Supporting Information for:

Intramolecular Hydrogen Bonding-Assisted Cyclocondensation of *α*-Diazoketones with Various Amines: A Strategy for Catalytic Wolff 1,2,3-Triazole Synthesis

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I. General Information

All reagents were purchased from commercial sources and used without treatment, unless otherwise indicated. The products were purified by column chromatography over silica gel. ^{1}H NMR and ^{13}C NMR spectra were recorded at 25 °C at 300MHz or 400MHz or 500 MHz and 100MHz or 125 MHz, respectively, with TMS as internal standard. Mass spectra were recorded on BRUKER AutoflexIII Smartbeam MS-spectrometer. High resolution mass spectra (HRMS) were recorded on Bruck microTof by using ESI method.

II. Synthesis and analytical data for α-diazo-β-oxoamides and 1,2,3-trizaoles.

Synthesis of *α*-diazo-1,3-dicarbonyl compounds (with **1a** as an example): To a solution of 3-oxo-*N*-phenylbutanamide (30 mmol) and triethylamine (60 mmol) in 50 mL of acetonitrile was added tosyl azide (33 mmol). The solution was stirred for 12 h at room temperature until the 3-oxo-*N*-phenylbutanamide disappeared (monitored by TLC). The reaction mixture was then treated with 250 mL brine, and extracted with dichloromethane (2 × 100 mL). The combined organic layer was washed with brine (3 × 100 mL), dried over $MgSO_4$ and filtered. The filtrate was concentrated in *vacuum*, and purified by silica gel column chromatography to give **1a** as a yellowish solid.

2-diazo-3-oxo-*N*-phenylbutanamide

(**1a**) Yellowish solid; mp 118-120 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 10.17 (s, 1H), 7.57-7.60 (m, 2H), 7.30-7.36 (m, 2H), 7.09-7.14 (m, 1H), 2.42 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 125Hz) δ 189.8, 158.0, 137.8, 128.9, 124.3, 119.9, 78.3, 26.6. **HRMS** Calcd for $C_{10}H_{10}N_3O_2$ ($[M + H]^+$) 204.0773; Found 204.0776.

2-diazo-3-oxo-*N*-(*o*-tolyl)butanamide

(**1b**) Yellow solid; mp 133-135 °C. **^{1}H-NMR** ($CDCl_3$, 500 MHz) δ 10.13 (s, 1H), 8.12 (d, J = 10.0Hz, 1H), 7.18-7.22 (m, 2H), 7.03-7.06 (m, 1H), 2.40 (s, 3H), 2.35 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 125Hz) δ 190.0, 158.0, 136.3, 130.3, 127.7, 126.6, 124.4, 121.2, 78.5, 26.6, 17.9. **HRMS** Calcd for $C_{11}H_{12}N_3O_2$ ($[M + H]^+$) 218.0930; Found

218.0912.

2-diazo-3-oxo-*N*-(*p*-tolyl)butanamide

(**1c**) Yellow solid; mp 137-139 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 10.08 (s, 1H), 7.45-7.48 (m, 2H), 7.09-7.16 (m, 2H), 2.41 (s, 3H), 2.32 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 125Hz) δ 189.8, 157.8, 135.2, 133.9, 129.4, 119.9, 78.2, 26.6, 20.8. **HRMS** Calcd for $C_{11}H_{12}N_3O_2$ ([M + H]$^+$) 218.0930; Found 218.0916.

2-diazo-*N*-(2-methoxyphenyl)-3-oxobutanamide

(**1d**) Yellow solid; mp 138-140 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 10.61 (s, 1H), 8.34-8.37 (m, 1H), 7.03-7.09 (m, 1H), 6.89-6.98 (m, 2H), 3.94 (s, 3H), 2.41 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 189.5, 157.8, 148.5, 127.9, 124.0, 120.8, 120.0, 110.1, 78.4, 55.8, 26.6. **HRMS** Calcd for $C_{11}H_{12}N_3O_3$ ([M + H]$^+$) 234.0879; Found 234.0858.

2-diazo-*N*-(4-methoxyphenyl)-3-oxobutanamide

(**1e**) Yellow solid; mp 111-113 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 10.03 (s, 1H), 7.47-7.50 (m, 2H), 6.85-6.88 (m, 2H), 3.79 (s, 3H), 2.41 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 189.7, 157.6, 156.1, 130.9, 121.3, 113.8, 77.9, 55.2, 26.4. **HRMS** Calcd for $C_{11}H_{12}N_3O_3$ ([M + H]$^+$) 234.0879; Found 234.0866.

N-(2-chlorophenyl)-2-diazo-3-oxobutanamide

(**1f**) Yellow solid; mp 112-114 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 10.71 (s, 1H), 8.38-8.42 (m, 1H), 7.38-7.41 (m, 1H), 7.24-7.29 (m, 1H), 7.02-7.08 (m, 1H), 2.43 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 125Hz) δ 189.5, 158.4, 135.0, 129.1, 127.4, 124.6, 123.2, 121.5, 78.5, 26.5. **HRMS** Calcd for $C_{10}H_9ClN_3O_2$ ($[M + H]^+$) 238.0383; Found 238.0367.

N-(4-chlorophenyl)-2-diazo-3-oxobutanamide

(**1g**) Yellow solid; mp 134-136 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 10.20 (s, 1H), 7.52-7.55 (m, 2H), 7.27-7.30 (m, 2H), 2.41 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 125Hz) δ 189.8, 158.1, 136.4, 129.1, 128.9, 121.1, 78.2, 26.6. **HRMS** Calcd for $C_{10}H_9ClN_3O_2$ ($[M + H]^+$) 238.0383; Found 238.0362.

2-diazo-*N*-(2,4-dimethylphenyl)-3-oxobutanamide

(**1h**) Yellow solid; mp 129-131 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 10.04 (s, 1H), 7.90-7.97 (m, 1H), 7.00-7.02 (m, 2H), 2.41 (s, 3H), 2.30 (s, 3H), 2.29 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 190.0, 157.8, 134.0, 133.7, 130.9, 127.7, 127.0, 121.3, 78.3, 26.5, 20.7, 17.7. **HRMS** Calcd for $C_{12}H_{14}N_3O_2$ ($[M + H]^+$) 232.1086; Found 232.1040.

2-diazo-3-oxo-*N*-(4-(trifluoromethyl)phenyl)butanamide

(**1i**) Yellow solid; mp 137-139 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 10.41 (s, 1H), 7.72 (d, *J* = 9.0Hz, 2H), 7.58 (d, *J* = 9.0Hz, 2H), 2.44 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 189.9, 158.6, 140.8, 129.6, 126.2, 125.4, 122.7, 119.6, 78.4, 26.6. **HRMS** Calcd for $C_{11}H_9F_3N_3O_2$ ($[M + H]^+$) 272.0647; Found 272.0611.

2-diazo-3-oxo-*N*-(pyridin-2-yl)butanamide

(**1j**) Yellowish solid; mp 155-158 °C. **^{1}H-NMR** ($CDCl_3$, 400 MHz) δ 10.61 (s, 1H), 8.31-8.32 (m, 1H), 8.14 (d, *J* = 8.4Hz, 1H), 7.66-7.70 (m, 1H), 7.02-7.05 (m, 1H), 2.41 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 188.9, 158.6, 151.0, 148.0, 138.0, 119.8, 114.0, 78.0, 26.6. **HRMS** Calcd for $C_9H_8N_4NaO_2$ ([M + Na]$^+$) 227.0545; Found 227.0690.

2-diazo-3-oxo-*N*-phenylhexanamide

(**1k**) Yellow solid; mp 68-70 °C. **^{1}H-NMR** ($CDCl_3$, 400 MHz) δ 10.26 (s, 1H), 7.56-7.61 (m, 2H), 7.29-7.36 (m, 2H), 7.08-7.14 (m, 1H), 2.60 (t, *J* = 7.6Hz, 2H), 1.72-1.79 (m, 2H), 1.02 (t, *J* = 7.6Hz, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 192.8, 158.3, 137.9, 128.9, 124.2, 119.8, 77.5, 40.8, 17.5, 13.4. **HRMS** Calcd for $C_{12}H_{14}N_3O_2$ ([M + H]$^+$) 232.1086; Found 232.1097.

2-diazo-3-oxo-*N*,3-diphenylpropanamide

(**1l**) Yellowish solid; mp 95-98 °C. **^{1}H-NMR** ($CDCl_3$, 400 MHz) δ 10.46 (s, 1H), 7.59-7.68 (m, 5H), 7.53 (t, *J* = 8.0Hz, 2H), 7.36 (t, *J* = 8.0Hz, 2H), 7.14 (t, *J* = 8.0Hz, 1H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 187.9, 158.5, 137.8, 136.5, 132.7, 129.0, 127.0, 124.4, 120.0, 77.6. **HRMS** Calcd for $C_{15}H_{12}N_3O_2$ ([M + H]$^+$) 266.0930; Found 266.0914.

2-diazo-5,5-dimethylcyclohexane-1,3-dione

(**S2**) Yellowish solid; mp 97-100 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 2.44 (s, 4H), 1.11

(s, 6H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 189.7, 75.2, 50.4, 31.0, 28.2. **HRMS** Calcd for $C_8H_{11}N_2O_2$ ($[M + H]^+$) 167.0821; Found 167.0802.

3-diazopiperidine-2,4-dione

(**S3**) White solid; mp 137-139 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 6.22 (s, 1H), 3.50 (t, *J* = 6.0Hz, 2H), 2.65 (t, *J* = 6.0Hz, 2H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 188.0, 163.5, 75.4, 36.9, 36.2. **HRMS** Calcd for $C_5H_6N_3O_2$ ($[M + H]^+$) 140.0460; Found 140.0433.

2-diazo-*N*-methyl-3-oxo-*N*-phenylbutanamide

(**S4**) Yellow oil; **^{1}H-NMR** (CDCl3, 300 MHz) δ 7.40-7.45 (m, 2H), 7.31-7.36 (m, 1H), 7.20 (d, *J* = 6.0Hz, 2H), 3.38 (s, 3H), 2.50 (s, 3H). **^{13}C-NMR** (CDCl3, 125Hz) δ 191.6, 160.6, 142.7, 130.0, 127.7, 126.0, 74.2, 38.2, 28.2. **HRMS** Calcd for $C_{11}H_{11}N_3O_2$ ($[M + H]^+$) 217.0851; Found 217.0850.

Synthesis of 1,2,3-triazoles (with **2a1** as an example): To a solution of **1a** (5 mmol) and aniline (5 mmol) in DMF (10 mL) was added 0.2 equiv of $FeCl_2$ (1 mmol). The mixture was warmed to 80 °C and stirred for 10 h. When **1a** disappeared (monitored by TLC), the reaction mixture was then treated with 50 mL brine, and extracted with dichloromethane (2 × 50 mL). The combined organic layer was washed with brine (3 × 50 mL), dried over $MgSO_4$ and filtered. The filtrate was concentrated in *vacuum*, and then purified by silica gel column chromatography to give **2a1** as a white solid.

5-methyl-*N*,1-diphenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2a1**) White solid; mp 149-151 °C.**^{1}H-NMR** ($CDCl_3$, 400 MHz) δ 9.07 (s, 1H), 7.72 (d, *J* = 8.0Hz, 2H), 7.55-7.64 (m, 3H), 7.46-7.51 (m, 2H), 7.39 (t, *J* = 8.0Hz, 2H), 7.16 (t, *J* = 7.6Hz, 1H), 2.69 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 125Hz) δ 159.2, 138.5, 137.6, 137.3, 135.4, 130.0, 129.6, 129.0, 125.2, 124.3, 119.7, 9.8. **HRMS** Calcd for $C_{16}H_{15}N_4O$ ($[M + H]^+$) 279.1246; Found 279.1240.

5-methyl-1-phenyl-*N*-(*o*-tolyl)-1*H*-1,2,3-triazole-4-carboxamide

(**2a2**) White solid; mp 104-106 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.03 (s, 1H), 8.12 (d, *J* = 6.0Hz, 1H), 7.55-7.65 (m, 3H), 7.46-7.52 (m, 2H), 7.21-7.32 (m, 2H), 7.08-7.13 (m, 1H), 2.69 (s, 3H), 2.43 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.0, 138.6, 137.1, 135.5, 130.4, 129.9, 129.5, 128.3, 126.6, 125.1, 124.6, 121.8, 17.6, 9.6. **HRMS** Calcd for $C_{17}H_{17}N_4O$ ($[M + H]^+$) 293.1402; Found 293.1400.

5-methyl-1-phenyl-*N*-(*p*-tolyl)-1*H*-1,2,3-triazole-4-carboxamide

(**2a3**) White solid; mp 152-154 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.02 (s, 1H), 7.55-7.65 (m, 5H), 7.45-7.52 (m, 2H), 7.19 (d, *J* = 8.1Hz, 2H), 2.68 (s, 3H), 2.35 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.0, 138.6, 137.2, 135.4, 135.1, 133.8, 129.9, 129.6, 129.5, 125.2, 119.7, 20.8, 9.7. **HRMS** Calcd for $C_{17}H_{17}N_4O$ ($[M + H]^+$) 293.1402; Found 293.1399.

N-(2-methoxyphenyl)-5-methyl-1-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2a4**) Yellow solid; mp 184-185 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.70 (s, 1H), 8.49-8.54 (m, 1H), 7.55-7.63 (m, 3H), 7.46-7.52 (m, 2H), 6.92-7.13 (m, 3H), 3.96 (s, 3H), 2.69 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.1, 148.4, 138.9, 137.1, 135.5, 129.9, 129.6, 127.4, 125.2, 123.8, 120.8, 119.6, 110.0, 55.7, 9.7. **HRMS** Calcd for

$C_{17}H_{17}N_4O_2$ ($[M + H]^+$) 309.1352; Found 309.1343.

N-(4-methoxyphenyl)-5-methyl-1-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2a5**) White solid; mp 163-166 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 8.98 (s, 1H), 7.57-7.67 (m, 5H), 7.45-7.52 (m, 2H), 6.89-6.96 (m, 2H), 3.82 (s, 3H), 2.68 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.00, 156.4, 138.6, 137.1, 135.5, 130.8, 130.0, 129.6, 125.2, 121.5, 114.2, 55.4, 9.7. **HRMS** Calcd for $C_{17}H_{17}N_4O_2$ ($[M + H]^+$) 309.1352; Found 309.1350.

N-(2-chlorophenyl)-5-methyl-1-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2a6**) Yellow solid; mp 127-129 °C. **^{1}H-NMR** ($CDCl_3$, 400 MHz) δ 9.68 (s, 1H), 8.52-8.56 (m, 1H), 7.56-7.63(m, 3H), 7.41-7.51 (m, 3H), 7.30-7.34 (m, 1H), 7.06-7.11 (m, 1H), 2.69 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 125Hz) δ 159.2, 138.4, 137.5, 135.4, 134.5, 130.0, 129.6, 129.2, 127.5, 125.2, 124.6, 123.3, 121.2, 9.8. **HRMS** Calcd for $C_{16}H_{14}ClN_4O$ ($[M + H]^+$) 313.0856; Found 313.0850.

N-(4-chlorophenyl)-5-methyl-1-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2a7**) White solid; mp 175-177 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.07 (s, 1H), 7.66-7.69 (m, 2H), 7.56-7.63 (m, 3H), 7.45-7.51 (m, 2H), 7.32-7.36 (m, 2H), 2.68 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.2, 138.4, 137.5, 136.3, 135.4, 130.2, 129.7, 129.3, 129.1, 125.2, 121.0, 9.8. **HRMS** Calcd for $C_{16}H_{14}ClN_4O$ ($[M + H]^+$) 313.0856; Found 313.0844.

N-(2,4-dimethylphenyl)-5-methyl-1-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2a8**) White solid; mp 162-164 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 8.94 (s, 1H), 7.94 (d, *J* = 9.0Hz, 1H), 7.56-7.64 (m, 3H), 7.45-7.51 (m, 2H), 7.04-7.08 (m, 2H), 2.68 (s, 3H), 2.38 (s, 3H), 2.32 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.2, 138.8, 137.1, 135.6, 134.5, 132.9, 131.2, 130.0, 129.7, 128.8, 127.2, 125.2, 122.3, 20.8, 17.7, 9.8. **HRMS** Calcd for $C_{18}H_{19}N_4O$ ([M + H]$^+$) 307.1559; Found 307.1542.

5-methyl-1-phenyl-*N*-(4-(trifluoromethyl)phenyl)-1*H*-1,2,3-triazole-4-carboxamide

(**2a9**) White solid; mp 212-214 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.23 (s, 1H), 7.86 (d, *J* = 8.4 Hz, 2H), 7.58-7.68 (m, 5H), 7.48-7.52 (m, 2H), 2.70 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.4, 140.8, 138.2, 137.8, 135.3, 130.2, 129.7, 126.31, 126.3, 125.4, 125.2, 122.7, 119.3, 9.8. **HRMS** Calcd for $C_{17}H_{14}F_3N_4O$ ([M + H]$^+$) 347.1120; Found 347.1117.

5-methyl-1-phenyl-N-(pyridin-2-yl)-1*H*-1,2,3-triazole-4-carboxamide

(**2a10**) White solid; mp 105-108 °C. **^{1}H-NMR** ($CDCl_3$, 400 MHz) δ 9.69 (s, 1H), 8.31-8.39 (m, 2H), 7.72-7.77 (m, 1H), 7.56-7.62 (m, 3H), 7.46-7.51 (m, 2H), 7.05-7.10 (m, 1H), 2.69 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.5, 151.1, 148.1, 138.1, 137.6, 135.4, 130.0, 129.6, 125.1, 119.7, 113.8, 109.5, 9.8. **HRMS** Calcd for $C_{15}H_{14}N_5O$ ([M + H]$^+$) 280.1198; Found 280.1199.

N,1-diphenyl-5-propyl-1*H*-1,2,3-triazole-4-carboxamide

(**2a11**) Yellow solid; mp 110-112 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.12 (s, 1H), 7.72 (d, *J* = 6.0 Hz, 2H), 7.59-7.61 (m, 3H), 7.44-7.47 (m, 2H), 7.39 (t, *J* = 6.0 Hz, 2H), 7.15 (t, *J* = 7.5 Hz, 1H), 3.00-3.10 (m, 2H), 1.56-1.66 (m, 2H), 0.88 (t, *J* = 7.5 Hz, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.0, 141.8, 138.2, 137.6, 135.7, 130.2, 129.6, 129.0, 125.7, 124.2, 119.8, 25.0, 22.2, 13.8. **HRMS** Calcd for $C_{18}H_{19}N_4O$ ($[M + H]^+$) 307.1559; Found 307.1548.

N,1,5-triphenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2a12**) Yellow solid; mp 101-104 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.22 (s, 1H), 7.71 (d, *J* = 6.0 Hz, 2H), 7.27-7.46 (m, 12H), 7.14 (t, *J* = 7.5Hz, 1H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 158.0, 139.5, 138.7, 137.6, 135.9, 131.7, 130.5, 129.9, 129.5, 129.3, 129.0, 128.3, 125.2, 124.3, 119.8. **HRMS** Calcd for $C_{21}H_{17}N_4O$ ($[M + H]^+$) 341.1402; Found 341.1400.

5-methyl-*N*-phenyl-1-(*o*-tolyl)-1*H*-1,2,3-triazole-4-carboxamide

(**2b1**) White solid; mp 143-145 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.10 (s, 1H), 7.70-7.75 (m, 2H), 7.36-7.55 (m, 5H), 7.23-7.27 (m, 1H), 7.12-7.19 (m, 1H), 2.50 (s, 3H), 2.07 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.2, 138.2, 138.0 137.6, 135.4, 134.2, 131.4, 130.7, 129.0, 127.1, 127.0, 124.2, 119.7, 17.1, 9.1. **HRMS** Calcd for $C_{17}H_{17}N_4O$ ($[M + H]^+$) 293.1402; Found 293.1401.

1-(2-methoxyphenyl)-5-methyl-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b2**) Yellow solid; mp 110-112 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.10 (s, 1H), 7.70-7.75 (m, 2H), 7.52-7.60 (m, 1H), 7.35-7.43 (m, 3H), 7.09-7.20 (m, 3H), 3.83 (s, 3H), 2.52 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.3, 153.8, 139.3, 137.7, 132.0, 128.9, 128.3, 124.0, 123.9, 120.9, 119.6, 112.1, 55.7, 9.0. **HRMS** Calcd for $C_{17}H_{17}N_4O_2$ ([M + H]$^+$) 309.1352; Found 309.1352.

5-methyl-*N*-phenyl-1-(*p*-tolyl)-1*H*-1,2,3-triazole-4-carboxamide

(**2b3**) Yellow solid; mp 210-213 °C. ^{1}**H-NMR** ($CDCl_3$, 300 MHz) δ 9.08 (s, 1H), 7.68-7.76 (m, 2H), 7.33-7.42 (m, 6H), 7.13-7.19 (m, 1H), 2.66 (s, 3H), 2.48 (s, 3H). ^{13}**C-NMR** ($CDCl_3$, 100Hz) δ 159.2, 140.4, 138.4, 137.7, 137.3, 133.0, 130.2, 129.0, 125.0, 124.2, 119.7, 21.2, 9.7. **HRMS** Calcd for $C_{17}H_{17}N_4O$ ([M + H]$^+$) 293.1402; Found 293.1379.

1-(4-methoxyphenyl)-5-methyl-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b4**) White solid; mp 214-216 °C. ^{1}**H-NMR** ($CDCl_3$, 300 MHz) δ 9.08 (s, 1H), 7.69-7.75 (m, 2H), 7.35-7.42 (m, 4H), 7.12-7.19 (m, 1H), 7.05-7.11 (m, 2H), 3.90 (s, 3H), 2.65 (s, 3H). ^{13}**C-NMR** ($CDCl_3$, 100Hz) δ 160.7, 159.3, 138.4, 137.7, 137.5, 129.0, 128.3, 126.6, 124.2, 119.8, 114.8, 55.6, 9.7. **HRMS** Calcd for $C_{17}H_{17}N_4O_2$ ([M + H]$^+$) 309.1352; Found 309.1349.

1-(4-chlorophenyl)-5-methyl-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b5**) White solid; mp 203-205 °C. ^{1}**H-NMR** ($CDCl_3$, 300 MHz) δ 9.05 (s, 1H), 7.70-7.74 (m, 2H), 7.55-7.62 (m, 2H), 7.35-7.46 (m, 4H), 7.12-7.20 (m, 1H), 2.69 (s, 3H). ^{13}**C-NMR** ($CDCl_3$, 100Hz) δ 159.0, 138.7, 137.5, 137.3, 136.2, 133.9, 129.9, 129.0, 126.4, 124.4, 119.8, 9.8. **HRMS** Calcd for $C_{16}H_{14}ClN_4O$ ([M + H]$^+$) 313.0856; Found 313.0852.

5-methyl-*N*-phenyl-1-(pyridin-2-yl)-1*H*-1,2,3-triazole-4-carboxamide

(**2b6**) Yellow solid; mp 119-122 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.10 (s, 1H), 8.60-8.64 (m, 1H), 7.97-8.01 (m, 2H), 7.70-7.75 (m, 2H), 7.33-7.48 (m, 3H), 7.13-7.18 (m, 1H), 3.02 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.1, 150.0, 148.4, 139.0, 138.2, 137.5, 128.9, 128.8, 124.2, 124.0, 119.7, 118.0, 10.8. **HRMS** Calcd for $C_{15}H_{14}N_5O$ ([M + H]$^{+}$) 280.1198; Found 280.1185.

5-methyl-1-(naphthalen-1-yl)-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b7**) Yellow solid; mp 189-191 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.16 (s, 1H), 8.12 (d, *J* = 8.4 Hz, 1H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.75 (d, *J* = 7.5 Hz, 2H), 7.51-7.70 (m, 4H), 7.41 (t, *J* = 9.0 Hz, 2H), 7.13-7.22 (m, 2H), 2.49 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.2, 139.4, 138.1, 137.7, 134.1, 131.5, 131.2, 129.3, 129.0, 128.4, 128.2, 127.3, 125.2, 125.0, 124.3, 121.8, 119.8, 9.2. **HRMS** Calcd for $C_{20}H_{17}N_4O$ ([M + H]$^{+}$) 329.1402; Found 329.1390.

1-benzyl-5-methyl-*N*-phenyl-1H-1,2,3-triazole-4-carboxamide

(**2b8**) Yellow solid; mp 127-128 °C. **^{1}H-NMR** ($CDCl_3$, 500 MHz) δ 9.04 (s, 1H), 7.66-7.71 (m, 2H), 7.31-7.39 (m, 5H), 7.15-7.19 (m, 2H), 7.13 (t, *J* = 7.5Hz, 1H), 5.52 (s, 2H), 2.54 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 125Hz) δ 159.2, 138.7, 137.6, 136.8, 133.8, 129.0, 129.0, 128.5, 127.1, 124.2, 119.7, 51.8, 8.8. **HRMS** Calcd for $C_{17}H_{17}N_4O$ ([M + H]$^{+}$) 293.1402; Found 293.1405.

5-methyl-*N*-phenyl-1-propyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b9**) White solid; mp 116-118 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.02 (s, 1H), 7.65-7.72 (m, 2H), 7.33-7.41 (m, 2H), 7.11-7.17 (m, 1H), 4.27 (t, *J* = 7.2Hz, 2H), 2.67 (s, 3H), 1.90-1.98 (m, 2H), 1.00 (t, *J* = 7.5Hz, 3H). **^{13}C-NMR** ($CDCl_3$, 125Hz) δ 159.3, 138.2, 137.6, 136.2, 129.0, 124.1, 119.6, 49.4, 23.1, 11.0, 8.7. **HRMS** Calcd for $C_{13}H_{17}N_4O$ ($[M + H]^+$) 245.1402; Found 245.1397.

5-methyl-1-octadecyl-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b10**) White solid; mp 71-73 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.02 (s, 1H), 7.68 (d, *J* = 7.8Hz, 2H), 7.37 (t, *J* = 7.5Hz, 2H), 7.13 (t, *J* = 7.5Hz, 1H), 4.29 (t, *J* = 7.5Hz, 2H), 2.66 (s, 3H), 1.25-1.33 (m, 32H), 0.87 (t, *J* = 6.0Hz, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.4, 138.4, 137.7, 136.2, 129.0, 124.2, 119.7, 48.0, 31.9, 29.6, 29.5, 29.3, 29.0, 26.5, 22.6, 14.1, 8.7. **HRMS** Calcd for $C_{28}H_{46}N_4O$ ($[M + H]^+$) 454.3672; Found 454.3670.

1-cyclopropyl-5-methyl-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b11**) White solid; mp 102-105 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 8.98 (s, 1H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.36 (t, *J* = 8.4 Hz, 2H), 7.13 (t, *J* = 7.2 Hz, 1H), 3.42-3.54 (m, 1H), 2.74 (s, 3H), 1.21-1.40 (m, 4H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.2, 138.5, 138.3, 137.6, 128.8, 124.0, 119.6, 29.0, 8.8, 6.3. **HRMS** Calcd for $C_{13}H_{15}N_4O$ ($[M + H]^+$) 243.1246; Found 243.1245.

1-cyclohexyl-5-methyl-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b12**) White solid; mp 146-148 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.03 (s, 1H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.36 (t, *J* = 7.5 Hz, 2H), 7.13 (t, *J* = 7.5 Hz, 1H), 4.07-4.21 (m, 1H), 2.67 (s, 3H), 1.75-2.14 (m, 7H), 1.30-1.54 (m, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.5, 138.0, 137.7, 135.5, 128.9, 124.0, 119.7, 58.1, 32.6, 25.4, 24.9, 8.6. **HRMS** Calcd for $C_{16}H_{21}N_4O$ ($[M + H]^+$) 285.1715; Found 285.1710.

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methyl 2-(5-methyl-4-(phenylcarbamoyl)-1*H*-1,2,3-triazol-1-yl)acetate

(**2b13**) White solid; mp 122-123 °C. **^{1}H-NMR** ($CDCl_3$, 400 MHz) δ 8.98 (s, 1H), 7.64-7.70 (m, 2H), 7.33-7.39 (m, 2H), 7.10-7.16 (m, 1H), 5.11 (s, 2H), 3.81 (s, 3H), 2.62 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 166.0, 159.0, 138.6, 137.8, 137.5, 129.0, 124.3, 119.8, 53.1, 48.6, 8.6. **HRMS** Calcd for $C_{13}H_{15}N_4O_3$ ($[M + H]^+$) 275.1144; Found 275.1147.

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tert-butyl (2-(5-methyl-4-(phenylcarbamoyl)-1*H*-1,2,3-triazol-1-yl)ethyl)carbamate

(**2b14**) White solid; mp 168-170 °C. **^{1}H-NMR** ($CDCl_3$, 500 MHz) δ 9.00 (s, 1H), 7.66-7.70 (m, 2H), 7.34-7.39 (m, 2H), 7.11-7.16 (m, 1H), 4.84 (s, 1H), 4.41-4.44 (m, 2H), 3.64-3.67 (m, 2H), 2.65 (s, 3H), 1.43 (s, 9H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.2, 155.8, 138.4, 137.6, 137.4, 129.0, 124.3, 119.8, 80.2, 47.2, 40.3, 28.3, 8.6. **HRMS** Calcd for $C_{17}H_{24}N_5O_3$ ($[M + H]^+$) 346.1879; Found 346.1877.

1-(2-hydroxyethyl)-5-methyl-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b15**) White solid; mp 104-106 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 8.97 (s, 1H), 7.62-7.71 (m, 2H), 7.32-7.41 (m, 2H), 7.14 (t, *J* = 7.5Hz, 1H), 4.33-4.43 (m, 2H), 4.04-4.17 (m, 2H), 2.15-2.70 (m, 4H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.2, 137.9, 137.7, 137.3, 128.9, 124.3, 119.9, 60.7, 50.1, 8.7. **HRMS** Calcd for $C_{12}H_{15}N_4O_2$ ($[M + H]^+$) 247.1195; Found 247.1187.

1-allyl-5-methyl-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b16**) White solid; mp 85-87 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.01 (s, 1H), 7.69 (d, *J* = 7.8Hz, 2H), 7.37 (t, *J* = 7.8Hz, 2H), 7.14 (t, *J* = 7.5 Hz, 1H), 5.89-6.07 (m, 1H), 5.35 (d, *J* = 10.2Hz, 1H), 5.11 (d, *J* = 17.1Hz, 1H), 4.94-5.00 (m, 2H), 2.64 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.2, 138.5, 137.6, 136.7, 130.4, 128.9, 124.1, 119.7, 119.0, 50.3, 8.6. **HRMS** Calcd for $C_{13}H_{15}N_4O$ ($[M + H]^+$) 243.1246; Found 243.1246.

5-methyl-*N*-phenyl-1-(prop-2-yn-1-yl)-1*H*-1,2,3-triazole-4-carboxamide

(**2b17**) White solid; mp 93-95 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 8.99 (s, 1H), 7.68 (d, *J* = 7.8Hz, 2H), 7.37 (t, *J* = 8.0 Hz, 2H), 7.14 (t, *J* = 7.5 Hz, 1H), 5.15 (d, *J* = 2.4Hz, 2H), 2.77 (s, 3H), 2.51 (t, *J* = 2.4Hz, 1H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.0, 138.8, 137.5, 137.0, 129.0, 124.3, 119.7, 75.3, 74.5, 37.9, 8.7. **HRMS** Calcd for $C_{13}H_{13}N_4O$ ($[M + H]^+$) 241.1089; Found 241.1078.

(*R*)-5-methyl-*N*-phenyl-1-(1-phenylethyl)-1*H*-1,2,3-triazole-4-carboxamide

(**2b18**) White solid; mp 105-108 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.05 (s, 1H), 7.67 (d, *J* = 9.0 Hz, 2H), 7.31-7.39 (m, 5H), 7.09-7.22 (m, 3H), 5.58 (q, *J* = 7.0 Hz, 1H), 2.50 (s, 3H), 2.09 (d, *J* = 6.0 Hz, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.3, 139.7, 138.7, 137.6, 136.5, 129.0, 128.9, 128.3, 125.9, 124.1, 119.7, 58.8, 21.6, 8.7. **HRMS** Calcd for $C_{13}H_{13}N_4O$ ($[M + H]^+$) 241.1089; Found 241.1078.

5-methyl-1-(methylamino)-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b19**) Yellowish solid; mp 123-125 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 8.97 (s, 1H), 7.68 (d, *J* = 9.0 Hz, 2H), 7.37 (t, *J* = 9.0 Hz, 2H), 7.14 (t, *J* = 7.5 Hz, 1H), 3.05 (s, 3H), 2.63 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.0, 137.5, 137.0, 136.0, 128.9, 124.1, 119.7, 39.9, 8.2. **HRMS** Calcd for $C_{11}H_{14}N_5O$ ($[M + H]^+$) 232.1198; Found 232.1194.

1-methoxy-5-methyl-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2b20**) Yellowish solid; mp 86-89 °C. **^{1}H-NMR** ($CDCl_3$, 400 MHz) δ 8.93 (s, 1H), 7.67 (d, *J* = 8.0Hz, 2H), 7.37 (t, *J* = 8.0Hz, 2H), 7.14 (t, *J* = 7.5 Hz, 1H), 4.31 (s, 3H), 2.64 (s, 3H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 158.5, 137.3, 136.5, 130.7, 128.9, 124.2, 119.6, 67.3, 7.5. **HRMS** Calcd for $C_{11}H_{13}N_4O_2$ ($[M + H]^+$) 233.1039; Found 233.1035.

Synthesis of N*H*-1,2,3-triazoles 2c1-2c5 (with **2c1** as an example): To a solution of **1a** (5 mmol) and ammonium acetate (7.5mmol) dissolved in 10 mL of DMF was added 0.2 equiv of $FeCl_2$. The mixture was warmed to 80 °C and stirred for 5 h. When **1a** had disappeared (TLC). The reaction mixture was treated with 50 mL brine, and then extracted with dichloromethane (2 × 50 mL). The combined organic layer was washed with brine (3 × 50 mL), dried over $MgSO_4$ and filtered. The filtrate was concentrated in *vacuum*, and then purified by silica gel column chromatography to give **2c1** as a white solid.

5-methyl-*N*-phenyl-1*H*-1,2,3-triazole-4-carboxamide

(**2c1**) White solid; mp 196-198 °C. **^{1}H-NMR** (DMSO, 500 MHz) δ 10.29 (s, 1H), 7.83 (d, *J* = 10.0 Hz, 2H), 7.32 (t, *J* = 10.0 Hz, 2H), 7.07 (t, *J* = 7.5Hz, 1H), 2.52 (s, 3H). **^{13}C-NMR** (DMSO, 125Hz) δ 159.9, 138.7, 137.6, 128.6, 123.6, 120.3, 120.1, 9.3. **HRMS** Calcd for $C_{10}H_{11}N_4O$ ($[M + H]^+$) 203.0933; Found 203.0929.

N-(4-methoxyphenyl)-5-methyl-1*H*-1,2,3-triazole-4-carboxamide

(**2c2**) White solid; mp 180-182 °C. **^{1}H-NMR** (DMSO, 300 MHz) δ 15.30 (s, 1H), 10.17 (s, 1H), 7.73 (d, *J* = 9.0Hz, 2H), 6.90 (d, *J* = 9.0Hz, 2H), 3.73 (s, 3H), 2.51 (s, 3H). **^{13}C-NMR** (DMSO, 100Hz) δ 159.6, 155.6, 137.8, 131.9, 122.0, 113.7, 55.2, 9.3. **HRMS** Calcd for $C_{11}H_{13}N_4O_2$ ([M + H]$^+$) 233.1039; Found 233.1040.

N-(2,4-dimethylphenyl)-5-methyl-1*H*-1,2,3-triazole-4-carboxamide

(**2c3**) White solid; mp 184-187 °C. **^{1}H-NMR** (DMSO, 300 MHz) δ 15.31 (s, 1H), 9.62 (s, 1H), 7.39 (d, *J* = 6.0 Hz, 1H), 7.06 (s, 1H), 7.00 (d, *J* = 6.0 Hz, 1H), 2.50 (s, 3H), 2.27 (s, 3H), 2.21 (s, 3H). **^{13}C-NMR** (DMSO, 100Hz) δ 159.6, 134.4, 133.3, 131.7, 130.8, 129.3, 126.5, 125.6, 124.9, 20.5, 17.6, 9.1. **HRMS** Calcd for $C_{12}H_{15}N_4O$ ([M + H]$^+$) 231.1246; Found 231.1246.

N-(4-chlorophenyl)-5-methyl-1*H*-1,2,3-triazole-4-carboxamide

(**2c4**) White solid; mp 204-206 °C. **^{1}H-NMR** (DMSO, 400 MHz) δ 15.32 (s, 1H), 10.41 (s, 1H), 7.86 (d, *J* = 8.8Hz, 2H), 7.33 (d, *J* = 8.8Hz, 2H), 2.50 (s, 3H). **^{13}C-NMR** (DMSO, 100Hz) δ 159.9, 137.8, 137.5, 137.4, 128.4, 127.2, 121.8, 9.2. **HRMS** Calcd for $C_{10}H_{10}ClN_4O$ ([M + H]$^+$) 237.0543; Found 237.0504.

N-phenyl-5-propyl-1*H*-1,2,3-triazole-4-carboxamide

(**2c5**) White solid; mp 106-109 °C. **^{1}H-NMR** (DMSO, 500 MHz) δ 10.30 (s, 1H), 7.84 (d, *J* = 10.0 Hz, 2H), 7.31 (t, *J* = 7.5 Hz, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 2.96 (t, *J* = 7.5 Hz, 2H), 1.62-1.73 (m, 2H), 0.89 (t, *J* = 7.5 Hz, 3H). **^{13}C-NMR** (DMSO, 125Hz) δ 159.8, 138.8, 129.3, 128.6, 125.7, 123.6, 120.3, 25.3, 21.8, 13.6. **HRMS** Calcd for $C_{12}H_{15}N_4O$ ($[M + H]^+$) 231.1246; Found 231.1292.

Synthesis of bis-triazoles 3a and 3b (with **3a** as an example): To a solution of **1h** (5 mmol) and 1,6-diaminohexane (2 mmol) dissolved in 10 mL of DMF was added 0.2 equiv of $FeCl_2$. The mixture was warmed to 80 °C and stirred for 10 h. When **1h** had disappeared (TLC). The reaction mixture was treated with 50 mL brine, and then extracted with dichloromethane (2 × 50 mL). The combined organic layer was washed with brine (3 × 50 mL), dried over $MgSO_4$ and filtered. The filtrate was concentrated in *vacuum*, and then purified by silica gel column chromatography to give **3a** as a pale pink solid.

1,1'-(hexane-1,6-diyl)bis(*N*-(2,4-dimethylphenyl)-5-methyl-1*H*-1,2,3-triazole-4-carb-oxamide)

(**3a**) Pale pink; mp 175-178 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 8.86 (s, 2H), 7.90 (d, *J* = 9.0 Hz, 2H), 7.00-7.09 (m, 4H), 4.29 (t, *J* = 6.0 Hz, 4H), 2.65 (s, 6H), 2.34 (s, 6H), 2.31 (s, 6H), 1.84-1.99 (m, 4H), 1.35-1.48 (m, 4H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.2, 138.5, 135.9, 134.2, 132.8, 131.0, 128.5, 127.0, 122.0, 47.4, 29.2, 26.8, 20.7, 17.5, 8.6. **HRMS** Calcd for $C_{30}H_{39}N_8O_2$ ($[M + H]^+$) 543.3196; Found 543.3176.

1,1'-(hexane-1,6-diyl)bis(*N*-phenyl-5-propyl-1*H*-1,2,3-triazole-4-carboxamide)

(**3b**) Yellow solid; mp 188-190 °C. **^{1}H-NMR** ($CDCl_3$, 300 MHz) δ 9.03 (s, 2H), 7.68 (d, *J* = 7.5 Hz, 4H), 7.36 (t, *J* = 7.8 Hz, 4H), 7.13 (t, *J* = 7.5 Hz, 2H), 4.28 (t, *J* = 7.2 Hz, 4H), 2.98-3.07 (m, 4H), 1.90-2.01 (m, 4H), 1.63-1.75 (m, 4H), 1.39-1.48 (m, 4H), 1.02 (t, *J* = 7.5Hz, 6H). **^{13}C-NMR** ($CDCl_3$, 100Hz) δ 159.1, 140.4, 138.1, 137.6, 129.0, 124.1, 119.7, 47.6, 29.7, 26.0, 24.7, 22.3, 13.8. **HRMS** Calcd for $C_{30}H_{39}N_8O_2$ ($[M + H]^+$) 543.3196; Found 543.3185.

III. Single-crystal X-ray diffraction data for compound **2a1**

Single-crystal X-ray diffraction data for compound **2a1** was collected at room temperature on a Oxford Diffraction Gemini R Ultra diffractometer, the X-ray generator using Mo-Kα (λ = 0.71073 Å) radiation with a ω scan technique. the crystal structures were solved by direct method of SHELXS-97 and refined by full-matrix least-squares techniques using the SHELXL-97 program.[1] Non-hydrogen atoms were refined anisotropic, and hydrogen atoms of the ligands were refined as rigid groups. Basic information of crystal parameters and structure refinement are listed in Table 1-4

1 (*a*) G. M. Sheldrick, *SHELXS-97, Program for Solution of Crystal Structures, University of Göttingen*, Germany, 1997; (*b*) G. M. Sheldrick, *SHELXL-97, Program for Refinement of Crystal Structures, University of Göttingen,* Germany, 1997.

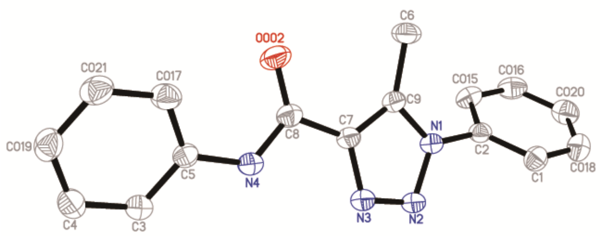
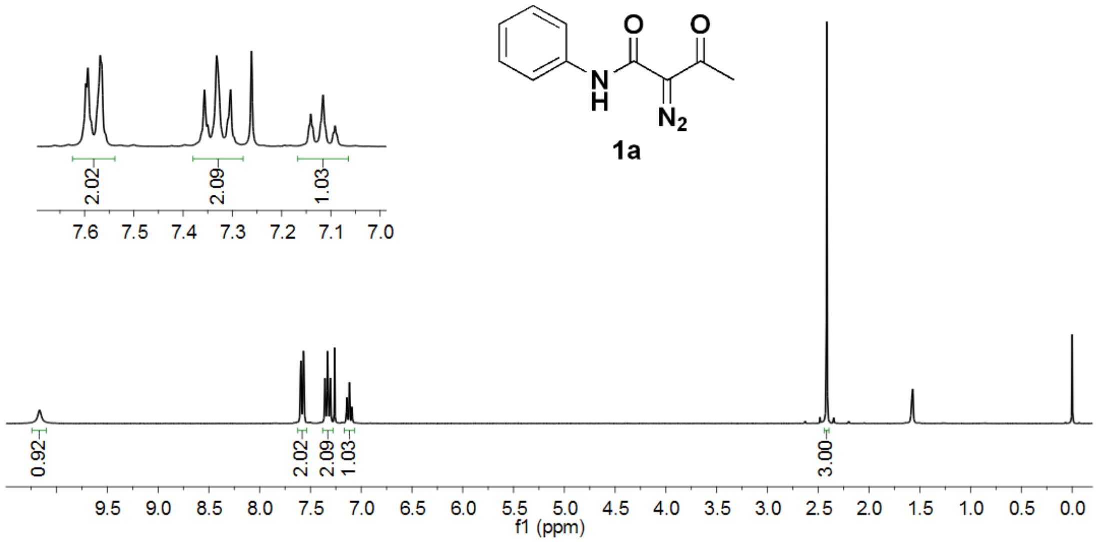
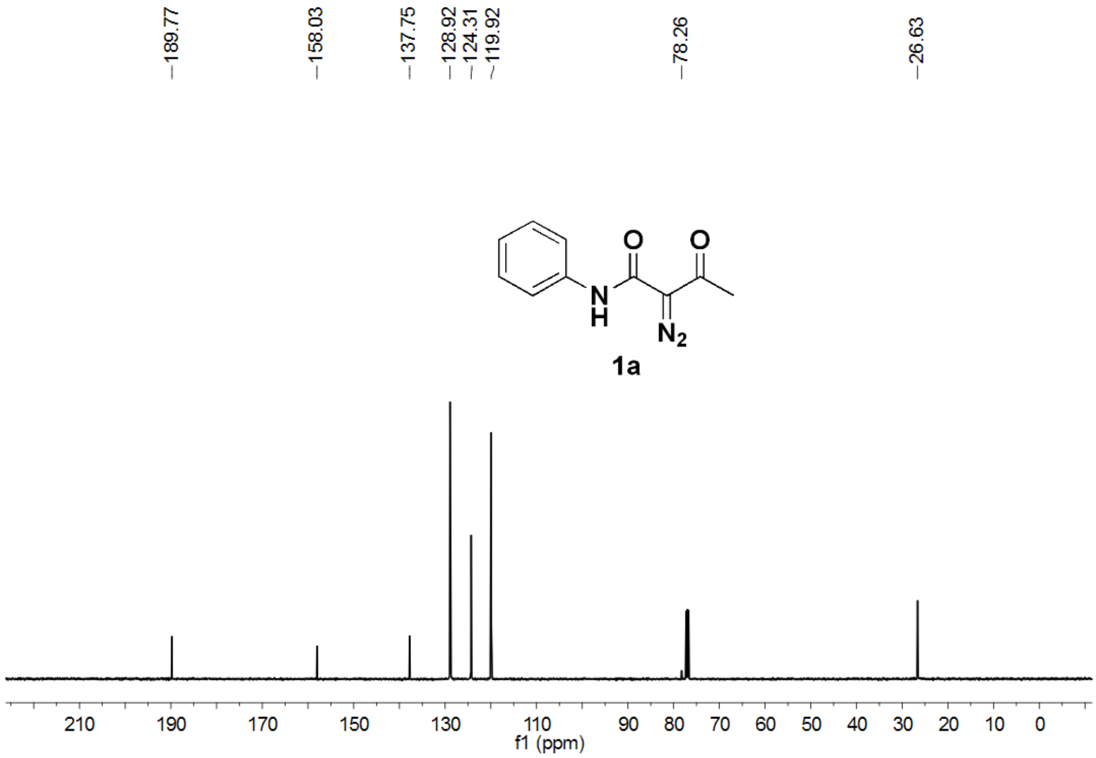


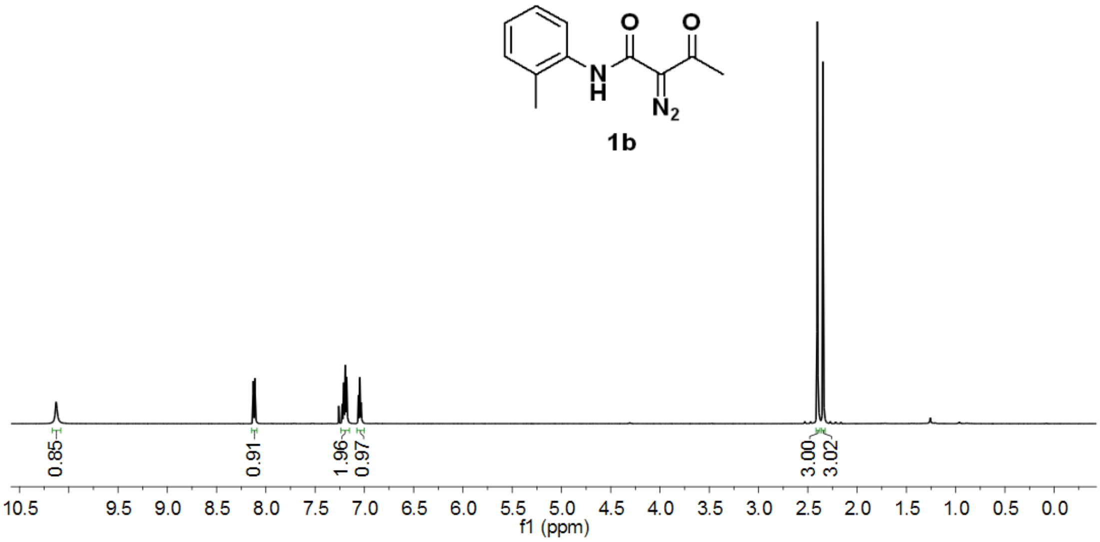
Table 1. Crystal data and structure refinement of **2a1**.

Empirical formula	$C_{33}H_{28}N_2SCl$
Formula weight	520.08
Temperature	293(2) K
Crystal system	Orthorhombic
Space group	Pna21
Unit cell dimensions	a = 13.0268(10) Å
	b = 26.8385(14) Å
	c =7.9778(6) Å
	alpha = 90.00 deg.
	beta = 90.00 deg.
	gamma = 90.00 deg.
Volume	2789.2(3) $Å^3$
Z	4
Calculated density	1.239 Mg/m^3
Absorption coefficient	0.236 mm^{-1}
F(000)	1092
Crystal size	0.34 x 0.19 x 0.14 mm
Theta range for data collection	2.76 to 25.02 deg.
Reflections collected / unique	16740 / 4413 [R(int) = 0.0529]
Data / restraints / parameters	4413 / 1 / 347
Goodness-of-fit on F^2	1.050
Final R indices [I>2sigma(I)]	R1 = 0.0643, wR2 = 0.1306
R indices (all data)	R1 = 0.1087, wR2 = 0.1492

IV. ^{1}H- and ^{13}C-NMR Spectra Copies



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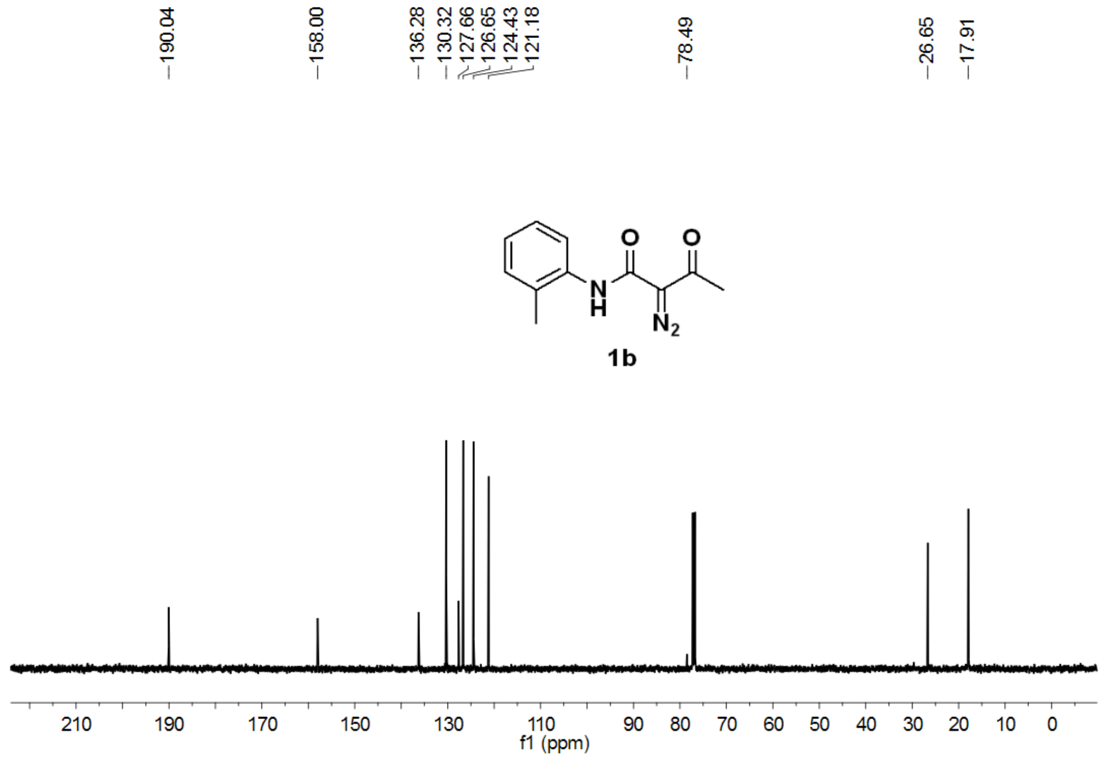
0.91
1.96
0.97
3.00
3.02
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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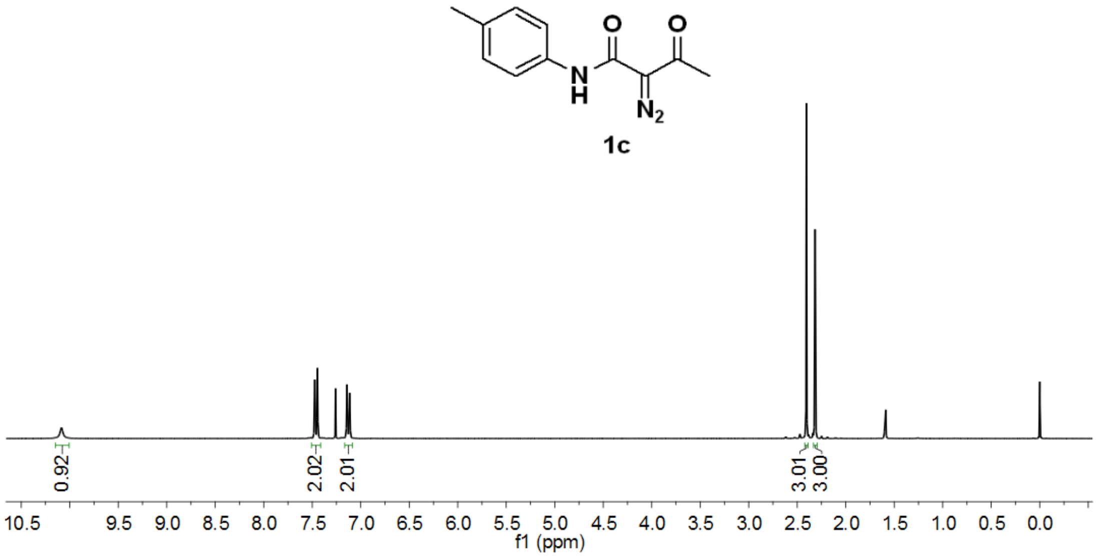
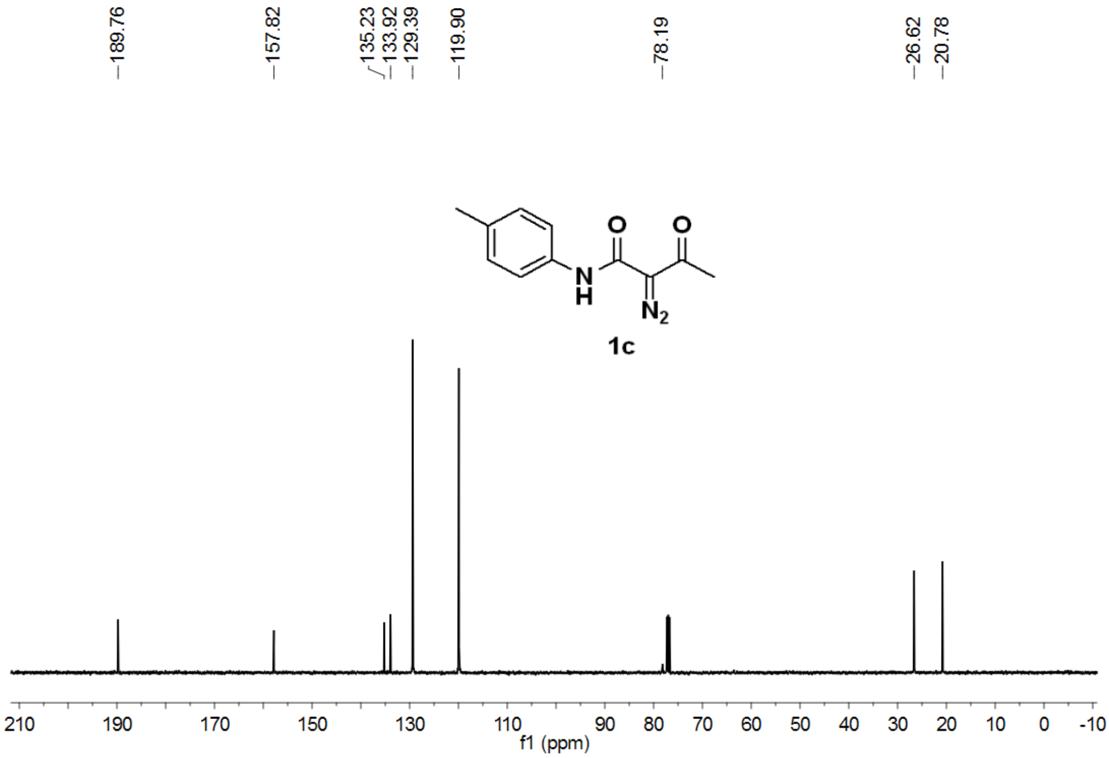
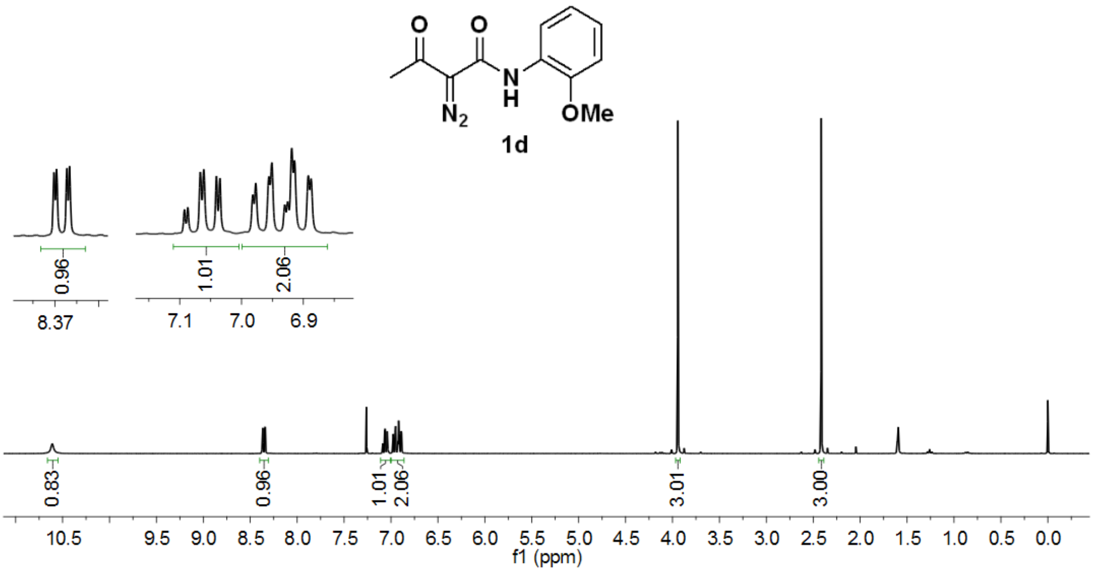
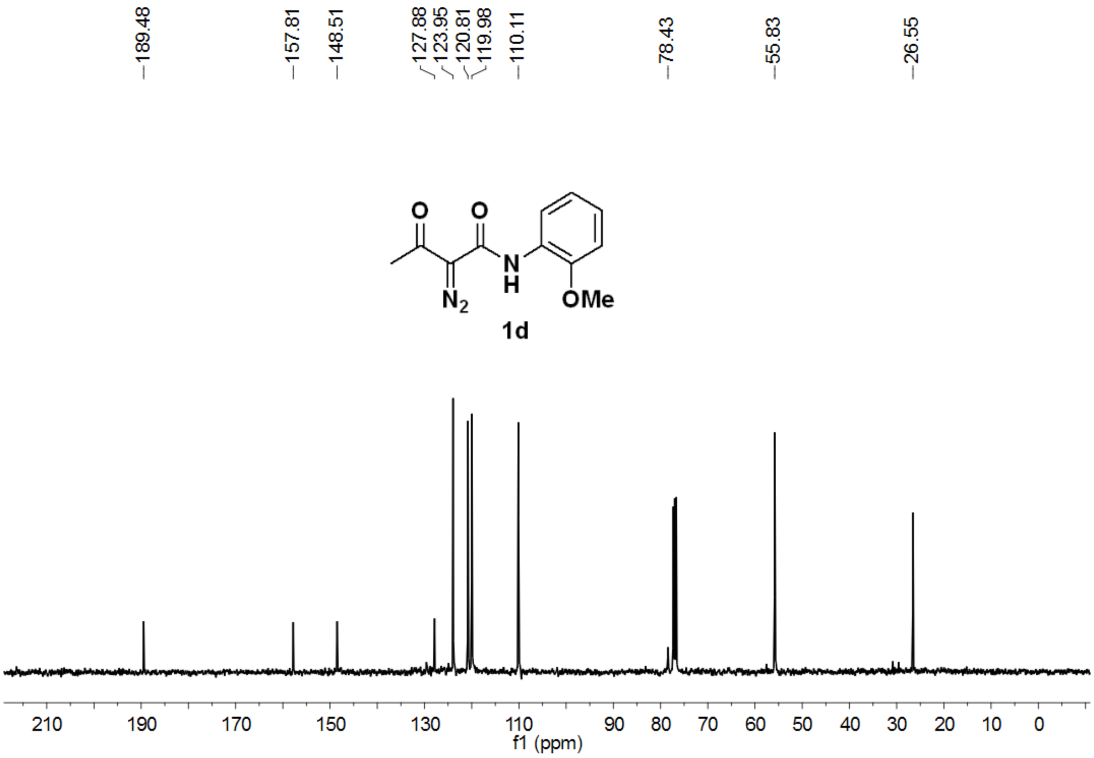
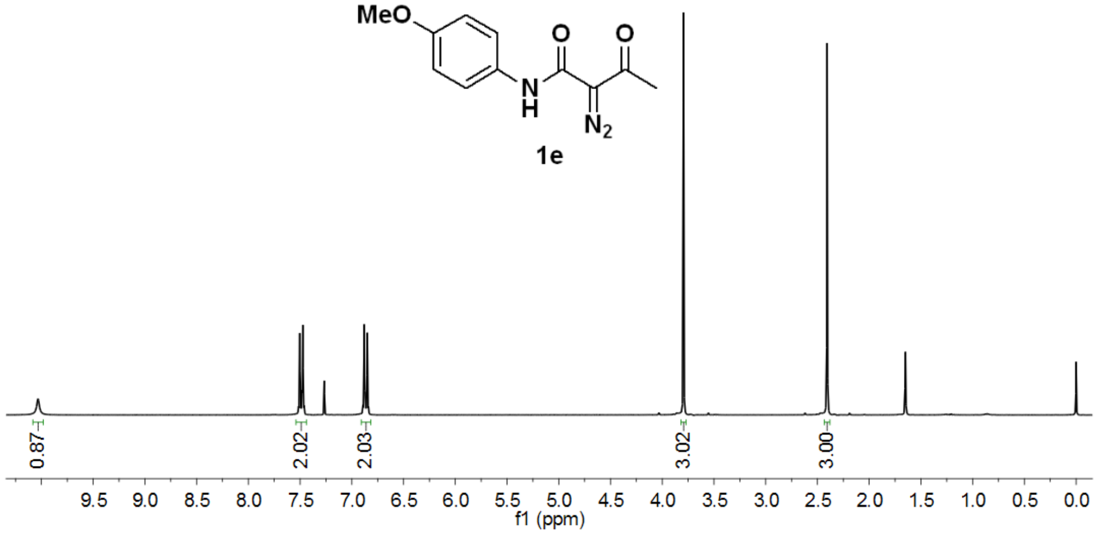
127.66
126.65
124.43
121.18
78.49
26.65
17.91
1b
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

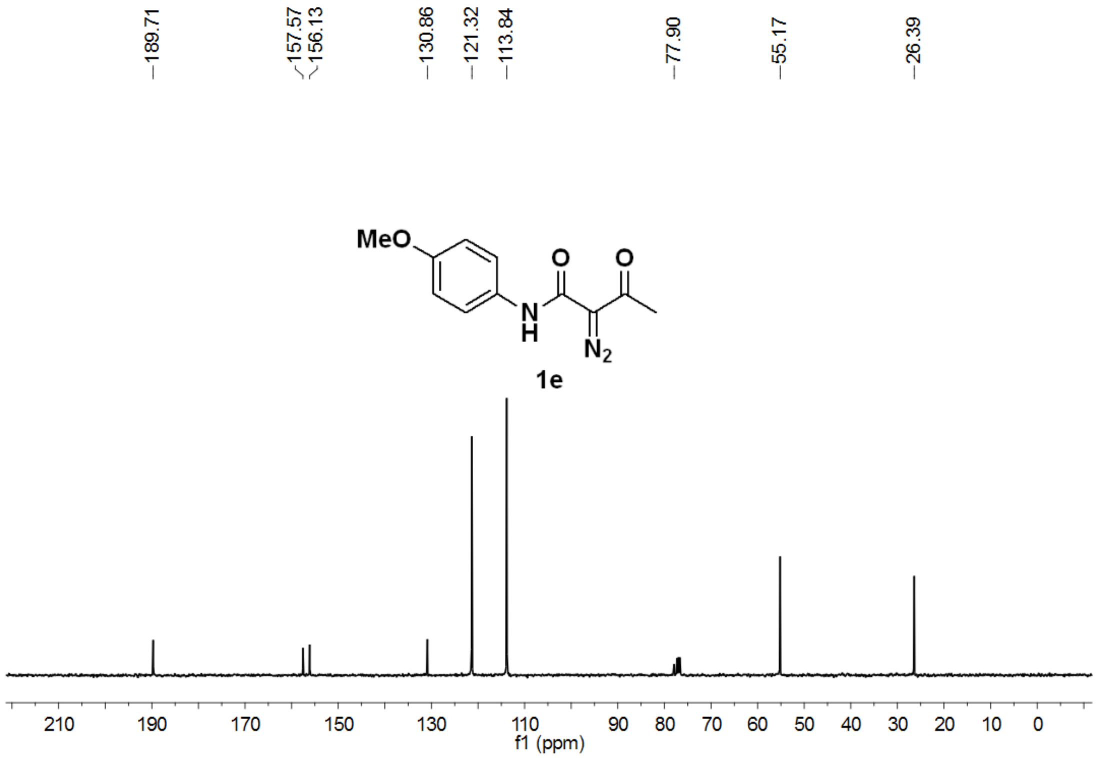

O
O
N
H
N2
1c
0.92
2.02
2.01
3.01
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

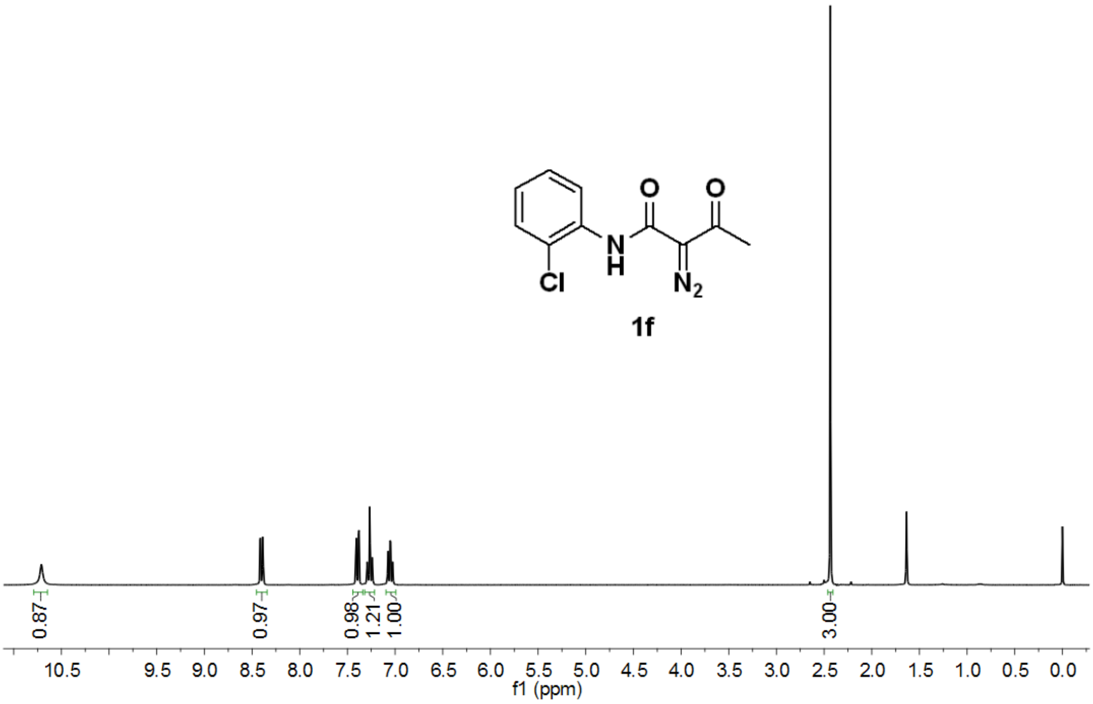

189.76
157.82
135.23
133.92
129.39
119.90
78.19
26.62
20.78
O
O
N
H
N2
1c
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

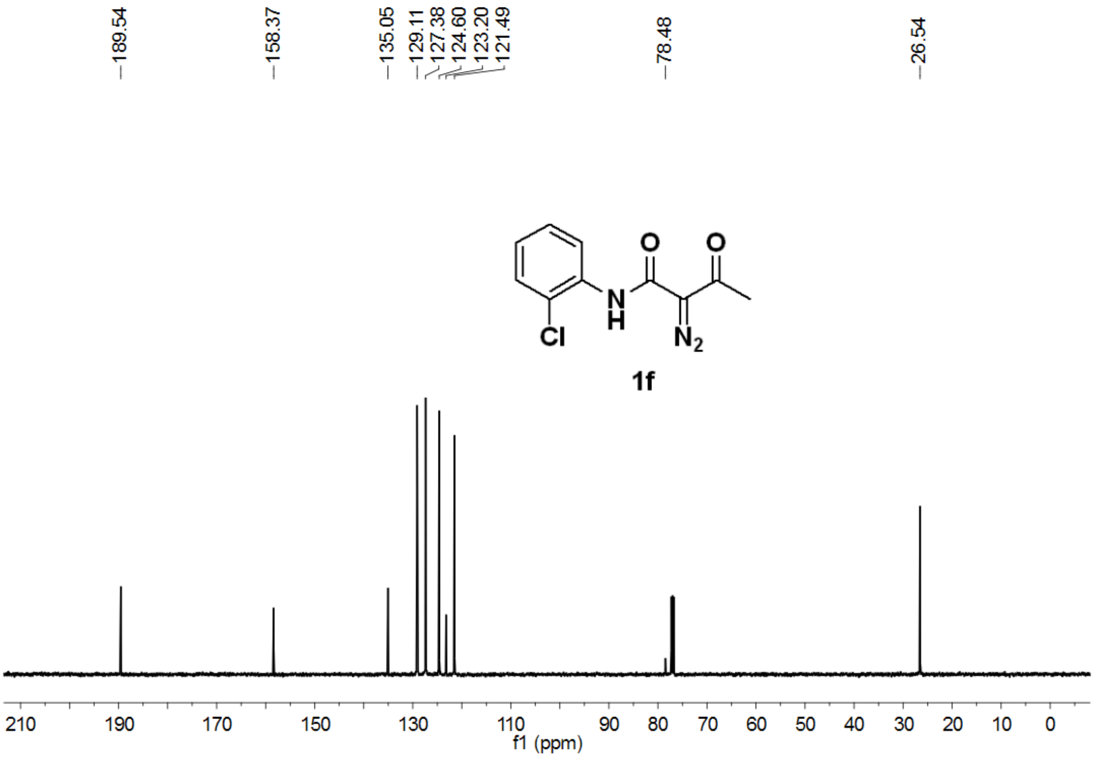

O
O
N
H
N2
OMe
1d
0.96
1.01
2.06
8.37
7.1
7.0
6.9
0.83
0.96
1.01
2.06
3.01
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


189.48
157.81
148.51
127.88
123.95
120.81
119.98
110.11
78.43
55.83
26.55
O
O
N
H
N2
OMe
1d
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


MeO
O
O
N
H
N2
1e
0.87
2.02
2.03
3.02
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


189.71
157.57
156.13
130.86
121.32
113.84
77.90
55.17
26.39
MeO
O
O
N
H
N2
1e
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


1f
0.87
0.97
0.98
1.21
1.00
3.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


189.54
158.37
135.05
129.11
127.38
124.60
123.20
121.49
78.48
26.54
1f
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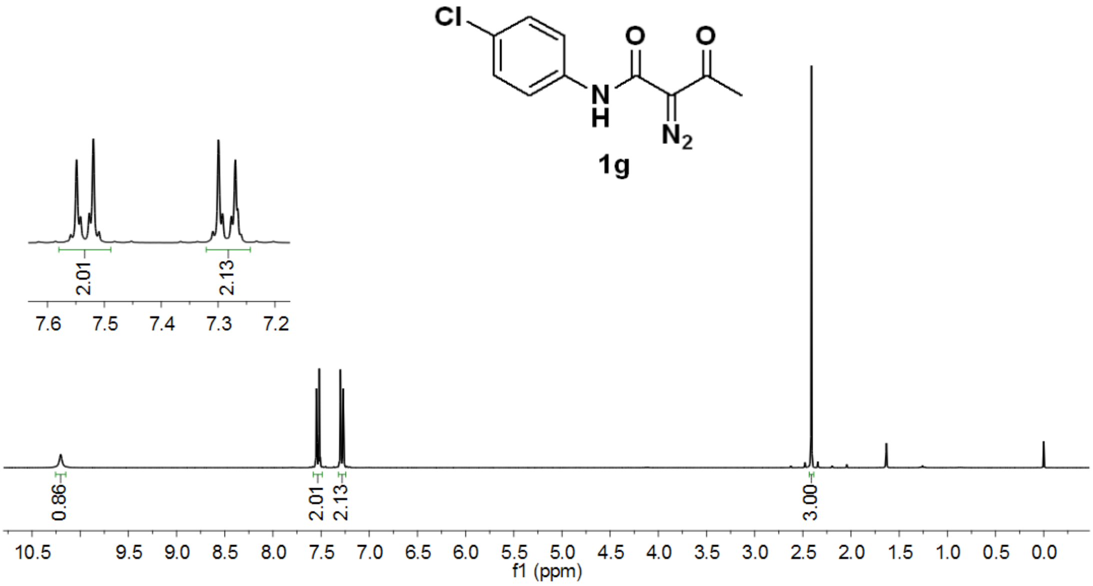
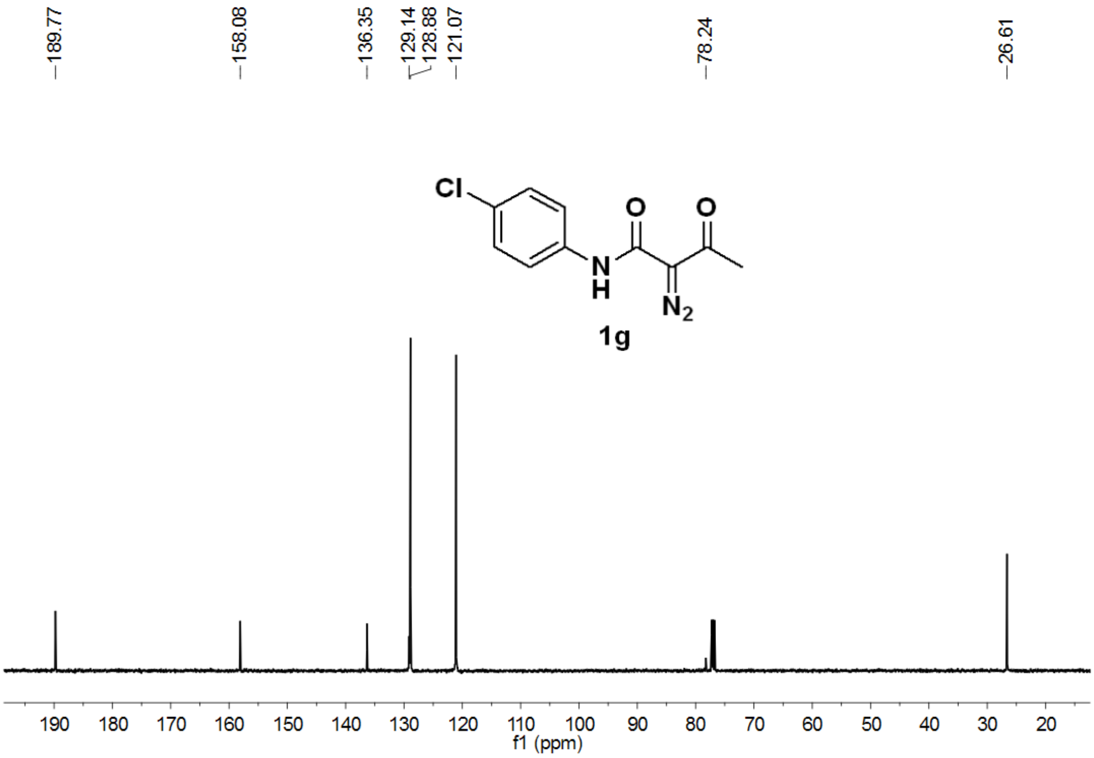
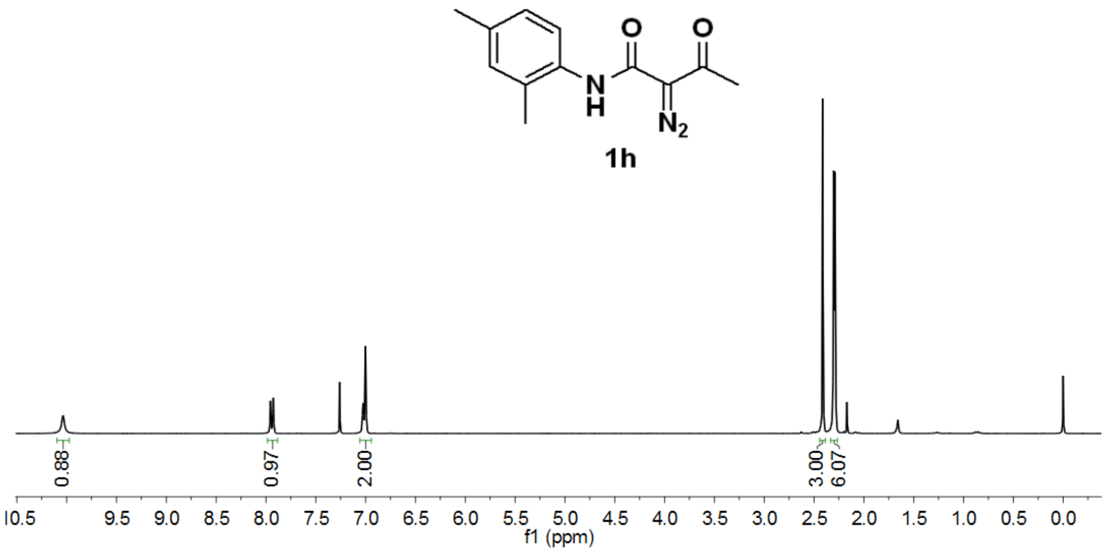
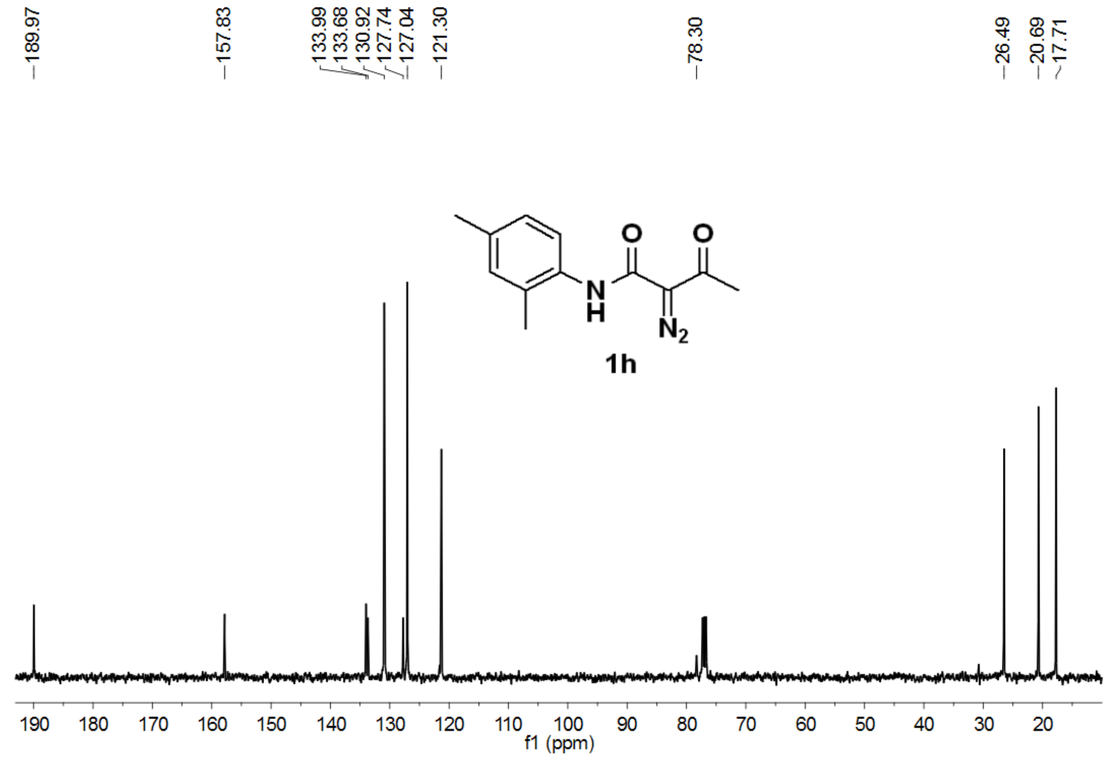
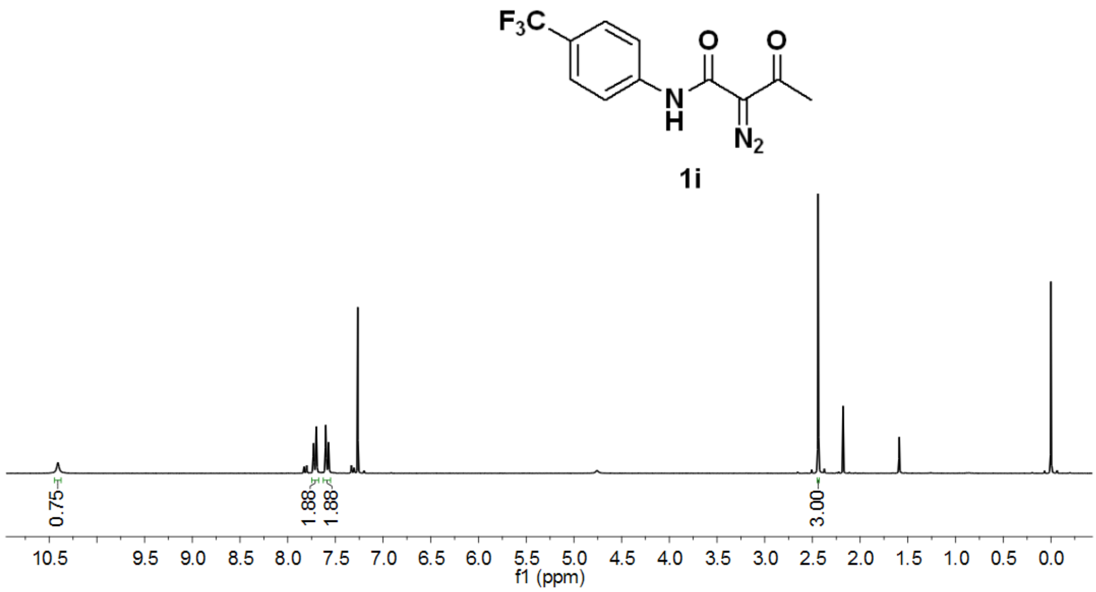
f1 (ppm)

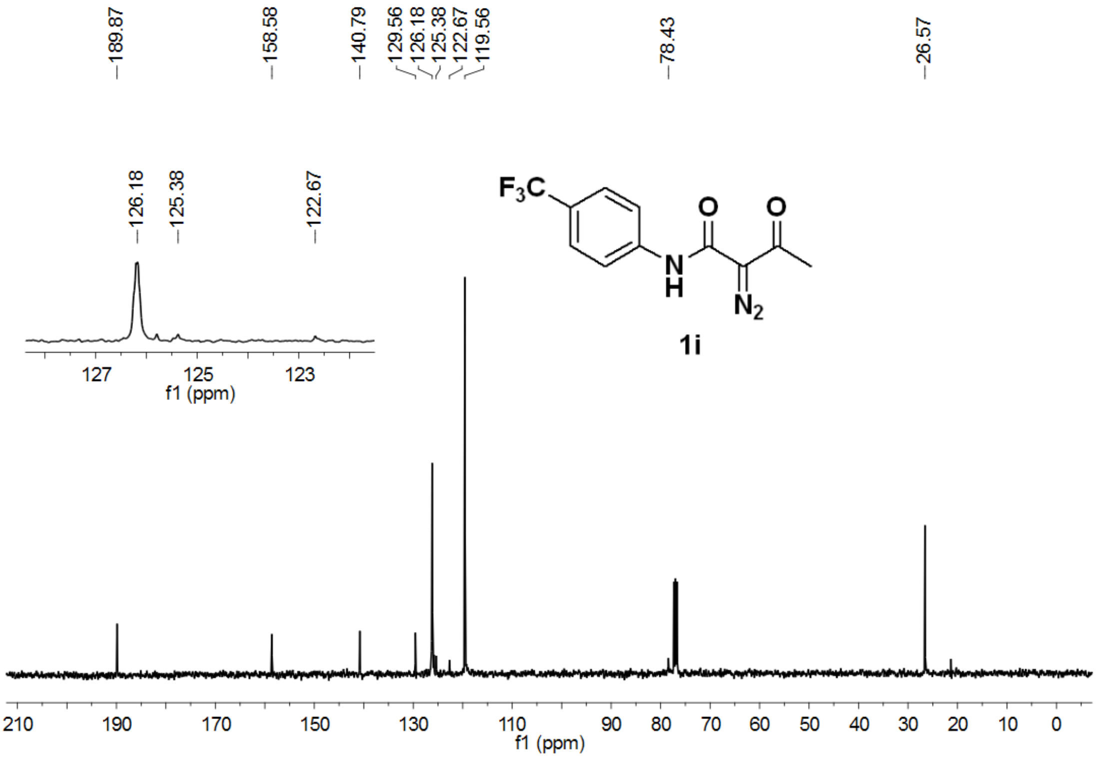

Cl
O
O
N
H
N2
1g
2.01
2.13
7.6 7.5 7.4 7.3 7.2
0.86
2.01
2.13
3.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

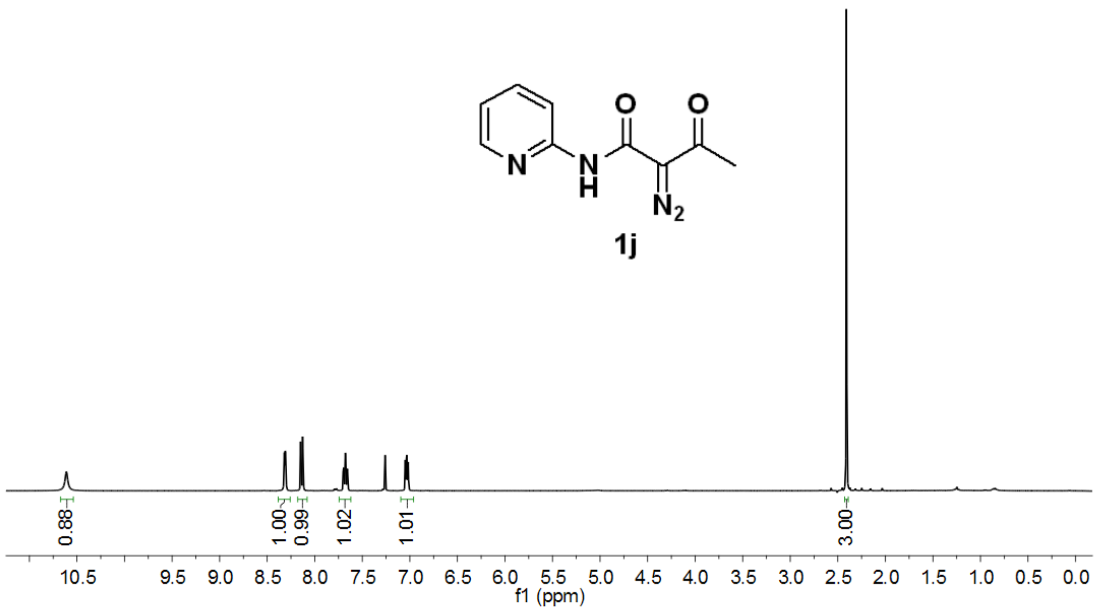

189.77
158.08
136.35
129.14
128.88
121.07
78.24
26.61
Cl
O
O
N
H
N2
1g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)

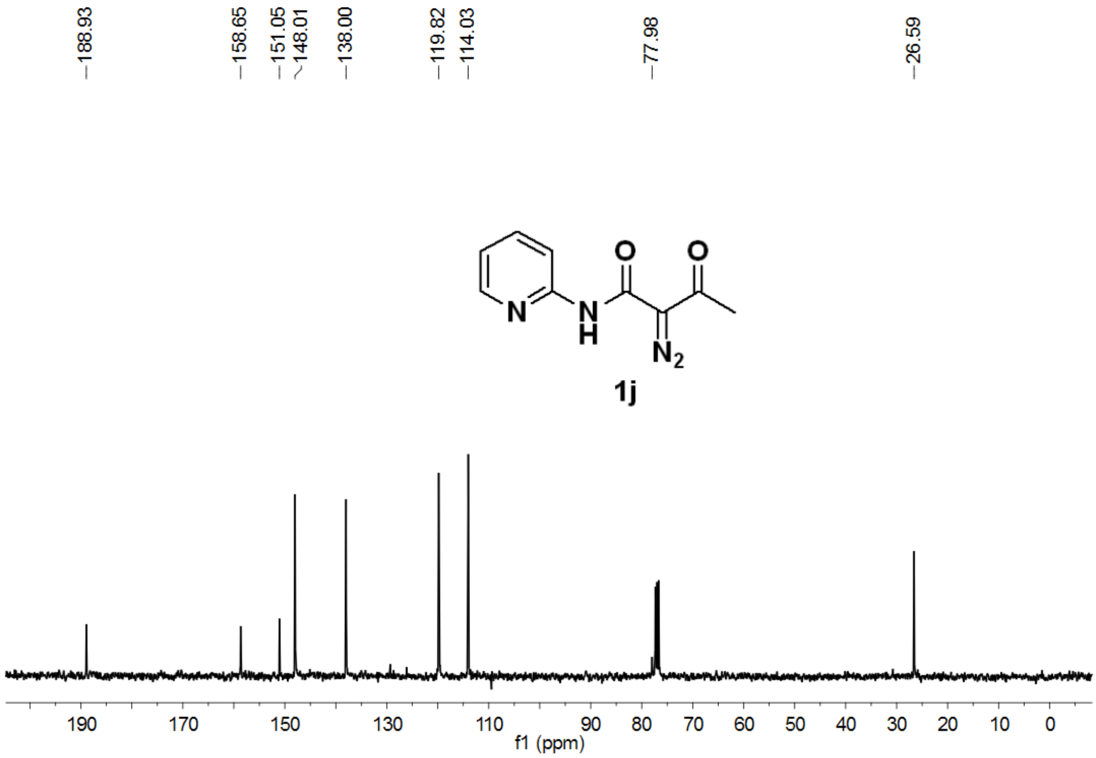

1h
0.88
0.97
2.00
3.00
6.07
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

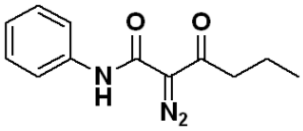

189.97
157.83
133.99
133.68
130.92
127.74
127.04
121.30
78.30
26.49
20.69
17.71
1h
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


F3C
O
O
N
H
N2
1i
0.75
1.88
1.88
3.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

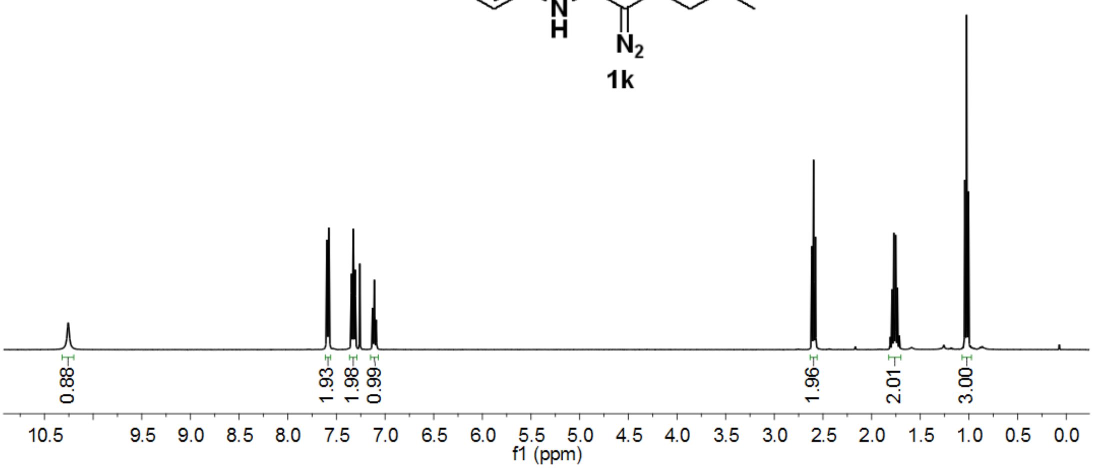

189.87
158.58
140.79
129.56
126.18
125.38
122.67
119.56
78.43
26.57
126.18
125.38
122.67
127 125 123
f1 (ppm)
F3C
O
O
N
H
N2
1i
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

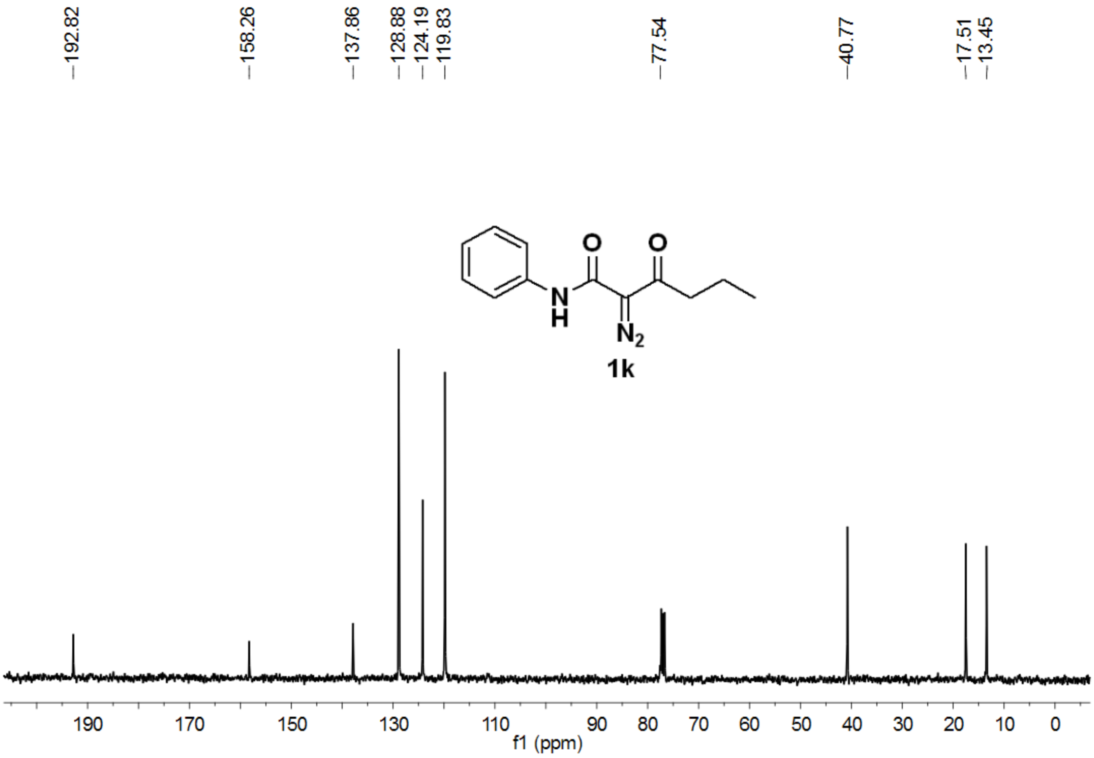

O
O
N
N
H
N2
1j
0.88
1.00
0.99
1.02
1.01
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


188.93
158.65
151.05
148.01
138.00
119.82
114.03
77.98
26.59
O
O
N
N
H
N2
1j
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

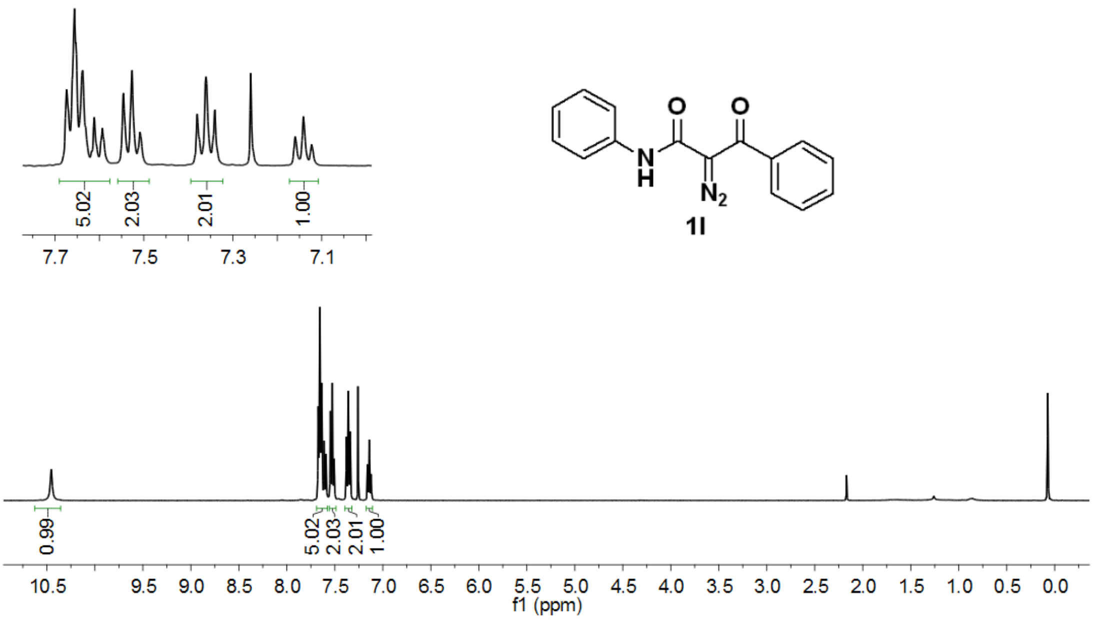

O
O
N
H
N2

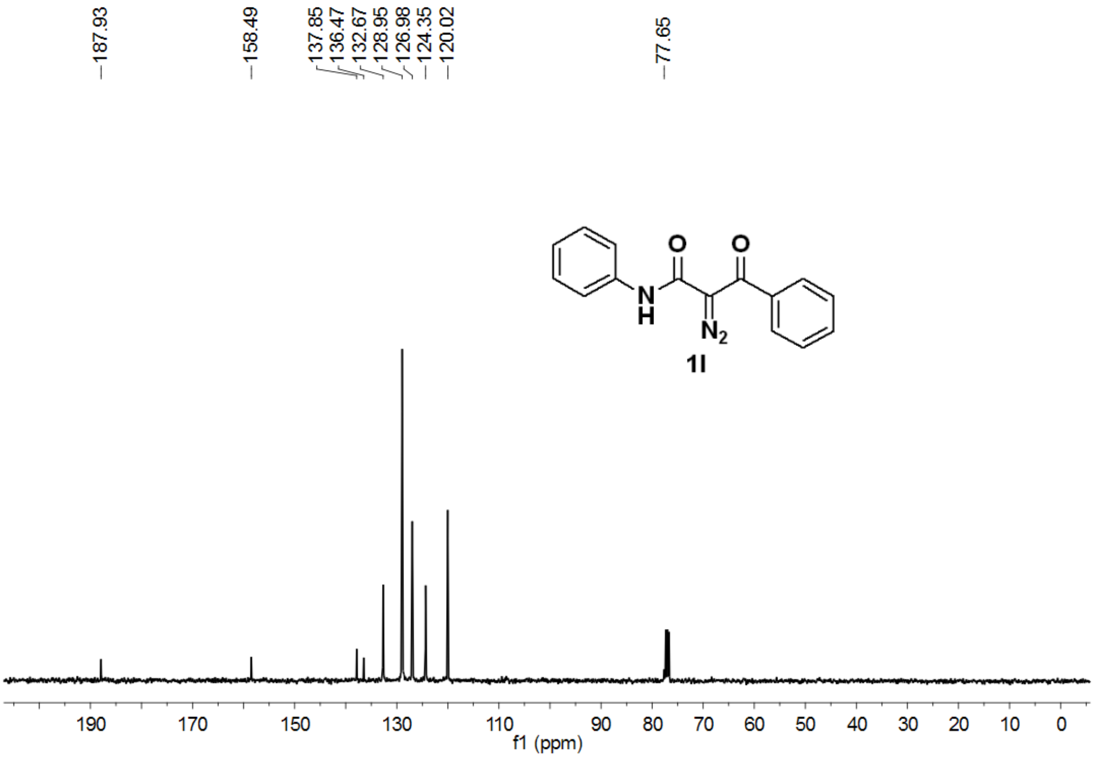
1k

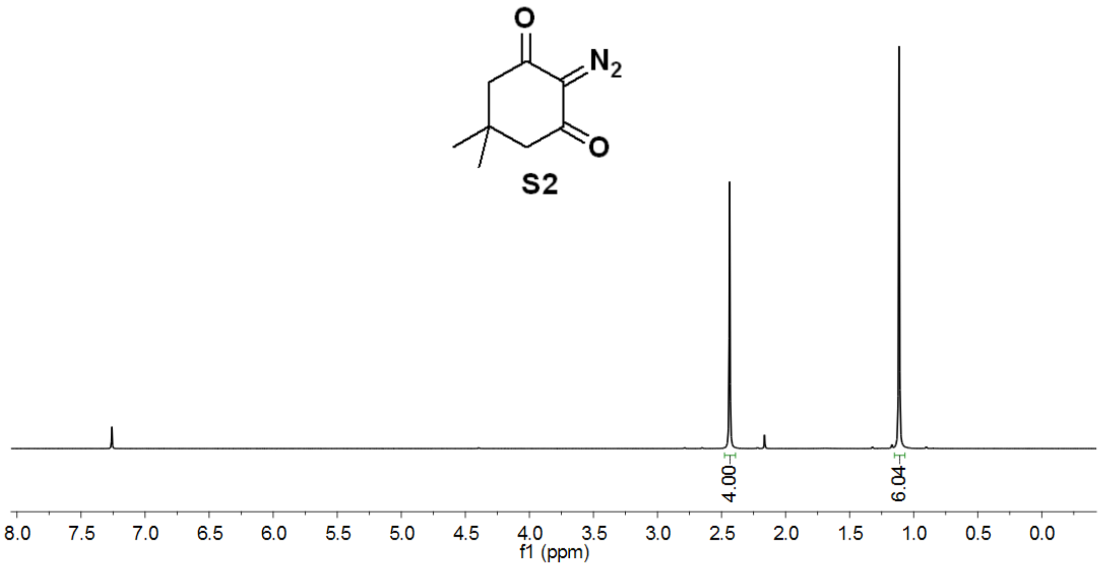

0.88
1.93
1.98
0.99
1.96
2.01
3.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

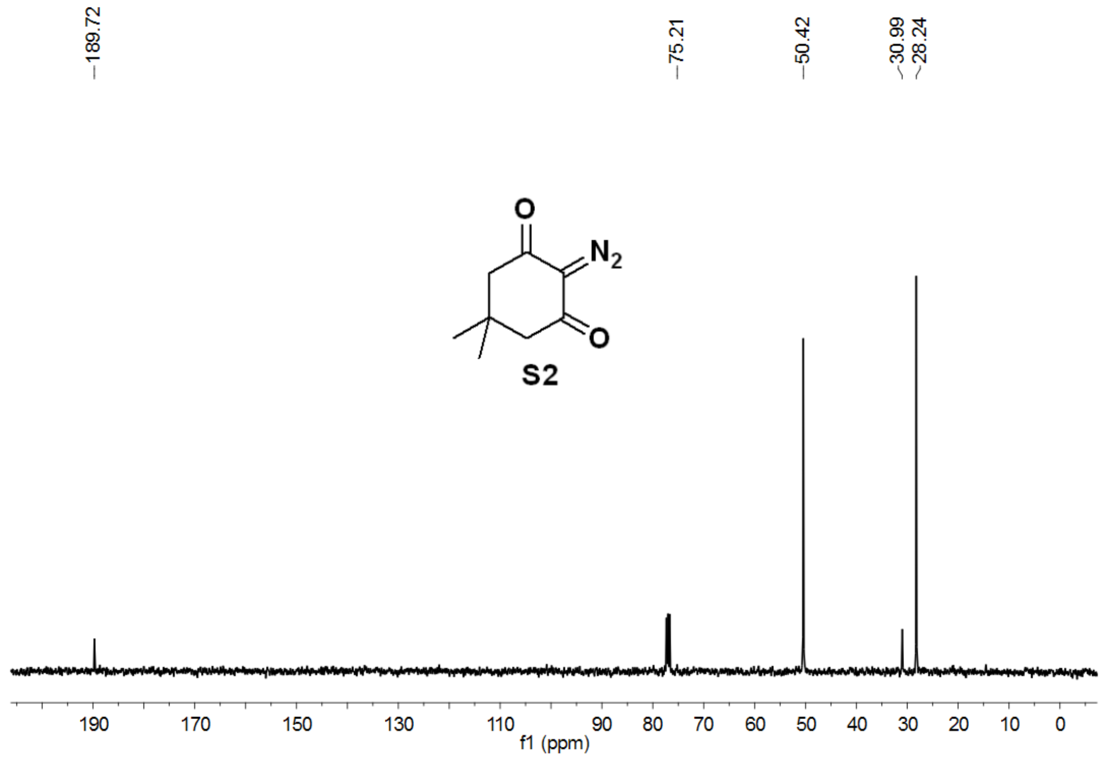

192.82
158.26
137.86
128.88
124.19
119.83
77.54
40.77
17.51
13.45
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
O
O
N
H
N2

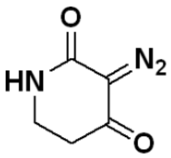
1k


5.02
2.03
2.01
1.00
7.7
7.5
7.3
7.1
1l
0.99
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

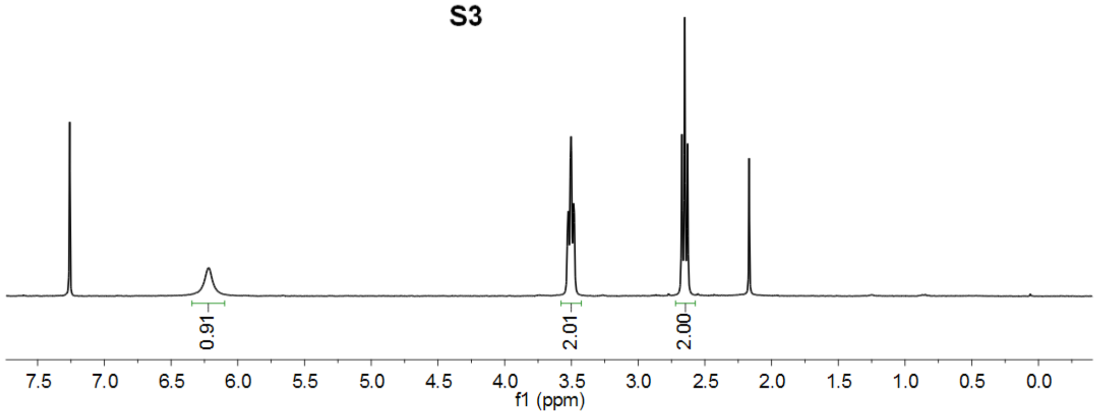

187.93
158.49
137.85
136.47
132.67
128.95
126.98
124.35
120.02
77.65
1l
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

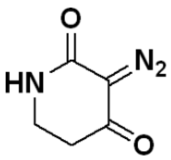

O
N2
O
S2
4.00
6.04
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


189.72
75.21
50.42
30.99
28.24
O
N2
O
S2
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

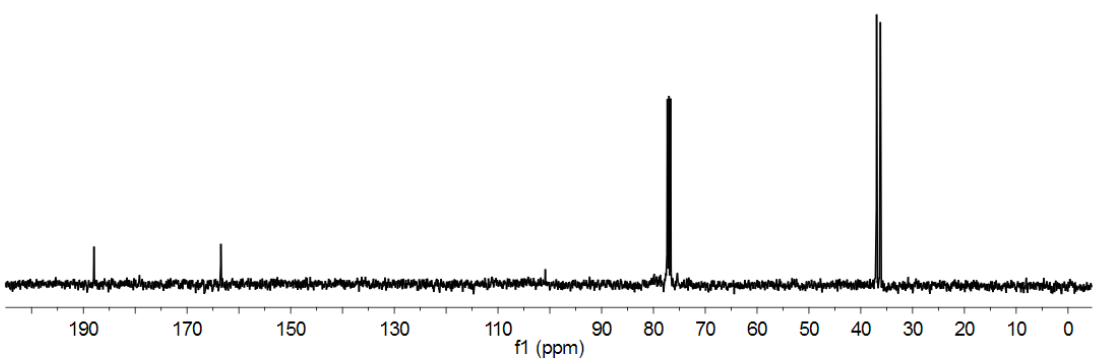

O
HN
N2
O

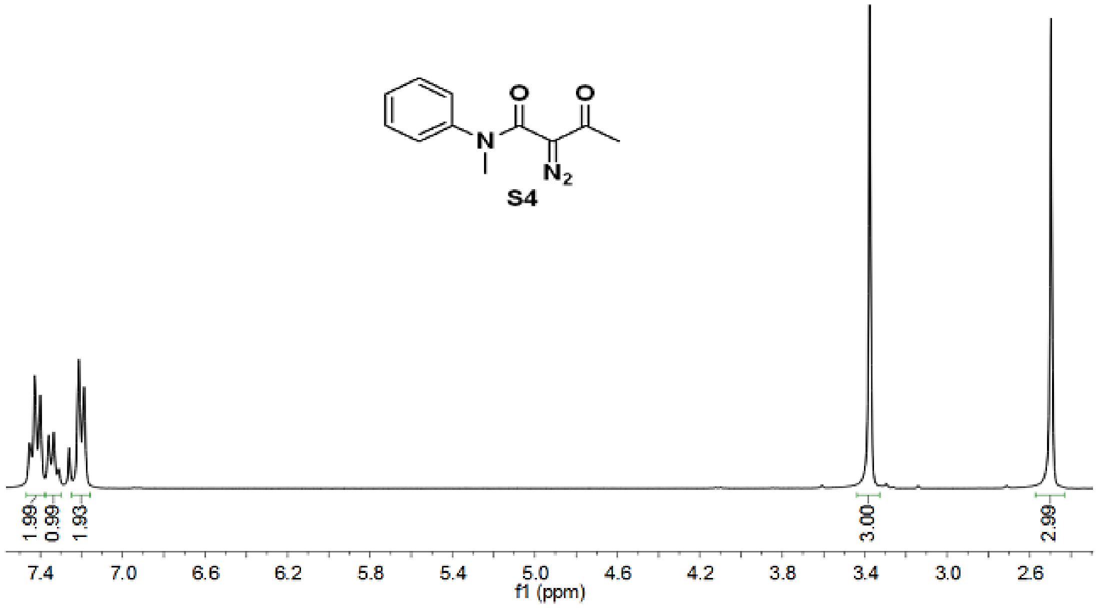
S3

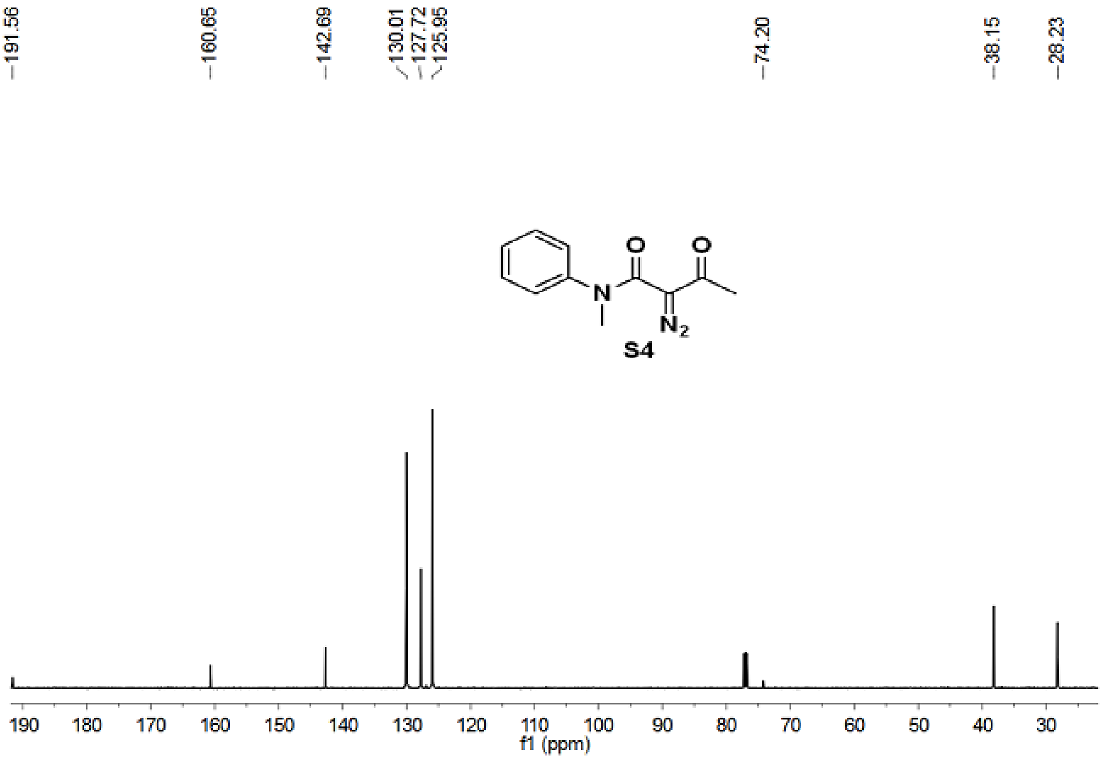

0.91
2.01
2.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

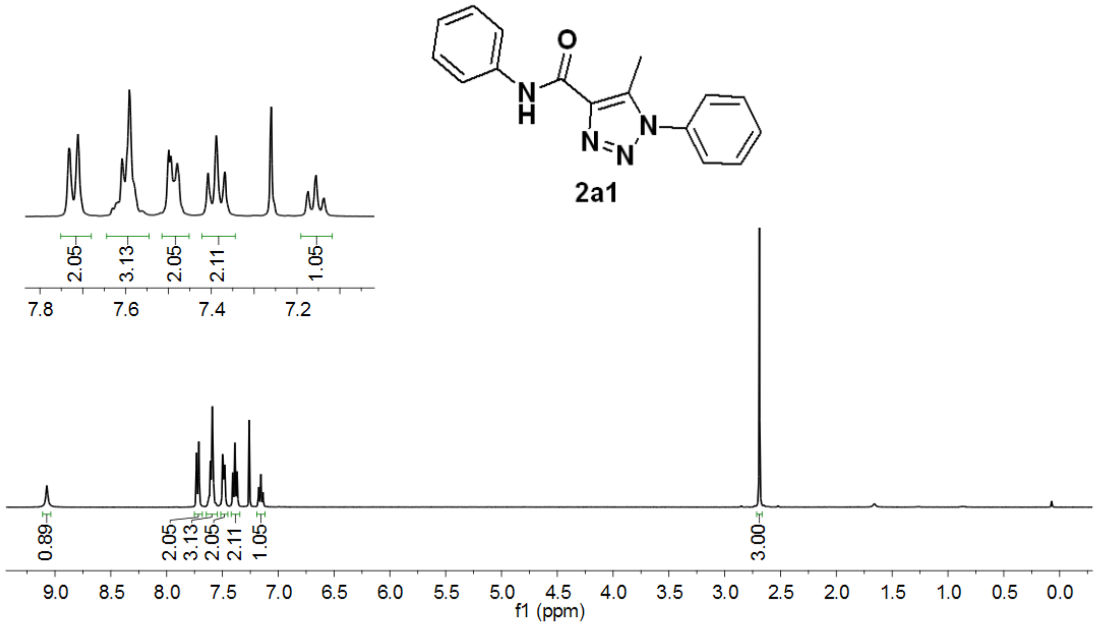

O
HN
N2
O

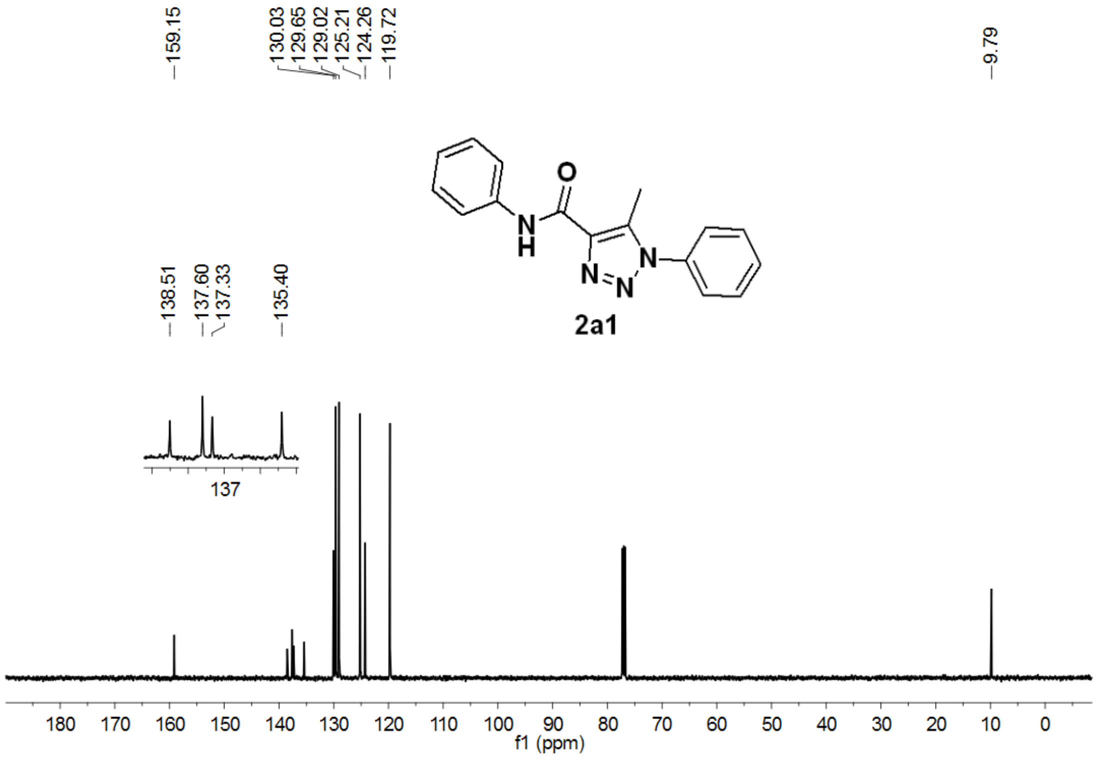
S3

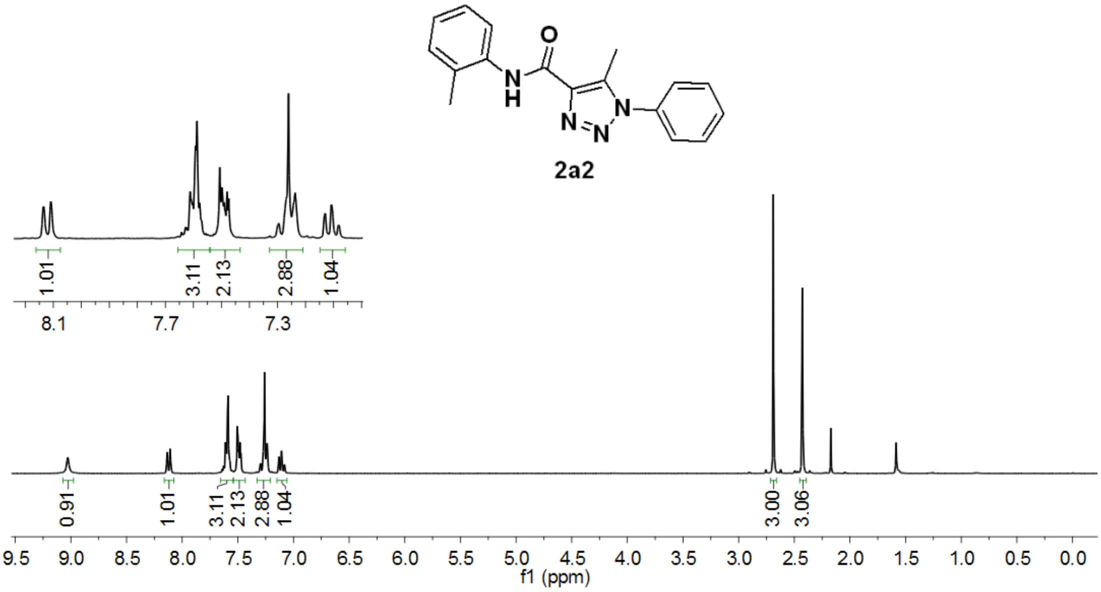

187.96
163.47
75.39
36.94
36.20
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

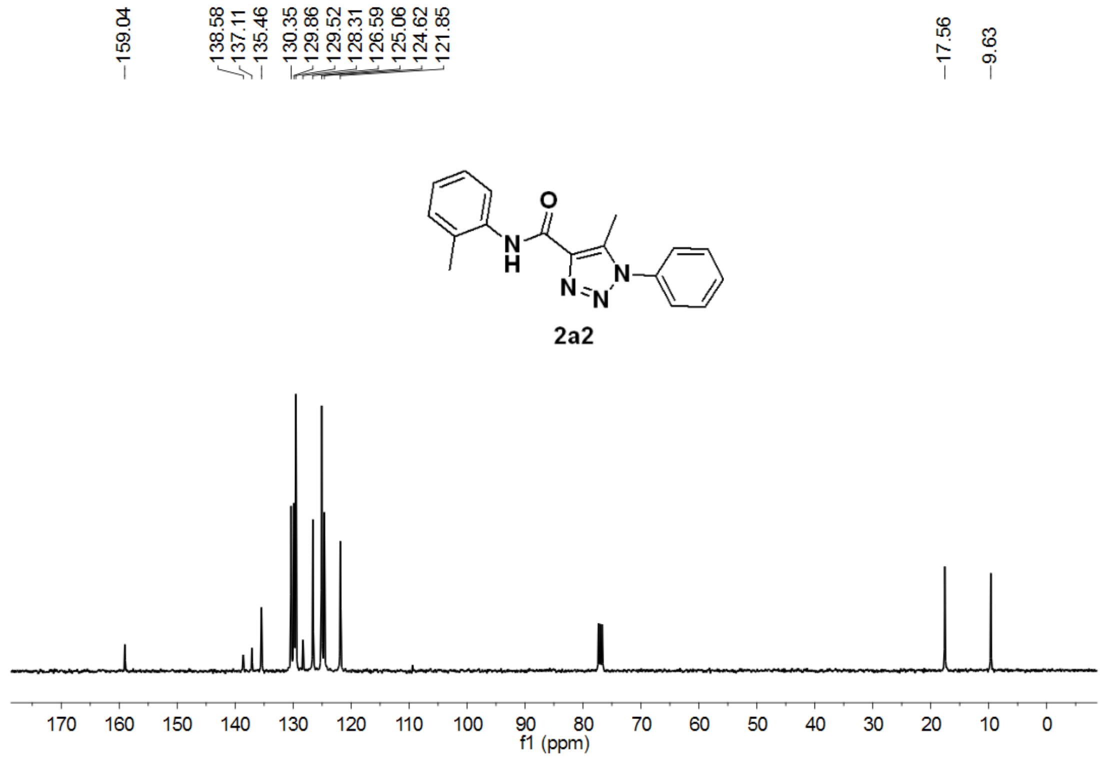

S4
1.99
0.99
1.93
3.00
2.99
f1 (ppm)

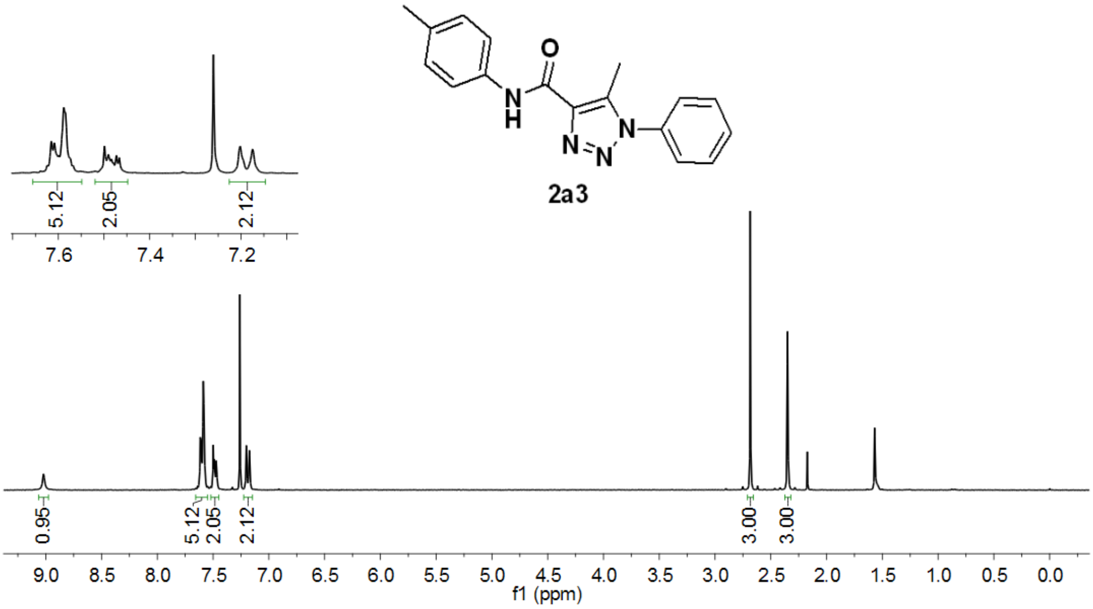

191.56
160.65
142.69
130.01
127.72
125.95
74.20
38.15
28.23
S4
f1 (ppm)

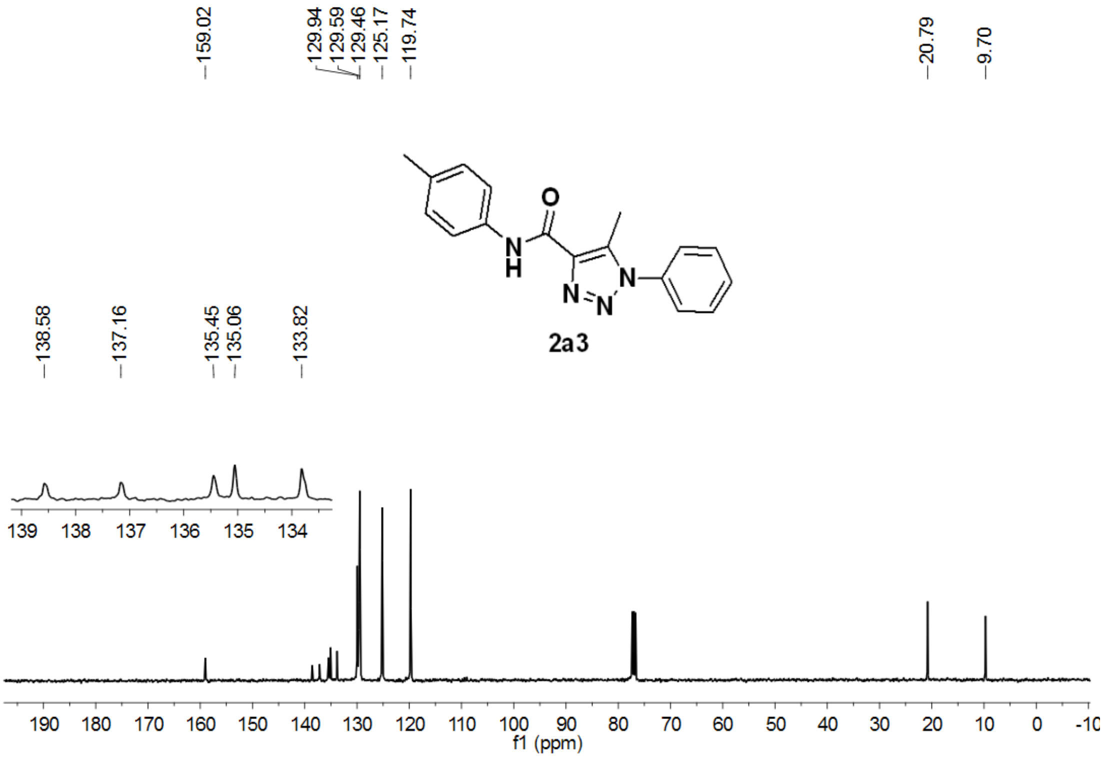

2a1
0.89
2.05
3.13
2.05
2.11
1.05
3.00
f1 (ppm)

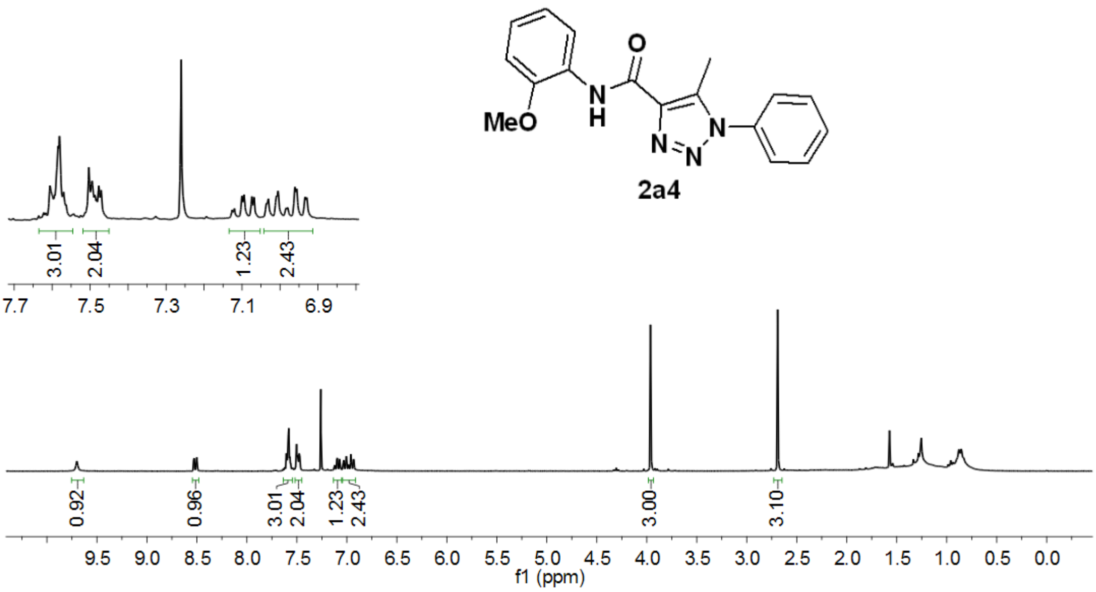

159.15
130.03
129.65
129.02
125.21
124.26
119.72
9.79
138.51
137.60
137.33
135.40
2a1
137
f1 (ppm)

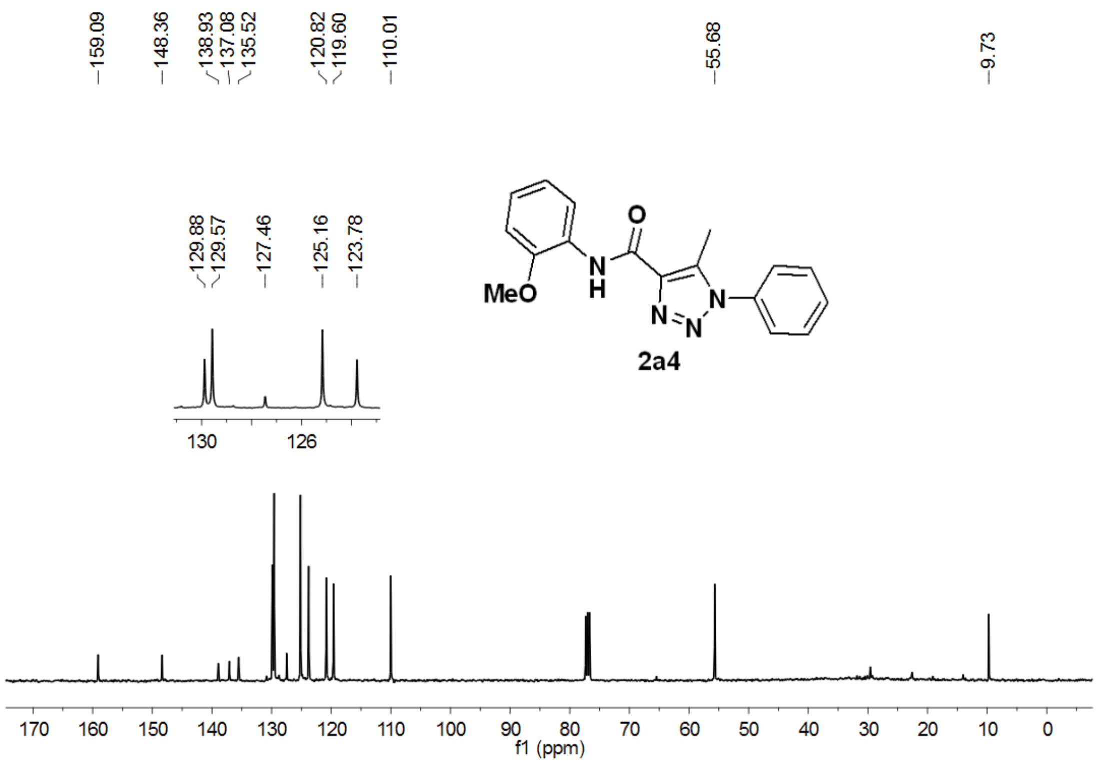

2a2
1.01
3.11
2.13
2.88
1.04
8.1
7.7
7.3
0.91
1.01
3.11
2.13
2.88
1.04
3.00
3.06
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

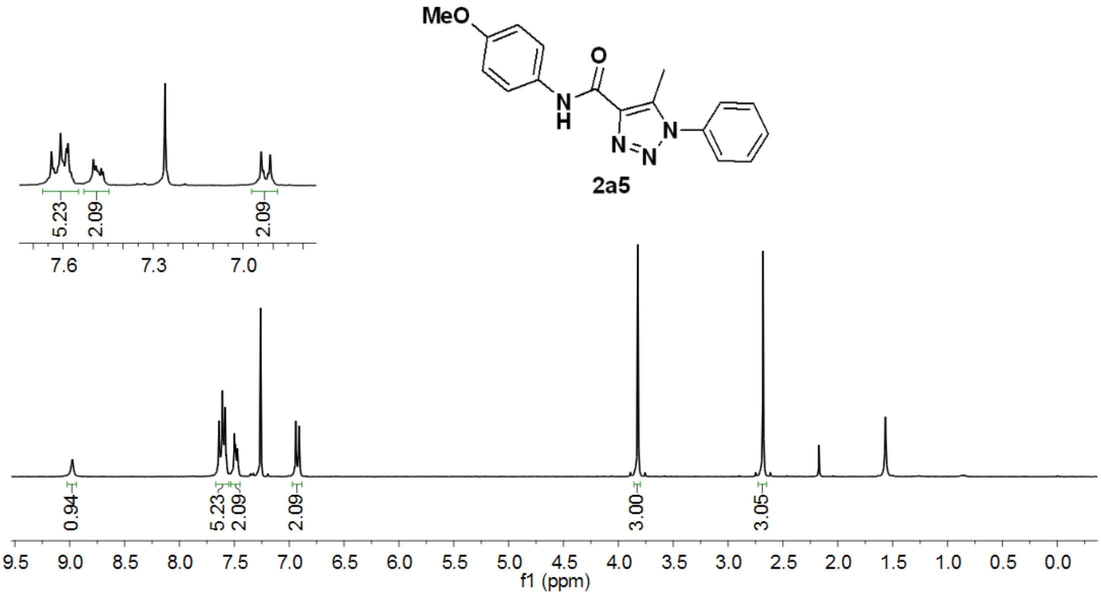

159.04
138.58
137.11
135.46
130.35
129.86
129.52
128.31
126.59
125.06
124.62
121.85
17.56
9.63
2a2
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

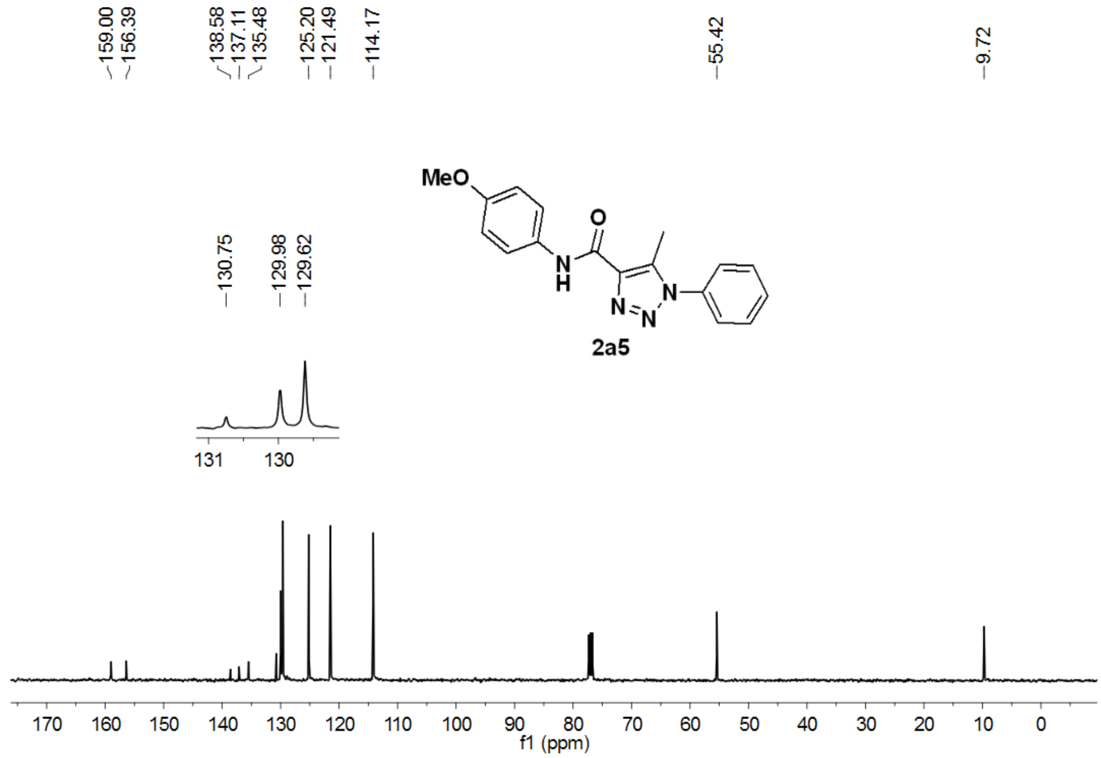

2a3
5.12
2.05
2.12
7.6
7.4
7.2
0.95
5.12
2.05
2.12
3.00
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

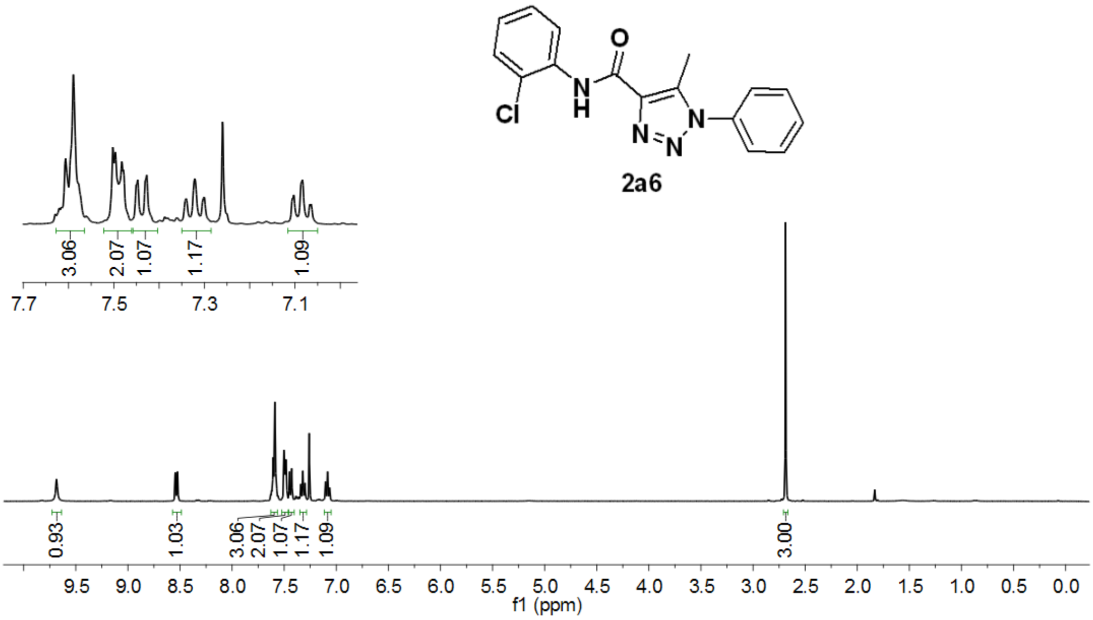

159.02
129.94
129.59
129.46
125.17
119.74
20.79
9.70
2a3
138.58
137.16
135.45
135.06
133.82
139 138 137 136 135 134
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

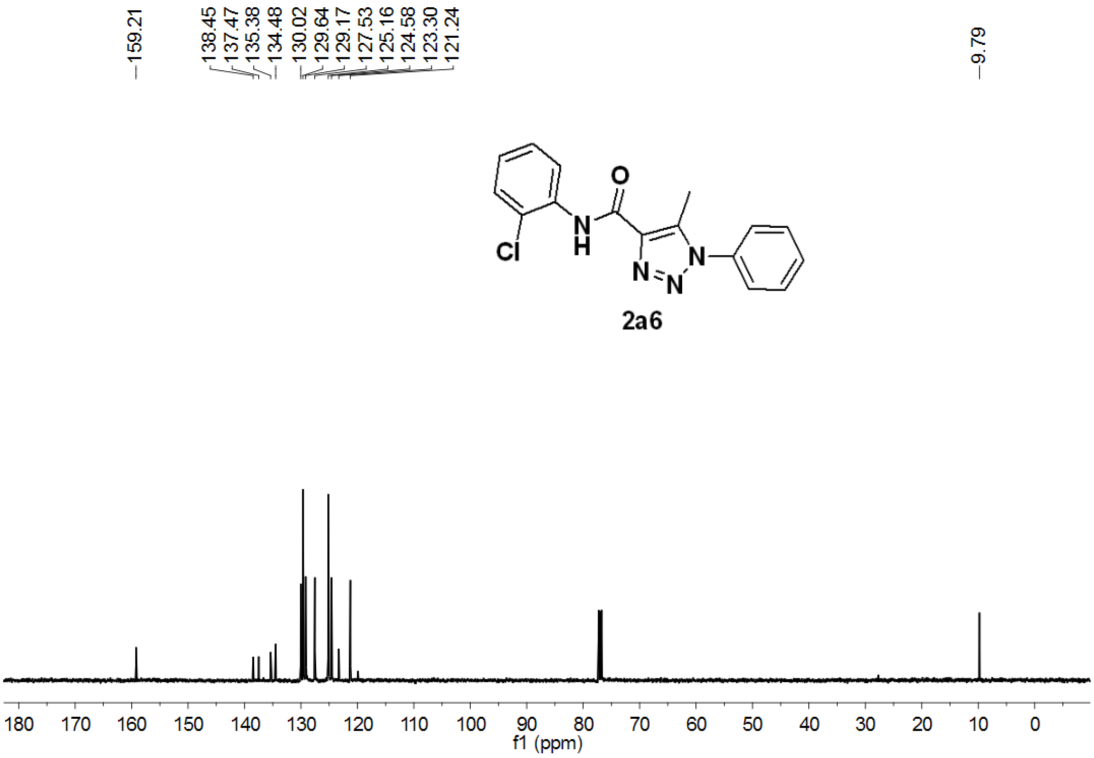

MeO
N
H
O
N
N
N
2a4
3.01
2.04
1.23
2.43
7.7
7.5
7.3
7.1
6.9
0.92
0.96
3.01
2.04
1.23
2.43
3.00
3.10
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

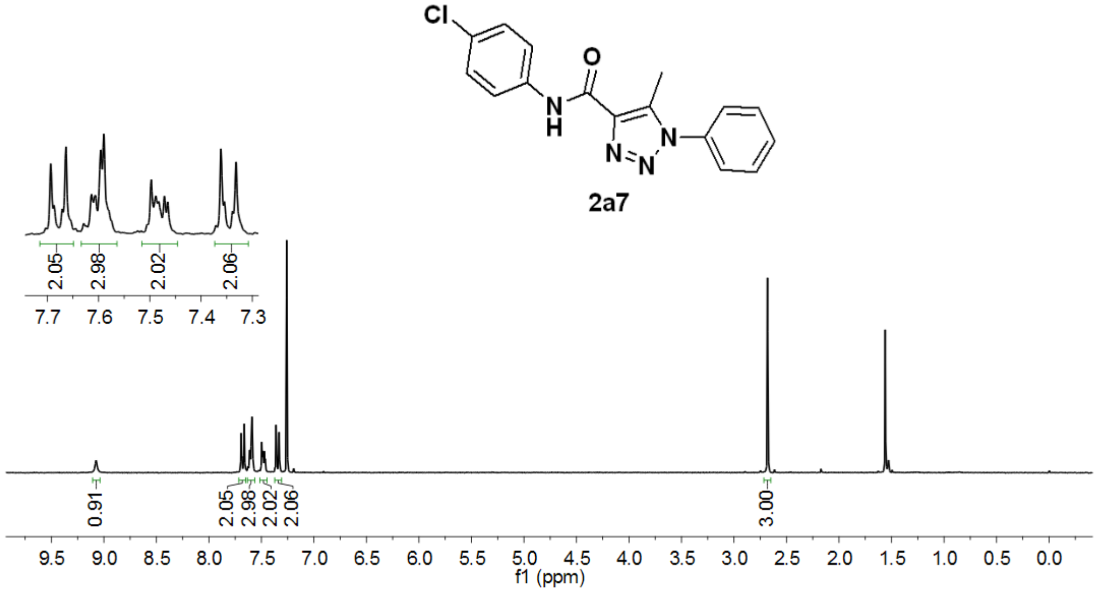

159.09
148.36
138.93
137.08
135.52
120.82
119.60
110.01
55.68
9.73
129.88
129.57
127.46
125.16
123.78
130
126
MeO
N
H
O
N
N
N
2a4
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

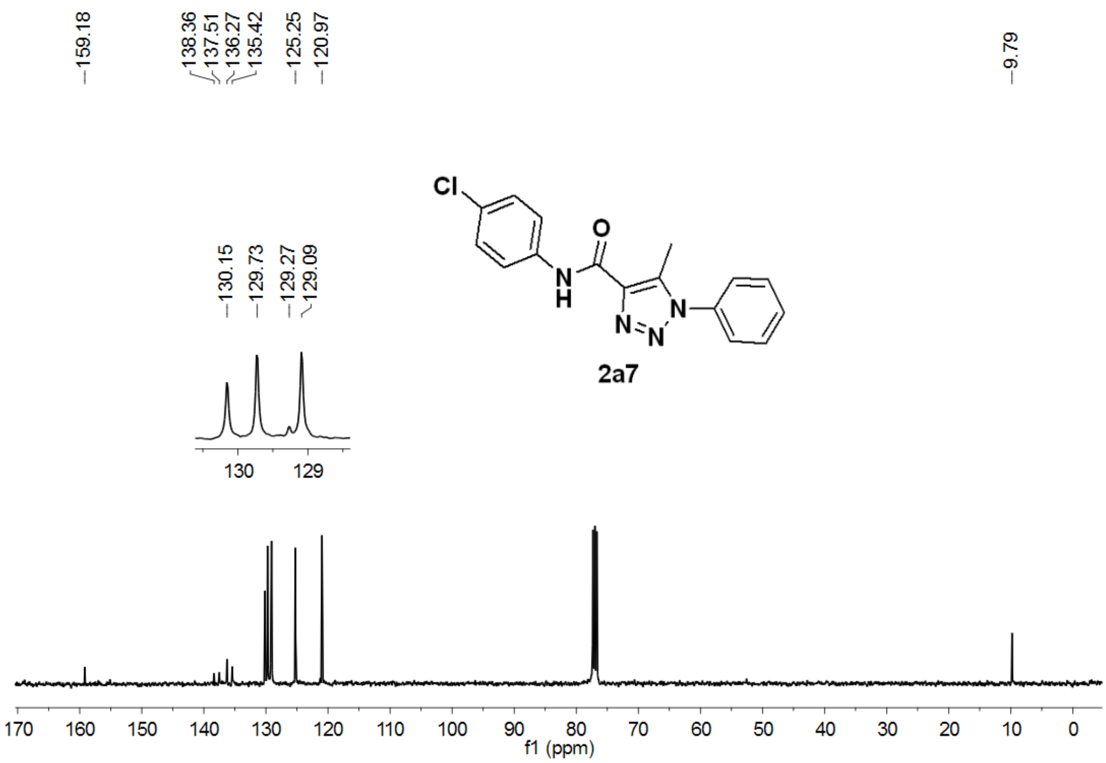

MeO
O
N
H
N
N
N
2a5
5.23
2.09
2.09
7.6
7.3
7.0
0.94
5.23
2.09
2.09
3.00
3.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

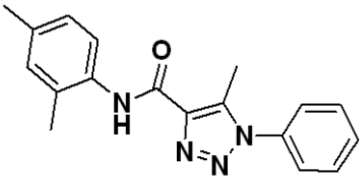

159.00
156.39
138.58
137.11
135.48
125.20
121.49
114.17
55.42
9.72
MeO
O
N
H
N
N
N
2a5
130.75
129.98
129.62
131
130
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
N
H
Cl
N
N
N
2a6
3.06
2.07
1.07
1.17
1.09
7.7
7.5
7.3
7.1
0.93
1.03
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

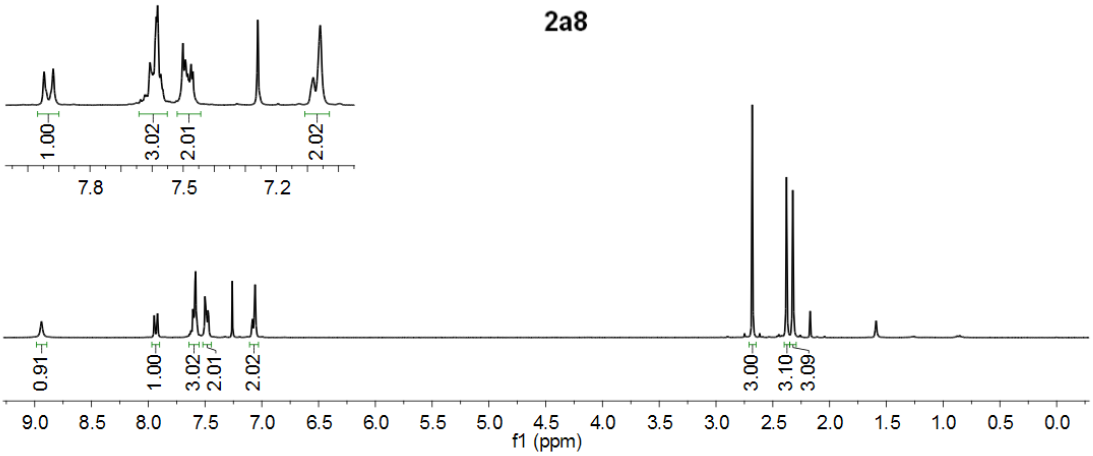

159.21
138.45
137.47
135.38
134.48
130.02
129.64
129.17
127.53
125.16
124.58
123.30
121.24
9.79
O
N
H
Cl
N
N
N
2a6
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

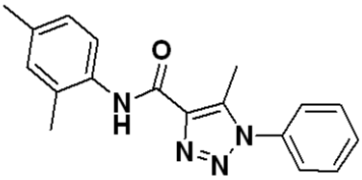

Cl
O
N
H
N
N
N
2a7
2.05
2.98
2.02
2.06
7.7
7.6
7.5
7.4
7.3
0.91
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


159.18
138.36
137.51
136.27
135.42
125.25
120.97
9.79
Cl
O
N
H
N
N
N
2a7
130.15
129.73
129.27
129.09
130
129
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

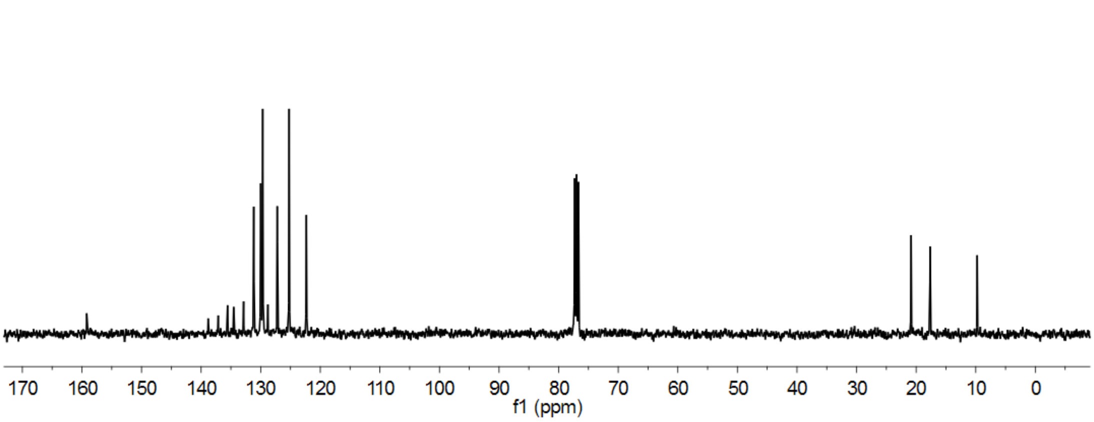


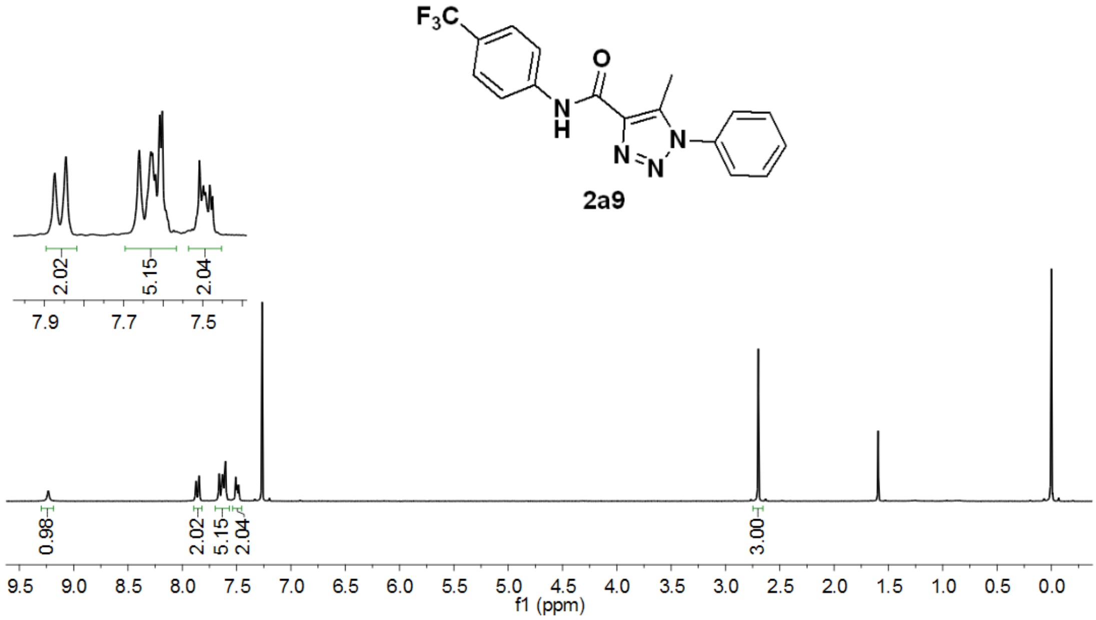
2a8

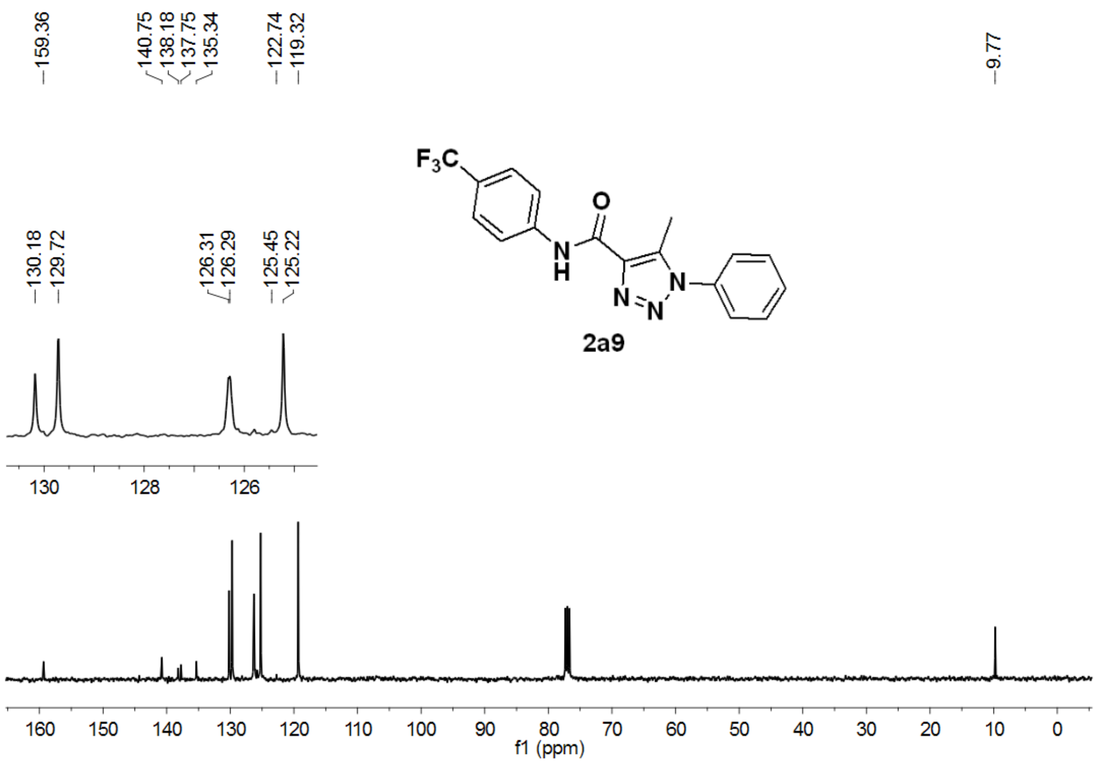




2a8




F3C
O
N
H
N
N
N
2a9
2.02
5.15
2.04
7.9
7.7
7.5
0.98
2.02
5.15
2.04
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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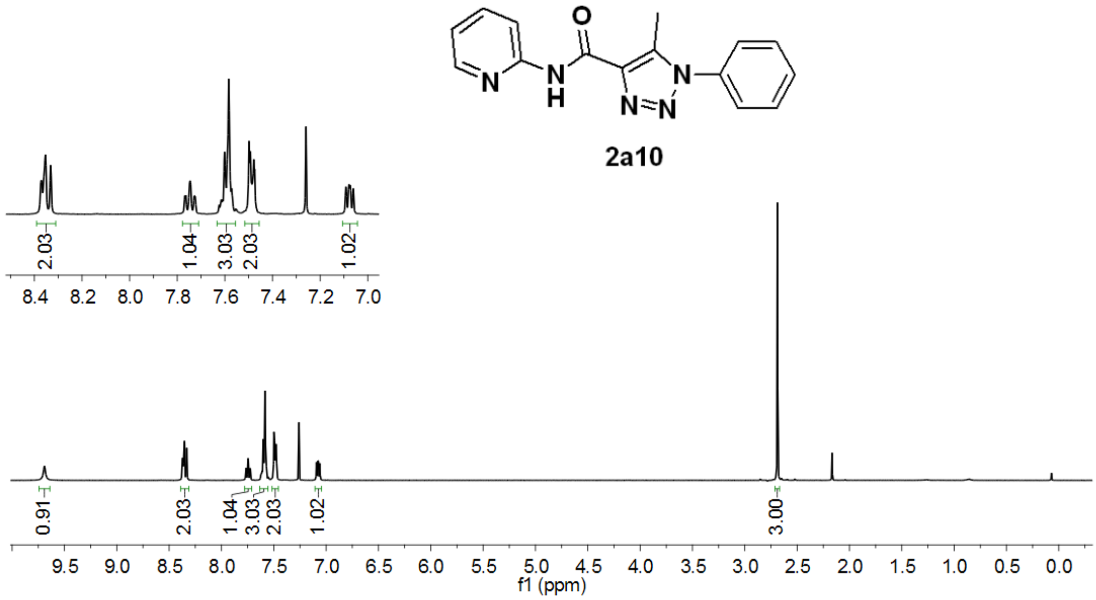
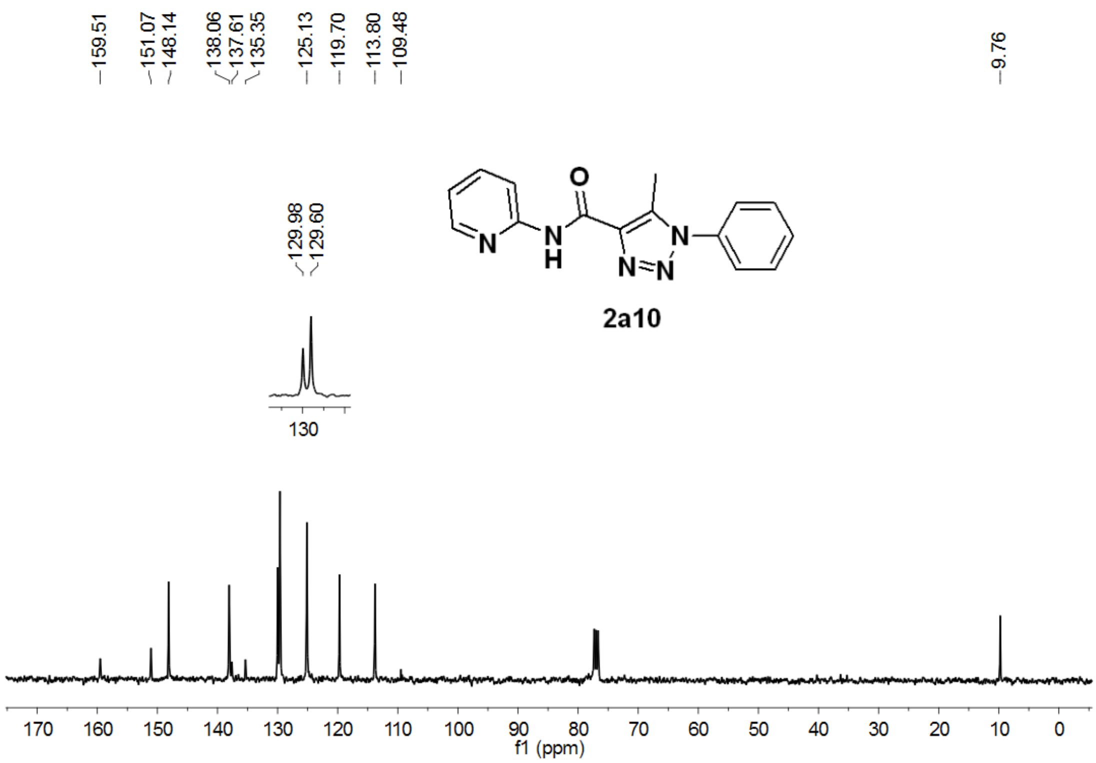
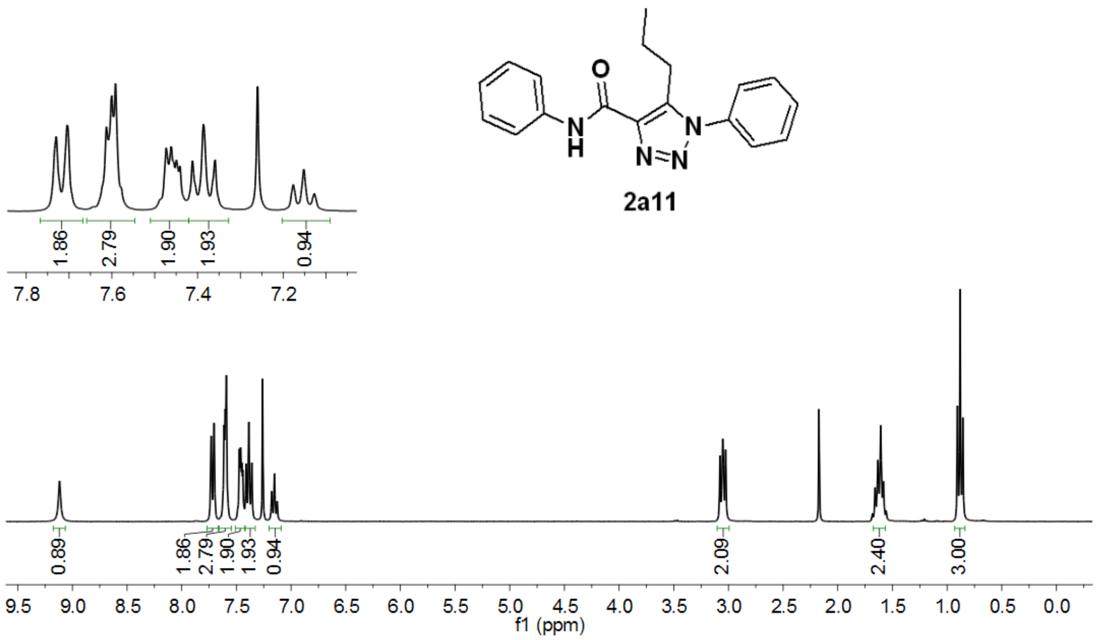
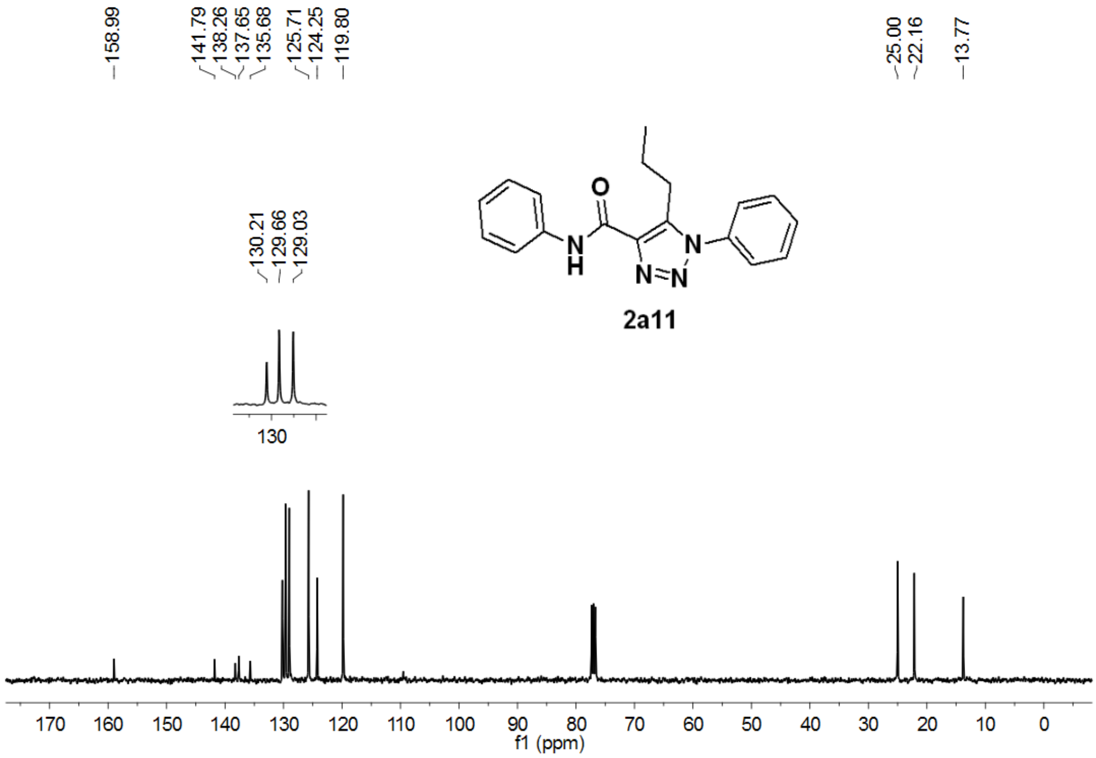
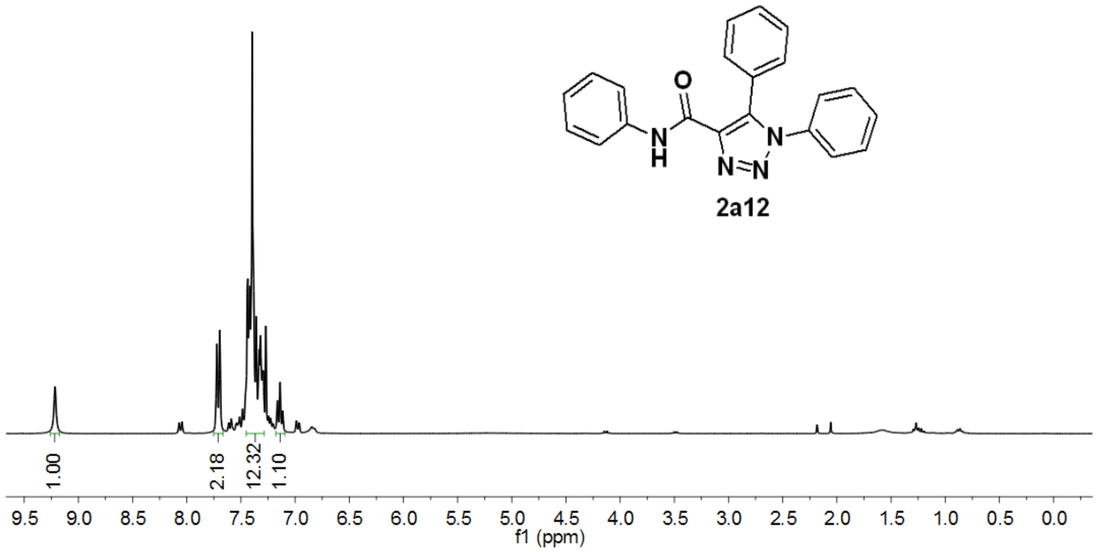
140.75
138.18
137.75
135.34
122.74
119.32
9.77
130.18
129.72
126.31
126.29
125.45
125.22
130
128
126
F3C
O
N
H
N
N
N
2a9
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

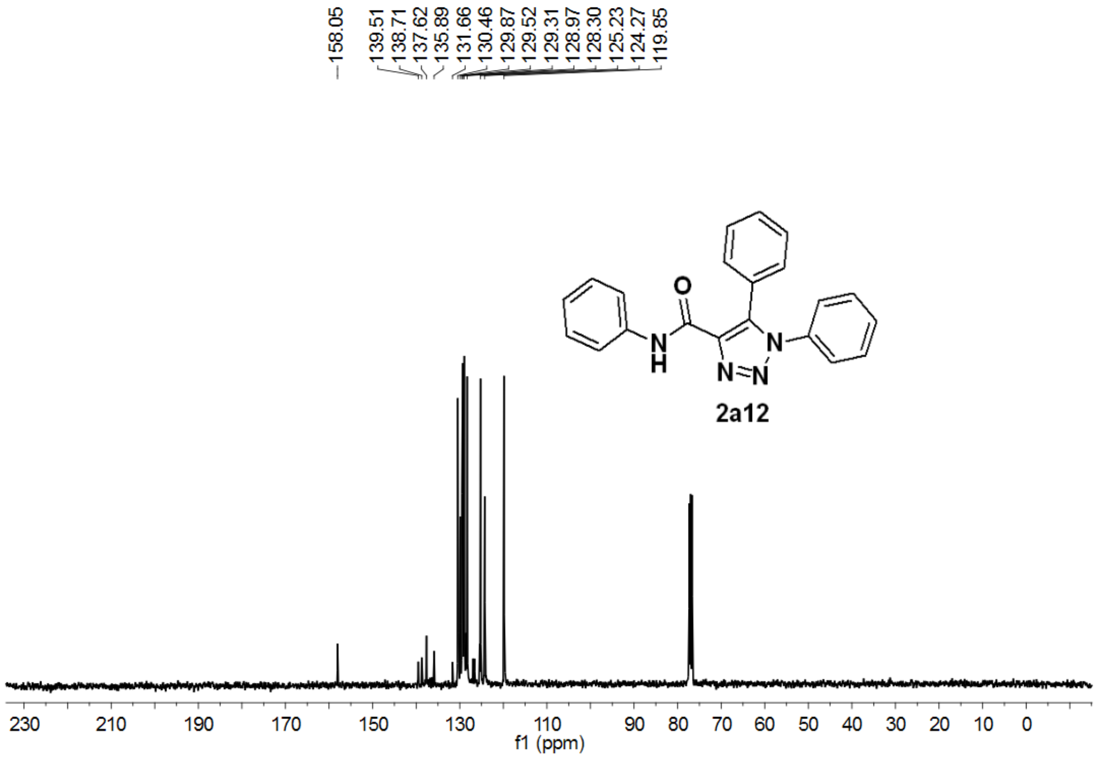

2a10
2.03
1.04
3.03
2.03
1.02
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
0.91
2.03
1.04
3.03
2.03
1.02
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

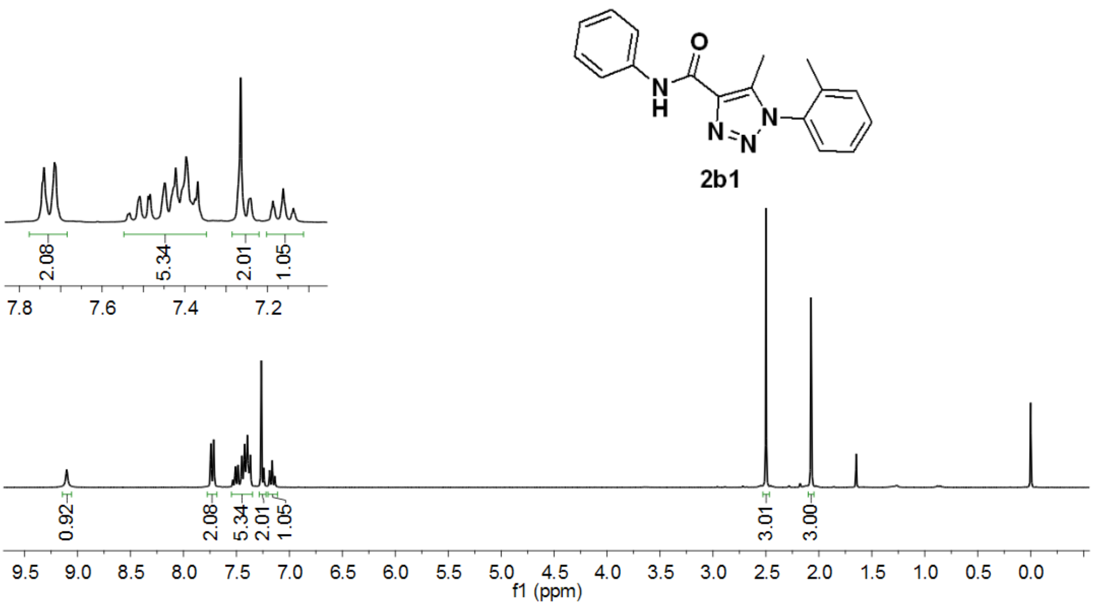

159.51
151.07
148.14
138.06
137.61
135.35
125.13
119.70
113.80
109.48
9.76
129.98
129.60
130
2a10
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

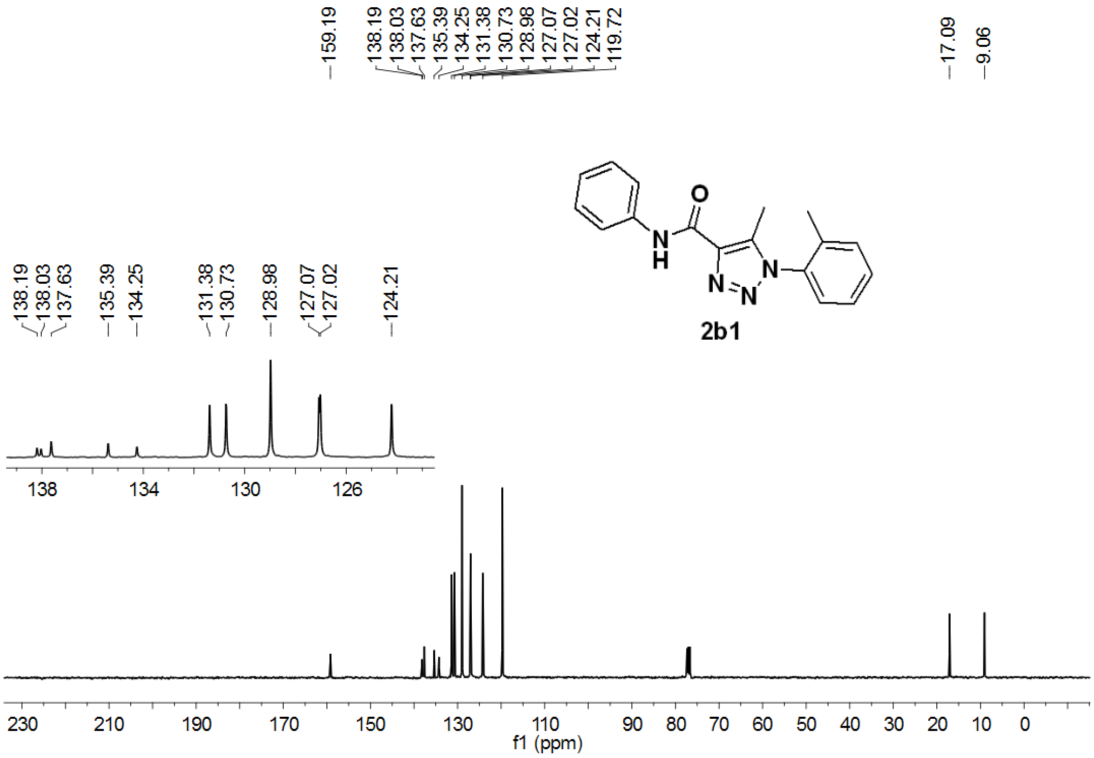

O
N
H
N
N=N
2a11
1.86
2.79
1.90
1.93
0.94
7.8
7.6
7.4
7.2
0.89
2.09
2.40
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

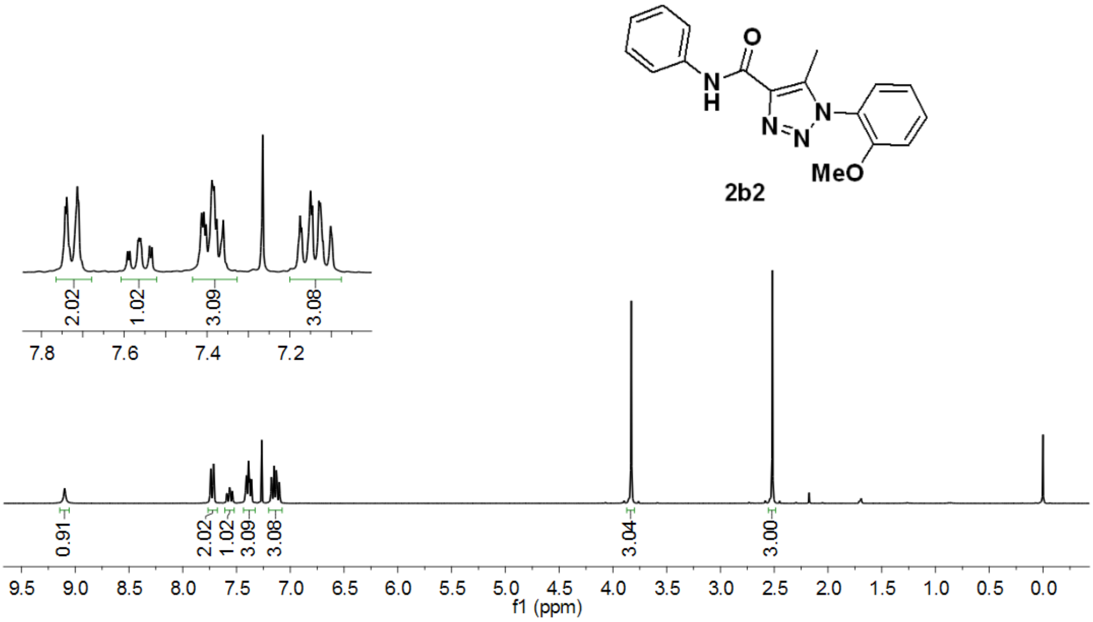

158.99
141.79
138.26
137.65
135.68
125.71
124.25
119.80
25.00
22.16
13.77
130.21
129.66
129.03
130
O
N
H
N
N=N
2a11
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

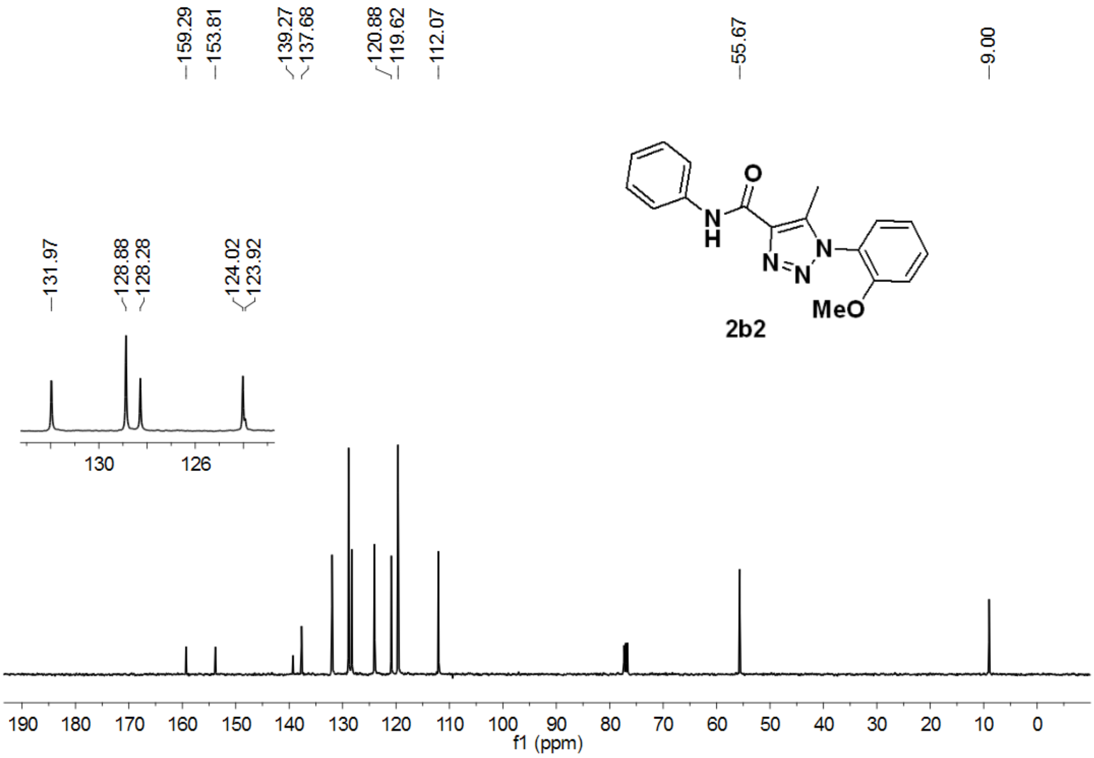

2a12
1.00
2.18
12.32
1.10
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

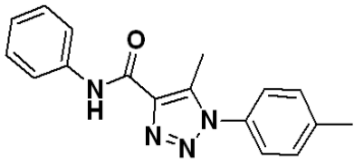

158.05
139.51
138.71
137.62
135.89
131.66
130.46
129.87
129.52
129.31
128.97
128.30
125.23
124.27
119.85
2a12
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


2b1
2.08
5.34
2.01
1.05
7.8
7.6
7.4
7.2
0.92
3.01
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

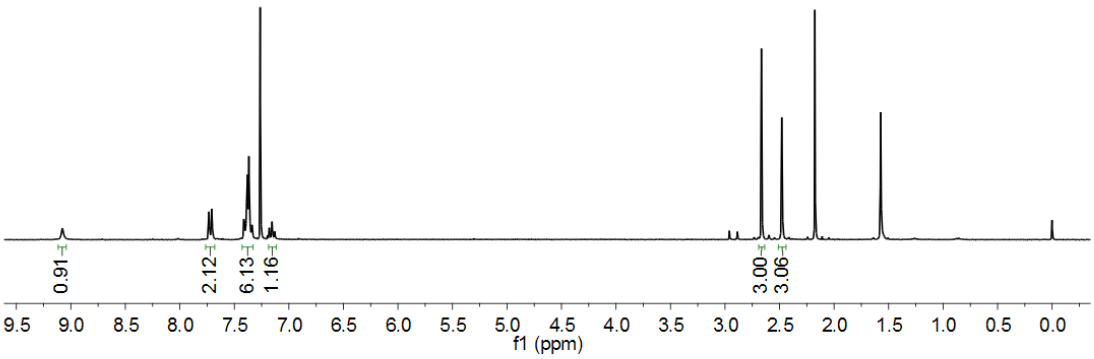

159.19
138.19
138.03
137.63
135.39
134.25
131.38
130.73
128.98
127.07
127.02
124.21
119.72
17.09
9.06
2b1
138
134
130
126
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

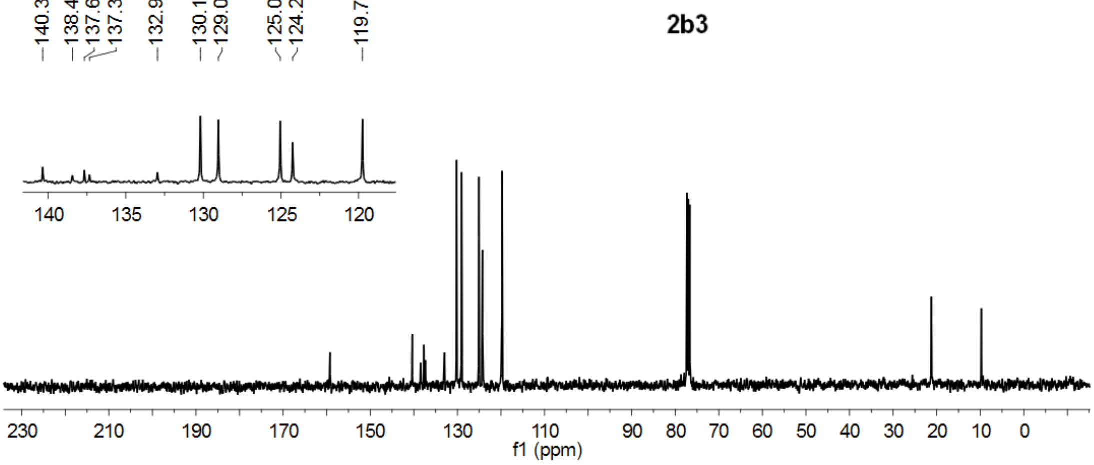

2b2
MeO
2.02
1.02
3.09
3.08
7.8
7.6
7.4
7.2
0.91
2.02
1.02
3.09
3.08
3.04
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

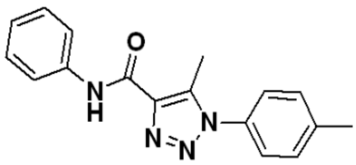

159.29
153.81
139.27
137.68
120.88
119.62
112.07
55.67
9.00
2b2
MeO
131.97
128.88
128.28
124.02
123.92
130
126
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
N
H
N
N
N

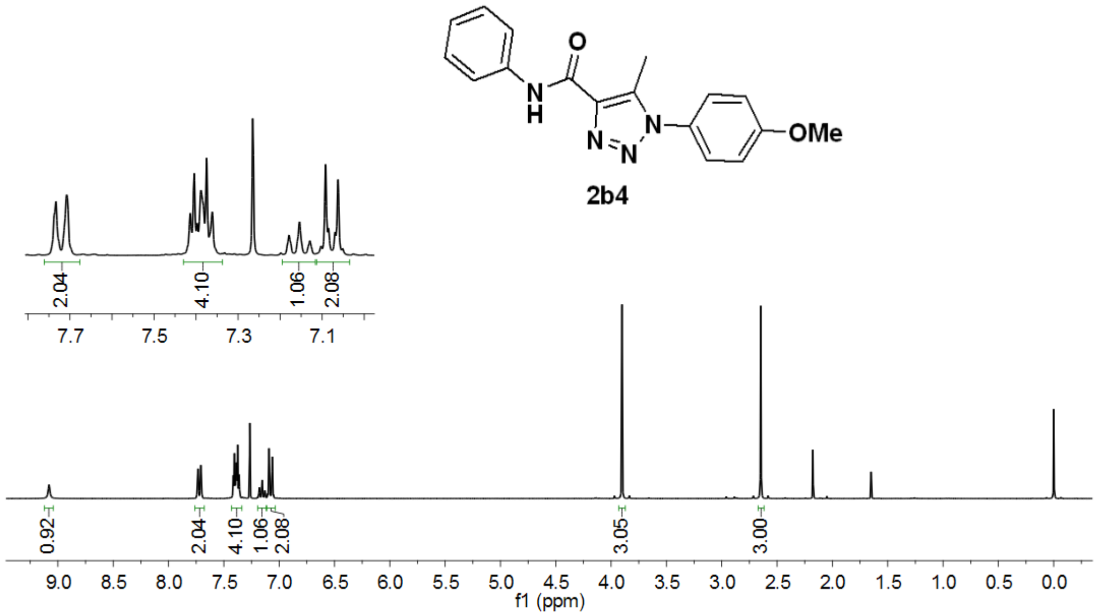
2b3

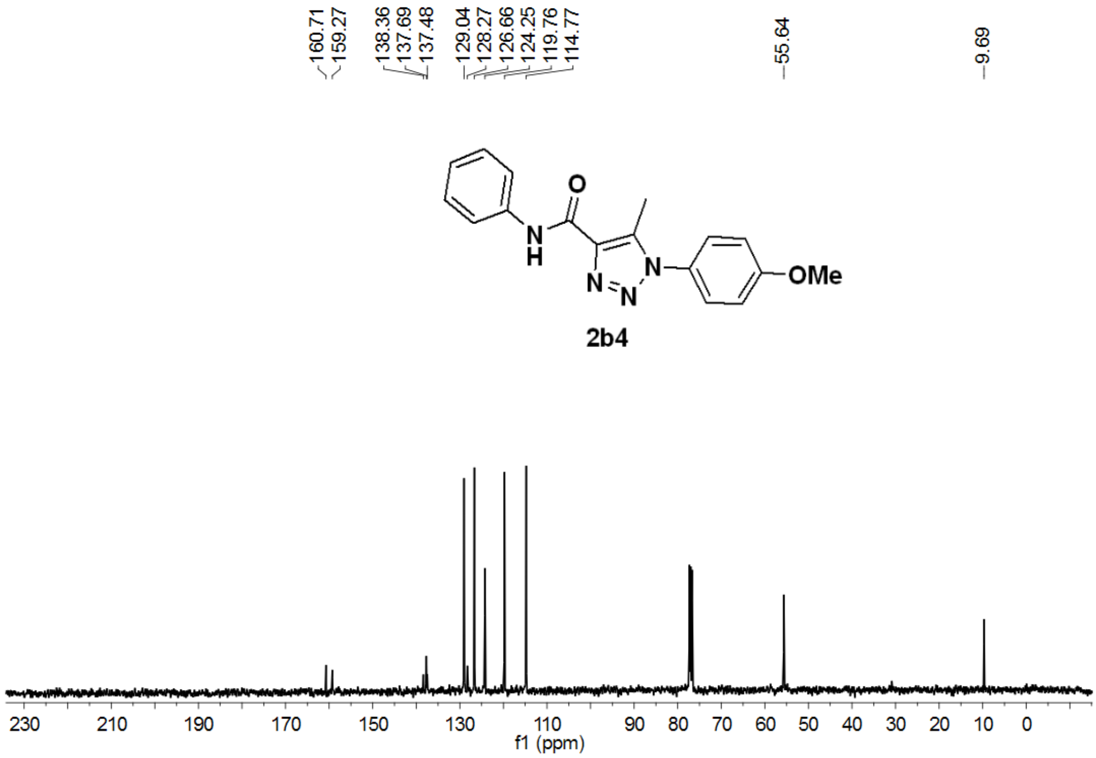

0.91
2.12
6.13
1.16
3.00
3.06
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.25
140.35
138.44
137.67
137.34
132.96
130.19
129.03
125.04
124.24
119.75
21.22
9.74
140 135 130 125 120
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
N
H
N
N
N

2b3


OMe
2b4
2.04
4.10
1.06
2.08
7.7
7.5
7.3
7.1
0.92
2.04
4.10
1.06
2.08
3.05
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


160.71
159.27
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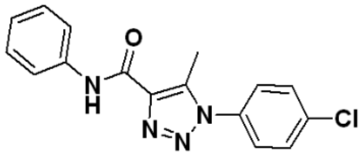
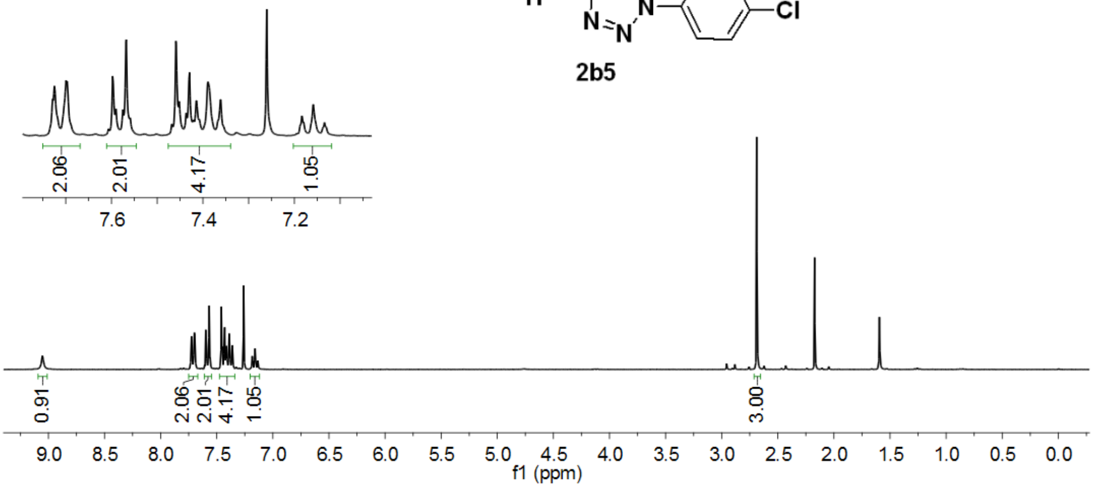
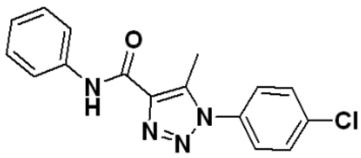
137.69
137.48
129.04
128.27
126.66
124.25
119.76
114.77
55.64
9.69
OMe
2b4
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

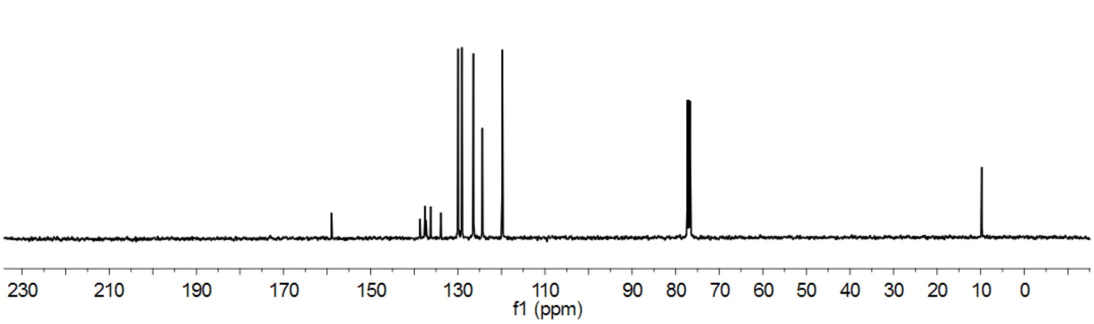

O
N
H
N
N
N
Cl

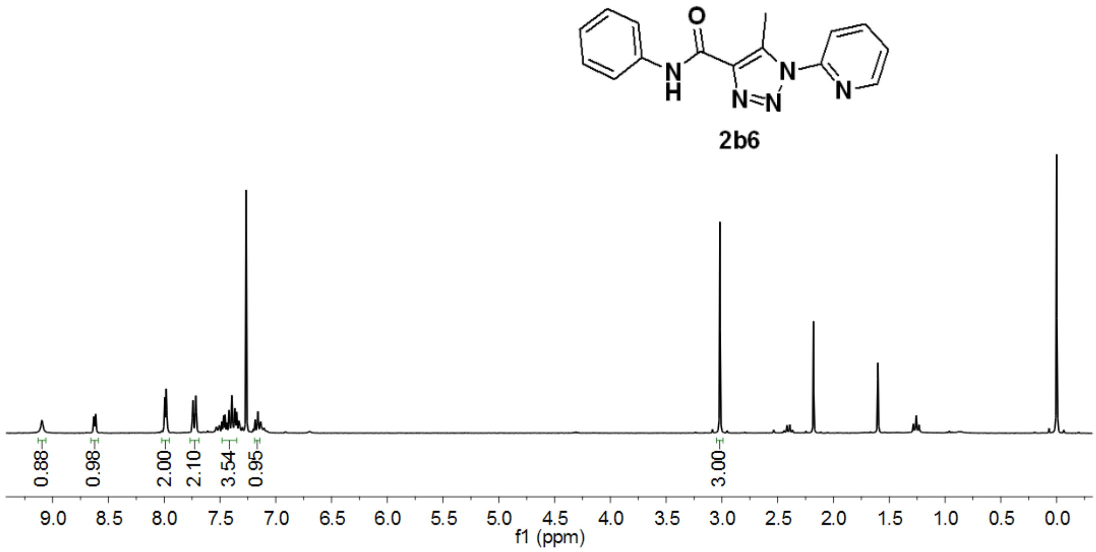
2b5

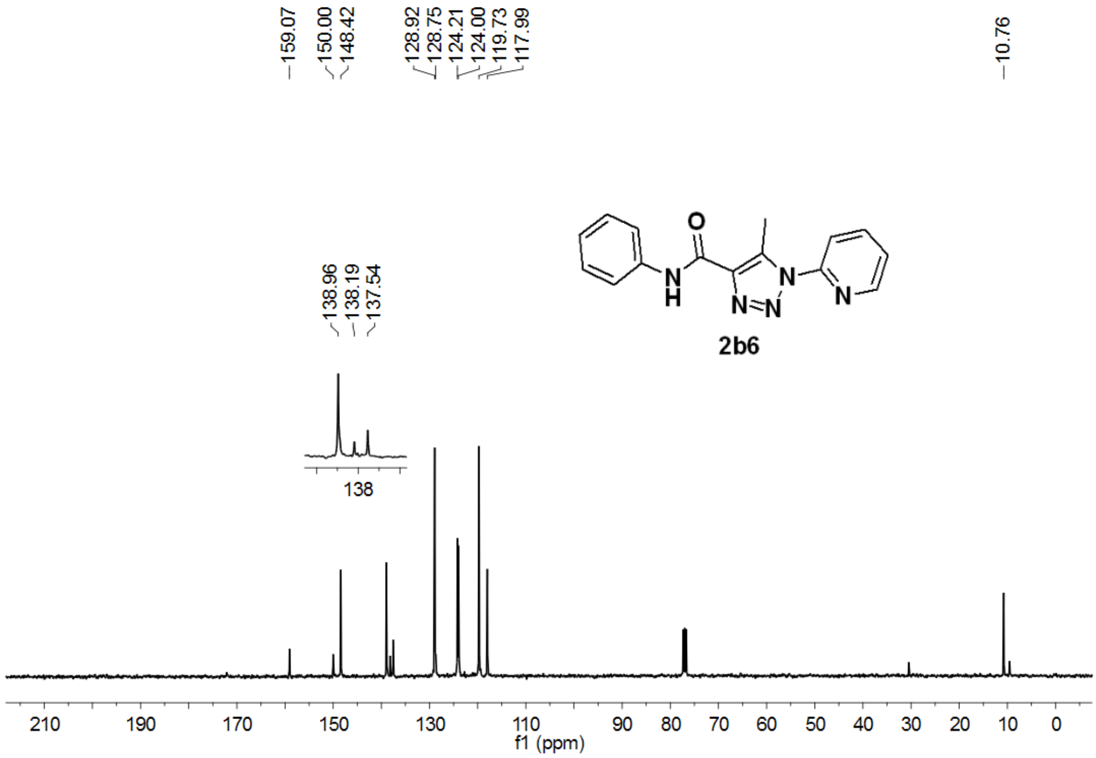

2.06
2.01
4.17
1.05
7.6
7.4
7.2
0.91
2.06
2.01
4.17
1.05
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


O
N
H
N
N
N
Cl

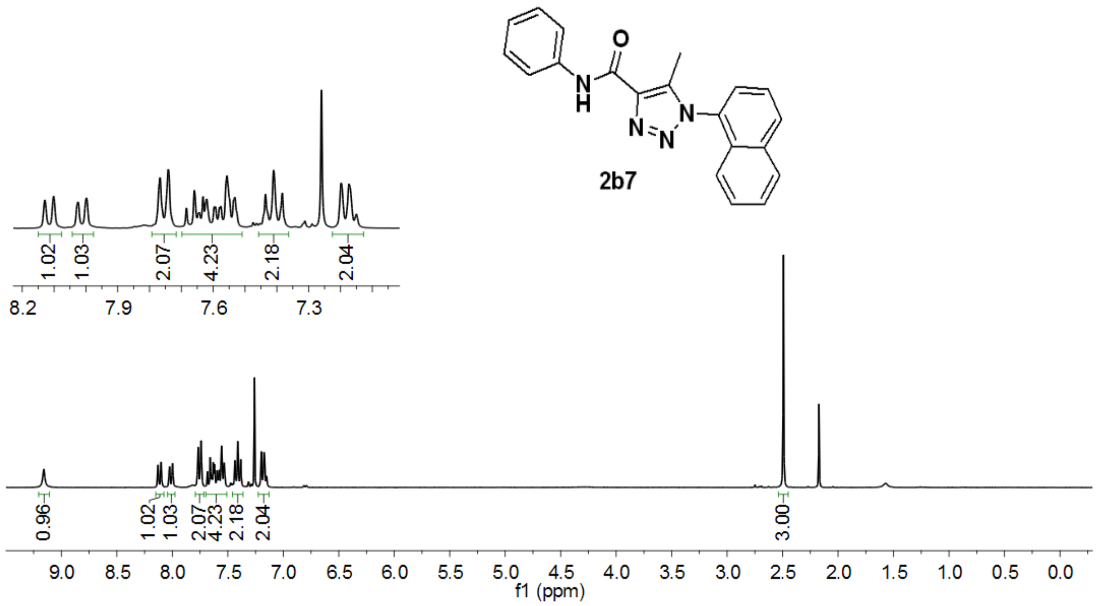
2b5

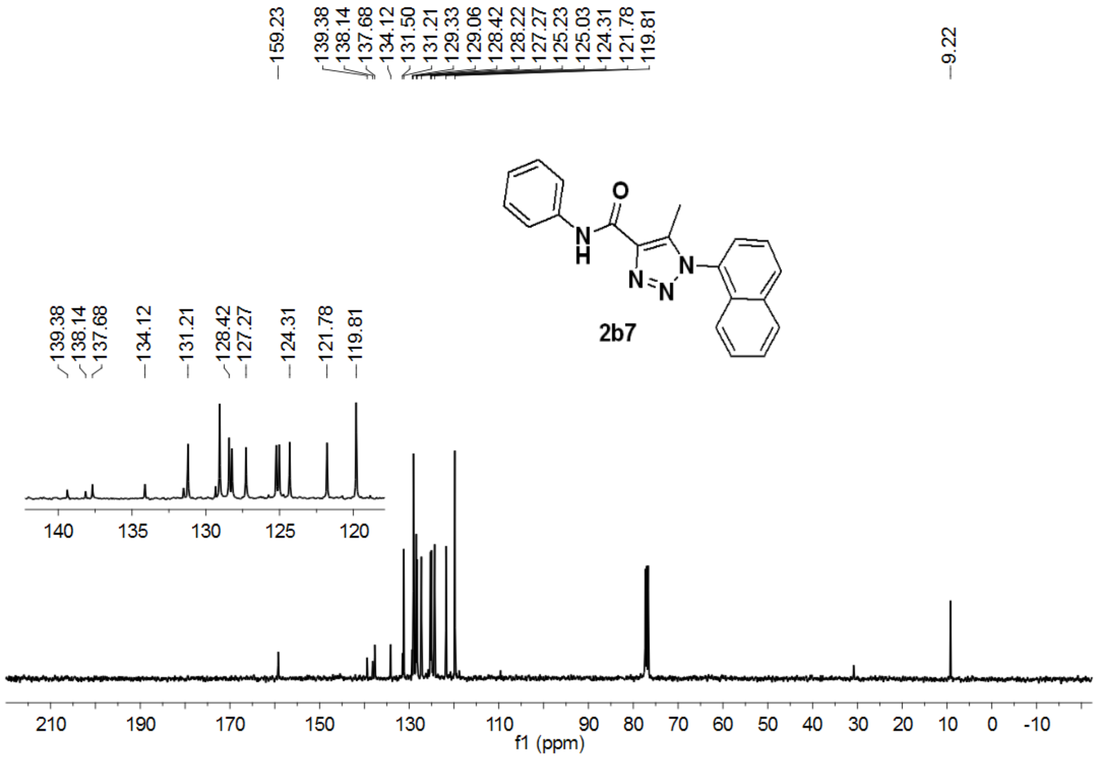

158.99
138.69
137.55
137.34
136.23
133.90
129.94
129.05
126.43
124.37
119.79
9.76
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

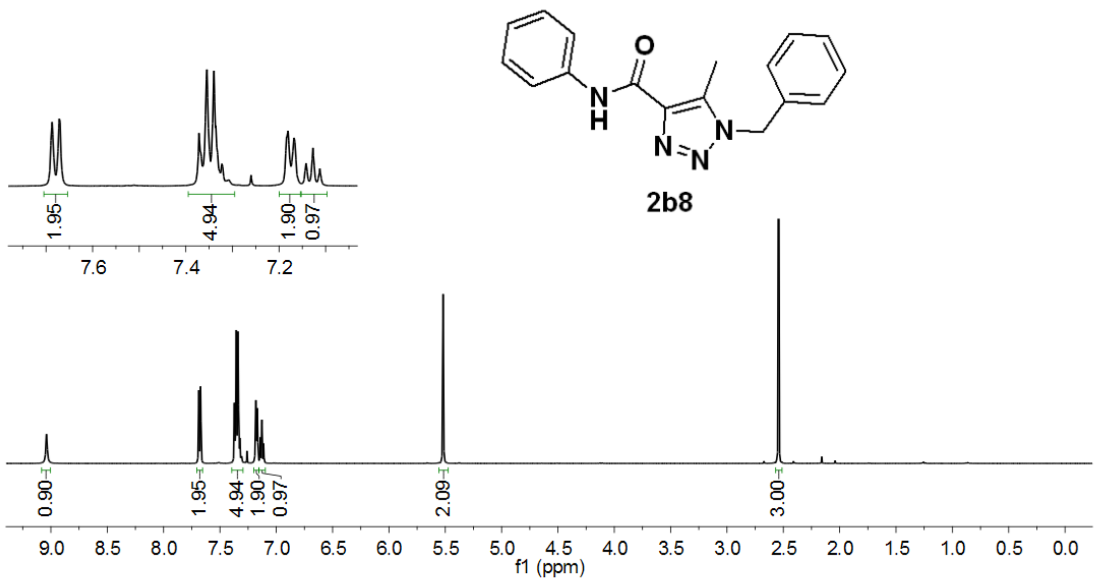

2b6
0.88
0.98
2.00
2.10
3.54
0.95
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

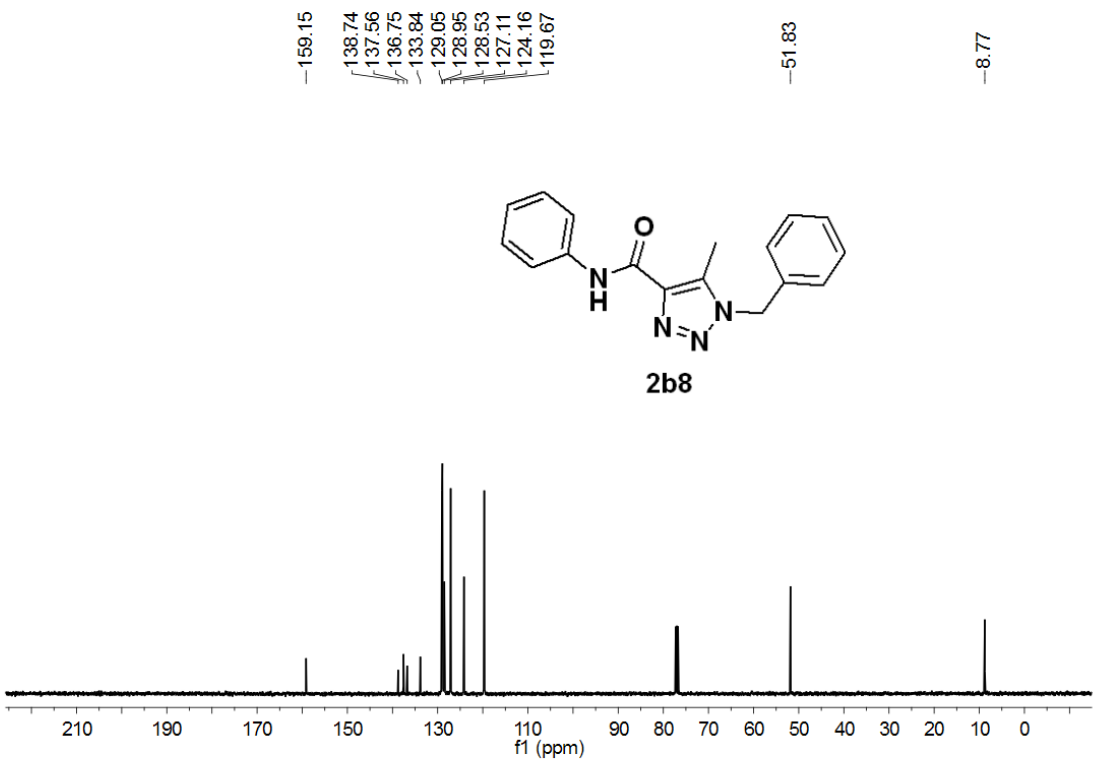

159.07
150.00
148.42
128.92
128.75
124.21
124.00
119.73
117.99
10.76
138.96
138.19
137.54
138
2b6
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

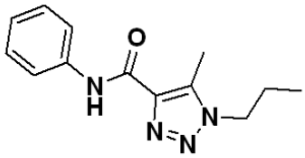
Electronic Supplementary Material (ESI) for Chemical Communications



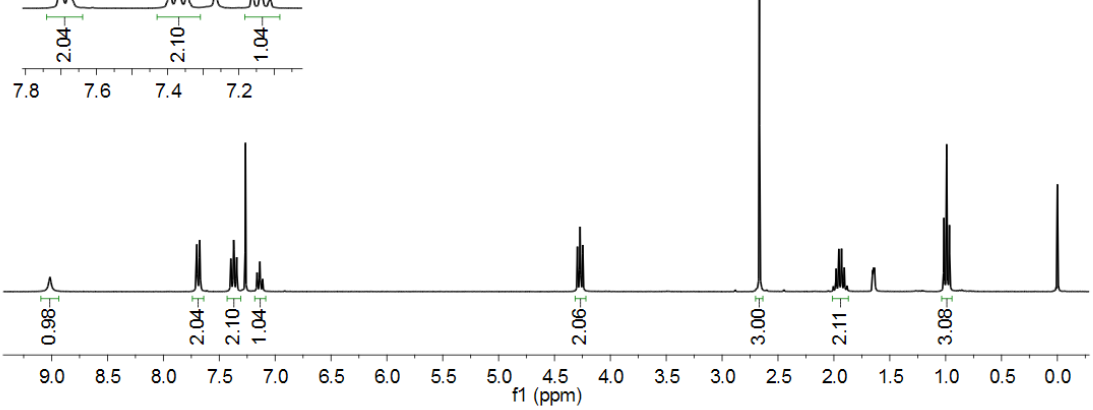


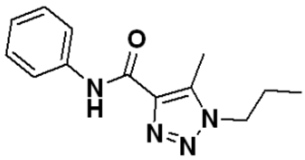

2b8
1.95
4.94
1.90
0.97
7.6
7.4
7.2
0.90
1.95
4.94
1.90
0.97
2.09
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.15
138.74
137.56
136.75
133.84
129.05
128.95
128.53
127.11
124.16
119.67
51.83
8.77
2b8
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

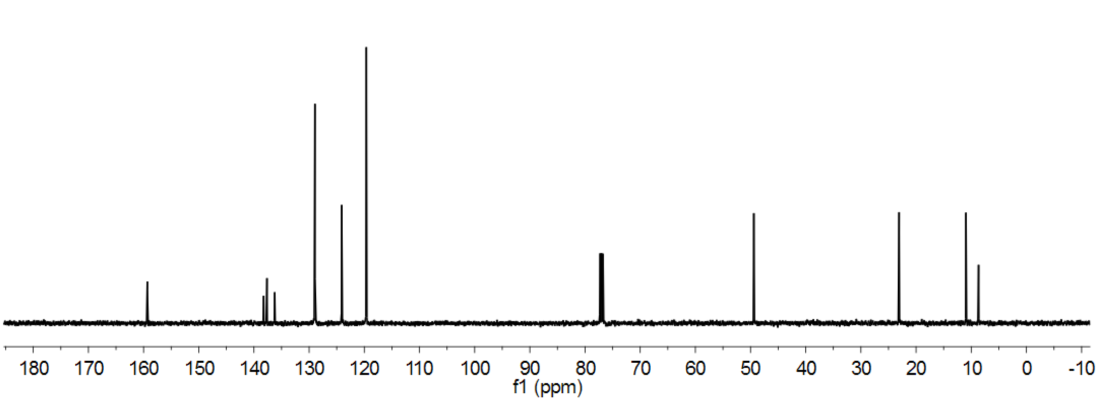

O
N
H
N
N
N

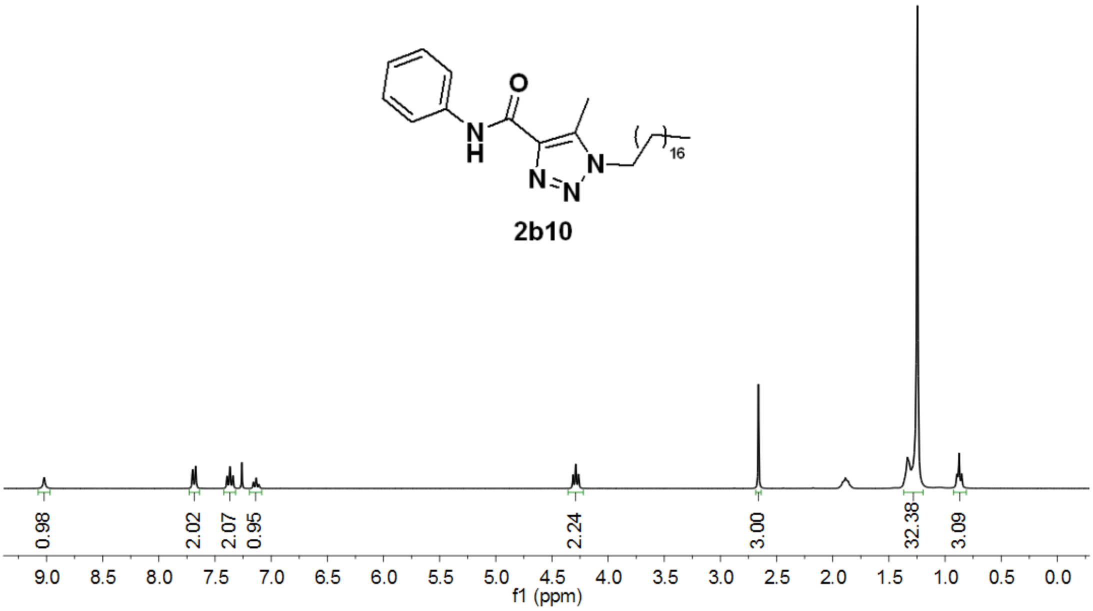
2b9

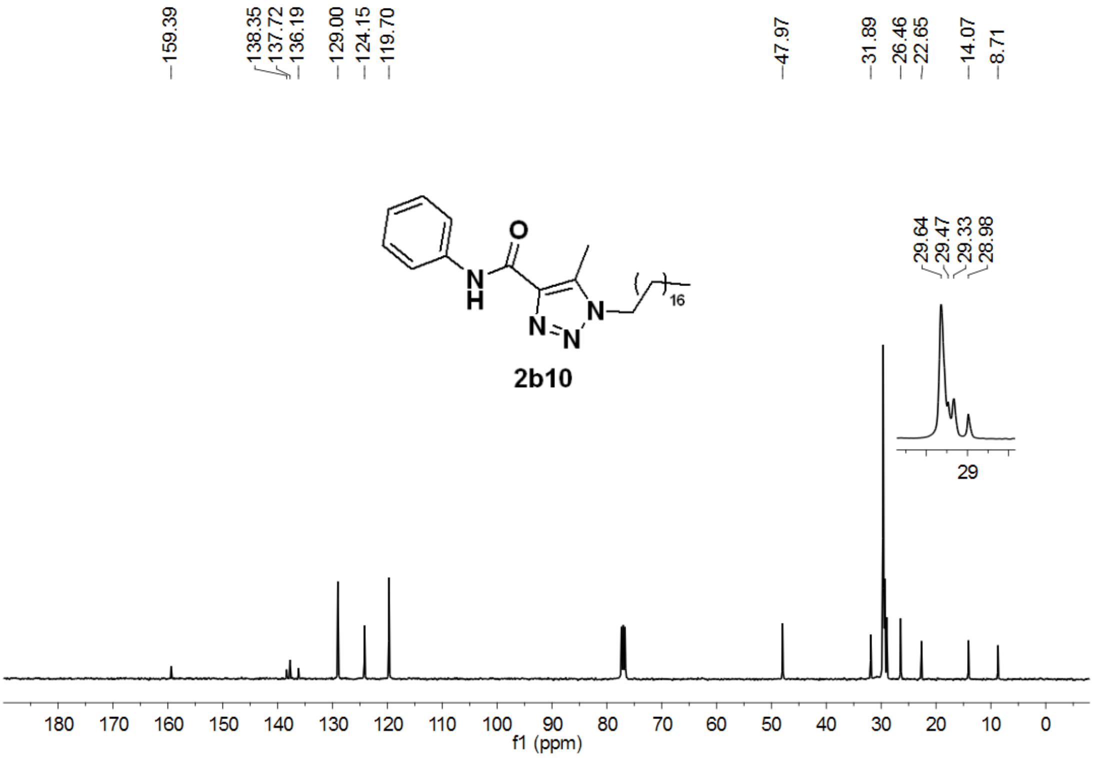

2.04
2.10
1.04
7.8
7.6
7.4
7.2
0.98
2.04
2.10
1.04
2.06
3.00
2.11
3.08
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

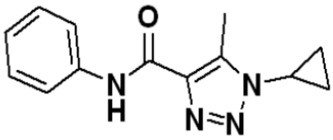

O
N
H
N
N
N

2b9

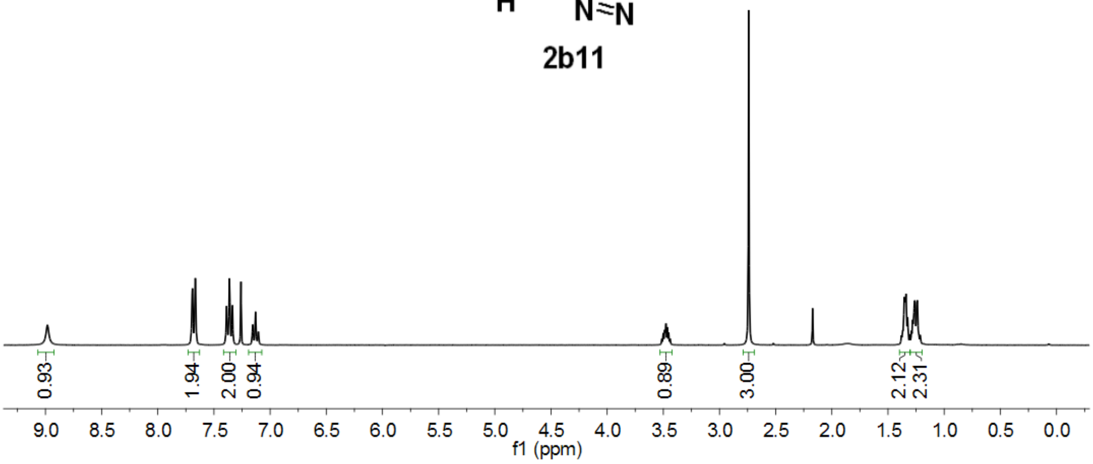

159.33
138.24
137.64
136.23
128.95
124.10
119.63
49.40
23.08
10.96
8.67
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

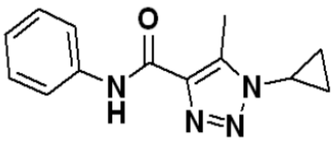

O
N
H
N
N
N
16
2b10
0.98
2.02
2.07
0.95
2.24
3.00
32.38
3.09
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.39
138.35
137.72
136.19
129.00
124.15
119.70
47.97
31.89
26.46
22.65
14.07
8.71
29.64
29.47
29.33
28.98
29
2b10
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

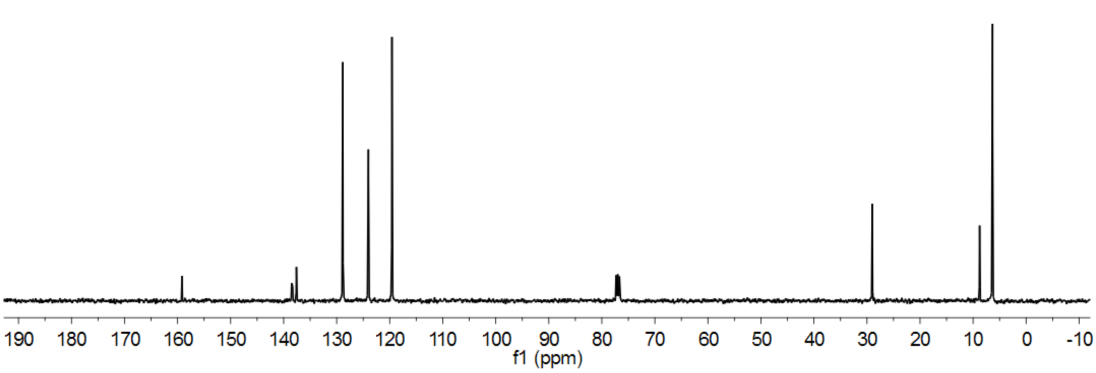

O
N
H
N
N=N

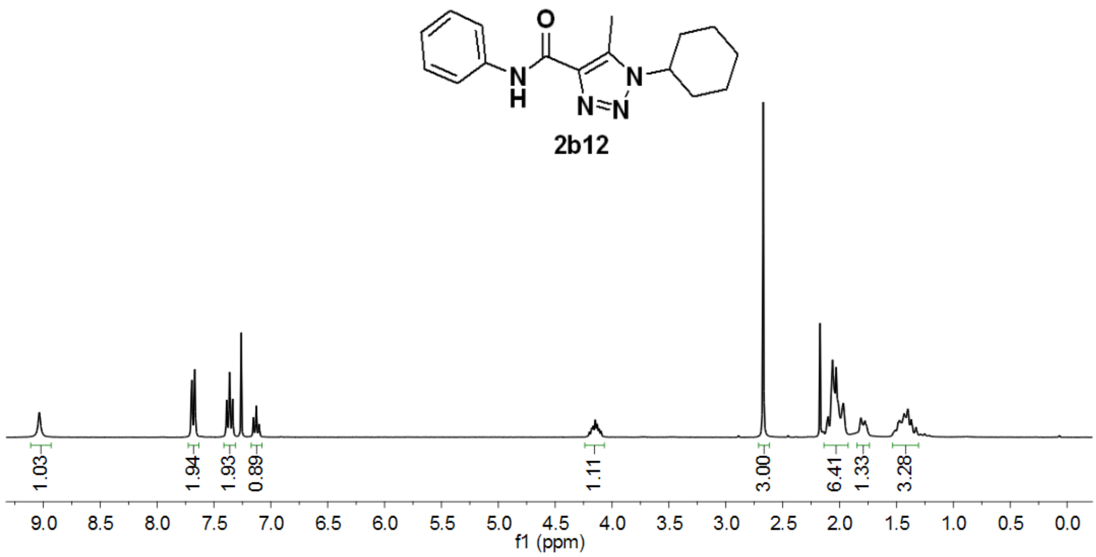
2b11

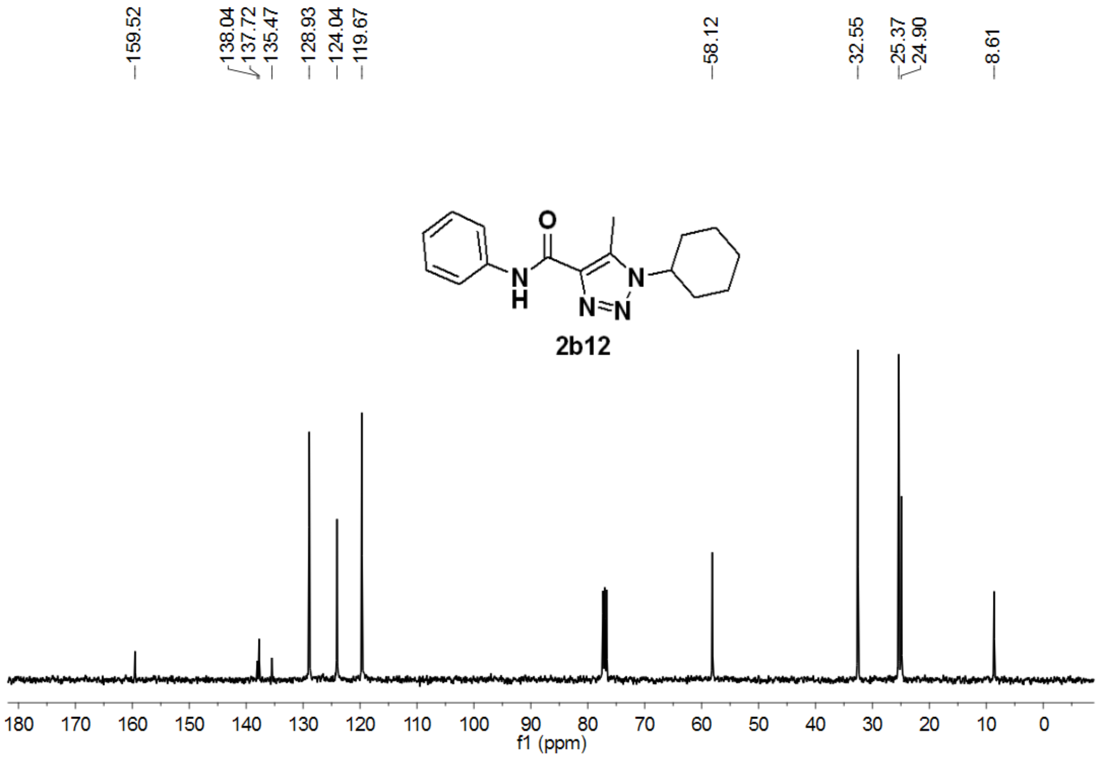

0.93
1.94
2.00
0.94
0.89
3.00
2.12
2.31
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

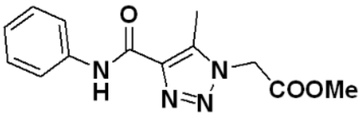

O
N
H
N
N=N

2b11

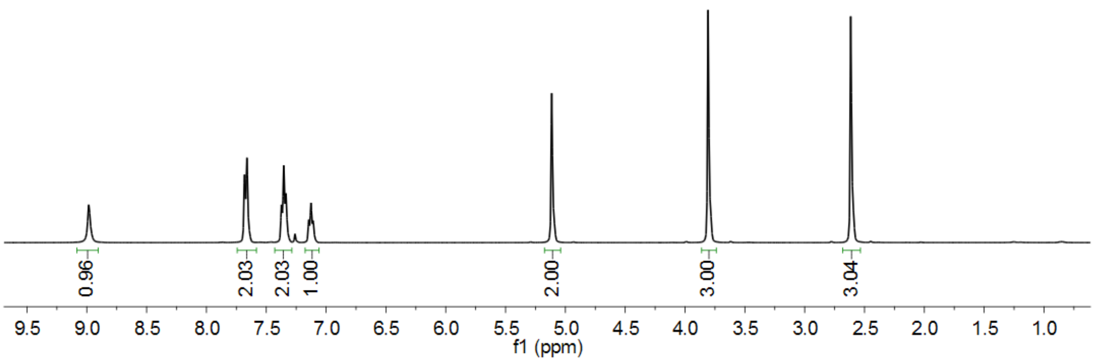

159.16
138.49
138.30
137.59
128.85
124.02
119.58
29.01
8.76
6.33
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

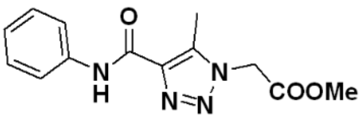

O
N
H
N
N=N
2b12
1.03
1.94
1.93
0.89
1.11
3.00
6.41
1.33
3.28
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.52
138.04
137.72
135.47
128.93
124.04
119.67
58.12
32.55
25.37
24.90
8.61
O
N
H
N
N=N
2b12
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

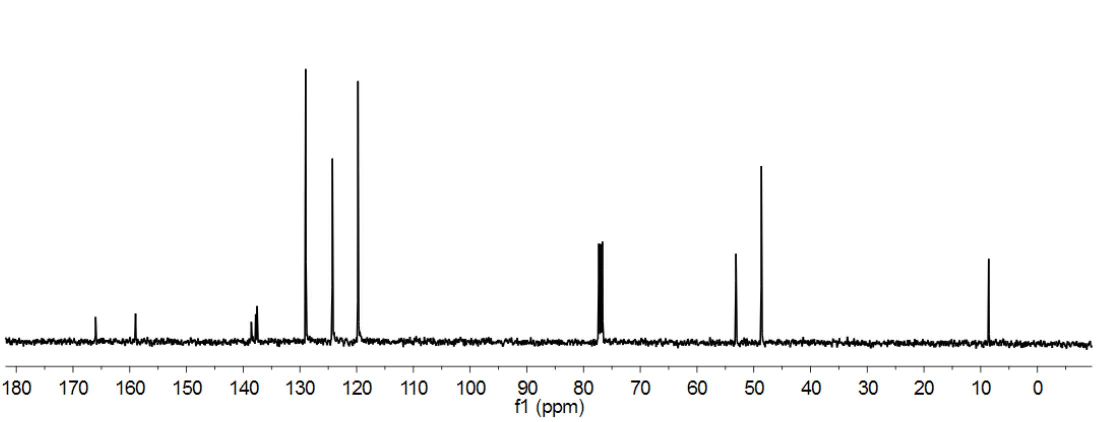


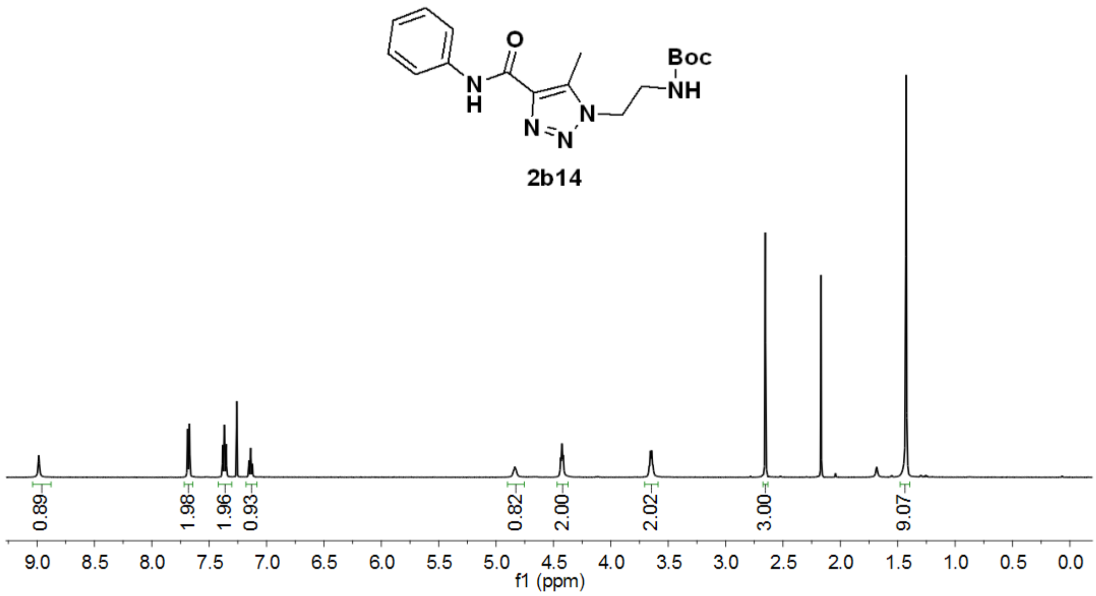
2b13

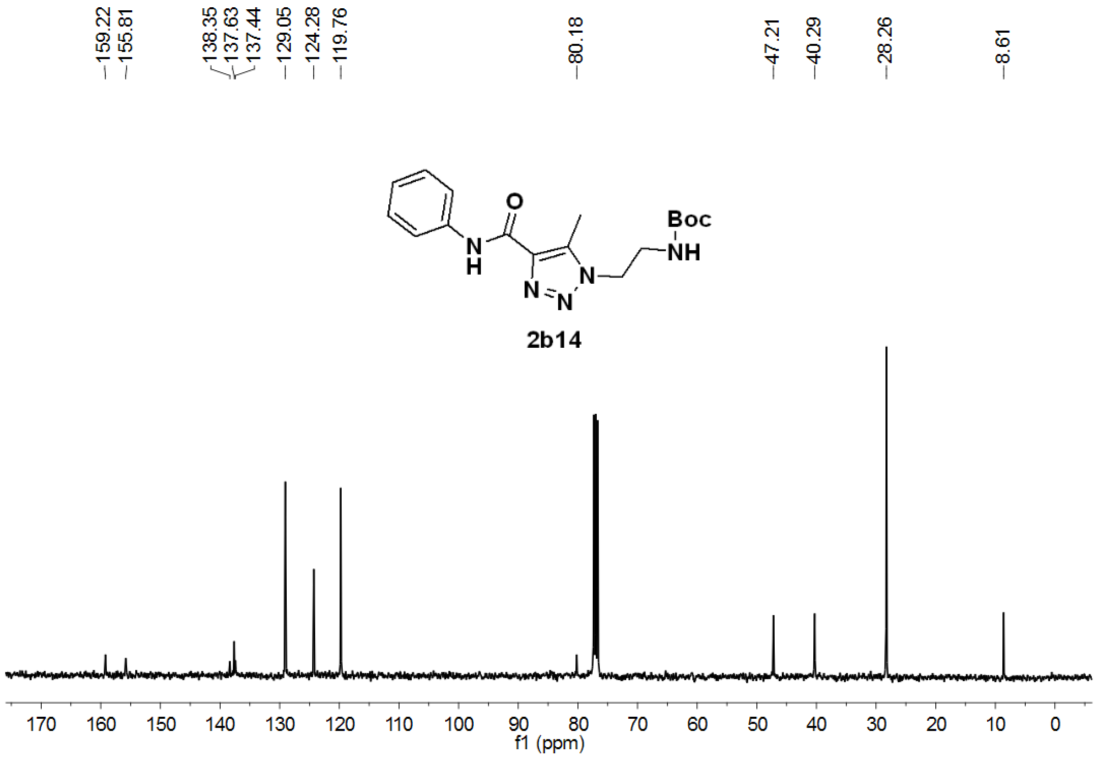


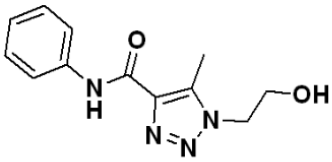


2b13

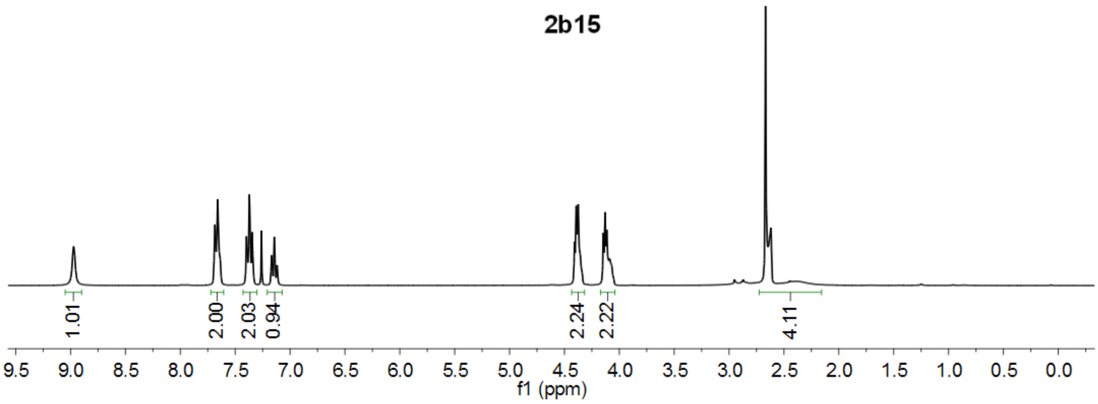



O
N
H
N
N
N
Boc
NH
2b14
0.89
1.98
1.96
0.93
0.82
2.00
2.02
3.00
9.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

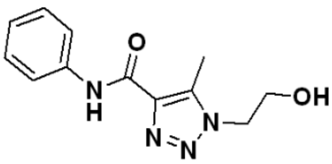

159.22
155.81
138.35
137.63
137.44
129.05
124.28
119.76
80.18
47.21
40.29
28.26
8.61
O
N
H
N
N
N
Boc
NH
2b14
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
N
H
N
N
N
OH

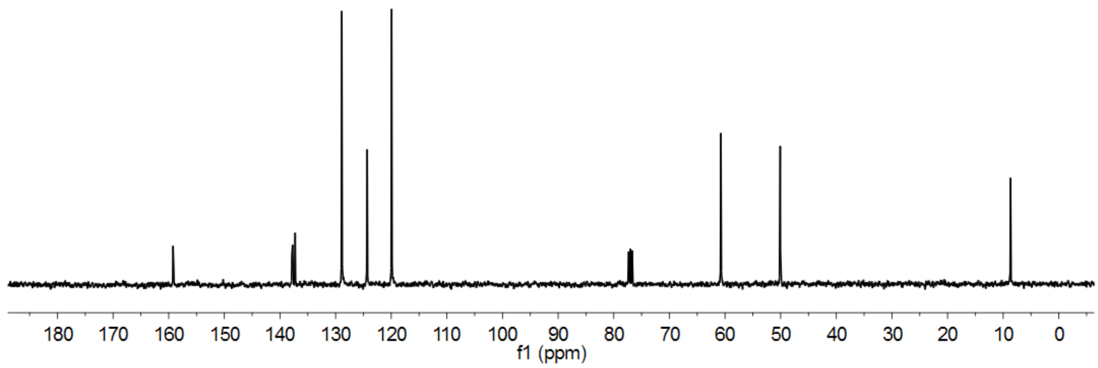
2b15

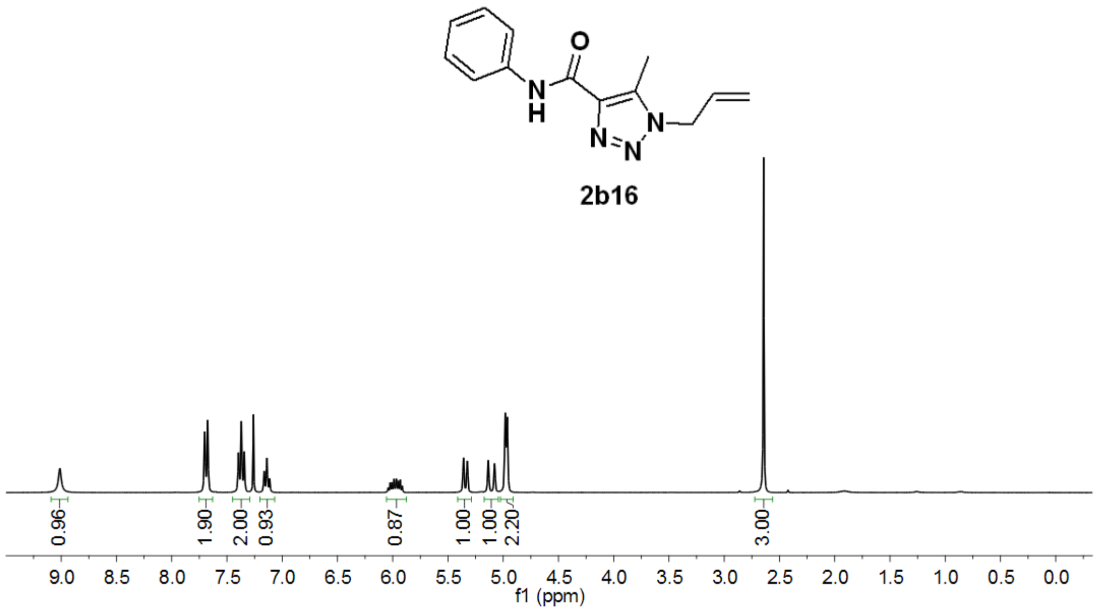

1.01
2.00
2.03
0.94
2.24
2.22
4.11
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

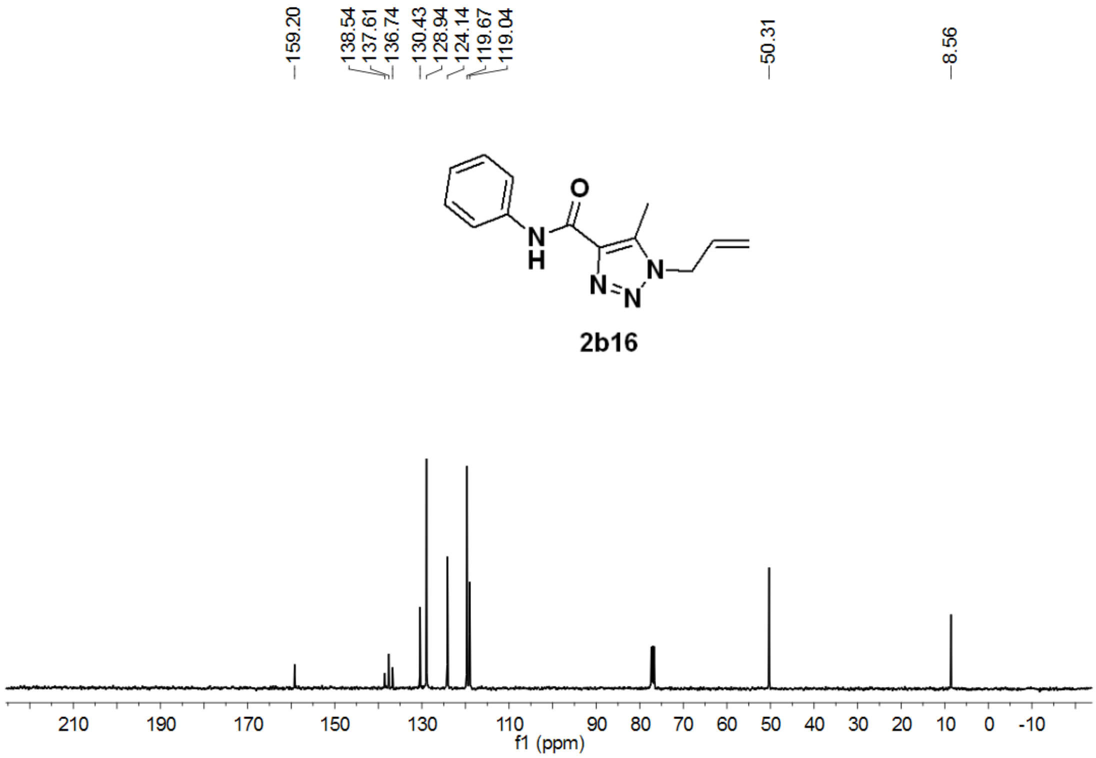

159.22
137.86
137.73
137.27
128.89
124.33
119.93
60.74
50.08
8.67

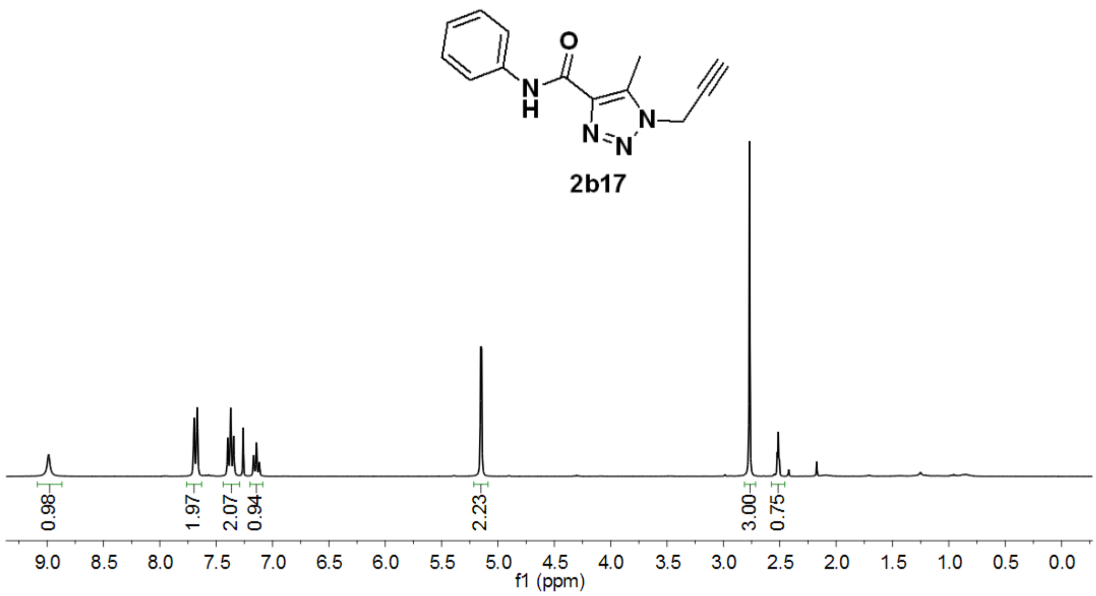

O
N
H
N
N
N
OH

2b15

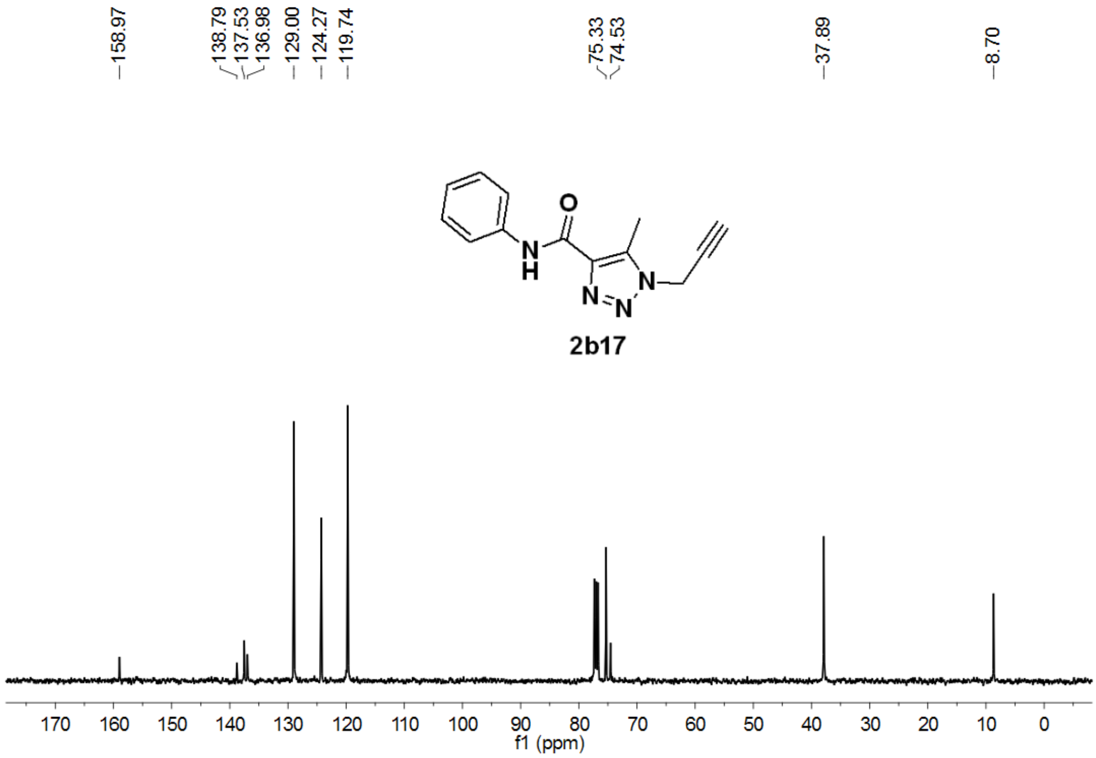

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


2b16
0.96
1.90
2.00
0.93
0.87
1.00
1.00
2.20
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

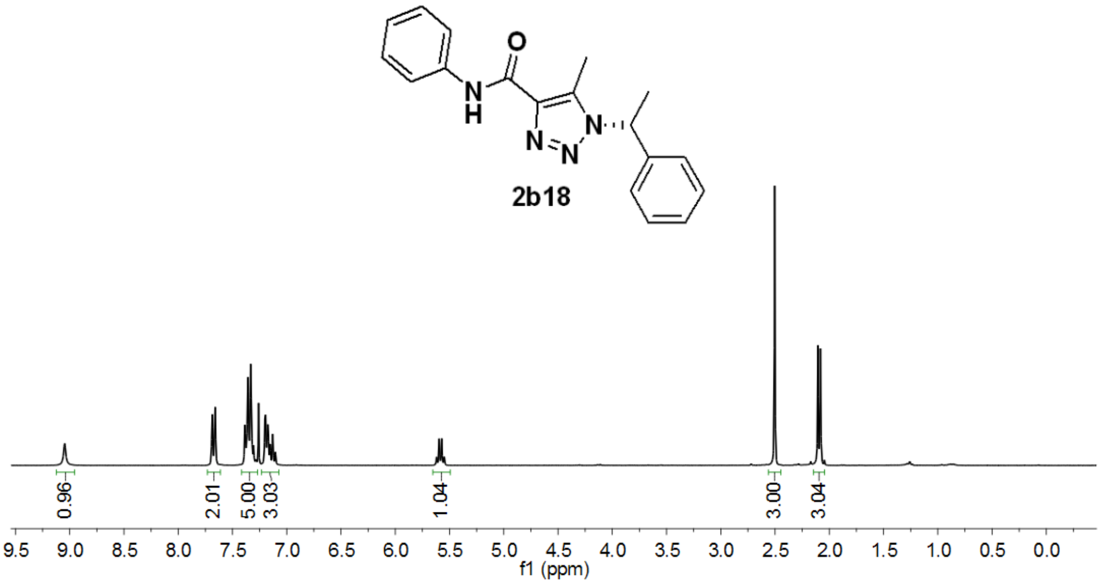

159.20
138.54
137.61
136.74
130.43
128.94
124.14
119.67
119.04
50.31
8.56
2b16
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

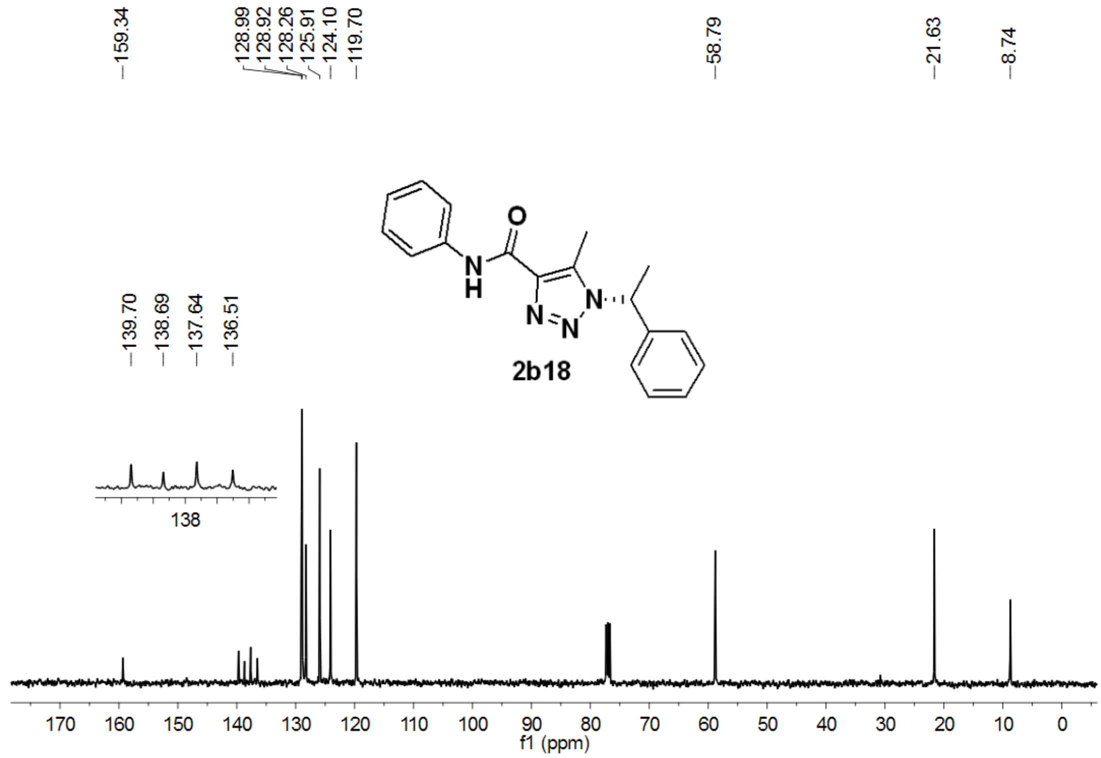


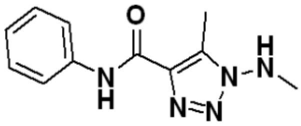
O
N
H
N
N
N
2b17
0.98
1.97
2.07
0.94
2.23
3.00
0.75
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



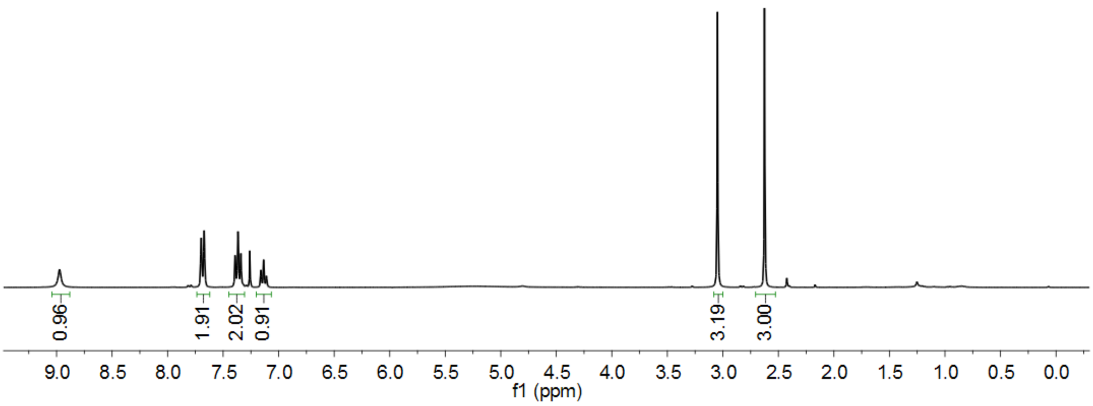
158.97
138.79
137.53
136.98
129.00
124.27
119.74
75.33
74.53
37.89
8.70
O
N
H
N
N
N
2b17
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
N
H
N
N
N
2b18
0.96
2.01
5.00
3.03
1.04
3.00
3.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

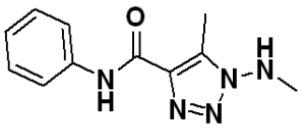

159.34
128.99
128.92
128.26
125.91
124.10
119.70
58.79
21.63
8.74
139.70
138.69
137.64
136.51
138
O
N
H
N
N
N
2b18
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



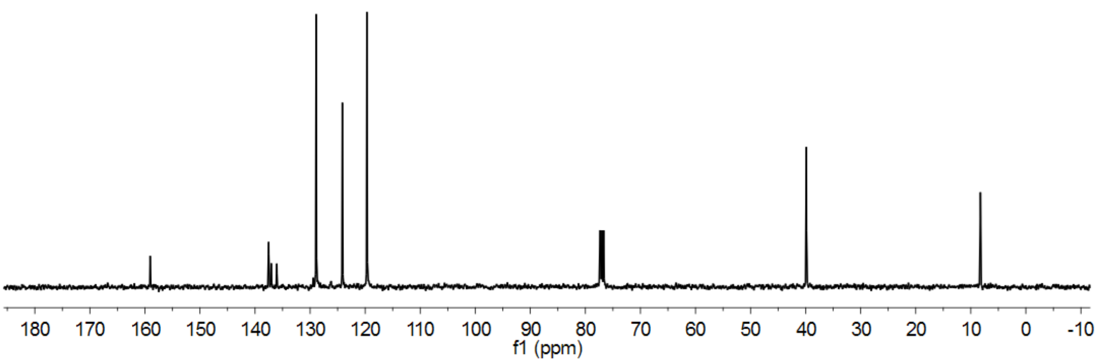
2b19

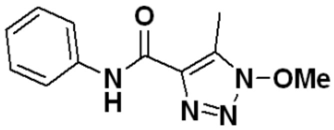

0.96
1.91
2.02
0.91
3.19
3.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.05
137.52
137.05
136.04
128.91
124.14
119.68
39.87
8.25

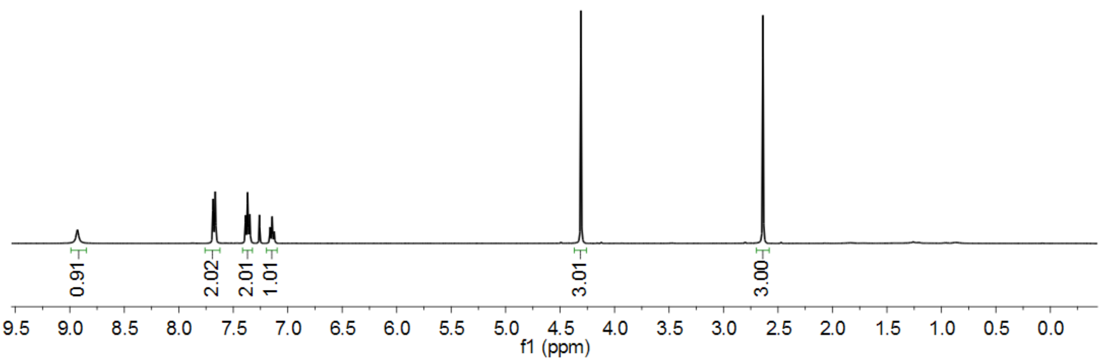


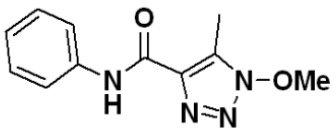
2b19


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

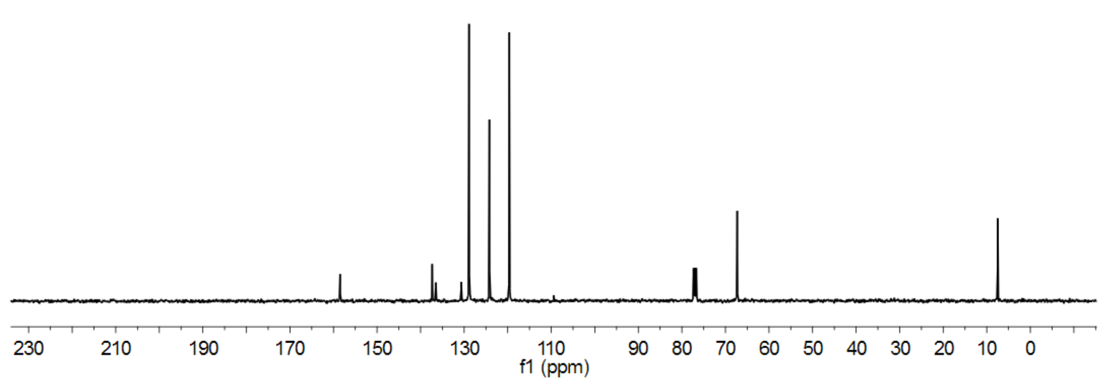

O
N
H
N–OMe
N=N

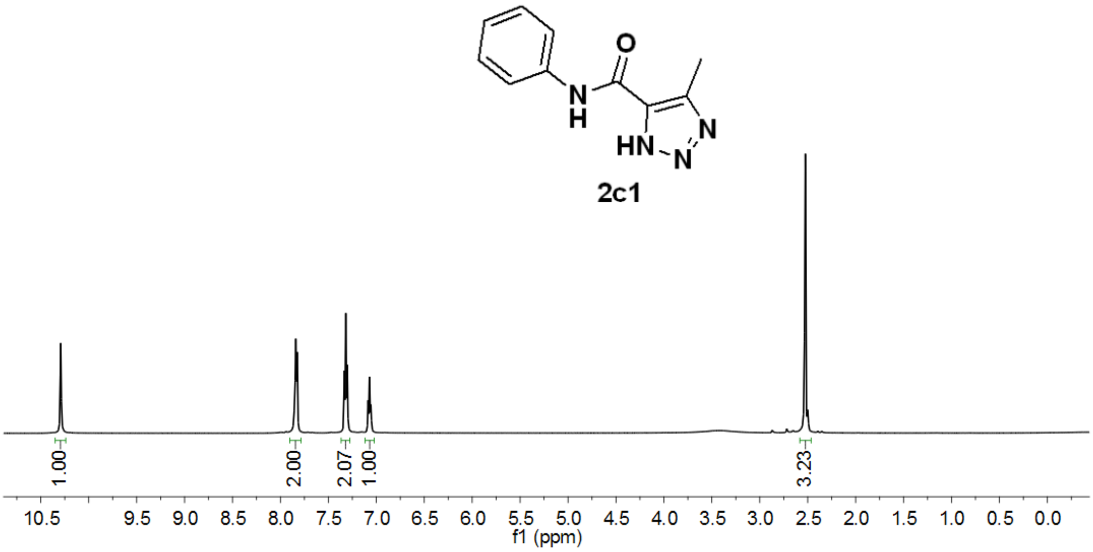
2b20

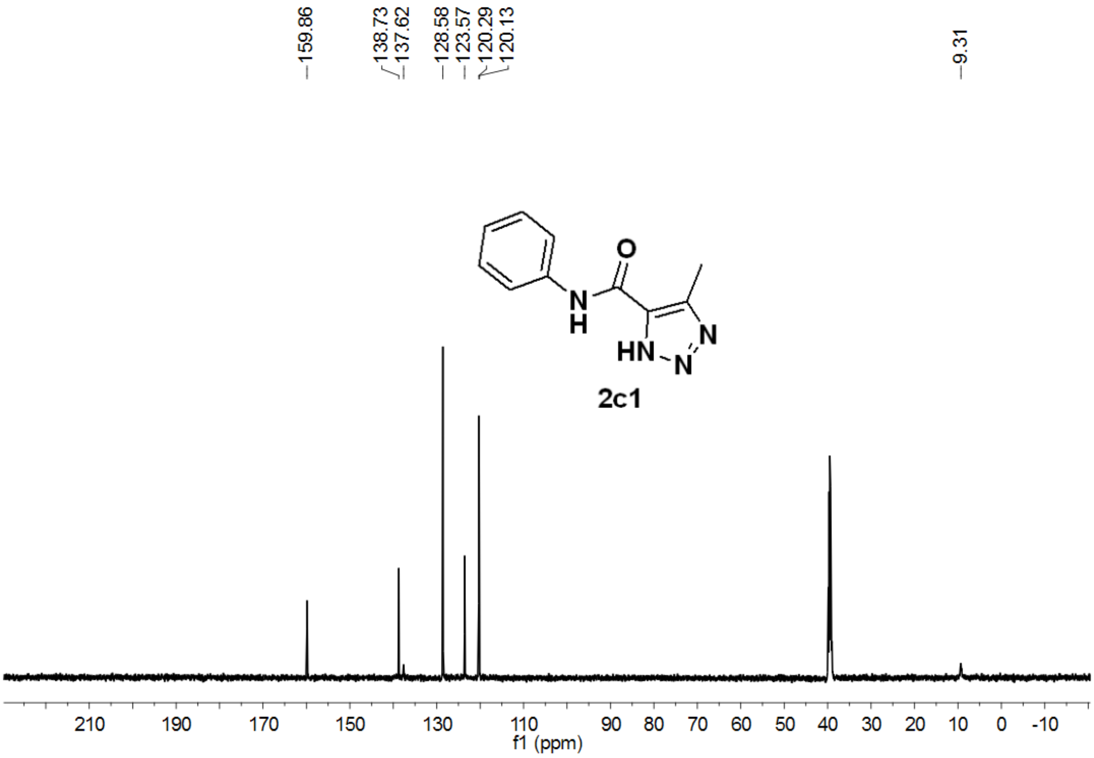

0.91
2.02
2.01
1.01
3.01
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

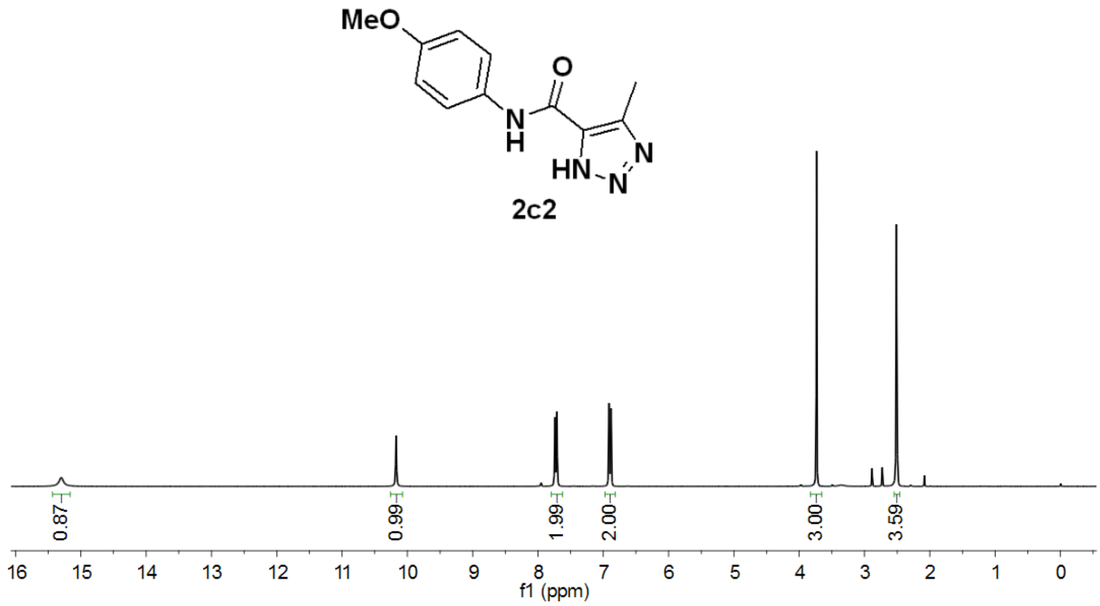

O
N
H
N–OMe
N=N

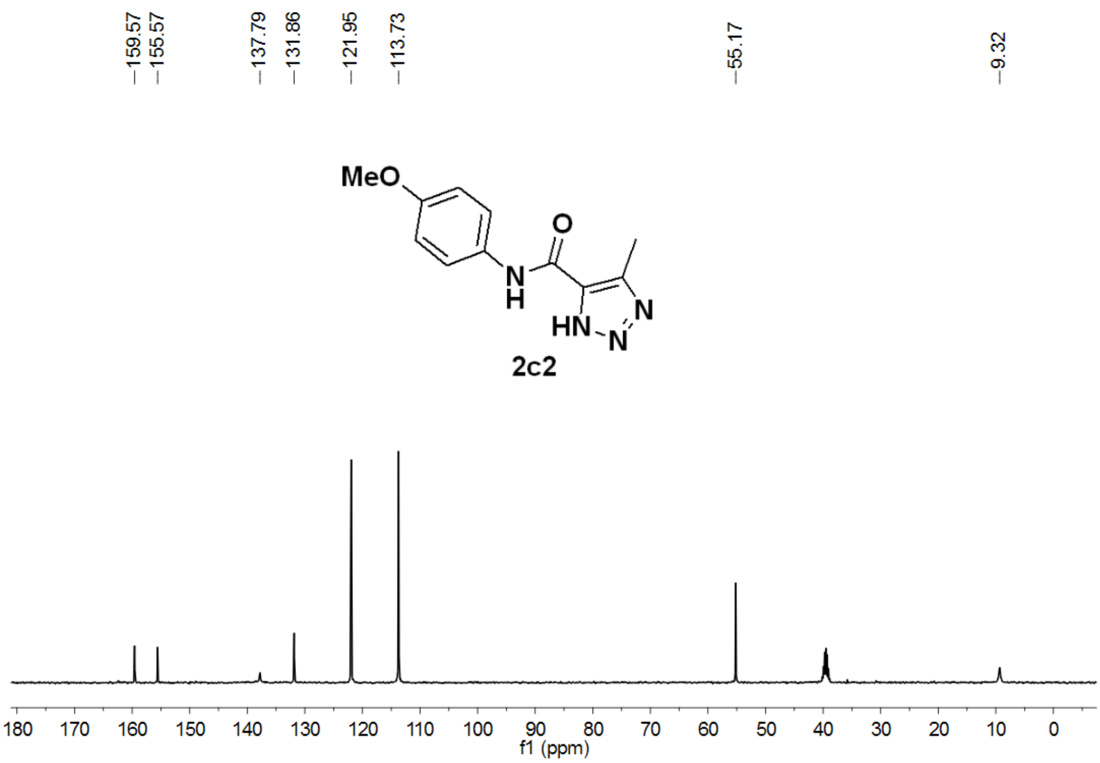
2b20

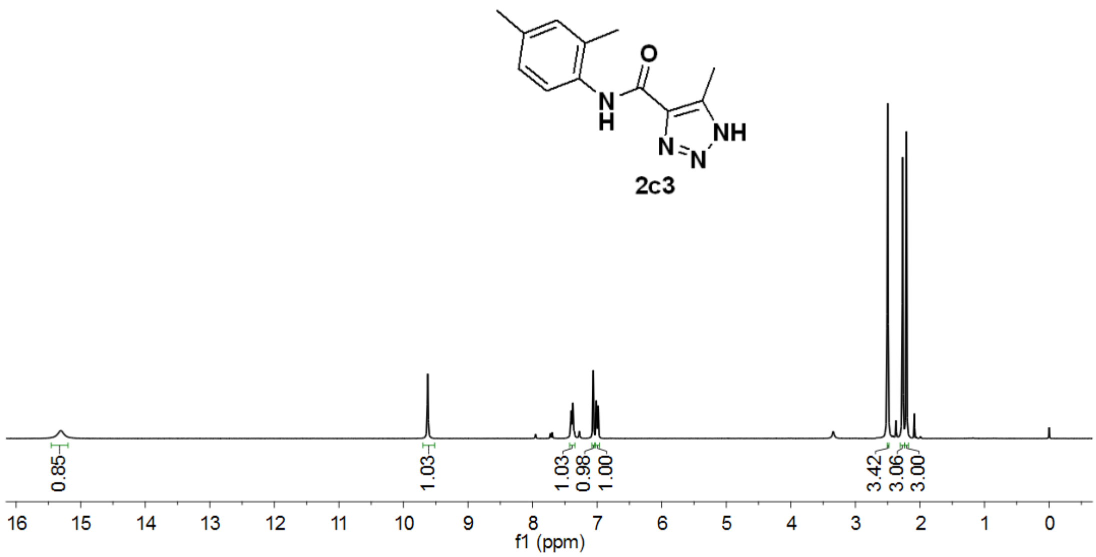

158.48
137.33
136.50
130.67
128.88
124.21
119.62
67.30
7.47
230 210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

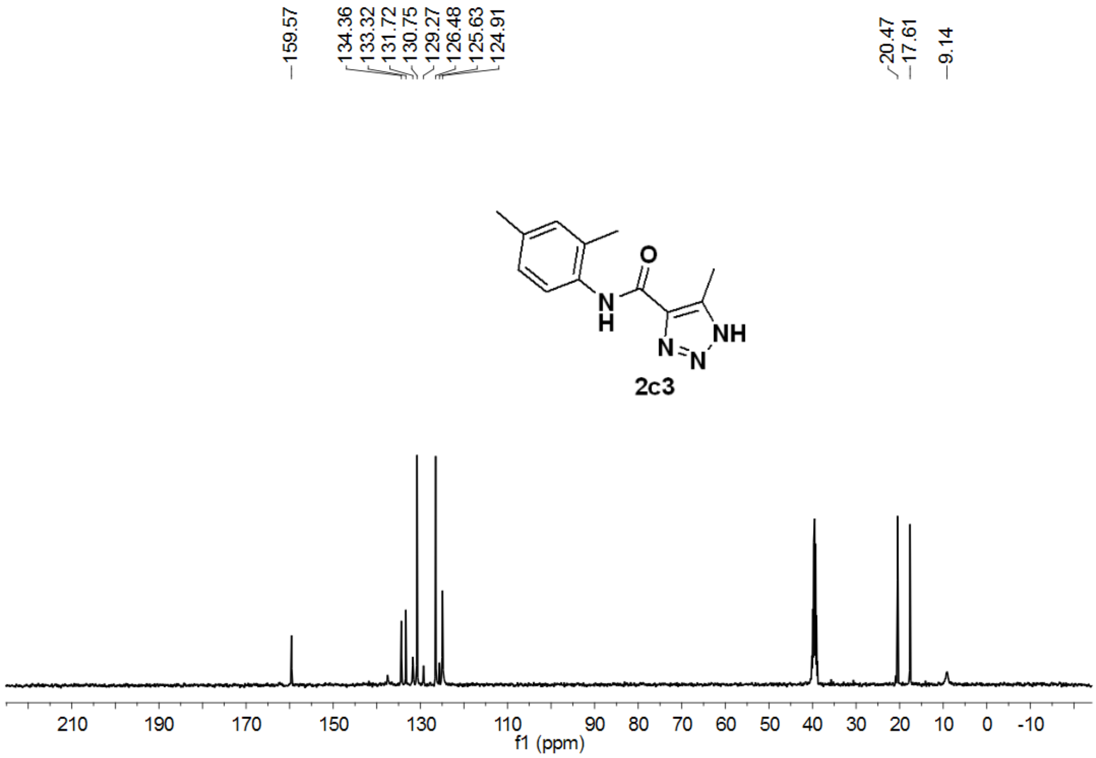

O
N
H
HN
N
N
2c1
1.00
2.00
2.07
1.00
3.23
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

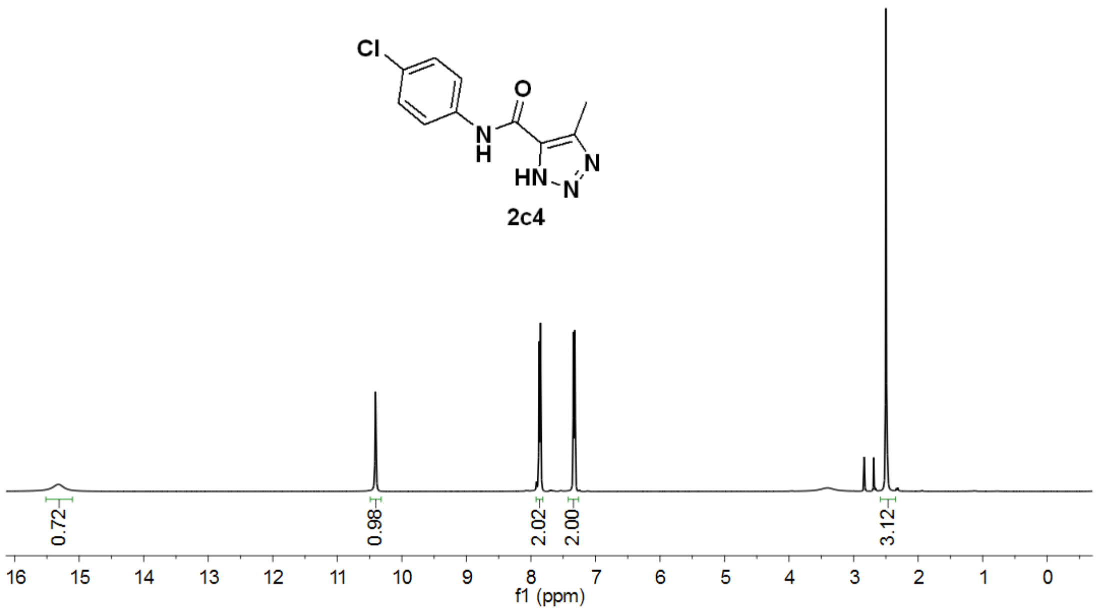

159.86
138.73
137.62
128.58
123.57
120.29
120.13
9.31
O
N
H
HN
N
N
2c1
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

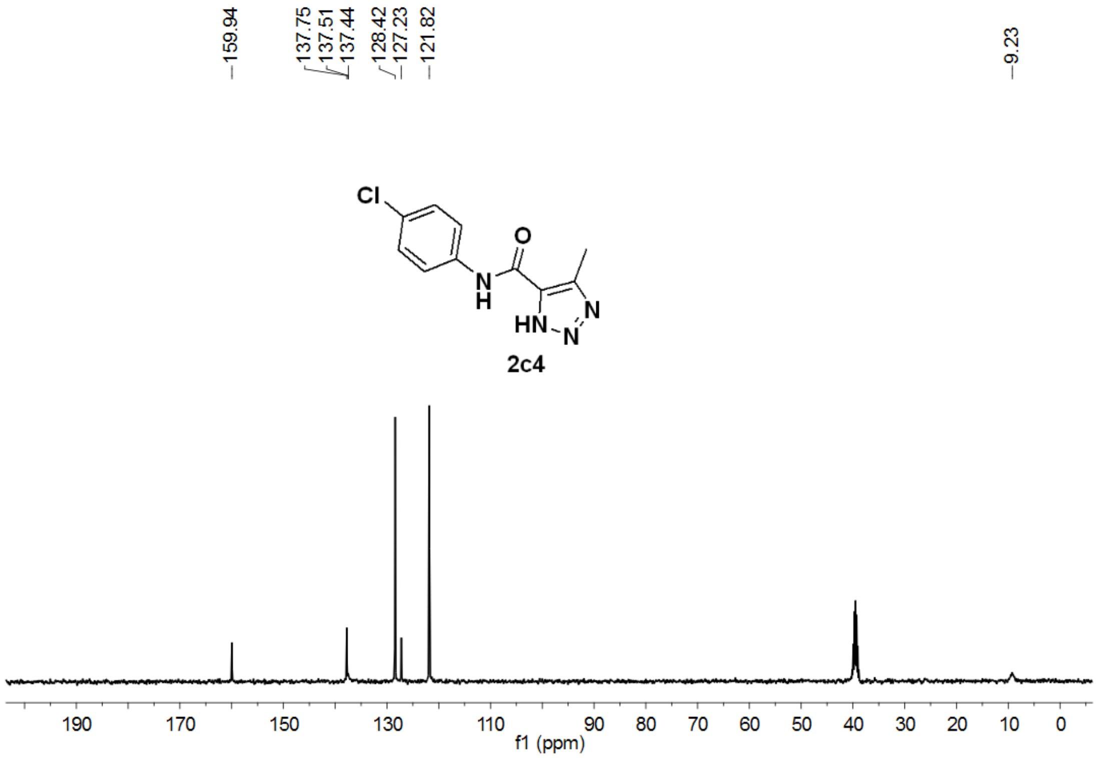

MeO
O
N
H
HN
N
N
2c2
0.87
0.99
1.99
2.00
3.00
3.59
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)

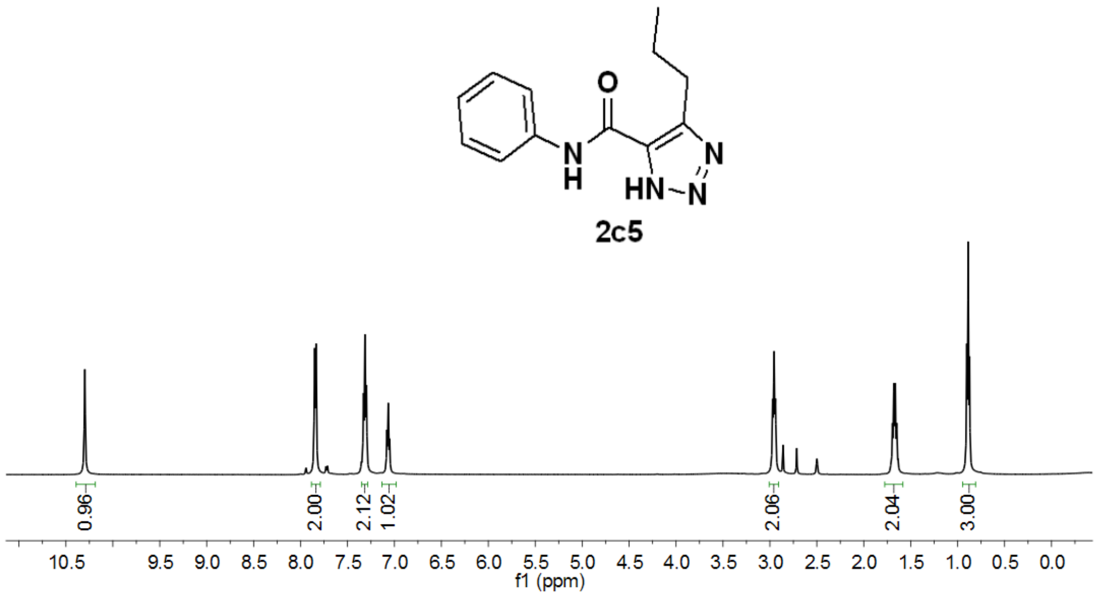

159.57
155.57
137.79
131.86
121.95
113.73
55.17
9.32
MeO
O
N
H
HN
N
N
2c2
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

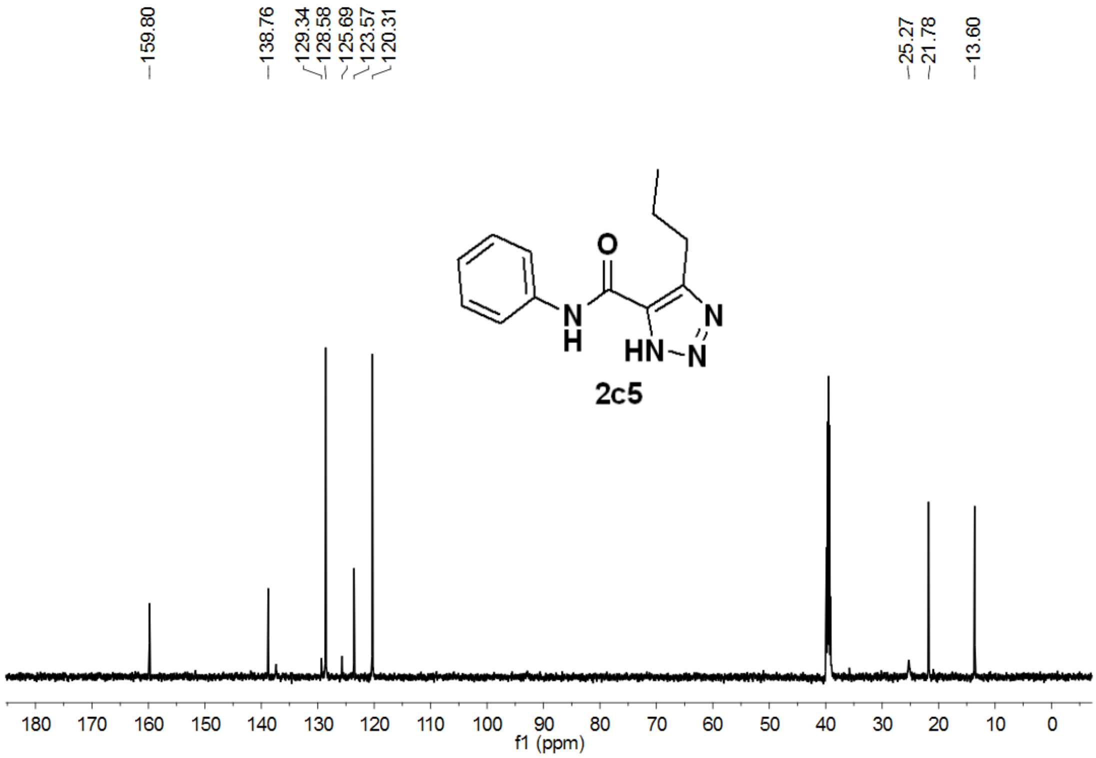

O
N
H
NH
N
N
2c3
0.85
1.03
1.03
0.98
1.00
3.42
3.06
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0
f1 (ppm)

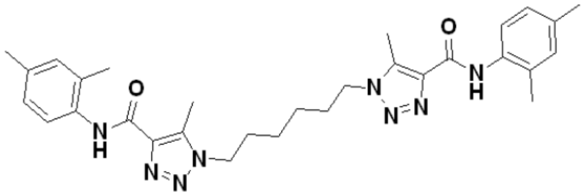

159.57
134.36
133.32
131.72
130.75
129.27
126.48
125.63
124.91
20.47
17.61
9.14
O
N
H
NH
N
N
2c3
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Cl
O
N
H
HN
N
N
2c4
0.72
0.98
2.02
2.00
3.12
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

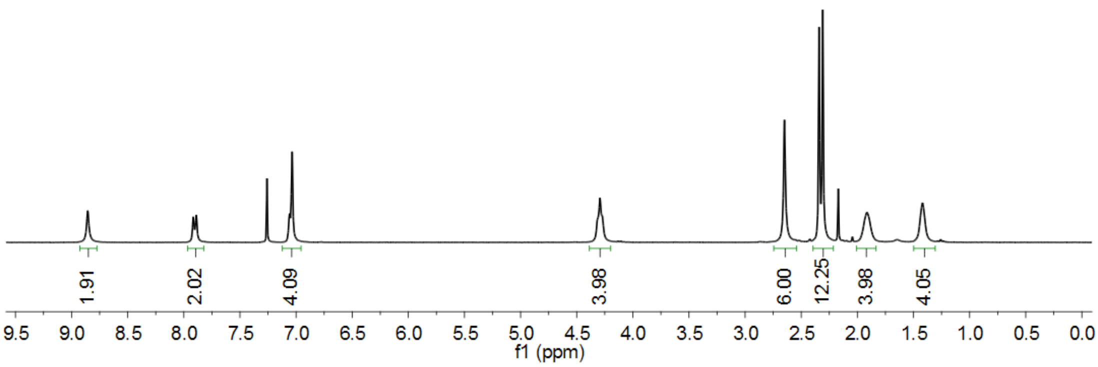

159.94
137.75
137.51
137.44
128.42
127.23
121.82
9.23
Cl
O
N
H
HN
N
N
2c4
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

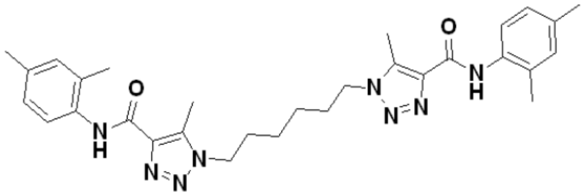

2c5
0.96
2.00
2.12
1.02
2.06
2.04
3.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.80
138.76
129.34
128.58
125.69
123.57
120.31
25.27
21.78
13.60
2c5
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

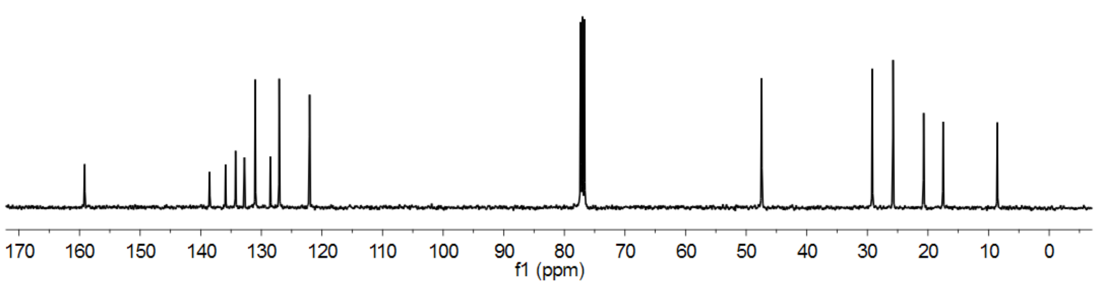


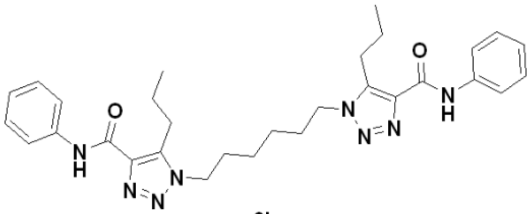
3a


1.91
2.02
4.09
3.98
6.00
12.25
3.98
4.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

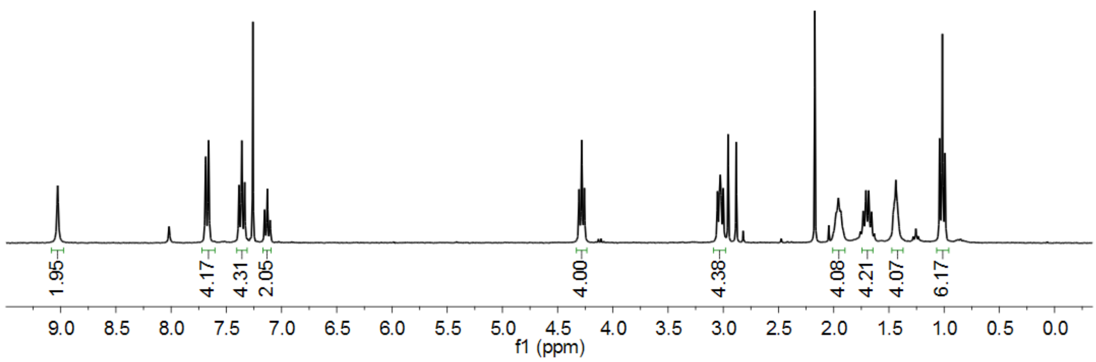


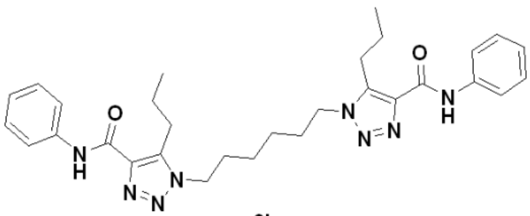
3a


159.15
138.53
135.90
134.25
132.79
131.00
128.51
127.05
122.04
47.45
29.19
25.76
20.71
17.50
8.55
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

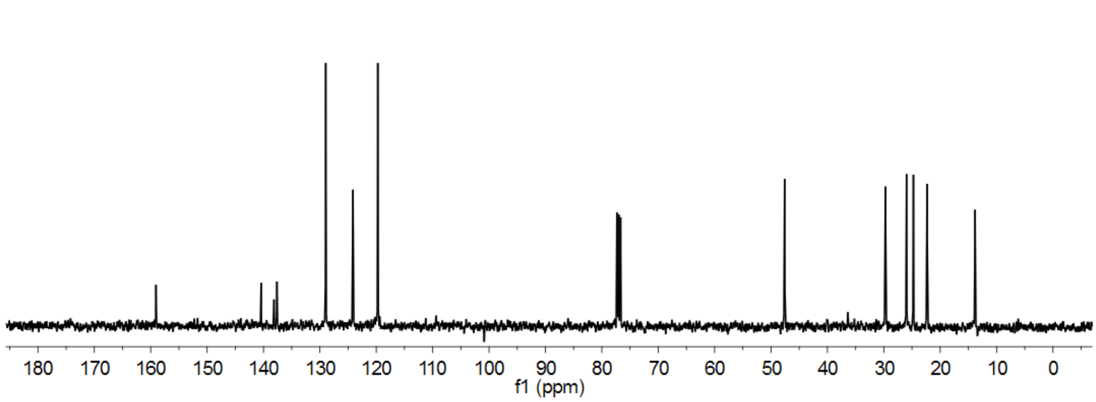

O
N
H
N
N
N
N
N
N
O
N
H

3b


1.95
4.17
4.31
2.05
4.00
4.38
4.08
4.21
4.07
6.17
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


O
N
H
N
N
N
N
N
N
O
N
H

3b


159.06
140.41
138.14
137.62
128.96
124.14
119.72
47.58
29.71
25.96
24.74
22.32
13.82
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)