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A Synthetic Route Towards 3, 4-disubstituted Pyrrolidin-2-ones via Michael Addition and Reductive Ring Closing Strategy

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EXPERIMENTAL SECTION

General remarks

Chemicals and solvents received from commercial sources were used without further purification. ^{1}H NMR spectra and ^{13}C NMR spectra were recorded on Bruker (500 MHz and 400 MHz) spectrometer. Coupling constants (*J*) are reported in hertz (Hz), and chemical shifts are reported in parts per million (δ). Melting points were determined using a Thomas Hoover capillary melting point apparatus and uncorrected. Column chromatography was performed using silica gel (100-200 mesh). Exact mass measurements were performed on Bruker impact HD Q-TOF analyser in the ESI mode. IR spectra were recorded by a Shimadzu FT-IR 8400 Spectrometer. The routine monitoring of reactions were performed using TLC (Merck kieselgel 60 0.20 mm layer, UV254). In case of all Michael adducts, exchangeable protons were not observed in ^{1}H NMR in $CDCl_3$.

Synthetic Procedures and Characterization Data

Syntheses of **1** and **2**: The nitroalkenes[1] (**1a-j**) and isoxazolone derivatives[2] (**2a**, **b, c**) were prepared according to literature methods from the corresponding β-keto esters.

General Procedure for the Synthesis of Michael Adducts (3a-o)

Nitroalkene **1** (1.24 mmol), isoxazolone **2** (1.24 mmol), and DABCO (1.24 mmol) were stirred in dry DCM (10 mL) under N_2 at room temperature. The progress of the reaction was monitored by TLC. When the reaction was complete, DCM was evaporated off. The crude sample was purified by column chromatography on silica gel (with petroleum ether/EtOAc or $CHCl_3$/MeOH as the eluent) to afford the corresponding Michael adduct **3**.

4-(2-Nitro-1-phenylethyl)-3-phenyl-1,2-oxazol-5-ol ***(3a)***

[(*E*)-2-nitroethenyl]benzene (**1a**) (185 mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 30 mins. The product was obtained as a brown semisolid (343 mg, 89% yield). R_f 0.56 (pet ether/EtOAc = 50/50); ^{1}H NMR (500 MHz, $CDCl_3$ with DABCO) δ 7.41-7.36 (m, 7H), 7.31-7.28 (m, 2H), 7.23-7.21(m, 1H), 5.49 (dd, *J* = 11.9, 10.4 Hz, 1H), 4.72 (dd, *J* = 12.1, 6.1 Hz, 1H), 4.49 (dd, *J* = 10.2, 6.1 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$ with DABCO) δ 176.6, 165.2, 141.3, 132.2, 131.5, 128.8, 128.5, 128.2, 127.9, 127.1, 82.6, 78.0, 39.6; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.50-7.48

(m, 3H), 7.42-7.38 (m, 4H), 7.32-7.28 (m, 2H), 7.24-7.20 (m, 1H), 6.52 (br s, 1H), 5.33 (dd, *J* = 12.8, 8.8 Hz, 1H), 5.11 (dd, *J* = 12.8, 7.3 Hz, 1H), 4.38 (t, *J* = 8.1 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 171.1, 162.4, 139.0, 131.3, 129.4, 129.0, 127.7, 127.5, 127.4, 126.9, 126.7, 76.3, 38.0; FT-IR (neat) 3064, 2934, 1732, 1556, 1377, 756, 698 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 311.1022, calcd for $C_{17}H_{15}N_2O_4$ 311.1026.

4-[1-(4-Methoxyphenyl)-2-nitroethyl]-3-phenyl-1,2-oxazol-5-ol ***(3b)***

1-Methoxy-4-[(*E*)-2-nitroethenyl]benzene (**1b**) (222 mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 4 h. The product was obtained as a brown semisolid (359 mg, 85% yield). R_f 0.54 (pet ether/EtOAc = 50/50); ^{1}H NMR (500 MHz, $CDCl_3$ with DABCO) δ 7.38-7.34 (m, 7H), 6.83 (d, *J* = 8.7 Hz, 2H), 5.44 (dd, *J* = 11.9, 10.3 Hz, 1H), 4.70 (dd, *J* = 11.9, 6.3 Hz, 1H), 4.44 (dd, *J* = 10.2, 6.3 Hz, 1H), 3.78 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$ with DABCO) δ 176.7, 165.2, 158.6, 133.5, 131.7, 129.0, 128.7, 128.4, 128.1, 114.1, 82.3, 78.2, 55.2, 38.8; FT-IR (neat) 2933, 1721, 1603, 1556, 1514, 1378, 1253, 1174, 1030, 834 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 341.1135, calcd for $C_{18}H_{17}N_2O_5$ 341.1132.

4-[2-Nitro-1-(thiophen-2-yl)ethyl]-3-phenyl-1,2-oxazol-5-ol ***(3c)***

2-[(*E*)-2-nitroethenyl]thiophene (**1c**) (193mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 1 h. The product was obtained as a dark brown semisolid (342 mg, 87% yield). R_f 0.41 (pet ether/EtOAc = 50/50); ^{1}H NMR (500 MHz, $CDCl_3$ with DABCO) δ 7.46 (dd, *J* = 6.5, 2.9 Hz, 2H), 7.39-7.37 (m, 3H), 7.16 (dd, *J* = 5.0, 1.3 Hz, 1H), 6.94-6.92 (m, 2H), 5.41 (dd, *J* = 11.5, 9.5 Hz, 1H), 4.86-4.77 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$ with DABCO) δ 176.3, 164.9, 144.8, 131.4, 128.8, 128.4, 128, 127.1, 124.8, 124.5, 82.6, 77.8, 34.6; FT-IR (neat) 3066, 2923, 1711, 1553, 1426, 1376, 757, 699 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 317.0593, calcd for $C_{15}H_{13}N_2O_4S$ 317.0591.

4-(1-Nitropentan-2-yl)-3-phenyl-1,2-oxazol-5-ol ***(3d)***

(1*E*)-1-nitropent-1-ene (**1d**) (143 mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 24 h. The product was obtained as brown semisolid (182 mg, 53% yield). R_f 0.43 (pet ether/EtOAc = 50/50); ^{1}H NMR (500 MHz, $CDCl_3$ with DABCO) δ

7.51-7.49 (m, 2H), 7.44-7.39 (m, 3H), 4.98 (dd, *J* = 11.3, 9.8 Hz, 1H), 4.47 (dd, *J* = 11.4, 6.2 Hz, 1H), 3.31-3.25 (m, 1H), 1.69-1.63 (m, 1H), 1.40-1.36 (m, 1H), 1.30-1.25 (m, 1H), 1.14-1.12 (m, 1H), 0.75 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$ with DABCO) δ 176.1, 165. 8, 132.2, 129.5, 128.7, 128.5, 128.4, 78.4, 33.6, 33.6, 20.4, 13.9; FT-IR (neat) 3065, 2961, 1697,1549, 1379, 758, 696 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 277.1184, calcd for $C_{14}H_{17}N_2O_4$ 277.1183.

*4-(2-Nitro-1-phenylpropyl)-3-phenyl-1,2-oxazol-5-ol (**3e**)*

[(1*E*)-2-nitroprop-1-en-1-yl]benzene (**1e**) (202 mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 3 h. The product was obtained as orange solid (322 mg, 80% yield). R_f 0.47 (pet ether/EtOAc = 50/50); mp 84-86 °C; ^{1}H NMR (500 MHz, $CDCl_3$ with DABCO) δ 7.36-7.34 (m, 3H), 7.32-7.26 (m, 4H), 7.22-7.19 (m, 2H), 7.17-7.16 (m, 1H), 5.86 (dd, *J* = 11.2, 6.6 Hz, 1H), 3.94 (d, *J* = 11.2 Hz, 1H), 1.21 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$ with DABCO) δ 173.0, 164.1, 139.5, 130.6, 129.2, 129.0, 128.4, 128.2, 94.5, 83.8, 45.7, 19.0; FT-IR (neat) 3064, 2937, 1687,1549, 1414, 1388, 1359, 756, 699 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 325.1179, calcd for $C_{18}H_{17}N_2O_4$ 325.1183.

*Methyl 4-[1-(5-hydroxy-3-phenyl-1,2-oxazol-4-yl)-2-nitroethyl]benzoate (**3f**)*

Methyl 4-[(*E*)-2-nitroethenyl]benzoate (**1f**) (257 mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 3 h. The product was obtained as dark brown semisolid (352 mg, 77% yield). R_f 0.43 (pet ether/EtOAc = 50/50); ^{1}H NMR (500 MHz, $CDCl_3$ with DABCO) δ 7.96 (d, *J* = 8.3 Hz, 2H), 7.48 (d, *J* = 8.3 Hz, 2H), 7.37-7.34 (m, 5H), 5.46 (dd, *J* = 12.2, 9.9 Hz, 1H), 4.77 (dd, *J* = 12.2, 6.5 Hz, 1H), 4.53 (dd, *J* = 9.9, 6.5 Hz, 1H), 3.90 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$ with DABCO) δ 175.6, 166.8, 164.7, 146.0, 130.2, 129.4, 129.2, 128.7, 128.1, 127.9, 84.4, 52.2, 39.4, 29.7; FT-IR (neat) 3060, 2953, 1719,1556,1377, 1279, 1111, 759, 700 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 369.1081, calcd for $C_{19}H_{17}N_2O_6$ 369.1081.

*4-[1-(1,3-Benzodioxol-5-yl)-2-nitroethyl]-3-phenyl-1,2-oxazol-5-ol (**3g**)*

5-[(*E*)-2-nitroethenyl]-1,3-benzodioxole (**1g**) (240 mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 4 h. The product was obtained as brown semisolid (360 mg, 82% yield); R_f 0.41 (pet ether/EtOAc = 50/50); ^{1}H NMR (500 MHz, $CDCl_3$ with DABCO) δ 7.38-7.36 (m, 5H), 7.04 (s, 1H), 6.81 (d, J = 8.0 Hz, 1H), 6.72 (d, J = 8.0 Hz, 1H), 5.91 (dd, J = 3.5, 1.3 Hz, 2H), 5.38 (dd, J = 12.0, 9.9 Hz, 1H), 4.73 (dd, J = 12.0, 6.6 Hz, 1H), 4.38 (dd, J = 9.8, 6.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$ with DABCO) δ 176.5, 165.2, 147.9, 146.6, 135.4, 131.5, 128.9, 128.5, 128.2, 121.1, 108.5, 108.4, 101, 82.9, 78.3, 39.4; FT-IR (neat) 2926, 1724, 1557, 1490, 1377, 1256, 1039, 969, 754, 699 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 355.0928, calcd for $C_{18}H_{15}N_2O_6$ 355.0925.

*4-[1-(4-Chlorophenyl)-2-nitroethyl]-3-phenyl-1,2-oxazol-5-ol (**3h**)*

1-Chloro-4-[(*E*)-2-nitroethenyl]benzene (**1h**) (227 mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 4 h. The product was obtained as orange semisolid (266 mg, 62% yield); R_f 0.38 (pet ether/EtOAc = 50/50); ^{1}H NMR (500 MHz, $CDCl_3$ with DABCO) δ 7.38-7.35 (m, 7H), 7.27-7.25 (m, 2H), 5.41 (dd, J = 12.1, 10.0 Hz, 1H), 4.73 (dd, J = 12.1, 6.5 Hz, 1H), 4.44 (dd, J = 9.9, 6.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$ with DABCO) δ 175.4, 164.6, 139.3, 133.2, 130.0, 129.6, 129.3, 129.1, 128.8, 128.1, 85.7, 85.4, 38.9; FT-IR (neat) 2926, 1714,1553, 1492, 1377, 754, 699 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 345.0636, calcd for $C_{17}H_{14}ClN_2O_4$ 345.0637.

*4-(1-Cyclohexyl-2-nitroethyl)-3-phenyl-1,2-oxazol-5-ol (**3i**)*

[(*E*)-2-nitroethenyl]cyclohexane (**1i**) (192 mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (138 mg, 1.24 mmol) were stirred in DCM (10 mL) for 24 h. The product was obtained as brown semisolid (177 mg, 45% yield); R_f 0.4 (pet ether/EtOAc = 50/50); ^{1}H NMR (500 MHz, $CDCl_3$ without DABCO) δ 7.56-7.52 (m, 5H), 5.09 (dd, J = 14.1, 9.0 Hz, 1H), 4.65 (dd, J = 12.1, 4.3 Hz, 1H), 3.18 (ddd, J = 11.0, 9.3, 4.4 Hz, 1H), 1.80-1.78 (m, 1H), 1.71-1.59 (m, 5H), 1.22-1.14 (m, 2H), 1.06-1.03 (m, 1H), 0.93-0.9 (m, 1H), 0.76-0.69 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$ without DABCO) δ 171.6, 165.2, 131.6, 129.5, 128.1,

126.7, 99.9, 75.5, 39.4, 38.3, 31.33, 31.1, 26, 25.9, 1.1; FT-IR (neat) 3065, 2928, 2853, 1686, 1550, 1379, 758, 698 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 317.1499, calcd for $C_{17}H_{21}N_2O_4$ 317.1496.

*4-[1-(Naphthalen-2-yl)-2-nitroethyl]-3-phenyl-1,2-oxazol-5-ol (**3j**)*

2-[(*E*)-2-nitroethenyl]naphthalene (**1j**) (247 mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (124 mg, 0.62 mmol) were stirred in DCM (10 mL) for 12 h. The product was obtained as brown semisolid (264 mg, 59% yield); R_f 0.44 (pet ether/EtOAc = 50/50); ^1H NMR (500 MHz, $CDCl_3$ with DABCO) δ 7.79-7.77 (m, 4H), 7.58 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.45-7.43 (m, 2H), 7.39-7.37 (m, 2H), 7.34-7.33 (m, 3H), 5.57 (dd, *J* = 12.1, 10.2 Hz, 1H), 4.82 (dd, *J* = 12.1, 6.1 Hz, 1H), 4.66 (dd, *J* = 10.2, 6.1 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$ with DABCO) δ 176.9, 165.6, 138.7, 133.6, 132.6, 131.7, 128.8, 128.6, 128.4, 128.3, 128.0, 127.6, 126.5, 126.3, 126.1, 125.8, 82.0, 78.0, 39.8. FT-IR (neat) 3051, 2925, 1719, 1555, 1376, 753, 479 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 361.1184, calcd for $C_{21}H_{17}N_2O_4$ 361.1183.

*3-Methyl-4-(2-nitro-1-phenylethyl)-1,2-oxazol-5-ol (**3k**)*

[(*E*)-2-nitroethenyl]benzene (**1a**), (185 mg, 1.24 mmol), 3-methylisoxazol-5(4*H*)-one (**2b**) (123 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 3 h. The product was obtained as a yellow semisolid (237 mg, 77% yield). R_f 0.37 (pet ether/EtOAc = 50/50); ^1H NMR (500 MHz, $CDCl_3$ without DABCO) δ 7.41-7.39 (m, 2H), 7.37-7.32 (m, 2H), 7.30-7.29 (m, 1H), 5.37 (dd, *J* = 13.3, 9.4 Hz, 1H), 4.88 (dd, *J* = 13.3, 6.3 Hz, 1H), 4.47 (dd, *J* = 9.4, 6.3 Hz, 1H), 2.18 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$ without DABCO) δ 171.6, 162.2, 137.8, 129.2, 128.0, 127.5, 99.3, 76.1, 38.8, 10.8; FT-IR (neat) 3062, 2927, 1719, 1554, 1455, 1377, 1261, 754, 701 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 249.0869, calcd for $C_{12}H_{13}N_2O_4$ 249.0870.

*4-[1-(4-Methoxyphenyl)-2-nitroethyl]-3-methyl-1,2-oxazol-5-ol (**3l**)*

1-Methoxy-4-[(*E*)-2-nitroethenyl]benzene (**1b**) (222 mg, 1.24 mmol), 3-methylisoxazol-5(4*H*)-one (**2b**) (123 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 3 h. The product was obtained as a yellow semisolid (252 mg, 73% yield). R_f 0.50 (pet ether/EtOAc = 50/50); ^1H NMR (500 MHz, $CDCl_3$ without DABCO) δ 7.29 (d, *J* = 8.7 Hz, 2H), 6.85 (d, *J* = 8.7 Hz, 2H), 5.27 (dd, *J* = 13.1, 9.3 Hz, 1H),

4.83 (dd, *J* = 13.1, 6.7 Hz, 1H), 4.40 (dd, *J* = 9.2, 6.7 Hz, 1H), 3.77 (s, 3H), 2.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$ without DABCO) δ 172.2, 161.9, 159.4, 130.2, 128.7, 114.7, 97.9, 96.2, 55.4, 38.2, 10.7; FT-IR (neat) 2937, 1716, 1602, 1552, 1511, 1377, 1249, 1027, 832, 756 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 279.0974, calcd for $C_{13}H_{15}N_2O_5$ 279.0975.

*3-Methyl-4-[2-nitro-1-(thiophen-2-yl)ethyl]-1,2-oxazol-5-ol (**3m**)*

2-[(*E*)-2-nitroethenyl]thiophene (**1c**) (193 mg, 1.24 mmol), 3-methylisoxazol-5(4*H*)-one (**2b**) (123 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 1 h. The product was obtained as a greenish yellow semisolid (256 mg, 81% yield). R_f 0.40 (pet ether/EtOAc = 50/50); ^{1}H NMR (500 MHz, $CDCl_3$ without DABCO) δ 7.44 (br s, 1H), 7.16 (d, *J* = 5.0 Hz, 1H), 6.98 (d, *J* = 3.2 Hz, 1H), 6.92-6.9 (m, 1H), 5.20 (dd, *J* = 12.9, 9.6 Hz, 1H), 4.87 (dd, *J* = 13.1, 6.3 Hz, 1H), 4.74 (dd, *J* = 9.1, 6.5 Hz, 1H), 2.15 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$ without DABCO) δ 172.1, 161.4, 140.5, 127.4, 125.5, 125.1, 94.7, 76.6, 33.7, 10.6; FT-IR (neat) 3106, 2958, 1721, 1555, 1378, 1214, 751, 668 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 255.0438, calcd for $C_{10}H_{11}N_2O_4S$ 255.0434.

3-(4-chlorophenyl)-4-(2-nitro-1-phenylethyl)-1,2-oxazol-5-ol (***3n***)

[(*E*)-2-nitroethenyl]benzene (**1a**) (184 mg, 1.24 mmol), 3-(4-chlorophenyl)isoxazol-5(4*H*)-one (**2c**) (242 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 30 mins. The product was obtained as a yellow semisolid (340 mg, 79% yield). R_f 0.29 ($CHCl_3$/MeOH = 98/02); ^{1}H NMR (500 MHz, $CDCl_3$ with DABCO) δ 7.39-7.38 (m, 3H), 7.32 -7.27 (m, 5H), 7.23-7.2 (m, 1H), 5.52 (t, J = 11.4 Hz, 1H), 4.67 (dd, J = 11.5, 5.8 Hz, 1H), 4.43 (dd, J = 11, 5.8 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$ with DABCO) δ 176.9, 164.5, 141.1, 134.8, 131.2, 129.6, 128.9, 128.7, 128, 127.3, 82.1, 39.7, 29.8; FT-IR (neat) 2937, 1747, 1554, 1350 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 345.0635, calcd for $C_{17}H_{14}ClN_2O_4$ 345.0637.

*4-(2-Nitro-1,2,3,4-tetrahydronaphthalen-1-yl)-3-phenyl-1,2-oxazol-5-ol (**3o**)*

3-Nitro-1,2-dihydronaphthalene (**1k**) (217 mg, 1.24 mmol), 3-phenylisoxazol-5(4*H*)-one (**2a**) (200 mg, 1.24 mmol), DABCO (139 mg, 1.24 mmol) were stirred in DCM (10 mL) for 24 h. The product was obtained as yellow solid (321 mg, 77% yield). R_f 0.45 (pet ether/EtOAc = 50/50); mp 145-147 °C; ^{1}H NMR (400 MHz,

$CDCl_3$ without DABCO) δ 7.59-7.5 (m, 5H), 7.19-7.12 (m, 3H), 6.98 (d, J = 7.4 Hz, 1H), 5.52 (td, J= 11.6, 3.5 Hz, 1H), 4.64 (d, J = 10.4 Hz, 1H), 3.16-3.09 (m, 1H), 3.03-2.99 (m, 1H), 2.58-2.55 (m, 1H), 2.40-2.30 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$ without DABCO) δ 170.6, 165.5, 134.9, 133.4, 132.1, 129.7, 129.0, 127.8, 127.4, 127.1, 126.5, 102.1, 84.4, 38.7, 28.7, 28.0; FT-IR (neat) 3065, 2956, 1709, 1547,1374,758, 698 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 337.1184, calcd for $C_{19}H_{17}N_2O_4$ 337.1183.

General Procedure for Reductive Cyclization (4a-n and 5)

To a stirred solution of Michael adduct **3** (0.5 mmol) in acetic acid (10 mL) was added zinc dust (5 mmol). The reaction mixture was stirred for 1 h at room temperature. After disappearance of the starting material (monitored by TLC), the reaction mixture was subjected for heating at 80 °C for 24 h and then filtered through celite. The reaction mixture was evaporated under reduced pressure to remove acetic acid. The residue was quenched with water, washed with saturated $NaHCO_3$ solution and extracted with ethyl acetate, dried over sodium sulfate and concentrated. Purification was done by column chromatography using chloroform/ MeOH as the eluent.

*(3S,4S)-3-Benzoyl-4-phenylpyrrolidin-2-one (**4a**)*

Michael adduct **3a** (170 mg) was used. The product was obtained as a brown solid (80 mg, 55% yield). >99:1 dr [determined by ^{1}H NMR]; R_f 0.59 ($CHCl_3$/MeOH = 98/02); mp 156-158 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.05-8.03 (m, 2H), 7.58-7.55 (m, 1H), 7.48-7.44 (m, 2H), 7.34-7.32 (m, 2H), 7.29-7.26 (m, 3H), 6.16 (br s, 1H), 4.53 (d, J = 7.0 Hz, 1H), 4.33 (dd, J = 15.0, 7.0 Hz, 1H), 3.96-3.89 (m, 1H), 3.53 (dd, J = 9.6, 6.9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.1, 173.6, 141.4, 136.3, 133.6, 129.5, 129.1, 128.6, 127.5,127 57.5, 47.7, 43.4; FT-IR (neat) 3240, 2927, 1701, 1674, 1494, 1270, 1054, 757,698 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 266.1174, calcd for $C_{17}H_{16}NO_2$ 266.1176.

*(3S,4S)-3-Benzoyl-4-(4-methoxyphenyl)pyrrolidin-2-one (**4b**)*

Michael adduct **3b** (178 mg) was used. The product was obtained as a brown solid (85 mg, 55% yield). >99:1 dr [determined by ^{1}H NMR]; R_f 0.55 ($CHCl_3$/MeOH = 98/02); mp 128-130 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 (d, J = 8.24 Hz, 2H), 7.55-7.58 (m, 1H), 7.47-7.44 (m, 2H), 7.19 (d, J = 8.5 Hz, 2H), 6.85 (d, J = 8.5 Hz,

2H), 5.96 (br s, 1H), 4.49 (d, J = 7.5 Hz, 1H), 4.27 (dd, J = 14.9, 7.5 Hz, 1H), 3.91-3.88 (m, 1H), 3.78 (s, 3H), 3.48 (dd, J = 9.6, 6.7 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.3, 173.0, 158.8, 136.4, 133.6, 133.4, 129.5, 128.6, 128.0, 114.4, 57.8, 55.3, 48.0, 42.8; FT-IR (neat) 3263, 2973, 1705, 1676, 1515, 1448, 1250, 1033, 766 cm^{-1}; HRMS (ESI) $[M + Na]^+$ found *m/z* 318.1100, calcd for $C_{18}H_{17}NNaO_3$ 318.1101.

*(3S,4R)-3-Benzoyl-4-(thiophen-2-yl)pyrrolidin-2-one (**4c**)*

Michael adduct **3c** (175 mg) was used. The product was obtained as a brown solid (78 mg, 52% yield). >99:1 dr [determined by ^{1}H NMR]; R_f 0.53 ($CHCl_3$/MeOH = 98/02); mp 77-79 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 (dd, J = 8.4, 1.2 Hz, 2H), 7.60-7.57 (m, 1H), 7.49-7.46 (m, 2H), 7.17 (dd, J = 4.8, 1.5 Hz, 1H), 6.93-6.9 (m, 2H), 6.62 (br s, 1H), 4.64 (dd, J = 15.3, 7.6 Hz, 1H), 4.54 (d, J = 7.9 Hz, 1H), 3.94-3.91 (m, 1H), 3.56 (dd, J = 9.6, 7.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 194.6, 172.4, 143.9, 136.3, 133.7, 129.5, 128.6, 127.1, 124.6 , 124.2, 58.4, 48.2, 39; FT-IR (neat) 3225, 2924, 1704, 1677, 1448, 1261, 1029, 750, 699 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 272.0741, calcd for $C_{15}H_{14}NO_2S$ 272.0740.

*(3S,4R)-3-Benzoyl-4-propylpyrrolidin-2-one (**4d**)*

Michael adduct **3d** (155 mg) was used. The product was obtained as a yellow solid (61 mg, 47% yield). >99:1 dr [determined by ^{1}H NMR]; R_f 0.56 ($CHCl_3$/MeOH = 98/02); mp >193 °C (compound decomposes above); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08-8.06 (m, 2H), 7.61-7.57 (m, 1H), 7.51-7.48 (m, 2H), 6.40 (br s, 1H), 4.12 (d, J = 7.02 Hz, 1H), 3.64 (t, J = 8.24 Hz, 1H), 3.12-3.05 (m, 2H), 1.49 (q, J = 7.32 Hz, 2H), 1.29-1.24 (m, 2H), 0.88 (t, J = 7.32 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.3, 174.0, 136.8, 133.6, 129.4, 128.7, 56.4, 46.6, 38.5, 36.5, 20.7, 14.1; FT-IR (neat) 3019, 2928, 1703, 1677, 1448, 1214, 1033, 748, 667 cm^{-1}; HRMS (ESI) $[M + Na]^+$ found *m/z* 254.1159, calcd for $C_{14}H_{17}NNaO_2$ 254.1151.

*(3S,4S,5S)-3-Benzoyl-5-methyl-4-phenylpyrrolidin-2-one (**4e**)*

Michael adduct **3e** (160 mg) was used. The product was obtained as a brown solid (85 mg, 62% yield). 65:15:12:8 dr [determined by ^{1}H NMR]; R_f 0.43 ($CHCl_3$/MeOH = 98/02); mp 92-94 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99-7.97 (m, 2H), 7.55-7.52 (m, 1H), 7.44-7.41 (m, 2H), 7.33-7.29 (m, 4H), 7.27-7.25 (m, 1H), 6.07 (br s, 1H), 4.65 (d, J = 9.4 Hz, 1H), 3.92-3.83 (m, 2H), 1.34 (d, J = 5.9 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ

196.1, 171.7, 139.6, 136.8, 133.6, 129.6, 129.1, 128.6, 127.8, 127.7, 58.7, 56.5, 52, 20.3; FT-IR (neat) 3019, 1706, 1680, 1214, 746, 668 cm^{-1}; HRMS (ESI) $[M + Na]^+$ found *m/z* 280.1337, calcd for $C_{18}H_{18}NO_2$ 280.1332.

*Methyl 4-[(3S,4S)-4-benzoyl-5-oxopyrrolidin-3-yl]benzoate (**4f**)*

Michael adduct **3f** (185 mg) was used. The product was obtained as an orange solid (84 mg, 52% yield). 94:6 dr [determined by ^1H NMR]; R_f 0.38 ($CHCl_3$: MeOH = 98/02); mp 100-102 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.05-8.03 (m, 2H), 8.00 (d, J = 8.4 Hz, 2H), 7.58 (t, J = 7.5, 1H), 7.47 (t, J = 7.8 Hz, 2H), 7.35 (d, J = 8.3 Hz, 2H), 6.15 (br s, 1H), 4.52 (d, J = 7.2 Hz, 1H), 4.41 (dd, J = 14.9, 6.9 Hz, 1H), 3.97 (d, J = 8.7 Hz, 1H), 3.90 (s, 3H), 3.54 (dd, J = 9.6, 6.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 194.7, 172.3, 166.6, 146.5, 136.1, 133.8, 130.4, 129.5, 129.4, 128.6, 127.1, 57.3, 52.2, 47.4, 43.2; FT-IR (neat) 3020, 1720, 1711, 1284, 1054, 743, 668 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 324.1233, calcd for $C_{19}H_{18}NO_4$ 324.1230.

*(3S,4S)-4-(1,3-Benzodioxol-5-yl)-3-benzoylpyrrolidin-2-one (**4g**)*

Michael adduct **3g** (176 mg) was used. The product was obtained as a pink solid (84 mg, 55% yield) 83:17 dr [determined by ^1H NMR]; R_f 0.36 ($CHCl_3$/MeOH = 98/02); mp 160-162 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.03 (dd, J = 8.3, 1.1 Hz, 2H), 7.60-7.56 (m, 1H), 7.48-7.45 (m, 2H), 6.77 (d, J = 1.1 Hz, 1H), 6.73-6.72 (m, 2H), 6.45 (br s, 1H), 5.93 (s, 2H), 4.47 (d, J = 7.4 Hz, 1H), 4.25 (dd, J = 15.0, 7.3 Hz, 1H), 3.89 (d, J = 9.0 Hz, 1H), 3.47 (dd, J = 9.6, 6.7 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.3, 173.0, 148.3, 147.0, 136.5, 135.2, 133.8, 130.2, 129.6, 128.7, 120.5, 108.7, 107.37, 101.3, 57.9, 48.15, 43.4; FT-IR (neat) 3020, 1708, 1679, 1506, 1479, 1214, 1055, 1033, 743, 668 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 310.1075, calcd for $C_{18}H_{16}NO_4$ 310.1074.

*(3S,4S)-3-Benzoyl-4-(4-chlorophenyl)pyrrolidin-2-one (**4h**)*

Michael adduct **3h** (172 mg) was used. The product was obtained as a yellow solid (87 mg, 58% yield). 81:19 dr [determined by ^1H NMR]; R_f 0.49 ($CHCl_3$/MeOH = 98/02); mp 116-118 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.04-8.03 (m, 2H), 7.60-7.57 (m, 1H), 7.47 (t, J = 7.8 Hz, 2H), 7.30 (d, J = 8.5 Hz, 2H), 7.22 (d, J = 8.5 Hz, 2H), 6.32 (br s, 1H), 4.48 (d, J = 7.3 Hz, 1H), 4.33 (dd, J = 14.8, 7.2 Hz, 1H), 3.94 (t, J = 8.9 Hz, 1H), 3.50 (dd, J = 9.7, 6.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.8, 139.9, 136.3, 133.9, 133.5, 129.8, 129.3, 128.8,

128.5, 57.6, 47.8, 42.8; FT-IR (neat) 3019, 2924, 1703, 1677, 1493, 1214, 1054, 1033, 751, 668 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 300.0787, calcd for $C_{17}H_{15}ClNO_2$ 300.0786.

*(3S,4R)-3-Benzoyl-4-cyclohexylpyrrolidin-2-one (**4i**)*

Michael adduct **3i** (160 mg) was used. The product was obtained as orange yellow solid (56 mg, 41% yield). >99:1 dr [determined by ^1H NMR]; R_f 0.51 ($CHCl_3$/MeOH = 98/02); mp 135-137 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.10-8.08 (m, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 2H), 6.05 (br s, 1H), 4.22 (d, *J* = 8.5 Hz, 1H), 3.60 (t, *J* = 8.8 Hz, 1H), 3.17-3.07 (m, 2H), 1.74-1.62 (m, 5H), 1.39-1.30 (m, 2H), 1.21-1.10 (m, 2H), 1.03-0.97 (m, 1H), 0.91-0.88 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 196.4, 173.9, 137.0, 133.5, 129.5, 128.7, 54.1, 44.9, 44.2, 42.2, 31.5, 30.6, 26.2, 26.1, 26.0; FT-IR (neat) 3225, 3017, 2924, 1701, 1678, 1448, 1339, 1214, 1055, 1033, 753, 667 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 272.1645, calcd for $C_{17}H_{22}NO_2$ 272.1645.

*(3S,4S)-3-Benzoyl-4-(naphthalen-2-yl)pyrrolidin-2-one (**4j**)*

Michael adduct **3j** (182 mg) was used. The product was obtained as a dark brown solid (70 mg, 44% yield). >99:1 dr [determined by ^1H NMR]; R_f 0.44 $CHCl_3$/MeOH = 98/02); mp 142-145 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 8.05-8.03 (m, 2H), 7.84-7.77 (m, 3H), 7.72 (d, *J* = 0.7 Hz, 1H), 7.57-7.54 (m, 1H), 7.49-7.4 (m, 5H), 6.32 (br s, 1H), 4.64 (d, *J* = 7.1 Hz, 1H), 4.5 (dd, *J* = 14.9, 6.7 Hz, 1H), 4.03-3.99 (m, 1H), 3.64 (dd, *J* = 9.5, 6.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 195.3, 173.1, 138.8, 136.4, 133.8, 133.5, 132.7, 129.6, 129.2, 128.7, 127.8, 127.7, 126.6, 126.2, 125.9, 124.9, 57.7, 48.0, 43.7; FT-IR (neat) 3226, 3057, 2923, 1701, 1674, 1487, 1268, 1033, 748, 687 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 316.1333, calcd for $C_{21}H_{18}NO_2$ 316.1332.

*(3S,4S)-3-Acetyl-4-phenylpyrrolidin-2-one (**4k**)*

Michael adduct **3k** (127 mg) was used. The product was obtained as a brown solid (58 mg, 56% yield). >99:1 dr [determined by ^1H NMR]; R_f 0.46 ($CHCl_3$/MeOH = 98/02); mp 111-113 °C; ^1H NMR (500 MHz, $CDCl_3$) δ 7.35-7.32 (m, 2H), 7.28-7.25 (m, 3H), 6.53 (br s, 1H), 4.19 (dd, *J* = 15.6, 8.2 Hz, 1H), 3.78-3.76 (m, 1H), 3.67 (d, *J* = 8.1 Hz, 1H), 3.42 (dd, *J* = 9.7, 7.2 Hz, 1H), 2.41 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 202.5, 172.5, 141.2, 129, 127.4, 127.1, 62.4, 47.5, 41.3, 30.6; FT-IR (neat) 3284, 2923, 1688, 1492, 1260, 1052, 754, 698 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 204.1019, calcd for $C_{12}H_{14}NO_2$ 204.1019.

*(3S,4S)-3-Acetyl-4-(4-methoxyphenyl)pyrrolidin-2-one (**4l**)*

Michael adduct **3l** (152 mg) was used. The product was obtained as a buff white solid (64 mg, 50% yield). >99:1 dr [determined by ^{1}H NMR]; R_f 0.59 ($CHCl_3$/MeOH = 98/02); mp 110-112 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.18 (d, J = 8.7 Hz, 2H), 6.87 (d, J = 8.7 Hz, 2H), 6.50 (br s, 1H), 4.14 (dd, J = 15.9, 8.2 Hz, 1H), 3.80 (s, 3H), 3.75 (t, J = 9.2 Hz, 1H), 3.63 (d, J = 8.3 Hz, 1H), 3.39 (dd, J = 9.7, 7.4 Hz, 1H), 2.40 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 202.7, 172.6, 133.1, 158. 8, 133.1, 128.1, 114.3, 62.6, 55.3, 47.6, 40.7, 30.7; FT-IR (neat) 3261, 2956, 1687, 1514, 1441, 1246, 1031, 828 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 234.1127, calcd for $C_{13}H_{16}NO_3$ 234.1125.

*(3S,4R)-3-Acetyl-4-(thiophen-2-yl)pyrrolidin-2-one (**4m**)*

Michael adduct **3m** (135 mg) was used. The product was obtained as a brown semisolid (52 mg, 47% yield). >99:1 dr [determined by ^{1}H NMR]; R_f 0.61 ($CHCl_3$/MeOH = 98/02); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.19 (dd, J = 5.1, 1.1 Hz, 1H), 6.94 (dd, J = 5.1, 3.5 Hz, 1H), 6.91 (d, J = 3.4 Hz, 1H), 5.96 (br s, 1H), 4.49 (dd, J = 16.1, 8.0 Hz, 1H), 3.80 (t, J = 9.0 Hz, 1H), 3.65 (d, J = 8.3 Hz, 1H), 3.46 (dd, J = 9.5, 7.6 Hz, 1H), 2.44 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 202.1, 171.6, 144.1, 127.2, 124.7, 124.3, 63, 47.7, 37, 30.8; FT-IR (neat) 3262, 3018, 2923, 1697, 1486, 1215, 1055, 749, 668 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 210.0588, calcd for $C_{10}H_{12}NO_2S$ 210.0583.

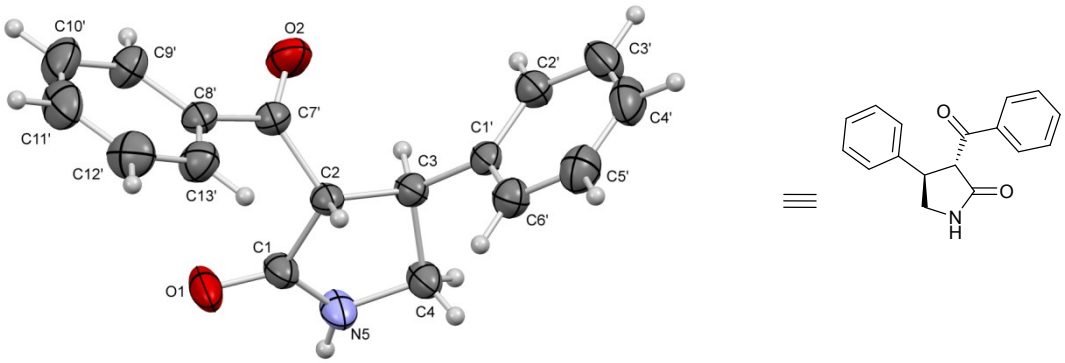
(3S,4S)-3-(4-chlorobenzoyl)-4-phenylpyrrolidin-2-one (**4n**)

Michael adduct **3n** (173 mg) was used. The product was obtained as a yellow semisolid (77 mg, 51% yield). >99:1 dr [determined by ^{1}H NMR]; R_f 0.33 ($CHCl_3$/MeOH = 98/02); ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.98 (d, J = 8.5 Hz, 2H), 7.43 (d, J = 8.4 Hz, 2H), 7.39-7.31 (m, 3H), 7.28-7.27 (m, 2H), 6.18 (br s, 1H), 4.46 (d, J = 7.3 Hz, 1H), 4.34 (dd, J = 14.8, 7.4 Hz, 1H), 3.93 (t, J = 9.0 Hz, 1H), 3.53 (dd, J = 9.6, 6.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 193.9, 172.6, 141.3, 134.7, 131.1, 129.7, 129.2, 129.0, 127.7, 127.1, 57.8, 47.9, 43.2; FT-IR (neat) 3032, 2955, 1708, 1489, 1220, 1014, 1033, 759 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 300.0785, calcd for $C_{17}H_{15}ClNO_2$ 300.0786.

*2-Phenyl-3H-benzo[e]indole (**5**)*[3]

Michael adduct **3n** (178 mg, 0.5 mmol) was reacted with Zn (0.346 mg, 5 mmol) according to the general procedure for reductive cyclization. After heating the reaction mass at 80 °C for 12 h, Zn was removed and the mixture was further refluxed in AcOH for 48 h in open air. After usual work up; the mixture was purified by column chromatography. The product was obtained as a white solid (80 mg, 62% yield). R_f 0.55 (pet ether/EtOAc = 90/10); mp 135-137 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.67 (br s, 1H), 8.27 (d, J = 7.7 Hz, 1H), 7.90 (d, J = 8.1 Hz, 1H), 7.60-7.55 (m, 2H), 7.62-7.53 (m, 3H), 7.48-7.41 (m, 3H), 7.38-7.37 (m, 1H), 7.34-7.31 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136, 133.2, 132.4, 129.3, 129.2, 128.6, 128, 127.3, 125.8, 124.8, 124.3, 123.5, 123.3, 122.9, 112.5, 99.3; FT-IR (neat) 3372, 3019, 2927, 1719, 1604, 1284, 1214, 1074, 754 cm^{-1}; HRMS (ESI) $[M + H]^+$ found *m/z* 244.1125, calcd for $C_{18}H_{14}N$ 244.1121.

Crystal data and structure of 4a



Supplementary crystallographic data for the compound **4a** (CCDC **1489354**) can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

Crystal data and structure refinement for4a

Identification code	cu_M35_R_0m	
Empirical formula	$C_{17}H_{15}NO_2$	
Formula weight	265.30	
Temperature	296(2) K	
Wavelength	1.54178 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 5.6408(8) Å	alpha= 74.130(5)°.
	b = 10.3136(1) Å	beta= 89.422(6)°.
	c = 12.233(2) Å	gamma= 86.861(6)°.
Volume	683.52(16) $Å^3$	
Z	2	
Density (calculated)	1.289 Mg/m^3	
Absorption coefficient	0.679 mm^{-1}	
F(000)	496	
Crystal size	0.49 x 0.47 x 0.45mmm^3	
Theta range for data collection	2.72 to 25.00°.	
Index ranges	6<=h<=6, 11<=k<=11, 27<=l<=27	
Reflections collected	25501	
Independent reflections	2073 [R(int) = 0.0365]	
Completeness to theta =	25.00° 99.8 %	
Max. and min. transmission	0.9594 and 0.9561	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2424 / 0 / 181	
Goodness-of-fit on F^2	1.157	

Final R indices [I>2sigma(I)]	R1 = 0.0817, wR2 = 0.2499
R indices (all data)	R1 = 0.0979, wR2 = 0.2749
Extinction coefficient	n/a
Largest diff. peak and hole	0.318 and -0.398 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for cu_m35_r_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3313(4)	5560(2)	3620(2)	61(1)
O(2)	-1800(3)	3674(2)	2807(2)	54(1)
N(5)	4174(4)	3434(2)	4785(2)	45(1)
C(1)	3314(5)	4328(2)	3848(2)	40(1)
C(2)	2383(4)	3535(2)	3066(2)	31(1)
C(3)	2251(4)	2104(2)	3844(2)	33(1)
C(4)	4179(5)	2056(2)	4742(2)	40(1)
C(1')	2554(4)	980(2)	3274(2)	33(1)
C(2')	893(5)	-4(2)	3439(2)	41(1)
C(3')	1183(5)	-1061(3)	2956(3)	55(1)
C(4')	3130(6)	-1164(3)	2293(2)	54(1)
C(5')	4786(6)	-188(3)	2114(2)	53(1)
C(6')	4519(5)	862(3)	2606(2)	42(1)
C(7')	49(4)	4177(2)	2492(2)	34(1)
C(8')	134(4)	5437(2)	1556(2)	35(1)
C(9')	-1831(5)	6357(3)	1374(2)	46(1)
C(10')	-1871(6)	7508(3)	488(3)	58(1)
C(11')	25(6)	7770(3)	-248(2)	58(1)
C(12')	1980(6)	6878(3)	-81(2)	58(1)
C(13')	2031(5)	5726(3)	820(2)	45(1)

Table 3. Bond lengths [Å] and angles [°] for cu_m35_r_0m.

O(1)-C(1)	1.224(3)
O(2)-C(7')	1.203(3)
N(5)-C(1)	1.336(3)
N(5)-C(4)	1.436(3)
N(5)-H(5)	0.8600
C(1)-C(2)	1.531(3)
C(2)-C(7')	1.528(3)
C(2)-C(3)	1.528(3)
C(2)-H(2)	0.9800
C(3)-C(1')	1.508(3)
C(3)-C(4)	1.543(3)
C(3)-H(3)	0.9800
C(4)-H(4A)	0.9700
C(4)-H(4B)	0.9700
C(1')-C(6')	1.390(3)
C(1')-C(2')	1.392(3)
C(2')-C(3')	1.377(4)
C(2')-H(2')	0.9300
C(3')-C(4')	1.375(4)
C(3')-H(3')	0.9300
C(4')-C(5')	1.383(5)
C(4')-H(4')	0.9300
C(5')-C(6')	1.376(4)
C(5')-H(5')	0.9300
C(6')-H(6')	0.9300
C(7')-C(8')	1.481(3)
C(8')-C(13')	1.383(3)
C(8')-C(9')	1.398(3)
C(9')-C(10')	1.371(4)
C(9')-H(9')	0.9300
C(10')-C(11')	1.380(4)
C(10')-H(10')	0.9300
C(11')-C(12')	1.377(4)
C(11')-H(11')	0.9300
C(12')-C(13')	1.381(4)
C(12')-H(12')	0.9300

C(13')-H(13')	0.9300
C(1)-N(5)-C(4)	114.0(2)
C(1)-N(5)-H(5)	123.0
C(4)-N(5)-H(5)	123.0
O(1)-C(1)-N(5)	127.3(2)
O(1)-C(1)-C(2)	125.1(2)
N(5)-C(1)-C(2)	107.64(19)
C(7')-C(2)-C(3)	115.36(19)
C(7')-C(2)-C(1)	111.63(18)
C(3)-C(2)-C(1)	103.62(17)
C(7')-C(2)-H(2)	108.7
C(3)-C(2)-H(2)	108.7
C(1)-C(2)-H(2)	108.7
C(1')-C(3)-C(2)	115.93(18)
C(1')-C(3)-C(4)	113.76(18)
C(2)-C(3)-C(4)	102.30(18)
C(1')-C(3)-H(3)	108.2
C(2)-C(3)-H(3)	108.2
C(4)-C(3)-H(3)	108.2
N(5)-C(4)-C(3)	102.95(18)
N(5)-C(4)-H(4A)	111.2
C(3)-C(4)-H(4A)	111.2
N(5)-C(4)-H(4B)	111.2
C(3)-C(4)-H(4B)	111.2
H(4A)-C(4)-H(4B)	109.1
C(6')-C(1')-C(2')	117.9(2)
C(6')-C(1')-C(3)	121.7(2)
C(2')-C(1')-C(3)	120.4(2)
C(3')-C(2')-C(1')	121.1(3)
C(3')-C(2')-H(2')	119.4
C(1')-C(2')-H(2')	119.4
C(4')-C(3')-C(2')	120.4(3)
C(4')-C(3')-H(3')	119.8
C(2')-C(3')-H(3')	119.8
C(3')-C(4')-C(5')	119.1(2)
C(3')-C(4')-H(4')	120.5
C(5')-C(4')-H(4')	120.5
C(6')-C(5')-C(4')	120.7(3)

C(6')-C(5')-H(5')	119.7
C(4')-C(5')-H(5')	119.7
C(5')-C(6')-C(1')	120.8(3)
C(5')-C(6')-H(6')	119.6
C(1')-C(6')-H(6')	119.6
O(2)-C(7')-C(8')	121.4(2)
O(2)-C(7')-C(2)	120.4(2)
C(8')-C(7')-C(2)	118.1(2)
C(13')-C(8')-C(9')	118.0(2)
C(13')-C(8')-C(7')	123.2(2)
C(9')-C(8')-C(7')	118.7(2)
C(10')-C(9')-C(8')	120.7(3)
C(10')-C(9')-H(9')	119.6
C(8')-C(9')-H(9')	119.6
C(9')-C(10')-C(11')	120.4(3)
C(9')-C(10')-H(10')	119.8
C(11')-C(10')-H(10')	119.8
C(12')-C(11')-C(10')	119.8(3)
C(12')-C(11')-H(11')	120.1
C(10')-C(11')-H(11')	120.1
C(11')-C(12')-C(13')	119.8(3)
C(11')-C(12')-H(12')	120.1
C(13')-C(12')-H(12')	120.1
C(12')-C(13')-C(8')	121.3(2)
C(12')-C(13')-H(13')	119.4
C(8')-C(13')-H(13')	119.4

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for cu_m35_r_0m. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	94(2)	26(1)	63(1)	-10(1)	-38(1)	-3(1)
O(2)	36(1)	50(1)	65(1)	5(1)	-4(1)	-5(1)
N(5)	64(2)	33(1)	37(1)	-8(1)	-21(1)	-4(1)
C(1)	51(2)	30(1)	40(1)	-7(1)	-16(1)	-6(1)
C(2)	35(1)	27(1)	29(1)	-6(1)	-6(1)	-3(1)
C(3)	37(1)	30(1)	32(1)	-4(1)	-5(1)	-5(1)
C(4)	52(2)	30(1)	34(1)	-3(1)	-13(1)	-7(1)
C(1')	38(1)	26(1)	32(1)	-3(1)	-10(1)	-1(1)
C(2')	43(1)	35(1)	43(1)	-7(1)	-7(1)	-6(1)
C(3')	65(2)	35(2)	65(2)	-14(1)	-15(2)	-14(1)
C(4')	77(2)	36(2)	54(2)	-20(1)	-15(1)	7(1)
C(5')	60(2)	48(2)	49(2)	-15(1)	-3(1)	9(1)
C(6')	45(1)	36(1)	41(1)	-6(1)	-4(1)	-2(1)
C(7')	34(1)	32(1)	36(1)	-8(1)	-6(1)	-1(1)
C(8')	36(1)	34(1)	34(1)	-7(1)	-11(1)	-1(1)
C(9')	48(2)	39(2)	47(2)	-5(1)	-9(1)	7(1)
C(10')	65(2)	44(2)	57(2)	-3(1)	-16(1)	14(1)
C(11')	82(2)	40(2)	44(2)	4(1)	-19(2)	-3(1)
C(12')	63(2)	59(2)	42(2)	2(1)	-3(1)	-10(1)
C(13')	45(2)	43(2)	42(2)	-3(1)	-5(1)	1(1)

Table 5. Torsion angles [°] for cu_m35_r_0m.

C(4)-N(5)-C(1)-O(1)	-172.7(3)
C(4)-N(5)-C(1)-C(2)	5.3(3)
O(1)-C(1)-C(2)-C(7')	-42.4(4)
N(5)-C(1)-C(2)-C(7')	139.6(2)
O(1)-C(1)-C(2)-C(3)	-167.1(3)
N(5)-C(1)-C(2)-C(3)	14.8(3)
C(7')-C(2)-C(3)-C(1')	86.2(2)
C(1)-C(2)-C(3)-C(1')	-151.5(2)
C(7')-C(2)-C(3)-C(4)	-149.39(19)
C(1)-C(2)-C(3)-C(4)	-27.1(2)
C(1)-N(5)-C(4)-C(3)	-22.9(3)
C(1')-C(3)-C(4)-N(5)	155.8(2)
C(2)-C(3)-C(4)-N(5)	29.9(2)
C(2)-C(3)-C(1')-C(6')	54.3(3)
C(4)-C(3)-C(1')-C(6')	-63.9(3)
C(2)-C(3)-C(1')-C(2')	-128.0(2)
C(4)-C(3)-C(1')-C(2')	113.8(2)
C(6')-C(1')-C(2')-C(3')	0.0(4)
C(3)-C(1')-C(2')-C(3')	-177.8(2)
C(1')-C(2')-C(3')-C(4')	-0.2(4)
C(2')-C(3')-C(4')-C(5')	-0.4(4)
C(3')-C(4')-C(5')-C(6')	1.1(4)
C(4')-C(5')-C(6')-C(1')	-1.4(4)
C(2')-C(1')-C(6')-C(5')	0.8(4)
C(3)-C(1')-C(6')-C(5')	178.6(2)
C(3)-C(2)-C(7')-O(2)	13.1(3)
C(1)-C(2)-C(7')-O(2)	-104.9(3)
C(3)-C(2)-C(7')-C(8')	-167.81(19)
C(1)-C(2)-C(7')-C(8')	74.3(3)
O(2)-C(7')-C(8')-C(13')	-148.9(3)
C(2)-C(7')-C(8')-C(13')	32.0(3)
O(2)-C(7')-C(8')-C(9')	28.4(4)
C(2)-C(7')-C(8')-C(9')	-150.7(2)
C(13')-C(8')-C(9')-C(10')	0.2(4)
C(7')-C(8')-C(9')-C(10')	-177.2(3)
C(8')-C(9')-C(10')-C(11')	0.7(5)

C(9')-C(10')-C(11')-C(12')	-0.8(5)
C(10')-C(11')-C(12')-C(13')	0.1(5)
C(11')-C(12')-C(13')-C(8')	0.9(5)
C(9')-C(8')-C(13')-C(12')	-1.0(4)
C(7')-C(8')-C(13')-C(12')	176.3(3)

Symmetry transformations used to generate equivalent atoms:

X-Ray data of 4l

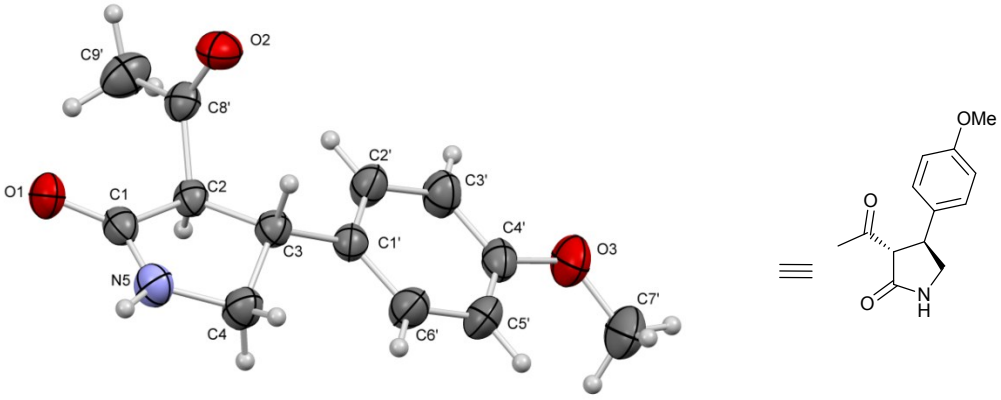


Fig: crystal structure of **4l**

Supplementary crystallographic data for the compound **4l (**CCDC **1492205)** can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

Crystal data and structure refinement for 4l

Identification code	mo_md42_0m	
Empirical formula	$C_{13}H_{15}NO_3$	
Formula weight	233.26	
Temperature	250(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 5.21520(7)Å	alpha= 90°.
	b = 9.89840(14) Å	beta= 94.5570 (5)°.
	c =23.0406 (3) Å	gamma= 90°.
Volume	1185.65(3) $Å^3$	
Z	4	
Density (calculated)	1.307 Mg/m^3	
Absorption coefficient	0.093mm^{-1}	
F(000)	496	
Crystal size	0.49 x 0.47 x 0.45mm^3	
Theta range for data collection	2.72 to 25.00°.	
Index ranges	6<=h<=6, 11<=k<=11, 27<=l<=27	
Reflections collected	25501	
Independent reflections	2073 [R(int) = 0.0365]	
Completeness to theta =	25.00° 99.8 %	
Max. and min. transmission	0.9594 and 0.9561	
Refinement method	Full matrix leastsquares on F^2	

Data / restraints / parameters	2073 / 0 / 157
Goodness-of-fit on F^2	1.091
Final R indices [I>2sigma(I)]	R1 = 0.0418, wR2 = 0.1128
R indices (all data)	R1 = 0.0469, wR2 = 0.1172
Extinction coefficient	0.026(4)
Largest diff. peak and hole	0.291 and -0.185 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for mo_md42_0m. U (eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	-2321(2)	6087(1)	-96(1)	47(1)
O(2)	-1110(2)	8977(1)	788(1)	53(1)
O(3)	5947(3)	7283(2)	3423(1)	63(1)
N(5)	-3354(3)	5116(1)	756(1)	45(1)
C(1)	-2007(3)	5918(2)	434(1)	38(1)
C(2)	-2(3)	6651(2)	844(1)	34(1)
C(3)	-1068(3)	6479(2)	1440(1)	36(1)
C(4)	-2496(3)	5125(2)	1374(1)	47(1)
C(1')	848(3)	6611(2)	1964(1)	36(1)
C(2')	2773(3)	7582(2)	1983(1)	45(1)
C(3')	4454(3)	7770(2)	2472(1)	49(1)
C(4')	4238(3)	6983(2)	2960(1)	43(1)
C(5')	2353(4)	6011(2)	2953(1)	52(1)
C(6')	674(4)	5835(2)	2459(1)	49(1)
C(7')	6084(5)	6407(2)	3899(1)	79(1)
C(8')	257(3)	8087(2)	629(1)	38(1)
C(9')	2216(4)	8314(2)	204(1)	57(1)

Table 3. Bond lengths [Å] and angles [°] for mo_md42_0m.

O(1)-C(1)	1.2309(19)
O(2)-C(8')	1.2082(19)
O(3)-C(4')	1.368(2)
O(3)-C(7')	1.394(3)
N(5)-C(1)	1.326(2)
N(5)-C(4)	1.459(2)
N(5)-H(5)	0.8700
C(1)-C(2)	1.534(2)
C(2)-C(8')	1.515(2)
C(2)-C(3)	1.531(2)
C(2)-H(2)	0.9900
C(3)-C(1')	1.511(2)
C(3)-C(4)	1.536(2)
C(3)-H(3)	0.9900
C(4)-H(4A)	0.9800
C(4)-H(4B)	0.9800
C(1')-C(6')	1.383(2)
C(1')-C(2')	1.388(2)
C(2')-C(3')	1.383(2)
C(2')-H(2')	0.9400
C(3')-C(4')	1.381(2)
C(3')-H(3')	0.9400
C(4')-C(5')	1.374(3)
C(5')-C(6')	1.392(2)
C(5')-H(5')	0.9400
C(6')-H(6')	0.9400
C(7')-H(7'1)	0.9700
C(7')-H(7'2)	0.9700
C(7')-H(7'3)	0.9700
C(8')-C(9')	1.487(2)
C(9')-H(9'1)	0.9700
C(9')-H(9'2)	0.9700
C(9')-H(9'3)	0.9700
C(4')-O(3)-C(7')	117.92(17)
C(1)-N(5)-C(4)	114.04(13)
C(1)-N(5)-H(5)	123.0

C(4)-N(5)-H(5)	123.0
O(1)-C(1)-N(5)	126.99(15)
O(1)-C(1)-C(2)	125.23(14)
N(5)-C(1)-C(2)	107.75(13)
C(8')-C(2)-C(3)	116.46(13)
C(8')-C(2)-C(1)	108.48(12)
C(3)-C(2)-C(1)	102.78(12)
C(8')-C(2)-H(2)	109.6
C(3)-C(2)-H(2)	109.6
C(1)-C(2)-H(2)	109.6
C(1')-C(3)-C(2)	116.33(12)
C(1')-C(3)-C(4)	115.99(13)
C(2)-C(3)-C(4)	102.51(12)
C(1')-C(3)-H(3)	107.1
C(2)-C(3)-H(3)	107.1
C(4)-C(3)-H(3)	107.1
N(5)-C(4)-C(3)	102.12(12)
N(5)-C(4)-H(4A)	111.3
C(3)-C(4)-H(4A)	111.3
N(5)-C(4)-H(4B)	111.3
C(3)-C(4)-H(4B)	111.3
H(4A)-C(4)-H(4B)	109.2
C(6')-C(1')-C(2')	116.92(14)
C(6')-C(1')-C(3)	121.91(14)
C(2')-C(1')-C(3)	121.04(13)
C(3')-C(2')-C(1')	121.96(15)
C(3')-C(2')-H(2')	119.0
C(1')-C(2')-H(2')	119.0
C(4')-C(3')-C(2')	119.97(16)
C(4')-C(3')-H(3')	120.0
C(2')-C(3')-H(3')	120.0
O(3)-C(4')-C(5')	125.53(16)
O(3)-C(4')-C(3')	115.06(16)
C(5')-C(4')-C(3')	119.39(15)
C(4')-C(5')-C(6')	119.97(16)
C(4')-C(5')-H(5')	120.0
C(6')-C(5')-H(5')	120.0
C(1')-C(6')-C(5')	121.79(16)

C(1')-C(6')-H(6')	119.1
C(5')-C(6')-H(6')	119.1
O(2)-C(8')-C(9')	122.91(15)
O(2)-C(8')-C(2)	120.91(15)
C(9')-C(8')-C(2)	116.16(14)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for mo_md42_0m. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	51(1)	53(1)	35(1)	-3(1)	-7(1)	-2(1)
O(2)	64(1)	40(1)	55(1)	4(1)	8(1)	11(1)
O(3)	72(1)	71(1)	43(1)	-2(1)	-19(1)	4(1)
N(5)	46(1)	47(1)	42(1)	-2(1)	-7(1)	-11(1)
C(1)	38(1)	38(1)	37(1)	-5(1)	-4(1)	6(1)
C(2)	32(1)	37(1)	32(1)	-1(1)	-2(1)	4(1)
C(3)	36(1)	38(1)	34(1)	0(1)	0(1)	2(1)
C(4)	51(1)	48(1)	41(1)	4(1)	-5(1)	-10(1)
C(1')	38(1)	38(1)	32(1)	-1(1)	2(1)	4(1)
C(2')	50(1)	52(1)	33(1)	4(1)	-1(1)	-6(1)
C(3')	50(1)	56(1)	40(1)	-2(1)	-3(1)	-9(1)
C(4')	47(1)	48(1)	34(1)	-7(1)	-5(1)	10(1)
C(5')	68(1)	52(1)	35(1)	9(1)	-3(1)	2(1)
C(6')	57(1)	49(1)	41(1)	7(1)	-3(1)	-9(1)
C(7')	112(2)	76(2)	45(1)	1(1)	-22(1)	7(1)
C(8')	37(1)	42(1)	34(1)	2(1)	-7(1)	1(1)
C(9')	53(1)	62(1)	57(1)	15(1)	11(1)	1(1)

Table 5. Torsion angles [°] for mo_md42_0m.

C(4)-N(5)-C(1)-O(1)	179.46(16)
C(4)-N(5)-C(1)-C(2)	-2.36(18)
O(1)-C(1)-C(2)-C(8')	36.0(2)
N(5)-C(1)-C(2)-C(8')	-142.19(13)
O(1)-C(1)-C(2)-C(3)	159.91(14)
N(5)-C(1)-C(2)-C(3)	-18.31(16)
C(8')-C(2)-C(3)-C(1')	-83.74(16)
C(1)-C(2)-C(3)-C(1')	157.84(12)
C(8')-C(2)-C(3)-C(4)	148.62(13)
C(1)-C(2)-C(3)-C(4)	30.20(15)
C(1)-N(5)-C(4)-C(3)	21.90(18)
C(1')-C(3)-C(4)-N(5)	-159.16(13)
C(2)-C(3)-C(4)-N(5)	-31.31(16)
C(2)-C(3)-C(1')-C(6')	-144.36(16)
C(4)-C(3)-C(1')-C(6')	-23.7(2)
C(2)-C(3)-C(1')-C(2')	39.8(2)
C(4)-C(3)-C(1')-C(2')	160.49(15)
C(6')-C(1')-C(2')-C(3')	0.2(3)
C(3)-C(1')-C(2')-C(3')	176.25(15)
C(1')-C(2')-C(3')-C(4')	-0.3(3)
C(7')-O(3)-C(4')-C(5')	10.7(3)
C(7')-O(3)-C(4')-C(3')	-170.96(18)
C(2')-C(3')-C(4')-O(3)	-178.08(15)
C(2')-C(3')-C(4')-C(5')	0.4(3)
O(3)-C(4')-C(5')-C(6')	177.85(16)
C(3')-C(4')-C(5')-C(6')	-0.4(3)
C(2')-C(1')-C(6')-C(5')	-0.3(3)
C(3)-C(1')-C(6')-C(5')	-176.27(15)
C(4')-C(5')-C(6')-C(1')	0.4(3)
C(3)-C(2)-C(8')-O(2)	-26.8(2)
C(1)-C(2)-C(8')-O(2)	88.51(17)
C(3)-C(2)-C(8')-C(9')	154.94(14)
C(1)-C(2)-C(8')-C(9')	-89.80(16)

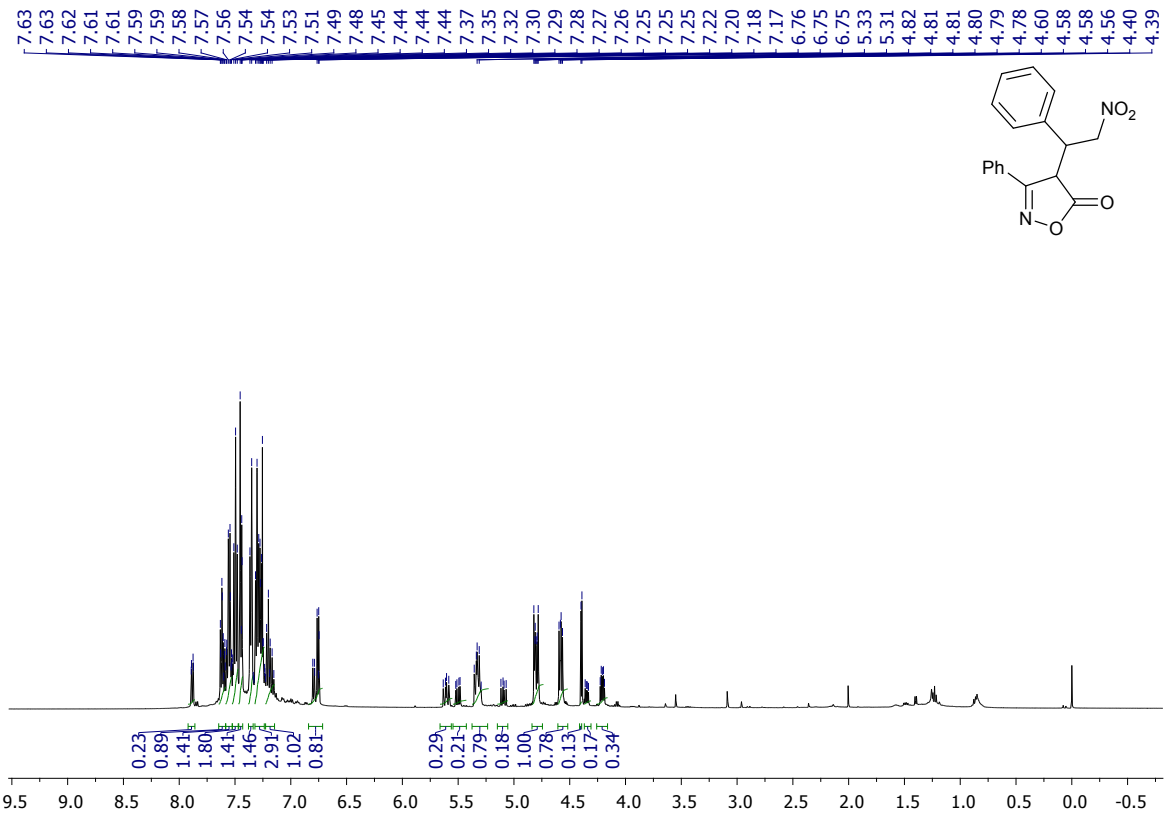
Symmetry transformations used to generate equivalent atoms:

References

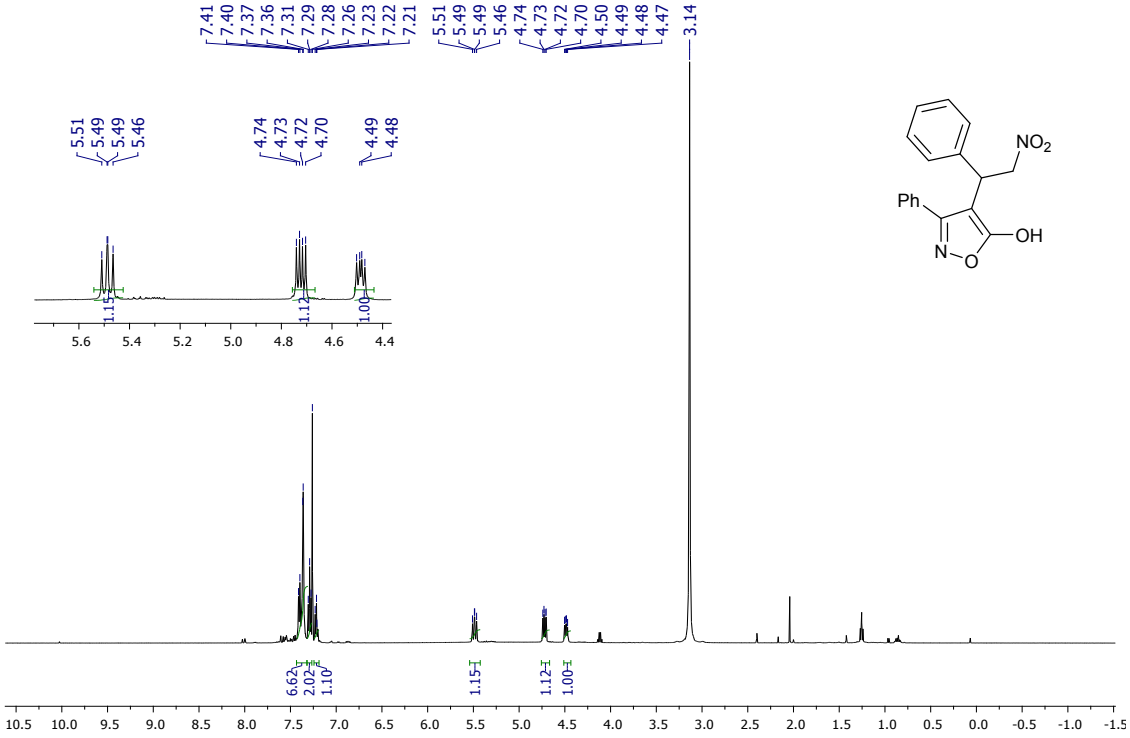
1. (a) J. Lopchuk, R. Hughes and G. Gribble, *Org. Lett*., 2013, **15**, 5218; (b) T. Naveen, S. Maity, U. Sharma and D. Maiti *J. Org. Chem.* 2013, **78**, 5949; (c) S. Cai, S. Zhang, Y. Zhao and D. Wang, *Org. Lett*., 2013, **15**, 2660; (d) L. Munoz, A. Rodriguez, G. Rosell, M. Boscha and A. Guerrero *Org. Biomol. Chem*., 2011, **9**, 8171; (e) B. Trost, and C. Muller, *J. Am. Chem. Soc*., 2008, **130**, 2438.
2. (a) A. Griesbeck, M. Franke, J. Neudörfl and H. Kotaka, *Beilstein J. Org. Chem.*, 2011, **7**, 127; (b) P. Too, Y. Wang and S. Chiba, *Org. Lett.*, 2010, **12**, 5688; (c) I. Dias-Jurberg, F. Gagosz and S. Zard, *Org. Lett*., 2010, **12**, 416.
3. E. Farney and T. Yoon, *Angew. Chem. Int. Ed.*, 2014, **53**, 793.

^{1}H and ^{13}C NMR spectra of compounds

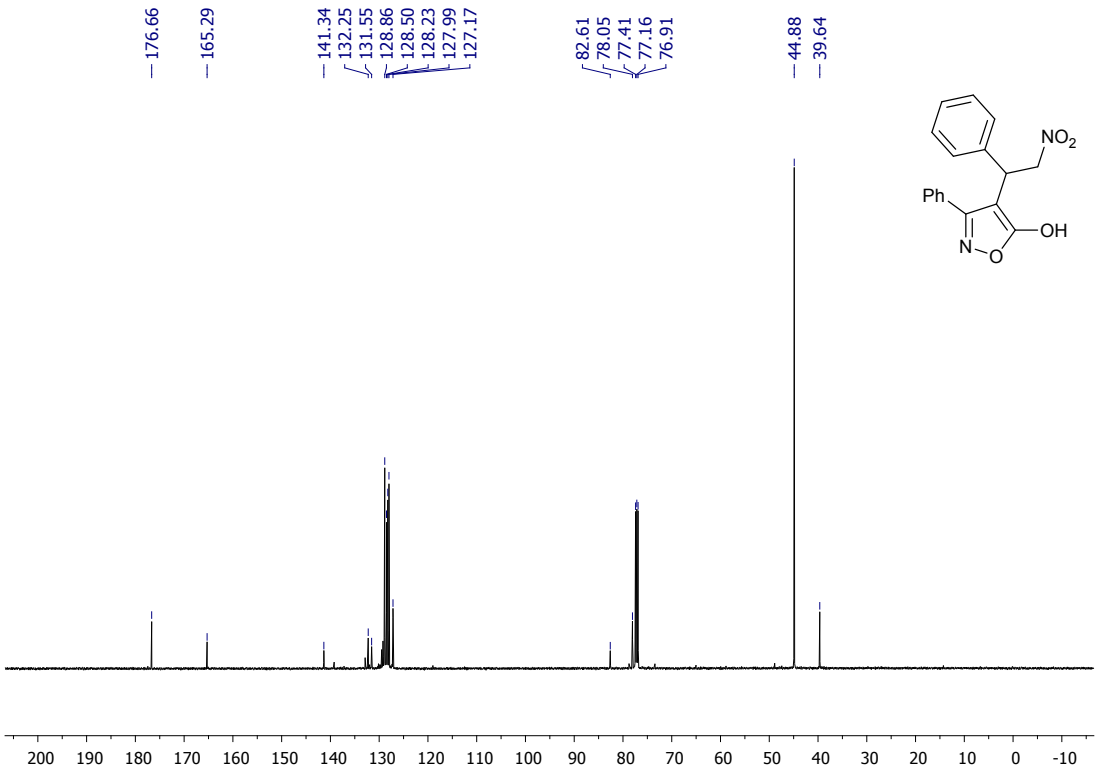
3a ^{1}H-NMR (500 MHz, $CDCl_3$):



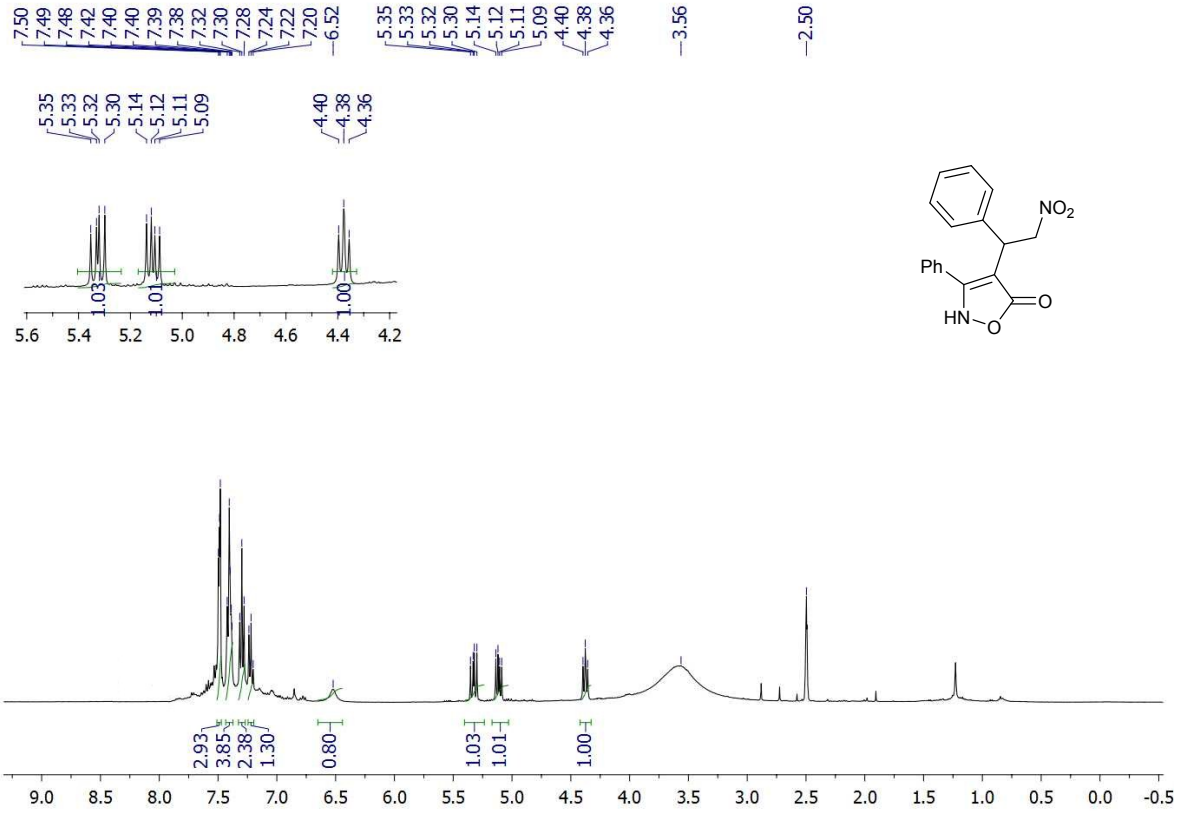
3a ^{1}H NMR (500 MHz, $CDCl_3$): In presence of DABCO (δ 2.9-3.1 ppm)



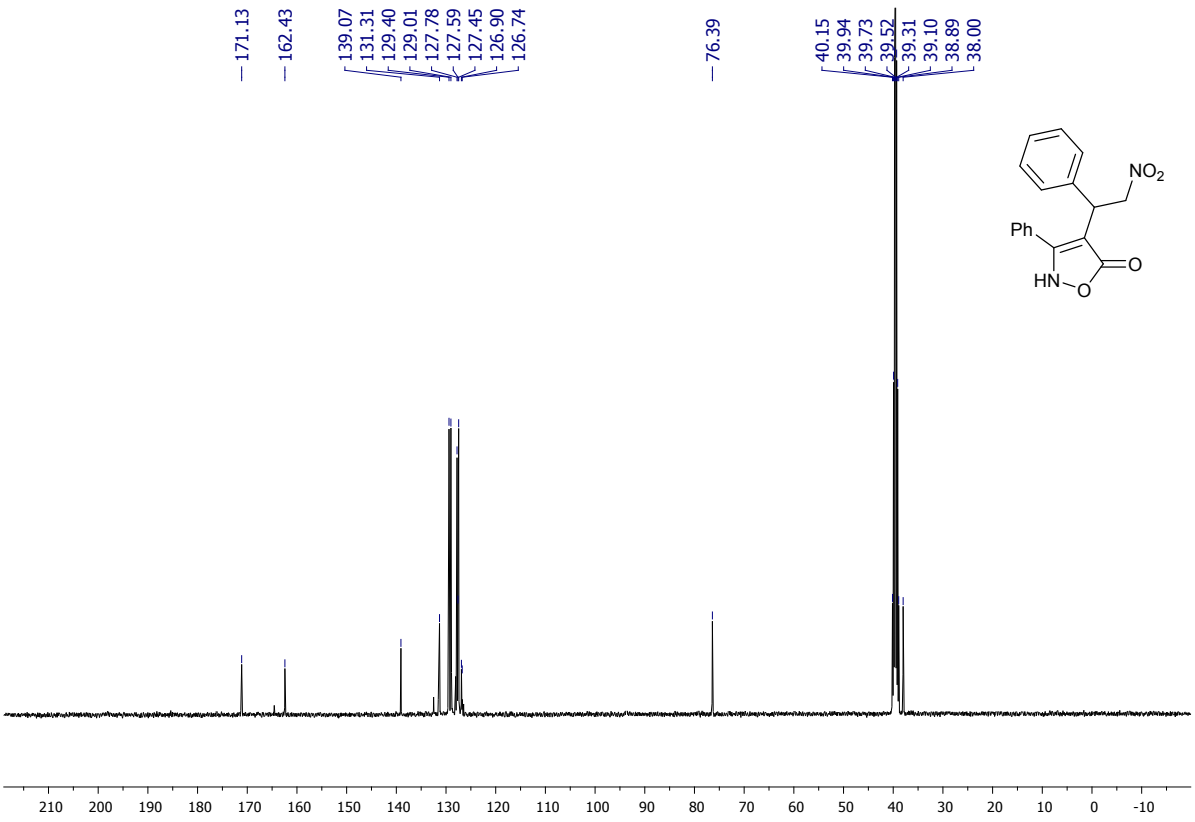
3a ^{13}C-NMR (125 MHz, $CDCl_3$): In presence of DABCO (δ 44-46 ppm)



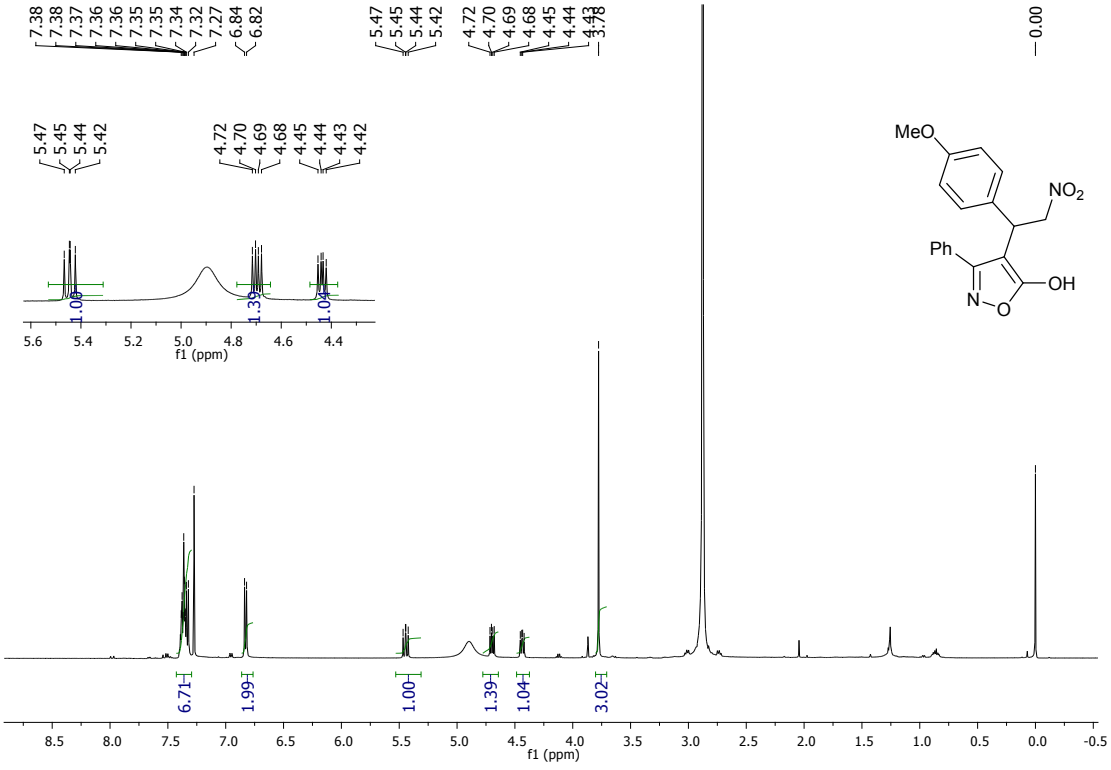
3a ^{1}H-NMR (400 MHz, DMSO-d_6)



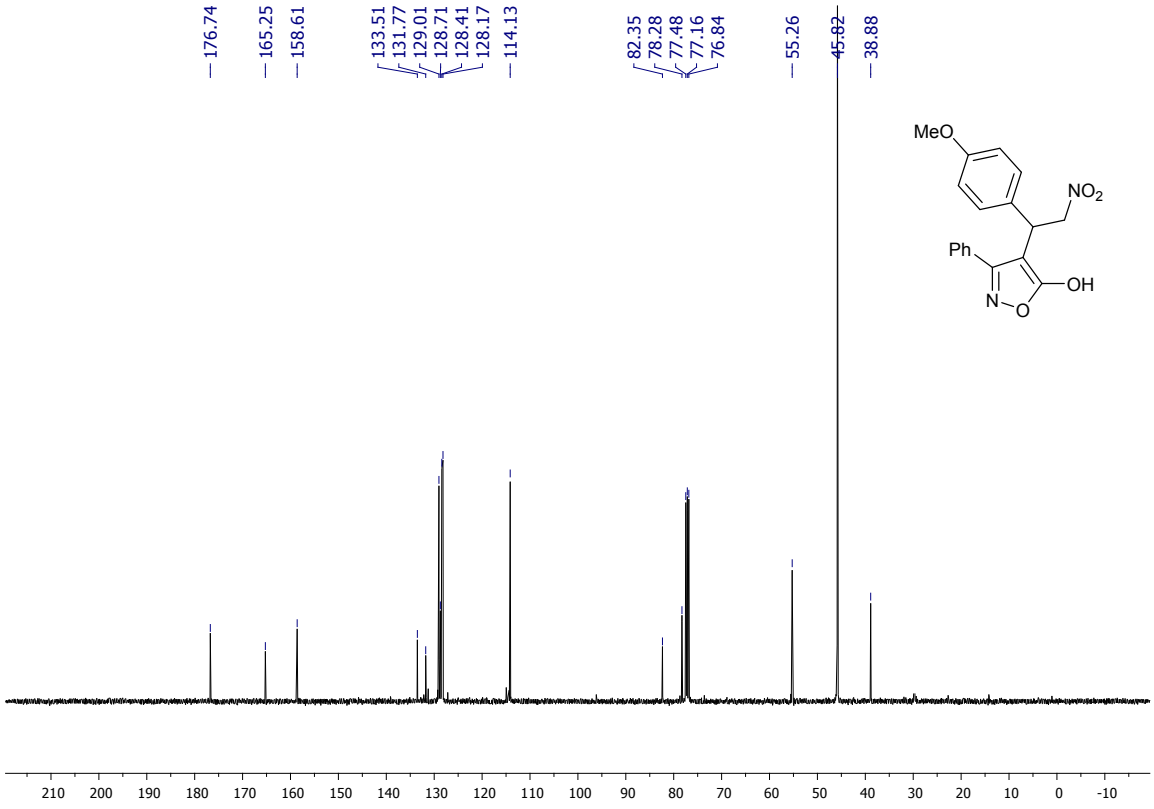
3a ^{13}C-NMR (100 MHz, DMSO-d_6)



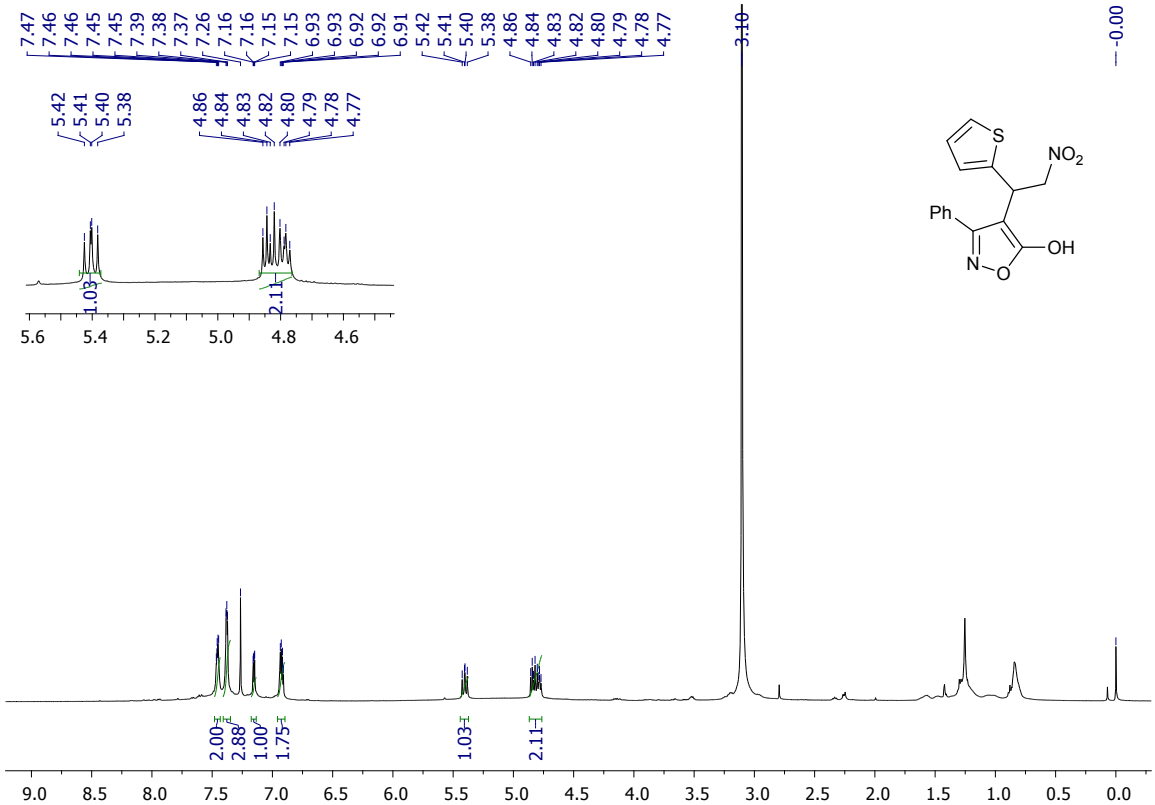
3b ^{1}H NMR (500 MHz, $CDCl_3$): In presence of DABCO (δ 2.9-3.1 ppm)



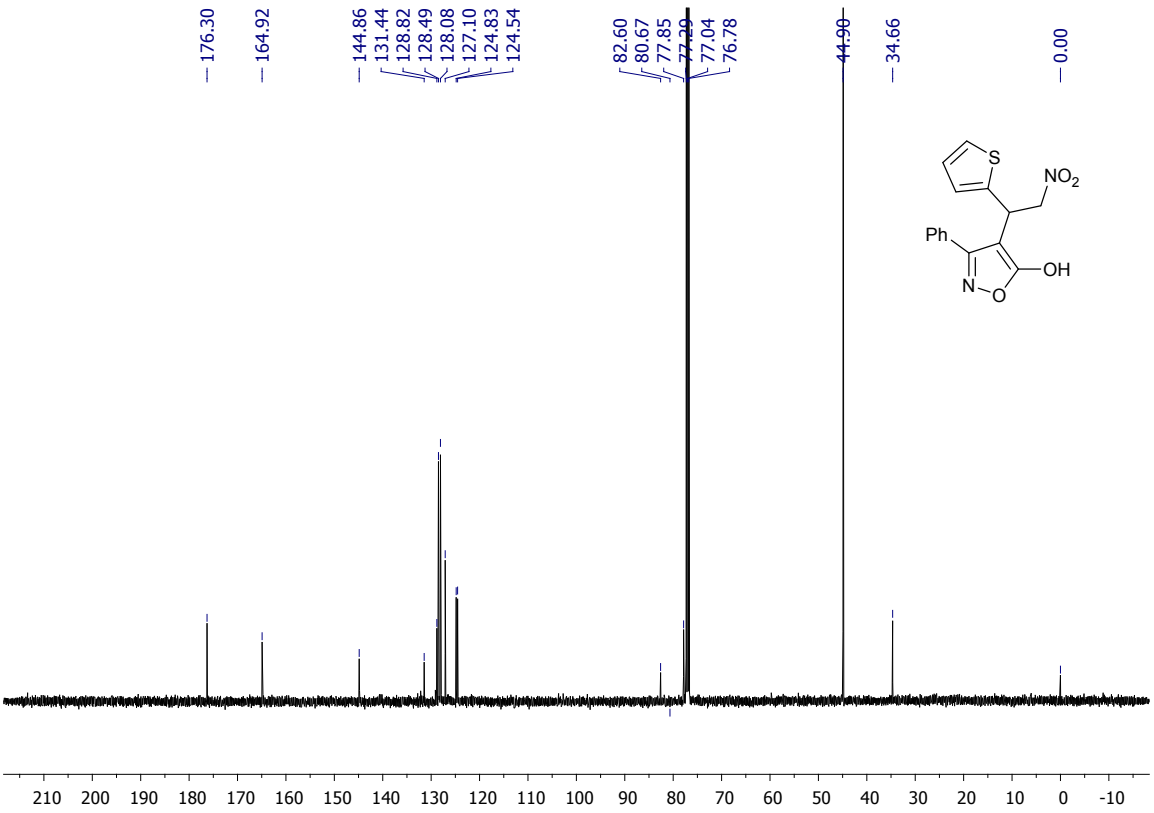
3b ^{13}C-NMR (100 MHz, $CDCl_3$): In presence of DABCO (δ 44-46 ppm)



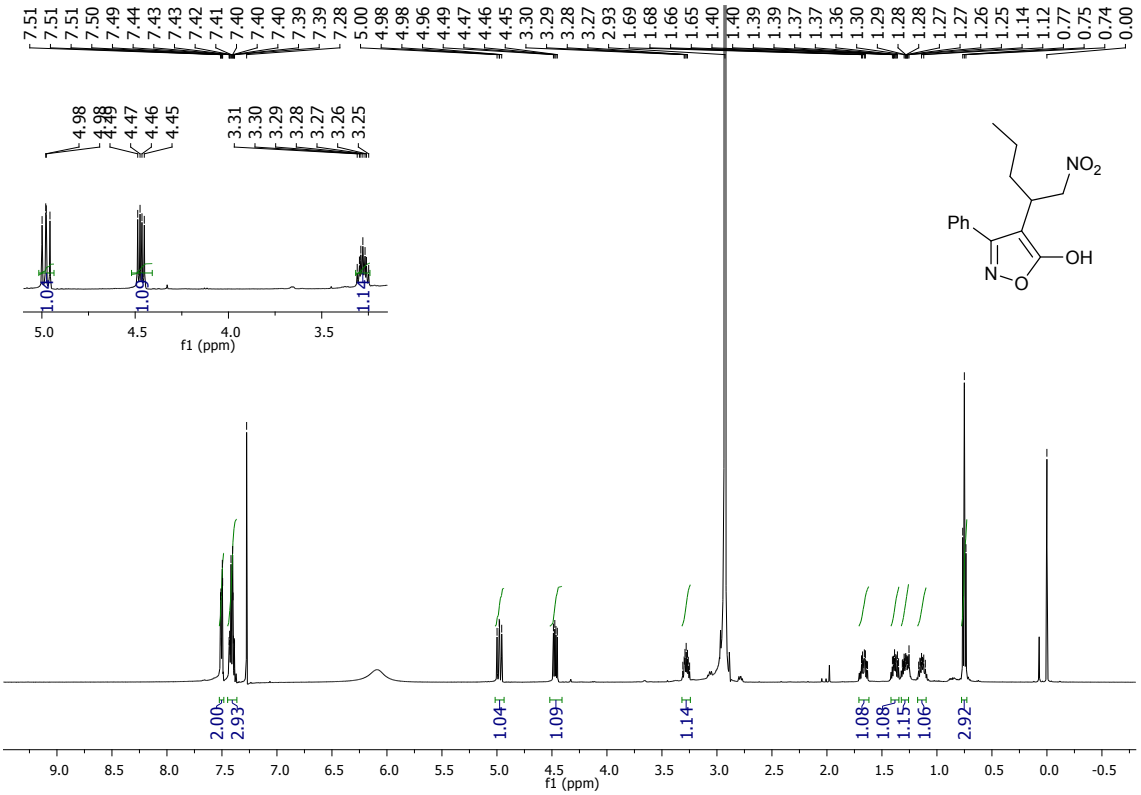
3c ^{1}H-NMR (500 MHz, $CDCl_3$): In presence of DABCO (δ 2.9-3.1 ppm)



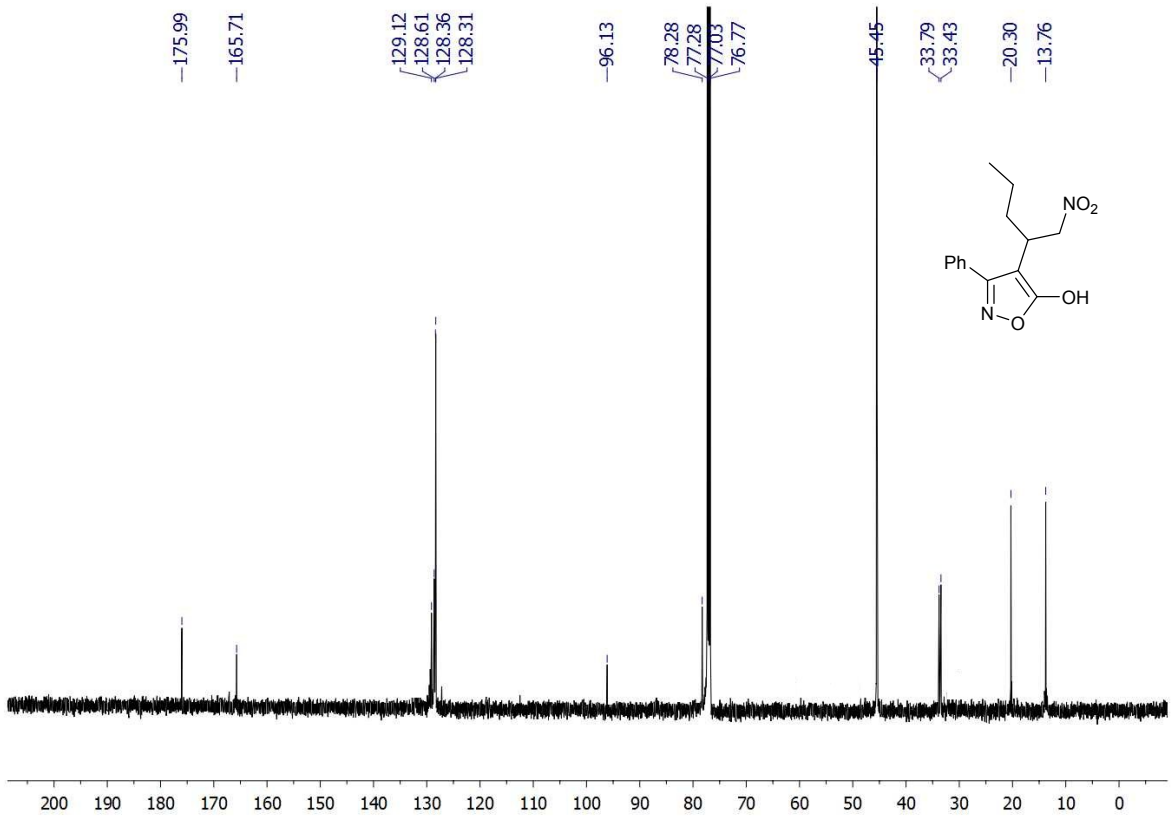
3c ^{13}C-NMR (125 MHz, $CDCl_3$): In presence of DABCO (δ 44-46 ppm)



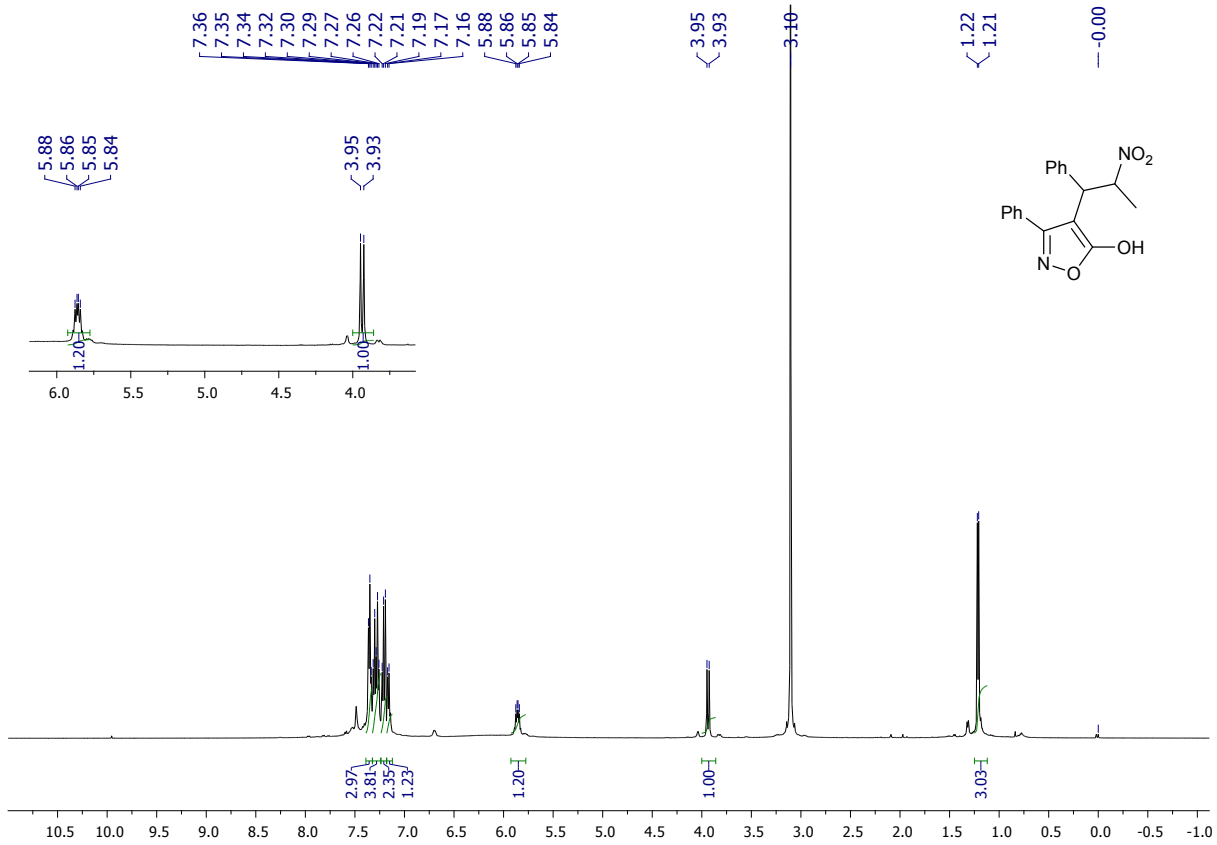
3d ^{1}H-NMR (500 MHz, $CDCl_3$): In presence of DABCO (δ 2.9-3.1 ppm)



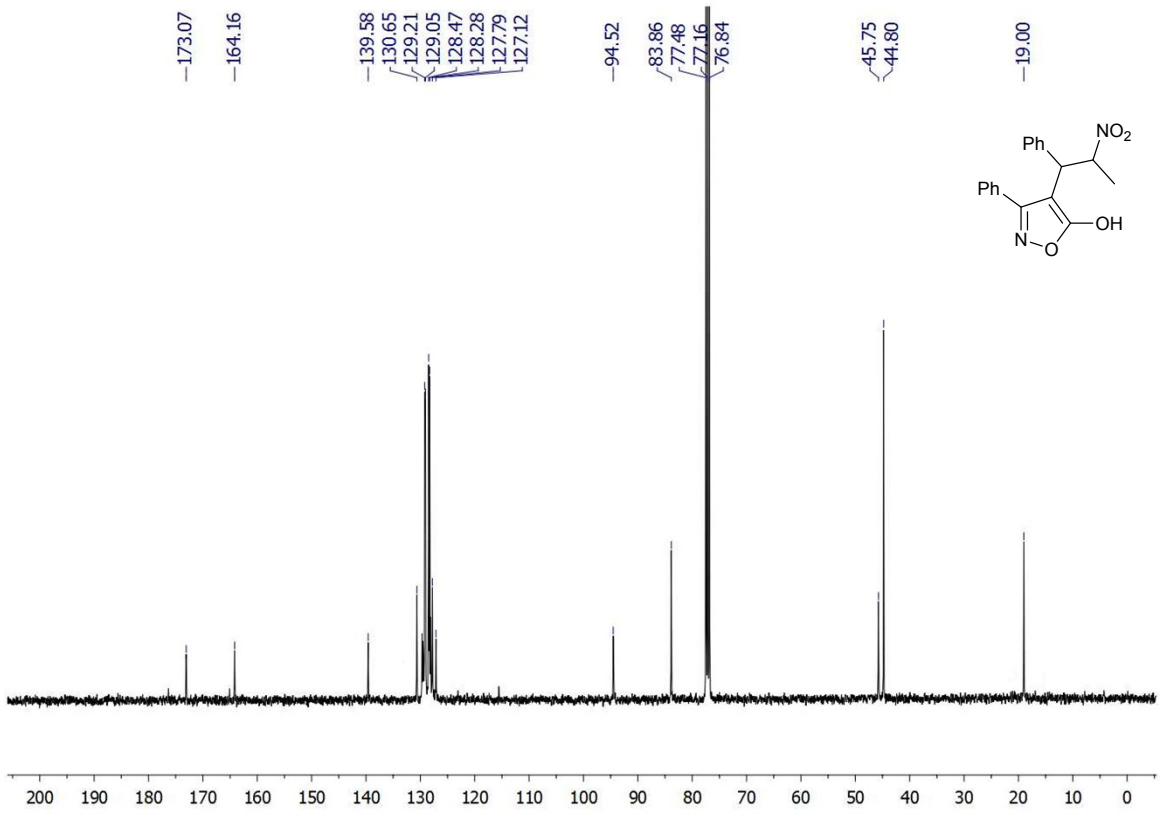
3d ^{13}C-NMR (125 MHz, $CDCl_3$): In presence of DABCO (δ 44-46 ppm)



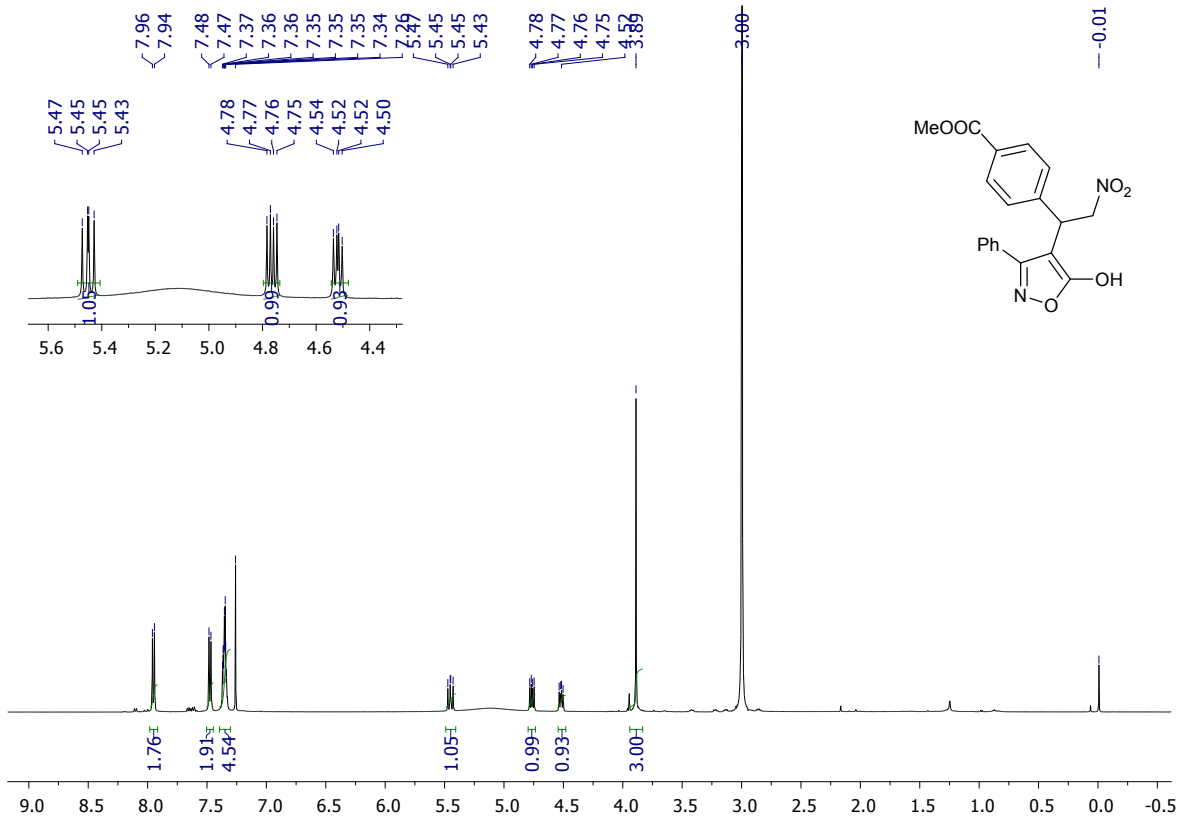
3e ^{1}H-NMR (500 MHz, $CDCl_3$): In presence of DABCO (δ 2.9-3.1 ppm)



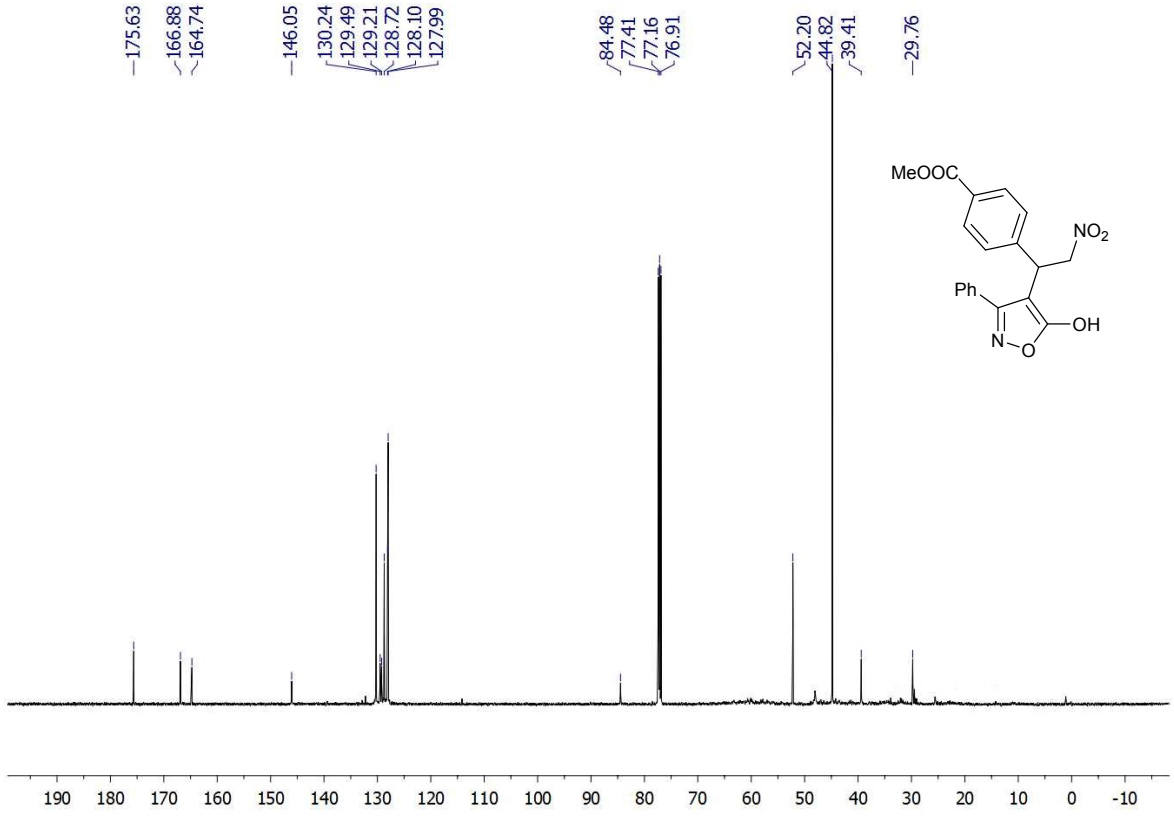
3e ^{13}C-NMR (100 MHz, $CDCl_3$): In presence of DABCO (δ 44-46 ppm)



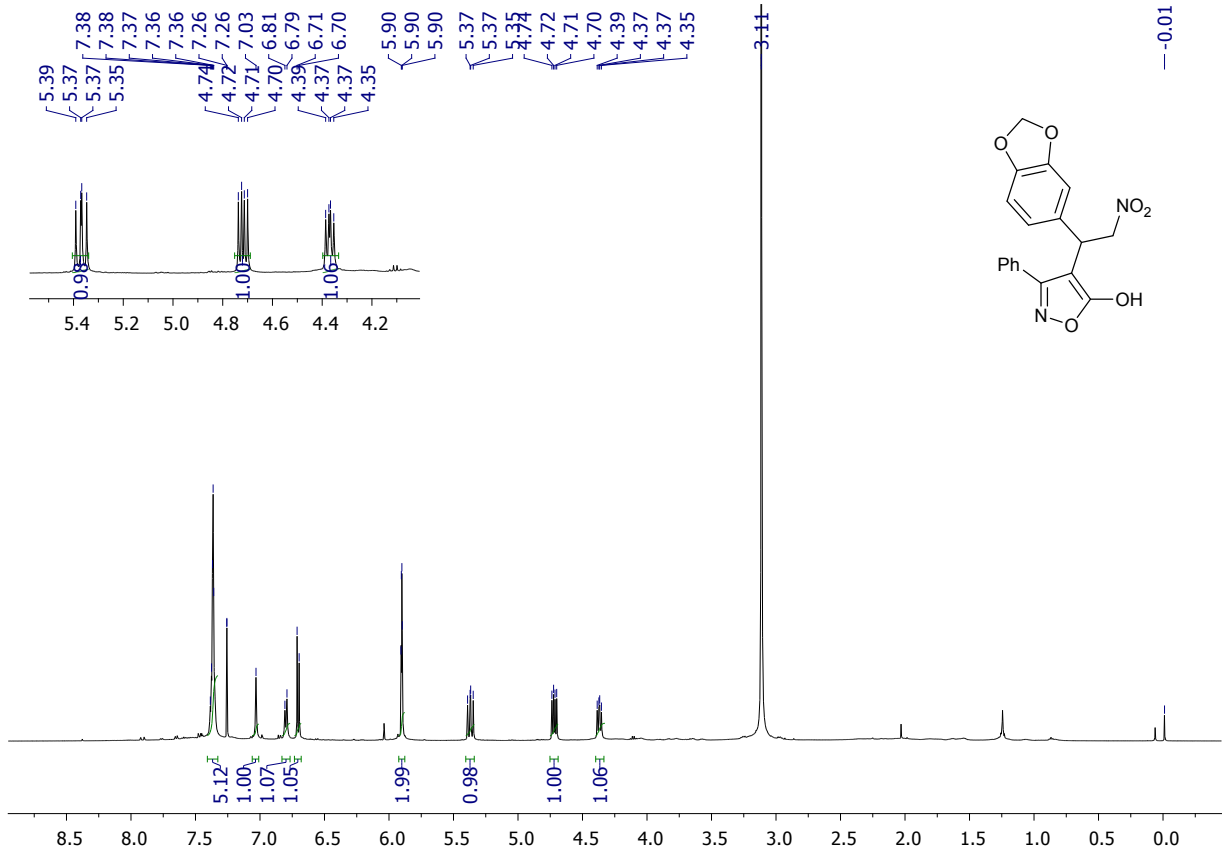
3f ^{1}H-NMR (500 MHz, $CDCl_3$): In presence of DABCO (δ 2.9-3.1 ppm)



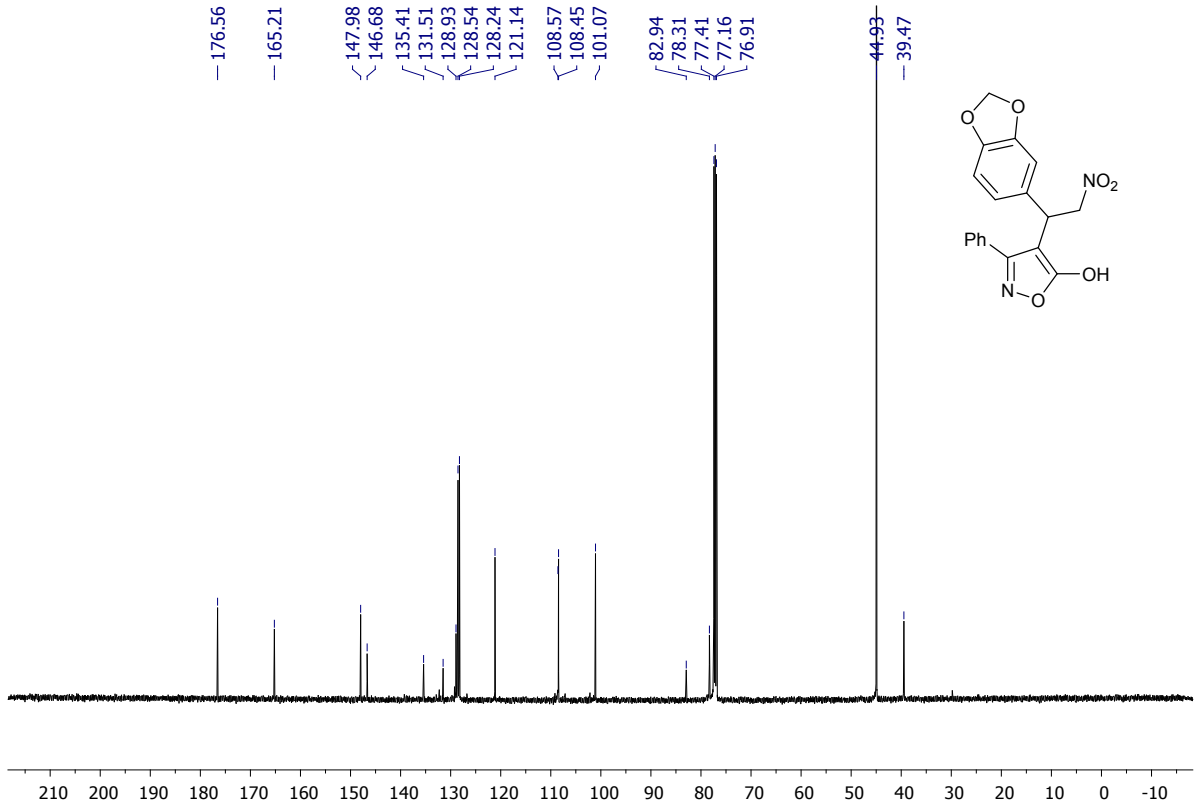
3f ^{13}C-NMR (125 MHz, $CDCl_3$): In presence of DABCO (δ 44-46 ppm)



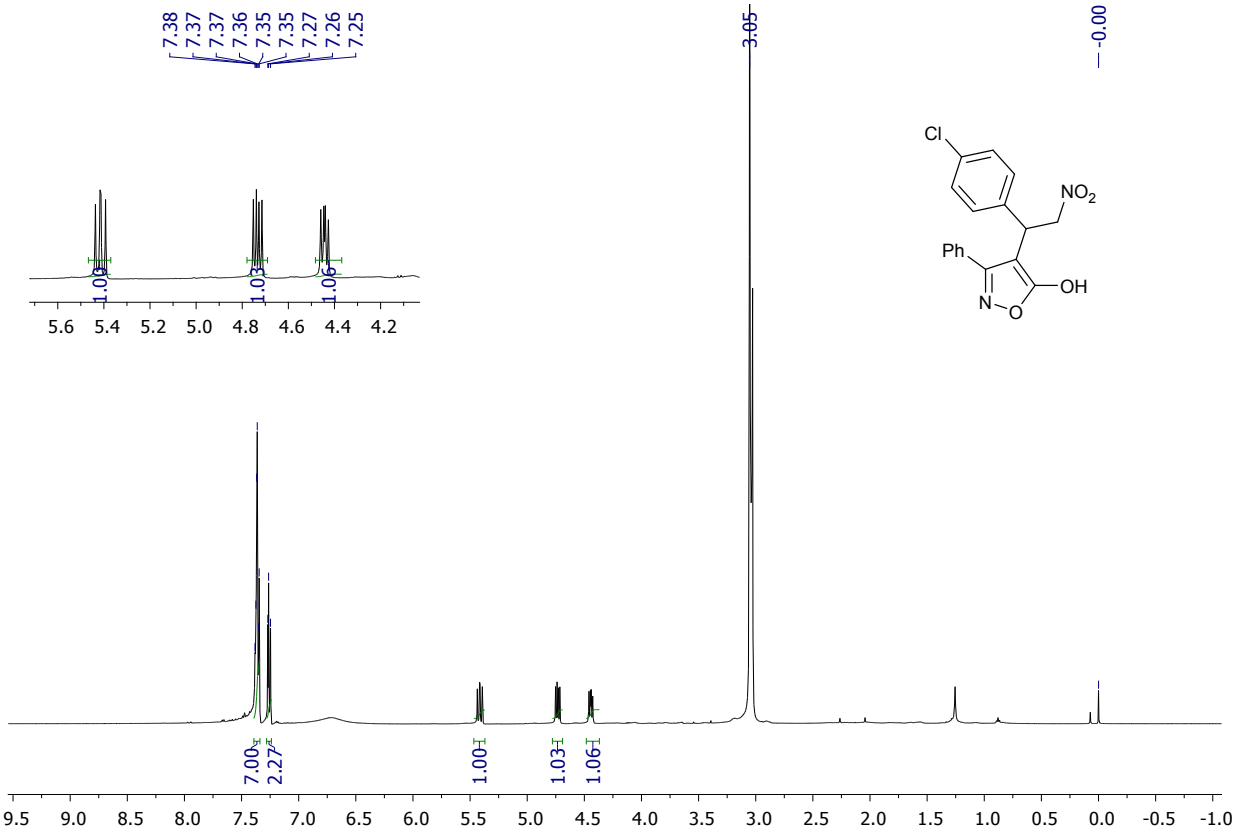
3g ^{1}H-NMR (500 MHz, $CDCl_3$): In presence of DABCO (δ 2.9-3.1 ppm)



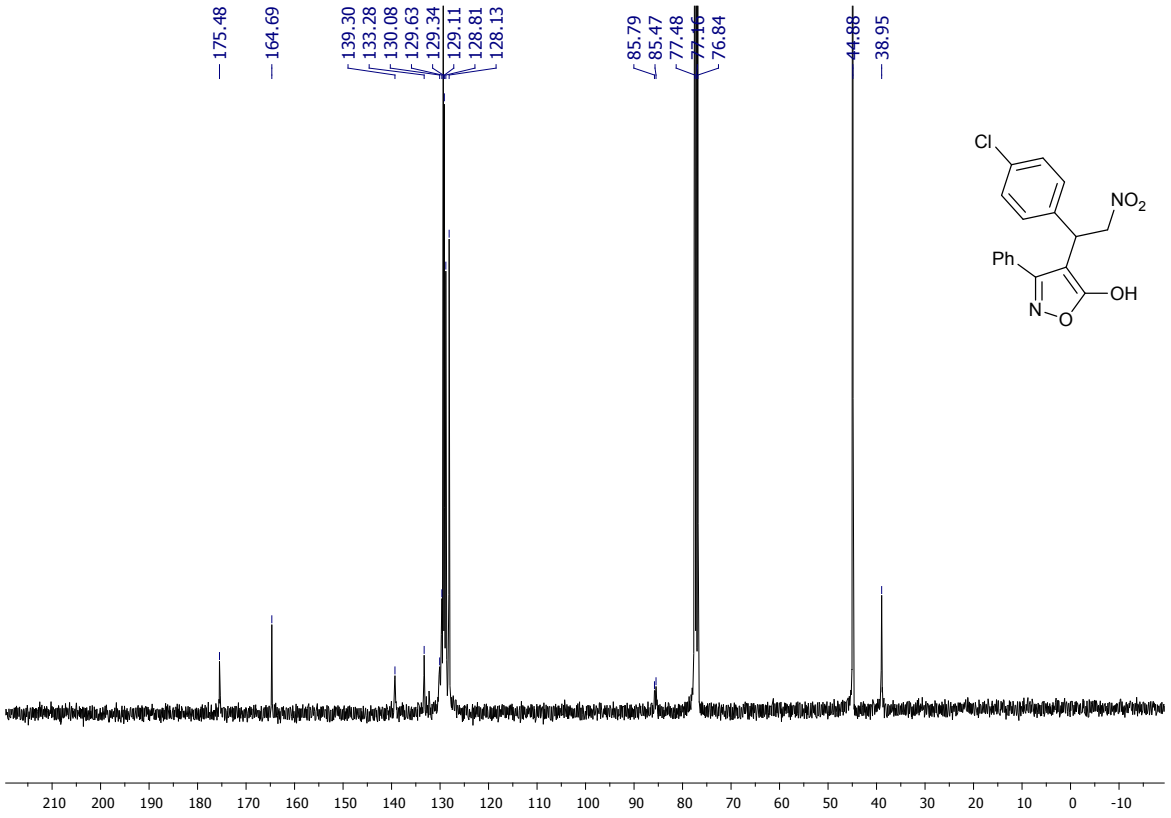
3g ^{13}C-NMR (125 MHz, $CDCl_3$): In presence of DABCO (δ 44-46 ppm)



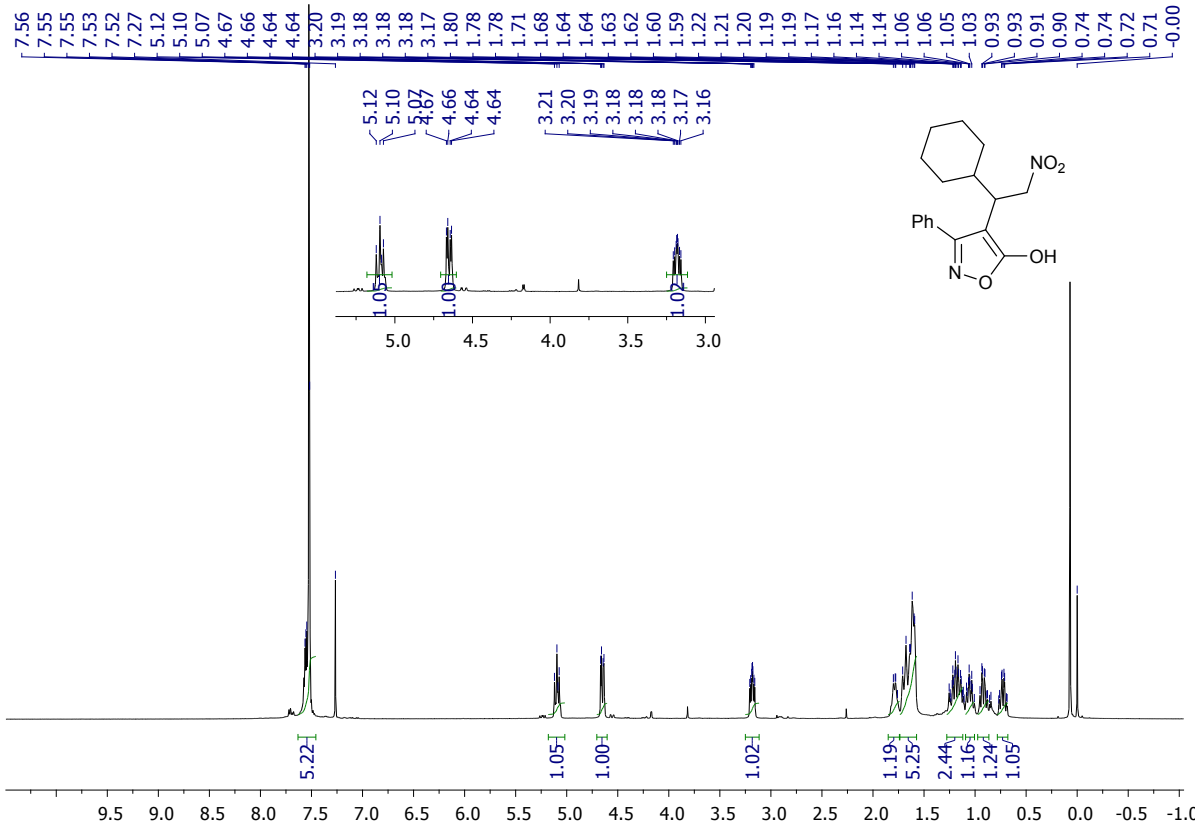
3h ^{1}H-NMR (500 MHz, $CDCl_3$): In presence of DABCO (δ 2.9-3.1 ppm)



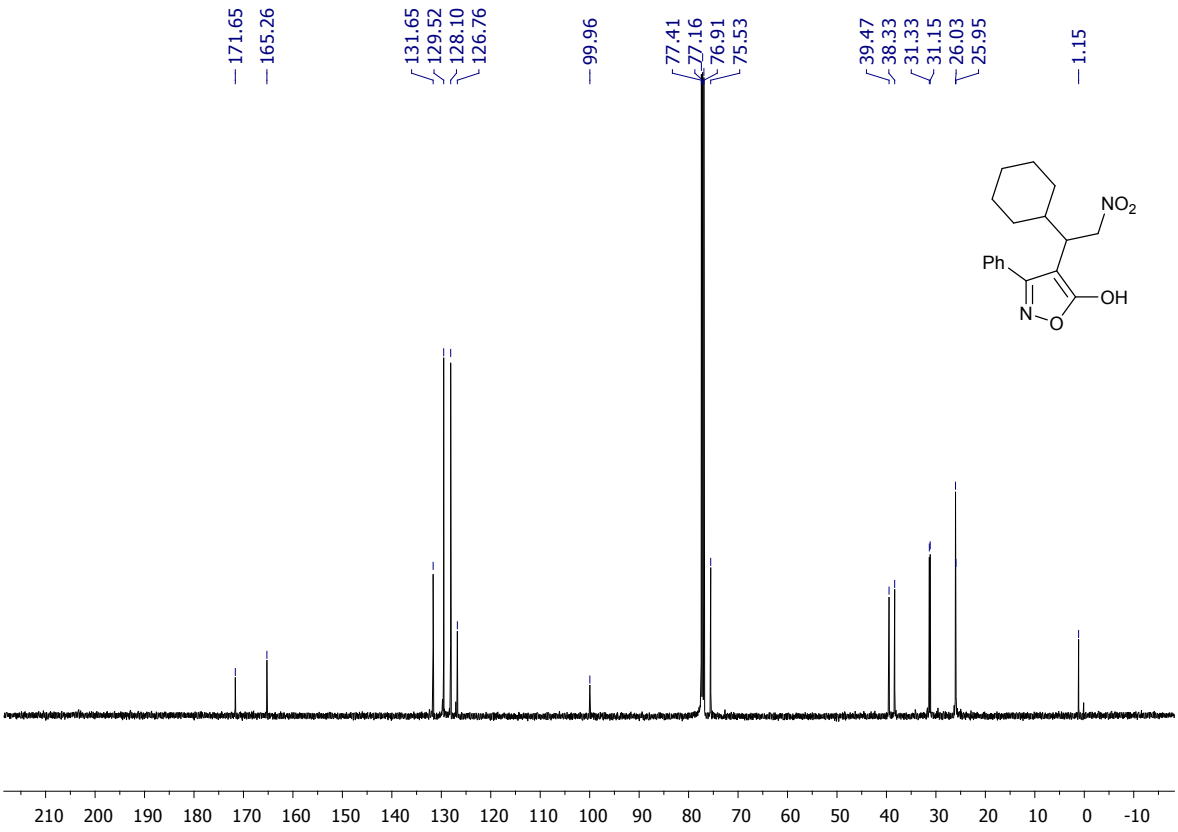
3h ^{13}C-NMR (100 MHz, $CDCl_3$): In presence of DABCO (δ 44-46 ppm)



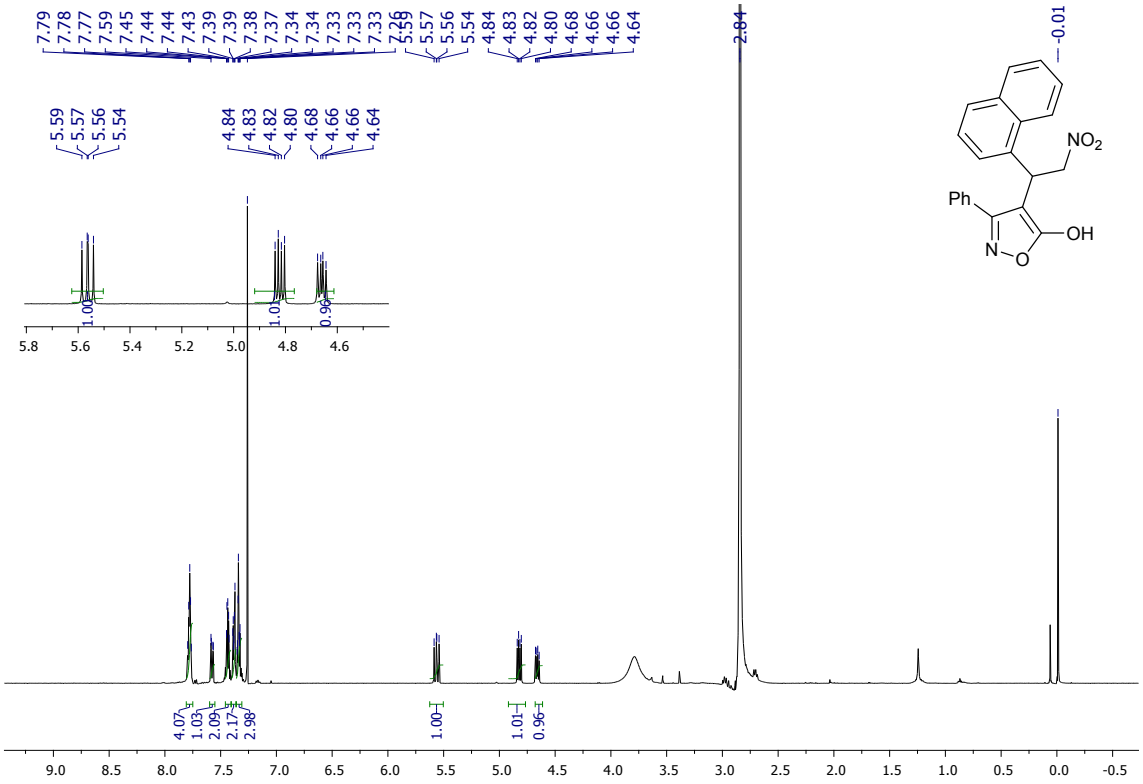
3i ^{1}H-NMR (500 MHz, $CDCl_3$): In absence of DABCO



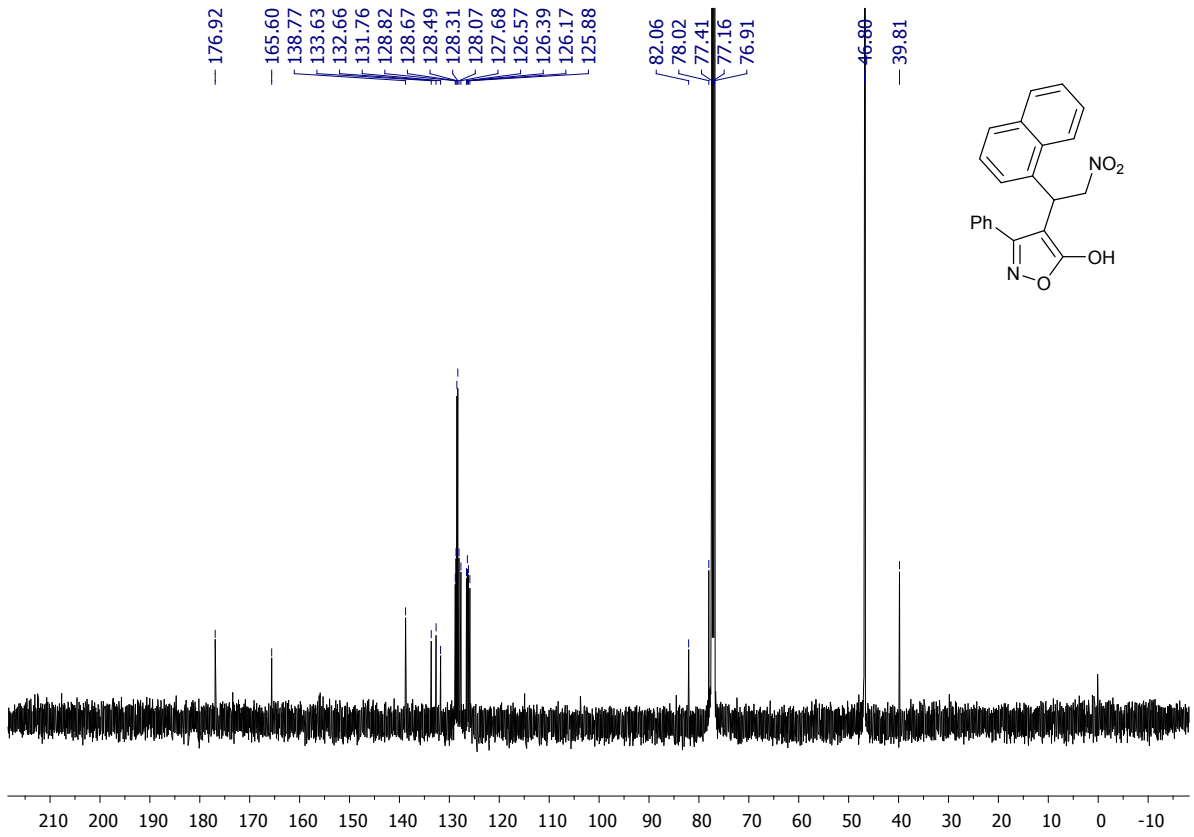
3i ^{13}C-NMR (125 MHz, $CDCl_3$): In absence of DABCO



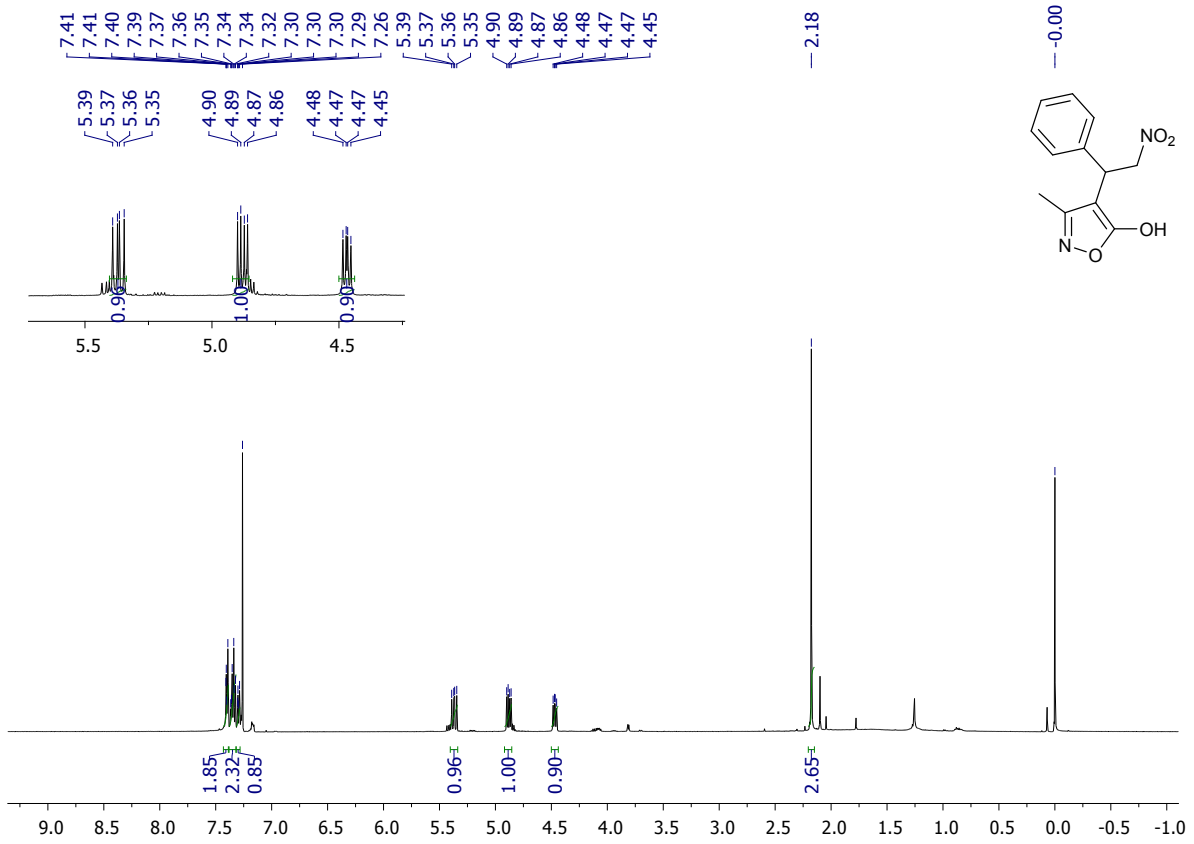
3j ^{1}H-NMR (500 MHz, $CDCl_3$): In presence of DABCO (δ 2.9-3.1 ppm)



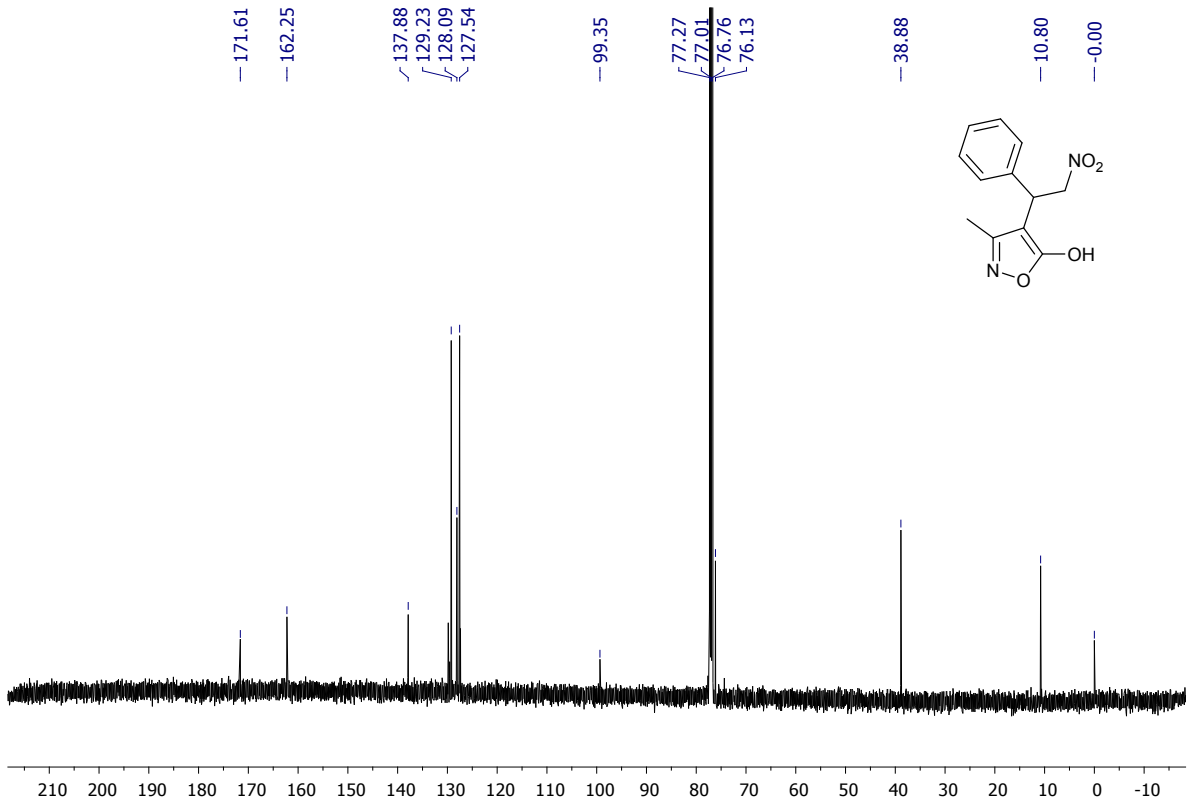
3j ^{13}C-NMR (125 MHz, $CDCl_3$): In presence of DABCO (δ 44-46 ppm)



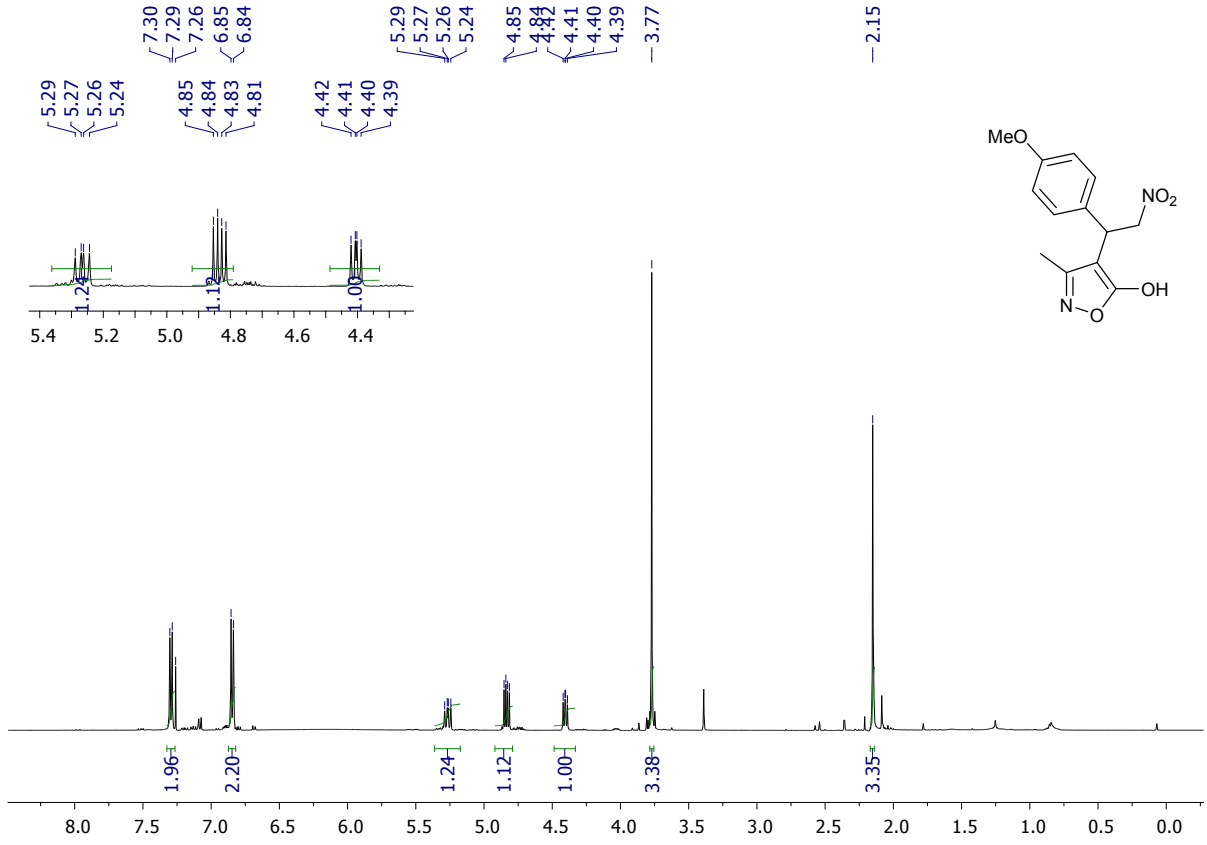
3k ^{1}H-NMR (500 MHz, $CDCl_3$): In absence of DABCO



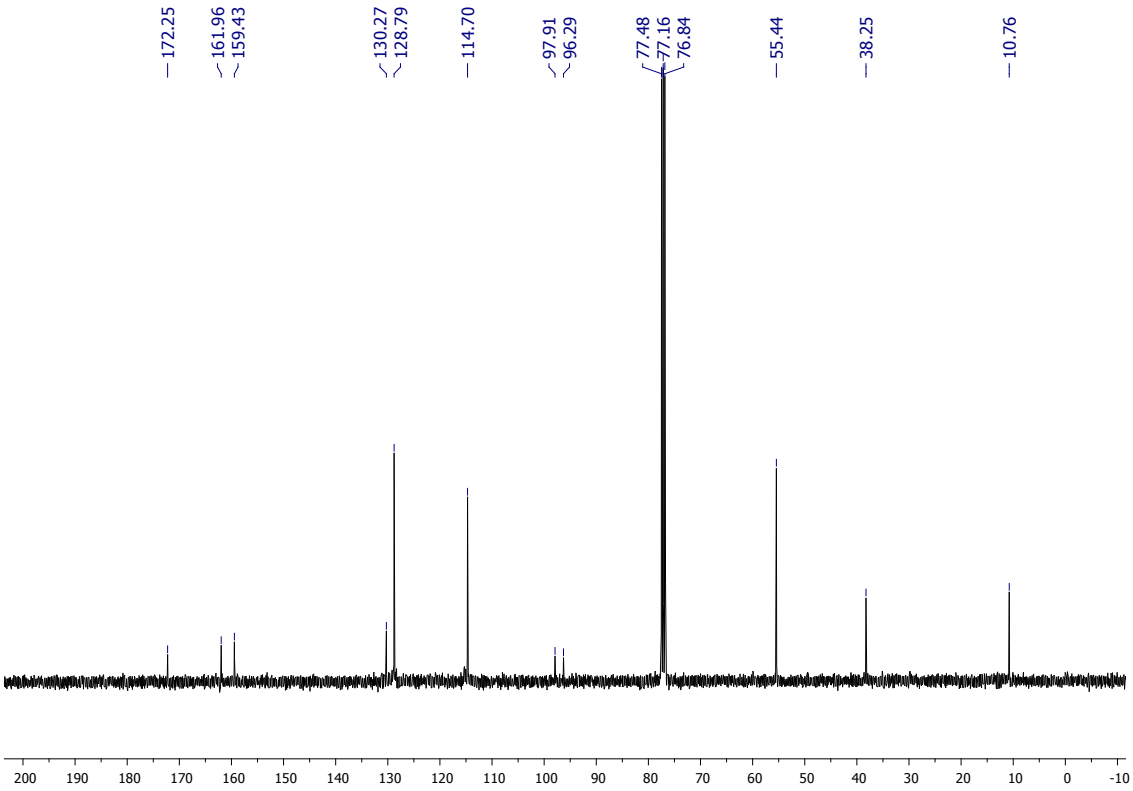
3k ^{13}C-NMR (125 MHz, $CDCl_3$): In absence of DABCO



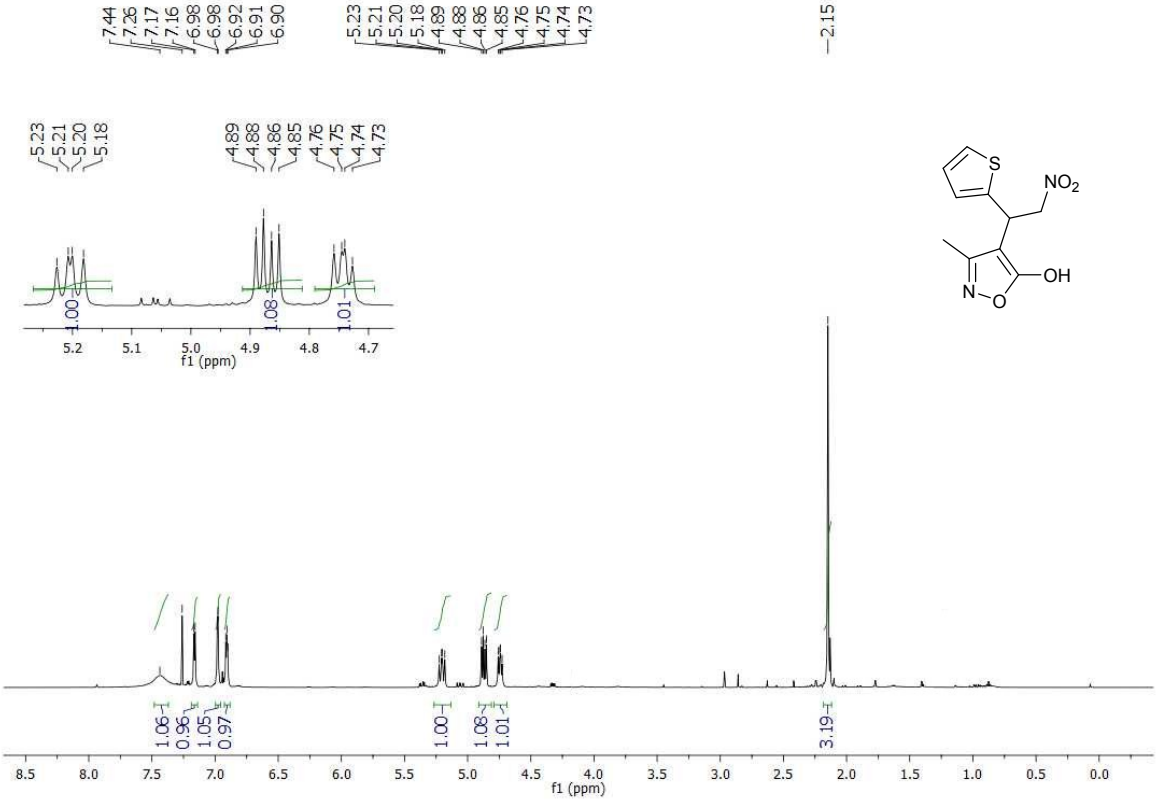
3l ^{1}H-NMR (500 MHz, $CDCl_3$): In absence of DABCO



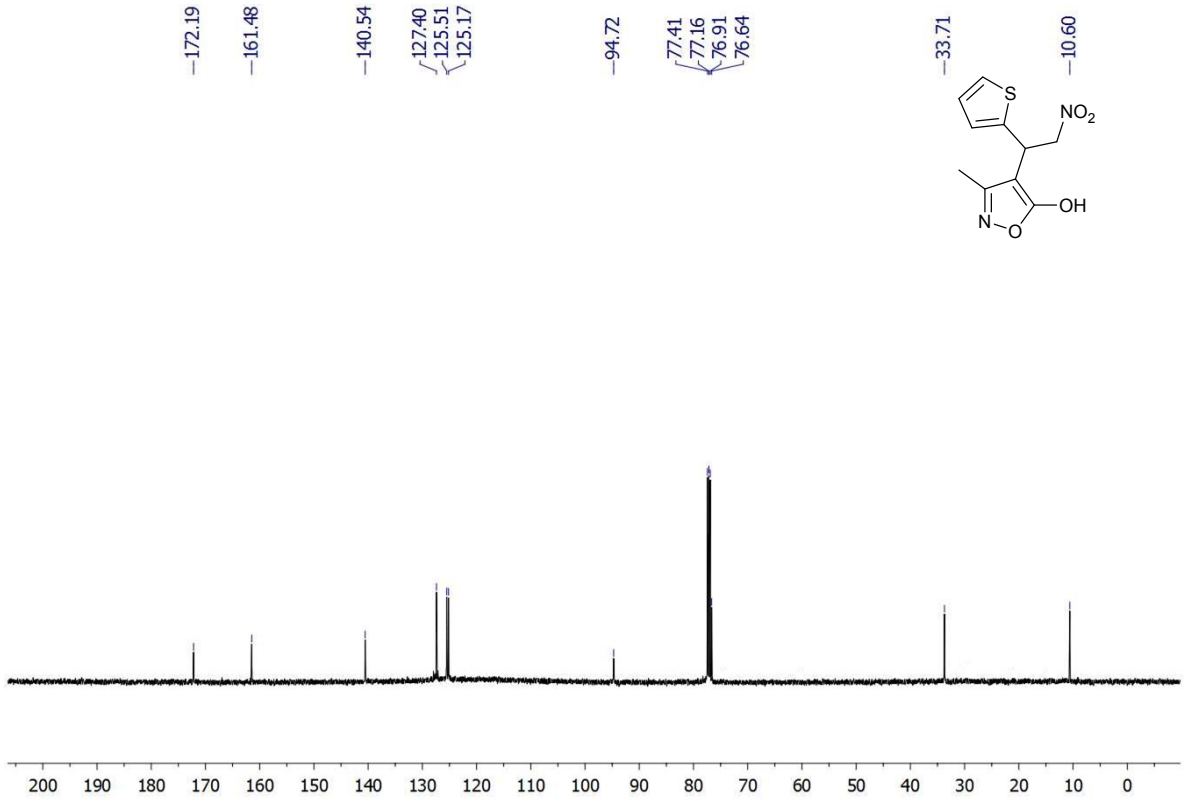
3l ^{13}C-NMR (100 MHz, $CDCl_3$): In absence of DABCO



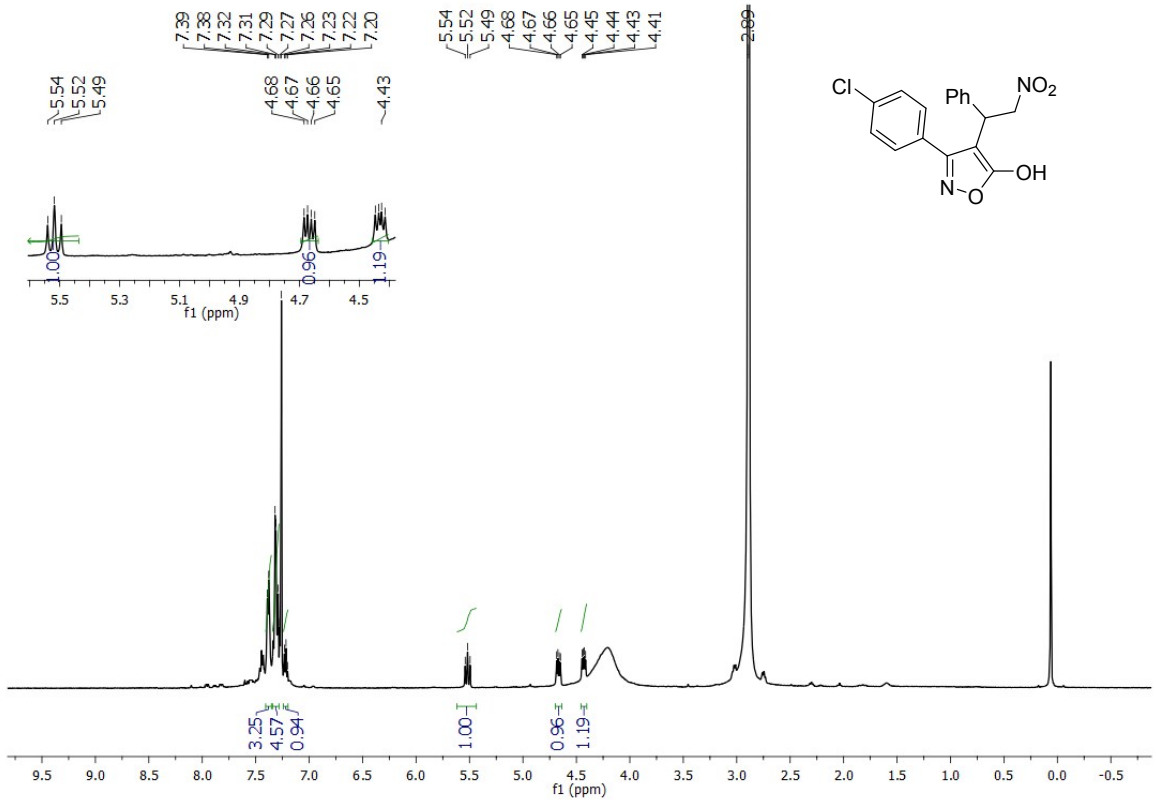
3m ^{1}H-NMR (500 MHz, $CDCl_3$): In absence of DABCO



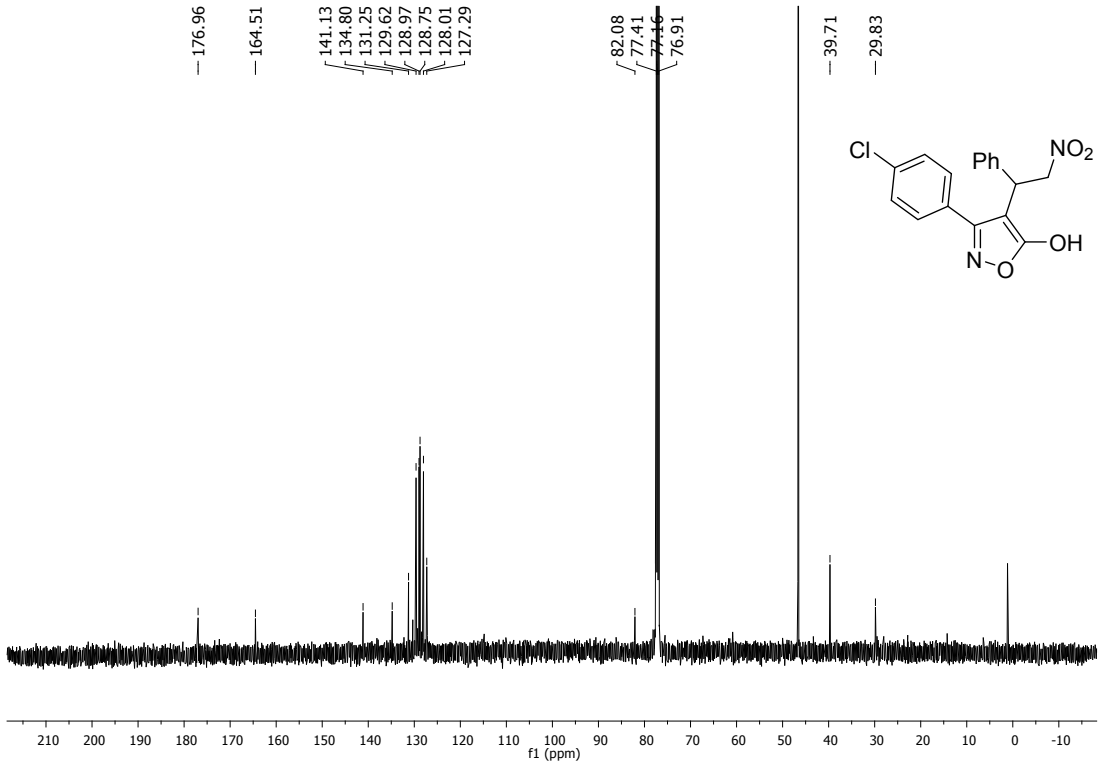
3m ^{13}C-NMR (125 MHz, $CDCl_3$): In absence of DABCO



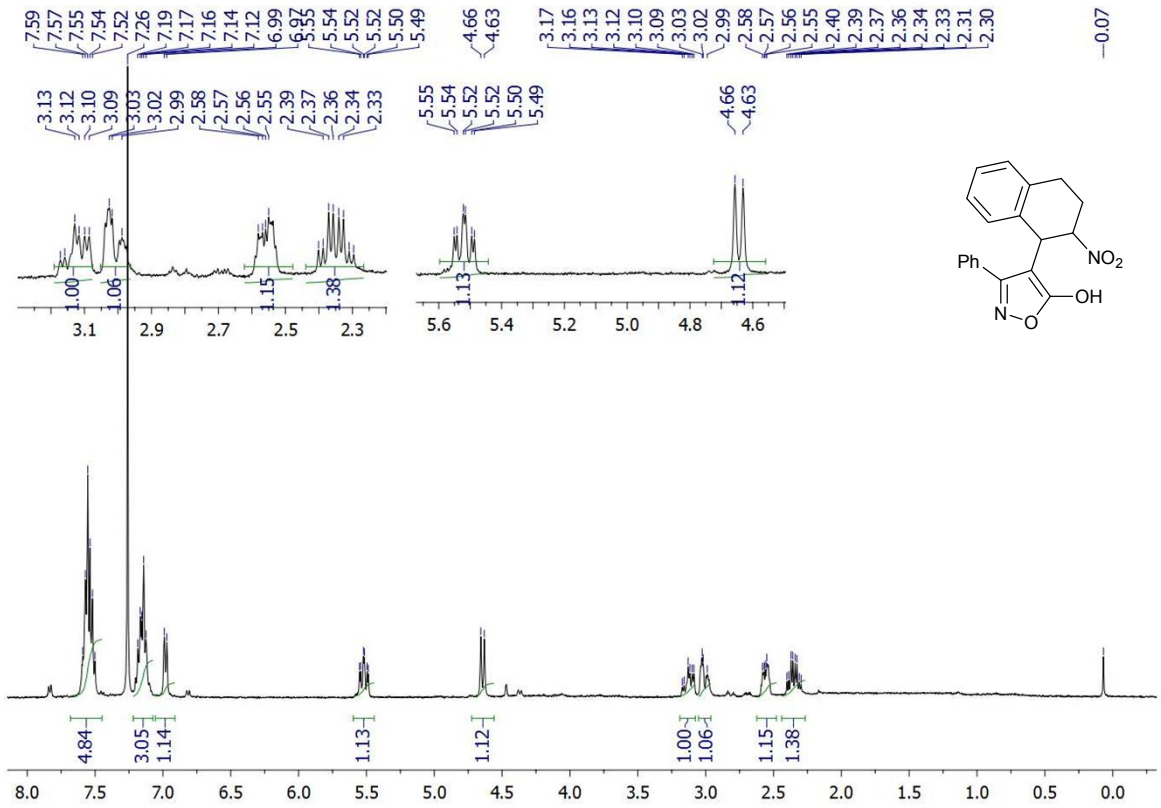
3n ^{1}H-NMR (500 MHz, $CDCl_3$): In presence of DABCO (δ 2.9-3.1 ppm)



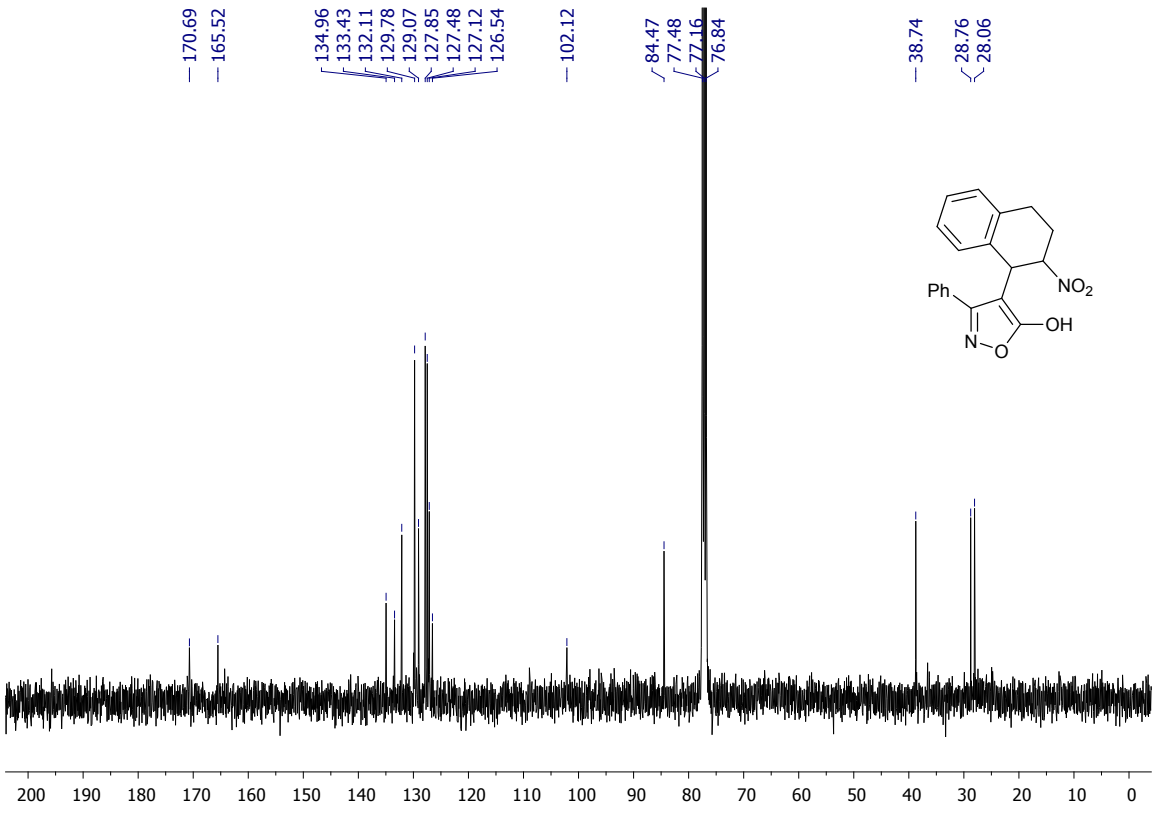
3n ^{13}C-NMR (125 MHz, $CDCl_3$): In presence of DABCO (δ 44-46 ppm)



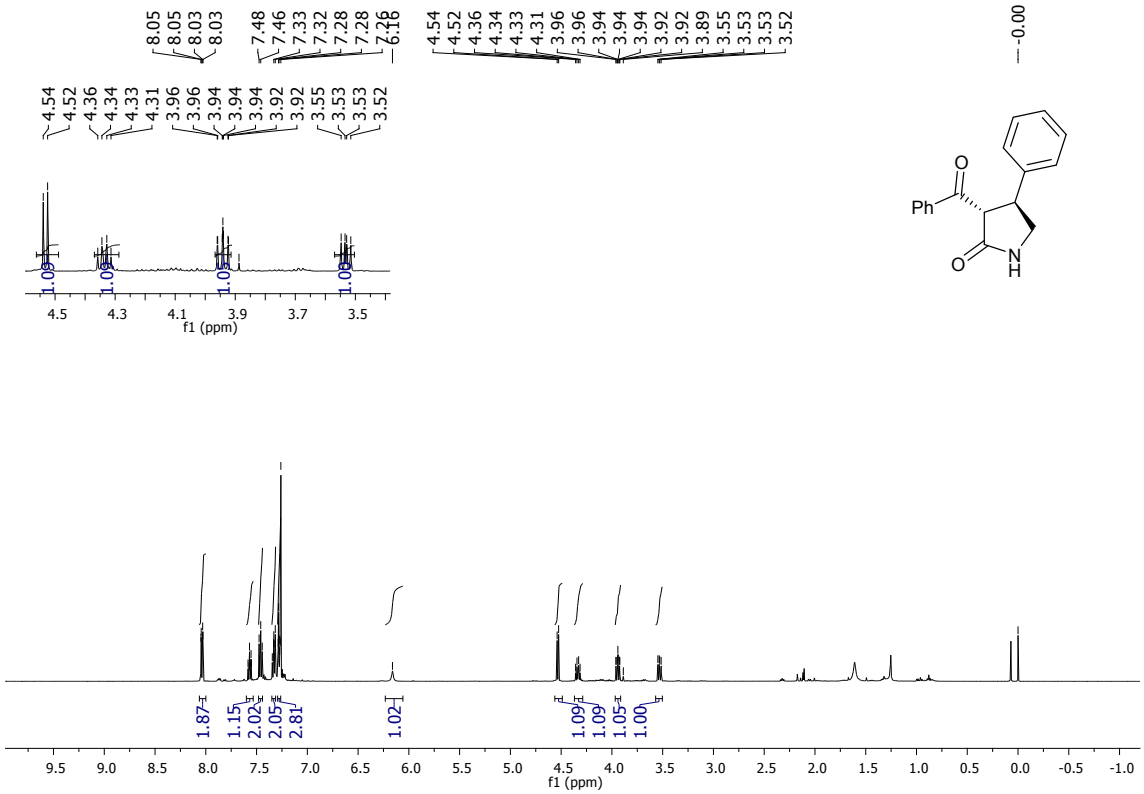
3o ^{1}H-NMR (400 MHz, $CDCl_3$): In absence of DABCO



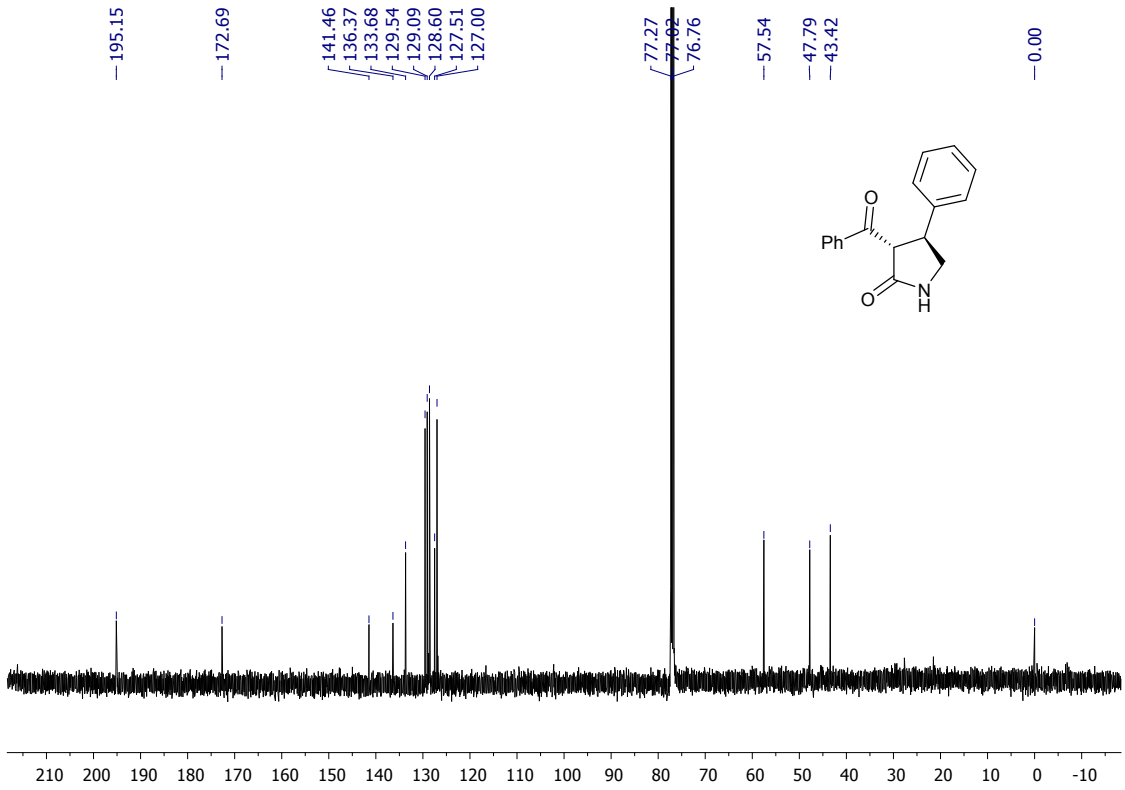
3o ^{13}C-NMR (100 MHz, $CDCl_3$): In absence of DABCO



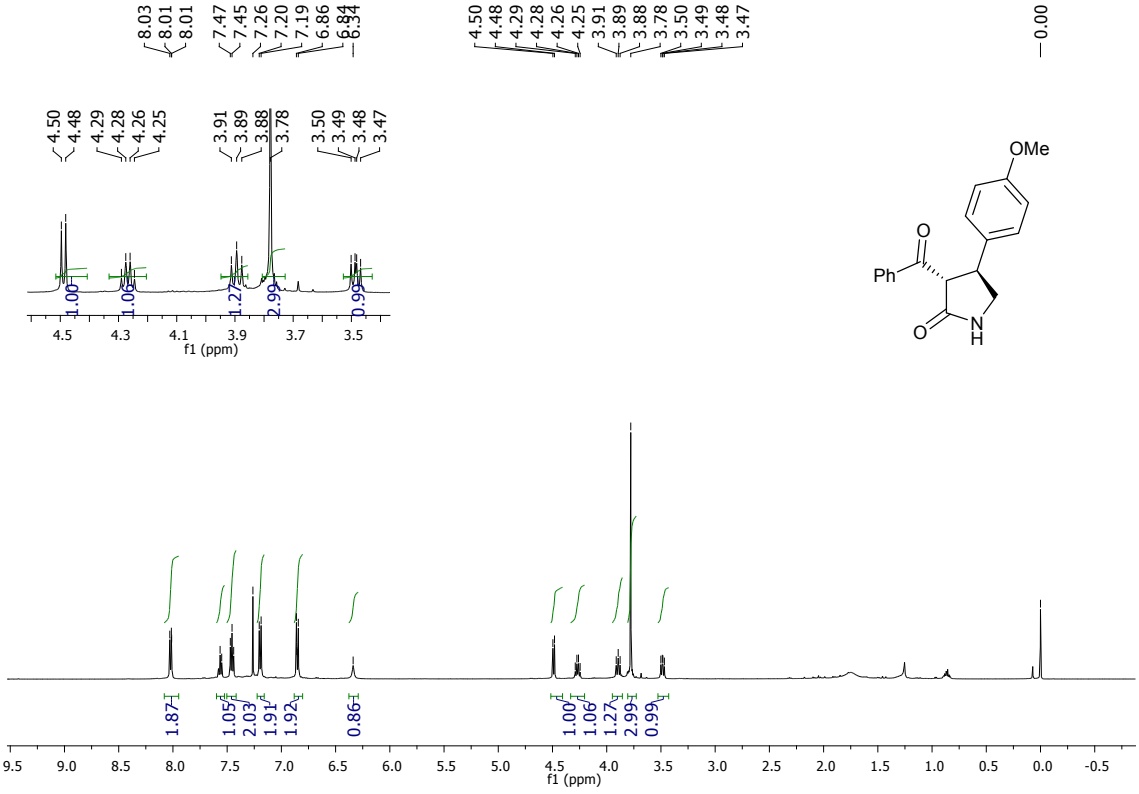
4a ^{1}H-NMR (500 MHz, $CDCl_3$)



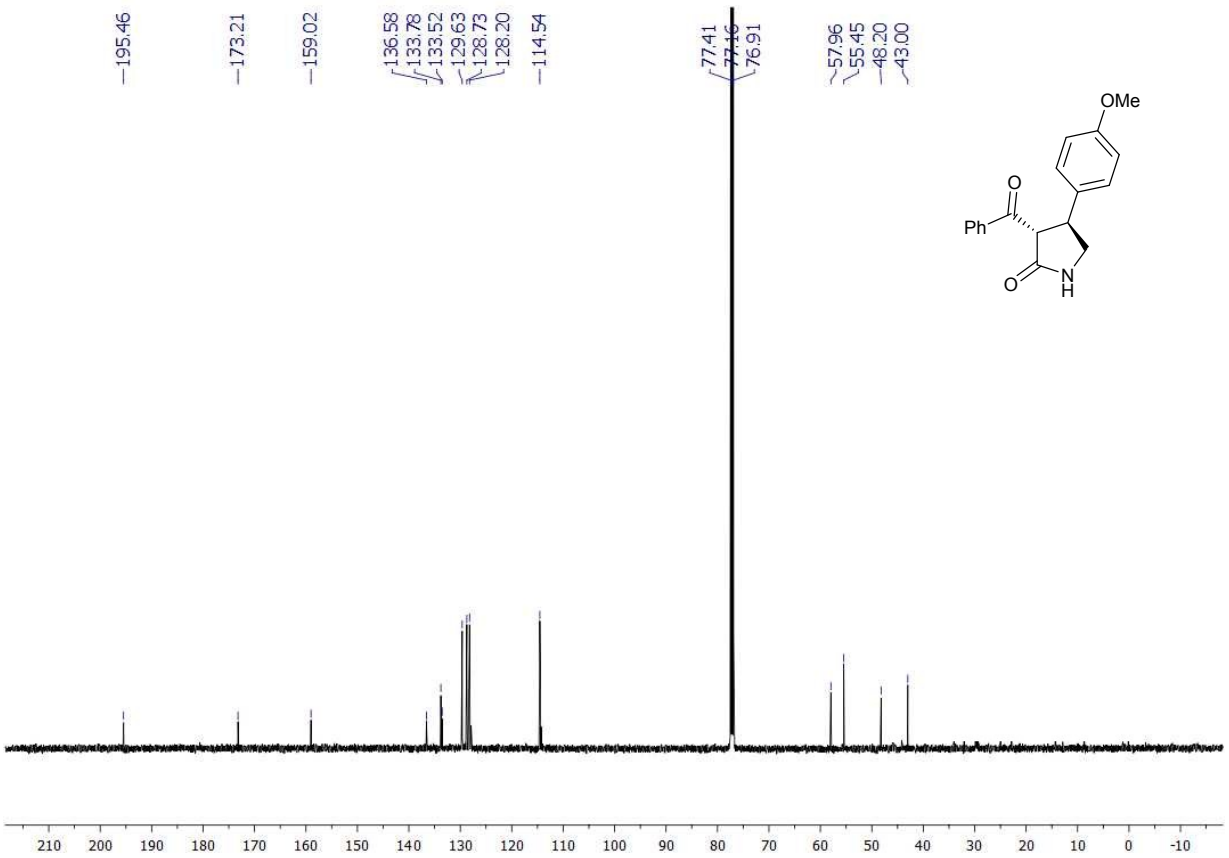
4a ^{13}C-NMR (125 MHz, $CDCl_3$)



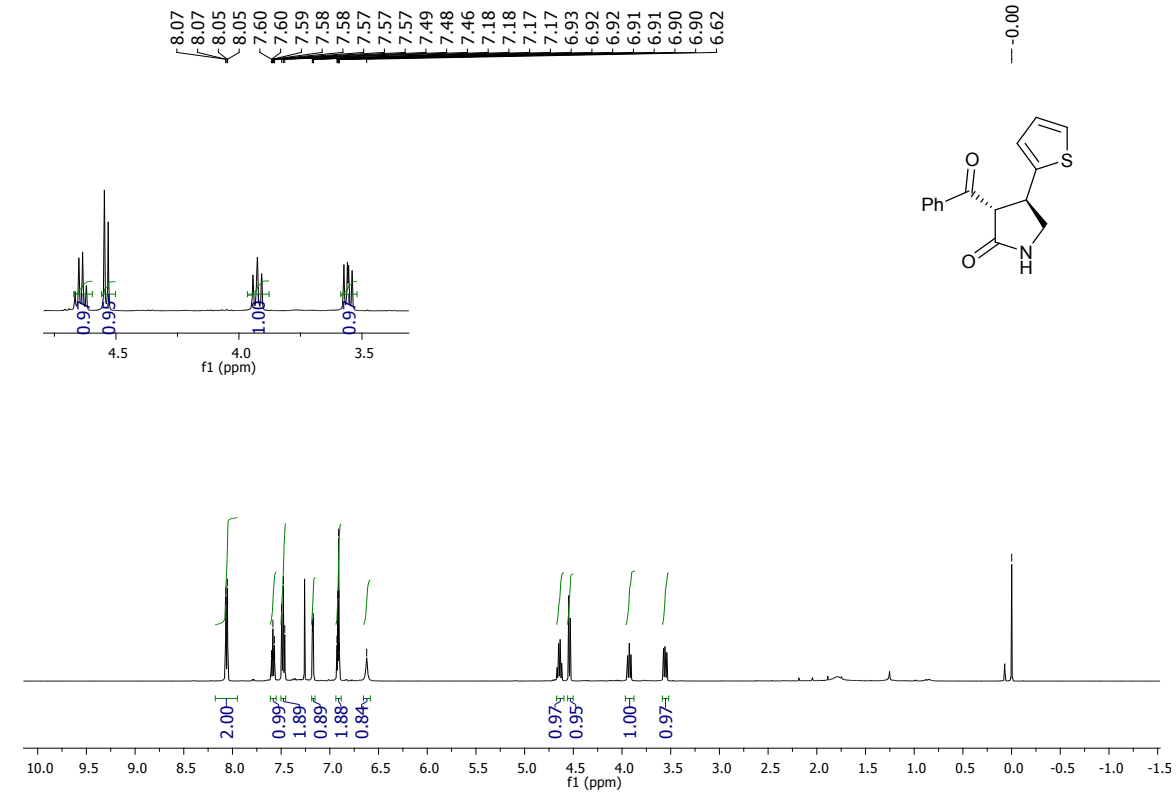
4b ^{1}H-NMR (500MHz, $CDCl_3$)



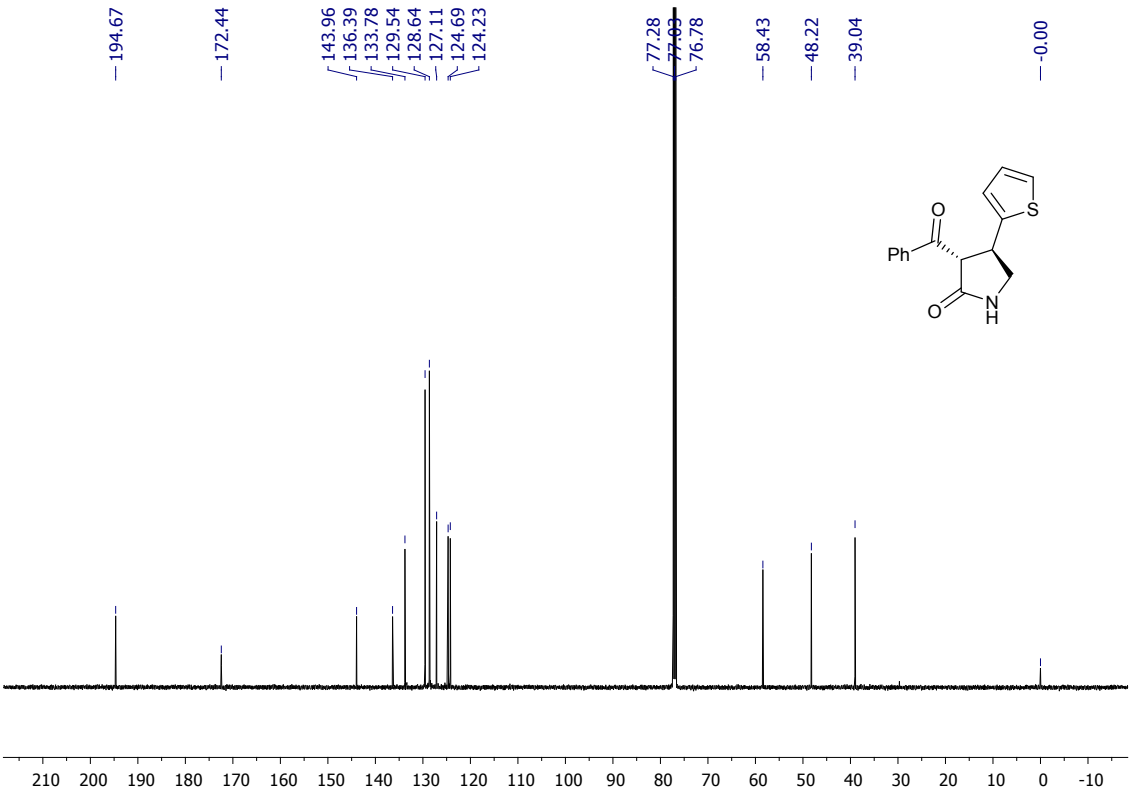
4b ^{13}C-NMR (125 MHz, $CDCl_3$)



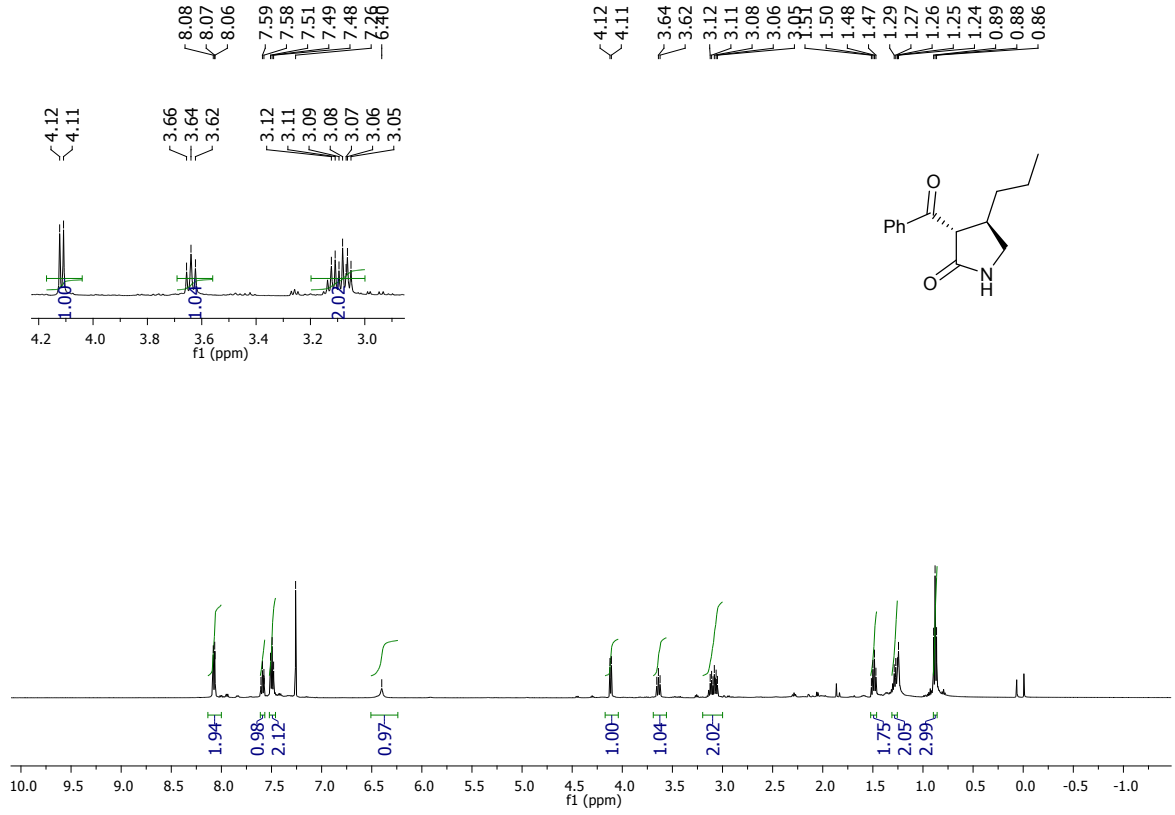
4c ^{1}H-NMR (500 MHz, $CDCl_3$)



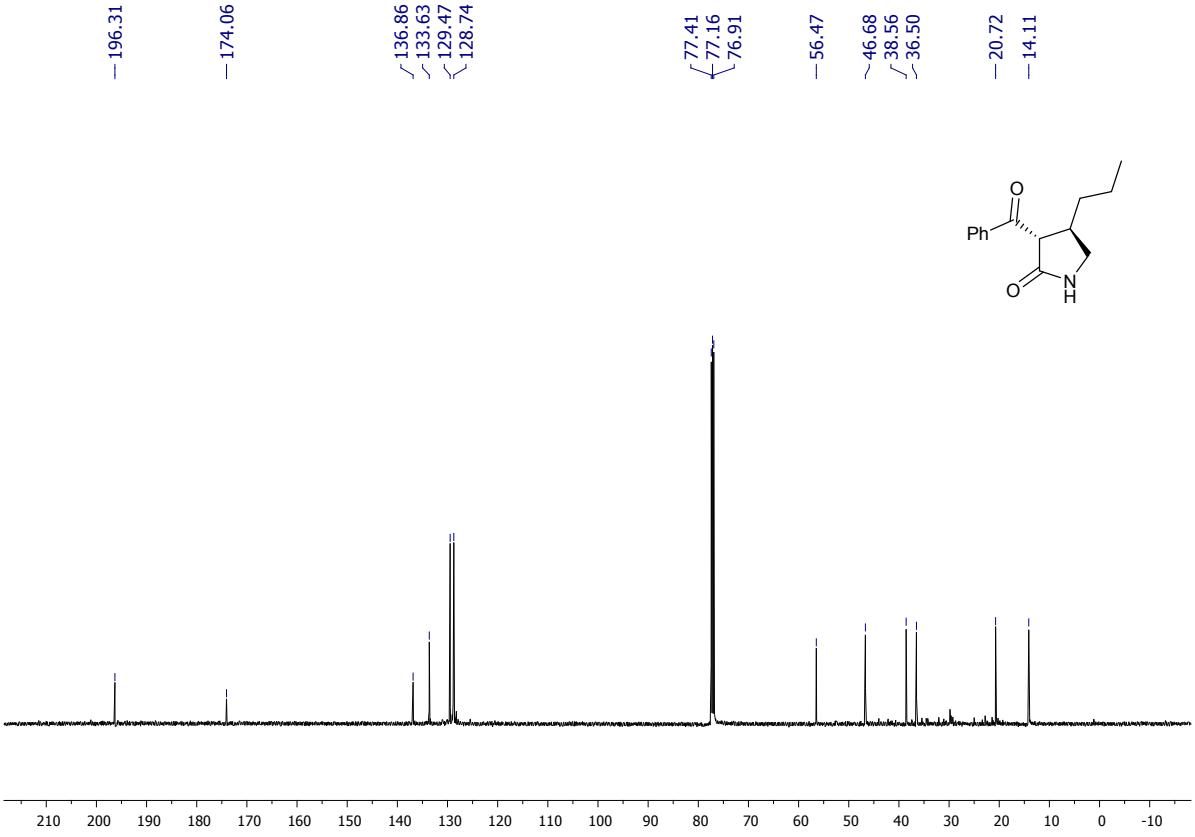
4c ^{13}C-NMR (125 MHz, $CDCl_3$)



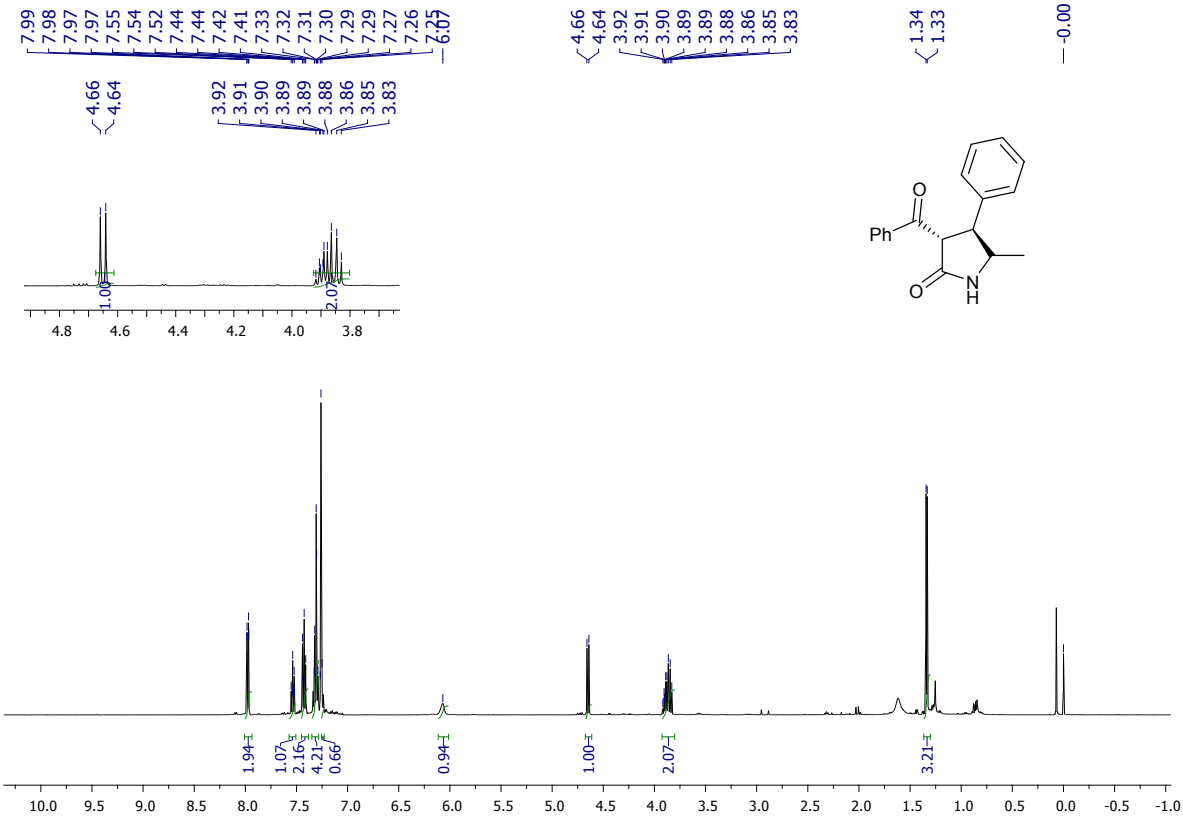
4d ^{1}H-NMR (500 MHz, $CDCl_3$)



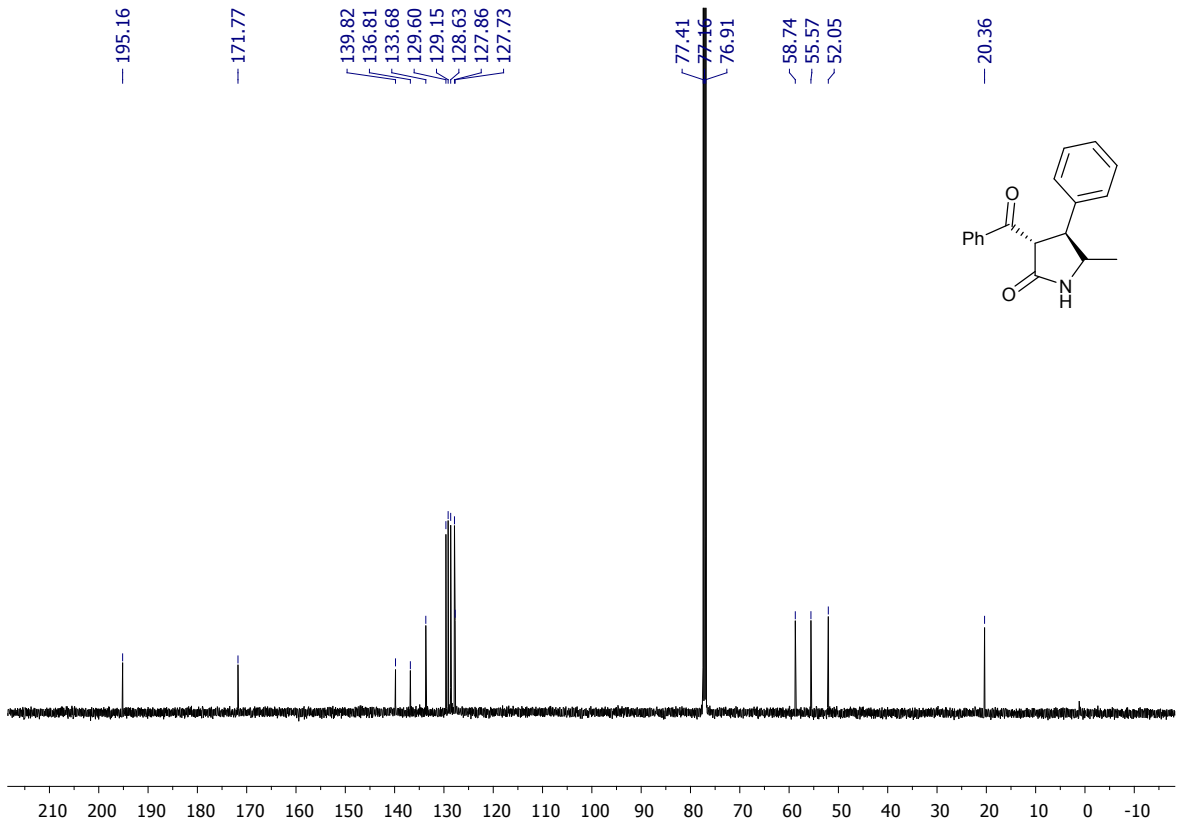
4d ^{13}C-NMR (125 MHz, $CDCl_3$)



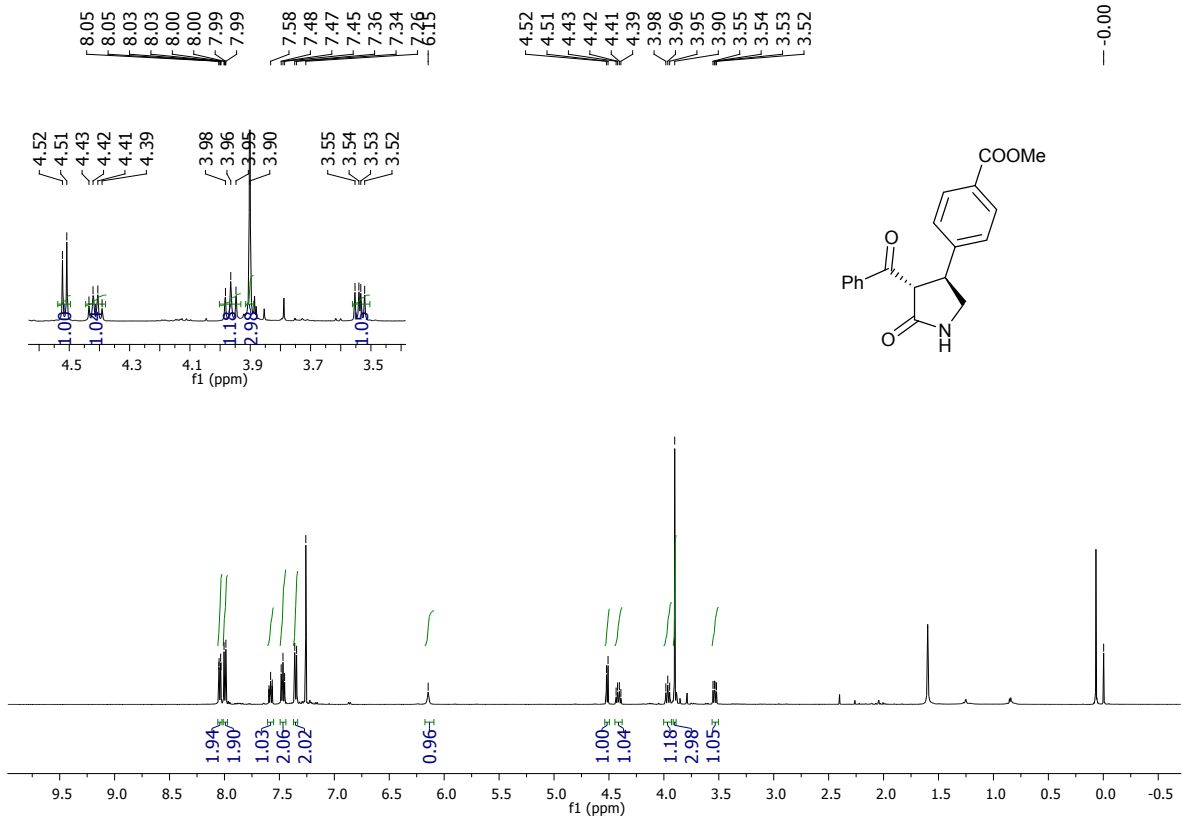
4e ^{1}H-NMR (500 MHz, $CDCl_3$)



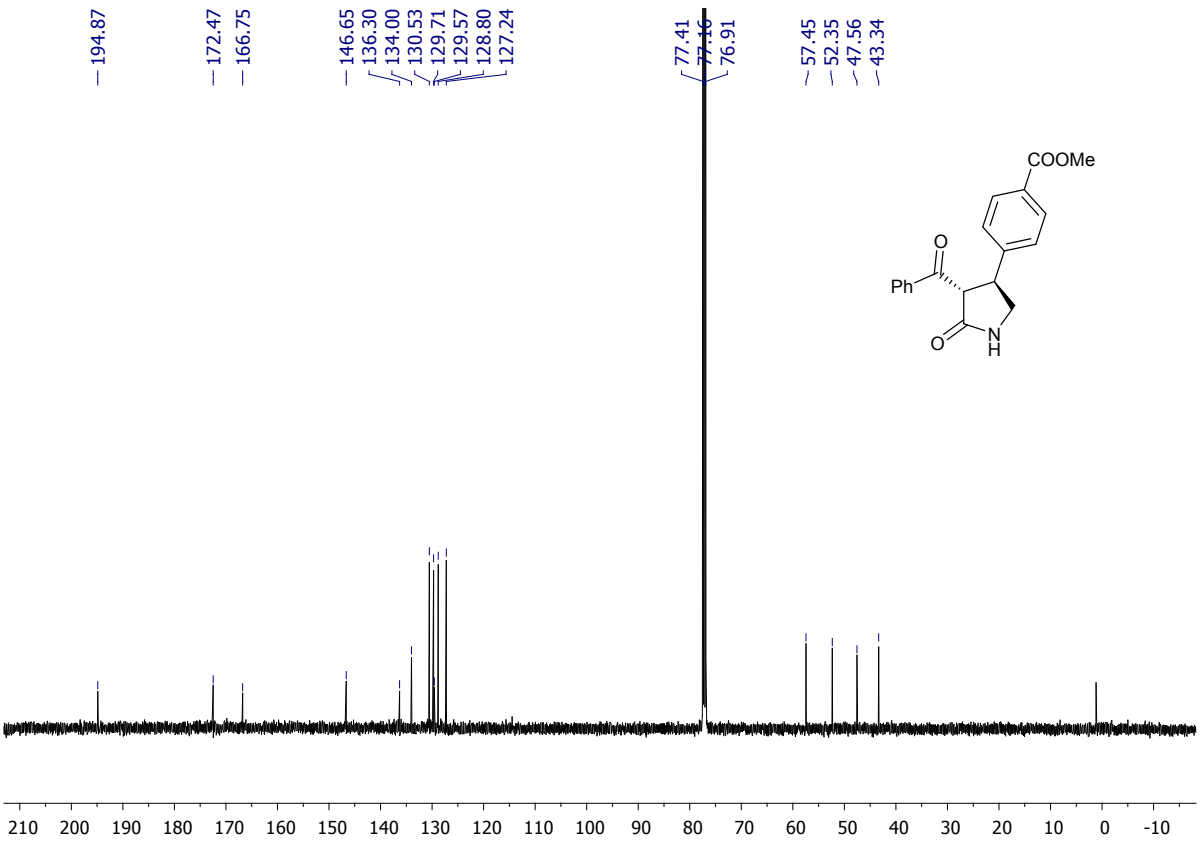
4e ^{13}C-NMR (125 MHz, $CDCl_3$)



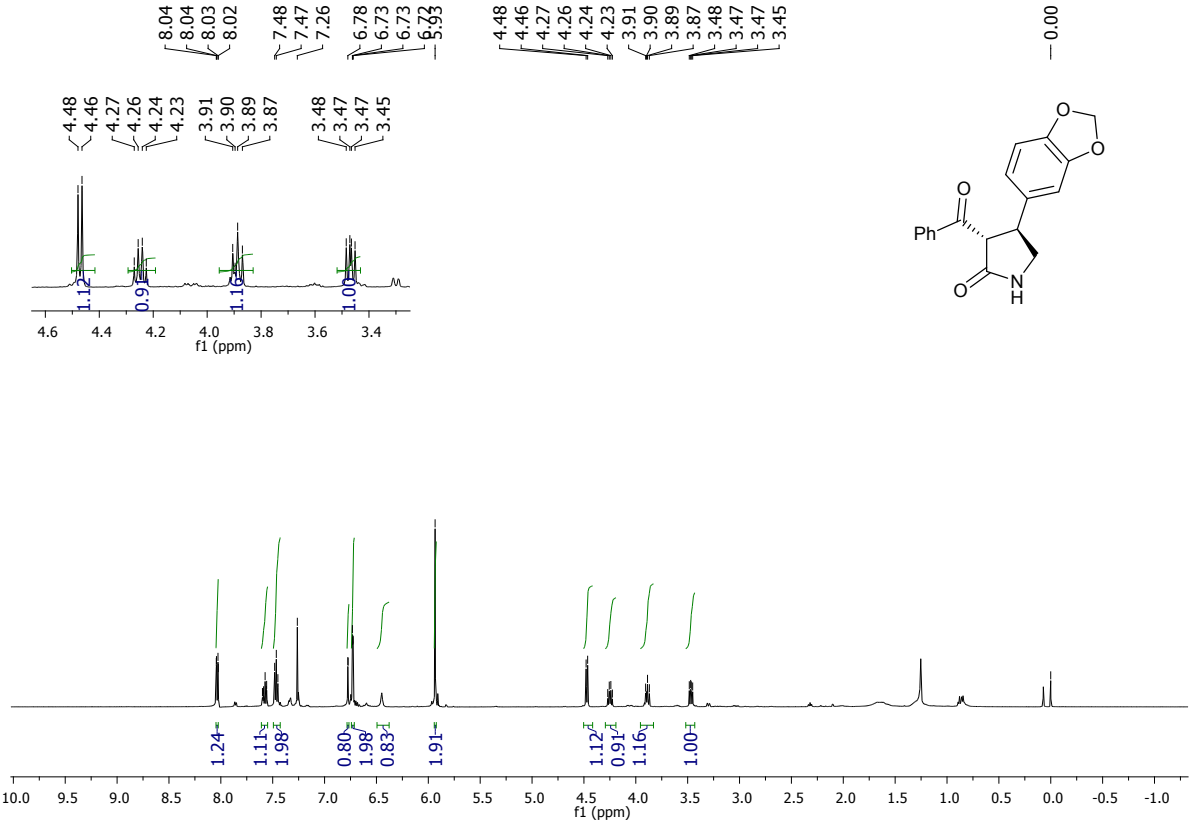
4f ^{1}H-NMR (500 MHz, $CDCl_3$)



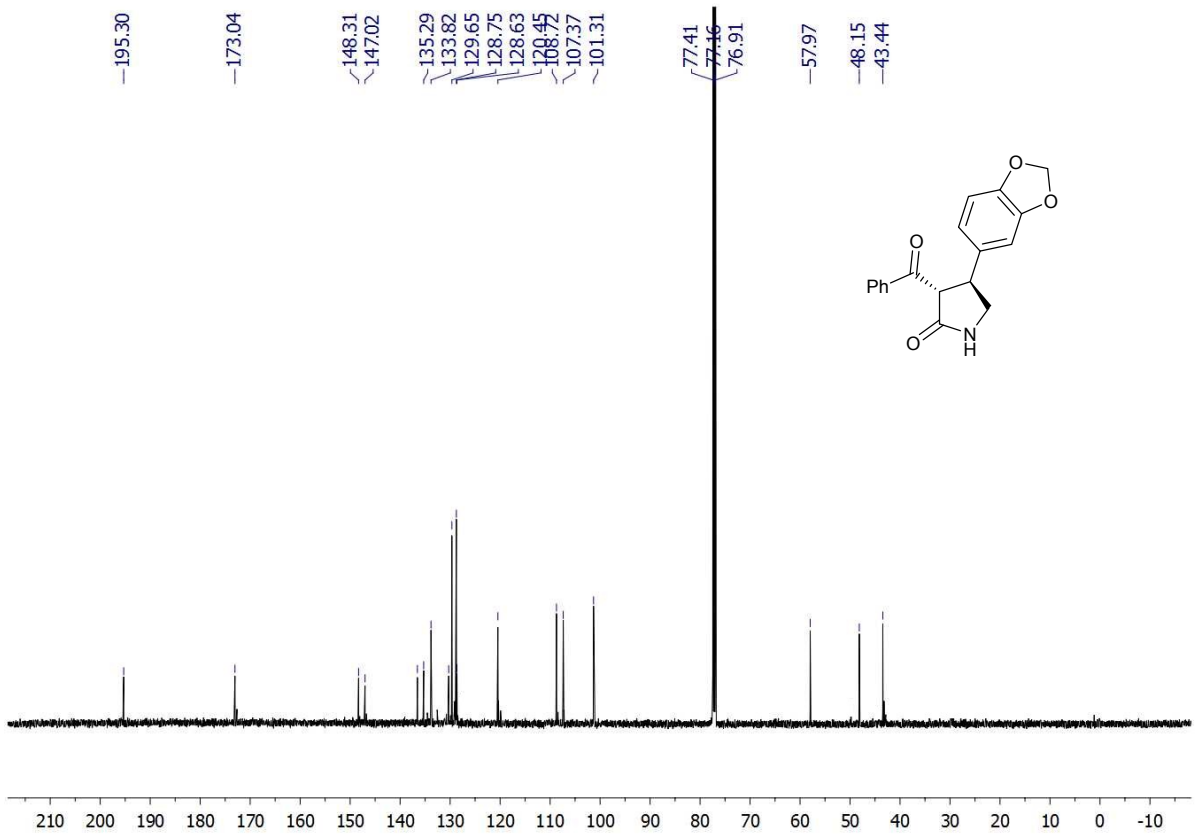
4f ^{13}C -NMR (125 MHz, $CDCl_3$)



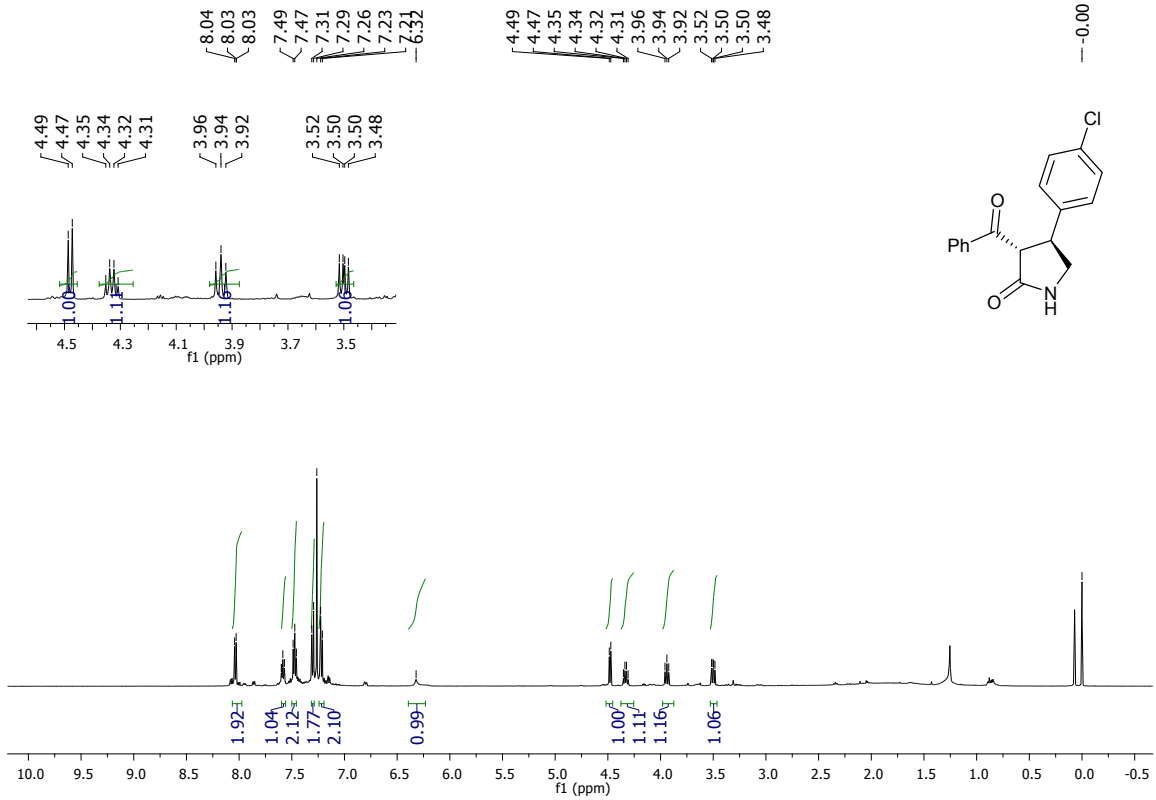
4g ^{1}H -NMR (500 MHz, $CDCl_3$)



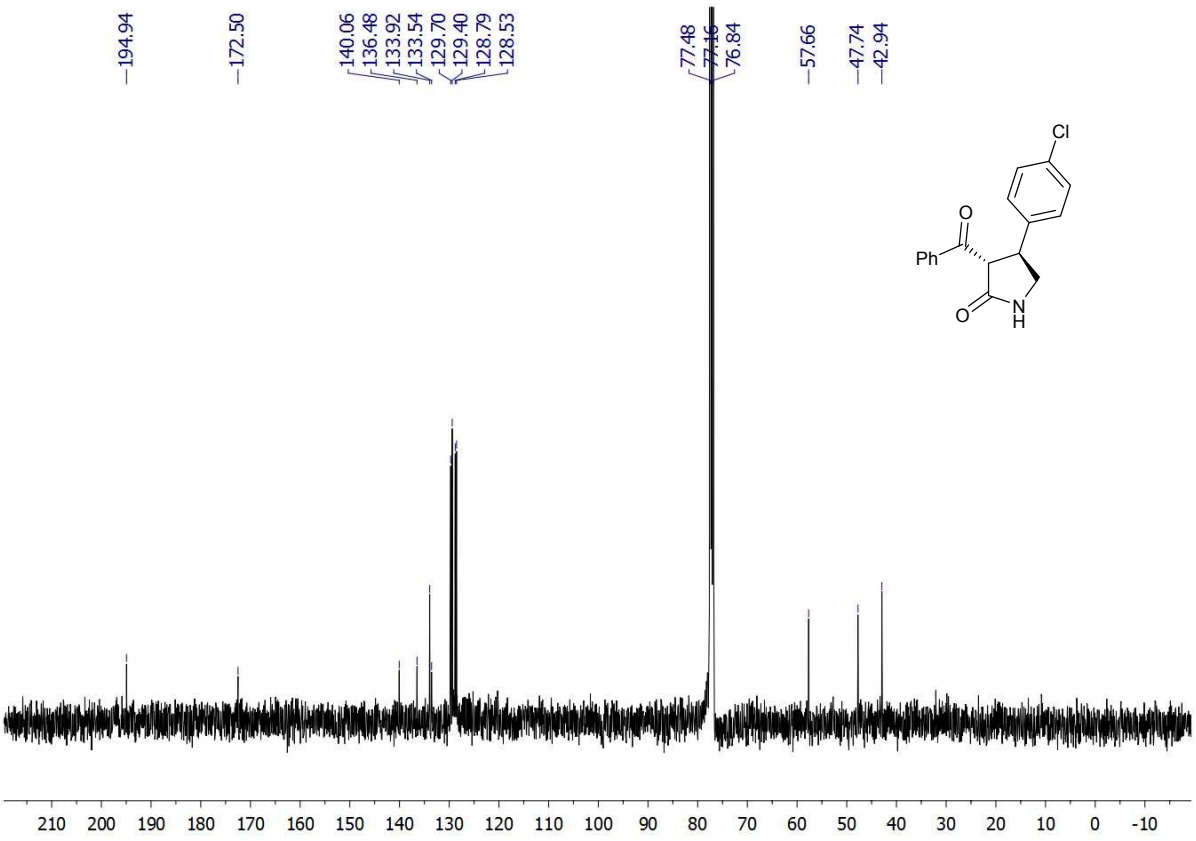
4g ^{13}C -NMR (125 MHz, $CDCl_3$)



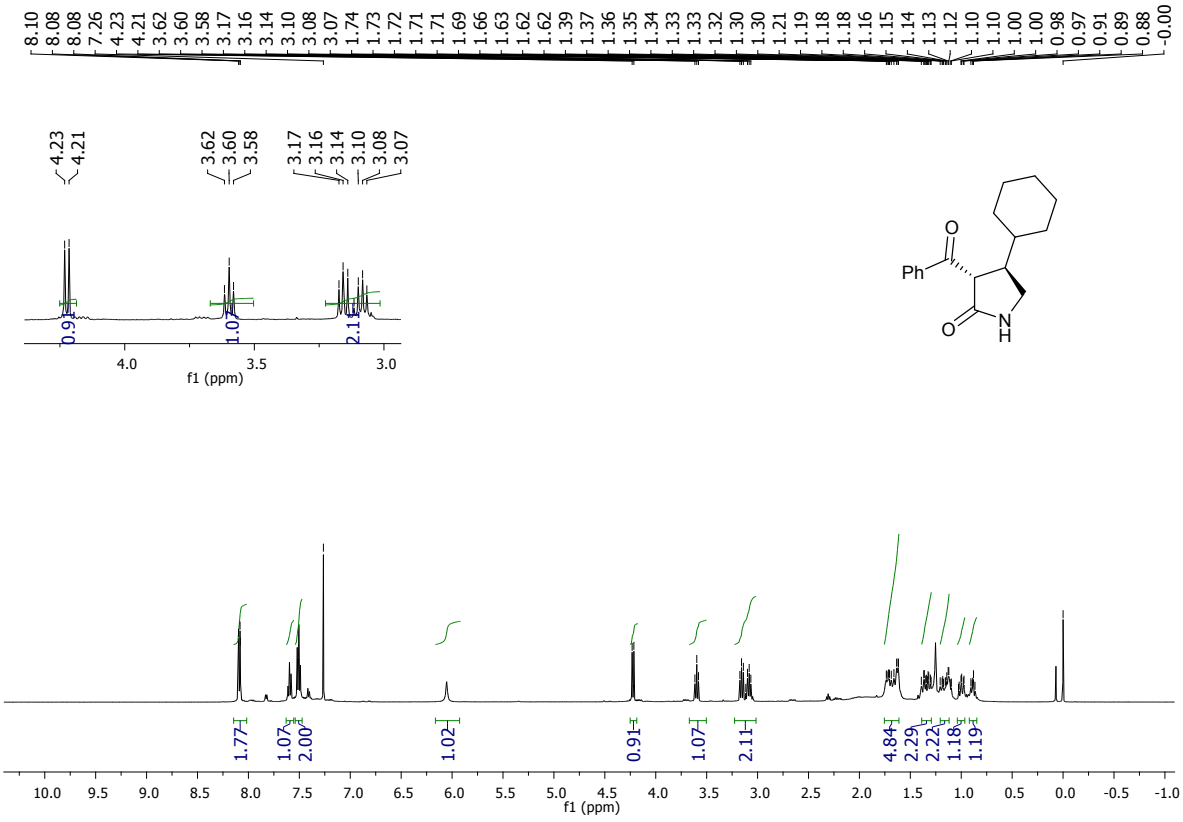
4h ^{1}H -NMR (500 MHz, $CDCl_3$)



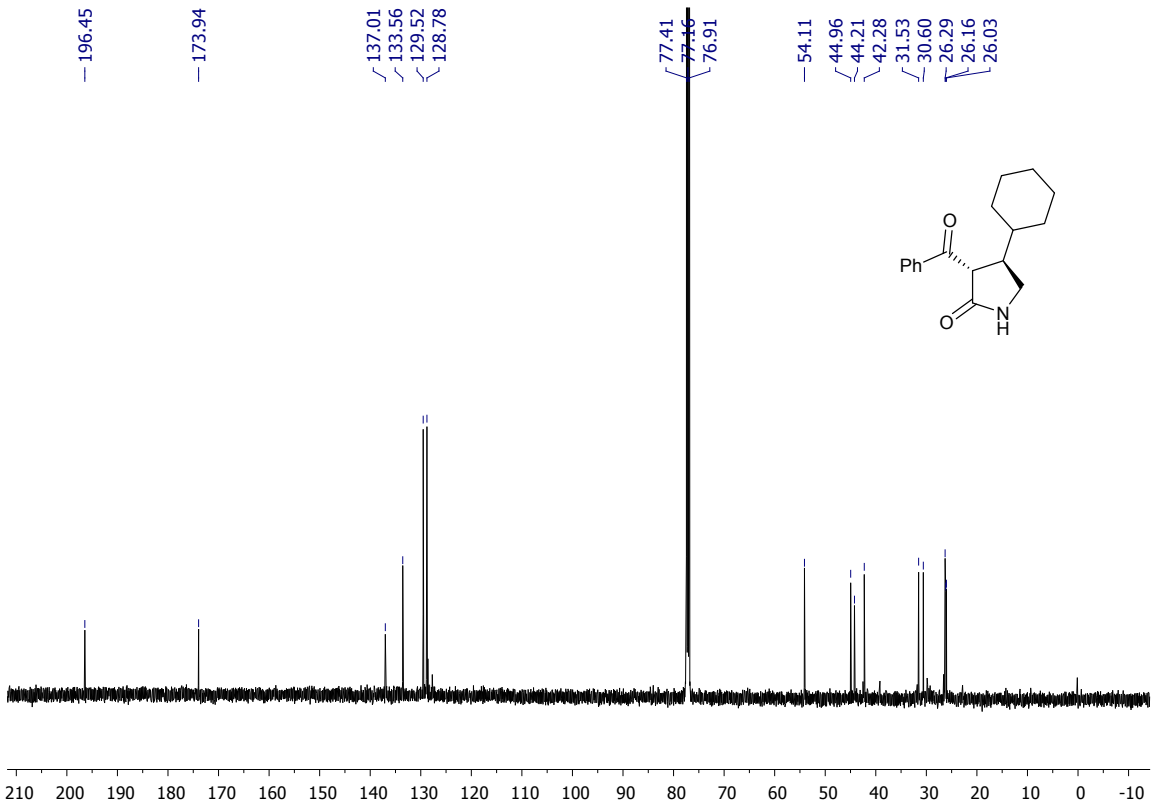
4h ^{13}C -NMR (100 MHz, $CDCl_3$)



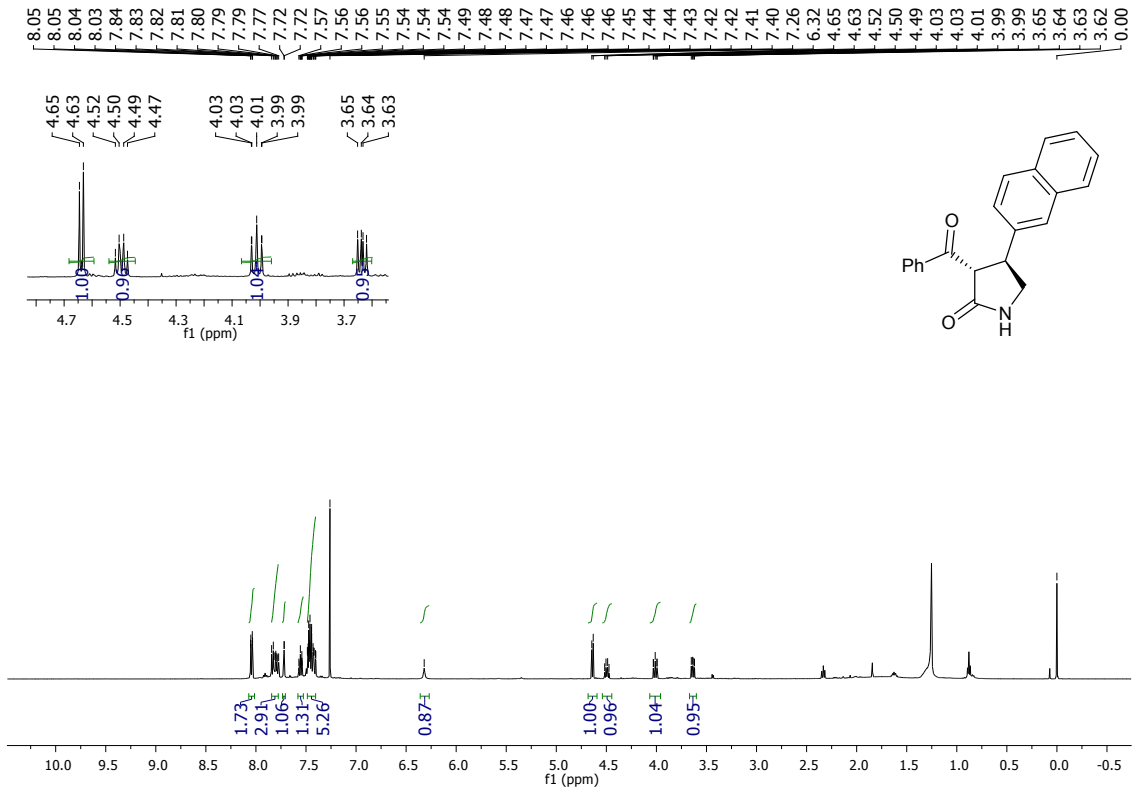
4i ^{1}H -NMR (500 MHz, $CDCl_3$)



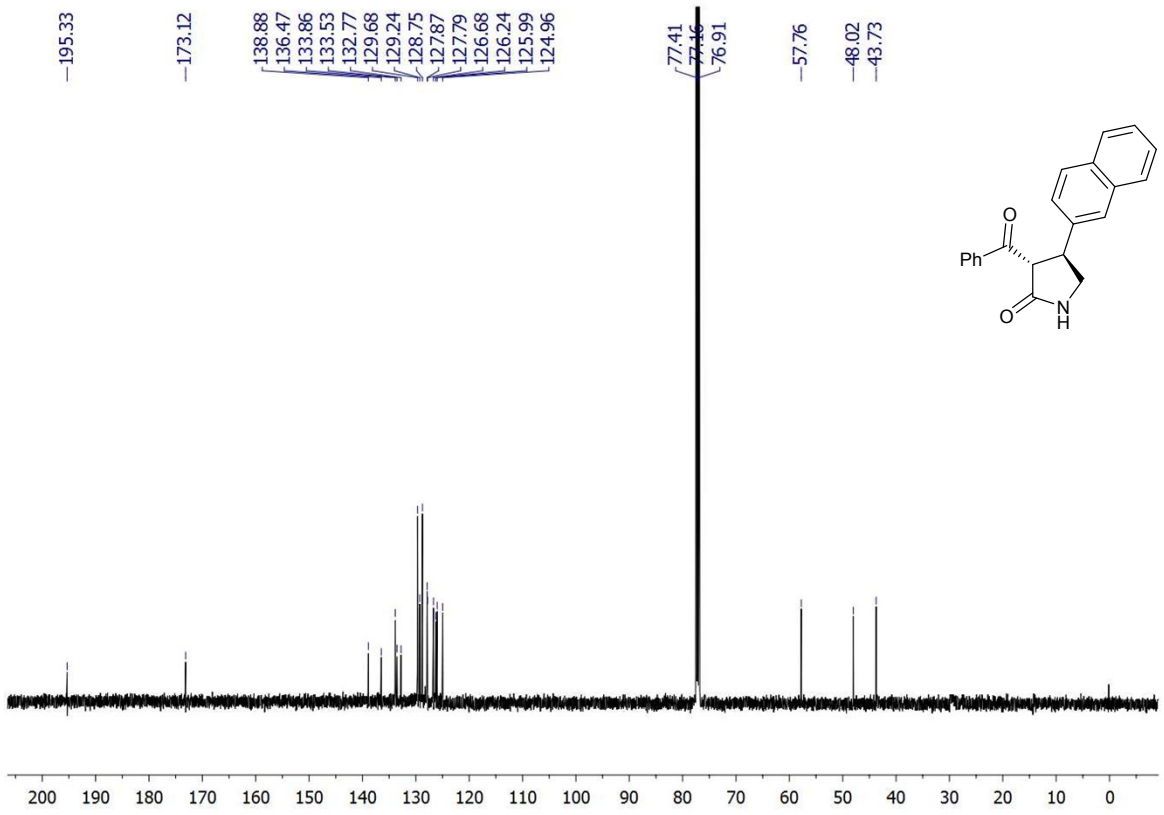
4i ^{13}C -NMR (125 MHz, $CDCl_3$)



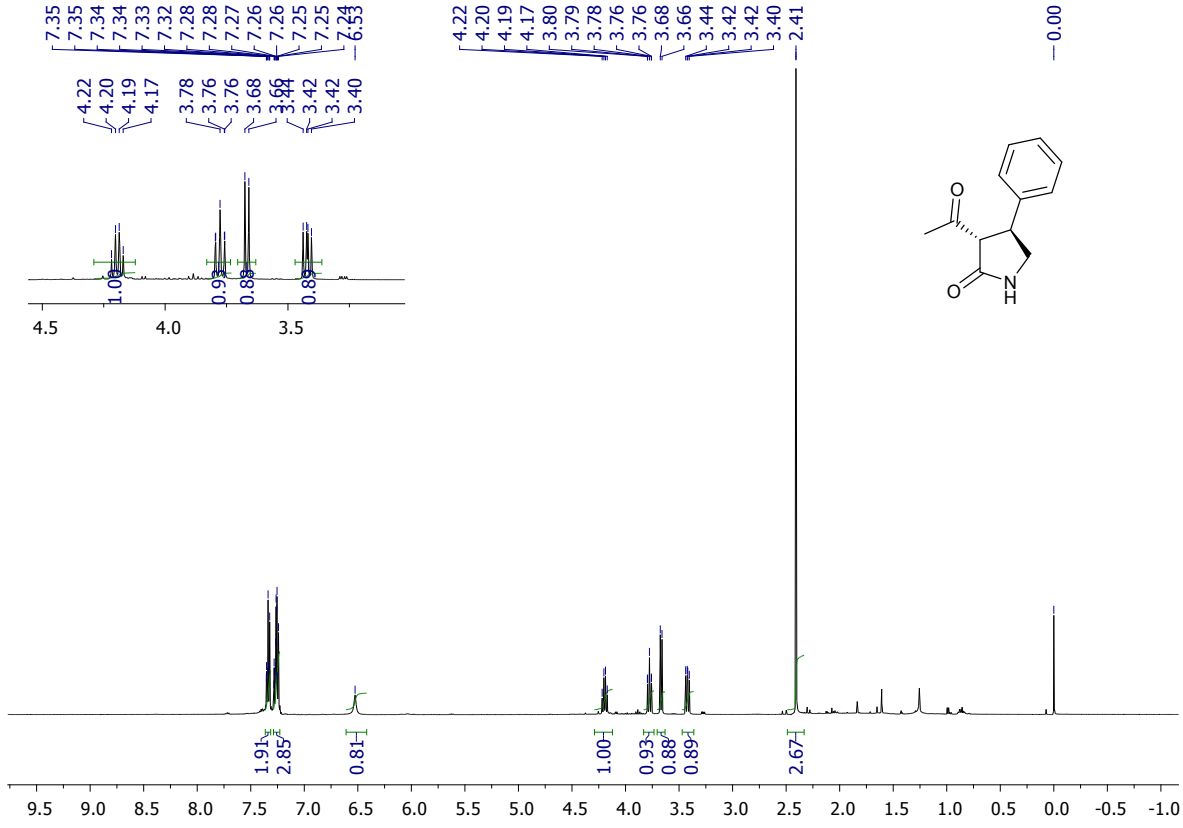
4j ^{1}H -NMR (500 MHz, $CDCl_3$)



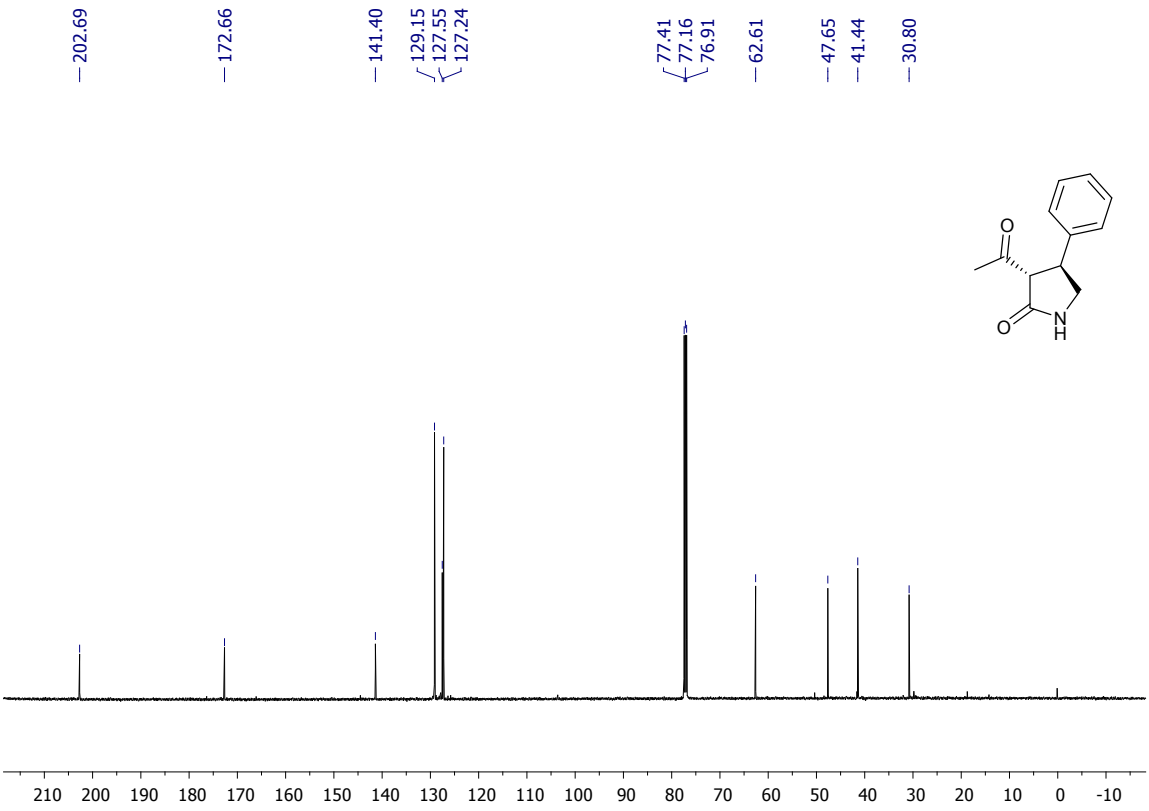
4j ^{13}C -NMR (125 MHz, $CDCl_3$)



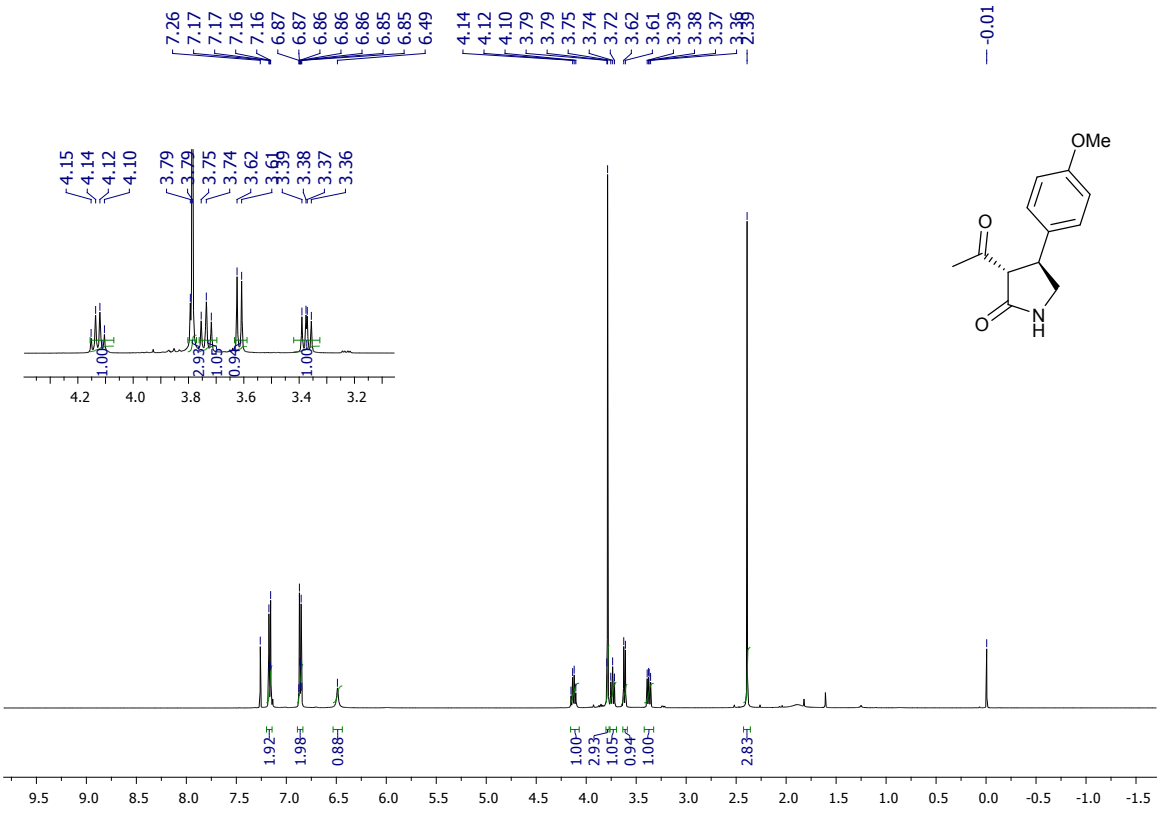
4k ^{1}H NMR (500 MHz, $CDCl_3$)



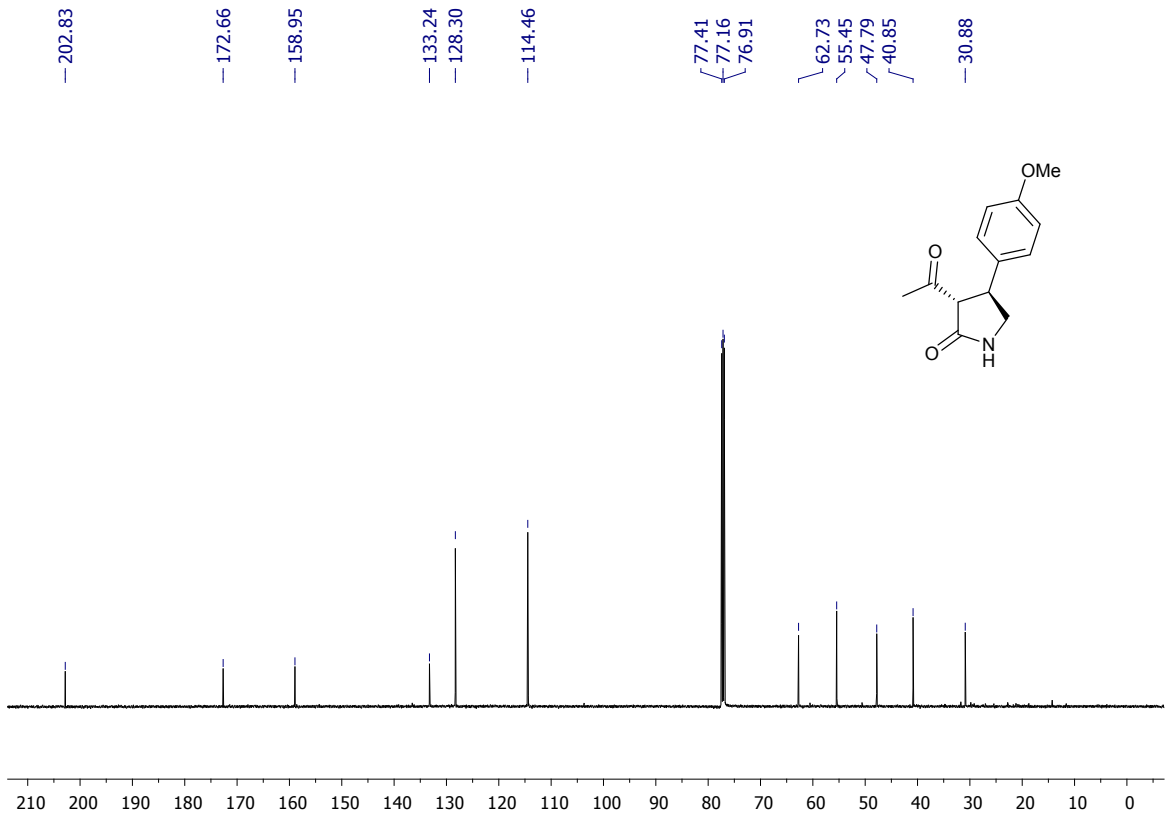
4k ^{13}C-NMR (125 MHz, $CDCl_3$)



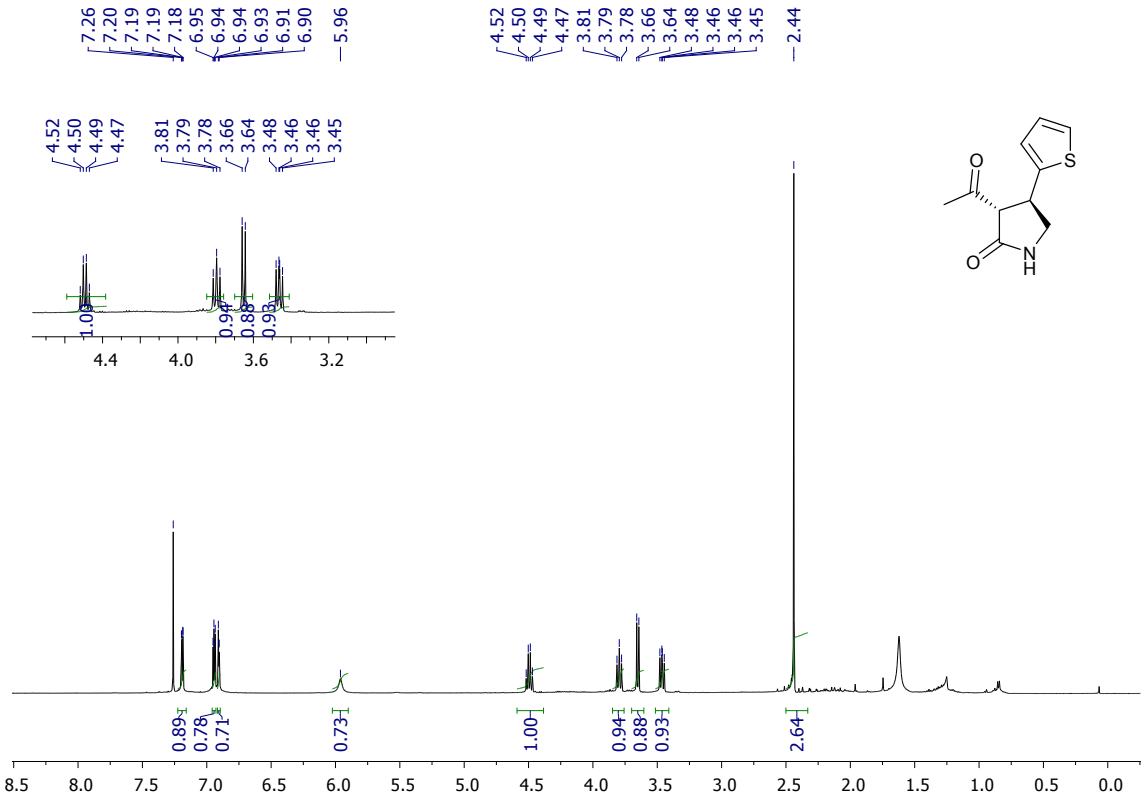
4l ^{1}H-NMR (500 MHz, $CDCl_3$)



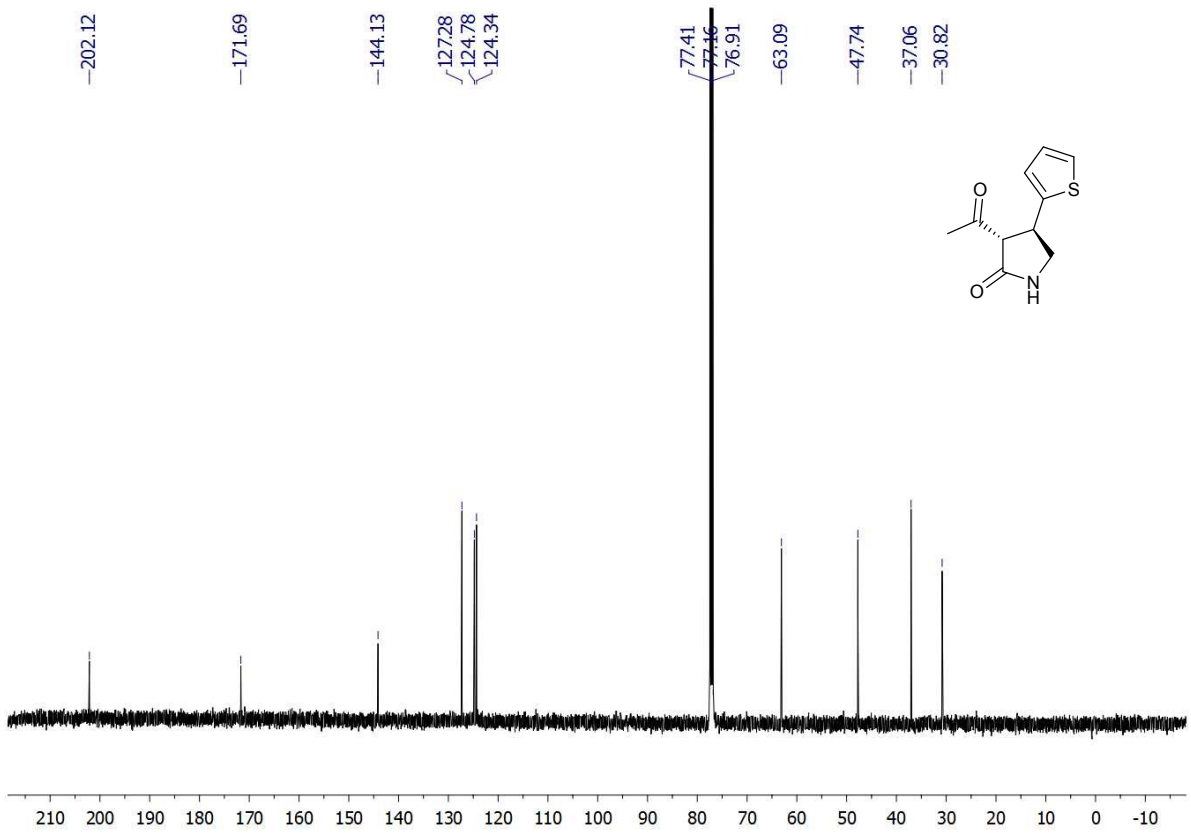
4l ^{13}C-NMR (125 MHz, $CDCl_3$)



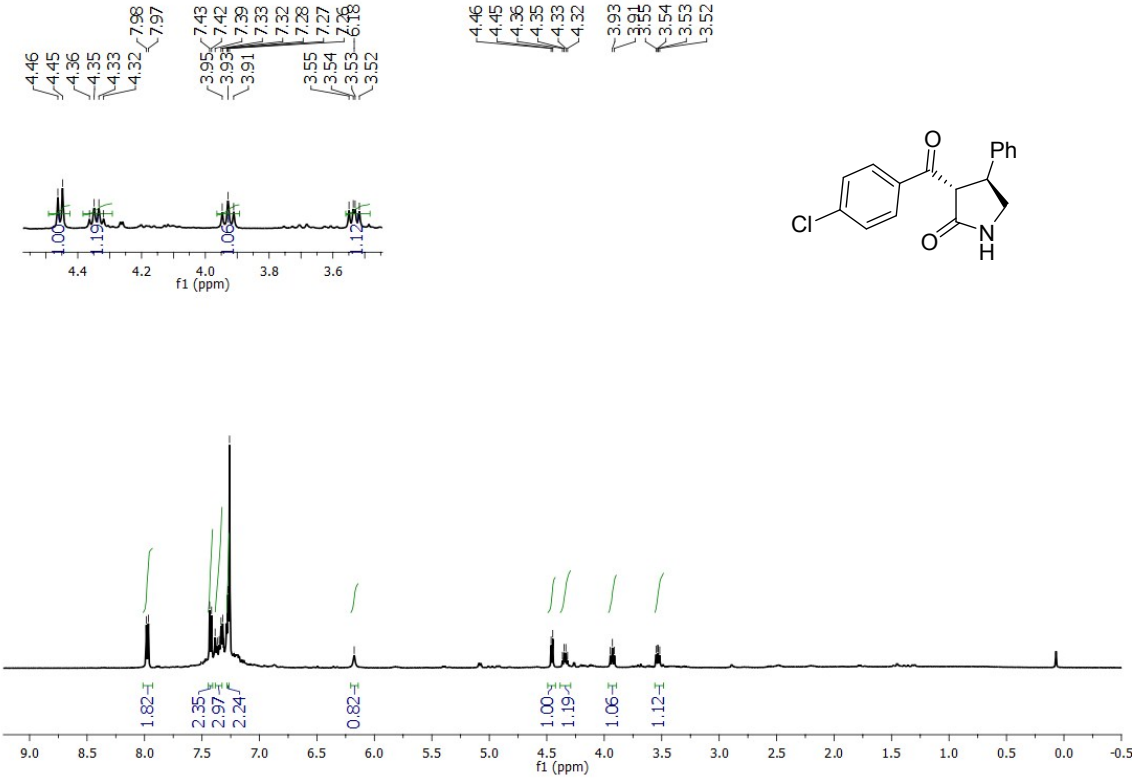
4m ^{1}H-NMR (500 MHz, $CDCl_3$)



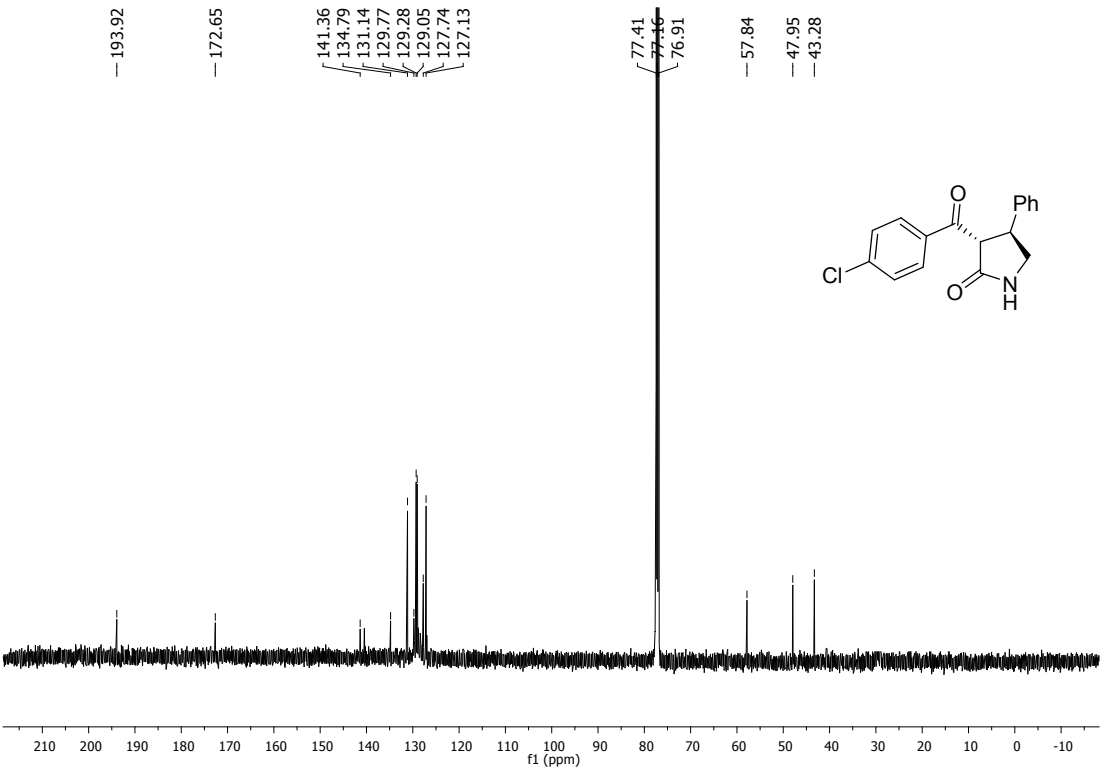
4m ^{13}C-NMR (125 MHz, $CDCl_3$)



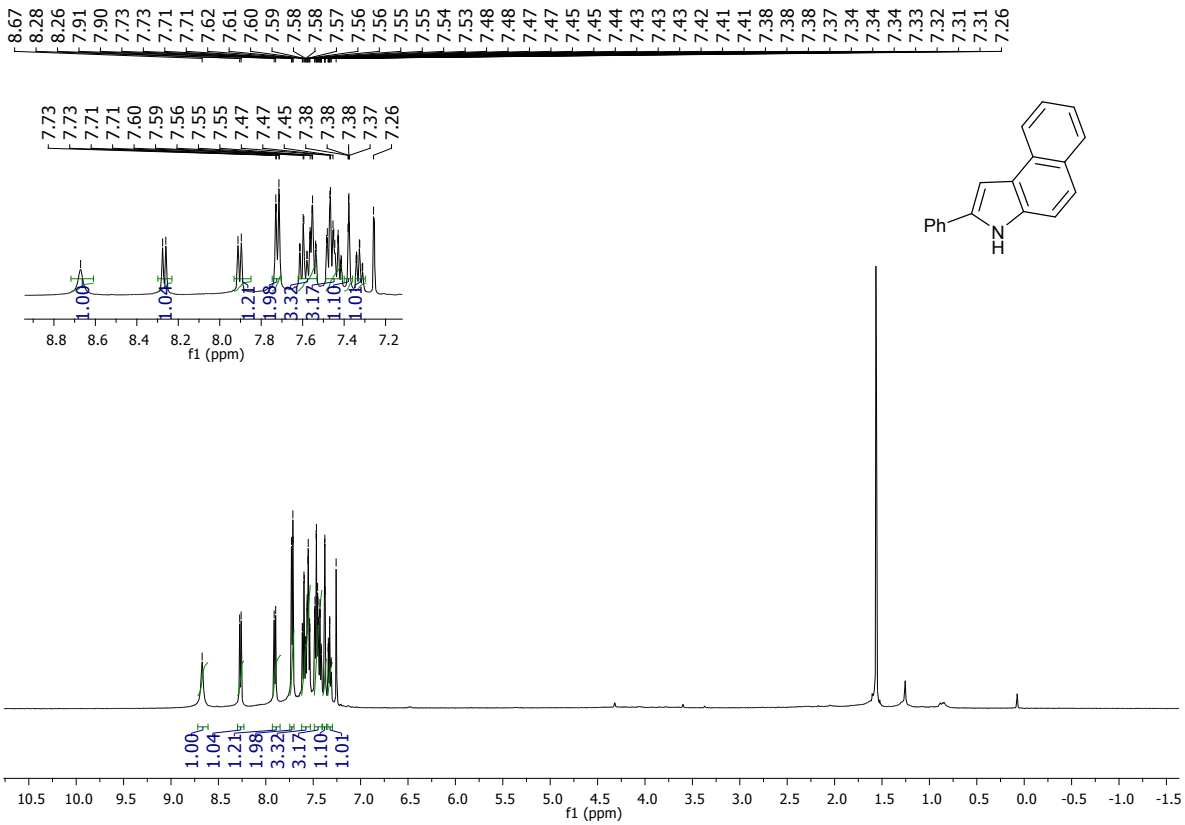
4n ^{1}H-NMR (500 MHz, $CDCl_3$)



4n ^{13}C-NMR (125 MHz, $CDCl_3$)



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



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