CHEMMEDCHEM
Chemistry & Drug Discovery

Supporting Information

Novel 1,4-Substituted-1,2,3-Triazoles as Antitubercular Agents

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cmdc_201500051_sm_miscellaneous_information.pdf

General Experimental

All ^{1}H and ^{13}C NMR spectra were recorded on a Jeol JNM-EX 270 MHz, Jeol JNM-EX 400 MHz or Bruker AVANCE III 500 MHz standard bore (solution) as indicated. Samples were dissolved in deuterated chloroform ($CDCl_3$) with the residual solvent peak used as an internal reference ($CDCl_3$ – δ H 7.26 ppm). Proton spectra are reported as follows: chemical shift δ (ppm), (integral, multiplicity (s = singlet, br s = broad singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, m = multiplet), coupling constant J (Hz), assignment).

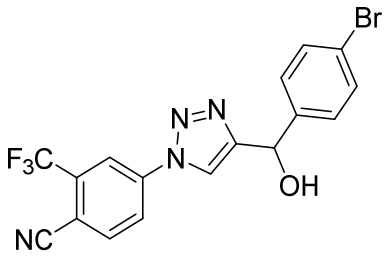
Thin Layer Chromatography (TLC) was performed using aluminium-backed Merck TLC Silica gel 60 F254 plates, and samples were visualised using 254 nm ultraviolet (UV) light, and potassium permanganate/potassium carbonate oxidising dip (1:1:100 $KMnO_4$:K_2CO_3:H_2O w/w).

Column Chromatography was performed using silica gel 60 (70-230 mesh). All solvents used were AR grade. Specialist reagents were obtained from Sigma-Aldrich Chemical Company and used without further purification. Petroleum spirits refers to the fraction boiling between 40-60 °C.

HRMS was found via a 6210 MSD TOF mass spectrometer under the conditions: gas temperature (350 ^{0}C), vaporizer (28 ^{0}C), capillary voltage (3.0 kV), cone voltage (40 V), nitrogen flow rate (7.0 L/min), nebuliser (15 psi). Samples were dissolved in MeOH.

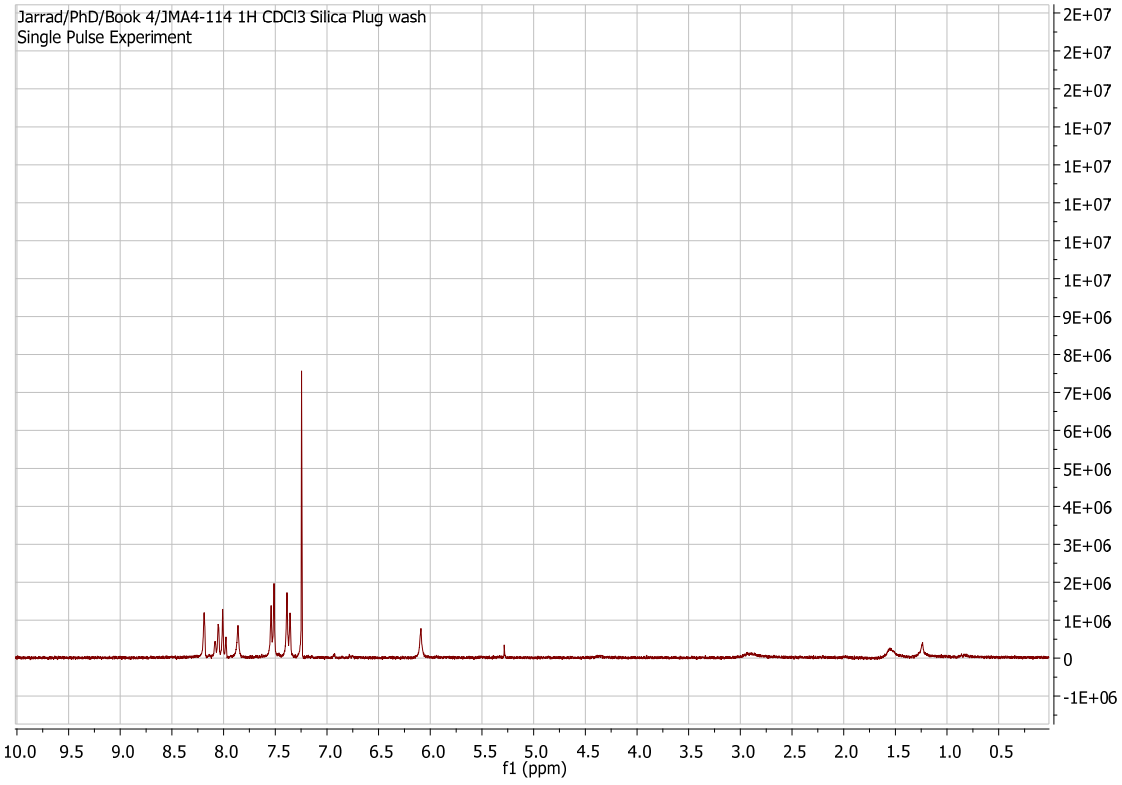
Melting points were found on a Stuart Scientific Melting Point Apparatus SMP3, v.5 and are uncorrected.

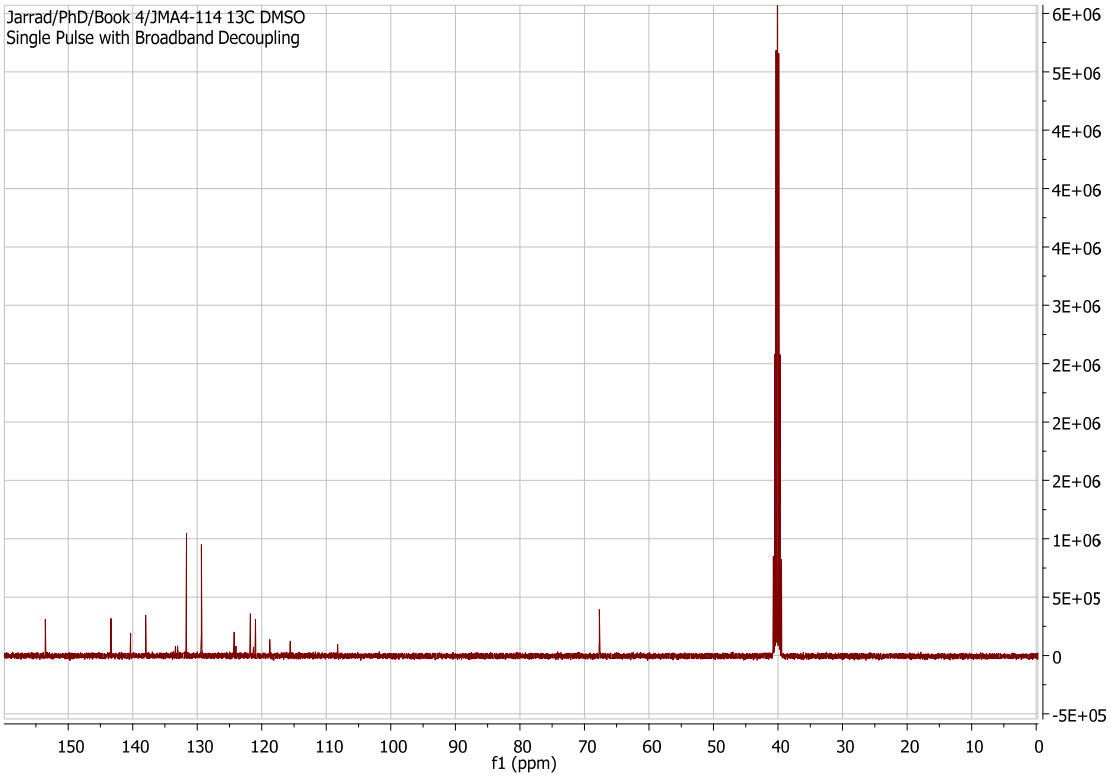
Solubility of the compounds was tested by dissolving an amount of the compound in DMSO and then taking an aliquot and diluting in H_2O to achieve the required concentration at 0.5% DMSO in H_2O.



4-(4-((4-Bromophenyl)(hydroxy)methyl)-1H-1,2,3-triazol-1-yl)-2-(trifluoromethyl)benzonitrile **6i**

mp 68.0-69.3°C; ^{1}H NMR ($CDCl_3$, 270 MHz): δ 8.19 (1H, s, Ar-H) 8.08-7.98 (2H, m, Ar-H) 7.86 (1H, s, Ar-H) 7.53 (2H, d, J = 8.1 Hz, Ar-H) 7.38 (2H, d, J = 8.1 Hz, Ar-H) 6.09 (1H, s, CH) 2.94 (1H, br s, OH); ^{13}C NMR (d_6-DMSO, 100 MHz): δ 153.6, 143.4, 140.3, 138.0, 133.2 (q, $J^{2}_{C\text{-}F}$ = 33 Hz) 131.7, 129.4, 124.3, 122.6 (q, $J^{1}_{C\text{-}F}$ = 273 Hz) 121.8, 121.0, 118.7, 115.6, 108.2, 67.7; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -62.2; IR (cm^{-1}): 3359, 3143, 1615, 1509, 1402, 1183, 1010, 738; HRMS (ES) calced for $(C_{17}H_{10}BrF_3N_4ONa)^{+}$ 444.9823, found 444.9846

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3E+06
2E+06
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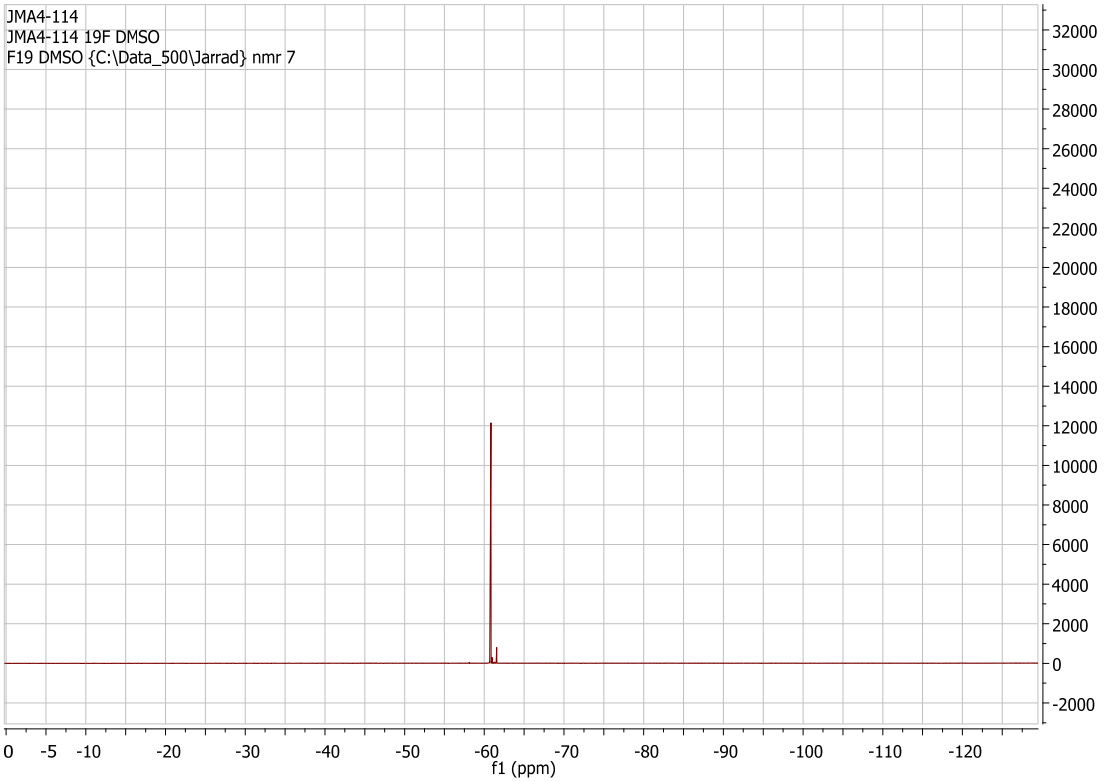
f1 (ppm)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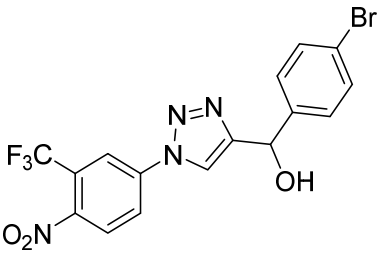
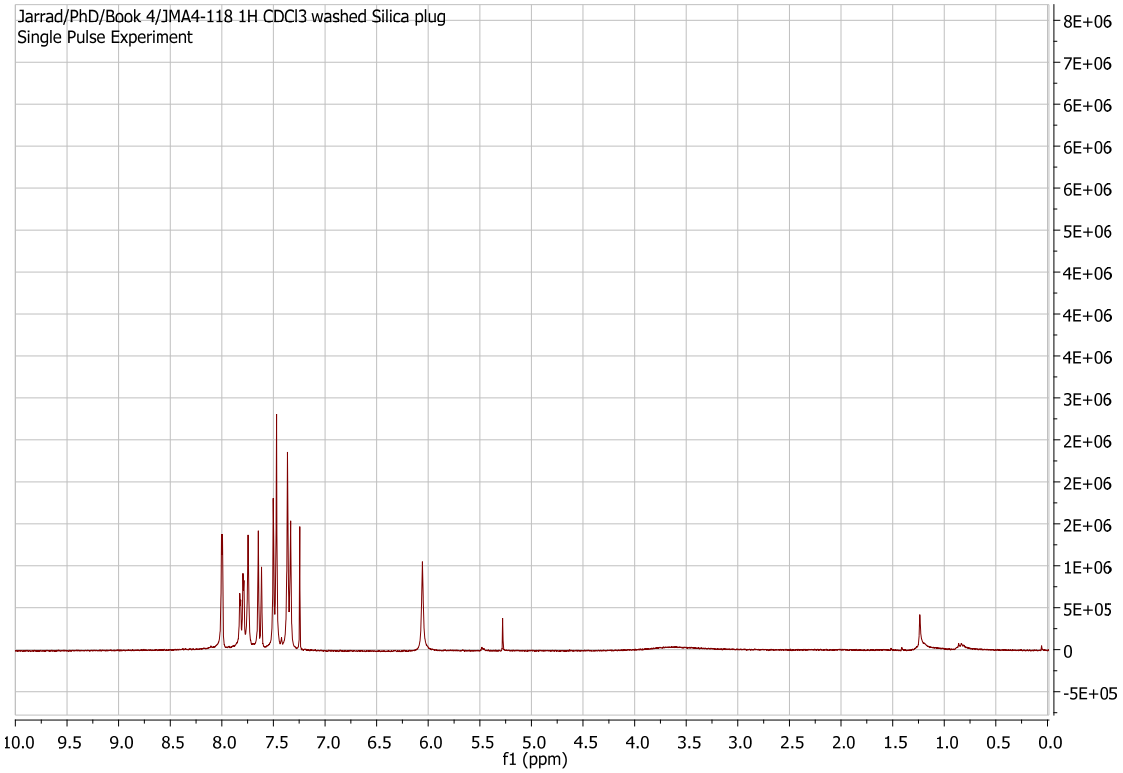
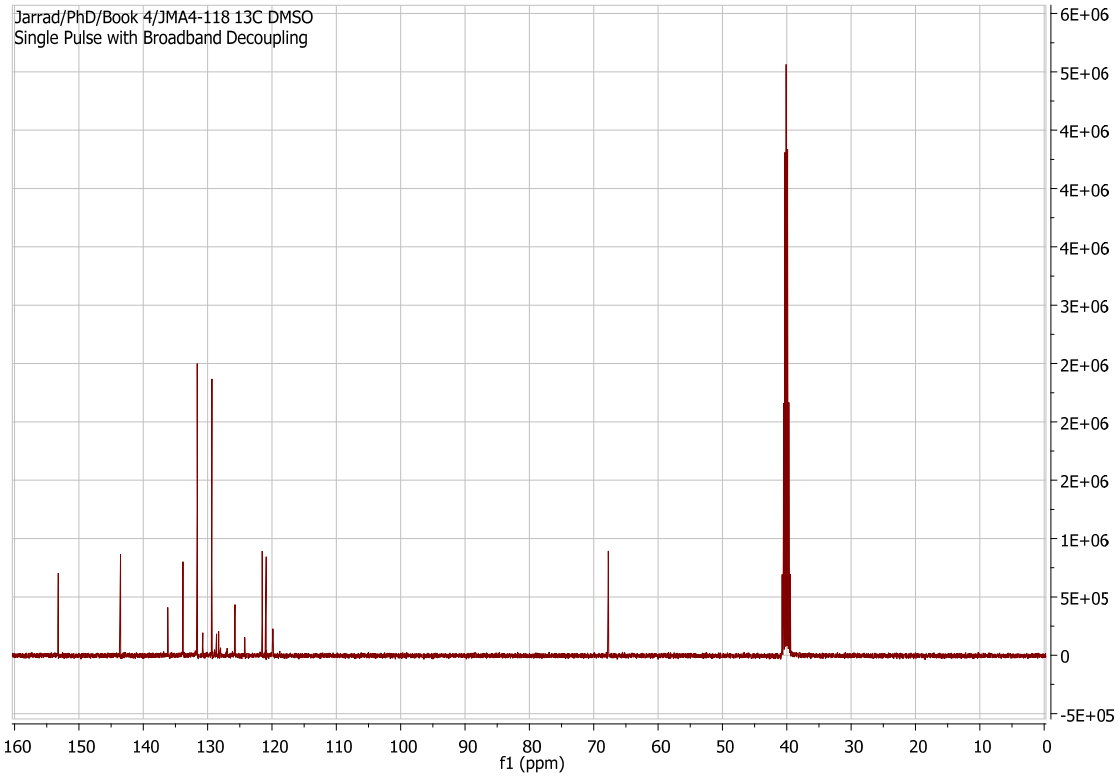
f1 (ppm)



(4-Bromophenyl)(1-(4-nitro-3-(trifluoromethyl)phenyl)-1H-1,2,3-triazol-4-yl)methanol **6j**

mp 66.5-67.5°C; ^{1}H NMR ($CDCl_3$, 270 MHz): δ 8.00 (1H, s, Ar-H) 7.83-7.74 (2H, m, Ar-H) 7.63 (1H, d, J = 10 Hz, Ar-H) 7.49 (2H, J = 8.1 Hz, Ar-H) 6.06 (1H, s, CH), OH not observed ^{13}C NMR (d_6-DMSO, 100 MHz): δ 153.2, 143.5, 136.2, 133.8, 131.6, 129.3, 128.4 (q, $J^{2}_{C\text{-}F}$ = 31 Hz) 125.8, 122.9 (q, $J^{1}_{C\text{-}F}$ = 272 Hz) 121.5, 120.9, 119.9 (q, $J^{3}_{C\text{-}F}$ = 6 Hz) 67.7; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -63.0; IR (cm^{-1}): 3353, 3153, 1490, 1290, 1143, 1036, 825; HRMS (ES) calced for $(C_{16}H_{11}BrF_3N_4O_3)^{+}$ 442.9961, found 442.9924

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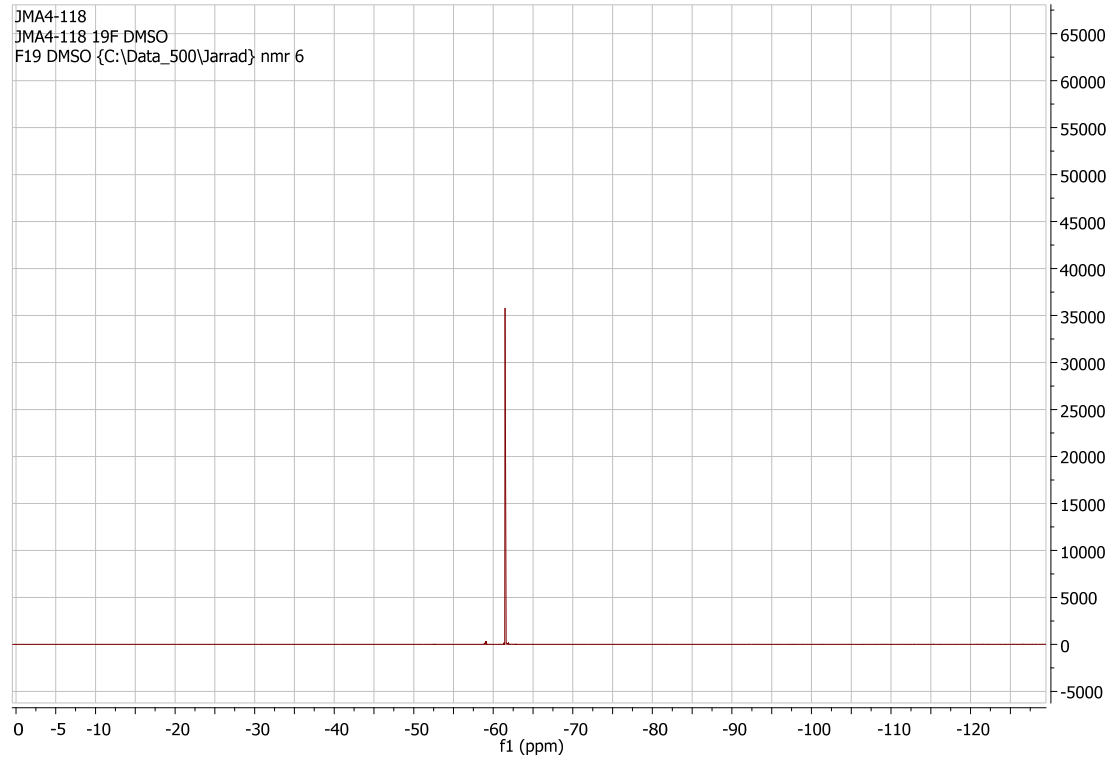
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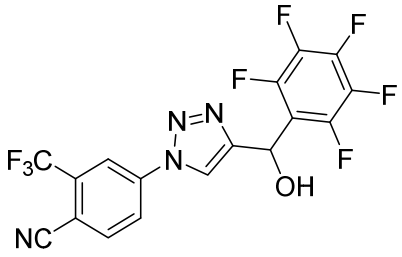
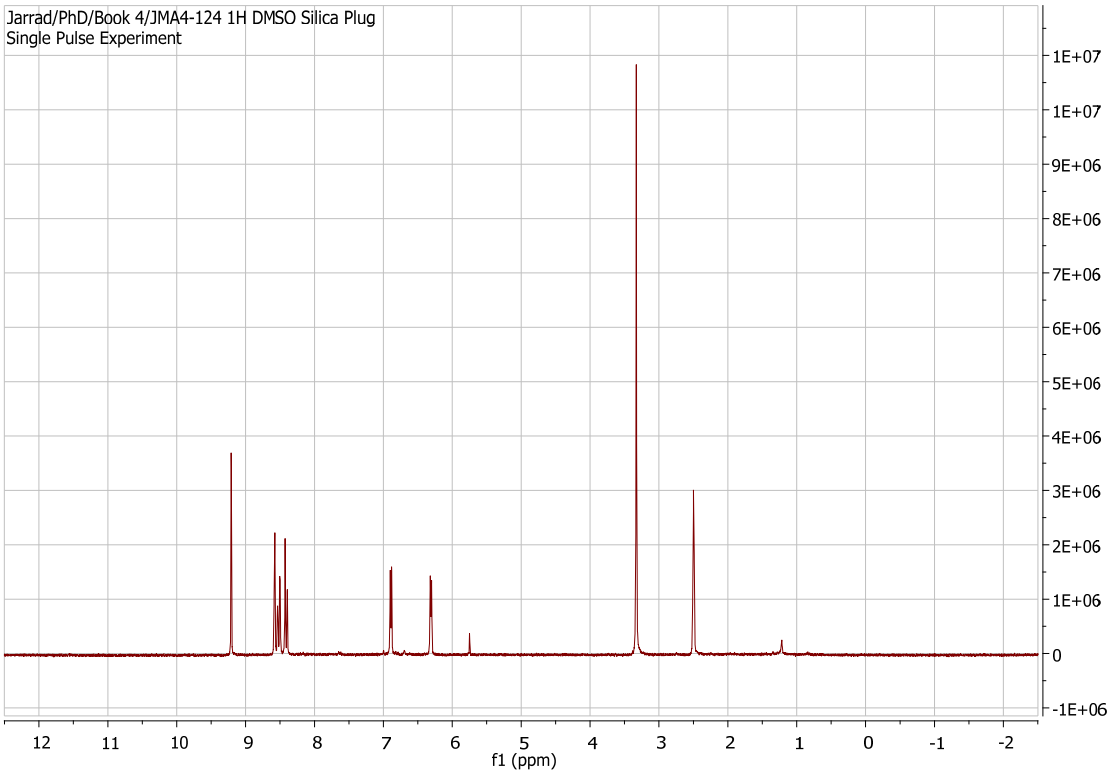
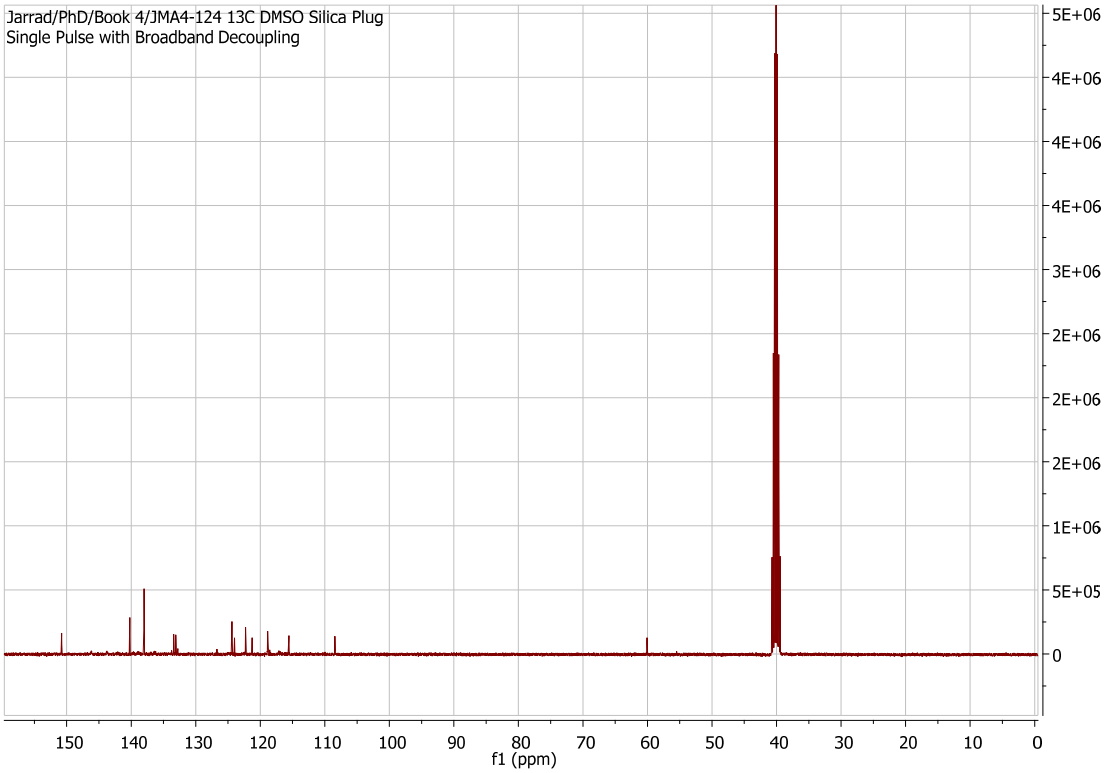
f1 (ppm)



(1-(4-Nitro-3-(trifluoromethyl)phenyl)-1H-1,2,3-triazol-4-yl)(perfluorophenyl)methanol **6e**

mp 153.6-155.3°C; ^{1}H NMR (d_6-DMSO, 270 MHz): δ 9.07 (1H, s, Ar-H) 8.58 (1H, s, Ar-H) 8.53 (1H, d, J = 8.1 Hz, Ar-H) 8.41 (1H, d, J = 10.8 Hz, Ar-H) 6.89 (1H, d, J= 5.4 Hz, CH) 6.31 (1H, d, J = 5.4 Hz, Ar-H); ^{13}C NMR (d_6-DMSO, 100 MHz): δ 150.8, 146.2 (m) 143.8 (m) 140.2, 138.8 (m) 138.0, 136.4 (m) 133.26 (q, $J^{2}_{C\text{-}F}$ = 32 Hz) 124.3, 123.6 (q, $J^{1}_{C\text{-}F}$ = 273 Hz) 122.3, 118.8 (q, $J^{3}_{C\text{-}F}$ = 5 Hz) 117.1 (m) 115.5, 108.4; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -62.2, -142.7, -152.7, -160.8; IR (cm^{-1}): 3384, 1616, 1506, 1312, 1124, 996; HRMS (ES) calced for $(C_{17}H_7F_8N_4O)^+$ 435.04866, found 435.04435.

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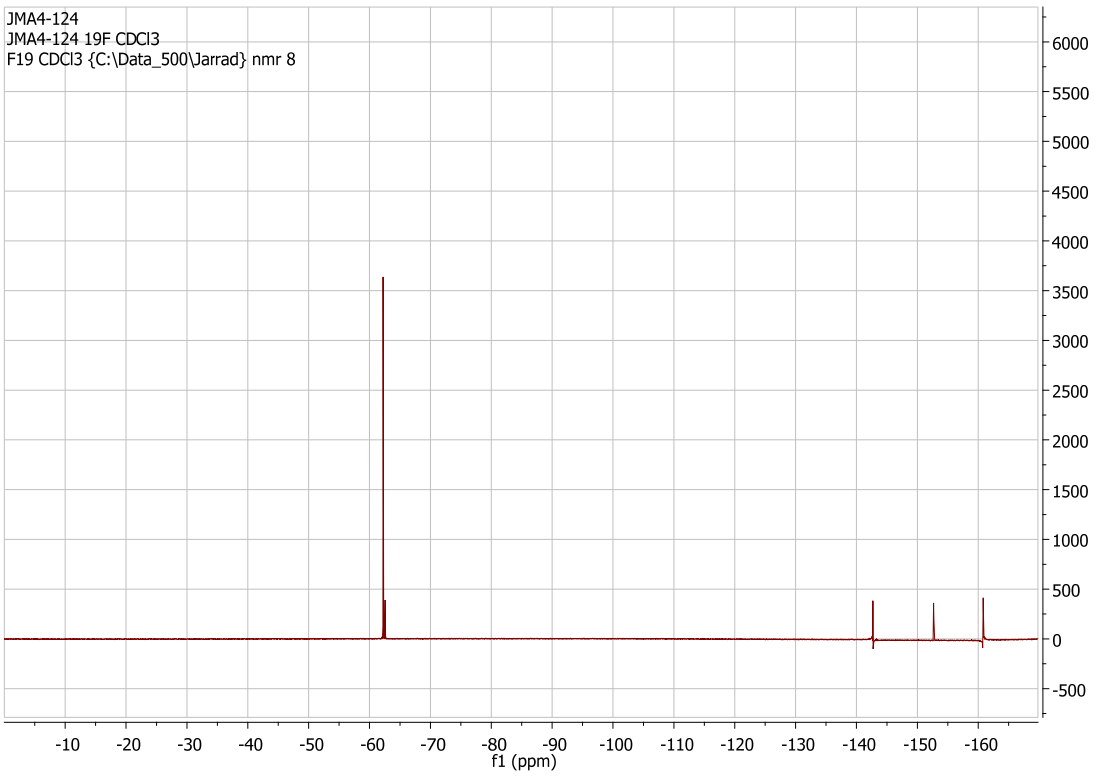
f1 (ppm)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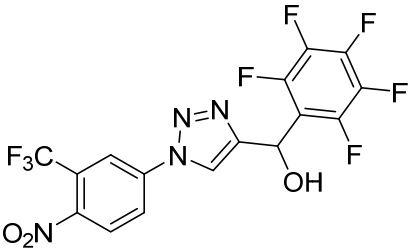
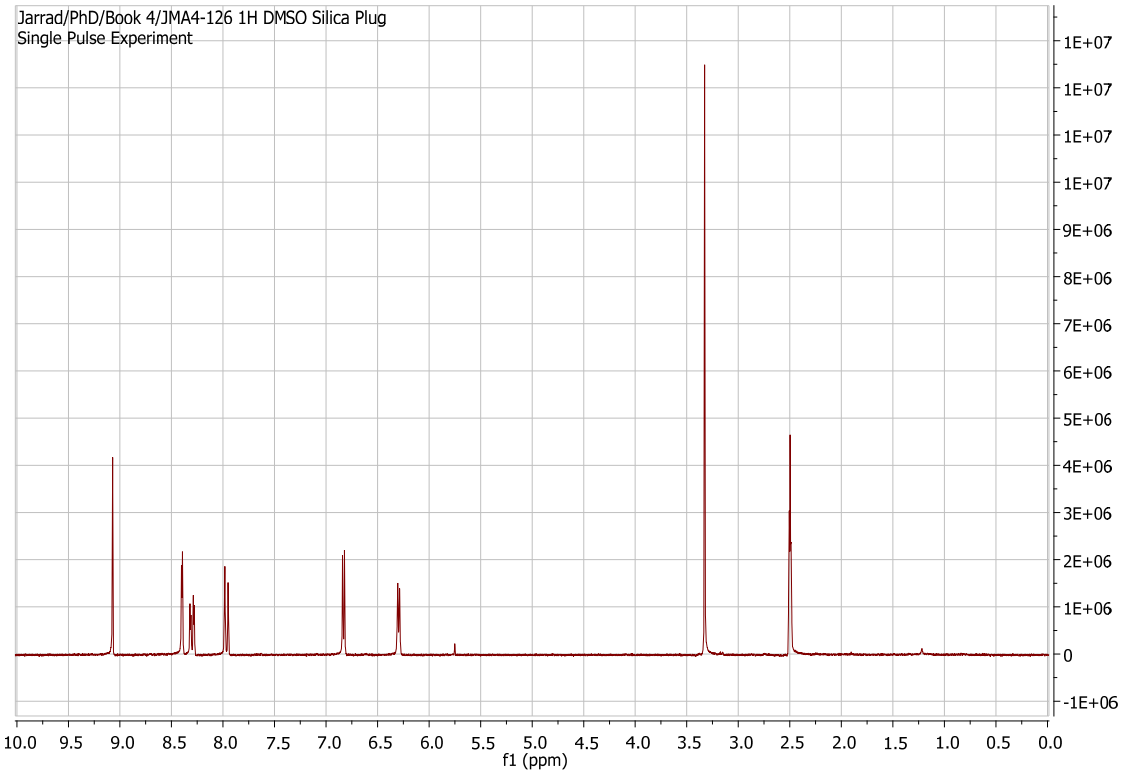
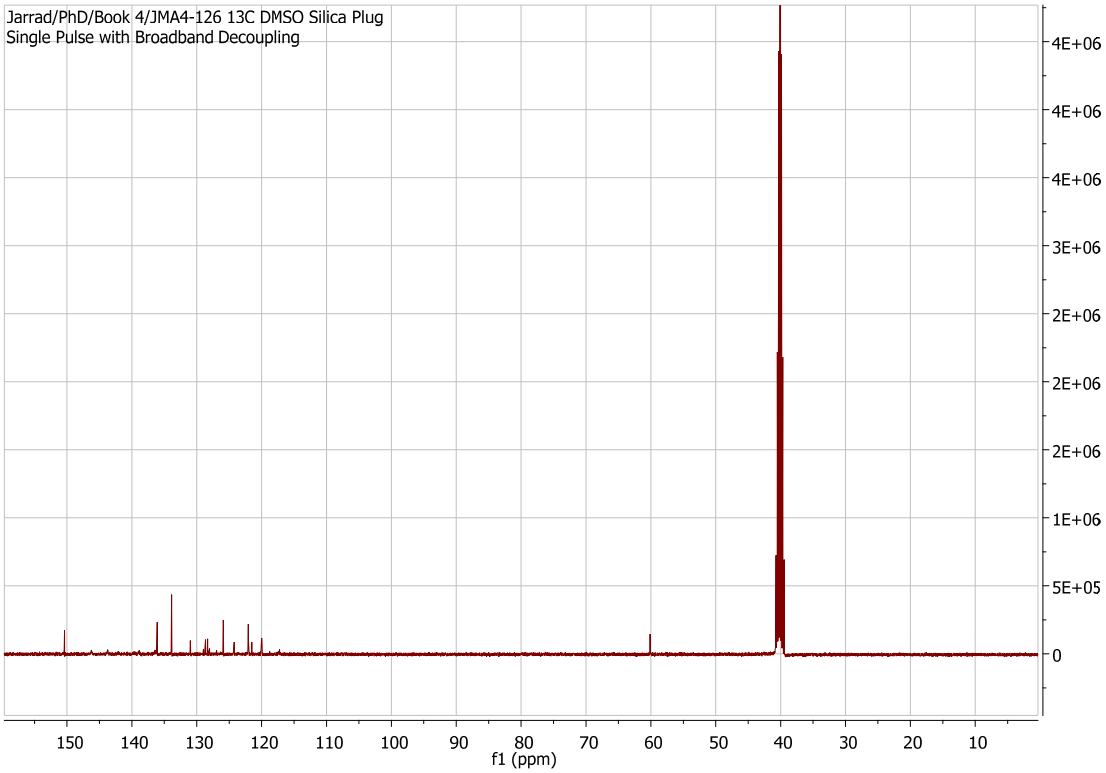
f1 (ppm)



(1-(4-Nitro-3-(trifluoromethyl)phenyl)-1H-1,2,3-triazol-4-yl)(perfluorophenyl)methanol **6f**

mp 148.0-149.8°C; ^{1}H NMR (d_6-DMSO, 270 MHz): δ 9.07 (1H, s, Ar-H) 8.40 (1H, d, J = 2.7 Hz, Ar-H) 8.32 (1H, d, J = 2.7 Hz, Ar-H) 8.29 (1H, d, J = 2.7 Hz, Ar-H) 7.97 (1H, d, J = 8.1 Hz, Ar-H) 6.83 (1H, d, J = 5.4 Hz, CH) 6.30 (1H, d, J = 2.7 Hz, OH); ^{13}C NMR (d_6-DMSO, 100 MHz): δ 150.4, 146.2 (m) 143.8 (m) 138.9 (m) 136.4 (m) 136.1, 133.9, 131.0, 128.5 (q, $J^{2}_{C\text{-}F}$ = 31 Hz) 125.9, 122.9 (q, $J^{1}_{C\text{-}F}$ = 272 Hz) 121.1, 120.0 (m) 117.2 (m); ^{19}F NMR (d_6-DMSO, 470 MHz): δ -63.1, -142.7, -153.1, -161.0; IR (cm^{-1}): 3337, 1522, 1311, 1179, 1037, 996: HRMS calced for $C_{16}H_7F_8N_4O_3$ $[MH]^+$ 455.0385, found 455.0410

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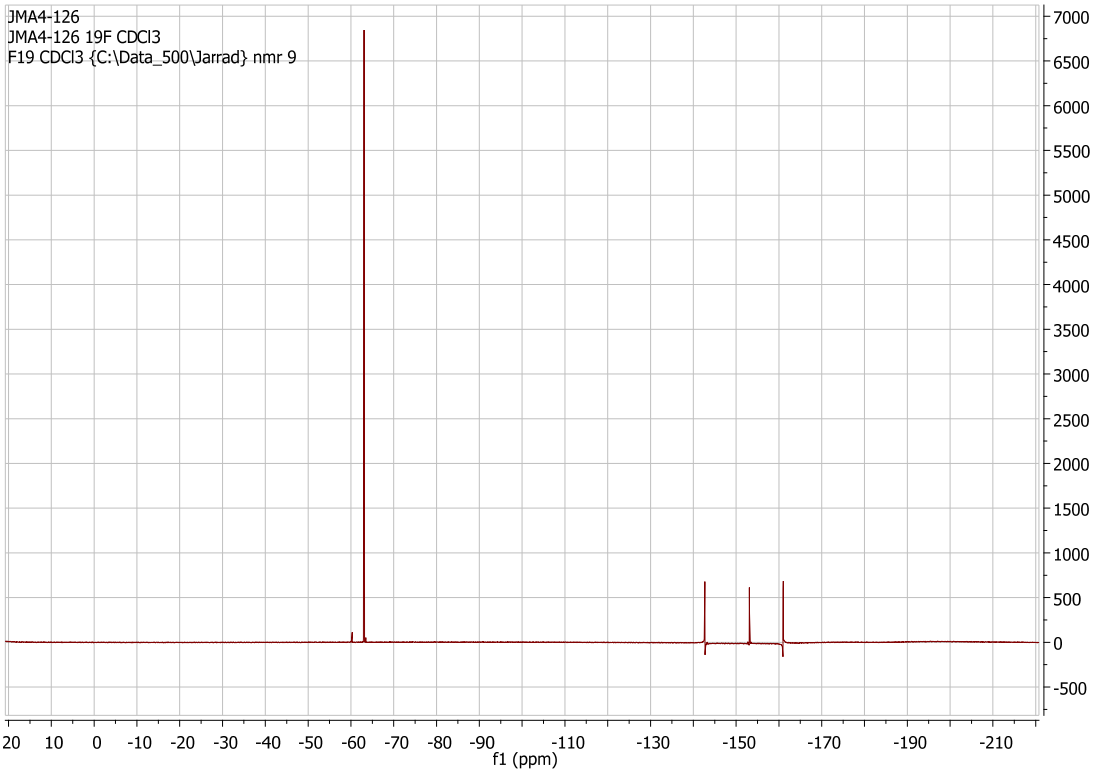
f1 (ppm)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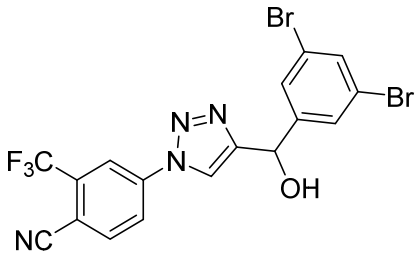
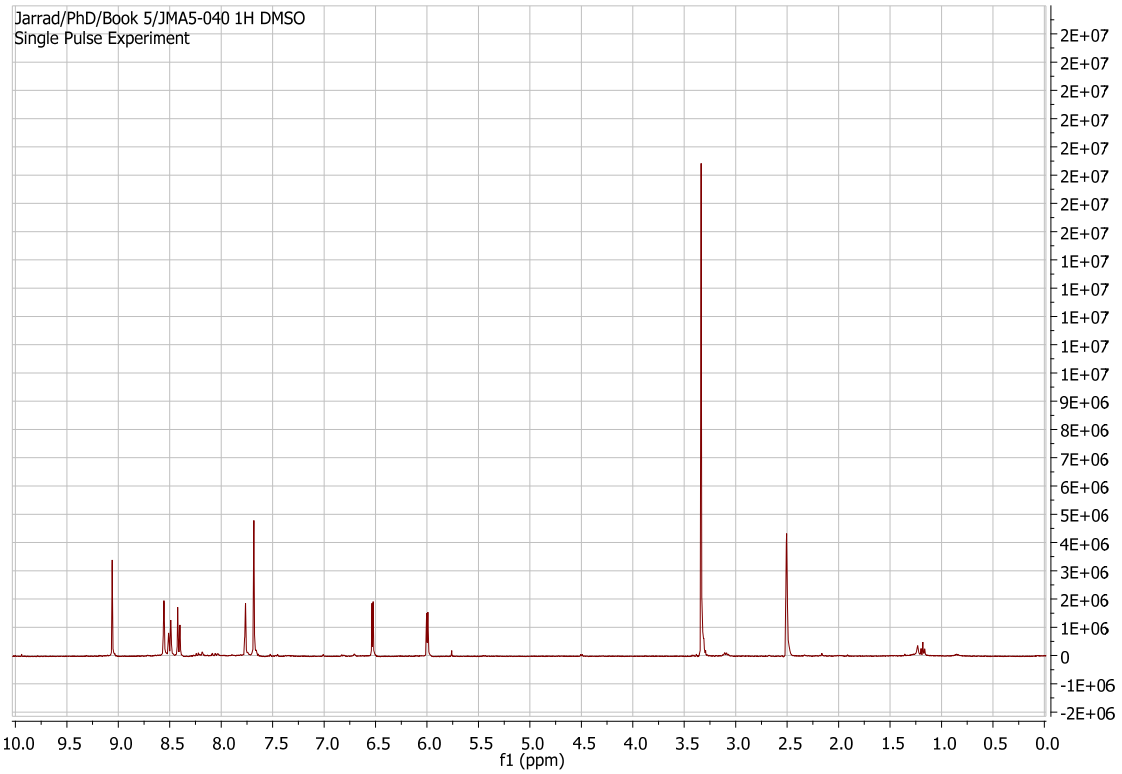
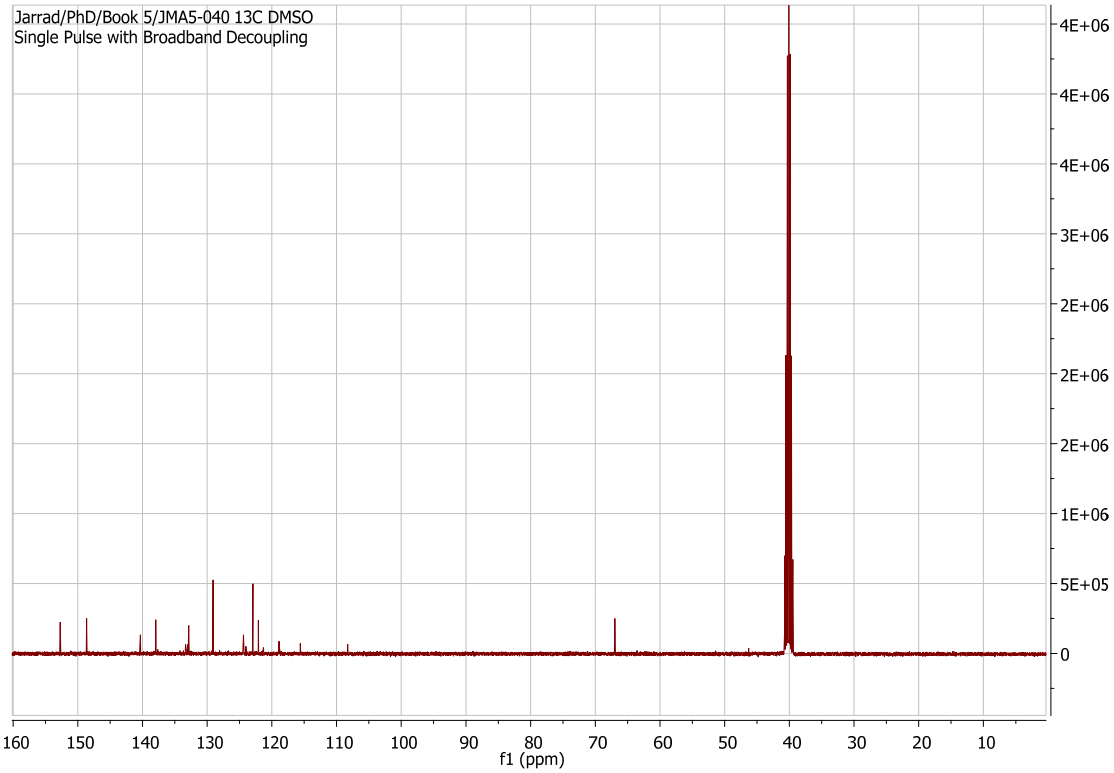
f1 (ppm)



4-(4-((3,5-Dibromophenyl)(hydroxy)methyl)-1*H*-1,2,3-triazol-1-yl)-2-(trifluoromethyl)benzonitrile **6g**

mp 164.6-165.4°C; ^{1}H NMR (d_6-DMSO, 400 MHz): δ 9.06 (1H, s, ArH) 8.56 (1H, s, ArH) 8.50 (1H, d, J = 8 Hz, ArH) 8.41 (1H, d, J = 8 Hz, ArH) 7.76 (1H, s, ArH) 7.68 (1H, s, ArH) 6.53 (1H, d, J = 4 Hz, CH), 6.00 (1H, d, J = 4 Hz, OH) ^{13}C NMR (d_6-DMSO, 100 MHz): δ 152.7, 148.6, 140.3, 139.0, 133.2 (q, $J^{2}_{C\text{-}F}$ = 32 Hz) 132.8, 129.1, 124.4, 122.9, 122.6 (q, $J^{1}_{C\text{-}F}$ = 272 Hz), 122.1, 118.8 (m) 115.6, 67; ^{19}F NMR (d_6-DMSO, 470 MHz): δ 59.1; IR (cm^{-1}): 3381, 3074, 1615, 1467, 1355, 1184, 1143, 998, 666, 50; HRMS calced for $C_{17}H_{10}Br_2F_3N_4O^+$ $[MH]^+$ 500.9168, found 500.9214

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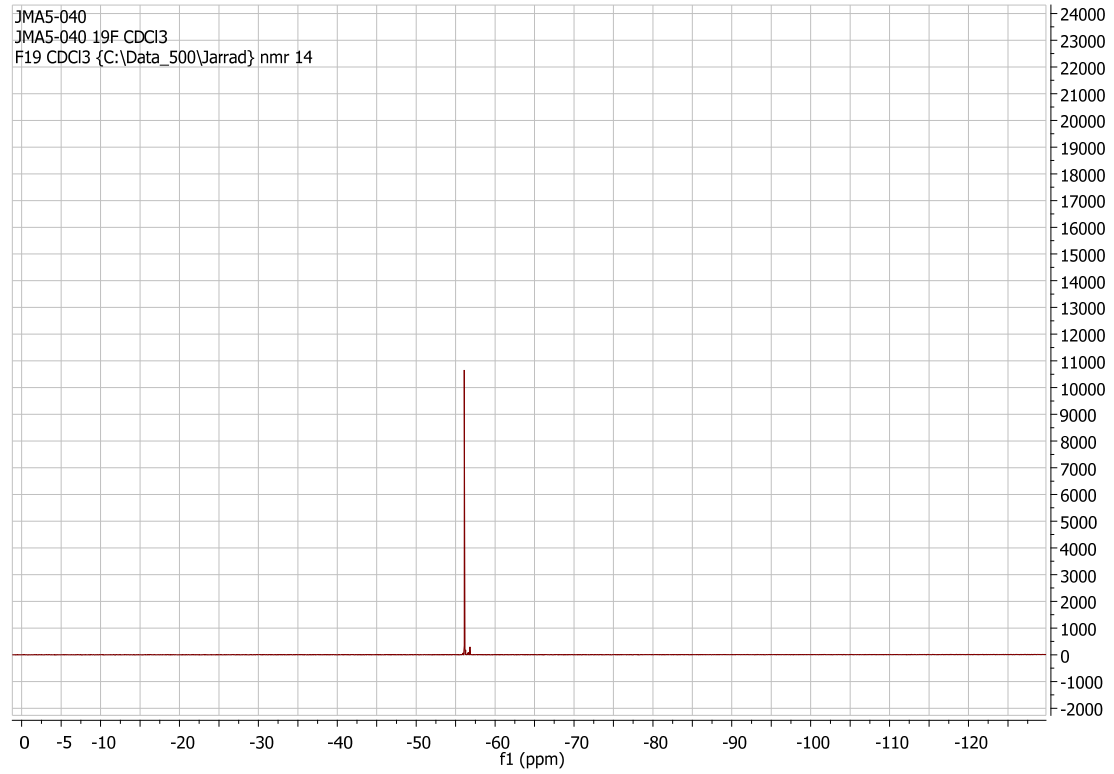
f1 (ppm)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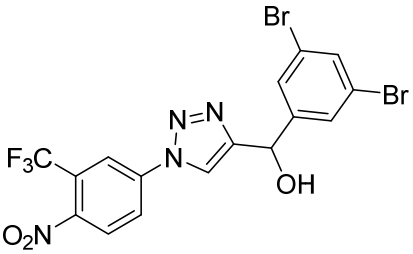
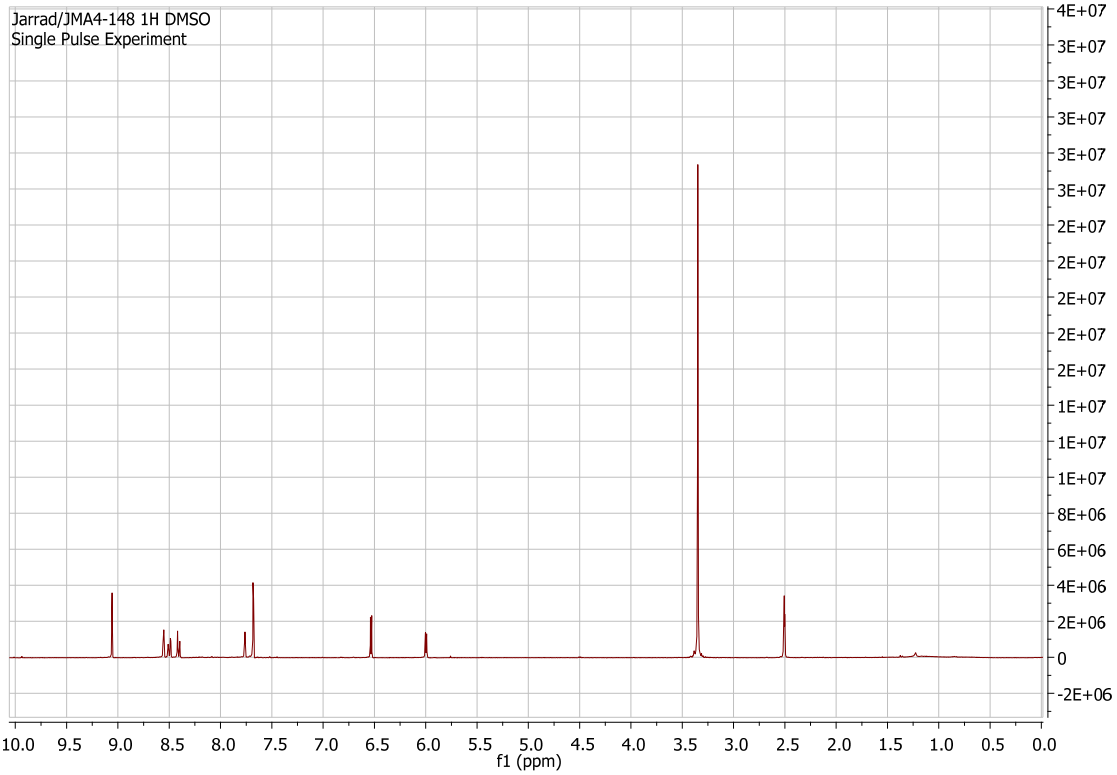
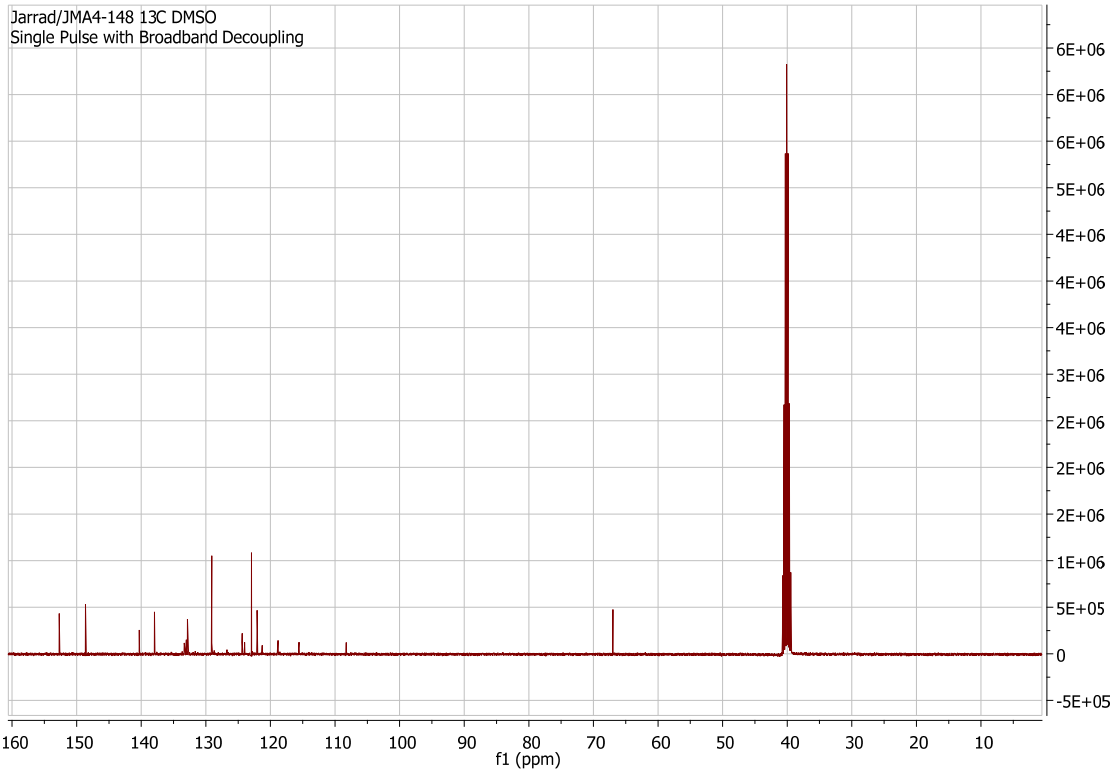
f1 (ppm)



(3,5-Dibromophenyl)(1-(4-nitro-3-(trifluoromethyl)phenyl)-1H-1,2,3-triazol-4-yl)methanol **6h**

mp 167.2-168°C; ^{1}H NMR (d_6-DMSO, 400 MHz): δ 9.06 (1H, s, Ar-H) 8.56 (1H, d, J = 2.7 Hz, Ar-H) 8.51-8.49 (1H, m, Ar-H) 8.41 (1H, d, J = 5.4z, Ar-H) 7.76 (1H, m, Ar-H) 7.68 (2H, m, Ar-H) 6.53 (1H, d, J = 2.7 Hz, CH) 6.00 (1H, d, J = 2.7 Hz, OH); ^{13}C NMR (d_6-DMSO, 100 MHz): δ 152.7, 148.6, 138.0, 133.2 (q, $J^{2}_{C\text{-}F}$ = 33 Hz) 132.8, 129.1, 124.4, 122.9, 122.6 (q, $J^{1}_{C\text{-}F}$ = 272 Hz) 122.1, 118.8, 115.6, 66.98; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -60.8; IR (cm^{-1}): 3449, 2853, 1615, 1585, 1313, 1183, 1143, 1082, 841, 740, HRMS calced for $C_{16}H_{10}Br_2F_3N_4O_3^+$ $[MH]^+$ 520.9066, found 520.9054

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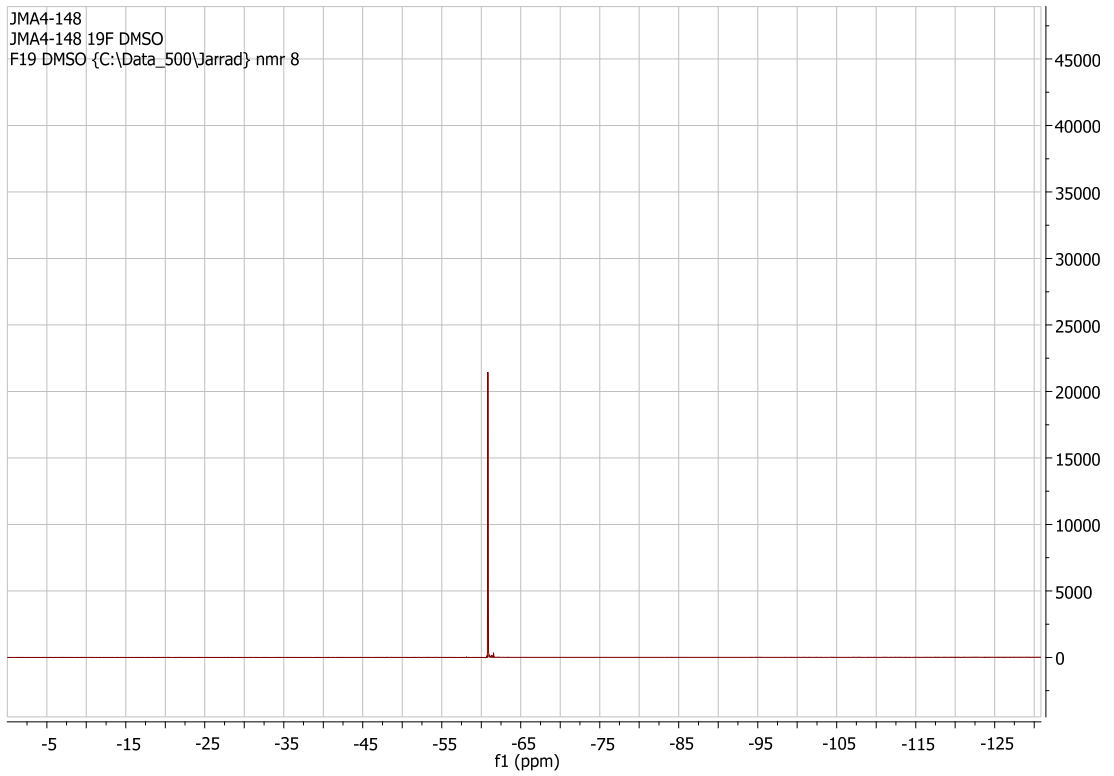
f1 (ppm)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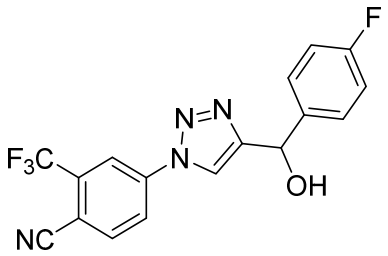
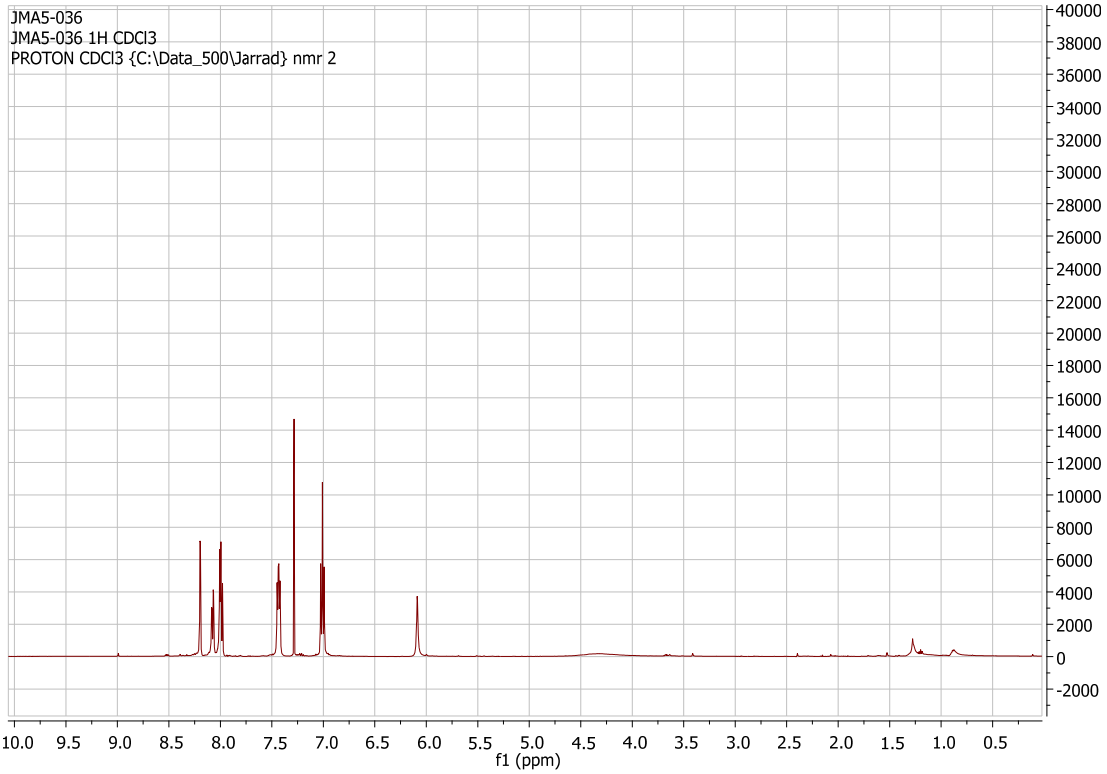
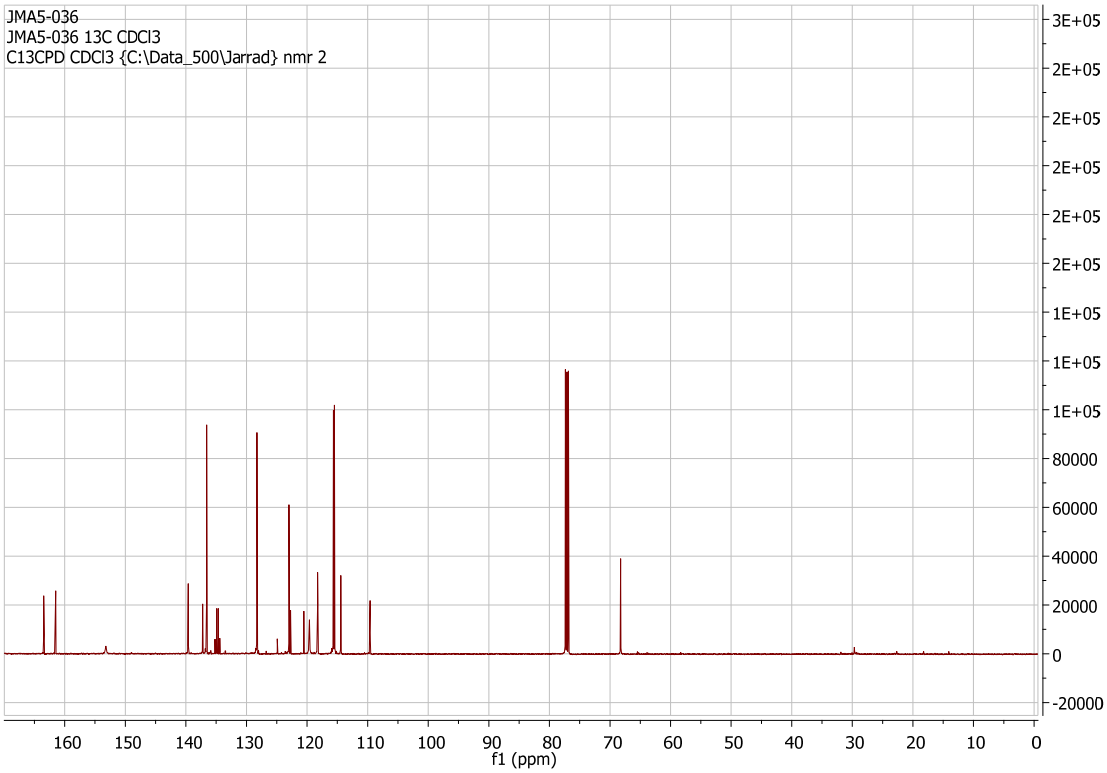
f1 (ppm)



4-(4-((4-Fluorophenyl)(hydroxy)methyl)-1*H*-1,2,3-triazol-1-yl)-2-(trifluoromethyl)benzonitrile **6k**

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.20 (1H, s, ArH) 8.08 (1H, d, J = 10 Hz, ArH) 8.01 (1H, d, J = 5 Hz, ArH) 7.98 (1H, s, ArH) 7.43 (2H, t, J = 10 Hz, ArH) 7.01 (2H, t, J = 10 Hz, ArH) 6.09 (1H, s, CH) No OH observed; ^{13}C NMR ($CDCl_3$, 125 MHz): δ 162.5 (d, $J^{1}_{C\text{-}F}$ = 250 Hz) 153.2, 139.6, 137.2, 136.6, 134.8 (q, $J^{2}_{C\text{-}F}$ = 33.75 Hz) 128.3 (d, $J^{3}_{C\text{-}F}$ = 8.75 Hz) 123.0, 121.6 (q, $J^{1}_{C\text{-}F}$ = 272.5 Hz) 119.6, 118.3 (m) 115.6 (d, $J^{2}_{C\text{-}F}$ = 22.5 Hz) 114.4, 109.6, 68.2 ; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -62.2, -113.6; IR (cm^{-1}): 3403, 3107, 1654, 1504, 1320, 1150, 990, 831; HRMS (ES) calced for $(C_{17}H_{11}F_4N_4O)^+$ 363.0864, found 363.0875

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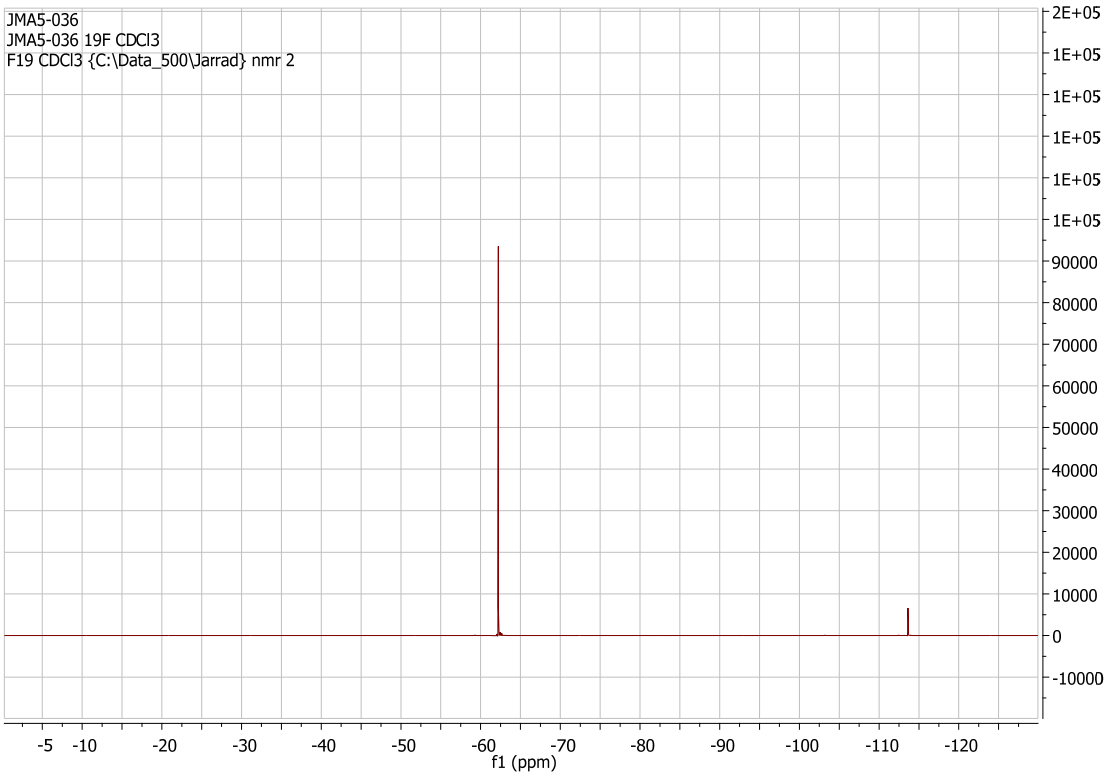
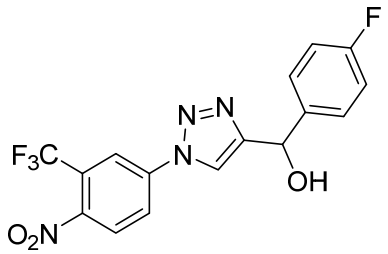
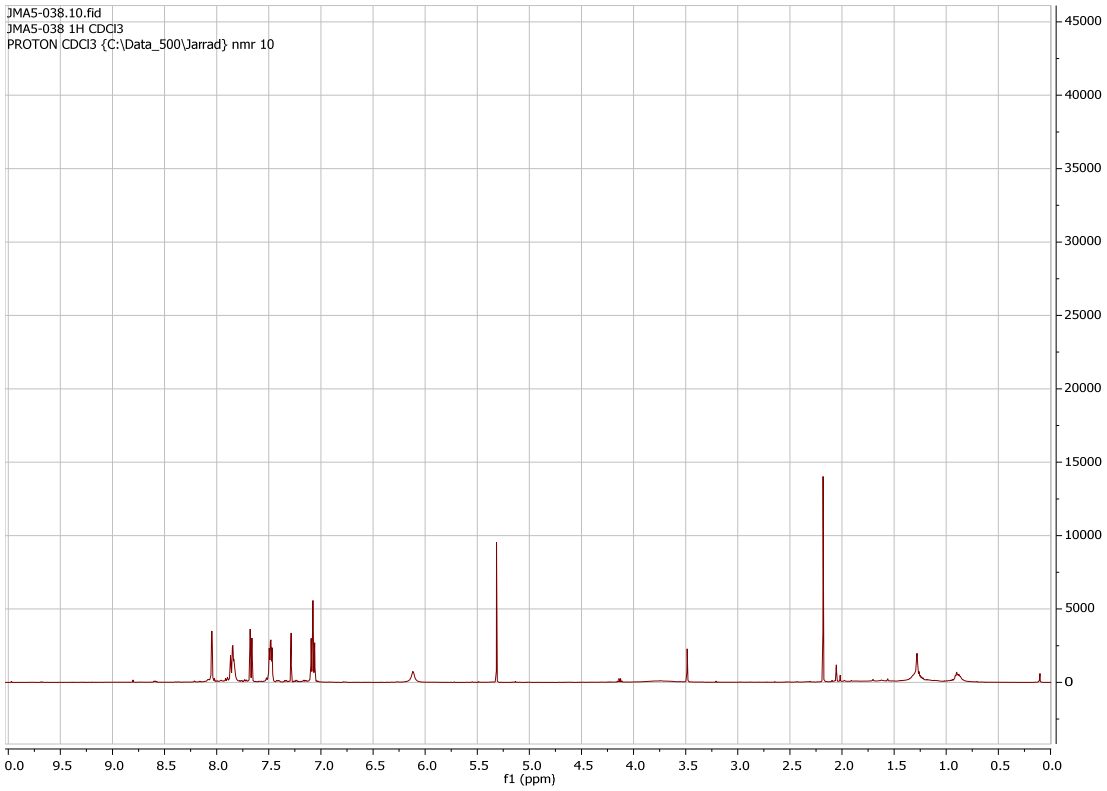
C13CPD CDCl3 {C:\Data_500\Jarrad} nmr 2
3E+05
2E+05
2E+05
2E+05
2E+05
2E+05
1E+05
1E+05
1E+05
80000
60000
40000
20000
0
-20000
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

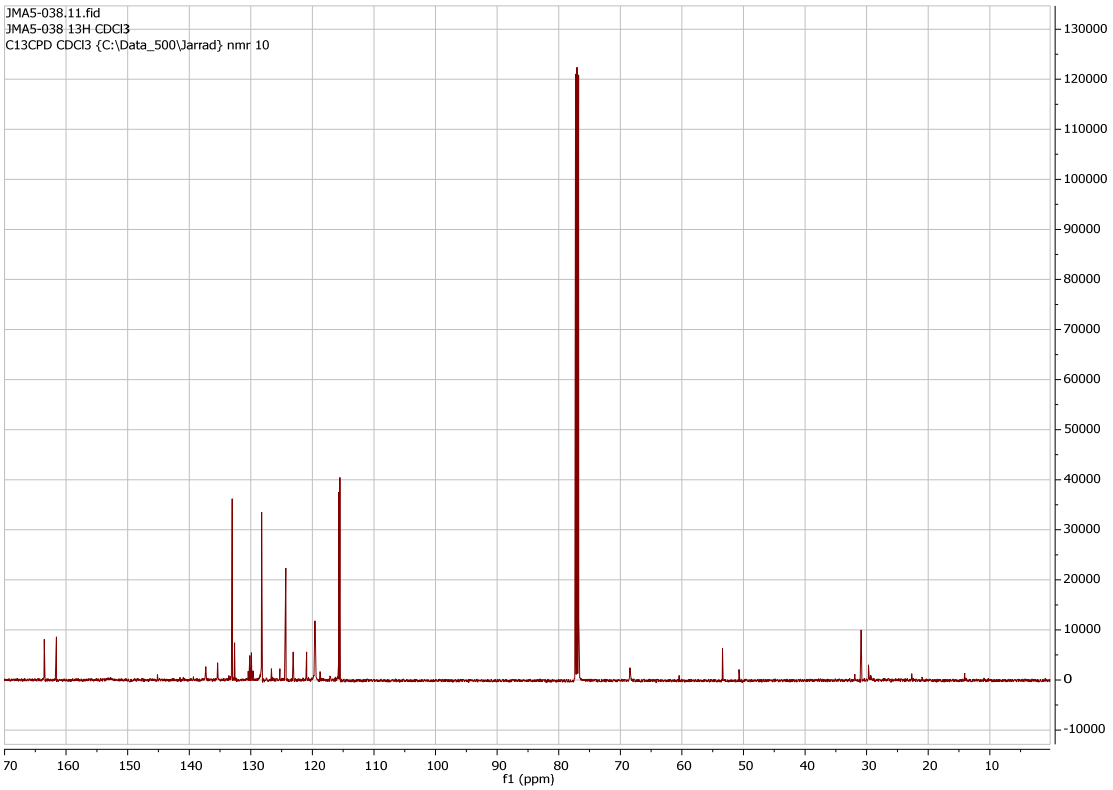

JMA5-036
JMA5-036 19F CDCl3
F19 CDCl3 {C:\Data_500\Jarrad} nmr 2
2E+05
1E+05
1E+05
1E+05
1E+05
1E+05
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-5 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120
f1 (ppm)



(4-Fluorophenyl)(1-(4-nitro-3-(trifluoromethyl)phenyl)-1H-1,2,3-triazol-4-yl)methanol **6l**

^{1}H NMR ($CDCl_3$, 500 MHz): δ 8.04 (1H, s, ArH) 7.87-7.84 (2H, m, ArH) 7.67 (1H, d, J = 10 Hz, ArH) 7.50-7.47 (2H, m, ArH) 7.08 (2H, t, J = 10 Hz, ArH) 6.12 (1H, s, CH) No OH peak observed; ^{13}C NMR ($CDCl_3$, 125 MHz): δ 162.6 (q, $J^{1}_{C\text{-}F}$ = 245 Hz) 137.3, 135.4, 133.0, 132.6, 130.0 (q, $J^{2}_{C\text{-}F}$=32.5 Hz) 128.2 (d, $J^{3}_{C\text{-}F}$=8.75 Hz) 126.7, 124.3, 122.0 (q, $J^{1}_{C\text{-}F}$ = 272.5) 119.6 (m) 115.6 (d, $J^{2}_{C\text{-}F}$ = 21.3 Hz) 53.4; ^{19}F NMR ($CDCl_3$, 470 MHz): δ -63.1,-113.7; IR (cm^{-1}): 3352, 2924, 1604, 1539, 1312, 1226, 1144, 1037, 829, 731; HRMS (ES) calced for $(C_{16}H_{11}F_4N_4O_3)^+$ 383.0762, found 383.0775

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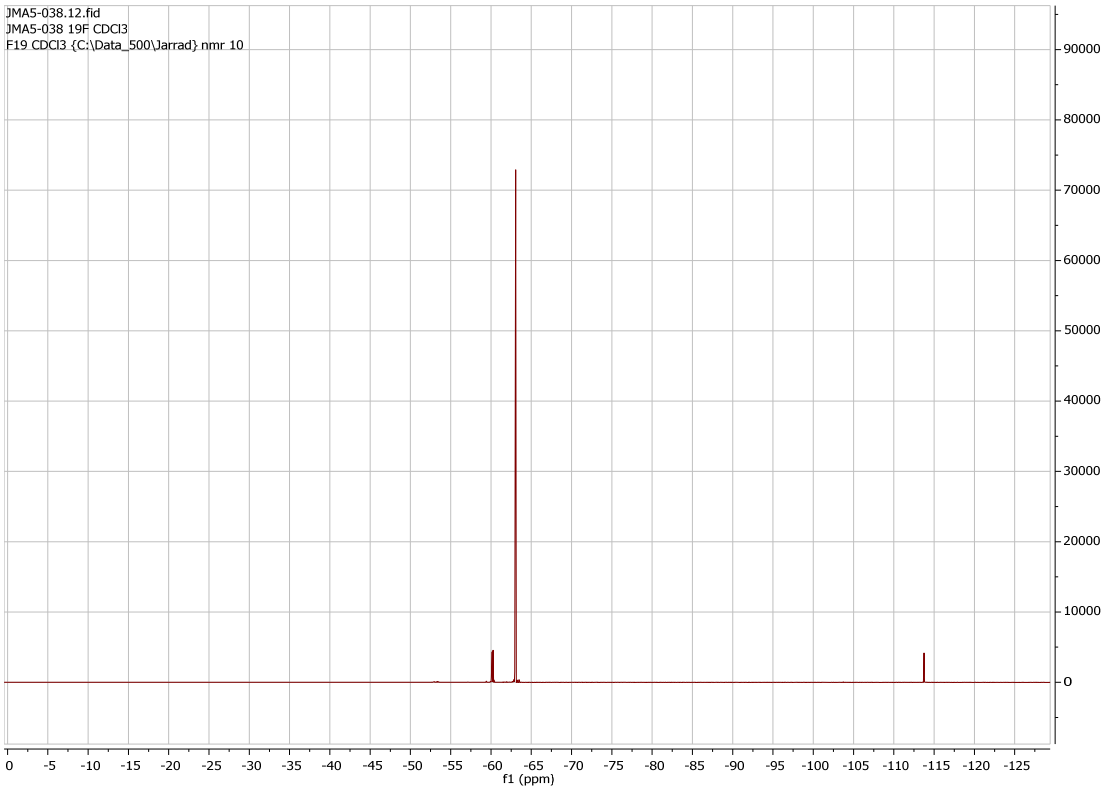
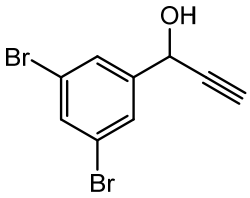
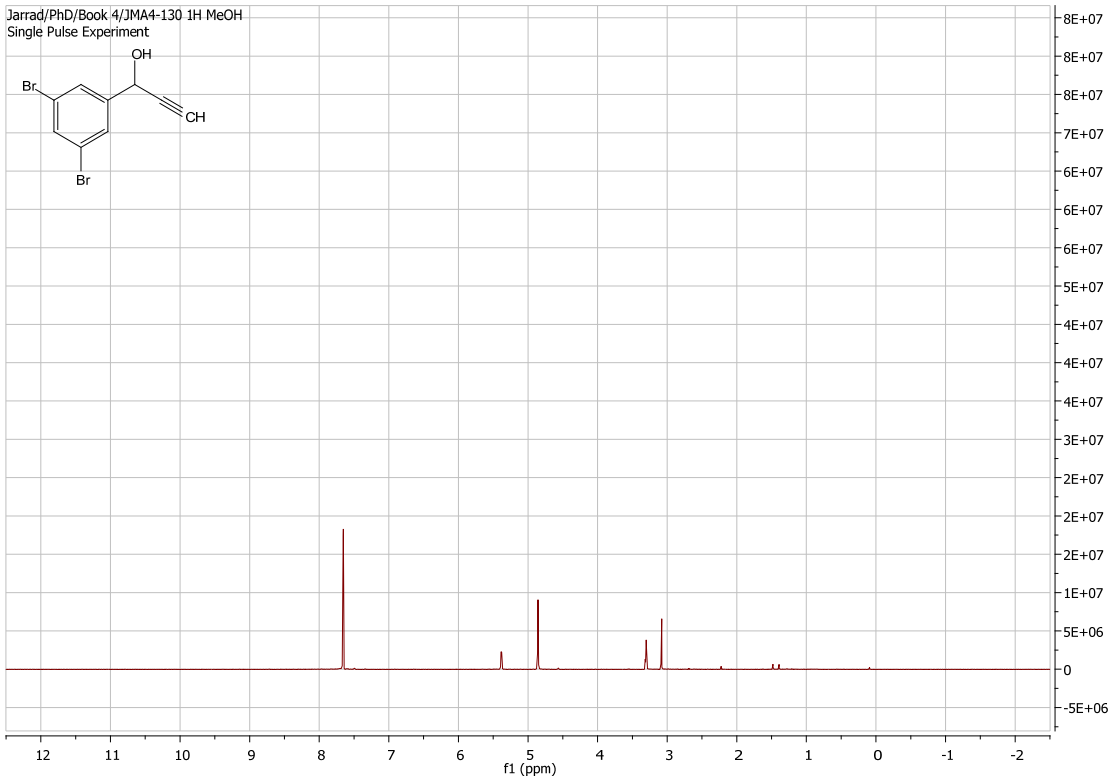
C13CPD CDCl3 {C:\Data_500\Jarrad} nmr 10
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

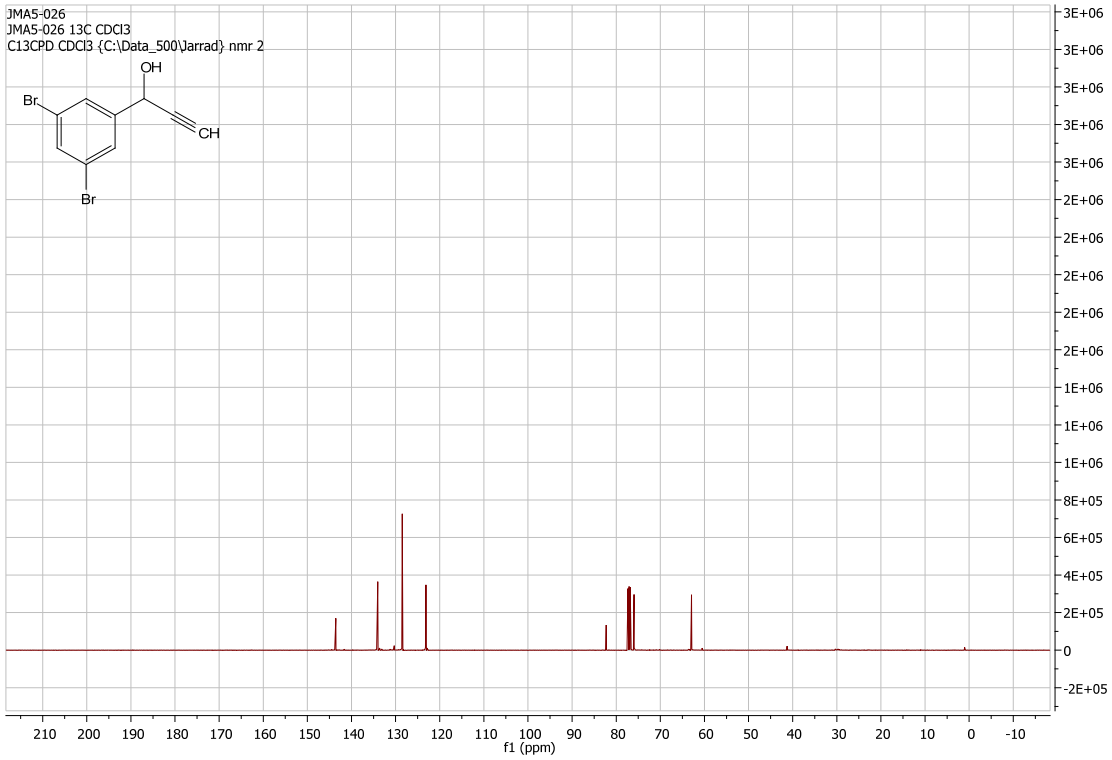

JMA5-038.12.fid
JMA5-038 19F CDCl3
F19 CDCl3 {C:\Data_500\Jarrad} nmr 10
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125
f1 (ppm)

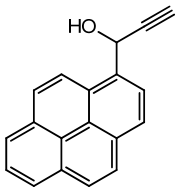


1-(3,5-dibromophenyl)prop-2-yn-1-ol

IR (thin film) υ, cm^{-1} 3844, 3724, 2361, 1694, 1548, 1426, 1029, 708, 406; ^{1}H NMR (d_3-MeOH, 270 MHz): 7.65 (3H, s, Ar-*H*) 5.39 (1H, s, C*H*) 4.86 (1H, s, O*H*) 3.08 (1H, s, C*H*); ^{13}C NMR (*d*-$CDCl_3$, 125 MHz): δ 143.6, 134.0, 128.5, 123.1, 82.3, 76.0, 62.9; m.p. 68.5 °C; HRMS calced for $C_9H_7Br_2O$ $[MH]^+$ 288.8858, found 288.8832.

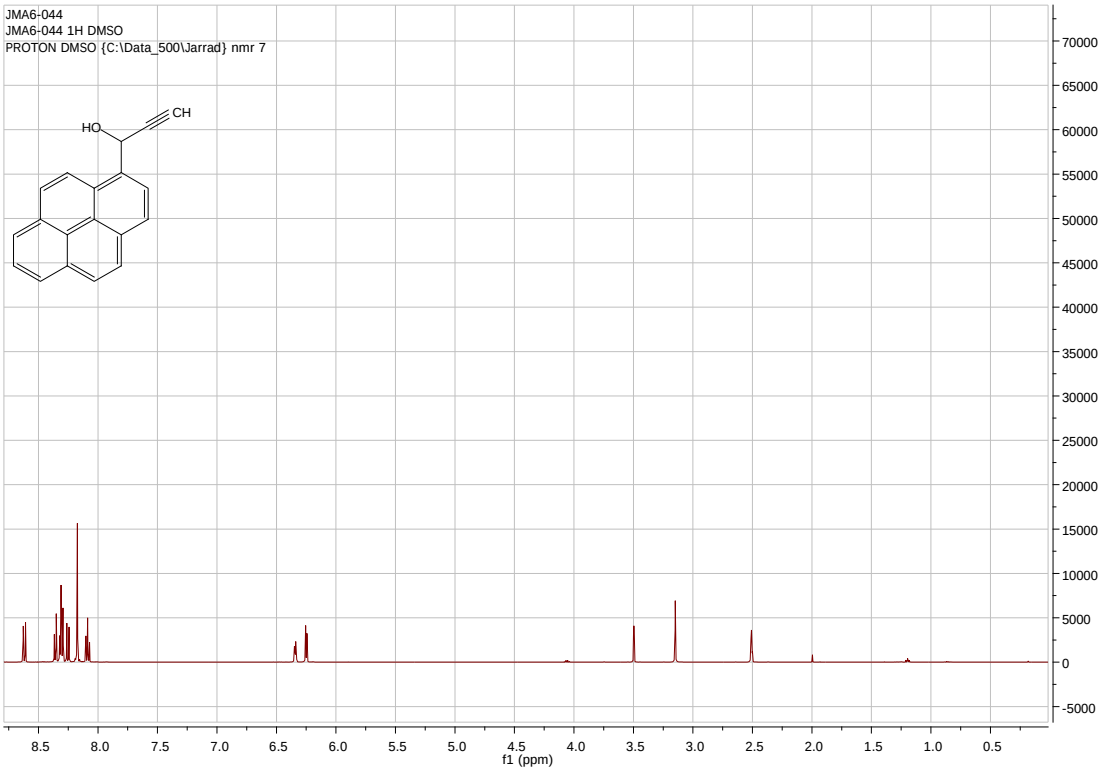






1-(pyren-2-yl)prop-2-yn-1-ol

IR (thin film) υ, cm^{-1} 3870, 3750, 3566, 2364, 1699, 1418, 1027, 740, 707; ^{1}H NMR (d_6-DMSO, 500 MHz): 8.62 (1H, d, J= 10 Hz, Ar-*H*) 8.36 (1H, d, J= 10 Hz, Ar-*H*) 8.31 (3H, apparent t, J= 5 Hz, Ar-*H*) 8.25 (1H, d, J= 10 Hz, Ar-*H*) 8.17 (2H, apparent s, Ar-*H*) 8.09 (1H, t, J= 5 Hz, Ar-*H*) 6.34 (1H, dd, J= 5, 3 Hz, C*H*) 6.25 (1H, d, J= 5.5 Hz, O*H*) 3.50 (1H, s, C*H*); ^{13}C NMR (d_6-DMSO, 125 MHz): δ 135.6, 131.3, 131.1, 130.7, 127.9, 127.7, 127.7, 127.3, 126.7, 125.8, 125.7, 125.1, 124.7, 124.5, 124.2, 86.1, 76.9, 61.5; m.p. 156.7 °C; HRMS predicted $C_{19}H_{13}O^{+}$ $[MH]^{+}$ 257.0961, found 257.0994.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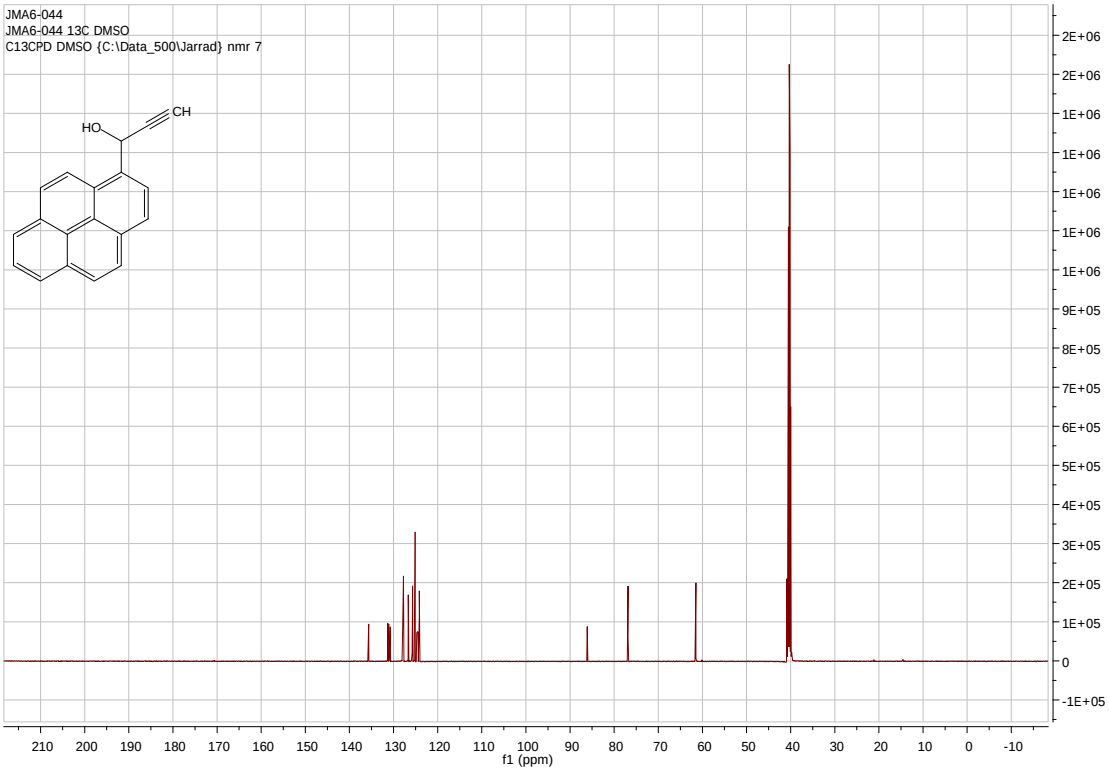
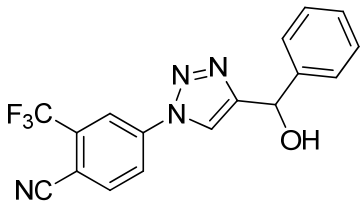
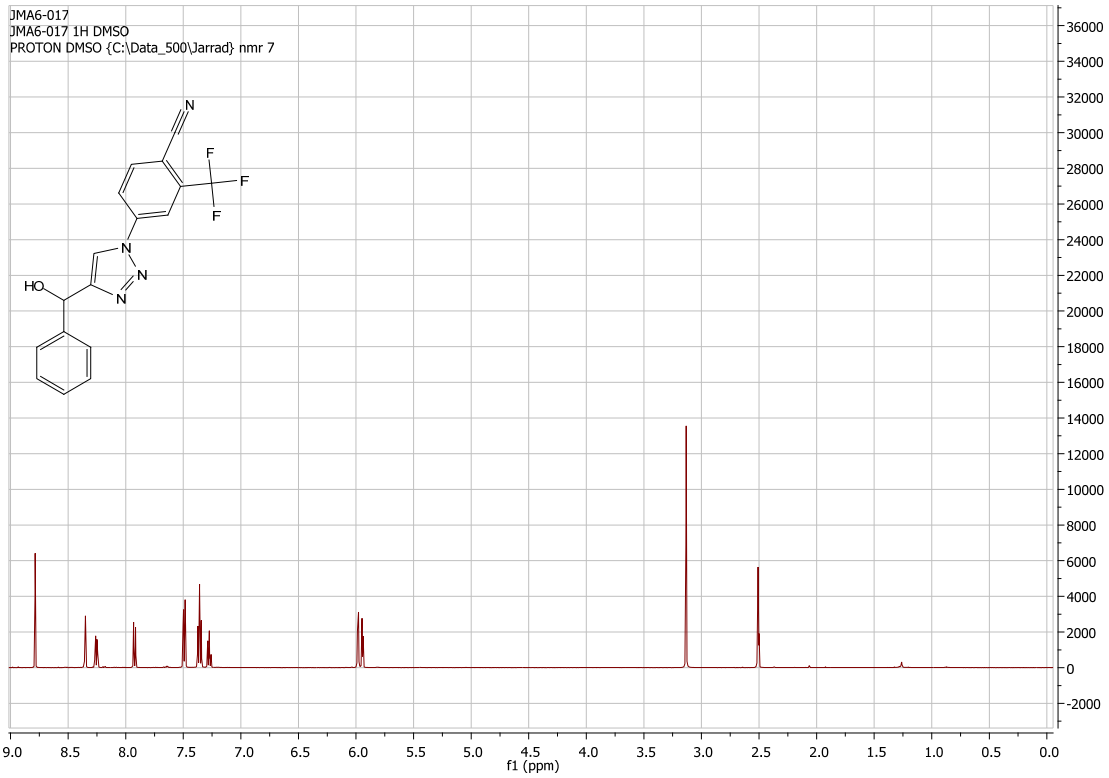
JMA6-044 1H DMSO
PROTON DMSO {C:\Data_500\Jarrad} nmr 7
HO
CH
f1 (ppm)

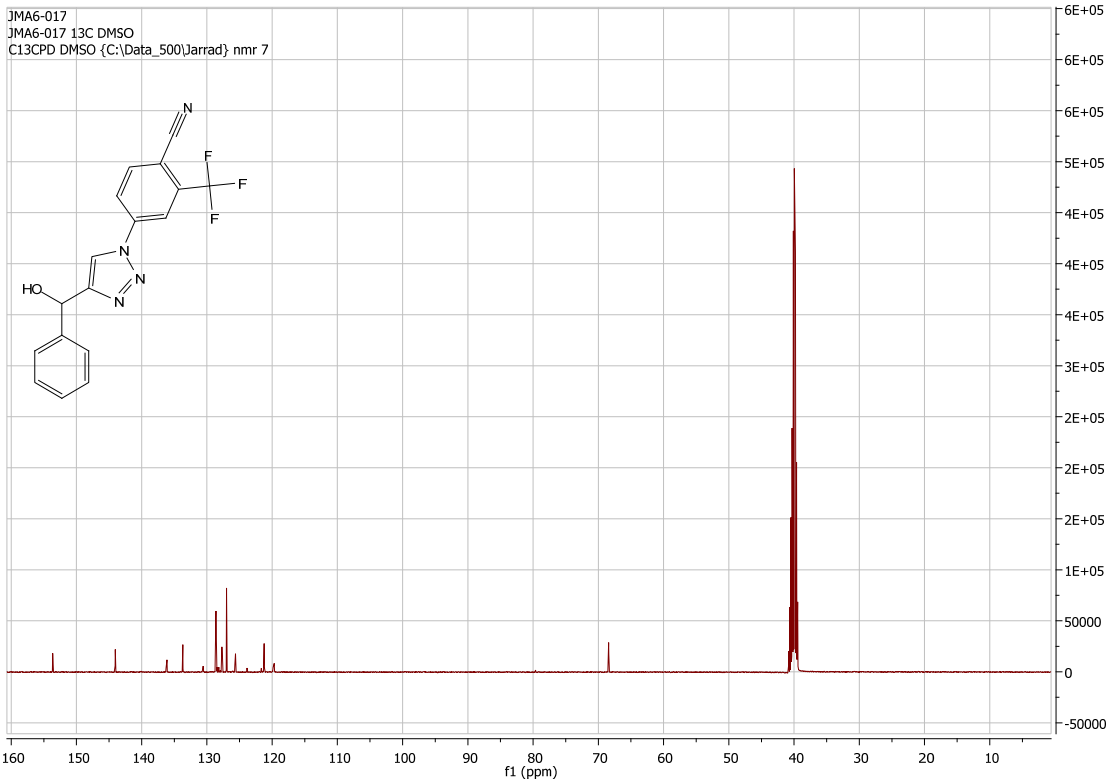

JMA6-044
JMA6-044 13C DMSO
C13CPD DMSO {C:\Data_500\Jarrad} nmr 7
HO
CH
f1 (ppm)

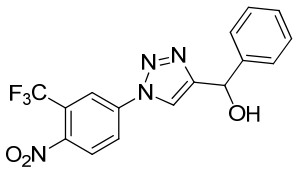


4-(4-[hydroxy(phenyl)methyl]-1H-1,2,3-triazol-1-yl}-2-(trifluoromethyl)benzonitrile 6a

IR (thin film) υ, cm^{-1}xxx; ^{1}H NMR (d_6-DMSO, 500 MHz): 8.79 (1H, s, Ar-*H*) 8.35 (1H, d, J= 2.5, Ar-*H*) 8.26 (1H,) 7.92 (1H, d, J= 8.5 Hz, Ar-*H*) 7.49 (2H, d, J= 7.5 Hz, Ar-*H*) 7.36 (2H, apparent t, J= 7 Hz, Ar-*H*) 7.29-7.25 (1H, m, Ar-*H*) 5.98 (1H, d, J= 4.5 Hz, O*H*) 5.94 (1H, d, 4.5, C*H*); ^{13}C NMR (d_6-DMSO, 125 MHz): δ 136.3, 136.11, 133.7, 133.7, 128.6, 128.2, 128.1 (q, J= 255, 102.5 Hz) 127.6, 127.0, 125.7, 125.6; HRMS predicted for ($C_{17}H_{12}F_3N_4O$) $[MH]^+$ 345.0958, found 345.0942.

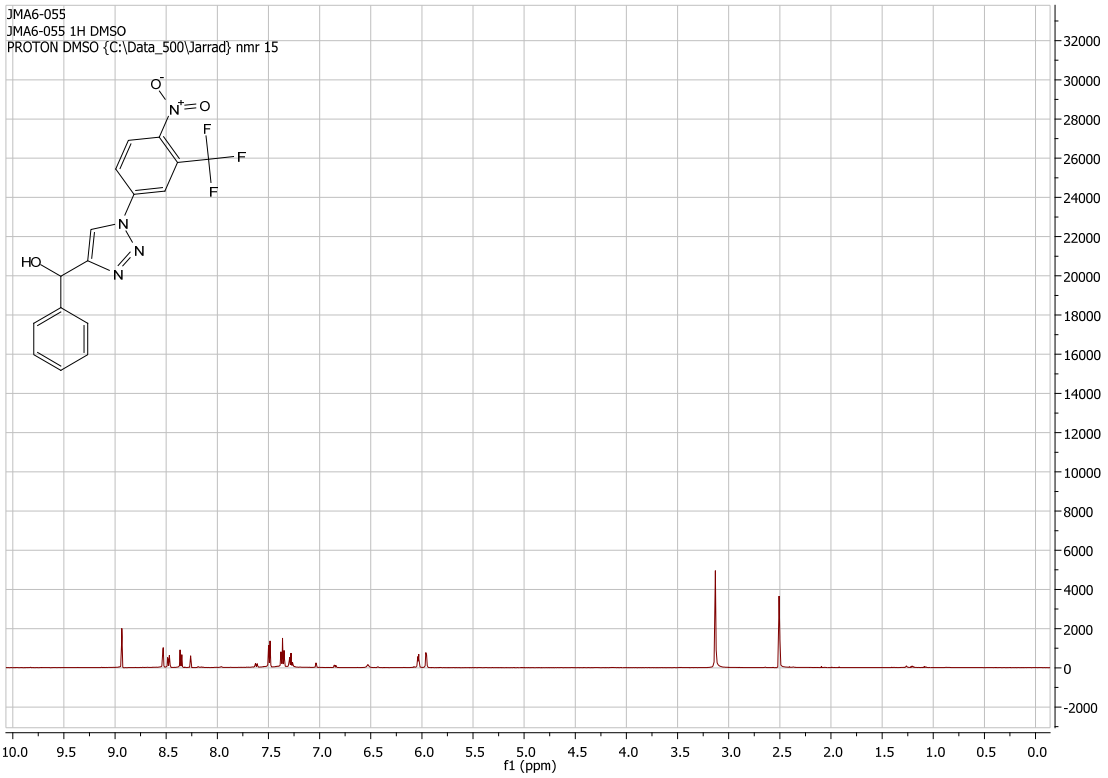






{1-[4-nitro-3-(trifluoromethyl)phenyl]-1H-1,2,3-triazol-4-yl}(pyren-1-yl)methanol 6b

IR (thin film) υ, cm^{-1}3368, 2822, 2233, 1613, 1444, 1312, 1180, 1083, 995, 844, 638, 480; ^{1}H NMR (d_6-DMSO, 500 MHz): 8.93 (1H, s, Ar-*H*) 8.53 (1H, d, J = 1.5 Hz, Ar-*H*) 8.48 (1H, dd, J = 2, 8.5 Hz, Ar-*H*) 8.36 (1H, d, J = 8.5, Ar-*H*) 7.50 (2H, d, J = 7.5 Hz, Ar-*H*) 7.36 (2H, t, J = 7.5 Hz, Ar-*H*) 7.28 (1H, t, J = 7.5 Hz, Ar-*H*) 6.04 (1H, d, J = 4.5 Hz, O*H*) 5.96 (1H, d, J = 4 Hz, C*H*); ^{13}C NMR (d_6-DMSO, 125 MHz): δ 153.9, 143.9, 140.4, 137.8, 136.7, 133.2 (q, $J^{3}_{C\text{-}F}$ = 32.5 Hz) 128.6, 127.7, 127.0, 124.3, 118.7 (q, J^{4}= 3.75 Hz) 68.5; HRMS predicted for $C_{16}H_{12}F_3N_4O_3^+$ $[MH]^+$ 365.0856, found 365.0845.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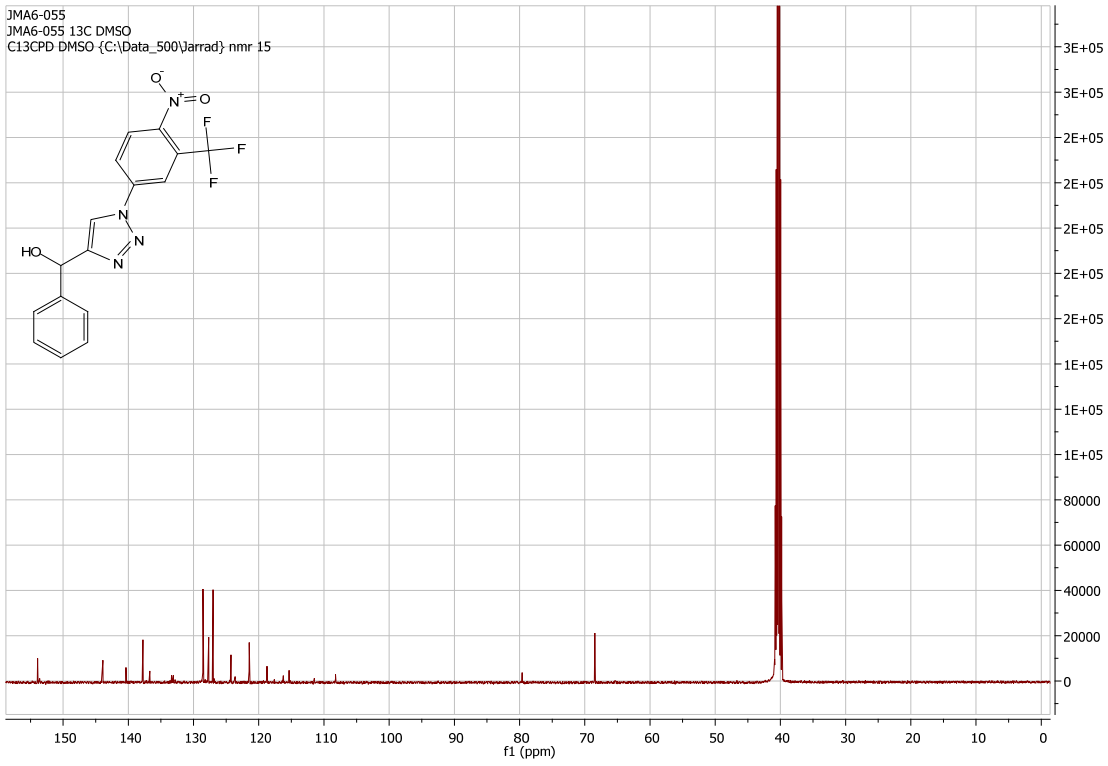
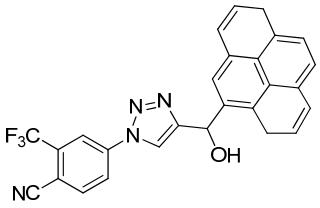
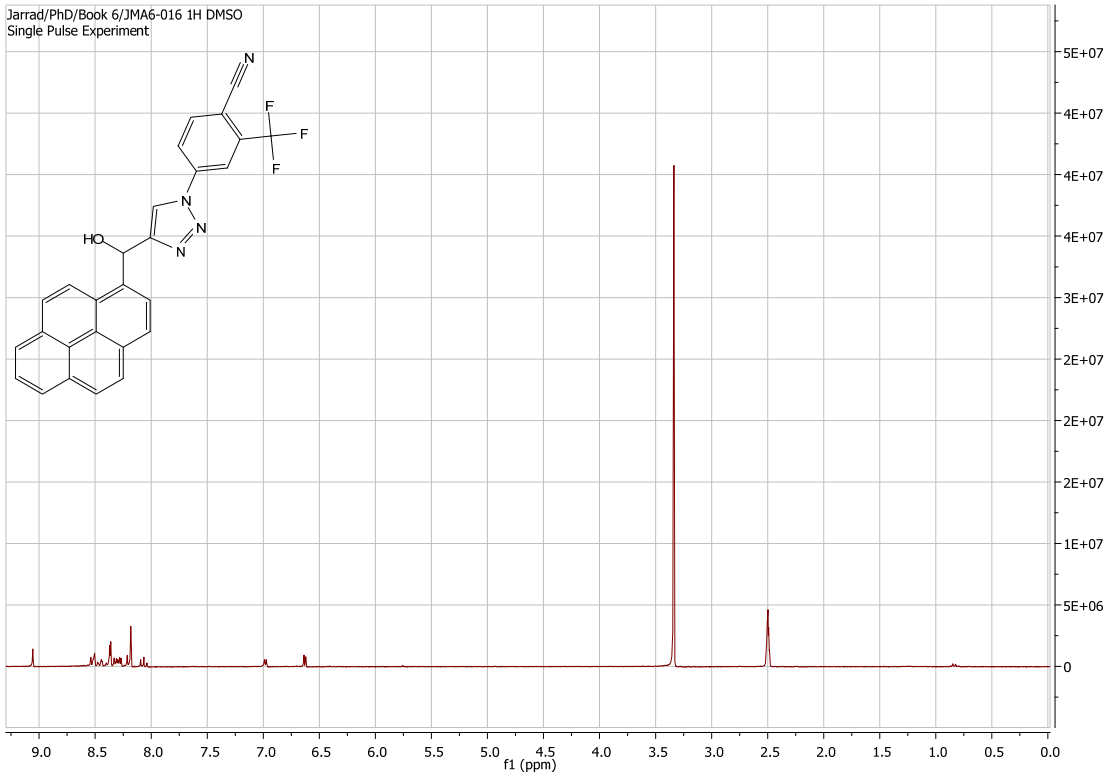
JMA6-055 1H DMSO
PROTON DMSO {C:\Data_500\Jarrad} nmr 15
f1 (ppm)

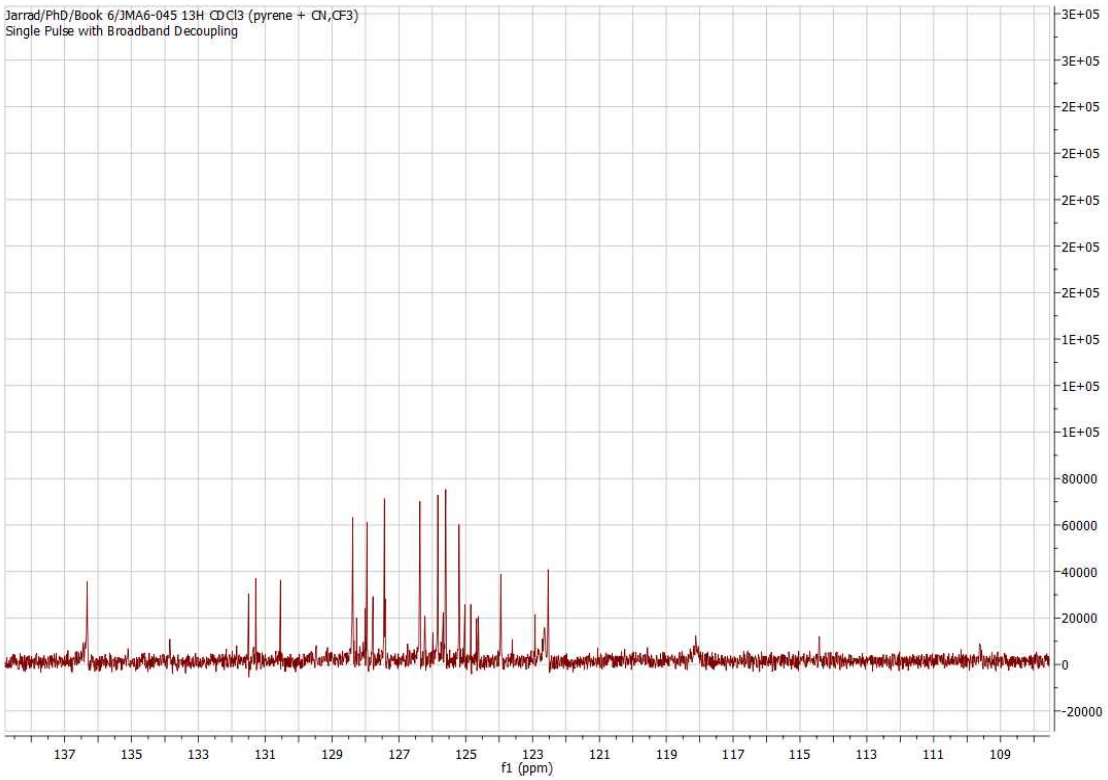

JMA6-055
JMA6-055 13C DMSO
C13CPD DMSO {C:\Data_500\Jarrad} nmr 15
f1 (ppm)



4-{4-[hydroxy(pyren-1-yl)methyl]-1H-1,2,3-triazol-1-yl}-2-(trifluoromethyl)benzonitrile 6c

Brown Solid; IR (thin film) υ, cm^{-1} 3902, 3801, 2364, 1792, 1653, 1396, 1026, 740, 717, 444; ^{1}H NMR (d_6-DMSO, 270 MHz): δ 9.06 (1H, s, Ar-*H*) 8.52 (2H, m, Ar-*H*) 8.46 (1H, d, J = 8.6, Ar-*H*) 8.37 (2H, d, J = 3Hz, Ar-*H*) 8.30 (3H, m, Ar-*H*) 8.21 (1H, s, Ar-*H*) 8.18 (2H, s, Ar-*H*) 8.07 (1H, t, J = 7.8 Hz, Ar-*H*) 6.98 (1H, d, J = 4.6 Hz, O*H*) 6.63 (1H, d, J = 4.6, C*H*); ^{13}C NMR (d-$CDCl_3$, 67.5 MHz): δ 136.3, 131.5, 131.3, 130.5, 128.4, 128.0, 127.9, 127.4, 126.4, 125.8, 125.7, 125.6, 125.2, 125.0, 124.9, 124.7, 124.6, 123.9, 122.9, 122.6, 122.5, 118.1, 62.9; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -60.85; m.p. 135.4 °C; HRMS predicted for ($C_{27}H_{18}F_3N_4O$) $[MH]^+$ 471.1427, found 471.1458.




Jarrad/PhD/Book 6/JMA6-045 13H CDCl3 (pyrene + CN,CF3)
Single Pulse with Broadband Decoupling
3E+05
3E+05
2E+05
2E+05
2E+05
2E+05
2E+05
1E+05
1E+05
1E+05
80000
60000
40000
20000
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-20000
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135
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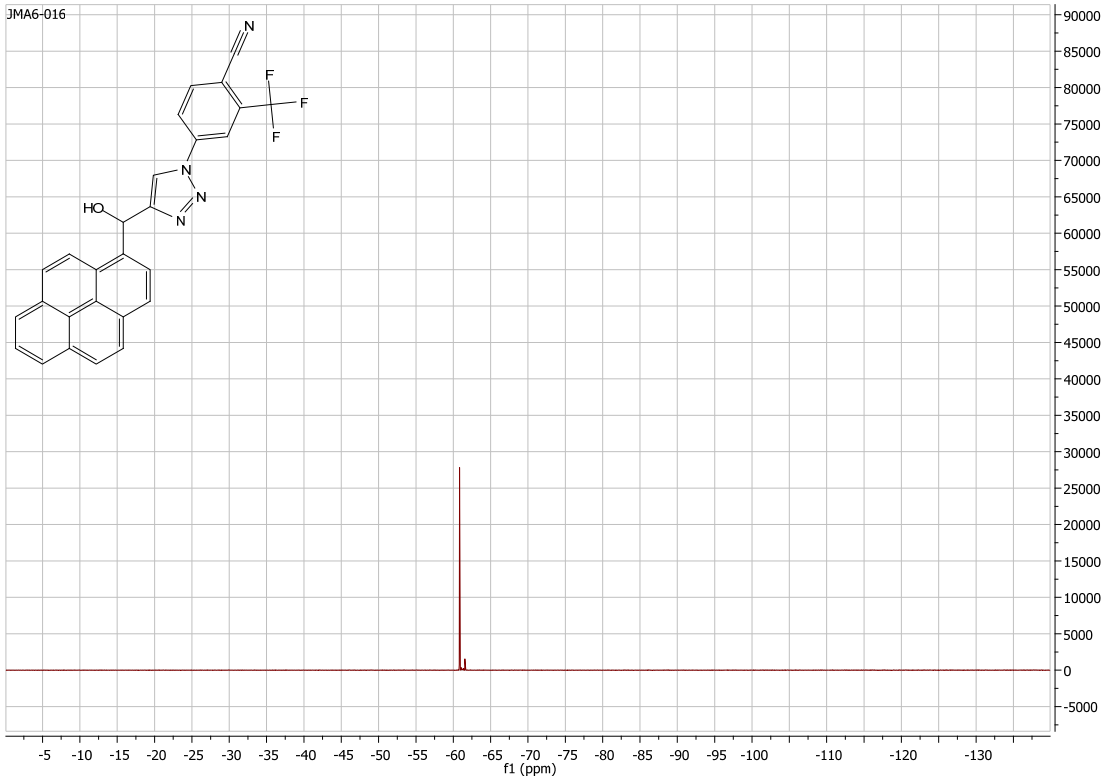
131
129
127
125
123
121
119
117
115
113
111
109
f1 (ppm)


JMA6-016
N
F
F
F
N
N
N
HO
90000
85000
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
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-75
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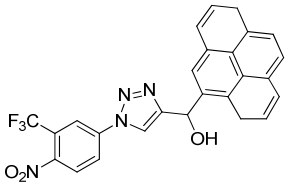
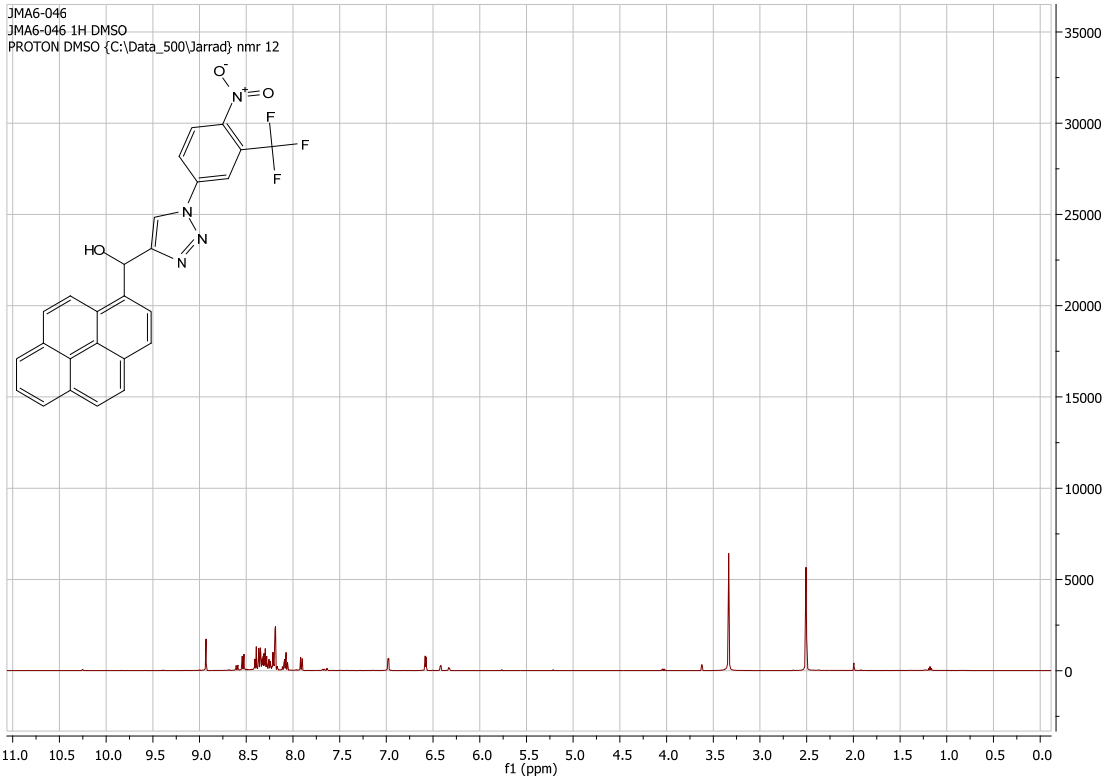
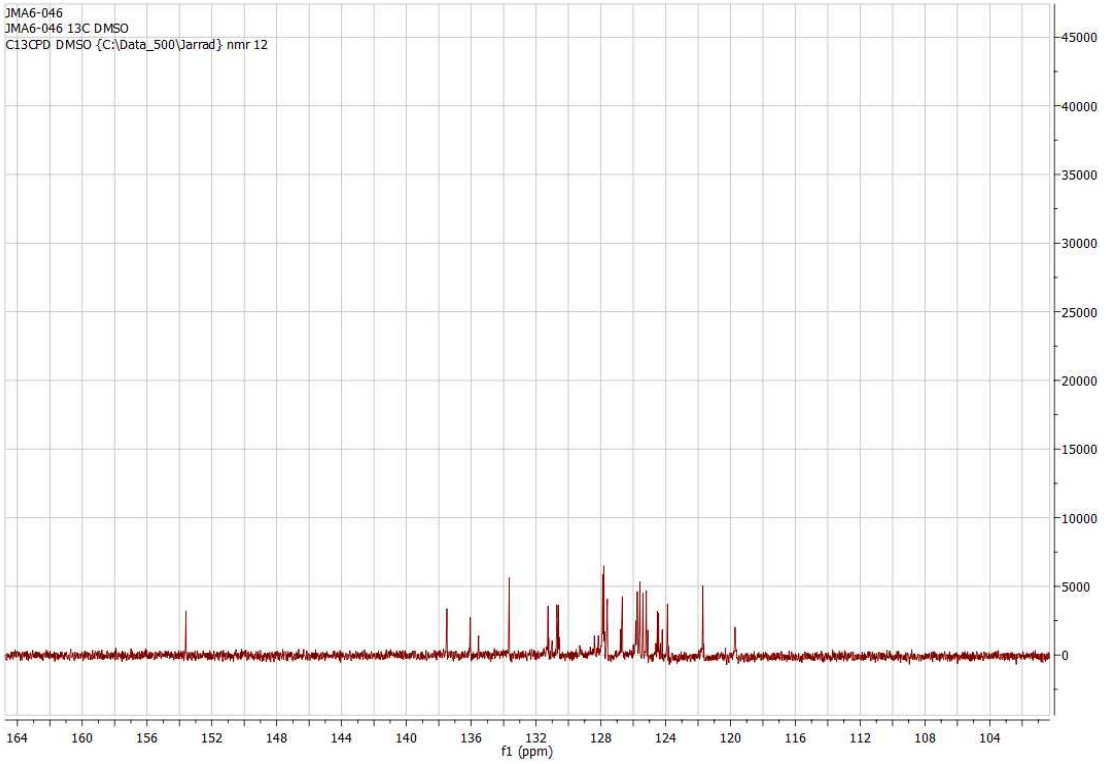
-120
-130
f1 (ppm)



{1-[4-Nitro-3-(trifluoromethyl)phenyl]-1*H*-1,2,3- triazol-4-yl}(pyren-1-yl)methanol 6d

Tan Solid; IR (thin film) υ, cm^{-1} 3902, 3801, 3566, 2342, 1868, 1733, 1540, 1396, 1060, 1027, 743, 703, 419; ^{1}H NMR (d_6-DMSO, 500 MHz): δ 8.93 (1H, s, Ar-*H*) 8.53 (1H, d, J = 9 Hz, Ar-*H*) 8.41-8.17 (11H, m, Ar-*H*) 8.12-8.06 (1H, m, Ar-*H*) 7.91 (1H, d, J = 8.5 Hz, Ar-*H*) 6.98 (1H, d, J = 4.5 Hz, O*H*) 6.58 (1H, d, J = 4.5 Hz, C*H*); ^{13}C NMR (d_6-DMSO, 125 MHz): δ 153.6, 137.5, 136.1, 135.4, 133.6, 131.3, 130.7, 130.6, 128.4, 127.9, 127.8, 127.64, 127.60, 126.8, 126.7, 125.8, 125.75, 125.6, 125.4, 125.2, 125.1, 124.5, 124.4, 124.2, 123.9, 121.7, 119.7, 65.9; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.49; m.p. 182.8 °C; m.p. 182.8 °C; HRMS predicted for ($C_{26}H_{18}F_3N_4O_3$) $[MH]^+$ 491.1326, found 491.1358. Note: due to the complexity in the ^{13}C NMR arising from the CF_3 group and the pyrene resolution of the C-F multiplets was not possible.

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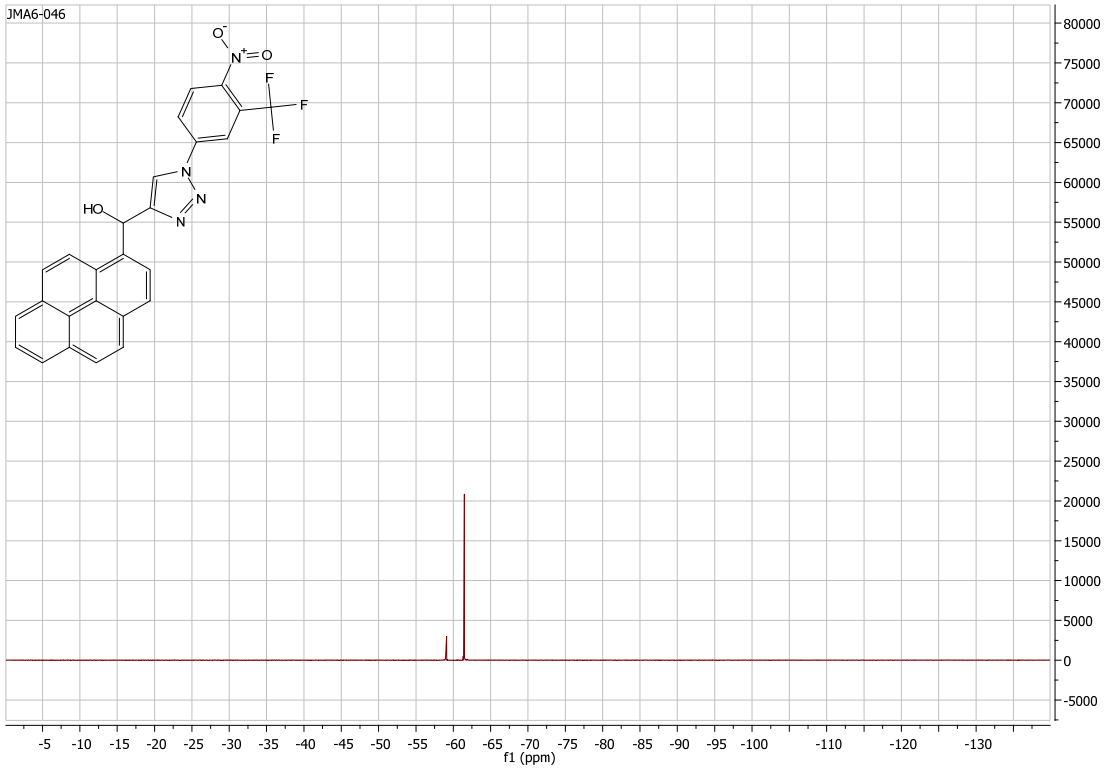
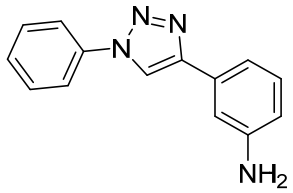
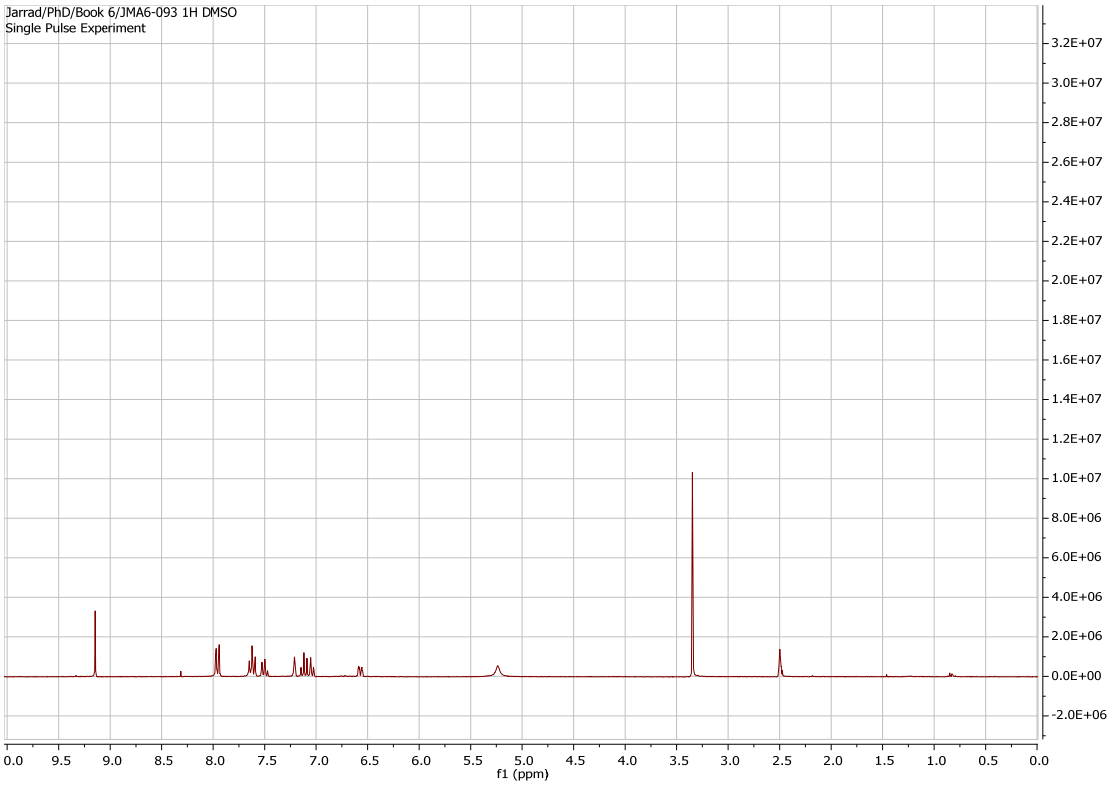
f1 (ppm)

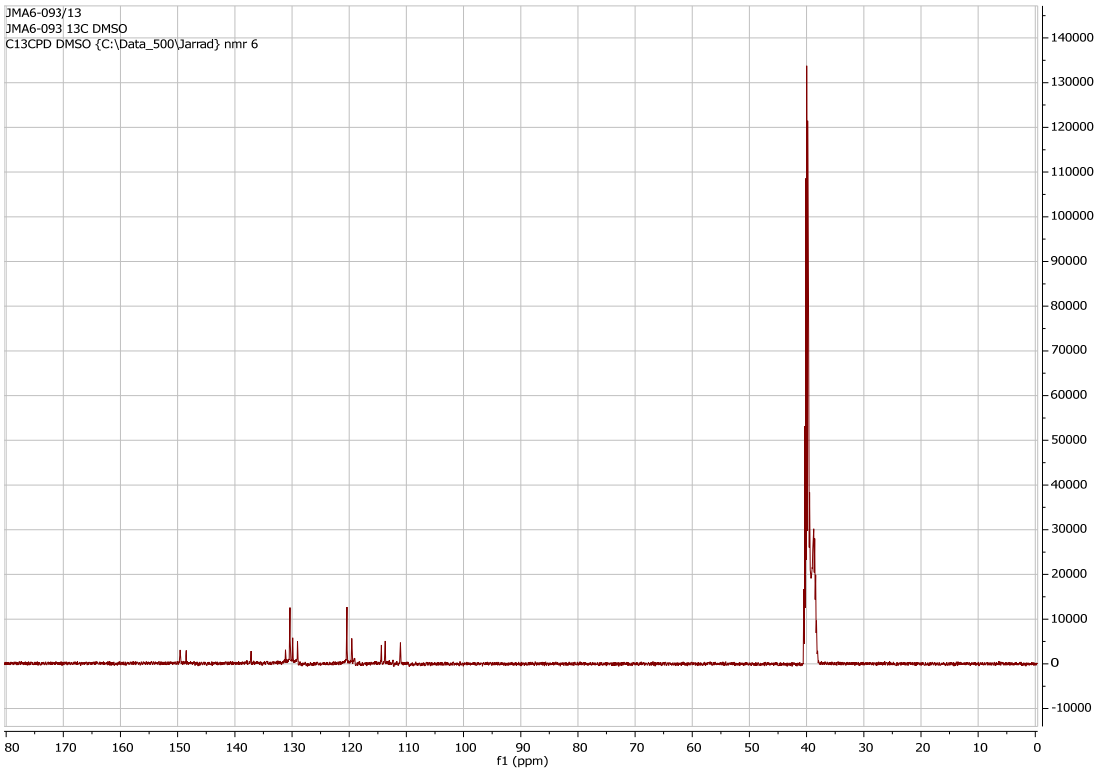

JMA6-046
HO
f1 (ppm)



3-(1-phenyl-1H-1,2,3-triazol-4-yl)aniline 13d

IR (thin film) υ, cm^{-1} 3344, 2969, 2931, 2360, 1378, 1108, 816, 624; mp 121.4 °C; ^{1}H NMR (DMSO-d_6, 270 MHz) δ 9.15 (1H, s, Ar-*H*) 7.94-7.98 (2H, m, Ar-*H*) 7.59-7.65 (2H, m, Ar-*H*) 7.47-7.53 (1H, m, Ar-*H*) 7.21 (1H, t, J = 2.7 Hz, Ar-*H*) 7.03-7.15 (2H, m, Ar-*H*) 6.55-6.59 (1H, m, N*H*); ^{13}C NMR (DMSO-d_6, 67.5 MHz) 149.5, 148.5, 137.2, 131.1, 130.4, 129.9, 129.0, 120.4, 119.6, 114.4, 113.7, 111.0; HRMS calced for $C_{14}H_{12}N_4$ $[MH]^+$ 237.11347, found 237.11310.

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130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
80
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160
150
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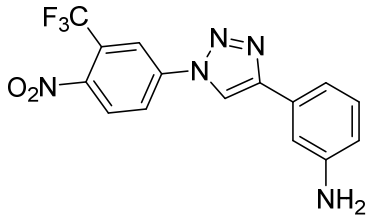
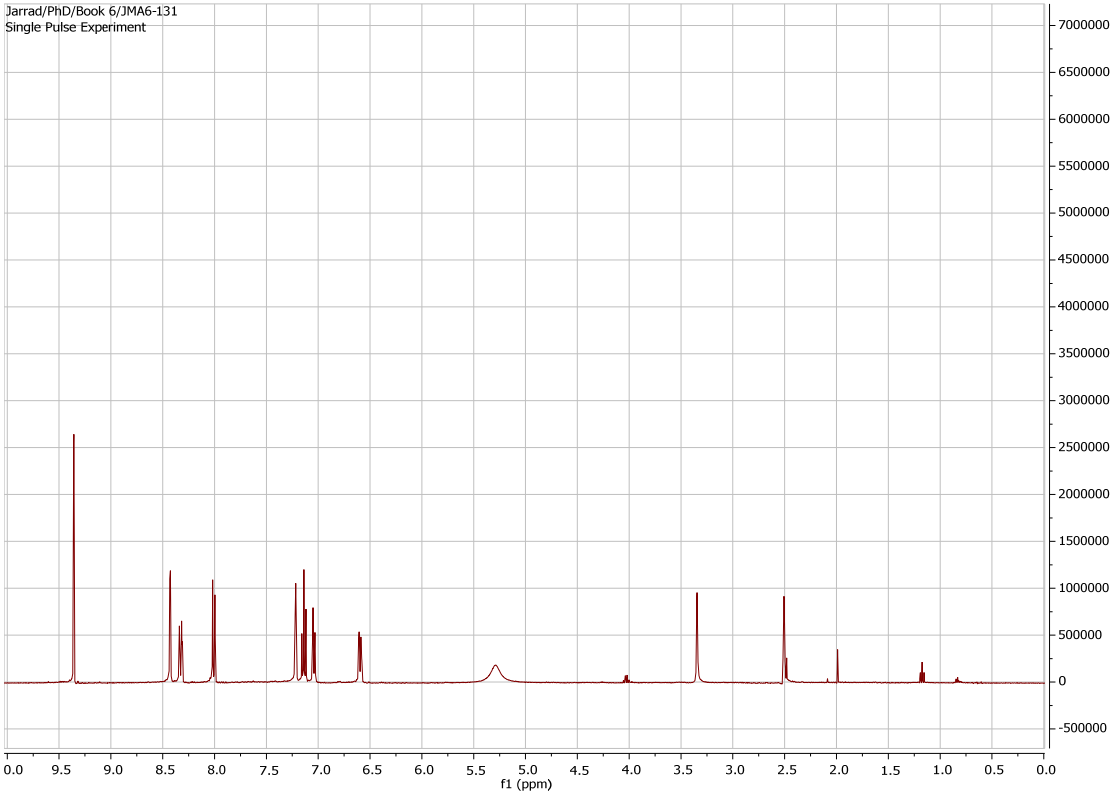
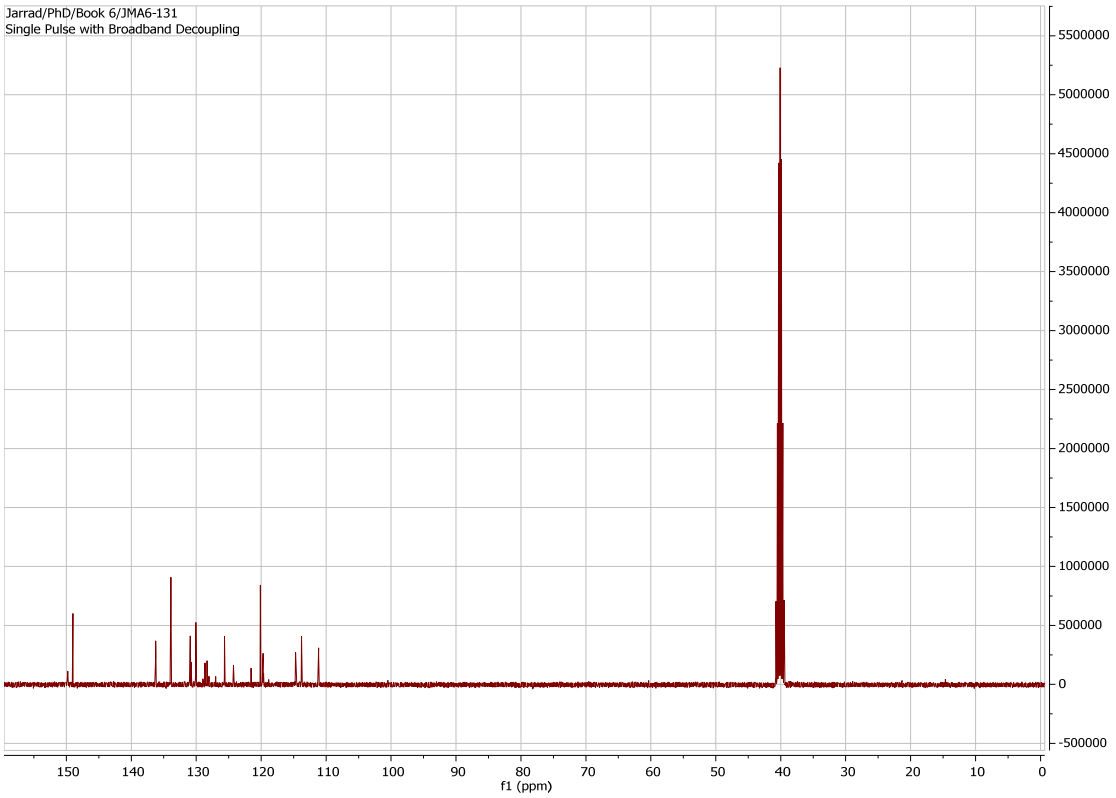
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



3-{1-[4-nitro-3-(trifluoromethyl)phenyl]-1H-1,2,3-triazol-4-yl}aniline 13l

IR (thin film) υ, cm^{-1} 3349, 2333, 1591, 1492, 1260, 1077, 749; mp 183.4 °C; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 9.36 (1H, s, Ar-*H*) 8.43 (1H, s, Ar-*H*) 8.33 (1H, d, J=5 Hz, Ar-*H*) 8.01 (1H, d, J = 5 Hz, Ar-*H*) 7.22 (1H, s, Ar-*H*) 7.14 (1H, t, J = 10 Hz, Ar-*H*) 7.05 (1H, d, J=5 Hz, Ar-*H*) 6.58-6.61 (1H, m, Ar-*H*) 5.29 (2H, br-s, NH_2); ^{13}C NMR (DMSO-d_6, 125 MHz) 149.8, 149.0, 136.2, 133.9, 130.9, 130.8, 130.1, 128.5 (q, $J^{2}_{C\text{-}F}$=37.5 Hz) 125.6, 123.0 (q, $J^{1}_{C\text{-}F}$=337.5 Hz) 120.1, 119.8 (q, $J^{3}_{C\text{-}F}$=12.5 Hz) 114.7, 113.8, 111.2 ; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.5 HRMS calced for $C_{15}H_{10}F_3N_5O_2$ $[MH]^+$ 350.08594, found 350.08574.




Jarrad/PhD/Book 6/JMA6-131
Single Pulse with Broadband Decoupling
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1000000
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50
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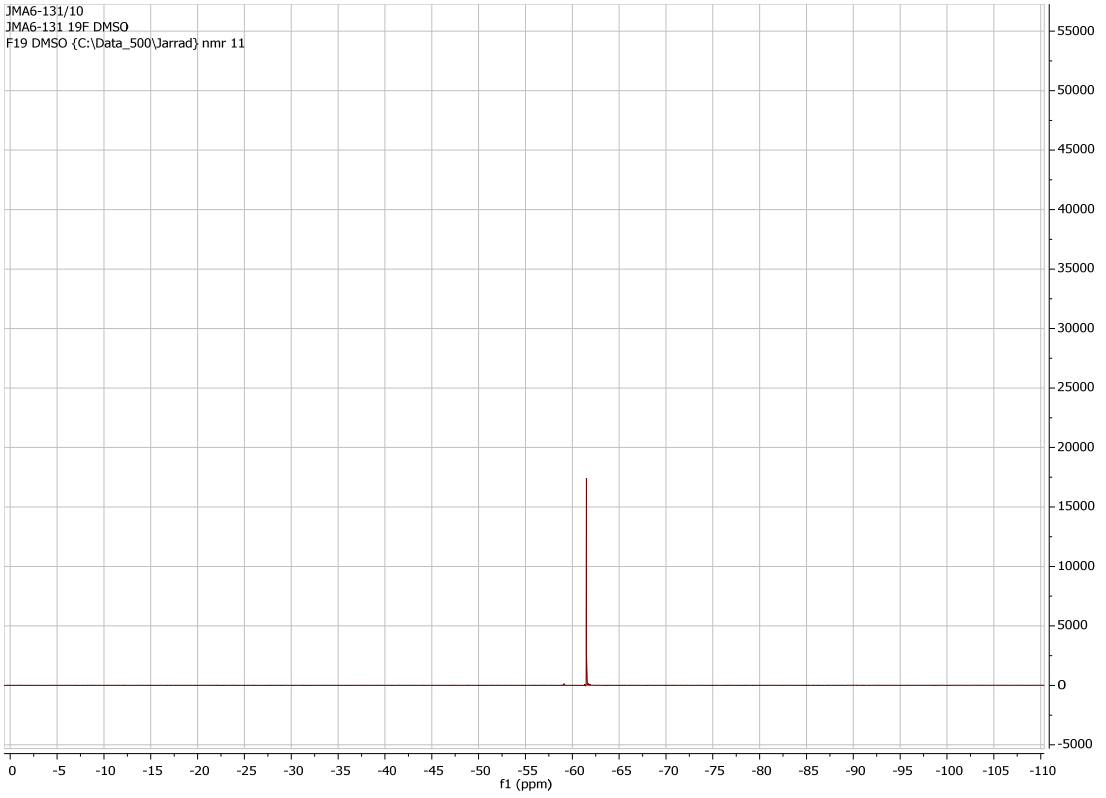
f1 (ppm)


JMA6-131/10
JMA6-131 19F DMSO
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30000
25000
20000
15000
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5000
0
-5000
0
-5
-10
-15
-20
-25
-30
-35
-40
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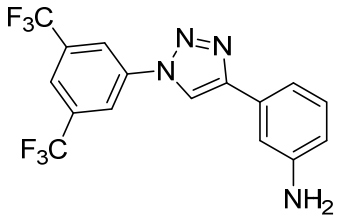
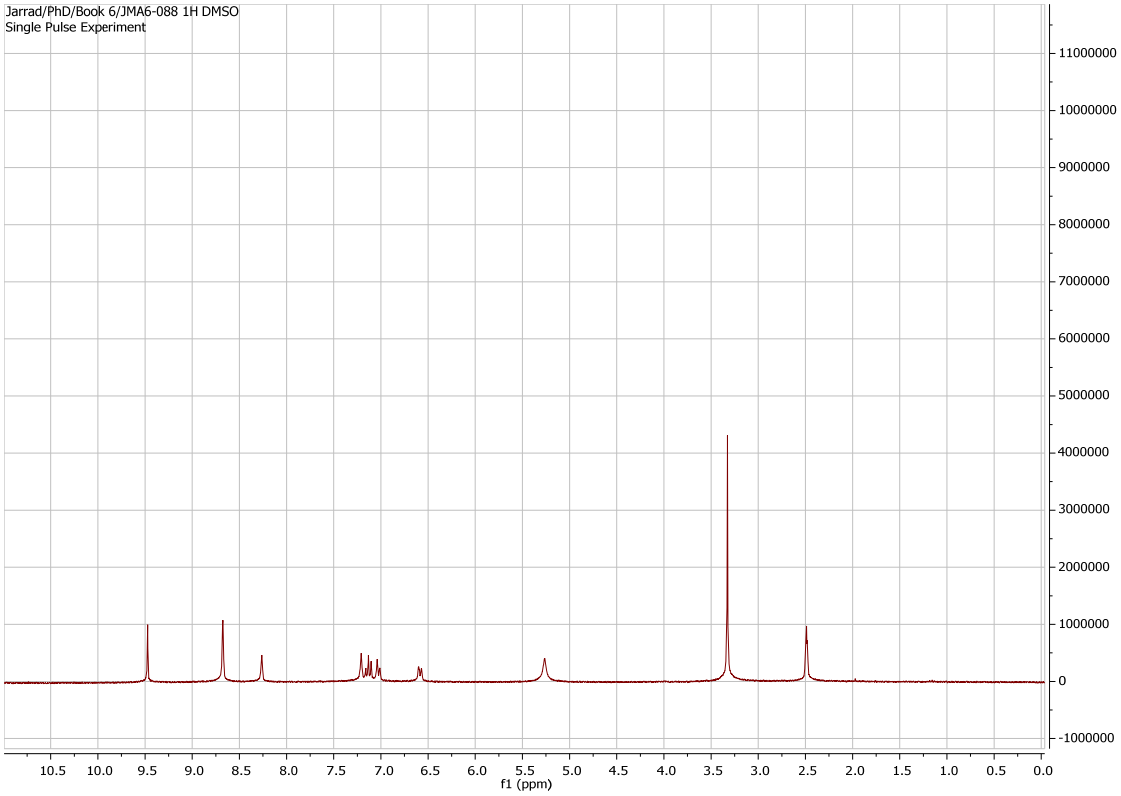
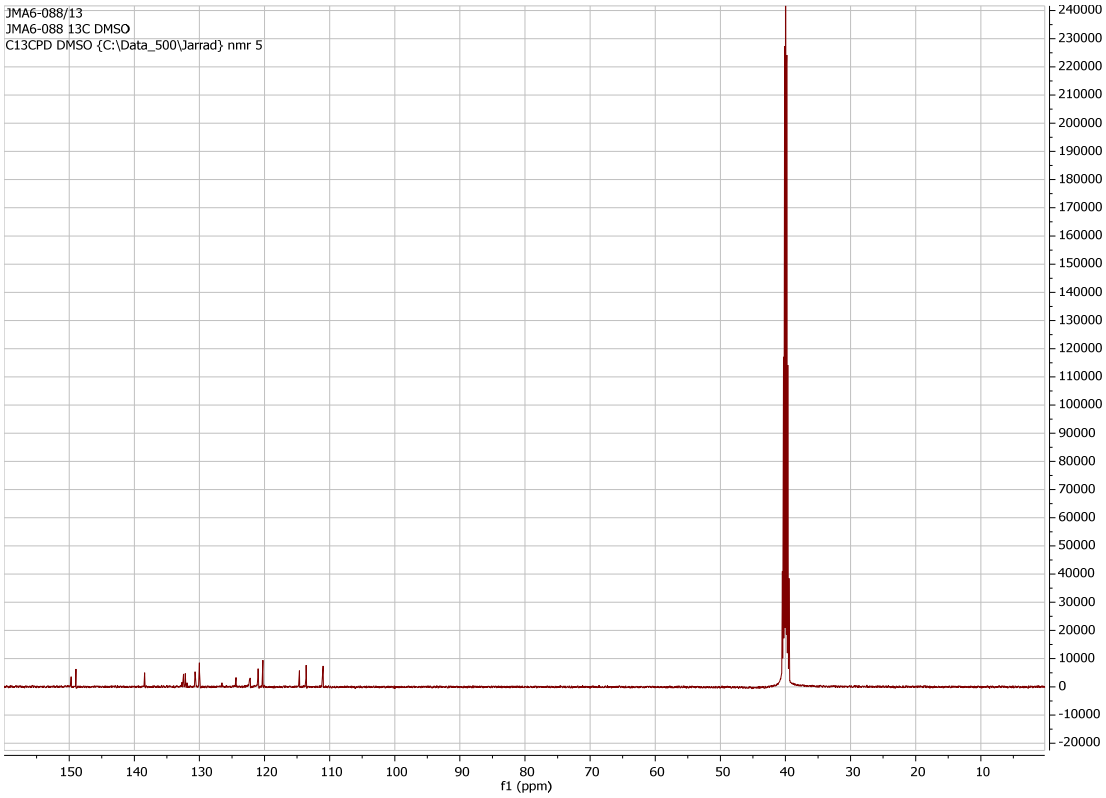
-110
f1 (ppm)



3-{1-[3,5-bis(trifluoromethyl)phenyl]-1H-1,2,3-triazol-4-yl}aniline 13h

IR (thin film) υ, cm^{-1} 3348, 2358, 1633, 1395, 1017, 773, 512, 402; mp 163.5 °C.; ^{1}H NMR (DMSO-d_6, 270 MHz) δ 9.41 (1H, s, Ar-*H*) 8.68 (2H, s, Ar-*H*) 8.27 (1H, s, Ar-*H*) 7.01-7.21 (4H, m, Ar-*H*) 6.59 (1H, d, J = 8.1 Hz, Ar-*H*) 5.26 (2H, br-s, NH_2); ^{13}C NMR (DMSO-d_6, 67.5 MHz) δ 149.7, 149.0, 138.4, 132.3 (q, $J^{2}_{C\text{-}F}$ = 20.25 Hz) 130.7, 130.0, 123.3 (q, $J^{1}_{C\text{-}F}$ = 148.5 Hz) 122.3, 121.0, 120.3, 114.6, 113.6, 111.0; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.3; HRMS calced for $C_{16}H_{10}F_6N_4$ $[MH]^+$ 373.08824, found 373.08835.

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160000
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
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-20000
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140
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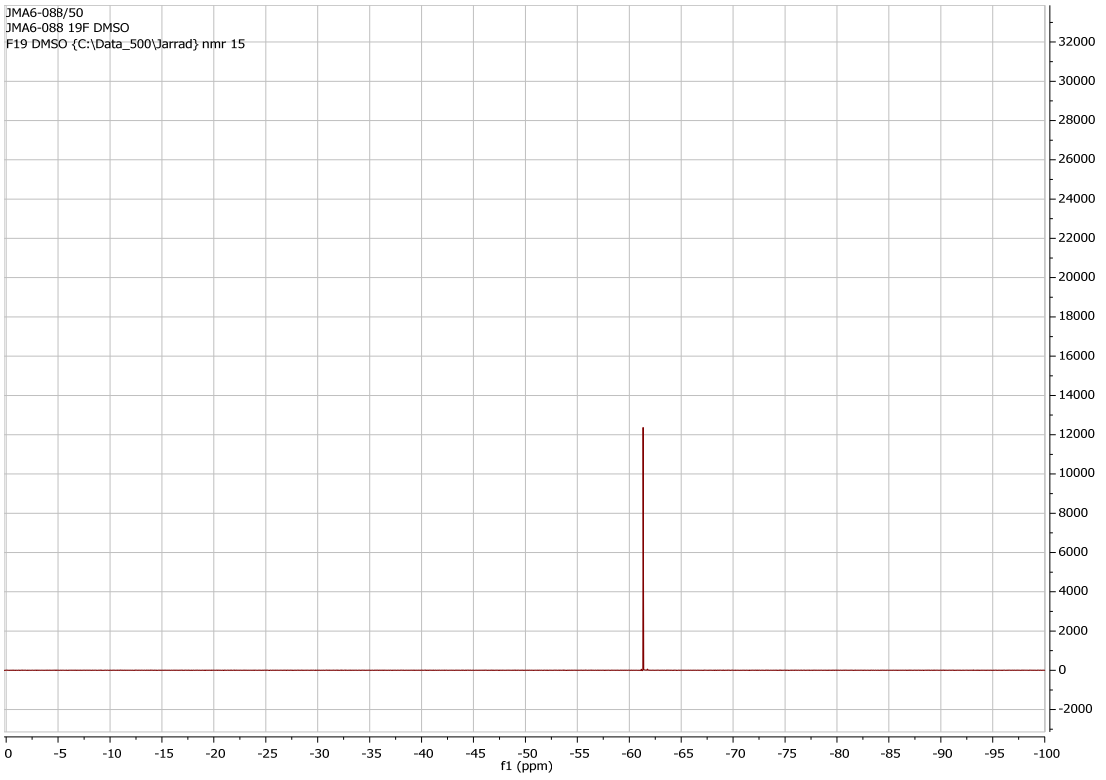
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


JMA6-088/50
JMA6-088 19F DMSO
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22000
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16000
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12000
10000
8000
6000
4000
2000
0
-2000
0
-5
-10
-15
-20
-25
-30
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-60
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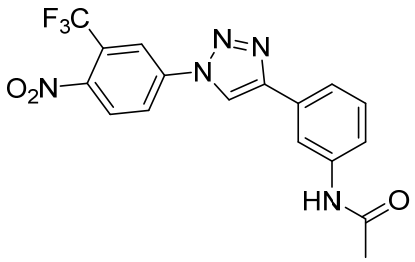
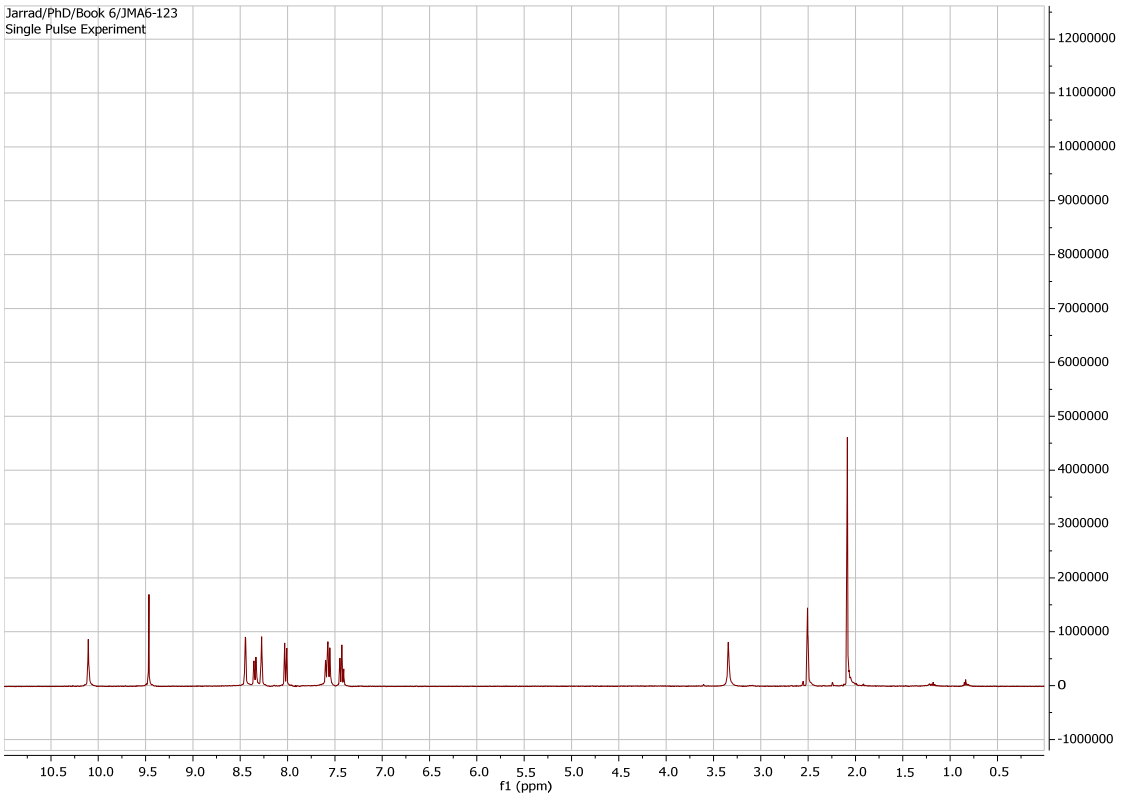
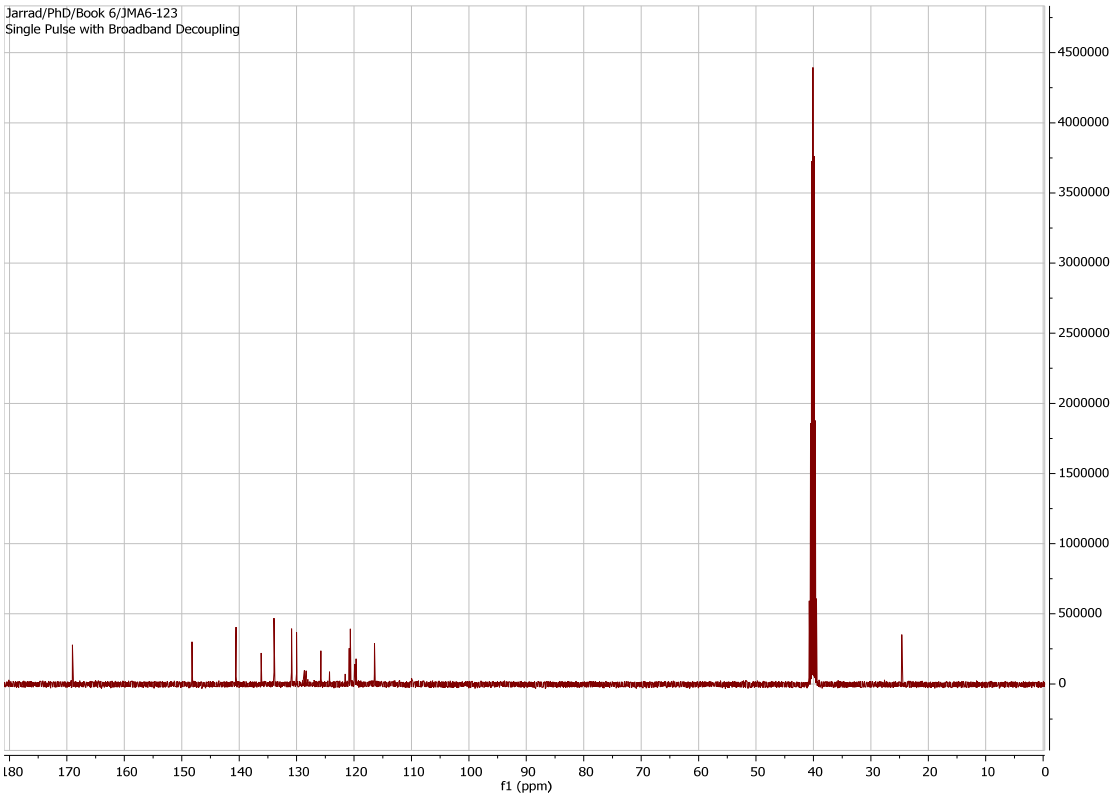
-70
-75
-80
-85
-90
-95
-100
f1 (ppm)



N-(3-{1-[4-nitro-3-(trifluoromethyl)phenyl]-1H-1,2,3-triazol-4-yl}phenyl)acetamide 14g

IR (thin film) υ, cm^{-1} 3401, 2334, 1899, 1620, 1507, 1205, 1050, 752, 510, 402; mp 181.7 °C; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 10.11 (1H, s, -N*H*) 9.47 (1H, s, Ar-*H*) 8.45 (1H, br-s, Ar-*H*) 8.35 (1H, d, J=5 Hz, Ar-*H*) 8.28 (1H, s, Ar-*H*) 8.02 (1H, d, J = 10 Hz, Ar-*H*) 7.55-7.60 (2H, m, Ar-*H*) 7.41-7.45 (1H, m, Ar-*H*); ^{13}C NMR (DMSO-d_6, 125 MHz) δ 169.0, 148.2, 140.6, 136.2, 133.9, 130.9, 130.0, 128.5 (q, $J^{2}_{C\text{-}F}$=37.5 Hz) 125.8, 124.3, 122.9 (q, $J^{1}_{C\text{-}F}$=350 Hz) 120.9, 120.7 119.9 (q, $J^{3}_{C\text{-}F}$=12.5 Hz) 119.6, 116.4, 24.6 ; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.5 HRMS calced for $C_{17}H_{12}F_3N_5O_3$ $[MH]^+$ 392.09650, found 392.09680.




Jarrad/PhD/Book 6/JMA6-123
Single Pulse with Broadband Decoupling
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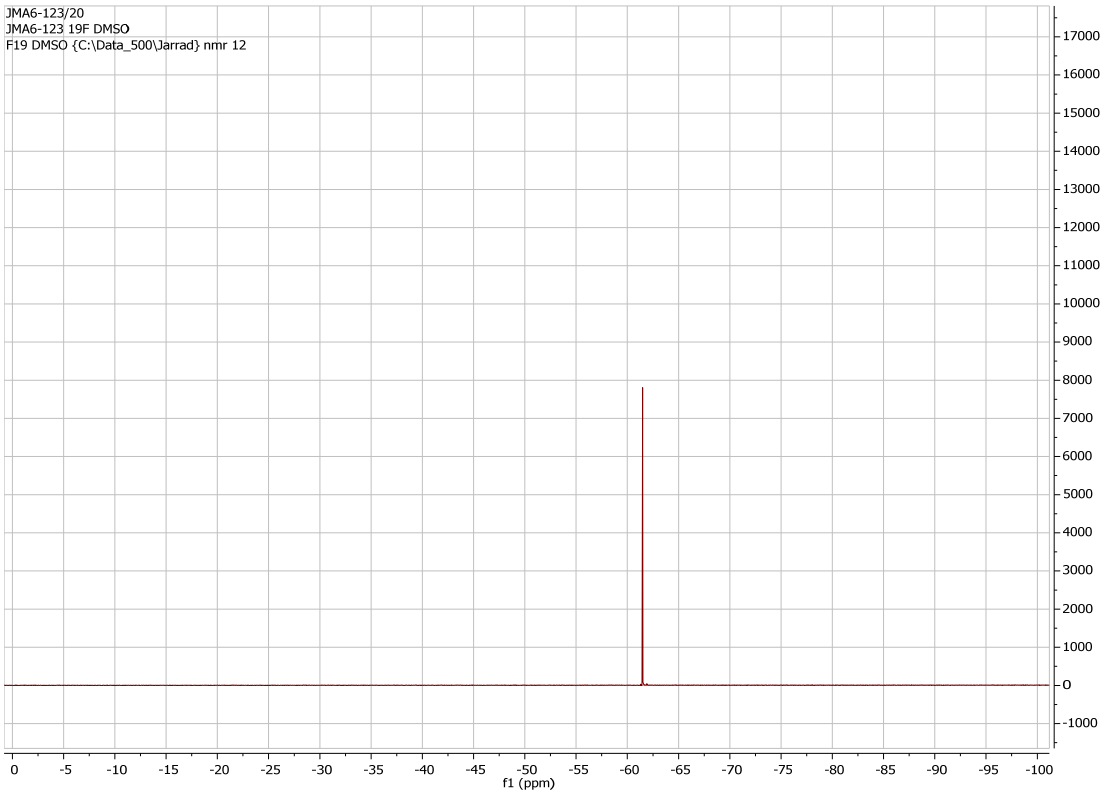
f1 (ppm)


JMA6-123/20
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15000
14000
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11000
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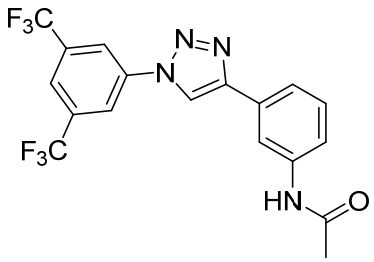
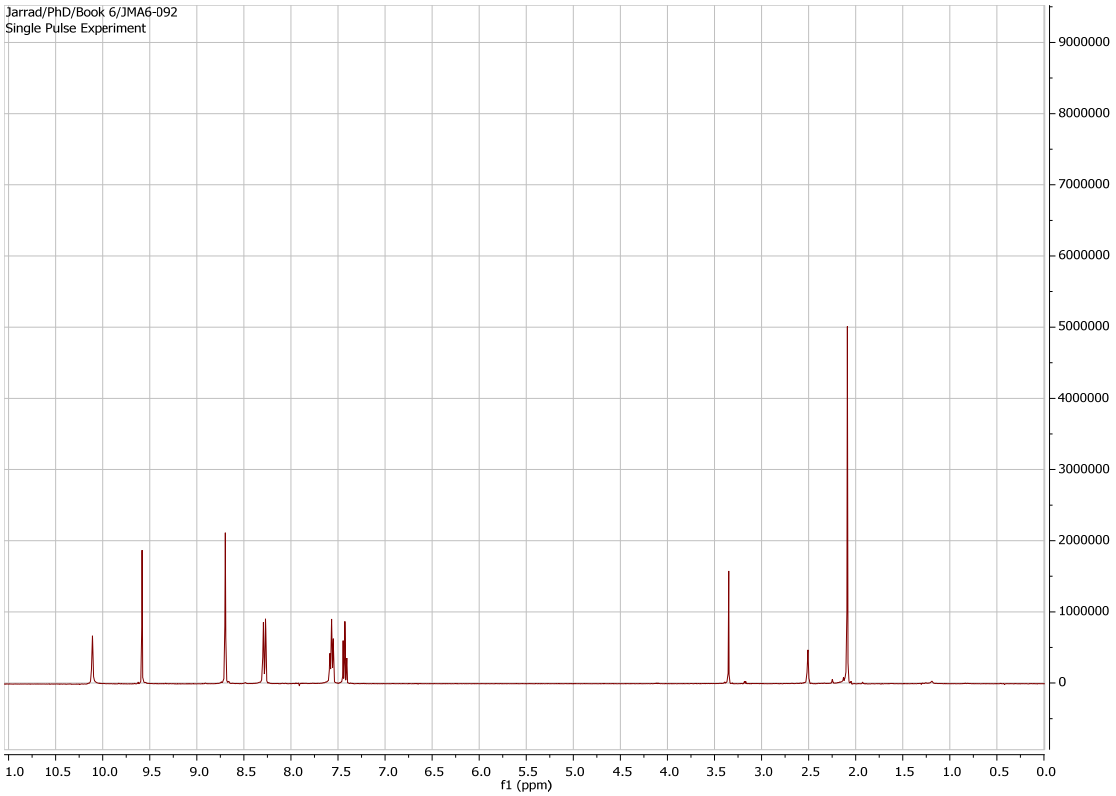
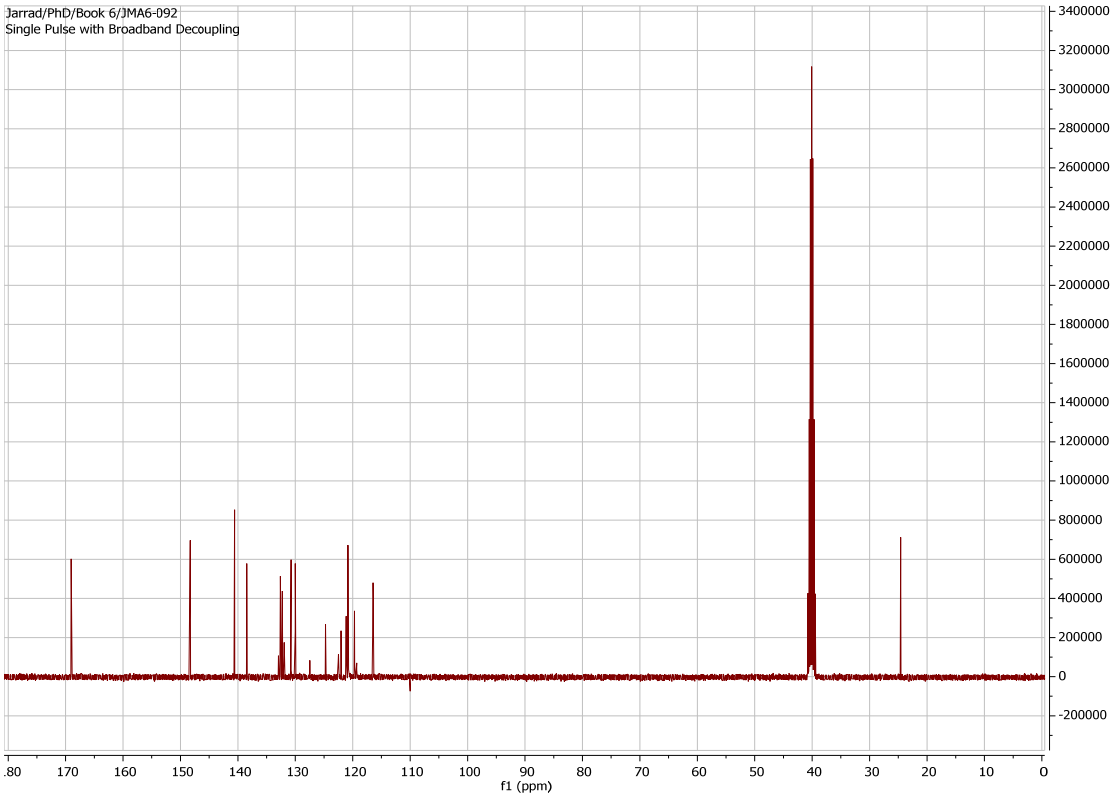
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100
f1 (ppm)

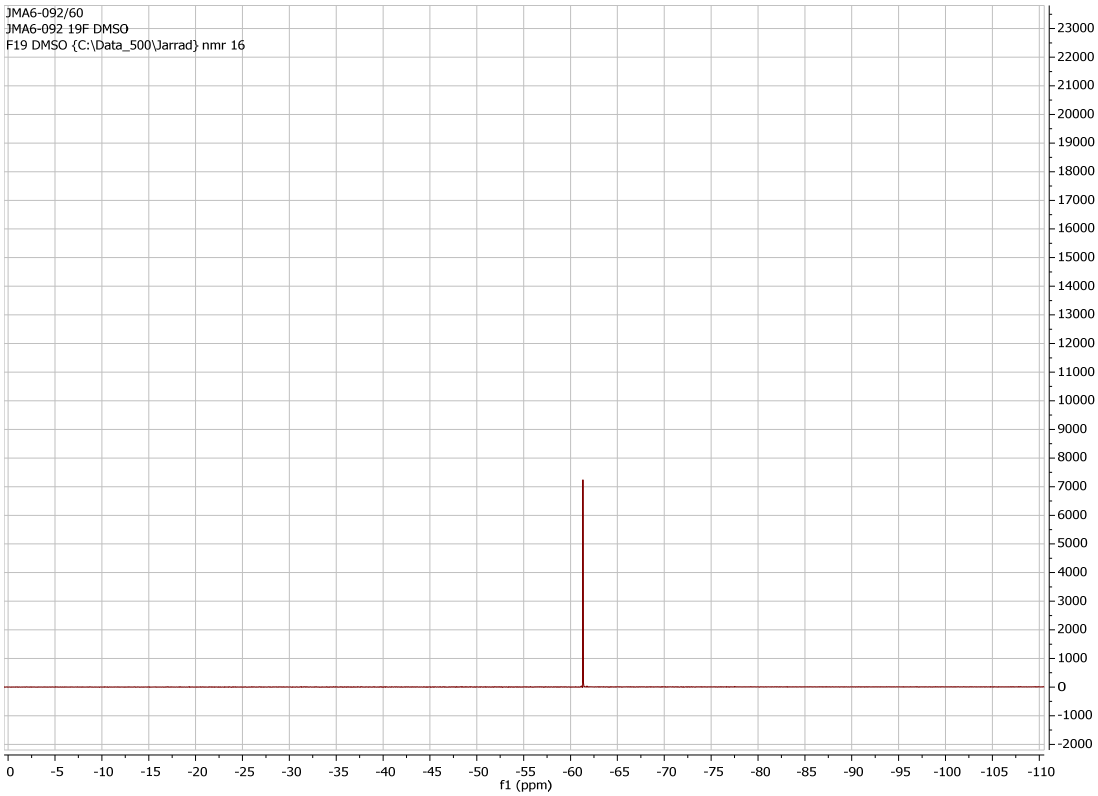


N-(3-{1-[3,5-bis(trifluoromethyl)phenyl]-1H-1,2,3-triazol-4-yl}phenyl)acetamide 14d

IR (thin film) υ, cm^{-1} 3321, 2454, 1600, 1620, 1100, 731, 560, 389; mp 203.6 °C; ^{1}H NMR (DMSO-d_6, 270 MHz): δ 10.11 (1H, s, Ar-*H*) 9.58 (1H, s, Ar-*H*) 8.70 (2H, s, Ar-*H*) 8.28 (2H, d, J = 5.4 Hz, Ar-*H*) 7.57 (2H, t, J=5.4 Hz, Ar-*H*) 7.41-7.45 (1H, m, Ar-*H*) 2.09 (3H, s, CH_3); ^{13}C NMR (DMSO-d_6, 67.5 MHz) δ 169.0, 148.3, 140.6, 138.5, 132.5 (q, $J^{2}_{C\text{-}F}$ = 20.25 Hz) 130.7, 130.0, 123.4 (q, $J^{1}_{C\text{-}F}$=182.3 Hz) 122.5, 121.2, 121.1, 120.8 119.7, 116.4, 24.6; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.3 HRMS calced for $C_{18}H_{12}F_6N_4O$ $[MH]^+$ 415.09881, found 415.09884.




Jarrad/PhD/Book 6/JMA6-092
Single Pulse with Broadband Decoupling
f1 (ppm)


JMA6-092/60
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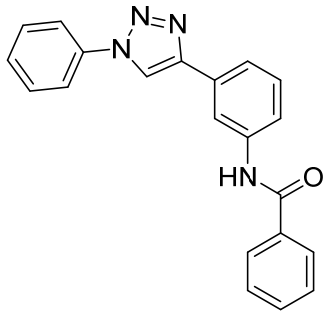
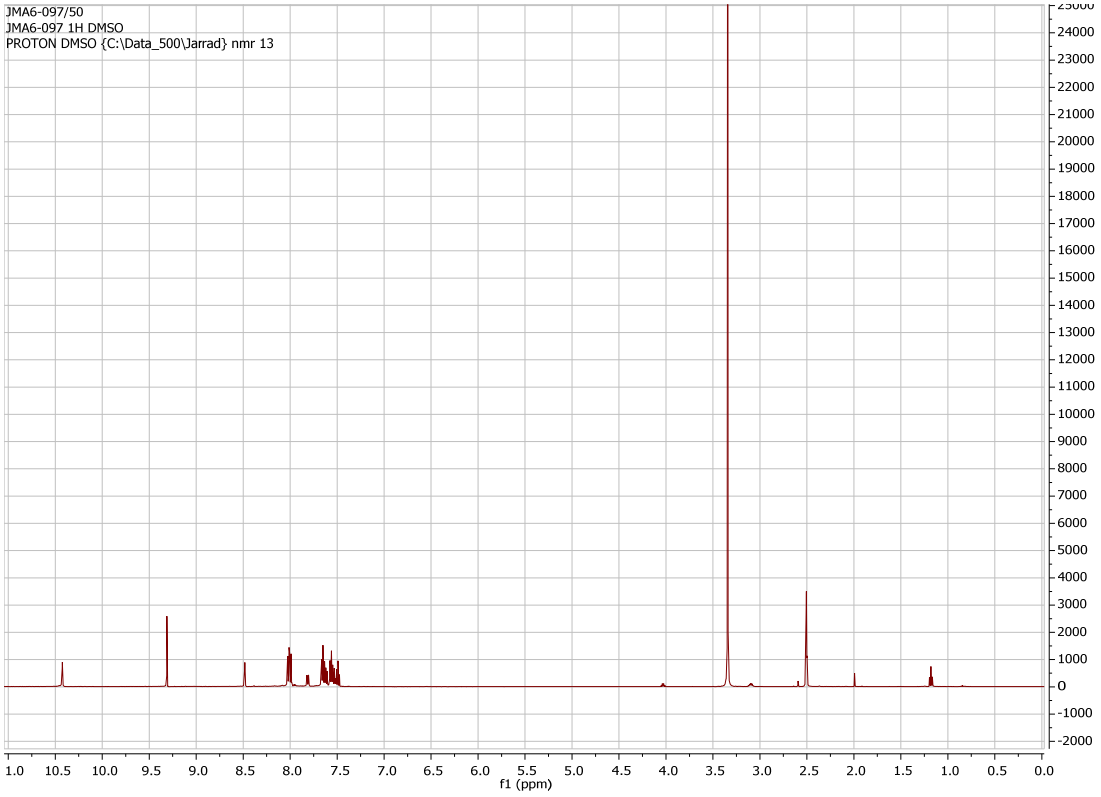
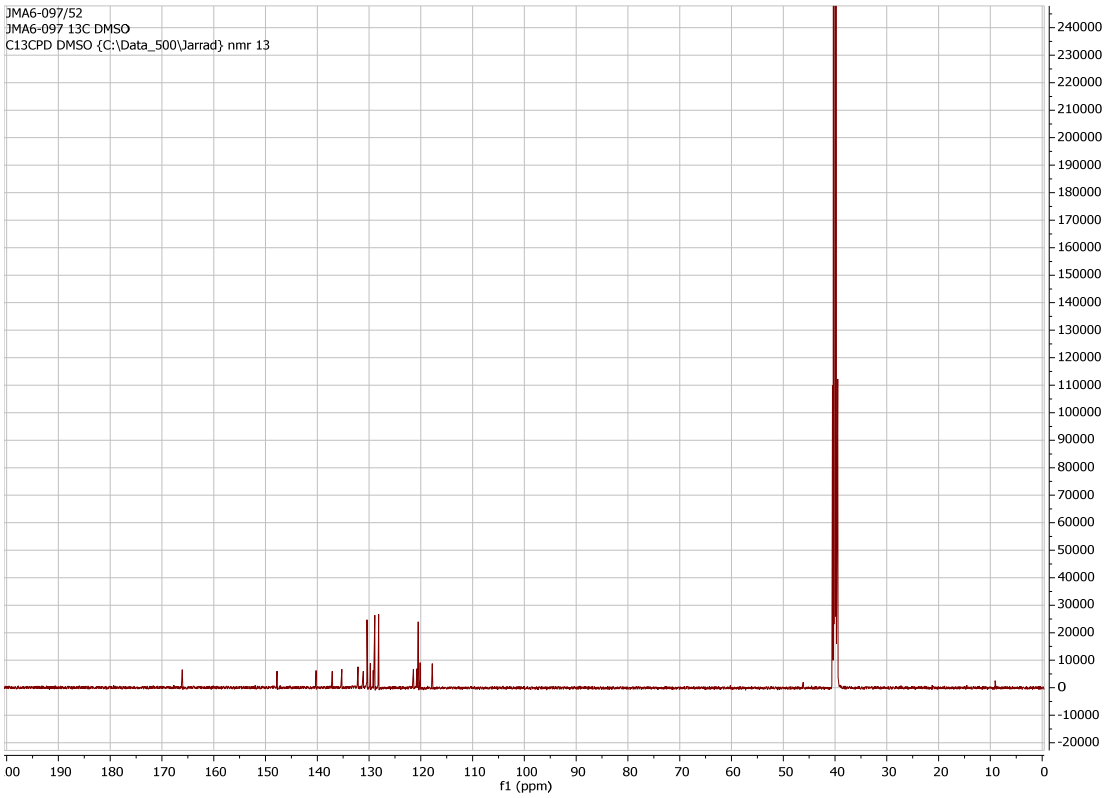
f1 (ppm)



N-[3-(1-phenyl-1H-1,2,3-triazol-4-yl) phenyl]benzamide 14c

IR (thin film) υ, cm^{-1} 3543, 3001, 2790, 2265, 1204, 800, 540, 480; mp 164.1 °C; ^{1}H NMR (DMSO-d_6, 500 MHz) δ 10.42 (1H, s, N*H*) 9.31 (1H, s, Ar-*H*) 8.48 (1H, s, Ar-*H*) 7.99-8.03 (4H, m, Ar-*H*) 7.81 (1H, d, J = 10, Ar-*H*) 7.47-7.67 (8H, m, Ar-*H*); ^{13}C NMR (DMSO-d_6, 125 MHz) 166.1, 147.8, 140.2, 137.1, 135.3, 132.1, 131.1, 130.4, 129.8, 129.2, 128.9, 128.2, 121.4, 120.8, 120.5, 120.1, 117.8; HRMS calced for $C_{21}H_{16}N_4O$ $[MH]^+$ 341.13969, found 341.13954.

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110000
100000
90000
80000
70000
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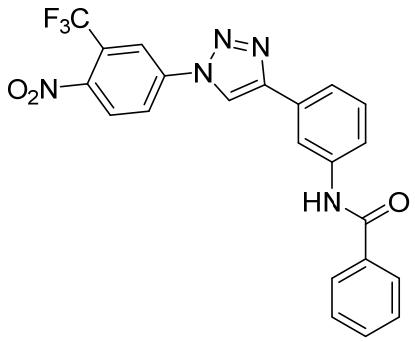
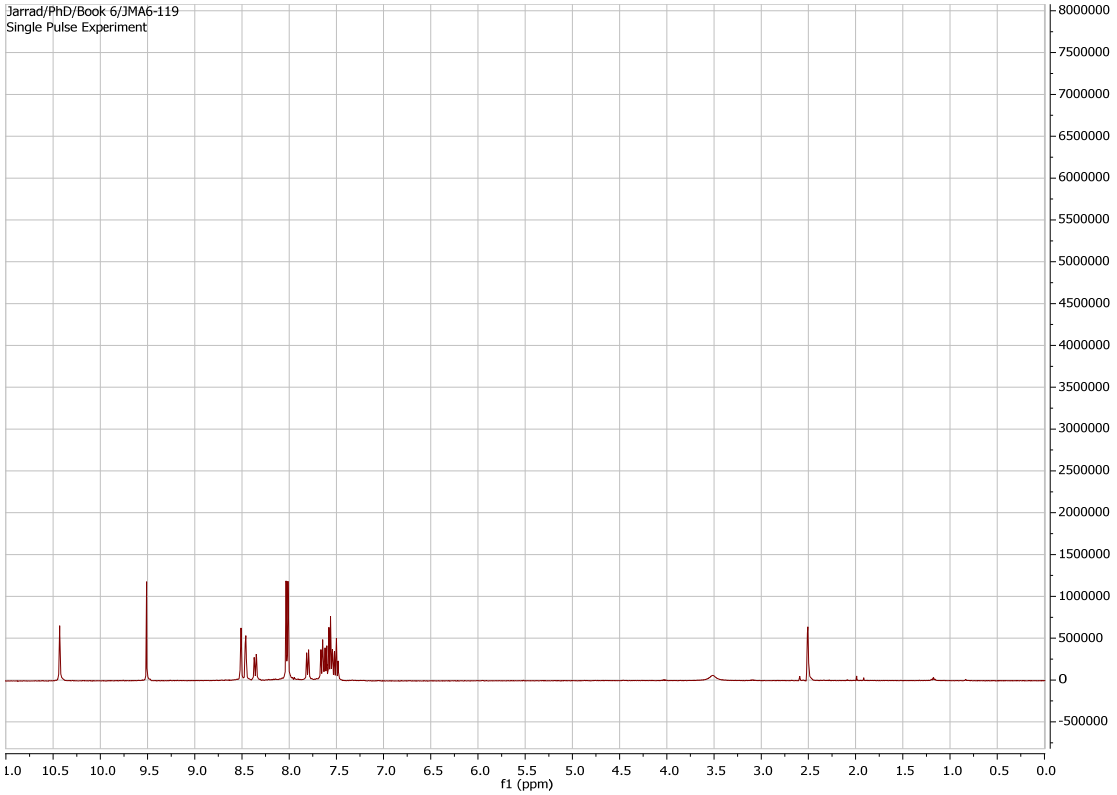
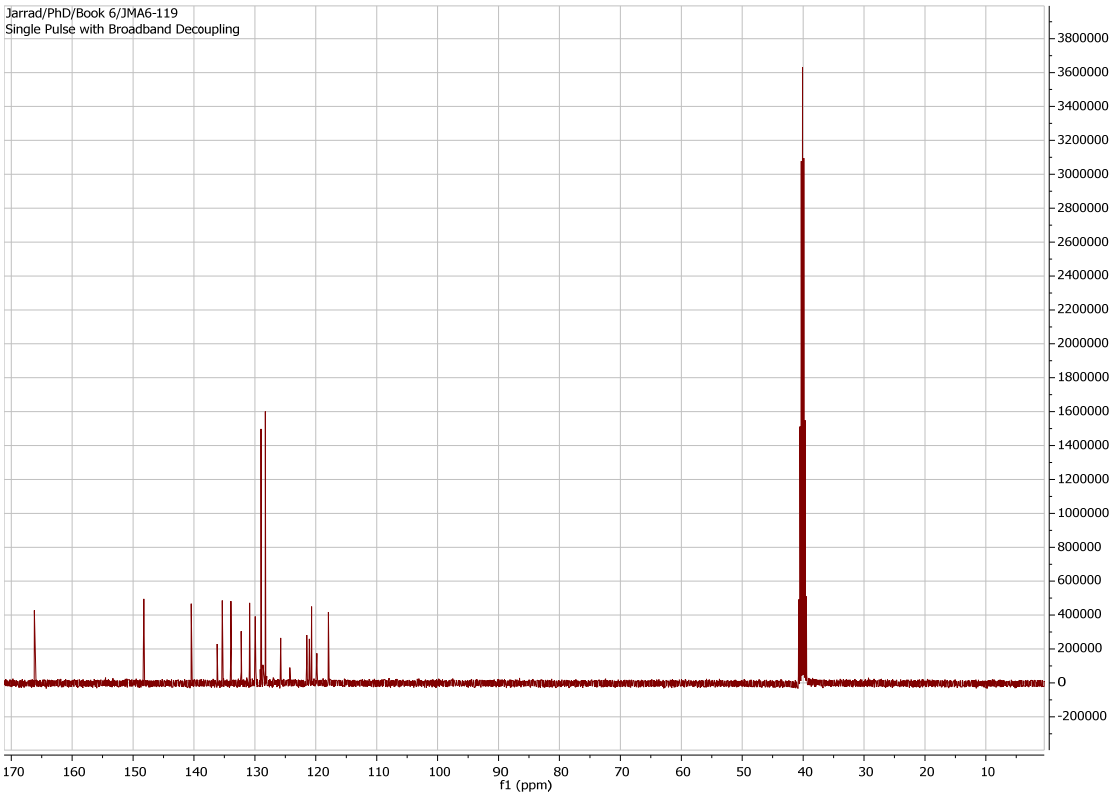
f1 (ppm)



N-(3-{1-[4-nitro-3-(trifluoromethyl)phenyl]-1H-1,2,3-triazol-4-yl}phenyl)benzamide 14i

IR (thin film) υ, cm^{-1} 3455, 3001, 2467, 1290, 1108, 801, 590; mp 218.1 °C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 10.43 (1H, s, Ar-*H*) 9.51 (1H, s, Ar-*H*) 8.46-8.52 (2H, m, Ar-*H*) 8.36 (1H, d, J = 5.4 Hz, Ar-*H*) 8.01-8.03 (3H, m, Ar-*H*) 7.79-7.82 (1H, m, Ar-*H*) 7.48-7.67 (6H, m, Ar-*H*); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 166.2, 148.2, 140.4, 136.2, 135.3, 133.9, 132.2, 130.9, 129.9, 129.0, 128.3, 125.8, 121.5, 121.0, 120.7, 119.8, 117.9; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.5 HRMS calced for $C_{22}H_{14}F_3N_5O_3$ $[MH]^+$ 454.11215, found 454.11235.




Jarrad/PhD/Book 6/JMA6-119
Single Pulse with Broadband Decoupling
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200000
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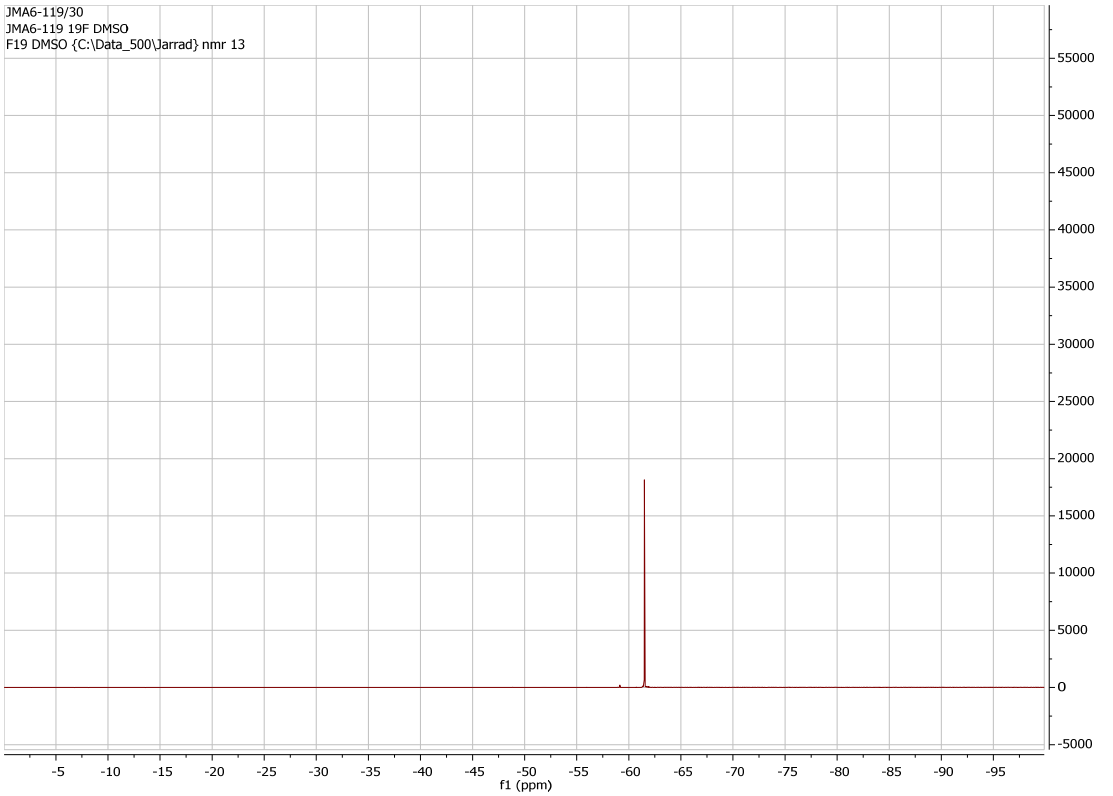
-200000
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


JMA6-119/30
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40000
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20000
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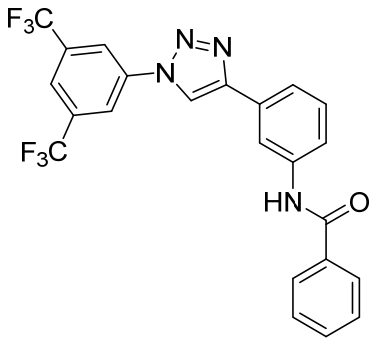
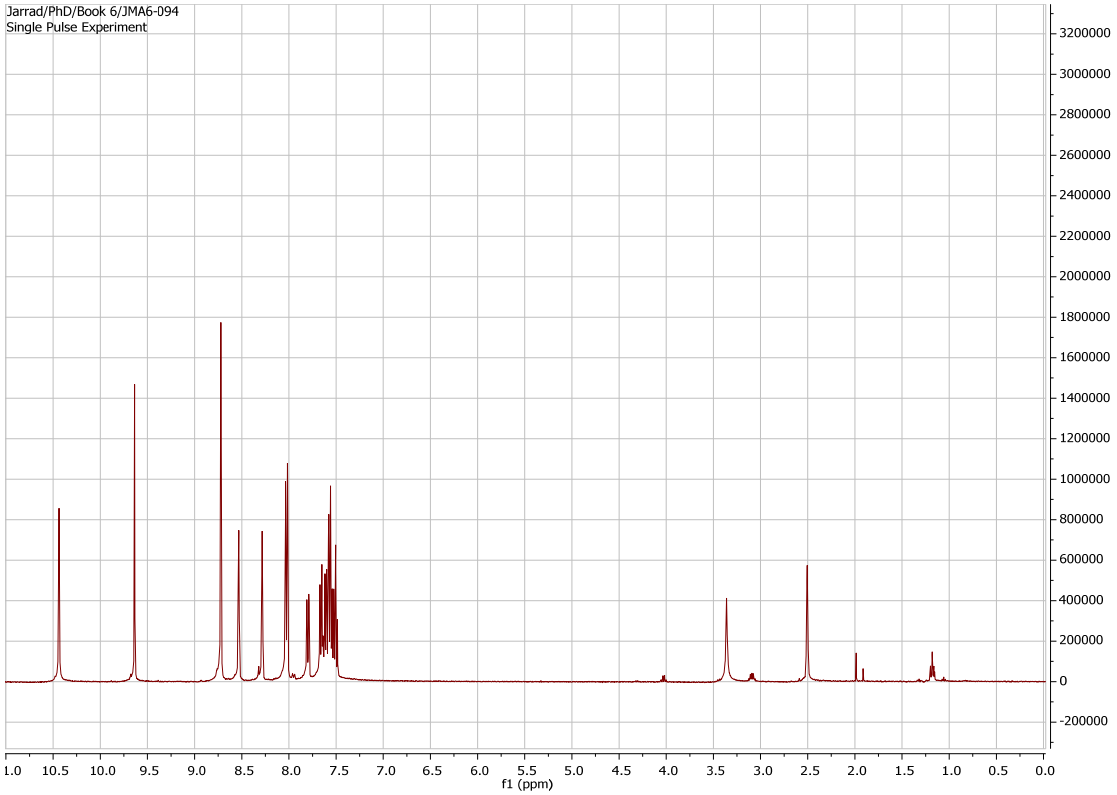
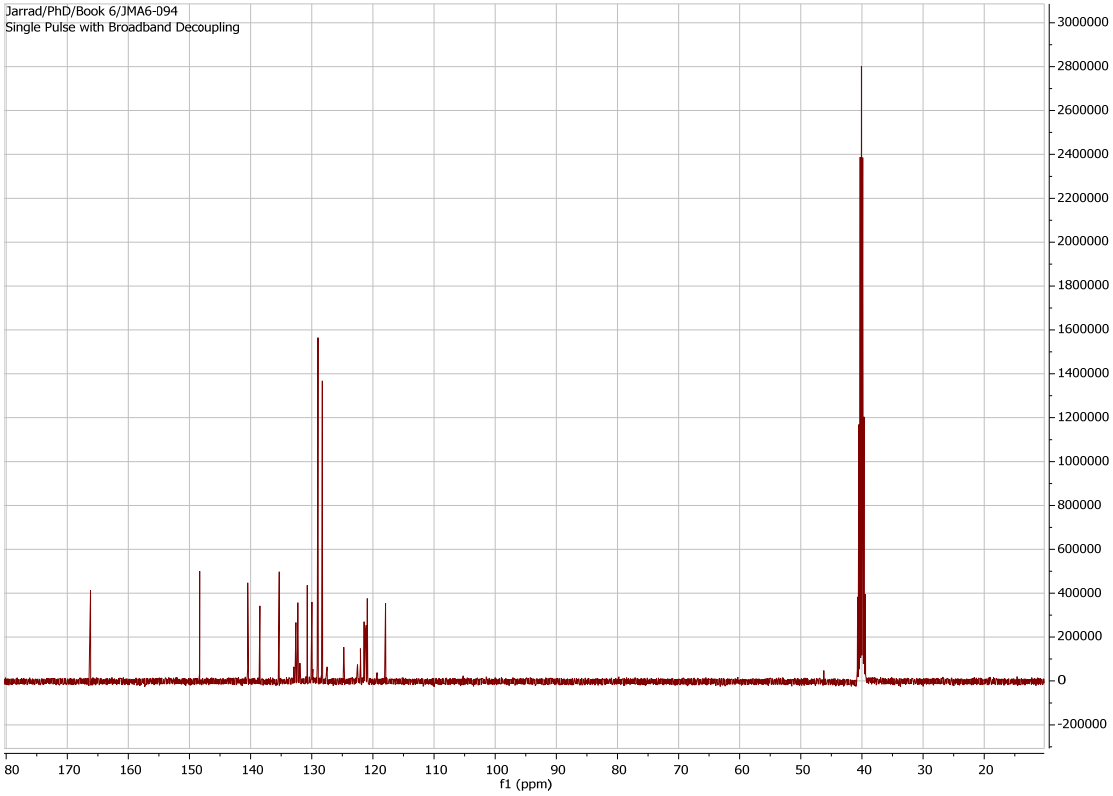
f1 (ppm)



N-(3-{1-[3,5-bis(trifluoromethyl)phenyl]-1H-1,2,3-triazol-4-yl}phenyl)benzamide 14f

IR (thin film) υ, cm^{-1} 3398, 3101, 2500, 1340, 1105, 701, 370; mp 218.9 °C; ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.44 (1H, s, Ar-*H*) 9.64 (1H, s, Ar-*H*) 8.72 (2H, s, Ar-*H*) 8.54 (1H, s, Ar-*H*) 8.28 (1H, s, Ar-*H*) 8.02-8.04 (2H, m, Ar-*H*) 7.79-7.81 (1H, m, Ar-*H*) 7.51-7.67 (5H, m, Ar-*H*); ^{13}C NMR (DMSO-d_6, 100 MHz) 166.2, 148.3, 140.3, 138.5, 135.3, 132.5 (q, $J^{2}_{C\text{-}F}$ = 30 Hz) 132.2, 130.7, 130.0, 129.0, 128.3, 123.4 (q, $J^{1}_{C\text{-}F}$=270 Hz) 122.5, 121.4, 121.1, 120.9, 117.9; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.3; HRMS calced for $C_{23}H_{14}F_6N_4O$ $[MH]^+$ 477.11446, found 477.11318.




Jarrad/PhD/Book 6/JMA6-094
Single Pulse with Broadband Decoupling
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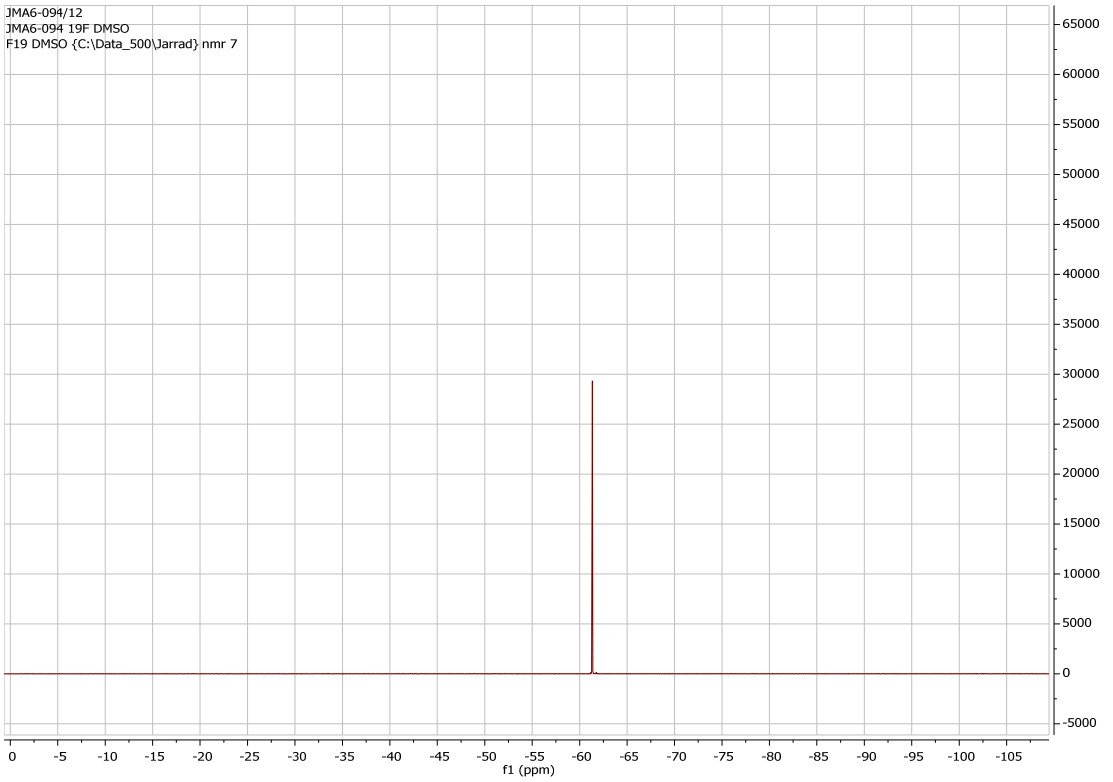
2600000
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
-200000
80 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)


JMA6-094/12
JMA6-094 19F DMSO
F19 DMSO {C:\Data_500\Jarrad} nmr 7
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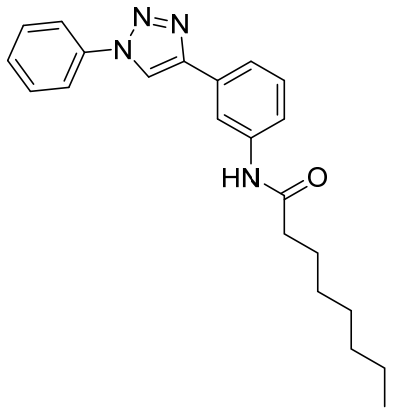
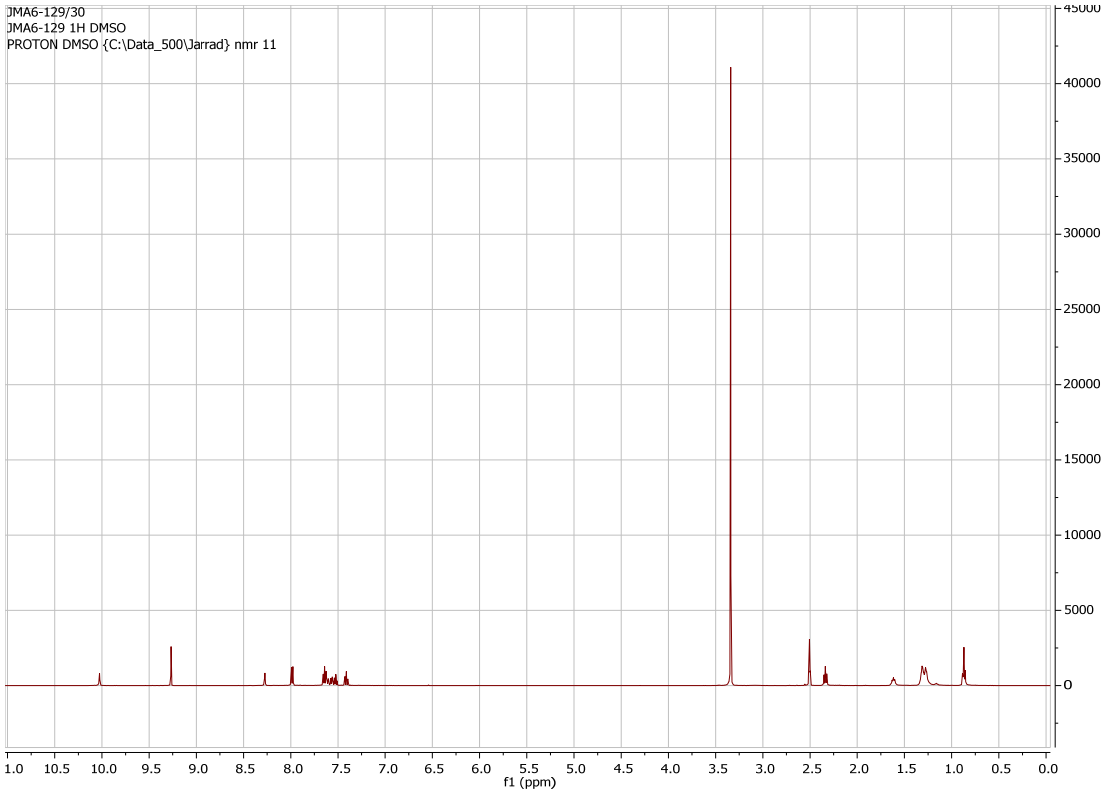
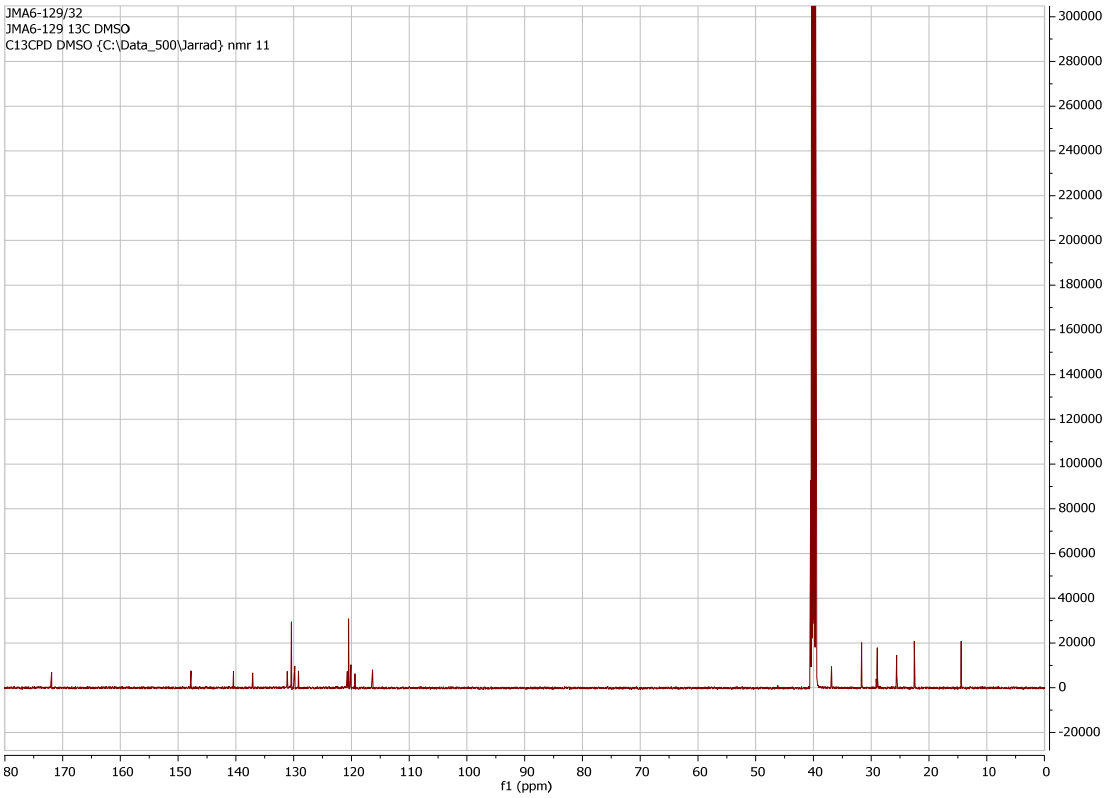
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105
f1 (ppm)



N-[3-(1-phenyl-1H-1,2,3-triazol-4-yl)phenyl]octanamide 14b

IR (thin film) υ, cm^{-1} 3254, 2958, 2900, 2460, 1300, 1100, 796, 524, 301; mp 140.9 °C; ^{1}H NMR (DMSO-d_6, 500 MHz) δ 10.03 (1H, s, N*H*) 9.27 (1H, s, Ar-*H*) 8.27 (1H, s, Ar-*H*) 7.98-8.00 (2H, m, Ar-*H*) 7.51-7.66 (5H, m, Ar-*H*) 2.34 (2H, t, J = 5 Hz, -CH_2) 1.60-1.63 (2H, m, -CH_3) 1.26-1.32 (8H, m, -CH_2); ^{13}C NMR (DMSO-d_6, 125 MHz) δ 171.9, 147.8, 140.4, 137.1, 131.1, 130.4, 129.8, 129.2, 120.7, 120.5, 120.1, 119.4, 116.3, 36.9, 31.7, 29.1, 29.0, 25.6, 22.5, 14.4; HRMS calced for $C_{22}H_{26}N_4O$ $[MH]^+$ 363.21794, found 363.21642.




JMA6-129/32
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240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
80
170
160
150
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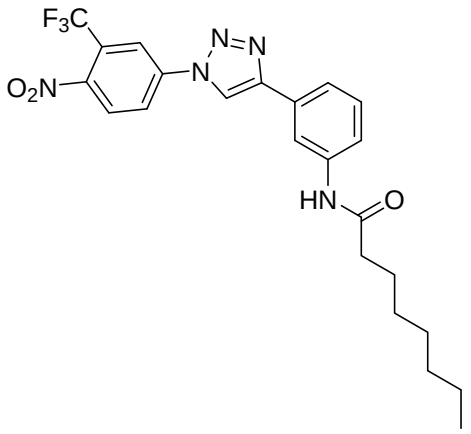
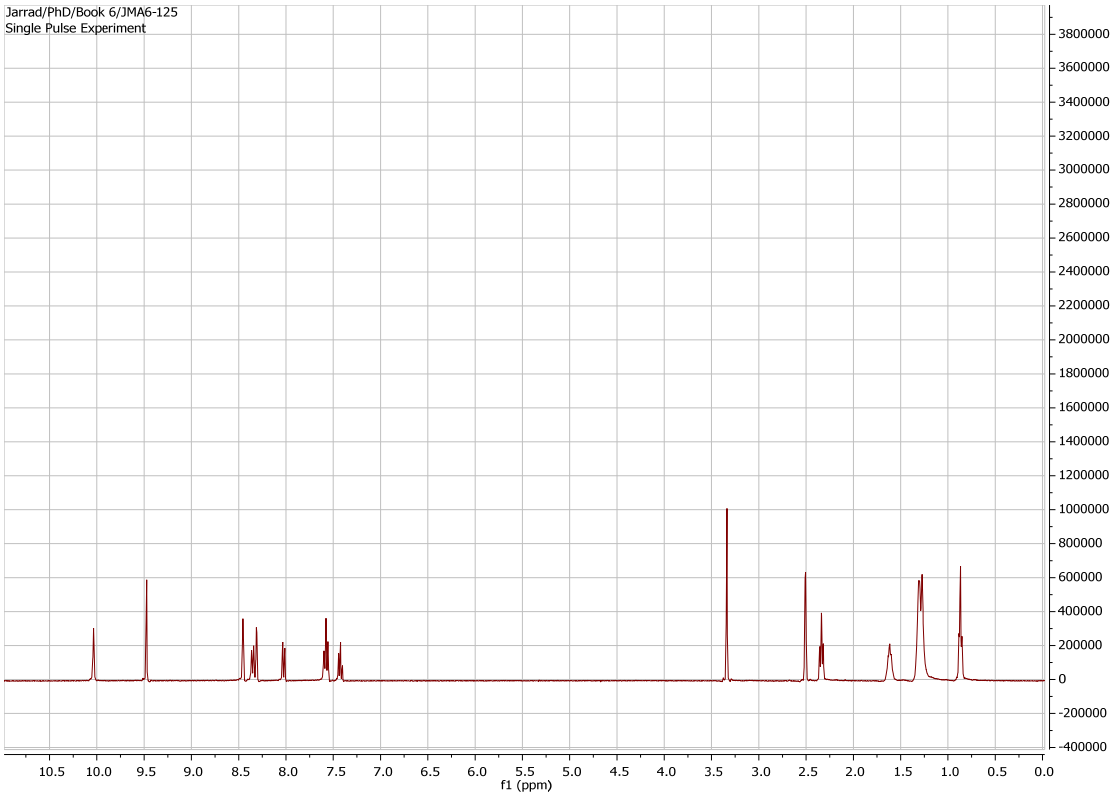
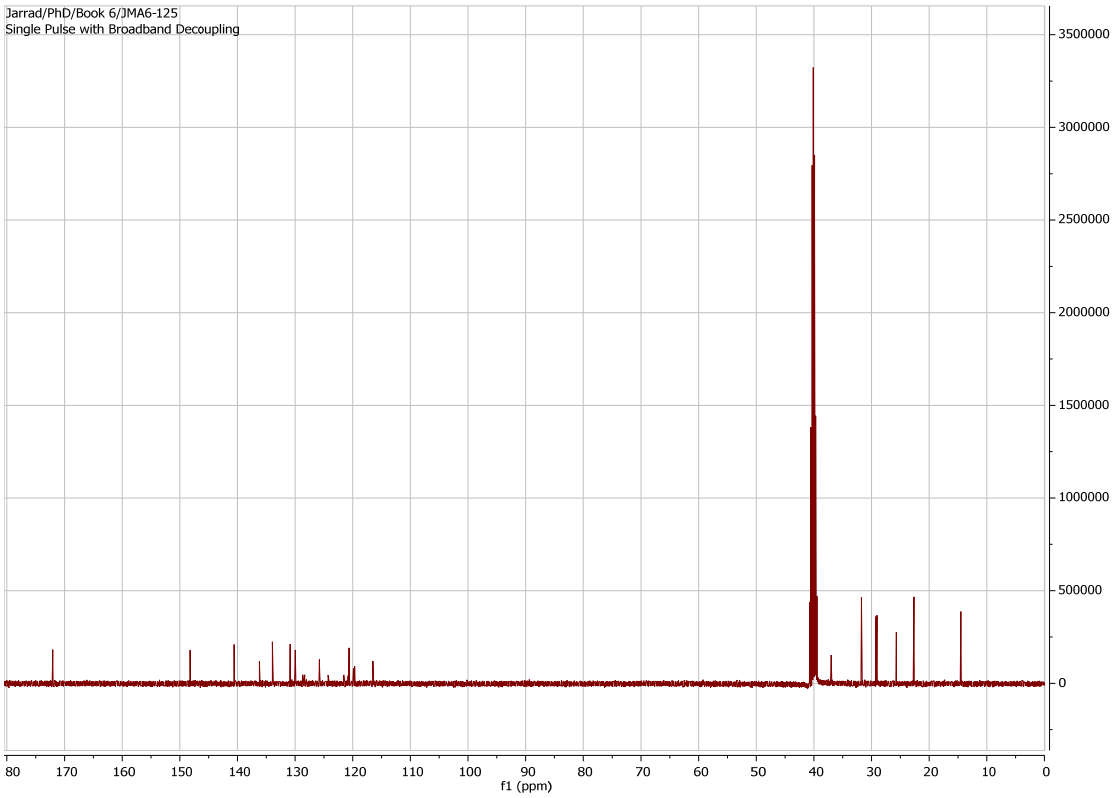
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



N-(3-{1-[4-nitro-3-(trifluoromethyl)phenyl]-1H-1,2,3-triazol-4-yl}phenyl)octanamide 14h

IR (thin film) υ, cm^{-1} 3365, 2221, 1602, 1500, 1200, 1021, 690; mp 153.5 °C; ^{1}H NMR (DMSO-d_6, 400 MHz) δ 10.03 (1H, s, N*H*) 9.48 (1H, s, Ar-*H*) 8.46 (1H, s, Ar-*H*) 8.30-8.36 (2H, m, Ar-*H*) 8.03 (1H, d, J=4 Hz, Ar-*H*) 7.55-7.60 (2H, m, Ar-*H*) 7.40-7.45 (1H, m, Ar-*H*) 2.34 (2H, t, J=8 Hz, CH_2) 1.62 (3H, t, J=8 Hz, CH_3) 1.26-1.31 (8H, m, CH_2); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 172.0, 148.2, 140.6, 136.2, 133.9, 130.9, 130.0, 128.5 (q, $J^{2}_{C\text{-}F}$=30 Hz) 125.8, 123.0 (q, $J^{1}_{C\text{-}F}$=270 Hz) 120.8, 120.6, 119.9, 119.7, 116.5, 37.0, 31.8, 29.1, 29.1, 25.7, 22.7, 14.5; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.5; HRMS calced for $C_{23}H_{24}F_3N_5O_3$ $[MH]^+$ 476.19040, found 476.19070.




Jarrad/PhD/Book 6/JMA6-125
Single Pulse with Broadband Decoupling
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1000000
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0
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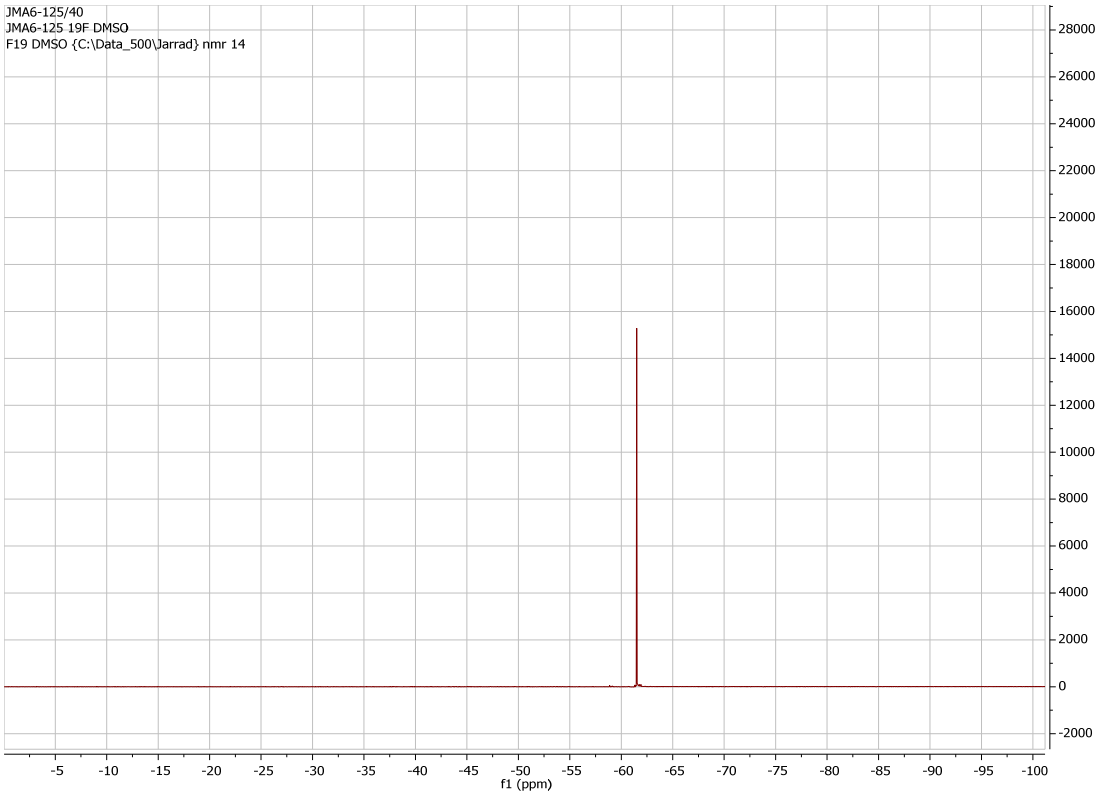
f1 (ppm)


JMA6-125/40
JMA6-125 19F DMSO
F19 DMSO {C:\Data_500\Jarrad} nmr 14
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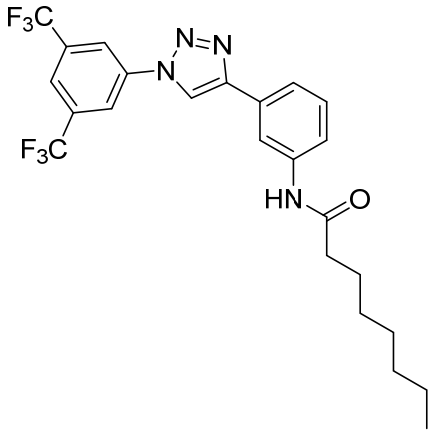
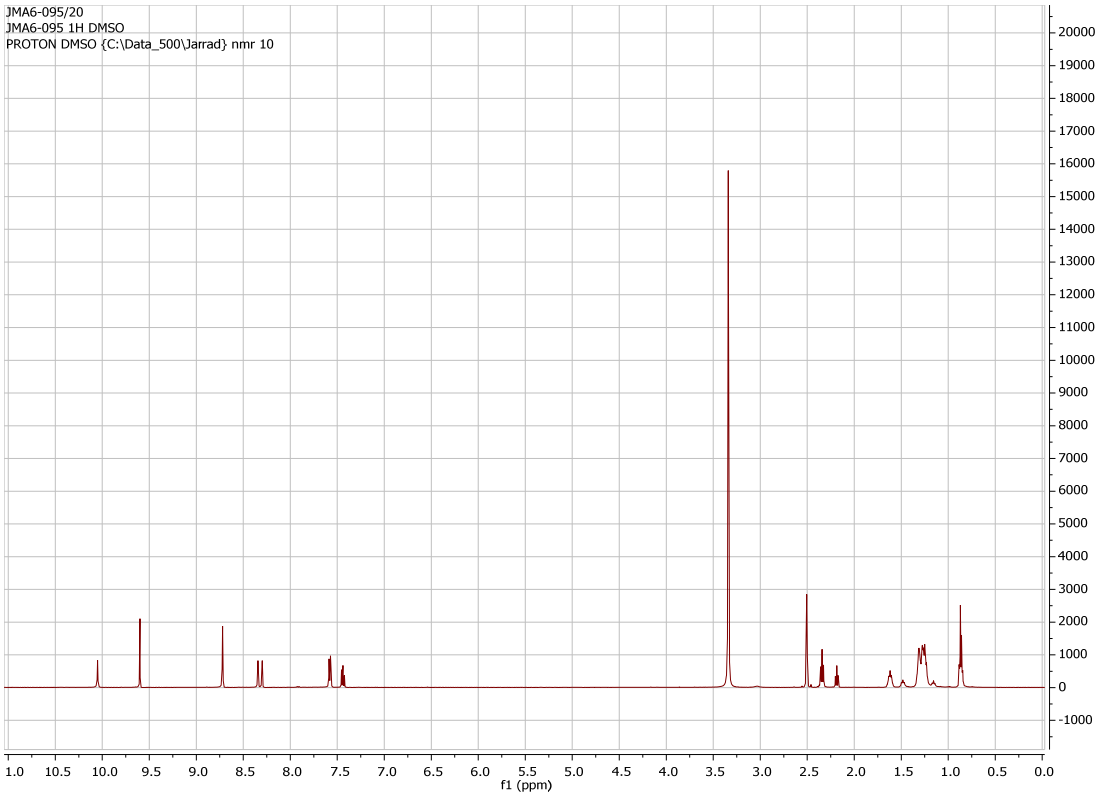
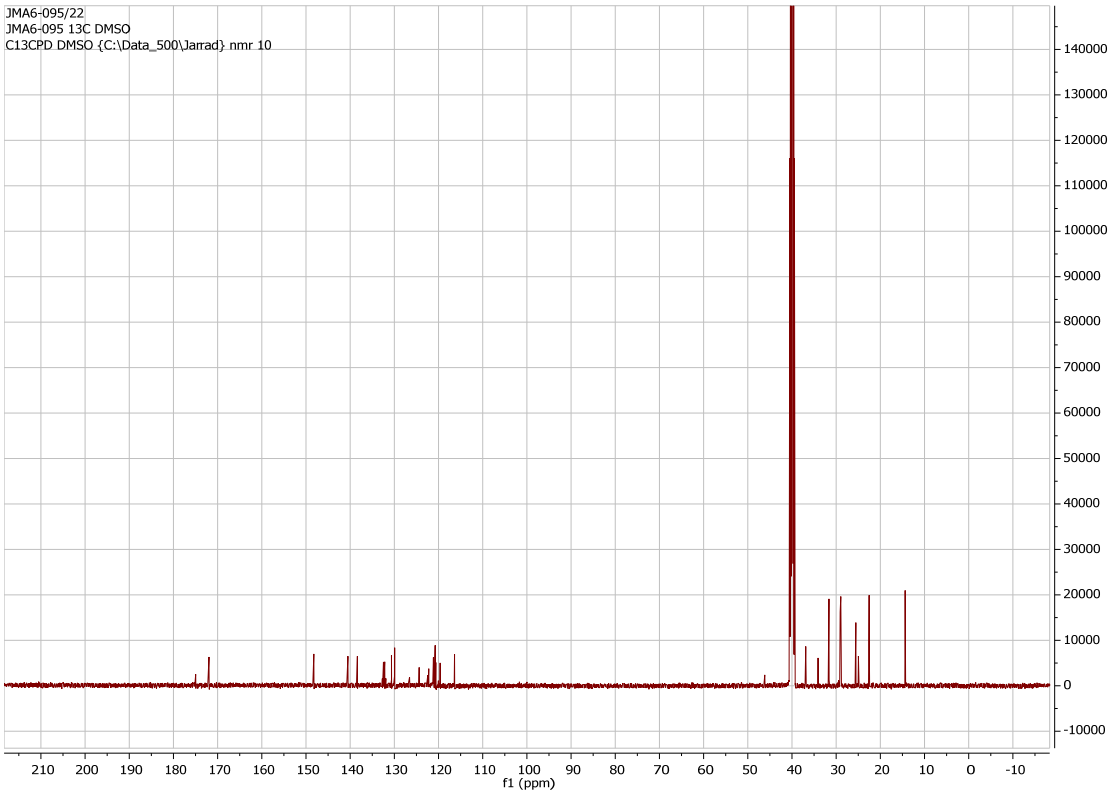
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
-5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100
f1 (ppm)



N-(3-{1-[3,5-bis(trifluoromethyl)phenyl]-1H-1,2,3-triazol-4-yl}phenyl)octanamide 14e

IR (thin film) υ, cm^{-1} 3290, 2418, 1589, 1401, 1117, 780, 487, 441; mp 148.9 °C; ^{1}H NMR (DMSO-d_6, 500 MHz) δ 10.05 (1H, s, N*H*) 9.60 (1H, s, Ar-*H*) 8.72 (2H, s, Ar-*H*) 8.32 (2H, d, J = 20 Hz, Ar-*H*) 7.57-7.59 (2H, m, Ar-*H*) 7.44 (1H, t, J = 10 Hz, Ar-*H*) 2.34 (2H, t, J = 10 Hz, CH_2) 1.60-1.63 (3H, m, CH_3) 1.23-1.32 (10H, m, CH_2); ^{13}C NMR (DMSO-d_6, 125 MHz) δ 175.0, 171.9, 148.2, 140.5, 138.4, 132.3 (q, $J^2_{C\text{-}F}$ = 25 Hz) 130.6, 129.9, 123.3 (q, $J^1_{C\text{-}F}$ = 275 Hz) 122.5, 121.2, 120.8, 120.6, 119.6, 116.4, 36.9, 34.1, 31.7, 29.1, 28.9, 25.6, 25.0, 22.5, 14.4; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.3; HRMS calced for $C_{24}H_{24}F_6N_4O$ $[MH]^+$ 499.19271, found 499.19381

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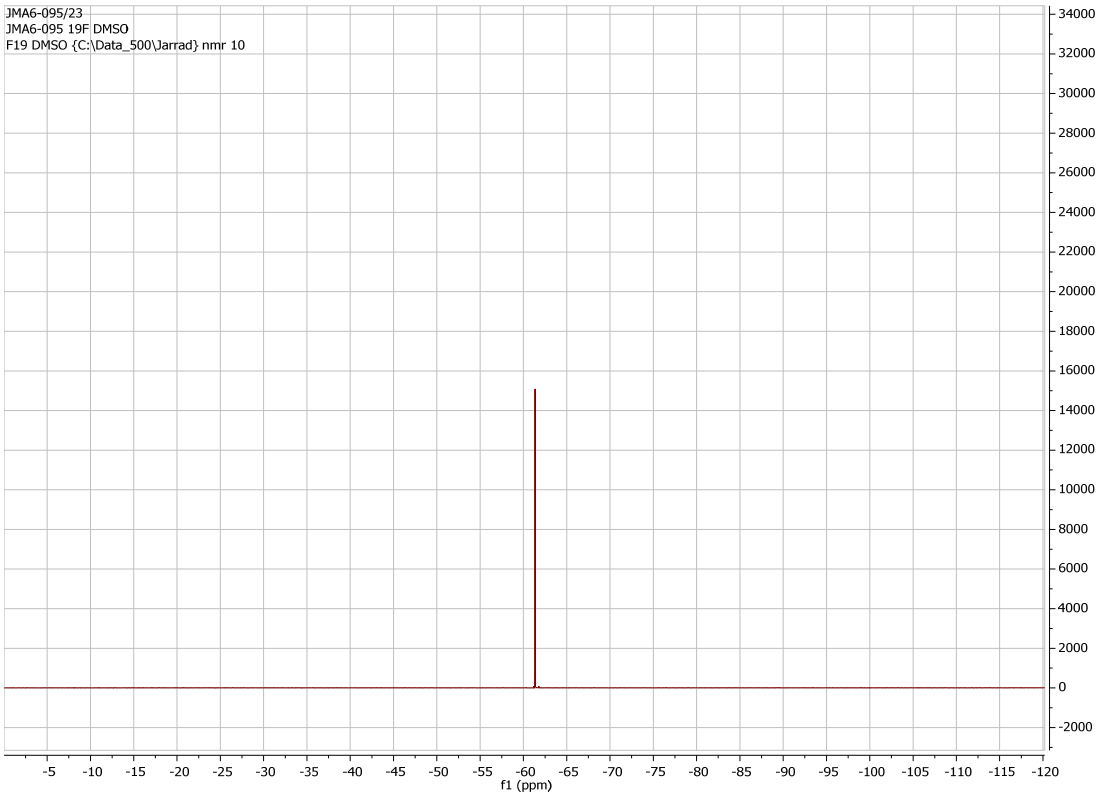
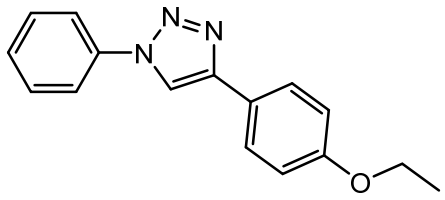
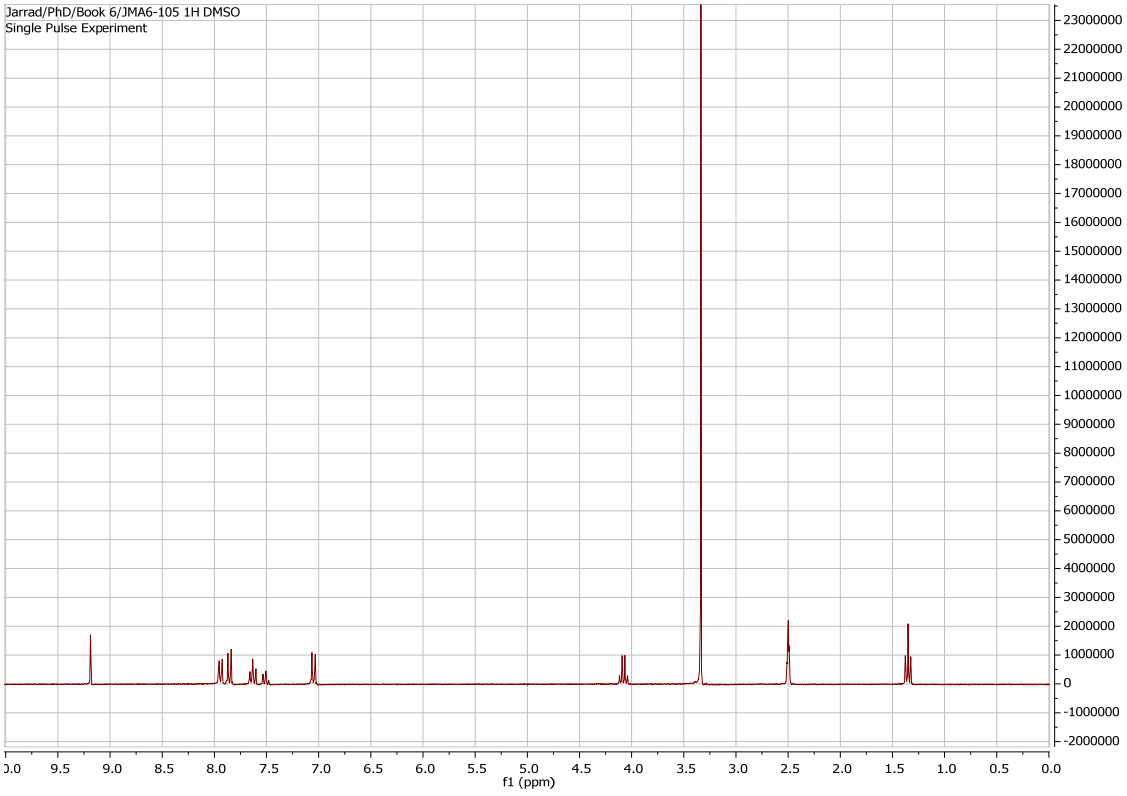
f1 (ppm)

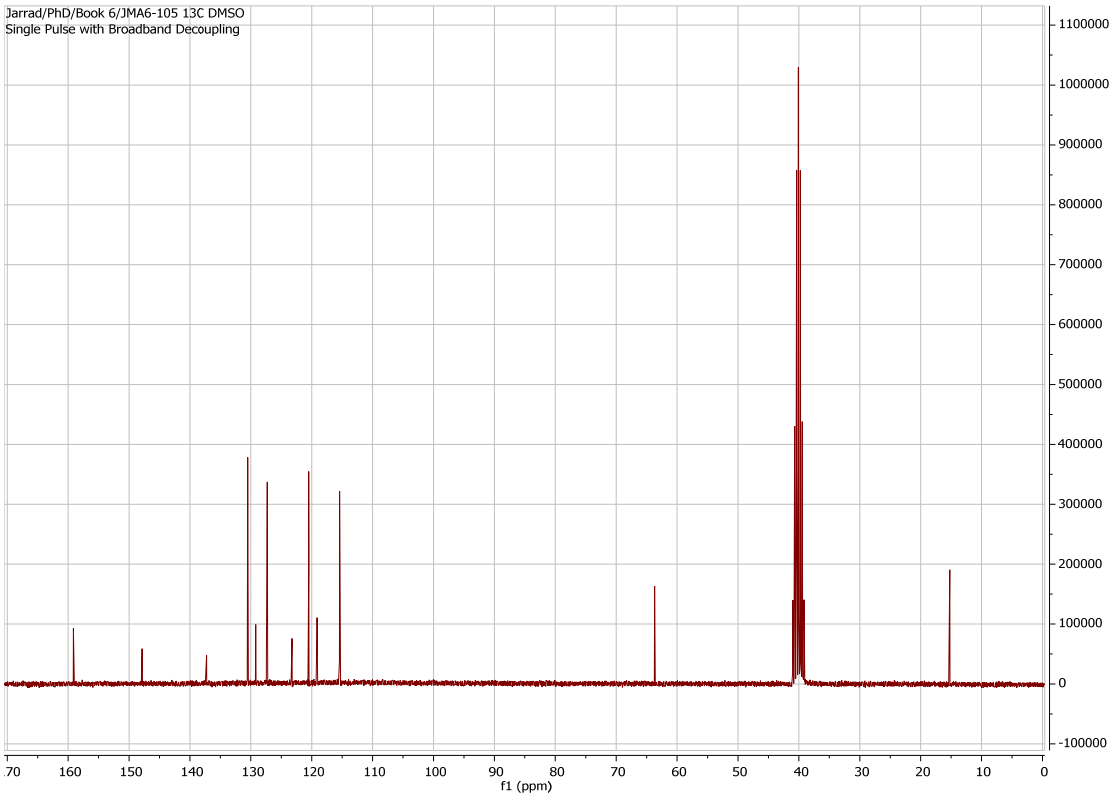

JMA6-095/23
JMA6-095 19F DMSO
F19 DMSO {C:\Data_500\Jarrad} nmr 10
f1 (ppm)



4-(4-methoxyphenyl)-1-phenyl-1H-1,2,3-triazole 13c

IR (thin film) υ, cm^{-1} 3201, 2954, 2100, 1578, 1100, 920, 650, 340; mp 164.7 °C; ^{1}H NMR (DMSO-d_6, 270 MHz) δ 9.19 (1H, s, Ar-H) 7.92-7.96 (2H, m, Ar-H) 7.84-7.87 (2H, m, Ar-H) 7.60-7.66 (2H, m, Ar-H) 7.48-7.54 (1H, m, Ar-H) 7.03-7.06 (2H, m, Ar-H) 4.08 (2H, q, J=8.1 Hz, CH_2) 1.35 (3H, t, J = 8.1 Hz, CH_3); ^{13}C NMR (DMSO-d_6, 67.5 MHz) δ 159.1, 147.9, 137.3, 130.5, 129.2, 127.3, 123.1, 120.5, 119.1, 115.4, 63.7, 15.2; HRMS calced for $C_{16}H_{15}N_3O$ $[MH]^+$ 266.12879, found 266.12862.




Jarrad/PhD/Book 6/JMA6-105 13C DMSO
Single Pulse with Broadband Decoupling
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1000000
900000
800000
700000
600000
500000
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100000
0
-100000
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90
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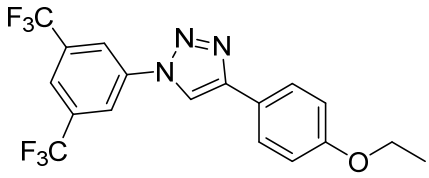
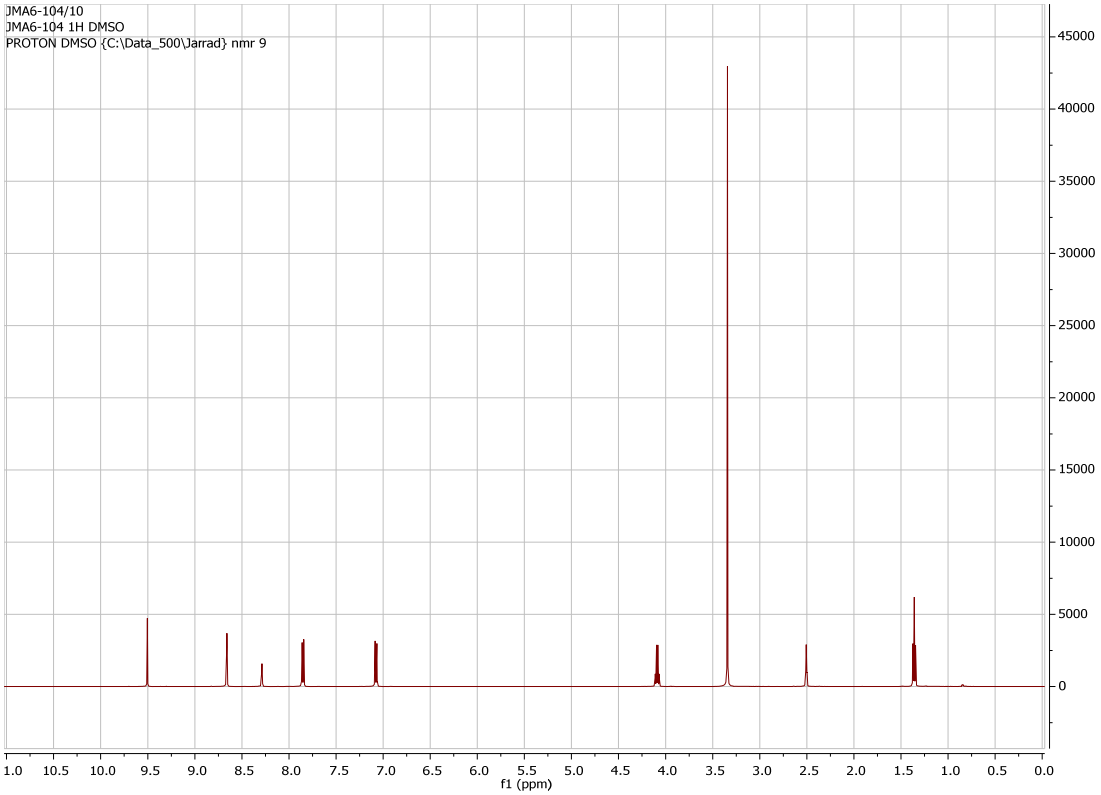
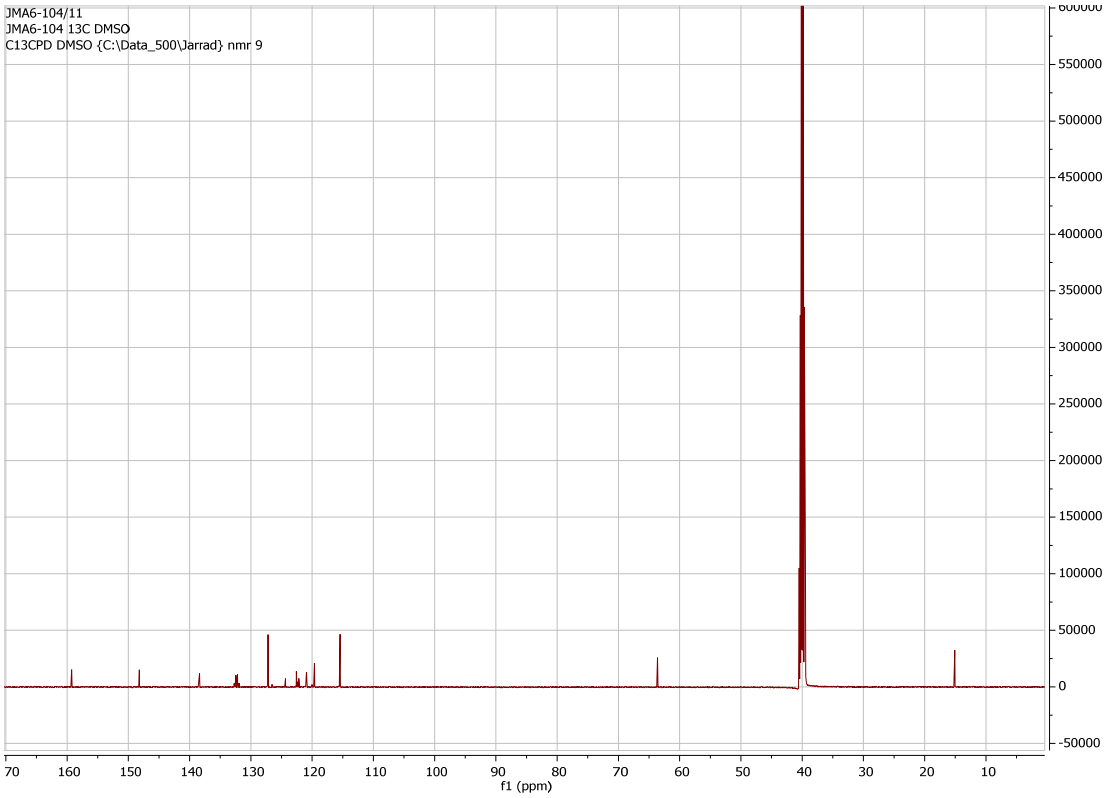
70
60
50
40
30
20
10
0
f1 (ppm)

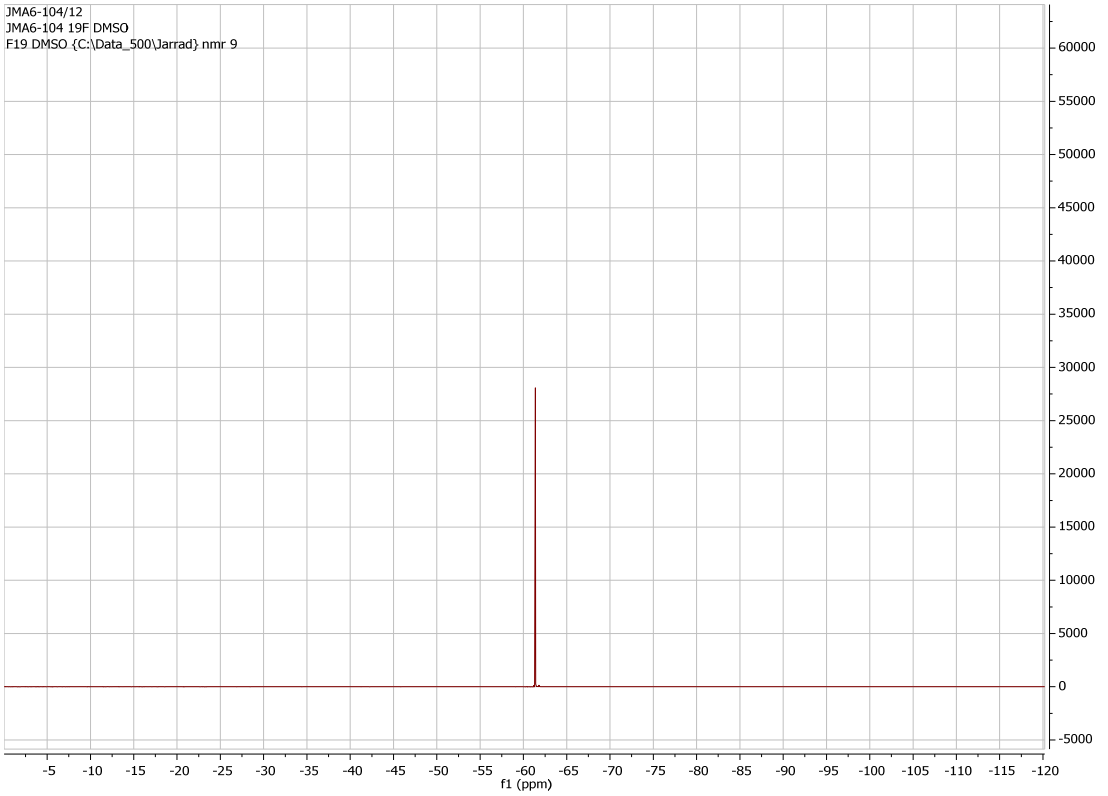


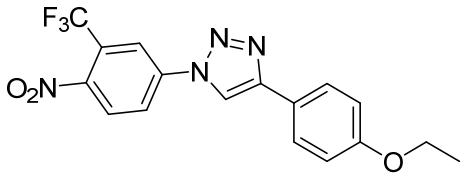
1-[3,5-bis(trifluoromethyl)phenyl]-4-(4-methoxyphenyl)-1H-1,2,3-triazole 13g

IR (thin film) υ, cm^{-1} 3054, 2245, 1604, 1348, 1001, 546, 482; mp 141.3 °C; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 9.50 (1H, s, Ar-*H*) 8.66 (2H, s, Ar-*H*) 8.29 (1H, s, Ar-*H*) 7.85 (2H, d, J = 10 Hz, Ar-*H*) 7.08 (2H, d, J = 5 Hz, Ar-*H*) 4.09 (2H, q, J = 5 Hz, CH_2) 1.36 (3H, t, J = 5 Hz, CH_3); ^{13}C NMR (DMSO-d_6, 125 MHz): δ 159.3, 148.2, 138.4, 132.4 (q, J=37.5 Hz) 127.2, 123.3 (q, J=275 Hz) 122.6, 122.4, 120.9, 119.6, 115.4, 63.6, 15.1; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.4; HRMS calced for $C_{18}H_{13}F_6N_3O$ $[MH]^+$ 402.10356, found 402.10316.



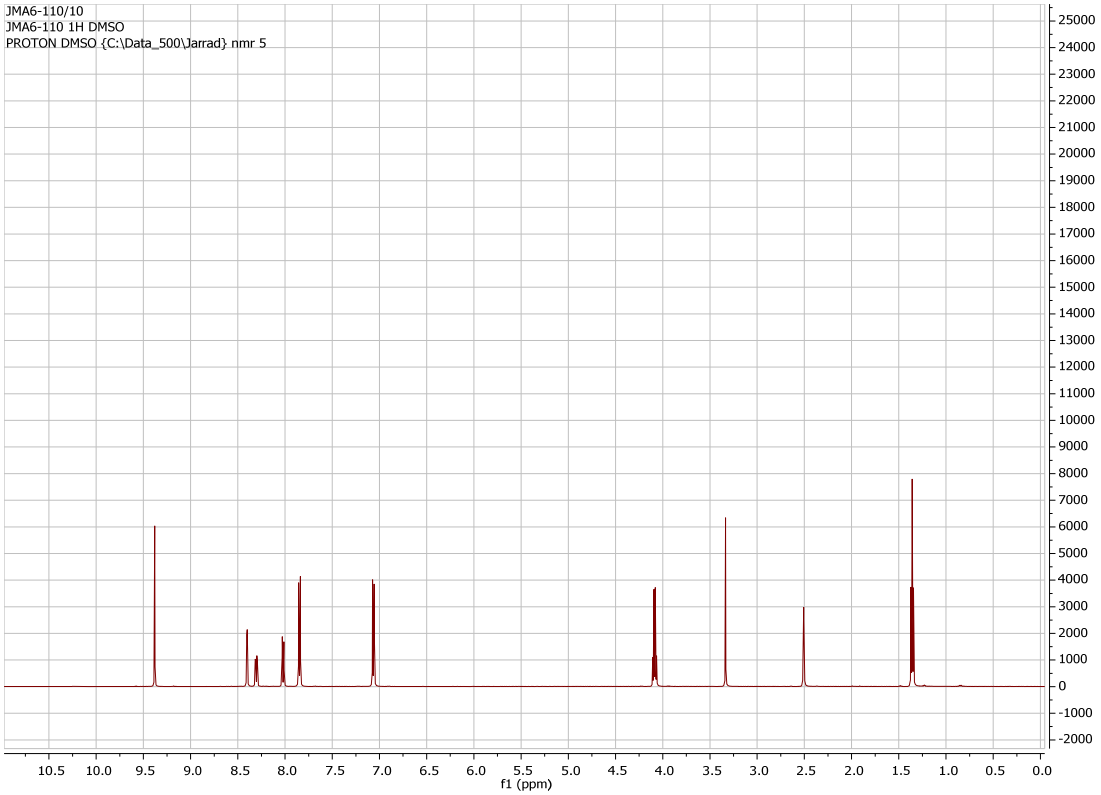

JMA6-104/11
JMA6-104 13C DMSO
C13CPD DMSO {C:\Data_500\Jarrad} nmr 9
f1 (ppm)

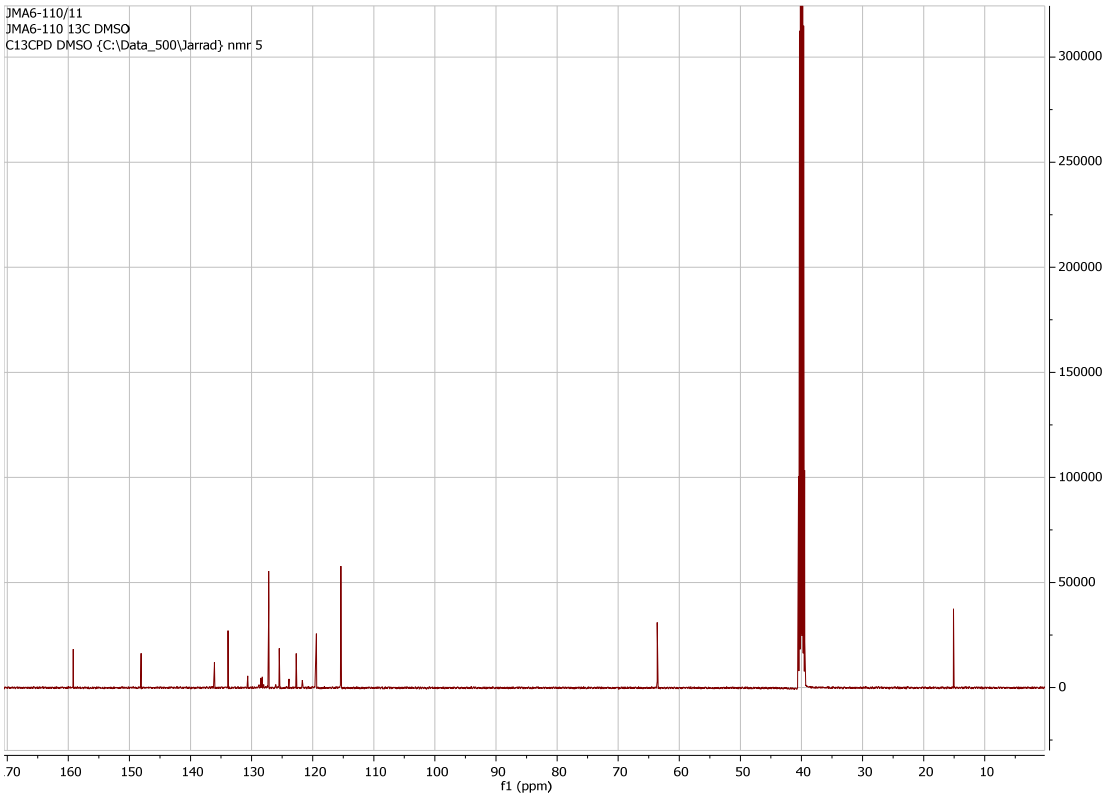

JMA6-104/12
JMA6-104 19F DMSO
F19 DMSO {C:\Data_500\Jarrad} nmr 9
f1 (ppm)



4-(4-methoxyphenyl)-1-[4-nitro-3-(trifluoromethyl)phenyl]-1H-1,2,3-triazole 13k

IR (thin film) υ, cm^{-1} 3189, 2617, 1476, 1241, 982, 754, 498, 354; mp 160.2 °C; ^{1}H NMR (DMSO-d_6, 270 MHz) δ 9.38 (1H, s, Ar-*H*) 8.40 (1H, s, Ar-*H*) 8.29-8.32 (1H, m, Ar-*H*) 8.02 (1H, d, J = 10 Hz, Ar-*H*) 7.84-7.86 (2H, m, Ar-*H*) 7.06-7.07 (2H, m, Ar-*H*) 4.09 (2H, d, J = 10 Hz, CH_2) 1.36 (3H, t, J = 5 Hz, CH_3); ^{13}C NMR (DMSO-d_6, 125 MHz) δ 159.2, 148.1, 136.1, 133.9, 130.6, 128.4 (q, $J^{2}_{C\text{-}F}$=25 Hz) 127.2, 125.5, 122.9 (q, $J^{1}_{C\text{-}F}$=275 Hz) 122.7, 119.5, 119.4, 115.4, 63.6, 15.1; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.5; HRMS calced for $C_{17}H_{13}F_3N_4O_3$ $[MH]^+$ 379.10125, found 379.10135.

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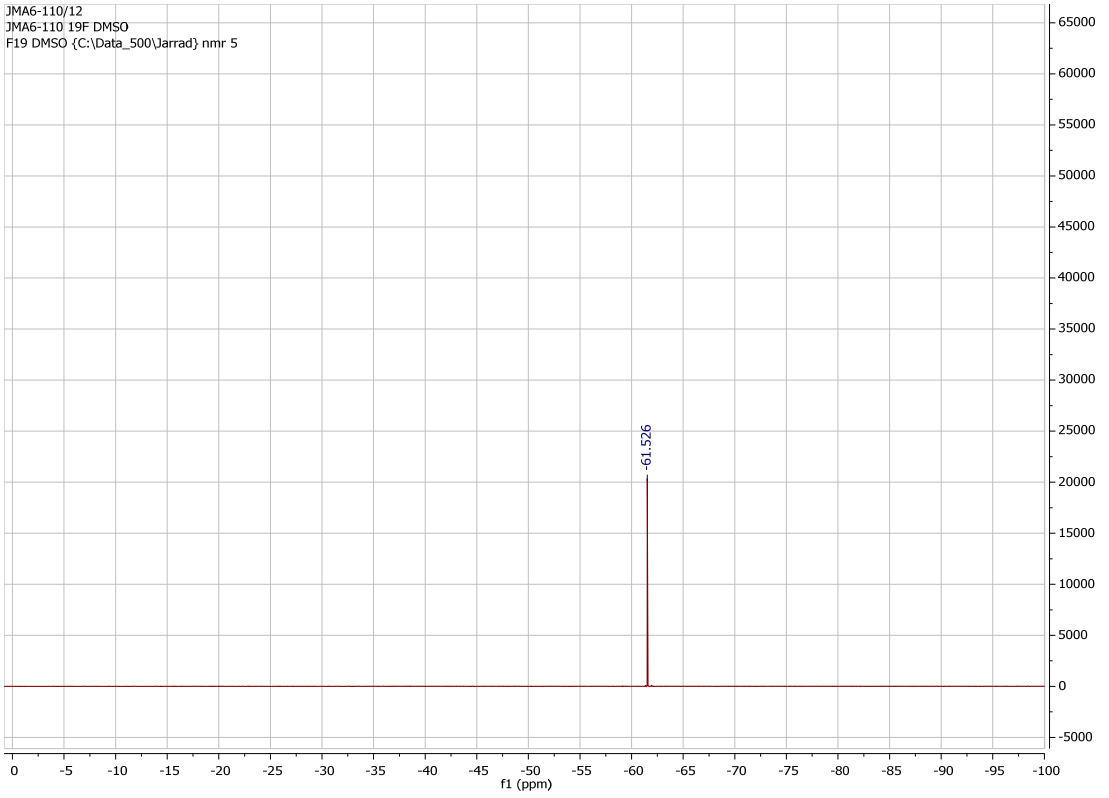
250000
200000
150000
100000
50000
0
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)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50000
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40000
35000
30000
25000
20000
15000
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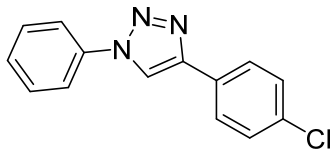
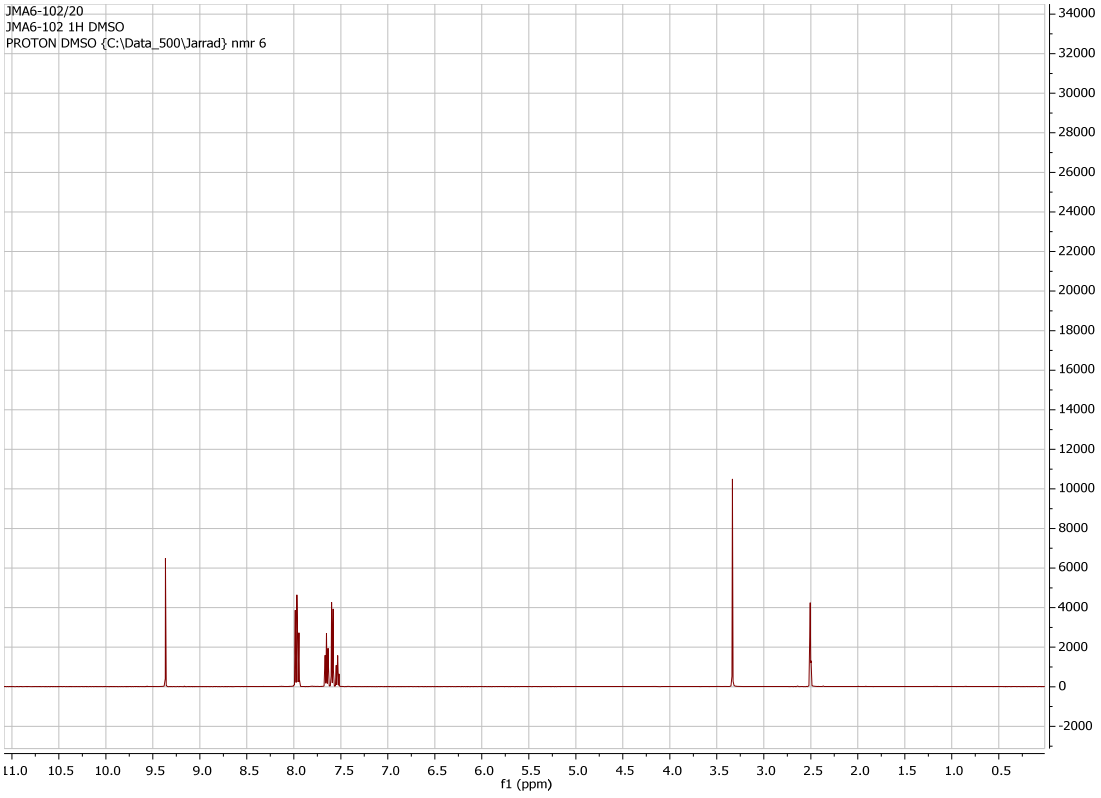
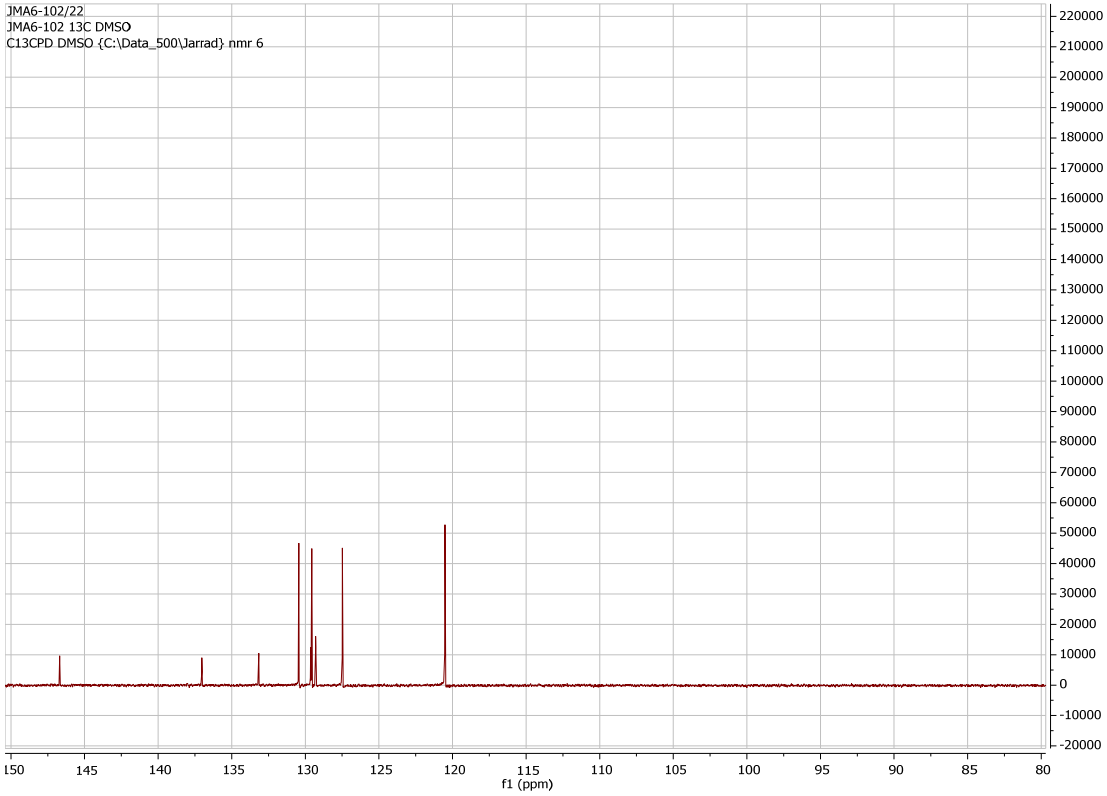
f1 (ppm)



4-(4-chlorophenyl)-1-phenyl-1H-1,2,3-triazole 13b

IR (thin film) υ, cm^{-1} 3604, 3102, 2567, 1301, 976, 644, 518; mp 218.4 °C; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 9.37 (1H, s, Ar-*H*) 7.94-7.98 (4H, m, Ar-*H*) 7.52-7.67 (5H, m, Ar-*H*); ^{13}C NMR (DMSO-d_6, 125 MHz): δ 146.7, 137.0, 133.2, 130.4, 129.6, 129.3, 127.5, 120.5; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.4; HRMS calced for $C_{14}H_{10}ClN_3$ $[MH]^+$ 256.06360, found 256.06370.




JMA6-102/22
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C13CPD DMSO {C:\Data_500\Jarrad} nmr 6
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200000
190000
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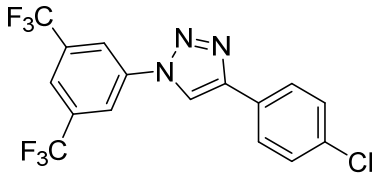
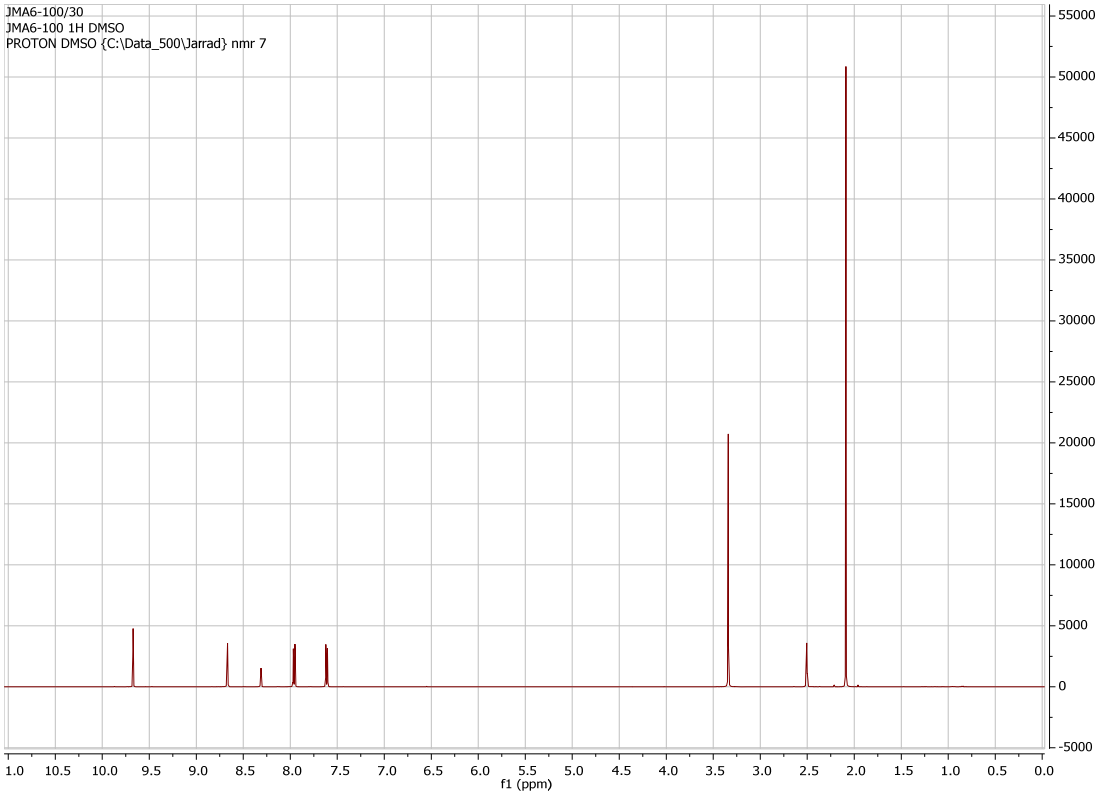
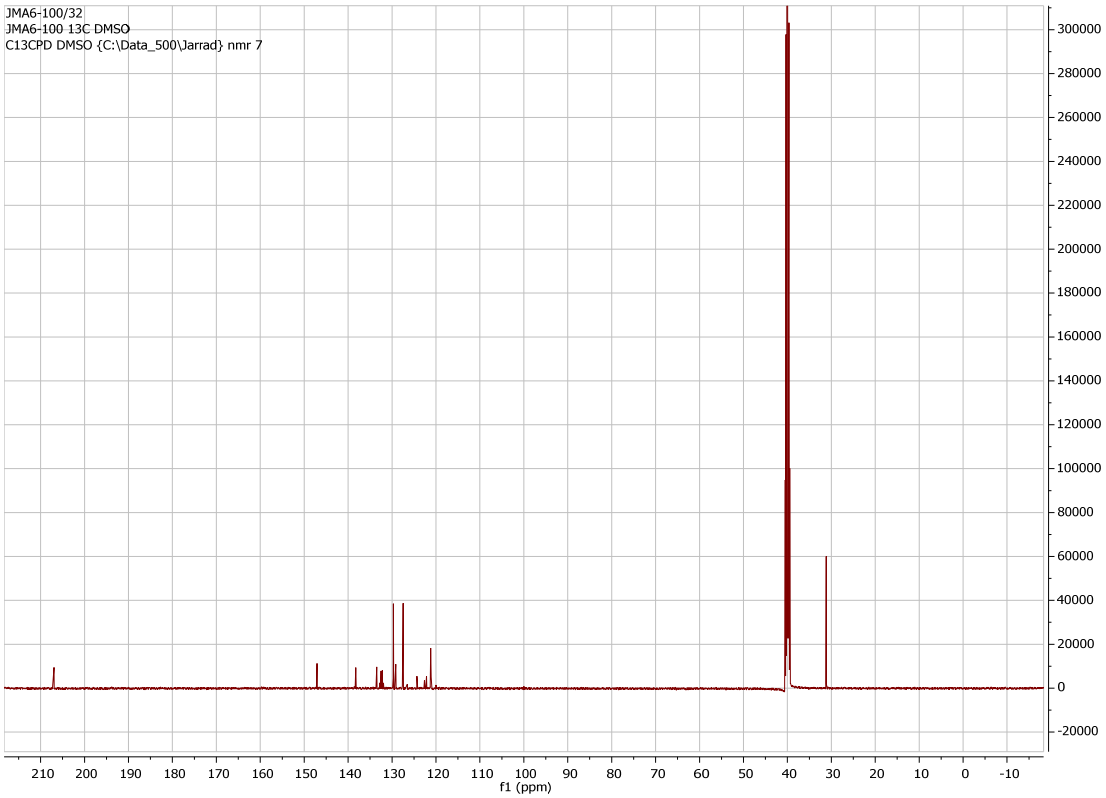
160000
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
-20000
150
145
140
135
130
125
120
115
110
105
100
95
90
85
80
f1 (ppm)

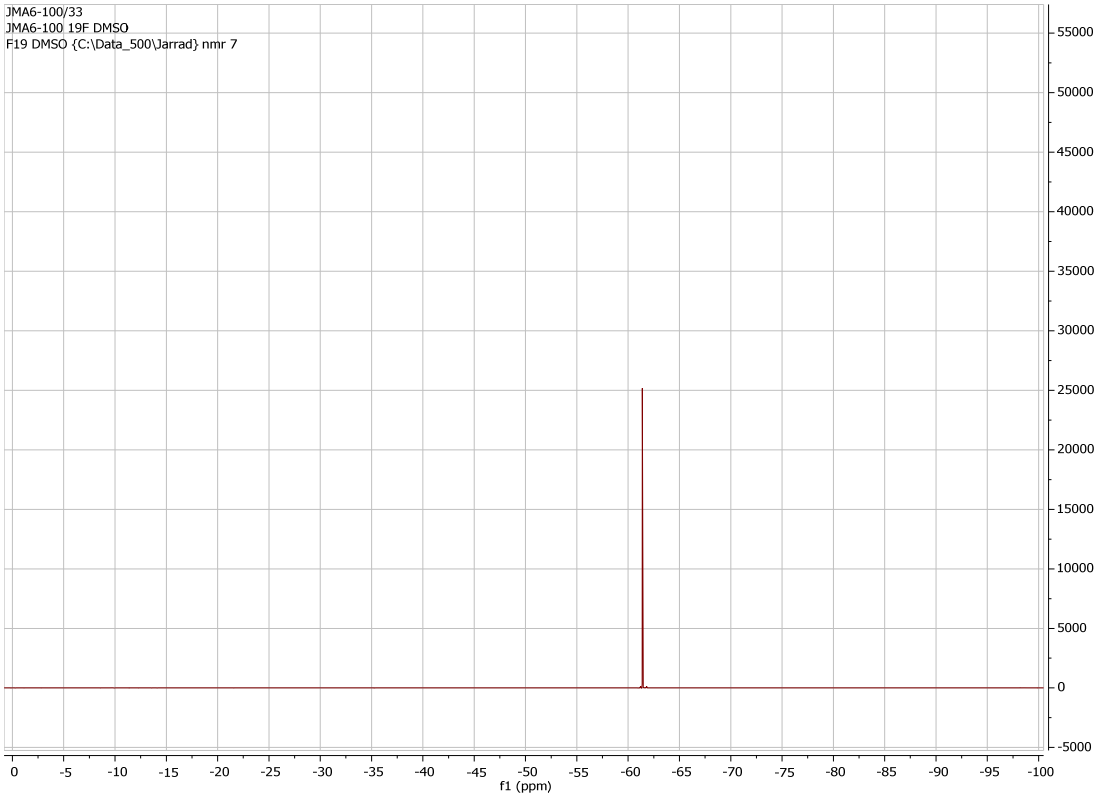


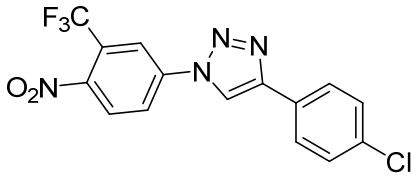
1-[3,5-bis(trifluoromethyl)phenyl]-4-(4-chlorophenyl)-1H-1,2,3-triazole 13f

IR (thin film) υ, cm^{-1} 3354, 2890, 2108, 1657, 1209, 983, 650, 421; mp 189.7 °C; ^{1}H NMR (DMSO-d_6, 500 MHz) δ 9.67 (1H, s, Ar-*H*) 8.67 (2H, s, Ar-*H*) 8.31 (1H, s, Ar-*H*) 7.95-7.97 (2H, m, Ar-*H*) 7.60-7.62 (2H, m, Ar-*H*); ^{13}C NMR (DMSO-d_6, 125 MHz) δ 206.9, 147.1, 138.3, 133.5, 132.4 (q, $J^{2}_{C\text{-}F}$ = 25 Hz) 129.7, 129.1, 127.5, 123.3 (q, $J^{1}_{C\text{-}F}$ = 275 Hz) 122.7, 121.2, 121.1; ^{19}F NMR (d_6-DMSO, 470 MHz): δ 61.4; HRMS calced for $C_{16}H_8ClF_6N_3$ $[MH]^+$ 392.03837, found 392.03837.



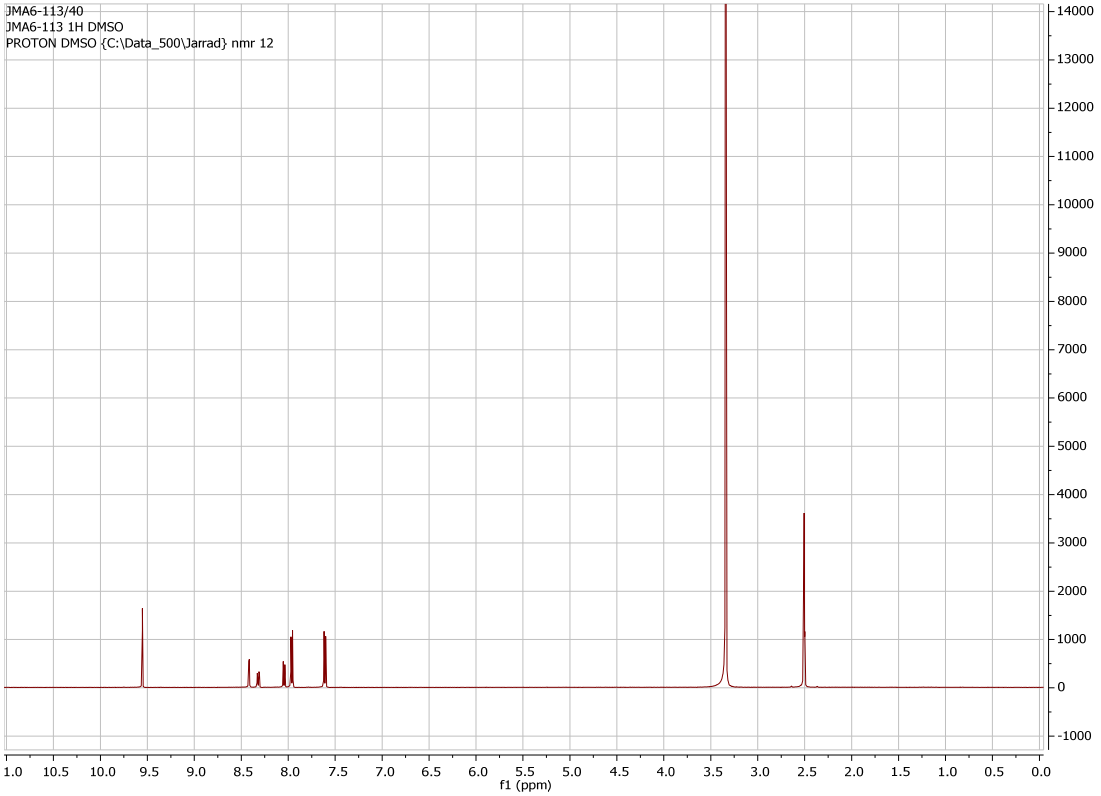

JMA6-100/32
JMA6-100 13C DMSO
C13CPD DMSO {C:\Data_500\Jarrad} nmr 7
300000
280000
260000
240000
220000
200000
180000
160000
140000
120000
100000
80000
60000
40000
20000
0
-20000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

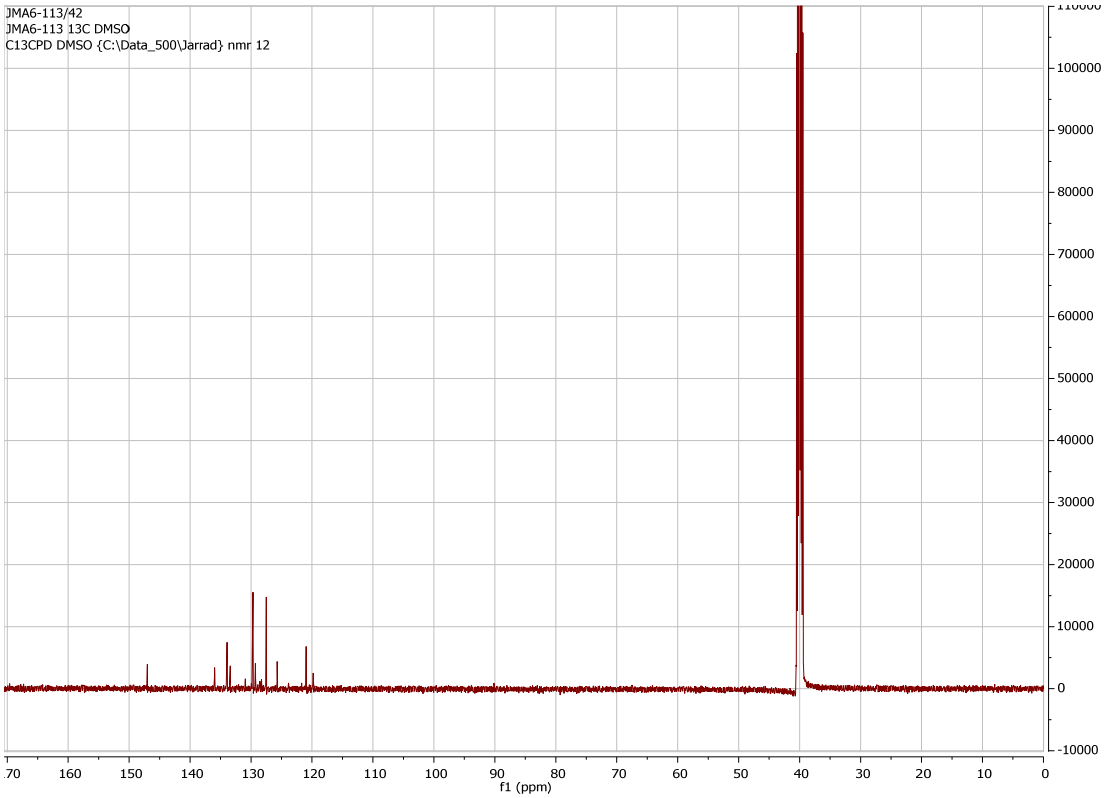

JMA6-100/33
JMA6-100 19F DMSO
F19 DMSO {C:\Data_500\Jarrad} nmr 7
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100
f1 (ppm)

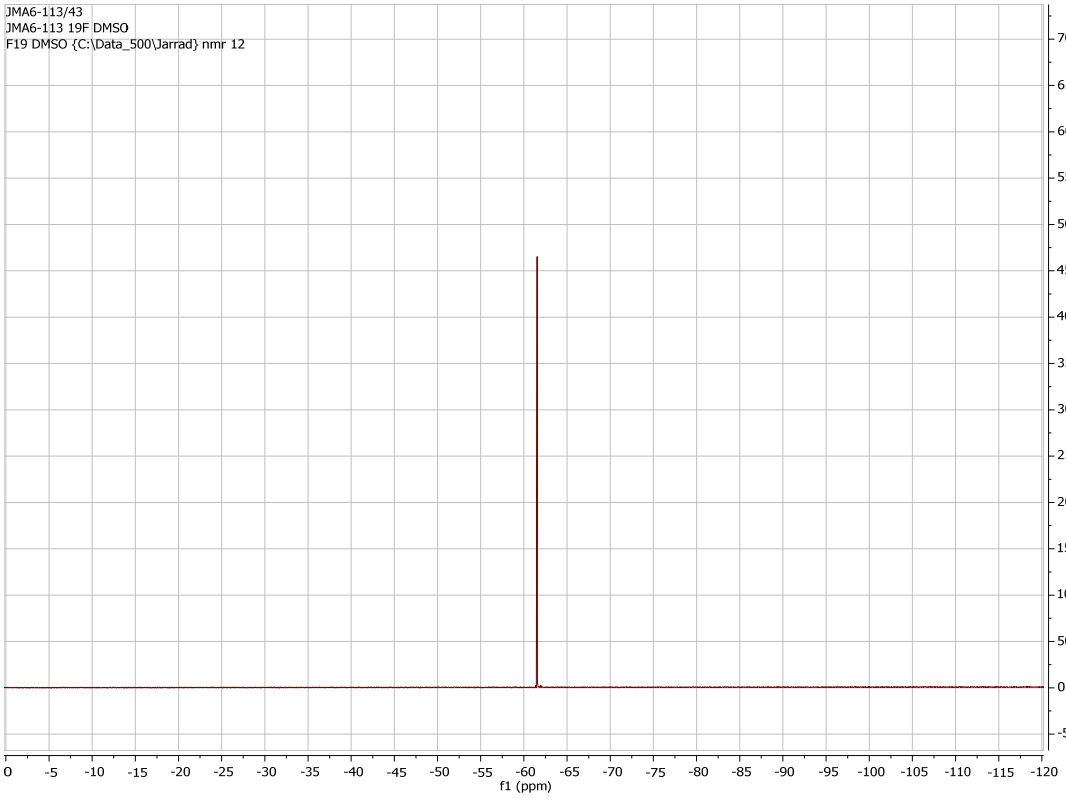


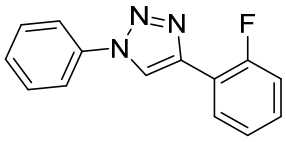
4-(4-chlorophenyl)-1-[4-nitro-3-(trifluoromethyl)phenyl]-1H-1,2,3-triazole 13j

IR (thin film) υ, cm^{-1} 3203, 2834, 2100, 1734, 1507, 1208, 985, 750, 430; mp 175.6 °C; ^{1}H NMR (DMSO-d_6, 500 MHz) δ 9.55 (1H, s, Ar-*H*) 8.42 (1H, d, J=5 Hz, Ar-*H*) 8.32 (1H, dd, J=2.5, 5 Hz, Ar-*H*) 8.05 (1H, d, J=5 Hz, Ar-*H*) 7.95-7.97 (2H, m, Ar-*H*) 7.60-7.62 (2H, m, Ar-*H*); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 147.0, 136.0, 133.9, 133.4, 130.9, 129.7, 129.3, 128.5 (q, $J^{2}_{C\text{-}F}$=25 Hz) 127.5, 125.7, 122.8 (q, $J^{1}_{C\text{-}F}$=275 Hz) 121.0, 119.8; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -61.5; HRMS calced for $C_{15}H_8ClF_3N_4O_2$ $[MH]^+$ 369.03606, found 369.03598.



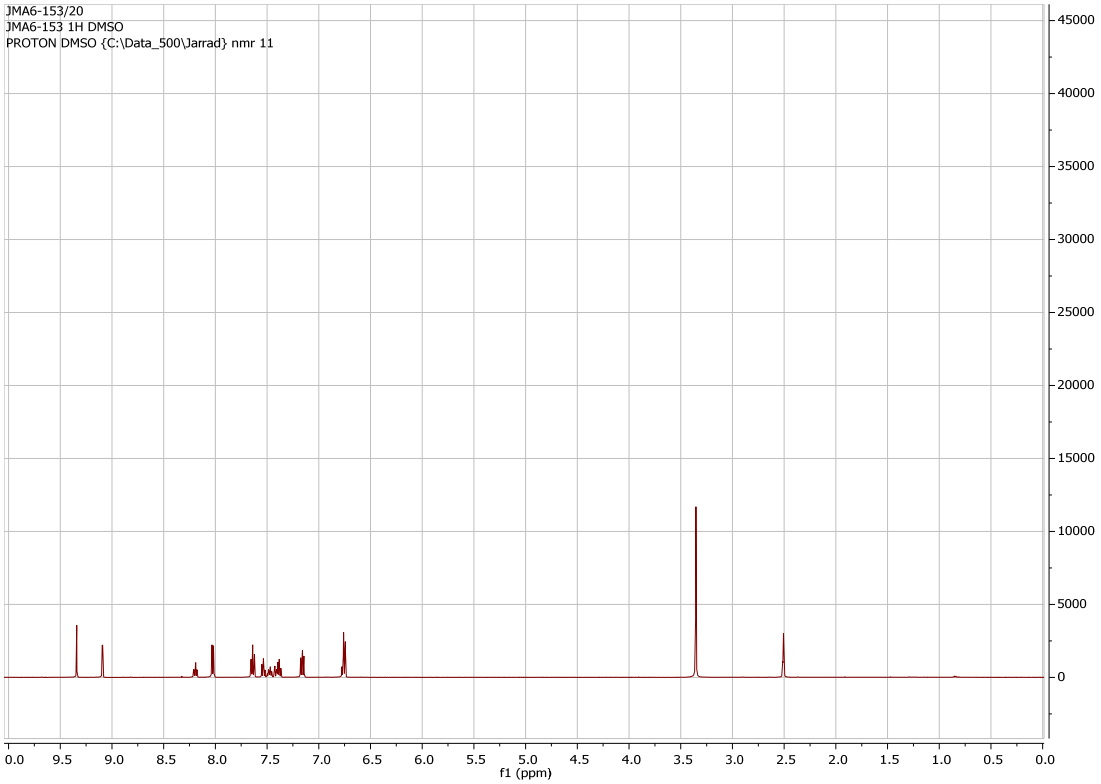

JMA6-113/42
JMA6-113 13C DMSO
C13CPD DMSO {C:\Data_500\Jarrad} nmr 12
f1 (ppm)

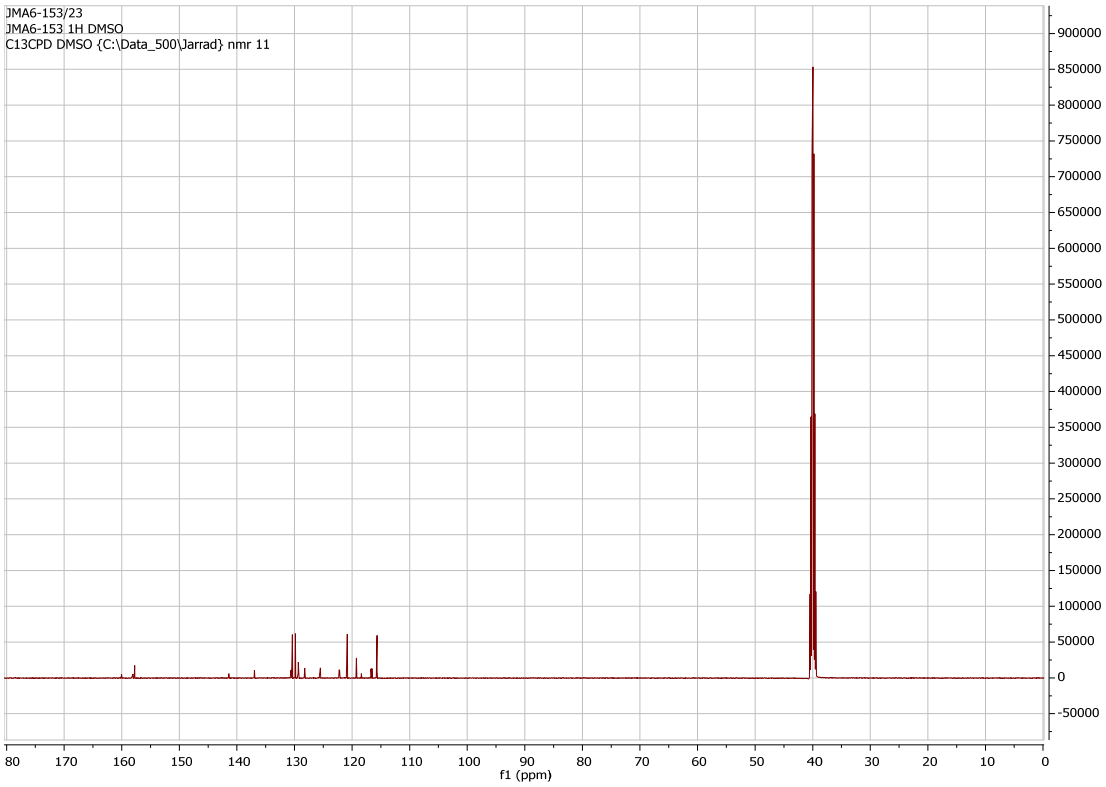

JMA6-113/43
JMA6-113 19F DMSO
F19 DMSO {C:\Data_500\Jarrad} nmr 12
f1 (ppm)

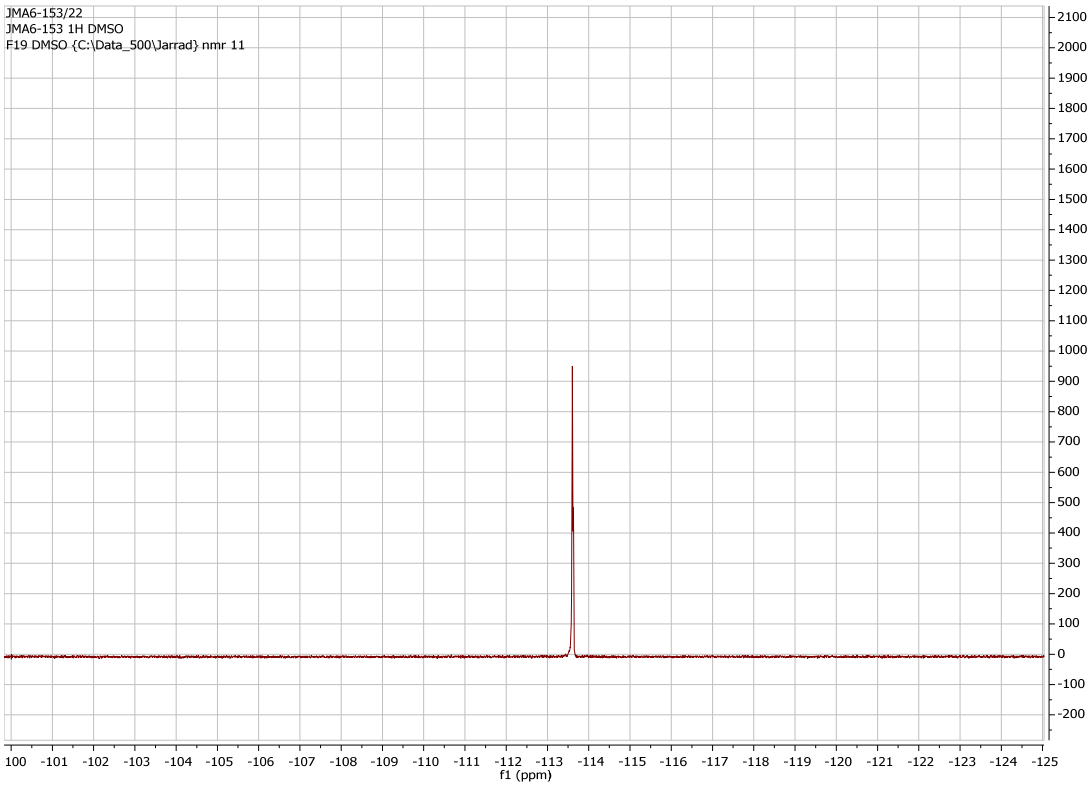


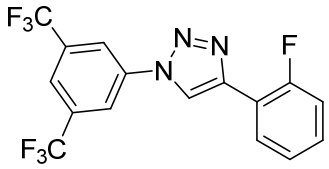
4-(2-fluorophenyl)-1-phenyl-1H-1,2,3-triazole 13a

IR (thin film) υ, cm^{-1} 2980, 2450, 1789, 1546, 1109, 983, 754, 589, 450; mp 88.3 °C; ^{1}H NMR (DMSO-d_6, 270 MHz) δ 9.34 (1H, s, Ar-*H*) 9.09 (1H, s, Ar-*H*) 8.18-8.21 (1H, m, Ar-*H*) 8.03 (1H, d, J = 10 Hz, Ar-*H*) 7.62-7.65 (1H, m, Ar-*H*) 7.37-7.55 (2H, m, Ar-*H*) 7.16-7.18 (1H, m, Ar-*H*) 6.74-6.78 (1H, m, Ar-*H*); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 159.0 (d, $J^{1}_{C\text{-}F}$ = 100 Hz) 157.8, 141.4, 137.0, 130.6 130.1 (d, $J^{2}_{C\text{-}F}$ = 60 Hz) 129.3 128.2, 125.5, 122.3, 120.8, 119.2, 116.6 (d, $J^{3}_{C\text{-}F}$ = 20 Hz) 115.7; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -113.6 ; HRMS calced for $C_{14}H_{10}FN_3$ $[MH]^+$ 240.09315, found 240.09284.



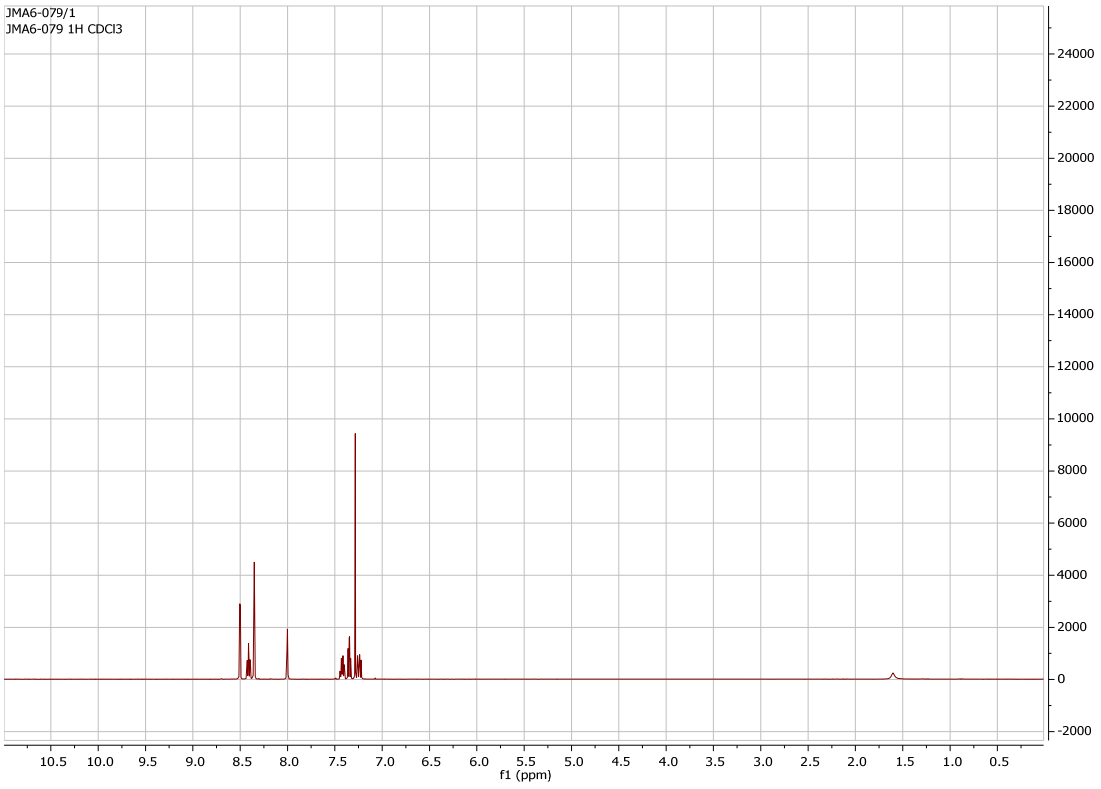

JMA6-153/23
JMA6-153 1H DMSO
C13CPD DMSO {C:\Data_500\Jarrad} nmr 11
f1 (ppm)

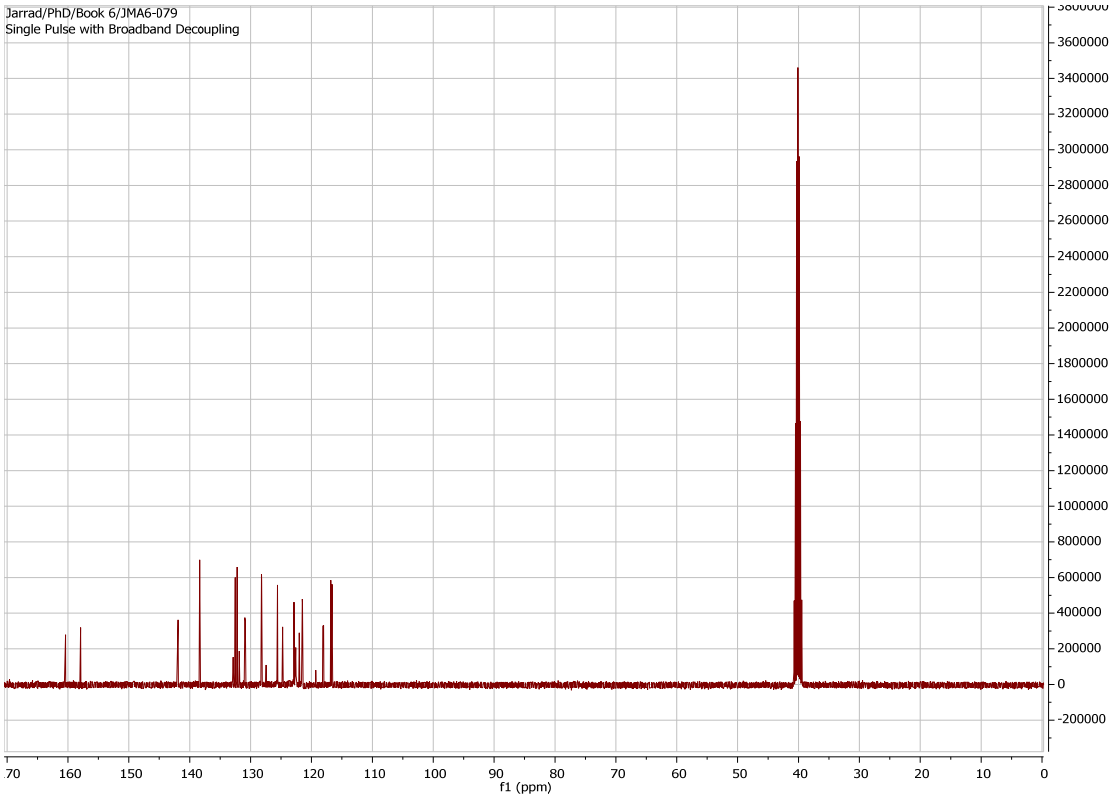

JMA6-153/22
JMA6-153 1H DMSO
F19 DMSO {C:\Data_500\Jarrad} nmr 11
f1 (ppm)

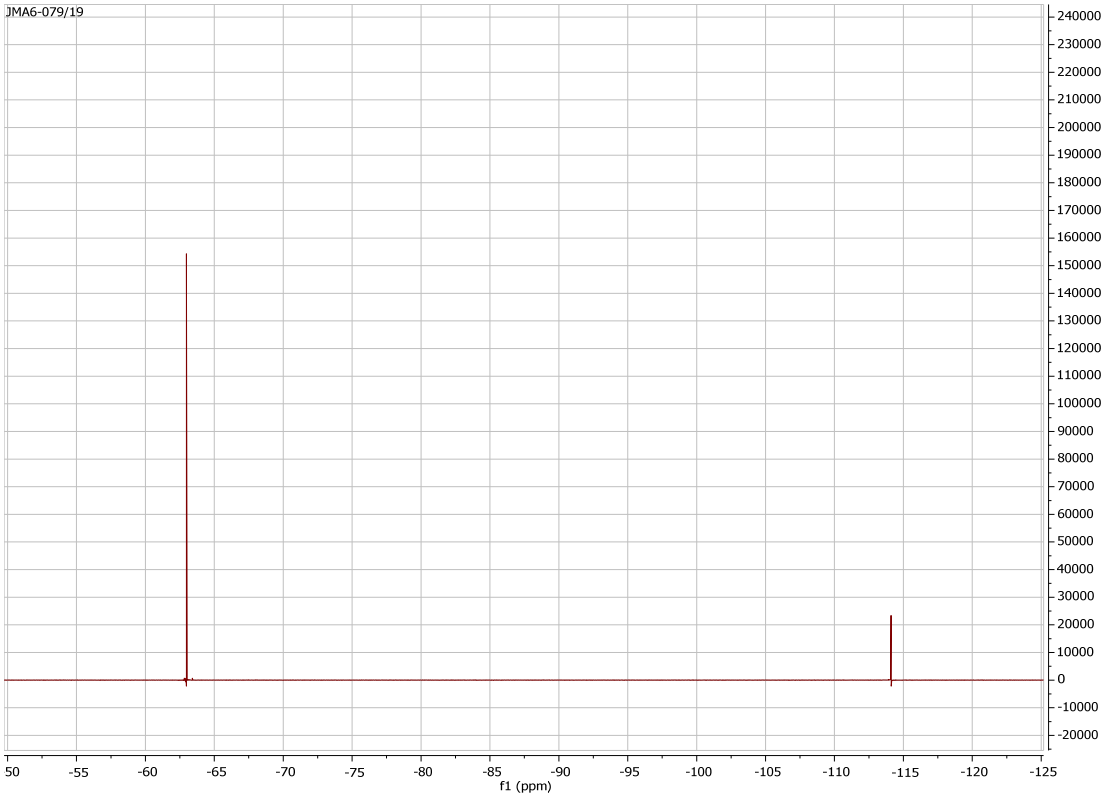


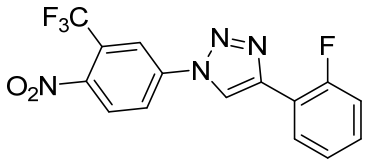
1-[3,5-bis(trifluoromethyl)phenyl]-4-(2-fluorophenyl)-1H-1,2,3-triazole 13e

IR (thin film) υ, cm^{-1} 3321. 2899, 1789, 1407, 1011, 746, 685, 534, 390; mp 168.1 °C; ^{1}H NMR ($CDCl_3$-d_3, 500 MHz) δ 8.50 (1H, d, J = 3 Hz, Ar-*H*) 8.41 (1H, td, J = 1.5, 7.5 Hz, Ar-*H*) 8.35 (2H, s, Ar-*H*) 8.00 (1H, s, Ar-*H*) 7.40-7.45 (1H, m, Ar-*H*) 7.46 (1H, td, J = 1.5, 7.5 Hz, Ar-*H*) 7.22-7.26 (1H, m, Ar-*H*); ^{13}C NMR (DMSO-d_6, 125 MHz) δ 160.4, 157.9, 141.9, 138.4, 132.4 (q, $J^{2}_{C\text{-}F}$ = 37.5 Hz) 130.9, 128.2, 125.6, 123.4 (q, $J^{1}_{C\text{-}F}$ = 337.5 Hz) 123.0 (d, $J^{1}_{C\text{-}F}$ = 12.5 Hz) 122.6, 121.5, 118.1 (d, $J^{2}_{C\text{-}F}$ = 12.5 Hz) 116.7 (d, $J^{1}_{C\text{-}F}$ = 25 Hz); ^{19}F NMR (d_6-DMSO, 470 MHz): δ -63.0, -114.1; HRMS calced for $C_{16}H_8F_7N_3$ $[MH]^+$ 376.06792, found 376.06813.



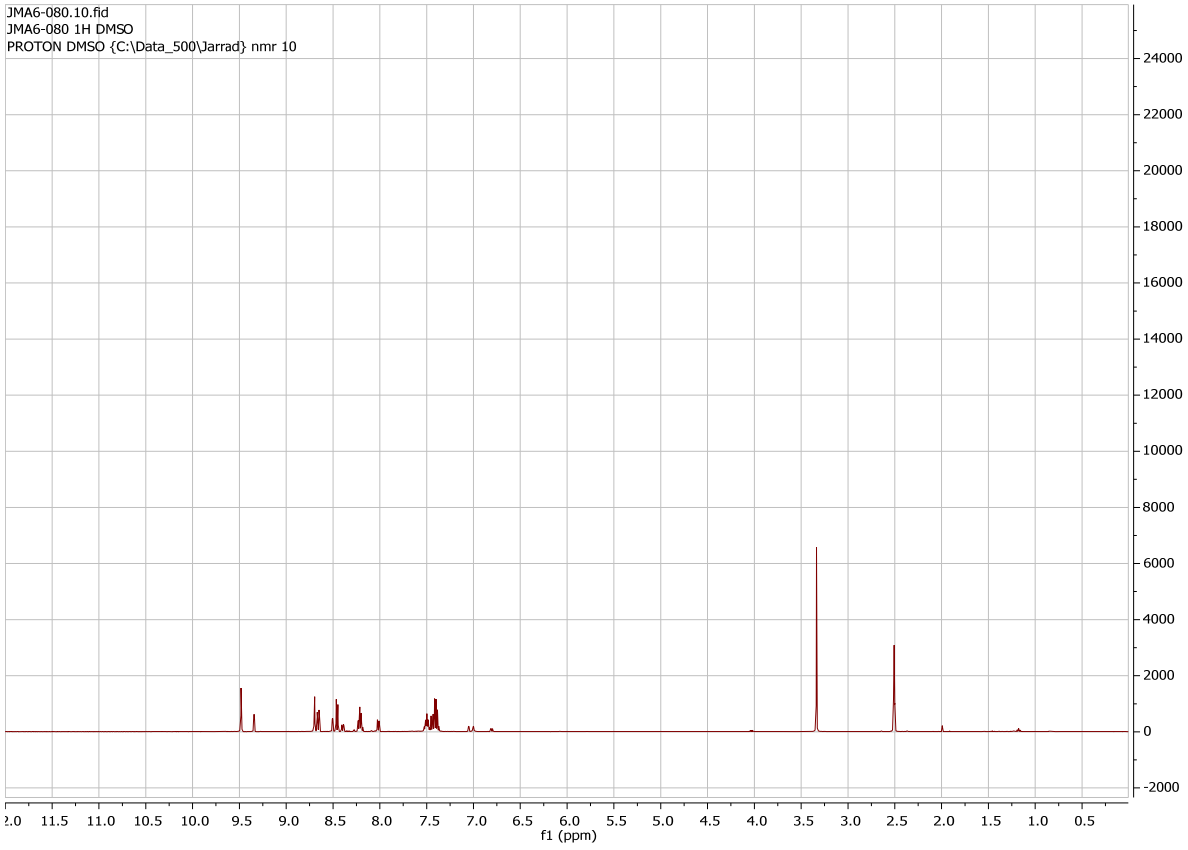

Jarrad/PhD/Book 6/JMA6-079
Single Pulse with Broadband Decoupling
f1 (ppm)

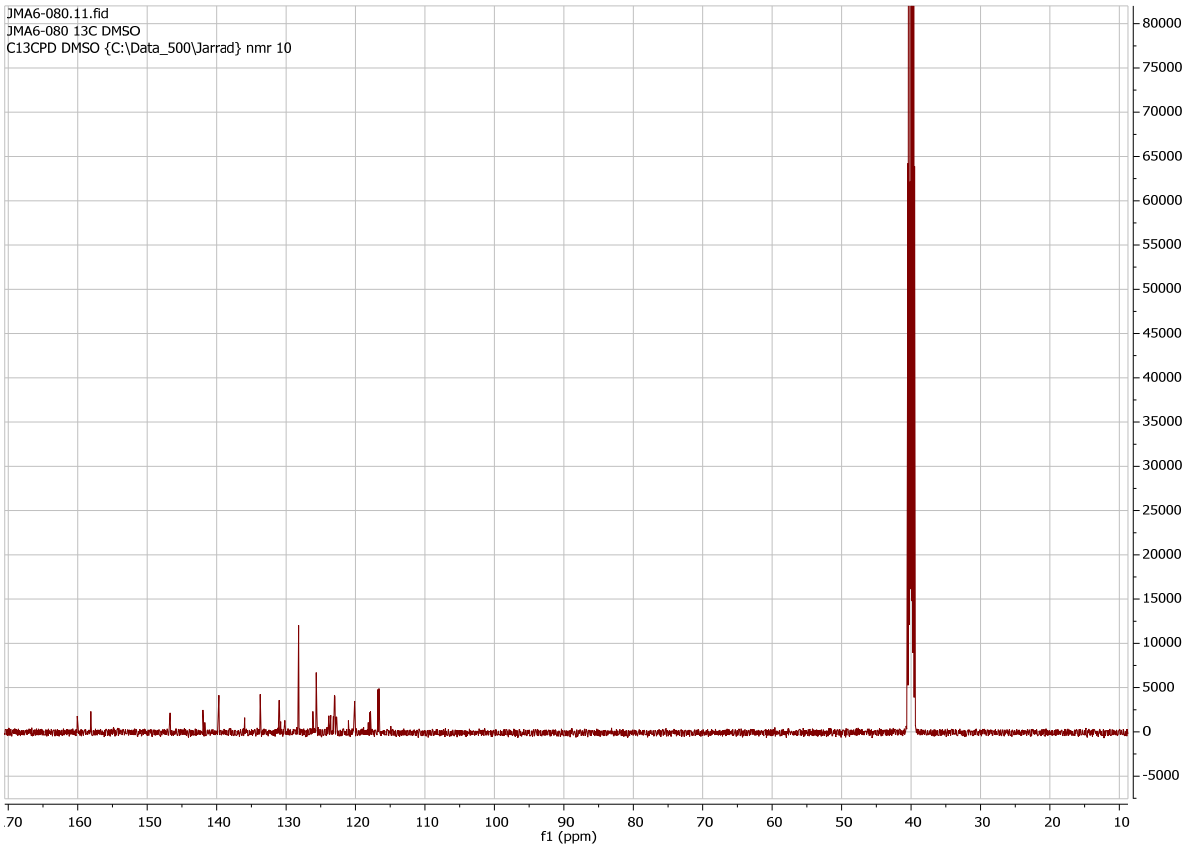

JMA6-079/19
f1 (ppm)

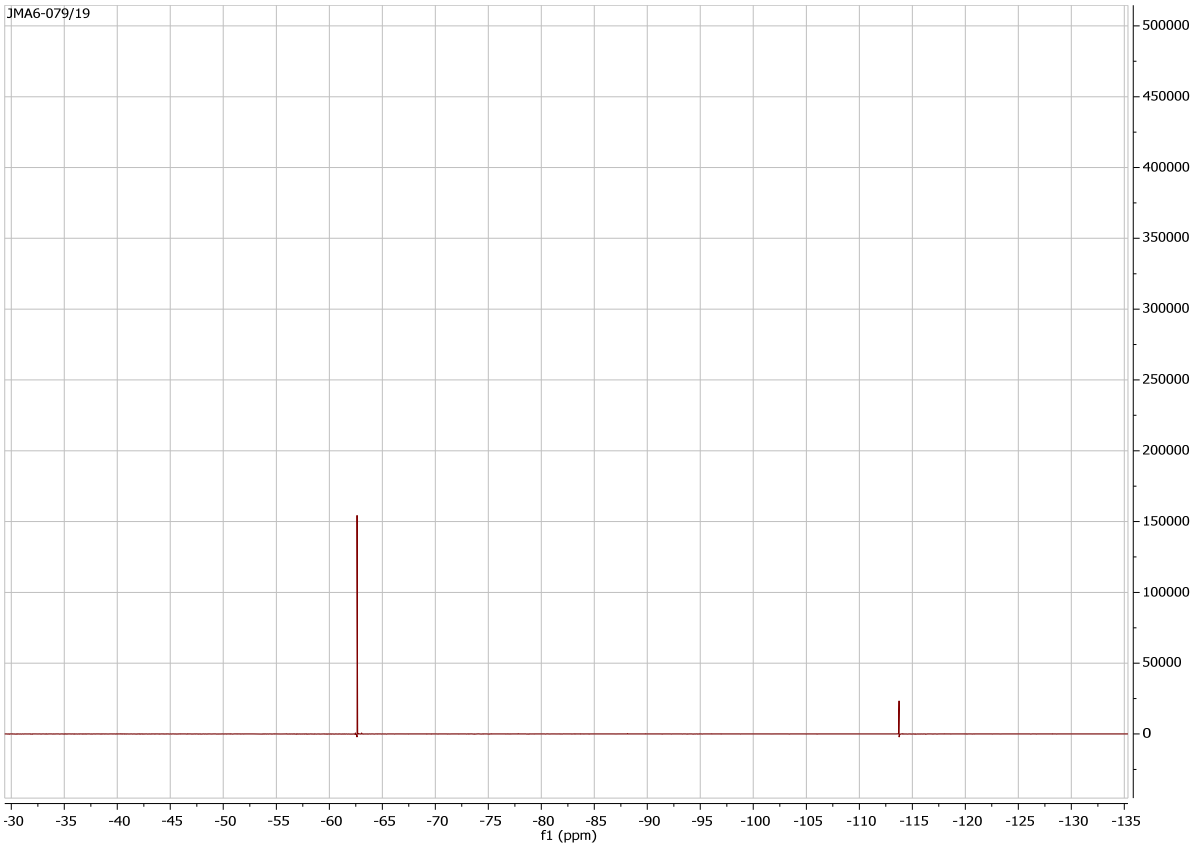


4-(2-fluorophenyl)-1-[4-nitro-3-(trifluoromethyl)phenyl]-1H-1,2,3-triazole 13i

IR (thin film) υ, cm^{-1} 3320, 2700, 1899, 1726, 1340, 1100, 988, 712, 542, 400; mp 173.2 °C; ^{1}H NMR (DMSO-d_6, 500 MHz): δ 9.48 (1H, s, Ar-*H*) 8.65-8.70 (2H, m, Ar-*H*) 8.45-8.50 (1H, m, Ar-*H*) 8.20-8.22 (1H, m, Ar-*H*) 7.38-7.52 (3H, m, Ar-*H*); ^{13}C NMR (DMSO-d_6, 125 MHz): δ 160.1, 158.1, 146.7, 142.0, 139.7, 133.7, 131.0, 128.2, 125.7, 125.6, 123.7 (q, $J^{2}_{C\text{-}F}$ = 25 Hz) 123.0, 120.1, 117.9 (d, $J^{3}_{C\text{-}F}$ = 12.5 Hz) 116.7 (d, $J^{2}_{C\text{-}F}$ = 25 Hz) ; ^{19}F NMR (d_6-DMSO, 470 MHz): δ -62.6, -113.7; HRMS calced for $C_{15}H_8F_4N_4O_2$ $[MH]^+$ 353.06561, found 353.06694.

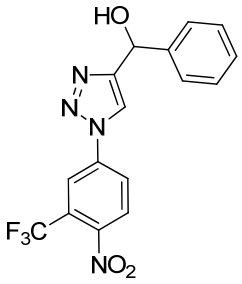



JMA6-080.11.fid
JMA6-080 13C DMSO
C13CPD DMSO {C:\Data_500\Jarrad} nmr 10
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
70 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


JMA6-079/19
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
-30 -35 -40 -45 -50 -55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135
f1 (ppm)

Example of procedure to evaluate compound solubility.

Solubility was evaluated by eye only. A representative procedure for this process is given below using compound **6b** as an example.



6b

Mwt: 364 g/mol

Therefore, a 100 μM solution (10 mL) of 0.5%DMSO/water requires 0.36 mg of **6b** in 50 μL of DMSO to be introduced to 9.95 mL of water.

A solution of **6b** (7.2 mg) in 1 mL of DMSO was prepared and 50 μL of this solution was added to 9.95 mL of water. The resulting solution was then evaluated by eye to determine if the compound was soluble.