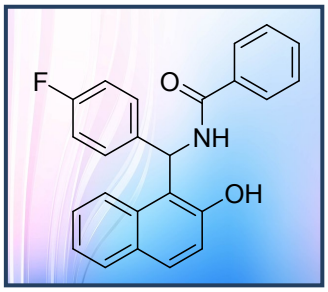
White solid, mp = 186 – 189 ^{o}C

IR (KBr, cm^{-1}): 3369, 3172, 2777, 1657, 1576, 1401, 769, 685, 633

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.35 (s, 1H), 9.02 (d, J = 8.0 Hz, 1H), 8.07 (d, J = 8.5 Hz, 1H), 7.90 – 7.80 (m, 4H), 7.56 (t, J = 7.5 Hz, 1H), 7.48 (dd, J = 13.5, 7.5 Hz, 3H), 7.34 (t, J = 7.5 Hz, 3H), 7.31 – 7.28 (m, 3H), 7.25 (d, J = 8.5 Hz, 1H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 166.4, 153.7, 141.6, 134.7, 132.7, 132.0, 131.6, 130.1, 129.1, 129.0, 128.9, 128.8, 128.6, 127.7, 127.3, 123.2, 119.1, 118.4, 49.2.

***N*-((4-Fluorophenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide[10] (4)**



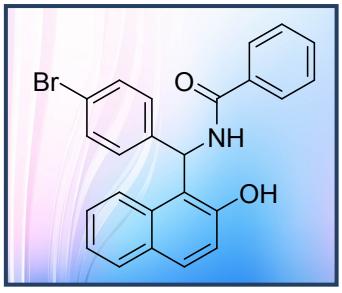
White solid, mp = 232 – 235 °C

IR (KBr, cm^{-1}): 3418, 3063, 2288, 1630, 1511, 1345, 1261, 1170, 825, 747, 712, 589.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.37 (s, 1H), 9.05 (d, J = 8.5 Hz, 1H), 8.08 (d, J = 8.5 Hz, 1H), 7.90 – 7.83 (m, 3H), 7.81 (d, J = 9.0 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.52 – 7.45 (m, 3H), 7.30 (td, J = 14.0, 7.5 Hz, 4H), 7.25 (d, J = 9.0 Hz, 1H), 7.11 (t, J = 9.0 Hz, 2H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 166.3, 162.4 (d, J = 245.7 Hz), 153.7, 138.6, 138.6, 134.7, 132.7, 131.9, 130.1, 130.0, 129.1, 129.0, 128.9 (d, J = 8.1 Hz), 127.7, 127.3, 123.2, 119.2, 118.6, 115.5 (d, J = 21.1 Hz), 49.3.

***N*-((4-Bromophenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide[6, 10, 11] (5)**



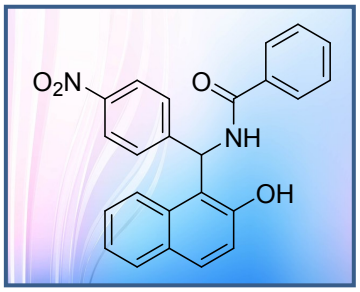
White solid, mp = 197 – 200 °C

IR (KBr, cm^{-1}): 3418, 3182, 1628, 1576, 1341, 810, 724, 585.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.36 (s, 1H), 9.02 (d, J = 8.5 Hz, 1H), 8.07 (d, J = 8.5 Hz, 1H), 7.89 – 7.84 (m, 3H), 7.82 (d, J = 9.0 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.53 – 7.44 (m, 5H), 7.33 (t, J = 7.5 Hz, 1H), 7.30 – 7.21 (m, 4H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 166.4, 153.7, 142.0, 134.7, 132.7, 132.0, 131.5, 130.1, 129.2, 129.1, 129.0, 128.9, 127.7, 127.3, 123.2, 120.1, 119.1, 118.3, 49.3.

***N*-((2-Hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl)benzamide[6-8] (6)**



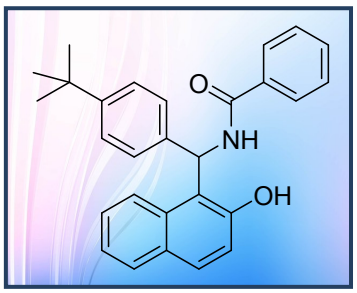
Yellowish solid, mp = 230 – 233 °C

IR (KBr, cm^{-1}): 3434, 3067, 2850, 1638, 1516, 1345, 851, 800, 736.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.40 (s, 1H), 9.08 (d, J = 8.0 Hz, 1H), 8.17 (d, J = 9.0 Hz, 2H), 8.08 (d, J = 8.5 Hz, 1H), 7.92 (d, J = 7.5 Hz, 2H), 7.85 (t, J = 9.0 Hz, 2H), 7.58 – 7.54 (m, 3H), 7.50 (t, J = 8.0 Hz, 3H),7.42 (d, J = 8.0 Hz, 1H), 7.33 (t, J = 7.5 Hz, 1H), 7.27 (d, J = 9.0 Hz, 1H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 166.8, 153.9, 150.7, 146.7, 134.4, 132.7, 132.1, 130.5, 129.2, 128.9, 128.9, 128.0, 127.9, 127.5, 123.9, 123.3, 123.1, 119.0, 117.9, 49.5.

***N*-((4-(*tert*-Butyl)phenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide[12] (7)**



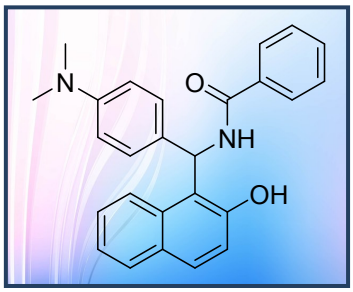
White solid, mp = 207 – 209 °C

IR (KBr, cm^{-1}): 3420, 3061, 2961, 1630, 1535, 1437, 875, 754, 689.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.33 (s, 1H), 9.04 (d, J = 8.5 Hz, 1H), 8.11 (d, J = 8.5 Hz, 1H), 7.90 – 7.82 (m, 3H), 7.79 (d, J = 9.0 Hz, 1H), 7.54 (t, J = 7.5 Hz, 1H), 7.50 – 7.42 (m, 3H), 7.35 – 7.19 (m, 7H), 1.22 (s, 9H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 166.0, 153.6, 149.4, 139.4, 134.9, 132.8, 131.9, 129.7, 129.1, 129.0, 128.8, 128.7, 127.9, 127.6, 127.2, 126.8, 125.5, 123.2, 119.2, 118.9, 49.6, 34.6, 31.6.

***N*-((4-(Dimethylamino)phenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide[7, 10, 13] (8)**



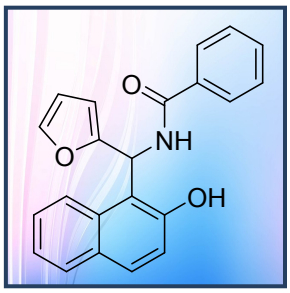
White solid, mp = 219 – 222 ^{o}C

IR (KBr, cm^{-1}): 3413, 3071, 2803, 1630, 1533, 1346, 1329, 1166, 813, 754, 686, 567.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.32 (s, 1H), 9.03 (d, J = 8.5 Hz, 1H), 8.09 (d, J = 9.0 Hz, 1H), 7.92 – 7.77 (m, 3H), 7.79 (d, J = 9.0 Hz, 1H), 7.56 (t, J = 7.5 Hz, 1H), 7.52 – 7.42 (m, 3H), 7.32 (t, J = 7.5 Hz, 1H), 7.26 (d, J = 9.0 Hz, 1H), 7.21 (d, J = 8.5 Hz, 1H), 7.13 (d, J = 8.5 Hz, 2H), 6.65 (d, J = 8.5 Hz, 2H), 2.82 (s, 6H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 165.8, 153.5, 149.9, 135.0, 132.7, 131.8, 129.9, 129.5, 129.0, 128.8, 127.9, 127.4, 127.1, 123.1, 119.3, 119.2, 112.9, 112.8, 49.6, 40.7.

***N*-(Furan-2-yl(2-hydroxynaphthalen-1-yl)methyl)benzamide[14] (9)**



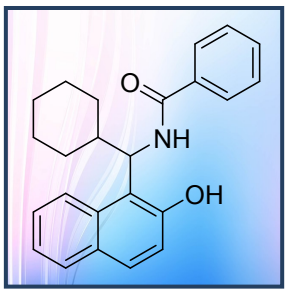
Brown solid, mp = 220 – 223 °C

IR (KBr, cm^{-1}): 3409, 2197, 1632, 1572, 1437, 816, 745, 597.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.21 (s, 1H), 9.09 (d, J = 8.0 Hz, 1H), 8.18 (d, J = 8.5 Hz, 1H), 7.89 – 7.77 (m, 4H), 7.59 – 7.52 (m, 2H), 7.47 (t, J = 7.5 Hz, 3H), 7.36 – 7.27 (m, 2H), 7.23 (d, J = 9.0 Hz, 1H), 6.36 (dd, J = 3.0, 2.0 Hz, 1H), 6.13 (d, J = 3.0 Hz, 1H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 166.1, 154.5, 153.9, 142.5, 142.5, 134.7, 132.7, 131.9, 130.0, 129.0, 128.9, 128.8, 128.7, 127.7, 127.0, 123.4, 123.0, 119.0, 116.8, 110.9, 110.9, 107.2, 107.2, 45.0.

***N*-(Cyclohexyl(2-hydroxynaphthalen-1-yl)methyl)benzamide (10)**



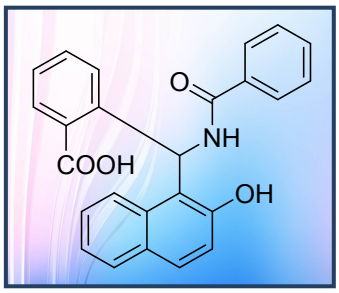
White solid, mp = 245 – 248 °C

IR (KBr, cm^{-1}): 3408, 3071, 2934, 1634, 1574, 1336, 858, 819, 695.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.23 (s, 1H), 8.46 (s, 1H), 8.19 (s, 1H), 7.79 (d, J = 7.0 Hz, 3H), 7.71 (d, J = 8.0 Hz, 1H), 7.55 – 7.41 (m, 4H), 7.32 – 7.26 (m, 1H), 7.19 (d, J = 8.5 Hz, 1H), 5.78 (s, 1H), 2.27 (s, 1H), 2.09 (s, 1H), 1.75 (d, J = 11.0 Hz, 1H), 1.56 (dd, J = 24.0, 11.0 Hz, 2H), 1.31 – 1.08 (m, 4H), 1.07 – 0.92 (m, 2H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 165.9, 153.4, 135.3, 131.6, 128.9, 127.3, 126.8, 123.2, 122.9, 119.6, 119.1, 55.6 , 53.6, 52.3, 41.3, 30.7, 26.4, 26.2, 26.1.

2-(Benzamido(2-hydroxynaphthalen-1-yl)methyl)benzoic acid (11)



White solid, mp = 236 – 238 °C

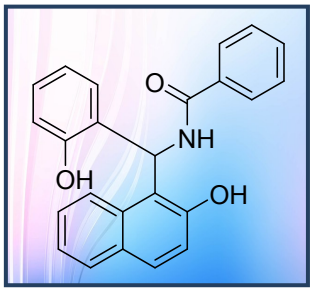
IR (KBr, cm^{-1}): 3227, 1716, 1628, 1513, 1302, 935, 818, 743, 711

^{1}H NMR (500 MHz, DMSO-d_6) δ 12.82 (s, 1H), 9.82 (s, 1H), 8.85 (d, J = 7.5 Hz, 1H), 8.14 (d, J = 8.5 Hz, 1H), 7.89 (d, J = 8.0 Hz, 1H), 7.85 (d, J = 7.5 Hz, 2H), 7.80 (d, J = 8.0 Hz, 1H), 7.75 (d, J = 9.0 Hz, 1H), 7.67 (dd, J = 7.5, 1.0 Hz, 1H), 7.50 (t, J = 7.5 Hz, 1H), 7.46 – 7.31 (m, 5H), 7.28 (t, J = 7.5 Hz, 2H), 7.15 (d, J = 8.5 Hz, 1H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 165.7, 154.2, 141.2, 134.9, 133.7, 131.5, 130.8, 129.7, 129.6, 130.0, 128.8, 128.8, 128.6, 127.9, 127.2, 126.7, 123.7, 122.8, 119.4, 118.5, 48.7, 31.2.

HRMS (ESI) *m/z* Cacld. $[M-H]^+$ $C_{25}H_{18}NO_4^+$ 396.1236; found 396.1240.

***N*-((2-Hydroxynaphthalen-1-yl)(2-hydroxyphenyl)methyl)benzamide[6, 10, 13] (12)**



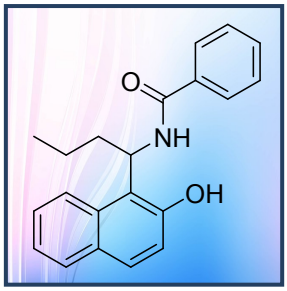
White solid, mp = 234 – 237 °C

IR (KBr, cm^{-1}): 3592, 3205, 2363, 1607, 1565, 1349, 816, 752, 687, 588.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.18 (s, 1H), 9.64 (s, 1H), 8.92 (s, 1H), 8.24 (d, J = 9.0 Hz, 1H), 7.84 (d, J = 7.0 Hz, 2H), 7.79 (d, J = 8.0 Hz, 1H), 7.73 (d, J = 9.0 Hz, 1H), 7.53 (t, J = 7.0 Hz, 1H), 7.50 – 7.37 (m, 4H), 7.34 (d, J = 8.0 Hz, 1H), 7.28 (t, J = 7.0 Hz, 1H), 7.20 (d, J = 9.0 Hz, 1H), 7.05 (t, J = 7.0 Hz, 1H), 6.80 (d, J = 7.5 Hz, 1H), 6.68 (t, J = 7.0 Hz, 1H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 165.6, 155.3, 153.6, 135.0, 133.0, 131.7, 129.5, 129.2, 128.9, 128.8, 128.5, 128.2, 127.6, 126.6, 123.8, 123.3, 122.9, 119.3, 119.3, 119.1, 115.8, 46.2.

***N*-(1-(2-Hydroxynaphthalen-1-yl)butyl)benzamide[15] (13)**



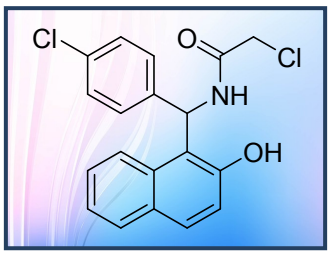
White solid, mp = 220 – 223 °C

IR (KBr, cm^{-1}): 3415, 3218, 30068, 2958, 2936, 2874, 1633, 1528, 1341, 815, 779, 690.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.10 (s, 1H), 8.61 (d, J = 6.5 Hz, 1H), 8.22 (d, J = 6.5 Hz, 1H), 7.85 – 7.77 (m, 3H), 7.70 (d, J = 8.5 Hz, 1H), 7.55 – 7.44 (m, 4H), 7.30 (t, J = 7.5 Hz, 1H), 7.19 (d, J = 8.5 Hz, 1H), 6.01 (q, J = 8.5 Hz, 1H), 2.21 – 2.11 (m, 1H), 1.93 – 1.83 (m, 1H), 1.54 – 1.42 (m, 1H), 1.35 – 1.22 (m, 1H), 0.92 (t, J = 7.5 Hz, 3H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 165.8, 153.4, 135.2, 132.6, 131.6, 129.0, 128.9, 128.9, 127.4, 126.8, 122.9, 120.4, 119.1, 47.1, 36.6, 20.1, 14.3.

2-Chloro-*N*-((4-chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)acetamide[16] (14)



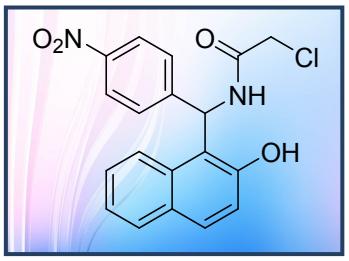
White solid, mp = 200 – 203 °C

IR (KBr, cm^{-1}): 3350, 3070, 2914, 1625, 1531, 1417, 1010, 858, 807, 774.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.28 (s, 1H), 8.96 (d, J = 8.5 Hz, 1H), 8.00 – 7.71 (m, 3H), 7.44 (m, J = 7.0 Hz 1H), 7.37 – 7.27 (m, 3H), 7.24 (d, J = 8.5 Hz, 1H), 7.20 (d, J = 8.5 Hz, 2H), 7.08 (d, J = 8.5 Hz, 1H), 4.33 (dd, J = 13.5, 10.5 Hz, 2H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 166.34, 153.71, 141.46, 132.54, 131.51, 130.27, 129.18, 128.58, 128.34, 123.16, 118.9, 118.0, 48.5, 43.3.

2-Chloro-*N*-((2-hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl)acetamide[17] (15)



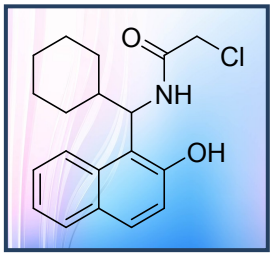
White solid, mp = 217 – 219 °C

IR (KBr, cm^{-1}): 3386, 3247, 1646, 1514, 1344, 948, 872, 784.

^{1}H NMR (500 MHz, DMSO-d_6) δ 9.01 (d, J = 7.5 Hz, 1H), 8.15 (d, J = 9.0 Hz, 2H), 7.84 (t, J = 7.0 Hz, 3H), 7.43 (d, J = 8.0 Hz, 3H), 7.31 (t, J = 7.0 Hz, 1H), 7.23 (d, J = 9.0 Hz, 1H), 7.16 (d, J = 8.0 Hz, 1H), 4.34 (q, J = 13.5 Hz, 2H), 3.83 (s, 1H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 168.4, 166.7, 153.8, 150.6, 146.6, 132.5, 130.7, 129.3, 128.8, 127.6, 123.9, 123.3, 118.8, 117.5, 48.9, 43.2.

2-Chloro-*N*-(cyclohexyl(2-hydroxynaphthalen-1-yl)methyl)acetamide (16)



White solid, mp = 216 – 218 °C

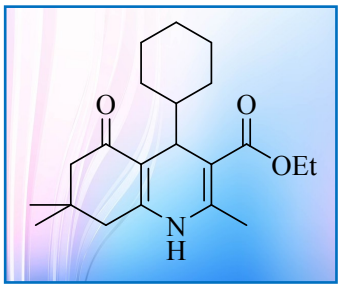
IR (KBr, cm^{-1}): 3278, 3070, 2934, 1647, 1581, 1472, 1143, 864, 811, 747.

^{1}H NMR (500 MHz, DMSO-d_6) δ 10.20 (s, 1H), 8.48 (d, J = 8.5 Hz, 1H), 8.11 (d, J = 8.5 Hz, 1H), 7.78 (d, J = 8.0 Hz, 1H), 7.72 (d, J = 8.5 Hz, 1H), 7.47 (t, J = 7.5 Hz, 1H), 7.28 (t, J = 7.5 Hz, 1H), 7.23 (d, J = 9.0 Hz, 1H), 5.63 (t, J = 9.5 Hz, 1H), 4.22 (d, J = 12.0 Hz, 2H), 2.19 (d, J = 9.5 Hz, 1H), 2.01 (d, J = 12.0 Hz, 1H), 1.75 (d, J = 11.0 Hz, 1H), 1.56 (d, J = 11.0 Hz, 1H), 1.49 (d, J = 11.0 Hz, 1H), 1.25 – 1.05 (m, 4H), 1.04 – 0.86 (m, 2H).

^{13}C NMR (125 MHz, DMSO-d_6) δ 165.5, 153.5, 133.4, 129.1, 129.0, 128.5, 126.9, 122.9, 118.9, 118.8, 51.8, 51.7, 43.5, 43.5, 43.4, 41.2, 30.7, 30.5, 26.4, 26.2, 26.1.

HRMS (ESI) *m/z* Cacld. $[M-H]^+$ $C_{19}H_{21}ClNO_2^+$ 330.1261; found 330.1262.

Ethyl 4-cyclohexyl-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydro-quinolin-3-carboxylate[18-20] (17)



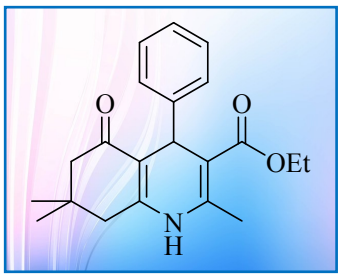
White solid, mp = 175 – 178 °C

IR (KBr, cm^{-1}): 3282, 2959, 1698, 1487, 1215.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 5.78 (br, 1H), 4.21–4.10 (m, 2H), 4.00–3.99 (d, J = 4.5 Hz, 1H), 2.31 (s, 3H), 2.31–2.25 (m, 4H), 1.63–1.55 (m, 8H), 1.30–1.27 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.12 (s, 3H), 1.09 (s, 3H), 0.93–0.87 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 197.1, 165.7, 164.6, 164.2, 137.3, 125.0, 124.7, 61.5, 51.9, 46.3, 32.9, 29.7, 28.3, 25.0, 14.3.

Ethyl 2,7,7-trimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate[21-23] (18)



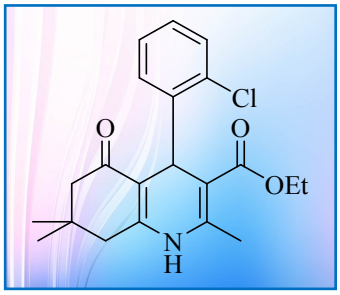
Pale yellow solid, mp = 201 – 203 °C

IR (KBr, cm^{-1}): 3285, 3082, 2954, 1697, 1609

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.30–7.29 (d, J = 7.5 Hz, 2H), 7.20–7.17 (t, J = 7.5 Hz, 2H), 7.10 – 7.07 (t, J = 7.5 Hz, 1H), 6.08 (s, 1H), 5.05 (s, 1H), 4.07–4.03 (m, 2H), 2.36 (s, 3H), 2.33–2.30 (m, 1H), 2.24–2.16 (m, 3H), 1.20–1.17 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.07 (s, 3H), 0.93 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.5, 167.5, 148.3, 147.0, 143.5, 128.0, 127.9, 126.0, 112.2, 106.1, 59.8, 50.8, 41.1, 36.6, 32.7, 29.4, 27.2, 19.3, 14.2.

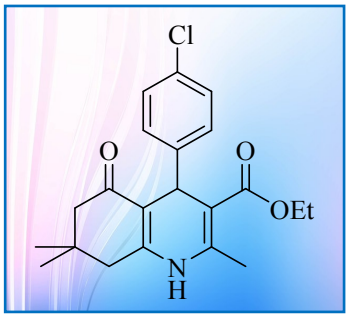
Ethyl 4-(2-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahyydroquinolin-3-carboxylate[21, 24] (19)



White solid, mp = 209 – 211 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.39–7.38 (d, J = 8.0 Hz, 1H), 7.24–7.22 (d, J = 8.0 Hz, 1H), 7.13–7.10 (m, 1H), 7.03–7.00 (m, 1H), 6.58 (br, 1H), 5.38 (s, 1H), 4.07–4.00 (m, 2H), 2.26 (s, 3H), 2.21–2.12 (m, 3H), 1.18–1.15 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.05 (s, 3H), 0.93 (s, 3H).

Ethyl 4-(4-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate[24, 25] (20)



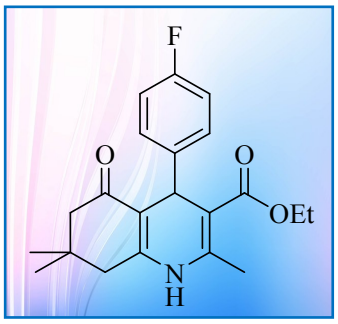
Pale yellow solid, mp = 246 – 248 °C

IR (KBr, cm^{-1}): 3273, 3190, 2958, 1703, 1602, 1485, 1211.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.24–7.22 (d, J = 7.0 Hz, 2H), 7.16–7.15 (d, J = 4.5 Hz, 2H), 6.20 (br, 1H), 5.02 (s, 1H), 4.07–4.03 (m, 2H), 2.36 (s, 3H), 2.33–2.16 (m, 4H), 1.20–1.17 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.06 (s, 3H), 0.92 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.6, 167.2, 148.4, 145.6, 143.7, 131.6, 129.4, 128.0, 111.8, 105.7, 59.9, 50.7, 41.0, 36.2, 32.7, 29.4, 28.1, 27.1, 19.4, 14.2, 13.7.

Ethyl 4-(4-fluorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate[26] (21)



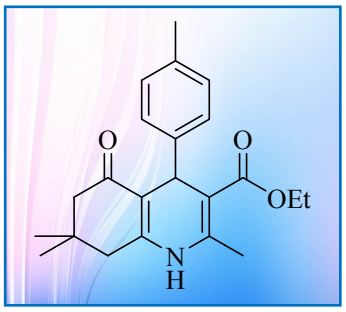
Pale yellow solid, mp = 184 – 187 °C

IR (KBr, cm^{-1}): 3273, 3194, 2957, 1705, 1604, 1488, 1211.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.30–7.27 (q, J = 5.2 Hz, 3H), 6.91–6.88 (t, J = 8.8 Hz, 2H), 5.82 (s, 1H), 5.05 (s, 1H), 4.12–4.06 (m, 2H), 2.41 (s, 3H), 2.38–2.35 (m, 1H), 2.24–2.19 (m, 3H), 1.24–1.21 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.10 (s, 3H), 0.95 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.4, 167.3, 161.3 (d, J = 241.9 Hz), 147.6, 143.3, 142.8, 129.5 (d, J = 7.9 Hz), 114.7 (d, J = 21.4 Hz), 114.6, 114.5, 112.3, 106.1, 59.9, 50.7, 41.2, 36.0, 32.7, 29.4, 28.1, 27.1, 19.5, 14.2, 13.7.

Ethyl 2,7,7-trimethyl-5-oxo-4-(*p*-tolyl)-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate[25] (22)



Pale yellow solid, mp = 261 – 263 °C

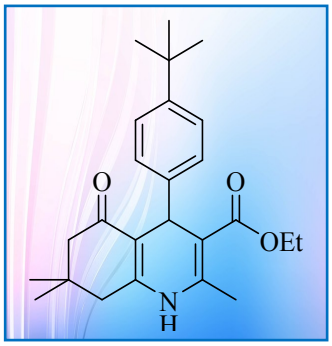
IR (KBr, cm^{-1}): 3273, 3196, 2958, 1699, 1602, 1490, 1211.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.19–7.17 (d, J = 8.0 Hz, 2H), 7.00–6.99 (d, J = 8.0 Hz, 2H), 5.73 (br, 1H), 5.01 (s, 1H), 4.08–4.04 (m, 2H), 2.36 (s, 3H), 2.35–2.31 (m,

1H), 2.25 (s, 3H), 2.24–2.14 (m, 3H), 1.22–1.19 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.07 (s, 3H), 0.95 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.4, 167.5, 147.6, 144.1, 143.1, 135.4, 133.4, 128.6, 127.9, 112.5, 106.4, 98.0, 66.6, 59.8, 50.7, 41.3, 36.1, 32.8, 29.4, 28.2, 27.3, 21.1, 19.5, 14.2.

Ethyl 4-(4-(*tert*-butyl)phenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate[21] (23)

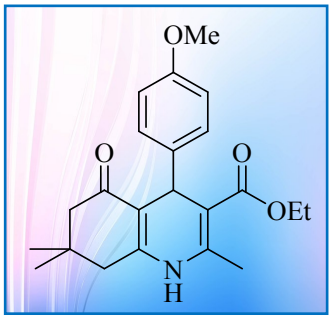


White solid, mp = 210 – 211 °C

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.23–7.19 (m, 4H), 7.03 (s, 1H), 5.04 (s, 1H), 4.10–4.07 (m, 2H), 2.34–2.30 (m, 2H), 2.28 (s, 3H), 2.23–2.19 (m, 3H), 1.25 (s, 9H), 1.24–1.21 (t, J = 7.5 Hz, 14.5 Hz, 3H), 1.08 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 196.0, 167.7, 149.4, 148.5, 144.1, 143.7, 127.5, 124.8, 111.8, 106.1, 59.8, 50.8, 40.9, 35.9, 34.3, 32.7, 31.4, 31.3, 29.4, 27.4, 19.1, 14.2.

Ethyl 4-(4-methoxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate[25] (24)



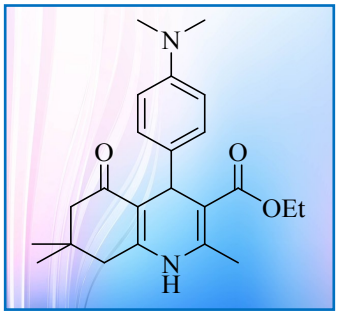
Pale yellow, mp = 255 – 257 °C

IR (KBr, cm^{-1}): 3275, 3076, 2956, 1699, 1602, 1490, 1211.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.22–7.20 (d, J = 9.0 Hz, 2H), 6.74–6.72 (d, J = 8.5 Hz, 2H), 5.71 (br, 1H), 4.99 (s, 1H), 4.08–4.04 (m, 2H), 3.74 (s, 3H), 2.37 (s, 3H), 2.35–2.32 (m, 1H), 2.22–2.14 (m, 3H), 1.21–1.19 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.08 (s, 3H), 0.94 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.5, 167.5, 157.8, 147.4, 139.5, 129.0, 113.3, 112.6, 106.4, 105.8, 59.8, 55.1, 50.7, 41.3, 35.7, 32.7, 29.4, 27.2, 19.5, 14.2.

Ethyl 4-(4-dimethylamino)phenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexayidroquinolin-3-carboxylate[27] (25)



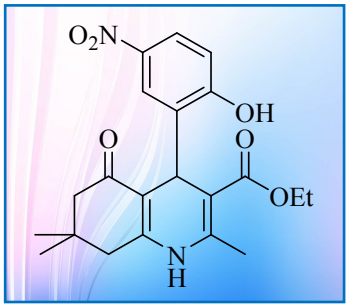
Pale yellow solid, mp = 225 – 227 ^{o}C

IR (KBr, cm^{-1}): 3283, 3078, 2956, 1700, 1607, 1488, 1380, 1279.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.20–7.18 (d, J = 8.5 Hz, 2H), 6.65 (s, 2H), 5.91 (brs, 1H), 4.98 (s, 1H), 4.11–4.07 (m, 2H), 2.90 (s, 6H), 2.38 (s, 3H), 2.24–2.22 (m, 1H), 2.22–2.19 (m, 3H), 1.25–1.23 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.09 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.5, 167.6, 147.5, 142.8, 128.7, 112.6, 106.5, 59.8, 50.8, 41.2, 35.4, 32.7, 29.4, 27.4, 19.5, 14.2.

Ethyl 4-(2-hydroxy-5-nitrophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate[21] (26)



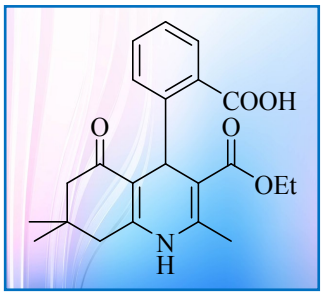
Pale yellow solid, mp = 210 – 213 °C

IR (KBr, cm^{-1}): 3294, 3086, 2958, 1699, 1589, 1483, 1332, 1220, 729.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 10.42 (s, 1H), 7.99–7.97 (dd, J = 3.0 Hz, 3.0 Hz, 1H), 7.84 (d, J = 2.5 Hz, 1H), 6.96–6.95 (d, J = 9.0 Hz, 1H), 6.14 (br, 1H), 5.10 (s, 1H), 4.01–3.97 (m, 2H), 2.54 (s, 3H), 2.38–2.34 (m, 4H), 1.11 (s, 3H), 1.06–1.03 (t, J = 7.0 Hz, 14.0 Hz, 3H), 0.92 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 198.5, 160.2, 151.2, 136.3, 134.5, 124.7, 124.3, 118.5, 110.7, 60.3, 55.1, 49.8, 49.2, 41.4, 32.8, 30.3, 29.0, 27.7, 27.3, 21.8, 19.5, 15.8, 13.9, 9.6.

Ethyl 4-(2-carboxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (27)



White solid, mp = 257 – 259 °C

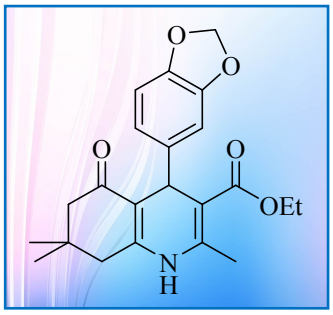
IR (KBr, cm^{-1}): 3292, 2956, 2873, 1699, 1575, 1479, 1390, 1220, 731.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 14.28 (s, 1H), 7.58–7.57 (d, J = 8.0 Hz, 1H), 7.39–7.36 (t, J = 8.0 Hz, 1H), 7.32–7.31 (d, J = 7.5 Hz, 1H), 7.23–7.20 (t, J = 7.5 Hz, 1H), 6.87 (br, 1H), 5.32 (s, 1H), 4.11–4.02 (m, 2H), 2.48–2.40 (m, 2H), 2.39 (s, 3H), 2.30–2.20 (m, 1H), 2.20–2.17 (m, 1H), 1.13–1.10 (q, J = 7.0 Hz, 14.0 Hz, 6H), 0.98 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 198.7, 171.1, 166.5, 152.6, 144.7, 142.7, 132.8, 131.5, 129.3, 129.0, 126.8, 111.3, 108.1, 60.4, 49.8, 41.1, 34.7, 33.0, 29.1, 27.1, 19.1, 14.2.

HR-ESI-MS *m/z*: found $[M+H]^+$ $C_{22}H_{26}NO_5^+$ 384.1806, calcd for 384.1810

Ethyl 4-(benzo[*d*][1,3]dioxol-5-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate[28] (28)



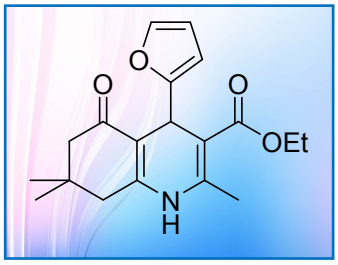
Pale yellow solid, mp = 225 – 228 °C

IR (KBr, cm^{-1}): 3278, 3080, 2954, 1485, 1685, 1598, 1379, 1209.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 6.79–6.76 (m, 2H), 6.64–6.63 (d, J = 8.0 Hz, 1H), 6.25 (br, 1H), 5.86–5.85 (d, J = 6.5 Hz, 2H), 4.97 (s, 1H), 4.09–4.05 (m, 2H), 2.34 (s, 3H), 2.32–2.29 (m, 1H), 2.22–2.18 (m, 3H), 1.23–1.20 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.06 (s, 3H), 0.95 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.7, 167.5, 148.3, 148.2, 147.2, 145.6, 143.3, 141.4, 121.1, 112.1, 108.7, 107.7, 106.2, 100.6, 59.9, 50.8, 41.0, 36.3, 32.7, 29.4, 27.3, 19.3, 14.3.

Ethyl 4-(furan-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate[29] (29)



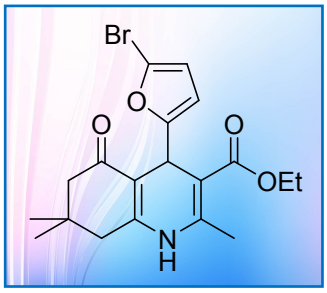
White solid, mp = 246 – 248 °C

IR (KBr, cm^{-1}): 3280, 3080, 2960, 1672, 2604, 1487, 1215.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.18 (s, 1H), 6.20–6.19 (dd, J = 1.5 Hz, 3.0 Hz, 1H), 6.01–6.00 (d, J = 3.0 Hz, 1H), 5.25 (s, 1H), 4.18–4.10 (m, 2H), 2.36 (s, 3H), 2.25–2.24 (d, J = 7.5 Hz, 2H), 1.25 (s, 6H), 1.09 (s, 3H), 1.01 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.3, 167.2, 157.9, 148.9, 144.3, 140.7, 110.2, 109.0, 104.8, 103.1, 59.9, 50.7, 41.3, 32.7, 30.2, 29.7, 29.5, 26.9, 22.7, 19.5, 14.3.

Ethyl 4-(5-bromofuran-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (30)



White solid, mp = 215 – 218 °C

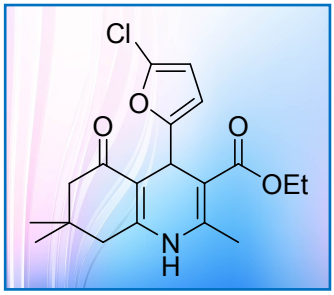
IR (KBr, cm^{-1}): 3277, 3078, 1678, 1604, 1485, 1211.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 6.23 (br, 1H), 6.11–6.10 (d, J = 3.5 Hz, 1H), 6.00 (d, J = 30 Hz, 1H), 5.20 (s, 1H), 4.21–4.10 (m, 2H), 2.35 (s, 3H), 2.33–2.22 (m, 4H), 1.28–1.26 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.10 (s, 3H), 1.05 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.4, 167.1, 159.9, 149.7, 144.7, 118.9, 112.0, 108.1, 107.7, 102.6, 60.0, 50.7, 41.1, 32.8, 30.6, 29.6, 26.8, 19.5, 14.3.

HR-ESI-MS *m/z*: calcd for $[M+H]^+$ $C_{19}H_{23}BrNO_4^+$: 408.0810, found: 408.0631

Ethyl 4-(5-chlorofuran-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (31)



White solid, mp = 195 – 198 °C

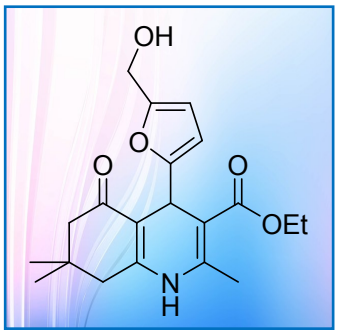
IR (KBr, cm^{-1}): 3277, 3076, 1677, 1602, 1485, 1215.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 6.67 (br, 1H), 6.01–6.00 (d, J = 3.0 Hz, 1H), 5.96 (d, J = 3.5 Hz, 1H), 5.18 (s, 1H), 4.21–4.12 (m, 2H), 2.34 (s, 3H), 2.27–2.24 (m, 4H), 1.28–1.26 (t, J = 7.0 Hz, 14.5 Hz, 3H), 1.10 (s, 3H), 1.05 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.6, 167.1, 157.5, 150.1, 144.9, 133.6, 107.9, 107.1, 106.8, 102.4, 60.0, 50.7, 41.0, 32.8, 30.6, 29.6, 26.8, 19.4, 14.3.

HR-ESI-MS *m/z*: calcd for $[M]^+$ $C_{19}H_{23}ClNO_4^+$: 363.1237, found: 363.1193

Ethyl 4-(5-(hydroxymethyl)furan-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (32)



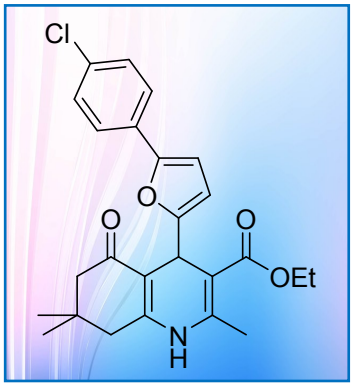
White solid, mp = 186 – 189 ^{o}C

IR (KBr, cm^{-1}): 3800, 3273, 3070, 2954, 1681, 1602, 1481, 1375, 1207.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 6.15 (br, 1H), 6.11 – 6.10 (d, J = 3.0 Hz, 1H), 5.95 – 5.94 (d, J = 3.0 Hz, 1H), 5.21 (s, 1H), 4.45 (s, 2H), 4.19–4.12 (m, 2H), 2.33 (s, 3H), 2.26 – 2.21 (m, 4H), 1.28 – 1.25 (t, J = 7.5 Hz, 14.5 Hz, 4H), 1.10 (s, 3H), 1.03 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.6, 167.3, 158.2, 152.1, 149.4, 144.4, 136.1, 108.7, 105.6, 103.0, 60.0, 57.6, 50.7, 41.2, 32.8, 30.4, 29.6, 26.8, 19.4, 14.3.

Ethyl 4-(5-(4-chlorophenyl)furan-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (33)



Pale yellow, mp = 185 – 188 °C

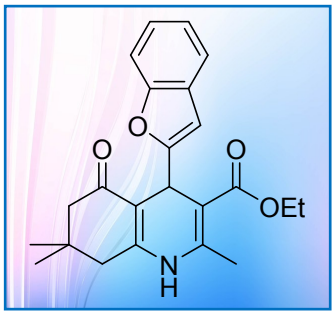
IR (KBr, cm^{-1}): 3278, 3078, 1693, 1602, 1479, 1211.

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.44–7.42 (d, J = 8.5 Hz, 2H), 7.26–7.24 (t, J = 6.0 Hz, 3H), 6.47–6.46 (d, J = 3.5 Hz, 1H), 6.07 (d, J = 3.0 Hz, 1H), 6.02 (br, 1H), 5.28 (s, 1H), 4.18–4.11 (m, 2H), 2.37 (s, 3H), 2.27–2.22 (m, 4H), 1.28–1.25 (t, J = 7.0 Hz, 14.0 Hz, 3H), 1.10 (s, 3H), 1.02 (s, 3H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.3, 167.2, 158.1, 150.9, 149.3, 144.5, 132.1, 129.8, 128.7, 124.4, 108.7, 107.1, 106.5, 103.0, 60.0, 50.7, 41.3, 32.8, 30.4, 29.7, 29.6, 26.8, 19.5, 14.4.

HR-ESI-MS *m/z*: calcd for $[M-H]^-$ $C_{25}H_{25}ClNO_4^-$: 438.1472, found: 438.1474.

Ethyl 4-(benzofuran-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (34)



White solid, mp = 205 – 208 °C

IR (KBr, cm^{-1}): 3280, 3084, 2953, 1699, 1602, 1489, 1379, 1209, 723

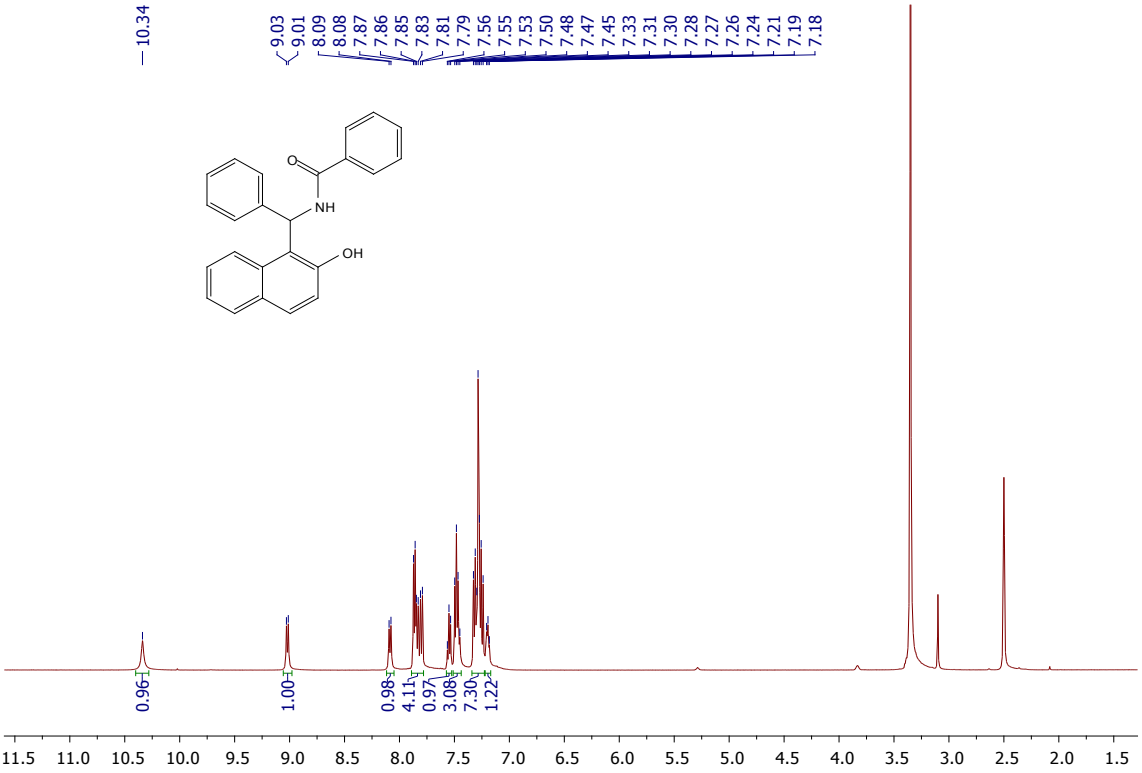
^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.42–7.41 (d, J = 6.5 Hz, 1H), 7.32–7.31 (d, J = 7.5 Hz, 1H), 7.15–7.09 (m, 2H), 6.41 (s, 1H), 6.37 (br, 1H), 5.40 (s, 1H), 4.18– 4.11 (m, 2H), 2.38 (s, 3H), 2.36–2.22 (m, 4H), 1.27–1.25 (t, J = 7.5 Hz, 14.5 Hz, 3H), 1.07 (s, 3H), 1.00 (s, 3H).

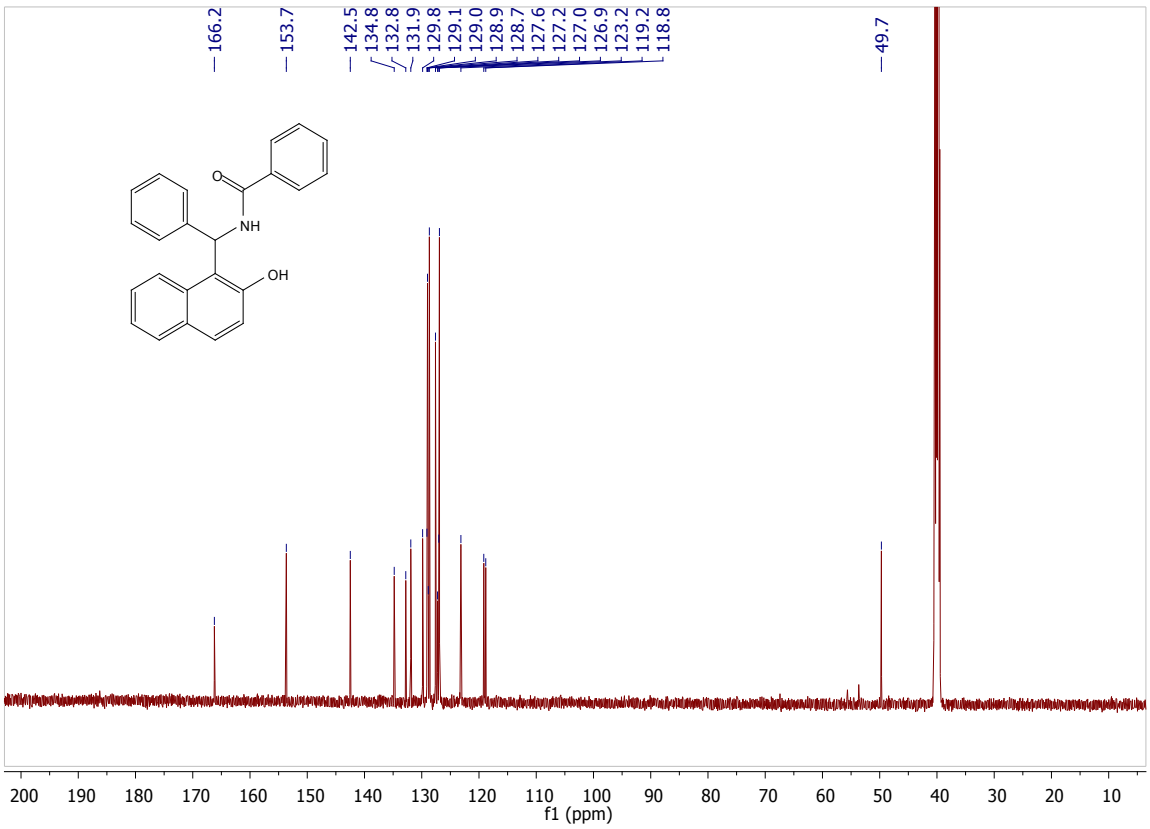
^{13}C NMR (125 MHz, $CDCl_3$) δ_C 195.5, 167.1, 160.7, 154.6, 149.8, 145.1, 129.0, 123.0, 122.2, 120.6, 110.9, 108.2, 102.3, 101.8, 60.0, 50.7, 41.1, 32.7, 30.9, 29.5, 27.0, 19.5, 14.3.

HS-ESI-MS *m/z*: calcd for $[M]^+$ $C_{23}H_{25}NO_4^+$: 379.1784, found: 379.1741.

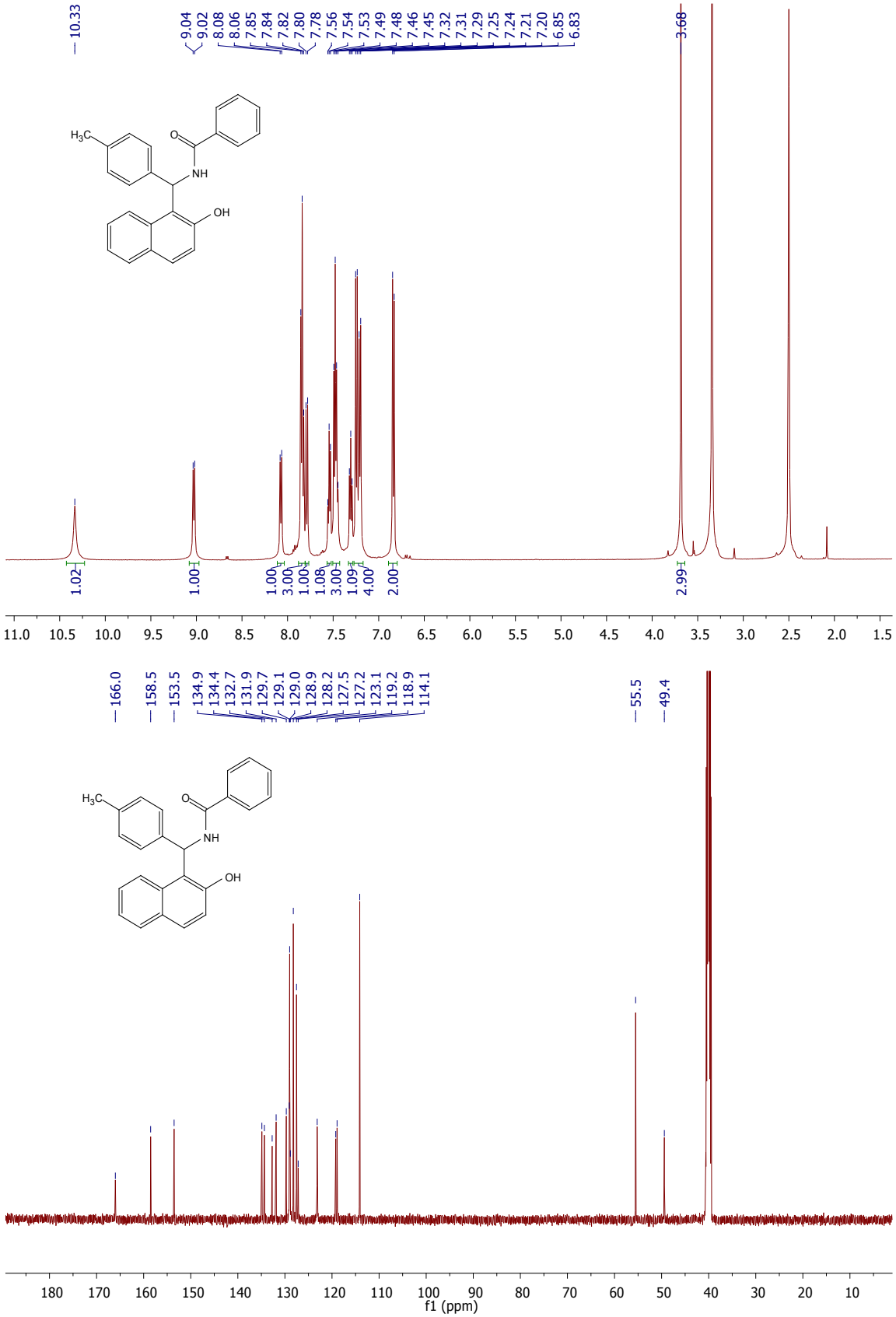
Section S5. ^{1}H, ^{13}C NMR and HRMS spectroscopy

^{1}H and ^{13}C NMR spectroscopy of *N*-((2-Hydroxynaphthalen-1-yl)(phenyl)methyl)benzamide (1)

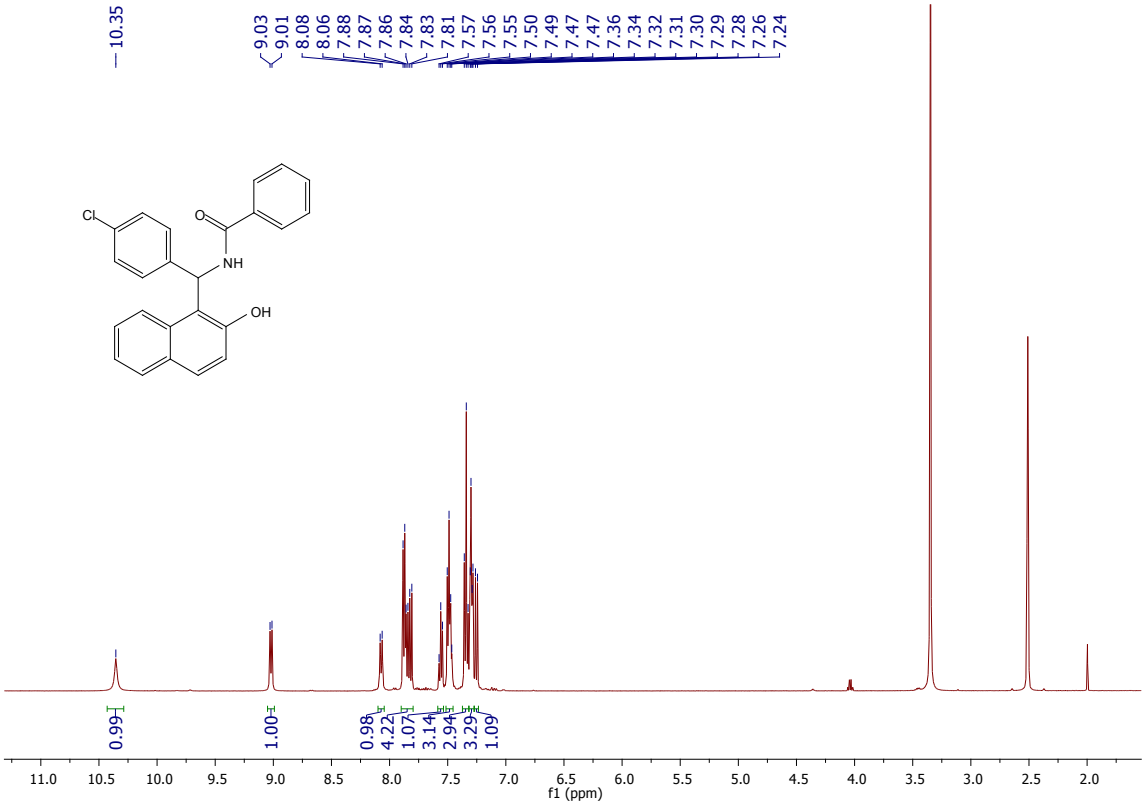


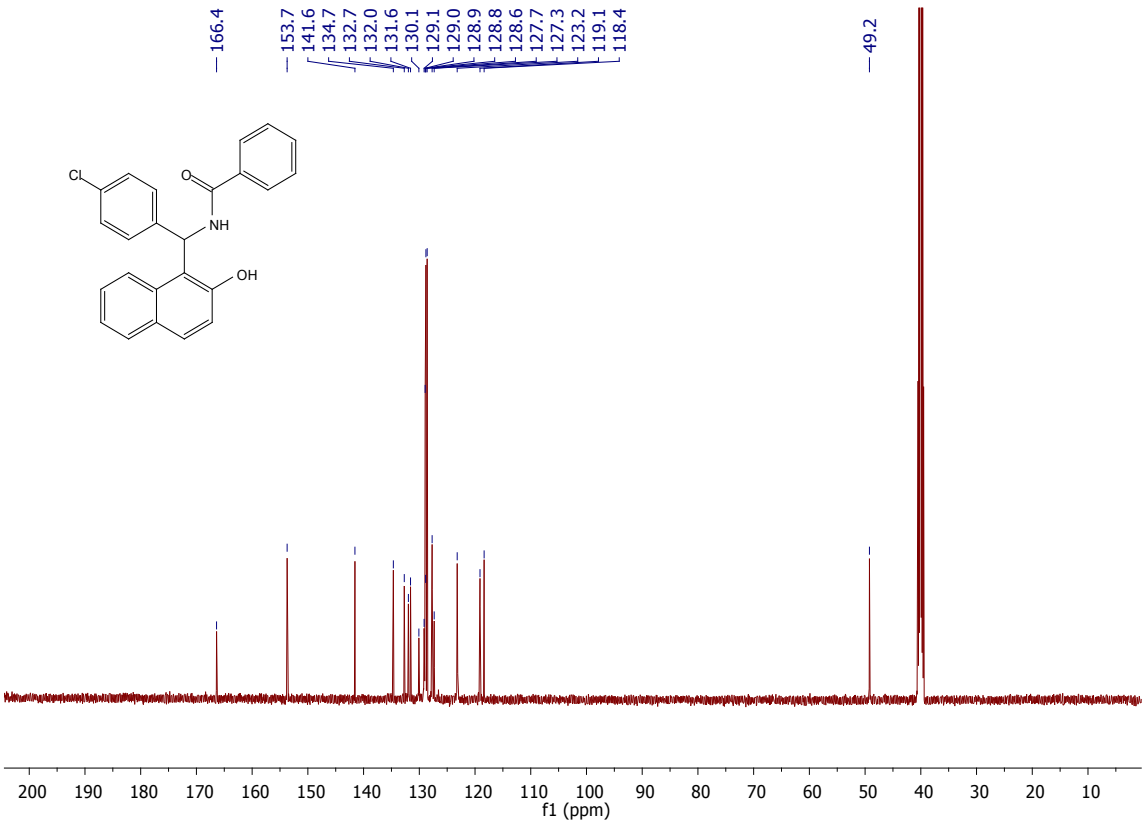


^{1}H and ^{13}C NMR spectroscopy of *N*-((2-hydroxynaphthalen-1-yl)(p-tolyl)methyl)benzamide (2)

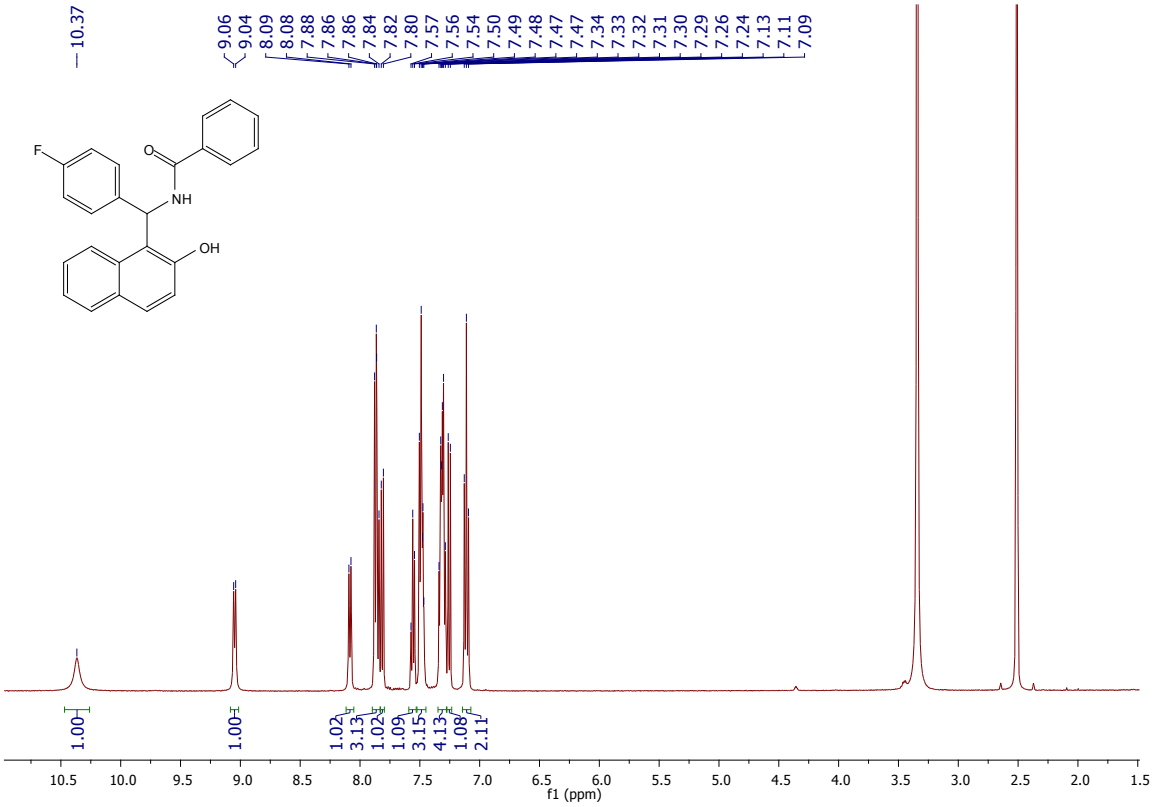


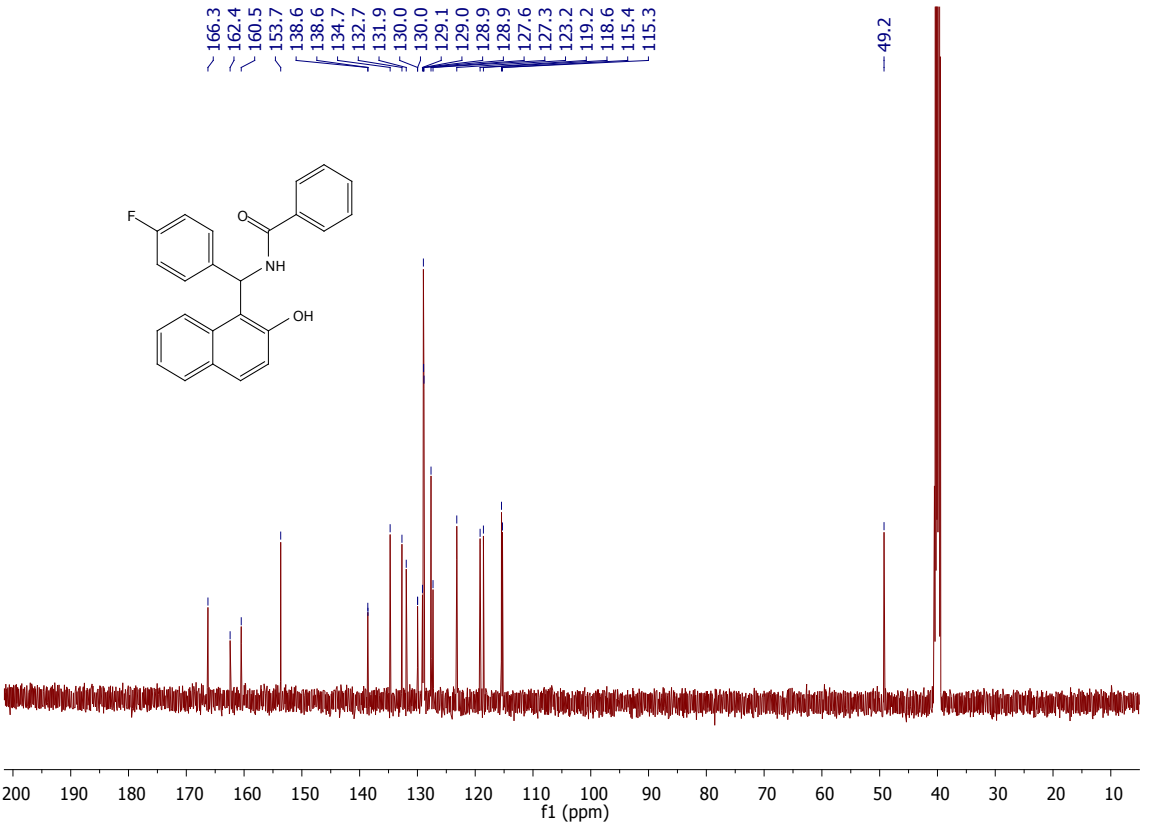
^{1}H and ^{13}C NMR spectroscopy of *N*-((4-Chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide (3)



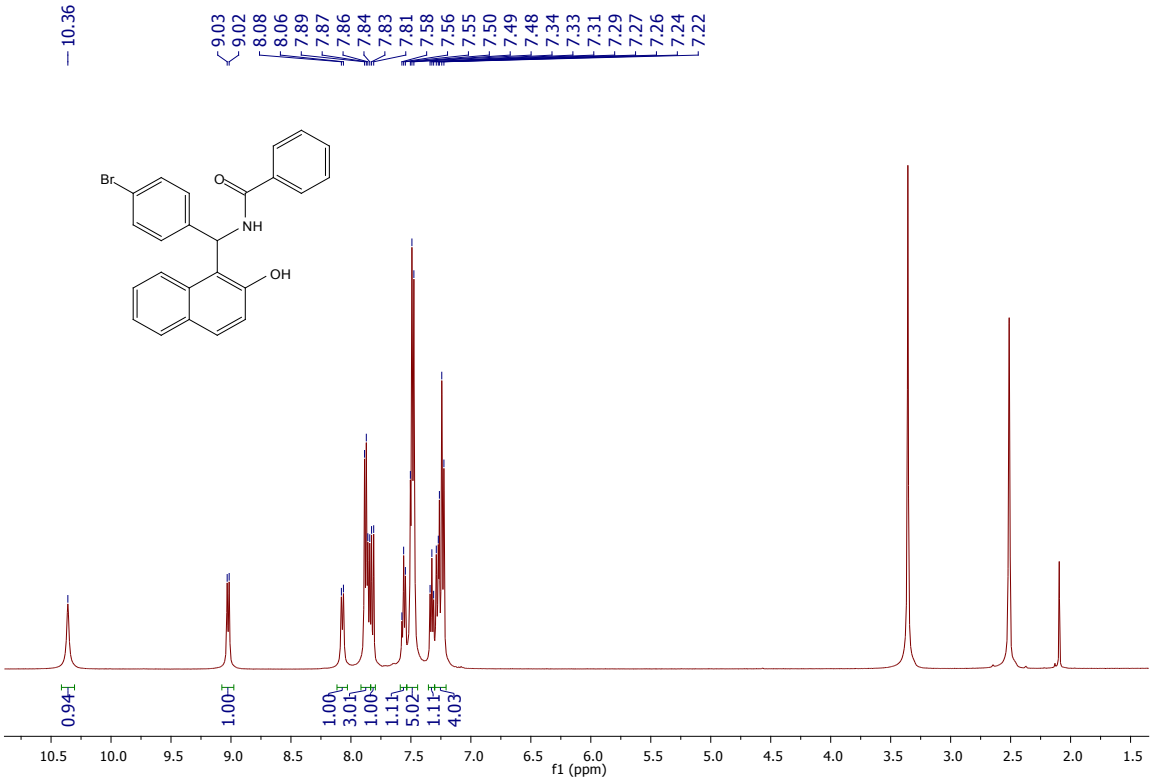


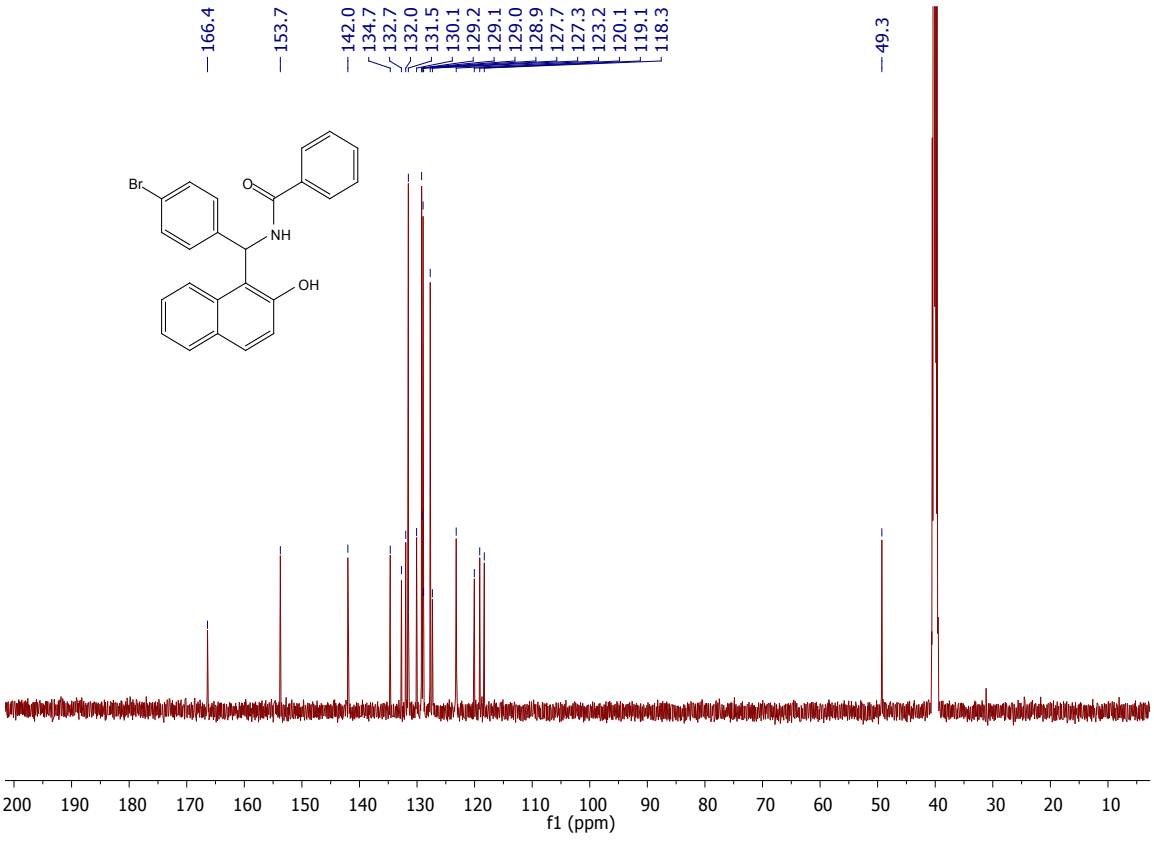
^{1}H and ^{13}C NMR spectroscopy of *N*-((4-Fluorophenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide (4)



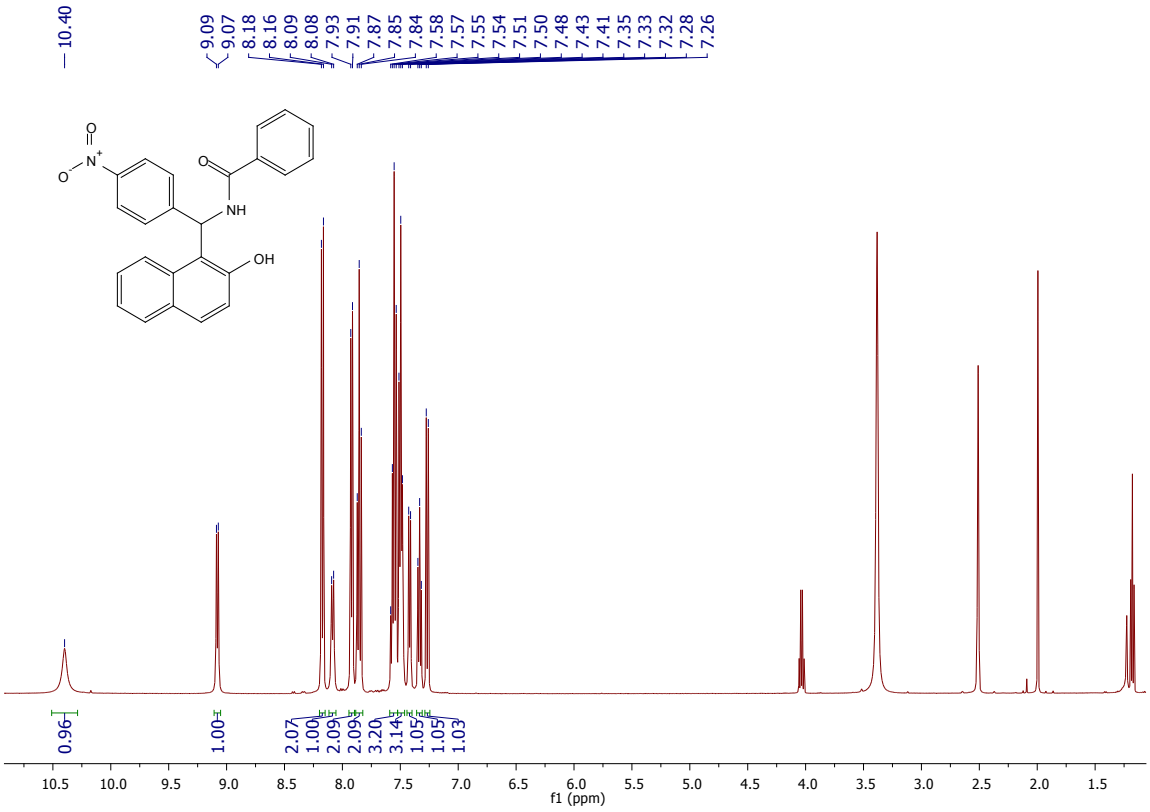


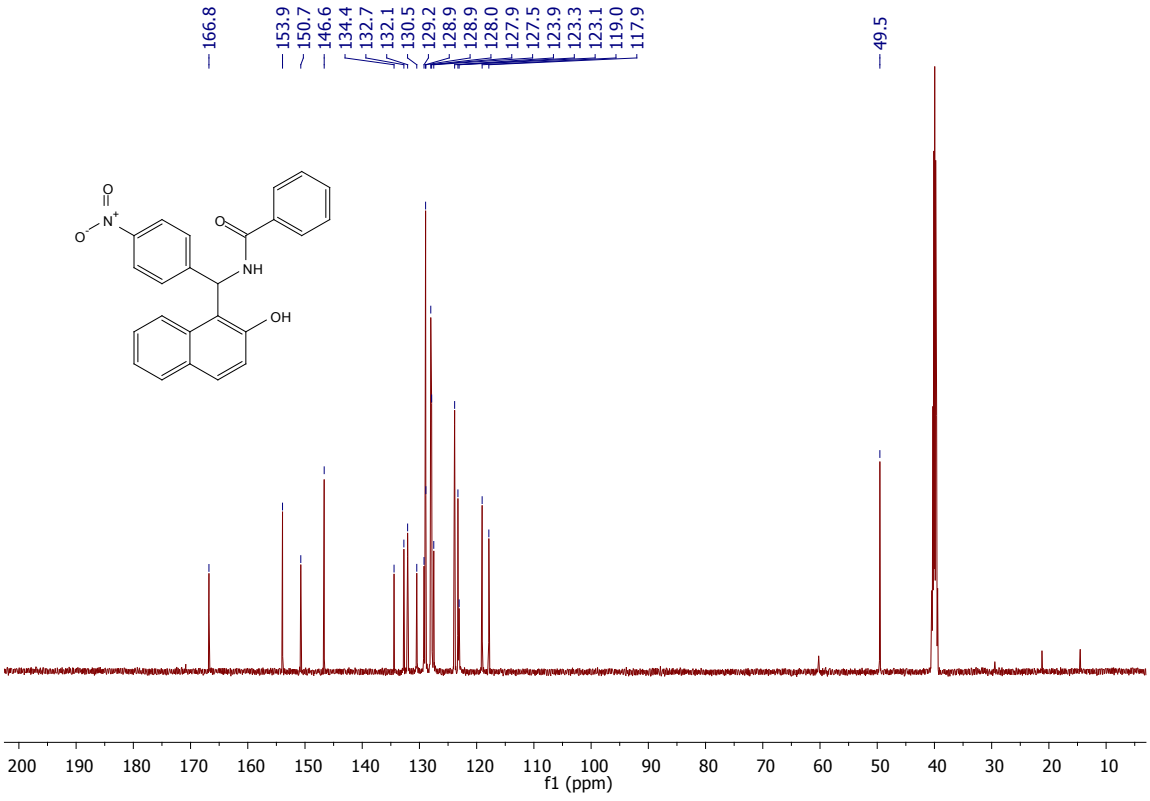
^{1}H and ^{13}C NMR spectroscopy of *N*-((4-Bromophenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide (5)



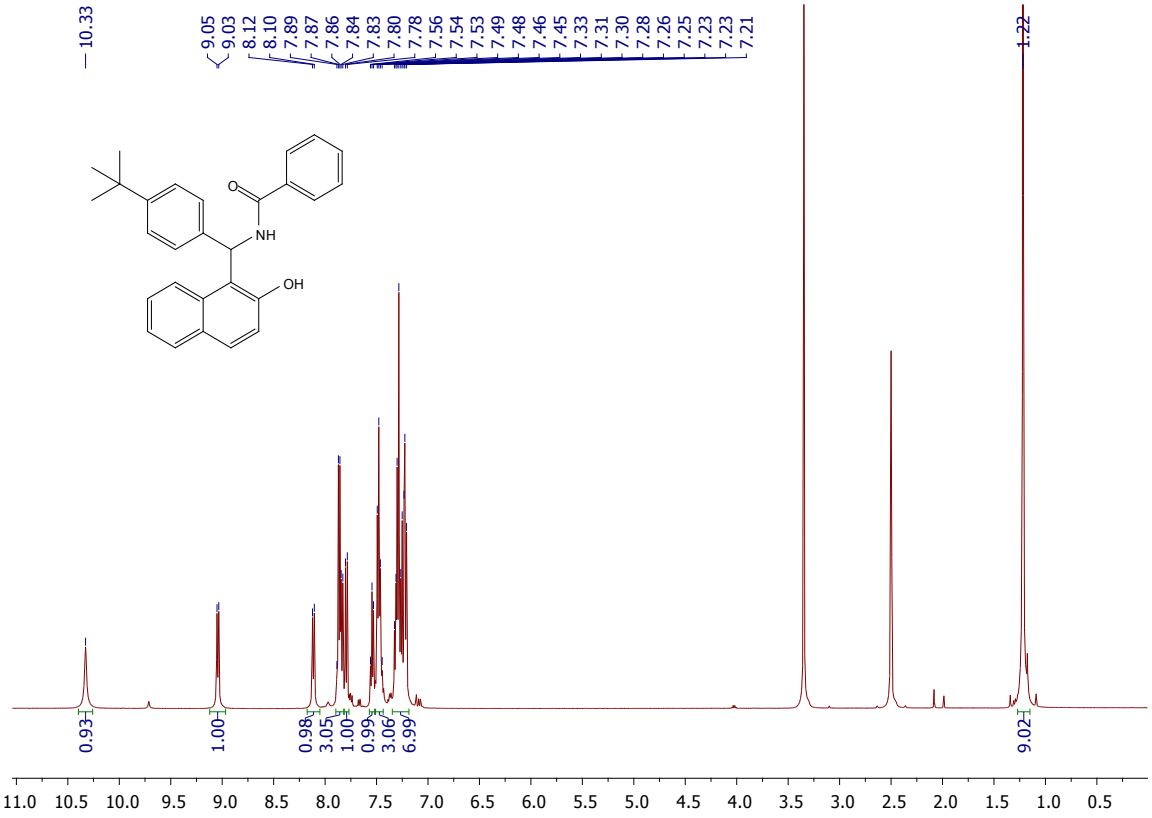


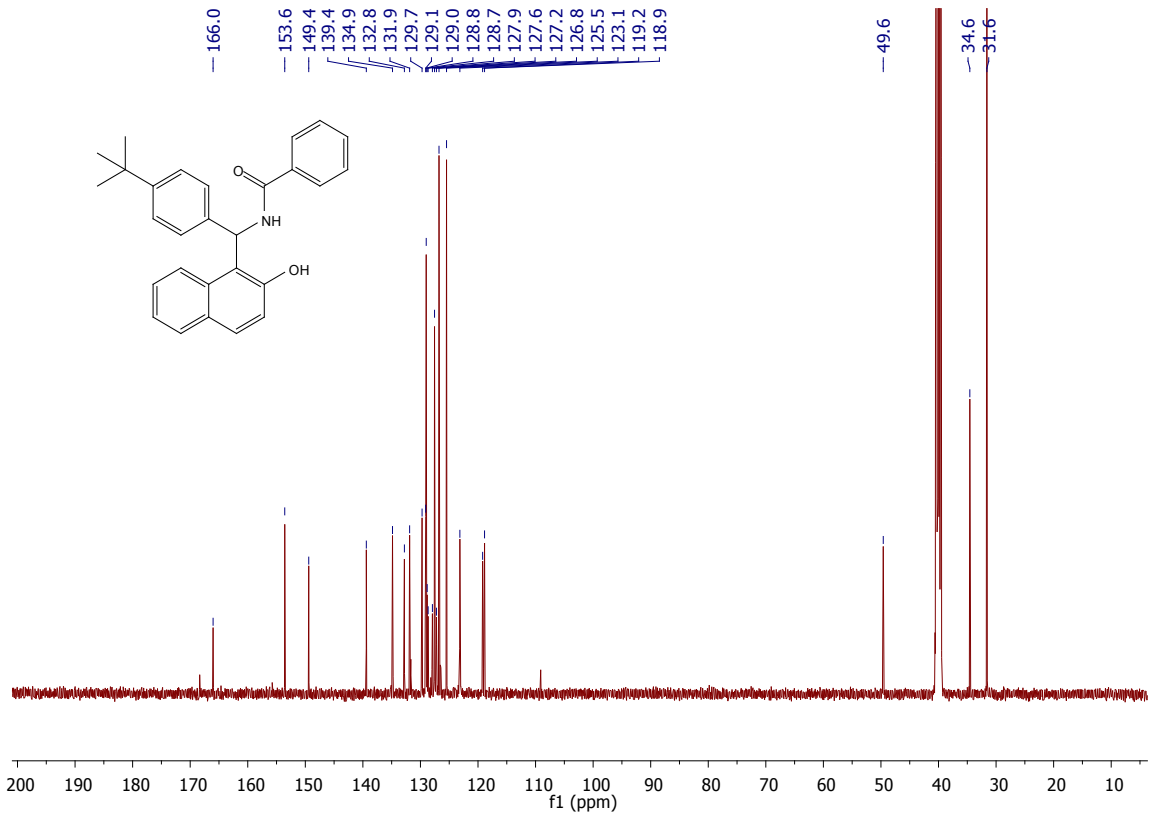
^{1}H and ^{13}C NMR spectroscopy of *N*-((2-Hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl)benzamide (6)



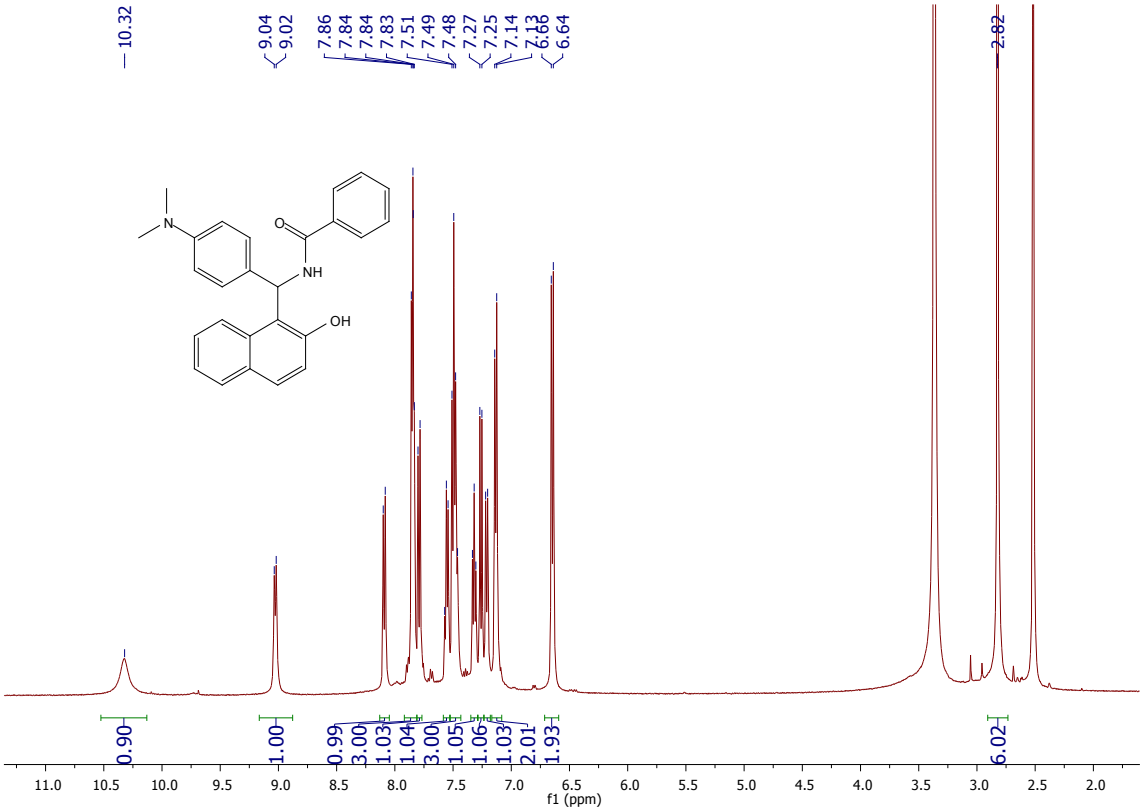


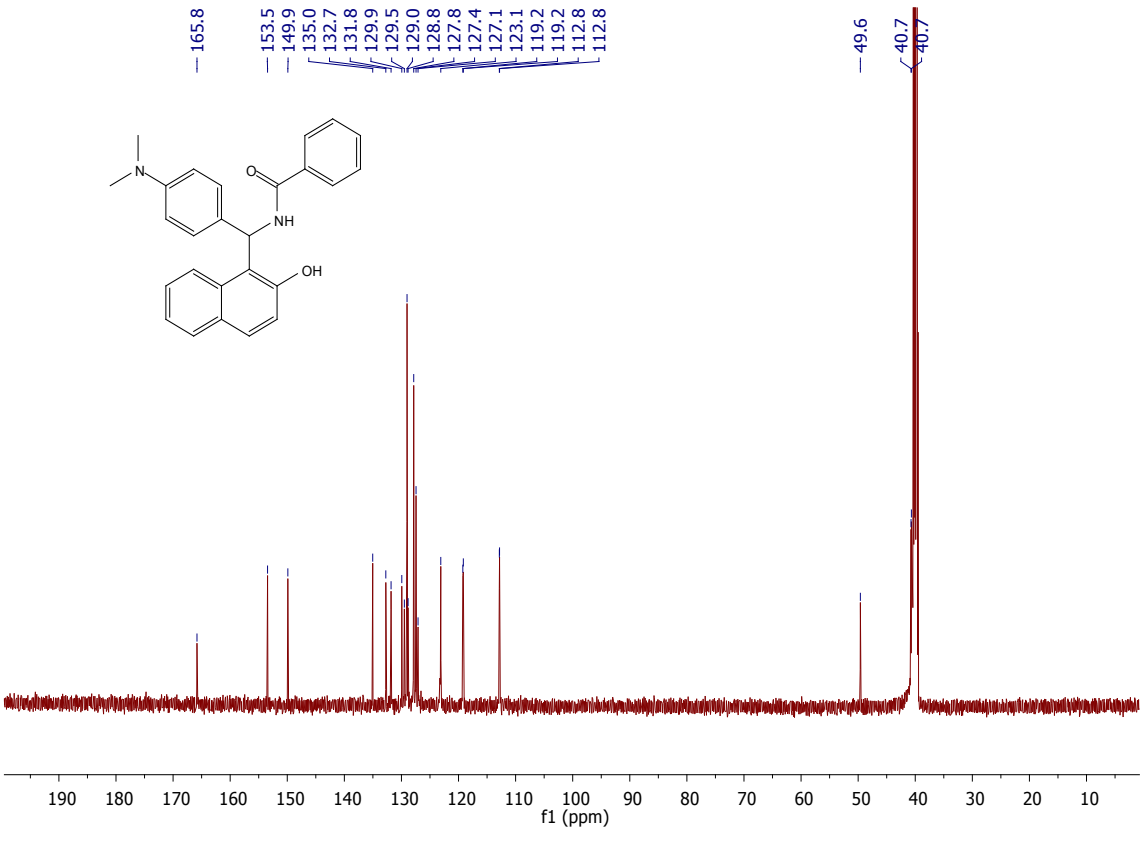
^{1}H and ^{13}C NMR spectroscopy of *N*-((4-(*tert*-Butyl)phenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide (7)



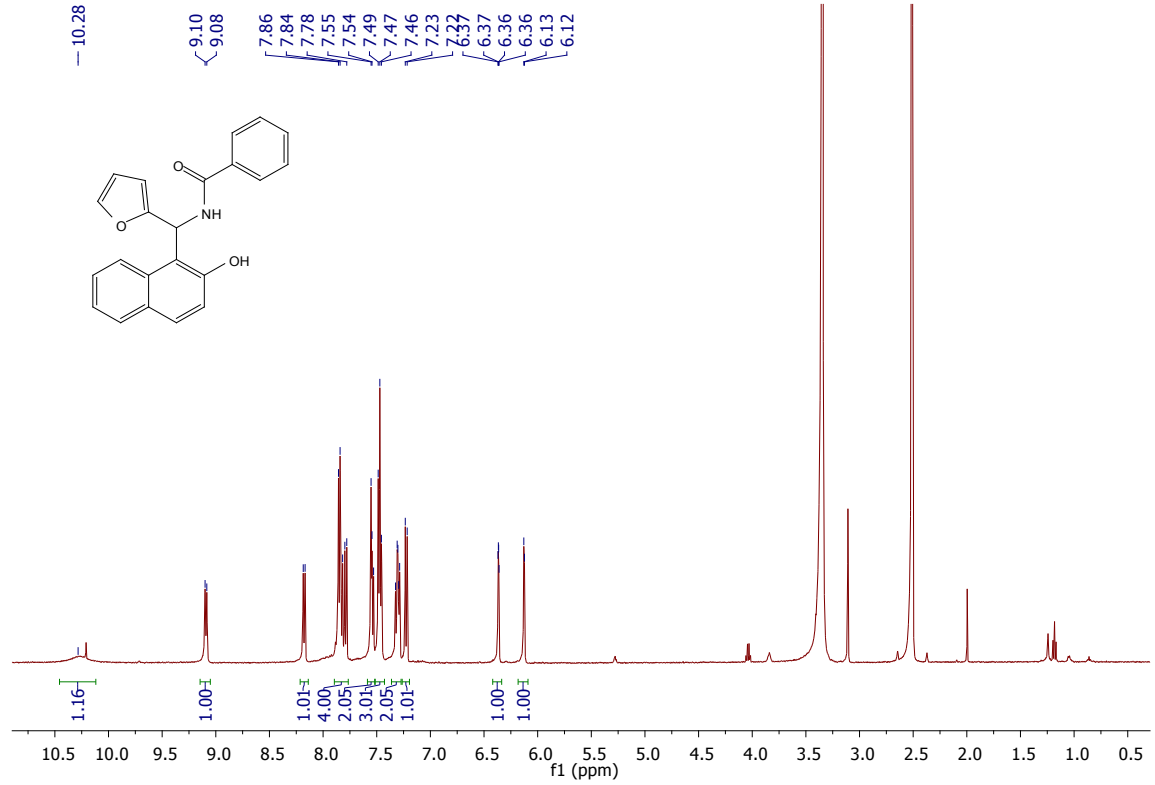


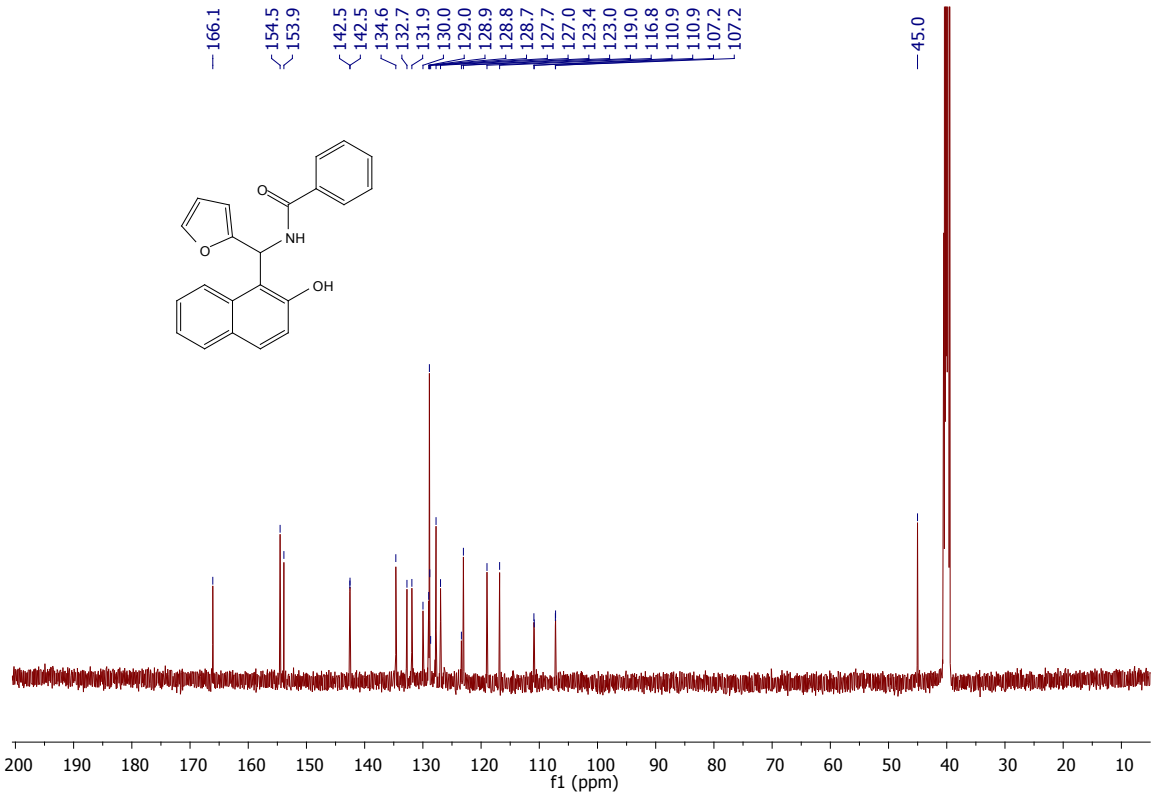
^{1}H and ^{13}C NMR spectroscopy of *N*-((4-(Dimethylamino)phenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide (8)



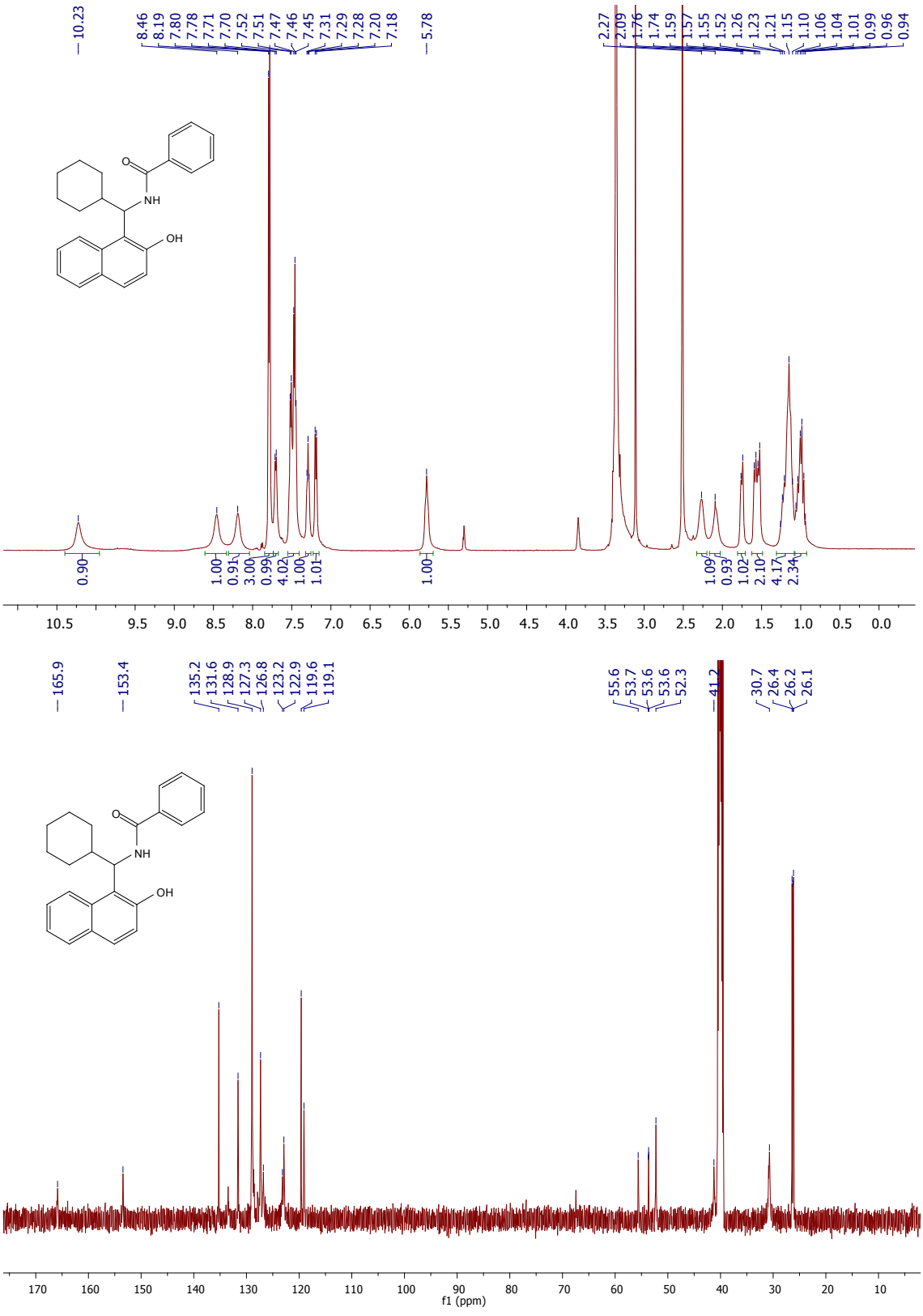


^{1}H and ^{13}C NMR spectroscopy of *N*-(Furan-2-yl(2-hydroxynaphthalen-1-yl)methyl)benzamide (9)

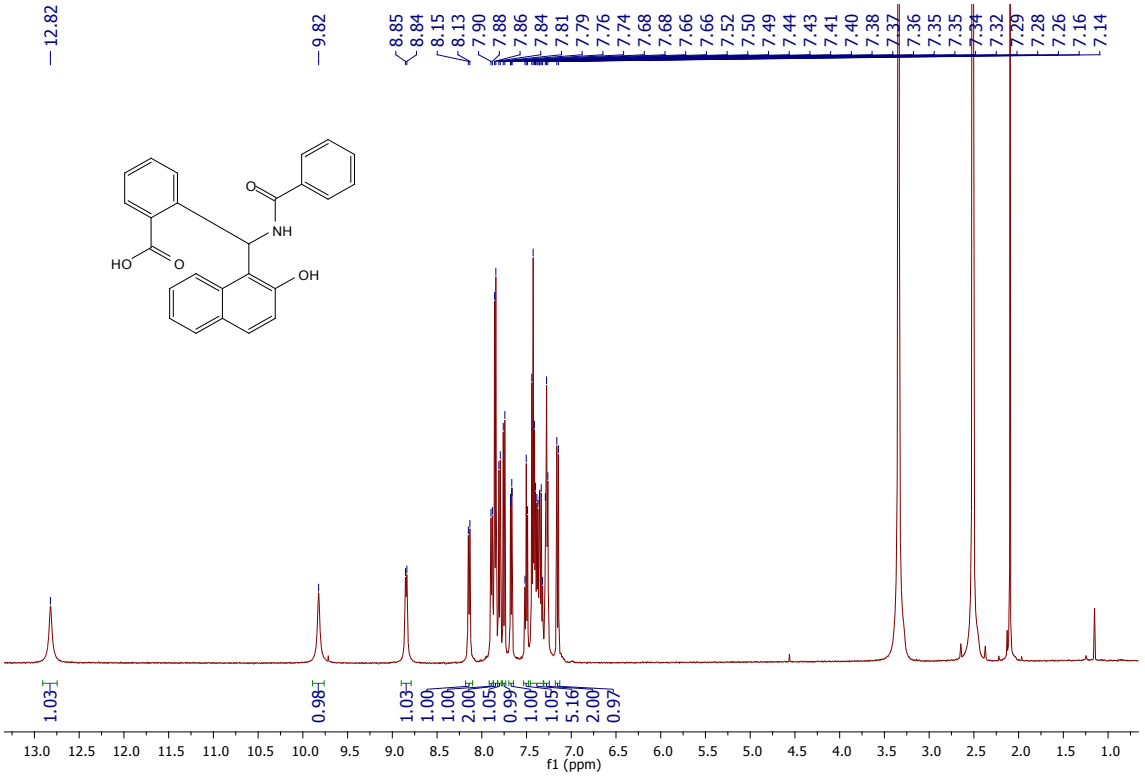


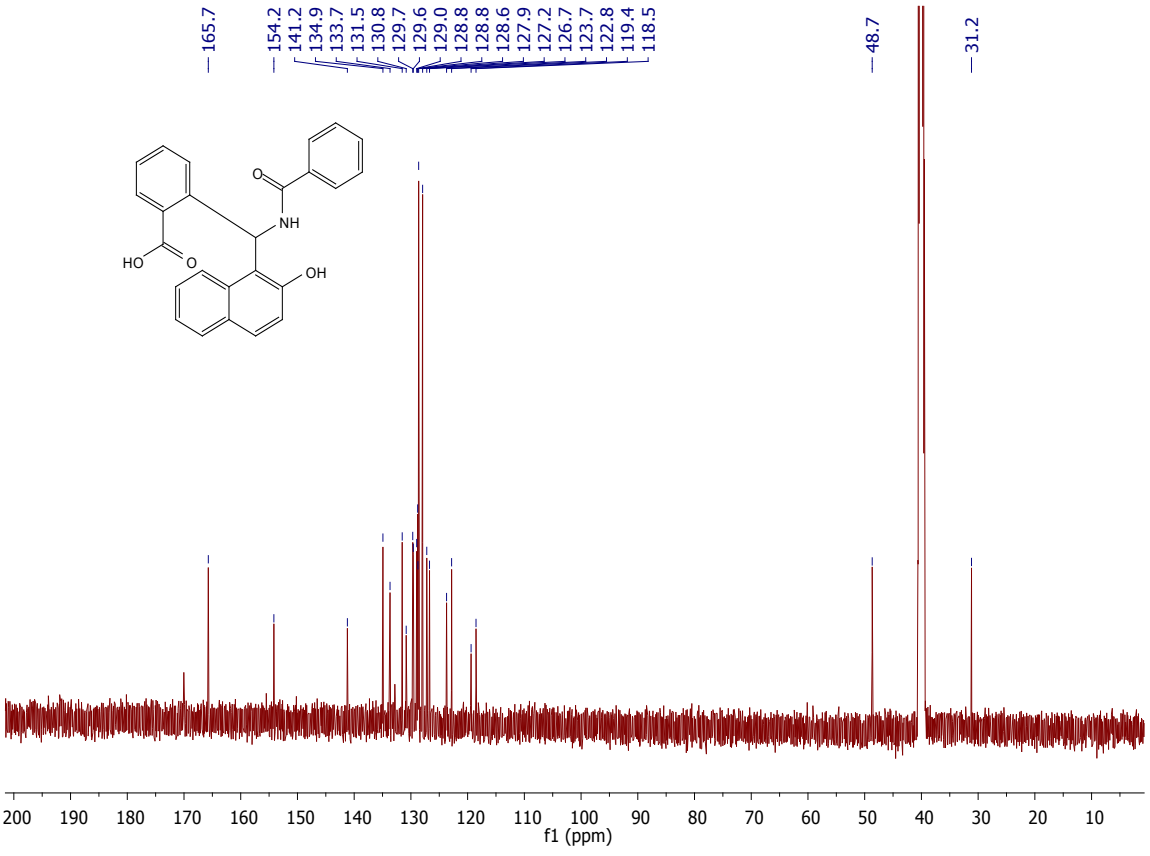


^{1}H and ^{13}C NMR spectroscopy of *N*-(Cyclohexyl(2-hydroxynaphthalen-1-yl)methyl)benzamide (10)



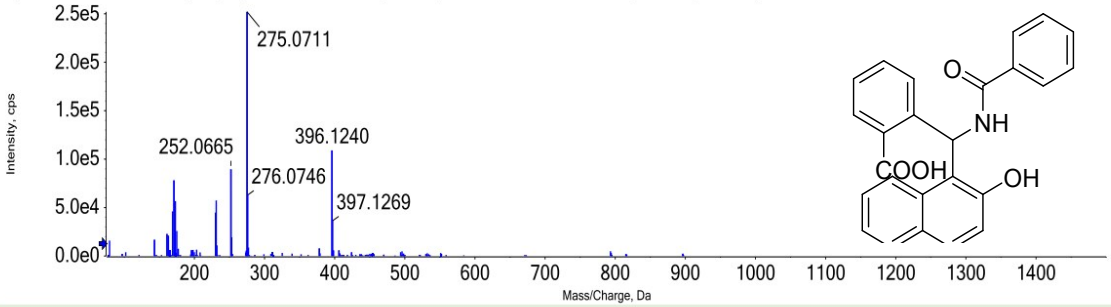
^{1}H and ^{13}C NMR spectroscopy and HRMS and HRMS of 2-(Benzamido(2-hydroxynaphthalen-1-yl)methyl)benzoic acid (11)





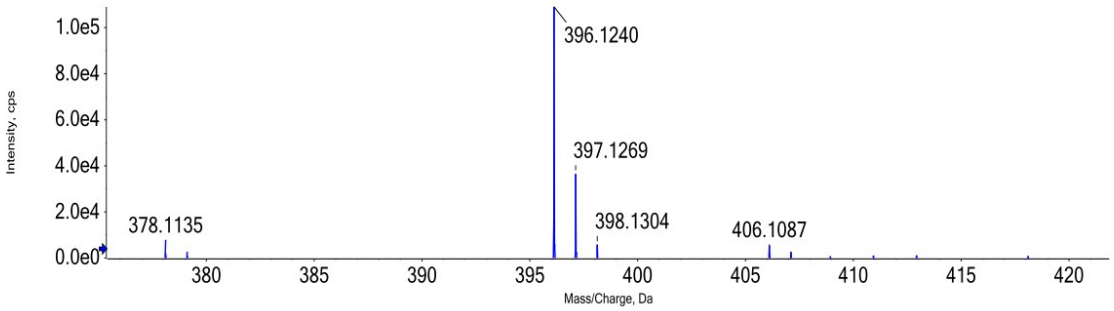
Full mass spectrum

Spectrum from VF6-ESI.wiff2 (sample 1) - VF6-ESI, -TOF MS (75 - 1500) from 0.129 min, noise filtered (noise multiplier = 1.5), Gaussian smoothed (0.5 points)

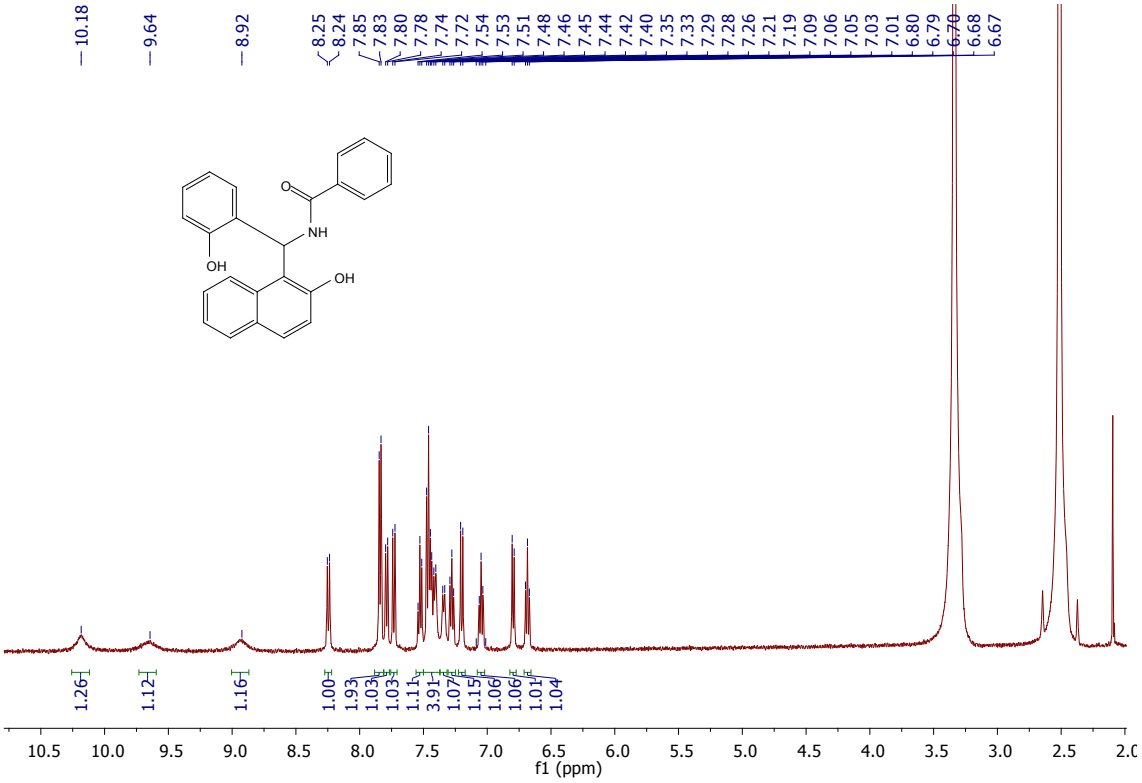


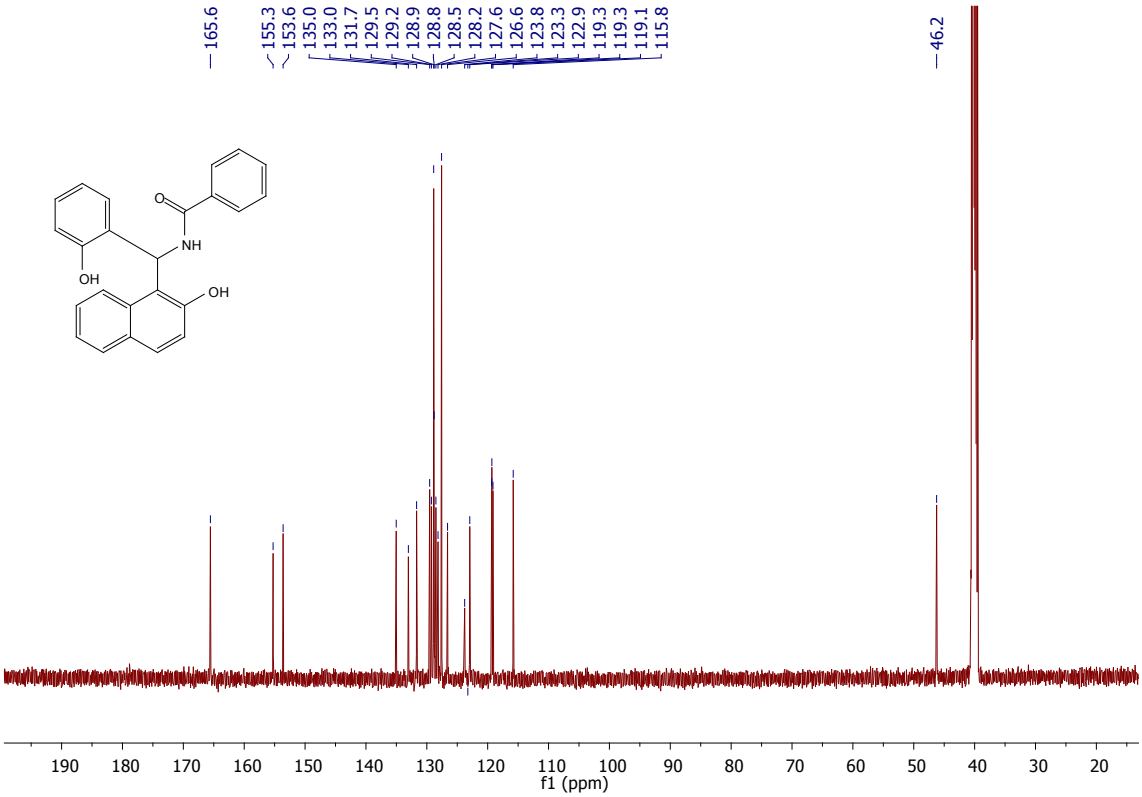
Expanded spectrum

Spectrum from VF6-ESI.wiff2 (sample 1) - VF6-ESI, -TOF MS (75 - 1500) from 0.129 min, noise filtered (noise multiplier = 1.5), Gaussian smoothed (0.5 points)

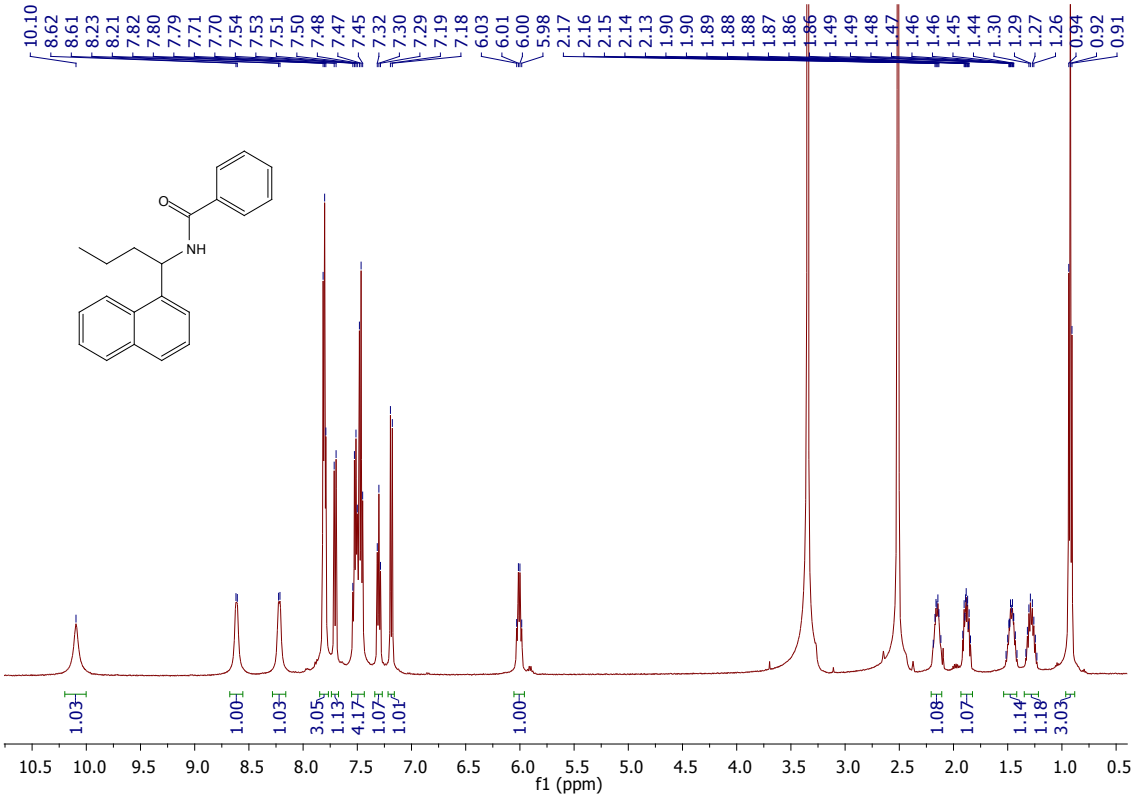


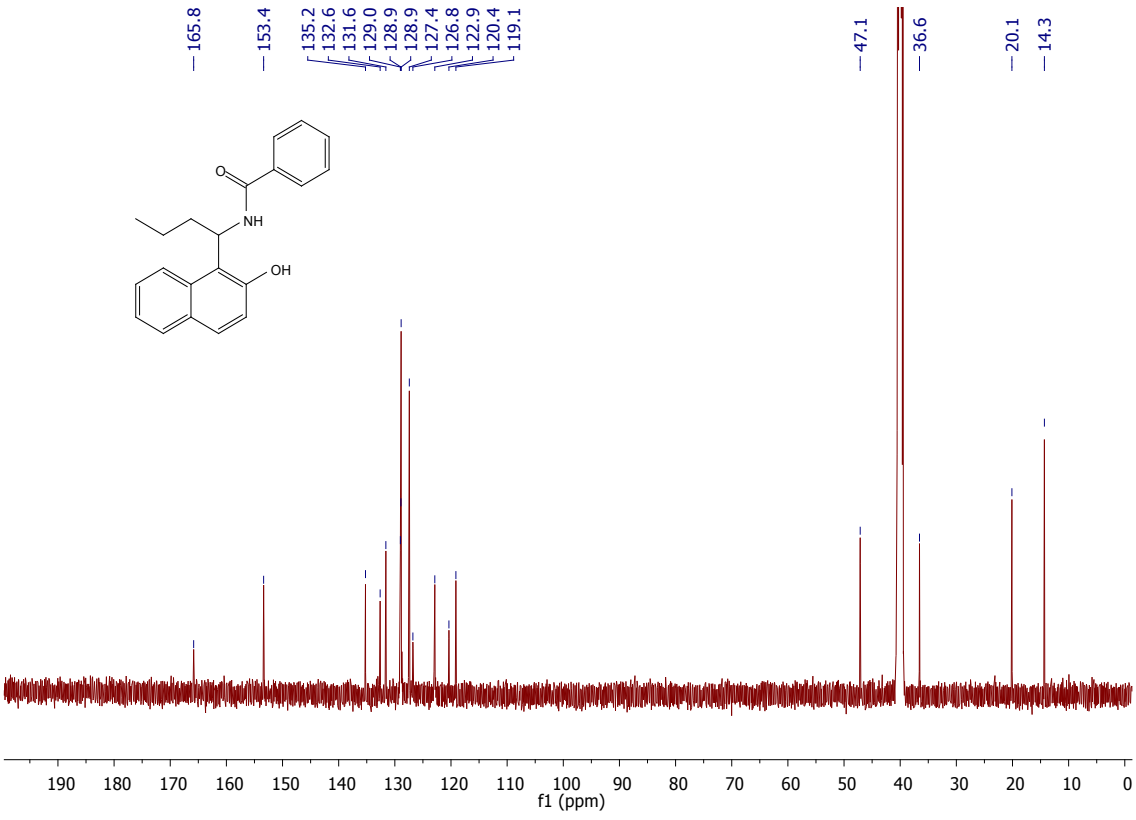
^{1}H and ^{13}C NMR spectroscopy of *N*-((2-Hydroxynaphthalen-1-yl)(2-hydroxyphenyl)methyl)benzamide (12)



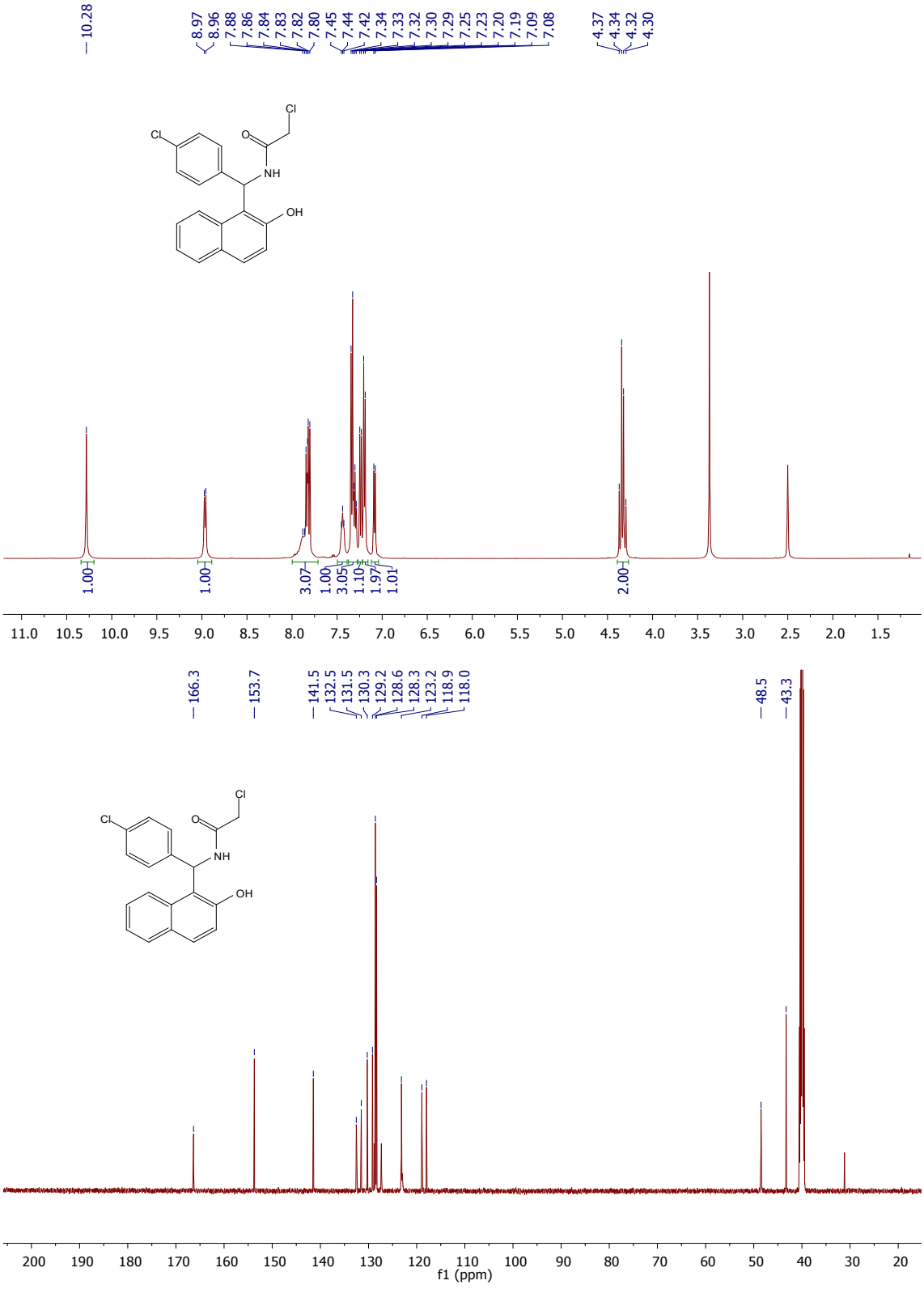


^{1}H and ^{13}C NMR spectroscopy of *N*-(1-(2-Hydroxynaphthalen-1-yl)butyl)benzamide (13)

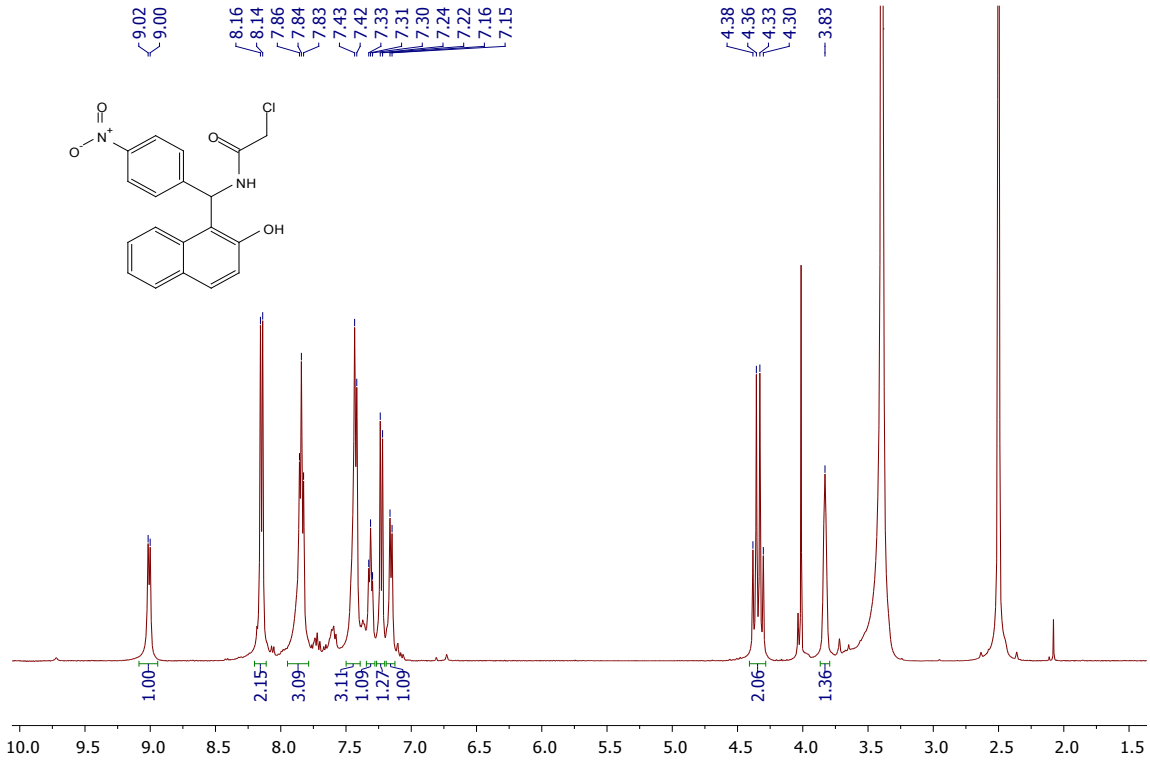


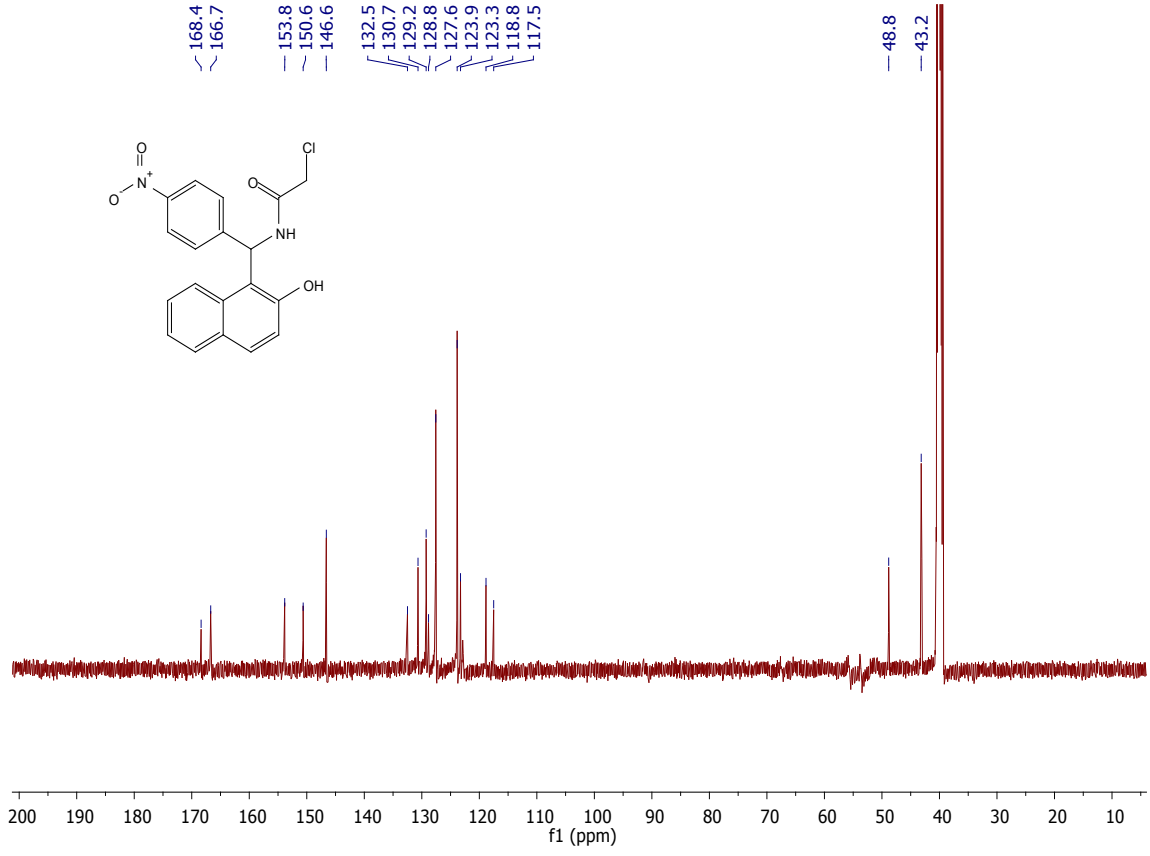


^{1}H and ^{13}C NMR spectroscopy of 2-Chloro-*N*-((4-chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)acetamide (14)

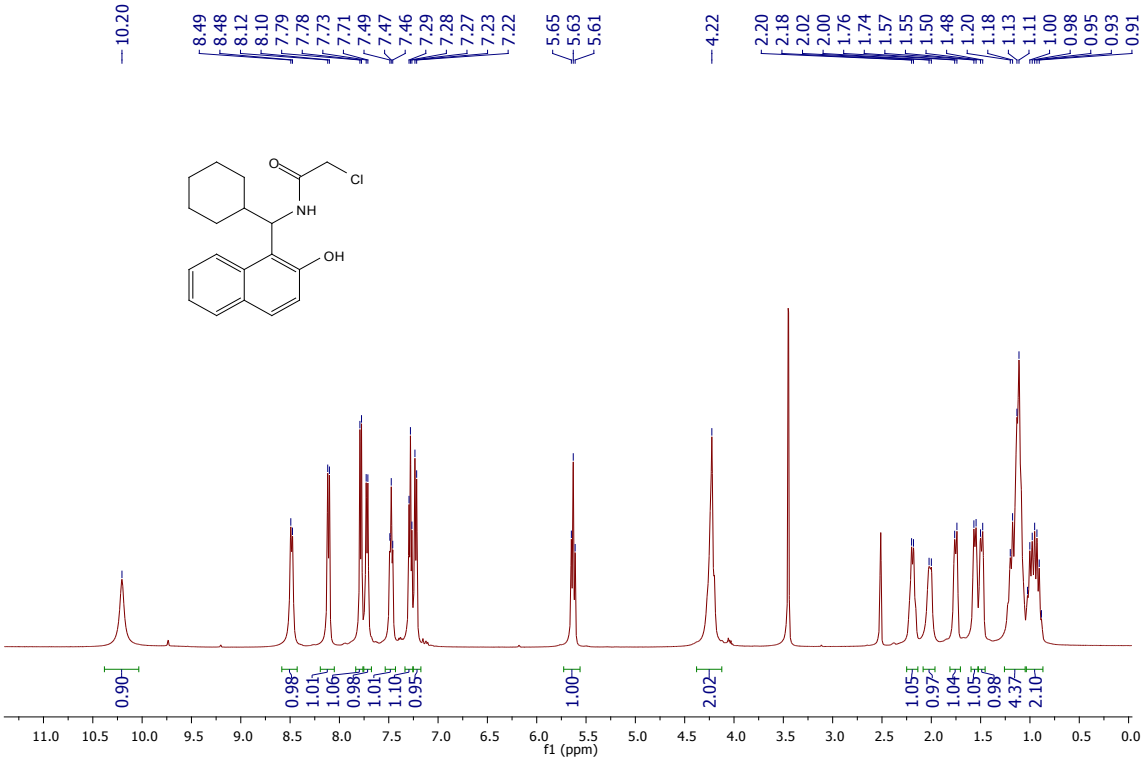


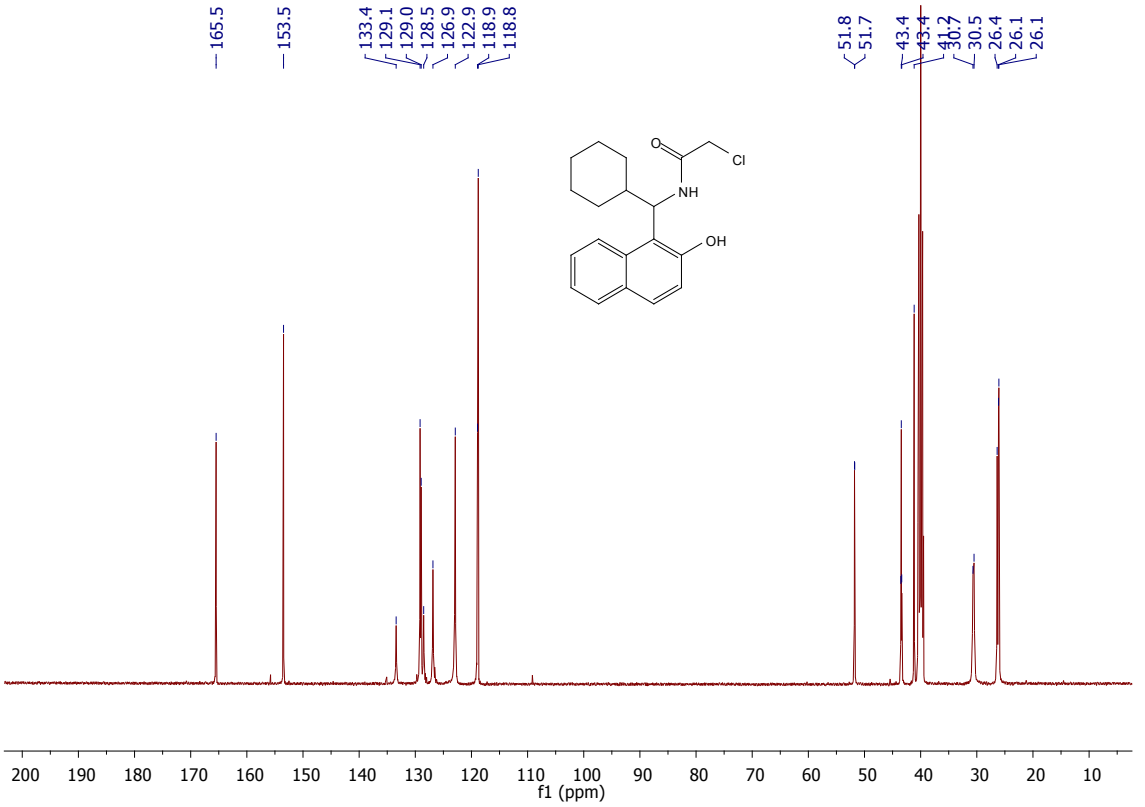
^{1}H and ^{13}C NMR spectroscopy of 2-Chloro-*N*-((2-hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl)acetamide (15)





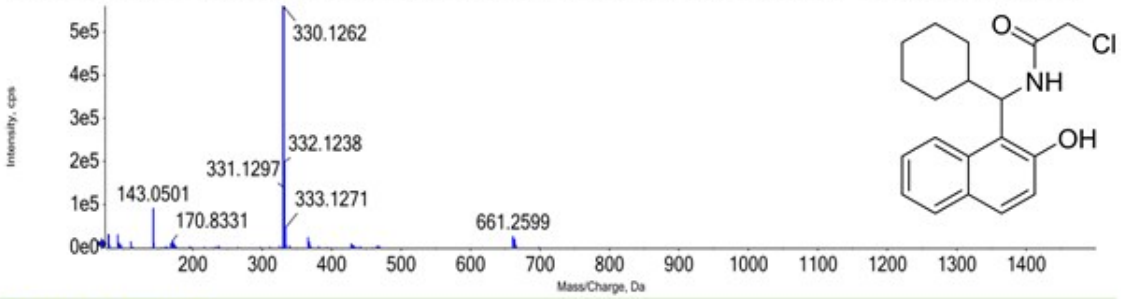
^{1}H and ^{13}C NMR spectroscopy and HRMS of 2-Chloro-*N*-(cyclohexyl(2-hydroxynaphthalen-1-yl)methyl)acetamide (16)





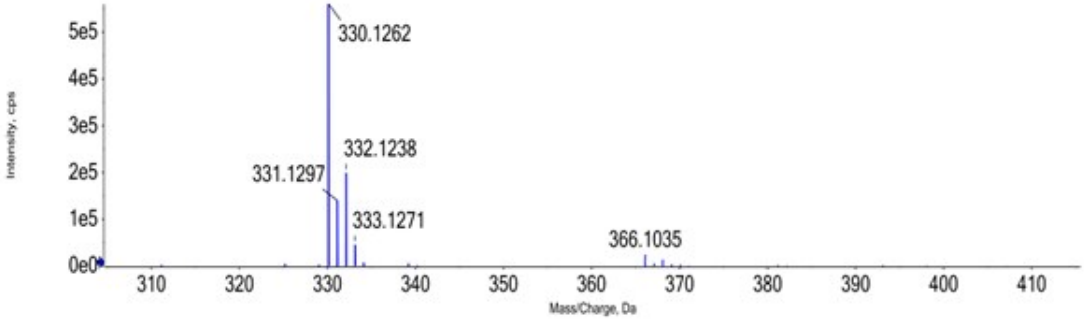
Full mass spectrum

Spectrum from CYCLOAMINO-ESI.wiff2 (sample 1) - CYCLOAMINO-ESI, -TOF MS (75 - 1500) from 0.111 min, noise filtered (noise multiplier = 1.5), Gaussian smoothed (0.5 points)

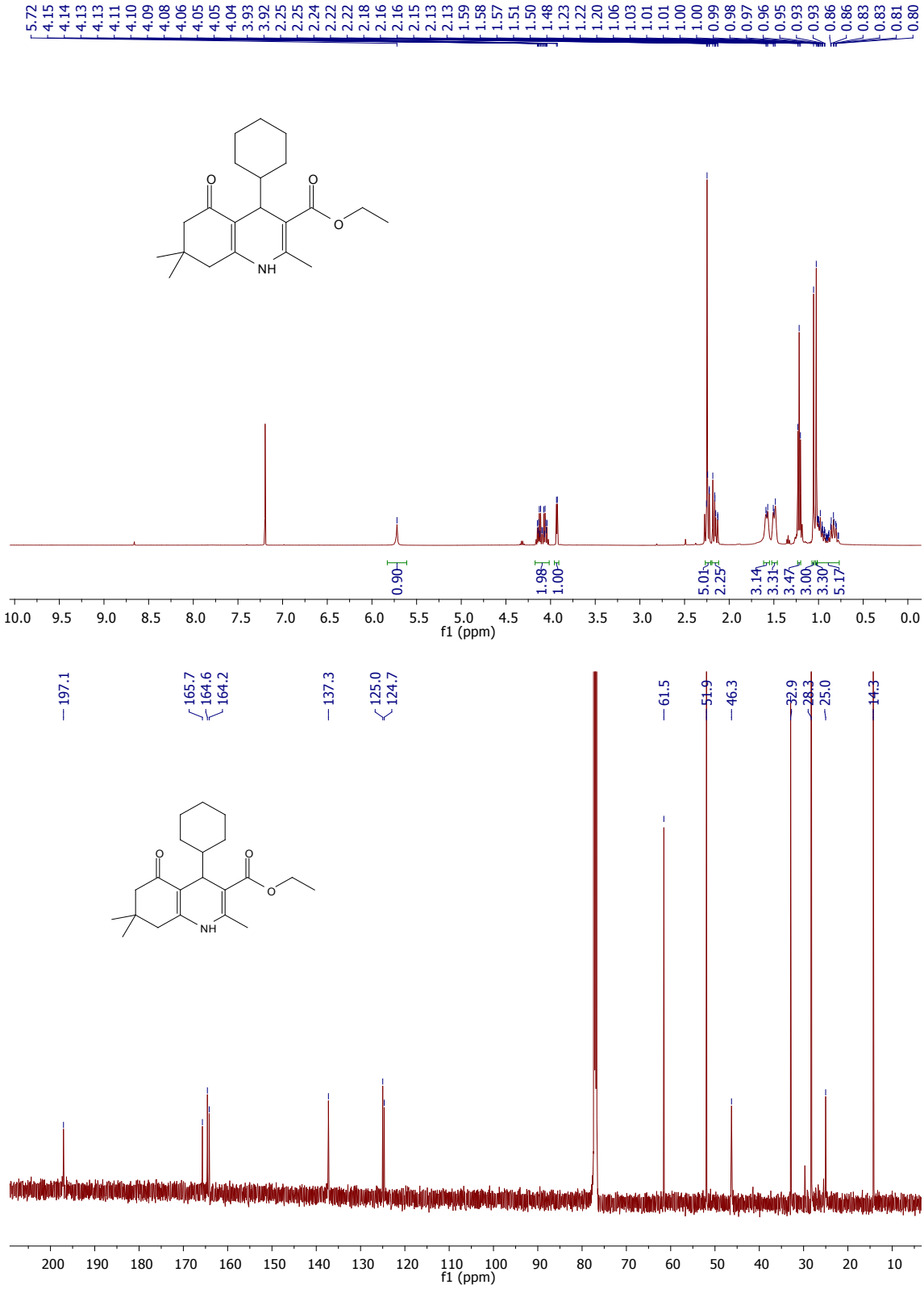


Expanded spectrum

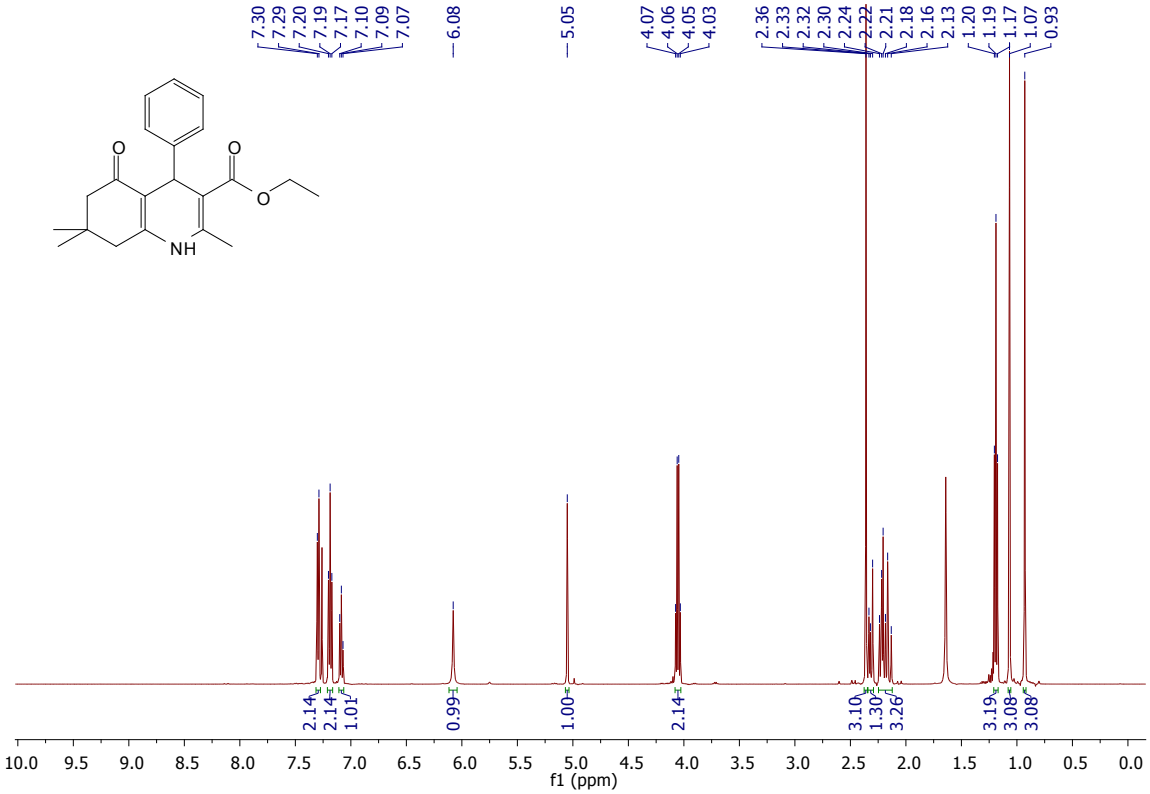
Spectrum from CYCLOAMINO-ESI.wiff2 (sample 1) - CYCLOAMINO-ESI, -TOF MS (75 - 1500) from 0.111 min, noise filtered (noise multiplier = 1.5), Gaussian smoothed (0.5 points)

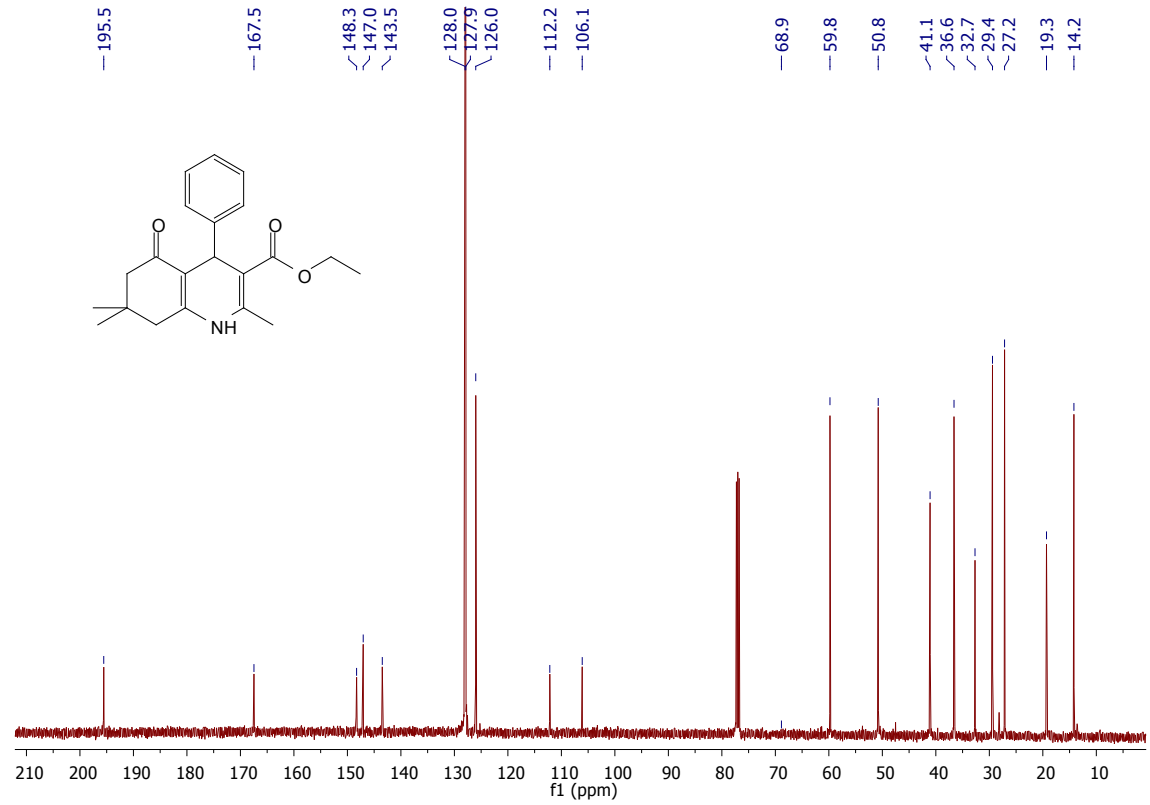


^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-cyclohexyl-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydro-quinolin-3-carboxylate (17)

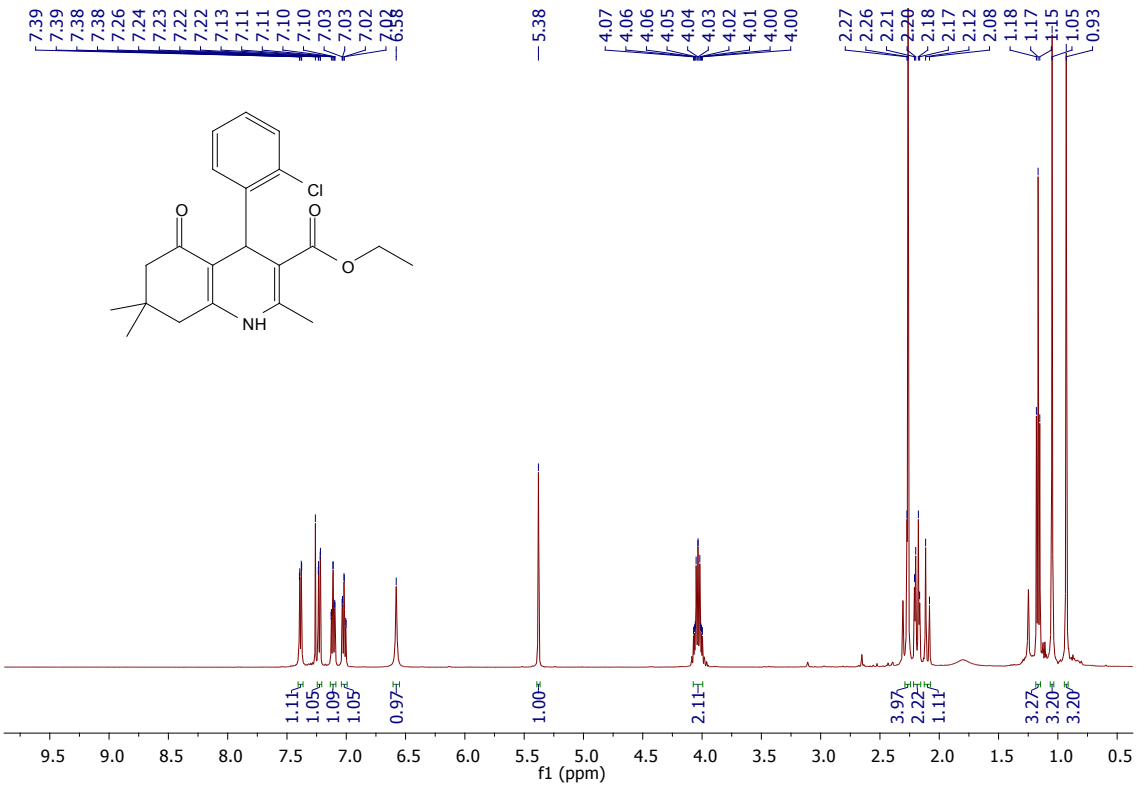


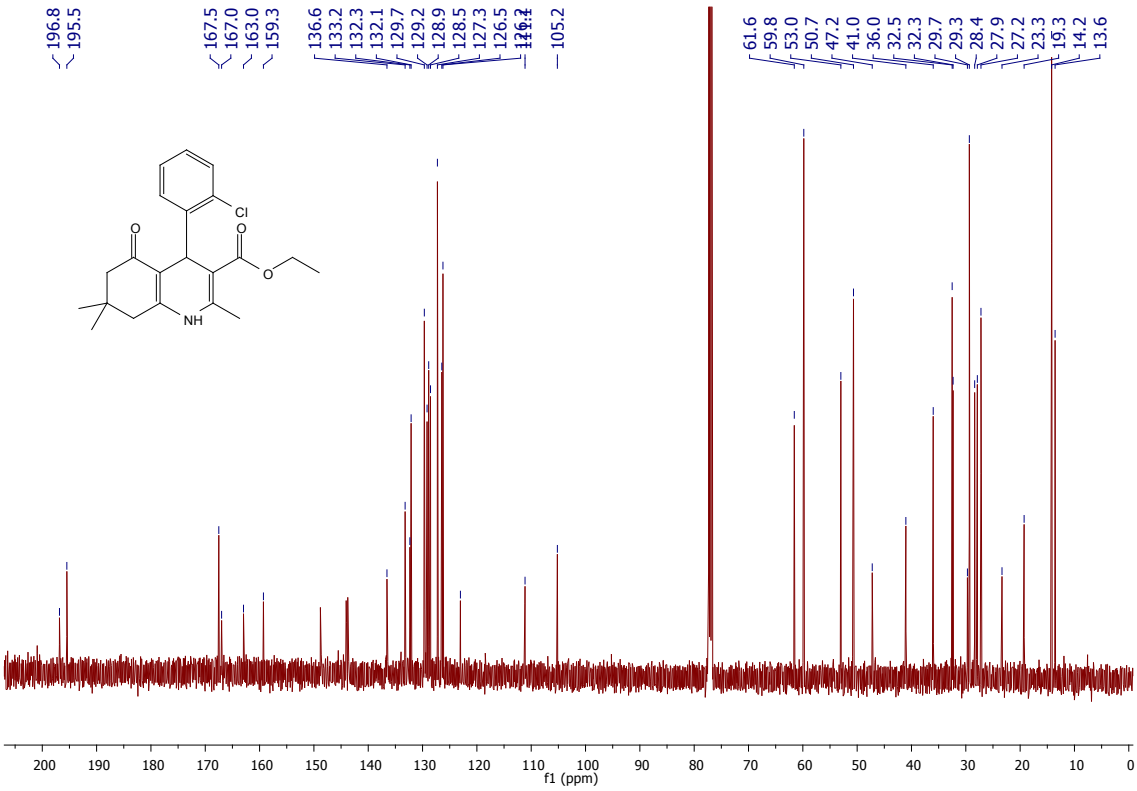
^{1}H and ^{13}C NMR spectroscopy of Ethyl 2,7,7-trimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (18)



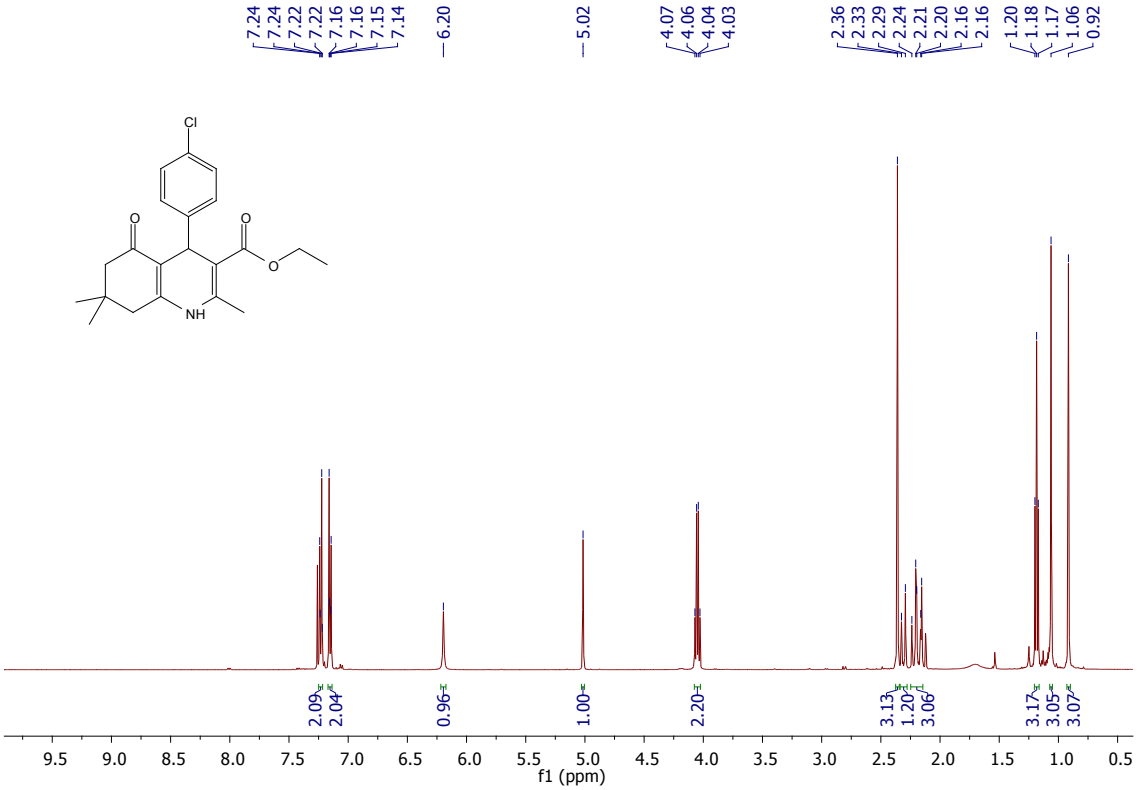


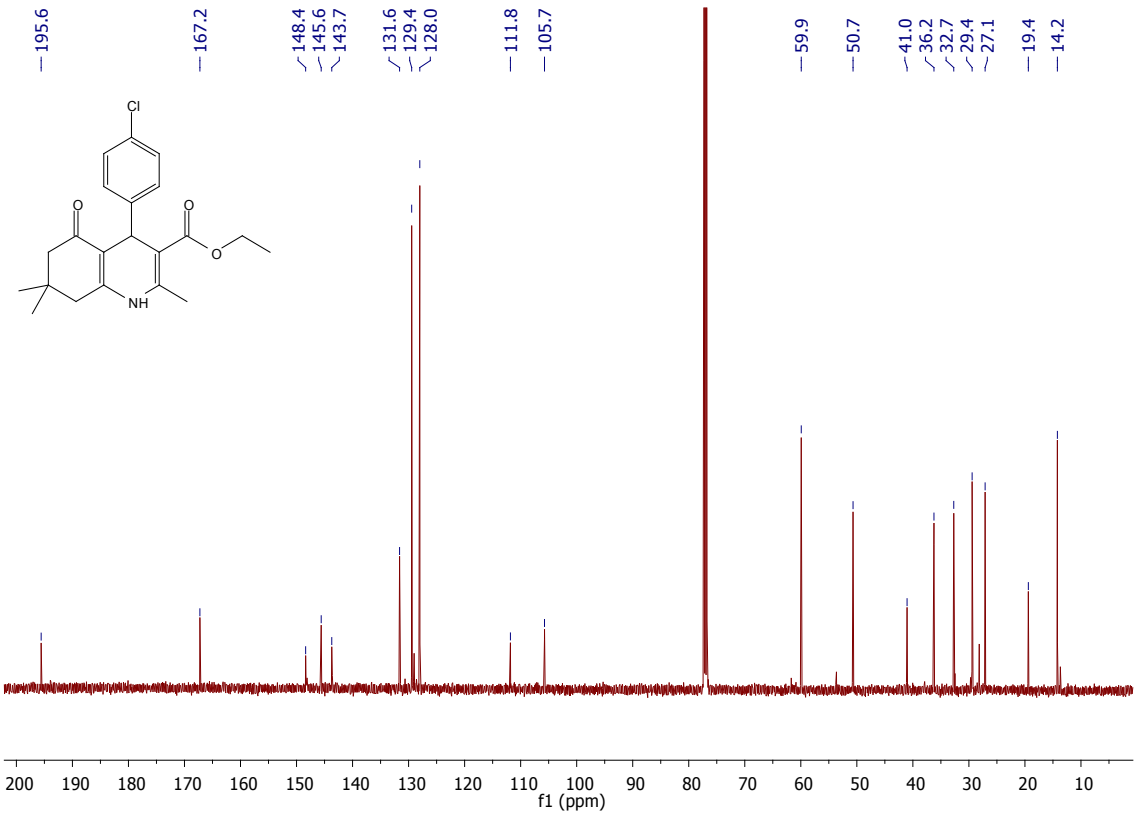
^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(2-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahyydroquinolin-3-carboxylate (19)



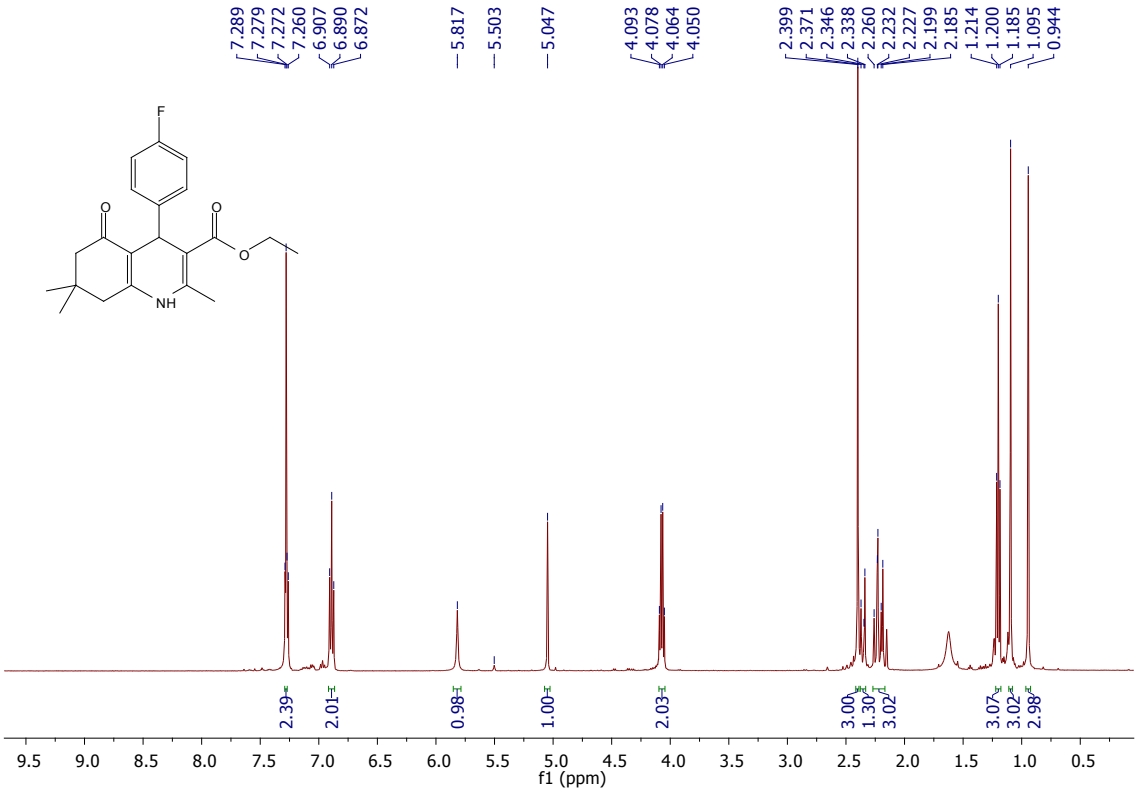


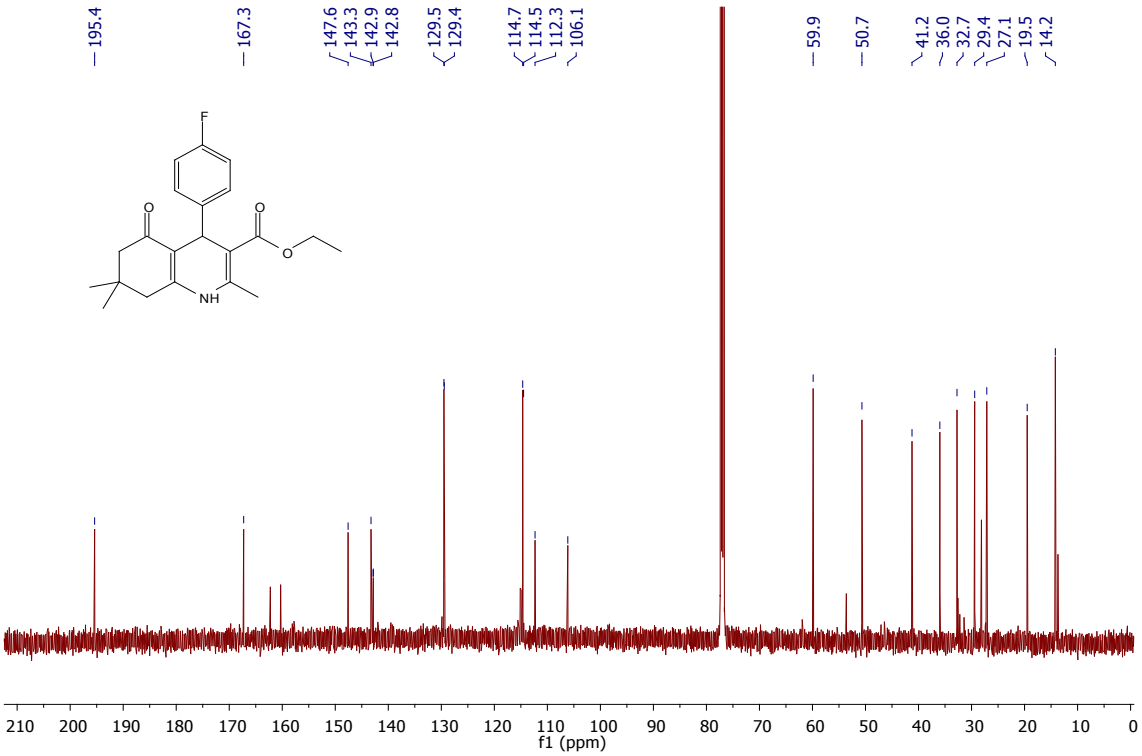
^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(4-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (20)



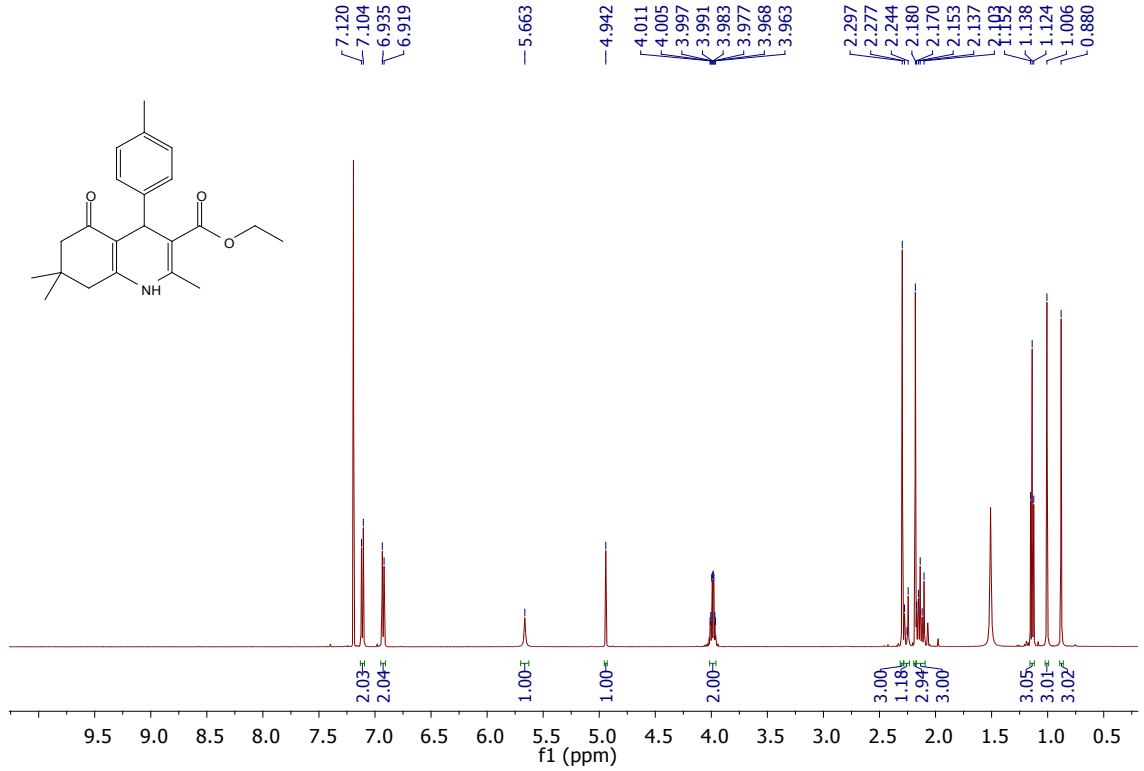


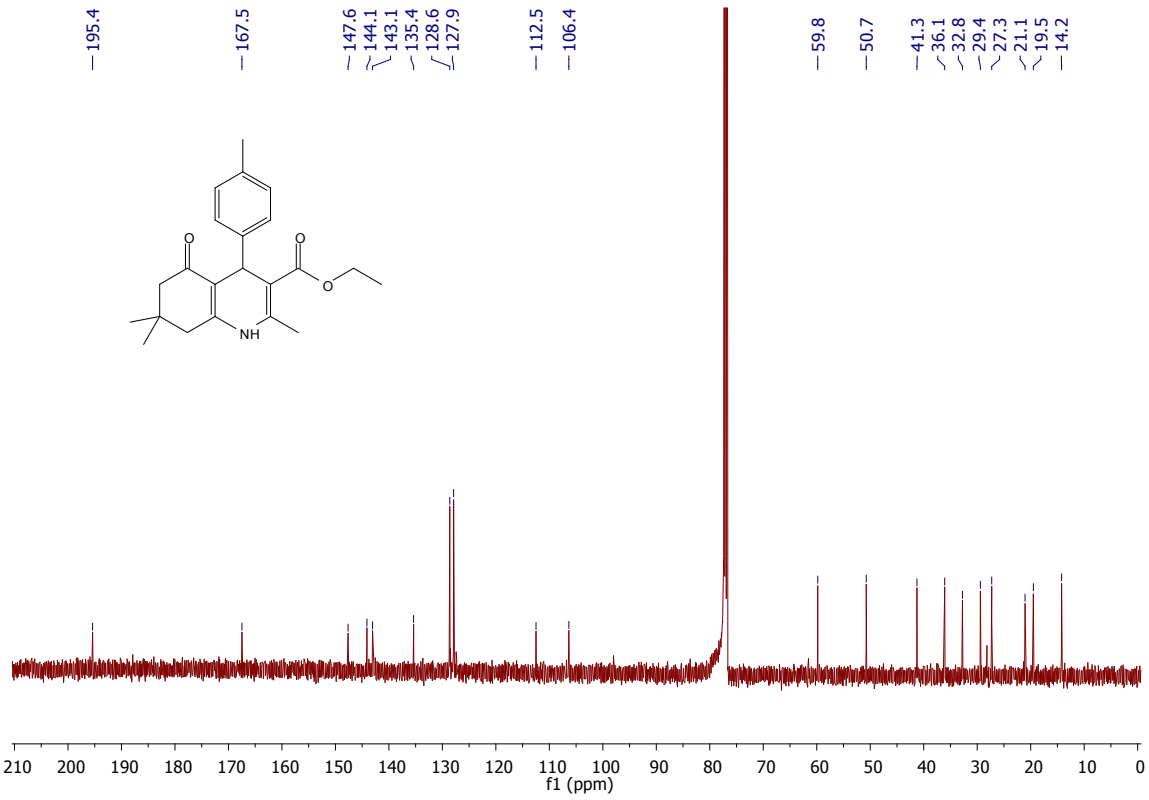
^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(4-fluorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (21)



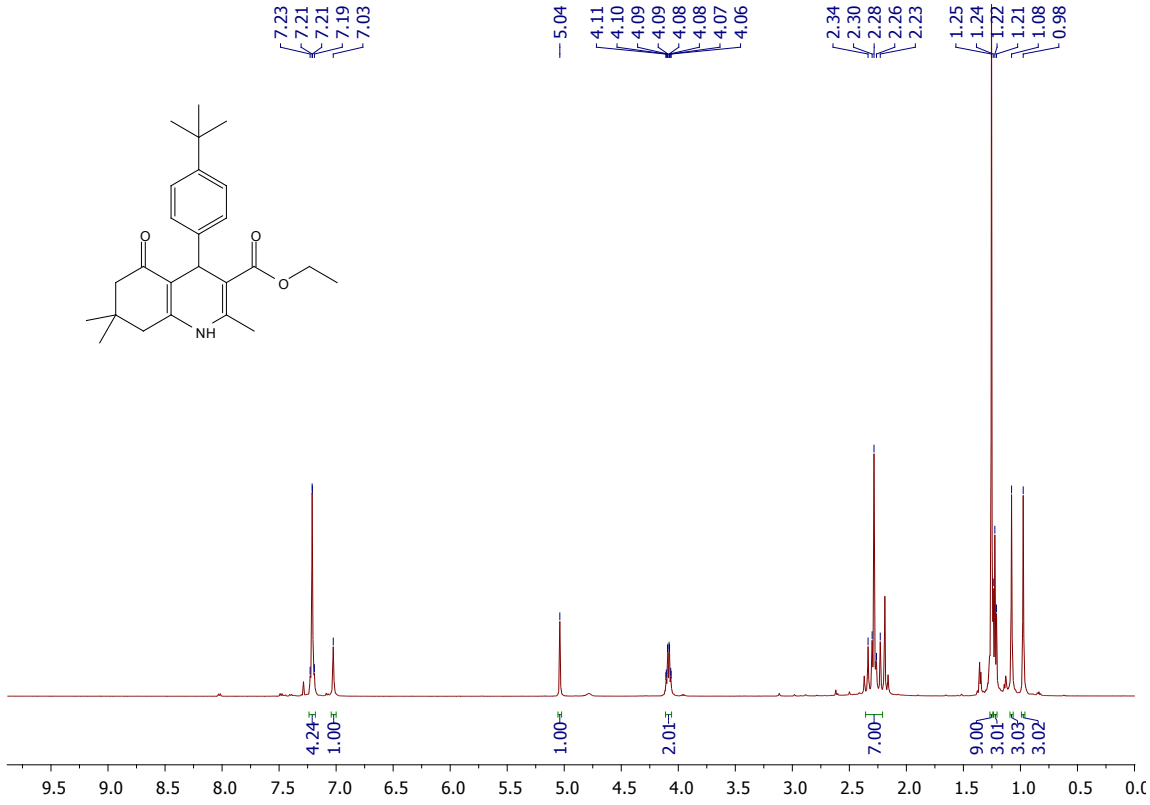


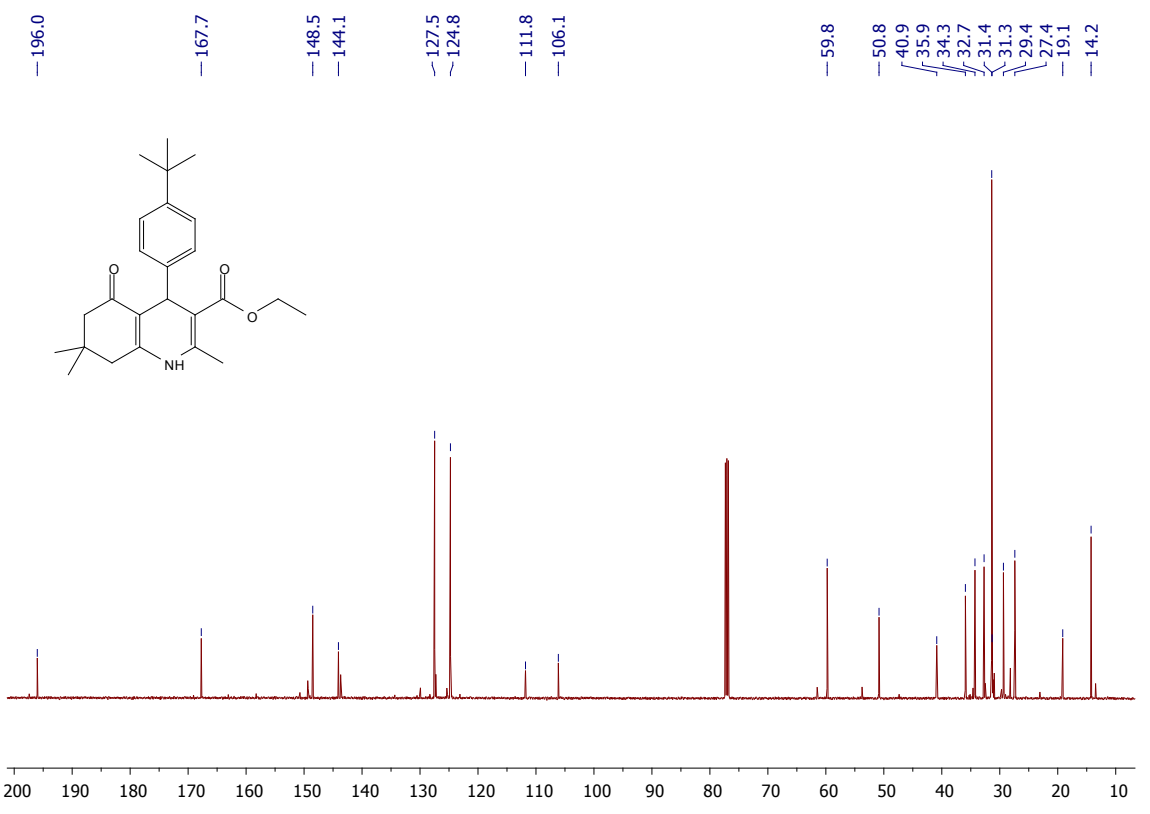
^{1}H and ^{13}C NMR spectroscopy of Ethyl 2,7,7-trimethyl-5-oxo-4-(*p*-tolyl)-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (22)



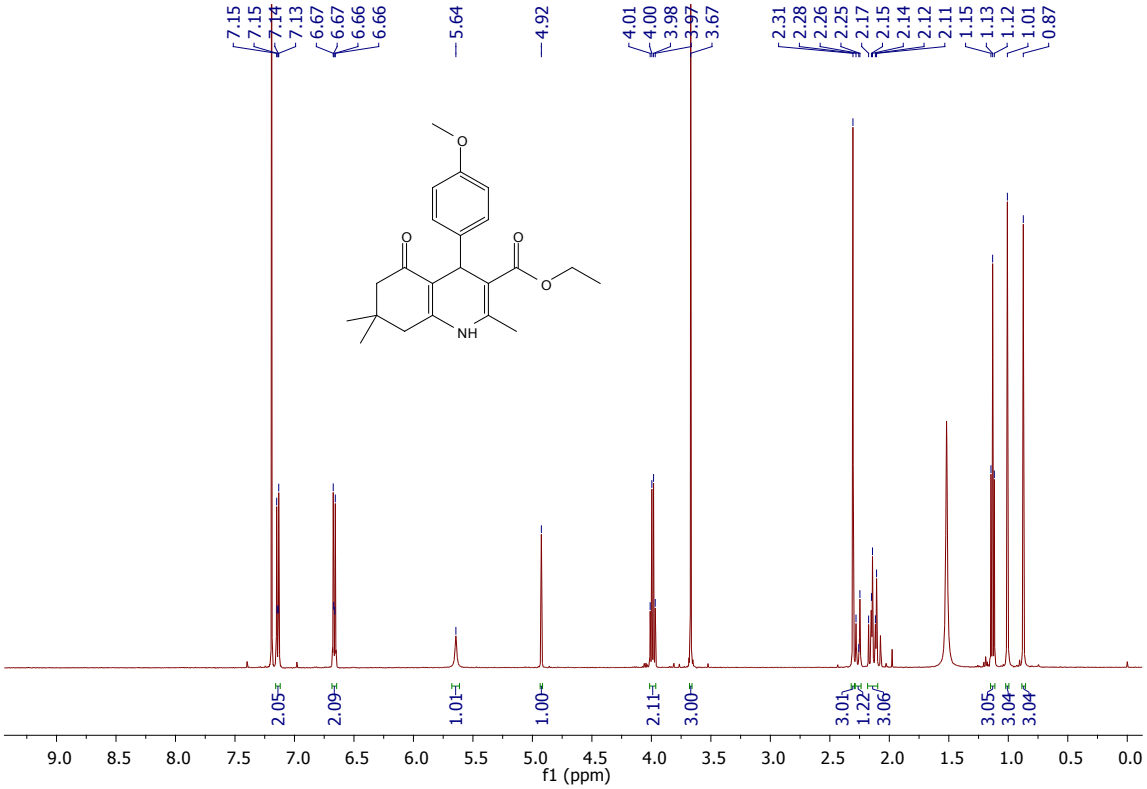


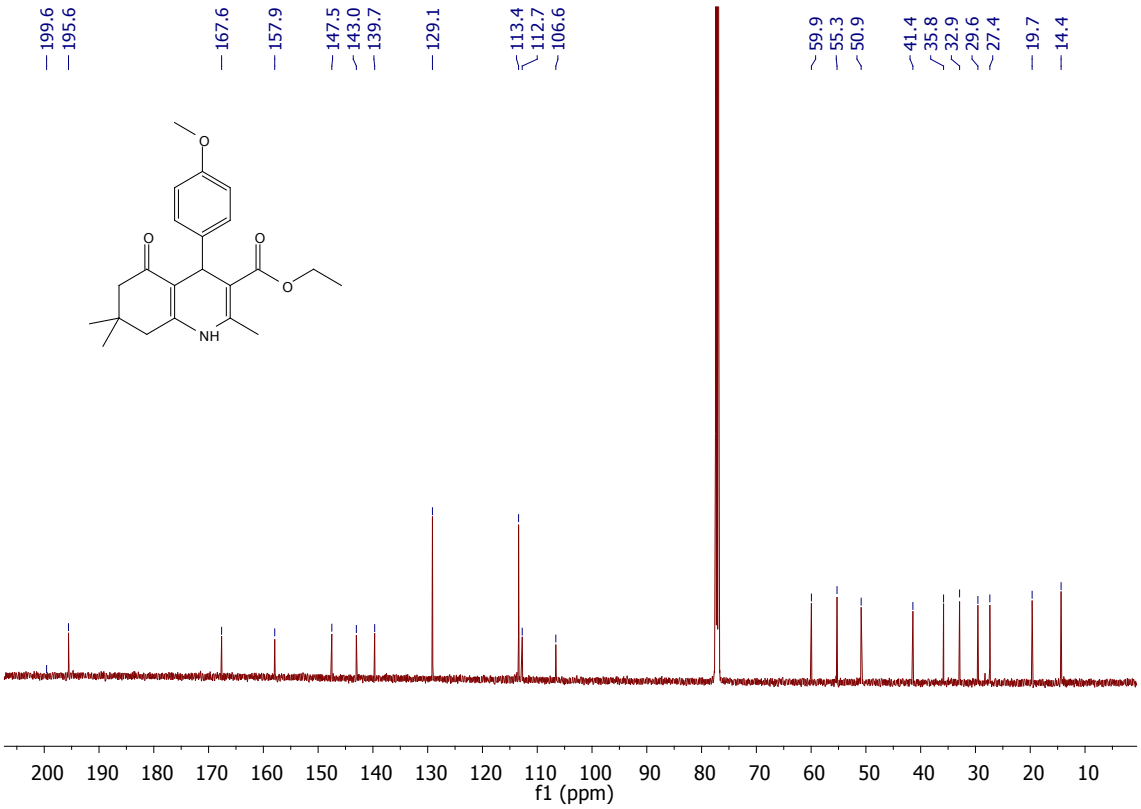
^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(4-(*tert*-butyl)phenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (23)



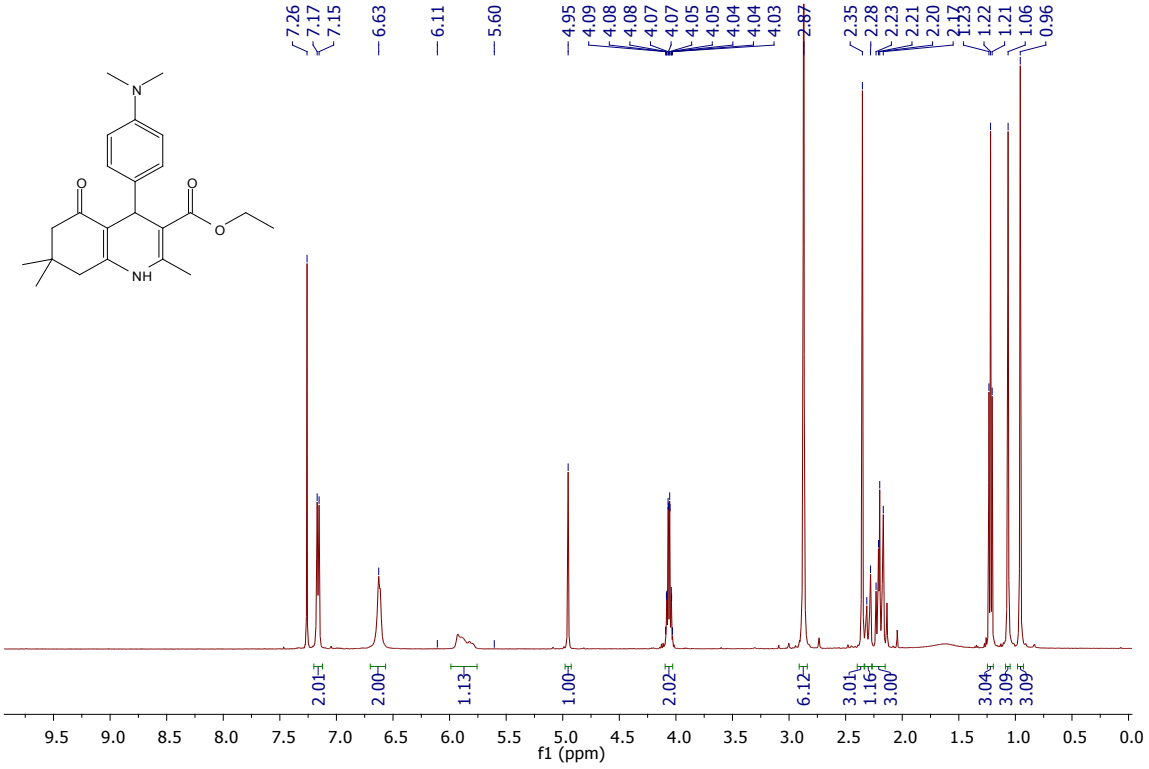


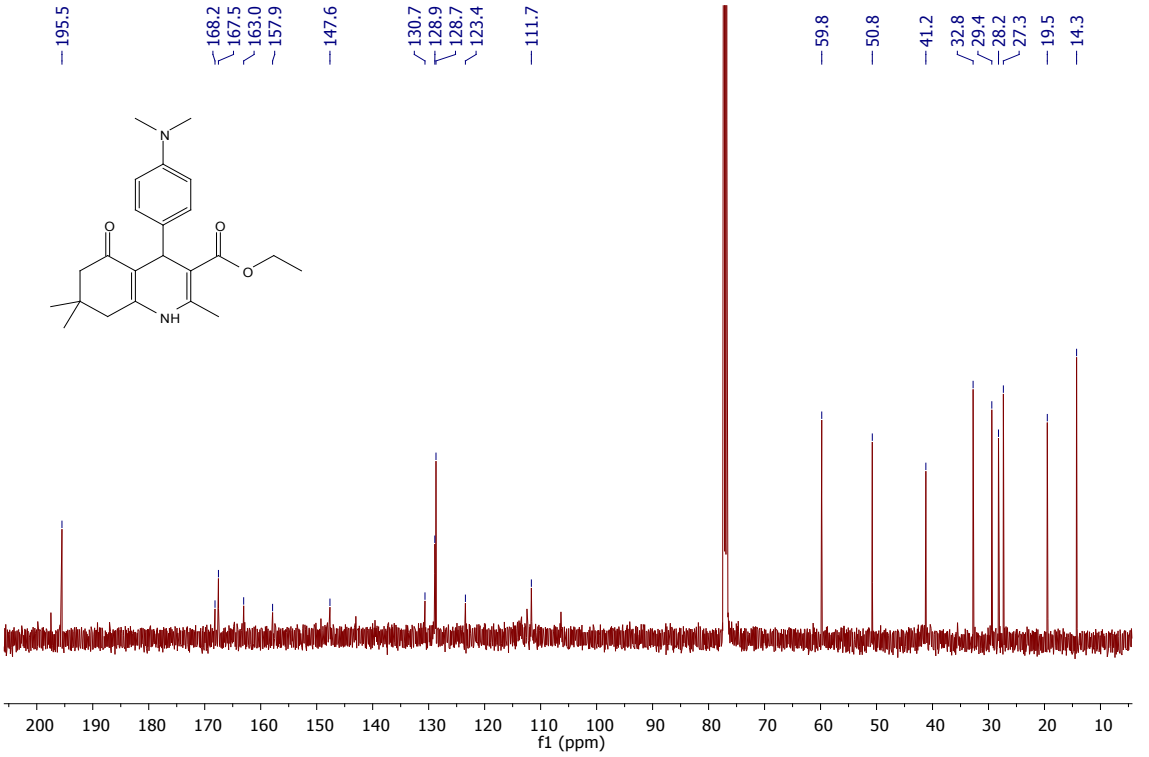
^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(4-methoxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (24)



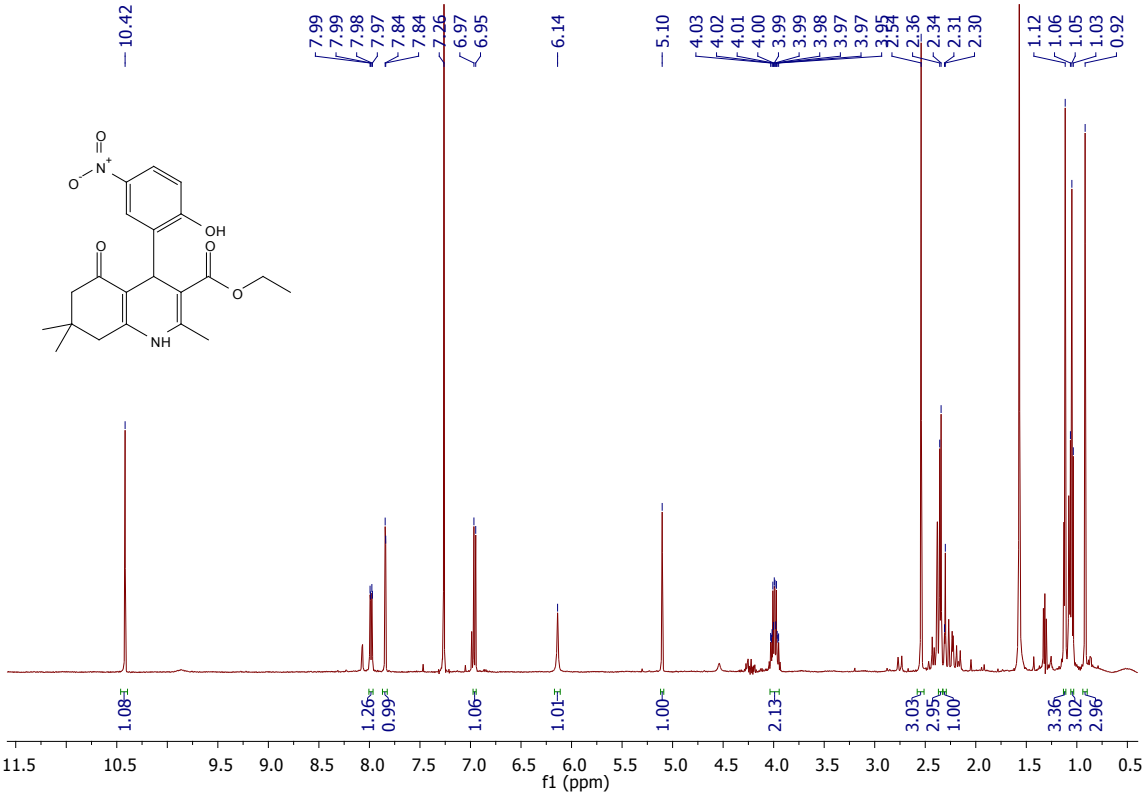


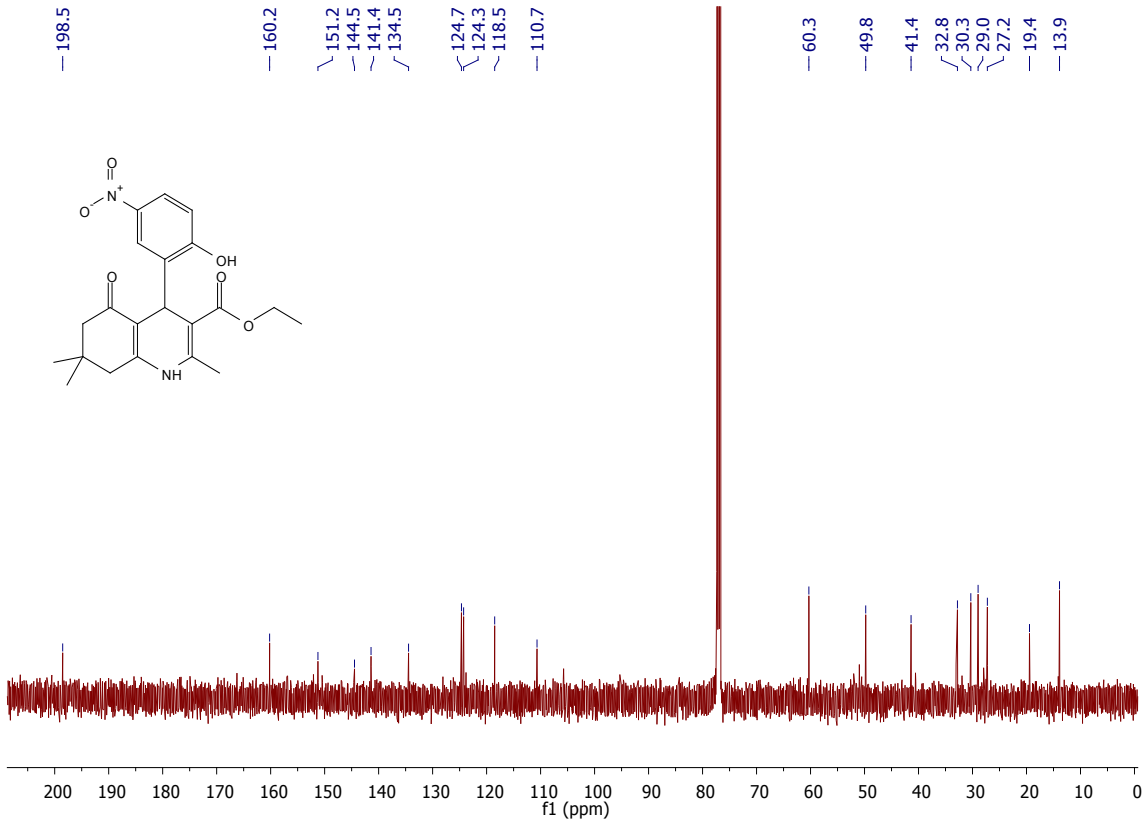
^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(4-dimethylamino)phenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexayidroquinolin-3-carboxylate (25)



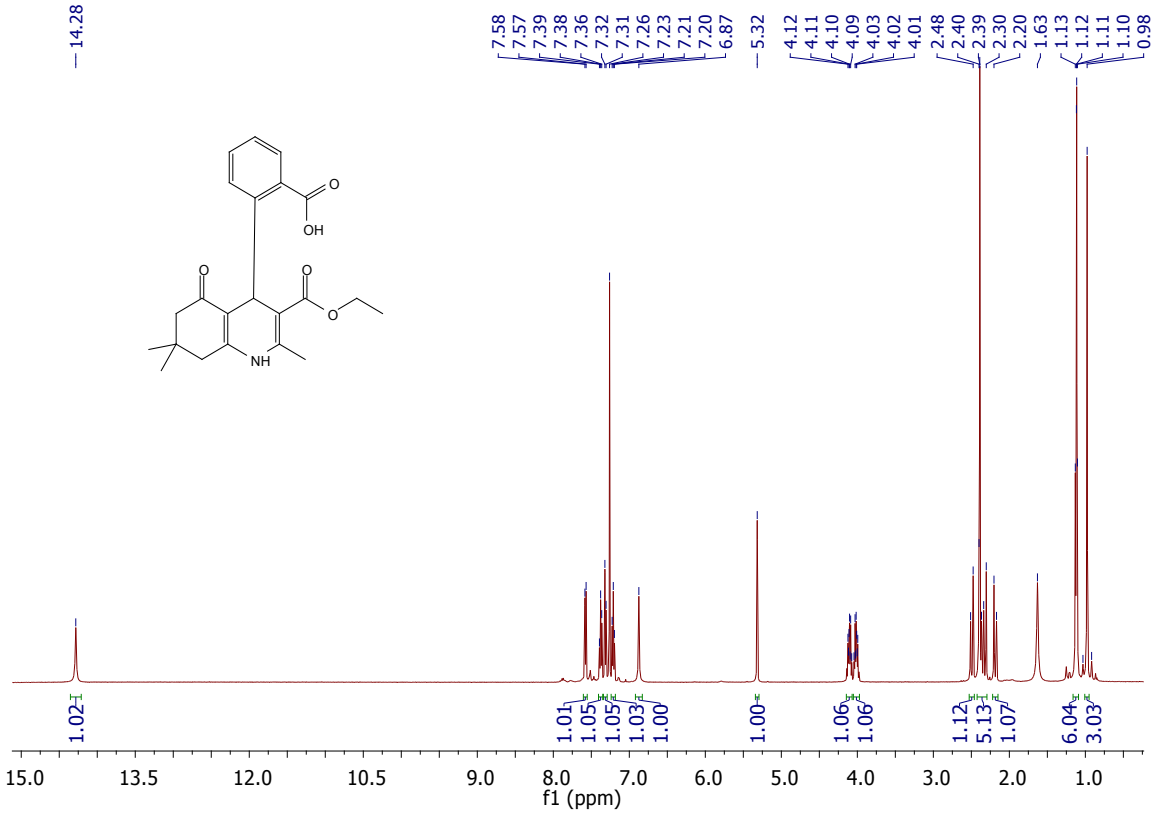


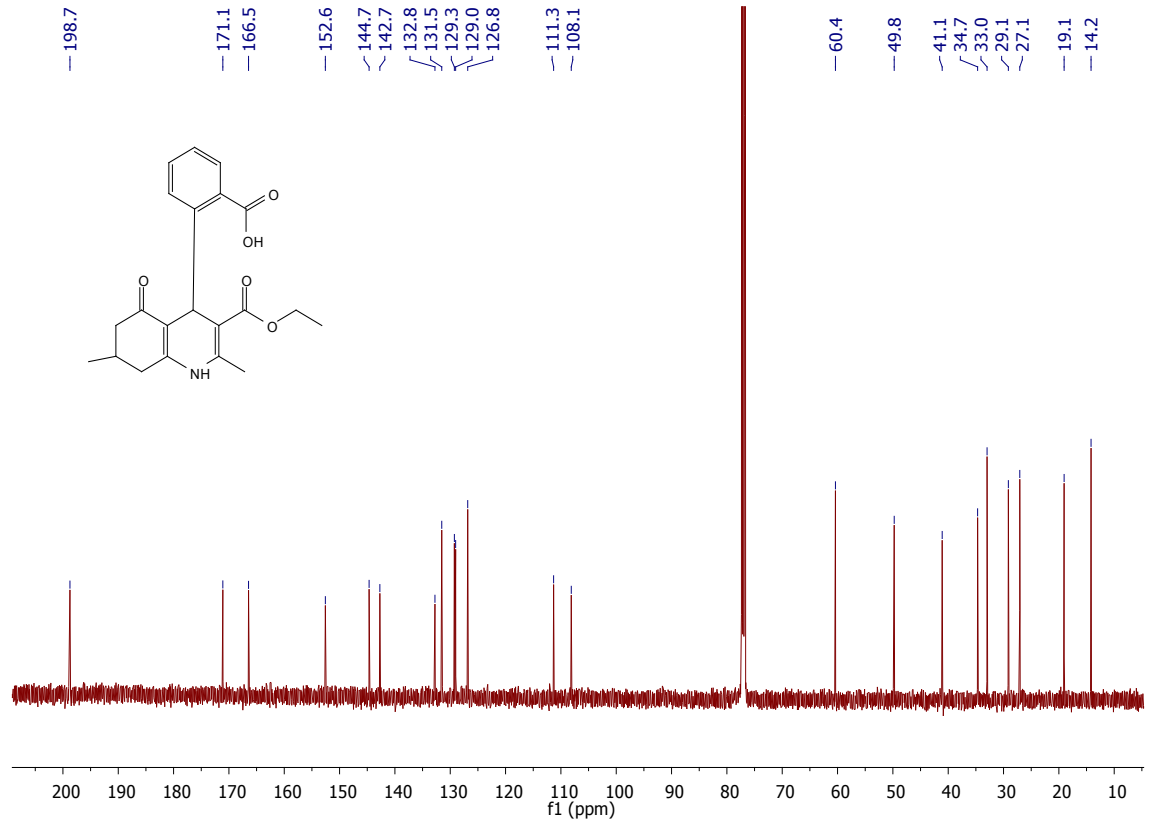
^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(2-hydroxy-5-nitrophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (26)



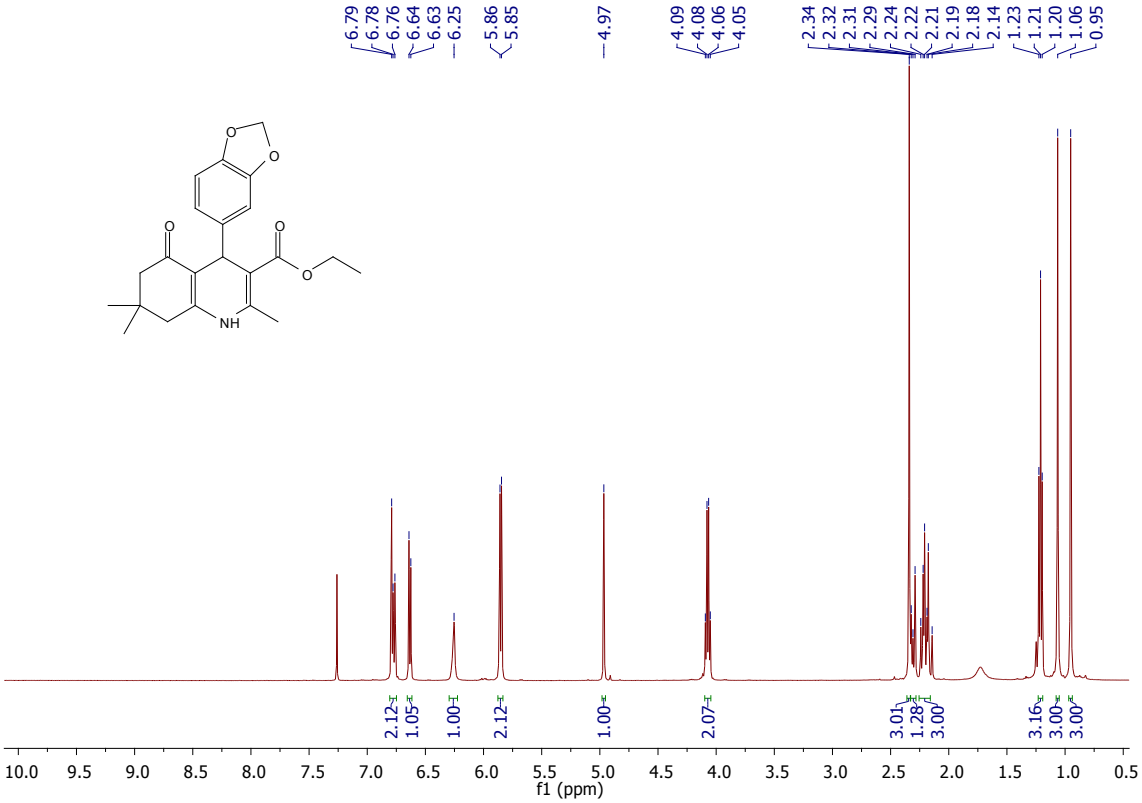


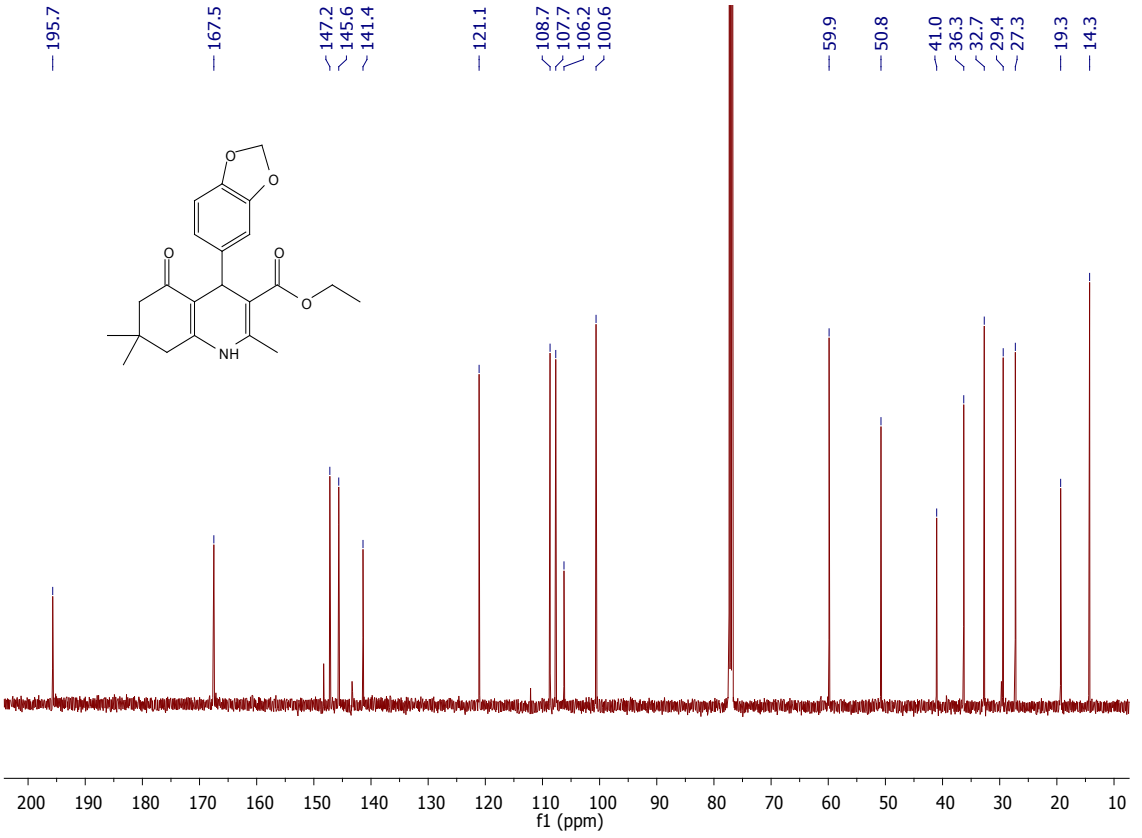
^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(2-carboxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (27)



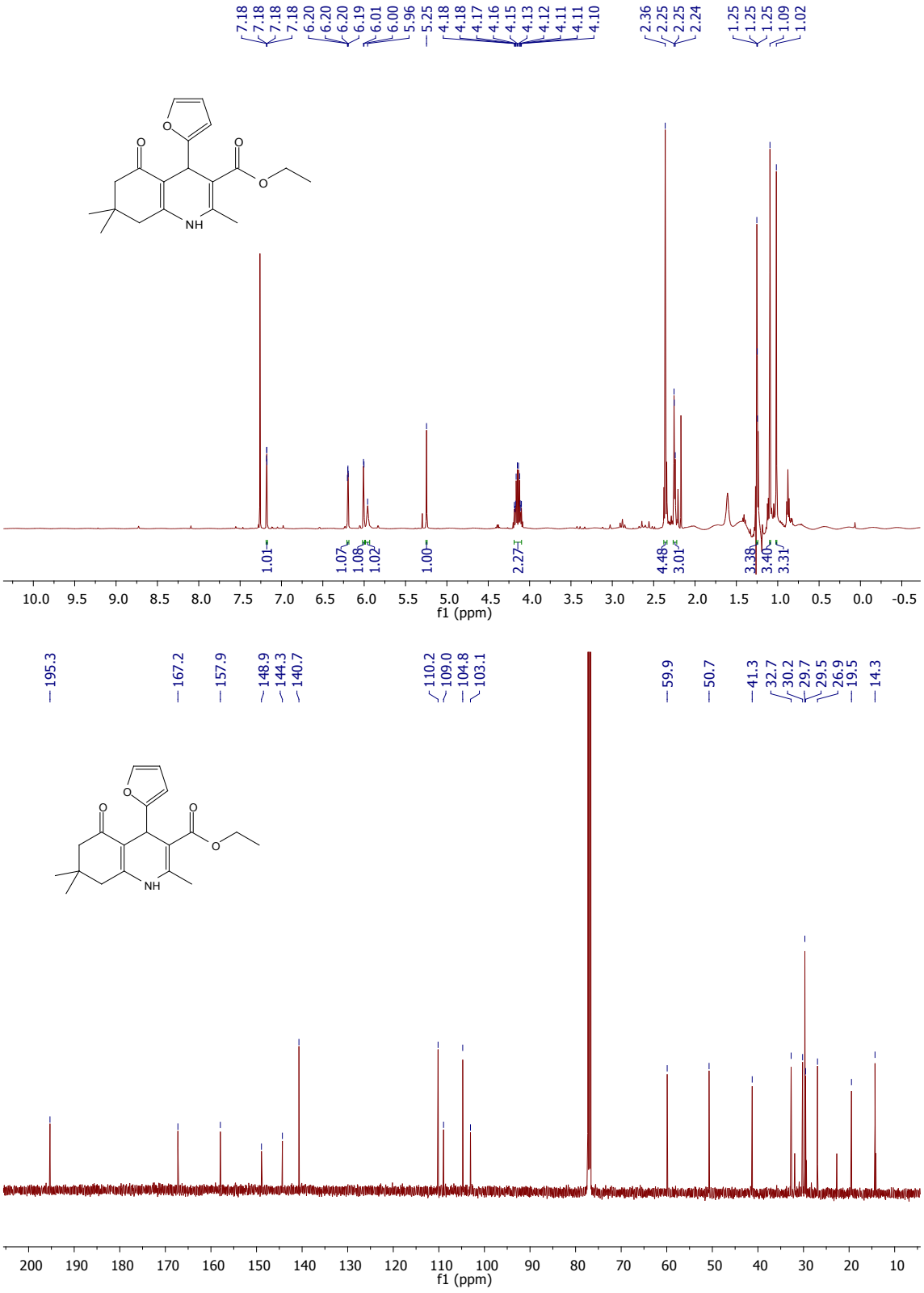


^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(benzo[*d*][1,3]dioxol-5-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (28)

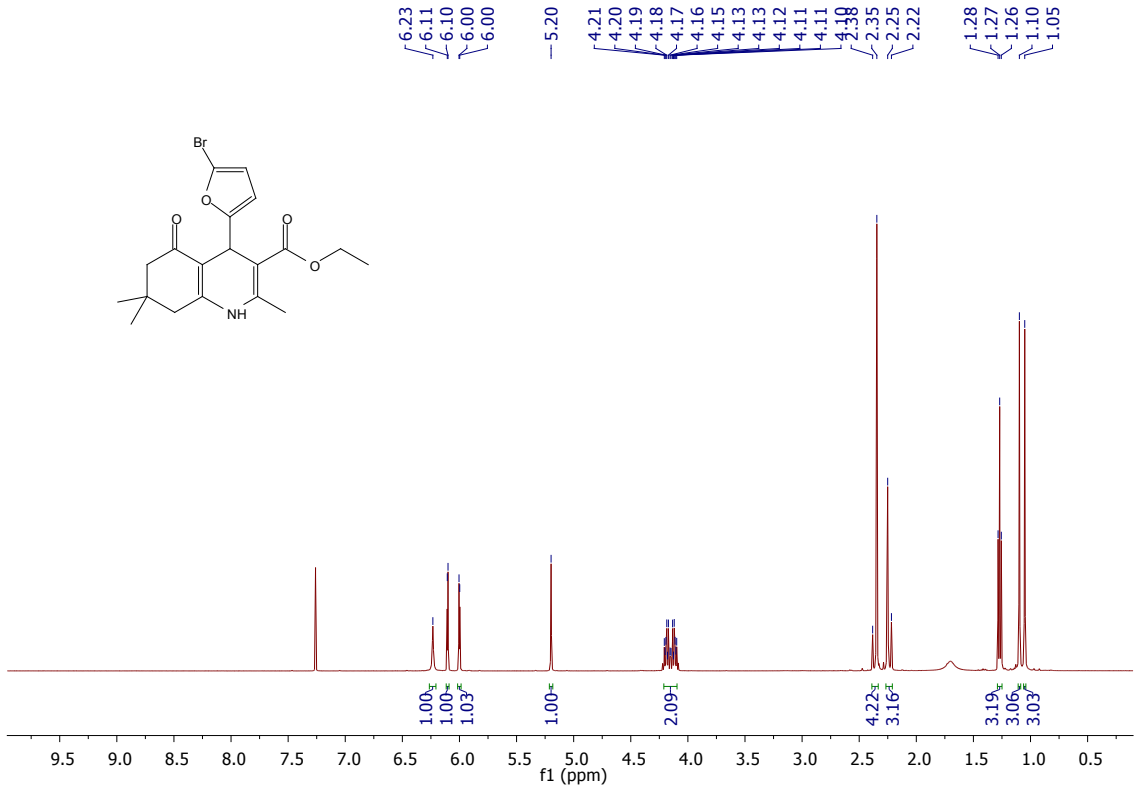


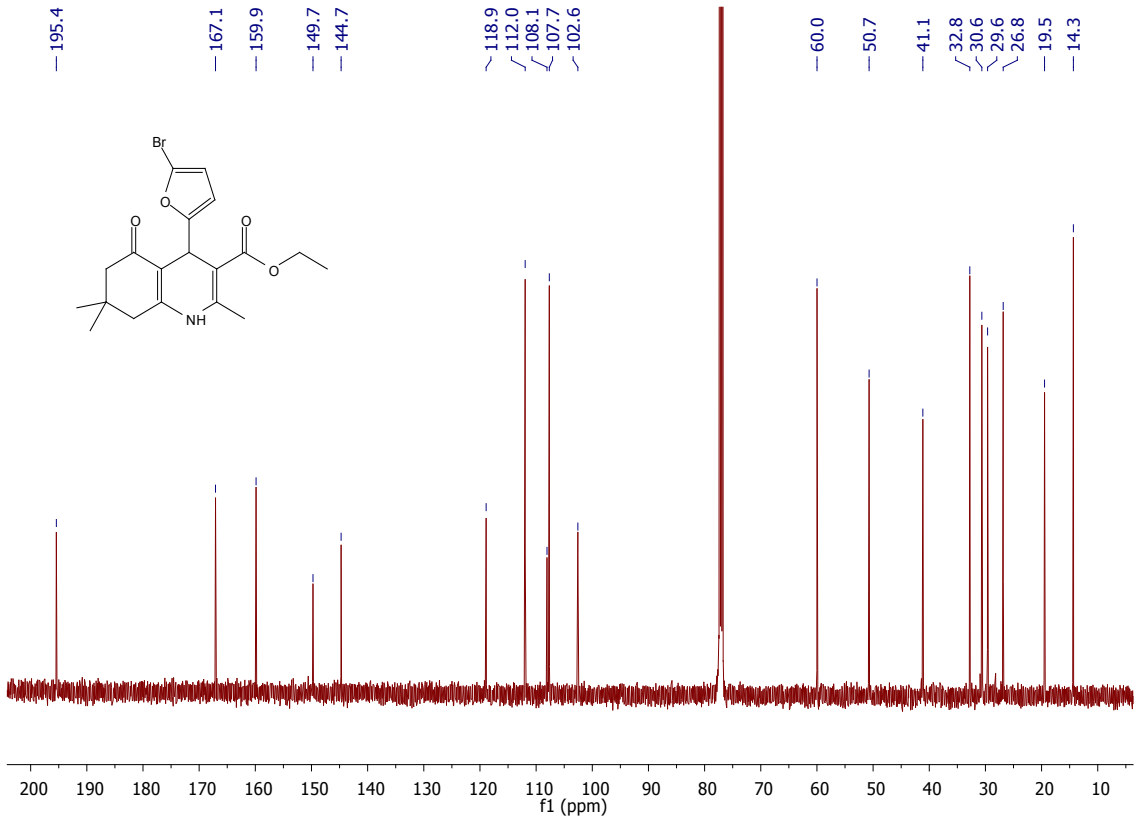


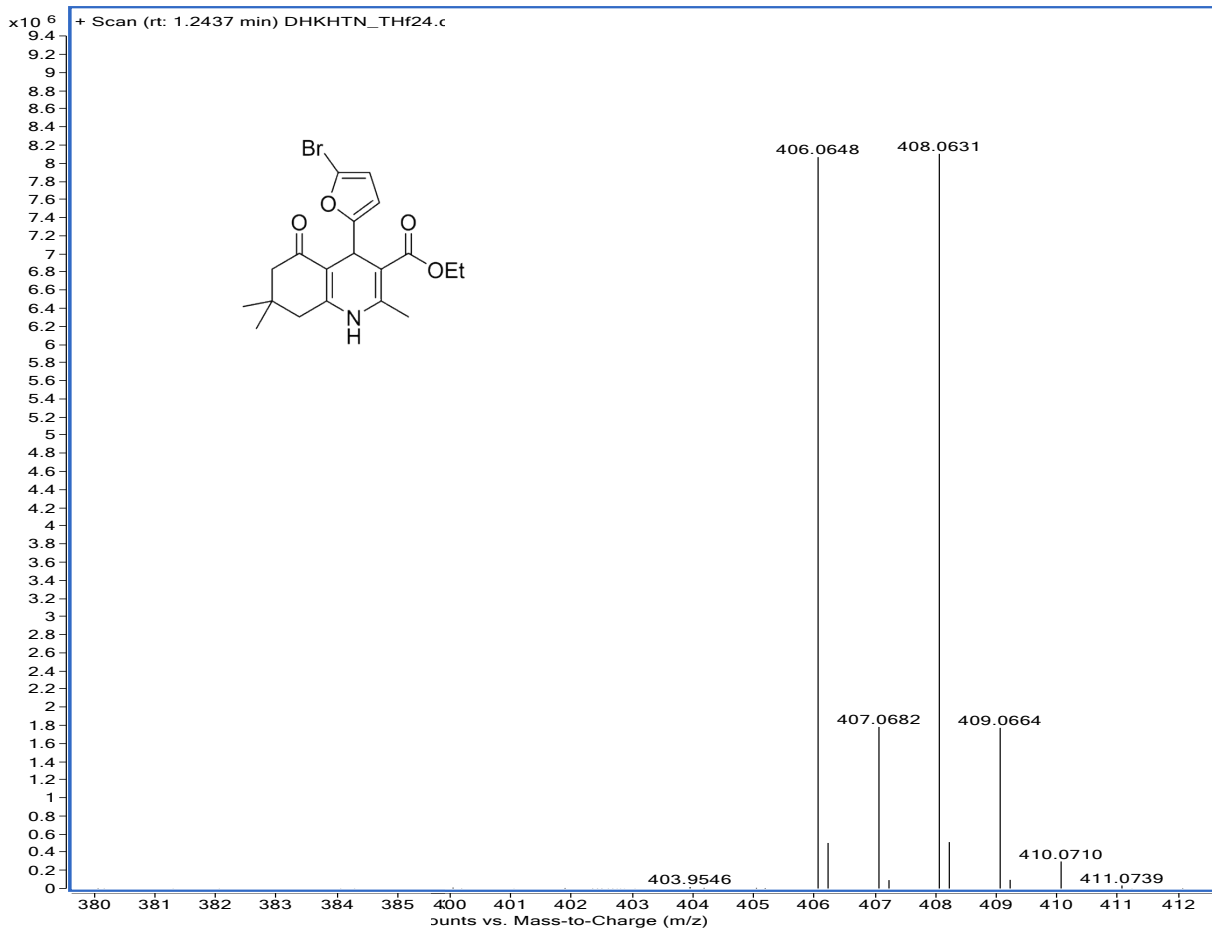
^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(furan-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (29)



^{1}H and ^{13}C NMR spectroscopy and HRMS of Ethyl 4-(5-bromofuran-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (30)






+ Scan (rt: 1.2437 min) DHKHTN_THf24.c
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406.0648
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409.0664
410.0710
411.0739
403.9546
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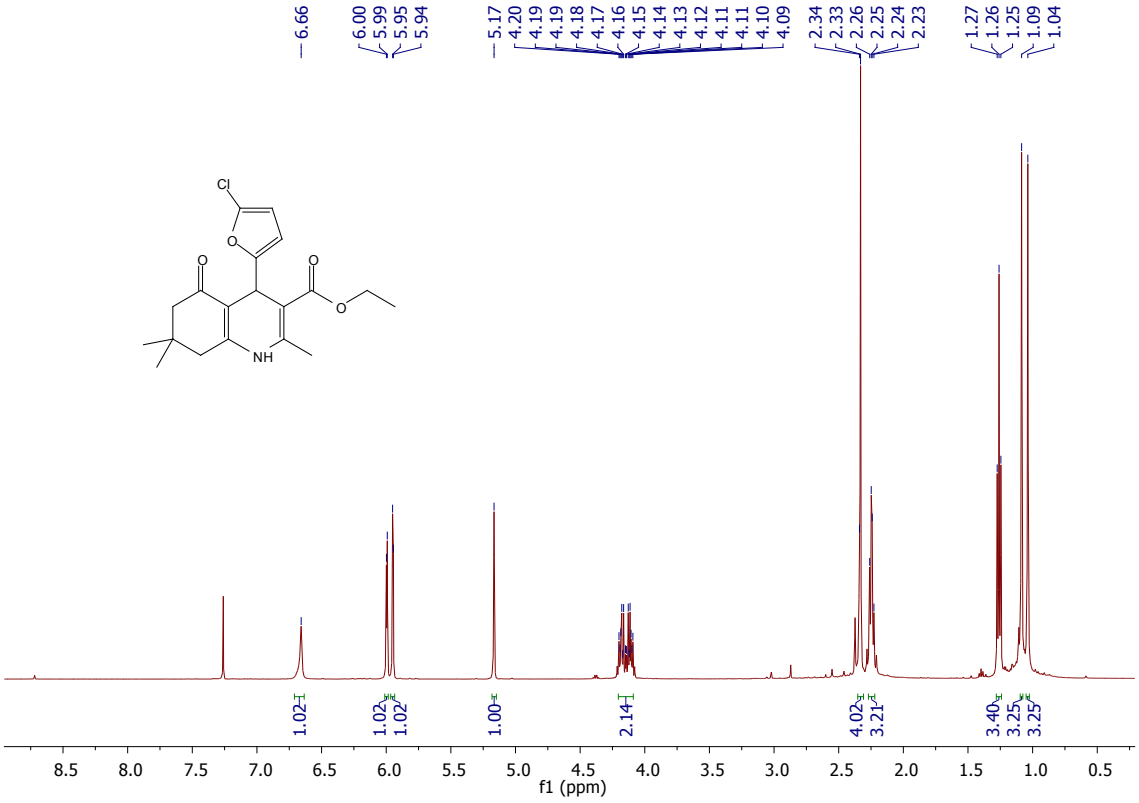
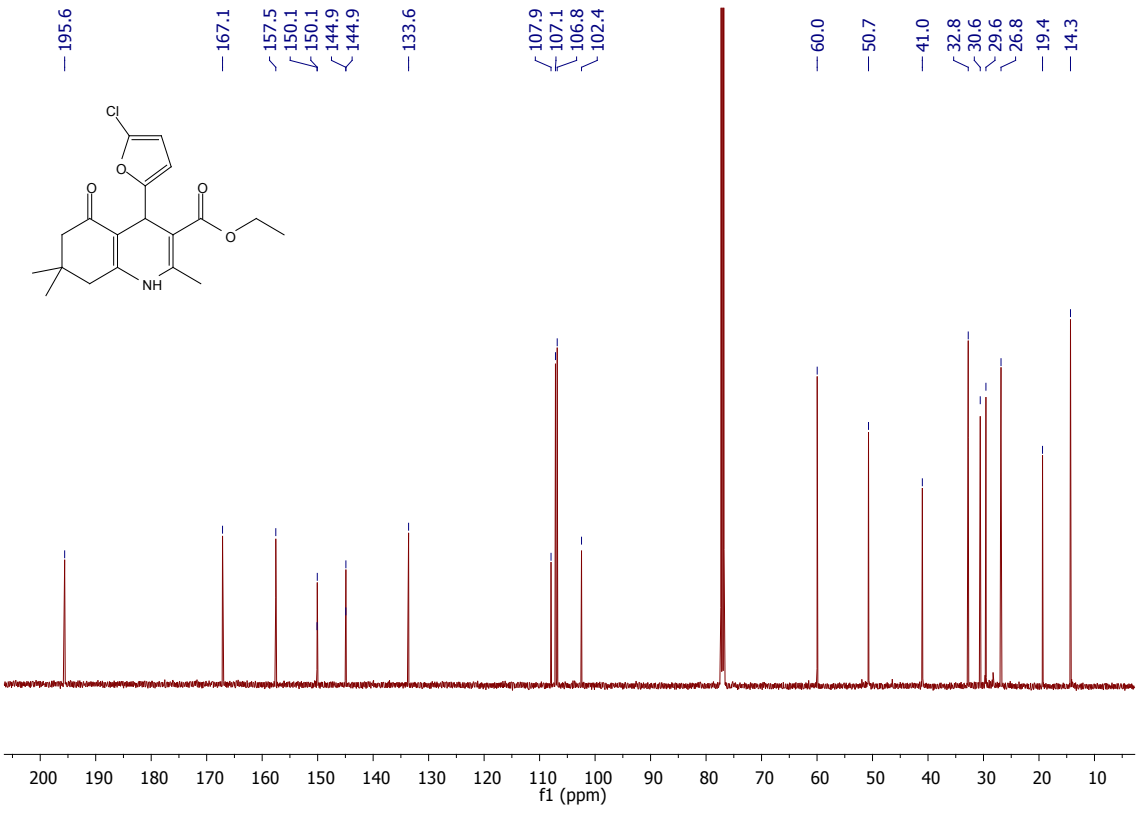
ounts vs. Mass-to-Charge (m/z)

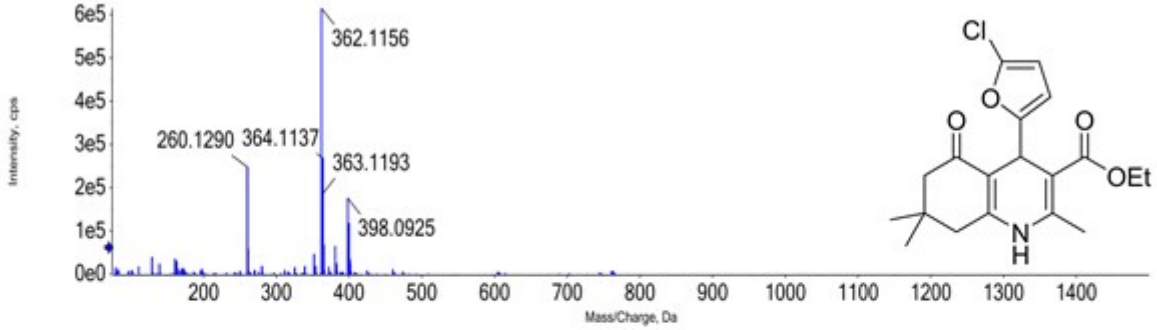
^{1}H and ^{13}C NMR spectroscopy and HRMS of Ethyl 4-(5-chlorofuran-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (31)





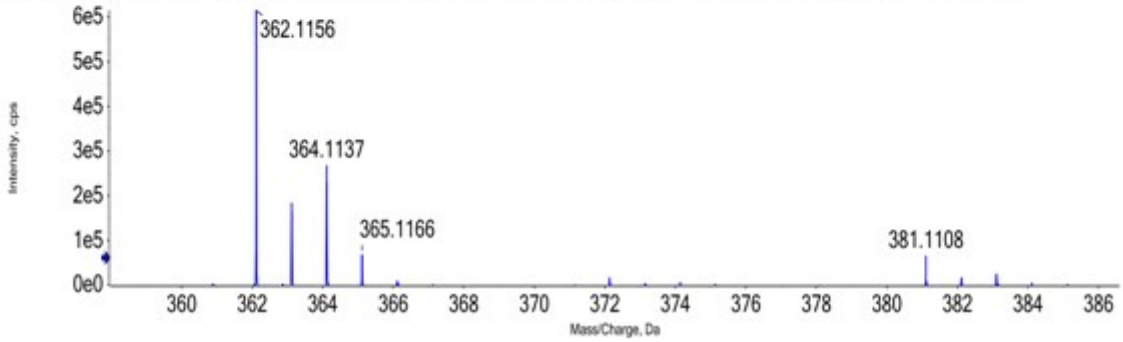
Full mass spectrum

Spectrum from FLU-CL-ESI.wiff2 (sample 1) - FU-CL-ESI, -TOF MS (75 - 1500) from 0.199 min, noise filtered (noise multiplier = 1.5), Gaussian smoothed (0.5 points)

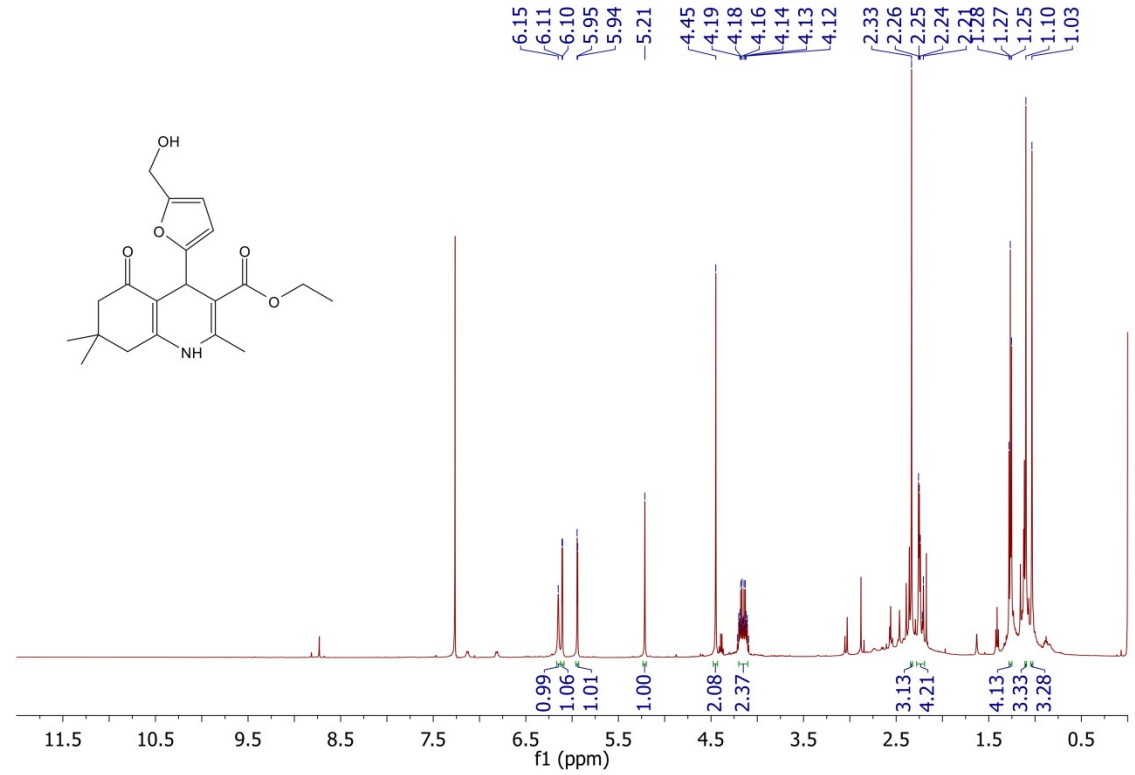


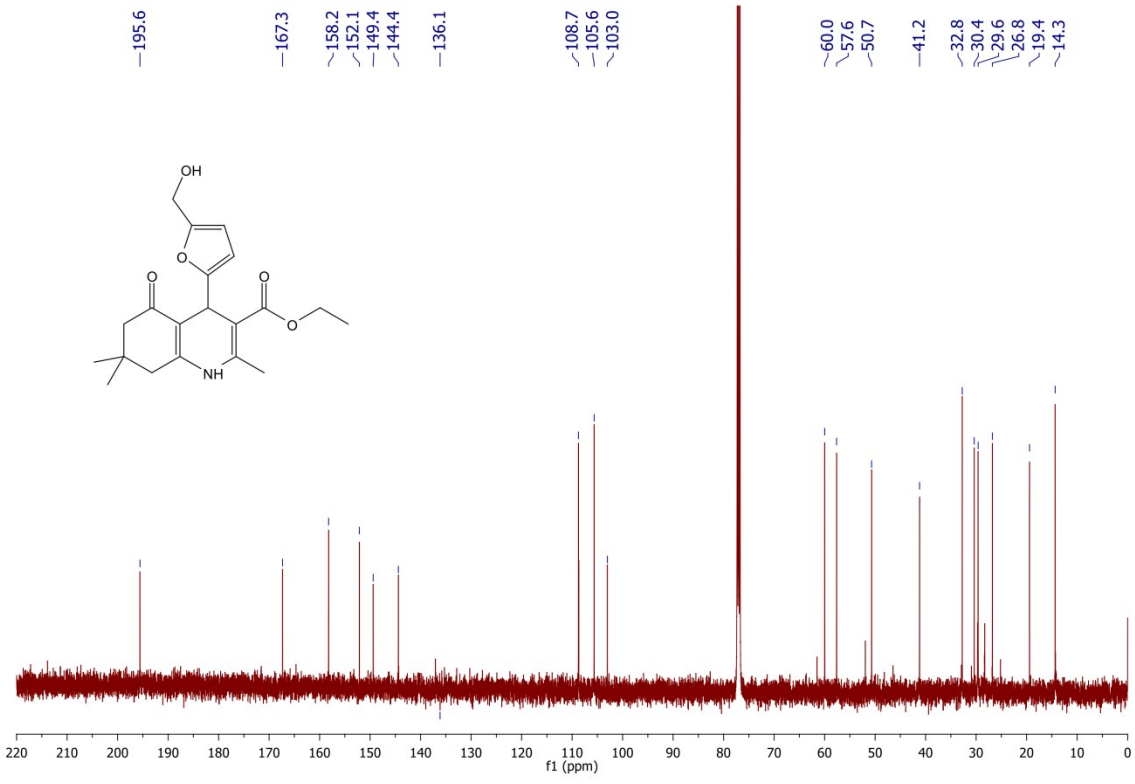
Expanded spectrum

Spectrum from FLU-CL-ESI.wiff2 (sample 1) - FU-CL-ESI, -TOF MS (75 - 1500) from 0.199 min, noise filtered (noise multiplier = 1.5), Gaussian smoothed (0.5 points)

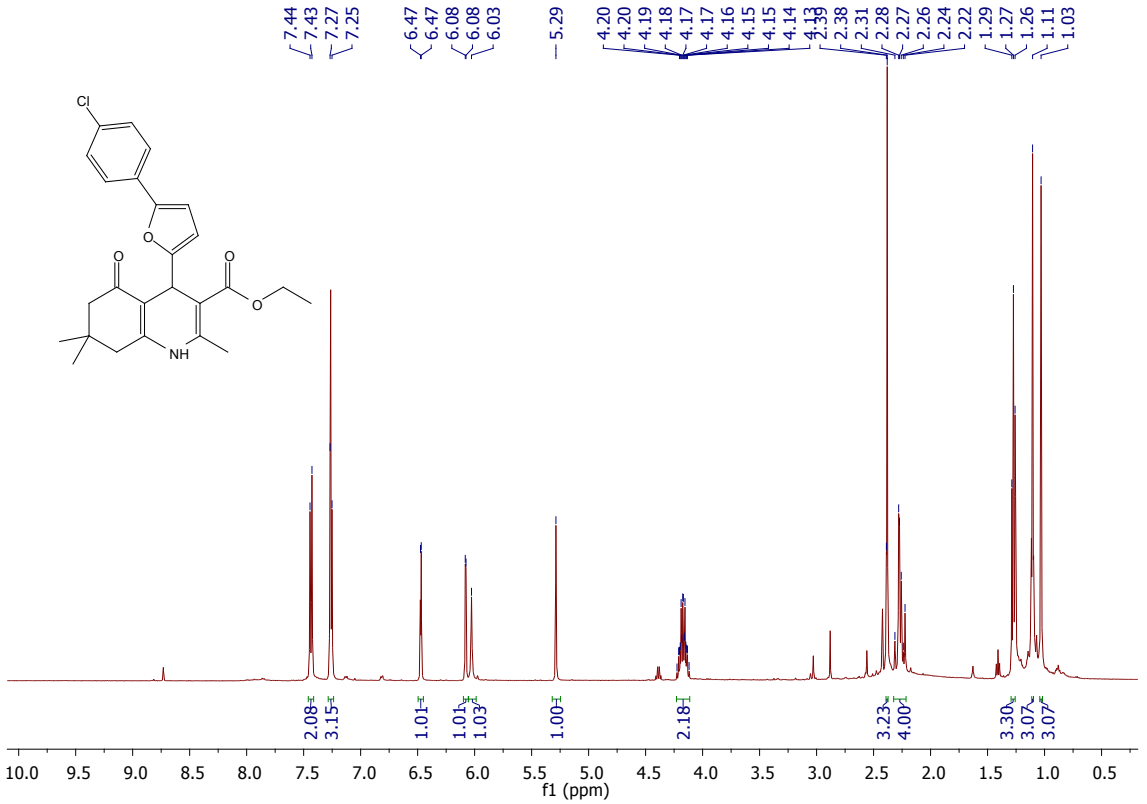


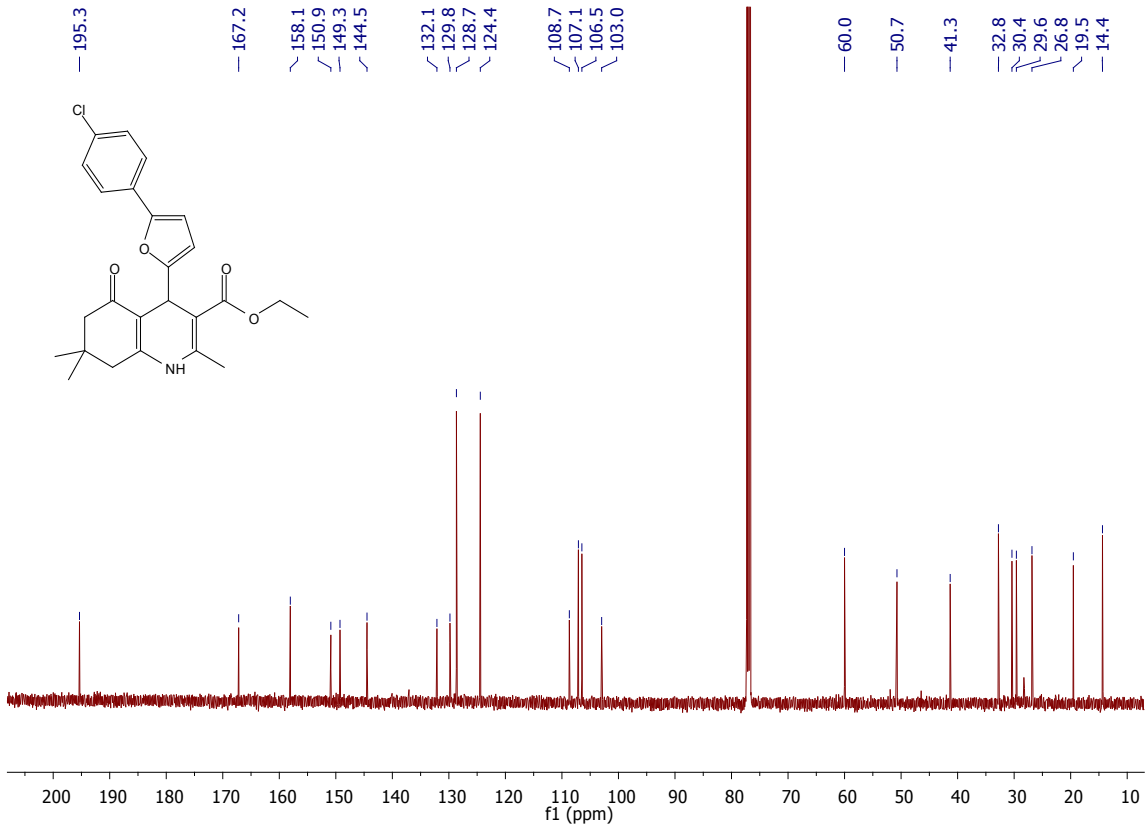
^{1}H and ^{13}C NMR spectroscopy of Ethyl 4-(5-(hydroxymethyl)furan-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (32)





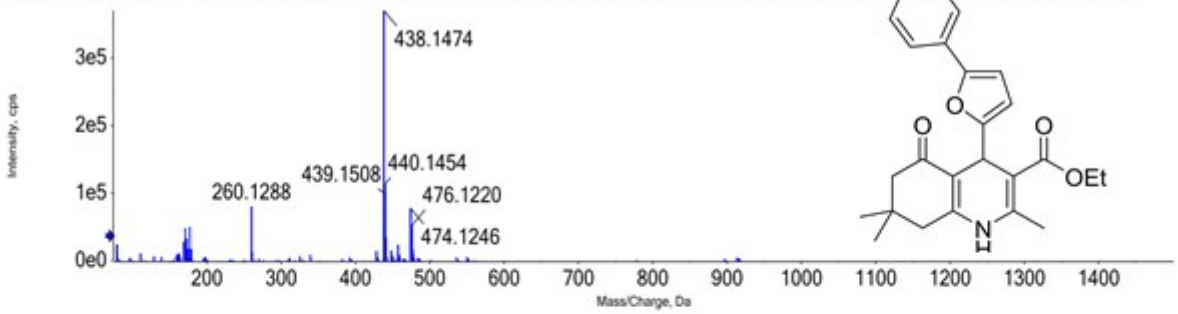
^{1}H and ^{13}C NMR spectroscopy and HRMS of Ethyl 4-(5-(4-chlorophenyl)furan-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (33)





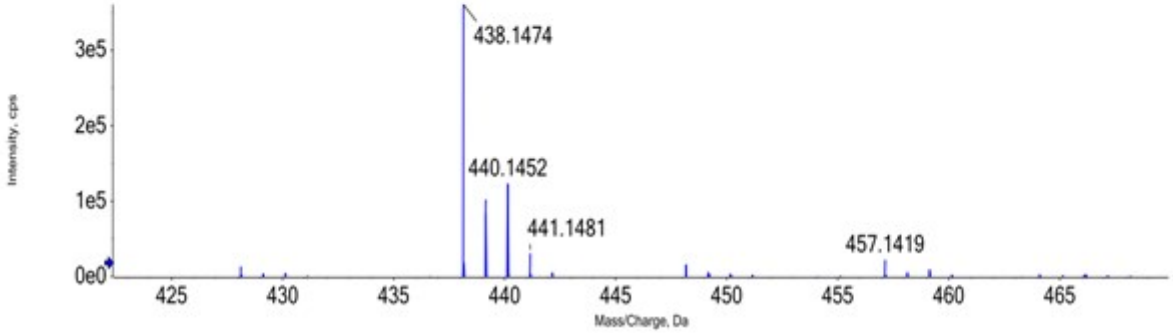
Full mass spectrum

Spectrum from FLU-PHE-ESI.wiff2 (sample 1) - FU-PHE-ESI, -TOF MS (75 - 1500) from 0.125 min, noise filtered (noise multiplier = 1.5), Gaussian smoothed (0.5 points)

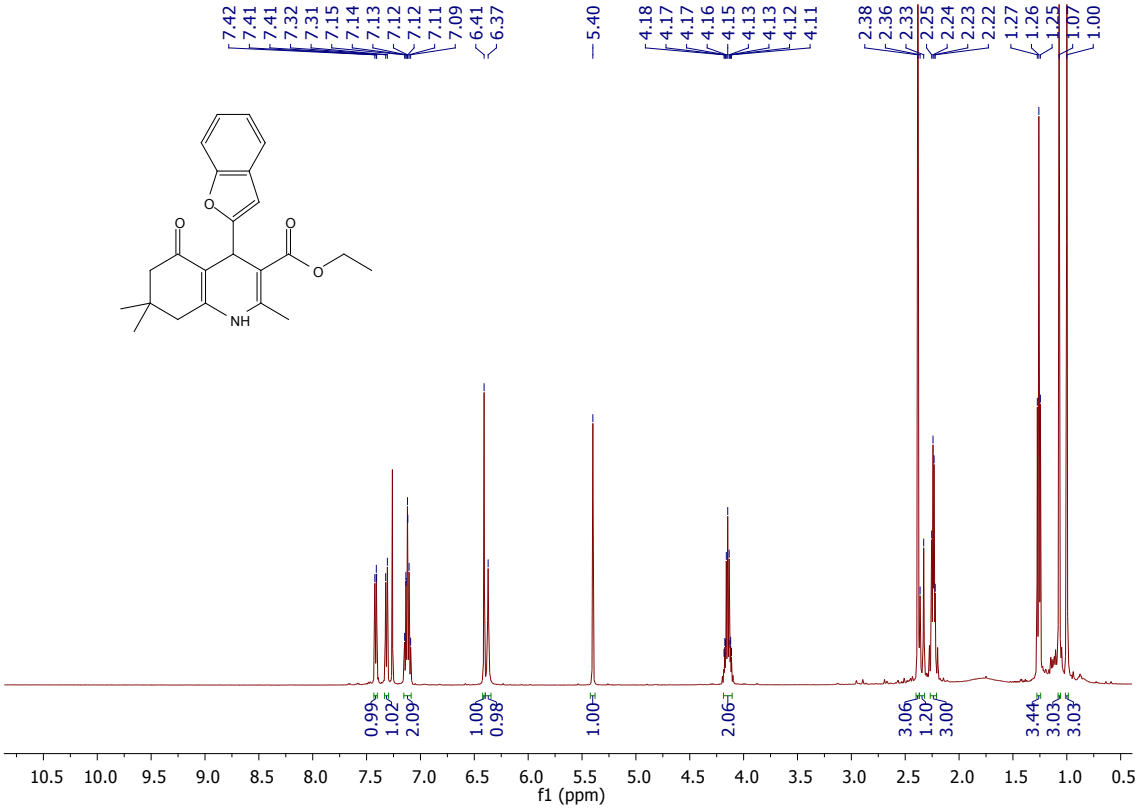


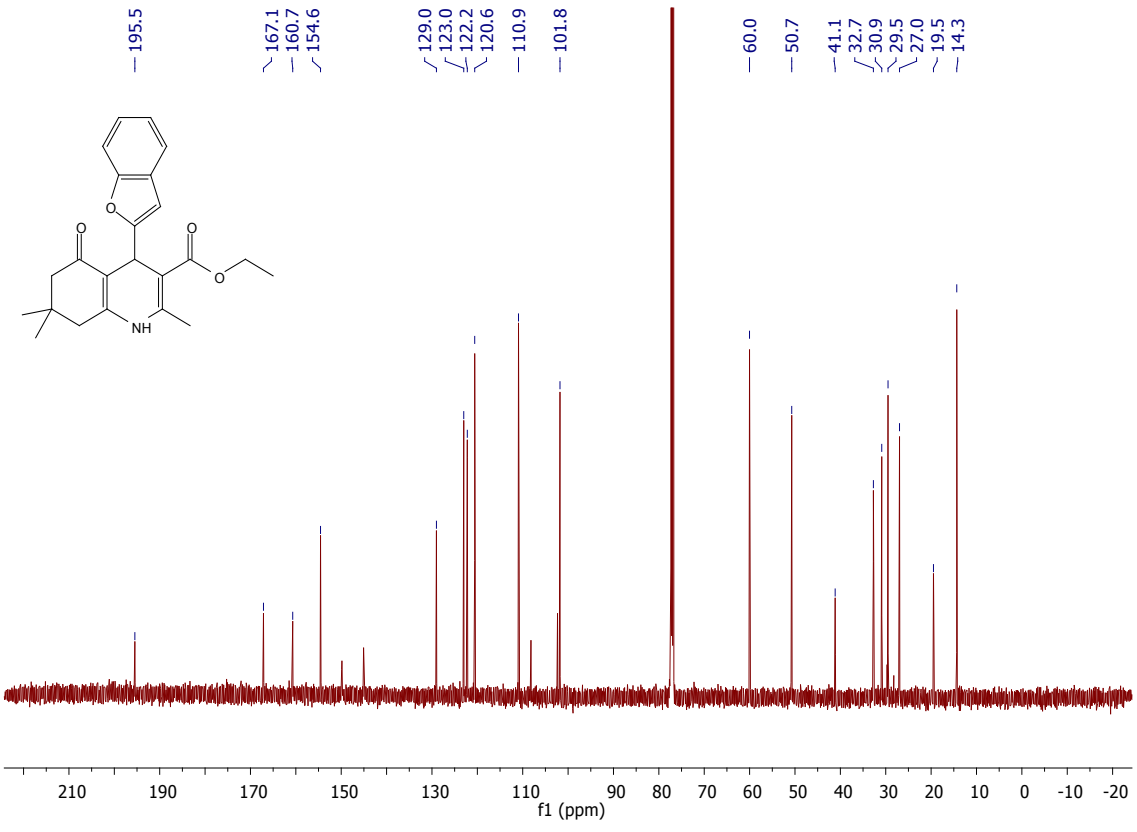
Expanded spectrum

Spectrum from FLU-PHE-ESI.wiff2 (sample 1) - FU-PHE-ESI, -TOF MS (75 - 1500) from 0.120 min, noise filtered (noise multiplier = 1.5), Gaussian smoothed (0.5 points)



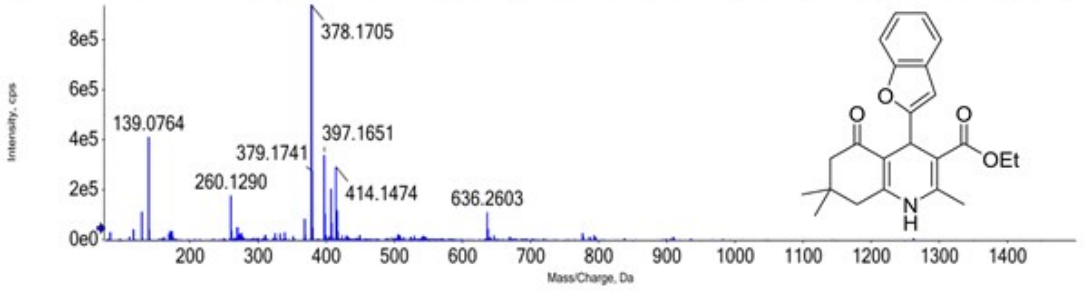
^{1}H and ^{13}C NMR spectroscopy and HRMS of Ethyl 4-(benzofuran-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (34)





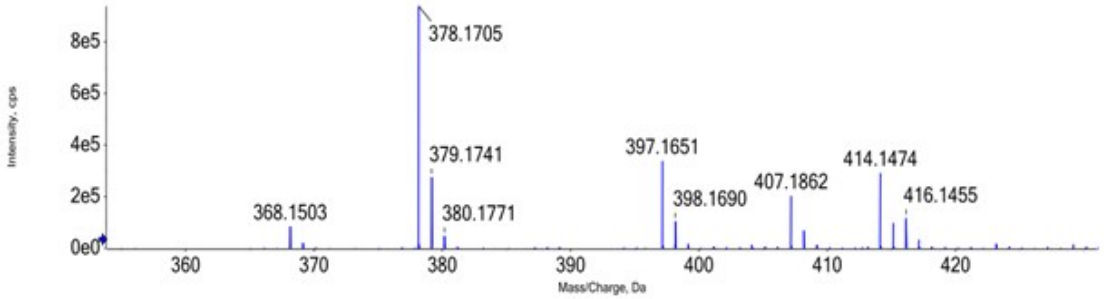
Full mass spectrum

Spectrum from BENZO-POLY-ESI.wiff2 (sample 1) - BENZO-POLY-ESI, -TOF MS (75 - 1500....153 min, noise filtered (noise multiplier = 1.5), Gaussian smoothed (0.5 points)



Expanded spectrum

Spectrum from BENZO-POLY-ESI.wiff2 (sample 1) - BENZO-POLY-ESI, -TOF MS (75 - 1500....153 min, noise filtered (noise multiplier = 1.5), Gaussian smoothed (0.5 points)



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