Supporting Information

2-Pyrrolecarbaldiminato-Cu(II) complexes catalyzed three-component 1,3-dipolar cycloaddition for 1,4-disubstituted 1,2,3-triazoles synthesis in water at room temperature

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Materials and Methods

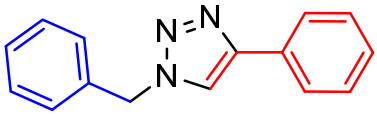
Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. Column chromatography was performed with silica gel (200-300 mesh) purchased from Qingdao Haiyang Chemical Co. Ltd. Thin-layer chromatography was carried out with Merck silica gel GF_{254} plates and visualized by exposure to UV light (254 nm). All 1,4-disubstituted 1,2,3-triazoles are characterized by ^{1}H NMR and ^{13}C NMR, which were compared with the previously reported data. ^{1}H NMR spectra and ^{13}C NMR spectra were recorded at room temperature on a Varian Inova-400 instrument at 400 MHz and 100 MHz, respectively.

General procedure for the synthesis of 1,2,3-triazoles 7a-7v: A 25 mL Schlenk tube was charged with Cu(II)-complex **3** (0.005 mmol), benzyl halides (0.5 mmol), NaN_3 (0.6 mmol), alkynes (0.6 mmol) and water (1 mL). The mixture was stirred at room temperature and monitored by TLC until the benzyl halide being consumed. The reaction mixture was then extracted with ethyl acetate (3×10 mL). The combined organic phases was washed with water and brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo. The residue was purified by flash column chromatograph on silica gel (ethyl acetate/petroleum ether as the eluent) to provide the target products.

General procedure for the synthesis of di-/tri-triazoles 7w and 7x: A 25 mL Schlenk tube was charged with Cu(II)-complex **3** (0.0025 mmol for dihalides and 0.00375 mmol for trihalides), benzyl halides (0.125 mmol), NaN_3 (0.3 mmol for dihalides and 0.45 mmol for trihalides), phenylacetylene (0.6 mmol for dihalides and 0.9 mmol for trihalides) and water (1 mL). The mixture was stirred at 60 °C and monitored by TLC until the benzyl halide being consumed. The reaction mixture was then extracted with ethyl acetate (3×10 mL). The combined organic phases was washed with water and brine, dried over anhydrous Na_2SO_4, and concentrated in vacuo. The residue was purified by flash column chromatograph on silica gel (ethyl acetate/petroleum ether as the eluent) to provide the target products.

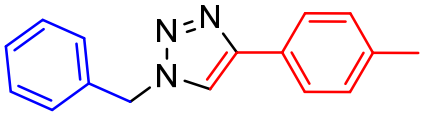
Characterization data for all 1,2,3-trizoles products

1-Benzyl-4-phenyl-1*H*-1,2,3-triazole (7a)[1]



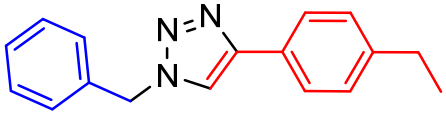
97% yield for benzyl bromide and 90 % for benzyl chloride. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81-7.79 (m, 2H), 7.67 (s, 1H), 7.36-7.42 (m, 5H), 7.29-7.33 (m, 3H), 5.58 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.2, 134.7, 130.5, 129.1, 128.8, 128.8, 128.1, 128.0, 125.7, 119.5, 54.2.

1-Benzyl-4-(p-tolyl)-1*H*-1,2,3-triazole (7b)[2]



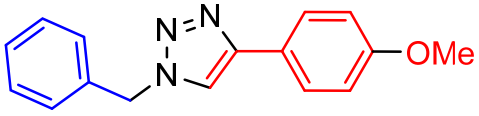
97% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (d, J = 8.4 Hz, 2H), 7.62 (s, 1H), 7.36-7.42 (m, 3H), 7.30-7.32 (m, 2H), 7.20 (d, J = 7.6 Hz, 2H), 5.57 (s, 2H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 138.0, 134.7, 129.5, 129.1, 128.7, 128.0, 127.7, 125.6, 119.2, 54.2, 21.3.

1-Benzyl-4-(4-ethylphenyl)-1*H*-1,2,3-triazole (7c) [3]



91% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, J = 8.4 Hz, 2H), 7.62 (s, 1H), 7.36-7.41 (m, 3H), 7.29-7.31 (m, 2H), 7.23 (d, J = 8.4 Hz, 2H), 5.57 (s, 2H), 2.66 (q, J = 7.6 Hz, 2H), 1.24 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 144.4, 134.9, 129.1, 128.7, 128.3, 128.1, 128.0, 125.7, 119.3, 54.2, 28.7, 15.6.

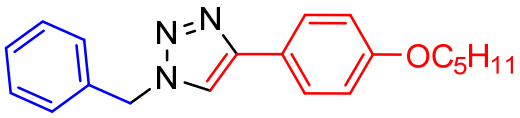
1-Benzyl-4-(4-methoxyphenyl)-1*H*-1,2,3-triazole (7d) [4]



91% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 (d, J = 8.8 Hz, 2H), 7.57 (s, 1H), 7.36-7.41 (m, 3H), 7.29-7.32 (m, 2H), 6.93 (d, J = 8.8 Hz, 2H), 5.56 (s, 2H), 3.83 (s,

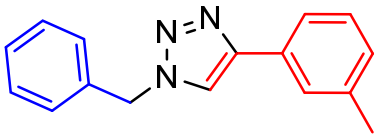
3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.7, 148.1, 134.9, 129.2, 128.8, 128.1, 127.1, 123.3, 118.8, 114.3, 55.4, 54.3.

1-Benzyl-4-(4-(pentyloxy)phenyl)-1*H*-1,2,3-triazole (7e) [4]



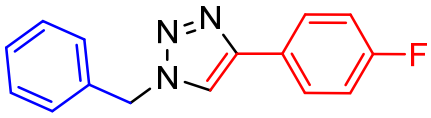
87% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70 (d, *J* = 8.8 Hz, 2H), 7.56 (s, 1H), 7.36-7.41 (m, 3H), 7.29-7.32 (m, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 5.56 (s, 2H), 3.97 (t, *J* = 6.4 Hz, 2H), 1.76-1.83 (m, 2H), 1.35- 1.47 (m, 4H), 0.93 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.2, 148.2, 134.8, 129.1, 128.7, 128.0, 126.9, 123.0, 118.6, 114.8, 68.0, 54.2, 28.9, 28.2, 22.5, 14.0.

1-Benzyl-4-(m-tolyl)-1*H*-1,2,3-triazole (7f) [5]



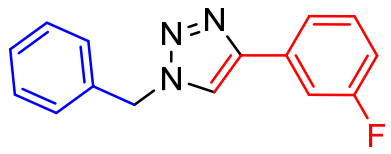
91% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (s, 1H), 7.64 (s, 1H), 7.55-7.58 (m, 1H), 7.36-7.42 (m, 3H), 7.26-7.32 (m, 3H), 7.11-7.14 (m, 1H), 5.58 (s, 2H), 2.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 138.5, 134.7, 130.4, 129.1, 128.9, 128.7, 128.7, 128.0, 126.3, 122.8, 119.5, 54.2, 21.4.

1-Benzyl-4-(4-fluorophenyl)-1*H*-1,2,3-triazole (7g) [6]



97% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74-7.79 (m, 2H), 7.61 (s, 1H), 7.37-7.42 (m, 3H), 7.30-7.32 (m, 2H), 7.06-7.12 (m, 2H), 5.57 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.8, 161.4, 147.3, 134.6, 129.2, 128.8, 128.0, 127.5, 127.4, 126.8, 126.8, 119.3, 115.9, 115.6, 54.2.

1-Benzyl-4-(3-fluorophenyl)-1*H*-1,2,3-triazole (7h) [7]

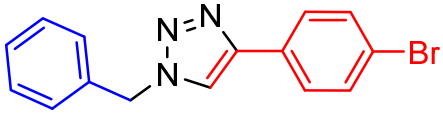


87% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66 (s, 1H), 7.51-7.57 (m, 2H), 7.34-7.43 (m, 4H), 7.30-7.33 (m, 2H), 6.98-7.03 (m, 1H), 5.58 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 164.3, 161.9, 147.2, 147.1, 134.5, 132.7, 132.6, 130.4, 130.3, 129.2, 128.9, 128.1, 121.3, 121.2, 119.9, 115.0, 114.8, 112.7, 112.5, 54.3.

1-Benzyl-4-(4-chlorophenyl)-1H-1,2,3-triazole (7i) [8]

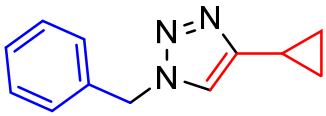
92% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, J = 8.4 Hz, 2H), 7.65 (s, 1H), 7.35-7.43 (m, 5H), 7.30-7.33 (m, 2H), 5.58 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.1, 134.5, 133.9, 129.2, 129.0, 129.0, 128.9, 128.1, 126.9, 119.6, 54.3.

1-Benzyl-4-(4-bromophenyl)-1*H*-1,2,3-triazole (7j) [9]



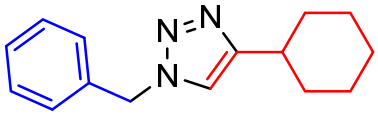
89% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67 (d, J = 8.8 Hz, 2H), 7.65 (s, 1H), 7.50-7.54 (m, 2H), 7.37-7.43 (m, 3H), 7.30-7.33 (m, 2H), 5.57 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.1, 134.5, 131.9, 129.5, 129.2, 128.8, 128.1, 127.2, 122.0, 119.6, 54.2.

1-Benzyl-4-cyclopropyl-1*H*-1,2,3-triazole (7k) [10]



60% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33-7.39 (m, 3H), 7.23-7.26 (m, 2H), 7.15 (s, 1H), 5.46 (s, 2H), 1.88-1.95 (m, 1H), 0.89-0.94 (m, 2H), 0.79-0.83 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.6, 135.0, 129.0, 128.6, 128.0, 120.0, 53.9, 7.7, 6.7.

1-Benzyl-4-cyclohexyl-1H-1,2,3-triazole (7l) [11]



77% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32-7.40 (m, 3H), 7.24-7.27 (m, 2H), 7.15 (s, 1H), 5.49 (s, 2H), 2.71-2.78 (m, 2H), 2.01-2.04 (m, 2H), 1.76-1.80 (m, 2H), 1.33-1.40 (m, 4H), 1.20-1.28 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.2, 135.0, 129.0, 128.6, 128.0, 119.2, 54.0, 35.3, 33.0, 26.1, 26.0.

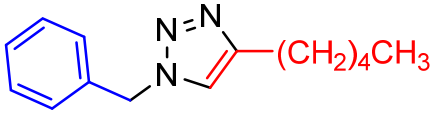
1-Benzyl-4-(cyclohex-1-en-1-yl)-1*H*-1,2,3-triazole (7m) [12]

75% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.38 (m, 4H), 7.24-7.29 (m, 2H), 6.49-6.51 (m, 1H), 5.50 (d, J = 5.2 Hz, 2H), 2.34-2.36 (m, 2H), 2.17-2.20 (m, 2H), 1.72-1.76 (m, 2H), 1.63-1.67 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.0, 135.0, 129.1, 128.7, 128.0, 127.3, 125.1, 118.3, 54.1, 26.4, 25.3, 22.5, 22.3.

2-(1-Benzyl-1H-1,2,3-triazol-4-yl)propan-2-ol (7n) [1]

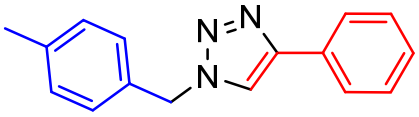
96% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36-7.41 (m, 3H), 7.35 (s, 1H), 7.27-7.29 (m, 2H), 5.50 (s, 2H), 2.62 (s, 1H), 1.61 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.1, 134.6, 129.1, 128.7, 128.1, 119.1, 68.5, 54.1, 30.4.

1-Benzyl-4-pentyl-1*H*-1,2,3-triazole (7o) [13]



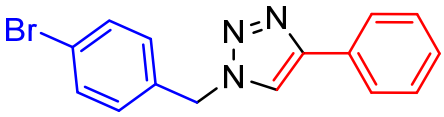
68% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33-7.39 (m, 3H), 7.24-7.26 (m, 2H), 7.19 (s, 1H), 5.49 (s, 2H), 2.68 (t, J = 8.0 Hz, 2H), 1.60-1.68 (m, 2H), 1.30-1.33 (m, 4H), 0.88 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.0, 135.0, 129.0, 128.6, 127.9, 120.5, 54.0, 31.5, 29.1, 25.7, 22.4, 14.0.

1-(4-Methylbenzyl)-4-phenyl-1*H*-1,2,3-triazole (7p) [2]



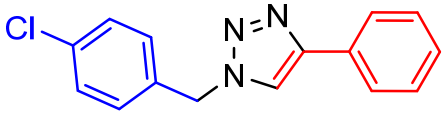
80% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78-7.80 (m, 2H), 7.63 (s, 1H), 7.37-7.41 (m, 2H), 7.28-7.33 (m, 1H), 7.18-7.23 (m, 4H), 5.53 (s, 2H), 2.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.7, 131.6, 129.8, 128.8, 128.1, 128.1, 125.7, 125.7, 125.6, 119.4, 54.0, 21.2.

1-(4-Bromobenzyl)-4-phenyl-1*H*-1,2,3-triazole (7q) [3]



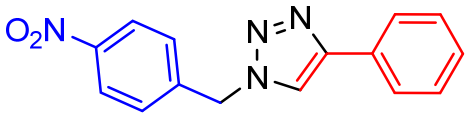
71% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79-7.81 (m, 2H), 7.66 (s, 1H), 7.50-7.54 (m, 2H), 7.39-7.43 (m, 2H), 7.30-7.35 (m, 1H), 7.19 (d, *J* = 8.4 Hz, 2H), 5.53 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.4, 133.7, 132.3, 130.3, 129.6, 128.8, 128.3, 125.7, 122.9, 119.5, 53.5.

1-(4-Chlorobenzyl)-4-phenyl-1*H*-1,2,3-triazole (7r) [14]



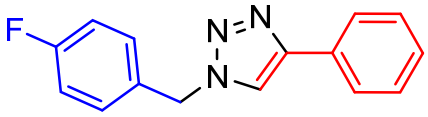
74% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 (d, *J* = 7.2 Hz, 2H), 7.67 (s, 1H), 7.31-7.43 (m, 5H), 7.26 (d, *J* = 7.2 Hz, 2H), 5.55 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 134.8, 133.2, 130.4, 129.3, 129.3, 128.8, 128.3, 125.7, 119.5, 53.4.

1-(4-Nitrobenzyl)-4-phenyl-1*H*-1,2,3-triazole (7s) [2]



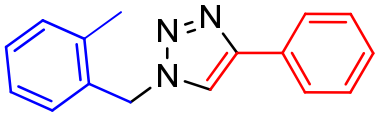
90% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.24 (d, *J* = 8.8 Hz, 2H), 7.82 (d, *J* = 7.2 Hz, 2H), 7.76 (s, 1H), 7.33-7.46 (m, 5H), 5.70 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.2, 141.9, 130.2, 129.0, 128.7, 128.6, 125.9, 124.4, 119.9, 53.3.

1-(4-Fluorobenzyl)-4-phenyl-1*H*-1,2,3-triazole (7t) [8]



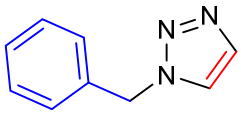
79% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79-7.82 (m, 2H), 7.66 (s, 1H), 7.39-7.43 (m, 2H), 7.29-7.35 (m, 3H), 7.06-7.11 (m, 2H), 5.55 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.1, 161.6, 148.3, 130.6, 130.5, 130.4, 130.0, 129.9, 128.8, 128.3, 125.7, 119.4, 116.2, 116.0, 53.5.

1-(2-Methylbenzyl)-4-phenyl-1H-1,2,3-triazole (7u) [15]



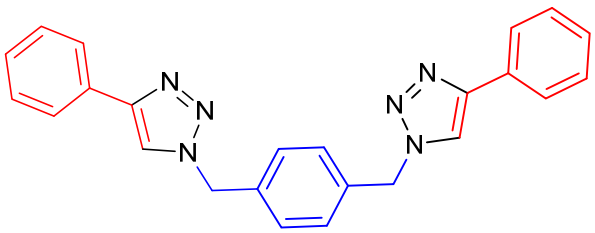
95% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.78-7.80 (m, 2H), 7.54 (s, 1H), 7.37-7.41 (m, 2H), 7.28-7.33 (m, 2H), 7.20-7.26 (m, 3H), 5.59 (s, 2H), 2.31 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.1, 137.1, 132.6, 131.2, 130.6, 129.5, 129.3, 128.9, 128.2, 126.8, 125.8, 119.4, 52.5, 19.1.

1-Benzyl-1*H*-1,2,3-triazole (7v) [16]



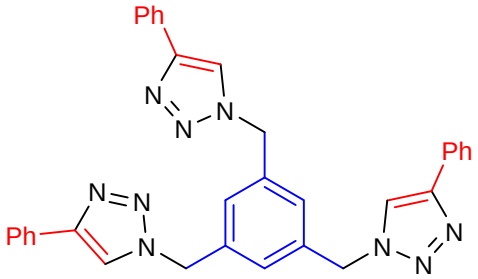
67% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 (s, 1H), 7.48 (s, 1H), 7.35-7.40 (m, 3H), 7.25-7.28 (m, 2H), 5.57 (s, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.6, 134.2, 129.1, 128.8, 128.0, 123.3, 54.0.

1,4-Bis((4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl)benzene (7w) [17]



55% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.62 (s, 2H), 7.83 (d, J = 7.2 Hz, 4H), 7.32-7.45 (m, 10H), 5.64 (s, 4H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 146.7, 136.0, 130.6, 128.9, 128.4, 127.9, 125.1, 121.6, 52.6.

1,3,5-Tris((4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl)benzene (7x) [18]



75% yield. ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.61 (s, 3H), 7.81 (d, J = 7.6 Hz, 6H), 7.42 (t, J = 6.8 Hz, 6H), 7.31-7.34 (m, 6H), 5.66 (s, 6H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 146.7, 137.3, 130.6, 128.9, 127.9, 127.2, 125.2, 121.6, 52.6.

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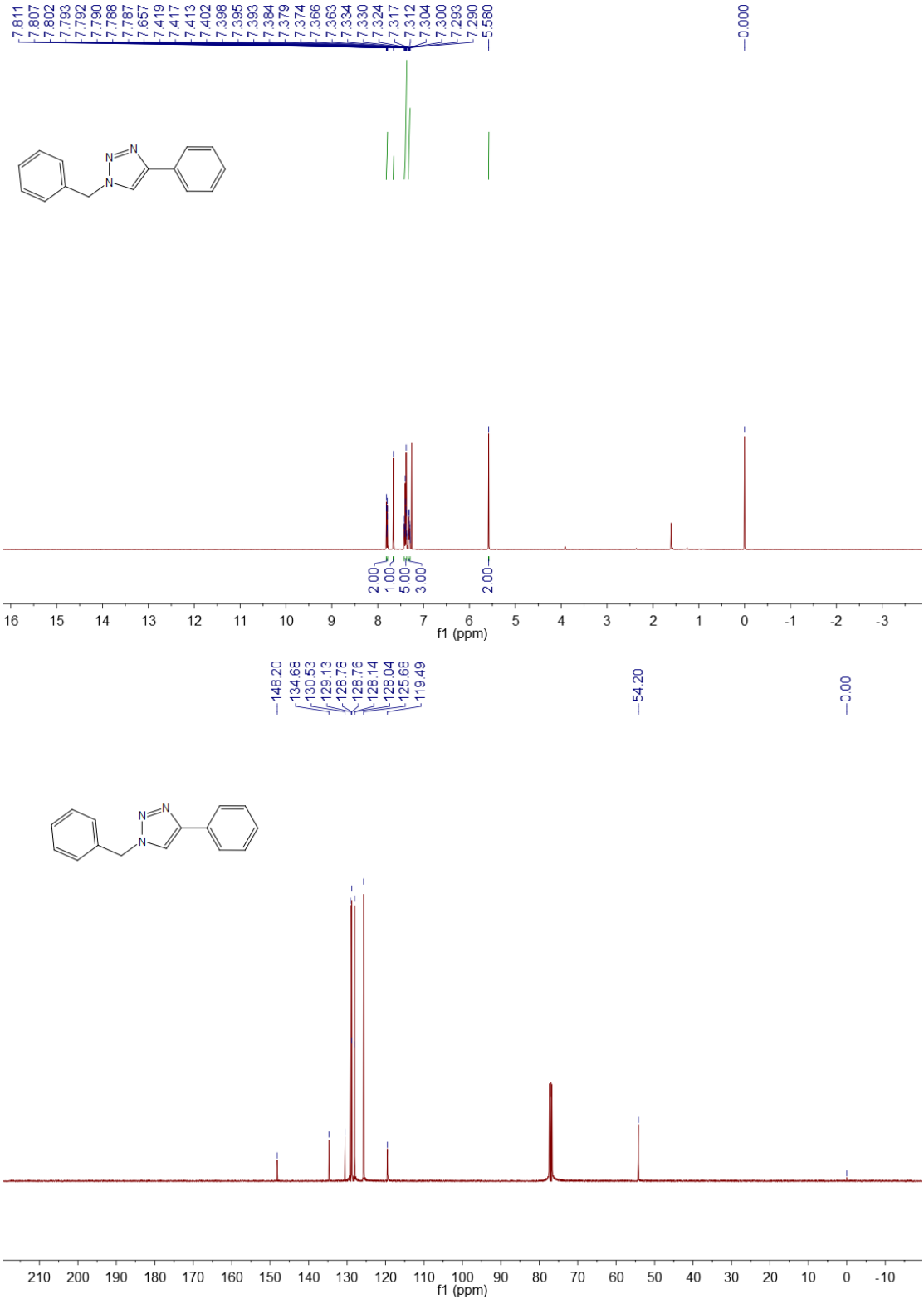
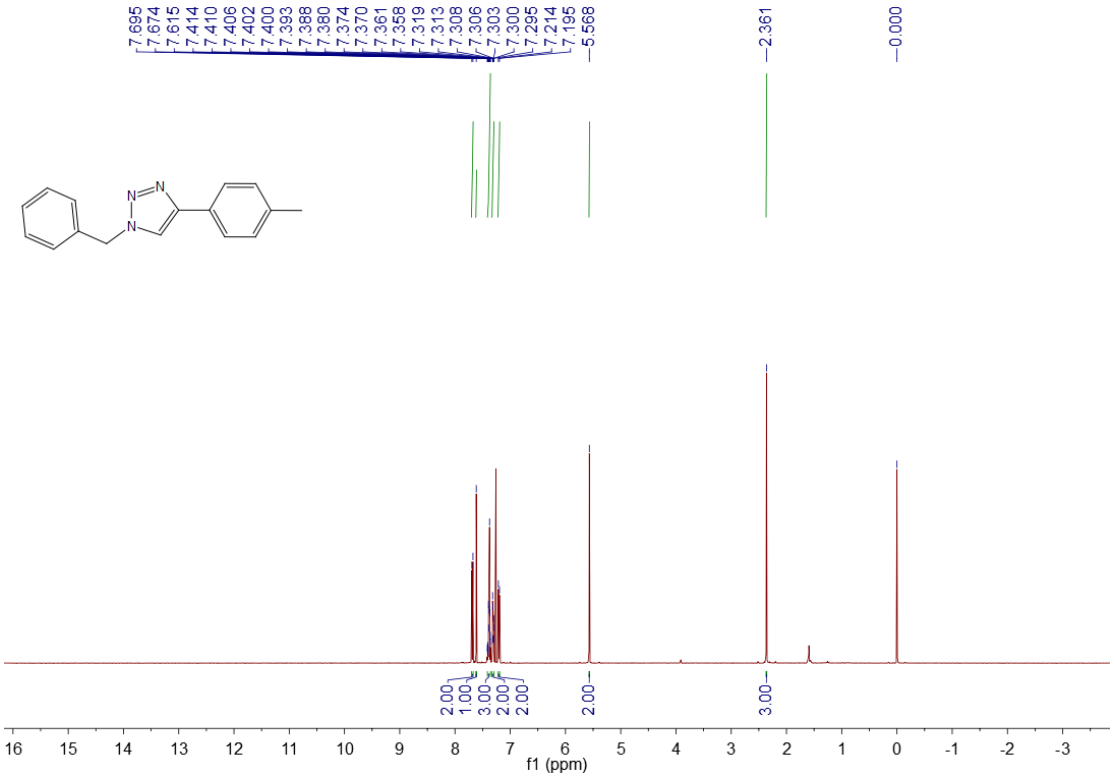
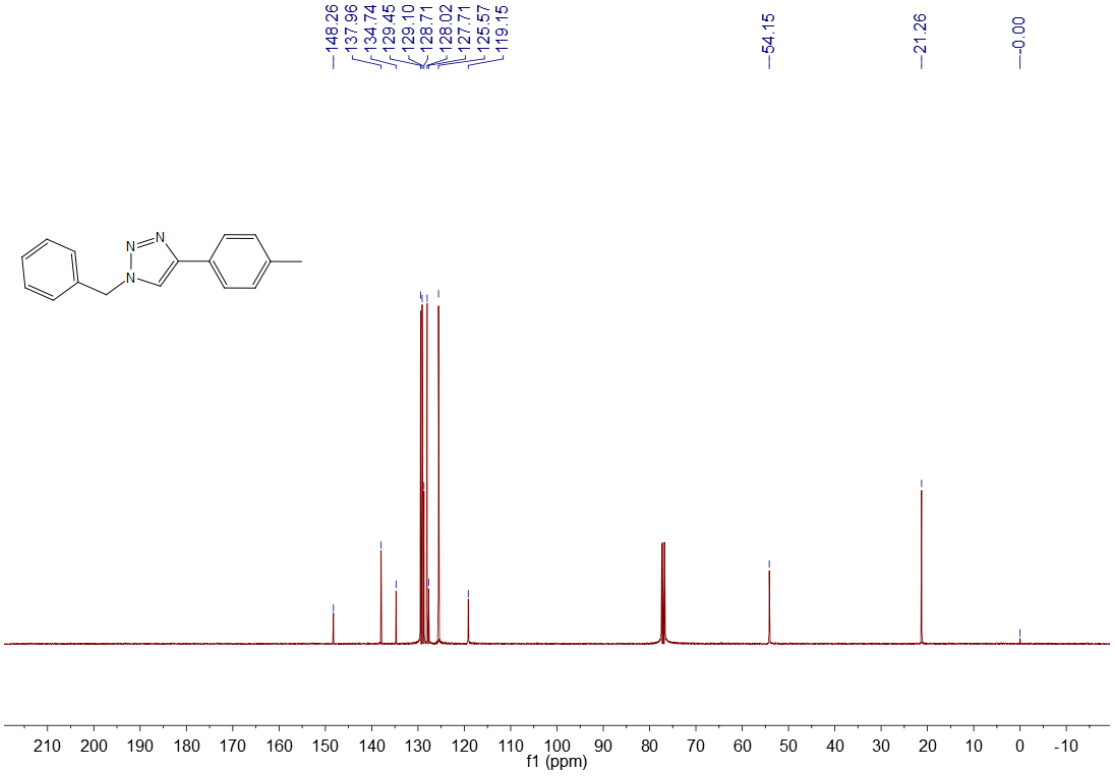
Copies of ^{1}H NMR and ^{13}C NMR spectra

1-Benzyl-4-phenyl-1*H*-1,2,3-triazole (7a)

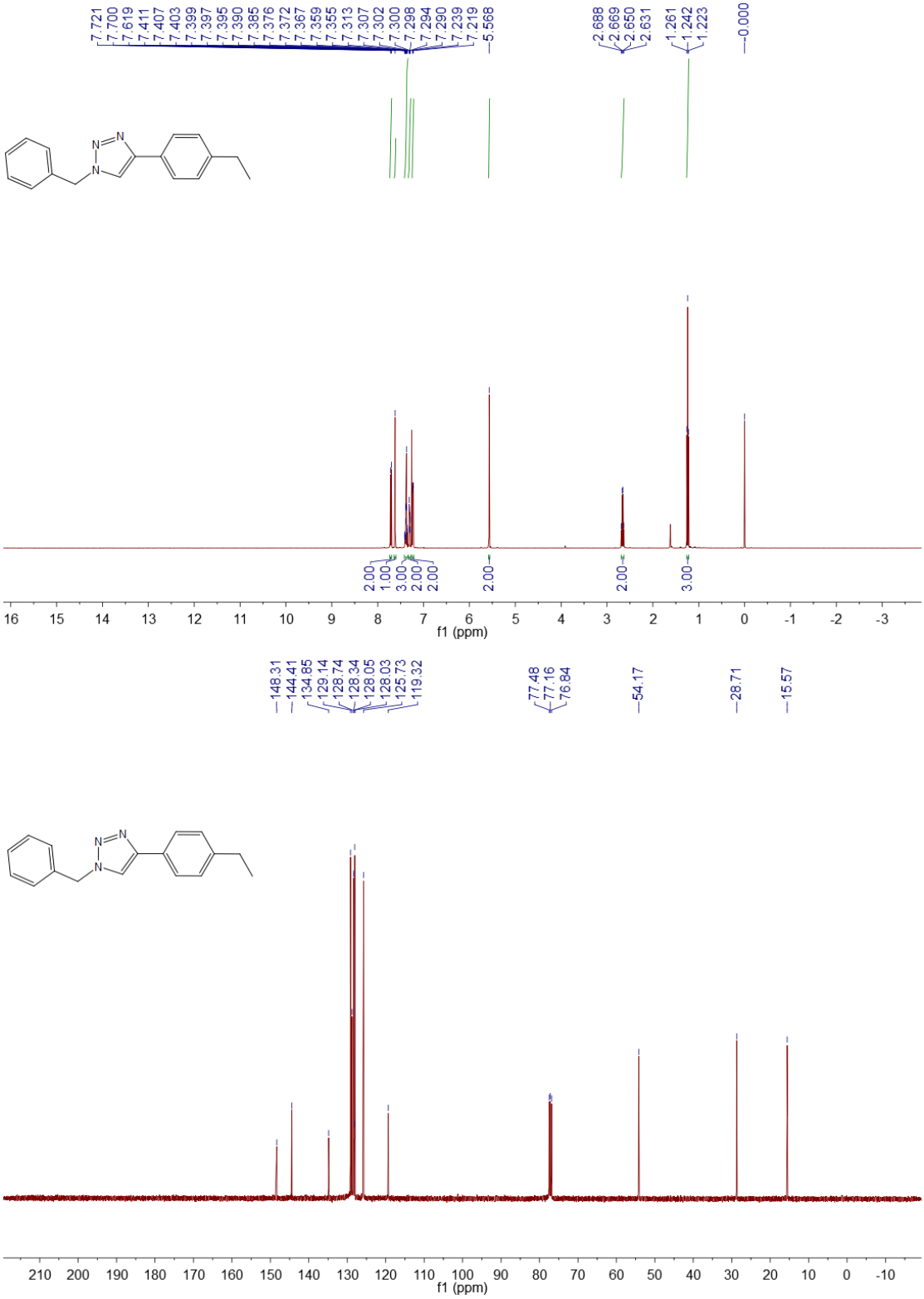


1-Benzyl-4-(p-tolyl)-1*H*-1,2,3-triazole (7b)

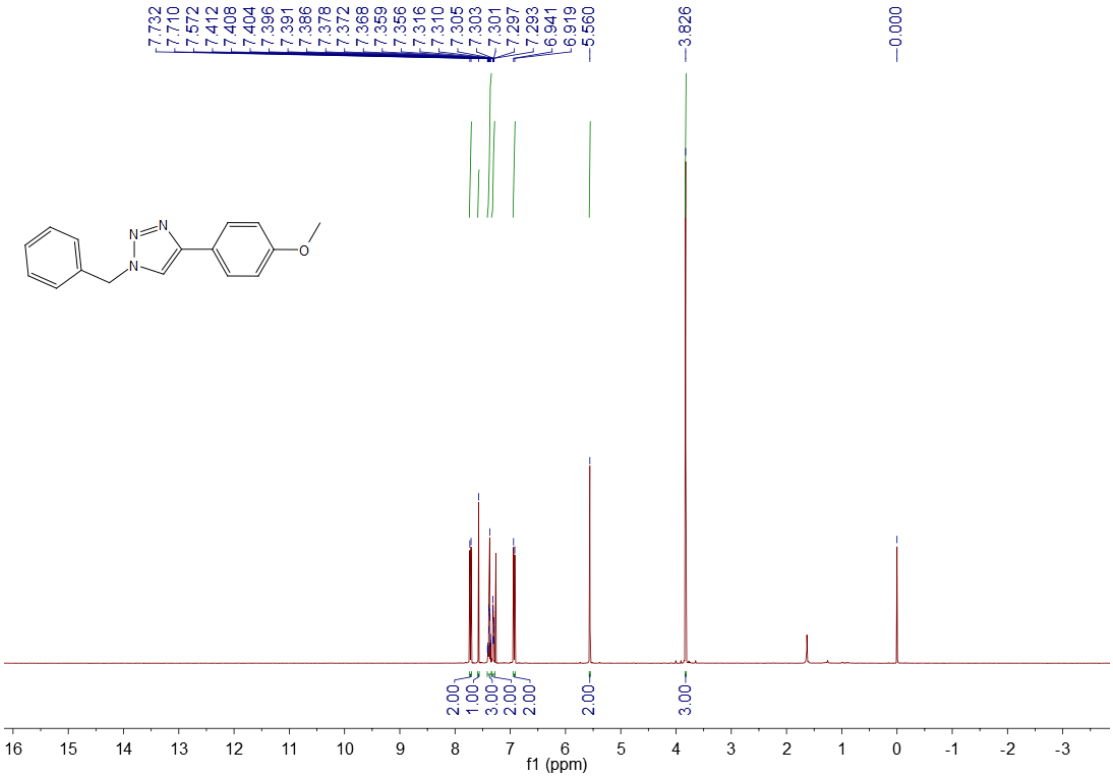


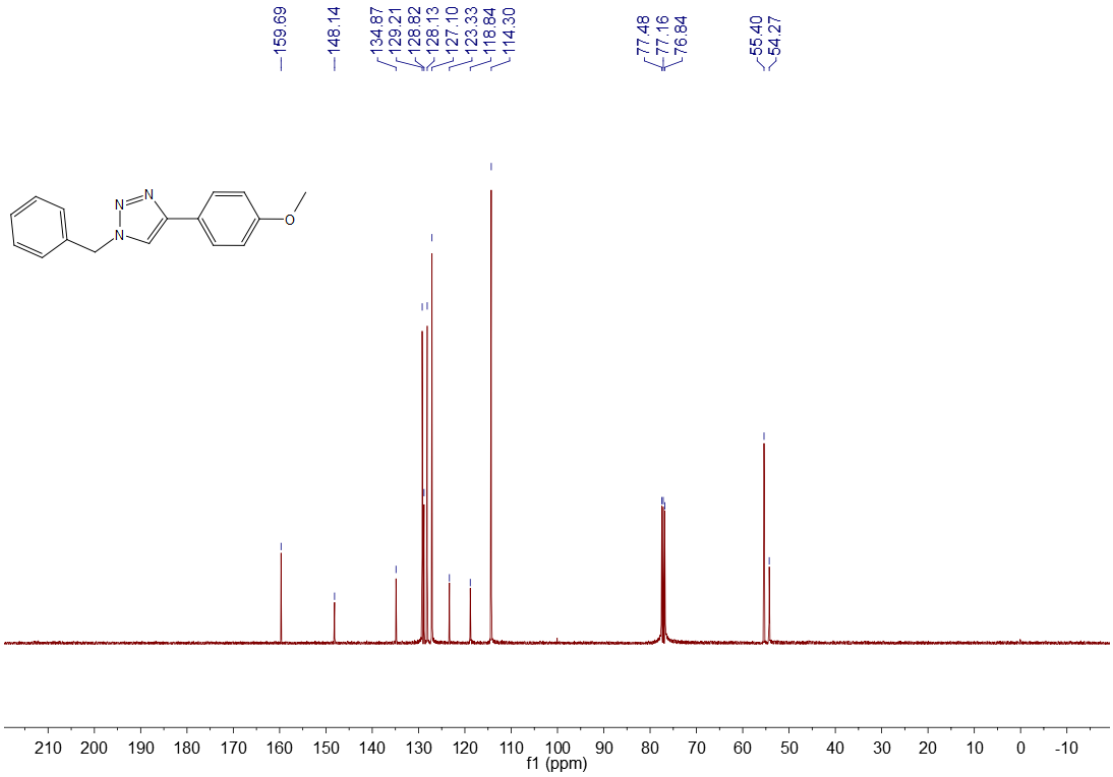


1-Benzyl-4-(4-ethylphenyl)-1*H*-1,2,3-triazole (7c)



1-Benzyl-4-(4-methoxyphenyl)-1*H*-1,2,3-triazole (7d)





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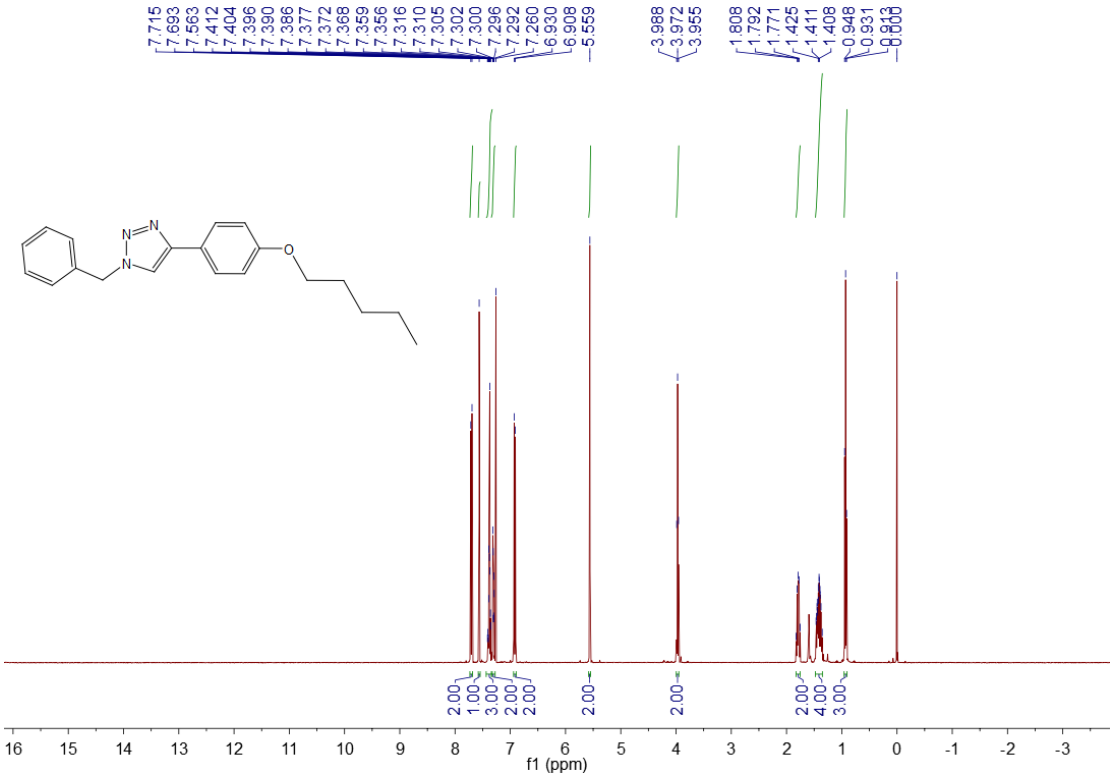

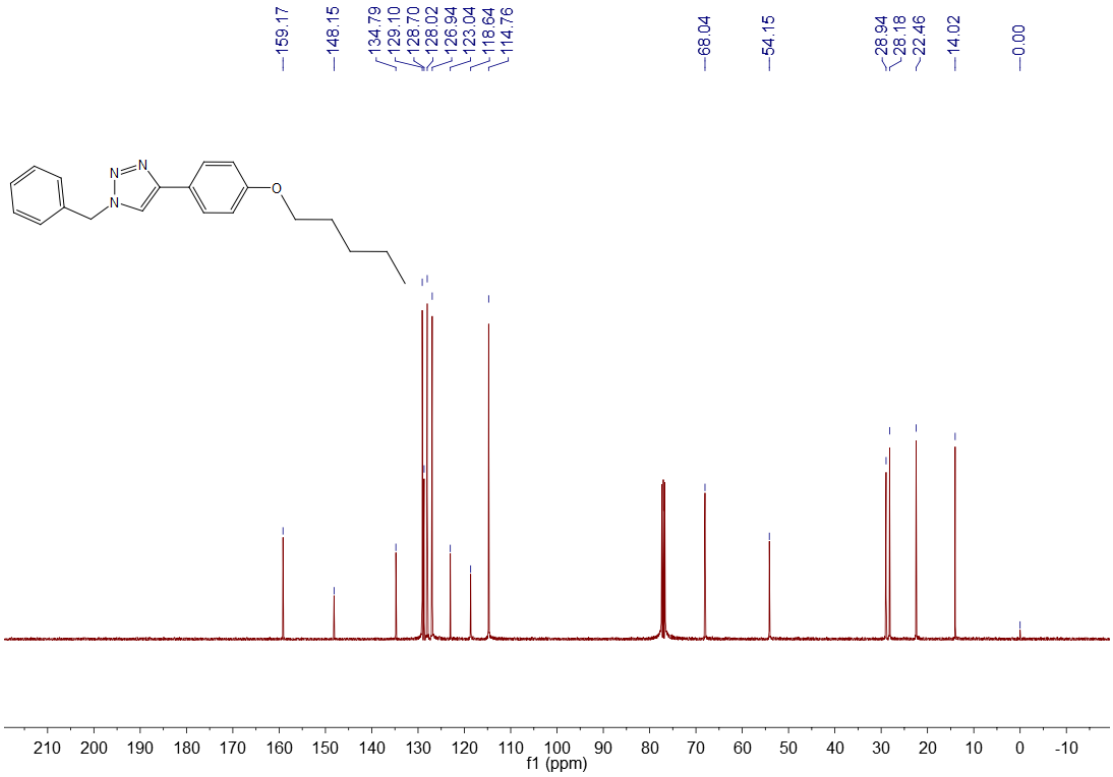

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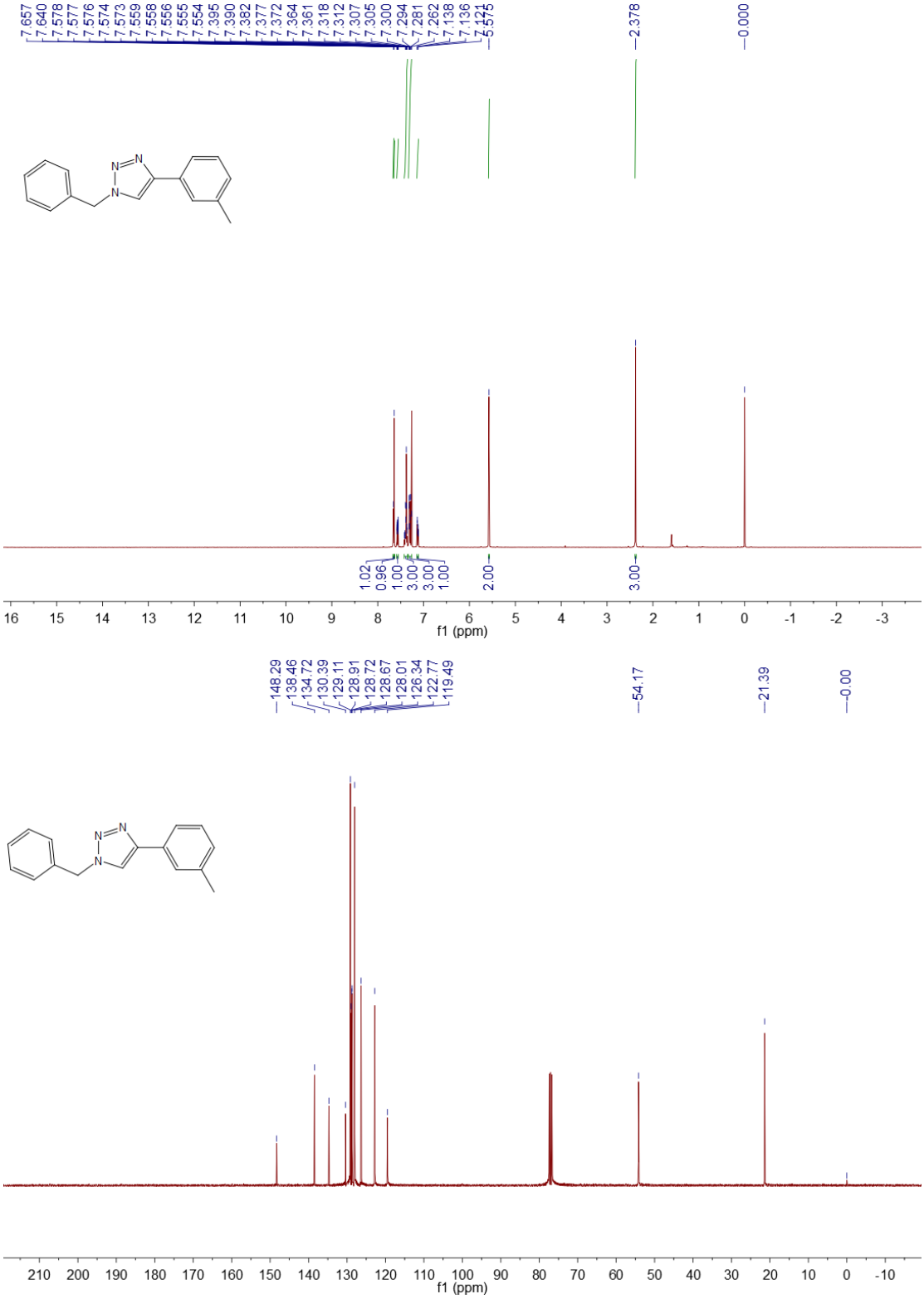
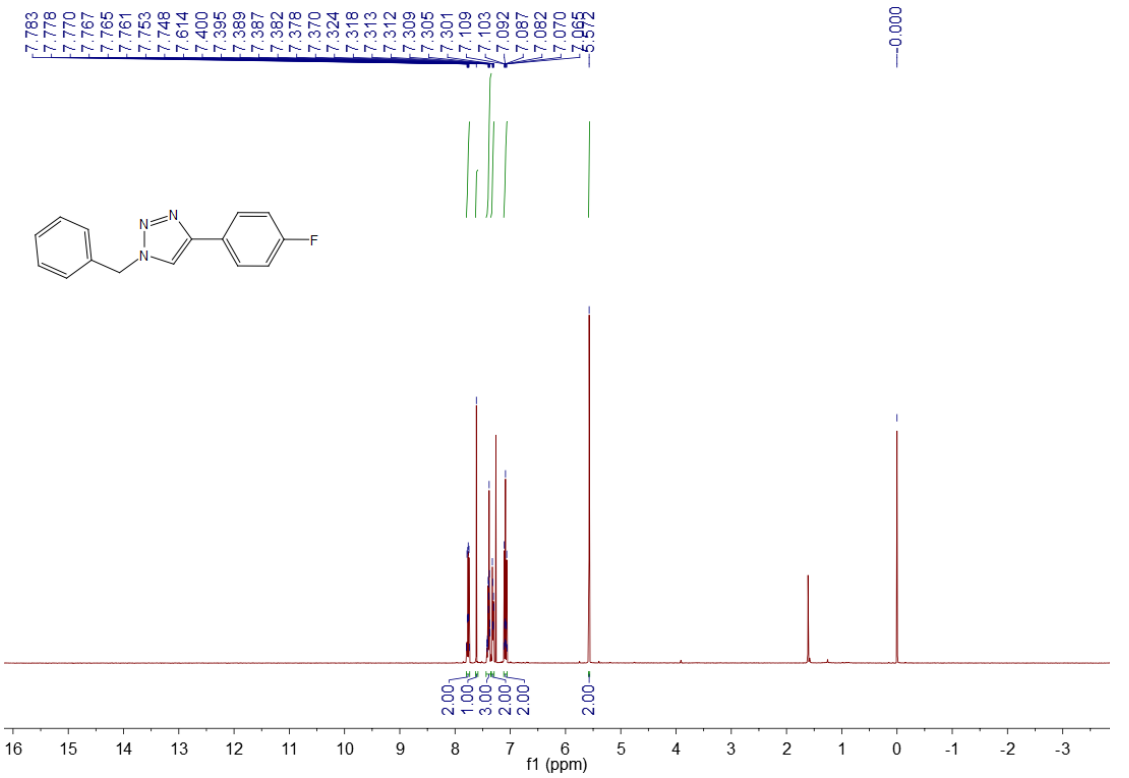
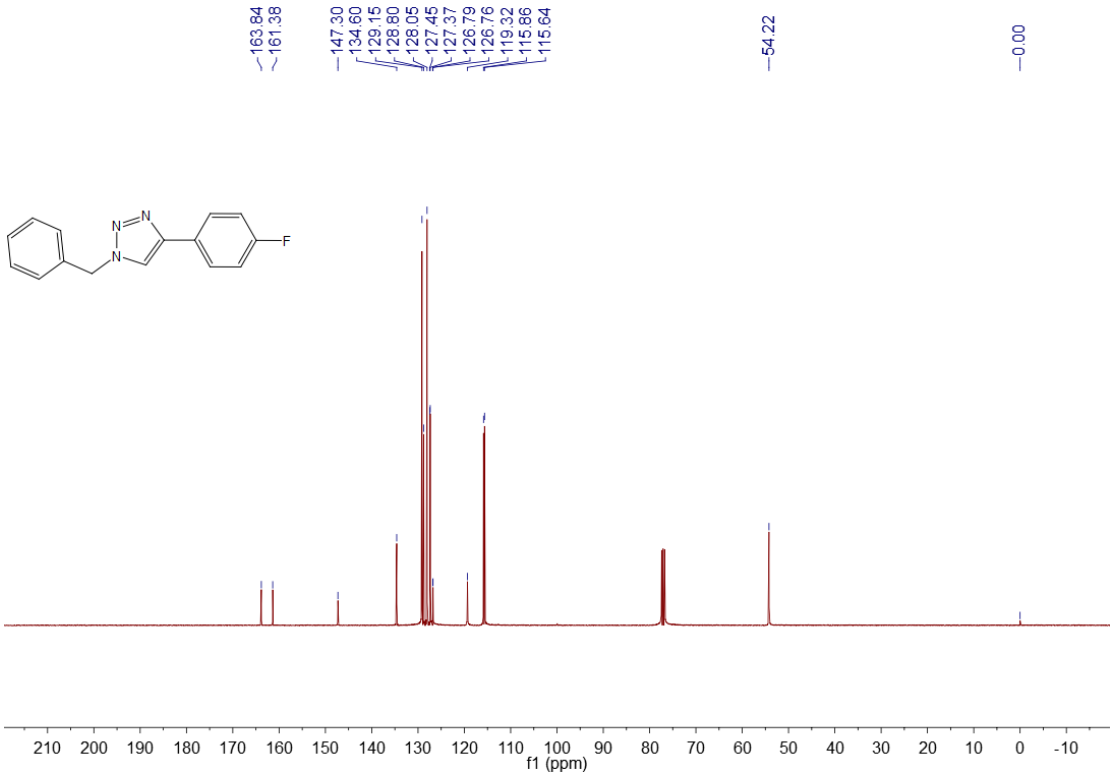


1-Benzyl-4-(4-fluorophenyl)-1*H*-1,2,3-triazole (7g)





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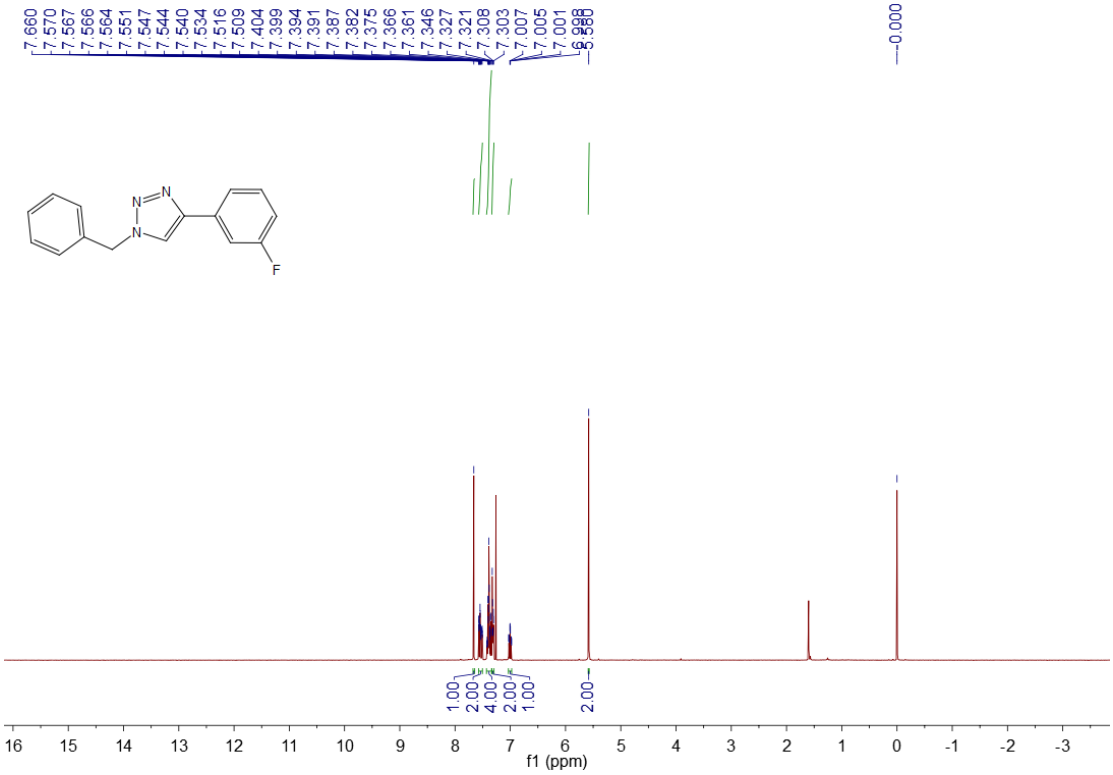
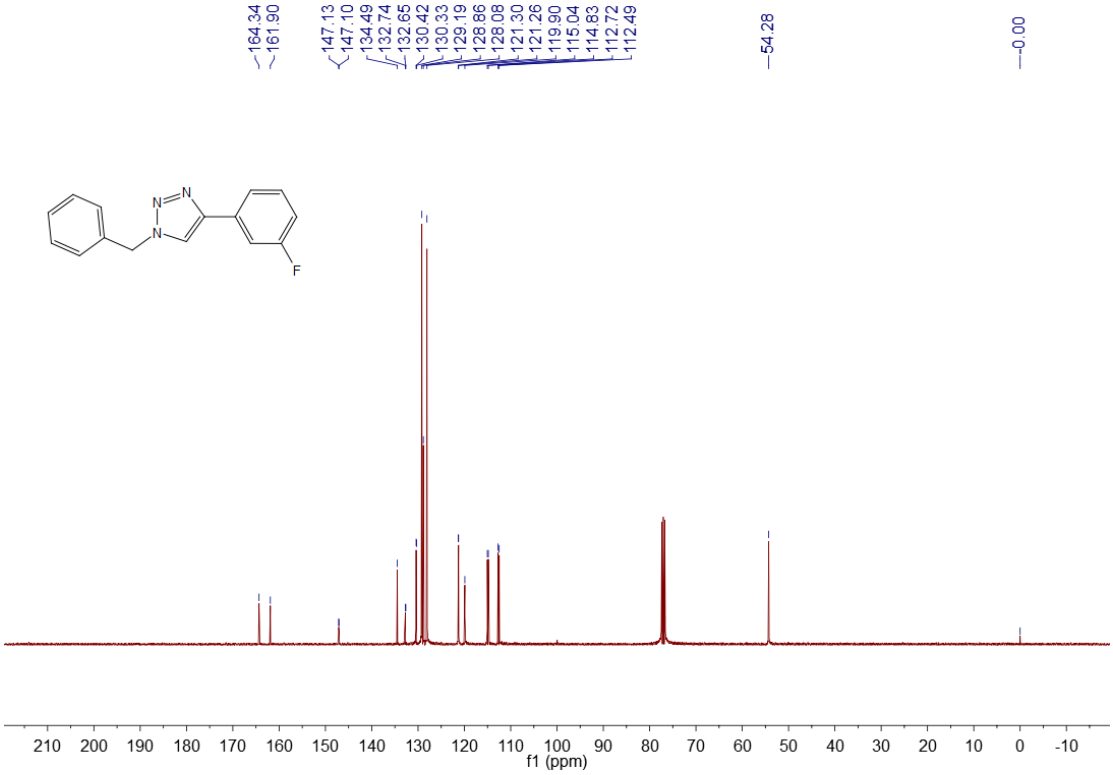
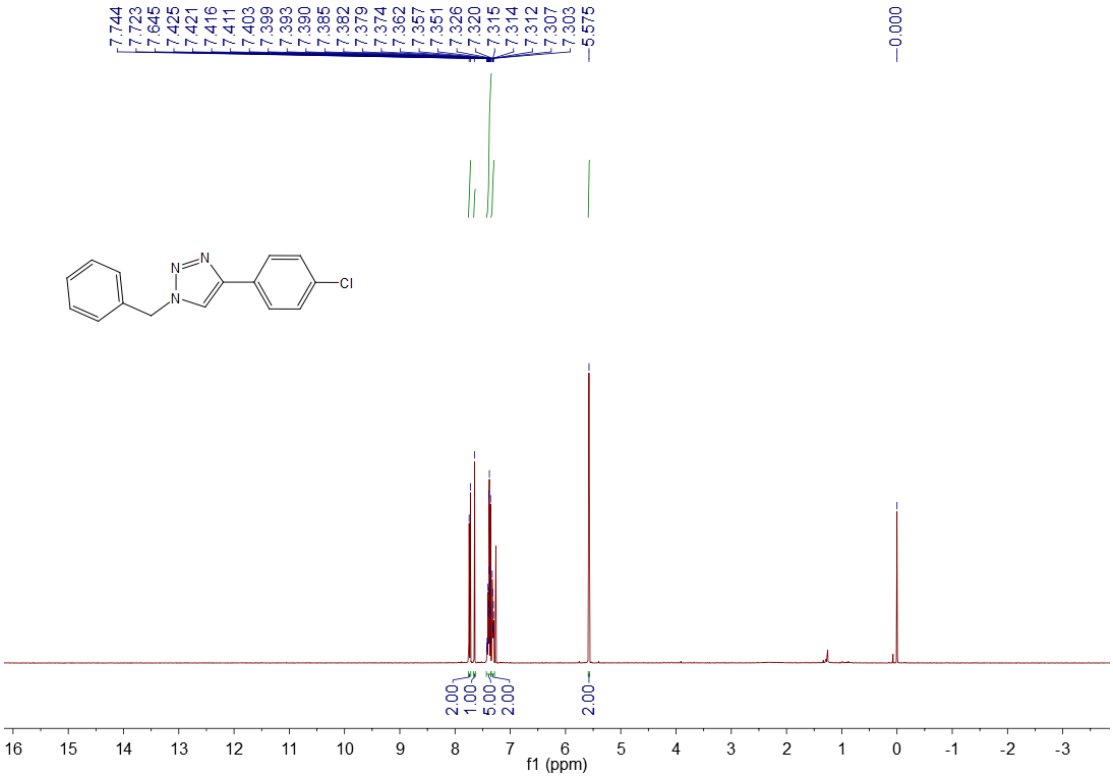
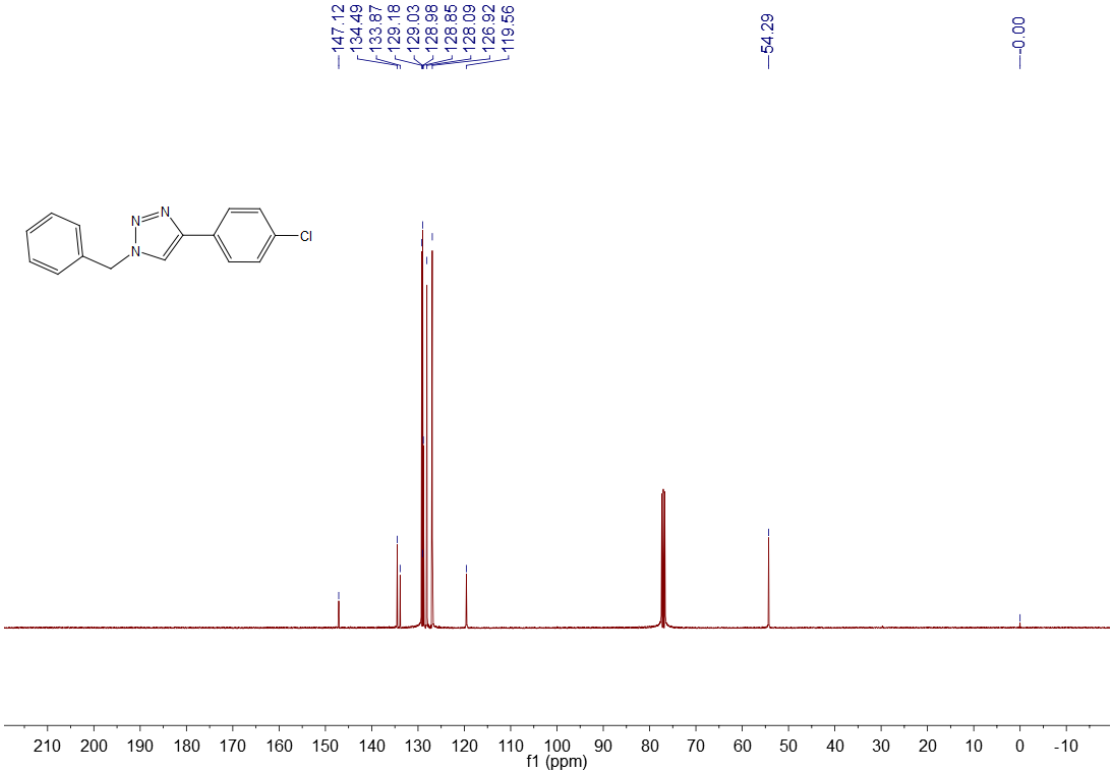


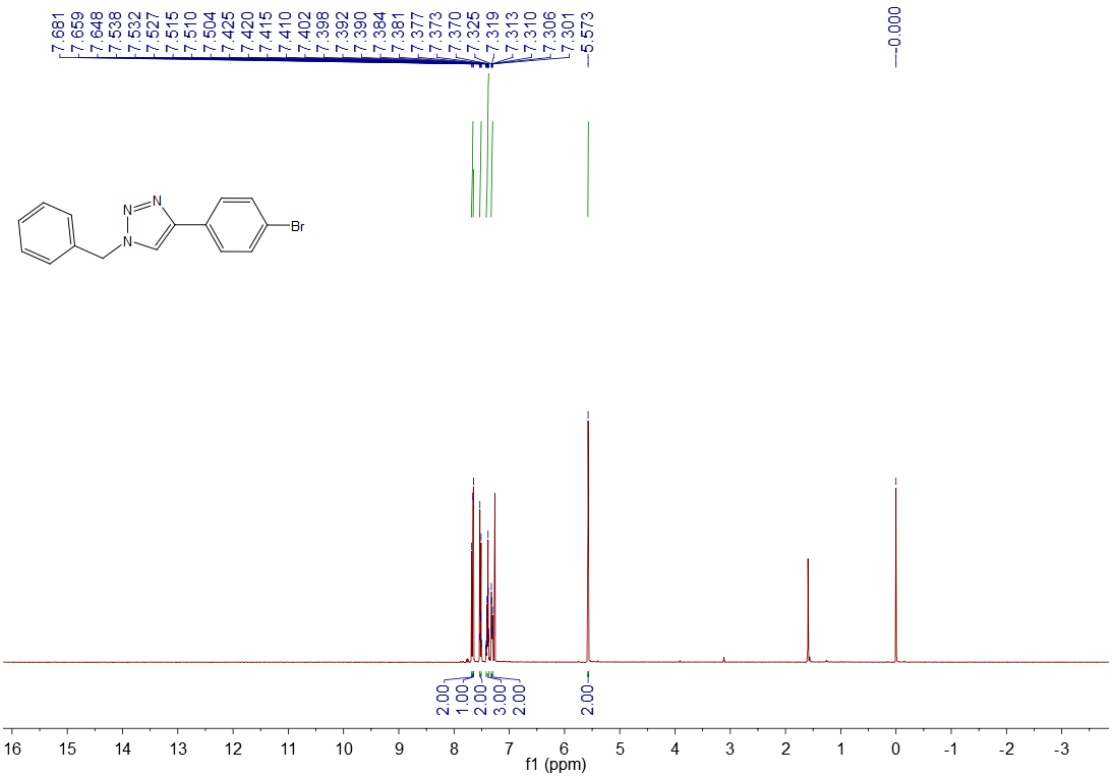


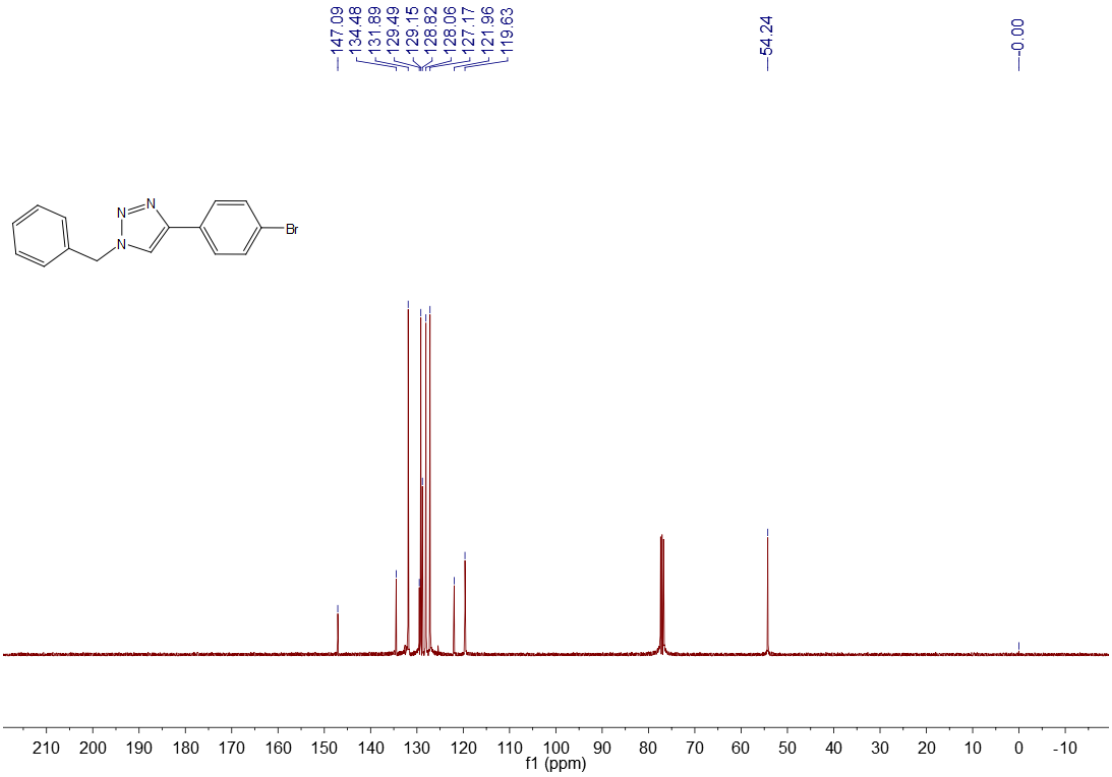
1-Benzyl-4-(4-chlorophenyl)-1H-1,2,3-triazole (7i)





1-Benzyl-4-(4-bromophenyl)-1*H*-1,2,3-triazole (7j)





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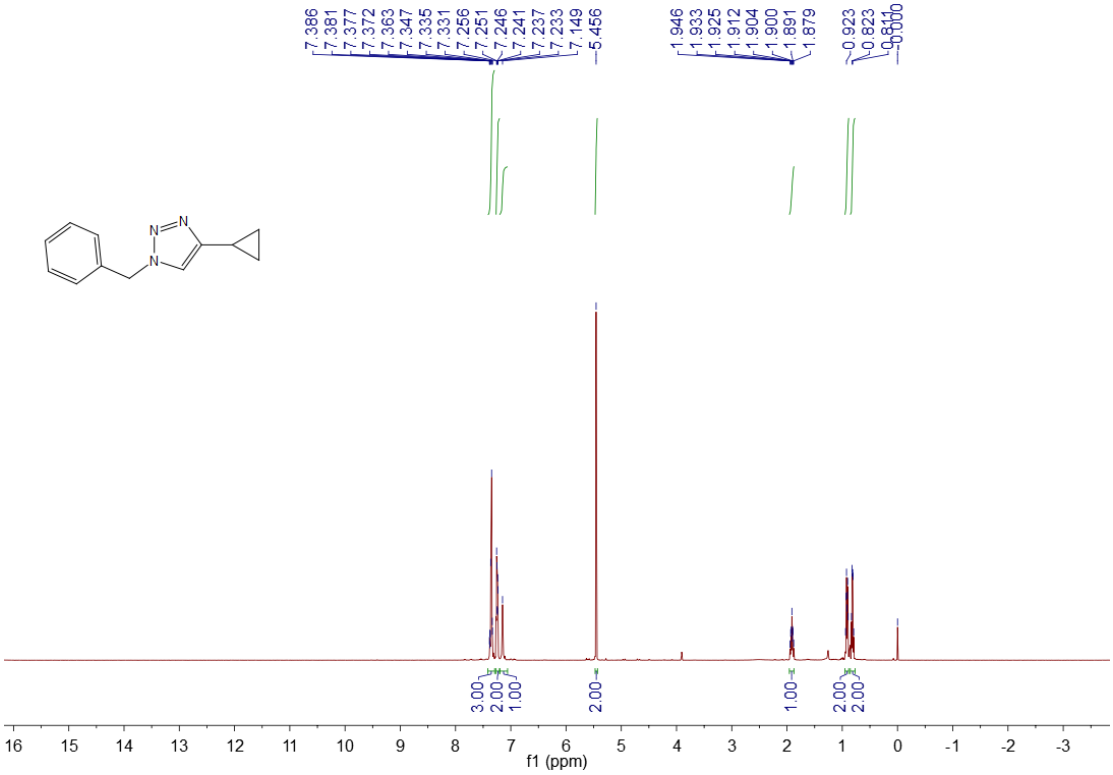
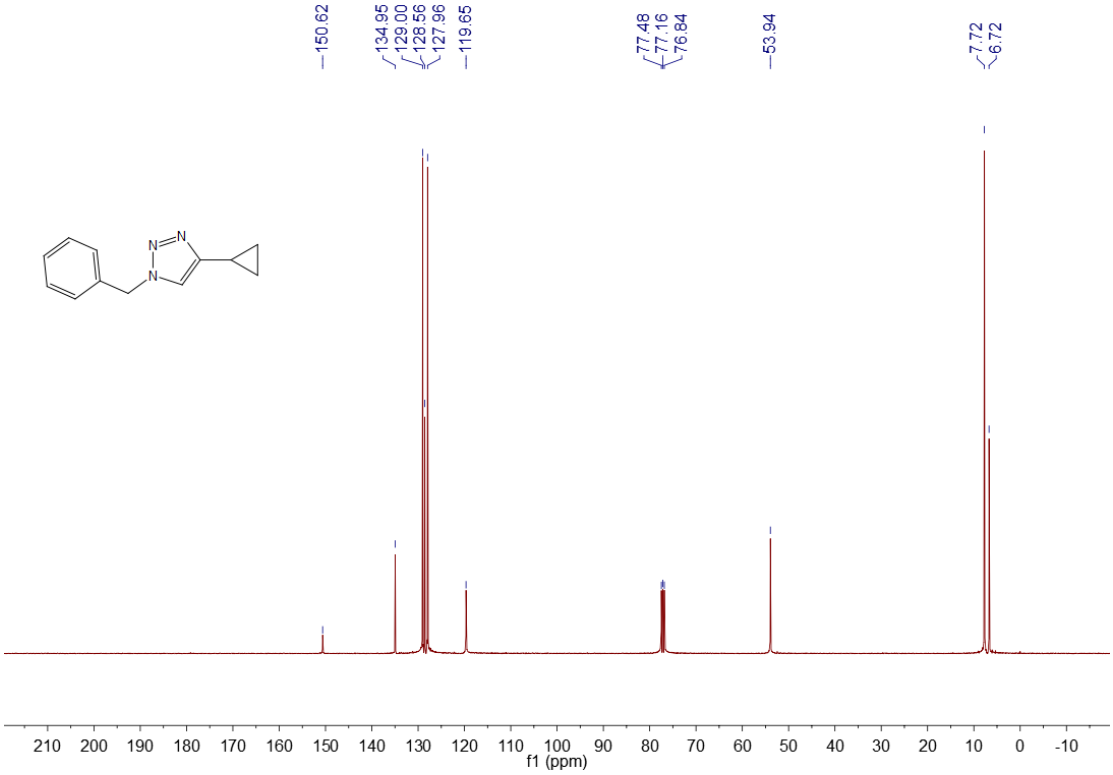
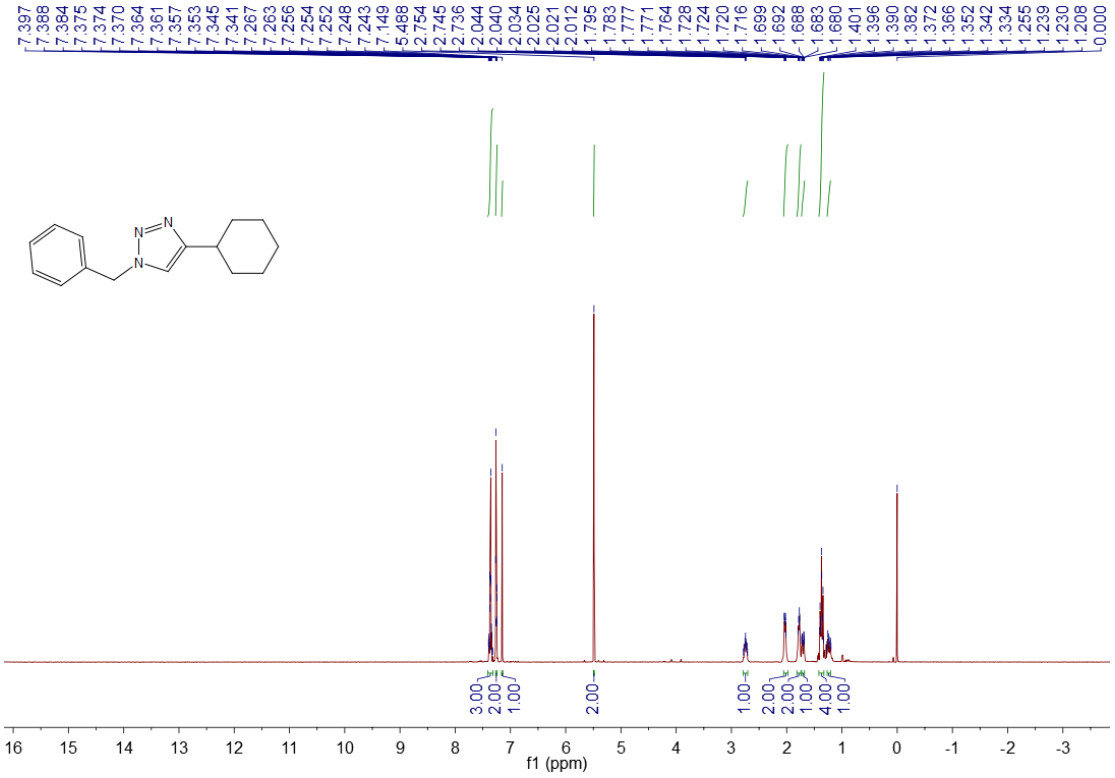
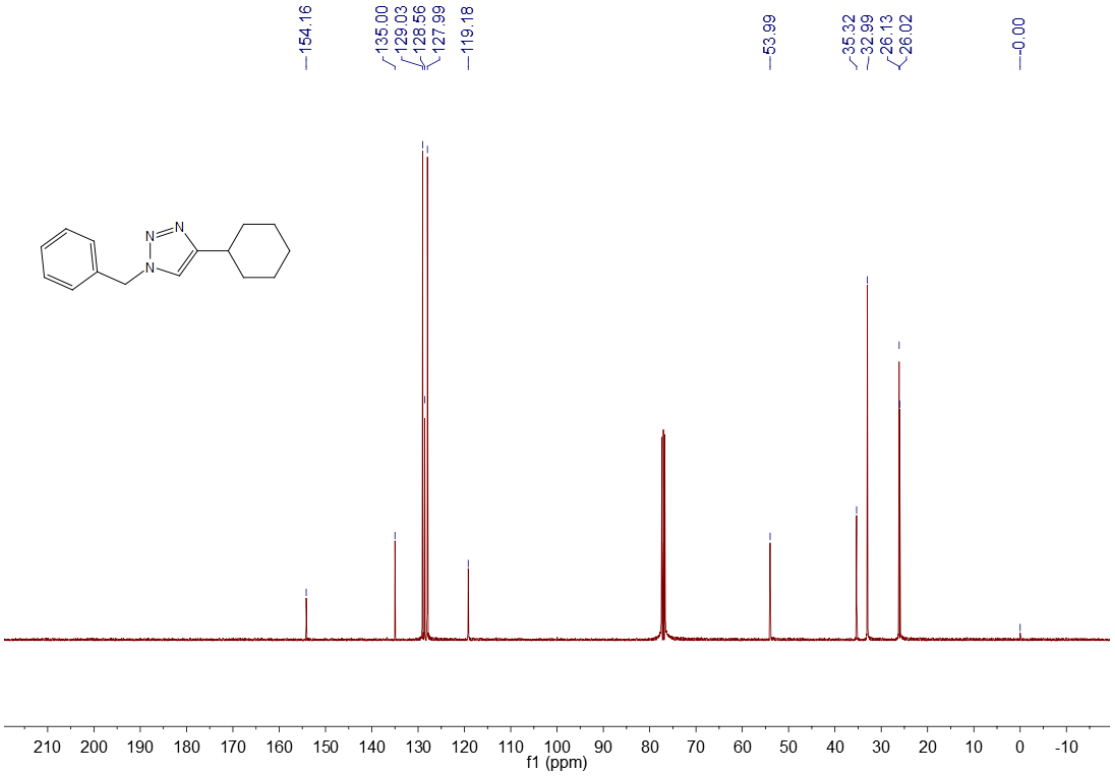




1-Benzyl-4-cyclohexyl-1H-1,2,3-triazole (7l)





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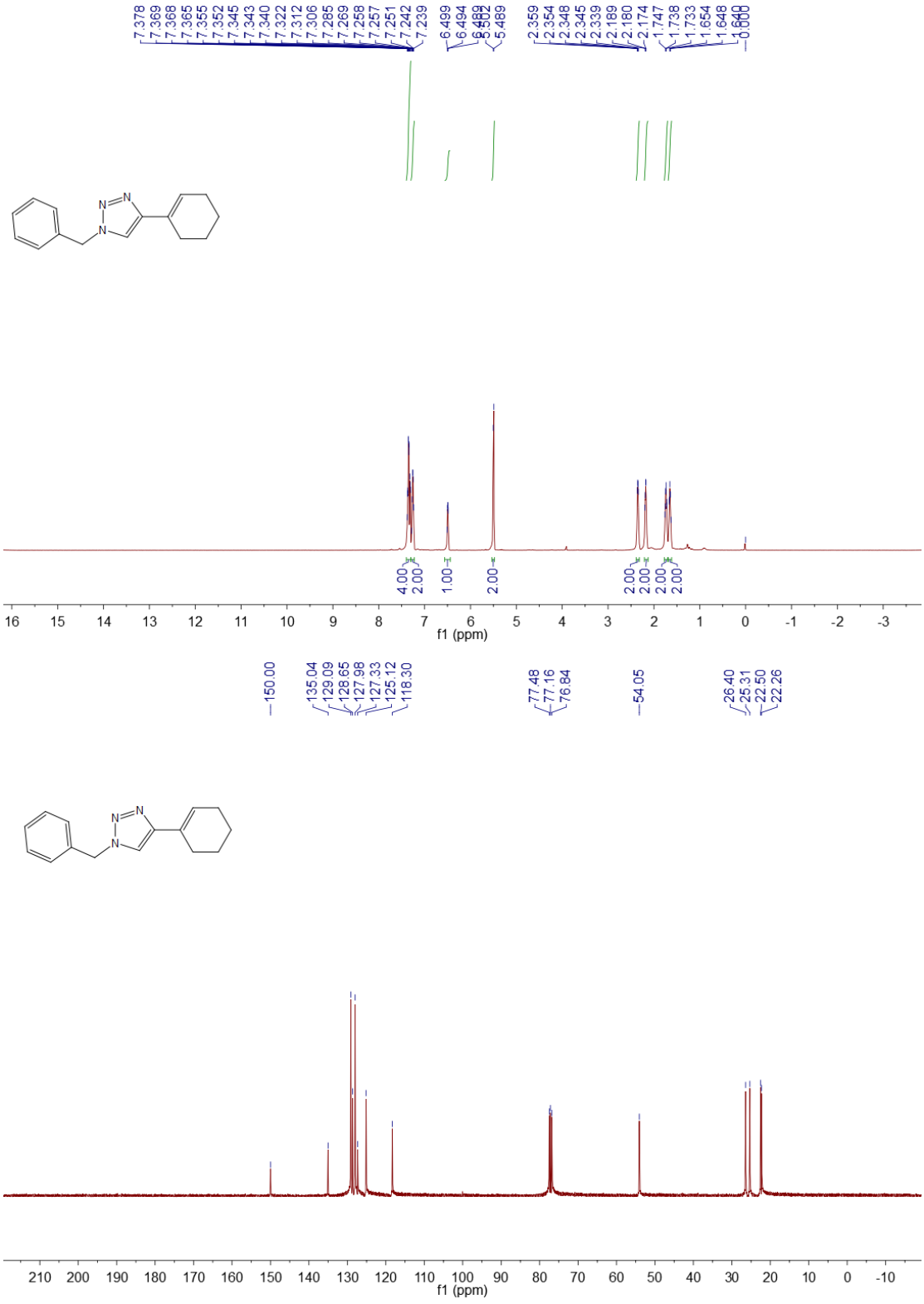
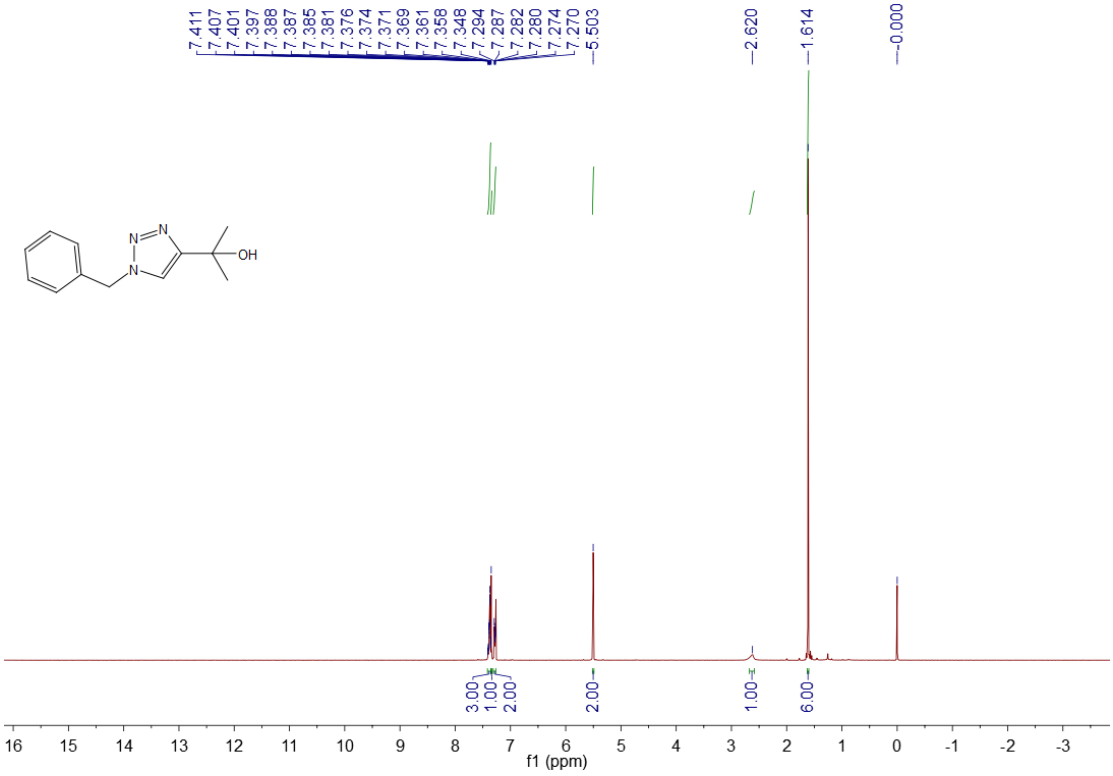
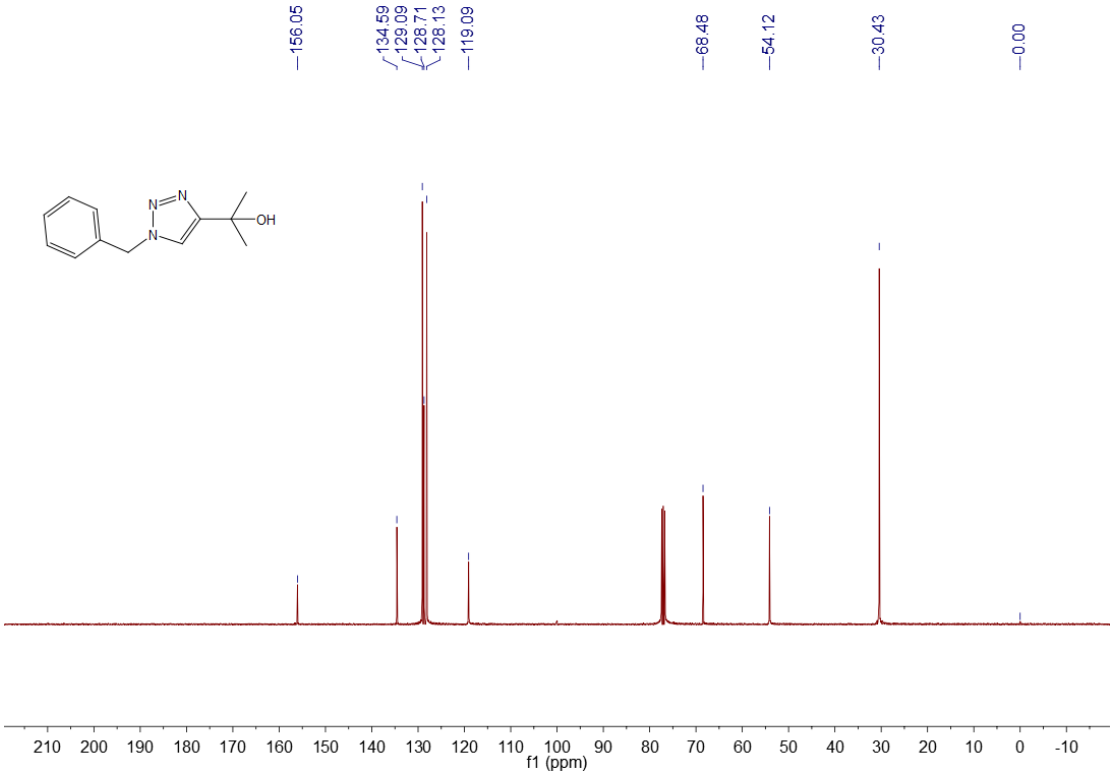
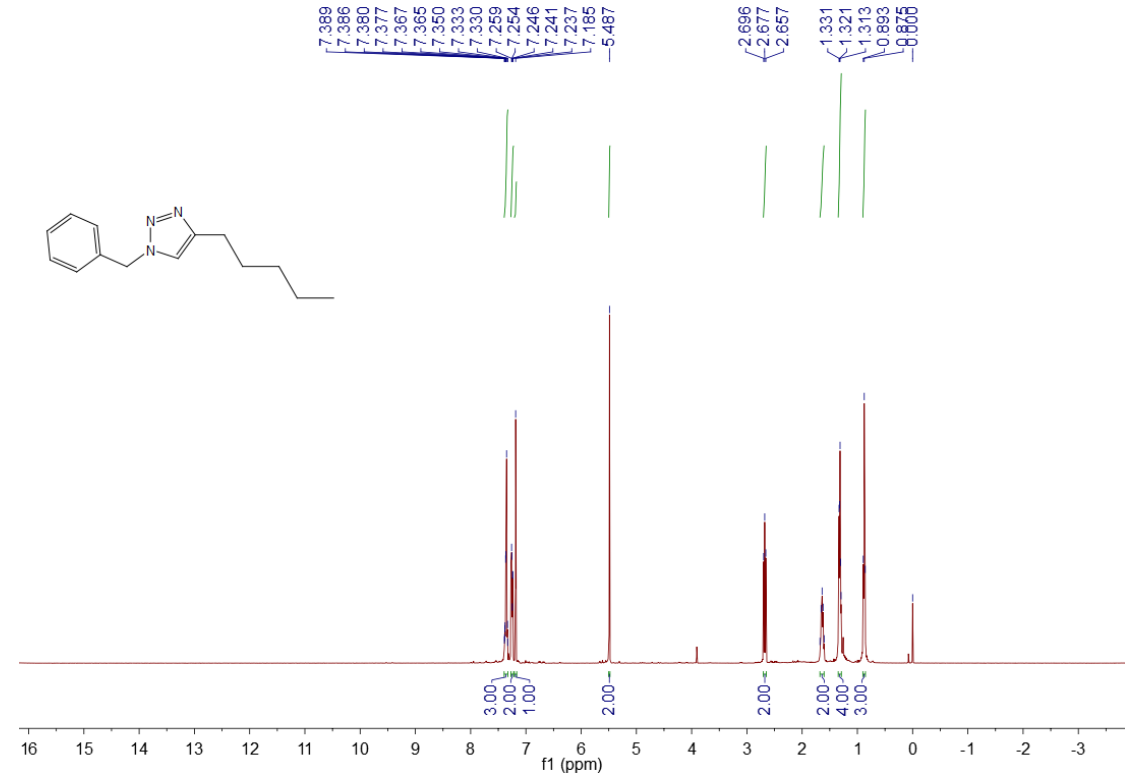


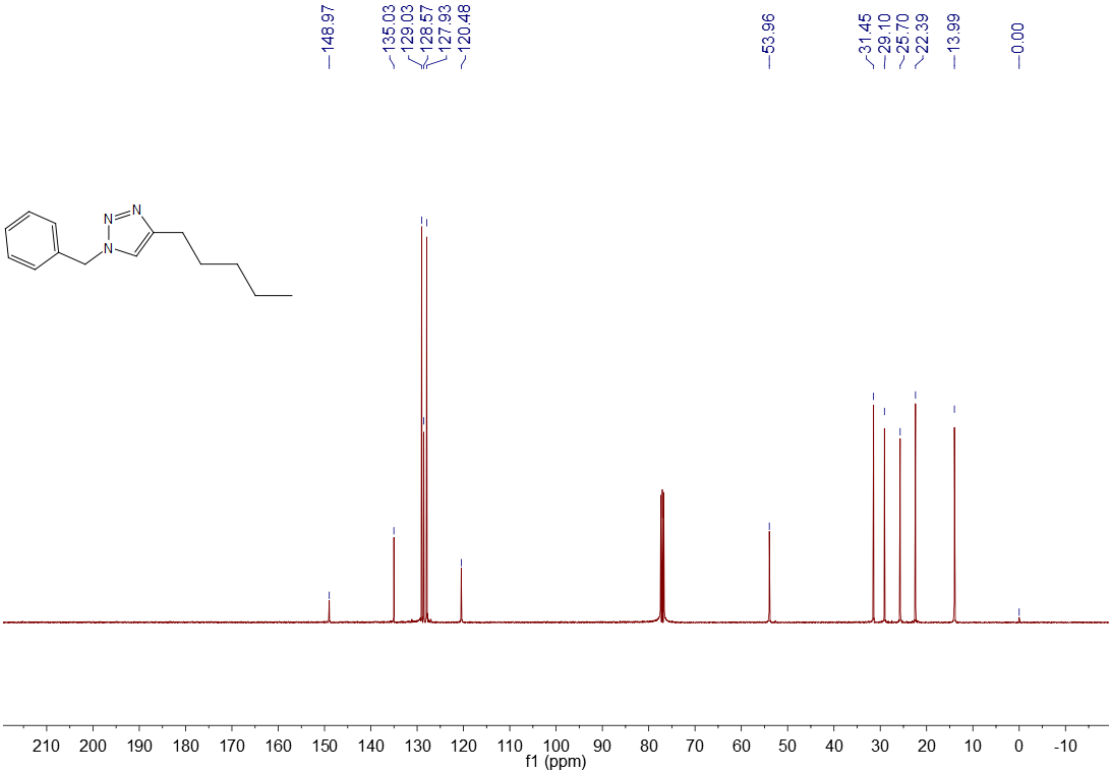
2-(1-Benzyl-1H-1,2,3-triazol-4-yl)propan-2-ol (7n)



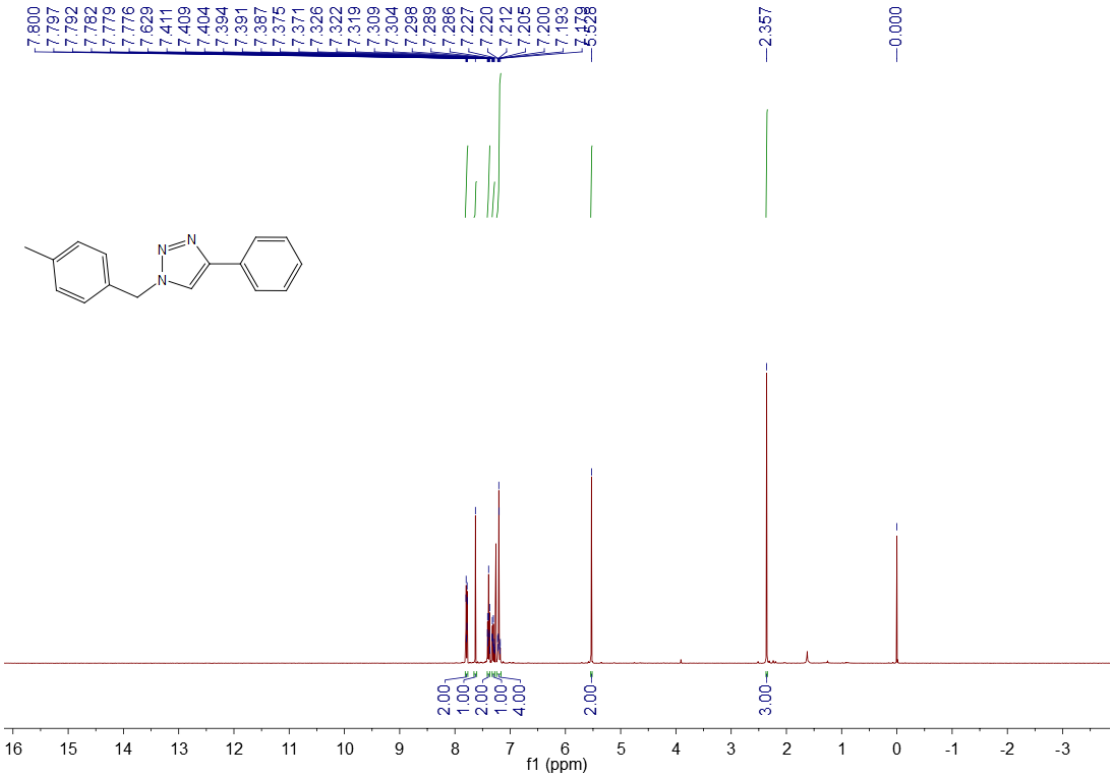


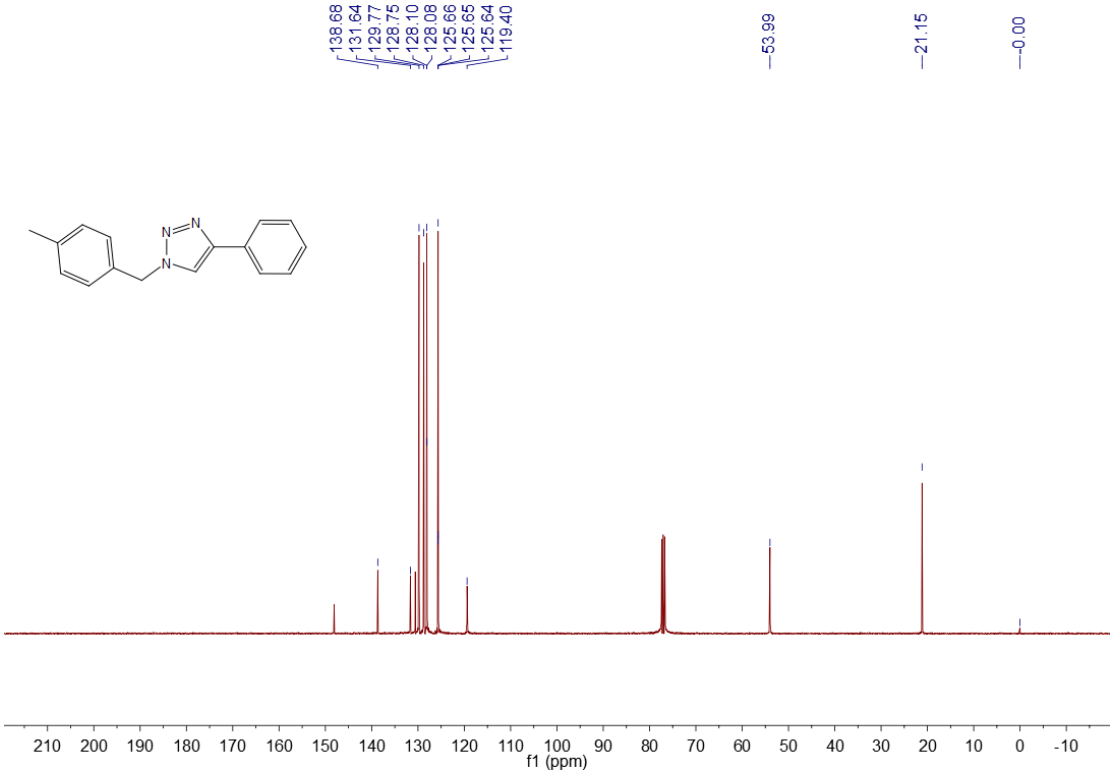
1-Benzyl-4-pentyl-1*H*-1,2,3-triazole (7o)



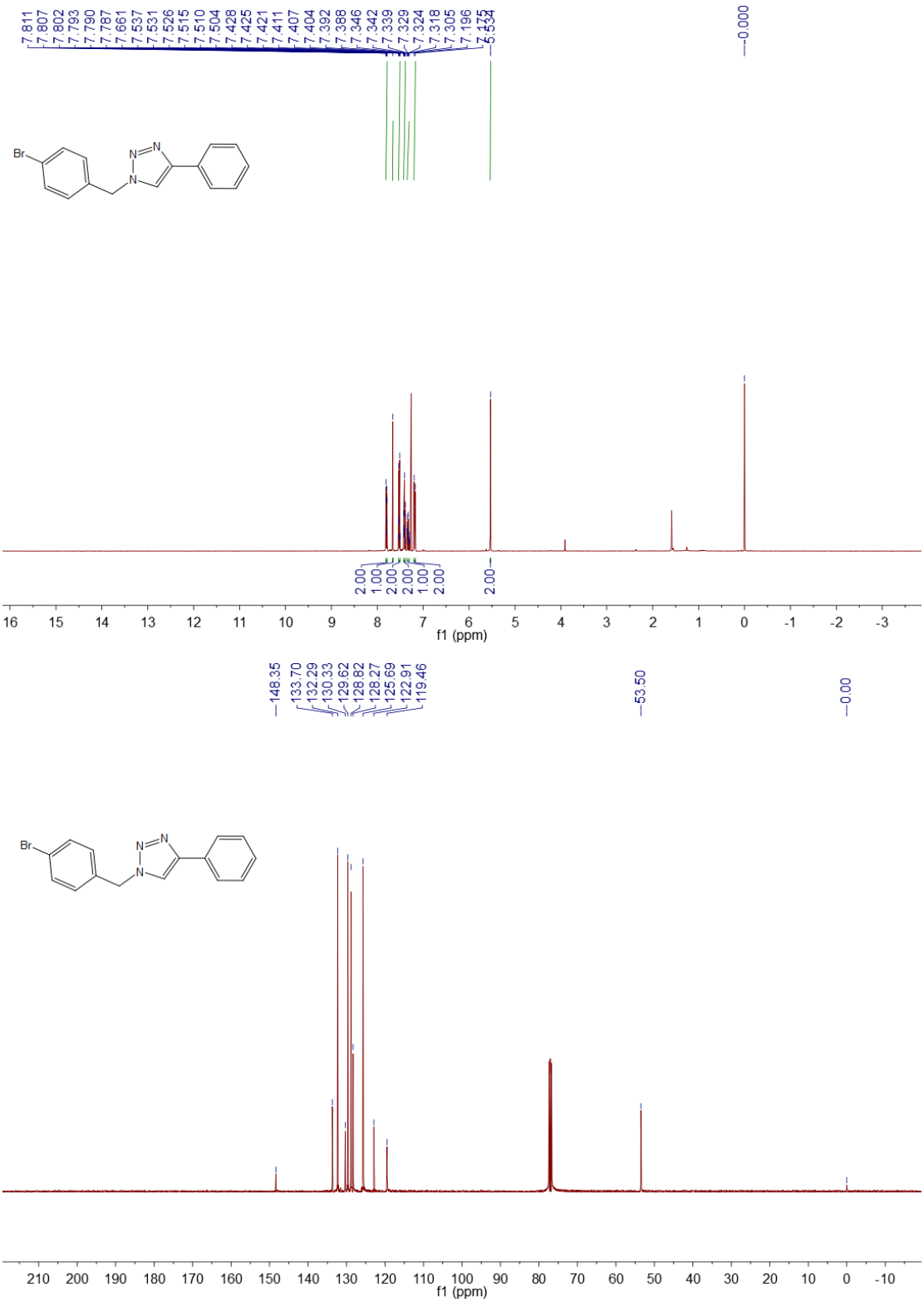


1-(4-Methylbenzyl)-4-phenyl-1*H*-1,2,3-triazole (7p)

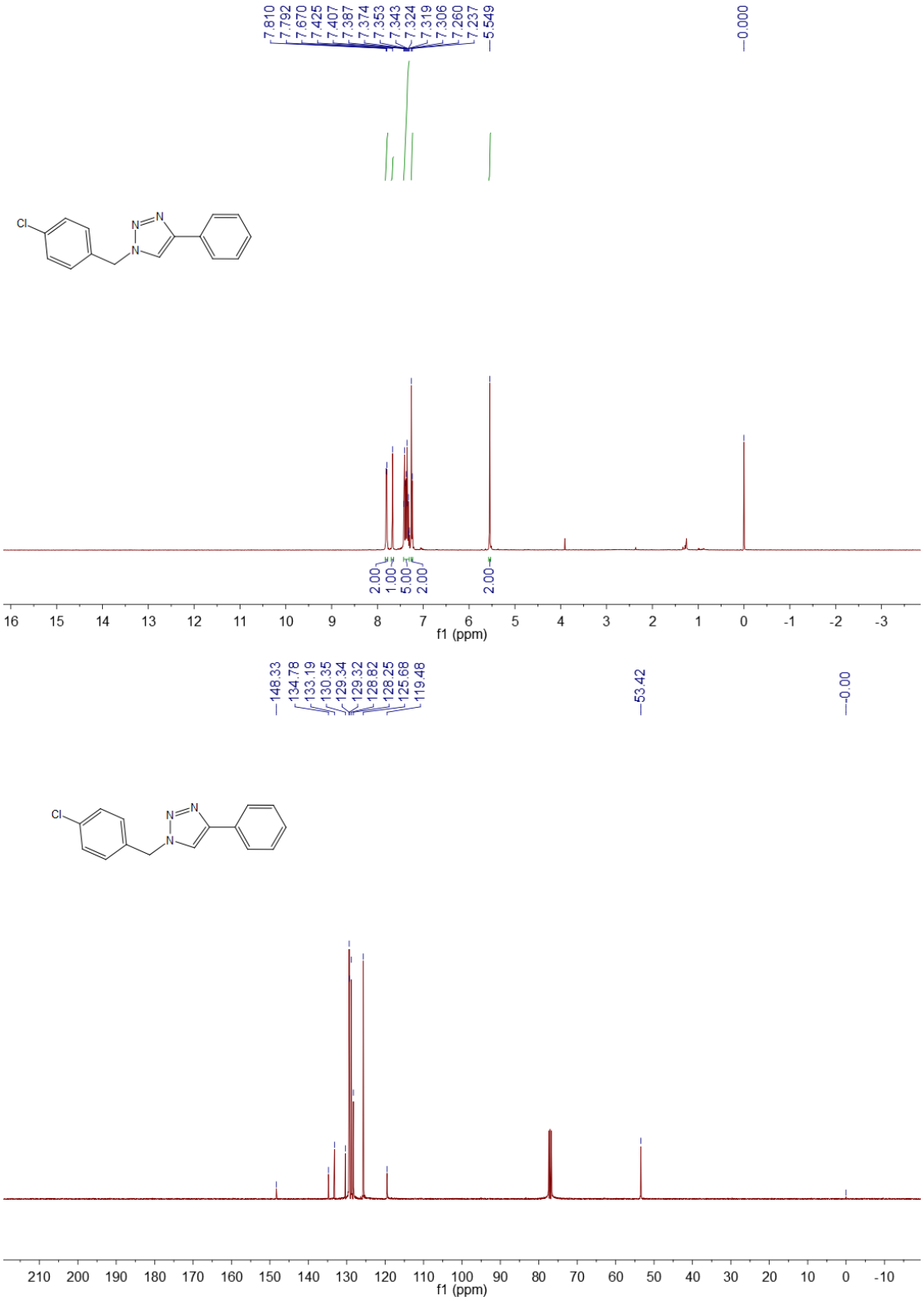




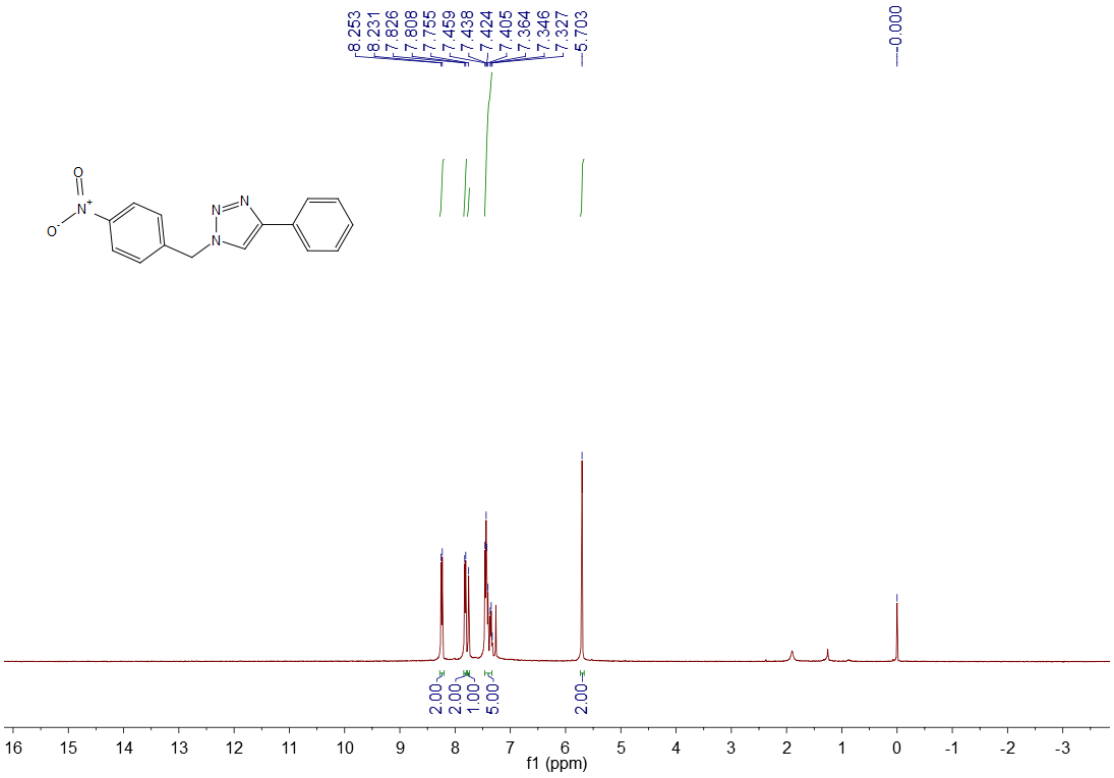
1-(4-Bromobenzyl)-4-phenyl-1*H*-1,2,3-triazole (7q)

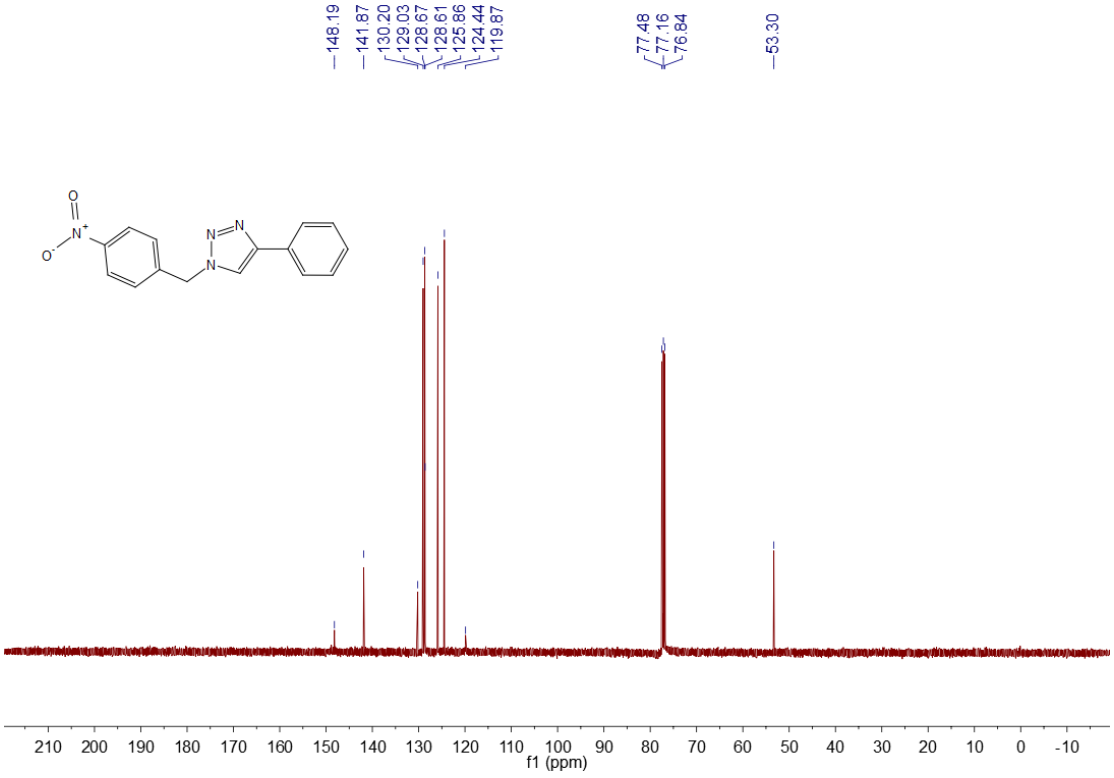


1-(4-Chlorobenzyl)-4-phenyl-1*H*-1,2,3-triazole (7r)

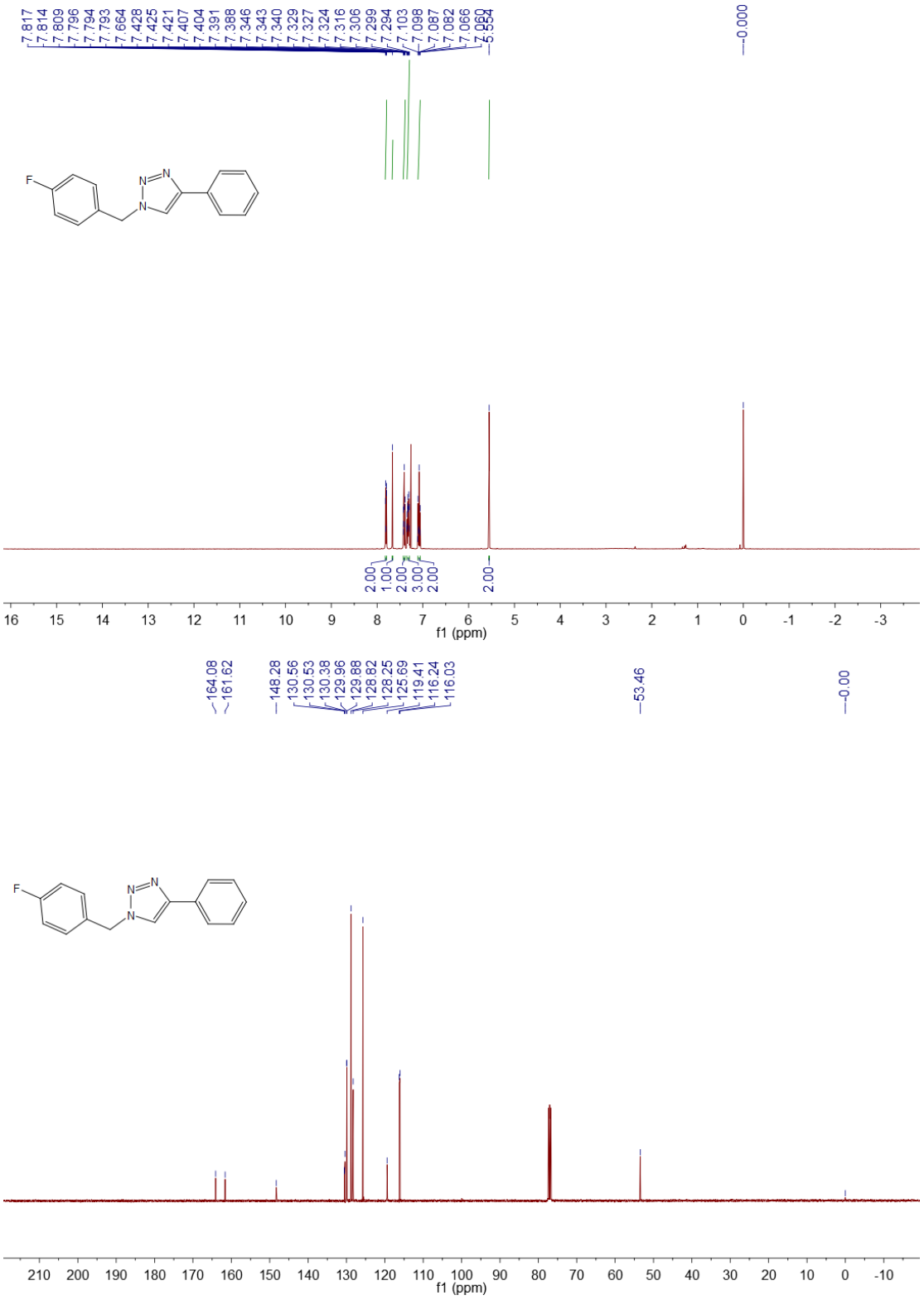


1-(4-Nitrobenzyl)-4-phenyl-1*H*-1,2,3-triazole (7s)





1-(4-Fluorobenzyl)-4-phenyl-1*H*-1,2,3-triazole (7t)



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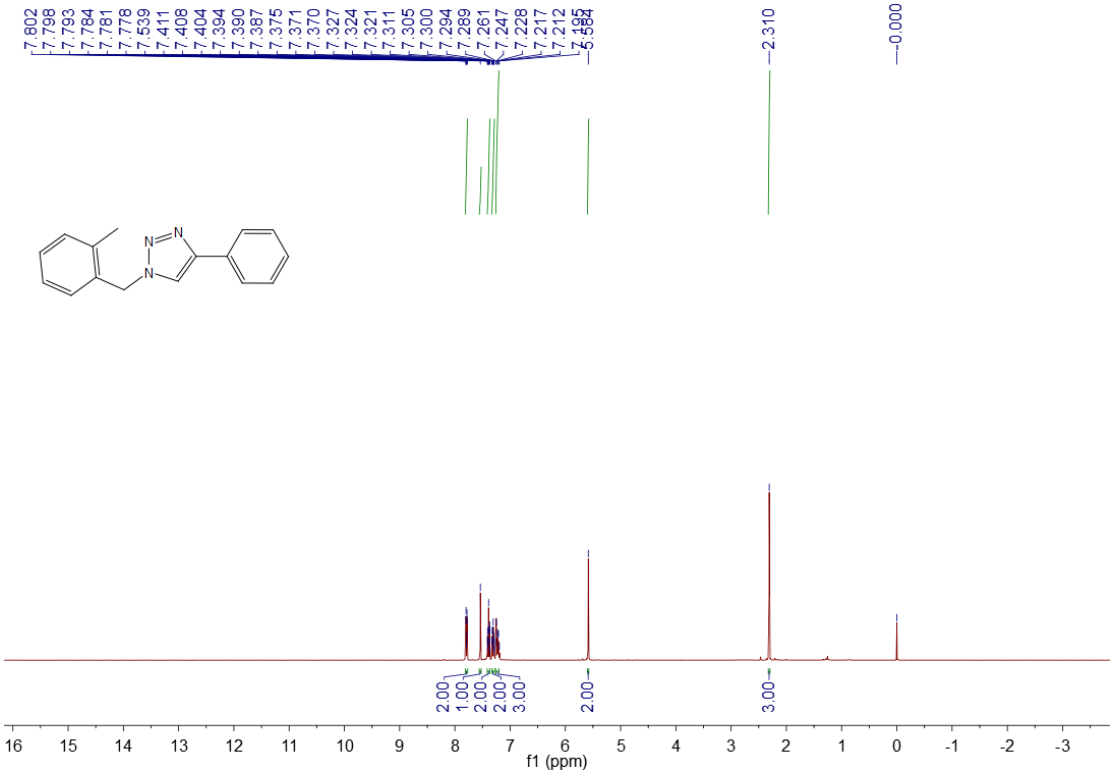
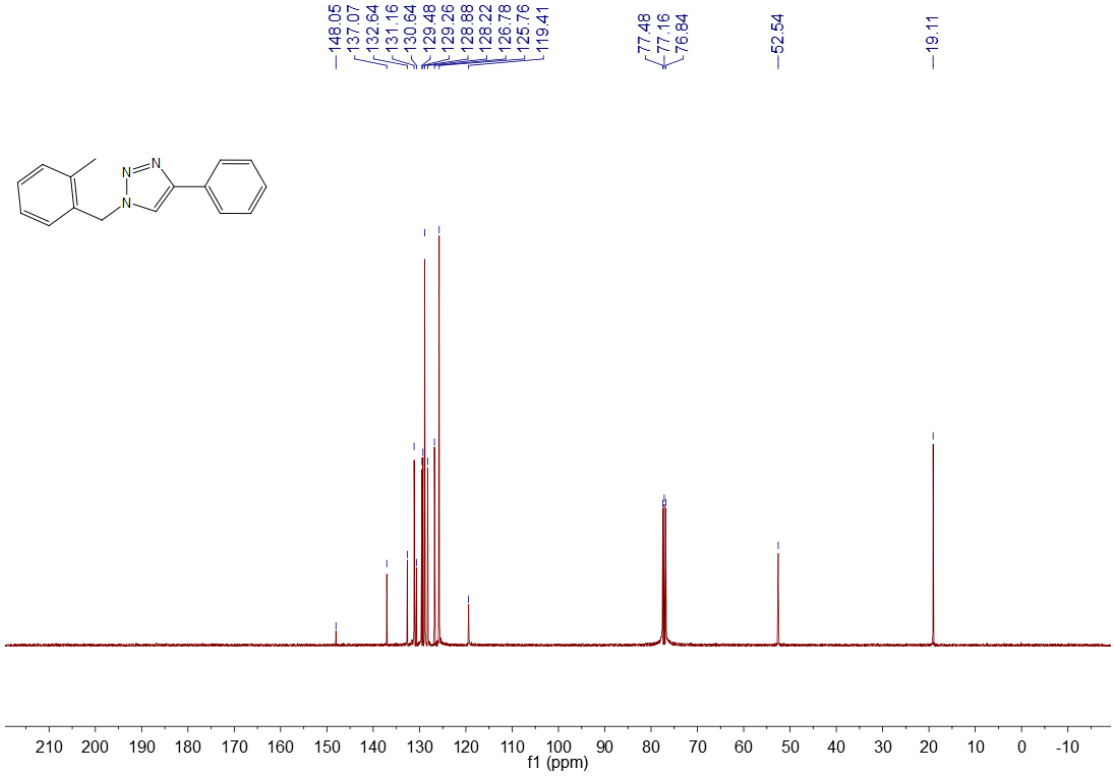
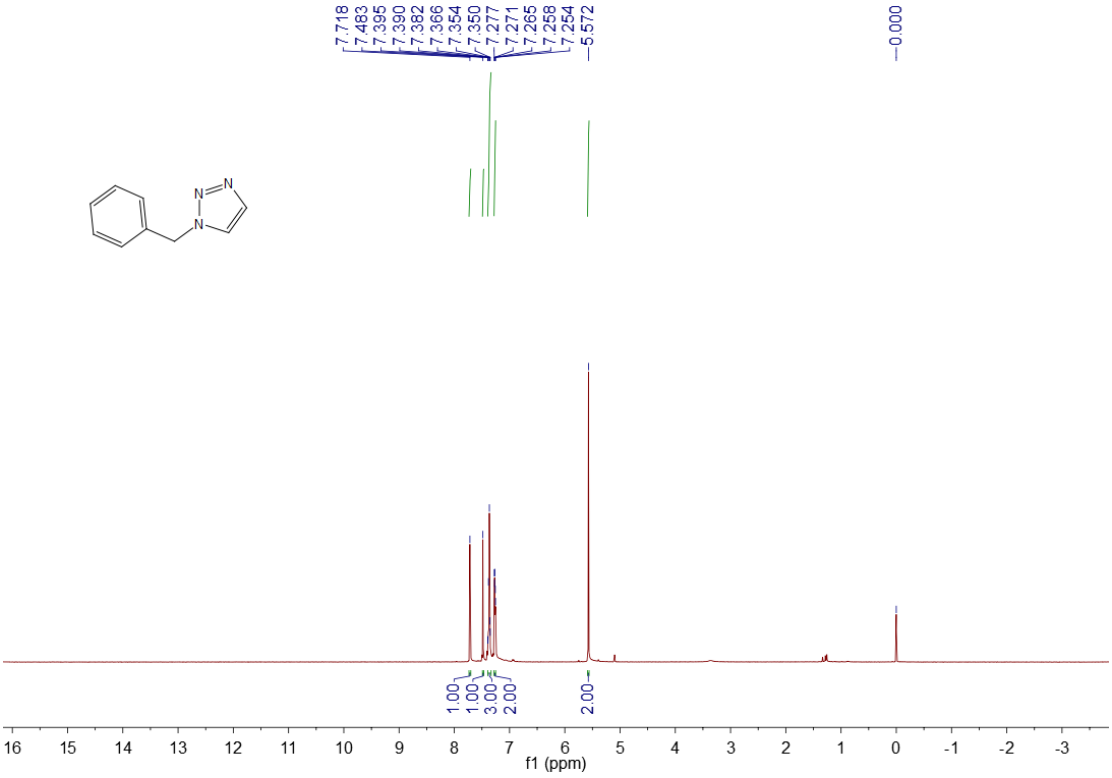
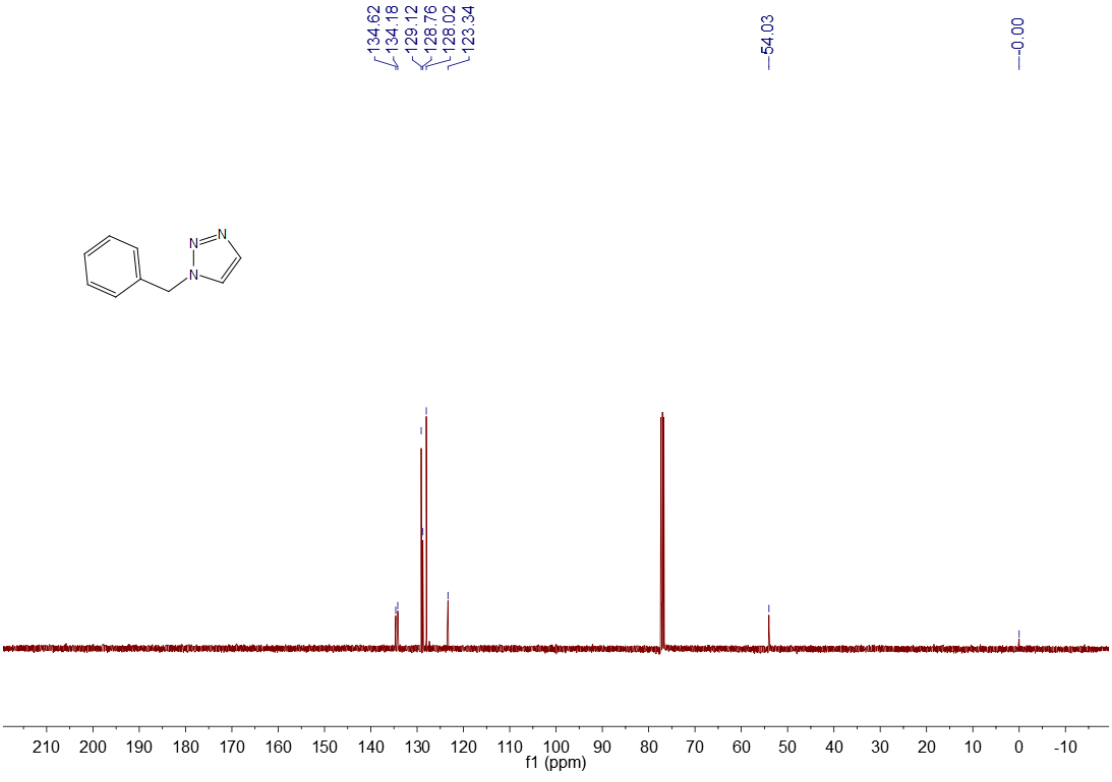


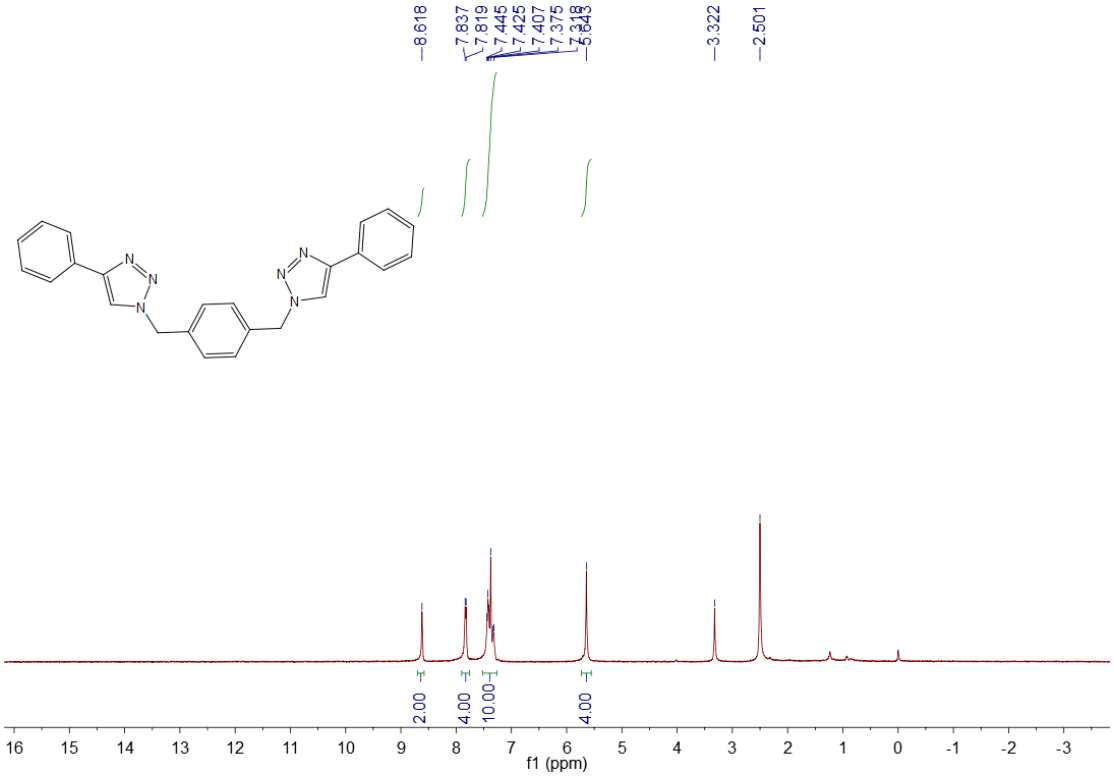


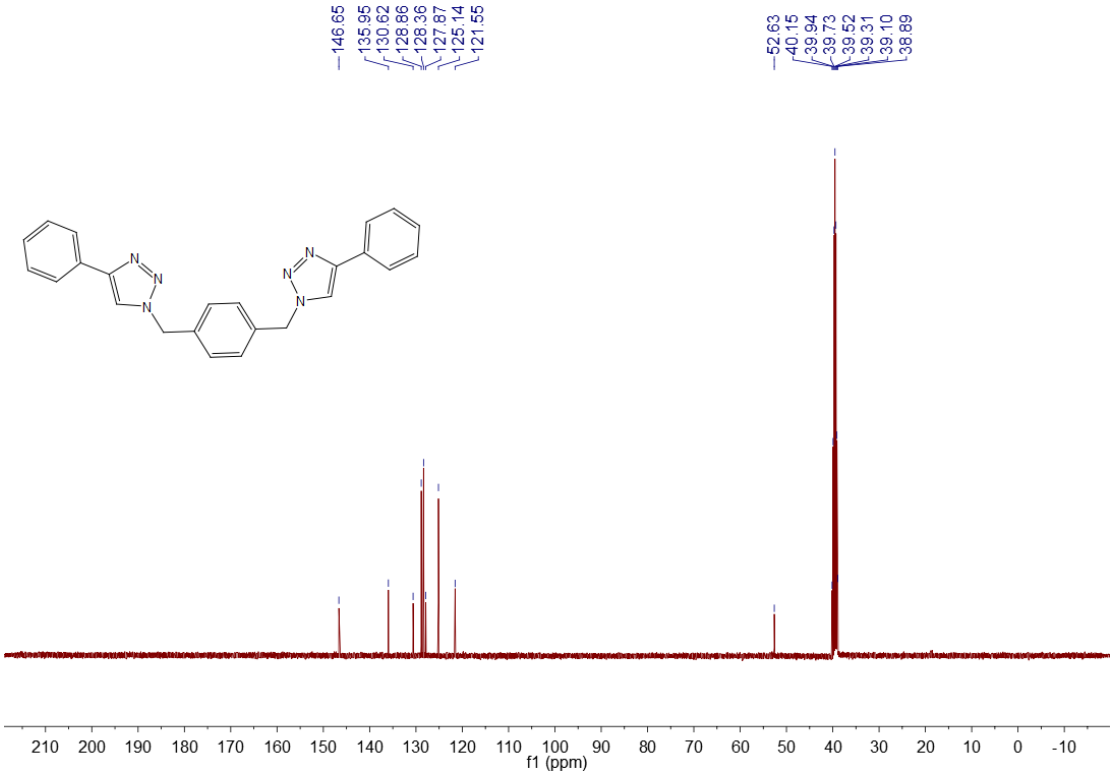
1-Benzyl-1*H*-1,2,3-triazole (7v)





1,4-Bis((4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl)benzene (7w)





1,3,5-Tris((4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl)benzene (7x)