Copper(II) Ionic Liquid Catalyzed Cyclization-Aromatization of Hydrazones with Dimethyl Acetylenedicarboxylate: A Green Synthesis of Fully Substituted Pyrazoles

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General information

Melting points were determined with a Stuart Scientific SMP2 apparatus and are uncorrected. FT-IR spectra were obtained as KBr pellets using a Nicolet-Impact 400D instrument in the range of 400-4000 cm^{-1}. ^{1}H NMR (400 and 500 MHz) and ^{13}C NMR (100 and 125 MHz) spectra were recorded on Bruker-Avance 400 and 500 spectrometers, respectively. All mass spectra were recorded on Micromass Platform II spectrometer from Micromass. EI mode at 70 eV. Elemental analysis was carried out with a LECO, CHNS-932 instrument.

General procedure for synthesis of pyrazoles via cyclization-aromatization of hydrazone with dimethyl acetylenedicarboxylate

A mixture of aldehyde (1 mmol), arylhydrazine (1 mmol) was stirred for 20 min. Then, DMAD (1.2 mmol) and catalyst (0.25 mmol) were added and the mixture was heated at 100 ^{o}C under solvent-free conditions for the appropriate time (Table 2). The progress of the reaction was monitored by TLC (eluent: *n*-hexane/ethyl acetate, 9:1). After completion of the reaction, the mixture was cooled to room temperature and water was added (30 ml). The mixture was filtered and the crude product was purified by recrystallization from EtOH to afford the pure product. If necessary, the product was purified by silica gel column chromatography (eluent: *n*-hexane/ethyl acetate: 12/1).

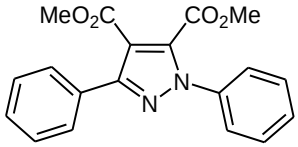
The structure of the products was identified by their IR, mass, ^{1}H and ^{13}C NMR spectra and elemental analysis. Furthermore, the structure of **4h** was confirmed byX-ray crystallographic analysis (CCDC 867509, Fig. 1).

Regarding to the number of peaks in^{13}CNMR and also the intensity of each peak, it is important to note that due to the use of polarization transfer (from ^{1}H to ^{13}C) *via* NOE effect in the pulse program applied to obtain the ^{13}C spectra, the carbon atoms attached to more number of hydrogen atoms acquire more polarization and thus, their peaks are more intense. Difference in the relaxation times is another source

of variation of the peak intensity. For example, the methyl group carbon atoms which are more exposed to solvent molecules has shorter relaxation times and thus, smaller intensities.

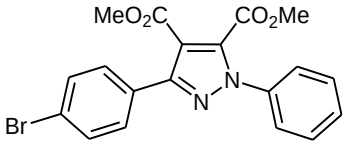
It is noteworthy that because of similar electronic environments, some ^{13}C nuclei have very close chemical shifts and appear at the same position. Therefore, the number of peaks in the ^{13}C NMR spectra correspond to the number of types of carbon atoms is less than the number of carbon atoms of the compounds.

Spectroscopic data of the products



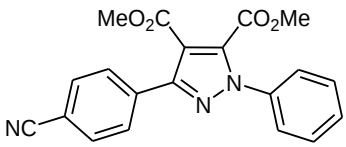
Dimethyl 1,3-diphenyl-1*H*-pyrazole-4,5-dicarboxylate (4a)[1,2]

Mp: 154-155 °C. IR (KBr): ν_{max} = 3008, 2951, 1732, 1594, 1497, 1269, 1107, 760 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ = 3.83 (s, 3H), 3.86 (s, 3H), 7.41-7.55 (m, 8H), 7.76 (dd, ^1J = 7.7 Hz, ^2J = 1.8 Hz, 2H).



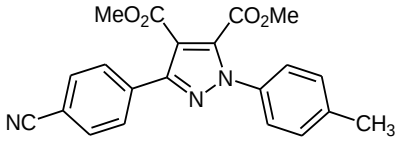
Dimethyl3-(4-bromophenyl)-1-phenyl-1*H*-pyrazole-4,5-dicarboxylate (4b)[1]

Mp: 85 °C. IR (KBr): ν_{max} = 3064, 2949, 1714, 1733, 1596, 1497, 1229, 1186, 759 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ = 3.83 (s, 3H), 3.86 (s, 3H), 7.46-7.54 (m, 5H), 7.56 (d, *J* = 8.4 Hz, 2H), 7.65 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 52.21, 53.23, 113.88, 123.36, 124.49, 129.24, 129.29, 130.36, 130.52, 131.39, 137.32, 138.95, 151.08, 160.65, 163.12.



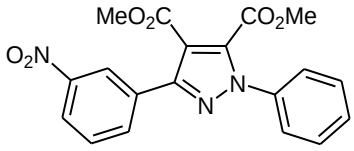
Dimethyl 3-(4-cyanophenyl)-1-phenyl-1*H*-pyrazole-4,5-dicarboxylate (4c)[1]

Mp: 137-138 °C. IR (KBr): ν_{max} = 3067, 2948, 2227, 1733, 1595, 1446, 1244, 1123, 824 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ = 3.84 (s, 3H), 3.87 (s, 3H), 7.49-7.54 (m, 5H), 7.73 (d, *J* = 8.2 Hz, 2H), 7.92 (d, *J* = 8.2 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 52.32, 53.33, 112.57, 114.02, 118.72, 124.42, 129.37, 129.46, 129.61, 131.95, 135.95, 137.78, 138.77, 150.30, 160.50, 162.80.



Dimethyl 3-(4-cyanophenyl)-1-*p*-tolyl-1*H*-pyrazole-4,5-dicarboxylate (4d)

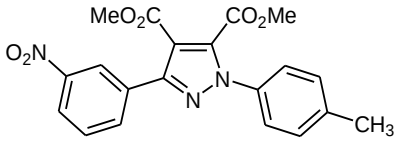
Mp: 140 °C. IR (KBr): ν_{max} = 3011, 2952, 2225, 1728, 1510, 1445, 1246, 1123, 819 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ = 2.42 (s, 3H), 3.83 (s, 3H), 3.87 (s, 3H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.71 (d, *J* = 8.1 Hz, 2H), 7.91 (d, *J* = 8.1 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 21.22, 52.26, 53.31, 112.47, 113.78, 118.75, 118.78, 124.23, 129.61, 129.92, 131.93, 136.04, 136.33, 137.70, 139.69, 150.12, 162.85. MS: m/z = 376.07 ($[M+2]^{\cdot+}$, 60.34), 375.06 ($[M]^{\cdot+}$, 93.97), 344.07 (84.48), 125.17 (79.74), 111.14 (89.22), 97.13 (48.28), 57.11 (95.26). Anal. Calcd for $C_{21}H_{17}N_3O_4$: C, 67.19; H, 4.56; N, 11.19. Found: C, 67.01; H, 4.68; N, 11.24.



Dimethyl 3-(3-nitrophenyl)-1-phenyl-1*H*-pyrazole-4,5-dicarboxylate (4e)

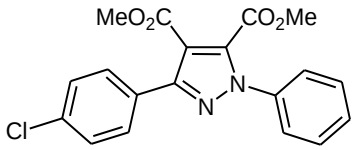
Mp: 111 °C. IR (KBr): ν_{max} = 3032, 2959, 1722, 1595, 1447, 1268, 1116, 758 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): δ = 3.86 (s, 3H), 3.89 (s, 3H), 7.49-7.57 (m, 5H), 7.62 (t, *J* = 8.0 Hz, 1H), 8.15 (d, *J* = 7.8 Hz, 1H), 8.27-8.29 (m, 1H), 8.70 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 52.33, 53.39, 113.81, 123.65, 124.19, 124.35, 129.10, 129.41, 129.46, 133.13, 135.02, 138.02, 138.73, 148.24, 149.98,

160.62, 162.67. MS: *m/z* = 381.04 ([M]$^{\cdot+}$, 25.82), 350.05 (22.95), 207.21 (11.27), 125.07 (85.66), 111.06 (93.85), 97.05 (95.08), 57.02 (100). Anal. Calcd for $C_{19}H_{15}N_3O_6$: C, 59.84; H, 3.96; N, 11.02. Found: C, 59.92; H, 4.05; N, 11.11.



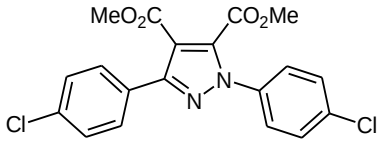
Dimethyl 3-(3-nitrophenyl)-1-*p*-tolyl-1*H*-pyrazole-4,5-dicarboxylate (4f)

Mp: 132-135 °C. IR (KBr): ν_{max} = 3030, 2951, 1727, 1522, 1497, 1354, 1128, 817 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.43 (s, 3H), 3.85 (s, 3H), 3.88 (s, 3H), 7.30 (d, *J* = 8.1 Hz, 2H), 7.42 (d, *J* = 8.1 Hz, 2H), 7.61 (t, *J* = 7.9 Hz, 1H), 8.14 (d, *J* = 7.2 Hz, 1H), 8.26-8.28 (m, 1H), 8.69 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 21.22, 52.29, 53.35, 113.60, 123.60, 124.16, 128.82, 129.07, 129.95, 133.21, 135.03, 136.30, 137.95, 139.68, 148.20, 149.80, 160.72, 162.72. MS: *m/z* = 396.99 ([M+2]$^{\cdot+}$, 15.24), 394.96 ([M]$^{\cdot+}$, 100), 363.94 (88.62), 331.95 (16.87), 229.00 (16.16, 129.95 (29.67), 90.95 (84.96), 76.95 (42.68). Anal. Calcd for $C_{20}H_{17}N_3O_6$: C, 60.76; H, 4.33; N, 10.63. Found: C, 60.63; H, 4.45; N, 10.66.



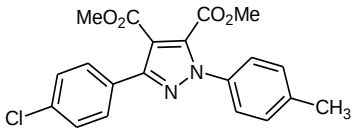
Dimethyl 3-(4-chlorophenyl)-1-phenyl-1*H*-pyrazole-4,5-dicarboxylate (4g)[1]

Mp: 63-65 °C. IR (KBr): ν_{max} = 3018, 2965, 1735, 1706, 1594, 1498, 1233, 1165, 839 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.83 (s, 3H), 3.87 (s, 3H), 7.41 (d, *J* = 8.5 Hz, 2H), 7.42-7.54 (m, 5H), 7.72 (d, *J* = 8.5 Hz, 2H).



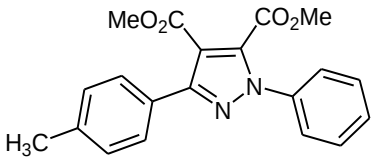
Dimethyl 1,3-bis(4-chlorophenyl)-1*H*-pyrazole-4,5-dicarboxylate (4h)

Mp: 130-132 °C. IR (KBr): ν_{max} = 3001, 2949, 1735, 1539, 1496, 1228, 1089, 834 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.83 (s, 3H), 3.88 (s, 3H), 7.41 (d, *J* = 7.6 Hz, 2H), 7.46 (s, 4H), 7.70 (d, *J* = 7.6 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 52.29, 53.30, 114.48, 125.87, 128.51, 129.45, 129.66, 130.12, 135.20, 135.23, 137.00, 137.45, 151.13, 160.37, 163.09. MS: *m/z* = 407.99 ($[M+4]^{\cdot+}$, 24.28), 405.99 ($[M+2]^{\cdot+}$, 75.00), 403.99 ($[M]^{\cdot+}$, 84.84), 372.98 (80.33), 184.56 (27.05), 125.13(34.43), 111.07 (100), 97.12 (87.30), 69.09 (87.70). Anal. Calcd for $C_{19}H_{14}Cl_2N_2O_4$: C, 56.31; H, 3.48; N, 6.91. Found: C, 56.24; H, 3.53; N, 6.90.



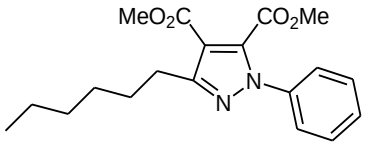
Dimethyl 3-(4-chlorophenyl)-1-*p*-tolyl-1*H*-pyrazole-4,5-dicarboxylate (4i)

Mp: 136 °C. IR (KBr): ν_{max} = 3013, 2947, 1726, 1602, 1511, 1277, 1102, 824 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 2.45 (s, 3H), 3.82 (s, 3H), 3.86 (s, 3H), 7.28 (d, *J* = 7.9 Hz, 2H), 7.40 (d, *J* = 7.9 Hz, 2H), 7.71 (d, *J* = 7.9 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ = 21.19, 52.14, 53.17, 113.68, 124.32, 128.40, 129.82, 129.98, 130.26, 134.98, 136.53, 137.25, 139.39, 150.88, 160.73, 163.18. MS: *m/z* = 386.02 ($[M+2]^{\cdot+}$, 34.12), 384.00 ($[M]^{\cdot+}$, 70.59), 353.01 (61.57), 179.20 (22.84), 111.12 (86.67), 97.12 (89.80), 69.09 (98.43), 57.09 (100). Anal. Calcd for $C_{20}H_{17}ClN_2O_4$: C, 62.42; H, 4.45; N, 7.28. Found: C, 62.23; H, 4.50; N, 7.23.



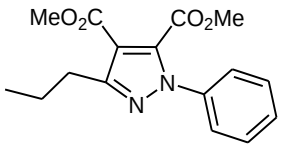
Dimethyl 1-phenyl-3-*p*-tolyl-1*H*-pyrazole-4,5-dicarboxylate (4j) [1, 2]

Mp: 66 °C. IR (KBr): ν_{max} = 3032, 2959, 1722, 1595, 1447, 1268, 1116, 758 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.41 (s, 3H), 3.85 (s, 3H), 3.87(s, 3H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.46-7.56 (m, 5H), 7.66 (d, *J* = 8.4 Hz, 2H).



Dimethyl 3-hexyl-1-phenyl-1*H*-pyrazole-4,5-dicarboxylate (4k)

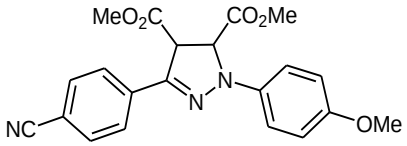
Oil. IR (neat): ν_{max} = 3042, 2955, 1725, 1597, 1504, 1255, 1108, 761 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 0.89 (s, 3H),1.26-141 (m, 6H), 1.68-1.74 (m, 2H), 2.91 (t, *J* = 7.7 Hz, 2H), 3.85 (s, 6H), 7.41-7.49 (m, 5H). ^{13}C NMR (125 MHz, $CDCl_3$): δ =14.08, 22.60, 27.61, 29.05, 29.21, 31.59, 51.70, 53.24, 100.01, 123.82, 128.74, 129.32, 134.14, 141.74, 145.73, 154.48, 158.03. MS: *m/z* = 345.14 ($[M+1]^{\cdot+}$, 31.10), 344.14 ($[M]^{\cdot+}$, 68.11), 313.14 (53.54), 255.10 (67.32), 113.09 (92.52), 77.02 (96.46), 55.05 (100). Anal. Calcd for $C_{19}H_{24}N_2O_4$: C, 66.26; H, 7.02; N, 8.13. Found: C, 66.29; H, 7.14; N, 8.21.



Dimethyl 3-propyl-1-phenyl-1*H*-pyrazole-4,5-dicarboxylate (4l)

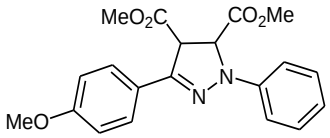
Oil. IR (neat): ν_{max} = 3024, 2957, 1744, 1724, 1597, 1504, 1255, 1106, 761 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.02 (t, *J* = 7.2 Hz, 3H), 1.72-1.82 (m, 2H), 2.92 (t, *J* = 8.0 Hz, 2H), 3.87 (s, 6H), 7.42-

7.51 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$): δ =14.02, 22.37, 29.50, 51.73, 53.28, 114.17, 123.78, 128.75, 129.33, 131.27, 139.13, 144.91, 155.08, 157.16. MS: *m/z* = 303.11 ($[M+1]^{\cdot+}$, 56.86), 302.10 ($[M]^{\cdot+}$, 82.35), 271.07 (84.31), 255.00 (4.11), 183.14 (67.06), 158.13 (88.63), 111.17 (74.17), 77.09 (100). Anal. Calcd for $C_{16}H_{18}N_2O_4$: C, 63.56; H, 6.00; N, 9.27. Found: C, 63.45; H, 5.90; N, 9.33.



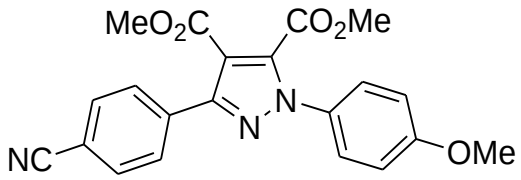
Dimethyl3-(4-cyanophenyl)-1-(4-methoxyphenyl)-4,5-dihydro-1*H*-pyrazole-4,5-dicarboxylate (5m)

Mp: 184-186 ^{o}C. IR (KBr): ν_{max} = 3039, 2931, 2224, 1742, 1509, 1238, 1153, 1011, 820 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.74 (s, 3H), 3.76 (s, 3H), 3.79 (s, 3H), 4.55 (d, *J* = 5.0 Hz, 1H), 5.26 (d, *J* = 5.0 Hz, 1H), 6.89 (d, *J* = 8.9 Hz, 2H),7.12 (d, *J* = 9.0 Hz, 2H), 7.64 (d, *J* = 8.4 Hz, 2H), 7.86 (d, *J* = 8.4 Hz, 2H).^{13}C NMR (125 MHz, $CDCl_3$): δ = 53.17, 53.36, 54.90, 55.65, 66.61, 111.33, 114.76, 115.25, 118.85, 126.28, 132.24, 135.66, 137.02, 140.47, 154.76, 168.83, 169.66. MS: *m/z* = 393.09 ($[M]^{\cdot+}$, 0.89), 334.03 (1.19), 273.06 (1.00), 129.99 (1.92), 101.96 (5.01), 59.01 (100). Anal. Calcd for $C_{21}H_{19}N_3O_5$: C, 64.12; H, 4.87; N, 10.68. Found: C, 64.05; H, 4.90; N, 10.64.



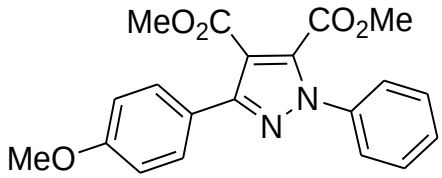
Dimethyl3-(4-methoxyphenyl)-1-phenyl-4,5-dihydro-1*H*-pyrazole-4,5-dicarboxylate (5n)[3]

Mp: 151-152 ^{o}C. IR (KBr): ν_{max} = 3053, 2953, 1744, 1595, 1500, 1242, 1143, 1015, 834 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.72 (s, 3H), 3.76 (s, 3H), 3.85 (s, 3H), 4.55 (d, *J* = 4.5 Hz, 1H), 5.15 (d, *J* = 5.0 Hz, 1H), 6.93-6.88 (m, 3H), 7.13 (d, *J* = 7.8 Hz, 2H),7.30 (t, *J* = 8.5 Hz, 2H), 7.75 (d, *J* = 8.9 Hz, 2H). MS: *m/z* = 368.07 ($[M]^{\cdot+}$, 5.86), 309.07 (7.09), 250.11 (7.48), 207.12 (8.57), 111.11 (15.87), 77.06 (46.63), 43.15 (100).



Dimethyl 3-(4-cyanophenyl)-1-(4-methoxyphenyl)-1*H*-pyrazole-4,5-dicarboxylate (4m)

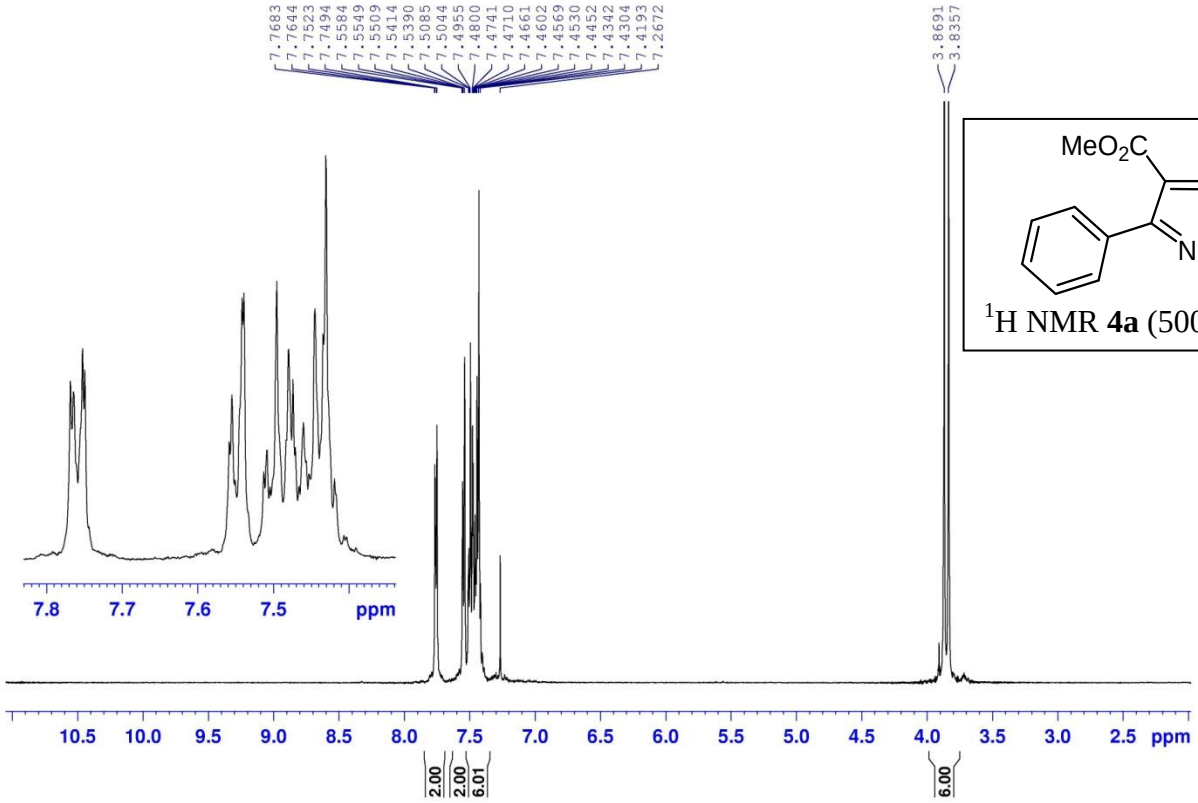
Mp: 125 ^{o}C. IR (KBr): ν_{max} = 3029, 2943, 2223, 1733, 1506, 1150, 1008, 828 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.84 (s, 3H), 3.866 (s, 3H), 3.868 (s, 3H), 6.99 (d, *J*= 8.8 Hz, 2H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.72 (d, *J* = 8.8 Hz, 2H), 7.90 (d, *J* = 9.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 52.34, 53.34, 55.61, 114.40, 118.76, 125.96, 128.80, 129.55, 130.62, 130.90, 131.68, 132.33, 132.46, 135.98, 150.03, 160.25, 160.52. MS: *m/z* = 391.10 ($[M]^{\cdot+}$, 15.70), 360.09 (8.52), 273.08 (2.38), 130.02 (23.99), 77.05 (38.12), 58.97 (100). Anal. Calcd for $C_{21}H_{17}N_3O_5$: C, 64.45; H, 4.38; N, 10.74. Found: C, 64.43; H, 4.41; N, 10.72.

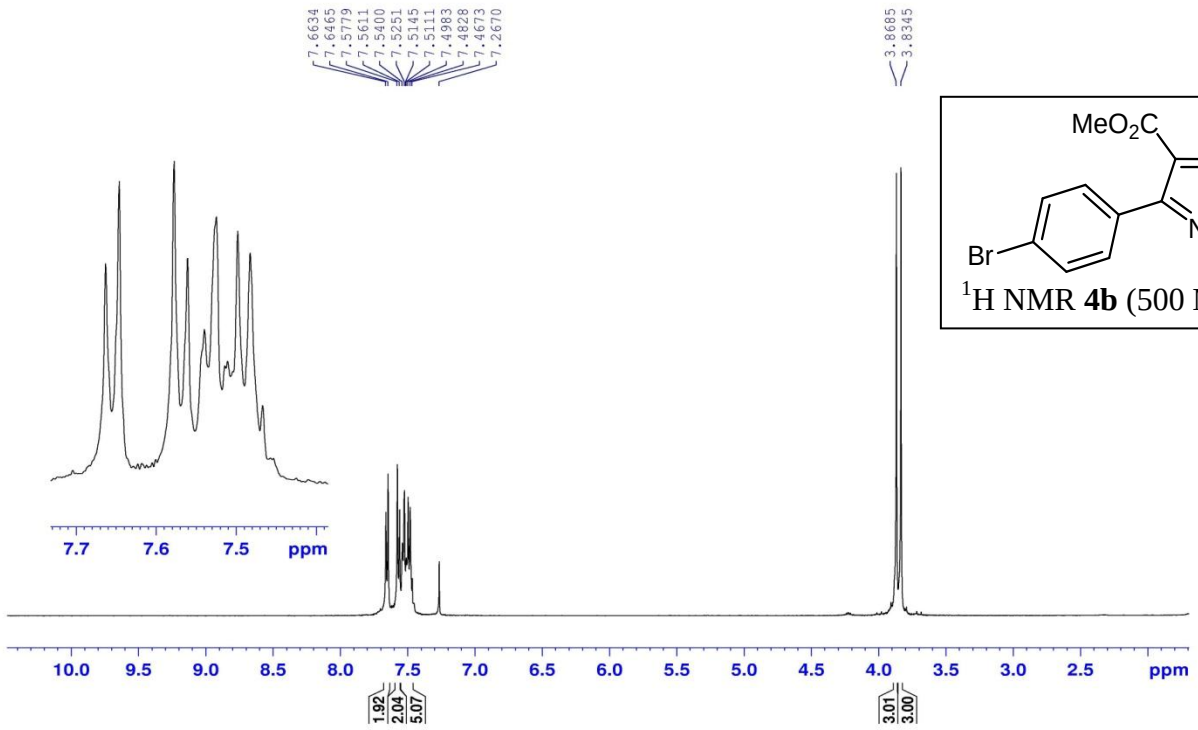


Dimethyl 3-(4-methoxyphenyl)-1-phenyl-1*H*-pyrazole-4,5-dicarboxylate (4n)

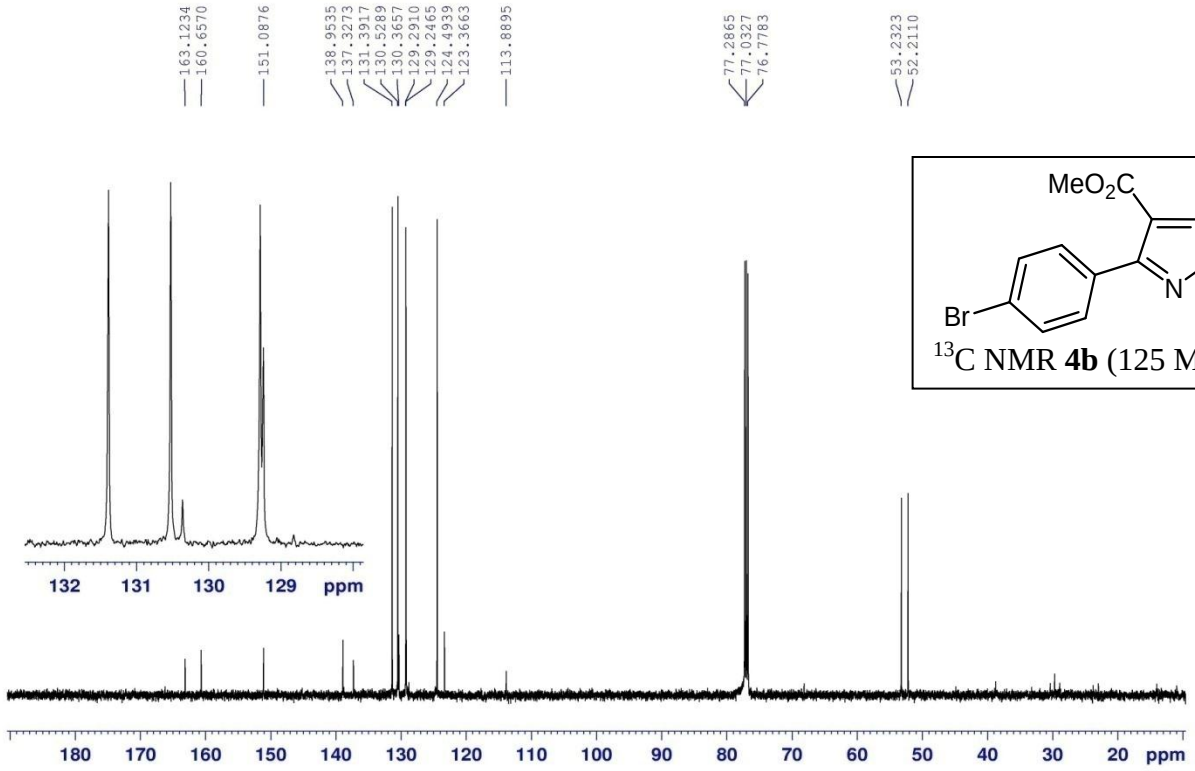
Mp: 110 ^{o}C. IR (KBr): ν_{max} = 3050, 2948, 1740, 1597, 1502, 1237, 830 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.83 (s, 3H), 3.857 (s, 3H), 3.859 (s, 3H), 6.97 (d, *J* = 9.2 Hz, 2H), 7.41-7.46 (m, 4H), 7.73-7.75 (M, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 52.18, 53.13, 55.57, 114.25, 126.14, 128.20, 128.48, 128.76, 128.80, 128.85, 130.89, 132.09, 133.11, 152.73, 160.03, 161.01. MS: *m/z* = 366.09 ($[M]^{\cdot+}$, 2.53), 335.09 (7.27), 149.06 (12.27), 111.10 (17.61), 77.06 (40.00), 59.05 (100). Anal. Calcd for $C_{20}H_{18}N_2O_5$: C, 65.57; H, 4.95; N, 7.65. Found: C, 65.53; H, 4.99; N, 7.70.

Copies of ^{1}H NMR and ^{13}C NMR of products:

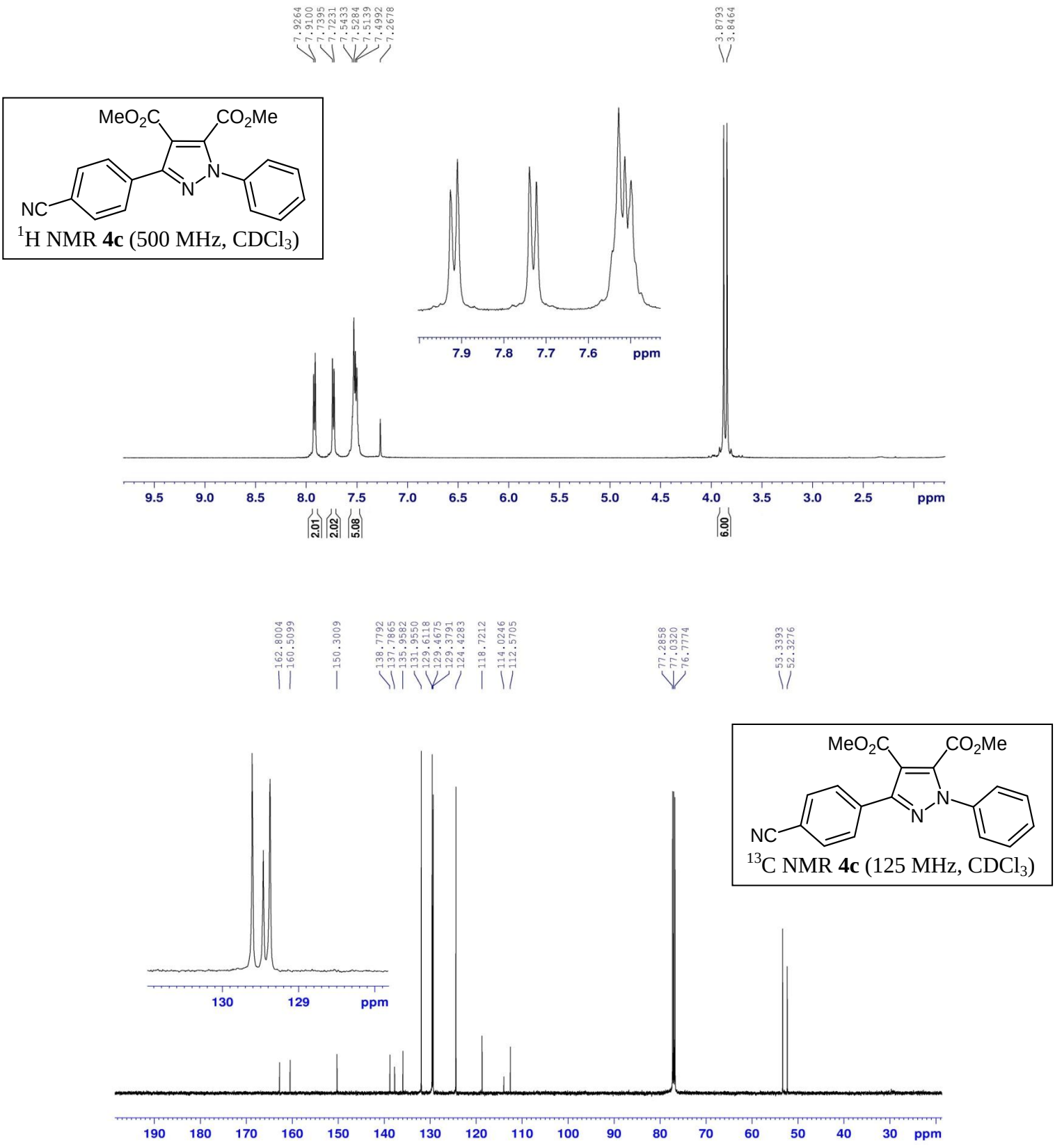


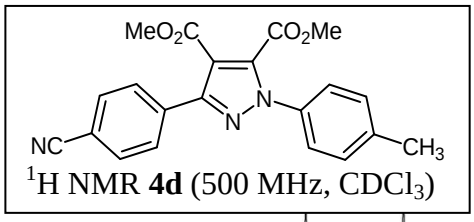


^{1}H NMR **4b** (500 MHz, $CDCl_3$)

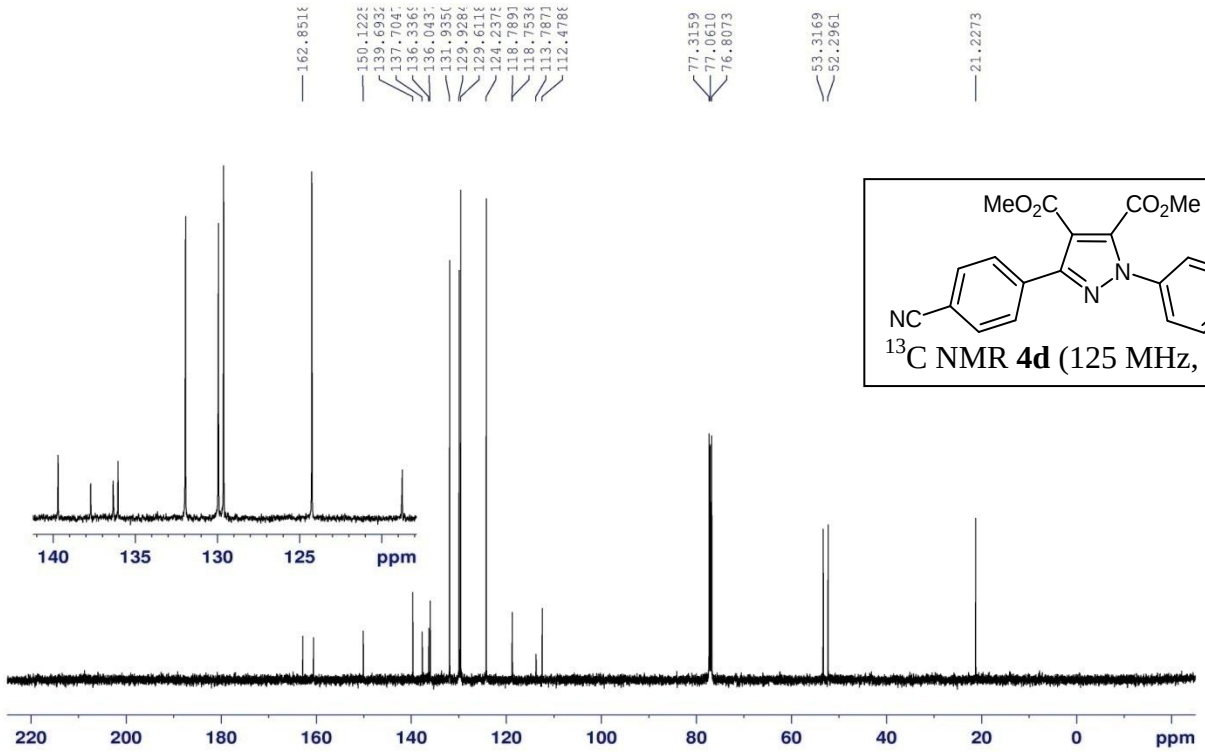


^{13}C NMR **4b** (125 MHz, $CDCl_3$)

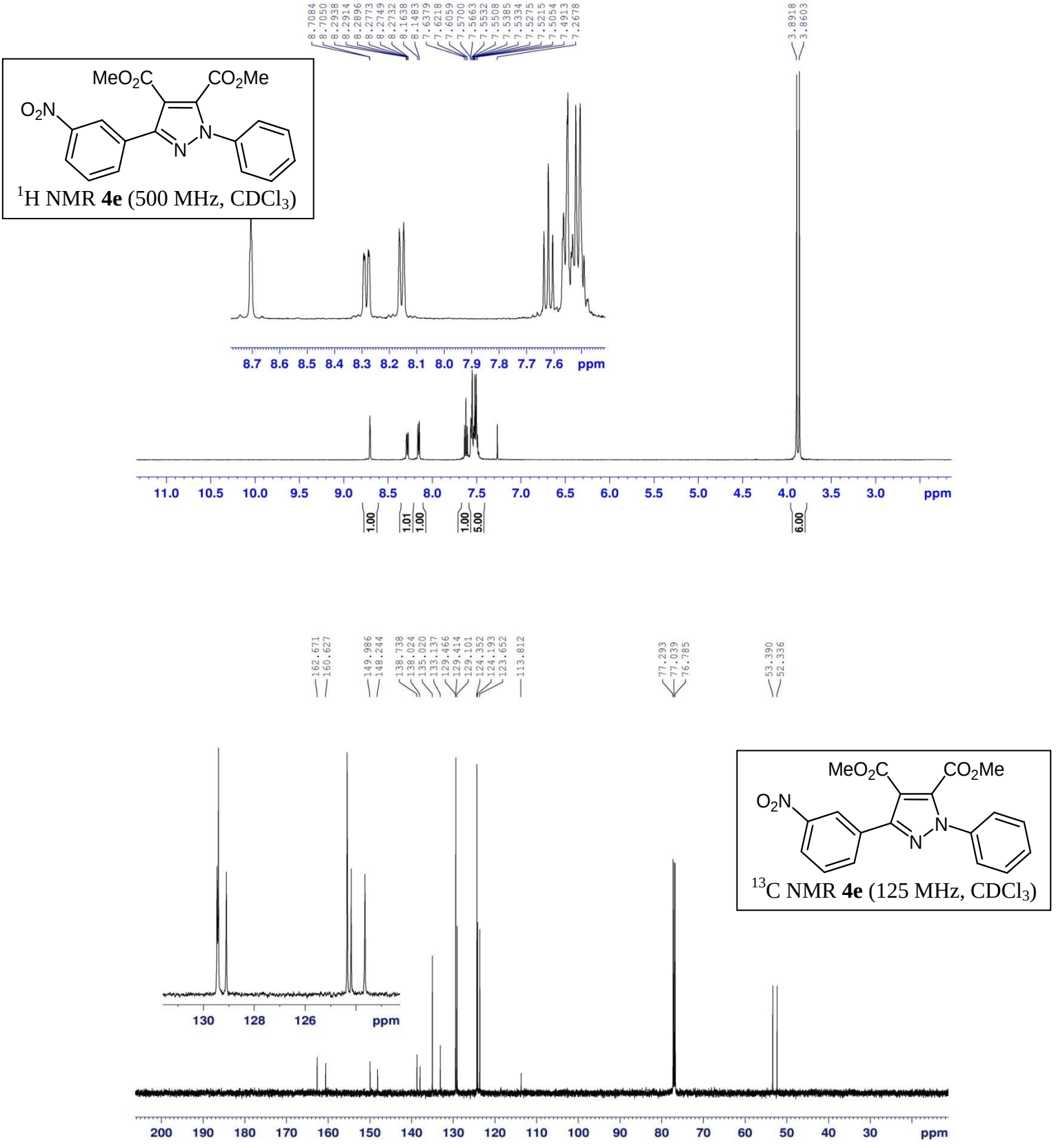


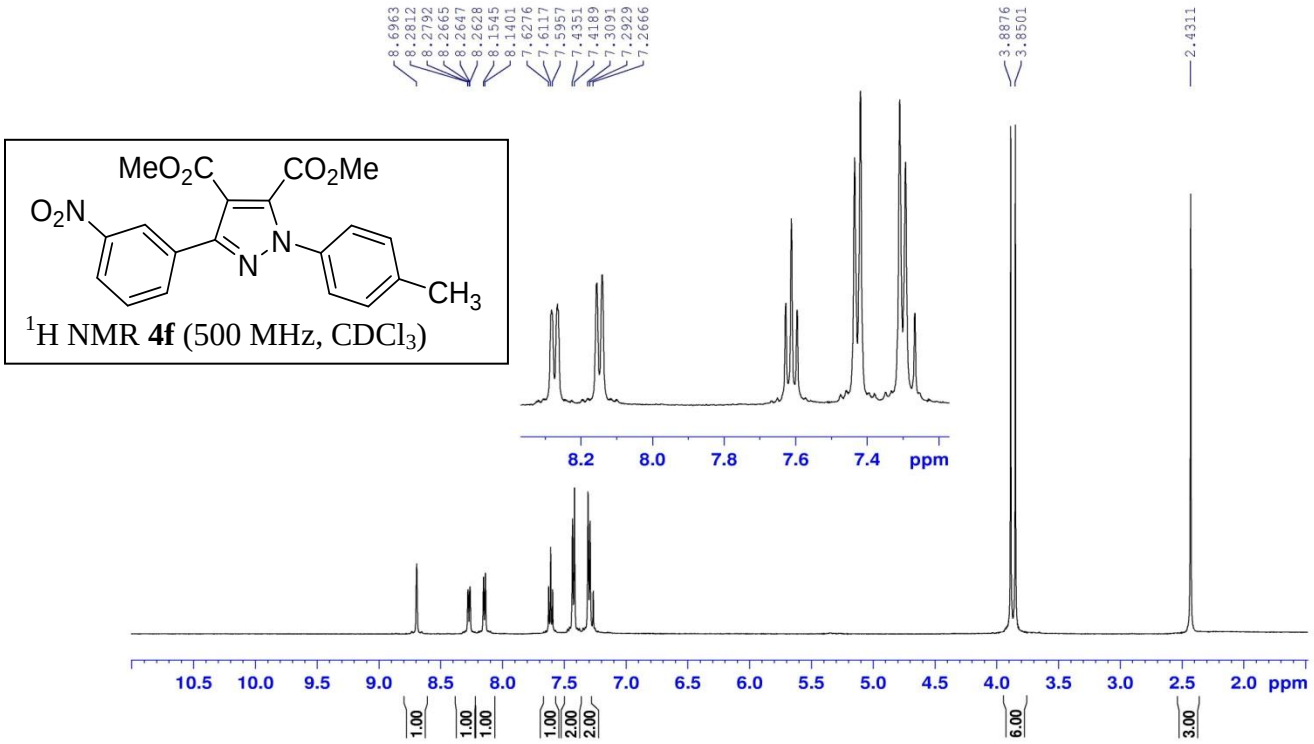


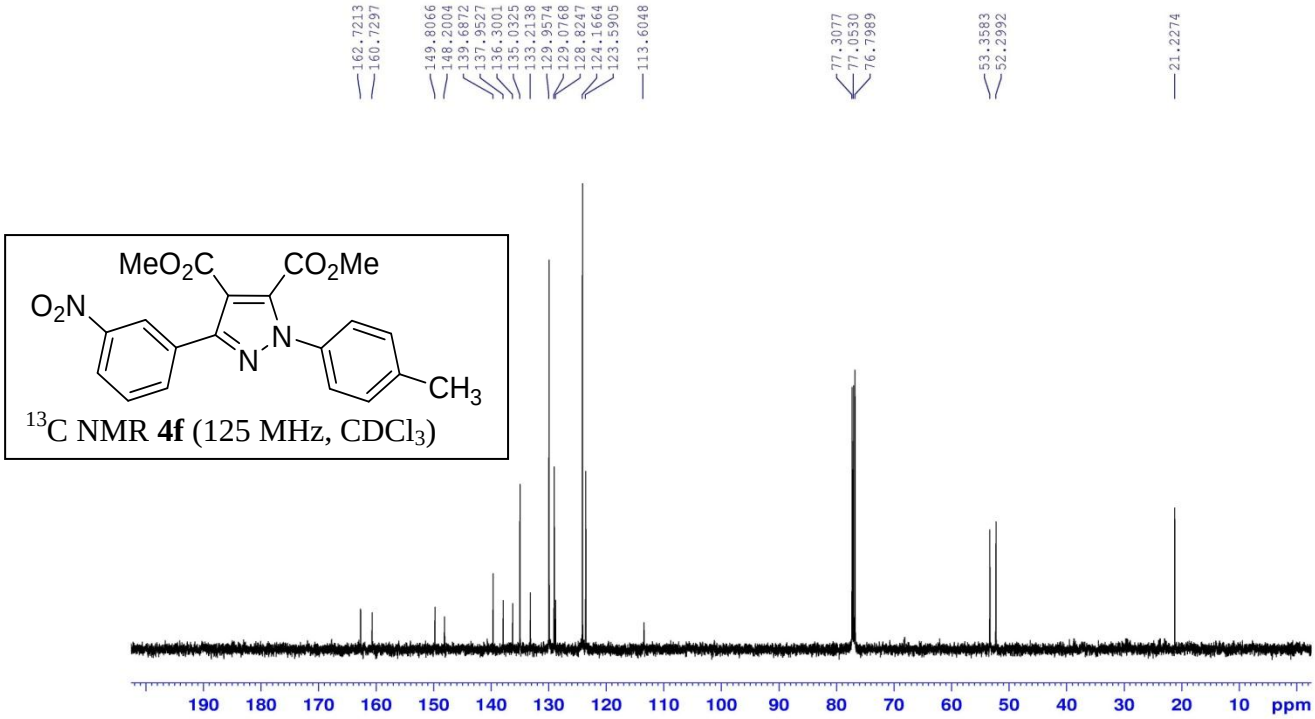
^{1}H NMR **4d** (500 MHz, $CDCl_3$)

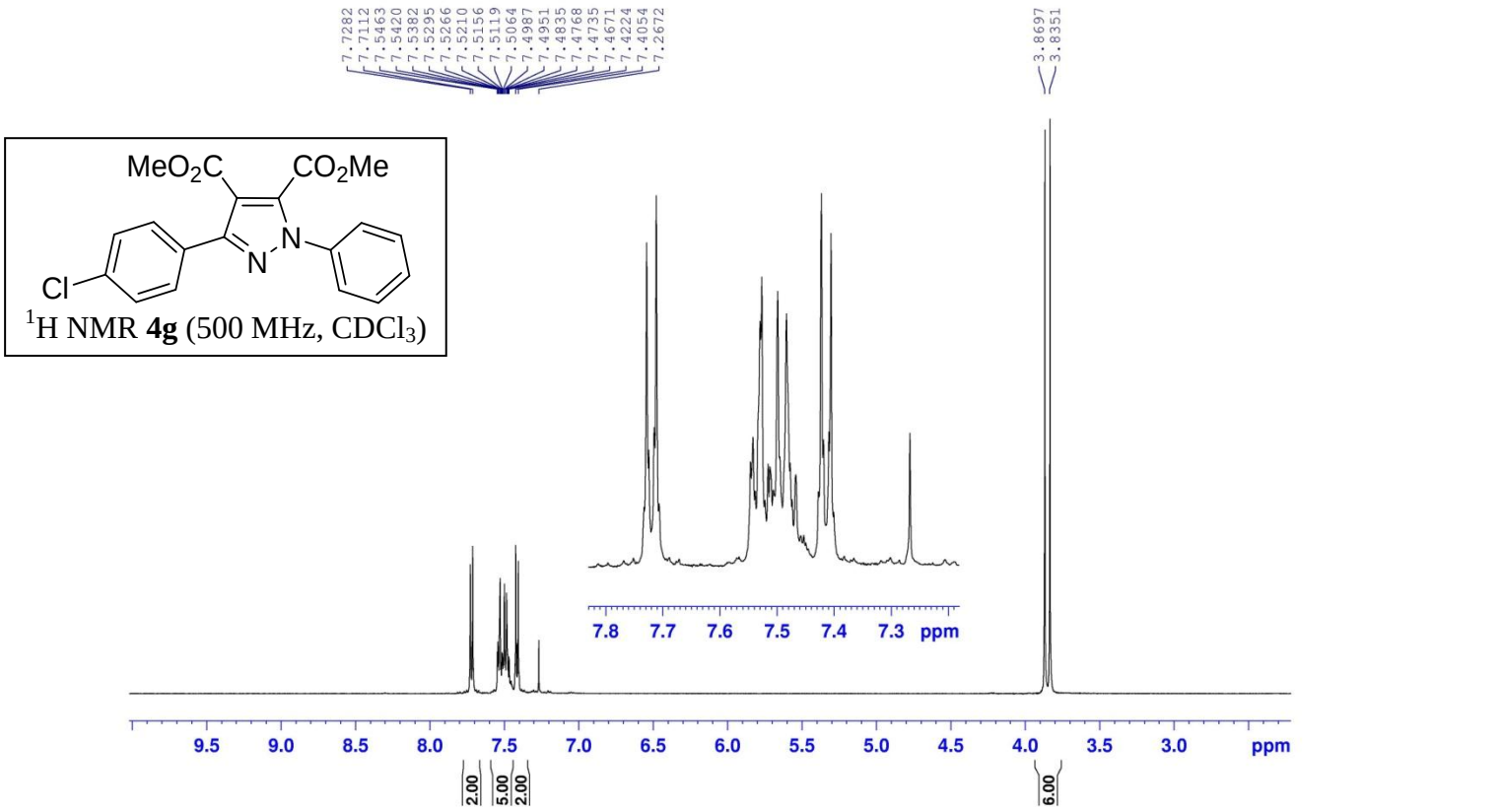


^{13}C NMR **4d** (125 MHz, $CDCl_3$)

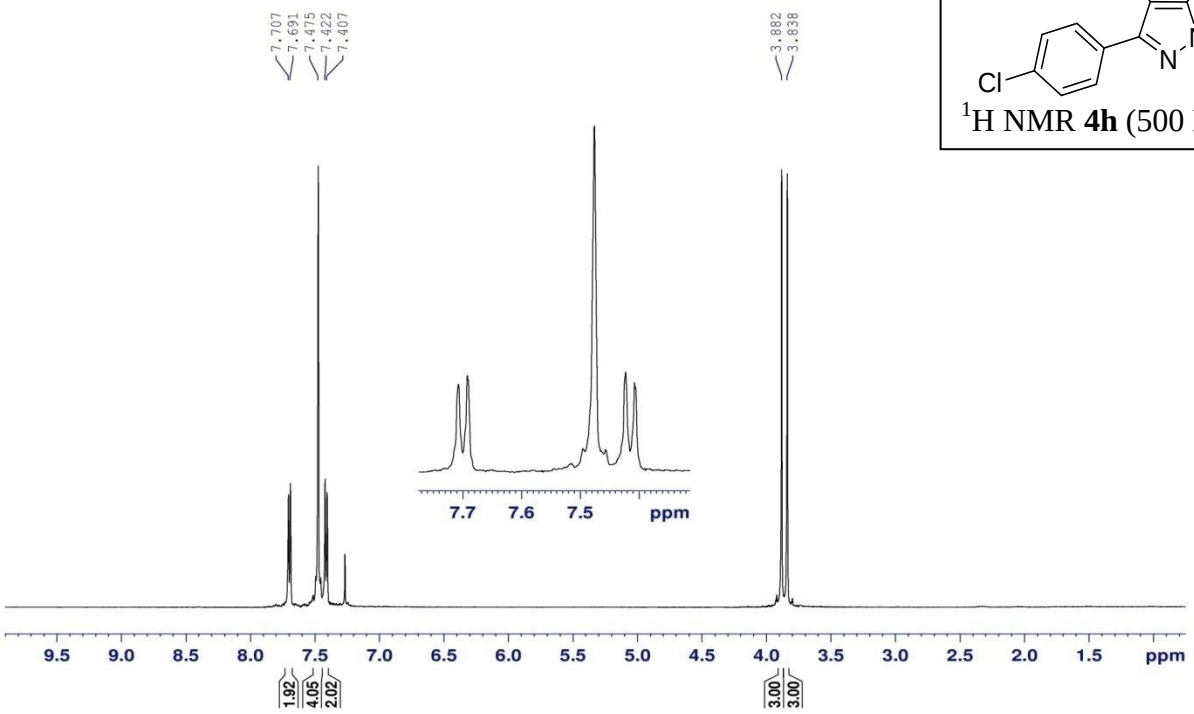


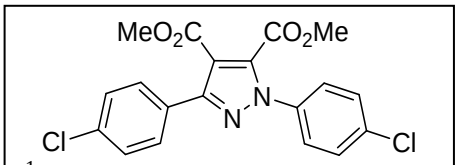




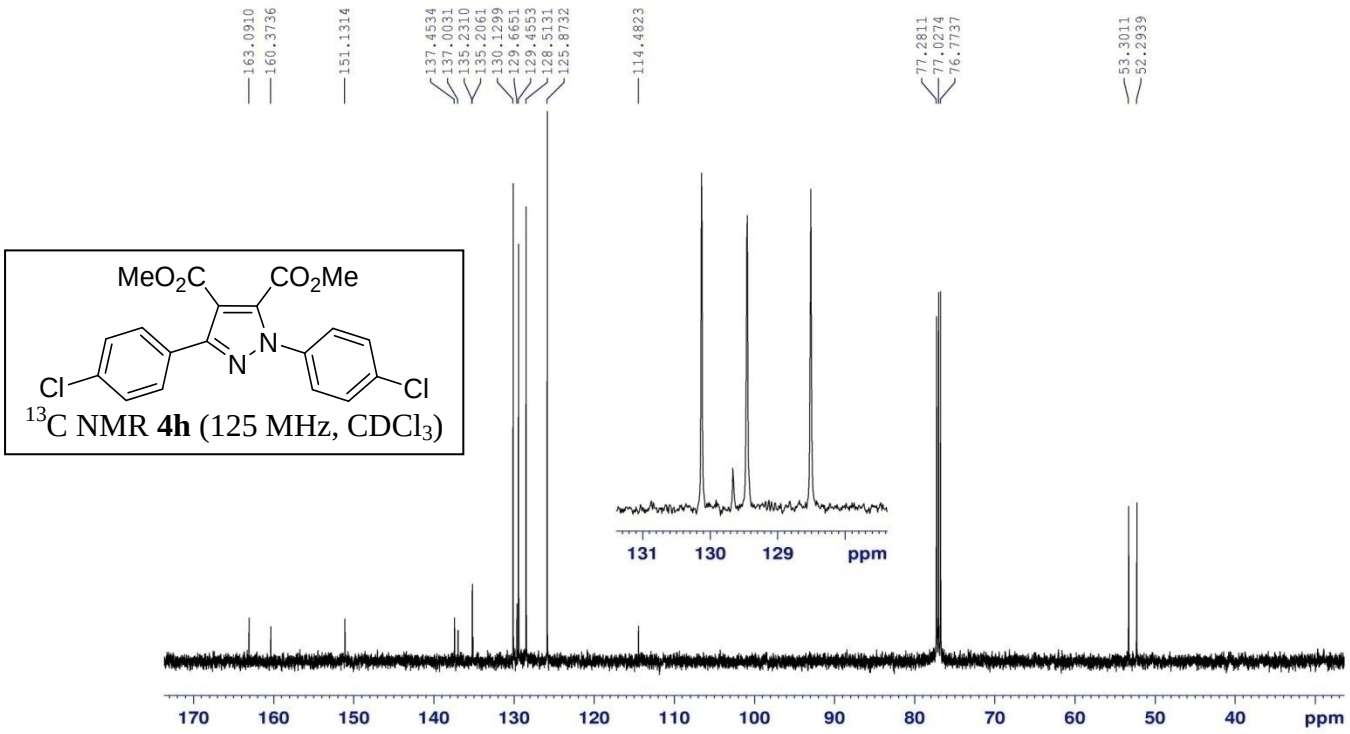


^{1}H NMR **4g** (500 MHz, $CDCl_3$)

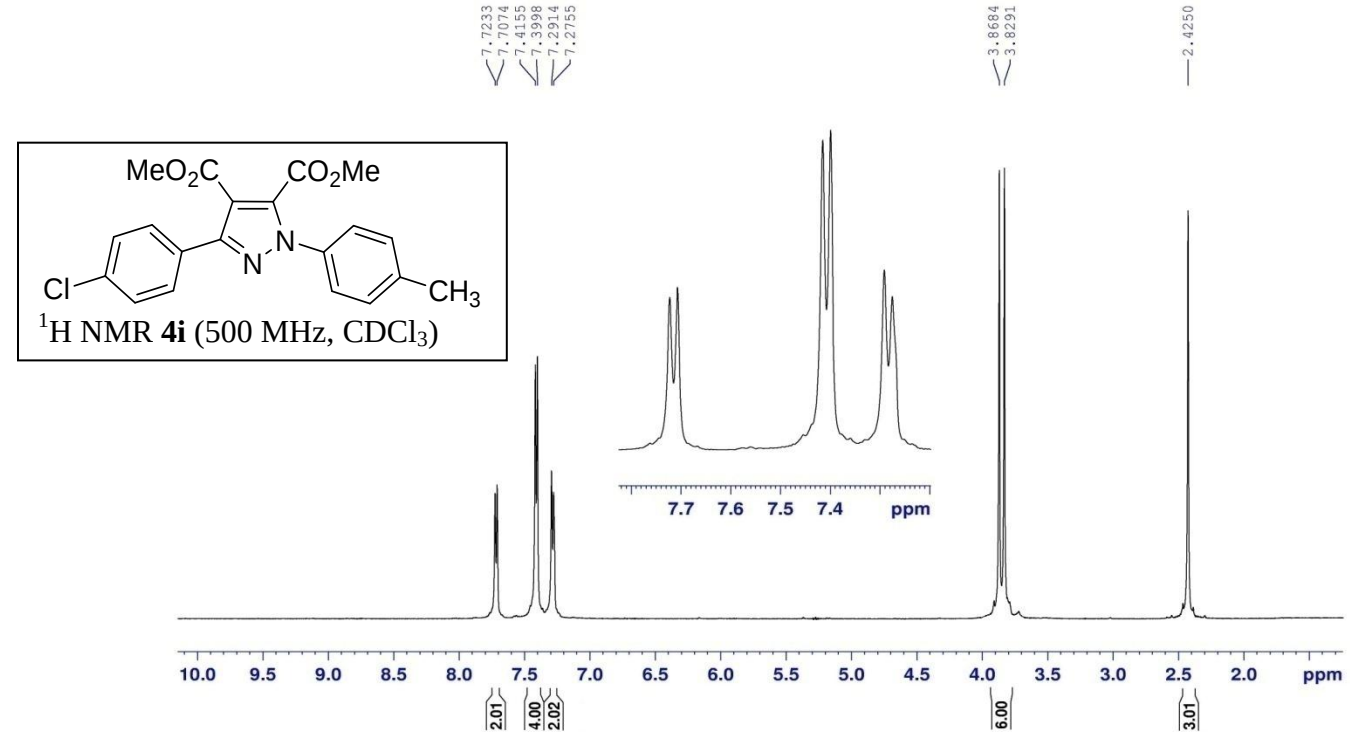




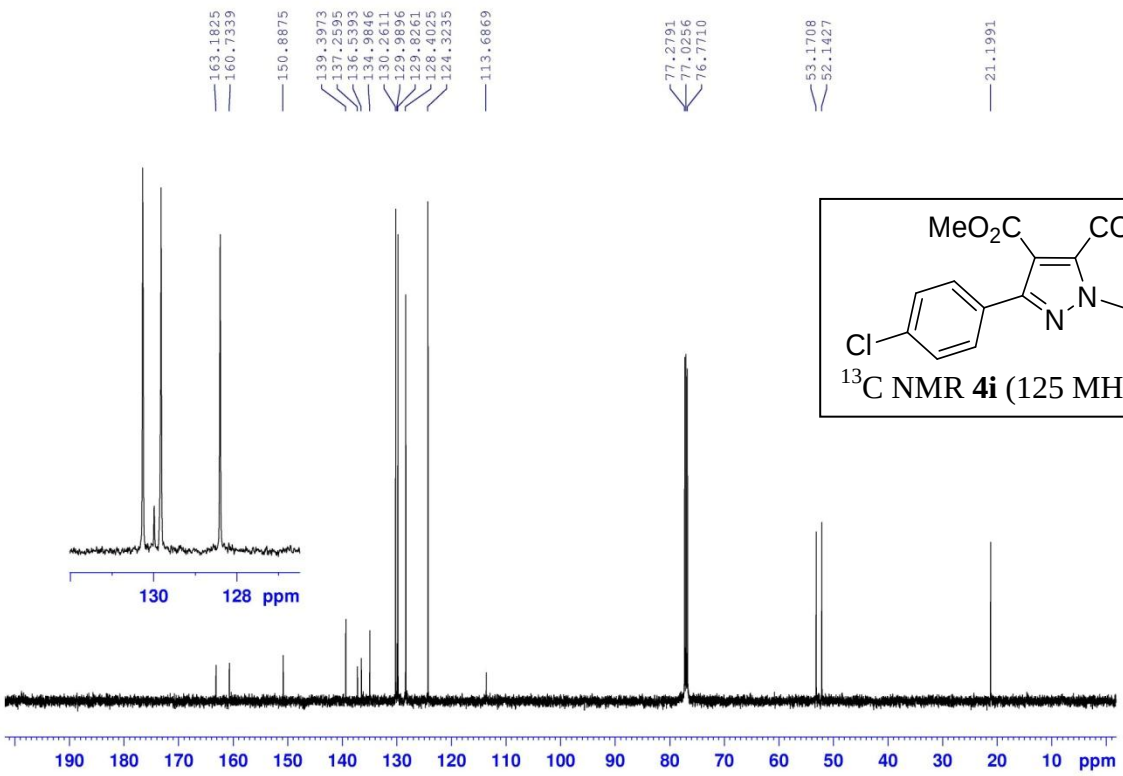
^{1}H NMR **4h** (500 MHz, $CDCl_3$)



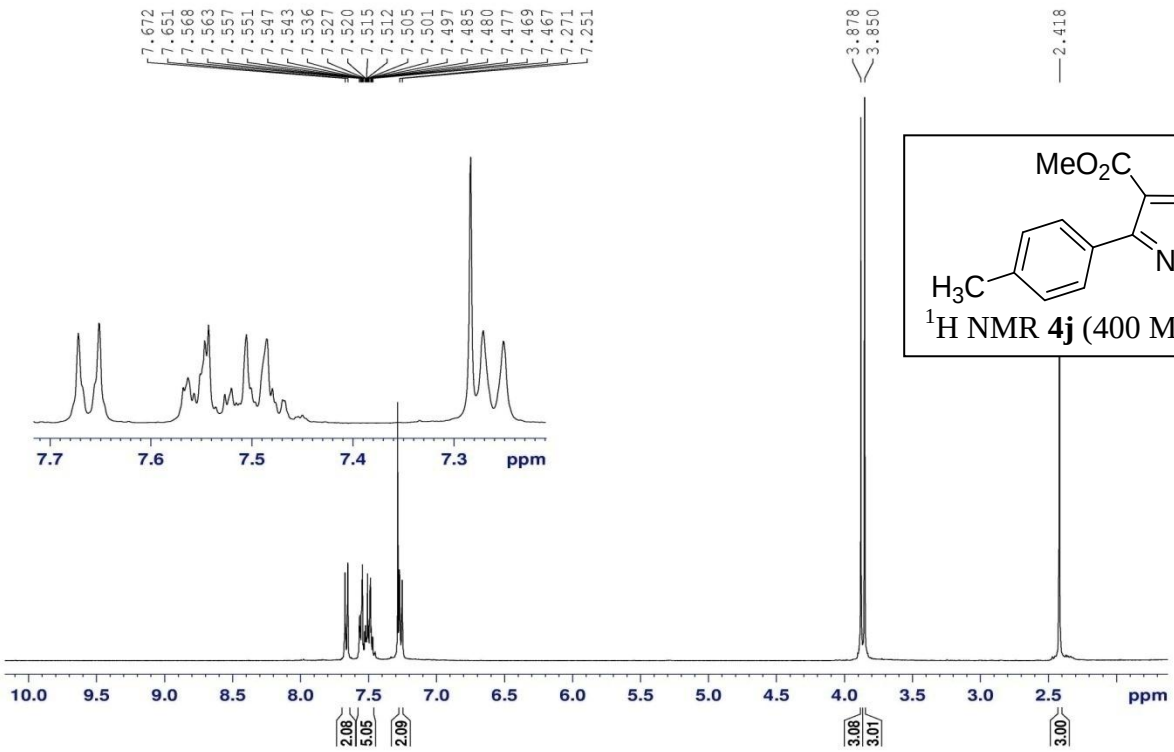
^{13}C NMR **4h** (125 MHz, $CDCl_3$)



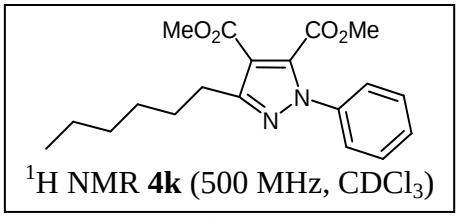
^{1}H NMR **4i** (500 MHz, $CDCl_3$)



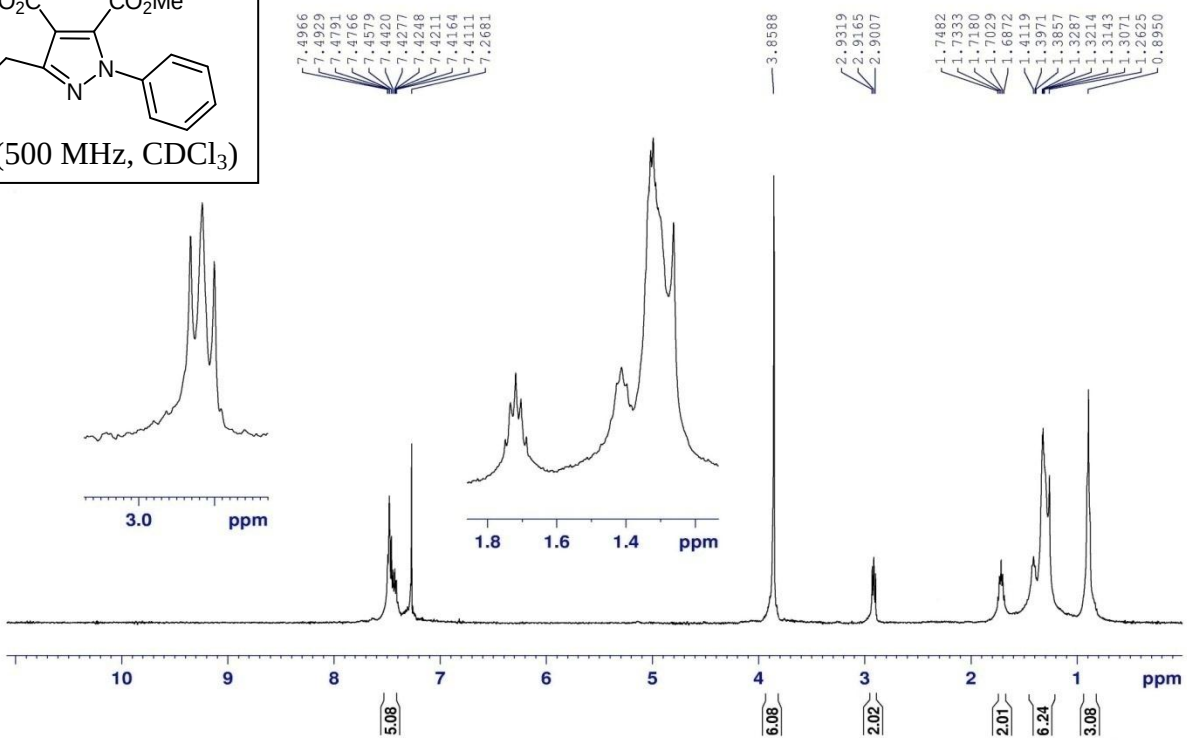
^{13}C NMR **4i** (125 MHz, $CDCl_3$)

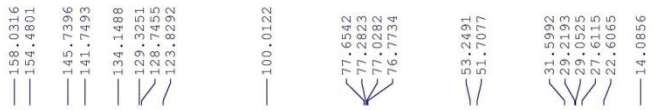


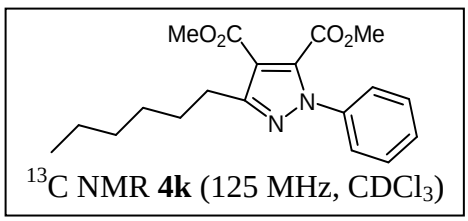
^{1}H NMR **4j** (400 MHz, $CDCl_3$)



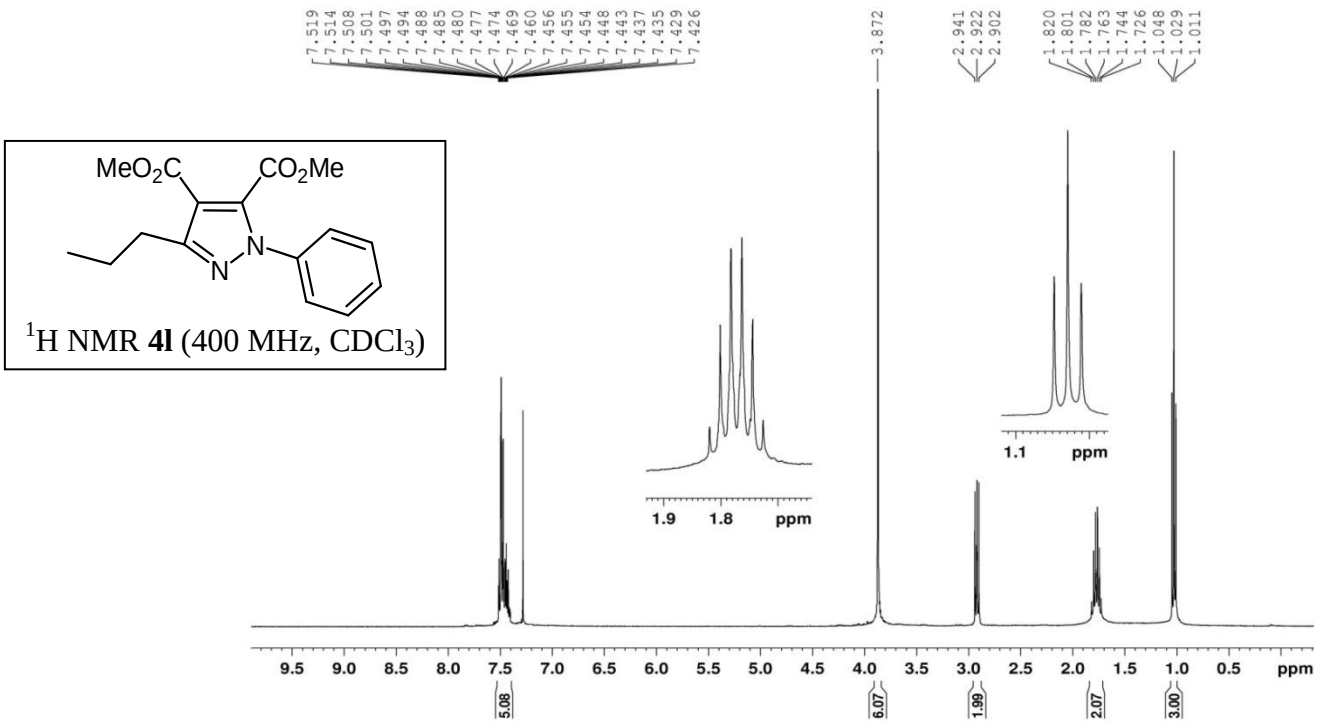
^{1}H NMR **4k** (500 MHz, $CDCl_3$)



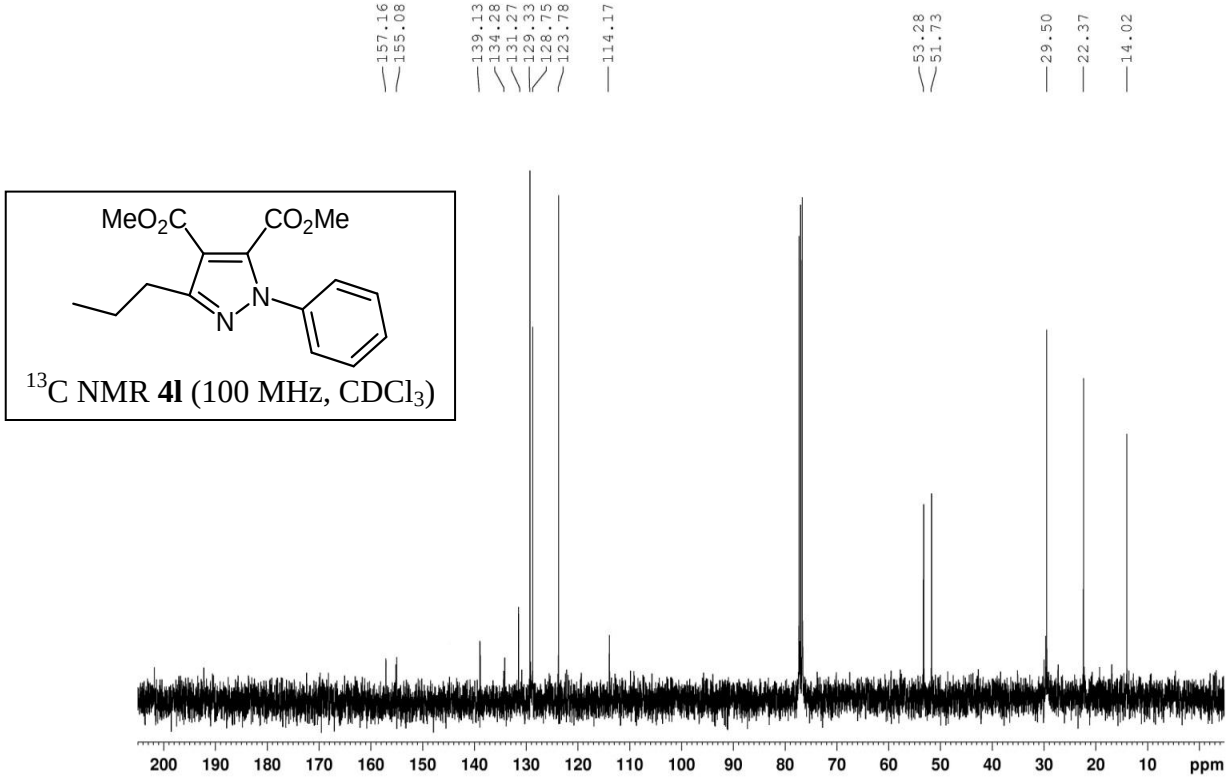




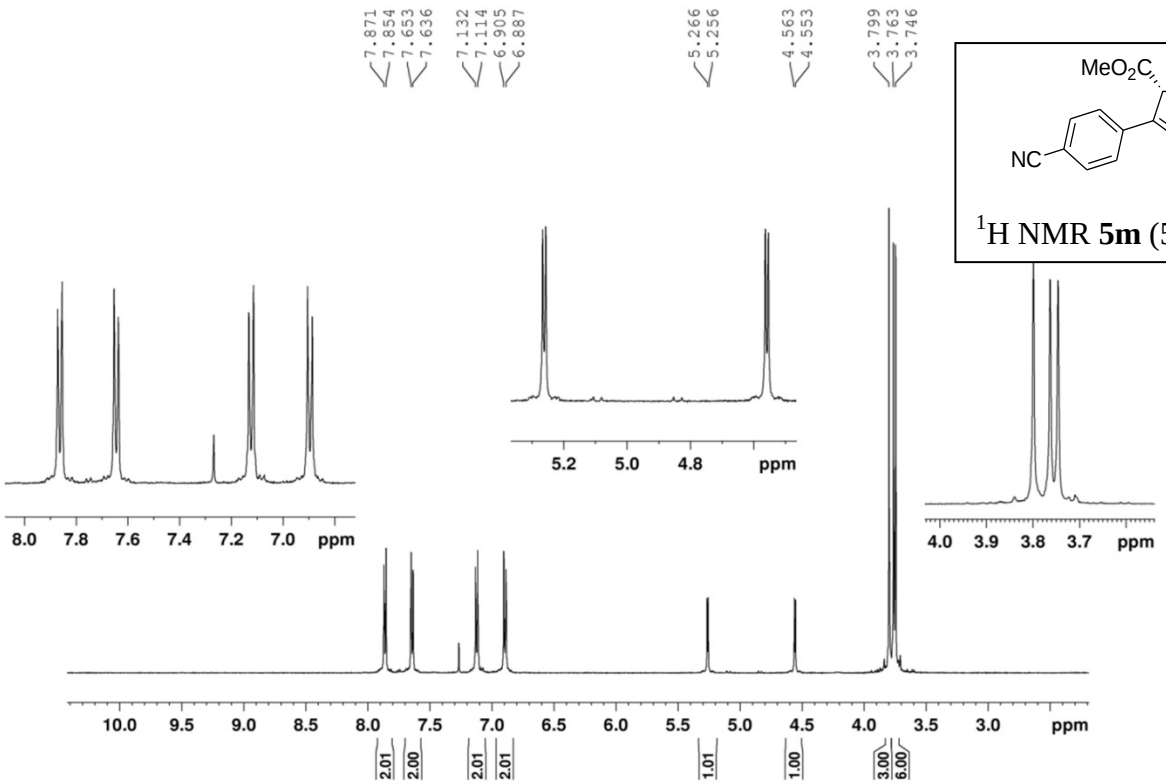
^{13}C NMR **4k** (125 MHz, $CDCl_3$)



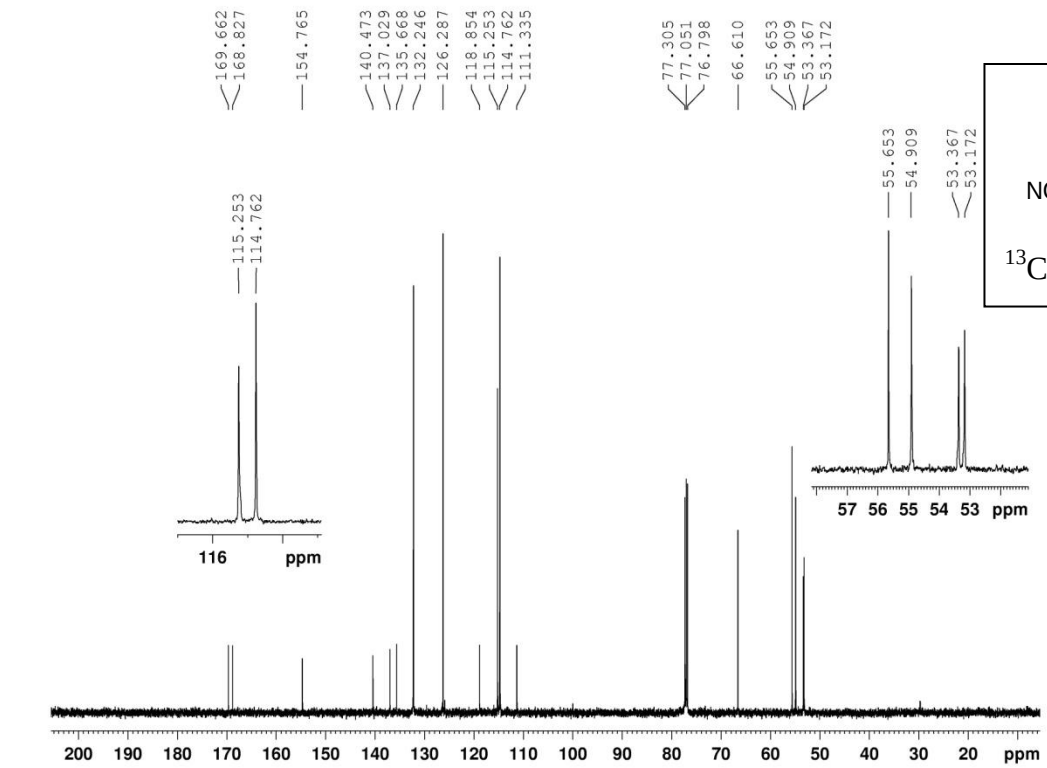
^{1}H NMR **4l** (400 MHz, $CDCl_3$)



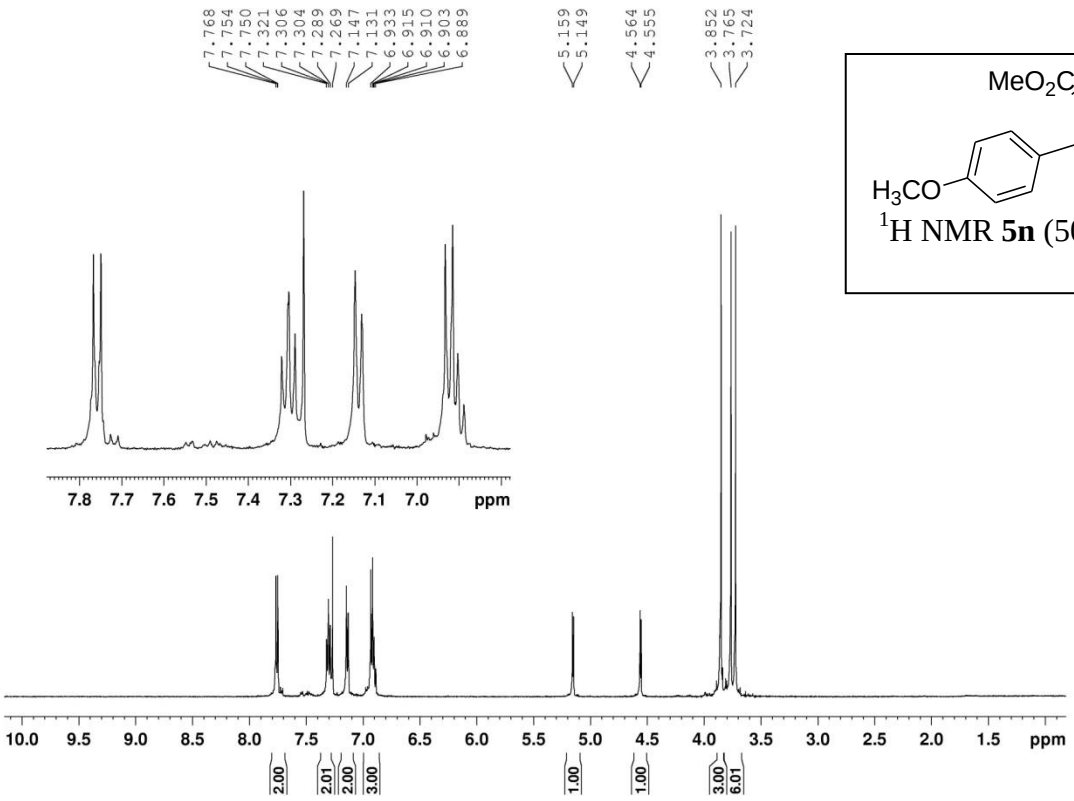
^{13}C NMR **4l** (100 MHz, $CDCl_3$)

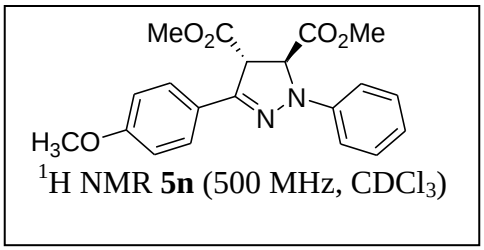


^{1}H NMR **5m** (500 MHz, $CDCl_3$)

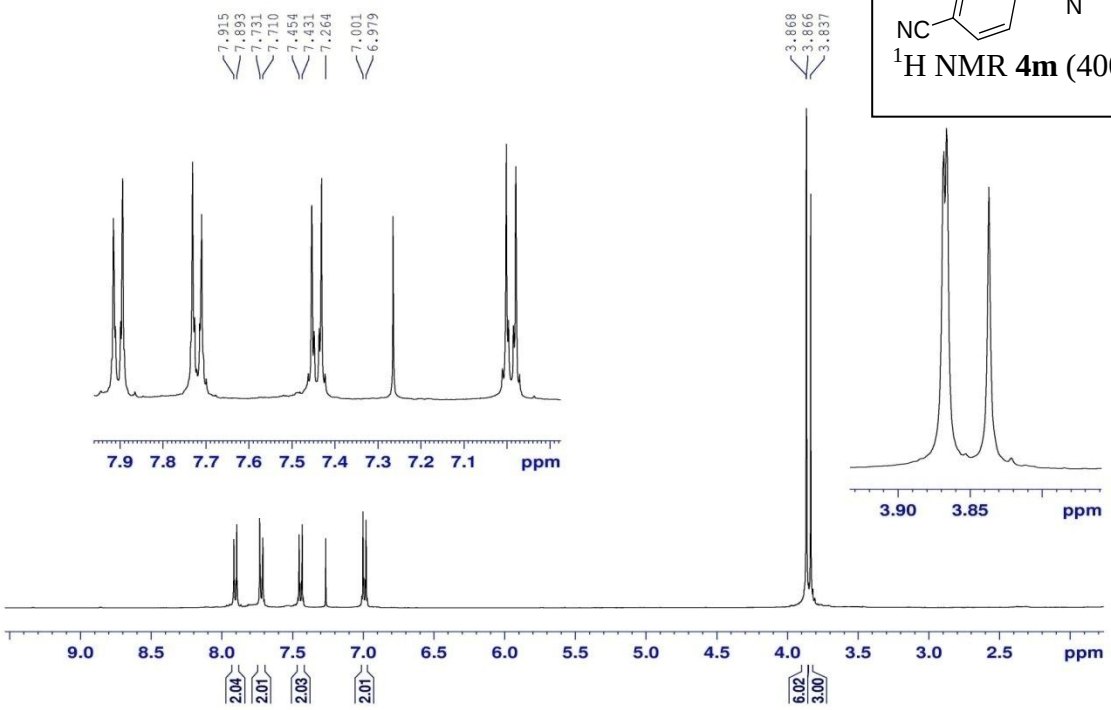


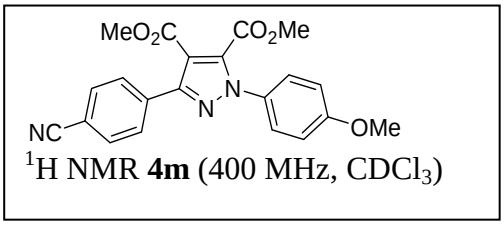
^{13}C NMR **5m** (125 MHz, $CDCl_3$)



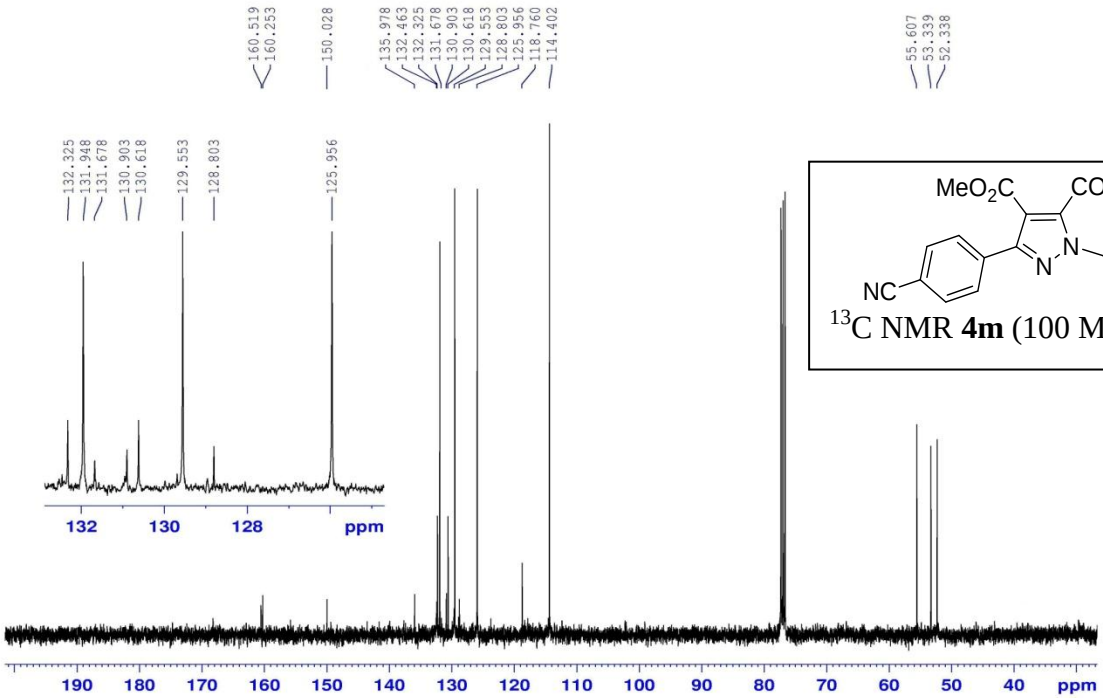


^{1}H NMR **5n** (500 MHz, $CDCl_3$)

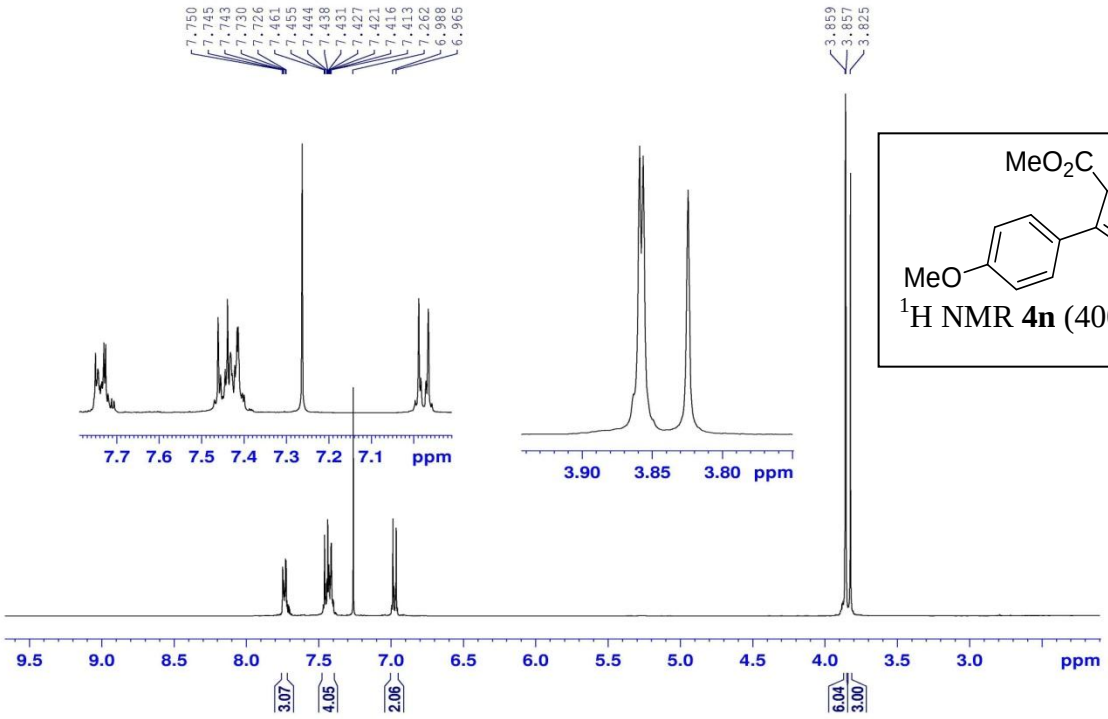




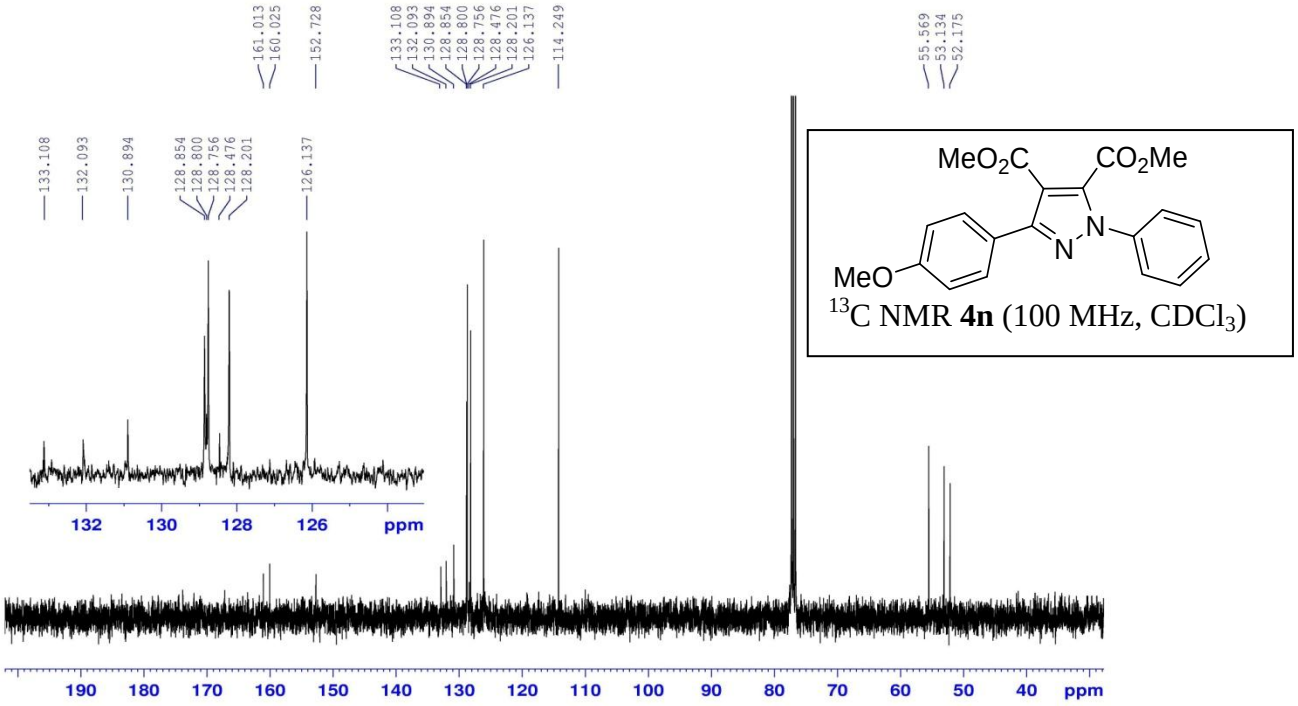
^{1}H NMR **4m** (400 MHz, $CDCl_3$)



^{13}C NMR **4m** (100 MHz, $CDCl_3$)



^{1}H NMR **4n** (400 MHz, $CDCl_3$)



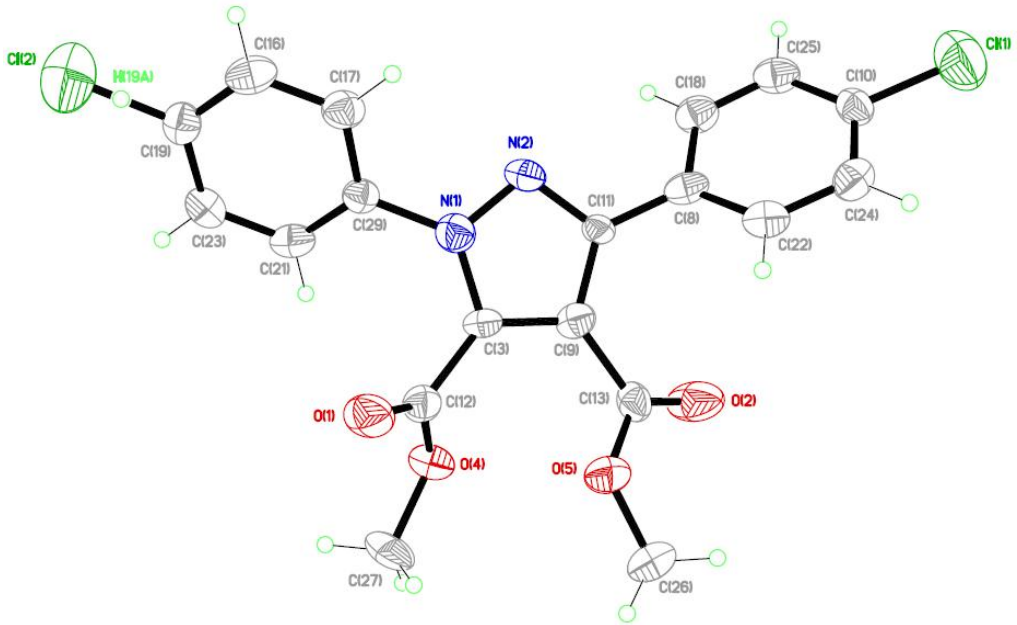


Fig. 1 The crystal structure of compound **4h**

Table 1.Crystal data and structure refinement for compound **4h**.

Identification code	1c	
Empirical formula	$C_{38}H_{28.71}Cl_{3.29}N_4O_8$	
Formula weight	785.99	
Temperature	293(2) K	
Wavelength	0.71069 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 29.363(5) Å	α= 90.000(5)^o
	b = 9.591(5) Å	β= 119.839(5)^o
	c = 15.551(5) Å	γ = 90.000(5)^o
Volume	3799(2) Å^3	
Z	4	
Density (calculated)	1.374 Mg/m^3	
Absorption coefficient	0.318 mm^{-1}	
F(000)	1619	
Crystal size	0.21 x 0.11 x 0.08 mm^3	
Theta range for data collection	2.50 to 29.33°.	
Index ranges	-40< h< 40, -13< k< 10, -18< l< 21	
Reflections collected	12034	
Independent reflections	5004 [R(int) = 0.092]	
Completeness to theta = 29.33°	95.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0000 and 0.9880	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5004 / 0 / 245	
Goodness-of-fit on F^2	0.644	
Final R indices [I>2sigma(I)]	R1 = 0.0578, wR2 = 0.1605	
R indices (all data)	R1 = 0.2934, wR2 = 0.2158	
Largest diff. peak and hole	0.195 and -0.329 e. Å^{-3}	

References:

1) Ma, C.; Li, Y.; Wen, P.; Yan, R.; Ren, Z.; Huang, G. *Synlett***2011**, 1321-1323.

2) Yavari, I.; Khalili, G.; Mirzaei, A. *Helv. Chim. Acta***2010**, *93*, 277-280.

3) Wang, Y.; Vera, C. I. R.; Lin, Q. *Org. Lett*. **2007**, *9*, 4155-4158.