Palladium catalyzed synthesis of naphthalene derivatives via direct fuse ring construction from amides with alkynes

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Supporting information

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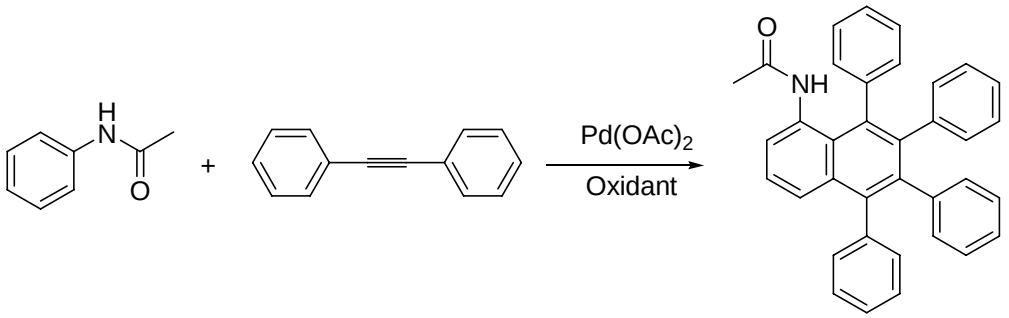
General Information

All reagents were used directly without further purification. Silica gel was purchased from Qing Dao Hai Yang Chemical Industry Co. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer. Mass spectra were obtained on a Waters Q-Tof MicroTM spectrometer. IR spectra were recorded on a Bruker VECTOR22 spectrometer. Fluorescence spectra were recorded on a Jobin Yvon Fluoroma-P fluorescence spectrometer in the solid state, slit width 1nm.

Reaction procedure:

Optimization Study for ring construction of acetanilide (1a) with diphenylacetylene (2a) via C-H bonds cleavage (Table S1). Pd catalyst (1~10 mol %), oxidant (1~2 equiv) and acetanilide (40.5 mg, 0.3mmol, 1 equiv)were weighed into the flask, diphenylacetylene (112.14mg, 0.63mmol, 2.1 equiv) was added followed by solvent (1.5 mL) and additive (0.15 mmol, 0.5 equiv). The resulting mixture was stirred at the indicated temperature for 16 h in an oil bath at air atmosphere. The reaction was cooled to room temperature, filtered through a short Silica gel column washing with EtOAc (50 mL). The filtrate was concentrated, and evaporated to dryness under high vacuum. The yield of desired product was determined by HPLC (C_{18} column, methanol/H_2O=80/20, 1.0mL/min, λ =242nm).

Table S1. Optimization Screen for ring construction of acetanilide (1a) with diphenylacetylene (2a) via C-H bonds cleavage[a]



1a 2a 3a

Entry	Pd catalyst	Solvent	Oxidant	Additive	Temp.(^{o}C)	Yield (%)[b]
1	$Pd(OAC)_2$	DMSO	BQ	TsOH	80	3
2	$Pd(OAC)_2$	CH_3CN	BQ	TsOH	80	15
3	$Pd(OAC)_2$	EtOH	BQ	TsOH	80	58
4	$Pd(OAC)_2$	HOAc	BQ	TsOH	80	58
5	$Pd(OAC)_2$	DMF	BQ	TsOH	80	36
6	$Pd(OAC)_2$	NMP	BQ	TsOH	80	38
7	$Pd(OAC)_2$	Dioxane	BQ	TsOH	80	39

8	$Pd(OAC)_2$	DCE	BQ	TsOH	80	52
9	$Pd(OAC)_2$	THF	BQ	TsOH	80	65
10	$Pd(OAC)_2$	$CHCl_3$	BQ	TsOH	80	71
11	$Pd(OAC)_2$	CH_2Cl_2	BQ	TsOH	80	53
12	$Pd(OAC)_2$	Toluene	BQ	TsOH	80	82(78)
13	-	Toluene	BQ	TsOH	80	No
14	$Pd(OAC)_2$	Toluene/HOAc	BQ	TsOH	80	66
15	$Pd(OAC)_2$	Toluene/H_2O	BQ	TsOH	80	20
16	$Pd(OAC)_2$	Toluene/EtOH	BQ	TsOH	80	68
17	$Pd(OAC)_2$	Toluene	-	TsOH	80	28
18	$Pd(OAC)_2$	Toluene	H_2O_2	TsOH	80	21
19	$Pd(OAC)_2$	Toluene	$Cu(OAc)_2$	TsOH	80	13
20	$Pd(OAC)_2$	Toluene	Ag_2O	TsOH	80	trace
21	$Pd(OAC)_2$	Toluene	Oxone	TsOH	80	80
22	$Pd(OAC)_2$	Toluene	AgI	TsOH	80	trace
23	$Pd(OAC)_2$	Toluene	$K_2S_2O_8$	TsOH	80	83(80)
24	$Pd(OAC)_2$	Toluene	$K_2S_2O_8$	TFA	80	10
25	$Pd(OAC)_2$	Toluene	$K_2S_2O_8$	TfOH	80	71
26	$Pd(OAC)_2$	Toluene	$K_2S_2O_8$	H_2SO_4	80	84
27	$Pd(OAC)_2$	Toluene	$K_2S_2O_8$	TsOH	60	65
28	$Pd(OAC)_2$	Toluene	$K_2S_2O_8$	TsOH	70	79
29	$Pd(OAC)_2$	Toluene	$K_2S_2O_8$	TsOH	100	83
30	$Pd(OAC)_2$[c]	Toluene	$K_2S_2O_8$	TsOH	80	84
31	$PdCl_2$	Toluene	$K_2S_2O_8$	TsOH	80	34
32	$Pd(PPh_3)_2Cl_2$	Toluene	$K_2S_2O_8$	TsOH	80	31
33	$Pd_2(dba)_3$[d]	Toluene	$K_2S_2O_8$	TsOH	80	80
34	$Pd(PPh_3)_4$	Toluene	$K_2S_2O_8$	TsOH	80	74
35	$Pd(CF_3COO)_2$	Toluene	$K_2S_2O_8$	TsOH	80	79

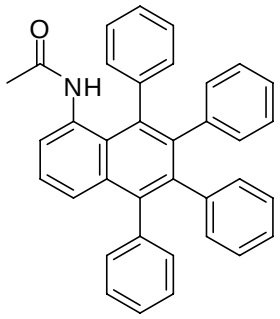
a Reaction conditions: **1a** (0.3 mmol), **2a** (0.63 mmol), Pd-catalyst (5 mol%), additives(0.15mmol), oxidant (0.6mmol), solvent (1.5 mL), 16 h. *b* Yield determined by HPLC analysis and based on the amount of **1a** used. Value in parentheses indicates yield after purification. c 10 mol % of $Pd(OAC)_2$ was used. d 2.5 mol % of $Pd_2(dba)_3$ was used.

General catalytic procedure (Table 2)

$Pd(OAc)_2$ (3.36 mg, 0.015mmol, 5%mol), $K_2S_2O_8$ (162.2mg, 0.60mmol, 2equiv), and anilide (0.3mmol, 1 equiv) were weighed into the flask, alkynes (0.63mmol, 2.1 equiv) was added followed by toluene (1.5 mL) and TsOH (25.8mg, 0.15 mmol, 0.5 equiv). The resulting mixture was stirred at the indicated temperature for 16 h in an oil bath at air atmosphere, then cooled down to room temperature. The mixture was diluted with 30 mL saturated $NaHCO_3$ and extracted with ether (3*20 mL). The combined organic phase was dried over Na_2SO_4. After evaporation of the solvents the residue was purified by silica gel

chromatography or thin layer chromatography (TLC) (elute: hexane-EtOAc).

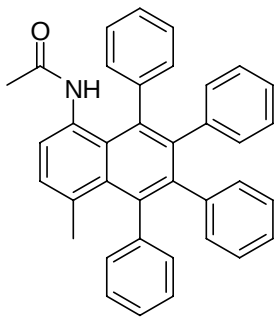
5, 6, 7, 8-tetraphenyl-N-acetyl-1-aminonaphthalene 3a



3a

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) ppm: 7.95 (d, J=7.45Hz, 1H), 7.50 (dd, J_1=7.74Hz, J_1=0.61Hz, 1H), 7.40 (t, J=7.66Hz, 1H), 7.26~7.17 (m, 10H), 6.99(s, 1H, N-H), 6.85~6.73 (m, 10H), 1.40 (s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 168.11, 142.21, 140.95, 140.13, 139.94, 139.66, 139.41, 138.84, 133.77, 132.97, 131.12, 131.08, 131.05, 131.00, 130.99, 130.96, 130.49, 128.21, 127.52, 127.04, 126.52, 126.45, 126.40, 125.78, 125.37, 125.28, 124.32, 123.33, 23.94. HRMS (ESI) Calcd. for $C_{36}H_{27}NO$: $[M+Na]^+$, 512.1990. Found: m/z 512.1987.

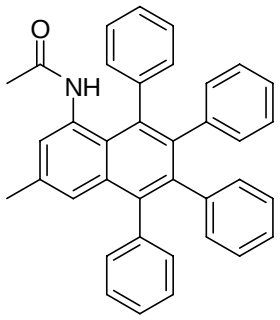
4-mehtyl-5, 6, 7, 8-tetraphenyl-N-acetyl-1-aminonaphthalene 3b



3b

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) ppm: 7.72 (d, J=7.71Hz, 1H), 7.26 (d, J=5.62Hz, 1H), 7.23~7.06 (m, 10H), 6.81~6.78 (m, 6H), 6.74 (s, 1H, N-H), 6.67~6.64 (m, 4H), 1.88 (s, 3H), 1.38 (s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 168.15, 142.65, 142.51, 140.25, 140.07, 139.90, 138.63, 134.75, 134.08, 132.97, 131.56, 131.24, 131.14, 131.07, 130.38, 130.26, 128.15, 126.91, 126.72, 126.54, 126.36, 126.31, 126.19, 125.11, 125.03, 123.88, 25.30, 23.74. HRMS (ESI) Calcd. for $C_{37}H_{29}NO$: $[M+Na]^+$, 526.2147. Found: m/z 526.2148.

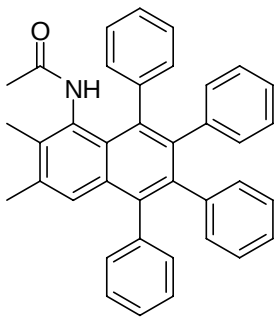
3-mehtyl-5, 6, 7, 8-tetraphenyl-N-acetyl-1-aminonaphthalene 3c



3c

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) ppm: 7.84(d, J=1.05Hz, 1H), 7.28~7.17 (m, 11H), 6.97 (s, 1H, N-H), 6.84~6.73 (m, 10H), 2.38(s, 3H), 1.40 (s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 168.13, 142.27, 140.32, 140.06, 140.02, 139.82, 138.91, 128.72, 135.67, 134.30, 133.89, 132.76, 131.22, 131.18, 130.99, 130.95, 130.50, 128.19, 127.52, 127.00, 126.55, 126.50, 126.45, 126.39, 126.36, 125.31, 125.22, 124.50, 122.62, 24.00, 21.80. IR (KBr): 3426, 3055, 3025, 2922, 2851, 1671, 1622, 1542, 1494, 1465, 1441, 1404, 1369, 1279, 734, 701cm^{-1}. HRMS (ESI) Calcd. for $C_{37}H_{29}NO$: $[M+Na]^+$, 526.2147. Found: m/z 526.2146.

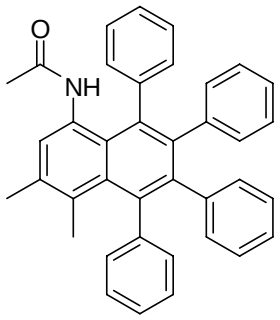
2, 3-dimehtyl-5, 6, 7, 8-tetraphenyl-N-acetyl-1-aminonaphthalene 3d



3d

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) ppm: 7.42 (s, 1H), 7.25~7.11 (m, 10H), 6.78~6.75 (m, 10H), 6.39 (s, 1H, N-H), 2.32(s, 3H), 2.17 (s, 3H), 1.38 (s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 168.50, 143.60, 140.37, 140.29, 140.23, 138.17, 138.12, 135.71, 135.53, 134.48, 131.70, 131.26, 131.22, 131.16, 131.10, 131.06, 131.03, 130.92, 130.89, 130.76, 129.58, 128.15, 128.10, 128.04, 127.29, 127.09, 126.51, 126.25, 126.18, 126.13, 126.06, 125.96, 125.92, 125.01, 124.83, 22.86, 21.27, 15.42. HRMS (ESI) Calcd. for $C_{38}H_{31}NO$: $[M+Na]^+$, 540.2303. Found: m/z 540.2302.

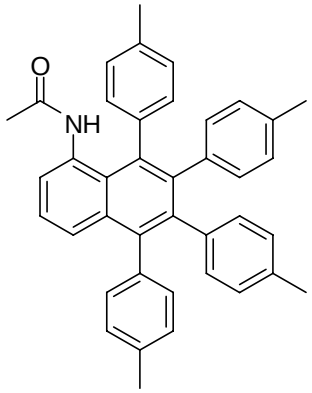
3, 4-dimehtyl-5, 6, 7, 8-tetraphenyl-N-acetyl-1-aminonaphthalene 3e



3e

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$)ppm: 7.18~7.08 (m, 11H), 6.78~6.64 (m, 10H), 6.21 (s, 1H, N-H), 2.23 (s, 3H), 1.87 (s, 3H), 1.39 (s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 168.19, 143.89, 142.46, 140.31, 140.28, 140.22, 139.19, 138.25, 135.54, 135.12, 134.94, 133.32, 131.52, 131.41, 131.05, 130.99, 130.93, 129.80, 128.33, 127.76, 126.69, 126.13, 126.07, 126.02, 126.00, 125.97, 125.78, 124.76, 25.16, 22.87, 18.67. HRMS (ESI) Calcd. for $C_{38}H_{31}NO$: $[M+Na]^+$, 540.2303. Found: m/z 540.2304.

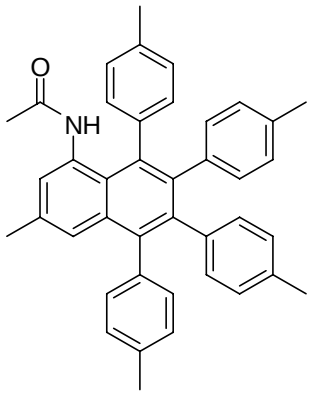
5, 6, 7, 8-tetra (4-methylphenyl)-N-acetyl-1-aminonaphthalene 3f



3f

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$)ppm: 7.95 (d, J=7.21Hz, 1H), 7.45 (d, J=8.35Hz, 1H), 7.34 (t, J=7.81Hz, 1H), 7.12~7.03 (m, 9H), 6.64~6.59 (m, 8H), 2.30 (s, 3H), 2.29 (s, 3H), 2.07 (s, 6H), 1.40 (s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 167.98,141.16, 139.31, 139.24, 139.05, 137.34, 137.09, 136.88, 136.47, 135.73, 134.36, 134.31, 134.29, 133.94, 132.97, 131.14, 130.96, 130.91, 130.75, 130.38, 128.82, 128.20, 127.20, 127.08, 125.41, 125.20, 124.32, 122.61, 23.91, 21.21, 21.09, 21.02. HRMS (ESI) Calcd. for $C_{40}H_{35}NO$: $[M+Na]^+$568.2616. Found: m/z 568.2617.

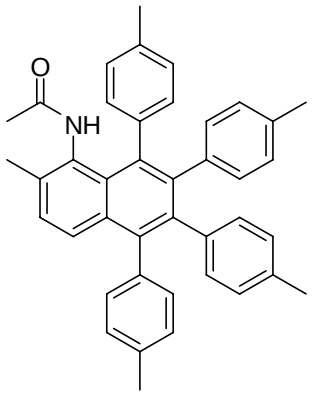
3-methyl-5, 6, 7, 8-tetra (4-methylphenyl)-N-acetyl-1-aminonaphthalene 3g



3g

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$)ppm: 7.84(d, J=1.27Hz, 1H), 7.24(s, 1H), 7.11~7.03(m, 9H), 6.64~6.61(m, 8H), 2.36(s, 3H), 2.31(s, 3H), 2.29(s, 3H), 2.07(s, 6H), 1.40(s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 167.92, 140.21, 139.30, 139.11, 138.65, 137.53, 137.21, 137.01, 136.39, 135.59, 135.21, 134.25, , 134.21, 134.15, 134.07, 132.79, 131.01, 130.79, 130.40, 129.48, 128.79, 128.19, 127.21, 127.17, 127.06, 127.01, 126.89, 126.34, 124.50, 124.28, 122.58, 23.95, 21.77, 21.25, 21.10, 21.07, 20.99. HRMS (ESI) Calcd. for $C_{41}H_{37}NO$: $[M+Na]^+$582.2773. Found: m/z 582.2773.

2-methyl-5, 6, 7, 8-tetra (4-methylphenyl)-N-acetyl-1-aminonaphthalene 3h

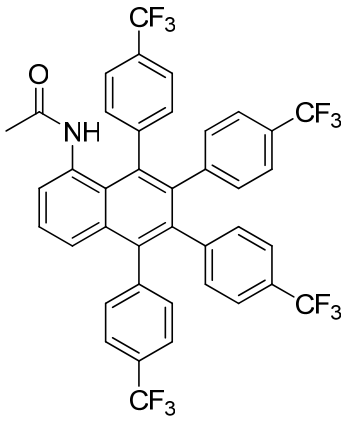


3h

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$)ppm: 7.51(d, J=8.73Hz, 1H), 7.25(d, J=7.61Hz, 1H), 7.05~7.03(m, 8H), 6.63~6.58(m, 8H), 6.35(s, 1H, N-H), 2.30(s, 3H), 2.28(s, 3H), 2.25(s, 3H), 2.07(s, 6H), 1.41(s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 168.14, 141.57, 140.76, 138.95, 138.43, 137.46, 137.37, 136.89, 135.60, 135.41, 135.36, 135.64, 134.19, 134.06, 132.58, 130.98, 130.96, 130.91, 130.80, 129.80, 128.69, 128.67, 128.64, 128.56, 128.20, 128.12, 127.20, 127.11, 126.91, 22.98, 21.21,

21.07, 21.03, 21.01, 19.23. IR (KBr): 3417, 3023, 2923, 2861, 1668, 1511, 1374, 1260, 1108, 1029, 811, 740cm^{-1}. HRMS (ESI) Calcd. for $C_{41}H_{37}NO$: $[M+Na]^+$582.2773. Found: m/z 582.2774.

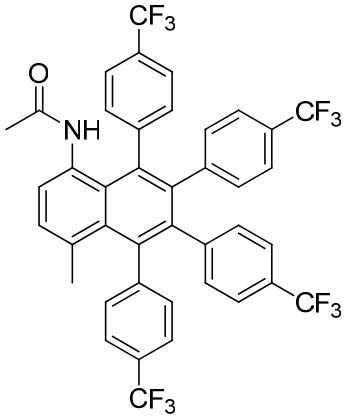
5, 6, 7, 8-tetra (4-trifluoromethylphenyl)-N-acetyl-1-aminonaphthalene 3i



3i

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$)ppm: 7.78 (d, J=6.88Hz, 1H), 7.55~7.46(m, 6H), 7.35~7.30(m, 4H), 7.15 (t, J=7.24Hz, 4H), 6.89(d, J=7.92Hz, 2H), 6.84(d, J=7.96Hz, 2H), 6.49(s, 1H, N-H), 1.39(s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 168.33, 145.42, 142.72, 142.66, 142.45, 139.05, 138.74, 137.06, 134.26, 133.61, 132.62, 131.19, 131.13, 130.94, 130.49, 129.68, 129.65, 129.35, 129.33, 128.68, 128.63, 128.35, 128.31, 127.06, 126.91, 126.16, 125.92, 125.26, 125.02~124.90(m), 124.17~124.06 (p, J=3.6Hz), 124.00~123.89 (p, J=3.7Hz), 122.55, 122.32, 122.29, 23.26. HRMS (ESI) Calcd. for $C_{40}H_{23}F_{12}NO$: $[M+Na]^+$784.1486. Found: m/z 784.1486.

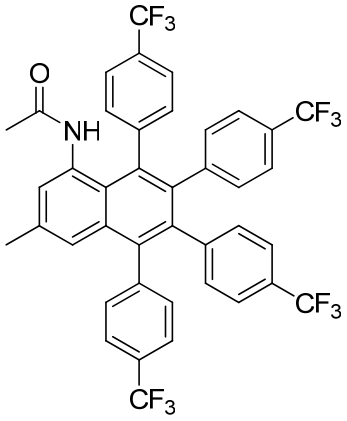
4-methyl-5, 6, 7, 8-tetra (4-trifluoromethylphenyl)-N-acetyl-1-aminonaphthalene 3j



3j

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$)ppm: 7.57(d, J=7.66Hz, 1H), 7.47(d, J=8.11Hz, 2H), 7.40(d, J=8.09Hz, 2H), 7.35(d, J=7.72Hz, 1H), 7.26(m, 2H), 7.20(d, J=8.01Hz, 2H), 7.11 (d, J=7.13Hz, 4H), 6.76(d, J=7.24Hz, 4H), 6.37(s, 1H, N-H), 1.89(s, 3H), 1.37(s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 168.28, 145.85, 145.47, 142.99, 142.84, 138.16, 137.94, 134.92, 134.65, 132.80, 131.53, 131.45, 131.14, 131.10, 131.03, 130.38, 129.34, 129.01, 128.68, 128.49, 128.42, 128.17, 128.10, 127.96, 127.77, 126.99, 125.24, 125.06~124.89 (m), 124.31~124.21 (q, J=36.), 123.95~123.81 (p, J=3.8), 122.53, 122.36, 122.32, 25.61, 23.11. IR (KBr): 3445, 3042, 3021, 2931, 2855, 1664, 1616, 1522, 1405, 1328, 1169, 1126, 1069, 1020, 844, 723cm^{-1}. HRMS (ESI) Calcd. for $C_{41}H_{25}F_{12}NO$: $[M+Na]^{+}$798.1642. Found: m/z 798.1642.

3-methyl-5, 6, 7, 8-tetra (4-trifluoromethylphenyl)-N-acetyl-1-aminonaphthalene 3k

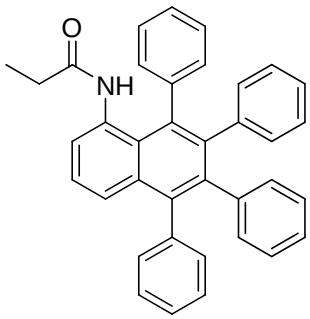


3k

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$)ppm: 7.62(d, J=1.17Hz, 1H), 7.53(m,

4H), 7.33(d, J=7.98Hz, 2H), 7.28(d, J=8.03Hz, 2H), 7.21(s, 1H), 7.13(m, 4H), 6.87(d, J=8.10Hz, 2H), 6.82(d, J=8.00Hz, 2H), 6.41(s, 1H, N-H), 2.41(s, 3H), 1.39(s, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 168.37, 145.54, 142.92, 142.78, 142.63, 138.07, 137.30, 137.12, 134.10, 133.75, 132.34, 131.25, 130.98, 130.50, 129.64, 129.31, 128.95, 125.28, 125.04~124.96(m), 124.12~124.08(q, J=4.0Hz), 123.96~123.93(q, J=3.8Hz), 122.34, 23.34, 21.70. HRMS (ESI) Calcd. for $C_{41}H_{25}F_{12}NO$: $[M+Na]^+$798.1642. Found: m/z 798.1641.

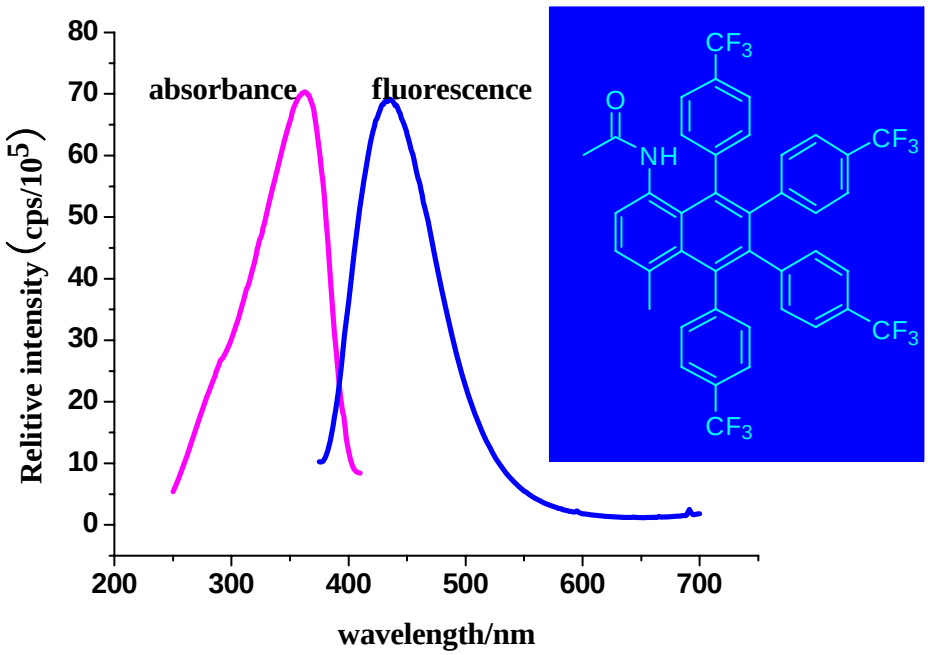
5, 6, 7, 8-tetraphenyl-N-propionyl-1-aminonaphthalene 3l



3l

Pale yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$)ppm: 8.01(d, J=7.45Hz, 1H), 7.48(d, J=8.29Hz, 1H), 7.39(t, J=8.30Hz, 1H), 7.25~7.16(m, 10H), 7.01(s, 1H, N-H), 6.84~6.72(m, 10H), 1.48(q, J=7.56Hz, 2H), 0.91(t, J=7.54Hz, 3H). ^{13}C NMR (100MHz, $CDCl_3$): 171.73, 142.20, 140.89, 14.017, 139.98, 139.71, 139.41, 138.80, 134.40, 133.79, 133.18, 131.15, 131.13, 130.97, 130.55, 128.18, 127.52, 127.04, 126.52, 126.40, 125.82, 125.37, 125.26, 125.07, 124.24, 122.94, 29.64, 9.38. HRMS (ESI) Calcd. for $C_{37}H_{29}NO$: $[M+Na]^+$, 526.2147. Found: m/z 526.2146.

Normalized absorption (purple) and fluorescence (blue) spectra of **3j** in the solid state



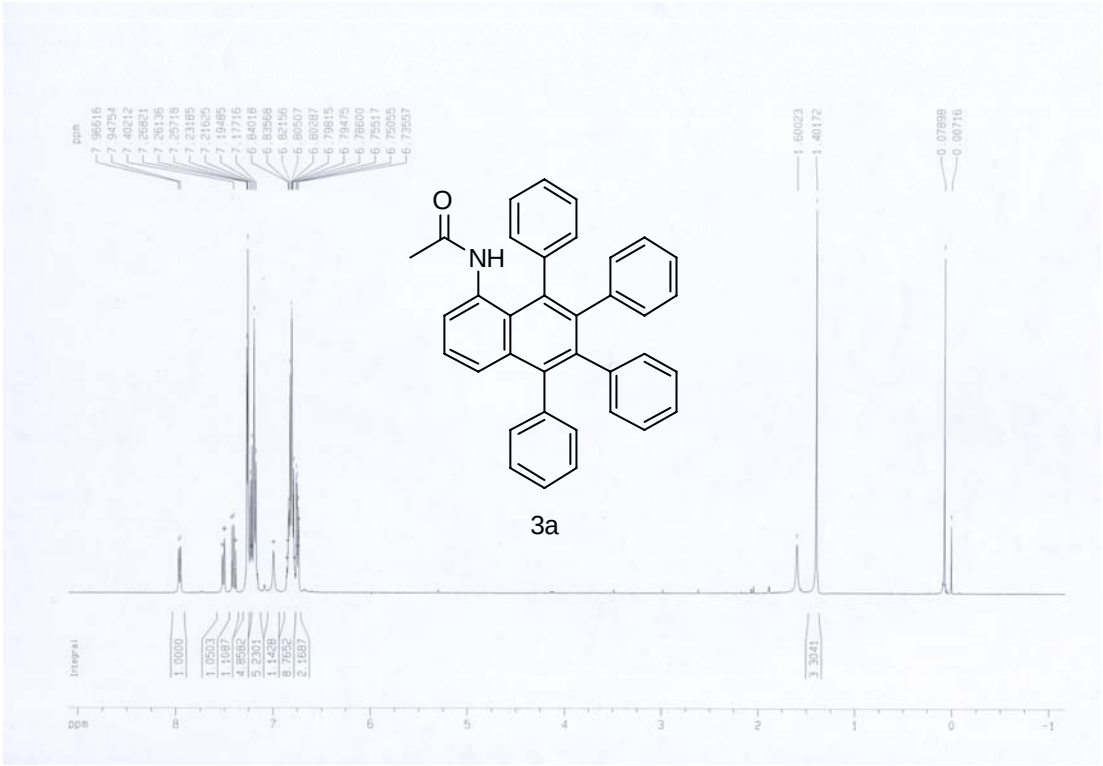


Fig. 1 ^{1}H NMR spectrum of compound **3a**

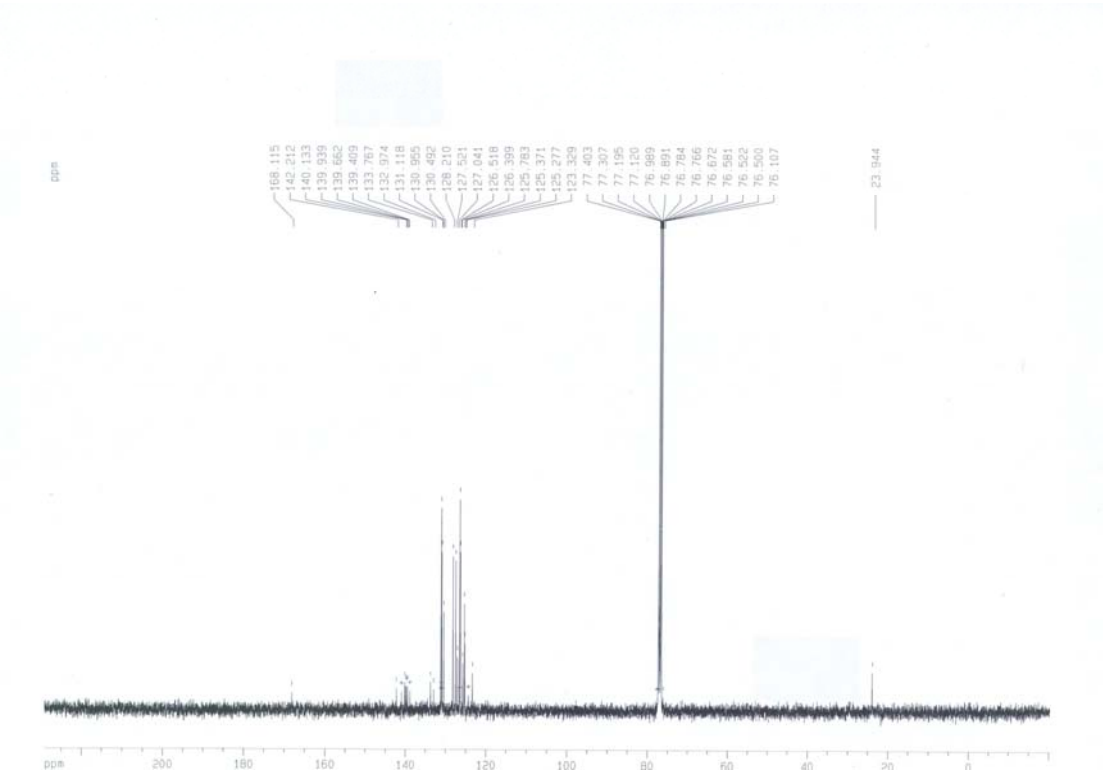


Fig. 2 ^{13}C NMR spectrum of compound **3a**

Fig. 3 ^{1}H NMR spectrum of compound **3b**

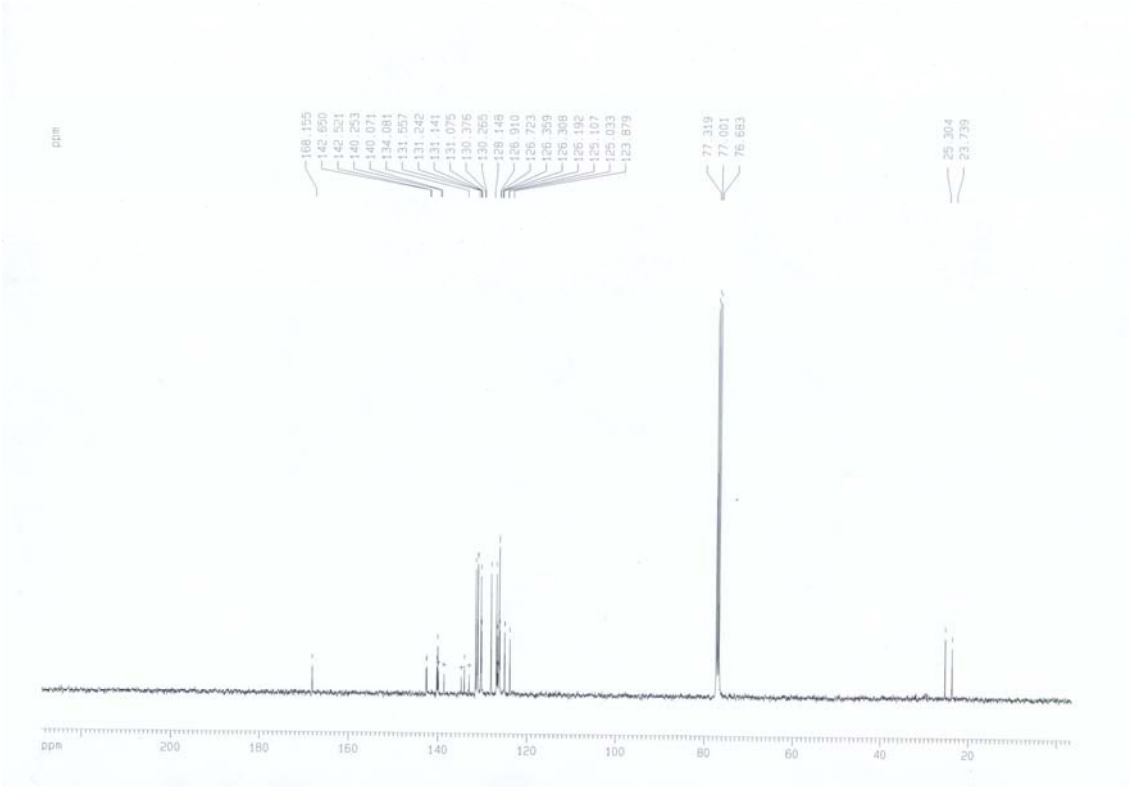


Fig. 4 ^{13}C NMR spectrum of compound **3b**

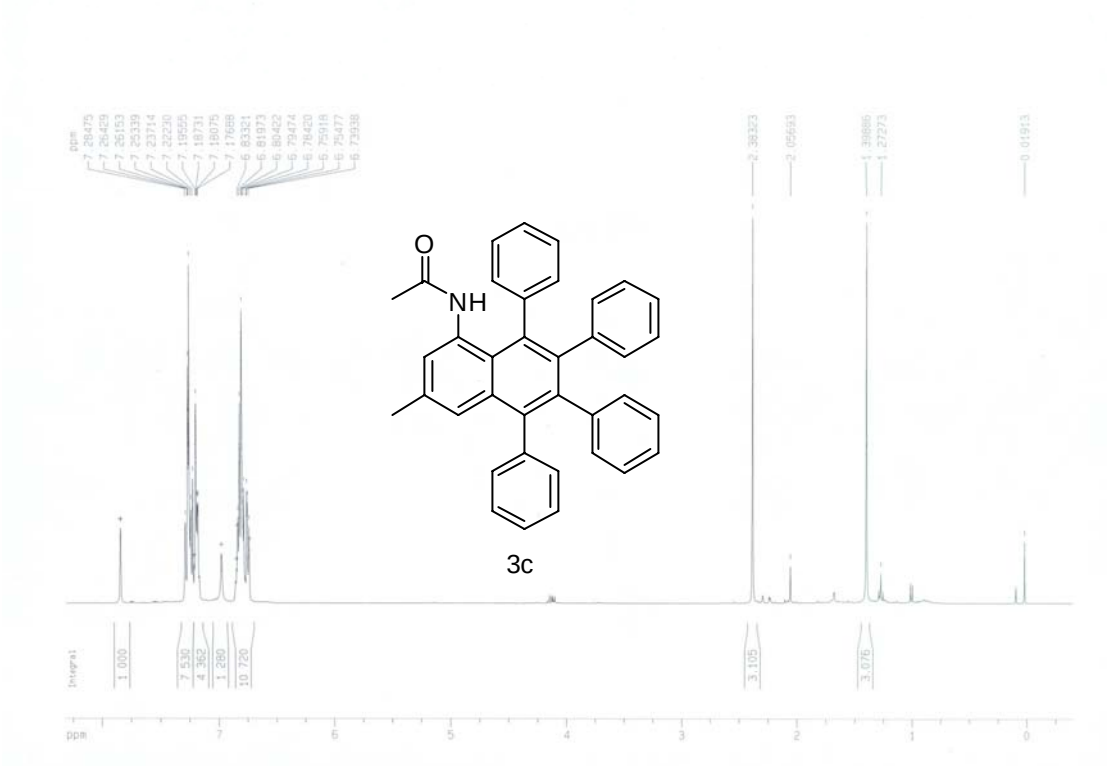


Fig. 5 ^{1}H NMR spectrum of compound **3c**

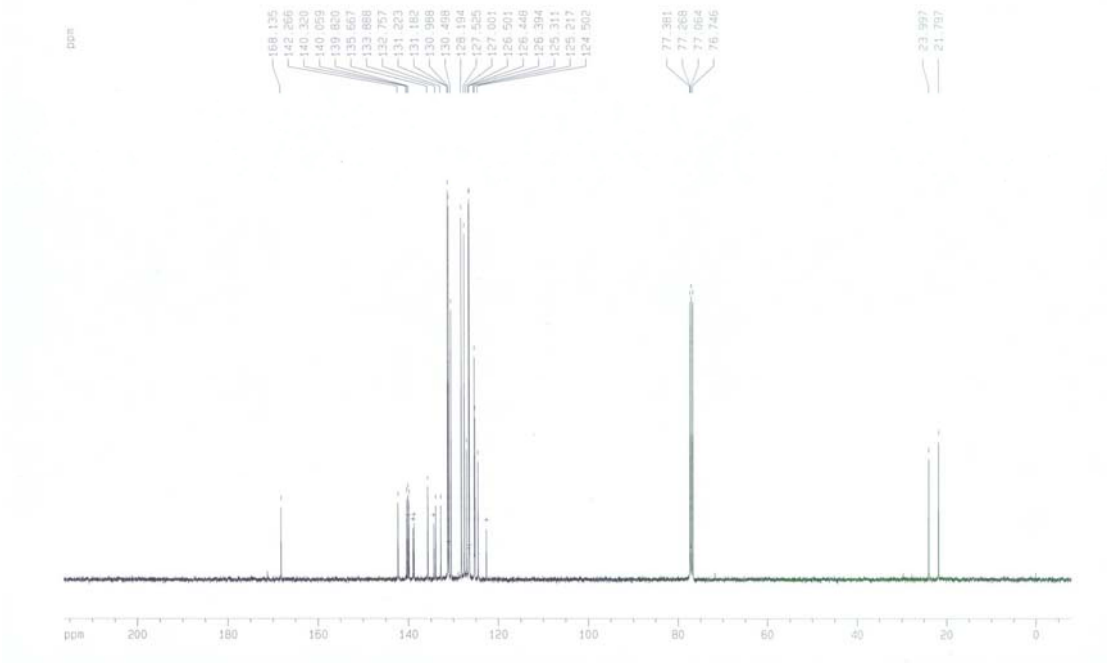


Fig. 6 ^{13}C NMR spectrum of compound **3c**

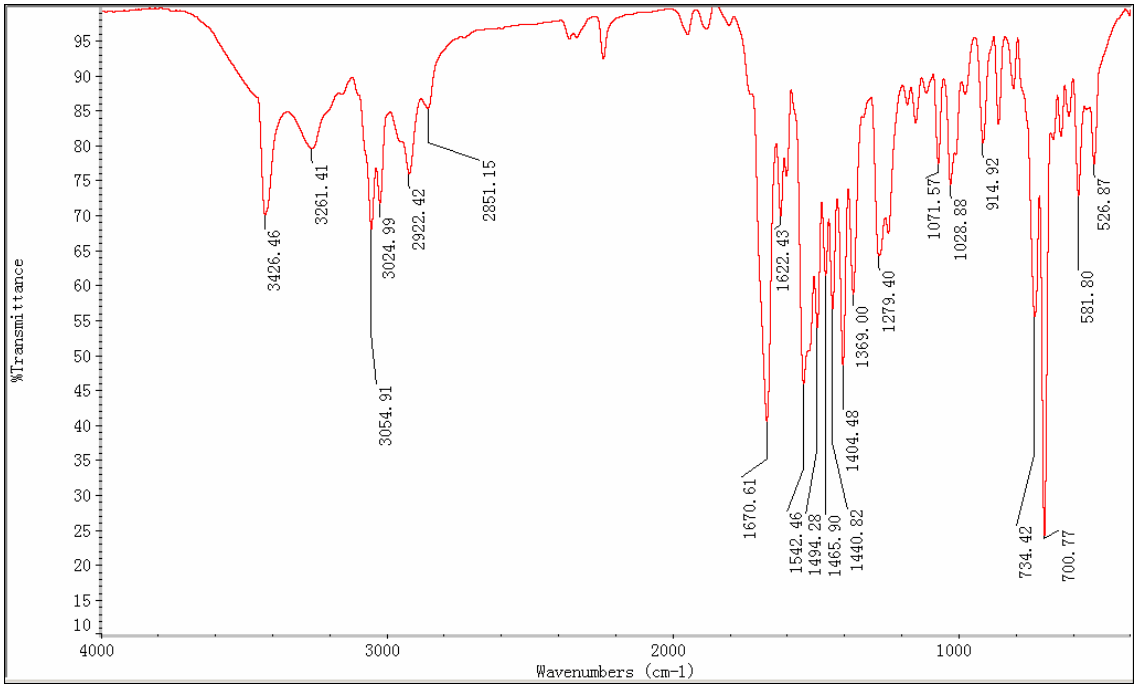


Fig. 7 IR spectrum of compound **3c**

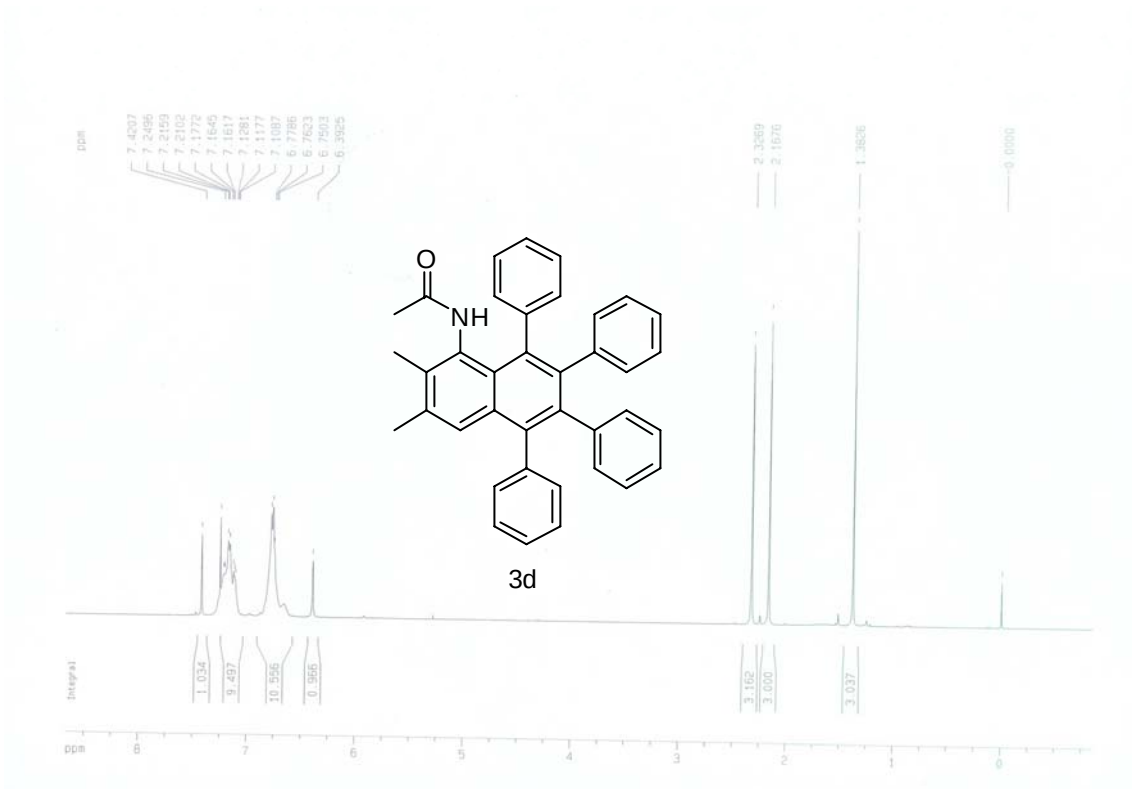


Fig. 8 ^{1}H NMR spectrum of compound **3d**

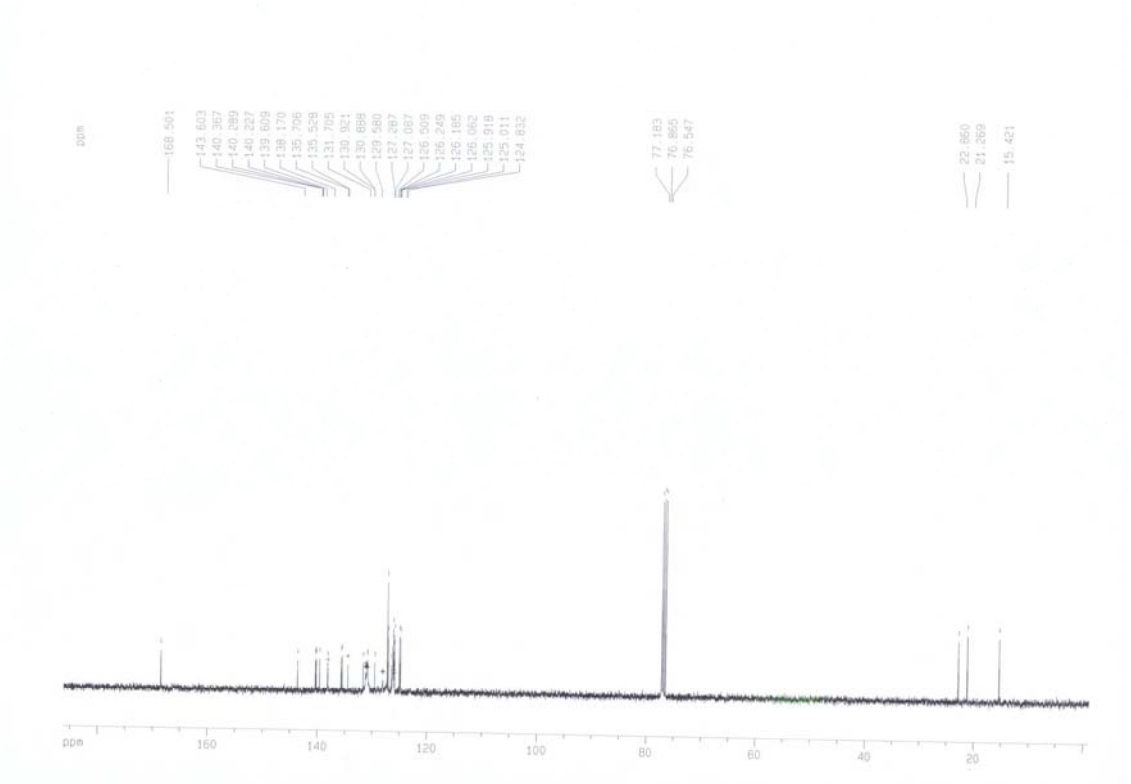
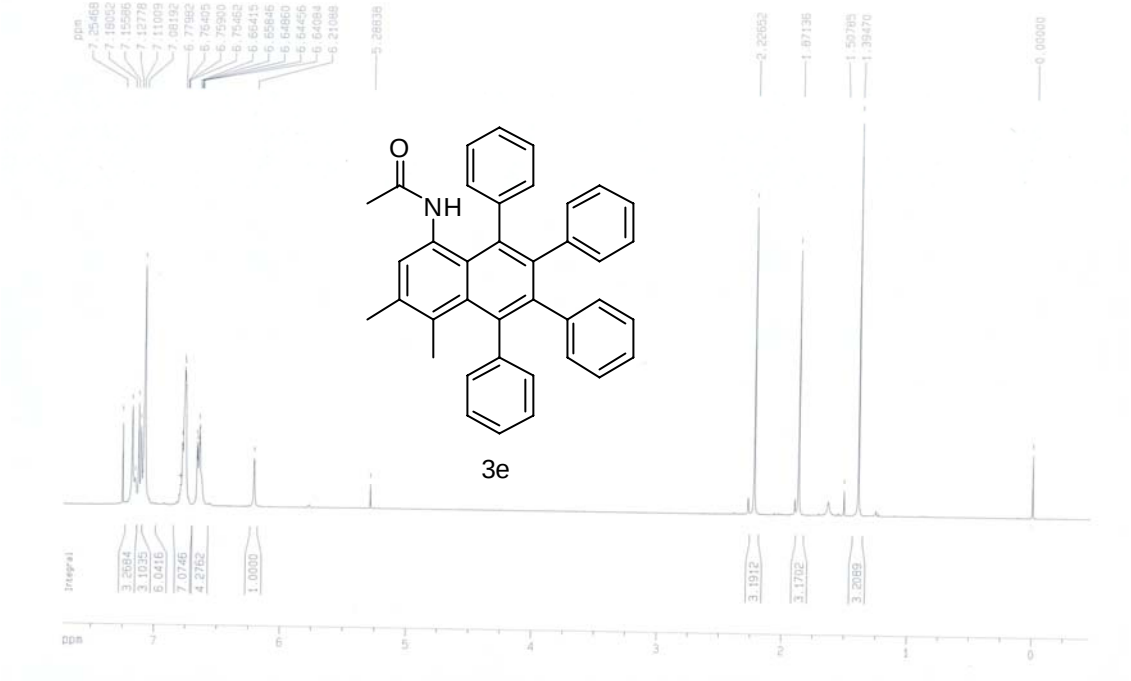


Fig. 9 ^{13}C NMR spectrum of compound **3d**



3e

Fig. 10 ^{1}H NMR spectrum of compound **3e**

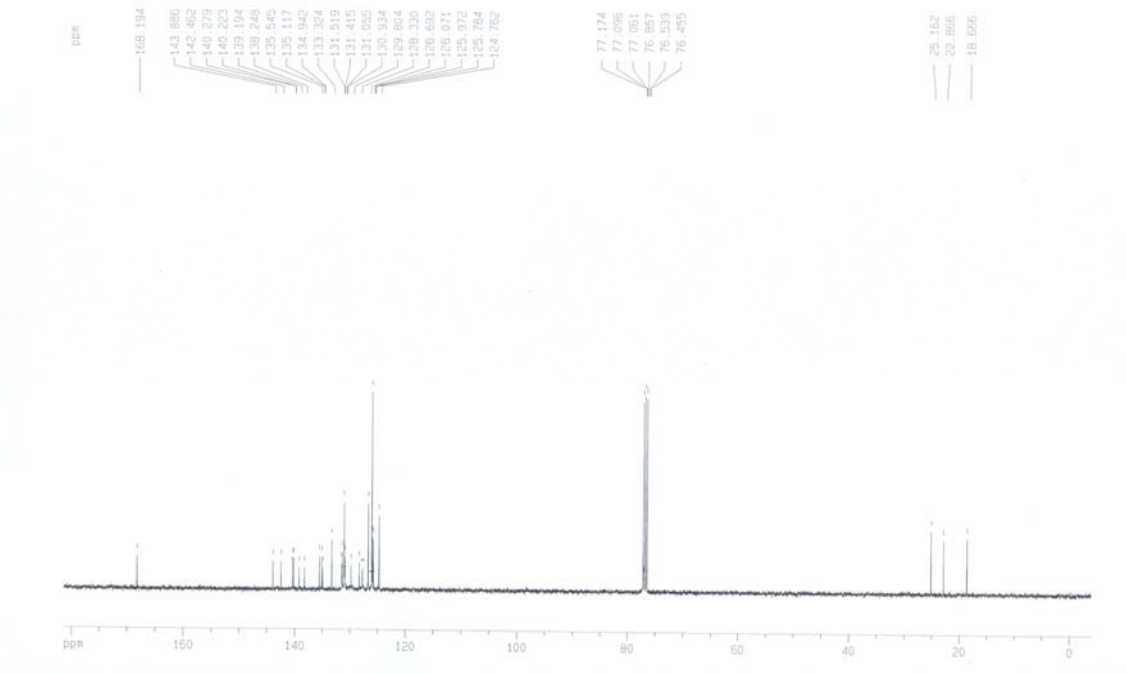


Fig. 11 ^{13}C NMR spectrum of compound **3e**

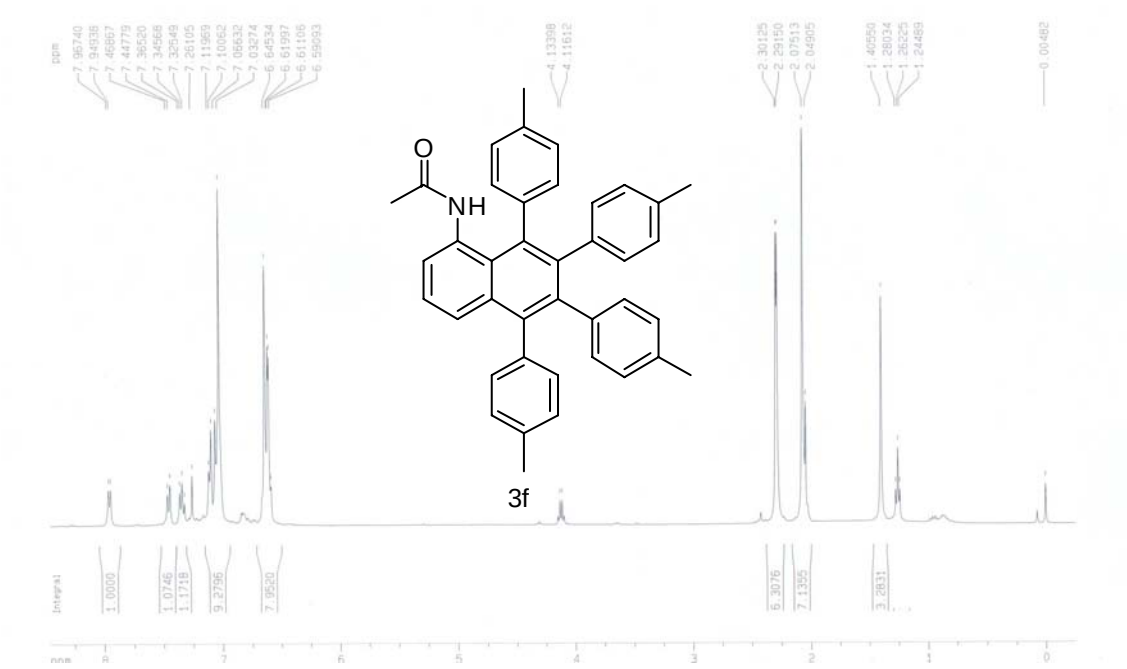


Fig. 12 ^{1}H NMR spectrum of compound **3f**

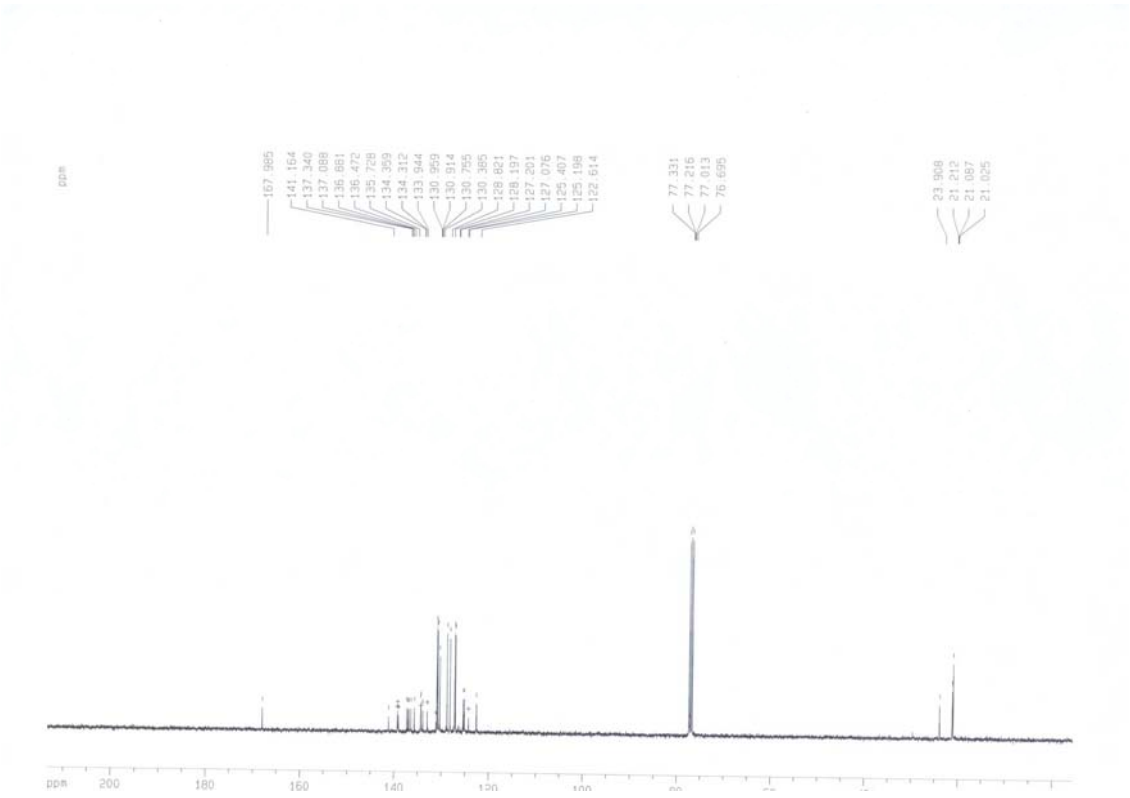
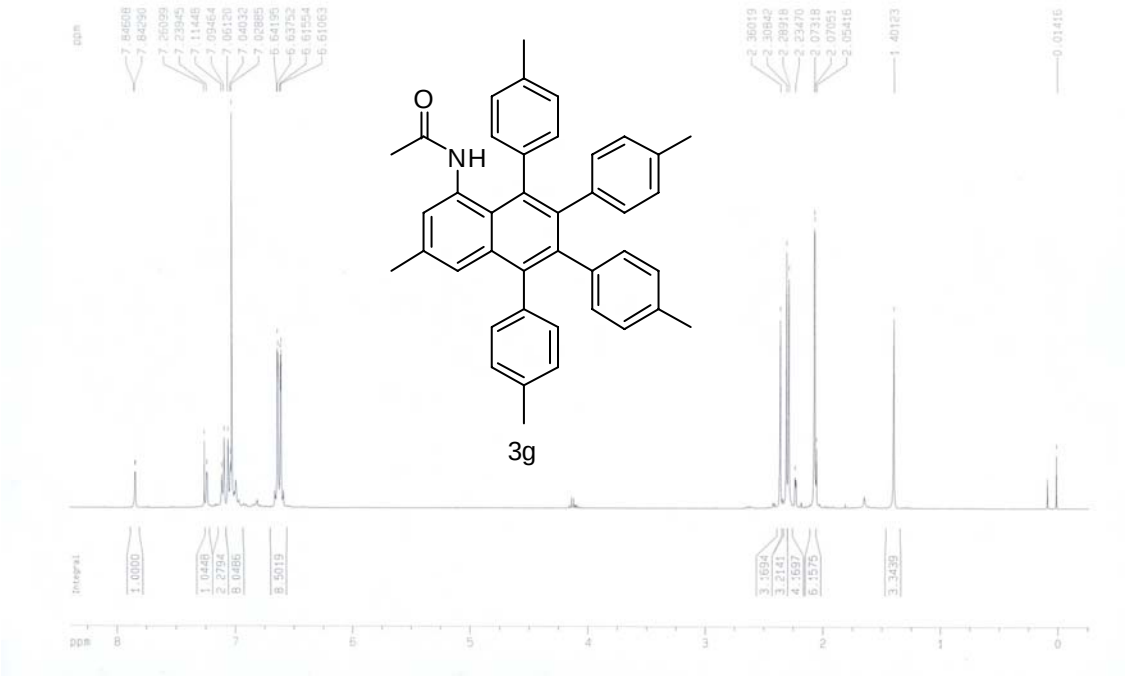


Fig. 13 ^{13}C NMR spectrum of compound **3f**



3g

Fig. 14 ^{1}H NMR spectrum of compound **3g**

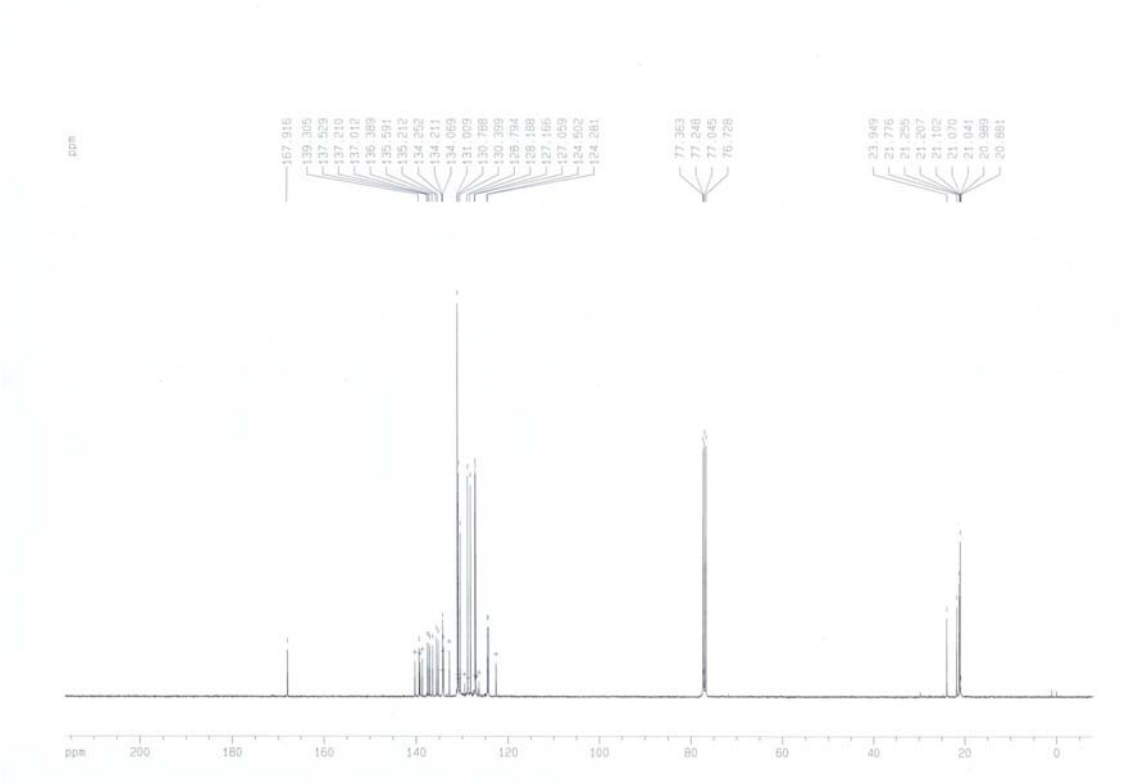


Fig. 15 ^{13}C NMR spectrum of compound **3g**

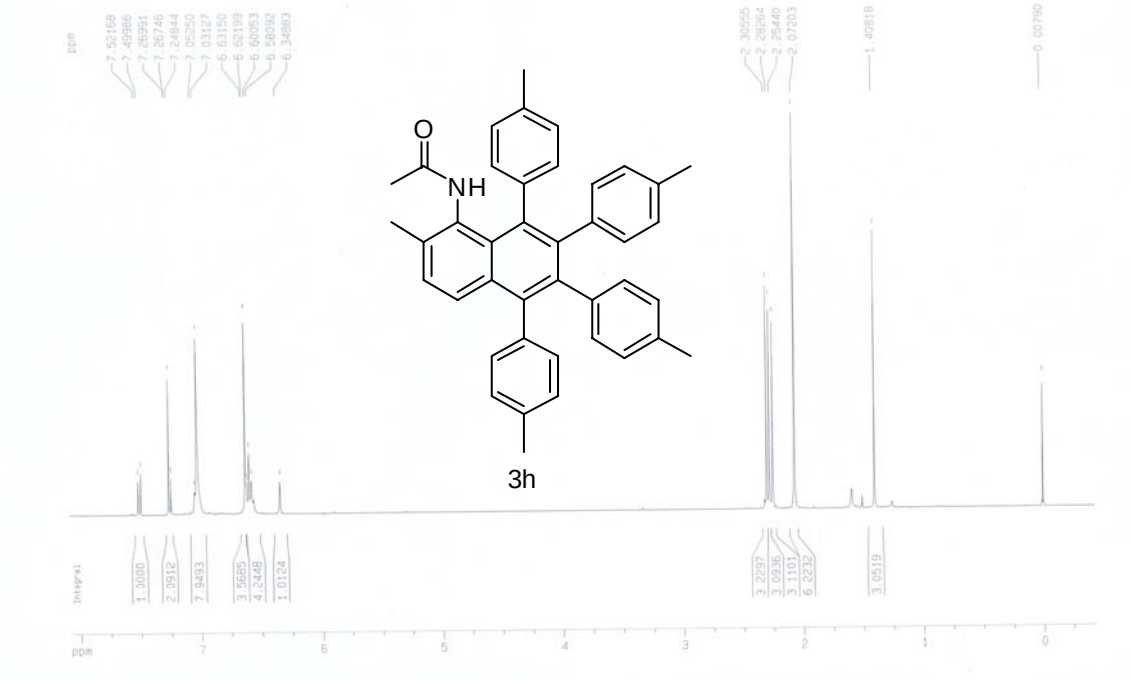


Fig. 16 ^{1}H NMR spectrum of compound **3h**

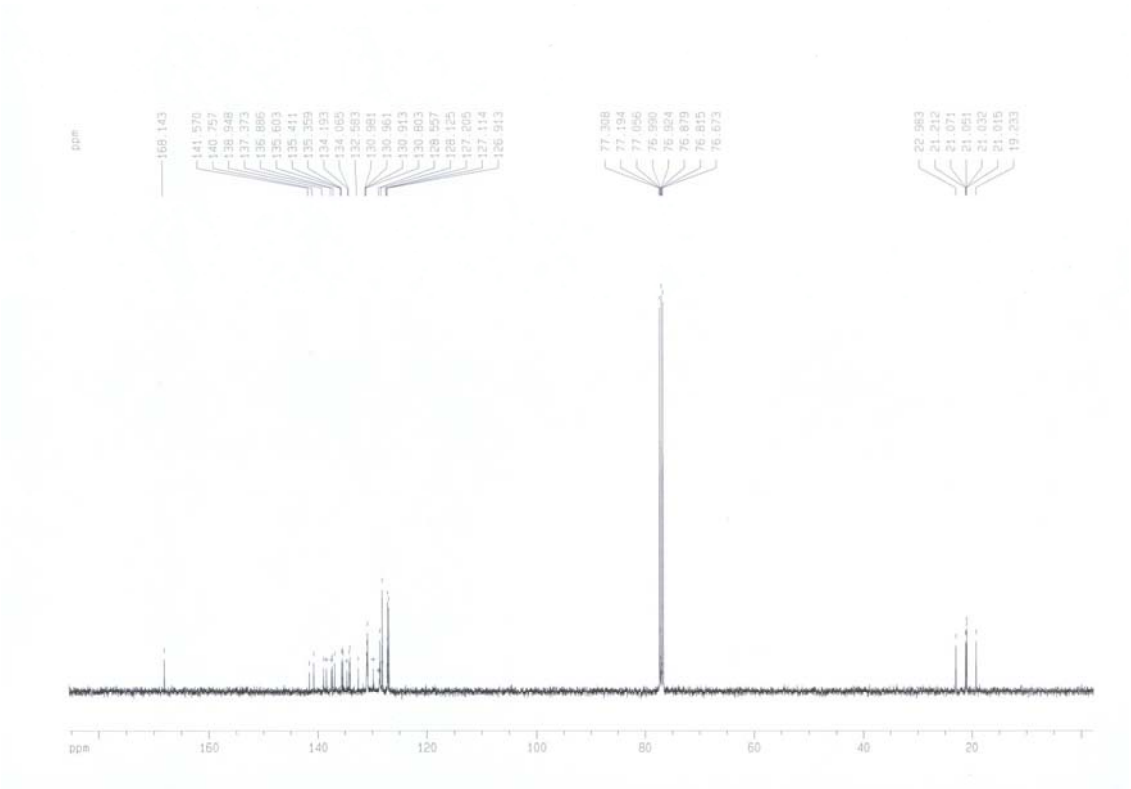


Fig. 17 ^{13}C NMR spectrum of compound **3h**

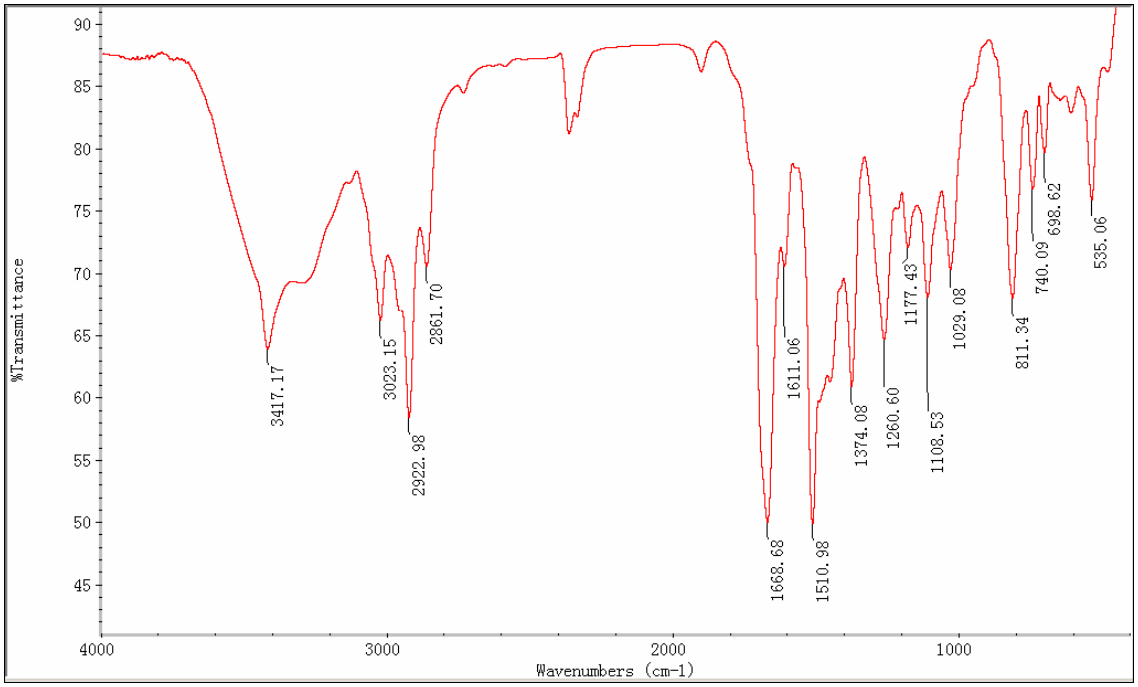


Fig. 18 IR NMR spectrum of compound **3h**

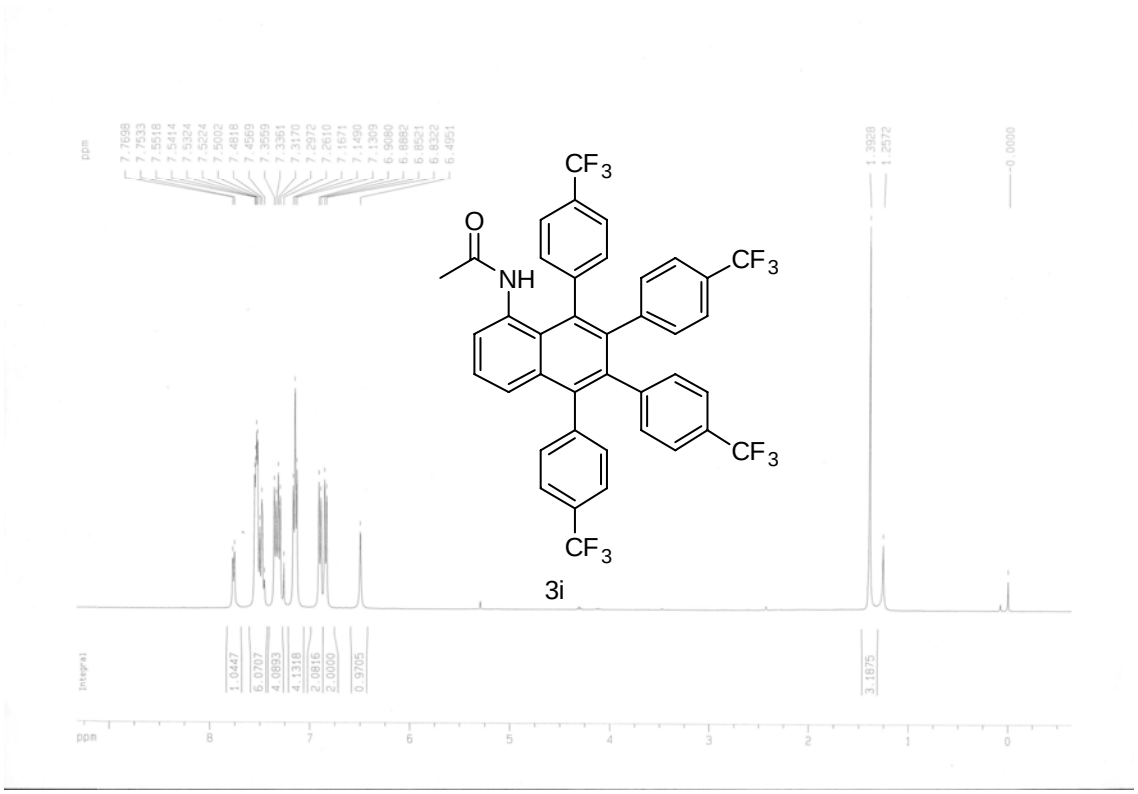


Fig. 19 ^{1}H NMR spectrum of compound **3i**

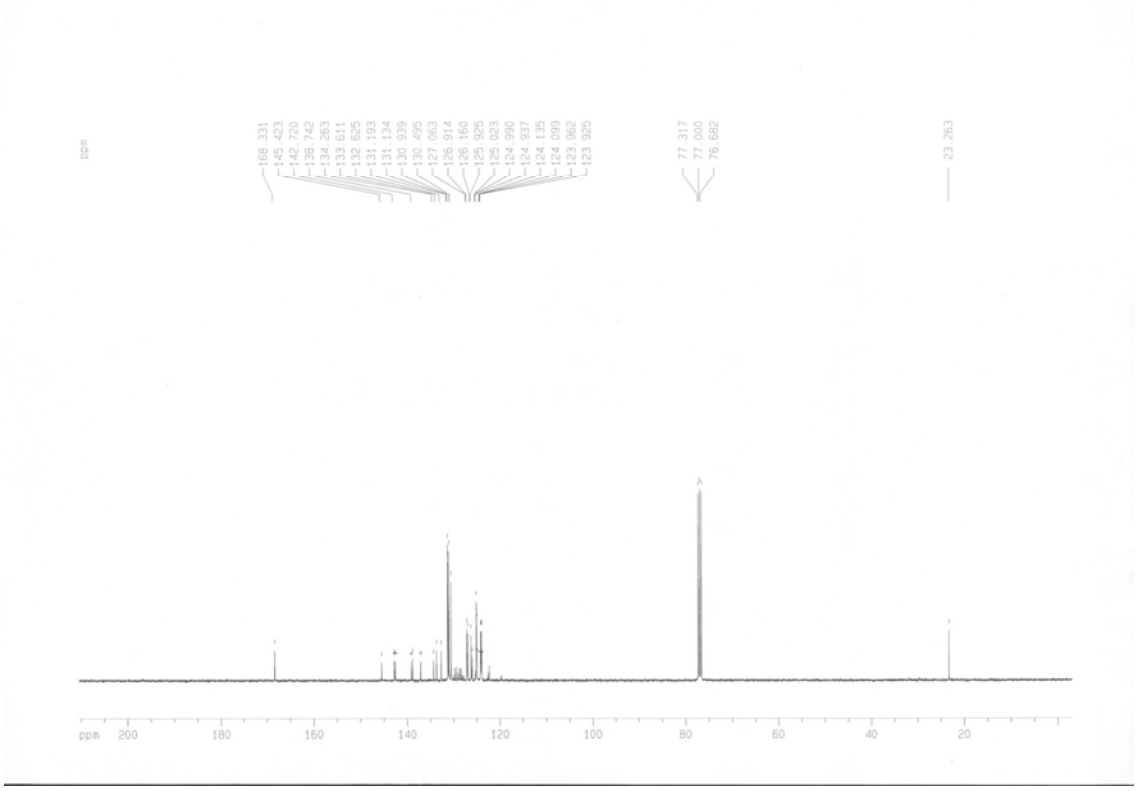


Fig. 23-a ^{13}C NMR spectrum of compound **3i**

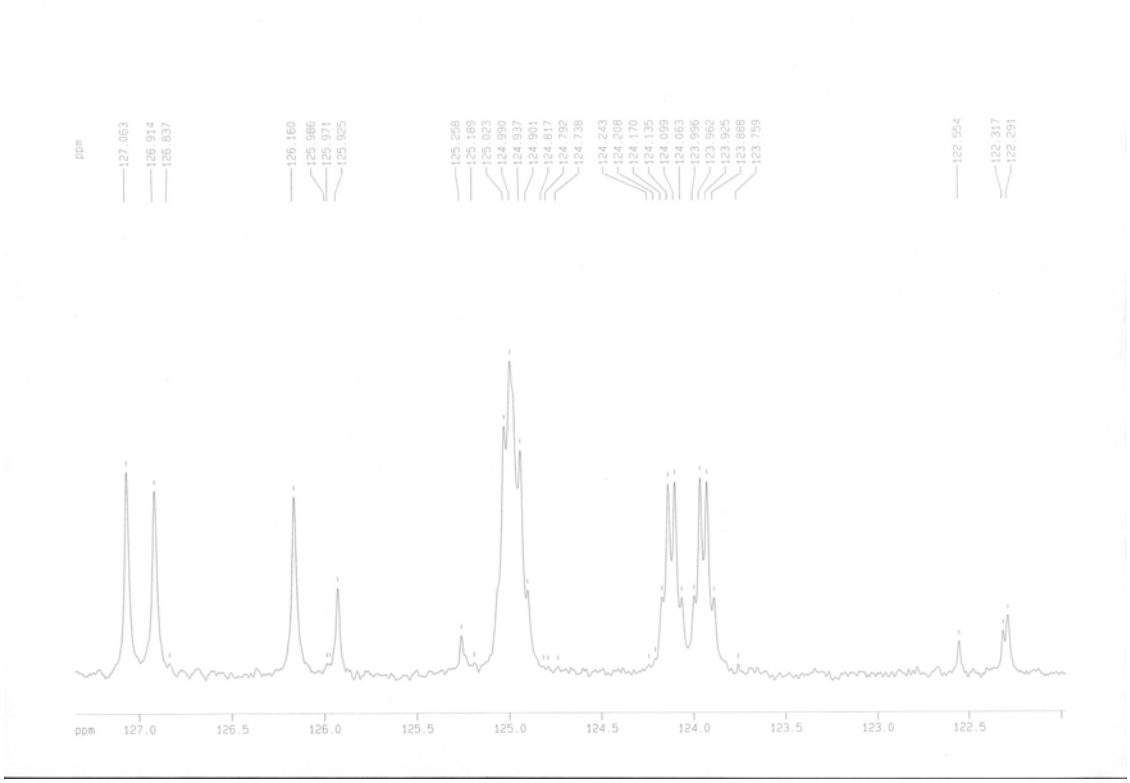


Fig. 23-b ^{13}C NMR spectrum of compound **3i**

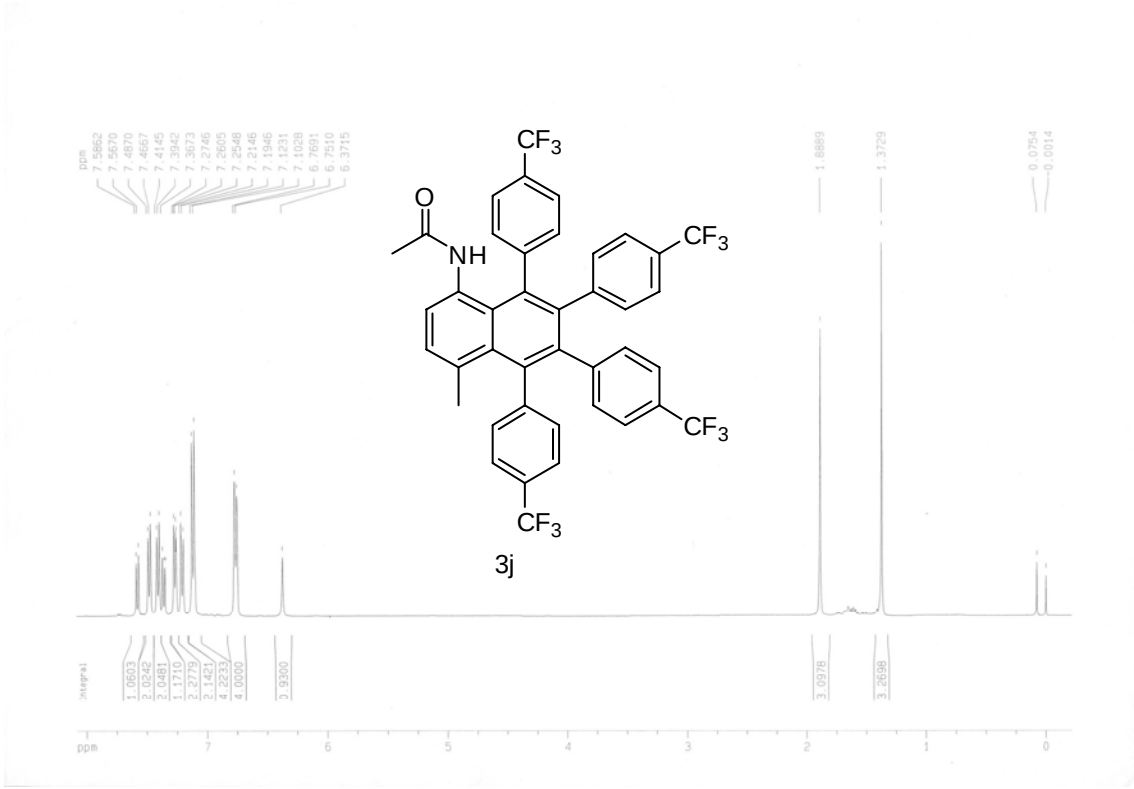


Fig. 24 ^{1}H NMR spectrum of compound **3j**

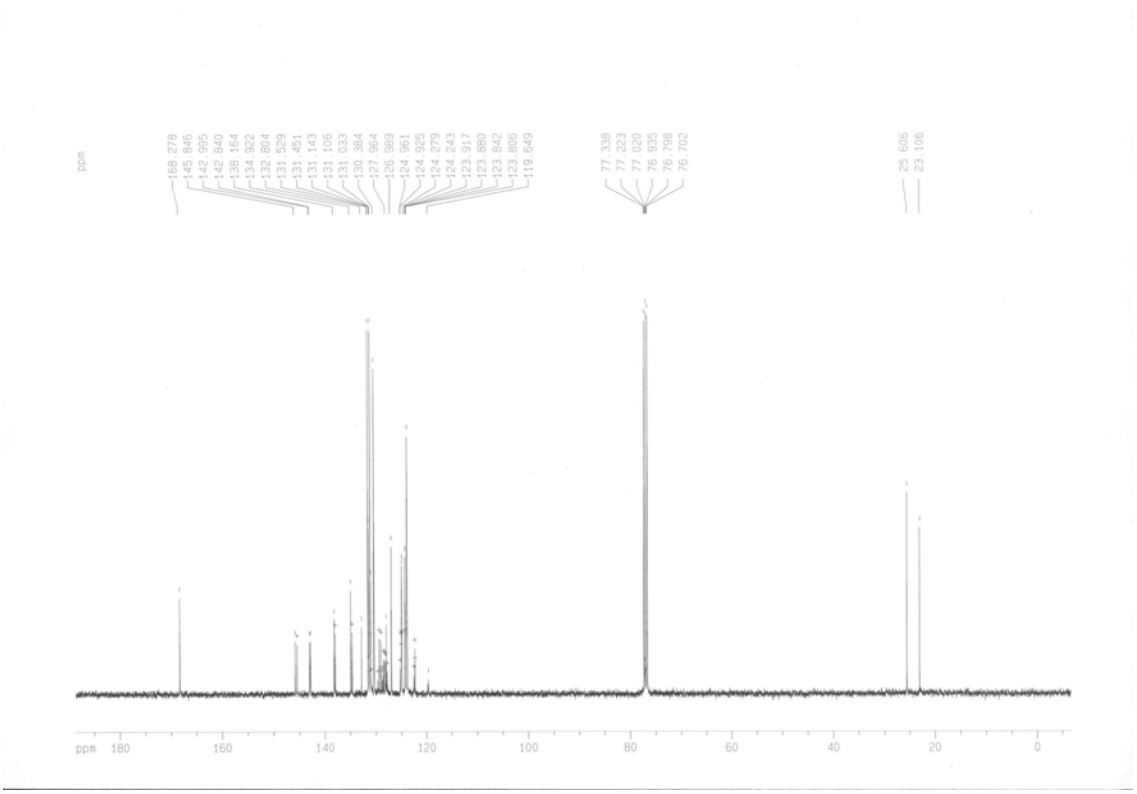


Fig. 25-a ^{13}C NMR spectrum of compound **3j**

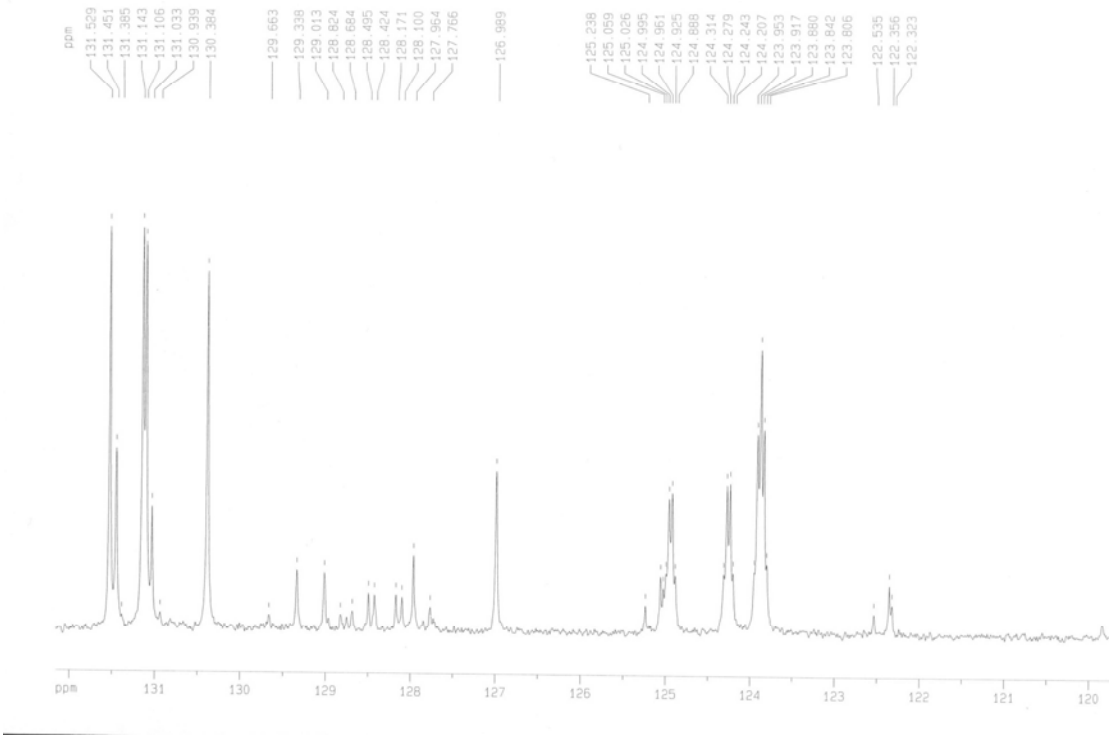


Fig. 25-b ^{13}C NMR spectrum of compound **3j**

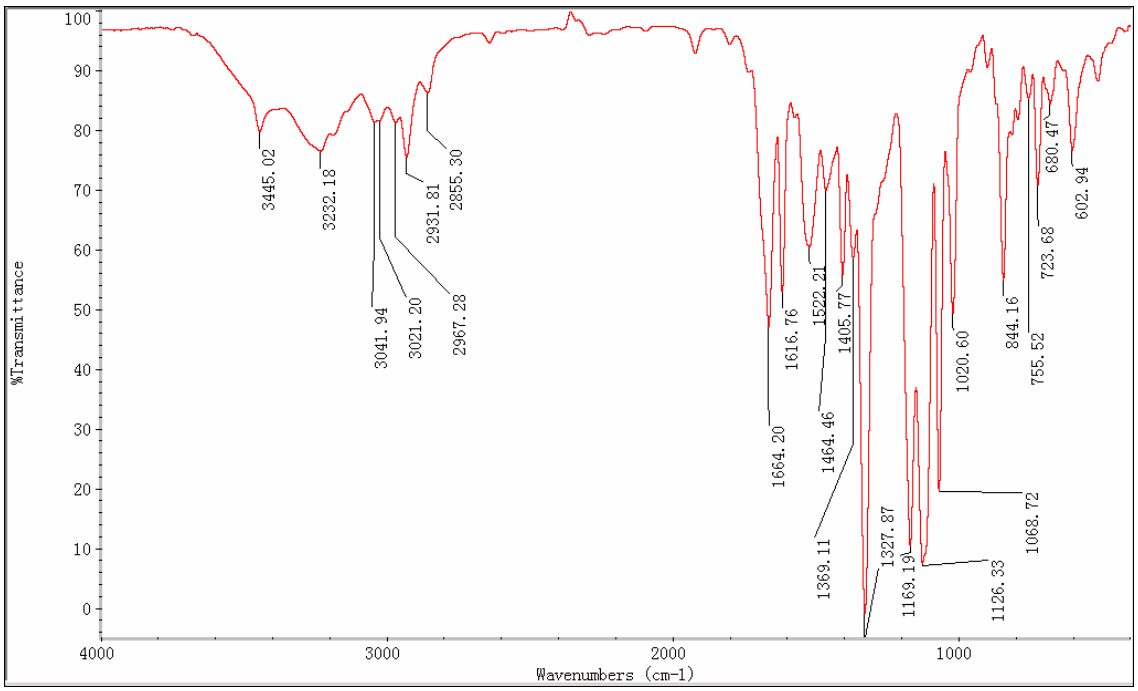


Fig. 26 IR spectrum of compound **3j**

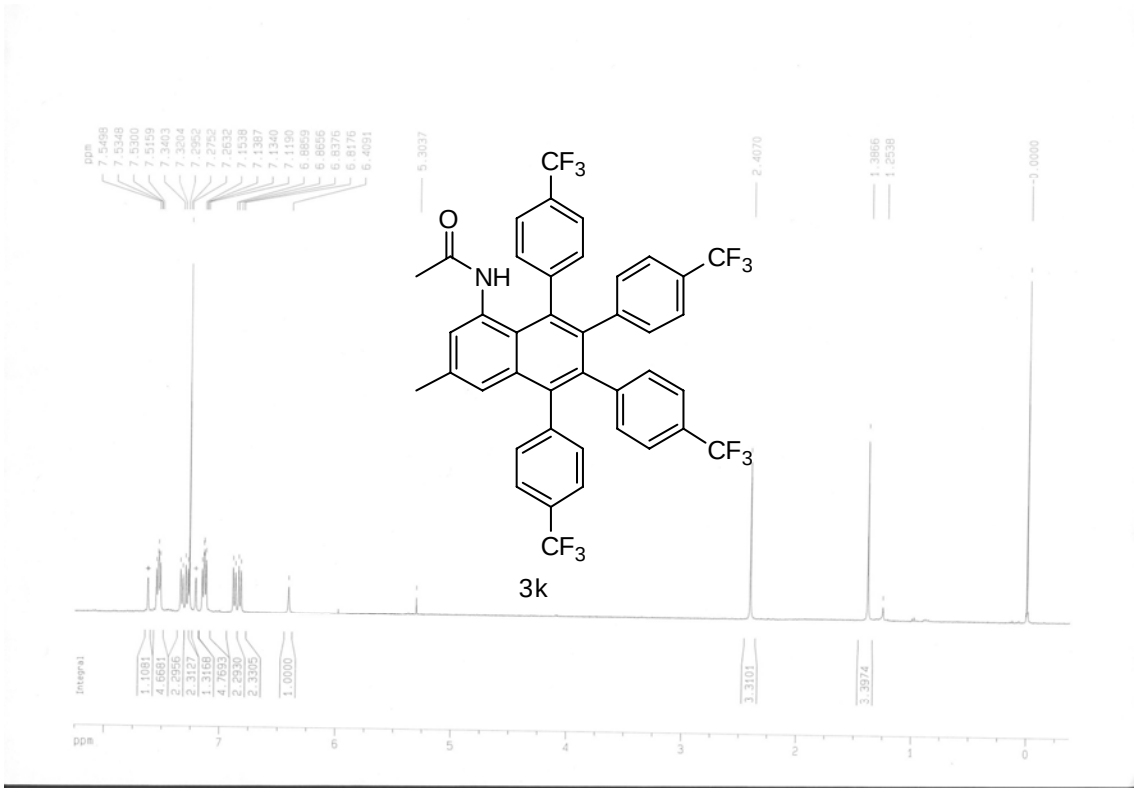


Fig. 27 ^{1}H NMR spectrum of compound **3k**

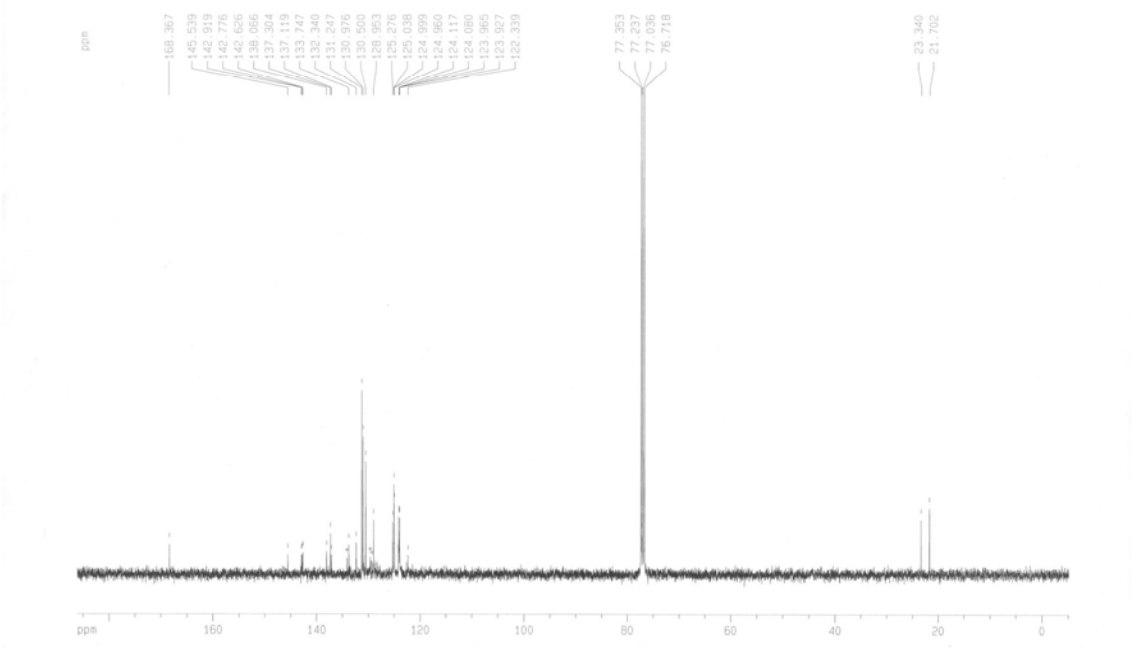


Fig. 28 ^{13}C NMR spectrum of compound**3k**

Fig. 29 ^{1}H NMR spectrum of compound **3l**

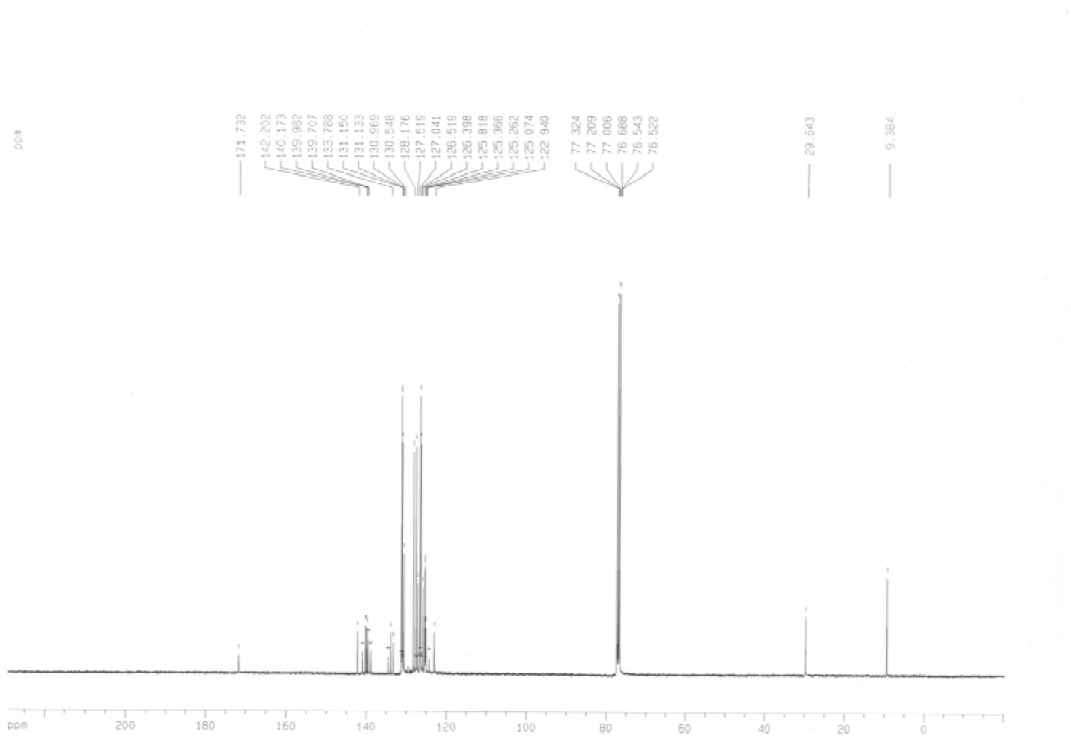
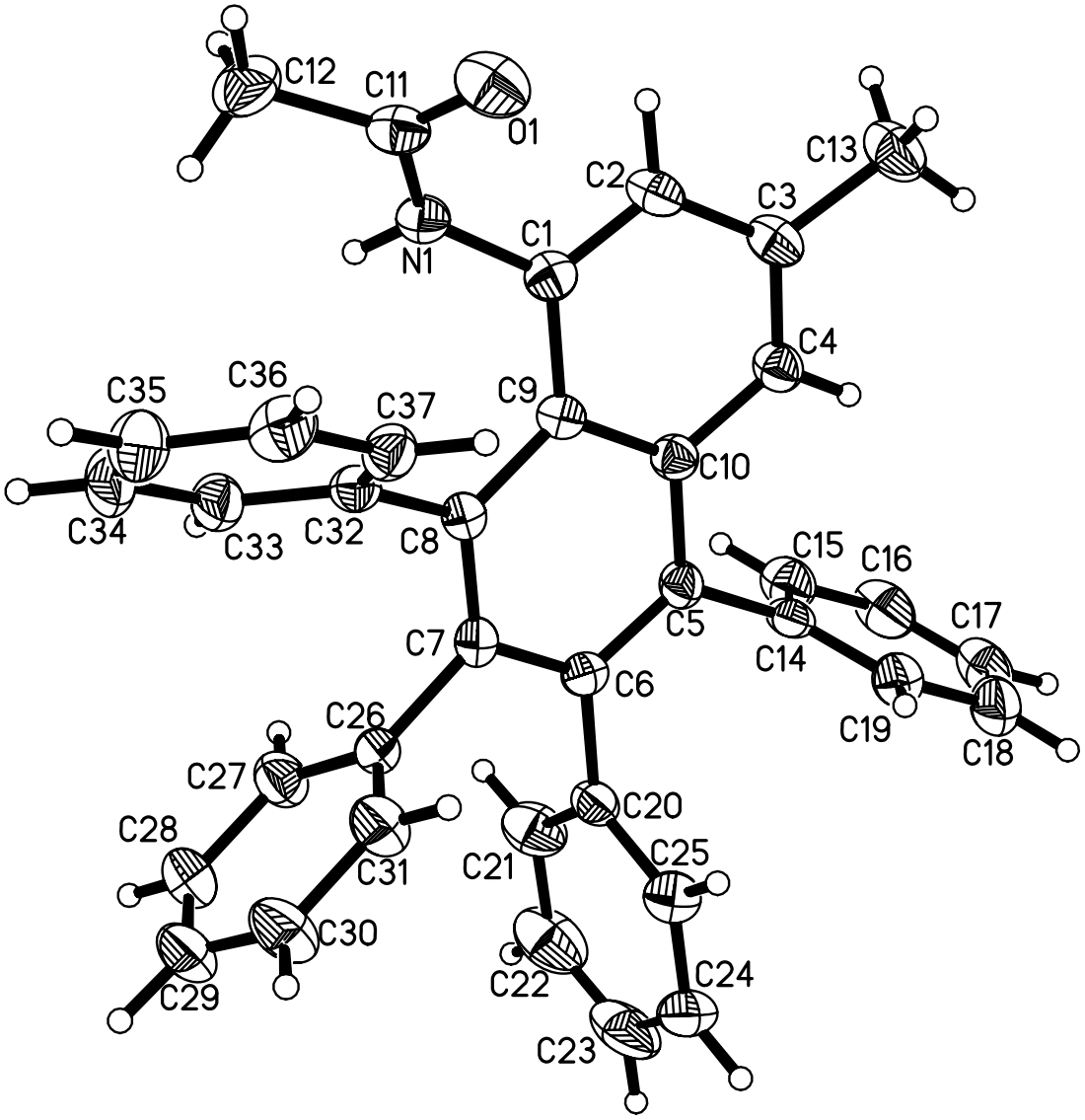
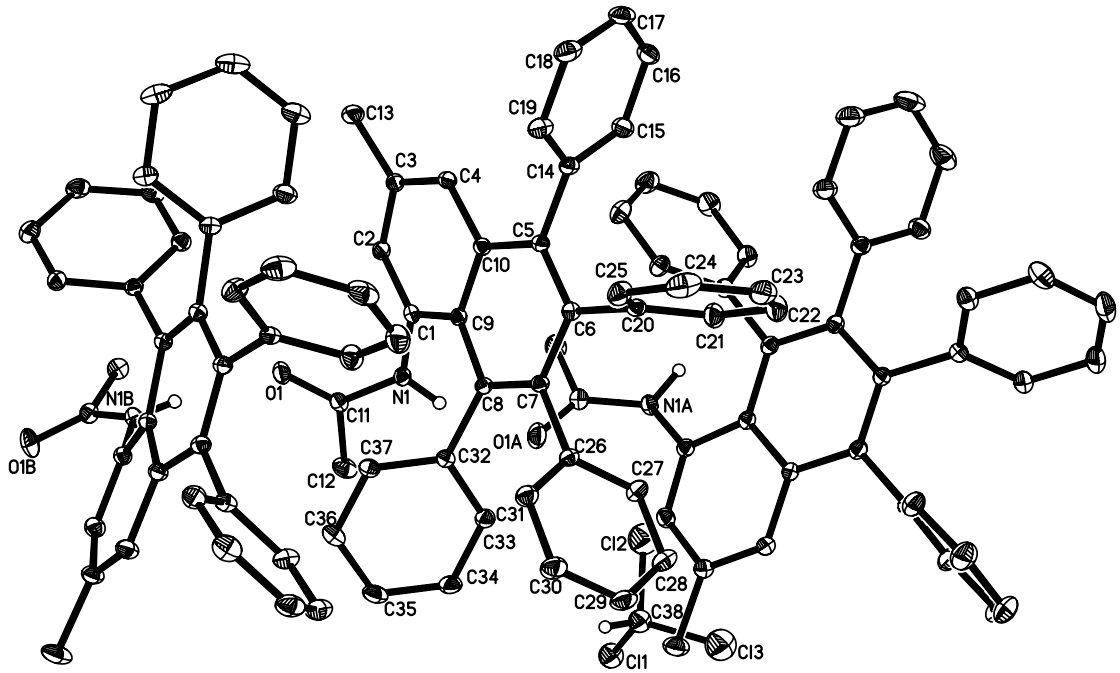


Fig. 30 ^{13}C NMR spectrum of compound **3l**

X-Ray Crystallographic Data of compound 3c




C17
C18
C16
C19
C15
C13
C3
C4
C14
C2
C5
C10
C25
C24
C23
C1
C9
C6
C20
C22
C21
O1
N1
C8
C7
N1A
N1B
C11
O1A
C37
C32
C26
C27
O1B
C12
C36
C31
C33
Cl2
C34
C30
C28
C29
C38
C35
Cl1
Cl3

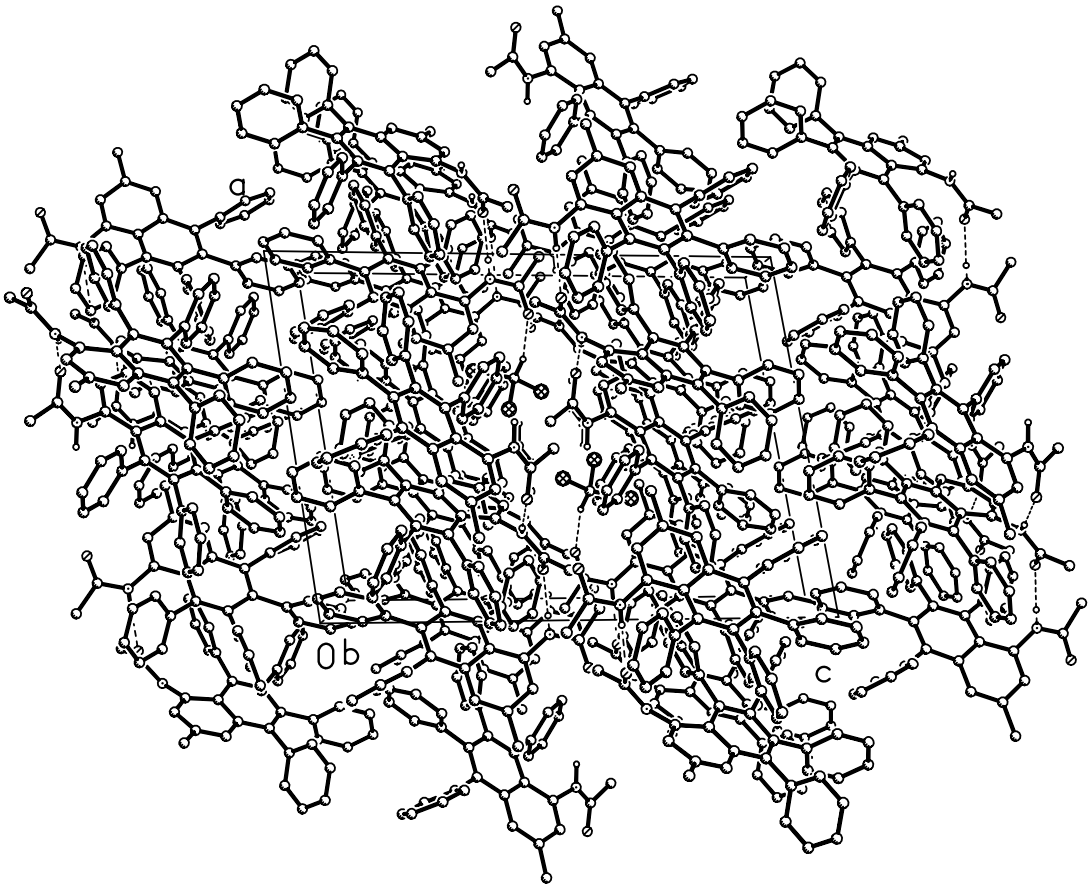

a
0 b
c

Table S2. Crystal data and structure refinement for compound 3c

Identification code	compound 3c
Empirical formula	C112 H88 Cl3 N3 O3
Formula weight	1630.20
Temperature	291(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 14.7772(10) A alpha = 101.132(6) deg. b = 15.6258(11) A beta = 96.957(5) deg. c = 20.3531(12) A gamma = 102.181(6) deg.
Volume	4442.7(5) A^3
Z, Calculated density	2, 1.219 Mg/m^3
Absorption coefficient	0.159 mm^-1
F(000)	1712
Crystal size	0.20 x 0.20 x 0.20 mm
Theta range for data collection	3.04 to 25.00 deg.
Limiting indices	-17<=h<=17, -18<=k<=18, -24<=l<=23
Reflections collected / unique	33044 / 15628 [R(int) = 0.0333]
Completeness to theta = 25.00	99.8 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9689 and 0.9689
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	15628 / 0 / 1088
Goodness-of-fit on F^2	1.050
Final R indices [I>2sigma(I)]	R1 = 0.0600, wR2 = 0.1455
R indices (all data)	R1 = 0.1400, wR2 = 0.1549
Extinction coefficient	0.0013(3)
Largest diff. peak and hole	0.724 and -0.746 e.A^-3