Supporting Information

Mixed Valence Cu(I)/Cu(II)-MWCNT as Heterogeneous Catalyst for [Click-Carbene Insertion]-Cascade Reaction

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1. General information:

The reagents were obtained from commercial suppliers and used without further purification. Commercially available terminal alkynes, ethyl diazoacetate, and $TMSN_3$were directly employed without any previous purification technique (distillation/use of molecular sieves). FT-IR spectra were recorded using Affinity-1 SHIMADZU. Powder X-ray diffraction (XRD) was recorded using a Rigaku X-ray diffractometer with Cu-Kα radiation at a wavelength of 1.5418 Å. FESEM and EDX analyses was recorded using a ZEISS sigma 300 scanning along with energy dispersive X-ray spectrometer (EDX). TEM analysis was performed using a JEOL JEM-2100 electron microscope. Thermogravimetric analysis (TGA) was performed on a Mettler Toledo TGA/DTA 1, STAR system analyzer. X-Ray Photoelectron Spectroscopy (XPS) data was determined using PHI 5000 VersaProbe III.Brunauer-Emmett-Teller (BET) data was recorded using BET Surface Area Analyser Model SAA-2000. Inductively coupled plasma-optical emission spectrometry (ICP-OES) was carried out on the Thermo Scientific iCAP 7600 instrument.^{1}H and ^{13}C spectra were recorded on a Bruker Ultrashield400MHz spectrometer. Chemical shift is given in units (δ) relative to tetramethylsilane (TMS) signal as an internal reference in $CDCl_3$. ^{1}H NMR coupling constants were reported in Hz and multiplicity was indicated as follows: s (singlet); d (doublet); t (triplet); q (quartet); m (multiplet); br (broad); dd (doublet of doublets). Chromatographic purification was performed using column chromatography over a manually packed column containing silica gel (230-400 mesh). Melting points were measured in Relitech melting point apparatus. The NMR spectra for all the prepared compounds are included in the following sections of this supporting information.

2. Experimental procedures and characterization details:

2.1 General procedure for the synthesis of *N*-substituted 1,2,3-triazoles

To a stirred mixture of the prepared catalyst (25wt%), terminal alkynes**1** (0.2 mmol), ethyl diazoacetate **2**(0.4 mmol), $TMSN_3$**3**(0.4 mmol) were added in acetonitrile solvent (2 ml)in the presence of K_2CO_3 (2 equiv)and then stirred at 85 ℃for 3 hours. After the completion of the reaction, the mixture was cooled to room temperature. The catalyst was recovered using centrifugation technique and washed with ethyl acetate several times to washed out the remained traces of the reaction mixture and then dried in oven for 6 hours at 60 ℃ for reusing purpose. The organic parts in the reaction mixture were further

separated out by adding ethyl acetate. The combined organic layer was dried over Na_2SO_4 and concentrated in vacuo. The crude residue was then purified by column chromatography on silica gel (ethyl acetate:petroleum ether, 3:7) to afford the desired product.

2.2 EDX spectra:

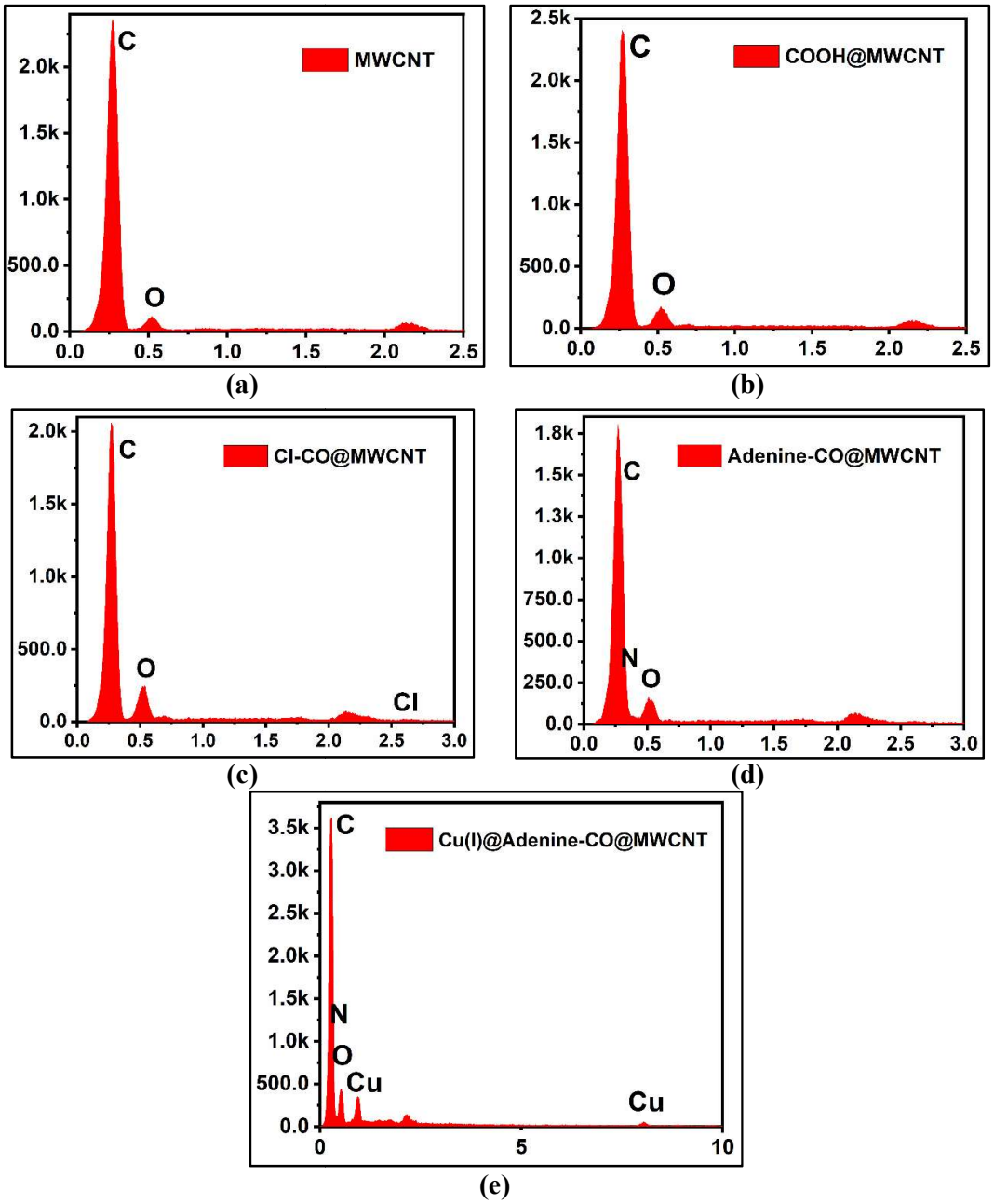


Figure S1. EDX spectra of (a) MWCNT, (b) COOH@MWCNT, (c) Cl-CO@MWCNT, (d) Adenine-CO@MWCNT, (e) Cu@Adenine-CO@MWCNT.

2.3Table of Elemental composition

Table S1. (a) Elemental composition of **MWCNT**		
Element	**Weight %**	**Atomic %**
C K	83.51	87.09
O K	16.49	12.91

Table S1. (b) Elemental composition of **COOH@MWCNT**		
Element	**Weight %**	**Atomic %**
C K	80.55	84.66
O K	19.45	15.34

Table S1. (c) Elemental composition of **Cl-CO@MWCNT**		
Element	**Weight %**	**Atomic %**
C K	78.29	82.96
O K	21.19	16.86
Cl K	0.52	0.19

Table S1. (d) Elemental composition of **Adenine-CO@MWCNT**		
Element	**Weight%**	**Atomic %**
C K	62.46	67.27
N K	20.73	19.14
O K	16.81	13.59

Table S1. (e) Elemental composition of **Cu@Adenine-CO@MWCNT**		
Element	**Weight %**	**Atomic %**
C K	63.01	72.16
N K	13.95	13.70
O K	14.24	12.24
Cu K	8.80	1.91

The raw data for all the characterizations such as FT-IR, P-XRD, XPS, EDX, and TGA has been uploaded to zenodo and are available at: https://doi.org/10.5281/zenodo.19971287.

2.4P-XRD pattern of reused catalyst:

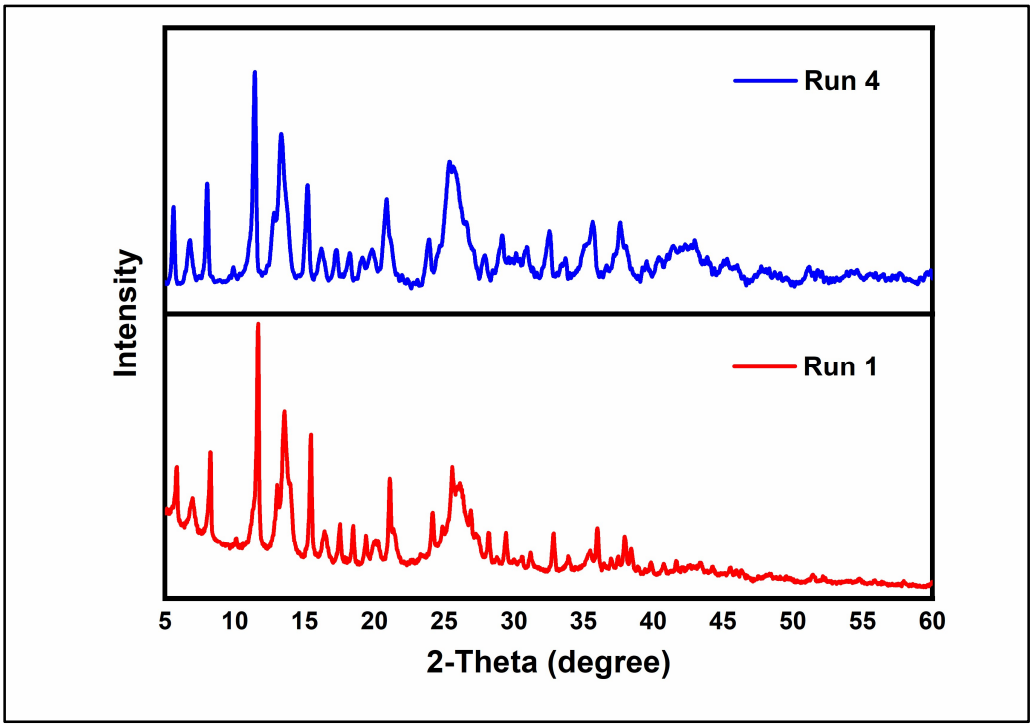
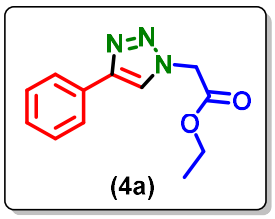


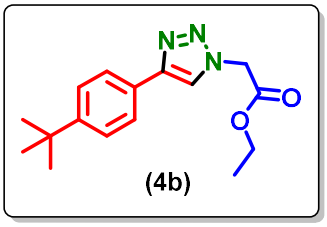
Figure S2. P-XRD pattern comparison of reused catalyst up to four runs.

3. **Spectroscopic data:**

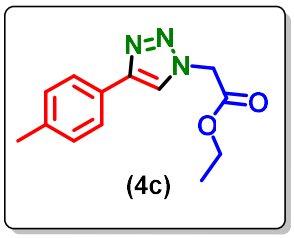
1. **Ethyl 2-(4-phenyl-1H-1,2,3-triazol-1-yl)acetate (4a):** Yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) =** 7.92 (s, 1H), 7.85 (d, *J* = 6.9 Hz, 2H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.35 (t, *J* = 7.4 Hz, 1H), 5.21 (s, 2H), 4.29 (q, *J* = 7.1 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 5H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.26, 148.28, 130.31, 128.84, 128.29, 125.82, 120.88, 62.49, 50.96, 14.08.



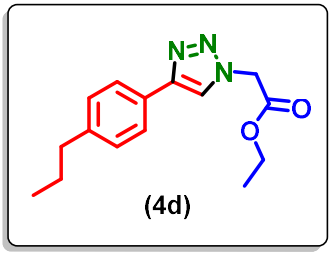
2. **Ethyl 2-(4-(4-(tert-butyl)phenyl)-1H-1,2,3-triazol-1-yl)acetate (4b):** White solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) =** 7.89 (s, 1H), 7.79 (d, *J* = 8.3 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 2H), 5.21 (s, 2H), 4.29 (q, *J* = 7.1 Hz, 2H), 1.36 (s, 9H), 1.32 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.28, 151.40, 148.27, 127.50, 125.76, 125.55, 120.62, 62.43, 50.95, 34.66, 31.27, 14.06.



3. **Ethyl 2-(4-(p-tolyl)-1H-1,2,3-triazol-1-yl)acetate (4c):** White solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) =** 7.88 (s, 1H), 7.75 (d, *J* = 8.1 Hz, 2H), 7.25 (d, *J* = 8 Hz, 2H), 5.21 (s, 2H), 4.3 (q, *J* = 7.1 Hz, 2H), 2.39 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.29, 138.15, 132.37, 129.51, 127.52, 125.73, 120.52, 62.45, 50.95, 21.28, 14.07.

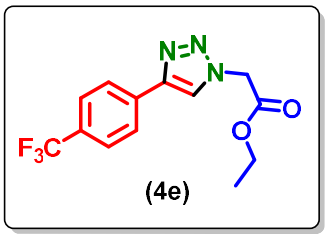


4. **Ethyl 2-(4-(4-propylphenyl)-1H-1,2,3-triazol-1-yl)acetate (4d):** Yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) =** 7.88 (s, 1H), 7.76



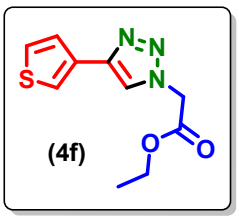
(d, *J* = 8.1 Hz, 2H), 7.24 (s, 2H), 5.21 (s, 2H), 4.3 (q, *J* = 7.1 Hz, 2H), 2.63 (t, *J* = 7.6 Hz, 2H), 1.70-1.65 (m, 2H), 1.32 (t, *J* = 7.2 Hz, 3H), 0.96 (t, *J* = 7.3 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.29, 148.43, 142.99, 128.94, 127.77, 125.74, 120.54, 62.45, 50.97, 37.82, 24.45, 14.07, 13.78.

5. **Ethyl 2-(4-(4-(trifluoromethyl)phenyl)-1H-1,2,3-triazol-1-yl)acetate (4e):** Yellow solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) =** 8.00 (d, *J* = 7.8 Hz, 1H), 7.93 (s, 1H), 7.77 (d, *J* = 7.9 Hz, 1H), 7.65 (t, *J* = 7.6 Hz, 1H), 7.53-7.49 (m, 1H), 5.25 (s, 2H), 4.3 (q, *J* = 7.2 Hz, 2H), 1.31 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.07, 144.65, 132.03, 131.75, 128.38, 126.09, 124.28, 122.75, 62.52, 51.01, 13.98.



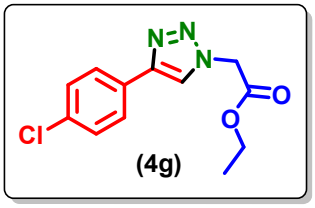
(4e)

6. **Ethyl 2-(4-(thiophen-3-yl)-1H-1,2,3-triazol-1-yl)acetate (4f):** Yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) =** 7.82 (s, 1H), 7.71 (dd, *J* = 2.9 Hz, 1.2 Hz, 1H), 7.47 (dd, *J* = 5 Hz, 1.1 Hz, 1H), 7.4 (dd, *J* = 5 Hz, 3 Hz, 1H), 5.2 (s, 2H), 4.29 (q, *J* = 7.1 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.24, 144.43, 131.54, 126.34, 125.82, 121.37, 120.67, 62.47, 50.90, 14.06.



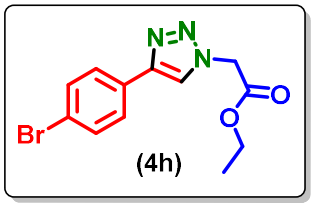
(4f)

7. **Ethyl 2-(4-(4-chlorophenyl)-1H-1,2,3-triazol-1-yl)acetate (4g):** White solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) =** 7.91 (s, 1H), 7.79 (d, *J* = 8.6 Hz, 2H), 7.41 (d, J = 8.6 Hz, 2H), 5.21 (s, 2H), 4.3 (q, *J* = 7.1 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.18, 147.21, 134.04, 129.05, 128.86, 127.04, 120.96, 62.53, 50.95, 14.05.



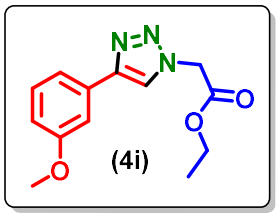
(4g)

8. **Ethyl 2-(4-(4-bromophenyl)-1H-1,2,3-triazol-1-yl)acetate (4h):** White solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm)** =7.92 (s, 1H), 7.73 (d, *J* = 8.5 Hz, 2H), 7.56 (d, *J* = 8.5 Hz, 2H), 5.21 (s, 2H), 4.3 (q, *J* = 7.1 Hz, 2H), 1.33 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.17, 147.24, 131.98, 129.30, 127.31, 122.18, 120.99, 62.54, 50.95, 14.05.



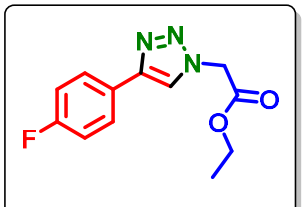
(4h)

9. **Ethyl 2-(4-(3-methoxyphenyl)-1H-1,2,3-triazol-1-yl)acetate (4i)[1]:** White solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm)** = 7.91 (s, 1H), 7.47 (dd, *J* = 2.3 Hz, 1.6 Hz, 1H), 7.40-7.32 (m, 2H), 6.9 (m, 1H), 5.21 (s, 2H), 4.3 (q, *J* = 7.2 Hz, 2H), 3.88 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.23, 160.03, 148.15, 131.64, 129.86, 121.09, 118.22, 114.43, 110.85, 62.48, 55.36, 50.96, 14.06; **HRMS (ESI) m/z** calcd. For $[M+H]^+$: 262.1186, found: 262.1185.



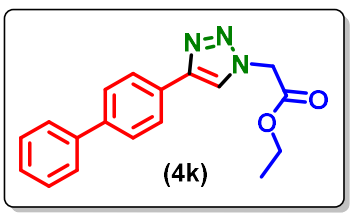
(4i)

10. **Ethyl 2-(4-(4-fluorophenyl)-1H-1,2,3-triazol-1-yl)acetate (4j)[1]:** Yellow solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm)** = 7.88 (s, 1H), 7.83 (dd, *J* = 8.9 Hz, 5.3 Hz, 2H), 7.13 (t, *J* = 8.8 Hz, 2H), 5.21 (s, 2H), 4.31 (q, *J* = 7.2 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.23, 161.52, 147.43, 127.60, 127.52, 120.64, 115.95, 115.73, 62.53, 50.96, 14.08; **HRMS (ESI) m/z** calcd. For $[M+H]^+$:250.0986, found: 250.0981.



(4j)

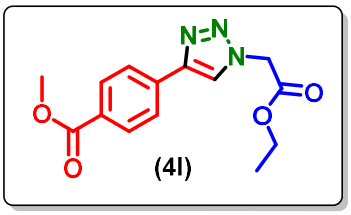
11. **Ethyl 2-(4-([1,1'-biphenyl]-4-yl)-1H-1,2,3-triazol-1-yl)acetate (4k)[1]:** White solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm)** = 7.96-7.92 (m, 3H), 7.70-7.64 (m, 4H), 7.47 (t, *J* = 7.6 Hz, 2H), 5.23 (s, 2H), 4.31 (q, *J* = 7.1 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz,**



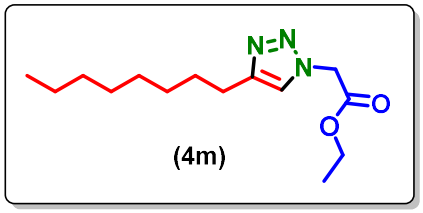
(4k)

$CDCl_3$) δ (ppm) = 166.25, 147.96, 141.03, 128.80, 127.52, 126.98, 126.19, 120.92, 62.49, 50.97, 14.07;**HRMS (ESI) m/z** calcd. For $[M+H]^+$: 308.1394, found: 308.1392.

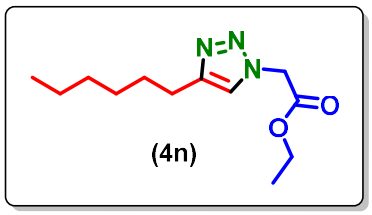
12. **Methyl 4-(1-(2-ethoxy-2-oxoethyl)-1H-1,2,3-triazol-4-yl)benzoate (4l)**[1]**:**White solid; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) =** 8.13 (d, *J* = 8.4 Hz, 2H), 8.02 (s, 1H), 7.94 (d, *J* = 8.4 Hz, 2H), 5.24 (s, 2H), 4.31 (q, *J* = 7.1 Hz, 2H), 3.95 (s, 3H), 1.34 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.23, 166.12, 147.23, 134.63, 132.44, 130.20, 129.54, 121.79, 62.57, 52.33, 50.97, 14.05;**HRMS (ESI) m/z** calcd. For $[M+H]^+$: 290.1135, found: 290.1134.



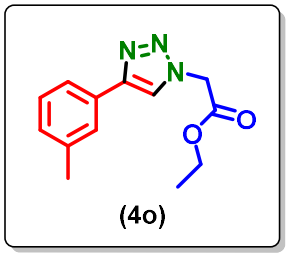
13. **Ethyl 2-(4-octyl-1H-1,2,3-triazol-1-yl)acetate (4m)**:Yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) =** 7.41 (s, 1H), 5.13 (s, 2H), 4.27 (q, *J* = 7.1 Hz, 2H), 2.74 (t, *J* = 7.8 Hz, 2H), 1.73-1.65 (m, 2H), 1.34-1.25 (m, 13H), 0.88 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.48, 148.97, 121.87, 62.30, 50.77, 31.84, 30.92, 29.68, 29.32, 29.21, 29.20.



14. **Ethyl 2-(4-hexyl-1H-1,2,3-triazol-1-yl)acetate**:Yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) =** 7.41 (s, 1H), 5.12 (s, 2H), 4.27 (q, *J* = 7.1 Hz, 2H), 2.74 (t, *J* = 7.7 Hz, 2H), 1.73-1.64 (m, 2H), 1.34-1.26 (m, 9H), 0.91-0.89 (m, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.48, 148.96, 121.87, 62.30, 50.77, 31.55, 29.28, 28.87, 25.65, 22.54, 14.05.



15. Ethyl 2-(4-(m-tolyl)-1H-1,2,3-triazol-1-yl)acetate: Yellow oil; **^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm)** =7.9 (s, 1H), 7.71 (s, 1H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.32 (t, *J* = 7.7 Hz, 1H), 7.16 (d, *J* = 7.5 Hz, 1H), 5.2 (s, 2H), 4.29 (q, *J* = 7.2 Hz, 2H), 2.41 (s, 3H), 1.31 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm)** = 166.48, 148.96, 121.87, 62.30, 50.77, 31.55, 29.28, 28.87, 25.65, 22.54, 14.05.

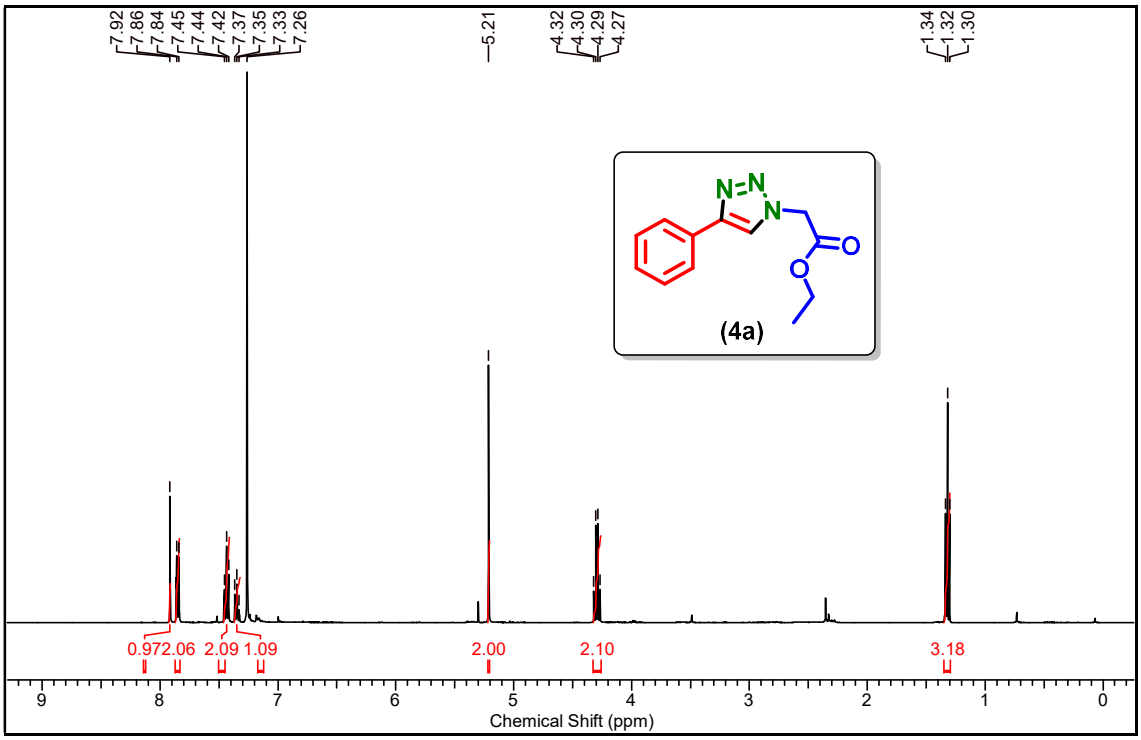


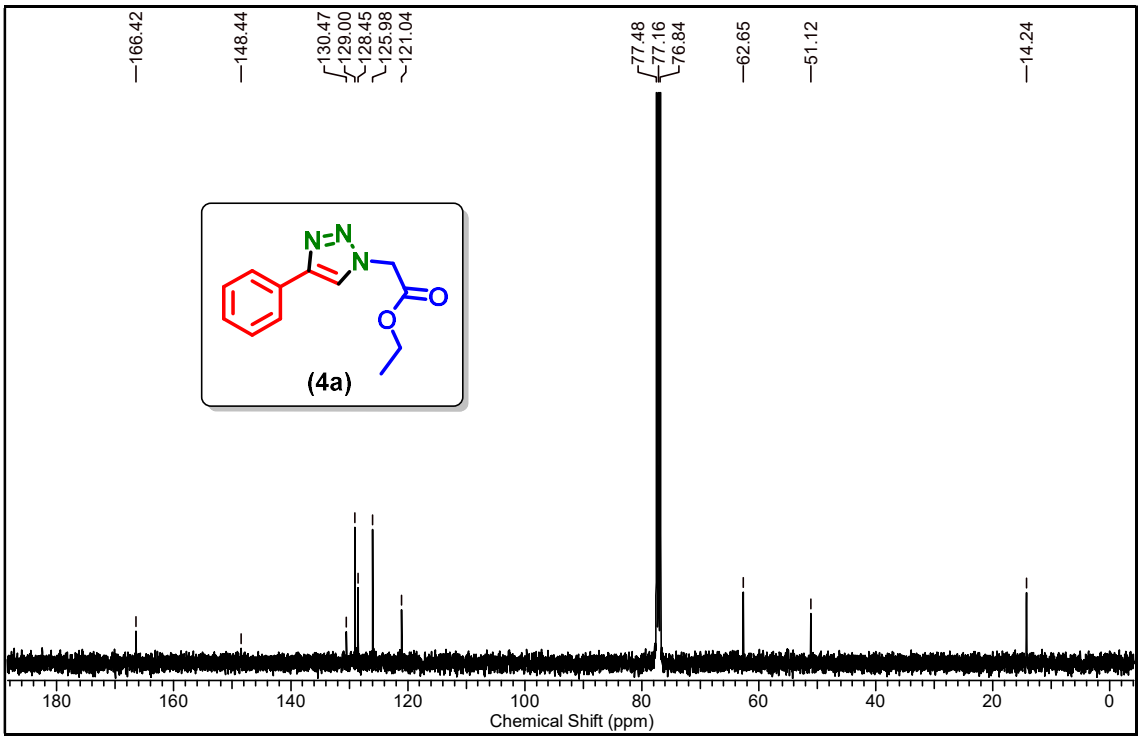
(4o)

4. References:

1. Y. Lv, Z. Wang, L. Song, J. Hao, S. Zhu, H. Yue, W. Wei and D. Yi, *J. Org. Chem.*, 2023, **88**, 17266–17273.

5. ^{1}H and ^{13}C NMR spectra:

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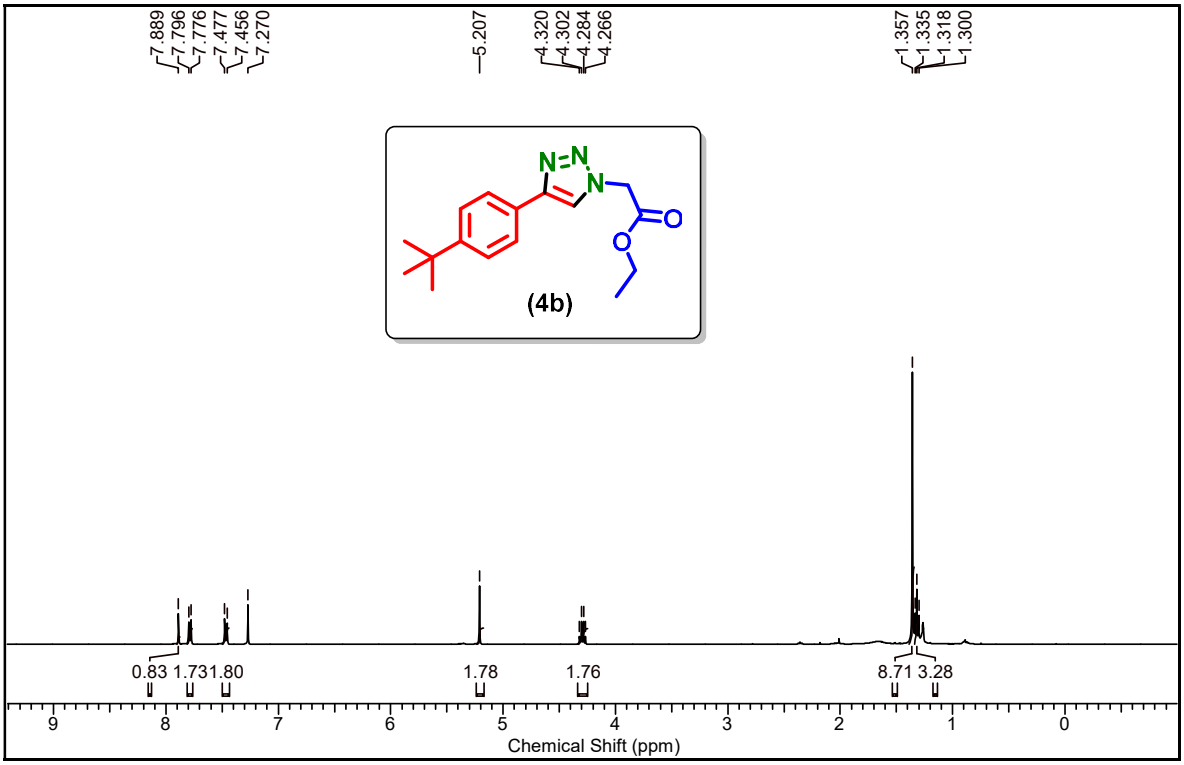
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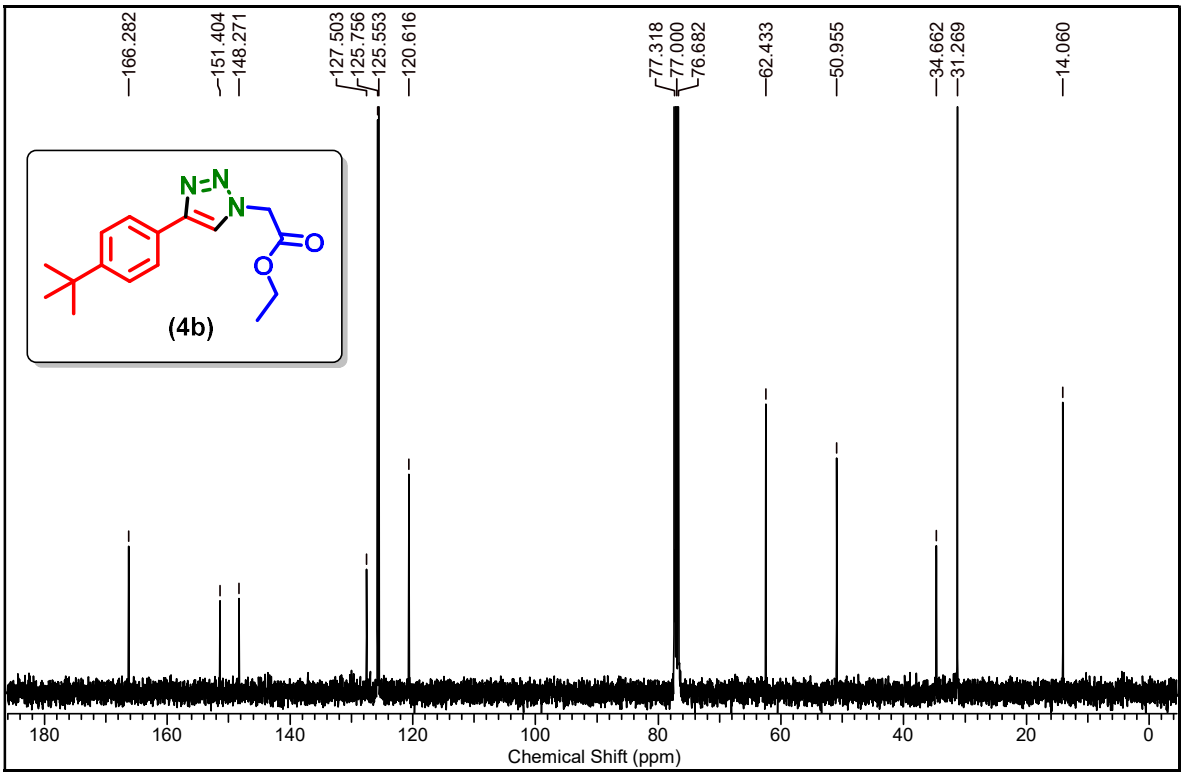
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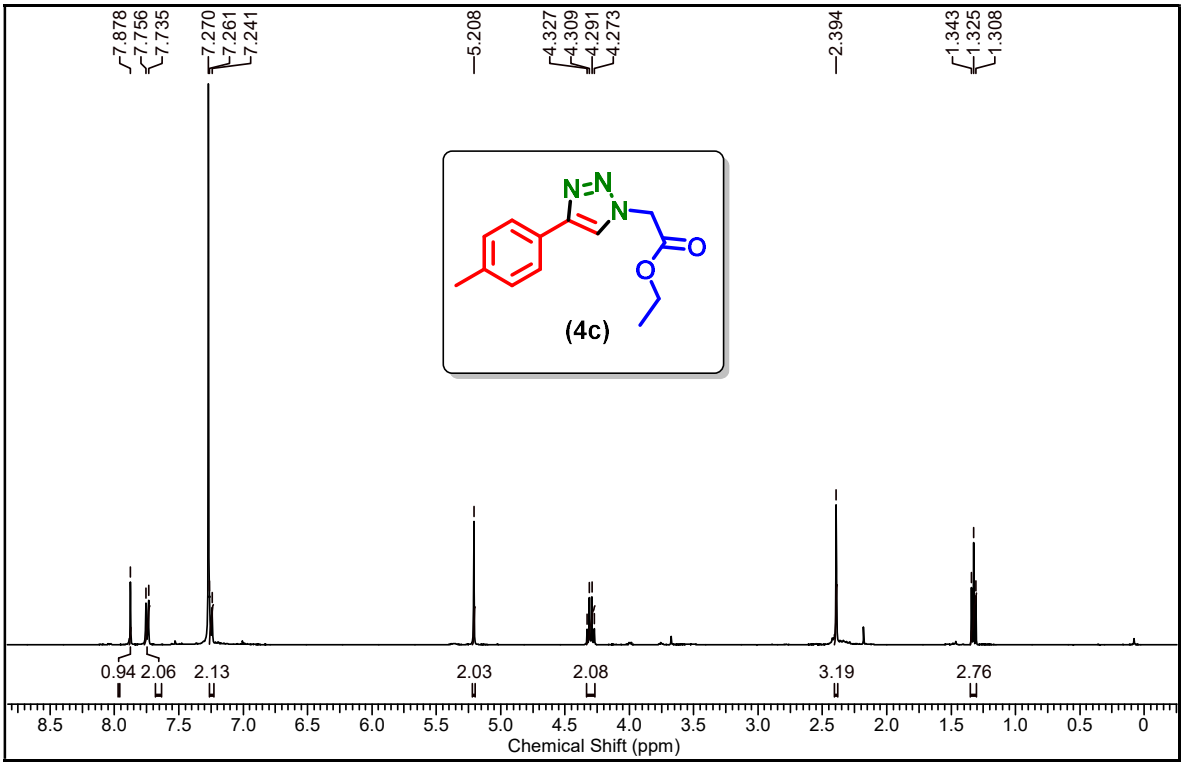
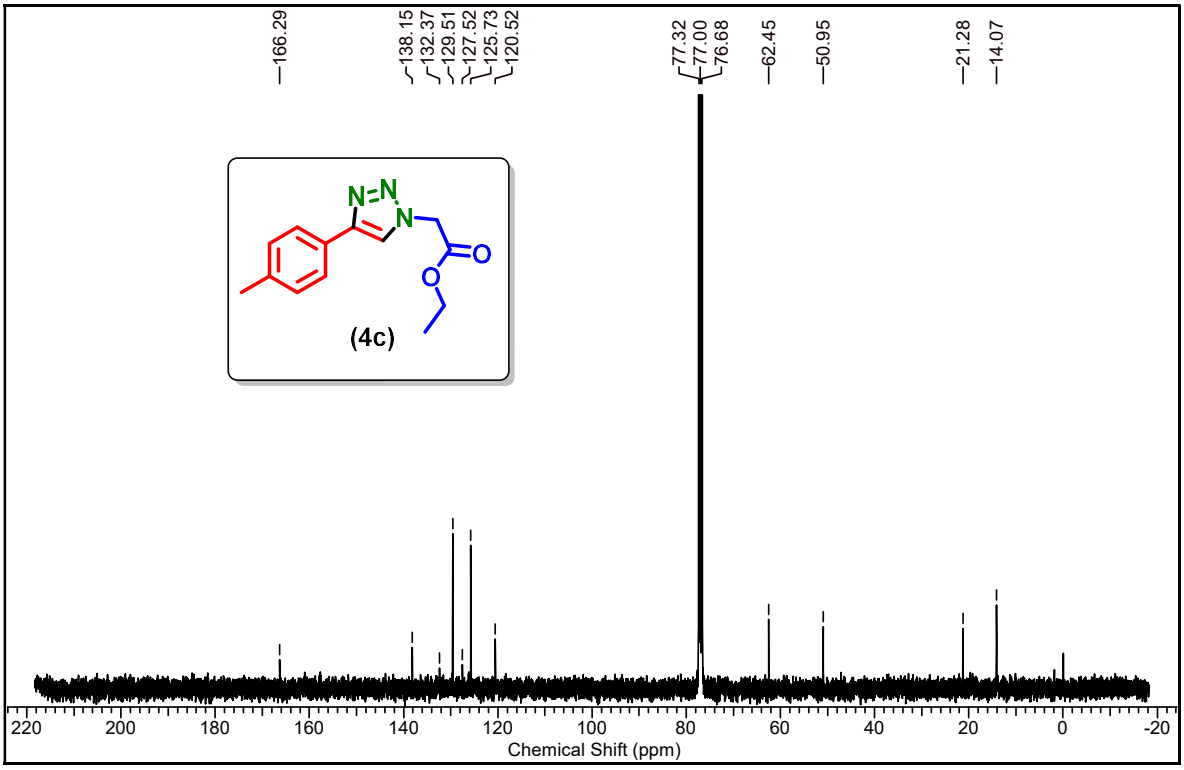
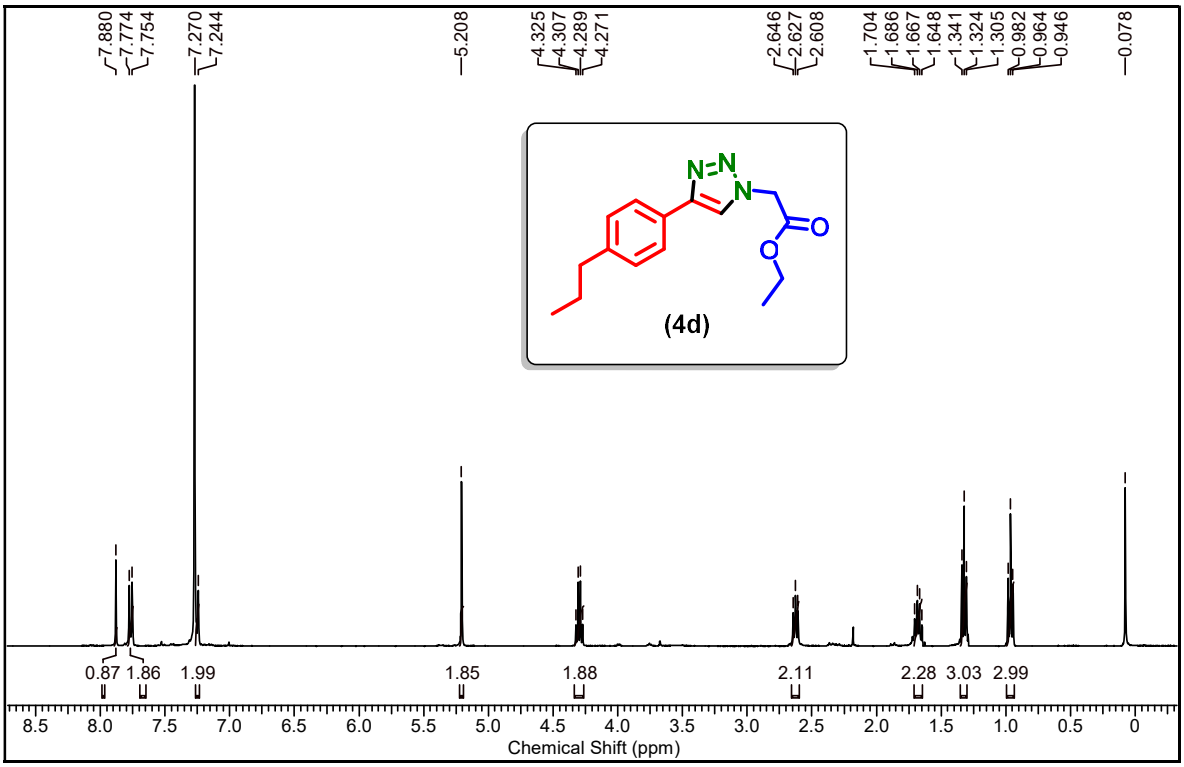
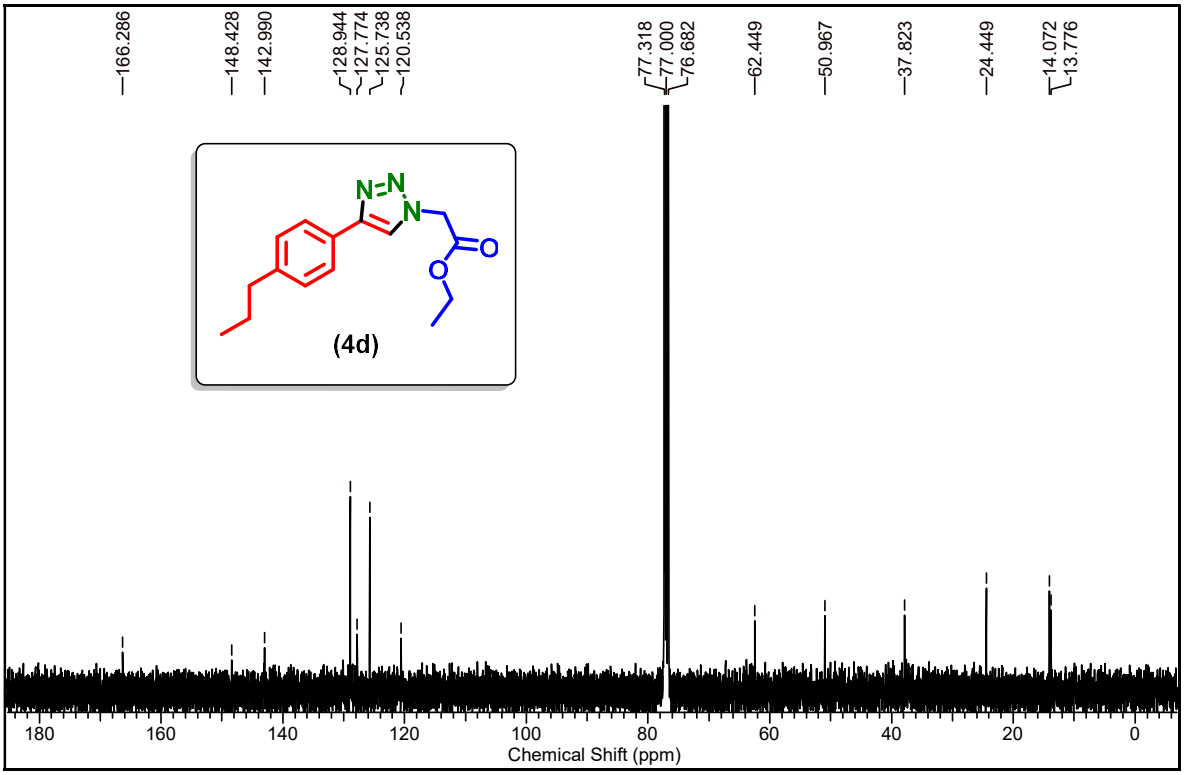
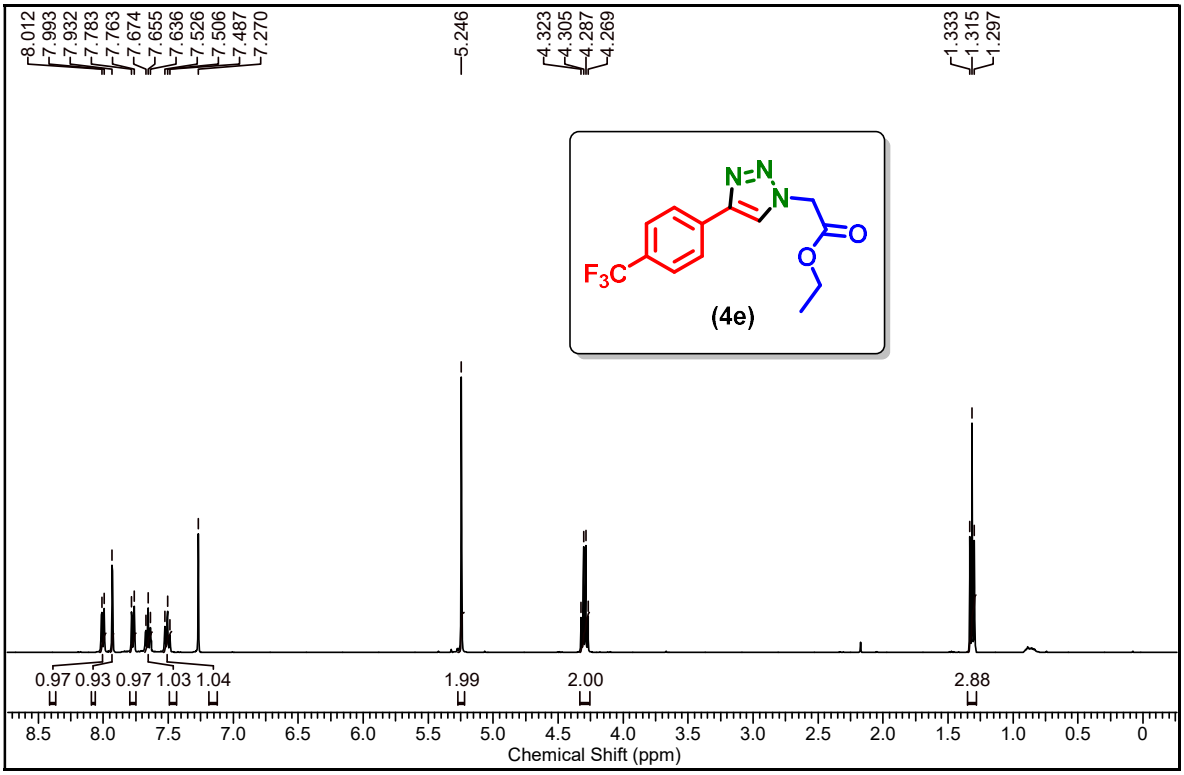
77.318
77.000
76.682
62.433
50.955
34.662
31.269
14.060
(4b)
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

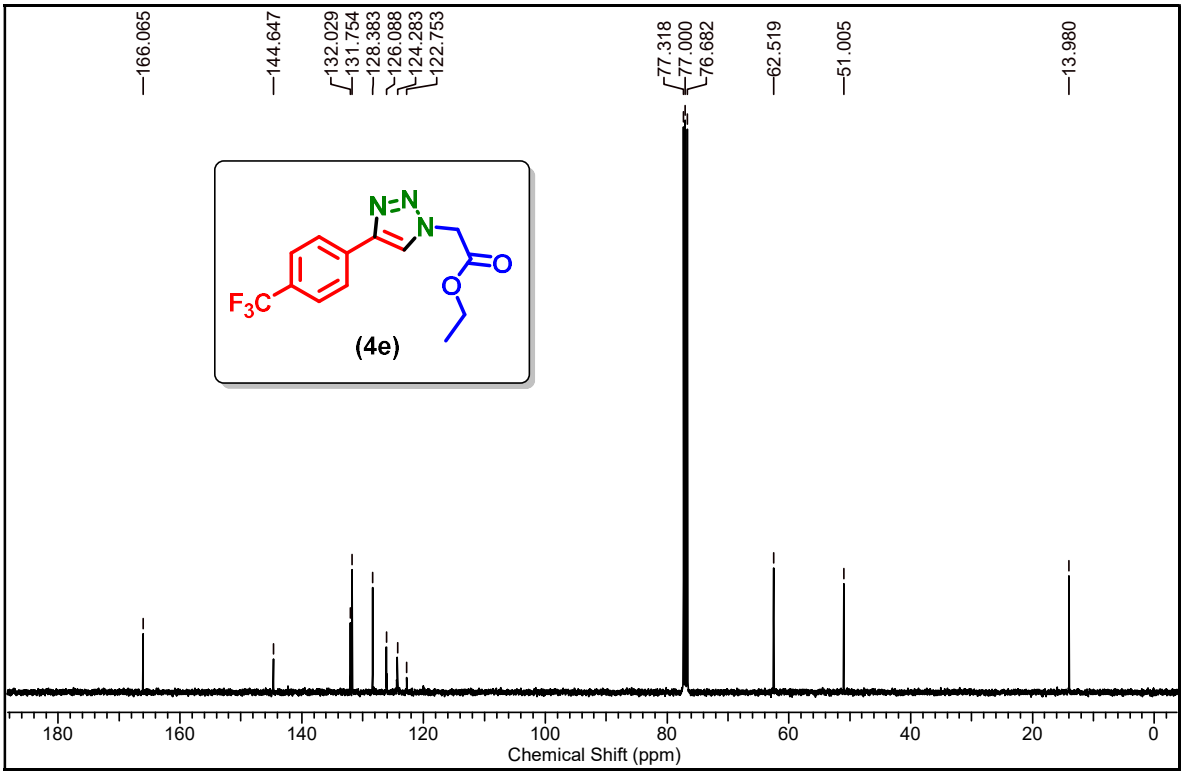

7.878
7.756
7.735
7.270
7.261
7.241
5.208
4.327
4.309
4.291
4.273
2.394
1.343
1.325
1.308
(4c)
0.94
2.06
2.13
2.03
2.08
3.19
2.76
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

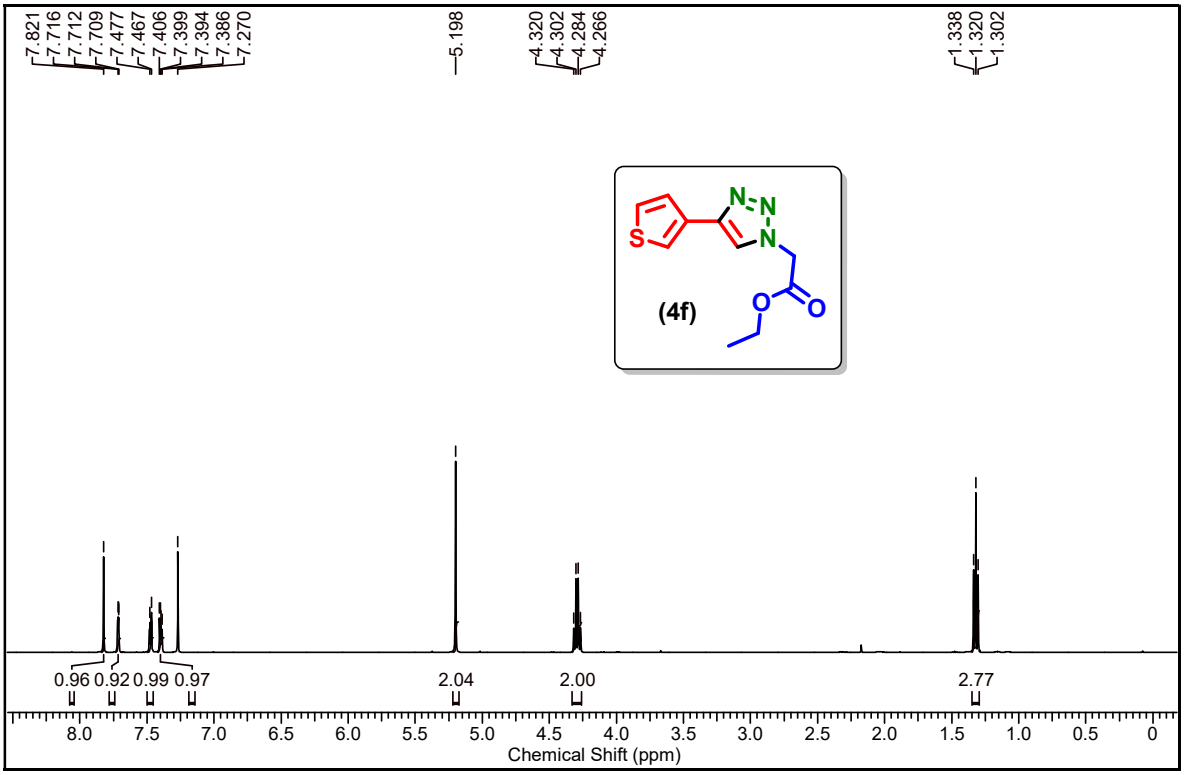

166.29
138.15
132.37
129.51
127.52
125.73
120.52
77.32
77.00
76.68
62.45
50.95
21.28
14.07
(4c)
220
200
180
160
140
120
100
80
60
40
20
0
-20
Chemical Shift (ppm)

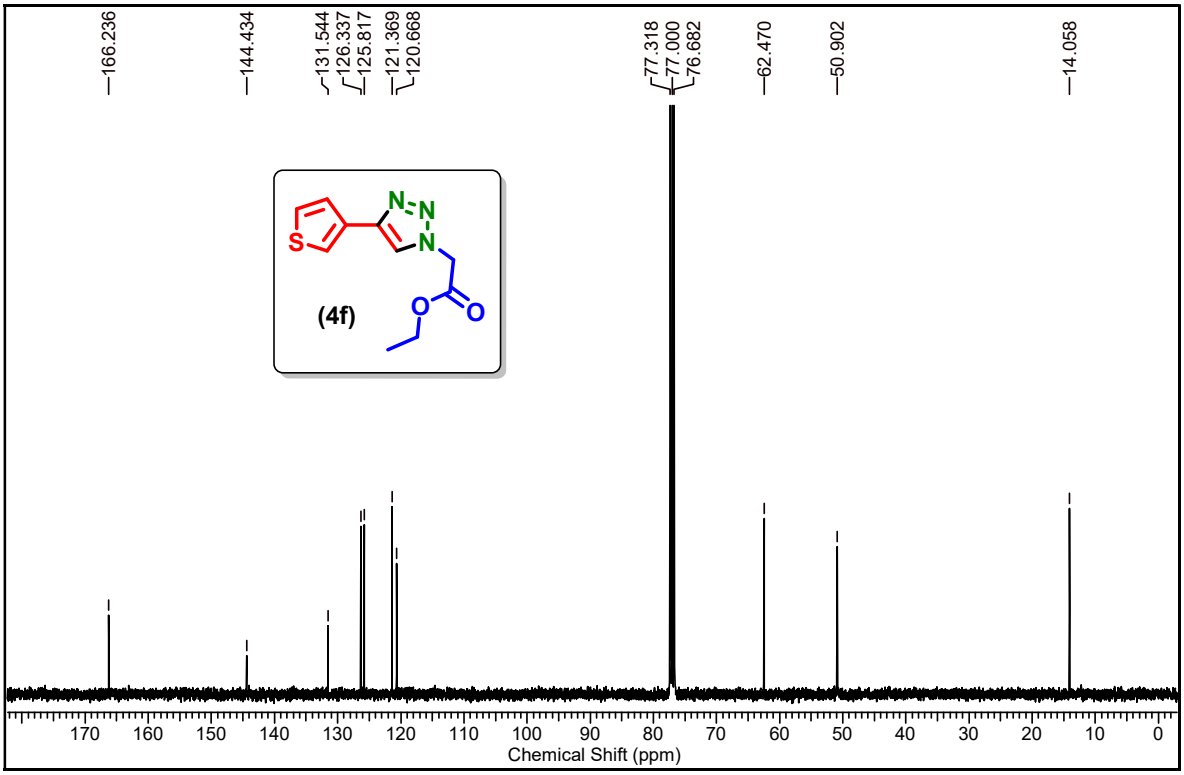

7.880
7.774
7.754
7.270
7.244
5.208
4.325
4.307
4.289
4.271
2.646
2.627
2.608
1.704
1.686
1.667
1.648
1.341
1.324
1.305
0.982
0.964
0.946
0.078
(4d)
0.87
1.86
1.99
1.85
1.88
2.11
2.28
3.03
2.99
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

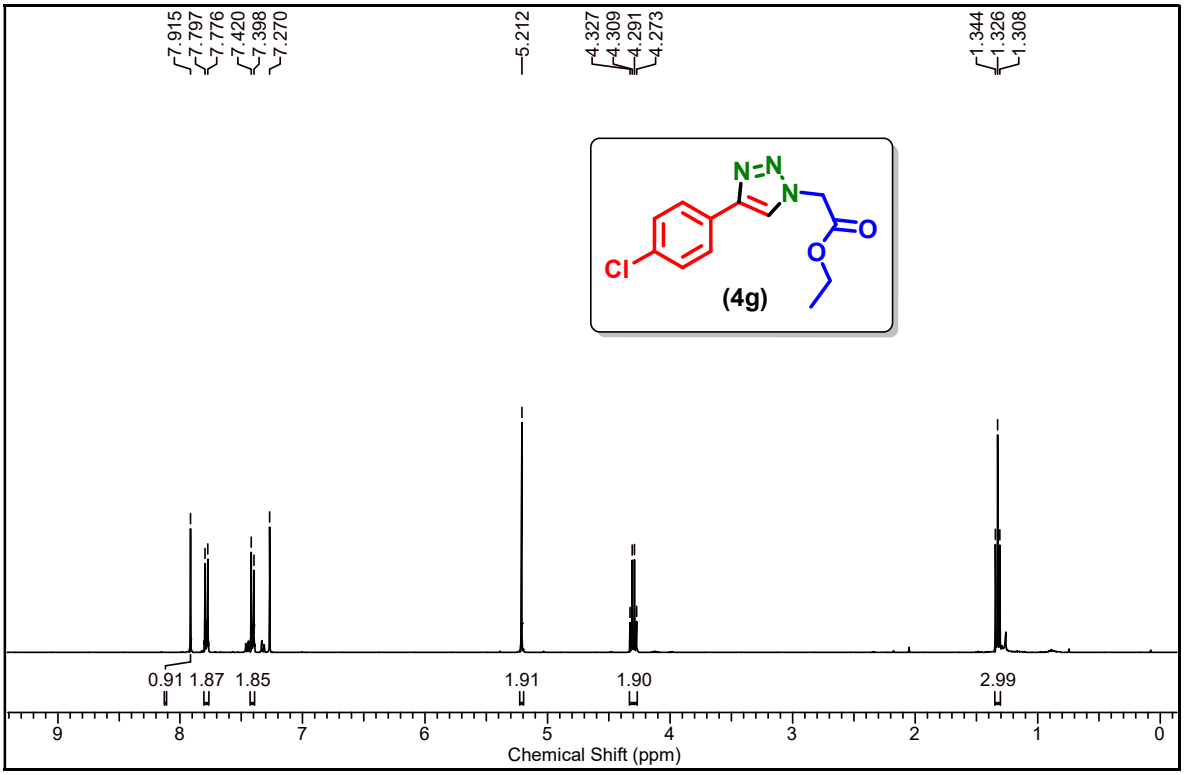

166.286
148.428
142.990
128.944
127.774
125.738
120.538
77.318
77.000
76.682
62.449
50.967
37.823
24.449
14.072
13.776
(4d)
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

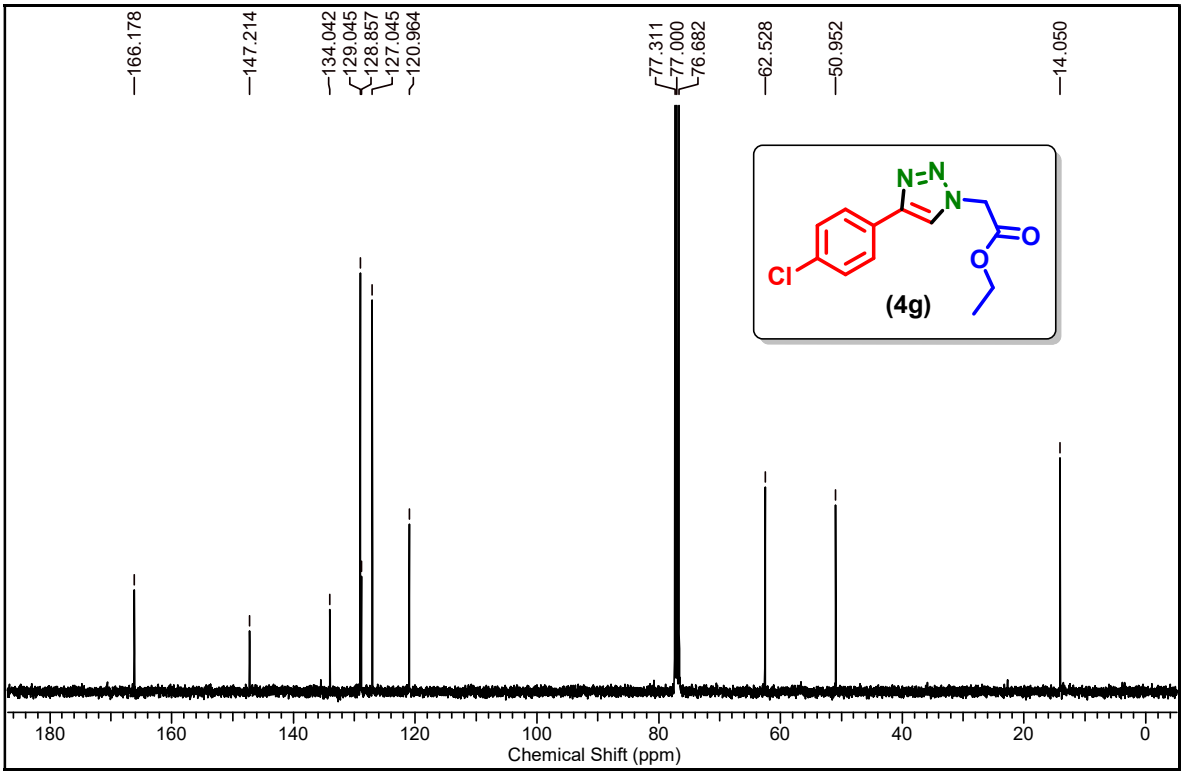

8.012
7.993
7.932
7.783
7.763
7.674
7.655
7.636
7.526
7.506
7.487
7.270
5.246
4.323
4.305
4.287
4.269
1.333
1.315
1.297
(4e)
0.97 0.93 0.97 1.03 1.04
1.99
2.00
2.88
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0
Chemical Shift (ppm)

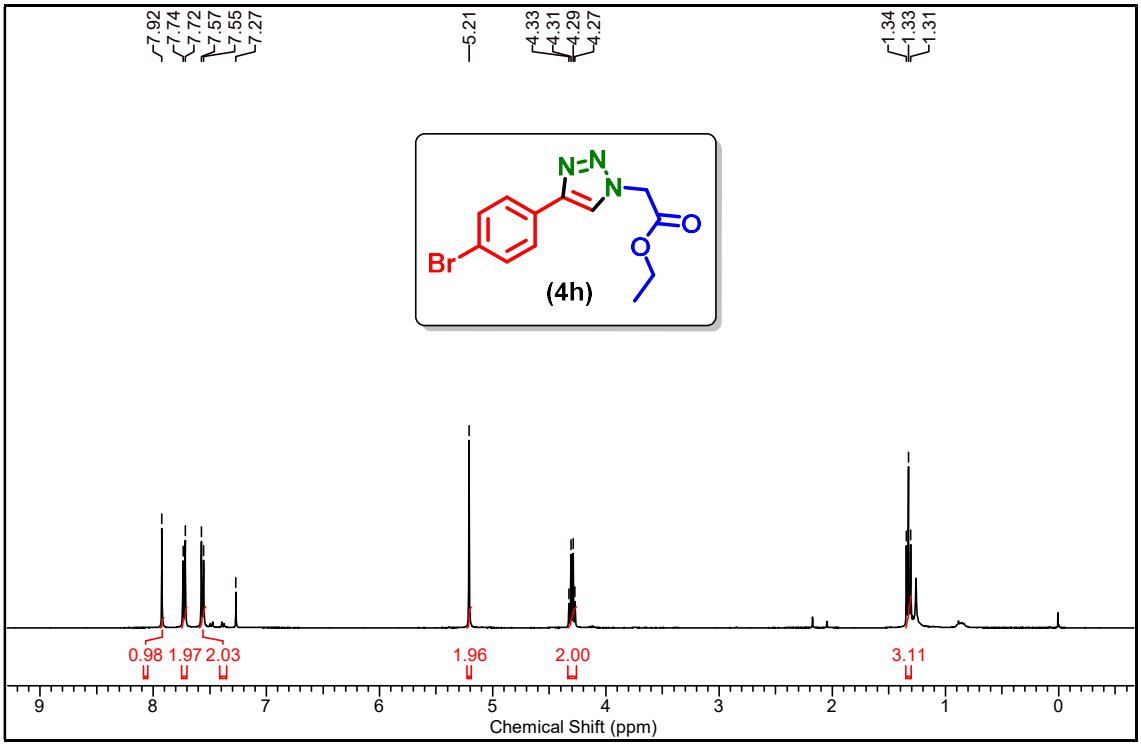

166.065
144.647
132.029
131.754
128.383
126.088
124.283
122.753
77.318
77.000
76.682
62.519
51.005
13.980
(4e)
Chemical Shift (ppm)

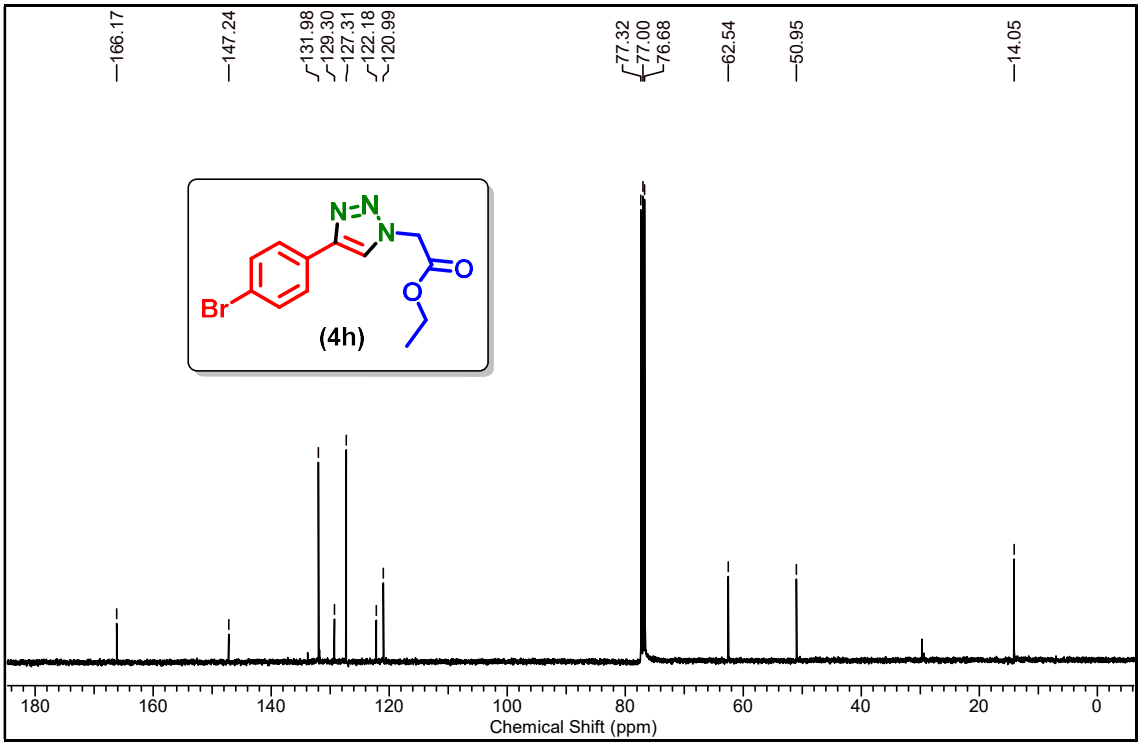

7.821
7.716
7.712
7.709
7.477
7.467
7.406
7.399
7.394
7.386
7.270
5.198
4.320
4.302
4.284
4.266
1.338
1.320
1.302
(4f)
0.96 0.92 0.99 0.97
2.04
2.00
2.77
Chemical Shift (ppm)

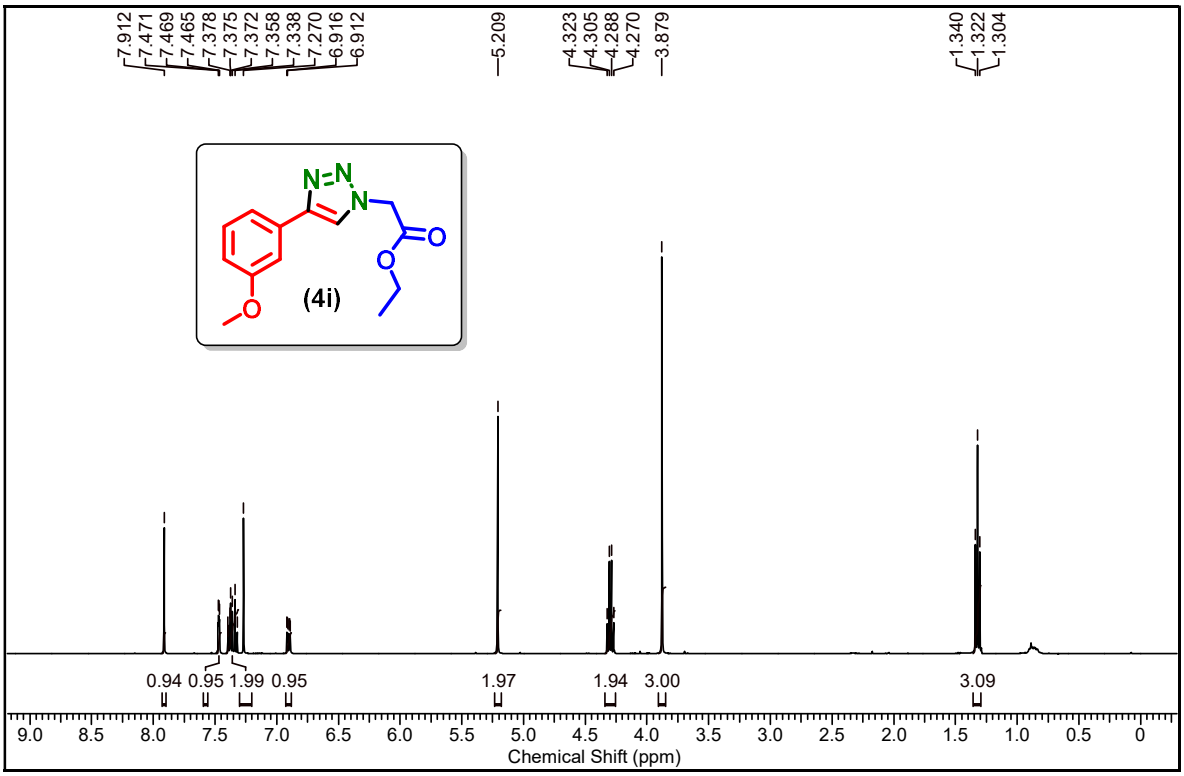

166.236
144.434
131.544
126.337
125.817
121.369
120.668
77.318
77.000
76.682
62.470
50.902
14.058
(4f)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
Chemical Shift (ppm)

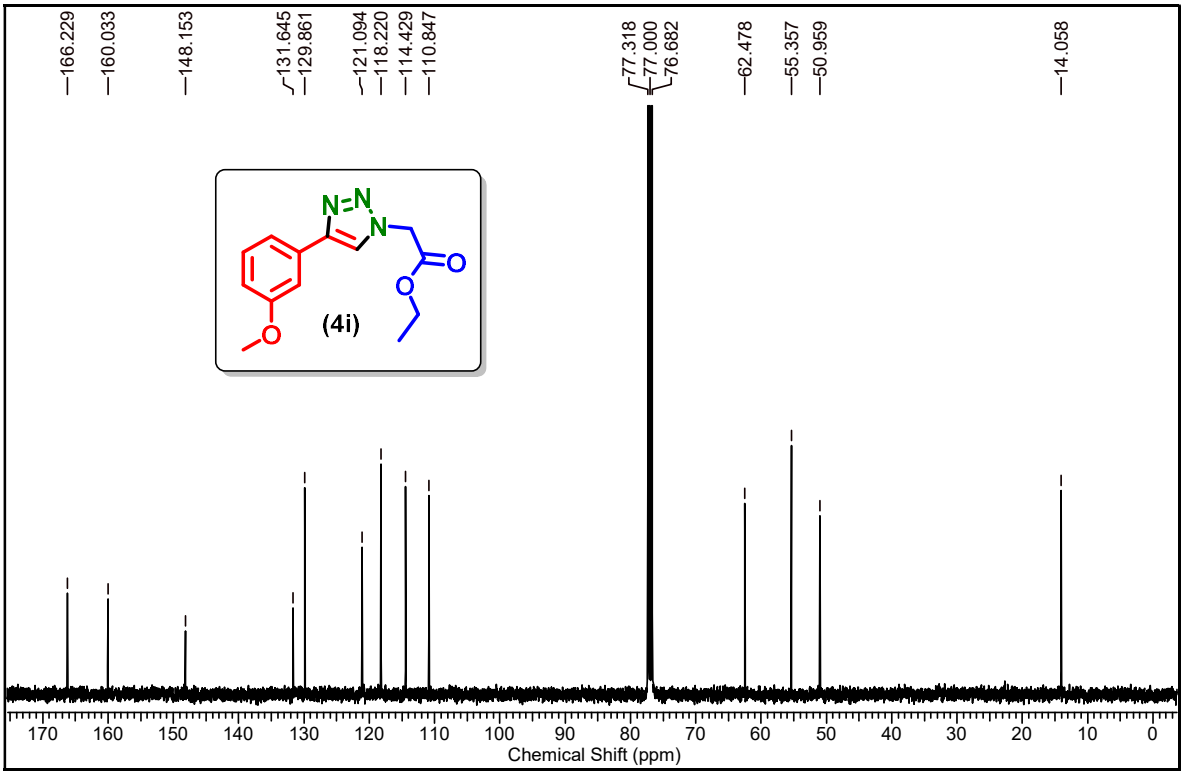

7.915
7.797
7.776
7.420
7.398
7.270
5.212
4.327
4.309
4.291
4.273
1.344
1.326
1.308
Cl
(4g)
0.91 1.87 1.85
1.91
1.90
2.99
9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)

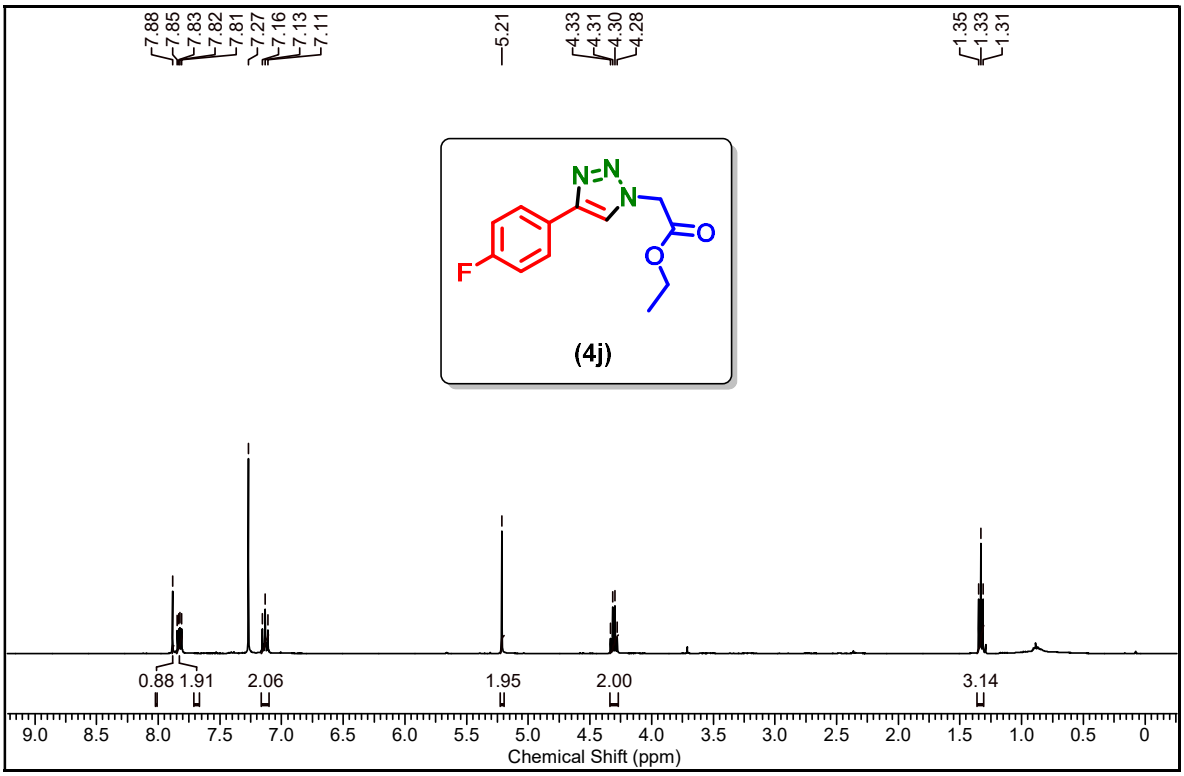

166.178
147.214
134.042
129.045
128.857
127.045
120.964
77.311
77.000
76.682
62.528
50.952
14.050
Cl
(4g)
Chemical Shift (ppm)

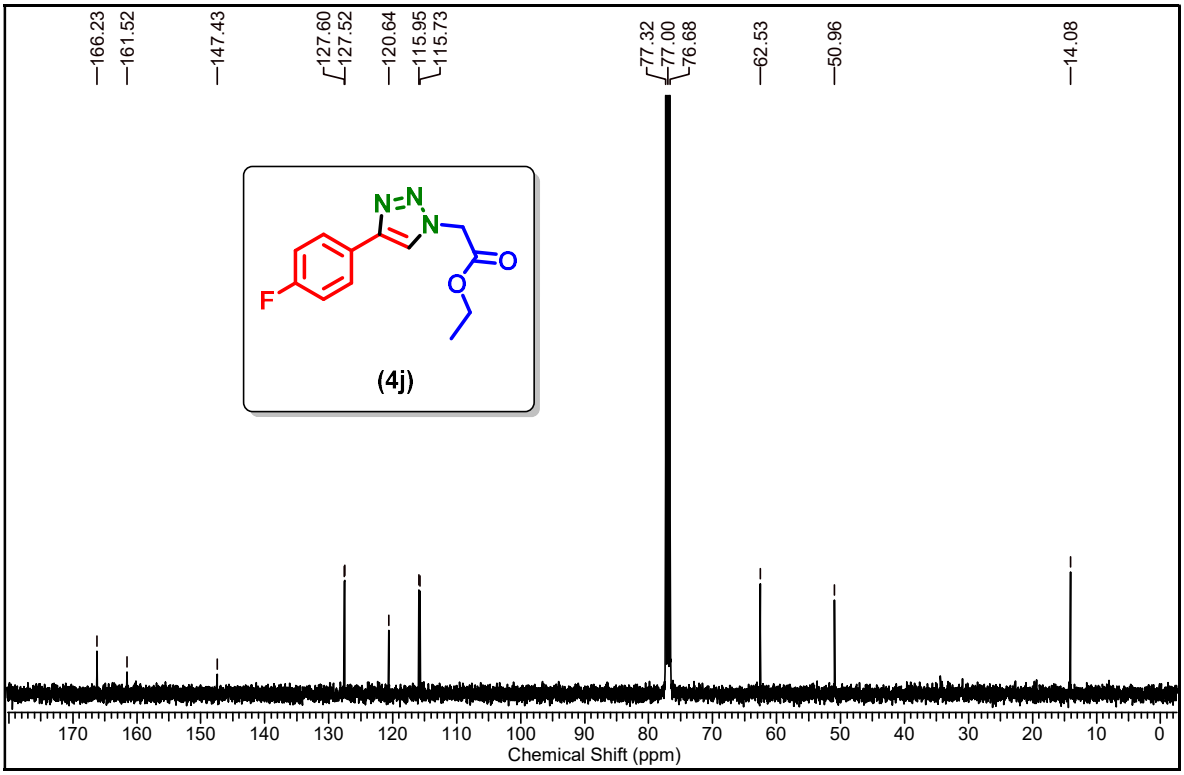

7.92
7.74
7.72
7.57
7.55
7.27
5.21
4.33
4.31
4.29
4.27
1.34
1.33
1.31
Br
(4h)
0.98
1.97
2.03
1.96
2.00
3.11
Chemical Shift (ppm)

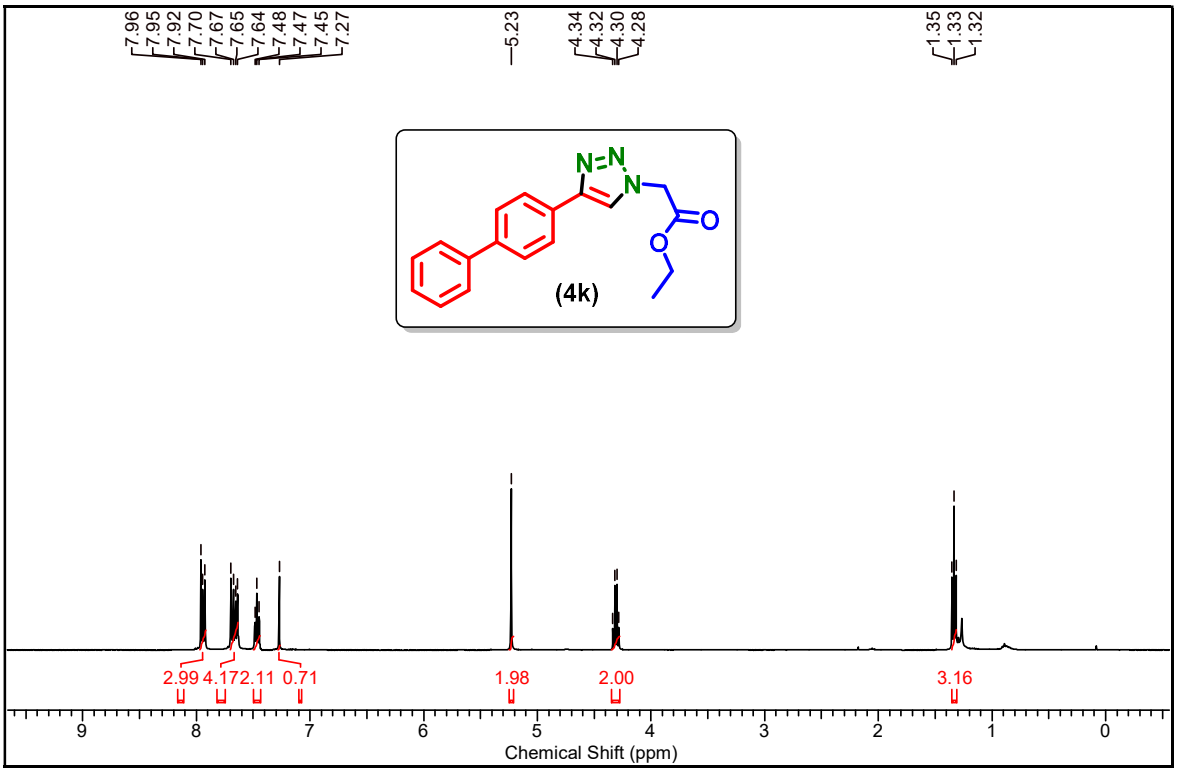

166.17
147.24
131.98
129.30
127.31
122.18
120.99
77.32
77.00
76.68
62.54
50.95
14.05
Br
(4h)
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

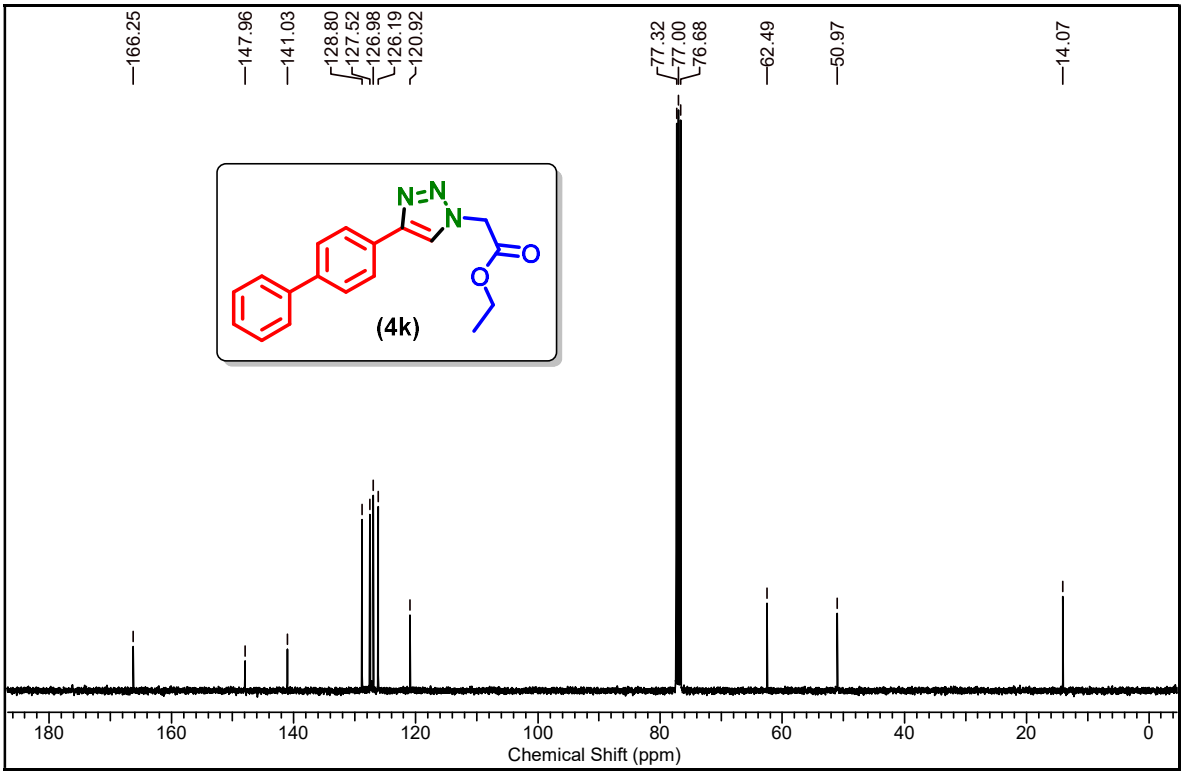

7.912
7.471
7.469
7.465
7.378
7.375
7.372
7.358
7.338
7.270
6.916
6.912
5.209
4.323
4.305
4.288
4.270
3.879
1.340
1.322
1.304
(4i)
0.94 0.95 1.99 0.95
1.97
1.94 3.00
3.09
Chemical Shift (ppm)

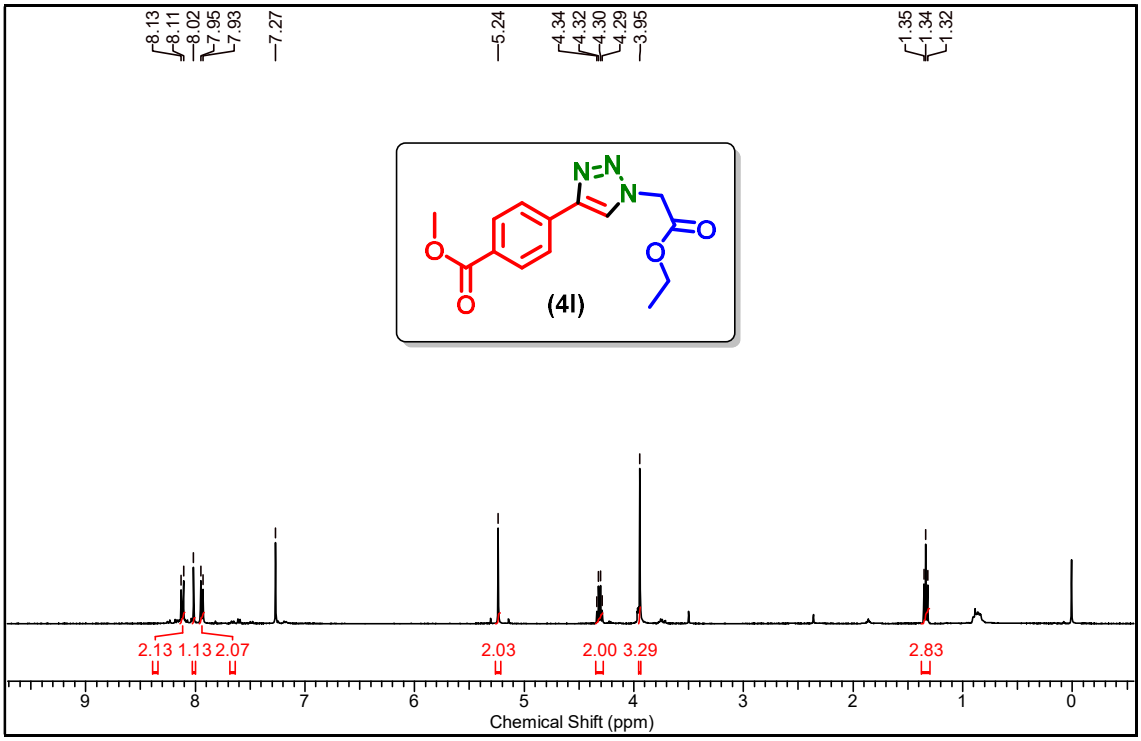

166.229
160.033
148.153
131.645
129.861
121.094
118.220
114.429
110.847
77.318
77.000
76.682
62.478
55.357
50.959
14.058
(4i)
Chemical Shift (ppm)

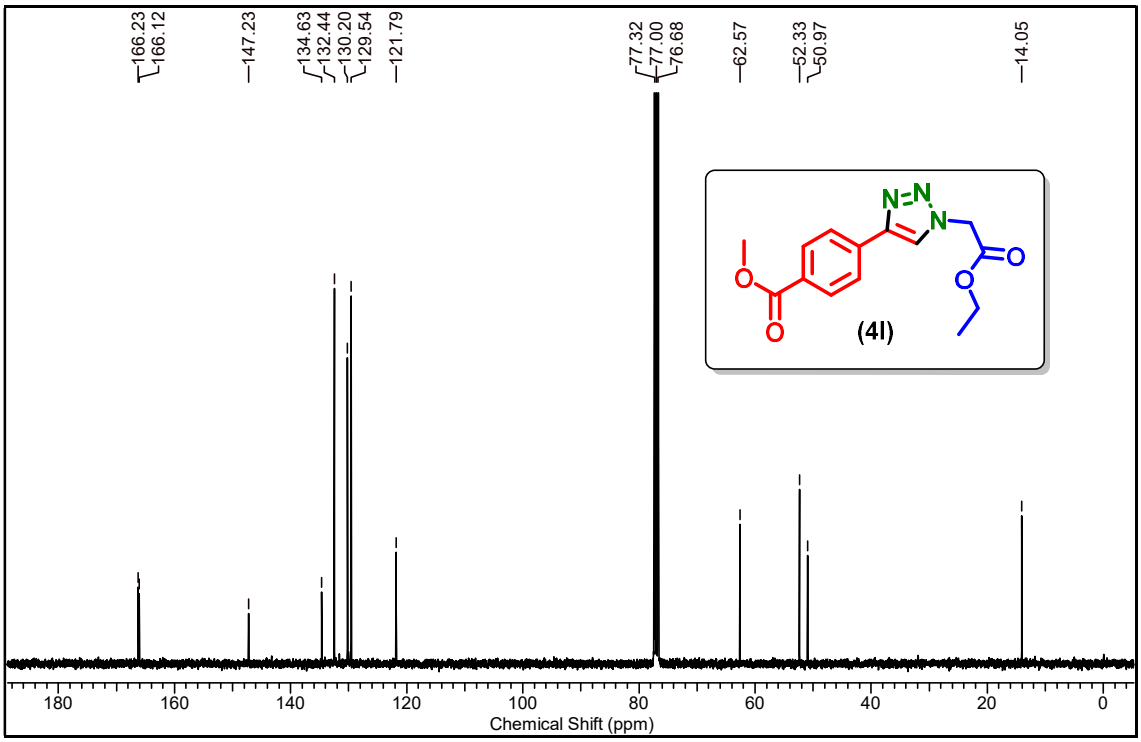

7.88
7.85
7.83
7.82
7.81
7.27
7.16
7.13
7.11
5.21
4.33
4.31
4.30
4.28
1.35
1.33
1.31
(4j)
0.88
1.91
2.06
1.95
2.00
3.14
Chemical Shift (ppm)

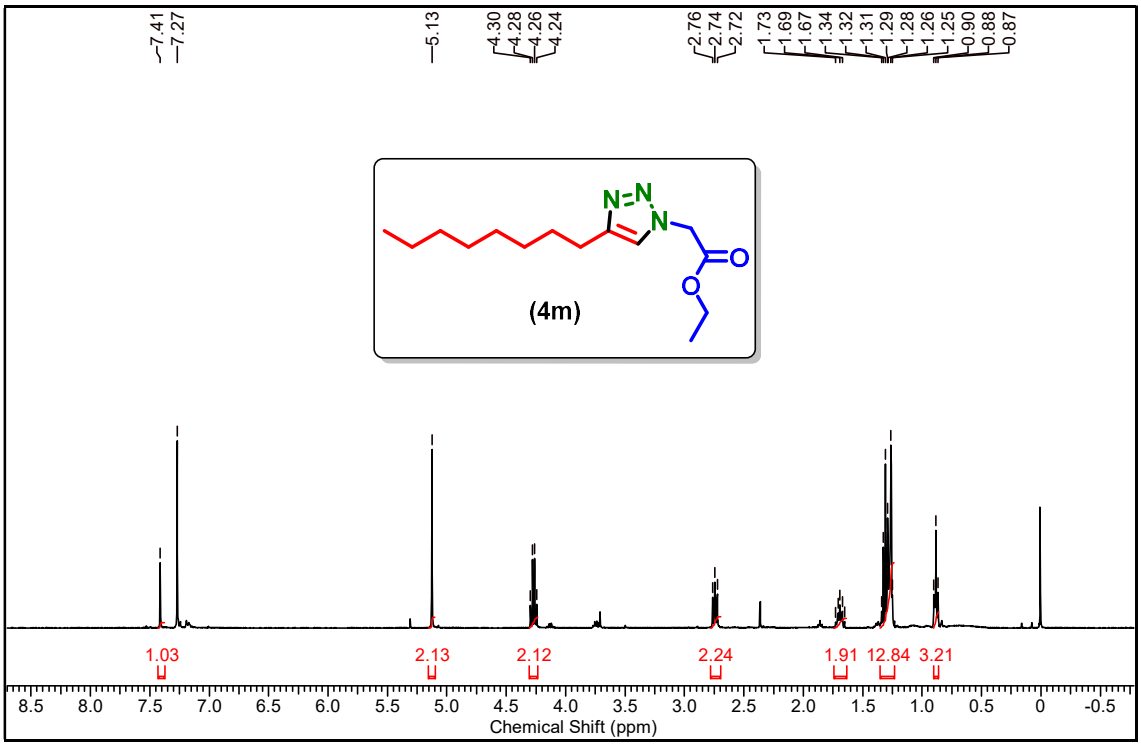

166.23
161.52
147.43
127.60
127.52
120.64
115.95
115.73
77.32
77.00
76.68
62.53
50.96
14.08
(4j)
Chemical Shift (ppm)

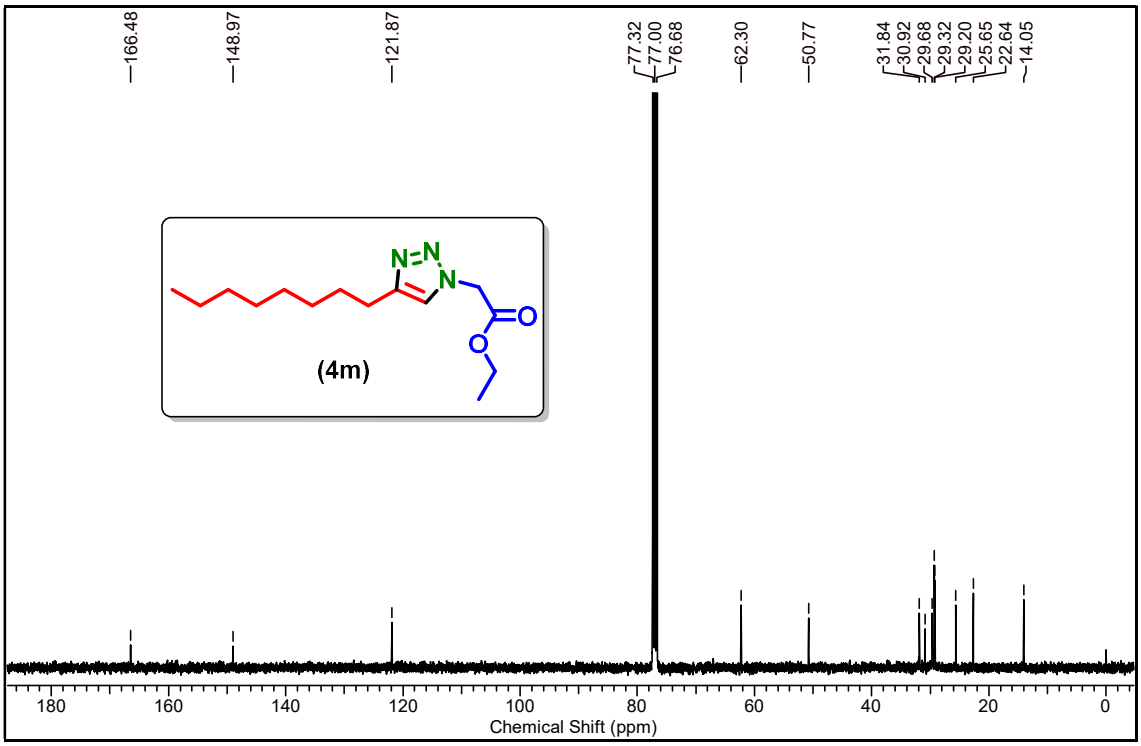

7.96
7.95
7.92
7.70
7.67
7.65
7.64
7.48
7.47
7.45
7.27
5.23
4.34
4.32
4.30
4.28
1.35
1.33
1.32
(4k)
2.99
4.17
2.11
0.71
1.98
2.00
3.16
Chemical Shift (ppm)

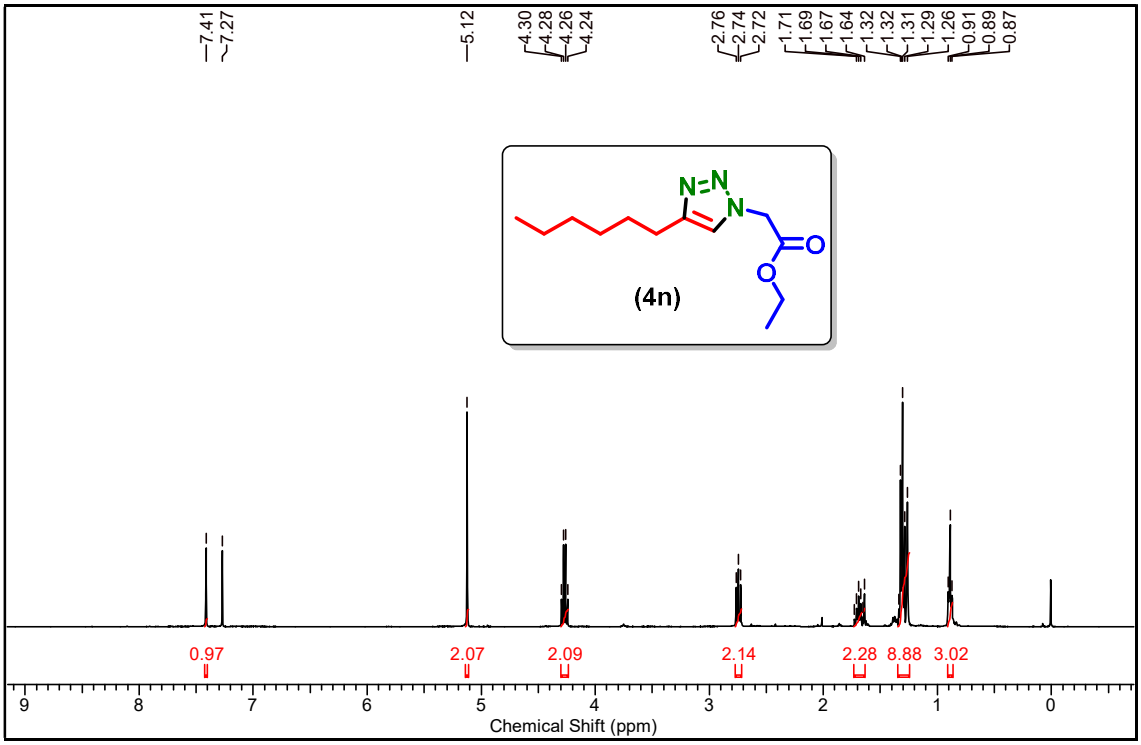

166.25
147.96
141.03
128.80
127.52
126.98
126.19
120.92
77.32
77.00
76.68
62.49
50.97
14.07
(4k)
Chemical Shift (ppm)

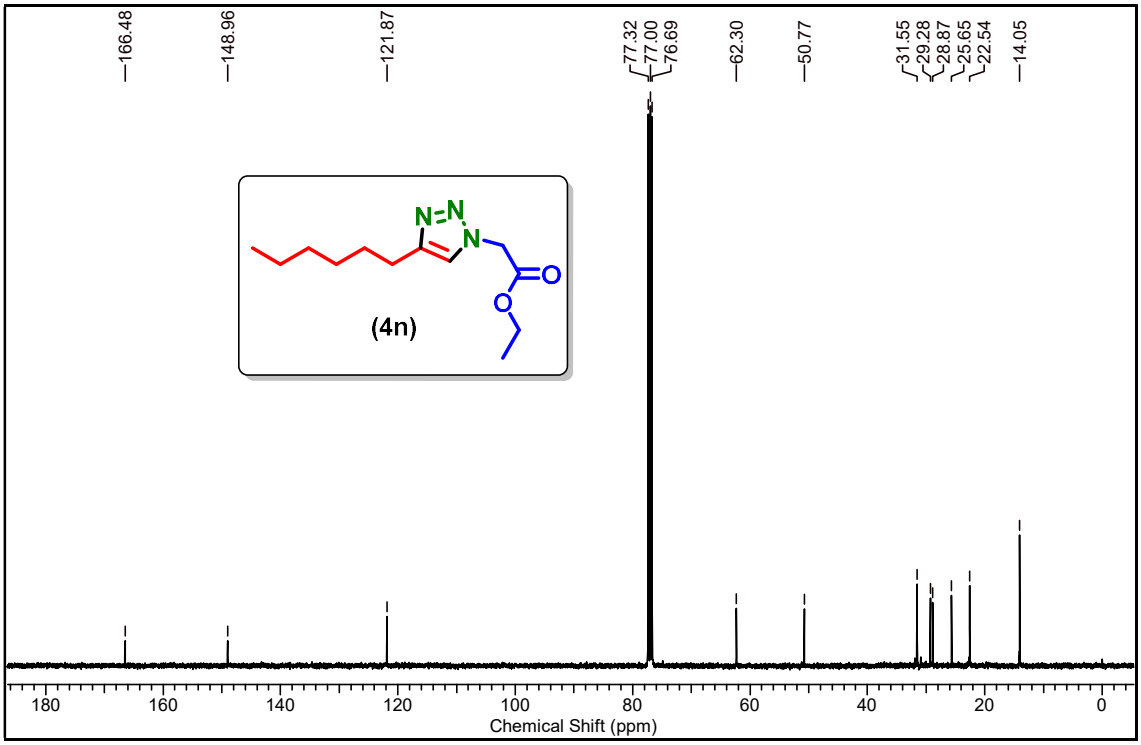

8.13
8.11
8.02
7.95
7.93
7.27
5.24
4.34
4.32
4.30
4.29
3.95
1.35
1.34
1.32
(4l)
2.13 1.13 2.07
2.03
2.00 3.29
2.83
Chemical Shift (ppm)

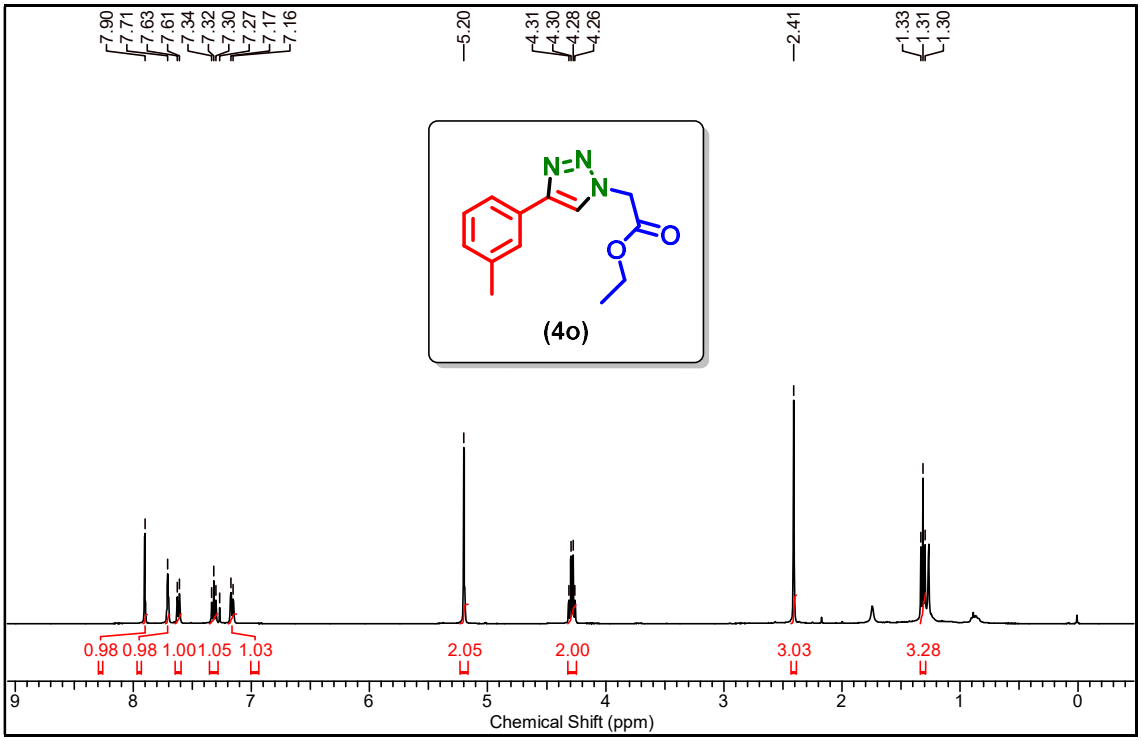

166.23
166.12
147.23
134.63
132.44
130.20
129.54
121.79
77.32
77.00
76.68
62.57
52.33
50.97
14.05
(4l)
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

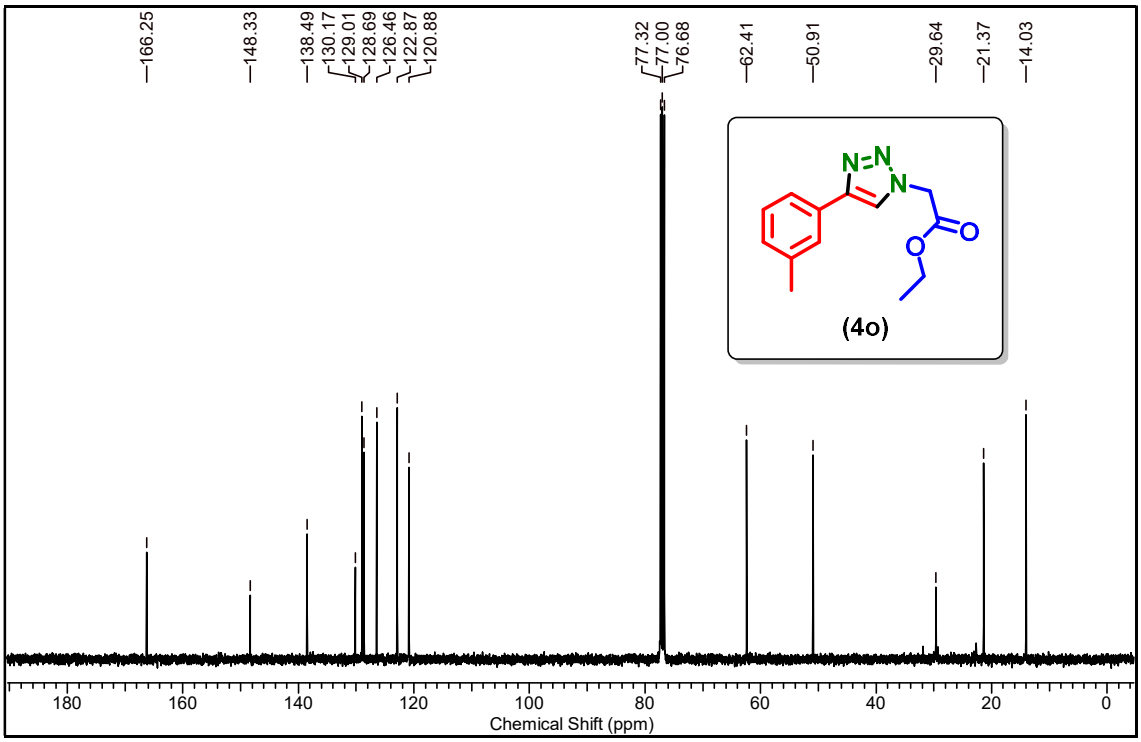

7.41
7.27
5.13
4.30
4.28
4.26
4.24
2.76
2.74
2.72
1.73
1.69
1.67
1.34
1.32
1.31
1.29
1.28
1.26
1.25
0.90
0.88
0.87
(4m)
1.03
2.13
2.12
2.24
1.91
12.84
3.21
Chemical Shift (ppm)


166.48
148.97
121.87
77.32
77.00
76.68
62.30
50.77
31.84
30.92
29.68
29.32
29.20
25.65
22.64
14.05
(4m)
Chemical Shift (ppm)

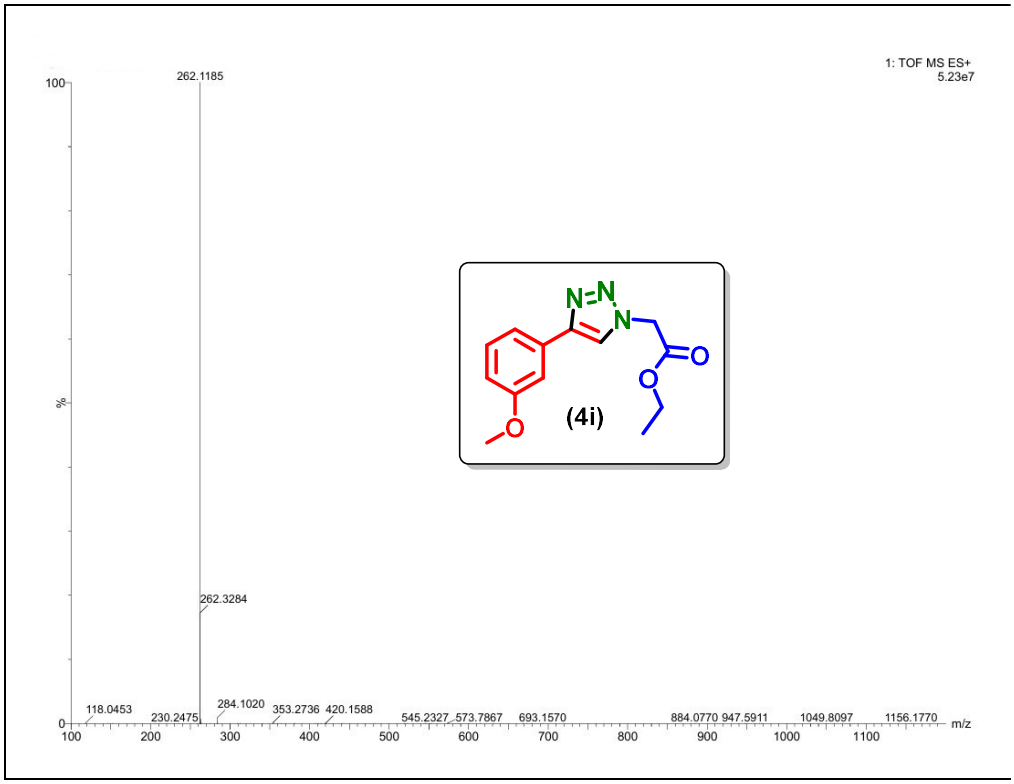

7.41
7.27
5.12
4.30
4.28
4.26
4.24
2.76
2.74
2.72
1.71
1.69
1.67
1.64
1.32
1.32
1.31
1.29
1.26
0.91
0.89
0.87
(4n)
0.97
2.07
2.09
2.14
2.28
8.88
3.02
Chemical Shift (ppm)

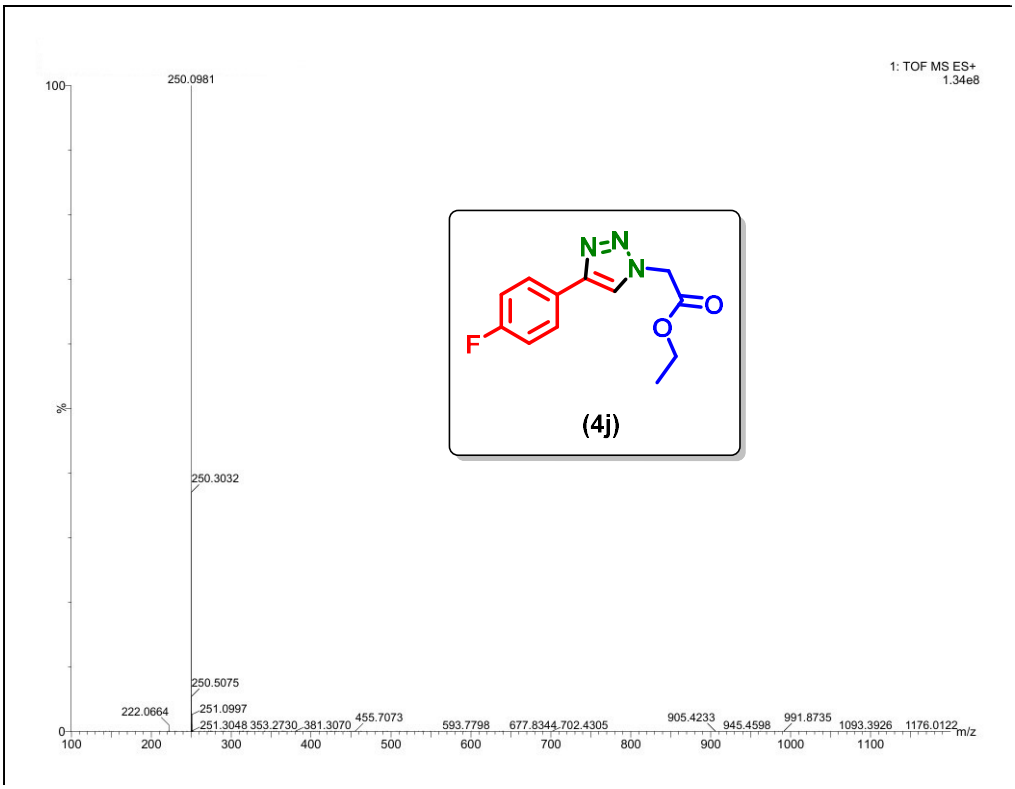

166.48
148.96
121.87
77.32
77.00
76.69
62.30
50.77
31.55
29.28
28.87
25.65
22.54
14.05
(4n)
Chemical Shift (ppm)

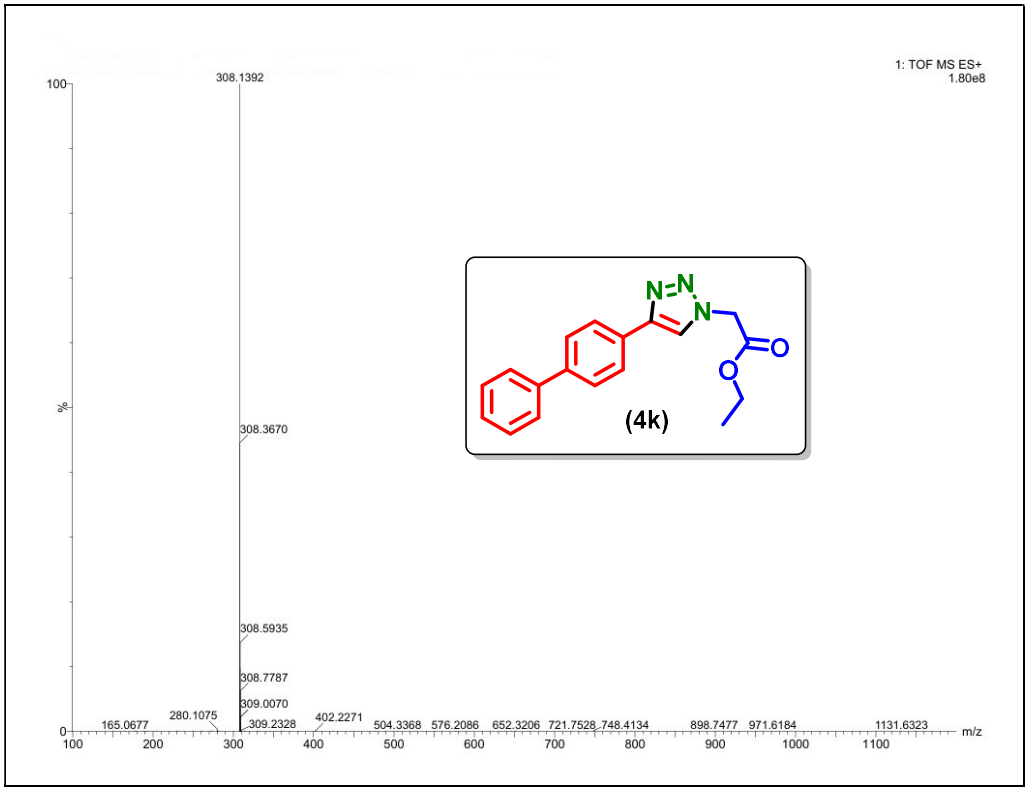

7.90
7.71
7.63
7.61
7.34
7.32
7.30
7.27
7.17
7.16
5.20
4.31
4.30
4.28
4.26
2.41
1.33
1.31
1.30
N=N
N
O
O
(4o)
0.98 0.98 1.00 1.05 1.03
2.05
2.00
3.03
3.28
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)

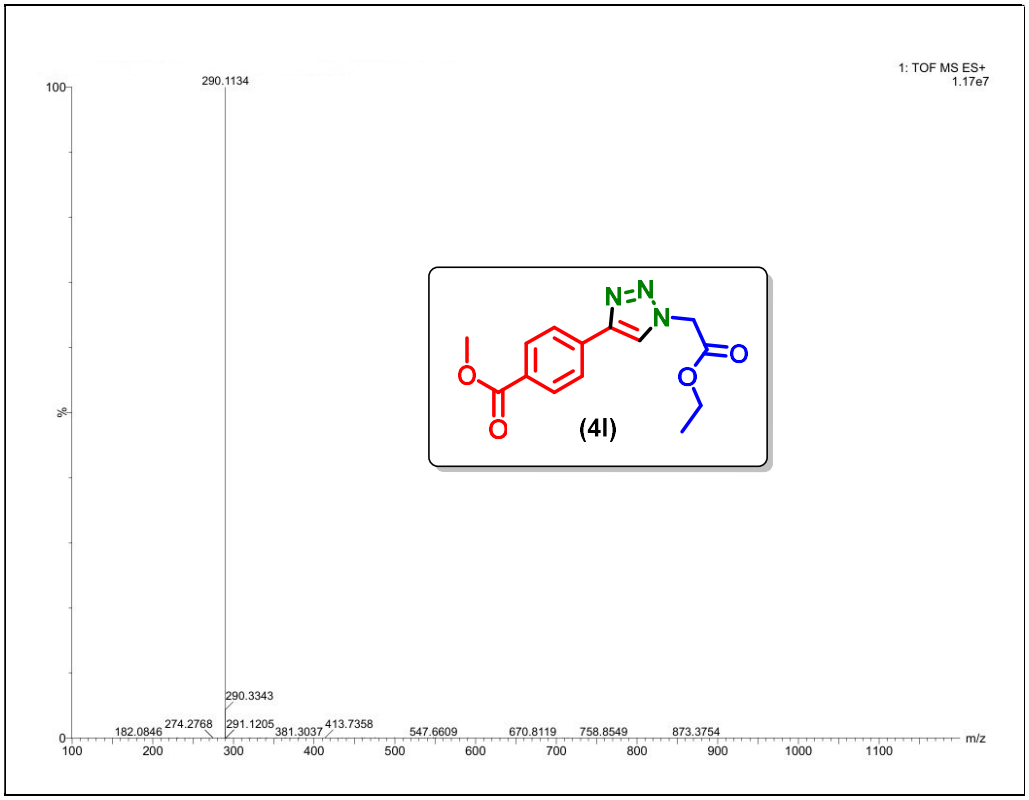


6. <u>HRMS spectra</u>:


1: TOF MS ES+
5.23e7
262.1185
100
%
0
262.3284
118.0453
230.2475
284.1020
353.2736
420.1588
545.2327
573.7867
693.1570
884.0770
947.5911
1049.8097
1156.1770
100
200
300
400
500
600
700
800
900
1000
1100
m/z
(4i)


1: TOF MS ES+
1.34e8
250.0981
100
%
0
250.3032
250.5075
251.0997
222.0664
251.3048
353.2730
381.3070
455.7073
593.7798
677.8344
702.4305
905.4233
945.4598
991.8735
1093.3926
1176.0122
100
200
300
400
500
600
700
800
900
1000
1100
m/z
F
(4j)


1: TOF MS ES+
1.80e8
308.1392
308.3670
308.5935
308.7787
309.0070
165.0677
280.1075
309.2328
402.2271
504.3368
576.2086
652.3206
721.7528
748.4134
898.7477
971.6184
1131.6323
m/z
(4k)


1: TOF MS ES+
1.17e7
290.1134
290.3343
182.0846
274.2768
291.1205
381.3037
413.7358
547.6609
670.8119
758.8549
873.3754
m/z
(4l)