Supporting Information

I_2/$CuCl_2$-promoted One-pot Three-component Synthesis of Aliphatic or Aromatic Substituted 1,2,3-thiadiazoles

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1. General

All substrates and reagents were commercially available and used without further purification. TLC analysis was performed using pre-coated glass plates. Column chromatography was performed using silica gel (200–300 mesh). IR spectra were recorded on a Perkin-Elmer PE-983 infrared spectrometer as KBr pellets with absorption in cm^{-1}. ^1H spectra were recorded in $CDCl_3$ or DMSO-d_6 on 400/600 MHz NMR spectrometers and resonances (δ) are given in parts per million relative to tetramethylsilane. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz) and integration. ^{13}C spectra were recorded in $CDCl_3$ or DMSO-d6 on 100/150 MHz NMR spectrometers and resonances (δ) are given in ppm. HRMS were obtained on a Bruker 7-tesla FT-ICR MS equipped with an electrospray source. Melting points were determined using XT-4 apparatus and not corrected.

2. Experimental Section

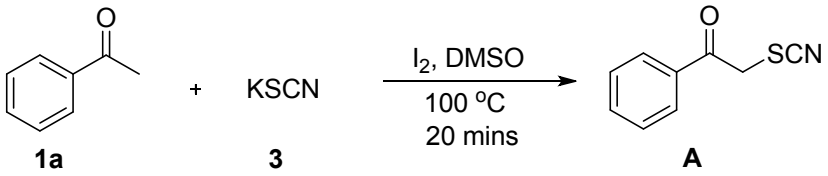
2.1 General procedure for the synthesis of 4 (4a as an example)

A sealed tube was charged with acetophenone (**1a**) (120 mg, 1.0 mmol), *p*-toluenesulfonyl hydrazide (**2**) (186.2 mg, 1.0 mmol), and potassium thiocyanate (**3**) (97.2 mg, 1.0 mmol), iodine (507.6 mg, 2.0 mmol) at room temperature, and DMSO (3 mL) was added. The resulting mixture was stirred at 100 °C for 1 h. After the reaction completed, the mixture was quenched with saturation $Na_2S_2O_3$ solution (50 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 20:1) to yield the desired product **4a** as a white solid.

2.2 General procedure for the synthesis of α- iodoacetophenone (1aa)[1]

A sealed tube was charged with acetophenone (**1a**) (1.2 g), iodine (2.79 g) and CuO (875 mg) at room temperature, and methanol (25 mL) was added. The resulting mixture was stirred at 65 °C until the reaction completed, the mixture was quenched with saturation $Na_2S_2O_3$ solution (80 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The desired product α- iodoacetophenone (**1aa**) is obtained as a brown oil.

2.3 General procedure for the synthesis of 1-phenyl-2-thiocyanatoethanone (A)



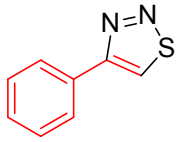
1a **3** **A**

A sealed tube was charged with acetophenone (**1a**) (1.20 g, 10.0 mmol), potassium thiocyanate (**3**) (972 mg, 10.0 mmol), iodine (5.08 g, 20.0 mmol) at room temperature, and DMSO (30 mL) was added. The resulting mixture was stirred at 100 °C for 20 mins. After the reaction completed, the mixture was quenched with saturation $Na_2S_2O_3$ solution (50 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 10:1) to yield the desired product 1-phenyl-2-thiocyanatoethanone (**A**) as a slight yellow solid. ^1H NMR (600 MHz, $CDCl_3$) δ 7.93 (m, *J* = 7.8 Hz, 2H), 7.66 (t, *J* = 7.2 Hz, 1H), 7.52 (t, *J*= 7.8 Hz, 2H), 4.74 (s, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 190.8, 134.7, 133.9, 129.1, 128.4, 111.8, 42.9.

2.4 General procedure for the synthesis of 4-methyl-N'-(1-phenylethylidene)benzenesulfonohydrazide (B)[2]

A solution of *p*-toluenesulfonyl hydrazide (**2**) (931 mg, 5.0 mmol) in methanol (5 mL) was stirred and heated to 60 °C until the *p*-toluenesulfonyl hydrazide was completely dissolved. Then acetophenone (**1a**) (660 mg, 5.5 mmol) was dropped to the mixture slowly. The reaction mixture was stirred at 60 °C for 30 mins. After the completion of reaction, the reaction mixture was cooled and the crude products was obtained as precipitates. The precipitates were washed by petroleum ether then were dried. The desired product 4-methyl-N'-(1-phenylethylidene)benzenesulfonohydrazide (**B**) is obtained as a white solid.

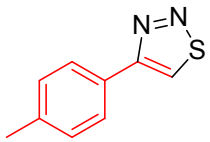
3. Characterization data for target compounds



4a

4-phenyl-1,2,3-thiadiazole (4a)[3]

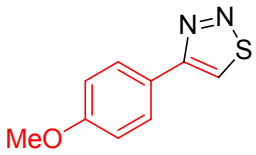
Yield 89%; 144.37 mg; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.51 (s, 1H), 7.88 (d, *J* = 7.2 Hz, 2H), 7.34 (t, *J* = 7.2 Hz, 2H), 7.29 (d, *J* = 6.6 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.5, 130.5, 130.1, 129.2, 128.9, 127.1. The spectroscopic data match a literature report.



4b

4-(p-tolyl)-1,2,3-thiadiazole (4b)[3]

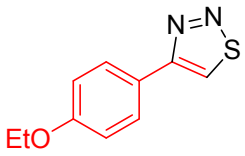
Yield 83%; 146.28 mg; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.56 (s, 1H), 7.90 (d, *J* = 7.8 Hz, 2H), 7.27 (d, *J* = 7.2Hz, 2H), 2.39 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.7, 139.3, 129.6, 129.4, 129.3, 127.1, 21.2. The spectroscopic data match a literature report.



4c

4-(4-methoxyphenyl)-1,2,3-thiadiazole (4c) [3]

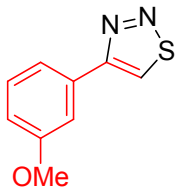
Yield 81%; 155.71 mg; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.51 (s, 1H), 7.95 (d, *J* = 7.8 Hz, 2H), 6.99 (d, *J* = 8.4 Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.4, 160.2, 129.6, 128.5, 123.3, 114.3, 55.2. The spectroscopic data match a literature report.



4d

4-(4-ethoxyphenyl)-1,2,3-thiadiazole (4d)

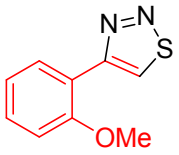
Yield 86%; 177.38 mg; white solid; mp 148-150 °C; IR (KBr): 3105, 1607, 1530, 1459, 1249, 1221, 1174, 1044, 842, 777 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.50 (s, 1H), 7.93 (d, *J* = 7.2 Hz, 2H), 6.97 (d, *J* = 7.8 Hz, 2H), 4.09–4.01 (m, 2H), 1.43 (t, *J* = 6.0 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.5, 159.7, 128.5, 128.4, 123.1, 114.8, 63.4, 14.6; HRMS (ESI) m/z calcd for $C_{10}H_{11}N_2OS^+$ $(M+H)^+$ 207.0587, found 207.0583.



4e

4-(3-methoxyphenyl)-1,2,3-thiadiazole (4e)[4]

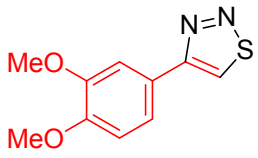
Yield 82%; 157.64 mg; ^1H NMR (400 MHz, $CDCl_3$) δ 8.64 (s, 1H), 7.66–7.63 (m, 1H), 7.56 (d, *J* = 7.6 Hz, 1H), 7.40 (t, *J* = 8.0 Hz, 1H), 7.00–6.96 (m, 1H), 3.88 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6, 160.1, 131.9, 130.3, 130.1, 119.6, 115.2, 112.6, 55.3. The spectroscopic data match a literature report.



4f

4-(2-methoxyphenyl)-1,2,3-thiadiazole (4f)[5]

Yield 87%; 168.99 mg; ^1H NMR (600 MHz, $CDCl_3$) δ 9.02 (s, 1H), 8.49 (d, *J* = 7.8 Hz, 1H), 7.37 (t, *J* = 7.8 Hz, 1H), 7.10 (t, *J* = 7.2 Hz, 1H), 7.00 (d, *J* = 7.8 Hz, 1H), 3.90 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 158.2, 156.1, 133.2, 130.1, 130.0, 120.9, 119.4, 111.0, 55.3. The spectroscopic data match a literature report.

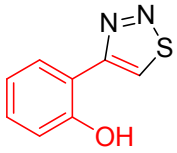


4g

4-(3,4-dimethoxyphenyl)-1,2,3-thiadiazole (4g)

Yield 81%; 180.03 mg; slight yellow solid; mp 144-146 °C; IR (KBr): 3106, 1599, 1531, 1477, 1290, 1234, 1137, 1026, 848, 796 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.49 (s, 1H), 7.55 (s, 1H), 7.40 (d, *J* = 7.8 Hz, 1H), 6.84 (d, *J* = 7.8

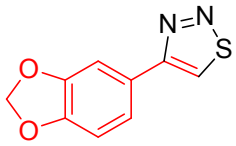
Hz, 1H), 3.84 (s, 3H), 3.81 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.3, 149.6, 148.9, 128.7, 123.3, 119.6, 111.1, 110.0, 55.63, 55.58; HRMS (ESI) m/z calcd for $C_{10}H_{11}N_2O_2S^+$ $(M+H)^+$ 223.0536, found 223.0532.



4h

2-(1,2,3-thiadiazol-4-yl)phenol (4h)

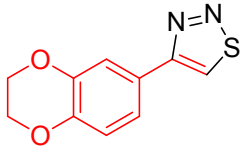
Yield 71%; 126.53 mg; yellow solid; mp 155-157 °C; IR (KBr): 3221, 3139, 2962, 1596, 1440, 1373, 1257, 1236, 768 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.55 (s, 1H), 8.79 (s, 1H), 7.62 (d, *J* = 7.2 Hz, 1H), 7.33 (t, *J* = 7.2 Hz, 1H), 7.11 (d, *J* = 7.8 Hz, 1H), 6.96 (t, *J* = 7.2 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.0, 155.8, 131.2, 130.1, 127.4, 120.0, 118.1, 114.4; HRMS (ESI) m/z calcd for $C_8H_7N_2OS^+$ $(M+H)^+$ 179.0274, found 179.0269.



4i

4-(benzo[d][1,3]dioxol-5-yl)-1,2,3-thiadiazole (4i)

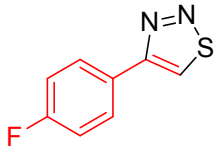
Yield 85%; 175.29 mg; white solid; mp 197-199 °C; IR (KBr): 3089, 1464, 1286, 1242, 1038, 931, 869, 779 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.51 (s, 1H), 7.56–7.51 (m, 2H), 6.93 (d, *J* = 7.8 Hz, 1H), 6.04 (s, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.5, 148.5, 148.3, 128.8, 124.9, 121.4, 108.8, 107.7, 101.4; HRMS (ESI) m/z calcd for $C_9H_7N_2O_2S^+$ $(M+H)^+$ 207.0223, found 207.0220.



4j

4-(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)-1,2,3-thiadiazole (4j)

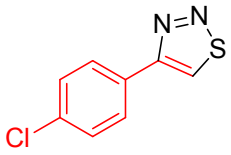
Yield 87%; 191.62 mg; slight yellow solid; mp 153-155 °C; IR (KBr): 3072, 1585, 1527, 1461, 1315, 1278, 1246, 1064, 888, 806 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.47 (s, 1H), 7.50 (s, 1H), 7.44 (d, *J* = 7.8 Hz, 1H), 6.90 (d, *J* = 8.4 Hz, 1H), 4.23 (s, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.1, 144.4, 143.7, 128.9, 124.0, 120.3, 117.6, 116.1, 64.2, 64.1; HRMS (ESI) m/z calcd for $C_{10}H_9N_2O_2S^+$ $(M+H)^+$ 221.0379, found 221.0376.



4k

4-(4-fluorophenyl)-1,2,3-thiadiazole (4k)[3]

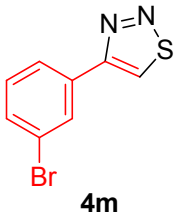
Yield 84%; 151.37 mg; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.64 (s, 1H), 8.05–7.98 (m, 2H), 7.18 (t, *J* = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 163.2 (d, $J_{C\text{-}F}$ = 249.0 Hz), 161.7, 129.8 (d, $J_{C\text{-}F}$ = 7.5 Hz), 129.1, 126.9, 116.1 (d, $J_{C\text{-}F}$ = 21.0 Hz). The spectroscopic data match a literature report.



4l

4-(4-chlorophenyl)-1,2,3-thiadiazole (4l)[3]

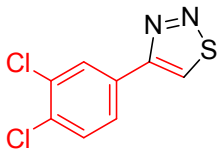
Yield 85%; 167.16 mg; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.66 (s, 1H), 7.98 (d, *J* = 7.8 Hz, 2H), 7.47 (d, *J* = 7.2 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 161.6, 135.3, 130.3, 130.2, 129.3, 128.5. The spectroscopic data match a literature report.



4m

4-(3-bromophenyl)-1,2,3-thiadiazole (4m)

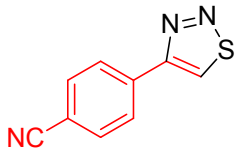
Yield 81%; 195.30 mg; slight yellow solid; mp 152-154 °C; IR (KBr): 3074, 1631, 1562, 1446, 1402, 1222, 1041, 787 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.69 (s, 1H), 8.19 (s, 1H), 7.96 (d, *J* = 7.8 Hz, 1H), 7.55 (d, *J* = 7.2 Hz, 1H), 7.36 (t, *J* = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 161.1, 132.5, 132.2, 130.8, 130.6, 130.2, 125.8, 123.1. This product could't be detected by HRMS (ESI), so we used GC-MS (EI) to detect it (see below).



4n

4-(3,4-dichlorophenyl)-1,2,3-thiadiazole (4n)

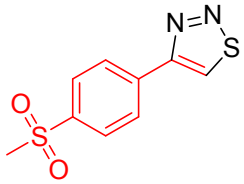
Yield 79%; 182.57 mg; white solid; mp 139-141 °C; IR (KBr): 3094, 1630, 1442, 1383, 1225, 1028, 825, 777 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.71 (s, 1H), 8.14 (s, 1H), 7.87 (d, *J* = 7.2 Hz, 1H), 7.56 (d, *J* = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 160.3, 133.4, 133.3, 131.1, 131.0, 130.5, 129.0, 126.3; HRMS (ESI) m/z calcd for $C_8H_5Cl_2N_2S^+$ $(M+H)^+$ 230.9545, found 230.9541.



4o

4-(1,2,3-thiadiazol-4-yl)benzonitrile (4o)[6]

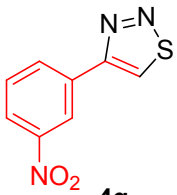
Yield 76%; 142.29 mg; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.84 (s, 1H), 8.20 (d, *J* = 7.8 Hz, 2H), 7.82 (d, *J* = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 160.7, 134.8, 132.9, 132.2, 127.8, 118.3, 112.8. The spectroscopic data match a literature report.



4p

4-(4-(methylsulfonyl)phenyl)-1,2,3-thiadiazole (4p)

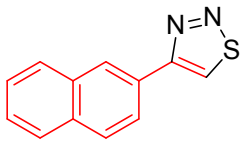
Yield 75%; 180.23 mg; brown solid; mp 219-221 °C; IR (KBr): 3093, 1664, 1598, 1407, 1295, 1147, 959, 929, 836, 771 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6) δ 9.84 (s, 1H), 8.41 (d, *J* = 8.4 Hz, 2H), 8.10 (d, *J* = 7.8 Hz, 2H), 3.30 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 160.1, 141.0, 135.8, 135.3, 127.94, 127.87, 43.5; HRMS (ESI) m/z calcd for $C_9H_9N_2O_2S_2^+$ $(M+H)^+$ 241.0100, found 241.0096.



4q

4-(3-nitrophenyl)-1,2,3-thiadiazole (4q)

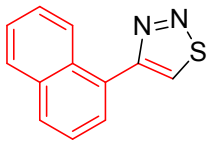
Yield 73%; 151.26 mg; purple solid; mp 209-211 °C; IR (KBr): 2924, 1663, 1631, 1530, 1442, 1408, 1229, 785 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.87 (s, 2H), 8.48 (d, *J* = 7.2 Hz, 1H), 8.32 (d, *J* = 7.8 Hz, 1H), 7.74 (t, *J* = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 160.4, 148.7, 133.1, 132.4, 131.8, 130.3, 124.0, 122.1; HRMS (ESI) m/z calcd for $C_8H_6N_3O_2S^+$ $(M+H)^+$ 208.0175, found 208.0172.



4r

4-(naphthalen-2-yl)-1,2,3-thiadiazole (4r)[3]

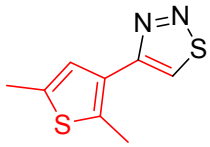
Yield 74%; 157.08 mg; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.65–8.62 (m, 1H), 8.53–8.50 (m, 1H), 8.04–8.00 (m, 1H), 7.92–7.85 (m, 2H), 7.85–7.79 (m, 1H), 7.52–7.47 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.7, 133.5, 133.3, 130.14, 130.11, 128.8, 128.4, 127.9, 127.7, 126.8, 126.7, 124.6. The spectroscopic data match a literature report.



4s

4-(naphthalen-1-yl)-1,2,3-thiadiazole (4s)[3]

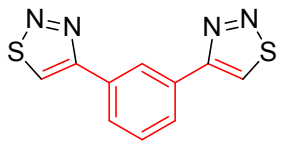
Yield 71%; 150.71 mg; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.67–8.61 (m, 1H), 8.11–8.04 (m, 1H),8.00–7.90 (m, 2H), 7.77–7.70 (m, 1H), 7.60–7.48 (m, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 161.8, 134.3, 134.2, 133.8, 131.3, 129.9, 128.5, 128.3, 127.0, 126.2, 125.2, 125.0. The spectroscopic data match a literature report.



4t

4-(2,5-dimethylthiophen-3-yl)-1,2,3-thiadiazole (4t)

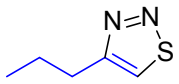
Yield 85%; 166.85 mg; brown solid; mp 99-101 °C; IR (KBr): 3077, 1630, 1513, 1438, 1326, 1198, 1020, 850, 814 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.41 (s, 1H), 7.10 (s, 1H), 2.64 (s, 3H), 2.46 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 158.6, 136.4, 135.9, 130.1, 127.1, 126.3, 15.0, 14.9; HRMS (ESI) m/z calcd for $C_8H_9N_2S_2^+$ $(M+H)^+$ 197.0202, found 197.0197.



4u

1,3-di(1,2,3-thiadiazol-4-yl)benzene (4u)

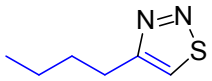
Yield 81%; 199.51 mg; white solid; mp 205-207 °C; IR (KBr): 3072, 1662, 1632, 1447, 1410, 1222, 890, 779 cm^{-1}; ^{1}H NMR (600 MHz, DMSO-d_6) δ 9.75 (s, 2H), 8.88 (s, 1H), 8.22 (d, J = 7.2 Hz, 2H), 7.70 (t, J = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 161.3, 134.0, 131.6, 130.1, 127.8, 125.7; HRMS (ESI) m/z calcd for $C_{10}H_7N_4S_2^+$ $(M+H)^+$ 247.010664, found 247.010655.



6a

4-propyl-1,2,3-thiadiazole (6a)

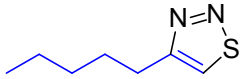
Yield 62%; 79.48 mg; yellow oil; IR (KBr): 2926, 1881, 1784, 1760, 1663, 1630, 1407, 830, 702 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.19 (s, 1H), 3.15 (t, *J* = 7.8 Hz, 2H), 1.90–1.83 (m, 2H), 1.02 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 163.9,131.3, 30.1, 22.9, 13.6; HRMS (ESI) m/z calcd for $C_5H_9N_2S^+$ $(M+H)^+$ 129.0481, found 129.0481.



6b

4-butyl-1,2,3-thiadiazole (6b)

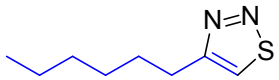
Yield 54%; 76.80 mg; yellow oil; IR (KBr): 3105, 2958, 2930, 1663, 1630, 1486, 1442, 1229, 882, 795 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.19 (s, 1H), 3.17 (t, *J* = 7.6 Hz, 2H), 1.86–1.77 (m, 2H), 1.48–1.37 (m, 2H), 0.97 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 131.1, 31.6, 27.8, 22.1, 13.7; HRMS (ESI) m/z calcd for $C_6H_{11}N_2S^+$ $(M+H)^+$ 143.06375, found 143.06334.



6c

4-pentyl-1,2,3-thiadiazole (6c)

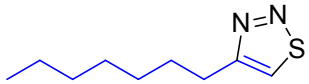
Yield 56%; 87.50 mg; yellow oil; IR (KBr): 3104, 2957, 2930, 1663, 1629, 1488, 1463, 1228, 982, 886, 805 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 3.16 (t, *J* = 7.6 Hz, 2H), 1.88–1.80 (m, 2H), 1.41–1.34 (m, 4H), 0.94–0.88 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 131.1, 31.2, 29.2, 28.1, 22.2, 13.8; HRMS (ESI) m/z calcd for $C_7H_{13}N_2S^+$ $(M+H)^+$ 157.07940, found 157.07904.



6d

4-hexyl-1,2,3-thiadiazole (6d)

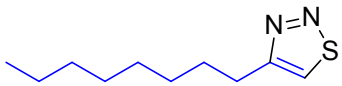
Yield 59%; 100.47 mg; yellow oil; IR (KBr): 3104, 2956, 2929, 1663, 1630, 1488, 1463, 1227, 982, 886, 792 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 3.19–3.12 (m, 2H), 1.87–1.77 (m, 2H), 1.44–1.29 (m, 6H), 0.92–0.85 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 131.1, 31.4, 29.5, 28.7, 28.1, 22.4, 13.9; HRMS (ESI) m/z calcd for $C_8H_{15}N_2S^+$ $(M+H)^+$ 171.09505, found 171.09468.



6e

4-heptyl-1,2,3-thiadiazole (6e)

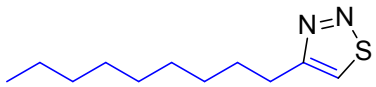
Yield 61%; 112.42 mg; yellow oil; IR (KBr): 3104, 2928, 2857, 1662, 1630, 1487, 1463, 1226, 980, 885, 804 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.23 (d, *J* = 2.4 Hz, 1H), 3.20–3.12 (m, 2H), 1.88–1.78 (m, 2H), 1.44–1.23 (m, 8H), 0.93–0.83 (m, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 164.0, 131.1, 31.5, 29.4, 28.9, 28.8, 28.0, 22.4, 13.8; HRMS (ESI) m/z calcd for $C_9H_{17}N_2S^+$ $(M+H)^+$ 185.11070, found 185.11047.



6f

4-octyl-1,2,3-thiadiazole (6f)

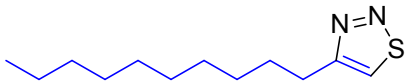
Yield 60%; 119.00 mg; yellow oil; IR (KBr): 3105, 2927, 2856, 1662, 1630, 1483, 1407, 1228, 980, 885, 791 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.21 (d, *J* = 1.8 Hz, 1H), 3.21–3.10 (m, 2H), 1.87–1.77 (m, 2H), 1.48–1.20 (m, 10H), 0.94–0.82 (m, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 164.0, 131.1, 31.6, 29.5, 29.1, 29.0, 28.1, 22.4, 13.9; HRMS (ESI) m/z calcd for $C_{10}H_{19}N_2S^+$ $(M+H)^+$ 199.12635, found 199.12631.



6g

4-nonyl-1,2,3-thiadiazole (6g)

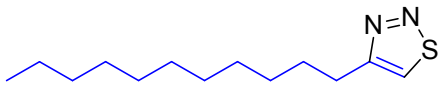
Yield 63%; 133.78 mg; yellow oil; IR (KBr): 3105, 2926, 2855, 1725, 1630, 1488, 1463, 1230, 981, 885, 803 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.16 (s, 1H), 3.11 (t, *J* = 7.8 Hz, 2H), 1.82–1.73 (m, 2H), 1.37–1.19 (m, 12H), 0.83 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 164.1, 131.2, 31.8, 29.6, 29.4, 29.3, 29.2, 29.1, 28.2, 22.6, 14.1; HRMS (ESI) m/z calcd for $C_{11}H_{21}N_2S^+$ $(M+H)^+$ 213.1420, found 213.1415.



6h

4-decyl-1,2,3-thiadiazole (6h)

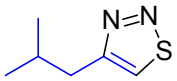
Yield 58%; 131.30 mg; yellow solid; mp 64-66 °C; IR (KBr): 3070, 2925, 2853, 1662, 1632, 1462, 1225, 986, 888, 816 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.21 (s, 1H), 3.15 (t, *J* = 7.2 Hz, 2H), 1.86–1.79 (m, 2H), 1.41–1.25 (m, 14H), 0.88 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 164.0, 131.0, 31.7, 29.5, 29.4, 29.3, 29.1, 29.0, 28.1, 22.5, 13.9; HRMS (ESI) m/z calcd for $C_{12}H_{23}N_2S^+$ $(M+H)^+$ 227.15765, found 227.15747.



6i

4-undecyl-1,2,3-thiadiazole (6i)

Yield 61%; 146.65 mg; slight yellow solid; mp 56-58 °C; IR (KBr): 3089, 2924, 2851, 1662, 1632, 1463, 1225, 988, 814 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.19 (s, 1H), 3.16 (t, *J* = 7.8 Hz, 2H), 1.86-1.79 (m, 2H), 1.42–1.23 (m, 16H), 0.88 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 164.1, 131.1, 31.8, 29.54, 29.49, 29.4, 29.2, 29.0, 28.1, 22.6, 14.0; HRMS (ESI) m/z calcd for $C_{13}H_{25}N_2S^+$ $(M+H)^+$ 241.1733, found 241.1727.



6j

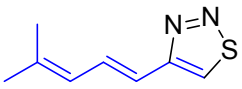
4-isobutyl-1,2,3-thiadiazole (6j)

Yield 69%; 98.13 mg; yellow oil; IR (KBr): 2926, 2855, 1663, 1590, 1530, 1443, 1382, 885, 807 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.15 (s, 1H), 3.00 (d, *J* = 7.2 Hz, 2H), 2.16-2.09 (m, 1H), 0.94 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 162.9, 131.8, 37.0, 29.0, 22.1; HRMS (ESI) m/z calcd for $C_6H_{11}N_2S^+$ $(M+H)^+$ 143.0637, found 143.0636.

6k

2-(1,2,3-thiadiazol-4-yl)propan-2-ol (6k)

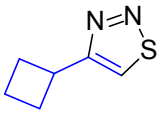
Yield 71%; 102.37 mg; yellow oil; IR (KBr): 3121, 2979, 2931, 1717, 1631, 1371, 1234, 1172, 955, 854, 800 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.49 (s, 1H), 3.68 (s, 1H), 1.73 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.4, 131.0, 70.7, 30.8; HRMS (ESI) m/z calcd for $C_5H_9N_2OS^+$ $(M+H)^+$ 145.0430, found 145.0428.



6l

(E)-4-(4-methylpenta-1,3-dien-1-yl)-1,2,3-thiadiazole (6l)

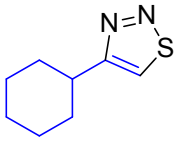
Yield 72%; 119.69 mg; brown solid; mp 150-152 °C; IR (KBr): 3106, 2979, 1667, 1630, 1377, 1234, 1099, 977, 854, 802 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.22 (s, 1H), 7.61-7.53 (m, 1H), 6.71 (d, *J* = 15.6 Hz, 1H), 6.06 (d, *J* = 11.4 Hz, 1H), 1.90 (s, 3H), 1.89 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 161.9, 140.3, 131.5, 129.0, 124.7, 117.5, 26.4, 18.7; HRMS (ESI) m/z calcd for $C_8H_{11}N_2S^+$ $(M+H)^+$ 167.0637, found 167.0636.



6m

4-cyclobutyl-1,2,3-thiadiazole (6m)

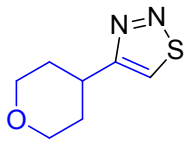
Yield 62%; 86.93 mg; yellow oil; IR (KBr): 3106, 2978, 2942, 1663, 1630, 1484, 1442, 1224, 968, 885, 798 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.14 (s, 1H), 3.99–3.89 (m, 1H), 2.44-2.35 (m, 2H), 2.34-2.24 (m, 2H), 2.06-1.97 (m, 1H), 1.93-1.85 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 167.7, 130.2, 33.6, 29.3, 18.5; HRMS (ESI) m/z calcd for $C_6H_9N_2S^+$ $(M+H)^+$ 141.0481, found 141.0478.



6n

4-cyclohexyl-1,2,3-thiadiazole (6n)

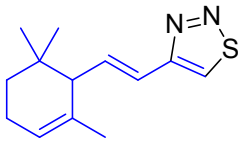
Yield 73%; 122.83 mg; slight yellow solid; mp 94-96 °C; IR (KBr): 3087, 2925, 2850, 1639, 1479, 1445, 1231, 1016, 971, 888, 826, 573 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.07 (s, 1H), 3.02-2.93 (m, 1H), 1.92 (d, *J* =12.0 Hz, 2H), 1.61 (d, *J* = 13.2 Hz, 2H), 1.51 (d, *J* = 12.6 Hz, 1H), 1.39-1.28 (m, 2H), 1.26-1.15 (m, 2H), 1.10-1.00 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.6, 129.7, 37.3, 32.6, 25.6, 25.3; HRMS (ESI) m/z calcd for $C_8H_{13}N_2S^+$ $(M+H)^+$ 169.0794, found 169.0791.



6o

4-(tetrahydro-2H-pyran-4-yl)-1,2,3-thiadiazole (6o)

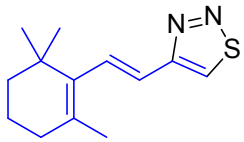
Yield 69%; 117.46 mg; slight yellow solid; mp 141-143 °C; IR (KBr): 3092, 2943, 2847, 1491, 1358, 1206, 1127, 1083, 978, 892, 827, 543 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.18 (s, 1H), 3.87 (d, *J* = 9.0 Hz, 2H), 3.39 (t, *J* = 12.0 Hz, 2H), 3.32-3.25 (m, 1H), 1.89 (d, *J* = 12.6 Hz, 2H), 1.78-1.70 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 166.9, 130.4, 67.0, 34.5, 32.2; HRMS (ESI) m/z calcd for $C_7H_{10}ON_2S^+$ $(M+H)^+$ 171.0587, found171.0583.



6p

(E)-4-(2-(2,6,6-trimethylcyclohex-2-en-1-yl)vinyl)-1,2,3-thiadiazole (6p)

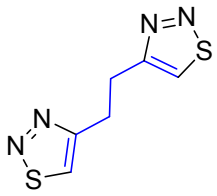
Yield 72%; 168.74 mg; yellow oil; IR (KBr): 3105, 2960, 2922, 1662, 1631, 1470, 1444, 1381, 1241, 979, 812 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.26 (s, 1H), 6.74 (d, *J* = 16.2 Hz, 1H), 6.70-6.64 (m, 1H), 5.50 (s, 1H), 2.36 (d, *J* = 9.0 Hz, 1H), 2.06 (s, 2H), 1.65 (s, 3H), 1.56–1.49 (m, 1H), 1.26-1.21 (m, 1H), 0.97 (s, 3H), 0.92 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 161.3, 138.4, 132.9, 129.2, 121.9, 120.0, 54.7, 32.5, 31.2, 27.8, 26.8, 23.0, 22.9; HRMS (ESI) m/z calcd for $C_{13}H_{20}N_2S^+$ $(M+H)^+$ 235.1263, found235.1259.



6q

(E)-4-(2-(2,6,6-trimethylcyclohex-1-en-1-yl)vinyl)-1,2,3-thiadiazole (6q)

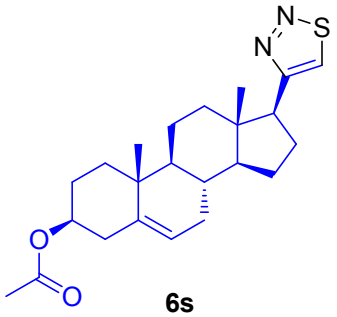
Yield 74%; 173.43 mg; brown oil; IR (KBr): 3103, 2930, 2865, 1663, 1621, 1467, 1379, 1213, 975, 883, 808 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.28 (s, 1H), 7.31 (d, *J* = 16.2 Hz, 1H), 6.72 (d, *J* = 16.2 Hz, 1H), 2.07 (s, 2H), 1.81 (s, 3H), 1.68-1.63 (m, 2H), 1.53–1.49 (m, 2H), 1.10 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 161.7, 136.8, 133.9, 131.4, 129.1, 121.2, 39.4, 34.1, 33.0, 28.8, 21.7, 19.0; HRMS (ESI) m/z calcd for $C_{13}H_{20}N_2S^+$ $(M+H)^+$ 235.1263, found235.1259.



6r

1,2-di(1,2,3-thiadiazol-4-yl)ethane (6r)

Yield 48%; 95.17 mg; brown solid; mp 148-150 °C; IR (KBr): 3112, 2927, 1663, 1594, 1443, 1380, 1217, 1167, 982, 884, 801 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.19 (s, 2H), 3.67 (s, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 161.7, 132.5, 27.9; HRMS (ESI) m/z calcd for $C_6H_7N_4S_2^+$ $(M+H)^+$ 199.0107, found199.0103.

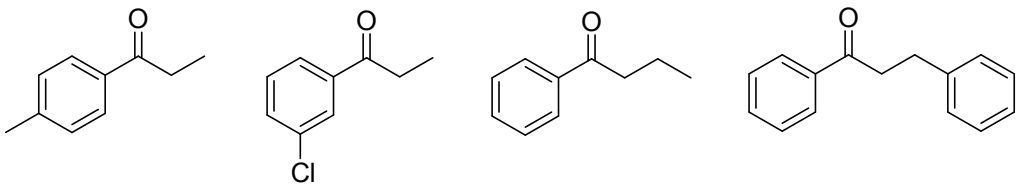


6s

(3S,8S,9S,10R,13S,14S,17S)-10,13-dimethyl-17-(1,2,3-thiadiazol-4-yl)-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl acetate (6s)

Yield 71%; 284.41 mg; yellow solid; mp 285-287 °C; IR (KBr): 3086, 2938, 2897, 1729, 1443, 1372, 1247, 1042, 894, 811 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.16 (s, 1H), 5.29 (s, 1H), 4.53–4.46 (m, 1H), 3.16 (t, *J* = 9.6 Hz, 1H), 2.27-2.18 (m, 4H), 2.16-2.07 (m, 2H), 1.92 (s, 3H), 1.81-1.73 (m, 5H), 1.58–1.40 (m, 6H), 1.37–1.27 (m, 4H), 1.25-1.17 (m, 2H), 1.10–1.02 (m, 2H), 1.00–0.94 (m, 2H), 0.91 (s, 3H), 0.39 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.0, 164.3, 139.3, 131.2, 121.9, 73.4, 55.6, 49.8, 49.7, 44.0, 37.7, 37.3, 36.6, 36.3, 31.8, 31.5, 27.3, 27.0, 24.3, 21.1, 20.3, 19.0, 12.7; HRMS (ESI) m/z calcd for $C_{23}H_{33}N_2O_2S^+$ $(M+H)^+$ 401.2257, found 401.2247.

Unsuccessful Substrates of α-substituted aryl ketones：



4. Reference

[1] A. Wu, Y. Pan, G. Yin, M. Gao, N. She and S. Hu, *Synthesis*, 2007, **2007**, 3113-3116.

[2] G. C. Senadi, W. P. Hu, T. Y. Lu, A. M. Garkhedkar, J. K. Vandavasi and J. J. Wang, *Org. Lett.*, 2015, **17**, 1521-1524.

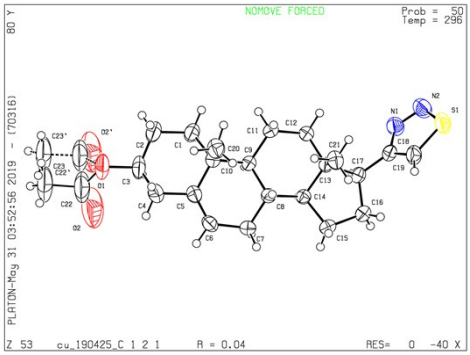
[3] A. Kumar, M. K. Muthyala, S. Choudhary, R. K. Tiwari and K. Parang, *J. Org. Chem.*, 2012, **77**, 9391-9396.

[4] J. Chen, Y. Jiang, J. T. Yu and J. Cheng, *J. Org. Chem.*, 2015, **81**, 271-275.

[5] M. Abramov, W. Dehaen, B. D'hooge, M. Petrov, S. Smeets, S. Toppet and M. Voets, *Tetrahedron*, 2000, **56**, 3933-3940.

[6] N. Lardiés, I. Romeo, E. Cerrada, M. Laguna and P. J. Skabara, *Dalton Trans.*, 2007, 5329-5338.

5. Crystallographic data and molecular structure of 6s



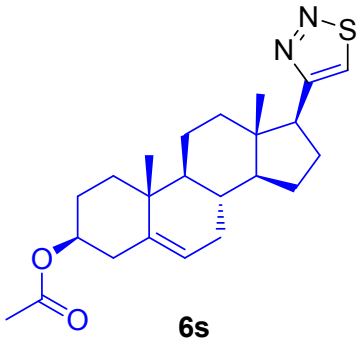


***Figure S1*.** X-ray crystal structure of 6s.

Crystal Data for Compound 6s: CCDC 1919871 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic.

Datablock: cu_190425_0m

Bond precision: C-C = 0.0038 A Wavelength=1.54178

Cell:	a=9.6526(5)	b=7.4265(4)	c=30.2283(16)
	alpha=90	beta=95.334(2)	gamma=90
Temperature:	296 K		

	Calculated	Reported
Volume	2157.5(2)	2157.5(2)
Space group	C 2	C 1 2 1
Hall group	C 2y	C 2y
Moiety formula	C23 H32 N2 O2 S	C23 H32 N2 O2 S
Sum formula	C23 H32 N2 O2 S	C23 H32 N2 O2 S
Mr	400.57	400.56
Dx,g cm-3	1.233	1.233
Z	4	4
Mu (mm-1)	1.486	1.486
F000	864.0	864.0
F000'	867.52	
h,k,lmax	11,9,37	11,9,37
Nref	4212[2274]	3764
Tmin,Tmax	0.837,0.985	0.323,0.753
Tmin'	0.837	

Correction method= # Reported T Limits: Tmin=0.323 Tmax=0.753
AbsCorr = MULTI-SCAN

Data completeness= 1.66/0.89 Theta(max)= 71.385

R(reflections)= 0.0408(3729) wR2(reflections)= 0.1140(3764)

S = 1.070 Npar= 268

The following ALERTS were generated. Each ALERT has the format
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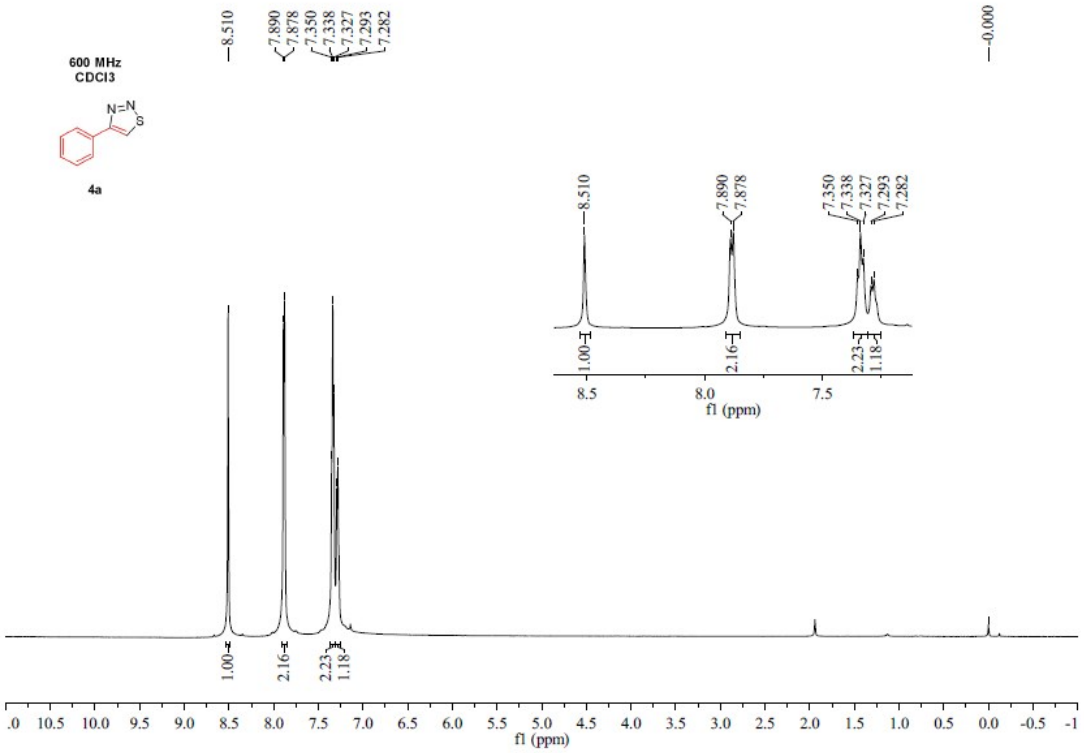
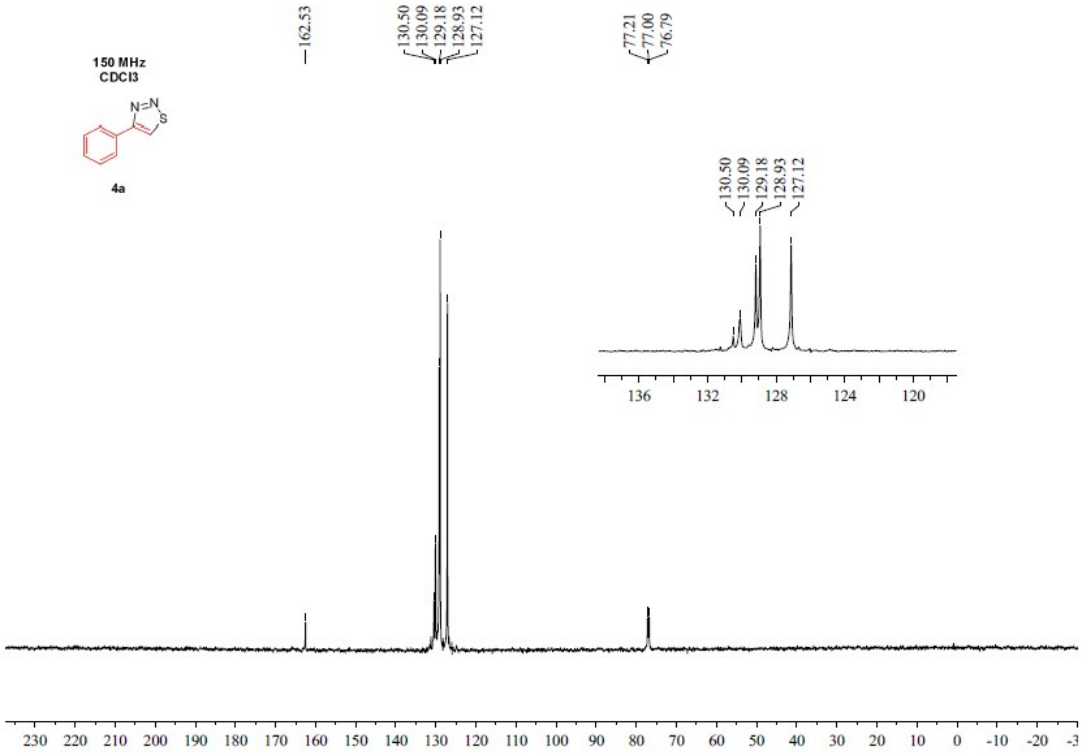
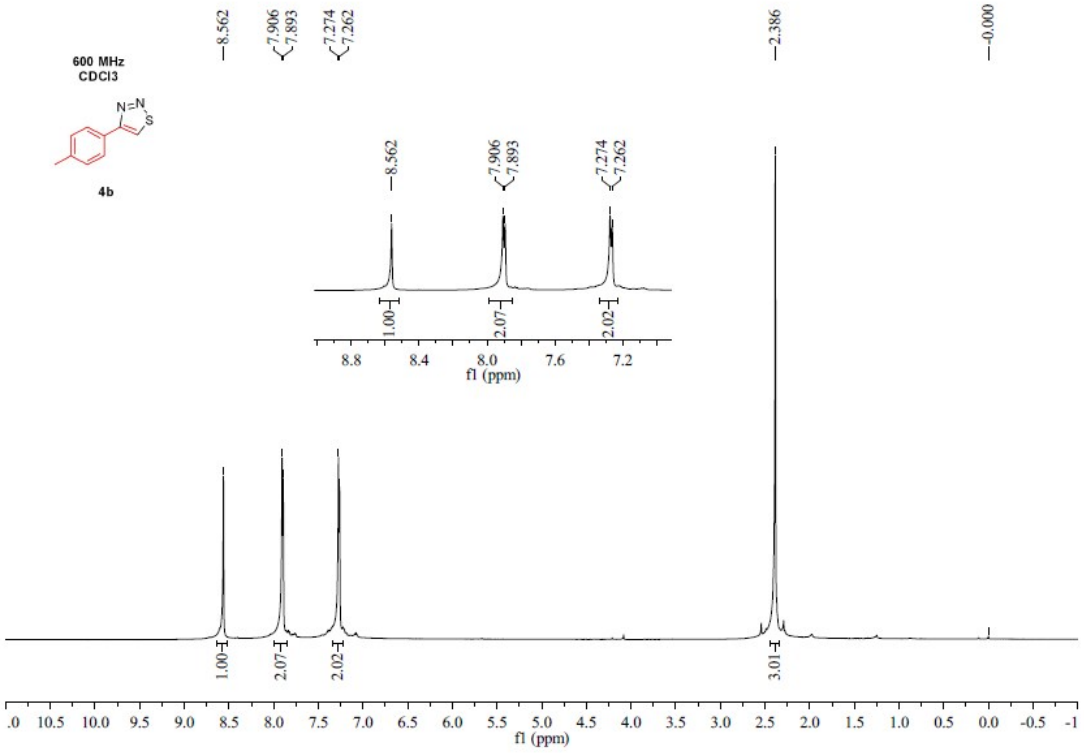
Click on the hyperlinks for more details of the test.

6. Copies of ^{1}H and ^{13}C NMR spectra



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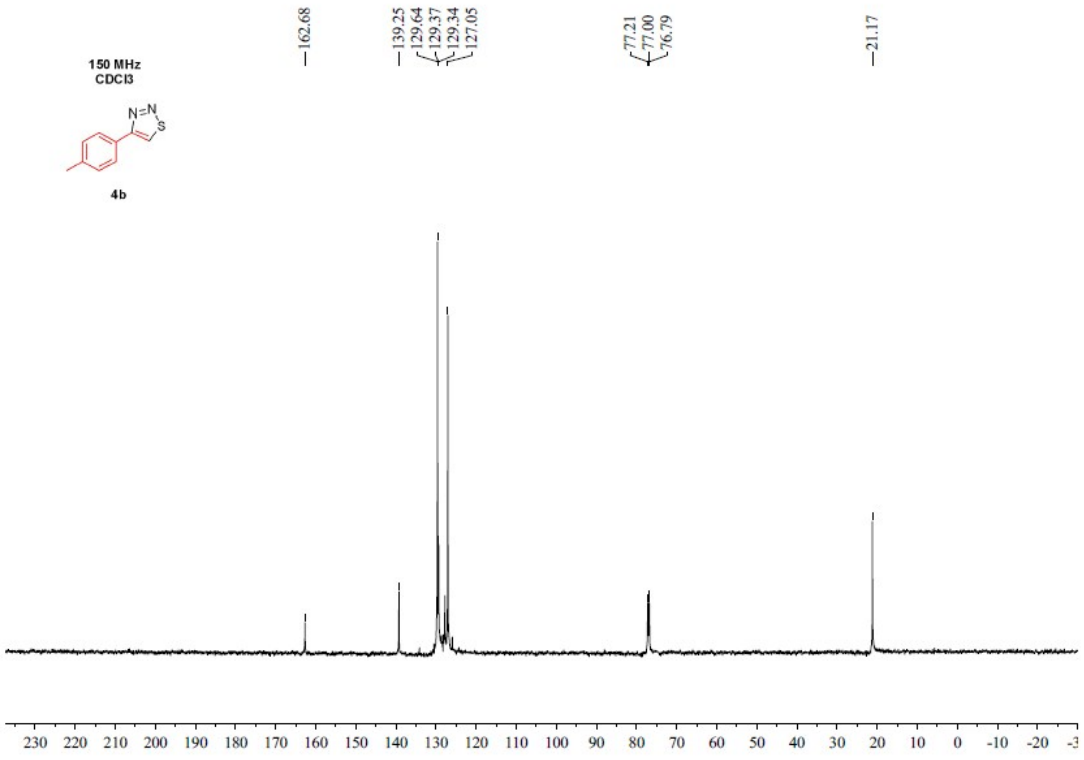
2.02
3.01
f1 (ppm)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77.00
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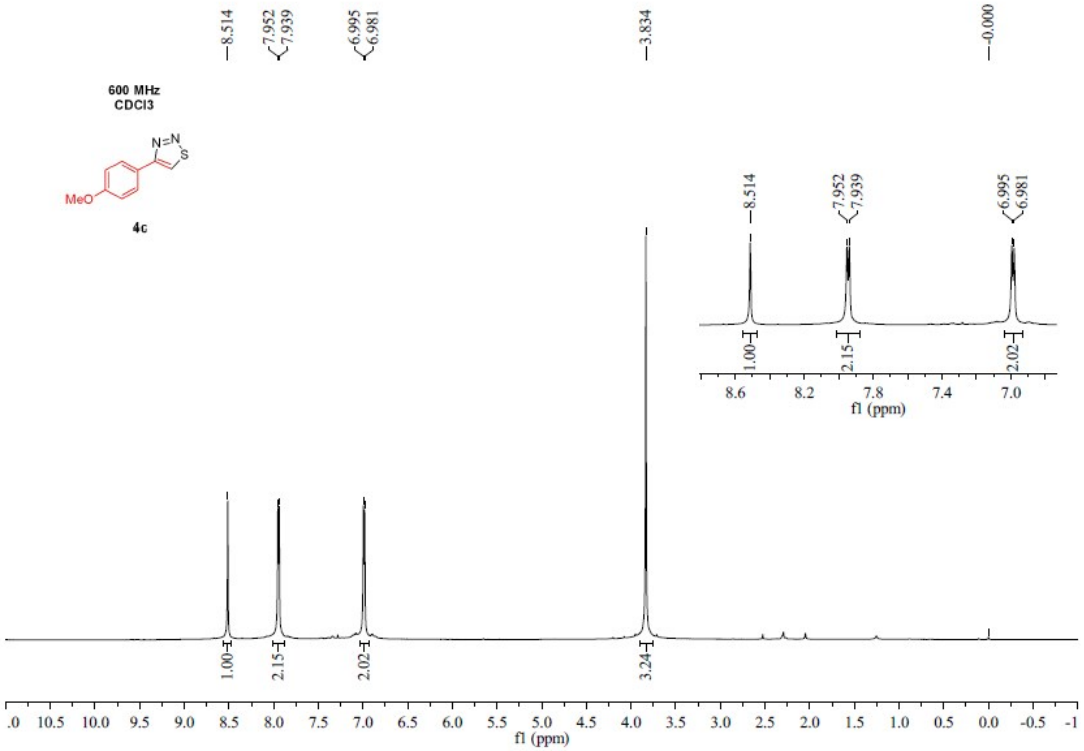
21.17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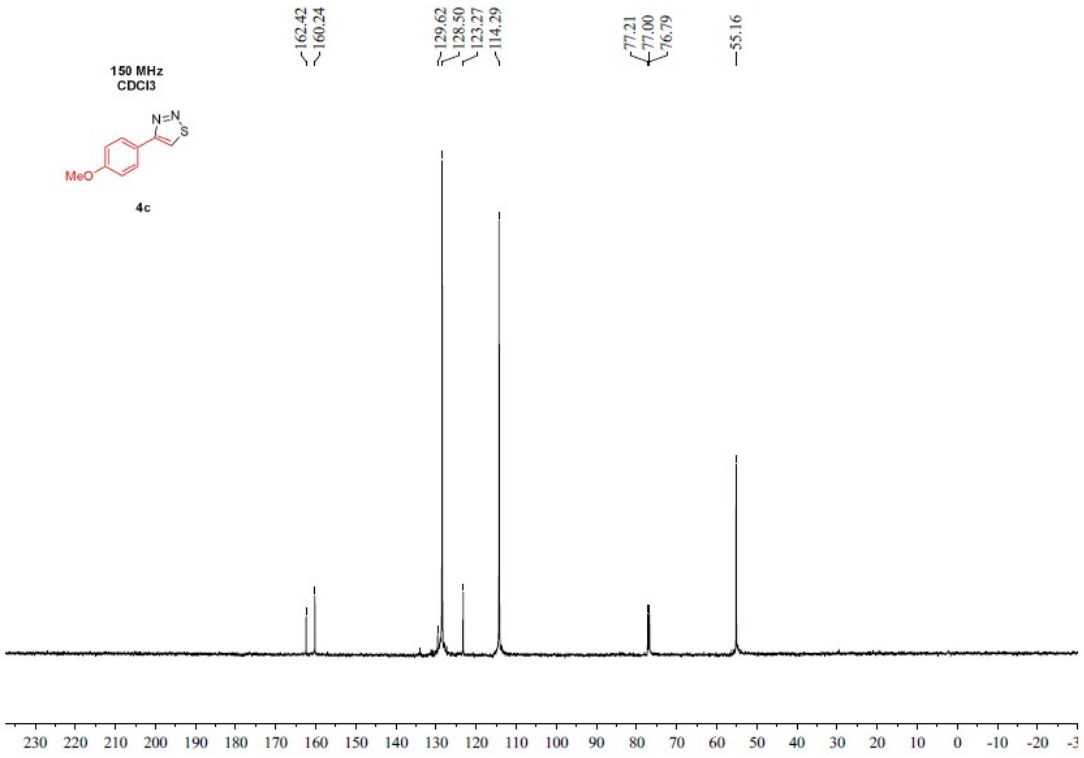
3.24
f1 (ppm)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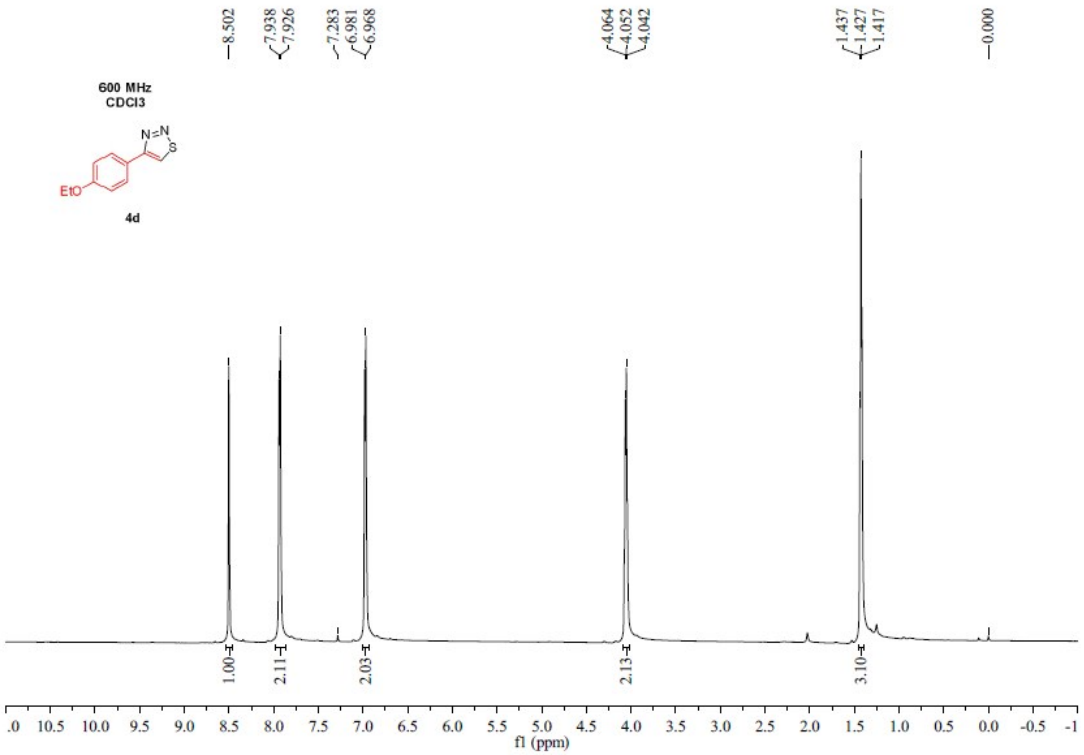
4c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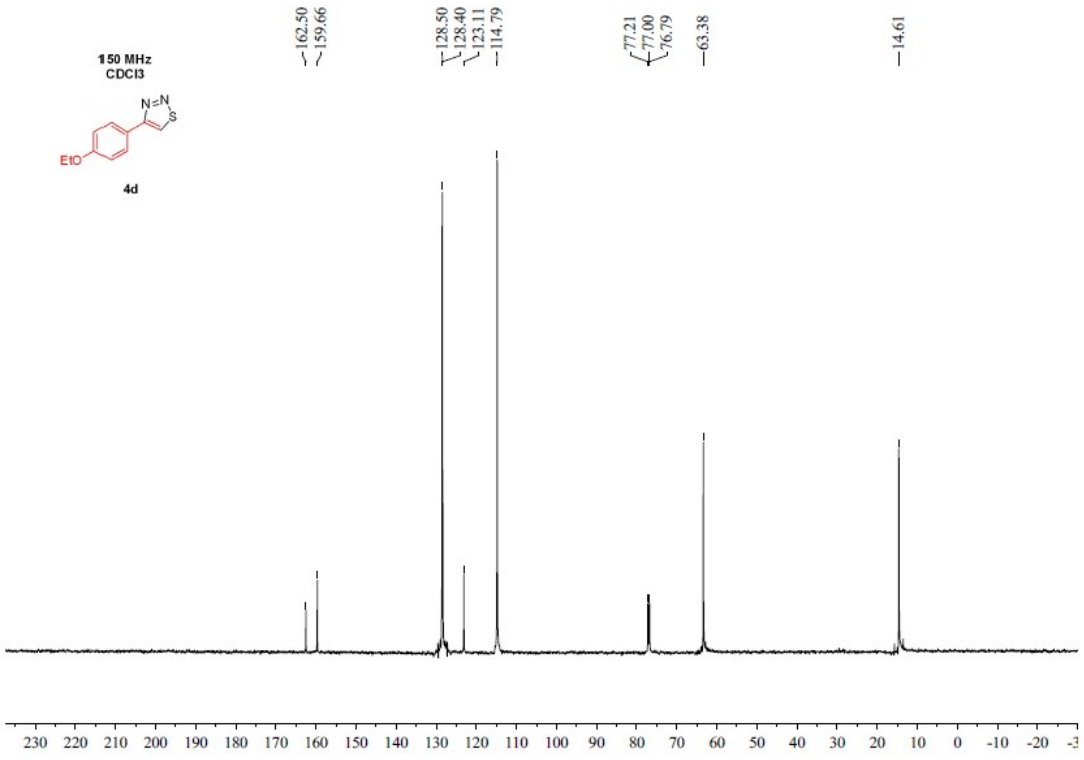
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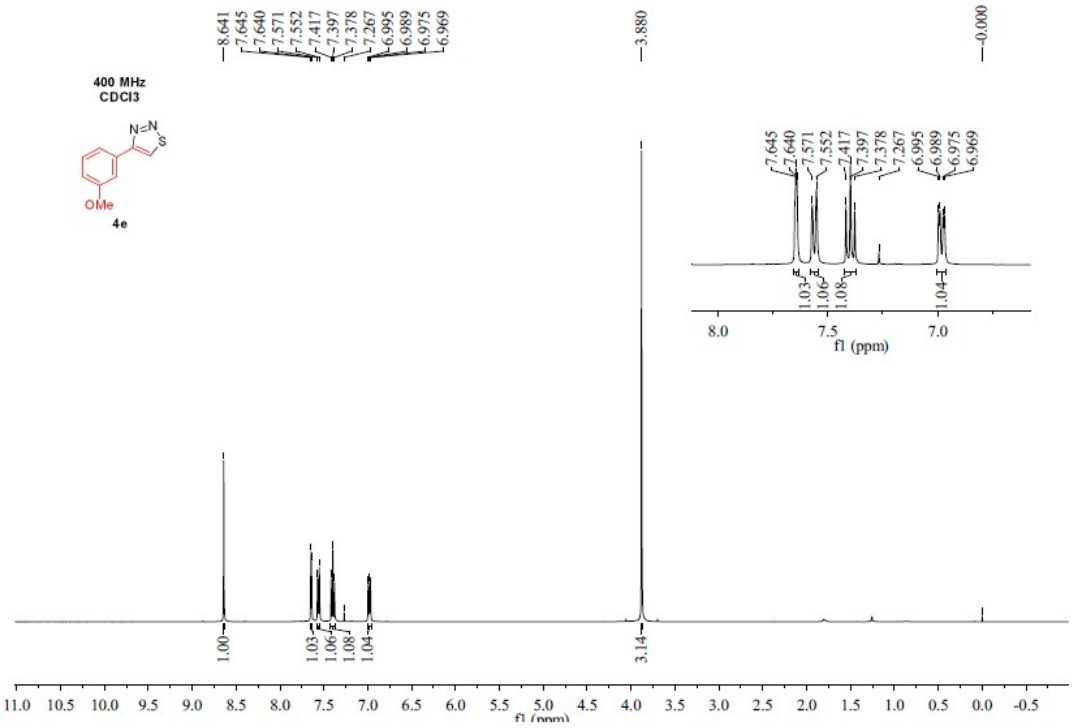
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6.969
3.880
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OMe
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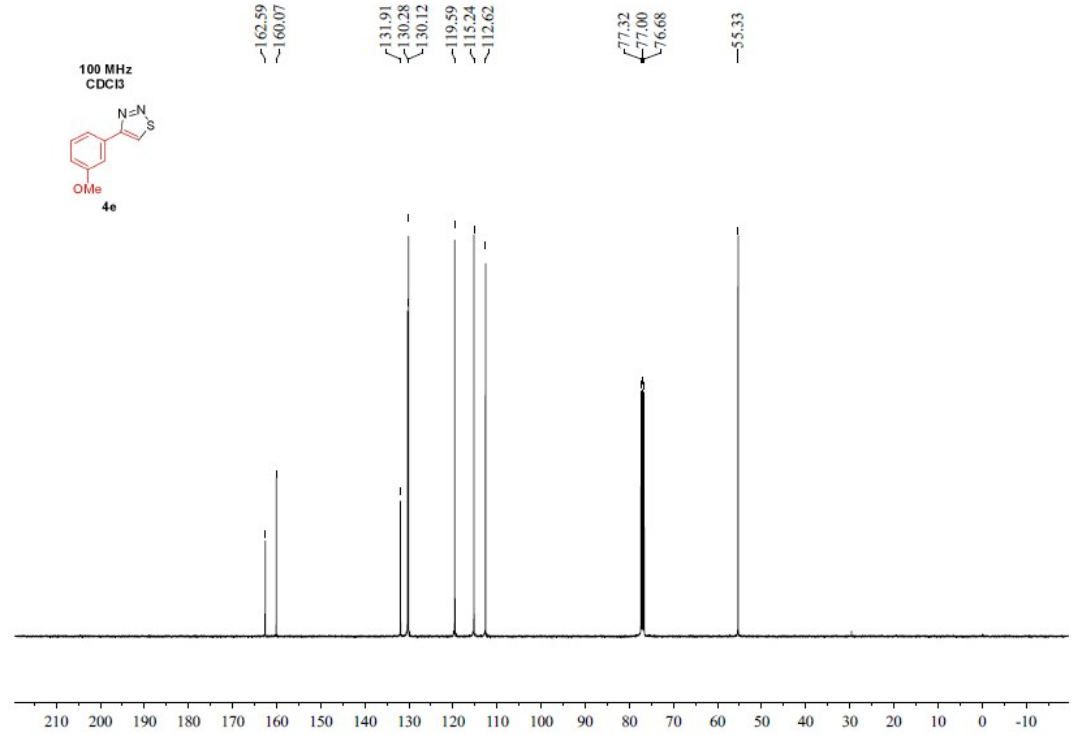
3.14
f1 (ppm)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55.33
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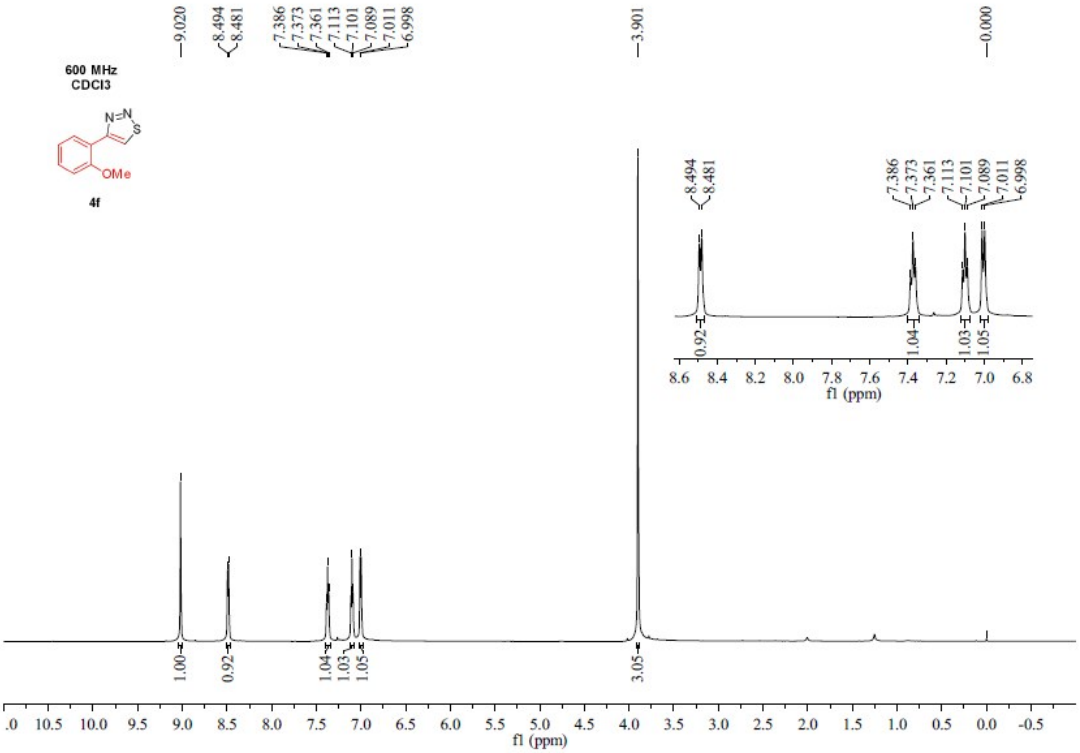
CDCl3
OMe
4e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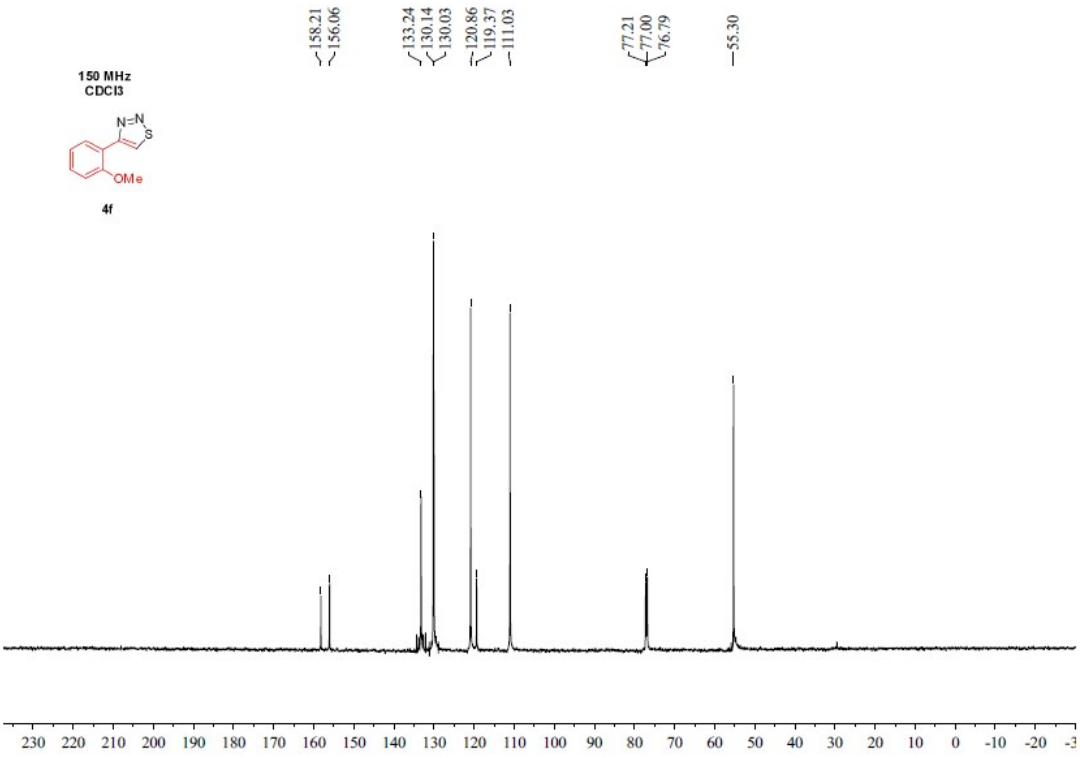
3.05
f1 (ppm)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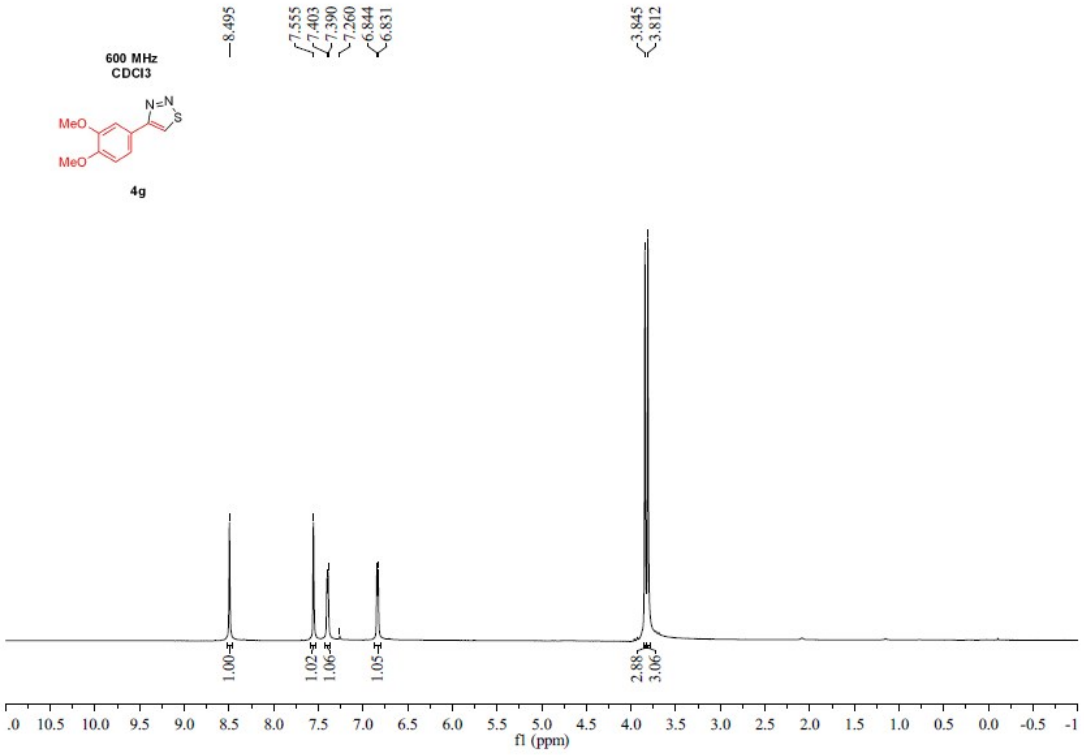
76.79
55.30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3.06
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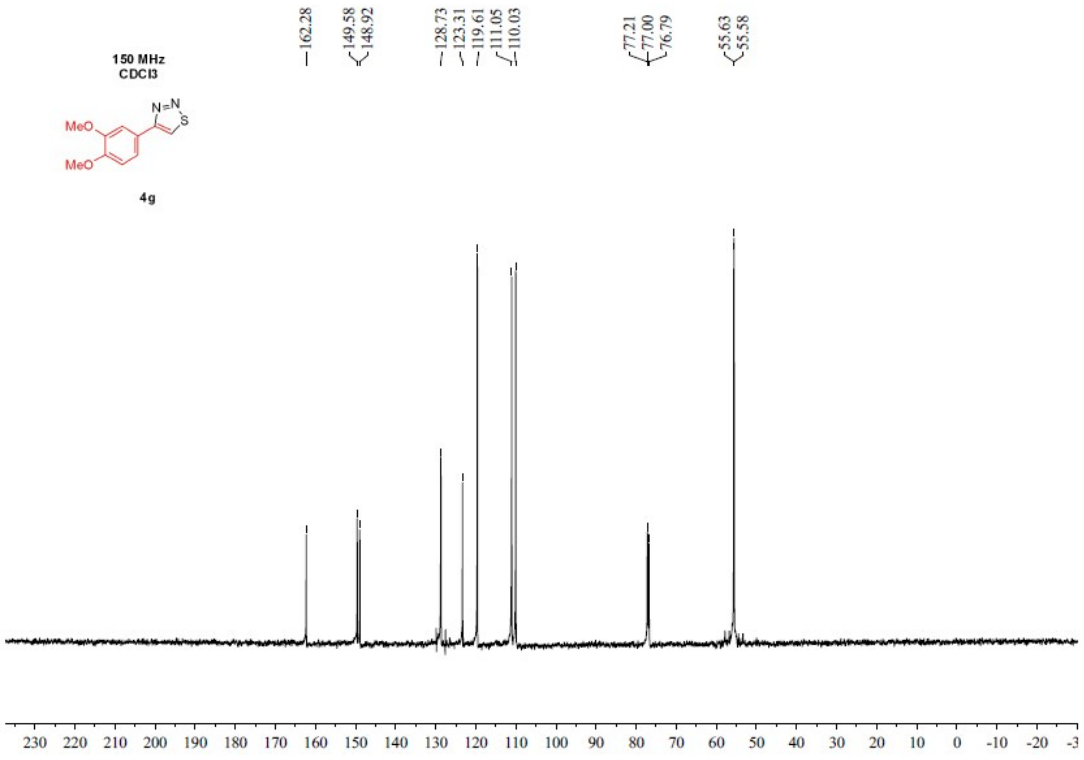
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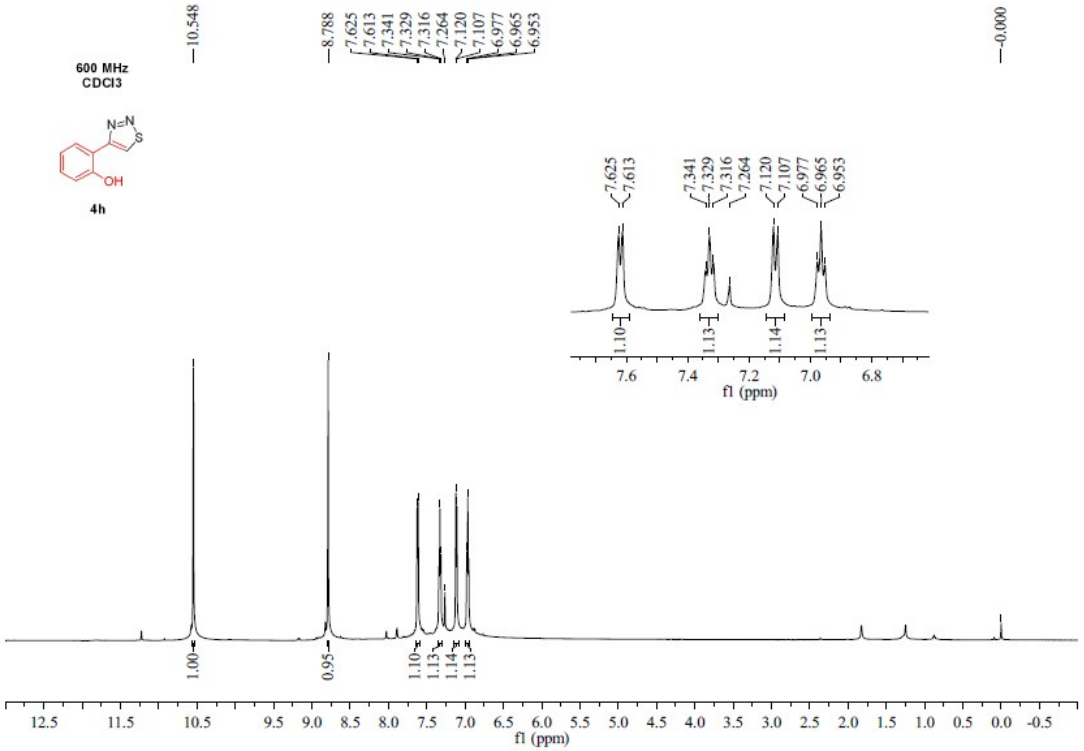
55.63
55.58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0.95
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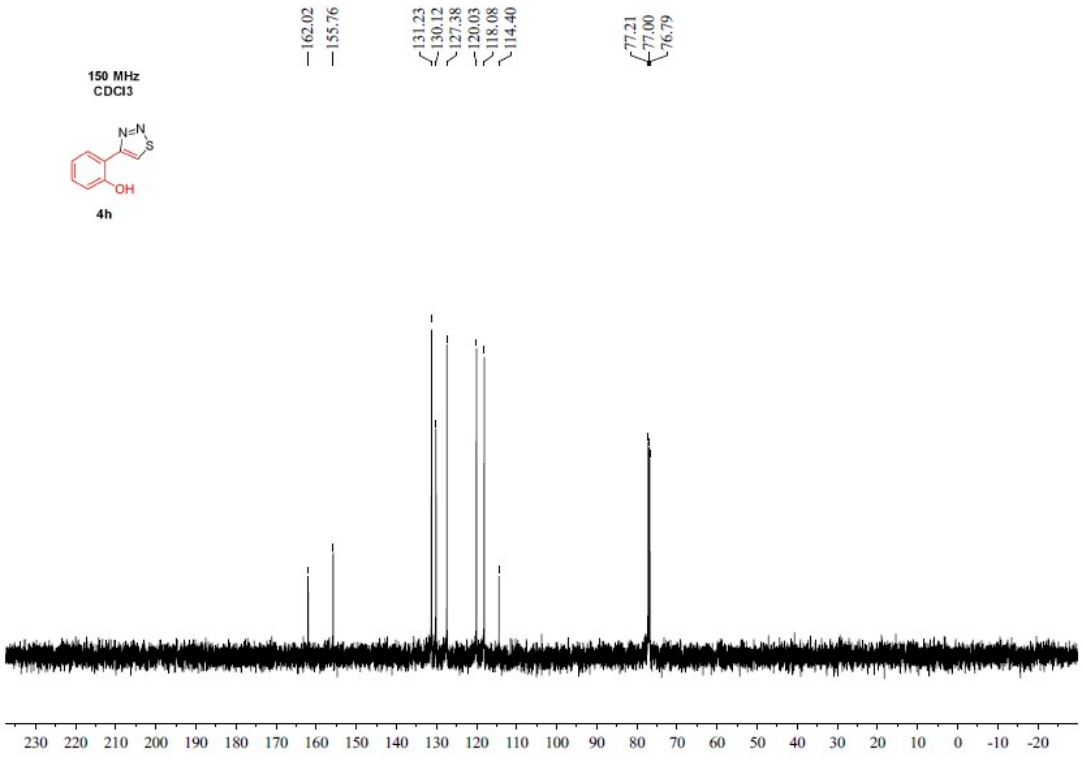
1.13
1.14
1.13
fl (ppm)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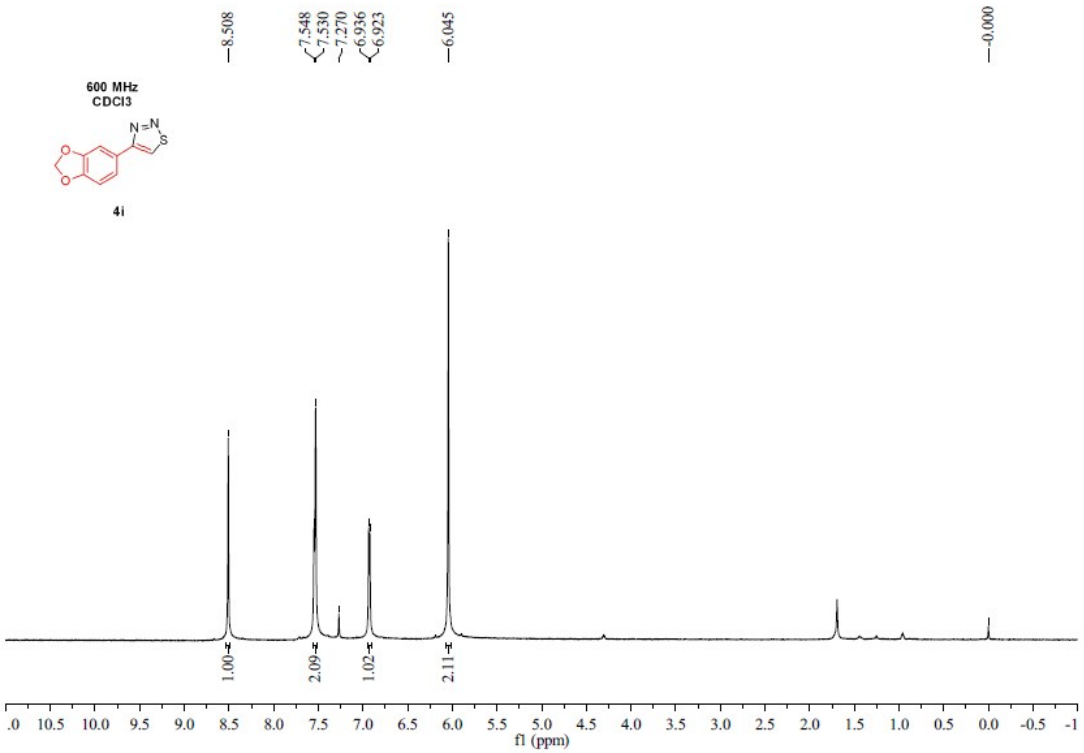
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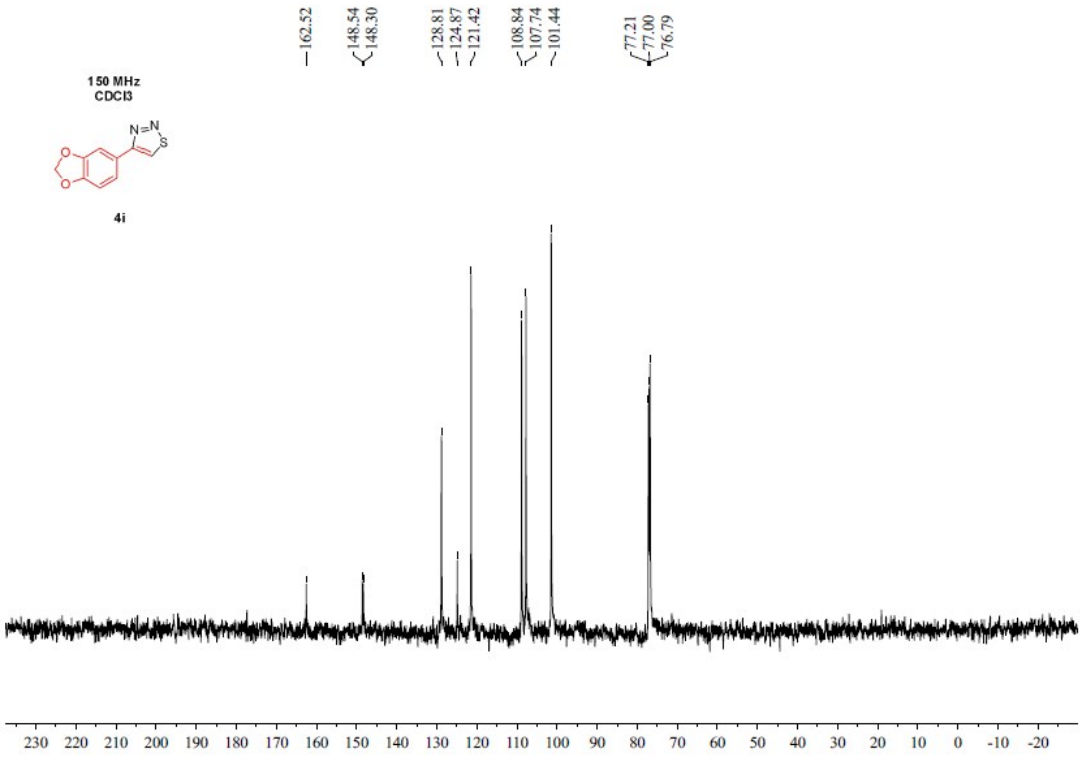
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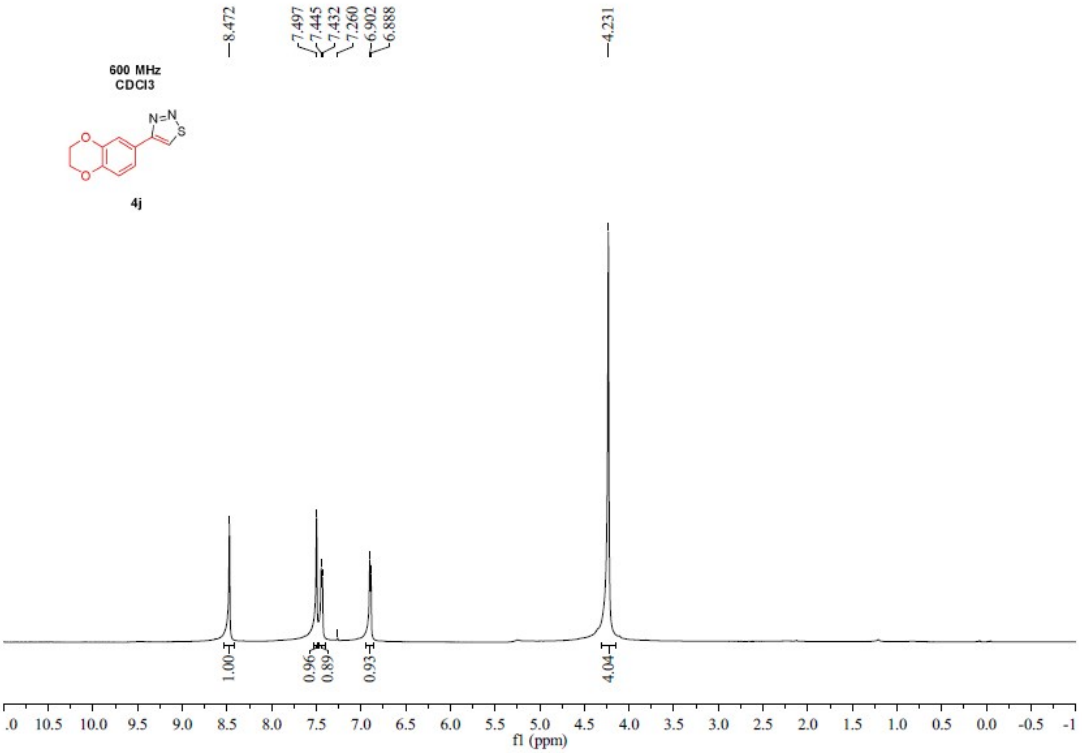
4i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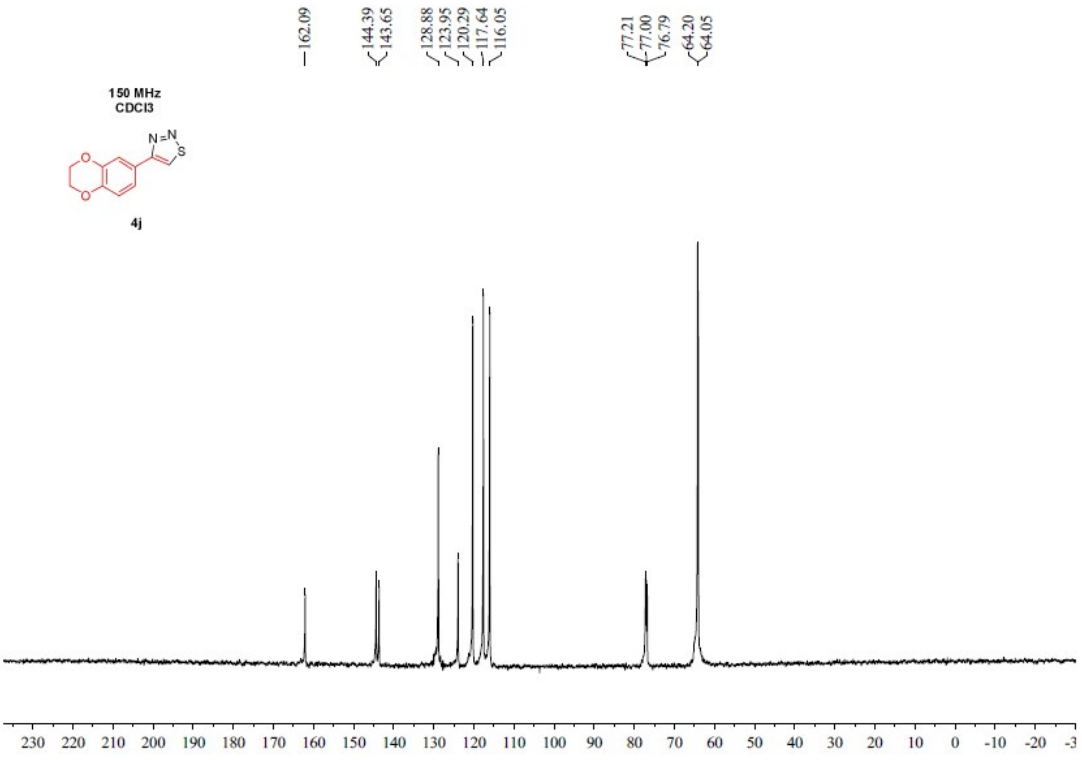
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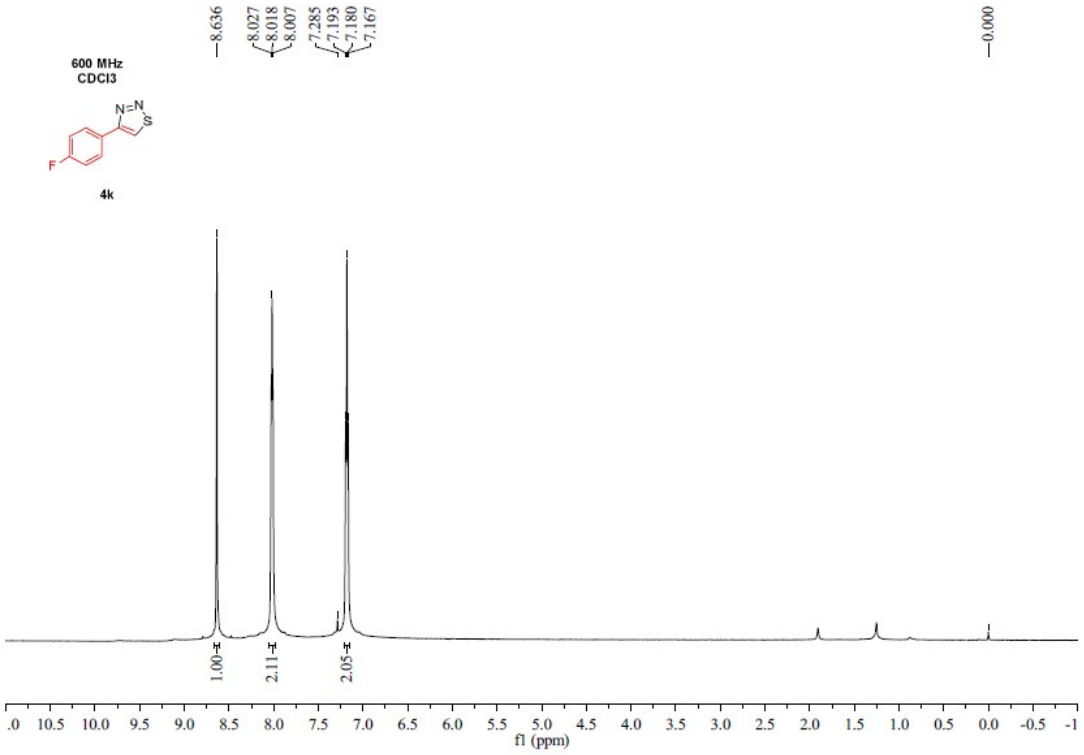
64.05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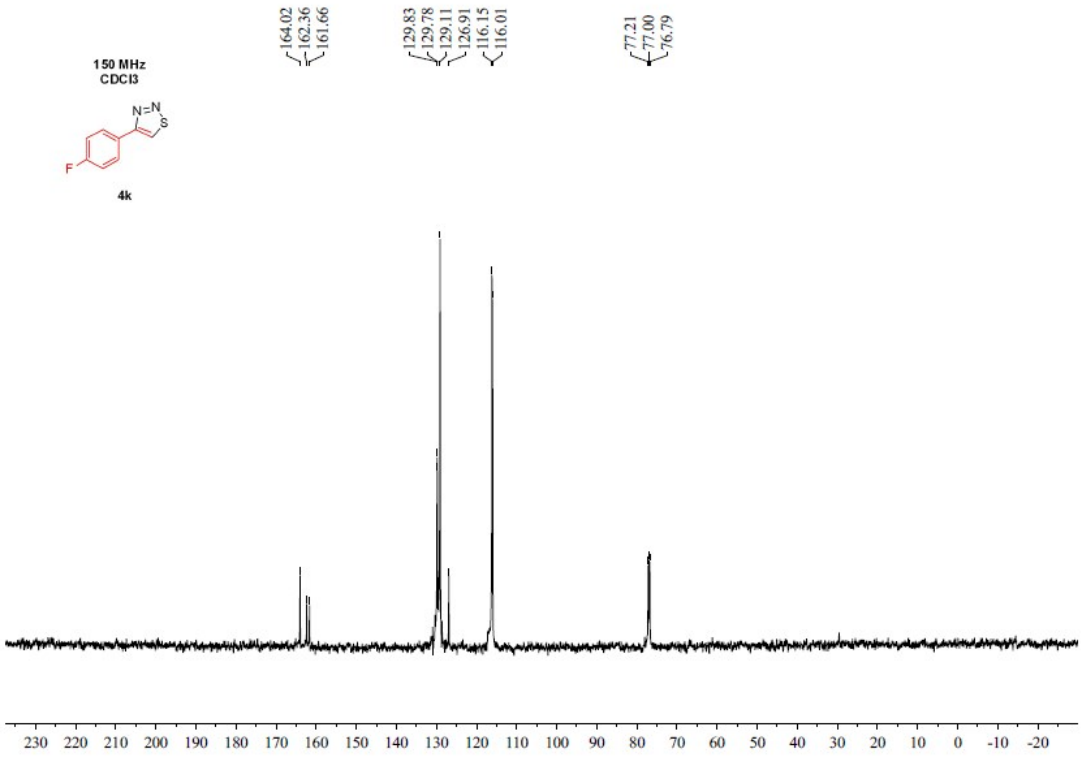
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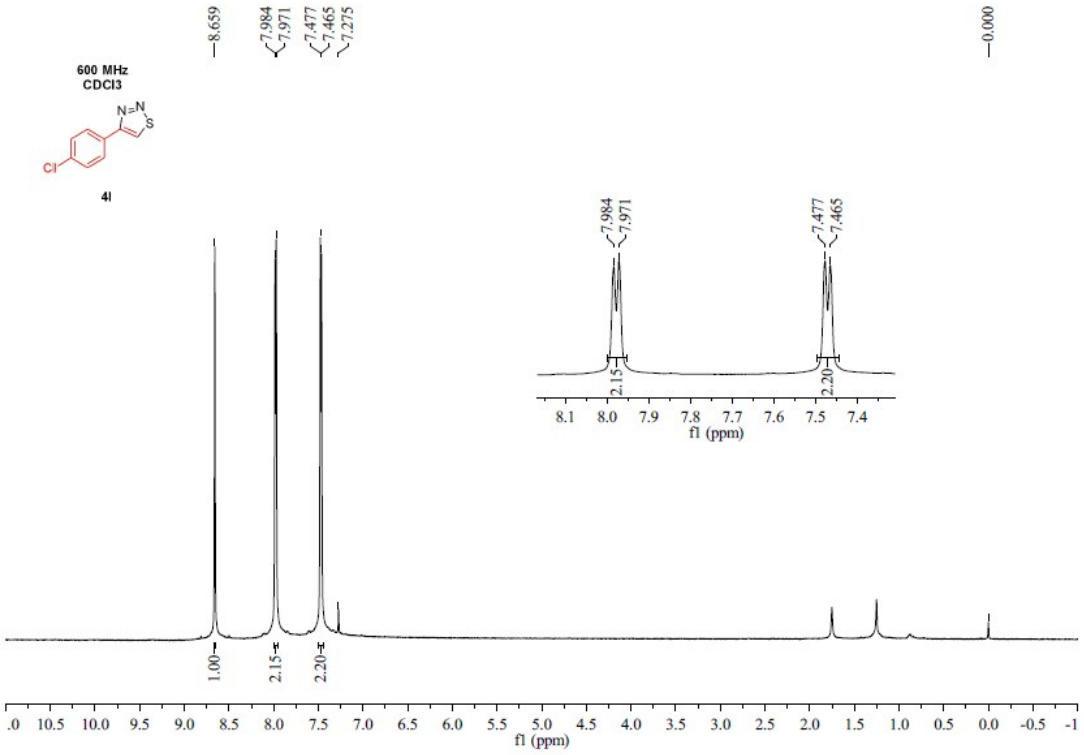
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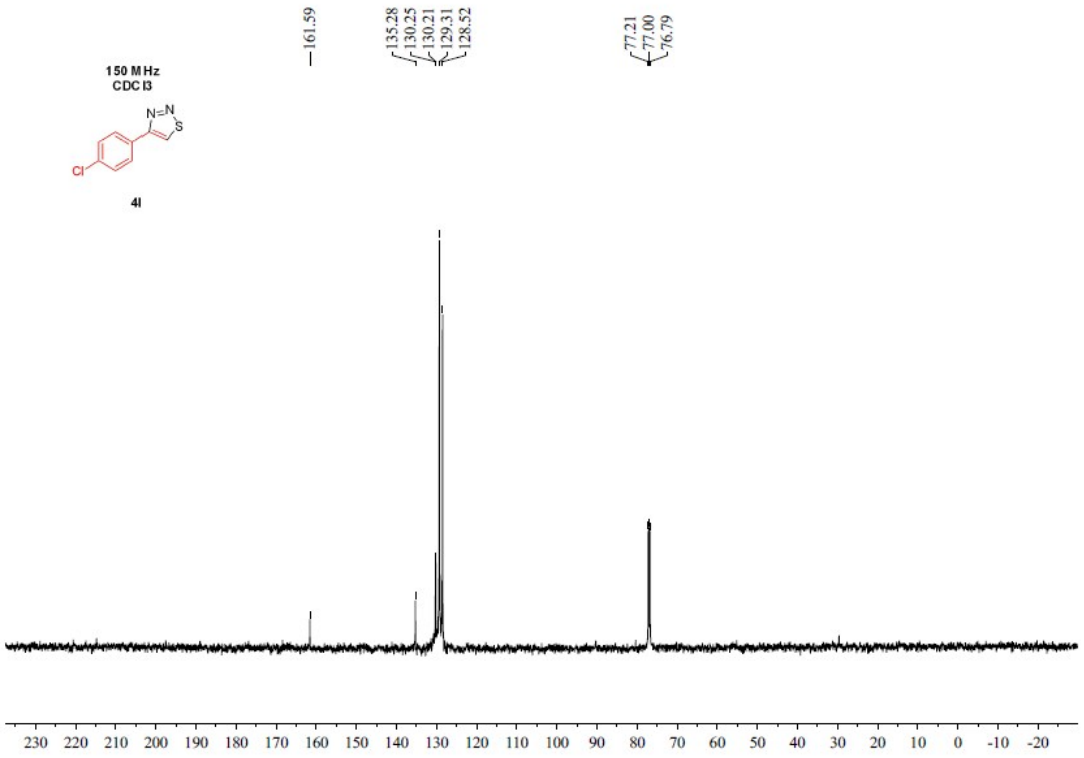
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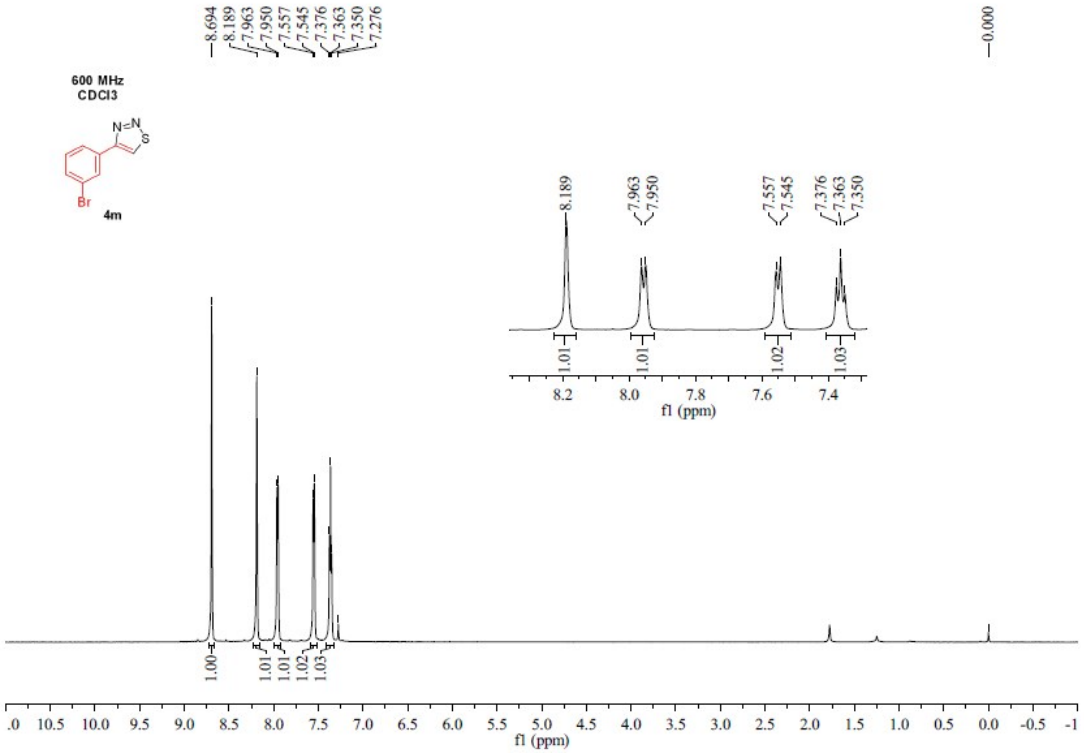
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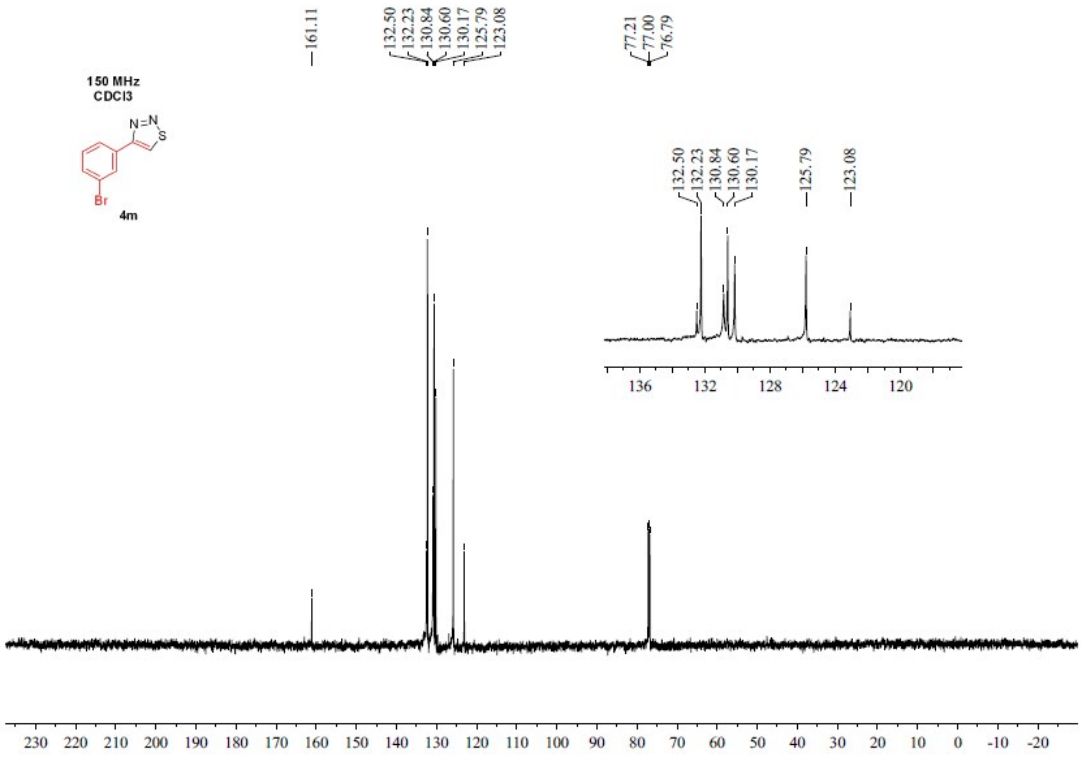
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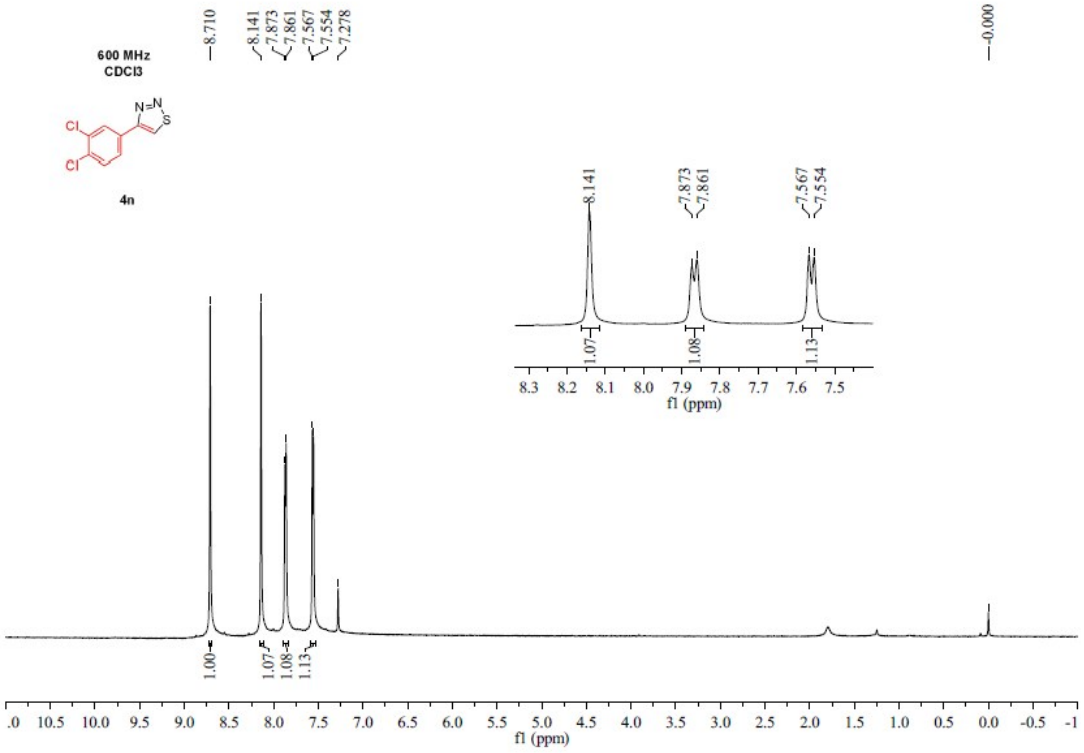
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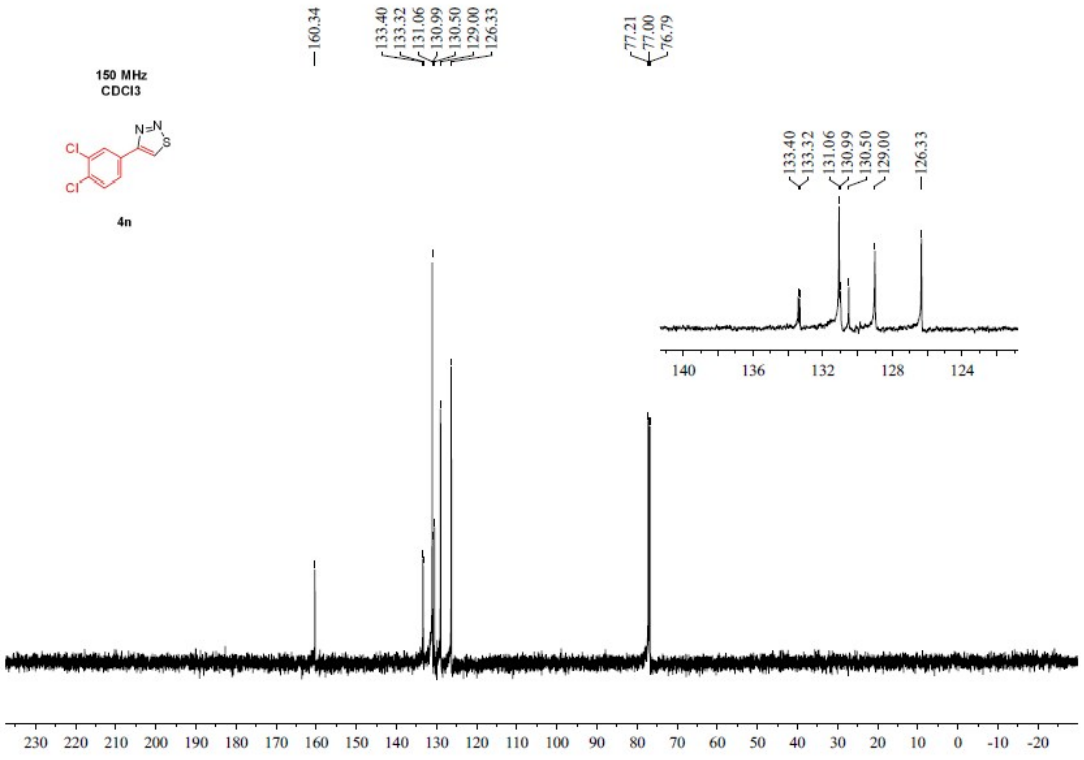
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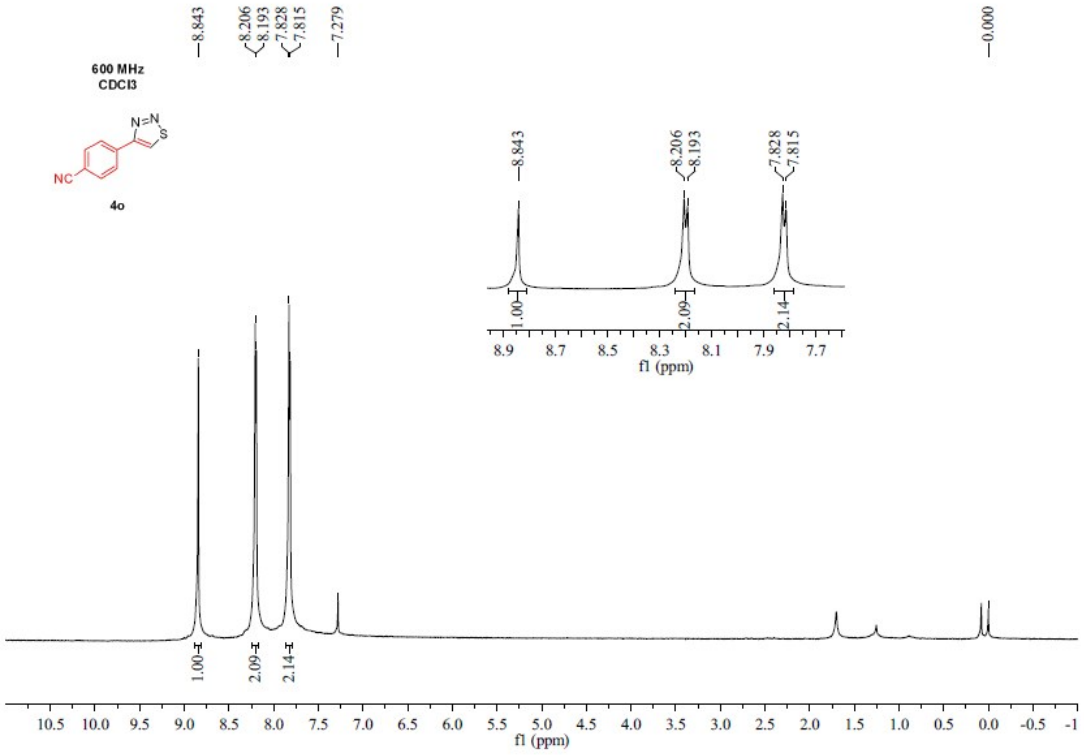
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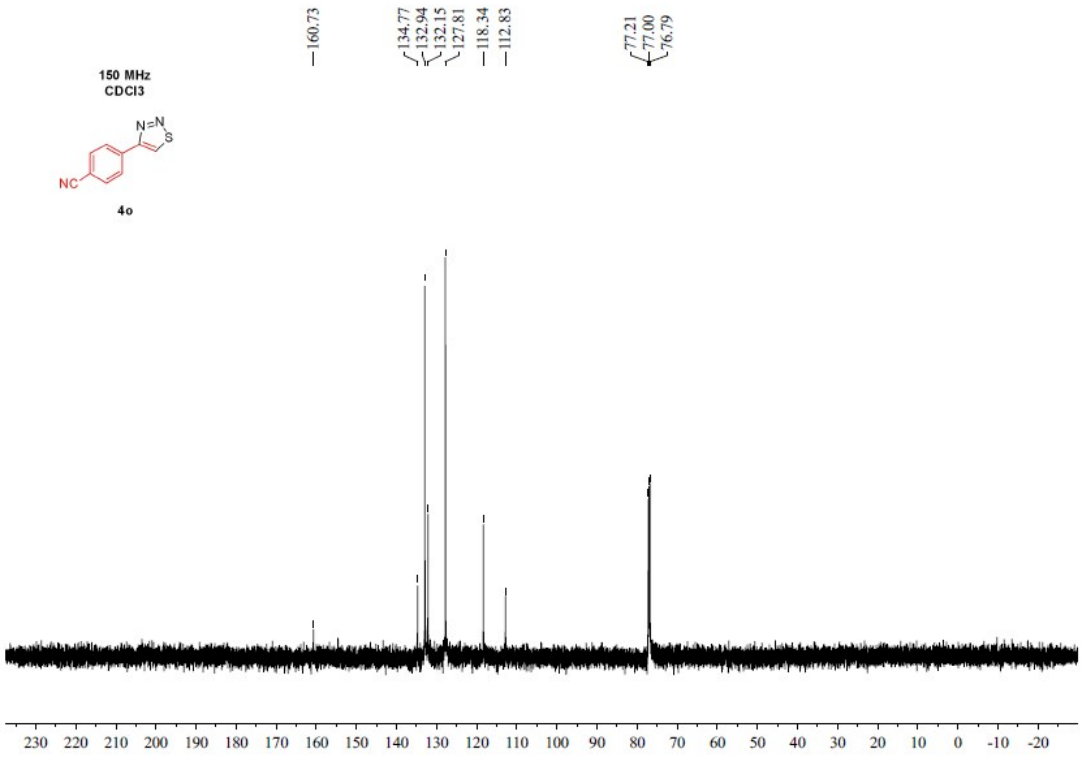
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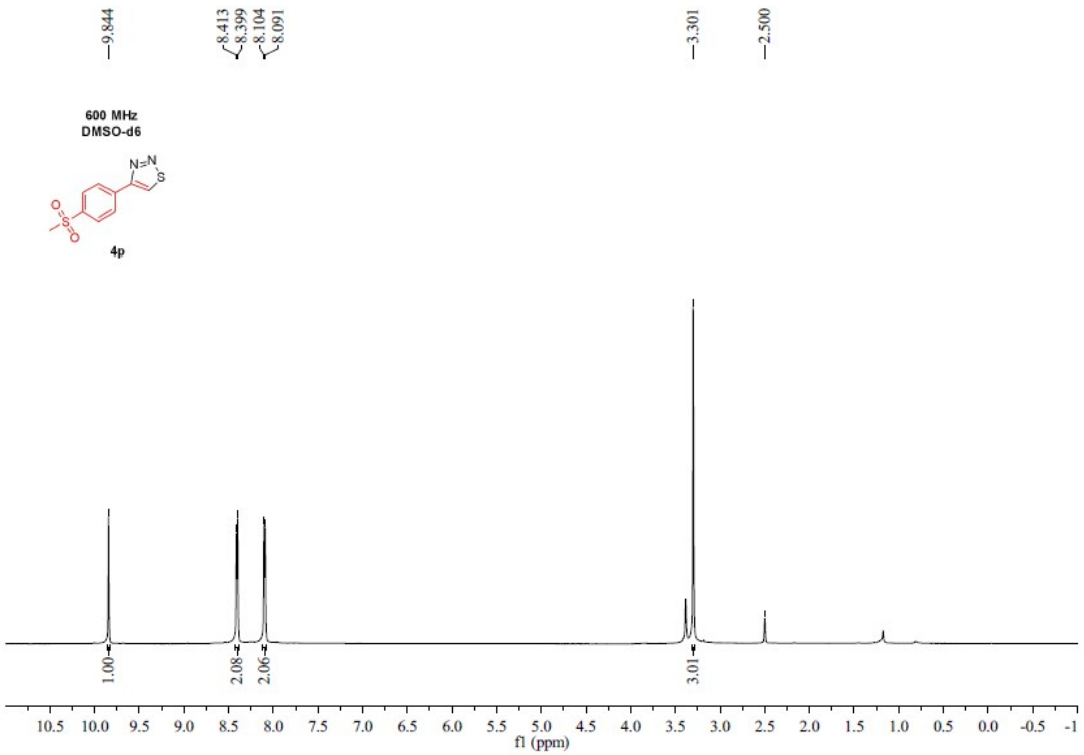
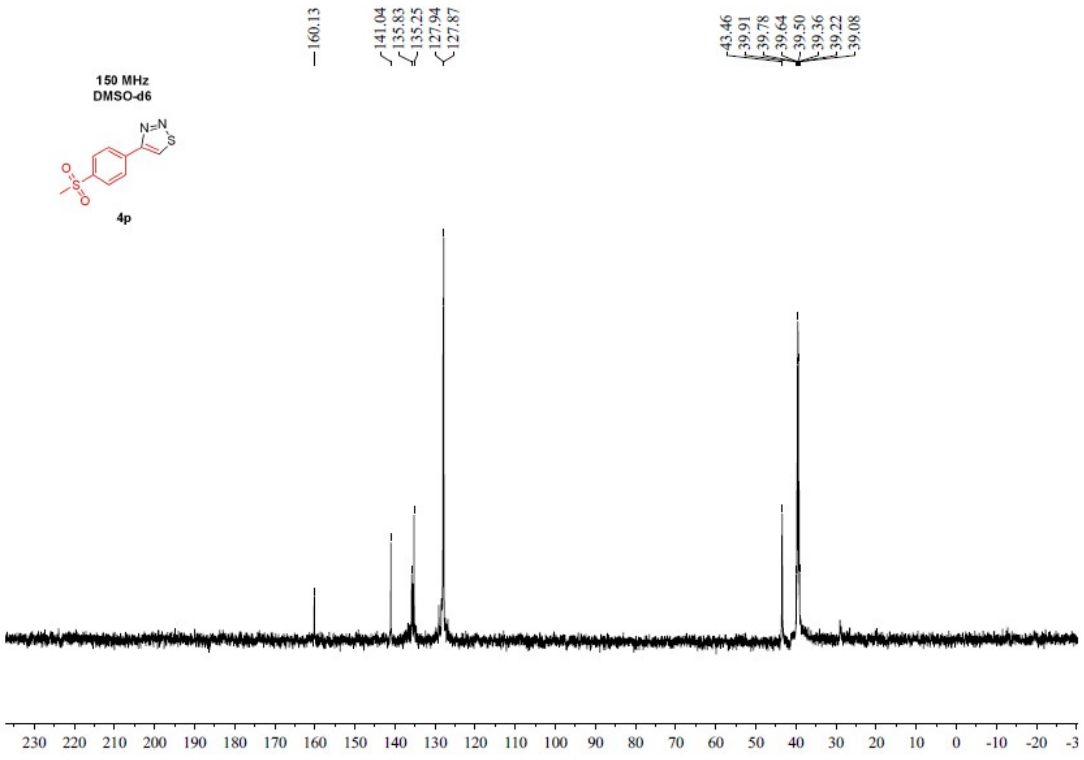
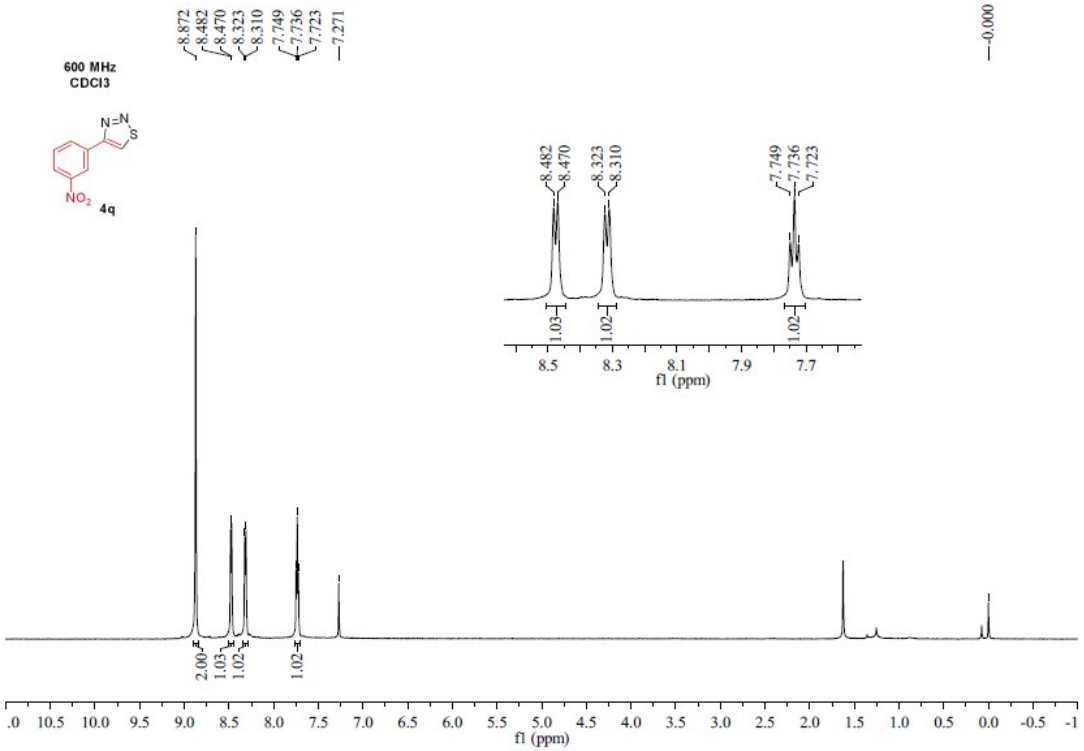
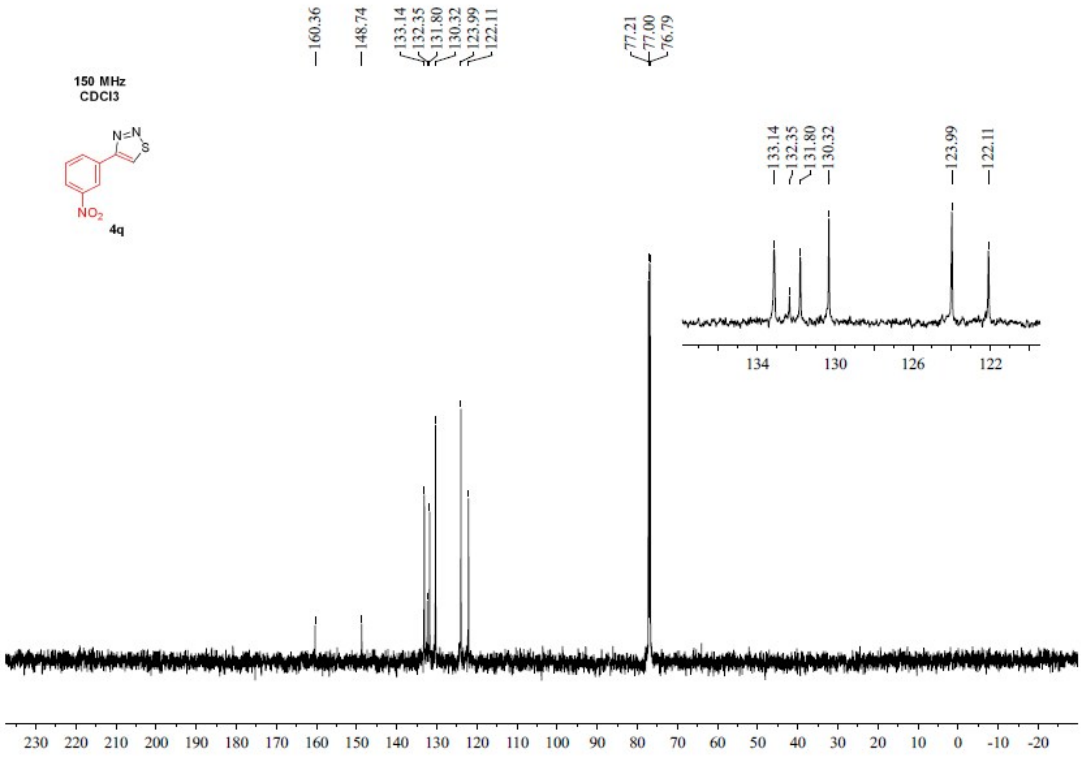
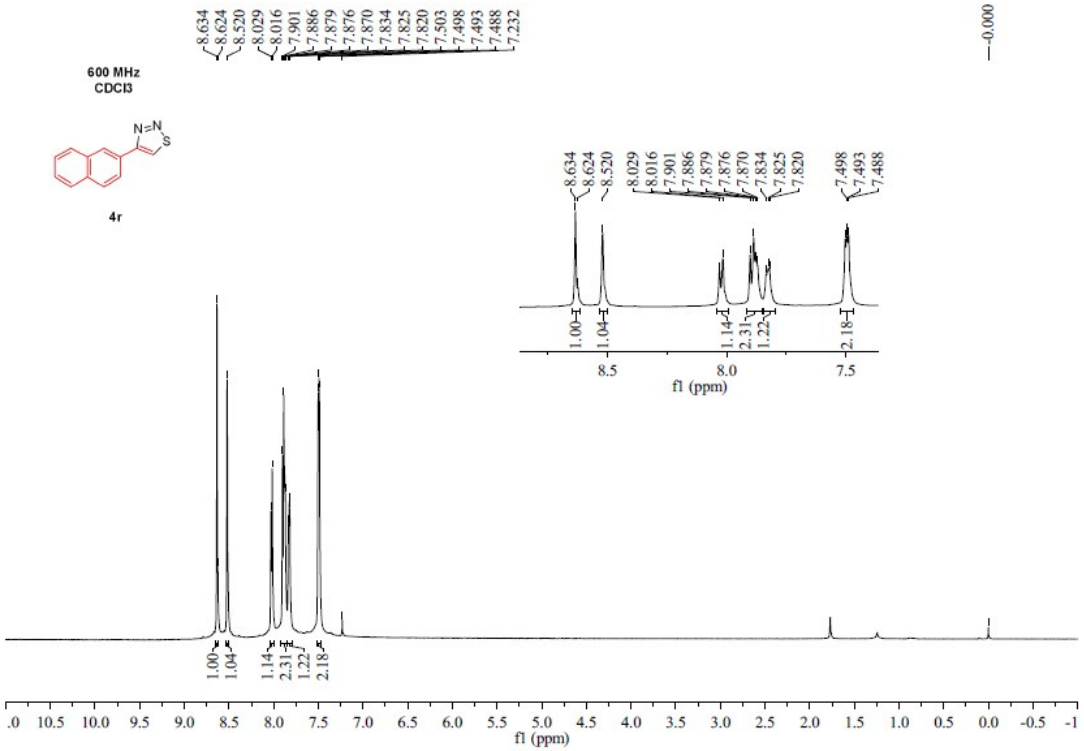
CDCl3
4o

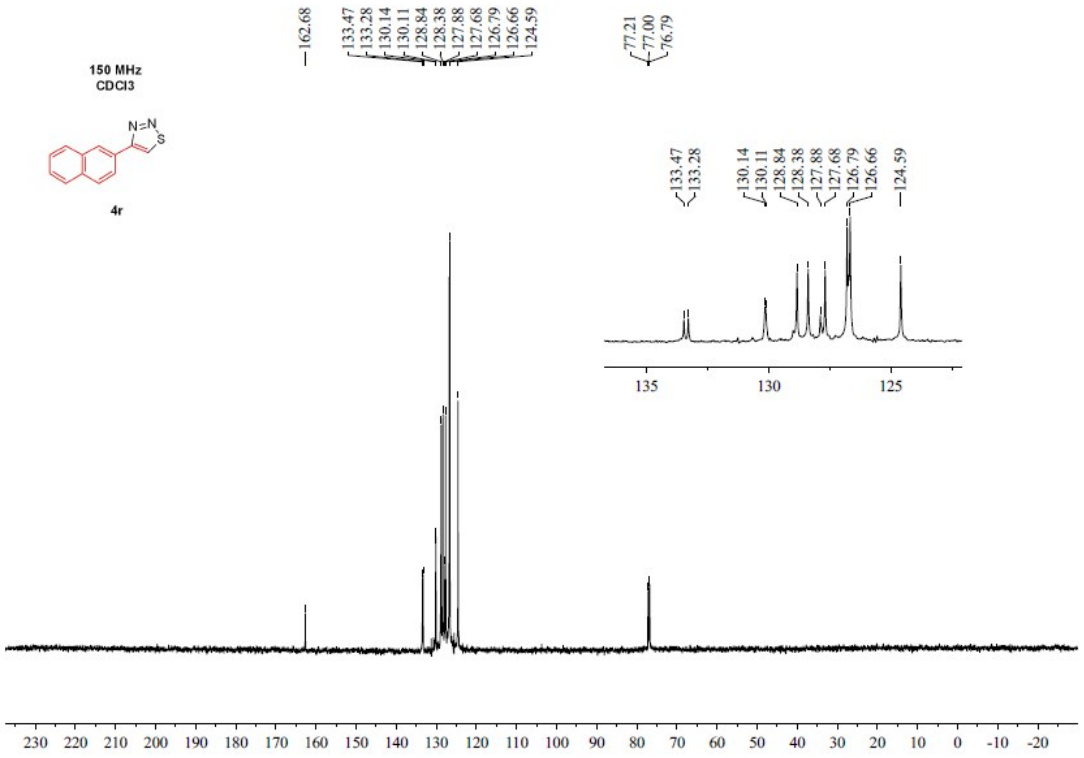

9.844
8.413
8.399
8.104
8.091
3.301
2.500
600 MHz
DMSO-d6
4p
1.00
2.08
2.06
3.01
f1 (ppm)

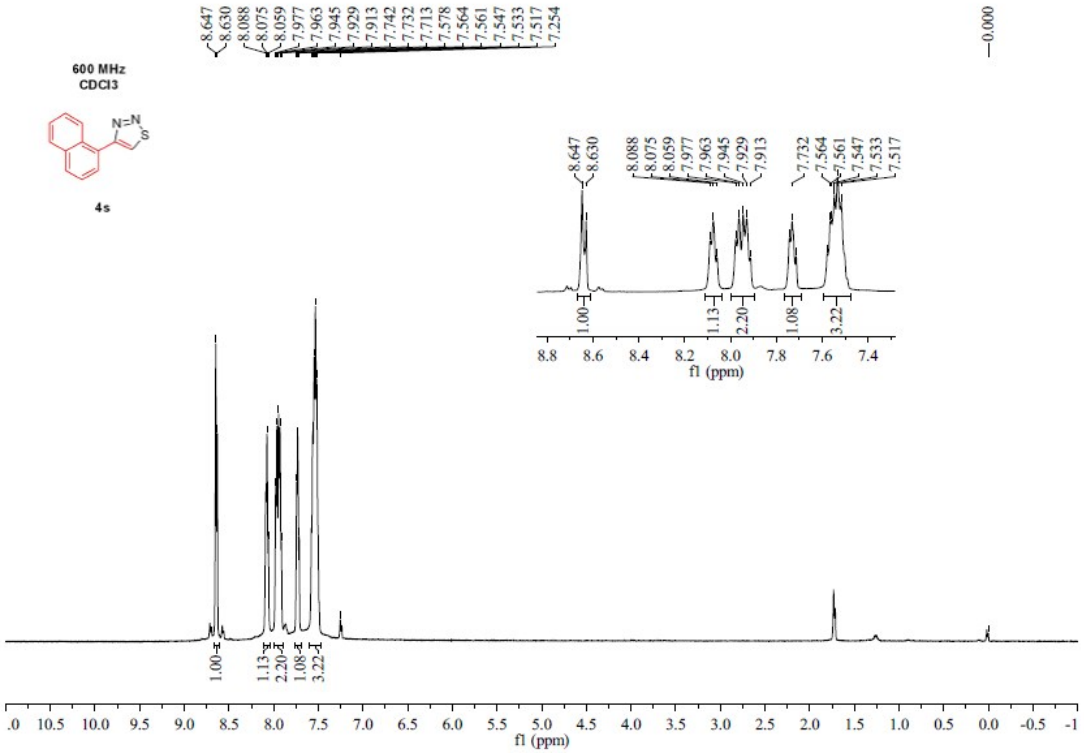

160.13
141.04
135.83
135.25
127.94
127.87
43.46
39.91
39.78
39.64
39.50
39.36
39.22
39.08
150 MHz
DMSO-d6
4p

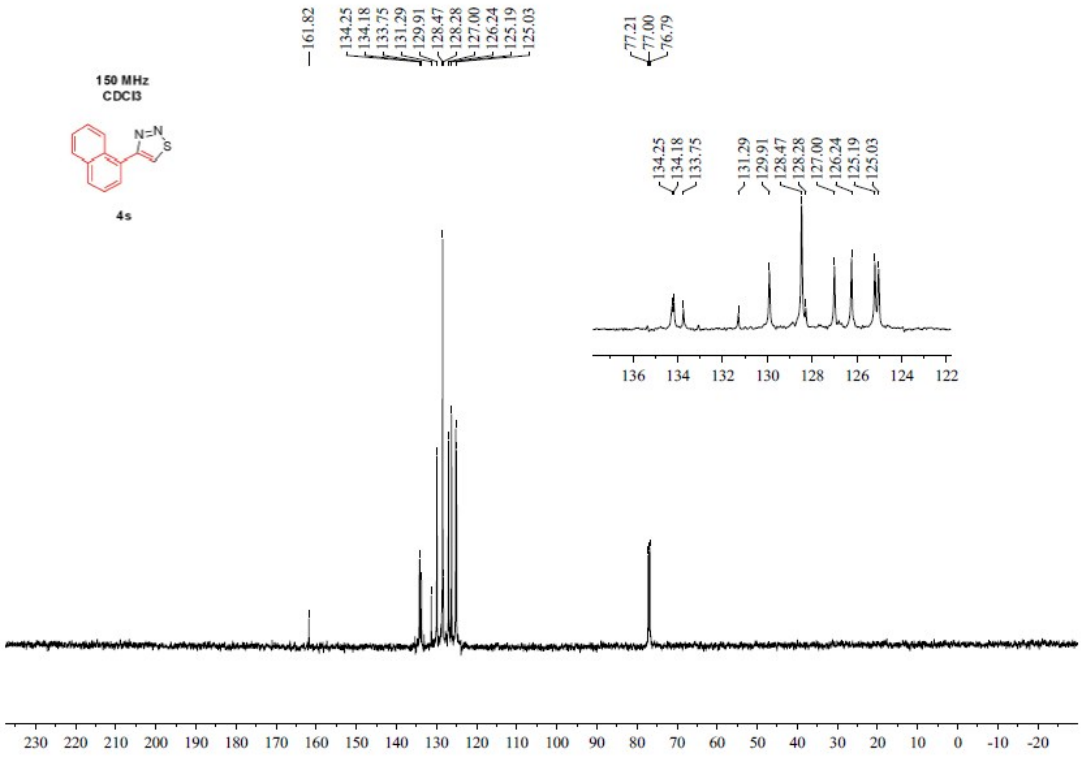

600 MHz
CDCl3
4q
8.872
8.482
8.470
8.323
8.310
7.749
7.736
7.723
7.271
0.000
2.00
1.03
1.02
1.02
f1 (ppm)

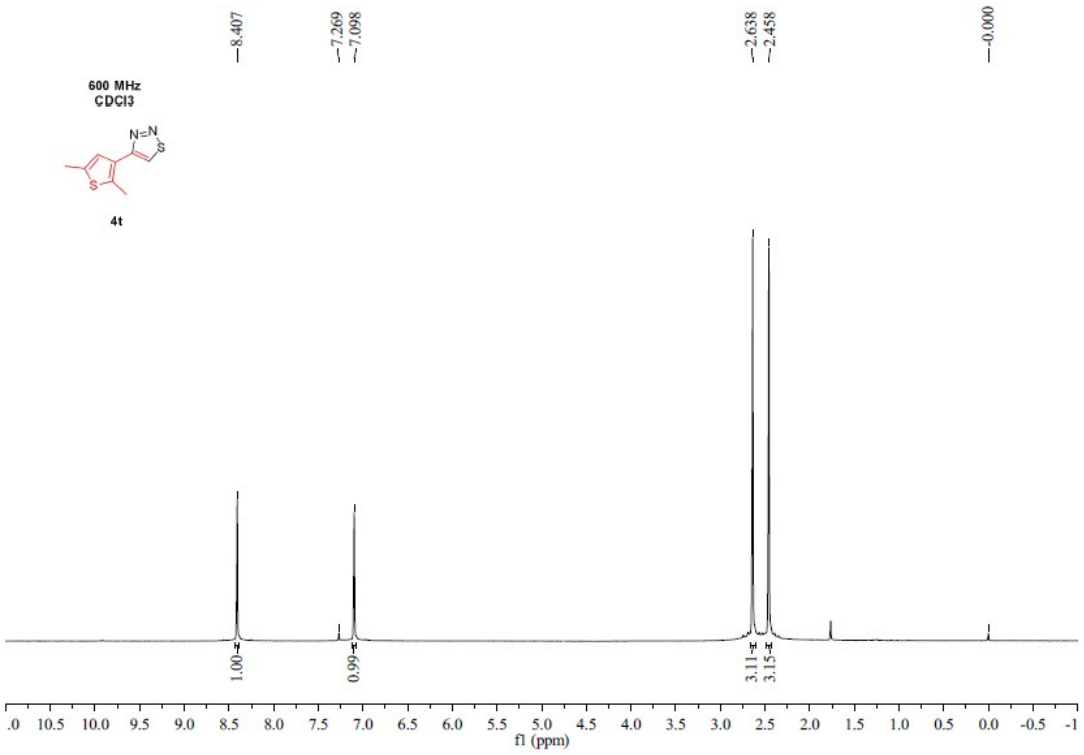

150 MHz
CDCl3
4q
160.36
148.74
133.14
132.35
131.80
130.32
123.99
122.11
77.21
77.00
76.79
f1 (ppm)

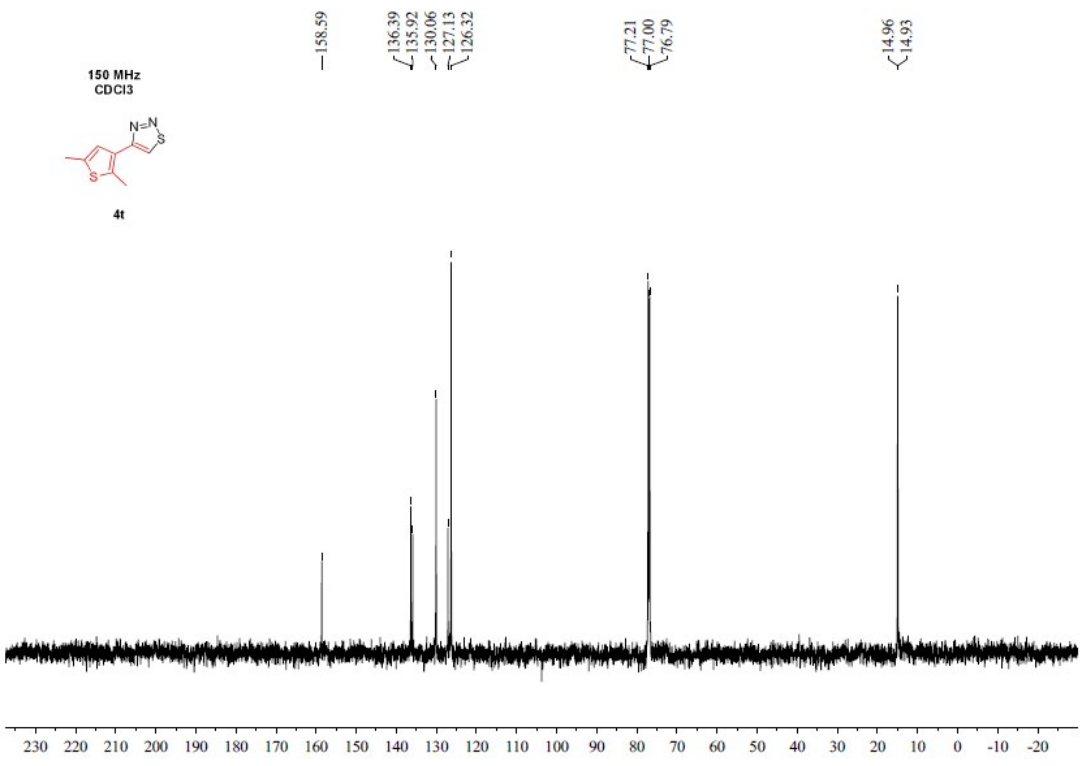

600 MHz
CDCl3
4r
8.634
8.624
8.520
8.029
8.016
7.901
7.886
7.879
7.876
7.870
7.834
7.825
7.820
7.503
7.498
7.493
7.488
7.232
-0.000
1.00
1.04
1.14
2.31
1.22
2.18
f1 (ppm)

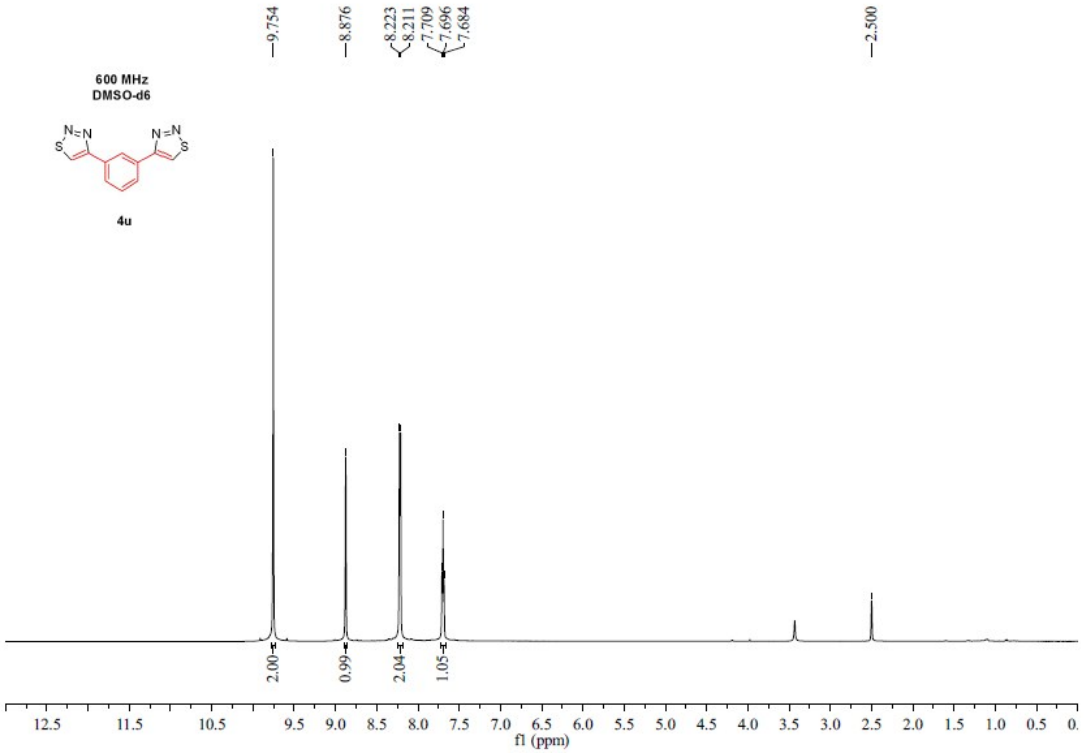

150 MHz
CDCl3
4r
162.68
133.47
133.28
130.14
130.11
128.84
128.38
127.88
127.68
126.79
126.66
124.59
77.21
77.00
76.79
f1 (ppm)

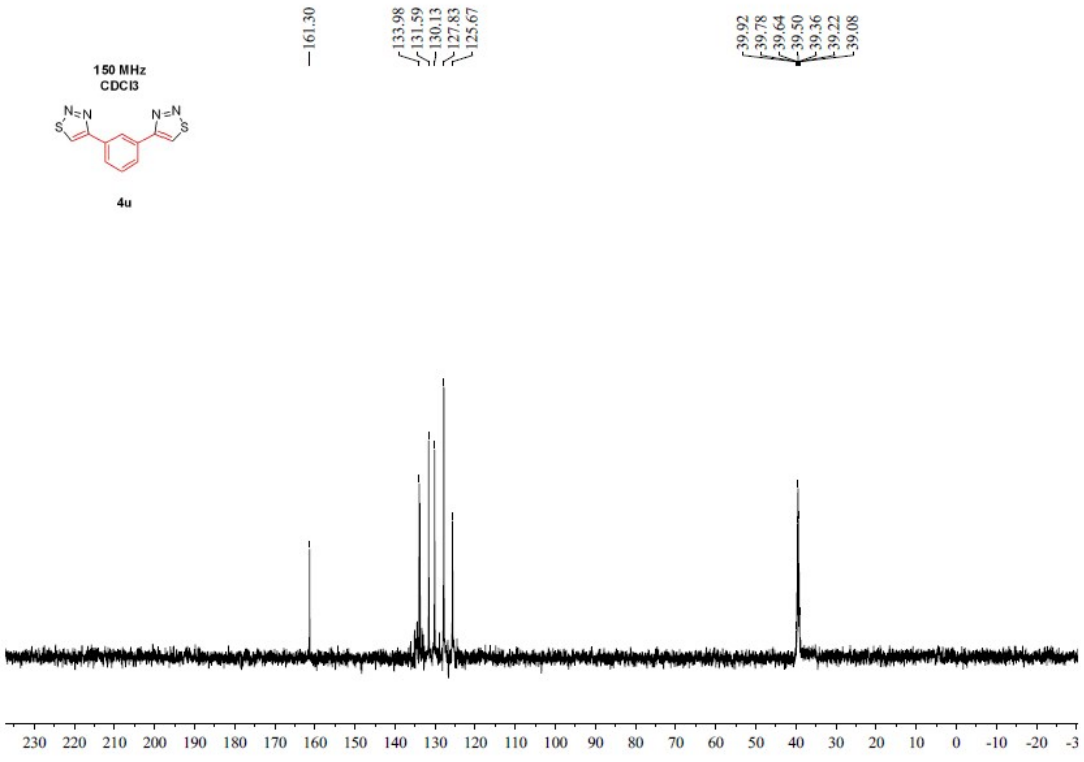

600 MHz
CDCl3
4s
8.647
8.630
8.088
8.075
8.059
7.977
7.963
7.945
7.929
7.913
7.742
7.732
7.713
7.578
7.564
7.561
7.547
7.533
7.517
7.254
0.000
1.00
1.13
2.20
1.08
3.22
f1 (ppm)

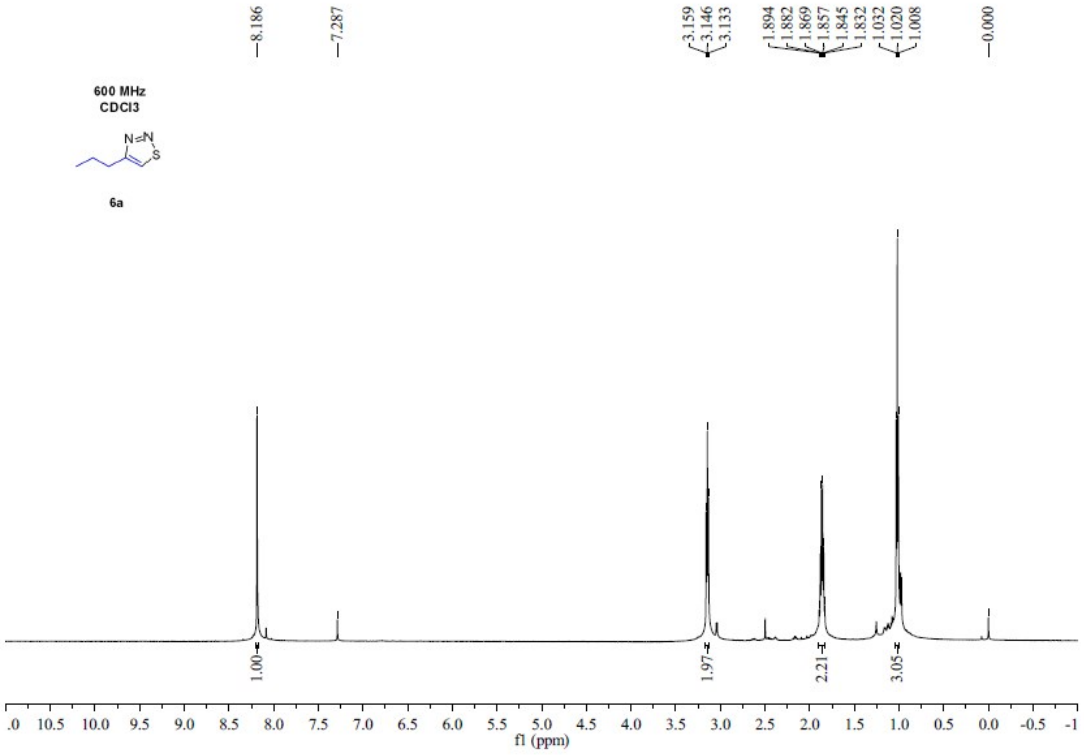

150 MHz
CDCl3
4s
161.82
134.25
134.18
133.75
131.29
129.91
128.47
128.28
127.00
126.24
125.19
125.03
77.21
77.00
76.79
f1 (ppm)

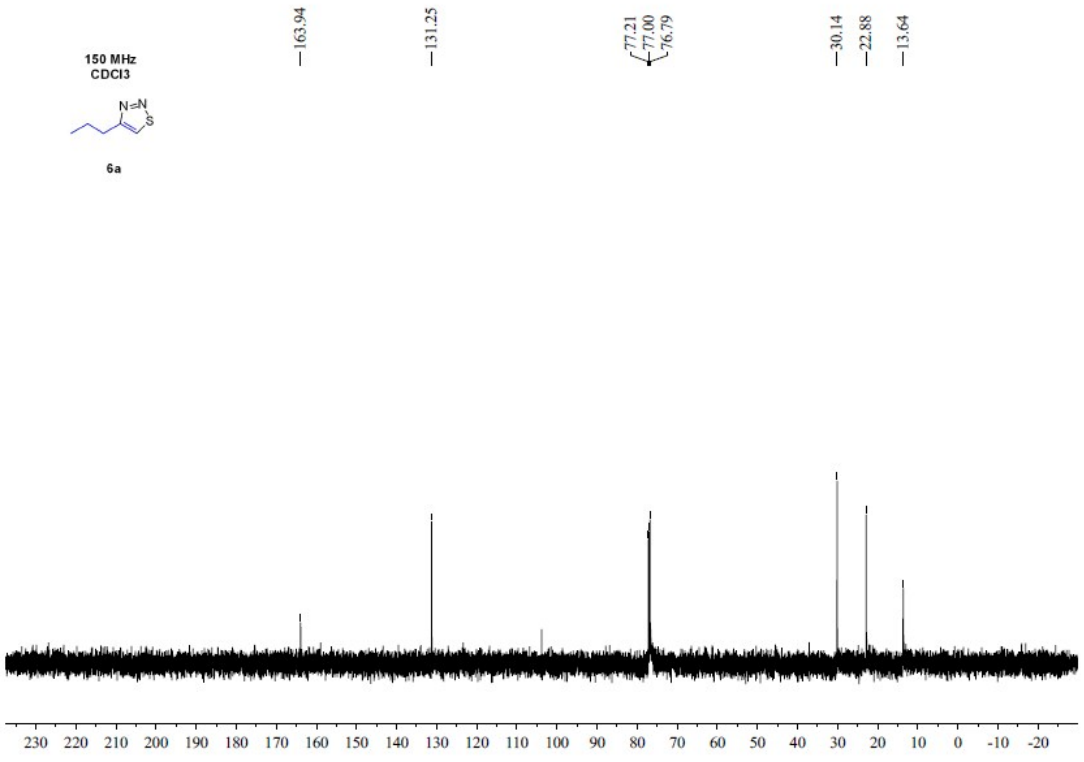

600 MHz
CDCl3
4t
8.407
7.269
7.098
2.638
2.458
0.000
1.00
0.99
3.11
3.15
f1 (ppm)

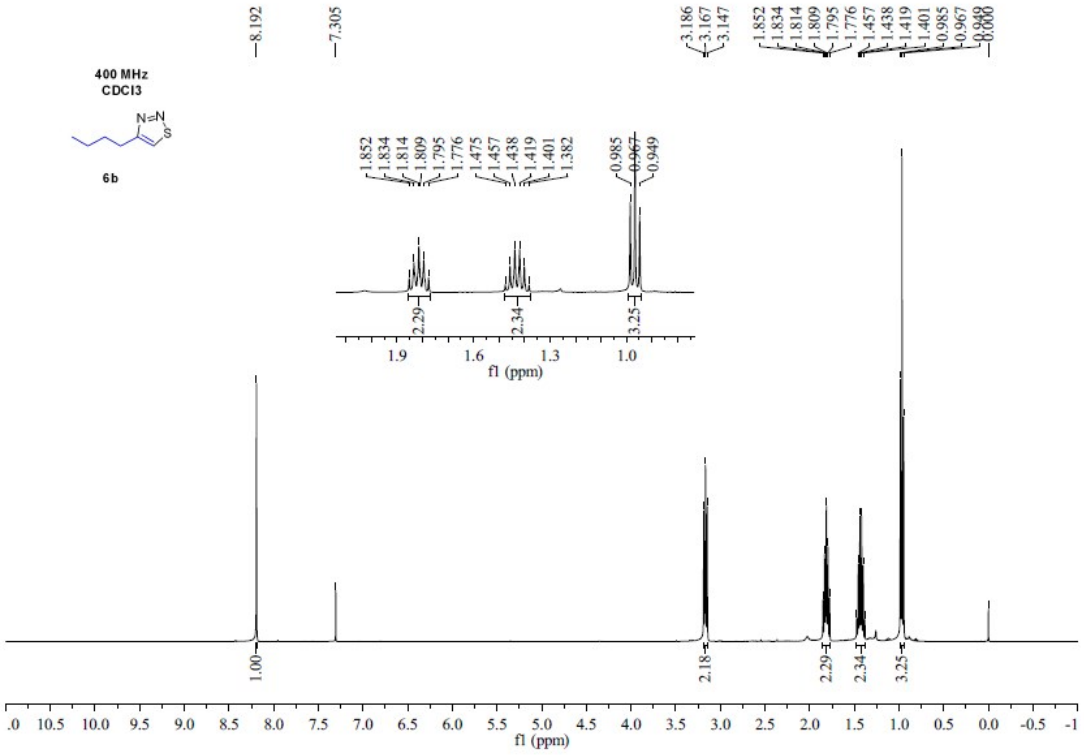

150 MHz
CDCl3
4t
158.59
136.39
135.92
130.06
127.13
126.32
77.21
77.00
76.79
14.96
14.93

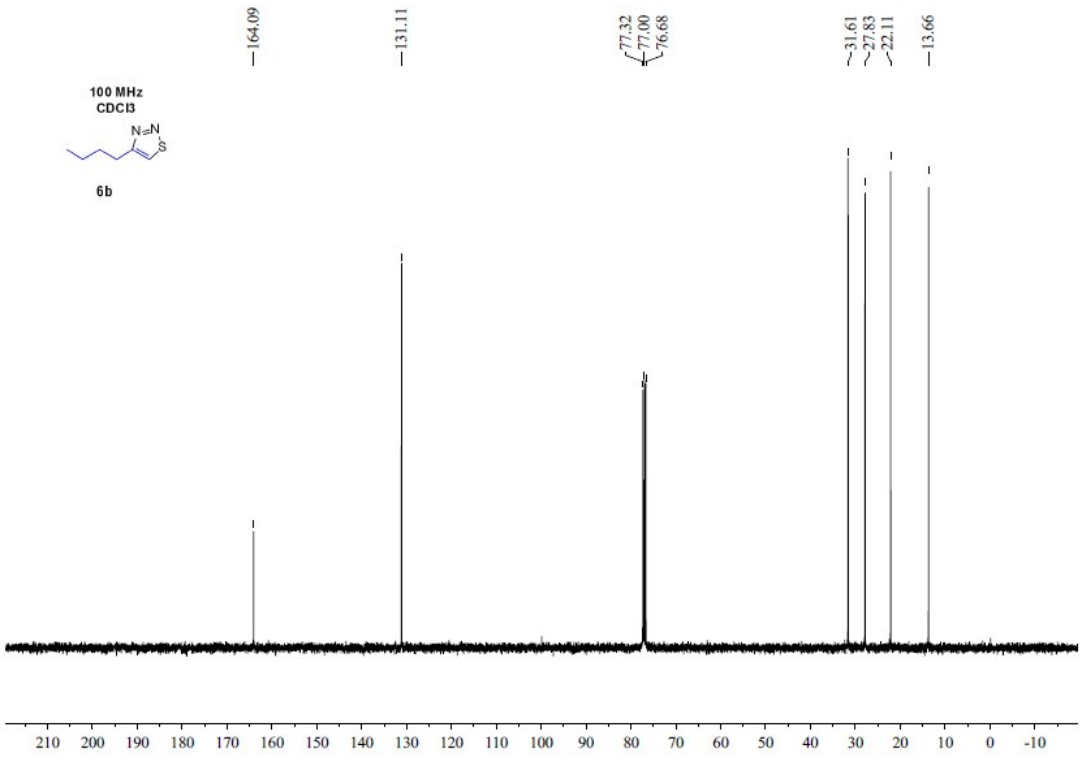

600 MHz
DMSO-d6
4u
9.754
8.876
8.223
8.211
7.709
7.696
7.684
2.500
2.00
0.99
2.04
1.05
f1 (ppm)

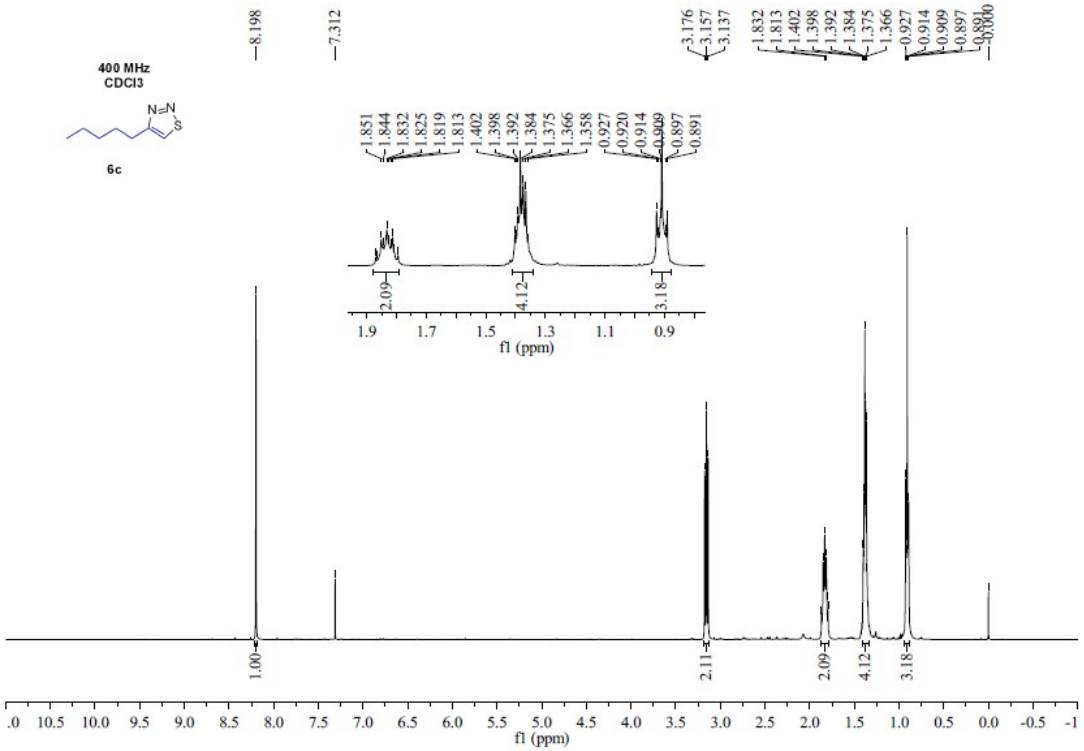

150 MHz
CDCl3
4u
161.30
133.98
131.59
130.13
127.83
125.67
39.92
39.78
39.64
39.50
39.36
39.22
39.08

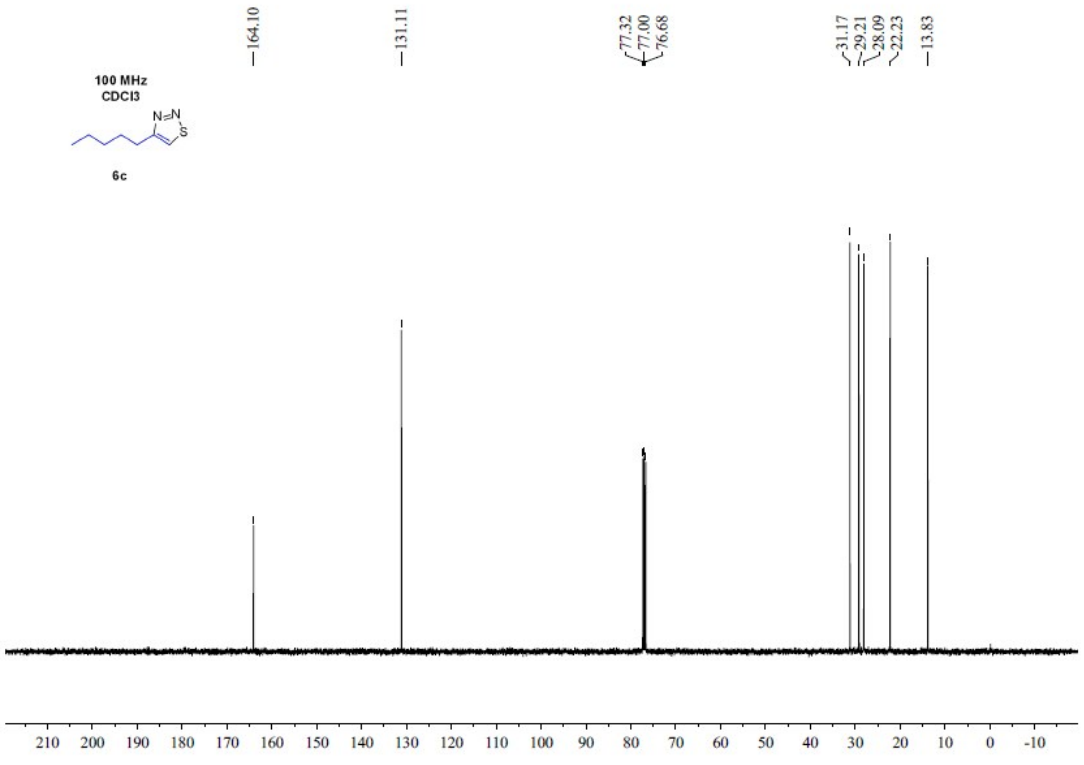

8.186
7.287
3.159
3.146
3.133
1.894
1.882
1.869
1.857
1.845
1.832
1.032
1.020
1.008
0.000
600 MHz
CDCl3
6a
1.00
1.97
2.21
3.05
f1 (ppm)

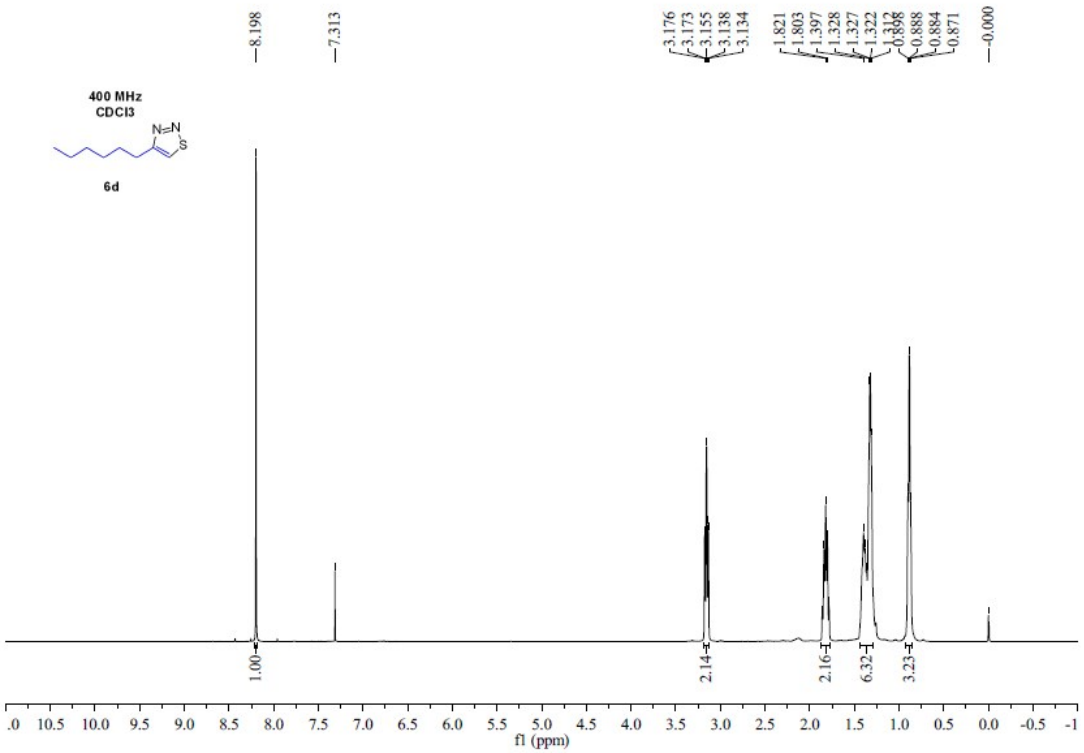

163.94
131.25
77.21
77.00
76.79
30.14
22.88
13.64
150 MHz
CDCl3
6a

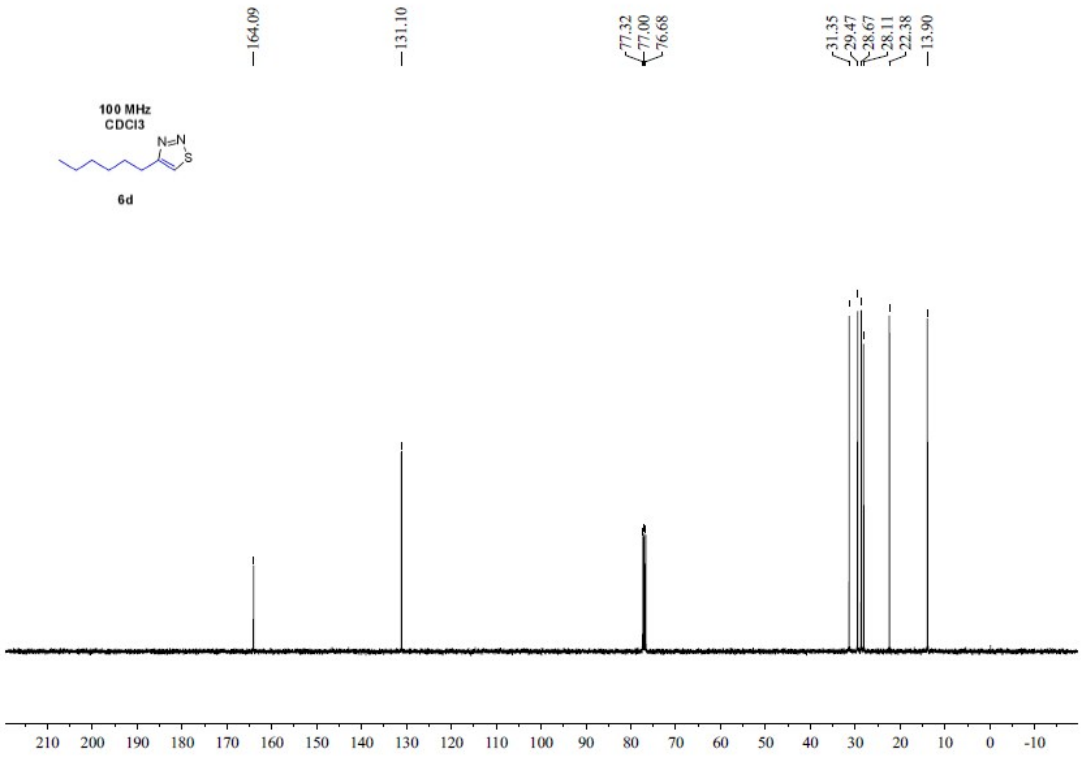

400 MHz
CDCl3
6b
8.192
7.305
3.186
3.167
3.147
1.852
1.834
1.814
1.809
1.795
1.776
1.475
1.457
1.438
1.419
1.401
1.382
0.985
0.967
0.949
2.29
2.34
3.25
1.9
1.6
1.3
1.0
f1 (ppm)
1.00
2.18
2.29
2.34
3.25
f1 (ppm)

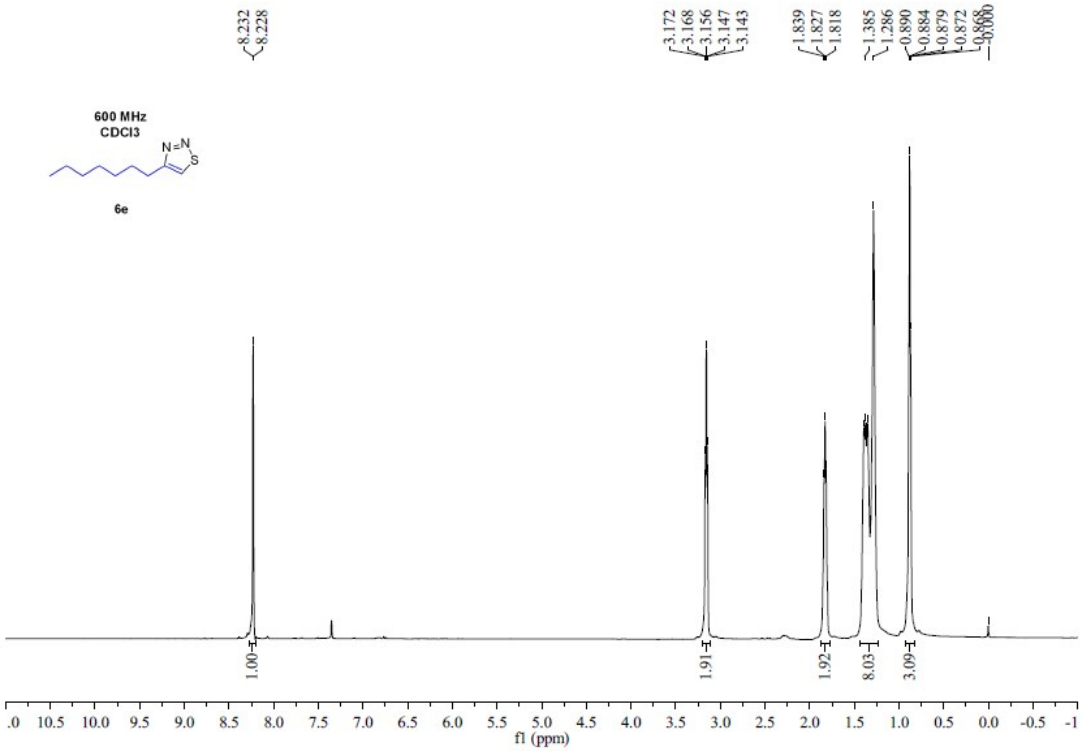

100 MHz
CDCl3
6b
164.09
131.11
77.32
77.00
76.68
31.61
27.83
22.11
13.66

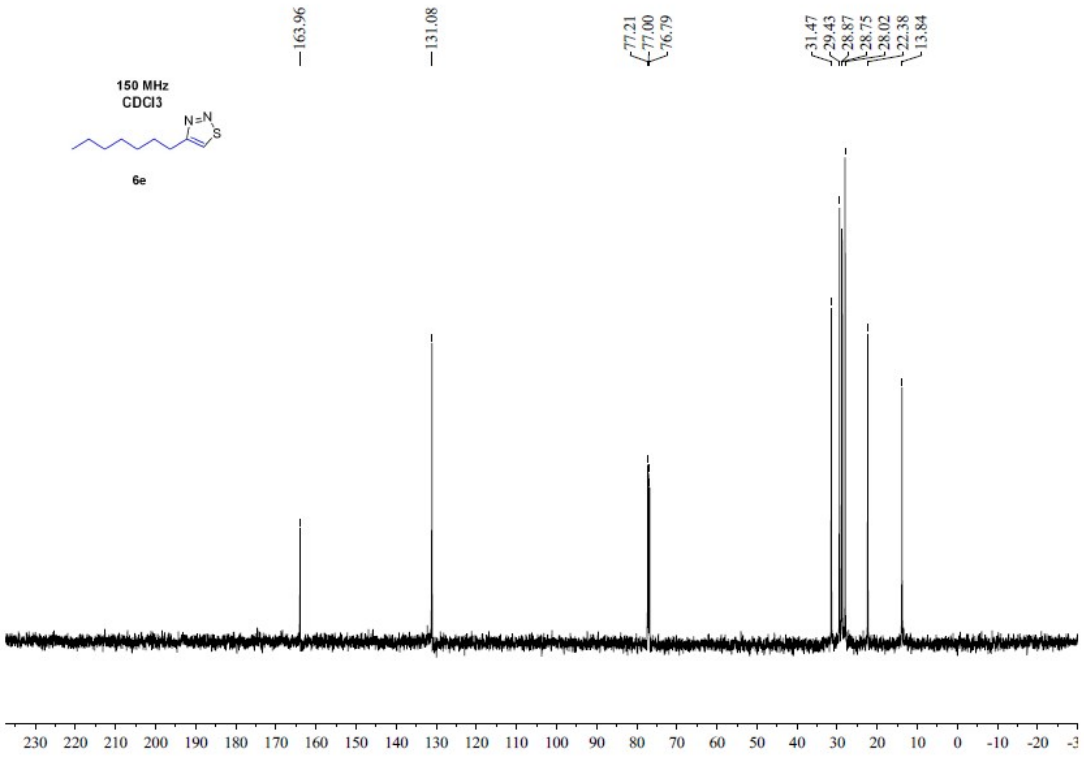

400 MHz
CDCl3
6c
8.198
7.312
3.176
3.157
3.137
1.832
1.813
1.402
1.398
1.392
1.384
1.375
1.366
0.927
0.914
0.909
0.897
0.891
0.000
1.851
1.844
1.832
1.825
1.819
1.813
1.402
1.398
1.392
1.384
1.375
1.366
1.358
0.927
0.920
0.914
0.909
0.897
0.891
2.09
4.12
3.18
1.9 1.7 1.5 1.3 1.1 0.9
f1 (ppm)
1.00
2.11
2.09
4.12
3.18
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)

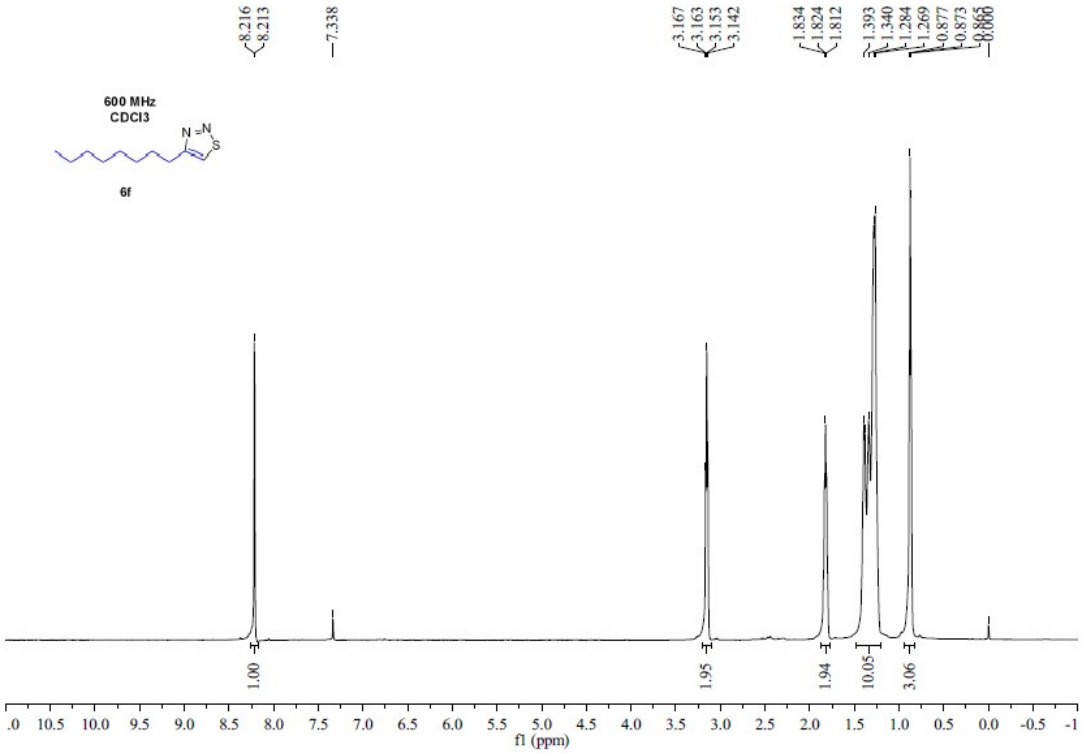

100 MHz
CDCl3
6c
164.10
131.11
77.32
77.00
76.68
31.17
29.21
28.09
22.23
13.83
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

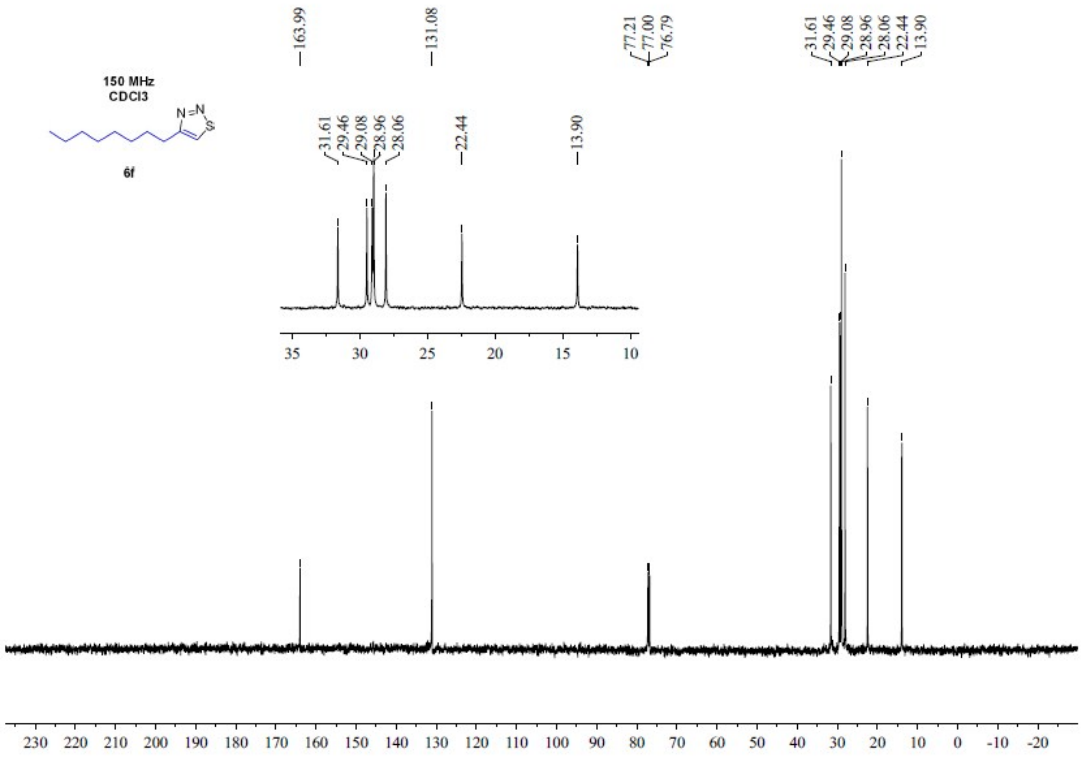

8.198
7.313
3.176
3.173
3.155
3.138
3.134
1.821
1.803
1.397
1.328
1.327
1.322
1.312
0.898
0.888
0.884
0.871
0.000
400 MHz
CDCl3
6d
1.00
2.14
2.16
6.32
3.23
f1 (ppm)

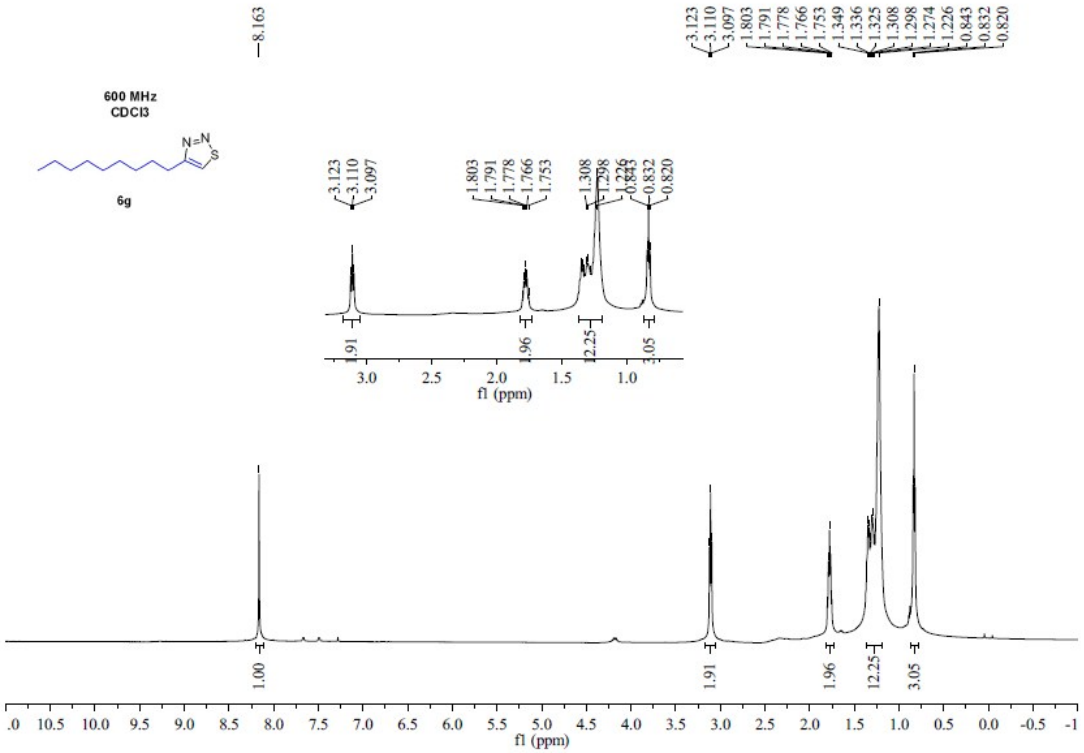

164.09
131.10
77.32
77.00
76.68
31.35
29.47
28.67
28.11
22.38
13.90
100 MHz
CDCl3
6d

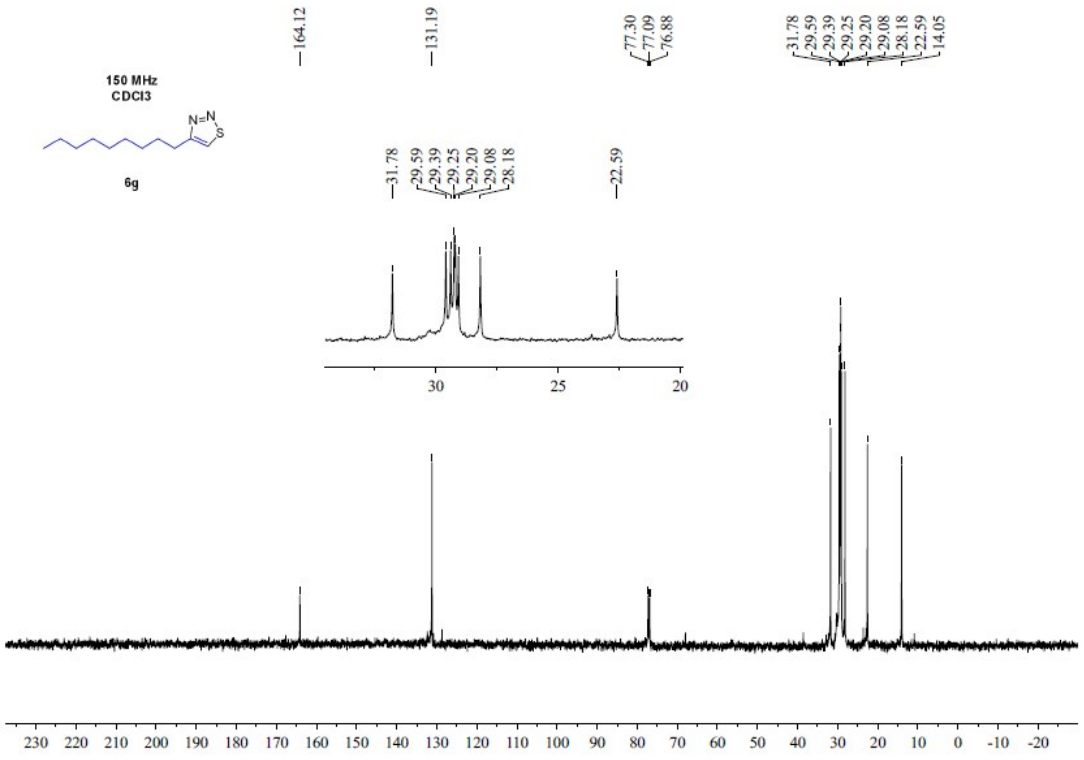

600 MHz
CDCl3
6e
8.232
8.228
3.172
3.168
3.156
3.147
3.143
1.839
1.827
1.818
1.385
1.286
0.890
0.884
0.879
0.872
0.868
0.000
1.00
1.91
1.92
8.03
3.09
f1 (ppm)

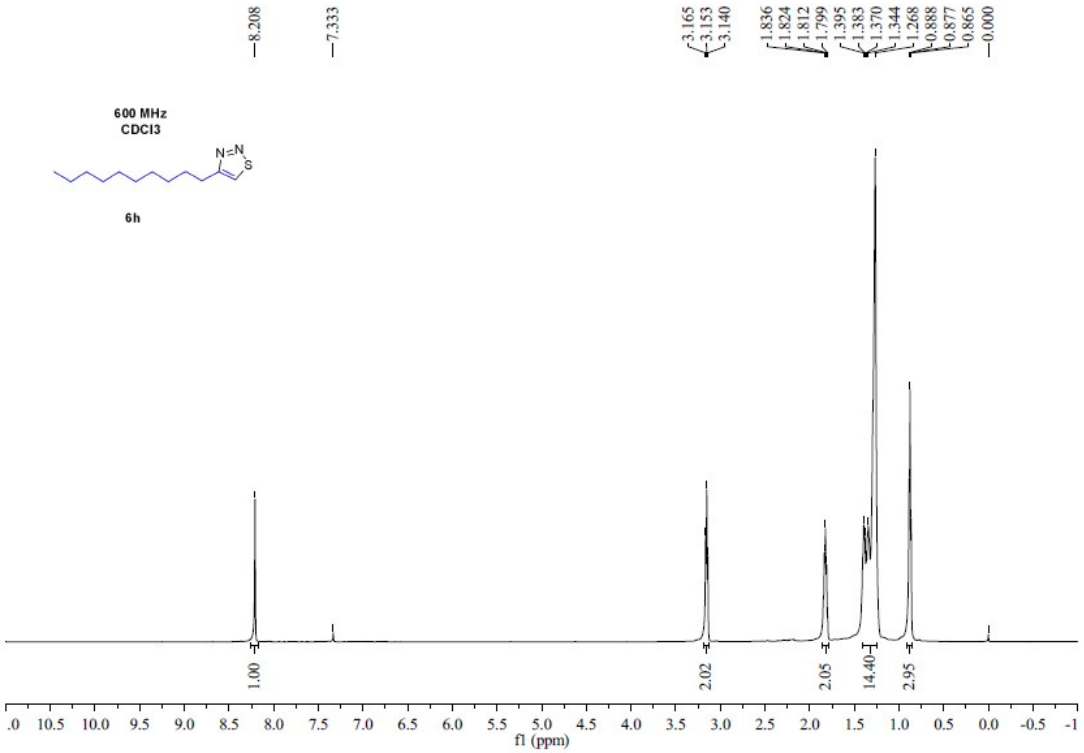

150 MHz
CDCl3
6e
163.96
131.08
77.21
77.00
76.79
31.47
29.43
28.87
28.75
28.02
22.38
13.84

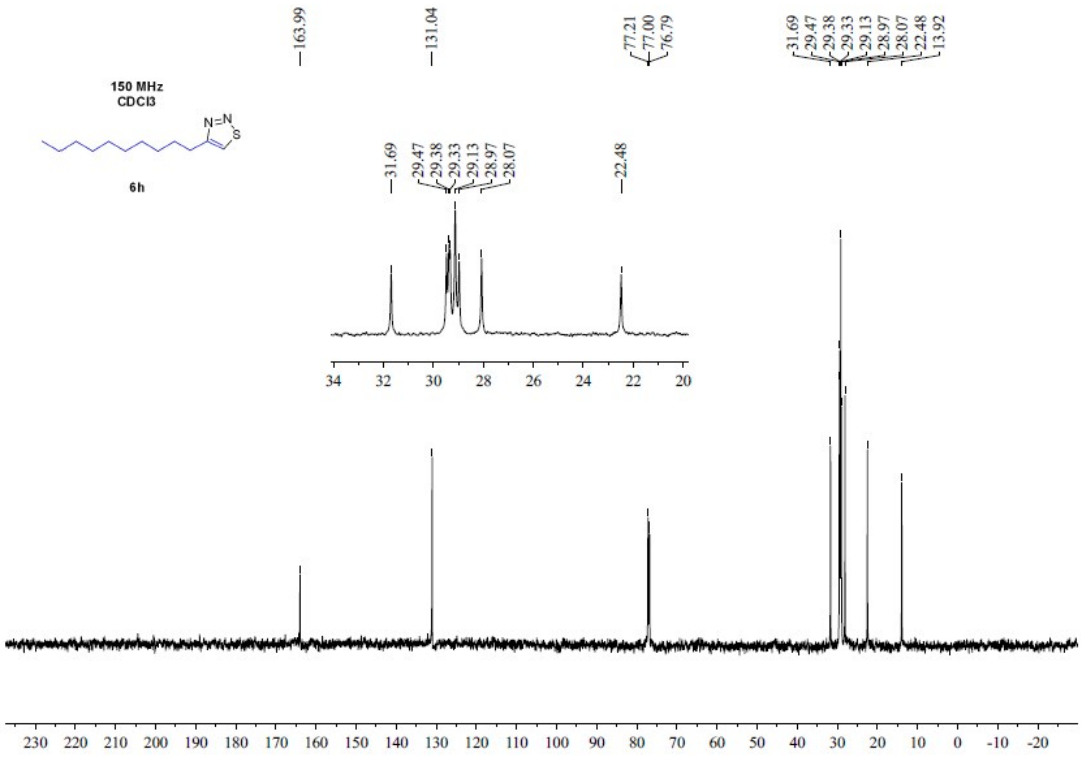

600 MHz
CDCl3
6f
8.216
8.213
7.338
3.167
3.163
3.153
3.142
1.834
1.824
1.812
1.393
1.340
1.284
1.269
0.877
0.873
0.865
0.860
1.00
1.95
1.94
10.05
3.06
f1 (ppm)

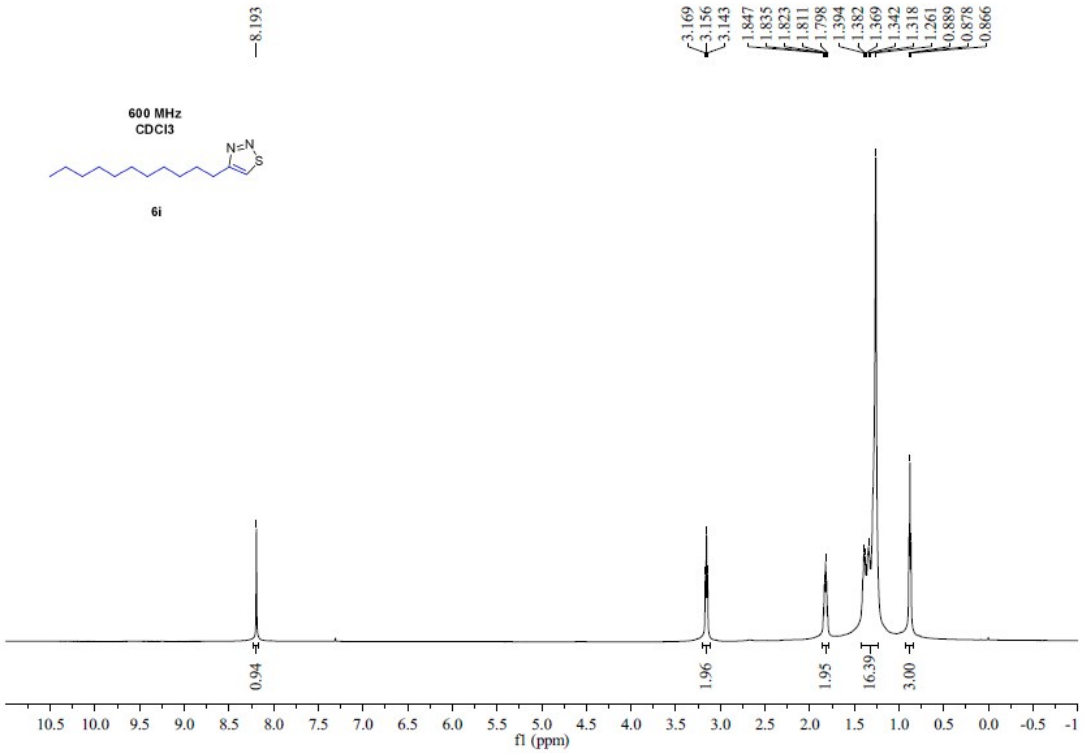

150 MHz
CDCl3
6f
163.99
131.08
77.21
77.00
76.79
31.61
29.46
29.08
28.96
28.06
22.44
13.90

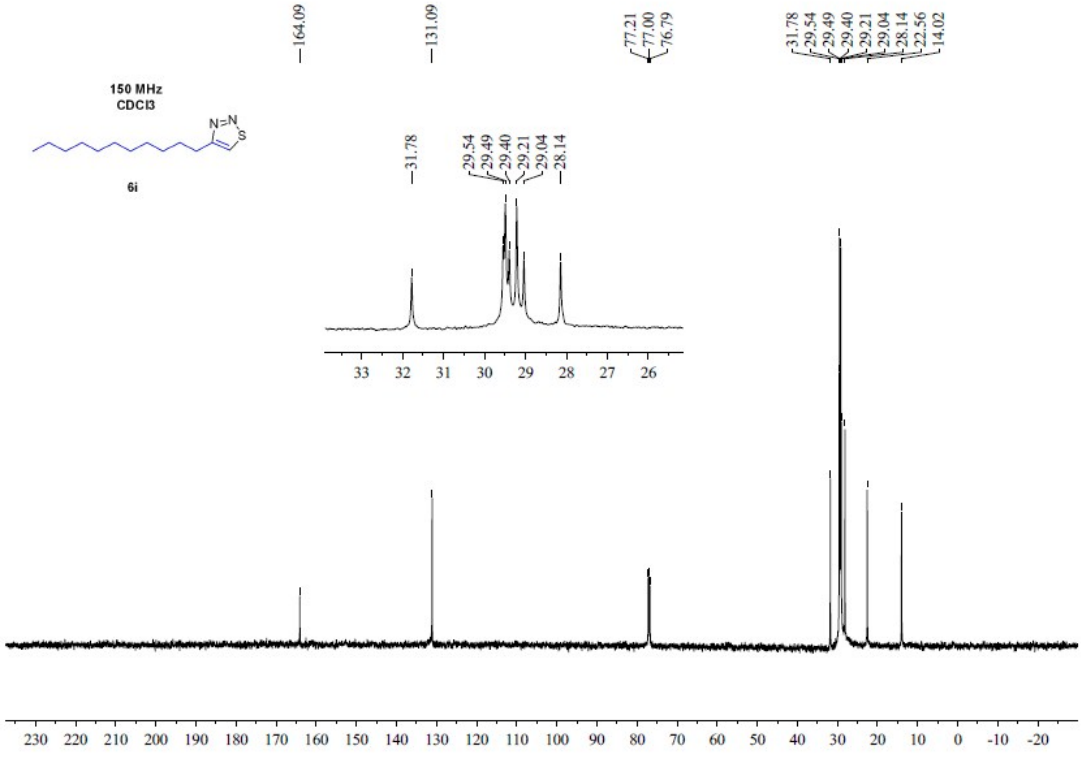

600 MHz
CDCl3
6g
8.163
3.123
3.110
3.097
1.803
1.791
1.778
1.766
1.753
1.349
1.336
1.325
1.308
1.298
1.274
1.226
0.843
0.832
0.820
1.00
1.91
1.96
12.25
3.05
f1 (ppm)

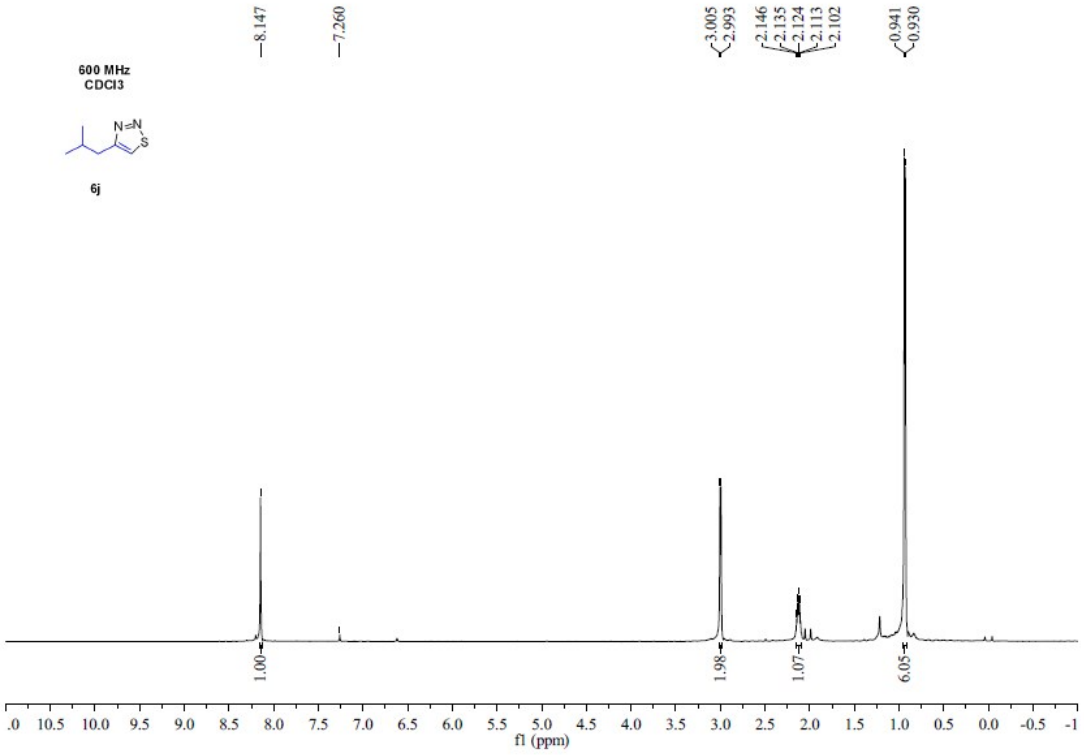

150 MHz
CDCl3
6g
164.12
131.19
77.30
77.09
76.88
31.78
29.59
29.39
29.25
29.20
29.08
28.18
22.59
14.05
f1 (ppm)

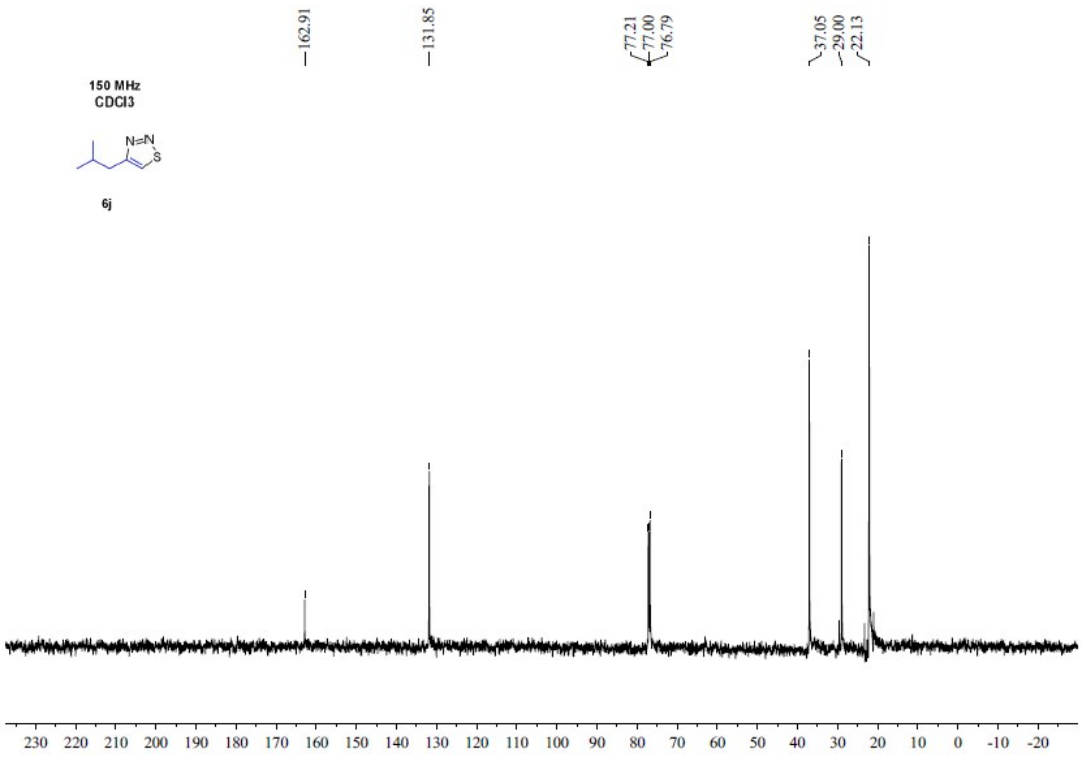

600 MHz
CDCl3
6h
8.208
7.333
3.165
3.153
3.140
1.836
1.824
1.812
1.799
1.395
1.383
1.370
1.344
1.268
0.888
0.877
0.865
0.000
1.00
2.02
2.05
14.40
2.95
f1 (ppm)

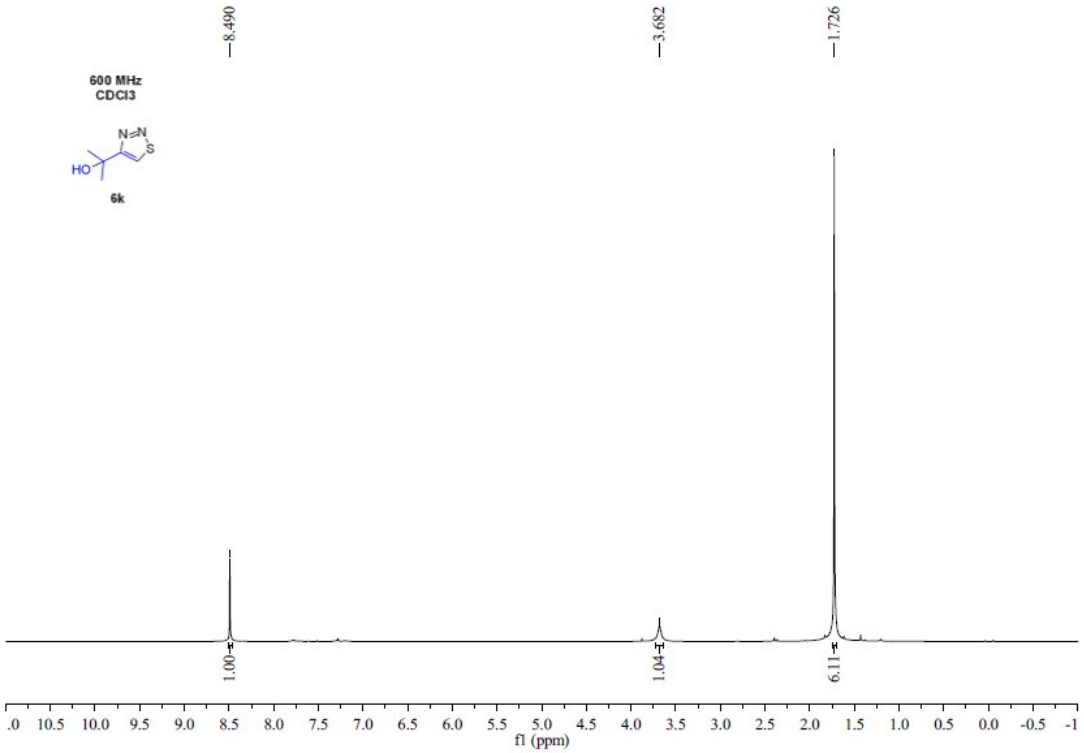

150 MHz
CDCl3
6h
163.99
131.04
77.21
77.00
76.79
31.69
29.47
29.38
29.33
29.13
28.97
28.07
22.48
13.92

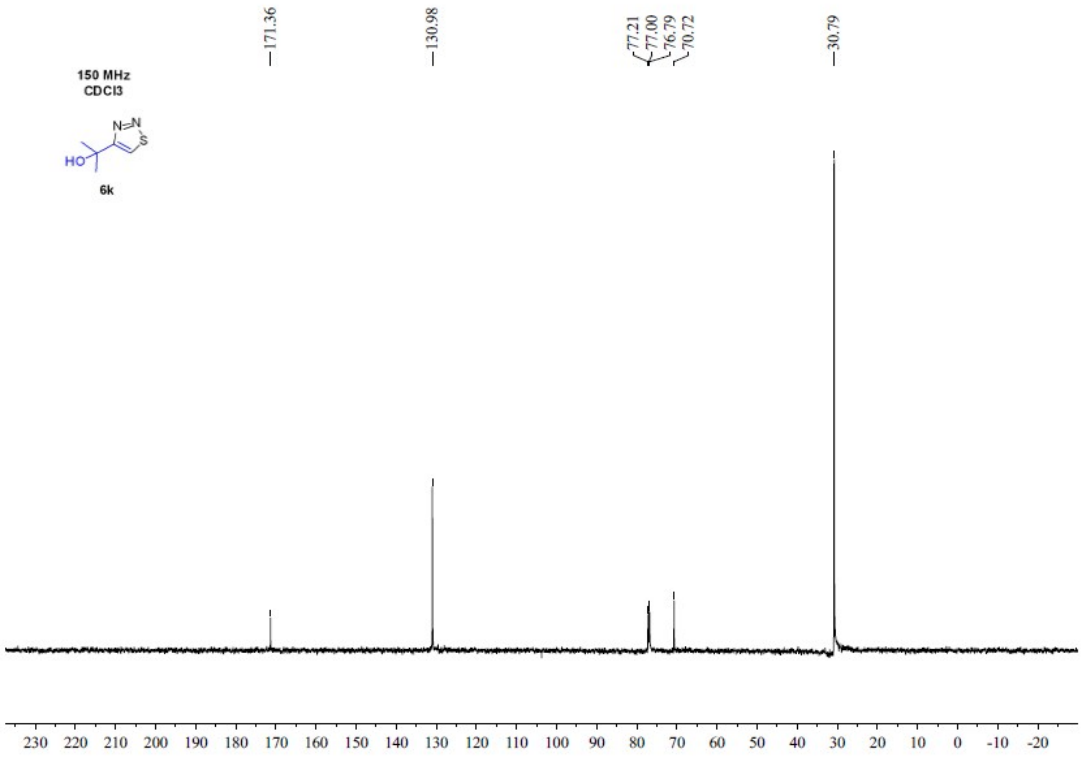

8.193
3.169
3.156
3.143
1.847
1.835
1.823
1.811
1.798
1.394
1.382
1.369
1.342
1.318
1.261
0.889
0.878
0.866
600 MHz
CDCl3
6i
0.94
1.96
1.95
16.39
3.00
f1 (ppm)

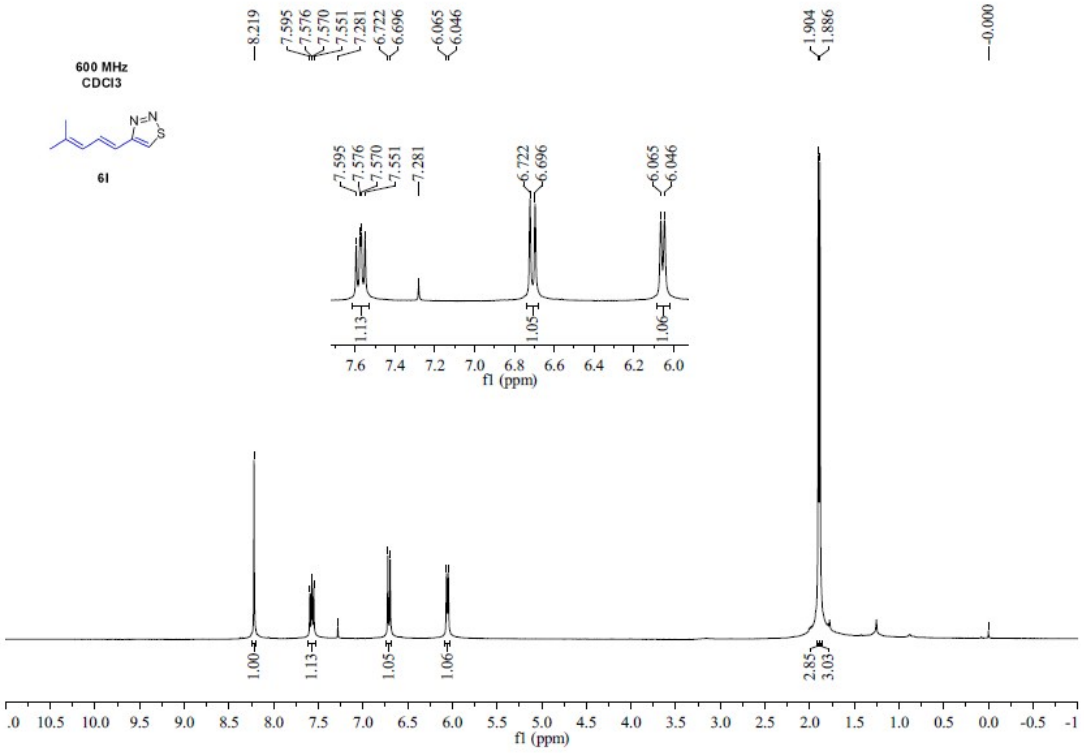

164.09
131.09
77.21
77.00
76.79
31.78
29.54
29.49
29.40
29.21
29.04
28.14
22.56
14.02
150 MHz
CDCl3
6i

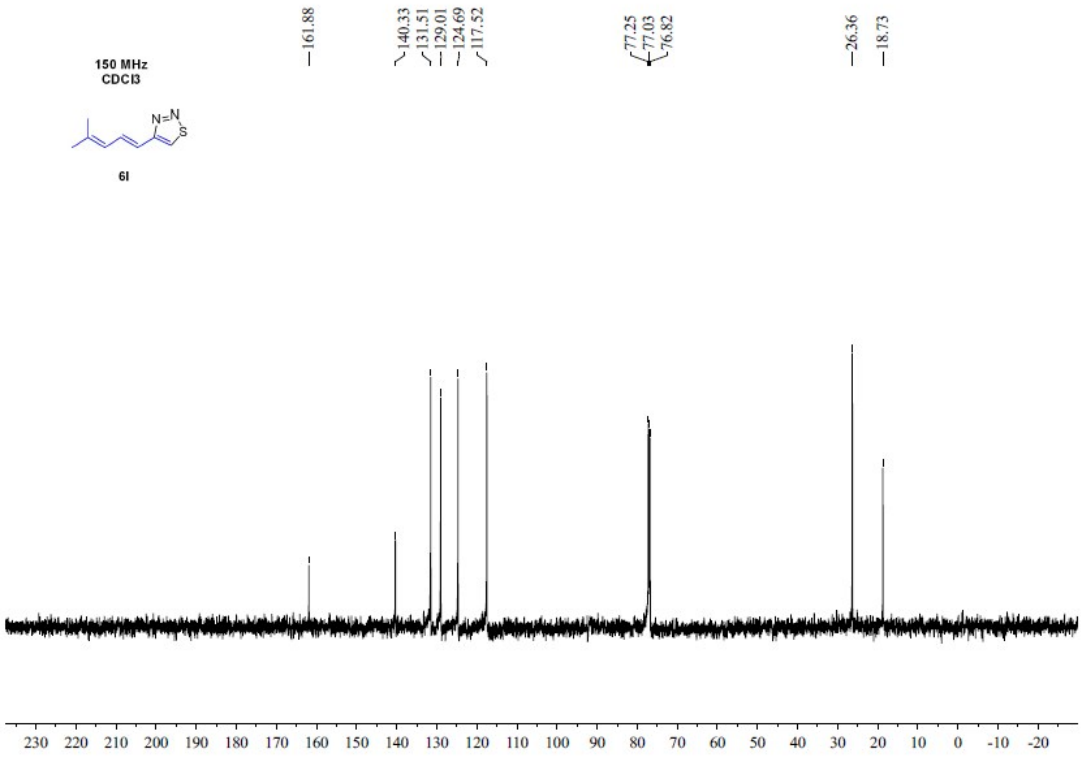

8.147
7.260
3.005
2.993
2.146
2.135
2.124
2.113
2.102
0.941
0.930
600 MHz
CDCl3
6j
1.00
1.98
1.07
6.05
f1 (ppm)

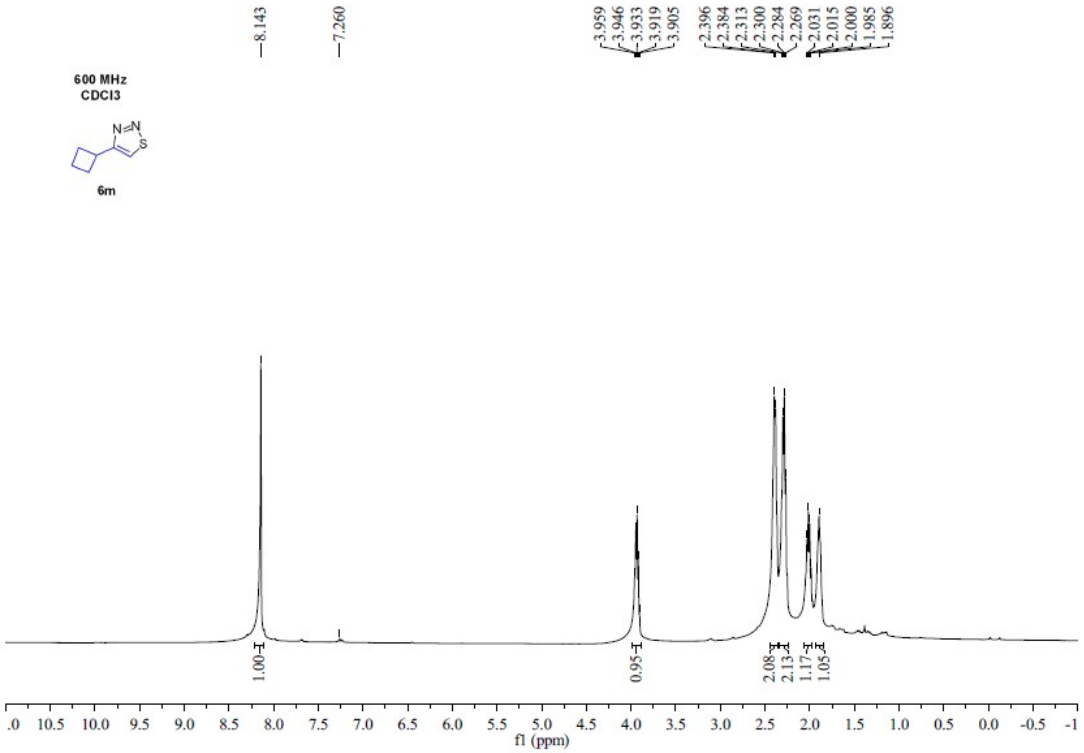

162.91
131.85
77.21
77.00
76.79
37.05
29.00
22.13
150 MHz
CDCl3
6j

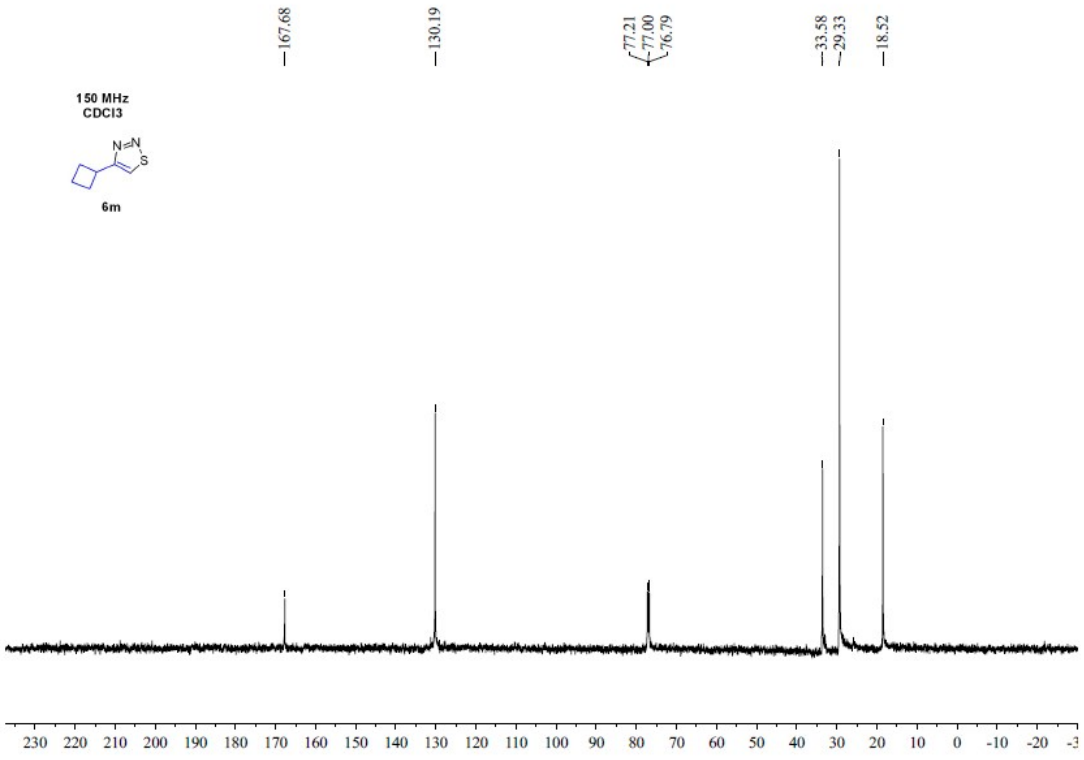

8.490
3.682
1.726
600 MHz
CDCl3
6k
1.00
1.04
6.11
f1 (ppm)

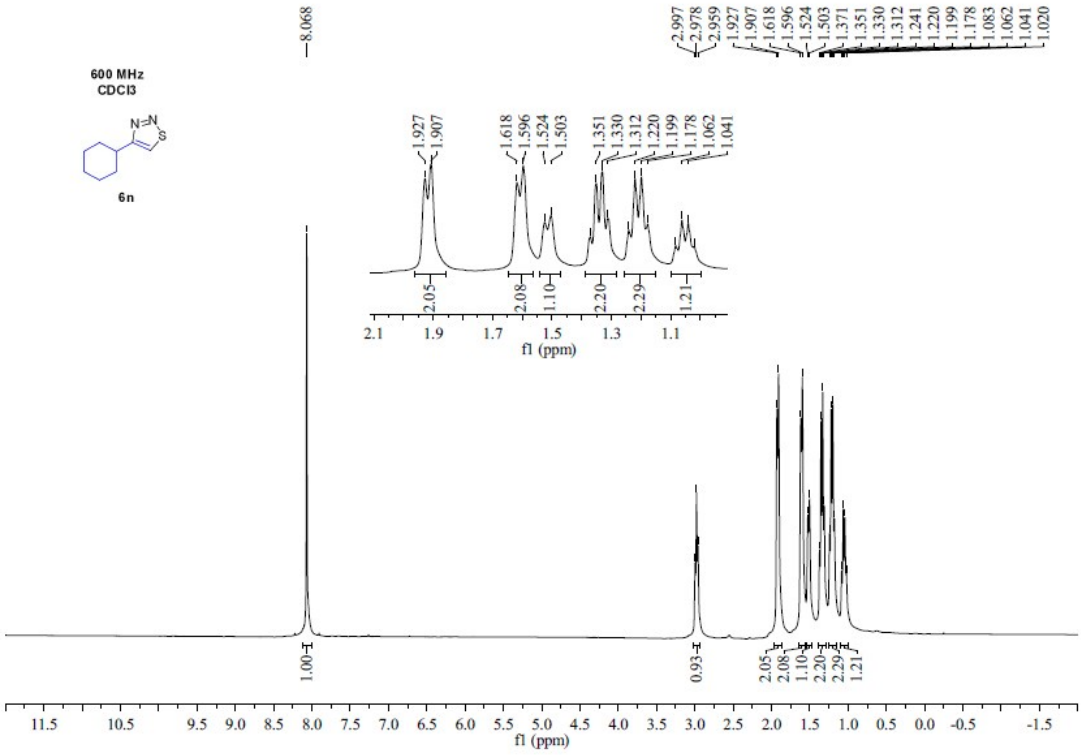

171.36
130.98
77.21
77.00
76.79
70.72
30.79
150 MHz
CDCl3
6k

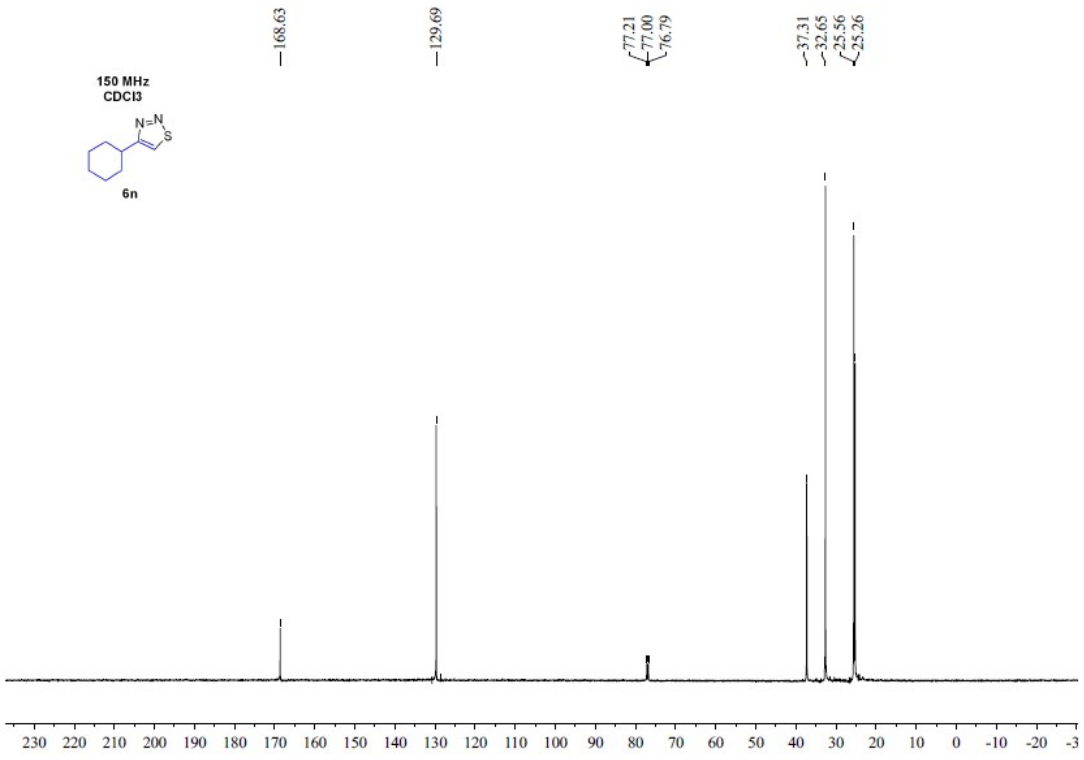

600 MHz
CDCl3
6l
8.219
7.595
7.576
7.570
7.551
7.281
6.722
6.696
6.065
6.046
1.904
1.886
0.000
1.00
1.13
1.05
1.06
2.85
3.03
f1 (ppm)

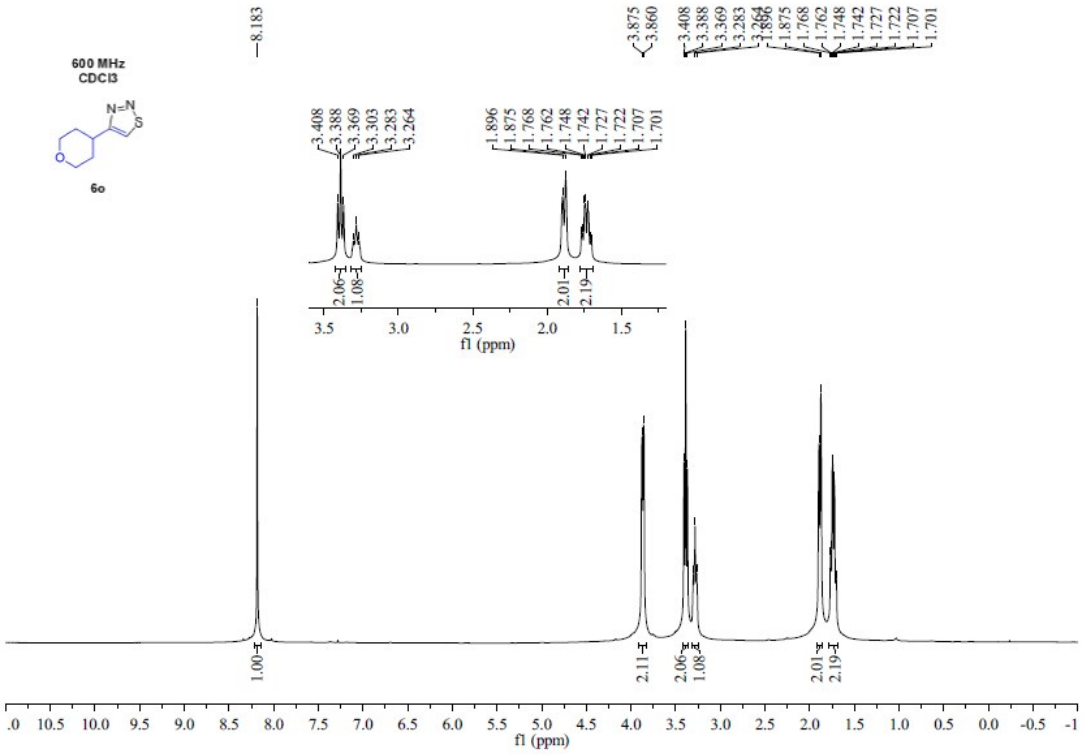

150 MHz
CDCl3
6l
161.88
140.33
131.51
129.01
124.69
117.52
77.25
77.03
76.82
26.36
18.73

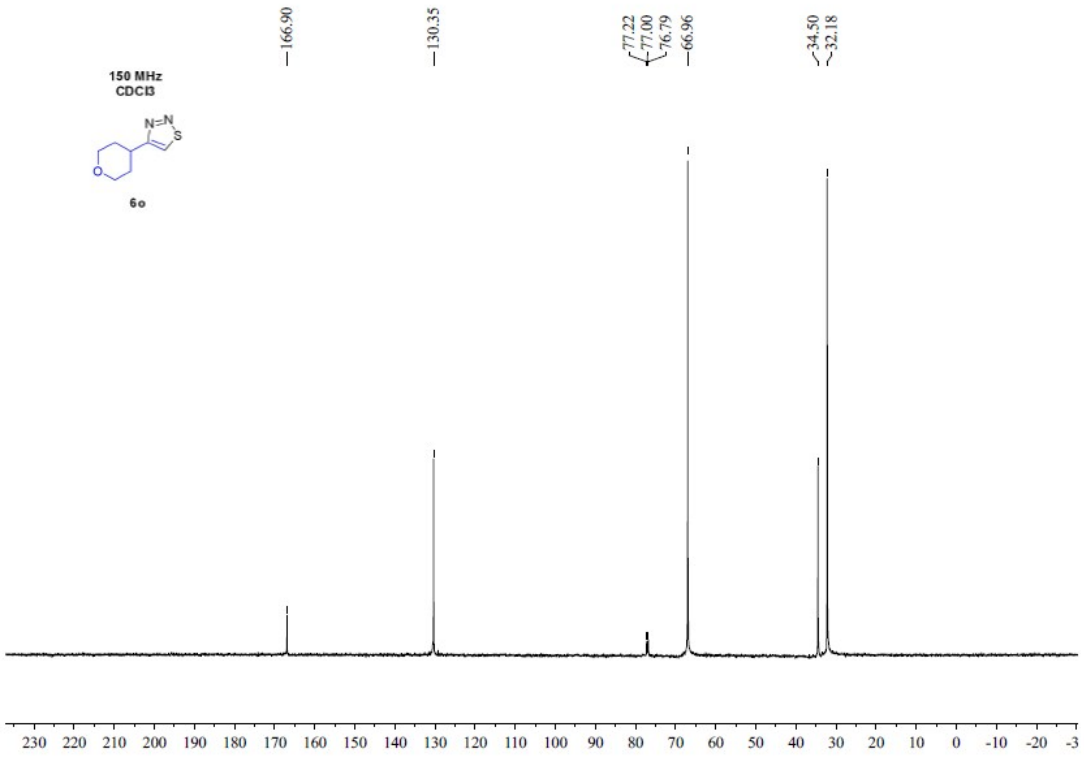

8.143
7.260
3.959
3.946
3.933
3.919
3.905
2.396
2.384
2.313
2.300
2.284
2.269
2.031
2.015
2.000
1.985
1.896
600 MHz
CDCl3
6m
1.00
0.95
2.08
2.13
1.17
1.05
f1 (ppm)

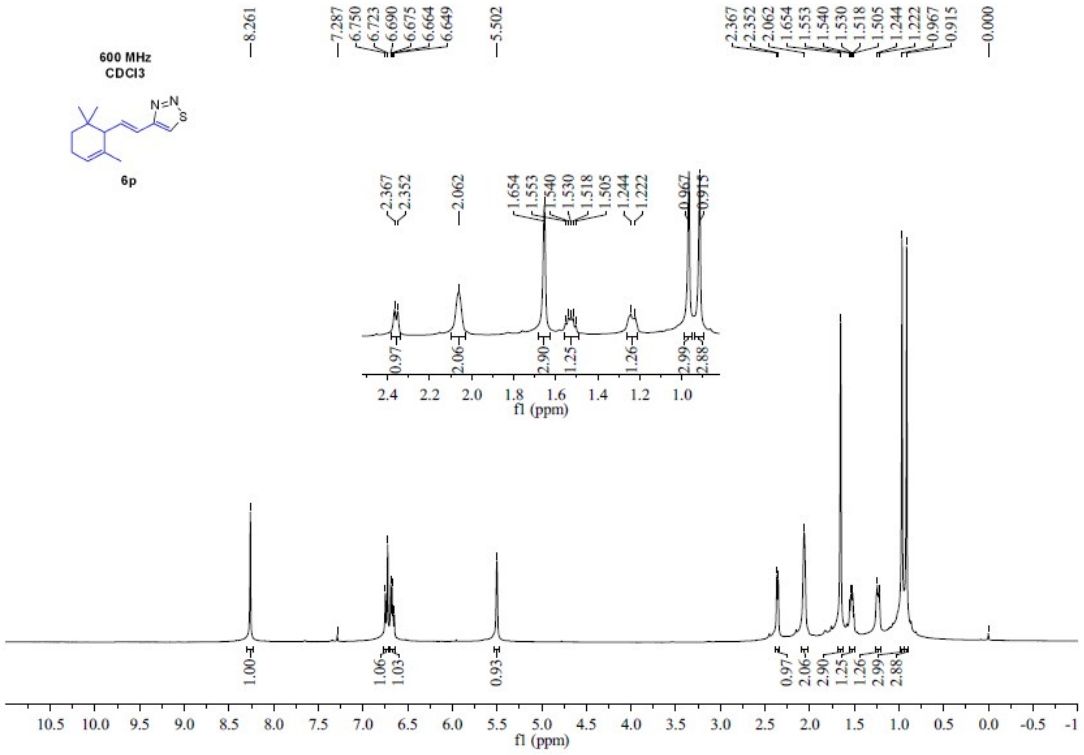

167.68
130.19
77.21
77.00
76.79
33.58
29.33
18.52
150 MHz
CDCl3
6m

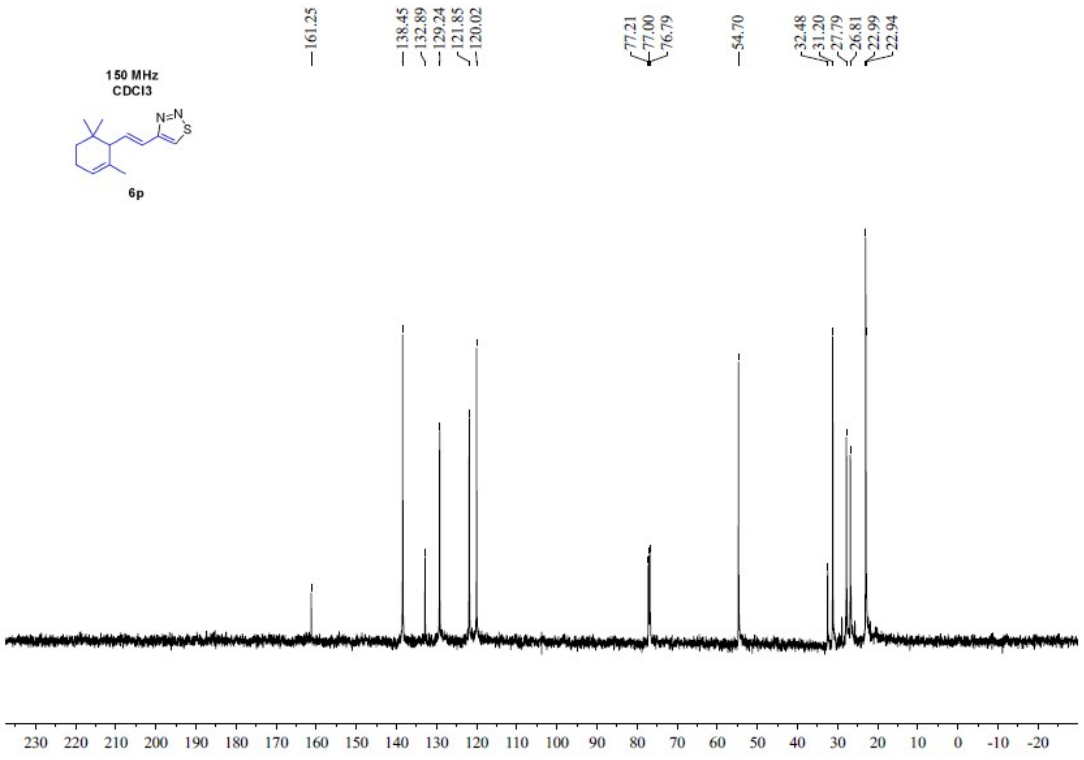

600 MHz
CDCl3
6n
8.068
2.997
2.978
2.959
1.927
1.907
1.618
1.596
1.524
1.503
1.371
1.351
1.330
1.312
1.241
1.220
1.199
1.178
1.083
1.062
1.041
1.020
2.05
2.08
1.10
2.20
2.29
1.21
f1 (ppm)
1.00
0.93
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.5
f1 (ppm)

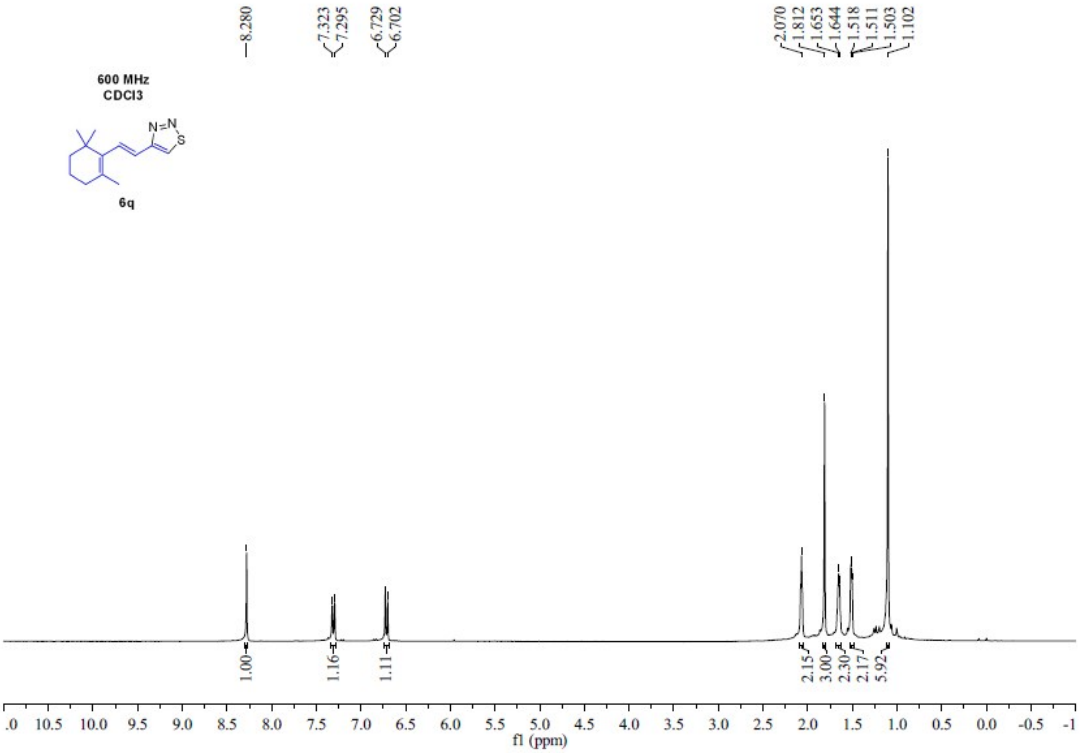

150 MHz
CDCl3
6n
168.63
129.69
77.21
77.00
76.79
37.31
32.65
25.56
25.26
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -3

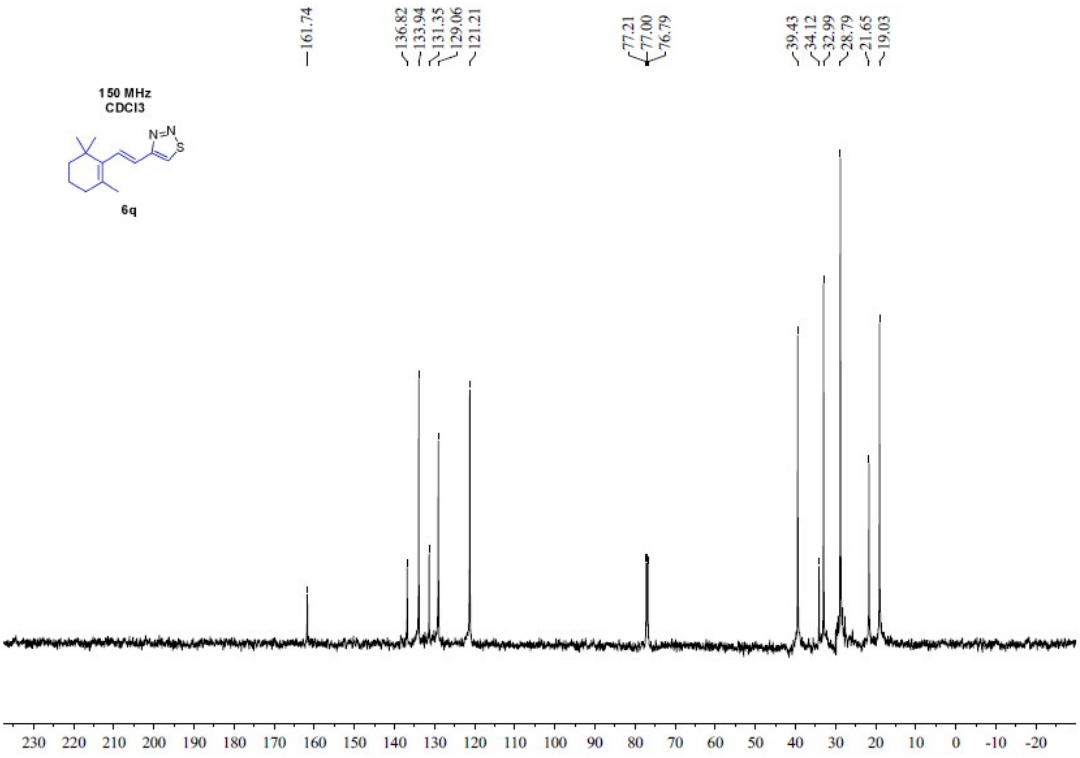

600 MHz
CDCl3
6o
8.183
3.875
3.860
3.408
3.388
3.369
3.303
3.283
3.264
1.896
1.875
1.768
1.762
1.748
1.742
1.727
1.722
1.707
1.701
2.06
1.08
2.01
2.19
3.5
3.0
2.5
2.0
1.5
f1 (ppm)
1.00
2.11
2.06
1.08
2.01
2.19
f1 (ppm)

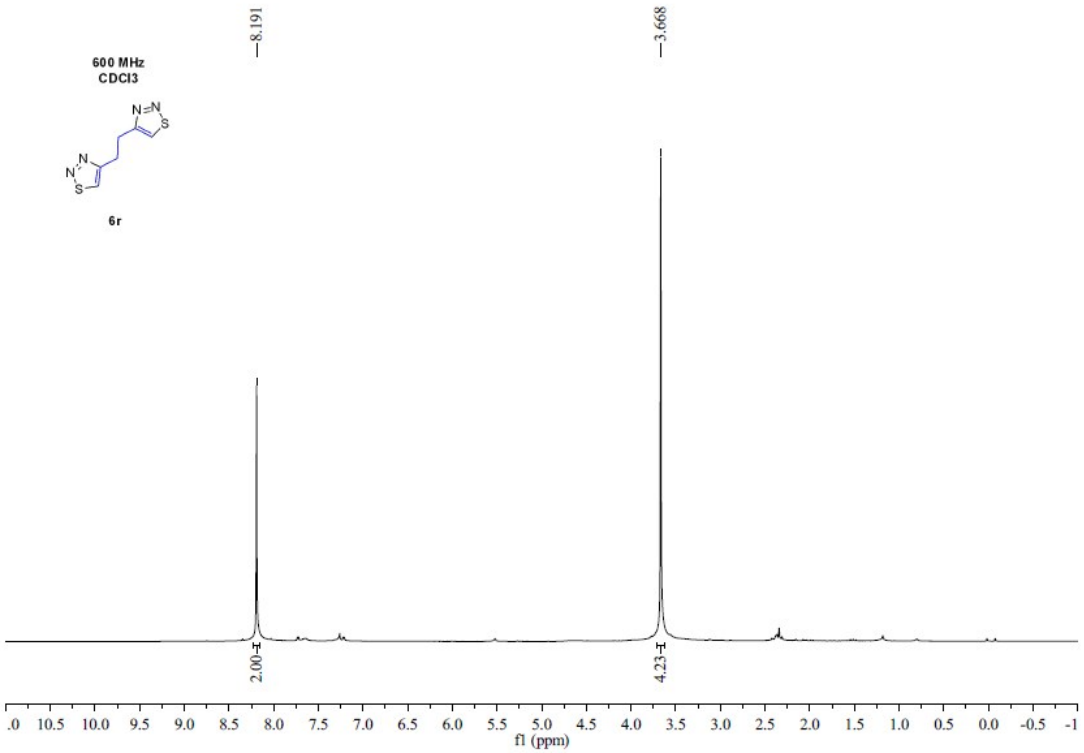

150 MHz
CDCl3
6o
166.90
130.35
77.22
77.00
76.79
66.96
34.50
32.18


600 MHz
CDCl3
6p
8.261
7.287
6.750
6.723
6.690
6.675
6.664
6.649
5.502
2.367
2.352
2.062
1.654
1.553
1.540
1.530
1.518
1.505
1.244
1.222
0.967
0.915
0.000
1.00
1.06
1.03
0.93
0.97
2.06
2.90
1.25
1.26
2.99
2.88
f1 (ppm)

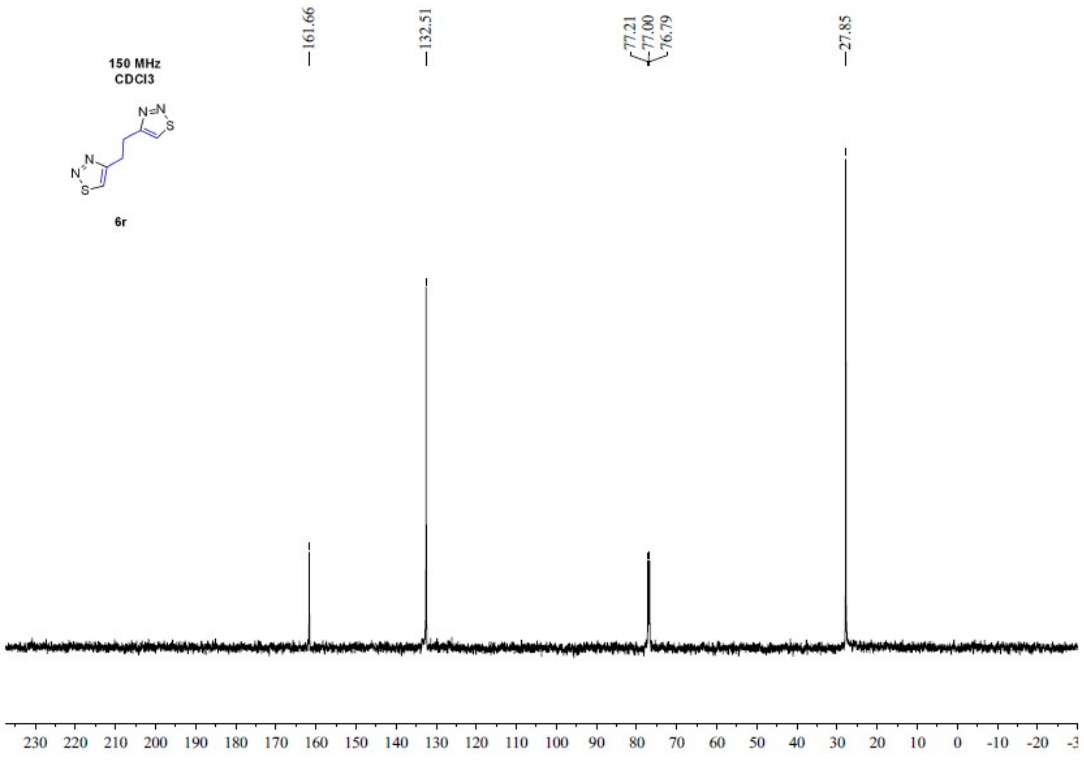

150 MHz
CDCl3
6p
161.25
138.45
132.89
129.24
121.85
120.02
77.21
77.00
76.79
54.70
32.48
31.20
27.79
26.81
22.99
22.94


600 MHz
CDCl3
6q
8.280
7.323
7.295
6.729
6.702
2.070
1.812
1.653
1.644
1.518
1.511
1.503
1.102
1.00
1.16
1.11
2.15
3.00
2.30
2.17
5.92
f1 (ppm)

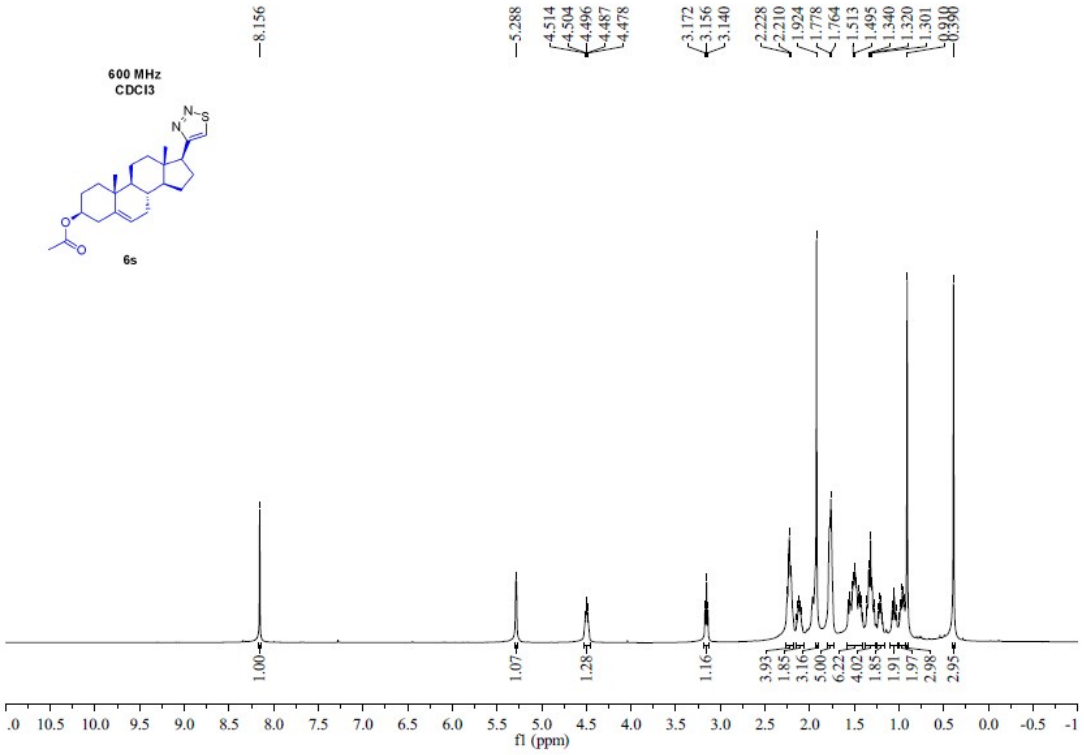

150 MHz
CDCl3
6q
161.74
136.82
133.94
131.35
129.06
121.21
77.21
77.00
76.79
39.43
34.12
32.99
28.79
21.65
19.03

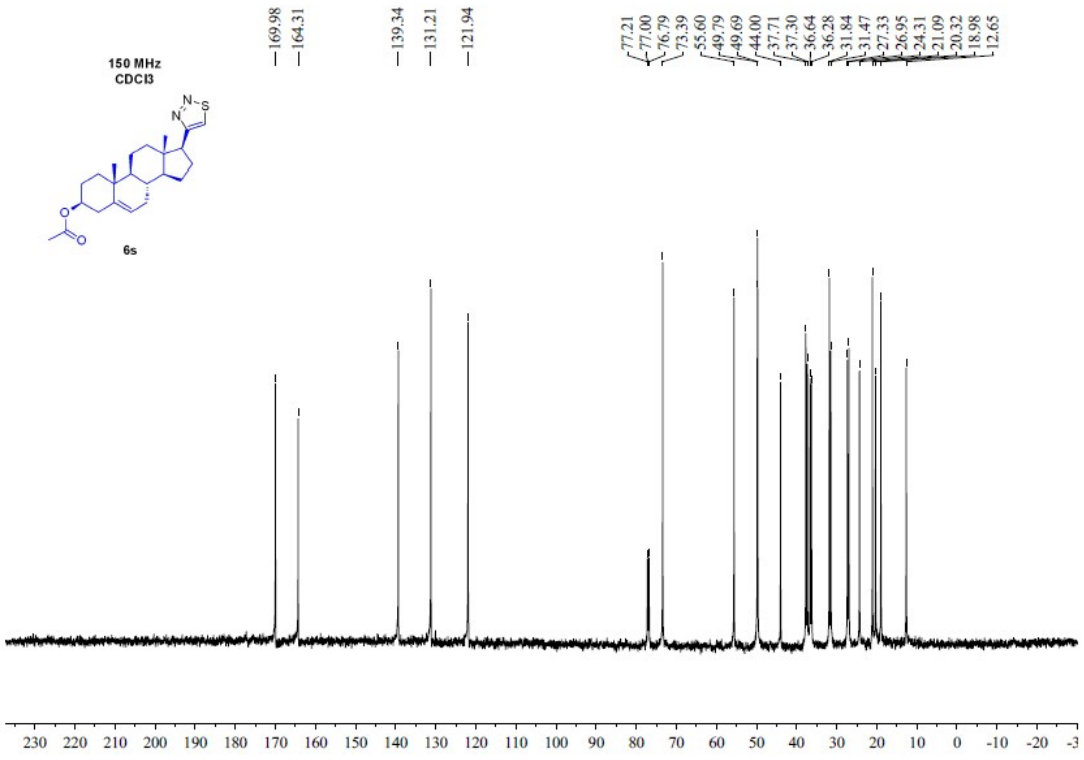

600 MHz
CDCl3
8.191
3.668
2.00
4.23
f1 (ppm)

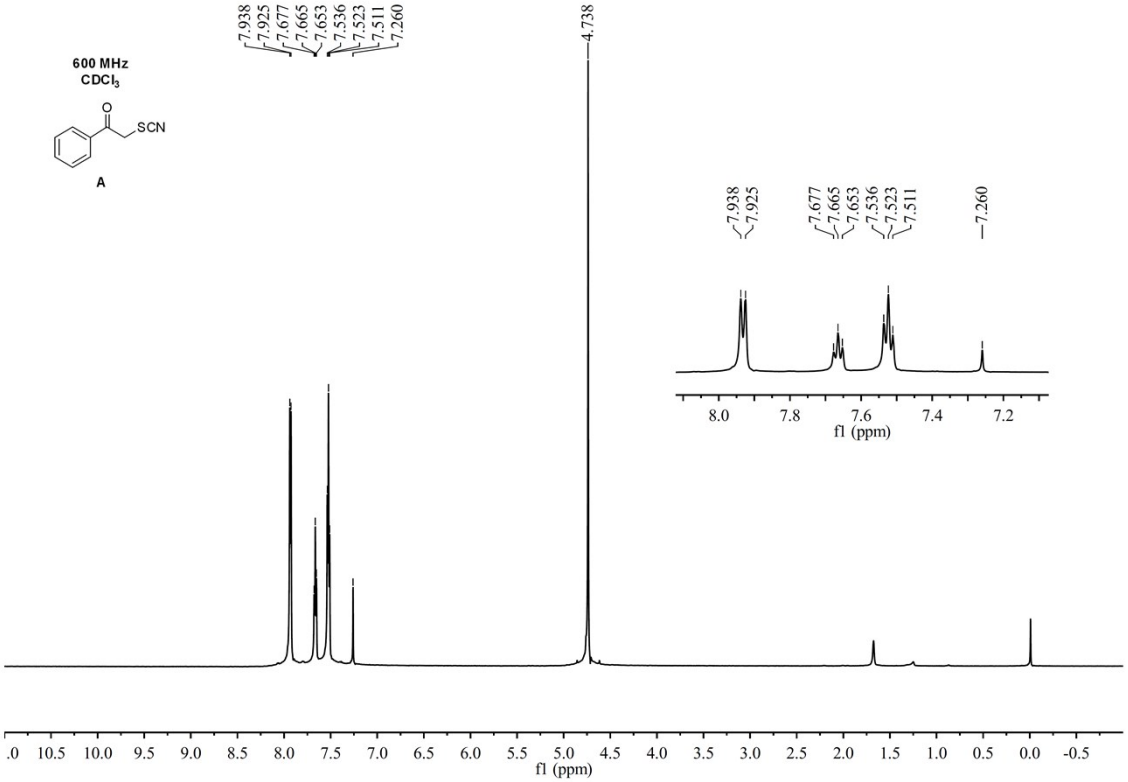
6r

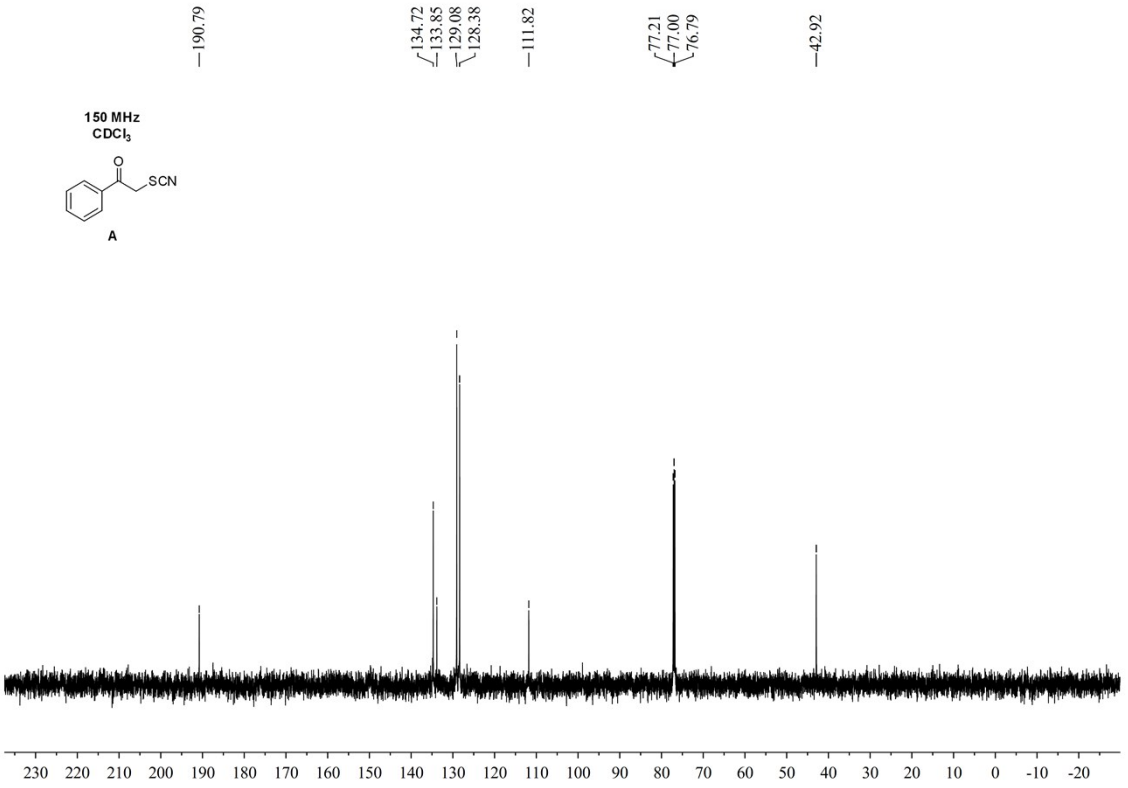

150 MHz
CDCl3
161.66
132.51
77.21
77.00
76.79
27.85

6r


600 MHz
CDCl3
6s
8.156
5.288
4.514
4.504
4.496
4.487
4.478
3.172
3.156
3.140
2.228
2.210
1.924
1.778
1.764
1.513
1.495
1.340
1.320
1.301
0.910
0.900
1.00
1.07
1.28
1.16
3.93
1.85
3.16
5.00
6.22
4.02
1.85
1.91
1.97
2.98
2.95
f1 (ppm)


150 MHz
CDCl3
6s
169.98
164.31
139.34
131.21
121.94
77.21
77.00
76.79
73.39
55.60
49.79
49.69
44.00
37.71
37.30
36.64
36.28
31.84
31.47
27.33
26.95
24.31
21.09
20.32
18.98
12.65


600 MHz
CDCl3
SCN
A
7.938
7.925
7.677
7.665
7.653
7.536
7.523
7.511
7.260
4.738
fl (ppm)


150 MHz
CDCl3
SCN
A
190.79
134.72
133.85
129.08
128.38
111.82
77.21
77.00
76.79
42.92

7. The product of 4m was detected by GC-MS