

Synthesis of Isoquinolines via Rh(III)-Catalyzed C–H Activation Using Hydrazone as a New Oxidizing Directing Group

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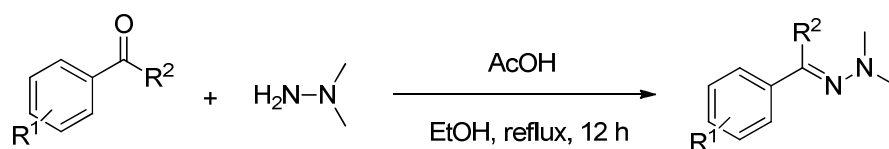
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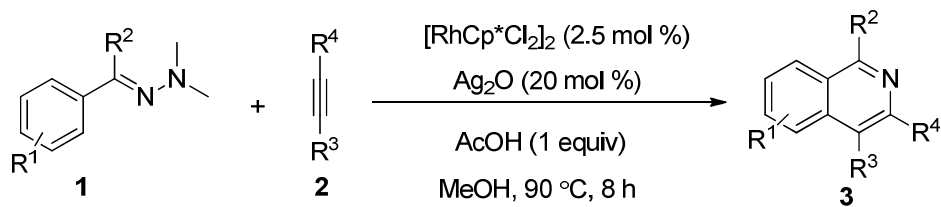
General. All reactions were conducted under nitrogen atmosphere on a dual-manifold Schlenk line unless otherwise mentioned and in oven-dried glass wares. All solvents were dried according to known methods and distilled prior to use.¹ Aryl hydrazones were synthesized using known literature procedure.² Other reagents were commercially available and used as purchased.

General Procedure for the Preparation of *N,N*-Dimethyl Hydrazones²



A mixture of arylketone (15 mmol), anhydrous dimethylhydrazine (1.8 g, 30 mmol) and AcOH (0.090g, 1.5 mmol) in absolute ethanol (30 ml) was refluxed for 12 h. Excess dimethylhydrazine and ethanol were removed by evaporation under reduced pressure. The crude product was dissolved in EtOAc and washed with water and concentrated by rotary evaporation. Further purification by a short silica gel column using *n*-hexane/EtOAc as eluent gave the corresponding *N,N*-dimethyl hydrazones in 80-99% yields.

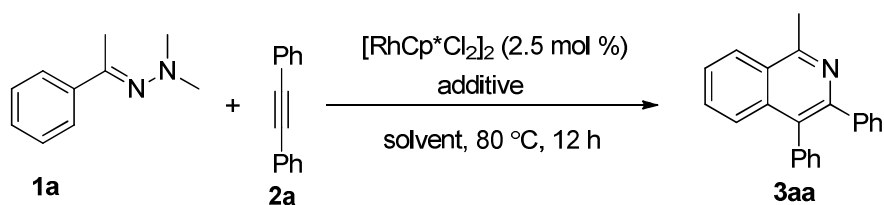
General Procedure for the Preparation of Isoquinolines from Arylhydrazones and Alkynes



A sealed tube containing aryl hydrazone **1** (0.20 mmol), alkyne **2** (0.24 mmol), [Cp*RhCl₂]₂ (0.0050 mmol, 2.50 mol %), and Ag₂O (0.04 mmol) was evacuated and purged with nitrogen gas three times. Then, AcOH (0.20 mmol) and MeOH (1.0 mL) were added to the system via a syringe under nitrogen atmosphere and the reaction was allowed to stir at 90 °C for 8-24 h. When the reaction was completed, the mixture was diluted with CH₂Cl₂ (10 mL) and filtered through a Celite pad and the Celite pad was washed several times with CH₂Cl₂ (50 mL). The combined filtrate was concentrated in vacuo and the residue was purified by column chromatography on a silica gel column using *n*-hexane/EtOAc as eluent to afford the desired pure product **3**.

It should be noted that for the synthesis of compounds **3aa**, **3ia**, **3ja**, **3ka**, **3la** and **3aj**, Ag₂O was not required (See Table 1 in main text).

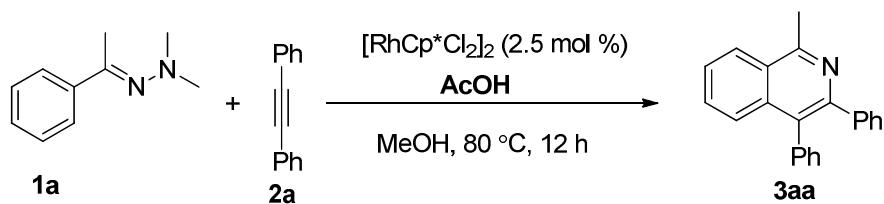
Table 1. Optimization Studies for the Rh(III)-Catalyzed Isoquinoline Formation^a



entry	additive	solvent	yield % ^b
1	AgOAc	MeOH	17
2	NaOAc	MeOH	30
3	KOAc	MeOH	23
4	CsOAc	MeOH	25
5	DCA	MeOH	25
6	AcOH	MeOH	98
7	AcOH	MeOH	60 ^c
8	AcOH	toluene	-
9	AcOH	1,4-dioxane	8
10	AcOH	EtOH	65
11	AcOH	DCE	12
12	AcOH	THF	-
13	AcOH	EtOAc	16
14	AcOH	acetone	20
15	AcOH	CH ₃ CN	-
16	-	MeOH	trace

^aUnless otherwise mentioned, all reactions were carried out using **1a** (33 mg, 0.2 mmol), **2a** (43 mg, 0.24 mmol), $[\text{RhCp}^*\text{Cl}_2]_2$ (3.0 mg, 0.0050 mmol) and additive (0.20 mmol) in 1.0 mL solvent at 80 °C, for 12 h under N₂ atmosphere. ^bYields were determined by ¹H NMR integration method using mesitylene as the internal standard. ^cUnder air. DCA = dichloroacetic acid.

Table 2. Effect of AcOH on Rh(III)-Catalyzed Isoquinoline Formation^a

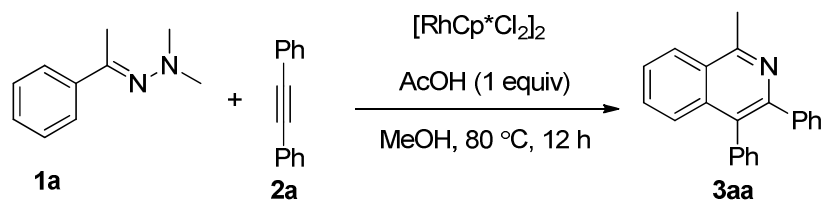


entry	AcOH (equiv)	yield (%) ^b
1	0.50	70
2	1.0	98
3	2.0	96
4	4.0	86
5	5.0	79
6	10.0	70

^aUnless otherwise mentioned, all reactions were carried out using **1a** (33 mg, 0.20 mmol), **2a** (43 mg, 0.24 mmol), $[\text{RhCp}^*\text{Cl}_2]_2$ (3.0 mg, 0.005 mmol), AcOH (indicated amount) in 1.0 mL MeOH at 80 °C, for 12 h under N₂ atmosphere.

^bYields were determined by ¹H NMR integration method using mesitylene as the internal standard.

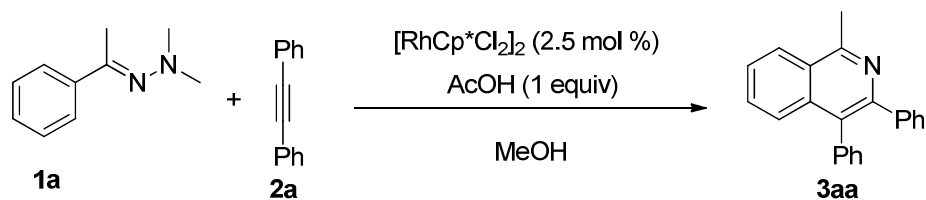
Table 3. Effect of Rh(III)-Catalyst Loading on Isoquinoline Formation^a



entry	$[\text{RhCp}^*\text{Cl}_2]_2$ (mol %)	yield (%)
1	0.5	16
2	1.5	55
3	2.0	81
4	2.0	91 ^c
5	2.5	98

^aUnless otherwise mentioned, all reactions were carried out using **1a** (33 mg, 0.20 mmol), **2a** (43 mg, 0.24 mmol), $[\text{RhCp}^*\text{Cl}_2]_2$ (mentioned amount), AcOH (12 mg, 0.20 mmol) in 1.0 mL MeOH at 80 °C, for 12 h under N₂ atmosphere. ^bYields were determined by ¹H NMR integration method using mesitylene as the internal standard. ^cReaction carried out for 24 h.

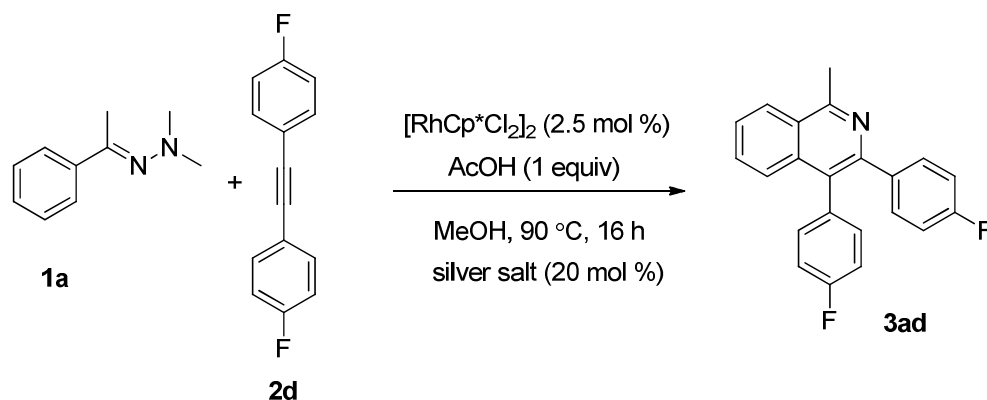
Table 4. Effect of Time and Temperature on Isoquinoline Formation^a



entry	temperature (°C)	time (h)	yield (%)
1	60	8	59
2	60	12	70
3	80	8	80
4	80	12	98
5	90	4	75
6	90	8	98

^aUnless otherwise mentioned, all reactions were carried out using **1a** (33 mg, 0.20 mmol), **2a** (43 mg, 0.24 mmol), $[\text{RhCp}^*\text{Cl}_2]_2$ (3 mg, 0.005 mmol), AcOH (12 mg, 0.20 mmol) in 1.0 mL MeOH at indicated temperature and time under N₂ atmosphere. ^bYields were determined by ¹H NMR integration method using mesitylene as the internal standard.

Table 5. Effect of Silver Salts on Isoquinoline Formation^a

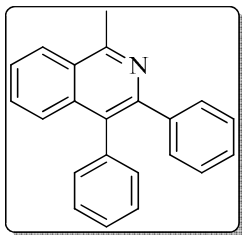


entry	silver salt (20 mol %)	yield (%)
1	-	52
2	AgBF ₄	50
3	Ag₂O	80
4	AgOTf	72
5	AgSbF ₆	64

^aUnless otherwise mentioned, all reactions were carried out using **1a** (33 mg, 0.20 mmol), **2d** (51 mg, 0.24 mmol), $[\text{RhCp}^*\text{Cl}_2]_2$ (3 mg, 0.005 mmol), AcOH (12 mg, 0.20 mmol) and silver salt (0.040 mmol) in 1.0 mL MeOH at 90 °C for 16 h under N₂ atmosphere. ^bYields were determined by ¹H NMR integration method using mesitylene as the internal standard.

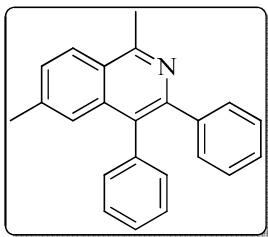
The spectral data and a copy of ^1H and ^{13}C NMR spectra of all compounds are listed below.

1-Methyl-3,4-diphenylisoquinoline (3aa)



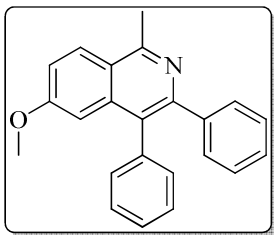
White solid; m.p. 157-158 °C; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.20-8.18 (m, 1H), 7.67-7.65 (m, 1H), 7.60-7.56 (m, 2H), 7.38-7.31 (m, 5H), 7.23-7.15 (m, 5H), 3.08 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 157.6, 149.3, 140.9, 137.5, 135.9, 131.3, 130.2, 129.8, 129.1, 128.1, 127.5, 127.0, 126.8, 126.4, 126.1, 126.1, 125.4, 22.7; HRMS (EI^+): 295.1360 (cal. for $\text{C}_{22}\text{H}_{17}\text{N}$ 295.1361); IR (cm^{-1}): 3054, 1550, 1504, 1434, 1388, 1334, 802.

1,6-Dimethyl-3,4-diphenylisoquinoline (3ba)



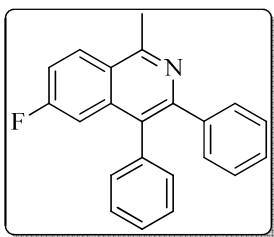
White solid; m.p. 158-160 °C; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.08 (d, $J = 8.8$ Hz, 1H), 7.41-7.40 (m, 2H), 7.36-7.32 (m, 5H), 7.22-7.15 (m, 5H), 3.04 (s, 3 H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 157.3, 149.5, 141.1, 140.1, 137.7, 136.1, 131.4, 130.2, 128.7, 128.6, 128.1, 127.5, 126.9, 126.7, 125.4, 125.0, 124.5, 22.6, 22.1; HRMS (EI^+): 309.1505 (cal. for $\text{C}_{23}\text{H}_{19}\text{N}$ 309.1517); IR (cm^{-1}): 3062, 2915, 1619, 1558, 1496, 1442, 1334, 1072, 1025, 809.

6-Methoxy-1-methyl-3,4-diphenylisoquinoline (3ca)



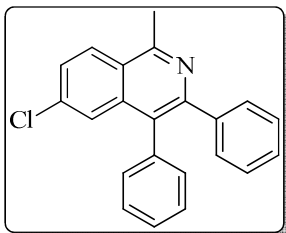
White solid; m.p. 175-177 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.09 (d, *J* = 8.8 Hz, 1H), 7.35-7.28 (m, 5H), 7.22-7.13 (m, 6H), 6.90 (d, *J* = 2.8 Hz, 1H), 3.70 (s, 3H), 3.00 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 160.4, 156.9, 150.0, 141.1, 138.0, 137.8, 131.2, 130.1, 128.5, 128.2, 127.5, 127.4, 127.0, 126.8, 121.8, 118.6, 104.4, 55.1, 22.6; **HRMS** (EI⁺): 325.1460 (cal. for C₂₃H₁₉NO 325.1467); **IR** (cm⁻¹): 3054, 2954, 1612, 1411, 1226, 1025, 817, 686.

6-Fluoro-1-methyl-3,4-diphenylisoquinoline (3da)



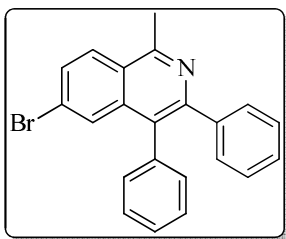
White solid; m.p. 153-154 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.22-8.18 (m, 1H), 7.36-7.29 (m, 6H), 7.25-2.22 (m, 1H), 7.19-7.14 (m, 5H), 3.04 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 164.4, 161.9, 157.5, 150.4, 140.6, 138.1, 137.1, 131.1, 130.1, 128.6, 128.5, 128.3, 127.6, 127.3, 127.1, 123.4, 116.7, 116.5, 109.9, 109.7, 22.8; **HRMS** (EI⁺): 313.1265 (cal. for C₂₂H₁₆FN 313.1267); **IR** (cm⁻¹): 3054, 2923, 1619, 1573, 1504, 1396, 1257, 1180, 979, 802.

6-Chloro-1-methyl-3,4-diphenylisoquinoline (3ea)



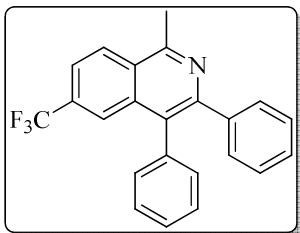
White solid; m.p. 166-167 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.11 (d, *J* = 8.8 Hz, 1H), 7.61 (d, *J* = 1.6 Hz, 1H), 7.50 (dd, *J* = 9.2, 1.6 Hz, 1H), 7.34-7.32 (m, 5H), 7.19-7.15 (m, 5H), 3.03 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 157.6, 150.5, 140.6, 137.1, 136.8, 136.3, 131.2, 130.1, 128.4, 127.6, 127.4, 127.3, 127.1, 125.0, 124.3, 22.7; **HRMS** (EI⁺): 329.0959 (cal. for C₂₂H₁₆ClN 329.0971); **IR** (cm⁻¹): 3062, 2915, 1604, 1565, 1488, 1442, 1388, 1326, 1095, 956, 879, 802.

6-Bromo-1-methyl-3,4-diphenylisoquinoline (3fa)



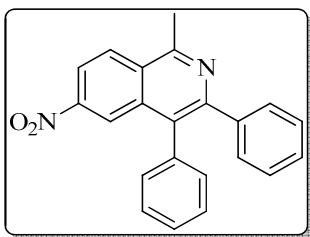
White solid; m.p. 191-193 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.03 (d, *J* = 8.8 Hz, 1H), 7.79 (d, *J* = 2.0 Hz, 1H), 7.64 (dd, *J* = 9.2, 2.0 Hz, 1H), 7.37-7.31 (m, 5H), 7.19-7.15 (m, 5H), 3.03 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 157.7, 150.5, 140.5, 137.3, 136.7, 131.2, 130.1, 129.9, 128.4, 128.3, 128.2, 127.6, 127.4, 127.2, 127.1, 125.0, 124.5, 22.6; **HRMS** (EI⁺): 373.0460 (cal. for C₂₂H₁₆BrN 373.0466); **IR** (cm⁻¹): 3062, 2915, 1596, 1558, 1488, 1442, 1388, 1326, 1072, 879, 809.

1-Methyl-3,4-diphenyl-6-(trifluoromethyl)isoquinoline (3ga)



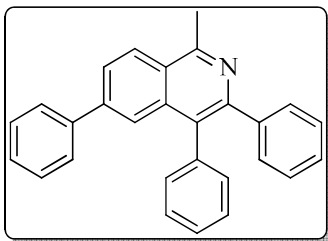
Pale yellow solid; m.p. 110-114 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.30 (d, *J* = 8.8 Hz, 1H), 7.95 (d, *J* = 0.8 Hz, 1H), 7.74 (dd, *J* = 8.8, 1.6 Hz, 1H), 7.39-7.32 (m, 5H), 7.21-7.17 (m, 5H), 3.09 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 157.8, 150.8, 140.3, 136.4, 135.4, 131.3, 131.2, 130.1, 129.6, 128.4, 127.7, 127.6, 127.3, 126.9, 126.8, 125.1, 123.8 (q, *J* = 4.7 Hz), 122.1 (q, *J* = 3.1 Hz), 22.7; **HRMS** (EI⁺): 363.1225 (cal. for C₂₃H₁₆F₃N 363.1235); **IR** (cm⁻¹): 3062, 2923, 1573, 1550, 1504, 1442, 1388, 1311, 1265, 1126, 1079, 956, 902, 802.

1-Methyl-6-nitro-3,4-diphenylisoquinoline (3ha)



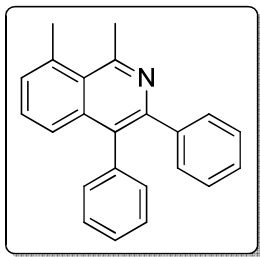
Yellow solid; m.p. 166-168 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.56 (s, 1H), 8.35-8.28 (m, 2H), 7.39-7.34 (m, 5H), 7.20-7.19 (m, 5H), 3.11 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 158.0, 151.5, 148.2, 139.9, 135.9, 135.8, 131.1, 130.4, 130.1, 128.7, 128.0, 127.79, 127.7, 127.5, 122.7, 119.8, 22.9; **HRMS** (EI⁺): 340.1198 (cal. for C₂₂H₁₆N₂O₂ 340.1212); **IR** (cm⁻¹): 2923, 2854, 1535, 1442, 1342, 1025, 910, 802.

1-Methyl-3,4,6-triphenylisoquinoline (3ia)



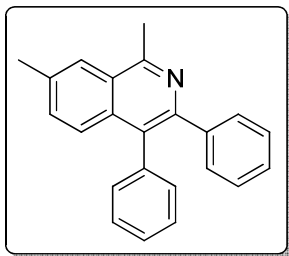
White solid; m.p. 176-178 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.26 (d, *J* = 9.6 Hz, 1H), 7.84-7.82 (m, 2H), 7.55-7.53 (m, 2H), 7.43-7.15 (m, 13H), 3.08 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 157.5, 149.9, 142.5, 140.9, 140.4, 137.4, 136.3, 131.4, 130.2, 129.4, 128.9, 128.2, 127.9, 127.6, 127.5, 127.1, 126.9, 126.2, 126.1, 125.2, 124.0, 22.7; **HRMS** (EI⁺): 371.1674 (cal. for C₂₈H₂₁N 371.1674); **IR** (cm⁻¹): 3054, 2923, 1612, 1565, 1488, 1442, 1396, 1342, 1265, 802.

1,8-Dimethyl-3,4-diphenylisoquinoline (3ma)



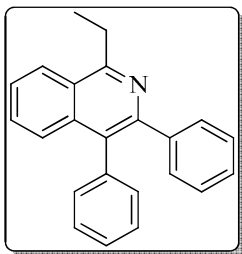
White solid; m.p. 125-127 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 7.47 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.39-7.29 (m, 7H), 7.20-7.13 (m, 5H), 3.23 (s, 3H), 2.99 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 157.6, 148.4, 140.7, 138.2, 138.0, 135.9, 131.4, 130.1, 129.4, 129.1, 128.1, 127.5, 127.0, 126.9, 125.0, 29.8, 25.9; **HRMS** (EI⁺): 309.1514 (cal. for C₂₃H₁₉N 309.1517); **IR** (cm⁻¹): 3062, 2915, 1619, 1550, 1496, 1442.

1,7-Dimethyl-3,4-diphenylisoquinoline (3na)



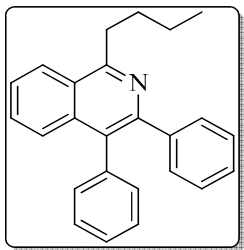
White solid; m.p. 132-134 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 7.95 (s, 1H), 7.56 (d, *J* = 8.4 Hz, 1H), 7.41 (d, *J* = 8.4 Hz, 1H), 7.37-7.30 (m, 5H), 7.23-7.15 (m, 5H), 3.05 (s, 3H), 2.56 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 156.9, 148.5, 141.0, 137.6, 136.3, 134.1, 132.0, 131.3, 130.2, 129.0, 128.0, 127.5, 126.9, 126.7, 126.2, 126.0, 124.4, 22.6, 21.8; **HRMS** (EI⁺): 309.1514 (cal. for C₂₃H₁₉N 309.1517); **IR** (cm⁻¹): 3062, 2915, 1619, 1550, 1496, 1442, 1072, 1025.

1-Ethyl-3,4-diphenylisoquinoline (3ja)



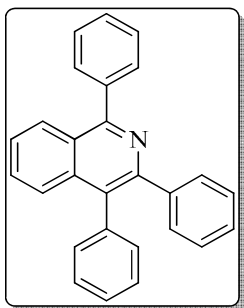
White solid; m.p. 110-112 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.25-8.22 (m, 1H), 7.67-7.65 (m, 1H), 7.57-7.55 (m, 2H), 7.39-7.31 (m, 5H), 7.24-7.15 (m, 5H), 3.44 (q, *J* = 7.2 Hz, 2H), 1.53 (t, *J* = 7.6 Hz, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 162.2, 149.2, 141.0, 137.7, 136.3, 131.3, 130.3, 129.6, 128.9, 128.1, 127.5, 127.0, 126.8, 126.3, 126.3, 125.2, 125.1, 28.7, 13.9; **HRMS** (EI⁺): 309.1515 (cal. for C₂₃H₁₉N 309.1517); **IR** (cm⁻¹): 3062, 2969, 1550, 1504, 1442, 1380, 1072, 1033, 971, 863, 809.

1-Butyl-3,4-diphenylisoquinoline (3ka)



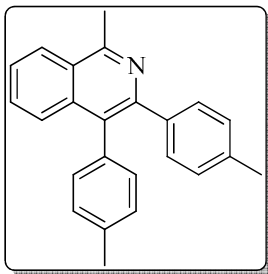
Pale yellow solid; m.p. 66-68 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.25-8.22 (m, 1H), 7.67-7.64 (m, 1H), 7.59-7.54 (m, 2H), 7.38-7.29 (m, 5H), 7.26-7.14 (m, 5H), 3.41 (t, *J* = 8.0 Hz, 2H), 1.97 (sxt, *J* = 7.6, Hz, 2H), 1.57 (sxt, *J* = 8.0 Hz, 2H), 1.03 (t, *J* = 7.6 Hz, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 161.4, 149.2, 141.0, 137.7, 136.3, 131.3, 130.3, 129.6, 128.9, 128.1, 127.5, 127.0, 126.8, 126.3, 125.4, 125.2, 35.4, 32.0, 23.0, 14.0; **HRMS** (EI⁺): 337.1821 (cal. for C₂₅H₂₃N 337.1830); **IR** (cm⁻¹): 3062, 2954, 2861, 1612, 1550, 1504, 1442, 1380, 1334, 1025, 802.

1,3,4-Triphenylisoquinoline (3la)



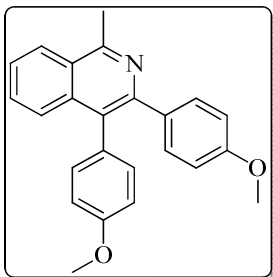
White solid; m.p. 181-183 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.18 (d, *J* = 8.4 Hz, 1H), 7.82 (d, *J* = 6.4 Hz, 2H), 7.71 (d, *J* = 8.4 Hz, 1H), 7.60-7.47 (m, 5H), 7.43-7.35 (m, 5H), 7.30-7.28 (m, 2H), 7.20-7.15 (m, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 159.7, 149.6, 140.8, 139.8, 137.5, 136.9, 131.3, 130.4, 130.2, 129.9, 129.7, 128.5, 128.2, 127.5, 127.4, 127.2, 126.9, 126.5, 126.0, 125.4; **HRMS** (EI⁺): 357.1510 (cal. for C₂₇H₁₉N 357.1517); **IR** (cm⁻¹): 3054, 2923, 1542, 1496, 1442, 1388, 1334, 1265, 979, 802.

1-Methyl-3,4-di-p-tolyloisoquinoline (3ab)



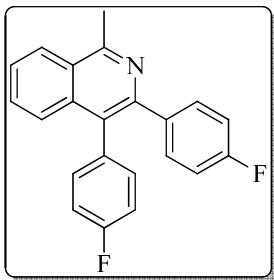
White solid; m.p. 130-132 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.18-8.15 (m, 1H), 7.65-7.62 (m, 1H), 7.57-7.54 (m, 2H), 7.25 (dd, *J* = 6.4, 1.6 Hz, 2H), 7.14 (d, *J* = 4.4 Hz, 2H), 7.10-7.07 (m, 2H), 6.99 (d, *J* = 8.4 Hz, 2H), 3.06 (s, 3H), 2.37 (s, 3H), 2.26 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 157.4, 136.7, 136.6, 136.3, 131.1, 130.1, 129.9, 128.9, 128.3, 126.4, 126.3, 126.0, 125.5, 22.6, 21.2, 21.1; **HRMS** (EI⁺): 323.1675 (cal. for C₂₄H₂₁N 323.1674); **IR** (cm⁻¹): 3023, 2915, 1612, 1565, 1511, 1434, 1388, 1025, 786.

3,4-Bis(4-methoxyphenyl)-1-methyloisoquinoline (3ac)



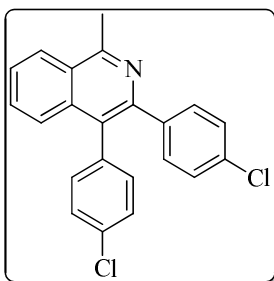
Pale yellow solid; m.p. 115-116 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.16-8.13 (m, 1H), 7.66-7.64 (m, 1H), 7.55-7.53 (m, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.12 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 8.4 Hz, 2H), 6.73 (d, *J* = 8.8 Hz, 2H), 3.83 (s, 3H), 3.75 (s, 3H), 3.03 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 158.5, 158.5, 157.3, 149.1, 136.4, 133.6, 132.4, 131.4, 129.9, 129.7, 128.2, 126.1, 126.1, 125.9, 125.4, 113.7, 113.1, 55.2, 55.1, 22.7; **HRMS** (EI⁺): 355.1568 (cal. for C₂₄H₂₁NO₂ 355.1572); **IR** (cm⁻¹): 2954, 2838, 1604, 1511, 1249, 1172, 1033, 802.

3,4-Bis(4-fluorophenyl)-1-methylisoquinoline (3ad)



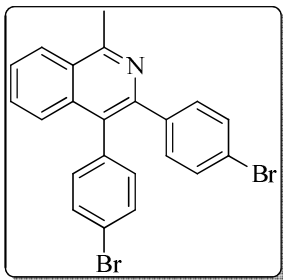
Yellow solid; m.p. 140-141 °C; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.20-8.17 (m, 1H), 7.60-7.57 (m, 3H), 7.32-7.28 (m, 2H), 7.17-7.14 (m, 2H), 7.07-7.03 (m, 2H), 6.91-6.87 (m, 2H), 3.05 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 163.2 (d, $J = 7.7$ Hz), 160.7 (d, $J = 7.7$ Hz), 158.0, 148.6, 136.8 (d, $J = 3.1$ Hz), 135.9, 133.2 (d, $J = 3.1$ Hz), 132.9 (d, $J = 7.8$ Hz), 131.9 (d, $J = 8.6$ Hz), 130.1, 128.0, 126.7, 126.1, 125.9, 125.6, 115.4 (d, $J = 21.7$ Hz), 114.6 (d, $J = 20.9$ Hz), 22.7; HRMS (EI^+): 331.1171 (cal. for $\text{C}_{22}\text{H}_{15}\text{F}_2\text{N}$ 331.1173); IR (cm^{-1}): 3070, 2923, 1604, 1511, 1434, 1388, 1226, 1157, 825.

3,4-Bis(4-chlorophenyl)-1-methylisoquinoline (3ae)



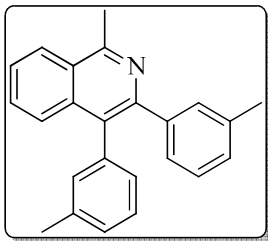
Pale yellow solid; m.p. 153-154 °C; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.20-8.17 (m, 1H), 7.61-7.56 (m, 3H), 7.35-7.26 (m, 4H), 7.19-7.12 (m, 4H), 3.04 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 158.3, 148.1, 139.1, 135.7, 135.7, 133.4, 133.2, 132.6, 131.5, 130.2, 128.7, 128.0, 127.9, 126.9, 126.2, 125.8, 125.6, 22.7; HRMS (EI^+): 363.0574 (cal. for $\text{C}_{22}\text{H}_{15}\text{Cl}_2\text{N}$ 363.0582); IR (cm^{-1}): 3070, 2923, 1573, 1496, 1434, 1388, 1095, 1018, 809.

3,4-Bis(4-bromophenyl)-1-methylisoquinoline (3af)



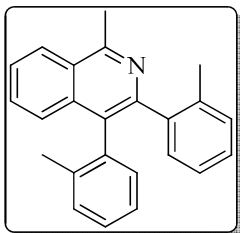
Yellow solid; m.p. 188-189 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.19-8.17 (m, 1H), 7.61-7.59 (m, 3H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 2H), 7.21 (d, *J* = 8.4 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 3.04 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 158.3, 148.1, 139.5, 136.2, 135.6, 132.9, 131.8, 131.6, 130.9, 130.3, 127.9, 126.9, 126.2, 125.8, 125.6, 121.6, 121.6, 22.7; **HRMS** (EI⁺): 450.9575 (cal. for C₂₂H₁₅Br₂N 450.9571); **IR** (cm⁻¹): 3070, 2915, 1565, 1496, 1388, 1072, 1010, 809.

1-Methyl-3,4-di-*m*-tolylisoquinoline (3ag)



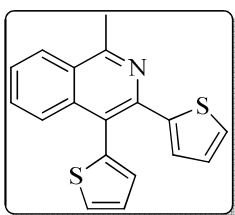
White solid; m.p. 124-126 °C; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 8.18-8.16 (m, 1H), 7.65-7.63 (m, 1H), 7.58-7.55 (m, 2H), 7.27 (d, *J* = 0.8 Hz, 1H), 7.22-7.18 (m, 1H), 7.11-6.94 (m, 6H), 3.05 (s, 3H), 2.30 (s, 3H), 2.24 (s, 3H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 157.4, 149.3, 140.8, 137.5, 137.5, 137.0, 136.1, 131.9, 130.9, 129.7, 129.2, 128.4, 127.9, 127.7, 127.6, 127.3, 127.2, 126.3, 126.3, 126.0, 125.4, 22.6, 21.4; **HRMS** (EI⁺): 323.1672 (cal. for C₂₄H₂₁N 323.1674); **IR** (cm⁻¹): 3039, 2915, 1612, 1573, 1504, 1434, 1388, 802.

1-Methyl-3,4-di-o-tolylisoquinoline (3ah)



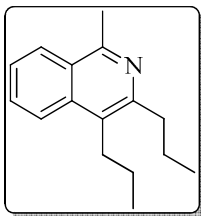
White solid; m.p. 154-156 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm): 8.20 (d, $J = 7.6$ Hz, 1H), 7.61-7.54 (m, 2H), 7.37-7.34 (m, 1H), 7.14-6.89 (m, 8H), 3.04 (s, 3H), 2.20 (s, 3H), 1.95 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm): 157.1, 150.5, 140.2, 136.9, 136.5, 136.2, 135.8, 130.9, 130.1, 129.9, 129.7, 129.5, 129.3, 127.4, 127.2, 126.5, 126.1, 126.0, 125.6, 125.2, 124.7, 22.4, 20.2, 20.0; **HRMS** (EI^+): 323.1665 (cal. for $\text{C}_{24}\text{H}_{21}\text{N}$ 323.1674); **IR** (cm^{-1}): 3062, 2923, 1558, 1504, 1442, 1388, 1334, 802.

1-Methyl-3,4-di(thiophen-2-yl)isoquinoline (3ai)



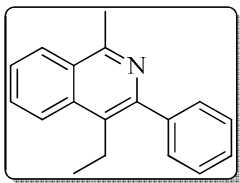
Pale yellow solid; m.p. 75-77 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm): 8.09 (d, $J = 8.0$ Hz, 1 H), 7.58-7.52 (m, 4H), 7.28-7.23 (m, 2H), 7.07-7.06 (m, 1H), 6.89-6.87 (m, 1H), 6.72 (d, $J = 3.6$ Hz, 1H), 3.02 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm): 158.8, 144.2, 137.9, 137.7, 130.4, 129.1, 127.8, 127.6, 127.6, 127.4, 127.3, 126.5, 126.0, 125.8, 125.4, 22.6; **HRMS** (EI^+): 307.0485 (cal. for $\text{C}_{18}\text{H}_{13}\text{NS}_2$ 307.0489); **IR** (cm^{-1}): 3070, 2923, 1558, 1434, 802.

1-Methyl-3,4-dipropylisoquinoline (3aj)



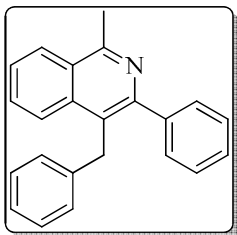
Colorless oily liquid; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.05 (d, $J = 8.8$ Hz, 1H), 7.94 (d, $J = 8.8$ Hz, 1H), 7.62 (t, $J = 7.6$ Hz, 1H), 7.47 (t, $J = 7.6$ Hz, 1H), 2.96 (t, $J = 8.4$ Hz, 2H), 2.90-2.86 (m, 5H), 1.74 (qt, $J = 7.6, 7.6$ Hz, 2H), 1.65 (qt, $J = 7.6, 7.6$ Hz, 2H), 1.07 (t, $J = 7.2$ Hz, 3H), 1.02 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 155.5, 151.6, 135.3, 129.3, 126.1, 126.0, 125.9, 125.2, 123.5, 37.4, 29.7, 24.1, 23.7, 22.3, 14.5, 14.3; HRMS (EI^+): 227.1667 (cal. for $\text{C}_{16}\text{H}_{21}\text{N}$ 227.1674); IR (cm^{-1}): 3070, 2962, 2869, 1565, 1457, 1388, 1334, 1087, 1025, 802.

4-Ethyl-1-methyl-3-phenylisoquinoline (3ak)



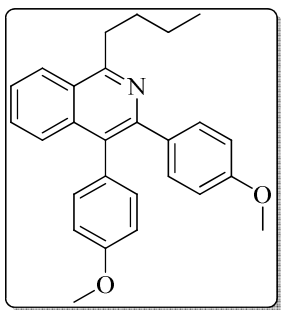
Brown solid; m.p. 124-125 °C; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.16 (d, $J = 8.8$ Hz, 1H), 8.06 (d, $J = 8.8$ Hz, 1H), 7.72 (t, $J = 8.0$ Hz, 1H), 7.58 (t, $J = 7.6$ Hz, 1H), 7.50-7.37 (m, 5H), 3.00-2.95 (m, 5H), 1.24 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 155.8, 150.7, 141.7, 135.1, 129.8, 129.2, 128.6, 128.1, 127.4, 126.7, 126.3, 126.2, 124.1, 22.4, 21.6, 15.7; HRMS (EI^+): 247.1350 (cal. for $\text{C}_{18}\text{H}_{17}\text{N}$ 247.1361); IR (cm^{-1}): 3062, 2969, 1565, 1442, 1388, 1025, 802.

4-Benzyl-1-methyl-3-phenylisoquinoline (3al)



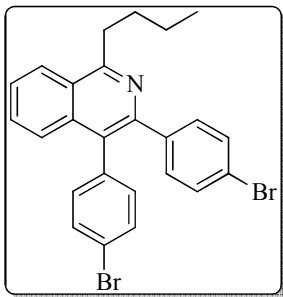
Brown solid; m.p. 146-148 °C; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.18 (d, $J = 7.6$ Hz, 1H), 7.82 (d, $J = 8.0$ Hz, 1H), 7.59-7.54 (m, 2H), 7.50-7.48 (m, 2H), 7.38-7.31 (m, 3H), 7.22-7.12 (m, 3H), 7.02 (d, $J = 6.8$ Hz, 2H), 4.41 (s, 2H), 3.04 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 157.2, 140.7, 136.2, 130.9, 129.3, 128.5, 128.3, 128.2, 128.0, 128.0, 127.8, 126.8, 126.6, 126.3, 126.0, 125.2, 34.6, 21.8; HRMS (EI^+): 309.1517 (cal. for $\text{C}_{23}\text{H}_{19}\text{N}$ 309.1517); IR (cm^{-1}): 3062, 2923, 1720, 1565, 1496, 1442, 809.

1-Butyl-3,4-bis(4-methoxyphenyl)isoquinoline (3kc)



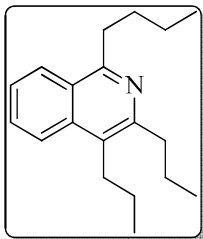
Brown solid; m.p. 56-58 °C; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.20-8.18 (m, 1H), 7.66-7.64 (m, 1H), 7.54-7.51 (m, 2H), 7.32 (d, $J = 8.8$ Hz, 2H), 7.12 (d, $J = 8.8$ Hz, 2H), 6.90 (d, $J = 8.0$ Hz, 2H), 6.73 (d, $J = 8.4$ Hz, 2H), 3.83 (s, 3H), 3.75 (s, 3H), 3.37 (t, $J = 8.4$ Hz, 2H), 1.93 (sxt, $J = 8.0, 7.6$ Hz, 2H), 1.55 (qt, $J = 8.0, 7.2$ Hz, 2H), 1.01 (t, $J = 7.6$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 161.1, 158.5, 158.4, 148.9, 136.7, 133.7, 132.3, 131.5, 130.0, 129.5, 127.9, 126.2, 126.0, 125.3, 125.2, 113.7, 113.0, 55.2, 55.1, 35.3, 31.9, 23.0, 14.0; HRMS (EI^+): 397.2040 (cal. for $\text{C}_{27}\text{H}_{27}\text{NO}_2$ 397.2042); IR (cm^{-1}): 2954, 1604, 1511, 1380, 1249, 1172, 1033, 786.

3,4-Bis(4-bromophenyl)-1-butylisoquinoline (3kf)



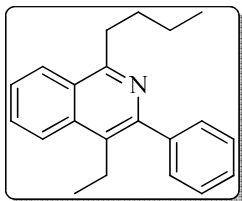
White solid; m.p. 80-82 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm): 8.23-8.20 (m, 1H), 7.58-7.57 (m, 3H), 7.49 (d, $J = 8.0$ Hz, 2H), 7.33 (d, $J = 8.4$ Hz, 2H), 7.21 (d, $J = 8.4$ Hz, 2H), 7.08 (d, $J = 8.0$ Hz, 2H), 3.36 (t, $J = 8.0$ Hz, 2H), 1.90 (tt, $J = 8.0, 7.6$ Hz, 2H), 1.53 (qt, $J = 8.0, 7.2$ Hz, 2H), 1.00 (t, $J = 7.6$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm): 162.1, 147.9, 139.7, 136.4, 135.9, 132.9, 131.9, 131.6, 130.9, 130.0, 127.6, 126.7, 125.9, 125.5, 125.3, 121.5, 121.5, 35.4, 31.9, 23.0, 14.0; **HRMS** (EI^+): 193.0002 (cal. for $\text{C}_{25}\text{H}_{21}\text{Br}_2\text{N}$ 493.0041); **IR** (cm^{-1}): 3070, 2954, 1565, 1488, 1380, 1072, 1010, 809.

1-Butyl-3,4-dipropylisoquinoline (3kj)



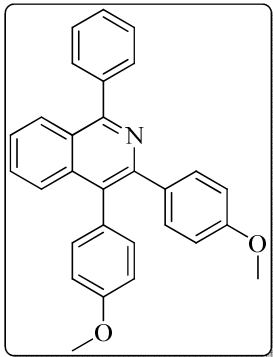
Yellow oily liquid; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm): 8.09 (d, $J = 8.0$ Hz, 1H), 7.94 (d, $J = 8.4$ Hz, 1H), 7.61 (t, $J = 7.6$ Hz, 1H), 7.45 (t, $J = 8.0$ Hz, 1H), 3.22 (t, $J = 8.0$ Hz, 2H), 2.97-2.88 (m, 4H), 1.83-1.73 (m, 4H), 1.66 (qt, $J = 8.0, 7.6$ Hz, 2H), 1.47 (qt, $J = 7.6, 7.2$ Hz, 2H), 1.07 (t, $J = 7.6$ Hz, 3H), 1.02 (t, $J = 7.6$ Hz, 3H), 0.95 (t, $J = 7.6$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm): 159.4, 151.6, 135.7, 129.1, 125.9, 125.8, 125.2, 125.0, 123.6, 37.3, 35.3, 32.3, 29.8, 24.1, 23.7, 22.9, 14.6, 14.2, 14.0; **HRMS** (EI^+): 269.2146 (cal. for $\text{C}_{19}\text{H}_{27}\text{N}$ 269.2143); **IR** (cm^{-1}): 2954, 2869, 1565, 1457, 1380, 802.

1-Butyl-4-ethyl-3-phenylisoquinoline (3kk)



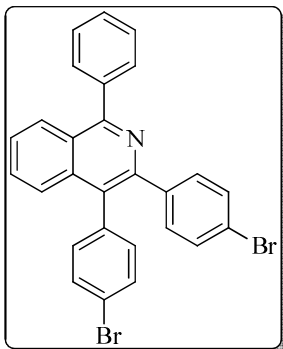
Yellow oily liquid; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm): 8.20 (d, $J = 8.8$ Hz, 1H), 8.07 (d, $J = 8.4$ Hz, 1H), 7.72-7.69 (m, 1H), 7.59-7.55 (m, 1H), 7.52-7.36 (m, 5H), 3.30 (t, $J = 8.4$ Hz, 2H), 2.98 (q, $J = 7.6$ Hz, 2H), 1.83 (tt, $J = 7.6, 7.6$ Hz, 2H), 1.50 (qt, $J = 7.6, 7.6$ Hz, 2H), 1.26 (t, $J = 7.6$ Hz, 3H), 0.96 (t, $J = 7.6$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm): 159.7, 150.6, 141.8, 135.4, 129.6, 129.2, 128.3, 128.0, 127.3, 126.0, 126.0, 125.9, 124.2, 35.4, 32.4, 23.0, 21.6, 15.6, 14.0; **HRMS** (EI^+): 289.1832 (cal. for $\text{C}_{21}\text{H}_{23}\text{N}$ 289.1830); **IR** (cm^{-1}): 3070, 2931, 2869, 1612, 1565, 1504, 1457, 1380, 1334, 1025, 802.

3,4-Bis(4-methoxyphenyl)-1-phenylisoquinoline (3lc)



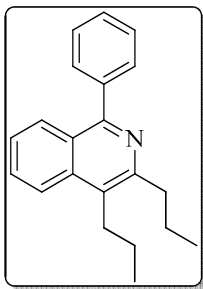
Yellow solid; m.p. 162-163 $^{\circ}\text{C}$; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm): 8.13 (d, $J = 8.0$ Hz, 1H), 7.79 (d, $J = 7.2$ Hz, 2H), 7.72 (d, $J = 8.4$ Hz, 1H), 7.57-7.44 (m, 5H), 7.37 (d, $J = 8.8$ Hz, 2H), 7.20 (d, $J = 8.4$ Hz, 2H), 6.94 (d, $J = 8.4$ Hz, 2H), 6.72 (d, $J = 8.8$ Hz, 2H), 3.85 (s, 3H), 3.74 (s, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm): 159.4, 158.7, 158.5, 149.3, 139.9, 137.3, 133.5, 132.3, 131.6, 130.2, 129.9, 129.7, 128.8, 128.4, 128.2, 127.4, 126.2, 125.9, 125.2, 113.9, 113.0, 110.0, 55.2, 55.1; **HRMS** (EI^+): 417.1723 (cal. for $\text{C}_{29}\text{H}_{23}\text{NO}_2$ 417.1729); **IR** (cm^{-1}): 3062, 2938, 2838, 1604, 1511, 1388, 1249, 1180, 1033, 840.

3,4-bis(4-bromophenyl)-1-phenylisoquinoline (3lf)



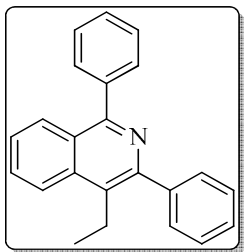
White solid; m.p. 196-198 °C; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm): 8.14 (d, $J = 8.0$ Hz, 1H), 7.74 (d, $J = 6.4$ Hz, 2H), 7.63-7.51 (m, 8H), 7.31-7.21 (m, 4H), 7.13 (d, $J = 5.6$ Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm): 160.3, 139.4, 136.7, 136.2, 132.9, 132.0, 131.8, 130.9, 130.4, 130.1, 128.7, 128.6, 128.3, 127.7, 127.0, 125.6, 125.5, 121.8, 121.7; **HRMS** (EI^+): 512.9725 (cal. for $\text{C}_{27}\text{H}_{17}\text{Br}_2\text{N}$ 512.9728); **IR** (cm^{-1}): 1488, 1380, 1265, 1064, 979, 809.

1-phenyl-3,4-dipropylisoquinoline (3lj)



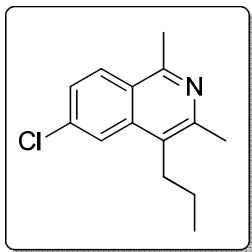
Yellow oily liquid; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm): 8.02 (dd, $J = 8.8, 7.6$ Hz, 2H), 7.67-7.63 (m, 3H), 7.52-7.39 (m, 4H), 3.08-3.00 (m, 4H), 1.85 (qt, $J = 8.0, 7.6$ Hz, 2H), 1.74 (qt, $J = 8.0, 7.6$ Hz, 2H), 1.13 (t, $J = 7.6$ Hz, 3H), 1.05 (t, $J = 7.6$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm): 158.0, 152.1, 140.0, 136.0, 132.3, 129.9, 129.4, 128.2, 128.1, 128.0, 127.1, 125.2, 125.2, 123.2, 37.3, 29.9, 24.1, 23.6, 14.6, 14.3; **HRMS** (EI^+): 289.1831 (cal. for $\text{C}_{21}\text{H}_{23}\text{N}$ 289.1830); **IR** (cm^{-1}): 3062, 2962, 2869, 1658, 1612, 1550, 1450, 1388, 1334, 1272, 1164, 1079, 917, 802.

4-Ethyl-1,3-diphenylisoquinoline (3lk)



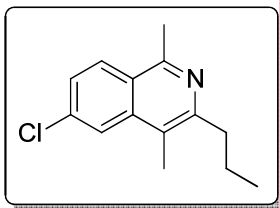
Yellow oily liquid; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 8.13 (t, $J = 8.4$ Hz, 2H), 7.75-7.68 (m, 3H), 7.58-7.36 (m, 9H), 3.08 (q, $J = 7.2$ Hz, 2H), 1.33 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 158.2, 151.0, 141.7, 139.8, 135.9, 130.1, 129.8, 129.4, 128.3, 128.2, 128.2, 128.0, 127.4, 126.2, 125.9, 123.8, 21.8, 15.6; HRMS (EI^+): 309.1521 (cal. for $\text{C}_{23}\text{H}_{19}\text{N}$ 309.1517); IR (cm^{-1}): 3054, 2969, 2877, 1550, 1496, 1442, 1388, 1334, 802.

6-Chloro-1,3-dimethyl-4-propylisoquinoline (3em)



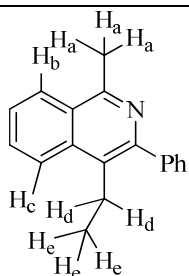
Colorless oily liquid; ^1H NMR (400 MHz, CDCl_3) δ (ppm): 7.97 (d, $J = 8.8$ Hz, 1 H), 7.86 (s, 1 H), 7.39 (dd, $J = 2.0$ Hz, $J = 9.2$ Hz, 1 H), 2.88 (t, $J = 8.0$ Hz, 2 H), 2.84 (s, 1 H), 1.61 (m, 2 H), 1.04 (t, $J = 7.2$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm): 155.3, 149.0, 136.2, 135.8, 127.8, 126.0, 125.9, 124.2, 122.4, 30.0, 23.2, 22.3, 22.2, 14.4; HRMS (EI^+): 233.0971 (cal. for $\text{C}_{14}\text{H}_{16}\text{ClN}$ 233.0967); IR (cm^{-1}): 3062, 2962, 1565, 1380.

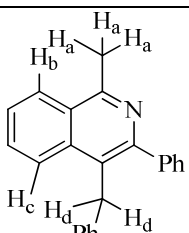
6-Chloro-1,4-dimethyl-3-propylisoquinoline (3em')

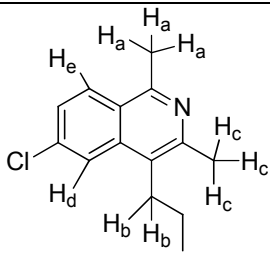


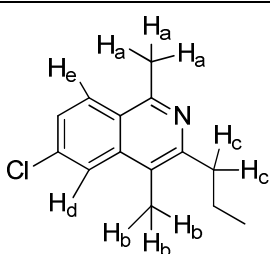
Colorless oily liquid; **¹H NMR** (400 MHz, CDCl₃) δ (ppm): 7.98 (d, *J* = 8.8 Hz, 1 H), 7.90, (s, 1 H), 7.42 (dd, *J* = 2.0 Hz, *J* = 8.8 Hz, 1 H), 2.90 (t, *J* = 7.6 Hz, 2 H), 2.86 (s, 3 H), 2.50 (s, 3 H), 1.71 (m, 2 H), 0.99 (t, *J* = 7.2 Hz, 3 H); **¹³C NMR** (100 MHz, CDCl₃) δ (ppm): 155.4, 153.0, 137.0, 135.8, 127.7, 126.1, 123.9, 122.7, 120.7, 38.0, 23.2, 22.2, 14.1, 13.6; **HRMS** (EI⁺):233.0971 (cal. for C₁₄H₁₆ClN 233.0967); **IR** (cm⁻¹): 3058, 2962, 2869,1565, 1025.

NOE Experiments of compounds 3ak and 3al

compound 3ak	irradiate	observed enhancement (%)
	Ha	Hb (0.52)
	Hb	Ha (2.60)
	Hc	Hd (3.86), He (1.43)
	Hd	Hc (1.17), He (0.90)
	He	Hc (0.45), Hd (0.78)

compound 3al	irradiate	observed enhancement (%)
	Ha	Hb (1.63)
	Hb	Ha (3.34)
	Hc	Hd (4.11)
	Hd	Hc (3.22)

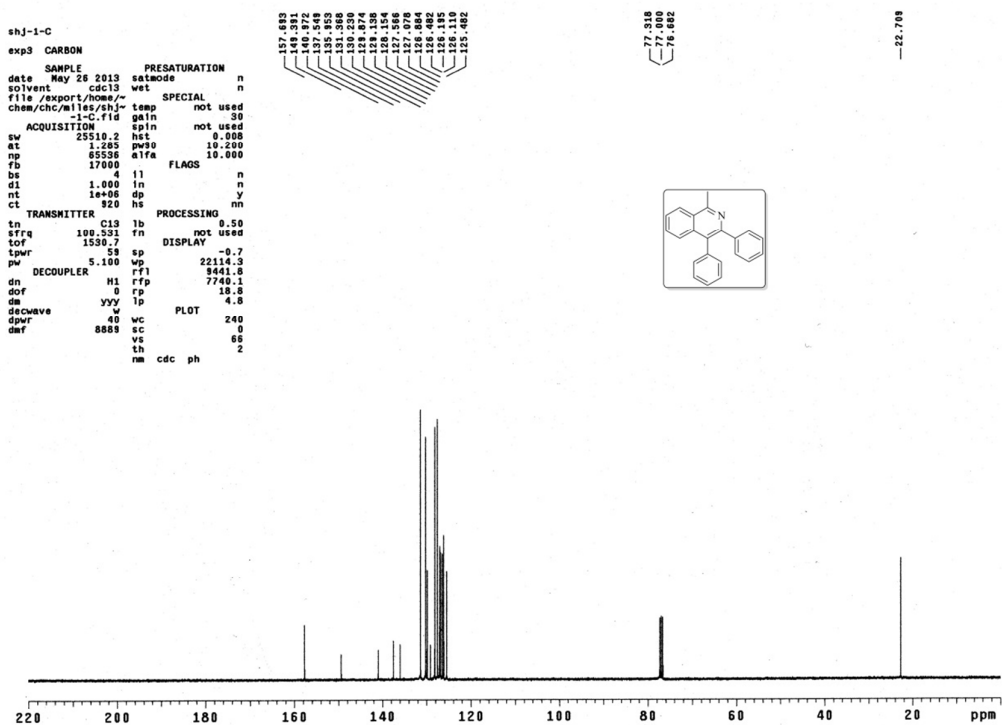
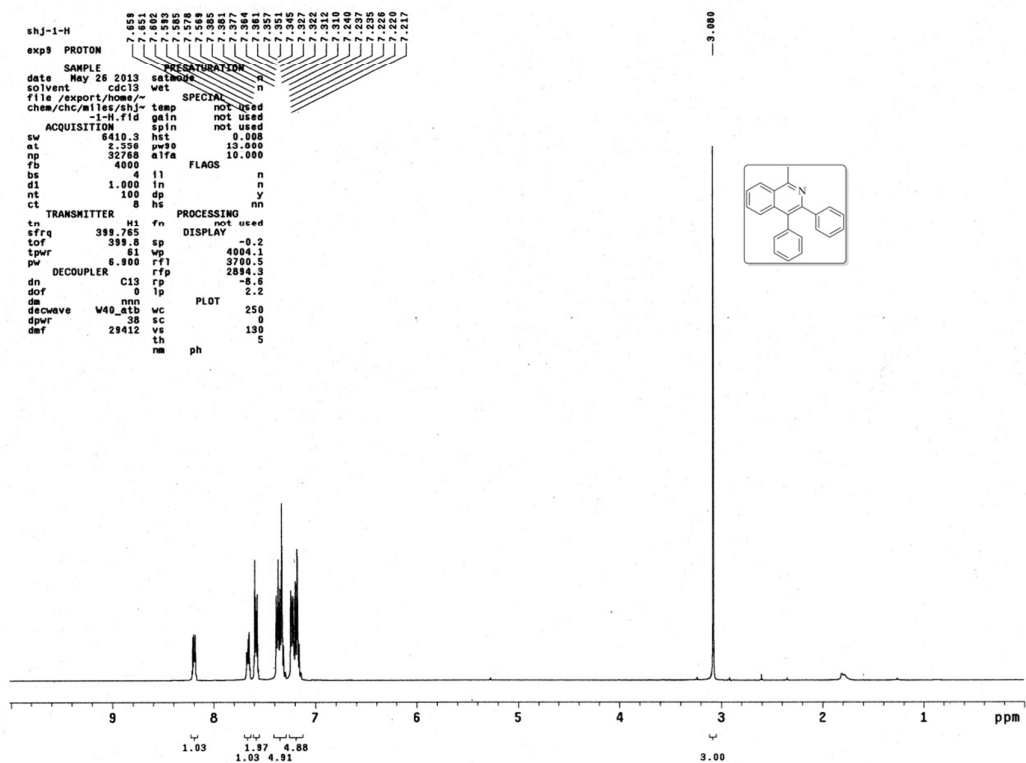
compound 3em	irradiate	observed enhancement (%)
	Ha	He (0.88)
	Hb	Hd (1.58)
	Hc	Hb (0.94)
	Hd	Hb (4.12)

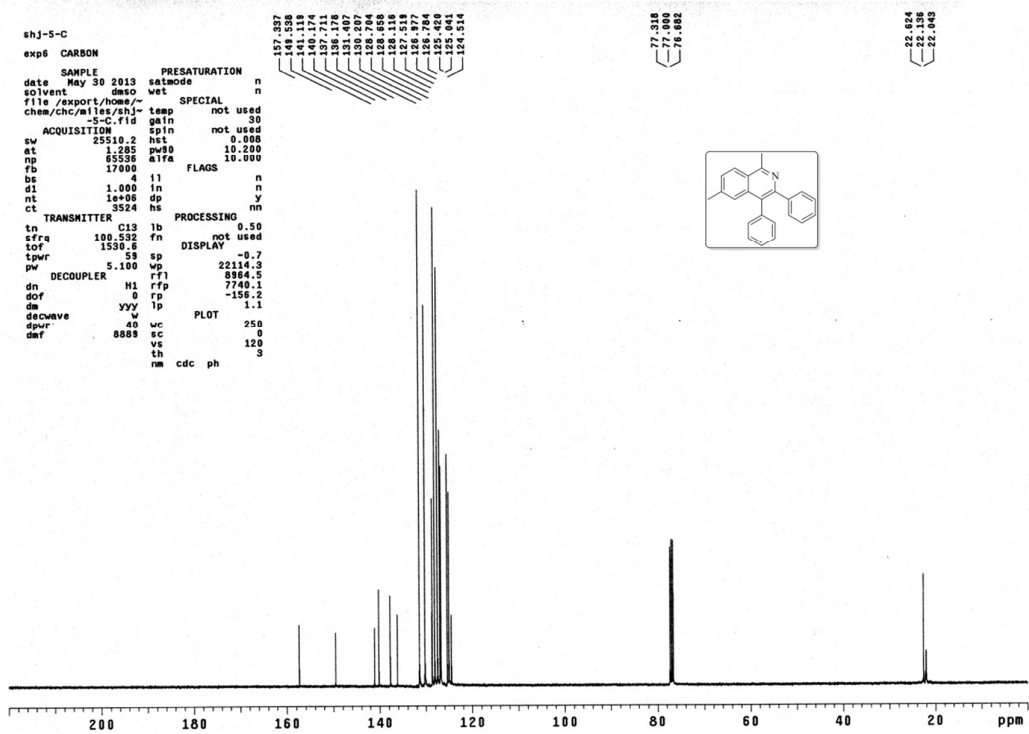
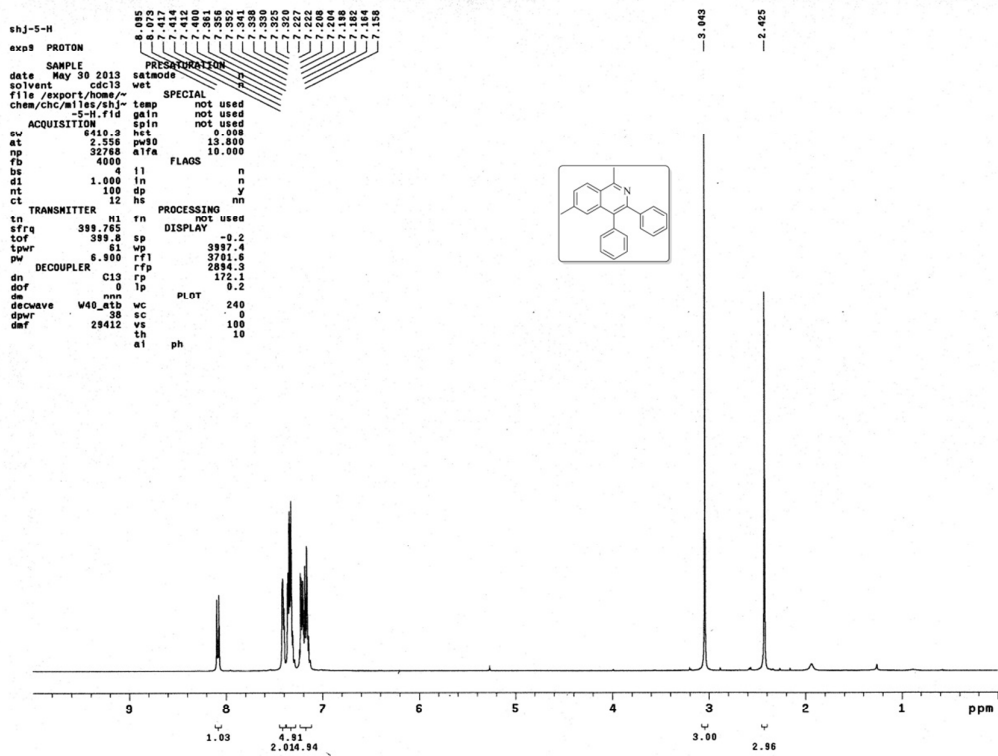
compound 3em'	irradiate	observed enhancement (%)
	Ha	He (0.84)
	Hb	Hd (1.04)
	Hc	Hb (1.02)
	Hd	Hb (3.33)

References

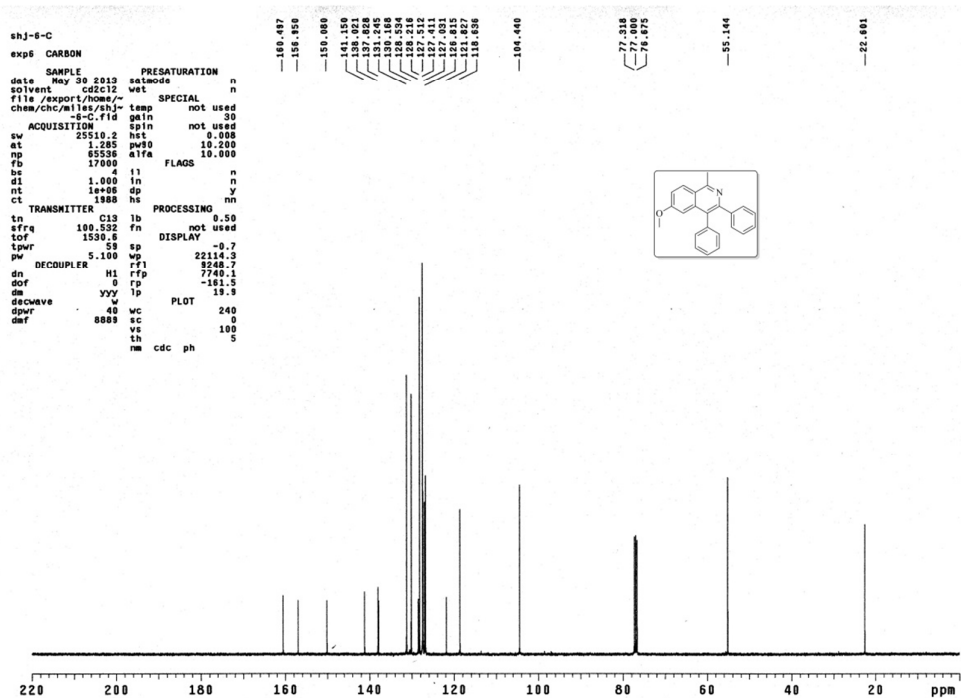
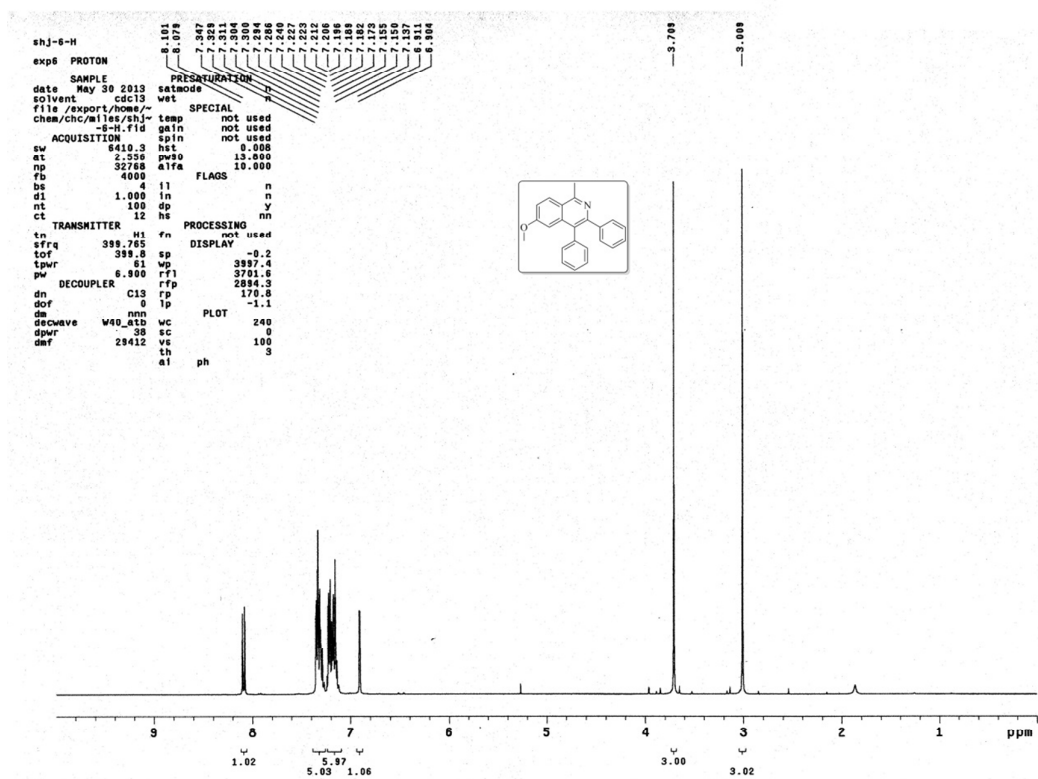
1. Perrin, D. D.; Armarego. W. L. F. *In Purification of Laboratory Chemicals*, 3rd ed.; Pergamon Press: New York, **1988**.
2. Newkome, G. R.; Fishel, D. L. *J. Org. Chem.* **1966**, *31*, 677.

^1H and ^{13}C NMR spectra of compound **3aa**

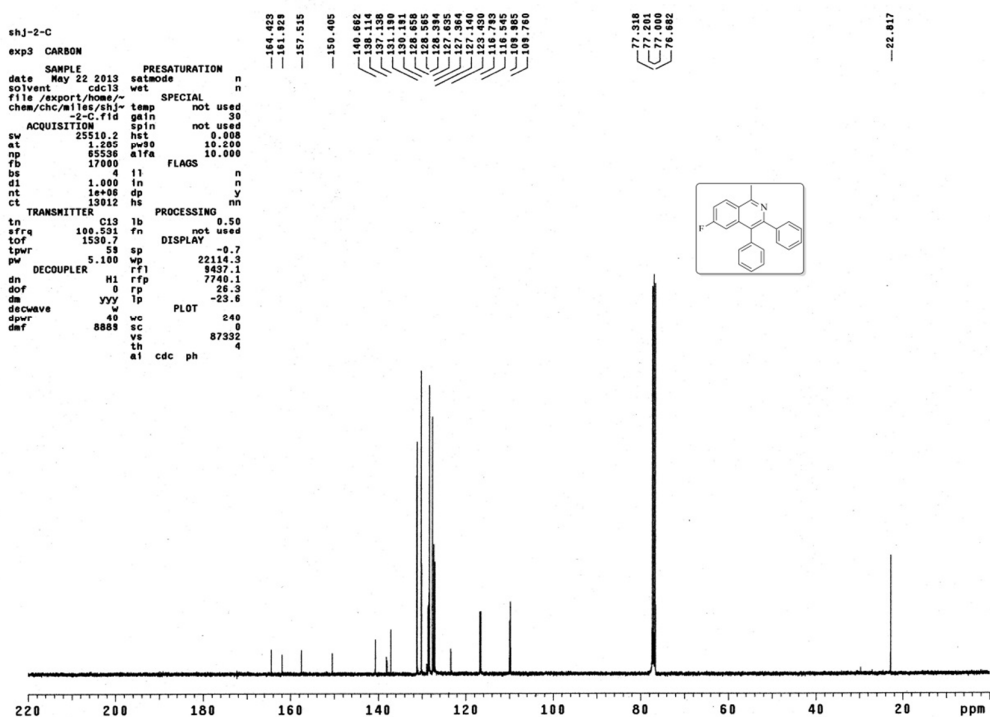
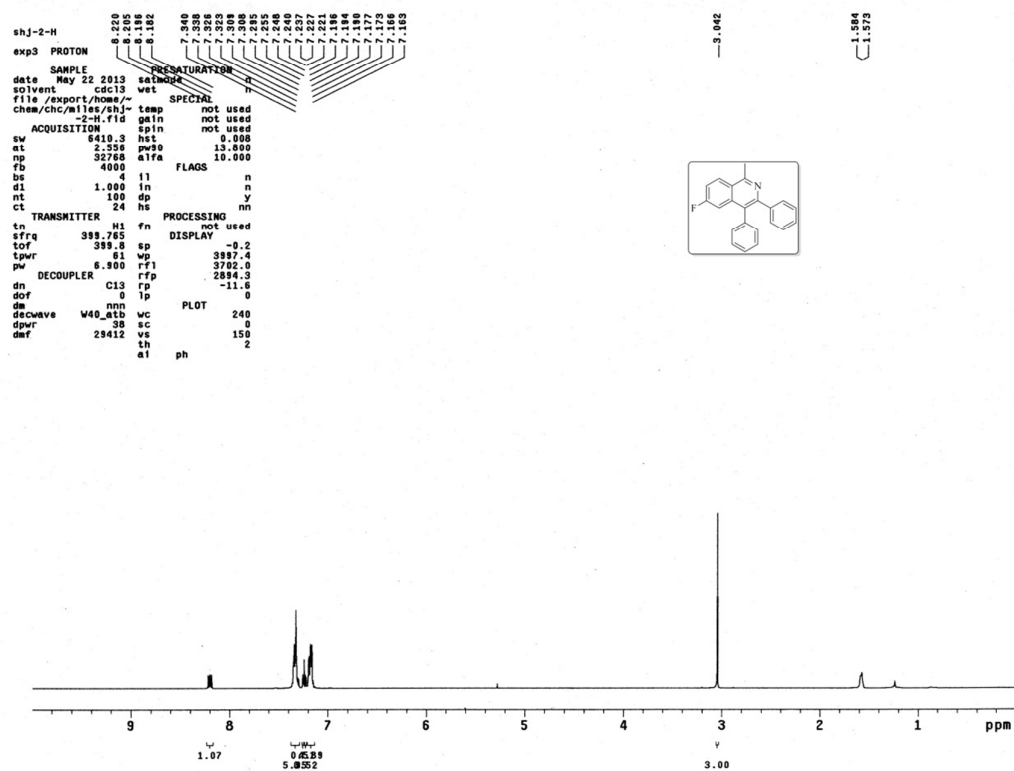


^1H and ^{13}C NMR spectra of compound **3ba**

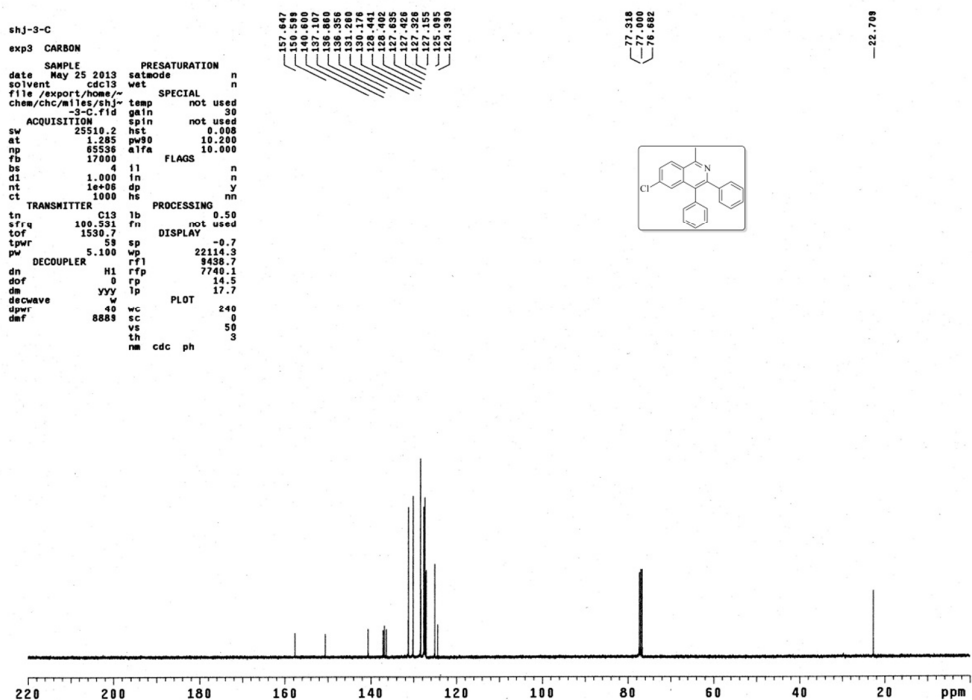
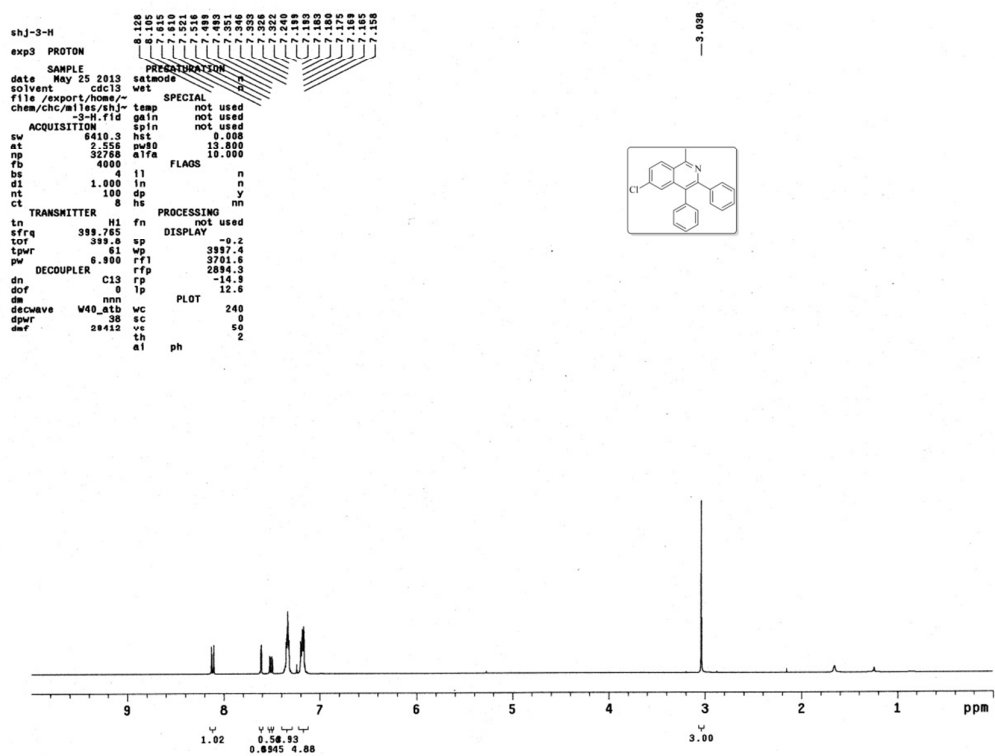
^1H and ^{13}C NMR spectra of compound **3ca**



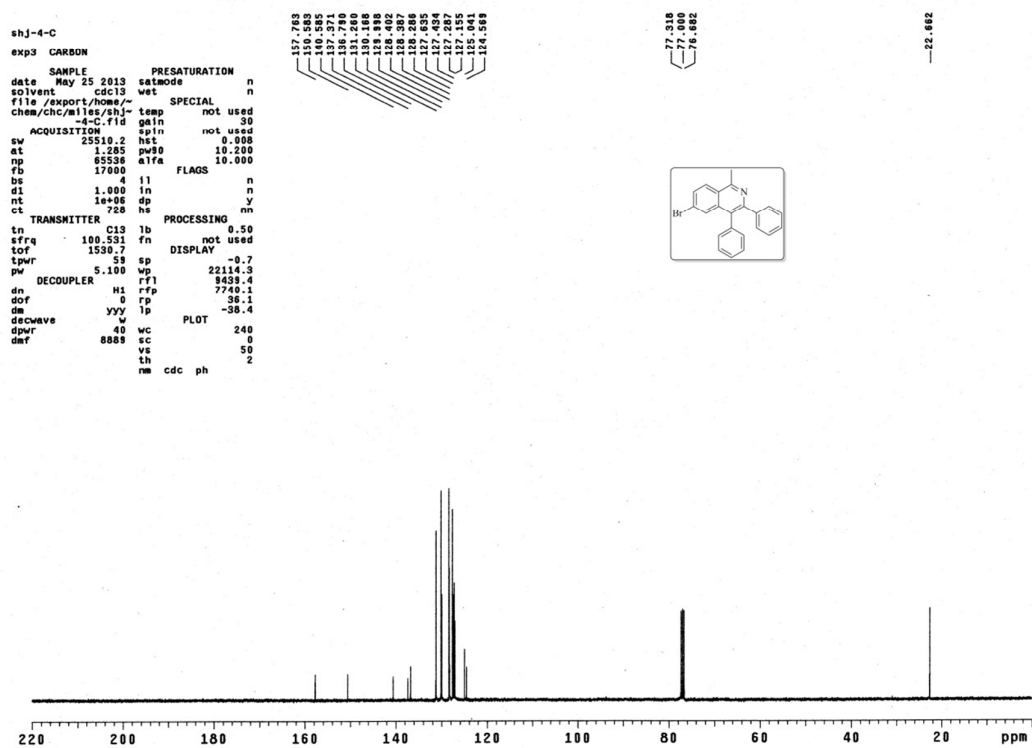
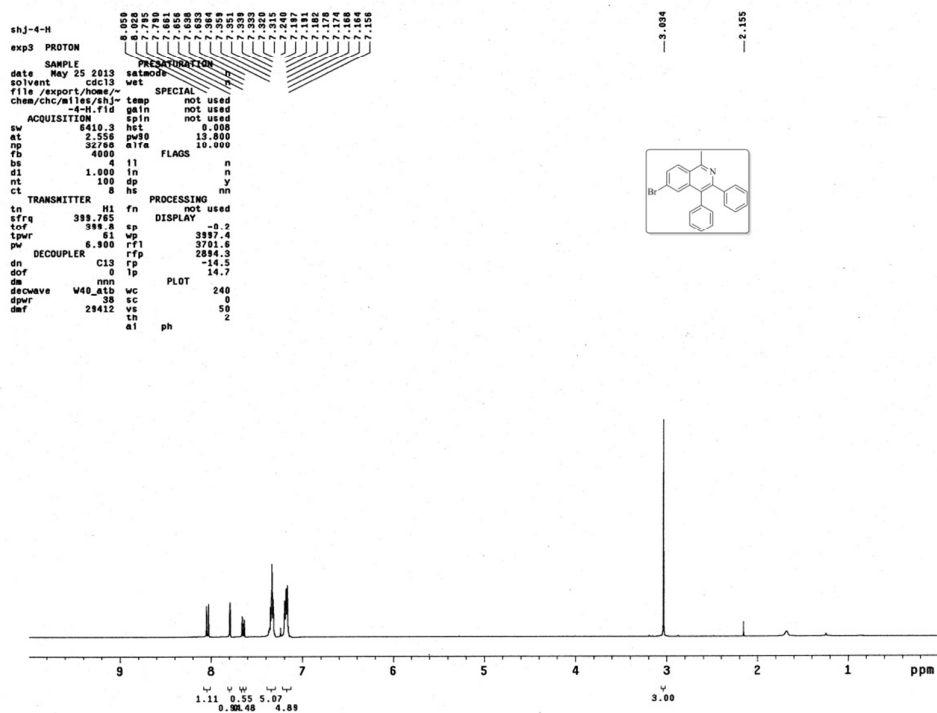
^1H and ^{13}C NMR spectra of compound **3da**

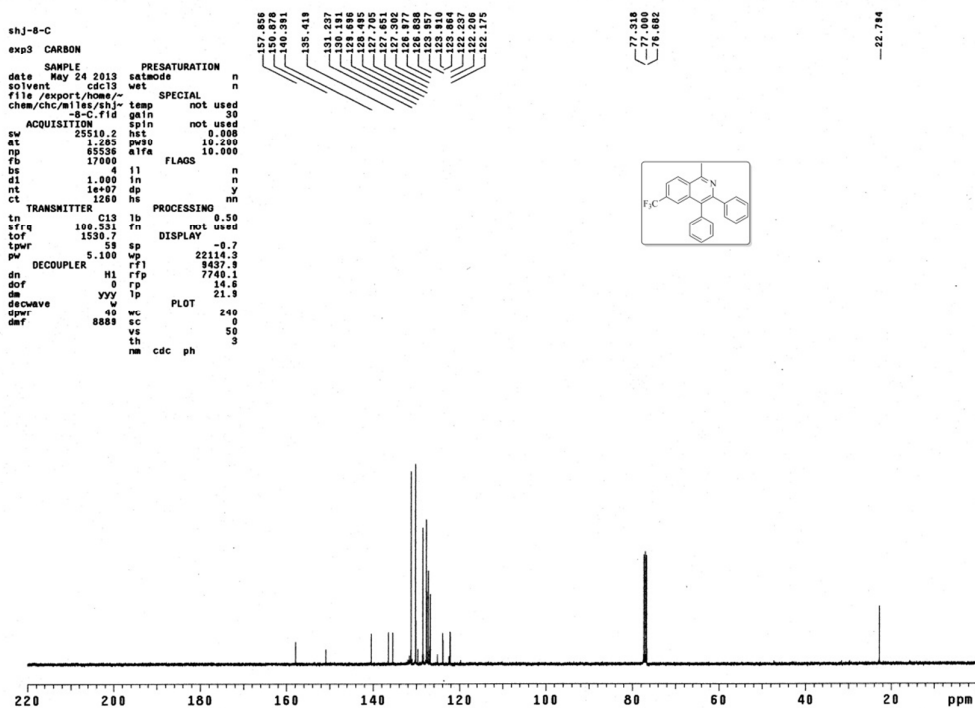
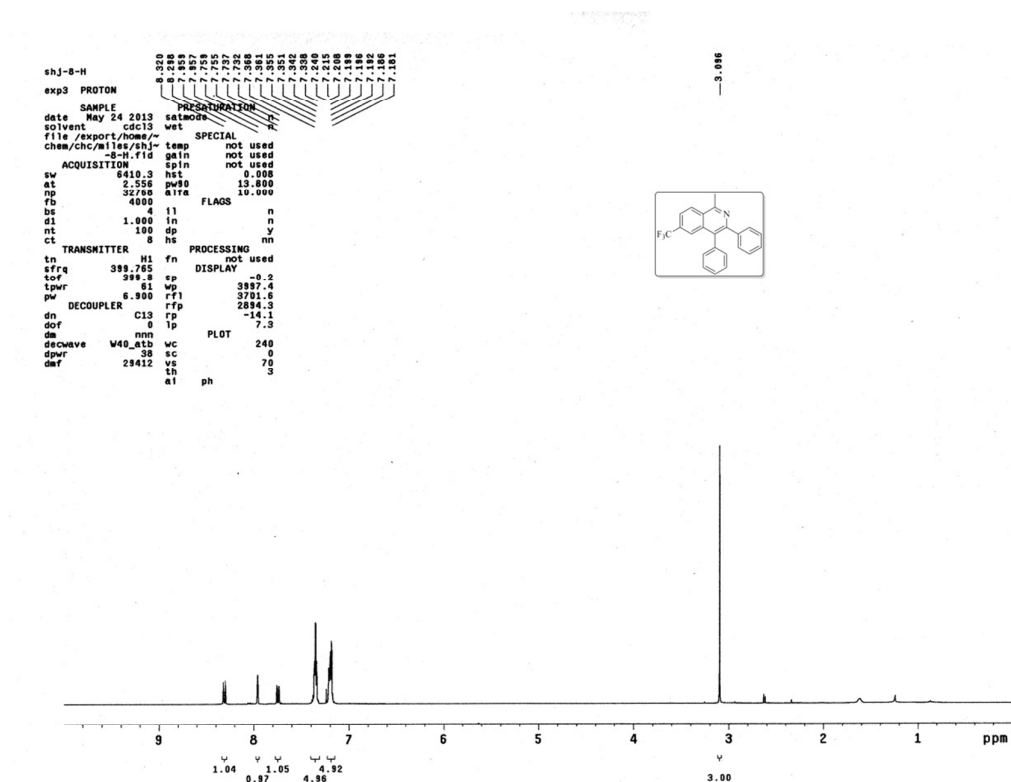


^1H and ^{13}C NMR spectra of compound **3ea**

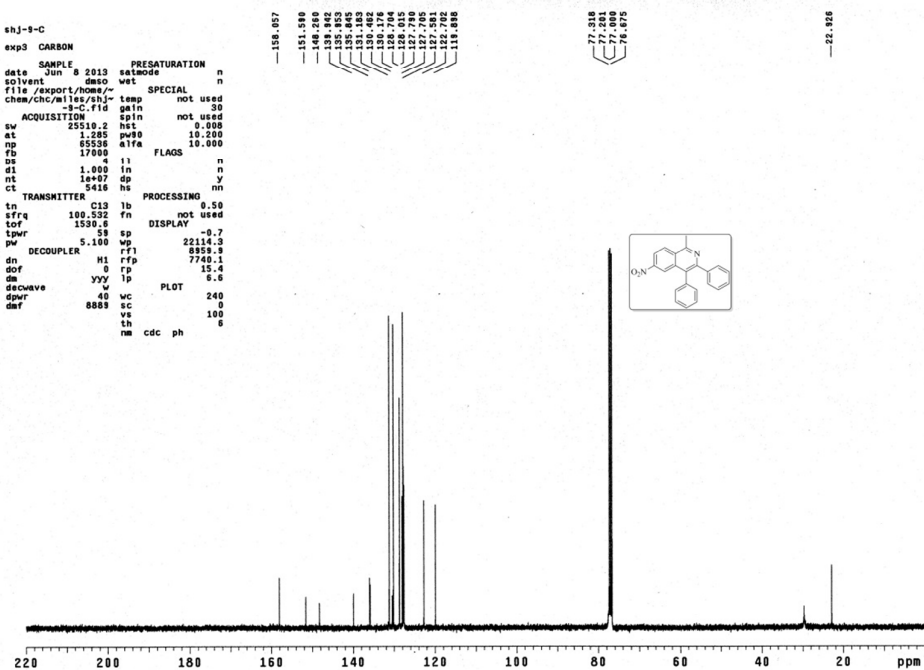
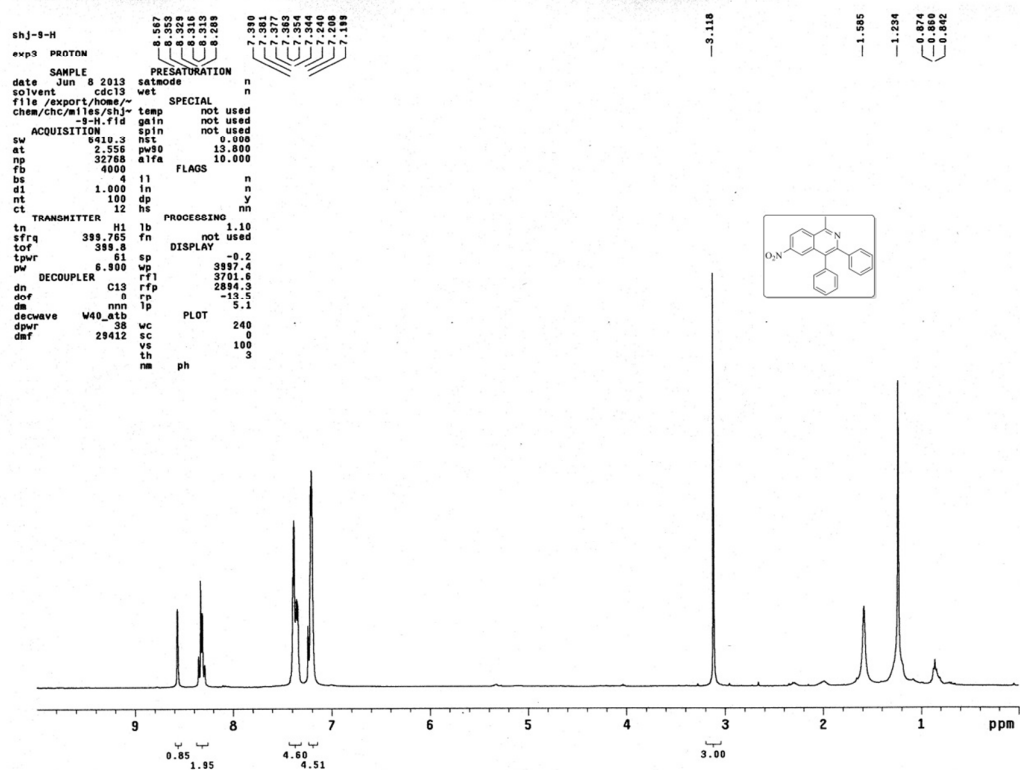


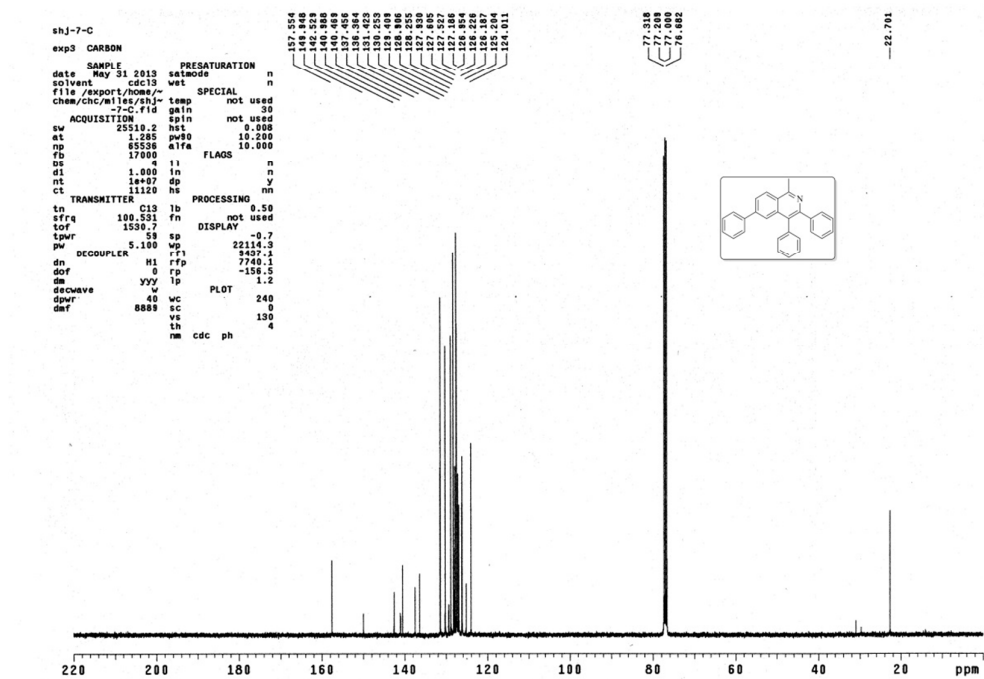
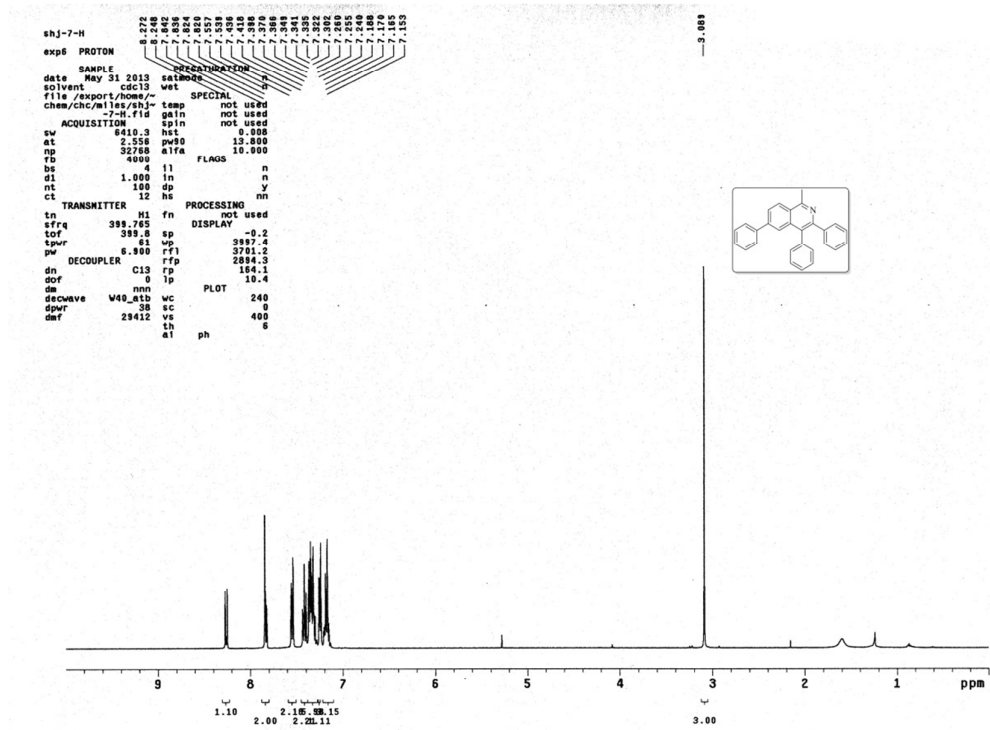
^1H and ^{13}C NMR spectra of compound **3fa**



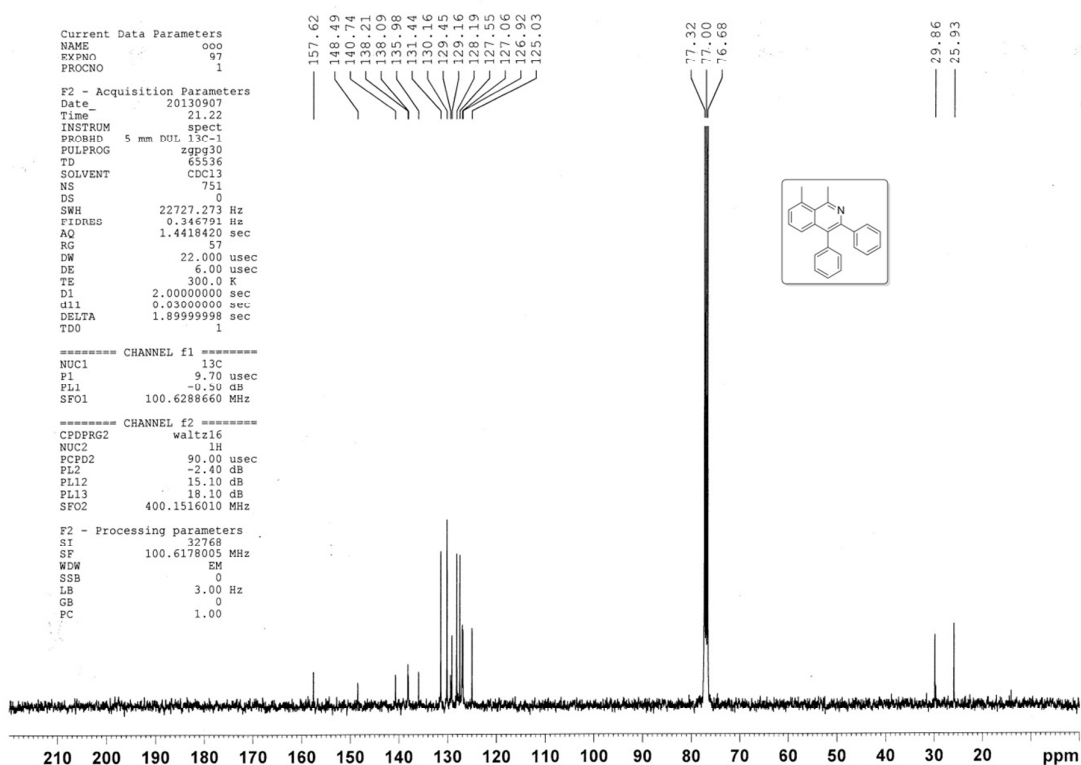
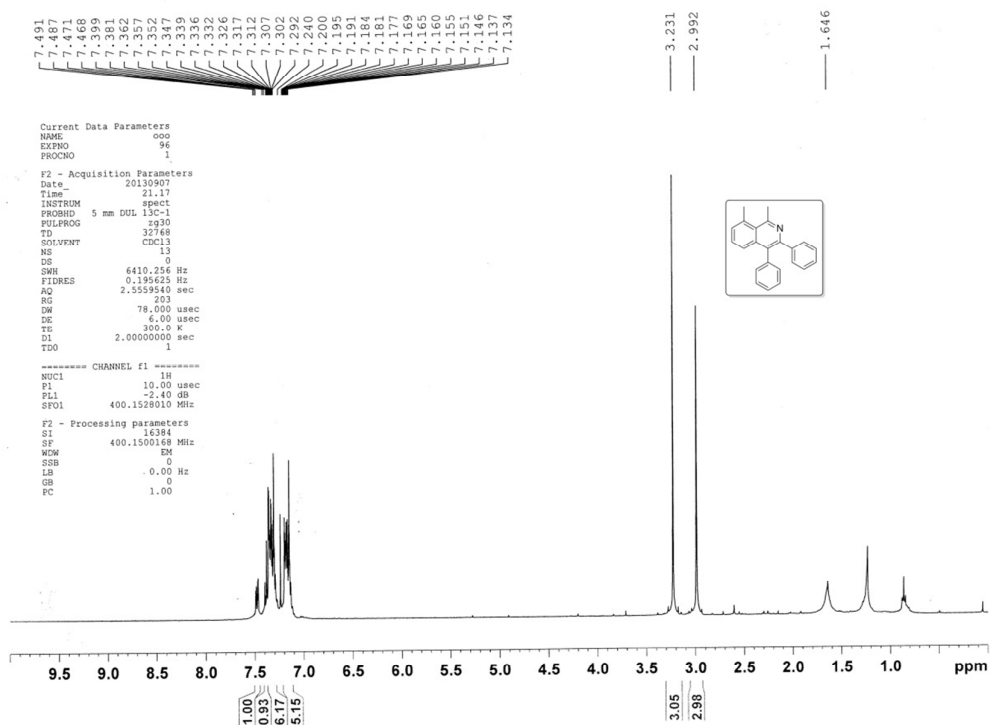


^1H and ^{13}C NMR spectra of compound **3ha**

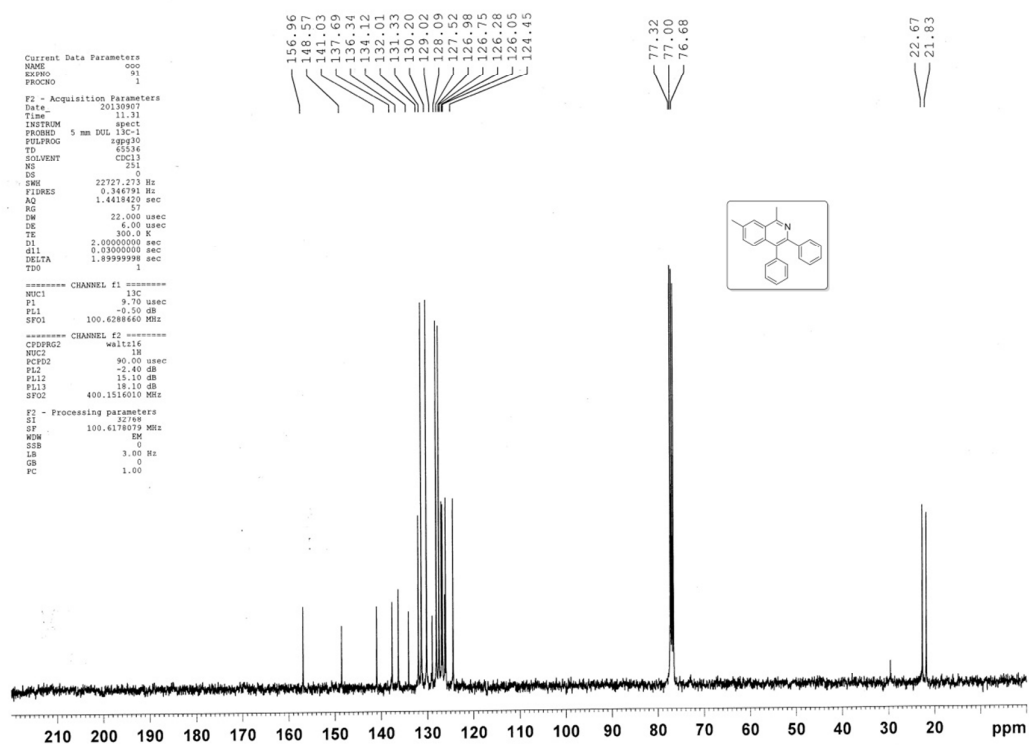
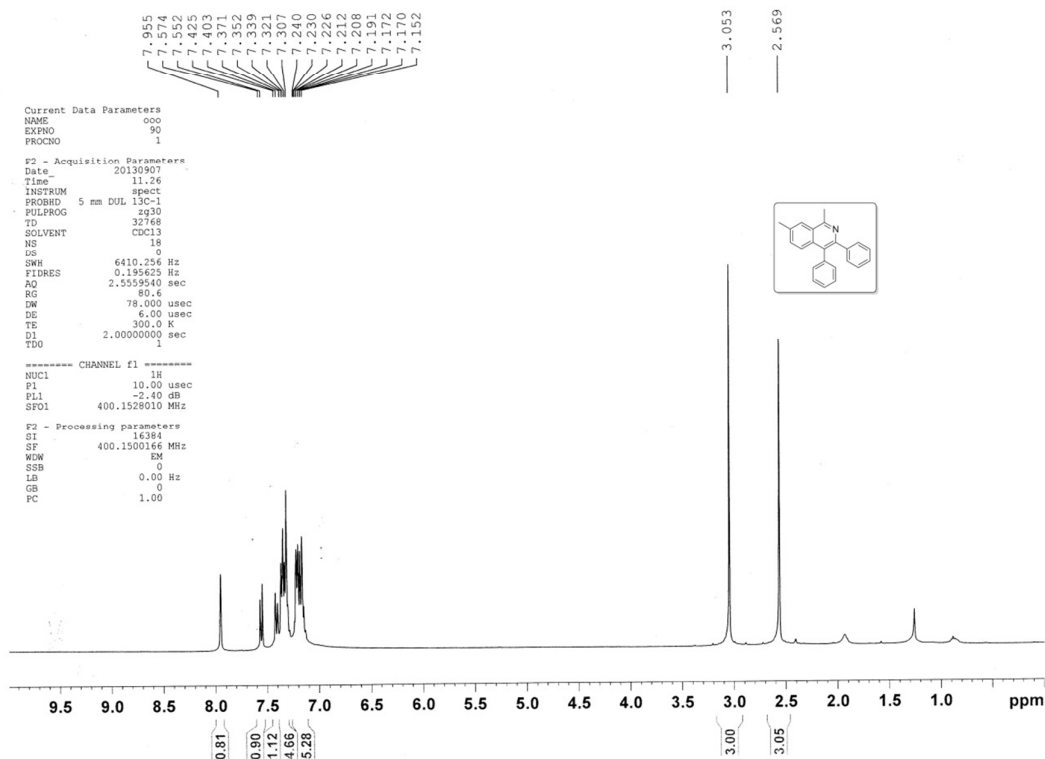


^1H and ^{13}C NMR spectra of compound **3ia**

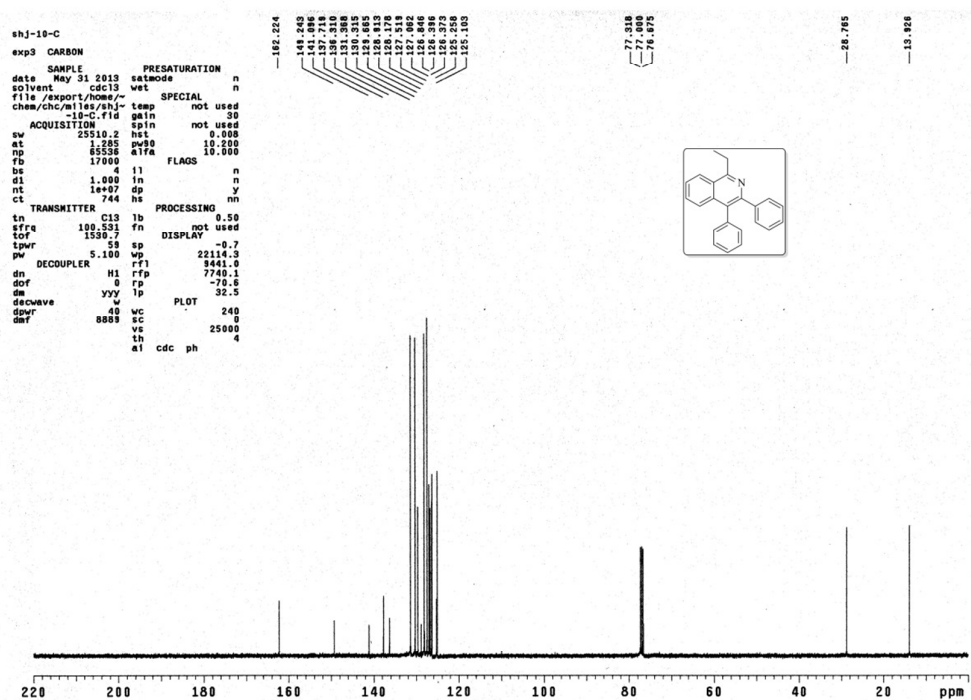
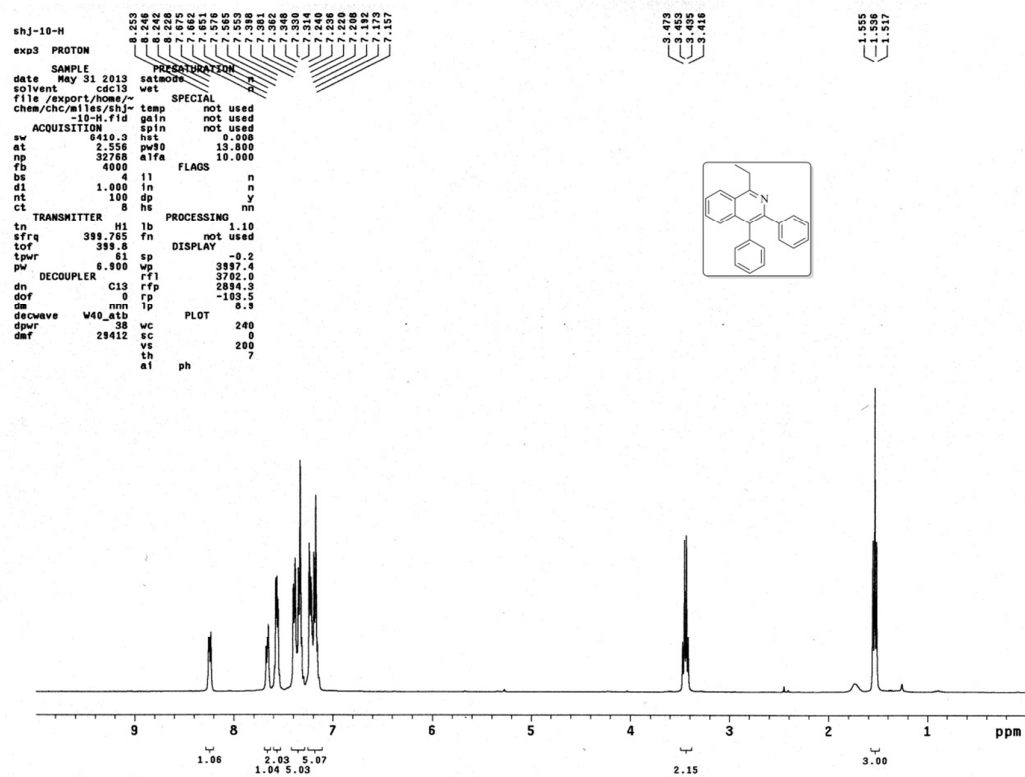
^1H and ^{13}C NMR spectra of compound **3ma**



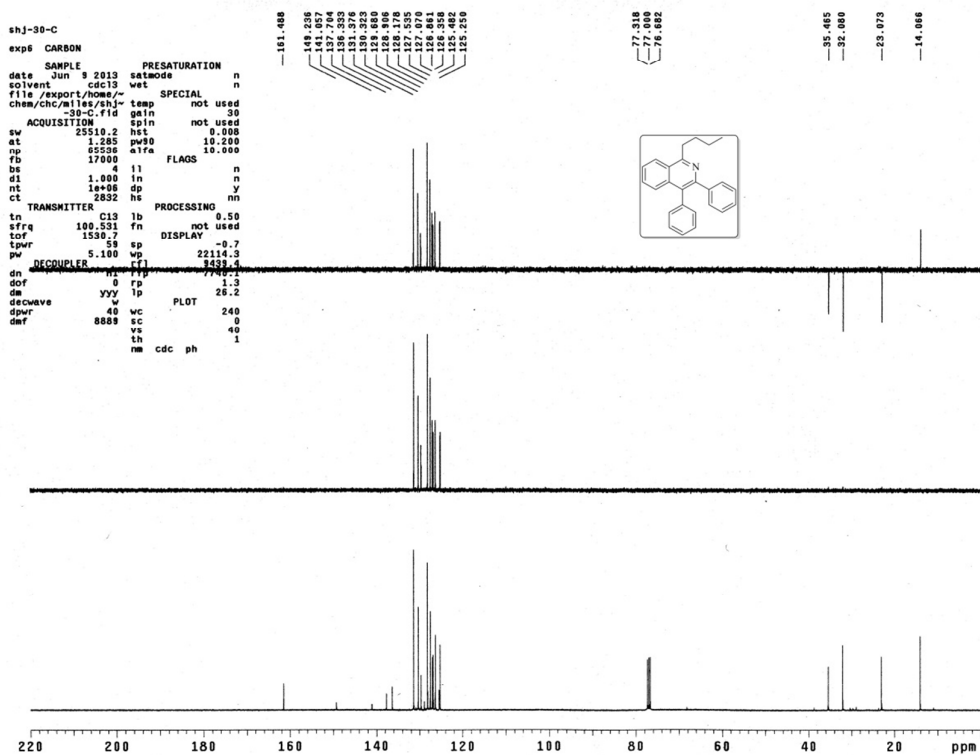
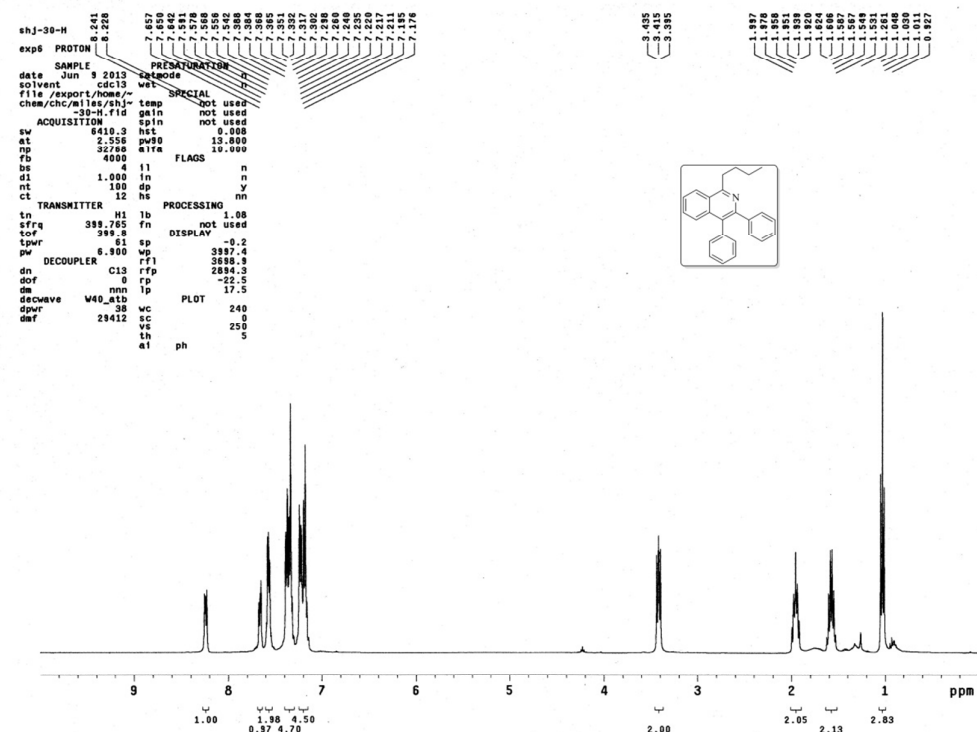
^1H and ^{13}C NMR spectra of compound **3na**



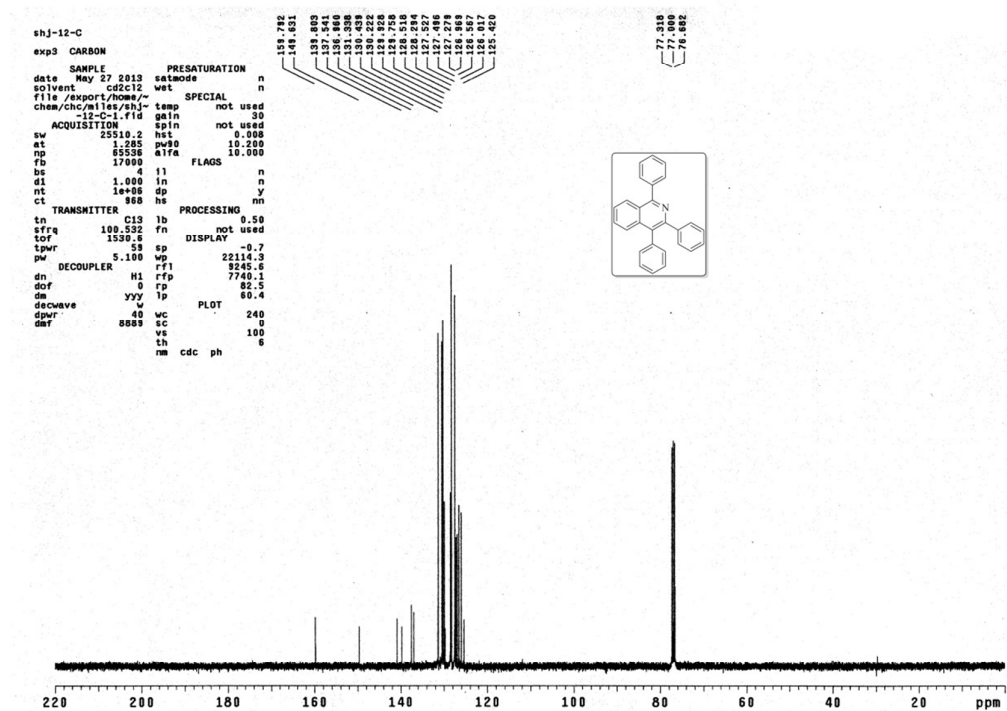
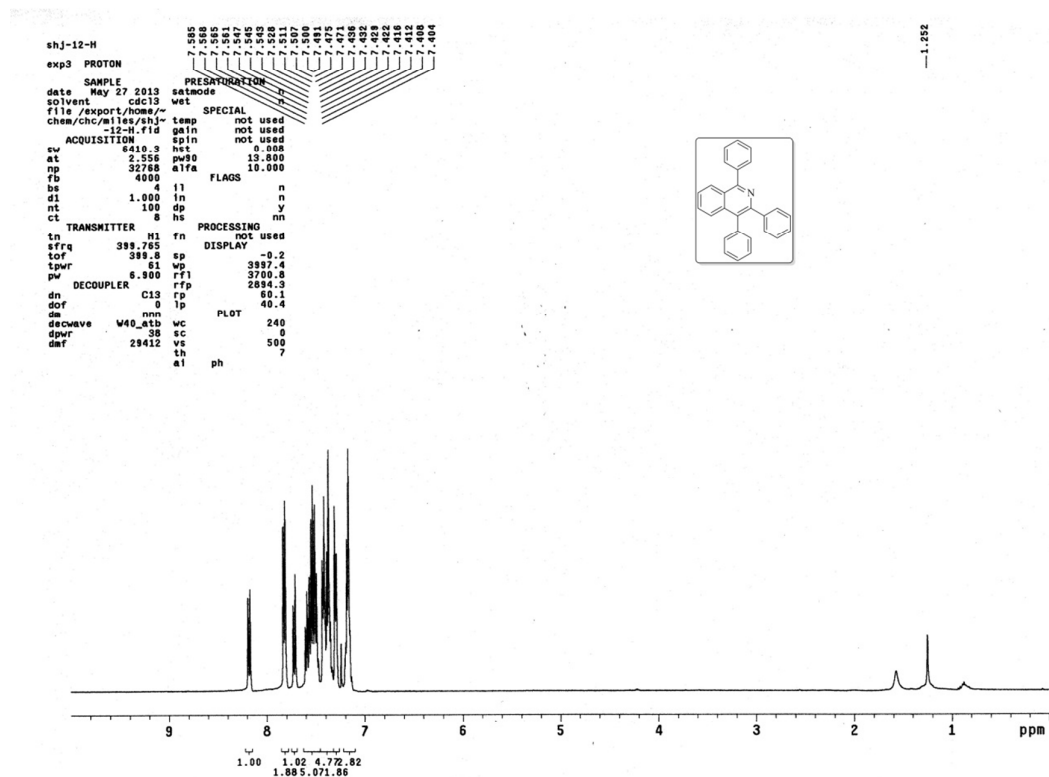
^1H and ^{13}C NMR spectra of compound **3ja**



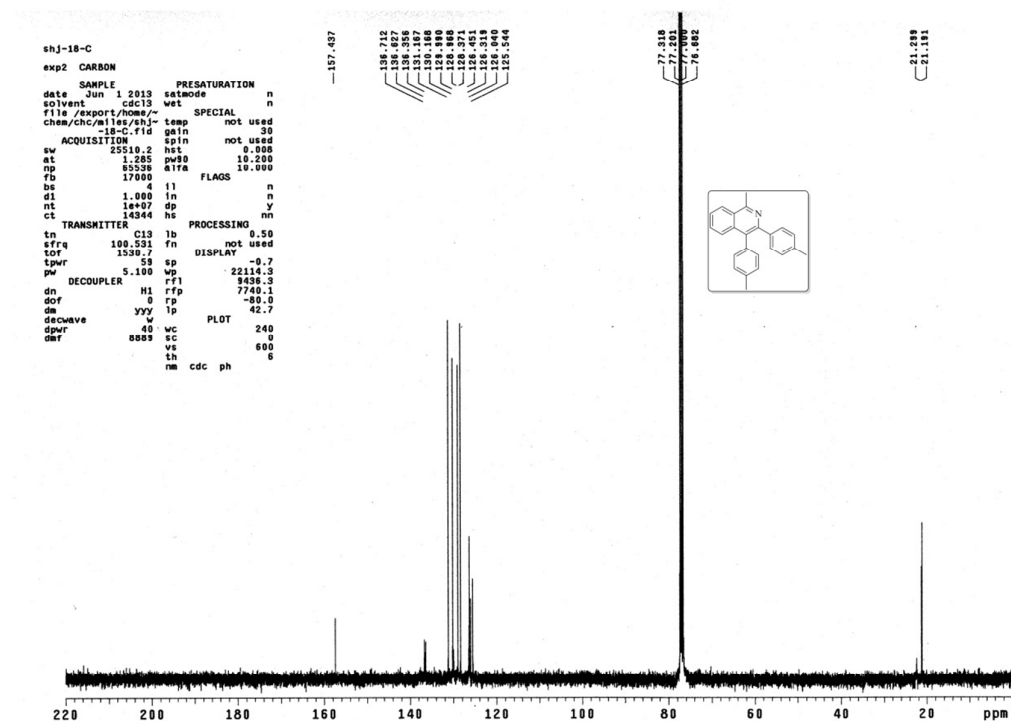
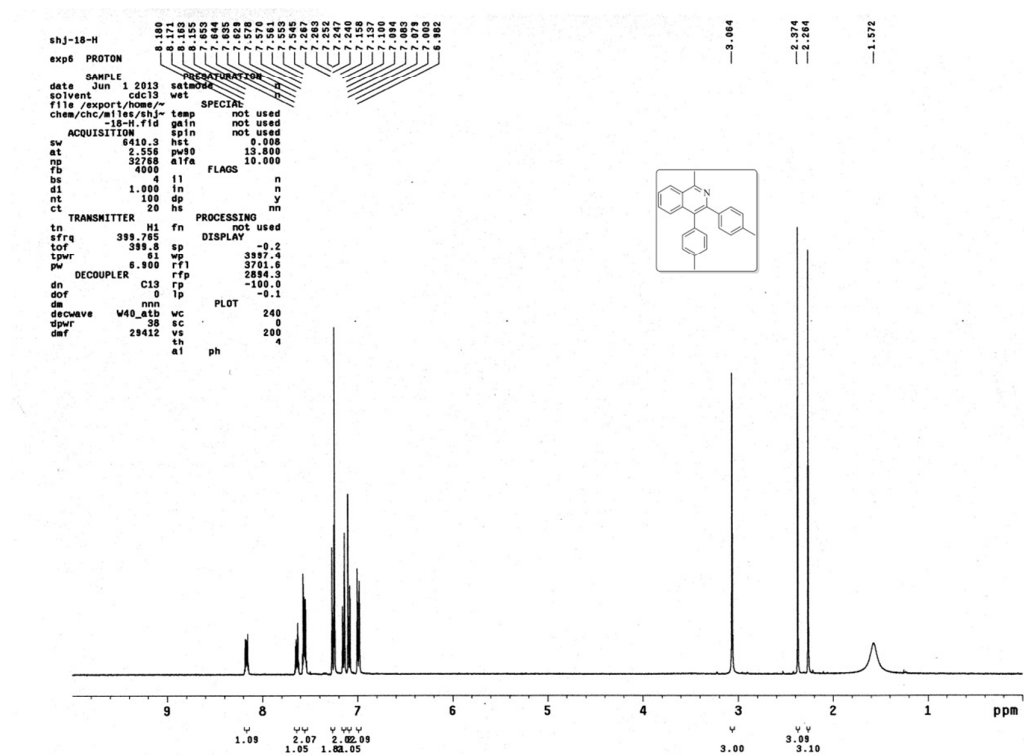
^1H and ^{13}C NMR spectra of compound **3ka**

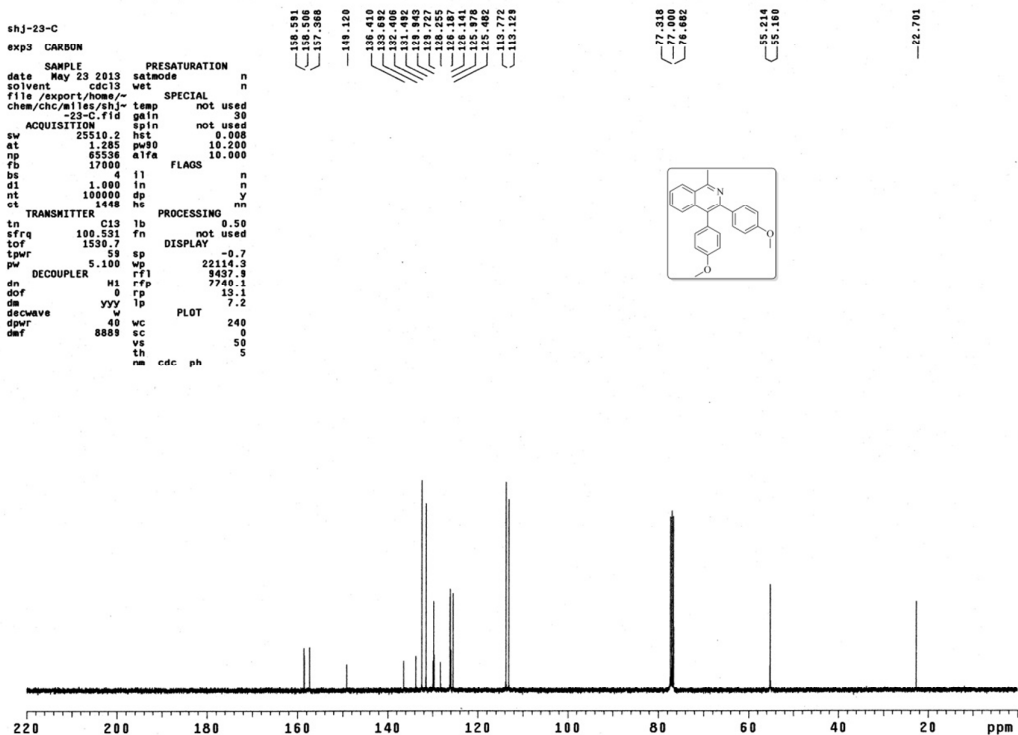
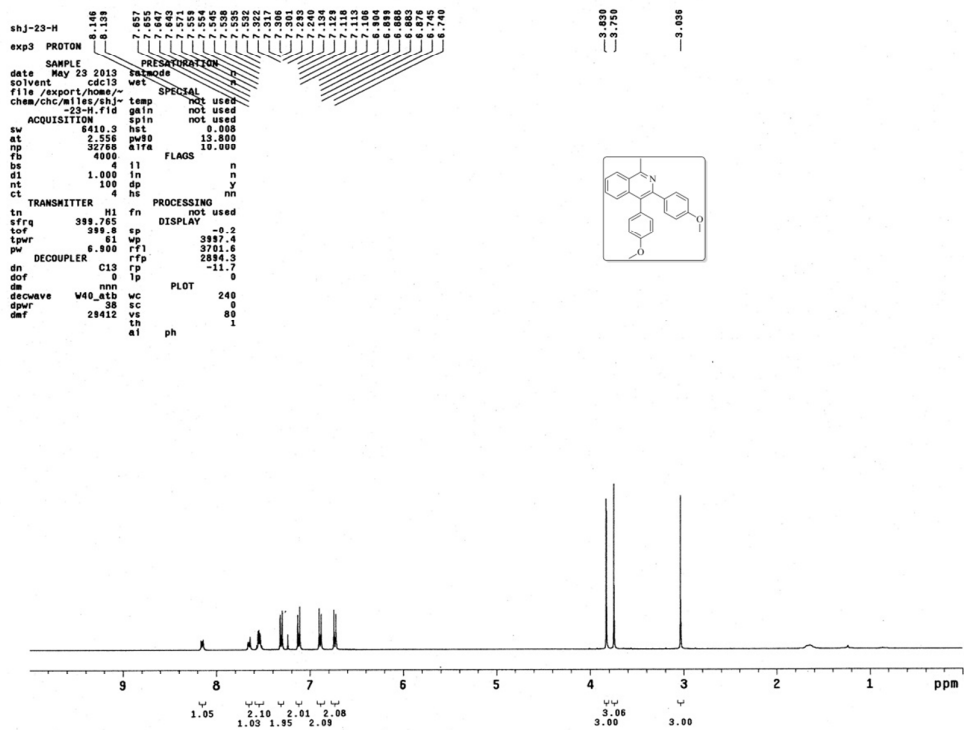


^1H and ^{13}C NMR spectra of compound **3la**

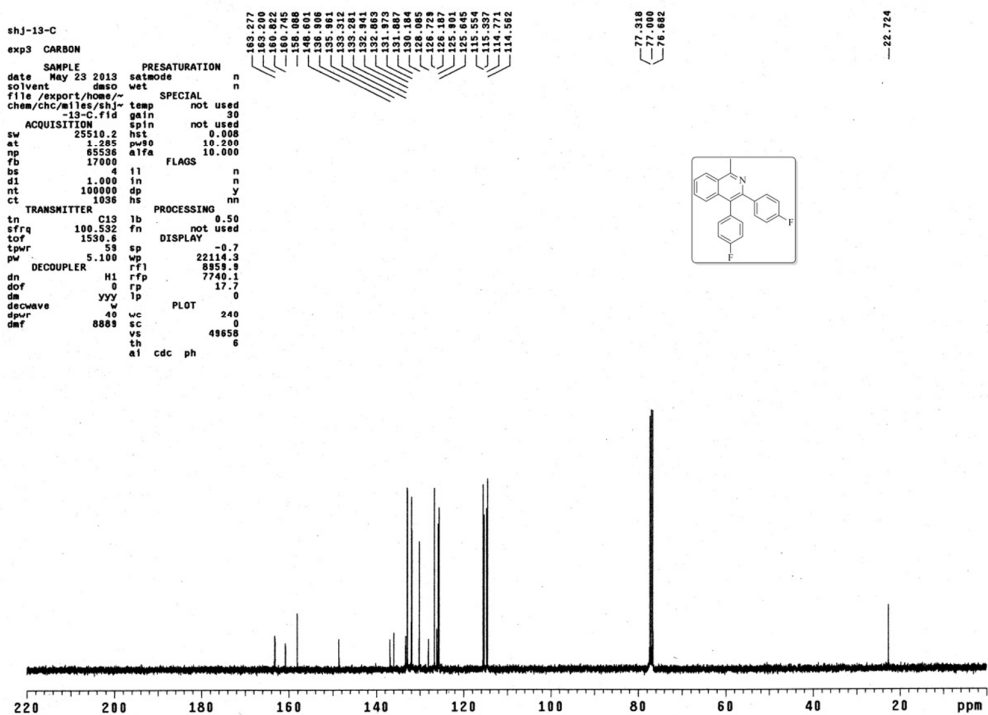
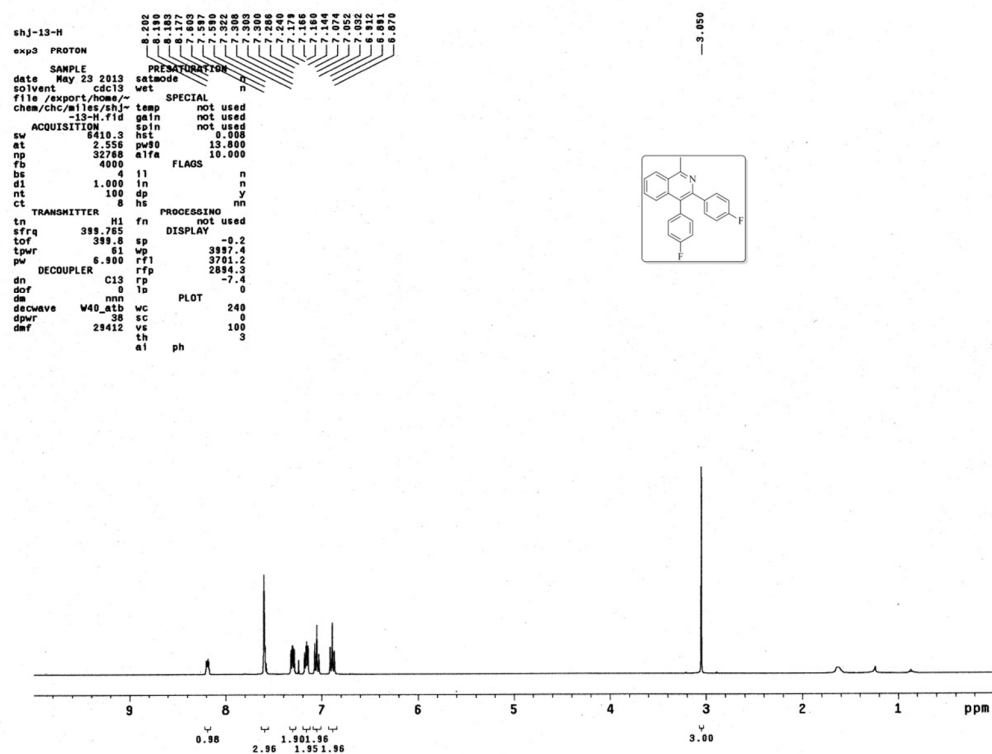


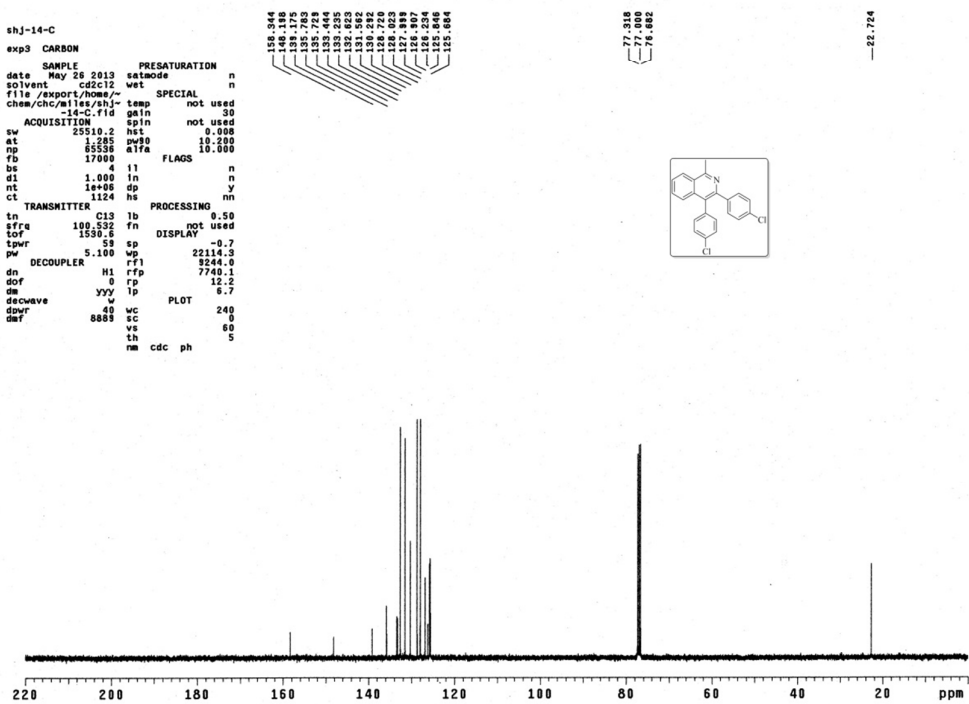
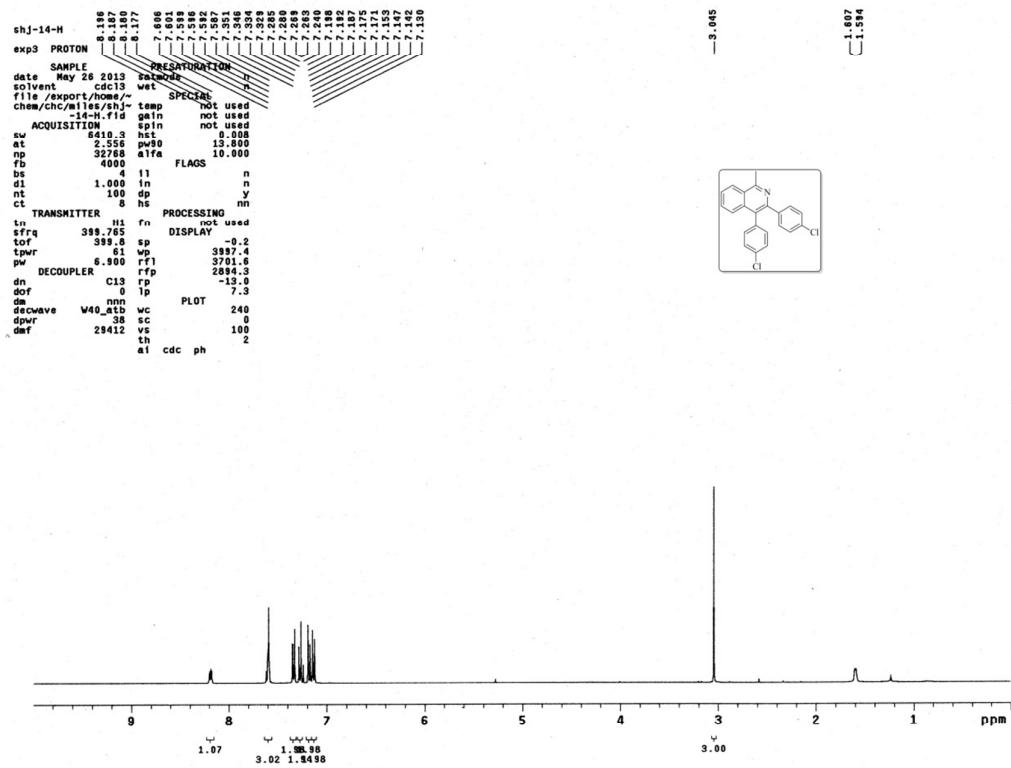
^1H and ^{13}C NMR spectra of compound **3ab**



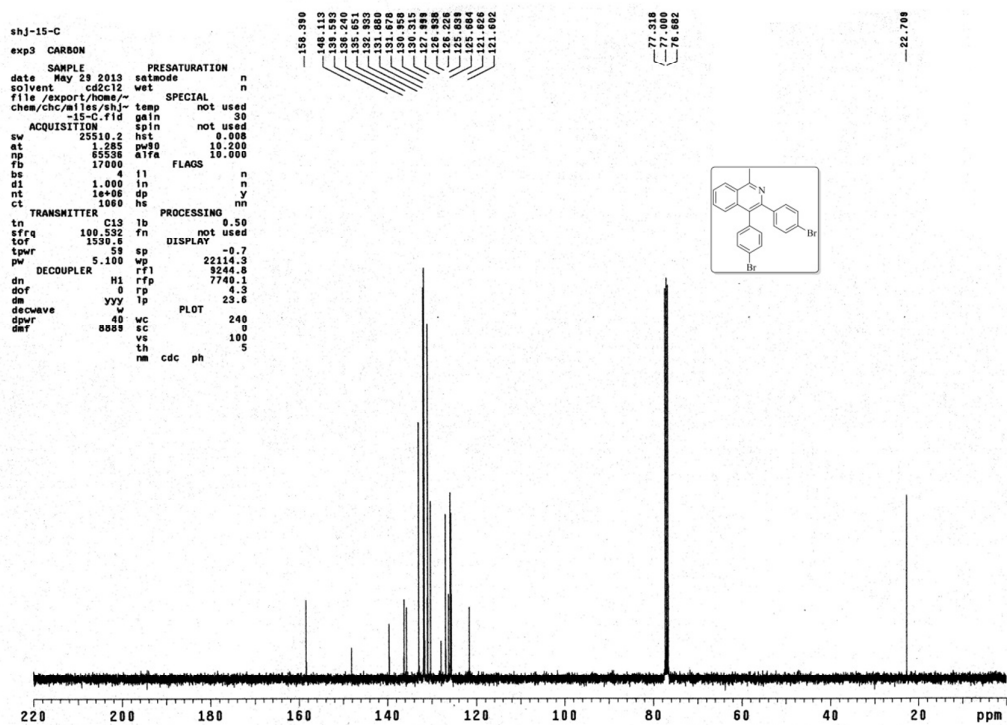
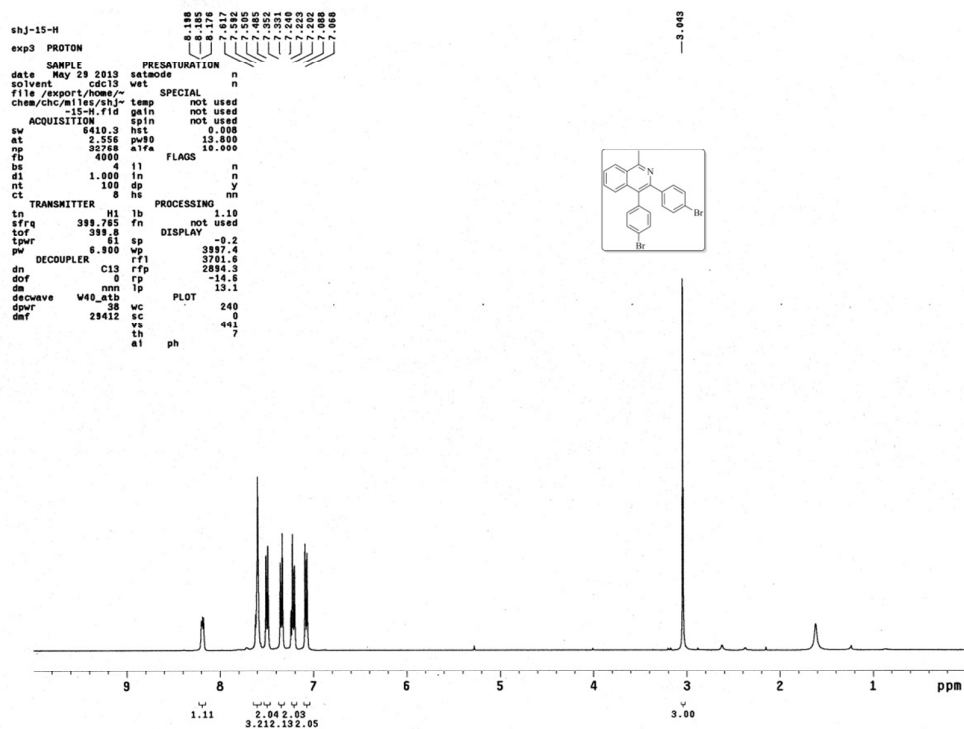
^1H and ^{13}C NMR spectra of compound **3ac**

^1H and ^{13}C NMR spectra of compound **3ad**

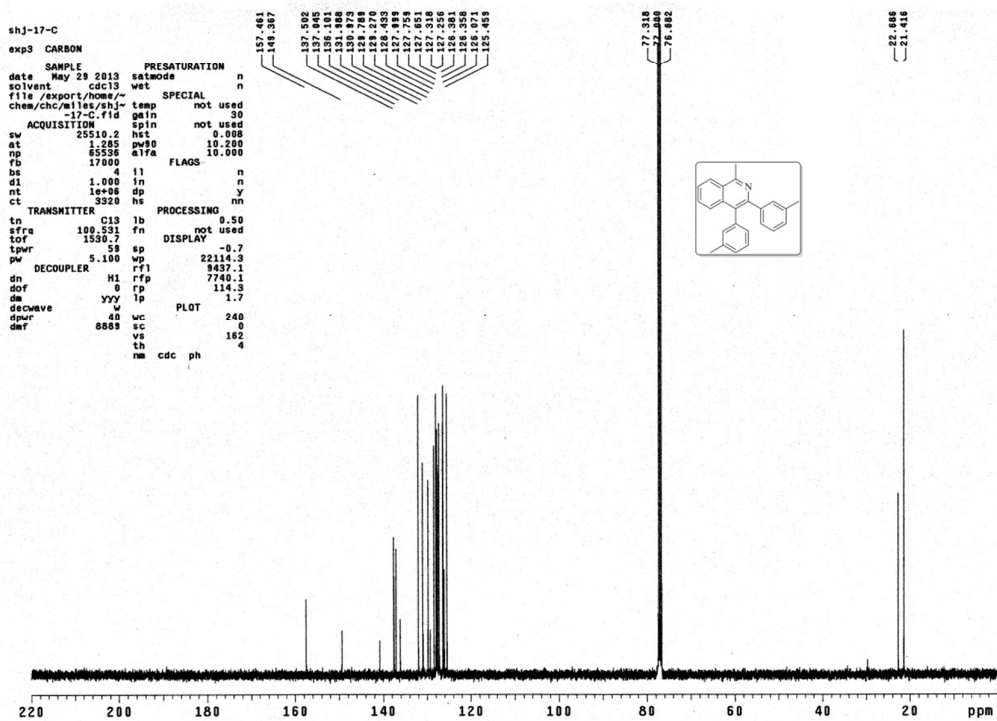
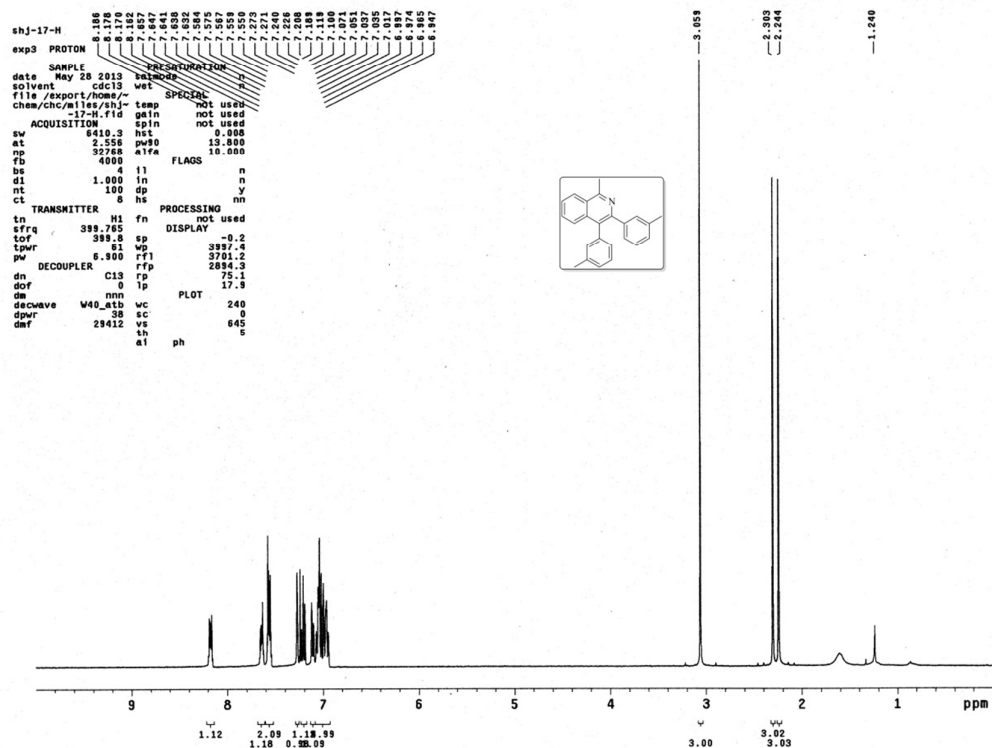


^1H and ^{13}C NMR spectra of compound **3ae**

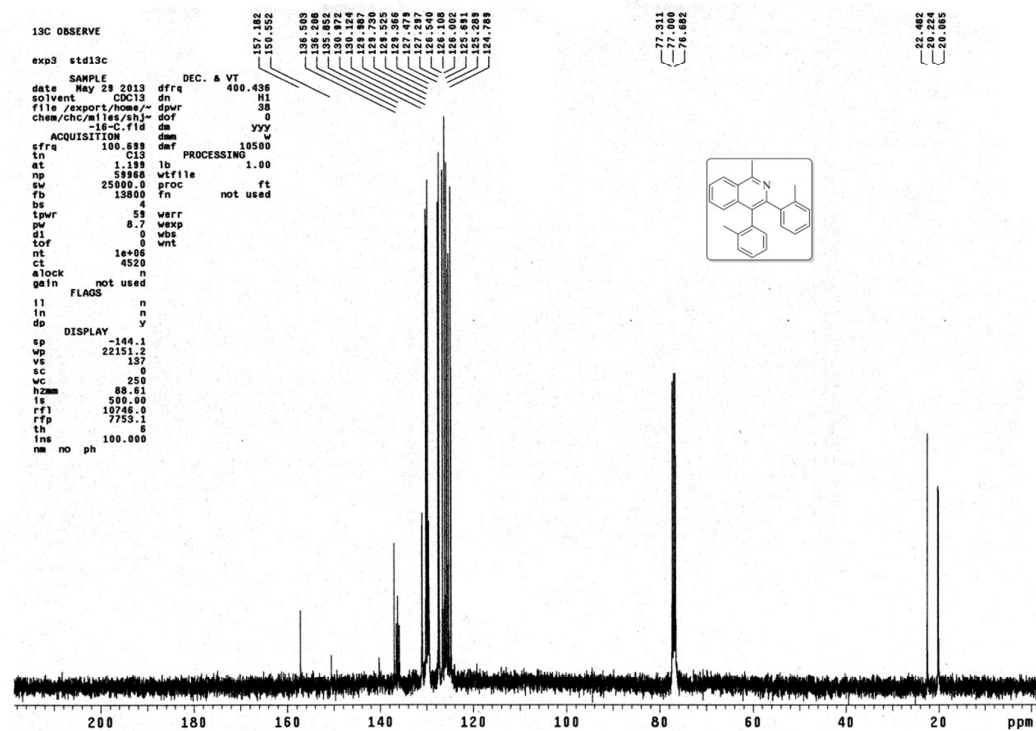
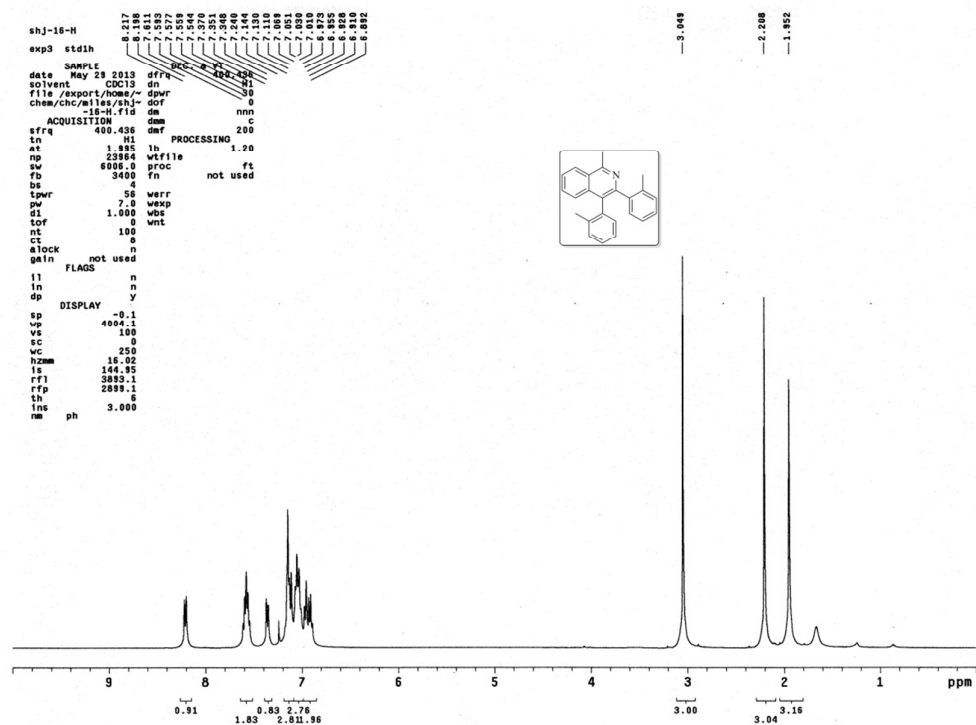
^1H and ^{13}C NMR spectra of compound **3af**

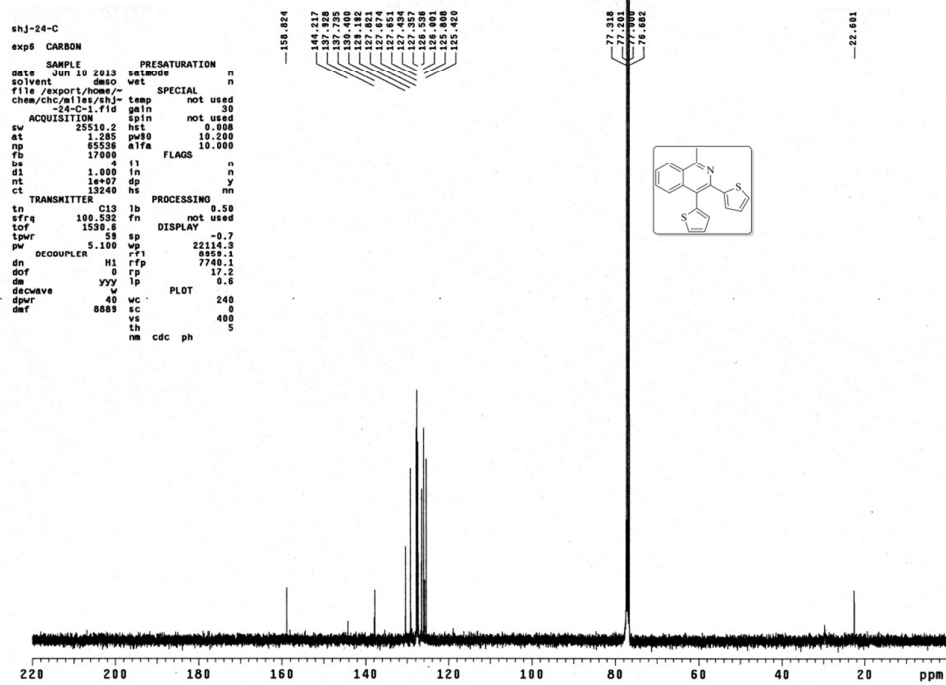
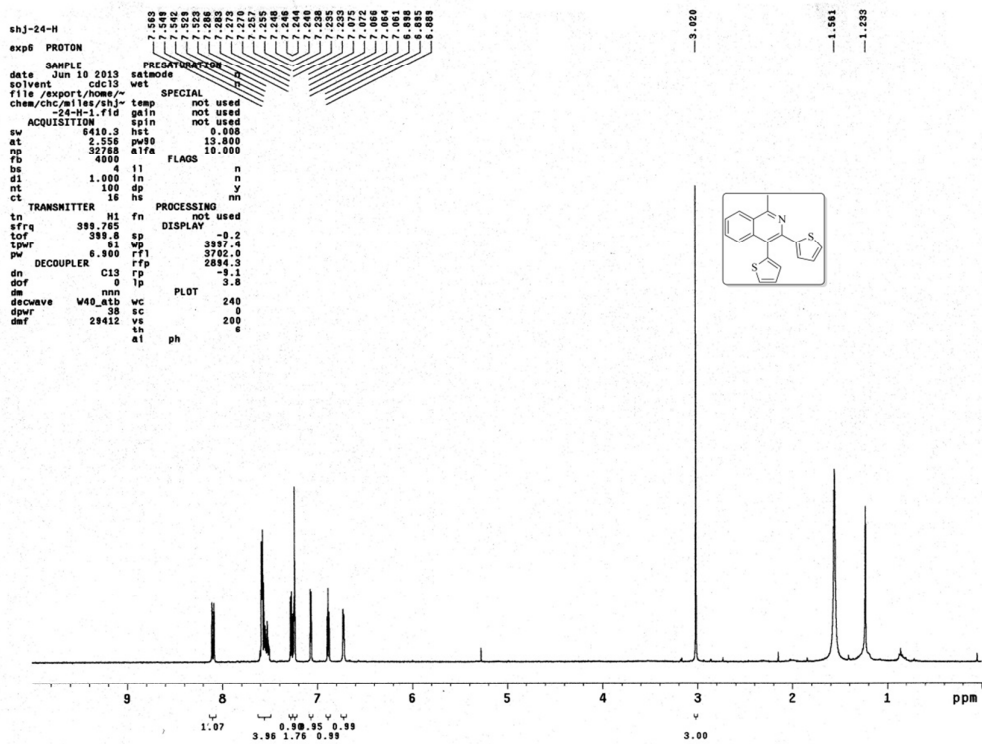


^1H and ^{13}C NMR spectra of compound **3ag**

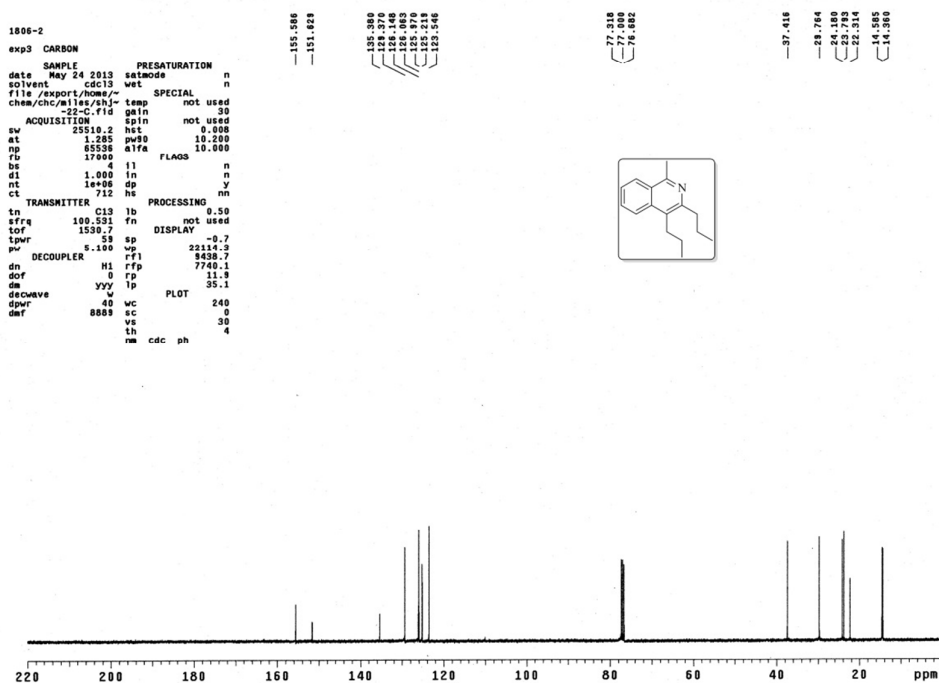
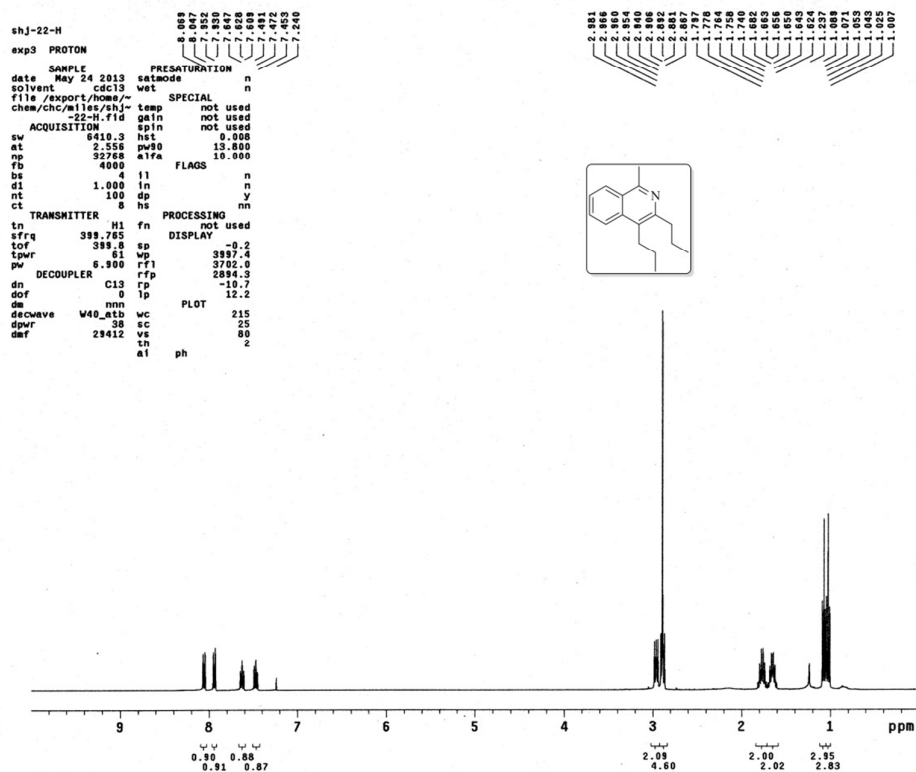


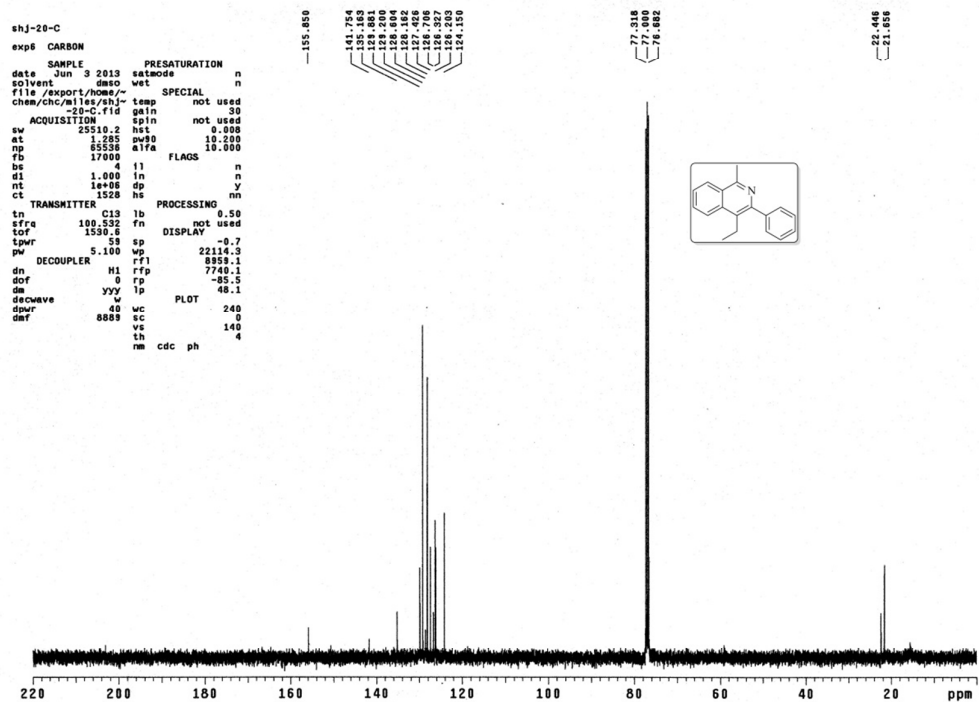
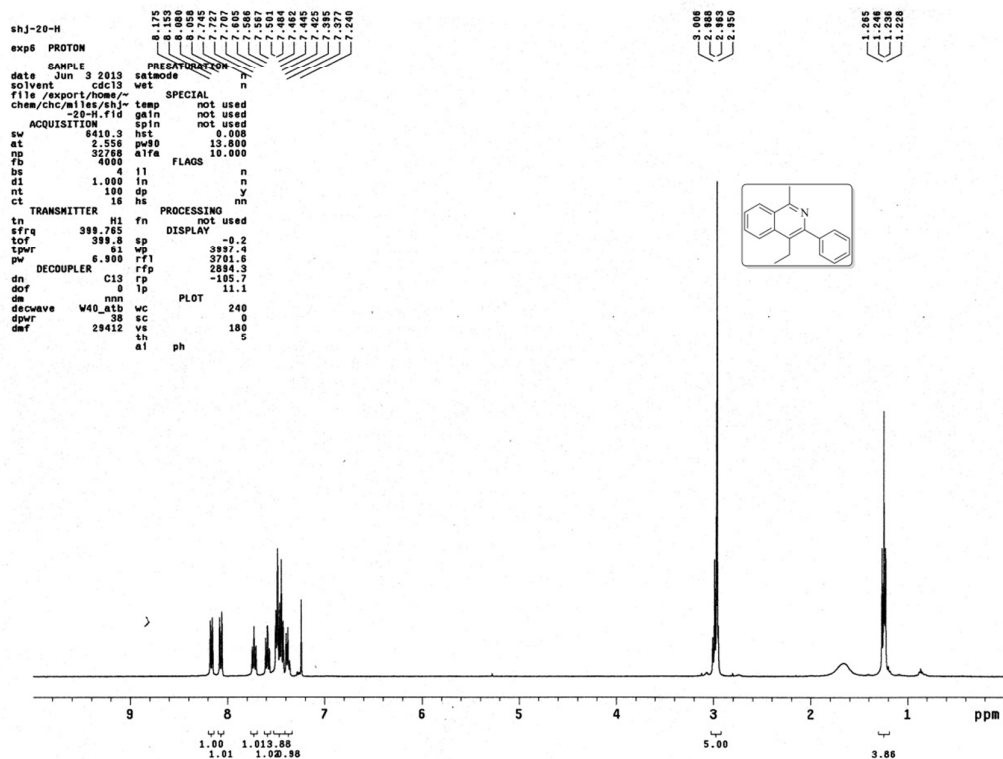
^1H and ^{13}C NMR spectra of compound **3ah**



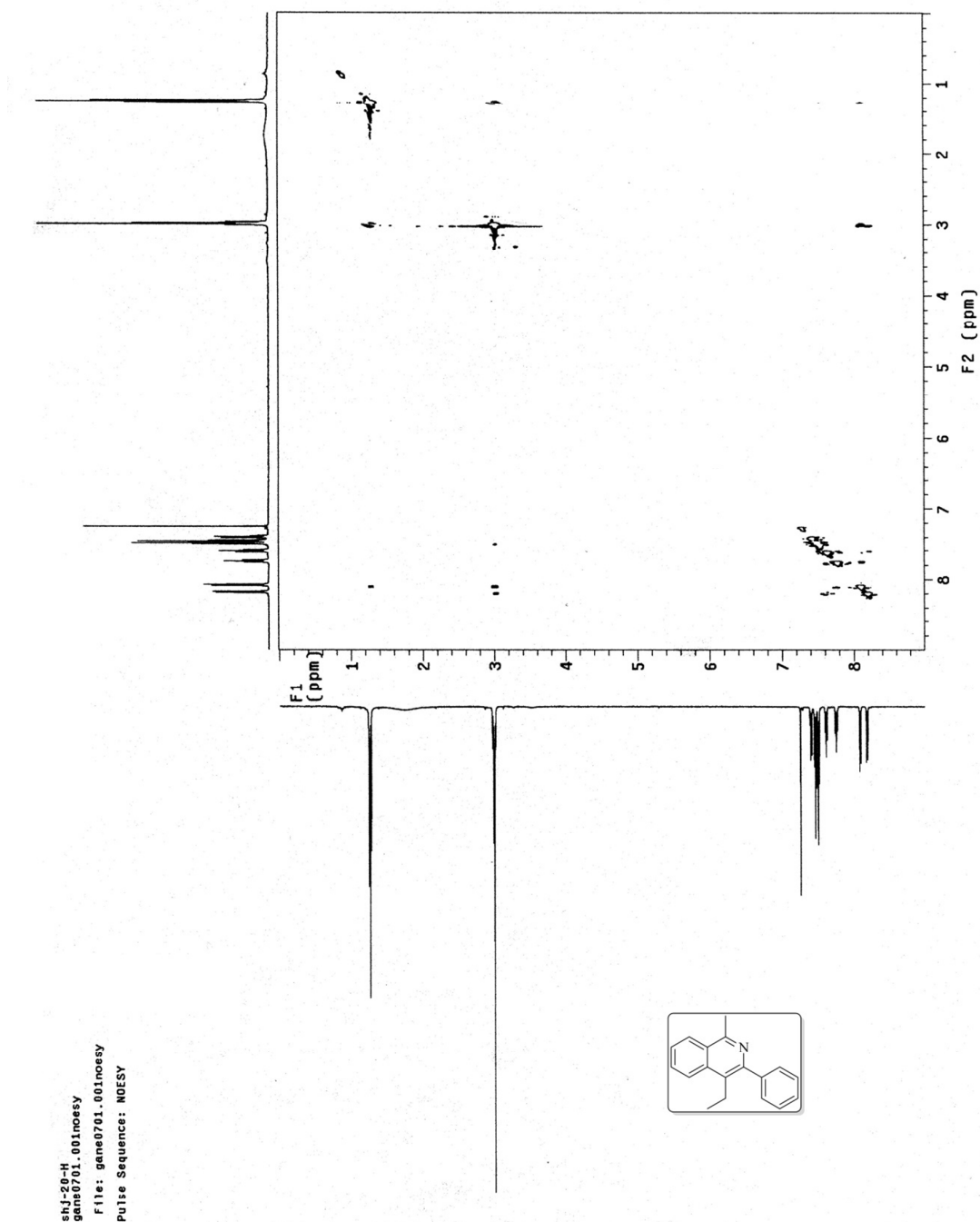
^1H and ^{13}C NMR spectra of compound **3ai**

^1H and ^{13}C NMR spectra of compound **3aj**

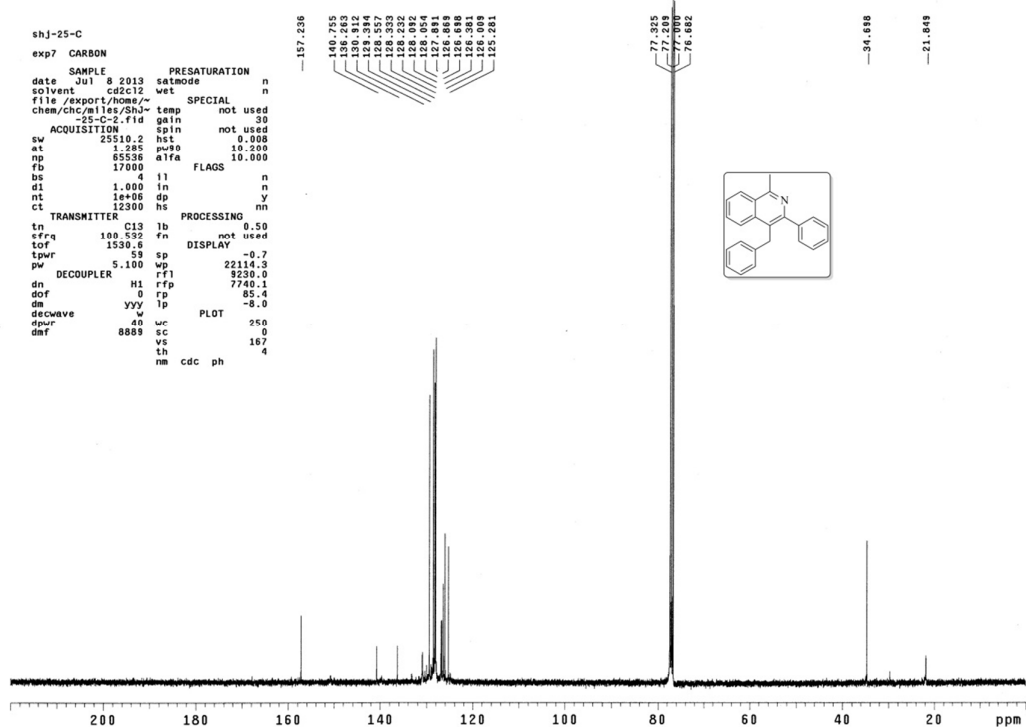
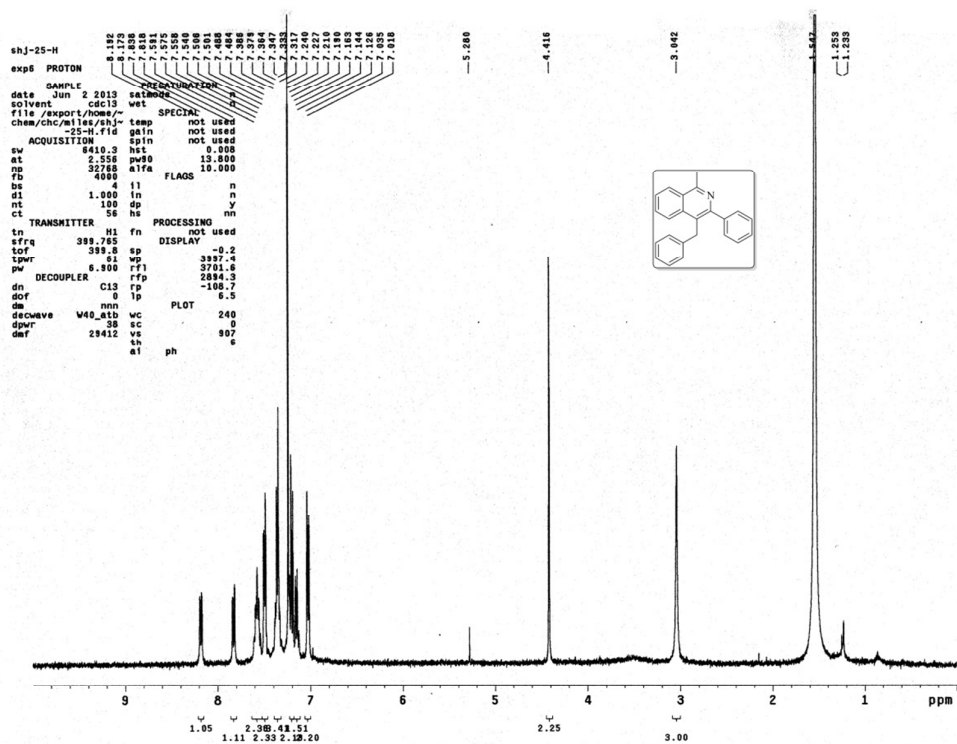


¹H and ¹³C NMR spectra of compound **3ak**

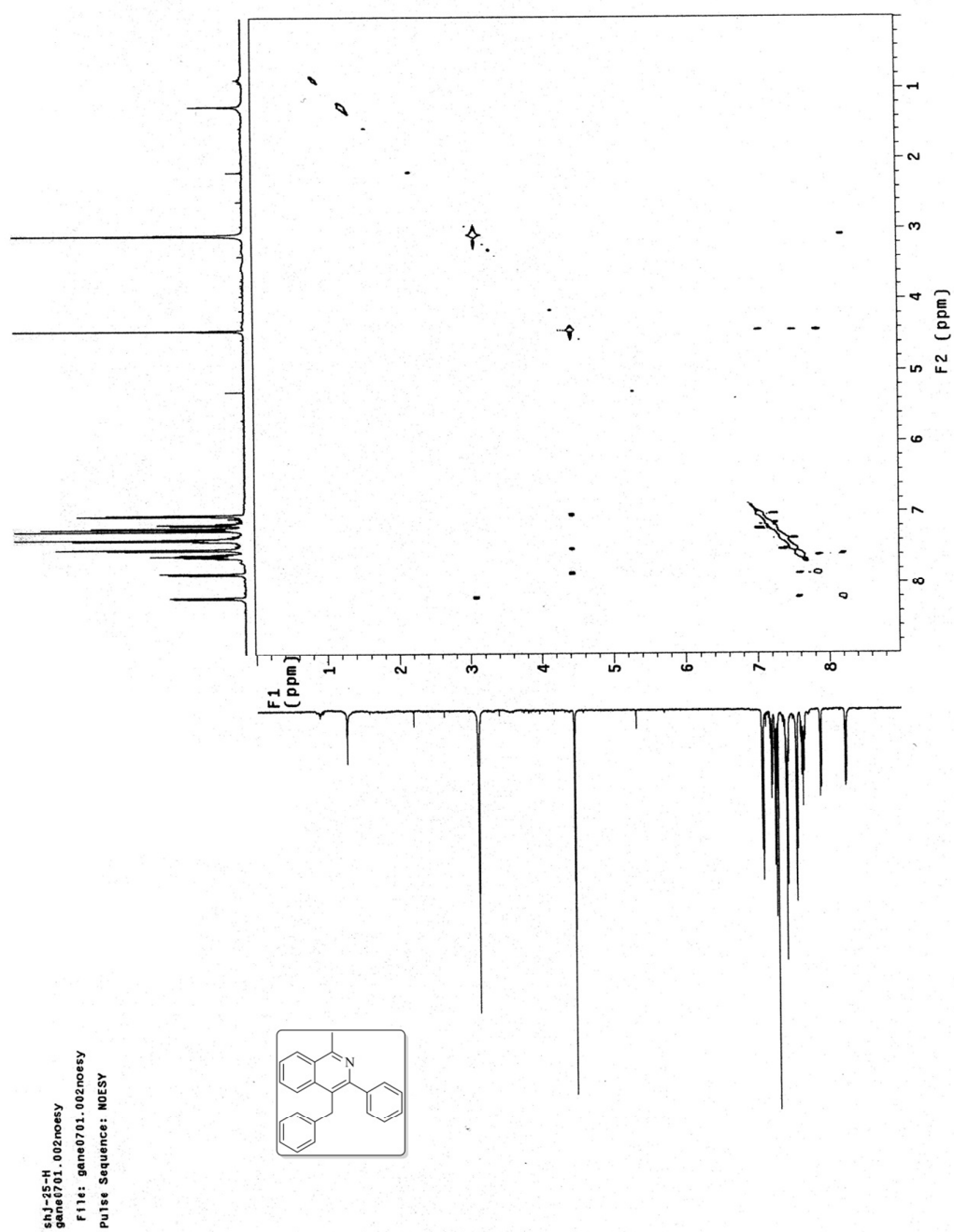
NOESY spectrum of **3ak**



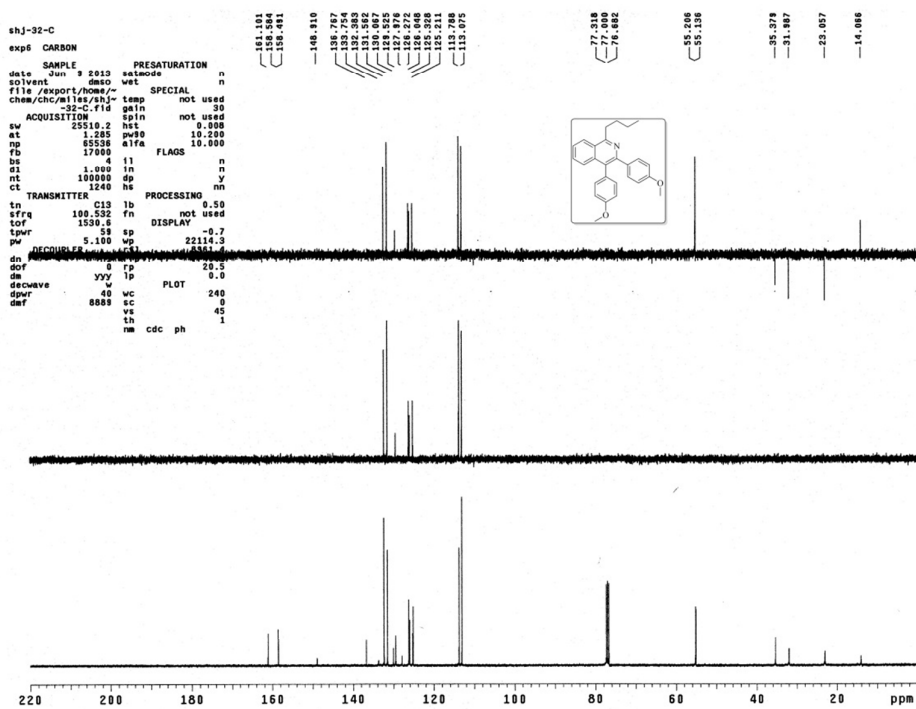
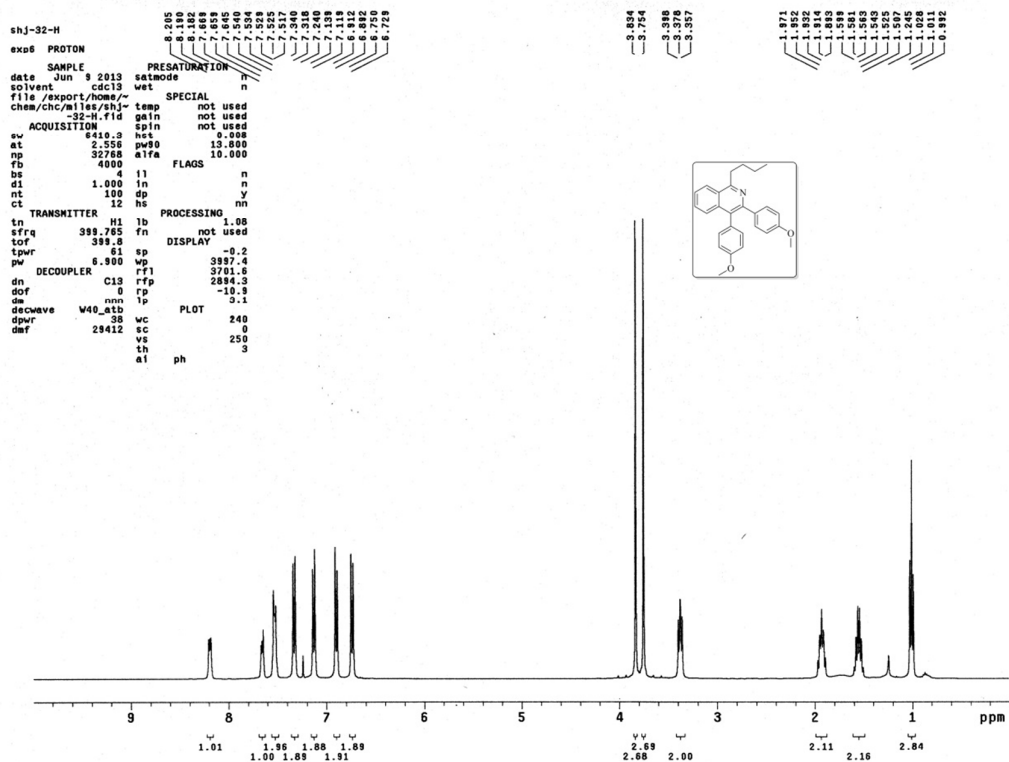
^1H and ^{13}C NMR spectra of compound **3al**



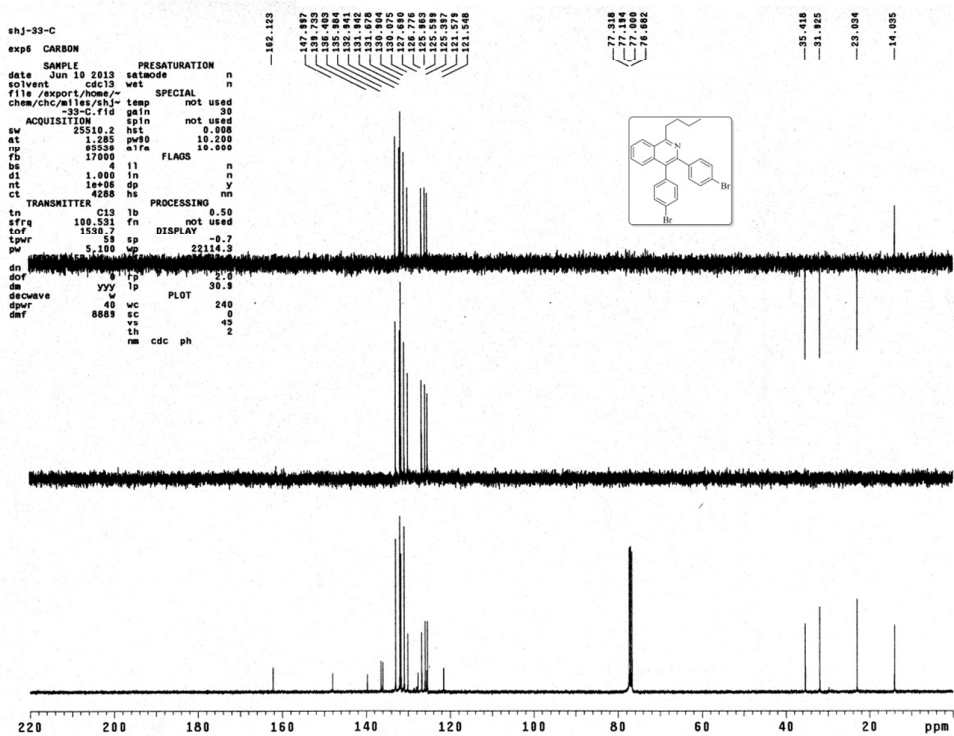
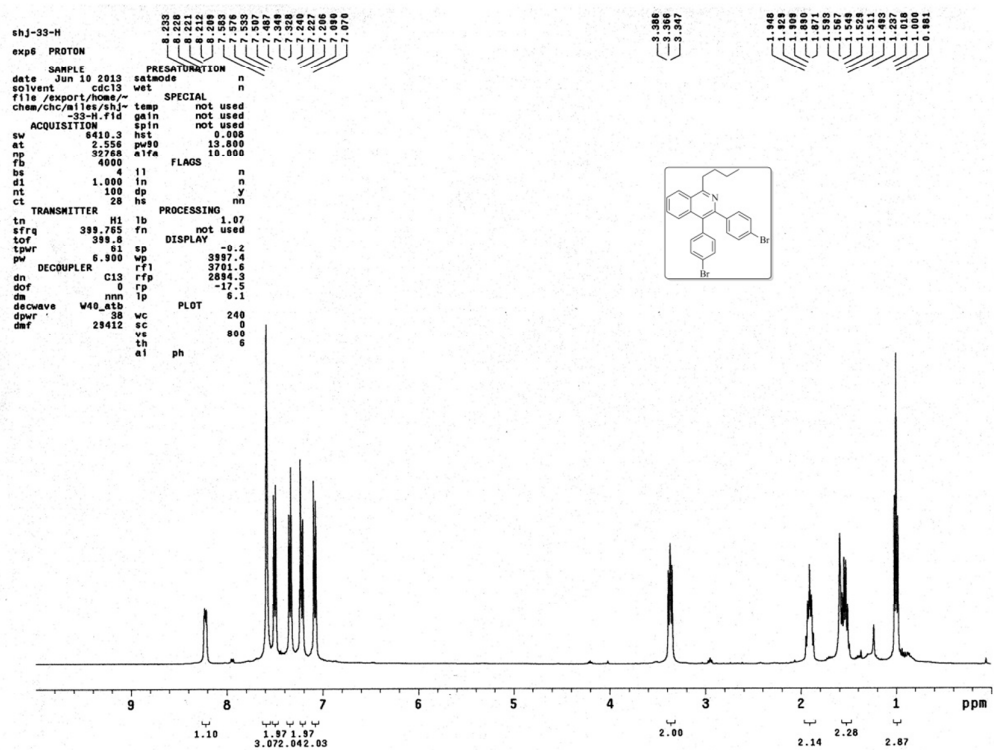
NOESY spectrum of **3al**



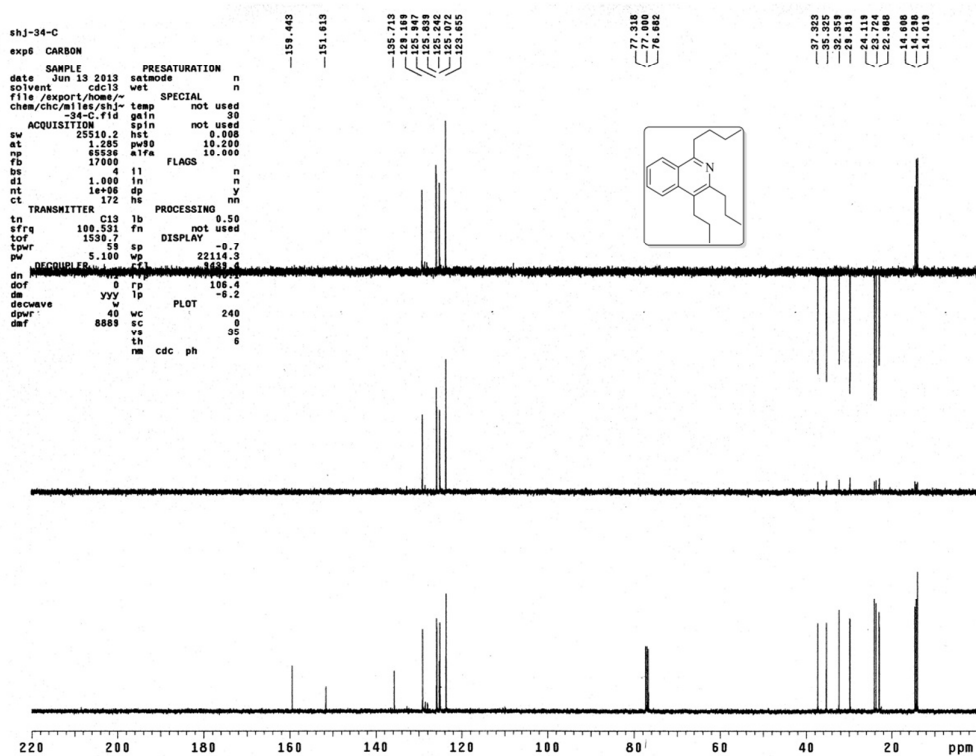
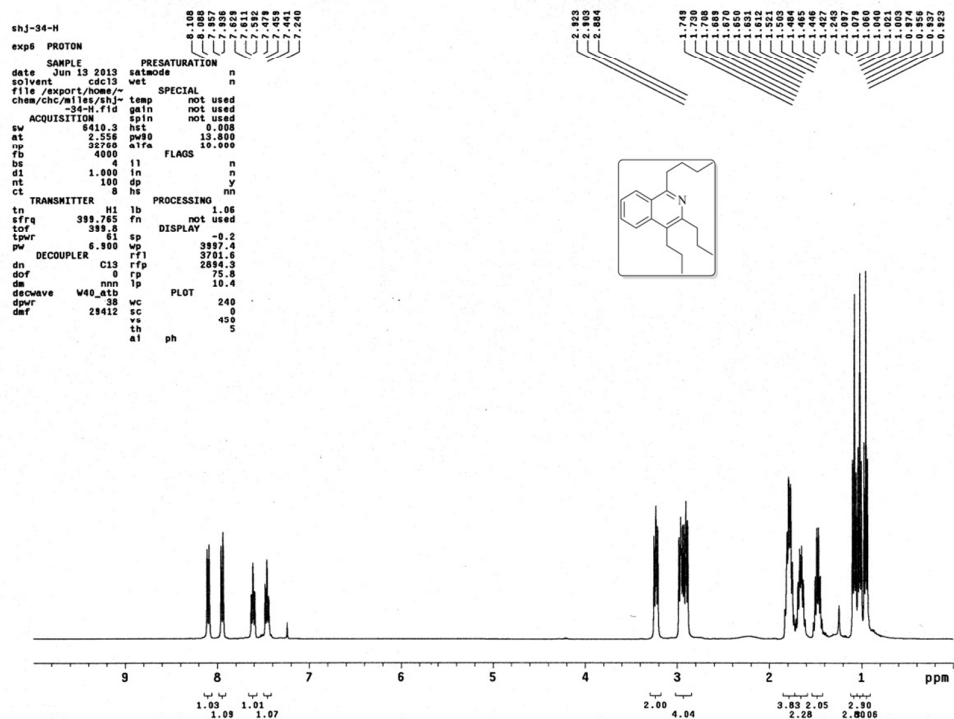
^1H and ^{13}C NMR spectra of compound **3kc**

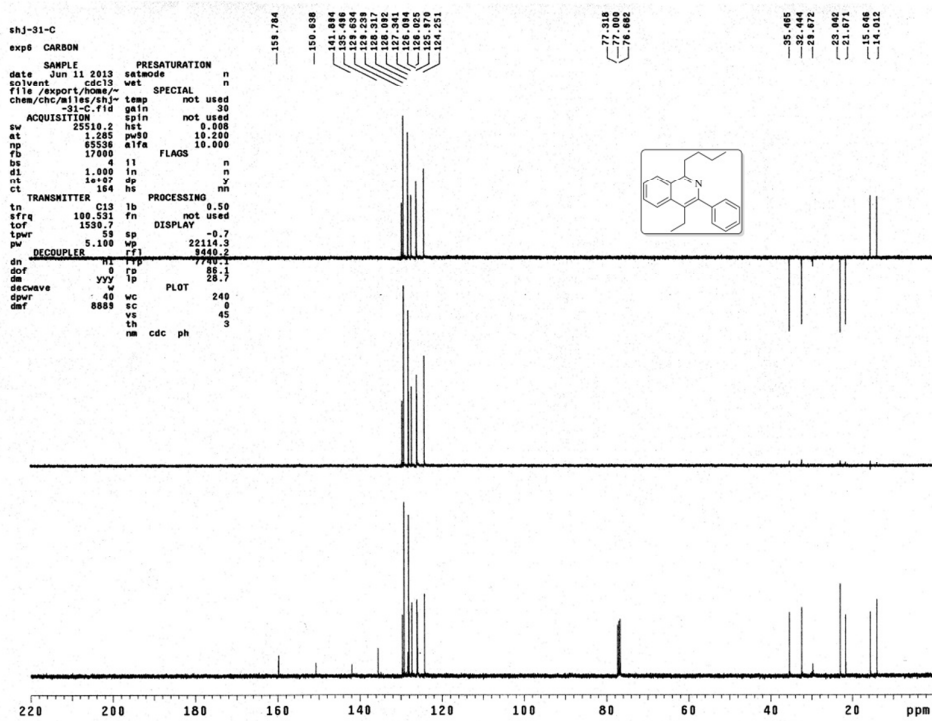
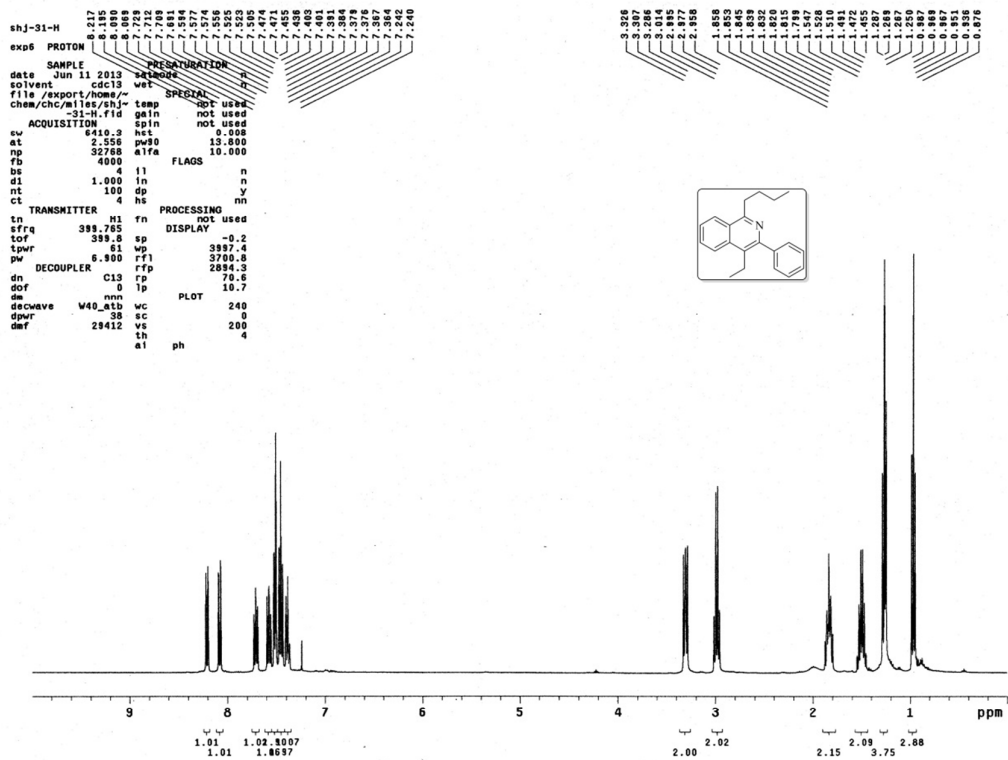


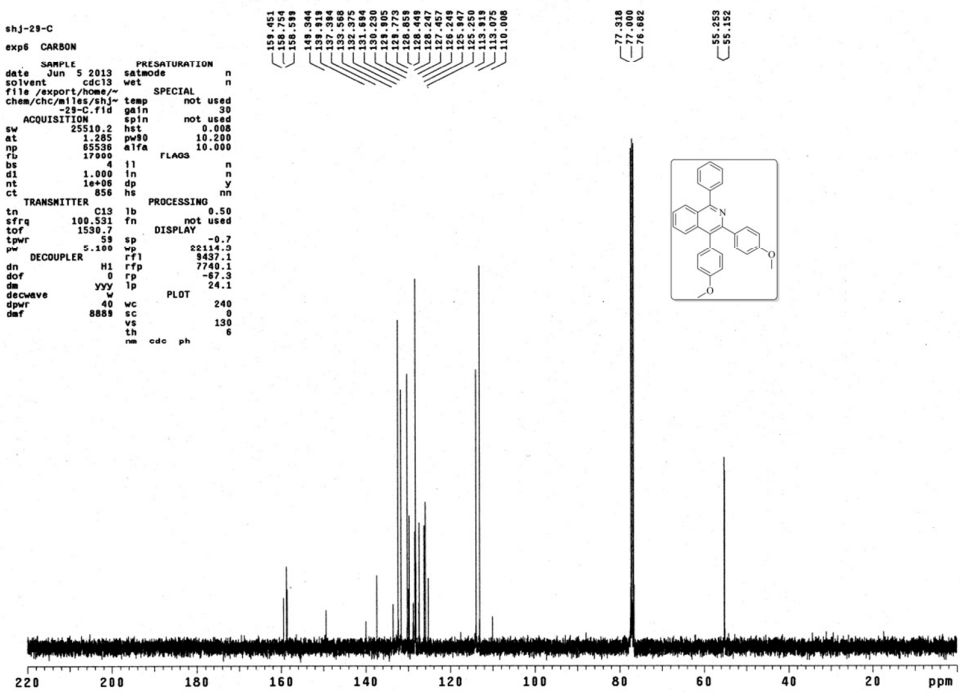
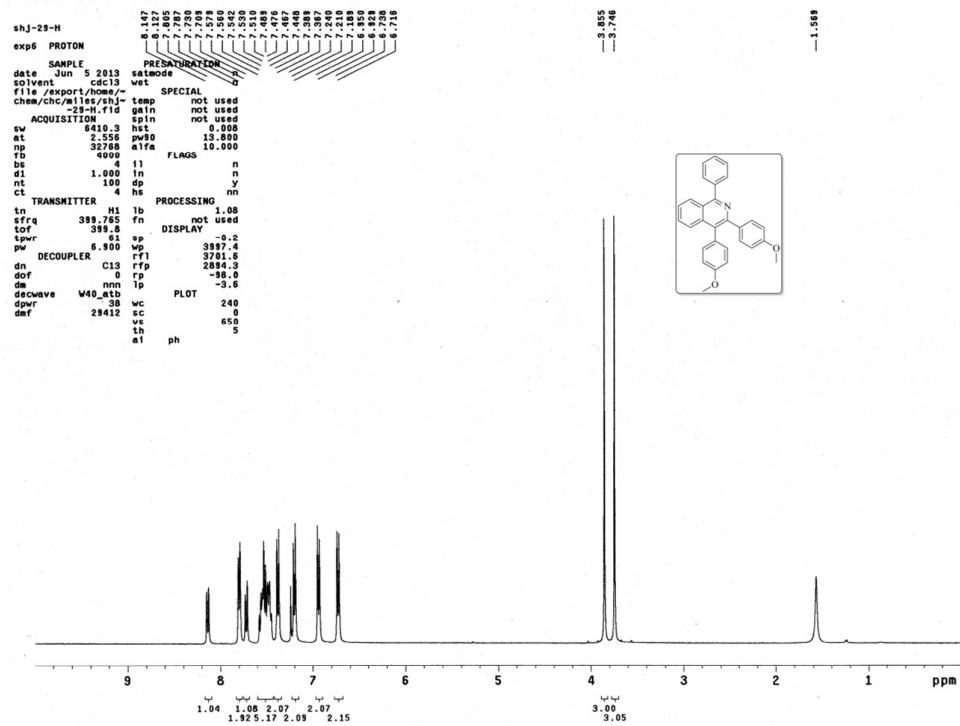
^1H and ^{13}C NMR spectra of compound **3kf**



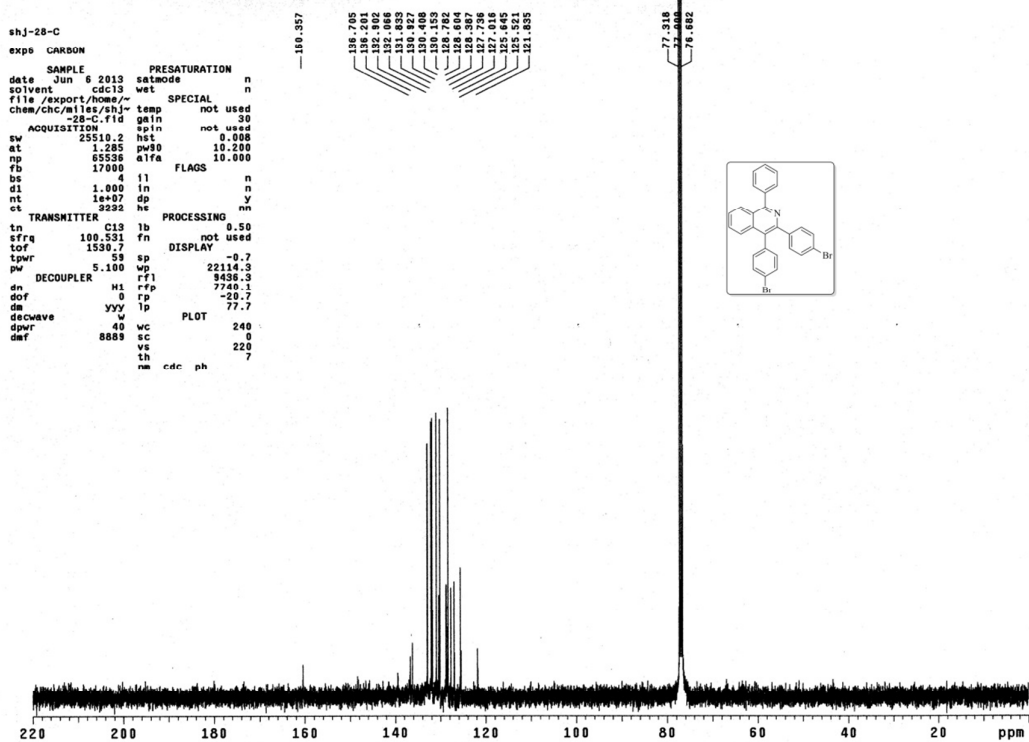
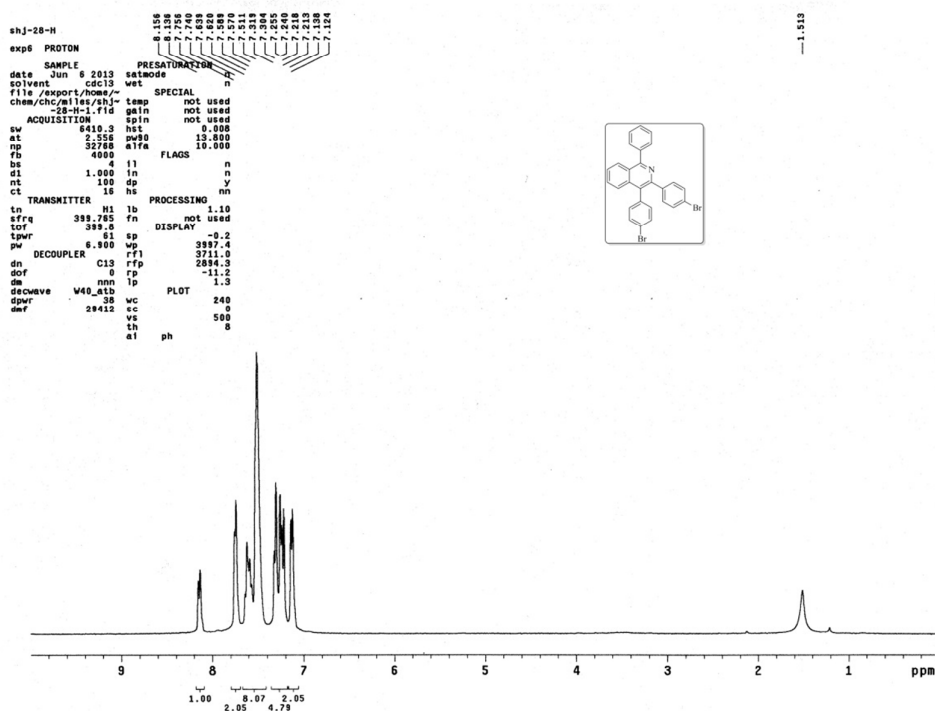
^1H and ^{13}C NMR spectra of compound **3kj**



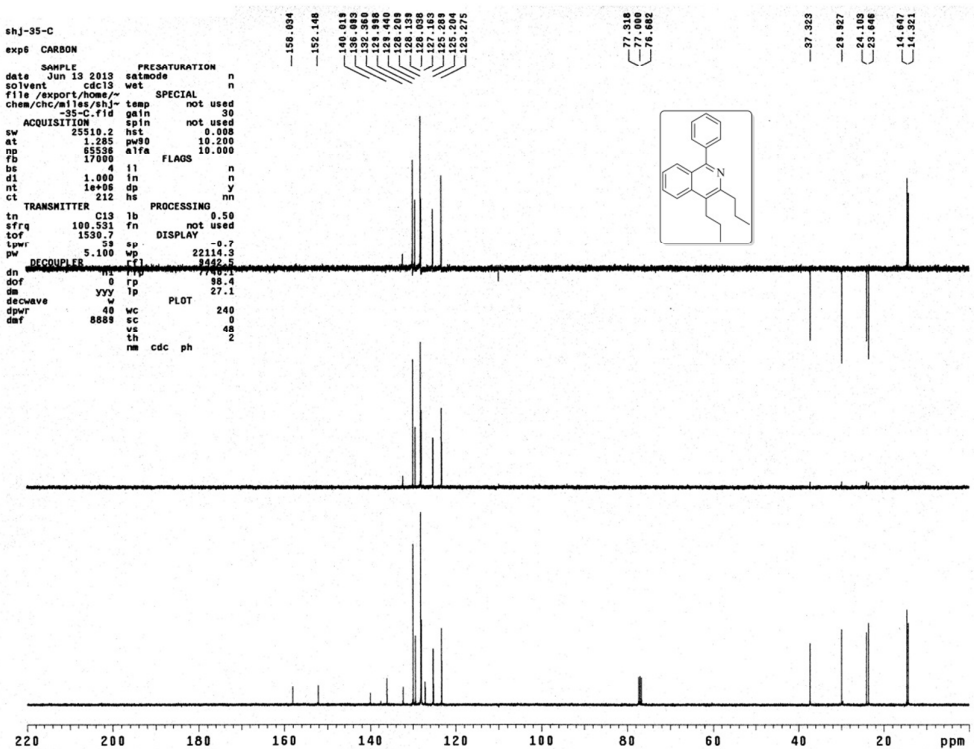
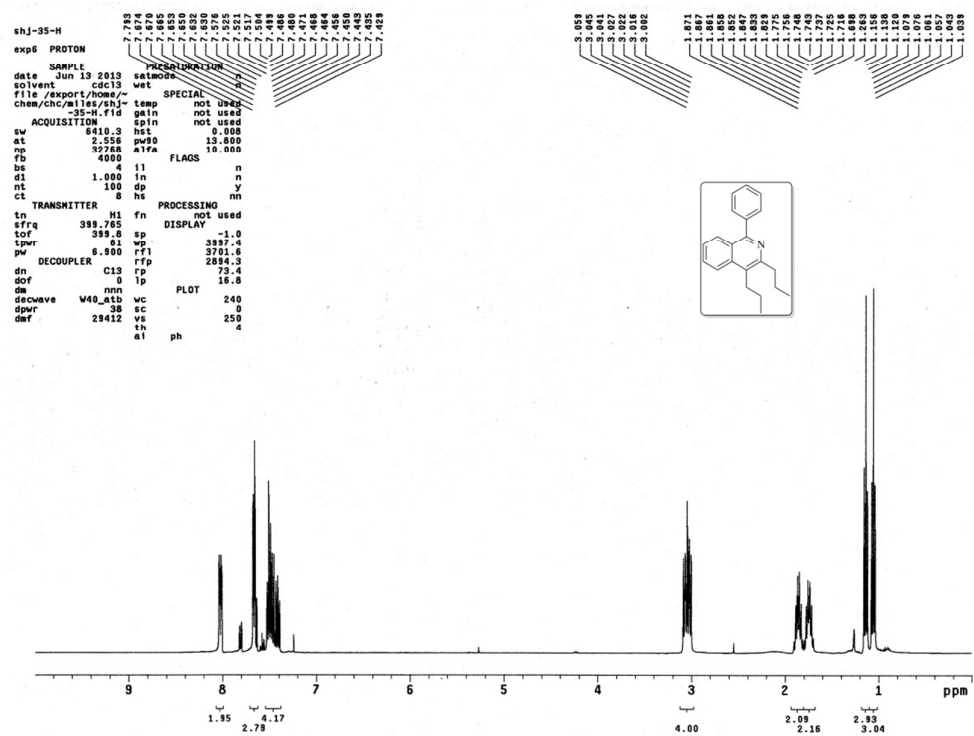
^1H and ^{13}C NMR spectra of compound **3kk**

¹H and ¹³C NMR spectra of compound **3lc**

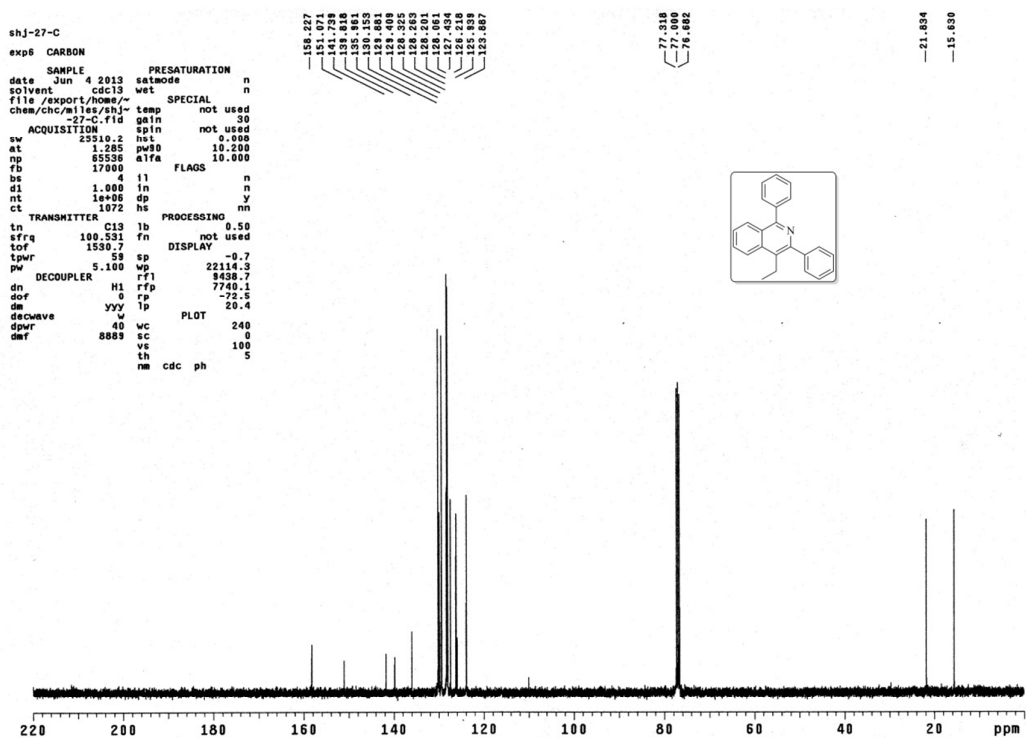
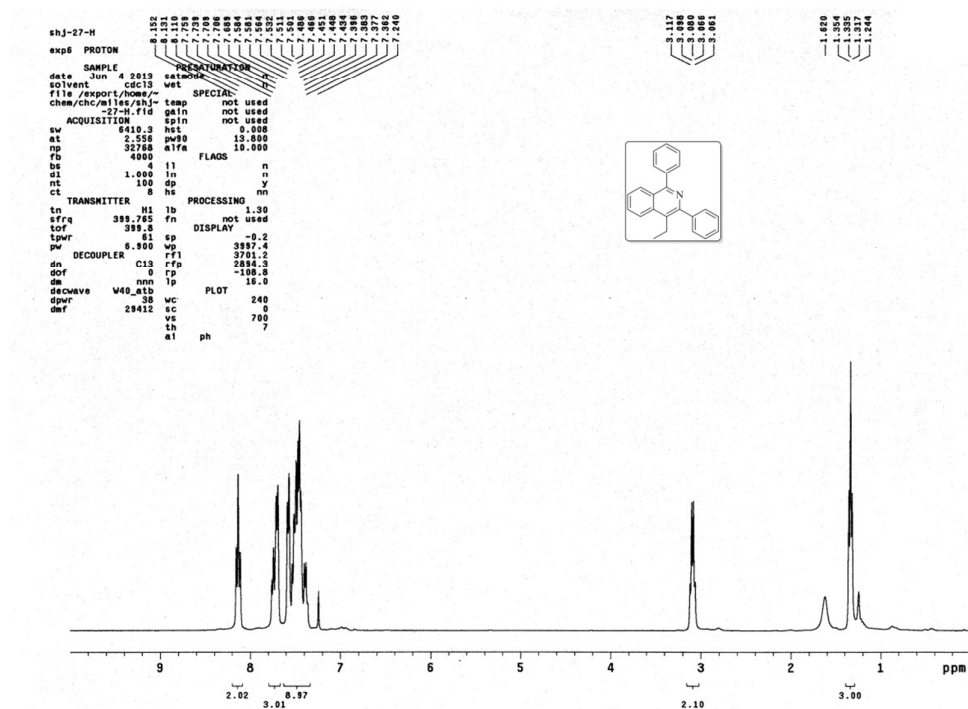
^1H and ^{13}C NMR spectra of compound **3If**



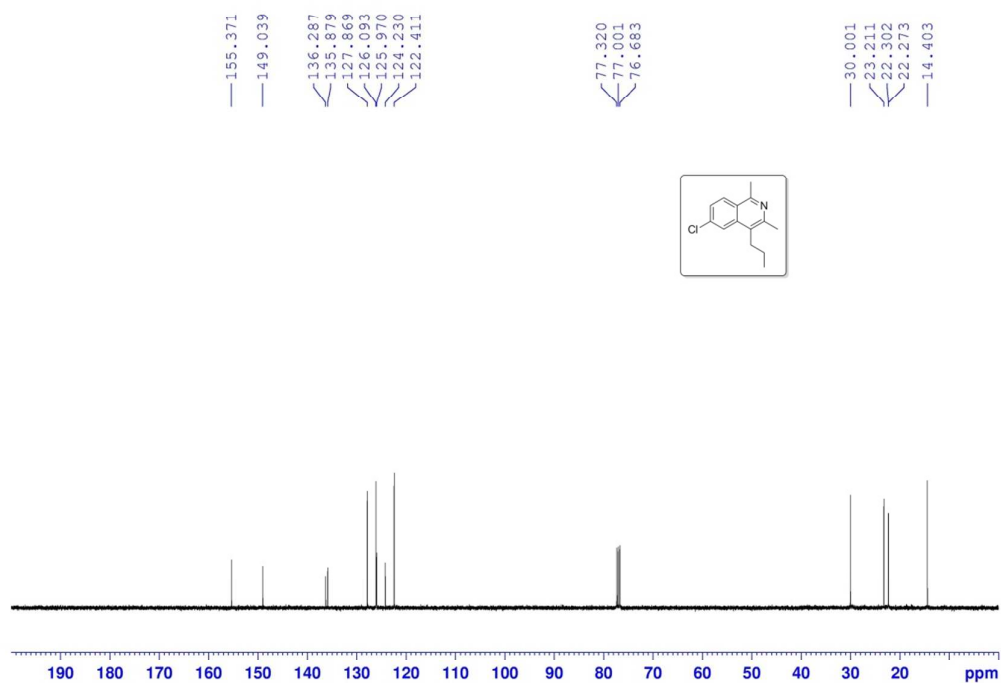
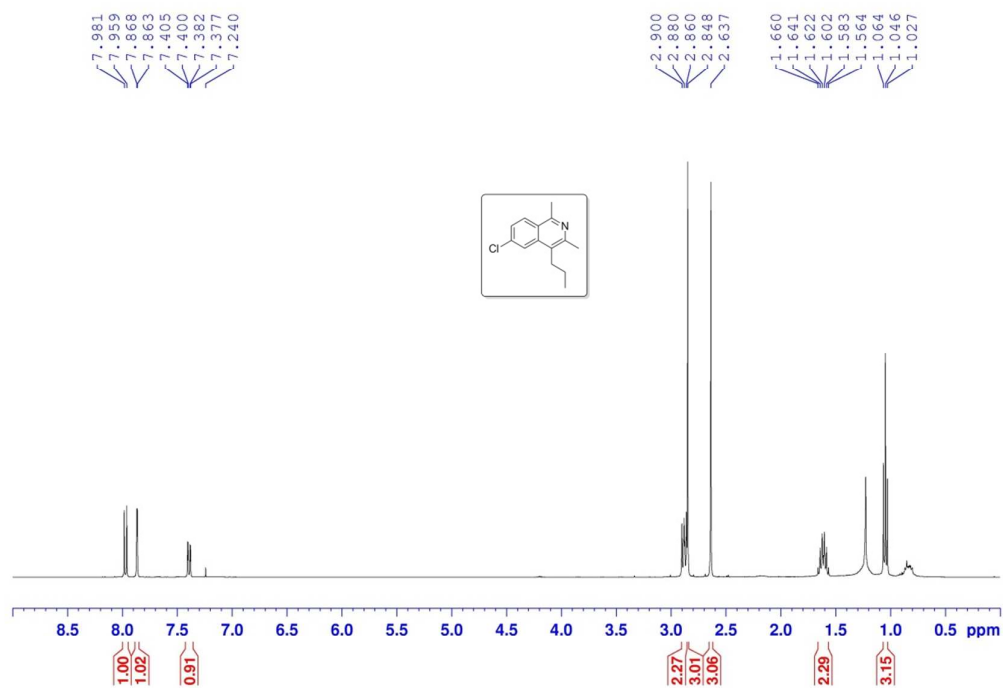
^1H and ^{13}C NMR spectra of compound **3lj**



^1H and ^{13}C NMR spectra of compound **3lk**



^1H and ^{13}C NMR spectra of compound **3em**



^1H and ^{13}C NMR spectra of compound **3em'**

