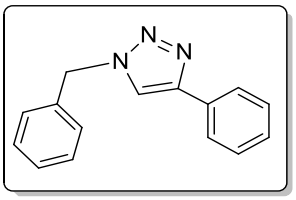
Metathesis Strategy for the Immobilization of Copper (II) onto Carboxymethylcellulose/Fe_3O_4 Nanohybrid Supports: Efficient and Recoverable Magnetic Catalyst for the CuAAC Reaction

Zhuan Zhang, Ping Song, Junbin Zhou, Yang Chen, Bijin Lin, and Yiqun Li *

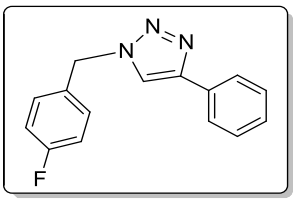
Department of Chemistry, Jinan University, Guangzhou 510632, China

Email: Yiqun Li* - tlyq@jnu.edu.cn

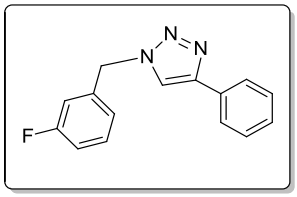
Characterization data for the compounds



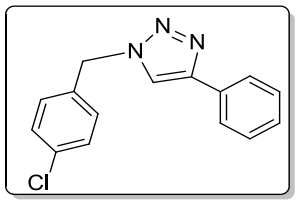
1-Benzyl-4-phenyl-1H-1,2,3-triazole(a). White solid. MP (°C): 128-130. ^{1}H NMR(300MHz, Chloroform-*d*) δ: 7.83 (d, *J*= 7.3 Hz, 2H), 7.70 (s, 1H), 7.46 –7.31 (m, 8H), 5.59 (s, 2H).



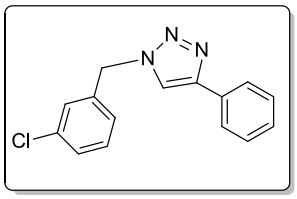
1-(4-Fluorobenzyl)-4-phenyl-1H-1,2,3-triazole(b). White solid. MP (°C): 136-138. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.83 (d, *J* = 7.5 Hz, 2H), 7.72 (s, 1H), 7.43 (t, *J*= 7.3 Hz, 2H), 7.38–7.28 (m, 3H), 7.10 (t, *J* = 8.5Hz, 2H), 5.56 (s, 2H).



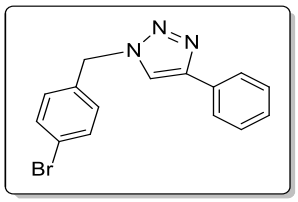
1-(3-Fluorobenzyl)-4-phenyl-1H-1,2,3-triazole(c). White solid. MP (°C): 104-106. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.85 (d, *J*= 7.2 Hz, 2H), 7.78 (s, 1H), 7.48–7.32 (m, 4H), 7.15–7.00 (m, 3H), 5.60 (s, 2H).



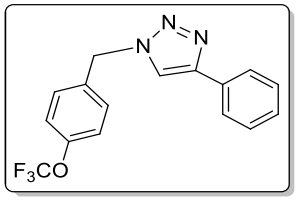
1-(4-Chlorobenzyl)-4-phenyl-1H-1,2,3-triazole(d). Yellow solid. MP (°C): 155-157. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.87–7.78 (m, 2H), 7.72 (s, 1H), 7.48–7.25 (m, 5H), 7.25 (d, *J* = 8.4 Hz, 2H), 5.55 (s, 2H).



1-(3-Chlorobenzyl)-4-phenyl-1H-1,2,3-triazole(e). White solid. MP (°C): 105-107. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.85 (d, *J* = 7.5 Hz, 2H), 7.75 (s, 1H), 7.45 (d, *J* = 7.4 Hz, 1H), 7.45–7.26 (m, 5H), 7.21 (d, *J*=6.5 Hz, 1H), 5.58 (s, 2H).

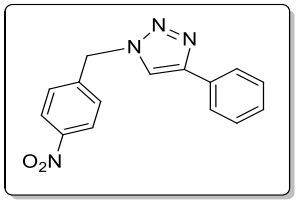


1-(4-Bromobenzyl)-4-phenyl-1H-1,2,3-triazole(f). White solid. MP (°C): 168-170. ^{1}H NMR (300MHz, Chloroform-*d*) δ: 7.83 (d, *J*= 7.5 Hz, 2H), 7.69 (s, 1H), 7.55 (d, *J*=8.0 Hz, 2H), 7.39 (dt, *J*=25.4, 7.5 Hz, 3H), 7.21 (d, *J*= 8.0 Hz, 2H), 5.56 (s, 2H).

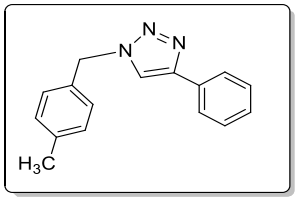


1-(4-Trifluoromethoxybenzyl)-4-phenyl-1H-1,2,3-triazole(g). White solid. MP (°C): 133-136. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.88 –7.79 (m, 2H), 7.74 (s, 1H), 7.43 (t, *J* =7.2 Hz, 2H),7.39–7.31 (m, 3H), 7.25 (d, *J* = 8.2 Hz, 2H), 5.59 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 149.44, 149.42, 133.45,

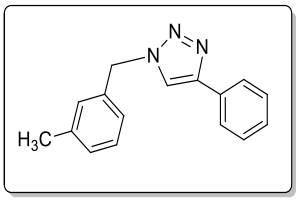
130.37, 129.50, 128.86, 128.31, 128.23, 125.72, 122.09, 121.58, 121.56, 118.67, 115.25, 53.35. IR (KBr) ν: 3130, 2952, 1510, 1462, 1301, 1161, 1073, 766, 695 cm^{-1}. HRMS (ESI) m/z: calcd for $C_{16}H_{12}F_3N_3O$ $(M + Na)^+$ 342.0825, Found 342.0833.



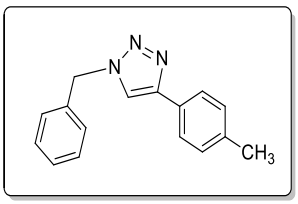
1-(4-Nitrobenzyl)-4 -phenyl-1H-1,2,3-triazole(h). White solid. MP (°C): 156-158. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 8.23 (d, *J*= 8.5 Hz, 2H), 7.84–7.75 (m, 3H), 7.47–7.31 (m, 5H), 5.70 (s, 2H).



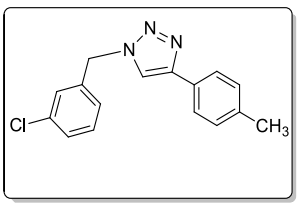
1-(4-Methylbenzyl)-4-phenyl-1H-1,2,3-triazole(i). White solid. MP (°C): 93-95. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.83 (d, *J*= 7.5 Hz, 2H), 7.72 (s, 1H), 7.48–7.30 (m, 4H), 7.23 (s, 3H), 5.55 (s, 2H), 2.39 (s, 3H).



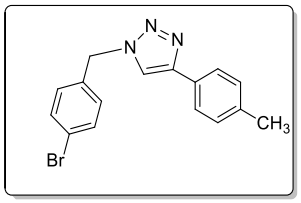
1-(3-Methylbenzyl)-4-phenyl-1H-1,2,3-triazole(j). White solid. MP (°C): 94-97. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.84 (d, *J*= 7.6 Hz, 2H), 7.71 (s, 1H), 7.49–7.25 (m, 4H), 7.25–7.10 (m, 3H), 5.56 (s, 2H), 2.38 (s, 3H).



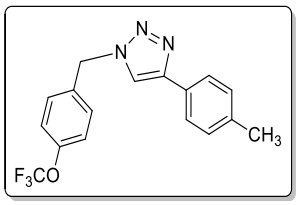
1-benzyl-4-p-tolyl-1H-1,2,3-triazole(k). White solid. MP (°C): 152-154. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.72 (d, *J*=7.7 Hz, 2H), 7.66 (s, 1H), 7.44–7.29 (m, 5H), 7.23 (d, *J*=7.8 Hz, 2H), 5.58 (s, 2H), 2.39 (s, 3H).



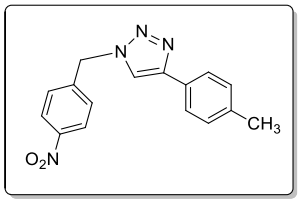
1-(3-Chlorobenzyl)-4-p-tolyl-1H-1,2,3-triazole(l). Yellow solid. MP (°C): 138-140. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.71 (d, *J*= 7.9 Hz, 3H), 7.34–7.28 (m, 3H), 7.24–7.15 (m, 3H), 5.54 (s, 2H), 2.37 (s, 3H).



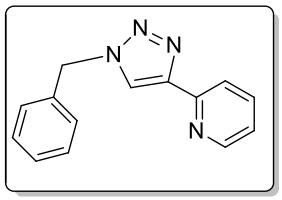
1-(4-Bromobenzyl)-4-p-tolyl-1H-1,2,3-triazole(m). White solid. MP (°C): 197-200. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.69 (t, *J* = 9.8 Hz, 3H), 7.57–7.47 (m, 2H), 7.20 (t, *J* = 8.6 Hz, 4H), 5.53 (s, 2H), 2.37 (s, 3H).



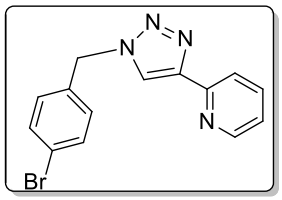
1-(4-Trifluoromethoxybenzyl)-4-p-tolyl-1H-1,2,3-triazole(n). White solid. MP (°C): 186-189. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 7.76 – 7.69 (m, 3H), 7.33 (d, *J* = 8.6 Hz, 2H), 7.24 (dd, *J* = 8.3, 6.2 Hz, 4H), 5.57 (s, 2H), 2.37 (s, 3H). ^{13}C NMR(75 MHz, $CDCl_3$) δ: 149.39, 149.37, 148.50, 138.18, 133.55, 129.53, 129.49, 127.56, 125.62, 122.09, 121.55, 121.53, 121.52, 121.51, 119.25, 53.27, 21.26. IR (KBr) ν: 3135, 2951, 1509, 1455, 1438, 1306, 1107, 1047, 813, 766, 612 cm^{-1}. HRMS (ESI) m/z: calcd for $C_{16}H_{15}F_3N_3O$ $(M+H)^+$ 334.1162, Found 334.1164.



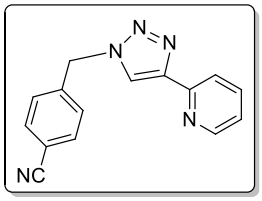
1-(4-Nitrobenzyl)-4-p-tolyl-1H-1,2,3-triazole(o). Yellow solid. MP (°C): 145-147. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 8.25 (d, *J*= 8.5 Hz, 2H), 7.73 (d, *J*= 8.0 Hz, 3H), 7.46 (d, *J*= 8.4 Hz, 2H), 7.29–7.22 (m, 2H), 5.71 (s, 2H), 2.40 (s, 3H).



2-(1-benzyl-1H-1,2,3-triazol-4-yl)pyridine(p). White solid. MP (°C): 120-123 101-104. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 8.56 (s, 1H), 8.30–7.97 (m, 2H), 7.81 (d, *J*=8.1 Hz, 1H), 7.31 (d, *J*=44.7 Hz, 6H), 5.61 (s, 2H).



2-(1-(4-bromobenzyl)-1H-1,2,3-triazol-4-yl)pyridine(q). White solid. MP (°C): 123-124. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 8.53 (d, *J*=4.6 Hz, 1H), 8.17 (dt, *J*=8.0, 1.1Hz, 1H), 8.06 (s, 1H), 7.77 (td, *J*=7.8, 1.8Hz, 1H), 7.56–7.45 (m, 2H), 7.25–7.16 (m, 3H), 5.53 (s, 2H).



4-((4-(pyridin-2-yl)-1H-1,2,3-triazol-1-yl)methyl)benzonitrile(r). White solid. MP (°C): 167-169. ^{1}H NMR (300 MHz, Chloroform-*d*) δ: 8.57 (s, 1H), 8.31–8.02 (m, 2H), 7.81 (t, *J*=7.8 Hz, 1H), 7.69 (d, *J*= 7.8 Hz, 2H), 7.42 (d, *J*=7.8 Hz, 2H), 7.28 (d, *J*=8.7 Hz, 1H), 5.68 (s, 2H).

Reference

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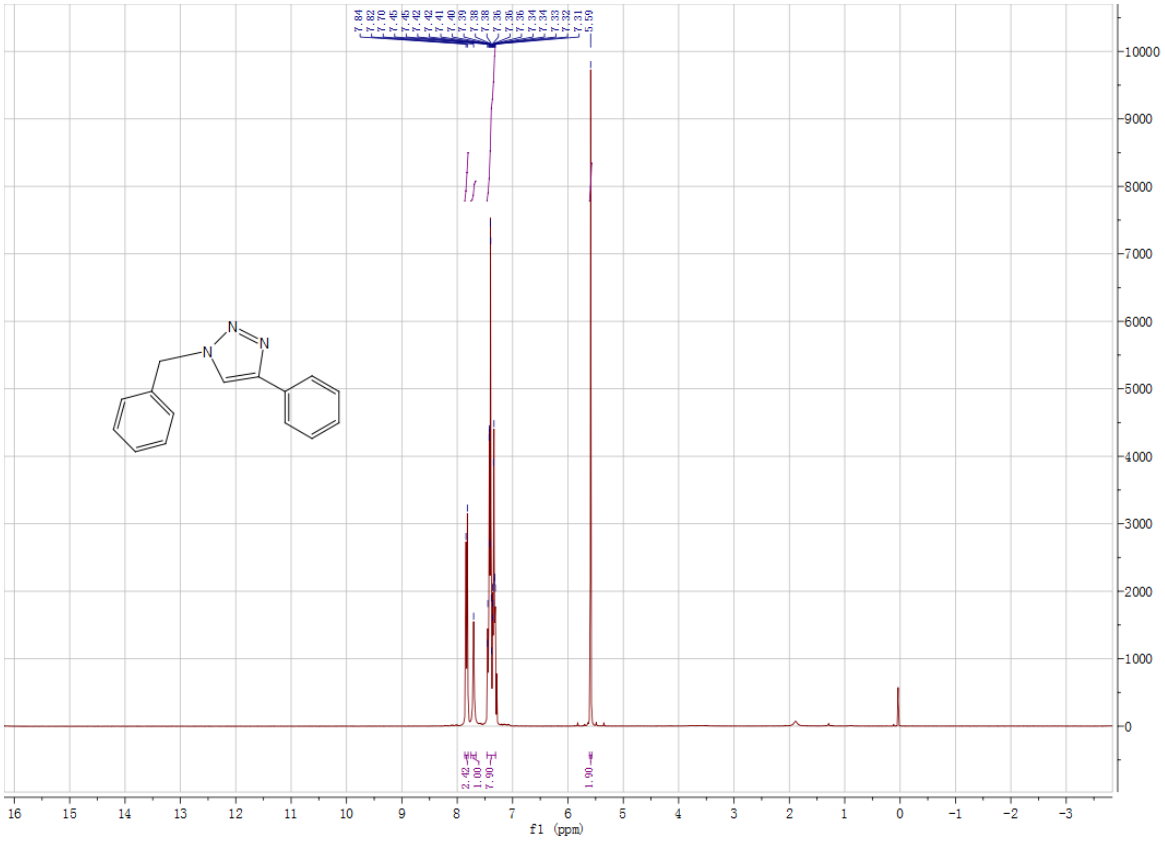
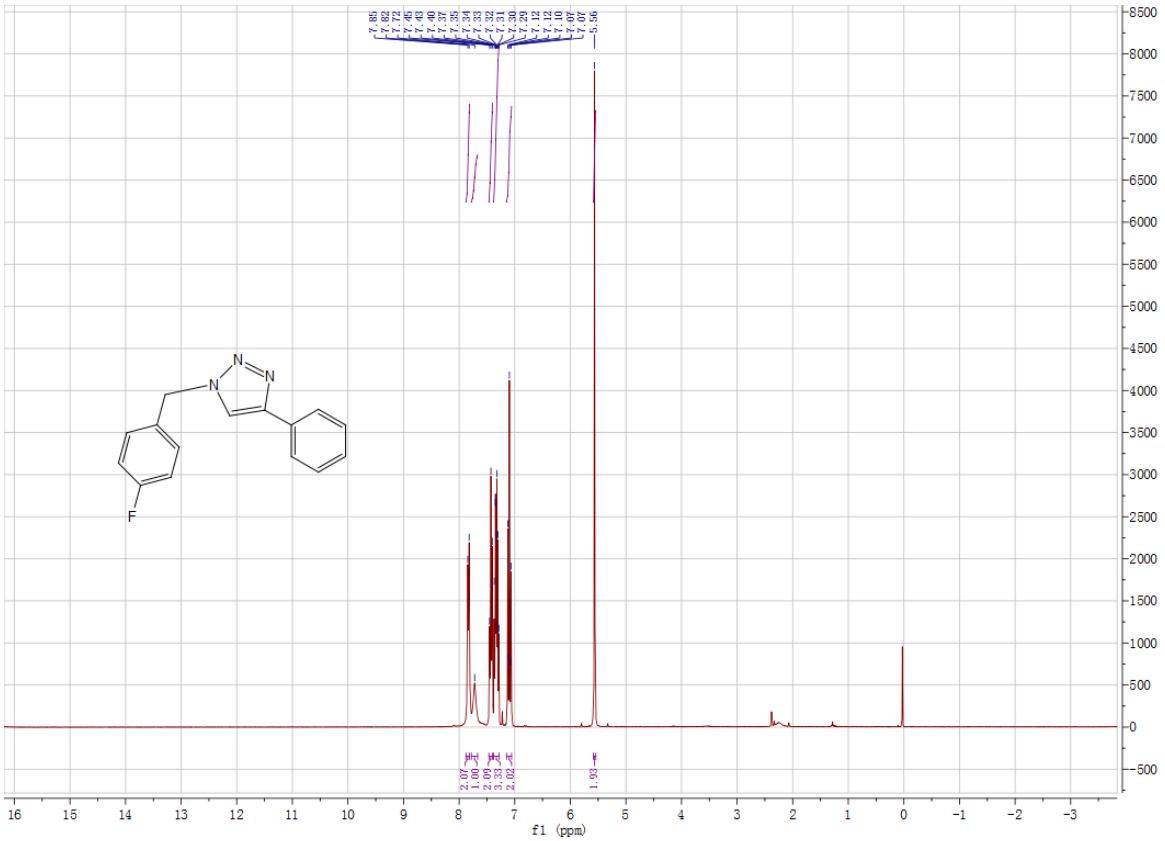
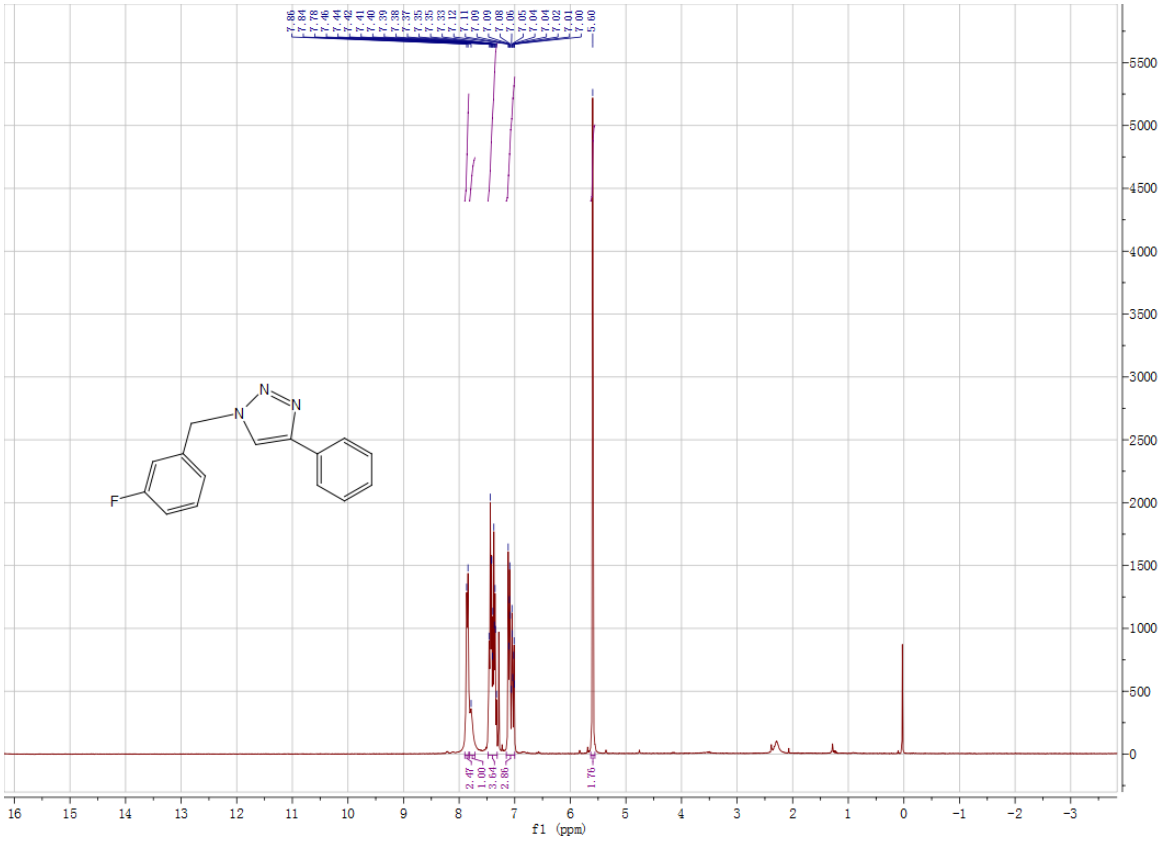
^{1}H NMR and ^{13}C NMR spectra of the products



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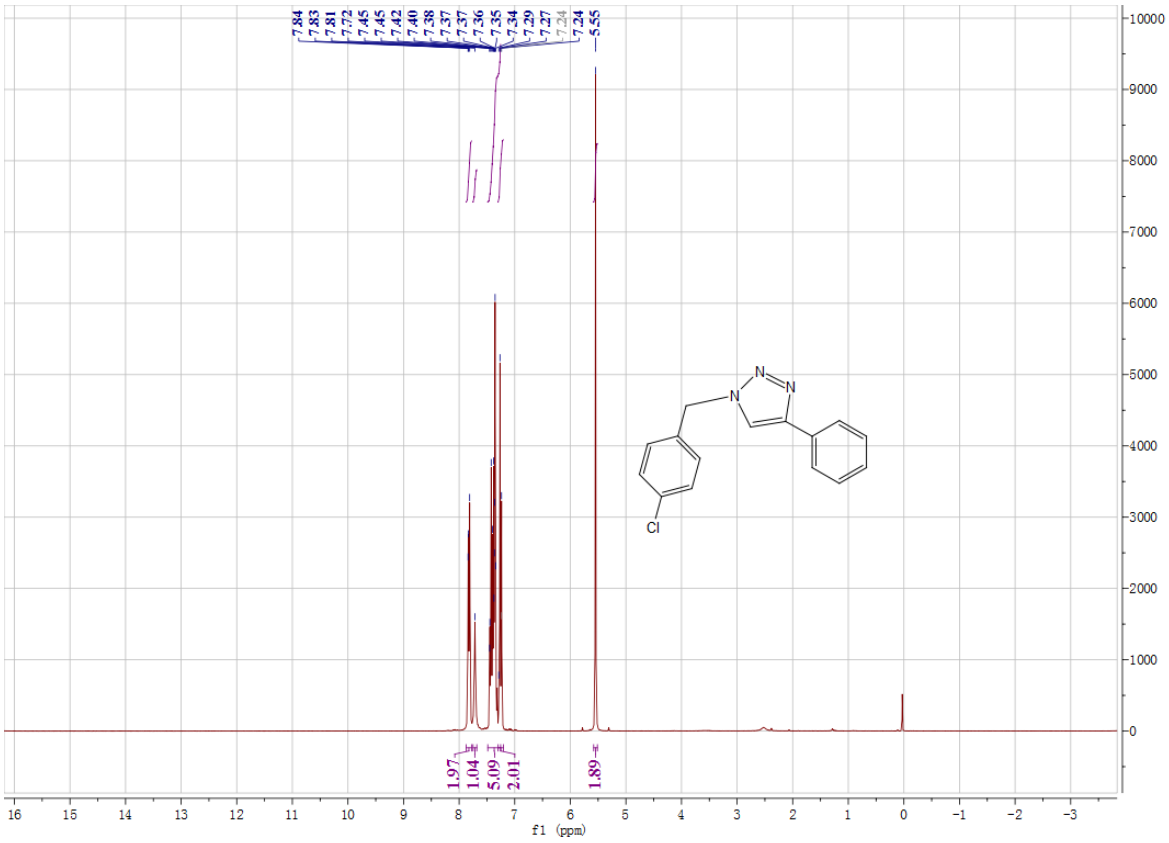
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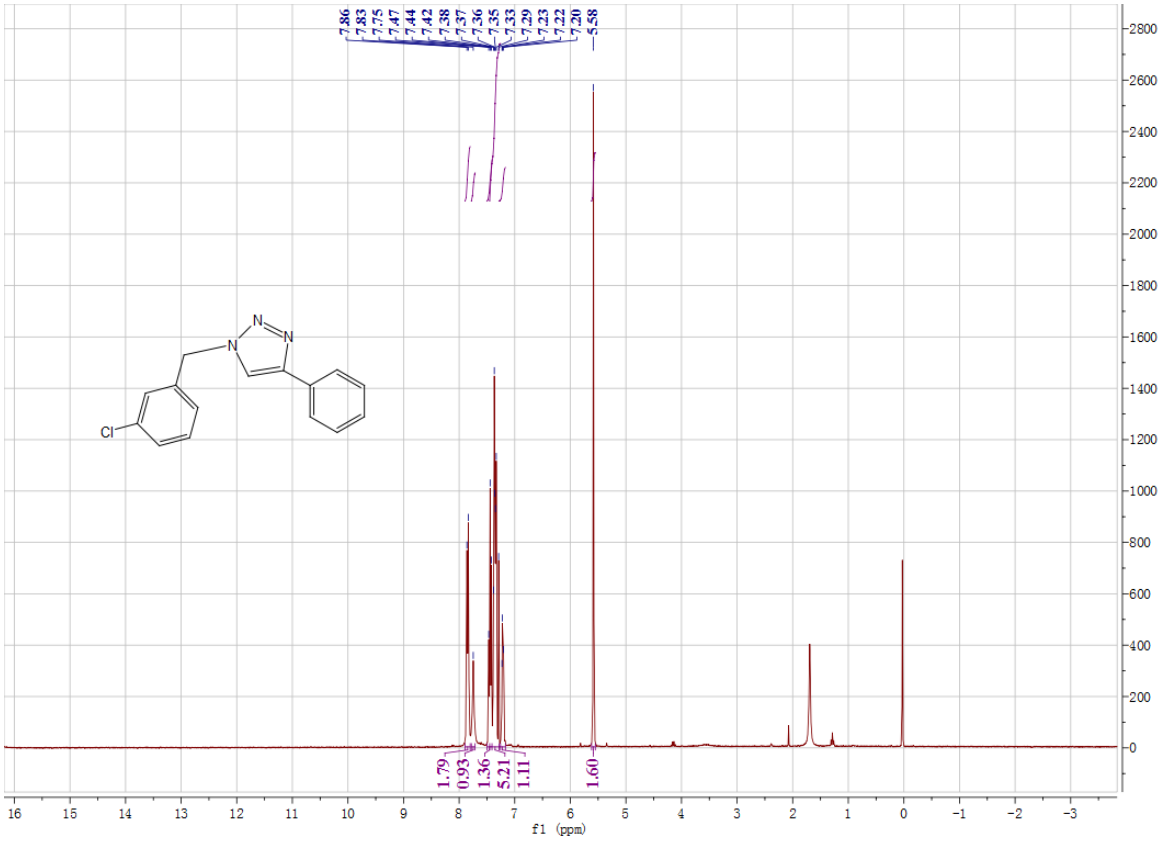
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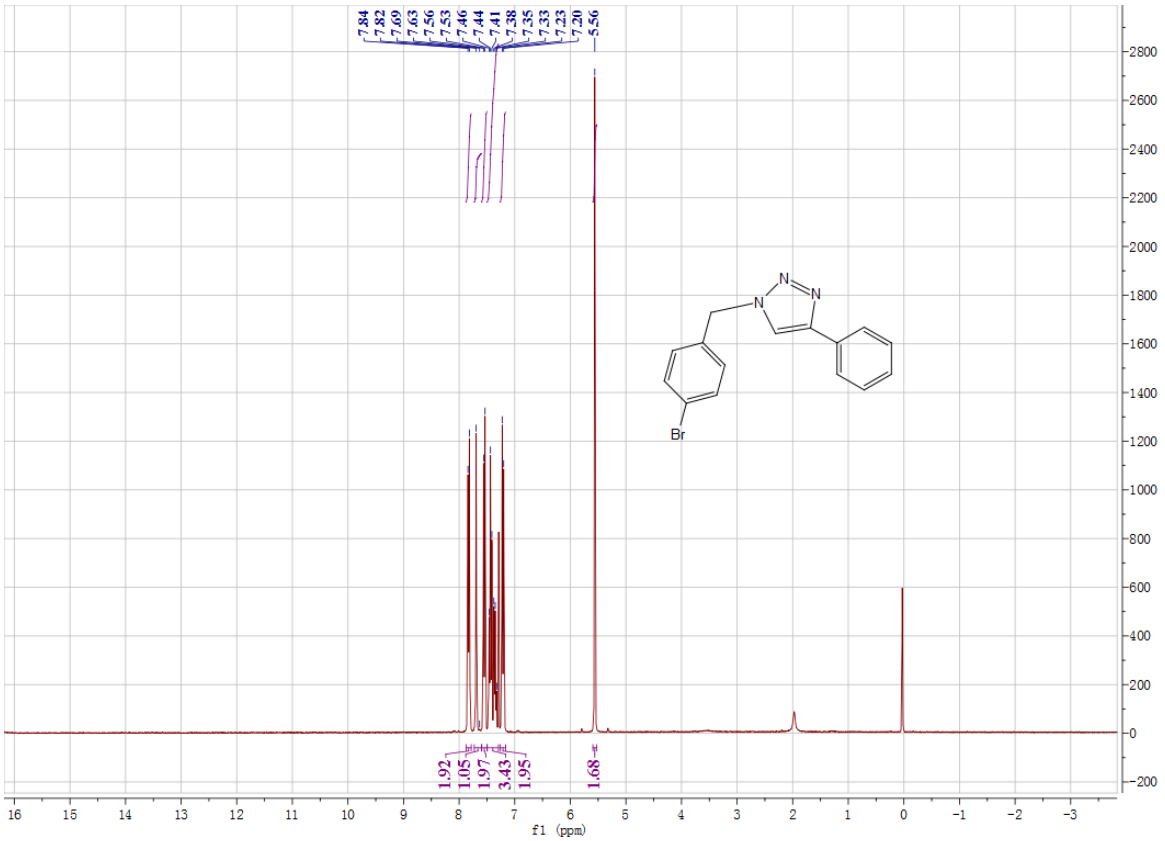
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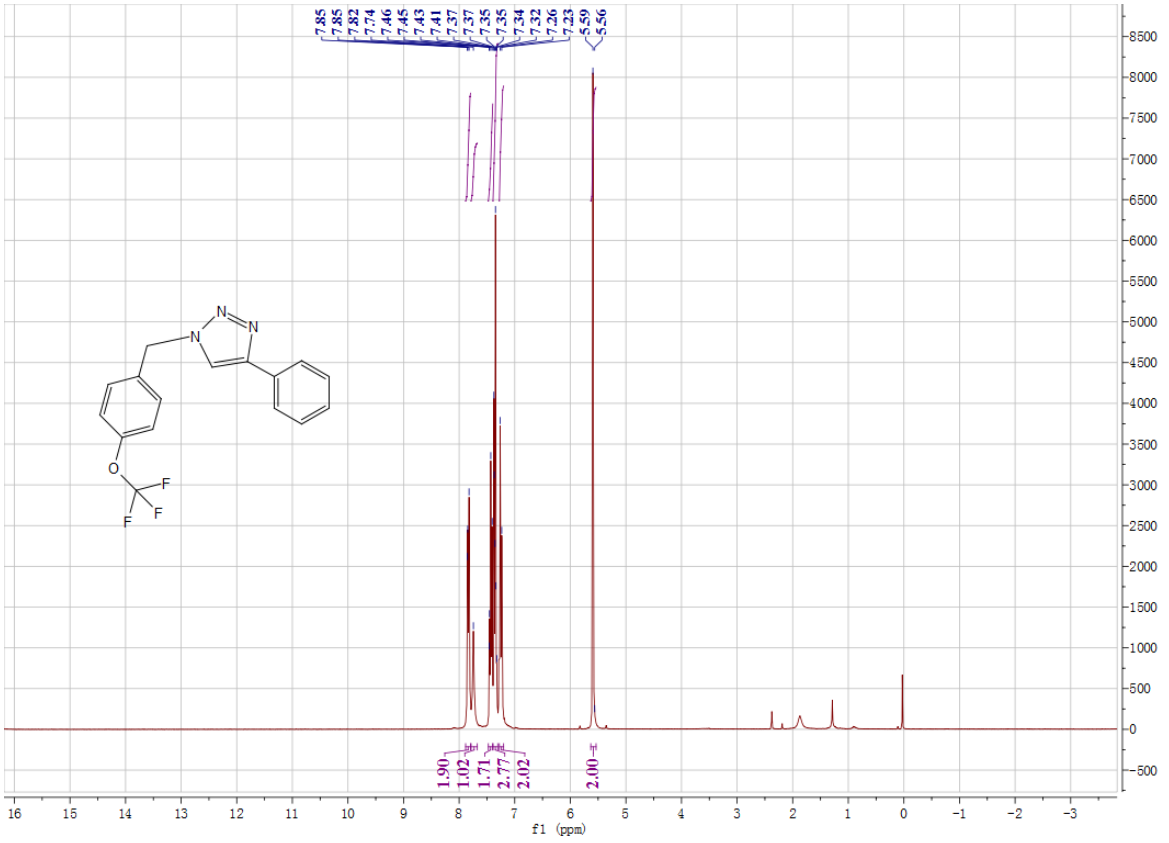
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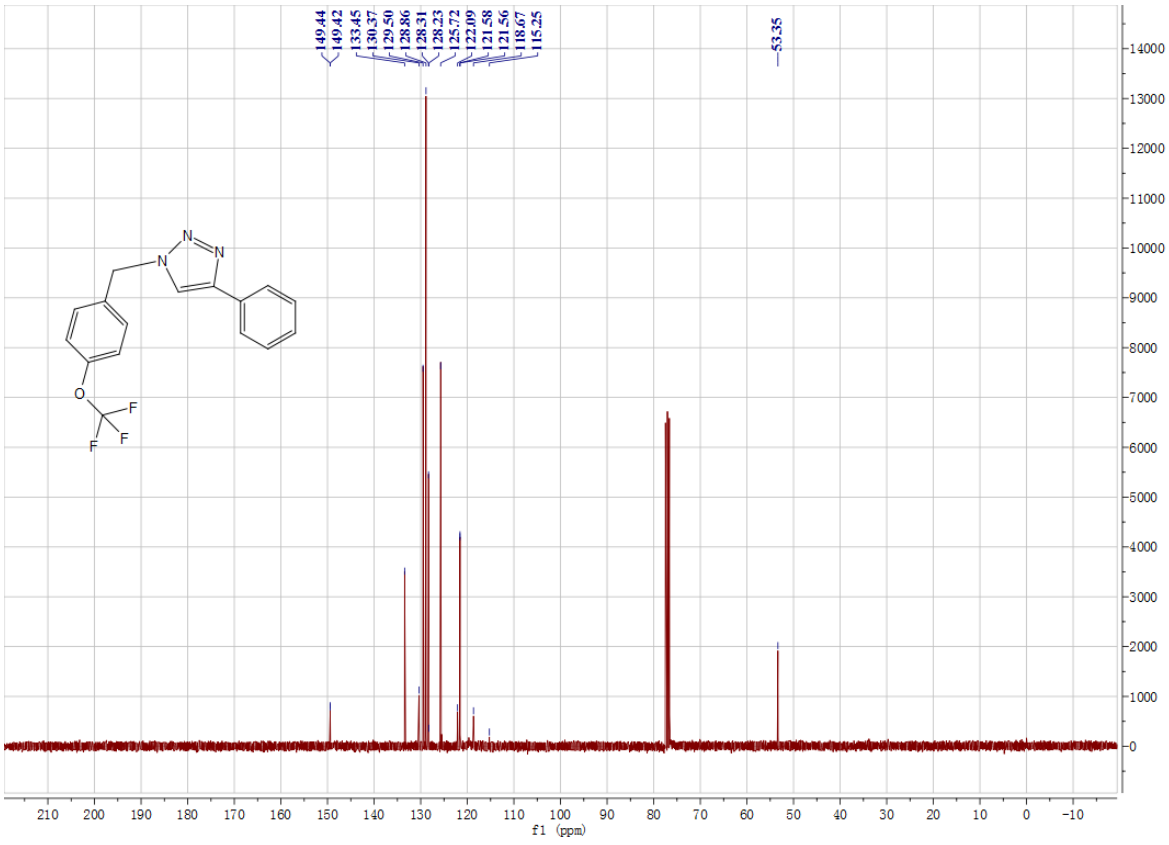
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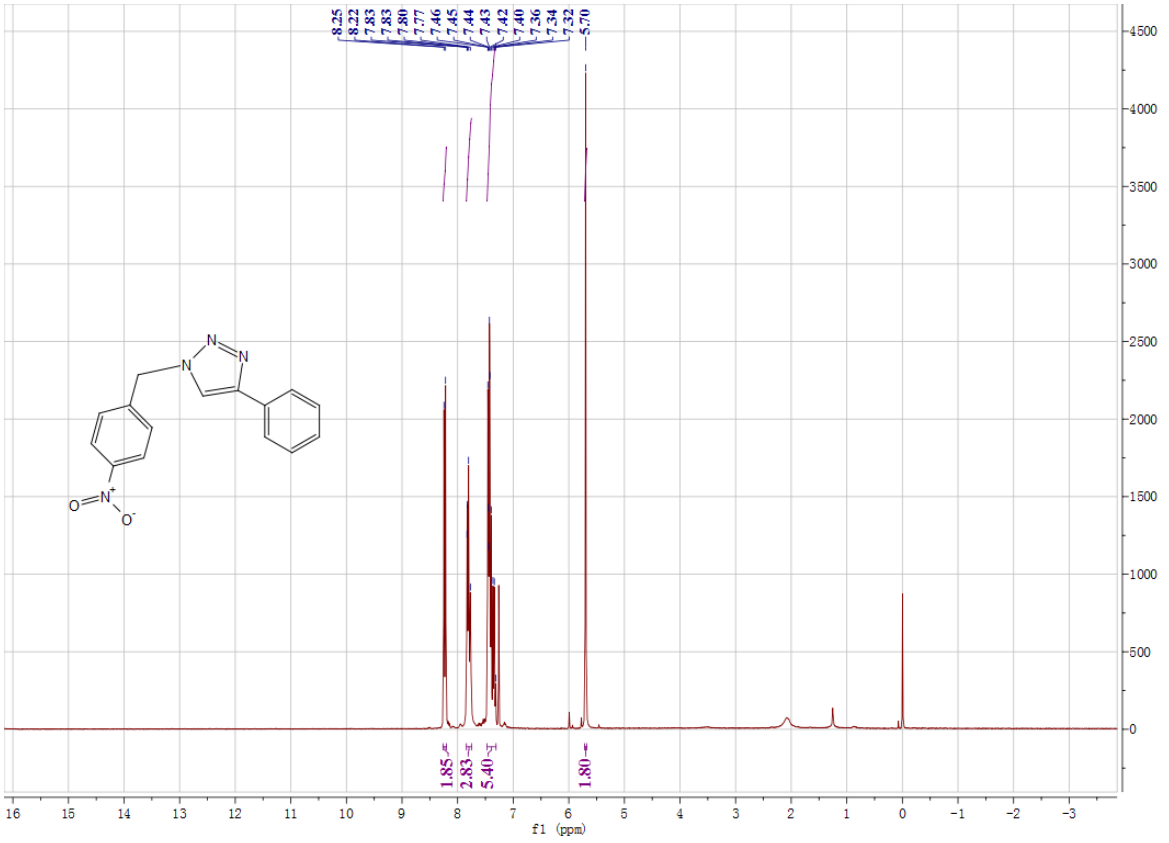
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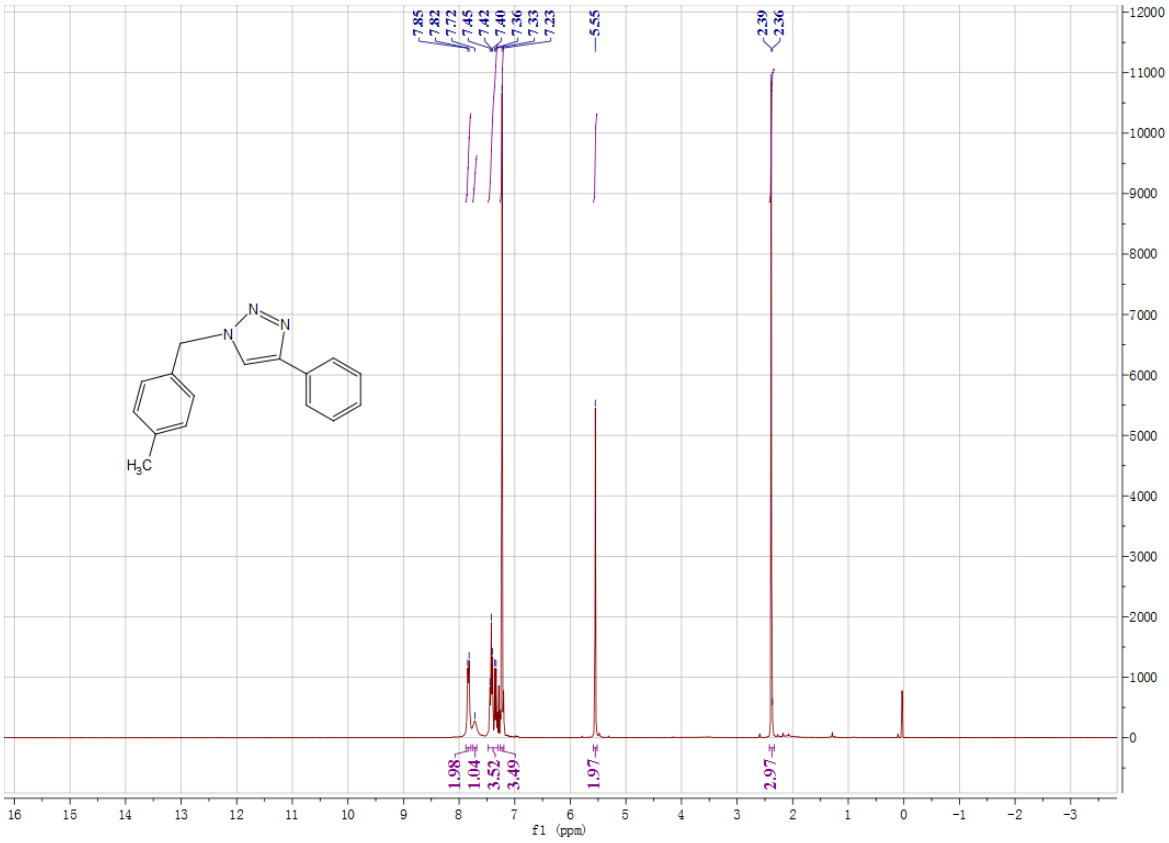
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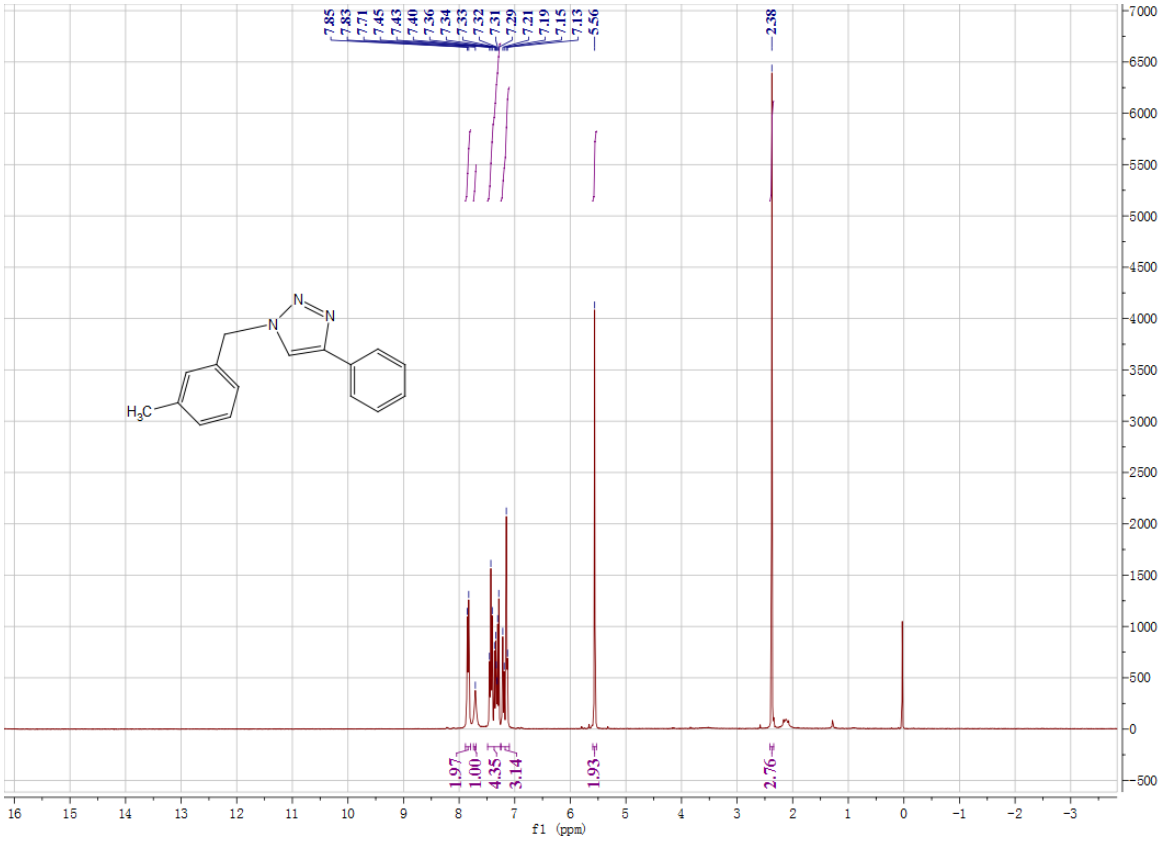
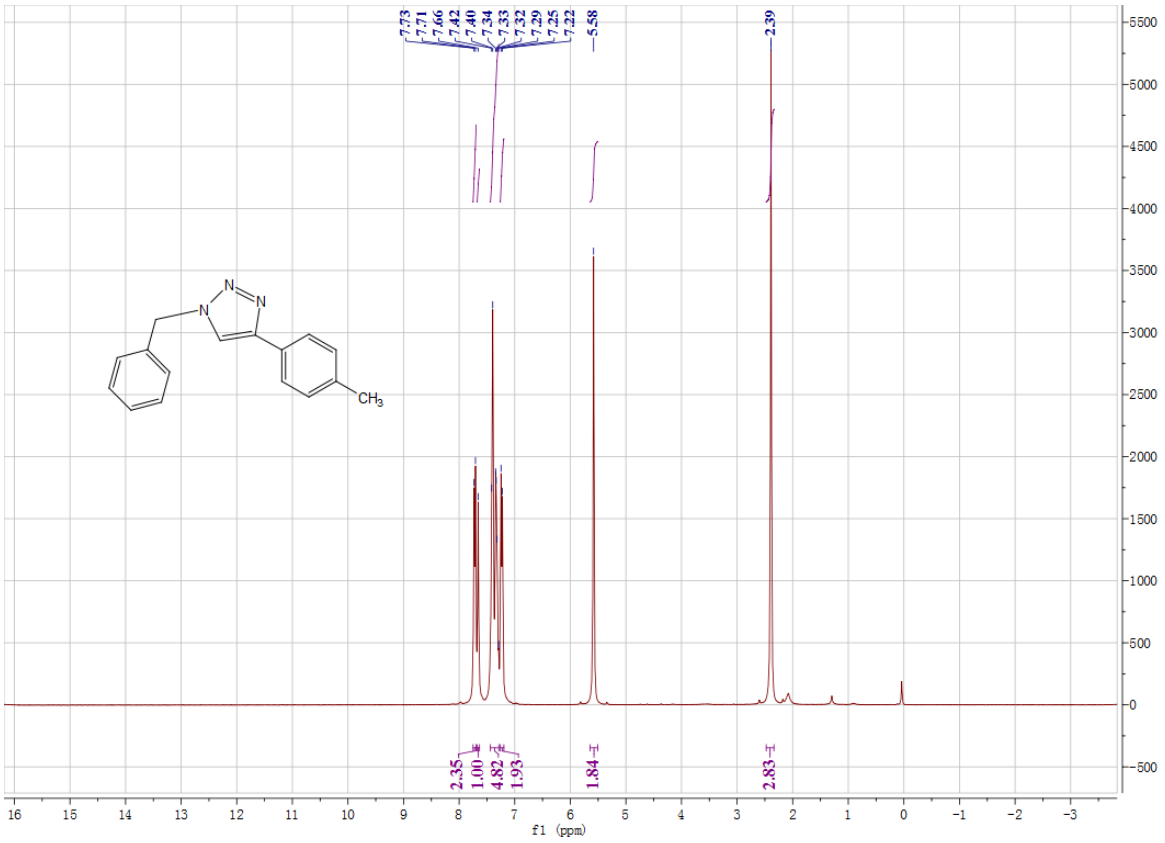
2.97
f1 (ppm)


7.85
7.83
7.71
7.45
7.43
7.40
7.36
7.34
7.33
7.32
7.31
7.29
7.21
7.19
7.15
7.13
5.56
2.38
H3C
1.97
1.00
4.35
3.14
1.93
2.76
f1 (ppm)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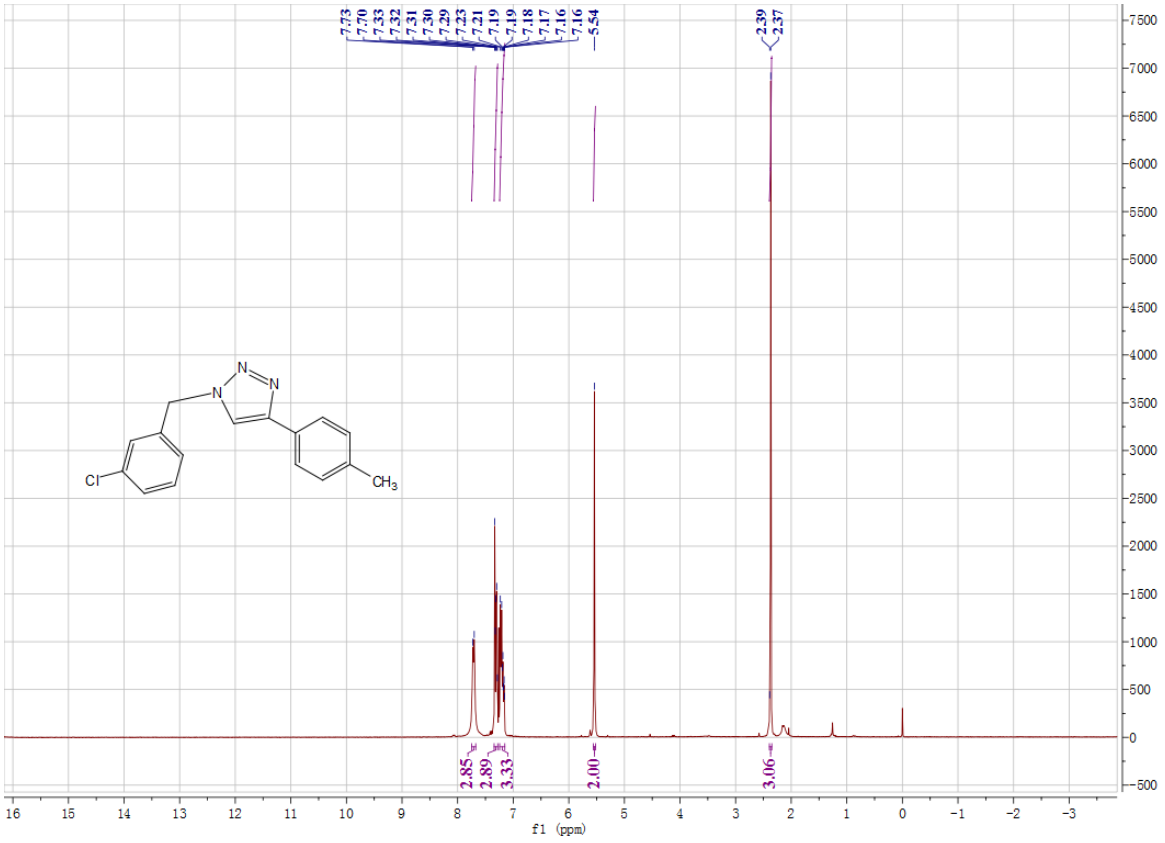
7.71
7.66
7.42
7.40
7.34
7.33
7.32
7.29
7.25
7.22
5.58
2.39
CH3
2.35
1.00
4.82
1.93
1.84
2.83
f1 (ppm)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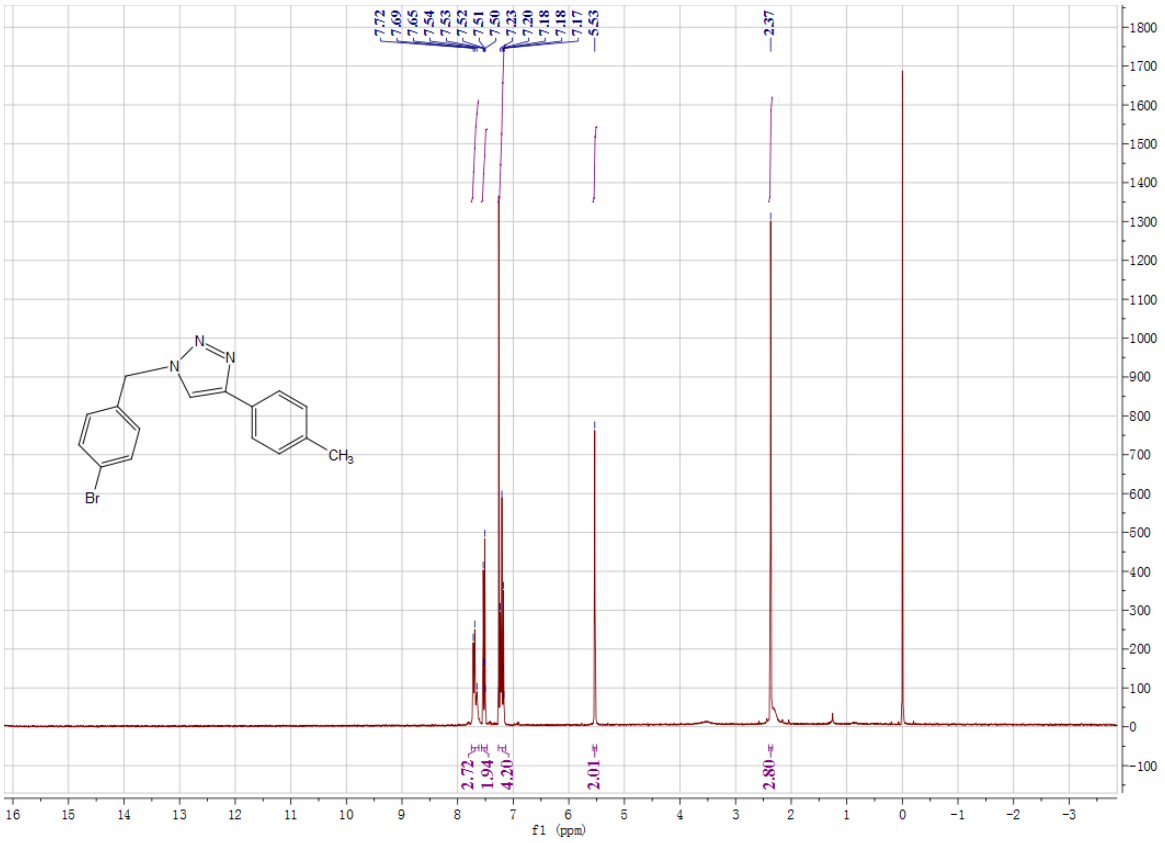
7.70
7.33
7.32
7.31
7.30
7.29
7.23
7.21
7.19
7.19
7.18
7.17
7.16
7.16
5.54
2.39
2.37
Cl
CH3
2.85
2.89
3.33
2.00
3.06
f1 (ppm)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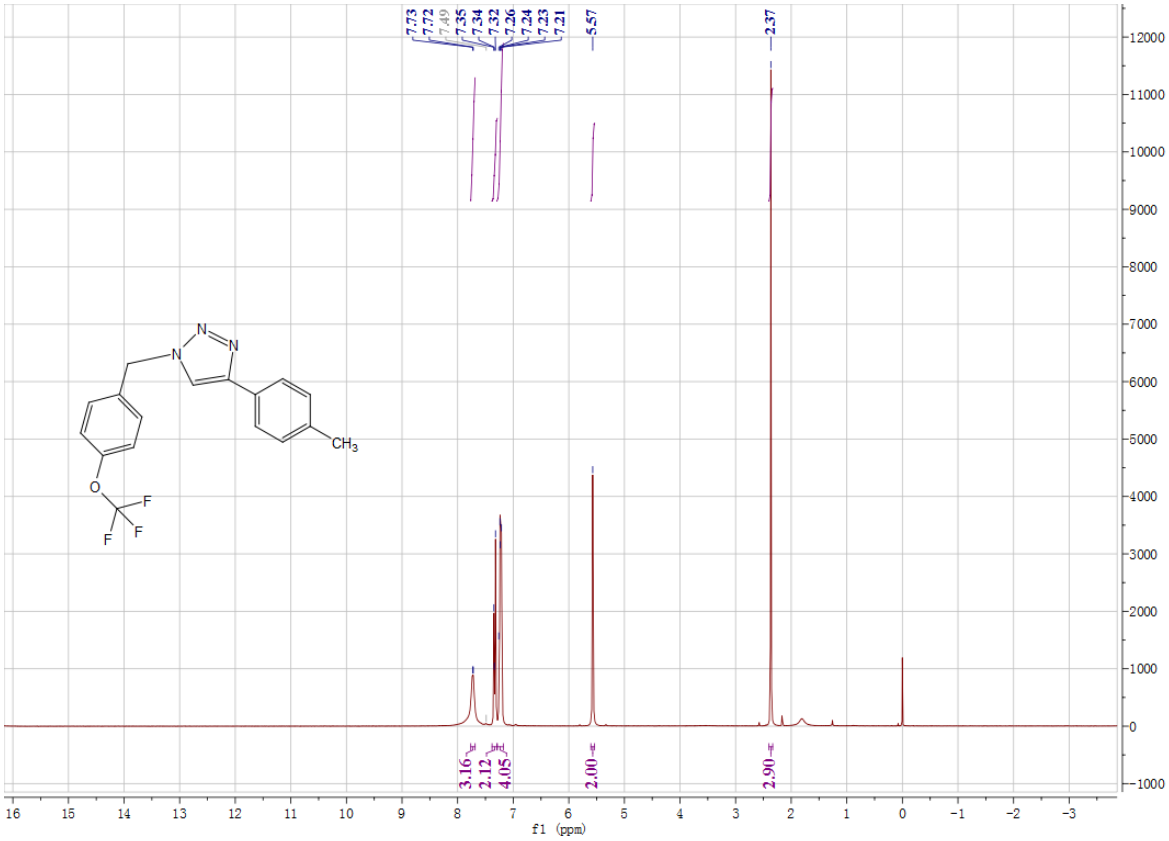
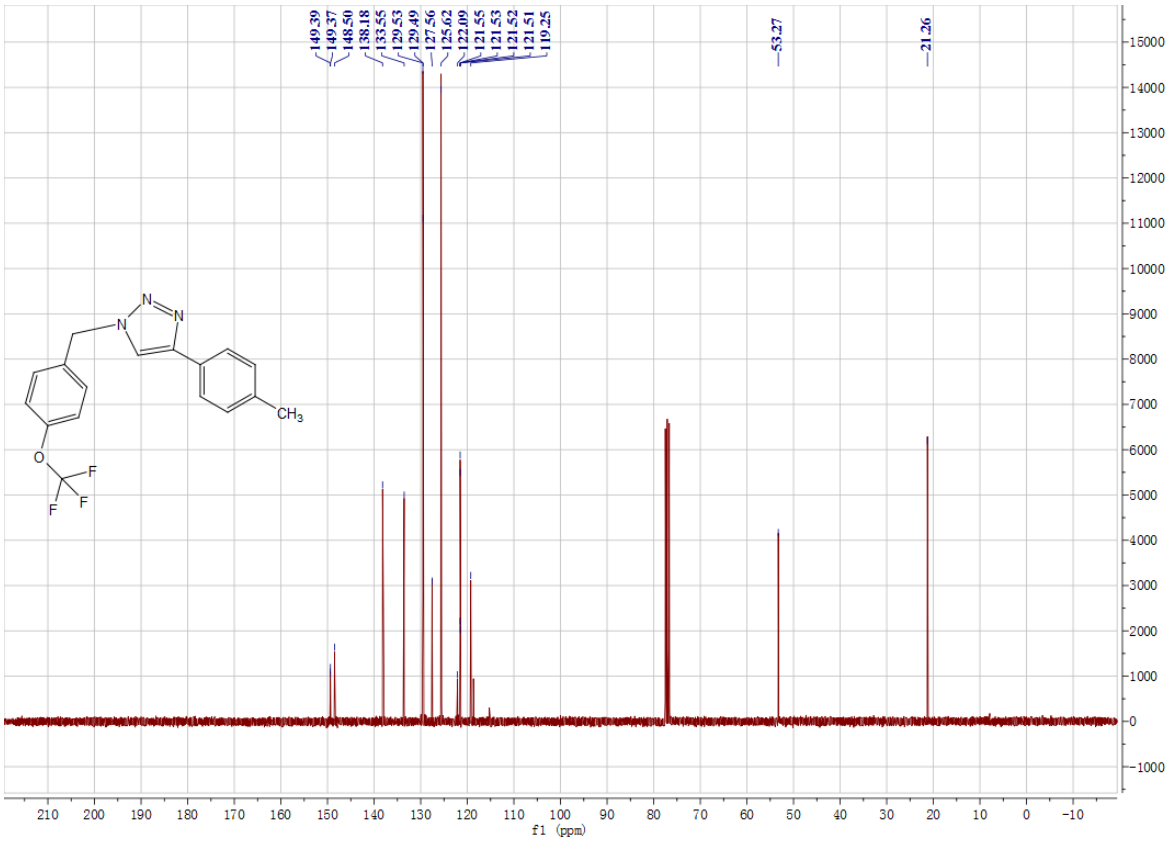
7.69
7.65
7.54
7.53
7.52
7.51
7.50
7.23
7.20
7.18
7.18
7.17
5.53
2.37
Br
CH3
2.72
1.94
4.20
2.01
2.80
f1 (ppm)


7.73
7.72
7.49
7.35
7.34
7.32
7.26
7.24
7.23
7.21
5.57
2.37
3.16
2.12
4.05
2.00
2.90
f1 (ppm)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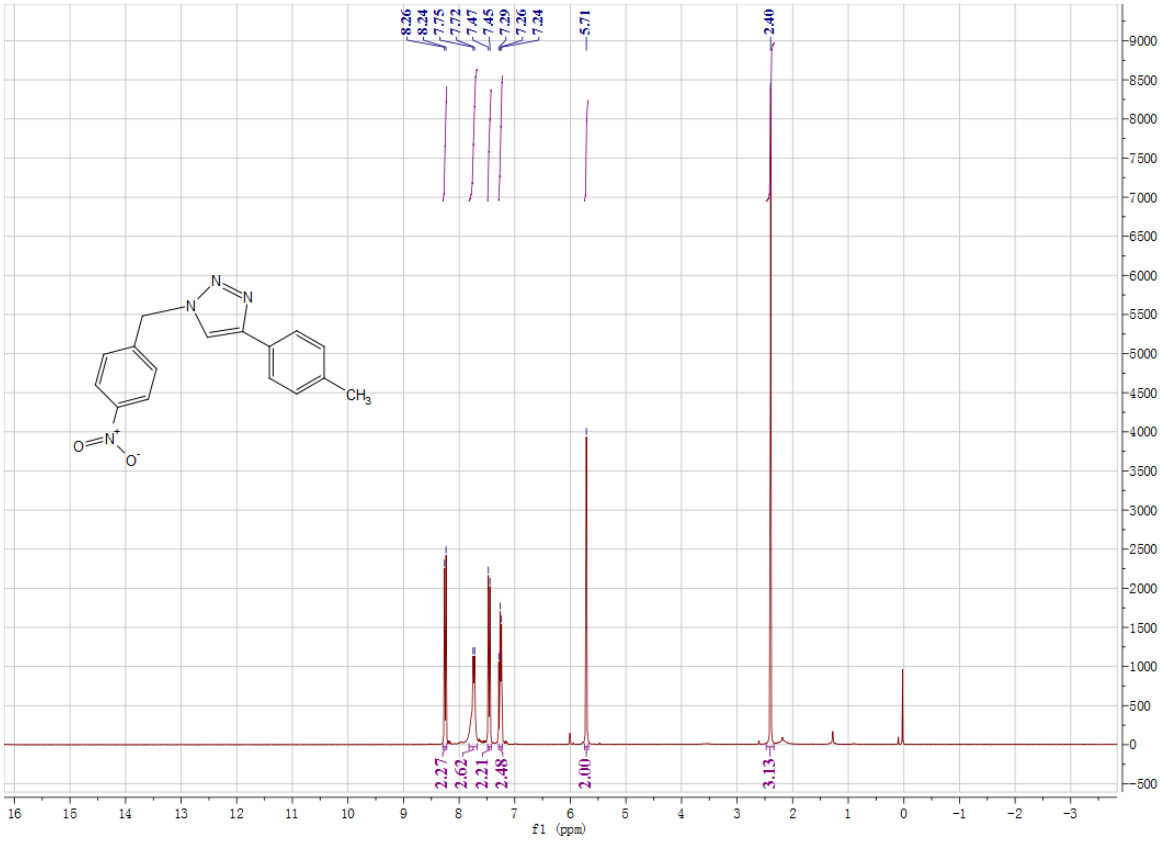
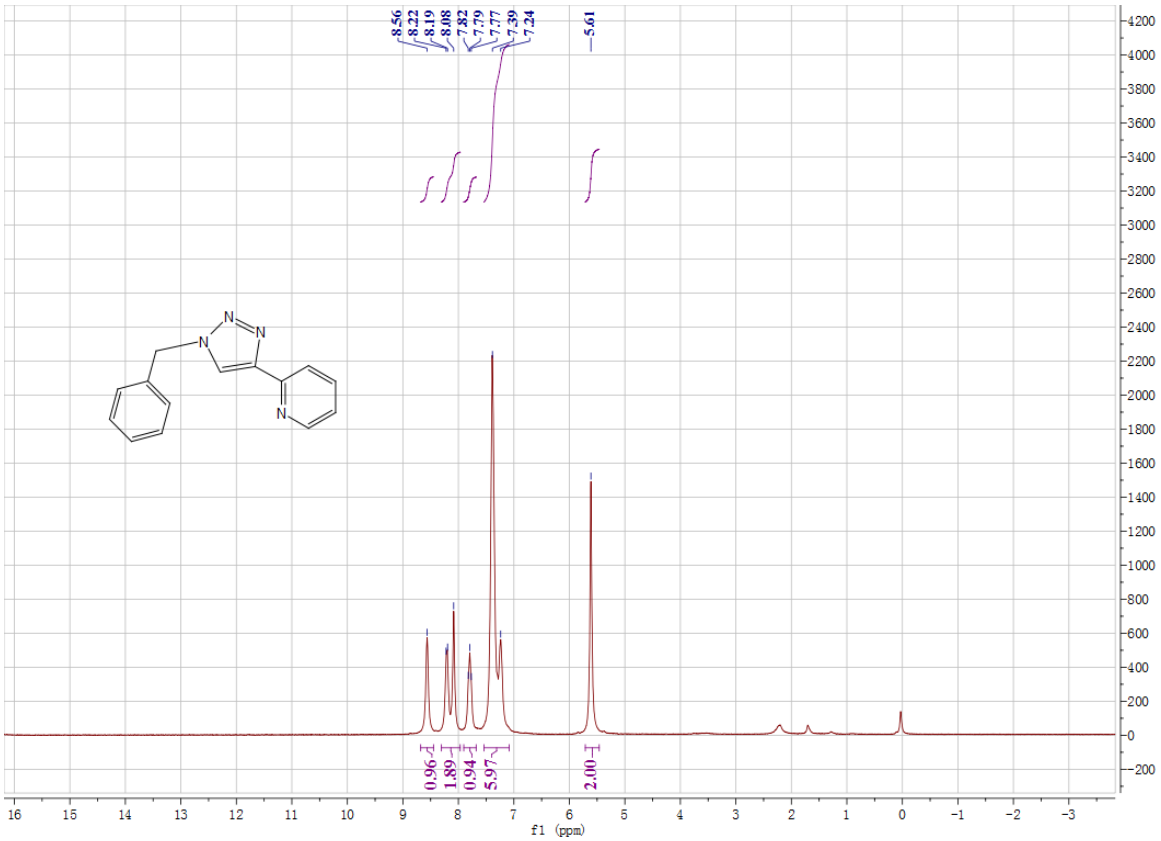
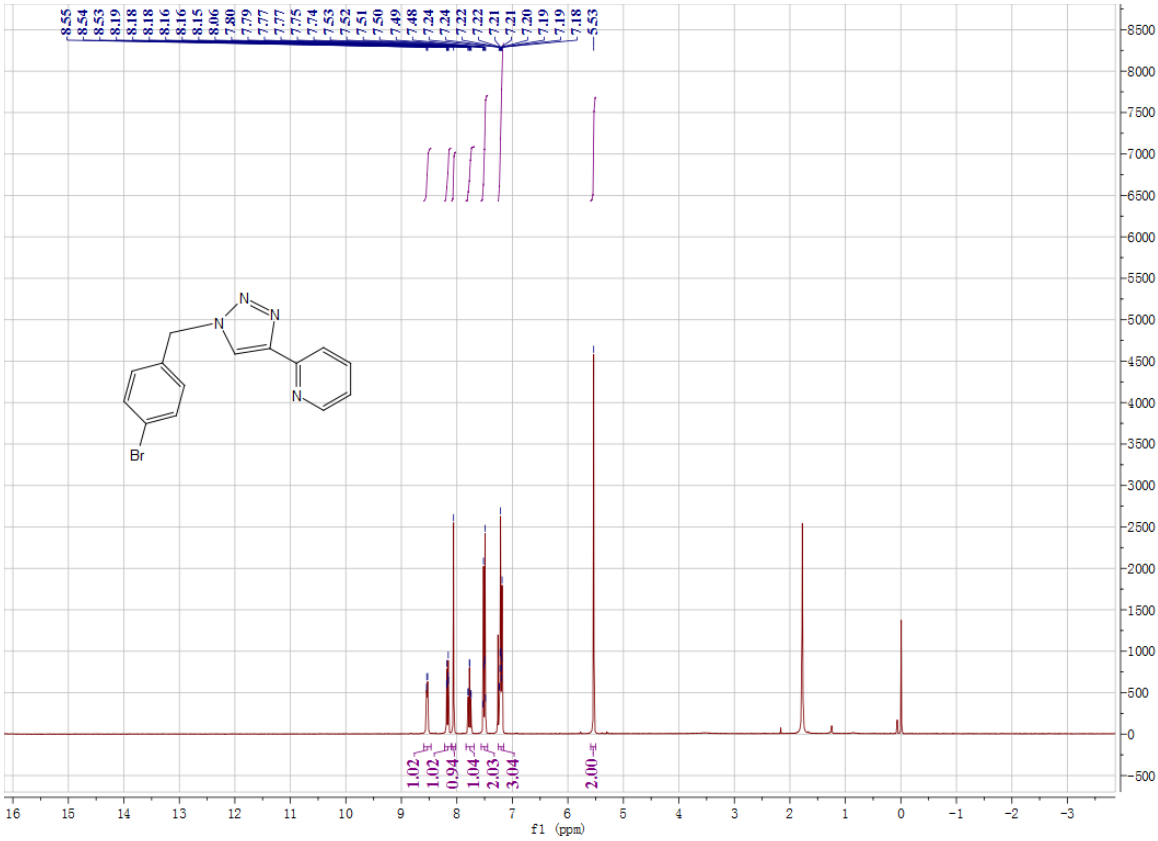
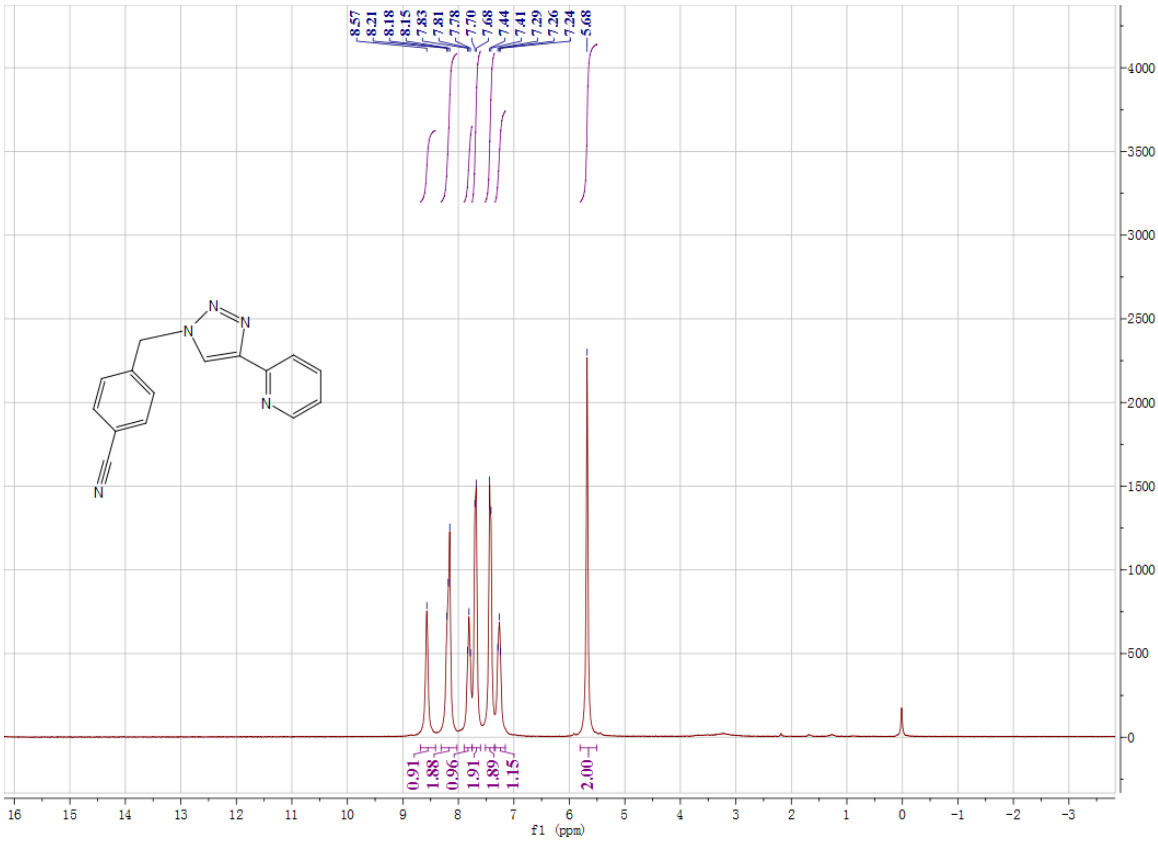
149.37
148.50
138.18
133.55
129.53
129.49
127.56
125.62
122.09
121.55
121.53
121.52
121.51
119.25
53.27
21.26
f1 (ppm)


8.26
8.24
7.75
7.72
7.47
7.45
7.29
7.26
7.24
5.71
2.40
2.27
2.62
2.21
2.48
2.00
3.13
f1 (ppm)


8.56
8.22
8.19
8.08
7.82
7.79
7.77
7.39
7.24
5.61
0.96
1.89
0.94
5.97
2.00
f1 (ppm)


8.55
8.54
8.53
8.19
8.19
8.18
8.18
8.16
8.16
8.15
8.06
7.80
7.79
7.77
7.77
7.75
7.74
7.53
7.52
7.51
7.50
7.49
7.48
7.24
7.24
7.22
7.22
7.21
7.21
7.20
7.19
7.19
7.18
5.53
N
N
N
N
Br
1.02
1.02
0.94
1.04
2.03
3.04
2.00
f1 (ppm)


8.57
8.21
8.18
8.15
7.83
7.81
7.78
7.70
7.68
7.44
7.41
7.29
7.26
7.24
5.68
N
N
N
N
0.91
1.88
0.96
1.91
1.89
1.15
2.00
f1 (ppm)