Electronic Supporting Information (ESI)

Metal-free DBU promoted regioselective synthesis of isoxazoles and isoxazolines†

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S1. EXPERIMENTAL PROCEDURES

S1.a. General Information

All chemicals were obtained from Sigma-Aldrich Company and used as received. ^{1}H, ^{13}C and DEPT NMR spectra were recorded on Brucker-Avance DPX FT-NMR 500 and 400 MHz instruments. Chemical data for protons are reported in parts per million (ppm) downfield from tetramethylsilane and are referenced to the residual proton in the NMR solvent ($CDCl_3$-7.26 and CD_3OD- 3.28 ppm). Carbon nuclear magnetic resonance spectra (^{13}C NMR) were recorded at 125 MHz or 100 MHz: chemical data for carbons are reported in parts per million (ppm, δ scale) downfield from tetramethylsilane and are referenced to the carbon resonance of the solvent ($CDCl_3$-77 and CD_3OD-50 ppm). ESI-MS and HR-ESIMS spectra were recorded on Agilent 1100 LC-Q-TOF and HR-ESIMS-6540-UHD machines. IR spectra were recorded on Perkin-Elmer IR spectrophotometer. Melting points were recorded on digital melting point apparatus.

S1.b. General procedure for preparation of aldooximes

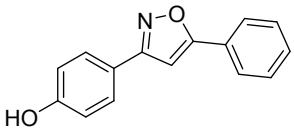
To the solution of hydroxylamine hydrochloride (1 g, 5.88 m mol) in water (5 ml) was added sodium hydroxide (307 mg, 0.5 m mol) and respective aldehydes (1 mmol) and reaction mixture was stirred for 2-3 hrs at room temperature. After completion of the reaction (monitoring by TLC), if compound precipitated out, was filtered on Buckner funnel or extracted with EtOAc (3 × 15 ml). The obtained product was taken for next step without further purification (> 90% yield).

S1.c. Optimized procedure for preparation of isoxazole and isoxazolines.

To the stirred solution of aldoximes (100 mg, 1 mmol) in DMF (3 ml) was added N-chlorosuccinimide (1.2 mmol) at room temperature and reaction was stirred for 0.5-1 h. Then, DBU (1 mmol) and alkynes/ alkenes (1.2 mmol) were added and reaction was further stirred for 1-8 h. After completion of the reaction (confirmed by TLC), chilled water (20 ml) was added and product was extracted with EtOAc (3 × 10 ml). The organic layer was collected, dried on anhydrous sodium sulphate and solvent was evaporated on rotary evaporator to get the crude product. The crude product was purified by silica gel (#100-200) column chromatography using 2 to 20% EtOAc: hexane to get pure isoxazole/ isoxazoline products. For synthesis of chloro-substituted isoxazoles/ isoxazolines, similar procedure was used with the use of 2.2 mmol of NCS instead of 1.2 mmol.

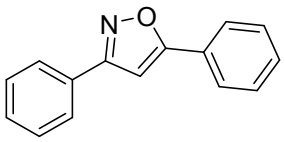
S2. Spectral data of isoxazole and isoxazolines

S2.1. 4-(5-phenylisoxazol-3-yl) phenol (4a):[1]



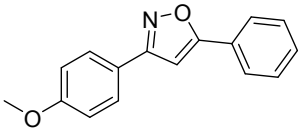
White solid; yield: 80%; m. p. 187-189 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.83 (dd, J = 4, 8 Hz, 2H), 7.76 (d, J = 8 Hz, 2H), 7.47 (m, 3H), 6.95 (d, J = 8 Hz, 2H), 6.78 (s, 1H); ^{13}C NMR ($CDCl_3$ +CD_3OD, 125 MHz): δ 170.0, 162.9, 158.8, 130.2, 128.9, 128.2, 127.2, 125.7, 120.0, 115.7, 97.3; IR ($CHCl_3$): ν_{max} 3433, 2925, 1631, 1450, 1353, 1095, 1017 cm^{-1}; ESI-MS: *m/z* 238.08 $[M+H]^+$; HR-ESIMS: *m/z* 238.0864 calcd for $C_{15}H_{11}NO_2+H^+$ (238.0864).

S2.2. 3, 5-diphenylisoxazole (4b):[2]



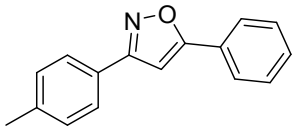
White solid; yield: 75%; m. p. 140-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.86 (m, 4H), 7.49 (m, 6H), 6.84 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 168.7, 161.3, 128.6, 128.4, 127.4, 127.3, 127.3, 125.8, 125.1, 124.2, 95.8; IR ($CHCl_3$): ν_{max} 3437, 3114, 2924, 1462, 1257, 1092, 763 cm^{-1}; ESI-MS: *m/z* 222.08 $[M+H]^+$; HR-ESIMS: *m/z* 222.0917 calcd for $C_{15}H_{11}NO+H^+$ (222.0913).

S2.3. 3-(4-Methoxyphenyl)-5-phenylisoxazole (4c):[1]



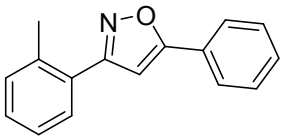
White solid; yield: 72%; m. p. 114-117 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.80-7.85 (m, 4H), 7.43-7.51 (m, 3H), 6.99 (d, J = 8 Hz, 2H), 6.78 (s, 1H), 3.87 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.1, 162.6, 161.0, 130.1, 129.0, 128.2, 127.5, 125.8, 121.6, 114.3, 97.2, 55.3; IR ($CHCl_3$): ν_{max} 3433, 2925, 1631, 1450, 1353, 1095, 1017 cm^{-1}; IR ($CHCl_3$): ν_{max} 3436, 2922, 2851, 1613, 1450, 1019, 841 cm^{-1}; ESI-MS: *m/z* 252.09 $[M+H]^+$; HR-ESIMS: *m/z* 252.1021 calcd for $C_{16}H_{13}NO_2+H^+$ (252.1019).

S2.4. 5-Phenyl-3-(p-tolyl)isoxazole (4d):[3]



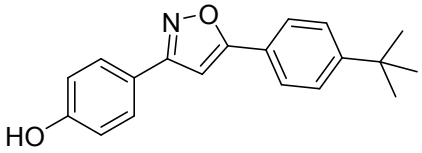
White solid; yield: 88%; m.p. 110-112 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.82-7.84 (m, 2H), 7.76 (d, *J* = 8.0 Hz, 2H), 7.42-7.50 (m, 3H), 7.28 (d, *J* = 8.0 Hz, 2H), 6.80 (s, 1H), 2.41 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.1, 162.6, 161.0, 130.1, 129.0, 128.2, 127.5, 125.8, 121.6, 114.3, 97.2, 55.3; IR ($CHCl_3$): ν_{max} 3433, 2925, 1631, 1450, 1353, 1095, 1017 cm^{-1}; ESI-MS: *m/z* 236.10 $[M+H]^+$; HR-ESIMS: *m/z* 236.1072 calcd for $C_{16}H_{13}NO+H^+$ (236.1070).

S2.5. 5-Phenyl-3-(o-tolyl)isoxazole (4e):[4]



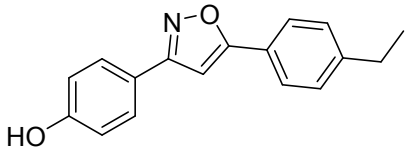
White liquid; yield: 87%; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): *δ* 7.83 (d, *J* = 8.0 Hz, 1H), 7.66-7.68 (m, 2H), 7.39-7.46 (m, 4H), 7.30 (t, *J* = 8.0 Hz, 2H), 6.06 (s, 1H), 2.58 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 158.8, 138.9, 134.4, 131.5, 131.5, 130.1, 128.9, 128.3, 126.4, 126.0, 121.0, 113.0, 22.2; IR ($CHCl_3$): ν_{max} 3433, 2925, 1631, 1450, 1353, 1095, 1017 cm^{-1}; ESI-MS: *m/z* 236.10 $[M+H]^+$; HR-ESIMS: *m/z* 236.1072 calcd for $C_{16}H_{13}NO+H^+$ (236.1070).

S2.6. 4-(5-(4-(tert-butyl) phenyl) isoxazol-3yl) phenol (4f)



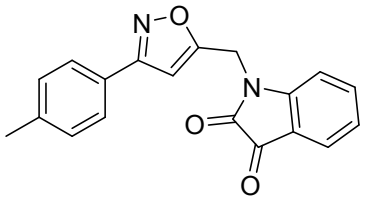
White solid; yield 70%; m. p. 167-170 °C ; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): *δ* 7.74-7.77 (m, 4H), 7.50 (d, *J* = 12.0 Hz, 2H), 6.95 (d, *J* = 8.0 Hz, 2H), 6.73(s, 1H), 1.27(s, 9H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.3, 162.6, 157.4, 153.6, 128.4, 125.9, 125.6, 124.7, 121.5, 115.9, 96.8, 34.4, 31.20 IR ($CHCl_3$): ν_{max} 3351, 2963, 2868, 1614, 1435, 1269, 840 cm^{-1}; ESI-MS: *m/z* 294.14 $[M+H]^+$; HR-ESIMS: *m/z* 294.1487 calcd for $C_{19}H_{19}NO_2+H^+$ (294.1489).

S2.7. 4-(5-(4-Ethylphenyl) isoxazole-3-yl) phenol (4g)



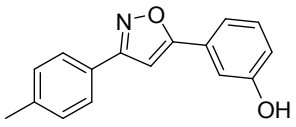
White solid; yield: 70%; m. p. 172-174 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.77-7.78 (m, 4H), 7.32 (d, J = 8.0 Hz, 2H), 6.94 (d, J = 8.0 Hz, 2H), 6.73 (s, 1H), 2.71(q, J = 8.0 Hz, 2H), 1.28 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.4, 162.6, 157.4, 146.8, 128.5, 128.4, 125.9, 124.9, 121.6, 115.9, 96.8, 28.8, 15.30; IR ($CHCl_3$): ν_{max} 3343, 2962, 2924, 1613, 1439, 1018, 838 cm^{-1}; ESI-MS: *m/z* 266.11 $[M+H]^+$; HR-ESIMS: *m/z* 266.1183 calcd for $C_{17}H_{15}NO_2+H^+$ (266.1176).

S2.8. 1-((3-(p-Tolyl) isoxazol-5-yl) methyl) indoline-2, 3-dione (4h)



Orange solid; yield: 50%; m. p. 167-169 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.62-7.66 (m, 4H), 7.24 (d, J = 8.0 Hz, 2H), 7.17 (t, J = 8.0 Hz, 1H), 7.09 (d, J = 8.0 Hz, 1H), 6.58 (s, 1H), 5.07 (s, 2H), 2.39 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 182.2, 165.7, 162.9, 157.7, 149.7, 140.6, 138.7, 129.7, 126.7, 125.7, 125.4, 124.4, 117.7, 110.7, 101.6, 35.5, 21.4 IR ($CHCl_3$): ν_{max} 3436, 2921, 1736, 1611, 1470, 1020 cm^{-1}; ESI-MS: *m/z* 319.11 $[M+H]^+$.

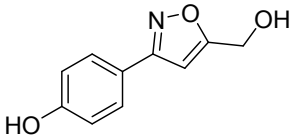
S2.9. 3-(3-(p-Tolyl)isoxazole-5-yl)phenol (4i)



White solid; yield: 80%; m. p. 215-218 °C ; ^{1}H NMR (400 MHz, CD_3OD, ppm): δ 7.67 (d, J = 8.0 Hz, 2H), 7.21-7.25 (m, 5H), 7.01 (s, 1H), 6.81 (m, 1H), 2.31 (s, 3H); ^{13}C NMR ($CDCl_3$+CD_3OD, 100 MHz): δ 169.7, 162.3, 156.7, 139.6, 129.4, 128.9, 127.6, 125.9, 125.1, 116.8, 116.5, 111.7, 96.9, 20.5; IR ($CHCl_3$): ν_{max} 3439, 3294, 2923, 2853, 1565, 1420, 1185, 686

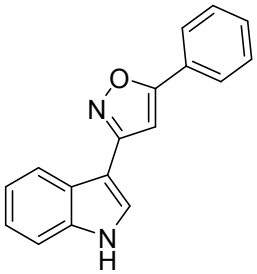
cm^{-1}; ESI-MS: *m/z* 252.09 $[M+H]^+$; HR-ESIMS: *m/z* 252.1017 calcd for $C_{16}H_{13}NO_2+H^+$ (252.1019).

S2.10. 4-(5-(Hydroxymethyl) isoxazole-3-yl) phenol (4j):[5]



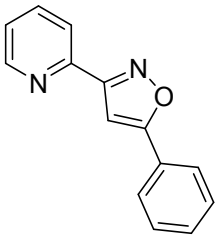
White solid; yield: 65%; m. p. 137-139 °C; ^{1}H NMR (400 MHz, CD_3OD, ppm): δ 7.56 (d, J = 8.0 Hz, 2H), 6.77 (d, J = 8.0 Hz, 2H), 6.50 (s, 1H), 4.59 (s, 2H); ^{13}C NMR ($CDCl_3$+CD_3OD, 100 MHz): δ 179.2, 172.3, 162.2, 128.2, 120.0, 115.7, 99.5, 55.8; IR ($CHCl_3$): ν_{max} 3435, 2922, 2851, 1705, 1612, 1427, 1019 cm^{-1}; ESI-MS: *m/z* 192.06 $[M+H]^+$; HR-ESIMS: *m/z* 192.0656 calcd for $C_{10}H_9NO_3+H^+$ (192.0655).

S2.11. 3-(1H-Indol-3-yl)-5-phenylisoxazole (4k)



White solid; yield: 60%; m. p. 194-197 °C ; 1H NMR (400 MHz, CD_3OD, ppm): δ 8.02-8.04 (m, 1H), 7.77-7.83 (m, 4H), 7.35-7.46 (m, 4H), 7.06-7.15(m, 3H); ^{13}C NMR ($CDCl_3$+CD_3OD, 100 MHz): δ 169.0, 159.1, 136.5, 130.0, 128.9, 127.9, 125.9, 125.0, 124.9, 122.7, 121.0, 120.9, 111.5, 106.5, 97.7; IR ($CHCl_3$): ν_{max} 3301, 2922, 1595, 1580, 1441, 1106, 1019, 740 cm^{-1}; ESI-MS: *m/z* 261.09 $[M+H]^+$; HR-ESIMS: *m/z* 261.1033 calcd for $C_{17}H_{12}N_2O+H^+$ (261.1022).

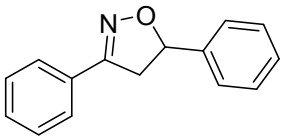
S2.12. 5-Phenyl-3-(pyridine-2-yl)isoxazole (4l):[6]



White solid; yield: 78%; m. p. 79-81°C ; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 8.72 (d, J = 8Hz, 1H), 8.14 (d, J = 8Hz, 1H), 7.80-7.88 (m, 3H), 7.46-7.52 (m, 3H), 7.38 (dd, J = 4, 8Hz, 1H), 7.21

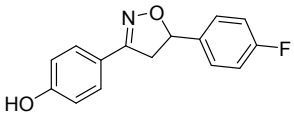
(s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.6, 163.7, 149.7, 148.5, 136.9, 130.2, 129.0, 127.4, 125.8, 124.5, 121.6, 98.3; IR ($CHCl_3$): ν_{max} 3436, 2925, 1593, 1567, 1486, 1460, 1401, 764 cm^{-1}; ESI-MS: *m/z* 223.08 $[M+H]^+$.

S2.13. 3, 5-Diphenyl-4, 5-dihydroisoxazole (6a):[3]



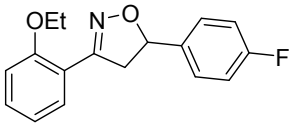
White solid; yield: 85%; m. p. 72-74 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): *δ* 7.70 (dd, *J* = 4.0, 8.0 Hz, 2H), 7.30- 7.42 (m, 8H), 5.75 (dd, *J* = 11.0, 8.3 Hz, 1H), 3.79(dd, *J* = 16.6, 11.0 Hz, 1H), 3.35 (dd, *J* = 16.6, 8.3 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 156.1, 140.9, 130.2, 129.4, 128.8, 128.2, 126.7, 125.9, 82.6, 43.2; IR ($CHCl_3$): ν_{max} 3433, 2920, 1730, 1446, 1352, 1120, 893, 751, 686 cm^{-1}; ESI-MS: *m/z* 224.10 $[M+H]^+$; HR-ESIMS: *m/z* 224.1084 calcd for $C_{15}H_{13}NO+H^+$ (224.1070).

S2.14. 4-(5-(4-Fluorophenyl)-4, 5-dihydroisoxazol-3-yl)phenol (6b)



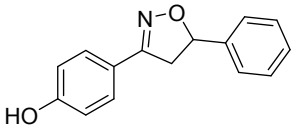
White solid; yield: 70%; m. p. 124 – 126 °C ; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): *δ* 7.54 (d, *J* = 8.0 Hz, 2H), 7.36 (dd, *J* = 4, 8.0 Hz, 2H), 7.06 (t, *J* = 8.0 Hz, 2H), 6.86 (d, *J* = 8.0 Hz, 2H), 5.67(dd, *J* = 10.4, 8.6 Hz, 1H), 3.75 (dd, *J* = 16.6, 10.8 Hz, 1H), 3.28 (dd, *J* = 16.6, 8.2 Hz, 1H); ^{13}C NMR ($CDCl_3$+CD_3OD, 125 MHz): δ 162.5 (d, $^1J_{CF}$ = 245 Hz), 159.0, 156.4, 136.6, 136.5, 128.4, 127.7, 127.6, 120.3, 115.6 (d, $^2J_{CF}$ = 21.2 Hz), 115.5, 81.5, 43.4; IR ($CHCl_3$): ν_{max} 3405, 2921, 2852, 1538, 1493, 1272, 1017 cm^{-1}; ESI-MS: *m/z* 258.11 $[M+H]^+$; HR-ESIMS: *m/z* 258.0955 calcd for $C_{15}H_{12}FNO_2+H^+$ (258.0925).

S2.15. 3-(2-Ethoxyphenyl)-5-(4-fluorophenyl)-4,5-dihydroisoxazole (6c)



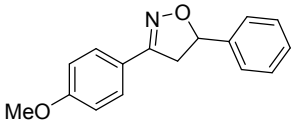
Colorless liquid; yield: 71%; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.79 (d, J = 8.0 Hz, 1H), 7.34- 7.40 (m, 3H), 7.06 (t, J = 8.0 Hz, 2H), 6.98 (t, J = 8.0 Hz, 1H), 6.91(d, J = 8.0 Hz, 1H), 5.66 (t, J = 9.3 Hz, 1H), 4.06 (q, J = 6.9 Hz, 2H), 3.90 (dd, J = 17.4, 10.8 Hz, 1H), 3.48 (dd, J = 17.4, 8.1 Hz, 1H), 1.39 (t, J = 6.9 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 162.5 (d, $^1J_{CF}$ = 245 Hz), 156.9, 156.0, 137.1, 131.4, 129.4, 127.8, 127.7, 120.7, 118.6, 115.5 (d, $^2J_{CF}$ = 21.2 Hz), 112.1, 81.9, 63.9, 45.8, 14.7; IR ($CHCl_3$): ν_{max} 3440, 2979, 2925, 1600, 1510, 1497, 1452, 1245, 1123, 1040 cm^{-1}; ESI-MS: *m/z* 286.12 $[M+H]^+$; HR-ESIMS: *m/z* 286.1260 calcd for $C_{17}H_{16}FNO_2+H^+$ (286.1238).

S2.16. 4-(5-phenyl-4, 5-dihydroisoxazole-3-yl) phenol (6d):[7]



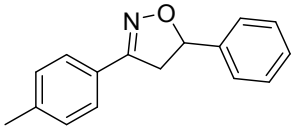
White solid; yield: 72%; m. p. 140-142 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.59 (d, J = 8.0 Hz, 2H), 7.31- 7.41 (m, 5H), 6.86 (d, J = 8.0 Hz, 2H), 5.71 (dd, J = 10.9, 8.3 Hz, 1H), 3.75 (dd, J = 16.5, 10.9 Hz, 1H), 3.31 (dd, J = 16.6, 8.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 156.9, 155.1, 139.7, 127.7, 127.5, 127.2, 124.8, 114.8, 81.2, 42.4; IR ($CHCl_3$): ν_{max} 3355, 2924, 2854, 1610, 1455, 1354, 1019, 836 cm^{-1}; ESI-MS: *m/z* 240.11 $[M+H]^+$.

S2.17. 3-(4-Methoxyphenyl)-5-phenyl-4, 5-dihydroisoxazole (6e):[2]



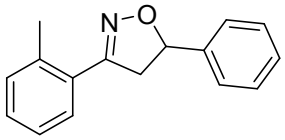
White solid; yield: 70%; m. p. 95- 97 °C ; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.63 (d, J = 12.0 Hz, 2H), 7.33- 7.39 (m, 5H), 6.92 (d, J = 12.0 Hz, 2H), 5.71 (dd, J = 10.9, 8.3 Hz, 1H), 3.84 (s, 3H), 3.76(dd, J = 16.5, 10.9 Hz, 1H), 3.32 (dd, J = 16.5, 8.2 Hz, 1H); IR ($CHCl_3$): ν_{max} 3435, 2954, 2919, 2836, 1609, 1457, 1236, 1178, 1020, 829, 695 cm^{-1}; ESI-MS: *m/z* 254.11 $[M+H]^+$; HR-ESIMS: *m/z* 254.1176 calcd for $C_{16}H_{15}NO_2+H^+$ (254.1176).

S2.18. 5-Phenyl-3-(p-tolyl)-4,5-dihydroisoxazole (6f):[3]



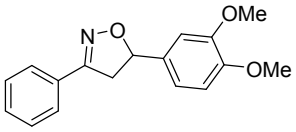
White solid; yield: 82%; m. p. 94- 96 °C ; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.58 (d, J = 8.0, 2H), 7.31-7.41 (m, 5H), 7.21 (d, J = 8.0, 2H), 5.72 (dd, J = 10.9, 8.3 Hz, 1H), 3.76 (dd, J = 16.4, 8.3 Hz, 1H), 3.30 (dd, J = 16.6, 8.2 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 156.1, 141.0, 140.4, 129.4, 128.7, 128.2, 126.7, 125.9, 82.4, 43.3, 21.4; IR ($CHCl_3$): ν_{max} 3439, 2923, 2853, 1456, 1430, 1238, 823 cm^{-1}; ESI-MS: *m/z* 238.12 $[M+H]^+$; HR-ESIMS: *m/z* 238.1228 calcd for $C_{16}H_{15}NO+H^+$ (238.1226).

S2.19. 5-phenyl-3-(*o*-tolyl)-4, 5-dihydroisoxazole (6g)



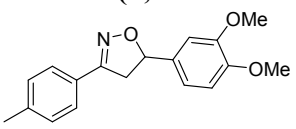
Colorless liquid; yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.21-7.42 (m, 9H), 5.69 (dd, J = 10.7, 8.1 Hz, 1H), 3.83 (dd, J = 16.6, 10.8 Hz, 1H), 3.39 (dd, J = 16.6, 8.0 Hz, 1H), 2.60 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 157.0, 141.1, 138.1, 131.6, 129.4, 128.8, 128.8, 128.5, 128.1, 125.8., 125.9, 82.4, 43.3, 21.4; IR ($CHCl_3$): ν_{max} 3435, 2923, 1587, 1494, 1454, 894 cm^{-1}; ESI-MS: *m/z* 238.12 $[M+H]^+$; HR-ESIMS: *m/z* 238.1235 calcd for $C_{16}H_{15}NO+H^+$ (238.1226).

S2.20. 5-(3, 4-Dimethoxyphenyl)-3-phenyl-4, 5-dihydroisoxazole (6h)



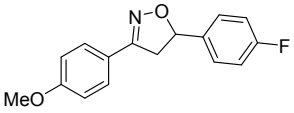
White solid; yield: 78%; m. p. 89-91 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.69-7.72 (m, 2H), 7.41-7.43 (m, 3H), 6.93-6.94 (m, 2H), 6.86 (d, J = 8Hz, 1H), 5.70 (dd, J = 10.7, 8.9 Hz, 1H), 3.89 (s, 6H), 3.75 (dd, J = 16.7, 10.9 Hz, 1H), 3.35 (dd, J = 16.7, 8.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 156.4, 149.2, 149.0, 133.1, 130.1, 129.5, 128.7, 118.5, 111.0, 108.9, 82.7, 55.9, 55.9, 43.0; IR ($CHCl_3$): ν_{max} 3435, 2924, 2851, 1593, 1516, 1261, 1025, cm^{-1}; ESI-MS: *m/z* 284.12 $[M+H]^+$; HR-ESIMS: *m/z* 284.1284 calcd for $C_{17}H_{17}NO_3+H^+$ (284.1281).

S2.21. 5-(3, 4-Dimethoxyphenyl)-3-(p-tolyl)-4, 5-dihydroisoxazole (6i)



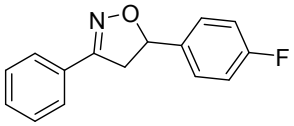
White solid; yield: 80%; m. p. 84-87 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.59 (d, J = 8 Hz, 2H), 7.22 (d, J = 8 Hz, 2H), 6.93 (d, J = 8Hz, 2H), 6.85 (d, J = 12 Hz, 1H), 5.67 (m, 1H), 3.88 (s, 6H), 3.72 (dd, J = 16.6, 10.8 Hz, 1H), 3.32 (dd, J = 16.6, 8.8 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 156.3, 149.3, 140.3, 133.3, 129.4, 126.7, 126.6, 118.4, 111.1, 109.0, 82.5, 55.9, 55.9, 43.1, 21.4; IR ($CHCl_3$): ν_{max} 3439, 3294, 2924, 2836, 1517, 1454, 1261, 1138, 1026, 816 cm^{-1}; ESI-MS: *m/z* 298.14 $[M+H]^+$; HR-ESIMS: *m/z* 298.1440 calcd for $C_{18}H_{19}NO_3+H^+$ (298.1438).

S2.22. 5-(4-fluorophenyl)-3-(4-methoxyphenyl)-4, 5-dihydroisoxazole (6j)



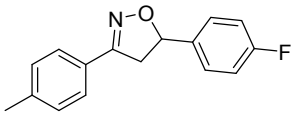
White solid; yield: 70%; m. p. 103-106 °C ; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.61-7.64 (m, 2H), 7.35-7.38 (m, 2H), 7.02-7.08 (m, 2H), 6.91-6.94 (m, 2H), 5.68 (dd, J = 10.8, 8.2 Hz, 1H) 3.84 (s, 3H), 3.74 (dd, J = 16.6, 10.9 Hz, 1H), 3.28 (dd, J = 16.6, 8.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 162.5 (d, $^1J_{CF}$ = 245 Hz), 161.1, 155.7, 136.8, 128.3, 127.7, 127.6, 121.8, 115.6 (d, $^2J_{CF}$ = 23.2 Hz), 114.2, 81.6, 55.3, 43.4; IR $CHCl_3$): ν_{max} 3437, 2923, 2880, 1608, 1515, 1244, 1021, 831cm^{-1}; ESI-MS: *m/z* 272.10 $[M+H]^+$; HR-ESIMS: *m/z* 272.1079 calcd for $C_{16}H_{14}FNO_2+H^+$ (272.1081).

S2.23. 5-(4-Fluorophenyl)-3-phenyl-4,5-dihydroisoxazole (6k):[8]



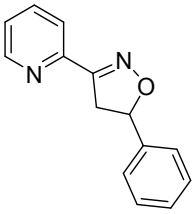
White solid; yield: 82%; m. p. 87-89 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.69 (dd, J = 8.0, 4.0 Hz, 2H), 7.36-7.43 (m, 5H), 7.04-7.09 (m, 2H), 5.73 (dd, J = 11.0, 8.2 Hz, 1H), 3.78 (dd, J = 16.7, 11.0 Hz, 1H), 3.31 (dd, J = 16.7, 8.2 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 162.5 (d, $^1J_{CF}$ = 245 Hz), 156.5, 136.7, 136.7, 130.2, 128.8, 127.7, 127.6, 126.7, 115.6 (d, $^2J_{CF}$= 21.2 Hz), 81.9, 43.2; IR ($CHCl_3$): ν_{max} 3439, 2925, 2854, 1602, 1512, 1446, 1018, 830 cm^{-1}; ESI-MS: *m/z* 242.09 $[M+H]^+$; HR-ESIMS: *m/z* 242.0982 calcd for $C_{15}H_{12}FNO+H^+$ (242.0976).

S2.24. 5-(4-fluorophenyl)-3-(p-tolyl)-4, 5-dihydroisoxazole (6l)



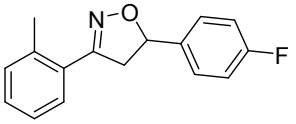
White solid; yield: 85%; m. p. 114-119 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.58 (d, J = 8.0, 2H), 7.35-7.38 (m, 2H), 7.22 (d, J= 8.0, 2H), 7.06-7.08 (m, 2H), 5.70 (dd, J = 10.8, 8.2Hz, 1H), 3.76 (dd, J = 16.6, 10.9 Hz, 1H), 3.29 (dd, J = 16.6, 8.2 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 162.5 (d, $^1J_{CF}$ = 245 Hz), 156.1, 140.5, 136.8, 129.5, 127.7, 127.7, 126.7, 126.5, 115.6 (d, $^2J_{CF}$ = 23.2 Hz), 81.7, 43.3, 21.4; IR ($CHCl_3$): ν_{max} 3439, 2923, 2854, 1515, 1432, 1050, 823 cm^{-1}; ESI-MS: *m/z* 256.11 $[M+H]^+$; HR-ESIMS: *m/z* 256.1134 calcd for $C_{16}H_{14}FNO+H^+$ (256.1132).

S2.25. 5-phenyl-3-(pyridine-2-yl)-4,5-dihydroisoxazole (6m):[9]



Colourless liquid; yield: 80%; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 8.59-8.61 (m, 1H), 8.07 (d, J = 8.0, Hz, 1H), 7.72-7.76 (m, 1H), 7.28-7.42 (m, 6H), 5.80 (dd, J = 10.6, 8.0 Hz, 1H), 3.94 (dd, J = 16.0, 10.7 Hz, 1H), 3.55(dd, J = 16.0, 8.0Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 158.1, 149.3, 140.7, 136.4, 128.7, 128.2, 125.9, 124.3, 121.8, 83.3, 42.5; IR ($CHCl_3$): ν_{max} 3437, 2924, 2853, 1583, 1457, 1019, 772 cm^{-1}; ESI-MS: *m/z* 225.09 $[M+H]^+$.

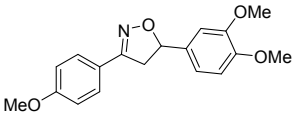
S2.26. 5-(4-Fluorophenyl)-3-(o-tolyl)-4, 5-dihydroisoxazole (6n)



Colorless liquid; yield: 82%; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.33-7.40 (m, 2H), 7.29-7.31 (m, 3H), 7.21-7.25 (m, 1H), 7.07 (t, J = 8.0 Hz, 2H), 5.68 (dd, J = 10.7, 8.1 Hz, 1H), 3.82 (dd, J = 16.6, 10.8 Hz, 1H), 3.34 (dd, J = 16.6, 8.0 Hz, 1H), 2.59 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 162.5 (d, $^1J_{CF}$= 245 Hz), 157.0, 138.1, 136.8, 136.8, 131.6, 129.4, 128.8, 128.4, 127.6, 127.6, 125.8, δ 115.6 (d, $^2J_{CF}$= 22 Hz), 80.9, 45.7, 22.9; IR ($CHCl_3$): ν_{max} 3436, 2923, 1604, 1454,

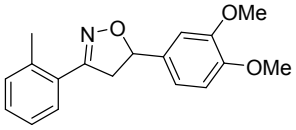
1224, 1019 cm^{-1}; ESI-MS: *m/z* 256.11 $[M+H]^+$; HR-ESIMS: *m/z* 256.1132 calcd for $C_{16}H_{14}FNO+H^+$ (256.1132).

S2.27. 5-(3, 4-Dimethoxyphenyl)-3-(4-methoxyphenyl)isoxazole (6o)



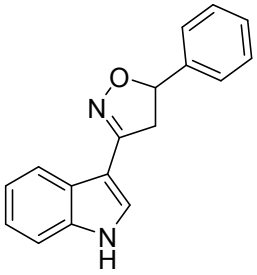
White solid; yield: 72%; m. p. 96-99 °C ; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.63 (d, J = 12.0 Hz, 2H), 6.84-6.94 (m, 4H), 6.83 (d, J = 8.0 Hz, 1H), 5.65(dd, J = 10.6, 8.9 Hz, 1H), 3.88(s, 6H), 3.84(s, 3H), 3.70 (dd, J = 16.6, 10.8 Hz, 1H), 3.31 (dd, J = 16.6, 8.8 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 161.1, 155.9, 149.3, 149.0, 133.4, 128.2, 122.1, 118.4, 114.1, 111.1, 108.9, 82.3, 55.9, 55.9, 55.3, 43.3;IR ($CHCl_3$): ν_{max} 3439, 2929, 2837, 1607, 1516, 1257, 1024, 1024 cm^{-1}; ESI-MS: *m/z* 314.13 $[M+H]^+$; HR-ESIMS: *m/z* 314.1387 calcd for $C_{18}H_{19}NO_4+H^+$ (314.1387).

S2.28. 5-(3, 4-Dimethoxyphenyl)-3-(o-tolyl)-4, 5-dihydroisoxazole (6p)



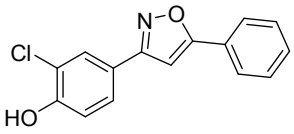
Colorless liquid; yield: 78%; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.23-7.35 (m, 4H), 6.92-6.95 (m, 2H), 6.86 (d, J = 8Hz, 1H), 5.65 (dd, J = 10.6, 8.5 Hz, 1H), 3.89 (s, 3H), 3.88 (s, 3H), 3.79 (dd, J = 16.6, 10.8 Hz, 1H), 3.39 (dd, J = 16.6, 8.4 Hz, 1H), 2.60 (s, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 157.2, 149.2, 148.9, 138.0, 133.3, 131.6, 129.4, 128.8, 128.5, 125.8, 118.3, 111.1, 108.8, 81.5, 55.9, 55.9, 45.6, 23.0; IR ($CHCl_3$): ν_{max} 3348, 2923, 1516, 1261, 1138, 1025, 759 cm^{-1}; ESI-MS: *m/z* 298.14 $[M+H]^+$; HR-ESIMS: *m/z* 298.1453 calcd for $C_{18}H_{19}NO_3+H^+$ (298.1438).

S2.29. 3-(1H-Indol-3yl)-5-phenyl-4, 5-dihydroisoxazole (6q)



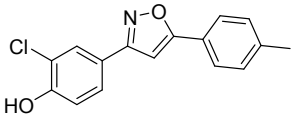
White solid; yield: 63%; m. p. 168-171 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 8.39 (s, 1-NH), 8.30 (d, J = 8.0, Hz, 1H), 7.25-7.45 (m, 9H), 5.67 (dd, J = 10.6, 8.0 Hz, 1H), 3.82 (dd, J = 16.0, 10.7 Hz, 1H), 3.38 (dd, J = 16.0, 8.0Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 152.4, 141.3, 136.5, 128.7, 128.0, 125.9, 125.8, 124.6, 123.6, 122.6, 121.5, 111.2, 107.7, 80.8, 44.6; IR ($CHCl_3$): ν_{max} 3414, 2922, 1595, 1433, 1019, 743 cm^{-1}; ESI-MS: *m/z* 263.11 $[M+H]^+$; HR-ESIMS: *m/z* 263.1184 calcd for $C_{17}H_{14}N_2O+H^+$ (263.1179).

S2.30. 2-Chloro-4-(5-phenylisoxazole-3-yl)phenol (7a)



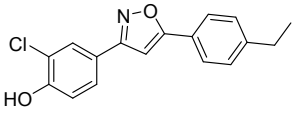
White solid; yield: 88%; m.p. 193-196 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.89 (d, J = 4.1, Hz, 1H), 7.82-7.84 (m, 2H), 7.68-7.71 (m, 1H), 7.47-7.50 (m, 3H), 7.13 (d, J = 8 Hz, 1H), 6.77(s, 1H), 5.78 (s, 1H); ^{13}C NMR ($CDCl_3$+CD_3OD, 125 MHz): δ 170.3, 161.9, 154.4, 130.3, 128.9, 128.3, 127.1, 126.3, 125.7, 121.0, 116.7, 97.2; IR ($CHCl_3$): ν_{max} 3346, 2922, 2851, 1594, 1422, 1019 cm^{-1}; ESI-MS: *m/z* 272.04 $[M+H]^+$; HR-ESIMS: *m/z* 272.0481 calcd for $C_{15}H_{10}ClNO_2+H^+$ (272.0473).

S2.31. 2-Chloro-4-(5-(p-tolyl)isoxazole-3-yl)phenol (7b)



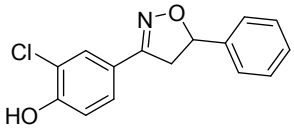
White solid; yield: 86 %; m. p. 184-186 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.88 (d, J = 4.1, Hz, 1H), 7.73 (s, 1H), 7.67-7.71 (m, 2H), 7.29 (d, J = 8.0, Hz, 2H), 7.12 (d, J = 8.0, Hz, 1H), 6.71(s, 1H), 5.78 (s, 1H), 2.42(s, 3H); ^{13}C NMR ($CDCl_3$+CD_3OD, 125 MHz): δ 170.5, 161.8, 154.3, 140.7, 129.6, 126.3, 125.6, 124.3, 121.3, 121.0, 116.7, 96.6, 21.32; IR ($CHCl_3$): ν_{max} 3128, 2922, 2851, 1602, 1425, 1300, 946, 779 cm^{-1}; ESI-MS: *m/z* 286.06 $[M+H]^+$; HR-ESIMS: *m/z* 286.0630 calcd for $C_{16}H_{12}ClNO_2+H^+$ (286.0629).

S2.32. 2-Chloro-4-(5-(4-ethylphenyl) isoxazole-3-yl) phenol (7c)



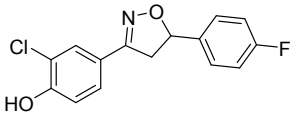
White solid; yield: 87%; m. p. 128-130 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.87 (d, J = 2.0 Hz, 1H), 7.74 (d, J = 8.2 Hz, 2H), 7.68 (dd, J = 8.5, 2.1 Hz, 1H), 7.31 (d, J = 8.2 Hz, 2H), 7.12 (d, J = 8.5 Hz, 1H), 6.71(s, 1H), 5.88 (s, OH), 2.71(q, J = 7.6 Hz, 2H), 1.28 (t, J = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.7, 161.5, 152.9, 146.9, 128.5, 127.6, 127.0, 125.9, 124.8, 122.7, 120.6, 116.7, 96.6, 28.8, 15.3; IR ($CHCl_3$): ν_{max} 3136, 2925, 2851, 1613, 1430, 1301, 1220, 1058 cm^{-1}; ESI-MS: *m/z* 300.07 $[M+H]^+$; HR-ESIMS: *m/z* 300.0793 calcd for $C_{17}H_{14}ClNO_2+H^+$ (300.0786).

S2.33. 2-Chloro-4-(5-phenyl-4,5-dihydroisoxazol-3-yl)phenol (7d)



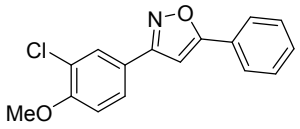
White solid; yield: 90%; m. p. 140 – 142 °C ; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.68 (d, J = 1.9, Hz, 1H), 7.50(dd, J = 8.5, 1.9.0 Hz, 1H), 7.39-7.30 (m, 5H), 7.04 (d, J = 8.5 Hz, 1H), 6.22(s, OH), 5.73 (dd, J = 10.9, 8.3 Hz, 1H), 3.72(dd, J = 16.6, 11.0 Hz, 1H), 3.29 (dd, J = 16.6, 8.2 Hz, 1H); ^{13}C NMR ($CDCl_3$+CD_3OD, 125 MHz): δ 155.5, 154.5, 140.5, 128.7, 128.4, 128.2, 126.4, 125.7, 121.6, 120.9, 116.6, 82.4, 43.1; IR ($CHCl_3$): ν_{max} 3344, 2919, 1603, 1510, 1338, 1292, 1206, 1057 cm^{-1}; ESI-MS: *m/z* 274.04 $[M+H]^+$; HR-ESIMS: *m/z* 274.0639 calcd for $C_{15}H_{12}ClNO_2+H^+$ (274.0629).

S2.34. 2-Chloro-4-(5-(4-fluorophenyl)-4,5-dihydroisoxazole-3yl)phenol (7e)



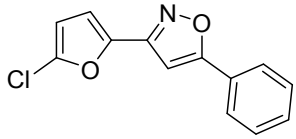
White solid; yield: 92%; m. p. 180 – 182 °C ; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.69 (d, J = 1.9 Hz, 1H), 7.51 (dd, J = 8.5, 1.9 Hz, 1H), 7.35 (dd, J = 8.6, 5.3 Hz, 2H), 7.06 (t, J = 8.6, 3H) 5.80 (s, OH), 5.71 (dd, J = 10.8, 8.3 Hz, 1H), 3.72 (dd, J = 16.6, 10.9 Hz, 1H), 3.25 (dd, J = 16.6, 8.2 Hz, 1H); ^{13}C NMR ($CDCl_3$+CD_3OD, 125 MHz): δ 161.5, 155.4, 154.6, 136.3, 128.4, 127.6, 127.5, 121.5, 120.9, 116.6, 115.6, 115.4, 81.7, 43.1; IR ($CHCl_3$): ν_{max} 3364, 2923, 2852, 1606, 1512, 1355, 1225, 834 cm^{-1}; ESI-MS: *m/z* 292.05 $[M+H]^+$; HR-ESIMS: *m/z* 292.0563 calcd for $C_{15}H_{11}ClFNO_2+H^+$ (292.0545).

S2.35. 3-(3-Chloro-4-methoxyphenyl)-5-phenylisoxazole (7f)



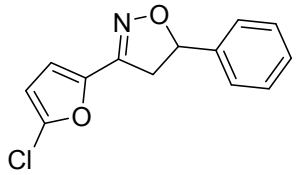
White solid; yield: 80%; m. p. 120-122 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 8.06 (d, J = 8.6 Hz, 2H), 7.86 (d, J = 8.6 Hz, 2H), 7.53 (m, 3H), 7.04(d, J = 8.6 Hz, 2H), 3.88(s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 163.9, 161.1, 160.3, 130.5, 129.6, 128.8, 128.7, 127.4, 126.6, 126.5,119.8 114.2, 104.4, 55.3; IR ($CHCl_3$): ν_{max} 3344, 2919, 1603, 1510, 1338, 1292, 1206, 1057 cm^{-1}; ESI-MS: *m/z* 286.06 $[M+H]^+$, HR-ESIMS: *m/z* 286.0632 calcd for $C_{16}H_{12}ClNO_2+H^+$ (286.0629).

S2.36. 3-(5-Chlorofuran-2-yl)-5-phenylisoxazole (7g)



White solid; yield: 90%; m. p. 115-117 °C ; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.82 (dd, J = 7.6,1.7 Hz, 2H), 7.56-7.44 (m, 3H), 6.95 (d, J = 3.5 Hz, 1H), 6.77(s, 1H), 6.33(d, J = 3.4, Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.4, 154.6, 143.9, 138.3, 130.4, 129.0, 125.9, 112.04, 108.5, 96.7; IR ($CHCl_3$): ν_{max} 3437, 3126, 2921, 1521, 1418, 1018, 759 cm^{-1}; ESI-MS: *m/z* 246.02 $[M+H]^+$; HR-ESIMS: *m/z* 246.0314 calcd for $C_{13}H_8ClNO_2+H^+$ (246.0316).

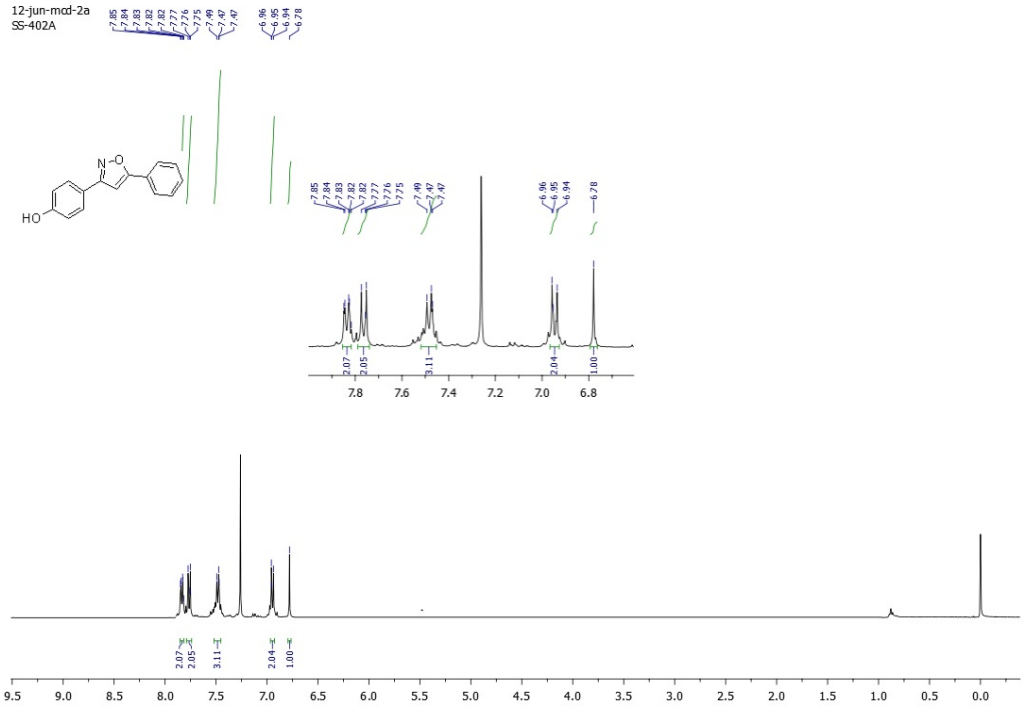
S2.37. 3-(5-chlorofuran-2-yl)-5-phenyl-4, 5-dihydroisoxazole (7h)

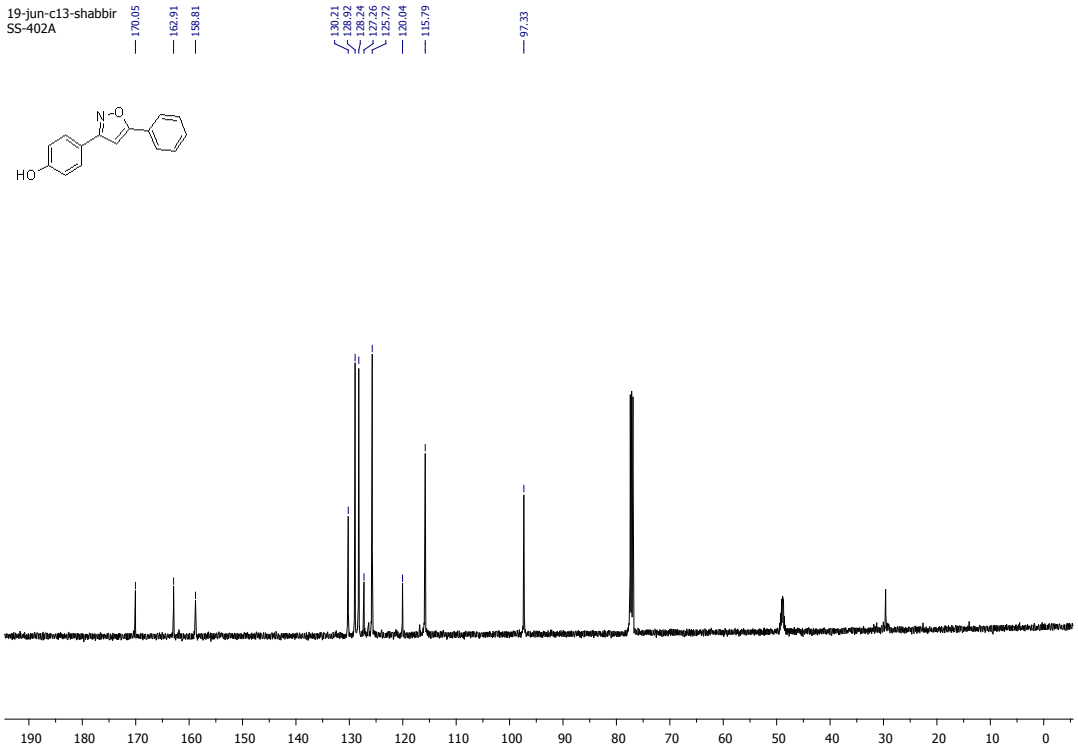


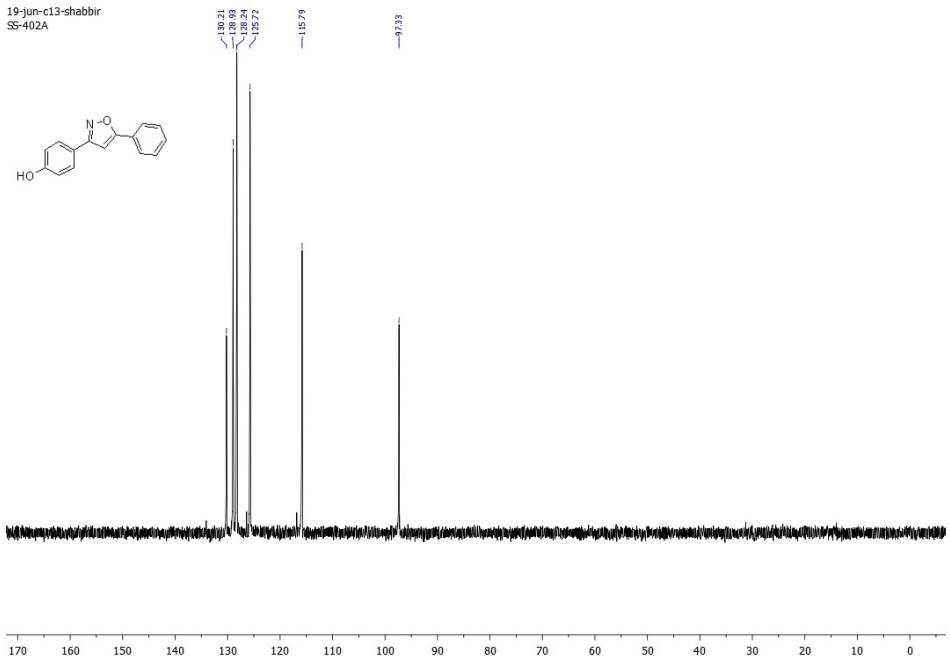
White solid; yield: 60%; m. p. 99- 101 °C; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 7.30-7.40 (m, 5H), 6.72 (d, J = 3.5 Hz, 1H), 6.27 (d, J = 3.5 Hz, 1H) 5.71(dd, J = 10.9, 8.2 Hz, 1H), 3.71(dd, J = 16.7, 11.0 Hz, 1H), 3.27(dd, J = 16.7, 8.2 Hz, 1H), ^{13}C NMR ($CDCl_3$, 125 MHz): δ 147.7, 144.2, 140.2, 138.9, 128.8, 128.4, 125.8, 113.7, 108.6, 82.4, 42.6; IR ($CHCl_3$): ν_{max} 3436, 2922, 1618, 1433, 1017cm^{-1}; ESI-MS: *m/z* 248.04 $[M+H]^+$; HR-ESIMS: *m/z* 248.0480 calcd for $C_{13}H_{10}ClNO_2+H^+$ (248.0473).

S3. Scanned copies of ^{1}H, ^{13}C and DEPT135 NMR spectra's of isoxazoles/ isoxazolines

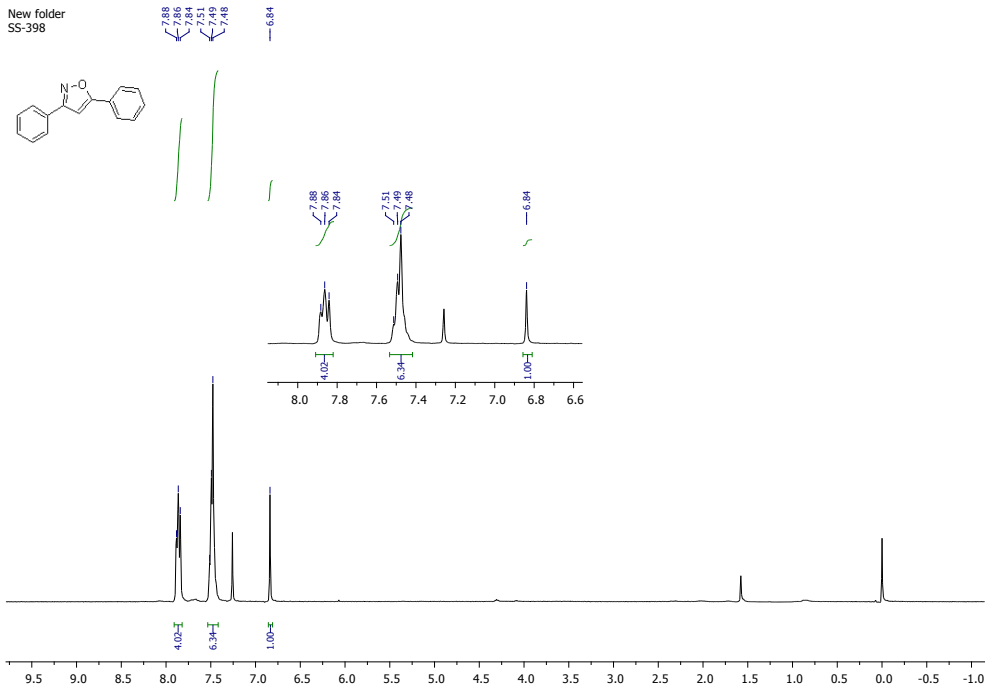
S3.1. ^{1}H, ^{13}C and DEPT135 NMR spectra of 4-(5-phenylisoxazol-3-yl) phenol (**4a**)

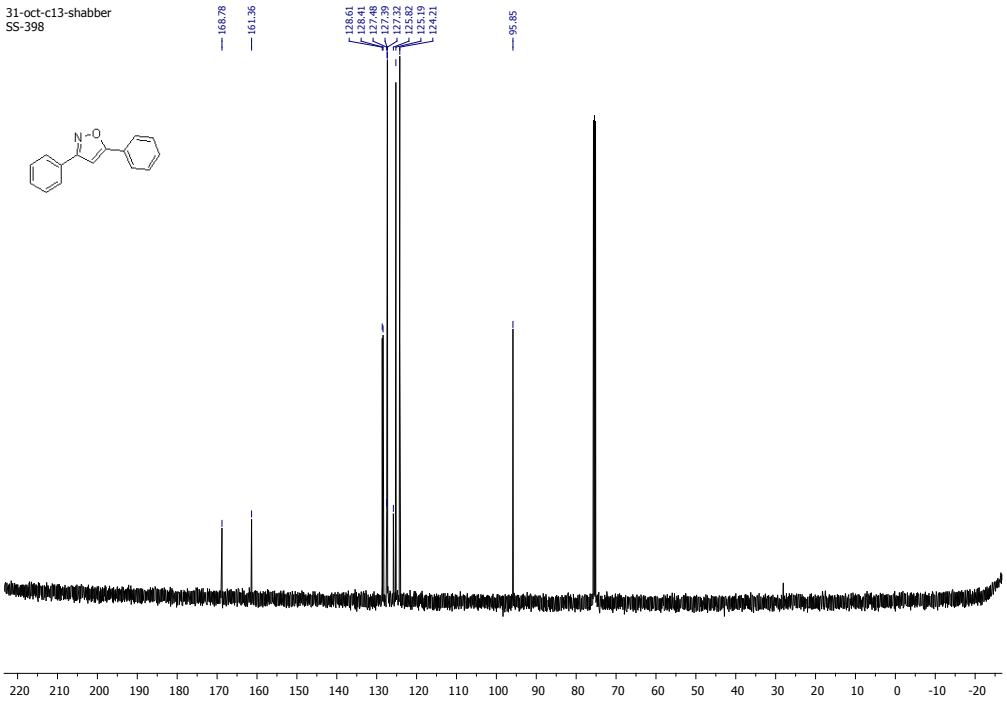




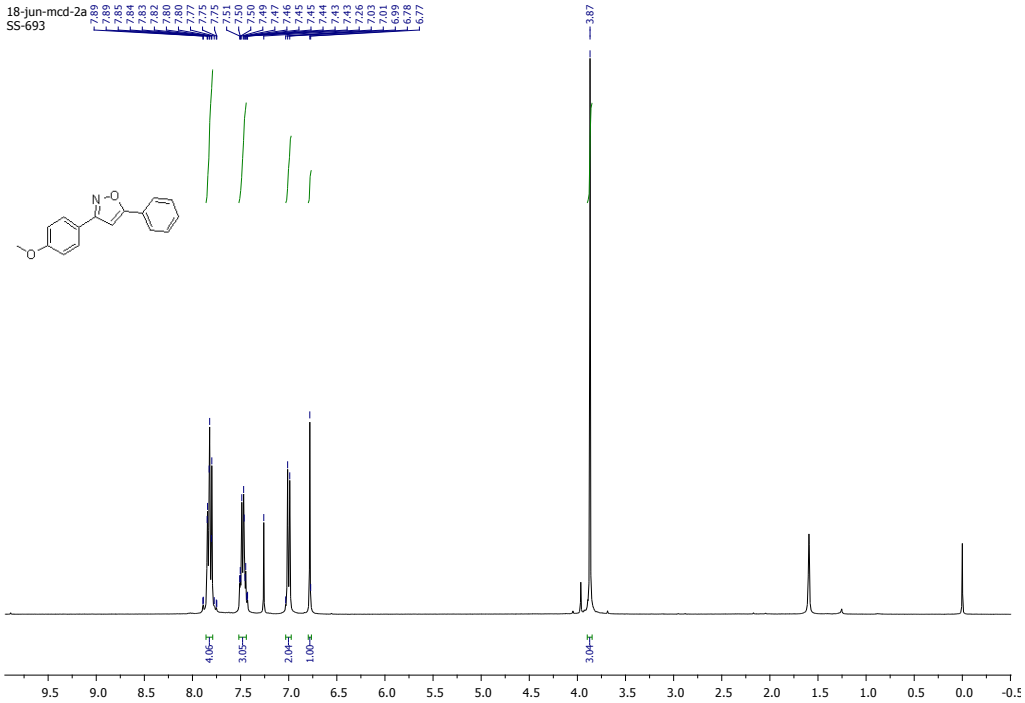

19-Jun-c13-shabbir
SS-402A
130.21
128.93
128.24
125.72
115.79
97.33
N
O
HO
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

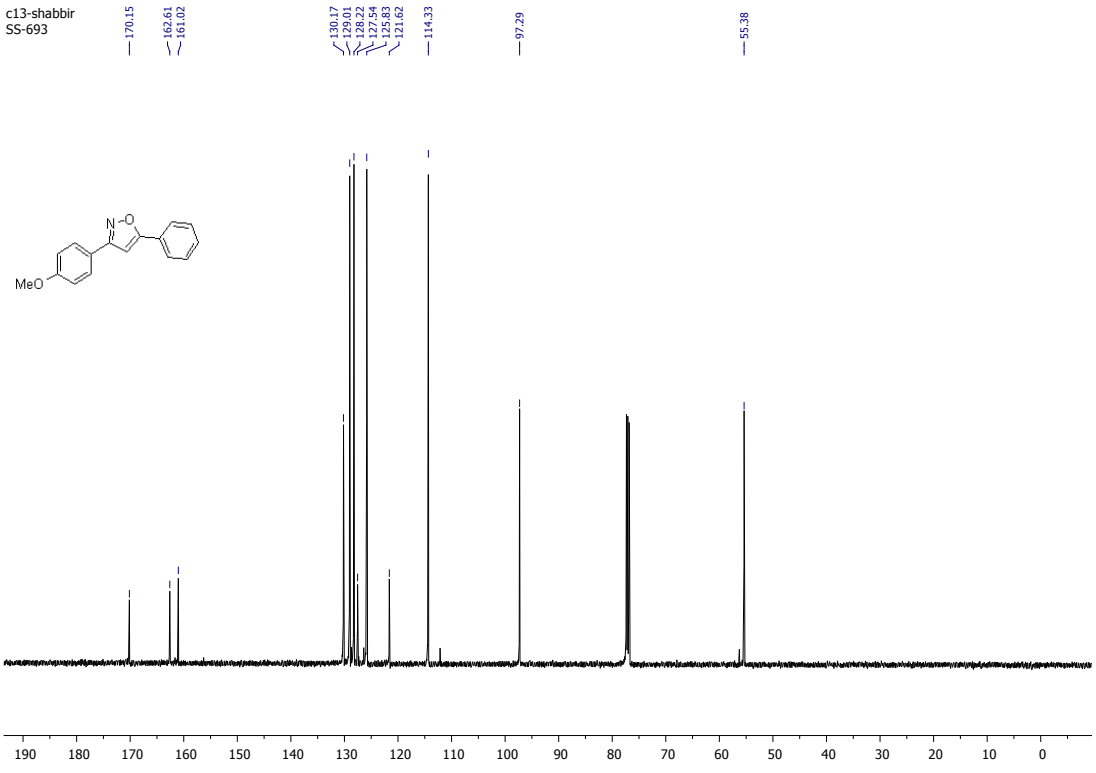
S3.2. ^{1}H, and ^{13}C NMR spectra of spectra's of 3,5-diphenylisoxazole (**4b**)

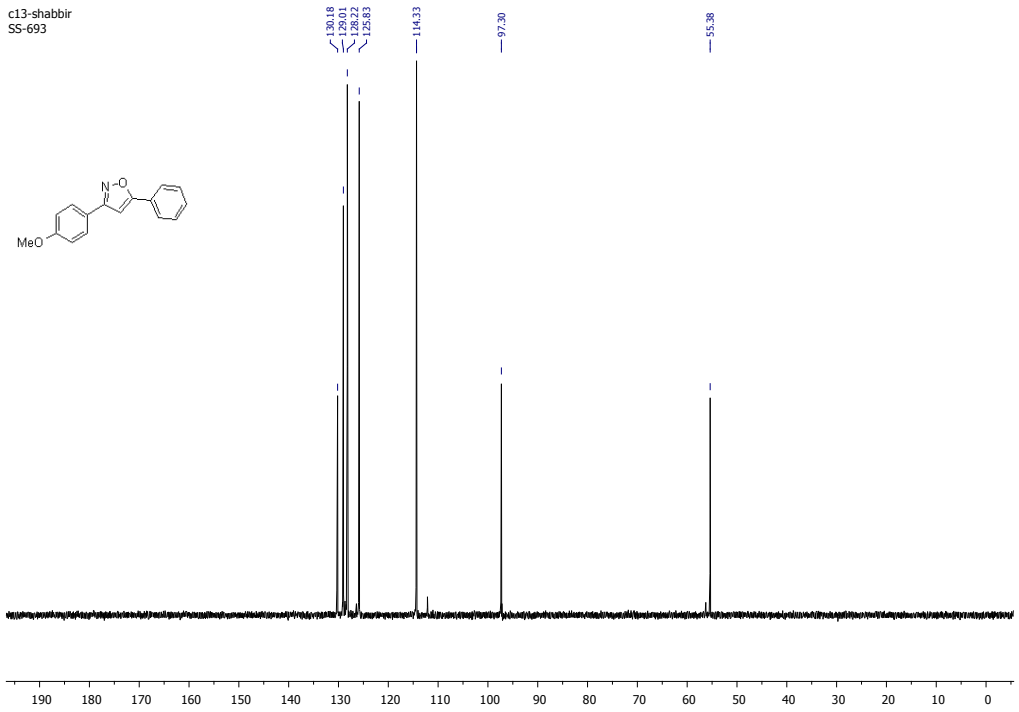




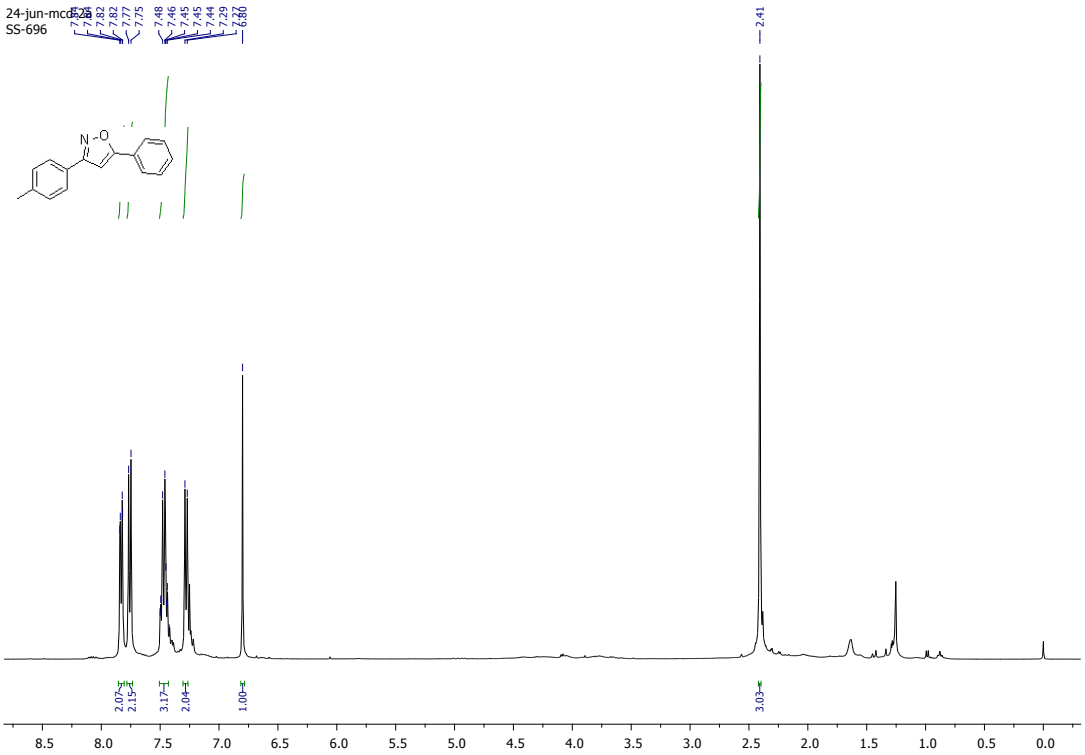
S3.3. ^{1}H, ^{13}C and DEPT135 NMR spectra of 3-(4-methoxyphenyl)-5-phenylisoxazole (**4c**)

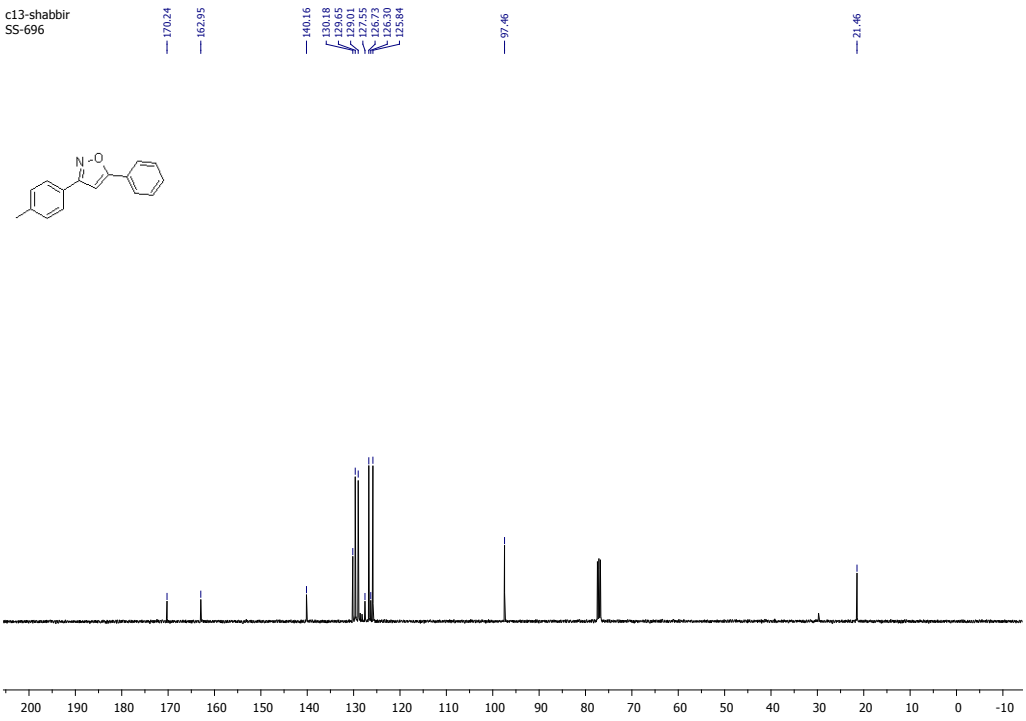


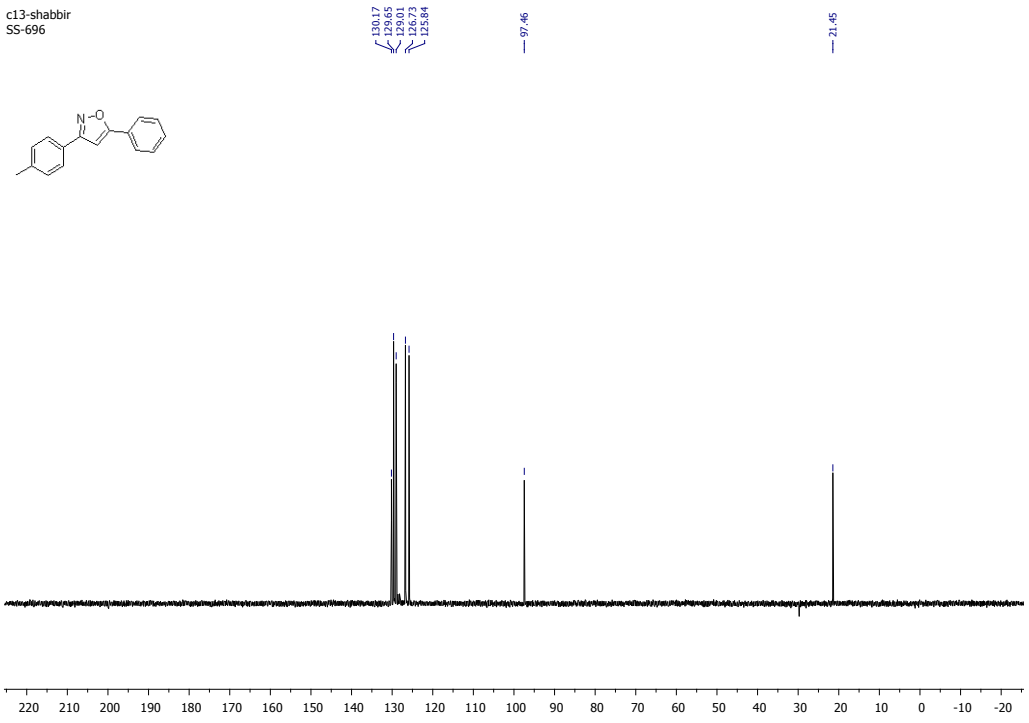



c13-shabbir
SS-693
130.18
129.01
128.22
125.83
114.33
97.30
55.38
MeO
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

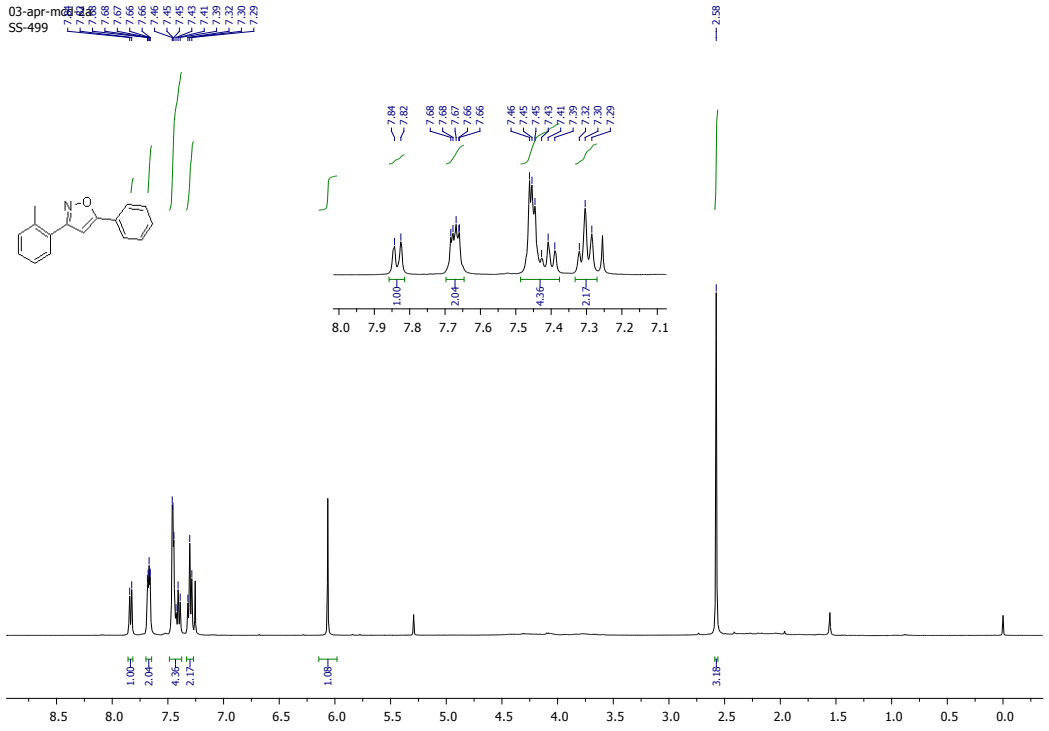
S3.4. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-phenyl-3-(p-tolyl)isoxazole (**4d**)

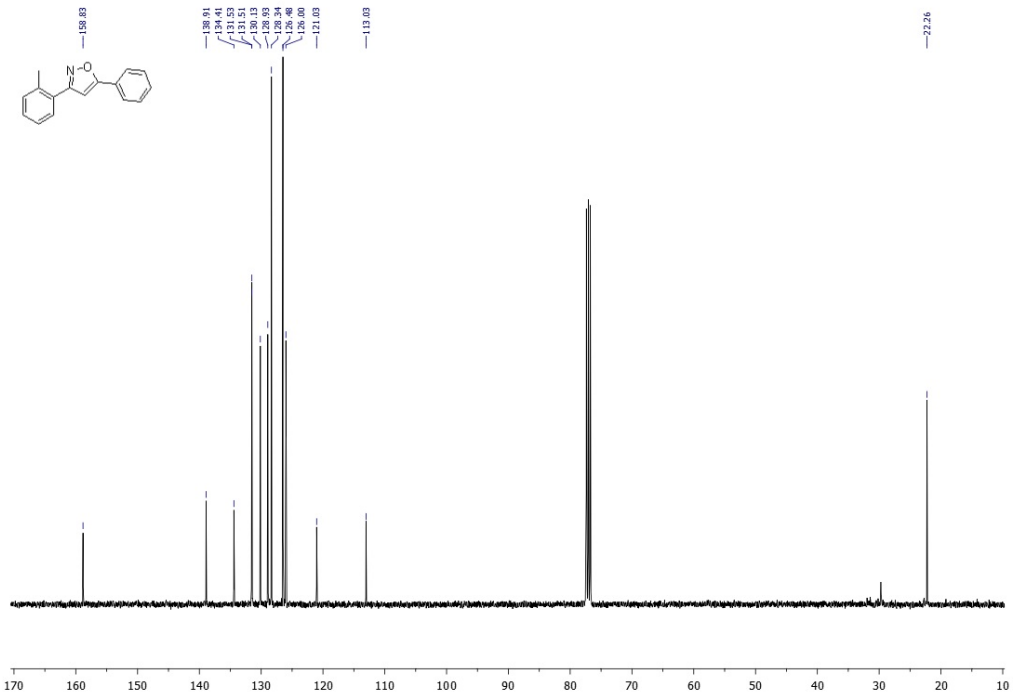


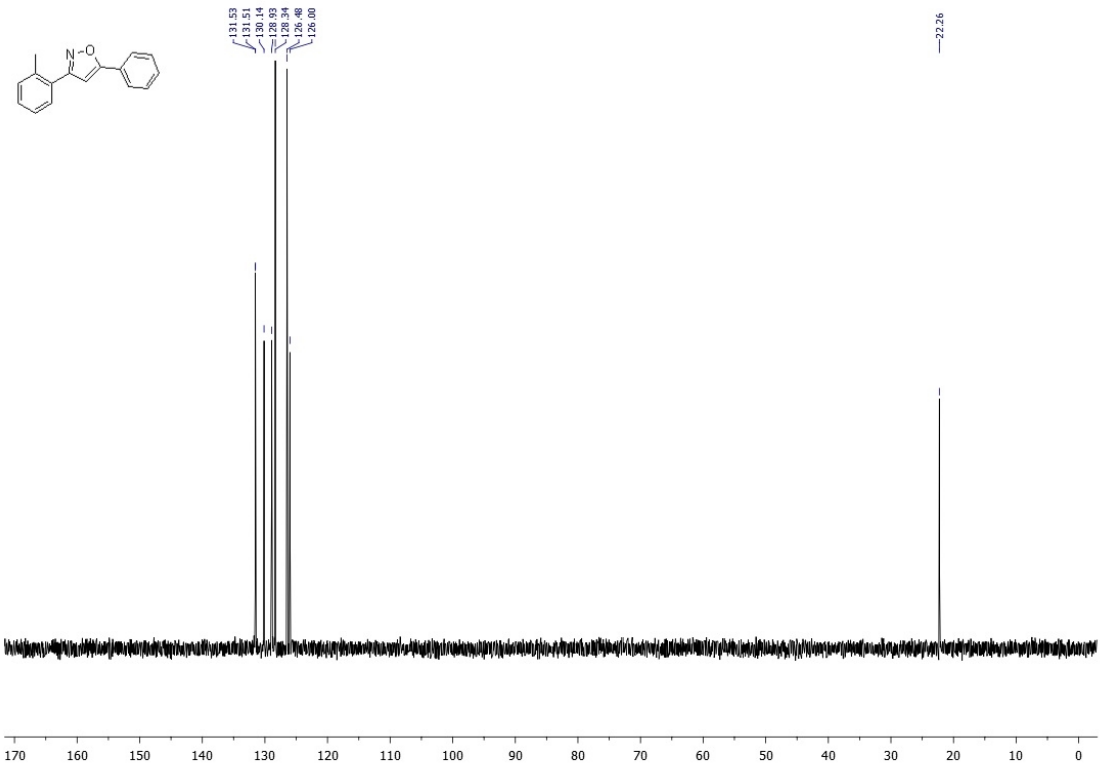



c13-shabbir
SS-696
130.17
129.65
129.01
126.73
125.84
97.46
21.45
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

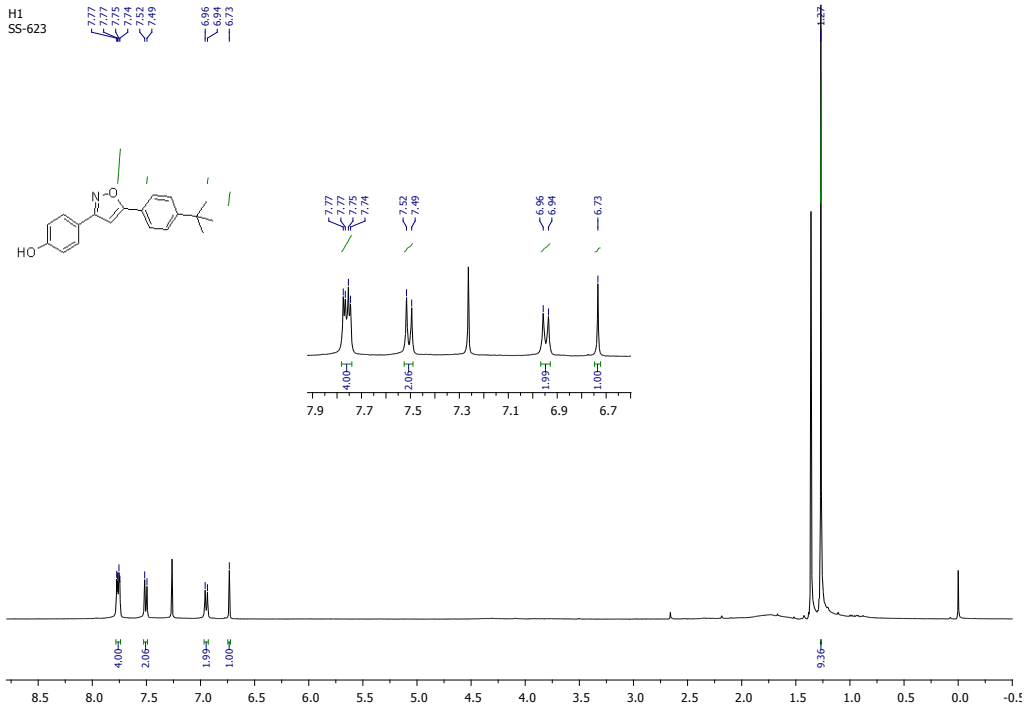
S3.5. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-phenyl-3-(o-tolyl)isoxazole (**4e**)

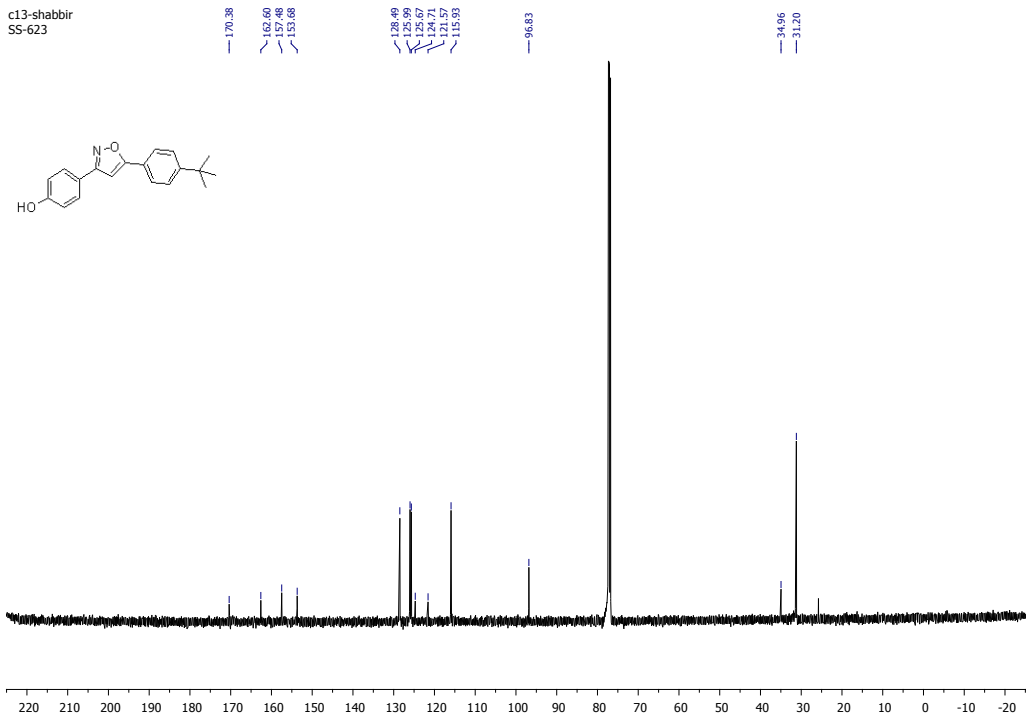


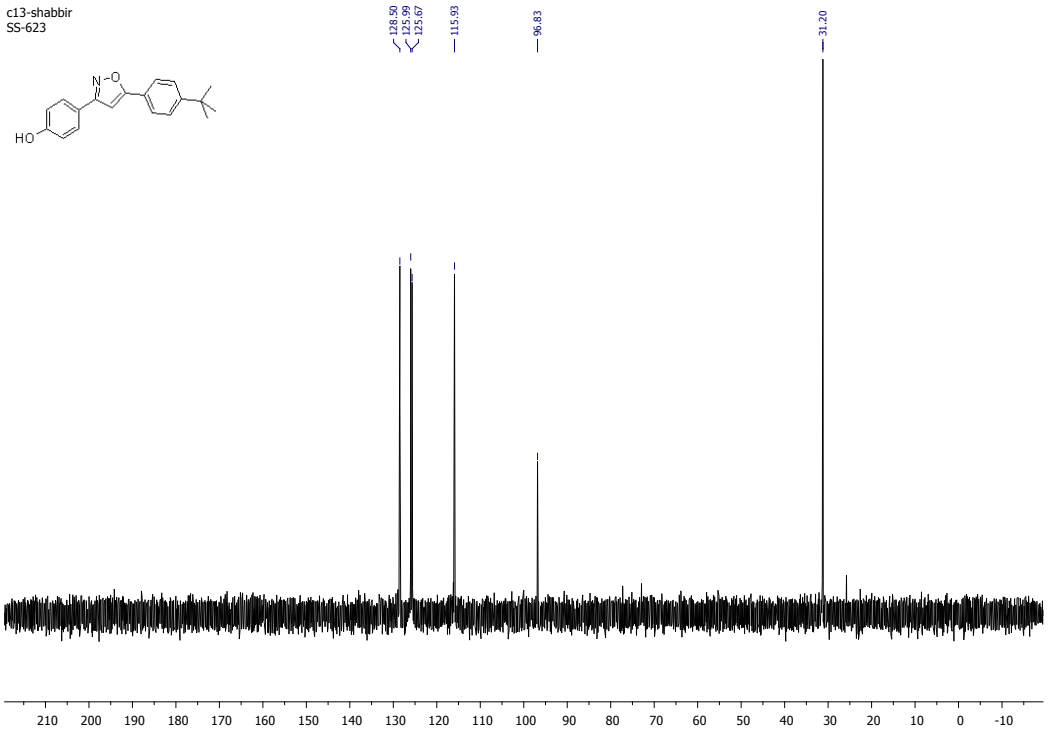



131.53
131.51
130.14
128.93
128.34
126.48
126.00
22.26
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

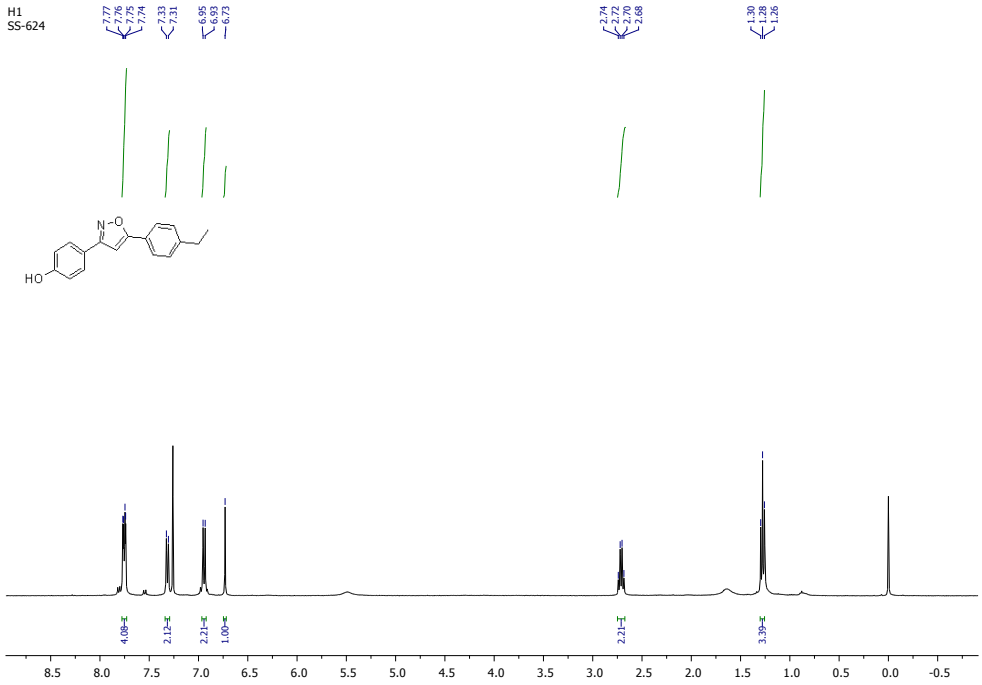
S3.6. ^{1}H, ^{13}C and DEPT135 NMR spectra of 4-(5-(4-(tert-butyl) phenyl) isoxazol-3yl) phenol (**4f**)

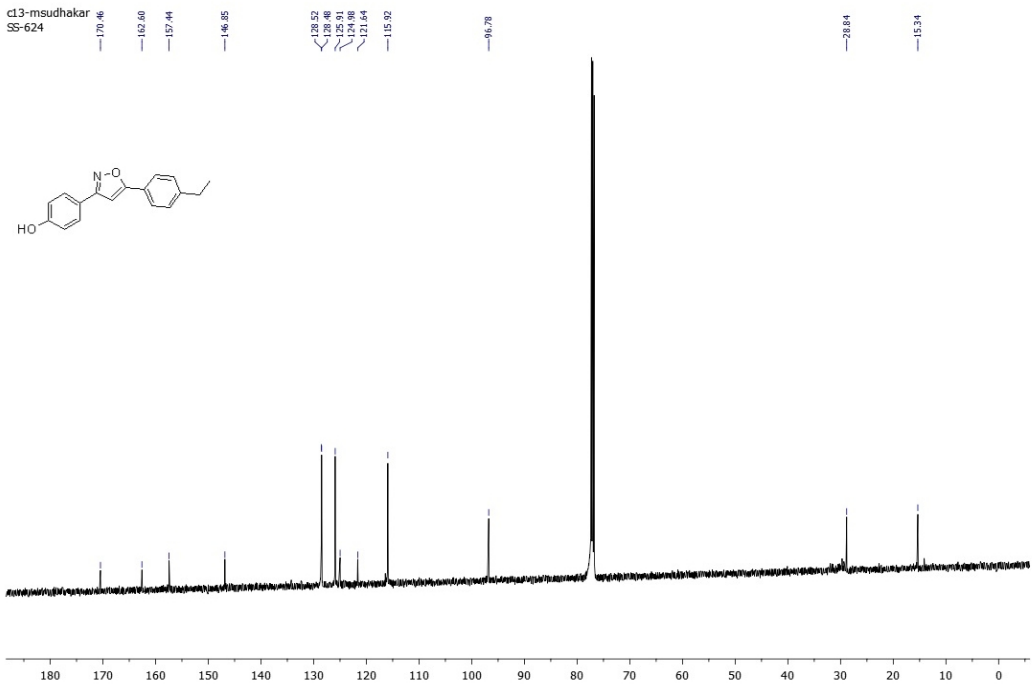


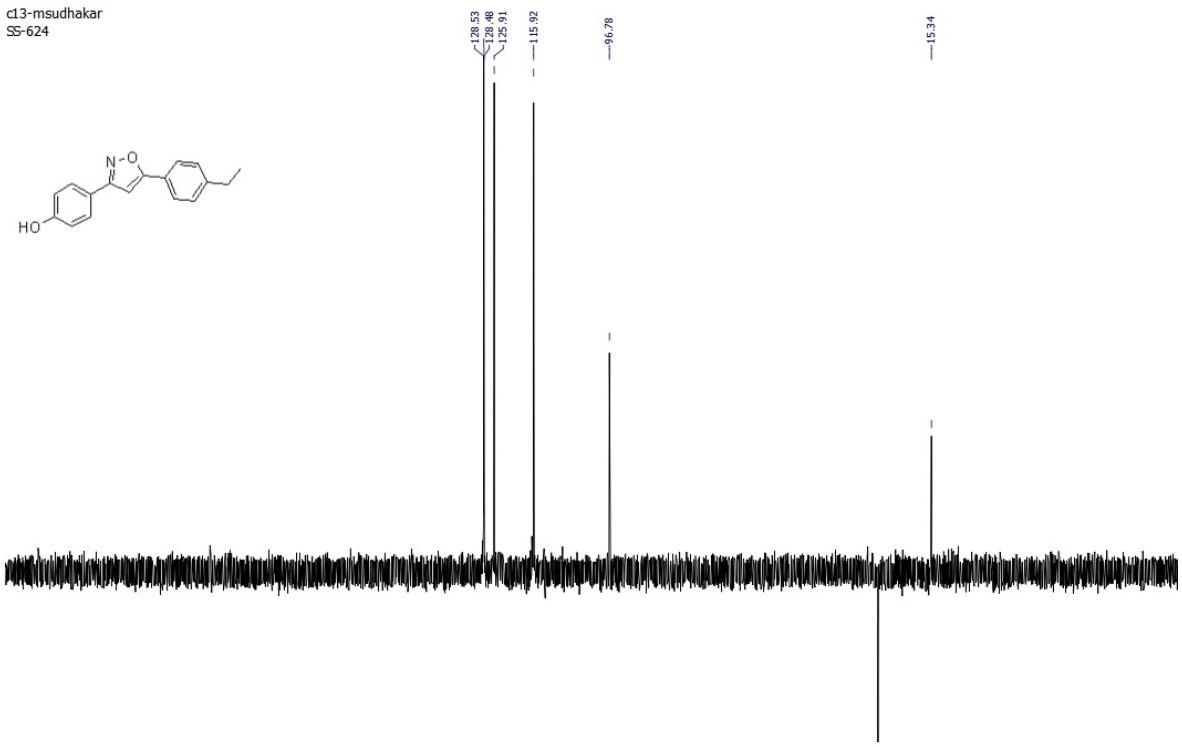



c13-shabbir
SS-623
128.50
125.99
125.67
115.93
96.83
31.20
HO
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

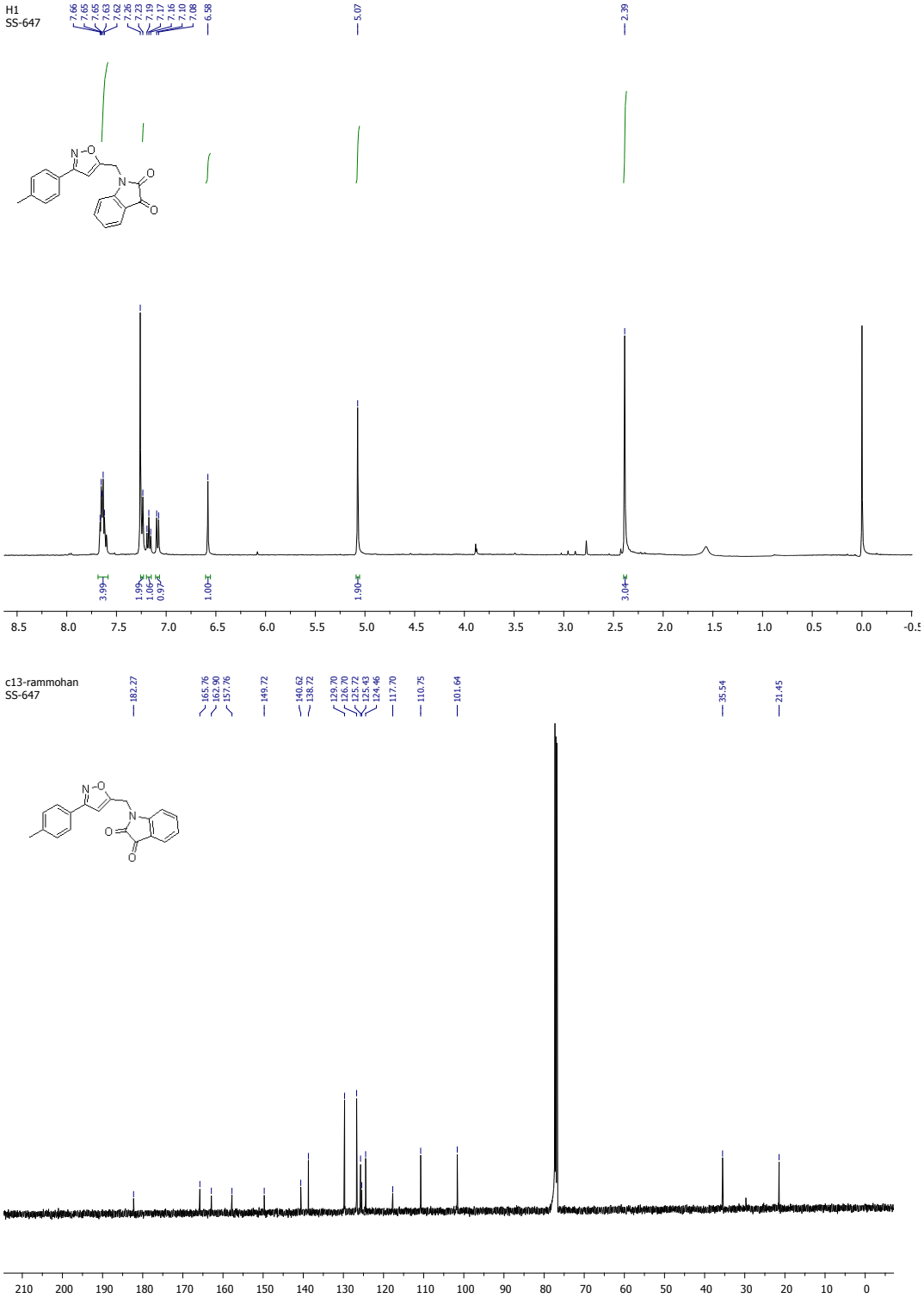
S3.7. ^{1}H, ^{13}C and DEPT135 NMR spectra of 4-(5-(4-ethylphenyl) isoxazole-3-yl) phenol (**4g**)

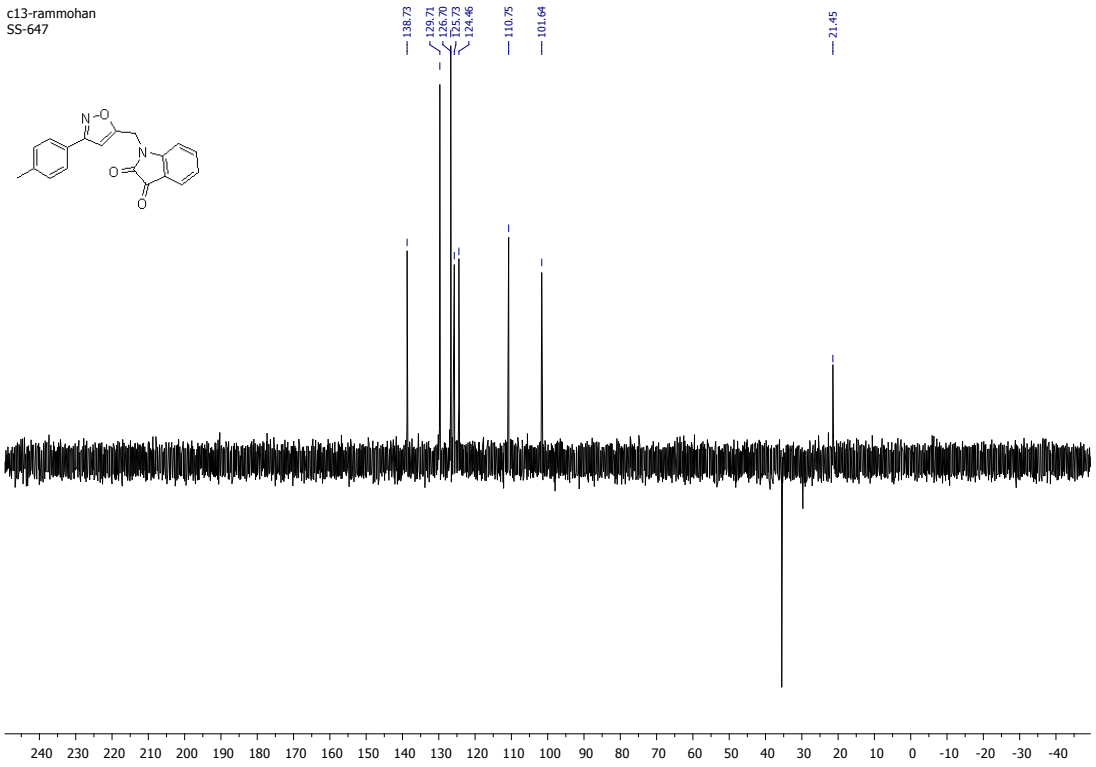




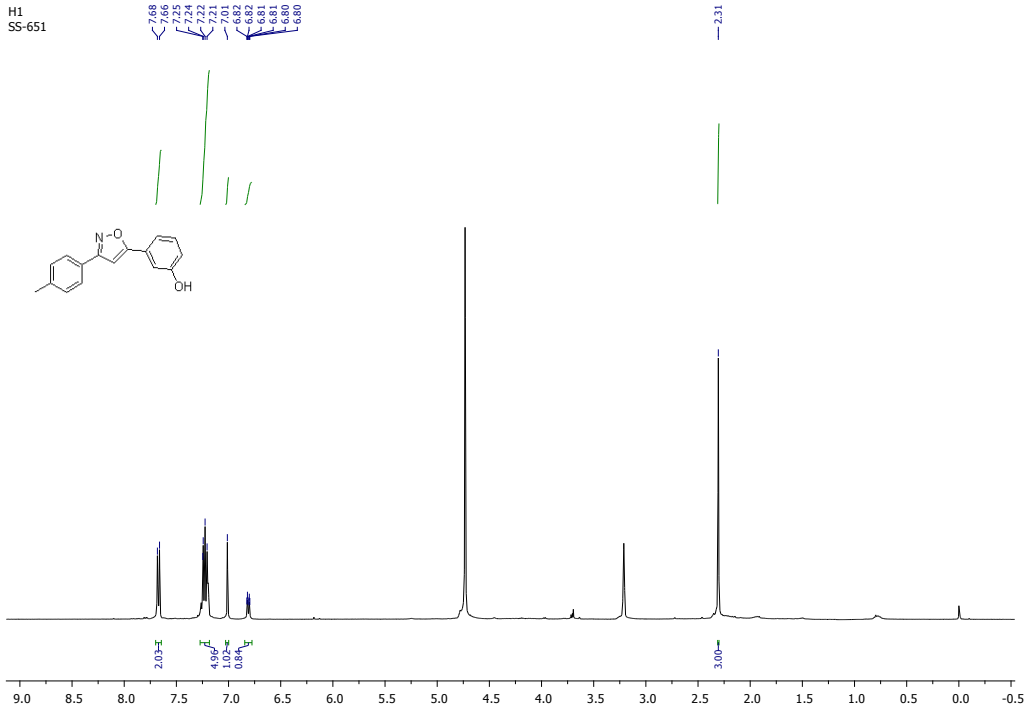

c13-msudhakar
SS-624
128.53
128.48
125.91
115.92
96.78
15.34
N
O
HO

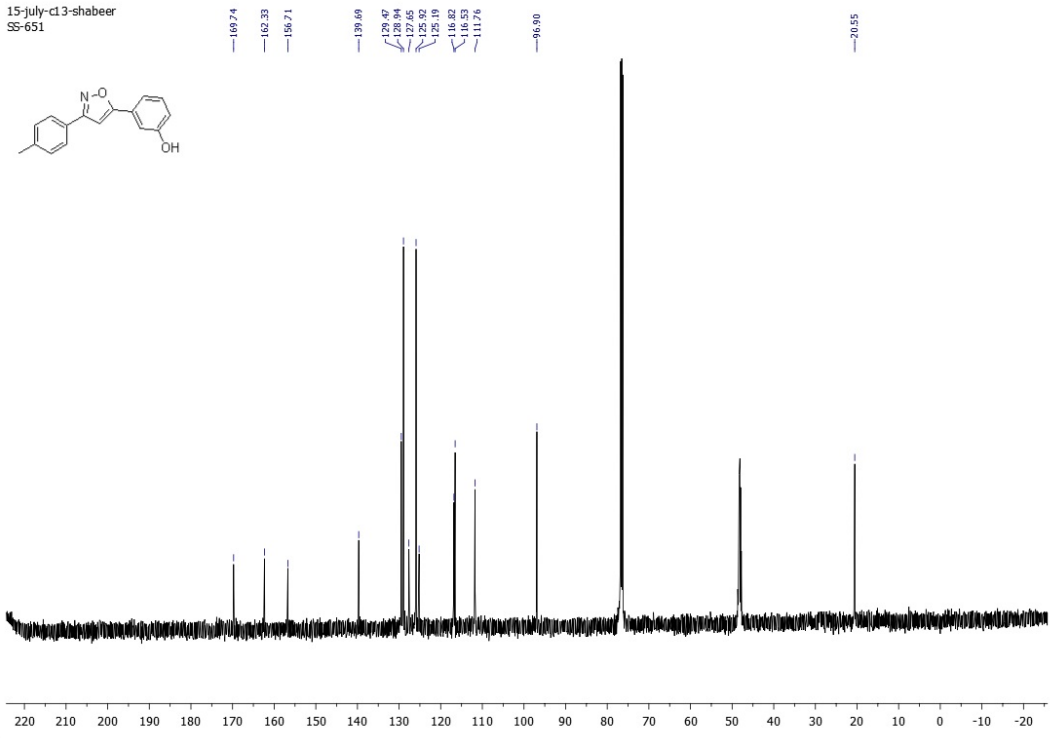
S3.8. ^{1}H, ^{13}C and DEPT135 NMR spectra of 3-((3-(p-tolyl) isoxazole-5yl) methyl)-1H-indene-1,2(3H)-dione (**4h**)

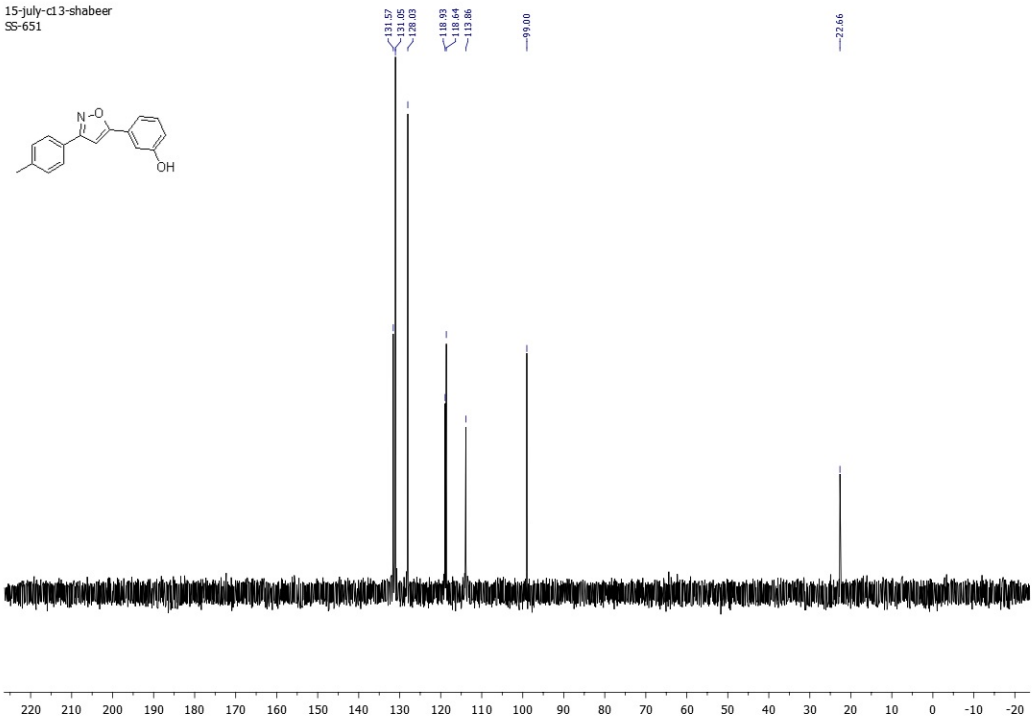



c13-rammohan
SS-647
138.73
129.71
126.70
125.73
124.46
110.75
101.64
21.45
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40

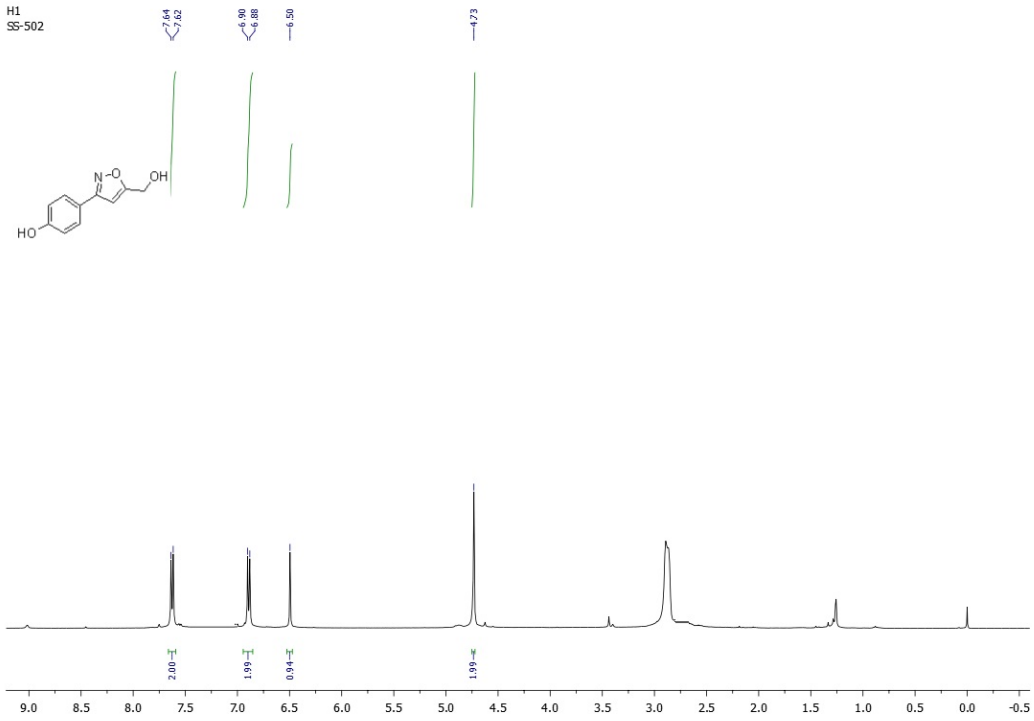
S3.9. ^{1}H, ^{13}C and DEPT135 NMR spectra of 3-(3-(p-tolyl) isoxazole-5-yl) phenol (**4i**)

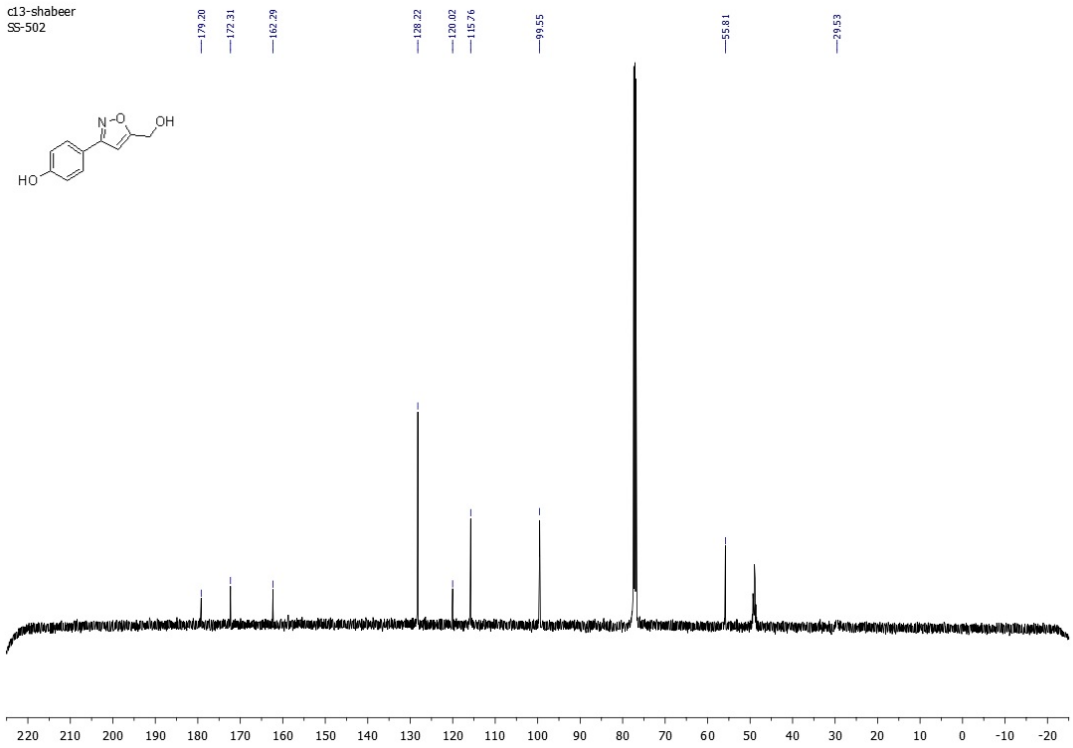


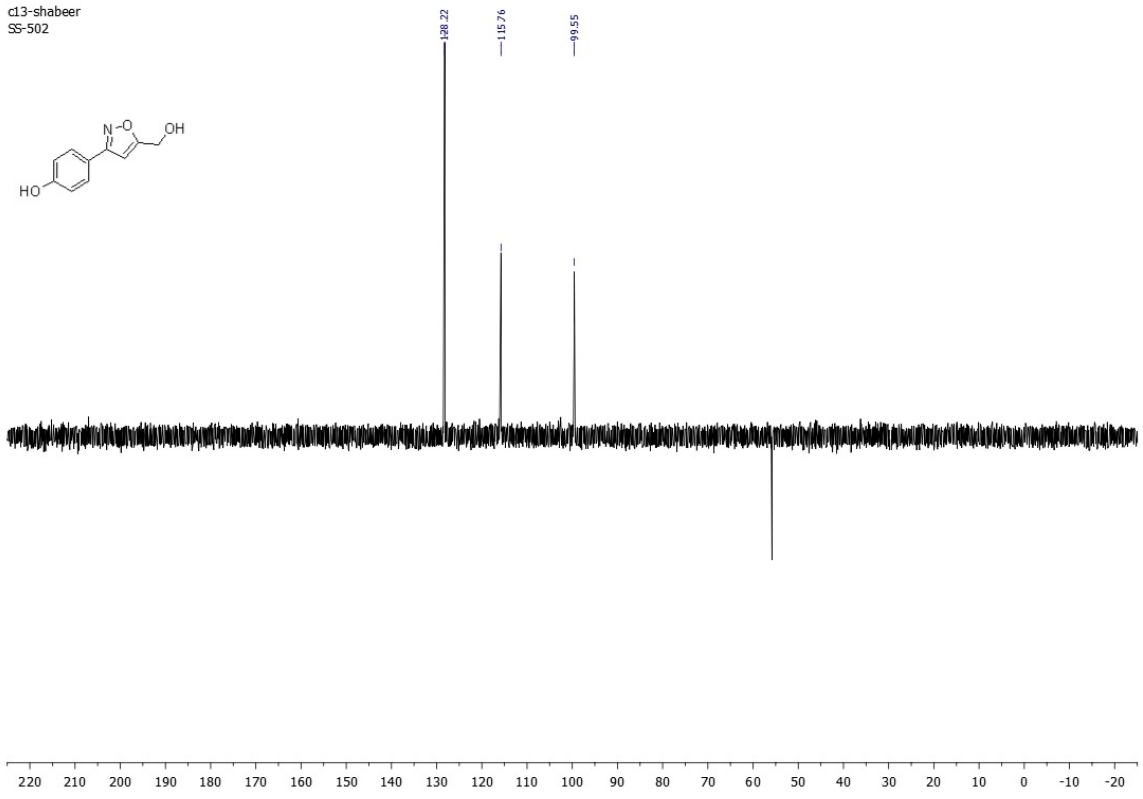



15-july-c13-shabeer
SS-651
131.57
131.05
128.03
118.93
118.64
113.86
99.00
22.66
N
O
OH
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

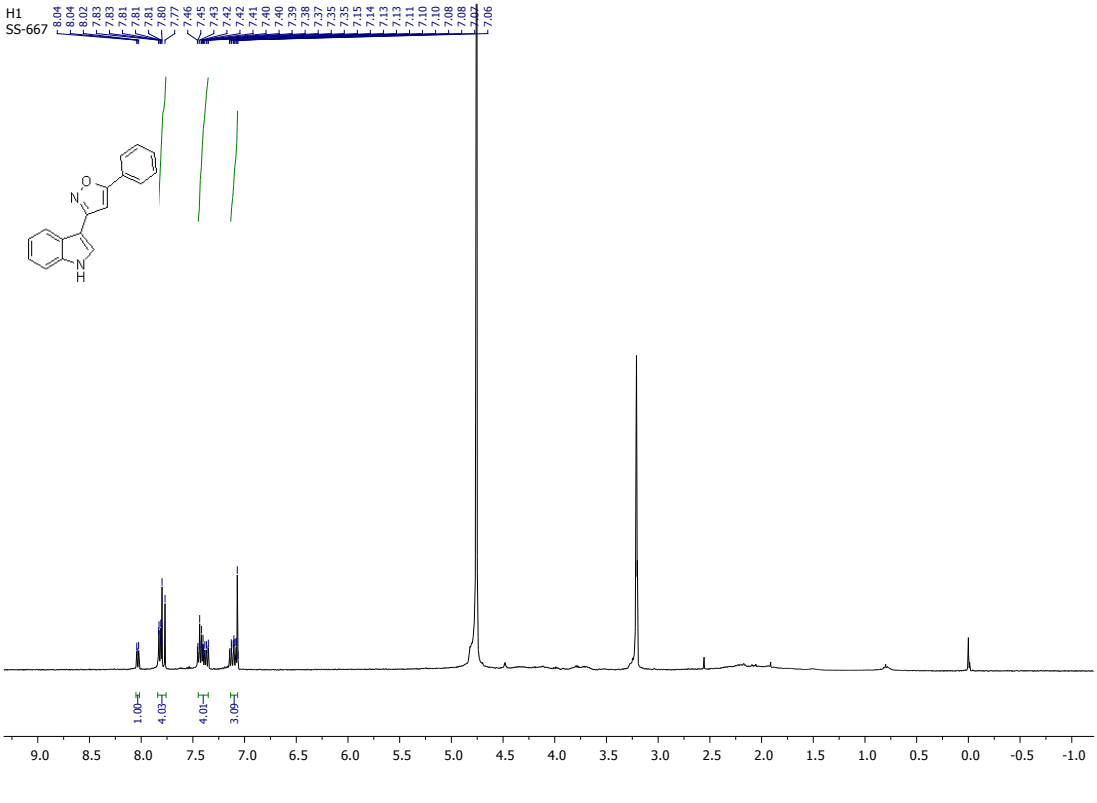
S3.10. ^{1}H, ^{13}C and DEPT135 NMR spectra of 4-(5-(hydroxymethyl) isoxazole-3-yl) phenol (**4j**)

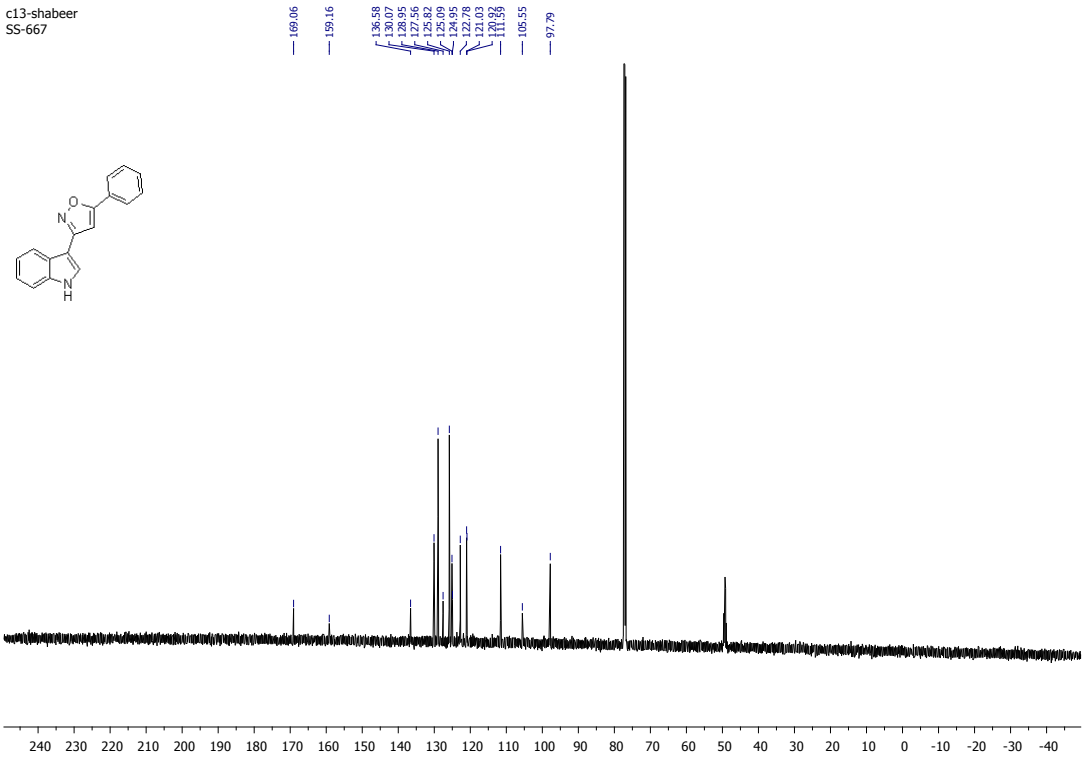


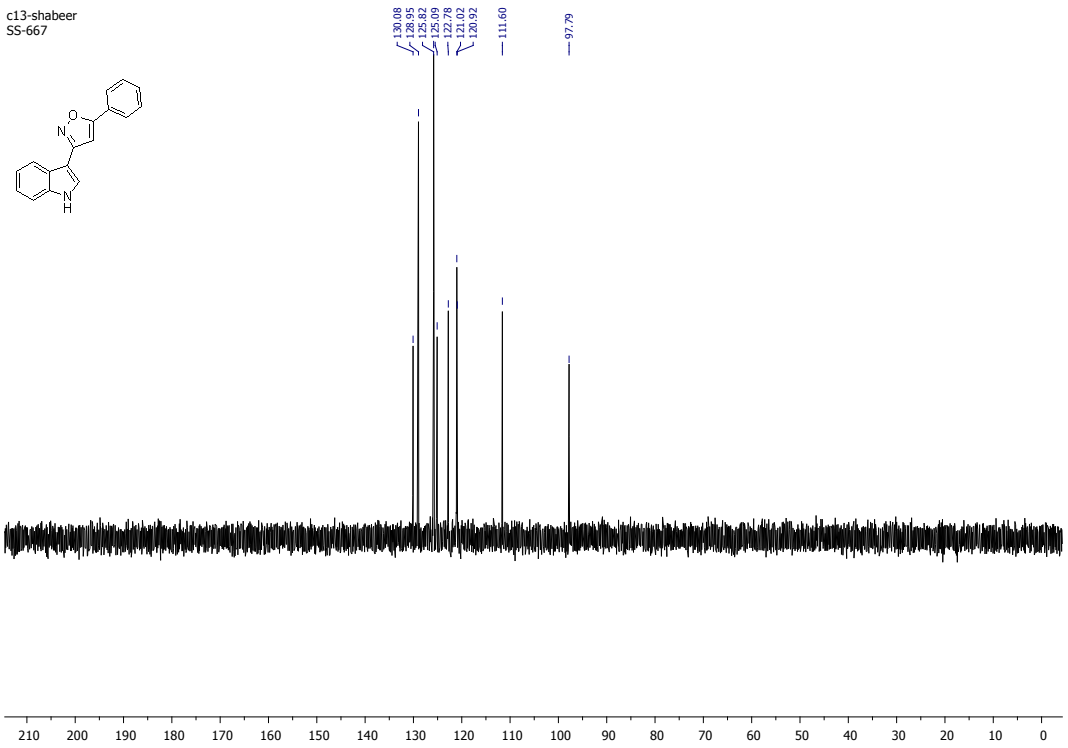



c13-shabeer
SS-502
128.22
115.76
99.55
N
O
OH
HO
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

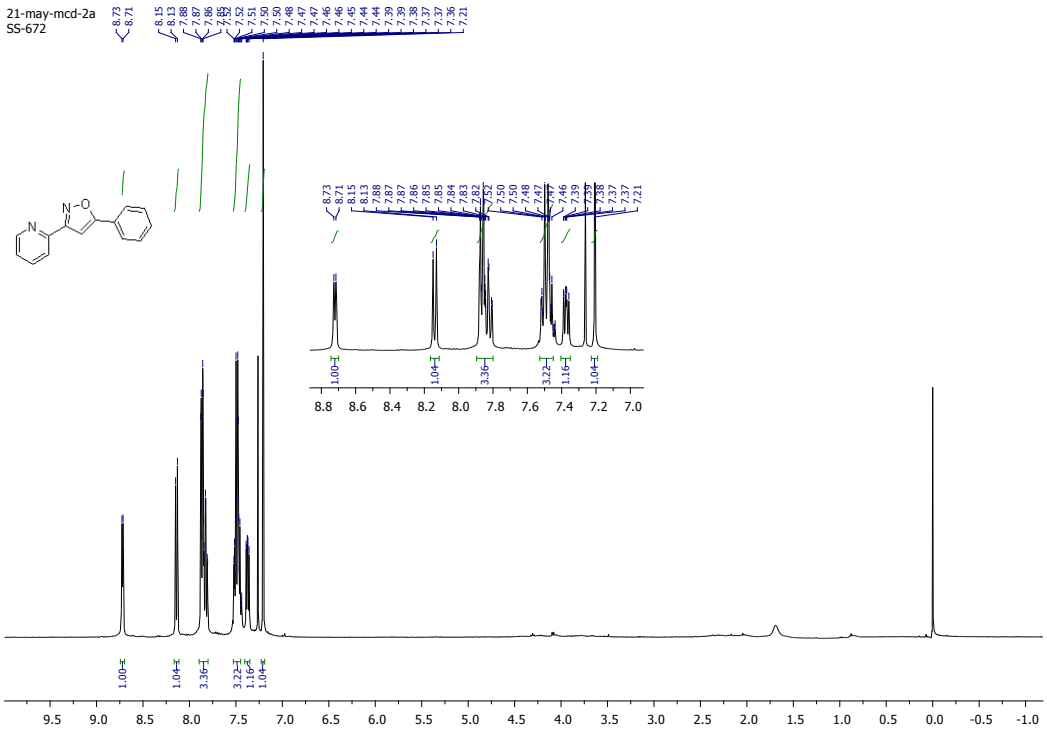
S3.11. ^{1}H, ^{13}C and DEPT135 NMR spectra of 3-(1H-indol-3-yl)-5-phenylisoxazole (**4k**)

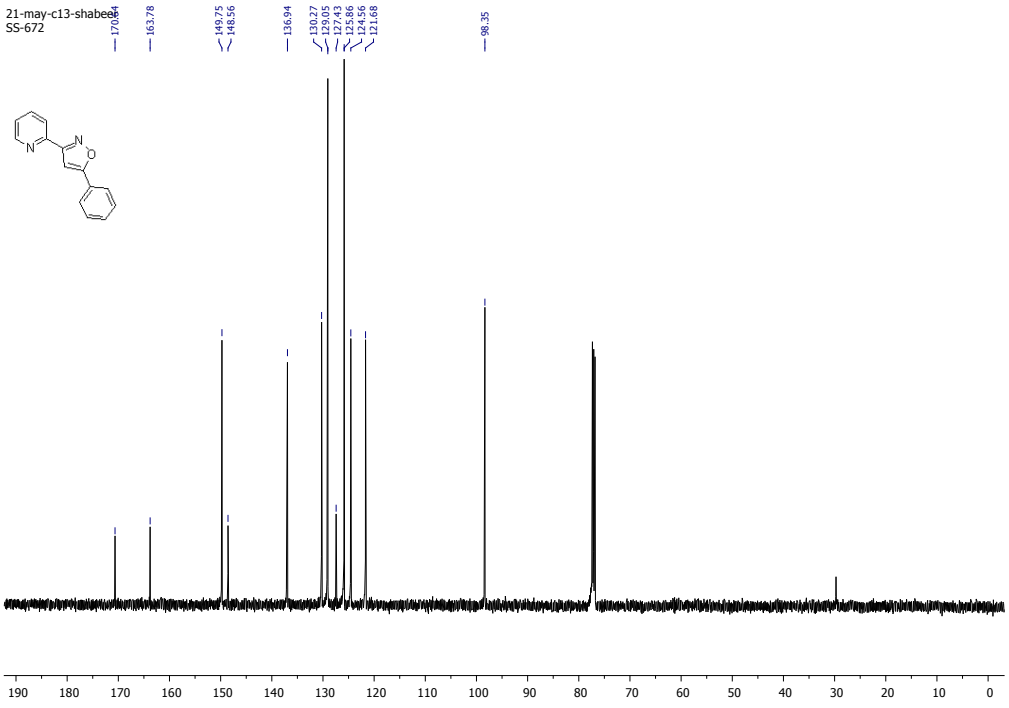


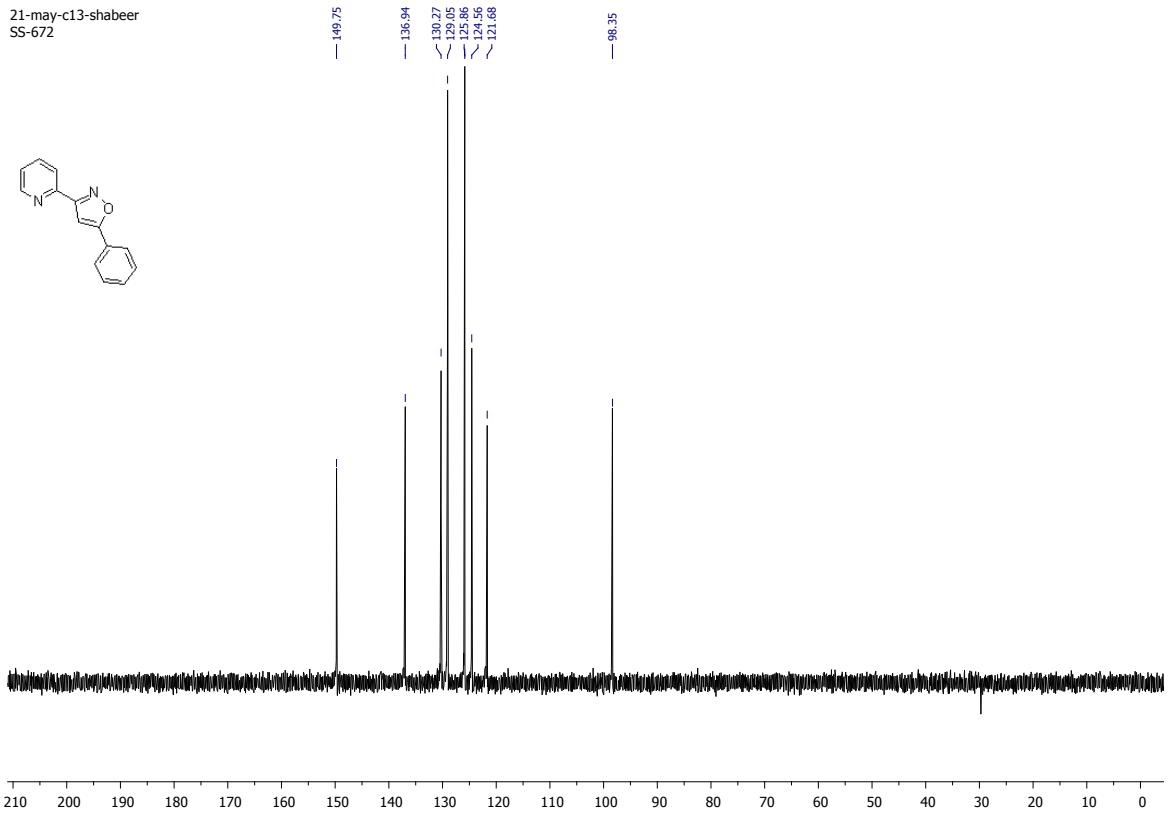



c13-shabeer
SS-667
130.08
128.95
125.82
125.09
122.78
121.02
120.92
111.60
97.79
O
N
N
H
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

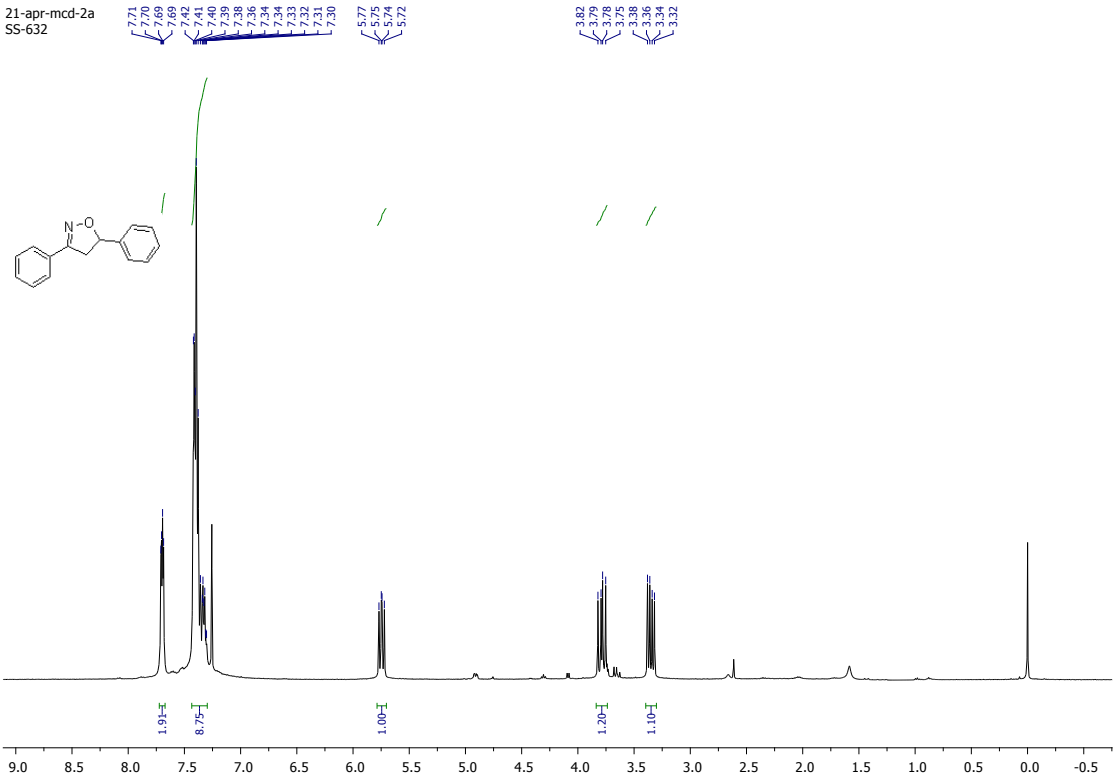
S3.12, ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-phenyl-3-(pyridine-2-yl) isoxazole (**4l**)

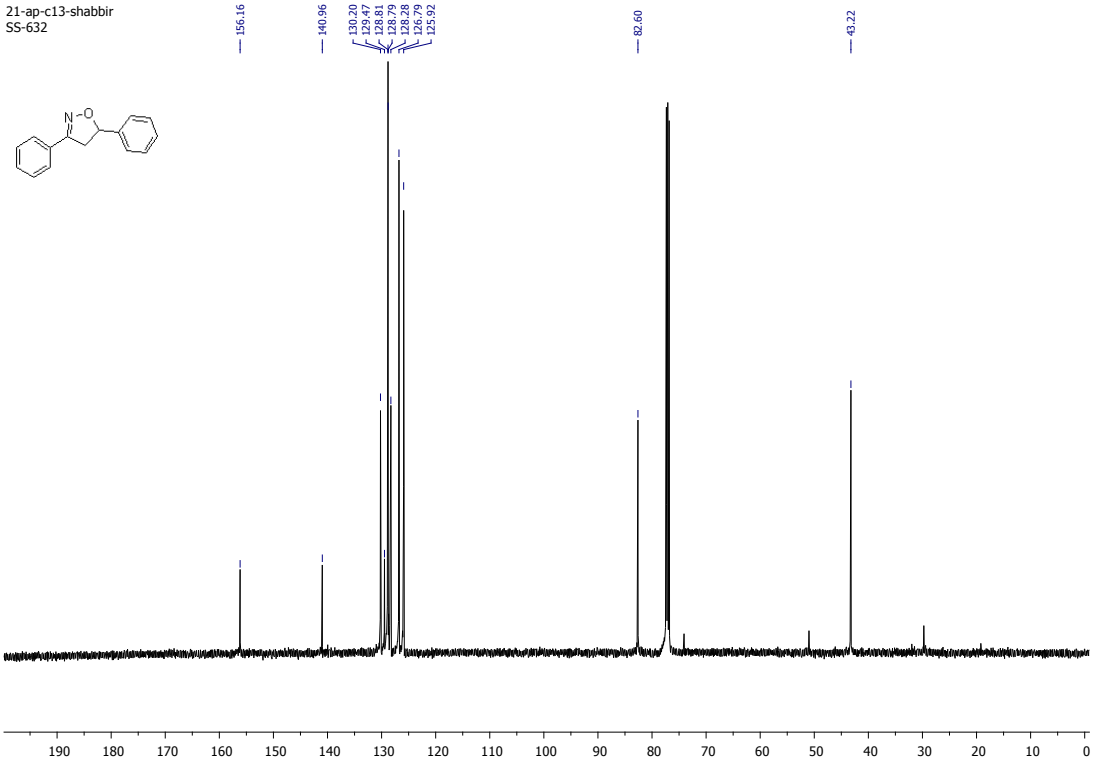


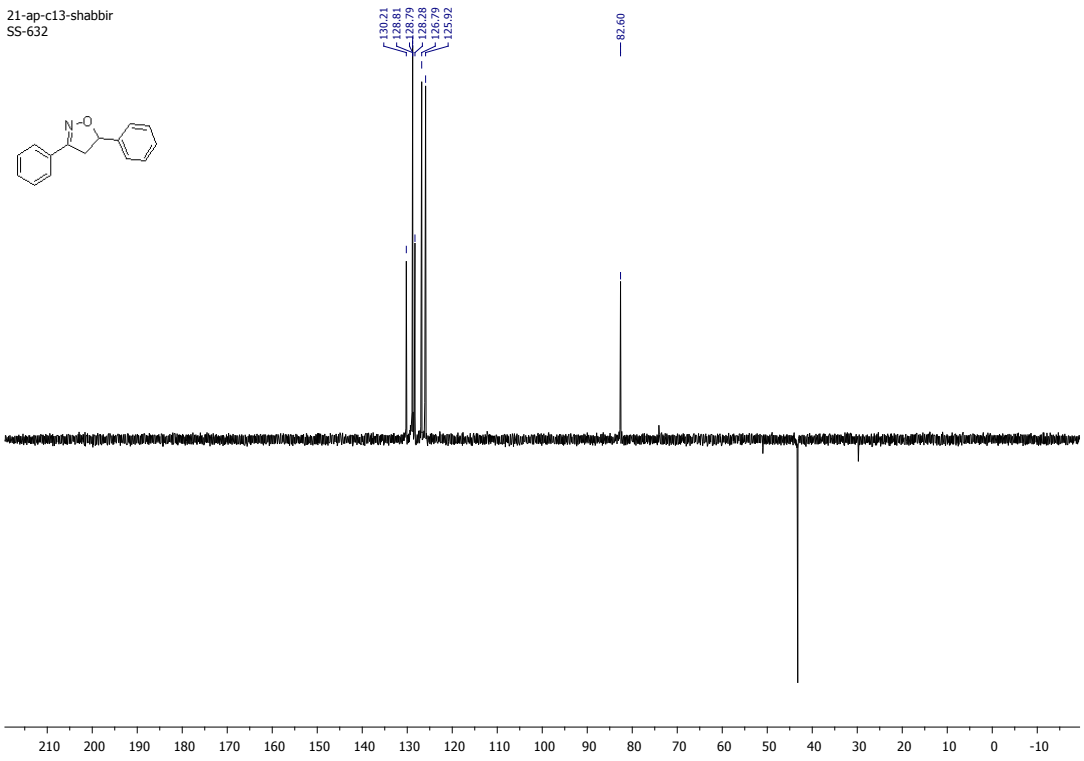



21-may-c13-shabeer
SS-672
149.75
136.94
130.27
129.05
125.86
124.56
121.68
98.35
N
N
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

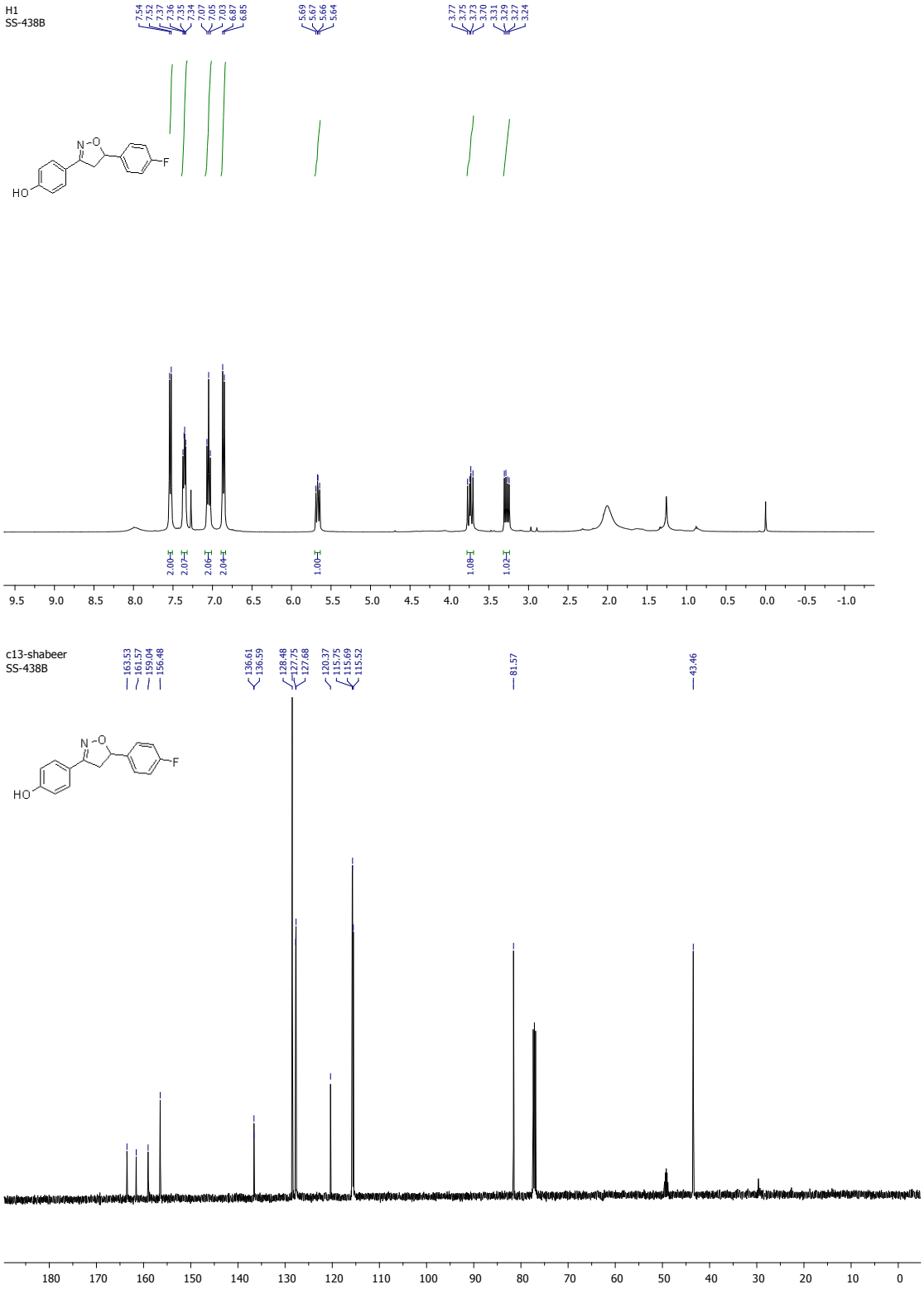
S3.13.^{1}H, ^{13}C, and DEPT135 spectra of 3,5-diphenyl-4,5-dihydroisoxazole (**6a**)

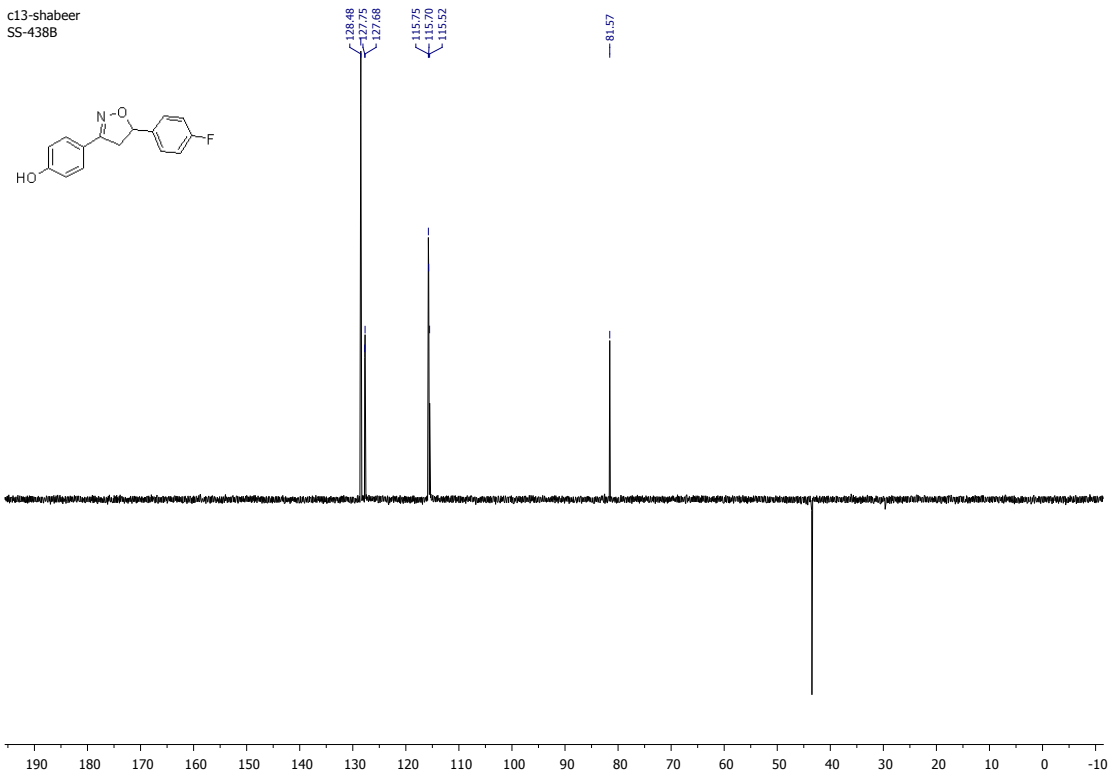




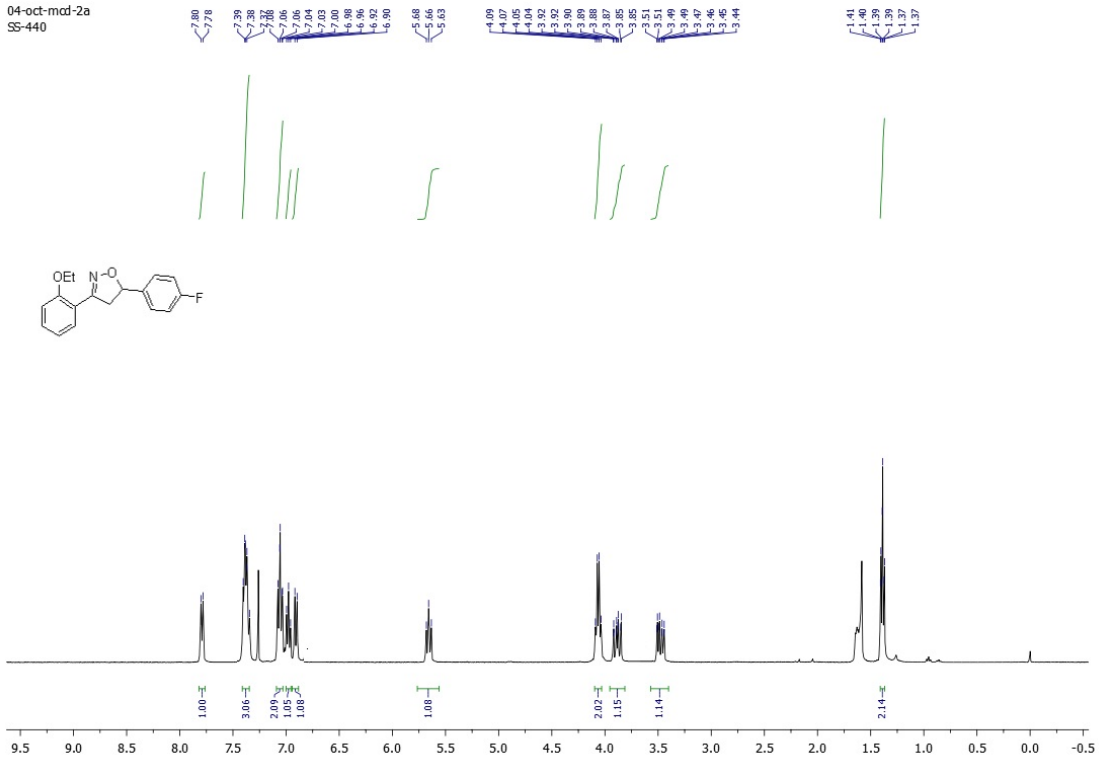

21-ap-c13-shabbir
SS-632
130.21
128.81
128.79
128.28
126.79
125.92
82.60
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

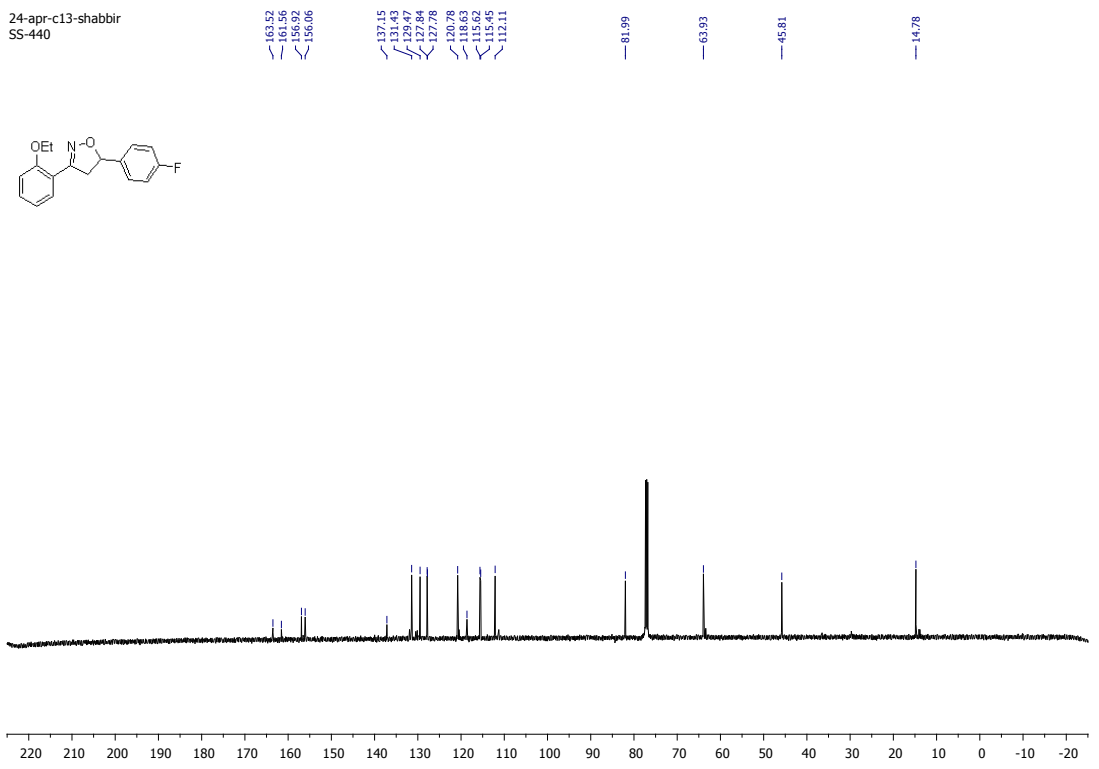
S3.14.^{1}H, ^{13}C, and DEPT135 spectra of 4-(5-(4-fluorophenyl)-4, 5-dihydroisoxazol-3-yl) phenol (**6b**)

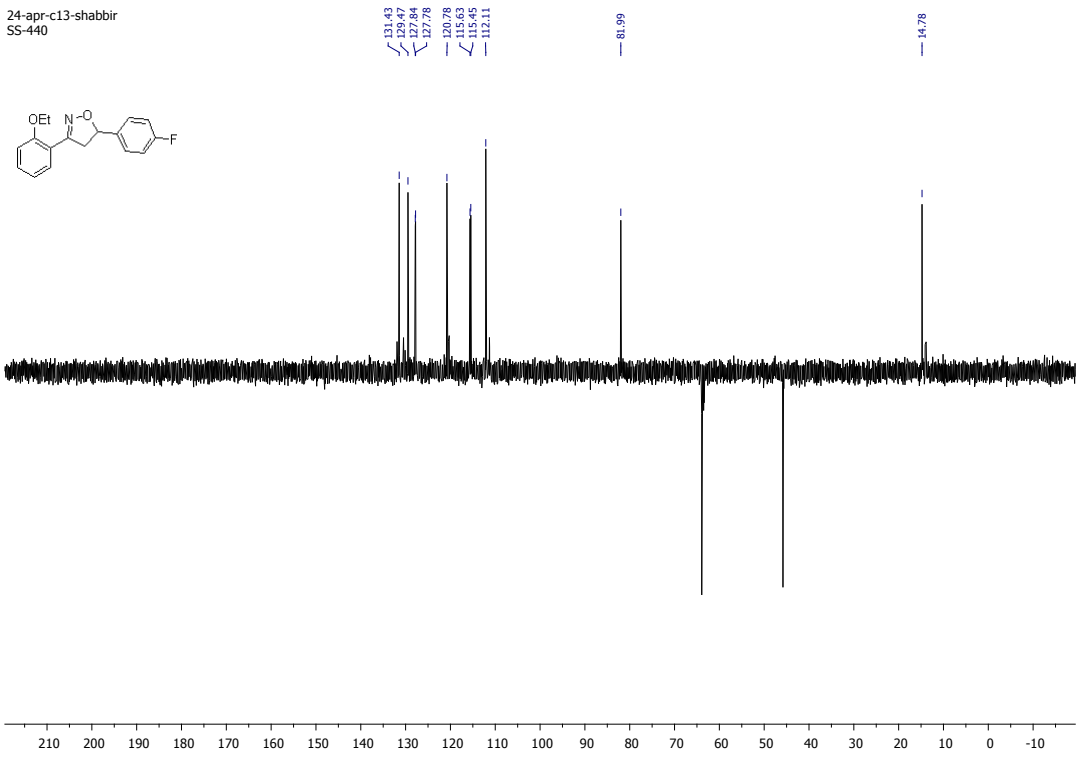



c13-shabeer
SS-438B
128.48
127.75
127.68
115.75
115.70
115.52
81.57
N
O
F
HO
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

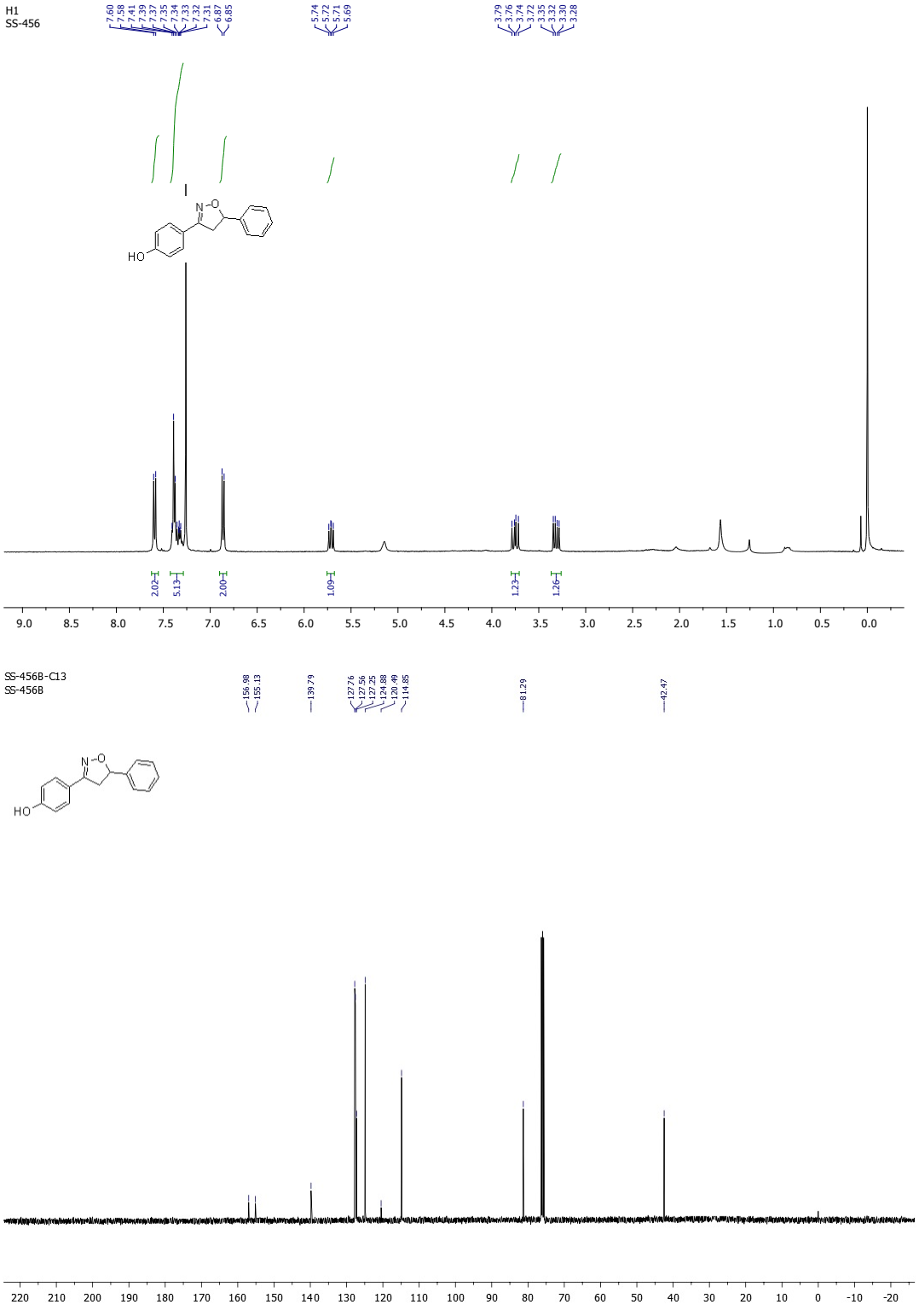
S3.15.^{1}H, ^{13}C, and DEPT135 spectra of 3-(2-ethoxyphenyl)-5-(4-fluorophenyl)-4,5-dihydroisoxazole (**6c**)

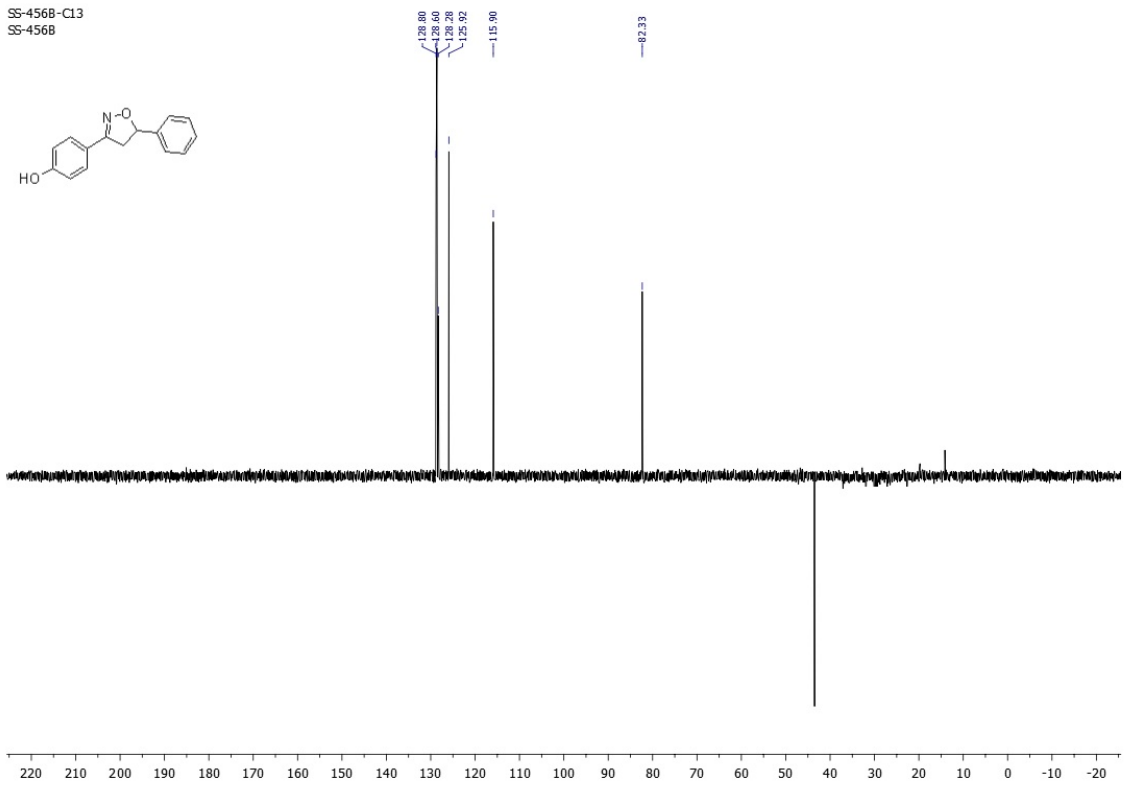




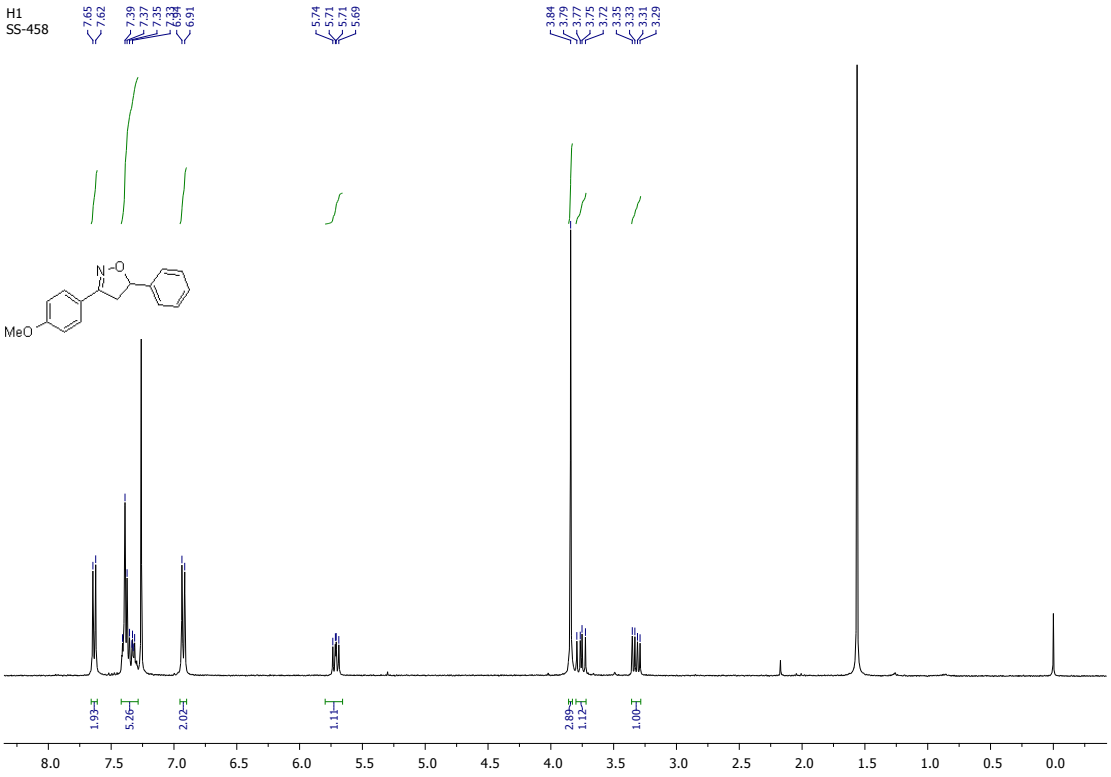

24-apr-c13-shabbir
SS-440
131.43
129.47
127.84
127.78
120.78
115.63
115.45
112.11
81.99
14.78
OEt
N
O
F
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

S3.16.^{1}H, ^{13}C, and DEPT135 spectra of 4-(5-phenyl-4, 5-dihydroisoxazole-3-yl) phenol (**6d**)

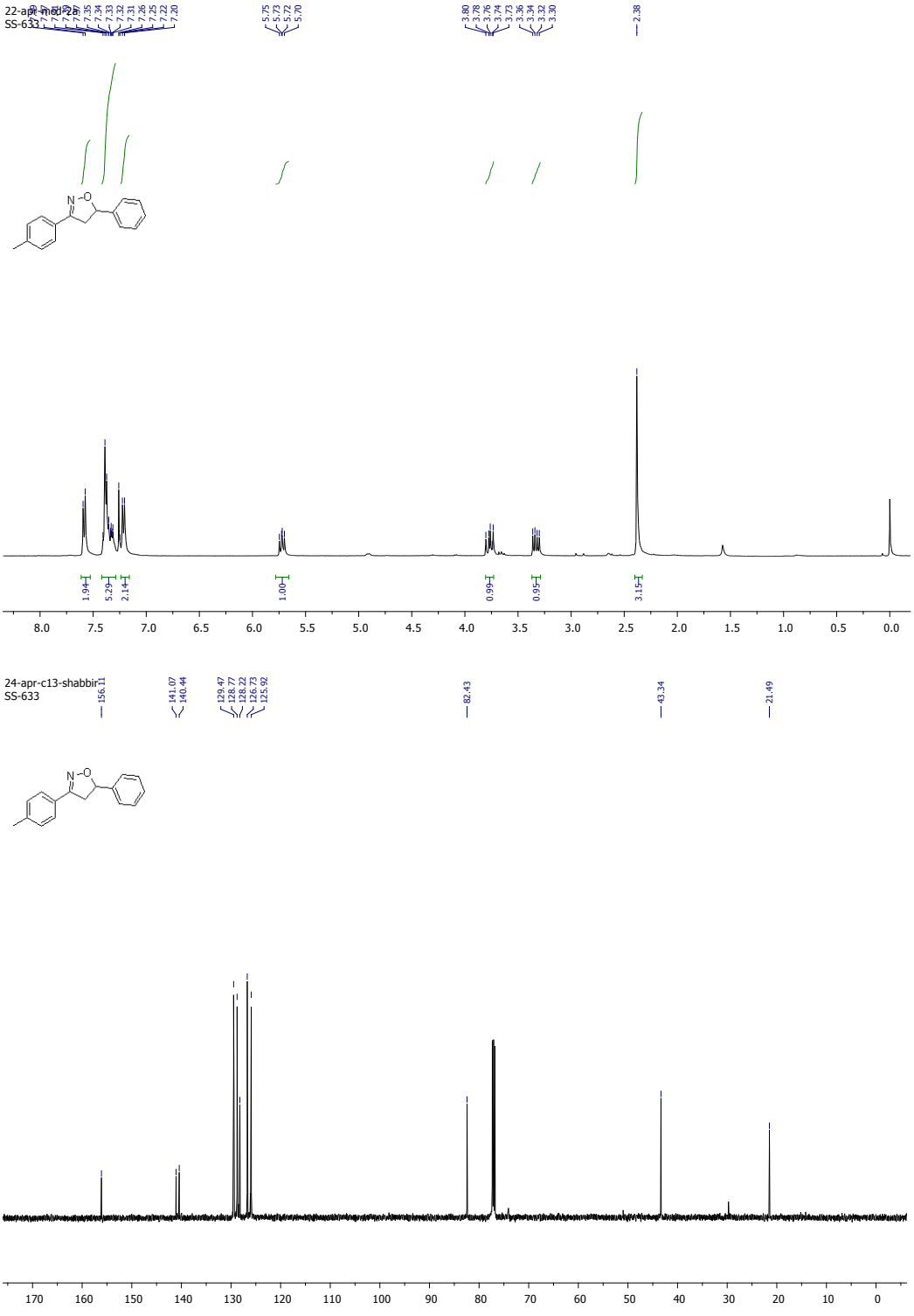


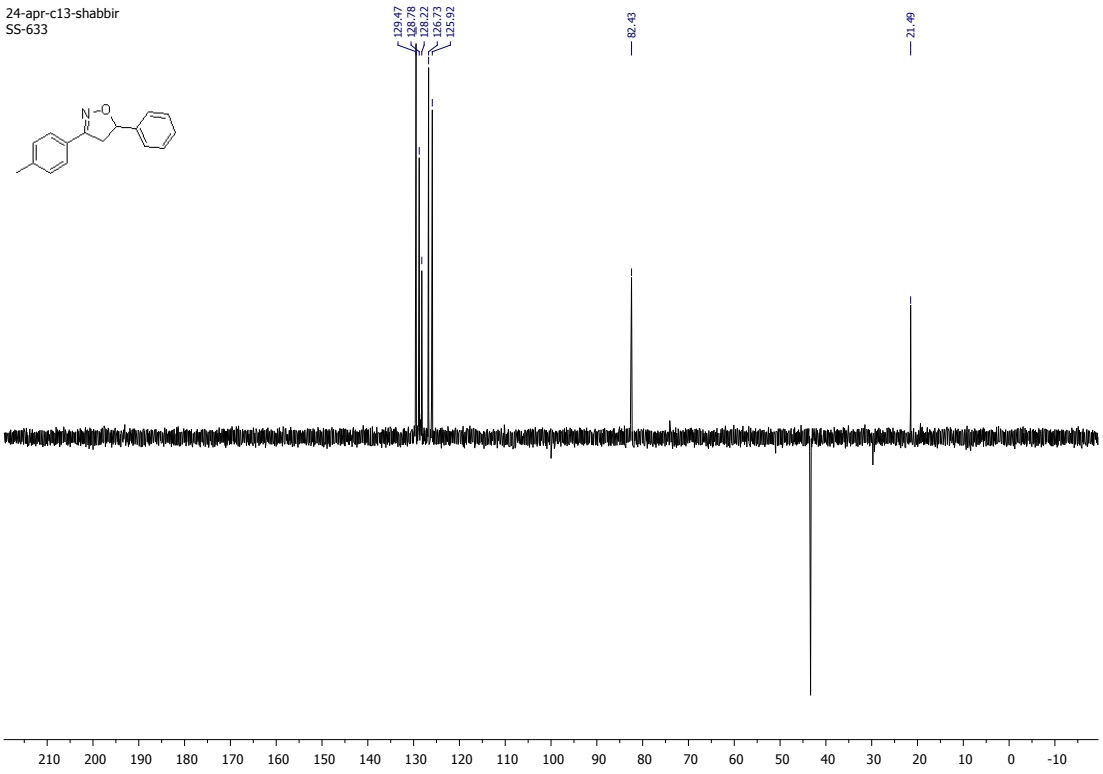

SS-456B-C13
SS-456B
128.80
128.60
128.28
125.92
115.90
82.33
HO
N
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

S3.17. ^{1}H, ^{13}C and DEPT135 NMR spectra of 3-(4-methoxyphenyl)-5-phenyl-4, 5-dihydroisoxazole (**6e**)

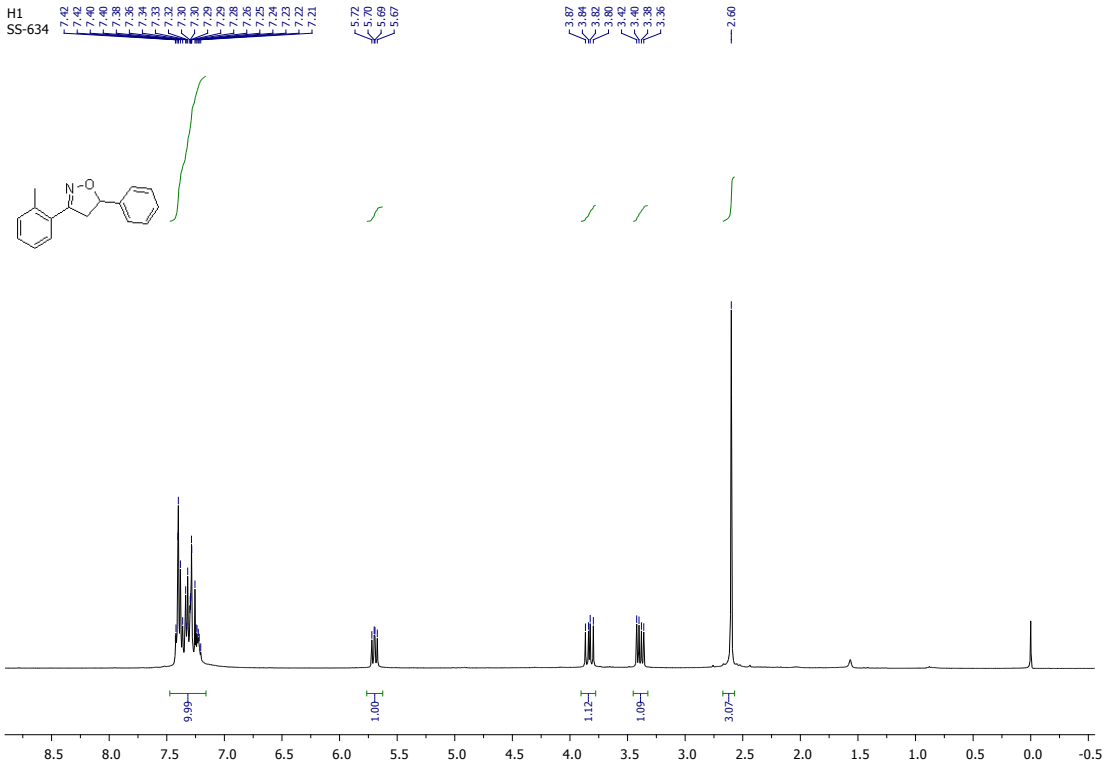


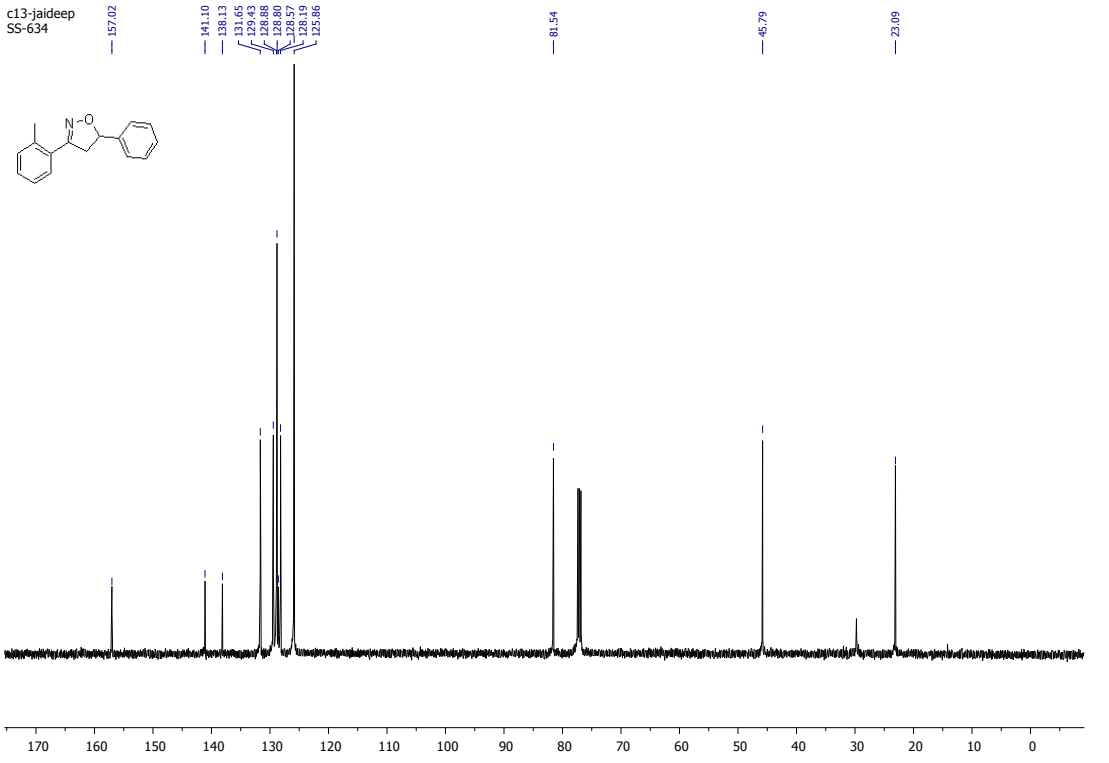
S3.18. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-phenyl-3-(p-tolyl)-4, 5-dihydroisoxazole (**6f**)

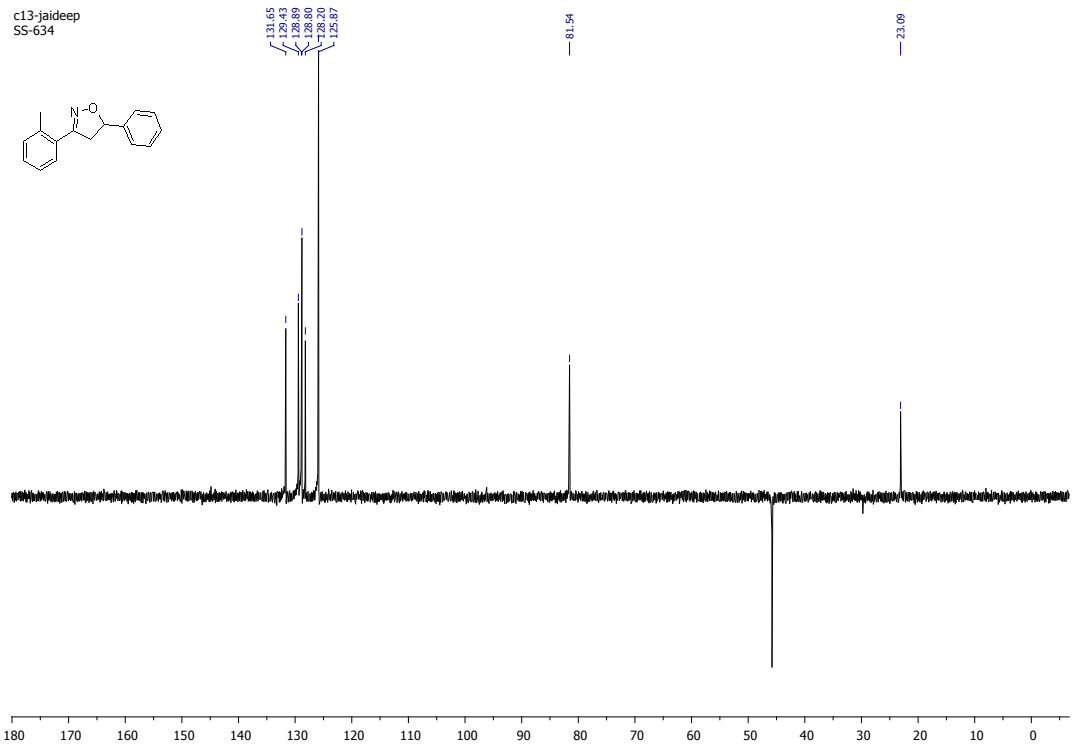



24-apr-c13-shabbir
SS-633
129.47
128.78
128.22
126.73
125.92
82.43
21.49
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

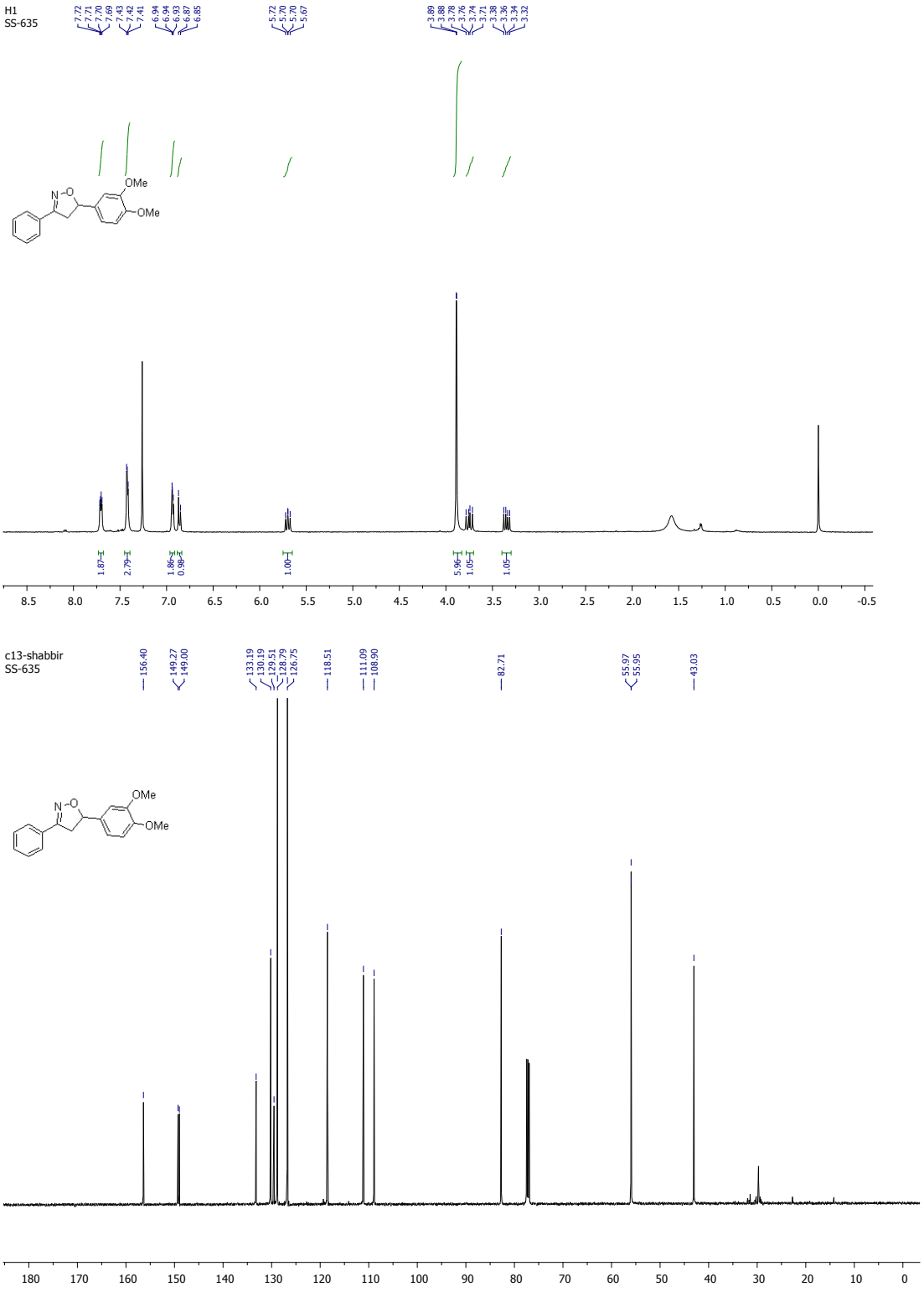
S3.19. ^{1}H, ^{13}C, and DEPT135 NMR spectra of 5-phenyl-3-(o-tolyl)-4, 5-dihydroisoxazole (**6g**)

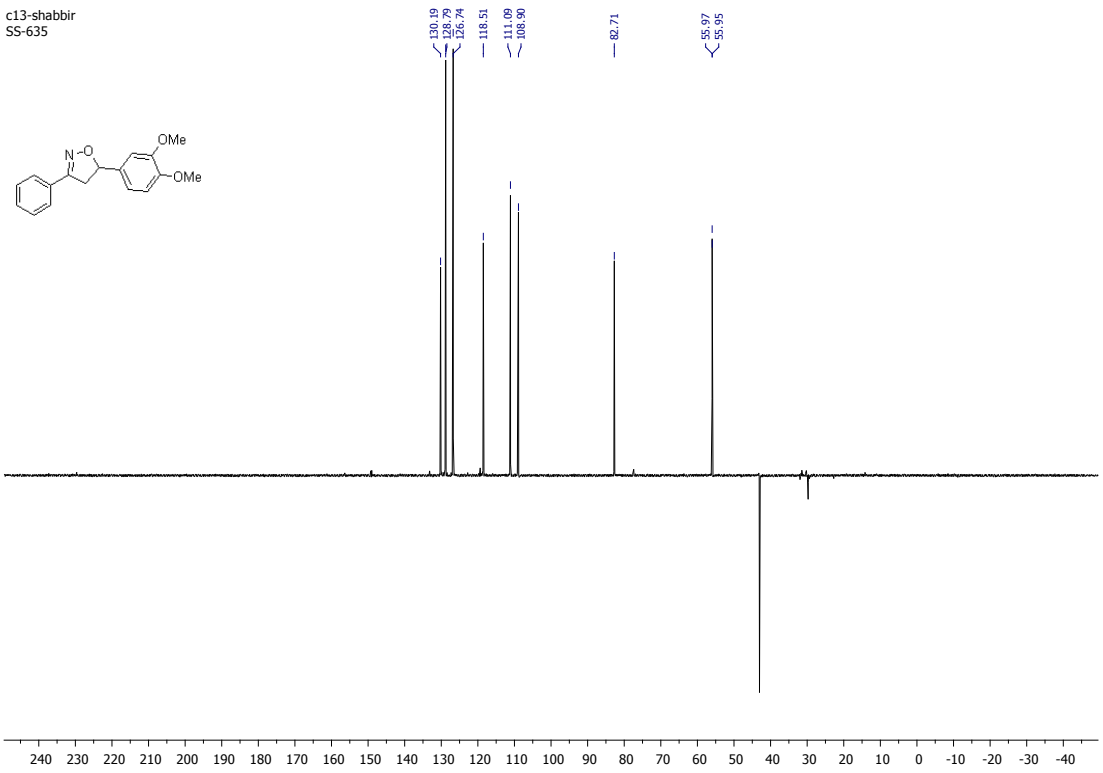




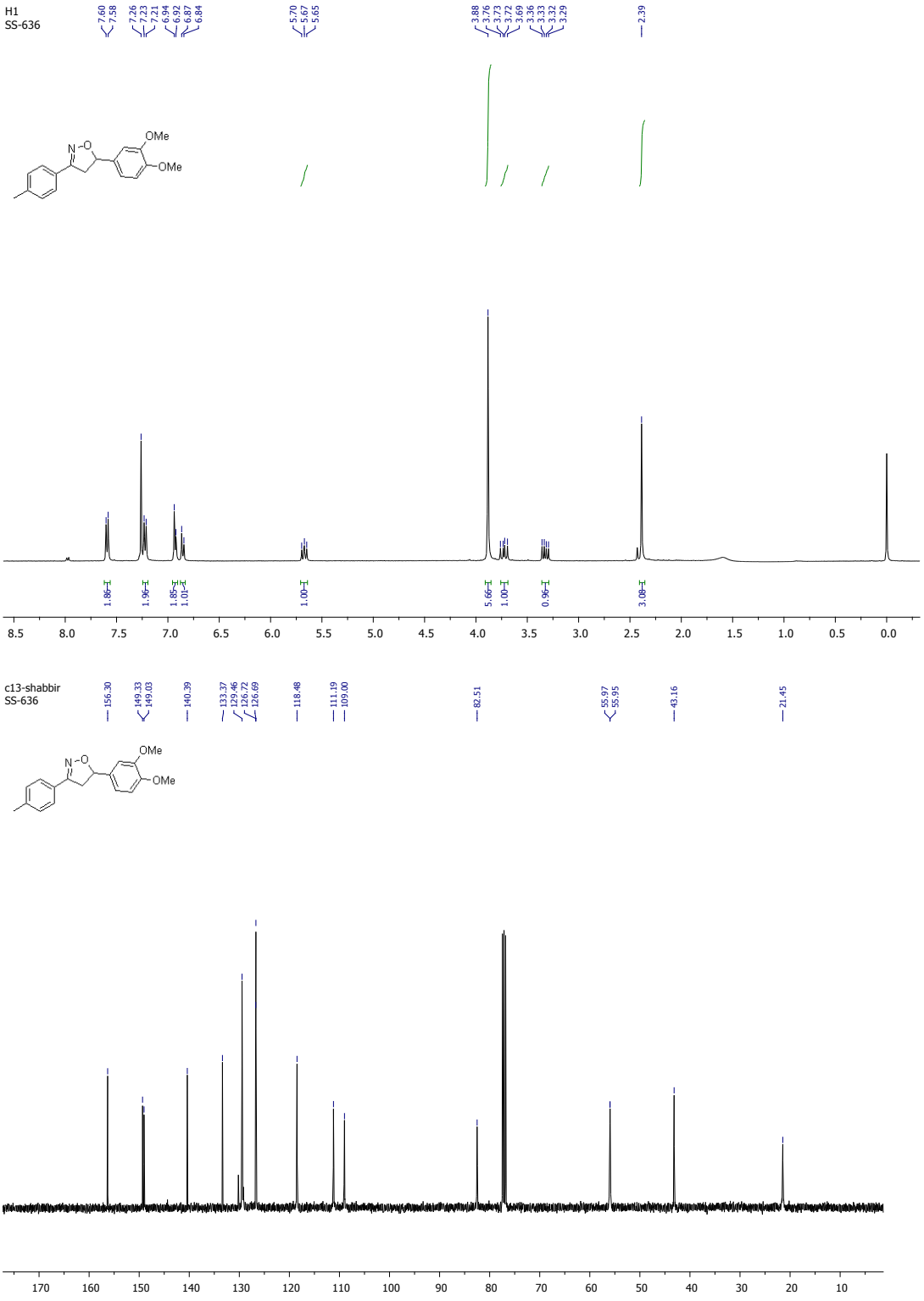

c13-jaideep
SS-634
131.65
129.43
128.89
128.80
128.20
125.87
81.54
23.09
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

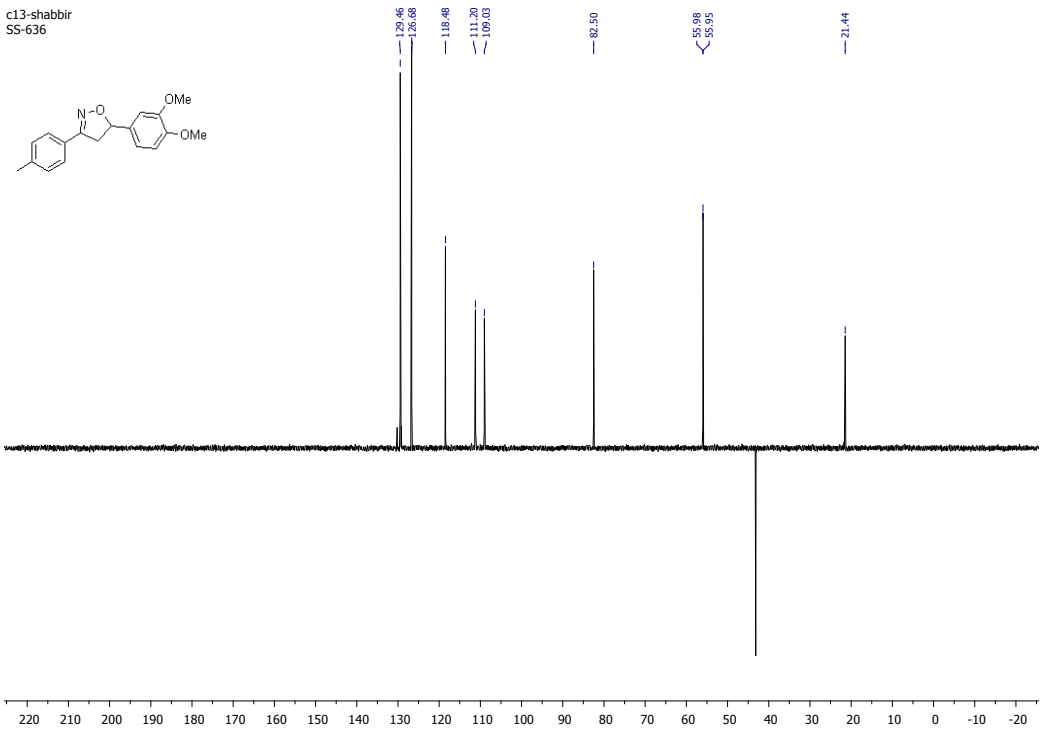
S3.20. ^{1}H, ^{13}C, and DEPT135 NMR spectra of 5-(3, 4-dimethoxyphenyl)-3-phenyl-4,5-dihydroisoxazole (**6h**)



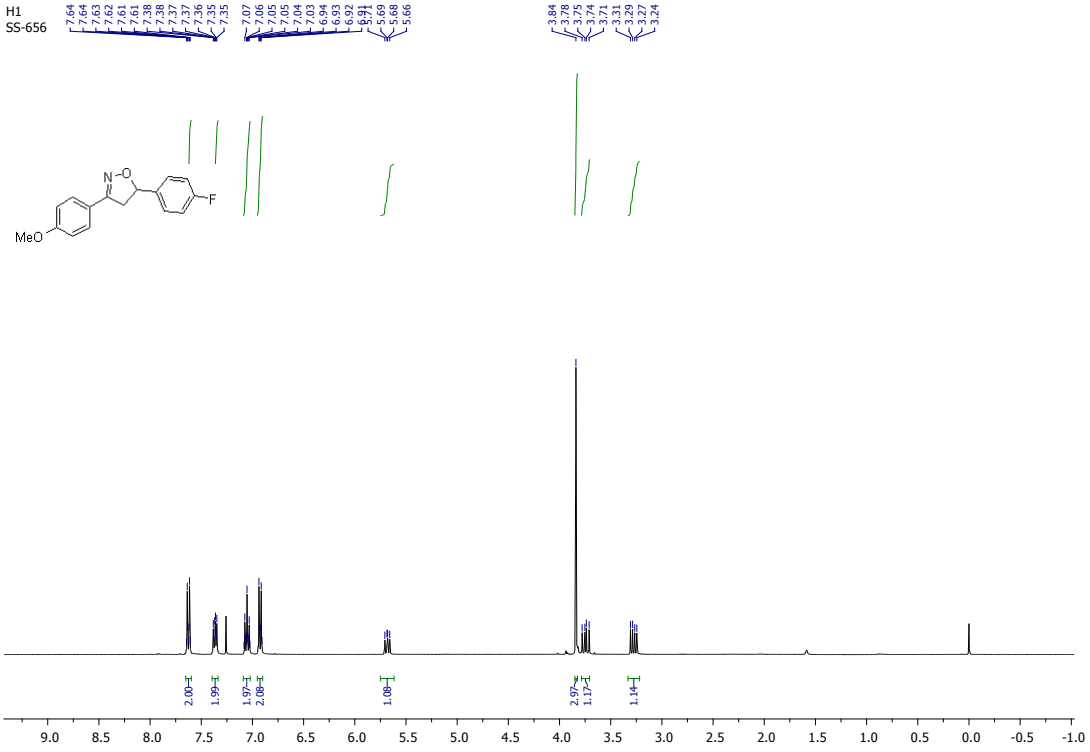

c13-shabbir
SS-635
130.19
128.79
126.74
118.51
111.09
108.90
82.71
55.97
55.95
OMe
OMe
N
O
240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40

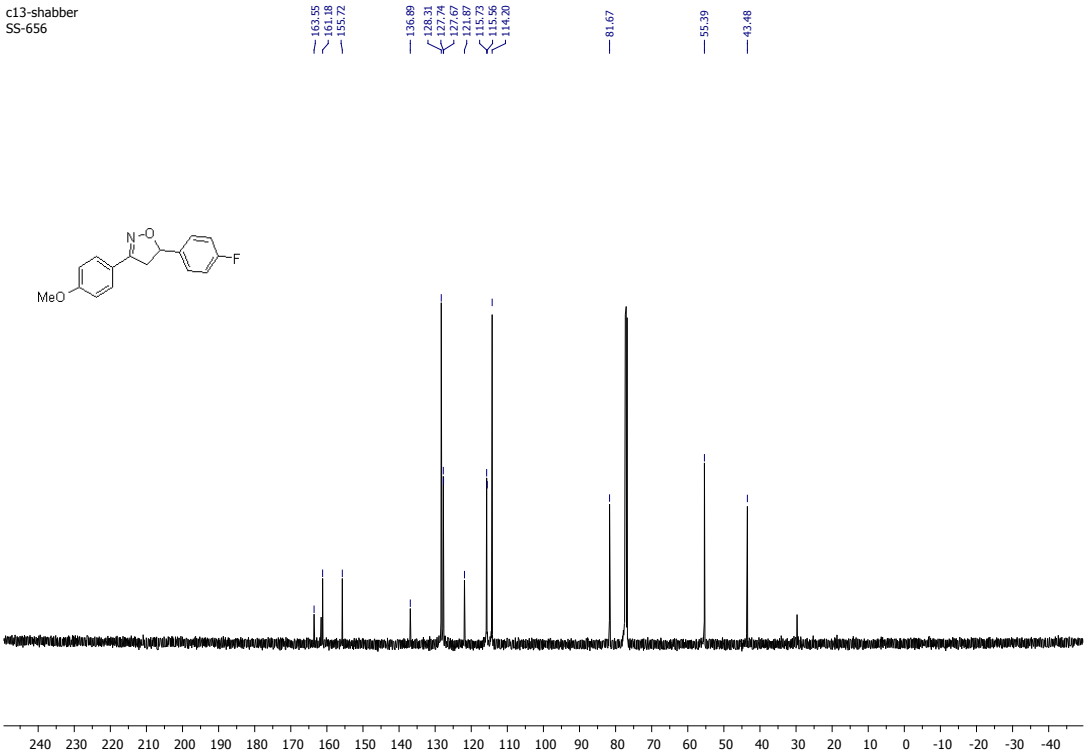
S3.21. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-(3,4-dimethoxyphenyl)-3-(p-tolyl)-4,5-dihydroisoxazole (**6i**)

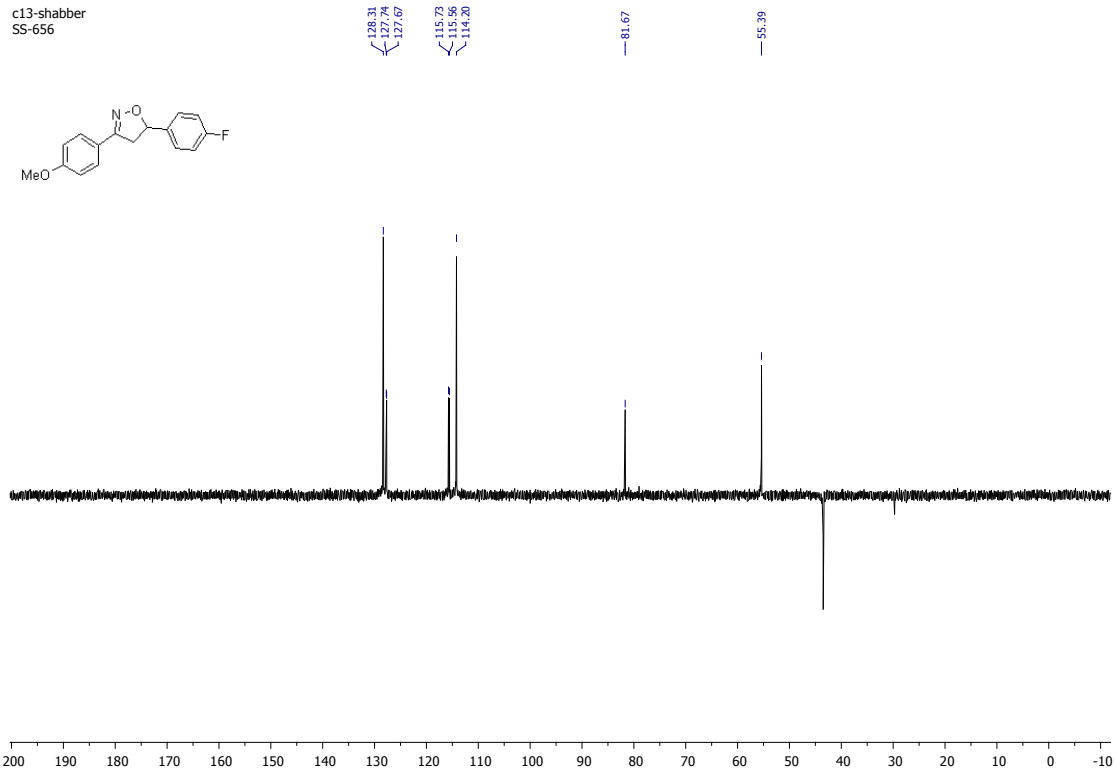



c13-shabbir
SS-636
129.46
126.68
118.48
111.20
109.03
82.50
55.98
55.95
21.44
OMe
OMe
N
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

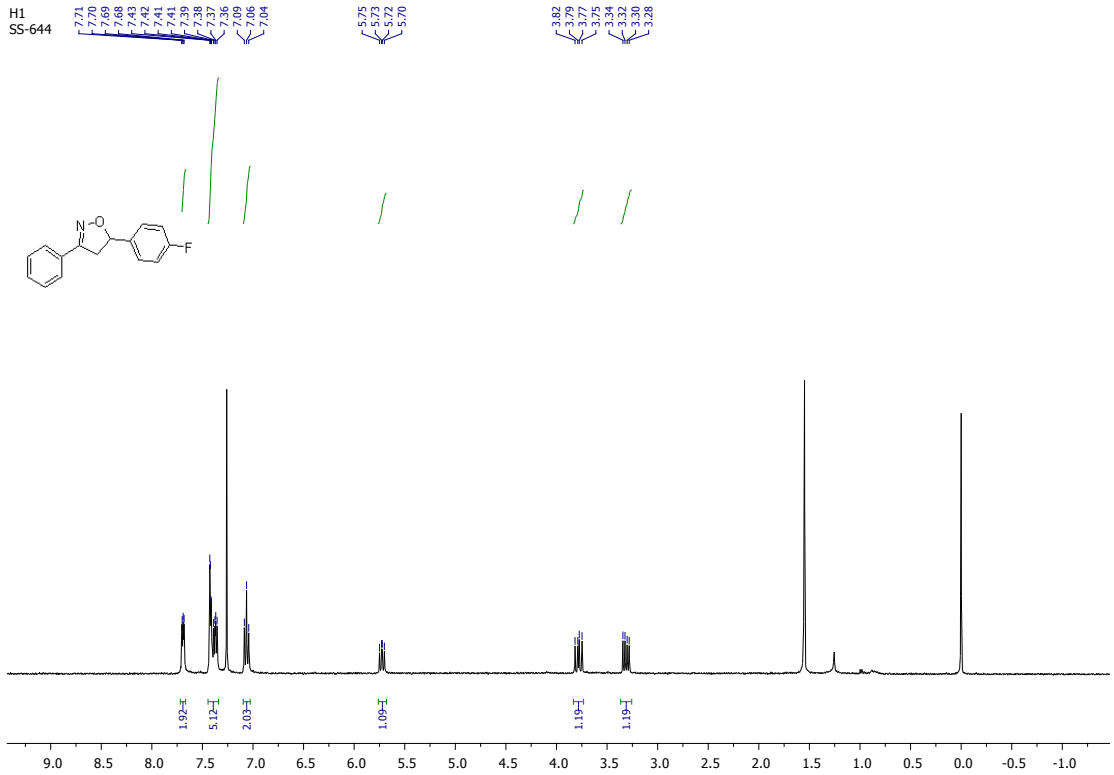
S3.22. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-(4-fluorophenyl)-3-(4-methoxyphenyl)-4, 5-dihydroisoxazole (**6j**)

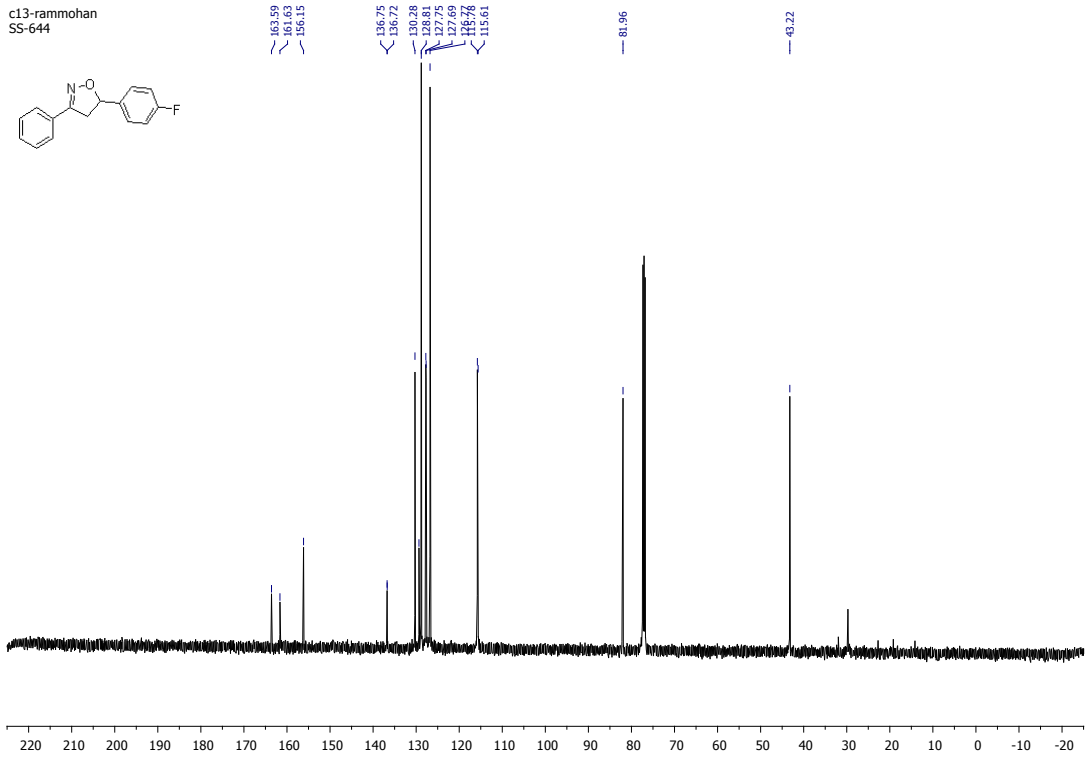


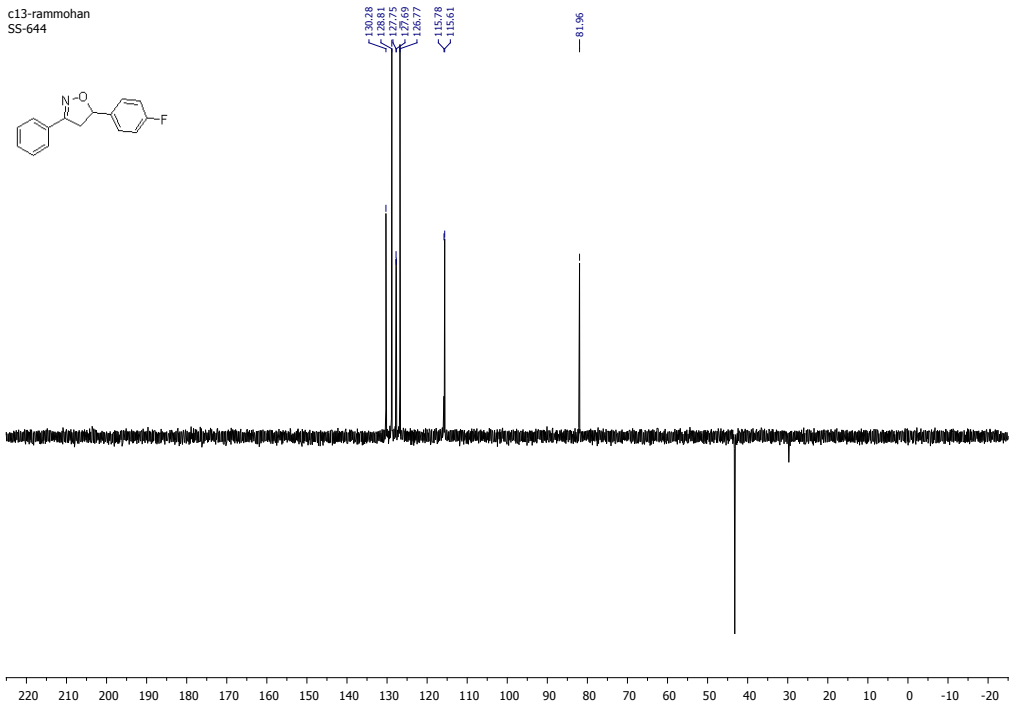



c13-shabber
SS-656
128.31
127.74
127.67
115.73
115.56
114.20
81.67
55.39
N
O
F
MeO
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

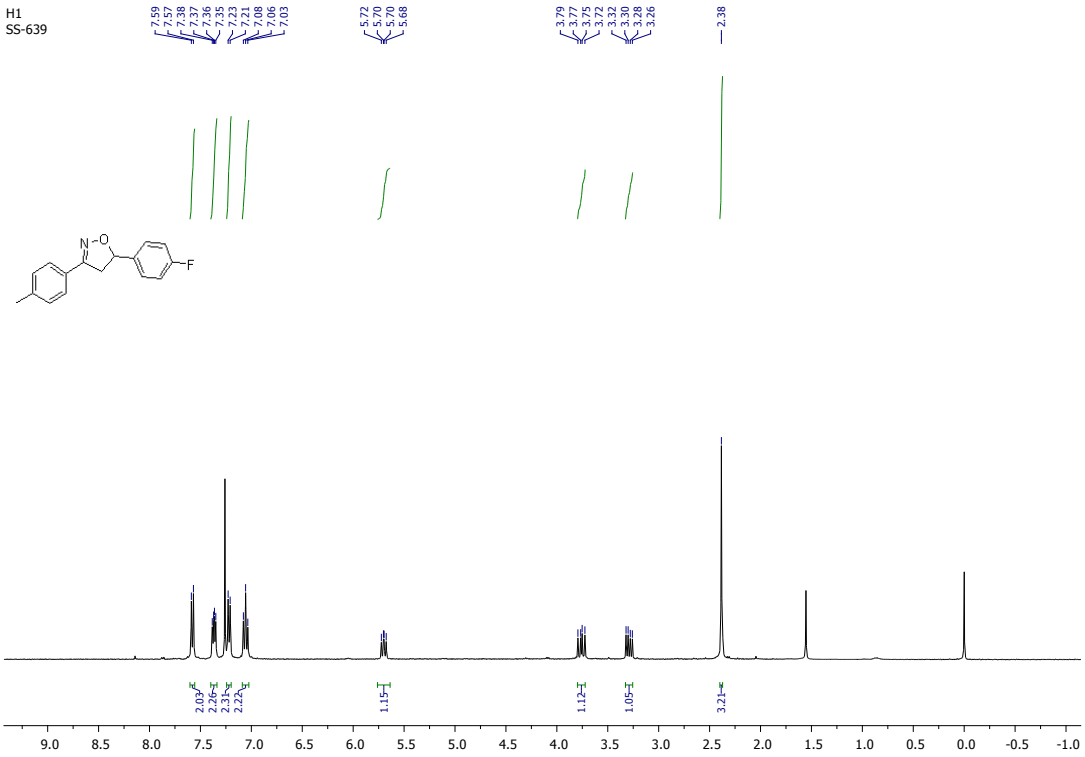
S3.23. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-(4-fluorophenyl)-3-phenyl-4, 5-dihydroisoxazole (**6k**)

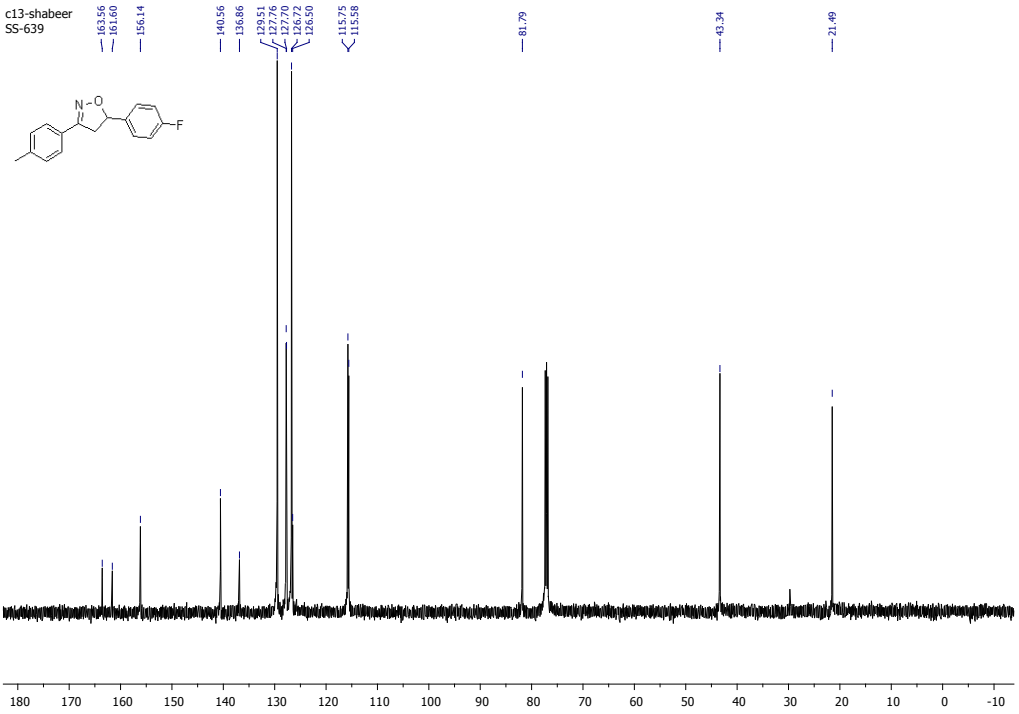


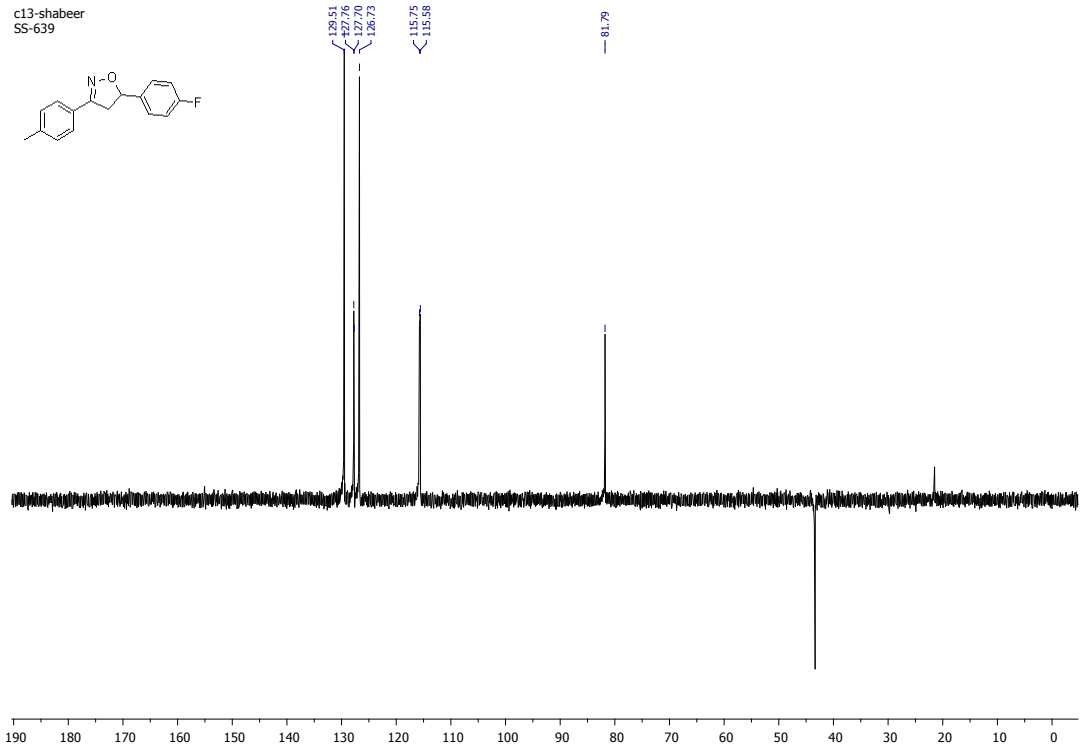



c13-rammohan
SS-644
130.28
128.81
127.55
127.49
126.77
115.78
115.61
81.96
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

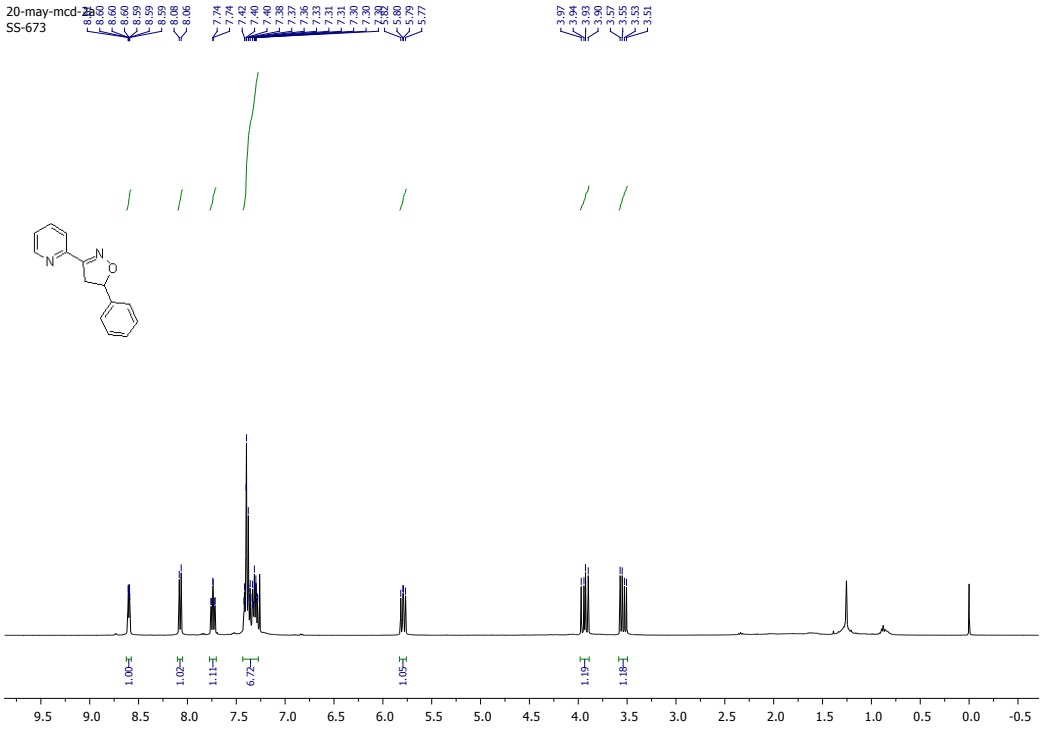
S3.24. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-(4-fluorophenyl)-3-(p-tolyl)-4, 5-dihydroisoxazole (**6l**)

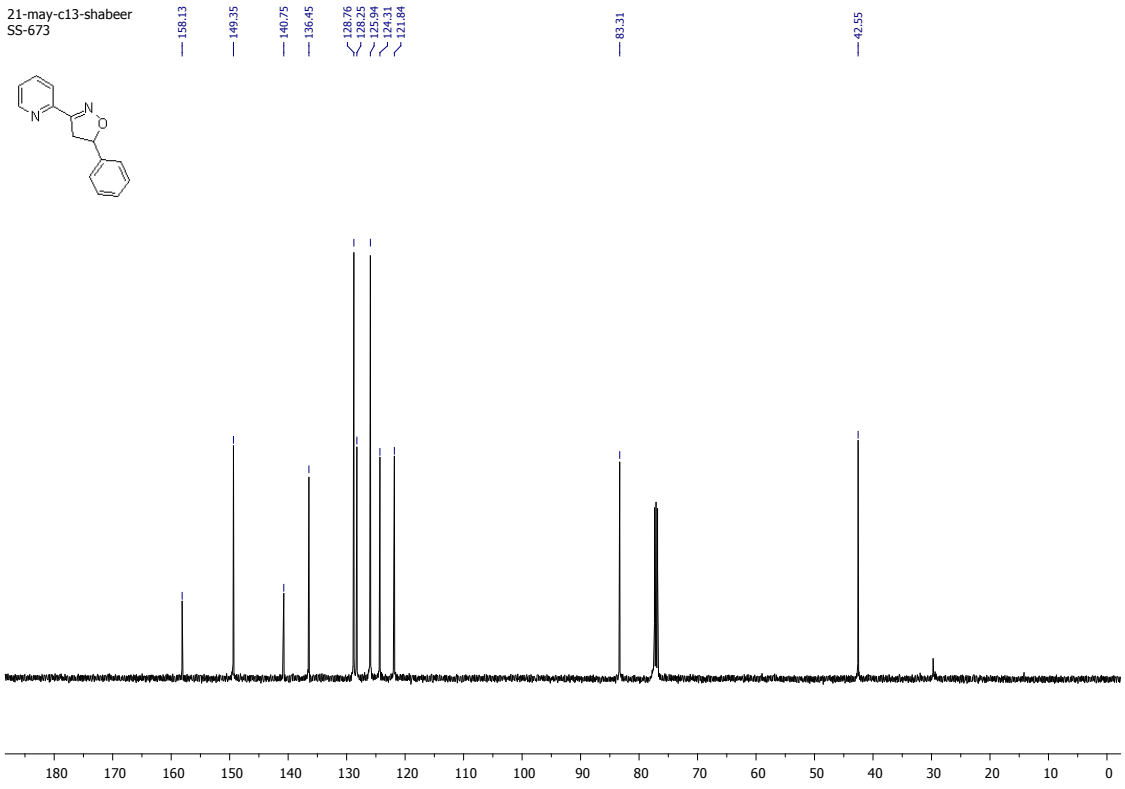


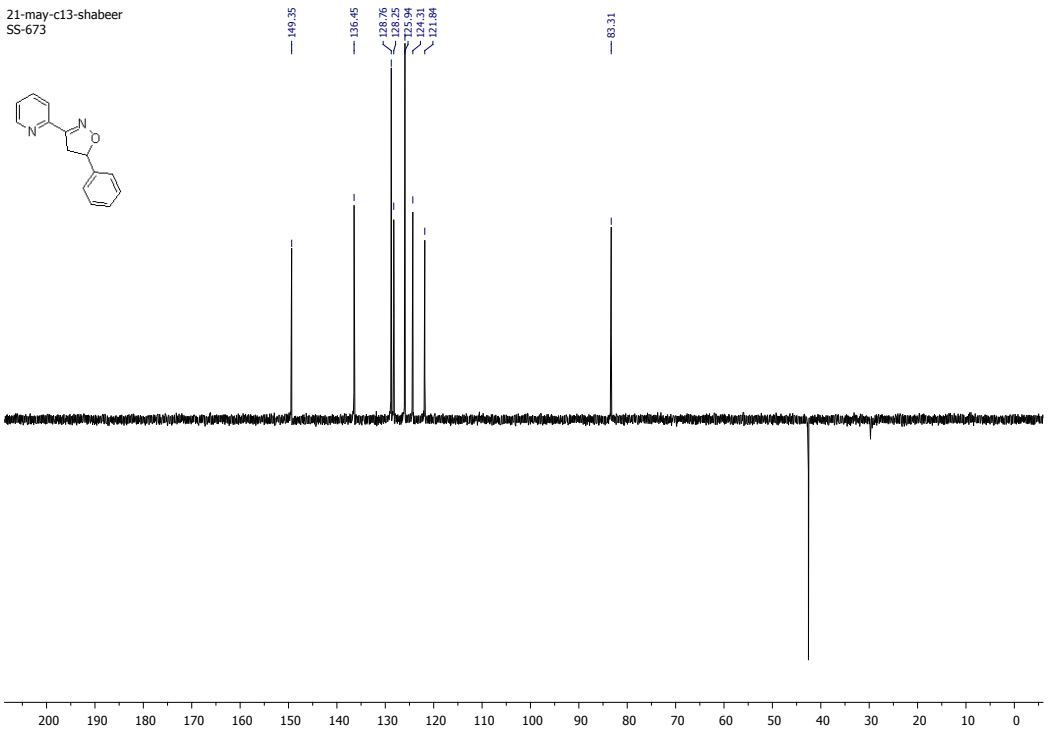



c13-shabeer
SS-639
129.51
127.76
127.70
126.73
115.75
115.58
81.79
N
O
F
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

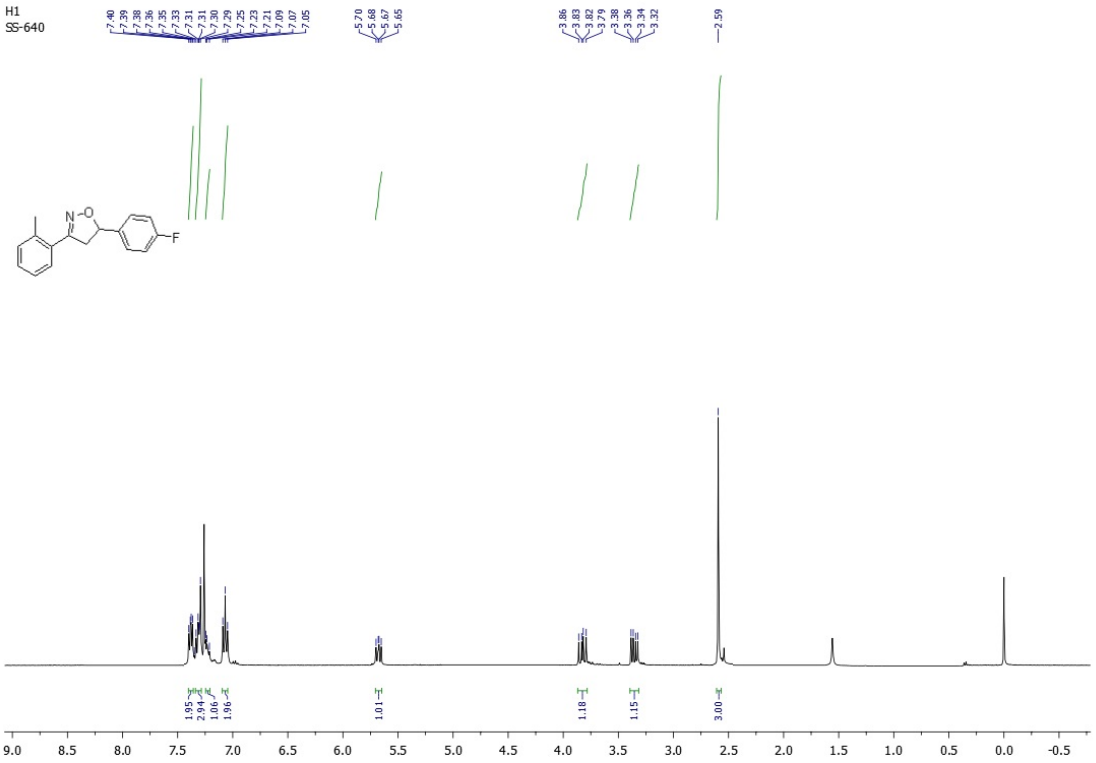
S3.25. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-phenyl-3-(pyridine-2-yl)-4, 5-dihydroisoxazole (**6m**)

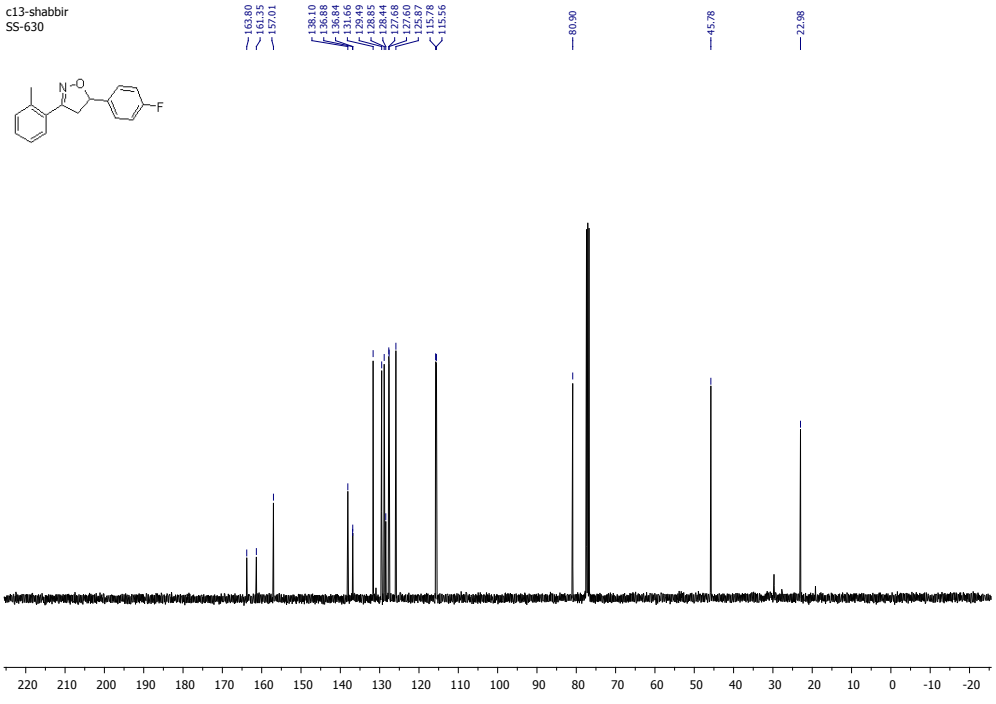


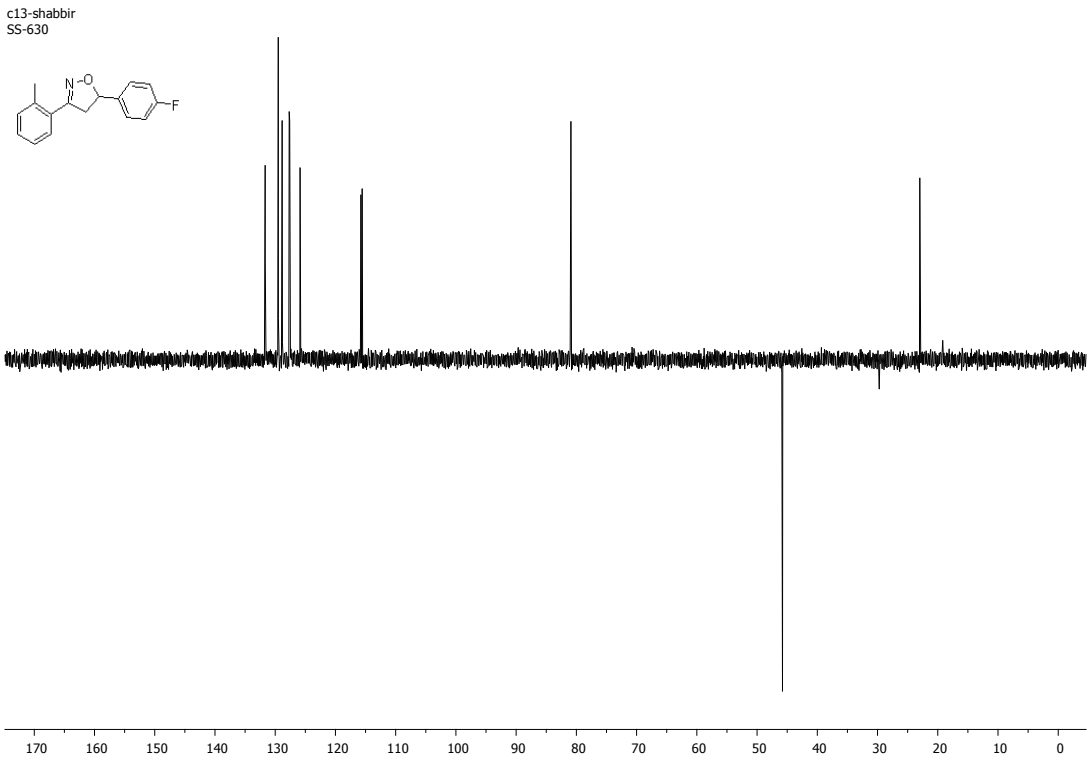



21-may-c13-shabeer
SS-673
149.35
136.45
128.76
128.25
125.94
124.31
121.84
83.31
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

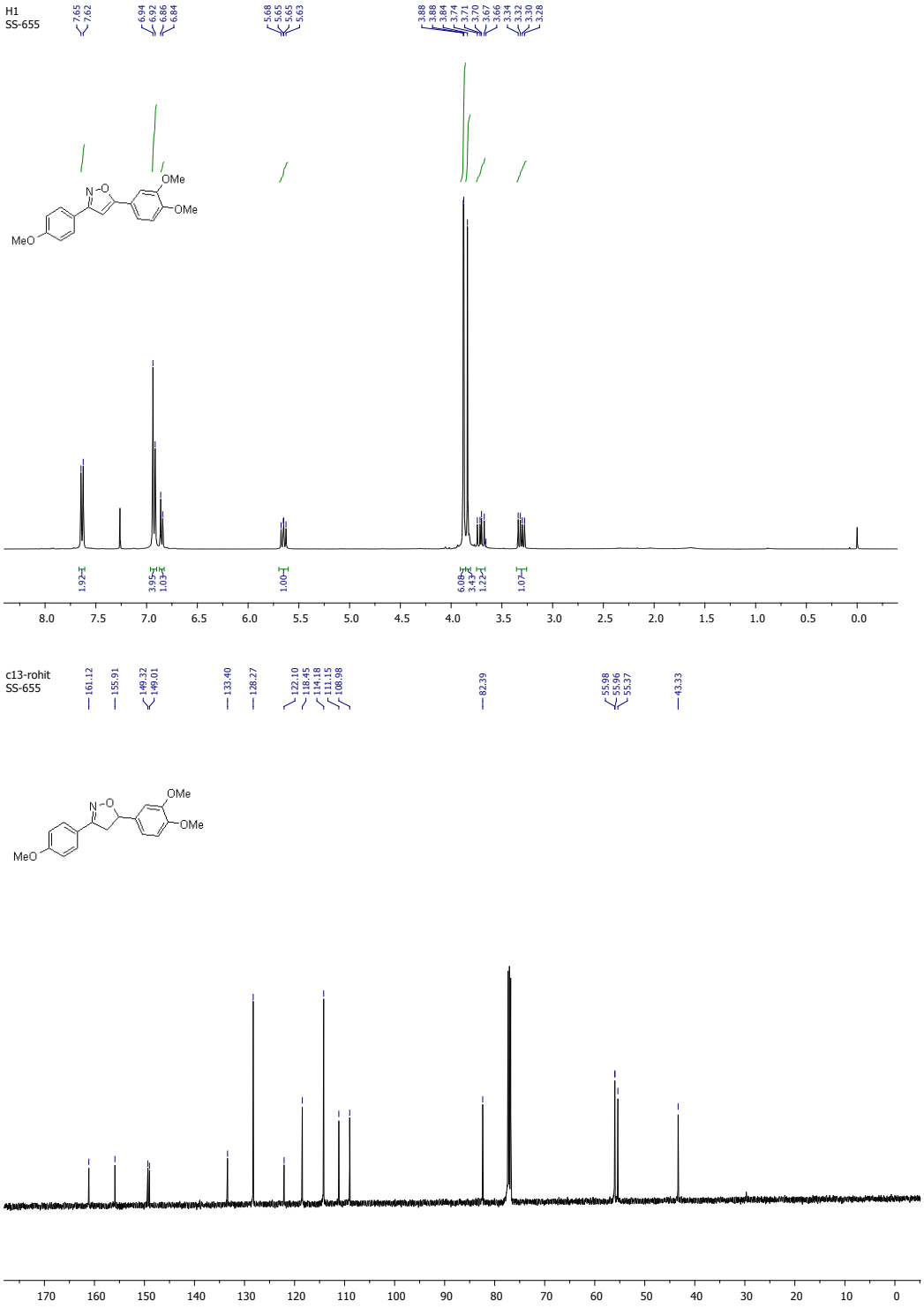
S3.26. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-(4-fluorophenyl)-3-(o-tolyl)-4, 5-dihydroisoxazole (**6n**)

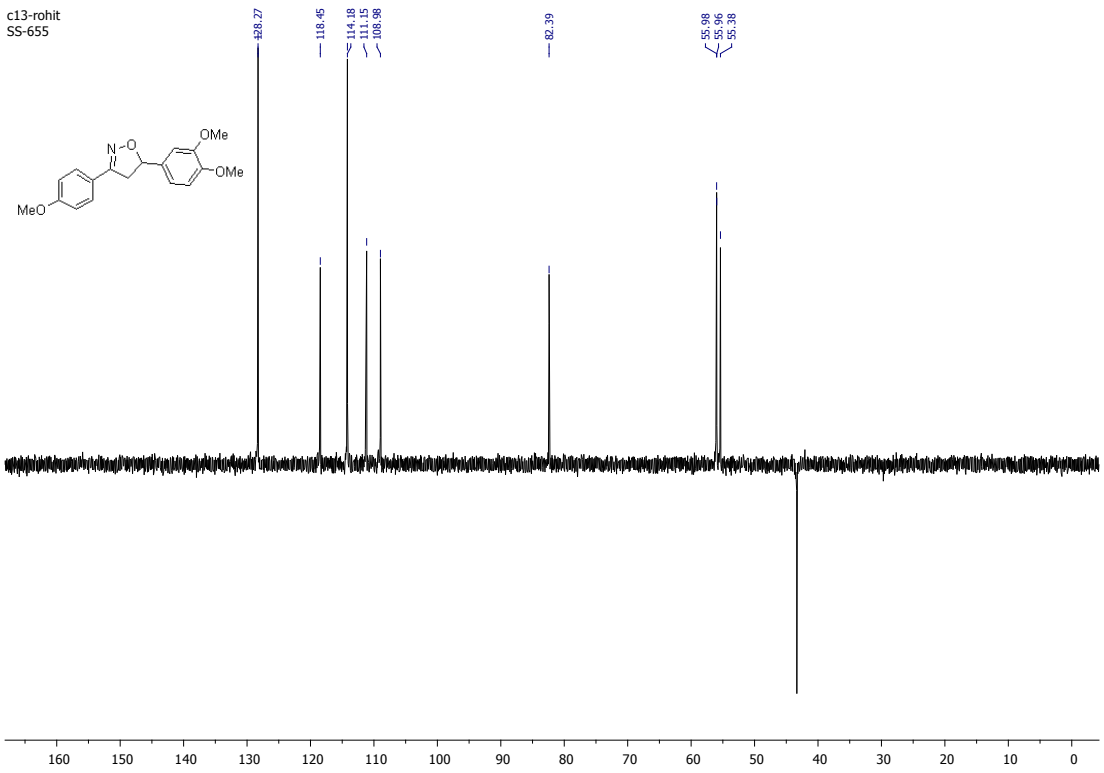




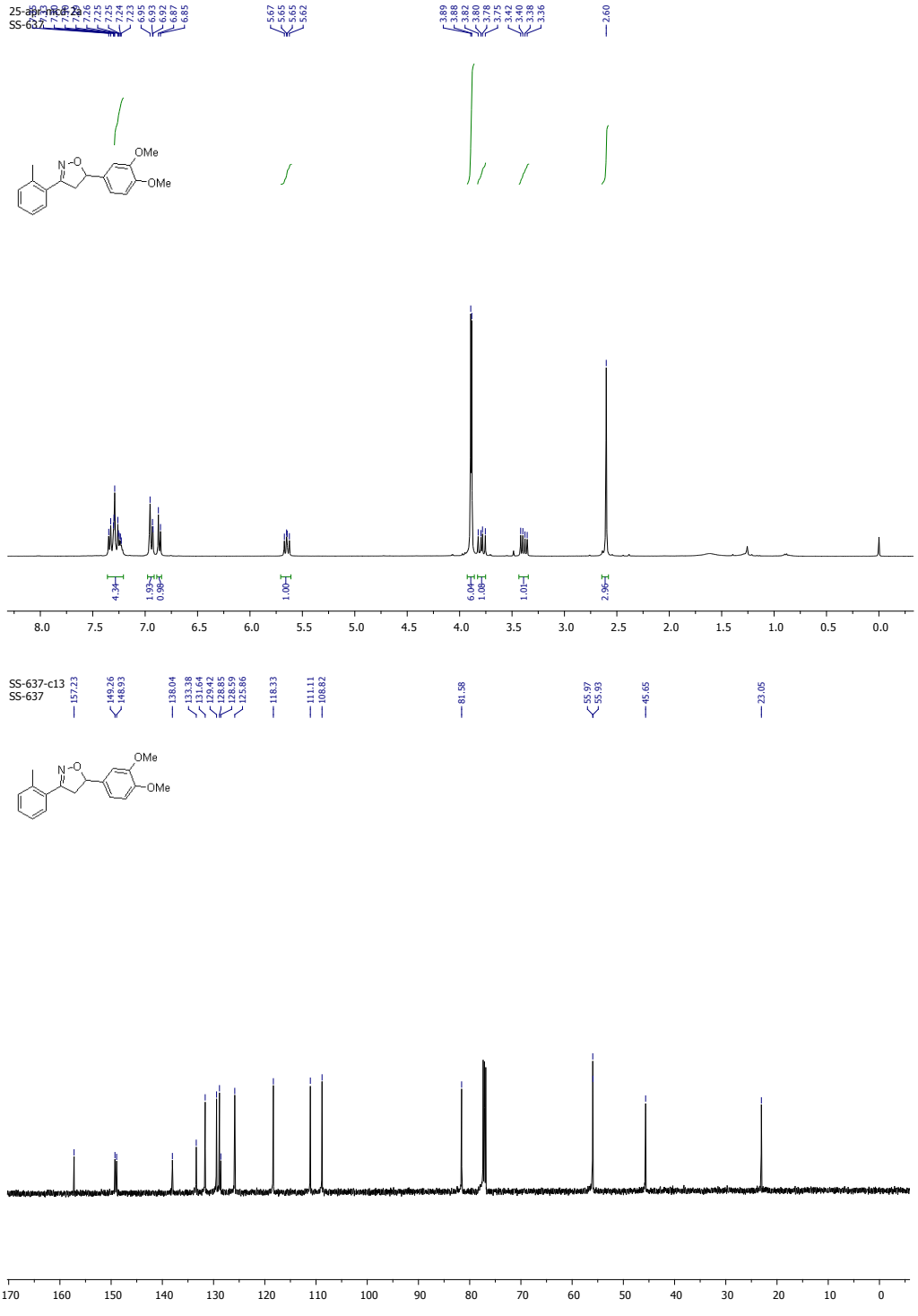

c13-shabbir
SS-630
N
O
F
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

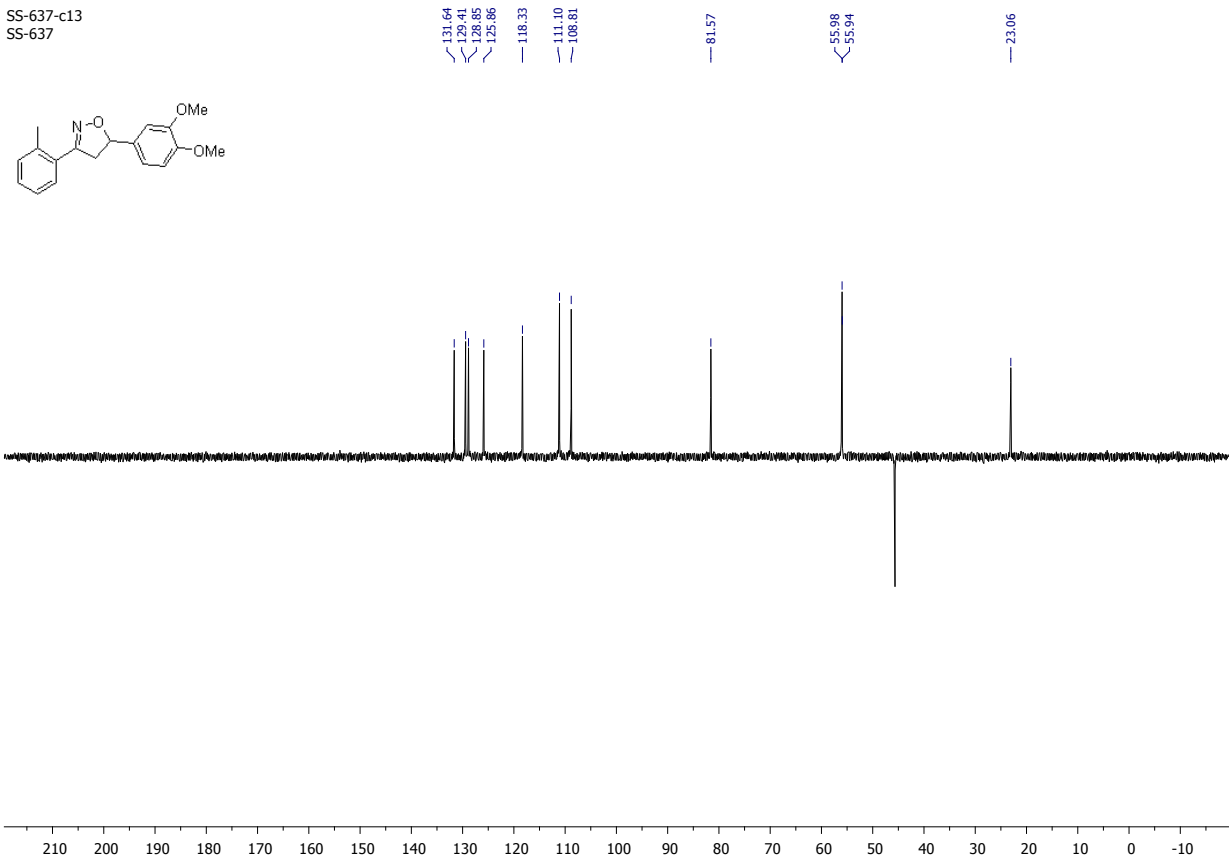
S3.27. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-(3, 4-dimethoxyphenyl)-3-(4-methoxyphenyl) isoxazole (**6o**)



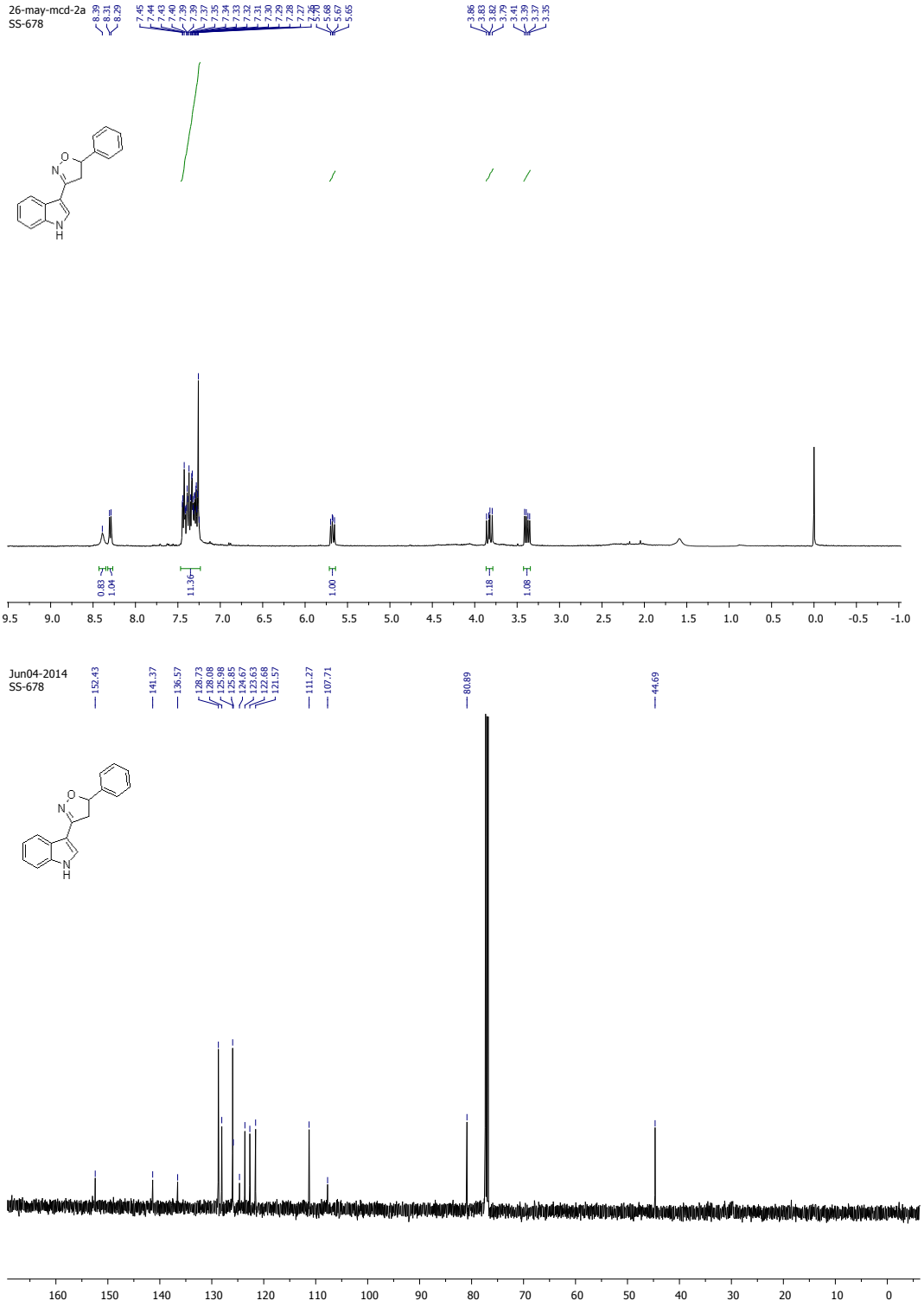

c13-rohit
SS-655
128.27
118.45
114.18
111.15
108.98
82.39
55.98
55.96
55.38
OMe
OMe
MeO
N
O
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

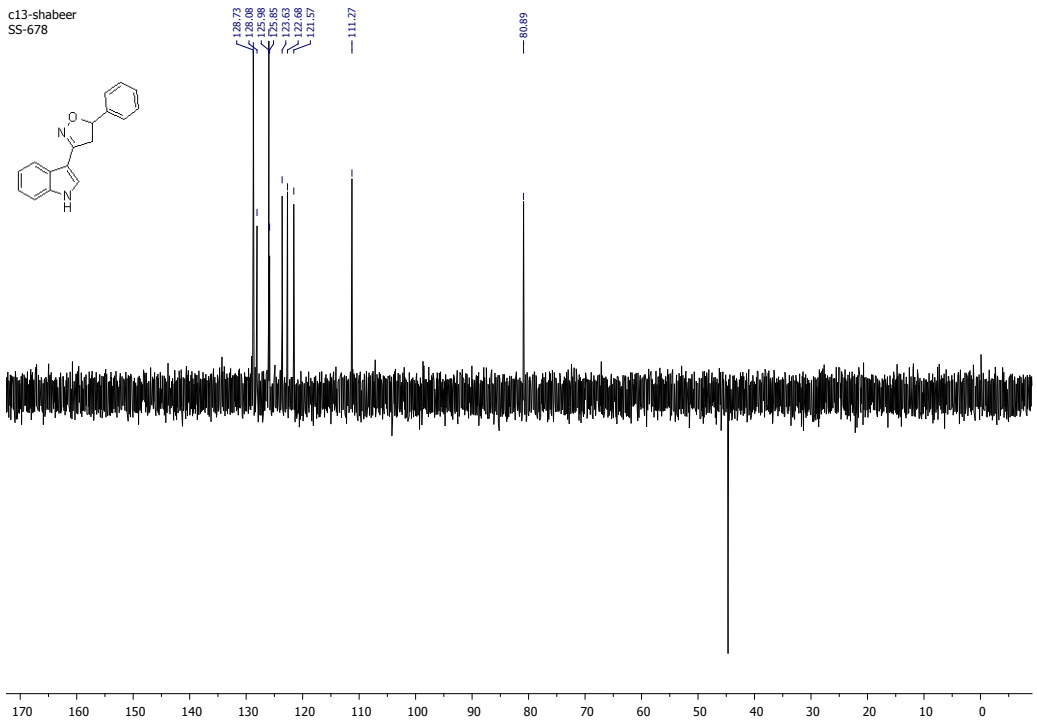
S3.28. ^{1}H, ^{13}C and DEPT135 NMR spectra of 5-(3, 4-dimethoxyphenyl)-3-(o-tolyl)-4,5-dihydroisoxazole (**6p**)



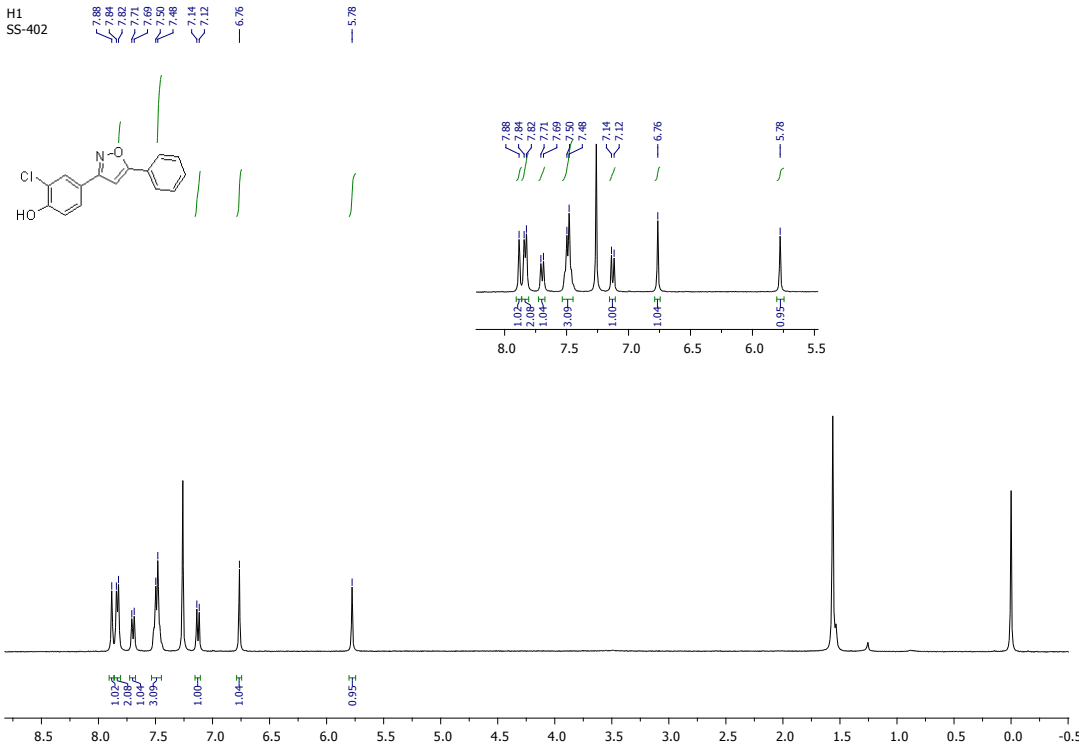

SS-637-c13
SS-637
131.64
129.41
128.85
125.86
118.33
111.10
108.81
81.57
55.98
55.94
23.06
OMe
OMe
N
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

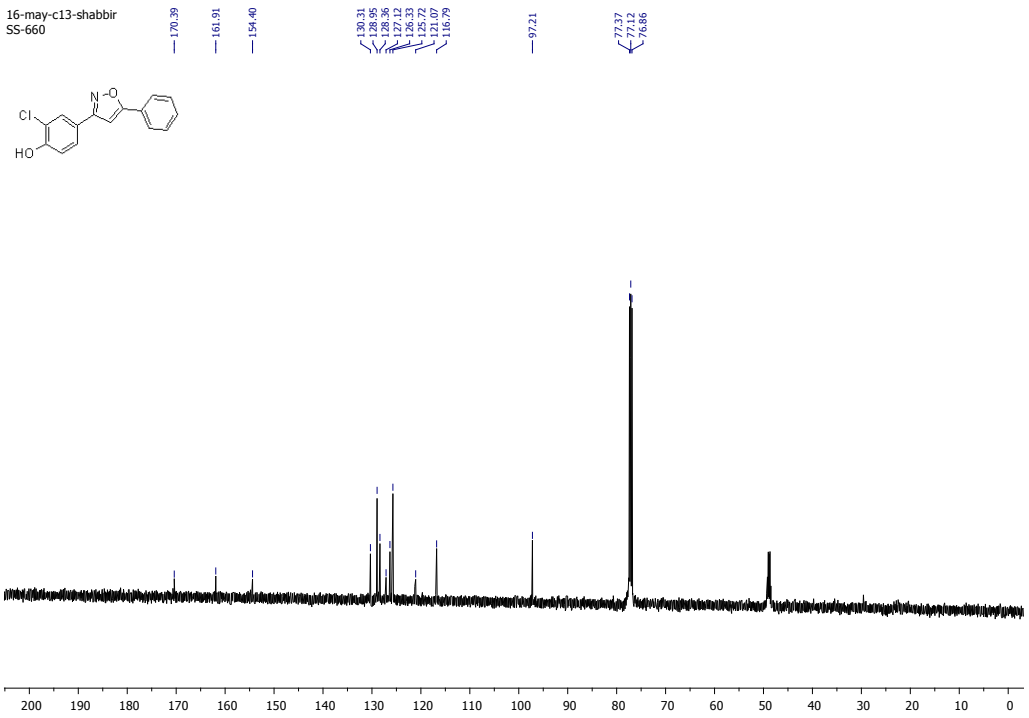
S3.29. ^{1}H, ^{13}C and DEPT135 NMR spectra of 3-(1H-indol-3yl)-5-phenyl-4, 5-dihydroisoxazole (**6q**)



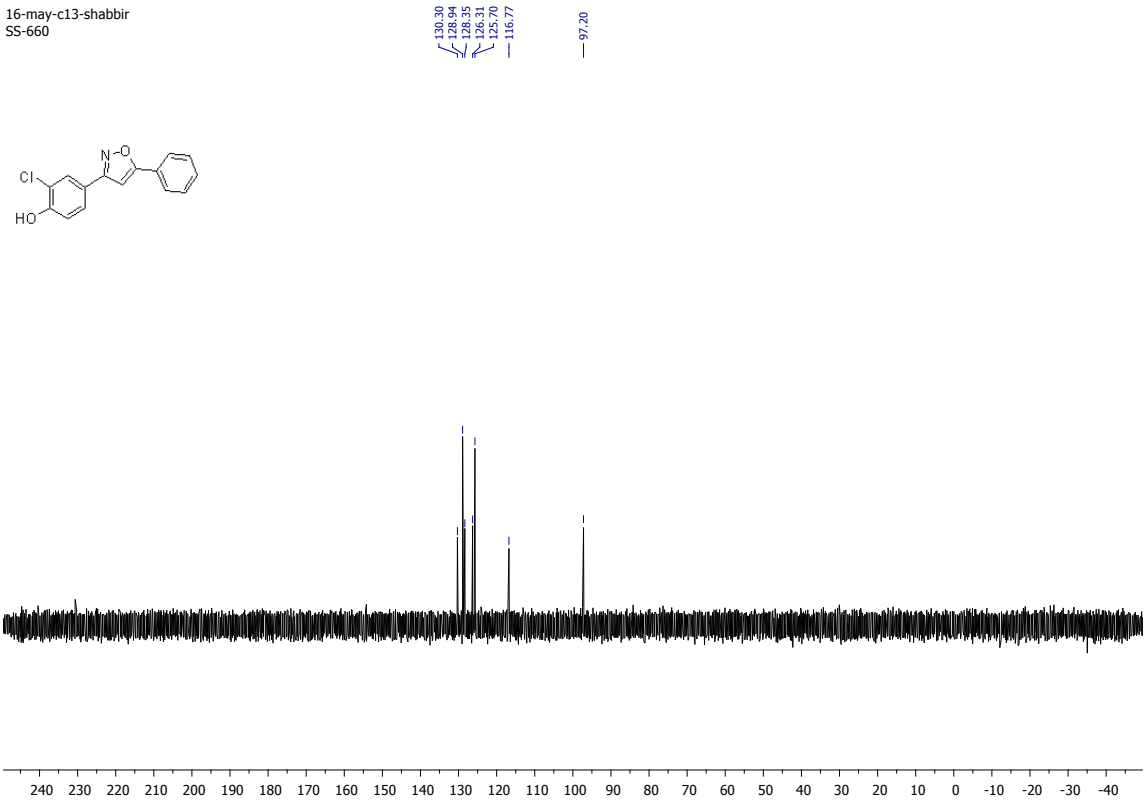

c13-shabeer
SS-678
128.73
128.08
125.98
125.85
123.63
122.68
121.57
111.27
80.89
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

S3.30. ^{1}H, ^{13}C and DEPT135 NMR spectra of 2-chloro-4-(5-phenylisoxazole-3-yl) phenol (**7a**)

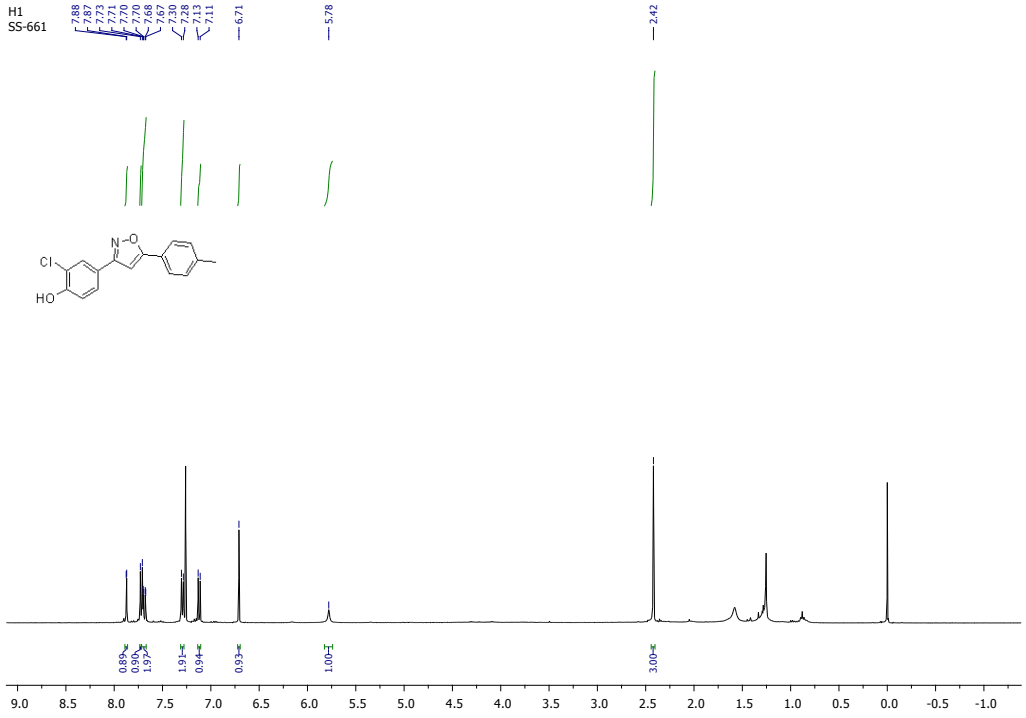


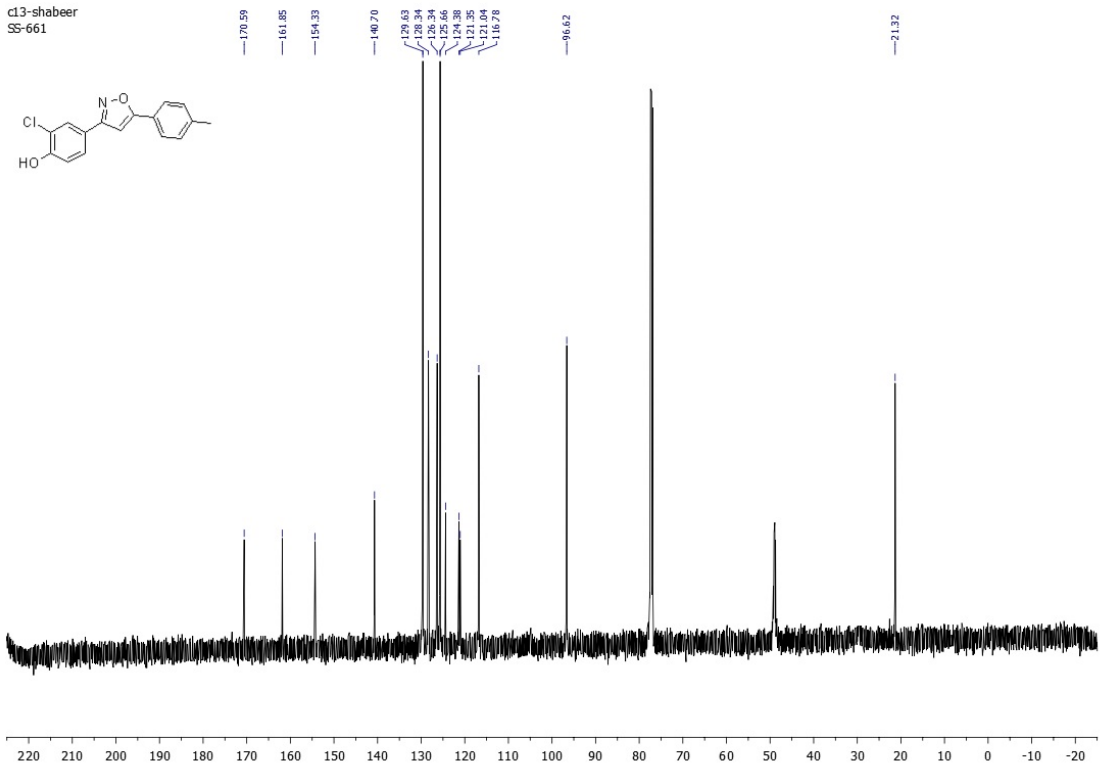


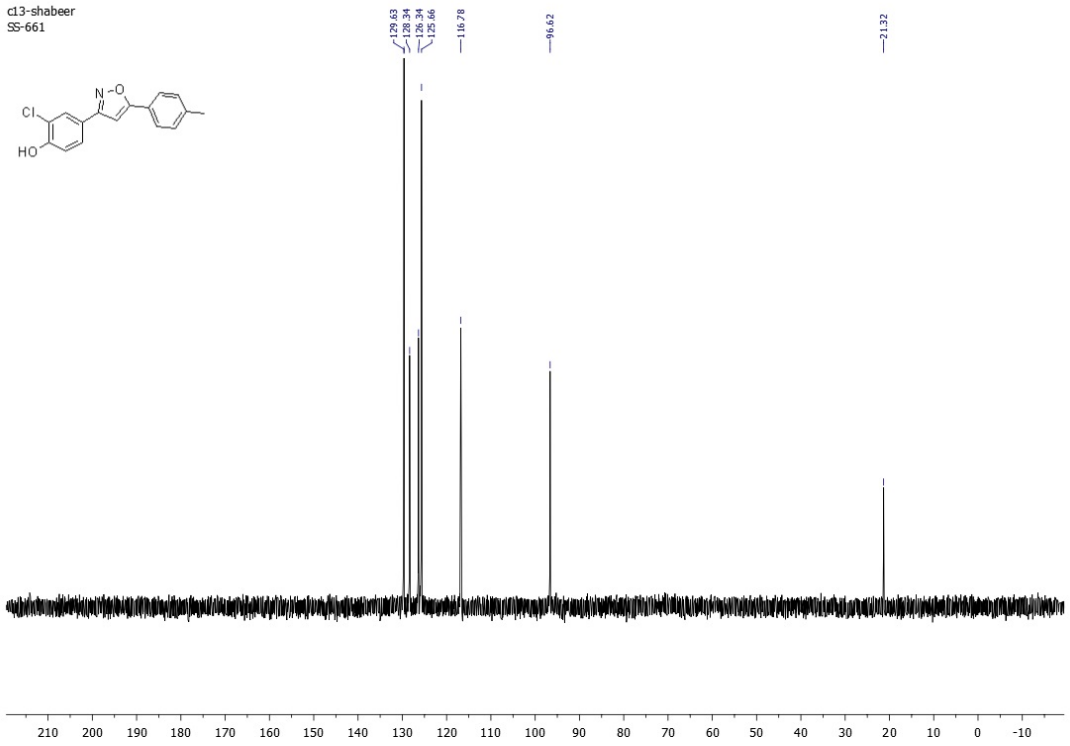
16-may-c13-shabbir
SS-660



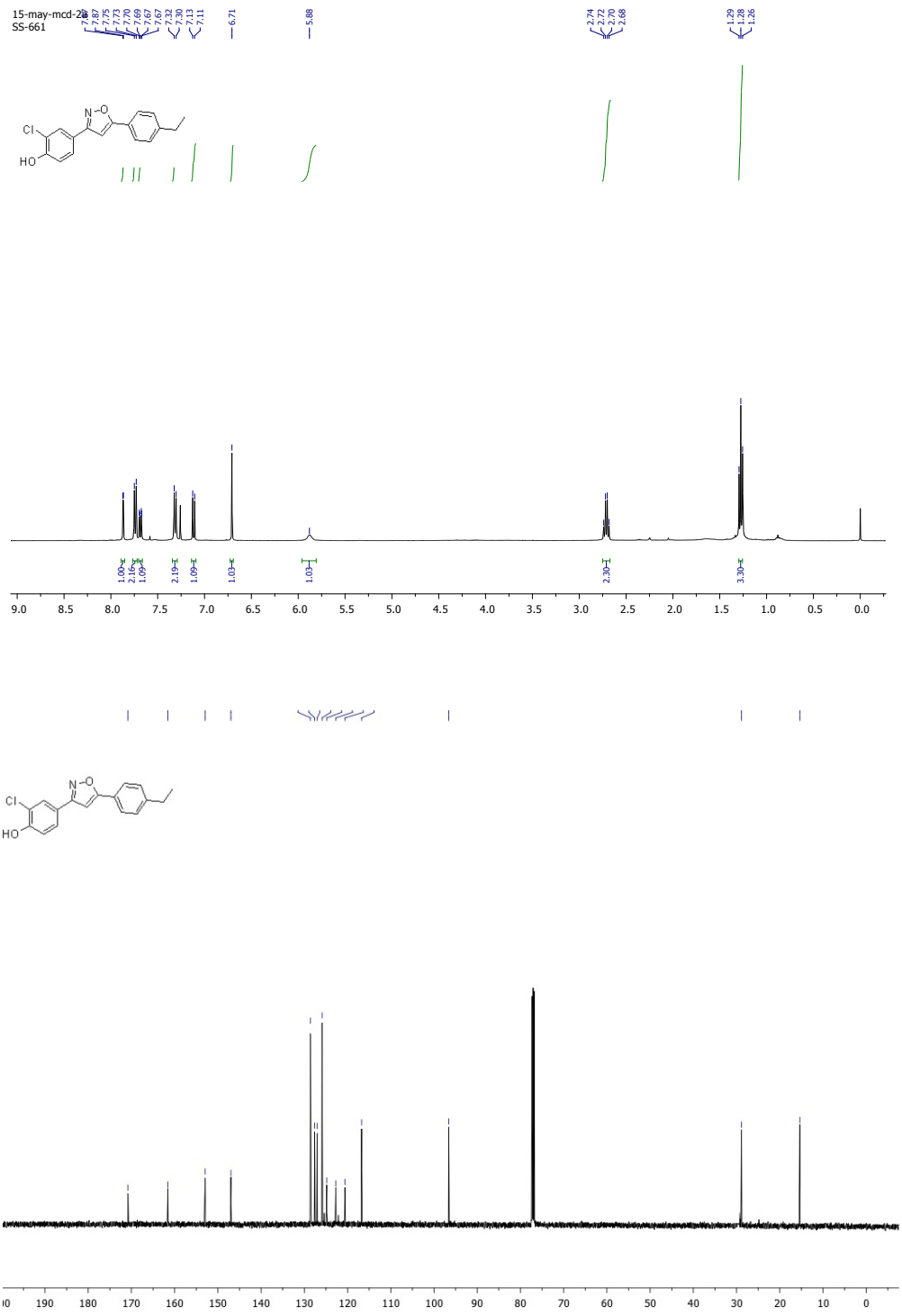
S3.31. ^{1}H, ^{13}C and DEPT135 NMR spectra of 2-chloro-4-(5-(p-tolyl) isoxazole-3-yl)phenol (**7b**)

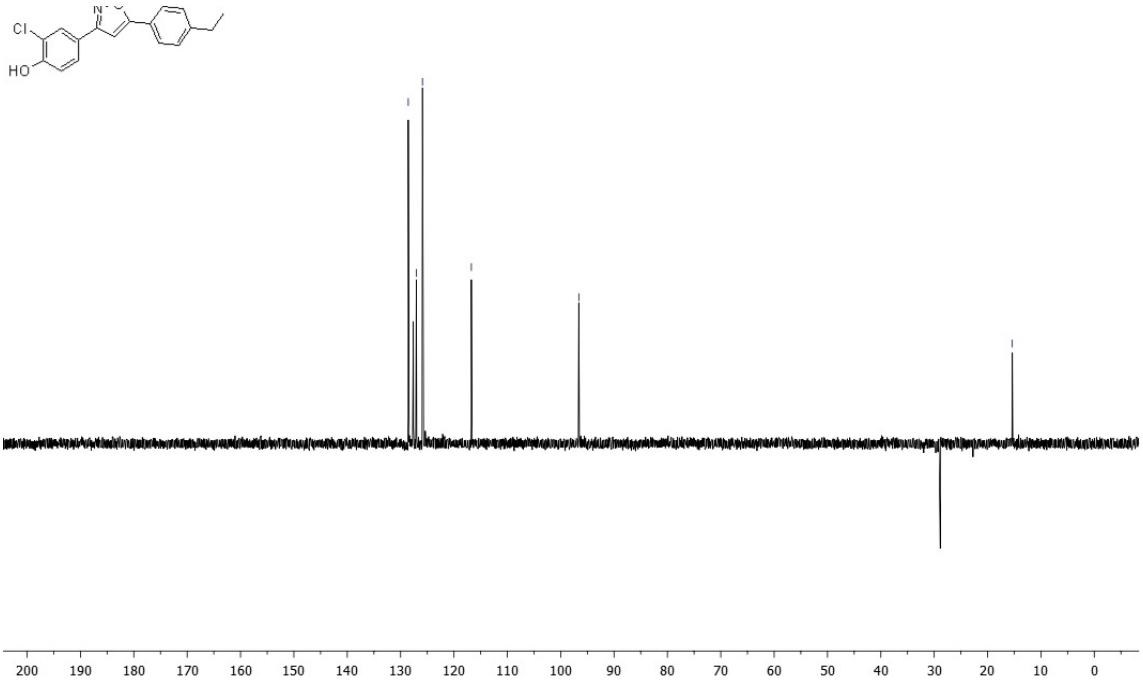




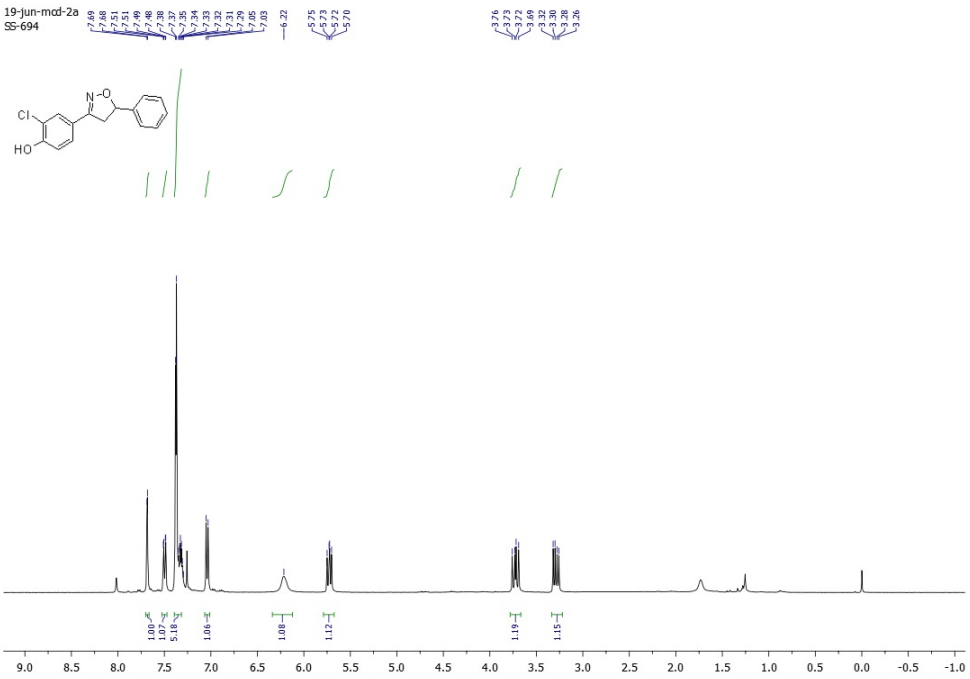

c13-shabeer
SS-661
129.63
128.34
126.34
125.66
116.78
96.62
21.32
Cl
HO
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

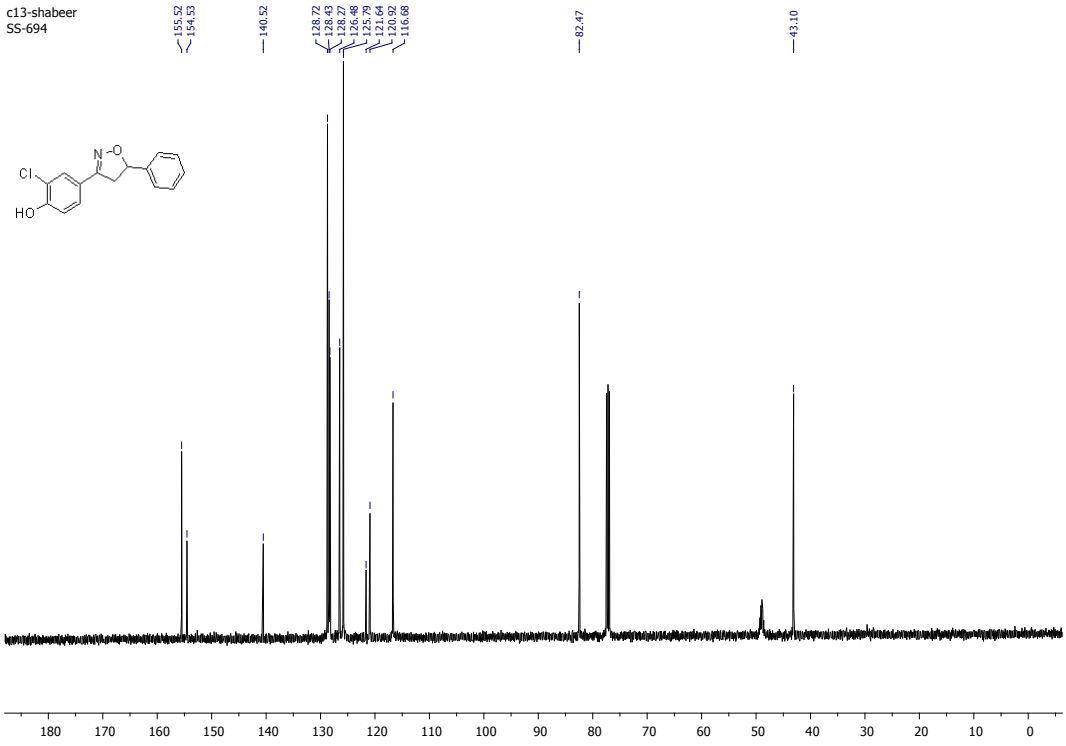
S3.32. ^{1}H, ^{13}C and DEPT135 NMR spectra of 2-chloro-4-(5-(4-ethylphenyl) isoxazole-3-yl) phenol (**7c**)

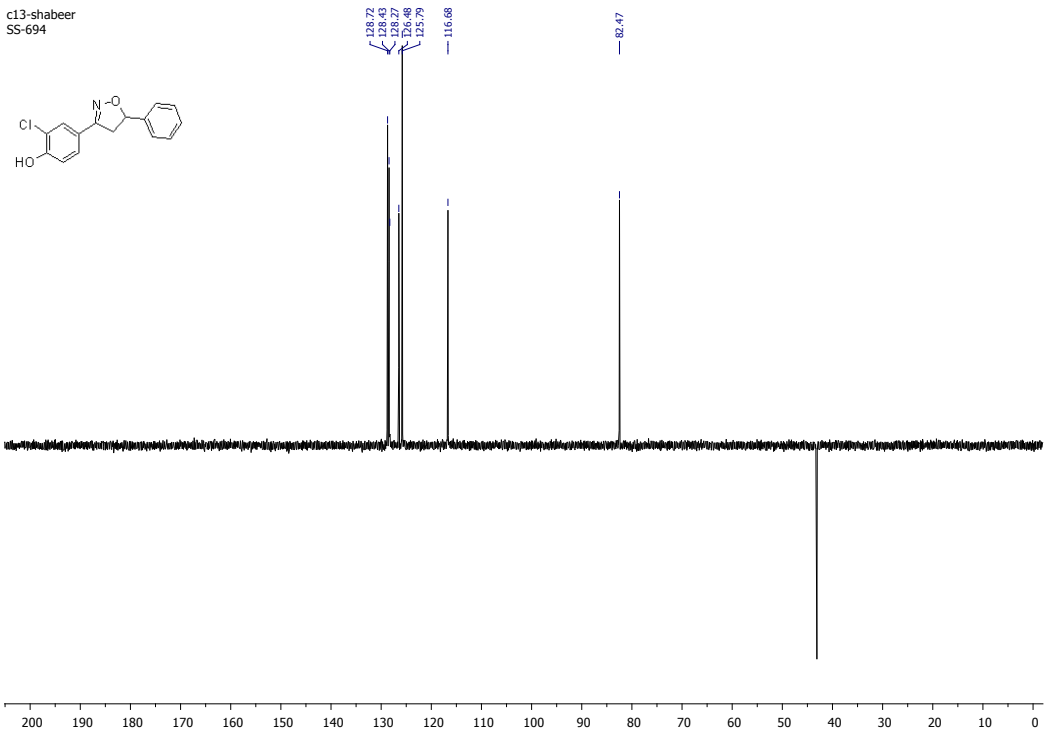



Cl
HO
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

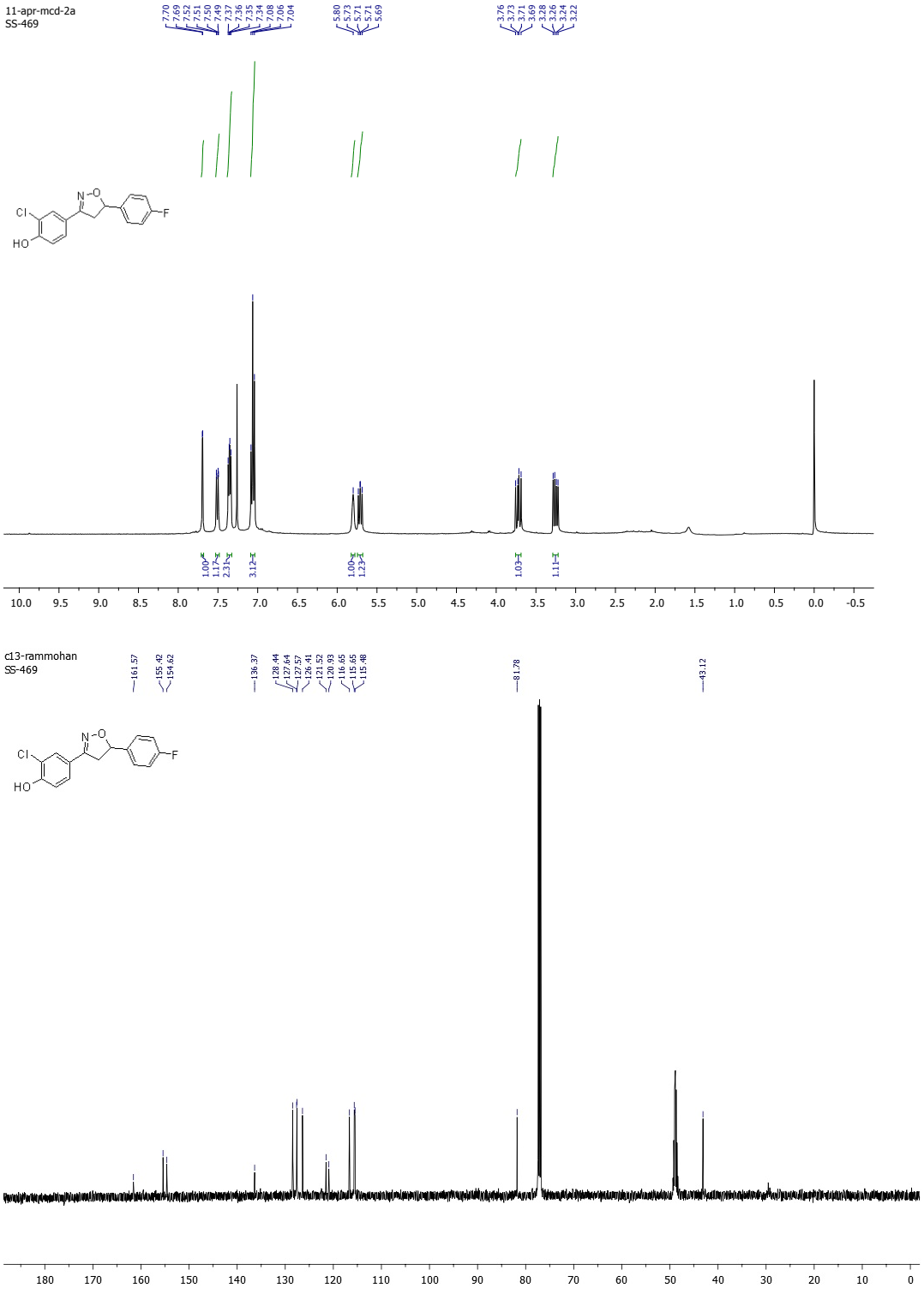
S3.33. ^{1}H, ^{13}C and DEPT135 NMR spectra of 2-chloro-4-(5-phenylisoxazol-3-yl) phenol (**7d**)

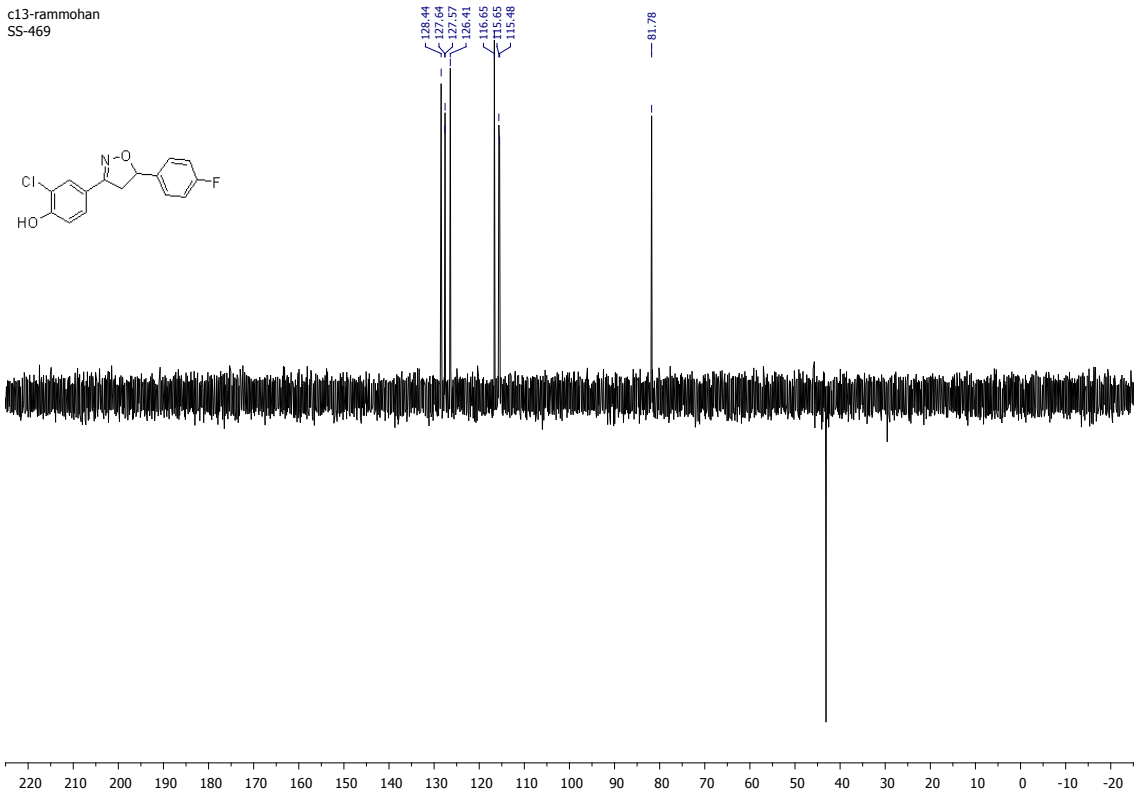




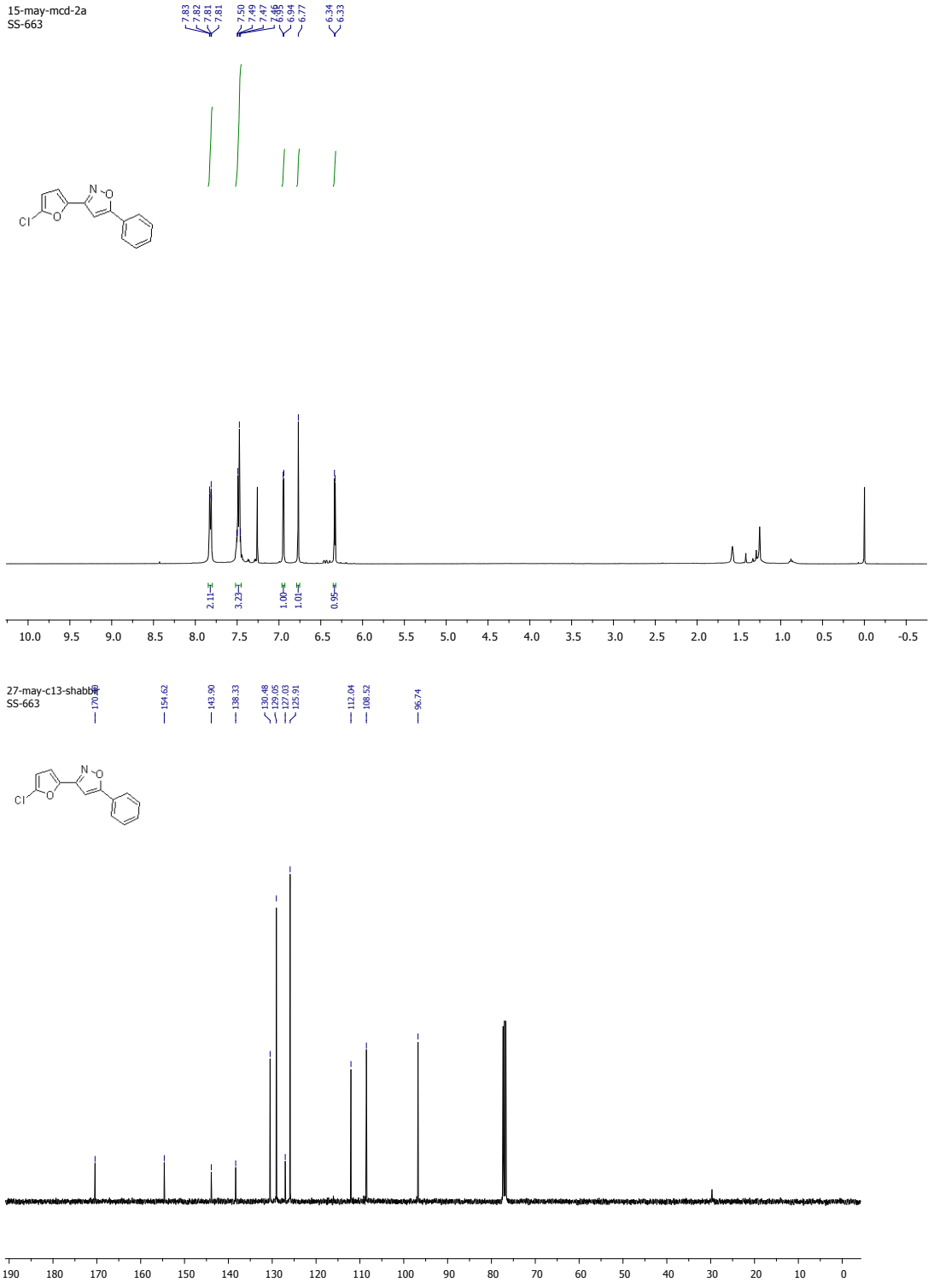

c13-shabeer
SS-694
128.72
128.43
128.27
126.48
125.79
116.68
82.47
Cl
HO
N
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

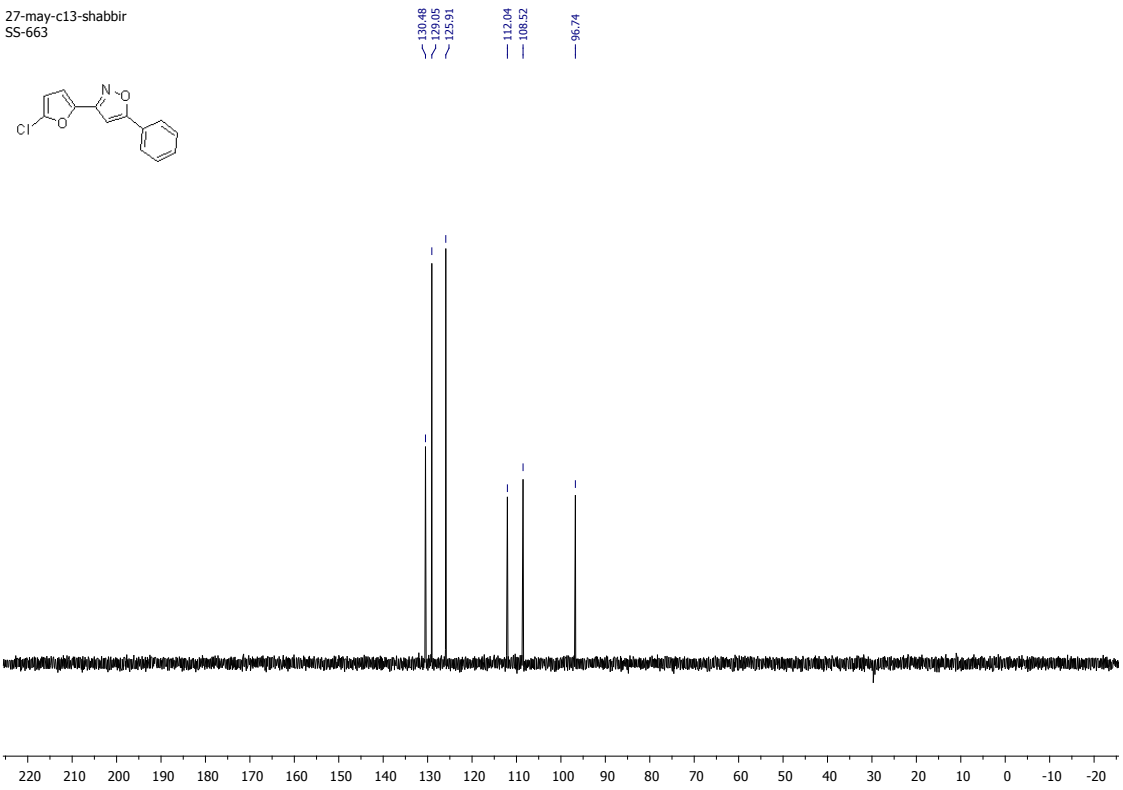
S3. 34. ^{1}H, ^{13}C and DEPT135 NMR spectra of 2-chloro-4-(5-(4-fluorophenyl)-4, 5-dihydroisoxazole-3yl)phenol (**7e**)



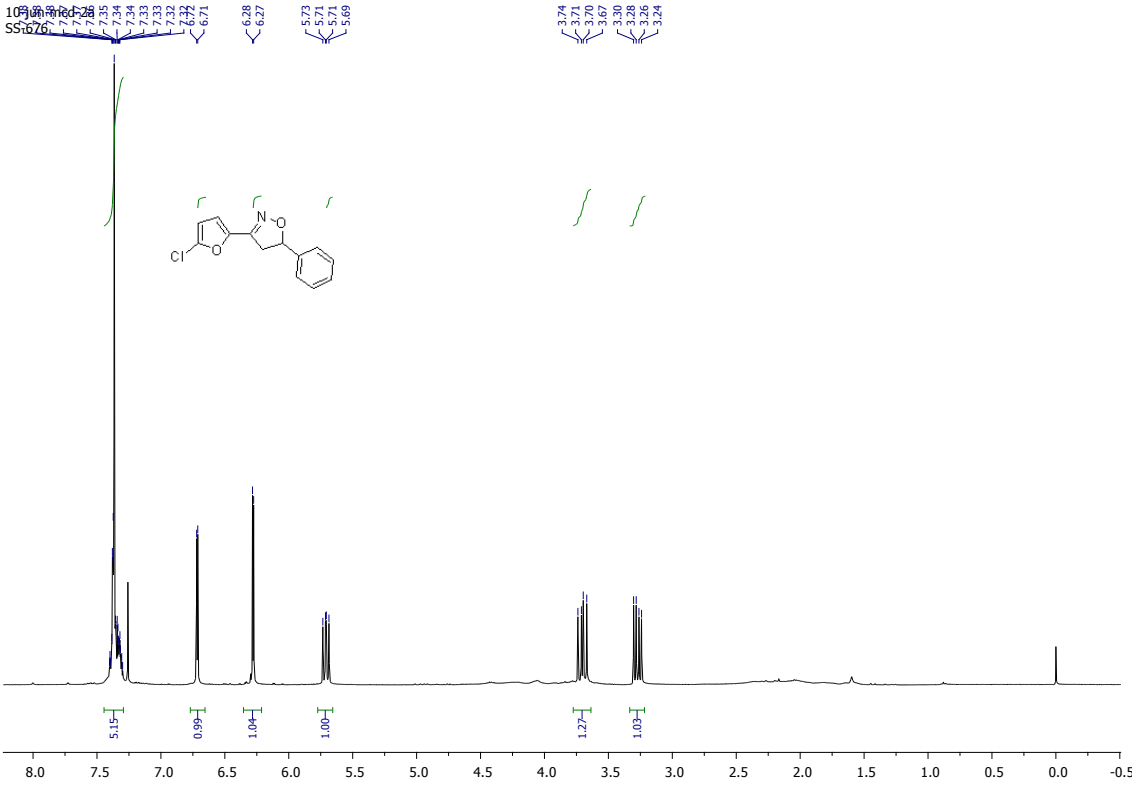

c13-rammohan
SS-469
128.44
127.64
127.57
126.41
116.65
115.65
115.48
81.78
Cl
HO
N
O
F
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

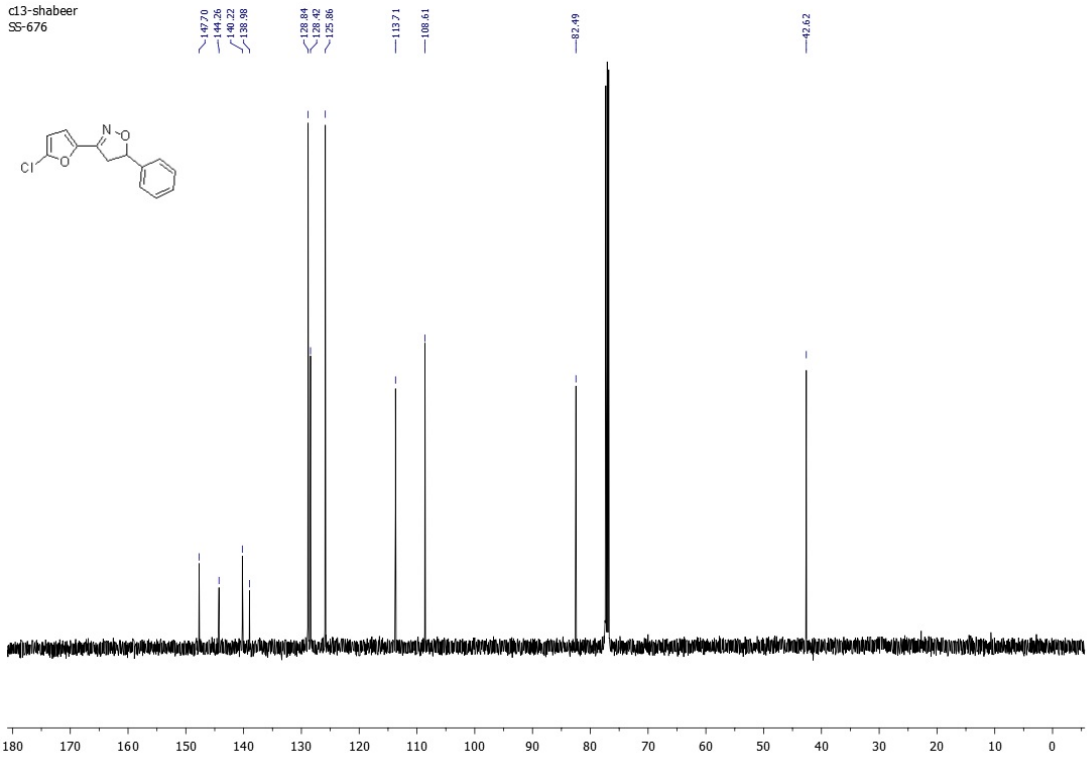
S3. 35. ^{1}H, ^{13}C and DEPT135 NMR spectra of 3-(5-chlorofuran-2-yl)-5-phenylisoxazole (**7f**)

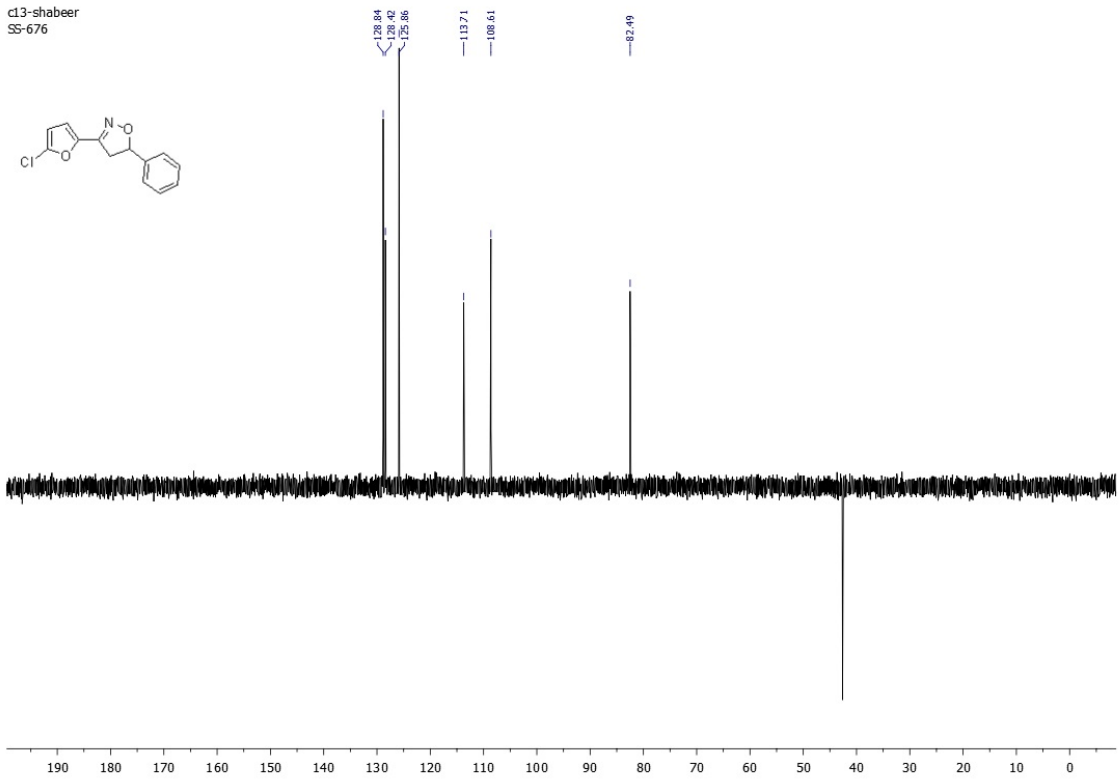



27-may-c13-shabbir
SS-663
130.48
129.05
125.91
112.04
108.52
96.74
Cl
O
N
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20

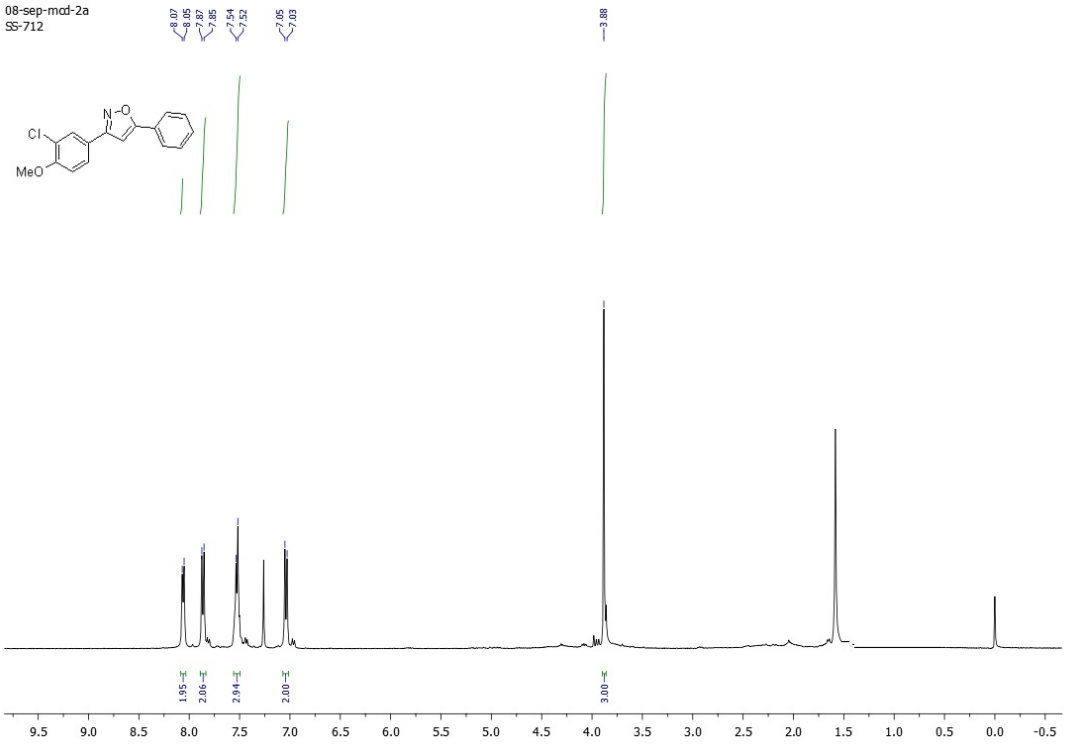
S3. 36. ^{1}H, ^{13}C and DEPT135 NMR spectra of 3-(5-chlorofuran-2-yl)-5-phenyl-4, 5-dihydroisoxazole (**7g**)

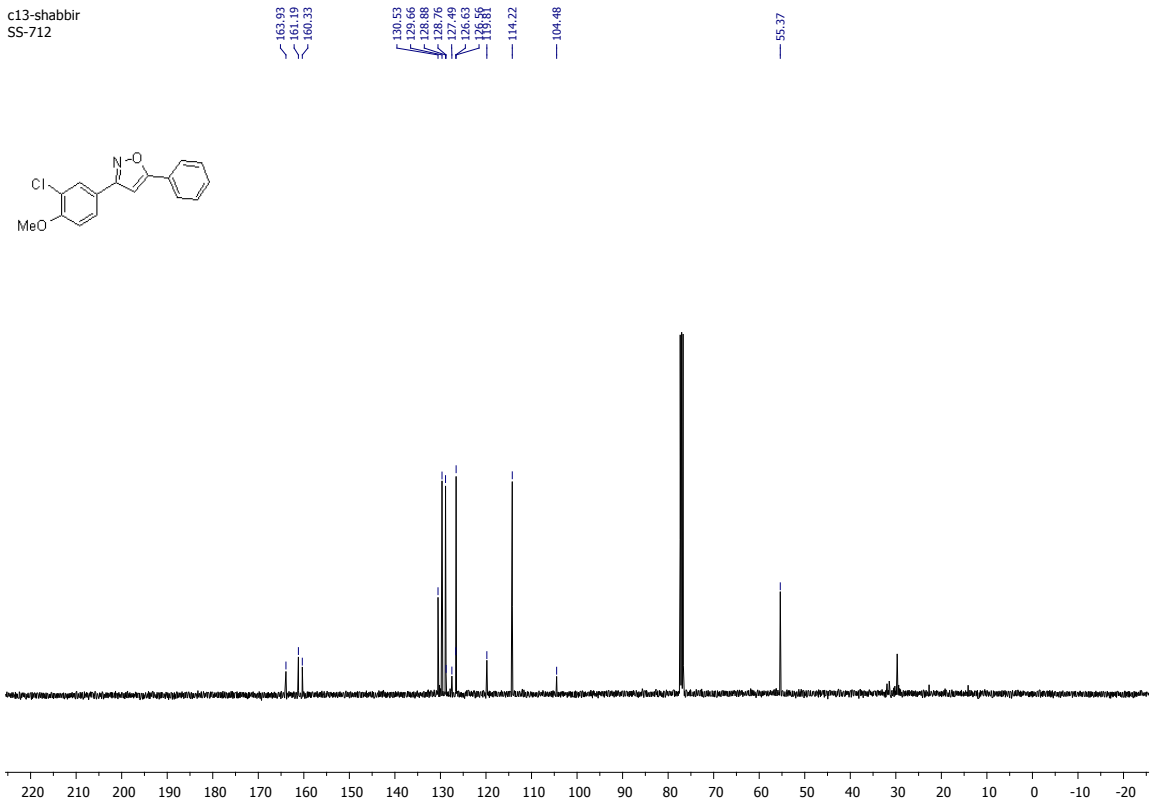



c13-shabeer
SS-676
147.70
144.26
140.22
138.98
128.84
128.42
125.86
113.71
108.61
82.49
42.62
Cl
O
N
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

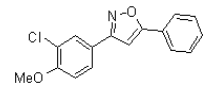

c13-shabeer
SS-676
128.84
128.42
125.86
113.71
108.61
82.49
Cl
O
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

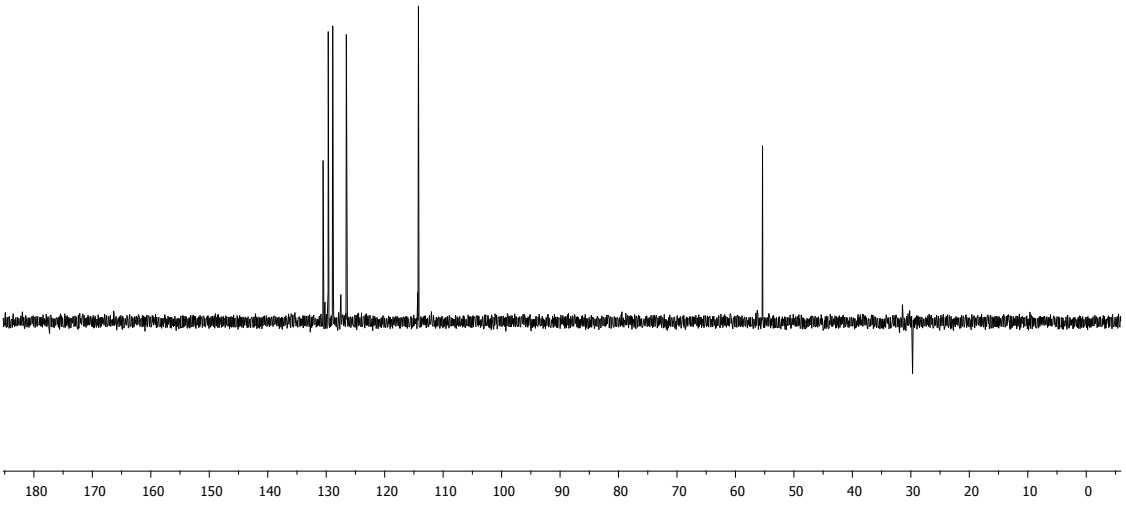
S3. 37. ^{1}H, ^{13}C and DEPT135 NMR spectra of 3-(3-chloro-4-methoxyphenyl)-5-phenylisoxazole (**7h**)





c13-shabbir
SS-712





S4. LCMS chromatogram at different time intervals.

LC-ESI-MS/MS analysis was carried out on Agilent Triple-Quad LC-MS/MS system (model 6410). Liquid chromatography analyses were carried out using an Agilent 1260 Infinity (Agilent, USA) quaternary pump equipped with an autosampler, column heater and online degasser. A Chromolith C_{18} (Merck, Germany) column (55 x 4.6 mm) was used at 30 °C temperature and the injection volume was 10 µl. The elution was carried out with binary solvent system consisting of 0.1% formic acid in water (solvent A) and acetonitrile (solvent B) at a constant flow-rate of 0.5 ml/min. The gradient elution was used, as depicted in Table S4-a.

Table S4-a. Gradient details used in LC-ESI-MS/MS analysis

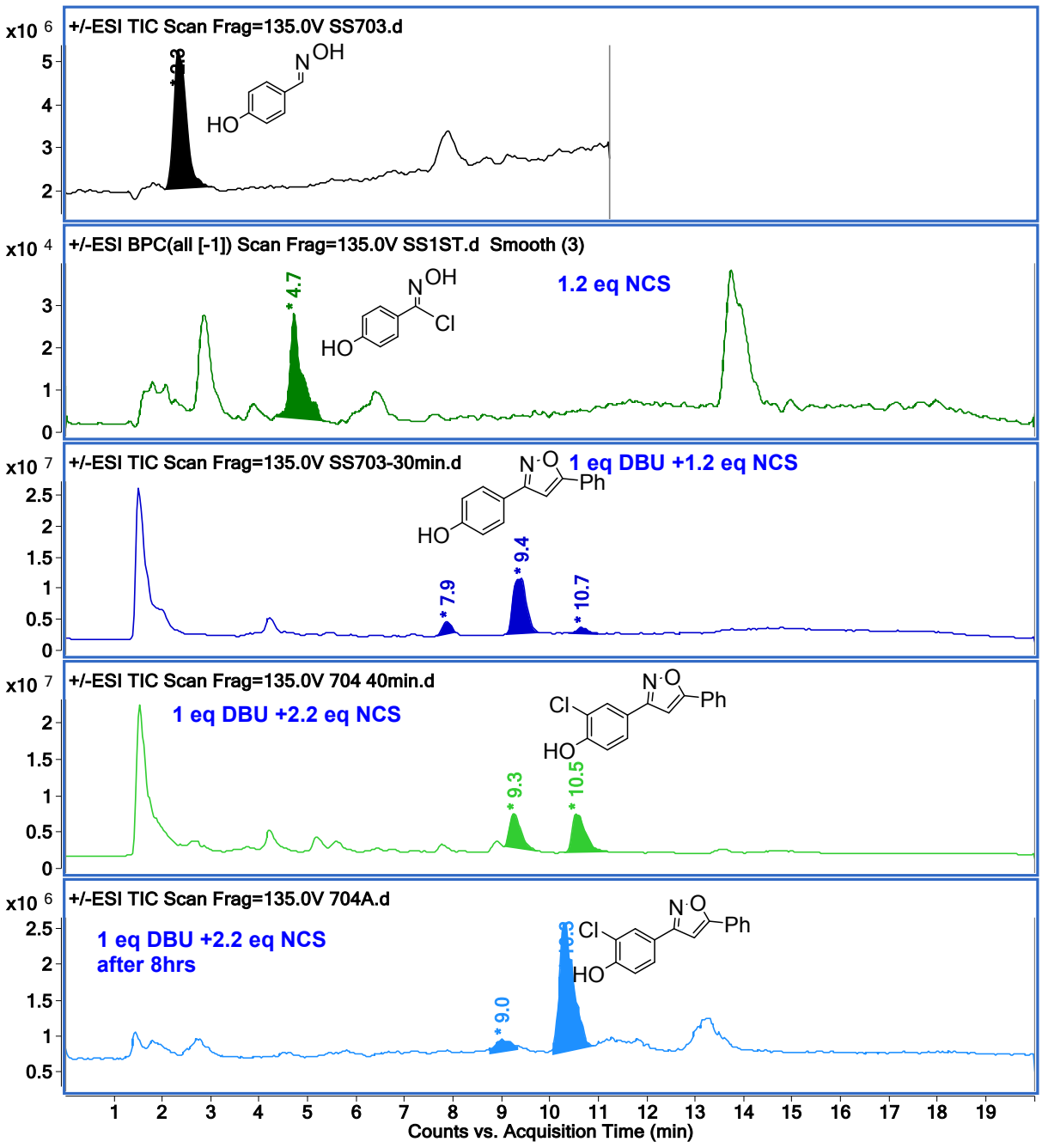
Time	**Solvent A (water – 0.1% formic acid)**	**Solvent B (acetonitrile)**	**Flow rate (ml/min)**
0	80	20	0.5
8	40	60	0.5
15	40	60	0.5
17	80	20	0.5
20	80	20	0.5

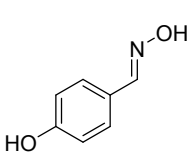
Other instrument parameters are listed in Table S4-b.

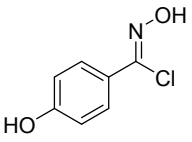
Table S4-b. Instrument parameters during LC-ESI-MS/MS analysis

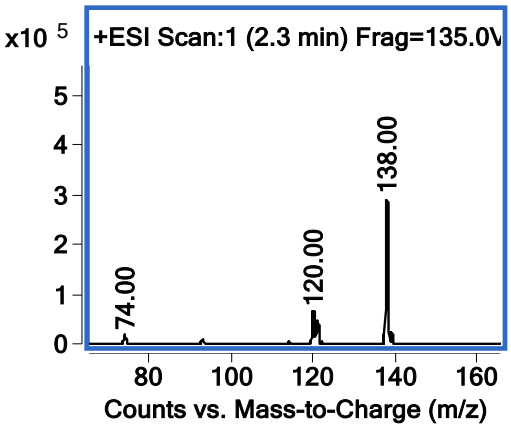
Source parameters		**Autosampler parameters**		**Quaternary pump parameters**	
Gas Temp.	300 °C	Injection volume	10 µl	Run time	15 min
Gas fgelow	8 L/min	Eject speed	200 µl/min	Flow rate	0.5 ml/min
Nebulizer pressure	50 psi	Draw speed	200 µl/min	Elution	Gradient*
Capillary voltage	3000 v				
Cell Acc (v)	7 v				

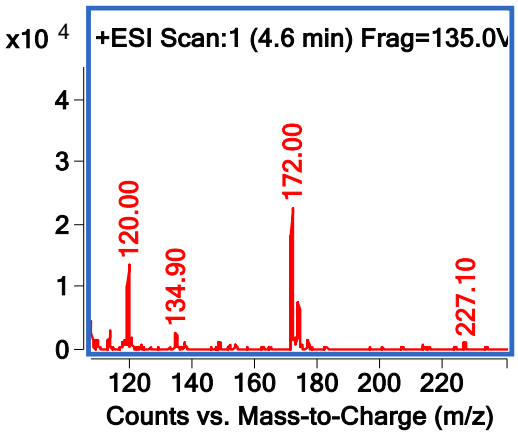
* Gradient system is provided in Table S4-a

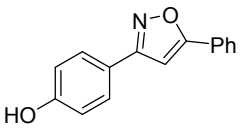

+/-ESI TIC Scan Frag=135.0V SS703.d
+/-ESI BPC(all [-1]) Scan Frag=135.0V SS1ST.d Smooth (3)
1.2 eq NCS
* 4.7
+/-ESI TIC Scan Frag=135.0V SS703-30min.d
1 eq DBU +1.2 eq NCS
* 7.9
* 9.4
* 10.7
+/-ESI TIC Scan Frag=135.0V 704 40min.d
1 eq DBU +2.2 eq NCS
* 9.3
* 10.5
+/-ESI TIC Scan Frag=135.0V 704A.d
1 eq DBU +2.2 eq NCS
after 8hrs
* 9.0
Counts vs. Acquisition Time (min)

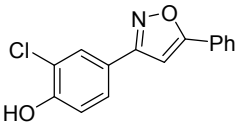

N
OH
HO

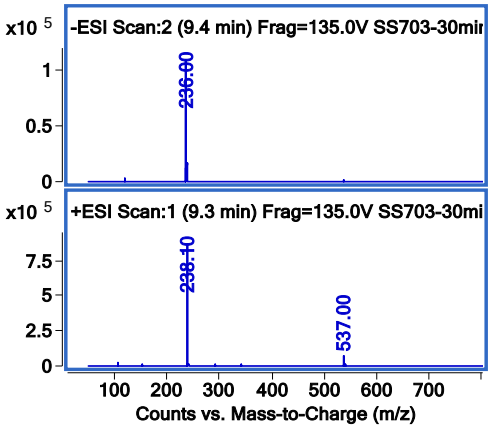

N
OH
Cl
HO

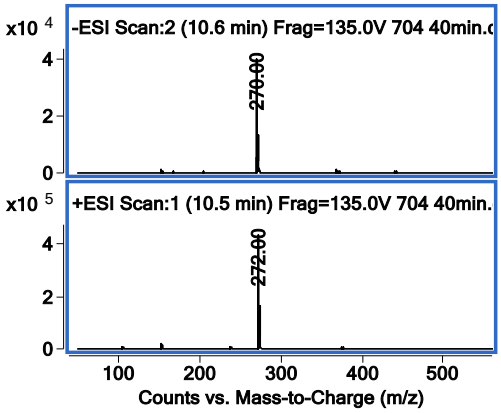

x10 5
+ESI Scan:1 (2.3 min) Frag=135.0V
74.00
120.00
138.00
Counts vs. Mass-to-Charge (m/z)

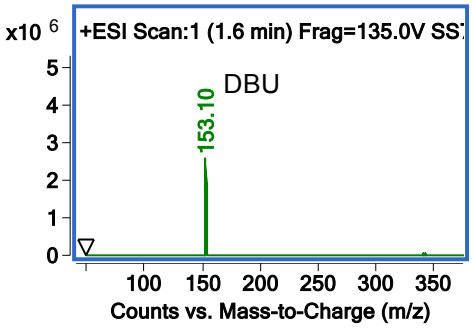

x10 4
+ESI Scan:1 (4.6 min) Frag=135.0V
120.00
134.90
172.00
227.10
Counts vs. Mass-to-Charge (m/z)


N
O
Ph
HO


N
O
Cl
Ph
HO


x10 5
-ESI Scan:2 (9.4 min) Frag=135.0V SS703-30mi
296.90
x10 5
+ESI Scan:1 (9.3 min) Frag=135.0V SS703-30mi
298.10
537.00
Counts vs. Mass-to-Charge (m/z)


x10 4
-ESI Scan:2 (10.6 min) Frag=135.0V 704 40min.
270.00
x10 5
+ESI Scan:1 (10.5 min) Frag=135.0V 704 40min.
272.00
Counts vs. Mass-to-Charge (m/z)


x10 6
+ESI Scan:1 (1.6 min) Frag=135.0V SS
DBU
153.10
Counts vs. Mass-to-Charge (m/z)

S5. References associated with ESI

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