Supporting information

Synthesis of polyhydroquinolines and propargylamines through one-pot multicomponent reactions using an acidic ionic liquid-immobilized onto magnetic Fe_3O_4 as an efficient heterogeneous catalyst under solvent-free sonication

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Section S1. Chemicals, supplies, and instruments

Chemicals and supplies

Imidazole (grade ReagentPlus®, assay 99%), tetraethyl orthosilicate (assay ≥99.5% (GC)), 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%), piperidine (grade ReagentPlus®, assay 99%), (3-chloropropyl)trimethoxysilane (assay 97%) were obtained from Sigma-Aldrich. 5,5-Dimethyl-1,3-cyclohexanedione (dimedone) (99%) was obtained from Across. Iron(III) chloride hexahydrate ($FeCl_3.6H_2O$) (for analysis EMSURE® ACS,Reag. Ph Eur), Iron(II) chloride tetrahydrate ($FeCl_2.4H_2O$) (for analysis EMSURE® ACS,Reag. Ph Eur), Potassium hydroxide (KOH) (pellets for analysis EMSURE®), ethanol (absolute for analysis EMSURE® ACS,ISO,Reag. Ph Eur), 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), ammonium acetate (EMPLURA®), phenylacetylene (for synthesis), 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 ^1H and ^{13}C NMR spectra were recorded on a Bruker Advance 500 instruments 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-assisted reactions 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. Synthesis and Characterization of LAIL@NMP

General procedure of magnetic Fe_3O_4 nanoparticles (MNPs)[1]

A co-precipitation method was used for the preparation of nano-Fe_3O_4. In a typical procedure $FeCl_3.6H_2O$ (20 mmol) and $FeSO_4.7H_2O$ (10 mmol) were dissolved in 100 mL deionized water. The mixture was stirred at 80 ^oC and then KOH (12 mmol) was added and stirred continuously within 2 h. The black precipitate, after being collected by a permanent magnet, was washed with water (3 x 100 mL) and ethanol (3 x 50 mL). This MNP material was dried at 60–70 ^oC under vacuum for 1 h

General procedure for the synthesis of LAIL@MNP[1]

A mixture of 3-chloroethoxypropylsilane (5.0 mmol, 1.2 mL) and imidazole (5.0 mmol, 0.34 g) was stirred at reflux for 17 h. The yellowish viscous liquid was diluted in 50 mL of ethanol-waterer (1:1 volume ratio) solution, and the mixture was sonicated at 40 °C for 4 h. The LAIL@MNP was washed with dichloromethane and dried at 70 °C *in vacuo*. A mixture of LAIL@MNP (1.0 g) and $ZnCl_2$ (1.0 mmol) in ethanol (50 mL) was refluxed for 24 h. After completion, the catalyst was separated by a magnet, washed with ethanol, and dried at 100 °C for 5 h.

Characterization of LAIL@MNP

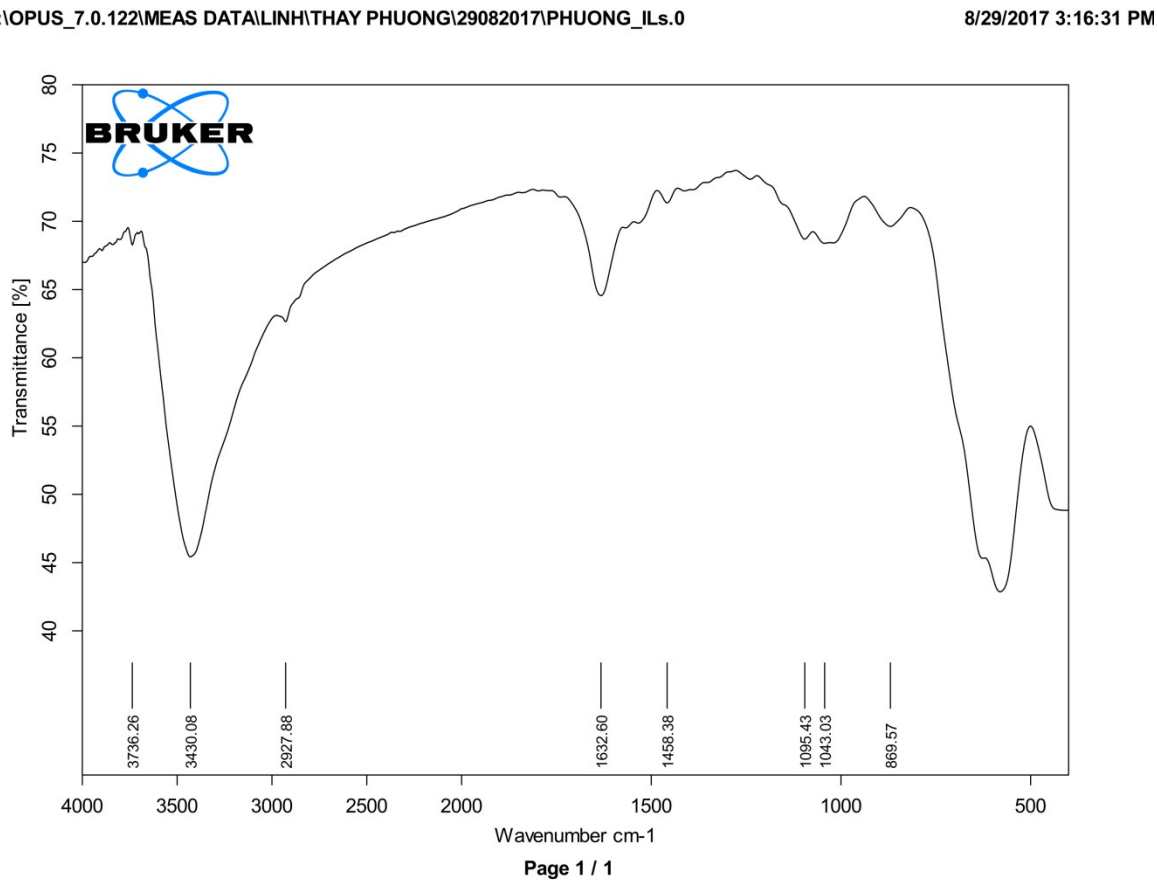
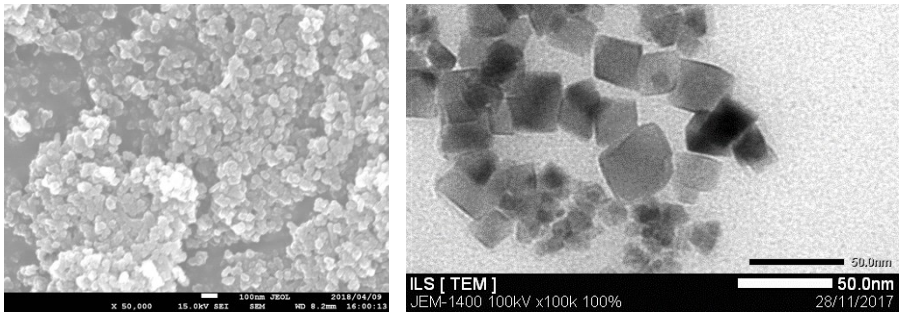


Figure S1. FT-IR spectra of LAIL@MNP



(**a**) (**b**)

Figure S2. SEM image of LAIL@MNP (**a**) and TEM images of as-prepared (**b**)

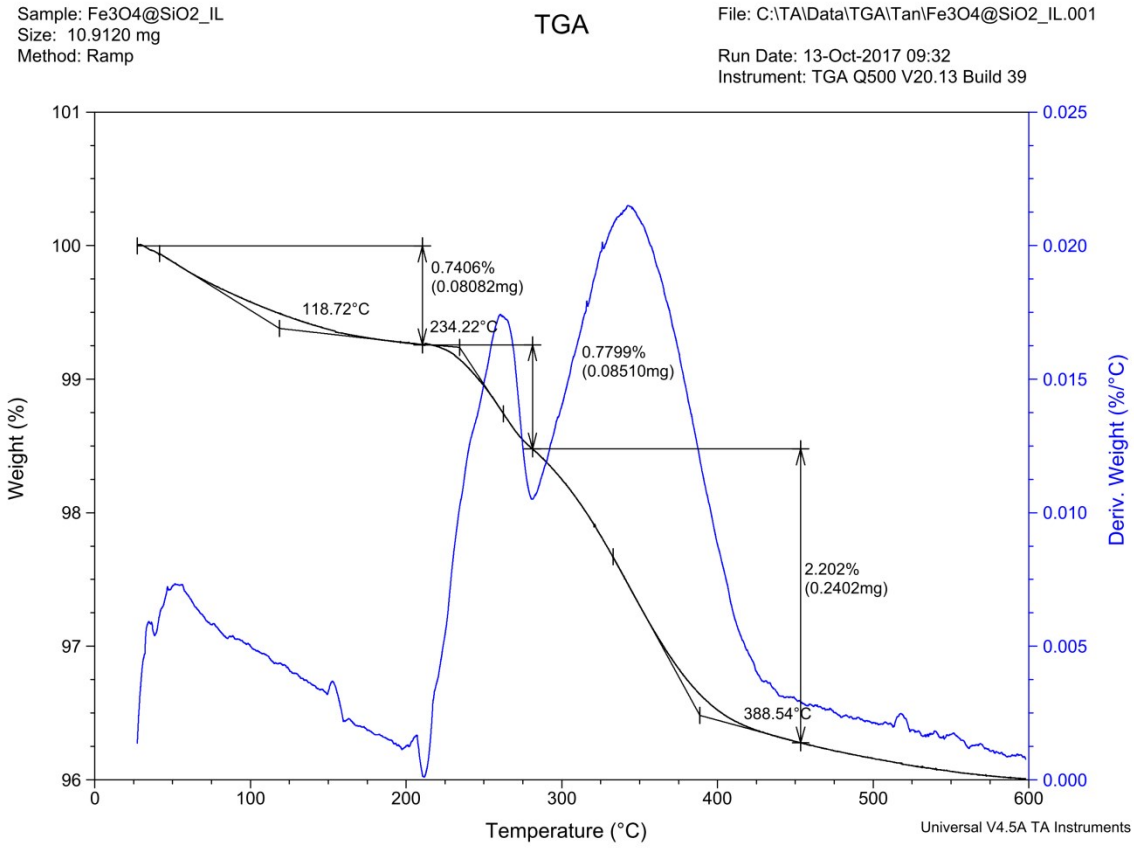
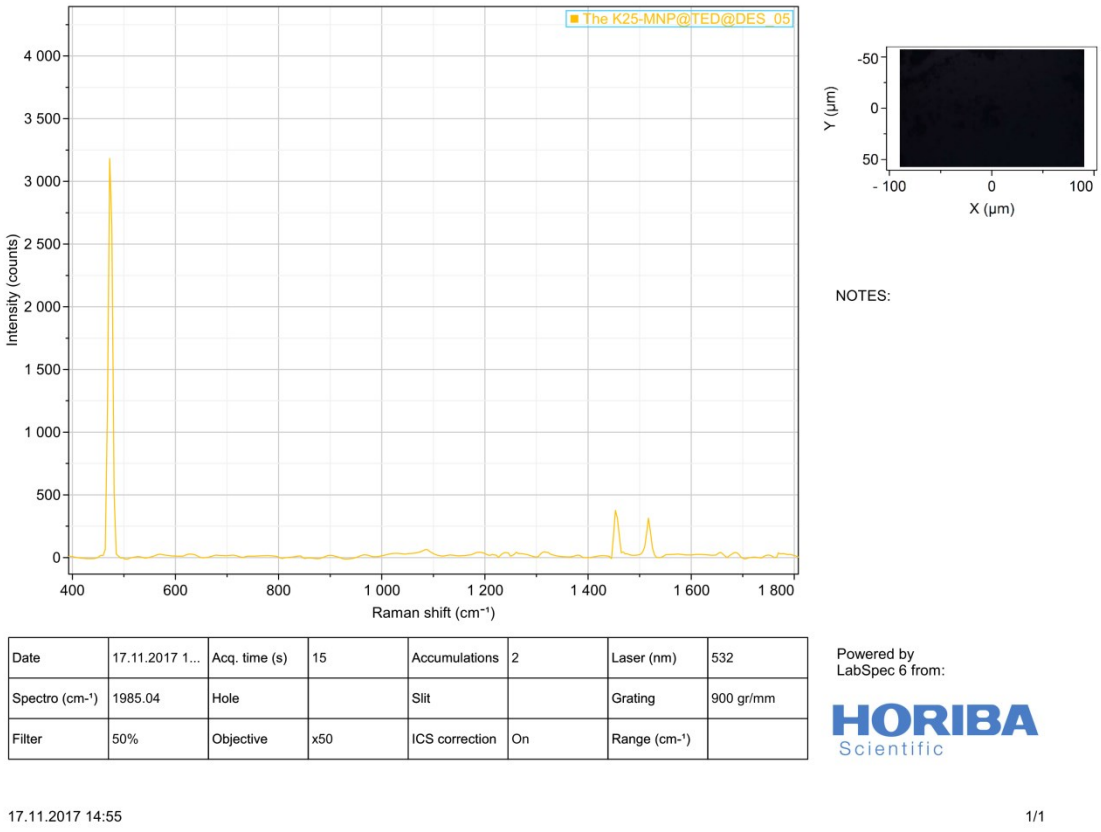


Figure S3. TGA curve of LAIL@MNP



Date	17.11.2017 1...	Acq. time (s)	15	Accumulations	2	Laser (nm)	532
Spectro (cm-¹)	1985.04	Hole		Slit		Grating	900 gr/mm
Filter	50%	Objective	x50	ICS correction	On	Range (cm-¹)	

Figure S4. Raman spectrum of LAIL@MNP

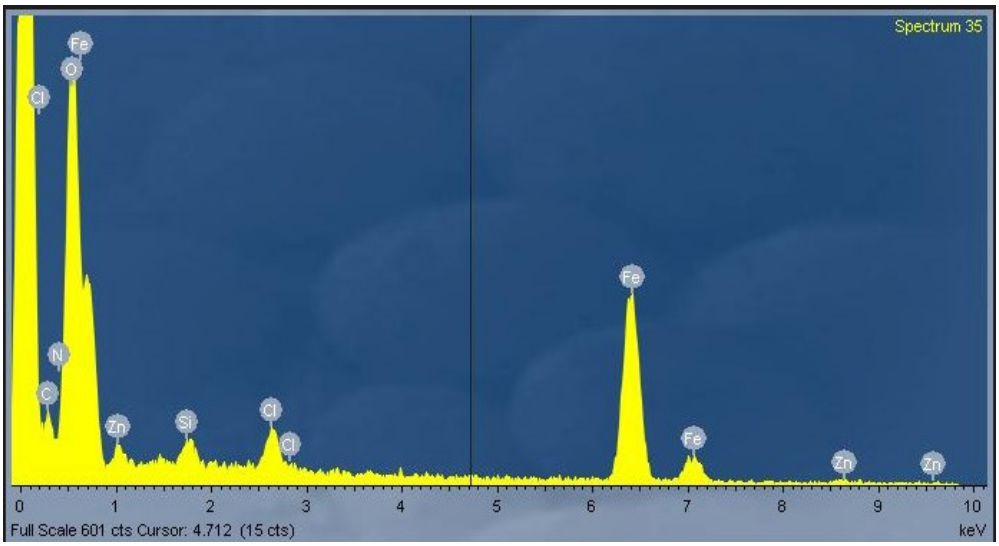


Figure S5. EDS spectrum of LAIL@MNP

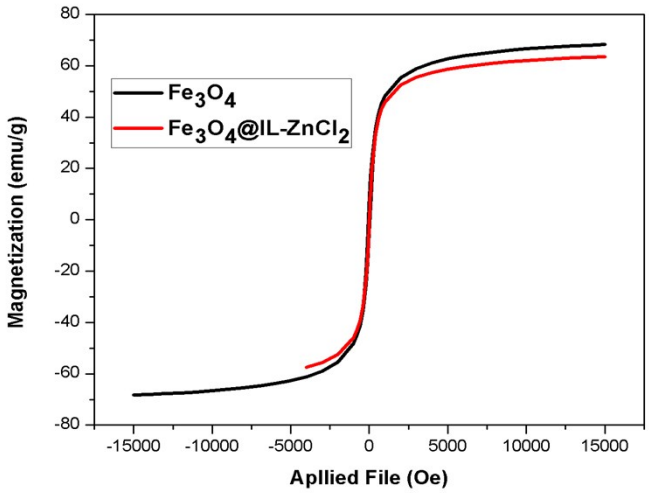


Figure S6. VSM curves for LAIL@MNP

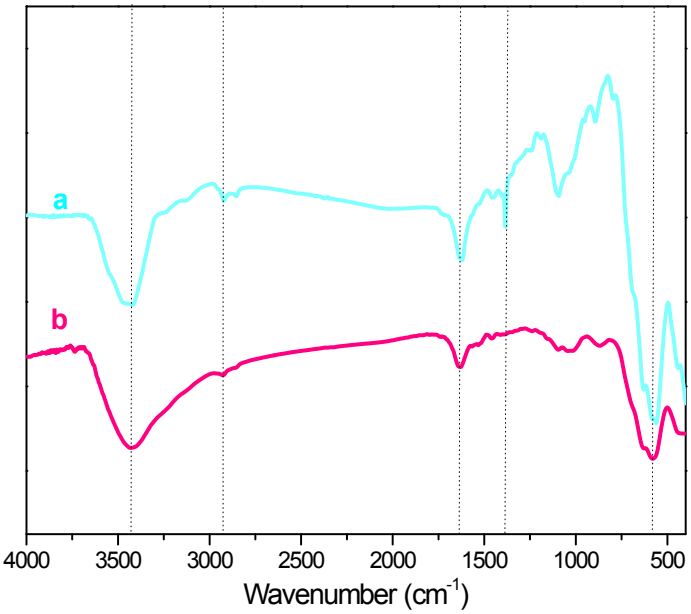
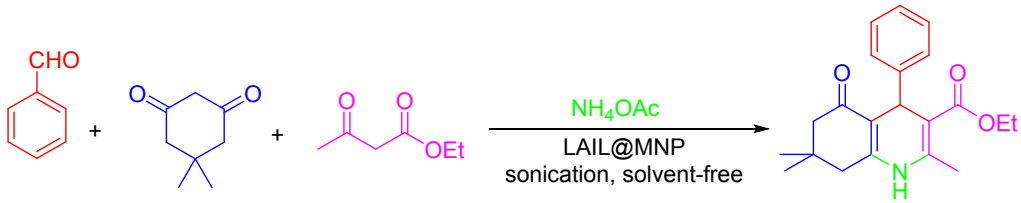


Figure S7. FT-IR spectra of fresh LAIL@MNP (a, blue), and LAIL@MNP after the fifth recovery (b, pink).

Section S3. Optimazation of the reaction condition

Table S1. Optimazation of reaction conditions[a]



Entry	Temperature (°C)	Time (min)	Catalyst loading (mg)	Ratio of a:b:c:d[b] (mmol)	Isolated yield (%)
1	30	10	15	1:1:1:2	37
2	30	20	15	1:1:1:2	49
3	30	30	15	1:1:1:2	55
4	30	45	15	1:1:1:2	63
5	30	60	15	1:1:1:2	68
6	80	10	15	1:1:1:2	65
7	80	20	15	1:1:1:2	77
8	80	30	15	1:1:1:2	84
9	**80**	**45**	**15**	**1:1:1:2**	**97**
10	80	60	15	1:1:1:2	98
11	80	45	15	1:1:1:1	75
12	80	45	15	1:1.2:1:1	84
13	80	45	15	1:1.5:1.1	78
14	80	45	15	1:2:1:1	74
15	80	45	15	1:1:1.2:1	88
16	80	45	15	1:1:1:1.5	81
17	80	45	0	1:1:1:2	34
18	80	45	1	1:1:1:2	45
19	80	45	5	1:1:1:2	63
20	80	45	10	1:1:1:2	77
21	80	45	20	1:1:1:2	97

[a] Under ultrasonic

[b] Benzaldehyde (a), dimedone (b), ethyl acetoacetate (c) and ammonium acetate (d)

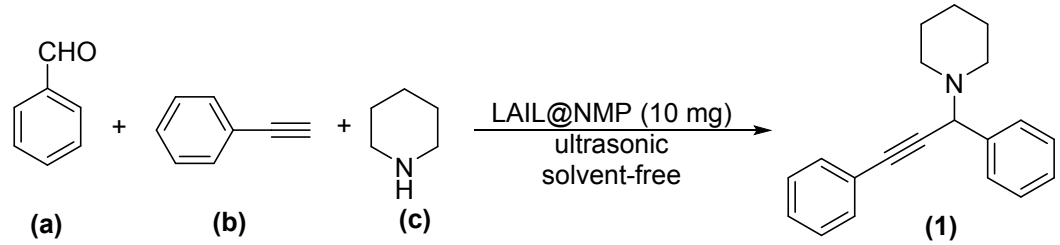
Table S2. Effect of catalysts and solvents

Entry	Catalyst	Solvent	Conversation[a] (%)
1	[BMIM]PF_6	Solvent-free	61
2	[EMIM]Cl	Solvent-free	58
3	$AlCl_3$	Solvent-free	97

Entry	Catalyst	Solvent	Conversation[a] (%)
4	$FeCl_3$	Solvent-free	96
5	Al_2O_3	Solvent-free	51
6	Fe_2O_3	Solvent-free	45
7	$CuFe_2O_4$	Solvent-free	44
8	$ZnFe_2O_4$	Solvent-free	40
9	γ-Fe_2O_3	Solvent-free	42
10	Fe_3O_4	Solvent-free	45
11	LAIL	Solvent-free	56
12	$ZnCl_2$	Solvent-free	95
13	None-catalyst	Solvent-free	34
14	**LAIL@NMP**	**Solvent-free**	**97**
15	LAIL@NMP	Ethanol	72
16	LAIL@NMP	Acetone	47
17	LAIL@NMP	Acetonitrile	41
18	LAIL@NMP	*n*-Hexane	79

[a]: Isolated yields

Table S3. Optimazation of reaction conditions



Entry	Temperature (°C)	Time (min)	Catalyst loading (mg)	Ratio of a:b:c (mmol)	Conversion[a] (%)
1	30	1	10	1:1.5:1.2	30
2	30	5	10	1:1.5:1.2	37
3	30	10	10	1:1.5:1.2	44
4	30	15	10	1:1.5:1.2	48
5	30	20	10	1:1.5:1.2	59
6	30	25	10	1:1.5:1.2	60
7	30	30	10	1:1.5:1.2	65
8	30	45	10	1:1.5:1.2	72
9	30	60	10	1:1.5:1.2	78
10	30	90	10	1:1.5:1.2	80
11	30	120	10	1:1.5:1.2	83
12	80	1	10	1:1.5:1.2	49
13	80	5	10	1:1.5:1.2	54
14	80	10	10	1:1.5:1.2	61
15	80	15	10	1:1.5:1.2	65
16	80	20	10	1:1.5:1.2	73
17	80	25	10	1:1.5:1.2	77
18	80	30	10	1:1.5:1.2	86
19	**80**	**45**	**10**	**1:1.5:1.2**	**95**
20	80	60	10	1:1.5:1.2	96
21	80	90	10	1:1.5:1.2	95
22	80	120	10	1:1.5:1.2	97
23	80	45	0	1:1.5:1.2	60
24	80	45	1	1:1.5:1.2	73

Entry	Temperature (°C)	Time (min)	Catalyst loading (mg)	Ratio of a:b:c (mmol)	Conversion[a] (%)
25	80	45	5	1:1.5:1.2	81
26	80	45	15	1:1.5:1.2	100
27	80	45	20	1:1.5:1.2	100
28	80	45	10	1:1:1	49
29	80	45	10	1:1.1:1	53
30	80	45	10	1:1.2:1	55
31	80	45	10	1:1.3:1	55
32	80	45	10	1:1.4:1	59
33	80	45	10	1:1.5:1	62
34	80	45	10	1:2:1	70
35	80	45	10	1:1.5:1.1	86

[a] Conversion was reported by GC-MS

Table S4. Effect of catalysts and solvents

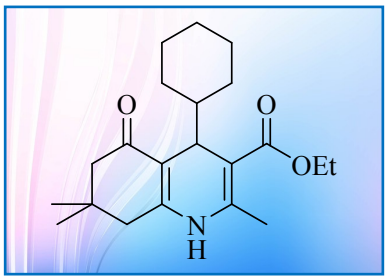
Entry	Catalyst	Solvent	Conversion[a] (%)
1	[BMIM]PF_6	Solvent-free	85
2	[EMIM]Cl	Solvent-free	81
3	[ButMIm]OTf	Solvent-free	88
4	$AlCl_3$	Solvent-free	95
5	$FeCl_3$	Solvent-free	96
6	$CuCl_2$	Solvent-free	95
7	$HfCl_4$	Solvent-free	93
8	Al_2O_3	Solvent-free	80
9	Fe_2O_3	Solvent-free	77
10	MgO	Solvent-free	70
11	CuO	Solvent-free	71
12	Cu_2O	Solvent-free	72
13	ZnO	Solvent-free	75
14	$CuFe_2O_4$	Solvent-free	83

Entry	Catalyst	Solvent	Conversion[a] (%)
15	$ZnFe_2O_4$	Solvent-free	81
16	γ-Fe_2O_3	Solvent-free	85
17	Fe_3O_4	Solvent-free	82
18	LAIL	Solvent-free	79
19	$ZnCl_2$	Solvent-free	94
20	None-catalyst	Solvent-free	47
21	**LAIL@NMP**	**Solvent-free**	**95**
22	LAIL@NMP	Dichloromethane	52
23	LAIL@NMP	Tetrahydrofurane	59
24	LAIL@NMP	2-Butanol	47
25	LAIL@NMP	Ethanol	55
26	LAIL@NMP	2-Propanol	53
27	LAIL@NMP	Acetone	46
28	LAIL@NMP	*N,N*-Dimethylformamide	50
29	LAIL@NMP	Acetonitrile	63
30	LAIL@NMP	Dimethyl sulfoxide	74
31	LAIL@NMP	Ethyl acetate	71
32	LAIL@NMP	Cyclopentyl methyl ether	69
33	LAIL@NMP	*n*-Hexane	70
34	LAIL@NMP	Toluene	78
35	LAIL@NMP	1,4-Dioxane	82
36	LAIL@NMP	Chloroform	69

[a] Conversion was reported by GC-MS

Section S4. Spectral data.

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



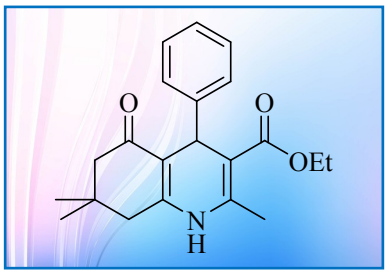
White solid, mp = 175 – 178 °C

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[5-7] (2)



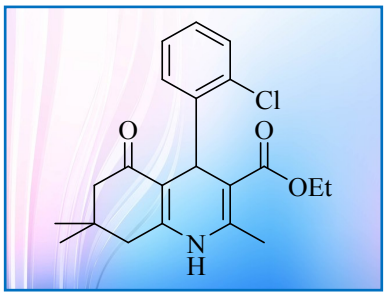
Pale yellow solid, mp = 201 – 203 °C

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.

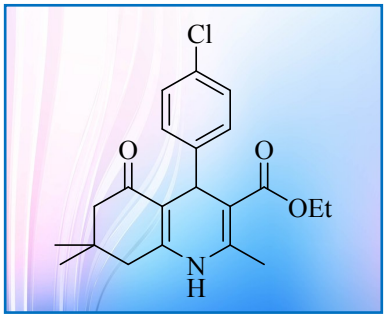
Ethyl 4-(2-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahyydroquinolin-3-carboxylate[5, 8] (3)



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[8, 9] (4)



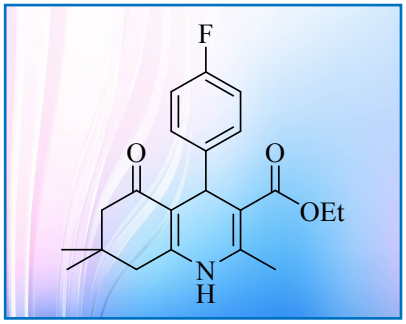
Pale yellow solid, mp = 246 – 248 °C

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[10] (5)



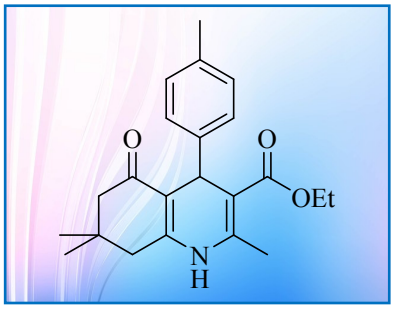
Pale yellow solid, mp = 184 – 187 °C

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[9] (6)



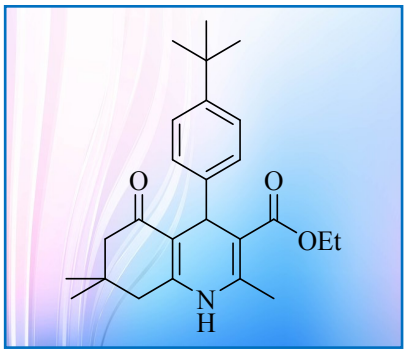
Pale yellow solid, mp = 261 – 263 °C

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[5] (7)

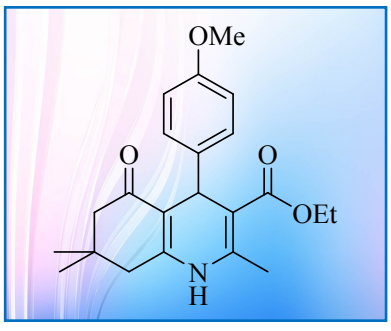


White solid, mp = 210 – 211 °C

^{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[9] (8)



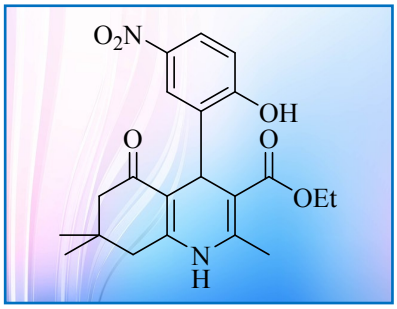
Pale yellow, mp = 255 – 257 °C

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-(2-hydroxy-5-nitrophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate[5] (9)



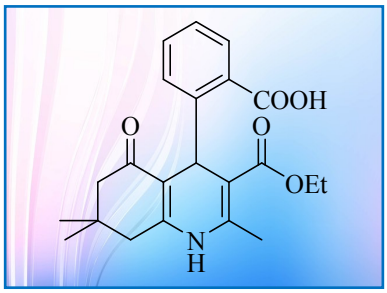
Pale yellow solid, mp = 210 – 213 °C

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 (10)



White solid, mp = 257 – 259 °C

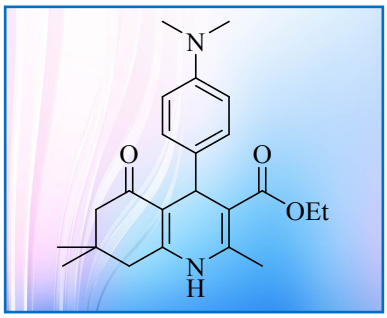
IR (KBr, cm^{-1}): 3292, 2956, 2873, 1699, 1575, 1479, 1390, 1220, 731.

^{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-(4-dimethylamino)phenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexayidroquinolin-3-carboxylate[11] (11)



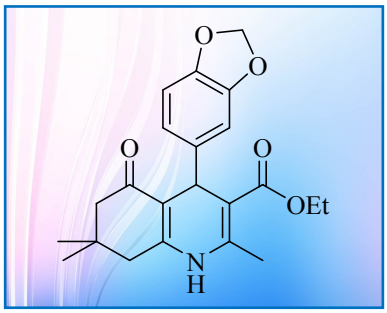
Pale yellow solid, mp = 225 – 227 °C

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-(benzo[*d*][1,3]dioxol-5-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate[12] (12)



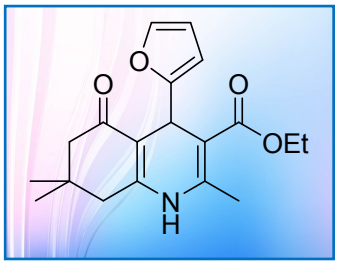
Pale yellow solid, mp = 225 – 228 °C

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[13] (13)



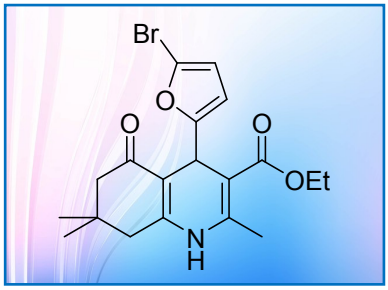
White solid, mp = 246 – 248 °C

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 (14)



White solid, mp = 215 – 218 °C

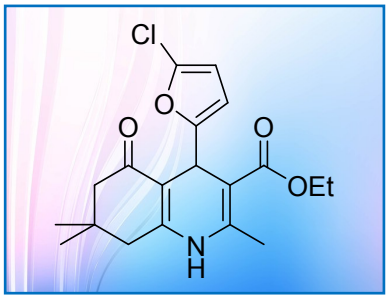
IR (KBr, cm^{-1}): 3277, 3078, 1678, 1604, 1485, 1211.

^{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 (15)



White solid, mp = 195 – 198 °C

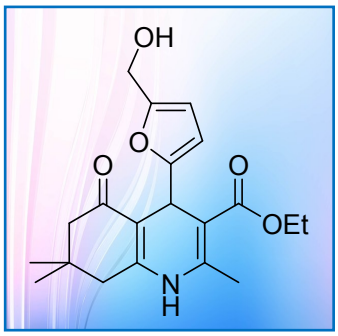
IR (KBr, cm^{-1}): 3277, 3076, 1677, 1602, 1485, 1215.

^{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 (16)



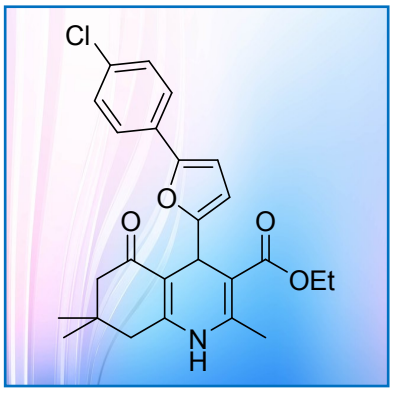
White solid, mp = 186 – 189 °C

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 (17)



Pale yellow, mp = 185 – 188 °C

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

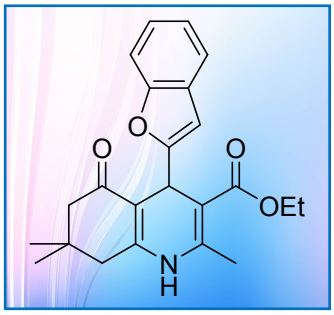
^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.44–7.42 (d, J = 8.5 Hz, 2H), 7.26–7.24 (t, J = 6.0 Hz, 3H), 6.47–6.46 (d, J = 3.5 Hz, 1H), 6.07 (d, J = 3.0 Hz, 1H), 6.02 (br, 1H), 5.28

(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 (18)



White solid, mp = 205 – 208 ^oC

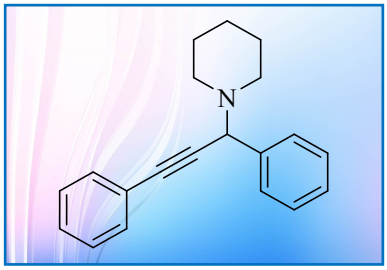
IR (KBr, cm^{-1}): 3280, 3084, 2953, 1699, 1602, 1489, 1379, 1209, 723

^{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.

(1,3-Diphenylprop-2-yn-1-yl)piperidine[14-16] (19)



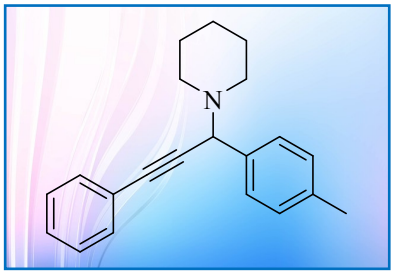
Yellow oil

^{1}H-NMR (500 MHz, $CDCl_3$): δ_H 7.65 (d, J = 7.5 Hz, 2H), 7.53 (m, 2H), 7.39 – 7.28 (m, 6H), 4.83 (s, 1H), 2.59 (t, J = 4.5 Hz, 4H), 1.65 – 1.57 (m, 4H), 1.46 (dd, J = 11.0, 5.0 Hz, 2H).

^{13}C-NMR (125 MHz, $CDCl_3$): δ_C 138.7, 132.0, 128.7, 128.4, 128.2, 127.6, 123.5, 88.0, 86.2, 62.5, 50.8, 29.8, 26.3, 24.6.

GC-MS (EI, 70 eV) *m/z* 275

1-(3-Phenyl-1-(*p*-tolyl)prop-2-yn-1-yl)piperidine[17] (20)



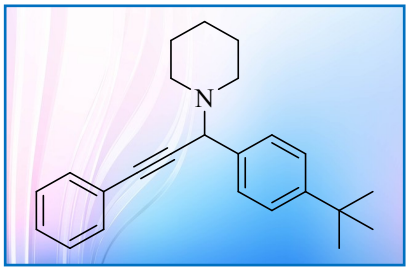
Yellow oil

^{1}H NMR (500 MHz, DMSO-d_6) δ_H δ 7.53 (d, J = 4.0 Hz, 2H), 7.48 (d, J = 7.0 Hz, 2H), 7.41 (d, J = 4.0 Hz, 3H), 7.22–7.19 (m, 2H), 4.87 (s, 1H), 2.54–2.49 (m, 4H), 2.32 (s, 3H), 1.56–1.50 (m, 4H), 1.40 (m, 2H).

^{13}C NMR (125 MHz, DMSO-d_6) δ_C 136.5, 135.3, 131.4, 128.6, 128.6, 128.3, 128.0, 122.5, 87.3, 86.2, 61.0, 50.0, 25.7, 24.0, 20.6.

GC-MS (EI, 70 eV) *m/z* 289

1-(1-(4-(*tert*-Butyl)phenyl)-3-phenylprop-2-yn-1-yl)piperidine[18] (21)



Yellow oil

^{1}H NMR (500 MHz, DMSO-d_6) δ_H 7.49–7.42 (m, 4H), 7.38–7.33 (m, 5H), 4.81 (s, 1H), 2.49–2.39 (m, 4H), 1.47 (m, 4H), 1.38–1.31 (m, 2H), 1.24 (s, 9H).

^{13}C NMR (125 MHz, DMSO-d_6) δ_C 149.8, 135.3, 131.4, 128.6, 128.4, 127.8, 124.8, 122.5, 87.3, 86.3, 60.9, 50.1, 34.2, 31.1, 25.7, 24.0.

GC-MS (EI, 70 eV) *m/z* 331

1-(1-(4-Chlorophenyl)-3-phenylprop-2-yn-1-yl)piperidine[17, 18] (22)

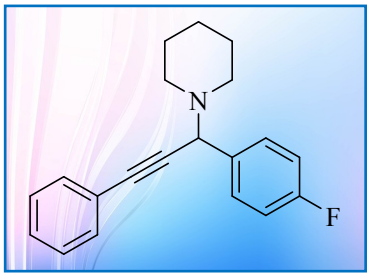
Yellow oil

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.59 (d, J = 8.5 Hz, 2H), 7.51 (m, 2H), 7.3 (m, 5H), 4.80 (s, 1H), 2.56 (t, J = 5.0 Hz, 4H), 1.64–1.56 (m, 4H), 1.46 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 143.4, 137.2, 133.5, 132.0, 130.0, 128.5, 128.4, 123.2, 88.5, 85.4, 61.8, 50.8, 26.2, 24.5.

GC-MS (EI, 70 eV) *m/z* 309

1-(1-(4-Fluorophenyl)-3-phenylprop-2-yn-1-yl)piperidine[17] (23)



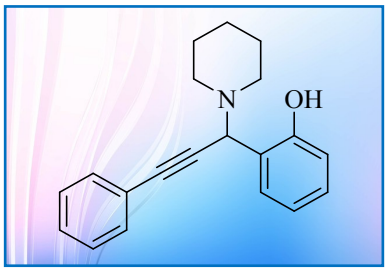
Yellow oil

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.61 (dd, J = 8.5, 5.5 Hz, 2H), 7.51 (m, 2H), 7.35–7.32 (m, 3H), 7.04 (t, J = 8.5 Hz, 2H), 4.80 (s, 1H), 2.56 (s, 4H), 1.59 (m, 4H), 1.46 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 163.3, 131.8, 130.2, 130.1, 128.3, 128.2, 115.0, 114.9, 61.6, 61.6, 50.6, 26.0, 24.3.

GC-MS (EI, 70 eV) *m/z* 293

1-(1-(2-Hydroxyphenyl)-3-phenylprop-2-in-1-yl)piperidine[19, 20] (24)

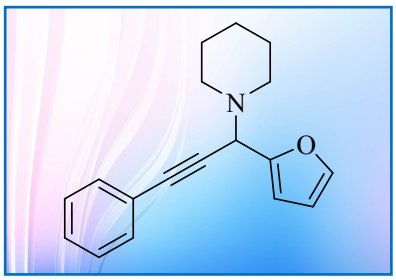


Yellow oil

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.61 – 7.56 (m, 3H), 7.40 – 7.37 (m, 3H), 7.26–7.23 (m, 1H), 6.91–6.87 (m, 2H), 5.12 (s, 1H), 2.78–2.73 (m, 3H), 1.70 (s, 4H), 1.54 (s, 1H).

GC-MS (EI, 70 eV) *m/z* 291

1-(1-(Furan-2-yl)-3-phenylprop-2-in-1-yl)piperidine[21, 22] (25)



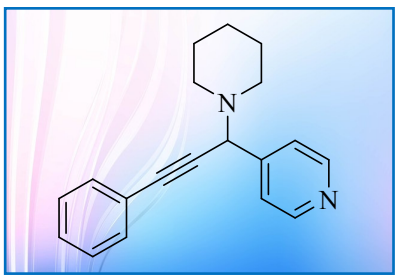
Yellow oil

^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.50–7.49 (m, 2H), 7.42 (s, 1H), 7.33–7.32 (t, J = 3.0 Hz, 6.5 Hz, 3H), 6.48 (d, J = 3.0 Hz, 1H), 6.35–6.34 (m, 1H), 4.87 (s, 1H), 2.61–2.56 (m, 4H), 1.69–1.59 (m, 4H), 1.47–1.43 (m, 2H).

^{13}C NMR (125 MHz, $CDCl_3$) δ_C 164.0, 160.7, 142.7, 132.0, 128.4, 117.3, 110.1, 109.4, 86.6, 84.0, 56.7, 50.7, 26.1, 24.5.

GC-MS (EI, 70 eV) *m/z* 265

4-(3-Phenyl-1-(piperidine-1-yl)prop-2-in-1-yl)pyridine[23, 24] (26)



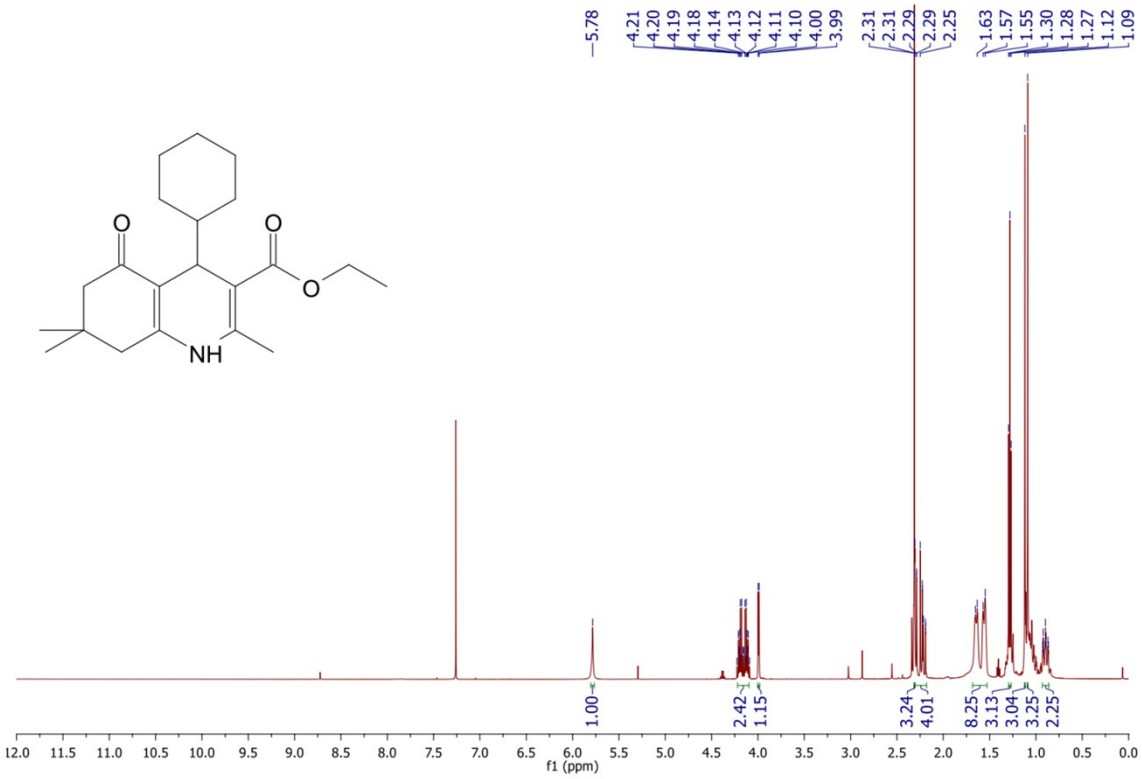
Yellow oil

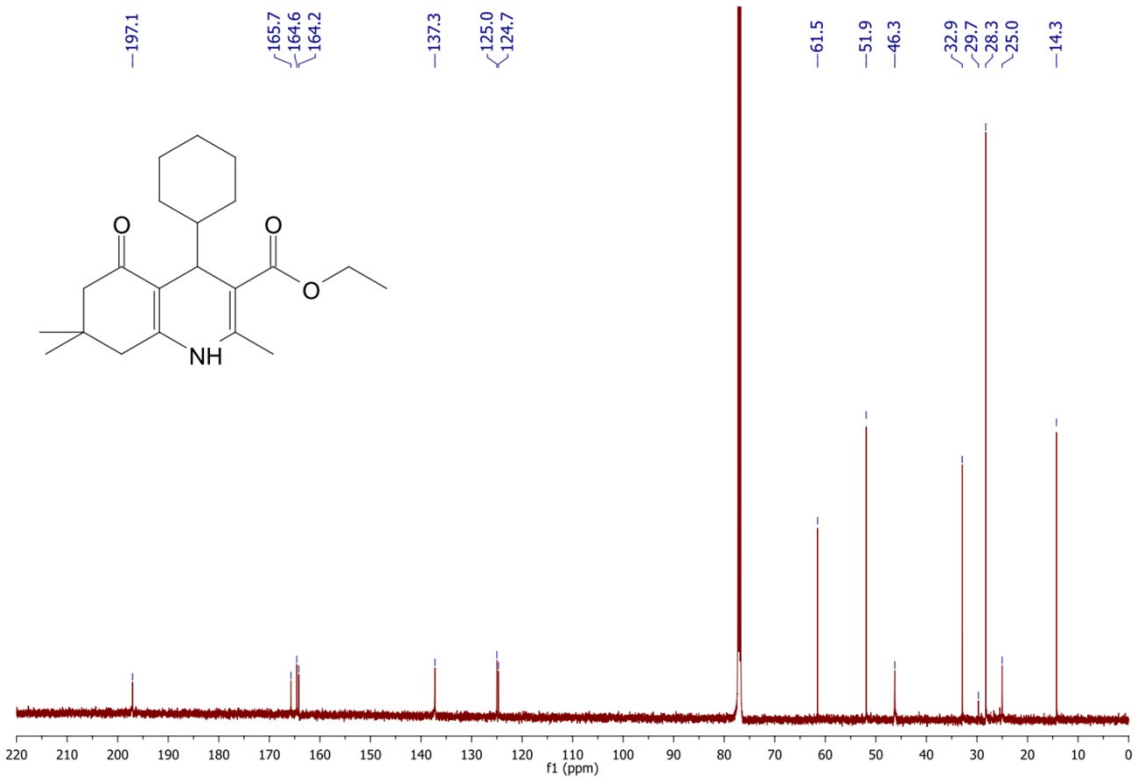
^{1}H NMR (500 MHz, $CDCl_3$) δ_H 7.64 (d, J = 7.5 Hz, 2H), 7.54–7.52 (m, 2H), 7.37–7.32 (m, 4H), 4.81 (s, 1H), 2.62–2.50 (m, 4H), 1.66–1.54 (m, 4H), 1.48–1.44 (m, 2H).

GC-MS (EI, 70 eV) *m/z* 276

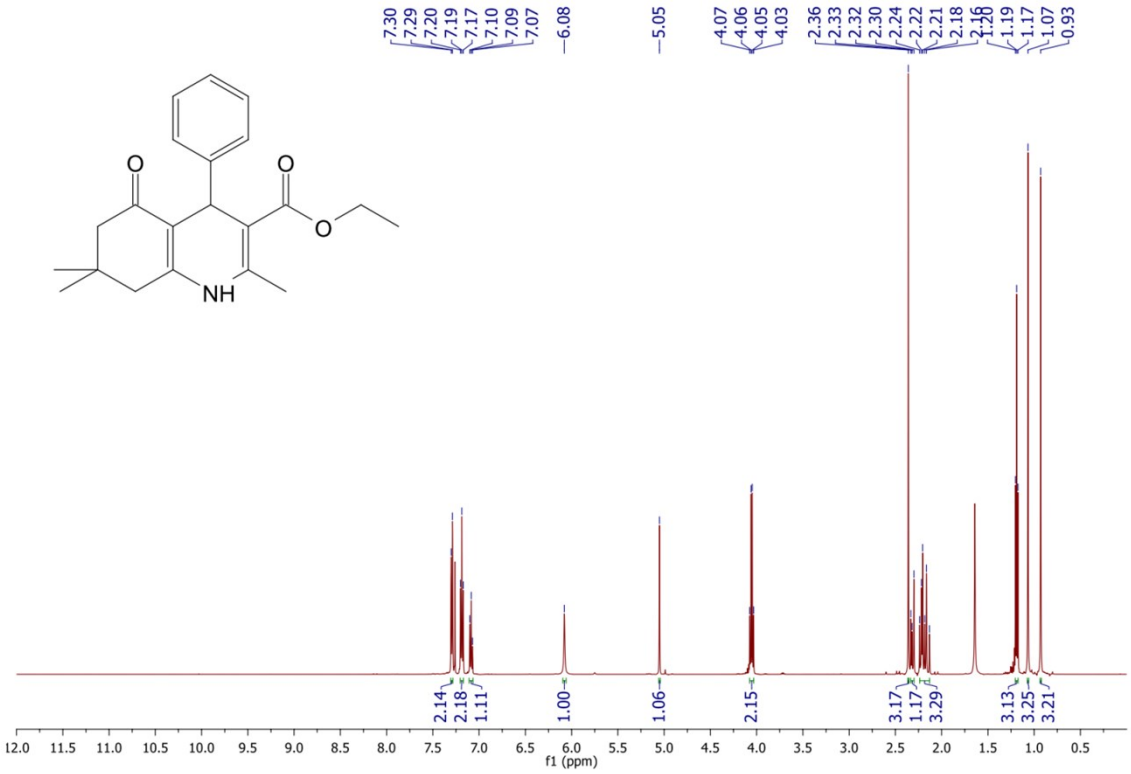
Section S5.

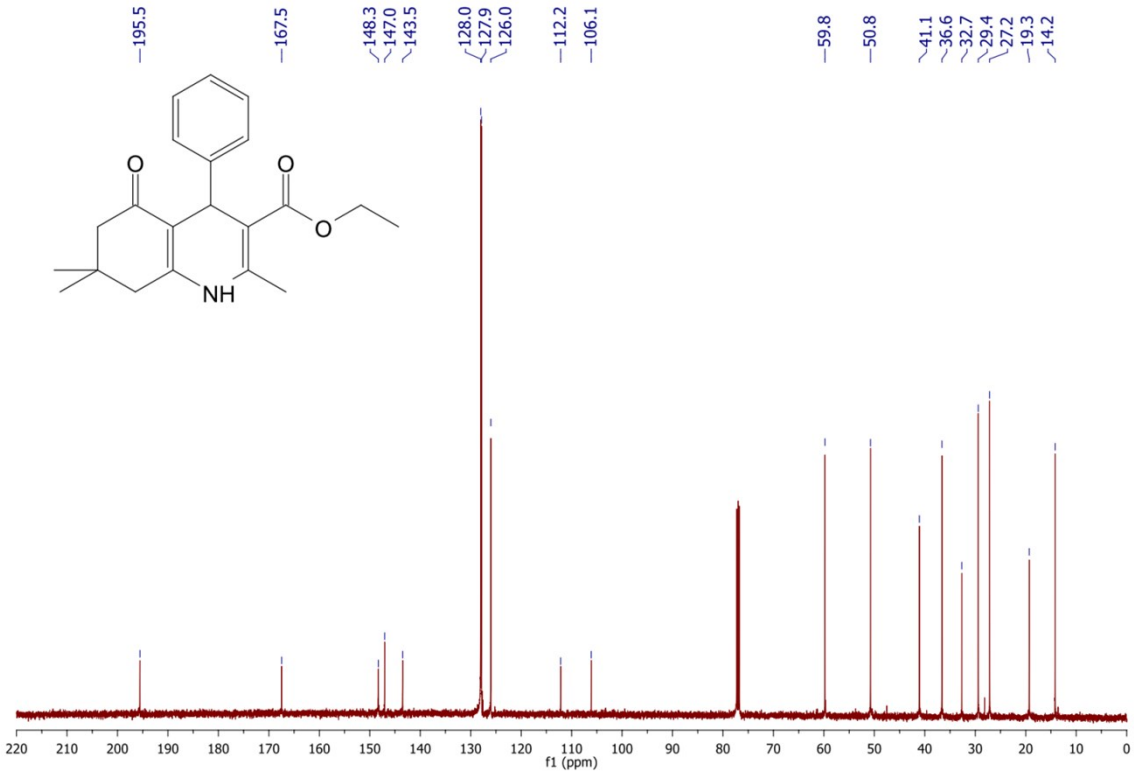
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-cyclohexyl-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (1)



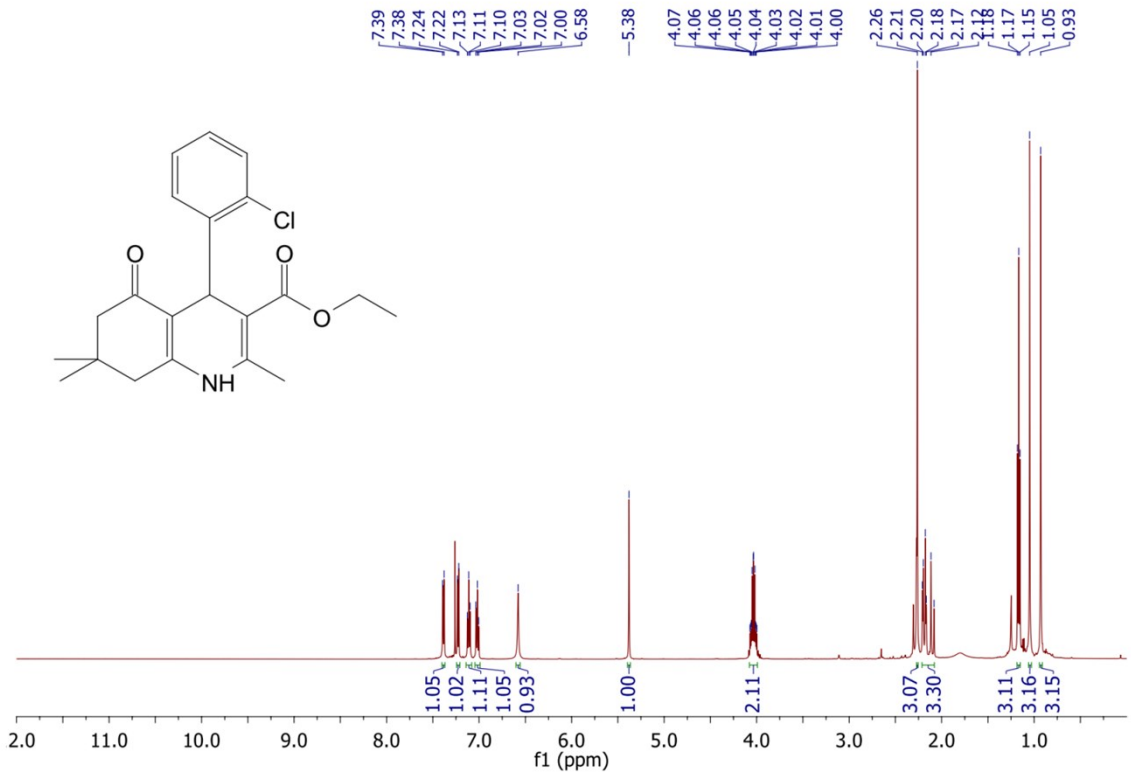


^{1}H and ^{13}C NMR spectroscopy of ethyl 2,7,7-trimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (2)

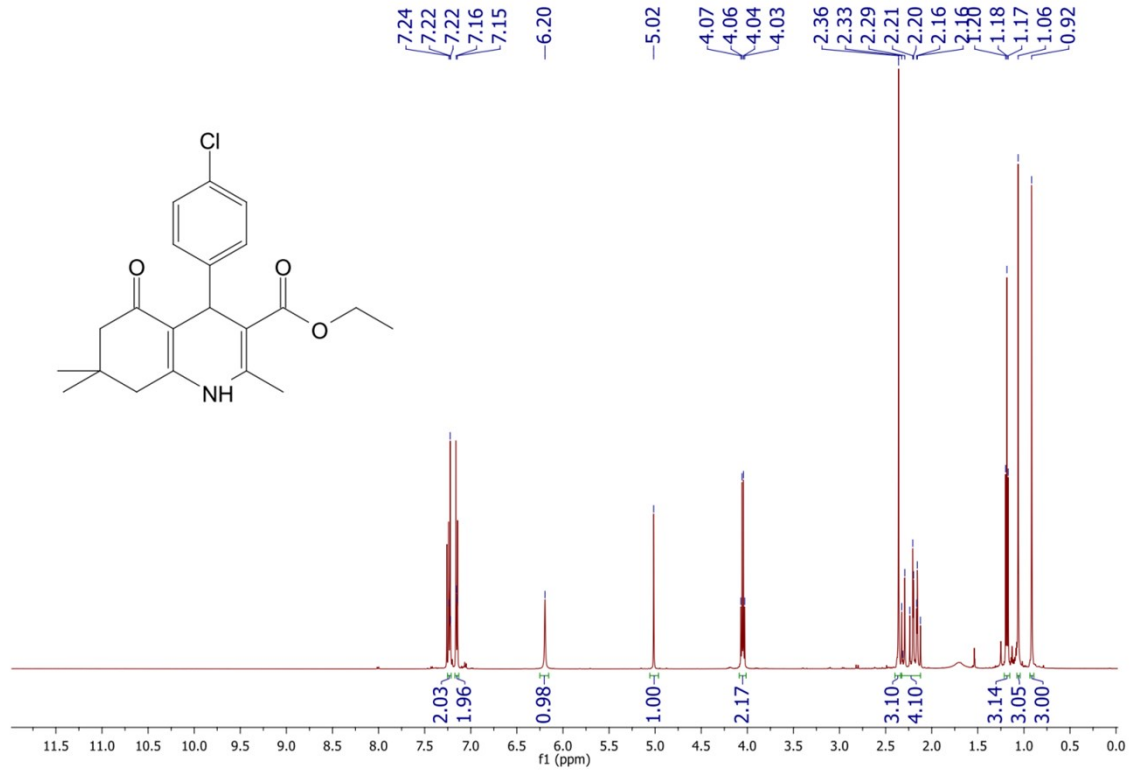


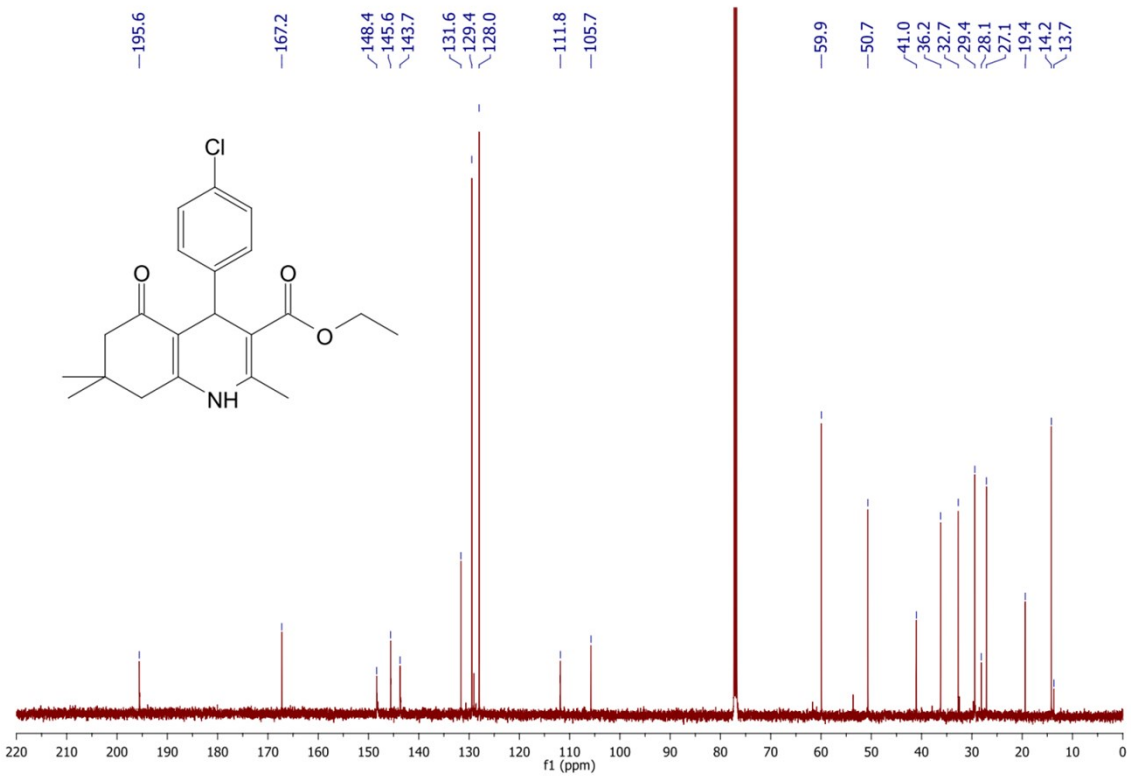


^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(2-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (3)

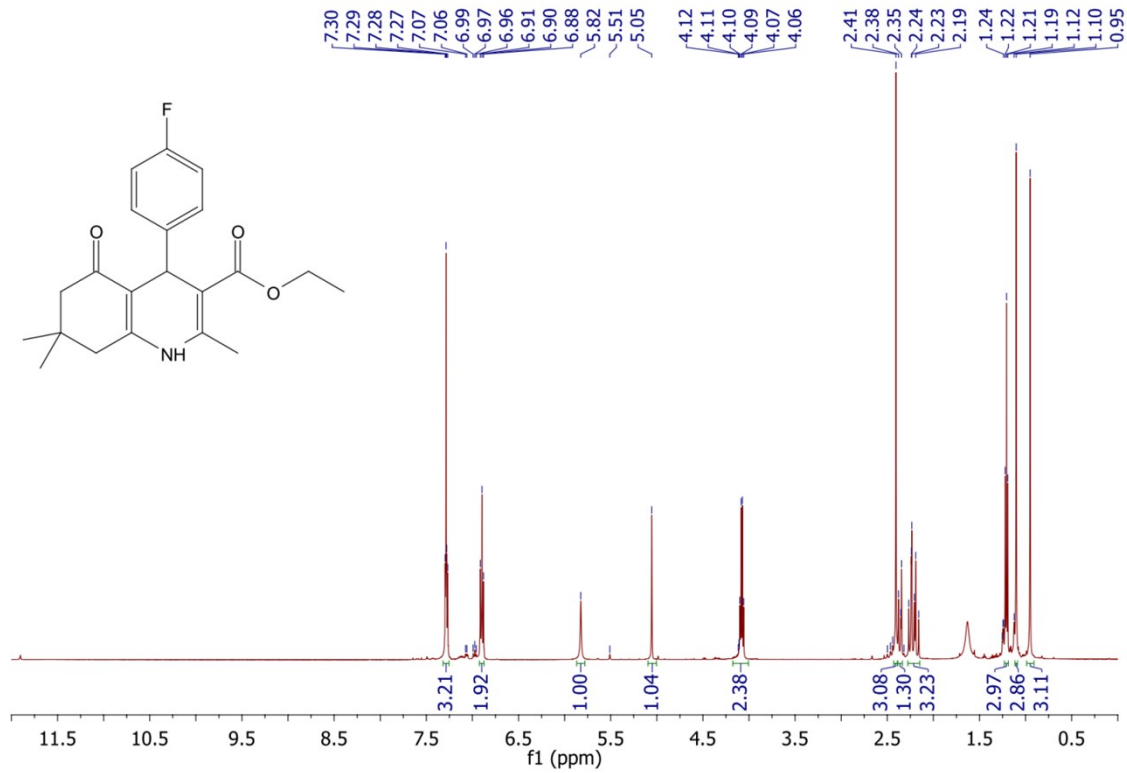


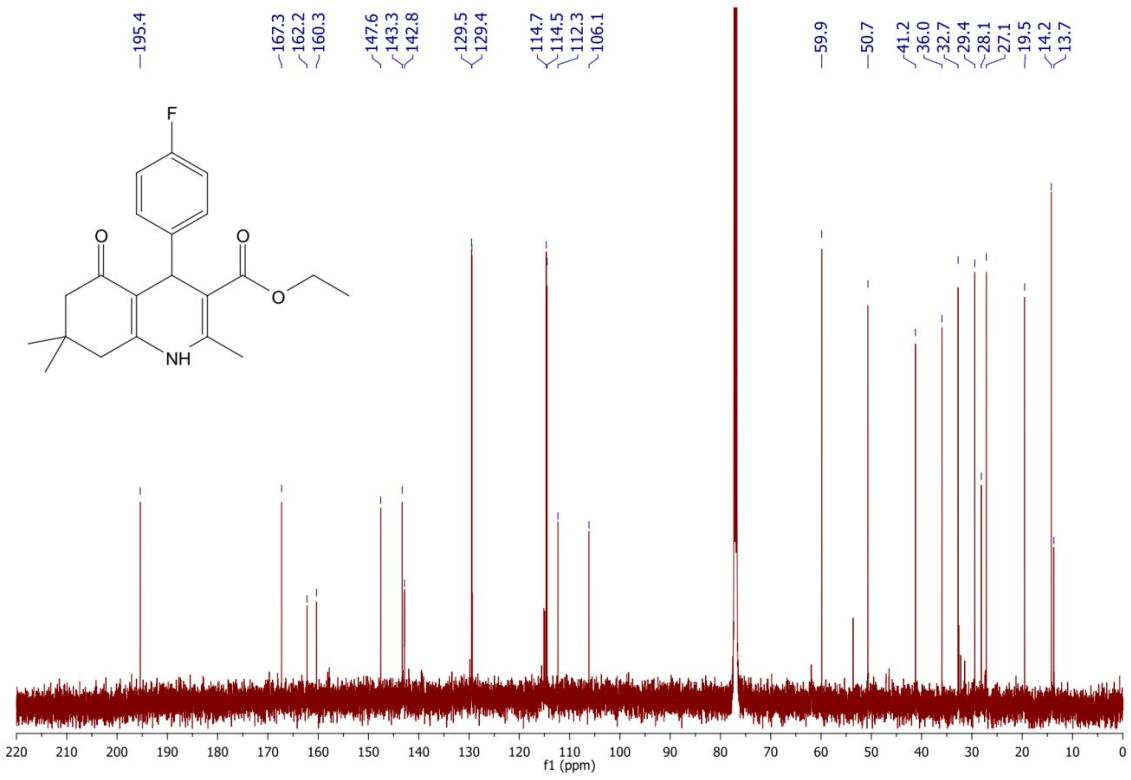
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(4-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (4)



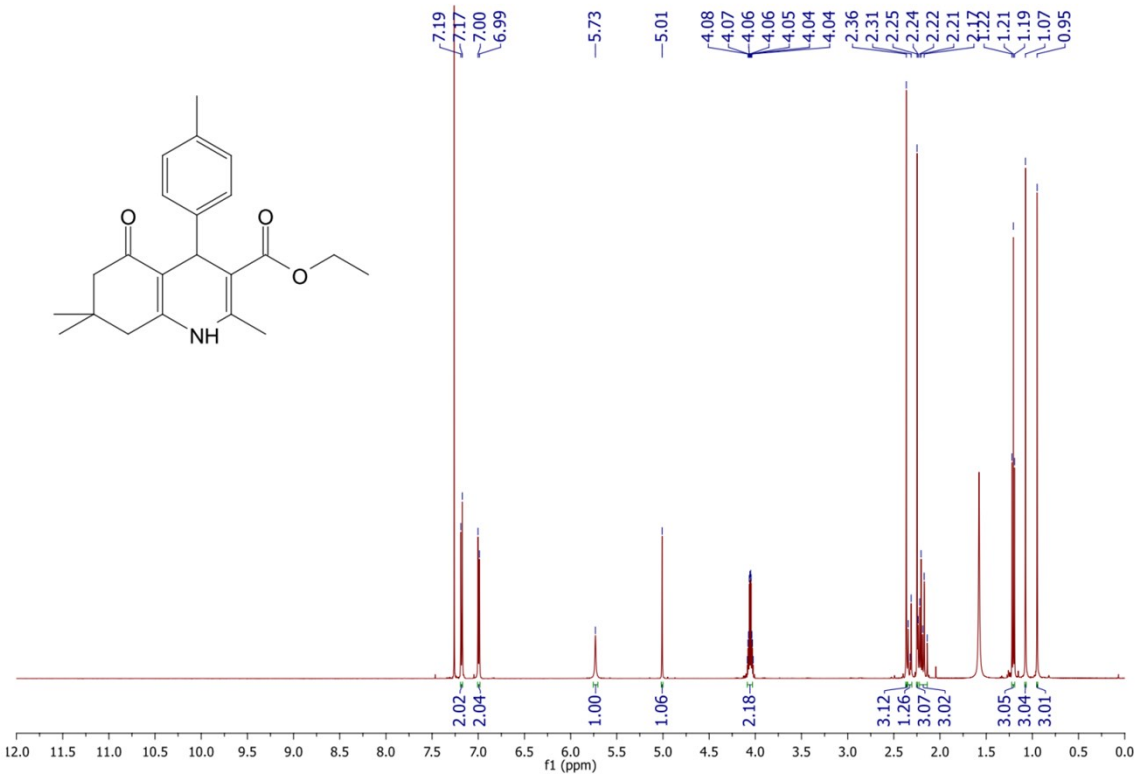


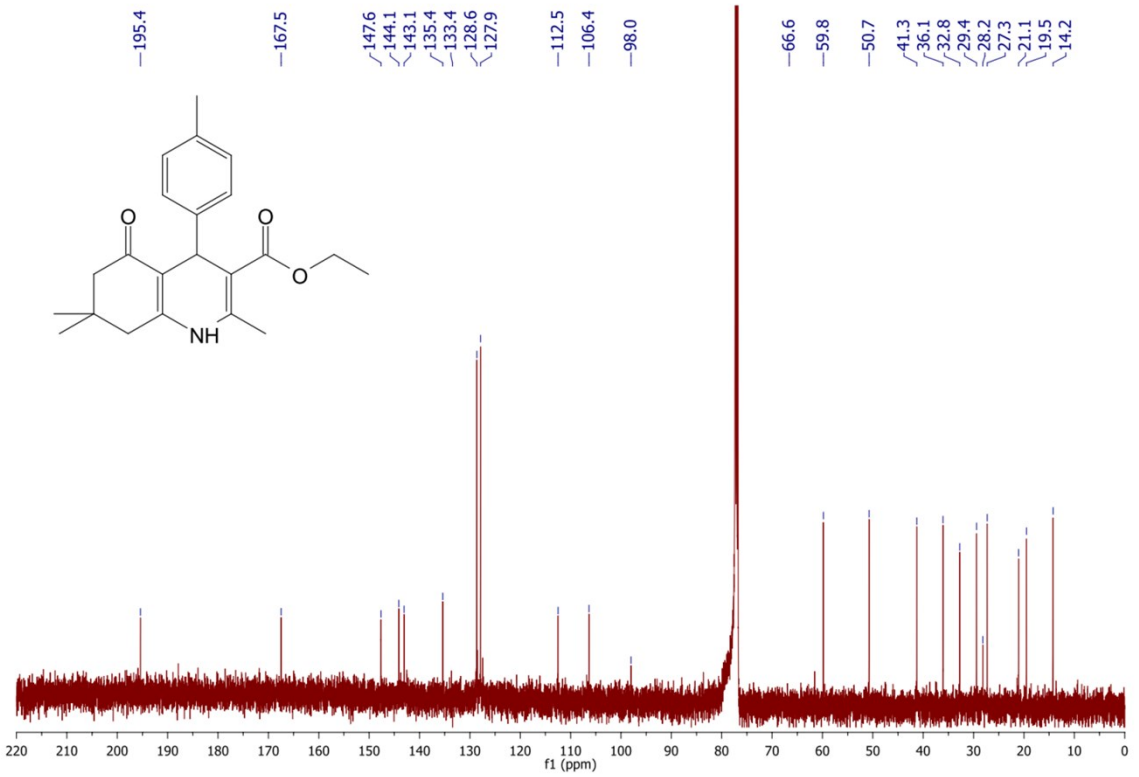
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(4-fluorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (5)



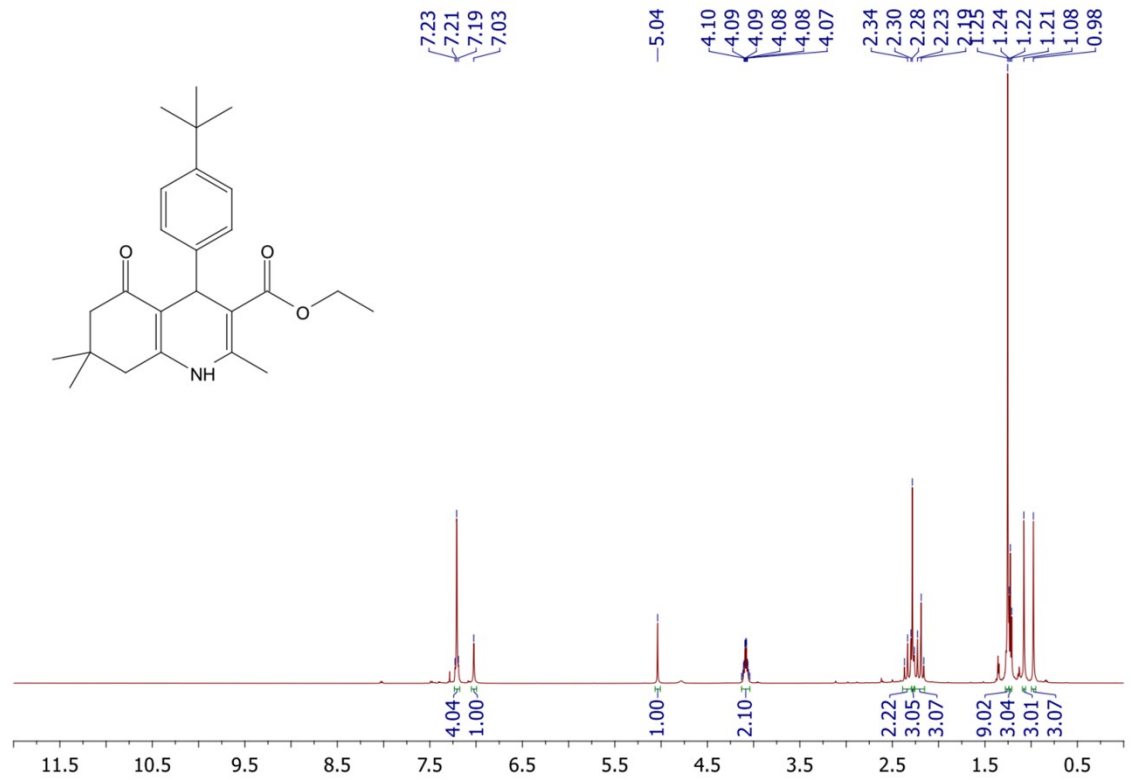


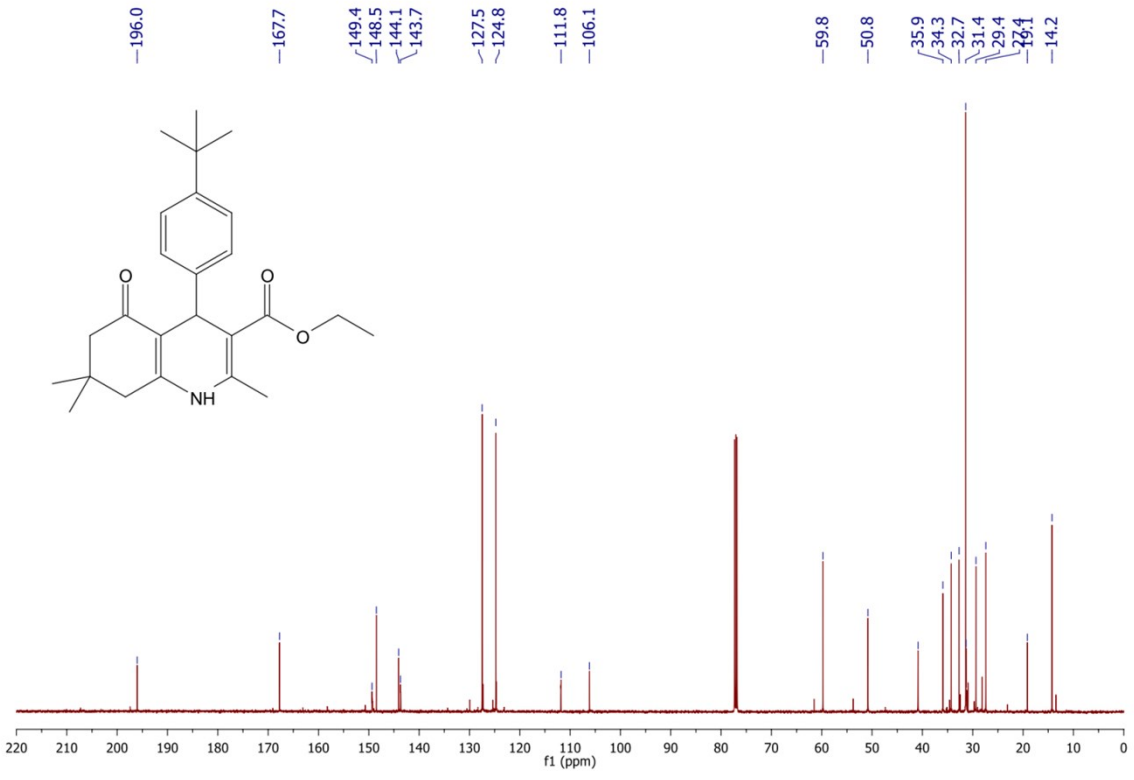
^{1}H and ^{13}C NMR spectroscopy of ethyl 2,7,7-trimethyl-5-oxo-4-(*p*-tolyl)-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (6)



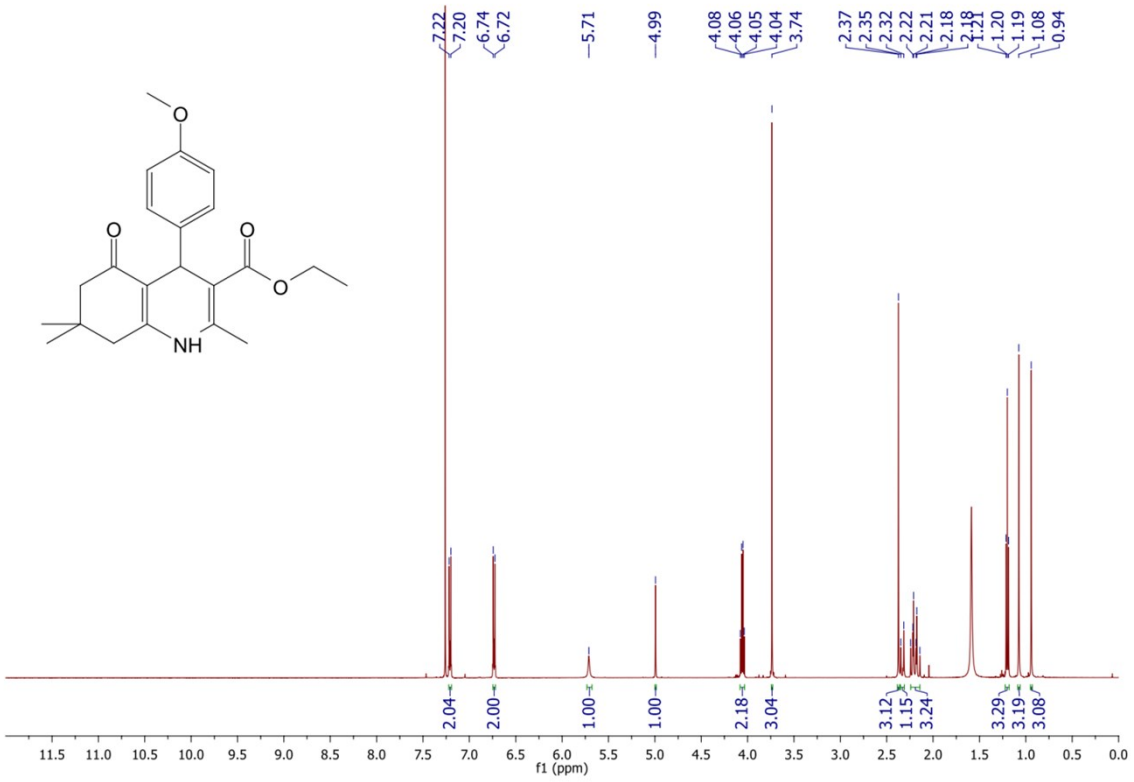


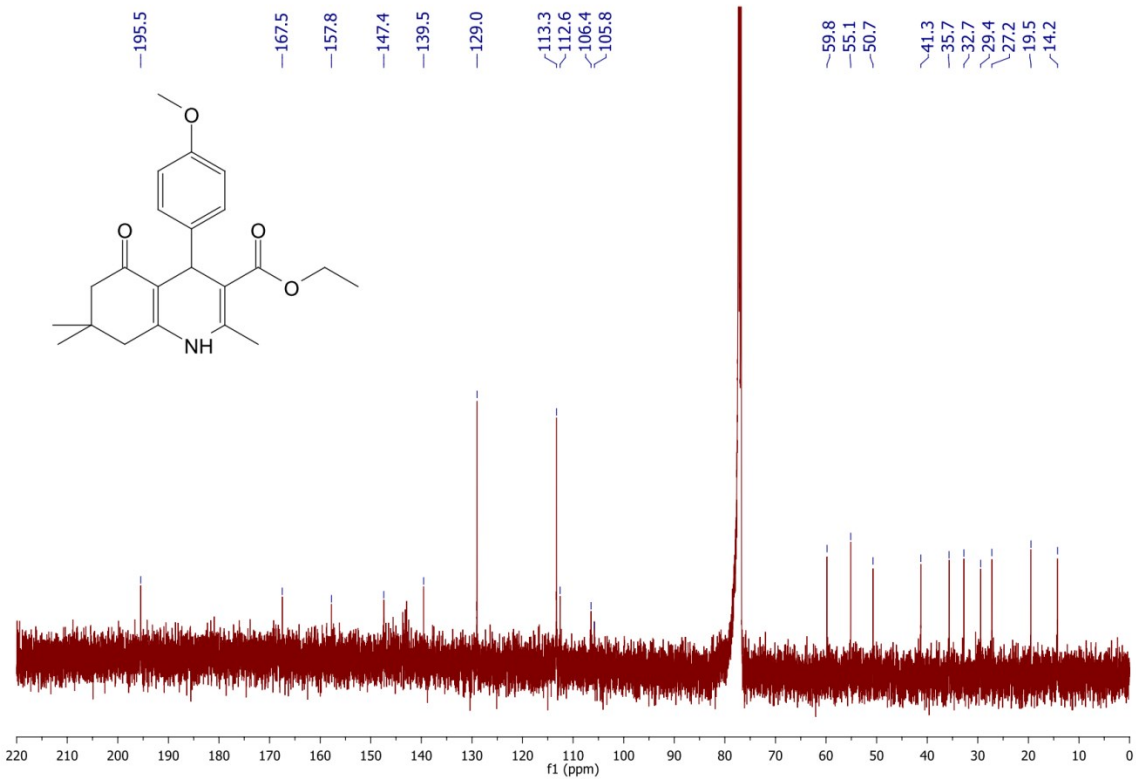
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(4-(*tert*-butyl)phenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (7)



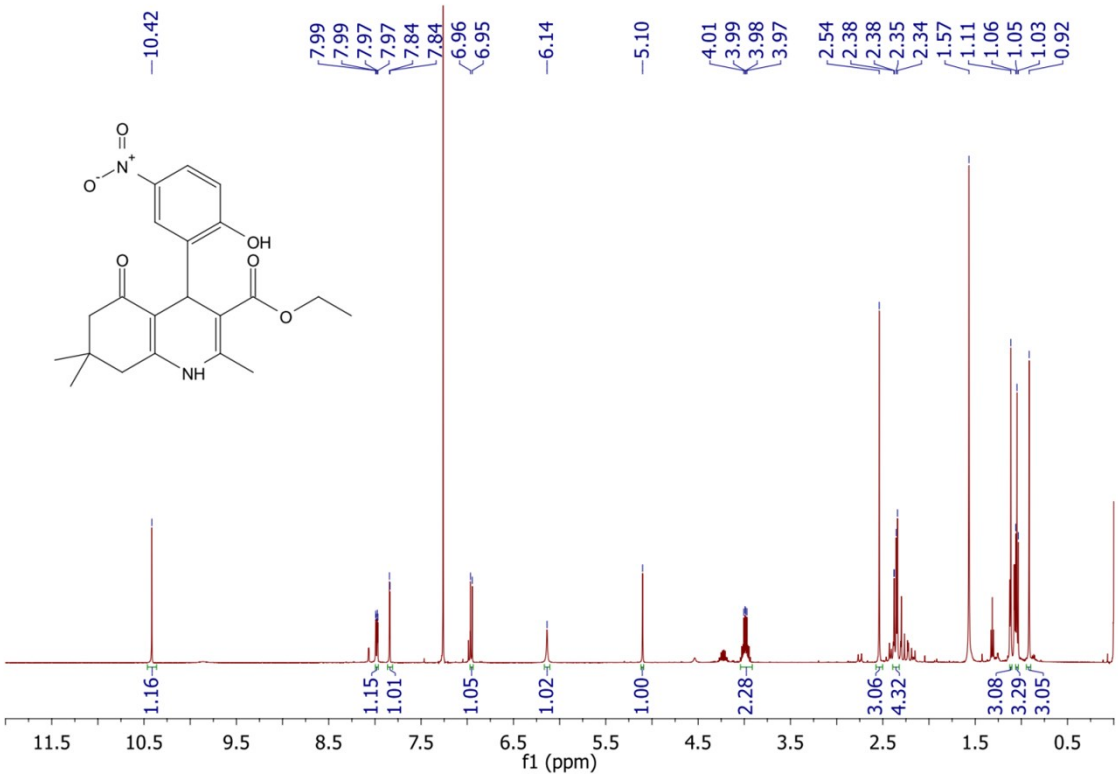


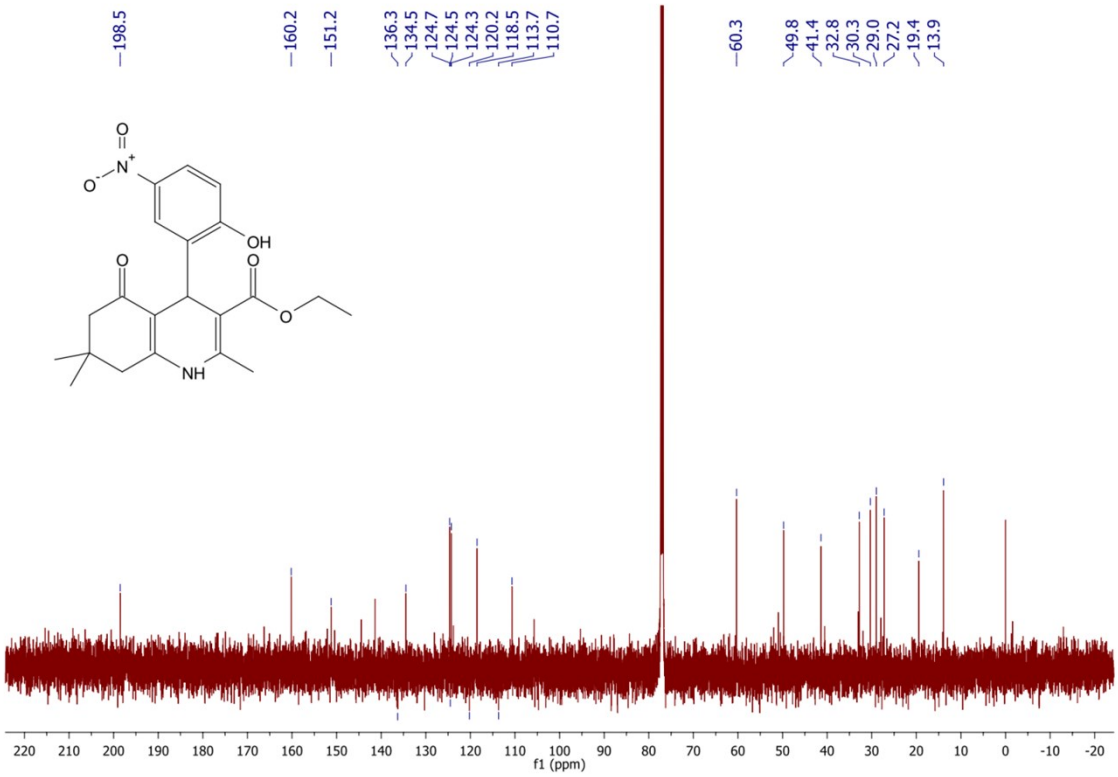
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(4-methoxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (8)



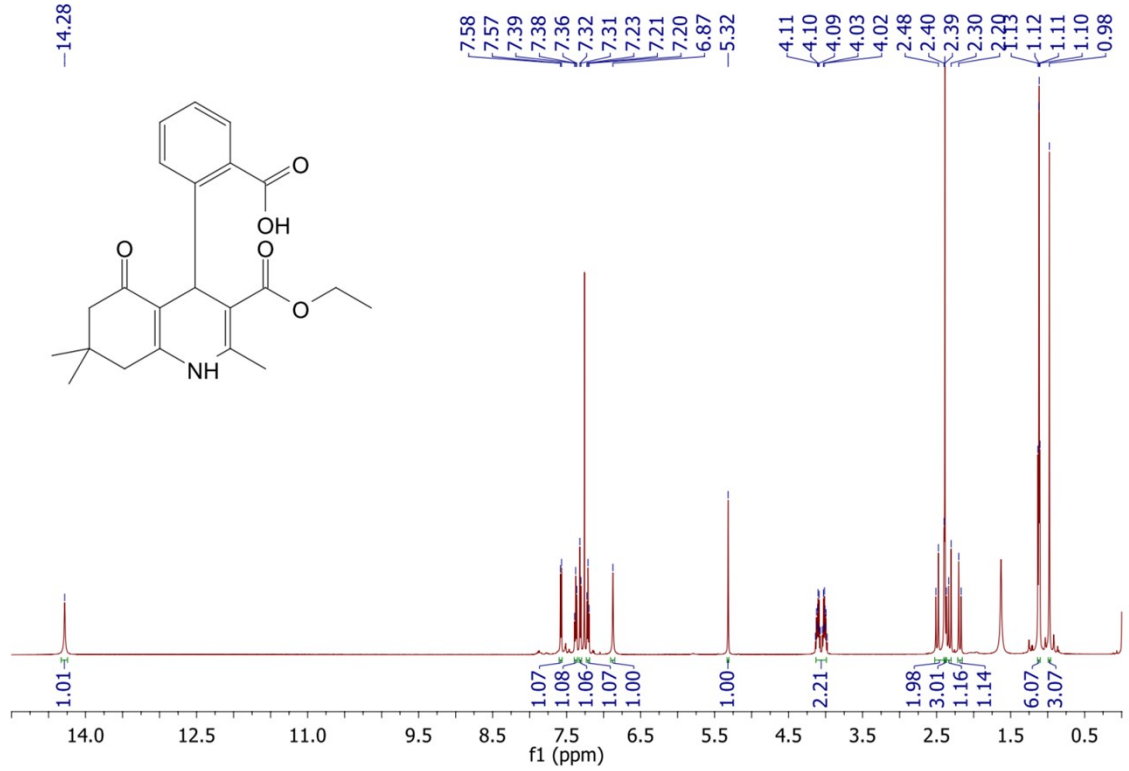


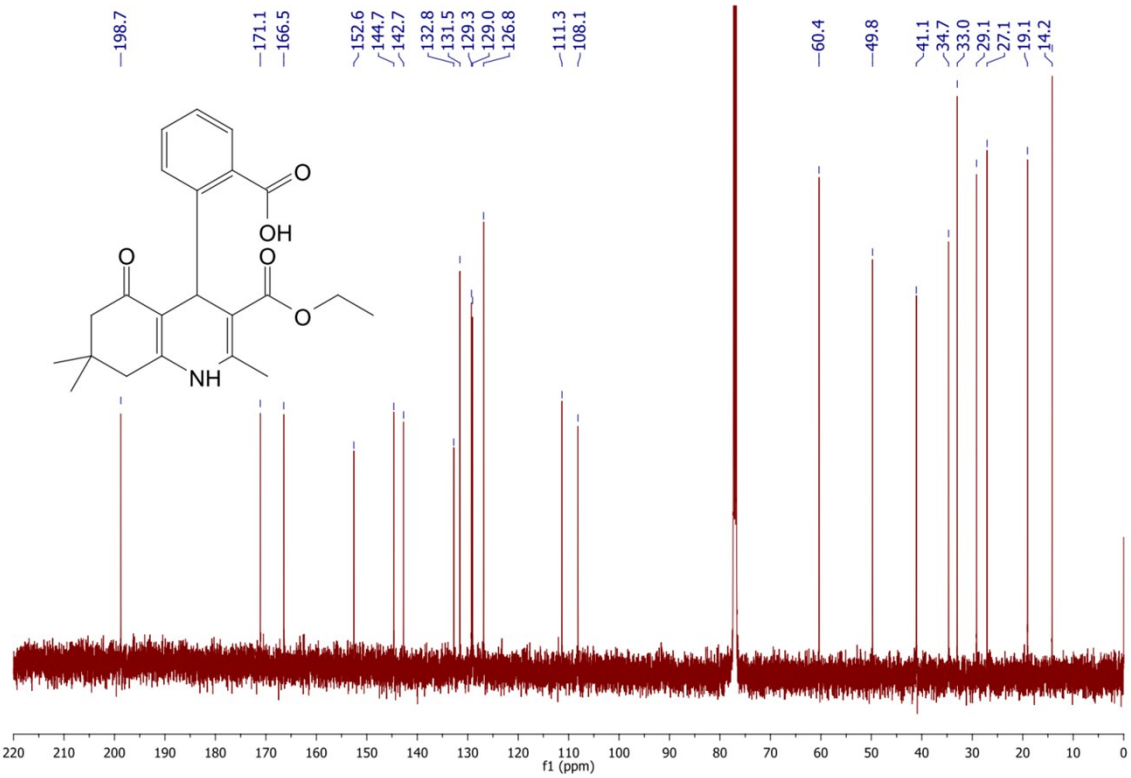
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(2-hydroxy-5-nitrophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (9)

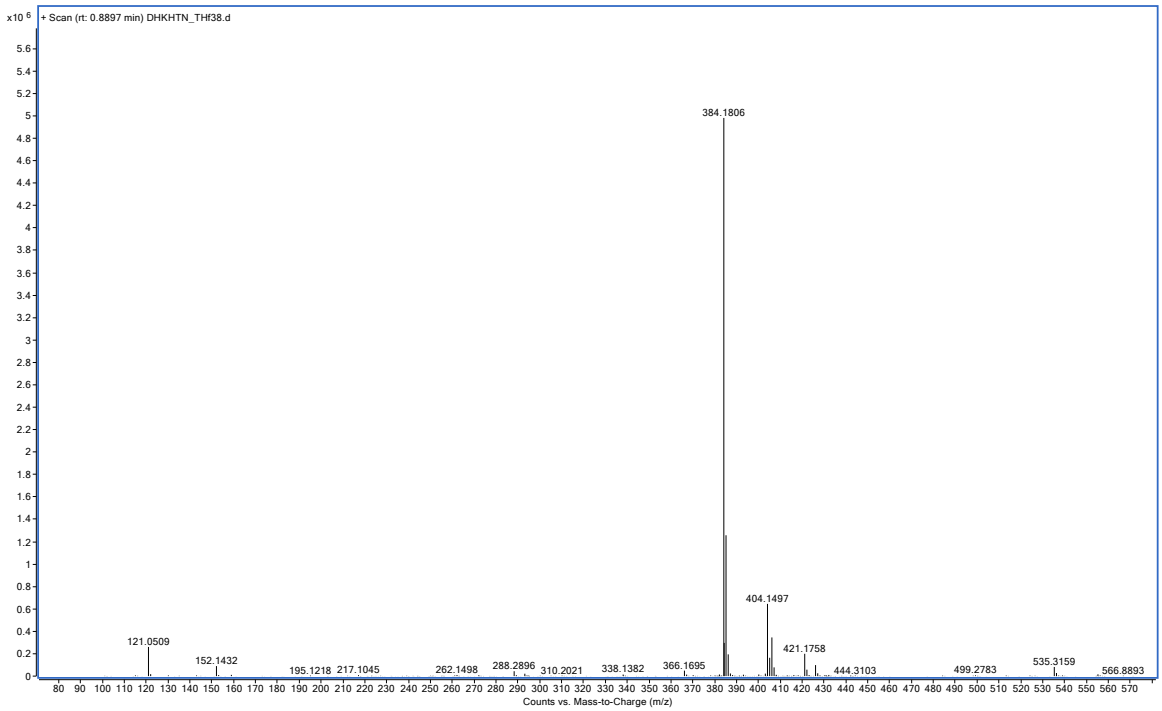




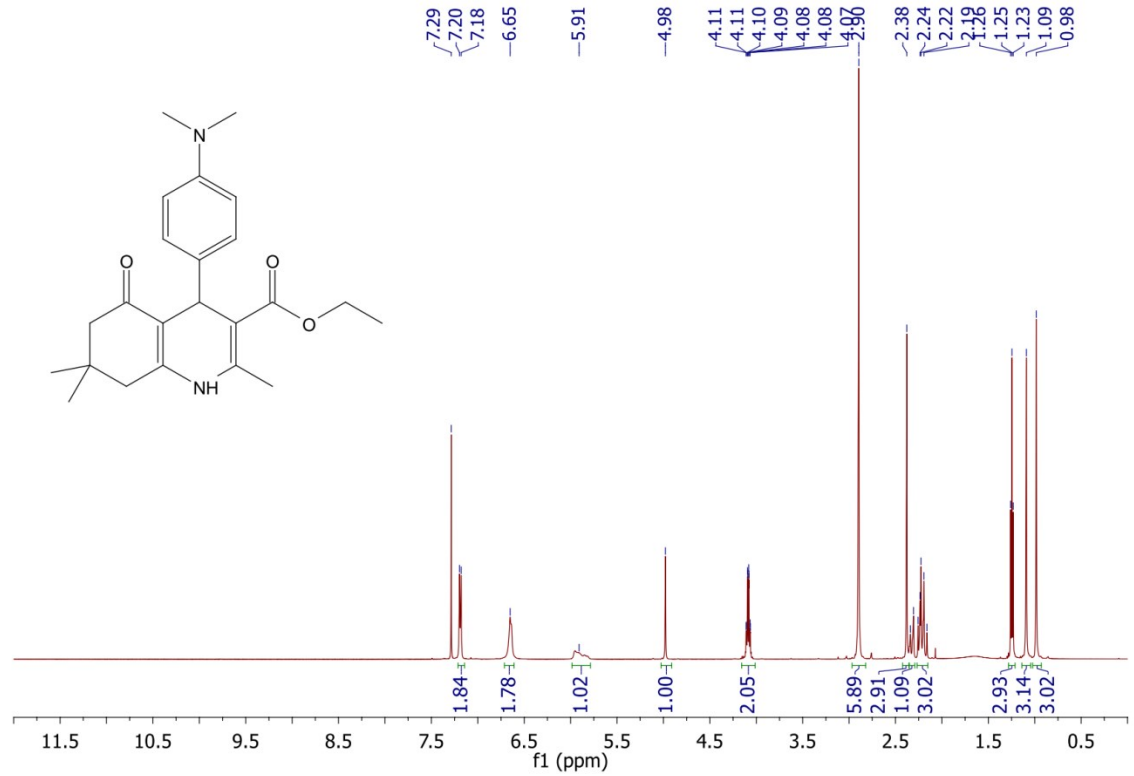
^{1}H, ^{13}C NMR and HR-ESI-MS spectroscopy of ethyl 4-(2-carboxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (10)

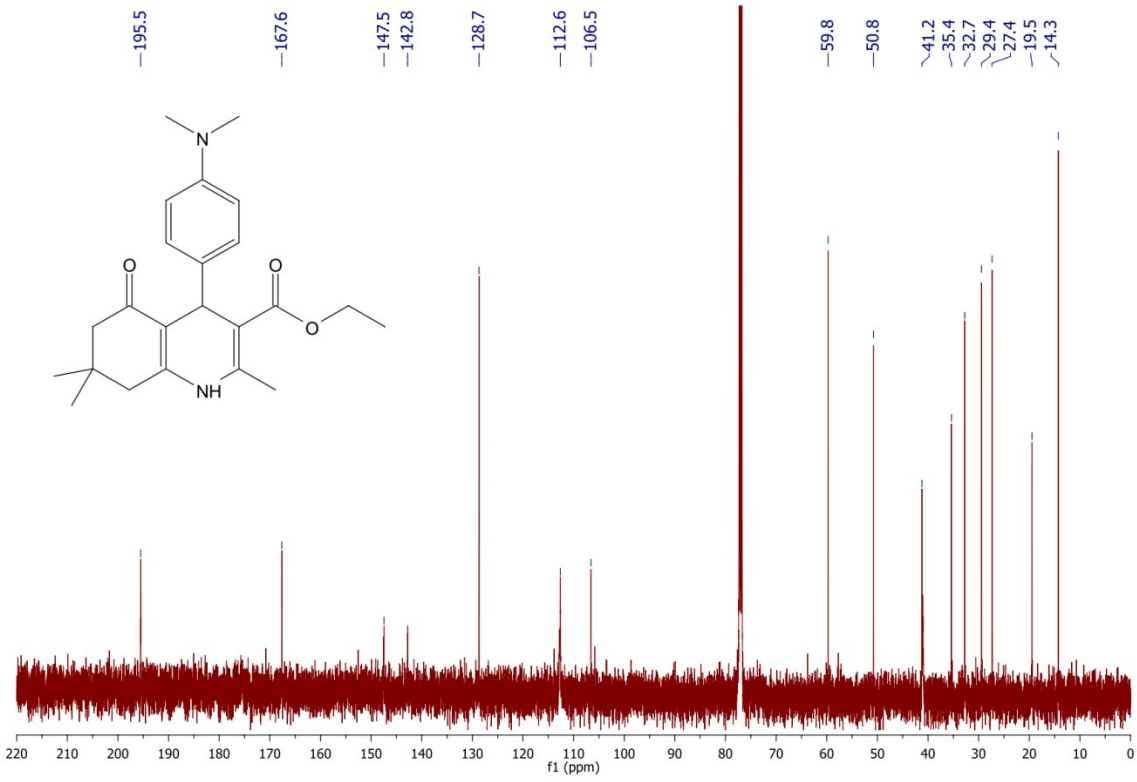




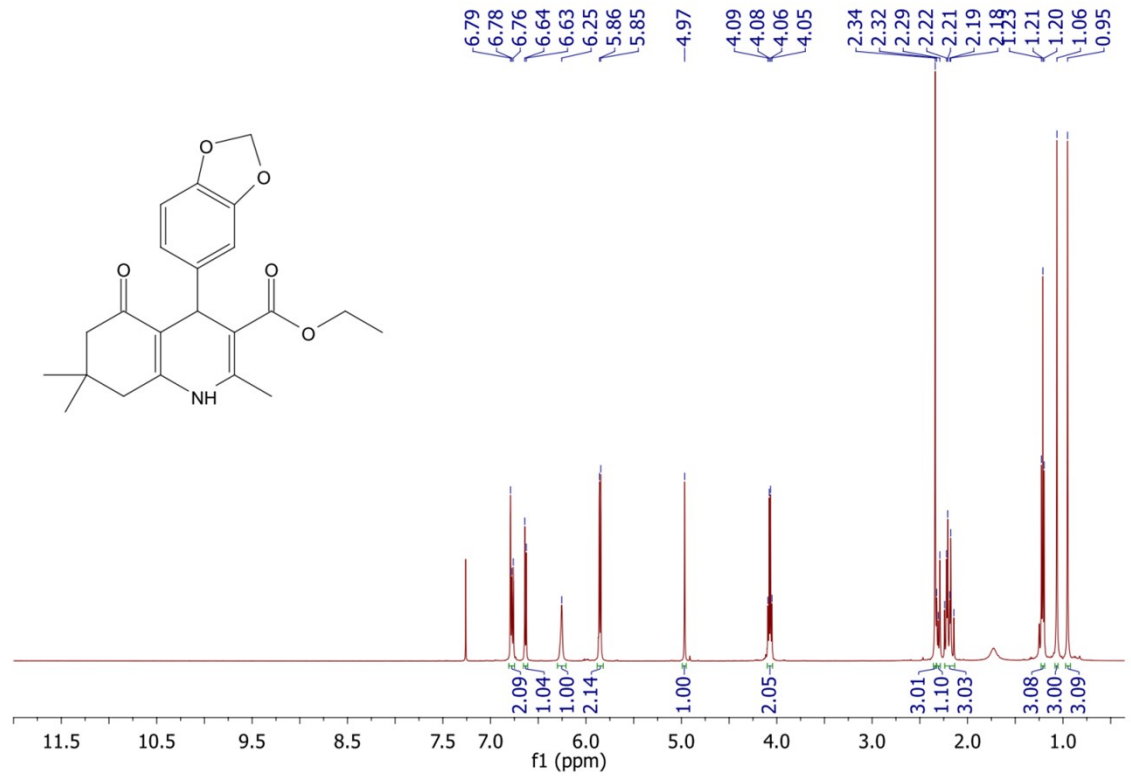

x10 6
+ Scan (rt: 0.8897 min) DHKHTN_THf38.d
384.1806
404.1497
121.0509
152.1432
421.1758
195.1218
217.1045
262.1498
288.2896
310.2021
338.1382
366.1695
444.3103
499.2783
535.3159
566.8893
Counts vs. Mass-to-Charge (m/z)

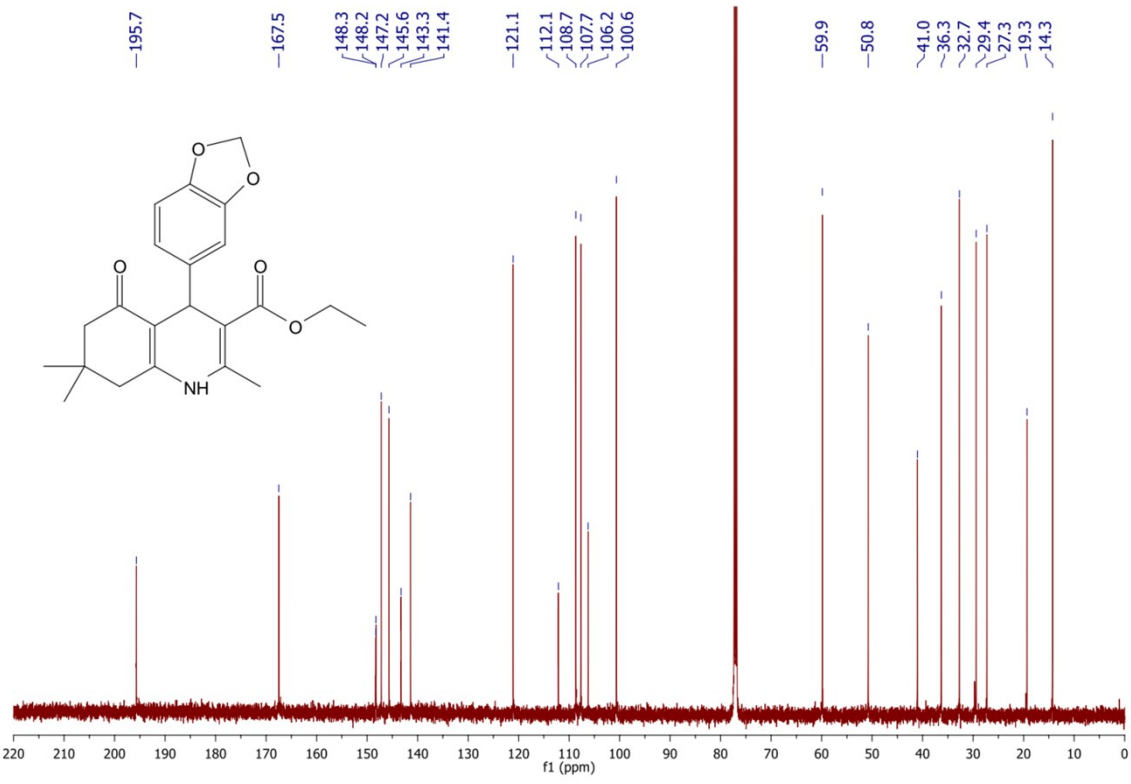
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(4-dimethylamino)phenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (11)



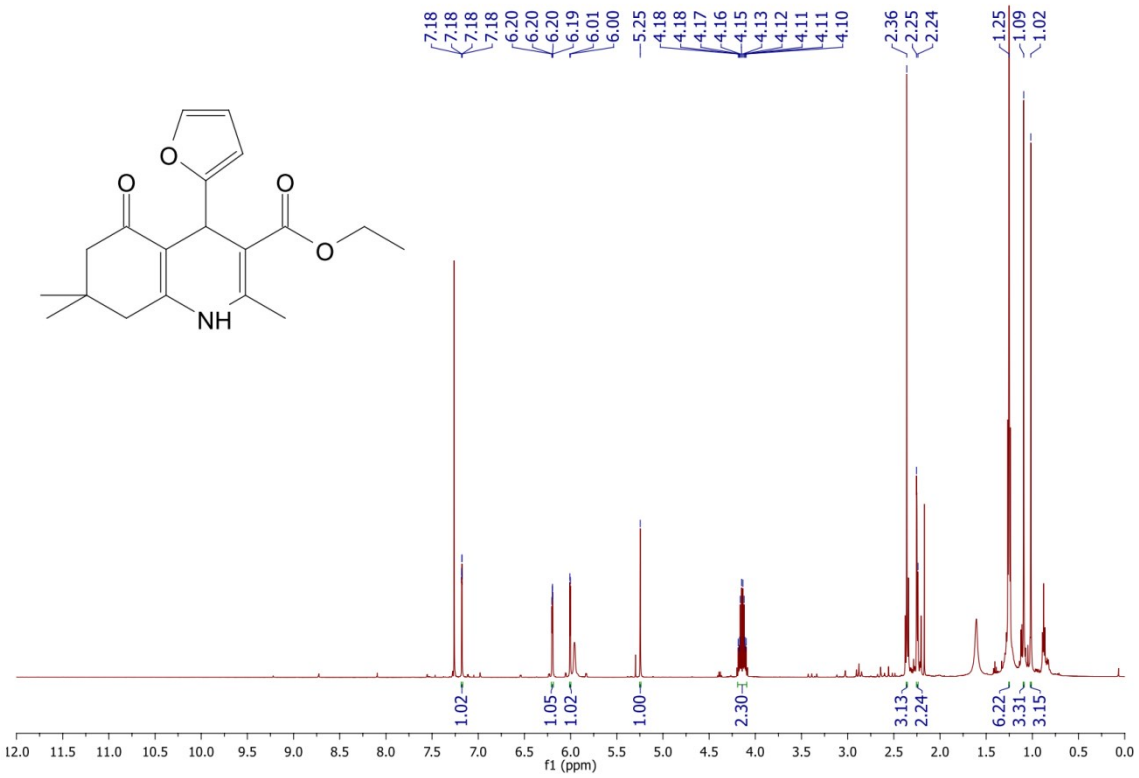


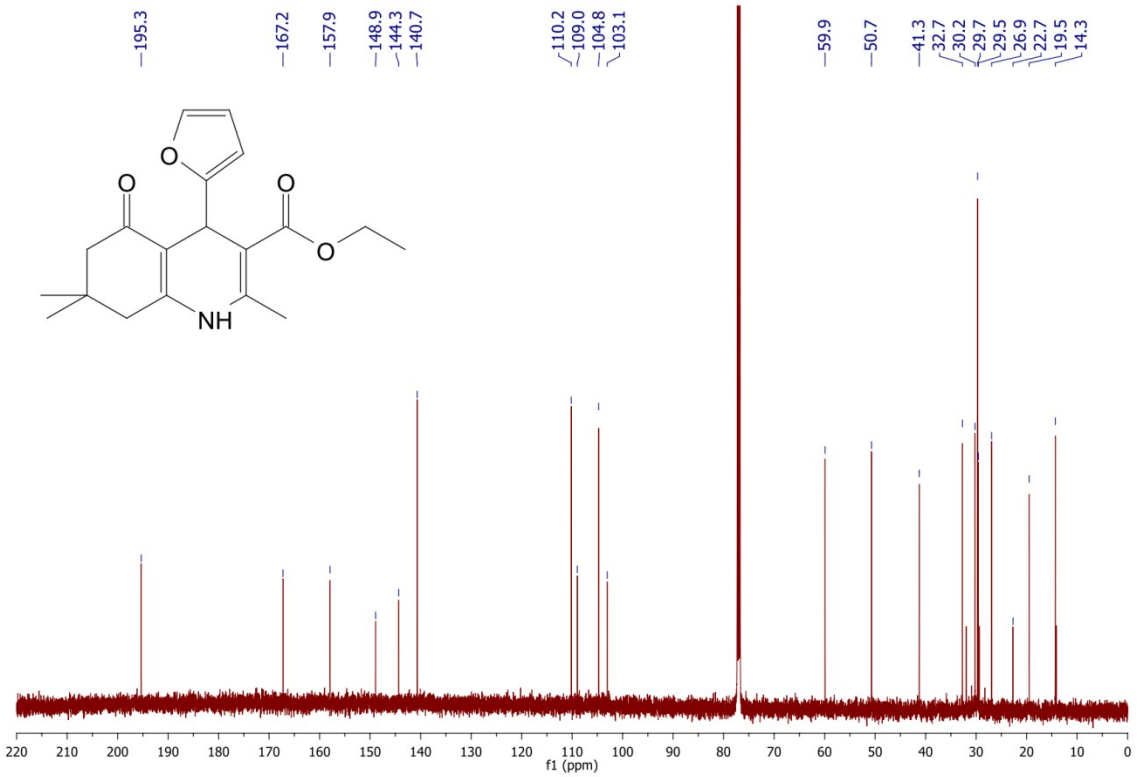
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(benzo[*d*][1,3]dioxol-5-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-3-carboxylate (12)



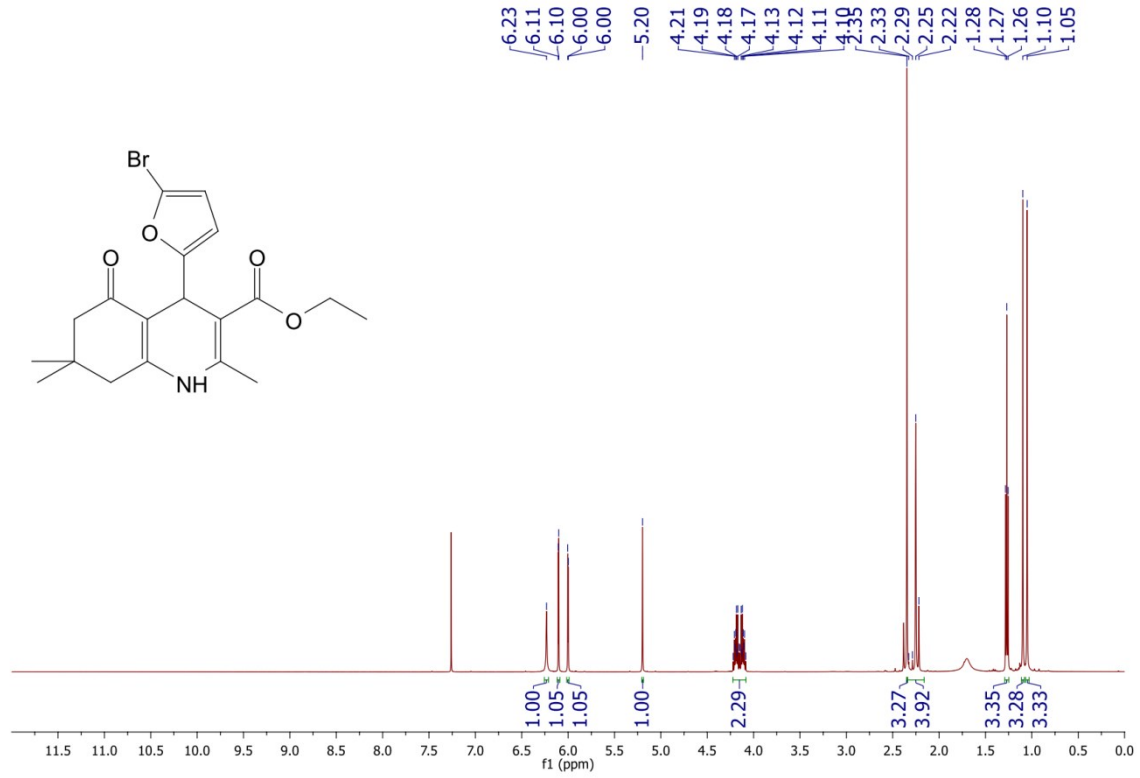


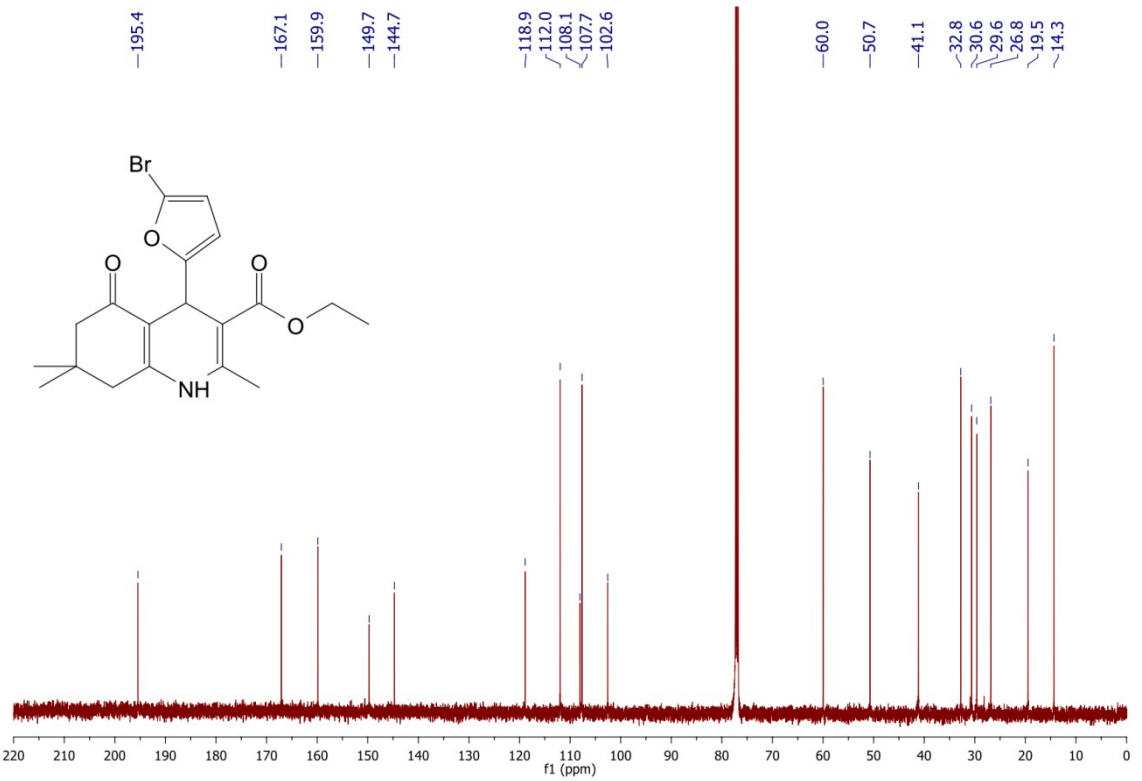
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(furan-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (13)

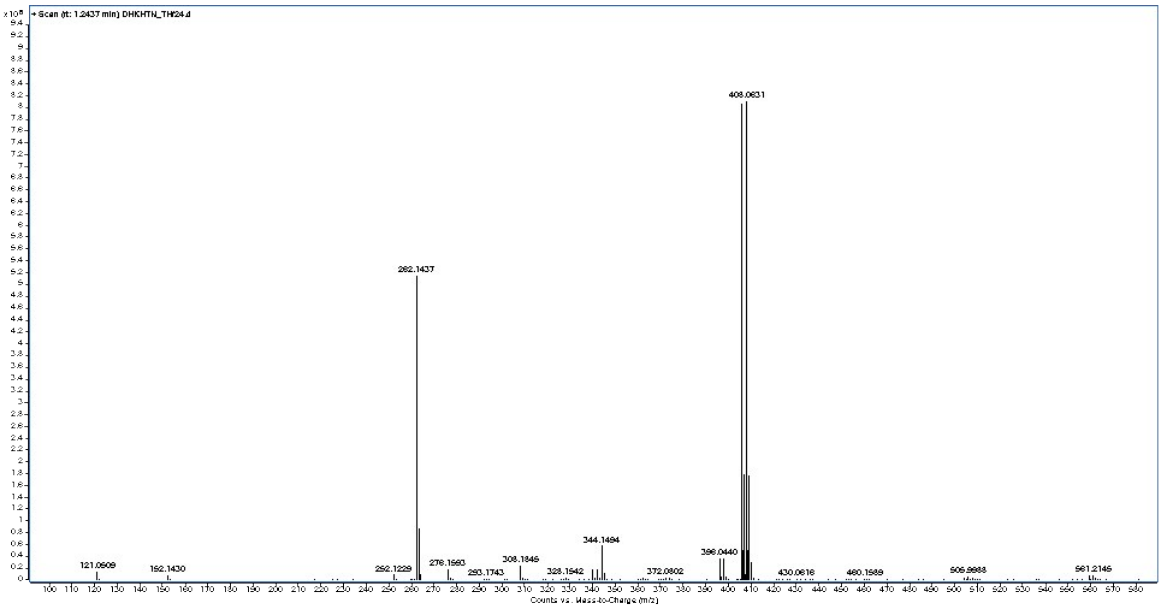




^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(5-bromofuran-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (14)



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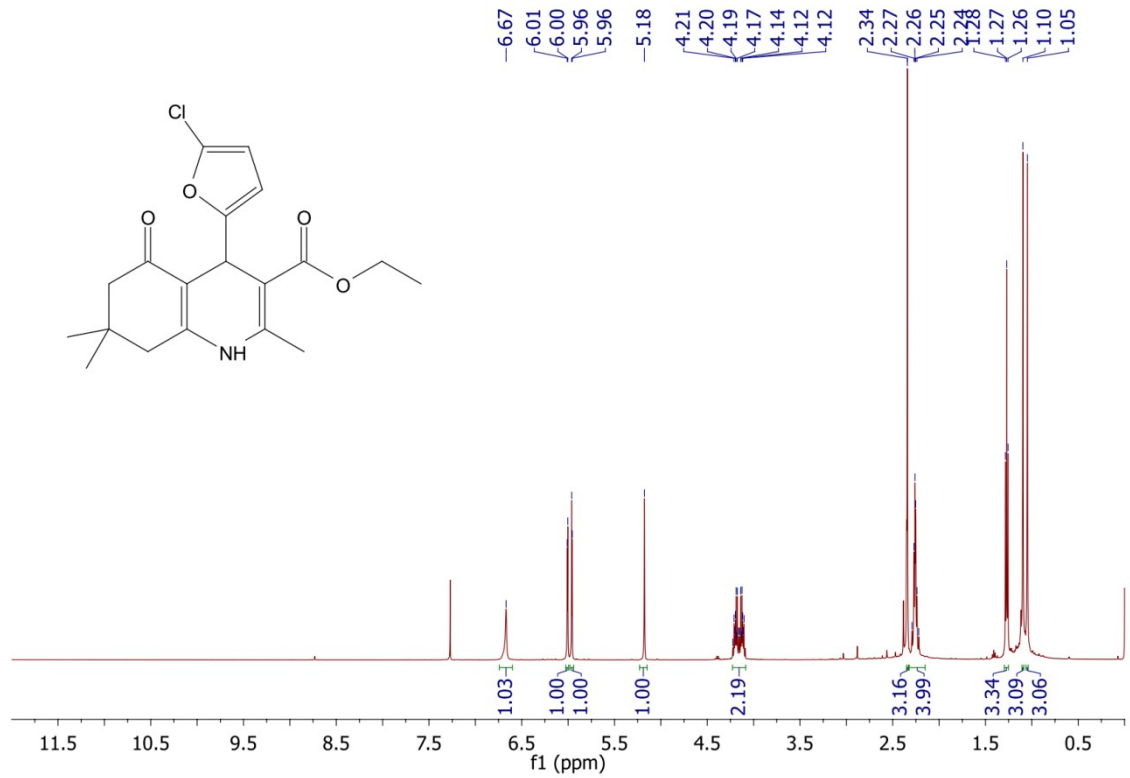
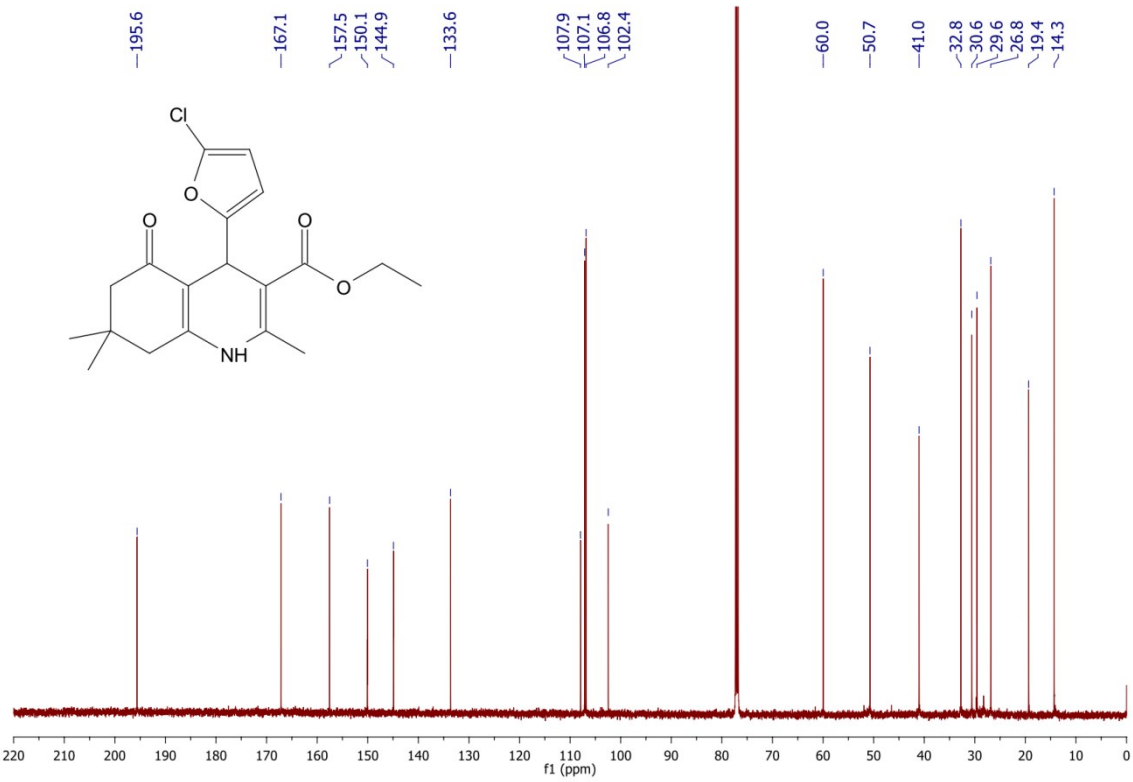
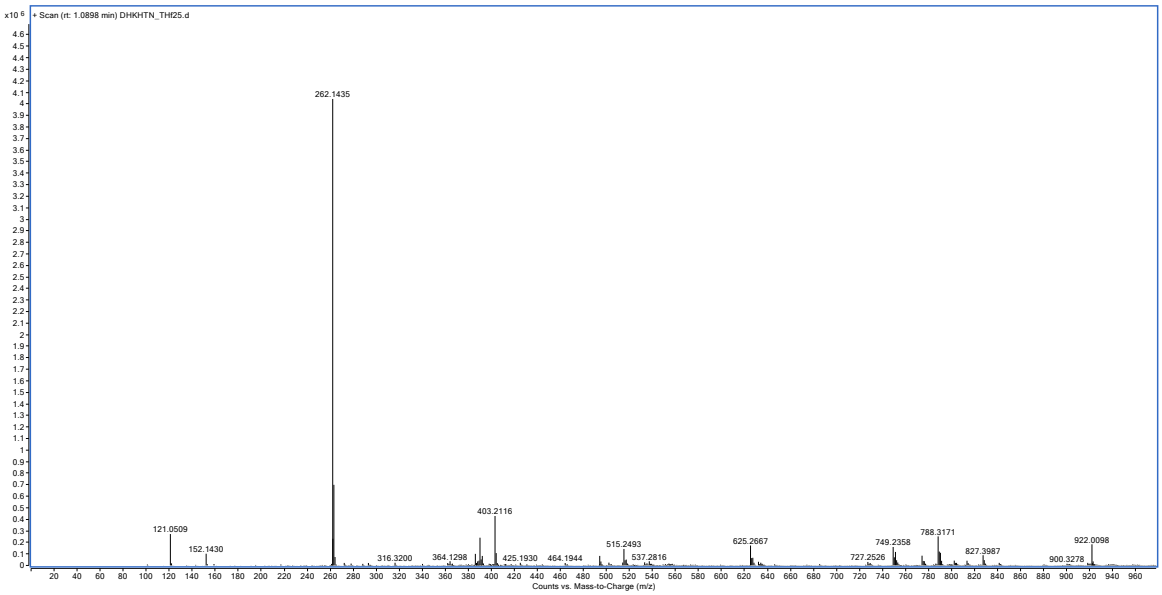
408.0831
282.1437
344.1494
396.0440
121.0509
162.1430
252.1229
278.1560
293.1743
308.1846
328.1642
372.0802
430.0618
460.1589
505.9988
561.2145
Counts vs. Mass-to-Charge (m/z)

^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(5-chlorofuran-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (15)



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+ Scan (rt: 1.0898 min) DHKHTN_THf25.d
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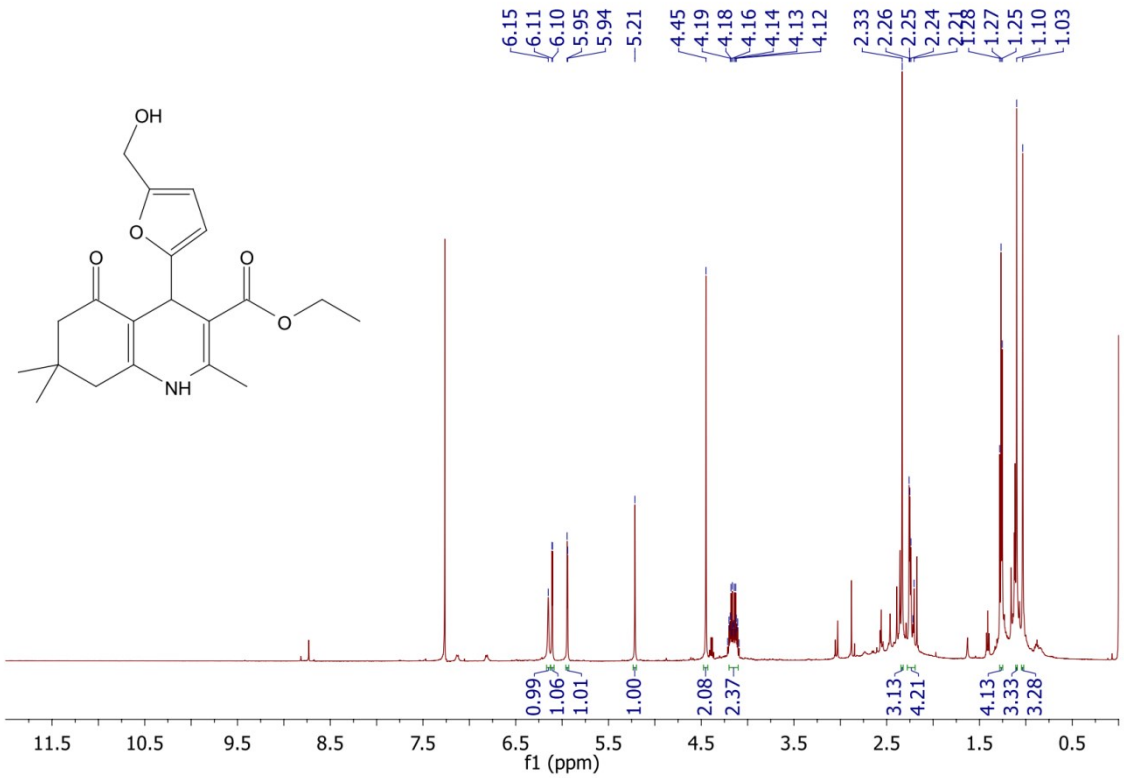
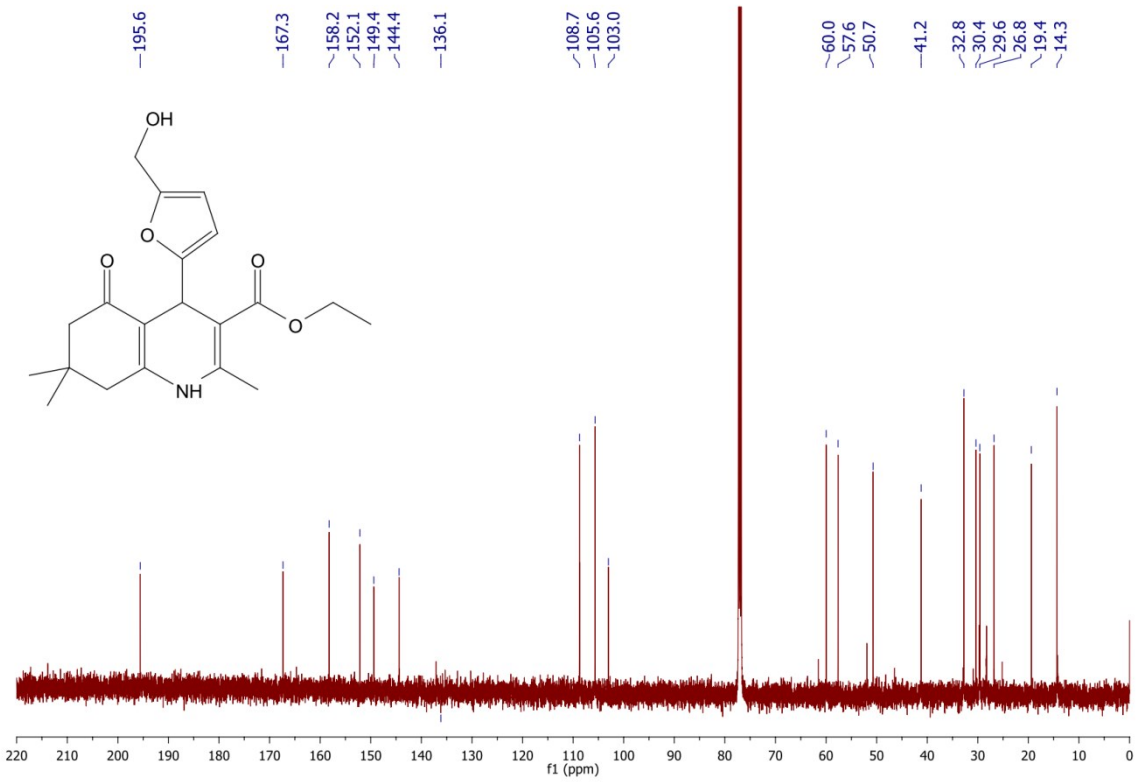
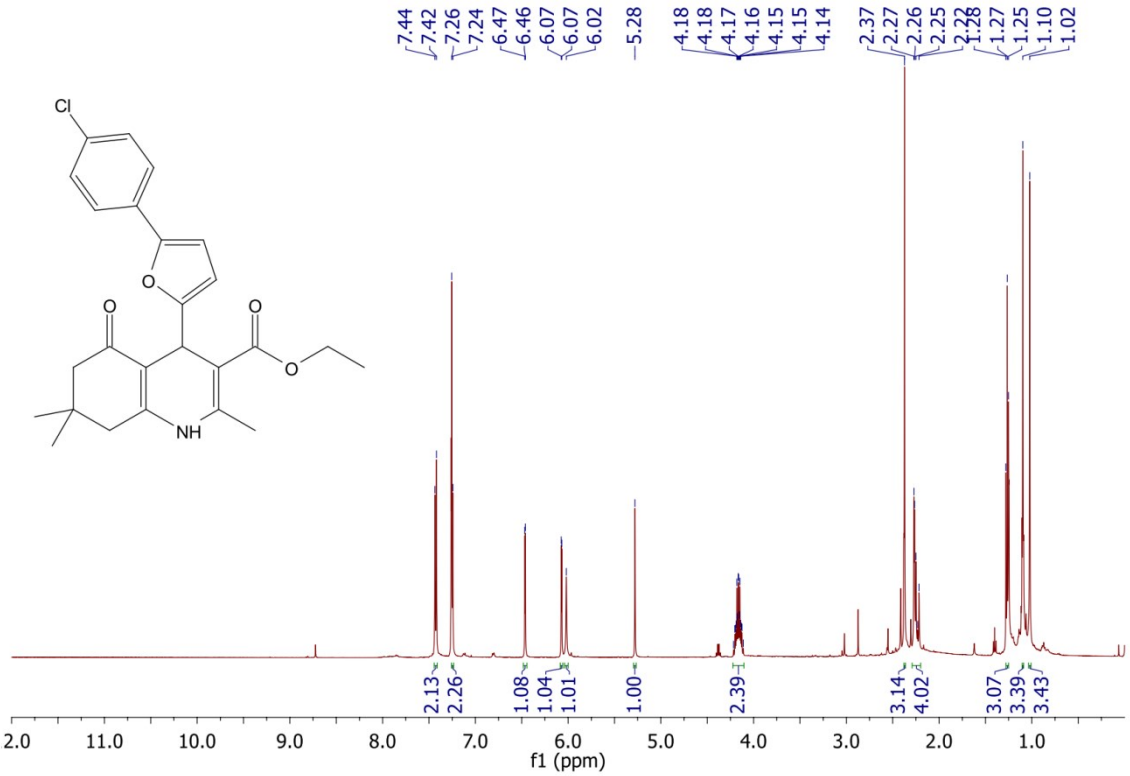
152.1430
316.3200
364.1298
403.2116
425.1930
464.1944
515.2493
537.2816
625.2667
727.2526
749.2358
788.3171
827.3987
900.3278
922.0098
Counts vs. Mass-to-Charge (m/z)

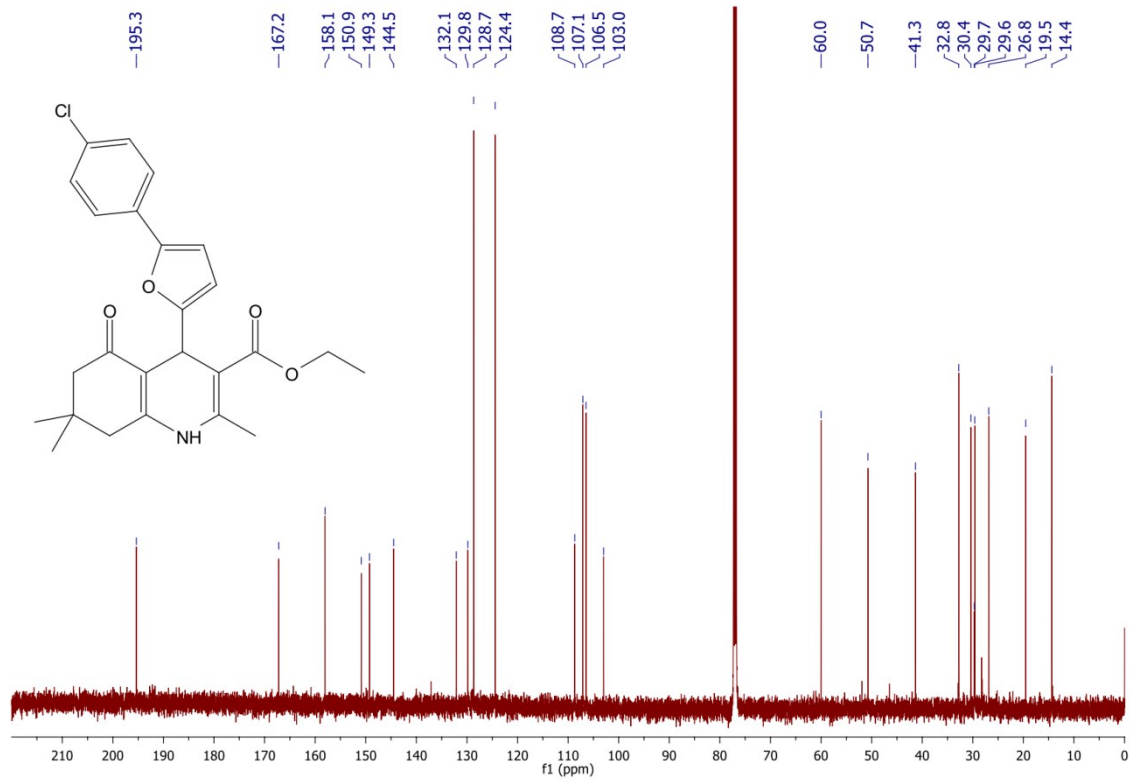
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(5-(hydroxymethyl)furan-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (16)

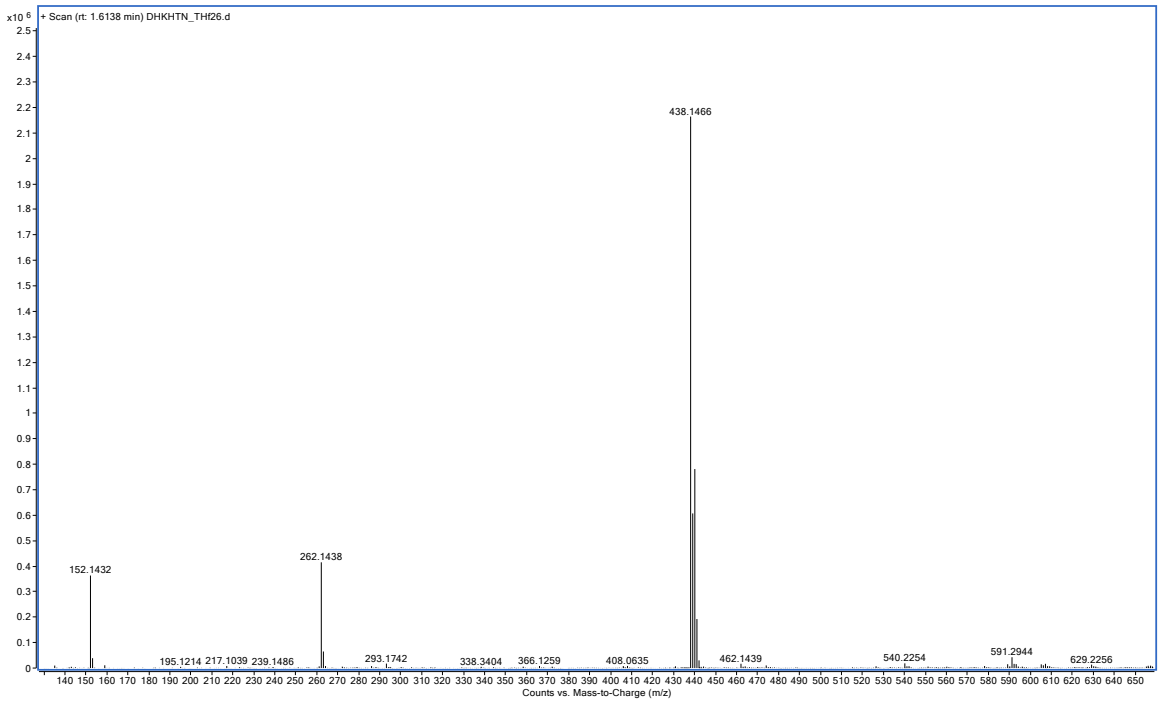




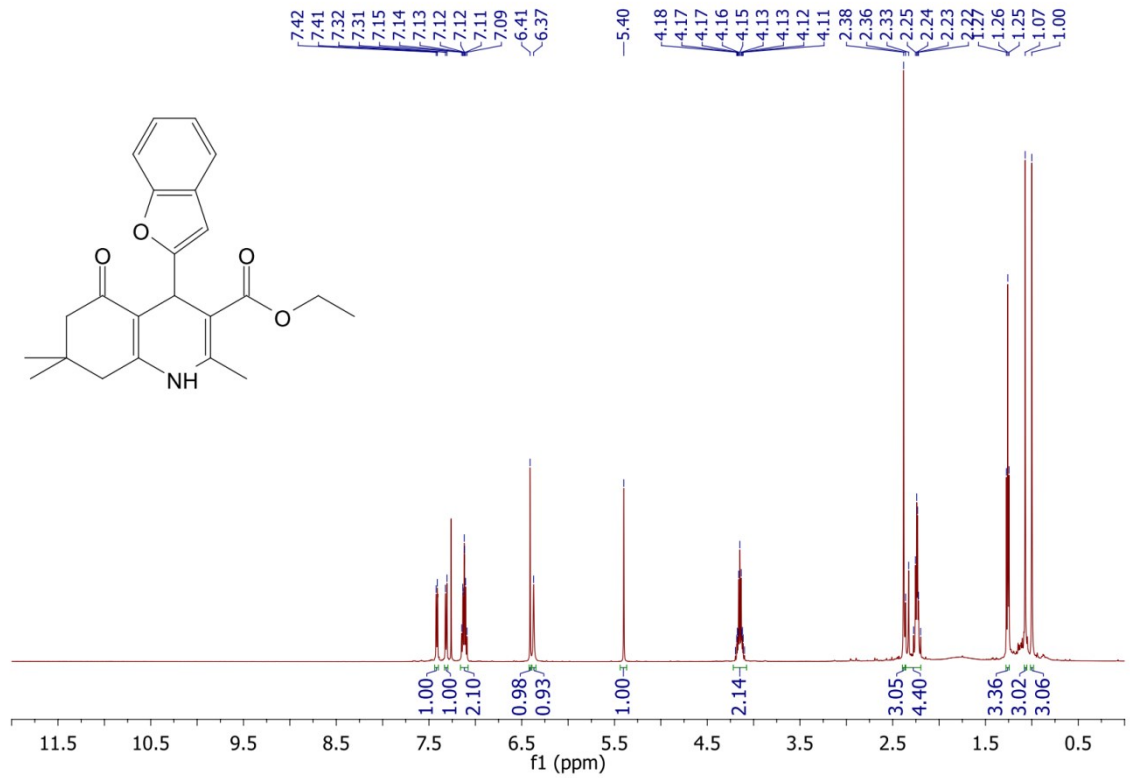
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(5-(4-chlorophenyl)furan-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (17)

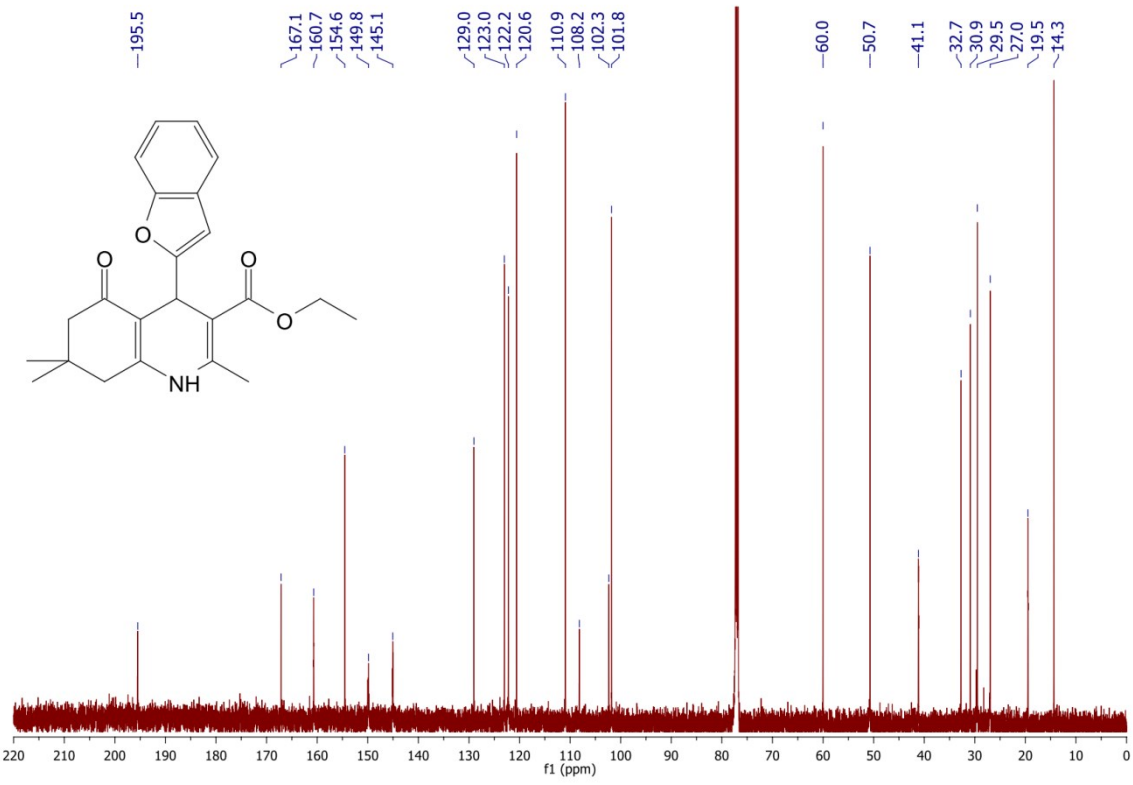


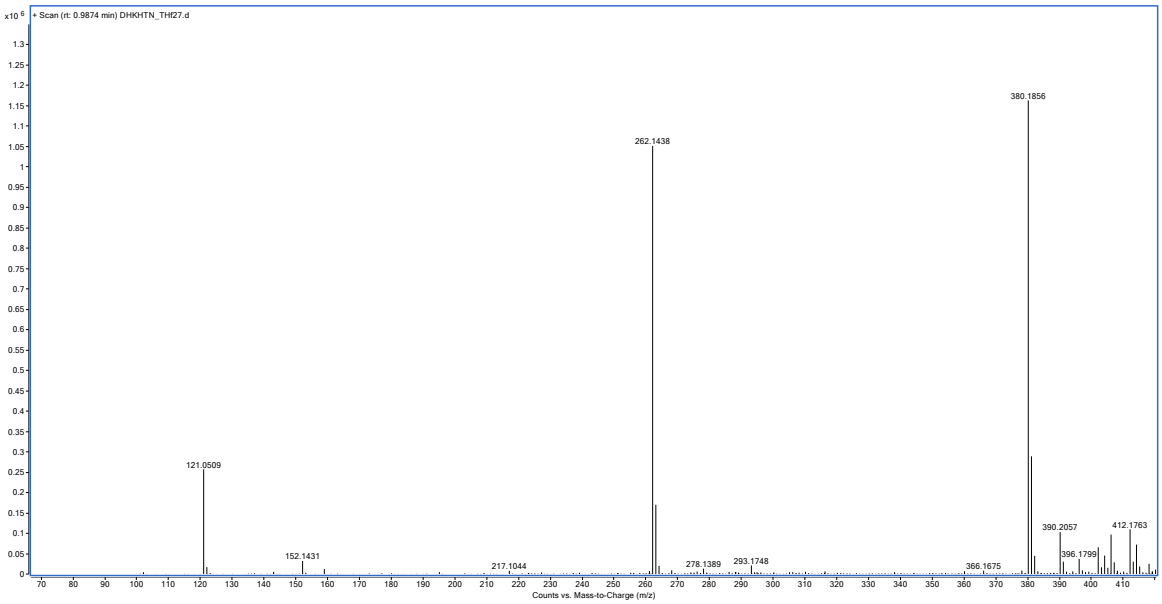



x10 6
+ Scan (rt: 1.6138 min) DHKHTN_THf26.d
438.1466
262.1438
152.1432
195.1214
217.1039
239.1486
293.1742
338.3404
366.1259
408.0635
462.1439
540.2254
591.2944
629.2256
Counts vs. Mass-to-Charge (m/z)

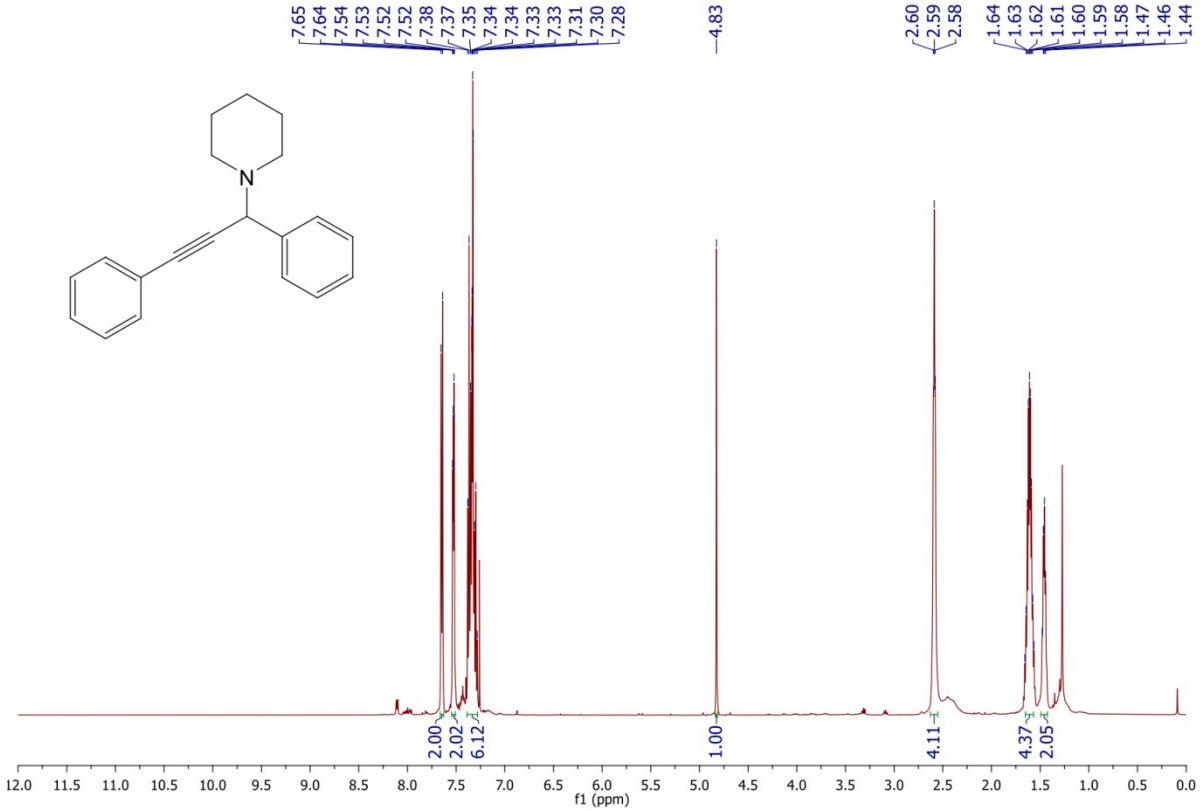
^{1}H and ^{13}C NMR spectroscopy of ethyl 4-(benzofuran-2-yl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (18)

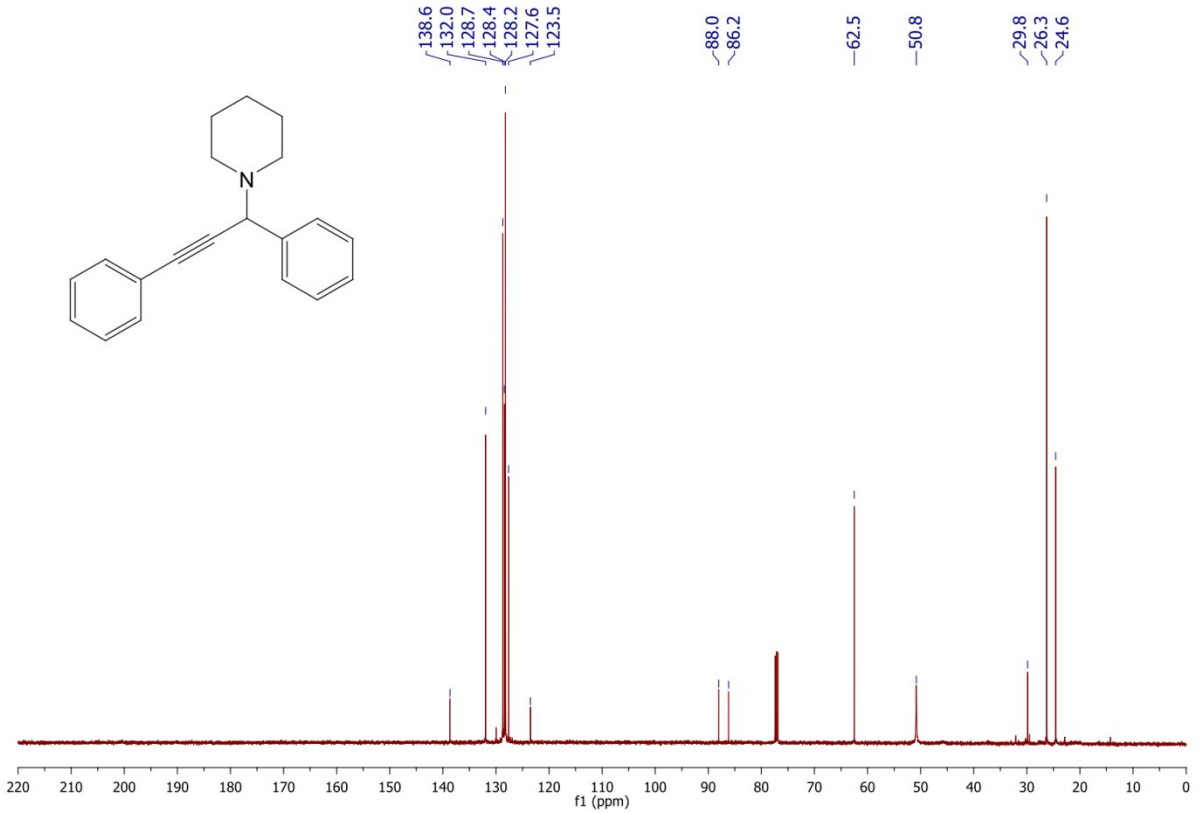




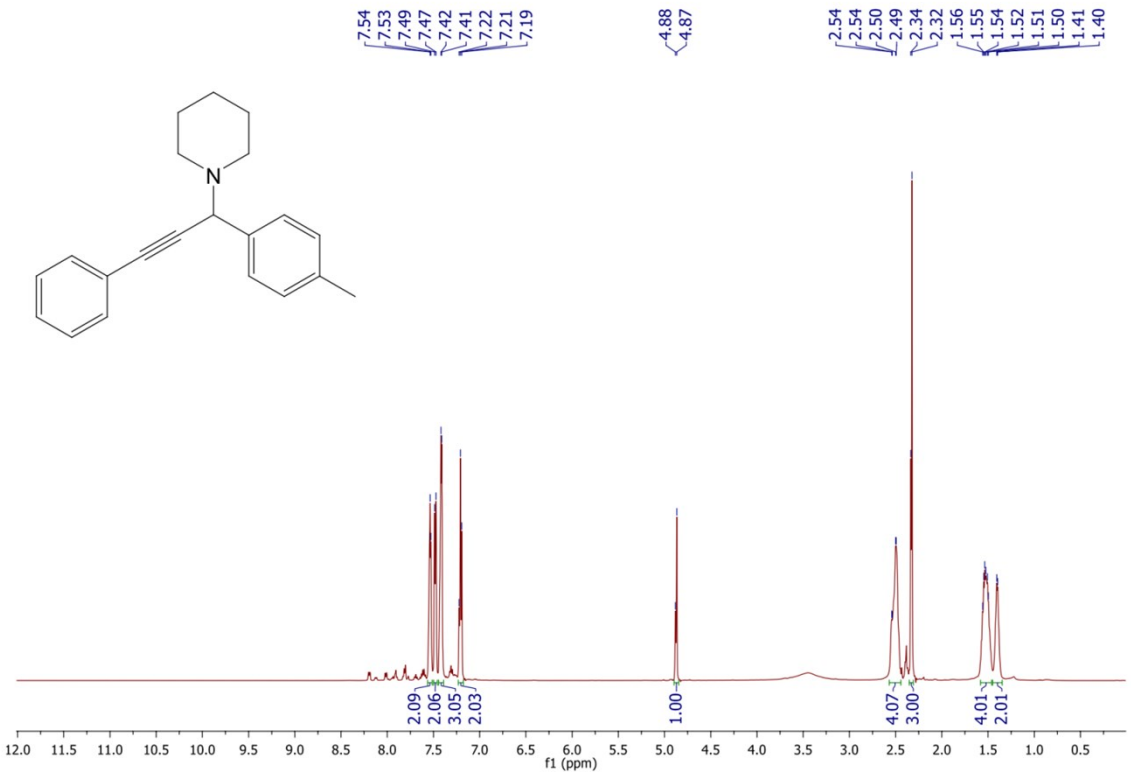

+ Scan (rt: 0.9874 min) DHKHTN_THf27.d
x10 6
380.1856
262.1438
121.0509
390.2057
412.1763
152.1431
396.1799
217.1044
278.1389
293.1748
366.1675
Counts vs. Mass-to-Charge (m/z)

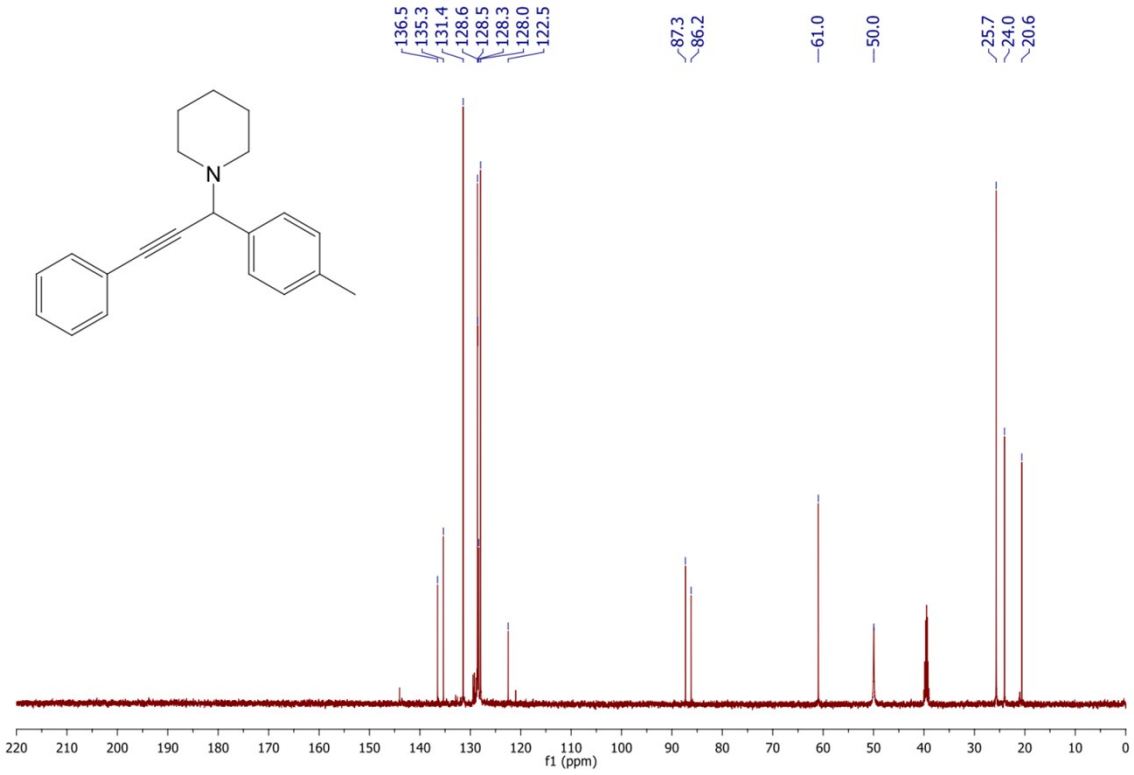
^{1}H and ^{13}C NMR spectroscopy of (1,3-diphenylprop-2-yn-1-yl)piperidine (19)



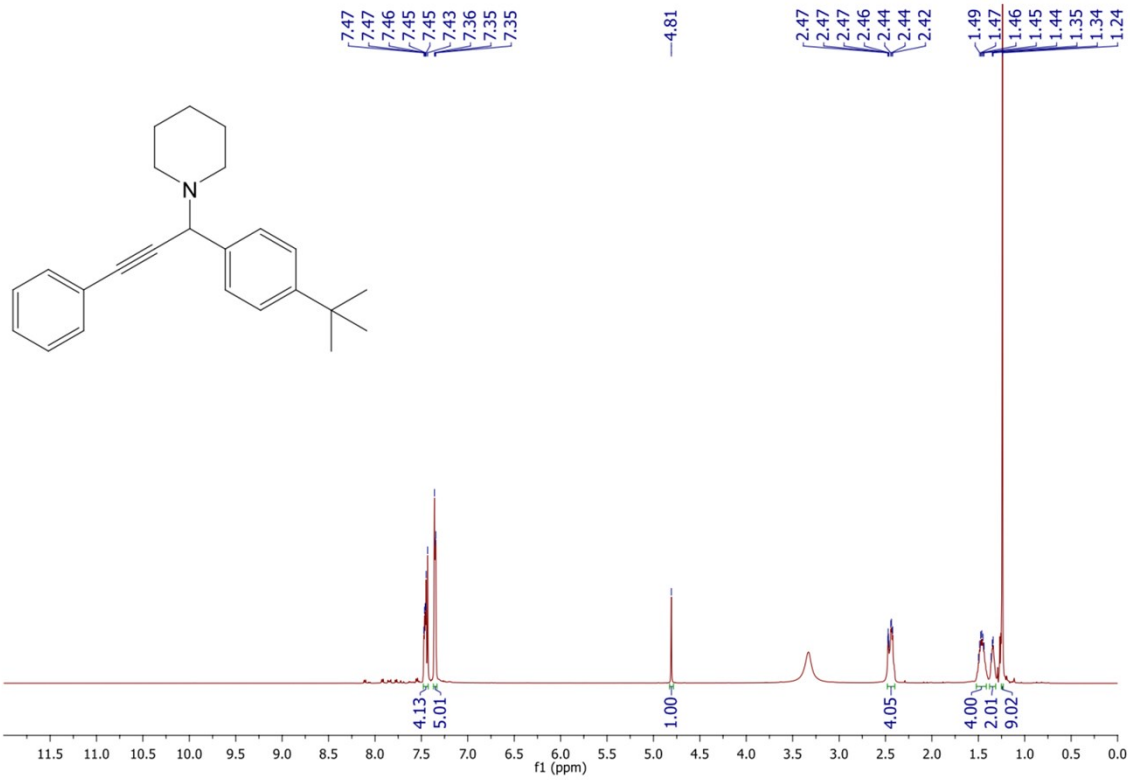


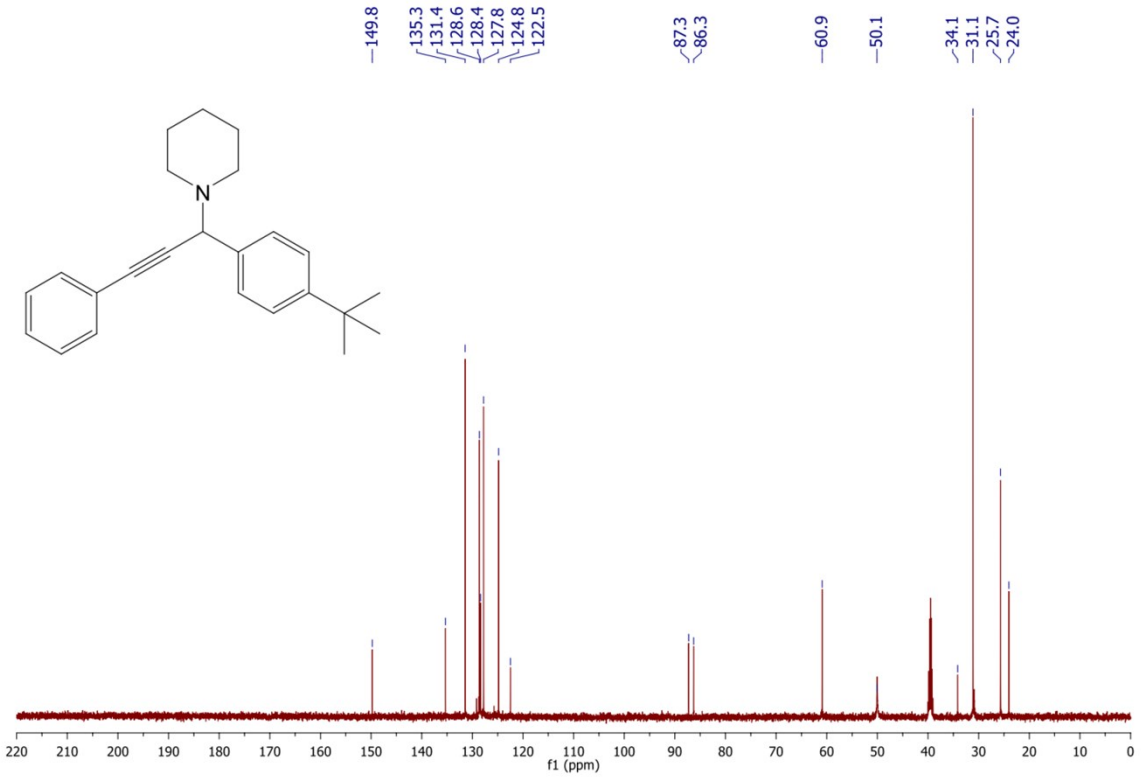
^{1}H and ^{13}C NMR spectroscopy of 1-(3-phenyl-1-(*o*-tolyl)prop-2-yn-1-yl)piperidine (20)



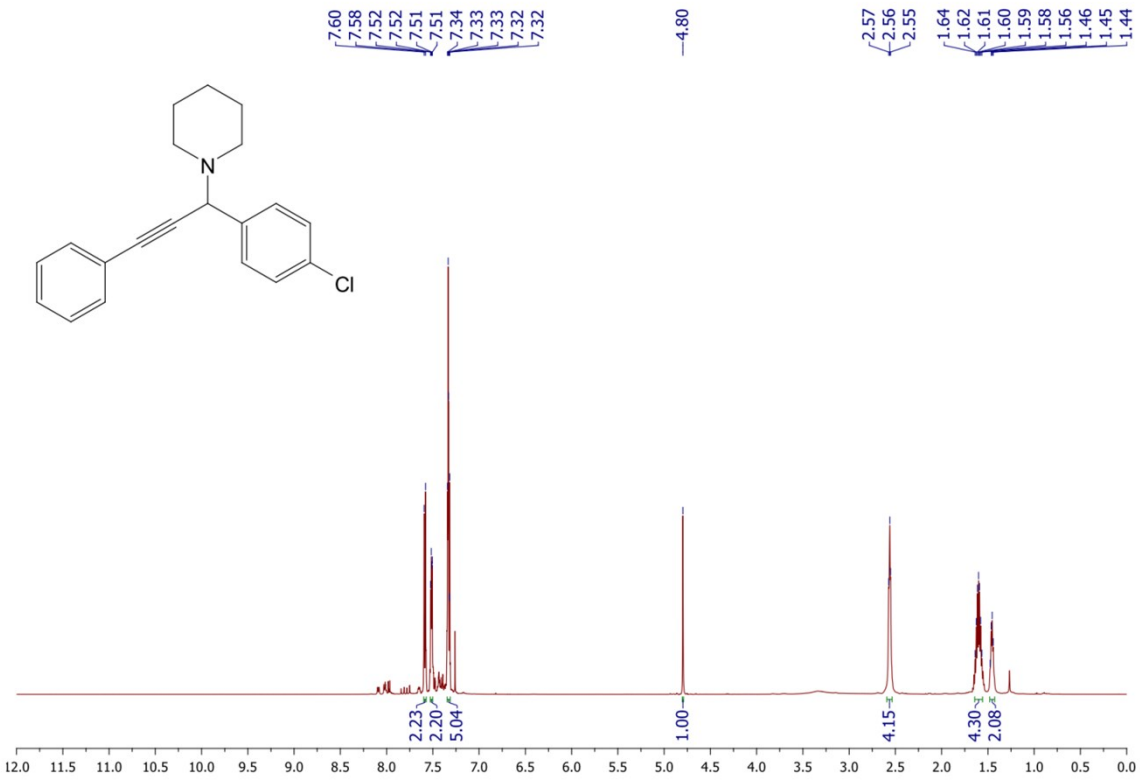


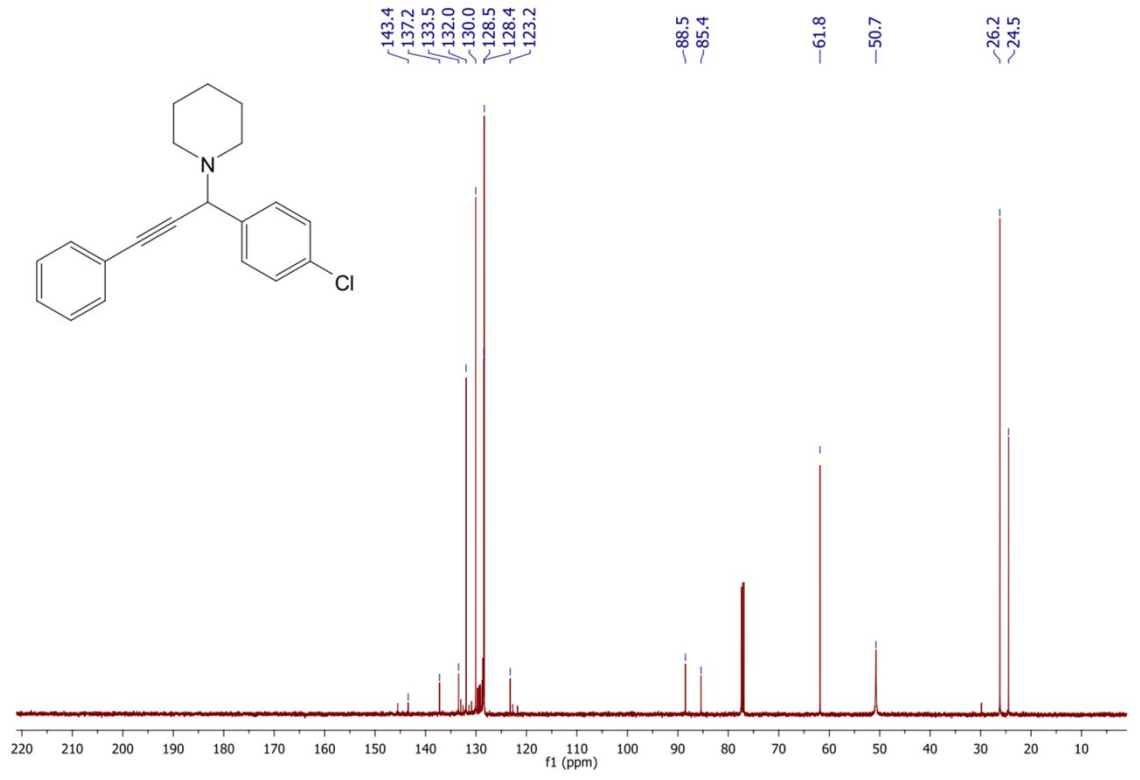
^{1}H and ^{13}C NMR spectroscopy of 1-(1-(4-(tert-butyl)phenyl)-3-phenylprop-2-yn-1-yl)piperidine (21)



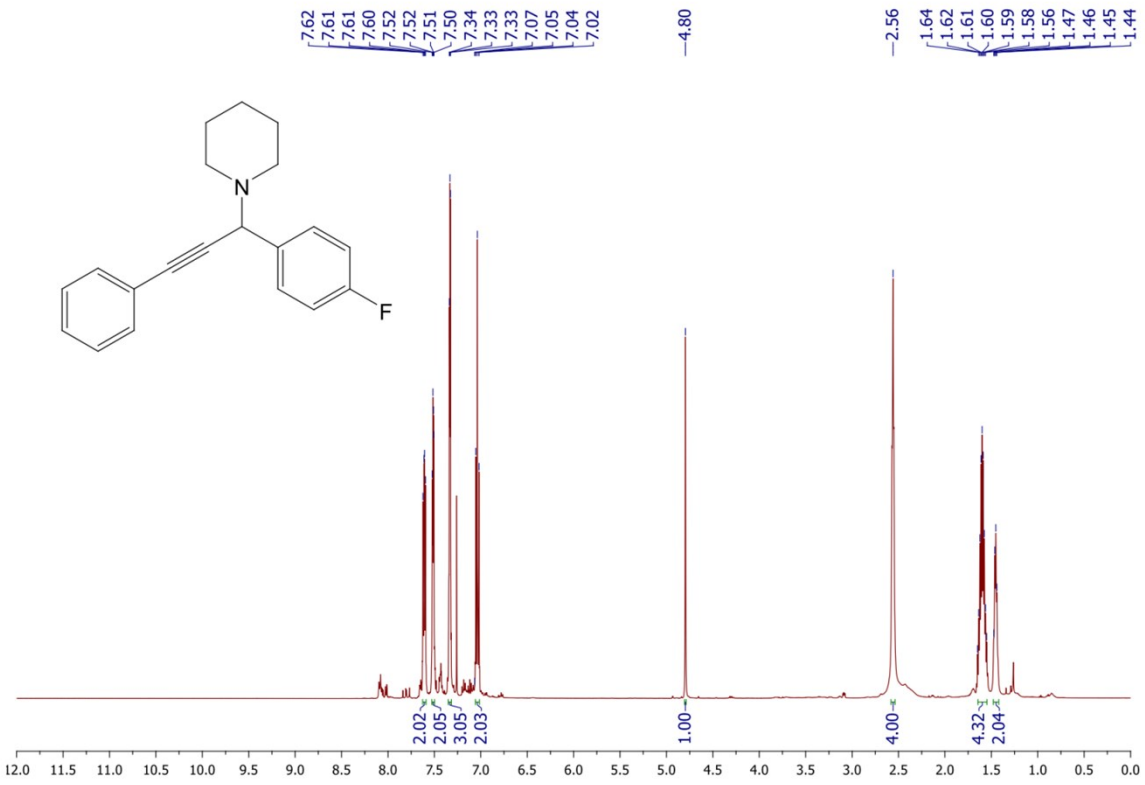


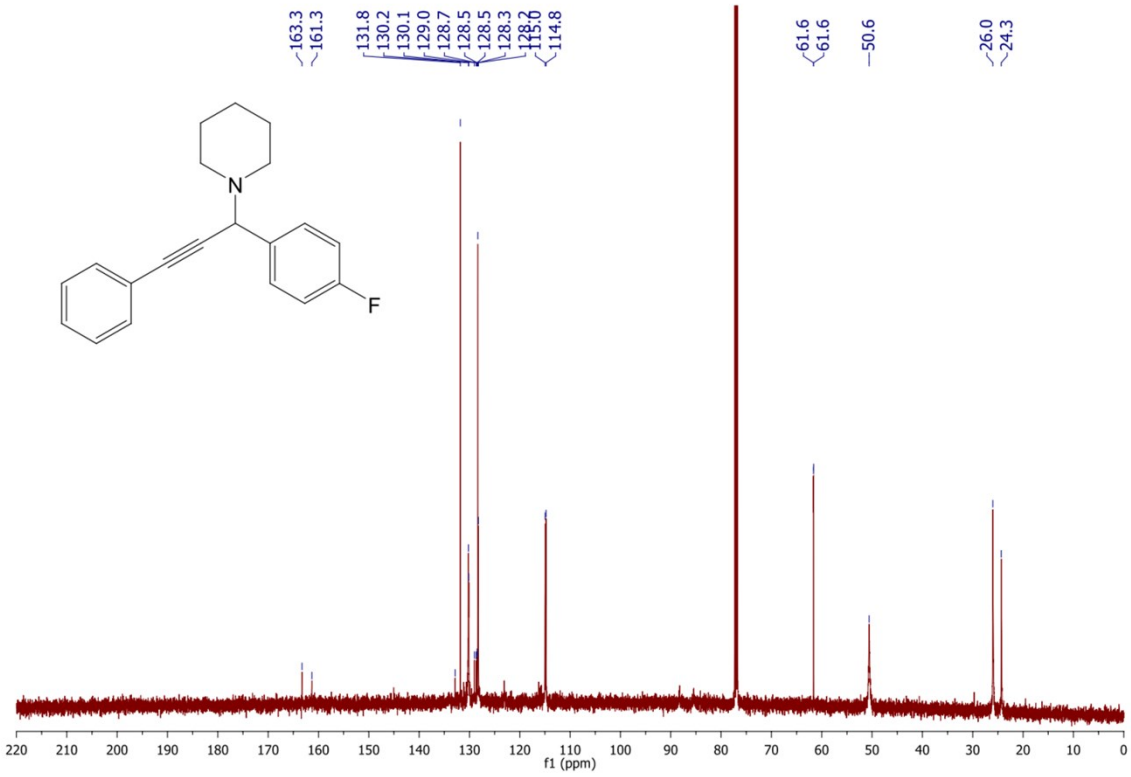
^{1}H and ^{13}C NMR spectroscopy of 1-(1-(4-chlorophenyl)-3-phenylprop-2-yn-1-yl)piperidine (22)



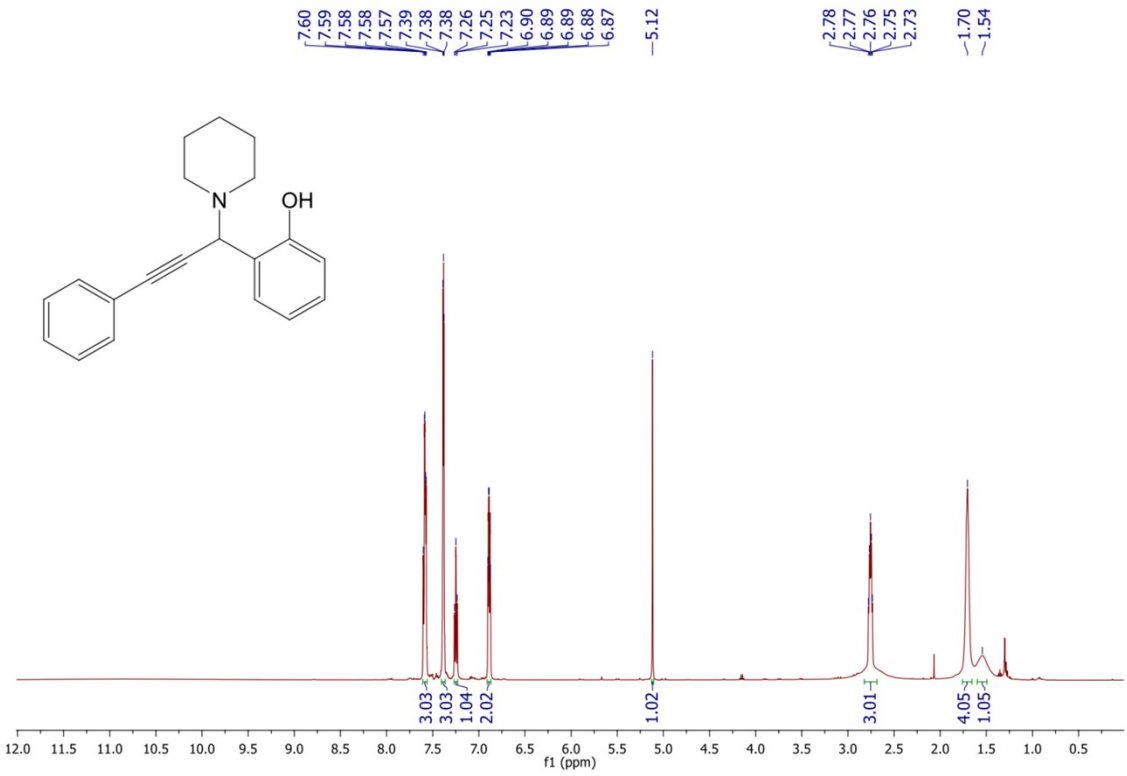


^{1}H and ^{13}C NMR spectroscopy of 1-(1-(4-fluorophenyl)-3-phenylprop-2-yn-1-yl)piperidine (23)

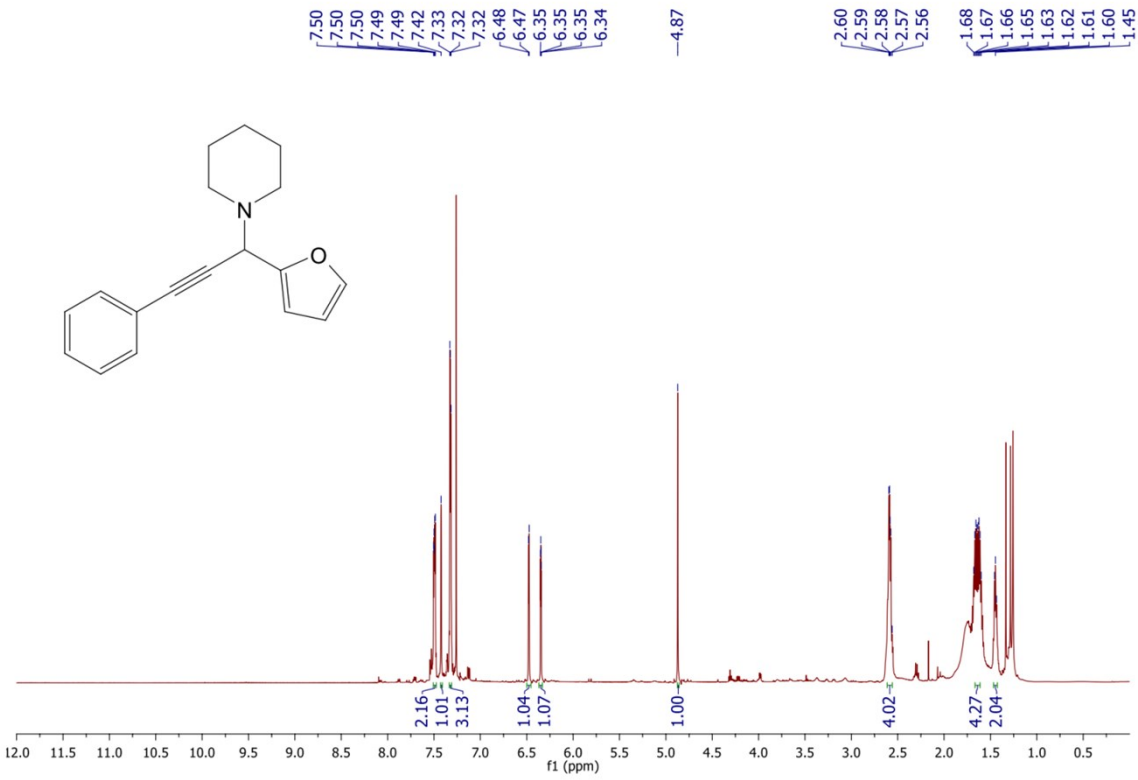


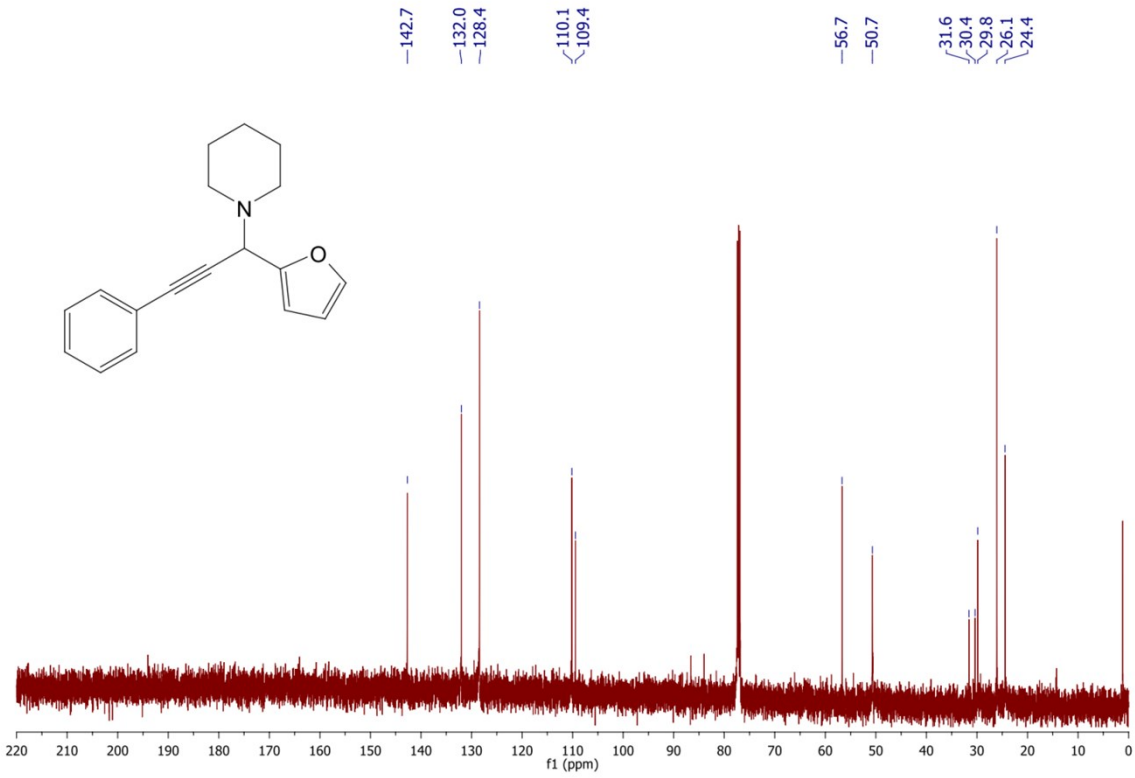


^{1}H NMR spectroscopy of ethyl 1-(1-(2-hydroxyphenyl)-3-phenylprop-2-in-1-yl)piperidine

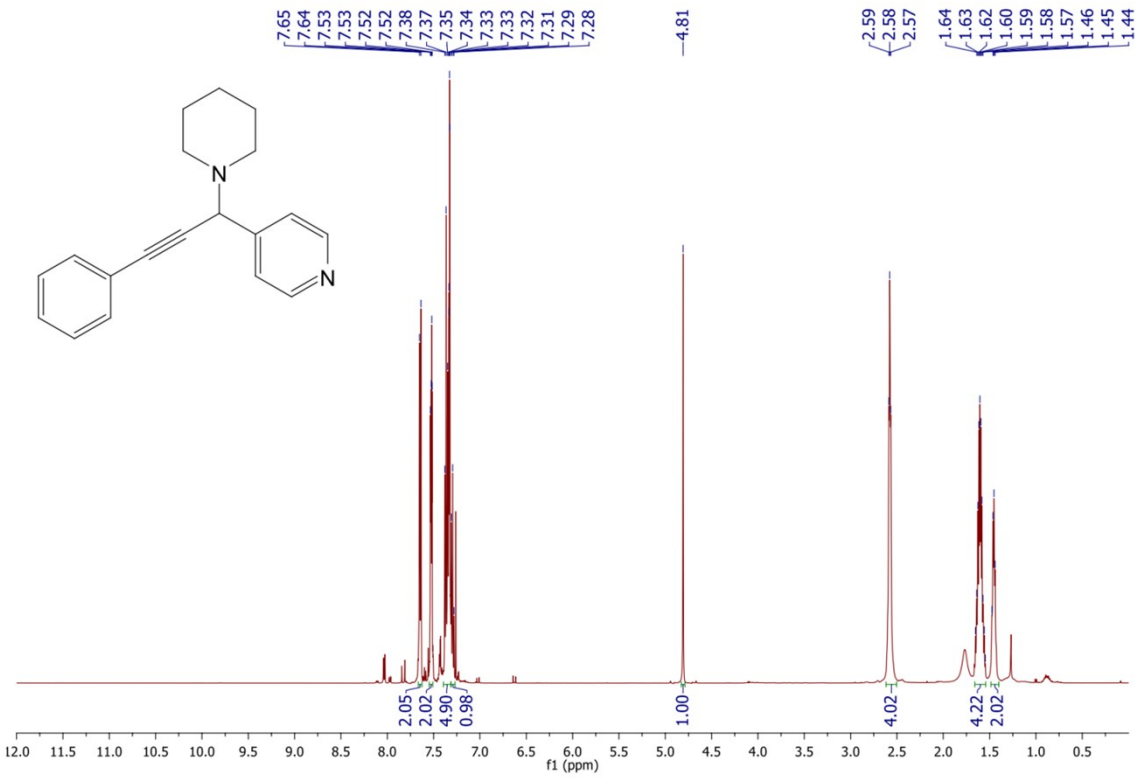


^{1}H and ^{13}C NMR spectroscopy of ethyl 1-(1-(furan-2-yl)-3-phenylprop-2-in-1-yl)piperidine





^{1}H NMR spectroscopy of ethyl 4-(3-phenyl-1-(piperidine-1-yl)prop-2-in-1-yl)pyridine



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