

Supporting Information

Visible-light- and bromide-mediated photoredox Minisci alkylation of *N*-heteroarenes with ester acetates

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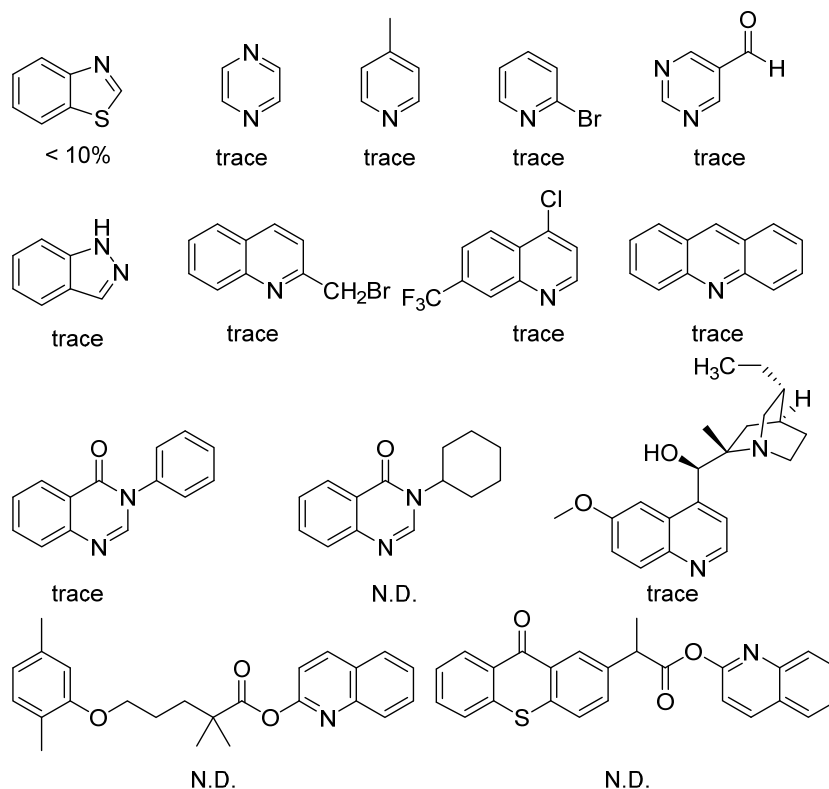
1. General information

The reaction via general procedure was carried out under an atmosphere of argon unless otherwise noted. Column chromatography was performed using silica gel (200-300 mesh) or thin layer chromatography was performed using silica gel (GF254). ^1H NMR and ^{13}C NMR spectra were recorded on Bruker-AV (400 and 100 MHz, respectively) instrument using CDCl_3 and $\text{DMSO-}d_6$ as solvents. Mass spectra were measured on Agilent 5975 GC-MS instrument (EI). High-resolution mass spectra (ESI) were obtained with the Thermo Scientific LTQ Orbitrap XL mass spectrometer. The structures of known compounds were further corroborated by comparing their ^1H NMR, ^{13}C NMR data and HRMS data with those in literature. Melting points were measured with a YUHUA X-5 melting point instrument and were uncorrected. All reagents were directly used without purification as received from commercial supplier.

2. General procedure for synthetic reaction

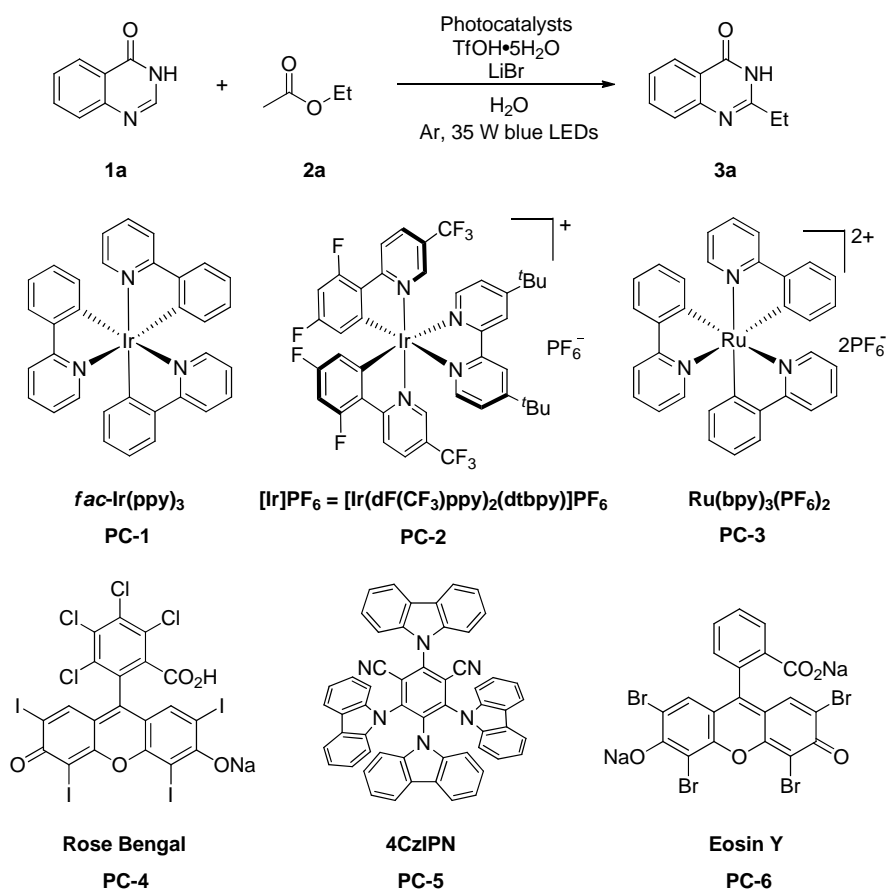
Standard reaction conditions: A 10 mL reaction vessel was charged with 4-hydroxyquinazoline (**1a**, 29.2 mg, 0.2 mmol), LiBr (8.6 mg, 0.1 mmol, 0.5 equiv), Ir[dF(CF₃)ppy]₂(dtbbpy)PF₆ (2.2 mg, 0.002 mmol), TfOH·5H₂O (5.5 M aq, 40 μL, 1.0 equiv, 0.22 mmol), H₂O (18 μL, 1.0 mmol). The atmosphere was exchanged by applying vacuum and backfilling with Ar (this process was conducted for three times), then EA (**2a**, 2mL) was added by syringe. The resulting mixture was stirred for 60 hours under irradiation with a 35 W blue LED. The crude reaction mixture was diluted using 10 mL EA, dried over sodium sulfate, filtered and the volatiles were removed under reduced pressure. Silica gel (200-300 mesh) or thin layer chromatography was performed using silica gel (GF254) to give product **3a**.

Substrates with no or very low reactivity:



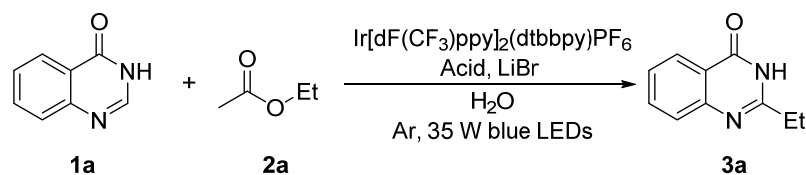
3. Optimization of Reaction Conditions

Table S1. Screening of photocatalyst^a



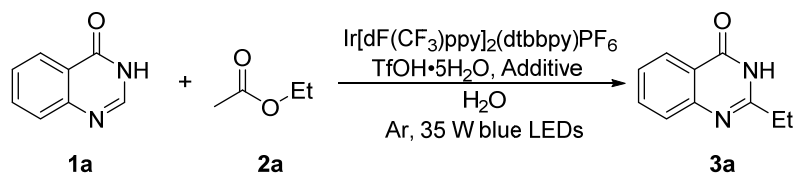
Entry	Photocatalyst	Yield (%) ^b
1	-	N.D.
2	PC-1	N.D.
3	PC-2	81
4	PC-3	N.D.
5	PC-4	N.D.
6	PC-5	N.D.
7	PC-6	N.D.
8 ^c	PC-2	N.D.

^a Reaction conditions: **1a** (0.2 mmol), **2a** (2.0 mL), Photocatalyst (1 mol %), LiBr (0.5 equiv), TfOH•5H₂O (aq., 1.0 equiv), H₂O (3.0 equiv), 60 °C under Ar, 60 h. ^b Isolated yield. ^c No light.

Table S2. Screening of Acid^a

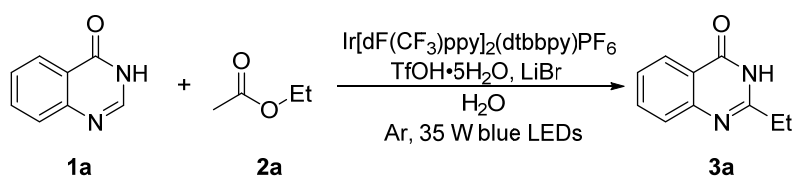
Entry	Acid	Yield (%) ^b
1	TfOH•5H ₂ O	81
2	-	N.D.
3	TsOH•5H ₂ O	Trace
4	AcOH•5H ₂ O	Trace
5	TFA•5H ₂ O	25
6	MAS•5H ₂ O	N.D.
7	TsOH	Trace

^a Reaction conditions: **1a** (0.2 mmol), **2a** (2.0 mL), Ir[dF(CF₃)ppy]₂(dtbbpy)PF₆ (1 mol %), LiBr (0.5 equiv), Acid (aq., 1.0 equiv), H₂O (3.0 equiv), 60 °C under Ar, 60 h. ^b Isolated yield.

Table S3. Screening of Additive^a

Entry	Additive	Yield (%) ^b
1	-	N.D.
2	LiBr (0.5 equiv)	81
3	NaBr (0.5 equiv)	63
4	NH ₄ Br (0.5 equiv)	71
5	LiCl (0.5 equiv)	N.D.
6	KI (0.5 equiv)	N.D.
7	ethyl 2-mercaptopropanoate (10 mol%)	N.D.
8	cyclohexanethiol (10 mol%)	N.D.
9	LiBr (0.5 equiv) + ethyl 2-mercaptopropanoate (10 mol%)	40

^a Reaction conditions: **1a** (0.2 mmol), **2a** (2.0 mL), Ir[dF(CF₃)ppy]₂(dtbbpy)PF₆ (1 mol %), TfOH•5H₂O (aq., 1.0 equiv), H₂O (3.0 equiv), 60 °C under Ar, 60 h. ^b Isolated yield.

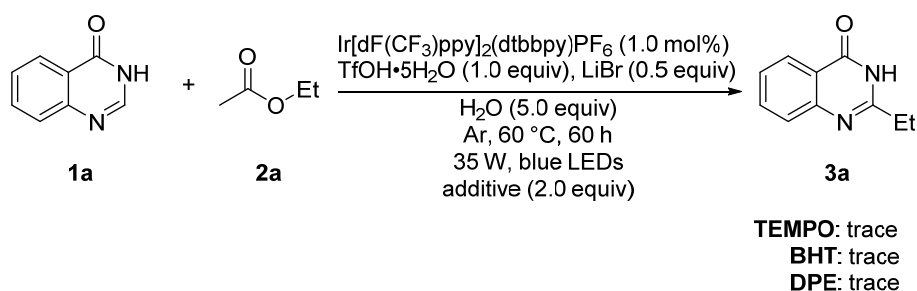
Table S4. Screening of H₂O^a

Entry	H ₂ O	Yield (%) ^b
1	-	Trace
2	3.0 equiv	81
3	5.0 equiv	85
4	10.0 equiv	83
5 ^c	5.0 equiv	trace

^a Reaction conditions: **1a** (0.2 mmol), **2a** (2.0 mL), Ir[dF(CF₃)ppy]₂(dtbbpy)PF₆ (1 mol %), LiBr (0.5 equiv), TfOH·5H₂O (aq., 1.0 equiv), H₂O, 60 °C under Ar, 60 h. ^b Isolated yield. ^c **2a** (20 equiv) combined with DMSO (2.0 mL) was used as the solvent.

4. Mechanistic studies

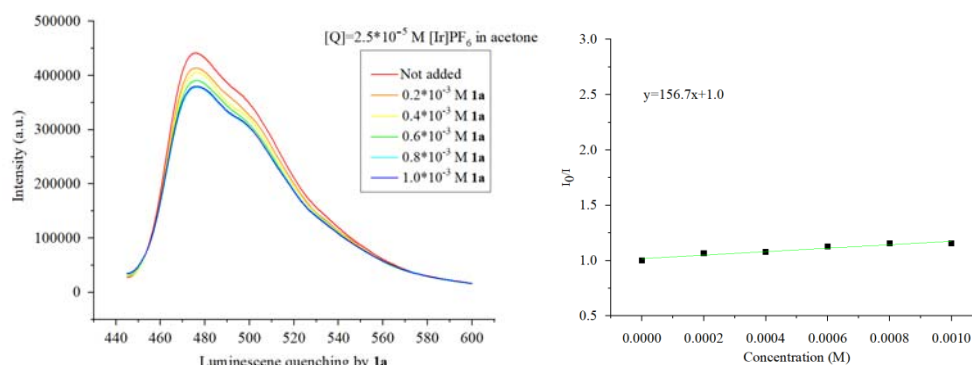
4.1 Radical trapping experiments



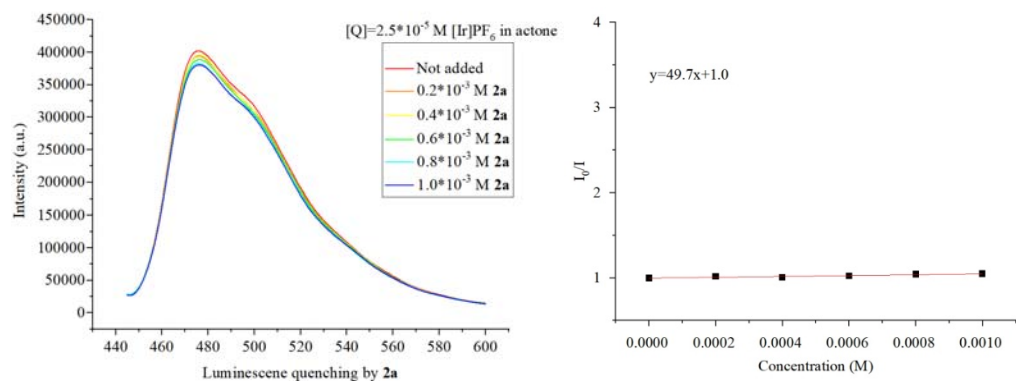
4.2 Stern–Volmer Quenching

Take 146.15 mg of 4-hydroxyquinazoline in a 10.0 mL volumetric flask and add methanol to the mark, the concentration is set to 0.1 mol/L. Take 97.6 μL of EtOAc in a 10.0 mL volumetric flask and add acetonitrile to the mark, the concentration is set to 0.1 M. Take 86.85 mg of LiBr in a 10.0 mL volumetric flask and add acetone to the mark, the concentration is set to 0.05 M. Photocatalyst (2.8 mg) was dissolved in acetone (10.0 mL) to set the concentration to be 0.1 mM. The resulting 0.1 mM solution (50.0 μL) was added to cuvette, this solution was then diluted to a volume of 2.0 mL by adding further acetone to prepare 2.5 μM solution. The resulting mixture was sparged with nitrogen for 3 minutes and then irradiated at 380 nm. Fluorescence emission spectra were recorded (3 trials per sample). Into this solution, 10.0 μL of a 4-Hydroxyquinazoline methanol solution was successively added and uniformly stirred, the resulting mixture was bubbled with nitrogen for 3 minutes and irradiated at 380 nm. Fluorescence emission spectra of 0 μL , 10.0 μL , 20.0 μL , 30.0 μL , 40.0 μL , 50.0 μL fluorescence intensity. Follow this method and make changes to the amount, relationship is obtained in turn.

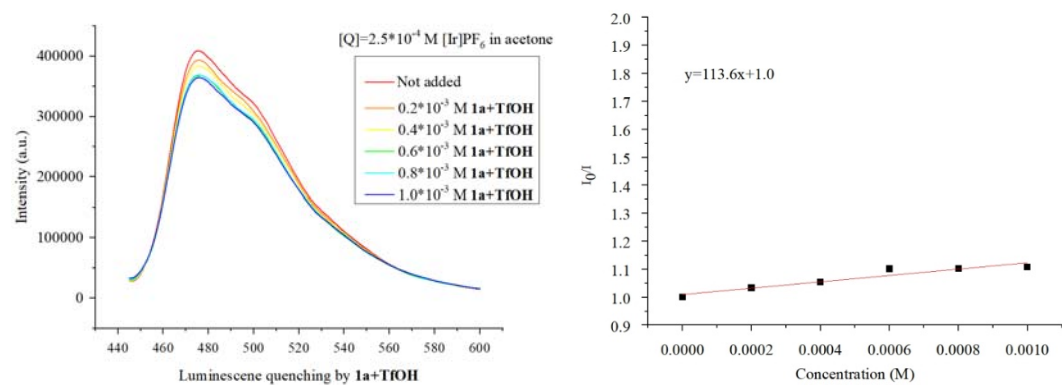
4.2.1 Quenching of photocatalyst by 4-hydroxyquinazoline



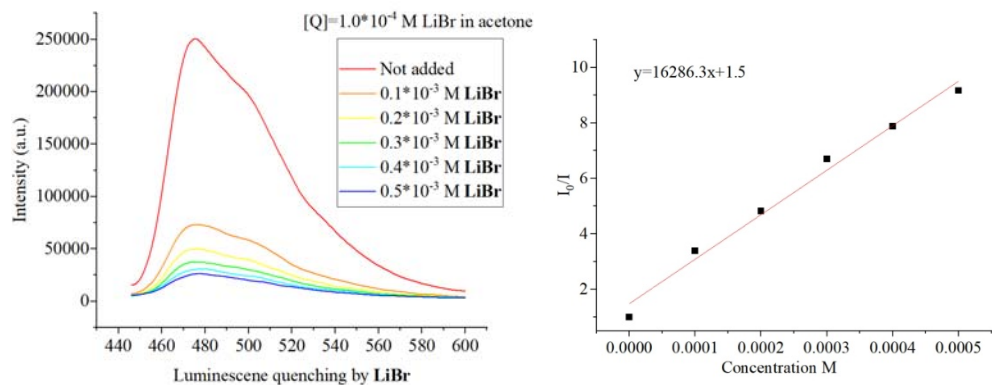
4.2.2 Quenching of photocatalyst by EtOAc



4.2.3 Quenching of photocatalyst by 4-hydroxyquinazoline (1a) + TfOH

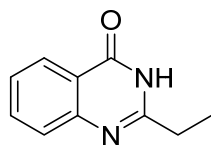


4.2.4 Quenching of photocatalyst by LiBr



5. Characterization data of products

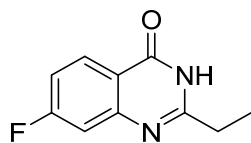
2-ethylquinazolin-4(3H)-one (3a)¹



The reaction was conducted with quinazolin-4(3H)-one (29.2 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 2/1) to yield **3a** (29.5 mg, 85%) as a white solid. mp: 214-216 °C.

¹H NMR (400 MHz, Chloroform-*d*) δ 12.13 (brs, 1H), 8.29 (d, J = 9.5 Hz, 1H), 7.79 – 7.74 (m, 1H), 7.71 (d, J = 8.0 Hz, 1H), 7.48 – 7.45 (m, J = 7.5 Hz, 1H), 2.85 (q, J = 7.6 Hz, 2H), 1.46 (t, J = 7.6 Hz, 3H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 164.5, 157.7, 149.5, 134.8, 127.2, 126.3, 126.2, 120.5, 29.1, 11.5.

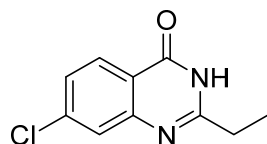
2-ethyl-7-fluoroquinazolin-4(3H)-one (3b)¹



The reaction was conducted with 2-ethylquinazolin-4(3H)-one (32.8 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 5/1) to yield **3b** (19.4 mg, 51%) as a white solid. mp: 225-227 °C.

¹H NMR (400 MHz, Chloroform-*d*) δ 11.87 (brs, 1H), 8.29 (dd, J = 8.8, 6.1 Hz, 1H), 7.36 (dd, J = 9.8, 2.4 Hz, 1H), 7.19 (m, 1H), 2.83 (q, J = 7.6 Hz, 2H), 1.44 (t, J = 7.6 Hz, 3H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 166.8 (d, J = 253.2 Hz), 163.1, 158.8, 129.0 (d, J = 10.8 Hz), 117.2, 115.3 (d, J = 23.3 Hz), 112.6 (d, J = 21.8 Hz), 29.1, 11.3. ¹⁹F NMR (376 MHz, Chloroform-*d*) δ -102.4.

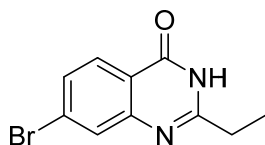
7-chloro-2-ethylquinazolin-4(3H)-one (3c)¹



The reaction was conducted with 7-chloroquinazolin-4(3H)-one (36.1 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 5/1) to yield **3c** (30.1 mg, 72%) as a white solid. mp: 239-241 °C.

^1H NMR (400 MHz, Chloroform-*d*) δ 12.28 (brs, 1H), 8.19 (d, $J = 8.5$ Hz, 1H), 7.69 (d, $J = 2.0$ Hz, 1H), 7.40 (dd, $J = 8.5, 2.0$ Hz, 1H), 2.83 (q, $J = 7.6$ Hz, 2H), 1.44 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 164.0, 159.1, 150.5, 141.0, 127.6, 127.0, 126.9, 118.9, 29.1, 11.4.

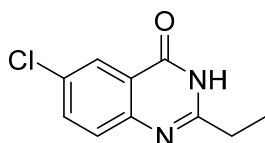
7-bromo-2-ethylquinazolin-4(3H)-one (3d)¹



The reaction was conducted with 7-bromoquinazolin-4(3H)-one (45.0 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 5/1) to yield **3d** (32.9 mg, 65%) as a white solid. mp: 236-238 °C.

^1H NMR (400 MHz, Chloroform-*d*) δ 11.89 (brs, 1H), 8.12 (d, $J = 8.5$ Hz, 1H), 7.90 (d, $J = 1.9$ Hz, 1H), 7.56 (dd, $J = 8.5, 1.9$ Hz, 1H), 2.83 (q, $J = 7.6$ Hz, 2H), 1.43 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 163.9, 158.9, 150.4, 130.1, 129.8, 129.6, 127.7, 119.3, 29.1, 11.3.

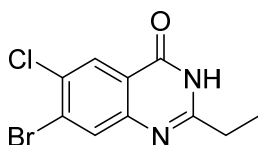
6-chloro-2-ethylquinazolin-4(3H)-one (3e)¹



The reaction was conducted with 7-bromoquinazolin-4(3H)-one (36.1mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 5/1) to yield **3e** (28.0 mg, 67%) as a white solid. mp: 229-231 °C.

^1H NMR (400 MHz, DMSO-*d*₆) δ 12.35 (brs, 1H), 7.97 (s, 1H), 7.76 (d, $J = 8.7$ Hz, 1H), 7.59 (d, $J = 8.7$ Hz, 1H), 2.59 (q, $J = 7.5$ Hz, 2H), 1.21 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (100 MHz, DMSO-*d*₆) δ 161.3, 159.4, 148.1, 134.8, 130.6, 129.5, 125.1, 122.5, 28.3, 11.6.

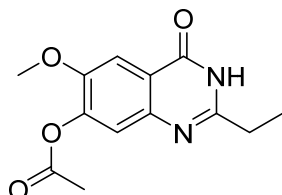
7-bromo-6-chloro-2-ethylquinazolin-4(3H)-one (3f)



The reaction was conducted with 7-bromo-6-chloroquinazolin-4(3H)-one (51.9 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 10/1) to yield **3f** (44.9 mg, 78%) as a white solid. mp: 256-258 °C.

^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 12.40 (brs, 1H), 8.02 (s, 1H), 7.87 (s, 1H), 2.58 (q, $J = 7.5$ Hz, 2H), 1.20 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (100 MHz, $\text{DMSO-}d_6$) δ 160.8, 160.7, 148.7, 132.2, 130.7, 128.3, 126.9, 121.7, 28.3, 11.5. HRMS (ESI) m/z calcd for $\text{C}_{10}\text{H}_8\text{BrClN}_2\text{NaO}^+$ ($\text{M}+\text{Na}$) $^+$ 308.9401, found 308.9400.

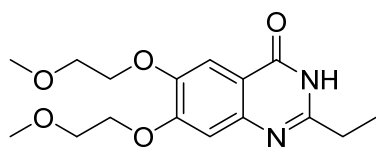
2-ethyl-6-methoxy-4-oxo-3,4-dihydroquinazolin-7-yl acetate (3g)



The reaction was conducted with 6-methoxy-4-oxo-3,4-dihydroquinazolin-7-yl acetate (46.8 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 10/1) to yield **3g** (36.7 mg, 78%) as a white solid. mp: 221-223 $^{\circ}\text{C}$.

^1H NMR (400 MHz, Chloroform- d) δ 12.34 (brs, 1H), 7.90 (s, 1H), 7.17 (s, 1H), 3.94 (s, 3H), 2.80 (q, $J = 7.6$ Hz, 2H), 2.34 (s, 3H), 1.42 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform- d) δ 168.9, 163.5, 158.5, 157.0, 149.7, 139.2, 119.5, 113.6, 108.7, 56.3, 29.1, 20.6, 11.6. HRMS (ESI) m/z calcd for $\text{C}_{13}\text{H}_{14}\text{N}_2\text{NaO}_4^+$ ($\text{M}+\text{Na}$) $^+$ 285.0846, found 285.0844.

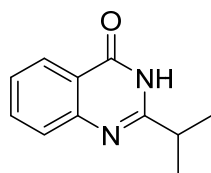
2-ethyl-6,7-bis(2-methoxyethoxy) quinazolin-4(3H)-one (3h)



The reaction was conducted with 6,7-bis(2-methoxyethoxy) quinazolin-4(3H)-one (58.9 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 5/1) to yield **3h** (22.6 mg, 78%) as a white solid. mp: 193-195 $^{\circ}\text{C}$.

^1H NMR (400 MHz, Chloroform- d) δ 11.66 (brs, 1H), 7.58 (s, 1H), 7.09 (s, 1H), 4.26 (q, $J = 5.3$ Hz, 4H), 3.86 – 3.84 (m, 4H), 3.48 (s, 3H), 3.47 (s, 3H), 2.80 (q, $J = 7.6$ Hz, 2H), 1.42 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform- d) δ 163.4, 156.4, 155.0, 148.2, 145.9, 113.7, 108.7, 106.9, 70.7, 70.5, 68.6, 68.4, 59.4, 59.4, 29.0, 11.7. HRMS (ESI) m/z calcd for $\text{C}_{16}\text{H}_{22}\text{N}_2\text{NaO}_5^+$ ($\text{M}+\text{Na}$) $^+$ 345.1421, found 345.1419.

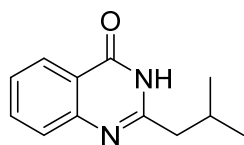
2-isopropylquinazolin-4(3H)-one (**3i**)²



The reaction was conducted with quinazolin-4(3H)-one (29.2 mg, 0.2 mmol) and isopropyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 5/1) to yield **3i** (7.0 mg, 18%) as a white solid.

¹H NMR (400 MHz, Chloroform-*d*) δ 11.90 (brs, 1H), 8.30 (d, *J* = 7.9 Hz, 1H), 7.78 – 7.71 (m, 2H), 7.48 – 7.44 (m, 1H), 3.10 – 3.04 (m, 1H), δ 1.46 (d, *J* = 7.0 Hz, 6H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 164.4, 161.0, 149.5, 134.7, 127.4, 126.3, 126.2, 120.7, 35.0, 20.4.

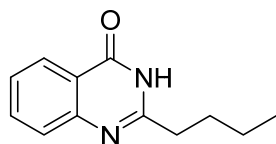
2-isobutylquinazolin-4(3H)-one (**3j**)²



The reaction was conducted with quinazolin-4(3H)-one (29.2 mg, 0.2 mmol) and isobutyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 5/1) to yield **3j** (10.1 mg, 25%) as a white solid.

¹H NMR (400 MHz, Chloroform-*d*) δ 12.41 (brs, 1H), 8.29 (d, *J* = 7.9 Hz, 1H), 7.78 – 7.70 (m, 2H), 7.48 – 7.44 (m, 1H), 2.69 (d, *J* = 7.4 Hz, 2H), 2.40 – 2.30 (m, 1H), 1.06 (d, *J* = 6.7 Hz, 6H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 164.6, 156.5, 149.5, 134.8, 127.3, 126.3, 126.2, 120.4, 44.7, 28.0, 22.4.

2-butylquinazolin-4(3H)-one (**3k**)¹

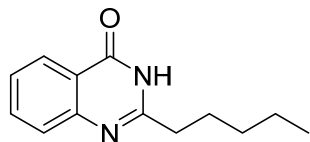


The reaction was conducted with quinazolin-4(3H)-one (29.2 mg, 0.2 mmol) and n-butyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 2/1) to yield **3k** (22.3 mg, 55%) as a white solid. mp: 135-137 °C.

¹H NMR (400 MHz, Chloroform-*d*) δ 12.15 (brs, 1H), 8.28 (d, *J* = 8.4 Hz, 1H), 7.79 – 7.77 (m, 1H), 7.71 (d, *J* = 8.1 Hz, 1H), 7.49 – 7.45 (m, 1H), 2.83 – 2.79 (m, 2H), 1.92-1.84 (m, 2H), 1.55 –

1.45 (m, 2H), 0.99 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 164.60, 157.2, 149.5, 134.8, 127.2, 126.3, 126.2, 120.5, 35.7, 29.7, 22.4, 13.8.

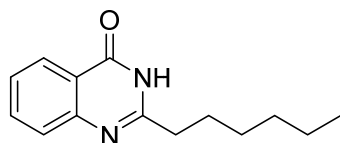
2-pentylquinazolin-4(3*H*)-one (**3l**)¹



The reaction was conducted with quinazolin-4(3*H*)-one (29.2 mg, 0.2 mmol) and n-pentyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 2/1) to yield **3l** (21.6 mg, 50%) as a white solid. mp: 156-158 °C.

^1H NMR (400 MHz, Chloroform-*d*) δ 12.29 (brs, 1H), 8.28 (d, $J = 7.9$ Hz, 1H), 7.78 – 7.69 (m, 2H), 7.48 – 7.44 (m, 1H), 2.82 – 2.79 (m, 2H), 1.94 – 1.86 (m, 2H), 1.49 – 1.37 (m, 4H), 0.92 (t, $J = 7.0$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 164.6, 157.2, 149.5, 134.8, 127.2, 126.3, 126.2, 120.5, 35.9, 31.4, 27.3, 22.4, 14.0.

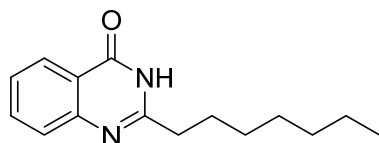
2-hexylquinazolin-4(3*H*)-one (**3m**)³



The reaction was conducted with quinazolin-4(3*H*)-one (29.2 mg, 0.2 mmol) and n-hexyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 2/1) to yield **3m** (23.1 mg, 50%) as a white solid. mp: 181-183 °C.

^1H NMR (400 MHz, Chloroform-*d*) δ 12.15 (brs, 1H), 8.29 (d, $J = 8.0$ Hz, 1H), 7.80 – 7.76 (m, 1H), 7.72 (d, $J = 7.7$ Hz, 1H), 7.49 – 7.45 (m, 1H), 2.83 – 2.79 (m, 2H), 1.93 – 1.85 (m, 2H), 1.51-1.43 (m, 2H), 1.39 – 1.32 (m, 4H), 0.89 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 164.4, 157.2, 149.4, 134.8, 127.2, 126.3, 126.2, 120.5, 36.0, 31.5, 28.9, 27.6, 22.5, 14.1.

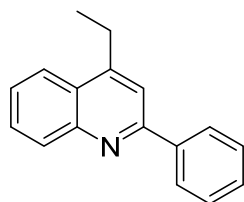
2-heptylquinazolin-4(3*H*)-one (**3n**)⁵



The reaction was conducted with quinazolin-4(3*H*)-one (29.2 mg, 0.2 mmol) and n-heptyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 2/1) to yield **3n** (14.6 mg, 50%) as a white solid. mp: 185-187 °C.

¹H NMR (400 MHz, Chloroform-*d*) δ 11.85 (brs, 1H), 8.29 (d, *J* = 8.0 Hz, 1H), 7.79 – 7.75 (m, 1H), 7.71 (d, *J* = 8.1 Hz, 1H), 7.49 – 7.45 (m, 1H), 2.82 – 2.78 (m, 2H), 1.92 – 1.95 (m, 2H), 1.50 – 1.46 (m, 2H), 1.44 – 1.34 (m, 2H), 1.30 – 1.25 (m, 4H), 0.86 (t, *J* = 6.7 Hz, 3H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 164.3, 157.1, 149.4, 134.8, 126.3, 126.2, 120.5, 36.0, 31.8, 29.3, 29.2, 29.1, 27.6, 22.6, 14.1.

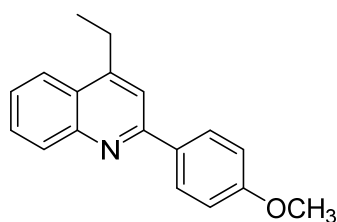
4-ethyl-2-phenylquinoline (**5a**)⁵



The reaction was conducted with 2-phenylquinoline (41.1 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether) to yield **5a** (23.3 mg, 50%) as a white solid. mp: 82-84 °C.

¹H NMR (400 MHz, Chloroform-*d*) δ 8.21 (d, *J* = 8.4 Hz, 1H), 8.16 (d, *J* = 7.0 Hz, 2H), 8.05 (d, *J* = 8.4 Hz, 1H), 7.73 (d, *J* = 7.0 Hz, 2H), 7.57 – 7.52 (m, 3H), 7.48 – 7.45 (m, 1H), 3.18 (q, *J* = 7.6 Hz, 2H), 1.46 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 157.3, 150.6, 148.3, 139.9, 130.4, 129.3, 129.2, 128.8, 127.6, 126.4, 126.1, 123.3, 117.9, 25.4, 14.3.

4-ethyl-2-(4-methoxyphenyl) quinoline (**5b**)

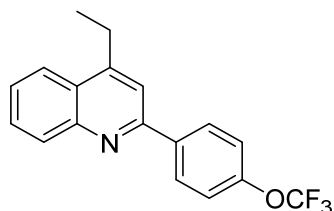


The reaction was conducted with 2-(4-methoxyphenyl) quinoline (47.1 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 100/1) to yield **5b** (29.5 mg, 56%) as a colorless liquid.

¹H NMR (400 MHz, Chloroform-*d*) δ 8.18 – 8.14 (m, 2H), 8.12 (s, 1H), 8.02 (d, *J* = 8.3 Hz, 1H), 7.71 – 7.67 (m, 2H), 7.53 – 7.49 (m, 1H), 7.05 (d, *J* = 8.7 Hz, 2H), 3.89 (s, 3H), 3.16 (q, *J* = 7.5

Hz, 2H), 1.45 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform- d) δ 160.7, 156.8, 150.4, 148.3, 132.4, 130.2, 129.2, 128.9, 126.2, 125.7, 123.3, 117.4, 114.2, 55.4, 25.5, 14.3. HRMS (ESI) m/z calcd for $\text{C}_{18}\text{H}_{17}\text{KNO}^+$ ($\text{M}+\text{K}$) $^+$ 302.0942, found 302.0945.

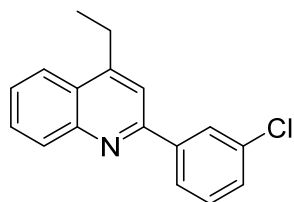
4-ethyl-2-(4-(trifluoromethoxy) phenyl) quinoline (5c)



The reaction was conducted with 2-(4-(trifluoromethyl) phenyl) quinoline (57.9 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 100/1) to yield **5e** (50.7 mg, 80%) as a white solid. mp: 80-82 °C.

^1H NMR (400 MHz, Chloroform- d) δ 8.21 – 8.17 (m, 3H), 8.05 (d, $J = 7.8$ Hz, 1H), 7.75 – 7.72 (m, 1H), 7.69 (s, 1H), 7.58 – 7.54 (m, 1H), 7.37 (d, $J = 7.8$ Hz, 2H), 3.18 (q, $J = 6.8$ Hz, 2H), 1.45 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform- d) δ 155.8, 150.9, 150.1, 148.3, 138.6, 130.4, 129.5, 129.1, 126.5, 126.3, 123.3, 121.1, 120.5 (q, $J = 255.7$ Hz), 117.5, 25.4, 14.2. ^{19}F NMR (376 MHz, Chloroform- d) δ -57.7. HRMS (ESI) m/z calcd for $\text{C}_{18}\text{H}_{15}\text{F}_3\text{NO}^+$ ($\text{M}+\text{H}$) $^+$ 318.1100, found 318.1107.

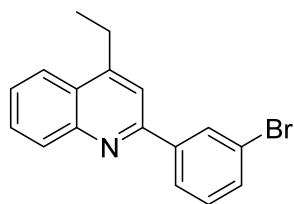
2-(4-chlorophenyl)-4-ethylquinoline (5d)



The reaction was conducted with 2-(4-chlorophenyl) quinoline (47.9 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 100/1) to yield **5c** (28.4 mg, 56%) as a colorless liquid.

^1H NMR (400 MHz, Chloroform- d) δ 8.19 (d, $J = 7.8$ Hz, 2H), 8.07 – 8.01 (m, 2H), 7.76 – 7.70 (m, 1H), 7.69 (s, 1H), 7.58 – 7.54 (m, 1H), 7.44 (d, $J = 7.2$ Hz, 2H), 3.17 (q, $J = 7.6$ Hz, 2H), 1.45 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform- d) δ 155.7, 150.9, 148.3, 141.7, 134.9, 130.5, 130.0, 129.5, 129.2, 127.7, 126.6, 126.4, 125.7, 123.3, 117.5, 25.5, 14.3. HRMS (ESI) m/z calcd for $\text{C}_{17}\text{H}_{14}\text{ClNNa}^+$ ($\text{M}+\text{Na}$) $^+$ 290.0707, found 290.0712.

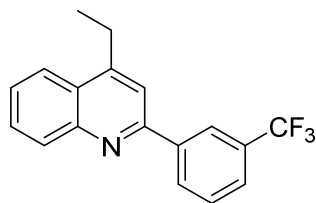
2-(3-bromophenyl)-4-ethylquinoline (5e)



The reaction was conducted with 2-(3-bromophenyl) quinoline (56.8 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 100/1) to yield **5f** (40.0 mg, 80%) as a colorless liquid.

^1H NMR (400 MHz, Chloroform-*d*) δ 8.34 (s, 1H), 8.18 (d, $J = 8.4$ Hz, 1H), 8.11 – 8.01 (m, 2H), 7.77 – 7.68 (m, 1H), 7.68 (s, 1H), 7.61 – 7.52 (m, 2H), 7.39 (m, 1H), 3.17 (q, $J = 7.6$ Hz, 2H), 1.45 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.5, 151.0, 148.2, 141.9, 132.1, 130.6, 130.5, 130.3, 129.5, 126.6, 126.4, 126.1, 123.3, 123.1, 117.5, 25.5, 14.3. HRMS (ESI) m/z calcd for $\text{C}_{17}\text{H}_{15}\text{NBr}^+$ ($\text{M}+\text{H}$) $^+$ 312.0382, found 312.0383.

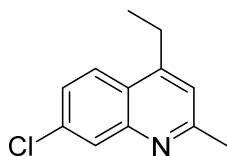
4-ethyl-2-(4-(trifluoromethyl) phenyl) quinoline (5f)



The reaction was conducted with 2-(4-(trifluoromethyl) phenyl) quinoline (54.7 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 100/1) to yield **5d** (42.8 mg, 71%) as a colorless liquid.

^1H NMR (400 MHz, Chloroform-*d*) δ 8.46 (s, 1H), 8.35 (d, $J = 7.8$ Hz, 1H), 8.21 (d, $J = 8.4$ Hz, 1H), 8.06 (d, $J = 8.4$ Hz, 1H), 7.77 – 7.70 (m, 3H), 7.64 (m, 1H), 7.60 – 7.55 (m, 1H), 3.19 (q, $J = 7.6$ Hz, 2H), 1.46 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.5, 151.1, 148.3, 140.7, 131.2 (q, $J = 32.3$ Hz), 130.8, 130.5, 129.4 (q, $J = 26.7$ Hz), 126.6 (q, $J = 10.1$ Hz), 125.8 (d, $J = 3.9$ Hz), 124.4 (q, $J = 3.8$ Hz), 124.3 (q, $J = 270.6$ Hz), 123.3, 117.4, 25.5, 14.3. ^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.4. HRMS (ESI) m/z calcd for $\text{C}_{18}\text{H}_{14}\text{F}_3\text{NNa}^+$ ($\text{M}+\text{Na}$) $^+$ 324.0971, found 324.0973.

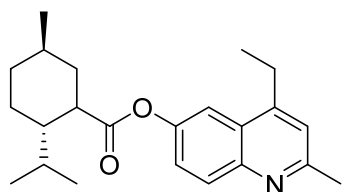
7-chloro-4-ethyl-2-methylquinoline (5g)



The reaction was conducted with 7-chloro-2-methylquinoline (35.5 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether) to yield **5g** (14.2 mg, 35%) as a colorless liquid.

^1H NMR (400 MHz, Chloroform-*d*) δ 8.02 (s, 1H), 7.91 (d, $J = 8.9$ Hz, 1H), 7.44 (dd, $J = 8.9, 2.2$ Hz, 1H), 7.13 (s, 1H), 3.04 (q, $J = 7.5$ Hz, 2H), 2.70 (s, 3H), 1.37 (t, $J = 7.5$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 160.1, 150.0, 148.4, 134.8, 128.2, 126.4, 124.6, 124.2, 120.9, 25.3, 25.0, 14.0. HRMS (ESI) m/z calcd for $\text{C}_{12}\text{H}_{13}\text{ClN}^+$ ($\text{M}+\text{H}$) $^+$ 206.0731, found 206.0730.

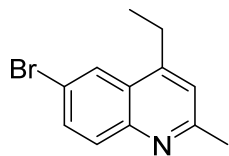
4-ethyl-2-methylquinolin-6-yl (2S,5R)-2-isopropyl-5-methylcyclohexane-1-carboxylate (**5h**)



The reaction was conducted with 2-methylquinolin-6-yl (2S,5R)-2-isopropyl-5-methylcyclohexane-1-carboxylate (65.1 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether) to yield **5h** (28.3 mg, 40%) as a colorless liquid.

^1H NMR (400 MHz, Chloroform-*d*) δ 8.76 (s, 1H), 8.25 (d, $J = 8.8$ Hz, 1H), 8.05 (d, $J = 8.8$ Hz, 1H), 7.20 (s, 1H), 5.03 – 4.97 (m, 1H), 3.20 – 3.09 (m, 2H), 2.74 (s, 3H), 2.19 – 2.15 (m, 1H), 2.04 – 1.96 (m, 1H), 1.78 – 1.74 (m, 2H), 1.64 – 1.59 (m, 2H), 1.41 (t, $J = 7.5$ Hz, 3H), 1.20-1.12 (m, 2H), 0.95 – 0.82 (m, 7H), 0.83 (d, $J = 6.9$ Hz, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 166.0, 161.2, 151.4, 149.8, 129.4, 128.7, 127.6, 126.5, 125.1, 121.3, 75.2, 47.3, 41.0, 34.3, 31.5, 26.7, 25.6, 24.9, 23.8, 22.1, 20.8, 16.7, 14.1. HRMS (ESI) m/z calcd for $\text{C}_{23}\text{H}_{31}\text{NNaO}_2^+$ ($\text{M}+\text{Na}$) $^+$ 376.2247, found 376.2247.

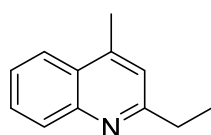
6-bromo-4-ethyl-2-methylquinoline (**5i**)



The reaction was conducted with 6-bromo-2-methylquinoline (41.4 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether) to yield **5i** (28.0 mg, 56%) as a white solid. mp: 85-87 °C.

¹H NMR (400 MHz, Chloroform-*d*) δ 8.11 (s, 1H), 7.88 (d, *J* = 8.9 Hz, 1H), 7.71 (d, *J* = 8.9 Hz, 1H), 7.15 (s, 1H), 3.01 (q, *J* = 7.6 Hz, 2H), 2.69 (s, 3H), 1.37 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 159.4, 149.0, 146.5, 132.4, 131.1, 127.1, 125.7, 121.4, 119.4, 25.4, 24.8, 13.9. HRMS (ESI) *m/z* calcd for C₁₂H₁₃BrN⁺ (M+H)⁺ 250.0226, found 250.0225.

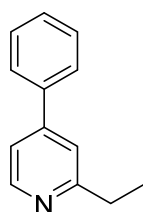
2-ethyl-4-methylquinoline (**5j**)⁷



The reaction was conducted with 4-methylquinoline (27 μL, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether) to yield **5j** (18.8 mg, 55%) as a colorless liquid.

¹H NMR (400 MHz, Chloroform-*d*) δ 8.04 (d, *J* = 8.3 Hz, 1H), 7.96 – 7.93 (m, 1H), 7.69 – 7.65 (m, 1H), 7.52 – 7.48 (m, 1H), 7.16 (s, 1H), 2.96 (q, *J* = 7.6 Hz, 2H), 2.68 (s, 3H), 1.38 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 163.7, 147.6, 144.4, 129.3, 129.1, 126.8, 125.4, 123.6, 121.6, 32.2, 18.7, 14.1.

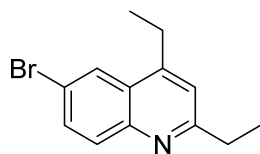
2-ethyl-4-phenylpyridine (**5k**)⁸



The reaction was conducted with 4-phenylpyridine (31.0 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 5/1) to yield **5k** (14.7 mg, 40%) as a colorless liquid.

¹H NMR (400 MHz, Chloroform-*d*) δ 8.57 (d, *J* = 5.2 Hz, 1H), 7.65 – 7.61 (m, 2H), 7.51 – 7.40 (m, 3H), 7.38 (s, 1H), 7.33 (d, *J* = 5.2 Hz, 1H), 2.89 (q, *J* = 7.6 Hz, 2H), 1.36 (t, *J* = 7.6 Hz, 3H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 164.0, 149.6, 148.9, 138.6, 129.0, 128.9, 127.1, 120.1, 119.1, 31.5, 14.0.

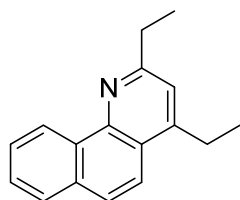
6-bromo-2-methylquinoline (5l)



The reaction was conducted with 6-bromoquinoline (41.6 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether) to yield **5l** (30.6 mg, 58%) as a white solid. mp: 83-85 °C.

¹H NMR (400 MHz, Chloroform-*d*) δ 8.10 (s, 1H), 7.90 (d, *J* = 8.9 Hz, 1H), 7.71 (d, *J* = 8.9 Hz, 1H), 7.16 (s, 1H), 3.01 (q, *J* = 7.5 Hz, 2H), 2.94 (q, *J* = 7.6 Hz, 2H), 1.39 – 1.35 (m, 6H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 164.3, 149.1, 146.5, 132.2, 131.3, 127.3, 125.7, 120.2, 119.4, 32.3, 25.0, 14.0, 13.9. HRMS (ESI) *m/z* calcd for C₁₃H₁₅BrN⁺ (M+H)⁺ 264.0382, found 264.0387.

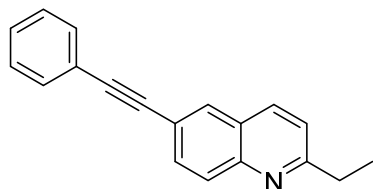
2,4-diethylbenzo[h]quinoline (5m)⁹



The reaction was conducted with benzo[h]quinoline (36.0 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether) to yield **5m** (40.0 mg, 86%) as a colorless liquid.

¹H NMR (400 MHz, Chloroform-*d*) δ 9.40 (d, *J* = 8.1 Hz, 1H), 7.88 – 7.84 (m, 2H), 7.75 – 7.61 (m, 3H), 7.23 (s, 1H), 3.12 – 3.01 (m, 4H), 1.47 (t, *J* = 7.6 Hz, 3H), 1.38 (t, *J* = 7.5 Hz, 3H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 162.2, 149.7, 145.8, 133.3, 132.0, 127.7, 127.5, 126.6, 126.2, 125.0, 123.1, 121.0, 120.2, 32.1, 25.5, 14.6, 13.8.

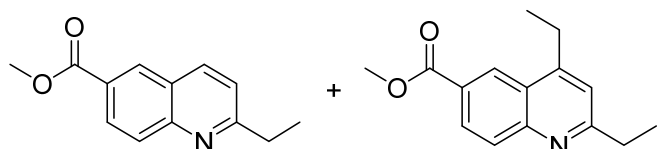
2-ethyl-6-(phenylethynyl) quinoline (5n)



The reaction was conducted with 6-(phenylethynyl) quinoline (45.9 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ ethyl acetate = 100/1) to yield **5n** (20.6 mg, 40%) as a white solid. mp: 82-84 °C.

^1H NMR (400 MHz, Chloroform-*d*) δ 8.05 – 7.98 (m, 3H), 7.79 (d, $J = 8.7$ Hz, 1H), 7.59 – 7.56 (m, 2H), 7.40 – 7.33 (m, 4H), 3.01 (q, $J = 7.6$ Hz, 2H), 1.40 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 164.8, 147.2, 136.1, 132.2, 131.7, 130.9, 128.9, 128.5, 128.4, 126.5, 123.1, 121.6, 120.6, 90.2, 89.2, 32.4, 14.0. HRMS (ESI) m/z calcd for $\text{C}_{19}\text{H}_{15}\text{NNa}^+$ ($\text{M}+\text{Na}$) $^+$ 280.1097, found 280.1099.

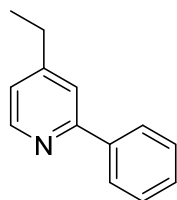
methyl 2-ethylquinoline-6-carboxylate (5o) + methyl 2,4-diethylquinoline-6-carboxylate (5p)



The reaction was conducted with methyl quinoline-6-carboxylate (37.4 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether) to yield **5p+5q** (28.2 mg, 58%) as a colorless liquid.

^1H NMR (400 MHz, Chloroform-*d*) δ 8.73 (d, $J = 1.9$ Hz, 0.37H), 8.52 (d, $J = 1.9$ Hz, 0.63H), 8.26 – 8.21 (m, 1.02H), 8.14 (d, $J = 8.5$ Hz, 0.65H), 8.05 (dd, $J = 8.8, 6.3$ Hz, 1.02H), 7.35 (d, $J = 8.5$ Hz, 0.65H), 3.97 (s, 0.37H), 3.97 (s, 1.11H), 3.96 (s, 2.16H). 3.15 – 2.95 (m, 2.74H), 1.39 (t, $J = 7.7$ Hz, 4.11H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 167.0, 166.8, 166.5, 166.3, 151.7, 149.8, 149.8, 130.7, 129.6, 129.0, 128.9, 128.6, 127.2, 126, 126.5, 125.8, 125.2, 52.4, 32.5, 32.4, 25.0, 14.2, 13.9, 13.8.

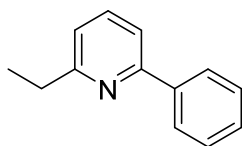
4-ethyl-2-phenylpyridine (5q)



The reaction was conducted with 2-phenylpyridine (31 μL , 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 5/1) to yield **5q** (17 mg, 47%) as a colorless liquid.

^1H NMR (400 MHz, Chloroform-*d*) δ 8.58 (d, $J = 5.0$ Hz, 1H), 7.98 (d, $J = 7.0$ Hz, 2H), 7.57 (s, 1H), 7.49 – 7.46 (m, 2H), 7.43 – 7.39 (m, 1H), 7.09 (d, $J = 5.0$ Hz, 1H), 2.72 (q, $J = 7.6$ Hz, 2H), 1.30 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 157.4, 153.9, 149.5, 139.5, 128.9, 128.7, 127.0, 121.9, 120.4, 28.6, 14.5. HRMS (ESI) m/z calcd for $\text{C}_{13}\text{H}_{13}\text{NNa}^+$ ($\text{M}+\text{Na}$) $^+$ 206.0940, found 206.0940.

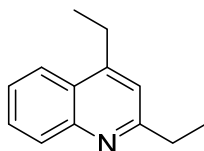
2-ethyl-6-phenylpyridine (**5r**)⁴



The reaction was conducted with 2-phenylpyridine (31 μ L, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether/ethyl acetate = 5/1) to yield **5r** (6.1 mg, 21%) as a colorless liquid.

¹H NMR (400 MHz, Chloroform-*d*) δ 8.00 (d, J = 7.0 Hz, 2H), 7.68 – 7.65 (m, 1H), 7.53 (d, J = 7.7 Hz, 1H), 7.48 – 7.45 (m, 2H), 7.42 – 7.40 (m, 1H), 7.11 (d, J = 7.5 Hz, 1H), 2.91 (q, J = 7.6 Hz, 2H), 1.37 (t, J = 7.6 Hz, 3H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 163.4, 156.8, 139.8, 137.0, 128.7, 128.7, 127.0, 120.4, 117.8, 31.5, 13.9.

2,4-diethylquinoline (**5s**)



The reaction was conducted with 4-chloroquinoline (32.7 mg, 0.2 mmol) and ethyl acetate (2.0 mL). Purification by thin layer chromatography was performed (petroleum ether) to yield **5s** (24.8 mg, 67%) as a colorless liquid.

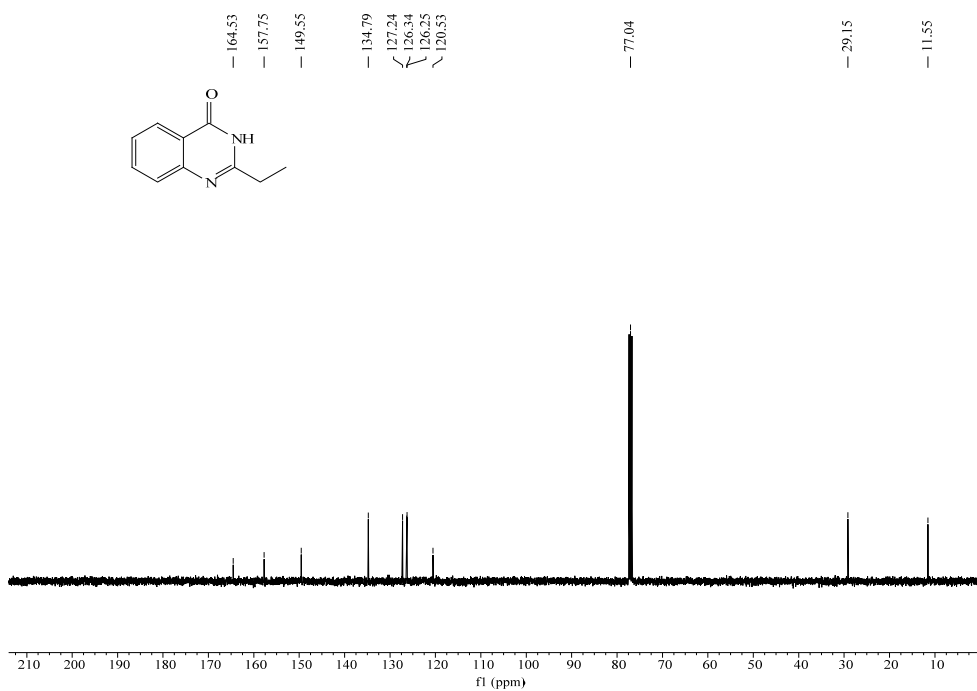
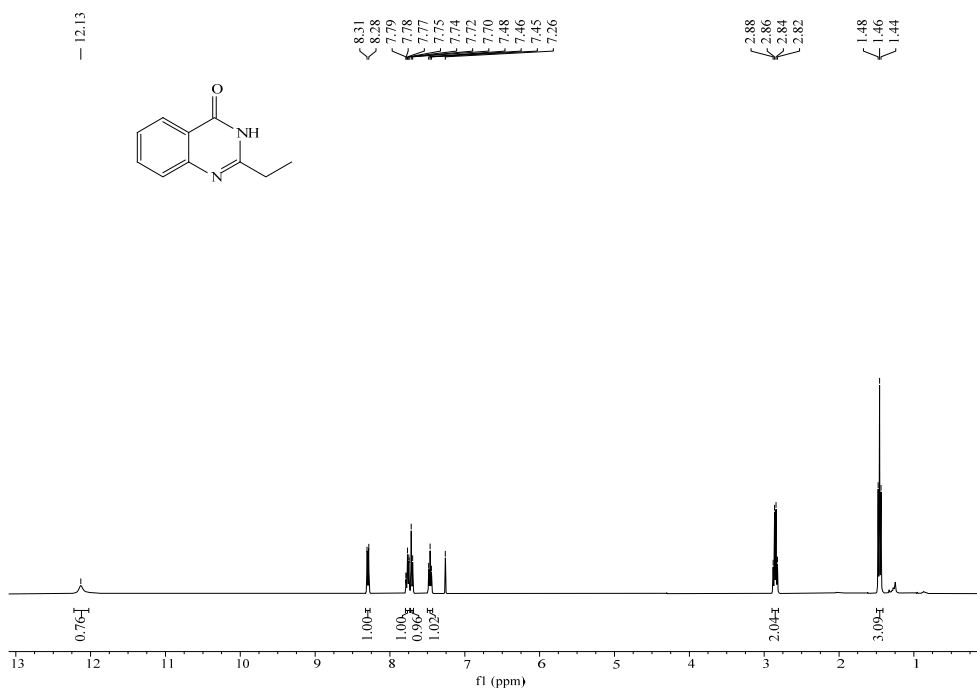
¹H NMR (400 MHz, Chloroform-*d*) δ 8.05 (d, J = 8.4 Hz, 1H), 7.98 (d, J = 8.4 Hz, 1H), 7.68 – 7.62 (m, 1H), 7.51 – 7.46 (m, 1H), 7.16 (s, 1H), 3.08 (q, J = 7.5 Hz, 2H), 2.97 (q, J = 7.6 Hz, 2H), 1.41 – 1.36 (m, 6H); ¹³C NMR (100 MHz, Chloroform-*d*) δ 163.9, 150.1, 147.8, 129.4, 128.9, 126.0, 125.4, 123.2, 119.5, 32.4, 25.1, 14.2, 14.1. HRMS (ESI) m/z calcd for C₁₃H₁₆N⁺ (M+H)⁺ 186.1277, found 186.1281.

6. References

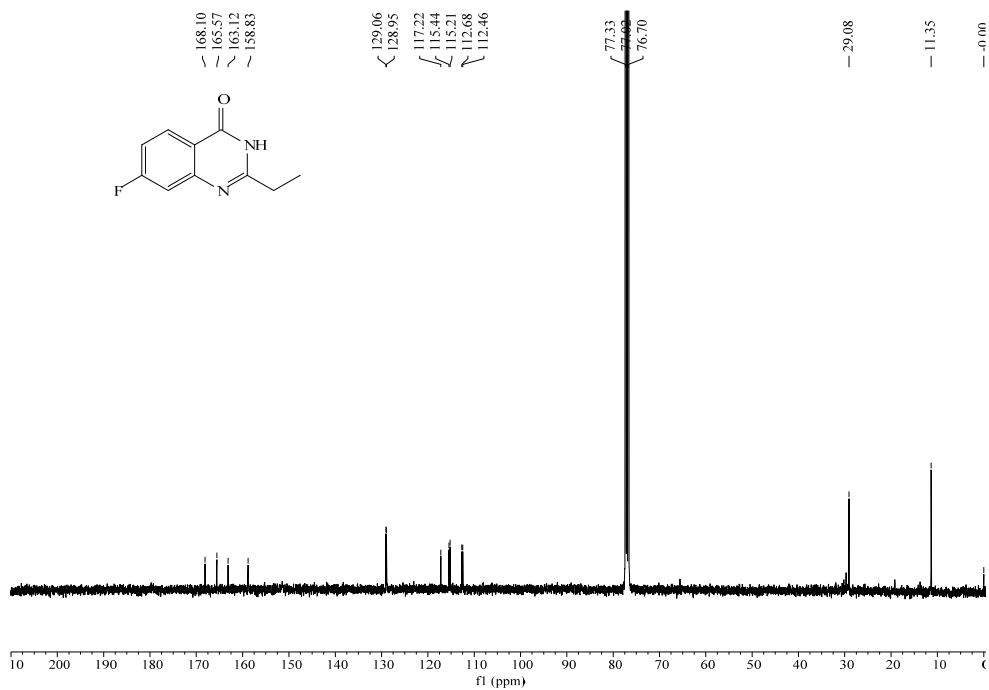
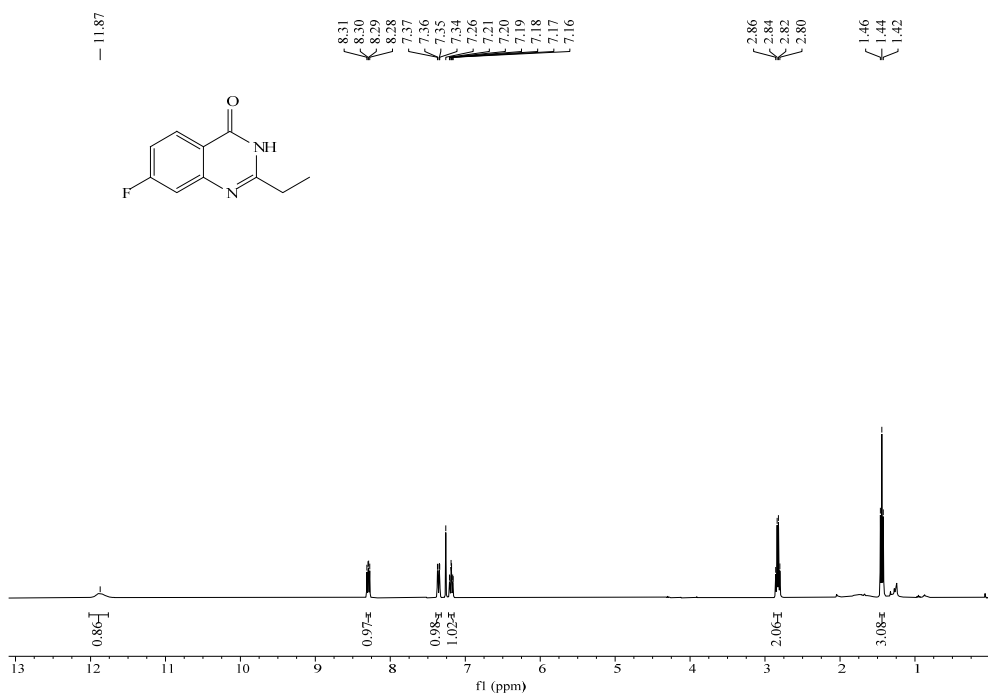
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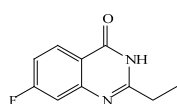
7. Copies of ^1H and ^{13}C NMR spectra of all products

^1H and ^{13}C NMR spectra of 3a

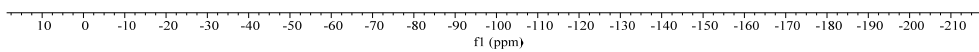


^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of 3b

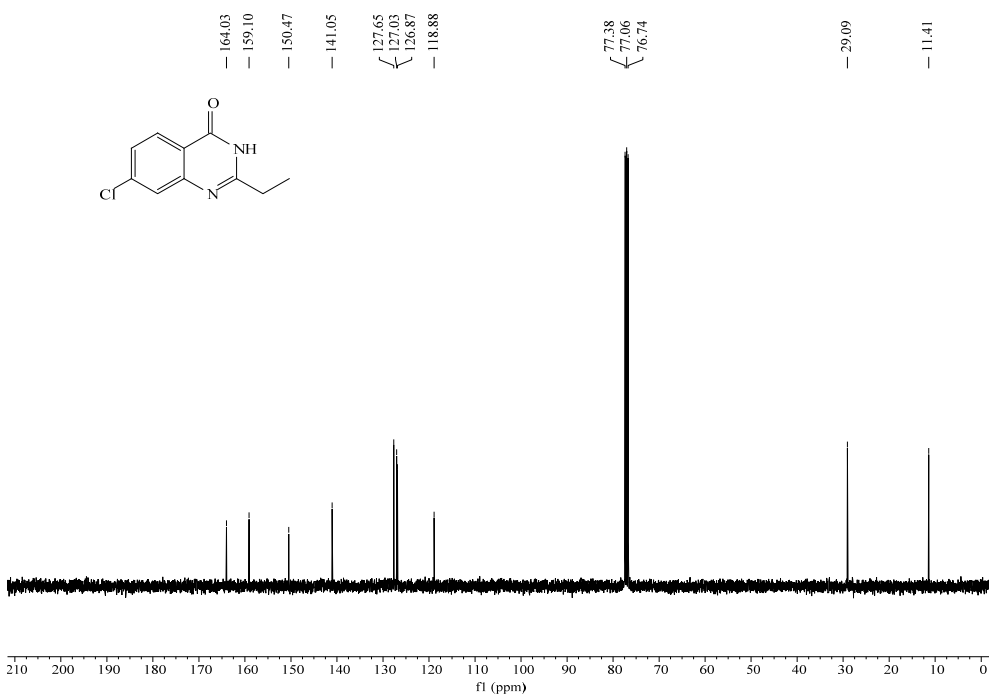
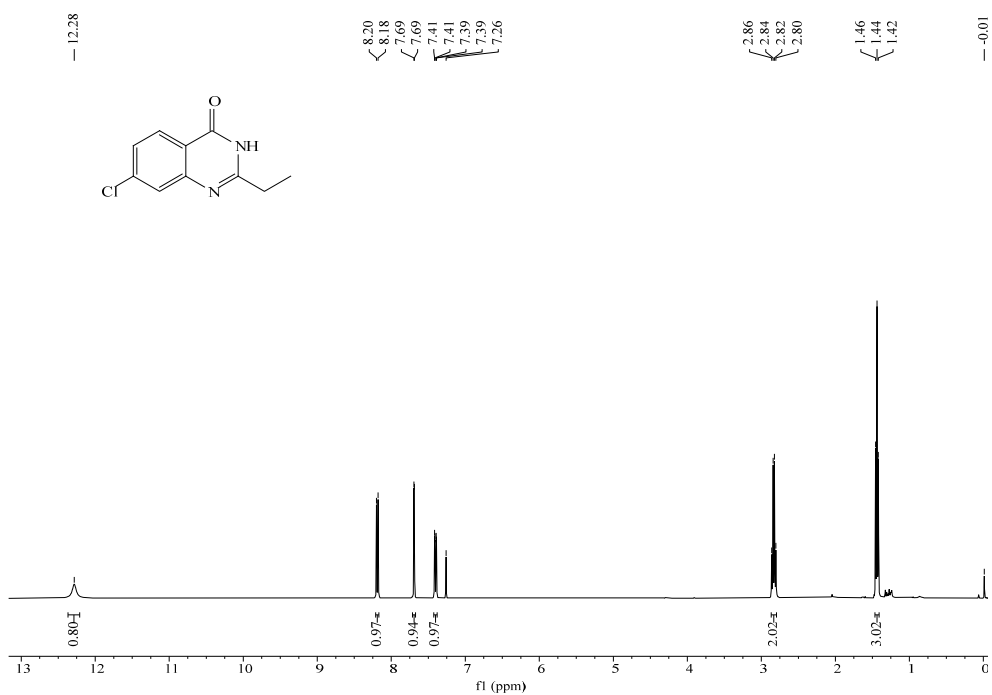




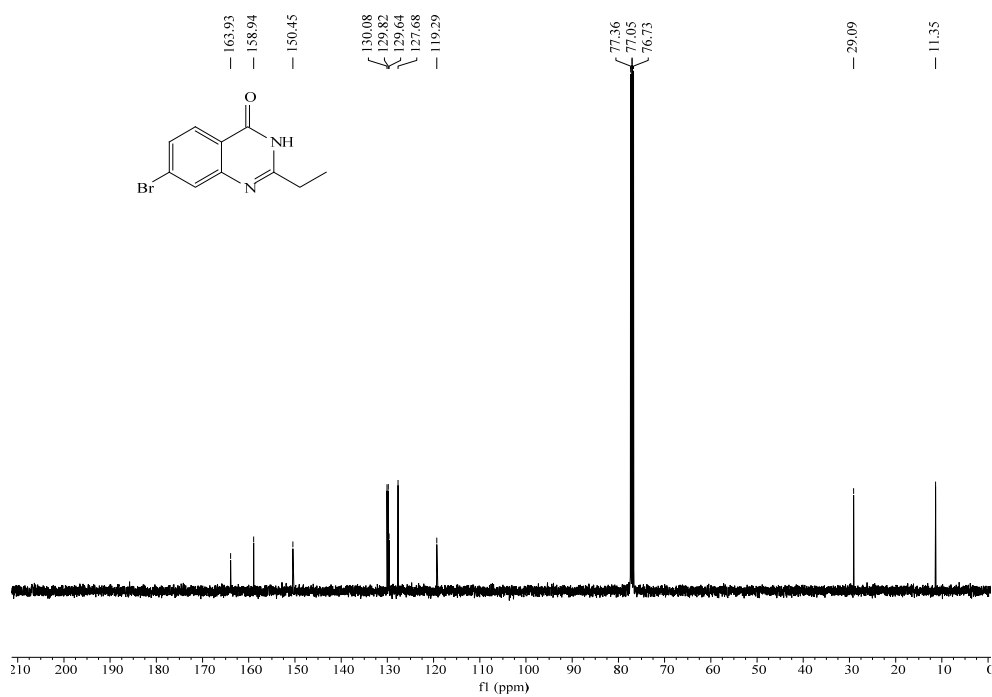
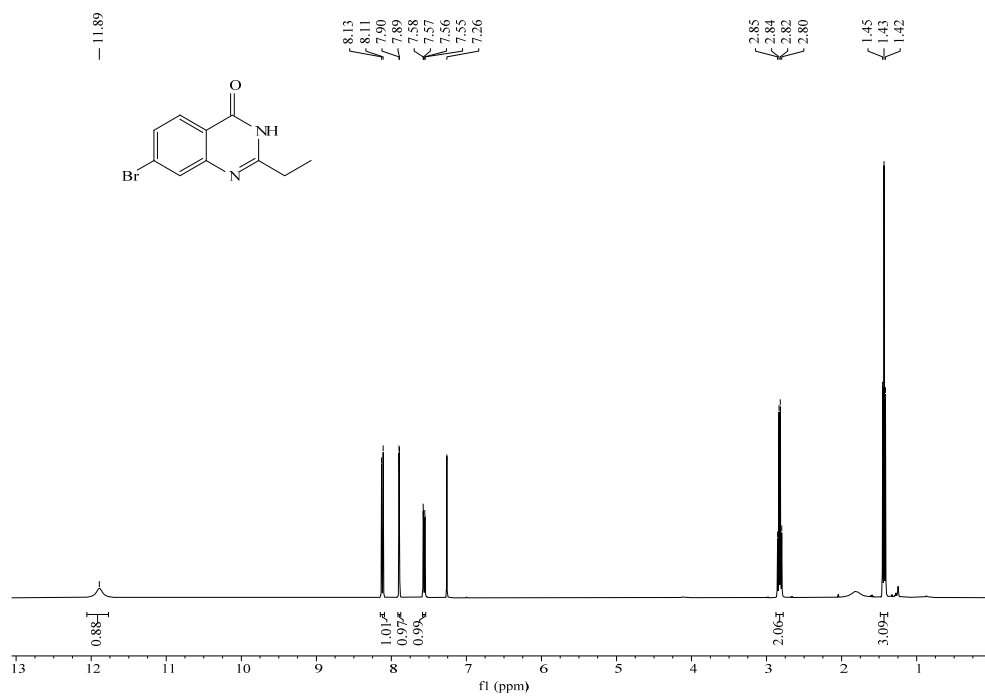
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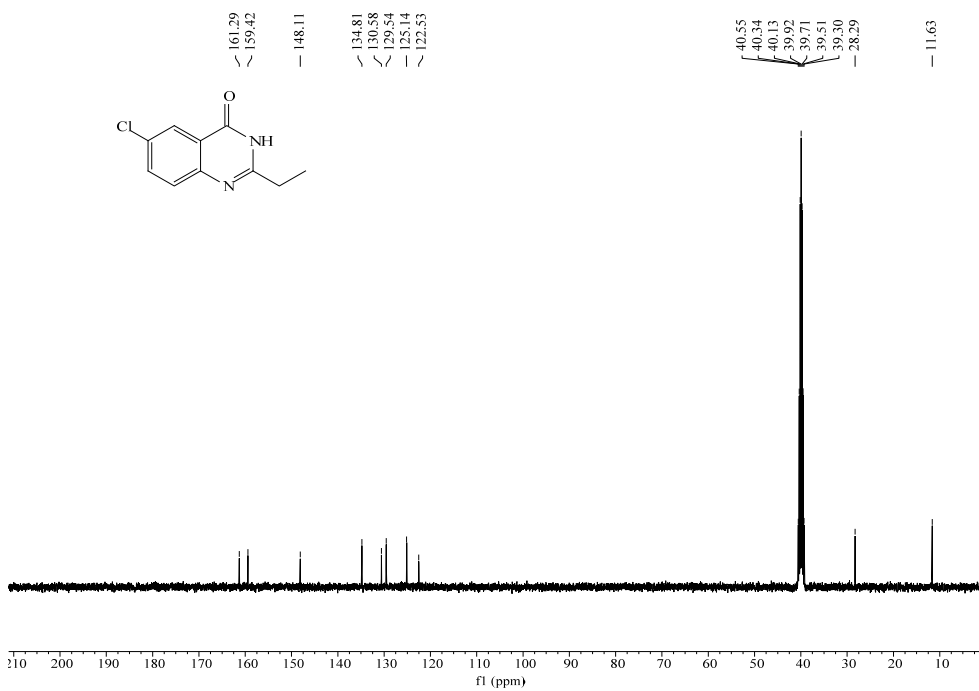
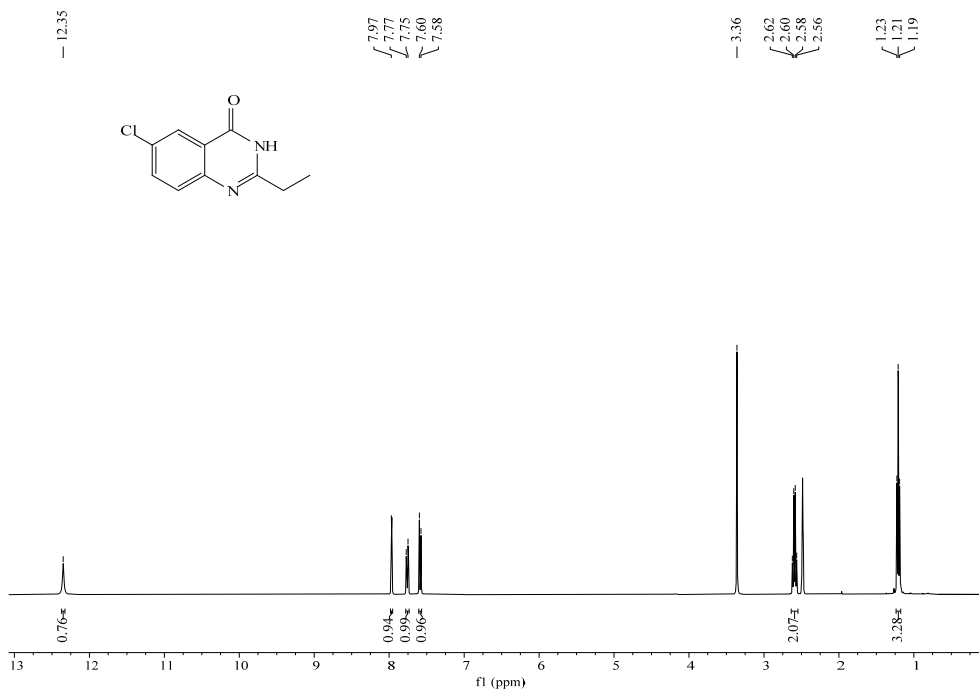
^1H and ^{13}C NMR spectra of 3c



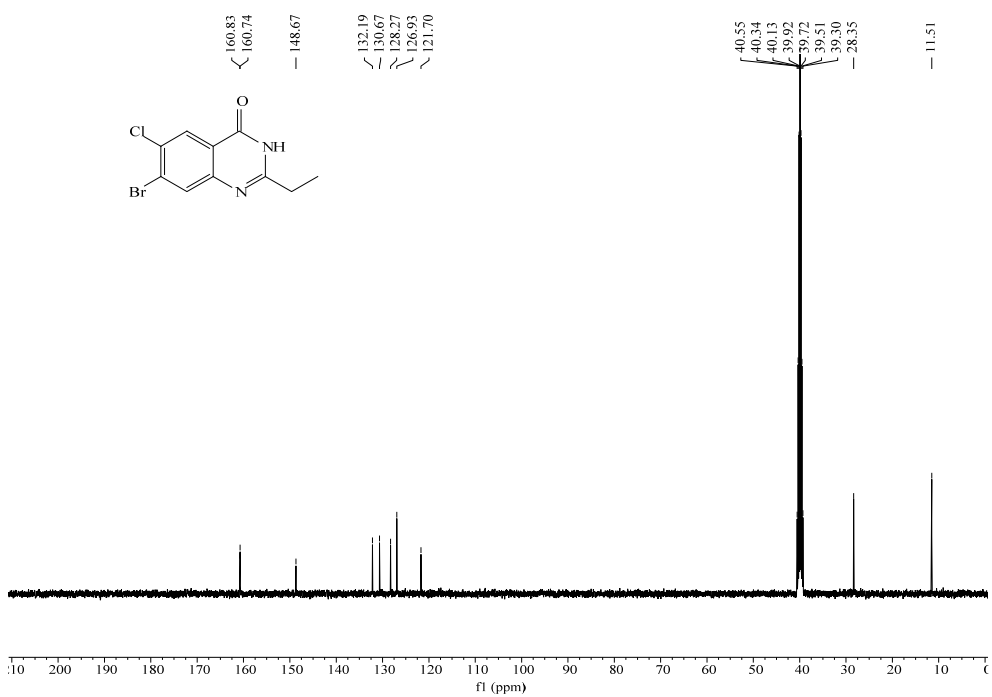
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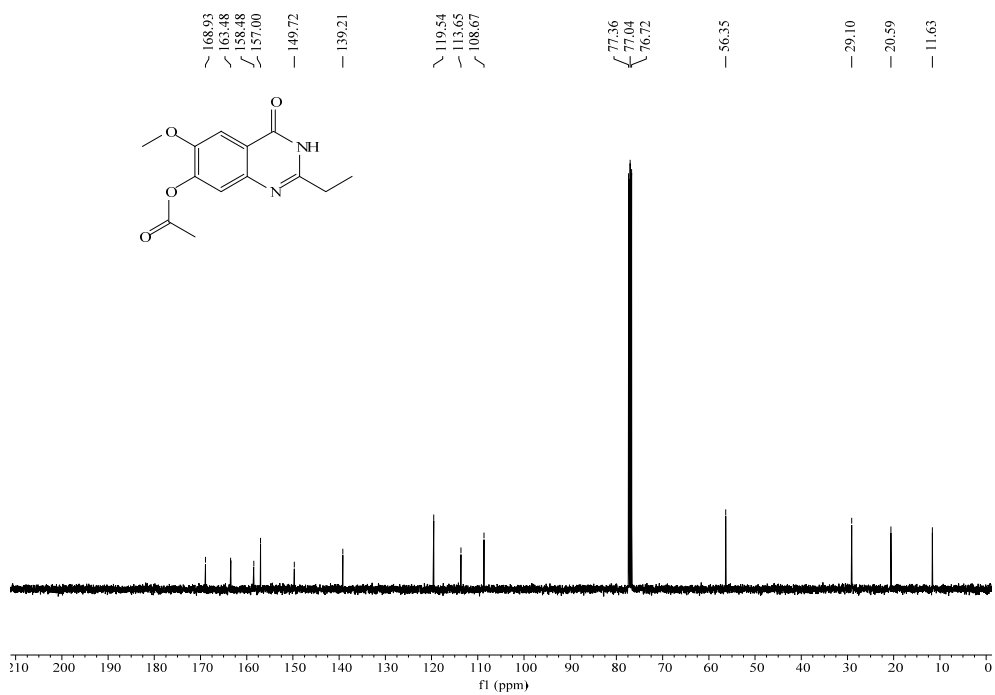
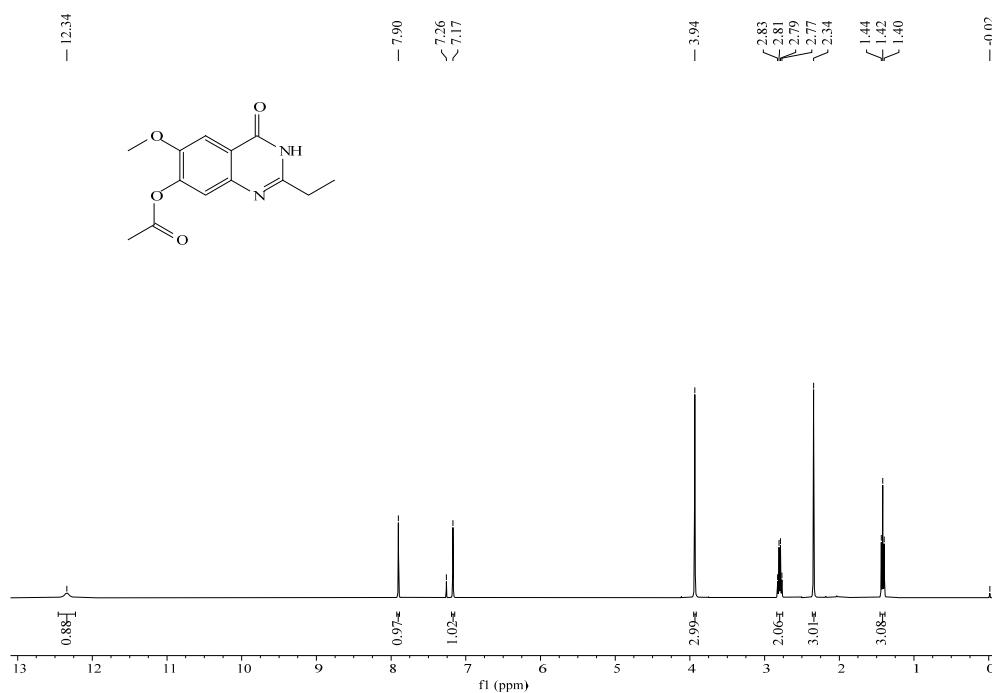
¹H and ¹³C NMR spectra of 3e



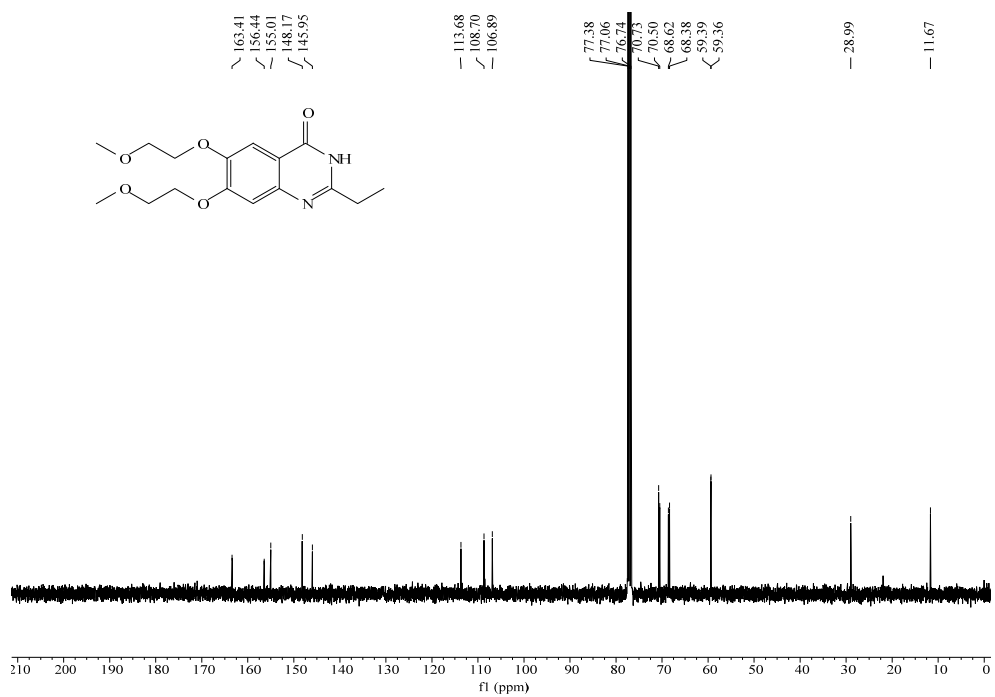
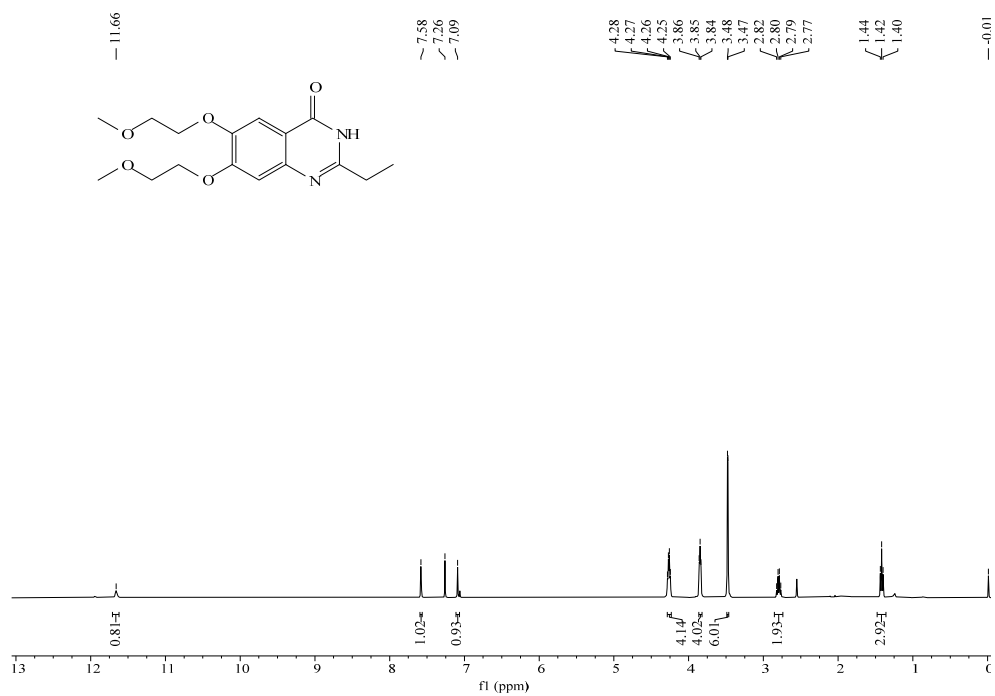
^1H and ^{13}C NMR spectra of 3f



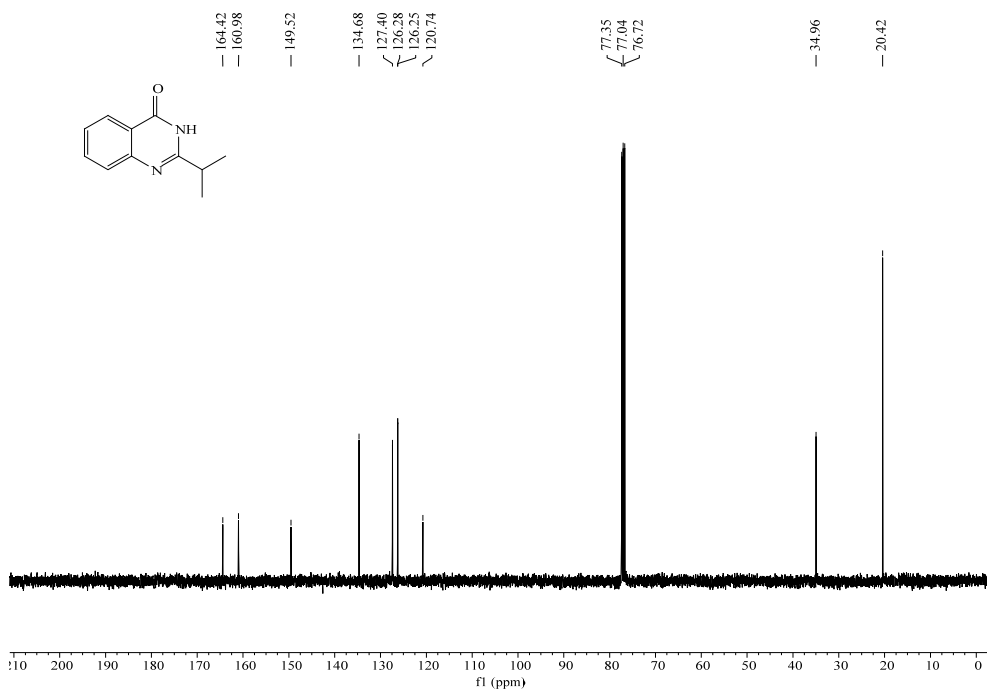
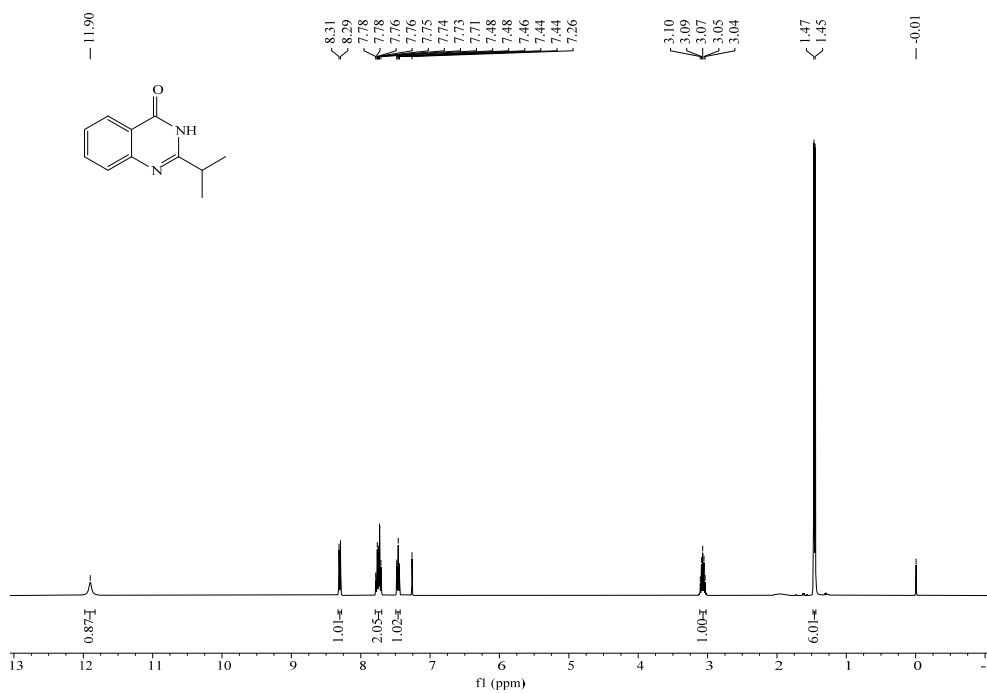
^1H and ^{13}C NMR spectra of 3g



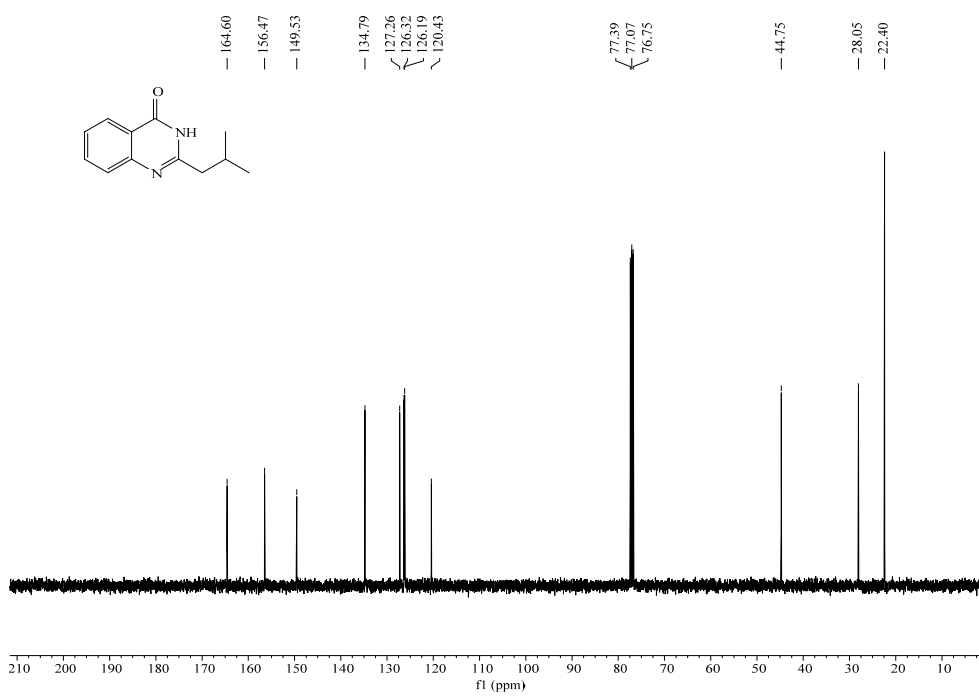
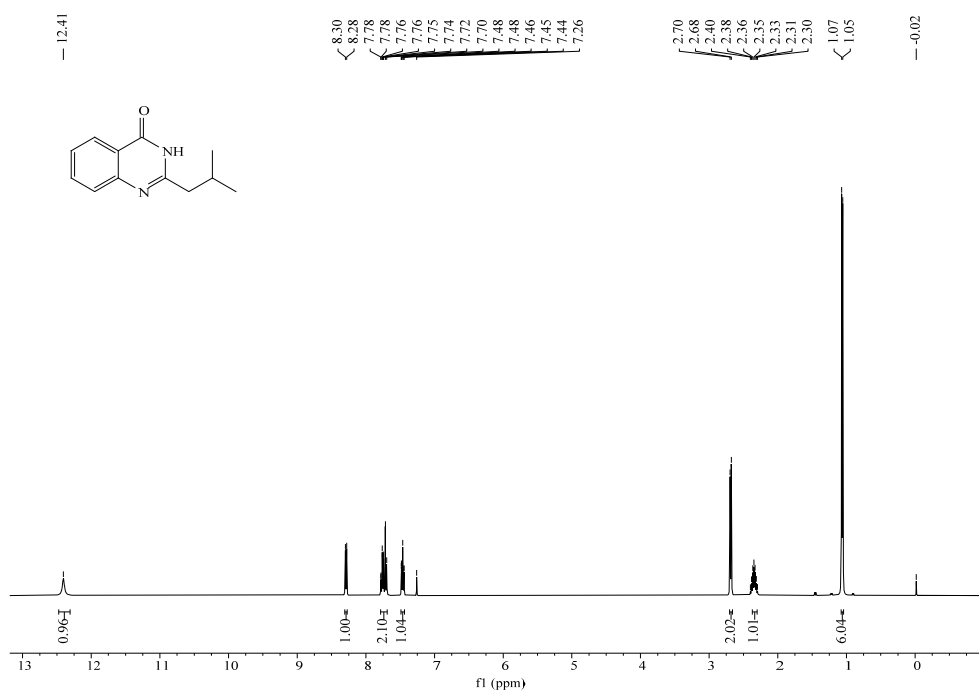
^1H and ^{13}C NMR spectra of 3h



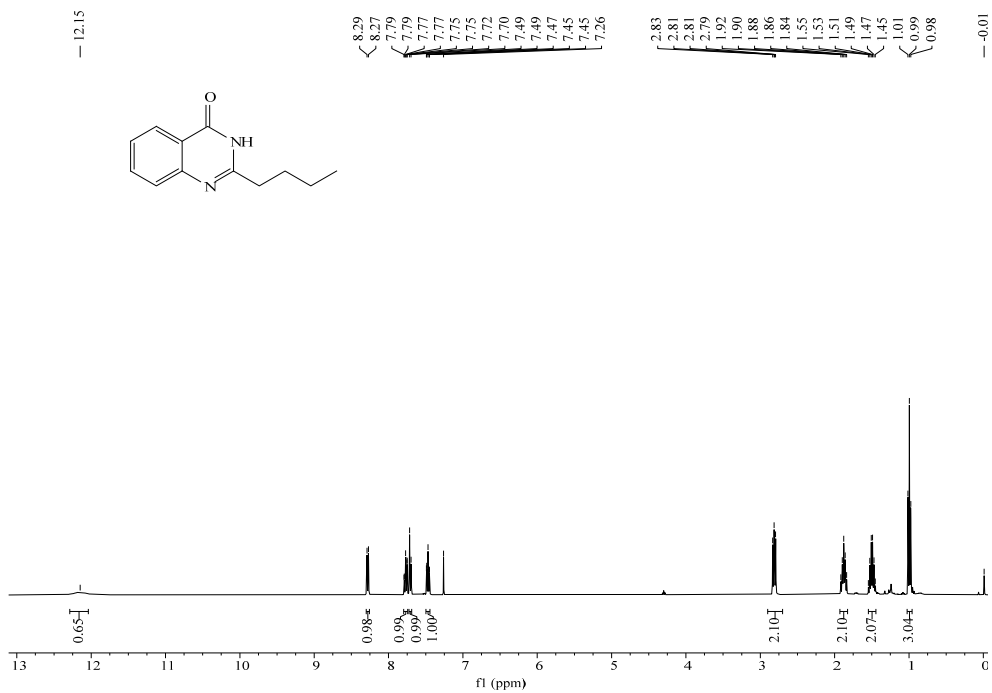
^1H and ^{13}C NMR spectra of 3i



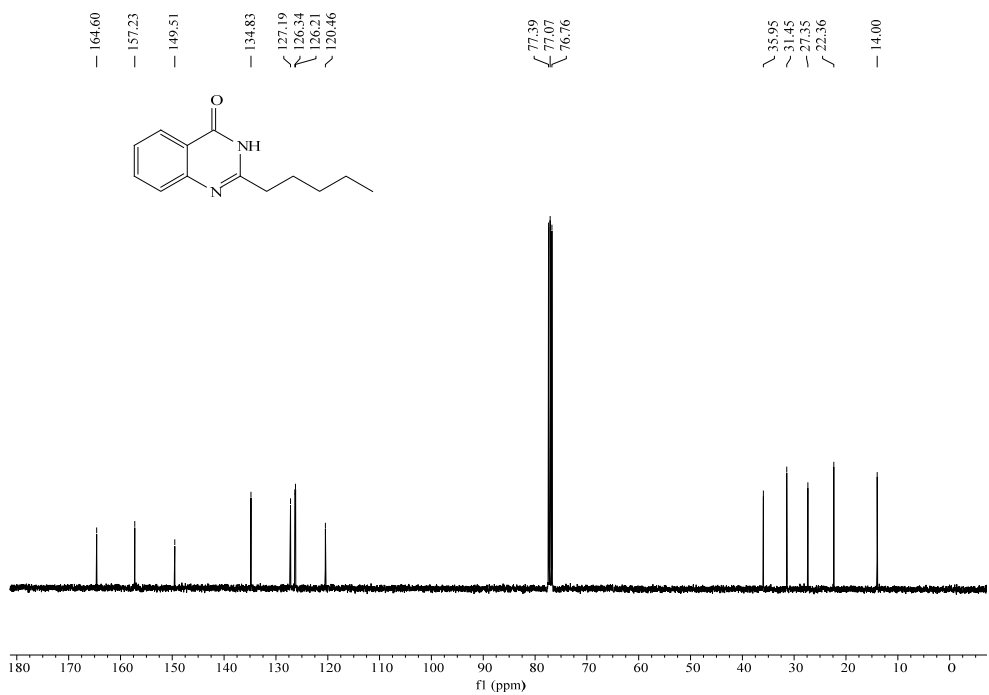
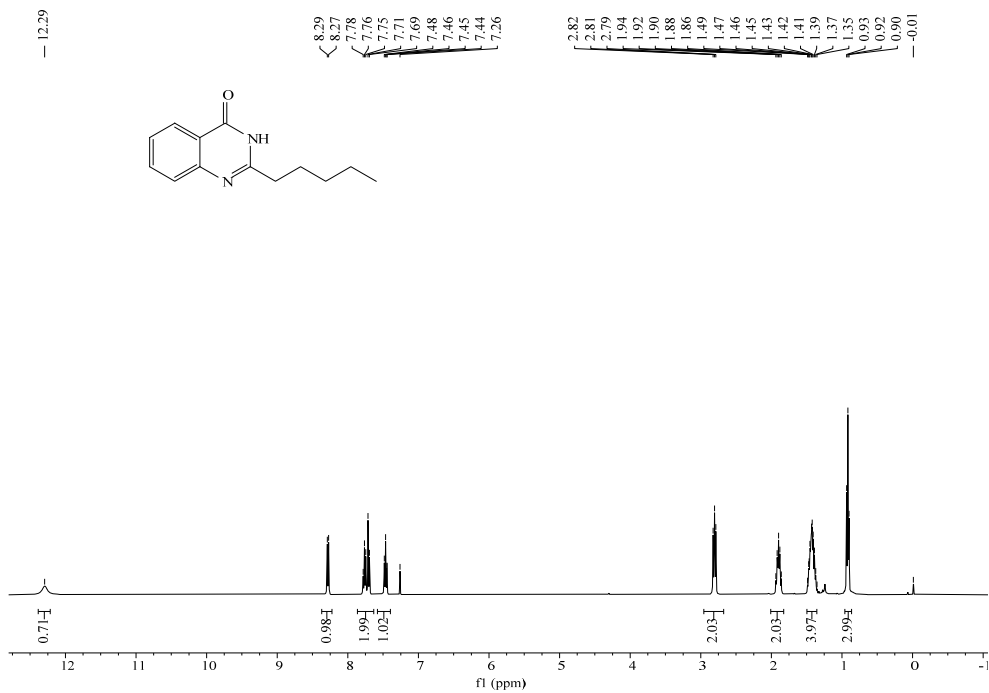
^1H and ^{13}C NMR spectra of 3j



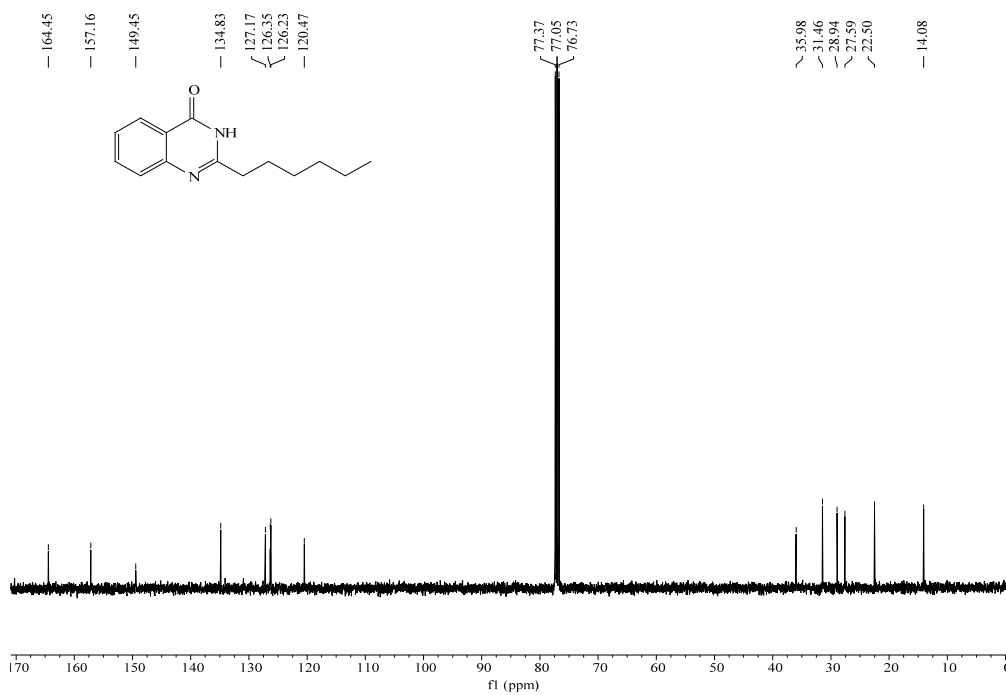
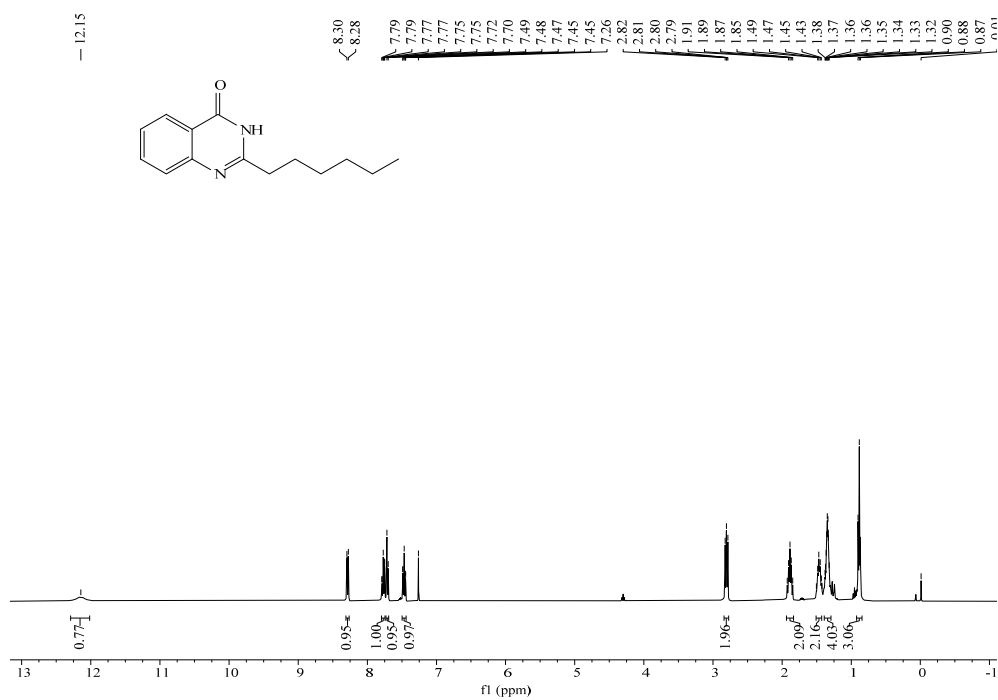
^1H and ^{13}C NMR spectra of 3k



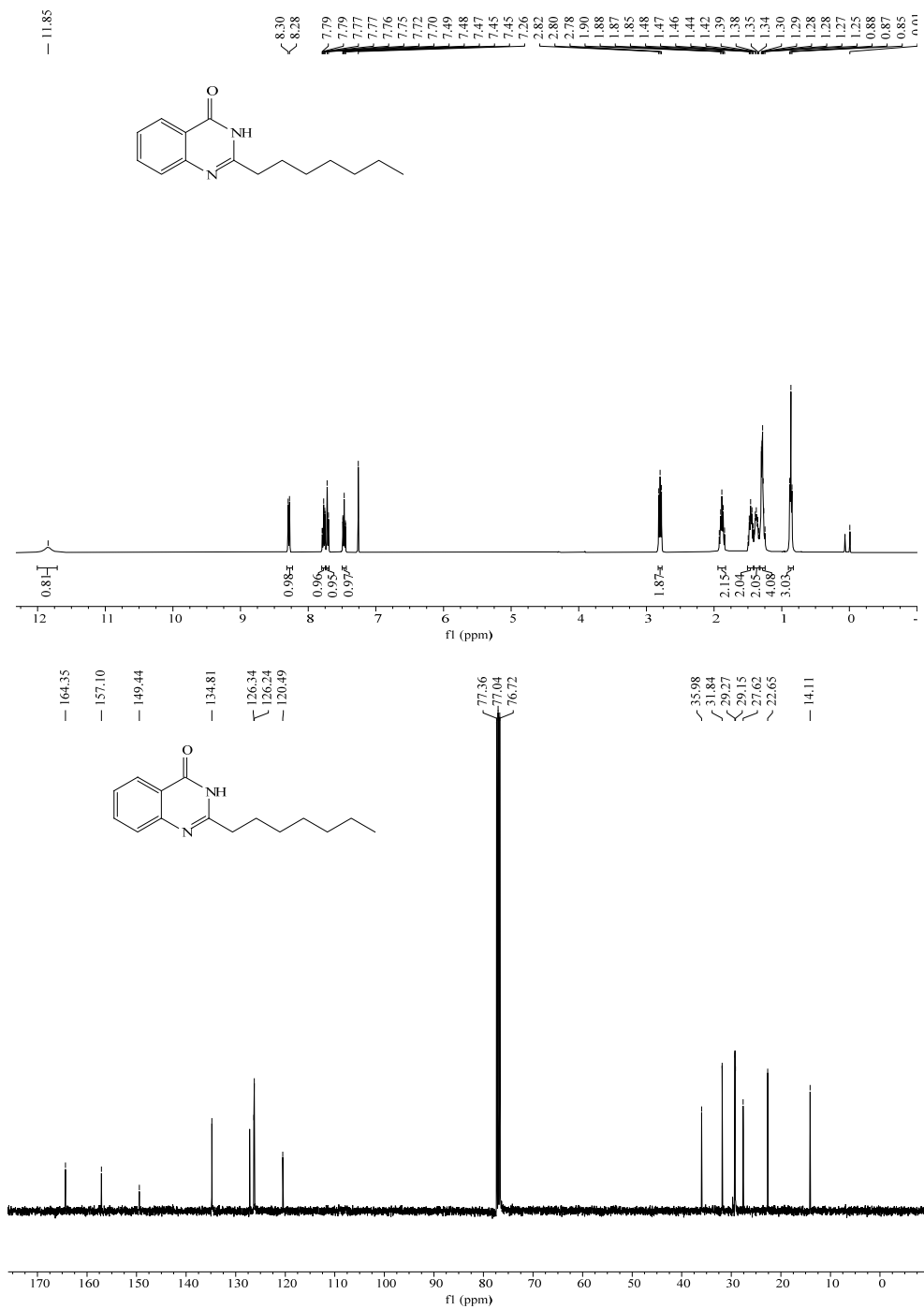
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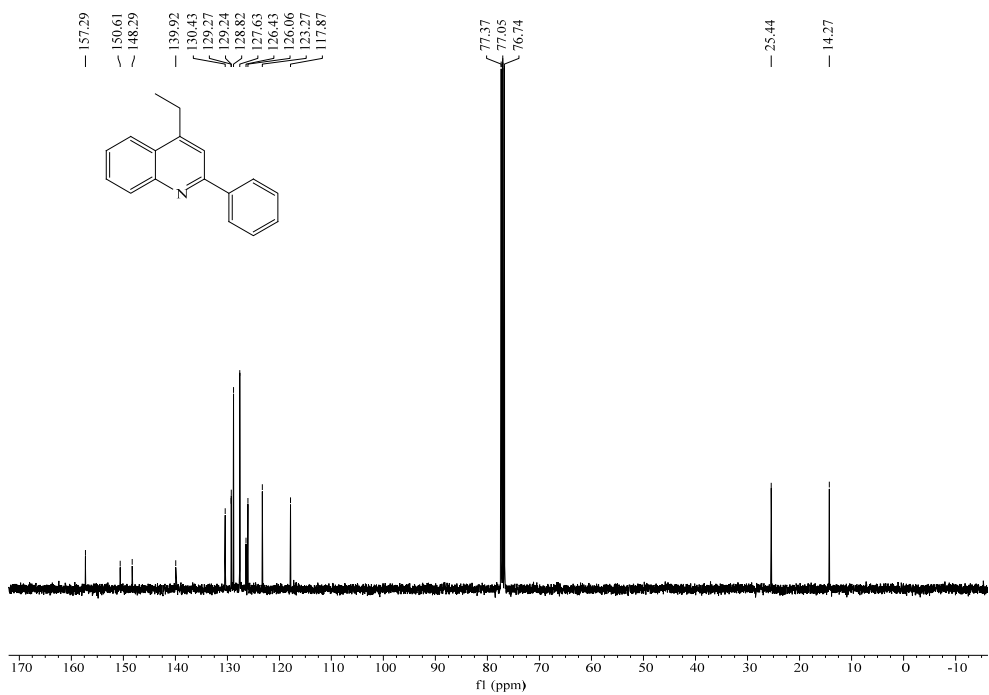
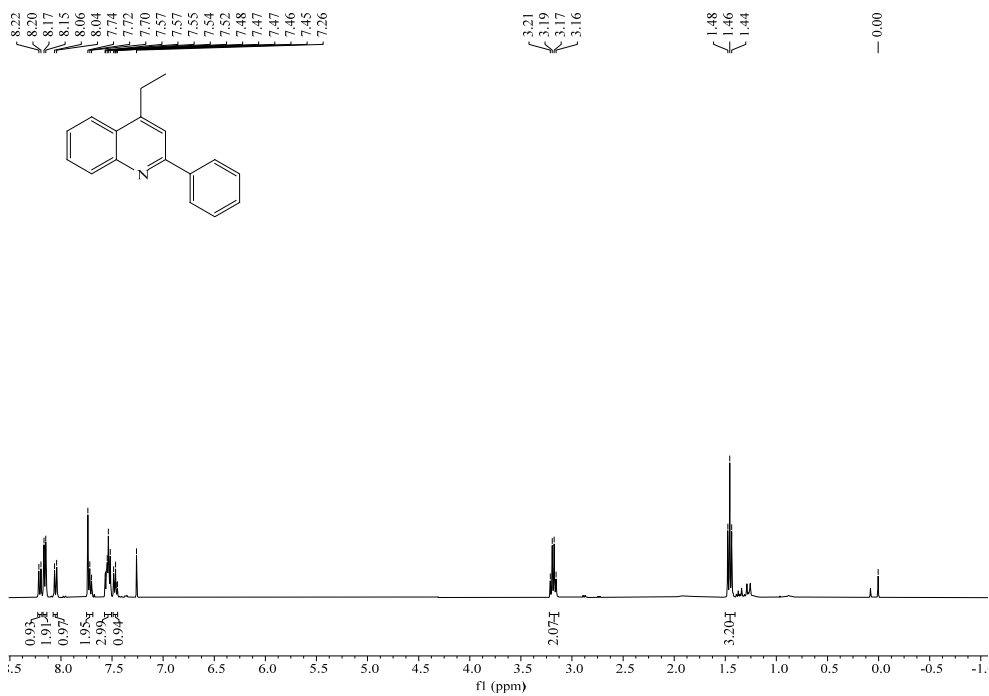
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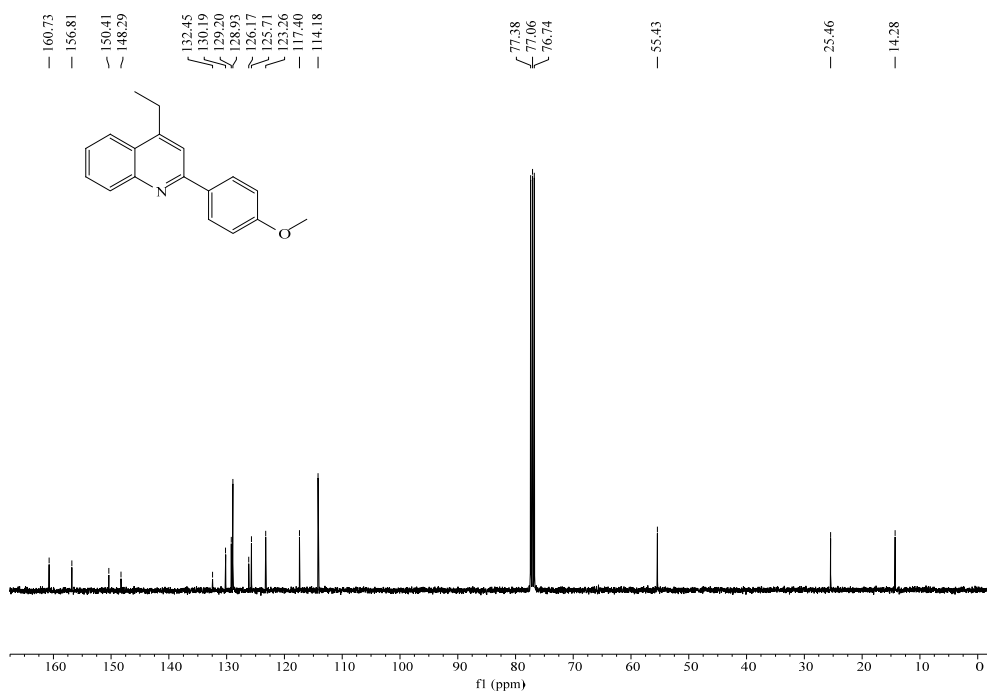
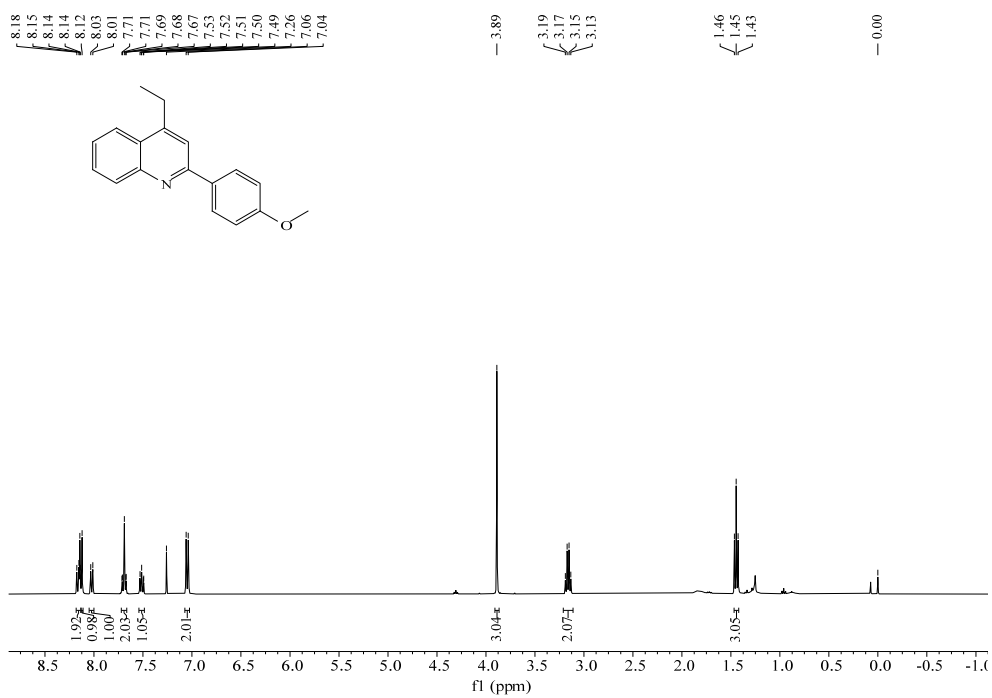
^1H and ^{13}C NMR spectra of 3n



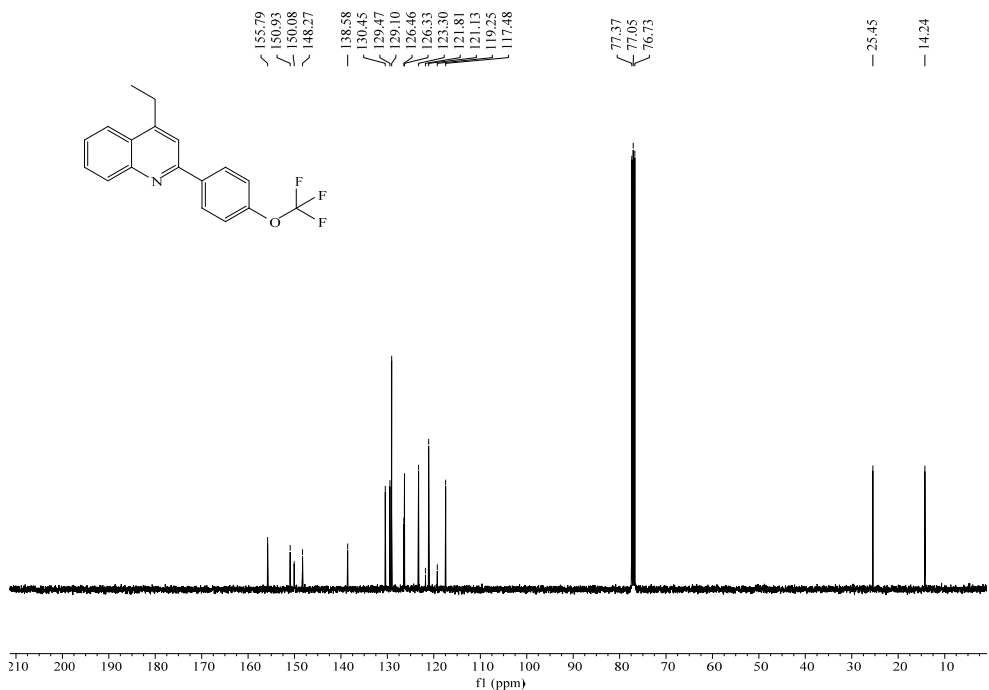
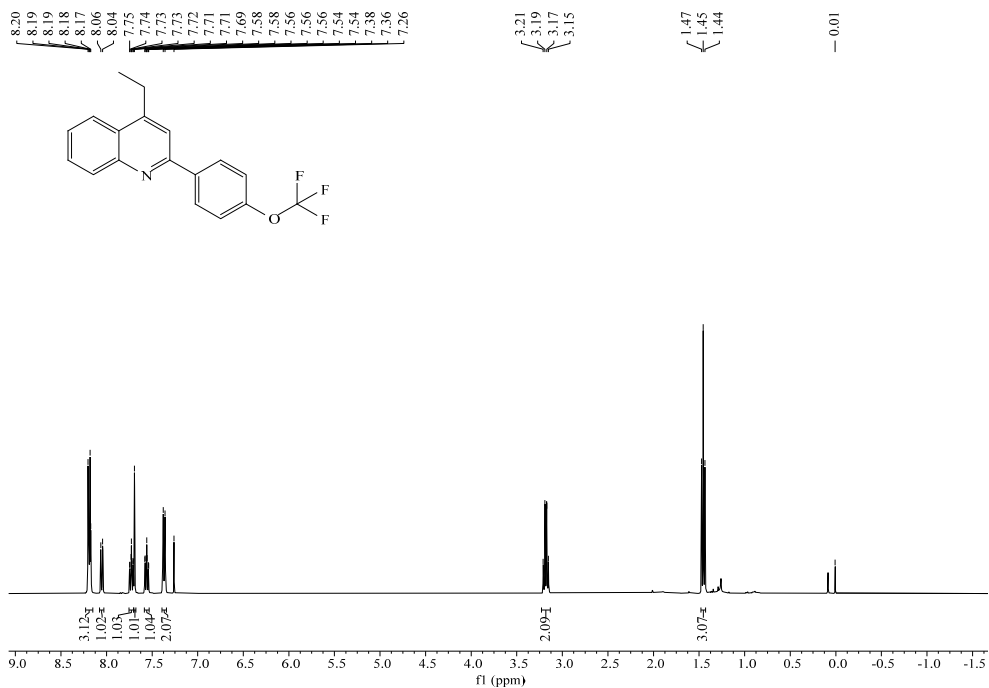
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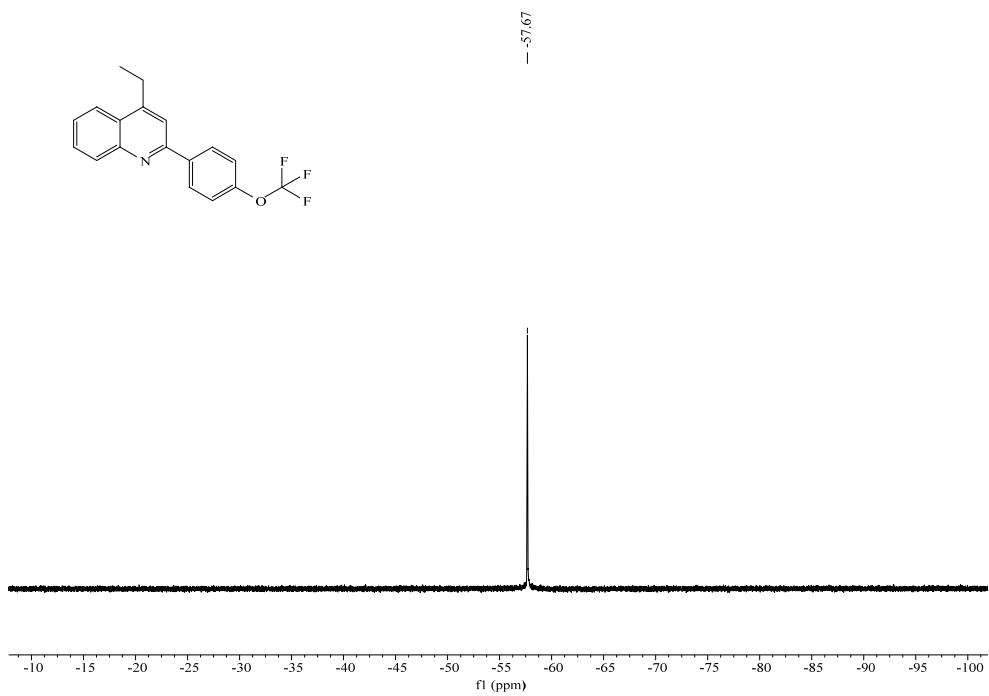


^1H and ^{13}C NMR spectra of 5b

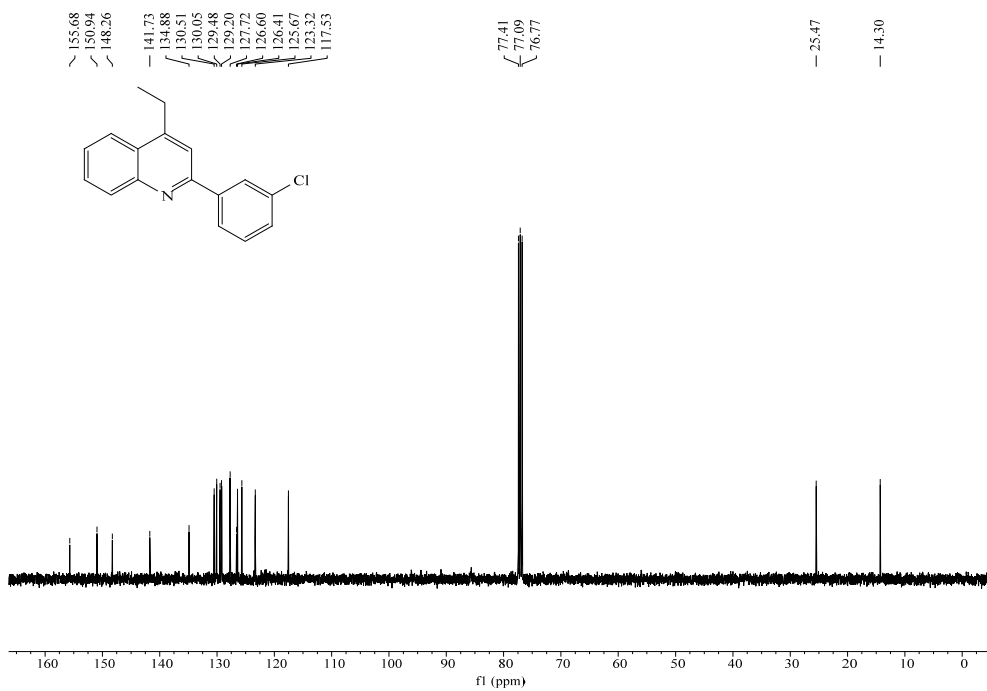
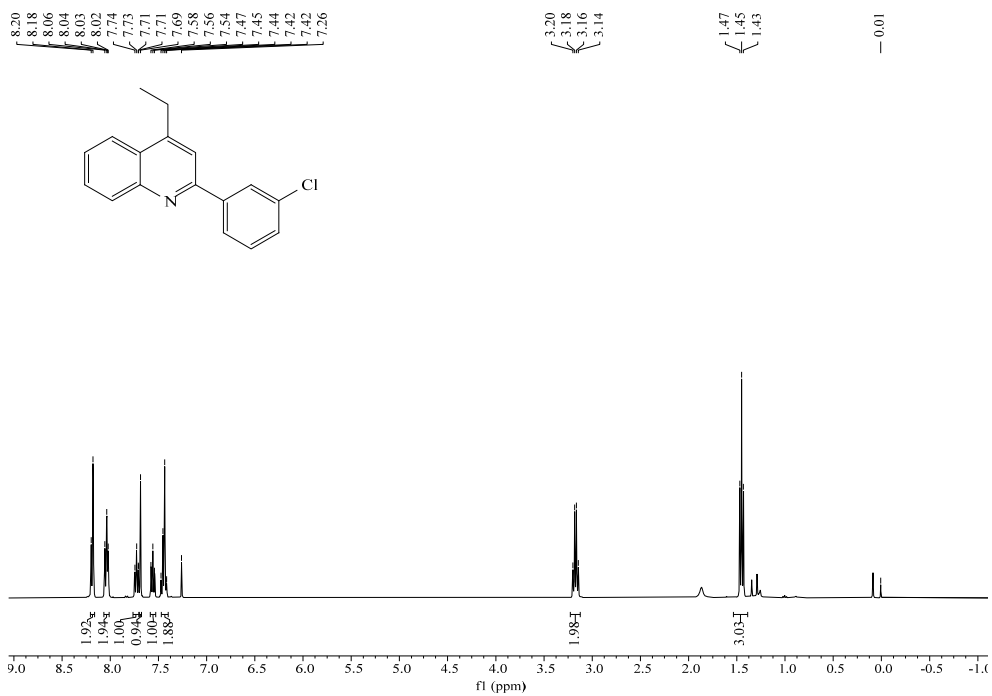


^1H NMR and ^{13}C NMR and ^{19}F NMR spectra of 5c

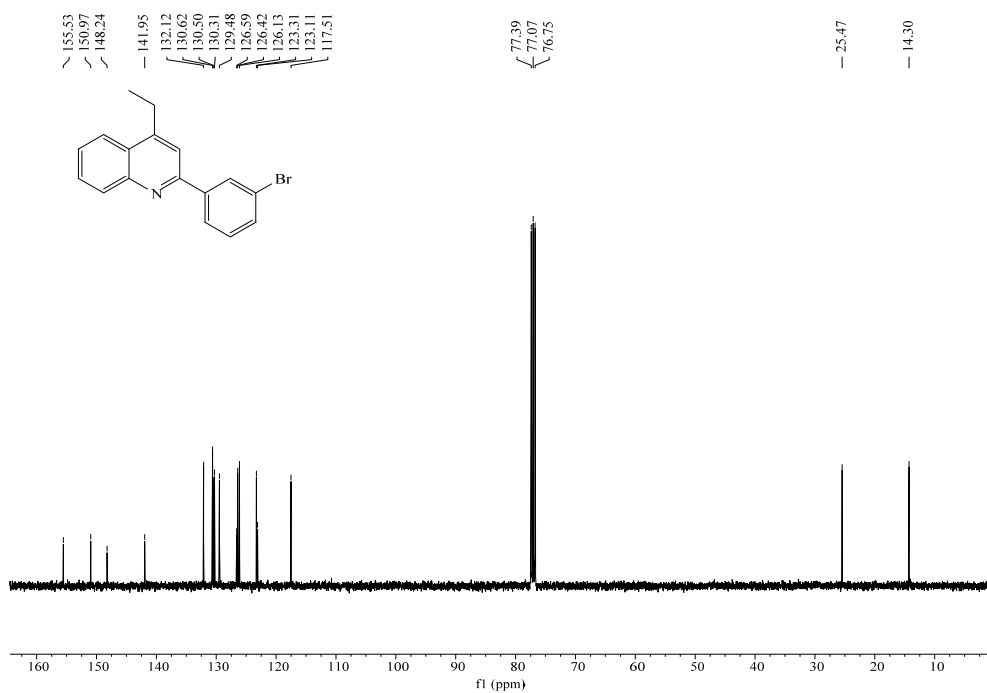
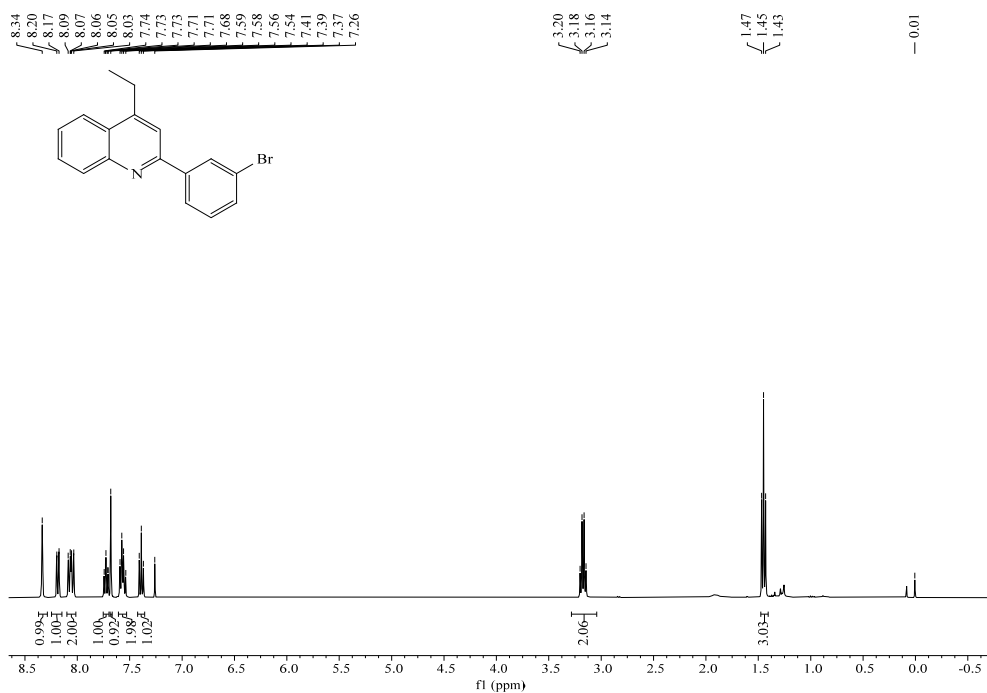




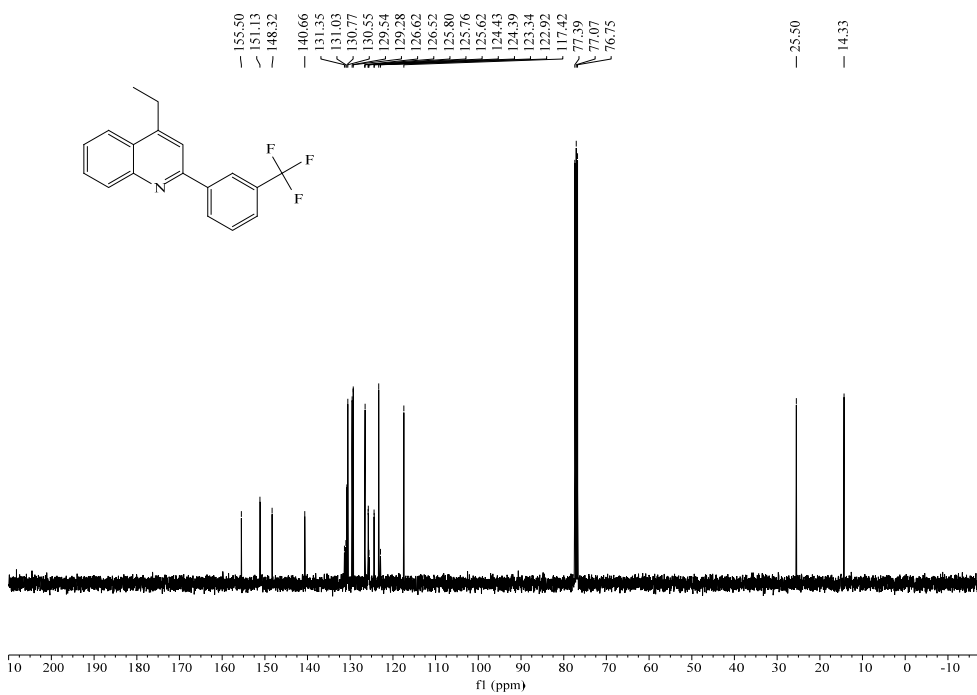
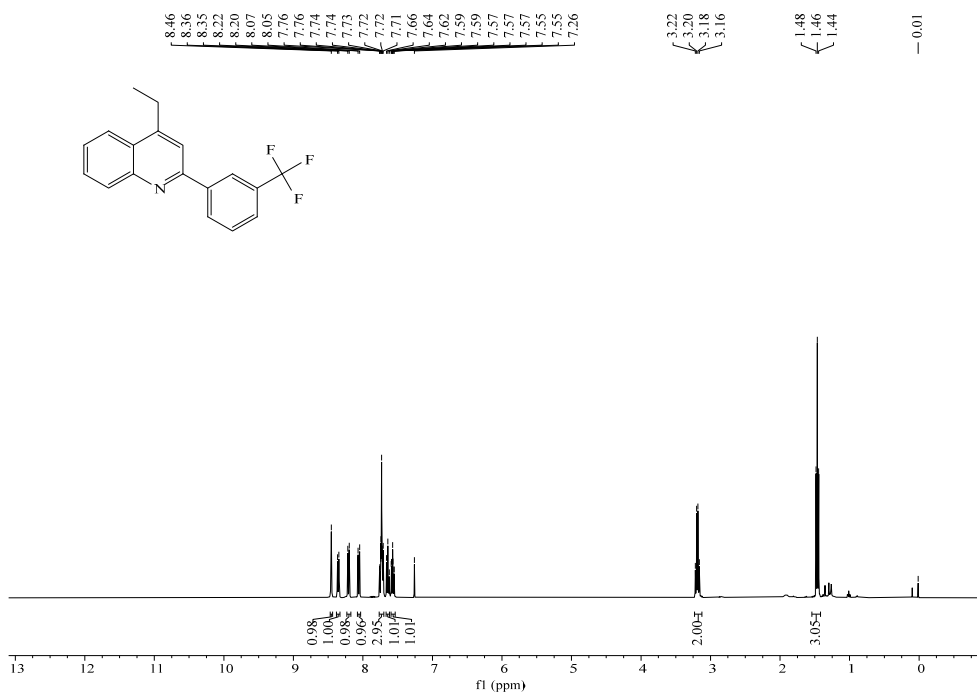
^1H and ^{13}C NMR spectra of 5d



^1H and ^{13}C NMR spectra of 5e

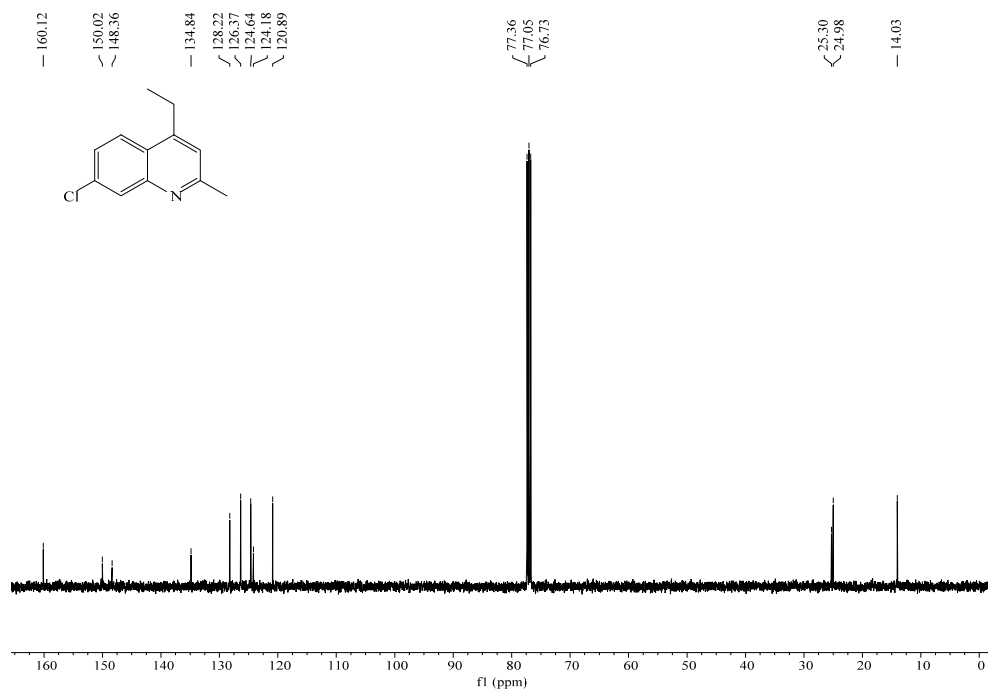
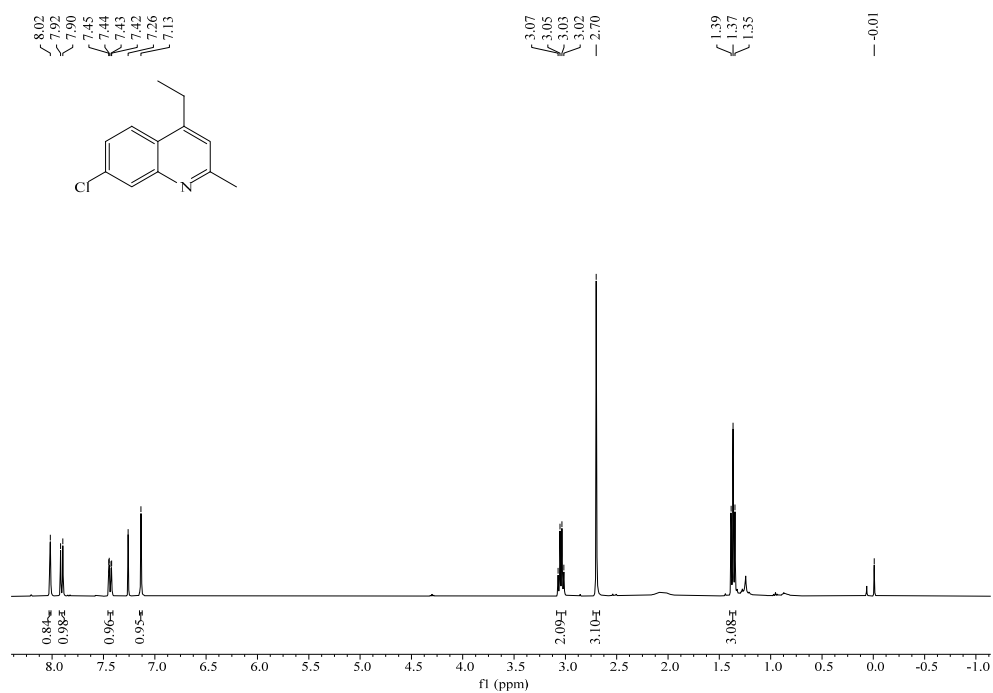


^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **5f**

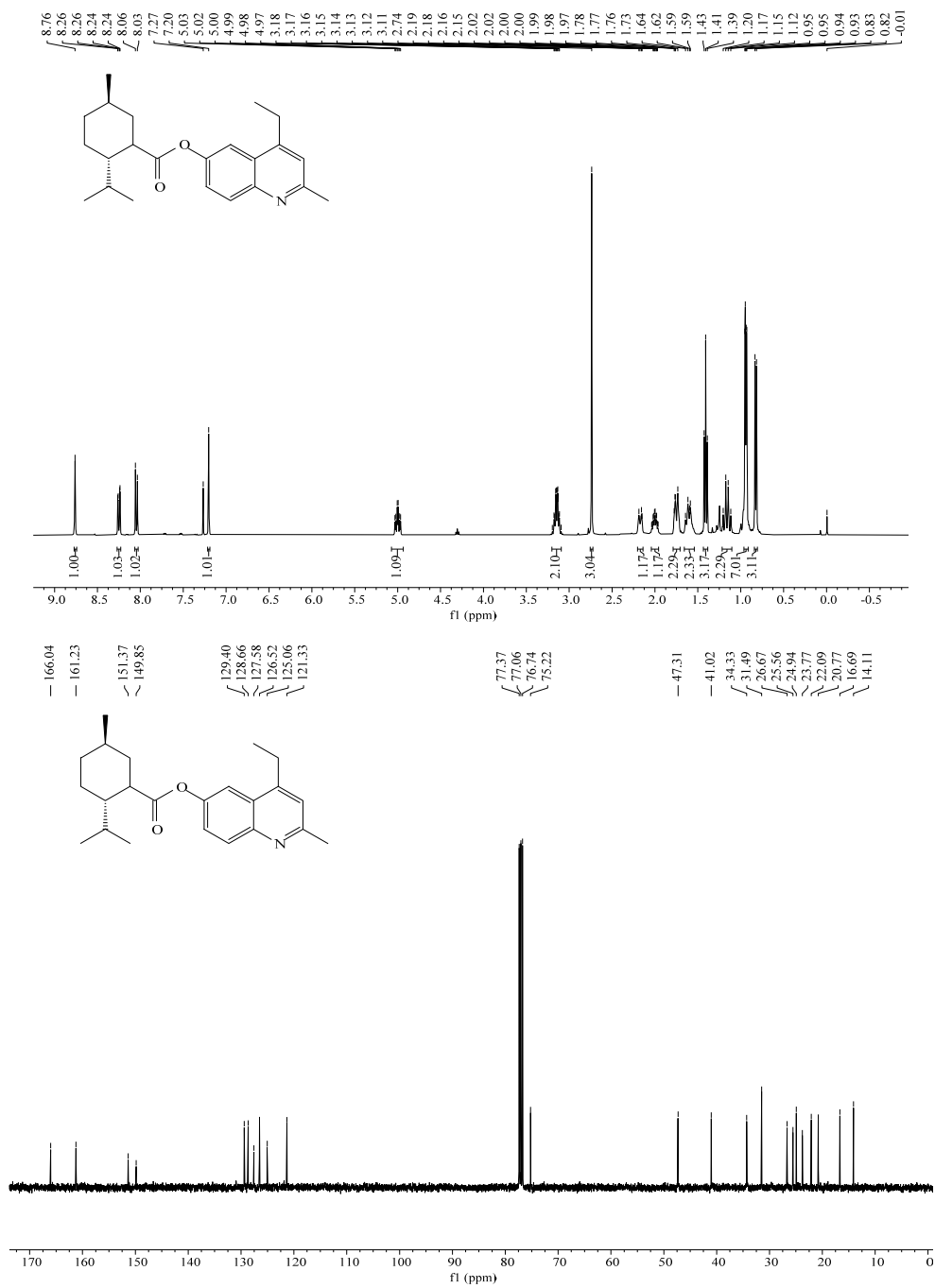




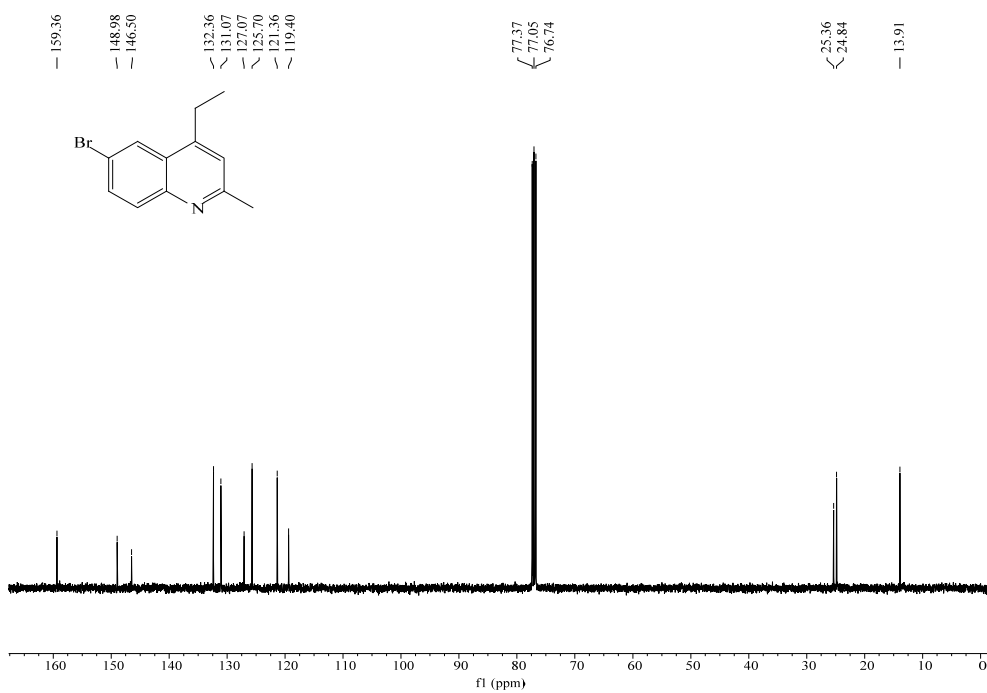
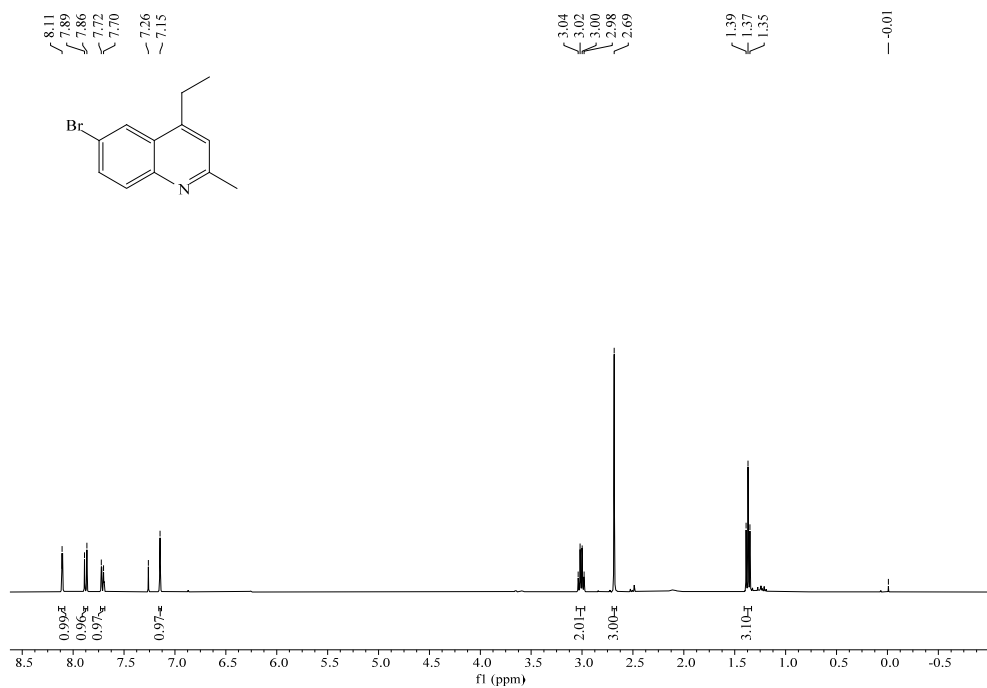
^1H and ^{13}C NMR spectra of 5g



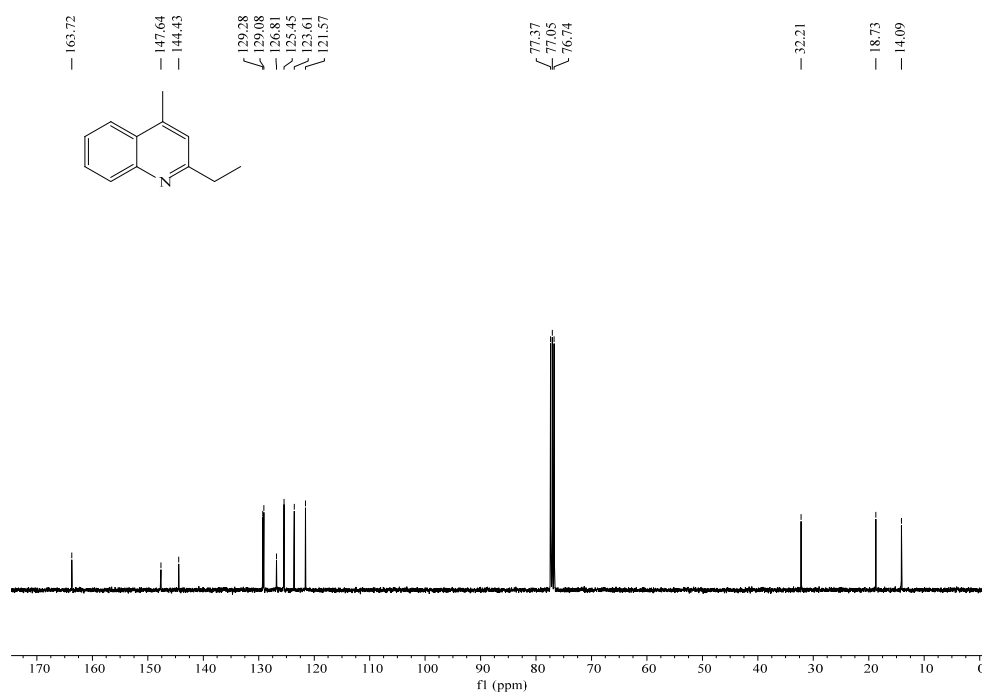
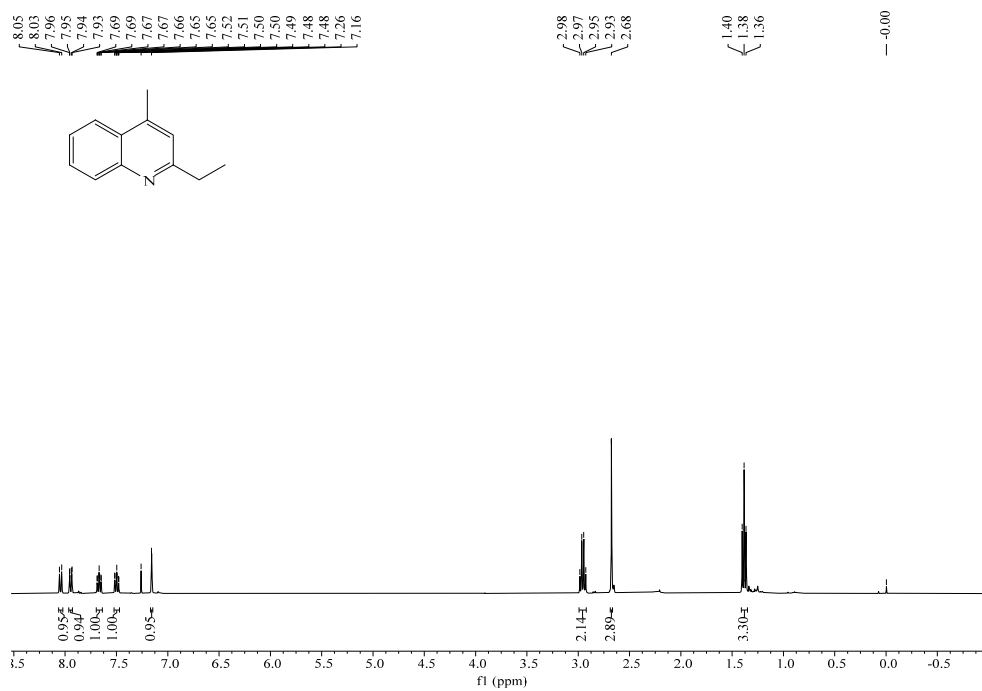
¹H and ¹³C NMR spectra of 5h



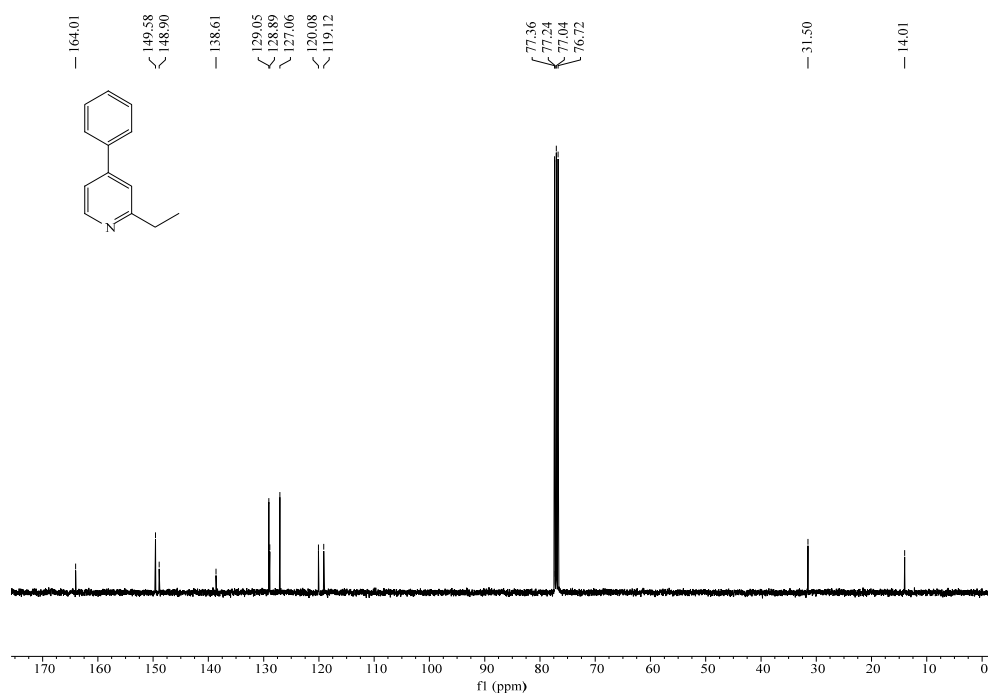
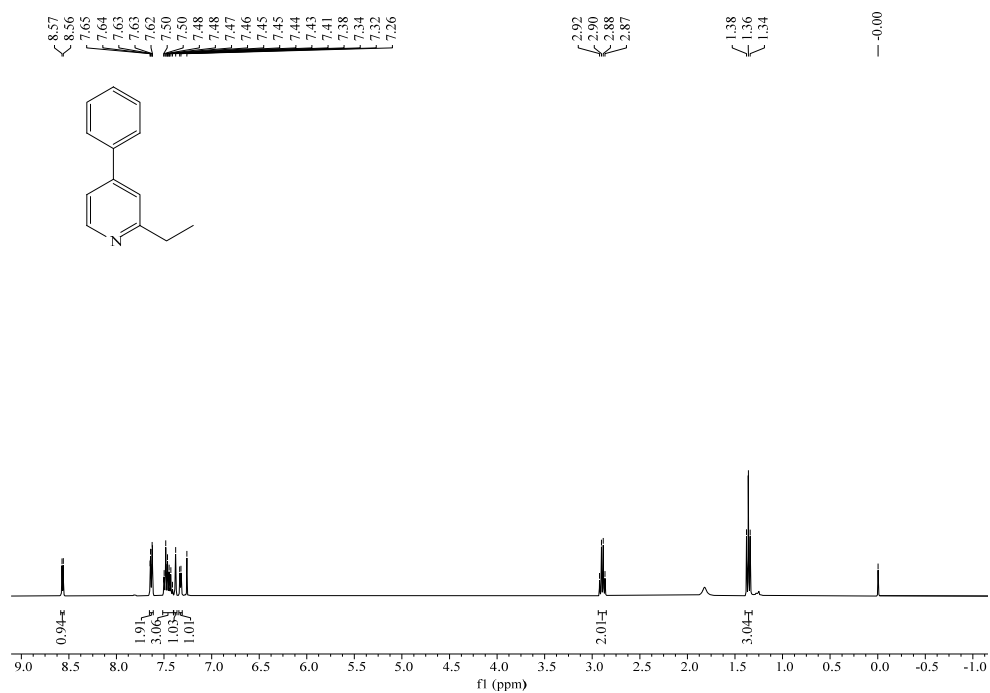
^1H and ^{13}C NMR spectra of 5i



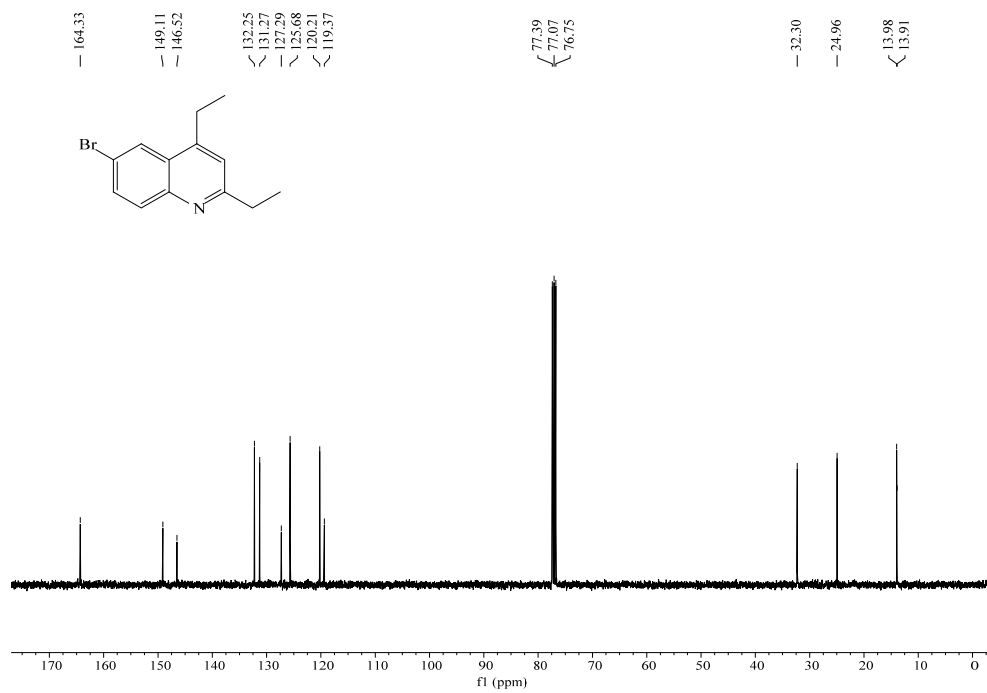
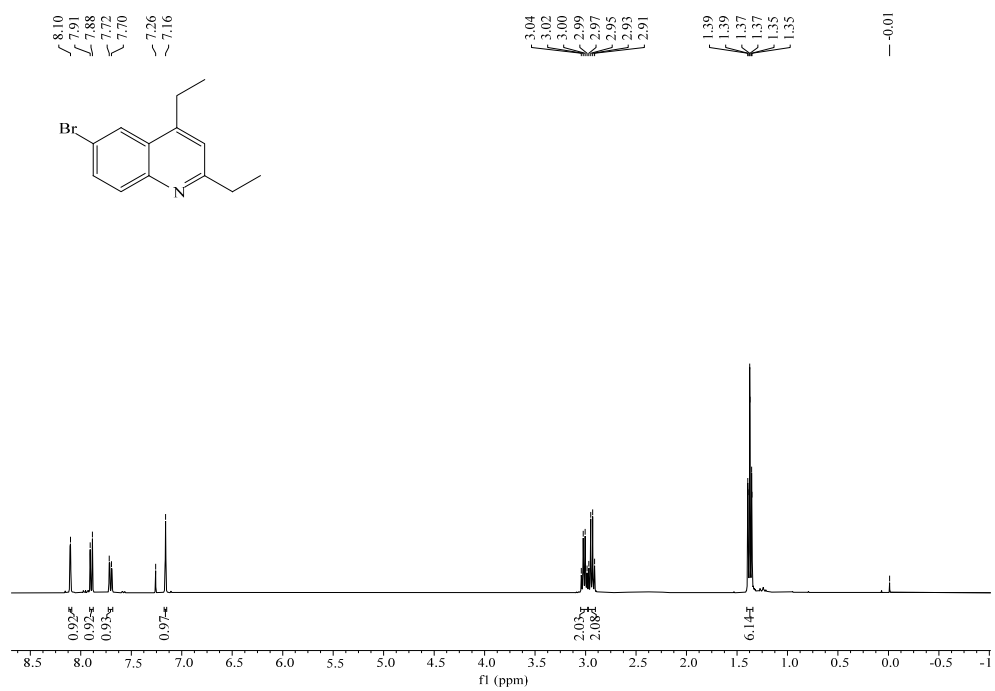
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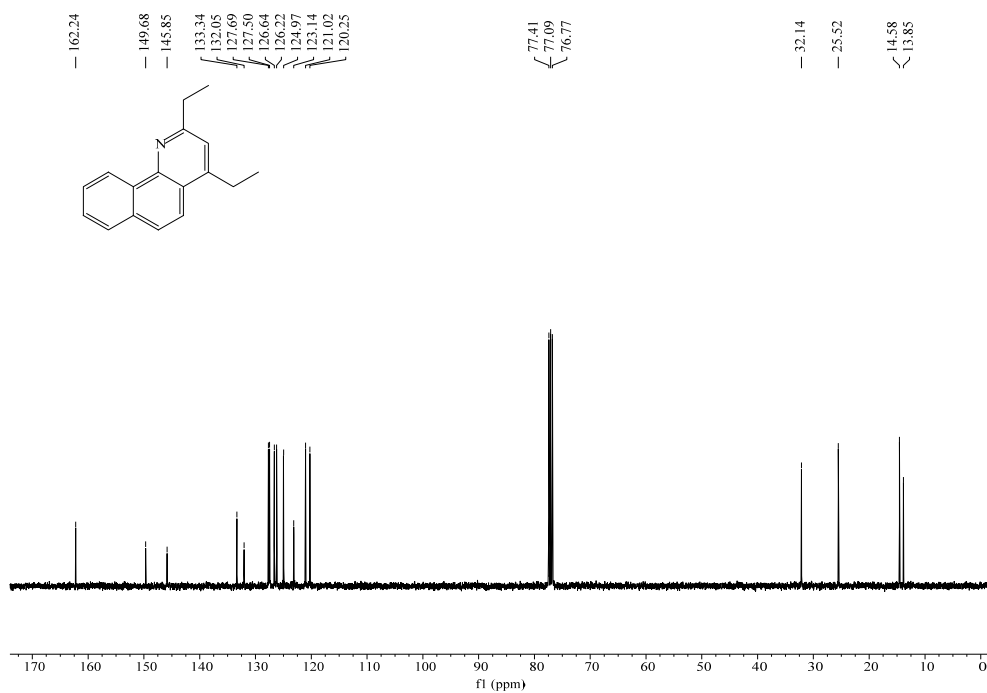
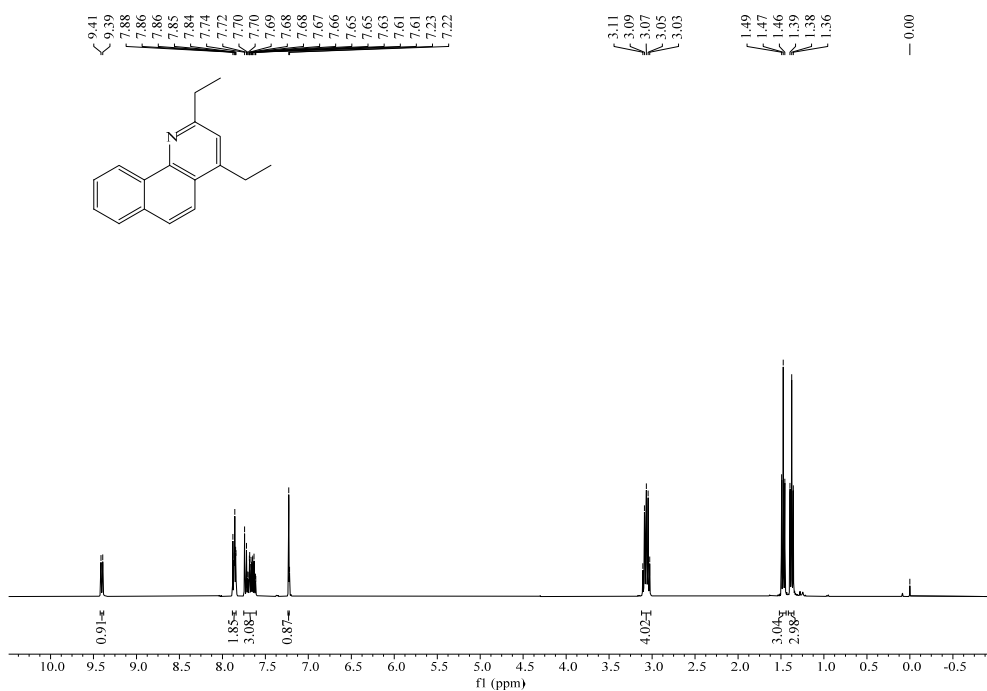
¹H and ¹³C NMR spectra of 5k



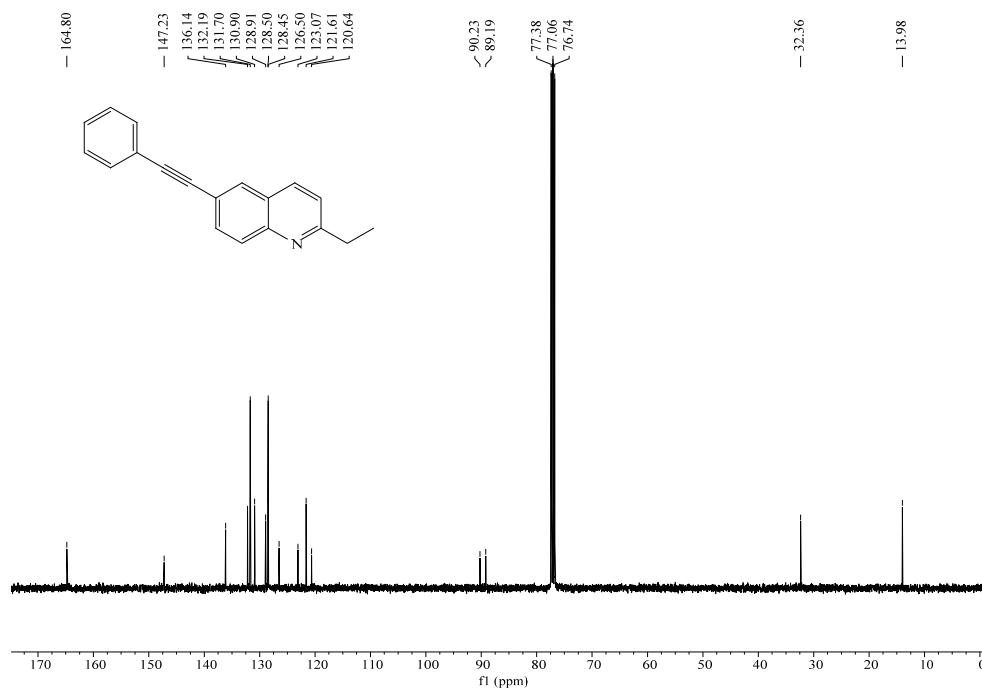
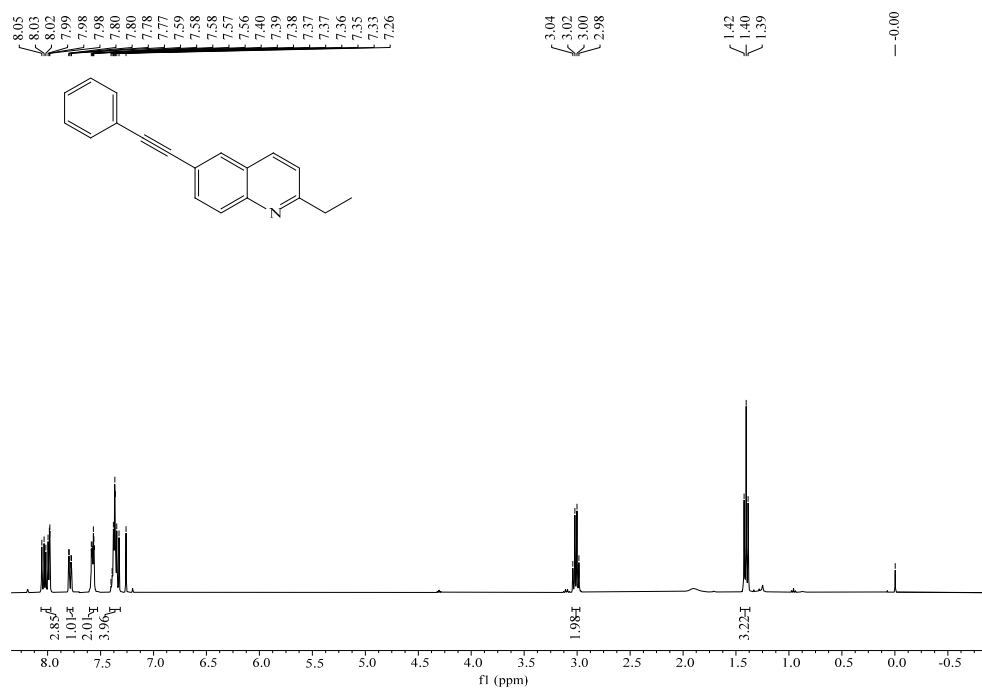
^1H and ^{13}C NMR spectra of 5l



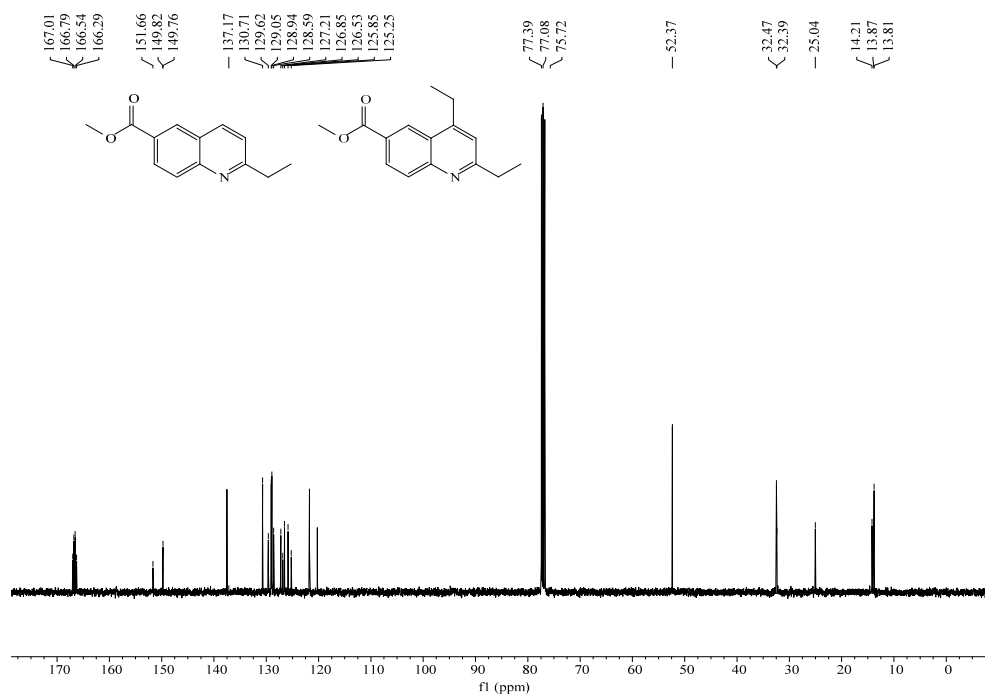
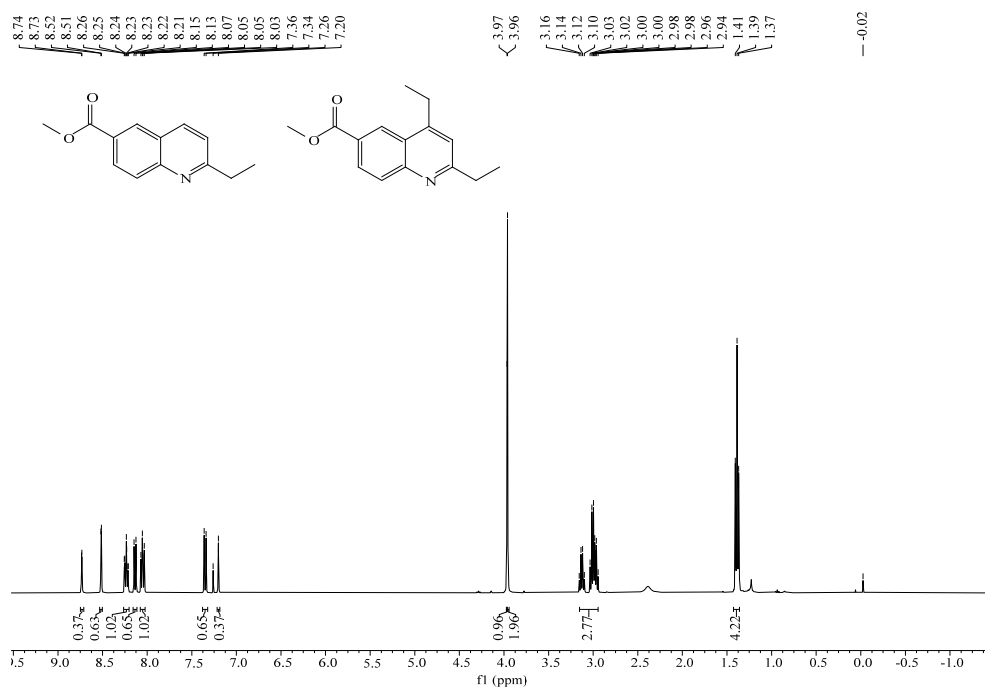
^1H and ^{13}C NMR spectra of 5n



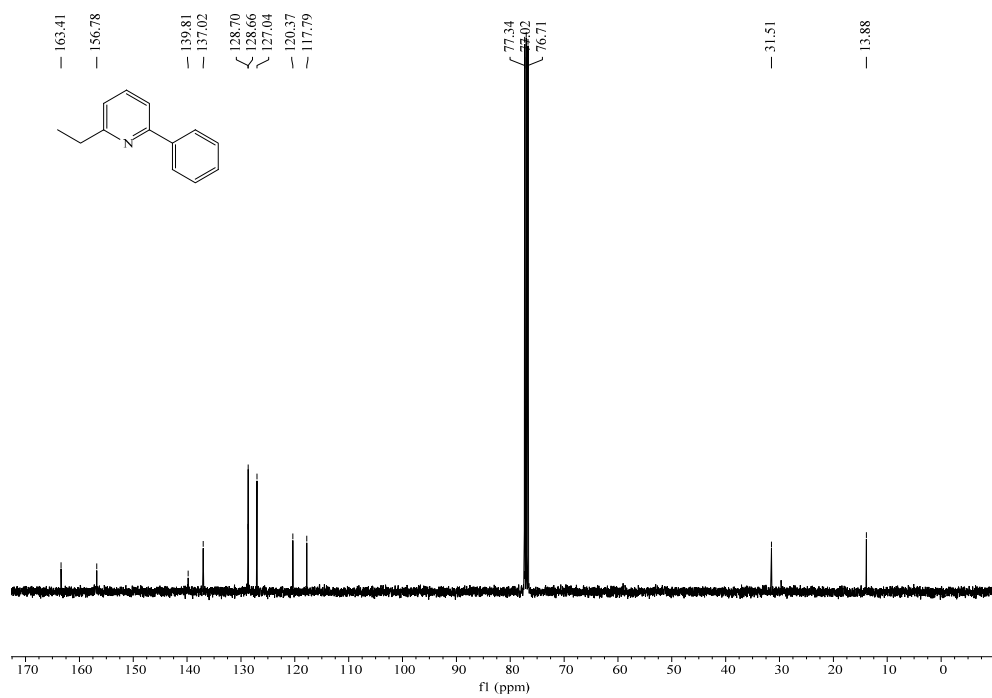
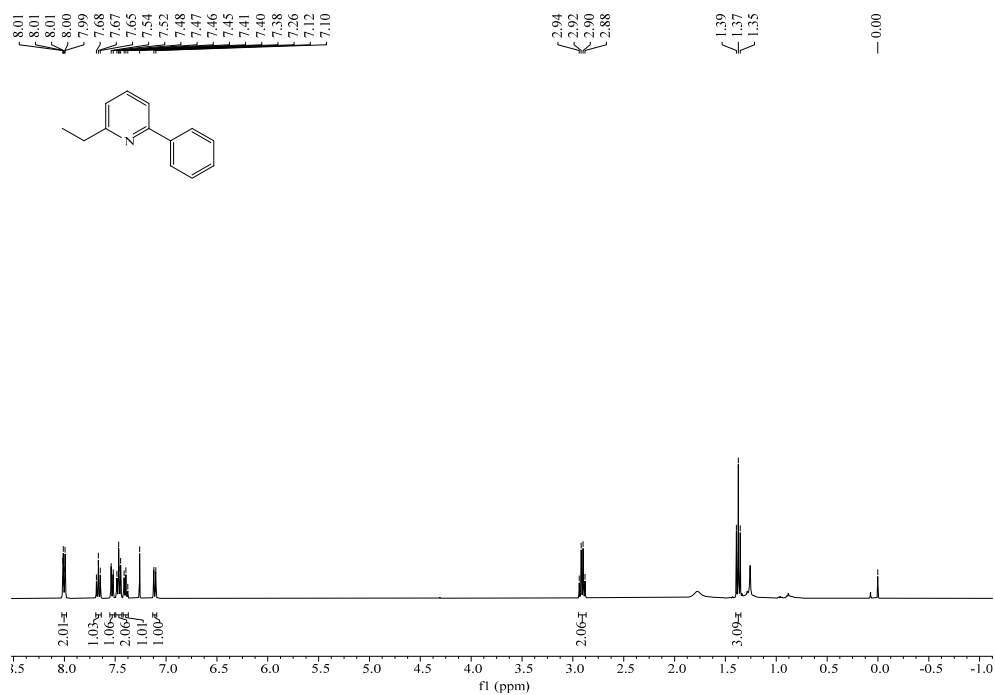
^1H and ^{13}C NMR spectra of 5n



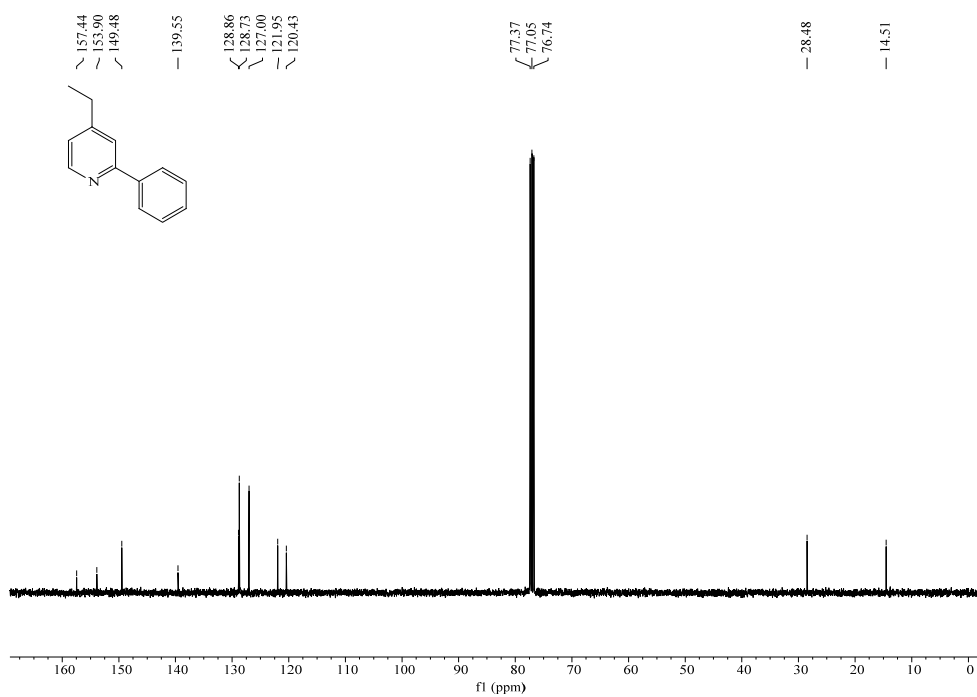
^1H and ^{13}C NMR spectra of 5o, 5p



¹H and ¹³C NMR spectra of 5q



^1H and ^{13}C NMR spectra of 5r



¹H and ¹³C NMR spectra of 5s

