

SUPPORTING INFORMATION

2-((4-Arylpiperazin-1-yl)methyl)phenol ligated Pd(II) complex: An efficient, versatile catalyst for Suzuki-Miyaura cross-coupling reaction

Srinivas Keesara^{*a} and Saiprathima Parvathaneni^b

^a*School of Chemistry, University of Hyderabad, Hyderabad-500046, India*

^b*Catalysis Division, CSIR-Indian Institute of Chemical Technology (IICT), India*

**Corresponding author. Tel.: +91-9848680094*

Received:, **E-mail:** drsrinivaskeesara@yahoo.com

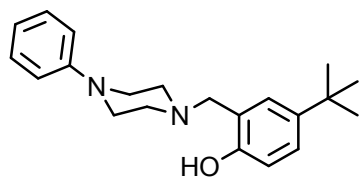
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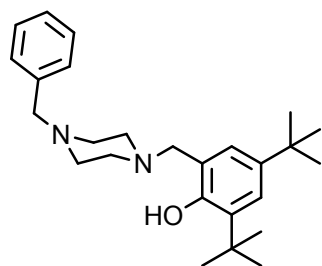
- **General Information**

All reagents were commercial grade materials and were used without further purification. All solvents were dried and distilled by standard methods. Purification of products was carried out by column chromatography using commercial column chromatography grade silica gel (60-120 mesh) using mixture of ethyl acetate and hexane as eluting agent. All known compounds were characterized and compared with the literature reports. The ^1H NMR, ^{13}C NMR and ^{19}F NMR spectra were obtained as solutions in CDCl_3 and TMS as the internal standard. IR spectra were obtained using KBr pellets. Mass spectra were determined on a LCQ ion trap mass spectrometer equipped with an ESI source or Shimadzu-LCMS-2010 a mass spectrometer. HR-MS spectra were determined on ESI-TOF maXis.

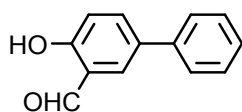
- Analytical Data**



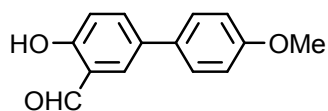
4-(tert-butyl)-2-((4-phenylpiperazin-1-yl)methyl)phenol (3a): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 10.56 (s, 1H), 7.28-7.19 (m, 3H), 6.99 (s, 1H), 6.93-6.85 (m, 3H), 6.78 (d, J = 8.33 Hz, 1H), 3.75 (s, 2H), 3.24 (s, 4H), 2.73 (s, 4H), 1.28 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 155.1, 150.9, 141.9, 129.1, 125.6, 125.4, 120.0 (d), 116.3, 115.4, 61.8, 52.5, 49.1, 33.9, 31.5; ESI-MS (m/z) (M) $^+$ = 324.



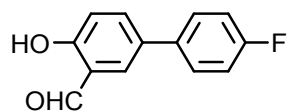
2,4-di-tert-butyl-6-((4-phenylpiperazin-1-yl)methyl)phenol (3b): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.40-7.31 (m, 6H), 6.92 (s, 1H), 3.76 (s, 2H), 3.60 (s, 2H), 2.70 (m, 8H), 1.52 (s, 9H), 1.37 (s, 9H); ^{13}C NMR (100 MHz, CDCl_3) δ 154.1, 140.4, 137.8, 135.3, 129.0, 128.2, 127.0, 123.3, 122.8, 120.5, 62.8, 62.0, 52.8, 52.2, 34.7, 34.0, 31.6, 29.5; ESI-MS (M) $^+$ = 394.



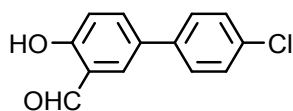
5-phenylsalicylaldehyde 8a (entry 1, Table 3): White solid, ^1H NMR (400 MHz, CDCl_3 , TMS) δ 11.00 (s, 1H), 9.96 (s, 1H), 7.77-7.75 (m, 2H), 7.55 (m, 2H), 7.47 (m, 2H), 7.37-7.33 (m, 1H), 7.08 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 196.5, 160.9, 139.3, 135.6, 133.3, 131.8, 128.9, 127.3, 126.5, 120.7, 118.1; LCMS (m/z) ($\text{M}-\text{H}$) $^+$ = 197.



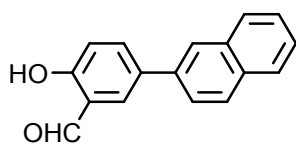
4-hydroxy-4'-methoxy-[1,1'-biphenyl]-3-carbaldehyde 8b (entry 2, Table 3): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 10.94 (s, 1H), 9.94 (s, 1H), 7.72-7.68 (m, 2H), 7.47 (d, J = 8.84 Hz, 2H), 7.05 (d, J = 8.58 Hz, 1H), 6.98 (d, J = 8.84 Hz, 2H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 196.6, 160.5, 159.2, 135.4, 133.0, 131.9, 131.3, 127.6, 120.7, 118.0, 114.4, 55.3; LCMS (m/z) ($\text{M}+\text{H}$) $^+$ = 229.



5-(-4-fluorophenyl)salicylaldehyde 8d (entry 4, Table 3): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 11.00 (s, 1H), 9.96 (s, 1H), 7.72-7.70 (m, 2H), 7.51-7.48 (m, 2H), 7.13 (t, J = 8.84 Hz, 2H), 7.07 (d, J = 7.83 Hz, 1H); ^{19}F NMR (376.46 MHz, CDCl_3) δ : -115.29 (s, 1F); ^{13}C NMR (100 MHz, CDCl_3) δ 196.5, 163.4 (d, J = 247.04 Hz), 160.9, 135.5, 132.4, 131.6, 128.2 (d, J = 8.17 Hz), 120.7, 118.2, 115.9 (d, J = 21.79 Hz); LCMS (m/z) ($\text{M}-\text{H}$) $^+$ = 215.

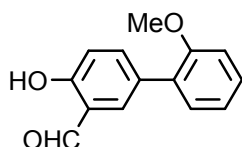


4'-chloro-4-hydroxy-[1,1'-biphenyl]-3-carbaldehyde 8e (entry 5, Table 3): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 10.91 (m, 1H), 9.84 (m, 1H), 7.62-7.59 (m, 2H), 7.37-7.30 (m, 4H), 6.98-6.94 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 196.5, 161.1, 137.8, 135.4, 133.5, 132.0, 131.7, 129.1, 127.8, 120.7, 118.3; LCMS (m/z) ($\text{M}+\text{H}$) $^+$ = 233.

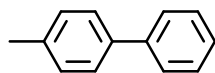


2-hydroxy-5-(naphthalen-2-yl)benzaldehyde 8g (entry 7, Table 3): Light yellow solid, mp:

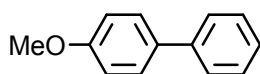
171 °C. ^1H NMR (400 MHz, CDCl_3 , TMS) δ 10.96 (s, 1H), 9.91 (s, 1H), 7.90 (s, 1H), 7.85-7.78 (m, 5H), 7.61 (d, J = 8.33 Hz, 1H), 7.44-7.41 (m, 2H), 7.04 (d, J = 8.58 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 196.7, 161.0, 136.6, 135.9, 133.6, 133.2, 132.5, 132.1, 128.7, 128.0, 127.7, 126.5, 126.1, 125.2, 124.9, 120.8, 118.2; HRMS: exact mass calculated for $\text{C}_{17}\text{H}_{13}\text{O}_2$ $[\text{M}+\text{H}]^+ = 249.0916$, found $m/z = 249.0919$.



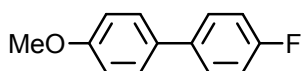
4-hydroxy-2'-methoxy-[1,1'-biphenyl]-3-carbaldehyde 8i (entry 9, Table 3): White solid, mp: 92 °C. ^1H NMR (400 MHz, CDCl_3 , TMS) δ 10.93 (s, 1H), 9.81 (s, 1H), 7.62 (m, 2H), 7.26-7.19 (m, 2H), 6.96-6.89 (m, 3H), 3.72 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 196.6, 160.5, 156.2, 138.3, 134.3, 130.2, 128.8, 128.5, 120.9, 120.2, 117.1, 111.2, 55.4; HRMS: exact mass calculated for $\text{C}_{14}\text{H}_{13}\text{O}_3$ $[\text{M}+\text{H}]^+ = 229.0865$, found $m/z = 229.0861$.



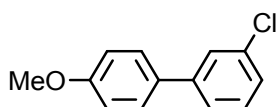
4-methylbiphenyl 9a (entry 1, table 4) ^1H NMR (300 MHz, CDCl_3 , TMS) δ 7.51 (d, J = 7.55 Hz, 2H), 7.44-7.34 (m, 4H), 7.26 (t, J = 7.17 Hz, 1H), 7.18 (d, J = 7.93 Hz, 2H), 2.38 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 141.1, 138.3, 136.9, 129.4, 128.6, 126.9, 21.0.



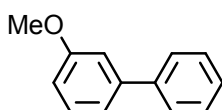
4-methoxybiphenyl 9b (entry 2, Table 4): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.49 (d, J = 8.00 Hz, 2H), 7.47 (d, J = 9.00 Hz, 2H), 7.34 (t, J = 8.00 Hz, 2H), 7.24 (m, 1H), 6.92 (d, J = 9.00 Hz, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) 159.0, 140.7, 133.7, 128.6, 128.0, 126.6 (d), 114.1, 55.2.



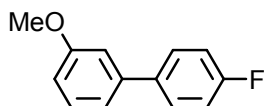
4-fluoro-4'-methoxy-1,1'-biphenyl 9c (entry 3, Table 4): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.49-7.44 (m, 4H), 7.08 (t, J = 8.84 Hz, 2H), 6.96 (d, J = 8.84 Hz, 2H), 3.82 (s, 3H); ^{19}F NMR (376.46 MHz, CDCl_3) δ : -116.71 (s, 1F); ^{13}C NMR (100 MHz, CDCl_3) 163.37 (d, J = 245.89 Hz), 159.1, 136.9, 132.8, 128.2 (d, J = 8.05 Hz), 127.9, 115.5 (d, J = 21.22 Hz), 114.2, 55.3.



3-chloro-4'-methoxy-1,1'-biphenyl 9d (entry 4, Table 4): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.42 (s, 1H), 7.39 (d, J = 8.84 Hz, 2H), 7.31 (d, J = 7.57 Hz, 1H), 7.21 (t, J = 7.57 Hz, 1H), 7.17 (d, J = 7.83 Hz, 1H), 6.87 (d, J = 8.84 Hz, 2H), 3.72 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) 159.5, 142.6, 134.5, 132.2, 129.8, 128.0, 126.7, 126.5, 124.7, 114.2, 55.2.

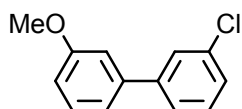


3-methoxybiphenyl 9e (entry 5, Table 4): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.58 (d, J = 7.07 Hz, 2H), 7.42 (t, J = 7.57 Hz, 2H), 7.36-7.32 (m, 2H), 7.18 (d, J = 6.82 Hz, 1H), 7.12 (m, 1H), 6.89 (d, J = 8.33 Hz, 1H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) 159.9, 142.7, 130.7, 129.7, 129.6, 128.6, 127.3, 127.1, 119.6, 112.8, 55.2.

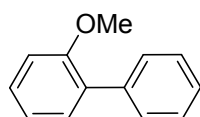


4'-fluoro-3-methoxy-1,1'-biphenyl 9f (entry 6, Table 4): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.47-7.43 (m, 2H), 7.26 (t, J = 8.08 Hz, 1H), 7.05-6.99 (m, 4H), 6.82 (d, J = 8.08 Hz, 1H), 3.77

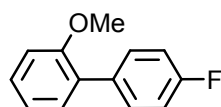
(s, 3H); ^{19}F NMR (376.46 MHz, CDCl_3) δ : -115.56 (s, 1F); ^{13}C NMR (100 MHz, CDCl_3) 163.7 (d, J = 245.89 Hz), 159.9, 141.7, 137.1, 129.8, 128.7 (d, J = 8.05 Hz), 119.5, 115.7 (d, J = 21.22 Hz), 112.8 (d, J = 29.27 Hz), 55.2.



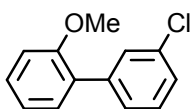
3-chloro-3'-methoxybiphenyl 9g (entry 7, Table 4): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.56 (m, 1H), 7.45 (d, J = 7.07 Hz, 1H), 7.36-7.29 (m, 3H), 7.14 (d, J = 7.83 Hz, 1H), 7.08 (m, 1H), 6.92 (d, J = 8.08 Hz, 1H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) 159.9, 142.9, 141.2, 134.5, 129.9, 129.8, 127.3, 127.2, 125.3, 119.5, 113.2, 112.8, 55.3.



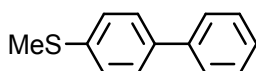
2-methoxybiphenyl 9h (entry 8, Table 4): ^1H NMR (300 MHz, CDCl_3 , TMS) δ 7.53 (d, J = 7.17 Hz, 2H), 7.40 (t, J = 7.17 Hz, 2H), 7.33-7.29 (m, 3H), 7.04 (d, J = 7.36 Hz, 1H), 6.97 (d, J = 8.68 Hz, 1H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 156.4, 138.5, 130.8, 129.5, 128.5, 127.9, 126.8, 120.8, 111.2, 55.5.



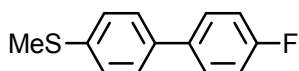
2-methoxy(-4'-fluorobiphenyl) 9i (entry 9, table 4) ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.55-7.51 (m, 2H), 7.34 (t, J = 8.08 Hz, 1H), 7.13-7.06 (m, 4H), 6.89 (d, J = 8.08 Hz, 1H), 3.85 (s, 3H); ^{19}F NMR (376.46 MHz, CDCl_3) δ : -115.82 (t, J = 5.45 and 2.72 Hz, 1F); ^{13}C NMR (100 MHz, CDCl_3) δ 162.9 (d, J = 246.13 Hz), 156.4, 134.4 (d, J = 3.63 Hz), 133.3, 131.1 (d, J = 8.17 Hz), 130.7, 128.7, 120.8, 114.8 (d, J = 20.89 Hz), 111.3, 55.5.



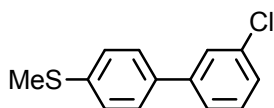
3'-chloro-2-methoxy-1,1'-biphenyl 9j (entry 10, Table 4): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.44 (s, 1H), 7.33 (d, J = 7.32 Hz, 1H), 7.28-7.20 (m, 4H), 6.96-6.89 (m, 2H), 3.73 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 156.4, 140.3, 133.3, 130.7, 129.6, 129.1 (d), 128.4, 127.6, 126.9, 121.7, 112.0, 56.1.



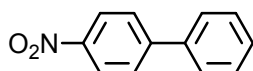
(biphenyl)-4-thiomethane 9k (entry 11, Table 4): ^1H NMR (300 MHz, CDCl_3 , TMS) δ 7.58-7.51 (m, 4H), 7.43 (t, J = 7.74 Hz, 2H), 7.33 (d, J = 8.49 Hz, 3H), 2.52 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 140.4, 138.0, 137.5, 128.7, 127.4, 127.1, 126.9, 126.7, 15.8.



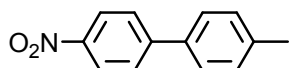
(4'-fluoro-[1,1'-biphenyl]-4-yl)(methyl)sulfane 9l (entry 12, Table 4): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.51-7.43 (m, 4H), 7.46 (d, J = 8.33 Hz, 2H), 7.10 (t, J = 8.84 Hz, 2H), 2.50 (s, 3H); ^{19}F NMR (376.46 MHz, CDCl_3) δ : -115.78 (s, 1F); ^{13}C NMR (100 MHz, CDCl_3) 163.5 (d, J = 245.89 Hz), 137.6, 137.0, 136.6 (d, J = 3.65 Hz), 128.4 ((d, J = 8.05 Hz)), 127.2, 126.9, 115.7 (d, J = 21.22 Hz), 15.8.



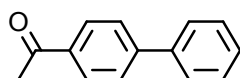
(3'-chloro-[1,1'-biphenyl]-4-yl)(methyl)sulfane 9m (entry 13, Table 4): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.54 (s, 1H), 7.49-7.42 (m, 3H), 7.36-7.28 (m, 4H), 2.51 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) 142.3, 138.5, 136.4, 134.7, 130.0, 127.4, 127.1, 126.9, 126.8, 124.9, 15.7.



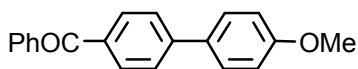
4-Nitrobiphenyl 9n (entry 1, Table 5): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.28 (d, J = 9.11 Hz, 2H), 7.71 (d, J = 9.11 Hz, 2H), 7.58 (d, J = 7.28 Hz, 2H), 7.46 (t, J = 8.20 Hz, 2H), 7.41 (m, 1H); ^{13}C NMR (100 MHz, CDCl_3) 147.6, 143.6, 138.7, 129.1, 128.9, 127.7, 127.3, 124.0.



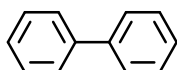
4-methyl-4'-nitrobiphenyl 9o (entry 2, Table 5): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.27 (d, J = 8.33 Hz, 2H), 7.71 (d, J = 8.58 Hz, 2H), 7.53 (d, J = 8.08 Hz, 2H), 7.30 (d, J = 7.83 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) 147.5, 146.7, 139.0, 135.7, 129.8, 127.3, 127.1, 124.0, 21.1.



4-Acetylbiphenyl 9p (entry 3, Table 5): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.01 (m, 2H), 7.70 (d, J = 8.33 Hz, 2H), 7.64 (d, J = 6.82 Hz, 2H), 7.47 (m, 2H), 7.42 (d, J = 6.92 Hz, 1H), 2.64 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) 197.7, 145.7, 139.8, 135.8, 128.9, 128.2, 127.2 (d), 26.7.

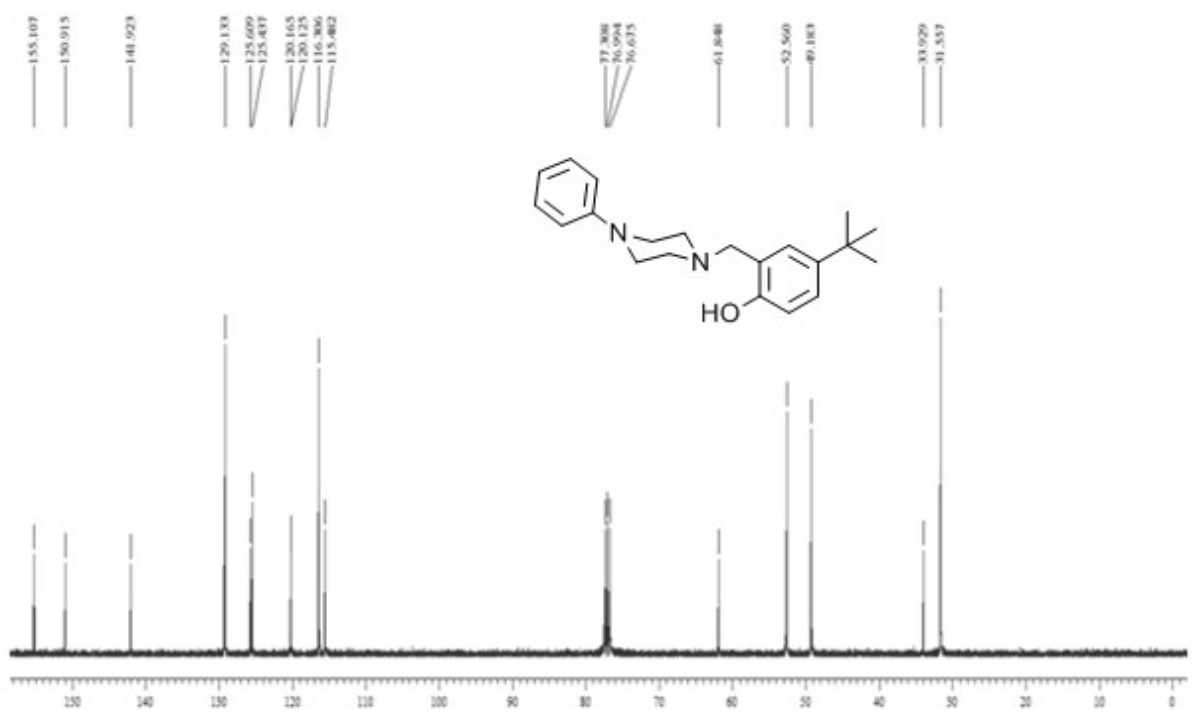
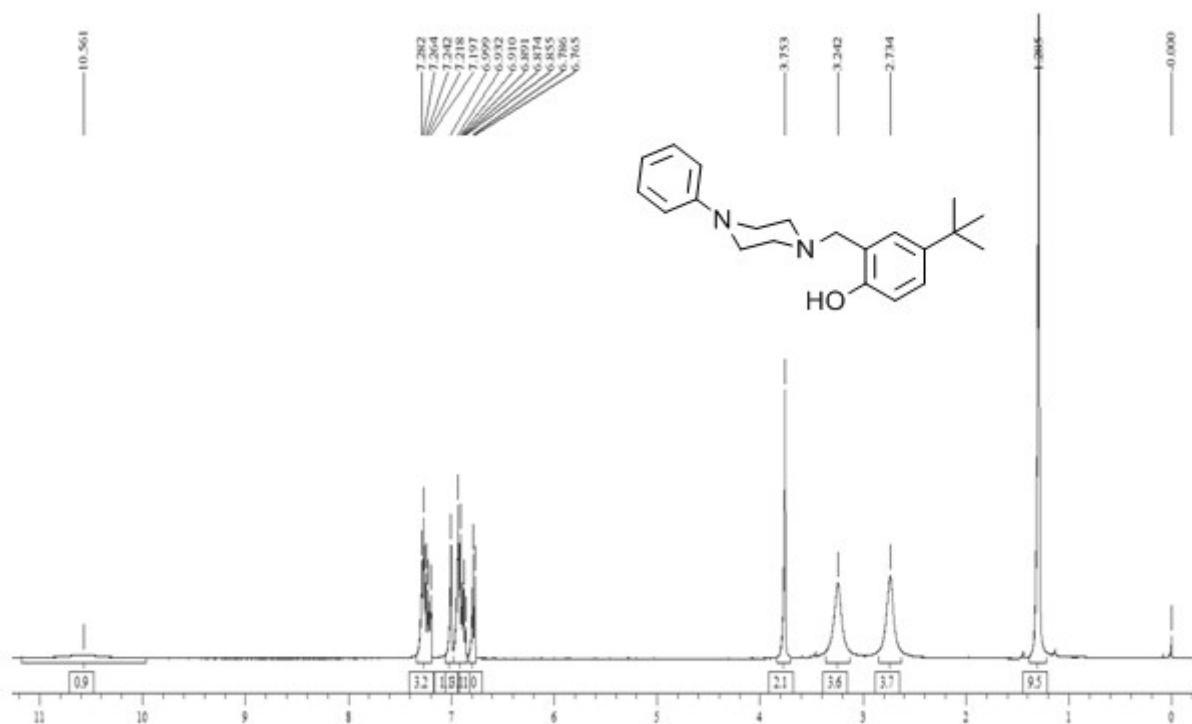


(4'-methoxybiphenyl-4-yl)(phenyl)methanone 9q (entry 4, Table 5): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 7.68 (t, J = 8.58 Hz, 6H), 7.51 (t, J = 7.57 Hz, 2H), 7.42-7.36 (m, 5H), 3.74 (s, 1H); ^{13}C NMR (100 MHz, CDCl_3) 195.4, 138.8, 137.1, 135.8, 133.4, 132.5, 131.4, 129.8, 128.5, 128.3, 127.6, 114.1, 55.2.

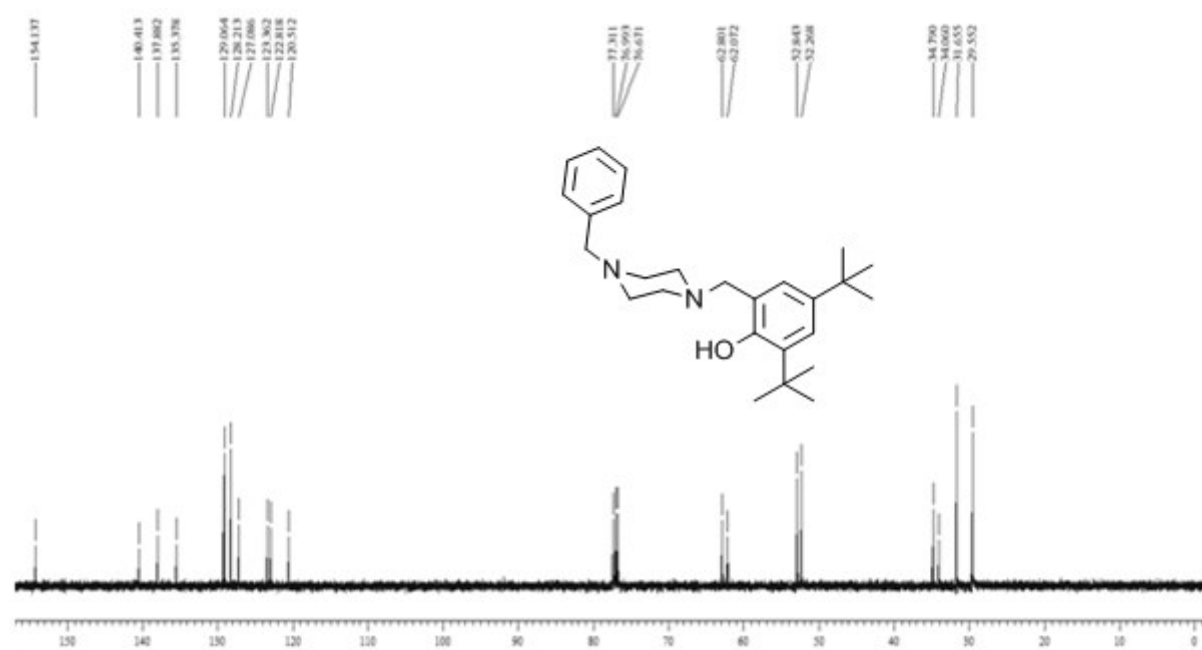
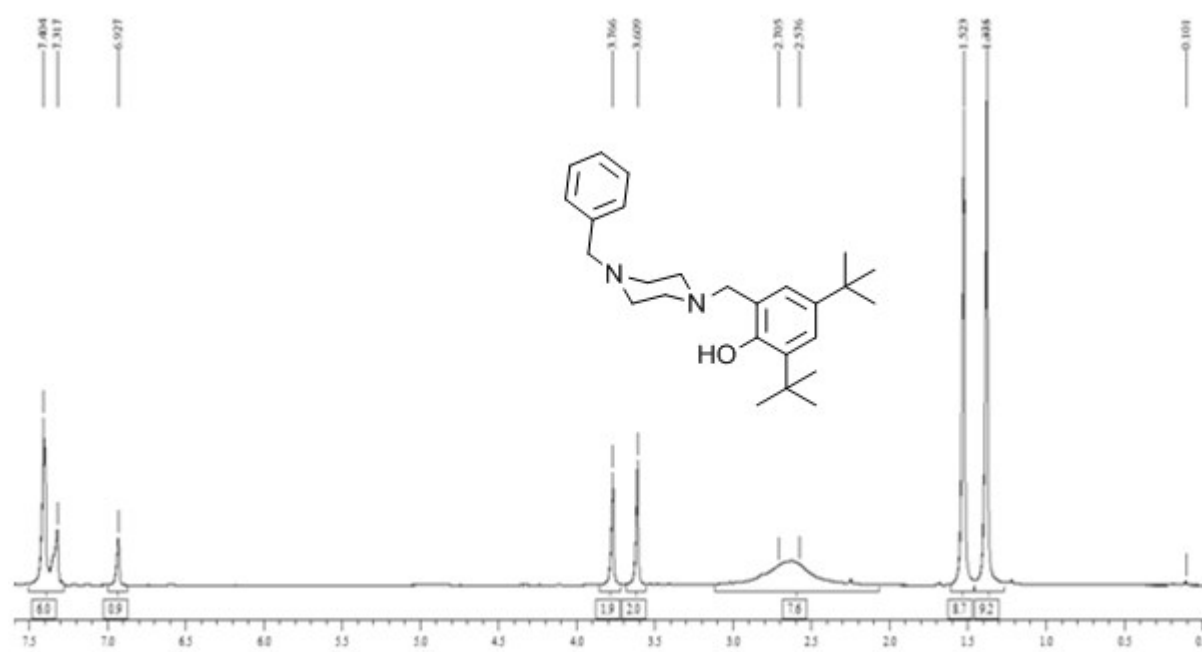


Biphenyl 9r (entry 5, Table 5): ^1H NMR (400 MHz, CDCl_3 , TMS) δ 8.21 (d, J = 6.79 Hz, 4H), 7.56 (d, J = 7.55 Hz, 2H), 7.46 (t, J = 6.79 and 7.55 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3) 141.3, 128.7, 127.2, 96.2.

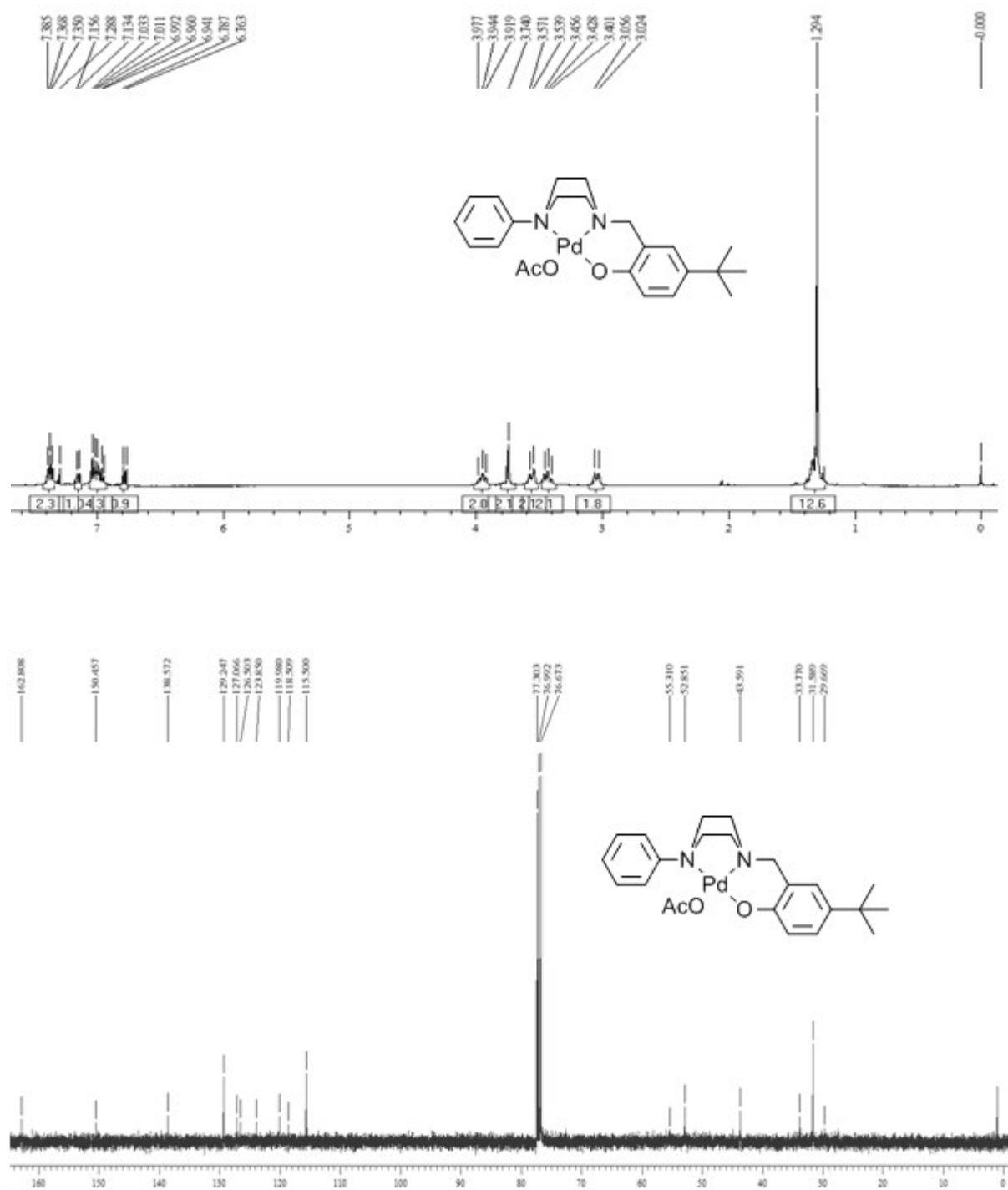
^1H & ^{13}C NMR spectrum of ligand 3a



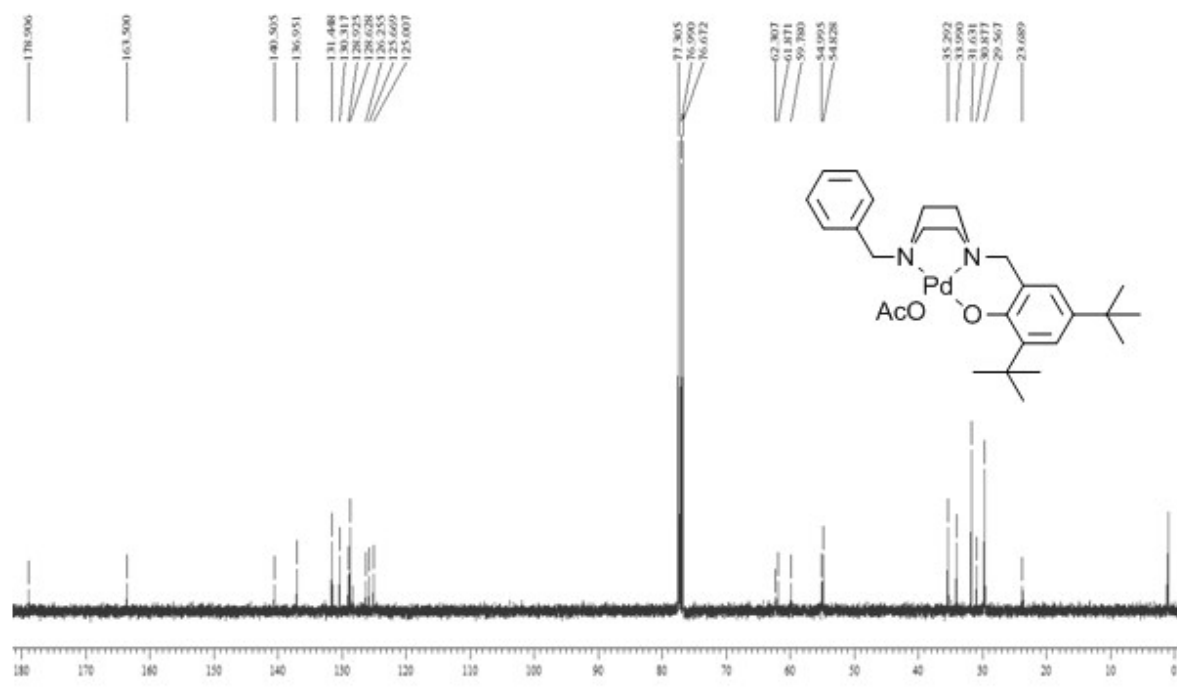
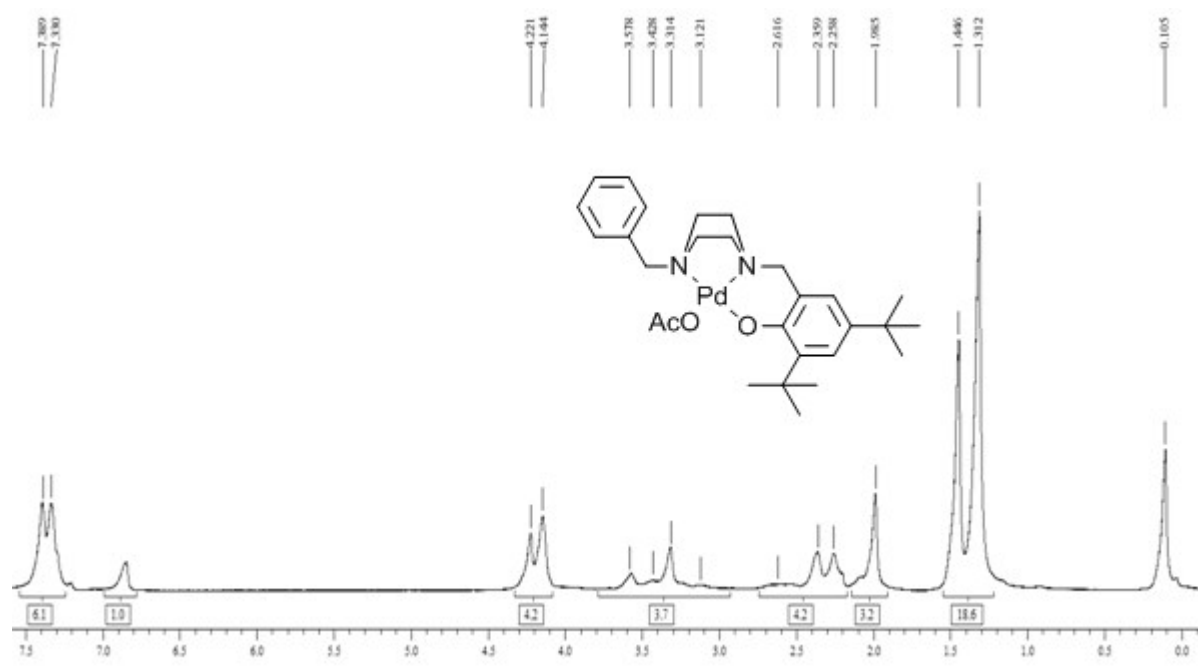
¹H & ¹³C NMR spectrum of ligand 3b

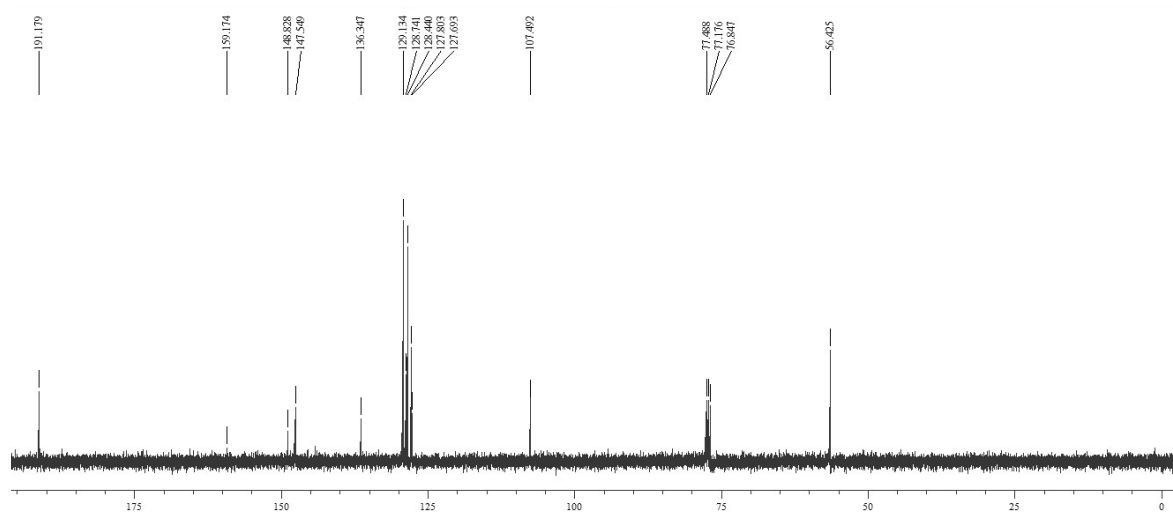
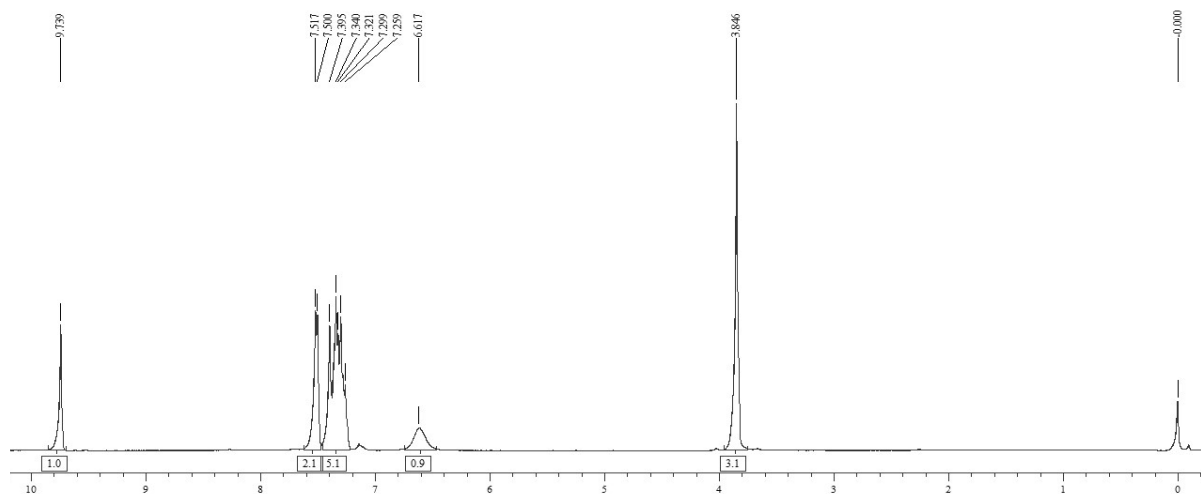


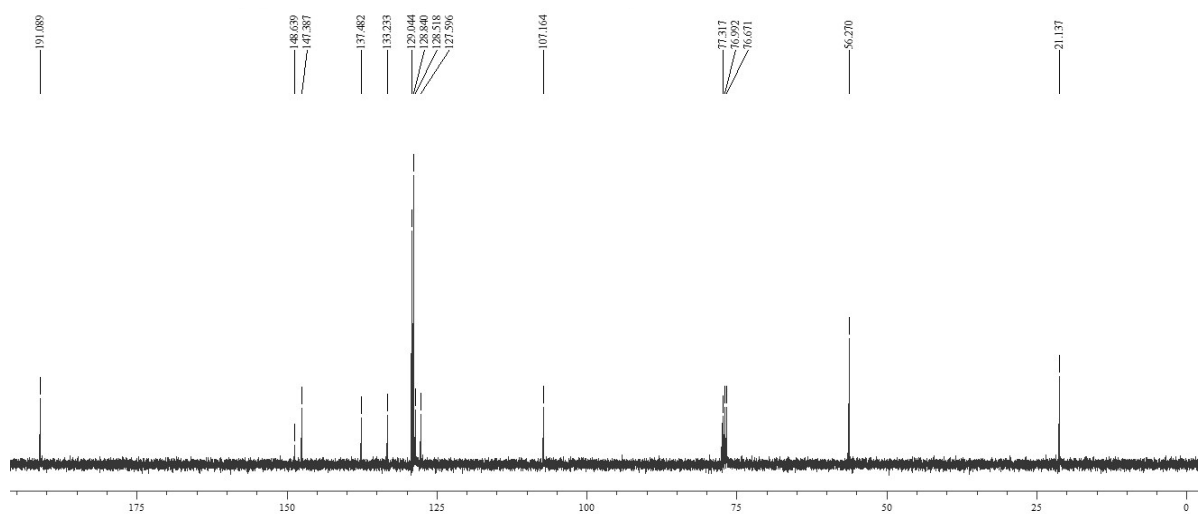
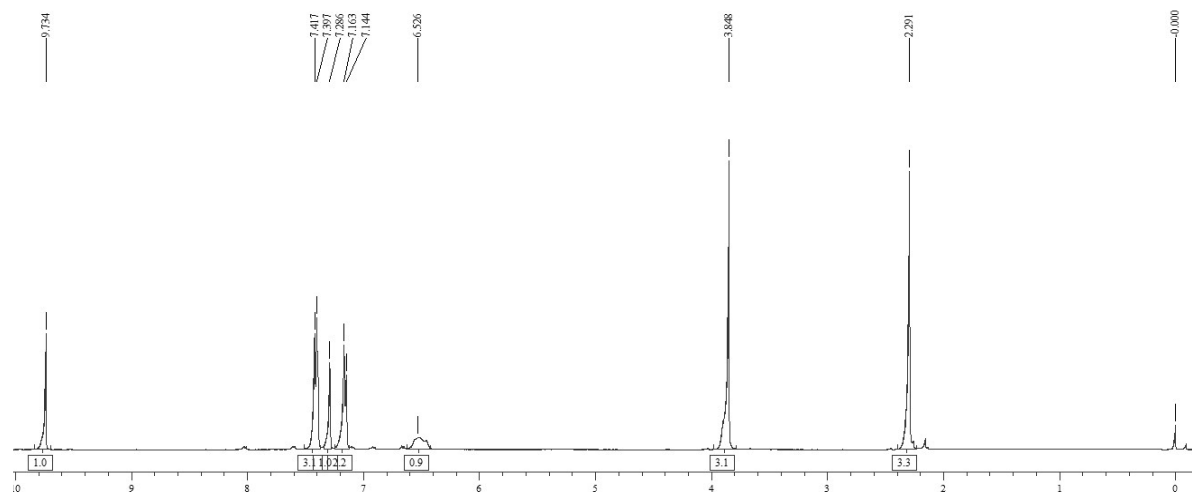
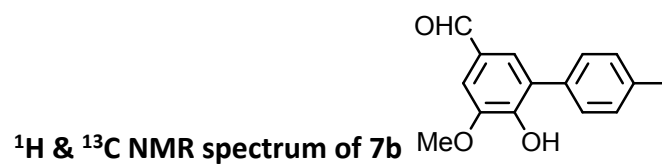
^1H & ^{13}C NMR spectrum of Pd(II) complex 4a

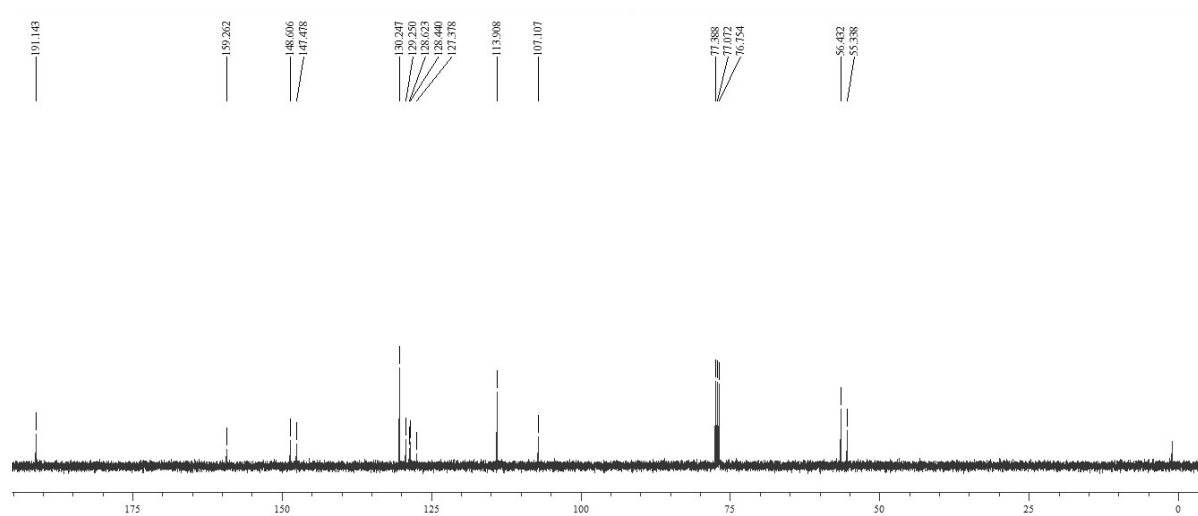
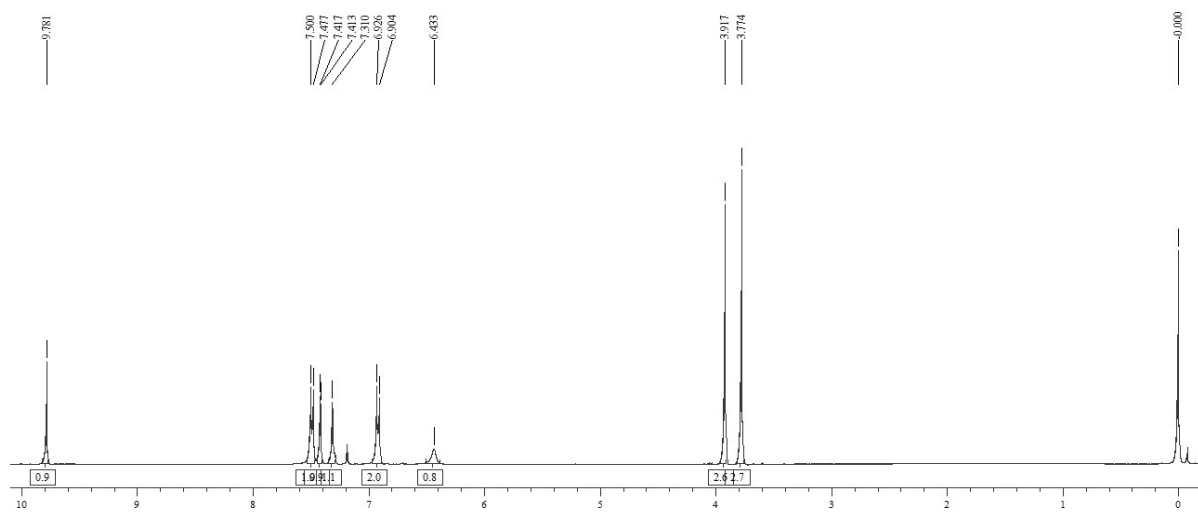
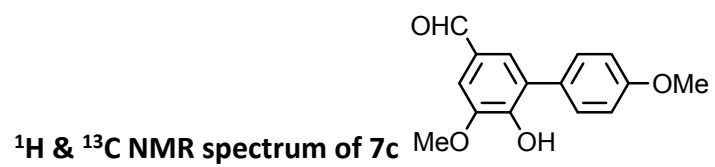


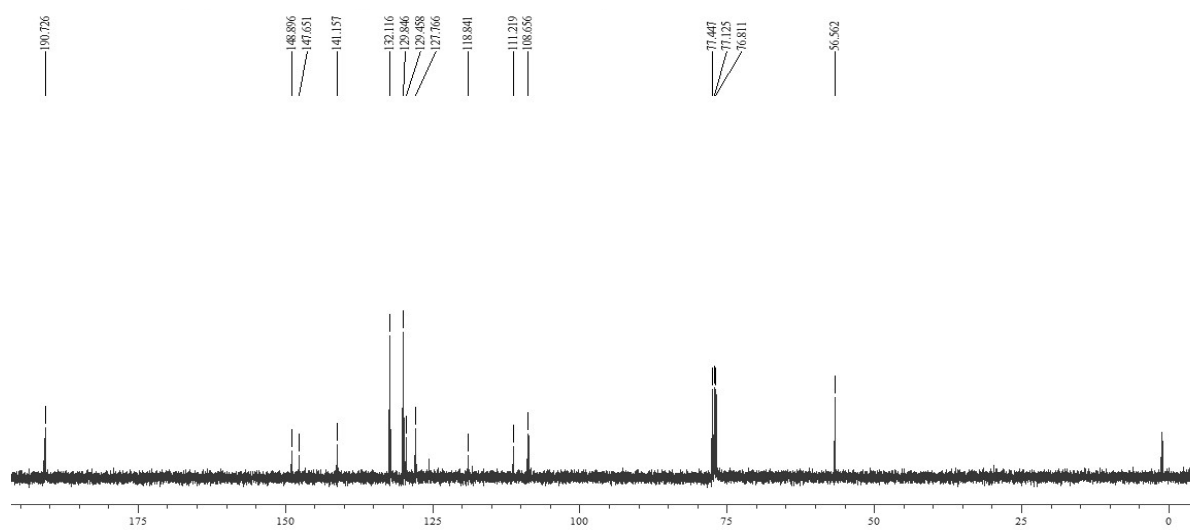
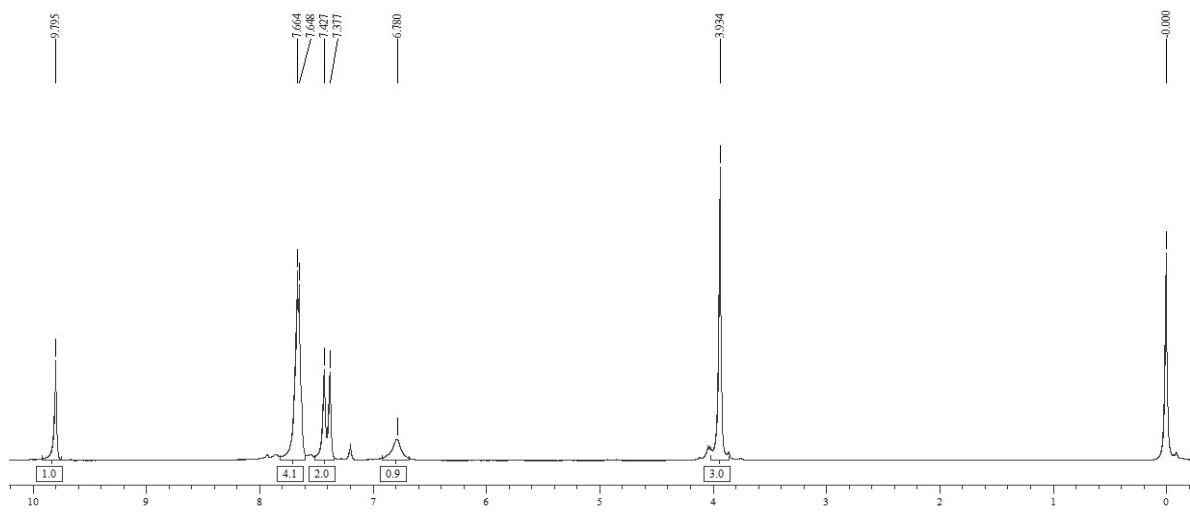
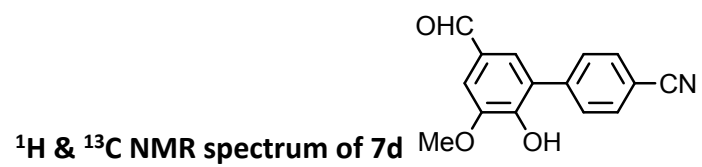
^1H & ^{13}C NMR spectrum of Pd(II) complex 4b

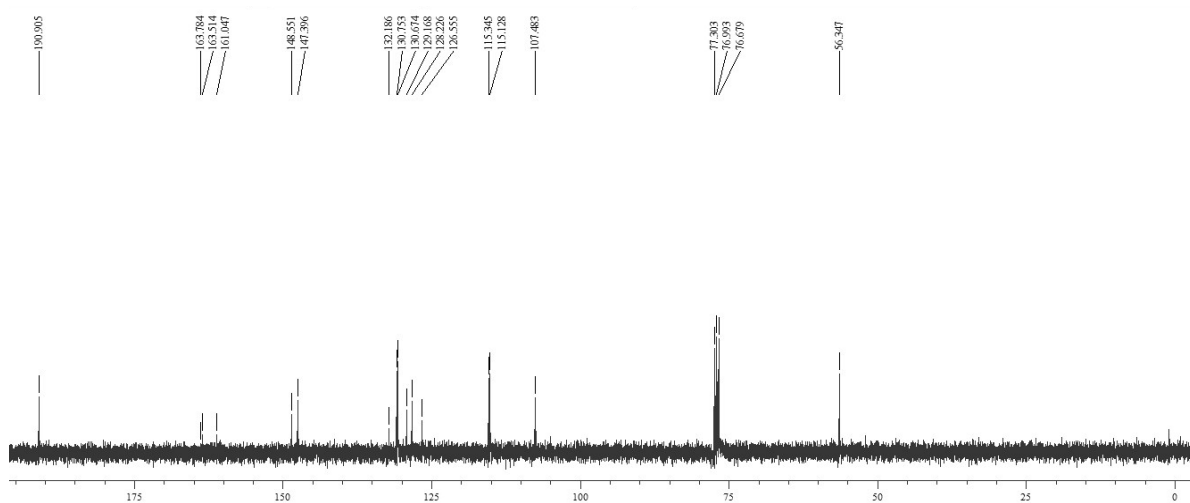
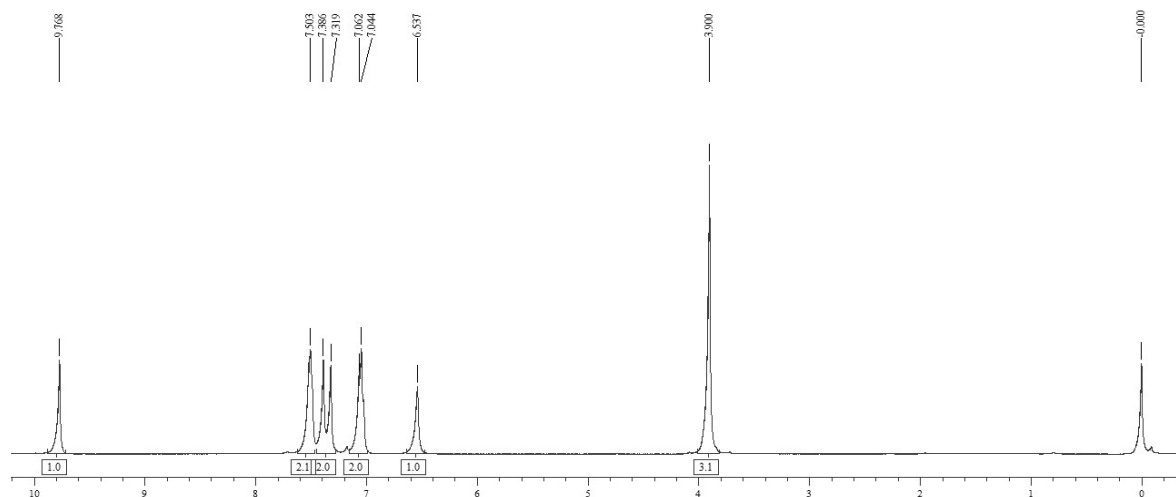
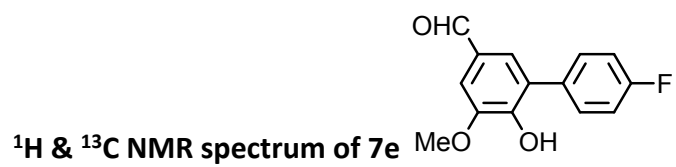


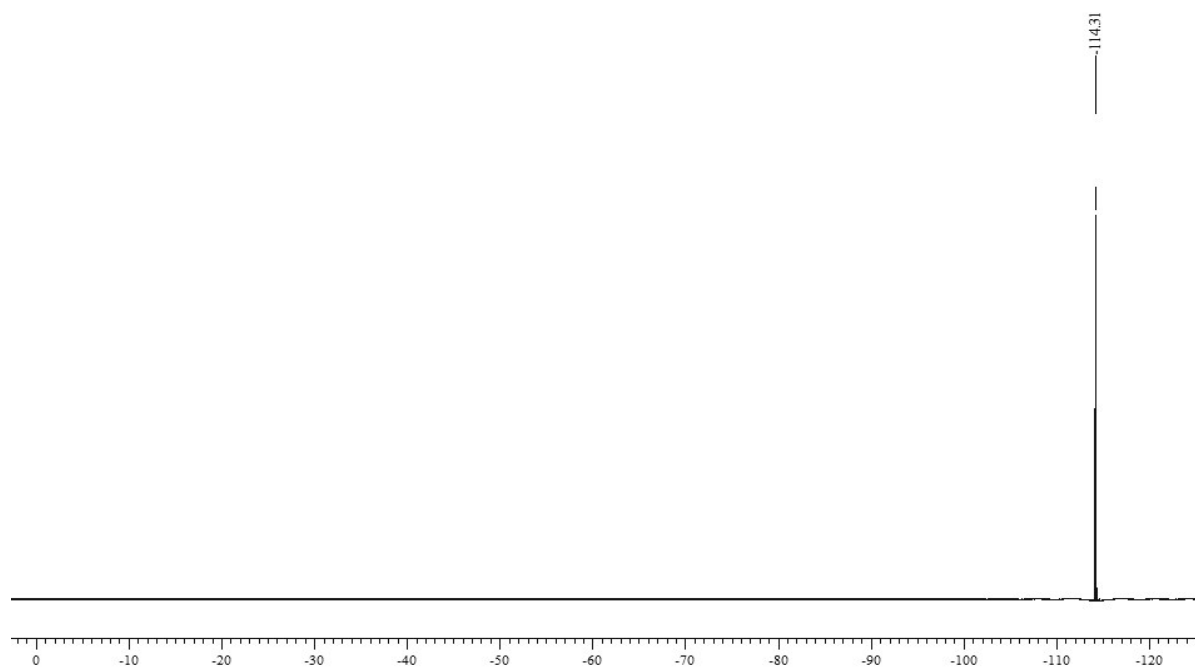
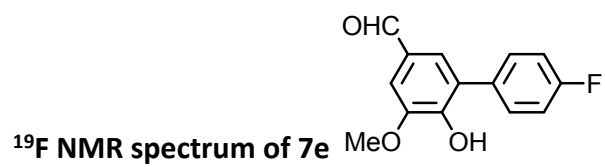


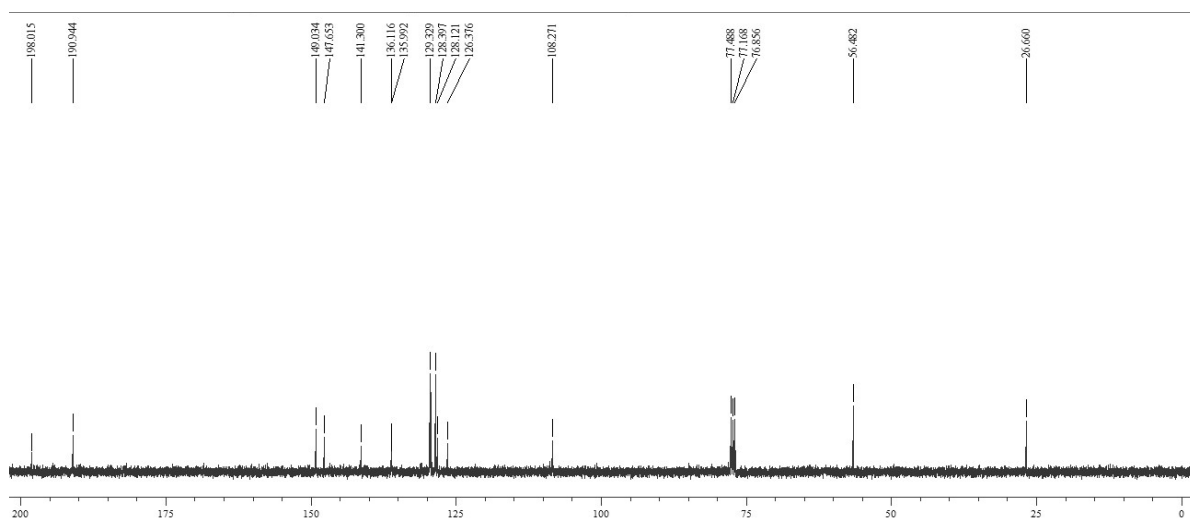
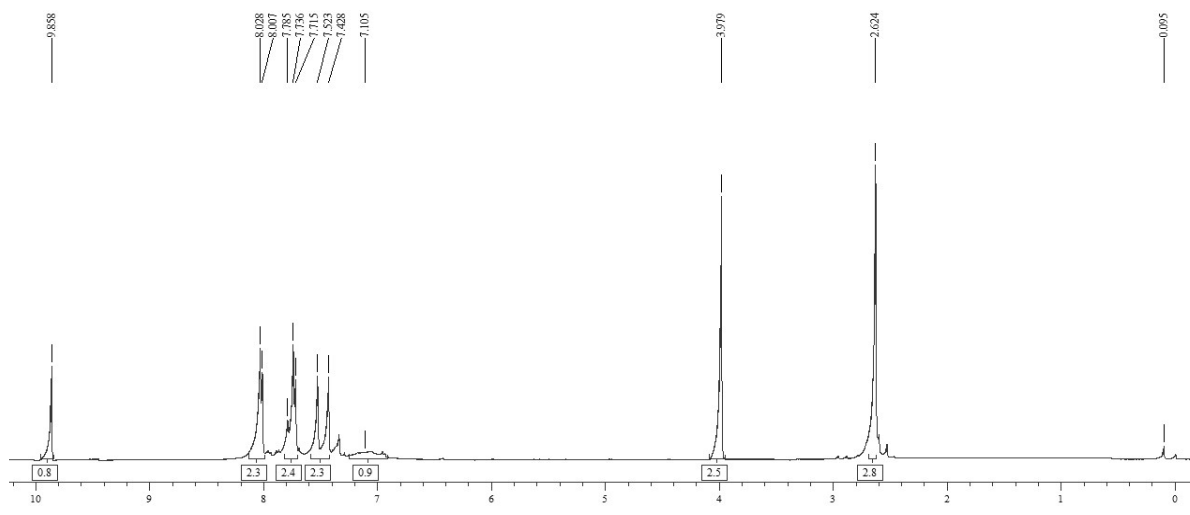
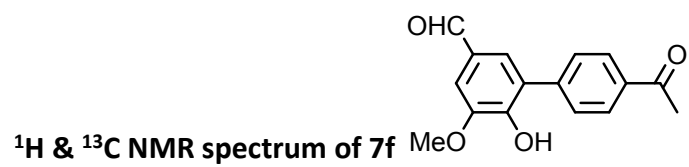


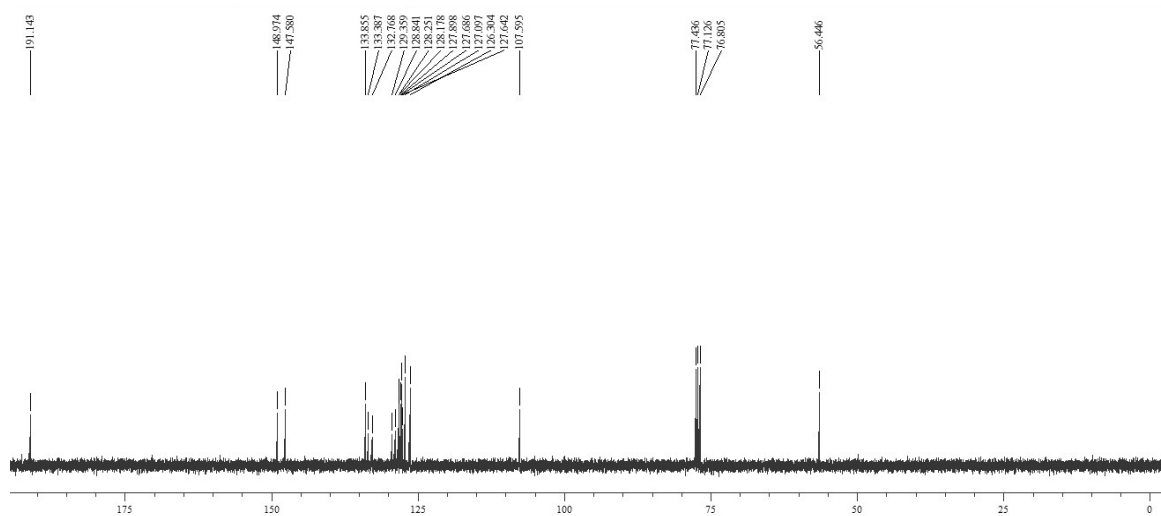
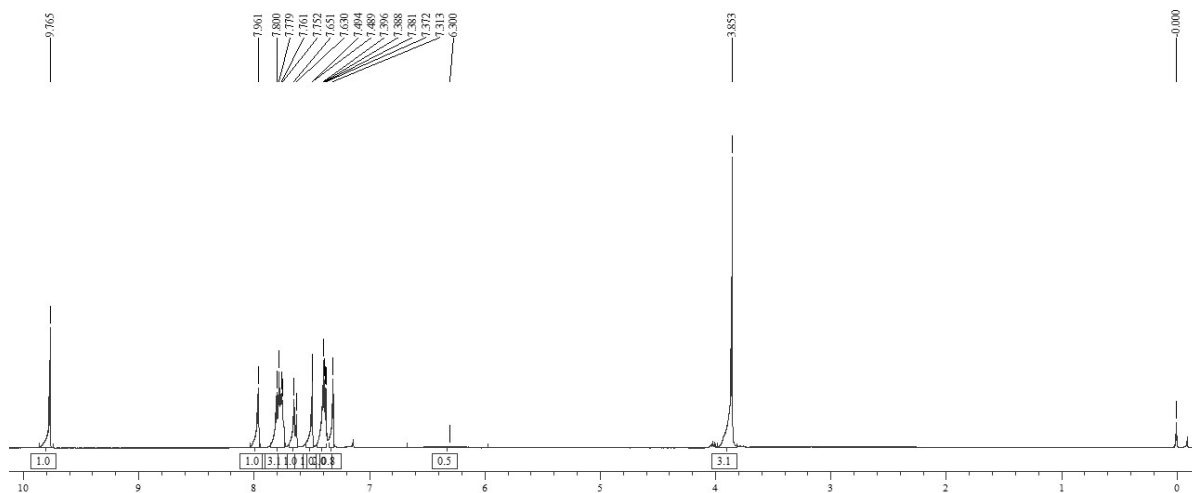
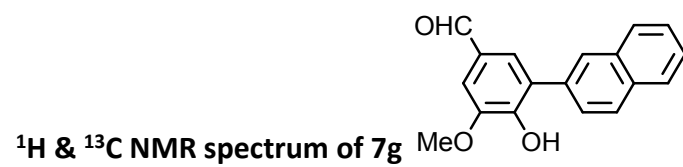


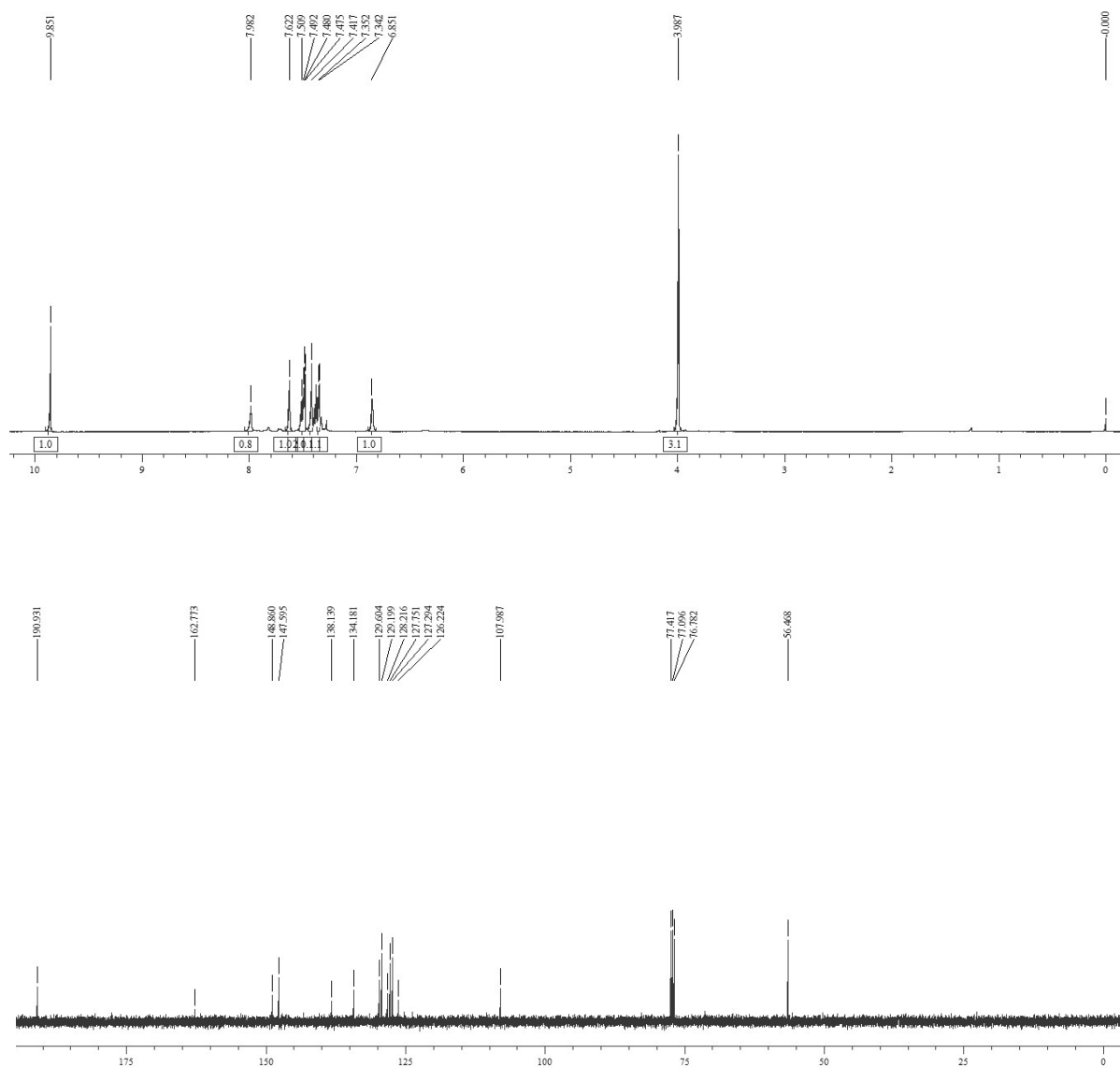


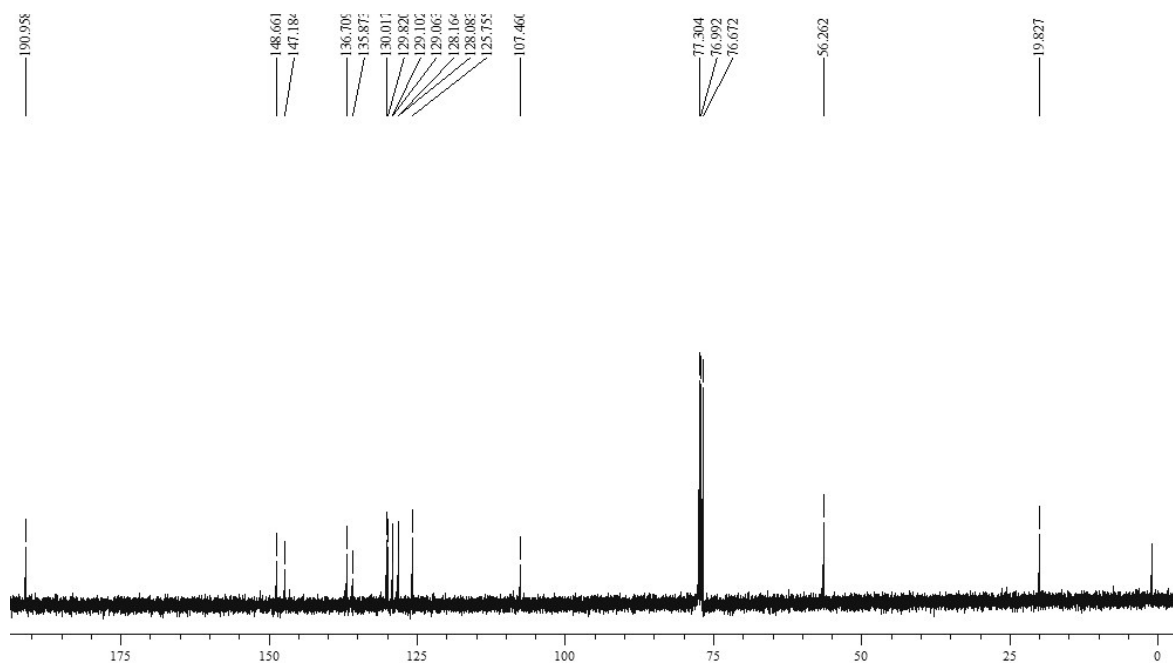
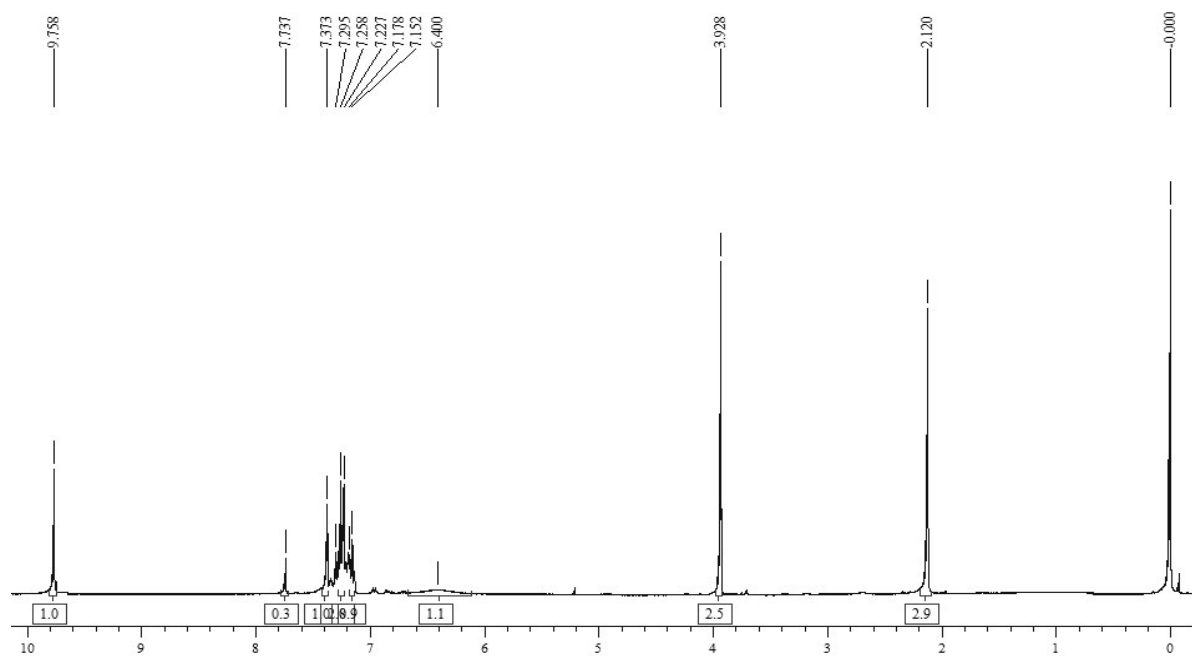
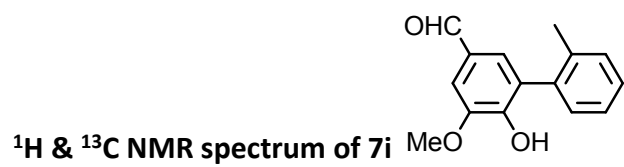


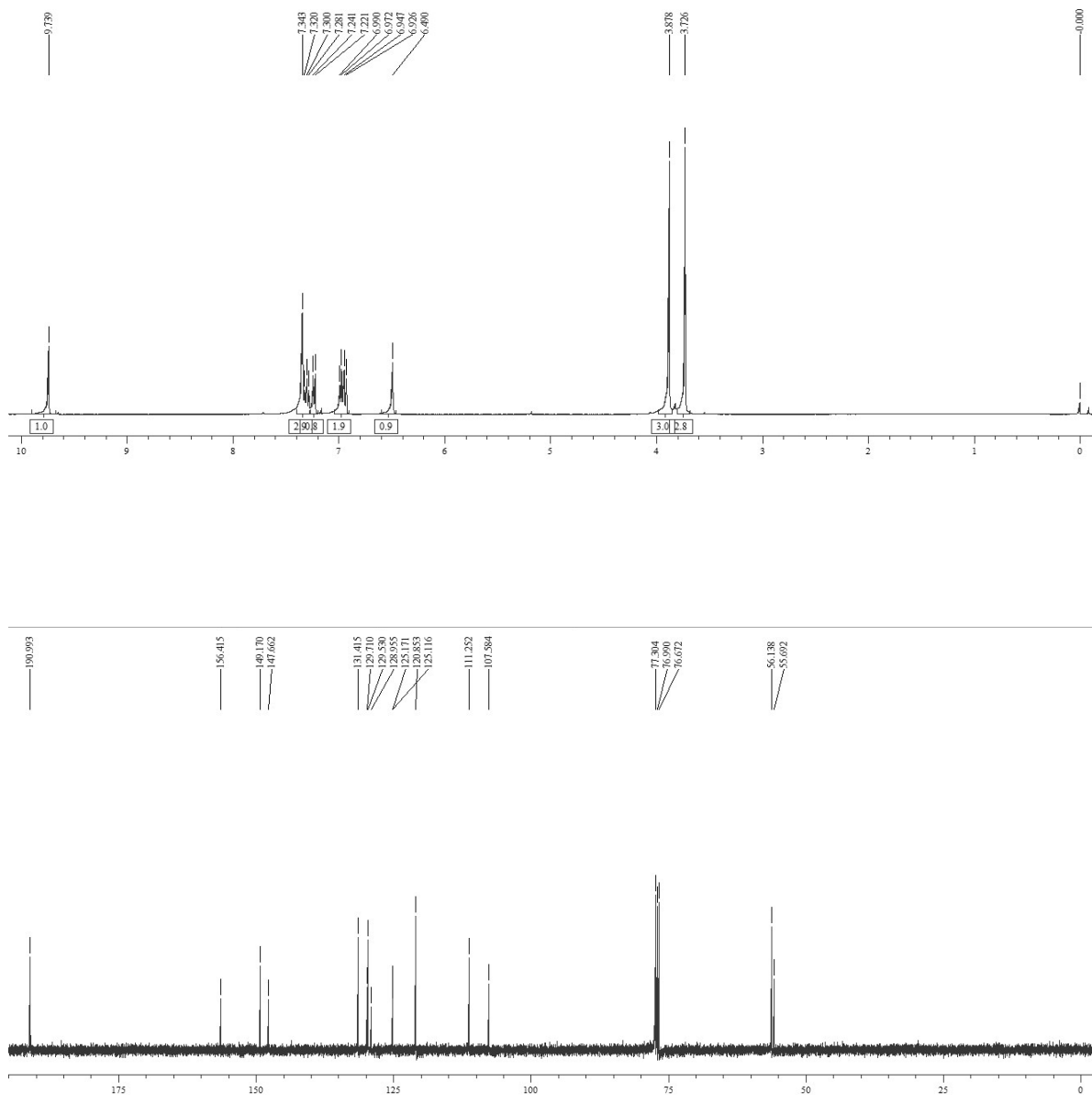
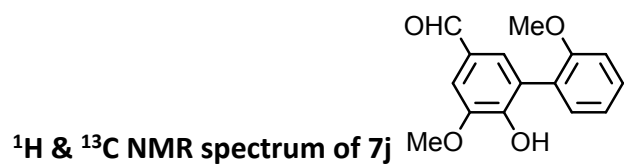


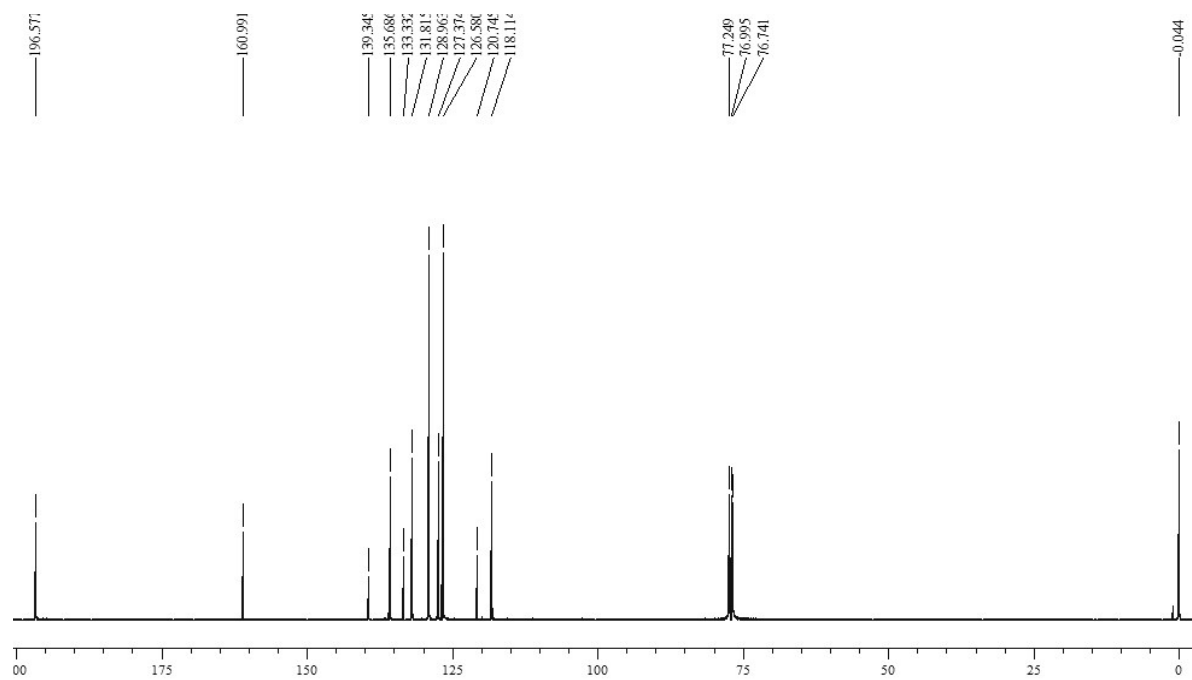
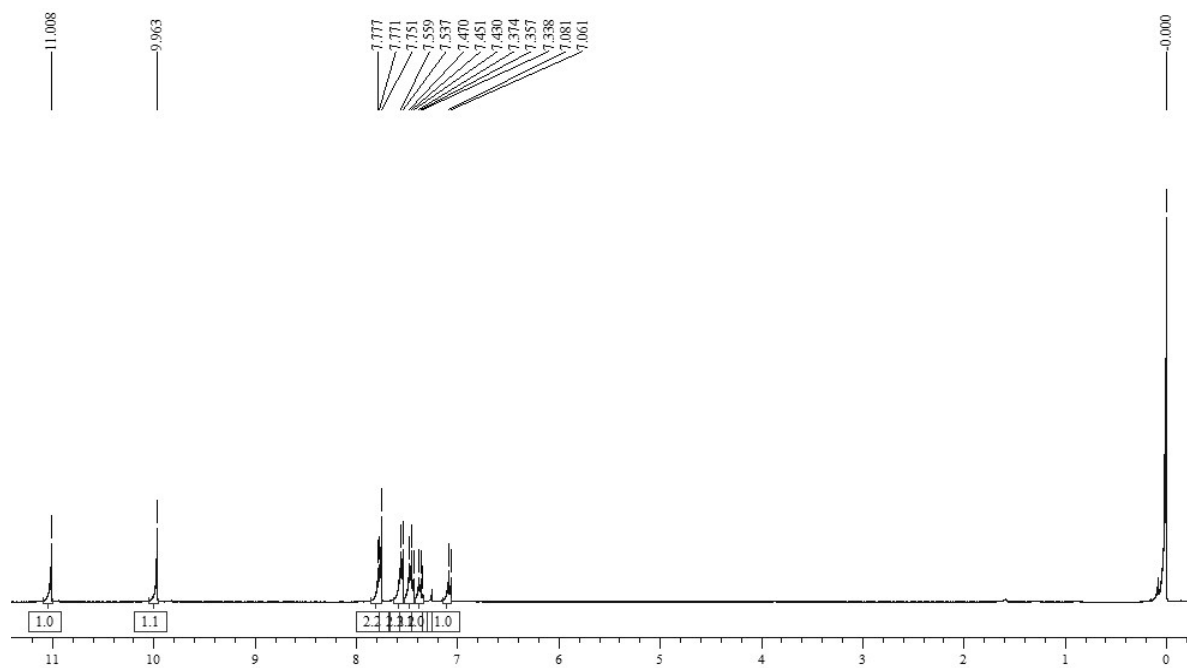
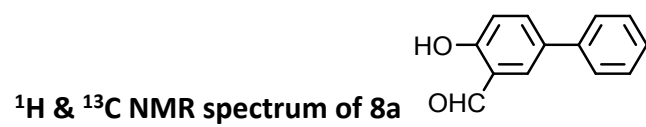


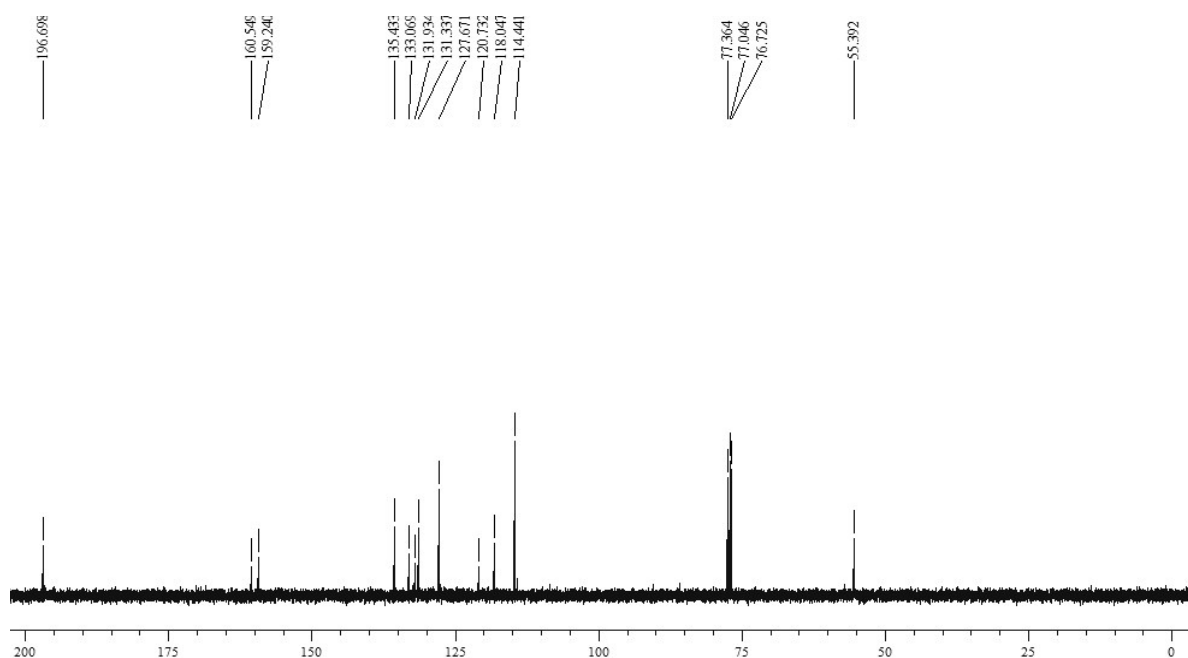
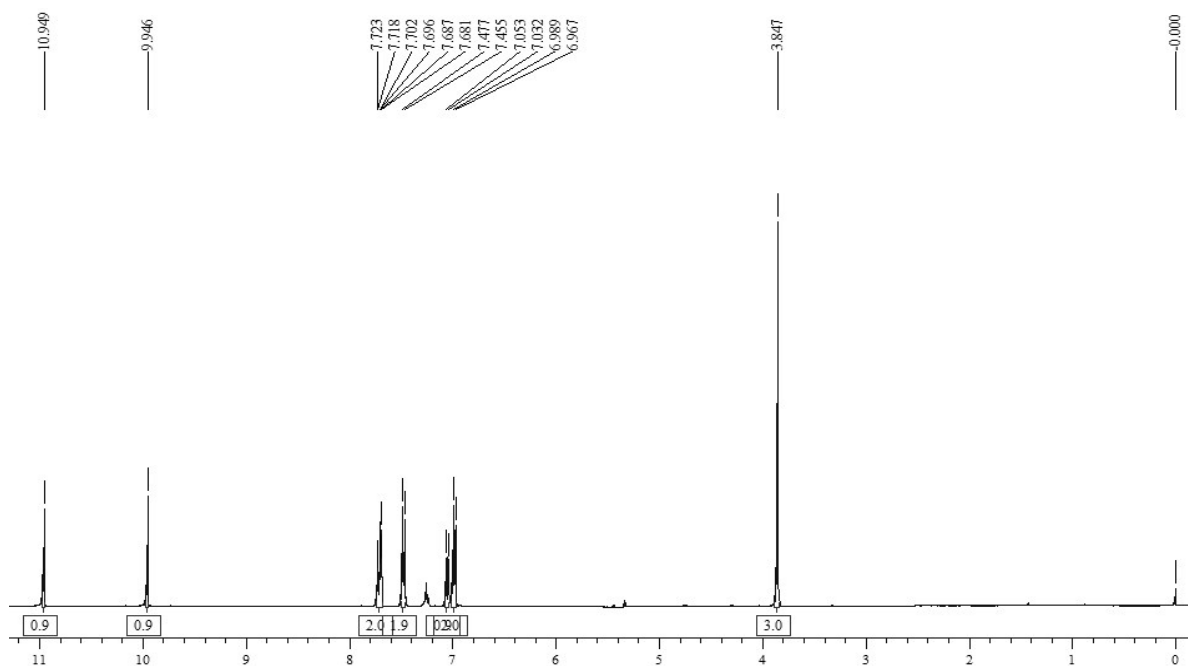
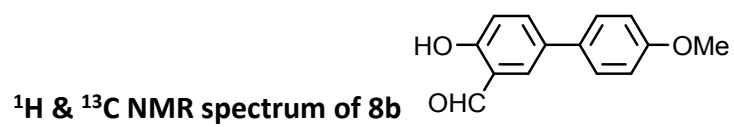


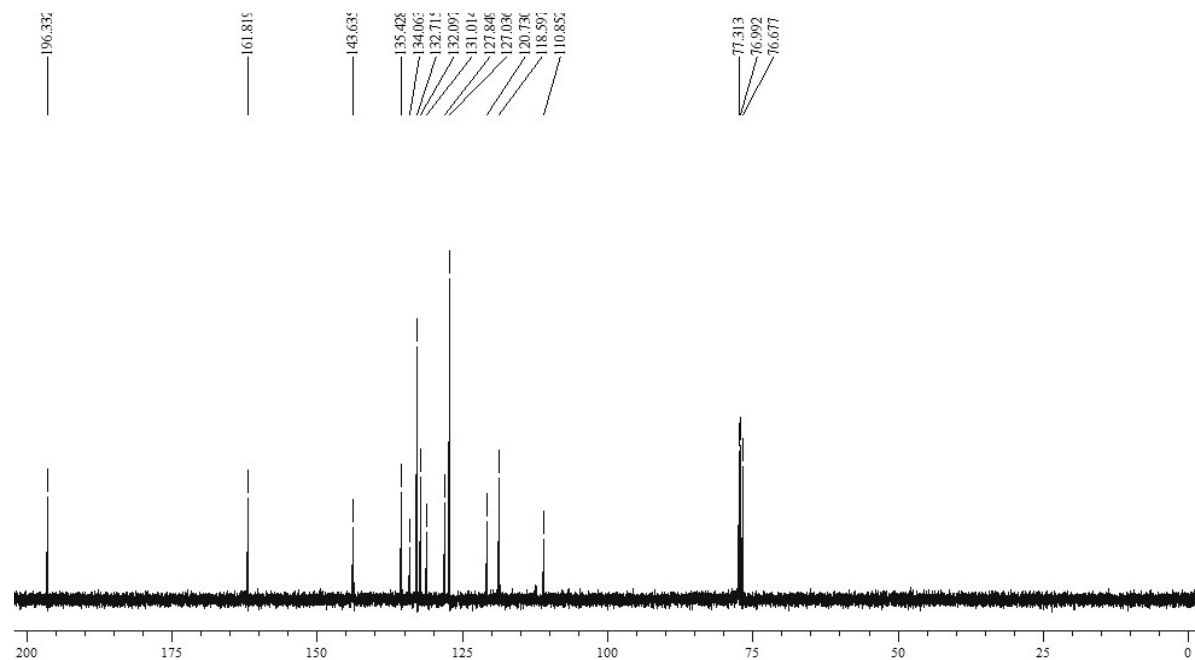
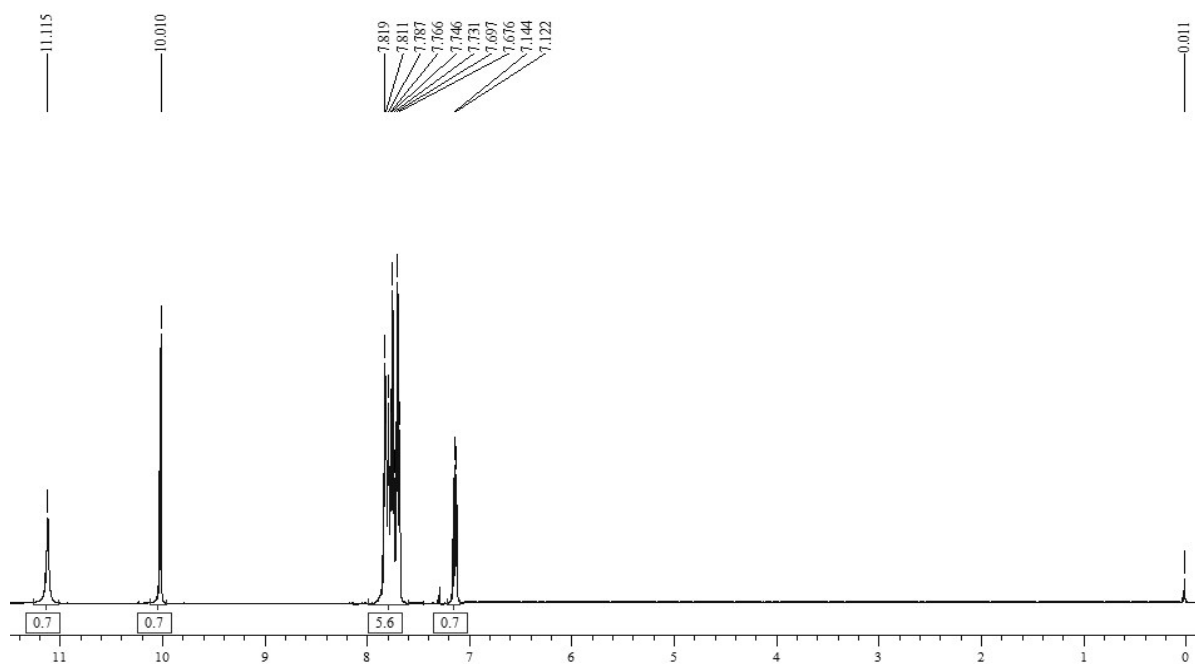
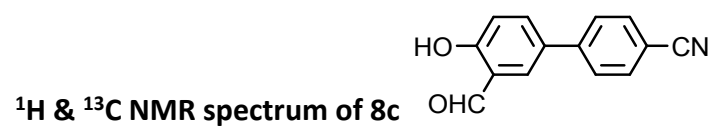




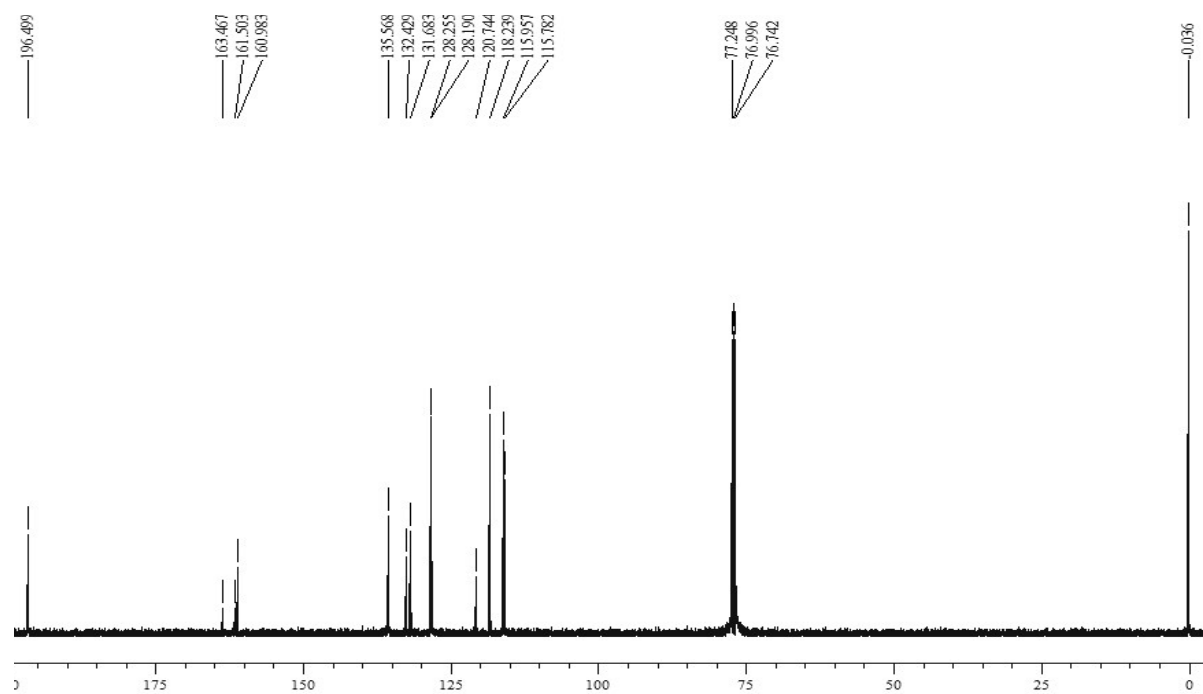
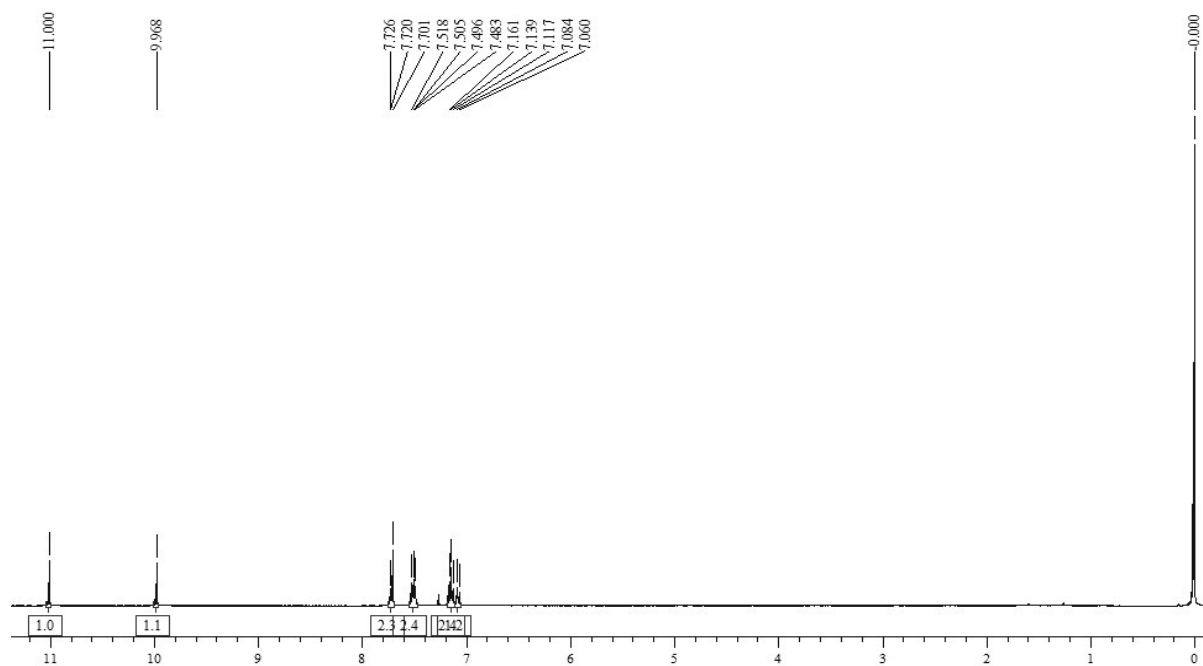
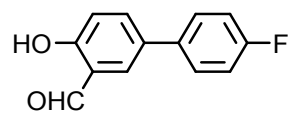


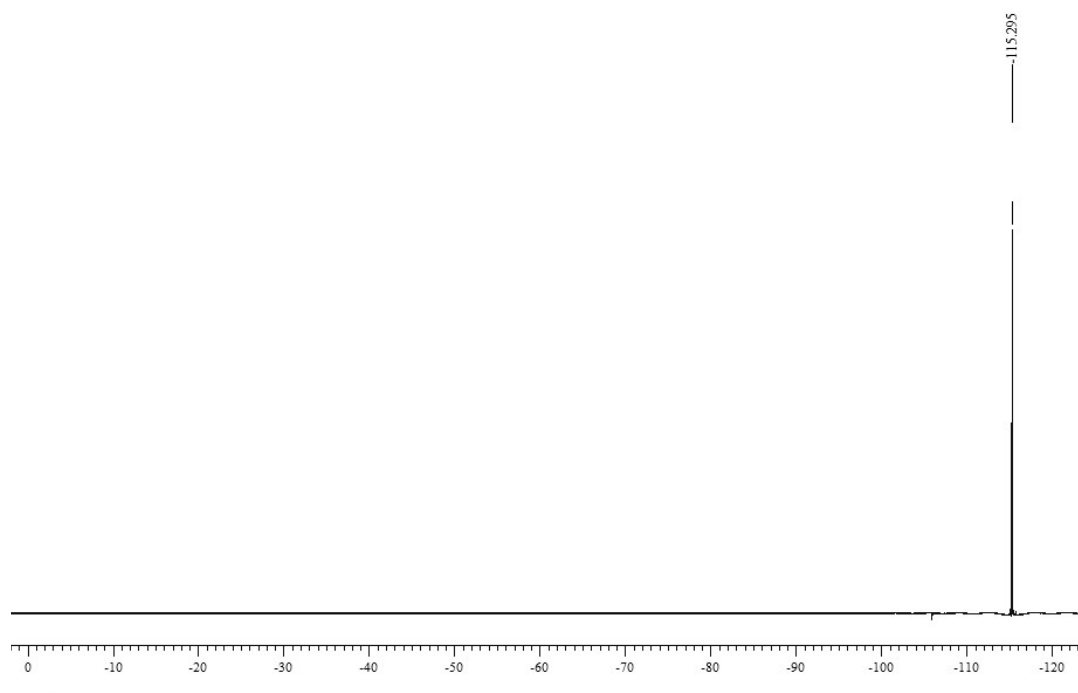
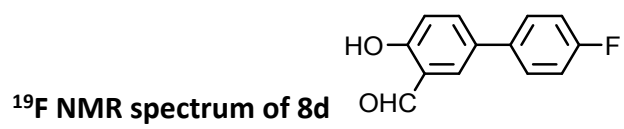


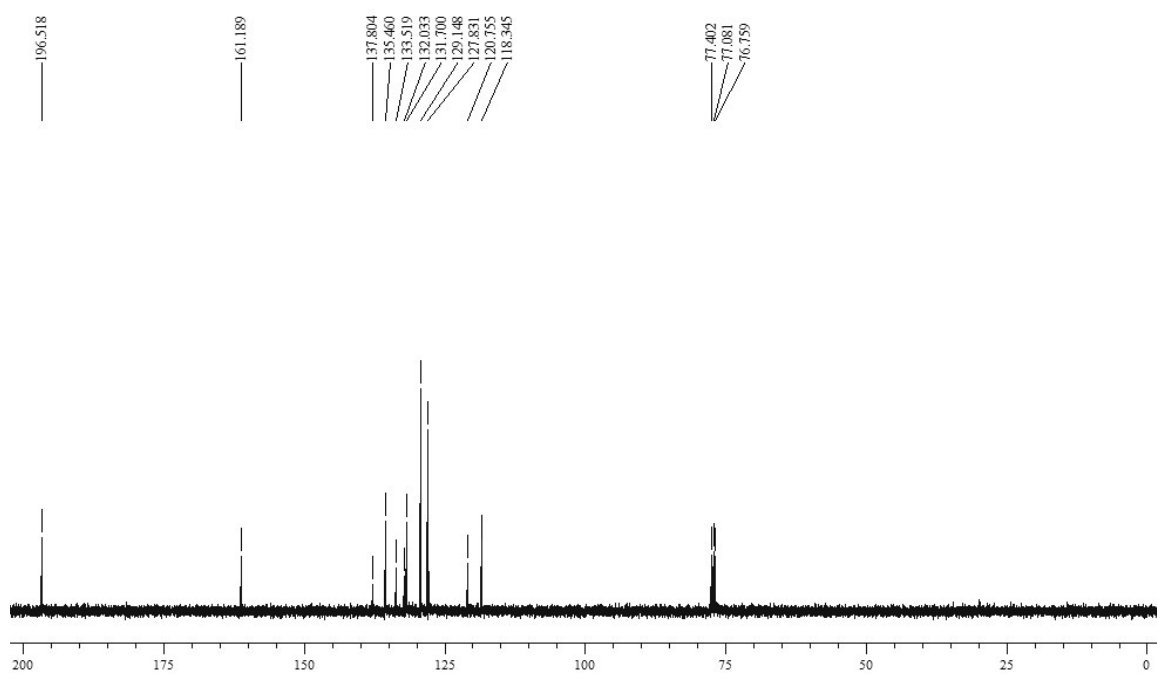
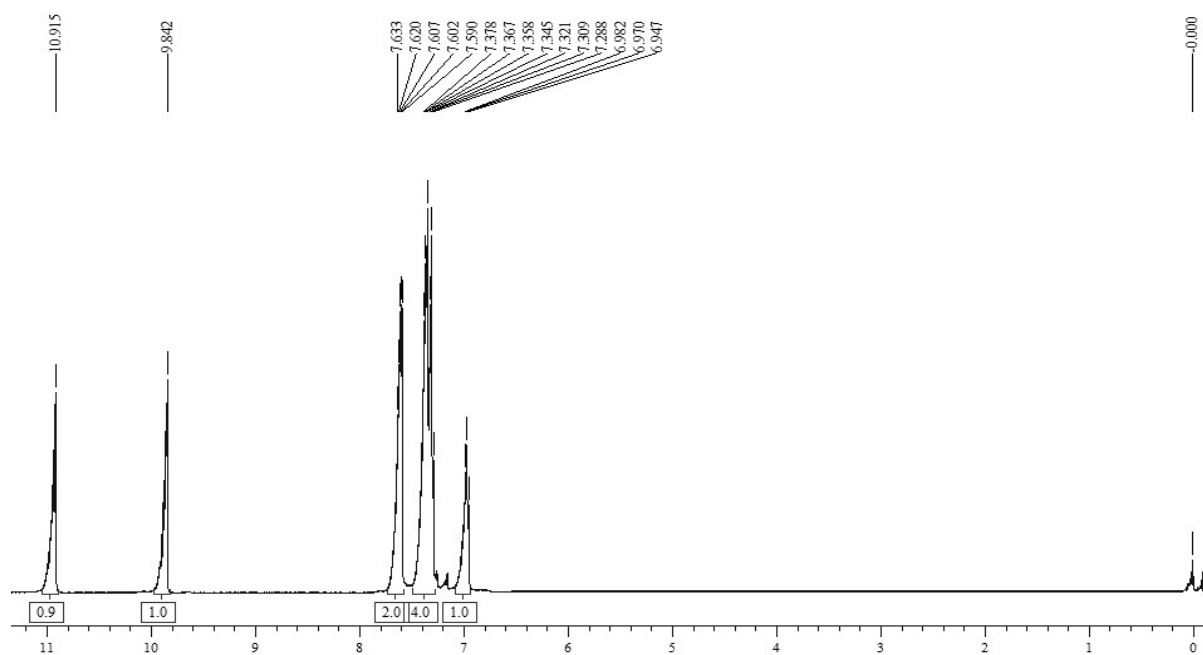




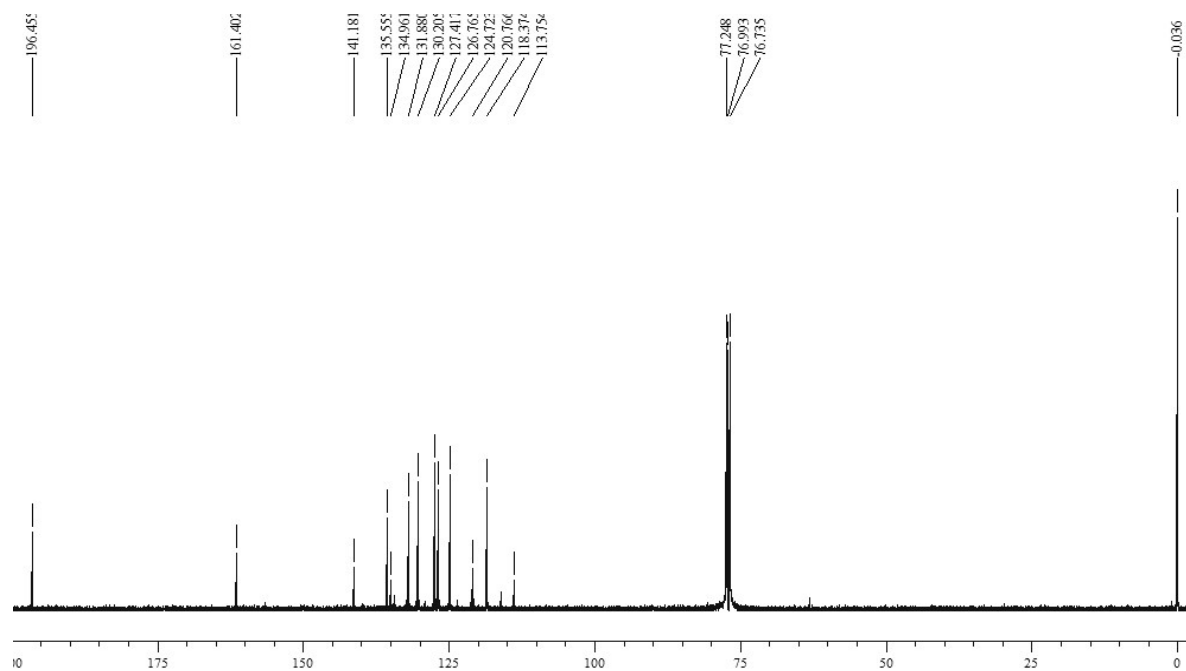
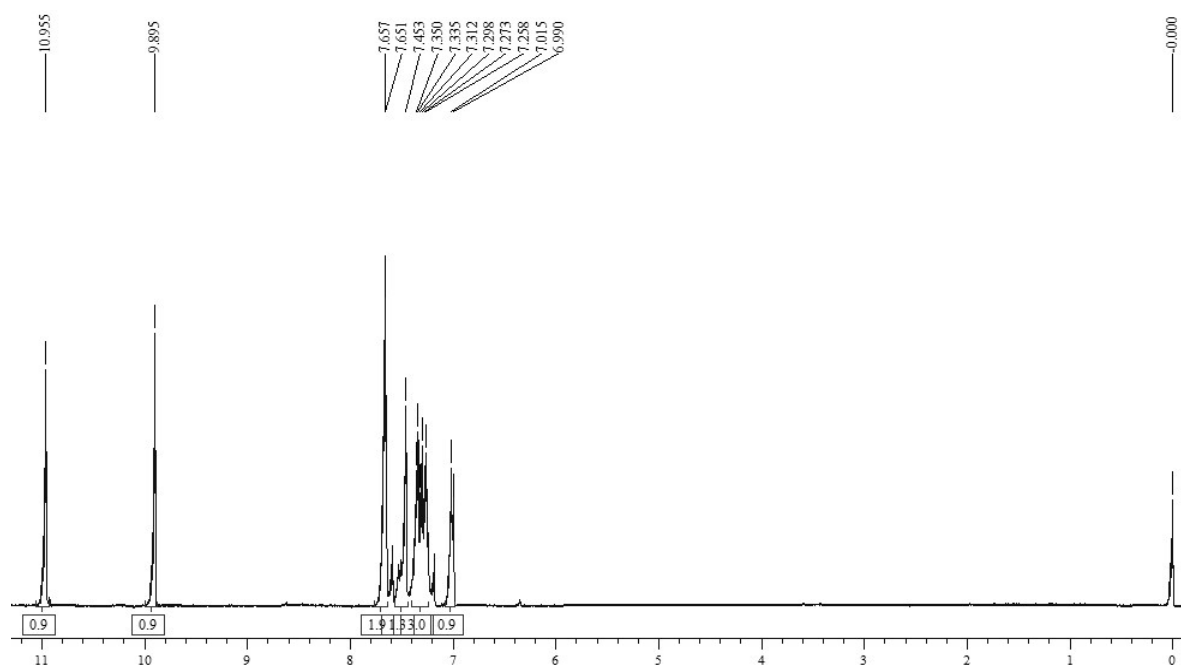
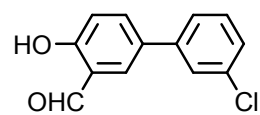
¹H & ¹³C NMR spectrum of 8d



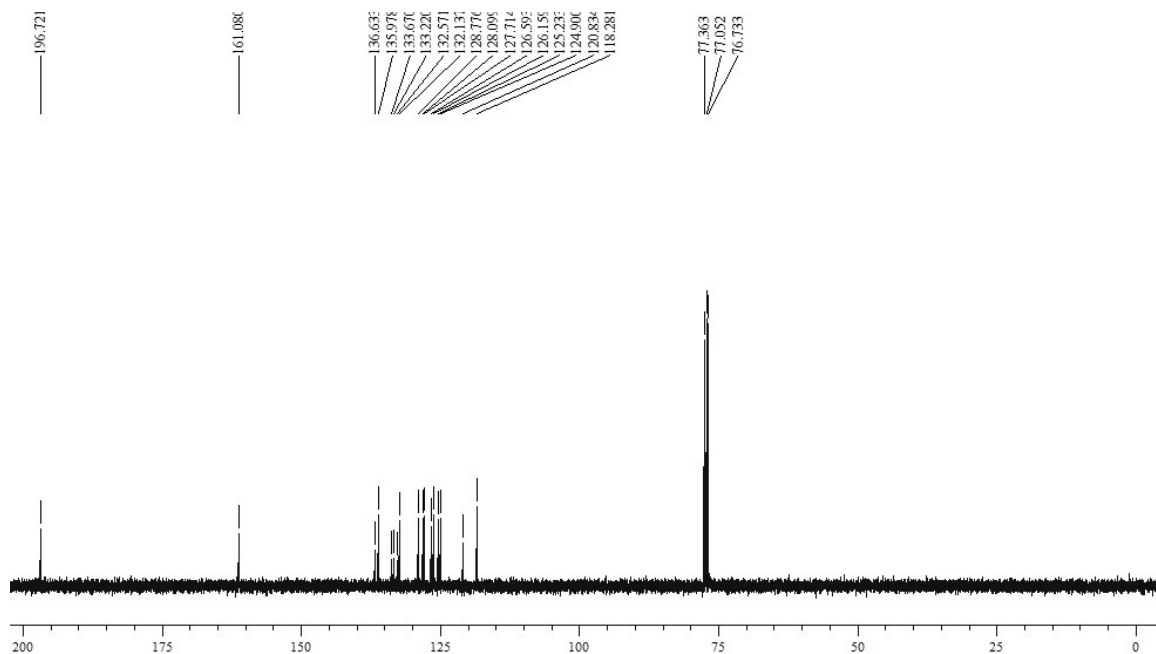
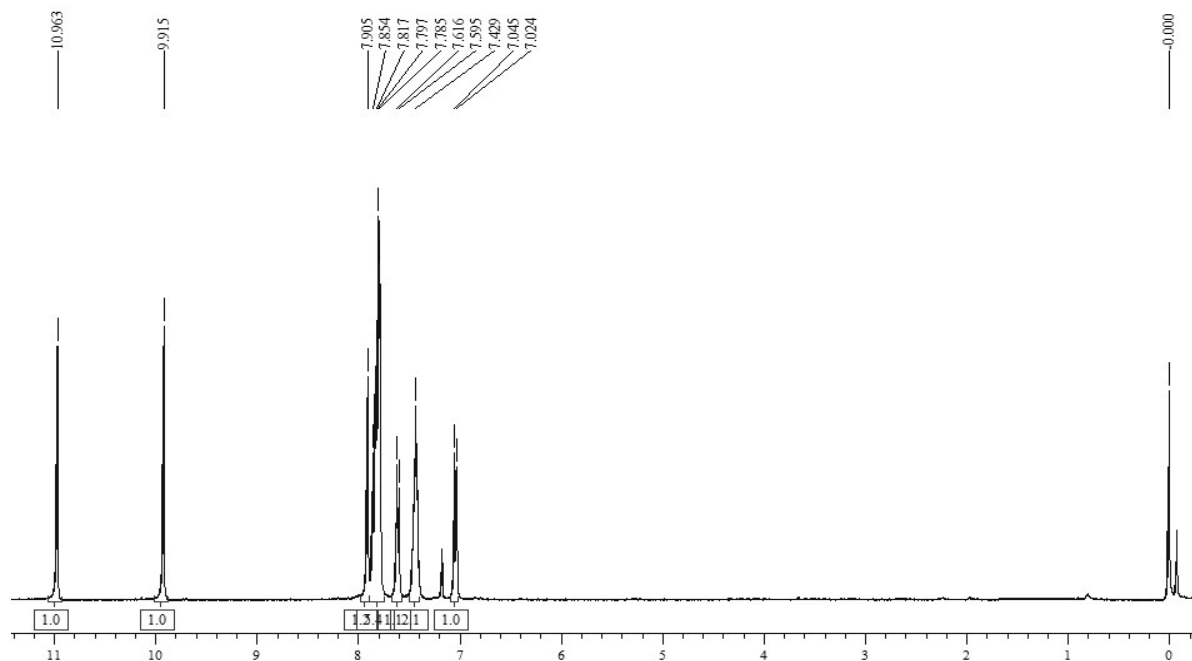
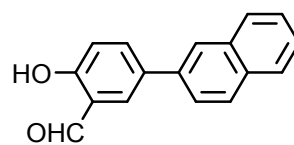




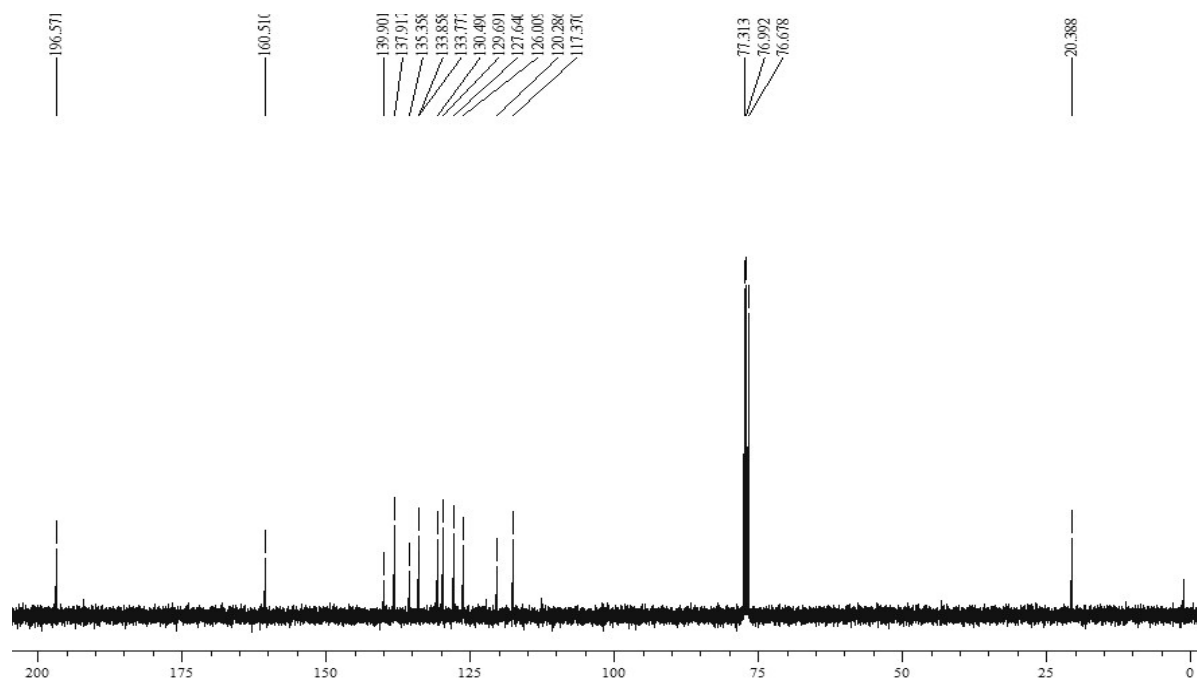
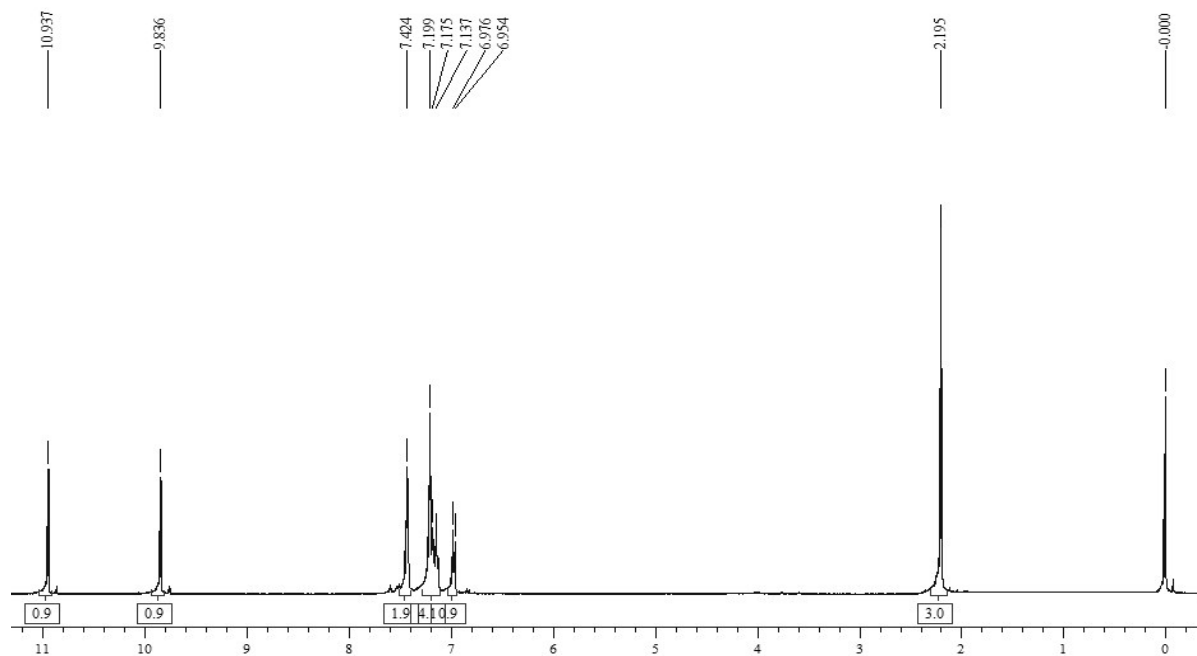
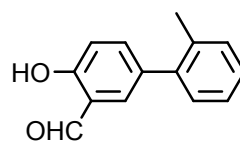
¹H & ¹³C NMR spectrum of 8f

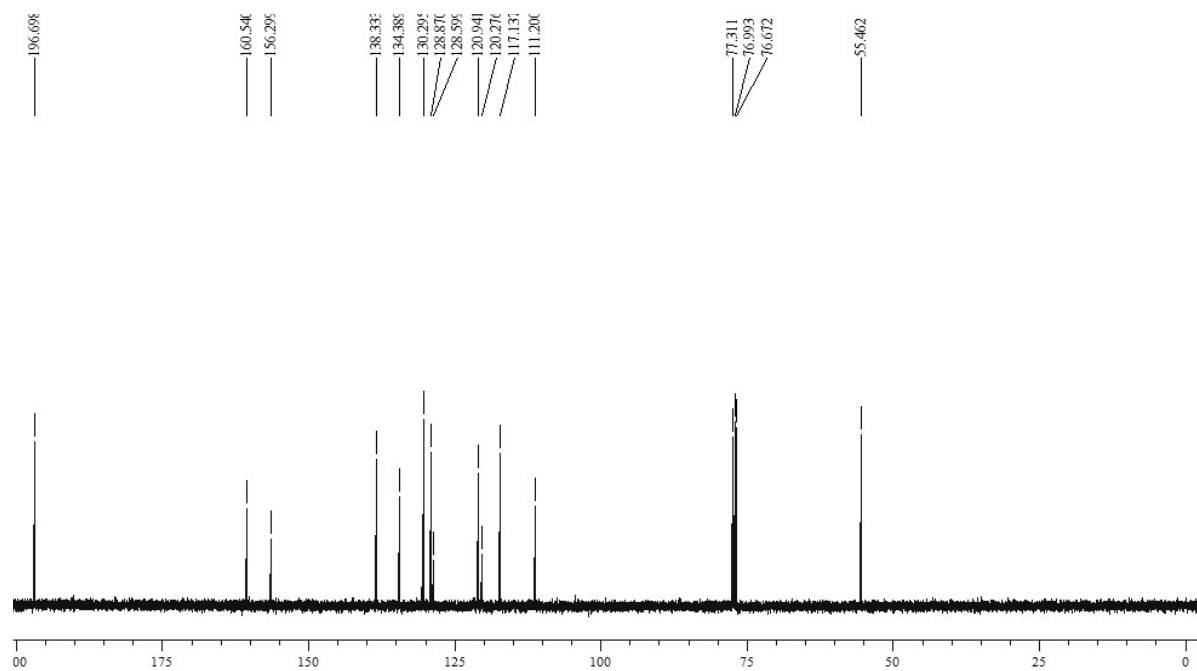
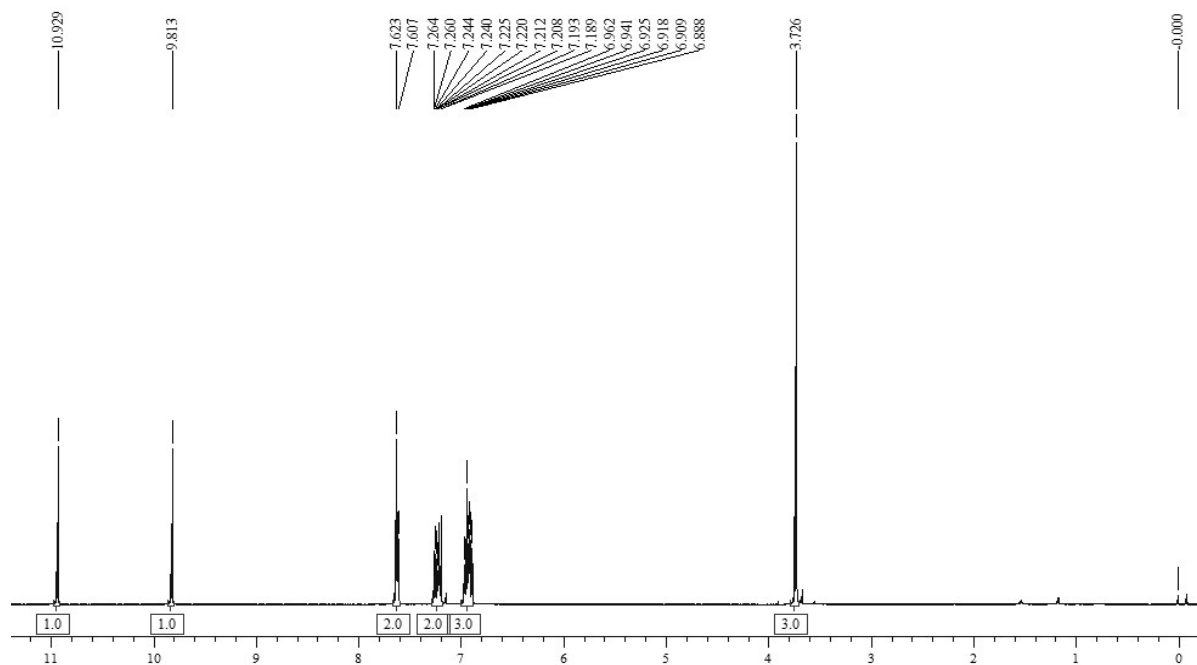
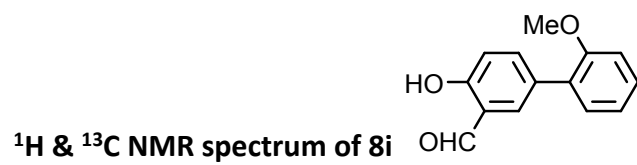


¹H & ¹³C NMR spectrum of 8g

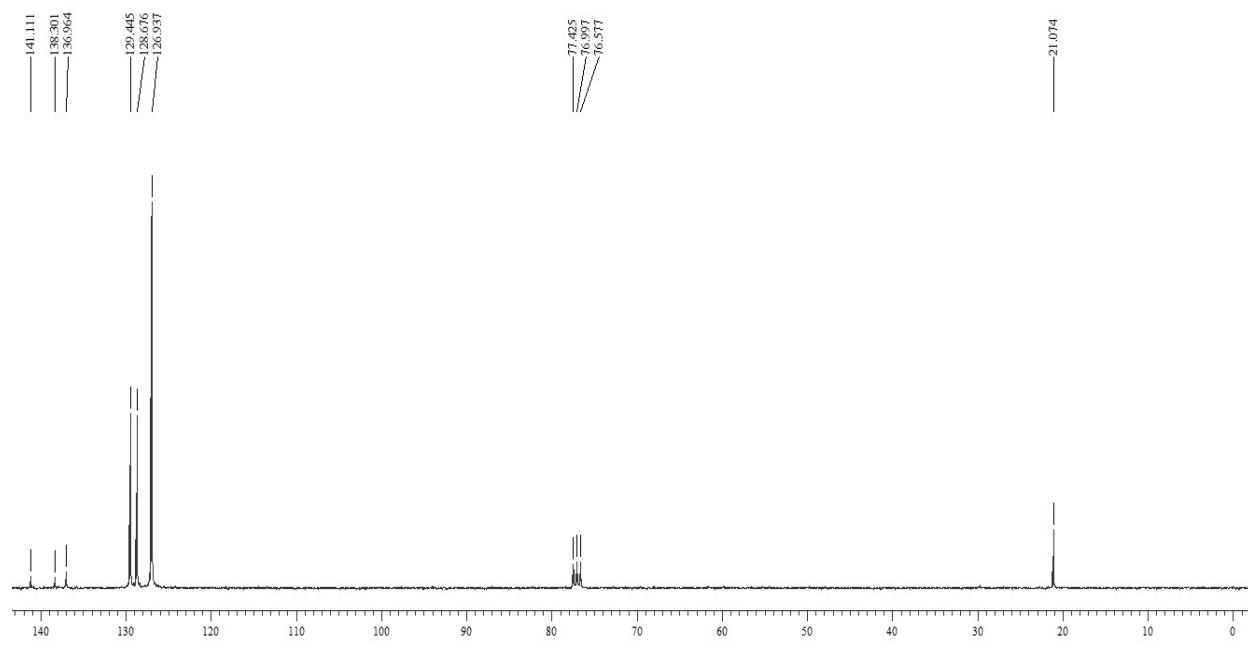
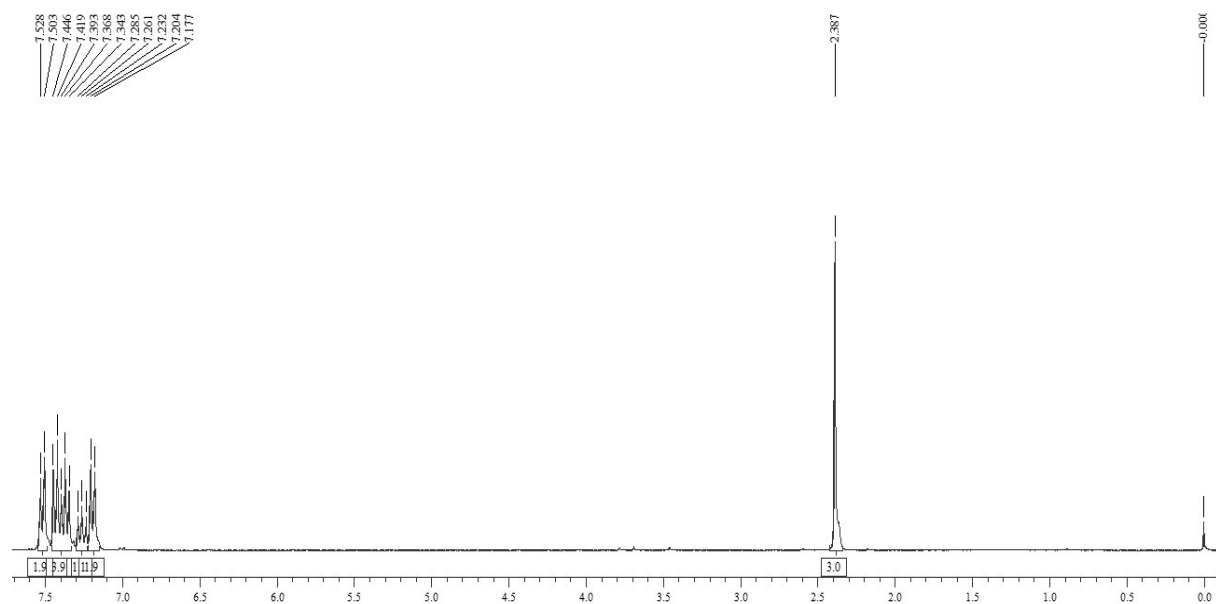
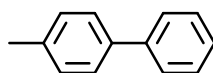


^1H & ^{13}C NMR spectrum of 8h

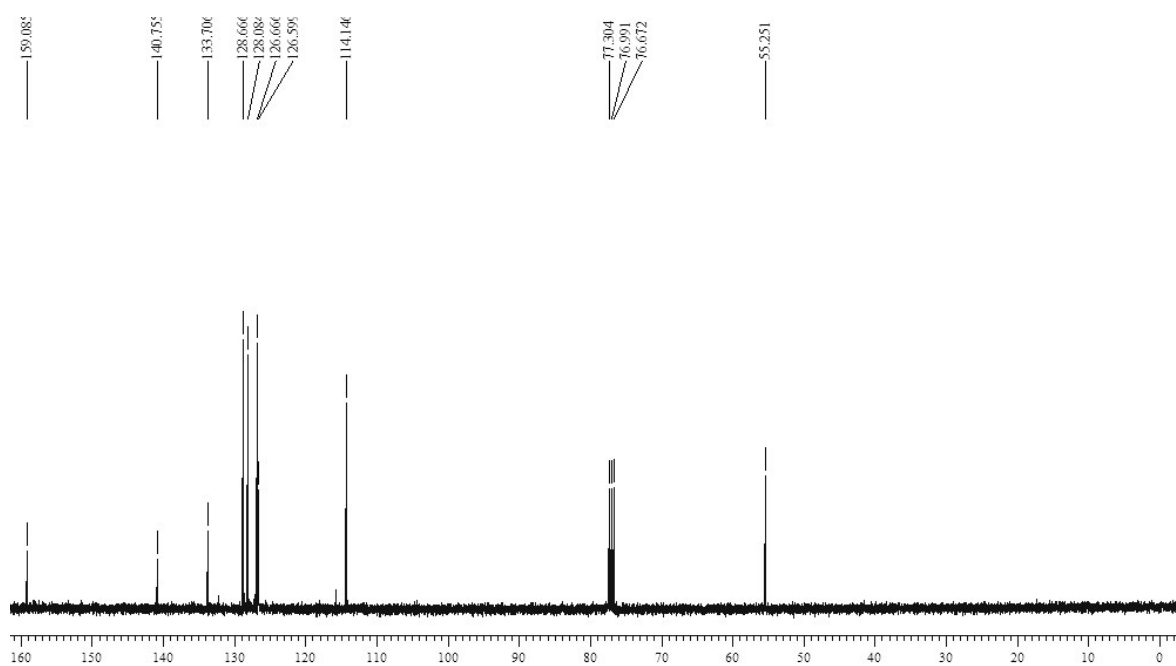
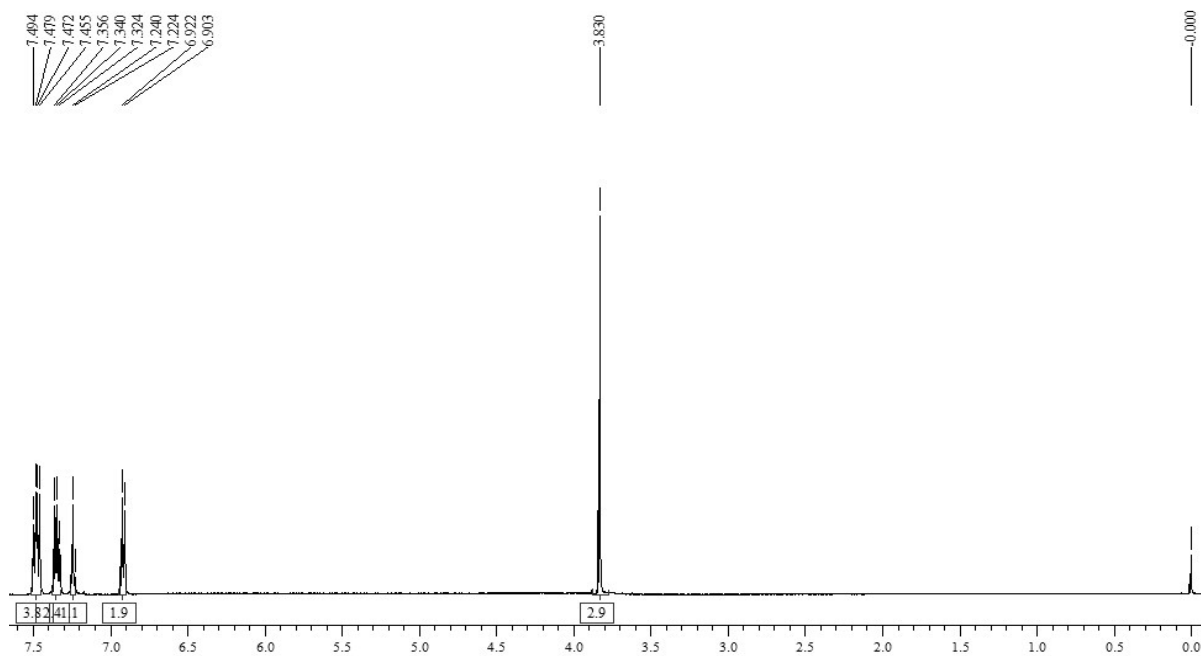
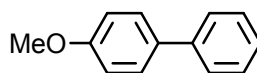




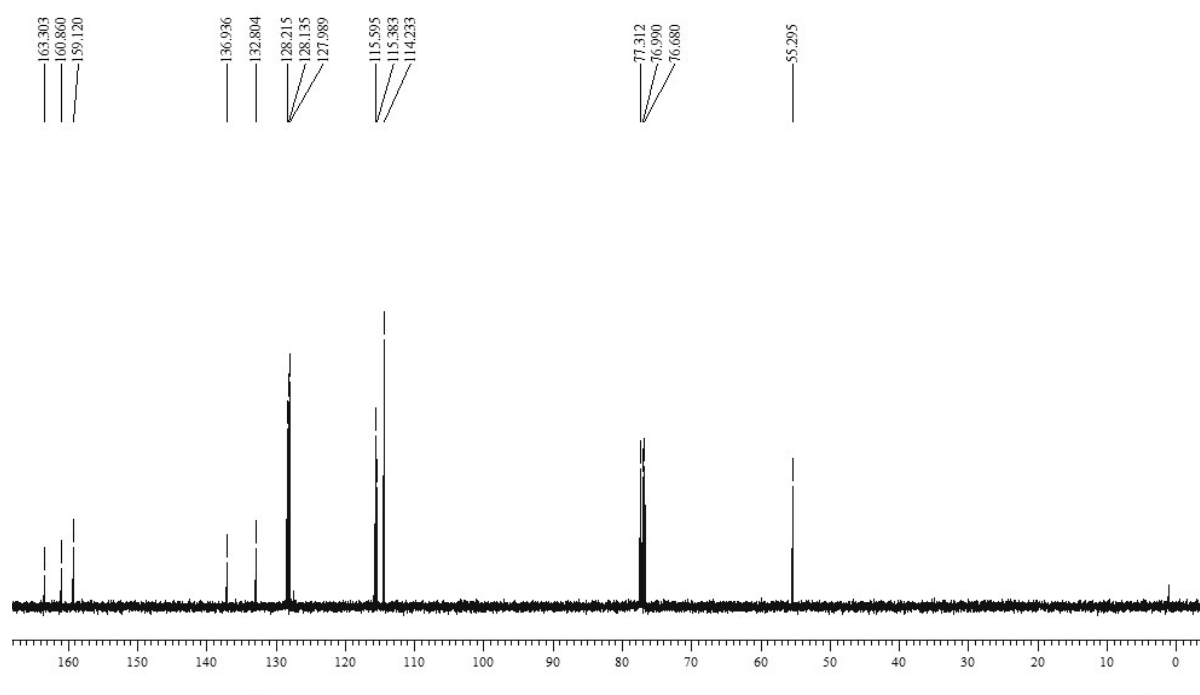
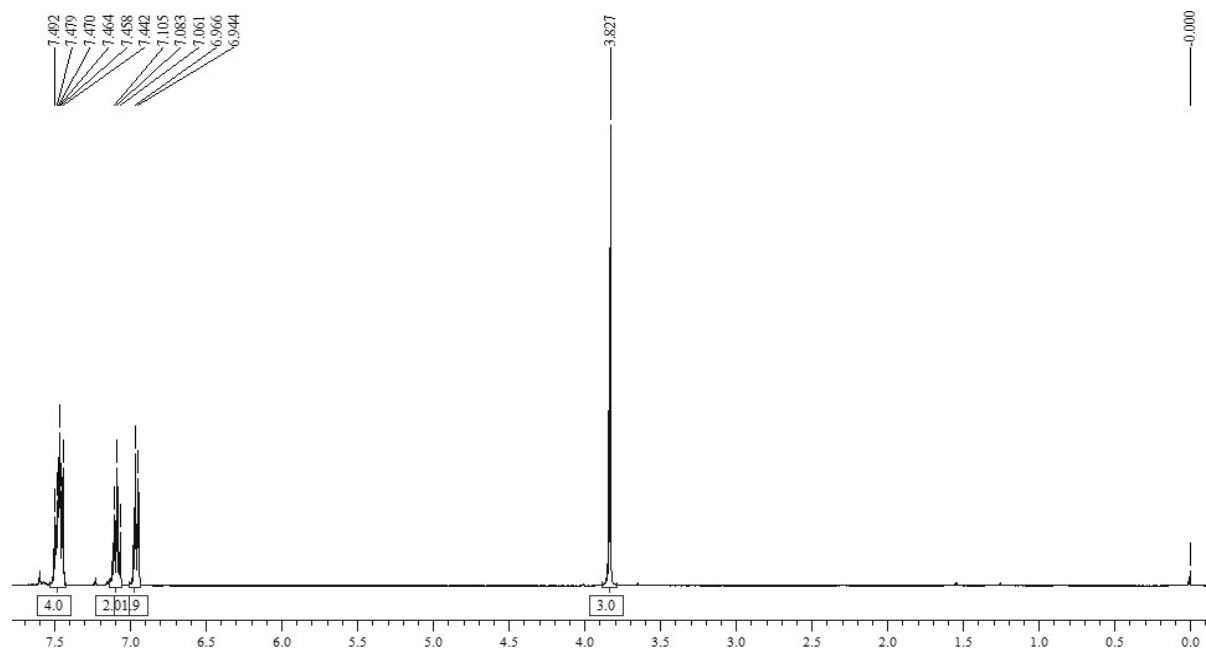
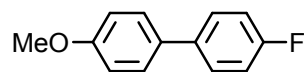
¹H & ¹³C NMR Spectrum of 9a



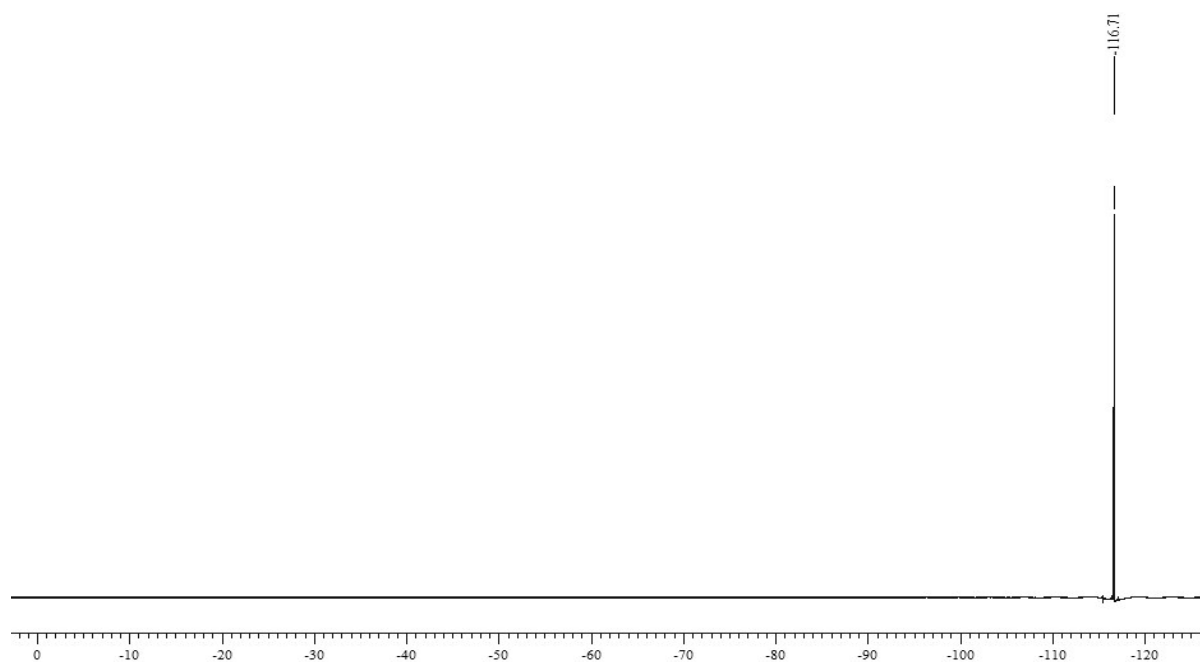
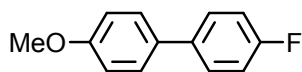
¹H & ¹³C NMR spectrum of 9b



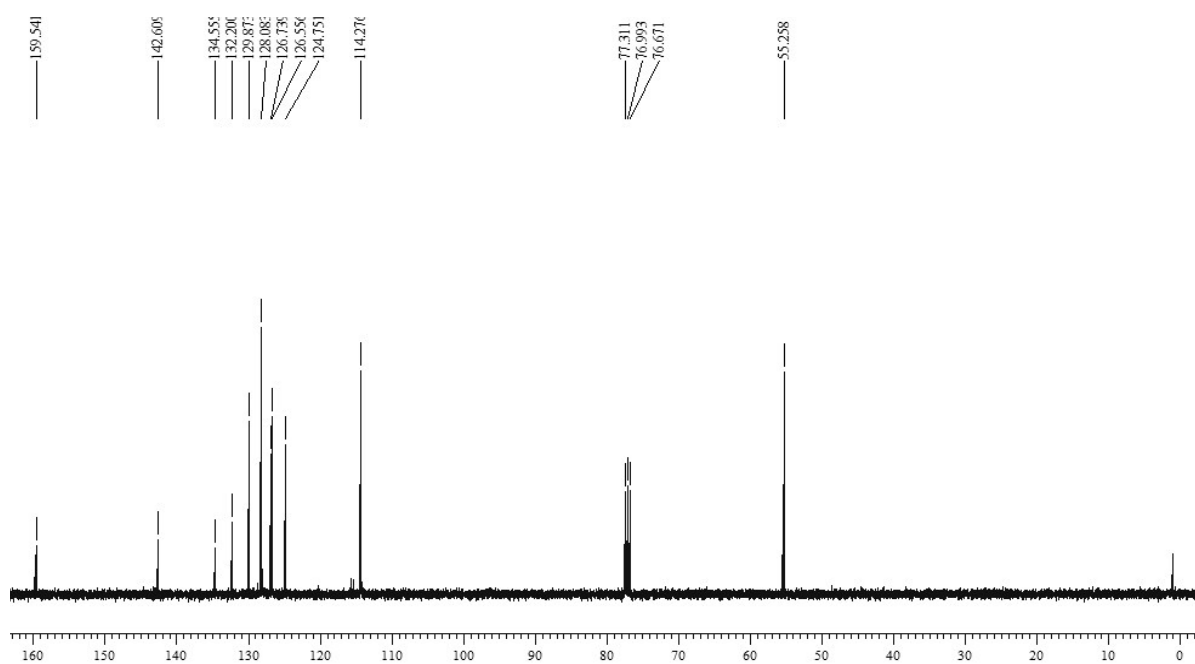
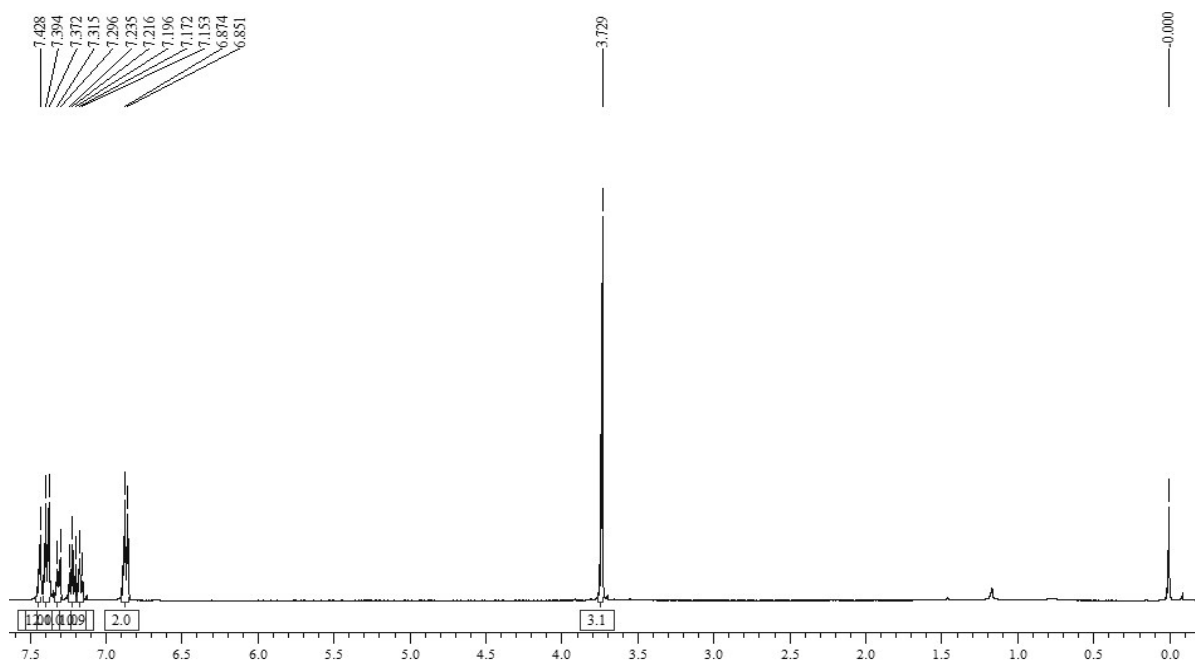
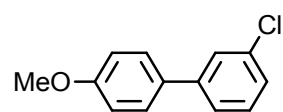
^1H & ^{13}C NMR spectrum of 9c

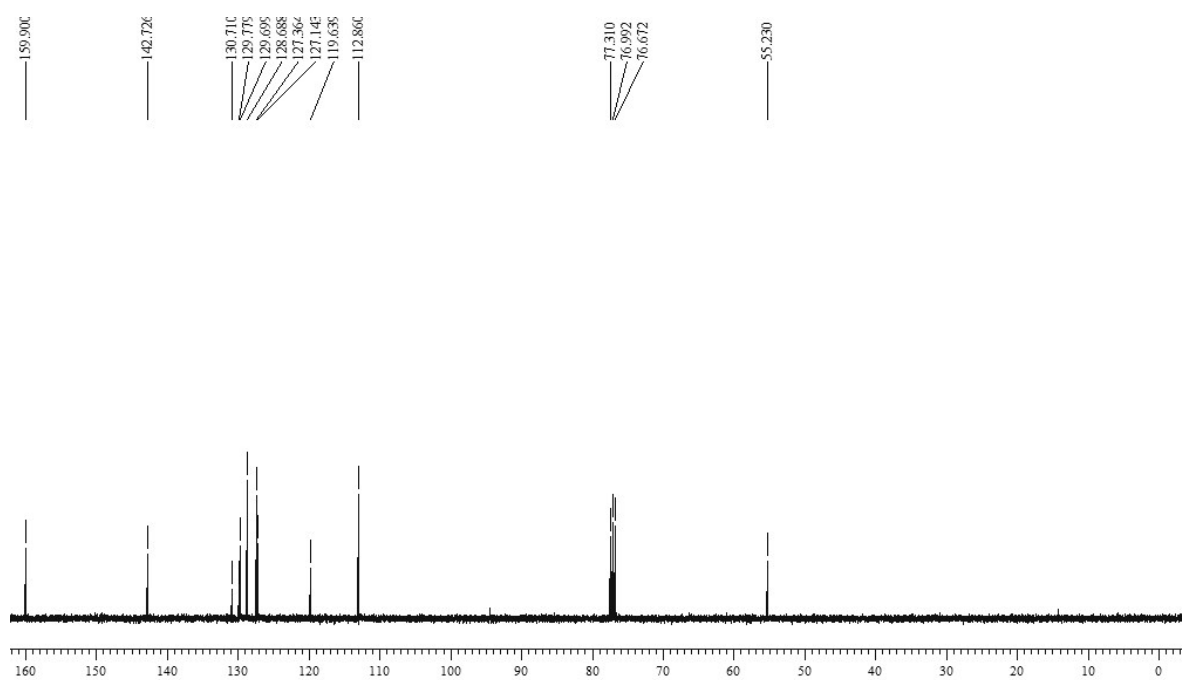
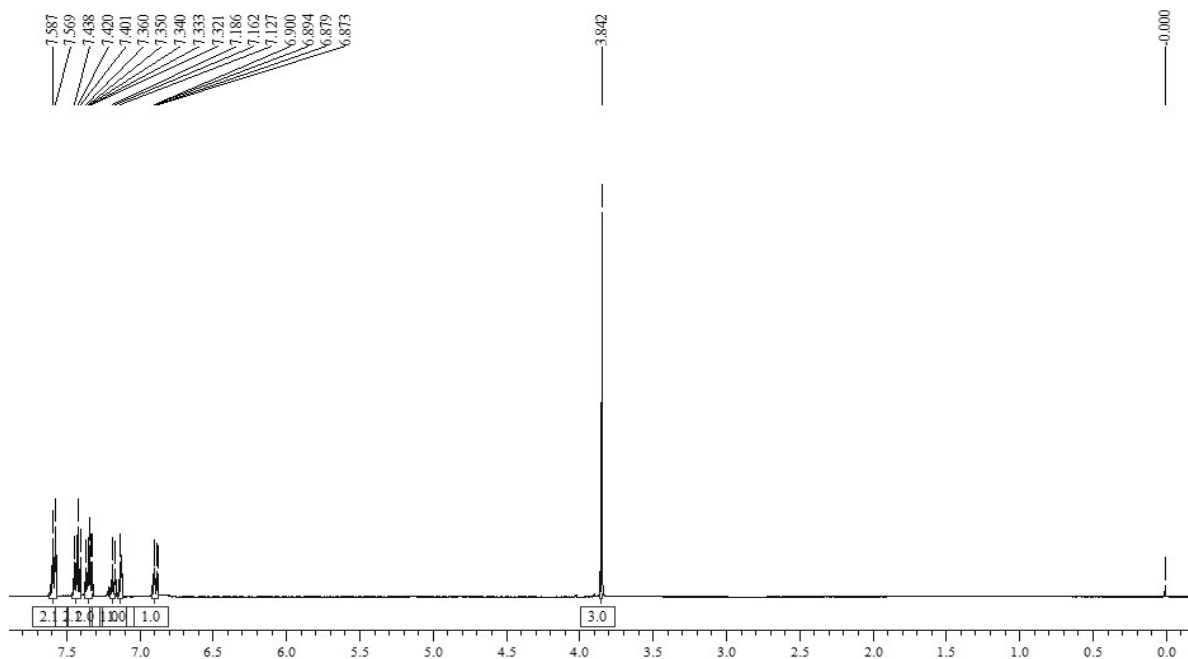


^{19}F NMR spectrum of 9c

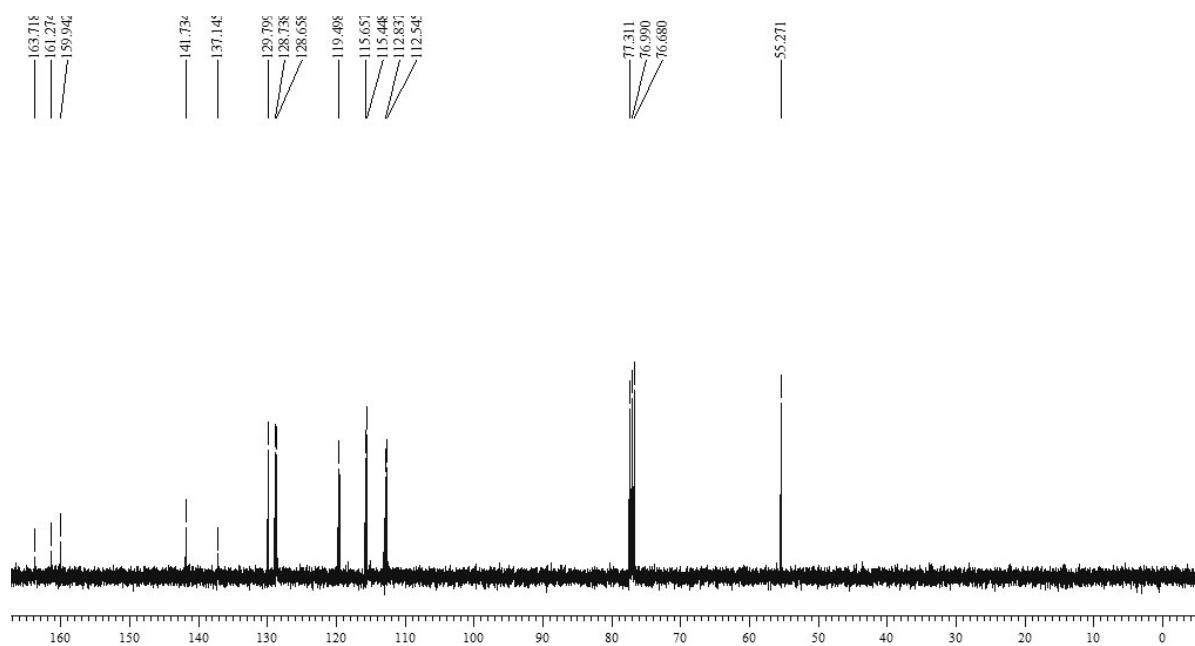
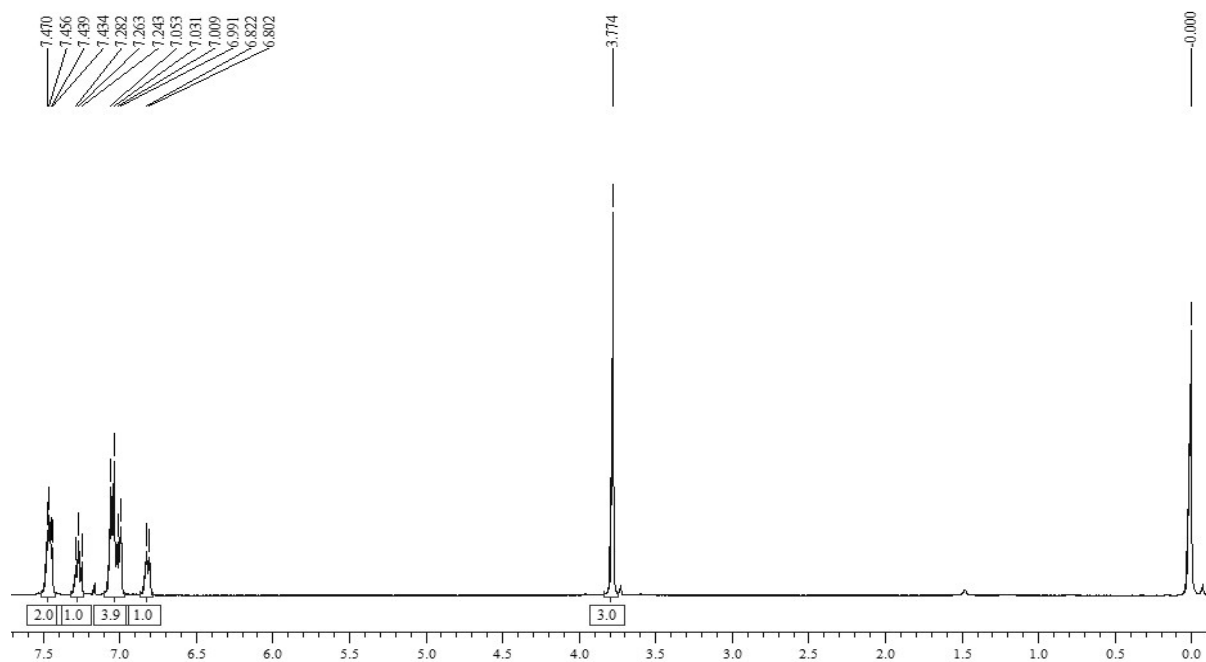
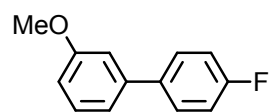


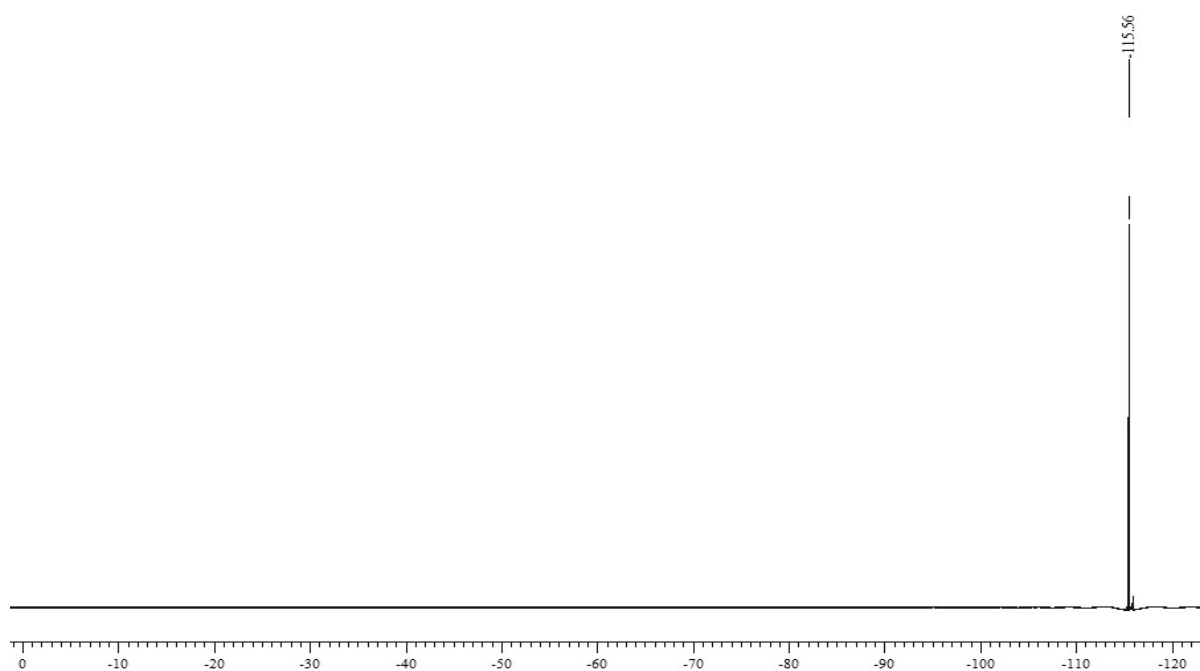
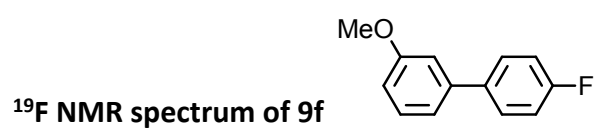
¹H & ¹³C NMR spectrum of 9d



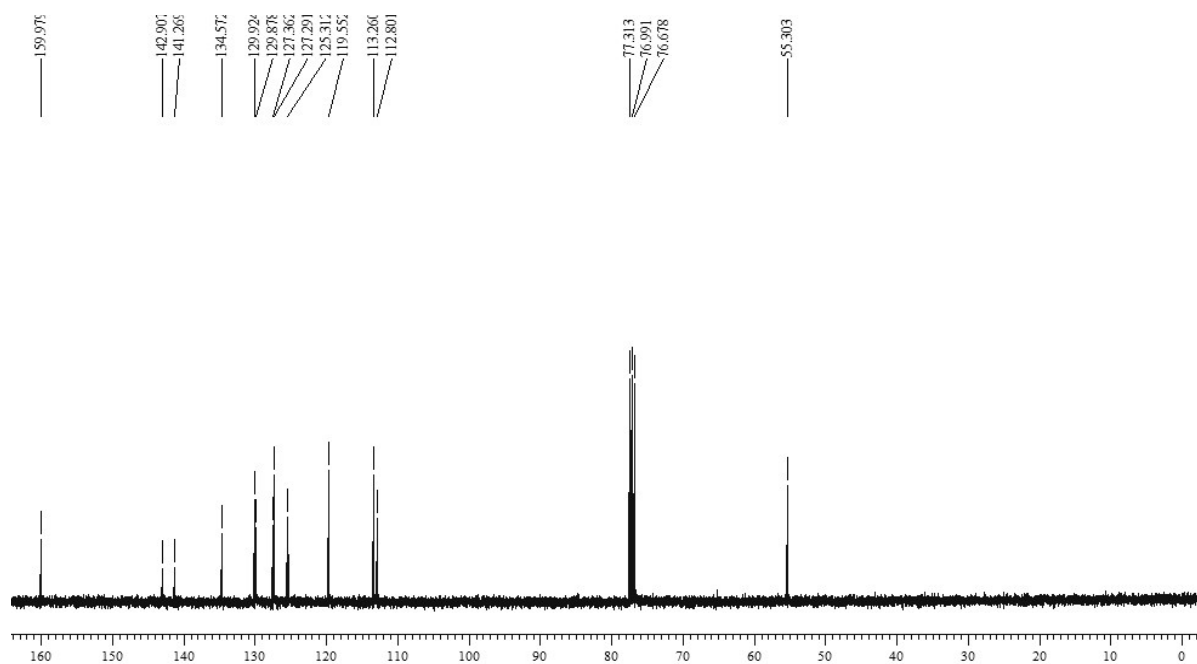
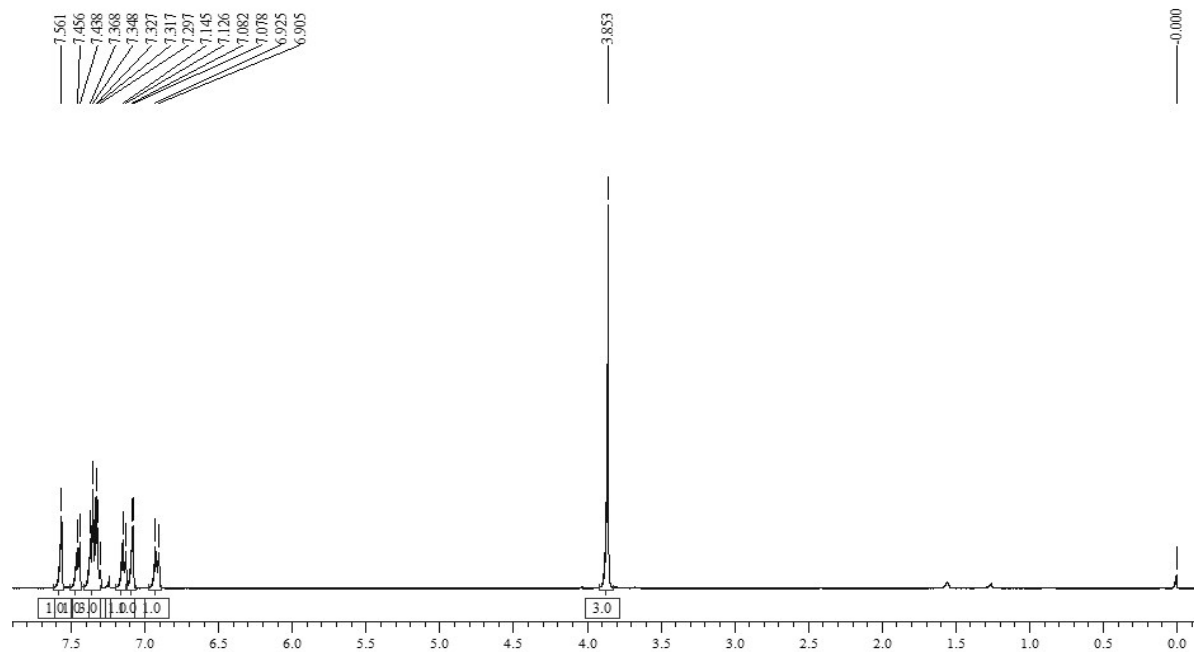
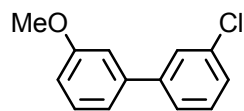


¹H & ¹³C NMR spectrum of 9f

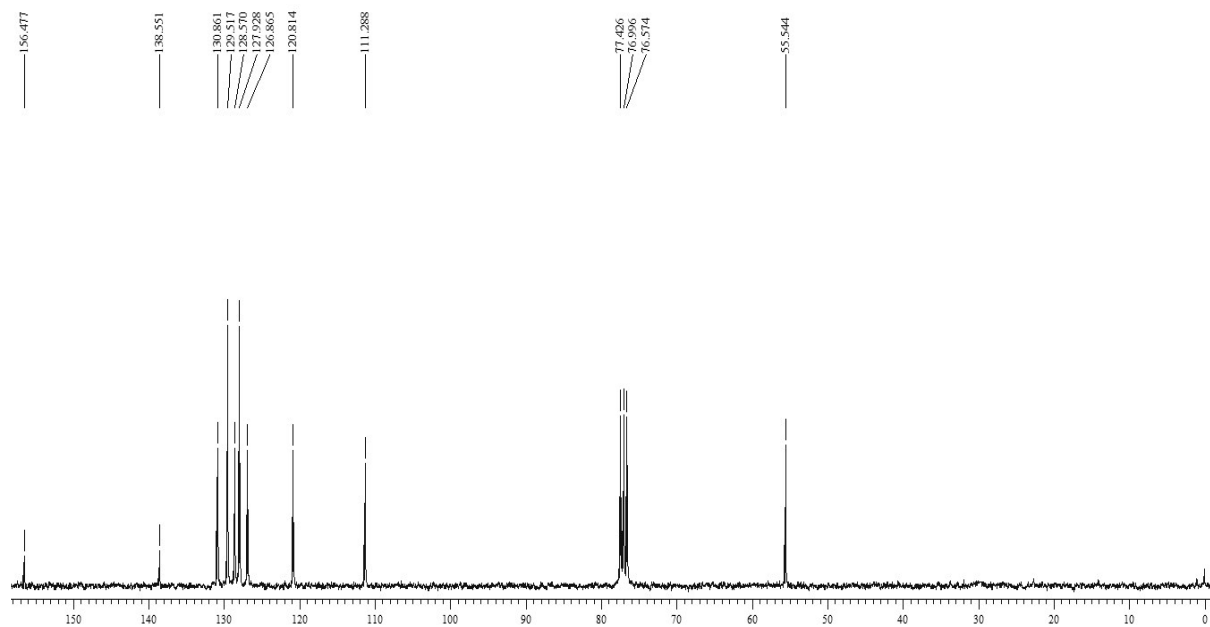
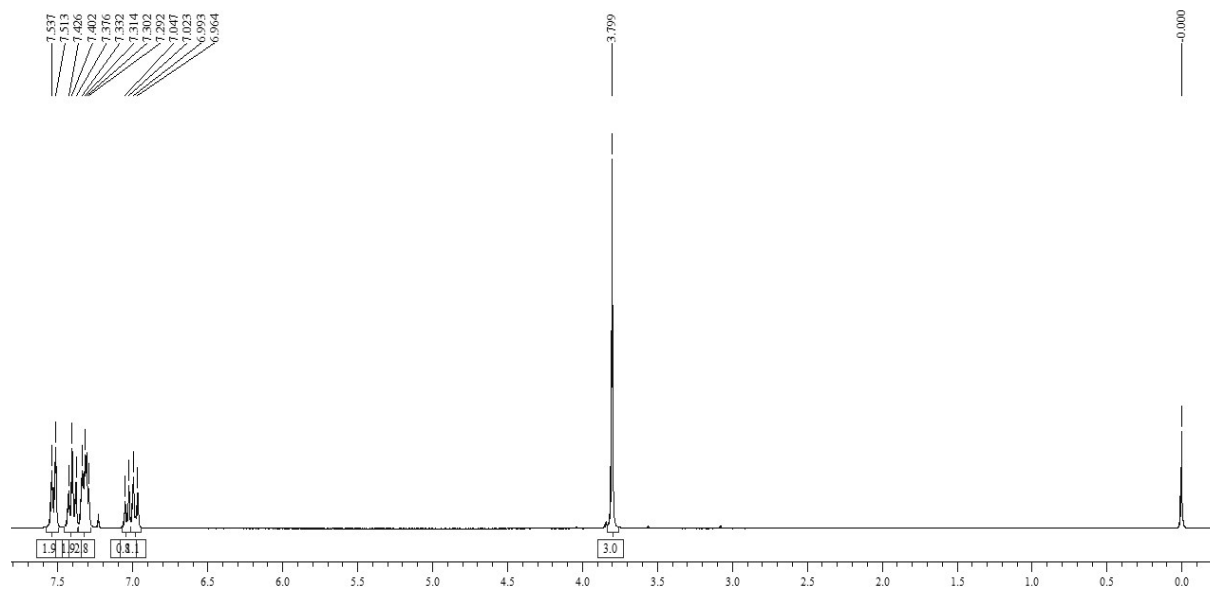
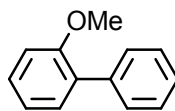




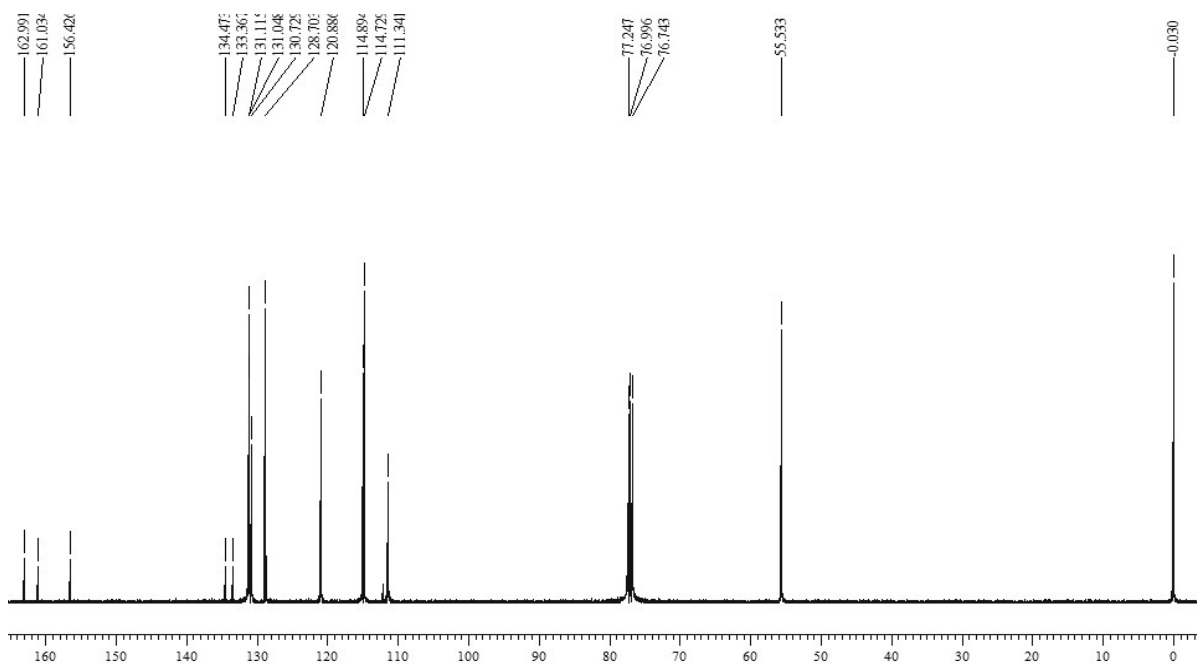
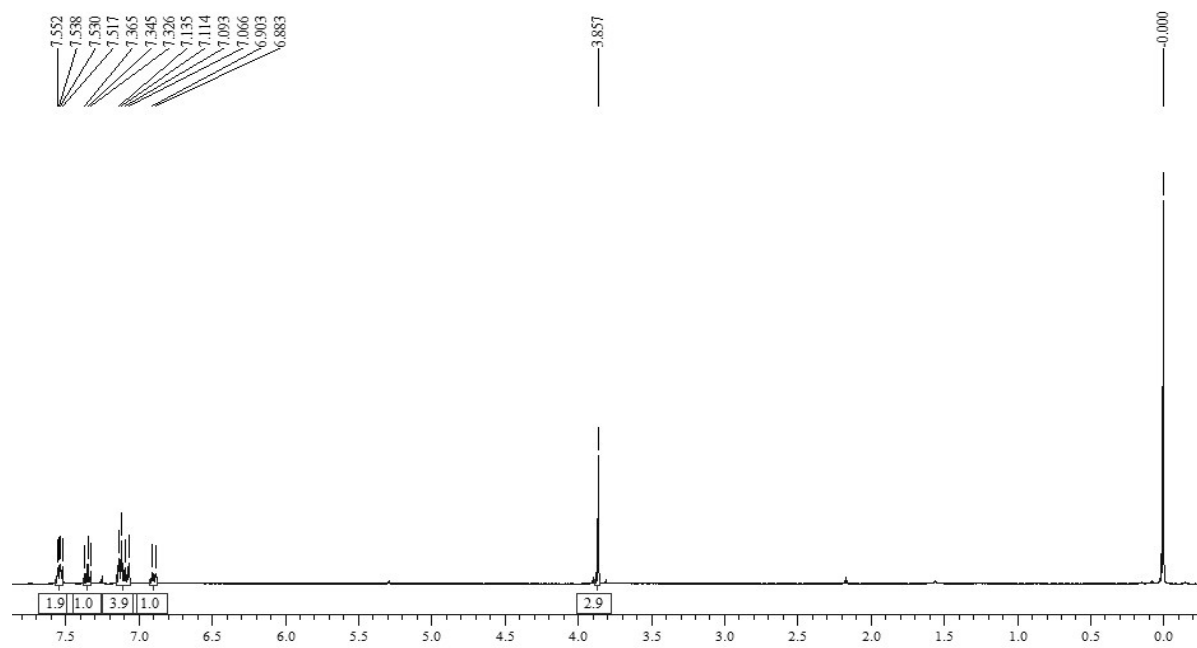
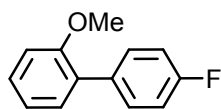
¹H & ¹³C NMR spectrum of 9g

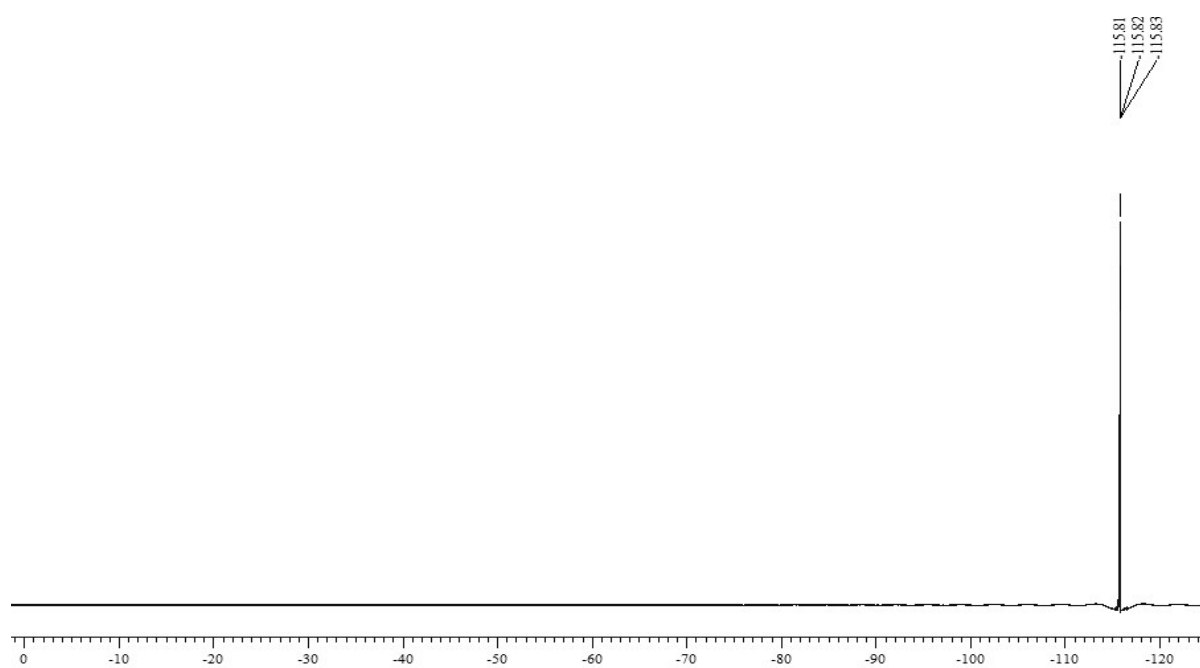
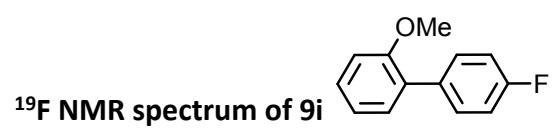


¹H & ¹³C NMR Spectrum of 9h

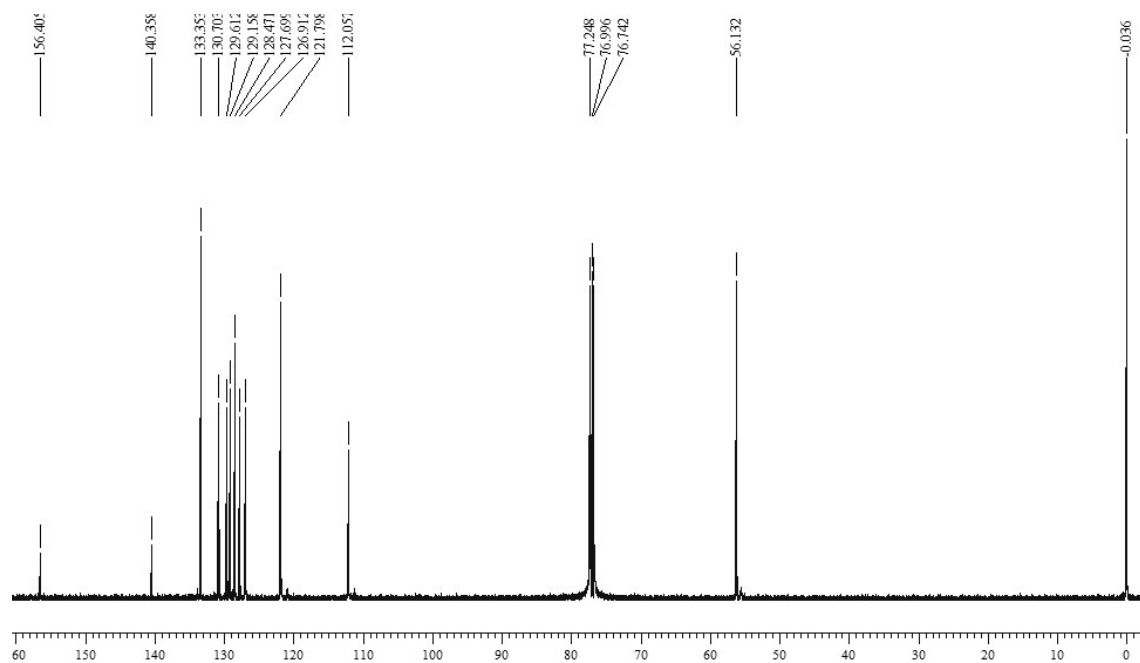
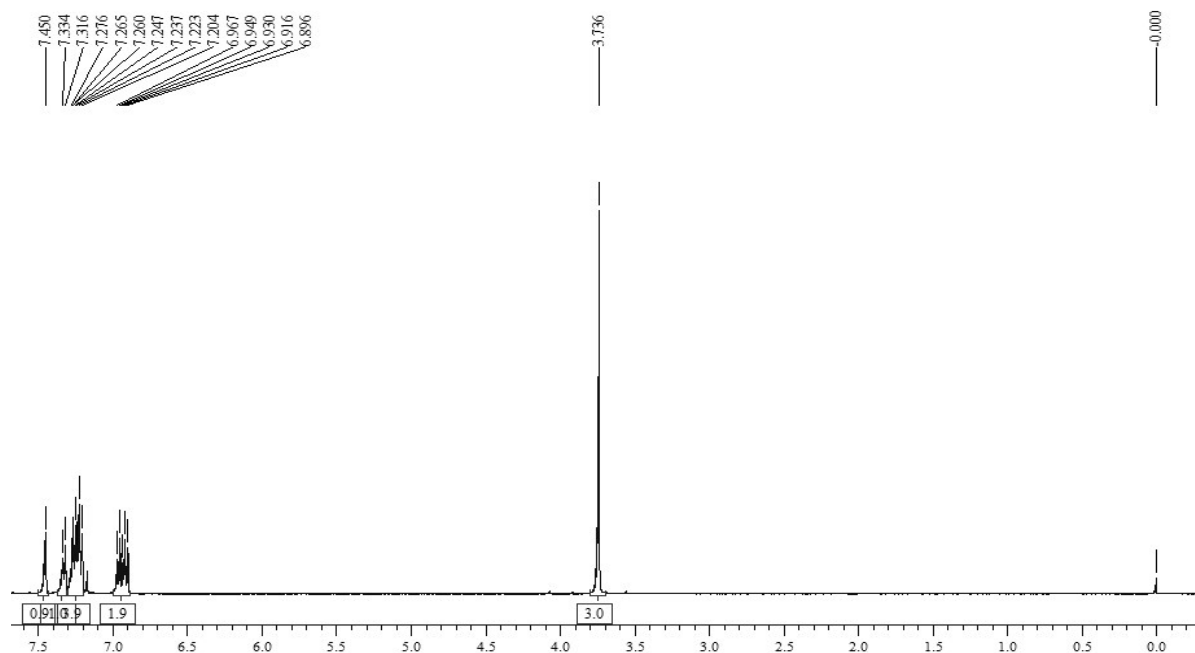
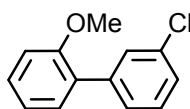


¹H & ¹³C NMR spectrum of 9i

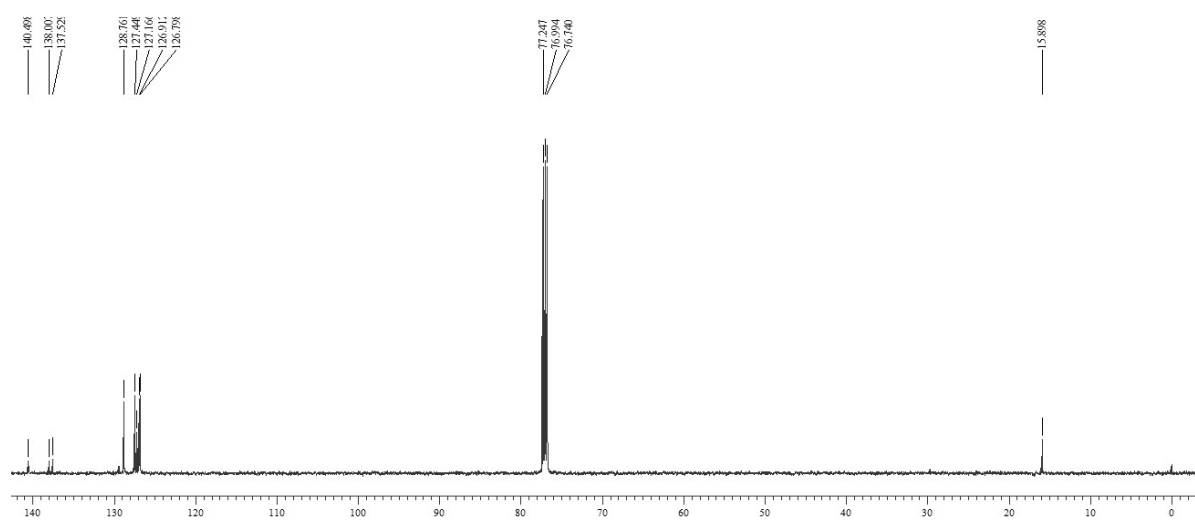
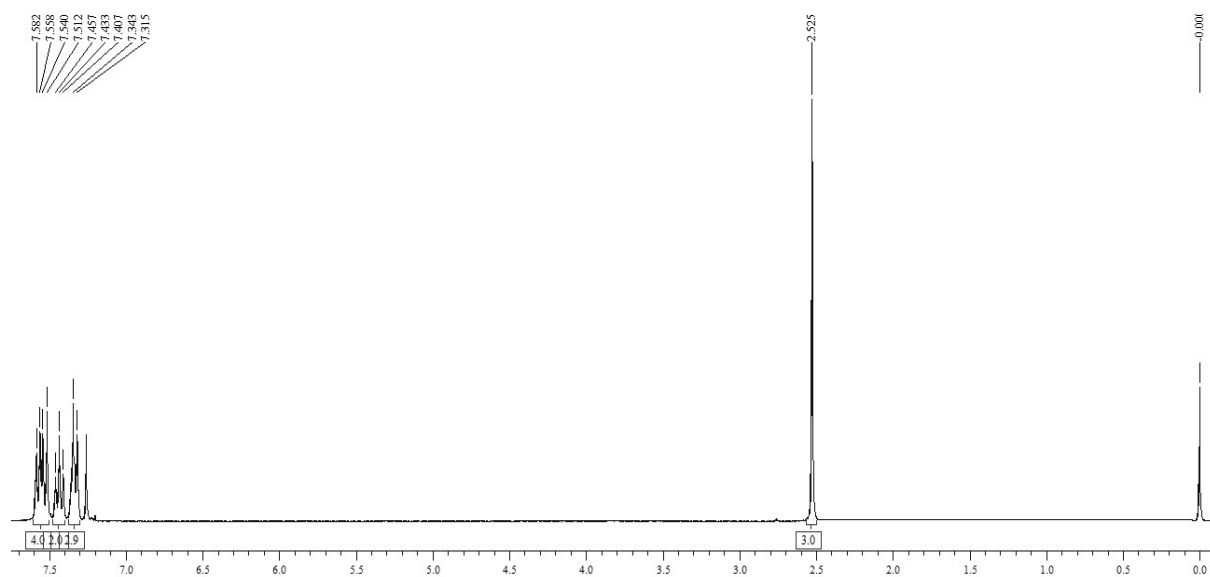
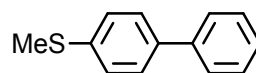




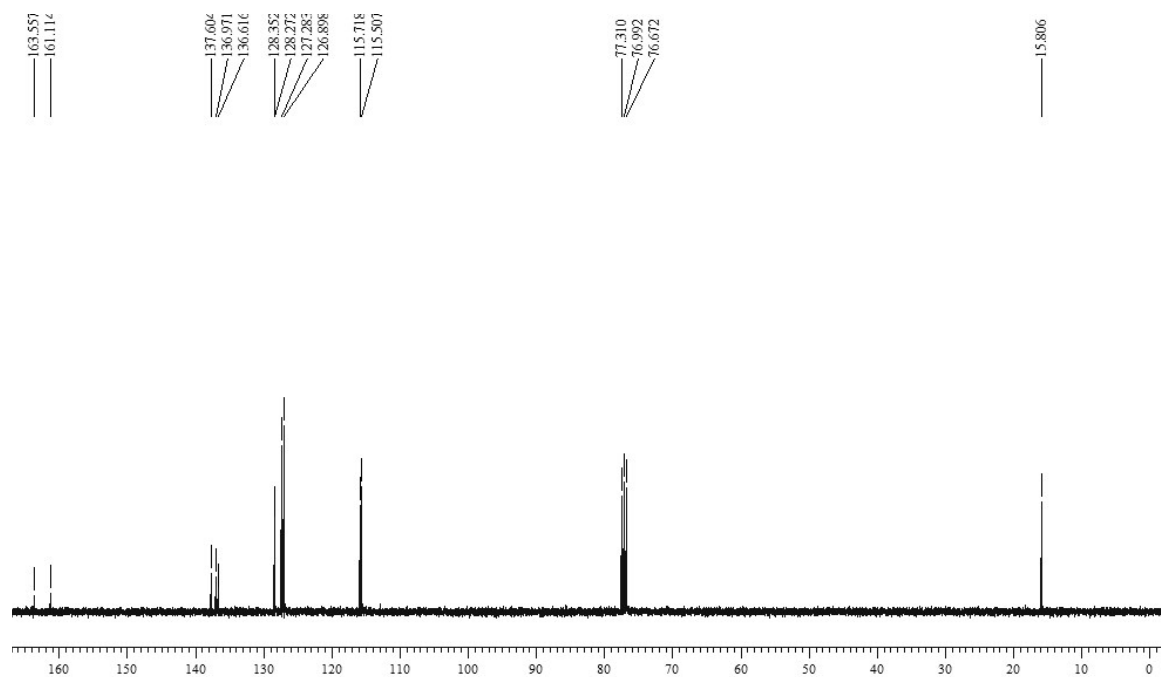
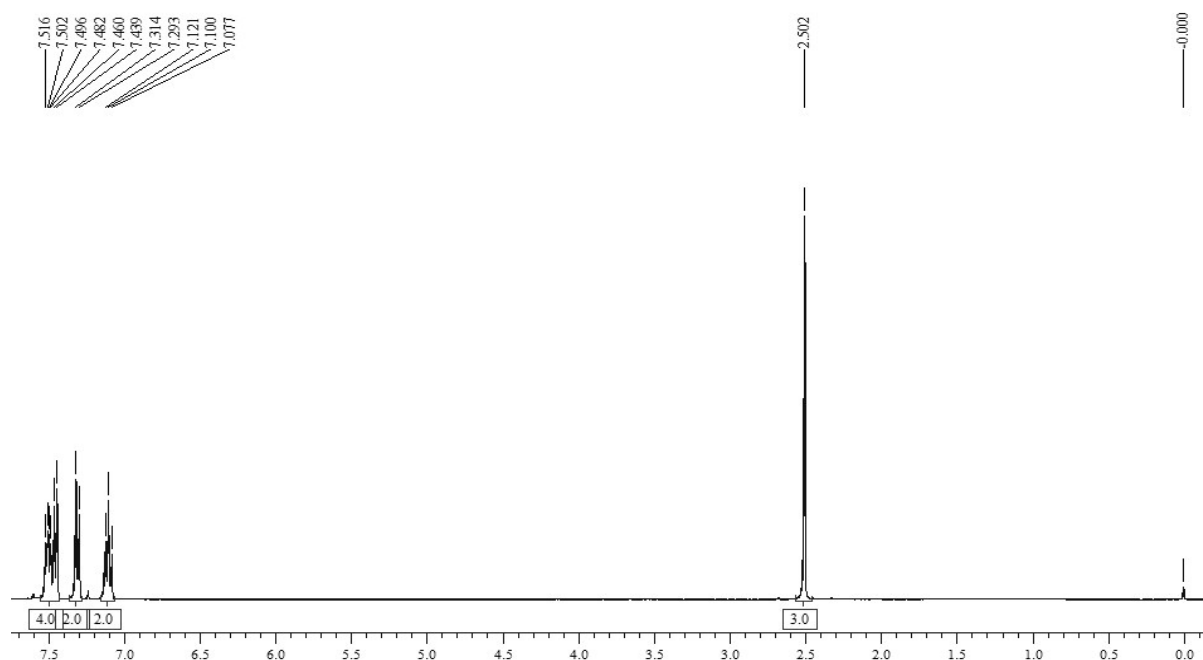
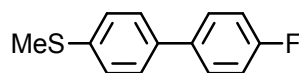
¹H & ¹³C NMR spectrum of 9j



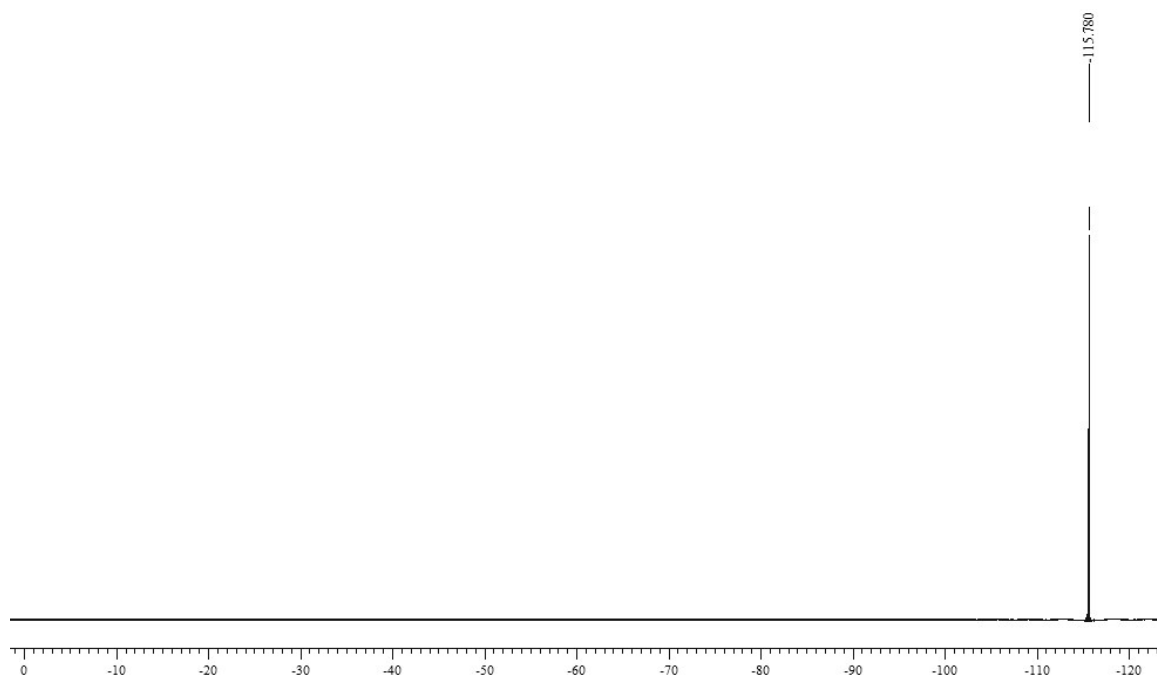
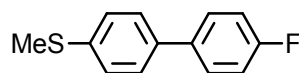
^1H & ^{13}C NMR Spectrum of 9k



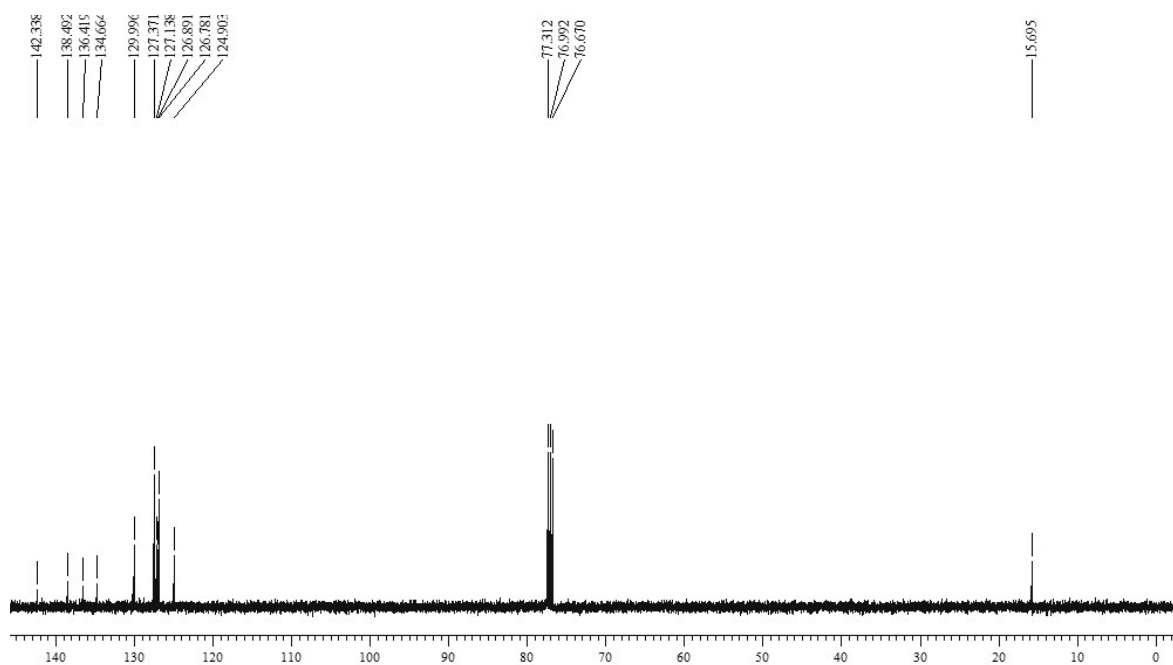
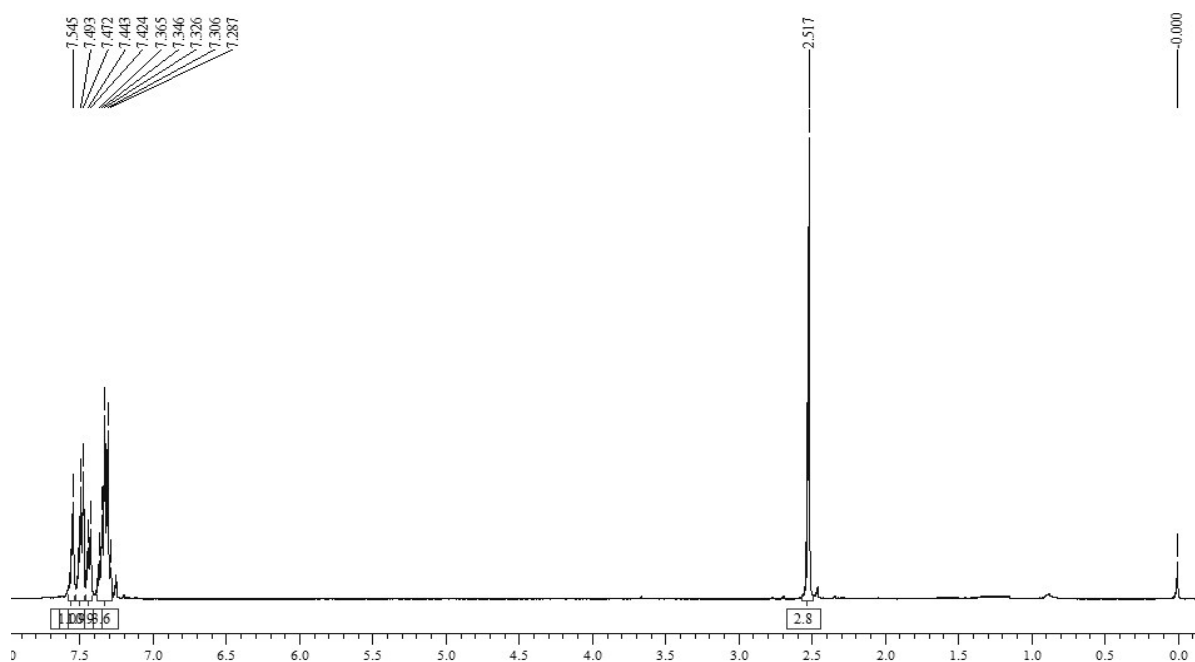
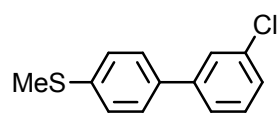
^1H & ^{13}C NMR spectrum of 9l



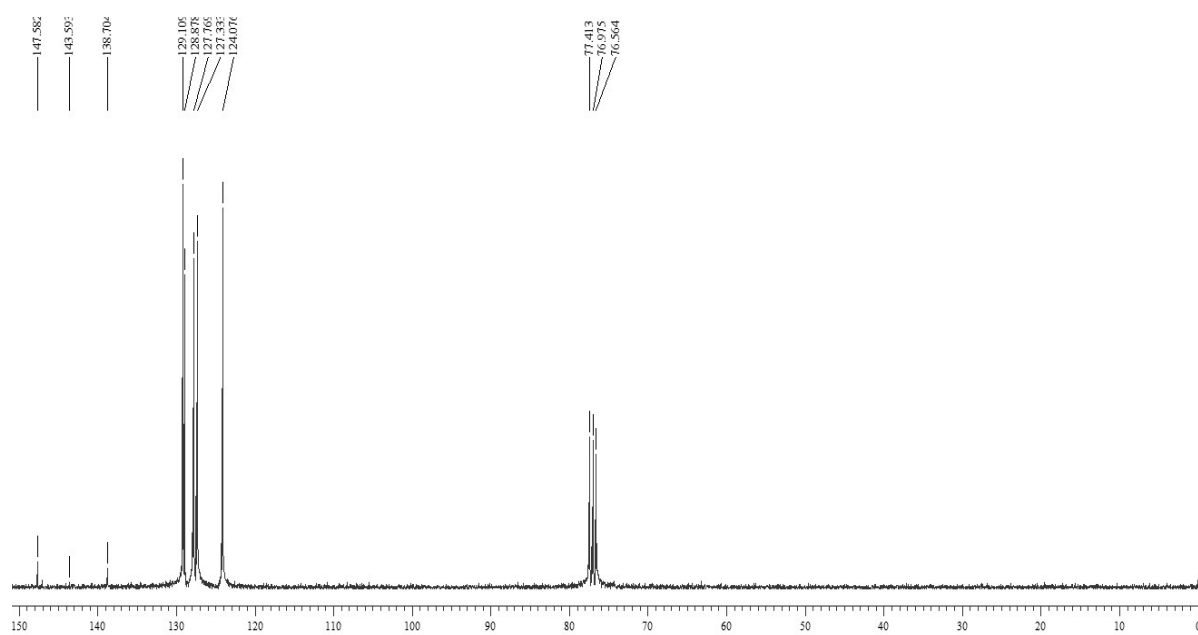
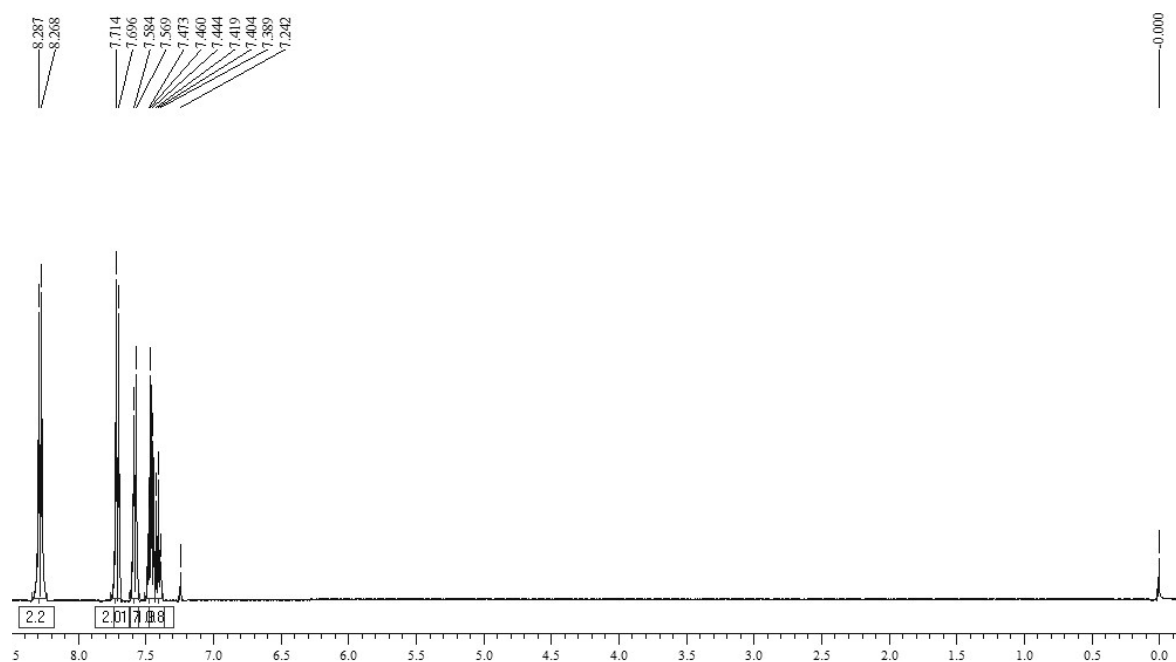
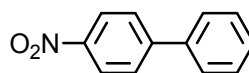
^{19}F NMR spectrum of 9l



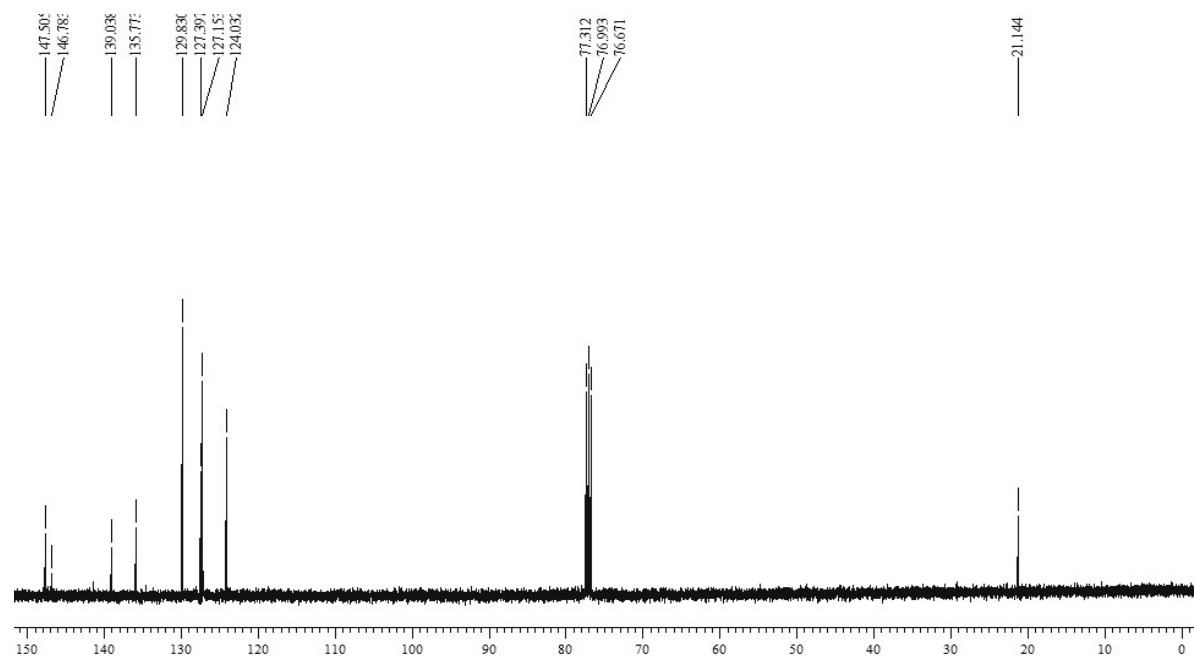
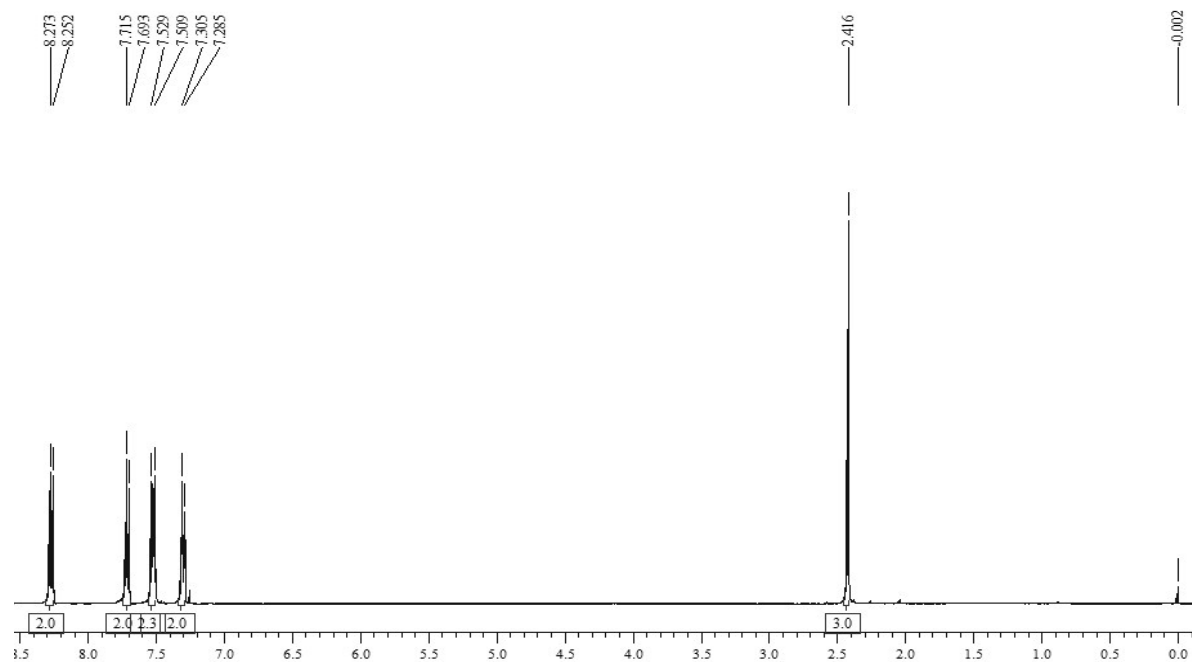
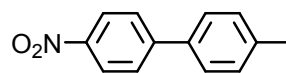
^1H & ^{13}C NMR spectrum of 9m



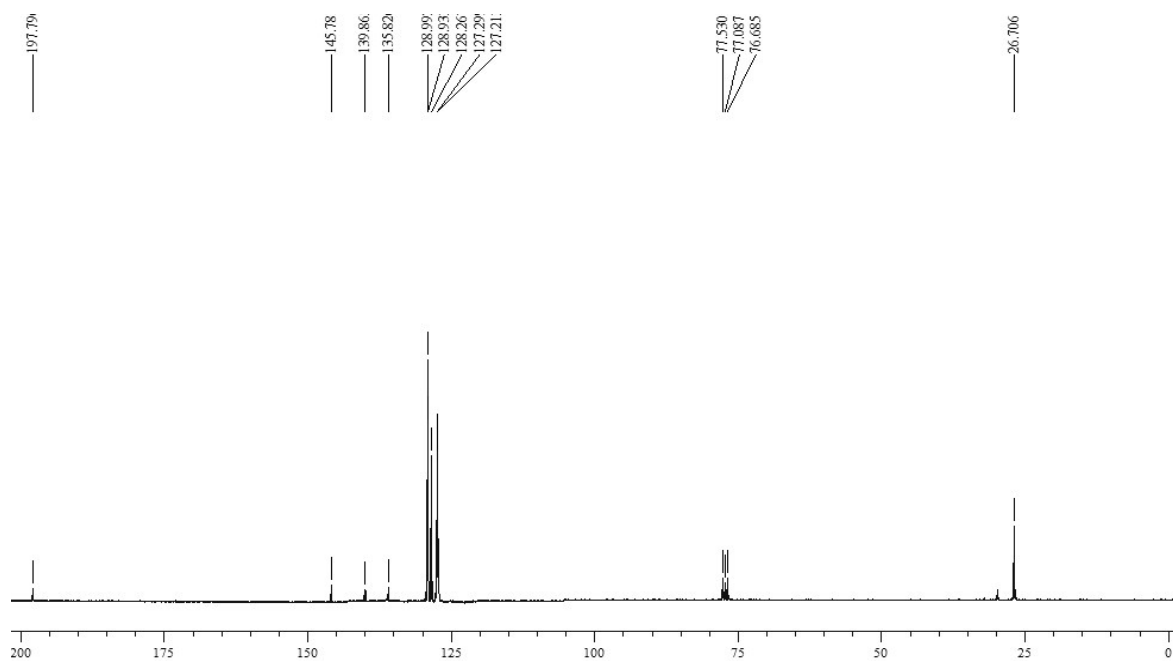
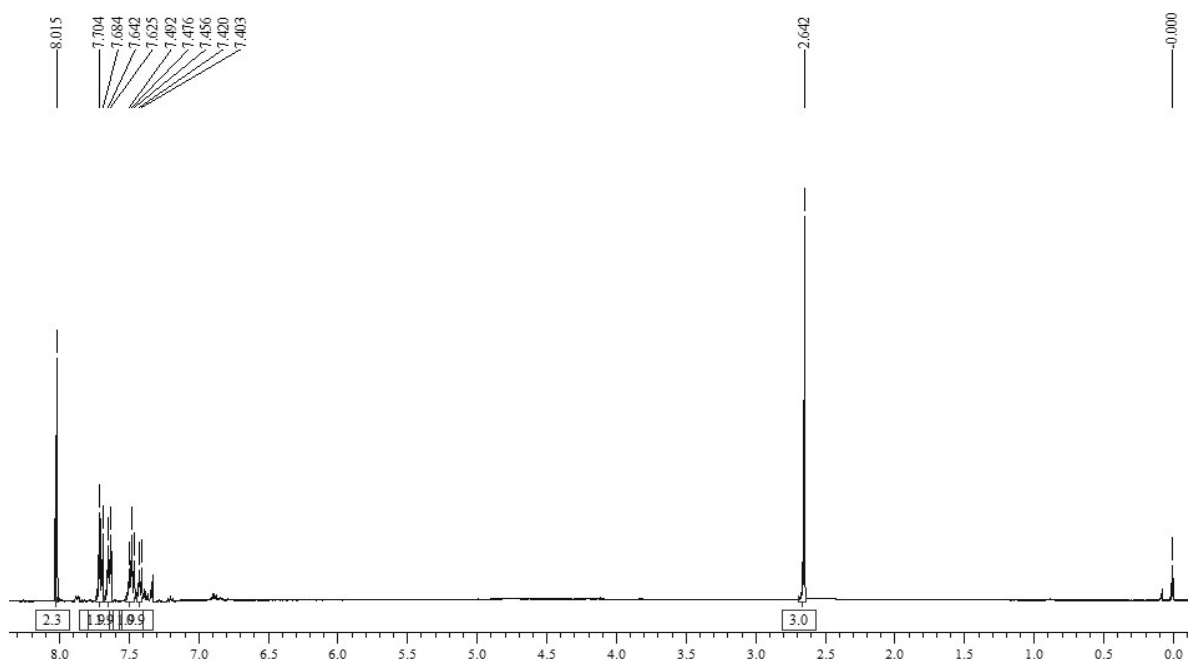
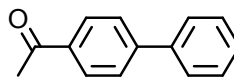
^1H & ^{13}C NMR spectrum of 9n



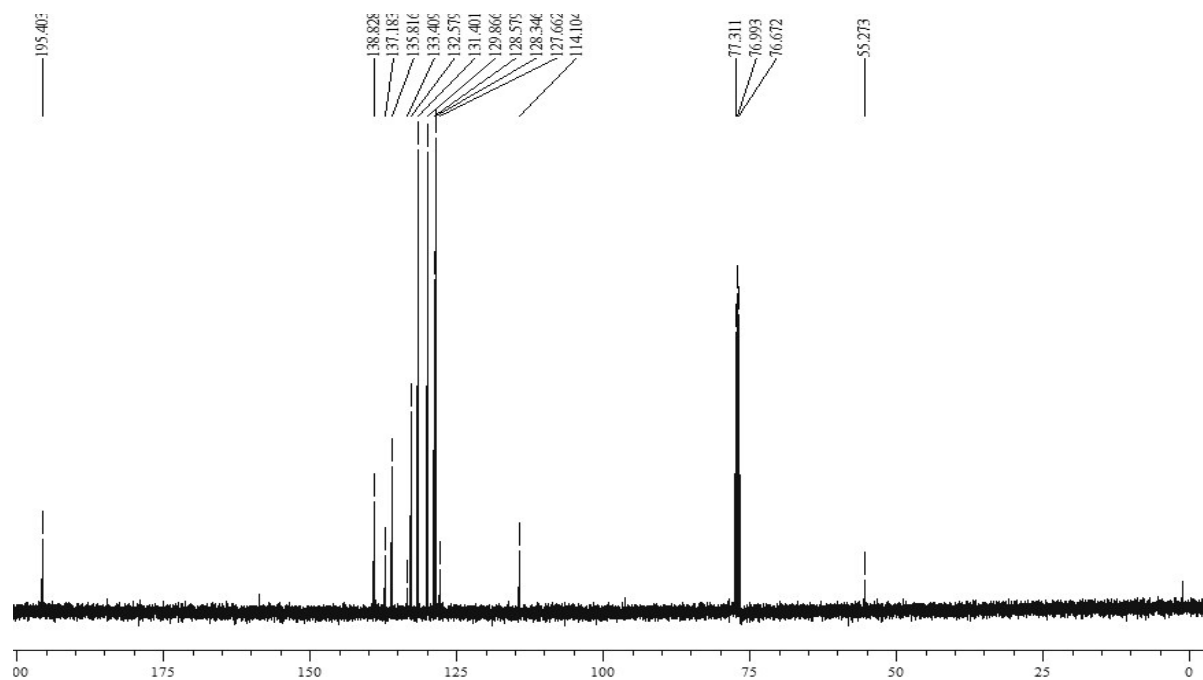
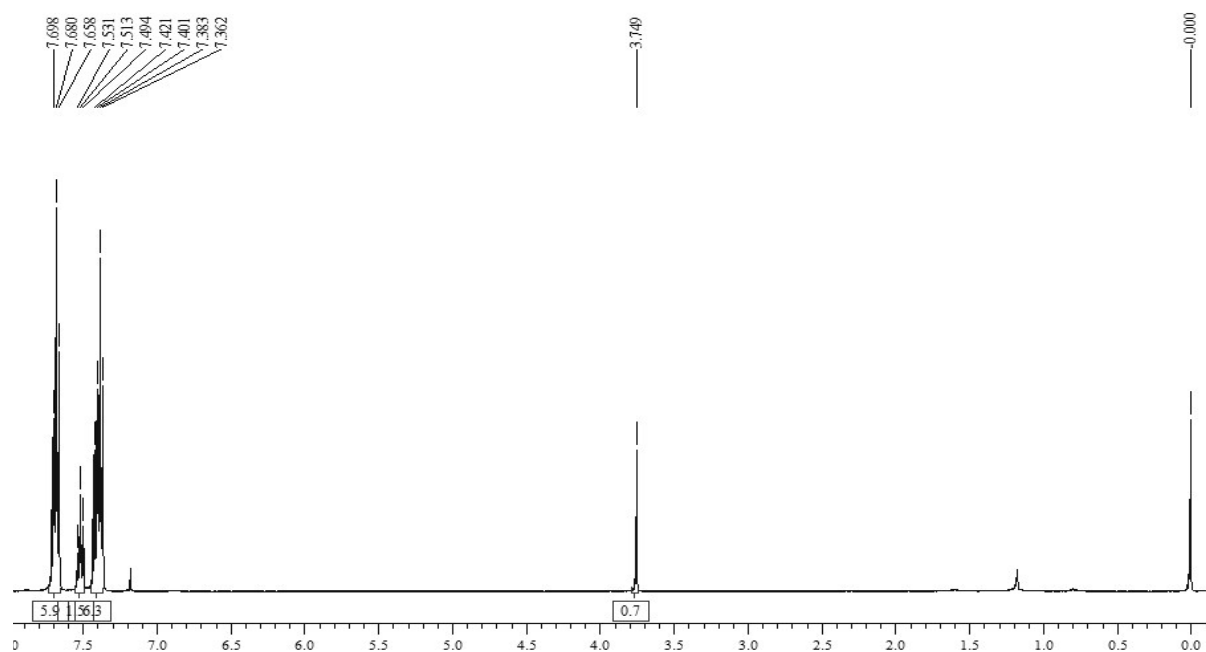
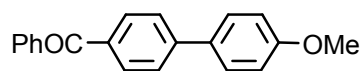
^1H & ^{13}C NMR spectrum of 9o



¹H & ¹³C NMR spectrum of 9p



¹H & ¹³C NMR spectrum of 9q



¹H & ¹³C NMR spectrum of 9r

