Supporting Information

Transition-Metal-Free catalyzed [3+2] Cycloadditions/Oxidative Aromatization Reactions for the synthesis of Annulated Indolizines

Qianwei Zhang, Bin Wang, Huifang Ma and Keyume Ablajan*

Key Laboratory of Oil & Gas Fine Chemicals, Ministry of Education & Xinjiang Uyghur Autonomous Region, College of Chemistry and Chemical Engineering, Xinjiang University, Urumqi 830046, PR China

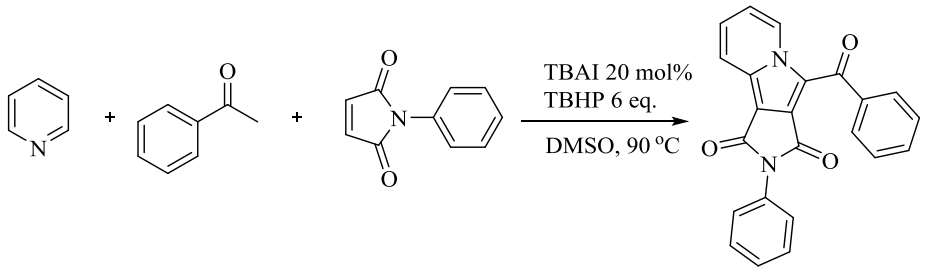
*E-mail: ablajan209@hotmail.com

Table of Contents

1. General information

All reagents and solvents were purchased from Adamas were used without further purification. The progress of the reaction was examined by thin-layer chromatography (TLC). Column chromatography was carried out using silica gel (200−400 mesh). NMR spectra (^{1}H NMR at 400 MHz, ^{13}C NMR at 100 MHz) were recorded on a Bruker Inova-400 instrument using tetramethylsilane (TMS) as an internal standard. High-resolution mass spectrometry (HRMS) data were obtained using a Micromass Q-TOF Global Tandem Mass Spectrometer. All the spectra data of compounds **4** and **5** are deposited in supporting information.

2. General procedure for the synthesis of 4 and 5 (4a as an example)

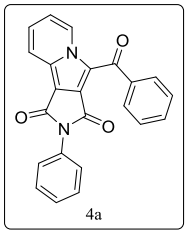


1a **2a** **3a** **4a**

A sealed tube was charged with pyridine **1a** (1.0 mmol), acetophenone **2a** (0.5 mmol), N-phenylmaleimide **3a** (0.75 mmol), TBAI (20 mmol%) and TBHP (6.0 equiv, 3 mmol) at room temperature, and DMSO (3 mL) was added. The resulting mixture was stirred for 18 hours at 90 ℃ and monitored by TLC. After the reaction completed, added 60 mL 10% $Na_2S_2O_3$ solution (w/w) to the mixture and extracted with EtOAc 3 times (3 × 60 mL). Combined organic layers were dried over anhydrous $MgSO_4$, and evaporated in vacuo to afford crude product. The crude product was purified by column chromatography on silica gel (petroleum ether/EtOAc = 8:1) to yield the desired product **4a** as a yellow solid.

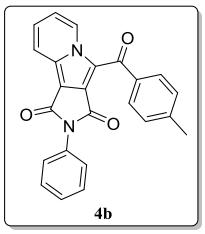
3. Spectral data of compounds obtained in this study:

4-benzoyl-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (4a):



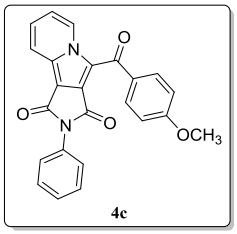
Yellow solid (159.6 mg, 87%); mp 222-224 °C; R_f = 0.51 (petroleum ether/ethyl acetate = 1:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.63 (dt, *J* = 7.3, 1.0 Hz, 1H), 8.01 (dt, *J* = 8.9, 1.2 Hz, 1H), 7.91-7.81 (m, 2H), 7.63-7.58 (m, 1H), 7.52-7.45 (m, 3H), 7.40 (ddd, *J* = 8.8, 3.5, 1.4 Hz, 2H), 7.34-7.27 (m, 3H), 7.14 (td, *J* = 7.1, 1.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.54, 162.56, 161.95, 138.71, 133.02, 132.49, 132.15, 130.03, 129.45, 128.85, 128.59, 128.32, 127.67, 126.92, 118.81, 117.66, 116.53, 110.62, 77.31, 76.99, 76.67. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{23}H_{14}N_2O_3$: 367.1006; found: 367.1008.

4-(4-methylbenzoyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (4b):



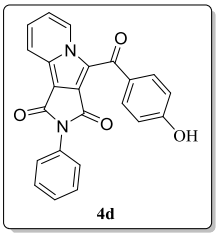
Yellow solid (163.6 mg, 86%); mp 238-240 °C; R_f = 0.46 (petroleum ether/ethyl acetate = 1:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.55 (d, *J* = 7.3 Hz, 1H), 7.99 (d, *J* = 8.9 Hz, 1H), 7.80 (d, *J* = 8.2 Hz, 2H), 7.50-7.45 (m, 1H), 7.44-7.38 (m, 2H), 7.36 -7.30 (m, 3H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.10 (td, *J* = 7.1, 1.3 Hz, 1H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.09, 162.60, 162.13, 144.04, 135.86, 132.38, 132.22, 129.97, 129.81, 129.59, 129.07, 128.85, 128.36, 127.66, 126.93, 118.78, 117.88, 116.30, 110.29, 21.84. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{24}H_{16}N_2O_3$: 381.1163; found: 381.1166.

4-(4-methoxybenzoyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (4c):



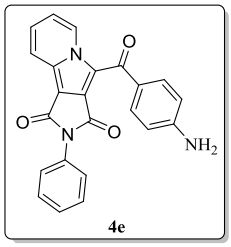
Yellow solid (166.5 mg, 84%); mp 276-277 °C; R_f = 0.28 (dichloromethane); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.47 (d, *J* = 7.3 Hz, 1H), 7.98 (d, *J* = 8.9 Hz, 1H), 7.91 (d, *J* = 8.8 Hz, 2H), 7.50-7.39 (m, 3H), 7.36-7.29 (m, 3H), 7.09 (dd, *J* = 10.0, 4.2 Hz, 1H), 6.95 (d, *J* = 8.9 Hz, 2H), 3.86 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.86, 163.92, 162.61, 162.28, 132.20, 131.01, 129.91, 129.09, 128.85, 128.16, 127.64, 126.88, 124.41, 123.93, 118.78, 118.03, 116.09,113.66, 109.97, 55.38. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{24}H_{16}N_2O_4$: 397.1112; found: 397.1115.

4-(4-hydroxybenzoyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (4d):



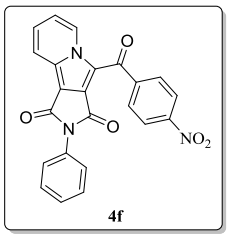
Yellow solid (156.8 mg, 82%); mp 278-280 °C; R_f = 0.45 (petroleum ether/ethyl acetate = 1:1); ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.40 (s, 1H), 9.25 (d, *J* = 7.3 Hz, 1H), 7.91 (d, *J* = 8.9 Hz, 1H), 7.80 (t, *J* = 8.8 Hz, 2H), 7.62-7.56 (m, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.38-7.29 (m, 3H), 7.24 (td, *J* = 7.1, 1.2 Hz, 1H), 6.80 (d, *J* = 8.7 Hz, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 183.27, 162.96, 162.66, 162.32, 133.15, 132.88, 131.79, 131.14, 130.26, 129.50, 129.46, 129.06, 128.89, 127.99, 127.85, 118.40, 118.09, 116.72, 115.54, 115.42, 109.31. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{23}H_{14}N_2O_4$: 383.0956; found: 383.0960.

4-(4-aminobenzoyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**4e**):



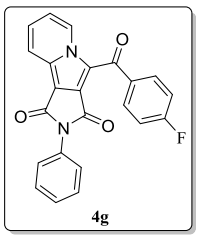
Yellow solid (143.0 mg, 75%); mp 268-270 °C; R_f = 0.40 (petroleum ether/ethyl acetate = 1:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.36 (d, *J* = 7.3 Hz, 1H), 7.95 (d, *J* = 8.9 Hz, 1H), 7.82 (d, *J* = 8.7 Hz, 2H), 7.45-7.38 (m, 3H), 7.36 (t, *J* = 4.3 Hz, 2H), 7.31 (t, *J* = 7.2 Hz, 1H), 7.03 (td, *J* = 7.2, 1.2 Hz, 1H), 6.65 (d, *J* = 8.7 Hz, 2H), 4.16 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.14, 162.70, 162.50, 151.82, 132.71, 132.36, 132.12, 130.75, 129.84, 128.81, 128.35, 128.10, 127.78, 127.55, 126.91, 118.71, 118.53, 115.71, 113.71, 109.99, 109.47. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{23}H_{15}N_3O_3$: 382.1115; found: 382.1117.

4-(4-nitrobenzoyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**4f**):



Yellow solid (154.3 mg, 75%); mp 289-290 °C; R_f = 0.48 (petroleum ether/ethyl acetate = 2:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (d, *J* = 7.2 Hz, 1H), 8.31 (d, *J* = 8.8 Hz, 2H), 8.07 (d, *J* = 8.9 Hz, 1H), 7.94 (d, *J* = 8.8 Hz, 2H), 7.63-7.57 (m, 1H), 7.45-7.38 (m, 2H), 7.36-7.25 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.57, 162.22, 161.86, 149.98, 144.21, 133.02, 131.81, 131.07, 130.32, 130.27, 129.93, 129.56, 129.01, 127.99, 126.80, 123.60, 119.01, 117.38, 116.66, 111.66. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{23}H_{13}N_3O_5$: 412.0857; found: 412.0860.

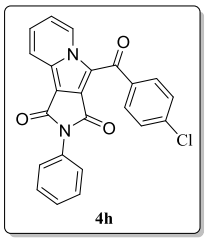
4-(4-fluorobenzoyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**4g**):



Yellow solid (138.4 mg, 72%); mp 223-224 °C; R_f = 0.24 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.58 (d, *J* = 7.3 Hz, 1H), 7.99 (d, *J* = 8.9 Hz, 1H), 7.93-7.85 (m, 2H), 7.52-7.46 (m, 1H), 7.45-7.39 (m, 2H), 7.36-7.29 (m, 3H), 7.13 (ddd, *J* = 6.9, 6.4, 1.7 Hz, 3H). ^{13}C NMR (100 MHz, CDCl3) δ 183.88, 167.09, 163.33 (d,

J = 245.0 Hz), 162.45, 134.87 (d, *J* = 3.0 Hz), 132.56, 132.16, 132.10, 132.07, 130.01, 129.94, 128.92, 128.70, 127.78, 126.86, 118.83, 117.40, 116.60, 115.57 (d, *J* = 22.0 Hz), 110.62. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{23}H_{13}FN_2O_3$: 385.0912; found: 385.0914.

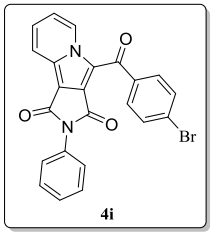
4-(4-chlorobenzoyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**4h**):



4h

Yellow solid (150.3 mg, 75%); mp 269-270 °C; R_f = 0.56 (petroleum ether/ethyl acetate = 2:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.62 (dt, *J* = 7.2, 1.0 Hz, 1H), 8.02 (dt, *J* = 8.9, 1.2 Hz, 1H), 7.85-7.77 (m, 2H), 7.52 (ddd, *J* = 8.8, 6.9, 1.0 Hz, 1H), 7.47-7.39 (m, 4H), 7.36 -7.27 (m, 3H), 7.15 (td, *J* = 7.1, 1.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.11, 162.43, 162.06, 139.49, 136.94, 132.64, 132.05, 130.87, 130.05, 128.94, 128.80, 128.68, 127.83, 126.88, 118.87, 117.27, 116.70, 110.77. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{23}H_{13}ClN_2O_3$: 401.0617; found: 401.0619.

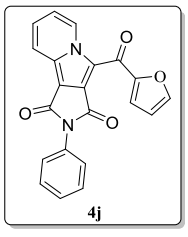
4-(4-bromobenzoyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**4i**):



4i

Yellow solid (155.8 mg, 70%); mp 281-282 °C; R_f = 0.37 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.62 (d, *J* = 7.3 Hz, 1H), 8.02 (d, *J* = 8.9 Hz, 1H), 7.76-7.71 (m, 2H), 7.63-7.59 (m, 2H), 7.55-7.50 (m, 1H), 7.43 (dd, *J* = 10.3, 4.9 Hz, 2H), 7.36-7.30 (m, 3H), 7.16 (td, *J* = 7.1, 1.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.25, 162.43, 162.06, 137.38, 132.66, 132.05, 131.65, 130.97, 130.07, 128.96, 128.83, 128.22, 127.85, 126.89, 118.88, 117.21, 116.73, 110.81. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{23}H_{13}BrN_2O_3$: 445.0112; found: 445.0115.

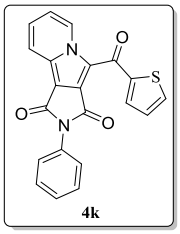
4-(furan-2-carbonyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**4j**):



4j

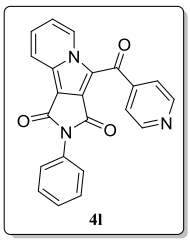
Yellow solid (149.6 mg, 84%); mp 261-263 °C; R_f = 0.28 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.41 (d, *J* = 7.3 Hz, 1H), 7.96 (d, *J* = 8.9 Hz, 1H), 7.66 (dd, *J* = 1.5, 0.6 Hz, 1H), 7.47-7.42 (m, 3H), 7.40- 7.30 (m, 4H), 7.07 (td, *J* = 7.1, 1.3 Hz, 1H), 6.61 (dd, *J* = 3.6, 1.7 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.56, 162.52, 162.30, 152.07, 147.61, 132.27, 129.65, 129.45, 128.96, 128.92, 128.85, 128.34, 127.73, 126.99, 126.92, 126.88, 120.12, 118.87, 116.31, 112.64, 110.60. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{21}H_{12}N_2O_4$: 357.0799; found: 357.0801.

2-phenyl-4-(thiophene-2-carbonyl)-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**4k**):



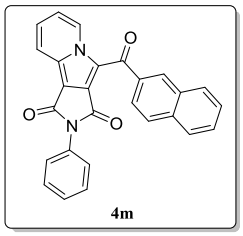
Yellow solid (128.5 mg, 69%); mp 275 °C; R_f = 0.29 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.36 (d, *J* = 7.3 Hz, 1H), 7.99 (d, *J* = 8.9 Hz, 1H), 7.85 (dd, *J* = 3.8, 1.0 Hz, 1H), 7.76 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.45 (dd, *J* = 15.1, 7.2 Hz, 3H), 7.35 (dd, *J* = 16.7, 7.9 Hz, 3H), 7.15 (dd, *J* = 4.9, 3.9 Hz, 1H), 7.08 (td, *J* = 7.1, 1.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 176.60, 162.47, 162.21, 142.91, 135.65, 135.09, 135.02, 132.31, 132.13, 129.82, 128.93, 128.79, 128.35, 128.11, 127.77, 126.90, 118.89, 117.65, 116.25, 115.08, 110.31. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{21}H_{12}N_2O_3S$: 373.0571; found: 373.0575.

4-isonicotinoyl-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**4l**):



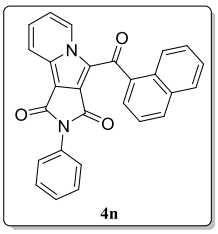
Yellow solid (132.2 mg, 72%); mp 265-267 °C; R_f = 0.29 (dichloromethane/ethyl acetate = 2:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.77 (d, *J* = 7.2 Hz, 1H), 8.76 (d, *J* = 5.8 Hz, 2H), 8.02 (d, *J* = 8.9 Hz, 1H), 7.62-7.54 (m, 3H), 7.40 (dd, *J* = 10.5, 4.7 Hz, 2H), 7.34-7.30 (m, 1H), 7.29-7.25 (m, 2H), 7.21 (td, *J* = 7.1, 1.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.81, 162.25, 161.69, 150.90, 149.94, 145.84, 132.96, 131.84, 131.13, 130.26, 129.67, 128.97, 127.93, 126.83, 122.32, 121.23, 118.94, 117.48, 116.37, 111.73. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{22}H_{13}N_3O_3$: 368.0959; found: 368.0962.

4-(2-naphthoyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**4m**):



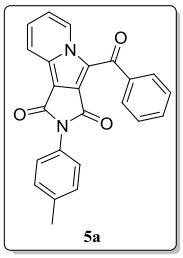
Yellow solid (170.7mg, 82%); mp 277-278 °C; R_f = 0.35 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.62 (d, *J* = 7.3 Hz, 1H), 8.40 (s, 1H), 8.02 (d, *J* = 8.9 Hz, 1H), 7.95-7.91 (m, 2H), 7.87 (t, *J* = 8.0 Hz, 2H), 7.58 -7.53 (m, 1H), 7.52-7.46 (m, 2H), 7.36 (dd, *J* = 8.2, 7.0 Hz, 2H), 7.31-7.25 (m, 3H), 7.13 (td, *J* = 7.1, 1.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.26, 162.60, 161.97, 135.80, 135.68, 132.53, 132.40, 132.19, 131.89, 130.09, 129.94, 129.62, 129.05, 128.83, 128.55, 128.35, 128.27, 127.91, 127.62, 126.81, 126.67, 124.93, 119.67, 118.85, 117.90, 116.47, 110.59. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{27}H_{16}N_2O_3$: 417.1163; found: 417.1165.

4-(1-naphthoyl)-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**4n**):



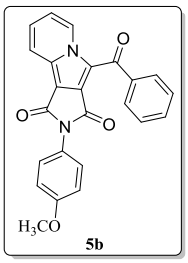
Yellow solid (179.1 mg, 86%); mp 215 °C; R_f = 0.41 (petroleum ether/ethyl acetate = 2:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.03 (d, *J* = 7.2 Hz, 1H), 8.18 (dd, *J* = 6.1, 3.4 Hz, 1H), 8.03 (d, *J* = 8.3 Hz, 2H), 7.91 (dd, *J* = 6.2, 3.3 Hz, 1H), 7.78-7.70 (m, 1H), 7.61-7.43 (m, 4H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.28-7.15 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.45, 162.58, 161.22, 136.82, 133.83, 132.60, 132.10, 132.02, 131.02, 130.67, 130.48, 129.06, 128.81, 128.66, 128.23, 127.63, 127.24, 126.95, 126.20, 124.80, 124.78, 124.30, 119.67, 118.82, 118.78, 117.02, 111.35. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{27}H_{16}N_2O_3$: 417.1163; found: 417.1167.

4-benzoyl-2-(p-tolyl)-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**5a**):



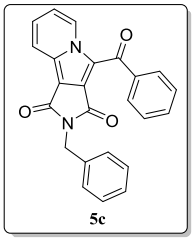
Yellow solid (163.6 mg, 86%); mp 227-229 °C; R_f = 0.33 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.62 (d, *J* = 7.2 Hz, 1H), 7.99 (d, *J* = 8.9 Hz, 1H), 7.89-7.81 (m, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.47 (q, *J* = 7.1 Hz, 3H), 7.23-7.16 (m, 4H), 7.12 (td, *J* = 7.1, 1.3 Hz, 1H), 2.34 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.53, 162.72, 162.09, 138.71, 137.68, 132.98, 132.44, 130.17, 130.01, 129.51, 129.46, 128.53, 128.31, 126.81, 119.70, 118.78, 117.60, 116.47, 110.71, 21.13. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{24}H_{16}N_2O_3$: 381.1163; found: 381.1166.

4-benzoyl-2-(4-methoxyphenyl)-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**5b**):



Yellow solid (166.5 mg, 84%); mp 240 °C; R_f = 0.46 (petroleum ether/ethyl acetate = 2:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.62 (d, *J* = 7.3 Hz, 1H), 7.99 (d, *J* = 8.9 Hz, 1H), 7.91-7.78 (m, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.48 (dt, *J* = 13.8, 4.1 Hz, 3H), 7.24-7.16 (m, 2H), 7.12 (td, *J* = 7.1, 1.3 Hz, 1H), 6.92 (d, *J* = 9.0 Hz, 2H), 3.78 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.54, 162.87, 162.25, 158.95, 138.73, 132.98, 132.44, 130.16, 130.02, 129.45, 128.53, 128.30, 124.86, 118.78, 117.62, 116.45, 114.23, 110.67, 55.45. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{24}H_{16}N_2O_4$: 397.1112; found: 397.1114.

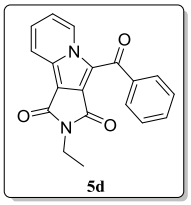
4-benzoyl-2-benzyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**5c**):



Yellow solid (171.2 mg, 90%); mp 209 °C; R_f = 0.51 (petroleum ether/ethyl acetate = 2:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.55 (d, *J* = 7.2 Hz, 1H), 7.90 (dt, *J* = 8.9, 1.1 Hz, 1H), 7.86-7.79 (m, 2H), 7.68-7.62 (m, 1H), 7.53-7.47 (m, 2H), 7.42 (ddd, *J* = 8.8, 6.9, 0.9

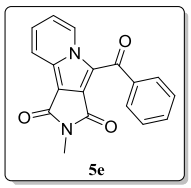
Hz, 1H), 7.36-7.30 (m, 2H), 7.28-7.18 (m, 3H), 7.05 (td, *J* = 7.1, 1.3 Hz, 1H), 4.69 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.35, 163.42, 162.65, 138.73, 136.89, 133.00, 132.12, 130.59, 129.90, 129.58, 128.49, 128.38, 128.25, 127.51, 118.56, 117.39, 116.20, 110.88, 41.66. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{24}H_{16}N_2O_3$: 381.1163; found: 381.1164.

4-benzoyl-2-ethyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**5d**):



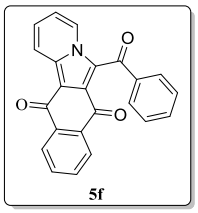
Yellow solid (149.6 mg, 94%); mp 204-205 °C; R_f = 0.44 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.58 (d, *J* = 7.2 Hz, 1H), 7.90 (d, *J* = 8.9 Hz, 1H), 7.81 (dd, *J* = 8.2, 1.2 Hz, 2H), 7.64 (t, *J* = 7.5 Hz, 1H), 7.49 (t, *J* = 7.7 Hz, 2H), 7.46-7.41 (m, 1H), 7.06 (td, *J* = 7.0, 1.3 Hz, 1H), 3.56 (q, *J* = 7.2 Hz, 2H), 1.15 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.46, 163.53, 162.80, 138.89, 132.84, 132.03, 130.92, 129.93, 129.45, 128.31, 128.21, 118.52, 117.25, 116.11, 111.18, 32.99, 14.04. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{19}H_{14}N_2O_3$: 319.1006; found: 381.1170.

4-benzoyl-2-methyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**5e**):



Yellow solid (140.0 mg, 92%); mp 246-247 °C; R_f = 0.33 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.58 (d, *J* = 7.2 Hz, 1H), 7.90 (d, *J* = 8.9 Hz, 1H), 7.85-7.73 (m, 2H), 7.63 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 7.7 Hz, 2H), 7.46-7.40 (m, 1H), 7.06 (td, *J* = 7.1, 1.3 Hz, 1H), 2.99 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.54, 163.74, 163.11, 138.93, 132.84, 132.08, 130.90, 129.94, 129.40, 128.37, 128.24, 118.56, 117.32, 116.15, 111.10, 24.15. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{18}H_{12}N_2O_3$: 305.0850; found: 305.0855.

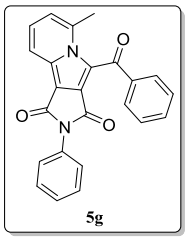
6-benzoylbenzo[f]pyrido[2,1-a]isoindole-7,12-dione (**5f**):



Red solid (158.1 mg, 90%); mp 253-255 °C; R_f = 0.41 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.80 (d, *J* = 7.0 Hz, 1H), 8.24 (dd, *J* = 7.7, 1.0 Hz, 1H), 8.00 (dd, *J* = 7.7, 1.0 Hz, 1H), 7.93 (d, *J* = 9.1 Hz, 1H), 7.90-7.85 (m, 2H), 7.71 (td, *J* = 7.6, 1.3 Hz, 1H), 7.62 (td, *J* = 7.5, 1.3 Hz, 1H), 7.59-7.54 (m, 1H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.37 (ddd, *J* = 9.0, 6.9, 1.1 Hz, 1H), 7.17 (td, *J* = 6.9, 1.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.65, 180.80, 174.87, 139.10, 138.92, 134.32, 133.73, 133.57, 133.01, 132.91, 129.23, 128.35, 128.28, 128.17,

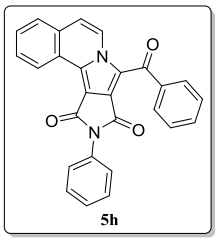
127.58, 127.11, 126.28, 121.29, 120.35, 117.62, 113.71. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{23}H_{13}NO_3$: 352.0897; found: 352.0898.

4-benzoyl-6-methyl-2-phenyl-1H-pyrrolo[3,4-a]indolizine-1,3(2H)-dione (**5g**):



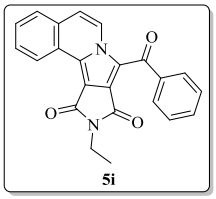
Yellow solid (144.6 mg, 76%); mp 178-180 °C; R_f = 0.28 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14-8.03 (m, 2H), 7.94 (d, *J* = 8.6 Hz, 1H), 7.64 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 2H), 7.42 (ddd, *J* = 15.2, 8.1, 4.5 Hz, 3H), 7.34-7.26 (m, 3H), 6.93 (d, *J* = 6.9 Hz, 1H), 2.51 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 184.19, 162.68, 162.34, 140.22, 137.59, 134.05, 133.99, 132.26, 130.52, 129.74, 128.78, 128.58, 127.96, 127.55, 126.88, 120.54, 117.63, 116.71, 109.39, 23.30. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{24}H_{16}N_2O_3$: 381.1163; found: 381.1165.

8-benzoyl-10-phenyl-9H-pyrrolo[3',4':3,4]pyrrolo[2,1-a]isoquinoline-9,11(10H)-dione (**5h**):



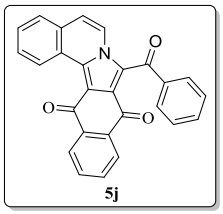
Light yellow solid (177.0 mg, 85%); mp 297-298 °C; R_f = 0.46 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.35 (dd, *J* = 6.9, 2.5 Hz, 1H), 9.15 (d, *J* = 7.6 Hz, 1H), 7.94 (dd, *J* = 8.2, 1.2 Hz, 2H), 7.83-7.77 (m, 1H), 7.76-7.70 (m, 2H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.50 (t, *J* = 7.7 Hz, 2H), 7.46-7.40 (m, 2H), 7.39-7.30 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.73, 162.97, 161.76, 138.47, 133.43, 132.36, 132.29, 130.62, 129.82, 129.71, 129.16, 128.85, 128.40, 128.05, 127.77, 127.74, 126.98, 126.71, 125.35, 123.90, 119.73, 116.64, 112.65. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{27}H_{16}N_2O_3$: 417.1163; found: 417.1166.

8-benzoyl-10-ethyl-9H-pyrrolo[3',4':3,4]pyrrolo[2,1-a]isoquinoline-9,11(10H)-dione (**5i**):



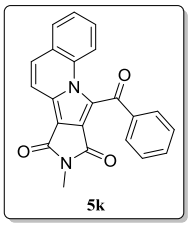
Yellow solid (163.9 mg, 89%); mp 228-229 °C; R_f = 0.51 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.23 (d, *J* = 7.4 Hz, 1H), 9.09 (d, *J* = 7.6 Hz, 1H), 7.95-7.85 (m, 2H), 7.77-7.73 (m, 1H), 7.72-7.63 (m, 3H), 7.52 (t, *J* = 7.8 Hz, 2H), 7.23 (d, *J* = 7.6 Hz, 1H), 3.63 (q, *J* = 7.2 Hz, 2H), 1.19 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.71, 163.80, 162.63, 138.63, 133.28, 131.82, 130.43, 129.72, 129.00, 128.91, 128.30, 127.60, 126.62, 125.34, 123.77, 119.20, 116.20, 113.22, 33.28, 14.03. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{23}H_{16}N_2O_3$: 369.1163; found: 369.1164.

8-benzoylbenzo[5,6]isoindolo[1,2-a]isoquinoline-9,14-dione (**5j**):



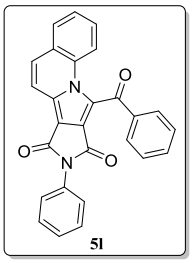
Red solid (170.6 mg, 85%); mp 311-312 °C; R_f = 0.56 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.49 (d, *J* = 7.4 Hz, 1H), 8.25 (dd, *J* = 7.7, 1.0 Hz, 1H), 8.09-7.99 (m, 3H), 7.90 (d, *J* = 8.1 Hz, 1H), 7.72 (ddd, *J* = 9.0, 5.6, 1.7 Hz, 2H), 7.66-7.51 (m, 3H), 7.49-7.38 (m, 3H), 7.32 (d, *J* = 7.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.92, 180.76, 175.22, 137.34, 134.52, 134.04, 133.77, 133.19, 133.16, 132.92, 129.51, 129.47, 129.23, 129.05, 128.90, 127.67, 126.90, 126.57, 124.74, 124.44, 124.40, 122.54, 117.60, 116.83, 109.98. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{27}H_{15}NO_3$: 402.1054; found: 402.1055.

10-benzoyl-8-methyl-7H-pyrrolo[3',4':3,4]pyrrolo[1,2-a]quinoline-7,9(8H)-dione (**5k**):



Yellow solid (157.7 mg, 89%); mp 256-257 °C; R_f = 0.47 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.79 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.77-7.70 (m, 3H), 7.65 (d, *J* = 9.2 Hz, 1H), 7.57 (td, *J* = 7.5, 1.6 Hz, 2H), 7.54-7.45 (m, 2H), 3.01 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.95, 163.88, 163.13, 137.25, 134.50, 133.40, 131.54, 130.48, 129.64, 129.54, 129.51, 129.35, 128.71, 126.19, 125.19, 123.02, 119.30, 116.42, 112.10, 24.20. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{22}H_{14}N_2O_3$: 355.1006; found: 355.1009.

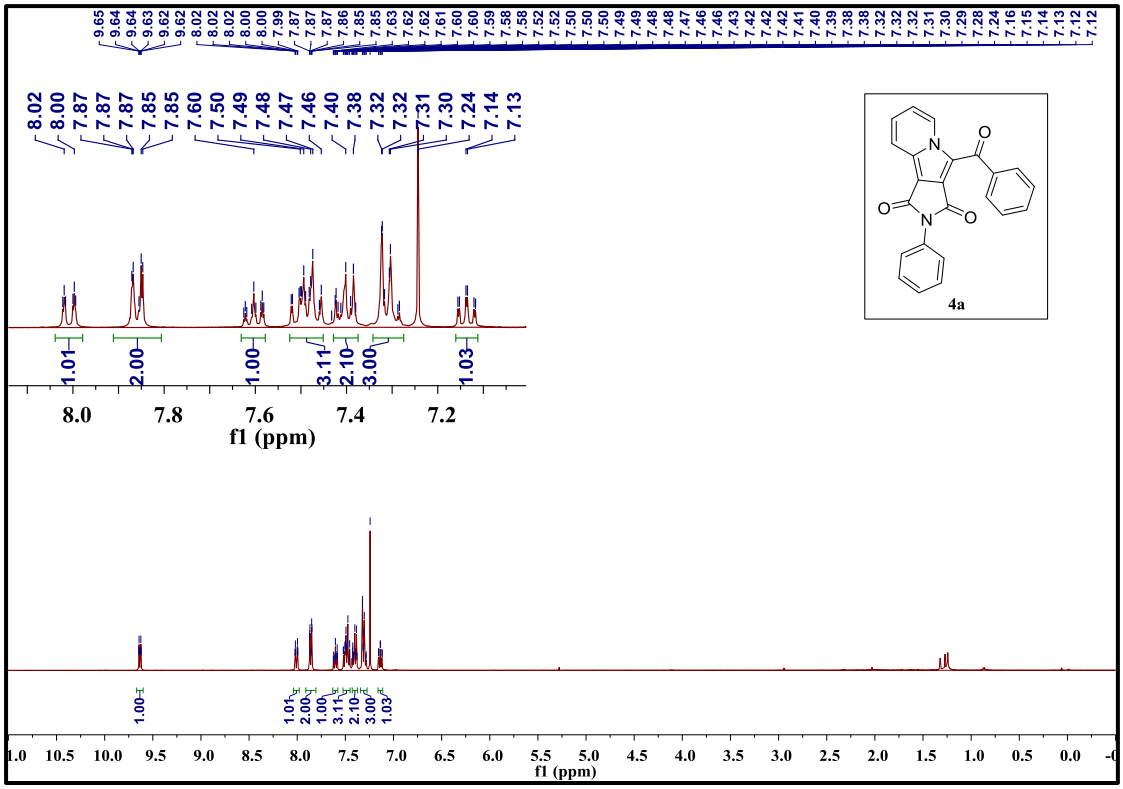
10-benzoyl-8-phenyl-7H-pyrrolo[3',4':3,4]pyrrolo[1,2-a]quinoline-7,9(8H)-dione (**5l**):

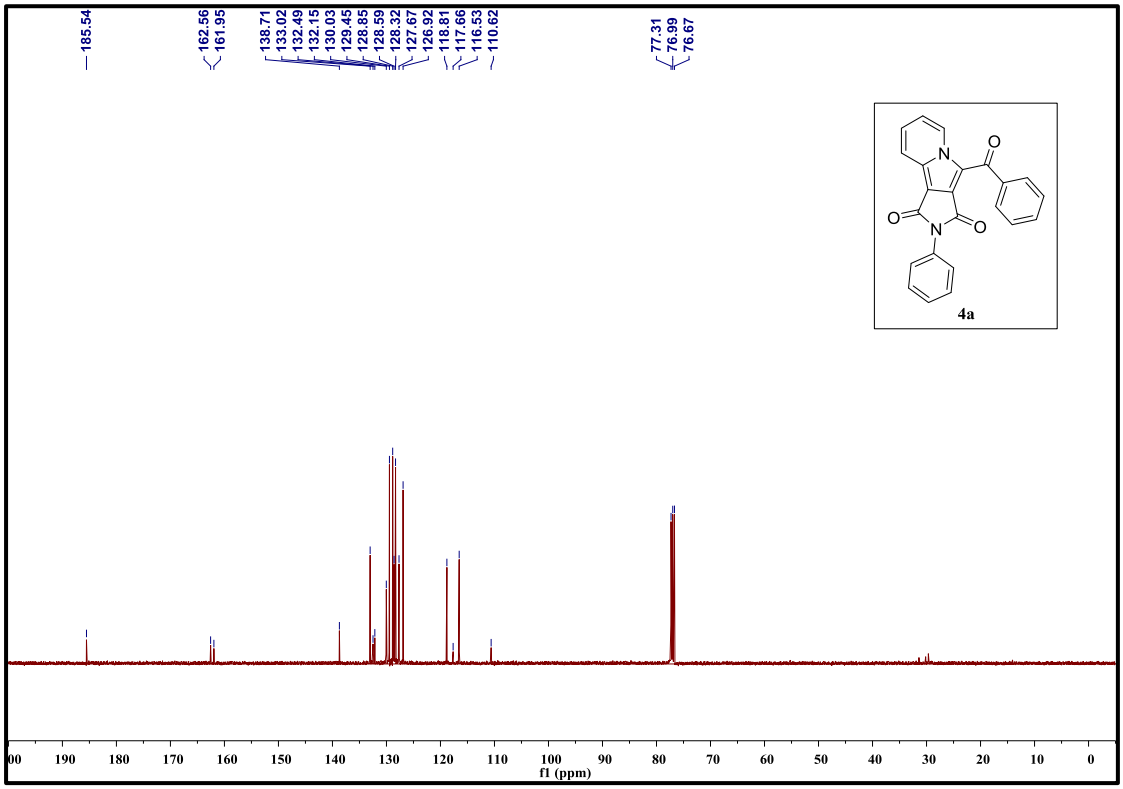


Yellow solid (172.8 mg, 83%); mp 220-222 °C; R_f = 0.45 (petroleum ether/ethyl acetate = 3:1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23- 8.14 (m, 2H), 7.82 (s, 2H), 7.78 (d, *J* = 7.7 Hz, 1H), 7.73-7.66 (m, 2H), 7.58-7.48 (m, 4H), 7.44-7.38 (m, 2H), 7.36 -7.27 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.09, 162.67, 161.95, 137.09, 134.57, 133.41, 132.21, 131.92, 130.50, 129.74, 129.62, 128.83, 128.80, 128.78, 128.45, 127.68, 126.87, 126.35, 125.31, 123.41, 119.33, 116.52, 111.64. HRMS (TOF MS ES+): m/z $[M+H]^+$ calcd for $C_{27}H_{16}N_2O_3$: 417.1163; found: 417.1166.

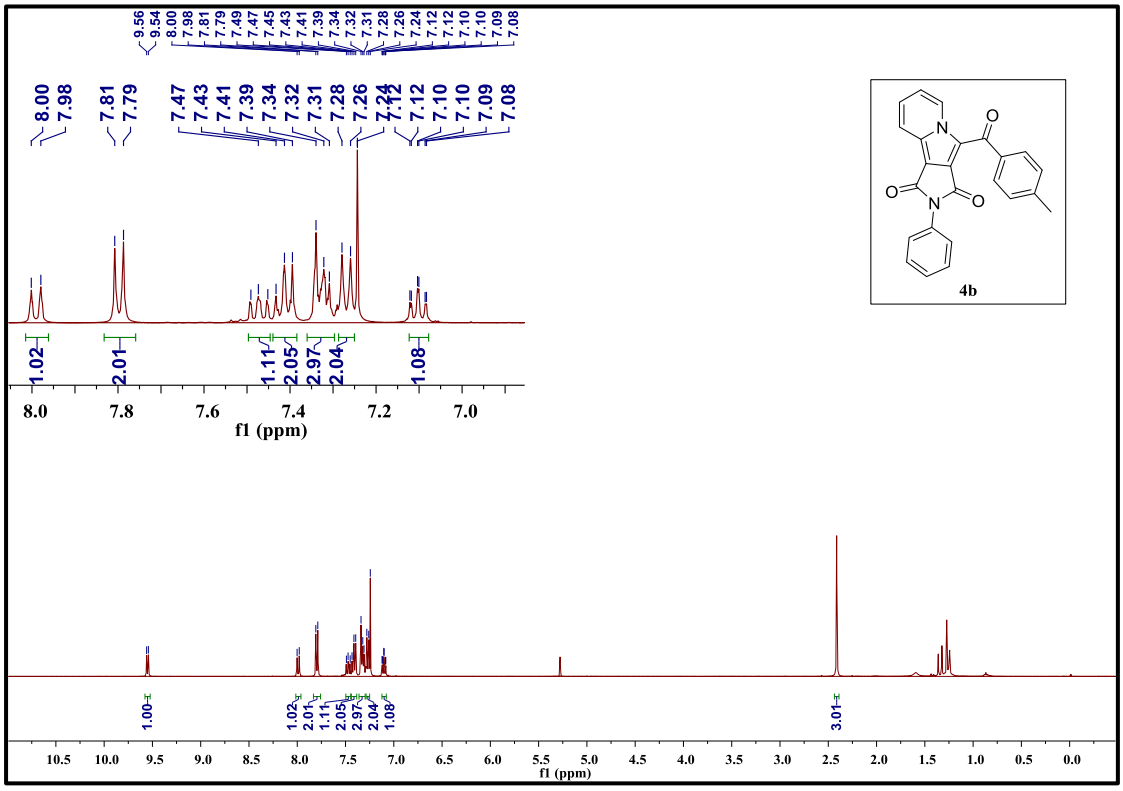
4. Appendix: spectral copies of 1H NMR, and 13C NMR

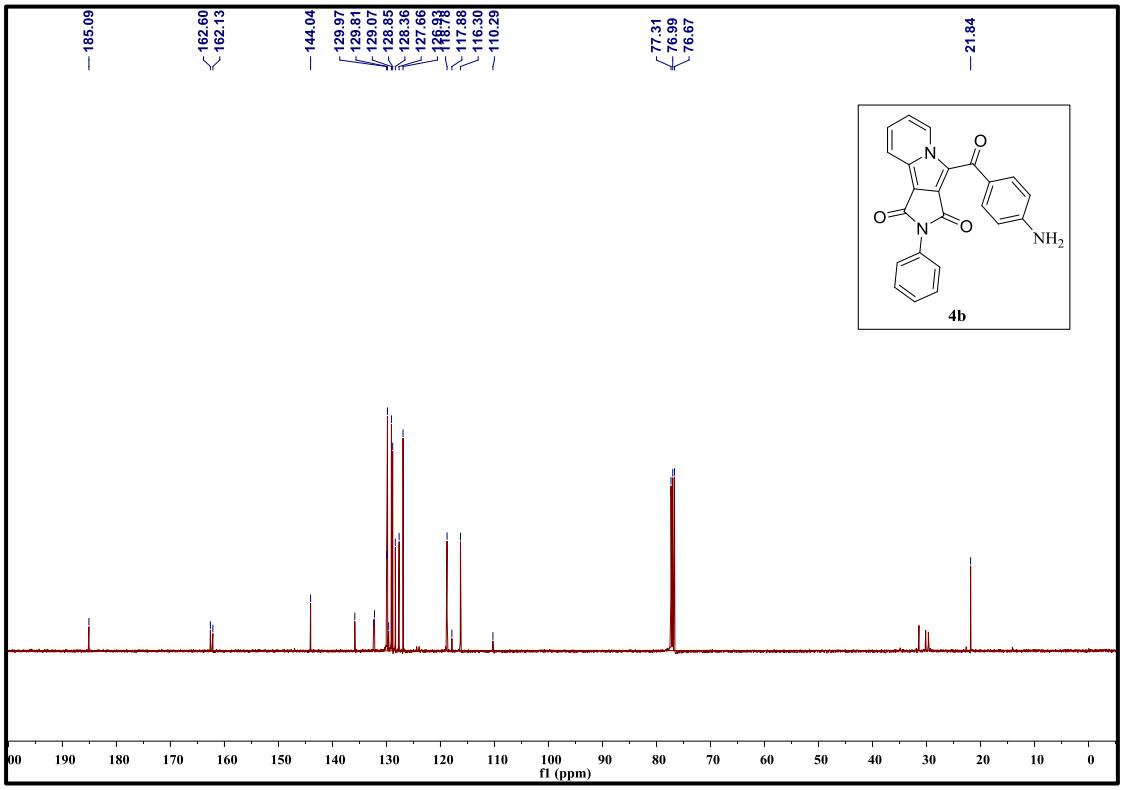
Compound 4a



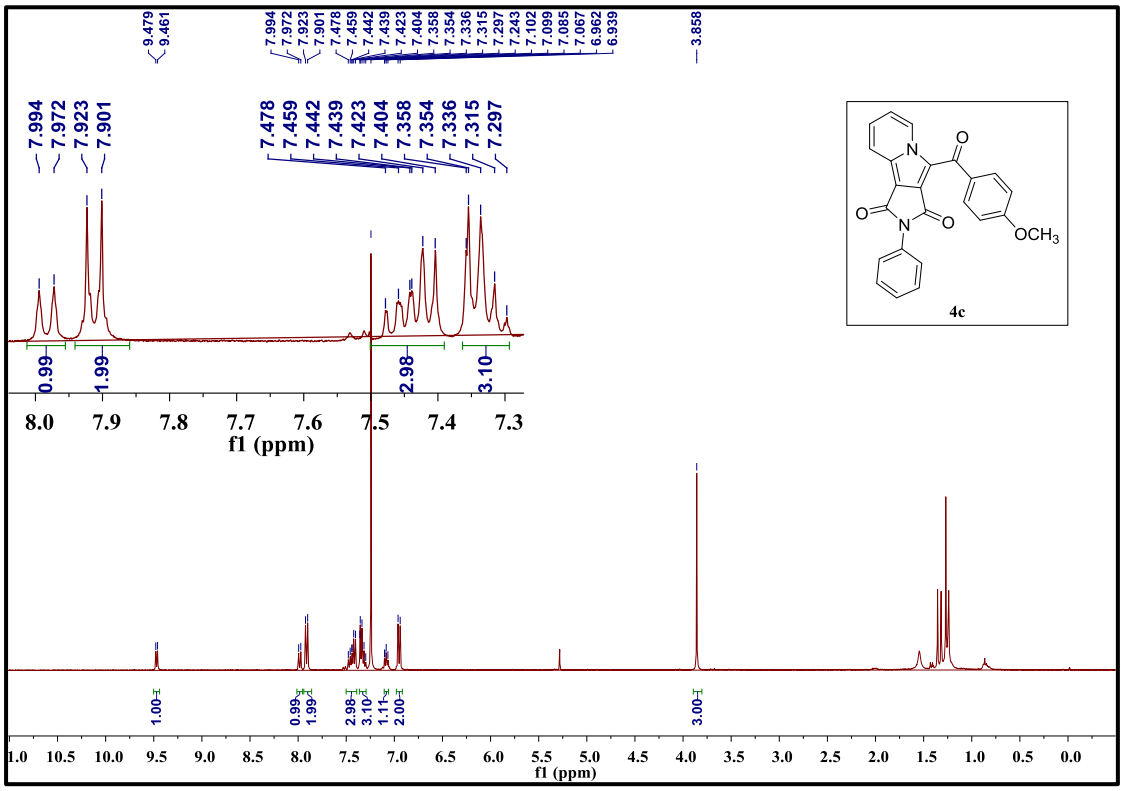


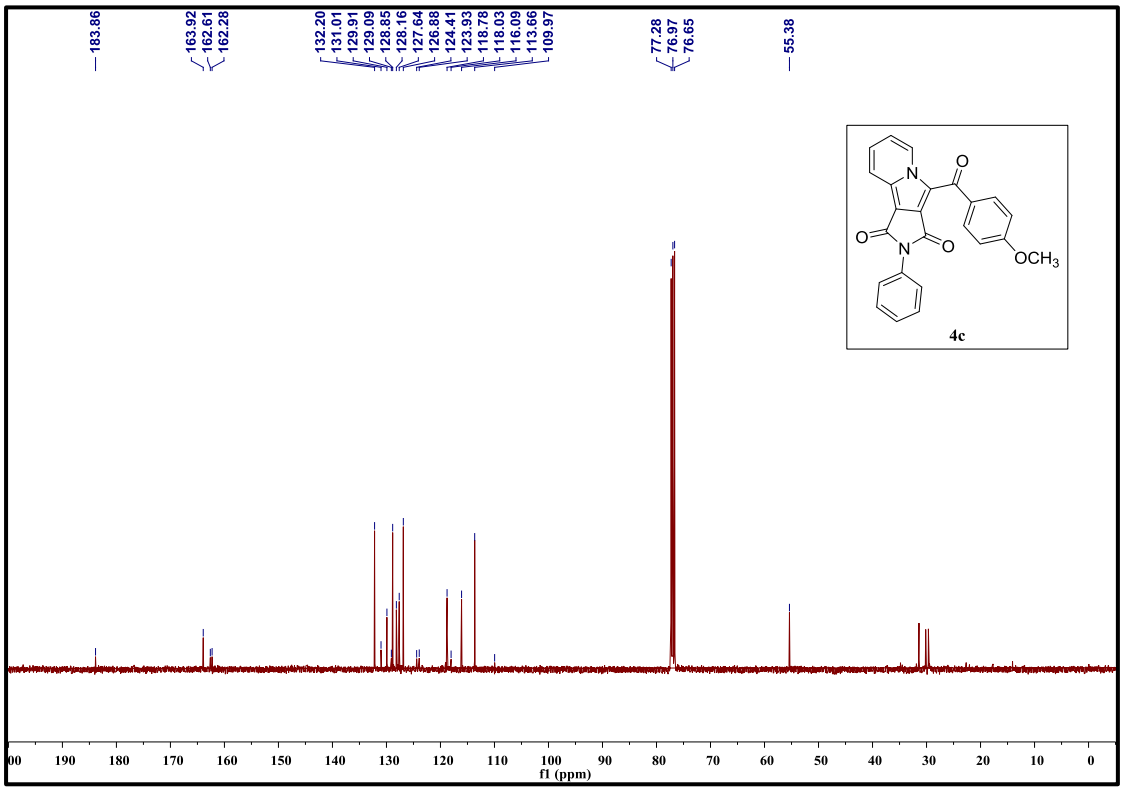
Compound 4b


9.56
9.54
8.00
7.98
7.81
7.79
7.49
7.47
7.45
7.43
7.41
7.39
7.34
7.32
7.31
7.28
7.26
7.24
7.12
7.10
7.09
7.08
1.02
2.01
1.11
2.05
2.97
2.04
1.08
8.0
7.8
7.6
7.4
7.2
7.0
f1 (ppm)
4b
1.00
3.01
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

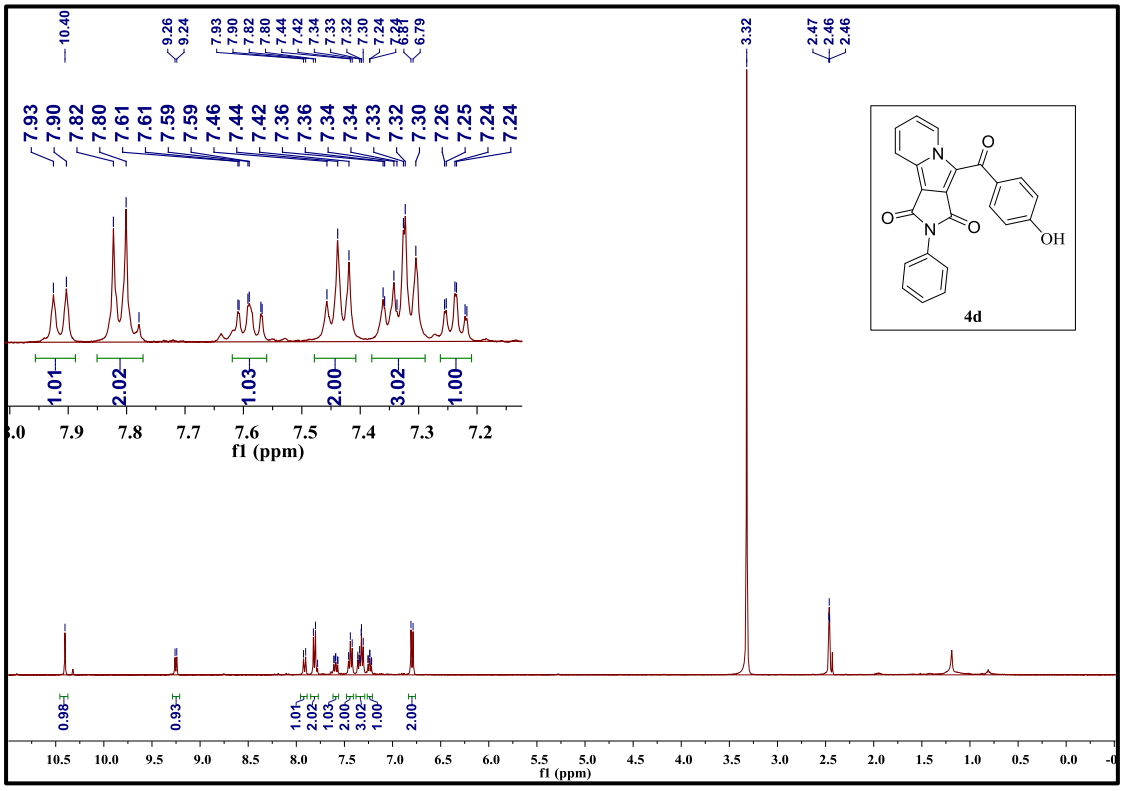

185.09
162.60
162.13
144.04
129.97
129.81
129.07
128.85
128.36
127.66
117.88
116.30
110.29
77.31
76.99
76.67
21.84
NH2
4b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

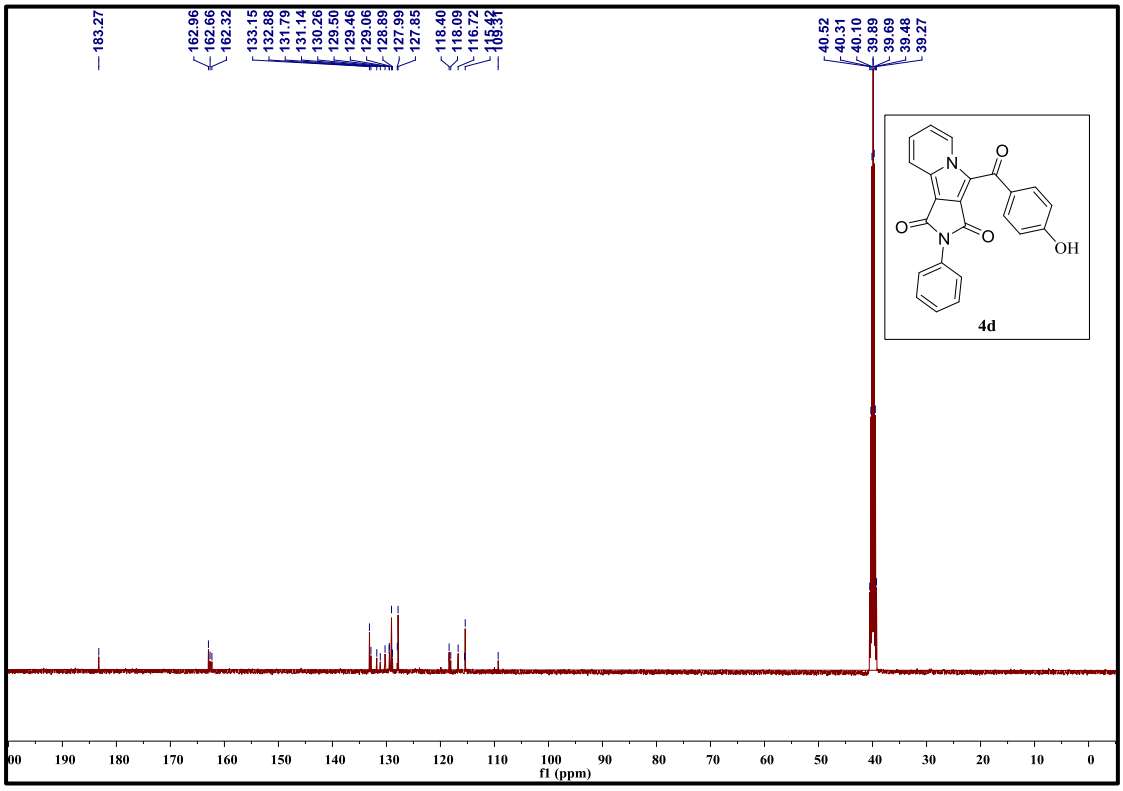
Compound 4c


9.479
9.461
7.994
7.972
7.923
7.901
7.478
7.459
7.442
7.439
7.423
7.404
7.358
7.354
7.336
7.315
7.297
7.243
7.102
7.099
7.085
7.067
6.962
6.939
3.858
0.99
1.99
2.98
3.10
1.00
0.99
1.99
2.98
3.10
1.11
2.00
3.00
f1 (ppm)
4c

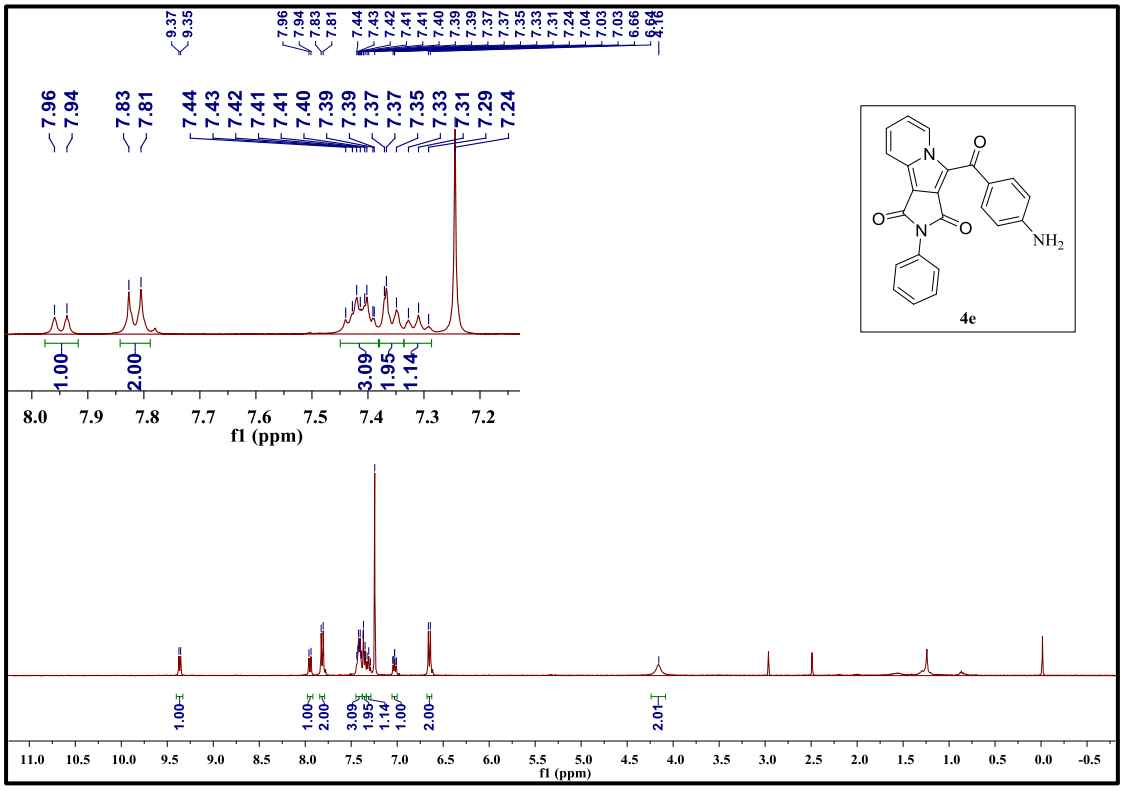

183.86
163.92
162.61
162.28
132.20
131.01
129.91
129.09
128.85
128.16
127.64
126.88
124.41
123.93
118.78
118.03
116.09
113.66
109.97
77.28
76.97
76.65
55.38
f1 (ppm)
4c

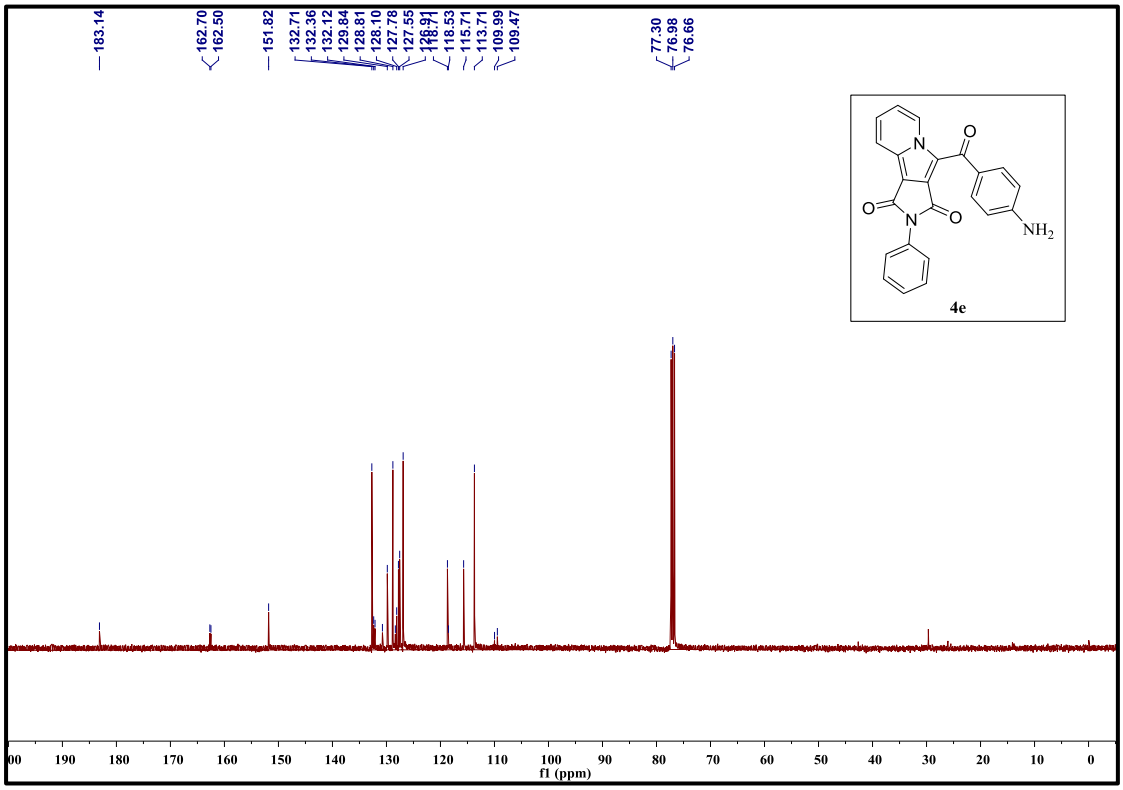
Compound 4d


10.40
9.26
9.24
7.93
7.90
7.82
7.80
7.61
7.59
7.46
7.44
7.42
7.36
7.34
7.33
7.32
7.30
7.26
7.25
7.24
6.81
6.79
3.32
2.47
2.46
2.46
1.01
2.02
1.03
2.00
3.02
1.00
0.98
0.93
f1 (ppm)
4d

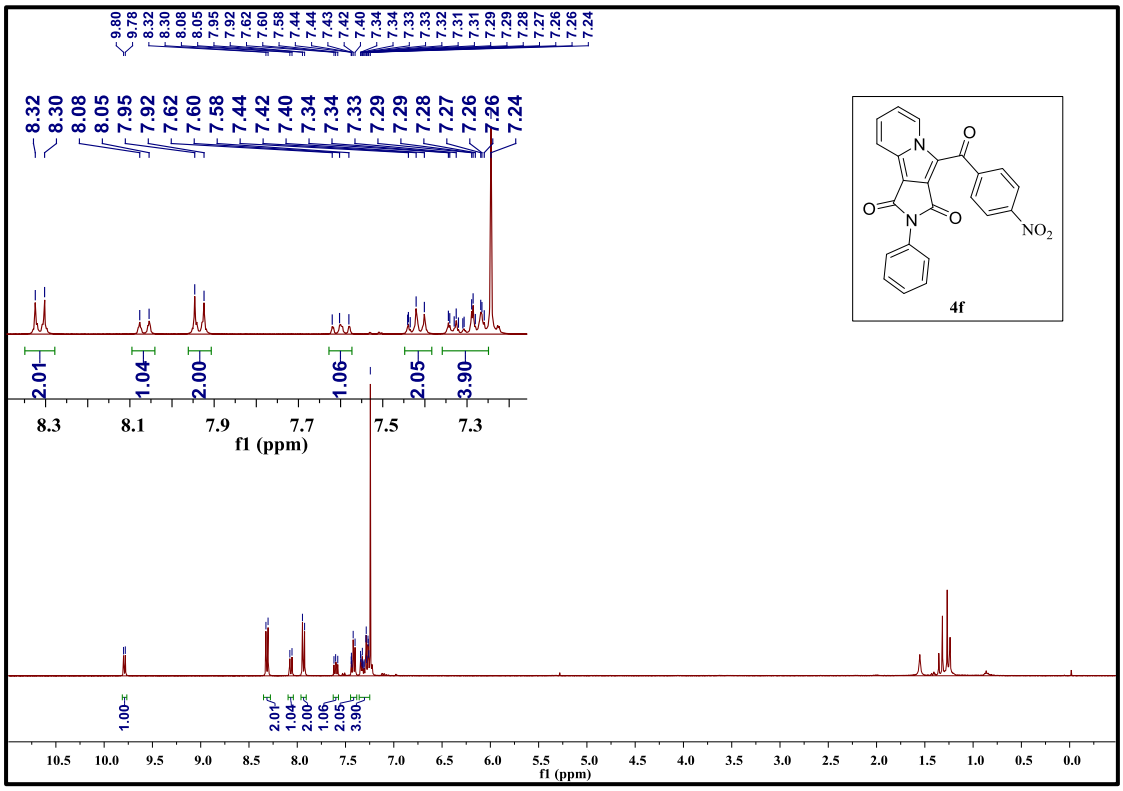

183.27
162.96
162.66
162.32
133.15
132.88
131.79
131.14
130.26
129.50
129.46
129.06
128.89
127.99
127.85
118.40
118.09
116.72
115.42
108.31
40.52
40.31
40.10
39.89
39.69
39.48
39.27
f1 (ppm)
4d

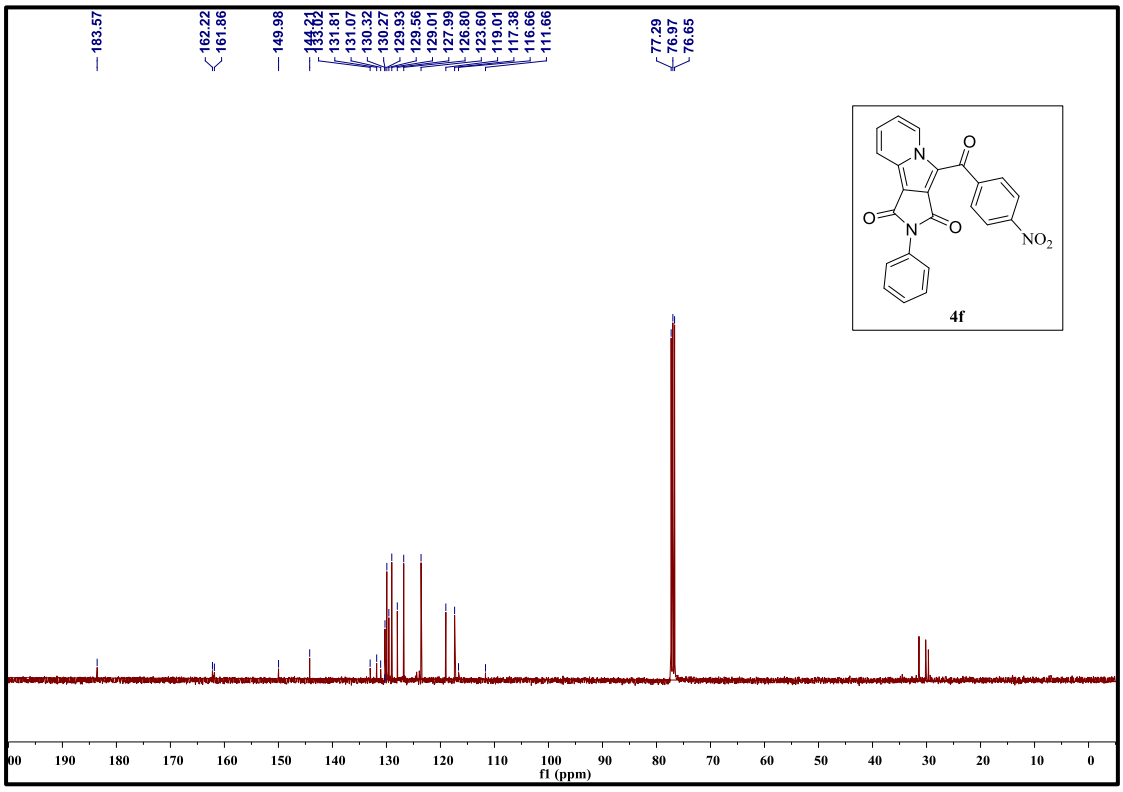
Compound 4e


9.37
9.35
7.96
7.94
7.83
7.81
7.44
7.43
7.42
7.41
7.41
7.40
7.39
7.39
7.37
7.37
7.35
7.33
7.31
7.29
7.24
7.04
7.03
7.03
6.66
6.64
6.16
1.00
2.00
3.09
1.95
1.14
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
f1 (ppm)
4e
1.00
2.00
3.09
1.95
1.14
1.00
2.00
2.01
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

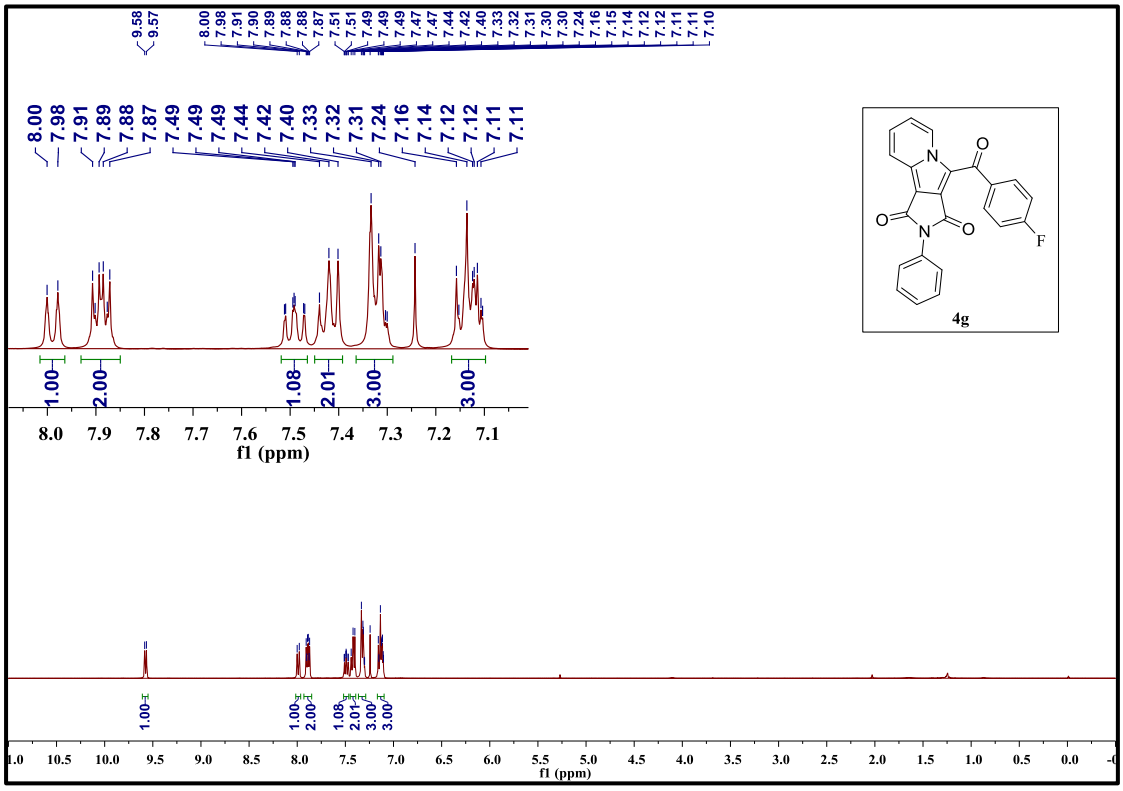

183.14
162.70
162.50
151.82
132.71
132.36
132.12
129.84
128.81
128.10
127.78
127.55
118.53
115.71
113.71
109.99
109.47
77.30
76.98
76.66
4e
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

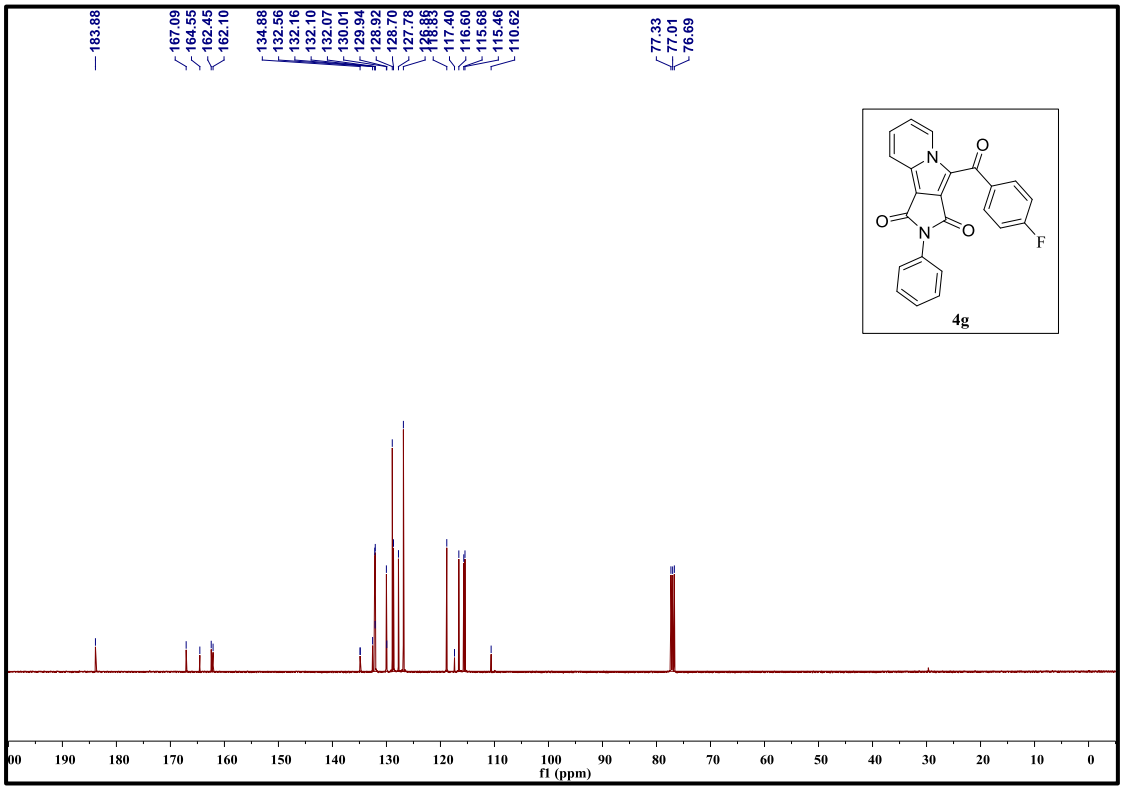
Compound 4f


9.80
9.78
8.32
8.30
8.08
8.05
7.95
7.92
7.62
7.60
7.58
7.44
7.44
7.43
7.42
7.40
7.34
7.34
7.33
7.33
7.32
7.31
7.31
7.29
7.29
7.28
7.27
7.26
7.26
7.24
2.01
1.04
2.00
1.06
2.05
3.90
f1 (ppm)
4f

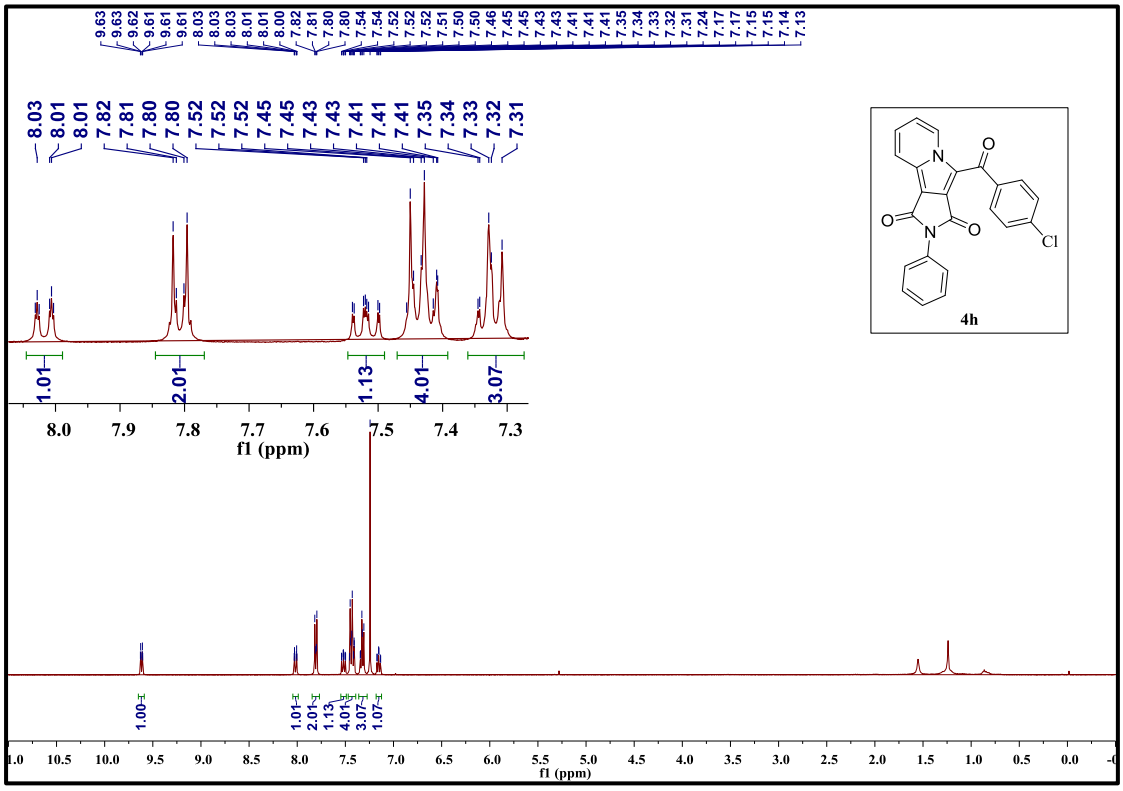

183.57
162.22
161.86
149.98
144.21
133.02
131.81
131.07
130.32
130.27
129.93
129.56
129.01
127.99
126.80
123.60
119.01
117.38
116.66
111.66
77.29
76.97
76.65
f1 (ppm)
4f

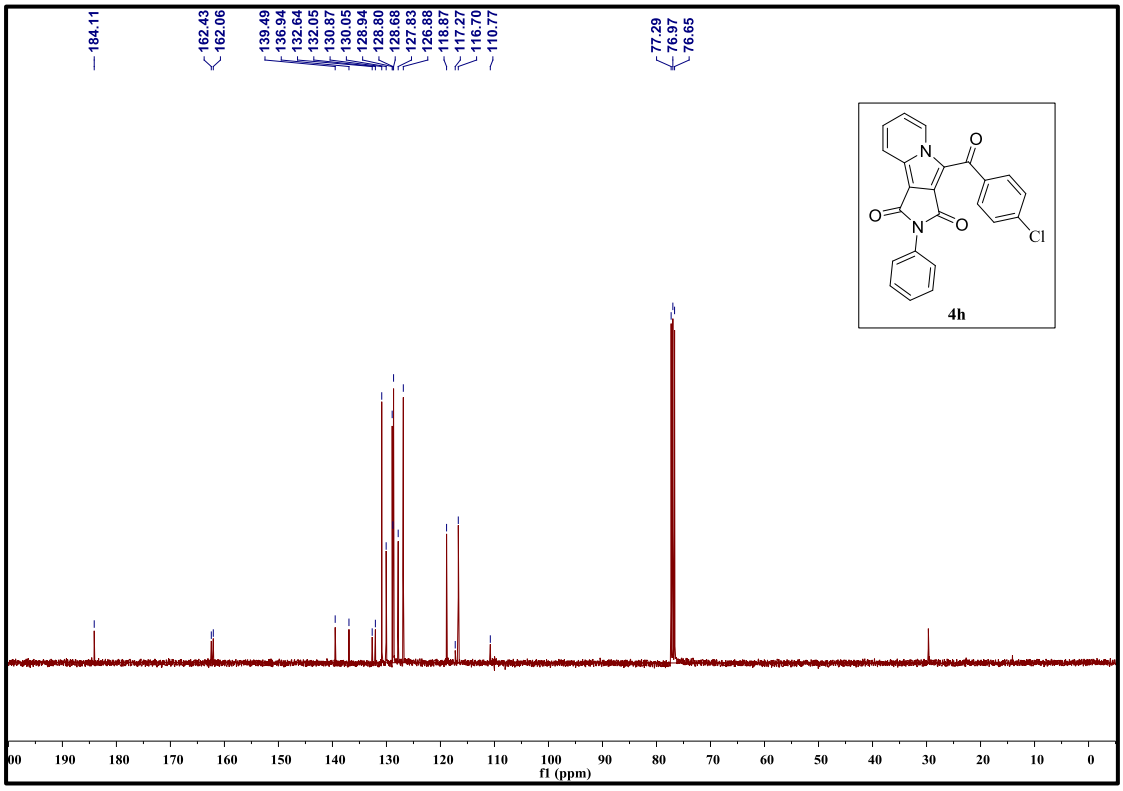
Compound 4g


9.58
9.57
8.00
7.98
7.91
7.90
7.89
7.88
7.87
7.51
7.49
7.49
7.47
7.47
7.44
7.42
7.40
7.33
7.32
7.31
7.30
7.30
7.24
7.16
7.15
7.14
7.12
7.12
7.11
7.11
7.10
1.00
2.00
1.08
2.01
3.00
3.00
f1 (ppm)
4g

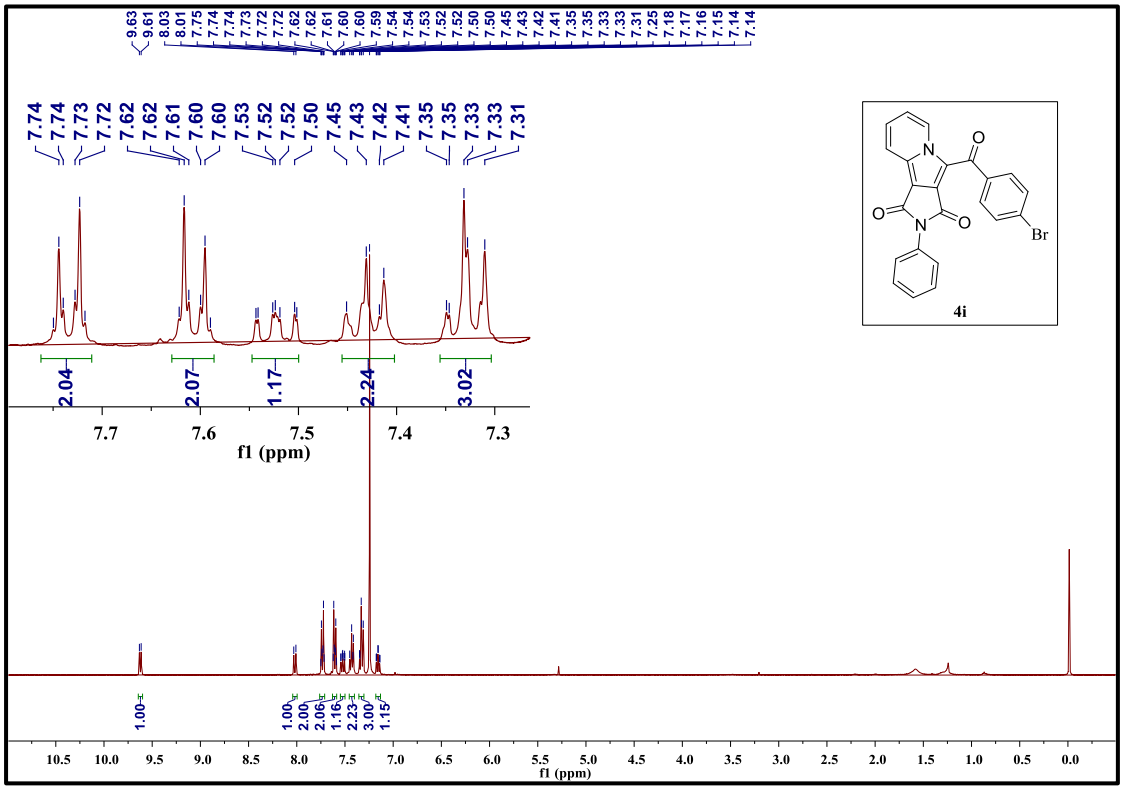

183.88
167.09
164.55
162.45
162.10
134.88
132.56
132.16
132.10
132.07
130.01
129.94
128.92
128.70
127.78
117.40
116.60
115.68
115.46
110.62
77.33
77.01
76.69
f1 (ppm)
4g

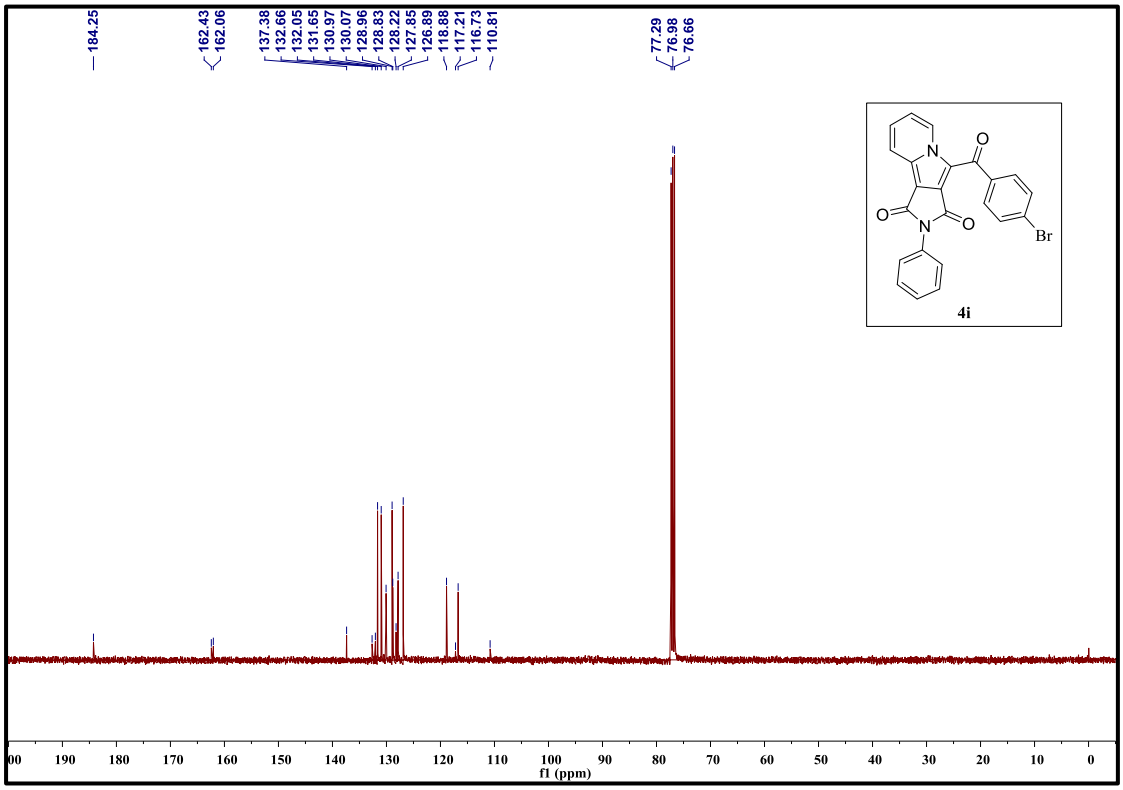
Compound 4h



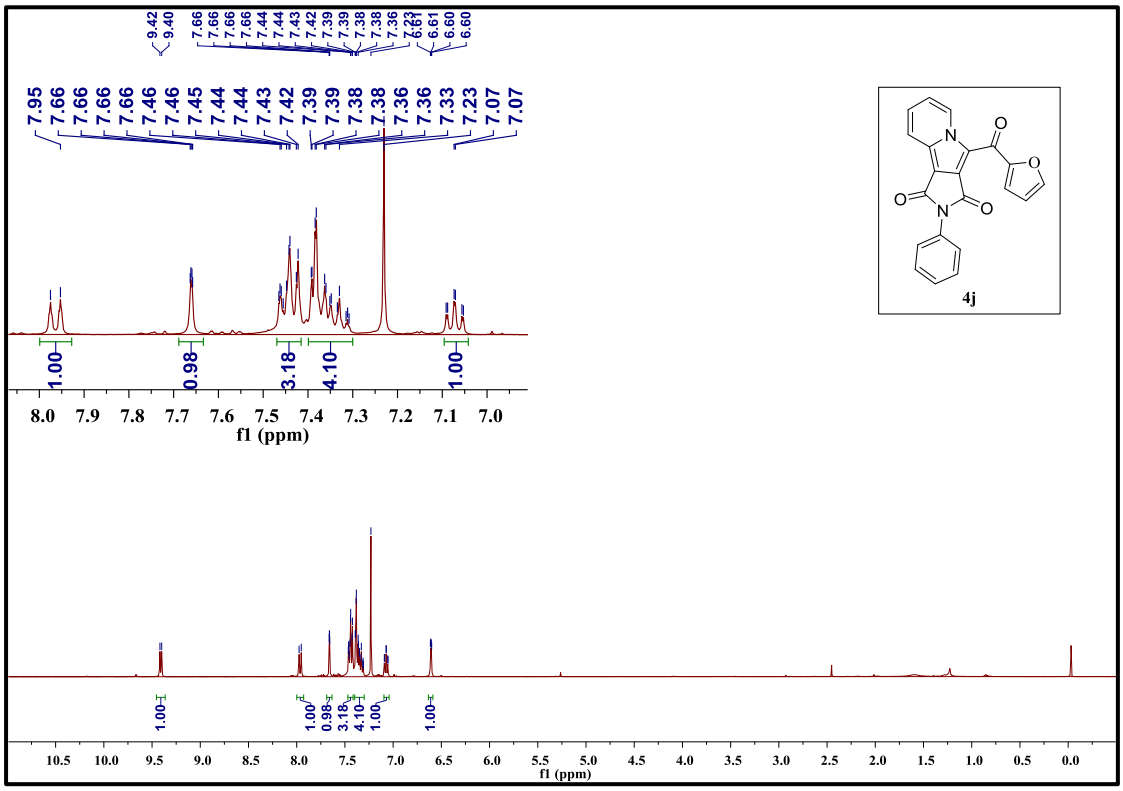


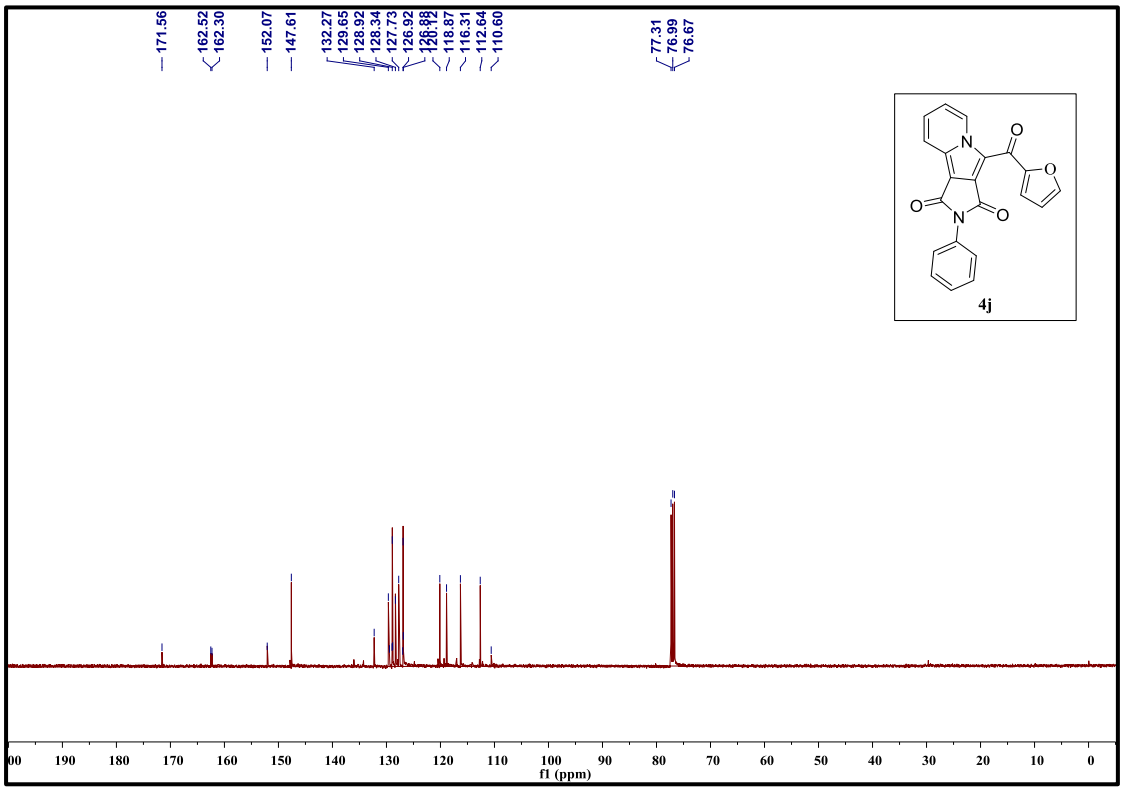
Compound 4i


9.63 9.61 8.03 8.01 7.75 7.74 7.74 7.73 7.72 7.72 7.62 7.62 7.61 7.60 7.60 7.59 7.54 7.54 7.53 7.52 7.52 7.50 7.50 7.45 7.43 7.42 7.41 7.35 7.35 7.33 7.33 7.31 7.25 7.18 7.17 7.16 7.15 7.14 7.14
7.74 7.74 7.73 7.72 7.62 7.62 7.61 7.60 7.60 7.53 7.52 7.52 7.50 7.45 7.43 7.42 7.41 7.35 7.35 7.33 7.33 7.31
2.04 2.07 1.17 2.24 3.02
7.7 7.6 7.5 7.4 7.3
f1 (ppm)
4i
1.00 1.00 2.00 2.06 1.16 2.23 3.00 1.15
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

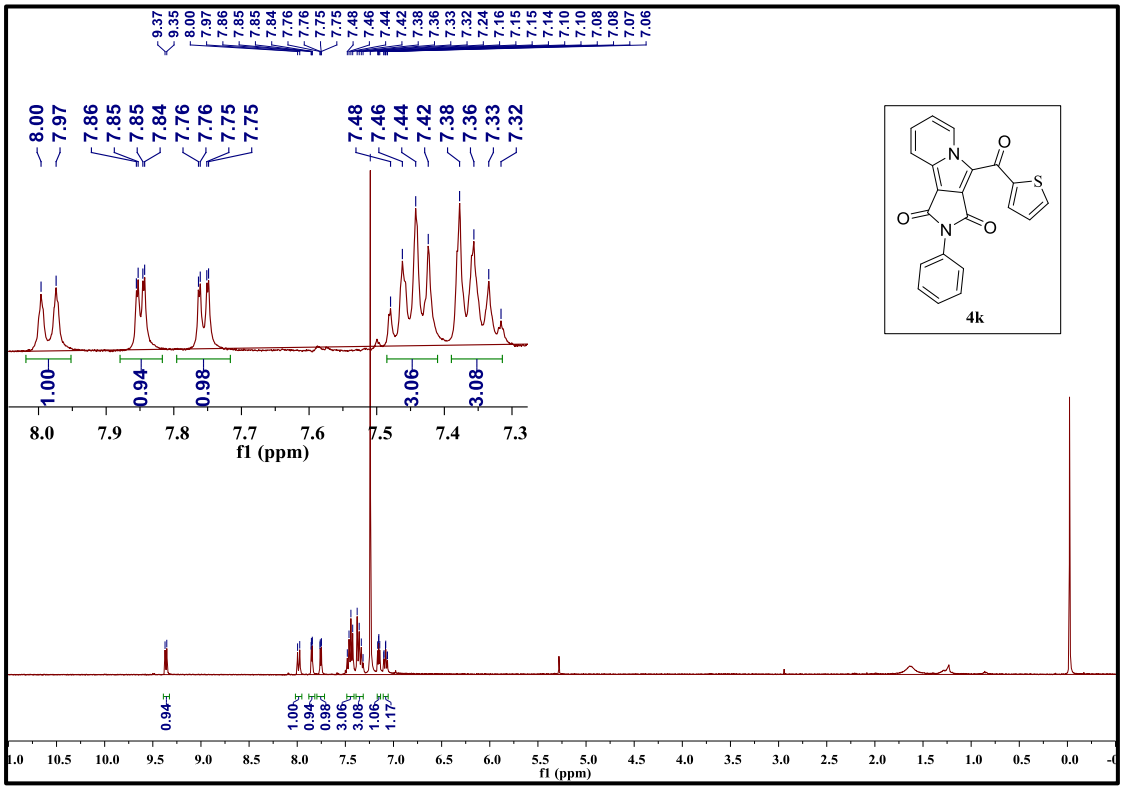

184.25
162.43 162.06
137.38 132.66 132.05 131.65 130.97 130.07 128.96 128.83 128.22 127.85 126.89 118.88 117.21 116.73 110.81
77.29 76.98 76.66
4i
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

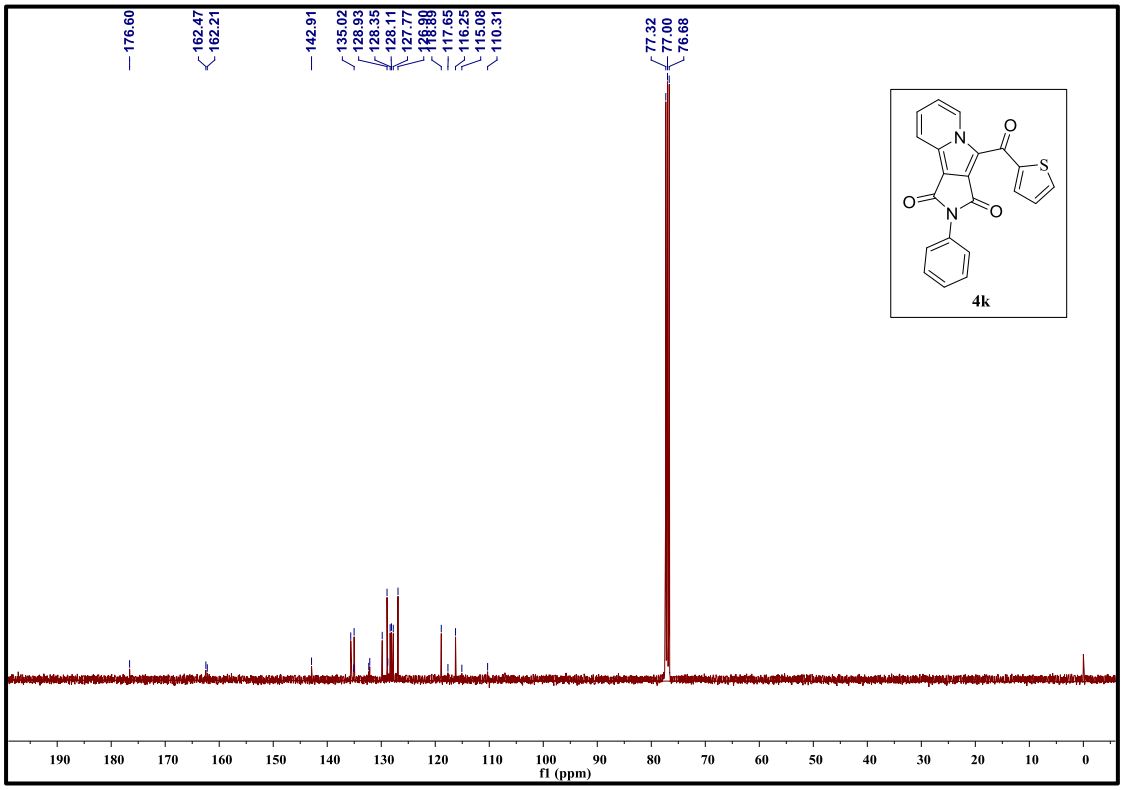
Compound 4j


9.42
9.40
7.66
7.66
7.66
7.66
7.44
7.44
7.43
7.42
7.39
7.39
7.38
7.38
7.36
7.33
6.61
6.60
6.60
7.95
7.66
7.66
7.66
7.46
7.46
7.45
7.44
7.44
7.43
7.42
7.39
7.39
7.38
7.38
7.36
7.36
7.33
7.23
7.07
7.07
1.00
0.98
3.18
4.10
1.00
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0
f1 (ppm)
4j
1.00
1.00
0.98
3.18
4.10
1.00
1.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

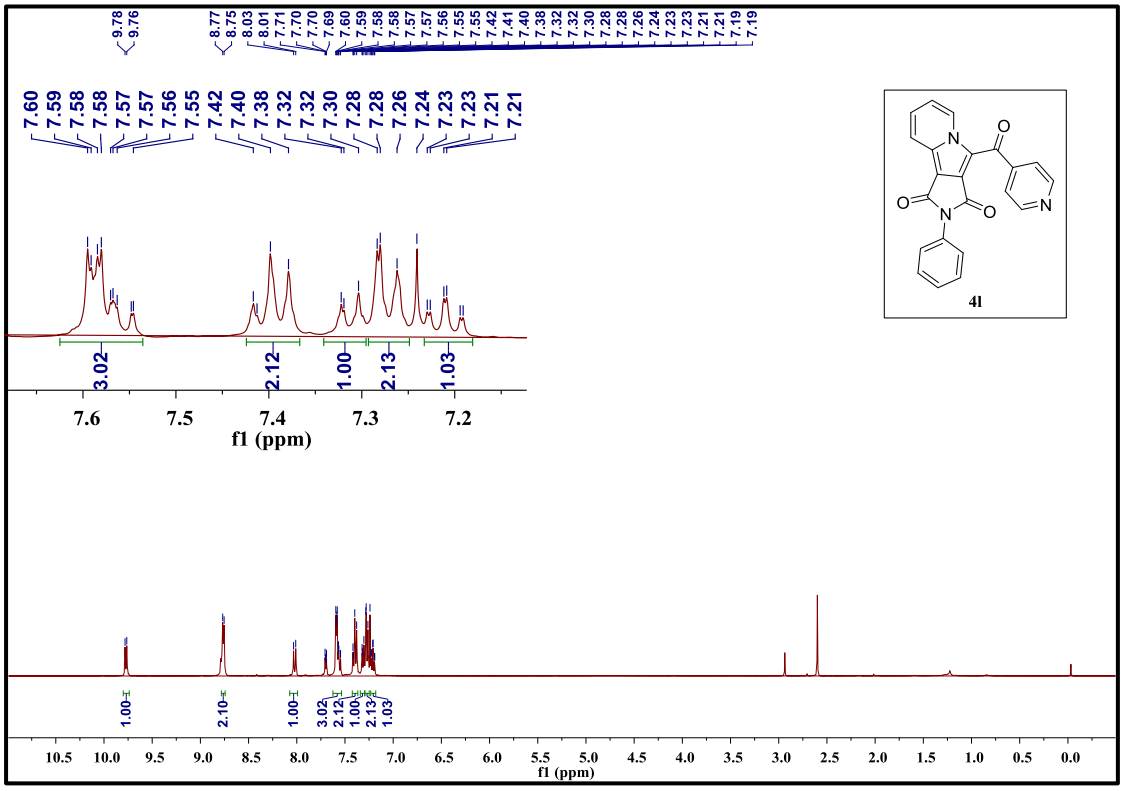

171.56
162.52
162.30
152.07
147.61
132.27
129.65
128.92
128.34
127.73
126.92
118.87
116.31
112.64
110.60
77.31
76.99
76.67
4j
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

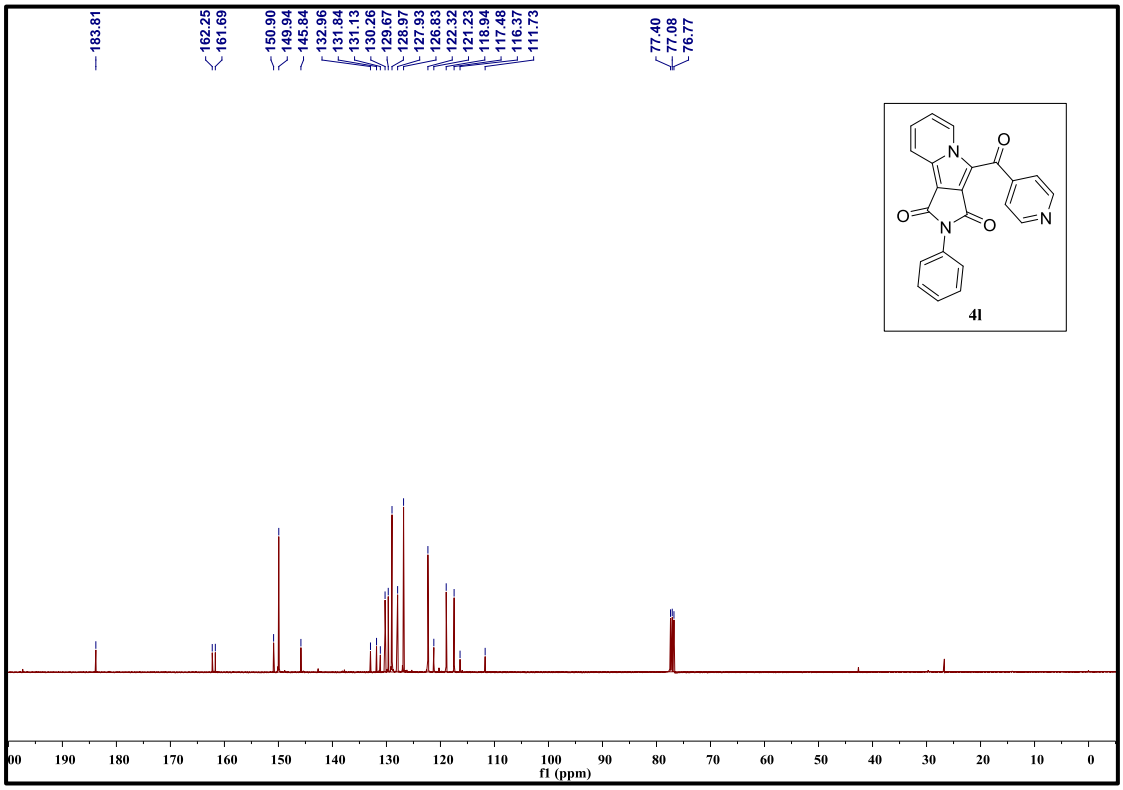
Compound 4k


9.37
9.35
8.00
7.97
7.86
7.85
7.85
7.84
7.76
7.76
7.75
7.75
7.48
7.46
7.44
7.42
7.38
7.36
7.33
7.32
7.24
7.16
7.15
7.15
7.14
7.10
7.10
7.08
7.08
7.07
7.06
1.00
0.94
0.98
3.06
3.08
f1 (ppm)
4k
0.94
1.00
0.94
0.98
3.06
3.08
1.06
1.17
f1 (ppm)

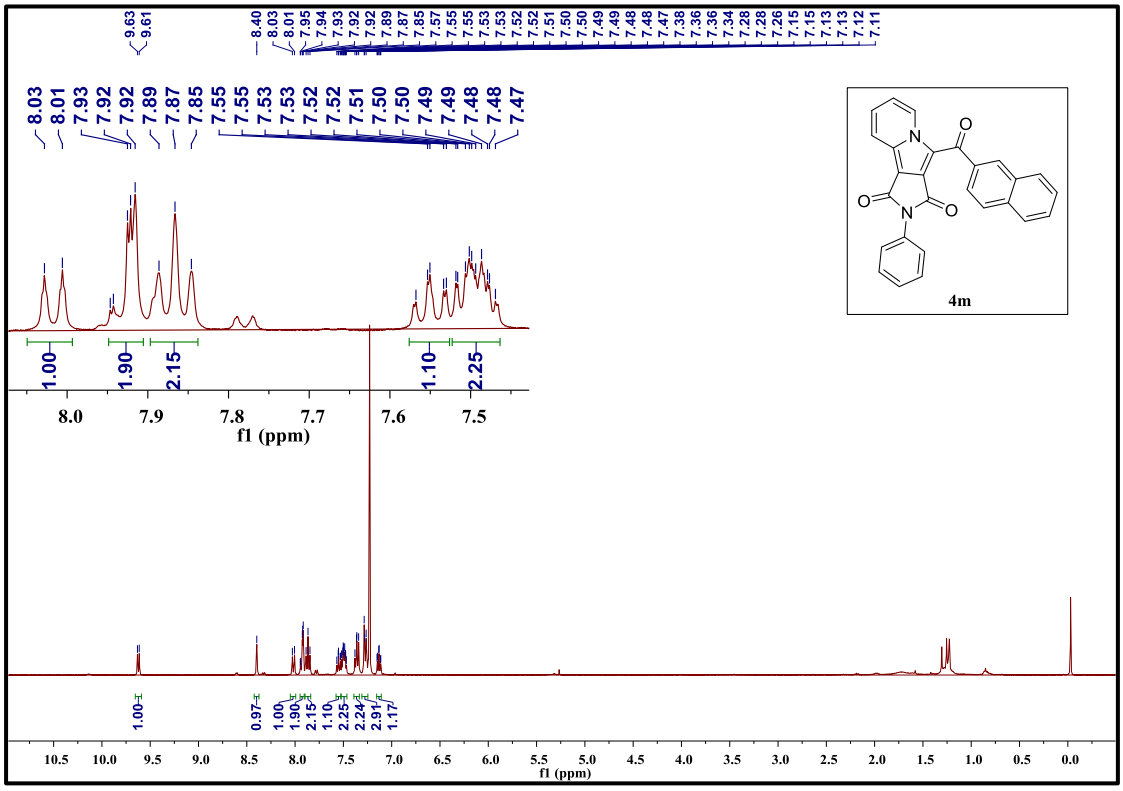

176.60
162.47
162.21
142.91
135.02
128.93
128.35
128.11
127.77
117.65
116.25
115.08
110.31
77.32
77.00
76.68
4k
f1 (ppm)

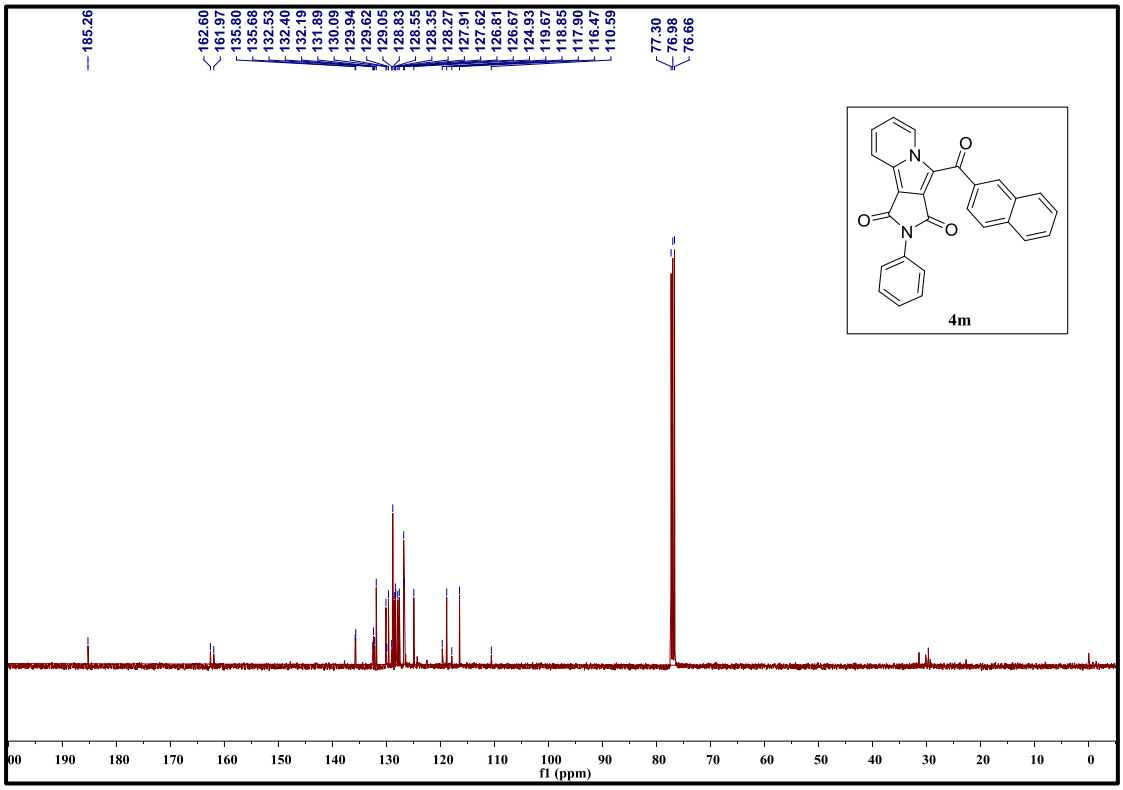
Compound 4l


9.78
9.76
8.77
8.75
8.03
8.01
7.71
7.70
7.70
7.69
7.60
7.59
7.58
7.58
7.57
7.57
7.56
7.55
7.55
7.42
7.41
7.40
7.38
7.32
7.32
7.30
7.28
7.28
7.26
7.24
7.23
7.23
7.21
7.21
7.19
7.19
7.60
7.59
7.58
7.58
7.57
7.57
7.56
7.55
7.42
7.40
7.38
7.32
7.32
7.30
7.28
7.28
7.26
7.24
7.23
7.23
7.21
7.21
3.02
2.12
1.00
2.13
1.03
7.6
7.5
7.4
7.3
7.2
f1 (ppm)
4l
1.00
2.10
1.00
3.02
2.12
1.00
2.13
1.03
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

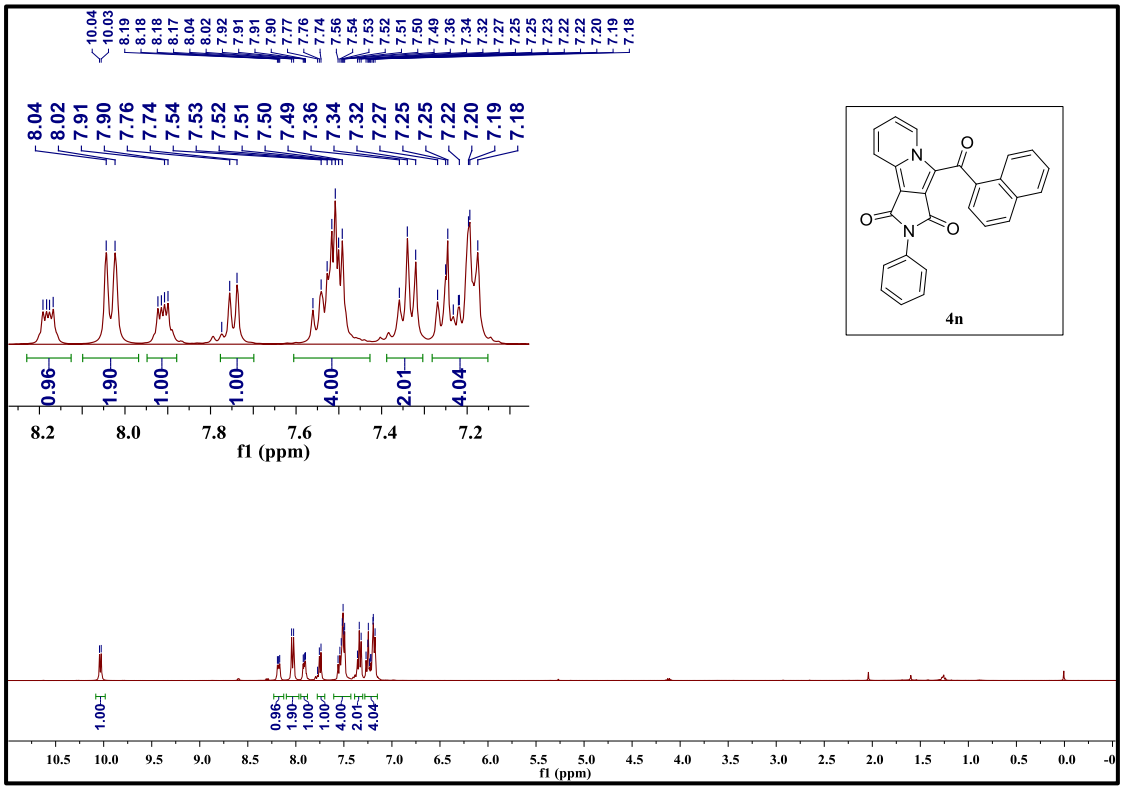

183.81
162.25
161.69
150.90
149.94
145.84
132.96
131.84
131.13
130.26
129.67
128.97
127.93
126.83
122.32
121.23
118.94
117.48
116.37
111.73
77.40
77.08
76.77
4l
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

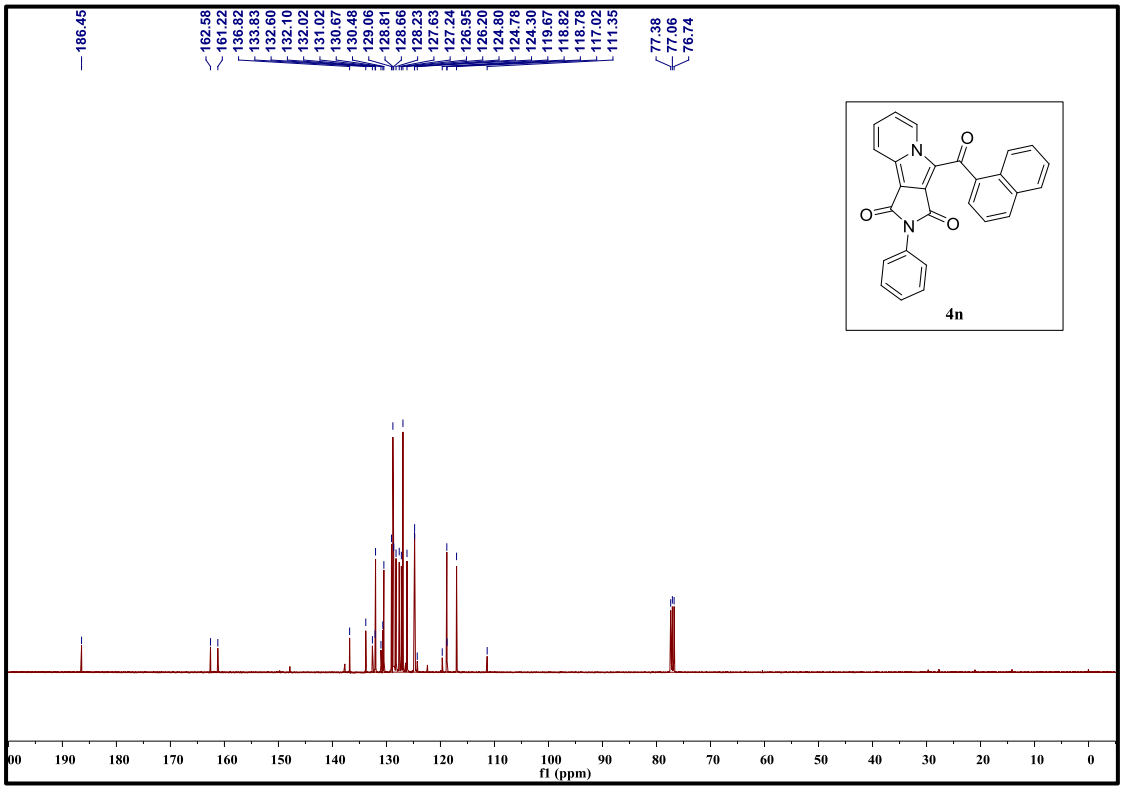
Compound 4m


9.63
9.61
8.40
8.03
8.01
7.95
7.94
7.93
7.92
7.92
7.89
7.87
7.85
7.57
7.55
7.55
7.53
7.53
7.52
7.52
7.51
7.50
7.50
7.49
7.49
7.48
7.48
7.47
7.38
7.36
7.36
7.34
7.28
7.28
7.26
7.15
7.15
7.13
7.13
7.12
7.11
8.03
8.01
7.93
7.92
7.92
7.89
7.87
7.85
7.55
7.55
7.53
7.53
7.52
7.52
7.51
7.50
7.50
7.49
7.49
7.48
7.48
7.47
1.00
1.90
2.15
1.10
2.25
8.0
7.9
7.8
7.7
7.6
7.5
f1 (ppm)
4m
1.00
0.97
1.00
1.90
2.15
1.10
2.25
2.24
2.91
1.17
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

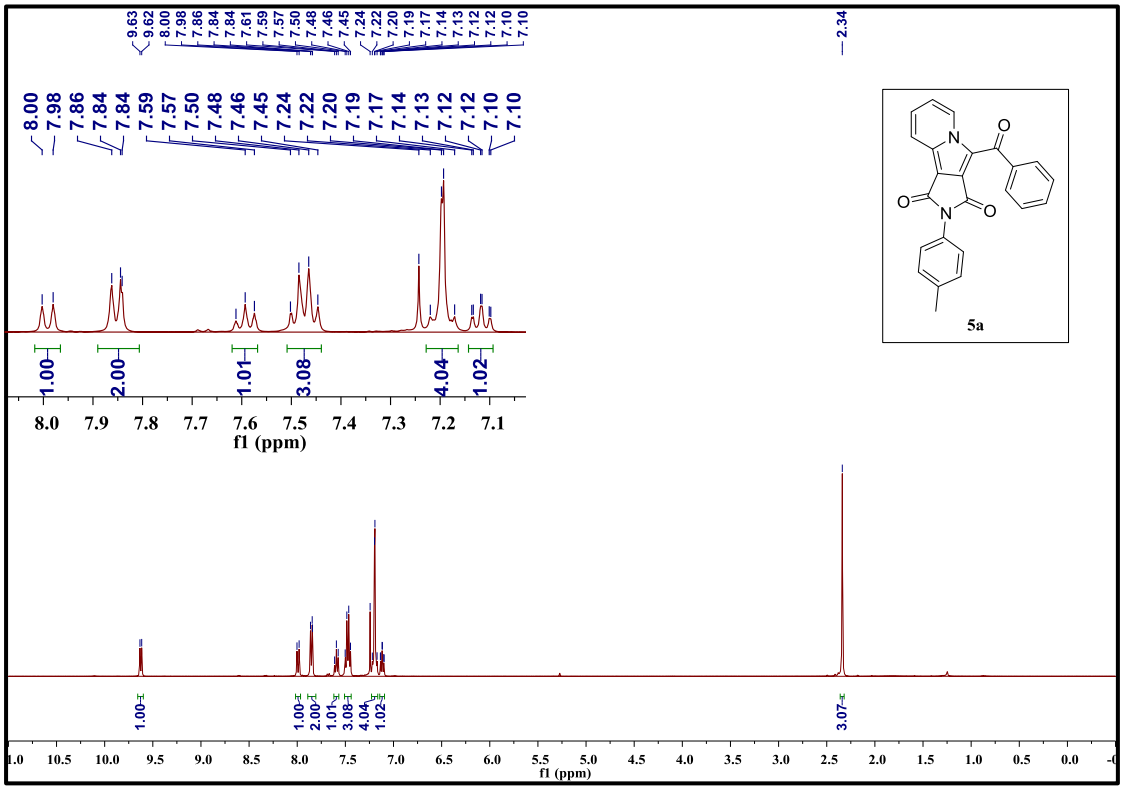

185.26
162.60
161.97
135.80
135.68
132.53
132.40
132.19
131.89
130.09
129.94
129.62
129.05
128.83
128.55
128.35
128.27
127.91
127.62
126.81
126.67
124.93
119.67
118.85
117.90
116.47
110.59
77.30
76.98
76.66
4m
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

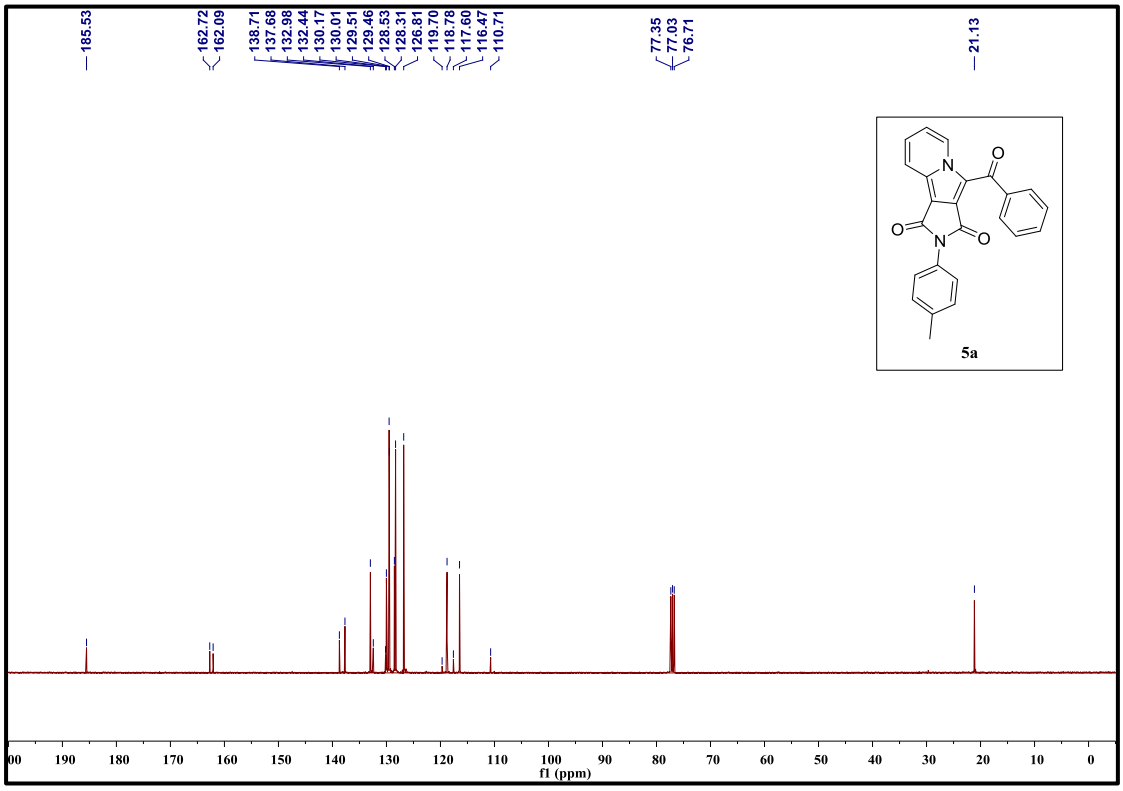
Compound 4n


10.04
10.03
8.19
8.18
8.18
8.17
8.04
8.02
7.92
7.91
7.91
7.90
7.77
7.76
7.74
7.56
7.54
7.53
7.52
7.51
7.50
7.49
7.36
7.34
7.32
7.27
7.25
7.25
7.23
7.22
7.22
7.20
7.19
7.18
8.04
8.02
7.91
7.90
7.76
7.74
7.54
7.53
7.52
7.51
7.50
7.49
7.36
7.34
7.32
7.27
7.25
7.25
7.22
7.20
7.19
7.18
0.96
1.90
1.00
1.00
4.00
2.01
4.04
8.2
8.0
7.8
7.6
7.4
7.2
f1 (ppm)
4n
1.00
0.96
1.90
1.00
1.00
4.00
2.01
4.04
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

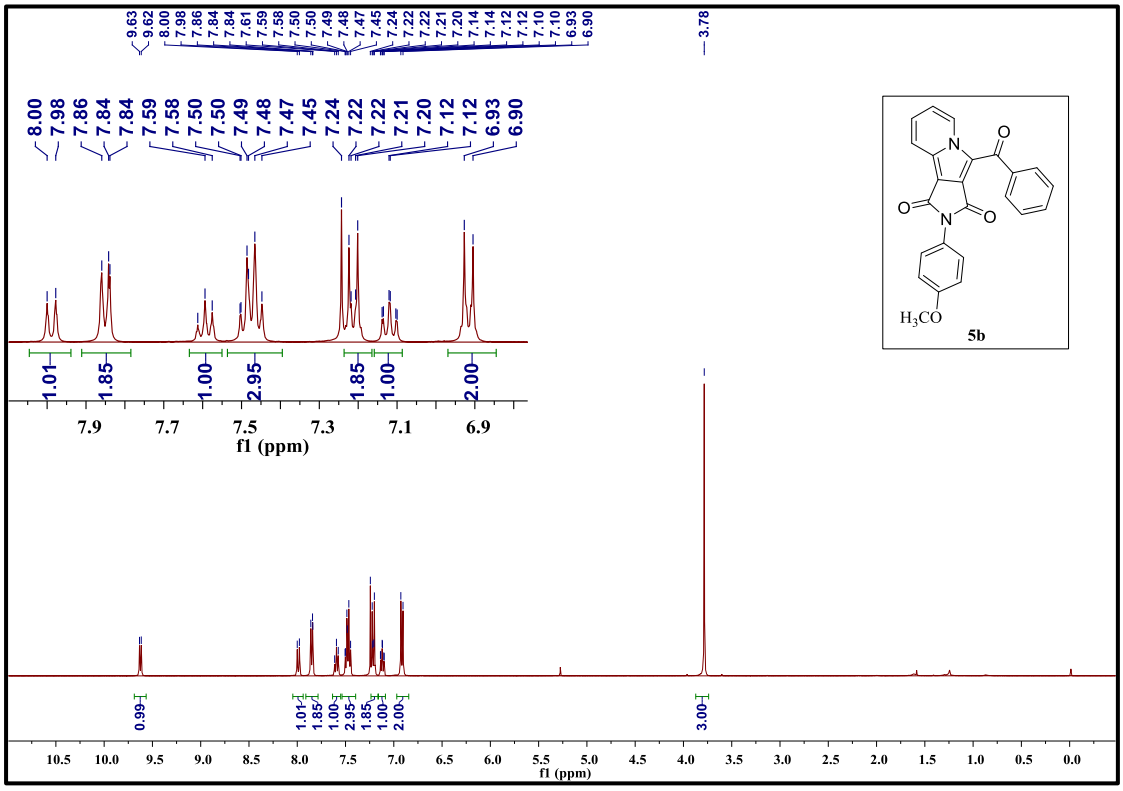

186.45
162.58
161.22
136.82
133.83
132.60
132.10
132.02
131.02
130.67
130.48
129.06
128.81
128.66
128.23
127.63
127.24
126.95
126.20
124.80
124.78
124.30
119.67
118.82
118.78
117.02
111.35
77.38
77.06
76.74
4n
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

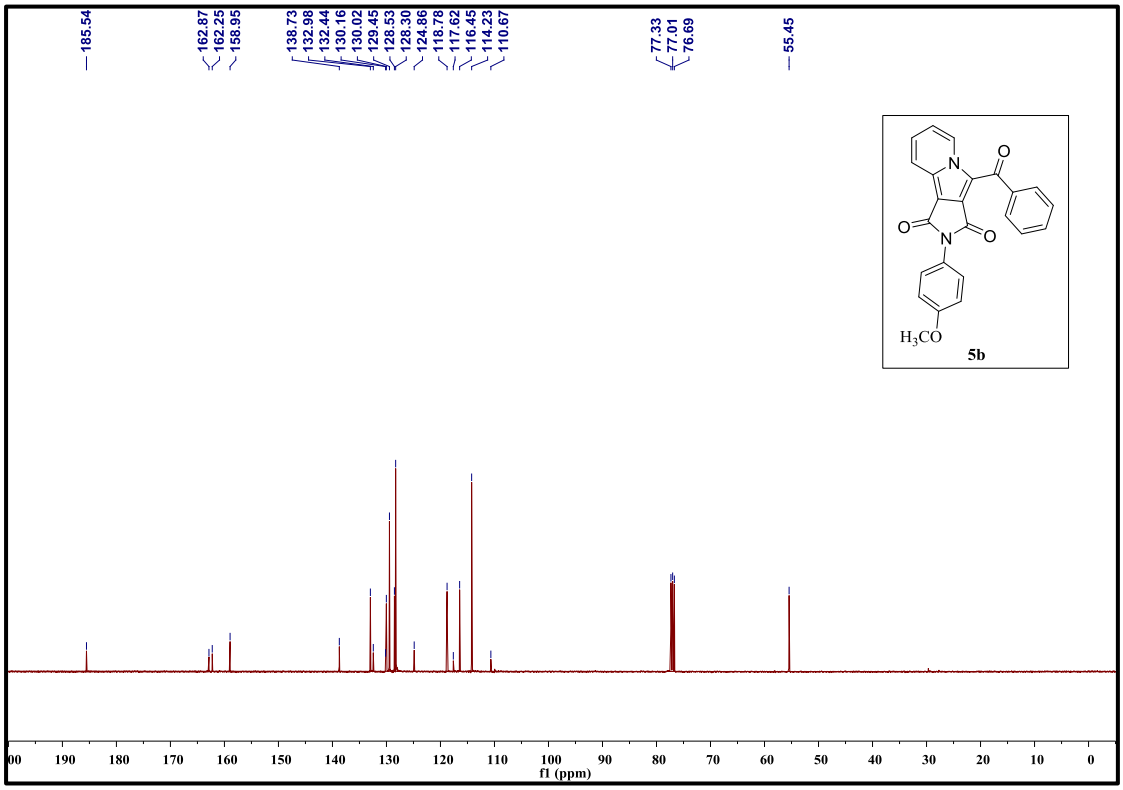
Compound 5a


9.63
9.62
8.00
7.98
7.86
7.84
7.84
7.61
7.59
7.57
7.50
7.48
7.46
7.45
7.24
7.22
7.20
7.19
7.17
7.14
7.13
7.12
7.12
7.10
7.10
2.34
1.00
2.00
1.01
3.08
4.04
1.02
f1 (ppm)
5a
3.07

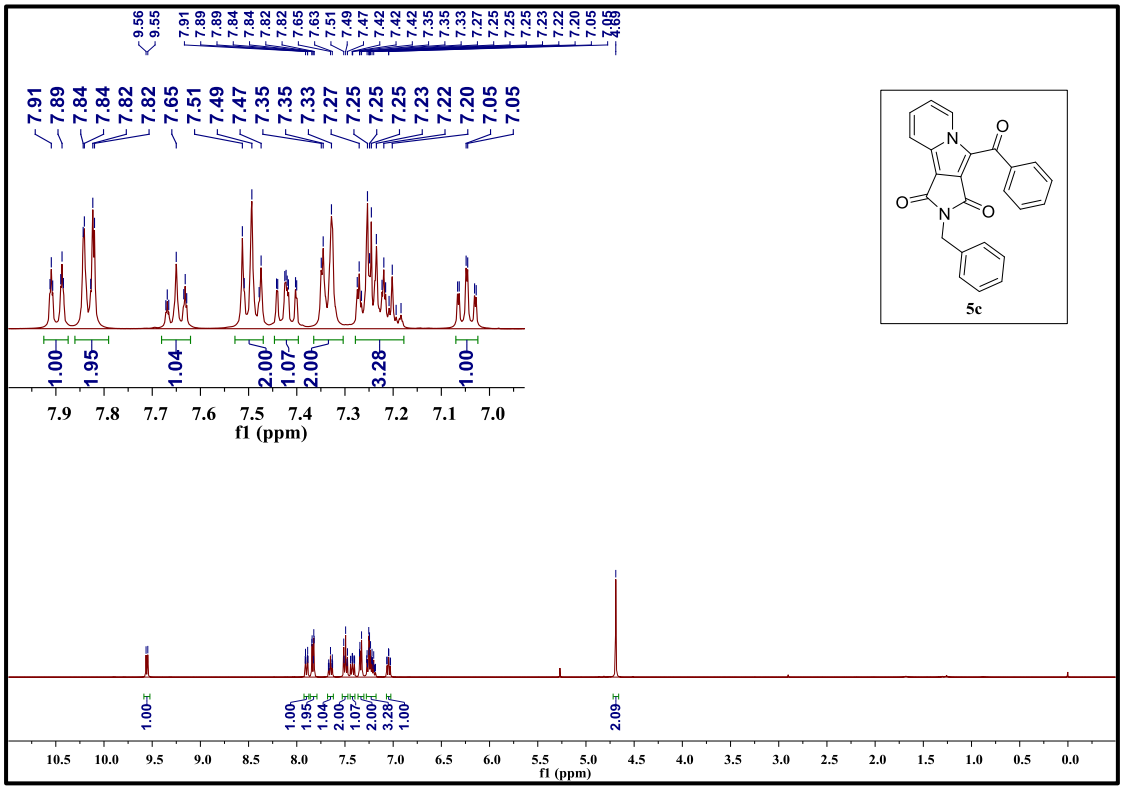

185.53
162.72
162.09
138.71
137.68
132.98
132.44
130.17
130.01
129.51
129.46
128.53
128.31
126.81
119.70
118.78
117.60
116.47
110.71
77.35
77.03
76.71
21.13
f1 (ppm)
5a

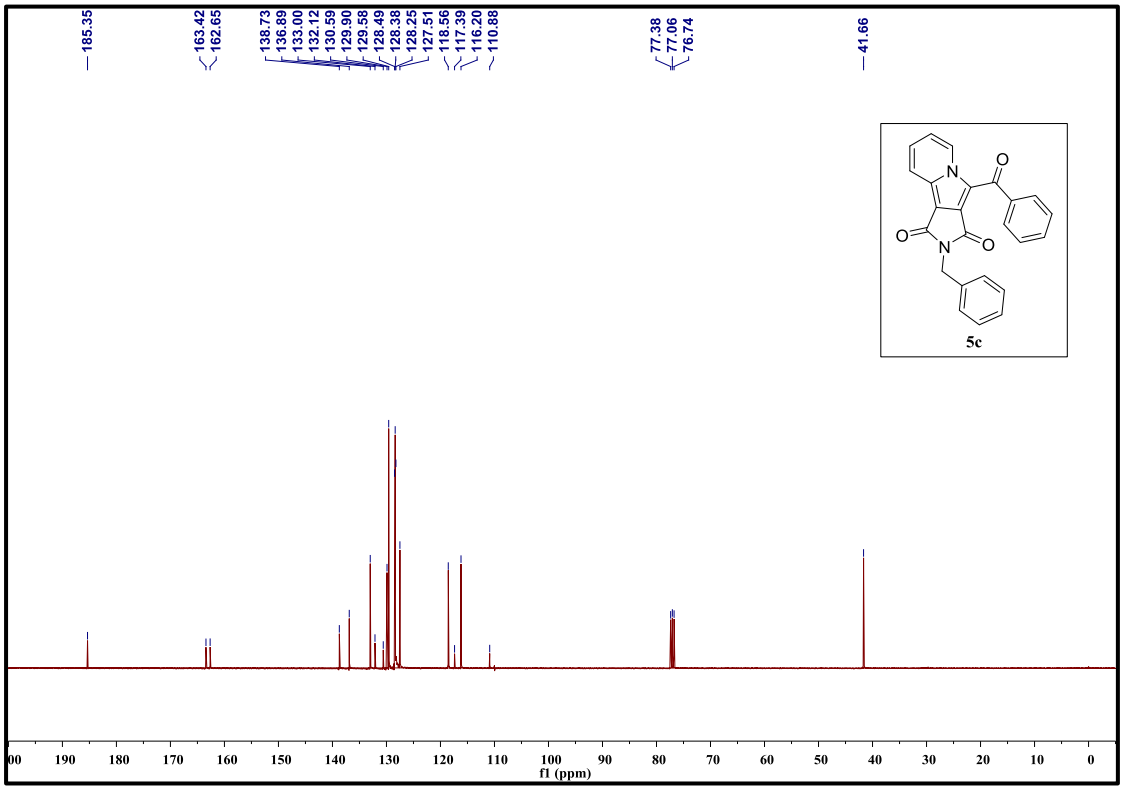
Compound 5b


9.63
9.62
8.00
7.98
7.86
7.84
7.84
7.61
7.59
7.58
7.50
7.50
7.49
7.48
7.47
7.45
7.24
7.22
7.22
7.21
7.20
7.14
7.12
7.12
7.10
6.93
6.90
3.78
1.01
1.85
1.00
2.95
1.85
1.00
2.00
7.9
7.7
7.5
7.3
7.1
6.9
f1 (ppm)
0.99
1.01
1.85
1.00
2.95
1.85
1.00
2.00
3.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
H3CO
5b

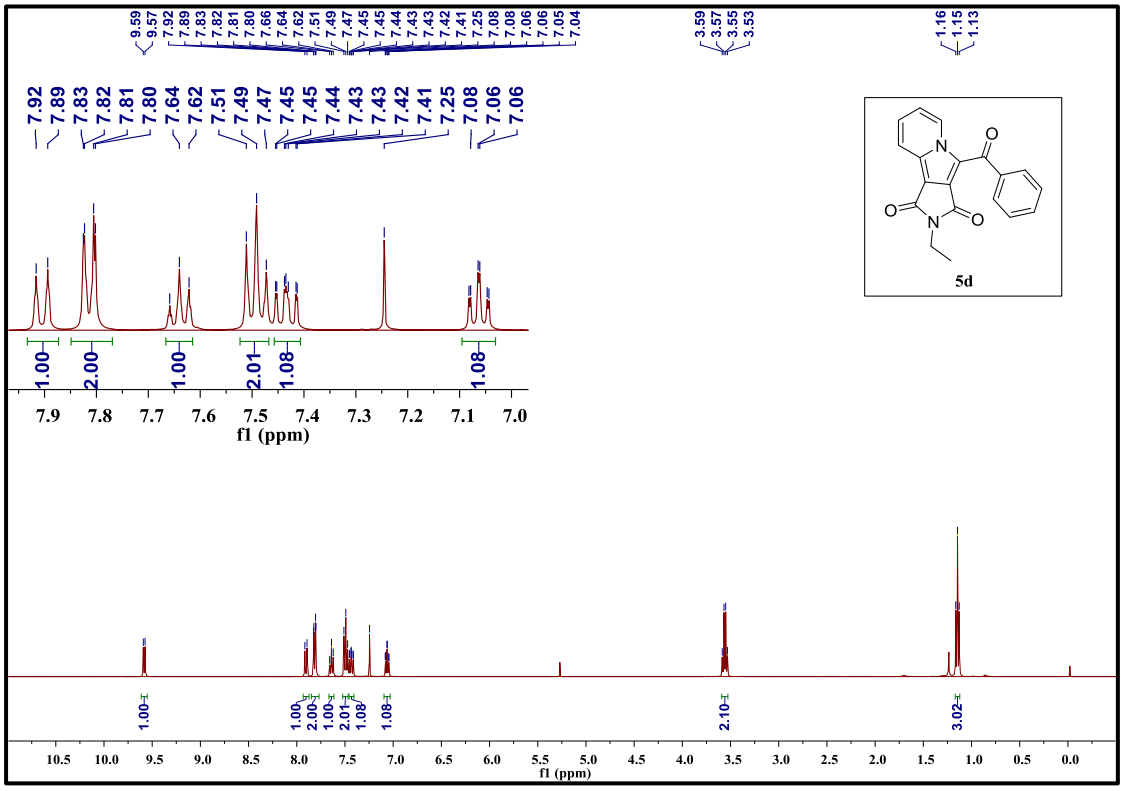

185.54
162.87
162.25
158.95
138.73
132.98
132.44
130.16
130.02
129.45
128.53
128.30
124.86
118.78
117.62
116.45
114.23
110.67
77.33
77.01
76.69
55.45
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
H3CO
5b

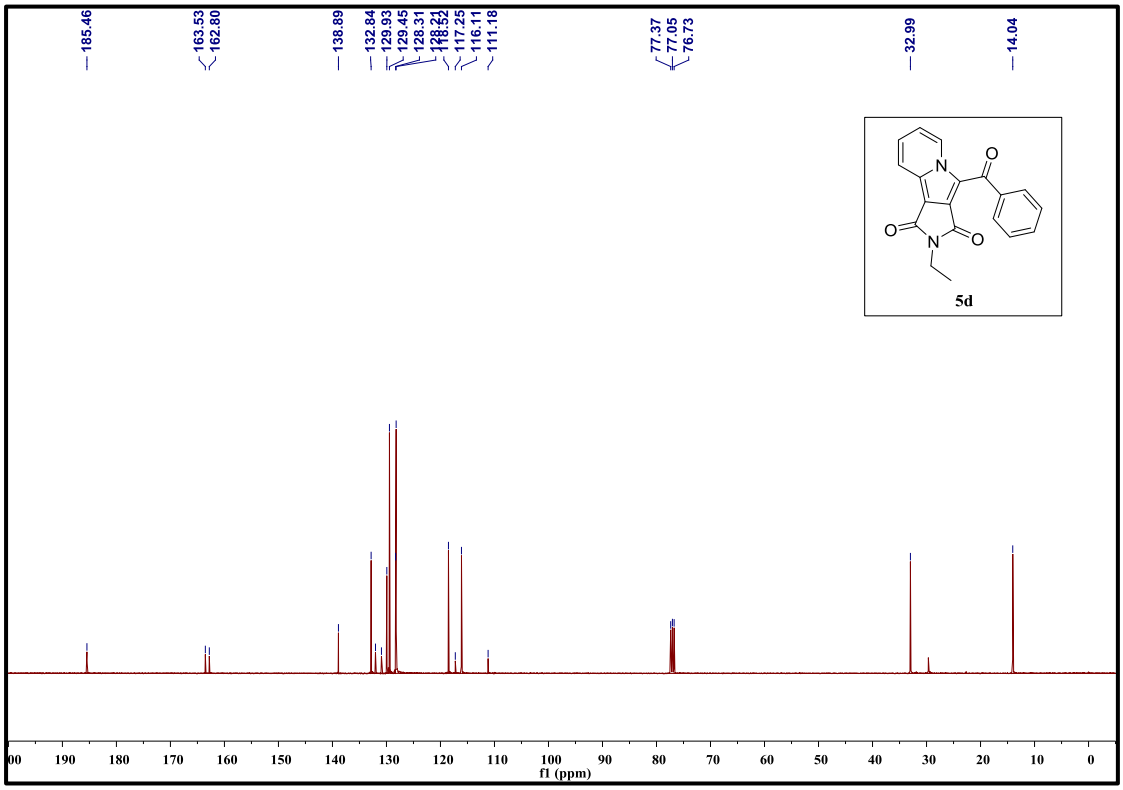
Compound 5c


9.56
9.55
7.91
7.89
7.89
7.84
7.84
7.82
7.82
7.65
7.63
7.51
7.49
7.47
7.42
7.42
7.35
7.35
7.33
7.27
7.25
7.25
7.23
7.22
7.20
7.05
7.05
4.69
1.00
1.95
1.04
2.00
1.07
2.00
3.28
1.00
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0
f1 (ppm)
5c
1.00
1.00
1.95
1.04
2.00
1.07
2.00
3.28
1.00
2.09
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

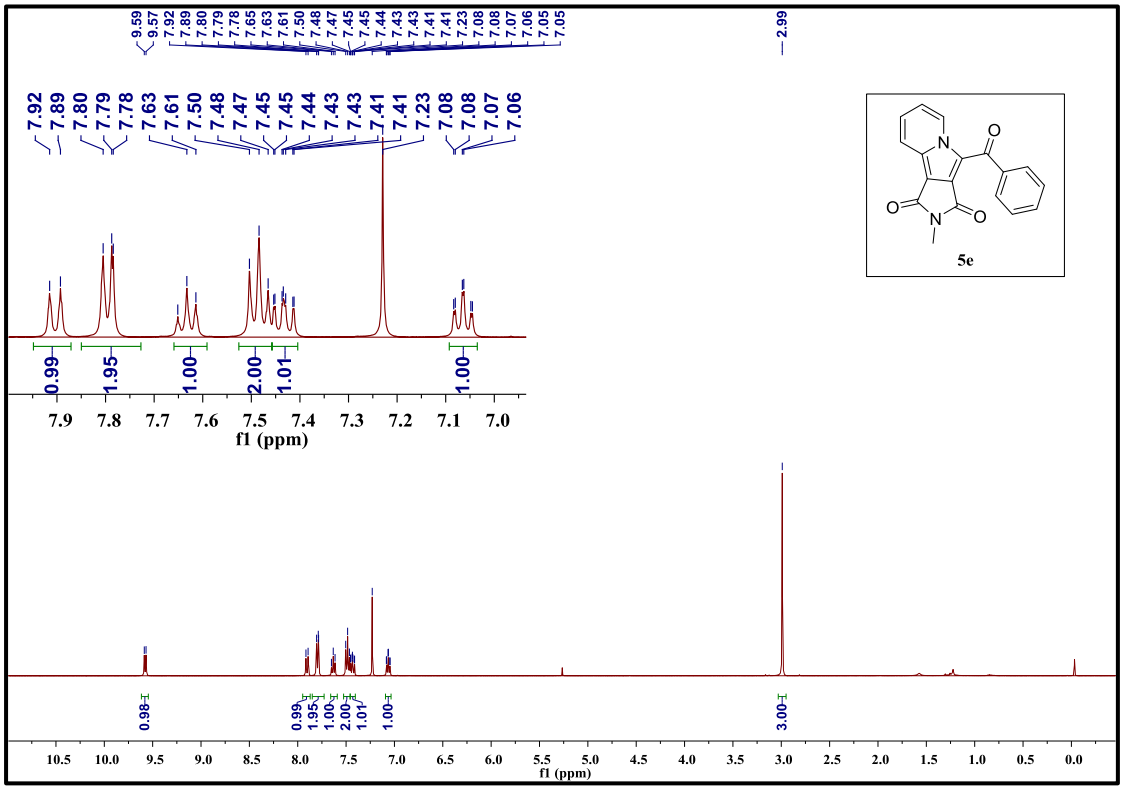

185.35
163.42
162.65
138.73
136.89
133.00
132.12
130.59
129.90
129.58
128.49
128.38
128.25
127.51
118.56
117.39
116.20
110.88
77.38
77.06
76.74
41.66
5c
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

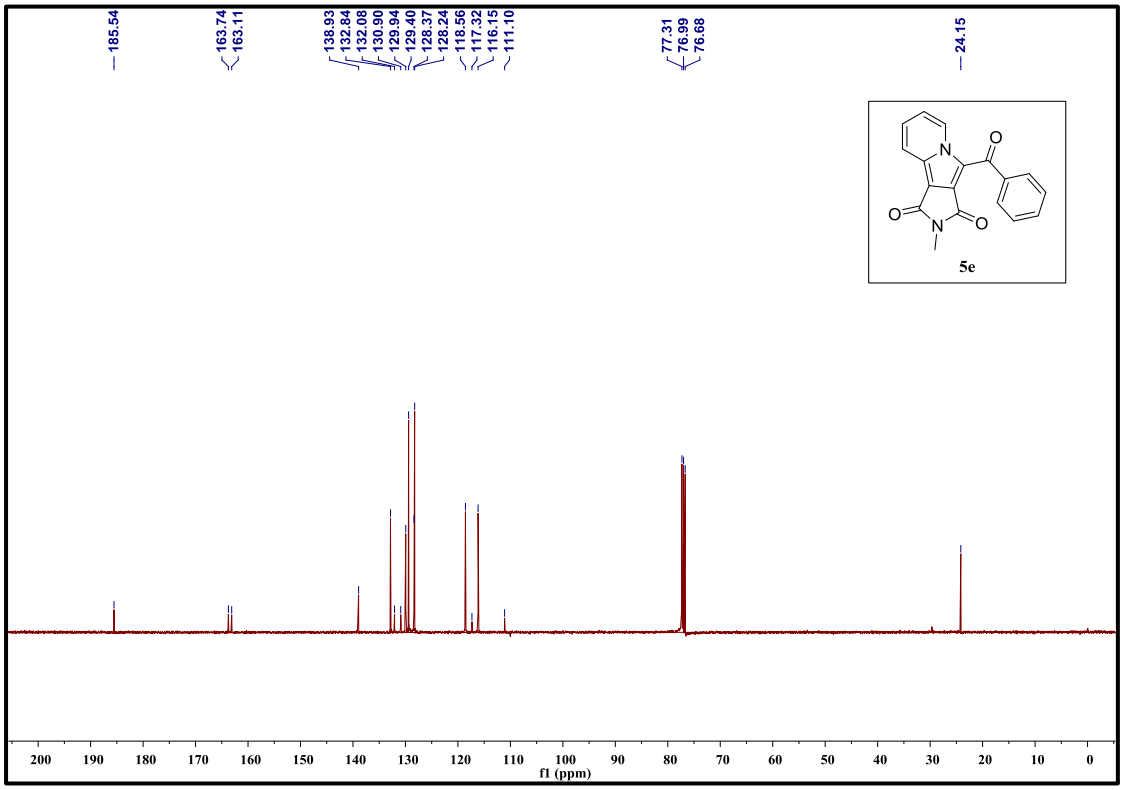
Compound 5d


9.59
9.57
7.92
7.89
7.83
7.82
7.81
7.80
7.66
7.64
7.62
7.51
7.49
7.47
7.45
7.45
7.44
7.43
7.43
7.42
7.41
7.25
7.08
7.06
7.06
7.05
7.04
3.59
3.57
3.55
3.53
1.16
1.15
1.13
1.00
2.00
1.00
2.01
1.08
1.08
1.00
2.00
1.00
2.01
1.08
1.08
2.10
3.02
f1 (ppm)
5d

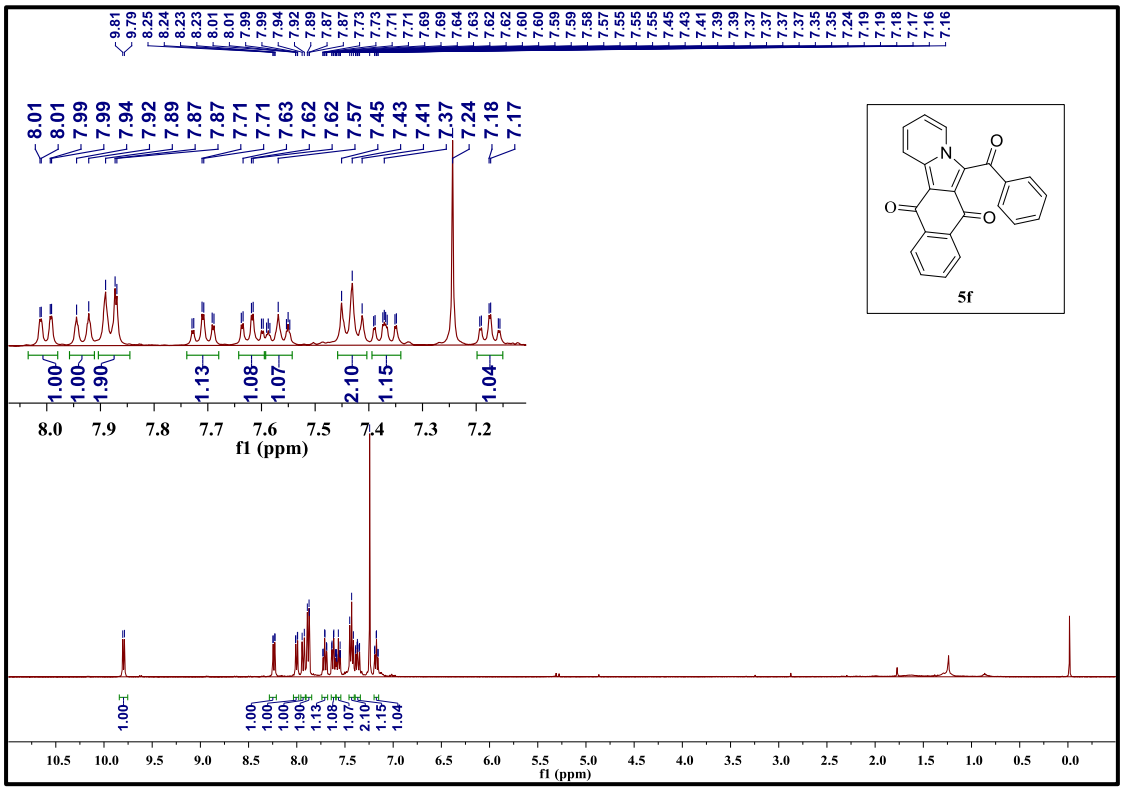

185.46
163.53
162.80
138.89
132.84
129.93
129.45
128.31
128.21
118.52
117.25
116.11
111.18
77.37
77.05
76.73
32.99
14.04
f1 (ppm)
5d

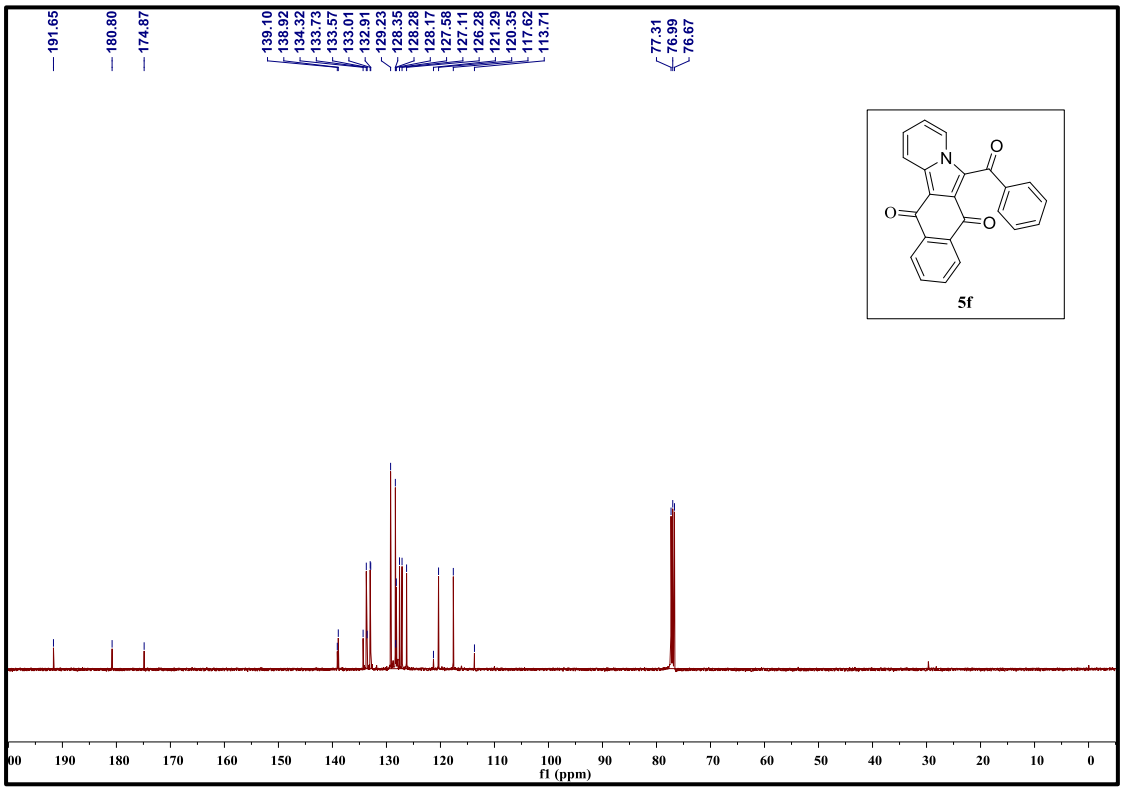
Compound 5e


9.59
9.57
7.92
7.89
7.80
7.79
7.78
7.65
7.63
7.61
7.50
7.48
7.47
7.45
7.45
7.44
7.43
7.43
7.41
7.41
7.23
7.08
7.08
7.07
7.06
7.05
7.05
2.99
0.99
1.95
1.00
2.00
1.01
1.00
0.98
0.99
1.95
1.00
2.00
1.01
1.00
3.00
f1 (ppm)
5e

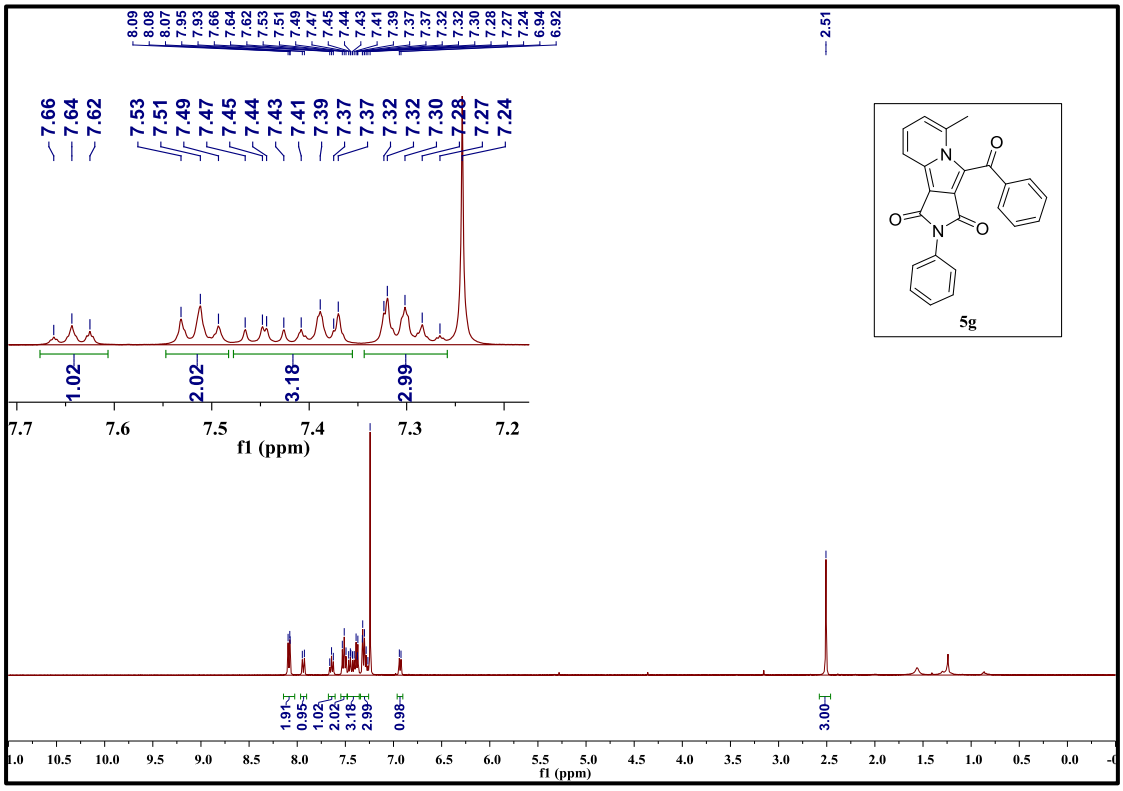

185.54
163.74
163.11
138.93
132.84
132.08
130.90
129.94
129.40
128.37
128.24
118.56
117.32
116.15
111.10
77.31
76.99
76.68
24.15
f1 (ppm)
5e

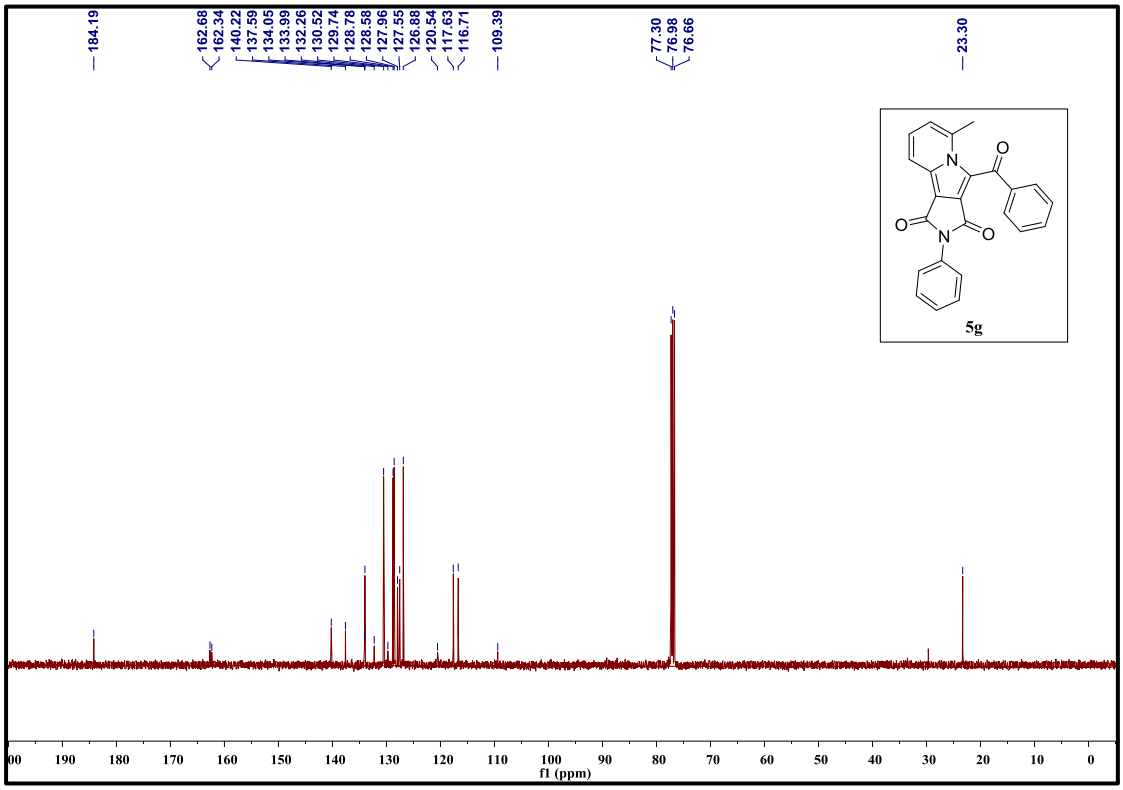
Compound 5f



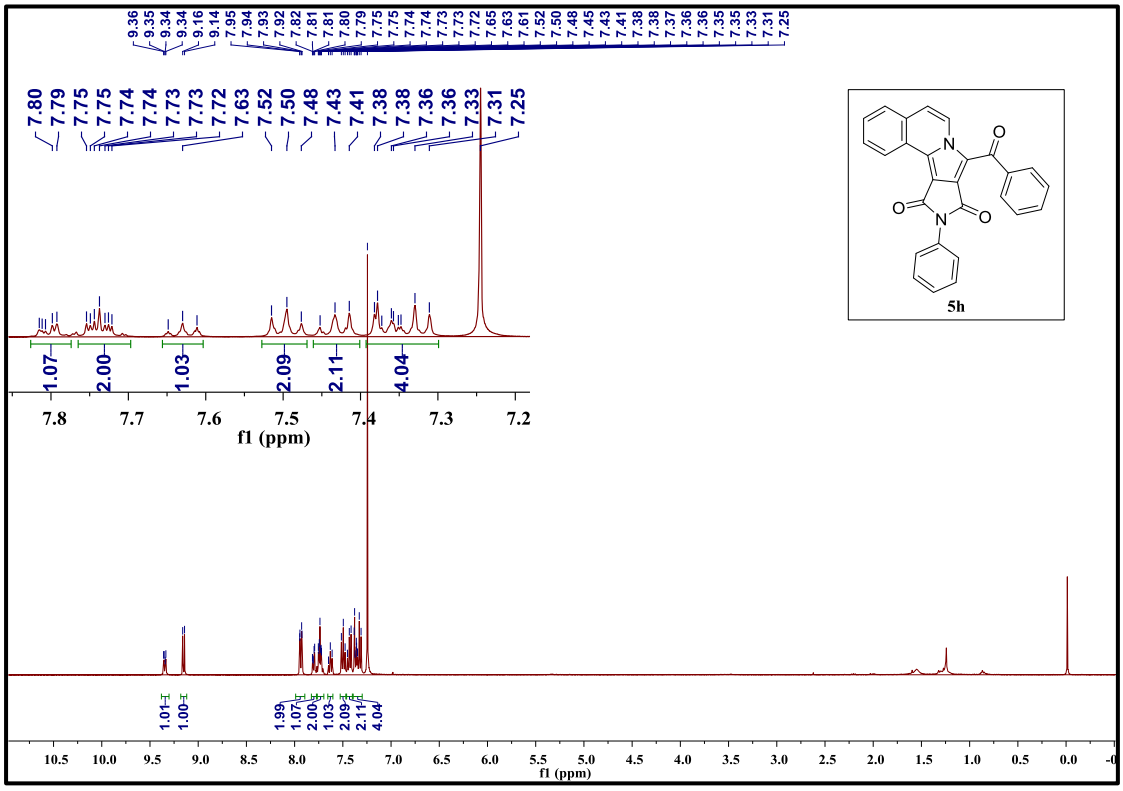


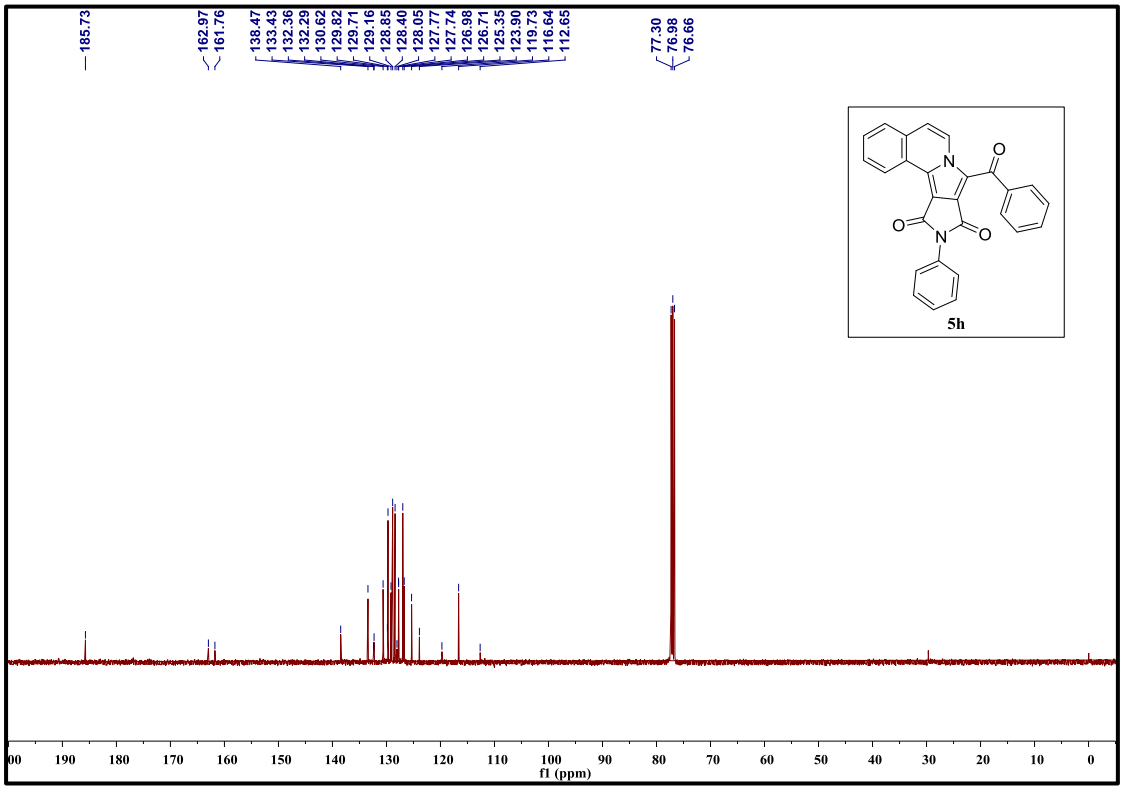
Compound 5g


8.09
8.08
8.07
7.95
7.93
7.66
7.64
7.62
7.53
7.51
7.49
7.47
7.45
7.44
7.43
7.41
7.39
7.37
7.37
7.32
7.30
7.28
7.27
7.24
6.94
6.92
2.51
1.02
2.02
3.18
2.99
f1 (ppm)
1.91
0.95
1.02
2.02
3.18
2.99
0.98
3.00
5g

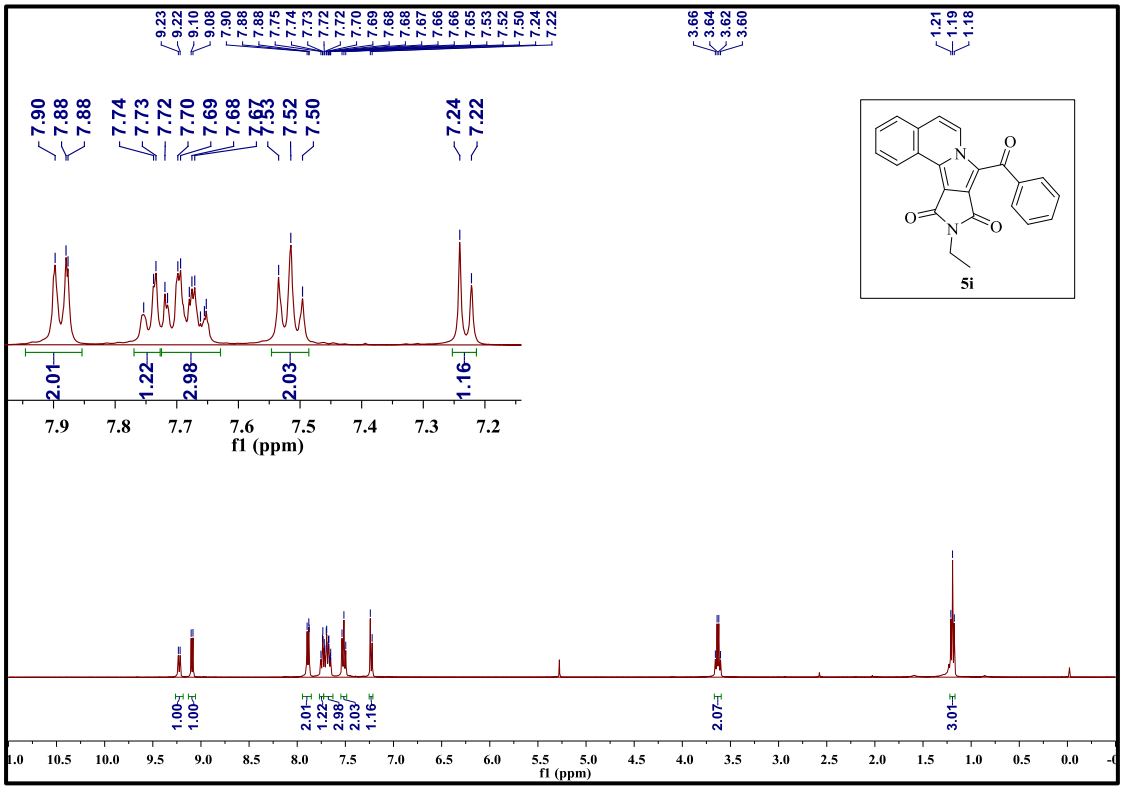

184.19
162.68
162.34
140.22
137.59
134.05
133.99
132.26
130.52
129.74
128.78
128.58
127.96
127.55
126.88
120.54
117.63
116.71
109.39
77.30
76.98
76.66
23.30
f1 (ppm)
5g

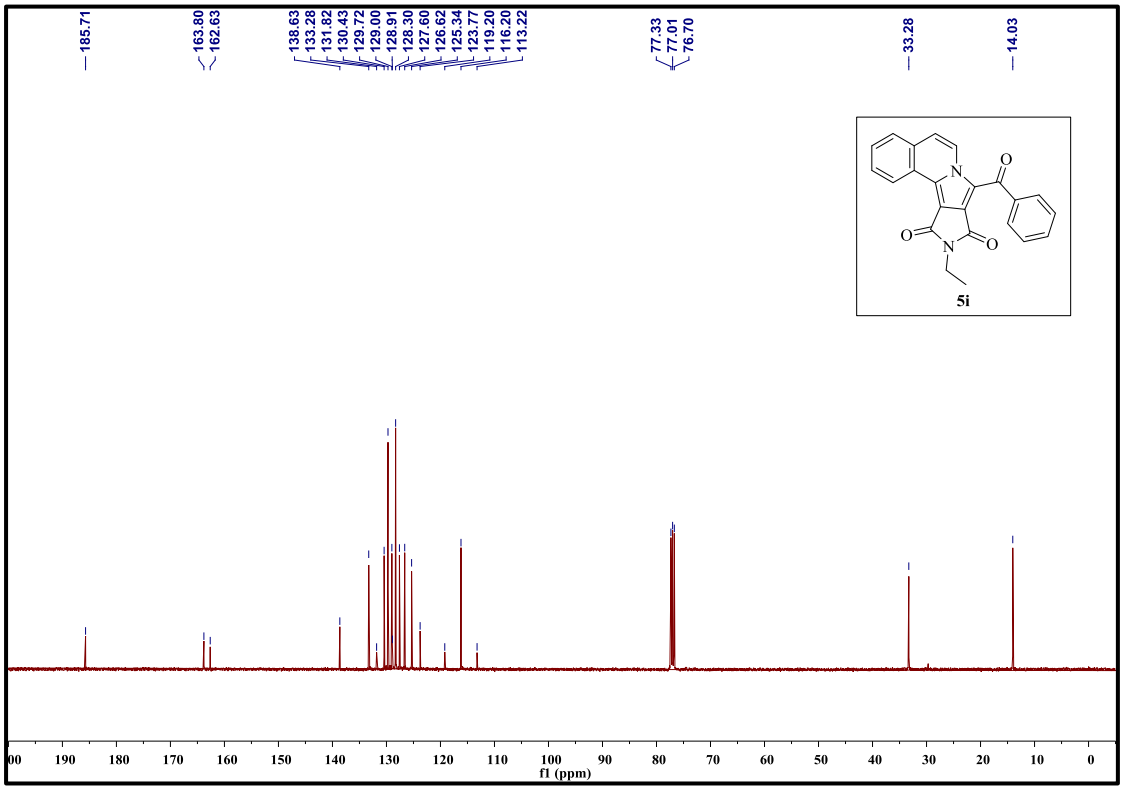
Compound 5h



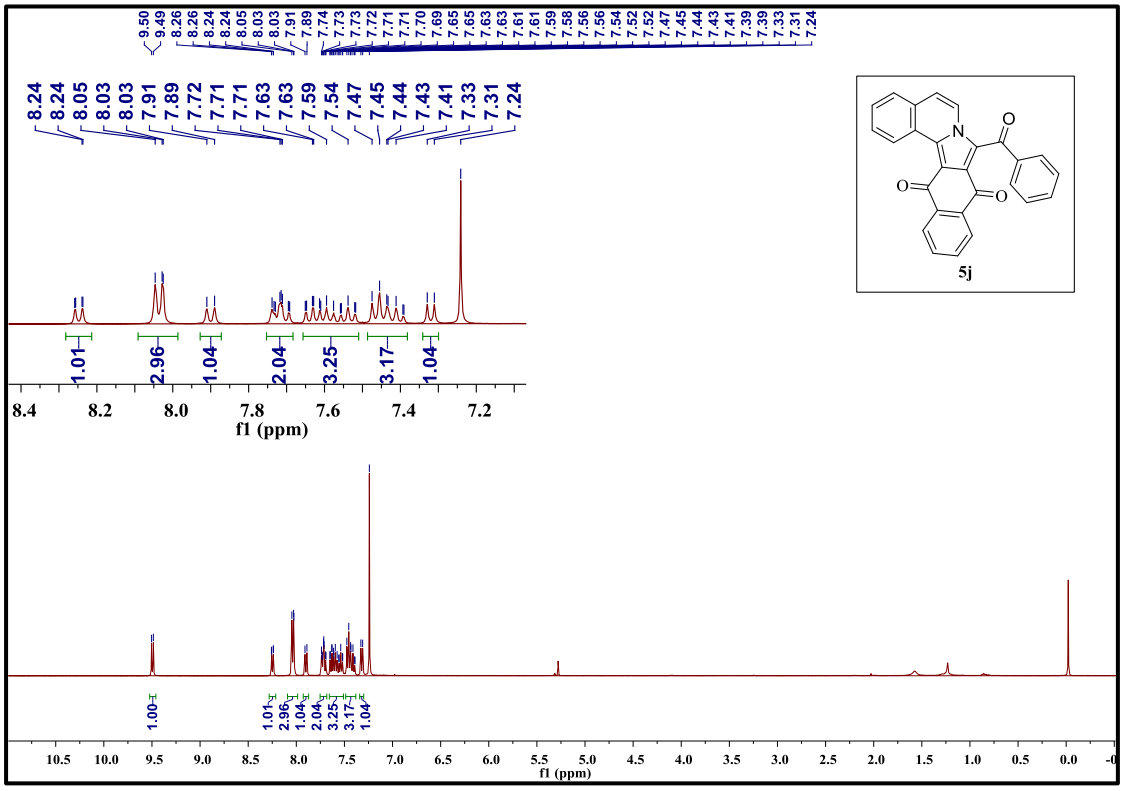


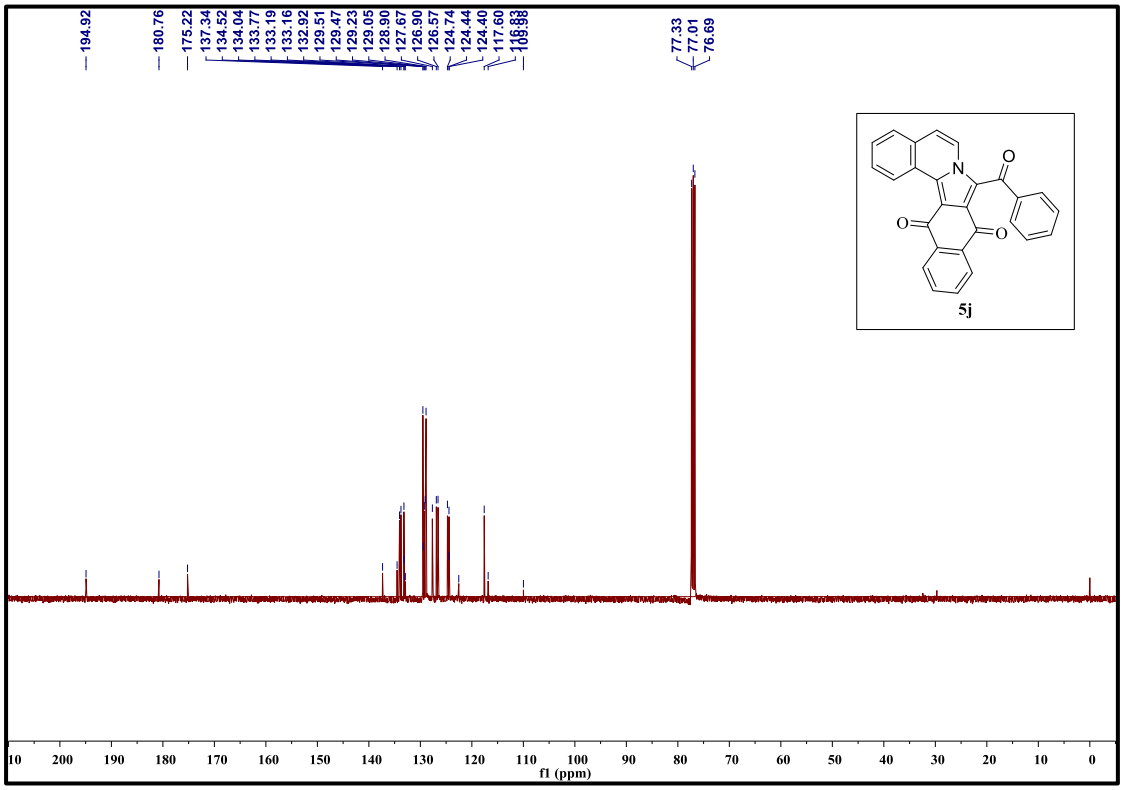
Compound 5i


9.23
9.22
9.10
9.08
7.90
7.88
7.88
7.75
7.74
7.73
7.72
7.72
7.70
7.69
7.68
7.68
7.67
7.66
7.66
7.65
7.53
7.52
7.50
7.24
7.22
3.66
3.64
3.62
3.60
1.21
1.19
1.18
7.90
7.88
7.88
7.74
7.73
7.72
7.70
7.69
7.68
7.67
7.53
7.52
7.50
7.24
7.22
2.01
1.22
2.98
2.03
1.16
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
f1 (ppm)
5i
1.00
1.00
2.01
1.22
2.98
2.03
1.16
2.07
3.01
f1 (ppm)

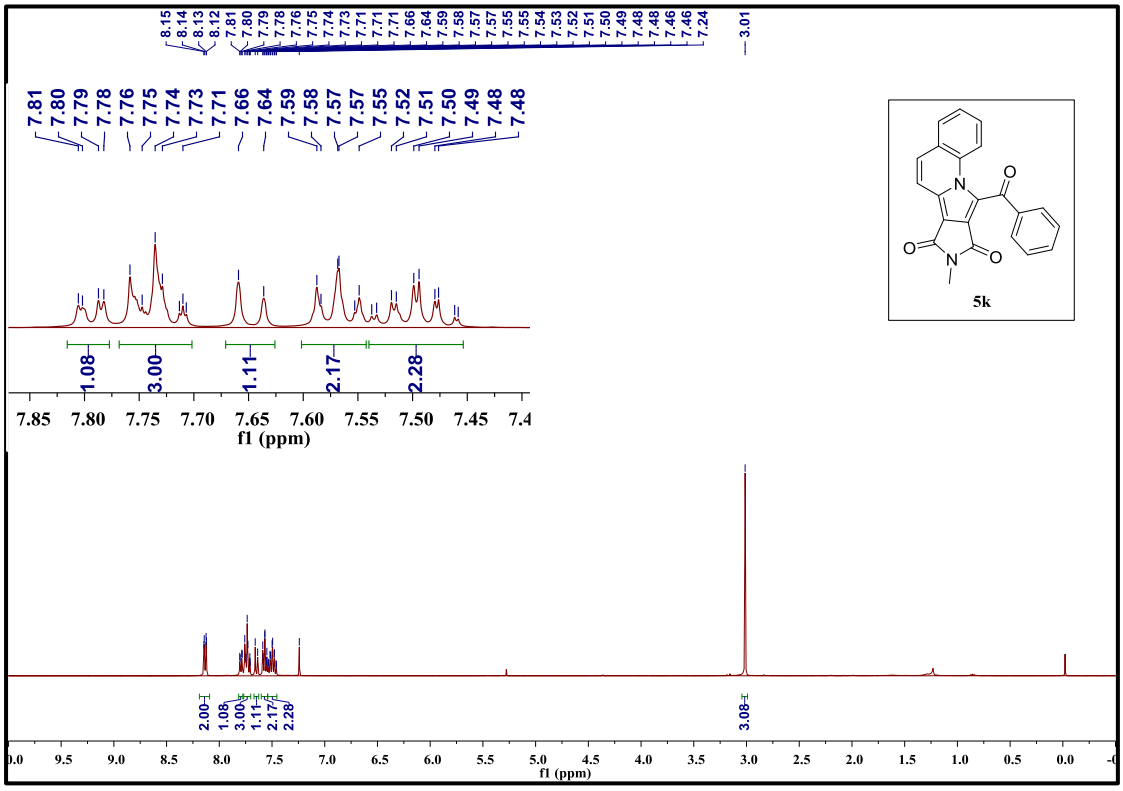

185.71
163.80
162.63
138.63
133.28
131.82
130.43
129.72
129.00
128.91
128.30
127.60
126.62
125.34
123.77
119.20
116.20
113.22
77.33
77.01
76.70
33.28
14.03
5i
f1 (ppm)

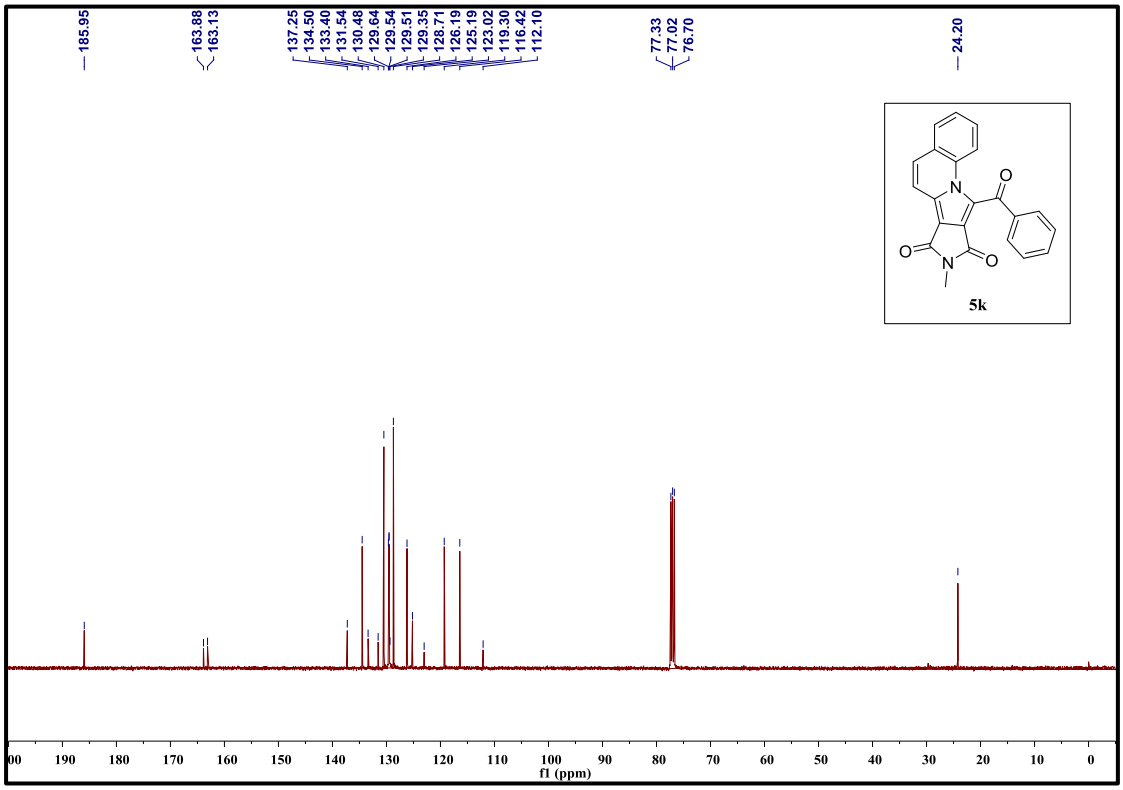
Compound 5j


9.50
9.49
8.26
8.24
8.24
8.05
8.03
8.03
7.91
7.89
7.74
7.73
7.72
7.71
7.71
7.70
7.69
7.65
7.65
7.63
7.63
7.61
7.61
7.59
7.58
7.56
7.56
7.54
7.52
7.52
7.47
7.45
7.44
7.43
7.41
7.39
7.39
7.33
7.31
7.24
8.24
8.24
8.05
8.03
8.03
7.91
7.89
7.72
7.71
7.71
7.63
7.63
7.59
7.54
7.47
7.45
7.44
7.43
7.41
7.33
7.31
7.24
1.01
2.96
1.04
2.04
3.25
3.17
1.04
8.4
8.2
8.0
7.8
7.6
7.4
7.2
f1 (ppm)
O
N
O
O
O
5j
1.00
1.01
2.96
1.04
2.04
3.25
3.17
1.04
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

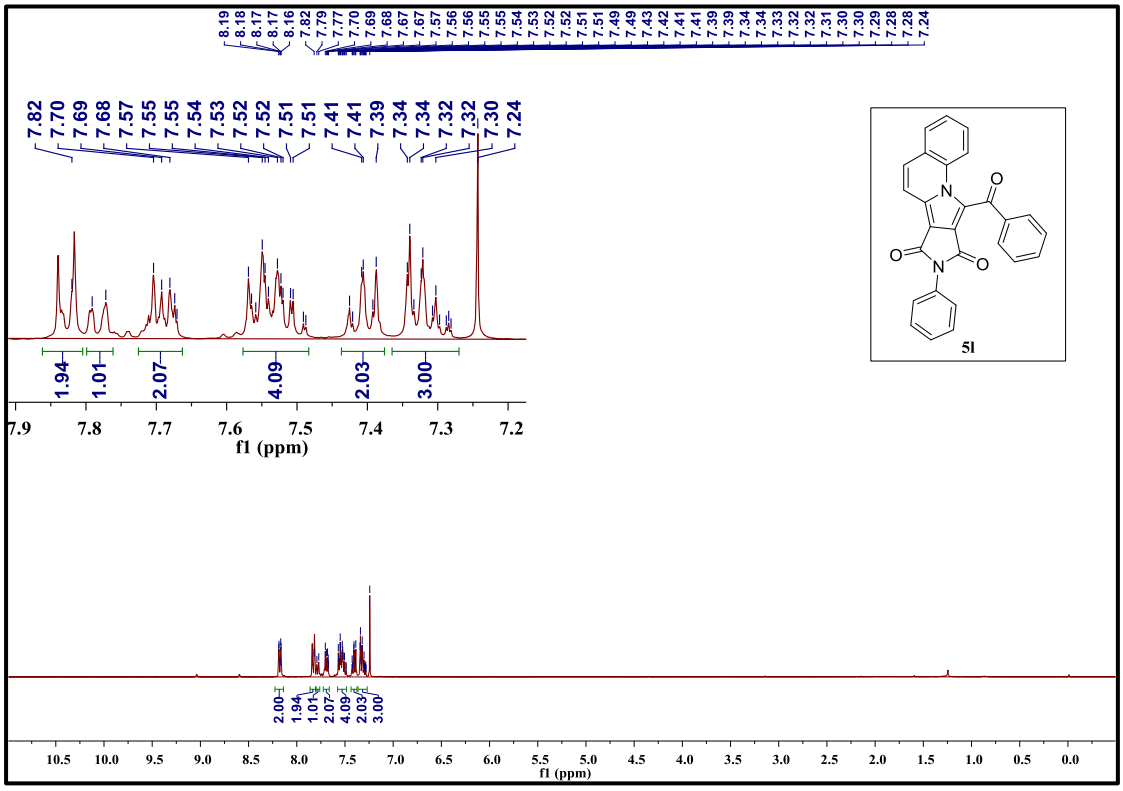

194.92
180.76
175.22
137.34
134.52
134.04
133.77
133.19
133.16
132.92
129.51
129.47
129.23
129.05
128.90
127.67
126.90
126.57
124.74
124.44
124.40
117.60
77.33
77.01
76.69
O
N
O
O
O
5j
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

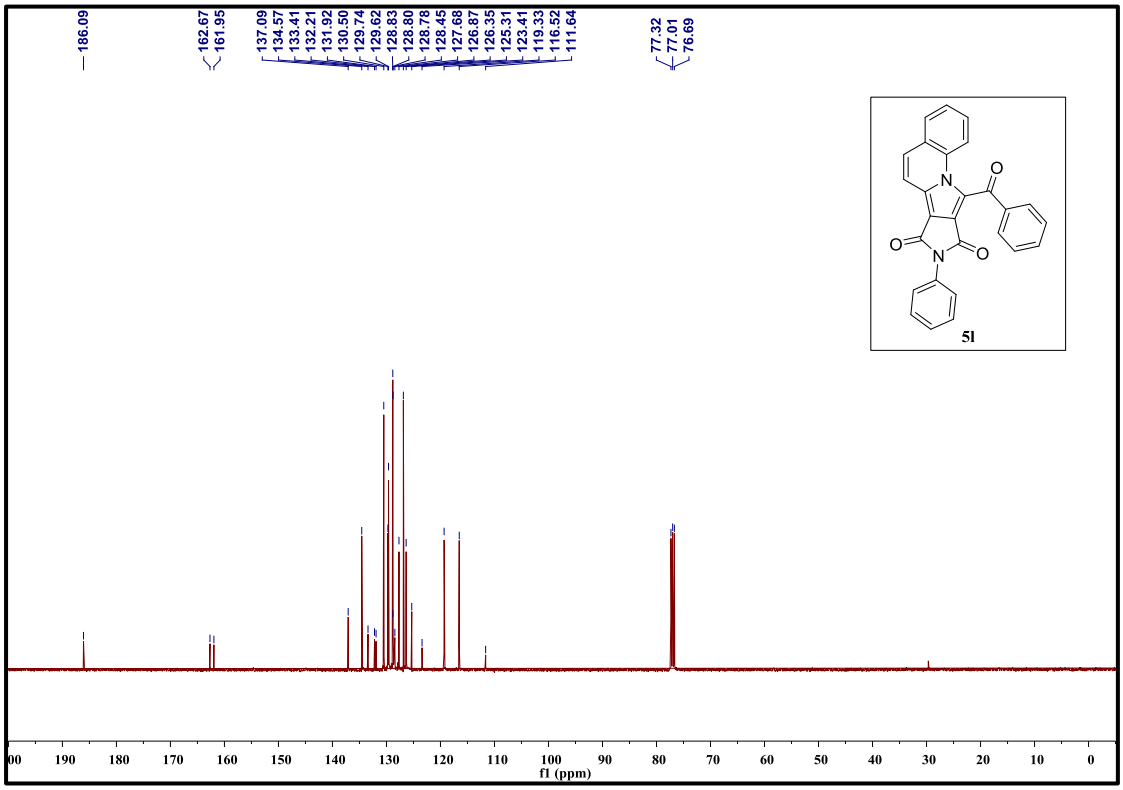
Compound 5k


8.15 8.14 8.13 8.12 7.81 7.80 7.79 7.78 7.76 7.75 7.74 7.73 7.71 7.71 7.66 7.64 7.59 7.58 7.57 7.57 7.55 7.52 7.51 7.50 7.49 7.48 7.48 7.46 7.46 7.24 3.01
1.08 3.00 1.11 2.17 2.28
f1 (ppm)
5k
2.00 1.08 3.00 1.11 2.17 2.28 3.08
f1 (ppm)


185.95 163.88 163.13 137.25 134.50 133.40 131.54 130.48 129.64 129.54 129.51 129.35 128.71 126.19 125.19 123.02 119.30 116.42 112.10 77.33 77.02 76.70 24.20
5k
f1 (ppm)

Compound 5l


8.19 8.18 8.17 8.16 7.82 7.79 7.77 7.70 7.69 7.68 7.67 7.67 7.57 7.56 7.55 7.55 7.54 7.53 7.52 7.52 7.51 7.51 7.49 7.49 7.43 7.42 7.41 7.41 7.39 7.39 7.34 7.34 7.33 7.32 7.31 7.30 7.30 7.29 7.28 7.28 7.24
7.82 7.70 7.69 7.68 7.57 7.55 7.55 7.54 7.53 7.52 7.52 7.51 7.51 7.41 7.41 7.39 7.34 7.34 7.32 7.32 7.30 7.24
1.94 1.01 2.07 4.09 2.03 3.00
7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2
f1 (ppm)
2.00 1.94 1.01 2.07 4.09 2.03 3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
5l


186.09
162.67 161.95
137.09 134.57 133.41 132.21 131.92 130.50 129.74 129.62 128.83 128.80 128.78 128.45 127.68 126.87 126.35 125.31 123.41 119.33 116.52 111.64
77.32 77.01 76.69
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
5l