# Copper Nitrate-Mediated Regio- and Stereoselective Difunctionalization of Alkynes: A Direct Approach to α-Chloro-β-nitroolefins

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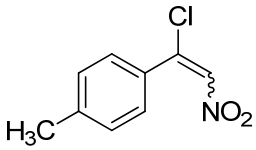
# Supporting Information

## CONTENTS:

## 1. General Information

All reagents and metal catalysts were obtained from commercial sources without further purification, and commercially available solvents were purified before use. All new compounds were fully characterized. All melting points were taken on a WRS-1A or a WRS-1B Digital Melting Point Apparatus without correction. Infrared spectra were obtained using an AVATAR 370 FT-IR spectrometer. $^{1}H$, $^{13}C$, and $^{19}F$ NMR spectra were recorded with a Bruker AV-500 spectrometer operating at 500 MHz, 125 MHz and 470 MHz, respectively, with chemical shift values being reported in ppm relative to chloroform ($\delta$ = 7.26 ppm), dimethyl sulfoxide ($\delta$ = 2.50 ppm) or TMS ($\delta$ = 0.00 ppm) for $^{1}H$ NMR; chloroform ($\delta$ = 77.16 ppm) or dimethyl sulfoxide ($\delta$ = 39.52 ppm) for $^{13}C$ NMR; and $C_6F_6$ ($\delta$ = -164.9 ppm) for $^{19}F$ NMR. Mass spectra (MS) and high resolution mass spectra (HRMS) were recorded with an Agilent 5975C using an Electron impact (EI) or Electrospray ionization (ESI) techniques. Silica gel plate GF254 was used for thin layer chromatography (TLC) and silica gel 300-400 mesh was used for flash column chromatography. Yields refer to chromatographically and spectroscopically pure compounds, unless otherwise indicated. $^{1}H$, $^{13}C$ and $^{19}F$ NMR data for minor isomer of products are recorded from the spectra of mixture with *E*/*Z* isomers. Unless commercially available alkynes, other alkynes were all prepared according to the literature reported procedures.[1-3]

## 2. Synthesis and Characterization of Products

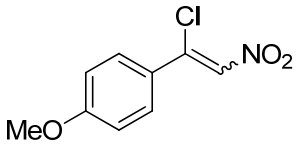



**1-(1-Chloro-2-nitrovinyl)-4-methylbenzene (2a):** To a test tube were added **1a** (34.8 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol), $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) and MeCN (1.5 mL). The tube was vacuumed and backfilled with $N_2$ for three times. The mixture was stirred at 40 $^oC$ for 4 h as monitored by TLC. Upon completion, the reaction mixture was cooled down to room temperature, and purified by flash column chromatography on silica gel to give the desired product **2a** as a yellow solid (53.3 mg, 90%), *E/Z* = 88/12.

***E* isomer**: IR (KBr, $cm^{-1}$): 1616, 1532, 1345, 816, 718; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.45 (s, 1H), 7.34 (d, $J$ = 8.5 Hz, 2H), 7.25 (d, $J$ = 8.5 Hz, 2H), 2.41 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 147.7 142.1, 135.3, 130.5, 129.4, 128.4, 21.7; EI-MS m/z (%): 199 (3) [$M^+$ ($^{37}Cl$)], 197 (8) [$M^+$ ($^{35}Cl$)], 182 (14), 139 (18), 134 (21), 116 (37), 115 (100), 89 (21), 77 (22), 63 (18); HRMS (EI) m/z calcd for $C_9H_8ClNO_2$ [$M^+$] 197.0244, found 197.0246.

***Z* isomer**: $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.73 (s, 1H), 7.57 (d, $J$ = 8.0 Hz, 2H), 7.28 (d, $J$ = 8.0 Hz, 2H), 2.42 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 143.2, 141.7, 133.8, 130.9, 130.0, 127.7, 21.6.

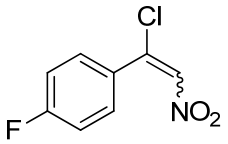
**Gram scale reaction for preparation of 2a**: To a 100 mL flask were added **1a** (1.16 g, 10 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (3.6240 g, 15 mmol), $SnCl_2 \cdot 2H_2O$ (2.2565 g, 10 mmol) and MeCN (50 mL). The mixture was stirred at 40 $^oC$ under $N_2$ for 4 h. Then the reaction mixture was cooled down to room temperature and filtered. The filtrate was concentrated and purified by column chromatography on silica gel to give the desired product as a yellow solid (1.5 g, 76%), *E/Z* = 96/4. Then the solid was recrystallized with hexane/ EtOAc and gave the pure *E*-product.




**1-(1-Chloro-2-nitrovinyl)-4-methoxybenzene (2b):** Following the general procedure as for **2a**, the reaction of **1b** (39.6 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 $^oC$ for 2 h afforded the desired product **2b** as a yellow solid (42.0 mg, 66%), *E/Z* = 7/93.

***Z* isomer**: IR (KBr, $cm^{-1}$): 1597, 1505, 1336, 1304, 1264, 1176, 1022, 831; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.71 (s, 1H), 7.64 (d, $J$ = 8.5 Hz, 2H), 6.97 (d, $J$ = 9.0 Hz, 2H), 3.87 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 163.0, 141.4, 132.8, 129.5, 125.6, 114.6, 55.6; EI-MS m/z (%): 215 (14) [$M^+$ ($^{37}Cl$)], 213 (43) [$M^+$ ($^{35}Cl$)], 155 (21), 135 (24), 132 (100), 117 (42), 89 (63), 63 (32); HRMS (EI) m/z calcd for $C_9H_8ClNO_3$ [$M^+$] 213.0193, found 213.0192.

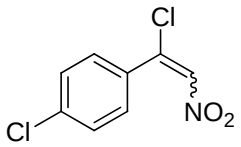
***E* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.42 (d, $J$ = 8.0 Hz, 2H), 7.26 (s, 1H), 6.93 (d, $J$ = 9.0 Hz, 2H), 3.85 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz) (partial data): $\delta$ 130.7, 113.9, 55.5.




**1-(1-Chloro-2-nitrovinyl)-4-fluorobenzene (2c):** Following the general procedure as for **2a**, the reaction of **1c** (36.0 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 $^oC$ for 4 h afforded the desired product **2c** as pale yellow oil (53.1 mg, 88%), *E/Z* = 92/8.

***E* isomer**: IR (KBr, $cm^{-1}$): 1607, 1529, 1344, 1236, 1164, 834, 716; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.50-7.41 (m, 3H), 7.20-7.09 (m, 2H); $^{19}F$ NMR ($CDCl_3$, 470 MHz): δ -107.3 (m, Ar-F); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 164.3 (d, $^1J_{C\text{-}F}$ = 252.0 Hz), 146.3, 135.9, 130.8 (d, $^3J_{C\text{-}F}$ = 9.0 Hz), 129.4 (d, $^4J_{C\text{-}F}$ = 3.5 Hz), 116.0 (d, $^2J_{C\text{-}F}$ = 22.0 Hz); EI-MS m/z (%): 203 (5) [$M^+$ ($^{37}Cl$)], 201 (14) [$M^+$ ($^{35}Cl$)], 143 (28), 123 (19), 120 (100), 107 (17), 99 (18); HRMS (EI) m/z calcd for $C_8H_5ClFNO_2$ [$M^+$] 200.9993, found 200.9994.

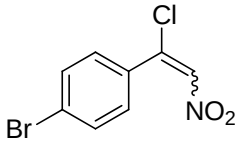
***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.71-7.65 (m, 3H), 7.20-7.09 (m, 2H); $^{19}F$ NMR ($CDCl_3$, 470 MHz): δ -106.3 (m, Ar-F); $^{13}C$ NMR ($CDCl_3$, 125 MHz) (partial data): $\delta$ 130.0 (d, $^3J_{C\text{-}F}$ = 9.0 Hz), 116.6 (d, $^2J_{C\text{-}F}$ = 22.0 Hz).




**1-Chloro-4-(1-chloro-2-nitrovinyl)benzene (2d):**[4,5] Following the general procedure as for **2a**, the reaction of **1d** (41.0 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 $^oC$ for 10 h afforded the desired product **2d** as a yellow solid (57.9 mg, 89%), *E/Z* = 93/7.

***E* isomer**: IR (KBr, $cm^{-1}$): 1621, 1527, 1346, 1093, 827, 722; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.47 (s, 1H), 7.42 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 9.0 Hz, 2H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 146.1, 137.6, 136.1, 131.8, 129.8, 129.0; LC-MS (ESI) m/z (%): 222 (12) [$M^+$ ($2\times^{37}Cl$)], 220 (65) [$M^+$ ($^{37}Cl$, $^{35}Cl$)], 218 (100) [$M^+$ ($2\times^{35}Cl$)].

***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.70 (s, 1H), 7.61 (d, *J* = 8.5 Hz, 2H), 7.42 (d, *J* = 8.5 Hz, 2H); $^{13}C$ NMR ($CDCl_3$, 125 MHz) (partial data): $\delta$ 129.6, 129.0.

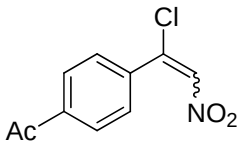



**1-Bromo-4-(1-chloro-2-nitrovinyl)benzene (2e):**[5] Following the general procedure as for **2a**, the reaction of **1e** (54.3 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 $^oC$ for 14 h afforded the desired product **2e** as a yellow solid (74.3 mg, 94%), *E/Z* = 90/10.

***E* isomer**: IR (KBr, $cm^{-1}$): 1612, 1525, 1341, 822, 720; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.60-7.56 (m, 2H), 7.47 (s, 1H), 7.35-7.28 (m, 2H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 146.1, 136.1, 132.3, 132.0, 129.9, 126.0; EI-MS m/z (%): 265 (6) [$M^+$ ($^{37}Cl$, $^{81}Br$)], 263 (25) {$M^+$ [($^{37}Cl$, $^{79}Br$) + ($^{35}Cl$, $^{81}Br$)]}, 261 (19) [$M^+$ ($^{35}Cl$, $^{79}Br$)], 198 (18), 182 (69), 152 (63), 136 (77), 101 (100), 75 (79), 50

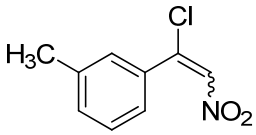
(38).

***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.71 (s, 1H), 7.64-7.60 (m, 2H), 7.56-7.52 (m, 2H); $^{13}$C NMR ($CDCl_3$, 125 MHz) (partial data): $\delta$ 132.6, 129.1.




**1-(4-(1-Chloro-2-nitrovinyl)phenyl)ethanone (2f):** Following the general procedure as for **2a**, the reaction of **1f** (43.3 mg, 0.3 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2{\cdot}2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 $^o$C for 10 h afforded the desired product **2f** as a yellow solid (57.9 mg, 86%), *E*/*Z* = 97/3.

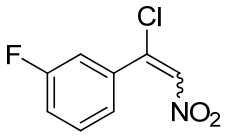
***E* isomer**: IR (KBr, cm$^{-1}$): 1678, 1607, 1515, 1348, 1264, 836, 723; $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 8.00 (d, *J* = 8.5 Hz, 2H), 7.50 (d, *J* = 8.5 Hz, 2H), 7.49 (s, 1H), 2.61 (s, 3H); $^{13}$C NMR ($CDCl_3$, 125 MHz): $\delta$ 197.1, 146.0, 138.7, 137.8, 136.6, 128.5, 128.4, 26.8; EI-MS m/z (%): 227 (0.67) [$M^+$ ($^{37}$Cl)], 225 (1.96) [$M^+$ ($^{35}$Cl)], 212 (21), 210 (63), 166 (31), 129 (100), 101 (46), 75 (27); HRMS (EI) m/z calcd for $C_{10}H_8ClNO_3$ [$M^+$] 225.0193, found 225.0195.




**1-(1-Chloro-2-nitrovinyl)-3-methylbenzene (2g):** Following the general procedure as for **2a**, the reaction of **1g** (39 μL, 0.3 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2{\cdot}2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 $^o$C for 4 h afforded the desired product **2g** as yellow oil (54.8 mg, 92%), *E*/*Z* = 90/10.

***E* isomer**: IR (KBr, cm$^{-1}$): 1612, 1527, 1345, 787, 718; $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.45 (s, 1H), 7.37-7.27 (m, 2H), 7.24 (s, 1H), 7.23 (d, *J* = 8.5, 1H), 2.40 (s, 3H); $^{13}$C NMR ($CDCl_3$, 125 MHz): $\delta$ 147.5, 138.5, 135.6, 133.4, 132.0, 128.6, 128.5, 125.2, 21.3; EI-MS m/z (%): 199 (2) [$M^+$ ($^{37}$Cl)], 197 (7) [$M^+$ ($^{35}$Cl)], 182 (14), 162 (16), 116 (34), 115 (100), 89 (27), 77 (26), 63 (20); HRMS (EI) m/z calcd for $C_9H_8ClNO_2$ [$M^+$] 197.0244, found 197.0247.

***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.72 (s, 1H), 7.51-7.47 (m, 2H), 7.37-7.27 (m, 2H), 2.42 (s, 3H); $^{13}$C NMR ($CDCl_3$, 125 MHz) (partial data): $\delta$ 139.1, 134.3, 133.6, 133.0, 129.1, 128.2, 124.9, 21.4.

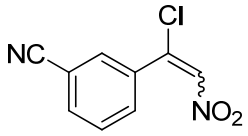



**1-(1-Chloro-2-nitrovinyl)-3-fluorobenzene (2h):** Following the general procedure as for **2a**, the reaction of **1h** (35 μL, 0.3 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2{\cdot}2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 $^o$C for 4 h afforded the desired product **2h** as yellow oil (53.2 mg, 88%), *E*/*Z* = 94/6.

***E* isomer**: IR (KBr, cm$^{-1}$): 1585, 1530, 1348, 1230, 790, 720; $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.47 (s, 1H), 7.46-7.38 (m, 1H), 7.22-7.12 (m, 3H); $^{19}$F NMR ($CDCl_3$, 470 MHz): δ -111.3 (m, Ar-F); $^{13}$C NMR ($CDCl_3$, 125 MHz): $\delta$ 162.4 (d, $^1J_{C\text{-}F}$ = 250.0 Hz), 145.5, 136.4, 135.3 (d, $^3J_{C\text{-}F}$ = 7.5 Hz),

130.5 (d, $^3J_{C\text{-}F}$ = 8.0 Hz), 124.0 (d, $^4J_{C\text{-}F}$ = 4.0 Hz), 118.3 (d, $^2J_{C\text{-}F}$ = 21.0 Hz), 115.5 (d, $^2J_{C\text{-}F}$ = 24.0 Hz); EI-MS m/z (%): 203 (1) [$M^+$ ($^{37}$Cl)], 201 (4) [$M^+$ ($^{35}$Cl)], 143 (33), 138 (21), 120 (100), 107 (23), 99 (27), 83 (23); HRMS (EI) m/z calcd for $C_8H_5ClFNO_2$ [$M^+$] 200.9993, found 200.9991.

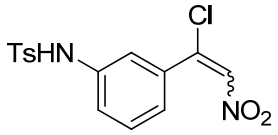
***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.71 (s, 1H), 7.46-7.38 (m, 3H), 7.22-7.12 (m, 1H); $^{19}$F NMR ($CDCl_3$, 470 MHz): δ -110.6 (m, Ar-F); $^{13}$C NMR ($CDCl_3$, 125 MHz) (partial data): 131.0 (d, $^3J_{C\text{-}F}$ = 7.5 Hz), 123.3 (d, $^4J_{C\text{-}F}$ = 4.0 Hz), 119.2 (d, $^2J_{C\text{-}F}$ = 20.0 Hz), 115.1 (d, $^2J_{C\text{-}F}$ = 25.0 Hz).




**3-(1-Chloro-2-nitrovinyl)benzonitrile (2i):** Following the general procedure as for **2a**, the reaction of **1i** (38.1 mg, 0.3 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2{\cdot}2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 $^o$C for 8 h afforded the desired product **2i** as a yellow solid (52.1 mg, 83%), *E*/*Z* = 93/7.

***E* isomer**: IR (KBr, cm$^{-1}$): 3104, 2227, 1610, 1520, 1333, 989, 799, 693; $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.77 (d, *J* = 8.0 Hz, 1H), 7.72 (s, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.58 (t, *J* = 8.0 Hz, 1H), 7.53 (s, 1H); $^{13}$C NMR ($CDCl_3$, 125 MHz): $\delta$ 144.5, 137.1, 134.9, 134.3, 132.3, 131.7, 129.7, 117.6, 113.3; EI-MS m/z (%): 210 (2) [$M^+$ ($^{37}$Cl)], 208 (6) [$M^+$ ($^{35}$Cl)], 150 (57), 145 (29), 127 (100), 126 (40), 117 (33), 100 (34), 76 (28), 75 (35); HRMS (EI) m/z calcd for $C_9H_5ClN_2O_2$ [$M^+$] 208.0040, found 208.0033.

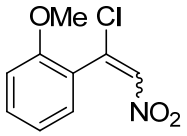
***Z* isomer** (partial data from spectra of mixture with *E*/*Z* isomers): $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.97 (s, 1H), 7.89 (d, *J* = 7.5 Hz, 1H), 7.82 (d, *J* = 7.5 Hz, 1H), 7.72 (s, 1H), 7.58 (t, *J* = 8.0 Hz, 1H).




***N*-(3-(1-Chloro-2-nitrovinyl)phenyl)-4-methylbenzenesulfonamide (2j):** Following the general procedure as for **2a**, the reaction of **1j** (81.4 mg, 0.3 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2{\cdot}2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 $^o$C for 10 h afforded the desired product **2j** as red oil (89.1 mg, 84%), *E*/*Z* = 91/9.

***E* isomer**: IR (KBr, cm$^{-1}$): 3259, 1526, 1337, 1158, 1091; $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.68 (d, *J* = 8.0 Hz, 2H), 7.63 (s, 1H), 7.42 (s, 1H), 7.33-7.20 (m, 4H), 7.15 (d, *J* = 7.5 Hz, 1H), 7.11 (d, *J* = 8.0 Hz, 1H), 2.36 (s, 3H); $^{13}$C NMR ($CDCl_3$, 125 MHz): $\delta$ 146.2, 144.5, 137.2, 136.3, 135.4, 134.7, 129.9, 129.8, 127.3, 124.6, 123.7, 120.5, 21.6; EI-MS m/z (%): 354 (4) [$M^+$ ($^{37}$Cl)], 352 (13) [$M^+$ ($^{35}$Cl)], 209 (27), 197 (33), 181 (100), 165 (28), 155 (36), 91 (85); HRMS (EI) m/z calcd for $C_{15}H_{13}ClN_2O_4S$ [$M^+$] 352.0285, found 352.0288.

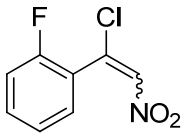
***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.74 (d, *J* = 8.0 Hz, 2H), 7.63 (s, 1H), 7.37-7.35 (m, 1H), 7.33-7.20 (m, 6H), 2.37 (s, 3H); $^{13}$C NMR ($CDCl_3$, 125 MHz) (partial data): $\delta$ 146.4, 144.6, 137.9, 135.6, 134.9, 130.3, 130.2, 130.0, 127.4, 124.2, 124.1, 119.7.




**1-(1-Chloro-2-nitrovinyl)-2-methoxybenzene (2k):** Following the general procedure as for **2a**, the reaction of **1k** (39 μL, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 30 °C for 4 h afforded the desired product **2k** as yellow oil (53.6 mg, 84%), *E*/*Z* = 92/8.

***E* isomer**: IR (KBr, $cm^{-1}$): 1625, 1528, 1349, 1286, 1252, 1022, 756; $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.49 (s, 1H), 7.44 (dt, *J* = 7.5, 1.5 Hz, 1H), 7.35 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.93 (d, *J* = 8.5 Hz, 1H), 3.81 (s, 3H); $^{13}$C NMR ($CDCl_3$, 125 MHz): $\delta$ 156.1, 143.6, 137.2, 132.6, 129.6, 123.0, 120.9, 111.2, 55.8; EI-MS m/z (%): 215 (24) [$M^+$ ($^{37}$Cl)], 213 (72) [$M^+$ ($^{35}$Cl)], 182 (38), 152 (63), 139 (34), 131 (72), 103 (41), 89 (100), 77 (42), 63 (52); HRMS (EI) m/z calcd for $C_9H_8ClNO_3$ [$M^+$] 213.0193, found 213.0192.

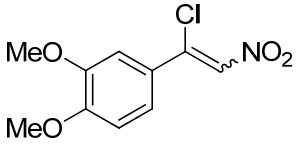
***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.87 (s, 1H), 7.44 (dt, *J* = 7.5, 1.5 Hz, 1H), 7.35 (dd, *J* = 7.5, 1.5 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.93 (d, *J* = 8.5 Hz, 1H), 3.90 (s, 3H); $^{13}$C NMR ($CDCl_3$, 125 MHz) (partial data): $\delta$ 132.8, 131.2, 121.1, 111.7, 100.1, 56.0.




**1-(1-Chloro-2-nitrovinyl)-2-fluorobenzene (2l):** Following the general procedure as for **2a**, the reaction of **1l** (34 μL, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 °C for 4 h afforded the desired product **2l** as pale yellow oil (49.3 mg, 82%), *E*/*Z* = 94/6.

***E* isomer**: IR (KBr, $cm^{-1}$): 1626, 1530, 1348, 1230, 828, 761, 718; $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.56 (s, 1H), 7.51-7.46 (m, 1H), 7.44-7.40 (m, 1H), 7.28-7.24 (m, 1H), 7.17-7.12 (m, 1H); $^{19}$F NMR ($CDCl_3$, 470 MHz): δ -111.4 (m, Ar-F); $^{13}$C NMR ($CDCl_3$, 125 MHz): $\delta$ 158.7 (d, $^1J_{C\text{-}F}$ = 250.0 Hz), 140.9, 138.1, 133.2 (d, $^3J_{C\text{-}F}$ = 9.0 Hz), 129.7, 124.7 (d, $^4J_{C\text{-}F}$ = 4.0 Hz), 122.0 (d, $^2J_{C\text{-}F}$ = 15.0 Hz), 116.1 (d, $^2J_{C\text{-}F}$ = 21.0 Hz); EI-MS m/z (%): 203 (6) [$M^+$ ($^{37}$Cl)], 201 (18) [$M^+$ ($^{35}$Cl)], 143 (48), 120 (100), 107 (23), 99 (29); HRMS (EI) m/z calcd for $C_8H_5ClFNO_2$ [$M^+$] 200.9993, found 200.9991.

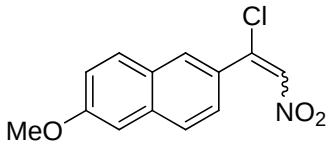
***Z* isomer** (partial data from spectra of mixture with *E*/*Z* isomers): $^1$H NMR ($CDCl_3$, 500 MHz): $\delta$ 7.76 (s, 1H), 7.72-7.66 (m, 1H), 7.51-7.46 (m, 1H), 7.31-7.28 (m, 1H), 7.22-7.17 (m, 1H); $^{19}$F NMR ($CDCl_3$, 470 MHz): δ -111.3 (m, Ar-F).




**4-(1-Chloro-2-nitrovinyl)-1,2-dimethoxybenzene (2m):** Following the general procedure as for **2a**, the reaction of **1m** (48.7 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 °C for 2 h afforded the desired product **2m** as a yellow solid (45.2 mg, 62%), *E*/*Z* = 30/70.

***Z* isomer**: IR (KBr, $cm^{-1}$): 1586, 1516, 1328, 1267, 1144, 1018, 808; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.72 (s, 1H), 7.31 (dd, $J$ = 8.5, 2.0 Hz, 1H), 7.14 (d, $J$ = 2.5 Hz, 1H), 6.91 (d, $J$ = 8.5 Hz, 1H), 3.94 (s, 3H), 3.93 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 152.7, 149.4, 141.5, 133.1, 126.0, 121.6, 111.1, 110.3, 56.3, 56.2; EI-MS m/z (%): 245 (21) [$M^+$ ($^{37}Cl$)], 243 (70) [$M^+$ ($^{35}Cl$)], 191 (100), 165 (64), 162 (60), 147 (34), 120 (38), 91 (53), 77 (40), 75 (42); HRMS (EI) m/z calcd for $C_{10}H_{10}ClNO_4$ [$M^+$] 243.0298, found 243.0300.

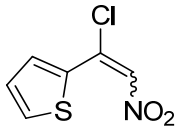
***E* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.41 (s, 1H), 7.08 (dd, $J$ = 8.5, 2.0 Hz, 1H), 6.96-6.85 (m, 2H), 3.92 (s, 3H), 3.88 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 151.8, 148.8, 147.3, 134.7, 125.3, 122.6, 111.5, 110.7, 56.2, 56.1.




**2-(1-Chloro-2-nitrovinyl)-6-methoxynaphthalene (2n):** Following the general procedure as for **2a**, the reaction of **1n** (54.7 mg, 0.3 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2{\cdot}2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 30 $^oC$ for 6 h afforded the desired product **2n** as a yellow solid (67.2 mg, 85%), *E*/*Z* = 81/19.

***E* isomer**: IR (KBr, $cm^{-1}$): 1598, 1510, 1337, 1224, 1163, 1025, 814; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.91 (s, 1H), 7.84-7.74 (m, 2H), 7.52 (s, 1H), 7.43 (d, $J$ = 8.5 Hz, 1H), 7.24-7.11 (m, 2H), 3.94 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 159.7, 148.0, 136.1, 135.3, 130.6, 129.2, 128.2, 127.9, 127.1, 125.5, 120.1, 105.9, 55.5; EI-MS m/z (%): 265 (16) [$M^+$ ($^{37}Cl$)], 263 (45) [$M^+$ ($^{35}Cl$)], 233 (21), 182 (63), 139 (100), 44 (50); HRMS (EI) m/z calcd for $C_{13}H_{10}ClNO_3$ [$M^+$] 263.0349, found 263.0345.

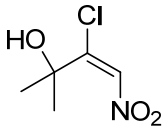
***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 8.17 (s, 1H), 7.86 (s, 1H), 7.84-7.74 (m, 2H), 7.63 (d, $J$ = 9.0 Hz, 1H), 7.24-7.11 (m, 2H), 3.95 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz) (partial data): $\delta$ 133.9, 130.7, 129.0, 128.3, 124.0, 120.5, 105.8, 55.6.



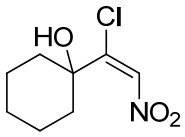

**2-(1-Chloro-2-nitrovinyl)thiophene (2o):** Following the general procedure as for **2a**, the reaction of **1o** (30 μL, 0.3 mmol), $Cu(NO_3)_2{\cdot}3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2{\cdot}2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 30 $^oC$ for 4 h afforded the desired product **2o** as yellow oil (49.6 mg, 87%), *E*/*Z* = 57/43.

***E* isomer**: IR (KBr, $cm^{-1}$): 3106, 1581, 1511, 1323, 722; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.92 (d, $J$ = 3.5 Hz, 1H), 7.74 (d, $J$ = 5.0 Hz, 1H), 7.41 (s, 1H), 7.15 (t, $J$ = 4.5 Hz, 1H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 139.5, 136.3, 134.8, 131.8, 131.7, 127.7; EI-MS m/z (%): 191 (4) [$M^+$ ($^{37}Cl$)], 189 (10) [$M^+$ ($^{35}Cl$)], 146 (37), 118 (40), 108 (100), 83 (43), 69 (33); HRMS (EI) m/z calcd for $C_6H_4ClNO_2S$ [$M^+$] 188.9651, found 188.9652.

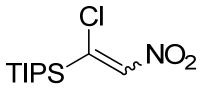
***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.84 (s, 1H), 7.67 (d, $J$ = 3.5 Hz, 1H), 7.58 (d, $J$ = 5.0 Hz, 1H), 7.15 (t, $J$ = 4.5 Hz, 1H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 136.7, 135.0, 133.7, 132.0, 131.6, 128.9.



**(*E*)-3-Chloro-2-methyl-4-nitrobut-3-en-2-ol (2p):** Following the general procedure as for **2a**, the reaction of **1p** (29 μL, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 60 $^oC$ for 21 h afforded the desired product **2p** as pale yellow oil (18.0 mg, 36%), *E/Z* > 19:1. IR (KBr, $cm^{-1}$): 3555, 2987, 2932, 1617, 1526, 1350, 1189, 832; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.75 (s, 1H), 2.11 (s, 1H), 1.56 (s, 6H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 148.8, 134.5, 74.7, 28.6; EI-MS m/z (%): 167 (0.87) [$M^+$ ($^{37}Cl$)], 165 (2.73) [$M^+$ ($^{35}Cl$)], 152 (23), 150 (72), 105 (49), 59 (99), 43 (100); HRMS (EI) m/z calcd for $C_5H_8ClNO_3$ [$M^+$] 165.0193, found 165.0195.



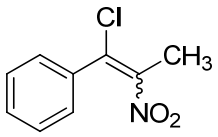
**(*E*)-1-(1-Chloro-2-nitrovinyl)cyclohexanol (2q):** Following the general procedure as for **2a**, the reaction of **1q** (37.3 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 60 $^oC$ for 26 h afforded the desired product **2q** as pale yellow oil (24.6 mg, 40%), *E/Z* > 19:1. IR (KBr, $cm^{-1}$): 3525, 2938, 2861, 1613, 1525; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.76 (s, 1H), 2.03-1.91 (m, 3H), 1.76-1.60 (m, 7H), 1.29-1.17 (m, 1H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 149.5, 134.9, 75.8, 34.8, 24.8, 21.1; EI-MS m/z (%): 207 (0.13) [$M^+$ ($^{37}Cl$)], 205 (0.36) [$M^+$ ($^{35}Cl$)], 170 (33), 99 (48), 81 (100), 55 (86); HRMS (EI) m/z calcd for $C_8H_{12}ClNO_3$ [$M^+$] 205.0506, found 205.0509.



**(1-Chloro-2-nitrovinyl)triisopropylsilane (2r):** Following the general procedure as for **2a**, the reaction of **1r** (67 μL, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 60 $^oC$ for 16 h afforded the desired product **2r** as pale yellow oil (23.6 mg, 30%), *E/Z* = 17/83.

***Z* isomer**: IR (KBr, $cm^{-1}$): 2954, 2873, 1522, 1463, 1353, 882, 669; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.83 (s, 1H), 1.64-1.56 (m, 3H), 1.16 (d, *J* = 7.5 Hz, 18H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 157.1, 149.3, 18.9, 12.5; EI-MS m/z (%): 265 (0.68) [$M^+$ ($^{37}Cl$)], 263 (1.90) [$M^+$ ($^{35}Cl$)], 222 (37), 220 (100), 160 (32), 149 (19), 132 (28), 103 (26); HRMS (EI) m/z calcd for $C_{11}H_{22}ClNO_2Si$ [$M^+$] 263.1108, found 263.1104.

***E* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.80 (s, 1H), 1.64-1.56 (m, 3H), 1.16 (d, *J* = 7.5 Hz, 18H); $^{13}C$ NMR ($CDCl_3$, 125 MHz) (partial data): $\delta$ 18.6, 12.3.

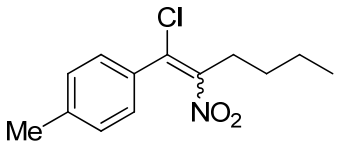


**(1-Chloro-2-nitroprop-1-en-1-yl)benzene (2s):**[6] Following the general procedure as for **2a**, the

reaction of **1s** (38 μL, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 60 °C for 12 h afforded the desired product **2s** as pale yellow oil (50.6 mg, 85%), *E*/*Z* = 70/30.

***E* isomer**: IR (KBr, $cm^{-1}$): 1532, 1442, 1348, 1027, 761, 697; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.55-7.29 (m, 5H), 2.51 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 146.4, 137.1, 135.2, 130.3, 128.8, 127.7, 18.0; EI-MS m/z (%): 199 (0.51) [$M^+$ ($^{37}Cl$)], 197 (1.60) [$M^+$ ($^{35}Cl$)], 169 (8), 134 (14), 116 (20), 115 (100), 105 (17), 103 (18), 89 (17), 77 (19), 63 (14).

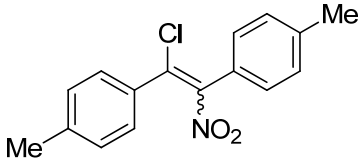
***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.55-7.29 (m, 5H), 2.26 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz) (partial data): 134.4, 130.2, 128.9, 127.6, 18.1.




**1-(1-Chloro-2-nitrohex-1-en-1-yl)-4-methylbenzene (2t):** Following the general procedure as for **2a**, the reaction of **1t** (51.7 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 40 °C for 18 h afforded the desired product **2t** as yellow oil (53.3 mg, 70%), *E*/*Z* = 55/45.

***E* isomer**: IR (KBr, $cm^{-1}$): 2960, 2930, 1533, 1458, 1357, 814; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.30-7.23 (m, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 2.90 (t, *J* = 8.0 Hz, 2H), 2.38 (s, 3H), 1.66-1.60 (m, 2H), 1.53-1.43 (m, 2H), 0.99 (t, *J* = 7.5 Hz, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 150.9, 140.6, 135.7, 132.2, 129.5, 128.6, 31.6, 28.7, 22.1, 21.5, 13.8; EI-MS m/z (%): 255 (3) [$M^+$ ($^{37}Cl$)], 253 (9) [$M^+$ ($^{35}Cl$)], 183 (22), 176 (27), 129 (99), 128 (60), 119 (100), 115 (31), 91 (23); HRMS (EI) m/z calcd for $C_{13}H_{16}ClNO_2$ [$M^+$] 253.0870, found 253.0874.

***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.32 (d, *J* = 8.5 Hz, 2H), 7.30-7.23 (m, 2H), 2.59 (t, *J* = 8.0 Hz, 2H), 2.43 (s, 3H), 1.53-1.43 (m, 2H), 1.34-1.30 (m, 2H), 0.86 (t, *J* = 7.5 Hz, 2H); $^{13}C$ NMR ($CDCl_3$, 125 MHz) (partial data): 140.5, 131.5, 129.6, 127.7, 127.0, 31.1, 29.2, 21.9, 13.7.

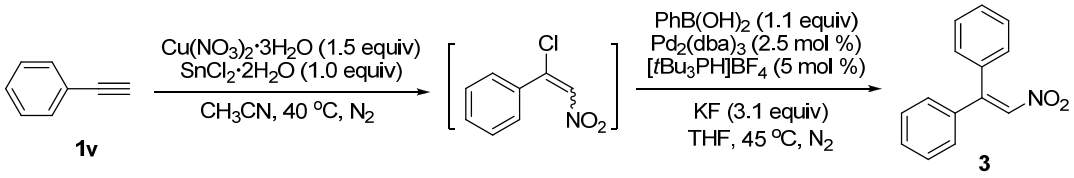



**4,4'-(1-Chloro-2-nitroethene-1,2-diyl)bis(methylbenzene) (2u):** Following the general procedure as for **2a**, the reaction of **1u** (61.9 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) in MeCN (1.5 mL) at 80 °C for 24 h afforded the desired product **2u** as a yellow solid (65.5 mg, 76%), *E*/*Z* = 62/38.

***E* isomer**: IR (KBr, $cm^{-1}$): 1531, 1360, 1184, 819, 750; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.22 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 4H), 2.33 (s, 3H), 2.31 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 141.0, 140.6, 129.8, 129.7, 129.6, 129.5, 129.4, 129.0, 128.9, 127.8, 21.5, 21.4; EI-MS m/z (%): 289 (9) [$M^+$ ($^{37}Cl$)], 287 (27) [$M^+$ ($^{35}Cl$)], 241 (66), 206 (100), 189 (23); HRMS (EI) m/z calcd for $C_{16}H_{14}ClNO_2$ [$M^+$] 287.0713, found 287.0715.

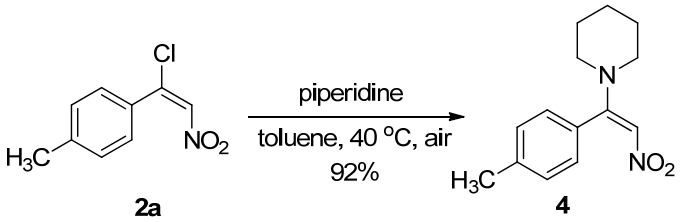
***Z* isomer** (data from spectra of mixture with *E*/*Z* isomers): $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.52 (d, *J* = 8.0 Hz, 2H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 2.42 (s, 3H), 2.39 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz) (partial data): $\delta$ 149.4, 141.0, 140.6, 134.9,

132.1, 131.0, 128.0, 127.9, 127.4, 21.6, 21.5.

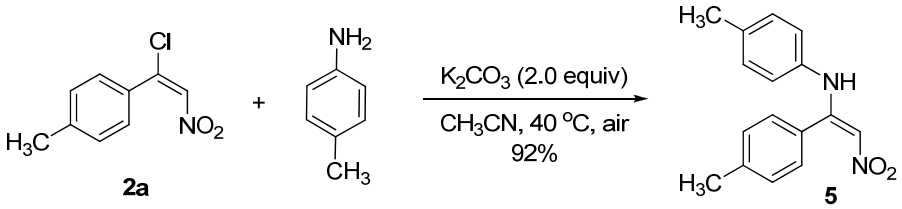



**(2-Nitroethene-1,1-diyl)dibenzene (3):**[7] To a test tube were added $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol), $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) and MeCN (1.5 mL), the tube was vacuumed and refilled with $N_2$ for three times, then **1v** (33μL, 0.3 mmol) was added, and the reaction was stirred at 40 °C for 4 h. After cooled down to room temperature, the mixture was filtered and the solvent was removed. To the residue were added $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), $[t\text{Bu}_3\text{PH}]BF_4$ (4.4 mg, 0.015 mmol), $PhB(OH)_2$ (40.2 mg, 0.33 mmol), KF (54.0 mg, 0.93 mmol) and THF (1.0 mL). The flask was vacuumed and backfilled with $N_2$, and the reaction was stirred at 45 °C for 24 h. Upon completion, the reaction was quenched with $H_2O$ and extracted with EA (3×10 mL). The combined organic layer was washed with brine. After dried over with anhydrous $Na_2SO_4$, the mixture was evaporated and purified by flash column chromatography on silica gel to give the desired product **3** (40.2 mg, 59%) as a yellow solid. m.p. 85-87 °C; IR (KBr, $cm^{-1}$): 1619, 1508, 1335, 691; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.51-7.34 (m, 7H), 7.32-7.27 (m, 2H), 7.25-7.18 (m, 2H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 150.6, 137.2, 135.7, 134.5, 131.0, 129.4, 129.1, 129.0, 128.9, 128.6; EI-MS m/z (%): 225 (15) [$M^+$], 178 (100), 168 (38), 165 (51), 152 (54).

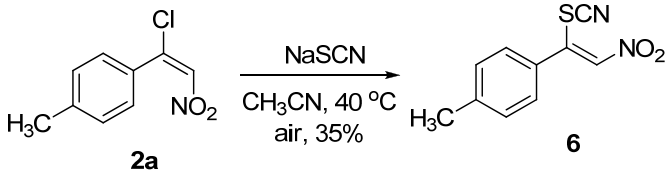
# 3. Application of Compound 2a



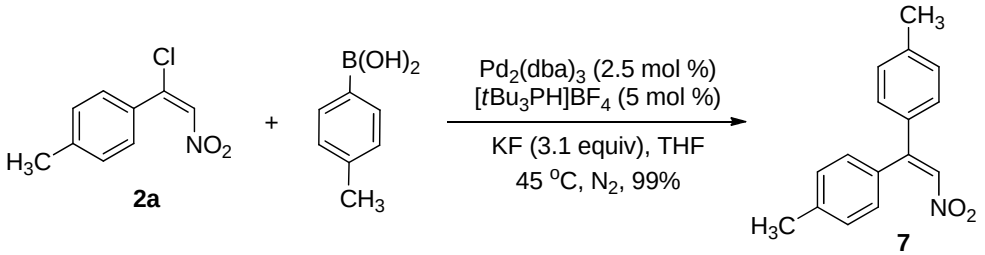

**(*E*)-1-(2-Nitro-1-(*p*-tolyl)vinyl)piperidine (4):** To a test tube were added **2a** (59.3 mg, 0.3 mmol), piperidine (63.9 mg, 0.75 mmol) and toluene (1.5 mL). The mixture was stirred at 40 °C for 3 h under air as monitored by TLC. Upon completion, the reaction mixture was cooled down to room temperature and purified by flash column chromatography to give the desired product **4** as a yellow solid (68.1 mg, 92%). m.p. 131-132 °C; IR (KBr, $cm^{-1}$): 2948, 1525, 1454, 1409, 1344, 1291, 1241, 1014, 782; $^1H$ NMR ($d_6$-DMSO, 500 MHz): $\delta$ 7.27 (d, $J$ = 7.5 Hz, 2H), 7.15 (s, 2H), 6.99 (s, 1H), 3.69-2.85 (m, 4H), 2.36 (s, 3H), 1.93-1.23 (m, 6H); $^{13}C$ NMR ($d_6$-DMSO, 125 MHz): $\delta$ 161.3, 139.2, 131.6, 129.8, 127.9, 113.6, 50.0, 25.9, 23.9, 21.4; EI-MS m/z (%): 246 (14) [$M^+$], 229 (18), 200 (51), 130 (20), 117 (54), 116 (22), 91 (27), 84 (100); HRMS (EI) m/z calcd for $C_{14}H_{18}N_2O_2$ [$M^+$] 246.1368, found 246.1370.




**(*E*)-4-Methyl-*N*-(2-nitro-1-(*p*-tolyl)vinyl)aniline (5):** To a test tube were added **2a** (59.3 mg, 0.3 mmol), *p*-toluidine (48.2 mg 0.45 mmol), $K_2CO_3$ (82.9 mg, 0.6 mmol) and $CH_3CN$ (1.5 mL). The mixture was stirred at 40 °C for 12 h under air as monitored by TLC. Upon completion, the reaction mixture was cooled down to room temperature, washed with HCl (0.1 M) for three times, and extracted with EA; the organic layer was then washed with $H_2O$ and brine, dried over anhydrous $Na_2SO_4$, filtered and concentrated in vacuo. The residue was purified by column chromatography on silica gel to give the desired product **5** as a yellow solid (74.0 mg, 92%). m.p. 112-113 °C; IR (KBr, $cm^{-1}$): 1598, 1553, 1464, 1355, 1272, 1182, 1102, 822; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 11.54 (s, 1H), 7.18 (d, $J$ = 8.0 Hz, 2H), 7.13 (d, $J$ = 8.0 Hz, 2H), 6.98 (d, $J$ = 8.0 Hz, 2H), 6.76 (s, 1H), 6.72 (d, $J$ = 8.5 Hz, 2H), 2.35 (s, 3H), 2.26 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 156.2, 141.4, 135.9, 135.2, 129.8, 129.7, 128.8, 128.6, 123.9, 113.8, 21.6, 21.0; EI-MS m/z (%): 268 (20) [$M^+$], 222 (100), 221 (31), 220 (25), 208 (45), 207 (49), 91 (37), 65 (24); HRMS (EI) m/z calcd for $C_{16}H_{16}N_2O_2$ [$M^+$] 268.1212, found 268.1215.



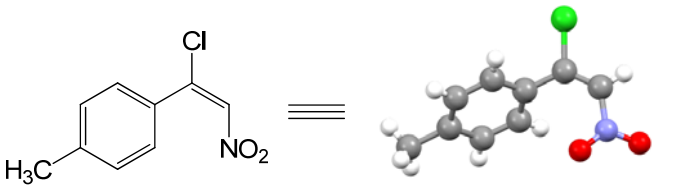

**(*Z*)-1-Methyl-4-(2-nitro-1-thiocyanatovinyl)benzene (6):** To a test tube were added **2a** (59.3 mg, 0.3 mmol), NaSCN (168.2 mg, 3.0 mmol) and $CH_3CN$ (1.5 mL). The mixture was stirred at 40 °C under air for 7 hours. Then the reaction mixture was cooled down to room temperature and purified by flash column chromatography to give the desired product **6** as a yellow solid (23.2 mg, 35%). m.p. 94-96 °C; IR (KBr, $cm^{-1}$): 2156, 1573, 1500, 1328, 819; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.37 (s, 1H), 7.34 (s, 4H), 2.44 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 148.7, 142.9, 135.2, 130.1, 129.2, 128.6, 108.3, 21.7; EI-MS m/z (%): 220 (16) [$M^+$], 119 (19), 116 (63), 115 (100), 91 (28); HRMS (EI) m/z calcd for $C_{10}H_8N_2O_2S$ [$M^+$] 220.0306, found 220.0304.




**4,4'-(2-Nitroethene-1,1-diyl)bis(methylbenzene) (7):**[7] To a test tube were added **2a** (59.3 mg, 0.3 mmol), *p*-tolylboronic acid (44.9 mg, 0.33 mmol), $Pd_2(dba)_3$ (6.9 mg, 0.0075 mmol), [$t$Bu$_3$PH]$BF_4$ (4.4 mg, 0.015 mmol), KF (54.0 mg, 0.93 mmol) and THF (1.0 mL). The flask was vacuumed and backfilled with $N_2$, and the reaction was stirred at 45 °C as monitored by TLC. Upon completion, the reaction was quenched with $H_2O$ and extracted with EA (3×10 mL). The combined organic

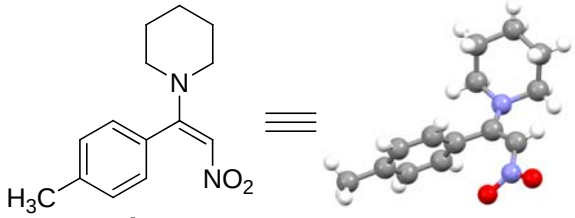
layer was washed with brine. After dried over with anhydrous $Na_2SO_4$, the mixture was evaporated and purified by flash column chromatography on silica gel to give the desired product **7** (75.2 mg, 99%) as a yellow solid. m.p. 111-113 $^oC$; IR (KBr, $cm^{-1}$): 1602, 1507, 1334, 1245, 816; $^1H$ NMR ($CDCl_3$, 500 MHz): $\delta$ 7.41 (s, 1H), 7.23 (d, $J$ = 7.5 Hz, 2H), 7.19 (s, 4H), 7.11 (d, $J$ = 7.5 Hz, 2H), 2.42 (s, 3H), 2.39 (s, 3H); $^{13}C$ NMR ($CDCl_3$, 125 MHz): $\delta$ 151.1, 141.6, 139.6, 134.7, 133.6, 132.8, 129.7, 129.3, 129.1, 129.0, 21.6, 21.5; EI-MS m/z (%): 253 (80) [$M^+$], 221 (53), 191 (97), 165 (100), 115 (91).

## 4. X-ray Crystallographic Data for Compounds (*E*)-2a, 4 and 6



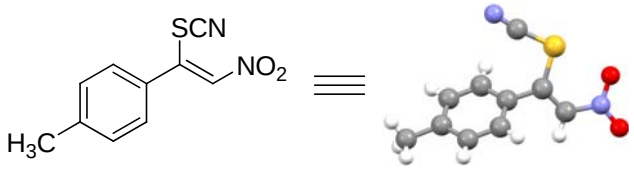

**(*E*)-2a** X-ray structure of **(*E*)-2a**

Crystallographic data for **(*E*)-2a**: $C_9H_8ClNO_2$, M = 197.61, Orthorhombic, P 21 21 21 (No. 19), a = 5.961 (5) Å, b = 7.819 (7) Å, c = 20.316 (18) Å, V = 946.9 (14) $Å^3$, Z = 4, Crystal size: 0.24 × 0.19 × 0.16 mm, T = 295 K, $\rho_{calcd}$ = 1.386 $g \cdot cm^{-3}$, $R_1$ = 0.0391 (I>4σ(I)), $wR_2$ = 0.1143 (all data), GOF = 1.060, reflections collected/unique: 5795 / 2227 (Rint = 0.0219), Data: 1833, restraints: 0, parameters: 123. CCDC 1487495 contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.




**4** X-ray structure of **4**

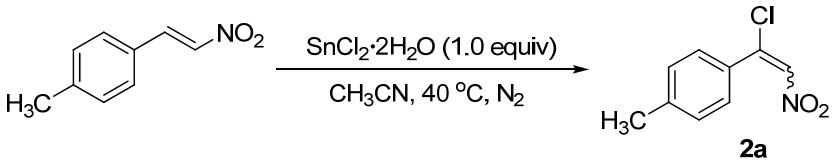
Crystallographic data for **4**: $C_{14}H_{18}N_2O_2$, M = 246.30, Monoclinic, P 21/c (No. 14), a = 8.018 (6) Å, b = 16.118 (13) Å, c = 10.272 (8) Å, V = 1322.7 (18) $Å^3$, Z = 4, Crystal size: 0.25 × 0.19 × 0.14 mm, T = 295 K, $\rho_{calcd}$ = 1.237 $g \cdot cm^{-3}$, $R_1$ = 0.0439 (I>4σ(I)), $wR_2$ = 0.1290 (all data), GOF = 1.050, reflections collected/unique: 8060 / 2984 (Rint = 0.0246), Data: 2243, restraints: 0, parameters: 168. CCDC 1487497 contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif




**6** X-ray structure of **6**

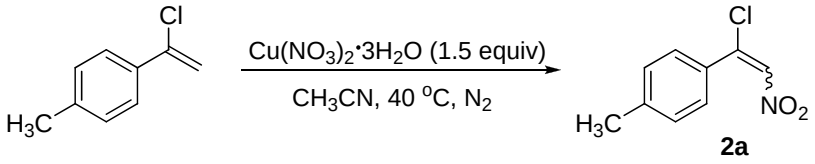
Crystallographic data for **6**: $C_{10}H_8N_2O_2S$, M = 220.24, Orthorhombic, P 21 21 21 (No. 19), a = 6.758 (5) Å, b = 8.245 (6) Å, c = 18.741 (15) Å, V = 1044.2 (14) $Å^3$, Z = 4, Crystal size: 0.23 × 0.19 × 0.15 mm, T = 295 K, $\rho_{calcd}$ = 1.401 $g \cdot cm^{-3}$, $R_1$ = 0.0329 (I>4σ(I)), $wR_2$ = 0.0830 (all data), GOF = 1.071, reflections collected/unique: 6187 / 2247 (Rint = 0.0302), Data: 1891, restraints: 0, parameters: 141. CCDC 1487496 contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

## 5. Mechanistic Studies



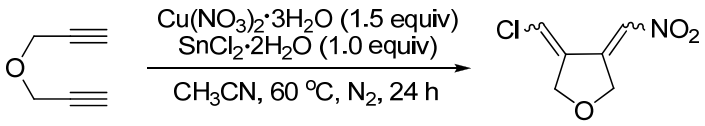

**2a**

To a test tube were added (*E*)-1-methyl-4-(2-nitrovinyl)benzene (49.0 mg, 0.3 mmol), $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) and MeCN (1.5 mL). The tube was vacuumed and backfilled with $N_2$ for three times. The mixture was stirred at 40 °C overnight, and no desired product was obtained.

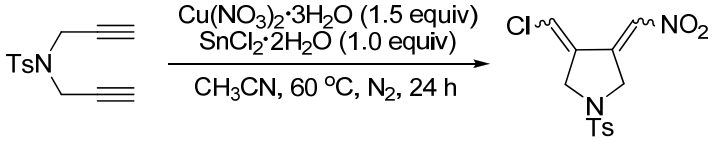



**2a**

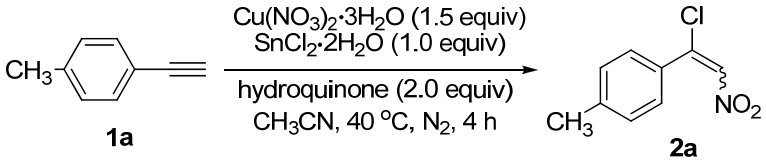
To a test tube were added 1-(1-chlorovinyl)-4-methylbenzene (45.8 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol) and MeCN (1.5 mL). The tube was vacuumed and backfilled with $N_2$ for three times. The mixture was stirred at 40 °C for 4 h, and no desired product was obtained.




To a test tube were added 3-(prop-2-yn-1-yloxy)prop-1-yne (28.2 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol), $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) and MeCN (1.5 mL). The tube was vacuumed and backfilled with $N_2$ for three times. The mixture was stirred at 60 °C for 24 h, and no desired cyclic product was obtained.

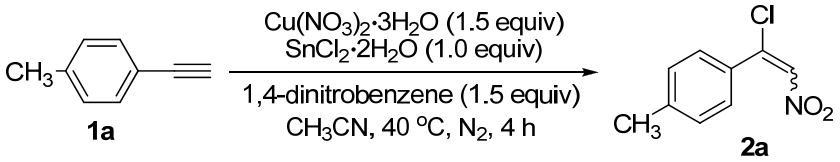



To a test tube were added 4-methyl-*N,N*-di(prop-2-yn-1-yl)benzenesulfonamide (34.8 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol), $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol) and MeCN (1.5 mL). The tube was vacuumed and backfilled with $N_2$ for three times. The mixture was stirred at 60 °C for 24 h, and no desired cyclic product was obtained.




**1a** **2a**

To a test tube were added **1a** (34.8 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol), $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol), hydroquinone (66.1 mg, 0.6 mmol) and MeCN (1.5 mL). The tube was vacuumed and backfilled with $N_2$ for three times. The mixture was stirred at 40 °C for 4 h. The reaction mixture was then cooled down to room temperature, and purified by flash column chromatography on silica gel to give the desired product **2a** as a yellow solid (40.8 mg, 69%), *E/Z* = 88/12.




**1a** **2a**

To a test tube were added **1a** (34.8 mg, 0.3 mmol), $Cu(NO_3)_2 \cdot 3H_2O$ (108.7 mg, 0.45 mmol), $SnCl_2 \cdot 2H_2O$ (67.7 mg, 0.3 mmol), 1,4-dinitrobenzene (75.6 mg, 0.45 mmol) and MeCN (1.5 mL). The tube was vacuumed and backfilled with $N_2$ for three times. The mixture was stirred at 40 °C for 4 h. The reaction mixture was then cooled down to room temperature, and purified by flash column chromatography on silica gel to give the desired product **2a** as a yellow solid (53.2 mg, 90%), *E/Z* = 85/15.

# References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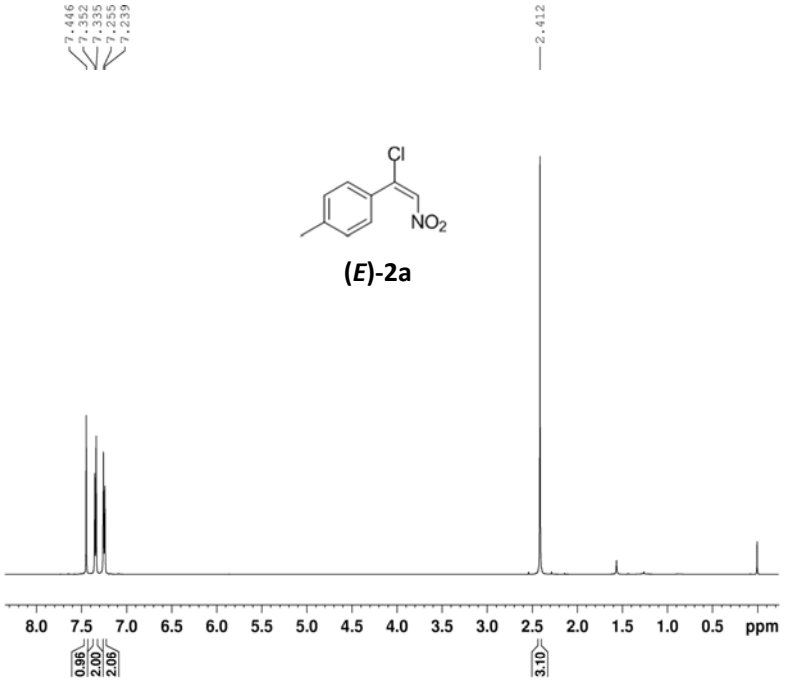


(*E*)-2a

GMC-11-1-4-2
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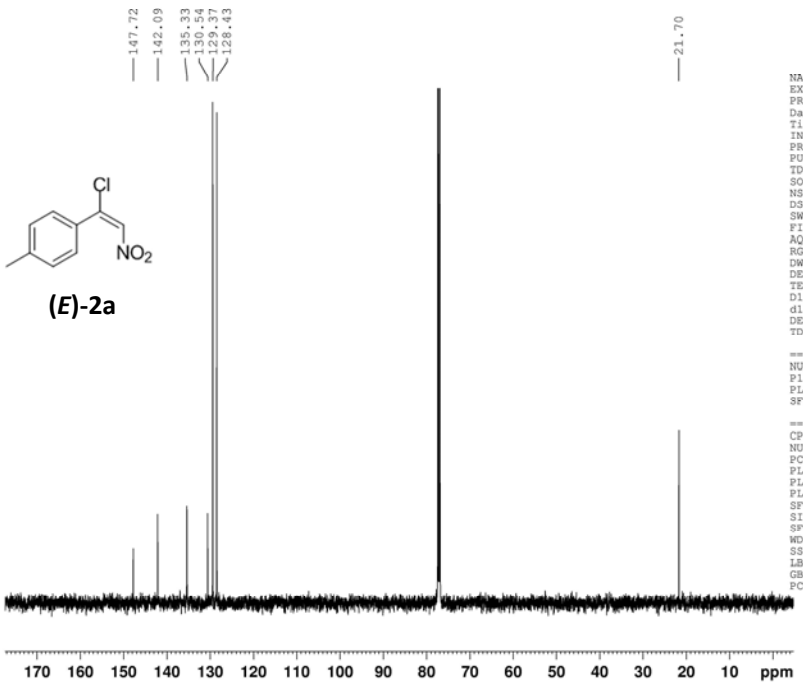


(*E*)-2a

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| NUC2 | 1H |
| PCPD2 | 80.00 usec |
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| PL12 | 16.05 dB |
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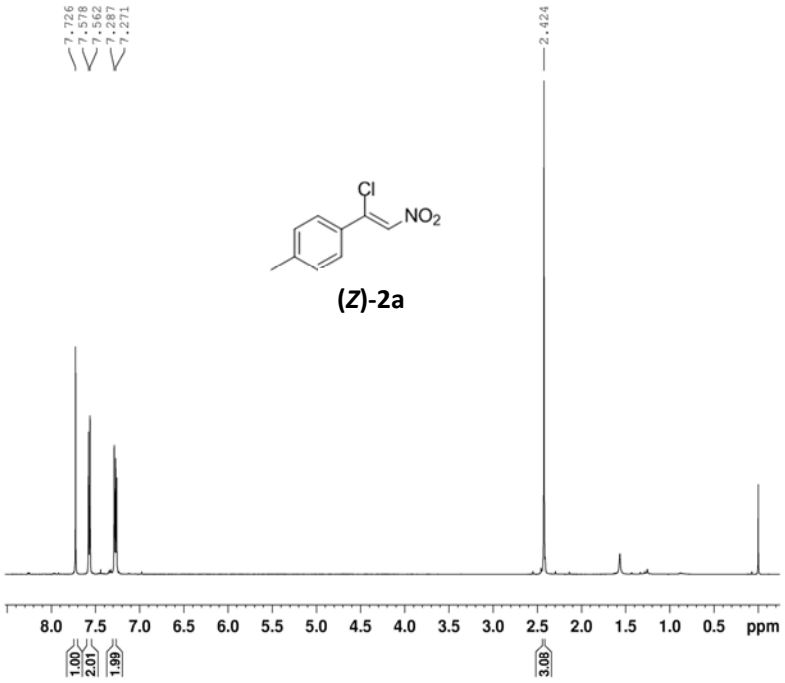


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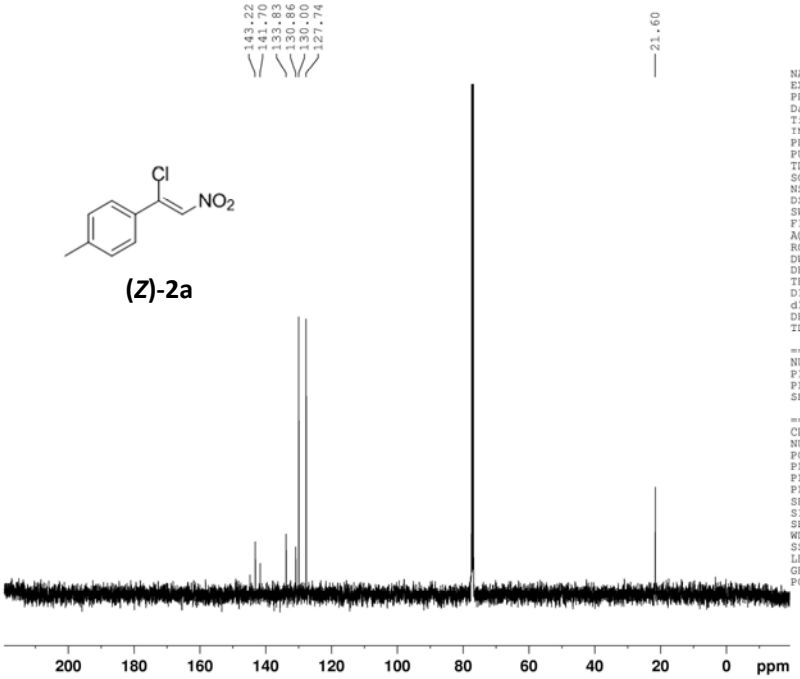


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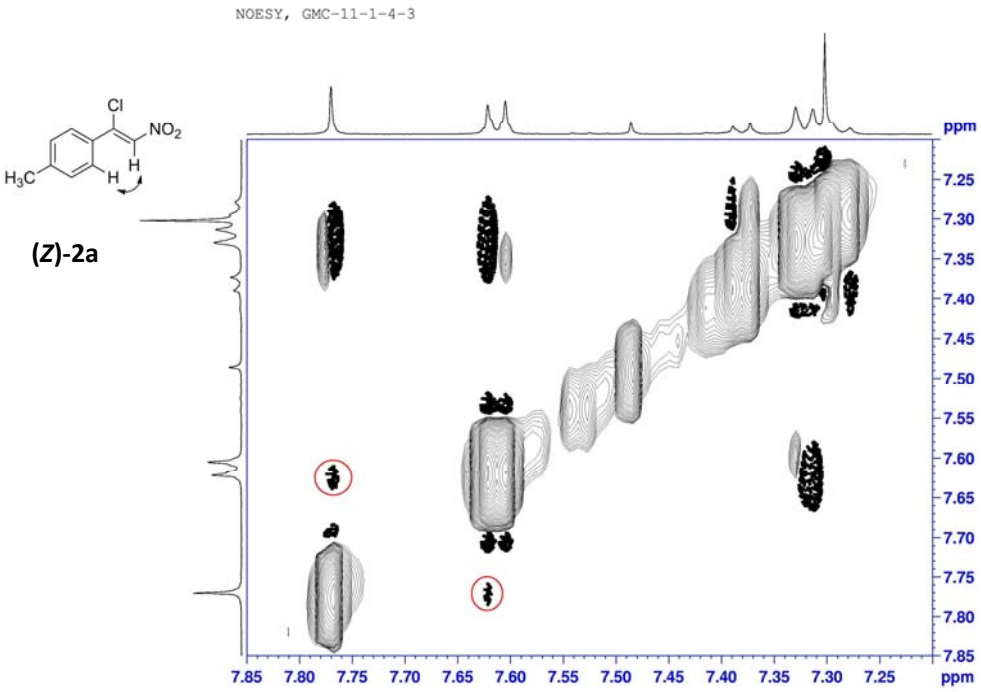
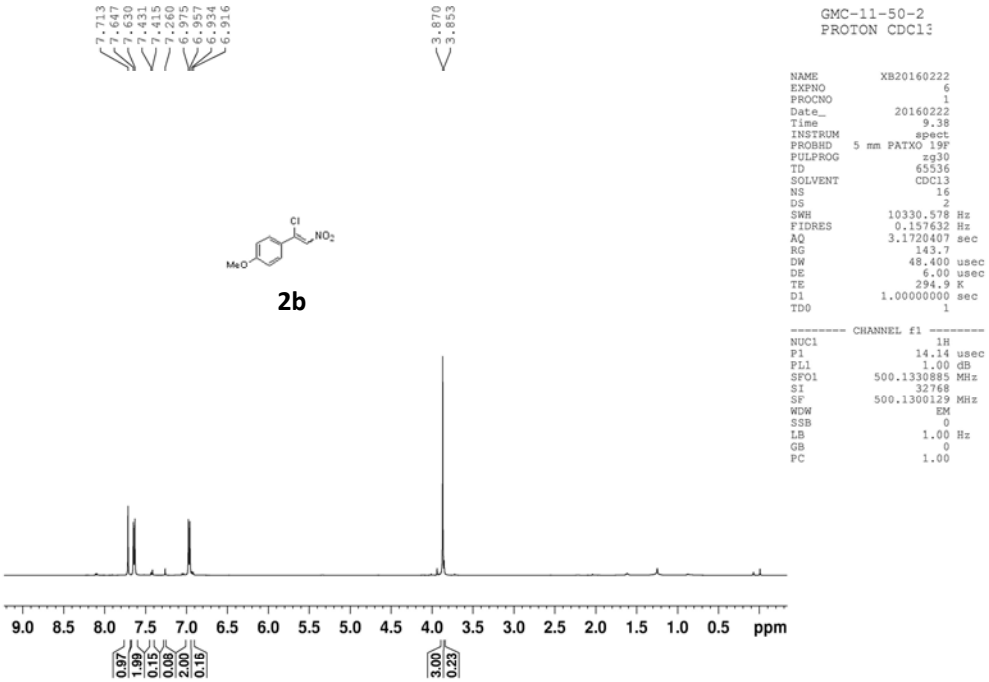
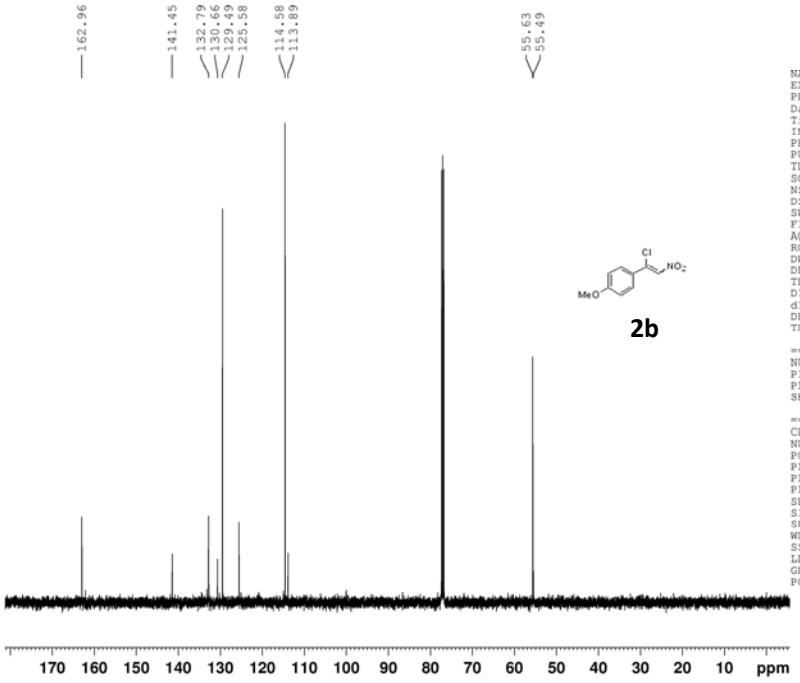
| DE | 6.00 usec |
| TE | 295.5 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577735 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 2.00 Hz |
| GB | 0 |
| PC | 1.00 |

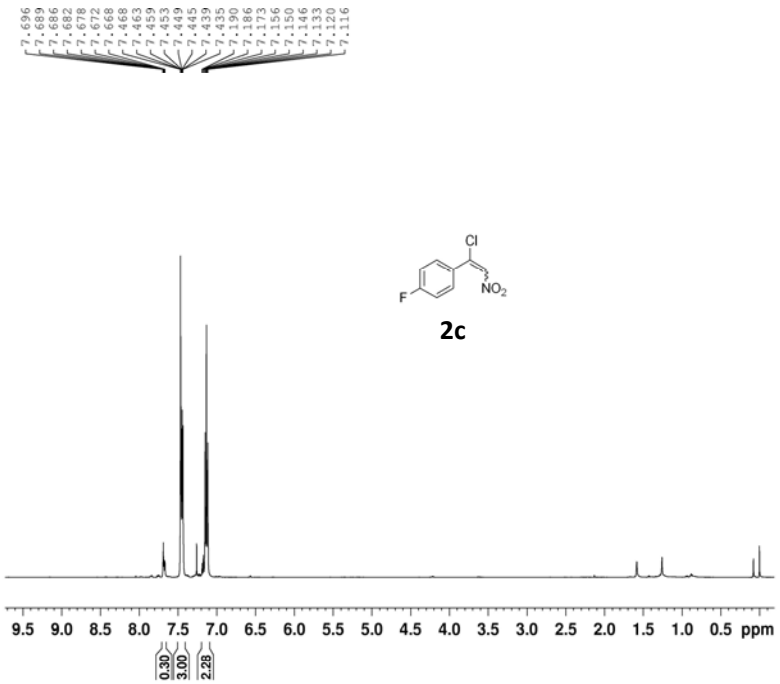

NOESY, GMC-11-1-4-3
Cl
NO2
H3C
H
H
(Z)-2a
ppm
7.25
7.30
7.35
7.40
7.45
7.50
7.55
7.60
7.65
7.70
7.75
7.80
7.85
7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45 7.40 7.35 7.30 7.25 ppm


7.713
7.647
7.630
7.431
7.415
7.260
6.975
6.957
6.934
6.916
3.870
3.853
GMC-11-50-2
PROTON CDCl3
NAME XB20160222
EXPNO 6
PROCNO 1
Date_ 20160222
Time 9.38
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 143.7
DW 48.400 usec
DE 6.00 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
Cl
NO2
MeO
2b
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 ppm
0.97
1.99
0.15
0.08
2.00
0.15
3.00
0.23



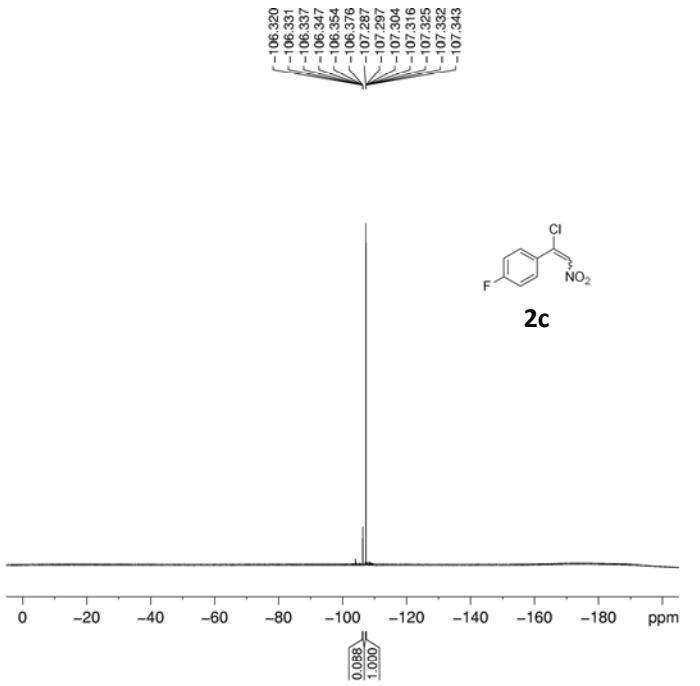

**2b**

```
GMC-11-50-2
C13CPD CDCl3

NAME          XB20160318
EXPNO                 20
PROCNO                 1
Date_           20160319
Time                2.07
INSTRUM            spect
PROBHD   5 mm PATXO 19F
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           30030.029 Hz
FIDRES        0.458222 Hz
AQ            1.0912410 sec
RG                  114
DW               16.650 usec
DE                6.00 usec
TE               296.4 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.50 usec
PL1                -0.50 dB
SFO1        125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.00 dB
PL12               16.05 dB
PL13               16.50 dB
SFO2        500.1320005 MHz
SI                 32768
SF          125.7577890 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
```




**2c**

```
GMC-11-96
PROTON CDCl3

NAME          XB20160322
EXPNO                 26
PROCNO                 1
Date_           20160322
Time               15.39
INSTRUM            spect
PROBHD   5 mm PATXO 19F
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1720407 sec
RG                203.2
DW               48.400 usec
DE                6.00 usec
TE               294.9 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.14 usec
PL1                1.00 dB
SFO1        500.1330885 MHz
SI                 32768
SF          500.1300129 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



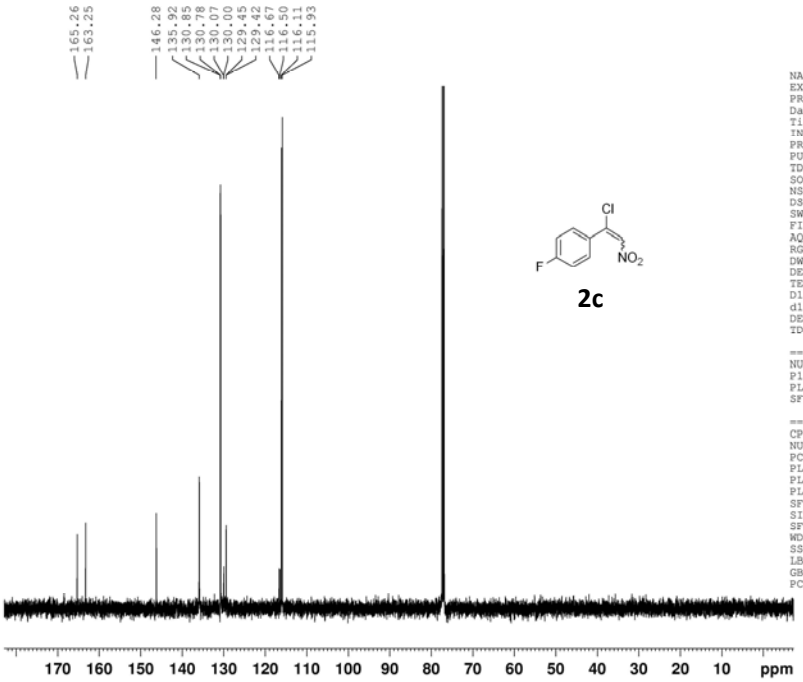

**2c**

GMC-11-96
19Fdeft CDCl3

| | |
|---|---|
| NAME | XB20160322 |
| EXPNO | 27 |
| PROCNO | 1 |
| Date_ | 20160322 |
| Time | 15.40 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg |
| TD | 131072 |
| SOLVENT | CDCl3 |
| NS | 16 |
| DS | 4 |
| SWH | 100000.000 Hz |
| FIDRES | 0.762939 Hz |
| AQ | 0.6554150 sec |
| RG | 362 |
| DW | 5.000 usec |
| DE | 6.00 usec |
| TE | 294.7 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |

======== CHANNEL f1 =======

| | |
|---|---|
| NUC1 | 19F |
| P1 | 19.30 usec |
| PL1 | 4.00 dB |
| SFO1 | 470.5453180 MHz |
| SI | 65536 |
| SF | 470.5923770 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |




**2c**

GMC-11-96
C13CPD CDCl3

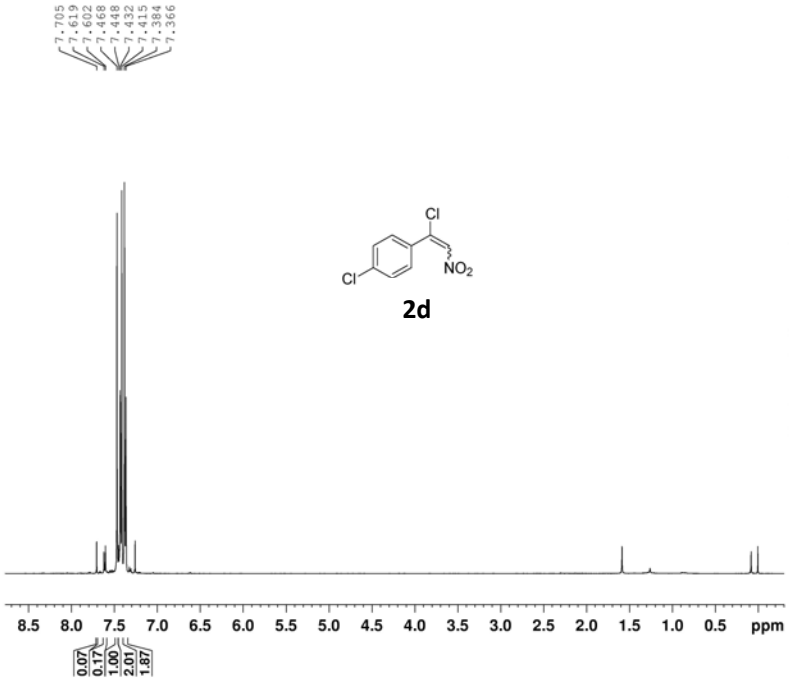
| | |
|---|---|
| NAME | XB20160322 |
| EXPNO | 38 |
| PROCNO | 1 |
| Date_ | 20160322 |
| Time | 20.24 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 161.3 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 295.4 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |

======== CHANNEL f1 =======

| | |
|---|---|
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |

======== CHANNEL f2 =======

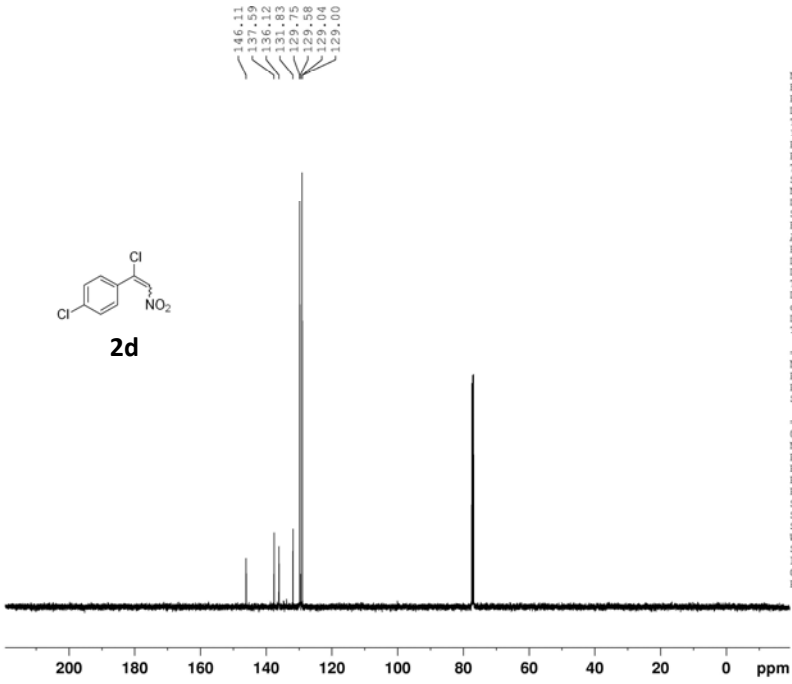
| | |
|---|---|
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577763 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |




**2d**

GMC-11-47-2
PROTON CDCl3

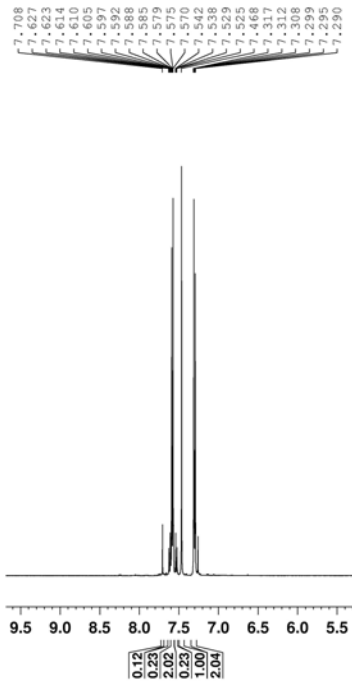
| | |
|---|---|
| NAME | XB20160223 |
| EXPNO | 2 |
| PROCNO | 1 |
| Date_ | 20160223 |
| Time | 12.24 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 8 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 161.3 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 294.4 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300126 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |

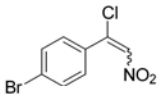



**2d**

GMC-11-47-2
C13CPD CDCl3

| | |
|---|---|
| NAME | XB20160325 |
| EXPNO | 8 |
| PROCNO | 1 |
| Date_ | 20160325 |
| Time | 10.57 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 101.6 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 295.3 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577791 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.70 Hz |
| GB | 0 |
| PC | 1.40 |

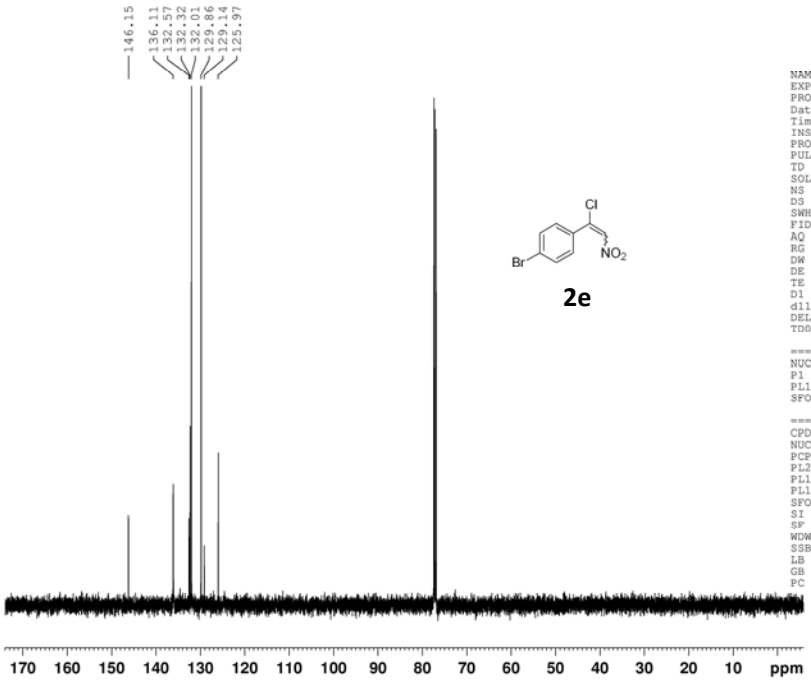



**2e**

GMC-11-54
PROTON CDC13

| | |
|---|---|
| NAME | XB20160222 |
| EXPNO | 9 |
| PROCNO | 1 |
| Date_ | 20160222 |
| Time | 9.56 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 16 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 143.7 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 294.3 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300129 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |



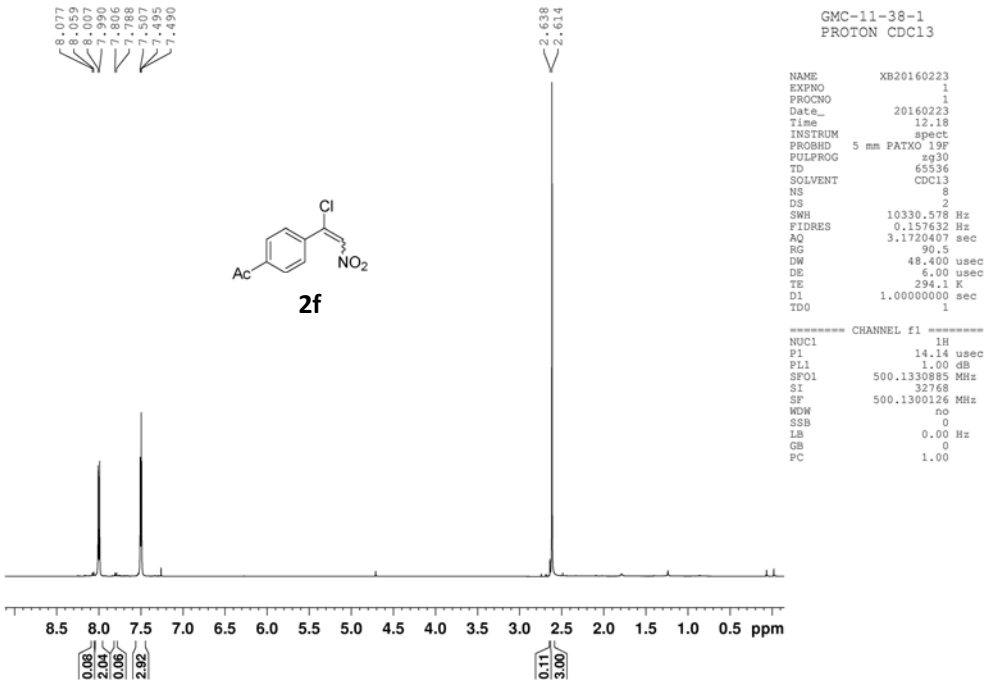




**2e**

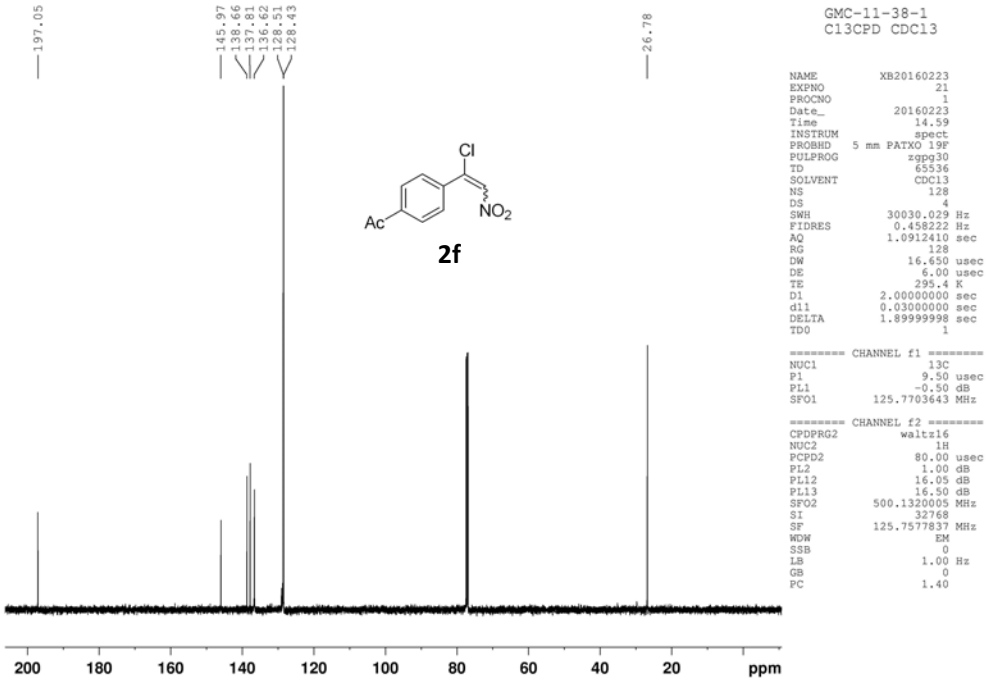
GMC-11-54
C13CPD CDC13

| | |
|---|---|
| NAME | XB20160325 |
| EXPNO | 9 |
| PROCNO | 1 |
| Date_ | 20160325 |
| Time | 11.04 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 94 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 181 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.3 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577791 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.70 Hz |
| GB | 0 |
| PC | 1.40 |



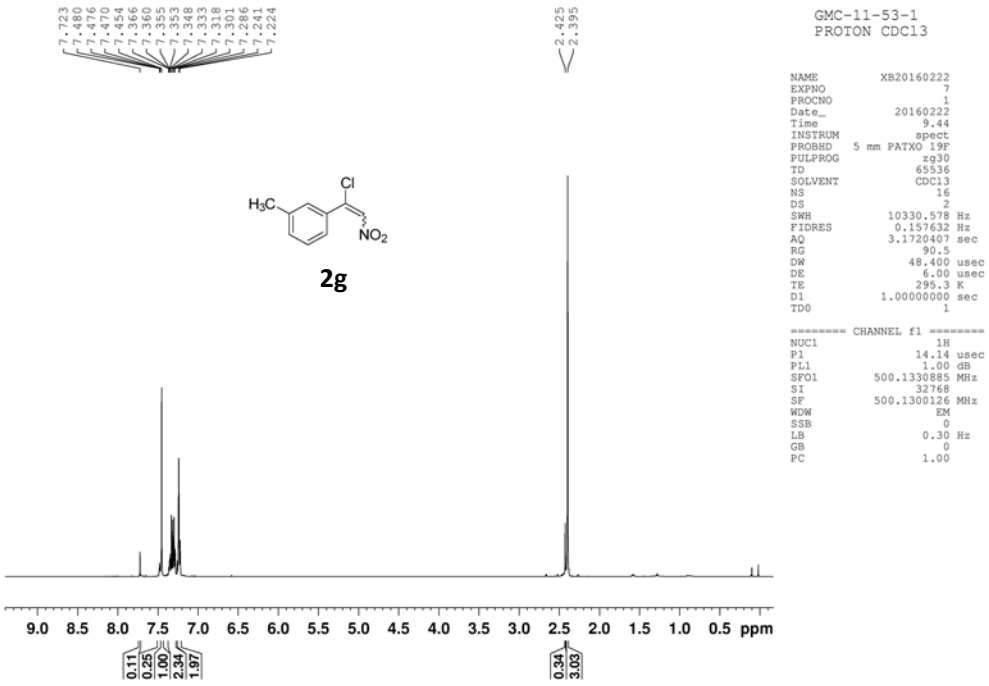

GMC-11-38-1
PROTON CDCl3

| | |
|---|---|
| NAME | XB20160223 |
| EXPNO | 1 |
| PROCNO | 1 |
| Date_ | 20160223 |
| Time | 12.18 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 8 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 90.5 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 294.1 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300126 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |



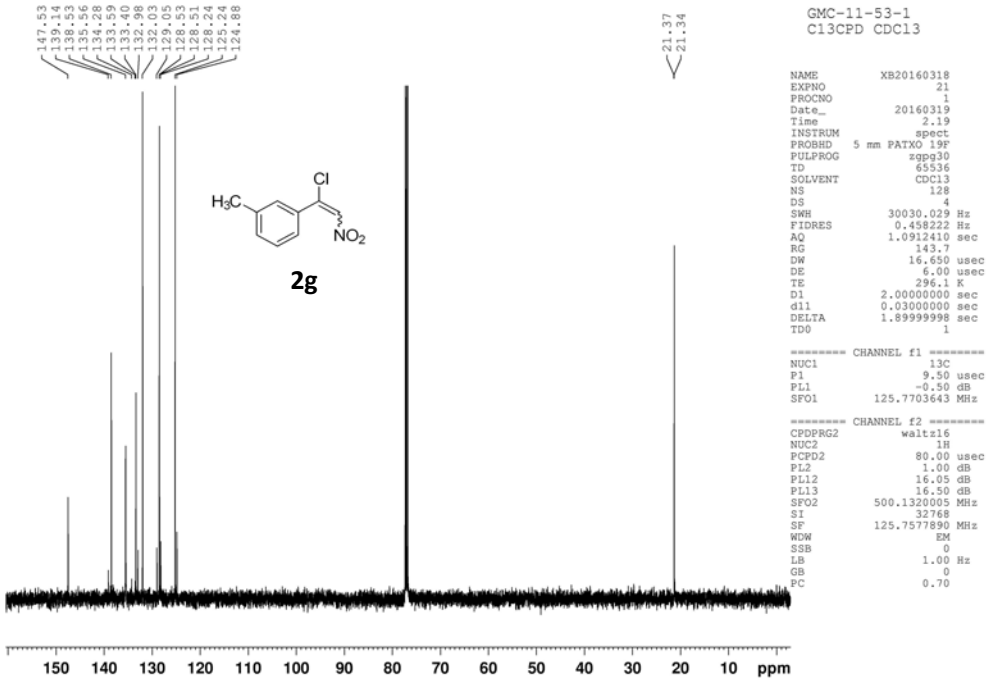

GMC-11-38-1
C13CPD CDCl3

| | |
|---|---|
| NAME | XB20160223 |
| EXPNO | 21 |
| PROCNO | 1 |
| Date_ | 20160223 |
| Time | 14.59 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 128 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 295.4 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577837 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |



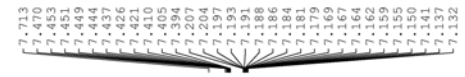

GMC-11-53-1
PROTON CDC13

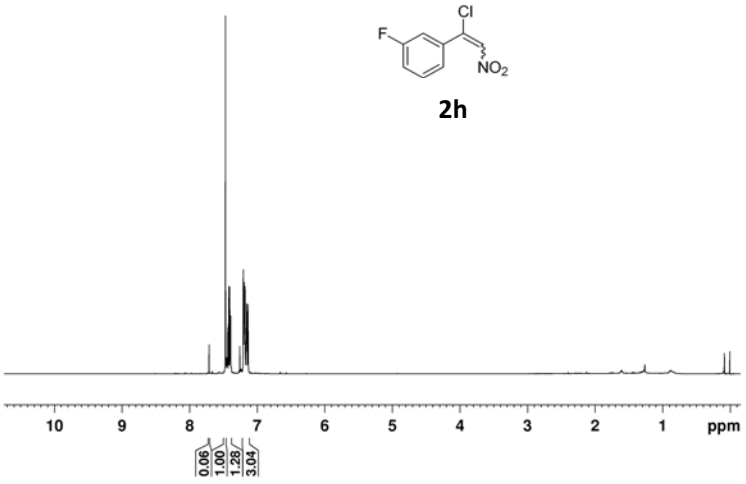
| | |
|---|---|
| NAME | XB20160222 |
| EXPNO | 7 |
| PROCNO | 1 |
| Date_ | 20160222 |
| Time | 9.44 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 16 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 90.5 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 295.3 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300126 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |




GMC-11-53-1
C13CPD CDC13

| | |
|---|---|
| NAME | XB20160318 |
| EXPNO | 21 |
| PROCNO | 1 |
| Date_ | 20160319 |
| Time | 2.19 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 143.7 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.1 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577890 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 0.70 |

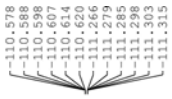


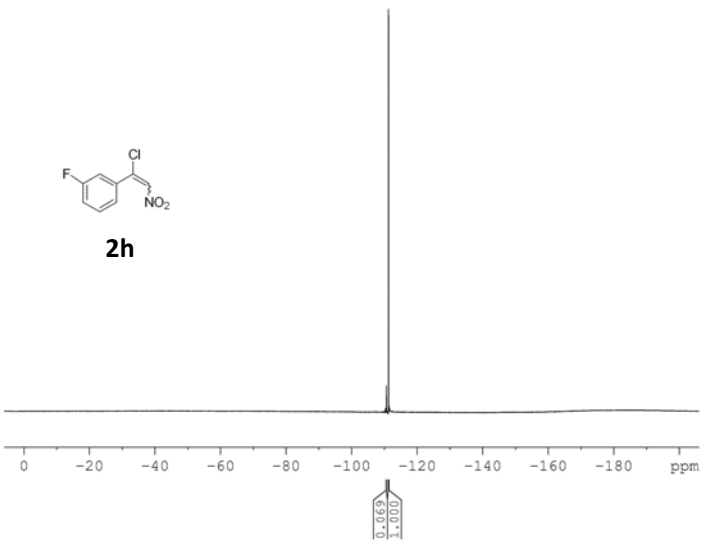




**2h**

GMC-11-53-2
PROTON CDCl3

| | |
|---|---|
| NAME | XB20160222 |
| EXPNO | 8 |
| PROCNO | 1 |
| Date_ | 20160222 |
| Time | 9.50 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 16 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 128 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 295.5 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300129 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |

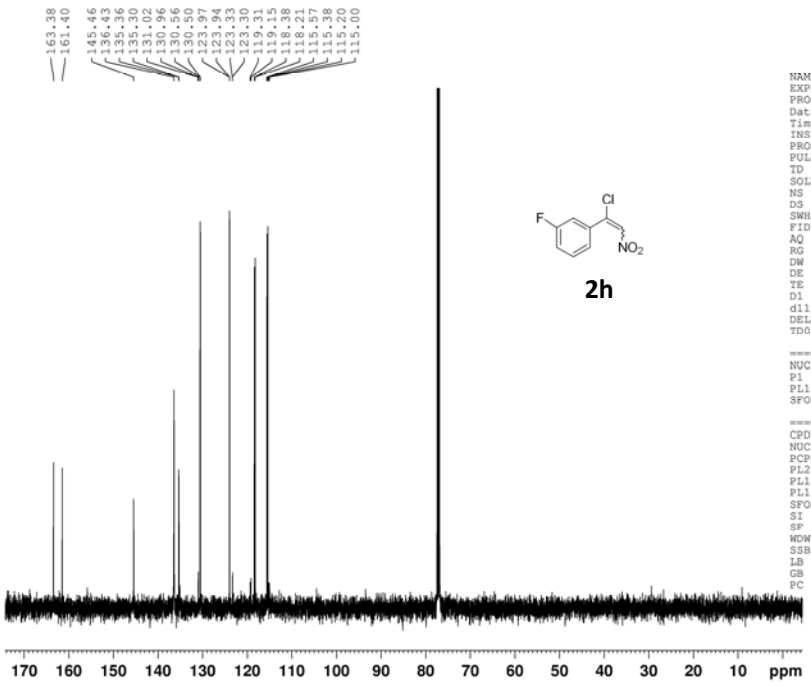



**2h**

GMC-11-53-2
19Fdeft CDCl3

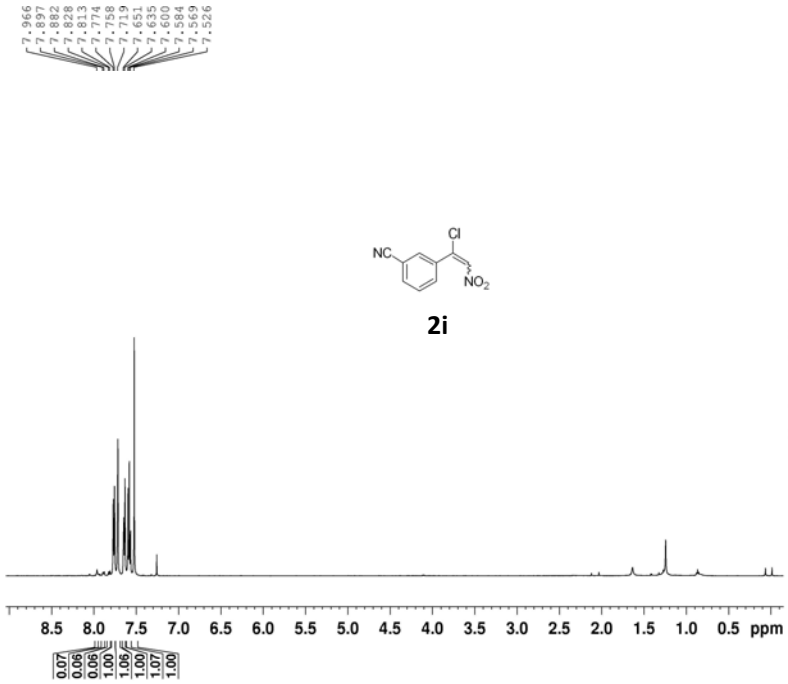
| | |
|---|---|
| NAME | XB20160322 |
| EXPNO | 36 |
| PROCNO | 1 |
| Date_ | 20160322 |
| Time | 20.05 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg |
| TD | 131072 |
| SOLVENT | CDCl3 |
| NS | 16 |
| DS | 4 |
| SWH | 100000.000 Hz |
| FIDRES | 0.762939 Hz |
| AQ | 0.6554150 sec |
| RG | 456.1 |
| DW | 5.000 usec |
| DE | 6.00 usec |
| TE | 294.1 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 19F |
| P1 | 19.30 usec |
| PL1 | 4.00 dB |
| SFO1 | 470.5453180 MHz |
| SI | 65536 |
| SF | 470.5923770 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.30 Hz |
| GB | 0 |
| PC | 1.00 |




**2h**

GMC-11-53-2
C13CPD CDCl3

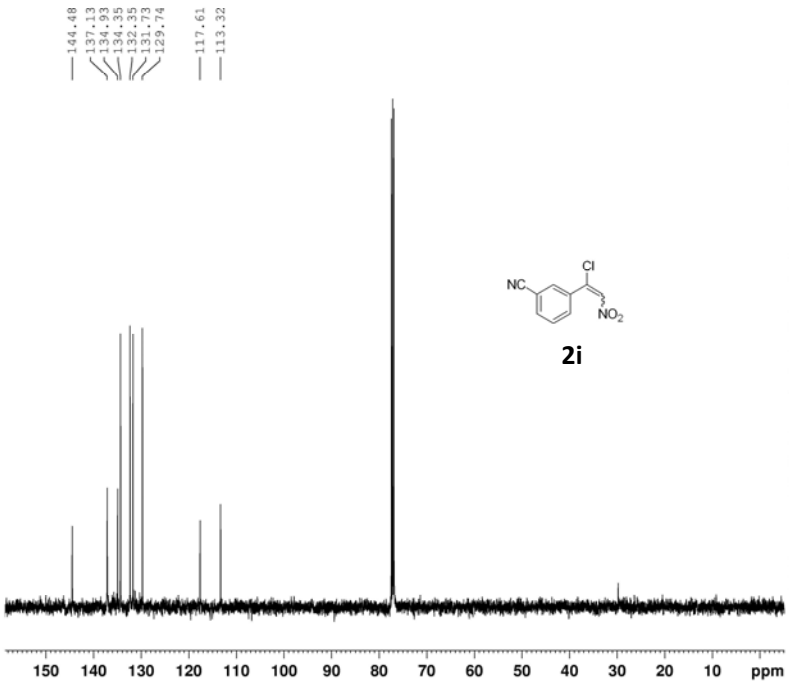
| | |
|---|---|
| NAME | XB20160322 |
| EXPNO | 37 |
| PROCNO | 1 |
| Date_ | 20160322 |
| Time | 20.14 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 161.3 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.0 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577772 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 0.50 |




**2i**

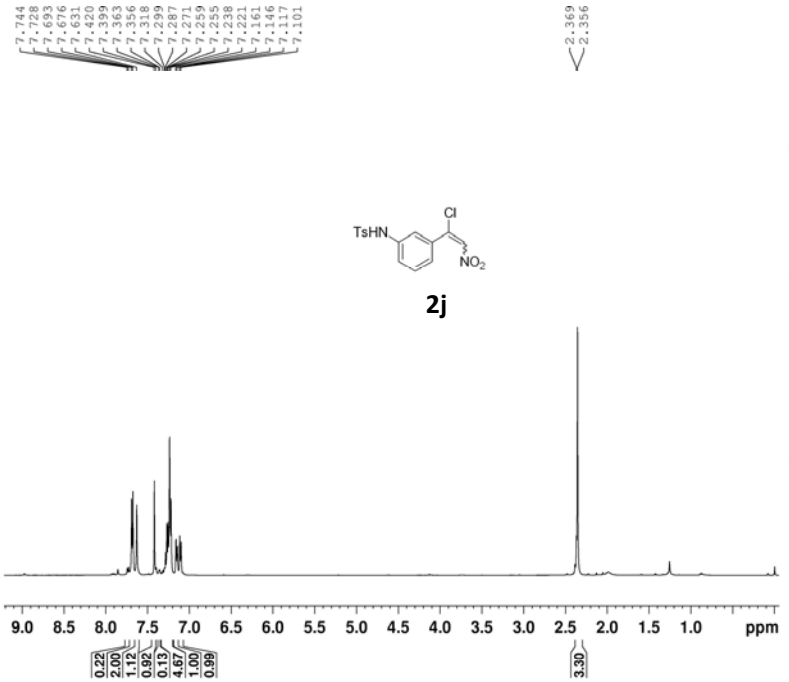
GMC-12-23
PROTON CDCl3

| | |
|---|---|
| NAME | XB20160516 |
| EXPNO | 20 |
| PROCNO | 1 |
| Date_ | 20160516 |
| Time | 15.11 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 4 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 181 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 295.1 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300129 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |



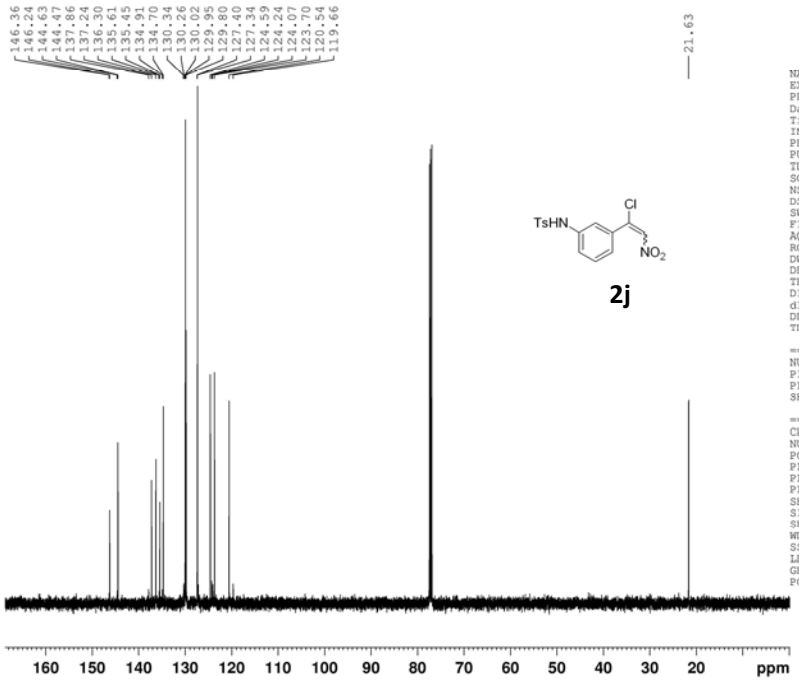

**2i**

```
GMC-12-23
C13CPD CDCl3

NAME            XB20160516
EXPNO                101
PROCNO                 1
Date_           20160516
Time                15.15
INSTRUM             spect
PROBHD   5 mm PATXO 19F
PULPROG             zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     69
DS                      4
SWH             30030.029 Hz
FIDRES              0.458222 Hz
AQ                1.0912410 sec
RG                    114
DW                 16.650 usec
DE                  6.00 usec
TE                 295.9 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.50 usec
PL1               -0.50 dB
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                1.00 dB
PL12              16.05 dB
PL13              16.50 dB
SFO2          500.1320005 MHz
SI                 32768
SF            125.7577809 MHz
WDW                   EM
SSB                    0
LB                 2.00 Hz
GB                     0
PC                 1.40
```




**2j**

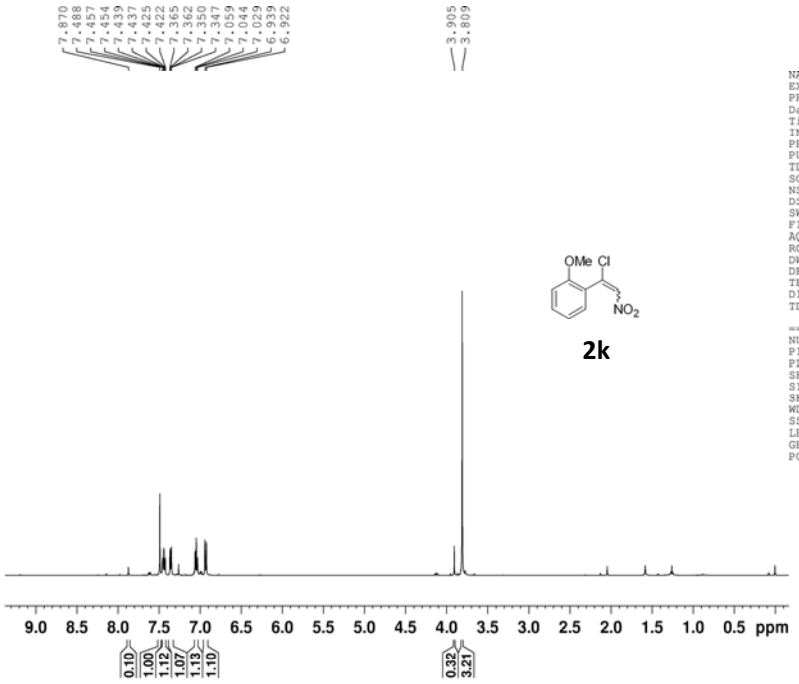
```
GMC-12-52
PROTON CDCl3

NAME            XB20160531
EXPNO                 15
PROCNO                 1
Date_           20160531
Time                12.51
INSTRUM             spect
PROBHD   5 mm PATXO 19F
PULPROG               zg30
TD                  65536
SOLVENT             CDCl3
NS                     8
DS                     2
SWH             10330.578 Hz
FIDRES              0.157632 Hz
AQ                3.1720407 sec
RG                   101.6
DW                 48.400 usec
DE                  6.00 usec
TE                 295.3 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.14 usec
PL1                1.00 dB
SFO1          500.1330885 MHz
SI                 32768
SF            500.1300129 MHz
WDW                   EM
SSB                    0
LB                 0.50 Hz
GB                     0
PC                 1.00
```




**2j**

GMC-12-52
C13CPD CDC13

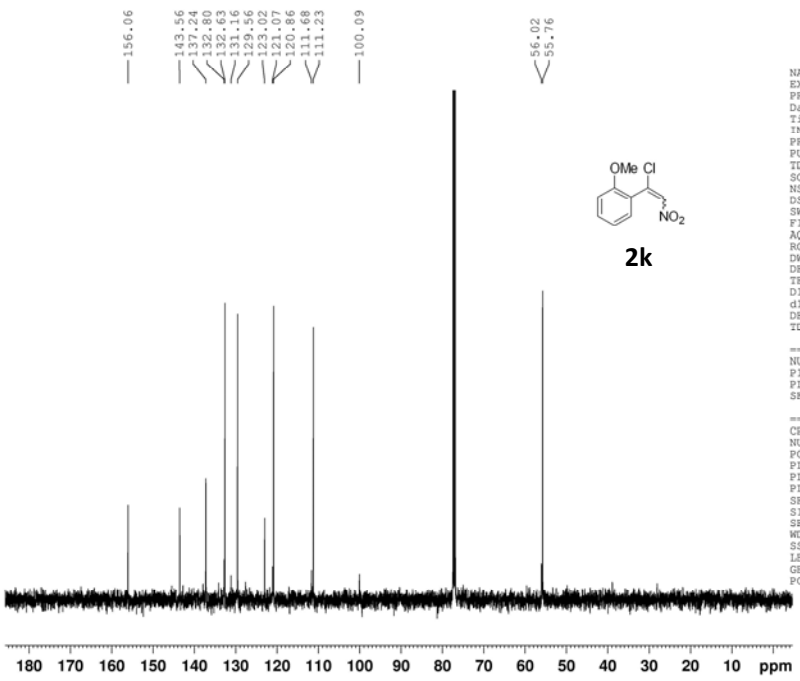
| | |
|---|---|
| NAME | XB20160531 |
| EXPNO | 20 |
| PROCNO | 1 |
| Date_ | 20160531 |
| Time | 14.42 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 362 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.4 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577809 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.50 Hz |
| GB | 0 |
| PC | 0.70 |




**2k**

GMC-11-149
PROTON CDC13

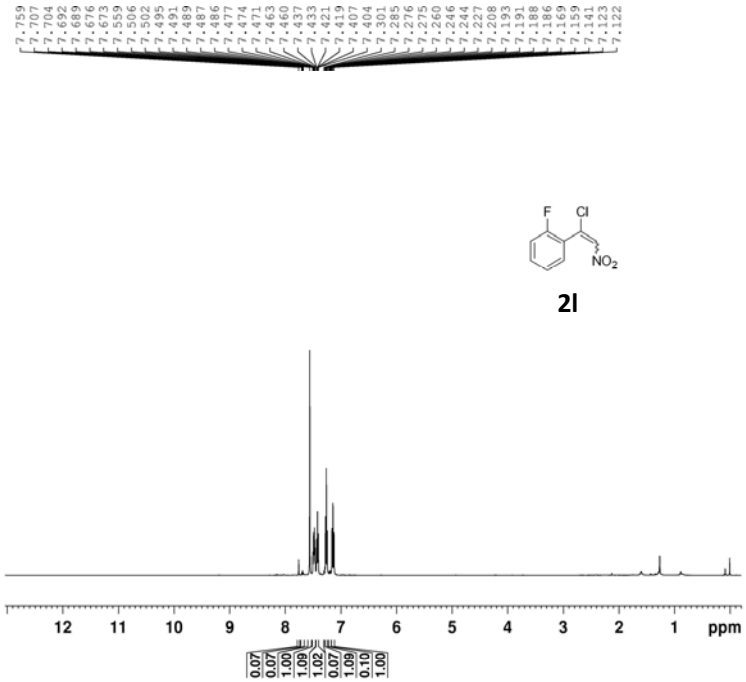
| | |
|---|---|
| NAME | XB20160425 |
| EXPNO | 13 |
| PROCNO | 1 |
| Date_ | 20160425 |
| Time | 10.27 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 16 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 181 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 294.5 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300129 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |




**2k**

GMC-11-149
C13CPD CDC13

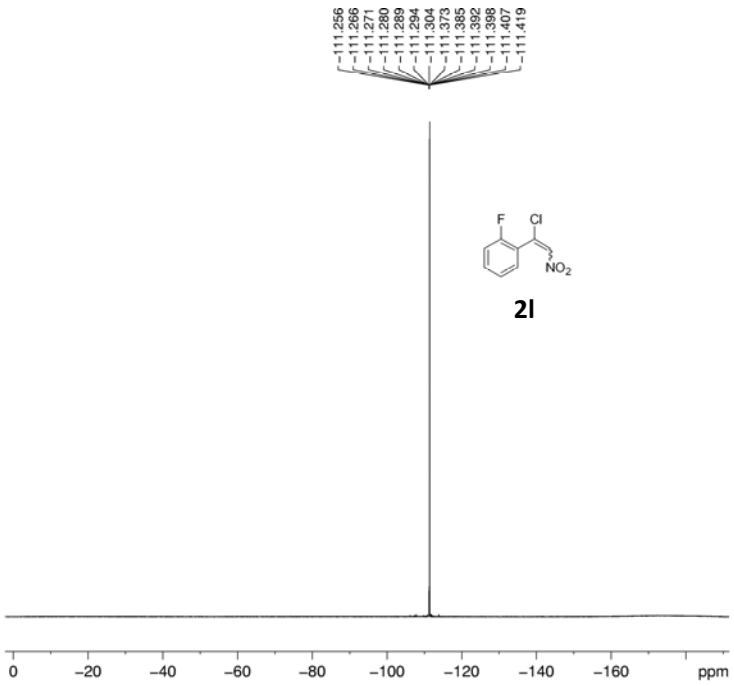
| | |
|---|---|
| NAME | XB20160425 |
| EXPNO | 16 |
| PROCNO | 1 |
| Date_ | 20160425 |
| Time | 18.01 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 161.3 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.2 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577763 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 2.00 Hz |
| GB | 0 |
| PC | 1.40 |




**2l**

GMC-12-34
PROTON CDC13

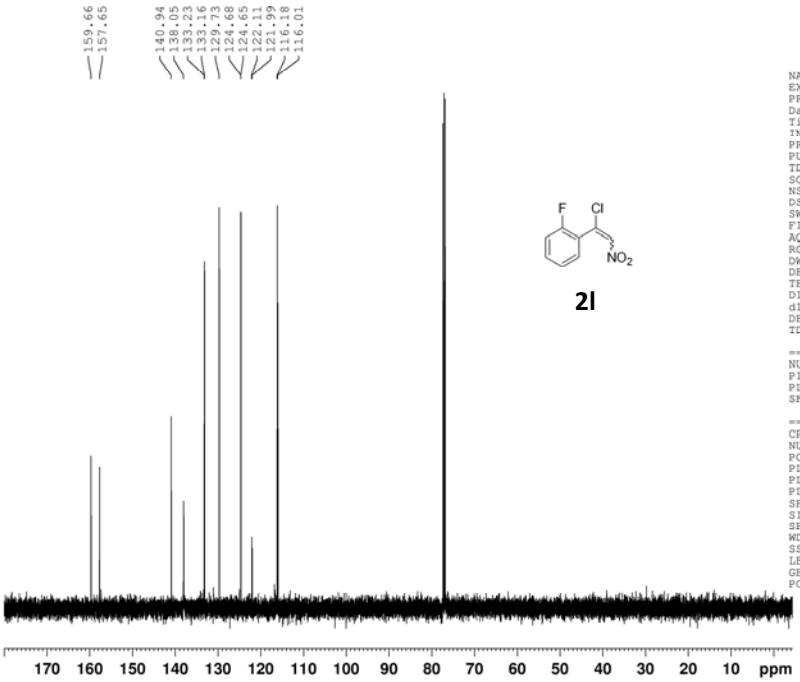
| | |
|---|---|
| NAME | XB20160519 |
| EXPNO | 17 |
| PROCNO | 1 |
| Date_ | 20160519 |
| Time | 17.21 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 16 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 161.3 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 295.5 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300129 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |




**2l**

GMC-12-34
19Fdeft CDCl3

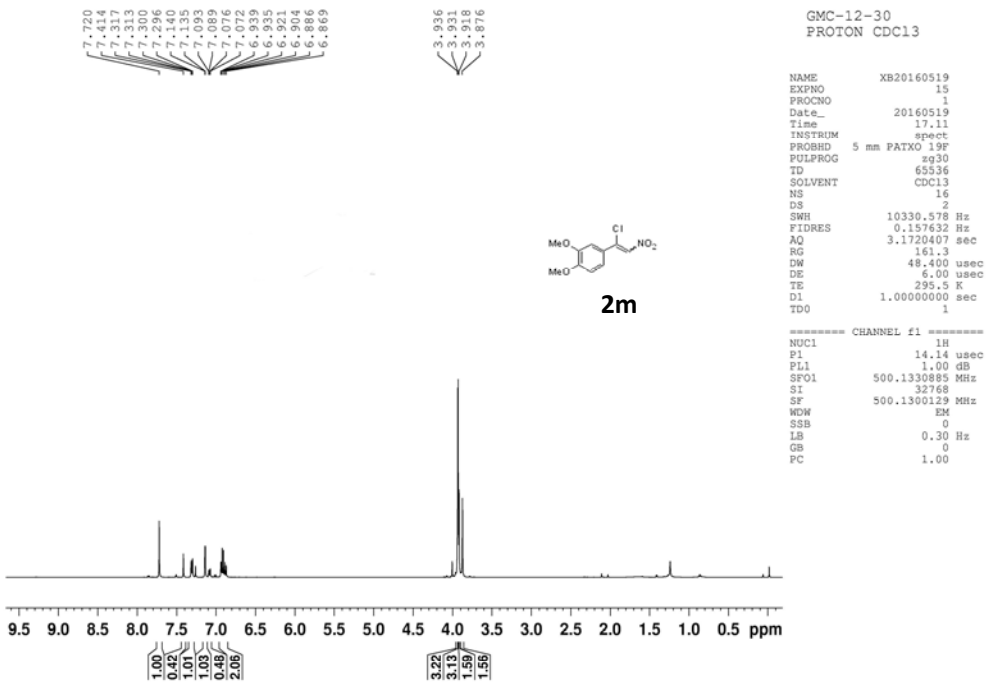
| | |
|---|---|
| NAME | XB20160519 |
| EXPNO | 18 |
| PROCNO | 1 |
| Date_ | 20160519 |
| Time | 17.22 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg |
| TD | 131072 |
| SOLVENT | CDCl3 |
| NS | 16 |
| DS | 4 |
| SWH | 100000.000 Hz |
| FIDRES | 0.762939 Hz |
| AQ | 0.6554150 sec |
| RG | 256 |
| DW | 5.000 usec |
| DE | 6.00 usec |
| TE | 295.6 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 19F |
| P1 | 19.30 usec |
| PL1 | 4.00 dB |
| SFO1 | 470.5453180 MHz |
| SI | 65536 |
| SF | 470.5923770 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |

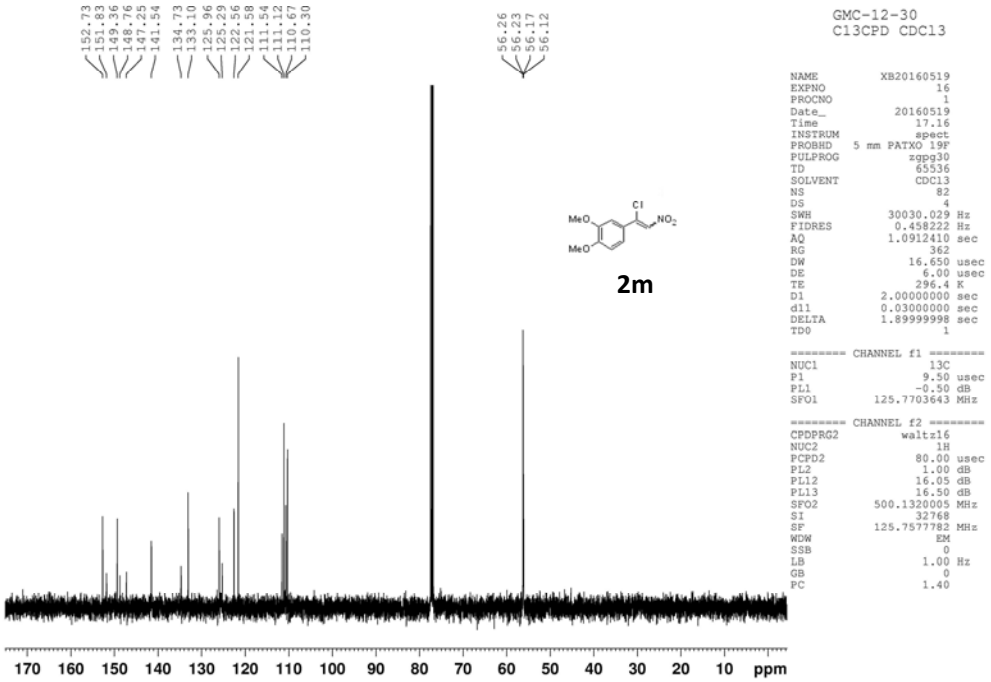


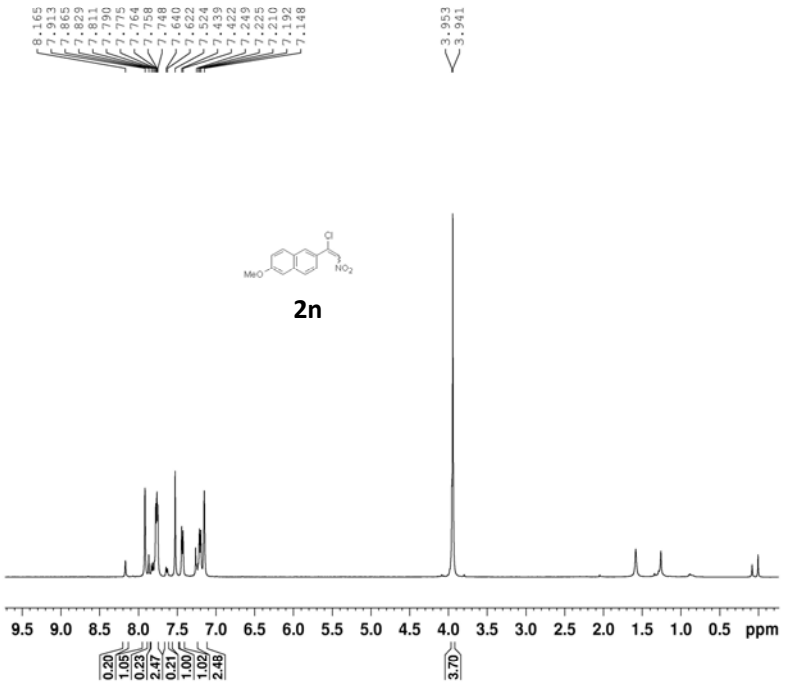

**2l**

GMC-12-34
C13CPD CDCl3

| | |
|---|---|
| NAME | XB20160519 |
| EXPNO | 19 |
| PROCNO | 1 |
| Date_ | 20160519 |
| Time | 17.27 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 362 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.2 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577782 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 0.70 |

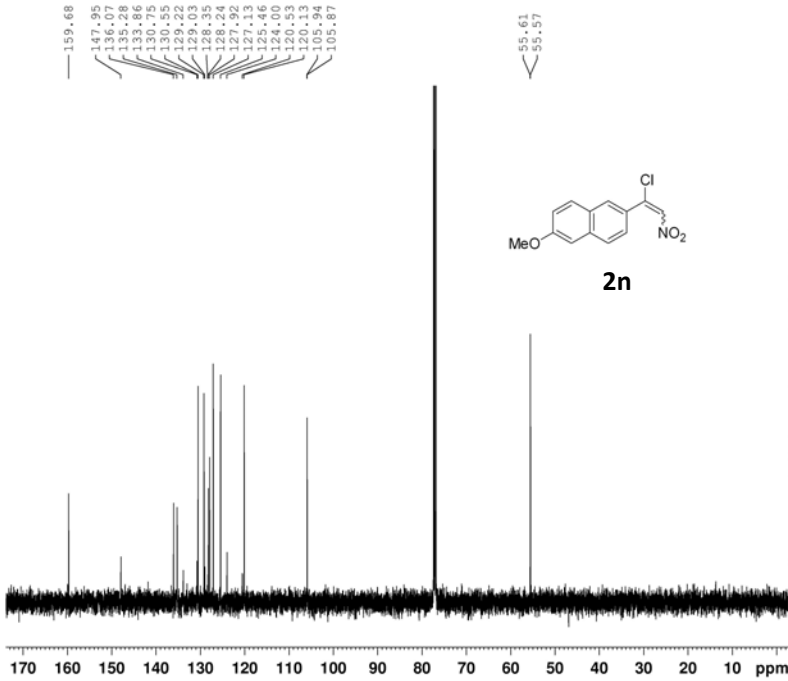

7.720
7.414
7.317
7.313
7.300
7.296
7.140
7.135
7.093
7.089
7.076
7.072
6.939
6.935
6.921
6.904
6.886
6.869
3.936
3.931
3.918
3.876
GMC-12-30
PROTON CDC13
NAME XB20160519
EXPNO 15
PROCNO 1
Date_ 20160519
Time 17.11
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 161.3
DW 48.400 usec
DE 6.00 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
MeO
Cl
NO2
2m
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 ppm
1.00
0.42
1.01
1.03
0.48
2.05
3.22
3.13
1.59
1.56


152.73
151.83
149.36
148.76
147.25
141.54
134.73
133.10
125.96
125.29
122.56
121.58
111.54
111.12
110.67
110.30
56.26
56.23
56.17
56.12
GMC-12-30
C13CPD CDC13
NAME XB20160519
EXPNO 16
PROCNO 1
Date_ 20160519
Time 17.16
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 82
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 362
DW 16.650 usec
DE 6.00 usec
TE 296.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577782 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
MeO
MeO
Cl
NO2
2m
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



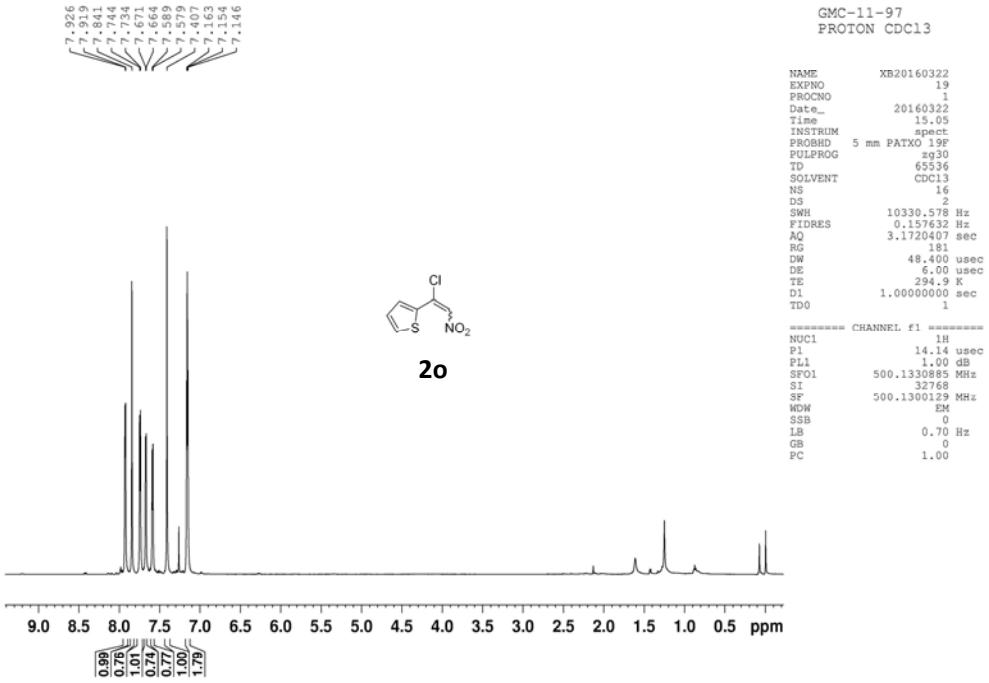

**2n**

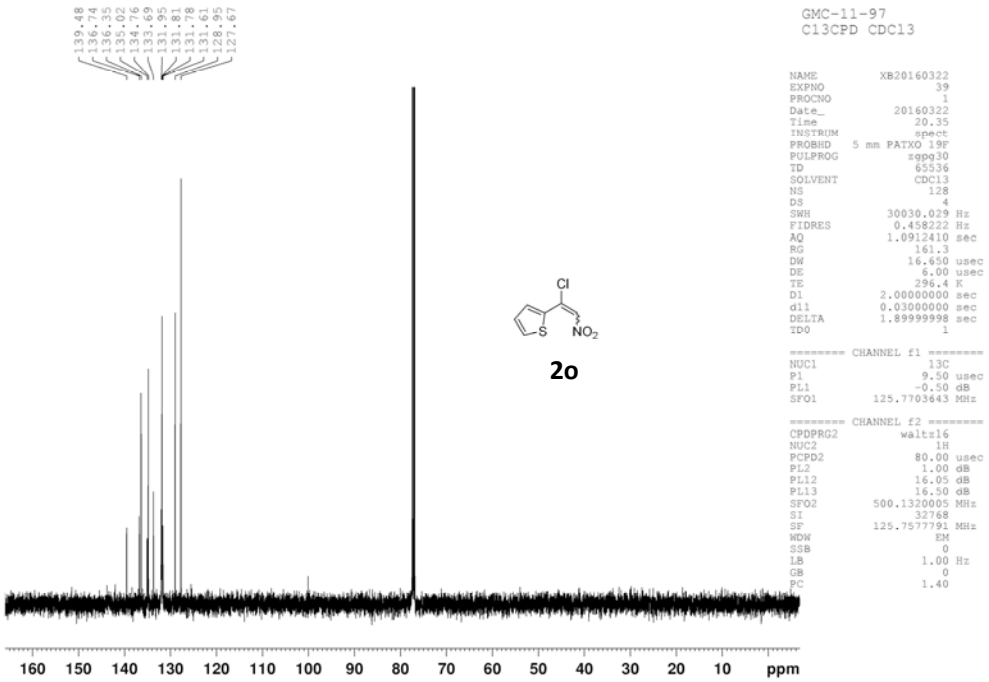
```
GMC-11-158
PROTON CDC13

NAME          XB20160505
EXPNO                  5
PROCNO                 1
Date_           20160505
Time               13.03
INSTRUM            spect
PROBHD   5 mm PATXO 19F
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                     8
DS                     2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                 203.2
DW                48.400 usec
DE                 6.00 usec
TE                295.7 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.14 usec
PL1                1.00 dB
SFO1         500.1330885 MHz
SI                 32768
SF           500.1300129 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

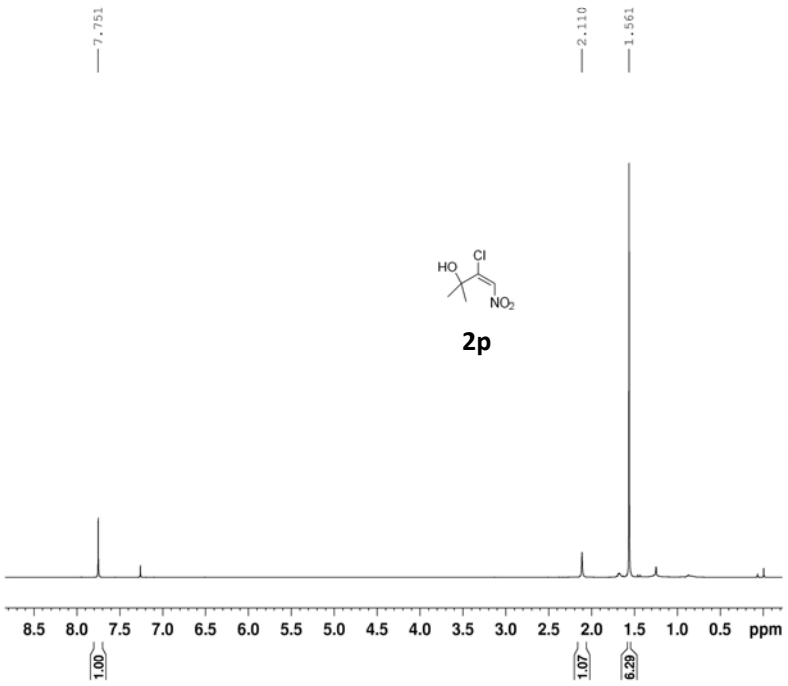



**2n**

```
GMC-11-158
C13CPD CDC13

NAME          XB20160505
EXPNO                 15
PROCNO                 1
Date_           20160505
Time               15.01
INSTRUM            spect
PROBHD   5 mm PATXO 19F
PULPROG           zgpg30
TD                 65536
SOLVENT            CDC13
NS                   128
DS                     4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ             1.0912410 sec
RG                 161.3
DW                16.650 usec
DE                 6.00 usec
TE                296.8 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 9.50 usec
PL1               -0.50 dB
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                1.00 dB
PL12              16.05 dB
PL13              16.50 dB
SFO2         500.1320005 MHz
SI                 32768
SF           125.7577754 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.00
```

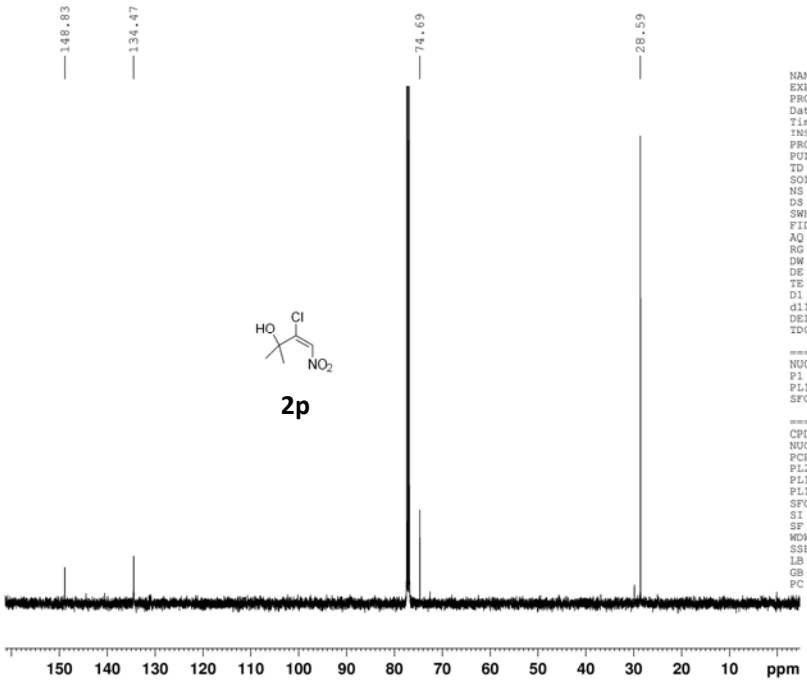

7.926
7.919
7.841
7.744
7.734
7.671
7.664
7.589
7.579
7.407
7.163
7.154
7.146
GMC-11-97
PROTON CDC13
NAME XB20160322
EXPNO 19
PROCNO 1
Date_ 20160322
Time 15.05
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 181
DW 48.400 usec
DE 6.00 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 1.00
Cl
S
NO2
2o
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 ppm
0.99
0.76
1.01
0.74
0.77
1.00
1.79


139.48
136.74
136.35
135.02
134.76
133.89
131.85
131.81
131.78
131.61
128.85
127.67
GMC-11-97
C13CPD CDC13
NAME XB20160322
EXPNO 39
PROCNO 1
Date_ 20160322
Time 20.35
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 128
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 161.3
DW 16.650 usec
DE 6.00 usec
TE 296.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577791 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cl
S
NO2
2o
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



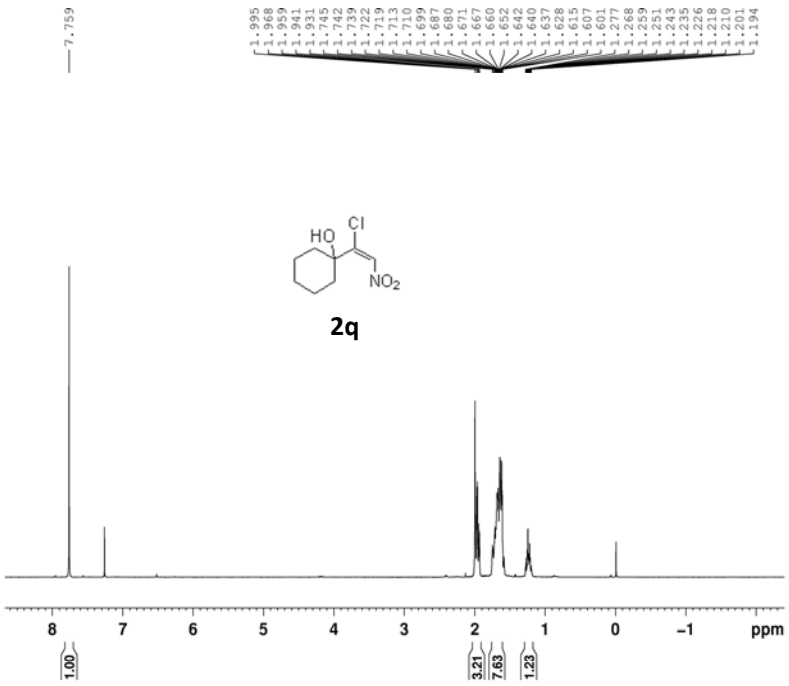

GMC-11-77-2-2
PROTON CDCl3

| | |
|---|---|
| NAME | XB20160307 |
| EXPNO | 13 |
| PROCNO | 1 |
| Date_ | 20160307 |
| Time | 14.57 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 16 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 228.1 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 294.8 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300129 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |



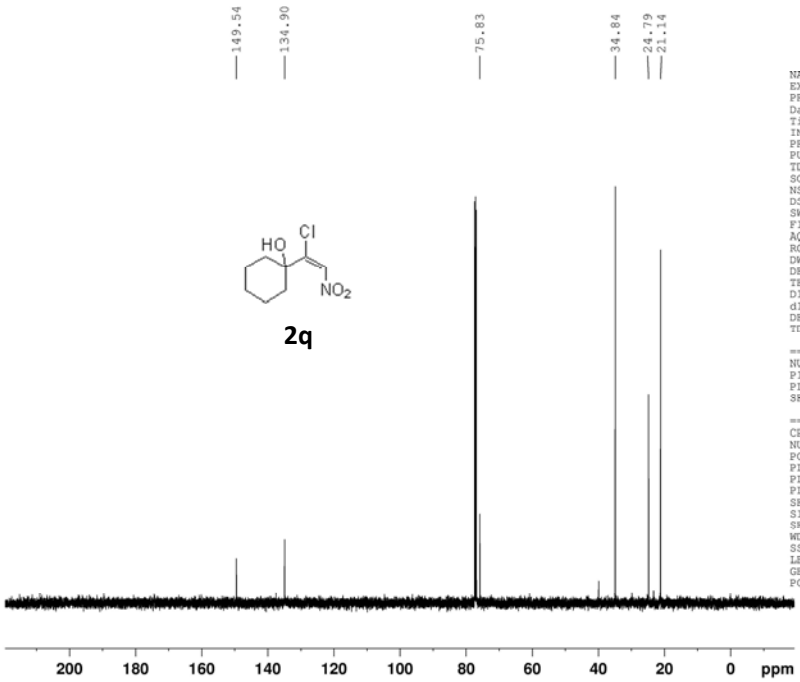

GMC-11-77-2-2
C13CPD CDCl3

| | |
|---|---|
| NAME | XB20160325 |
| EXPNO | 21 |
| PROCNO | 1 |
| Date_ | 20160326 |
| Time | 9.00 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDCl3 |
| NS | 512 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 128 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.0 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577727 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |



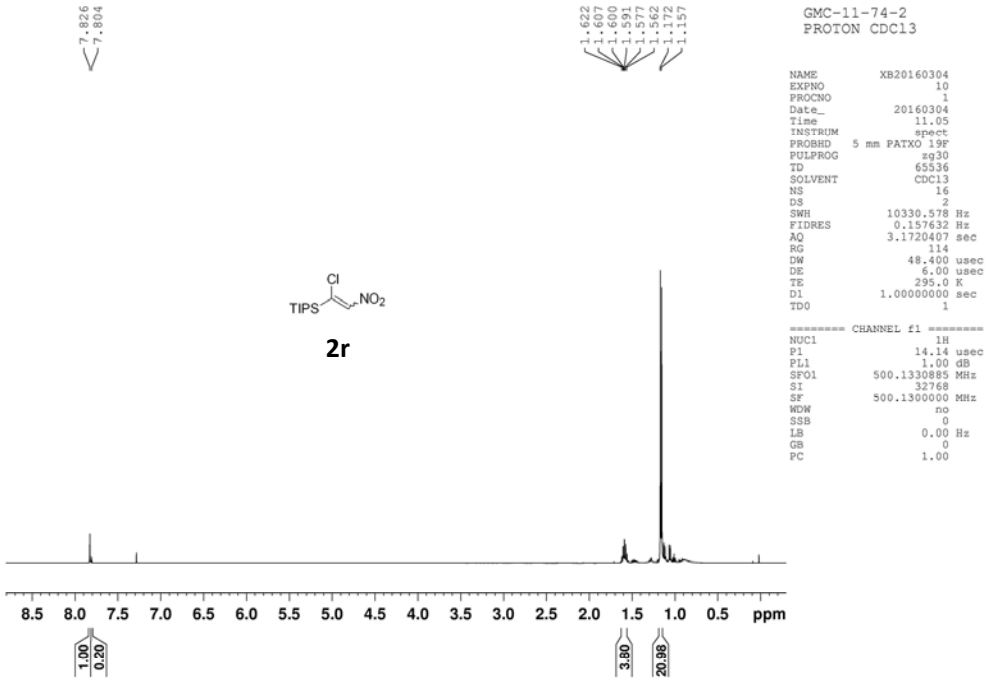

**2q**

```
GMC-11-92
PROTON CDCl3

NAME          XB20160510
EXPNO                 12
PROCNO                 1
Date_           20160510
Time               10.49
INSTRUM            spect
PROBHD   5 mm PATXO 19F
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1720407 sec
RG                   181
DW               48.400 usec
DE                6.00 usec
TE               295.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.14 usec
PL1                1.00 dB
SFO1         500.1330885 MHz
SI                 32768
SF           500.1300129 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                1.00
```

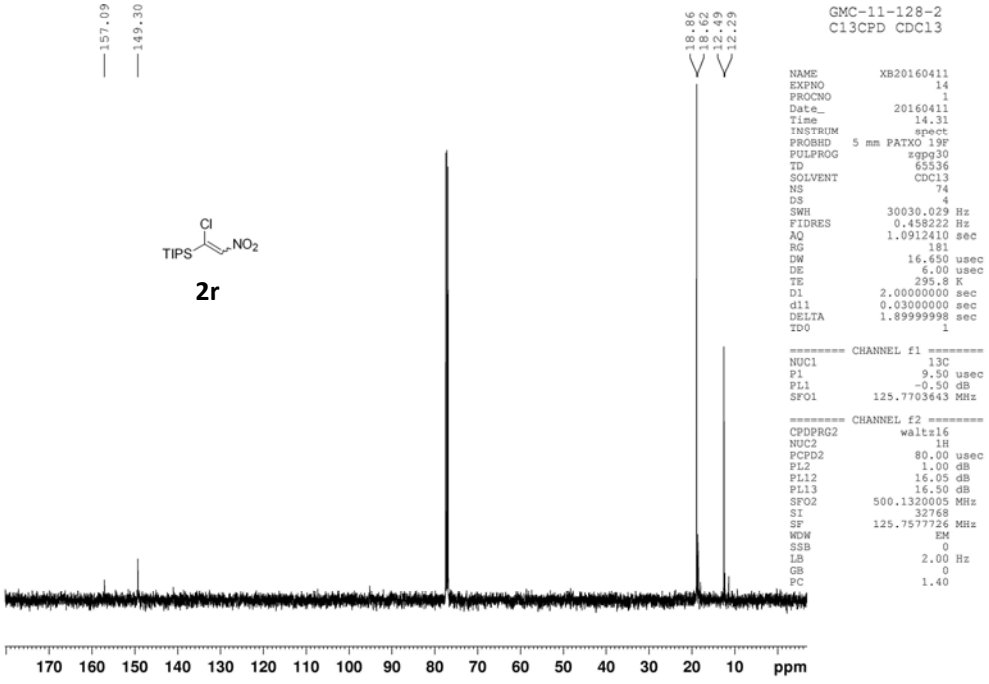



**2q**

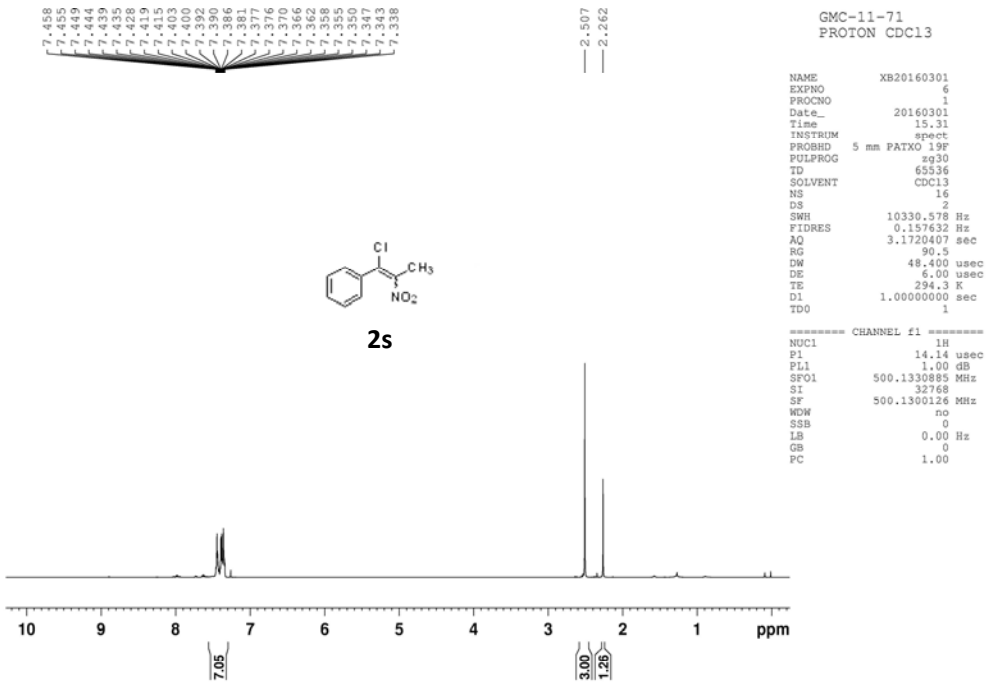
```
GMC-11-92
C13CPD CDCl3

NAME          XB20160322
EXPNO                 40
PROCNO                 1
Date_           20160322
Time               20.45
INSTRUM            spect
PROBHD   5 mm PATXO 19F
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     4
SWH           30030.029 Hz
FIDRES        0.458222 Hz
AQ            1.0912410 sec
RG                 161.3
DW               16.650 usec
DE                6.00 usec
TE               295.8 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                9.50 usec
PL1               -0.50 dB
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                1.00 dB
PL12              16.05 dB
PL13              16.50 dB
SFO2         500.1320005 MHz
SI                 32768
SF           125.7577745 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

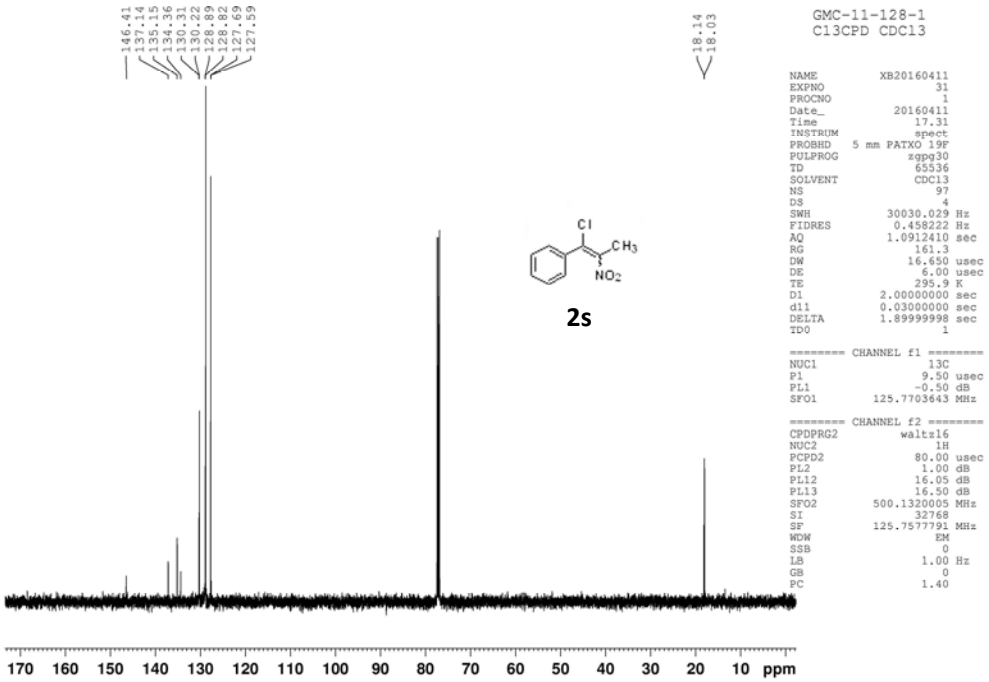


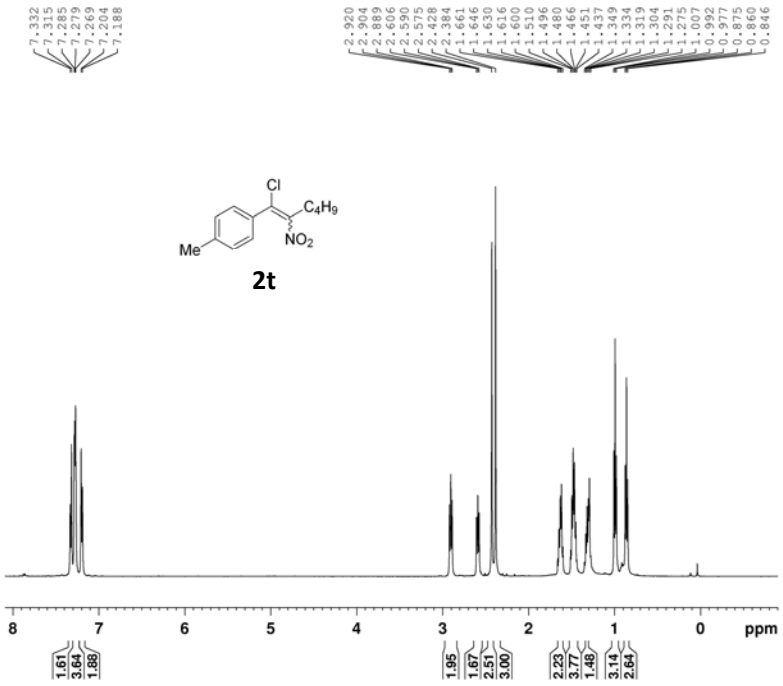

```
GMC-11-74-2
PROTON CDCl3

NAME          XB20160304
EXPNO             10
PROCNO             1
Date_          20160304
Time              11.05
INSTRUM           spect
PROBHD   5 mm PATXO 19F
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                16
DS                 2
SWH          10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1720407 sec
RG                114
DW             48.400 usec
DE              6.00 usec
TE             295.0 K
D1          1.00000000 sec
TD0                1

======== CHANNEL f1 ========
NUC1               1H
P1             14.14 usec
PL1             1.00 dB
SFO1      500.1330885 MHz
SI              32768
SF        500.1300000 MHz
WDW               no
SSB                0
LB              0.00 Hz
GB                 0
PC              1.00
```



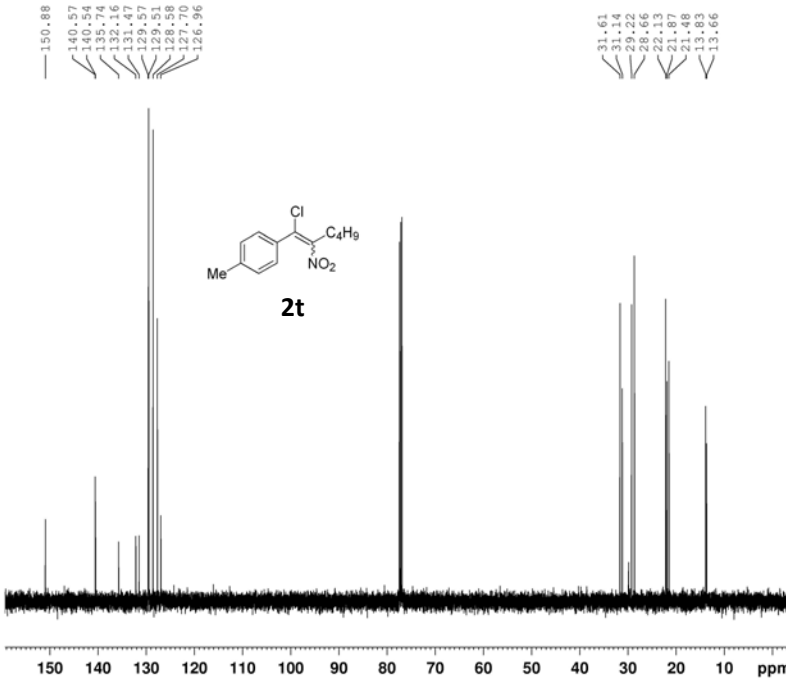

```
GMC-11-128-2
C13CPD CDCl3

NAME          XB20160411
EXPNO             14
PROCNO             1
Date_          20160411
Time              14.31
INSTRUM           spect
PROBHD   5 mm PATXO 19F
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                74
DS                 4
SWH          30030.029 Hz
FIDRES        0.458222 Hz
AQ            1.0912410 sec
RG                181
DW             16.650 usec
DE              6.00 usec
TE             295.8 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
TD0                1

======== CHANNEL f1 ========
NUC1              13C
P1              9.50 usec
PL1            -0.50 dB
SFO1      125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2               1H
PCPD2          80.00 usec
PL2             1.00 dB
PL12           16.05 dB
PL13           16.50 dB
SFO2      500.1320005 MHz
SI              32768
SF        125.7577726 MHz
WDW               EM
SSB                0
LB              2.00 Hz
GB                 0
PC              1.40
```


7.458
7.455
7.449
7.444
7.439
7.435
7.428
7.419
7.415
7.403
7.400
7.392
7.390
7.386
7.381
7.377
7.376
7.370
7.366
7.362
7.358
7.355
7.350
7.347
7.343
7.338
2.507
2.262
GMC-11-71
PROTON CDC13
NAME XB20160301
EXPNO 6
PROCNO 1
Date_ 20160301
Time 15.31
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 294.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300126 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Cl
CH3
NO2
2s
10 9 8 7 6 5 4 3 2 1 ppm
7.05
3.00
1.28

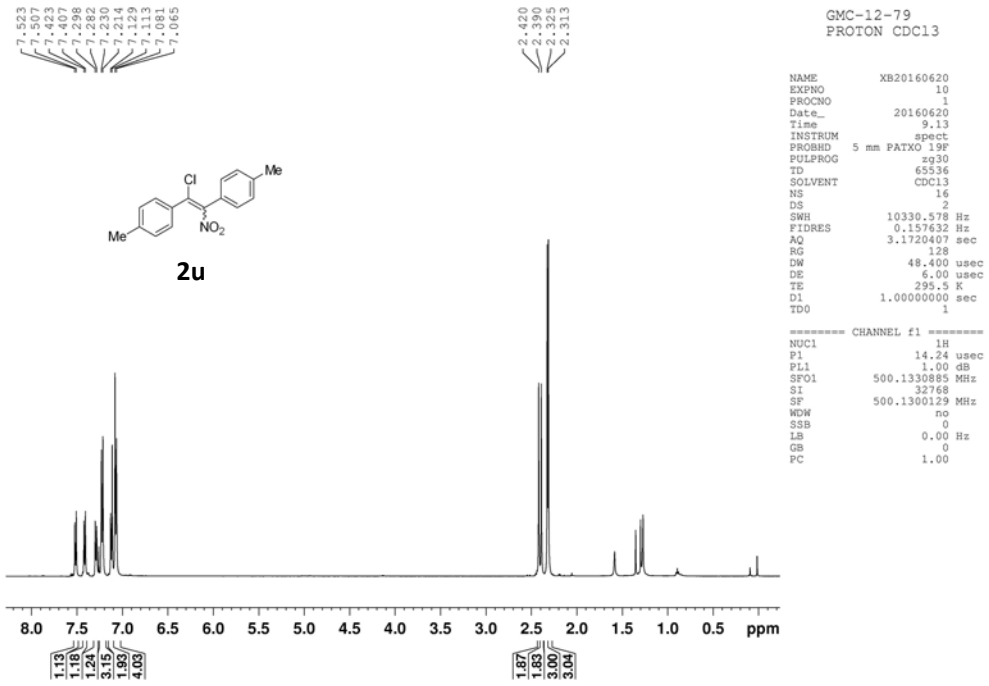

146.41
137.14
135.15
134.36
130.31
130.22
128.89
128.82
127.69
127.59
18.14
18.03
GMC-11-128-1
C13CPD CDC13
NAME XB20160411
EXPNO 31
PROCNO 1
Date_ 20160411
Time 17.31
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 97
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 161.3
DW 16.650 usec
DE 6.00 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577791 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
Cl
CH3
NO2
2s
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm




```
GMC-12-87-1
PROTON CDCl3

NAME          XB20160627
EXPNO                 15
PROCNO                 1
Date_           20160627
Time               11.53
INSTRUM            spect
PROBHD   5 mm PATXO 19F
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1720407 sec
RG                    57
DW               48.400 usec
DE                6.00 usec
TE               295.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.24 usec
PL1                1.00 dB
SFO1         500.1330885 MHz
SI                 32768
SF           500.1300000 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                1.00
```

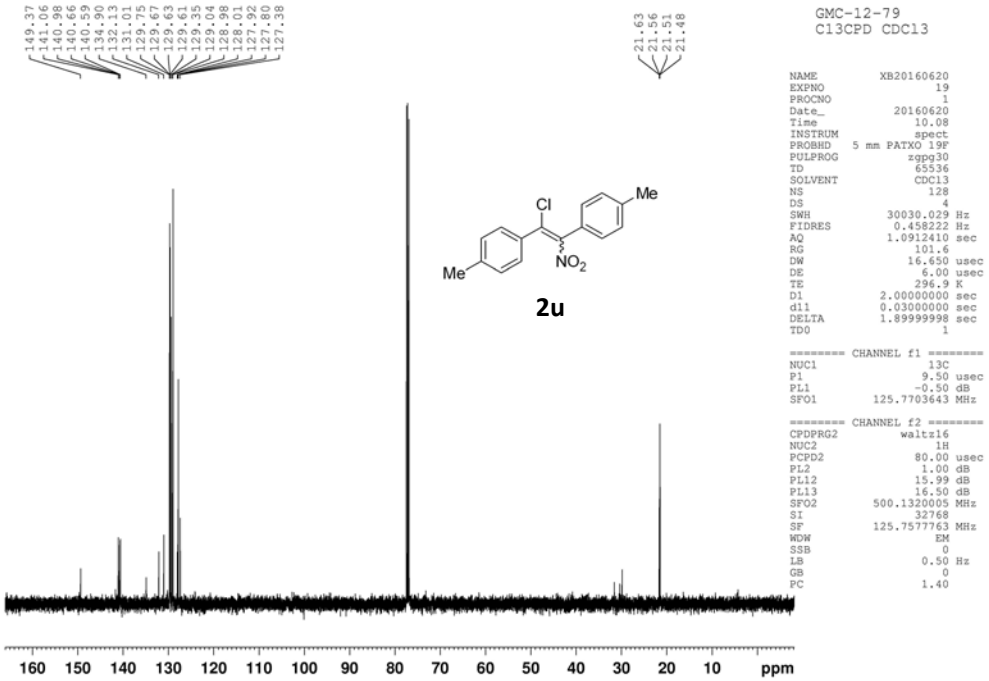



```
GMC-12-87-1
C13CPD CDCl3

NAME          XB20160627
EXPNO                 17
PROCNO                 1
Date_           20160627
Time               12.34
INSTRUM            spect
PROBHD   5 mm PATXO 19F
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     4
SWH           30030.029 Hz
FIDRES        0.458222 Hz
AQ            1.0912410 sec
RG                   181
DW               16.650 usec
DE                6.00 usec
TE               296.5 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                9.50 usec
PL1               -0.50 dB
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                1.00 dB
PL12              15.99 dB
PL13              16.50 dB
SFO2         500.1320005 MHz
SI                 32768
SF           125.7577773 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                1.40
```



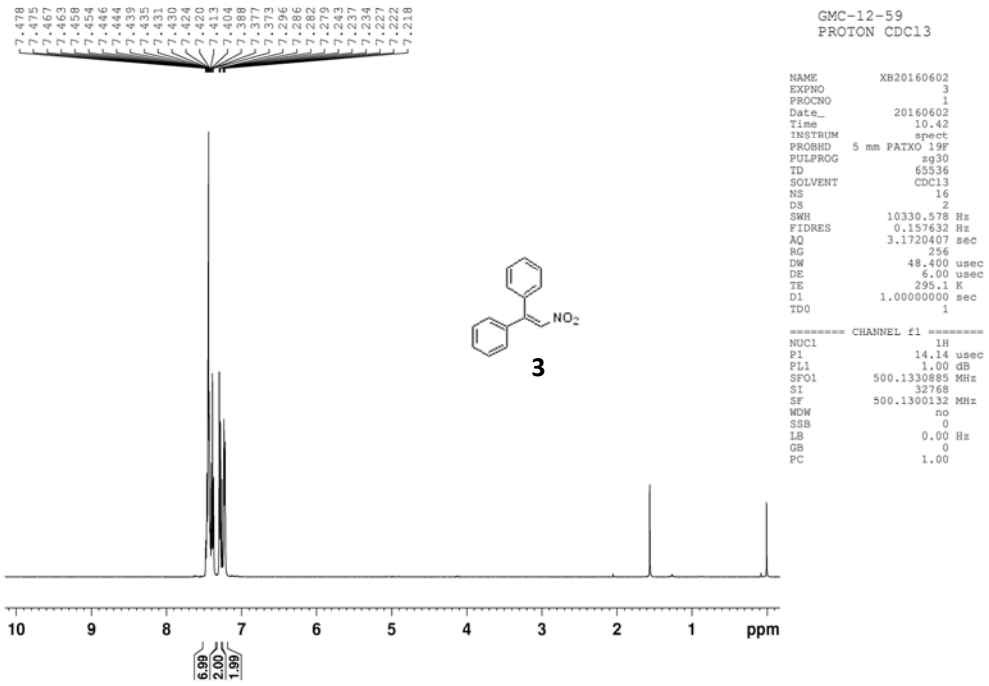

GMC-12-79
PROTON CDC13

| | |
|---|---|
| NAME | XB20160620 |
| EXPNO | 10 |
| PROCNO | 1 |
| Date_ | 20160620 |
| Time | 9.13 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 16 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 128 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 295.5 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.24 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300129 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |



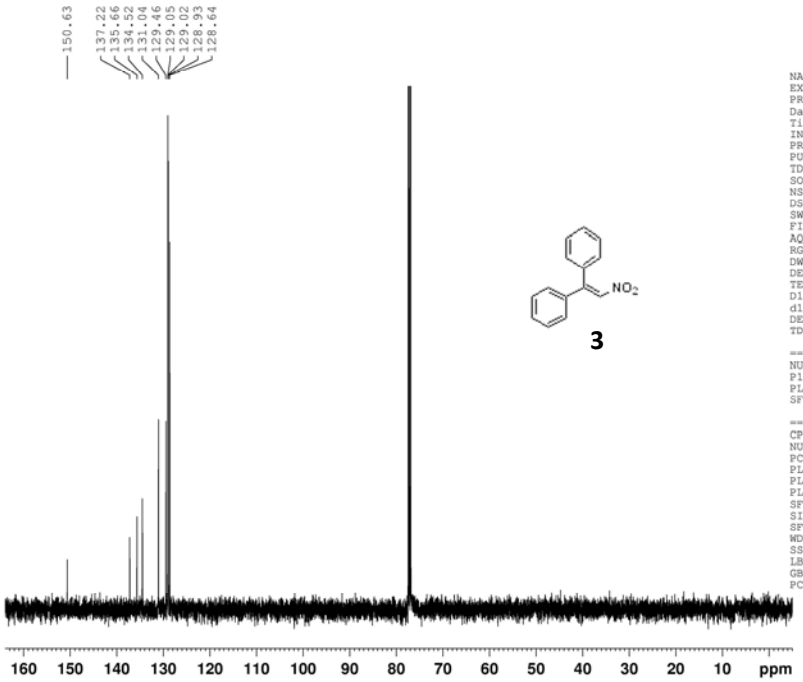

GMC-12-79
C13CPD CDC13

| | |
|---|---|
| NAME | XB20160620 |
| EXPNO | 19 |
| PROCNO | 1 |
| Date_ | 20160620 |
| Time | 10.08 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 101.6 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.9 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 15.99 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577763 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 0.50 Hz |
| GB | 0 |
| PC | 1.40 |



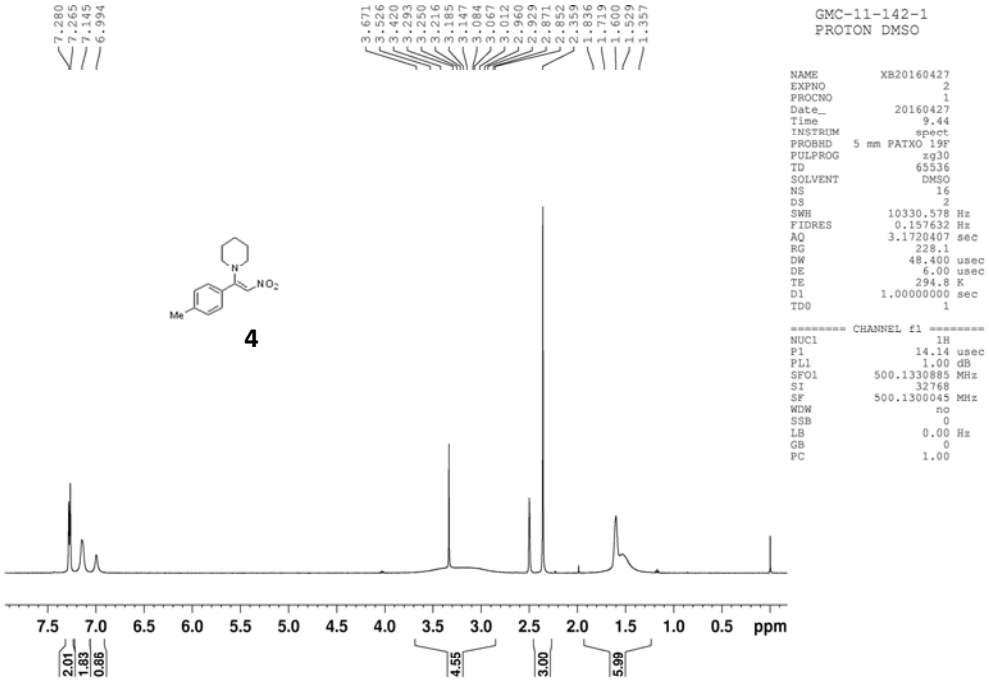

GMC-12-59
PROTON CDC13

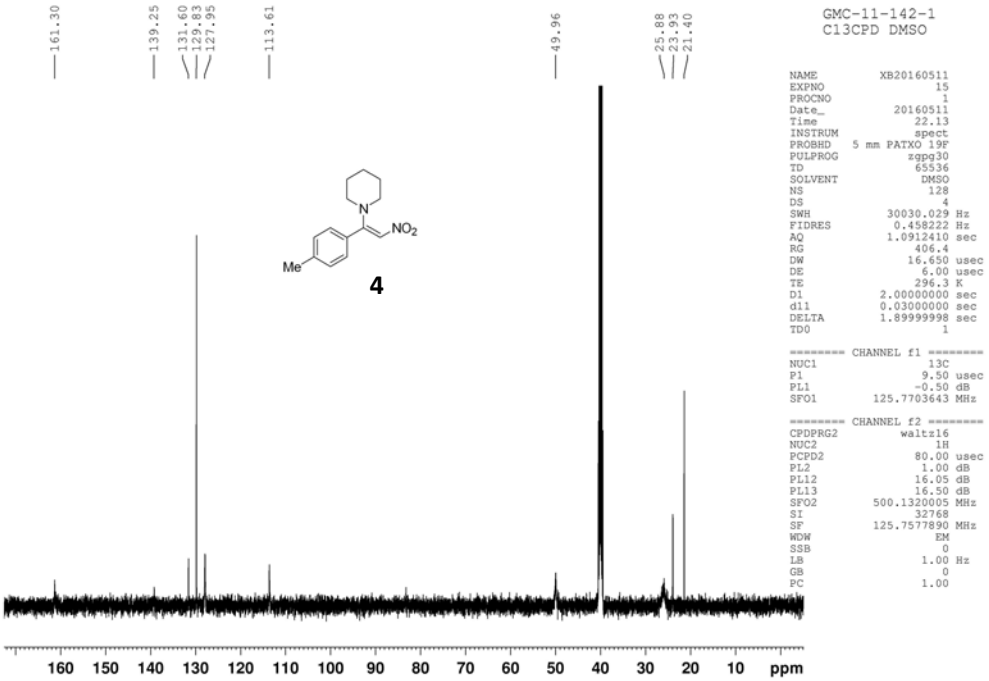
| | |
|---|---|
| NAME | XB20160602 |
| EXPNO | 3 |
| PROCNO | 1 |
| Date_ | 20160602 |
| Time | 10.42 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 16 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 256 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 295.1 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300132 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |

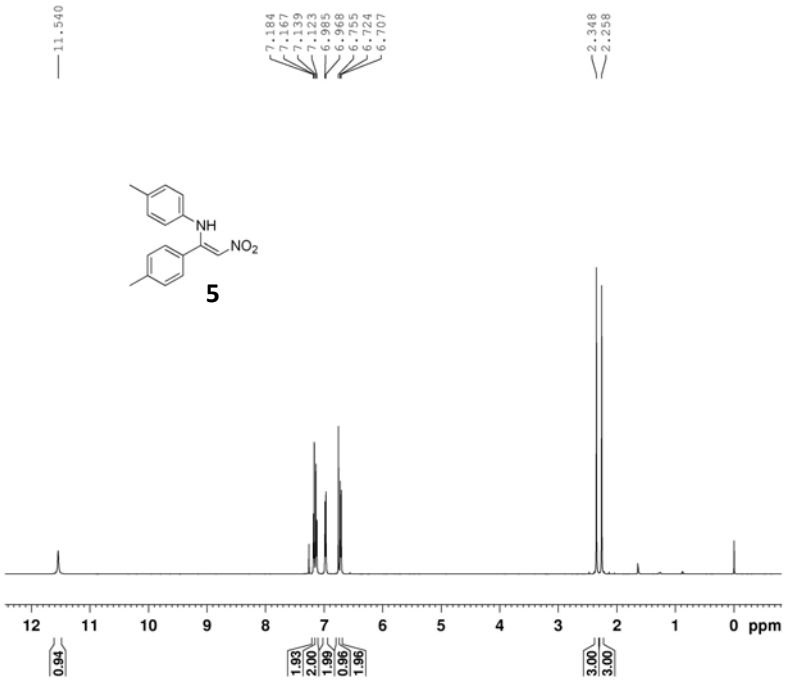



GMC-12-59
C13CPD CDC13

| | |
|---|---|
| NAME | XB20160603 |
| EXPNO | 1 |
| PROCNO | 1 |
| Date_ | 20160603 |
| Time | 8.27 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 161.3 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.5 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577745 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |

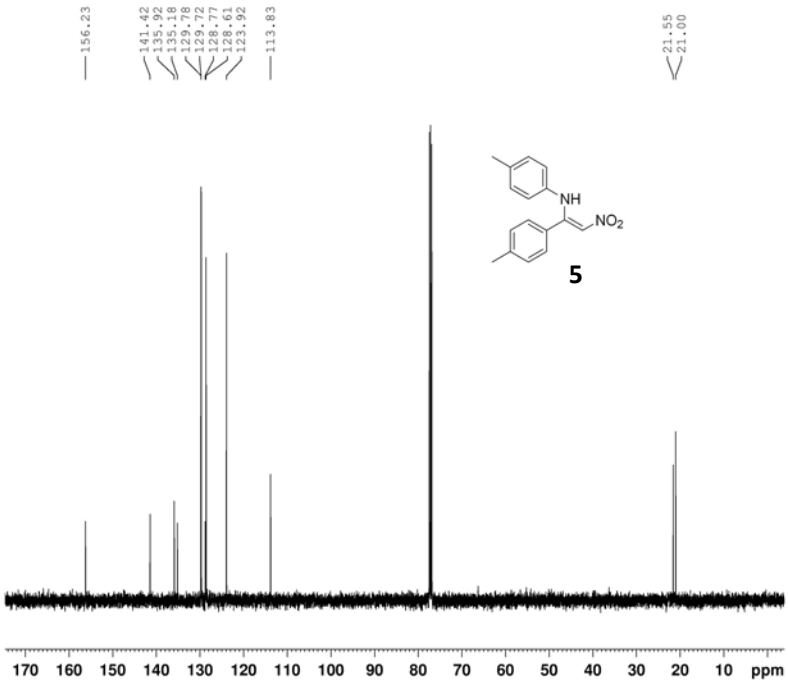

7.280
7.265
7.145
6.994
3.671
3.526
3.420
3.293
3.250
3.216
3.185
3.147
3.084
3.067
3.012
2.960
2.929
2.871
2.852
2.359
1.836
1.719
1.600
1.529
1.357
GMC-11-142-1
PROTON DMSO
NAME XB20160427
EXPNO 2
PROCNO 1
Date_ 20160427
Time 9.44
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 228.1
DW 48.400 usec
DE 6.00 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.14 usec
PL1 1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300045 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Me
NO2
4
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 ppm
2.01
1.83
0.86
4.55
3.00
5.99


161.30
139.25
131.60
129.83
127.95
113.61
49.96
25.88
23.93
21.40
GMC-11-142-1
C13CPD DMSO
NAME XB20160511
EXPNO 15
PROCNO 1
Date_ 20160511
Time 22.13
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 128
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 406.4
DW 16.650 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -0.50 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 16.05 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00
Me
NO2
4
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



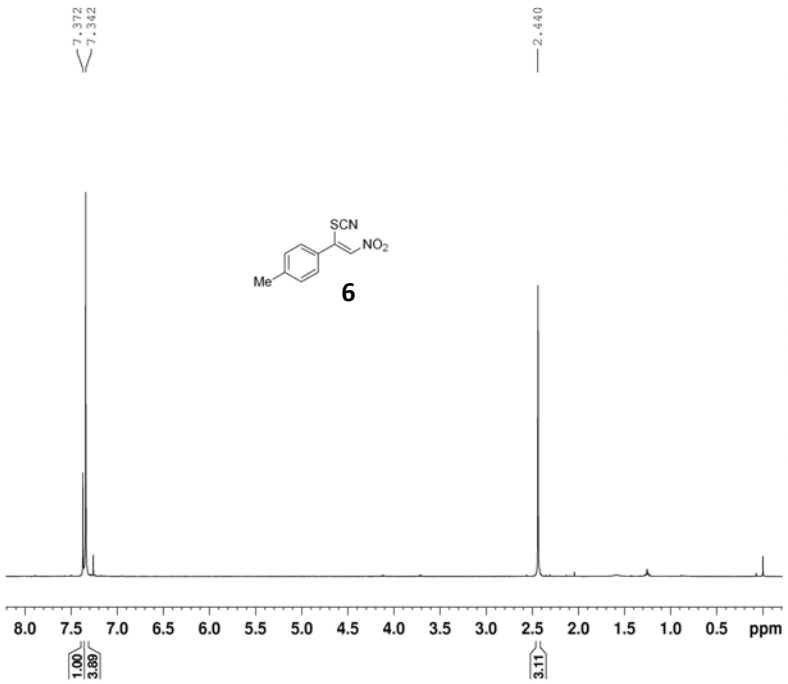

GMC-11-127-1
PROTON CDC13

| | |
|---|---|
| NAME | XB20160414 |
| EXPNO | 7 |
| PROCNO | 1 |
| Date_ | 20160414 |
| Time | 11.02 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 16 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 181 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 295.3 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300129 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |



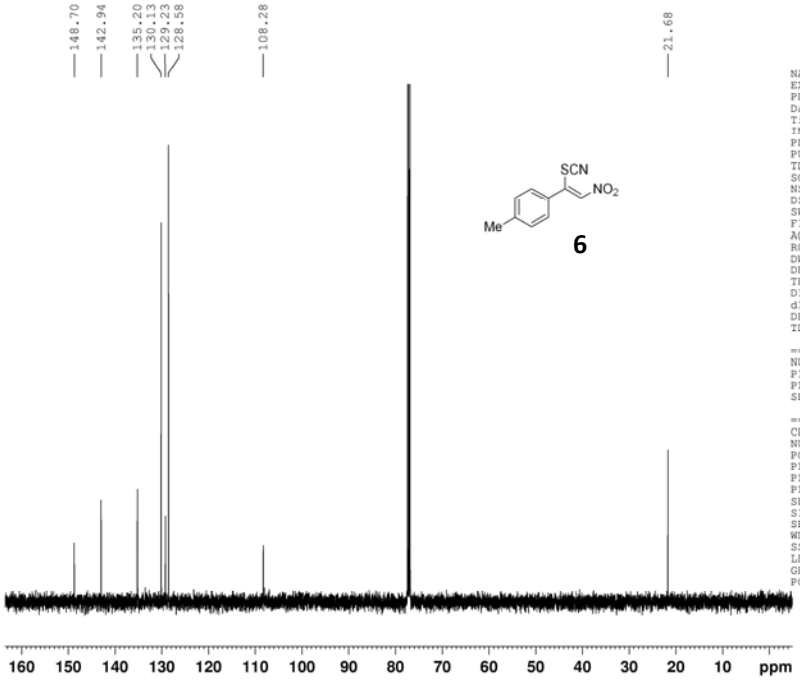

GMC-11-127-1
C13CPD CDC13

| | |
|---|---|
| NAME | XB20160414 |
| EXPNO | 9 |
| PROCNO | 1 |
| Date_ | 20160414 |
| Time | 11.13 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 114 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.5 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577773 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |



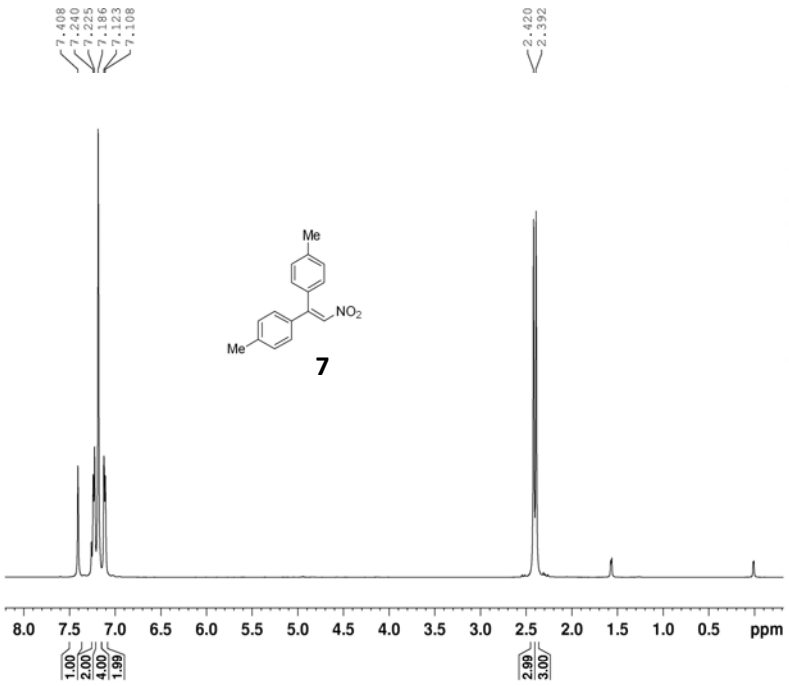

GMC-11-135-2
PROTON CDC13

| | |
|---|---|
| NAME | XB20160418 |
| EXPNO | 27 |
| PROCNO | 1 |
| Date_ | 20160418 |
| Time | 14.08 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 16 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 256 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 294.7 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300129 MHz |
| WDW | no |
| SSB | 0 |
| LB | 0.00 Hz |
| GB | 0 |
| PC | 1.00 |



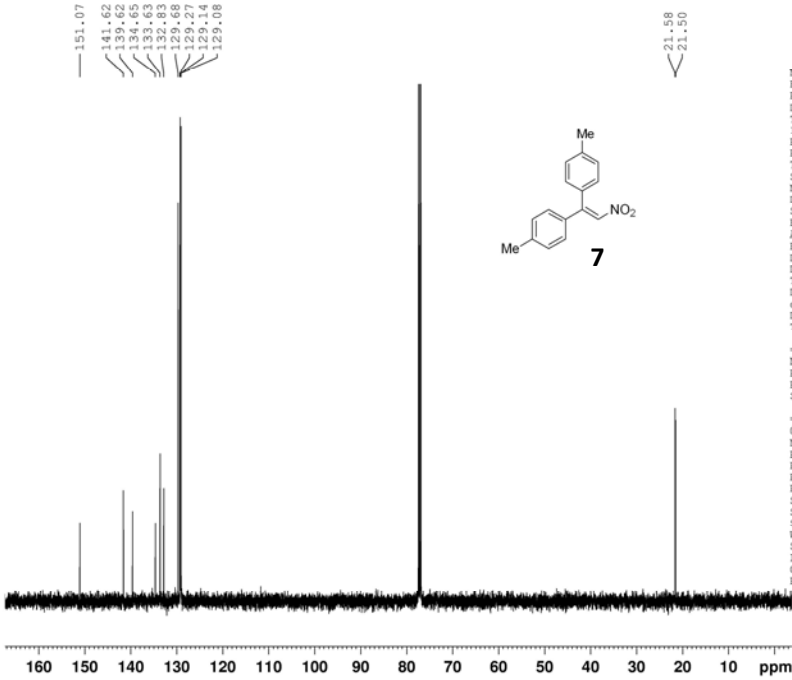

GMC-11-135-2
C13CPD CDC13

| | |
|---|---|
| NAME | XB20160418 |
| EXPNO | 37 |
| PROCNO | 1 |
| Date_ | 20160419 |
| Time | 7.23 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 114 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 295.9 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577763 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |




GMC-12-56
PROTON CDC13

| | |
|---|---|
| NAME | XB20160602 |
| EXPNO | 13 |
| PROCNO | 1 |
| Date_ | 20160602 |
| Time | 14.59 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 8 |
| DS | 2 |
| SWH | 10330.578 Hz |
| FIDRES | 0.157632 Hz |
| AQ | 3.1720407 sec |
| RG | 181 |
| DW | 48.400 usec |
| DE | 6.00 usec |
| TE | 296.1 K |
| D1 | 1.00000000 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 1H |
| P1 | 14.14 usec |
| PL1 | 1.00 dB |
| SFO1 | 500.1330885 MHz |
| SI | 32768 |
| SF | 500.1300129 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 2.00 Hz |
| GB | 0 |
| PC | 1.00 |




GMC-12-56
C13CPD CDC13

| | |
|---|---|
| NAME | XB20160603 |
| EXPNO | 16 |
| PROCNO | 1 |
| Date_ | 20160603 |
| Time | 11.48 |
| INSTRUM | spect |
| PROBHD | 5 mm PATXO 19F |
| PULPROG | zgpg30 |
| TD | 65536 |
| SOLVENT | CDC13 |
| NS | 128 |
| DS | 4 |
| SWH | 30030.029 Hz |
| FIDRES | 0.458222 Hz |
| AQ | 1.0912410 sec |
| RG | 161.3 |
| DW | 16.650 usec |
| DE | 6.00 usec |
| TE | 296.5 K |
| D1 | 2.00000000 sec |
| d11 | 0.03000000 sec |
| DELTA | 1.89999998 sec |
| TD0 | 1 |
| ======== CHANNEL f1 ======== | |
| NUC1 | 13C |
| P1 | 9.50 usec |
| PL1 | -0.50 dB |
| SFO1 | 125.7703643 MHz |
| ======== CHANNEL f2 ======== | |
| CPDPRG2 | waltz16 |
| NUC2 | 1H |
| PCPD2 | 80.00 usec |
| PL2 | 1.00 dB |
| PL12 | 16.05 dB |
| PL13 | 16.50 dB |
| SFO2 | 500.1320005 MHz |
| SI | 32768 |
| SF | 125.7577763 MHz |
| WDW | EM |
| SSB | 0 |
| LB | 1.00 Hz |
| GB | 0 |
| PC | 1.40 |