Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Microwave-assisted palladium mediated efficient synthesis of pyrazolo[3,4-*b*]pyridines, pyrazolo[3,4-*b*]quinolines, pyrazolo[1,5-*a*]pyrimidines and pyrazolo[1,5-*a*]quinazolines

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Table of Contents

General Information

Melting points were measured with a Buchi B-540 melting point apparatus and are uncorrected. IR spectra were recorded on Elmer FT-IR-2000 spectrometer on a thin film using chloroform. NMR spectra were recorded on Avance DPX 300 MHz FT-NMR spectrometer or Bruker Avance III 500 MHz FT-NMR spectrometer using tetramethylsilane (TMS) as an internal standard. Mass spectra were recorded on Trace DSQ GCMS instrument. All the commercially available regents were used as received. All experiments were monitored by thin layer chromatography (TLC). TLC was performed on pre-coated silica gel plates (Merck). Column chromatography was performed on silica gel (100-200 mesh, Merck). All MW reactions were carried out in a Synthos 3000 (Anton Paar) microwave reactor.

Microwave Instrumentation

All microwave reactions were carried out in a Synthos 3000 (Anton Paar) microwave reactor. The multitude microwave has a twin magnetron (2.45 GHz) with maximum output power of 1400 W. The output power can be controlled in unpulsed control mode over whole power which is adjustable in 1 W increment. A Motorola 68xxx series microprocessor system control is used to measure power, pressure, time and temperature during the reaction. The temperature and pressure were monitored throughout the reaction by an infrared detector. The temperature can be measured from 0 to 280 ^{o}C with uncertainty ± 1%. The temperature during the MW reaction was monitored by an externally calibrated IR sensor. The pressure can be measured from 0 to 86 bar with uncertainty ± 0.2 bar. The MW power is initially set at 700 W and the reaction is run. However, during the course of the reaction, once the set temperature and pressure limit is reached, the reactor automatically adjusts the power by lowering it.

General procedure for the synthesis of compounds 3a-p: A mixture of halo aldehyde (**1**, 1.0 mmol), 5-aminopyrrazole (**2**, 1.2 mmol), $Pd(OAc)_2$ (2.5 mol%), PPh_3 (5 mol%) and K_2CO_3 (2.1 mmol) was irradiated in a closed vessel in a Synthos 3000 microwave reactor at 700 Watt (120 ^{o}C and 14 bar) for 15 minutes. After completion of the reaction, the reaction mixture was treated with water (40 mL) and then extracted with ethylacetate (30 x 3 mL). The organic portion was washed with water, dried over anhydrous sodium sulfate and the solvent was removed in vacuo to obtain a crude product which on silica gel column chromatographic purification using EtOAc/hexane as the eluent afforded compounds **3a-p**.

3-Methoxy-8-methyl-10-phenyl-6,10-dihydro-5H-benzo[h]pyrazolo[3,4-b]quinoline ***(3a)****:* Yellow solid, mp: 162-163 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.43 (d, *J* = 7.6 Hz, 2H), 8.38

(d, *J* = 8.7 Hz, 1H), 7.70 (s, 1H), 7.52 (t, *J* = 7.4 Hz, 2H), 7.23 (t, *J* = 5.1 Hz, 1H), 6.93 (dd, *J* = 8.6 Hz & 2.6 Hz, 1H), 6.75 (d, *J* = 2.5 Hz, 1H), 3.86 (s, 3H), 3.02-2.98 (m, 2H), 2.95-2.89 (m, 2H), 2.59 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.8, 152.5, 150.5, 142.2, 141.1, 140.1, 128.9, 127.8, 127.7, 127.6, 125.1, 124.8, 120.2, 120.1, 115.5, 112.9, 112.8, 55.3, 29.0, 28.8, 12.5. IR ($CHCl_3$, cm^{-1}): 2932, 1599, 1505, 1381, 1242, 754. MS (EI, *m/z*): 341.2 [M^+]. Anal. calcd. for $C_{22}H_{19}N_3O$: C, 77.40; H, 5.61; N, 12.31. Found: C, 77.27; H, 5.76; N, 12.40.

*10-Ethyl-3-methoxy-6,10-dihydro-5H-benzo[h]pyrazolo[3,4-b]quinoline (**3b**):* Yellow solid, mp: 59-60 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.43 (d, *J* = 8.6 Hz, 1H), 7.88 (s, 1H), 7.75 (s, 1H), 6.93 (dd, *J* = 8.6 Hz & 2.6 Hz, 1H), 6.77 (d, *J* = 2.5 Hz, 1H), 4.62 (q, *J* = 7.3 Hz, 2H), 3.86 (s, 3H), 3.04-2.97 (m, 4H), 2.94-2.89 (m, 2H), 1.57 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.8, 152.1, 149.7, 141.3, 131.1, 127.9, 127.8, 127.5, 125.1, 114.1, 112.9, 112.8, 55.3, 41.9, 29.1, 28.9, 15.0. IR ($CHCl_3$, cm^{-1}): 2933, 1601, 1416, 1266, 780. MS (EI, *m/z*): 279.1 [M^+]. Anal. calcd. for $C_{17}H_{17}N_3O$: C, 73.10; H, 6.13; N, 15.04. Found: C, 73.12; H, 6.13; N, 15.08.

*3-Methoxy-8,10-diphenyl-6,10-dihydro-5H-benzo[h]pyrazolo[3,4-b]quinoline (**3c**):* Yellow solid, mp: 192-193 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.54 (d, *J* = 7.8 Hz, 2H), 8.42 (d, *J* = 8.6 Hz, 1H), 8.11 (s, 1H), 8.05 (d, *J* = 7.2 Hz, 1H), 7.59-7.23 (m, 7H), 6.96 (dd, *J* = 8.6 Hz & 2.5 Hz, 1H), 6.78 (d, *J* = 2.3 Hz, 1H), 3.87 (s, 3H), 3.11-3.05 (m, 2H), 2.99-2.81 (m, 2H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.9, 152.6, 151.1, 143.7, 141.3, 140.1, 133.2, 128.9, 128.8, 128.6, 128.4, 127.8, 127.6, 127.2, 126.3, 125.4, 120.8, 113.8, 113.1, 112.9, 55.4, 28.97, 28.9. IR ($CHCl_3$, cm^{-1}): 1598, 1497, 1380, 1245, 755. MS (EI, *m/z*): 403.1 [M^+]. Anal. calcd. for $C_{27}H_{21}N_3O$: C, 80.37; H, 5.25; N, 10.41. Found: C, 80.45; H, 5.29; N, 10.72.

*10-Ethyl-6,10-dihydro-5H-benzo[h]pyrazolo[3,4-b]quinoline (**3d**):* Red solid, mp: 68 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.35 (s, 1H), 8.29 (s, 1H), 8.01 (d, *J* = 7.6 Hz, 1H), 7.40-7.17 (m, 3H), 4.54 (q, *J* = 7.2 Hz, 2H), 2.92-2.79 (m, 4H), 1.48 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 150.4, 148.6, 139.3, 136.3, 132.4, 130.9, 129.6, 128.5, 127.3, 127.2, 125.0, 111.6, 53.5, 28.8, 25.4, 15.0. IR ($CHCl_3$, cm^{-1}): 2930, 1561, 1260, 1023, 772. MS (EI, *m/z*): 249.1 [M^+]. Anal. calcd. for $C_{16}H_{15}N_3$: C, 77.08; H, 6.06; N, 16.85. Found: C, 77.19; H, 5.85; N, 16.70.

*8-Methyl-10-phenyl-6,10-dihydro-5H-benzo[h]pyrazolo[3,4-b]quinoline (**3e**):* Yellow solid, mp: 152 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.44 (dd, *J* = 7.9 Hz & 1.2 Hz, 2H), 7.73 (s, 1H), 7.54-7.21 (m, 7H), 3.05-2.91 (m, 4H), 2.59 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 152.4, 150.5, 142.2, 140.1, 139.3, 134.8, 129.6, 128.9, 127.9, 127.2, 126.8, 126.3, 125.9, 124.9, 120.2, 116.1, 28.7, 28.6, 12.6. IR ($CHCl_3$, cm^{-1}): 2925, 1599, 1504, 1411, 1381, 1126, 761.

MS (EI, *m/z*): 311.1 [M^+]. Anal. calcd. for $C_{21}H_{17}N_3$: C, 81.00; H, 5.50; N, 13.49. Found: C, 81.35; H, 5.58; N, 13.71.

*10-Ethylchromeno[4,3-b]pyrazolo[4,3-e]pyridin-6(10H)-one (**3f**):* White solid, mp: 178 ^oC. ^1H NMR ($CDCl_3$, 300 MHz) δ 9.01 (s, 1H), 8.16 (s, 1H), 7.19- 8.63 (m, 4H), 4.63 (q, *J* = 6.0 Hz, 2H), 1.55 (t, *J* = 6.0 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 161.8, 152.7, 151.2, 150.4, 134.9, 134.2, 132.4, 125.1, 124.7, 119.6, 117.3, 116.2, 111.9, 42.4, 29.7. IR ($CHCl_3$, cm^{-1}): 1728, 1612, 1424, 1170, 750. MS (EI, *m/z*): 265.0 [M^+]. Anal. calcd. for $C_{15}H_{11}N_3O_2$: C, 67.92; H, 4.18; N, 15.84. Found: C, 67.82; H, 4.37; N, 15.68.

*1-Ethyl-6-phenyl-1H-pyrazolo[3,4-b]pyridine (**3g**):* Red solid, mp: 62 ^oC. ^1H NMR ($CDCl_3$, 300 MHz) δ 8.06 (d, *J* = 6.9 Hz, 1H), 8.01 (d, *J* = 8.4 Hz, 2H), 7.92 (s, 1H), 7.52 (d, *J* = 8.4 Hz, 1H), 7.48-7.33 (m, 3H), 4.58 (q, *J* = 7.2 Hz, 2H), 1.51 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.3, 150.1, 139.5, 131.6, 130.3, 129.2, 128.8, 128.5, 127.5, 114.4, 114.3, 41.9, 15.0. IR ($CHCl_3$, cm^{-1}): 2930, 1597, 1455, 1440, 927, 758. MS (EI, *m/z*): 223.1 [M^+]. Anal. calcd. for $C_{14}H_{13}N_3$: C, 75.31; H, 5.87; N, 18.82. Found: C, 75.32; H, 5.81; N, 18.94.

*3-Methyl-1,6-diphenyl-1H-pyrazolo[3,4-b]pyridine (**3h**):* Red solid, mp: 131 ^oC. ^1H NMR ($CDCl_3$, 300 MHz) δ 8.35 (d, *J* = 8.5 Hz, 2H), 8.14-8.08 (m, 3H), 8.01 (d, *J* = 8.3 Hz, 1H), 7.61-6.91 (m, 6H), 2.63 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 156.8, 151.1, 146.1, 142.6, 139.8, 139.2, 129.9, 128.8, 128.6, 127.6, 126.1, 125.6, 121.1, 120.6, 15.4. IR ($CHCl_3$, cm^{-1}): 1596, 1506, 1261, 799. MS (EI, *m/z*): 285.1 [M^+]. Anal. calcd. for $C_{19}H_{15}N_3$: C, 79.98; H, 5.30; N, 14.73. Found: C, 80.26; H, 5.35; N, 14.54.

*3β-Acetoxy-1′-ethyl-androst[16,17-f]-1H-pyrazolo[3,4-b]pyridine (**3i**):* Yellow solid, mp: 167 ^oC. ^1H NMR ($CDCl_3$, 300 MHz) δ 8.34 (s, 1H), 7.43 (s, 1H), 5.44-5.42 (m, 1H), 4.64-4.53 (m, 1H), 4.29 (q, *J* = 3.9 Hz, 2H), 2.04 (s, 3H), 1.12 (s, 3H), 1.05 (s, 3H), 2.82-0.84 (m, 20H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 170.6, 157.7, 155.6, 139.9, 138.5, 131.4, 122.3, 73.8, 55.4, 50.5, 46.9, 42.6, 38.1, 36.9, 35.1, 31.6, 30.5, 29.7, 27.7, 21.5, 20.7, 19.3, 16.4, 15.4. IR ($CHCl_3$, cm^{-1}): 1731, 1247, 1030. MS (EI, *m/z*): 373.2 [M-60$^+$]. Anal. calcd. for $C_{27}H_{35}N_3O_2$: C, 74.79; H, 8.14; N, 9.69. Found: C, 74.88; H, 8.21; N, 9.54.

*3β-Acetoxy-1′,3′-dimethyl-androst[16,17-f]-1H-pyrazolo[3,4-b]pyridine (**3j**):* Yellow solid, mp: 185 ^oC. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.69 (s, 1H), 5.43-5.45 (m, 1H), 4.57- 4.66 (m, 1H), 4.05 (s, 3H), 2.51 (s, 3H), 2.05 (s, 3H), 1.18 (s, 3H), 1.03 (s, 3H), 2.82-0.83 (m, 17H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 173.9, 170.5, 155.6, 140.1, 139.7, 128.9, 124.5, 122.1, 113.3, 73.8, 56.2, 53.4, 50.5, 45.4, 38.1, 36.9, 33.7, 33.6, 31.9, 31.3, 31.0, 29.7, 29.4, 27.7. IR

($CHCl_3$, cm^{-1}): 1733, 1243, 1032. MS (EI, *m/z*): 373.2 [M-60$^+$]. Anal. calcd. for $C_{27}H_{35}N_3O_2$: C, 74.79; H, 8.14; N, 9.69. Found: C, 74.49; H, 8.11; N, 9.82.

3β-Acetoxy-1′-methyl-3′-tert-butyl-androst[16,17-f]-1H-pyrazolo[3,4-b]pyridine ***(3k):*** Yellow solid, 149 ^oC. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.82 (s, 1H), 5.32-5.35 (m, 1H), 4.48-4.60 (m, 1H), 3.73 (s, 3H), 1.97 (s, 3H), 1.22 (s, 9H), 1.00 (s, 3H), 0.86 (s, 3H), 2.78-0.80 (m, 17H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 185.2, 170.6, 160.2, 146.6, 140.0, 122.0, 114.1, 92.2, 73.8, 52.7, 50.4, 46.7, 38.1, 37.0, 36.8, 34.4, 33.9, 32.1, 31.6, 31.3, 31.1, 27.7, 23.7, 21.4, 20.7, 19.4, 16.2. IR ($CHCl_3$, cm^{-1}): 1733, 1245, 1032. MS (EI, *m/z*): 415.3 [M-60$^+$]. Anal. calcd. for $C_{30}H_{41}N_3O_2$: C, 75.75; H, 8.69; N, 8.83. Found: C, 75.59; H, 8.64; N, 8.78.

3β-Acetoxy-1′-phenyl-3′-methyl-androst[16,17-f]-1H-pyrazolo[3,4-b]pyridine ***(3l):*** Yellow solid, mp: 242 ^oC. ^1H NMR ($CDCl_3$, 300 MHz) δ 8.38 (d, *J* = 7.7 Hz, 2H), 7.75 (s, 1H), 7.48 (t, *J* = 7.9 Hz, 2H), 7.23 (t, *J* = 7.4 Hz, 1H), 5.46-5.43 (m, 1H), 4.69-4.55 (m, 1H), 2.61 (s, 3H), 2.05 (s, 3H), 1.12 (s, 3H), 1.05 (s, 3H), 2.81-0.86 (m, 17H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.2, 170.6, 150.7, 142.2, 140.2, 130.2, 128.8, 124.8, 124.7, 122.1, 120.3, 115.5, 73.8, 56.2, 50.6, 45.5, 38.2, 36.9, 33.7, 31.4, 31.1, 30.1, 27.8, 21.5, 20.6, 19.4, 17.5, 12.4. IR ($CHCl_3$, cm^{-1}): 2931, 1733, 1596, 1508, 1386, 1246, 1028, 773. MS (EI, *m/z*): 435.2 [M-60$^+$]. Anal. calcd. for $C_{32}H_{37}N_3O_2$: C, 77.54; H, 7.52; N, 8.48. Found: C, 77.76; H, 7.68; N, 8.24.

3β-Acetoxy-1′-phenyl-3′-phenyl-androst[16,17-f]-1H-pyrazolo[3,4-b]pyridine ***(3m):*** White solid, mp: 239 ^oC. ^1H NMR ($CDCl_3$, 300 MHz) δ 8.50 (d, *J* = 7.8 Hz, 2H), 8.10 (s, 1H), 8.00 (d, *J* = 7.1 Hz, 2H), 7.55- 7.17 (m, 6H), 5.45-5.42 (m, 1H), 4.68-4.56 (m, 1H), 2.05 (s, 3H), 1.11 (s, 3H), 1.05 (s, 3H), 2.84-0.67 (m, 17H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 174.2, 170.6, 151.2, 143.9, 140.2, 140.1, 133.3, 131.3, 129.1, 128.9, 128.4, 128.2, 127.4, 125.7, 125.4, 122.1, 120.9, 113.9, 73.8, 56.2, 50.5, 45.6, 38.2, 36.9, 33.6, 31.9, 31.3, 31.1, 30.1, 29.7, 27.8, 22.7, 21.4, 20.6, 19.4, 17.5, 14.2. IR ($CHCl_3$, cm^{-1}): 2929, 1733, 1596, 1504, 1244, 1031, 769. MS (EI, *m/z*): 497.3 [M-60$^+$]. Anal. calcd. for $C_{37}H_{39}N_3O_2$: C, 79.68; H, 7.05; N, 7.53. Found: C, 79.73; H, 7.01; N, 7.79.

1-Ethyl-6-fluoro-1H-pyrazolo[3,4-b]quinoline ***(3n):*** White solid, mp: 97-98 ^oC. ^1H NMR ($CDCl_3$, 300 MHz) δ 8.90 (s, 1H), 8.26 (s, 1H), 8.13-8.07 (m, 1H), 7.66-7.61 (m, 1H), 7.56-7.46 (m, 1H), 4.61 (q, *J* = 7.2 Hz, 2H), 1.52 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 151.4, 146.6, 130.2, 127.3, 125.6, 124.8, 121.3, 113.1, 112.7, 109.5, 42.6, 15.3. IR ($CHCl_3$, cm^{-1}): 2925, 1588, 1570, 1450, 1239, 955, 772. MS (EI, *m/z*): 215.1 [M$^+$]. Anal. calcd. for $C_{12}H_{10}FN_3$: C, 66.97; H, 4.68; N, 19.52. Found: C, 67.16; H, 4.71; N, 19.61.

1-Ethyl-7-methyl-1H-pyrazolo[3,4-b]quinoline ***(3o):*** Yellow gum. ^1H NMR ($CDCl_3$, 300 MHz) δ 8.98 (s, 1H), 8.34 (s, 1H), 7.98-7.92 (m, 2H), 7.38 (dd, *J* = 8.3 & 1.1 Hz, 1H), 4.68

(q, *J* = 7.3 Hz, 2H), 2.61 (s, 3H), 1.59 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 152.4, 146.9, 142.4, 130.9, 130.6, 129.1, 127.1, 123.2, 121.9, 42.5, 22.3, 15.3. IR ($CHCl_3$, cm^{-1}): 2924, 1634, 1460, 775. MS (EI, *m/z*): 211 [M^+]. Anal. calcd. for $C_{13}H_{13}N_3$: C, 73.91; H, 6.20; N, 19.89. Found: C, 73.82; H, 6.35; N, 19.70.

*1-Ethyl-1H-[1,3]dioxolo[4,5-g]pyrazolo[3,4-b]quinoline (**3p**):* Yellow gum. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.82 (s, 1H), 8.26 (s, 1H), 7.36 (s, 1H), 7.32 (s, 1H), 6.15 (s, 2H), 4.66 (q, *J* = 7.2 Hz, 2H), 1.57 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 151.8, 150.5, 146.6, 130.1, 128.6, 121.1, 110.5, 105.6, 101.7, 100.1, 42.4, 15.2. IR ($CHCl_3$, cm^{-1}): 2921, 1598, 1457, 1247, 1037, 774. MS (EI, *m/z*): 241 [M^+]. Anal. calcd. for $C_{13}H_{11}N_3O_2$: C, 64.72; H, 4.60; N, 17.42. Found: C, 64.79; H, 4.66; N, 17.53.

*N-((1-Bromo-6-methoxy-3,4-dihydronaphthalen-2-yl)methylene)-3-methyl-1-phenyl-1H-pyrazol-5-amine (**4a**):* Yellow solid, mp: 142 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.96 (s, 1H), 7.73 (d, *J* = 7.7 Hz, 2H), 7.54-7.24 (m, 4H), 7.09 (d, *J* = 8.2 Hz, 1H), 6.83 (dd, *J* = 8.2 Hz & 2.6 Hz, 1H), 6.25 (s, 1H), 3.85 (s, 3H), 2.84-2.72 (m, 4H), 2.37 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.7, 158.5, 150.5, 149.4, 139.4, 136.0, 134.8, 132.9, 130.7, 128.5, 128.2, 126.5, 124.1, 115.4, 114.0, 93.7, 55.5, 26.6, 25.1, 14.2. IR ($CHCl_3$, cm^{-1}): 2932, 1594, 1243, 752. MS (EI, *m/z*): 421 [M^+], 423 [M + 2]. Anal. calcd. for $C_{22}H_{20}BrN_3O$: C, 62.57; H, 4.77; N, 9.95. Found: C, 62.68; H, 4.79; N, 9.81.

General procedure for the synthesis of compounds 6a-k: A mixture of halo aldehyde (**1**, 1.0 mmol), 3-aminopyrrazole/5-aminopyrrazole (**5**, 1.0 mmol), $Pd(OAc)_2$ (2.5 mol%), PPh_3 (5 mol%) and K_2CO_3 (2.1 mmol) was irradiated in a closed vessel in a Synthos 3000 microwave reactor at 700 Watt (120 ^{o}C and 14 bar) for 15 minutes. After completion of the reaction, the reaction mixture was treated with water (40 mL) and then extracted with ethylacetate (30 mL x 3). The organic portion was washed with water, dried over anhydrous sodium sulfate and the solvent was removed in vacuo to obtain a crude product which on silica gel column chromatographic purification using EtOAc/hexane as the eluent afforded compounds **6a-k**.

*9-Methoxy-5a,6,7,11b-tetrahydrobenzo[h]pyrazolo[1,5-a]quinazoline (**6a**):* Red solid, mp: 126 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.41 (d, *J* = 9.0 Hz, 1H), 8.39 (s, 1H), 8.18 (d, *J* = 2.4 Hz, 1H), 6.98 (dd, *J* = 9.0 Hz & 2.7 Hz, 1H), 6.89 (d, *J* = 2.4 Hz, 1H), 6.73 (d, *J* = 2.4 Hz, 1H), 3.91 (s, 3H), 2.97 (s, 4H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 161.6, 148.8, 143.9, 142.5, 140.0, 131.4, 119.4, 114.1, 111.5, 96.3, 55.4, 29.0, 24.1. IR ($CHCl_3$, cm^{-1}): 2927, 1606, 1412, 1266, 1190, 944, 772. MS (EI, m/z): 251. Anal. calcd. for $C_{15}H_{13}N_3O$: C, 71.70; H, 5.21; N, 16.72; Found: C, 71.99; H, 5.43; N, 16.64.

*9-Methoxy-2-methyl-6,7-dihydrobenzo[h]pyrazolo[1,5-a]quinazoline (**6b**):* Gum. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.45 (d, *J* = 8.7 Hz, 1H), 8.29 (s, 1H), 6.91 (dd, *J* = 8.7 Hz & 2.4 Hz, 1H), 6.86 (d, *J* = 2.4 Hz, 1H), 6.48 (s, 1H), 3.89 (s, 3H), 2.89-2.96 (m, 4H), 2.57 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 161.4, 154.1, 150.8, 148.5, 142.4, 139.2, 131.4, 119.6, 114.7, 114.1, 111.4, 95.4, 55.4, 31.9, 29.7, 24.1. IR ($CHCl_3$, cm^{-1}): 2924, 1602, 1493, 1244, 1188, 1038, 774. MS (EI, m/z): 265.1. Anal. calcd. for $C_{16}H_{15}N_3O$: C, 72.43; H, 5.70; N, 15.84; Found: C, 72.47; H, 5.75; N, 15.95.

*6,7-Dihydrobenzo[h]pyrazolo[1,5-a]quinazoline (**6c**):* Yellow solid, mp: 65 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 9.41 (dd, *J* = 6.9 & 4.8 Hz, 1H), 8.41 (s, 1H), 8.19 (d, *J* = 2.4 Hz, 1H), 7.32- 7.53 (m, 3H), 6.75 (d, *J* = 2.4 Hz, 1H), 2.99- 2.91 (m, 4H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 150.1, 149.3, 143.8, 139.8, 131.1, 129.1, 128.8, 128.1, 126.9, 126.7, 124.1, 116.9, 96.7, 28.6. IR ($CHCl_3$, cm^{-1}): 2930, 1607, 1509, 1451, 1233, 1131, 765. MS (EI, m/z): 221. Anal. calcd. for $C_{14}H_{11}N_3$: C, 76.00; H, 5.01; N, 18.99; Found: C, 76.08; H, 5.31; N, 19.15.

*7-(p-Tolyl)pyrazolo[1,5-a]pyrimidine (**6d**):* Gum. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.52 (d, *J* = 4.5 Hz, 1H), 8.17 (d, *J* = 2.4 Hz, 1H), 7.94 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 6.89 (d, *J* = 4.5 Hz, 1H), 6.78 (d, *J* = 2.4 Hz, 1H), 2.46 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 149.9, 149.1, 146.9, 144.6, 141.6, 129.4, 129.1, 128.2, 106.9, 96.9, 29.7. IR ($CHCl_3$, cm^{-1}): 2922, 1604, 1538, 1507, 1251, 1219, 1130, 808, 772. MS (EI, m/z): 209. Anal. calcd. for $C_{13}H_{11}N_3$: C, 74.62; H, 5.30; N, 20.08; Found: C, 74.74; H, 5.46; N, 19.87.

*2-Methyl-7-(naphthalen-2-yl)pyrazolo[1,5-a]pyrimidine (**6e**):* White solid, mp: 122 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.58 (s, 1H), 8.46 (d, *J* = 4.5 Hz, 1H), 8.07 (d, *J* = 7.0 Hz, 1H), 8.05-7.52 (m, 5H), 6.88 (d, *J* = 4.5 Hz, 1H), 6.59 (s, 1H), 2.54 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 155.1, 150.7, 148.6, 146.2, 134.4, 132.9, 129.8, 128.9, 128.6, 128.3, 127.8, 127.7, 126.7, 125.6, 106.9, 96.4, 14.9. IR ($CHCl_3$, cm^{-1}): 2925, 1601, 1532, 1505, 1352, 1259, 1208, 1067, 861, 810, 774. MS (EI, m/z): 259.1. Anal. calcd. for $C_{17}H_{13}N_3$: C, 78.74; H, 5.05; N, 16.20; Found: C, 78.82; H, 5.08; N, 16.21.

*2-(4-Methoxyphenyl)-7-(naphthalen-2-yl)pyrazolo[1,5-a]pyrimidine (**6f**):* Yellow solid, mp: 175 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.73 (s, 1H), 8.48 (d, *J* = 4.3 Hz, 1H), 8.18 (d, *J* = 8.6 Hz, 1H), 8.01-7.89 (m, 4H), 7.61-7.54 (m, 2H), 7.02 (s, 1H), 6.99-6.94 (m, 4H), 3.84 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.4, 155.9, 151.1, 148.6, 146.3, 134.4, 132.9, 130.2, 129.1, 128.4, 128.1, 127.9, 127.8, 127.7, 126.7, 125.8, 125.6, 114.2, 107.1, 92.9, 55.3. IR ($CHCl_3$, cm^{-1}): 2924, 1607, 1524, 1249, 1031, 773. MS (EI, m/z): 351.1. Anal. calcd. for $C_{23}H_{17}N_3O$: C, 78.61; H, 4.88; N, 11.96; Found: C, 78.68; H, 4.79; N, 11.94.

*2-Methyl-7-(thiophen-3-yl)pyrazolo[1,5-a]pyrimidine (**6g**):* Yellow solid, mp: 112 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.64 (d, *J* = 3.8 Hz, 1H), 7.73 (d, *J* = 5.0 Hz, 1H), 7.29-7.13 (m, 3H), 6.59 (s, 1H), 2.61 (s, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 155.1, 132.5, 131.6, 131.56, 127.7, 96.1, 14.9. MS (EI, m/z): 215. Anal. calcd. For $C_{11}H_9N_3S$: C, 61.37; H, 4.21; N, 19.52; Found: C, 61.42; H, 4.28; N, 19.39.

*7-(3-Nitrophenyl)pyrazolo[1,5-a]pyrimidine-3-carbonitrile (**6h**):* Brown solid, mp 215 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.93 (s, 1H), 8.86 (d, *J* = 4.4 Hz, 1H), 8.50 (d, *J* = 7.8 Hz, 1H), 8.48 (s, 1H), 8.41 (d, *J* = 7.8 Hz, 1H), 7.84 (t, *J* = 8.0 Hz, 1H), 7.28 (d, *J* = 6.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 152.6, 147.3, 135.1, 130.9, 130.1, 126.4, 124.6, 110.0. IR ($CHCl_3$, cm^{-1}): MS (EI, m/z): 265.0. Anal. calcd. For $C_{13}H_7N_5O_2$: C, 58.87; H, 2.66; N, 26.41; Found: C, 58.94; H, 2.57; N, 26.47.

*8-Methylpyrazolo[1,5-a]quinazoline (**6i**):* Yellow solid, mp: 127 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.83 (s, 1H), 8.28 (s, 1H), 8.09 (d, *J* = 2.1 Hz, 1H), 7.84-7.79 (m, 1H), 7.37-7.35 (m, 1H), 6.79 (d, *J* = 2.1 Hz, 1H), 2.63 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 151.5, 145.9, 145.8, 142.6, 136.3, 128.3, 126.9, 116.5, 114.4, 99.5, 29.7. IR ($CHCl_3$, cm^{-1}): 2920, 1624, 1599, 1286, 868, 773. MS (EI, m/z): 183. Anal. calcd. for $C_{11}H_9N_3$: C, 72.11; H, 4.95; N, 22.94; Found: C, 72.18; H, 4.90; N, 22.81.

*7-Fluoropyrazolo[1,5-a]quinazoline (**6j**):* Gum. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.84 (s, 1H), 8.52 -8.11 (m, 1H), 8.11 (d, *J* = 2.1 Hz, 1H), 7.68-7.59 (m, 2H), 6.84 (d, *J* = 2.1 Hz, 1H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 157.9, 150.7, 142.7, 122.9, 122.6, 117.2, 117.1, 113.1, 112.8, 100.2. IR ($CHCl_3$, cm^{-1}): 2923, 1560, 1244, 772. MS (EI, m/z): 187.0. Anal. calcd. for $C_{10}H_6FN_3$: C, 64.17; H, 3.23; N, 22.45; Found: C, 64.34; H, 3.20; N, 22.47.

*7-Fluoro-2-methylpyrazolo[1,5-a]quinazoline (**6k**):* White solid, mp: 130 ^{o}C. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.75 (s, 1H), 8.39 (m, 1H), 7.61- 7.51 (m, 2H), 6.61 (s, 1H), 2.55 (s, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 159.1 ($J_{C\text{-}F}$ = 244.7 Hz), 152.7, 150.3, 146.1, 132.8, 122.6 ($J_{C\text{-}F}$ = 24.6 Hz), 118.9 ($J_{C\text{-}F}$ = 7.8 Hz), 116.8 ($J_{C\text{-}F}$ = 8.1 Hz), 112.6 ($J_{C\text{-}F}$ = 22.6 Hz), 99.5, 29.5. IR ($CHCl_3$, cm^{-1}): 3054, 2923, 1607, 1558, 1253, 1244, 1152, 1132, 949, 826. MS (EI, m/z): 201. Anal. calcd. for $C_{11}H_8FN_3$: C, 65.66; H, 4.01; N, 20.88; Found: C, 65.74; H, 3.87; N, 20.79.

*7-(3-Aminophenyl)pyrazolo[1,5-a]pyrimidine-3-carbonitrile (**6l**):* To a stirring solution of compound **6h** (0.5 mmol) in ethanol catalytic amount of Pd/C was added. The reaction mixture was stirred under H_2 gas for 2 hours. After completion of the reaction, the catalyst was filtered off and the solvent was removed under vacuo. The crude product thus obtained on silica gel column chromatographic purification using EtOAc/hexane (2:3) as the eluent

afforded compound **6l**. Yellow crystals, mp: 227 ^{o}C. ^{1}H NMR (DMSO-d_6, 500 MHz) δ 8.86 (d, *J* = 4.0 Hz, 1H), 8.85 (s, 1H), 7.45 (dd, *J* = 4.5 Hz & 2 Hz, 1H), 7.27 (s, 1H), 7.25 (d, *J* = 8.5 Hz, 1H), 7.14 (d, *J* = 8.2 Hz, 1H), 6.82 (d, *J* = 8.2 Hz, 1H), 5.47 (bs, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ 154.0, 151.5, 149.0, 148.9, 147.5, 130.4, 129.6, 117.4, 117.4, 114.8, 113.9, 110.7, 81.5. IR ($CHCl_3$, cm^{-1}): 2917, 1446, 845, 781. MS (EI, m/z): 235. Anal. calcd. for $C_{13}H_9N_5$: C, 66.37; H, 3.86; N, 29.77; Found: C, 66.47; H, 3.89; N, 29.61.

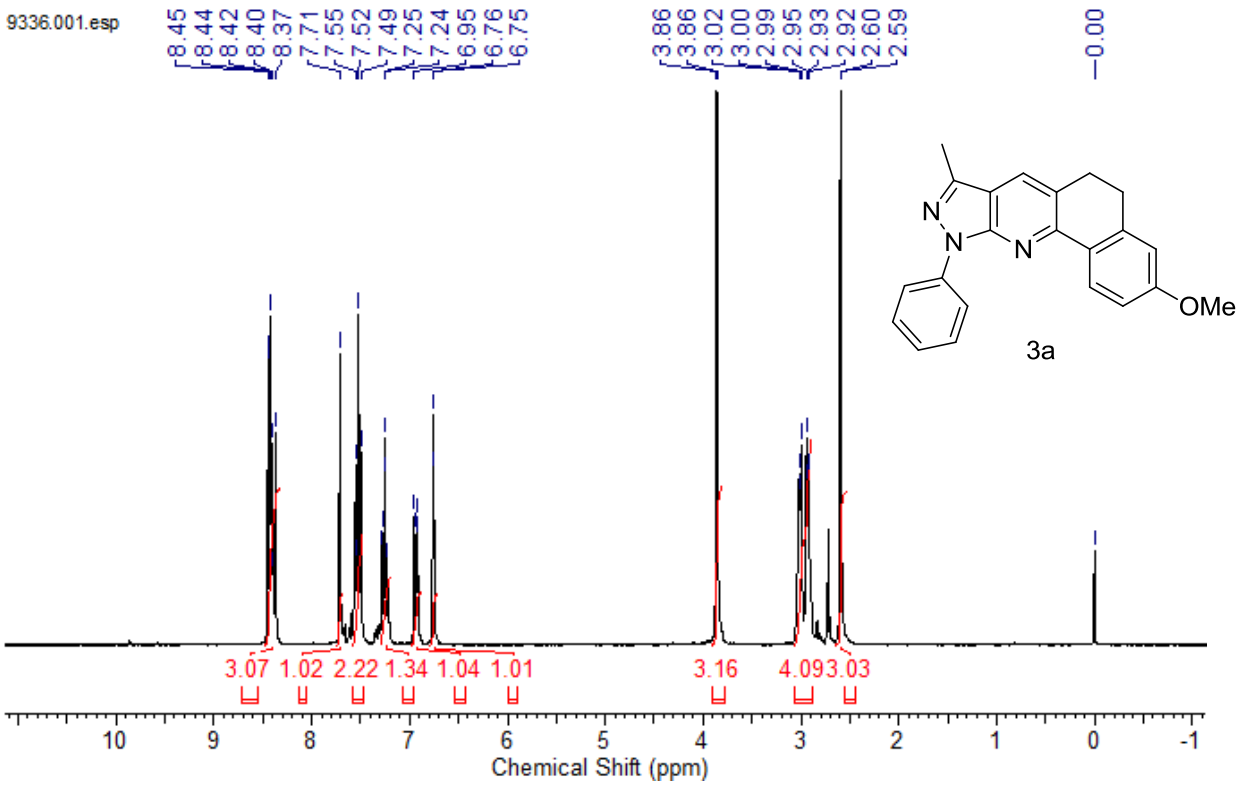
MTT ASSAY PROTOCOL

Cellular viability in the presence of test compounds was determined by MTT-micro cultured tetrazolium assay. The cells seeded to flat bottomed 96 (1000 cells/100 µl) well plates & cultured in the medium containing 10% serum and allowed to attach and recover for 24 hours in a hid chamber containing 5% CO_2. MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide; Sigma catalog No M2128) was dissolved in PBS at 5 mg/mL and filtered to sterilize. Different concentrations of compounds were added to the cells. After 48 hours, stock MTT solution (10 µl) was added to the culture plate. Cells were again kept in CO_2 incubator for 2 hours. After incubation 100 µL of DMSO was added and mixed. The absorbance was read at 562 nm in a plate reader. The results were represented as percentage of cytotoxicity/viability. All the experiments were carried out in triplicates. From the percentage of cytotoxicity the IC_{50} values were calculated.

Media used was MEM (Catalog No M0643), DPBS (Catalog No D5652), 1X antibiotic solution of 100X, (Catalog No A5955), 1% Sodium pyruvate (Catalog No.S8636), 1% Non essential amino acids (Catalog No M7145), 10% fetal bovine ser (Catalog No F2442), DMSO (Catalog No D5879), Trypsin-EDTA solution **(**0.25%, 2.5 g porcine trypsin and 0.2 g EDTA) (Catalog No T4049).

Reference

(1) S. Myadaraboina, M. Alla, V. Saddanapu, V. R. Bommena and A. Addlagatta, *Eur. J. Med. Chem.* 2010, 45, 5208.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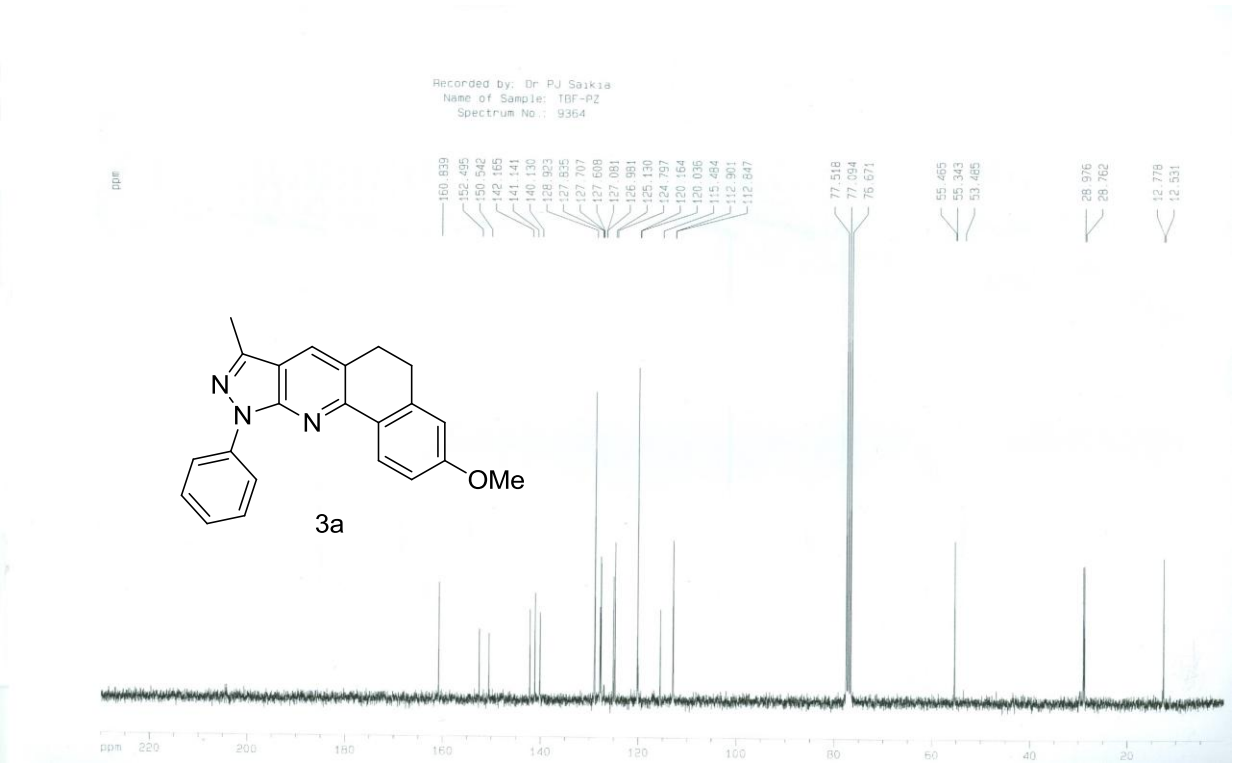
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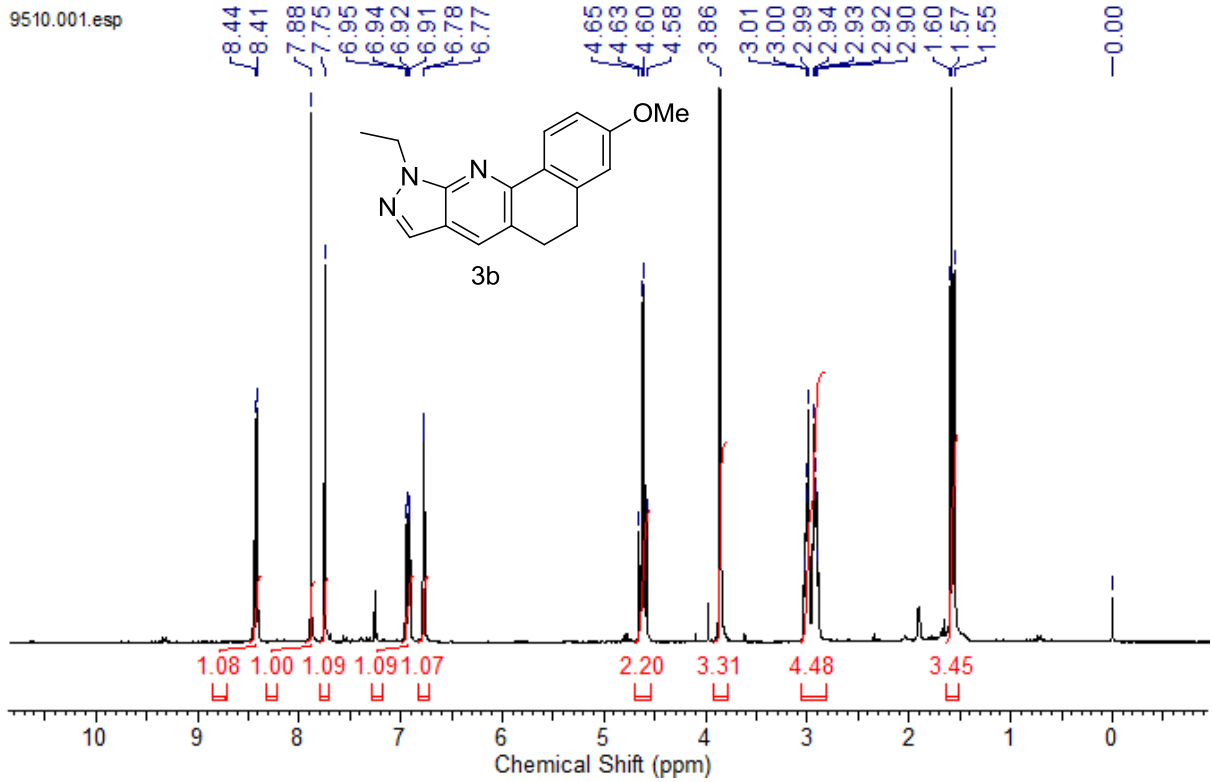
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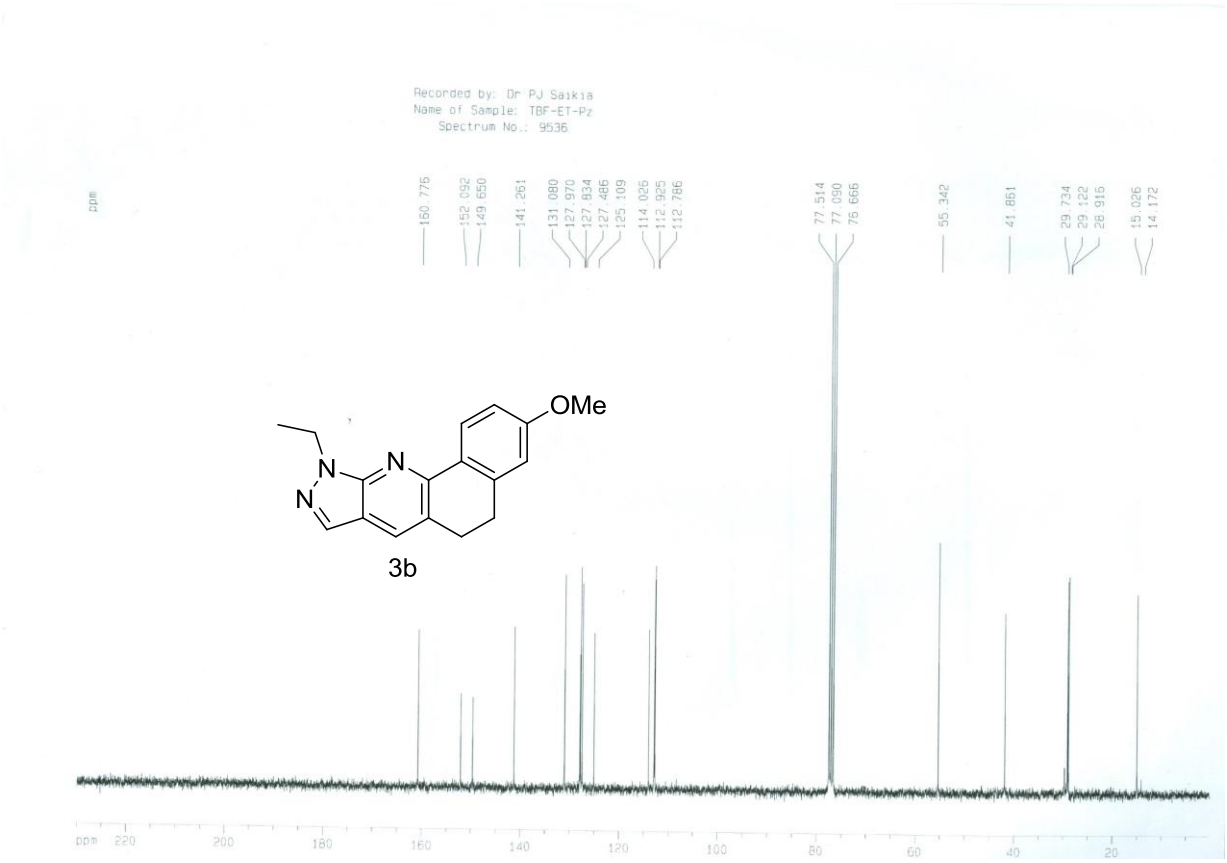
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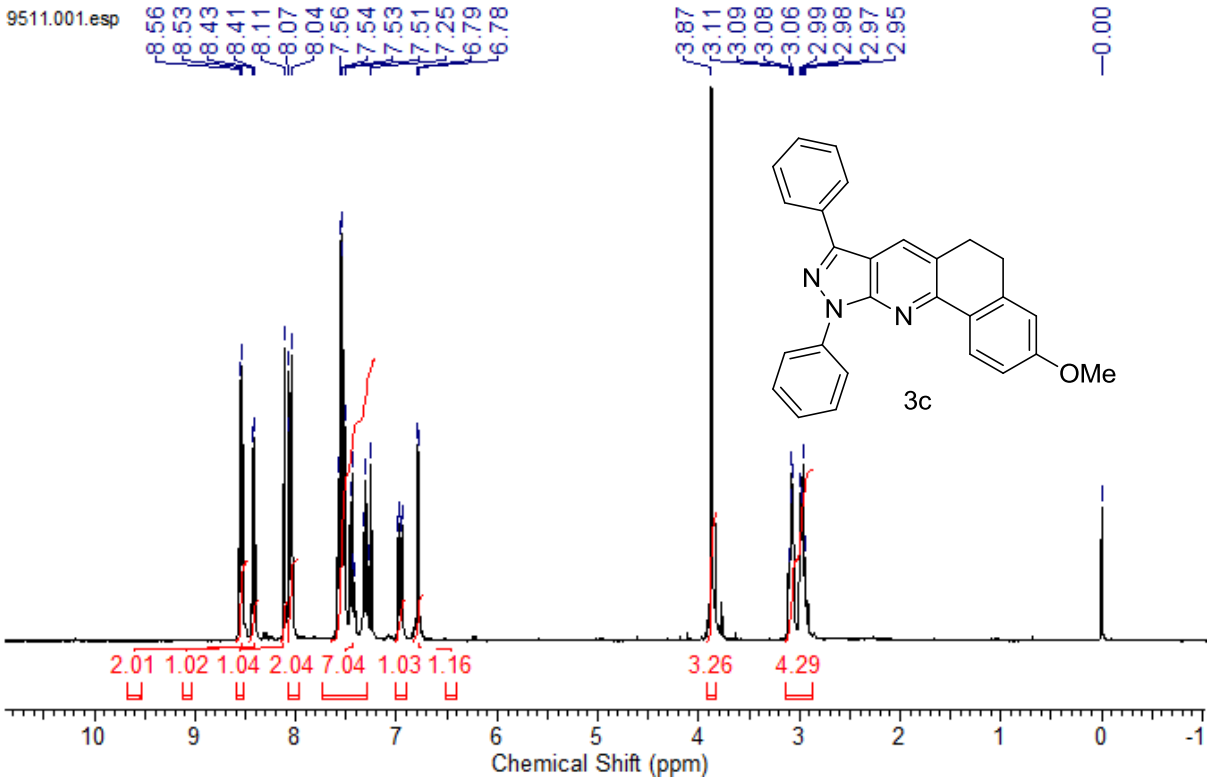
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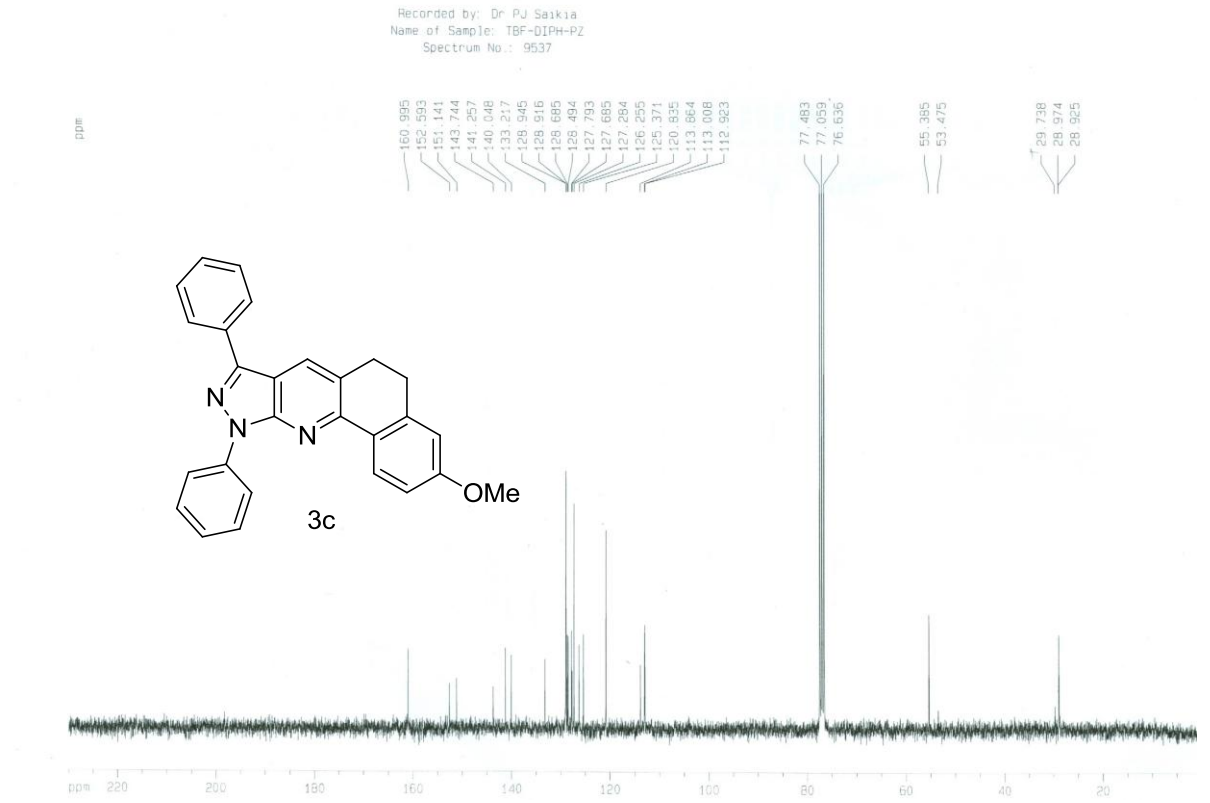
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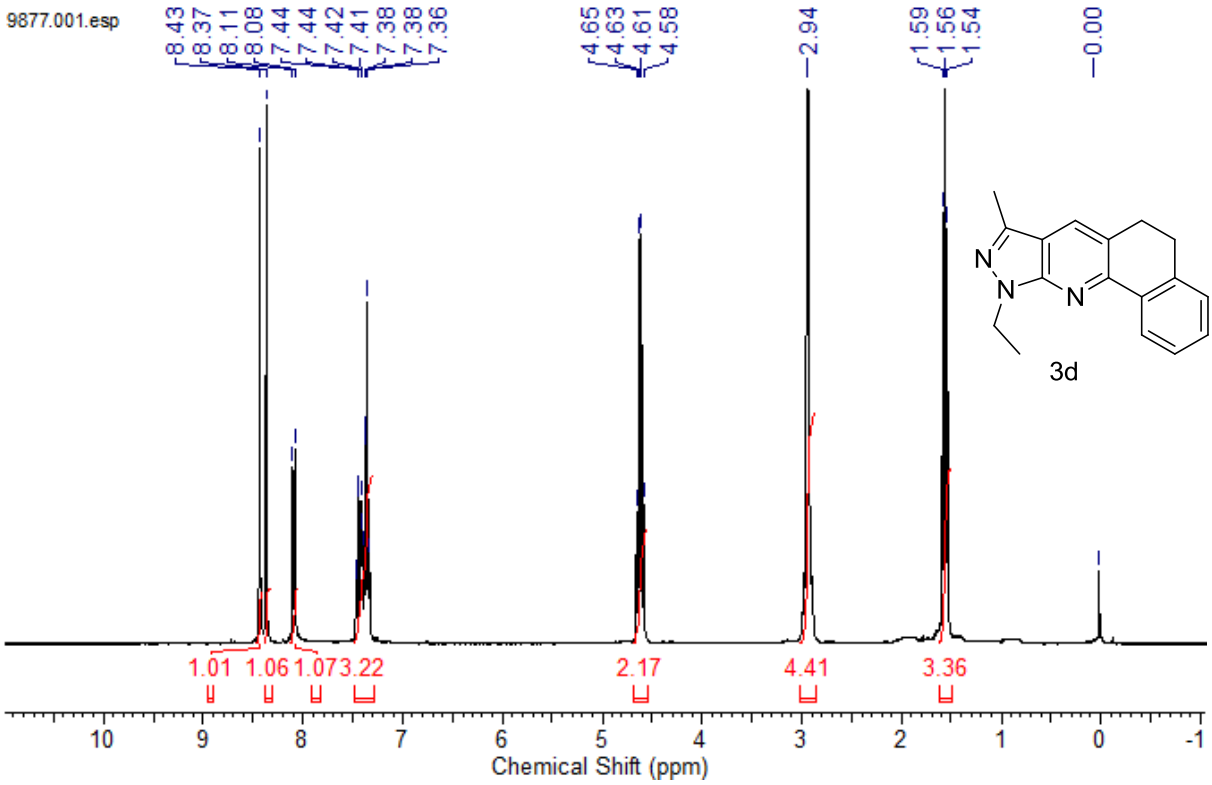
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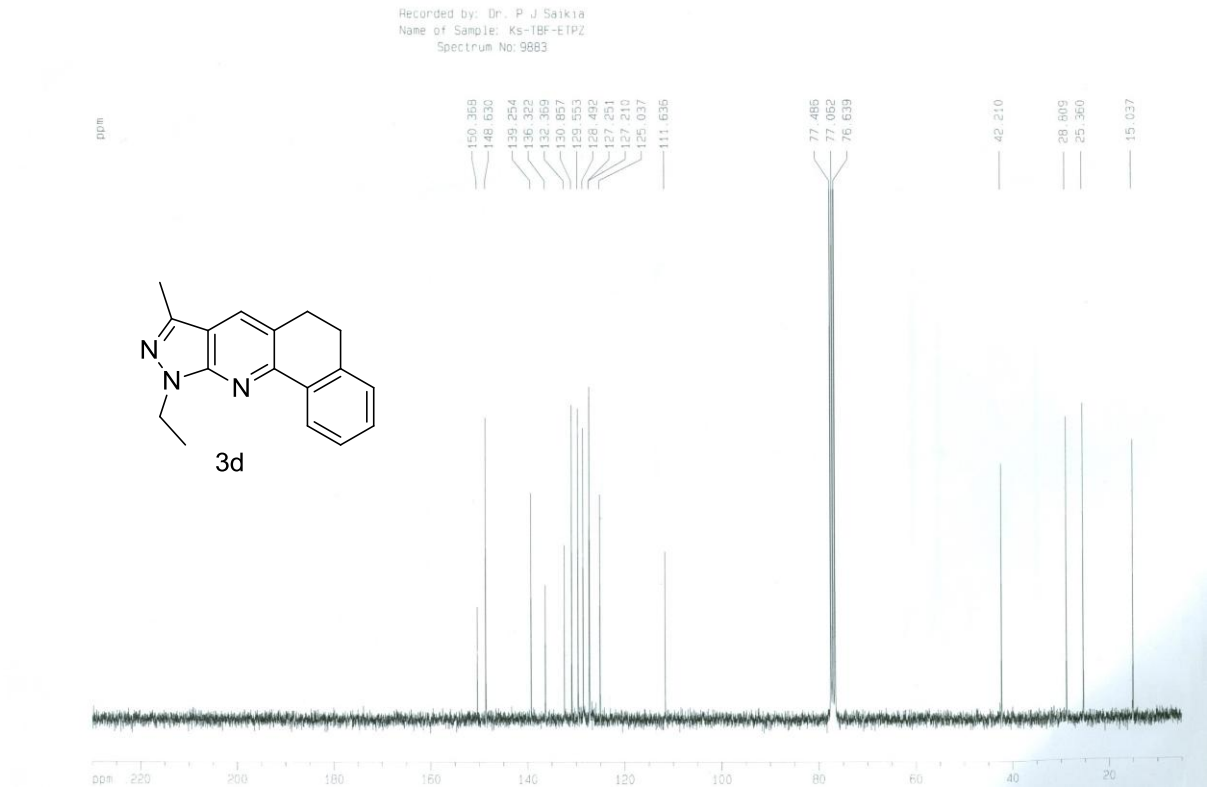
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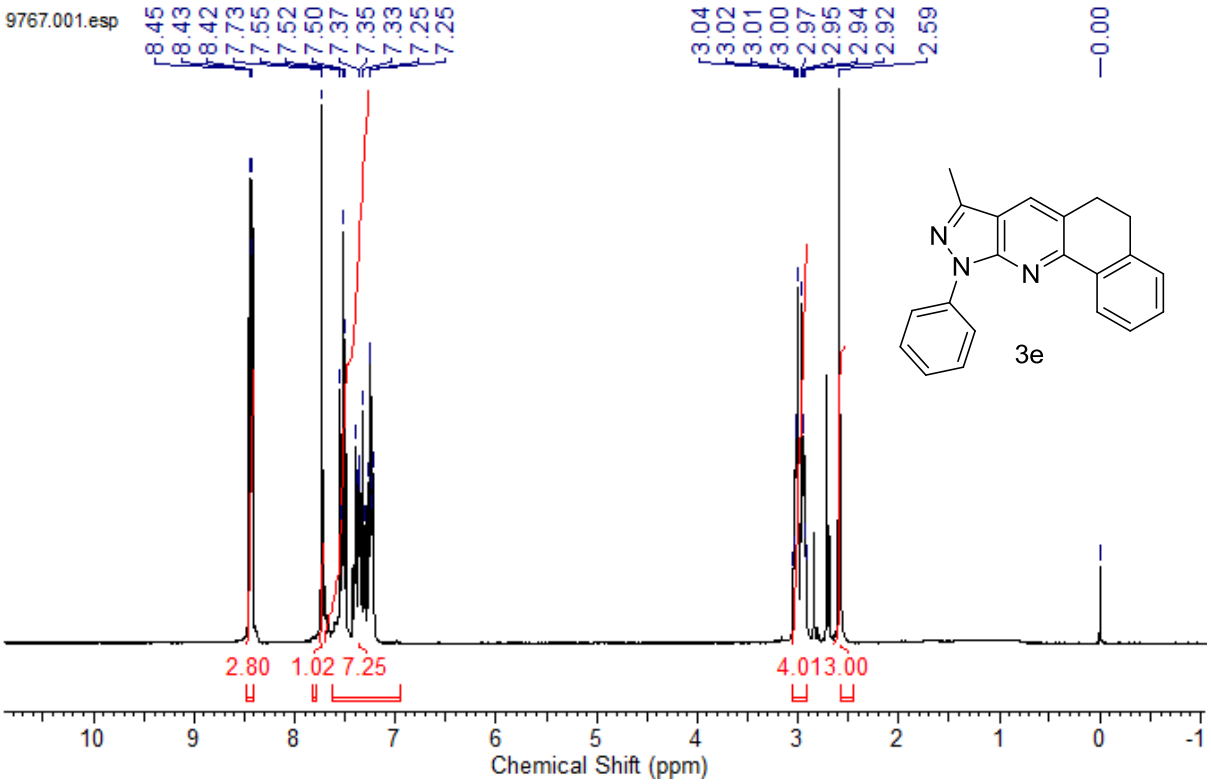
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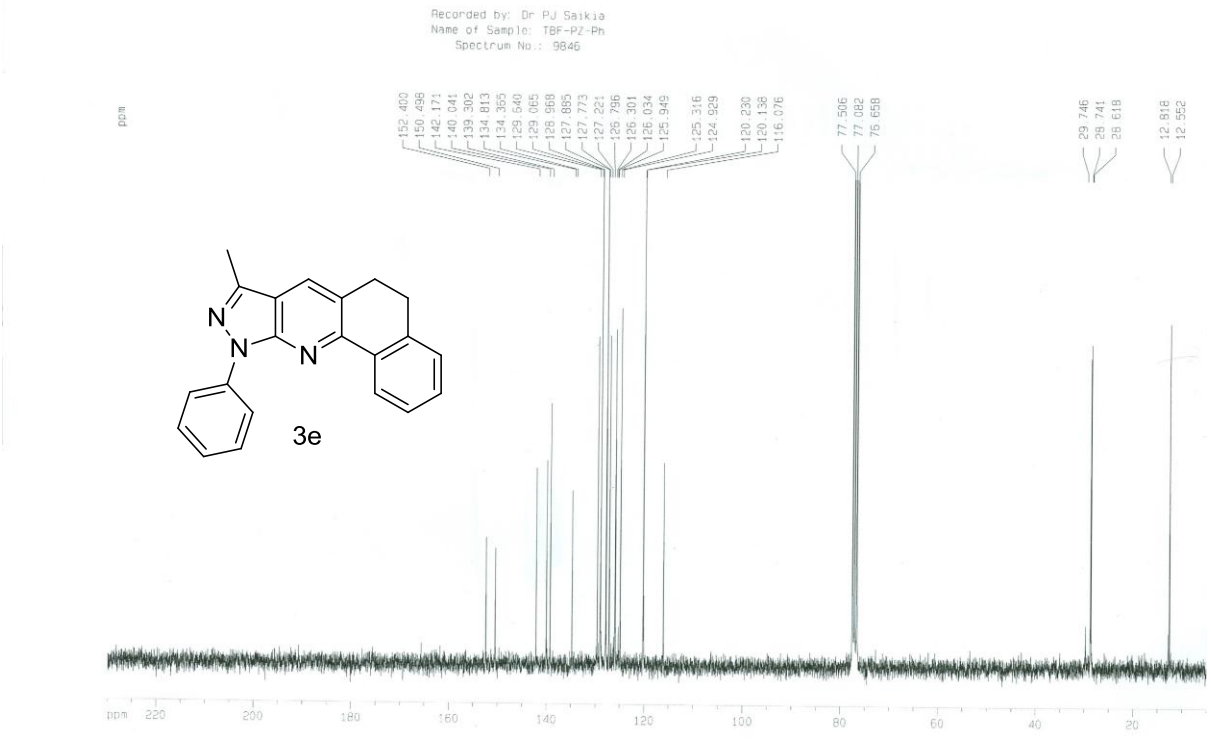
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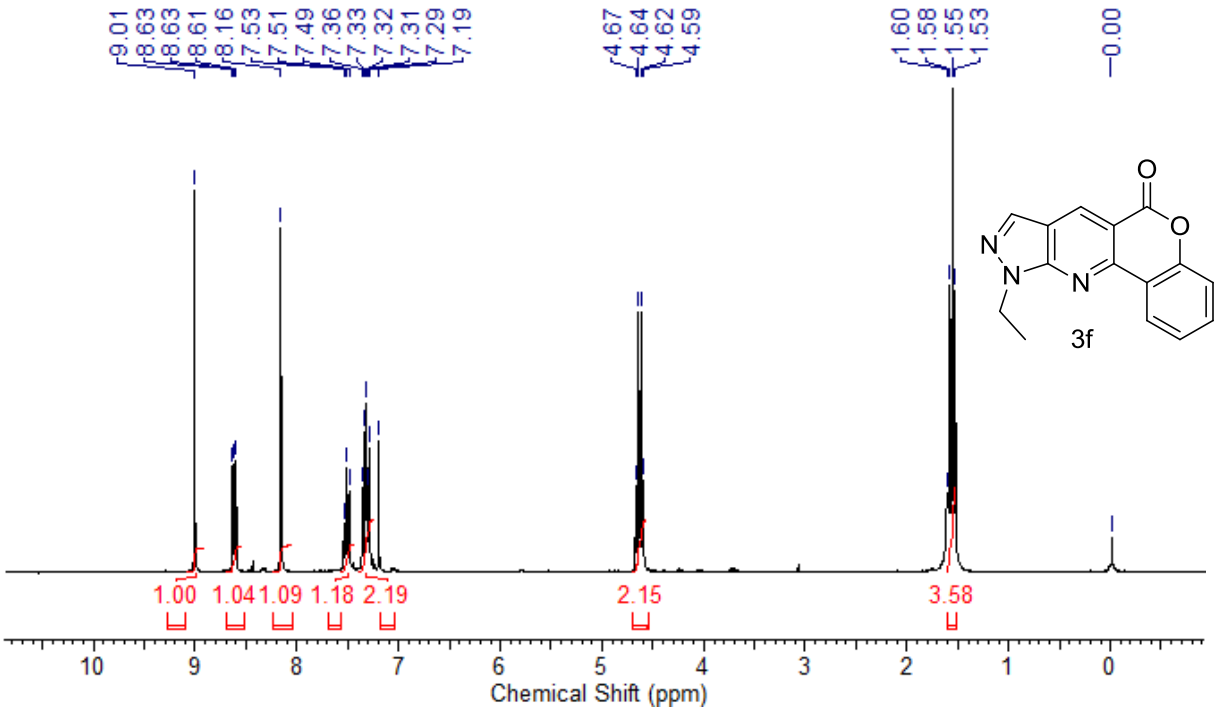
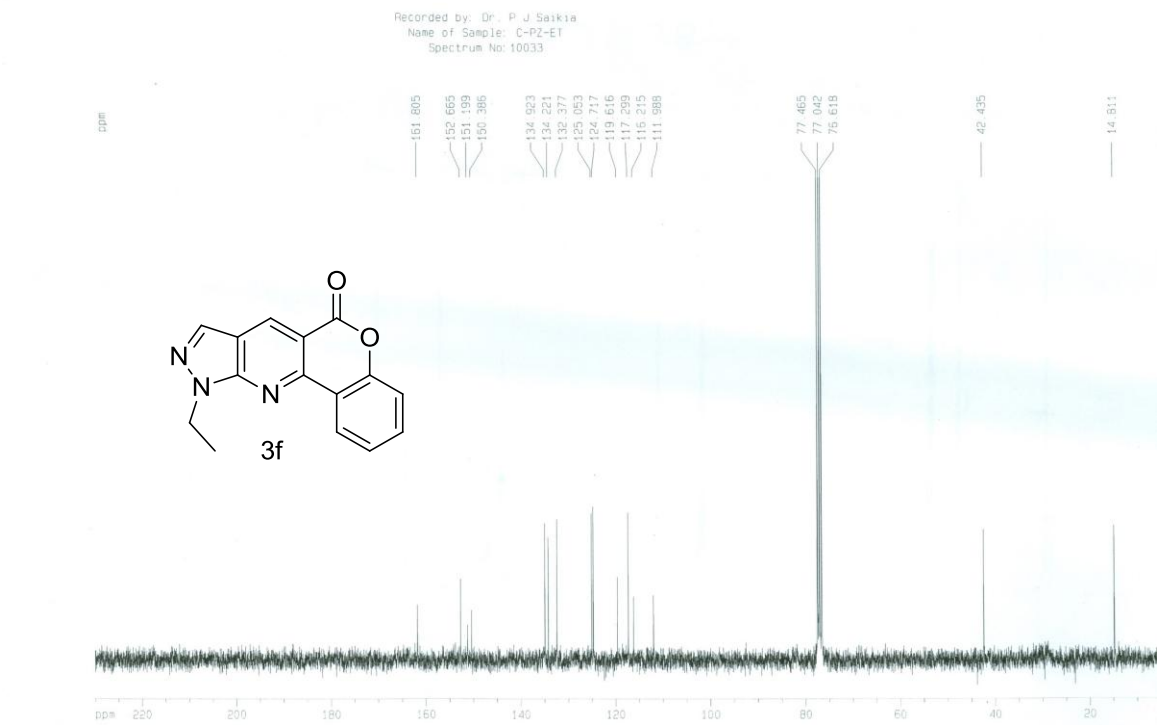
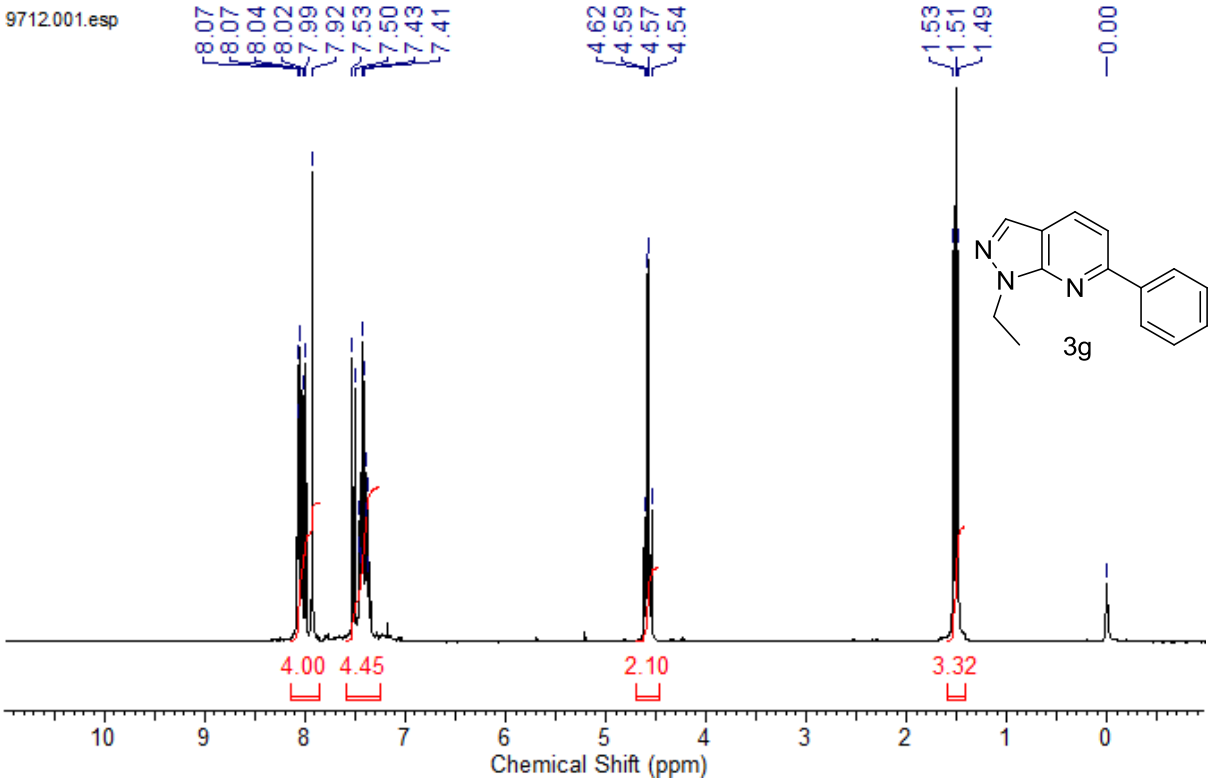
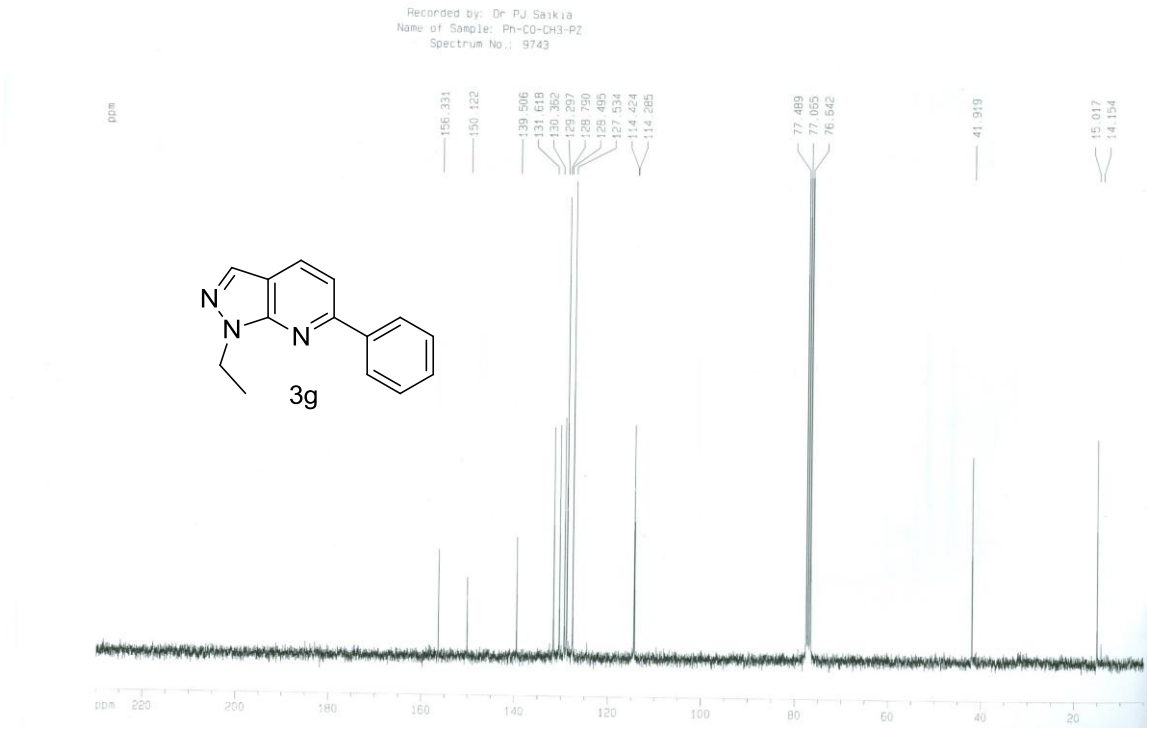
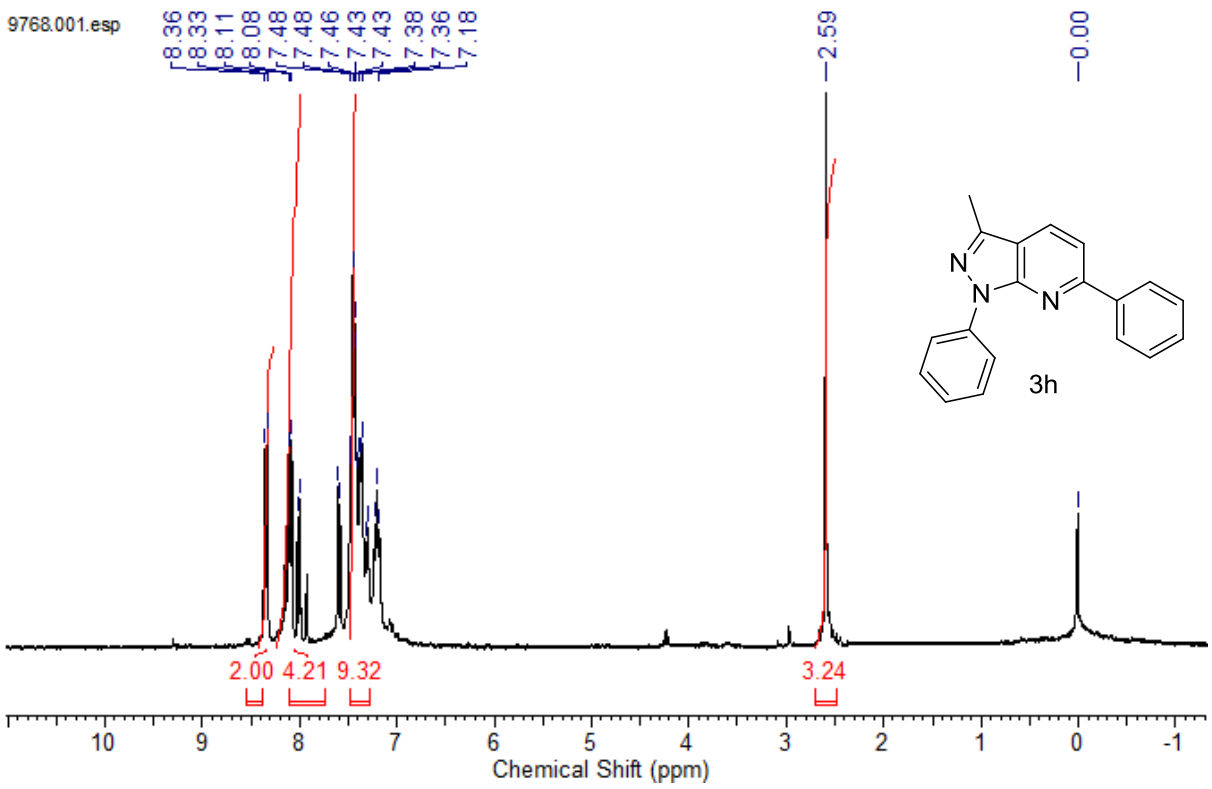
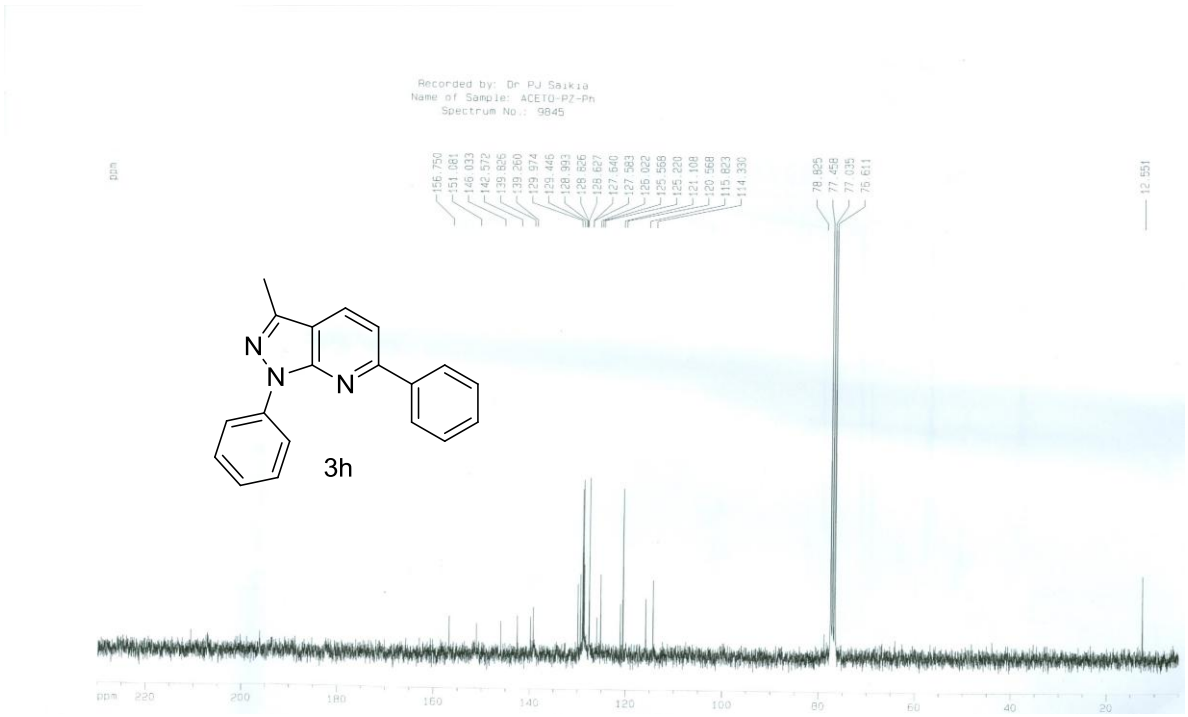


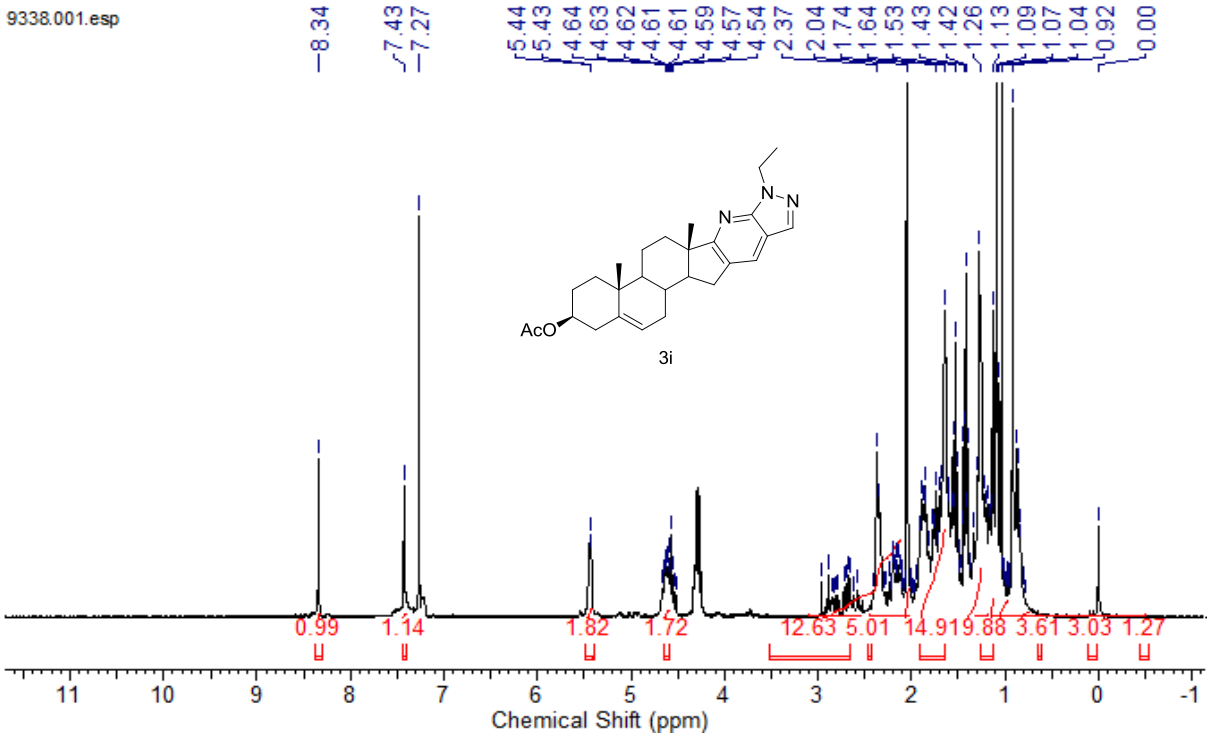


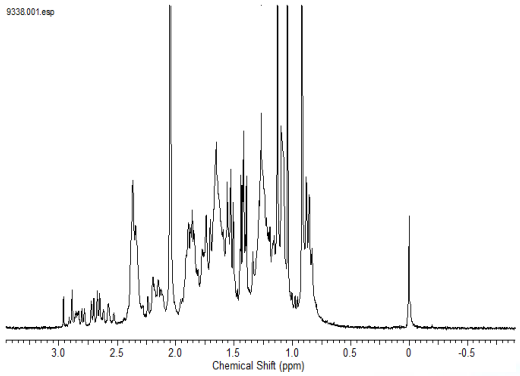

9712.001.esp
8.07
8.07
8.04
8.02
7.99
7.92
7.53
7.50
7.43
7.41
4.62
4.59
4.57
4.54
1.53
1.51
1.49
0.00
4.00
4.45
2.10
3.32
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
N
N
N
3g

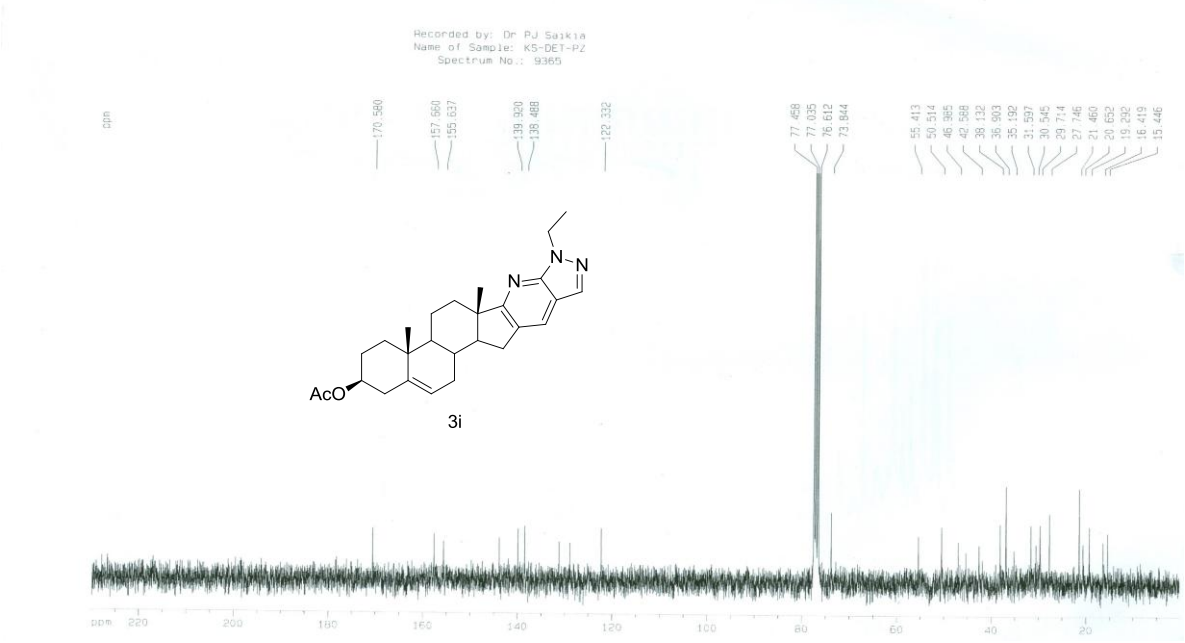

Recorded by: Dr PJ Saikia
Name of Sample: Ph-CO-CH3-PZ
Spectrum No.: 9743
ppm
156.331
150.122
139.506
131.618
130.362
129.297
128.790
126.495
127.534
114.424
114.285
77.489
77.065
76.642
41.919
15.017
14.154
N
N
N
3g
ppm
220
200
180
160
140
120
100
80
60
40
20

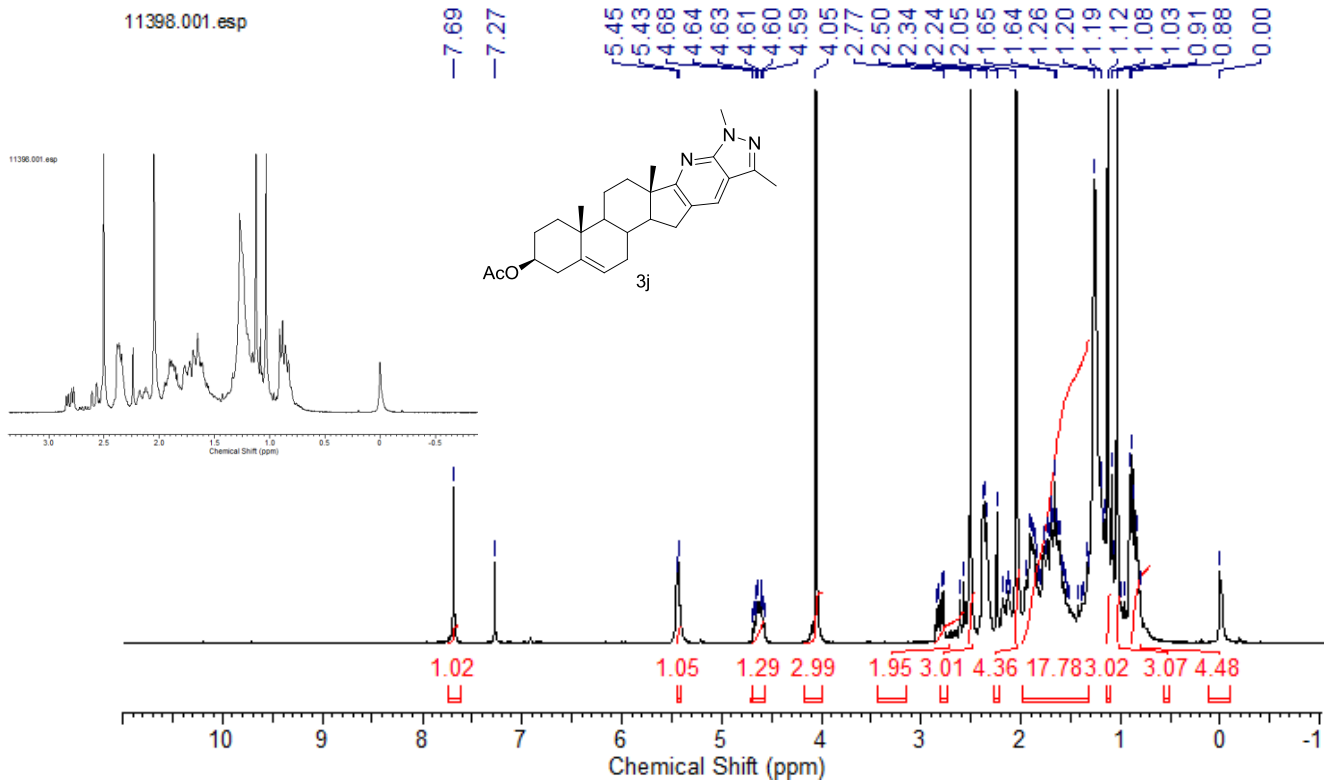

9768.001.esp
8.36
8.33
8.11
8.08
7.48
7.48
7.46
7.43
7.43
7.38
7.36
7.18
2.59
0.00
2.00
4.21
9.32
3.24
10
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)
3h

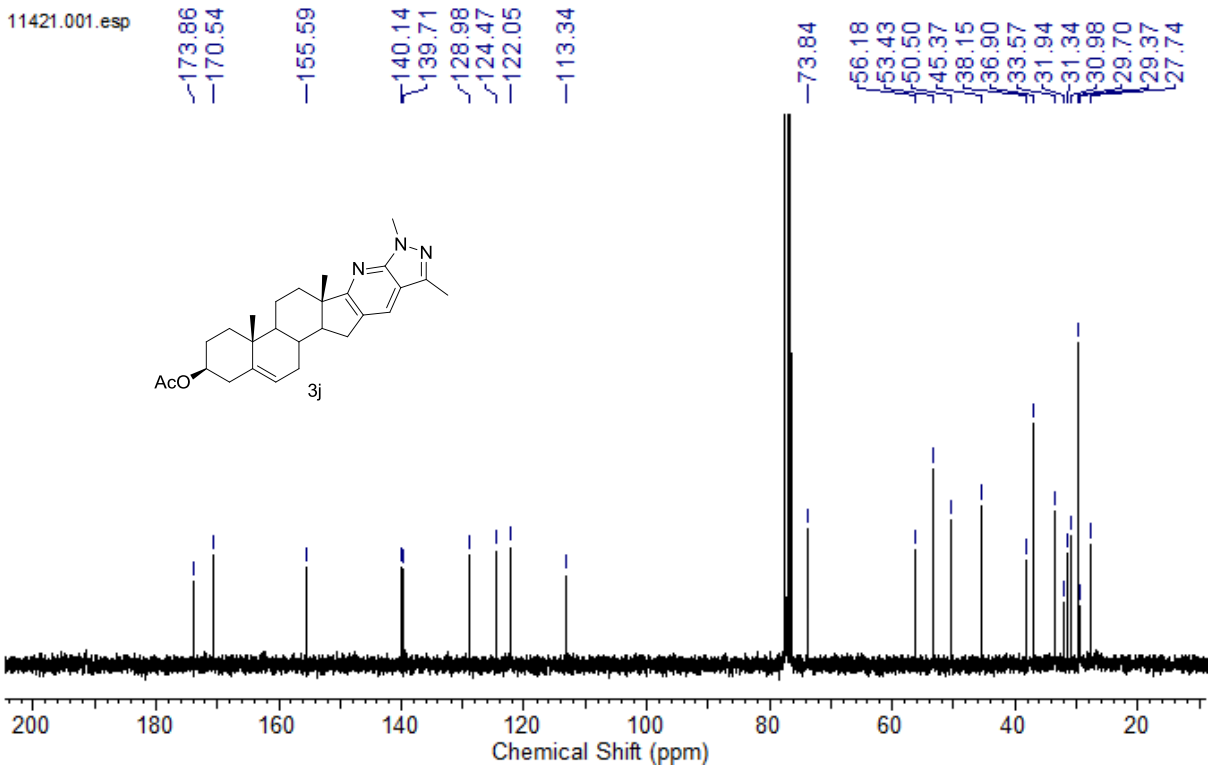

Recorded by: Dr PJ Saikia
Name of Sample: ACETO-PZ-Ph
Spectrum No.: 9845
156.750
151.081
146.033
142.572
139.826
139.260
129.974
129.446
128.993
128.826
128.627
127.840
127.583
126.022
125.586
125.220
121.109
120.568
115.823
114.330
78.925
77.458
77.035
76.611
12.551
ppm
220
200
180
160
140
120
100
80
60
40
20
3h

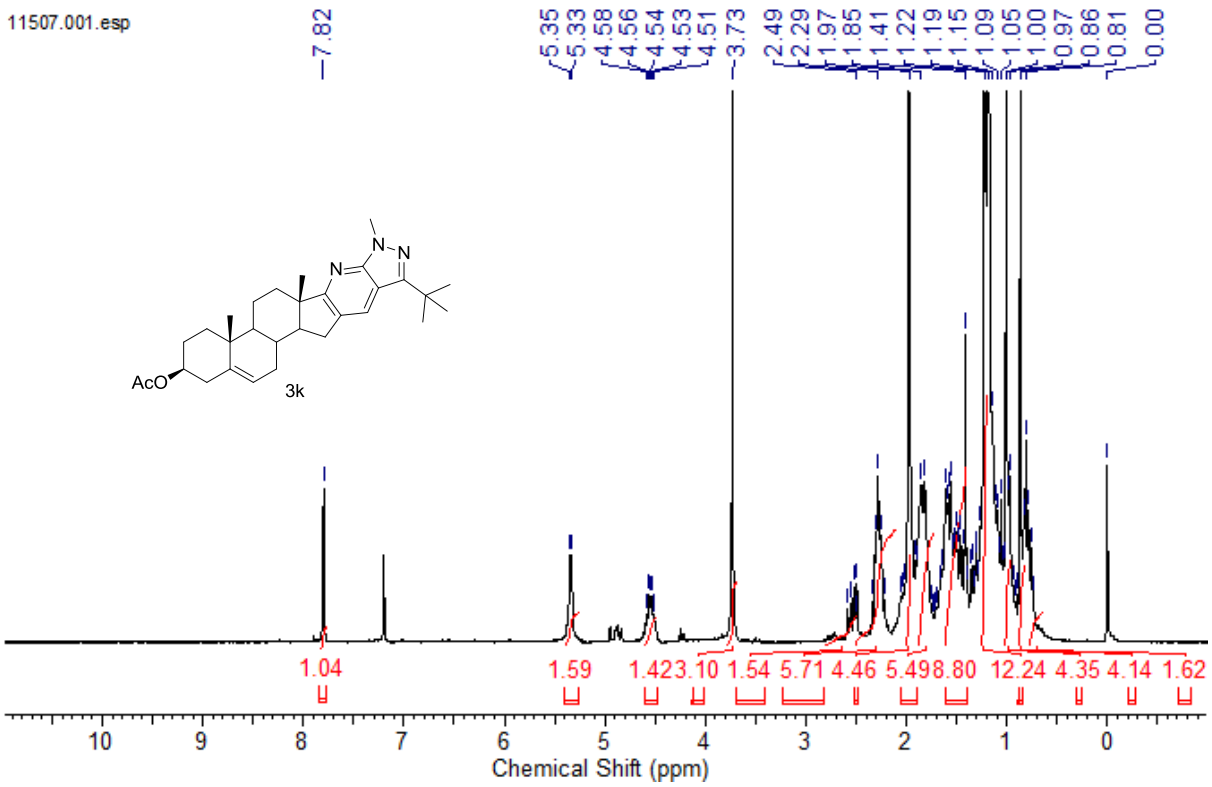

9338.001.esp
8.34
7.43
7.27
5.44
5.43
4.64
4.63
4.62
4.61
4.61
4.59
4.57
4.54
2.37
2.04
1.74
1.64
1.53
1.43
1.42
1.26
1.13
1.09
1.07
1.04
0.92
0.00
AcO
3i
0.99
1.14
1.82
1.72
12.63
5.01
14.91
9.88
3.61
3.03
1.27
Chemical Shift (ppm)

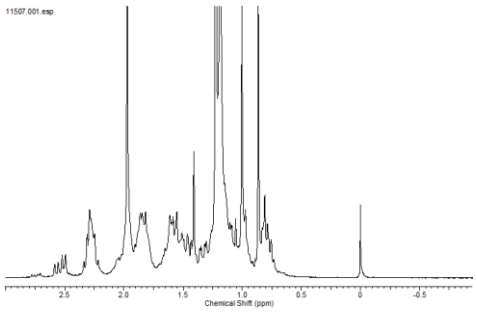

9338.001.esp
Chemical Shift (ppm)

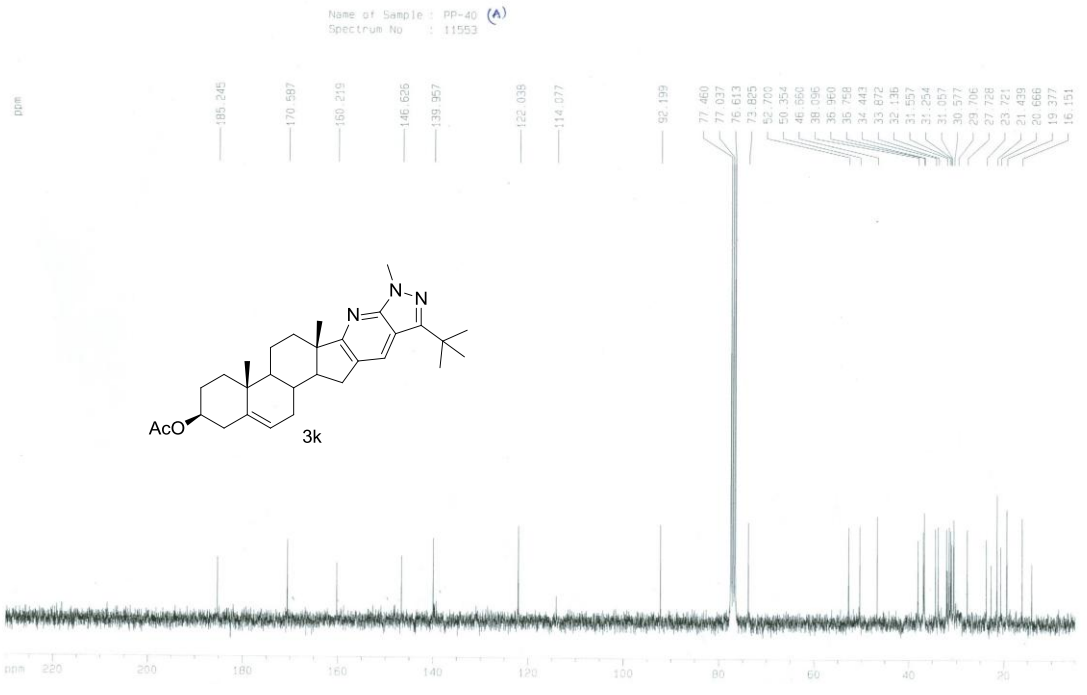

Recorded by: Dr PJ Saikia
Name of Sample: KS-DET-PZ
Spectrum No.: 9365
170.580
157.660
155.637
139.920
138.488
122.332
77.458
77.035
76.612
73.844
55.413
50.514
46.985
42.568
38.132
36.903
35.192
31.597
30.545
29.714
27.746
21.460
20.652
19.292
16.419
15.446
AcO
3i

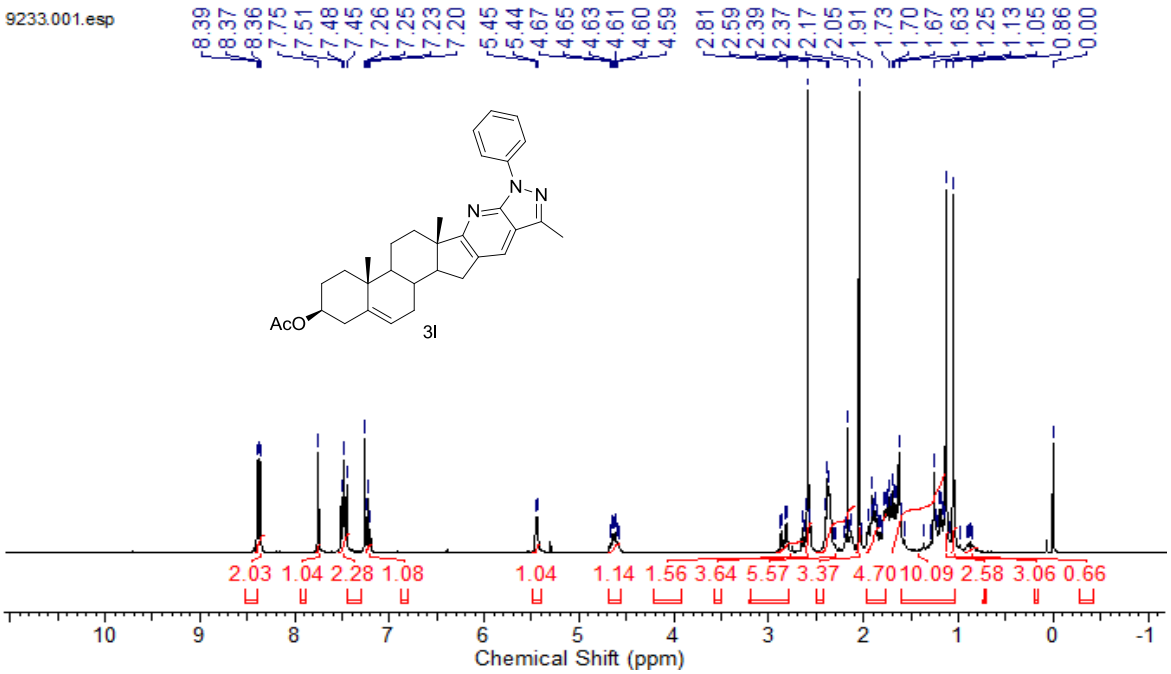

11398.001.esp
7.69
7.27
5.45
5.43
4.68
4.64
4.63
4.61
4.60
4.59
4.05
2.77
2.50
2.34
2.24
2.05
1.65
1.64
1.26
1.20
1.19
1.12
1.08
1.03
0.91
0.88
0.00
AcO
3j
1.02
1.05
1.29
2.99
1.95
3.01
4.36
17.78
3.02
3.07
4.48
Chemical Shift (ppm)

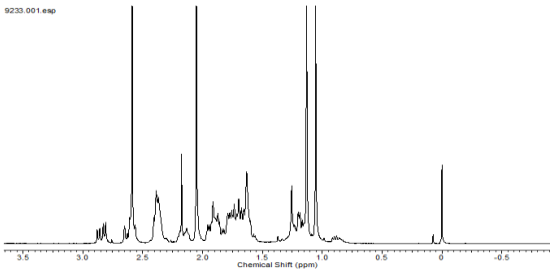

11421.001.esp
173.86
170.54
155.59
140.14
139.71
128.98
124.47
122.05
113.34
73.84
56.18
53.43
50.50
45.37
38.15
36.90
33.57
31.94
31.34
30.98
29.70
29.37
27.74
AcO
3j
Chemical Shift (ppm)

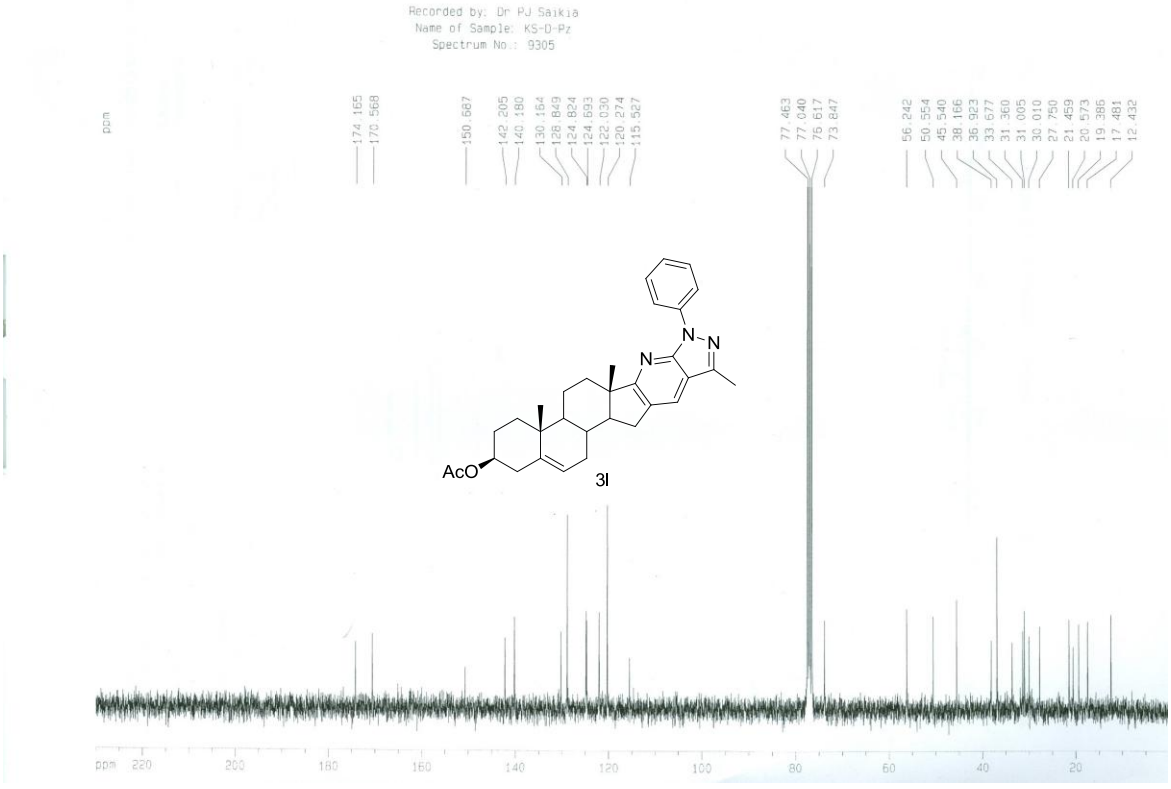

11507.001.esp
7.82
5.35
5.33
4.58
4.56
4.54
4.53
4.51
3.73
2.49
2.29
1.97
1.85
1.41
1.22
1.19
1.15
1.09
1.05
1.00
0.97
0.86
0.81
0.00
AcO
3k
1.04
1.59
1.42
3.10
1.54
5.71
4.46
5.49
8.80
12.24
4.35
4.14
1.62
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

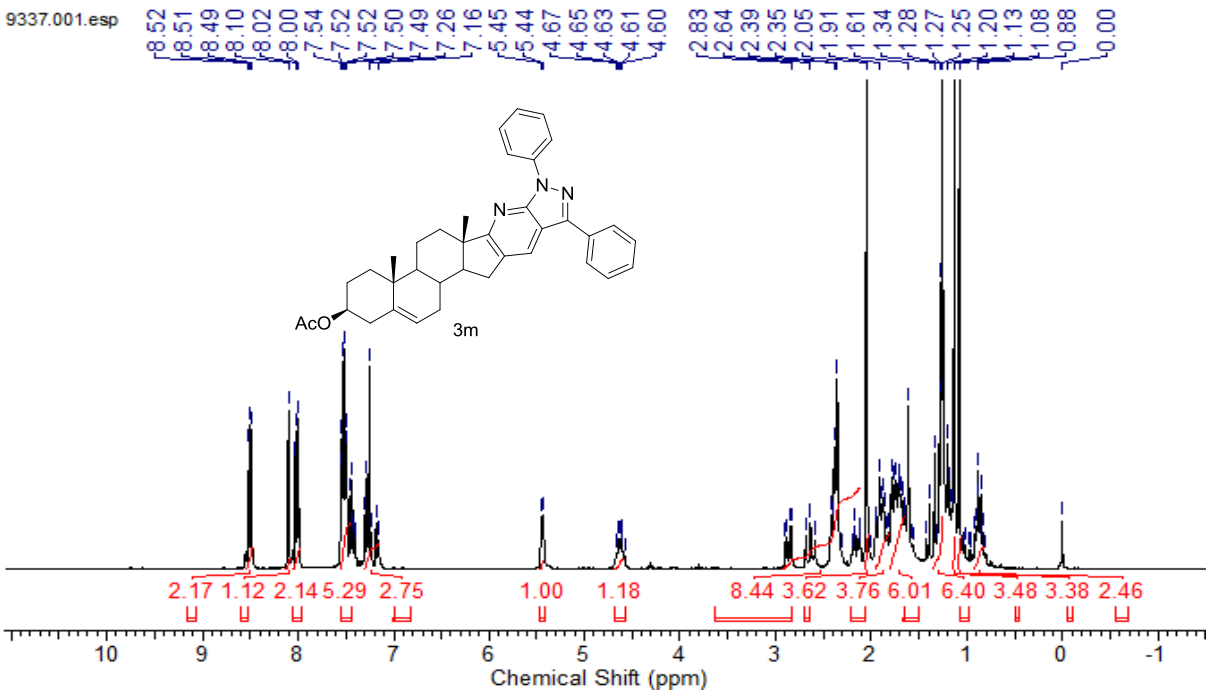

11507.001.esp
Chemical Shift (ppm)

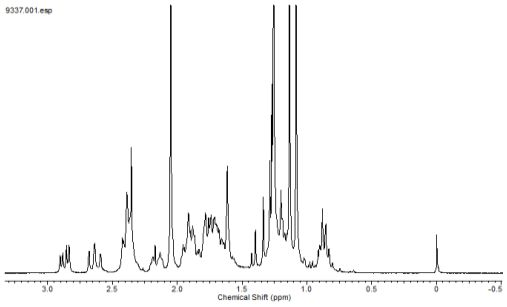

Name of Sample : PP-40
Spectrum No : 11553
ppm
185.245
170.587
160.219
146.626
139.957
122.038
114.077
92.199
77.460
77.037
76.613
73.825
52.700
50.354
46.560
38.096
36.960
35.758
34.443
33.872
32.136
31.557
31.254
31.057
30.577
29.706
27.728
23.721
21.439
20.666
19.377
16.151
AcO
3k
ppm
220
200
180
160
140
120
100
80
60
40
20

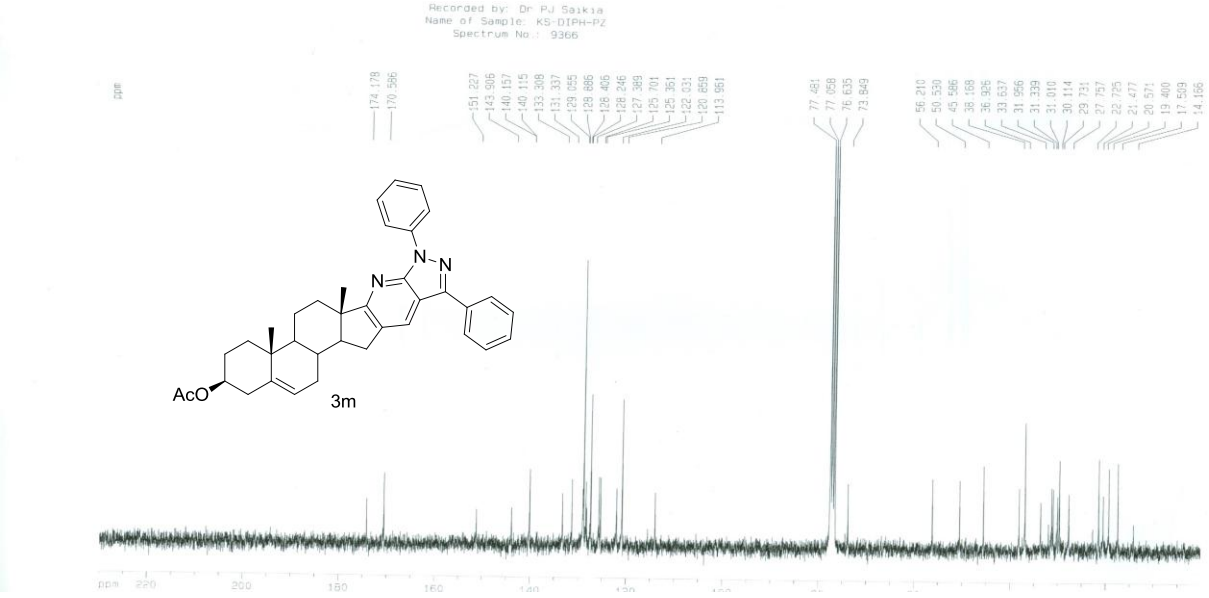

9233.001.esp
8.39 8.37 8.36 7.75 7.51 7.48 7.45 7.26 7.25 7.23 7.20 5.45 5.44 4.67 4.65 4.63 4.61 4.60 4.59 2.81 2.59 2.39 2.37 2.17 2.05 1.91 1.73 1.70 1.67 1.63 1.25 1.13 1.05 0.86 0.00
AcO
3l
2.03 1.04 2.28 1.08 1.04 1.14 1.56 3.64 5.57 3.37 4.70 10.09 2.58 3.06 0.66
10 9 8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

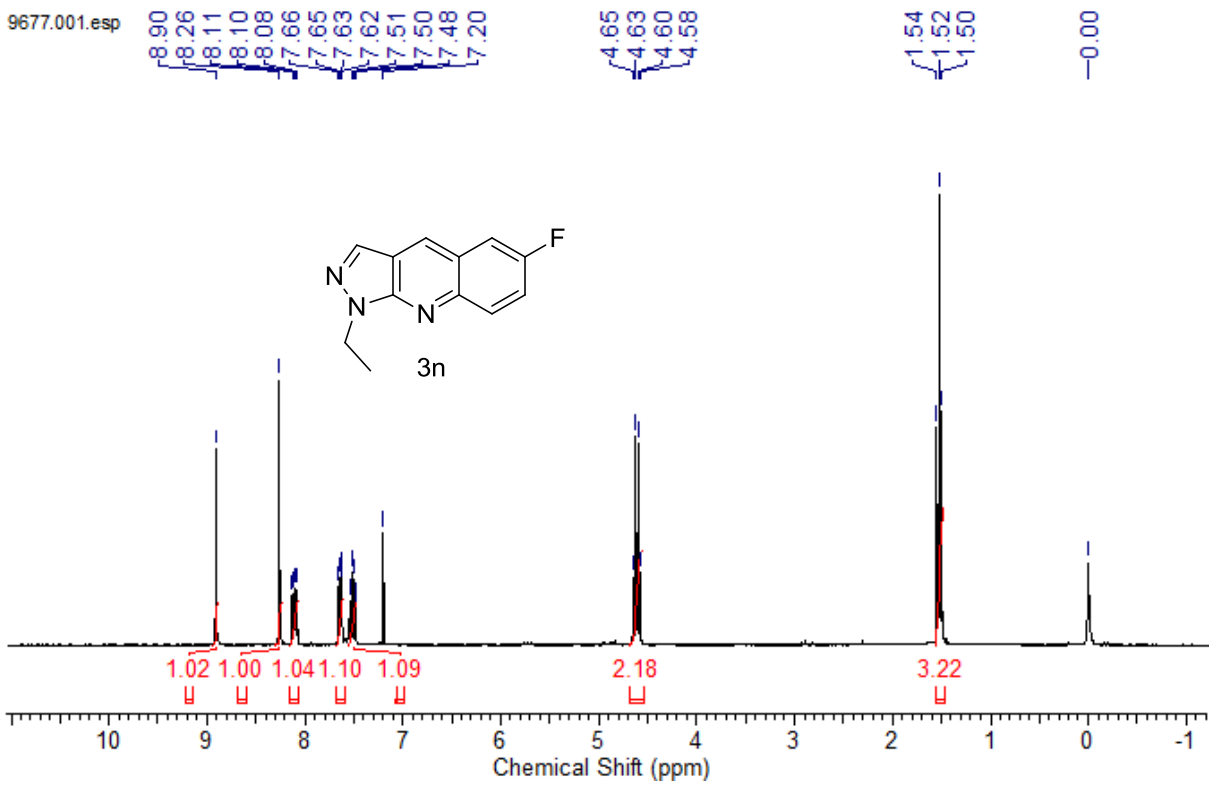

9233.001.esp
Chemical Shift (ppm)

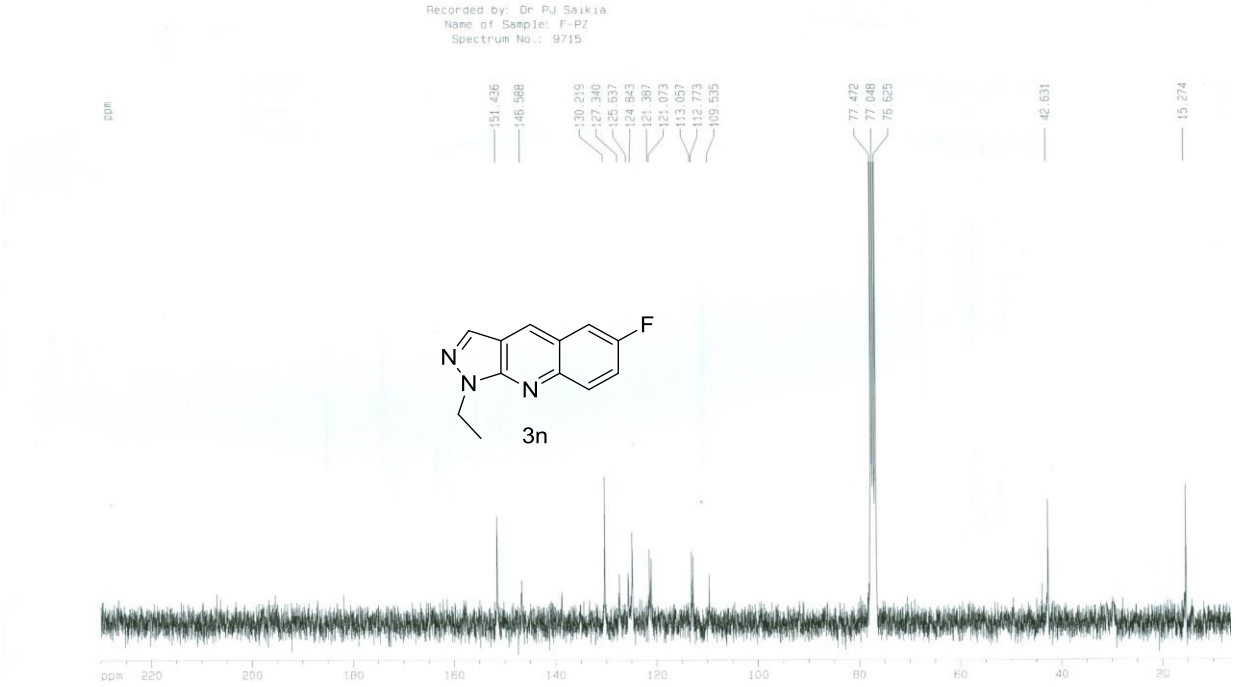

Recorded by: Dr PJ Saikia
Name of Sample: KS-D-Pz
Spectrum No.: 9305
ppm
174.165 170.568 150.687 142.205 140.180 130.164 128.849 124.824 124.693 122.030 120.274 115.527
77.463 77.040 76.617 73.847
56.242 50.554 45.540 38.166 36.923 33.677 31.360 31.005 30.010 27.750 21.459 20.573 19.386 17.491 12.432
AcO
3l
ppm 220 200 180 160 140 120 100 80 60 40 20

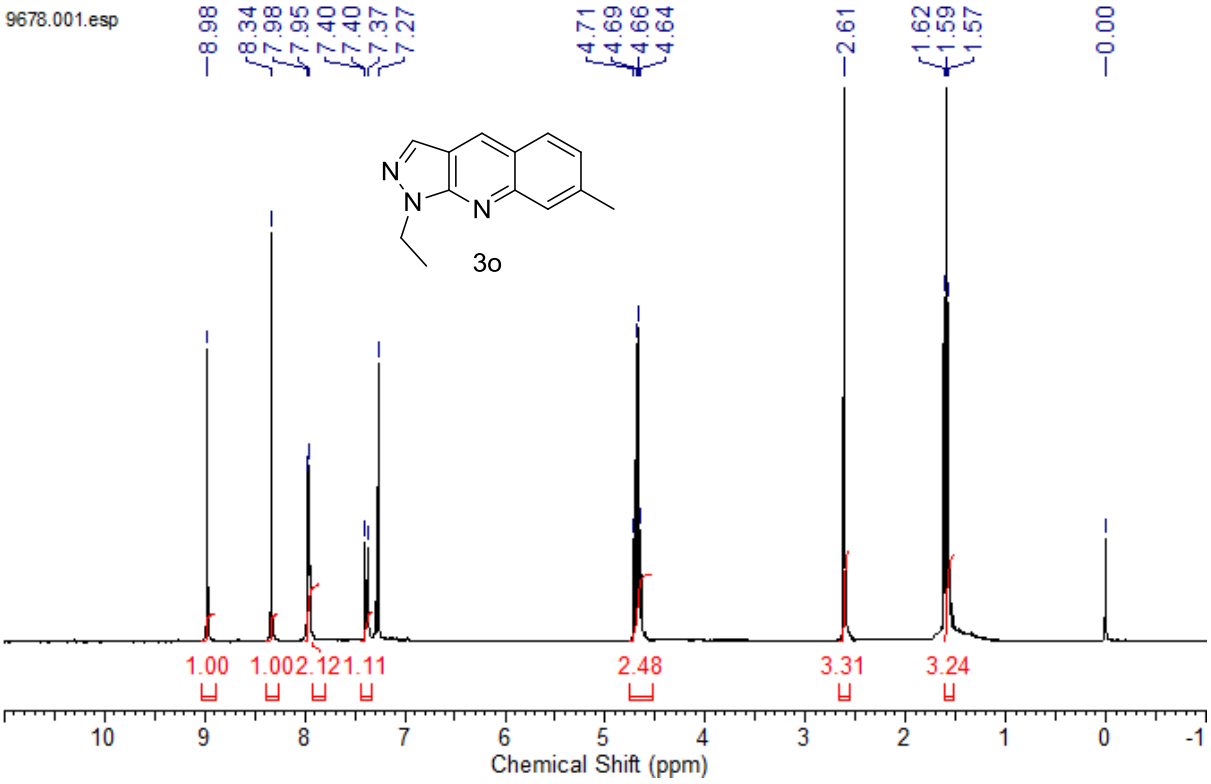

9337.001.esp
8.52 8.51 8.49 8.10 8.02 8.00 7.54 7.52 7.52 7.50 7.49 7.26 7.16 5.45 5.44 4.67 4.65 4.63 4.61 4.60 2.83 2.64 2.39 2.35 2.05 1.91 1.61 1.34 1.28 1.27 1.25 1.20 1.13 1.08 0.88 0.00
AcO
3m
2.17 1.12 2.14 5.29 2.75 1.00 1.18 8.44 3.62 3.76 6.01 6.40 3.48 3.38 2.46
10 9 8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

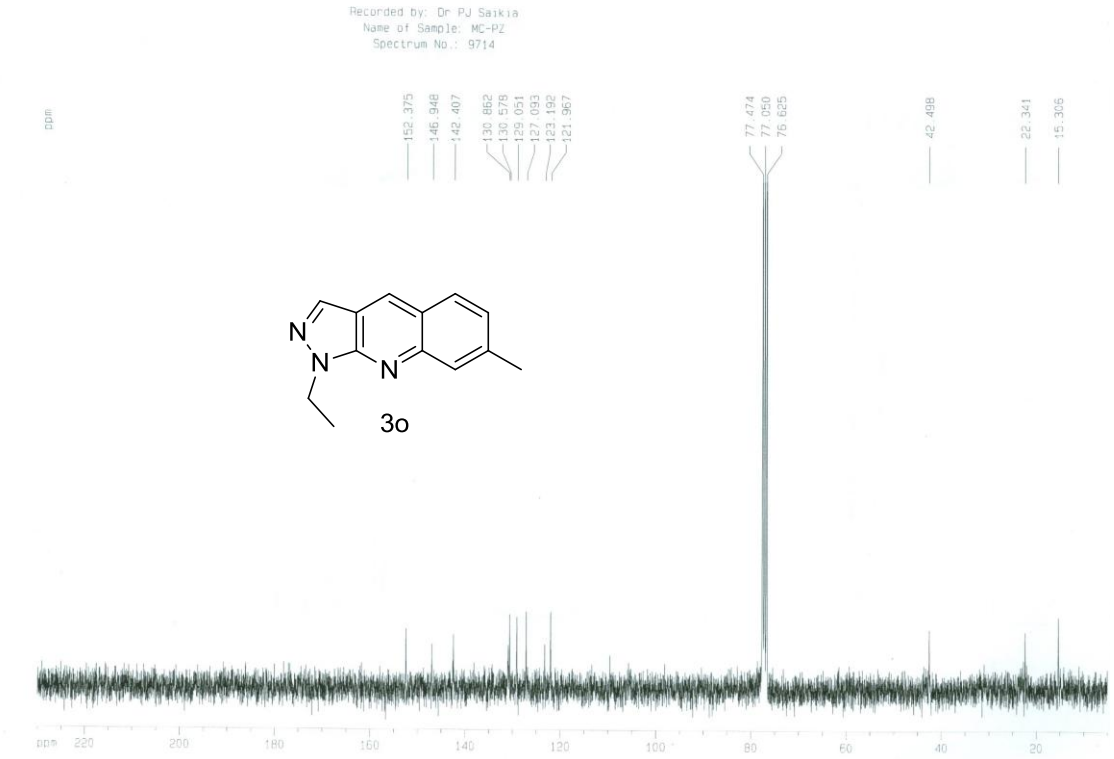

9337.001.esp
Chemical Shift (ppm)

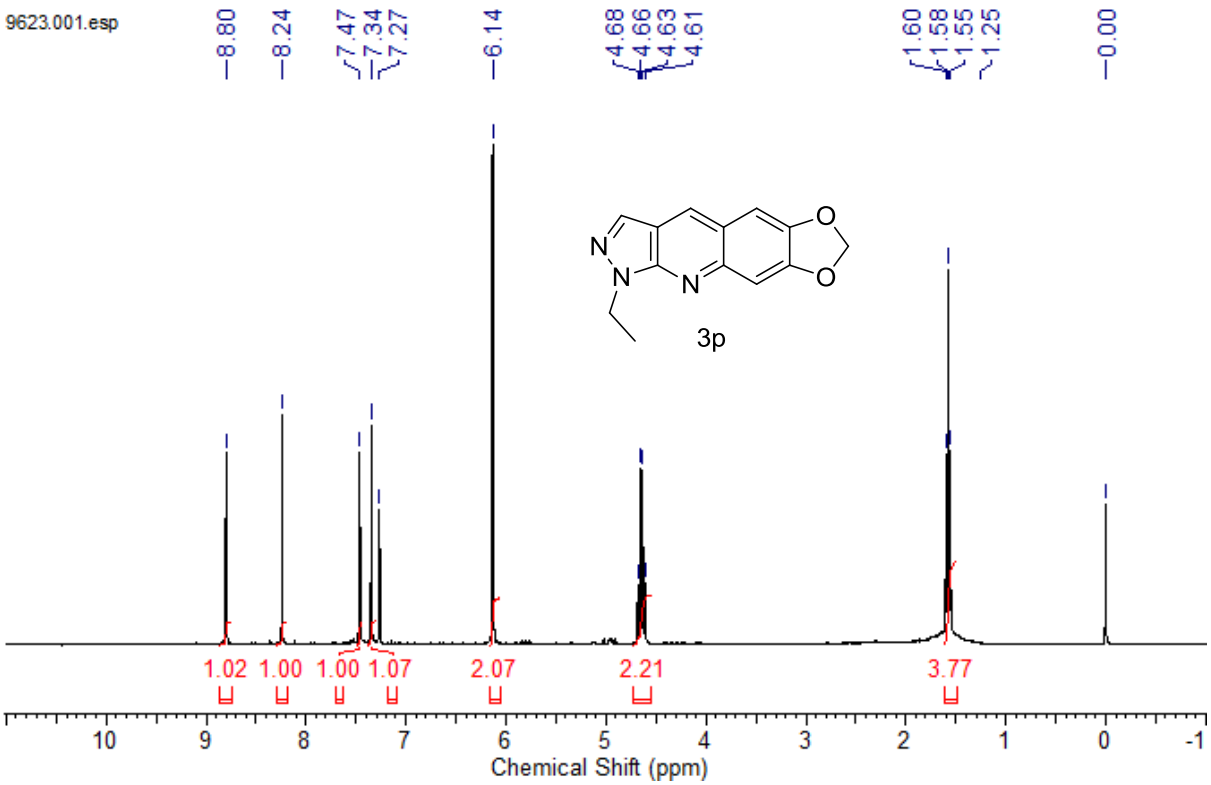

Recorded by: Dr PJ Saikia
Name of Sample: KS-DIPH-PZ
Spectrum No.: 9366
AcO
3m

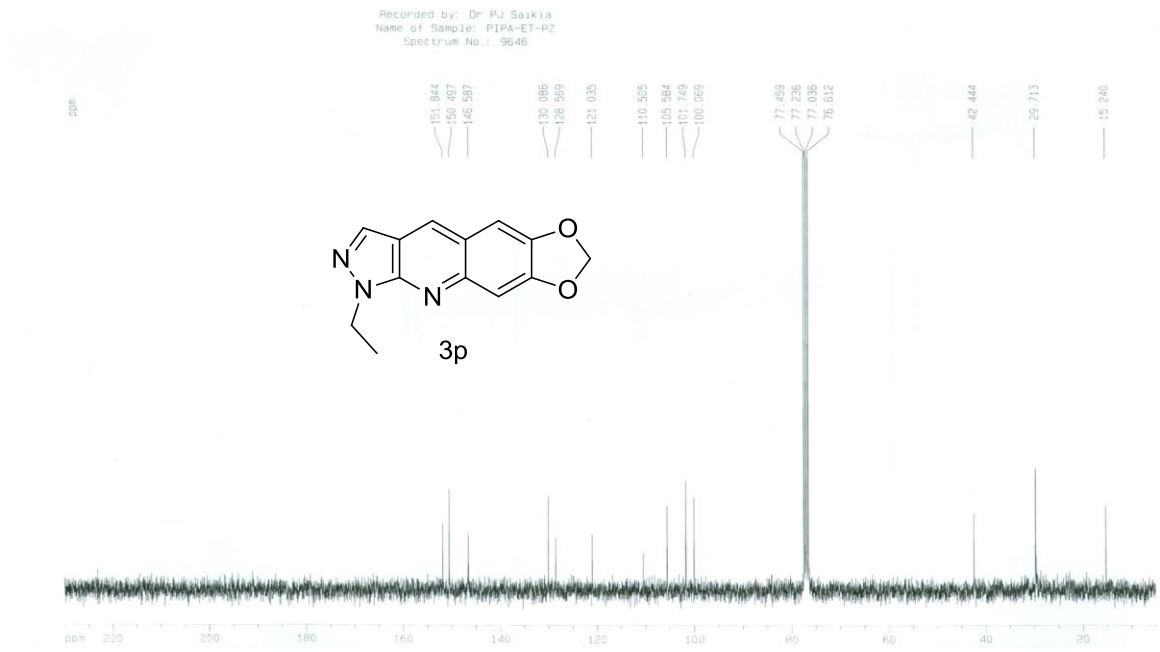

9677.001.esp
8.90
8.26
8.11
8.10
8.08
7.66
7.65
7.63
7.62
7.51
7.50
7.48
7.20
4.65
4.63
4.60
4.58
1.54
1.52
1.50
0.00
F
N
N
N
3n
1.02
1.00
1.04
1.10
1.09
2.18
3.22
10
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

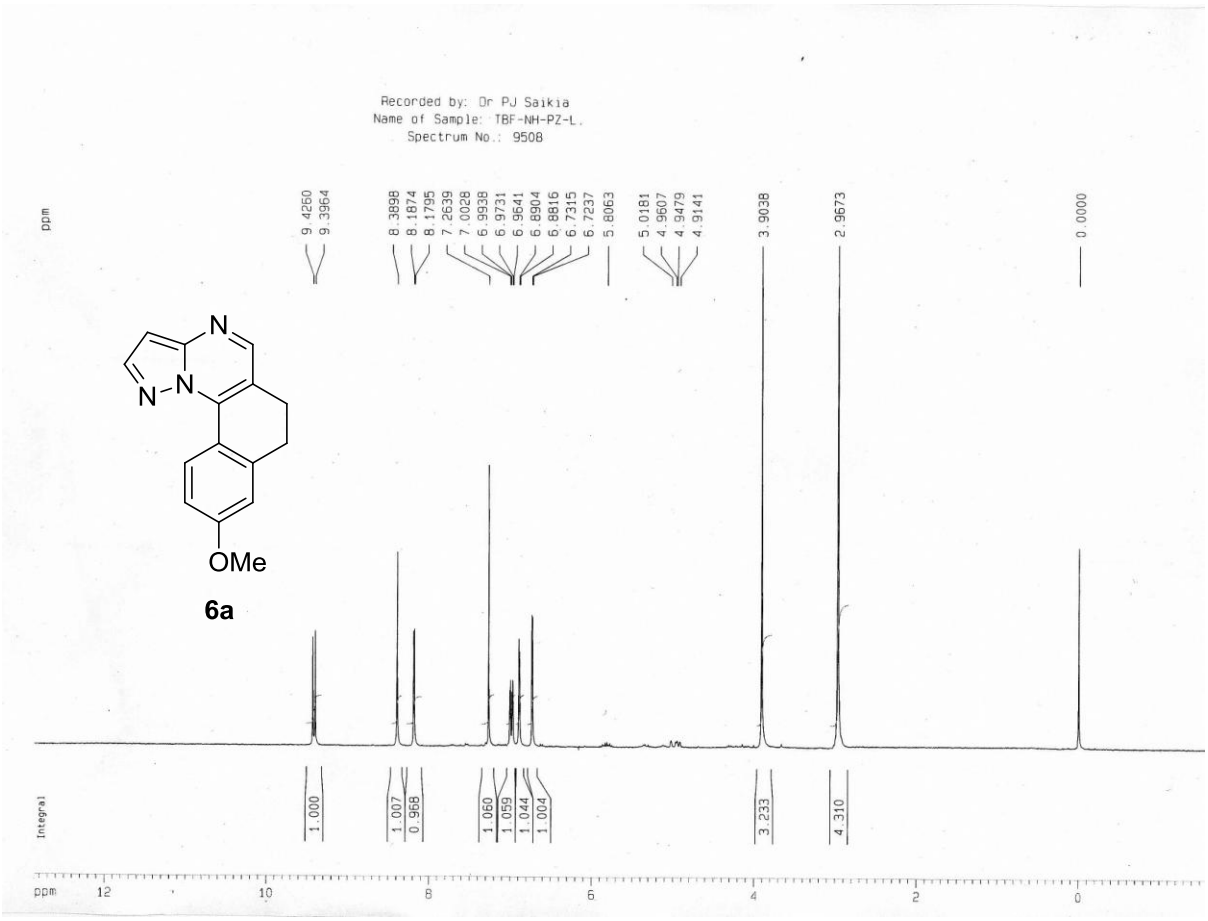

Recorded by: Dr PJ Saikia
Name of Sample: F-PZ
Spectrum No.: 9715
ppm
151.436
145.588
130.219
127.340
125.637
124.843
121.387
121.073
113.057
112.773
109.535
77.472
77.048
76.625
42.631
15.274
F
N
N
N
3n
ppm
220
200
180
160
140
120
100
80
60
40
20


9678.001.esp
8.98
8.34
7.98
7.95
7.40
7.40
7.37
7.27
4.71
4.69
4.66
4.64
2.61
1.62
1.59
1.57
0.00
3o
1.00
1.00
2.12
1.11
2.48
3.31
3.24
10
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

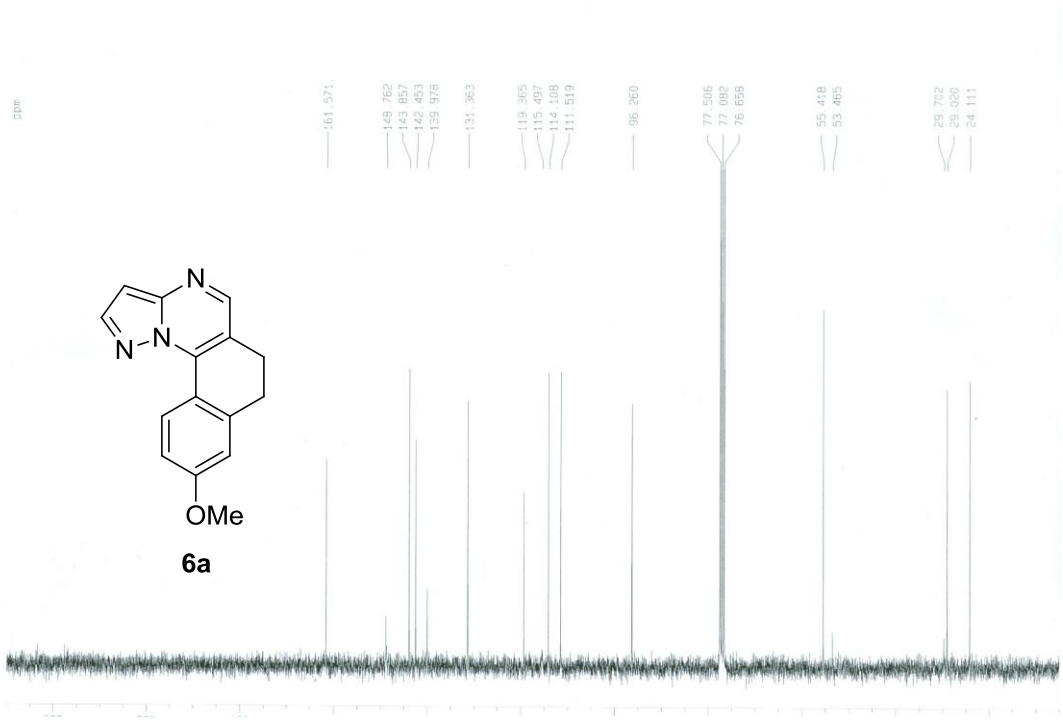

Recorded by: Dr PJ Saikia
Name of Sample: MC-PZ
Spectrum No.: 9714
ppm
152.375
146.948
142.407
130.862
130.578
129.051
127.093
123.192
121.967
77.474
77.050
76.625
42.498
22.341
15.306
3o
ppm
220
200
180
160
140
120
100
80
60
40
20


9623.001.esp
8.80
8.24
7.47
7.34
7.27
6.14
4.68
4.66
4.63
4.61
1.60
1.58
1.55
1.25
0.00
3p
1.02
1.00
1.00
1.07
2.07
2.21
3.77
Chemical Shift (ppm)

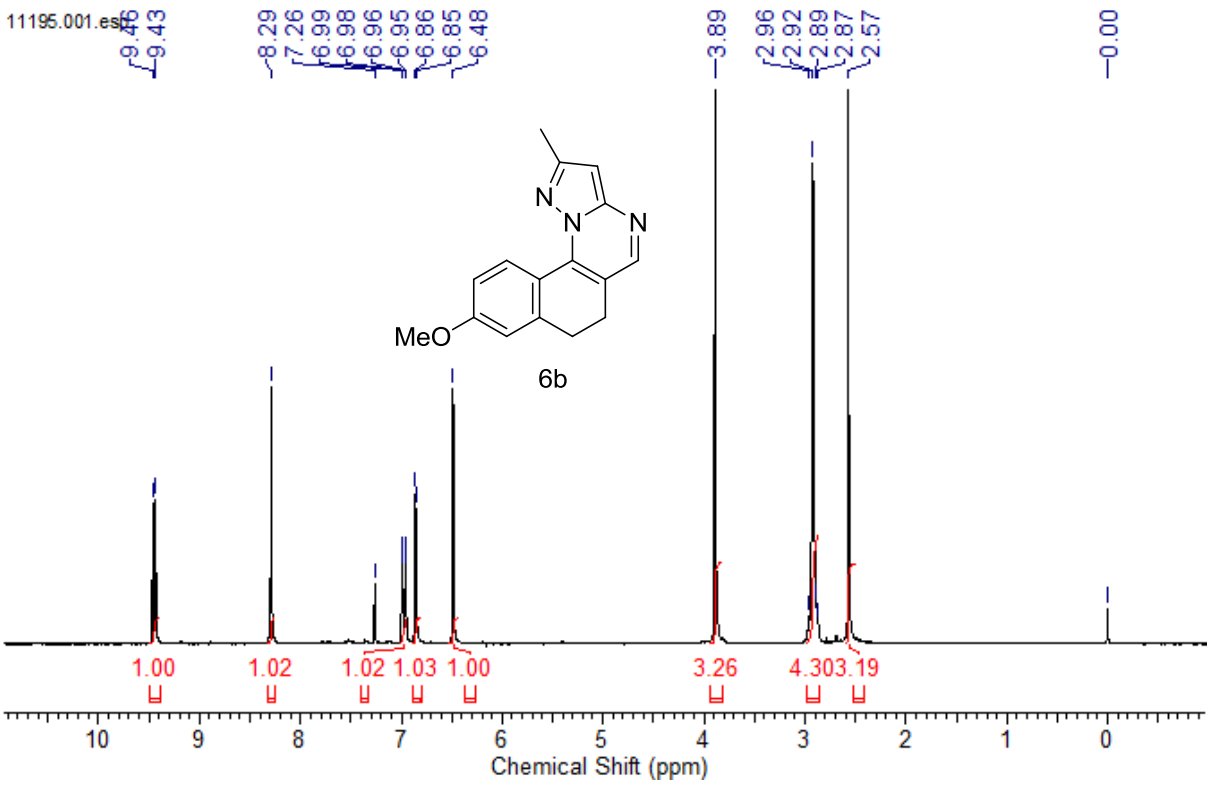

Recorded by: Dr PJ Saikia
Name of Sample: PIPA-ET-PZ
Spectrum No.: 9646
3p

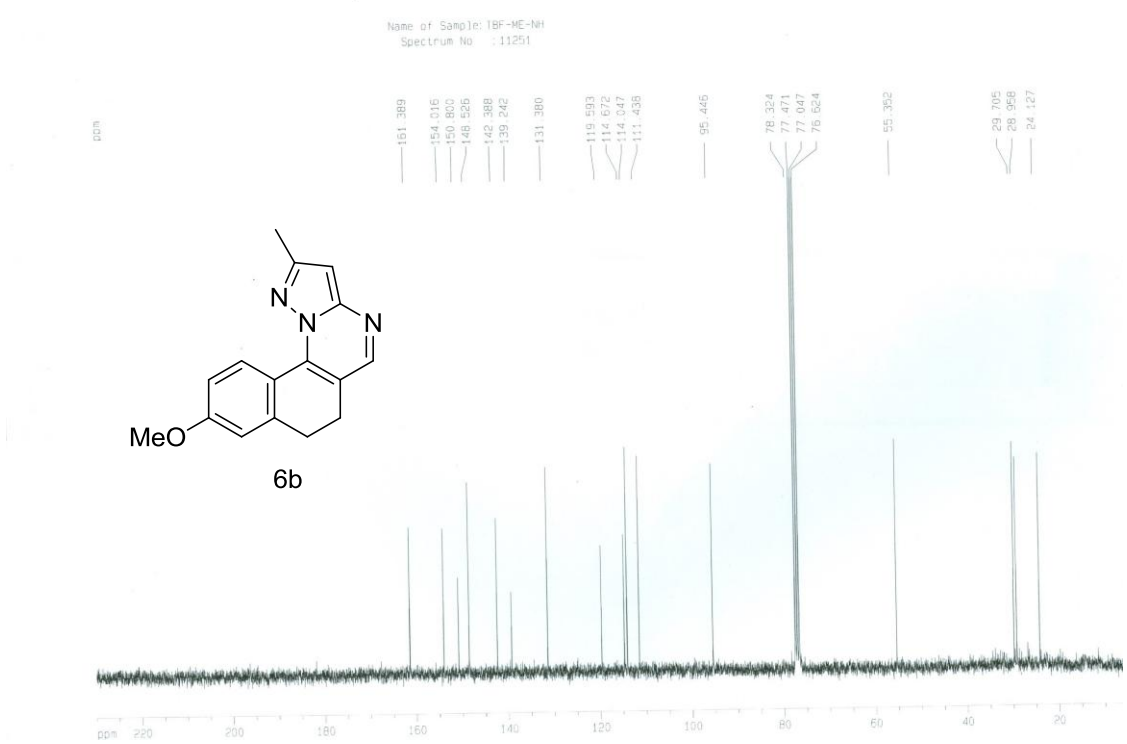

Recorded by: Dr PJ Saikia
Name of Sample: TBF-NH-PZ-L
Spectrum No.: 9508
ppm
9.4260
9.3964
8.3898
8.1874
8.1795
7.2639
7.0028
6.9938
6.9731
6.9641
6.8904
6.8816
6.7315
6.7237
5.8063
5.0181
4.9607
4.9479
4.9141
3.9038
2.9673
0.0000
Integral
1.000
1.007
0.968
1.060
1.059
1.044
1.004
3.233
4.310
ppm
12
10
8
6
4
2
0
N
N
N
OMe

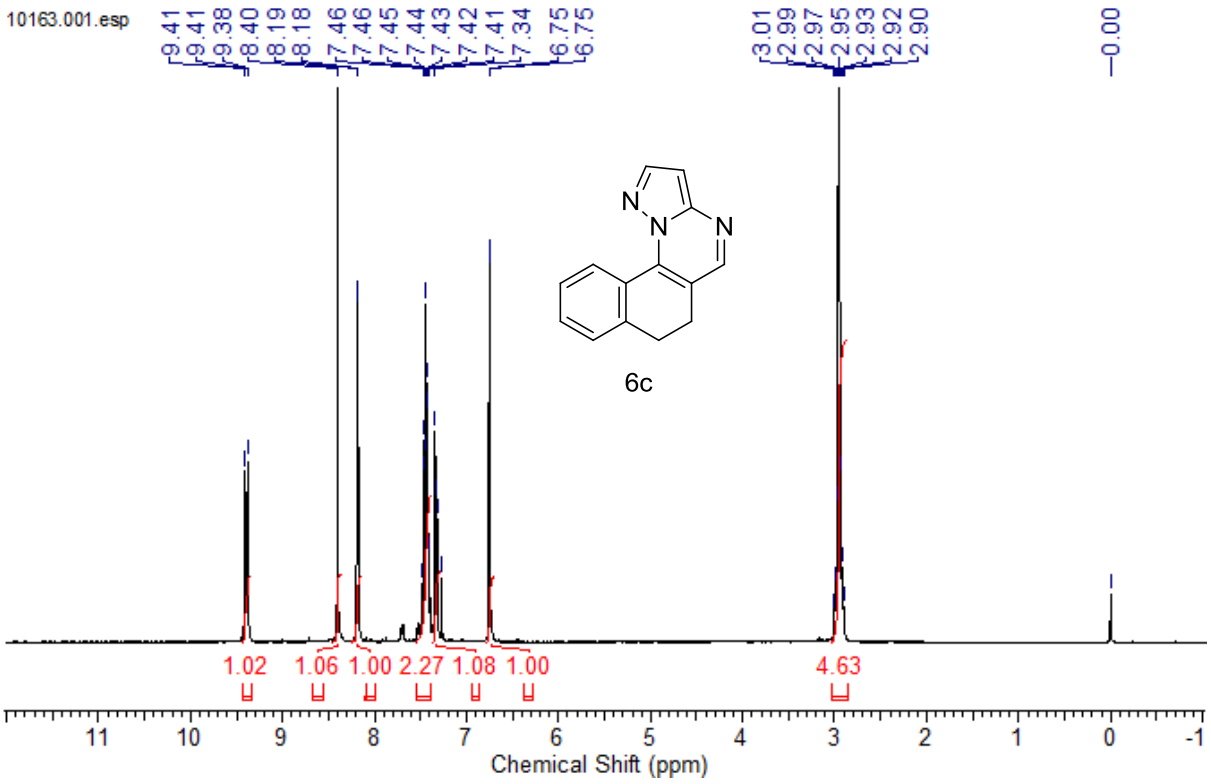
6a


ppm
161.571
148.762
143.857
142.453
139.976
131.363
119.365
115.497
114.108
111.519
96.260
77.506
77.082
76.658
55.418
53.465
29.702
29.020
24.111
N
N
N
OMe

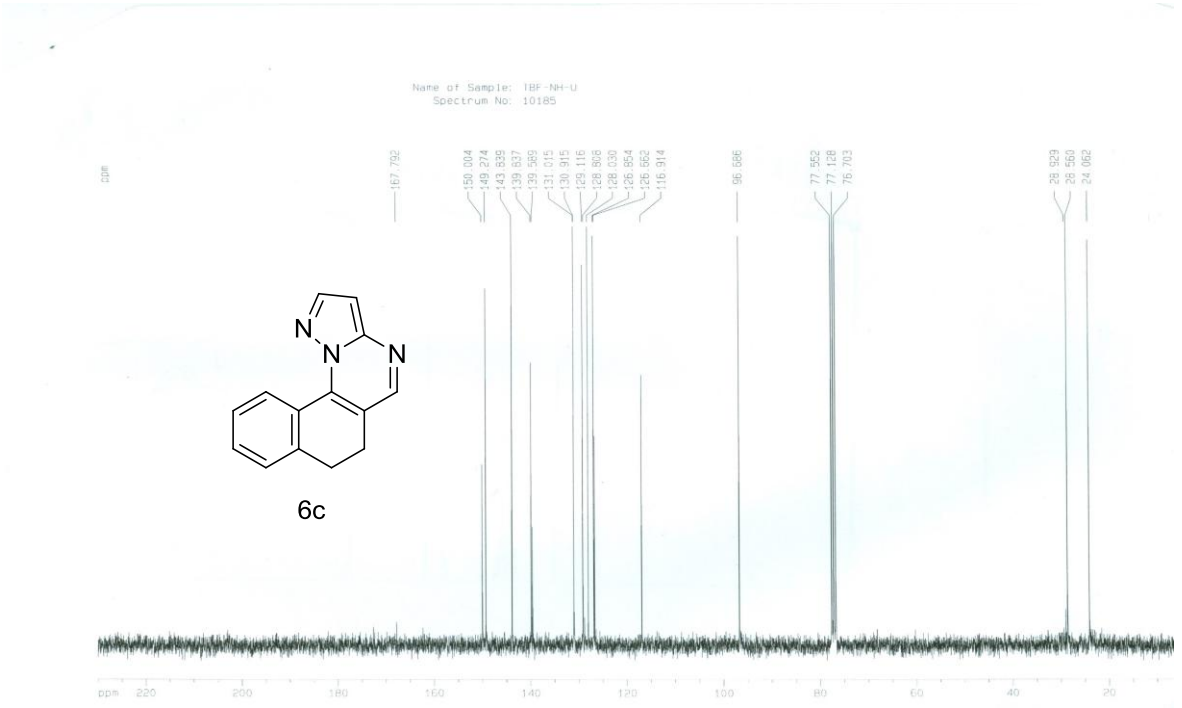
6a


11195.001.esp
9.46
9.43
8.29
7.26
6.99
6.98
6.96
6.95
6.86
6.85
6.48
3.89
2.96
2.92
2.89
2.87
2.57
0.00
MeO
6b
1.00
1.02
1.02
1.03
1.00
3.26
4.30
3.19
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

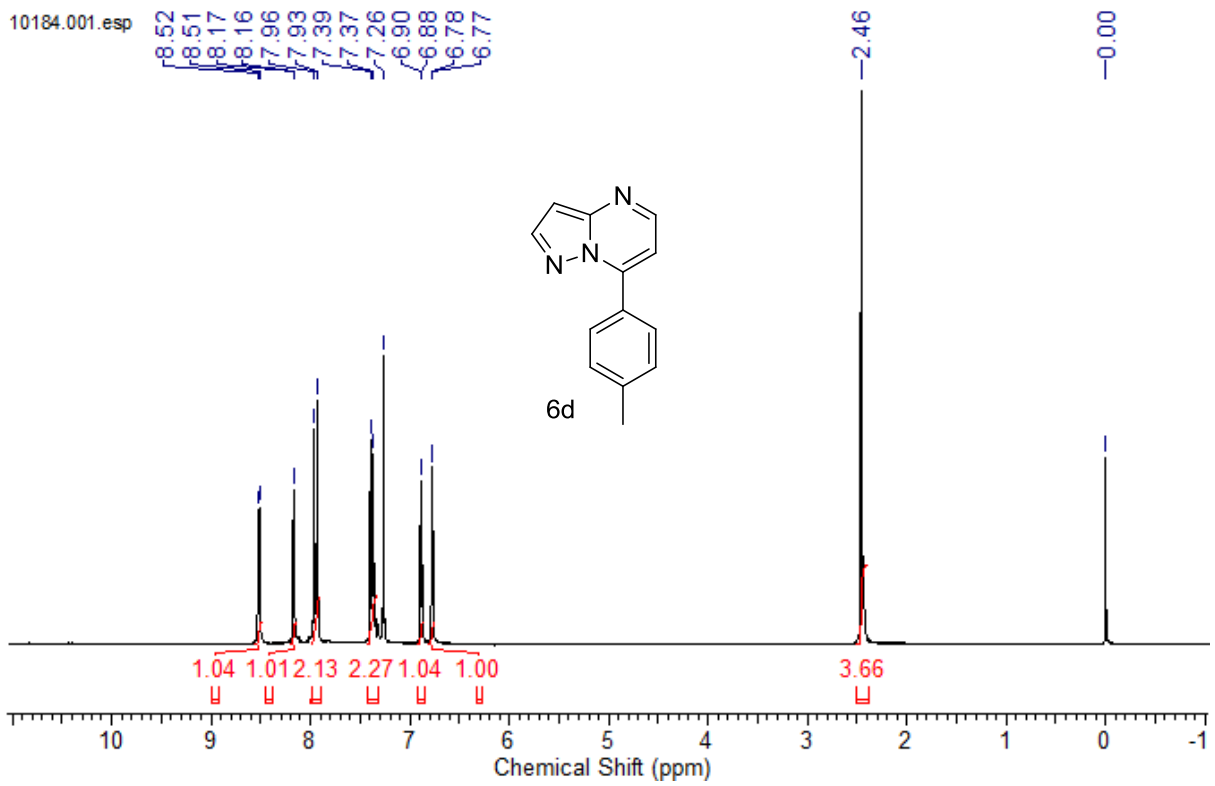

Name of Sample: TBF-ME-NH
Spectrum No. :11251
ppm
161.389
154.016
150.800
148.526
142.388
139.242
131.380
119.593
114.672
114.047
111.438
95.446
78.324
77.471
77.047
76.624
55.352
29.705
28.958
24.127
MeO
6b
ppm
220
200
180
160
140
120
100
80
60
40
20

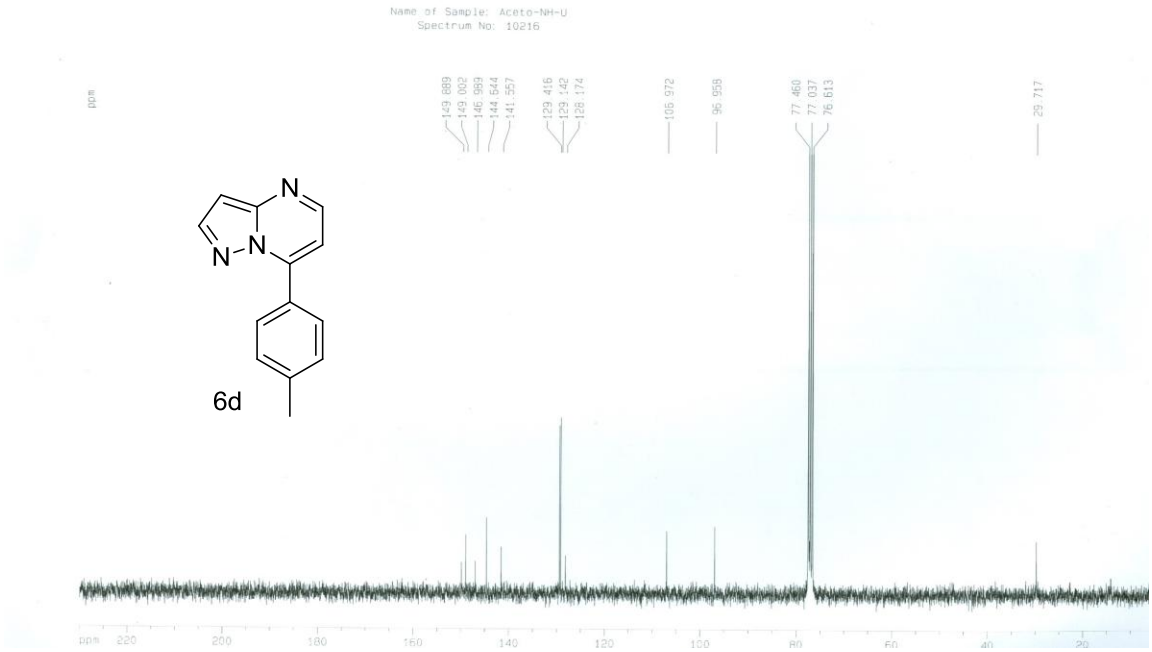

10163.001.esp
9.41
9.41
9.38
8.40
8.19
8.18
7.46
7.46
7.45
7.44
7.43
7.42
7.41
7.34
6.75
6.75
3.01
2.99
2.97
2.95
2.93
2.92
2.90
0.00
1.02
1.06
1.00
2.27
1.08
1.00
4.63
11
10
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

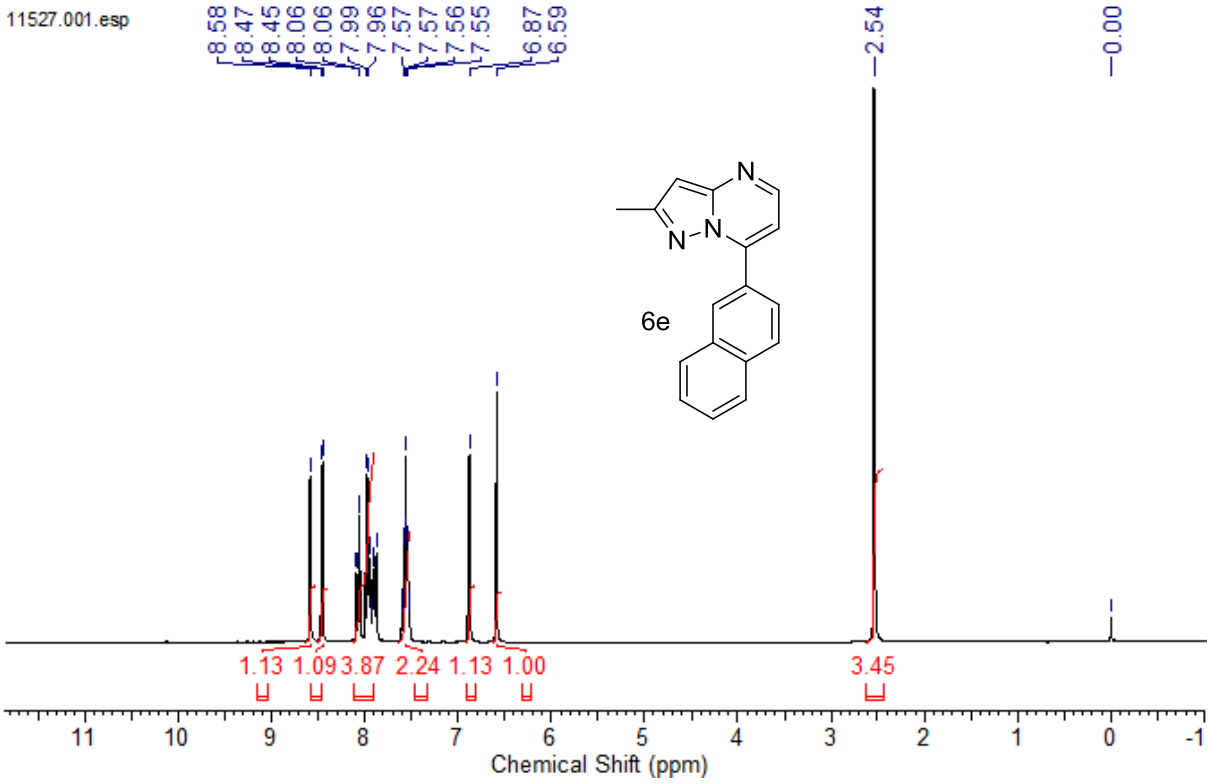
6c

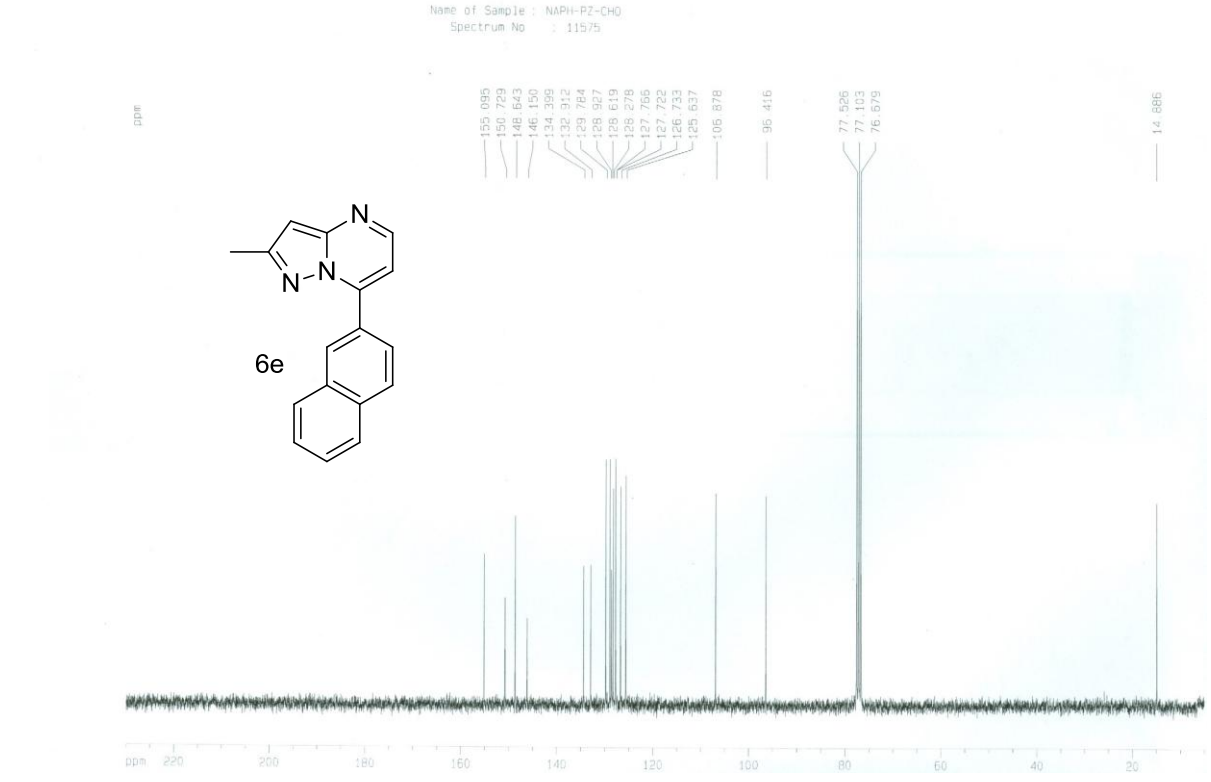

Name of Sample: TBF-NH-U
Spectrum No: 10185
ppm
167.792
150.004
149.274
143.839
139.837
139.589
131.015
130.915
129.116
128.806
128.030
126.854
126.662
116.914
96.666
77.552
77.128
76.703
28.929
28.560
24.062
220
200
180
160
140
120
100
80
60
40
20

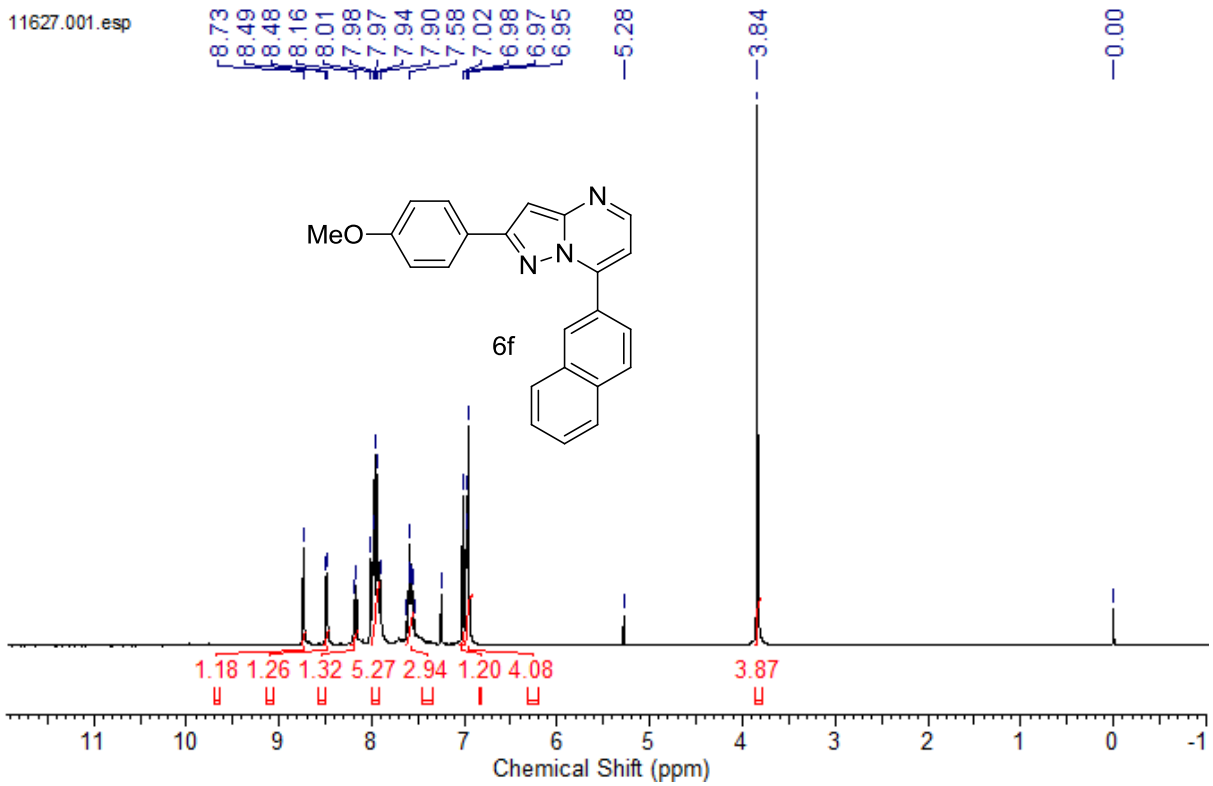
6c

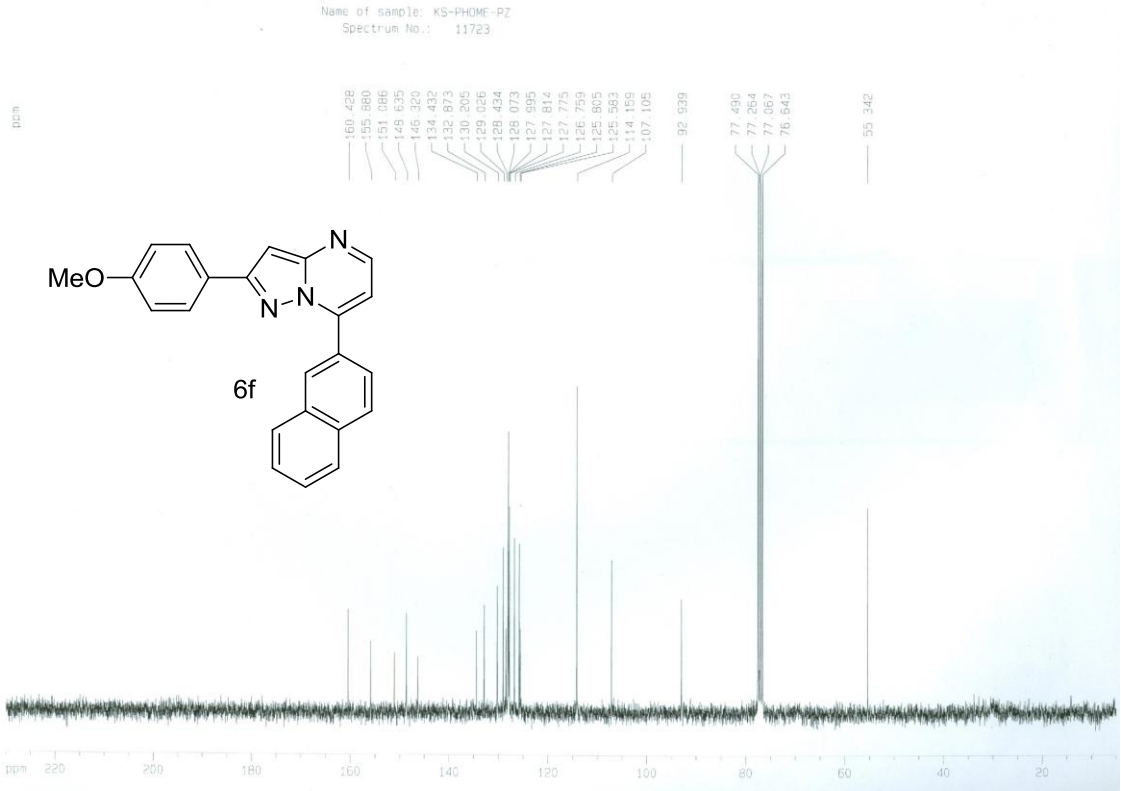

10184.001.esp
8.52
8.51
8.17
8.16
7.96
7.93
7.39
7.37
7.26
6.90
6.88
6.78
6.77
2.46
0.00
6d
1.04 1.01 2.13 2.27 1.04 1.00
3.66
10 9 8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

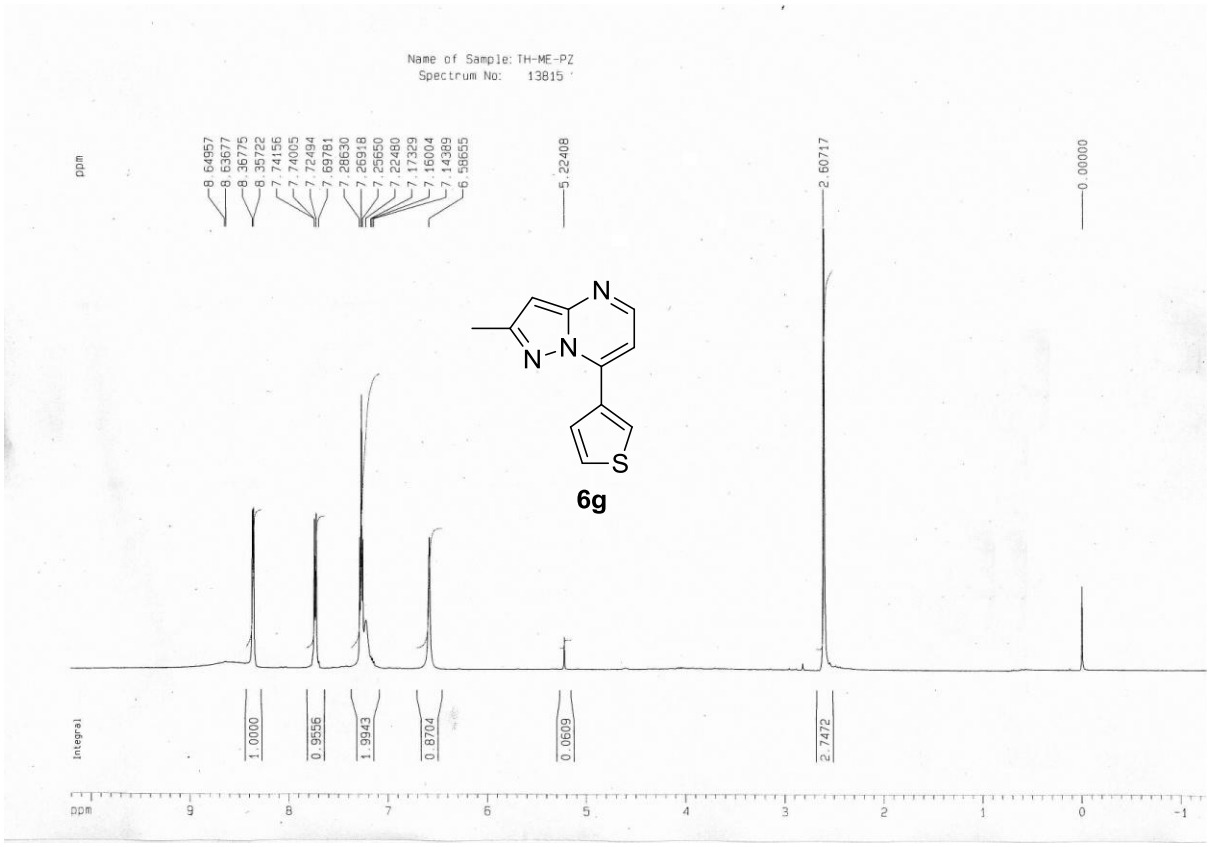

Name of Sample: Aceto-NH-U
Spectrum No: 10216
ppm
149.889
149.002
146.999
144.544
141.557
129.416
129.142
128.174
106.972
96.959
77.460
77.037
76.613
29.717
6d
ppm 220 200 180 160 140 120 100 80 60 40 20


11527.001.esp
8.58
8.47
8.45
8.06
8.06
7.99
7.96
7.57
7.57
7.56
7.55
6.87
6.59
2.54
0.00
6e
1.13 1.09 3.87 2.24 1.13 1.00
3.45
11 10 9 8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

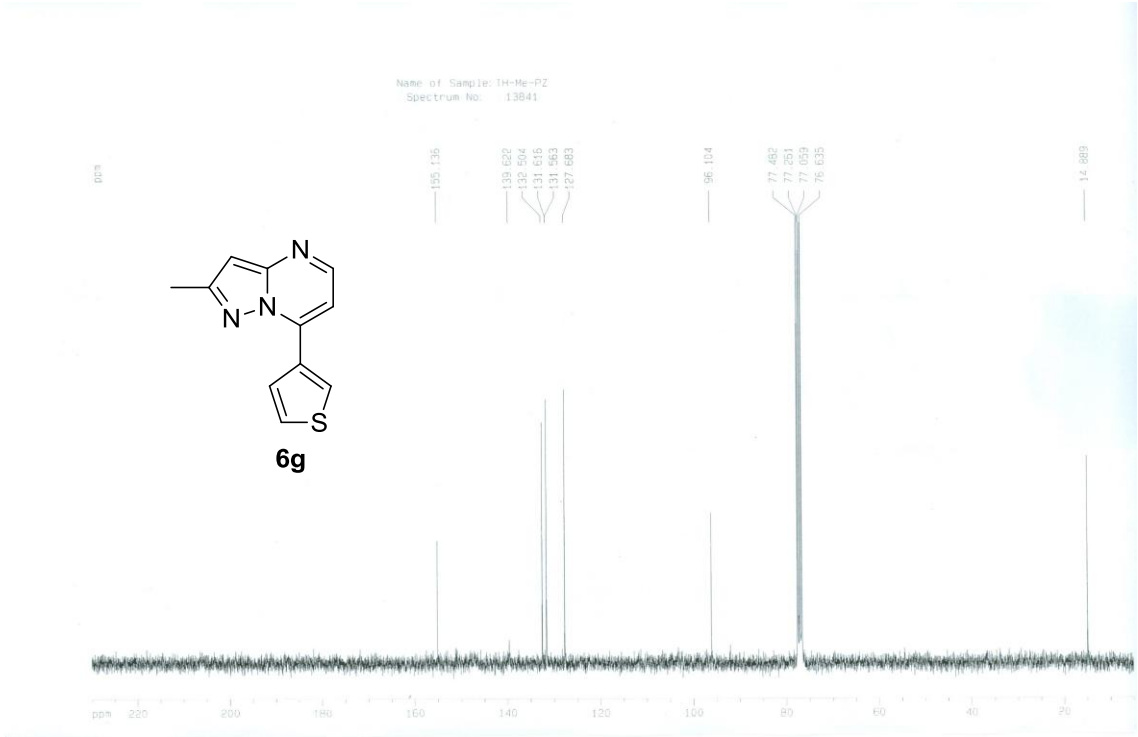

Name of Sample : NAPH-PZ-CHO
Spectrum No : 11575
ppm
155.095
150.729
148.643
146.150
134.399
132.912
129.784
128.927
128.619
128.278
127.766
127.722
126.733
125.637
106.878
96.416
77.526
77.103
76.679
14.886
6e
ppm 220 200 180 160 140 120 100 80 60 40 20


11627.001.esp
8.73
8.49
8.48
8.16
8.01
7.98
7.97
7.94
7.90
7.58
7.02
6.98
6.97
6.95
5.28
3.84
0.00
MeO
N
N
N
6f
1.18 1.26 1.32 5.27 2.94 1.20 4.08
3.87
11 10 9 8 7 6 5 4 3 2 1 0 -1
Chemical Shift (ppm)

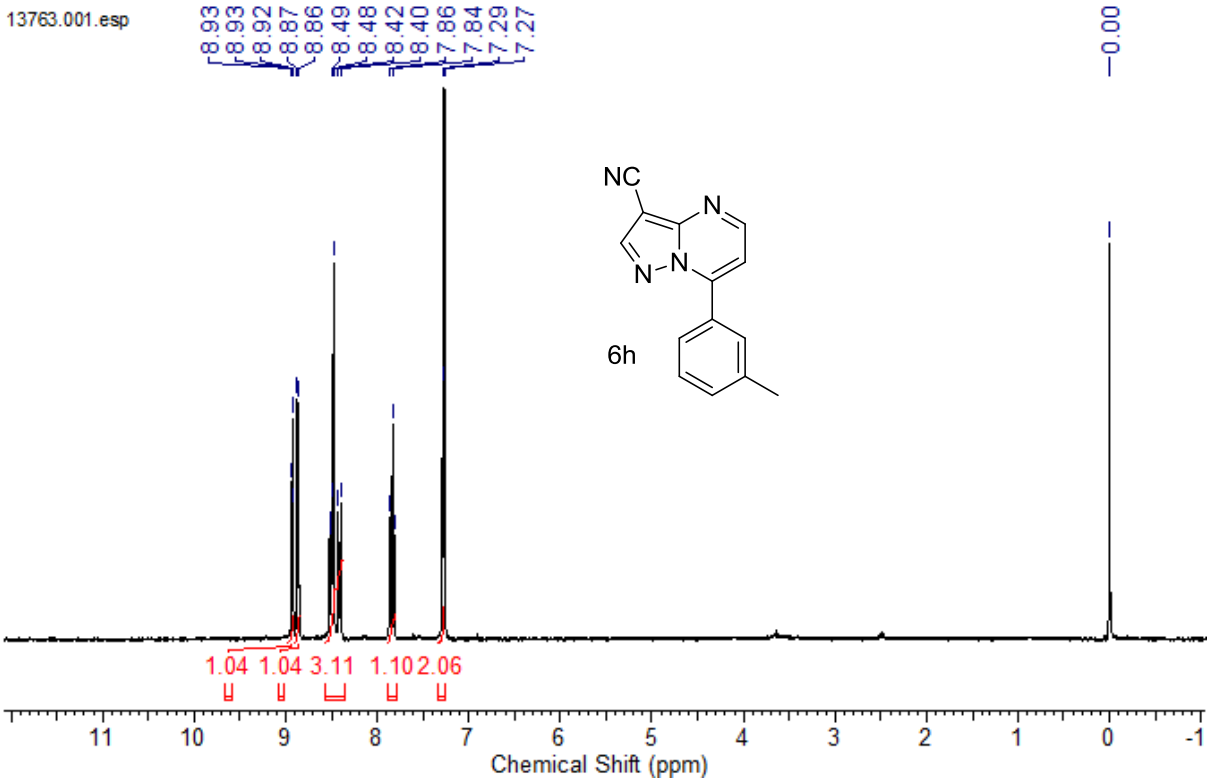

Name of sample: KS-PHOME-PZ
Spectrum No.: 11723
ppm
160.428
155.880
151.086
148.635
146.320
134.432
132.873
130.205
129.026
128.434
128.073
127.995
127.814
127.775
126.759
125.805
125.563
114.159
107.105
92.939
77.490
77.264
77.067
76.643
55.342
MeO
N
N
N
6f
ppm 220 200 180 160 140 120 100 80 60 40 20

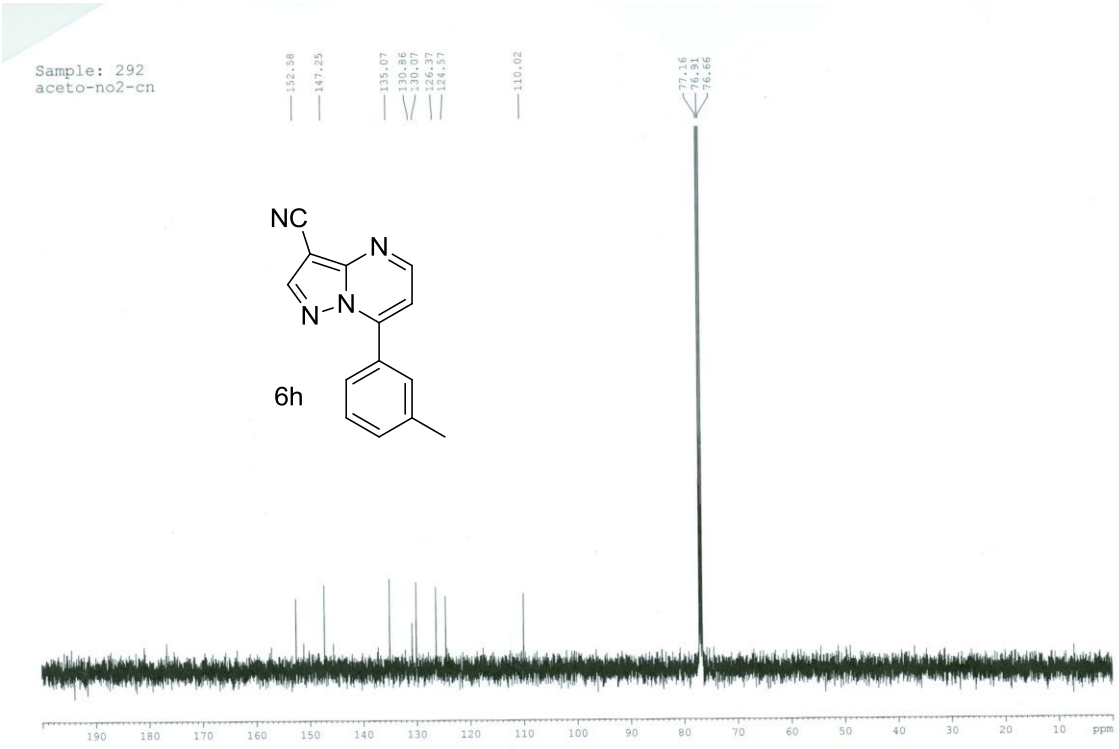

Name of Sample: TH-ME-PZ
Spectrum No: 13815
ppm
8.64957
8.63677
8.36775
8.35722
7.74156
7.74005
7.72494
7.69781
7.28630
7.26918
7.25650
7.22480
7.17329
7.16004
7.14389
6.59655
5.22408
2.60717
0.00000
Integral
1.0000
0.9556
1.9943
0.8704
0.0609
2.7472
ppm
9
8
7
6
5
4
3
2
1
0
-1

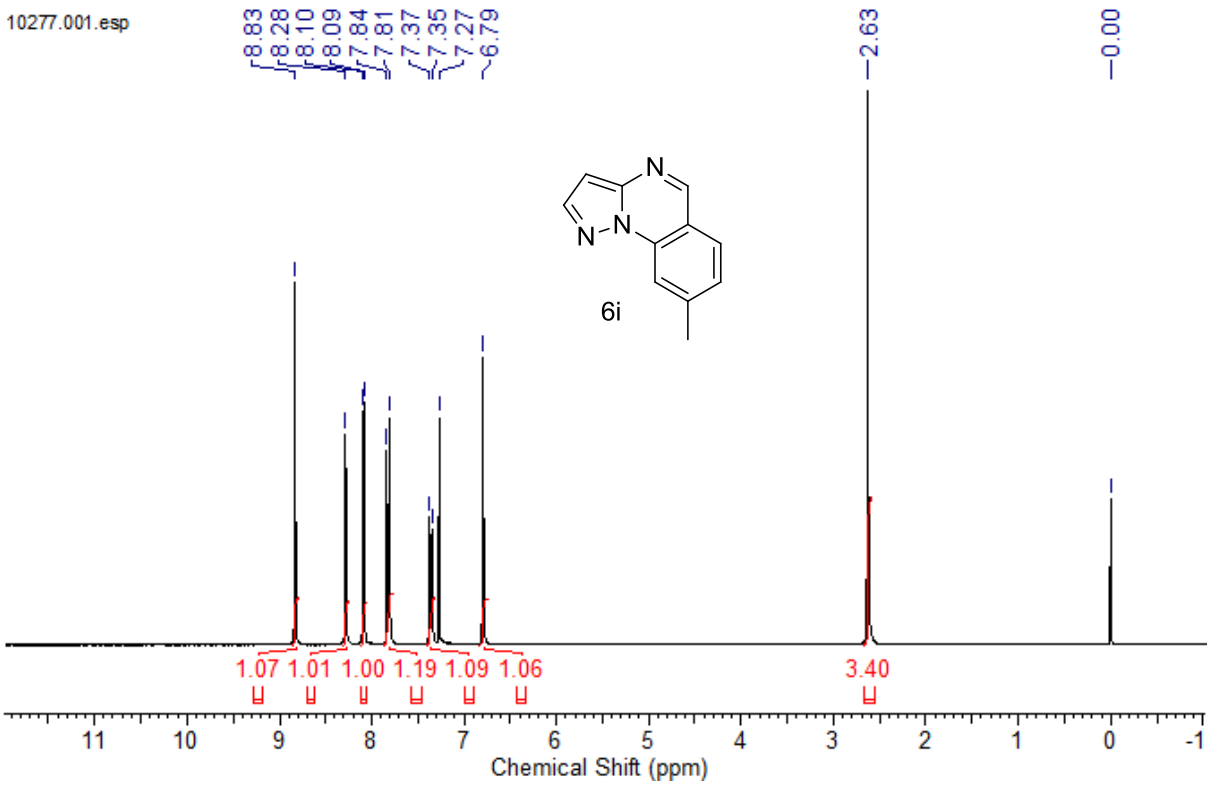
6g

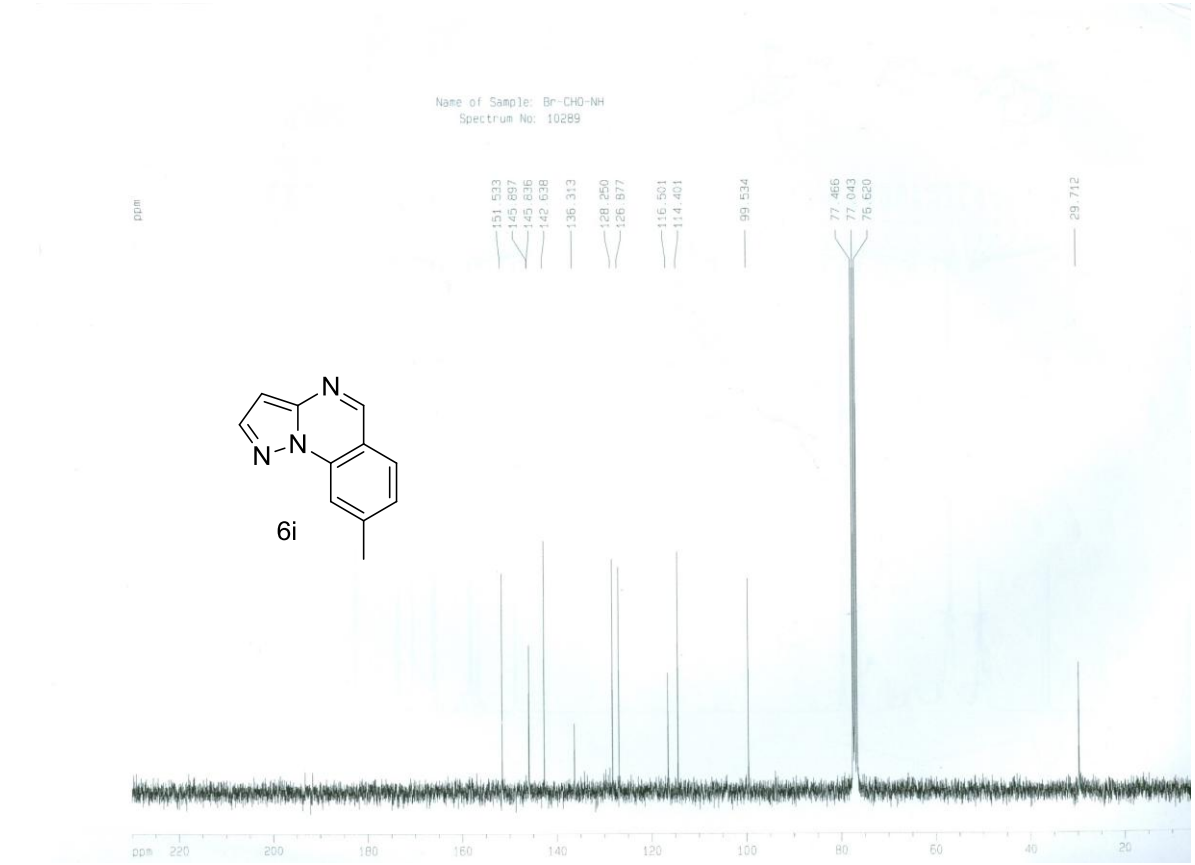

Name of Sample: TH-Me-PZ
Spectrum No: 13841
ppm
155.136
139.622
132.594
131.616
131.563
127.683
96.104
77.482
77.261
77.059
76.635
14.689
ppm
220
200
180
160
140
120
100
80
60
40
20

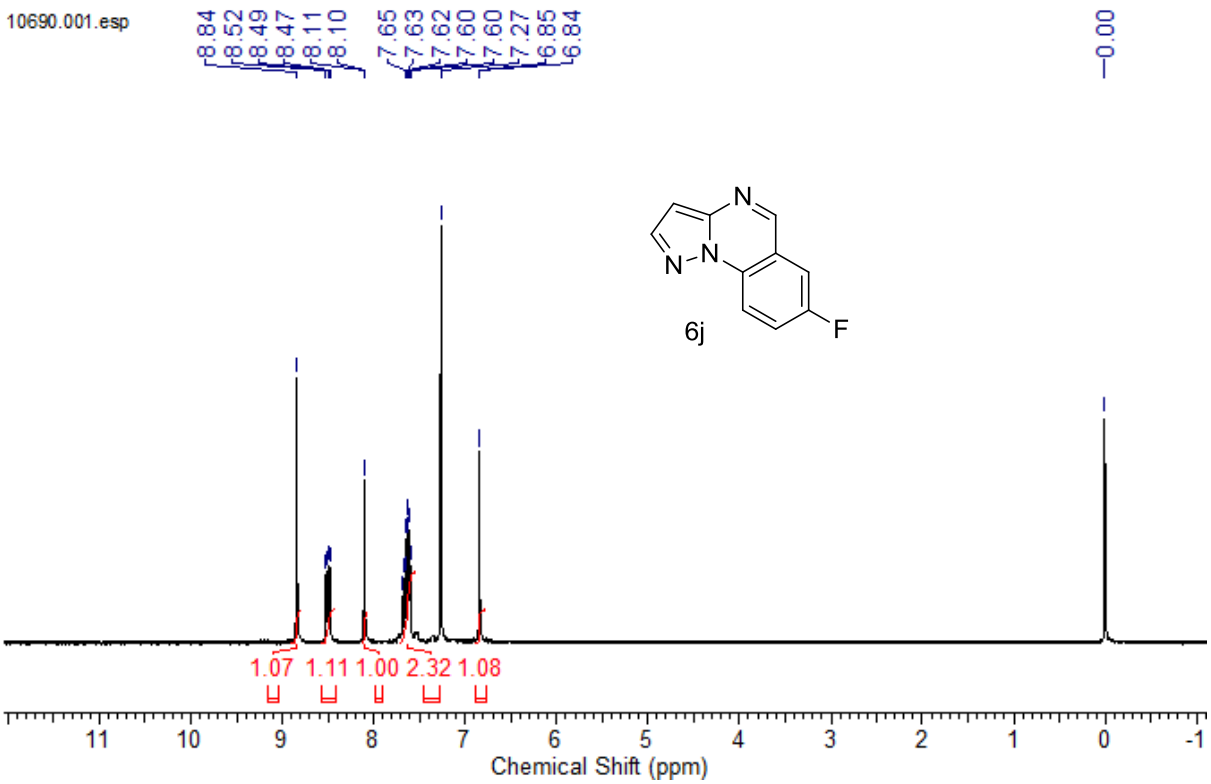
6g

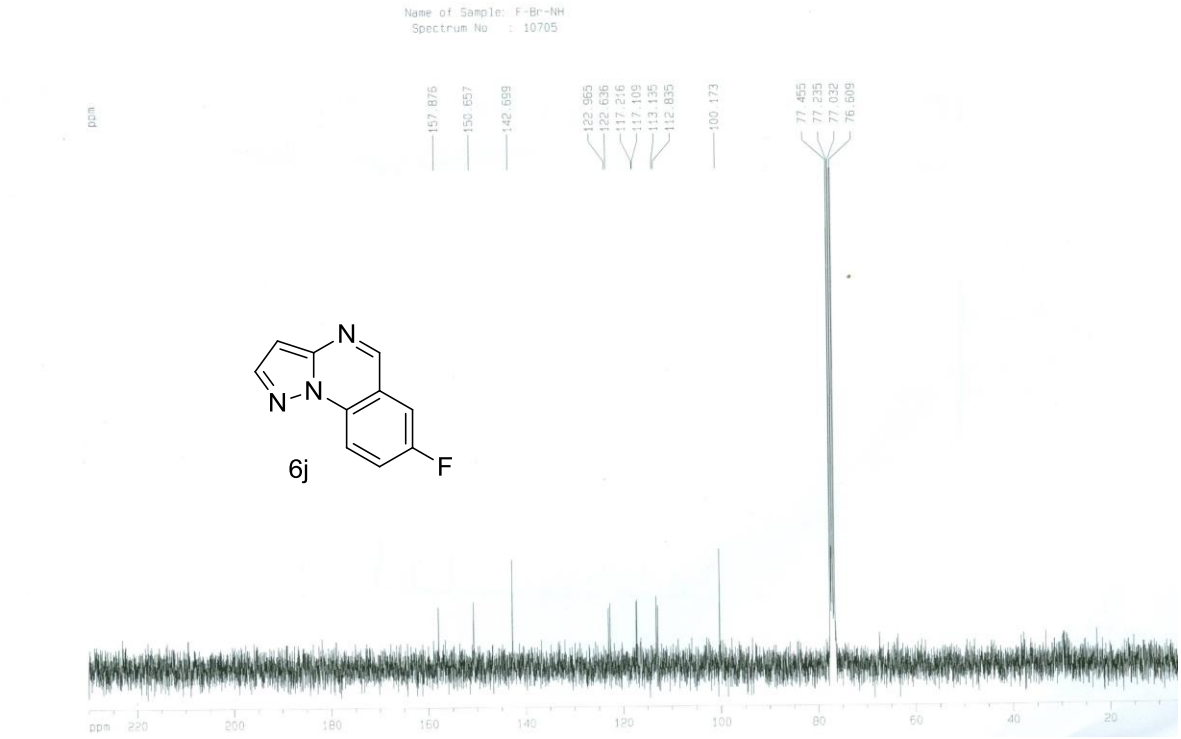

13763.001.esp
8.93
8.93
8.92
8.87
8.86
8.49
8.48
8.42
8.40
7.86
7.84
7.29
7.27
0.00
NC
N
N
N
6h
1.04
1.04
3.11
1.10
2.06
11
10
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)

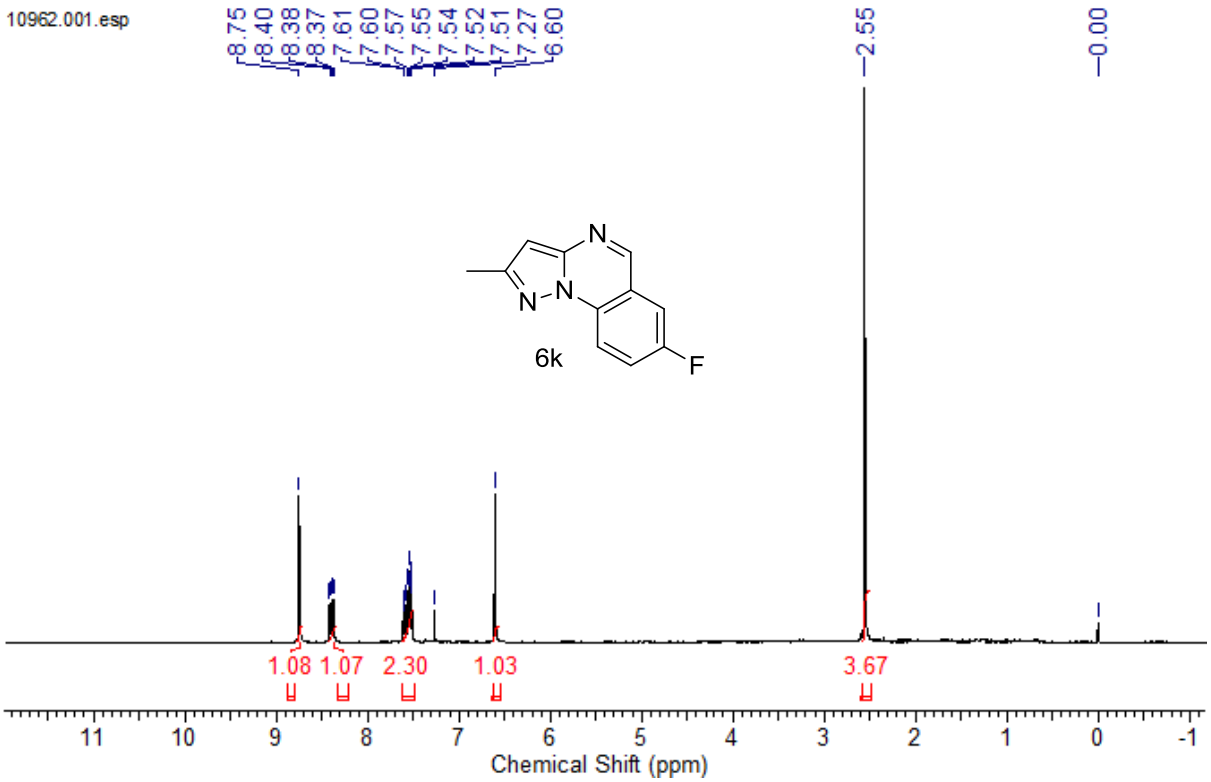

Sample: 292
aceto-no2-cn
152.58
147.25
135.07
130.86
130.07
126.37
124.57
110.02
77.16
76.91
76.66
NC
N
N
N
6h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

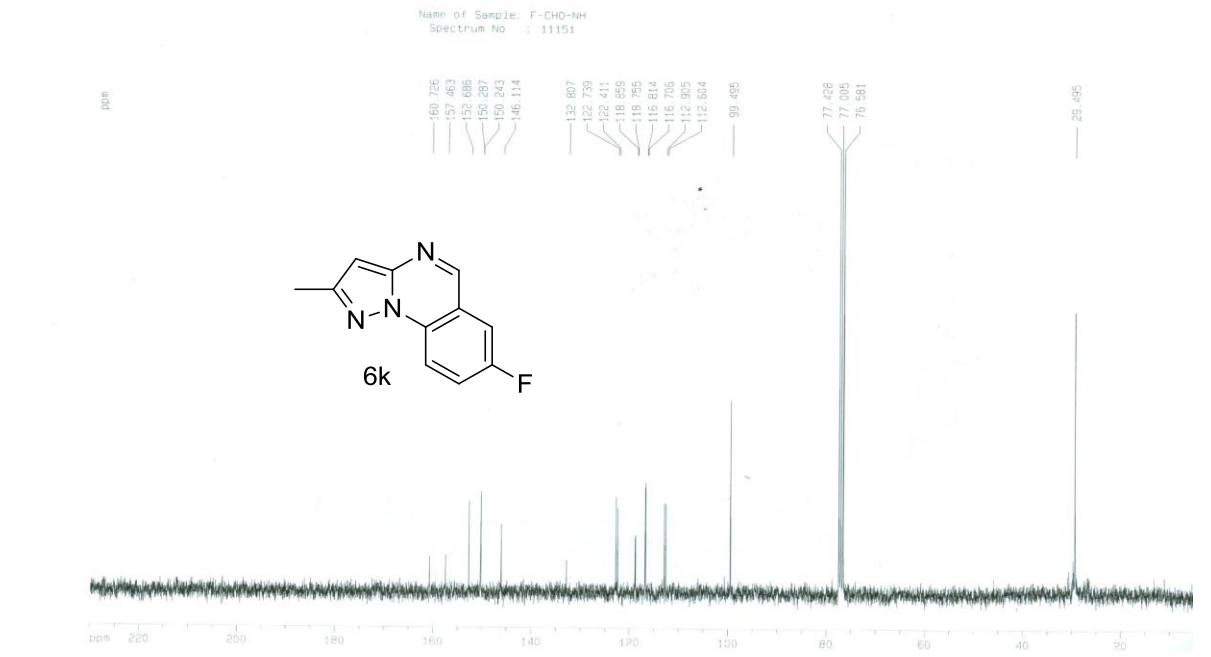

10277.001.esp
8.83
8.28
8.10
8.09
7.84
7.81
7.37
7.35
7.27
6.79
2.63
0.00
6i
1.07 1.01 1.00 1.19 1.09 1.06
3.40
Chemical Shift (ppm)


Name of Sample: Br-CHO-NH
Spectrum No: 10289
ppm
151.533
145.897
145.636
142.638
136.313
128.250
126.877
116.501
114.401
99.534
77.466
77.043
76.620
29.712
6i

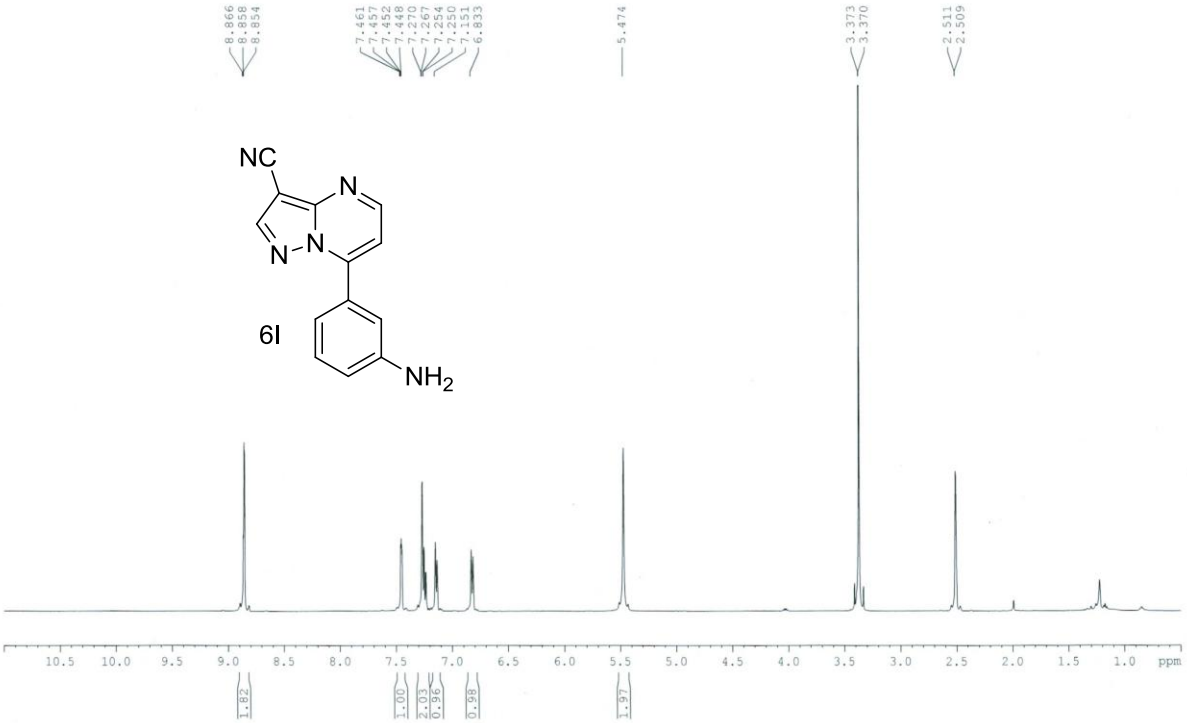

10690.001.esp
8.84
8.52
8.49
8.47
8.11
8.10
7.65
7.63
7.62
7.60
7.60
7.27
6.85
6.84
0.00
6j
F
1.07
1.11
1.00
2.32
1.08
11
10
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)


Name of Sample: F-Br-NH
Spectrum No : 10705
ppm
157.876
150.657
142.699
122.965
122.636
117.216
117.109
113.135
112.835
100.173
77.455
77.235
77.032
76.609
6j
F
ppm
220
200
180
160
140
120
100
80
60
40
20


10962.001.esp
8.75
8.40
8.38
8.37
7.61
7.60
7.57
7.55
7.54
7.52
7.51
7.27
6.60
2.55
0.00
N
N
N
F
6k
1.08
1.07
2.30
1.03
3.67
11
10
9
8
7
6
5
4
3
2
1
0
-1
Chemical Shift (ppm)


Name of Sample: F-CHO-NH
Spectrum No : 11151
ppm
160.726
157.463
152.686
150.287
150.243
146.114
132.807
122.739
122.411
118.859
118.755
116.814
116.706
112.905
112.604
99.495
77.428
77.005
76.581
28.495
N
N
N
F
6k
ppm
220
200
180
160
140
120
100
80
60
40
20

Sample Name: Aceto-NH2-PzCN
Spectrum No.:203



acetopznh2
359