Electronic Supplementary Material (ESI) for Chemical Communications.

Transition-Metal-Free Multinitrogenation of Amides by C-C Bond Cleavage: A New Approach to Tetrazoles

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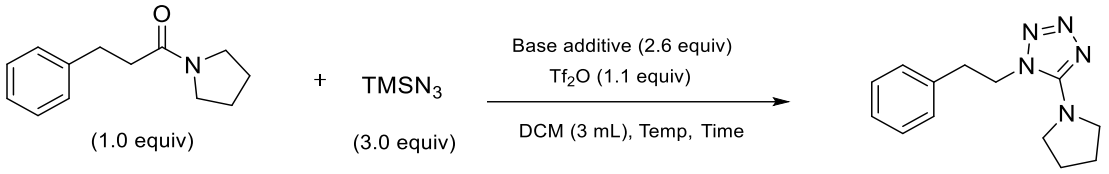
General information

Column chromatography was carried out on silica gel. **^{1}H NMR** spectra were recorded on 400 MHz in $CDCl_3$. **^{13}C NMR** spectra were recorded on 100 MHz in $CDCl_3$. Chemical shifts (ppm) were recorded with tetramethylsilane (TMS) as the internal reference standard. Multiplicities are given as s (singlet), d (doublet), t (triplet), dd (doublet of doublets), q (quartet), or m (multiplet). Their **^{1}H NMR** and **^{13}C NMR** spectra are provided in the Supporting Information. The **HRMS** was obtained using a Q-TOF instrument equipped with **ESI** source. Data collections for crystal structure were performed at room temperature (293 K) using Mo Kα radiation on a Bruker APEXII diffractometer. Melting points were measured with micro melting point apparatus.

The substituted amides were prepared according to the literature.[1] Trifluoromethanesulfonic anhydride (Tf_2O) was commercially available. Solvents were dried using standard methods. All commercially available reagents were used with further purification. Dichloromethane (DCM) was distilled over phosphorous pentoxide.

Optimization of the reaction conditions

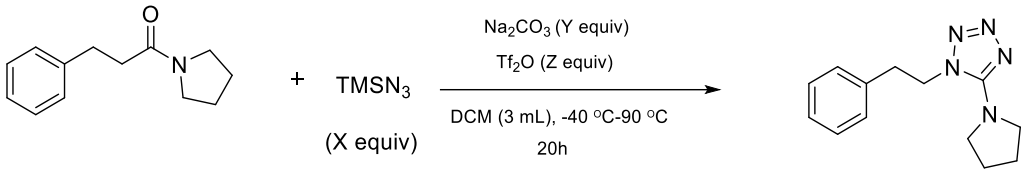
Table S1. Additional optimization of the reaction.[a]



Entry	Base additive	Temp(°C)	Time(h)	Solvent	Yield(%)[e]
1	2,6-lutidine[b]	-78-80	14	DCM	trace
2	2,6-dichloropyridine	-78-80	14	DCM	trace
3	2-methypyridine	-78-80	14	DCM	trace
4	2-chloropyridine	-78-80	14	DCM	trace
5	2-bromopyridine	-78-80	14	DCM	trace
6	2-iodopyridine	-78-80	14	DCM	trace
7	NaH	-78-80	14	DCM	41
8	Na_2CO_3	-78-80	14	DCM	50
9	K_2CO_3	-78-80	14	DCM	28
10	NaOAc	-78-80	14	DCM	46
11	$NaHCO_3$	-78-80	14	DCM	35
12	Na_2CO_3	-78-90	14	DCM	67
13	Na_2CO_3	-78-100	14	DCM	63
14	Na_2CO_3	-78-90	20	DCM	70
15	Na_2CO_3	-78-90	20	DCE	47
16	Na_2CO_3	-78-90	20	1,2-dibromoethane	60
17	Na_2CO_3	-78-90	20	toluene	trace
18	Na_2CO_3	-78-90	20	DMA	N,D
19	Na_2CO_3	-78-90	20	DMF	N,D
20	Na_2CO_3	-78-90	20	1,4-dioxane	N,D
21	Na_2CO_3	0-90[c]	20	DCM	N,D
22	Na_2CO_3	-40-90[d]	20	DCM	70

[a] Reaction conditions: To a mixture of amide **1a** (0.2 mmol, 1.0 equiv), $TMSN_3$ (3.0 equiv) and base additive (2.6 equiv) in DCM (3.0 mL) was added Tf_2O (1.1 equiv) at -78 °C under Ar atmosphere. After 20 min, the reaction mixture was stirred at reported temperature. [b] Base (2.0 equiv) was used. [c] Tf_2O was added at 0 °C. [d] Tf_2O was added at -40 °C. [e] Isolated yields. DCM=dichloromethane, DCE=1,2-dichloroethane, Tf=trifluoromethanesulfonyl, TMS=trimethylsilyl, DMA=dimethylacetamide, DMF=dimethyl formamide.

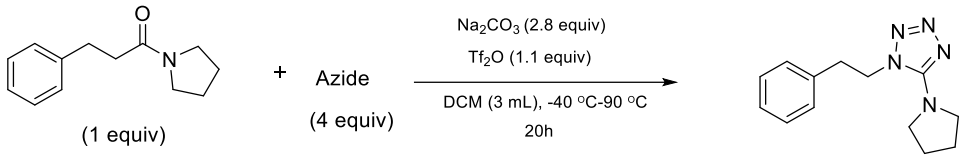
Table S2. Screening the ratio of the reagents of the reaction.[a]



Entry	$TMSN_3$ (X equiv)	Na_2CO_3 (Y equiv)	Tf_2O (Z equiv)	Yield[b] (%)
1	3	2.6	0.9	52
2	3	2.6	1.1	70
3	3	2.6	1.3	64
4	3	2.6	1.5	54
5	3	2.6	1.7	47
6	3	2.0	1.1	50
7	3	2.4	1.1	49
8	3	2.8	1.1	73
9	3	3.2	1.1	39
10	4	2.8	1.1	81
11	5	2.8	1.1	75
12	2	2.8	1.1	33

[a] Reaction conditions: To a mixture of amide **1a** (0.2 mmol, 1.0 equiv), $TMSN_3$ (X equiv) and base additive (Y equiv) in DCM (3.0 mL) was added Tf_2O (Z equiv) at -40 °C under Ar atmosohere. After 20 min, the reaction mixture was stirred at 90°C for 20 h. [b] Isolated yields.

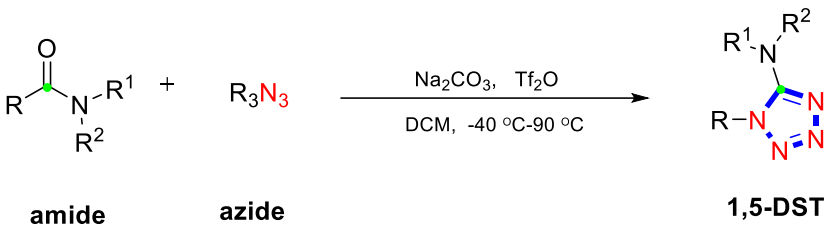
Table S3. Screening the azides of the reaction.[a]



entry	azide	yield(%)[b]
1	$TMSN_3$	81
2	NaN_3	33
3	TsN_3	N,D
4	(Azidomethyl)benzene	N,D
5	Diphenyl phosphorazidate	N,D
6	1-azidopentane	N,D
7	ethyl 2-azidoacetate	N,D

[a] Reaction conditions: To a mixture of amide (0.2 mmol, 1.0 equiv), azide (4.0 equiv) and base additive (2.8 equiv) in DCM (3.0 mL) was added Tf_2O (1.1 equiv) at -40 °C under Ar atmosphere. After 20 min, the reaction mixture was stirred at 90°C for 20 h. [b] Isolated yields.

General procedure for the synthesis of desired 1,5-DSTs

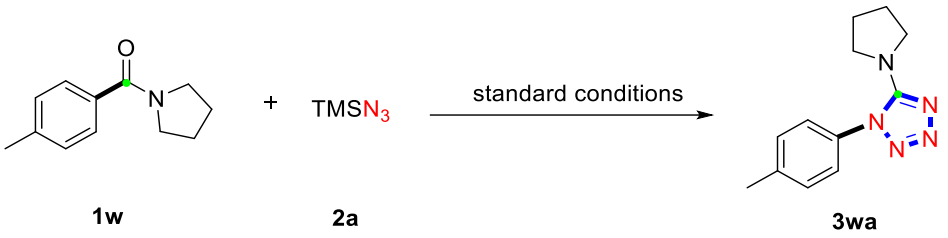


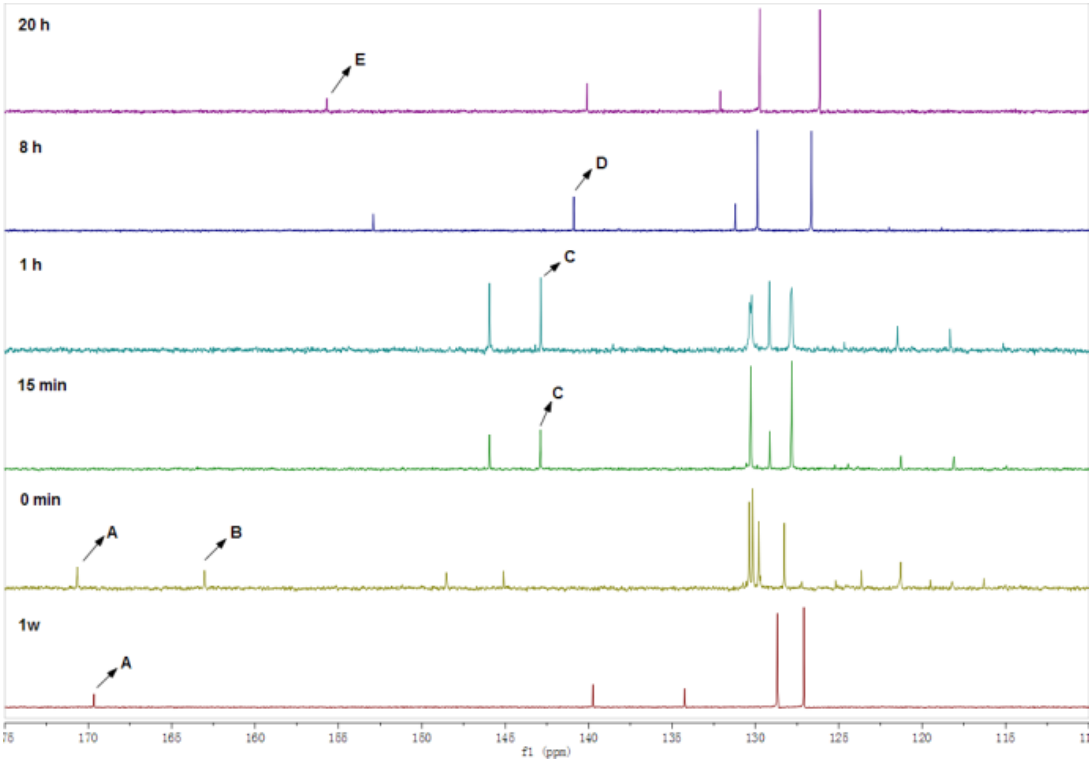
amide **azide** **1,5-DST**

The amide (0.2 mmol, 1.0 equiv), Na_2CO_3 (0.56 mmol) were added to a dried round bottom flask and put under Ar atmosphere. The azide (0.8 mmol), DCM (2.0 mL) were added and the solution was cooled to -40 °C, followed by addition of DCM (1.0 mL) solution of Tf_2O (0.22 mmol) via syringe. After 20 minutes, the reaction mixture was heated to 90 °C. After 20 hours, the mixture was quenched by the saturated $NaHCO_3$ solution and transferred to a separation funnel, diluted with DCM (15.0 mL) and the organic layer was washed with water (5.0 mL×2) and brine (5.0 mL), dried over anhydrous Na_2SO_4, concentrated in vacuum and subjected to column chromatography.

The monitoring experiment and NMR spectra

Transformation of 3wa monitored by ^{13}C NMR spectroscopy





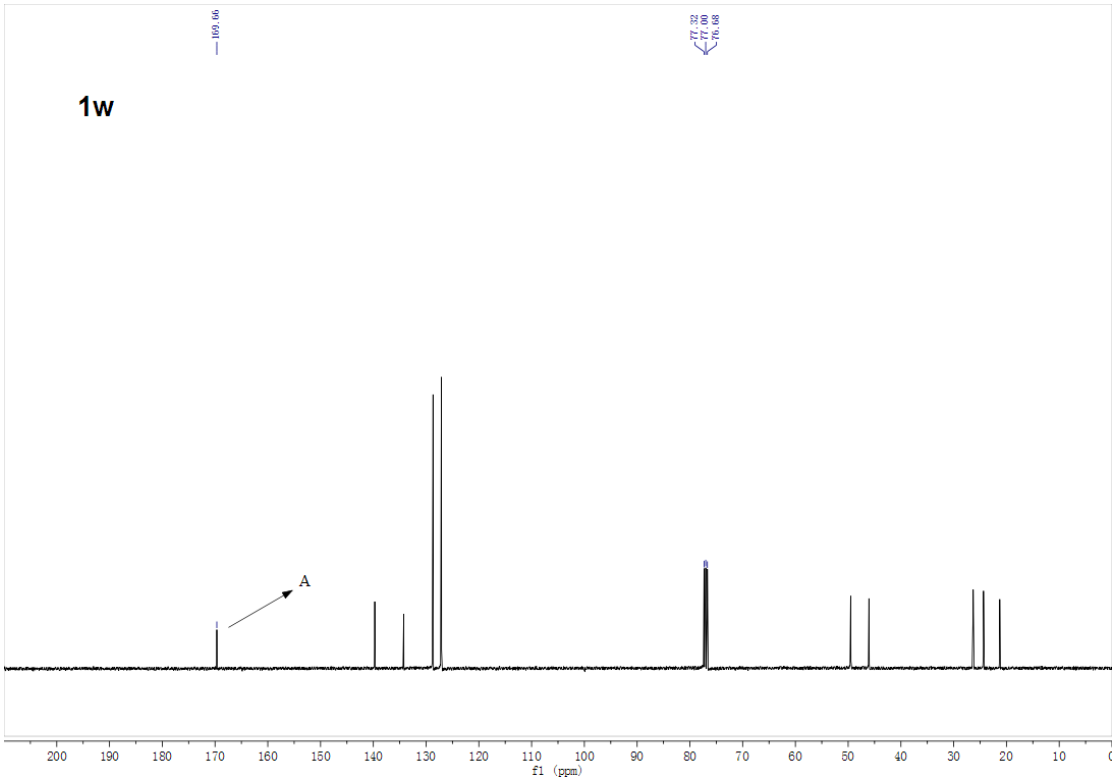
Transformation of **3wa** monitored by **^{13}C NMR** spectroscopy (**100MHz**, **$CDCl_3$**). **A** = **1w** carbonyl carbon, **B**, **C** and **D** = transforming signals assigned to original **1w** carbonyl carbon, **E** = **3wa** imine carbon.

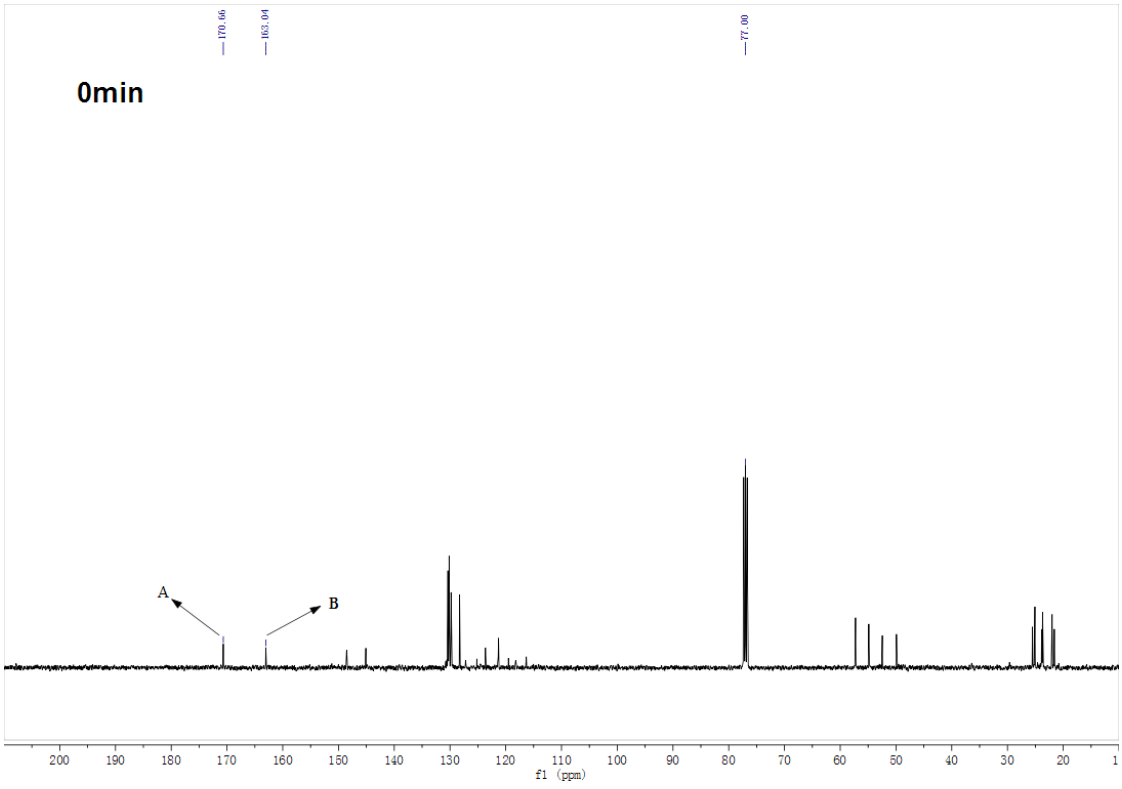
The first **^{13}C NMR** (100MHz, $CDCl_3$) was standard spectrum of substrate **1w**.

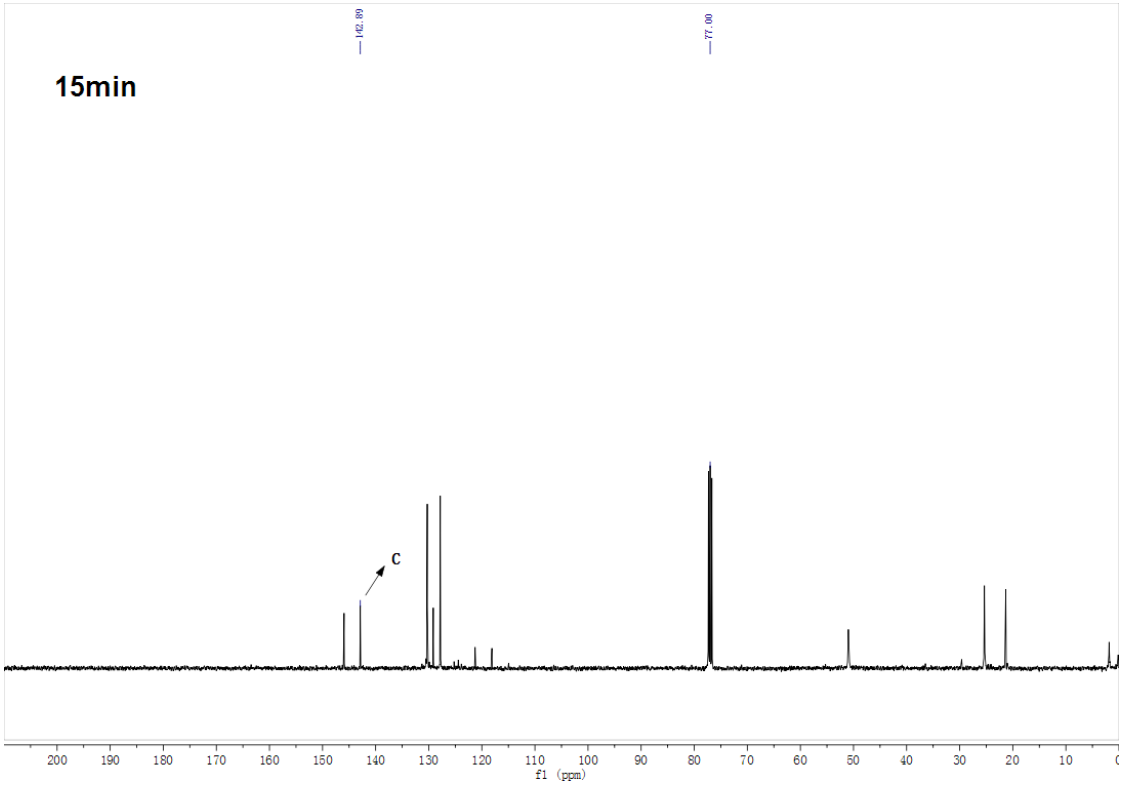
Four sets of the amide 1w (0.2 mmol, 1.0 equiv), Na_2CO_3 (0.56 mmol) were added to four dried round bottom flasks and put under Ar atmosphere, respectively. The $TMSN_3$ (0.8 mmol), DCM (2.0 mL) were each added to four flasks and the solutions were cooled to -40 °C, followed by addition of DCM (1.0 mL) solution of Tf_2O (0.22 mmol) via syringe, respectively. All of four reactions kept under -40 °C for 20 minutes. Then, the first reaction was stopped without heating and concentrated in vacuum. The mixture was added to NMR tube and the second **^{13}C NMR** was acquired. The second reaction was heated at 90 °C for 15 minutes and stopped and concentrated in vacuum. The mixture was added to NMR tube and the third **^{13}C NMR** was acquired. The third reaction was heated for 1 hour and stopped and concentrated in vacuum. The mixture was added to NMR tube and the fourth **^{13}C NMR** was acquired. The fourth reaction was heated for 8

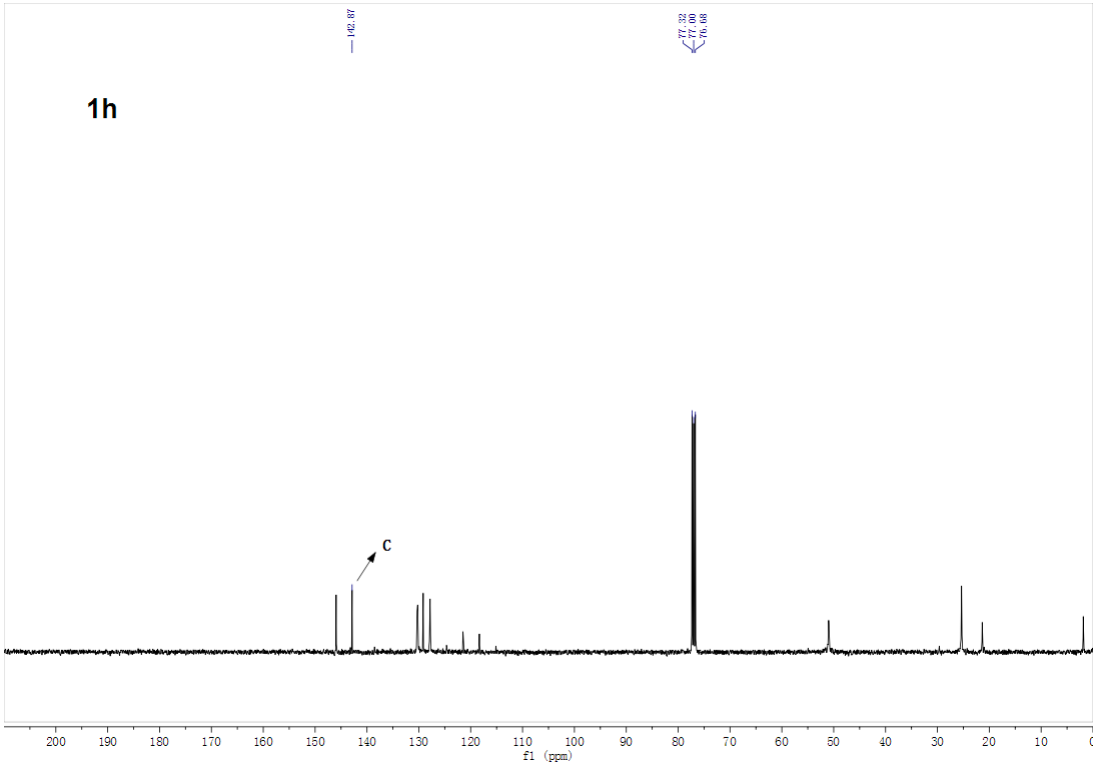
hours and stopped and concentrated in vacuum. The mixture was added to NMR tube and the fifth **^{13}C NMR** was acquired.

The sixth **^{13}C NMR** was standard spectrum of product **3wa**.

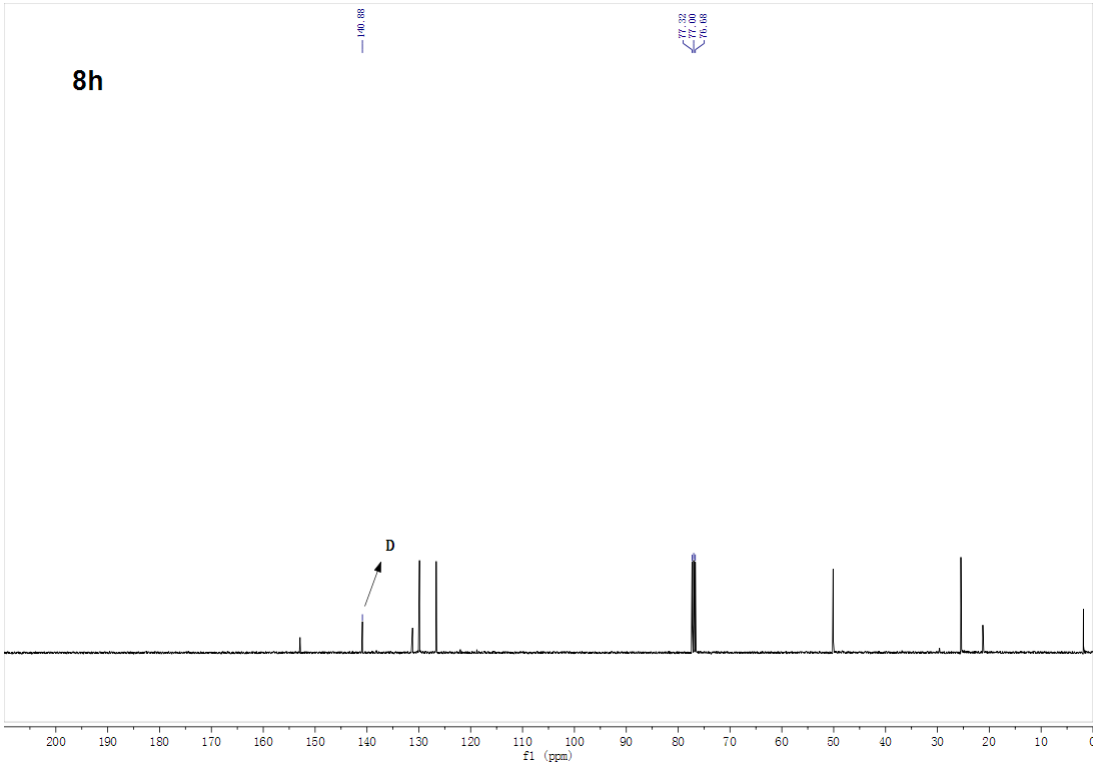





142.89
77.00
15min
C
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


142.87
77.32
77.00
76.68
1h
C
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

8h


140.88
77.32
77.00
76.68
D
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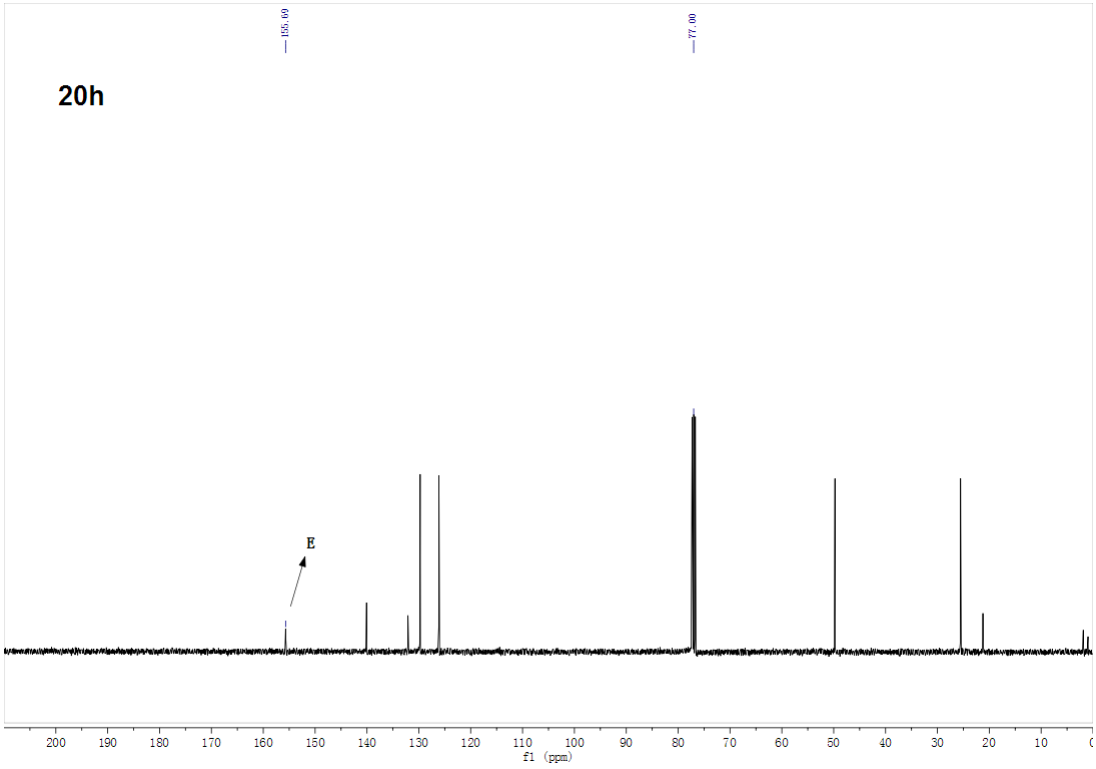
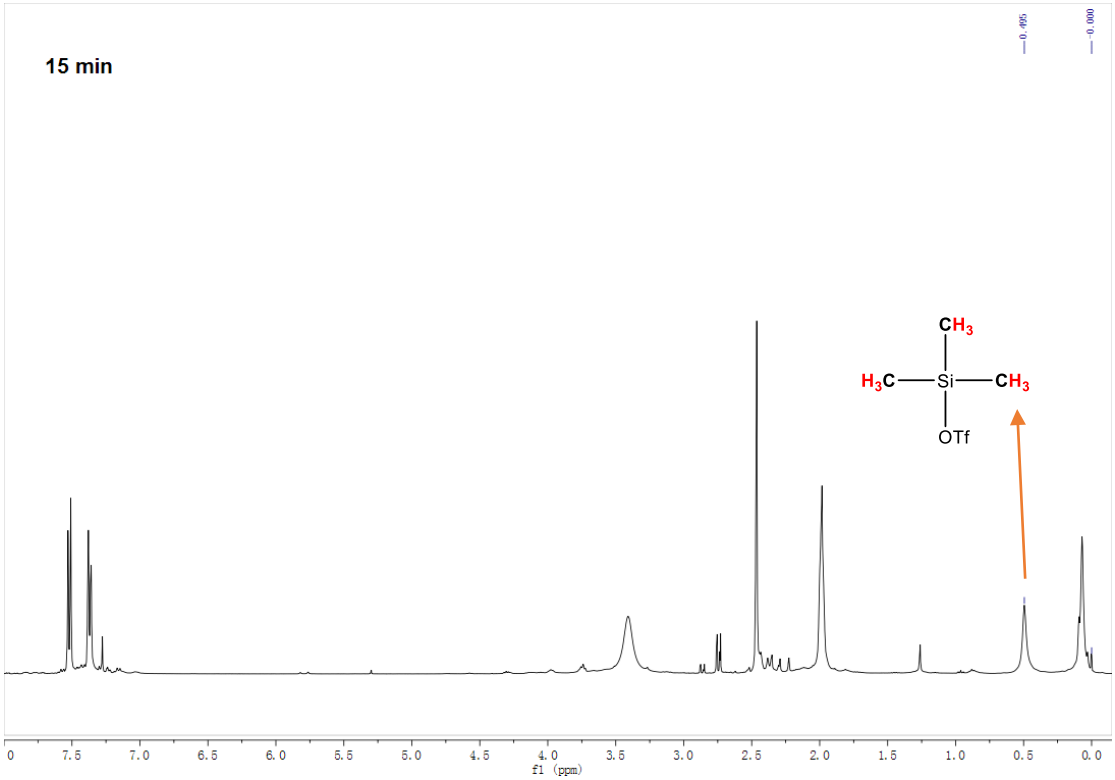
f1 (ppm)

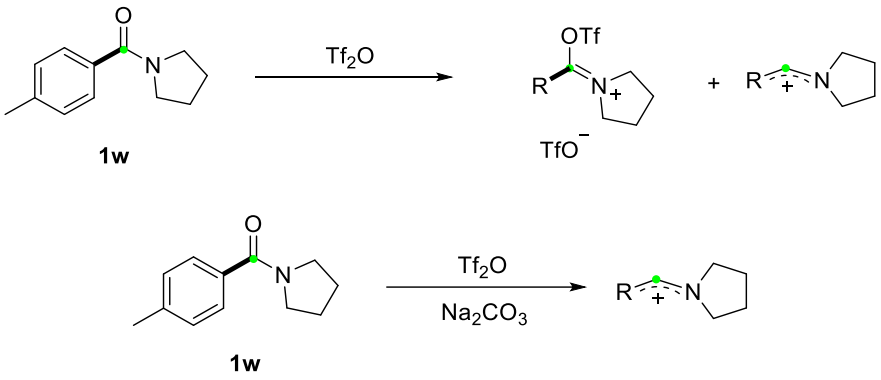
20h


155.69
77.00
E
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

TMSOTf signal has been documented in **^{1}H NMR**.[2]



The survey of the role of Na_2CO_3 and analysis: monitoring the transformation of 1w by ^{1}H NMR spectroscopy

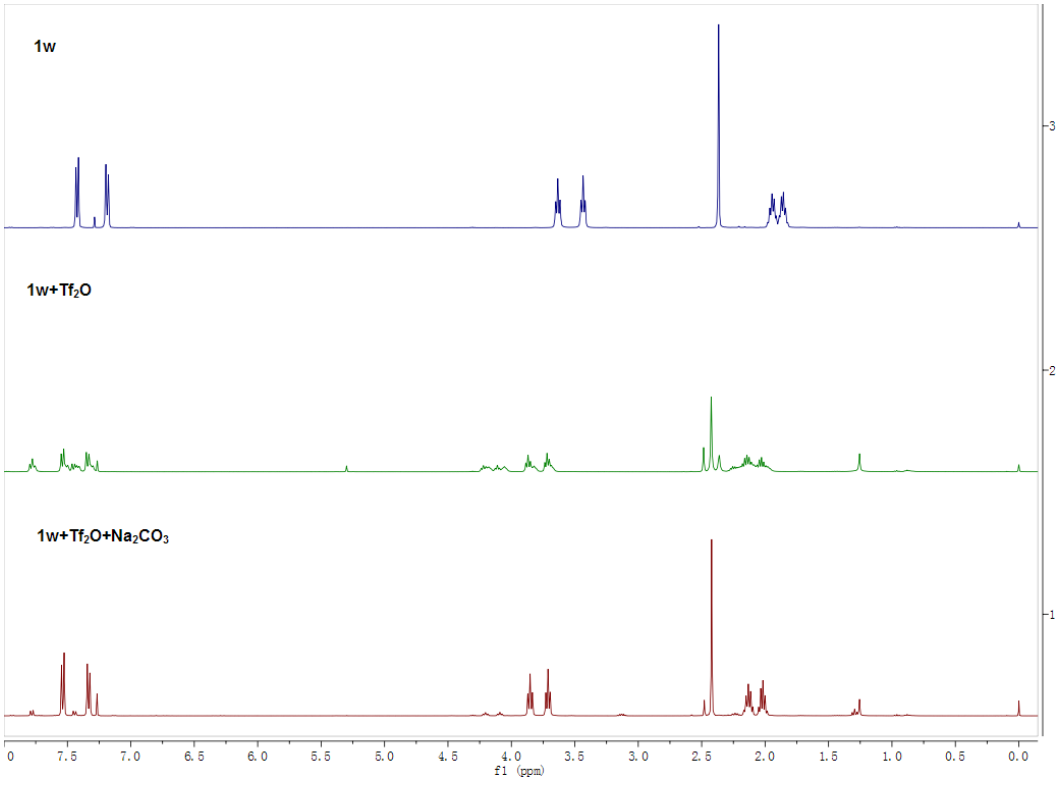


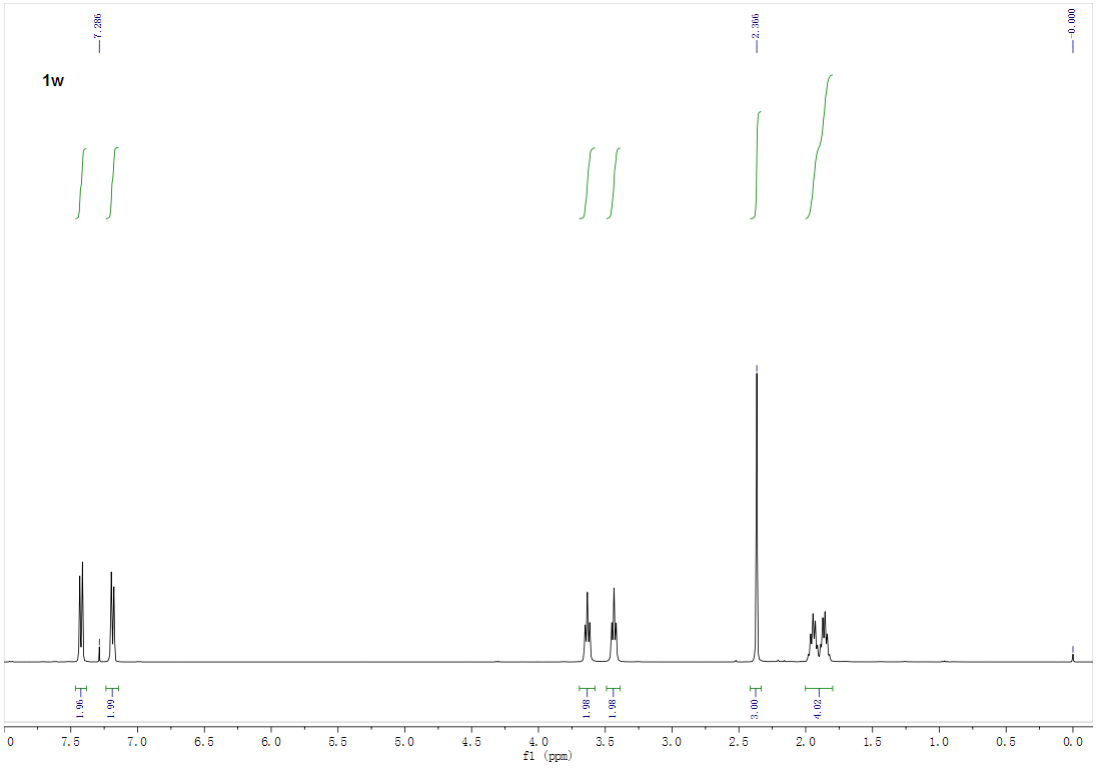
The amide **1w** (0.2 mmol, 1.0 equiv) was added to a dried round bottom flask and put under Ar atmosphere. The DCM (2.0 mL) was added to the flask and the solution was cooled to -40 °C, followed by addition of DCM (1.0 mL) solution of Tf_2O (0.22 mmol) via syringe. The reaction kept under -40 °C for 20 minutes. Then, the reaction was warmed to the room temperature and concentrated in vacuum. The mixture was added to NMR tube and the **^{1}H NMR** was acquired as 1w+Tf_2O.

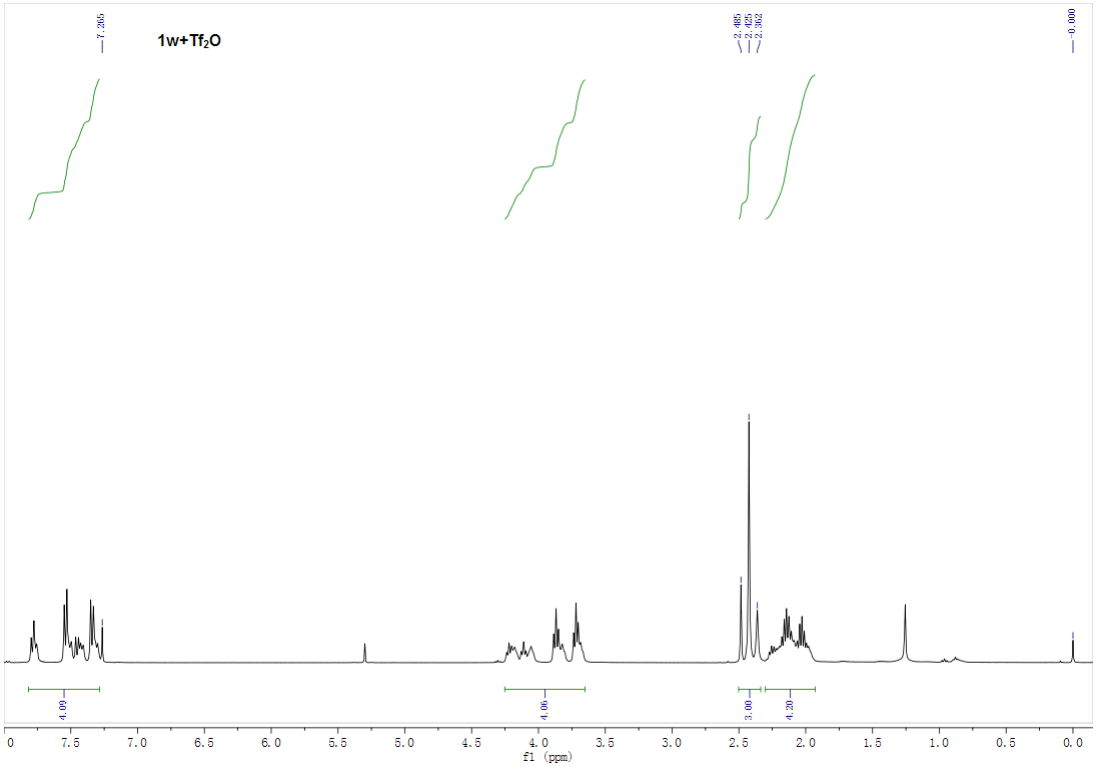
The amide **1w** (0.2 mmol, 1.0 equiv) and Na_2CO_3 (0.56 mmol) were added to a dried round bottom flask and put under Ar atmosphere. The DCM (2.0 mL) was added to the flask and the

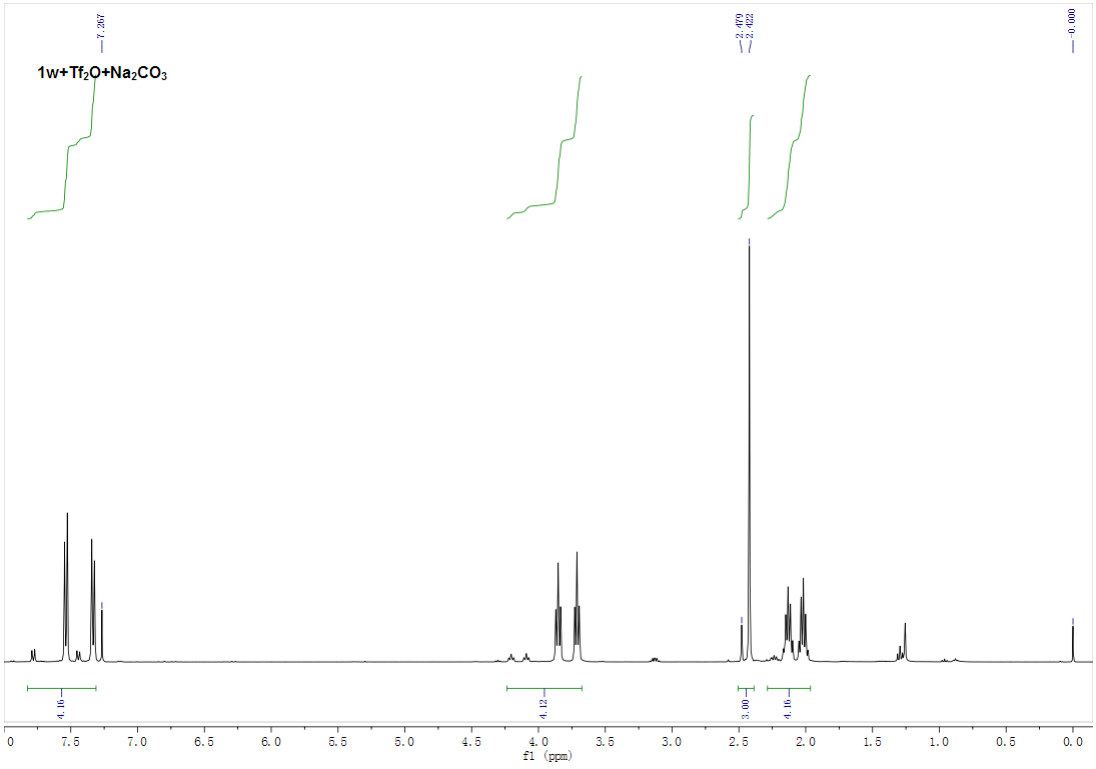
solution was cooled to -40 °C, followed by addition of DCM (1.0 mL) solution of Tf_2O (0.22 mmol) via syringe. The reaction kept under -40 °C for 20 minutes. Then, the reaction was warmed to the room temperature and concentrated in vacuum. The mixture was added to NMR tube and the **^{1}H NMR** was acquired as **1w**+Tf_2O+Na_2CO_3.

Analysis: Comparing the spectra of **1w**, **1w**+Tf_2O, **1w**+Tf_2O+Na_2CO_3, it was noticed that when the amide was treated with Tf_2O, the system was detected out three sets of CH_3 **^{1}H NMR** signals, and when the sets of CH_3 signals were integrated as 3H in total, the ratio of the CH_3 signals, the aryl part signals and the pyrrolidine part signals was perfectly integrated as 3:4:8. Treated with Tf_2O and Na_2CO_3, the system was detected out two sets of CH_3 **^{1}H NMR** signals, and when the sets of CH_3 signals were integrated as 3H in total, the aryl part signals and the pyrrolidine part signals were perfectly integrated as 4H and 8H as well. Therefore, it was assumed that the system of **1w**+Tf_2O turned into the mixture of **1w**, intermediate *O*-triflyliminium triflate, keteniminium ion, and the system of **1w**+Tf_2O+Na_2CO_3 turned into the mixture of **1w** and keteniminium ion. That showcased the influence of Na_2CO_3 to the reaction of Tf_2O-activation process, Na_2CO_3 could promote the transformation of *O*-triflyliminium triflate into keteniminium ion.

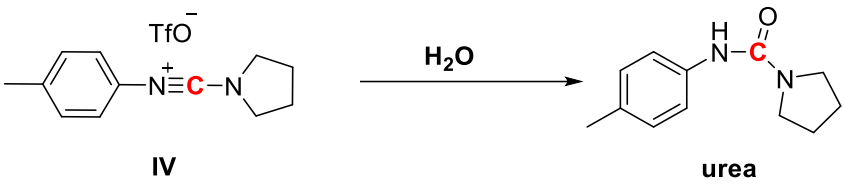



1w
7.286
2.306
0.000
1.96
1.99
1.98
1.98
3.00
4.02
f1 (ppm)


1w+Tf2O
7.295
2.485
2.425
2.362
0.000
4.09
4.06
3.00
4.20
f1 (ppm)

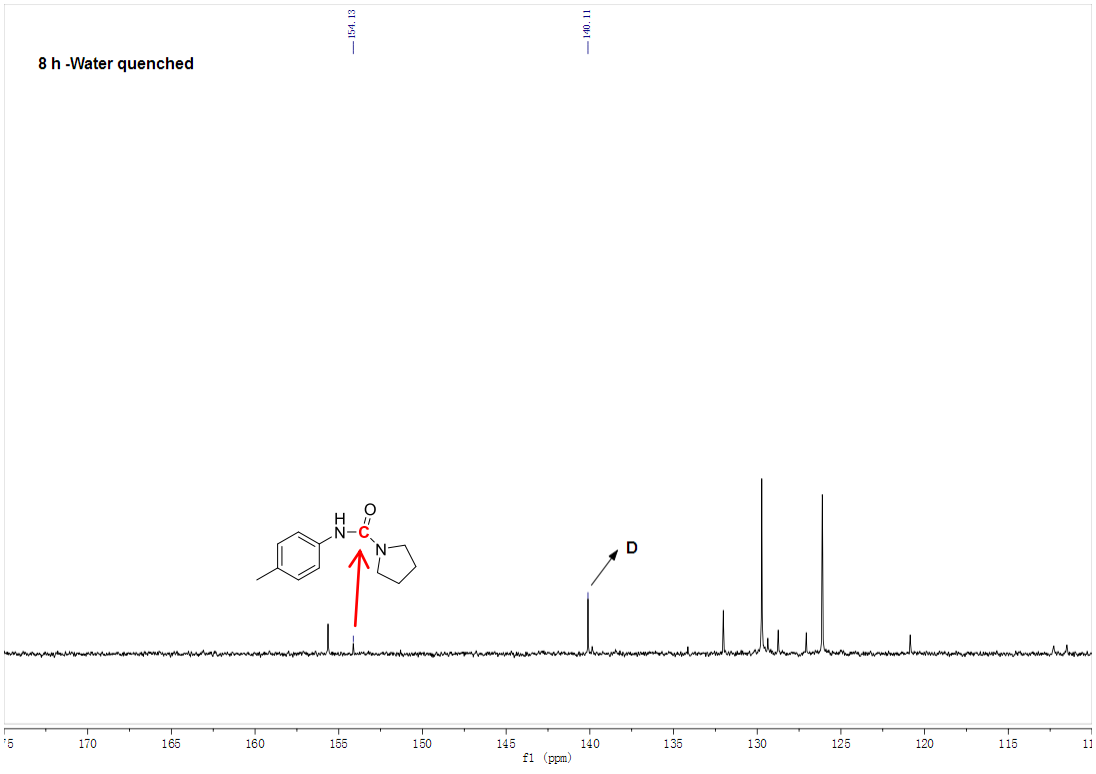


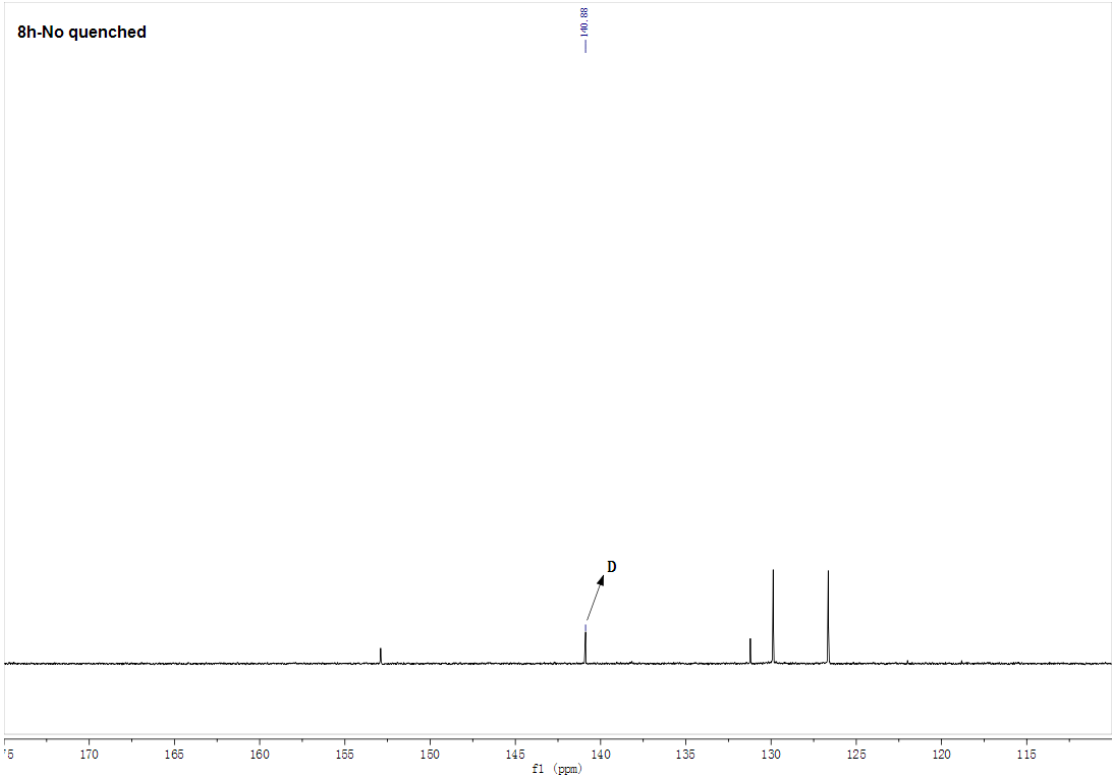
The survey of water-quenching intermediate IV and analysis

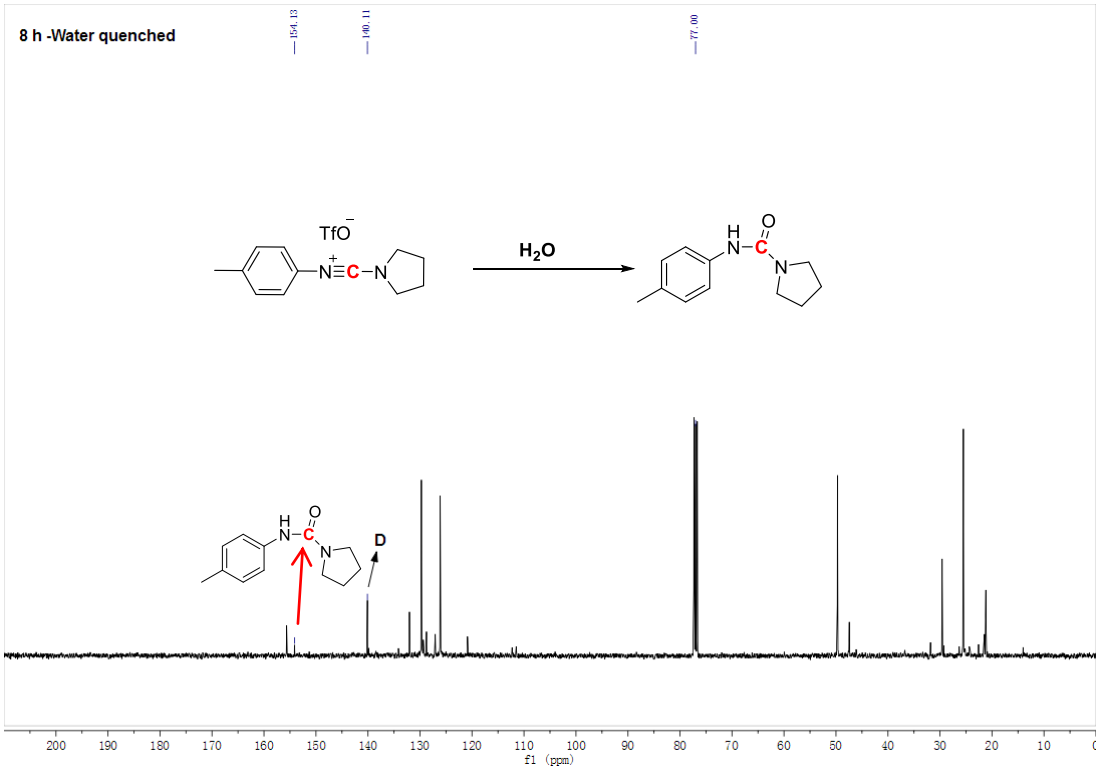


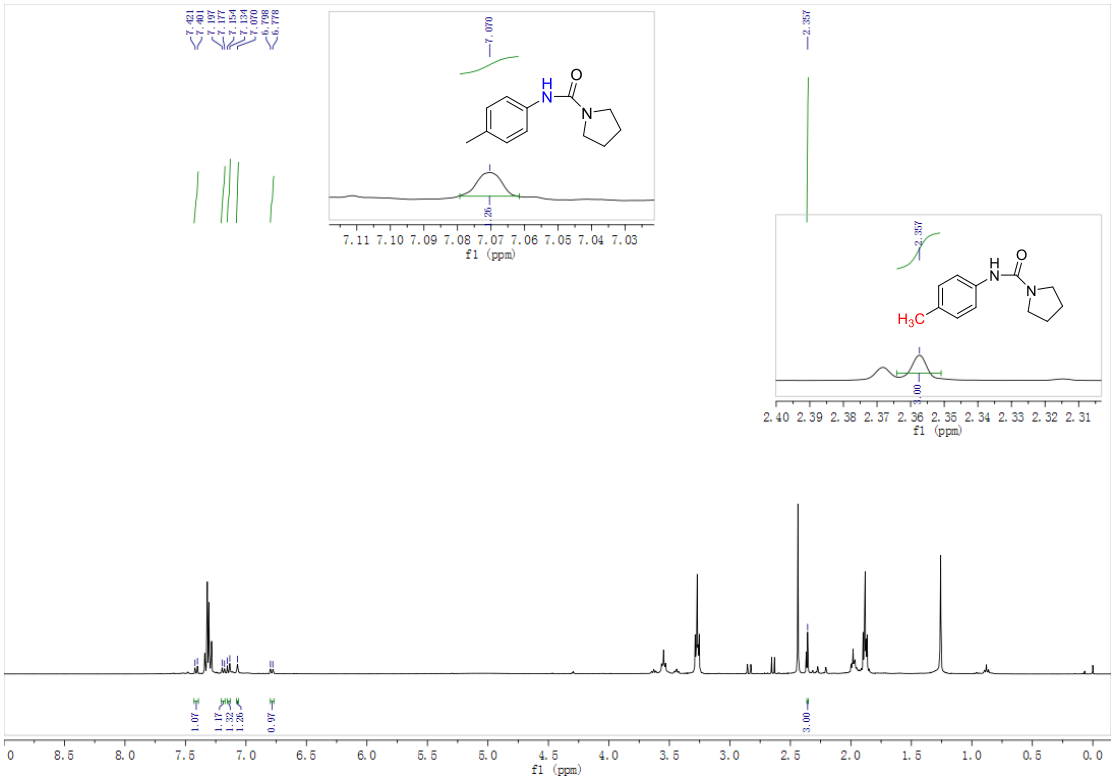
The amide **1w** (0.2 mmol, 1.0 equiv), Na_2CO_3 (0.56 mmol) were added to the dried round bottom flask and put under Ar atmosphere. The $TMSN_3$ (0.8 mmol), DCM (2.0 mL) were each added to flask and the solution was cooled to -40 °C, followed by addition of DCM (1.0 mL) solution of Tf_2O (0.22 mmol) via syringe. After 20 minutes, the reaction was heated for 8 hours and quenched by water, transferred to a separation funnel, diluted with DCM (15.0 mL) and the organic layer was washed with water (5.0 mL×2) and brine (5.0 mL), dried over anhydrous Na_2SO_4, concentrated in vacuum. The mixture was added to NMR tube and the **^{1}H NMR** and **^{13}C NMR** were acquired.

Analysis: From the spectra, it is clearly seen that after quenched by water, it could be found a new signal 154 ppm in 8 h-monitoring **^{13}C NMR** spectrum, pointed to the carbonyl carbon of urea, likewise, in the **^{1}H NMR** spectrum, it is also easy to pick up the signals of -NH-CO- and CH_3. It is the case that the intermediate IV could be led to a urea when the system has been quenched by water. The NMR spectra details of the same urea structure can be found in J. Org. Chem. 2018, 83, 913−920.


8 h -Water quenched
154.13
140.11
H
N
C
O
N
D
170
165
160
155
150
145
140
135
130
125
120
115
f1 (ppm)


8h-No quenched
140.88
D
170
165
160
155
150
145
140
135
130
125
120
115
f1 (ppm)

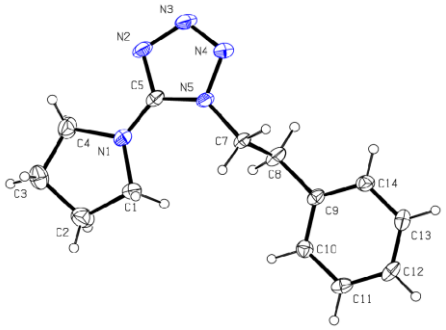

8 h -Water quenched
154.13
140.11
77.00
TfO⁻
H₂O
D
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

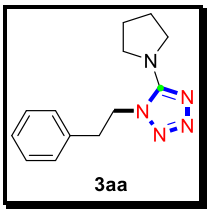

7.421
7.401
7.177
7.154
7.070
6.798
6.778
7.070
2.357
7.11 7.10 7.09 7.08 7.07 7.06 7.05 7.04 7.03
f1 (ppm)
2.40 2.39 2.38 2.37 2.36 2.35 2.34 2.33 2.32 2.31
f1 (ppm)
1.07
1.17
1.32
1.26
0.97
3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

X-ray structures of Tetrazoles: 3aa, 3ta, 3ua, 3Ra, 7aa

The crystal structure of product 3aa

Crystallorgraphic data for compound **3aa** (CCDC-1852258) has been deposited with Crystallorgraphic Data Centre, Copies of the data can be obtained, free of charge, on application to CCDC (Email: deposit@ccdc.cam.ac.uk)





The ellipsoid contour percent probability level is 30% in the caption of the thermal ellipsoid plot.

Bond precision: C-C = 0.0026 A Wavelength=1.54184

Cell: a=20.970(2) b=7.2564(6) c=17.3888(17)
alpha=90 beta=106.224(10) gamma=90

Temperature: 290 K

	Calculated	Reported
Volume	2540.6(4)	2540.6(4)
Space group	C 2/c	C 1 2/c 1
Hall group	-C 2yc	-C 2yc
Moiety formula	C13 H17 N5	C13 H17 N5
Sum formula	C13 H17 N5	C13 H17 N5
Mr	243.32	243.32
Dx,g cm-3	1.272	1.272
Z	8	8
Mu (mm-1)	0.644	0.644
F000	1040.0	1040.0
F000'	1042.82	
h,k,lmax	25,8,21	25,8,21
Nref	2409	2362
Tmin,Tmax	0.977,0.987	0.507,1.000
Tmin'	0.914	

Correction method= # Reported T Limits: Tmin=0.507 Tmax=1.000 AbsCorr = MULTI-SCAN

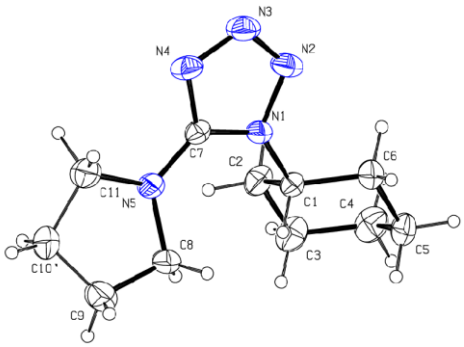
Data completeness= 0.980 Theta(max)= 69.990

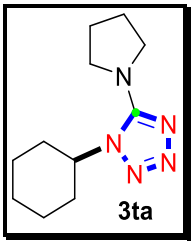
R(reflections)= 0.0453(1798) wR2(reflections)= 0.1257(2362)

S = 1.060 Npar= 163

The crystal structure of product 3ta

Crystallorgraphic data for compound **3ta** (CCDC-1852261) has been deposited with Crystallorgraphic Data Centre, Copies of the data can be obtained, free of charge, on application to CCDC (Email: deposit@ccdc.cam.ac.uk)





The ellipsoid contour percent probability level is 30% in the caption of the thermal ellipsoid plot.

Bond precision: C-C = 0.0037 A Wavelength=0.71073

Cell: a=6.8335(13) b=9.1979(15) c=10.0075(17)
alpha=94.814(14) beta=103.101(15) gamma=96.922(14)

Temperature: 293 K

	Calculated	Reported
Volume	604.13(19)	604.14(18)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C11 H19 N5	C11 H19 N5
Sum formula	C11 H19 N5	C11 H19 N5
Mr	221.31	221.31
Dx,g cm-3	1.217	1.217
Z	2	2
Mu (mm-1)	0.078	0.078
F000	240.0	240.0
F000'	240.06	
h,k,lmax	8,11,12	8,11,12
Nref	2378	2371
Tmin,Tmax	0.987,0.989	0.415,1.000
Tmin'	0.987	

Correction method= # Reported T Limits: Tmin=0.415 Tmax=1.000 AbsCorr = MULTI-SCAN

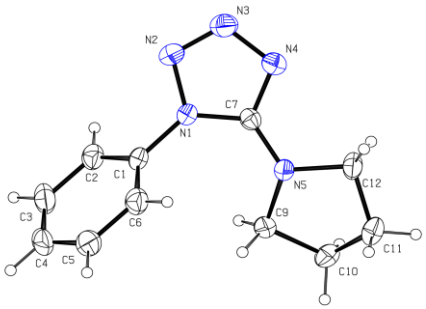
Data completeness= 0.997 Theta(max)= 26.020

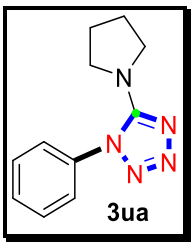
R(reflections)= 0.0627(1526) wR2(reflections)= 0.1699(2371)

S = 1.068 Npar= 162

The crystal structure of product 3ua

Crystallorgraphic data for compound **3ua** (CCDC-1852260) has been deposited with Crystallorgraphic Data Centre, Copies of the data can be obtained, free of charge, on application to CCDC (Email: deposit@ccdc.cam.ac.uk)





The ellipsoid contour percent probability level is 30% in the caption of the thermal ellipsoid plot.

Bond precision: C-C = 0.0048 A Wavelength=0.71073

Cell: a=10.2177(10) b=11.0976(11) c=19.154(2)

alpha=90 beta=90 gamma=90

Temperature: 189 K

	Calculated	Reported
Volume	2171.9(4)	2171.9(4)
Space group	P b c a	P b c a
Hall group	-P 2ac 2ab	-P 2ac 2ab
Moiety formula	C11 H13 N5	C11 H13 N5
Sum formula	C11 H13 N5	C11 H13 N5
Mr	215.26	215.26
Dx,g cm-3	1.317	1.317
Z	8	8
Mu (mm-1)	0.086	0.086
F000	912.0	912.0
F000'	912.24	
h,k,lmax	12,13,23	12,13,23
Nref	2139	2138
Tmin,Tmax	0.983,0.988	0.733,1.000
Tmin'	0.982	

Correction method= # Reported T Limits: Tmin=0.733 Tmax=1.000 AbsCorr = MULTI-SCAN

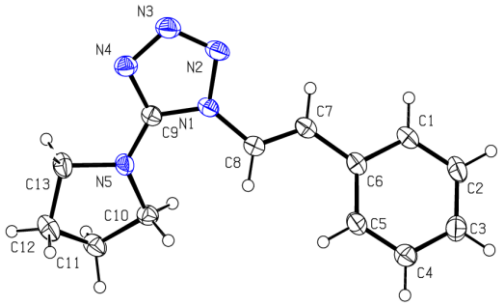
Data completeness= 1.000 Theta(max)= 26.010

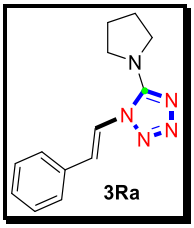
R(reflections)= 0.0621(1089) wR2(reflections)= 0.1694(2138)

S = 0.983 Npar= 145

The crystal structure of product 3Ra

Crystallorgraphic data for compound **3Ra** (CCDC-1852262) has been deposited with Crystallorgraphic Data Centre, Copies of the data can be obtained, free of charge, on application to CCDC (Email: deposit@ccdc.cam.ac.uk)





The ellipsoid contour percent probability level is 30% in the caption of the thermal ellipsoid plot.

Bond precision: C-C = 0.0024 A Wavelength=0.71073

Cell: a=12.0745(8) b=9.2802(7) c=11.0512(6)

alpha=90 beta=96.656(5) gamma=90

Temperature: 173 K

	Calculated	Reported
Volume	1229.98(14)	1229.98(14)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C13 H15 N5	C13 H15 N5
Sum formula	C13 H15 N5	C13 H15 N5
Mr	241.30	241.30
Dx,g cm-3	1.303	1.303
Z	4	4
Mu (mm-1)	0.084	0.084
F000	512.0	512.0
F000'	512.14	
h,k,lmax	14,11,13	14,11,13
Nref	2425	2421
Tmin,Tmax	0.987,0.990	0.786,1.000
Tmin'	0.987	

Correction method= # Reported T Limits: Tmin=0.786 Tmax=1.000 AbsCorr = MULTI-SCAN

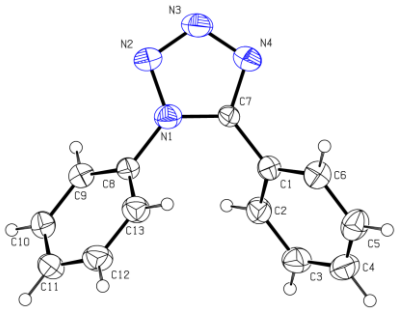
Data completeness= 0.998 Theta(max)= 26.020

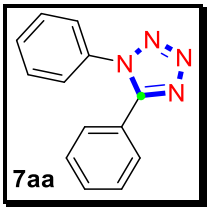
R(reflections)= 0.0459(1913) wR2(reflections)= 0.1112(2421)

S = 1.116 Npar= 163

The crystal structure of product 7aa

Crystallorgraphic data for compound **7aa** (CCDC-1852259) has been deposited with Crystallorgraphic Data Centre, Copies of the data can be obtained, free of charge, on application to CCDC (Email: deposit@ccdc.cam.ac.uk)





The ellipsoid contour percent probability level is 30% in the caption of the thermal ellipsoid plot.

Bond precision: C-C = 0.0039 A Wavelength=0.71073

Cell: a=10.7182(8) b=7.0388(5) c=14.6427(12)

alpha=90 beta=90.420(7) gamma=90

Temperature: 295 K

	Calculated	Reported
Volume	1104.66(15)	1104.66(14)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C13 H10 N4	C13 H10 N4
Sum formula	C13 H10 N4	C13 H10 N4
Mr	222.25	222.25
Dx,g cm-3	1.336	1.336
Z	4	4
Mu (mm-1)	0.085	0.085
F000	464.0	464.0
F000'	464.14	
h,k,lmax	13,8,18	13,8,18
Nref	2172	2163
Tmin,Tmax	0.986,0.990	0.386,1.000
Tmin'	0.986	

Correction method= # Reported T Limits: Tmin=0.386 Tmax=1.000 AbsCorr = MULTI-SCAN

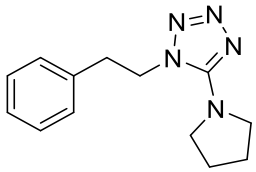
Data completeness= 0.996 Theta(max)= 26.010

R(reflections)= 0.0629(1401) wR2(reflections)= 0.1808(2163)

S = 1.057 Npar= 154

Characterization of compounds

(3aa) 1-phenethyl-5-(pyrrolidin-1-yl)-1H-tetrazole



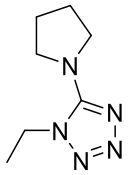
light yellow crystal, 39.4 mg, 81%, m.p. 63-64 °C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.17 (m, 3H), 7.14 – 7.00 (m, 2H), 4.46 (t, *J* = 7.2 Hz, 2H), 3.45 – 3.31 (m, 4H), 3.14 (t, *J* = 7.2 Hz, 2H), 2.09 – 1.77 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.0, 136.6, 128.5, 126.8, 49.1, 48.1, 36.0, 25.3.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{13}H_{17}N_5$) requires *m/z* 244.1557, found *m/z* 244.1555.

(3ba) 1-ethyl-5-(pyrrolidin-1-yl)-1H-tetrazole



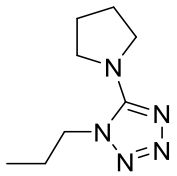
white crystal, 22.4 mg, 67%, m.p. 67-68 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.33 (q, *J* = 7.2 Hz, 2H), 3.64 – 3.51 (m, 4H), 2.08 – 1.98 (m, 4H), 1.49 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 49.6, 42.2, 25.52, 15.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_7H_{13}N_5$) requires *m/z* 168.1244, found *m/z* 168.1243.

(3ca) 1-propyl-5-(pyrrolidin-1-yl)-1H-tetrazole



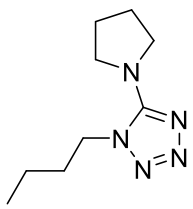
light yellow crystal, 24.9 mg, 69%, m.p. 33-34 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.23 (t, *J* = 7.2 Hz, 2H), 3.61 – 3.54 (m, 4H), 2.06 – 2.00 (m, 4H), 1.94 – 1.84 (m, 2H), 0.97 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 49.5, 48.6, 25.5, 23.1, 10.9.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_8H_{15}N_5$) requires *m/z* 182.1400, found *m/z* 182.1399.

(3da) 1-butyl-5-(pyrrolidin-1-yl)-1H-tetrazole



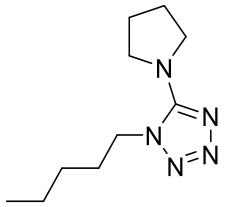
light yellow transparent liquid, 29.2 mg, 75%

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.26 (t, J = 7.2 Hz, 3H), 3.61 – 3.54 (m, 4H), 2.07 – 1.99 (m, 4H), 1.88 – 1.79 (m, 2H), 1.43 – 1.33 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 49.7, 46.9, 31.8, 25.6, 19.7, 13.5.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_9H_{17}N_5$) requires *m/z* 196.1557, found *m/z* 196.1555.

(3ea) 1-pentyl-5-(pyrrolidin-1-yl)-1H-tetrazole



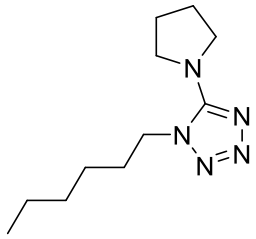
light yellow transparent liquid, 32.1 mg, 77%

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.25 (t, *J* = 6.4 Hz, 2H), 3.60 – 3.54 (m, 4H), 2.06 – 1.99 (m, 4H), 1.90 – 1.81 (m, 2H), 1.40 – 1.29 (m, 4H), 0.91 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 49.6, 47.1, 29.4, 28.5, 25.5, 22.0, 13.8.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{10}H_{19}N_5$) requires *m/z* 210.1713, found *m/z* 210.1714.

(3fa) 1-hexyl-5-(pyrrolidin-1-yl)-1H-tetrazole



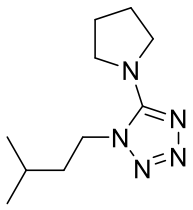
yellow oily liquid, 34.7 mg, 78%

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.28 – 4.22 (m, 2H), 3.60 – 3.54 (m, 4H), 2.06 – 2.00 (m, 4H), 1.89 – 1.80 (m, 2H), 1.38 – 1.26 (m, 6H), 0.93 – 0.84 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 49.6, 47.1, 31.1, 29.7, 26.0, 25.5, 22.3, 13.8.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{21}N_5$) requires *m/z* 224.1870, found *m/z* 224.1871.

(3ga) 1-isopentyl-5-(pyrrolidin-1-yl)-1H-tetrazole



light yellow transparent liquid, 30.1, 72%

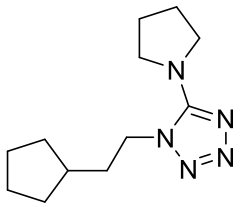
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.31 – 4.24 (m, 2H), 3.61 – 3.54 (m, 4H), 2.07 – 2.00 (m, 4H), 1.78 – 1.70 (m, 2H), 1.69 – 1.59 (m, 1H), 0.97 (d, *J* = 6.4 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 49.6, 45.6, 38.4, 25.6, 25.5, 22.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{10}H_{19}N_5$) requires *m/z* 210.1713, found *m/z*

210.1714.

(3ha) 1-(2-cyclopentylethyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



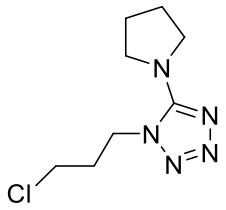
light yellow liquid, 32.0 mg, 68%

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.31 – 4.24 (m, 2H), 3.62 – 3.53 (m, 4H), 2.07 – 1.99 (m, 4H), 1.90 – 1.72 (m, 5H), 1.69 – 1.48 (m, 4H), 1.18 – 1.07 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 49.6, 46.6, 37.2, 35.9, 32.3, 25.5, 24.9.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{21}N_5$) requires *m/z* 236.1870, found *m/z* 236.1868.

(3ia) 1-(2-chloroethyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



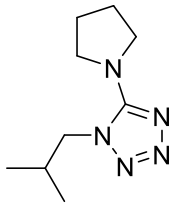
white powder, 23.2 mg, 54%, m.p. 39-40^{o}C.

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.46 (t, *J* = 7.2 Hz, 2H), 3.65 – 3.57 (m, 6H), 2.43 – 2.33 (m, 2H), 2.08 – 2.00 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.4, 49.7, 44.1, 41.3, 32.1, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_7H_{12}ClN_5$) requires *m/z* 216.1010, found *m/z* 216.1011.

(3ja) 1-isobutyl-5-(pyrrolidin-1-yl)-1H-tetrazole



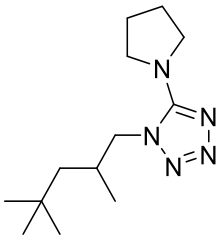
white crystal, 29.2 mg, 75%, m.p. 60-61 ^{o}C

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.07 (d, *J* = 7.2 Hz, 2H), 3.60 – 3.53 (m, 4H), 2.26 – 2.15 (m, 1H), 2.07 – 1.99 (m, 4H), 0.95 (d, *J* = 6.8 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.4, 54.1, 49.6, 29.0, 25.5, 19.7.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_9H_{17}N_5$) requires *m/z* 196.1557, found *m/z* 196.1556.

(3ka) 5-(pyrrolidin-1-yl)-1-(2,4,4-trimethylpentyl)-1H-tetrazole



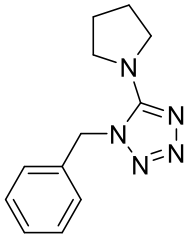
light yellow crystal, 36.1 mg, 72%, m.p. 64-66 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.12 (dd, *J* = 14.6, 7.6 Hz, 1H), 4.00 (dd, *J* = 14.0, 8.0 Hz, 1H), 3.59 – 3.52 (m, 4H), 2.17 – 2.09 (m, 1H), 2.06 – 2.00 (m, 4H), 1.26 (dd, *J* = 14.0, 2.8 Hz, 1H), 1.13 (dd, *J* = 14.1, 7.0 Hz, 1H), 0.94 (d, *J* = 6.4 Hz, 3H), 0.85 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 54.1, 49.6, 47.5, 30.8, 30.1, 29.6, 25.5, 20.0.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{13}H_{25}N_5$) requires *m/z* 252.2183, found *m/z* 252.2182.

(3la) 1-benzyl-5-(pyrrolidin-1-yl)-1H-tetrazole



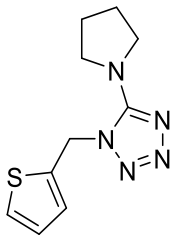
yellow crystal, 24.1 mg, 53%, m.p. 91-93 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.25 – 4.14 (m, 1H), 3.60 – 3.51 (m, 4H), 2.07 – 1.90 (m, 10H), 1.78 – 1.70 (m, 1H), 1.44 – 1.23 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.0, 135.3, 129.0, 128.2, 126.2, 50.2, 49.4, 25.5.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{15}N_5$) requires *m/z* 230.1400, found *m/z* 230.1395.

(3ma) 5-(pyrrolidin-1-yl)-1-(thiophen-2-ylmethyl)-1H-tetrazole



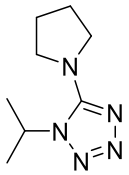
light yellow crystal, 7.5 mg, 16%, m.p. 81-82 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.25 (m, 1H), 6.98 – 6.91 (m, 2H), 5.63 (s, 2H), 3.60 – 3.55 (m, 4H), 2.00 – 1.95 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.7, 137.1, 127.2, 126.5, 126.2, 49.6, 45.7, 25.7.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{10}H_{13}N_5S$) requires *m/z* 236.0964, found *m/z* 236.0965.

(3na) 1-isopropyl-5-(pyrrolidin-1-yl)-1H-tetrazole



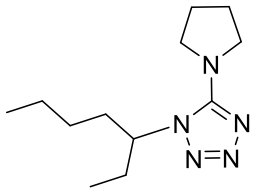
white crystal, 19.2 mg, 53%, m.p. 62-64 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.66 (hept, J = 6.6 Hz, 1H), 3.61 – 3.51 (m, 4H), 2.07 – 1.97 (m, 4H), 1.58 (d, J = 6.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 49.9, 25.5, 22.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_8H_{15}N_5$) requires m/z 182.1400, found m/z 182.1398.

(3oa) 1-(heptan-3-yl)-5-(pyrrolidin-1-yl)-1H-tetrazole



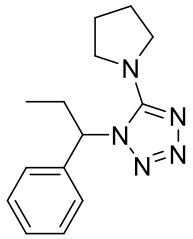
light yellow transparent liquid, 35.1mg, 74%

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.30 – 4.21 (m, 1H), 3.60 – 3.51 (m, 4H), 2.08 – 1.97 (m, 6H), 1.93 – 1.79 (m, 2H), 1.33 – 1.24 (m, 2H), 1.23 – 1.06 (m, 2H), 0.89 – 0.82 (m, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 157.4, 60.3, 50.1, 34.5, 28.2, 28.1, 25.5, 22.3, 13.7, 10.4.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{23}N_5$) requires m/z 238.2026, found m/z 238.2028.

(3pa) 1-(1-phenylpropyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



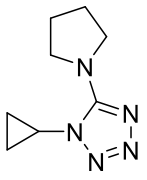
light yellow solid, 34.4 mg, 67%, m.p. 75-77 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.28 (m, 5H), 5.26 (dd, J = 8.8, 6.0 Hz, 1H), 3.61 – 3.54 (m, 2H), 3.46 – 3.39 (m, 2H), 2.61 – 2.48 (m, 1H), 2.28 – 2.17 (m, 1H), 1.98 – 1.87 (m, 4H), 0.93 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 157.3, 139.9, 128.8, 128.1, 126.4, 64.1, 50.2, 30.6, 25.5, 11.4.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{14}H_{19}N_5$) requires m/z 258.1713, found m/z 258.1713.

(3qa) 1-cyclopropyl-5-(pyrrolidin-1-yl)-1H-tetrazole



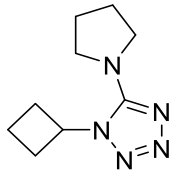
white crystal, 27.2 mg, 76%, m.p. 117-119 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.73 – 3.67 (m, 4H), 3.51 – 3.44 (m, 1H), 2.06 – 1.99 (m, 4H), 1.34 – 1.27 (m, 2H), 1.21 – 1.14 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 157.2, 49.4, 28.0, 25.5, 8.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_8H_{13}N_5$) requires m/z 180.1243, found m/z 180.1244.

(3ra) 1-cyclobutyl-5-(pyrrolidin-1-yl)-1H-tetrazole



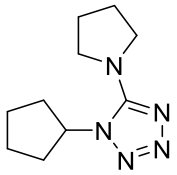
white crystal, 28.9 mg, 75%, m.p. 67-69 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.87 – 4.78 (m, 1H), 3.58 – 3.51 (m, 4H), 2.88 – 2.76 (m, 2H), 2.52 – 2.42 (m, 2H), 2.04 – 1.94 (m, 5H), 1.92 – 1.83 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.1, 51.2, 49.9, 30.4, 25.5, 14.9.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_9H_{15}N_5$) requires *m/z* 194.1400, found *m/z* 194.1399.

(3sa) 1-cyclopentyl-5-(pyrrolidin-1-yl)-1H-tetrazole



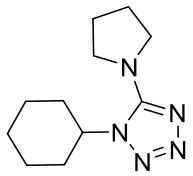
white crystal, 30.6 mg, 74%, m.p. 73-74 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.82 – 4.73 (m, 1H), 3.61 – 3.53 (m, 4H), 2.21 – 2.07 (m, 4H), 2.05 – 1.92 (m, 6H), 1.77 – 1.65 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 58.5, 50.0, 33.3, 25.5, 24.4.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{10}H_{17}N_5$) requires *m/z* 208.1557, found *m/z* 208.1556.

(3ta) 1-cyclohexyl-5-(pyrrolidin-1-yl)-1H-tetrazole



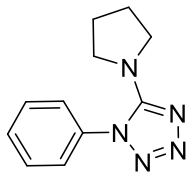
white crystal, 31.3 mg, 71%, m.p. 102-104 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.24 – 4.15 (m, 1H), 3.60 – 3.51 (m, 4H), 2.07 – 1.90 (m, 10H), 1.78 – 1.70 (m, 1H), 1.44 – 1.23 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 57.3, 50.0, 32.9, 25.5, 25.4, 24.9.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{19}N_5$) requires *m/z* 222.1713, found *m/z* 222.1709.

(3ua) 1-phenyl-5-(pyrrolidin-1-yl)-1H-tetrazole



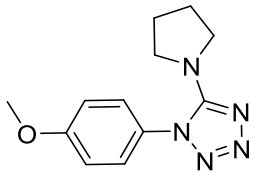
white crystal, 33.1 mg, 77%, m.p. 134-135 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.44 (m, 5H), 3.31 – 3.25 (m, 4H), 1.92 – 1.87 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.6, 134.6, 129.7, 129.1, 126.2, 49.8, 25.5.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{13}N_5$) requires *m/z* 216.1244, found *m/z* 216.1244.

(3va) 1-(4-methoxyphenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



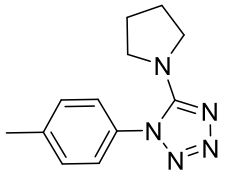
white crystal, 44.1 mg, 90%, m.p. 81-82 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.34 (m, 2H), 7.02 – 6.97 (m, 2H), 3.87 (s, 3H), 3.31 – 3.24 (m, 4H), 1.92 – 1.85 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 160.4, 155.6, 127.8, 127.2, 114.2, 55.6, 49.6, 25.5.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{15}N_5O$) requires *m/z* 246.1349, found *m/z* 246.1348.

(3wa) 5-(pyrrolidin-1-yl)-1-(p-tolyl)-1H-tetrazole



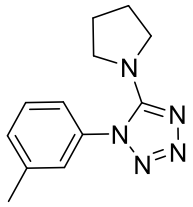
light yellow oily liquid, 38.0 mg, 83%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.28 (m, 4H), 3.31 – 3.24 (m, 4H), 2.44 (s, 3H), 1.92 – 1.85 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.6, 140.0, 132.1, 129.7, 126.1, 49.7, 25.5, 21.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{15}N_5$) requires *m/z* 230.1400, found *m/z* 230.1400.

(3xa) 5-(pyrrolidin-1-yl)-1-(m-tolyl)-1H-tetrazole



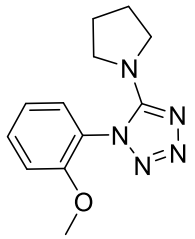
light yellow crystal, 35.7 mg, 78%, m.p. 81-82 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.36 (m, 1H), 7.33 – 7.27 (m, 2H), 7.26 – 7.23 (m, 1H), 3.31 – 3.26 (m, 4H), 2.43 (s, 3H), 1.92 – 1.87 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.6, 139.5, 134.5, 130.5, 128.9, 126.7, 123.2, 49.8, 25.5, 21.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{15}N_5$) requires *m/z* 230.1400, found *m/z* 230.1400.

(3ya) 1-(2-methoxyphenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



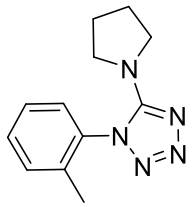
light yellow crystal, 32.3 mg, 66%, m.p. 93-95 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 – 7.47 (m, 1H), 7.37 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.08 – 7.02 (m, 2H), 3.81 (s, 3H), 3.30 – 3.24 (m, 4H), 1.89 – 1.84 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.7, 155.4, 131.9, 129.4, 123.6, 120.6, 111.8, 55.9, 48.7, 25.5.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{15}N_5O$) requires *m/z* 246.1349, found *m/z* 246.1350.

(3za) 5-(pyrrolidin-1-yl)-1-(o-tolyl)-1H-tetrazole



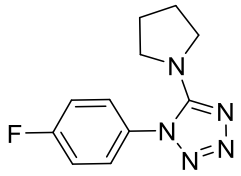
light yellow crystal, 25.6 mg, 56%, m.p. 103-104 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.41 (m, 1H), 7.37 – 7.31 (m, 3H), 3.26 – 3.19 (m, 4H), 2.14 (s, 3H), 1.90 – 1.85 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.5, 136.3, 133.8, 130.8, 130.5, 128.0, 126.7, 49.1, 25.5, 17.4.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{15}N_5$) requires *m/z* 230.1400, found *m/z* 230.1399.

(3Aa) 1-(4-fluorophenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



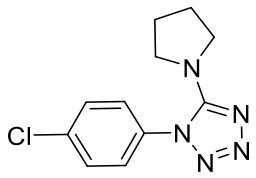
light yellow crystal, 38.2 mg, 82%, m.p. 148-150 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.44 (m, 2H), 7.25 – 7.18 (m, 2H), 3.32 – 3.25 (m, 4H), 1.94 – 1.89 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 161.7, 155.7, 130.7 (d, *J* = 3.0 Hz), 128.3 (d, *J* = 9.0 Hz), 116.3 (d, *J* = 23.0 Hz), 49.7, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{12}FN_5$) requires *m/z* 234.1150, found *m/z* 234.1150.

(3Ba) 1-(4-chlorophenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



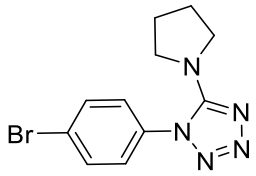
white crystal, 26.3 mg, 53%, m.p. 105-107 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.48 (m, 2H), 7.46 – 7.40 (m, 2H), 3.32 – 3.26 (m, 4H), 1.95 – 1.89 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.7, 135.8, 133.2, 129.5, 127.4, 50.0, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{12}ClN_5$) requires *m/z* 250.0584, found *m/z* 250.0587.

(3Ca) 1-(4-bromophenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



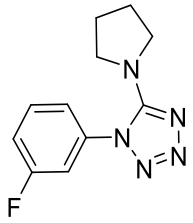
light yellow crystal, 46.8 mg, 80%, m.p. 137-138 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 – 7.64 (m, 2H), 7.39 – 7.34 (m, 2H), 3.32 – 3.27 (m, 4H), 1.94 – 1.89 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.7, 133.7, 132.5, 127.6, 123.8, 50.1, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{12}BrN_5$) requires *m/z* 294.0349, found *m/z* 294.0350.

(3Da) 1-(3-fluorophenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



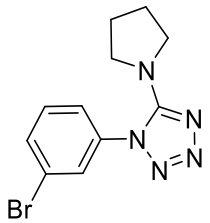
white crystal, 34.1 mg, 73%, m.p. 167-169°C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.48 (m, 1H), 7.34 – 7.19 (m, 3H), 3.39 – 3.22 (m, 4H), 2.00 – 1.87 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4 (d, *J* = 249.0 Hz), 155.7, 135.8 (d, *J* = 10.0 Hz), 130.5 (d, *J* = 8.0 Hz), 121.9 (d, *J* = 4.0 Hz), 116.9 (d, *J* = 21.0 Hz), 113.7 (d, *J* = 24.0 Hz), 50.0, 25.5.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{12}FN_5$) requires *m/z* 234.1150, found *m/z* 234.1150.

(3Ea) 1-(3-bromophenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



white crystal, 40.4 mg, 69%, m.p. 104°C

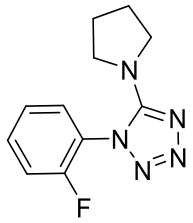
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 – 7.63 (m, 2H), 7.46 – 7.38 (m, 2H), 3.33 – 3.28 (m, 4H), 1.97 – 1.90 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.6, 135.7, 132.8, 130.4, 129.1, 124.7, 122.6, 50.1, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{12}BrN_5$) requires *m/z* 294.0349, found *m/z*

294.0351.

(3Fa) 1-(2-fluorophenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



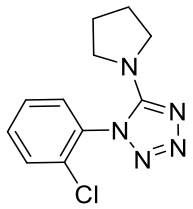
white crystal, 28.8 mg, 62%, m.p. 110-112 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 – 7.47 (m, 2H), 7.38 – 7.27 (m, 2H), 3.31 (t, *J* = 6.7 Hz, 4H), 1.95 – 1.86 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ158.6, 156.1, 155.6, 132.3 (d, *J* = 8.0 Hz), 129.5, 124.7 (d, *J* = 3.0 Hz), 122.9 (d, *J* = 13.0 Hz), 116.6 (d, *J* = 19.0 Hz), 49.0, 25.5.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{12}FN_5$) requires *m/z* 234.1150, found *m/z* 234.1154.

(3Ga) 1-(2-chlorophenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



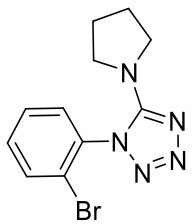
white crystal, 22.4 mg, 45%, m.p. 125-126 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.55 (m, 1H), 7.54 – 7.49 (m, 2H), 7.46 – 7.41 (m, 1H), 3.31 – 3.25 (m, 4H), 1.92 – 1.87 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.4, 133.0, 132.8, 131.8, 130.2, 130.0, 127.6, 48.9, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{12}ClN_5$) requires *m/z* 250.0584, found *m/z* 250.0586.

(3Ha) 1-(2-bromophenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



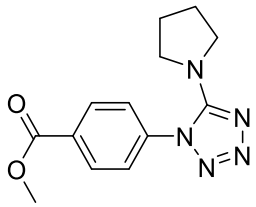
white crystal, 23.4 mg, 40%, m.p. 151-152 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 – 7.72 (m, 1H), 7.53 – 7.41 (m, 3H), 3.31 – 3.24 (m, 4H), 1.92 – 1.87 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.1, 134.4, 133.4, 131.9, 130.1, 128.2, 123.0, 48.9, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{12}BrN_5$) requires *m/z* 294.0349, found *m/z* 294.0352.

(3Ia) methyl 4-(5-(pyrrolidin-1-yl)-1H-tetrazol-1-yl)benzoate



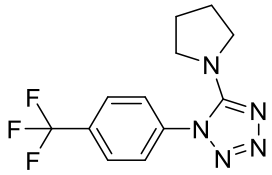
white crystal, 40.4 mg, 74%, m.p. 117-119 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.23 – 8.18 (m, 2H), 7.60 – 7.56 (m, 2H), 3.97 (d, *J* = 1.6 Hz, 3H), 3.34 – 3.27 (m, 4H), 1.95 – 1.89 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 165.7, 155.8, 138.2, 131.2, 130.6, 125.6, 52.5, 50.2, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{13}H_{15}N_5O_2$) requires *m/z* 274.1299, found *m/z* 274.1301.

(3Ja) 5-(pyrrolidin-1-yl)-1-(4-(trifluoromethyl)phenyl)-1H-tetrazole



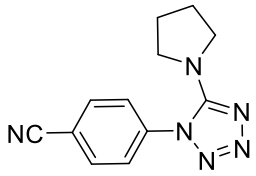
white crystal, 47.5 mg, 84%, m.p. 126-127 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.4 Hz, 2H), 3.34 – 3.28 (m, 4H), 1.97 – 1.91 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8, 137.6, 131.6 (q, *J* = 33.2 Hz), 126.4 (q, *J* = 3.7 Hz), 126.1, 123.3 (q, *J* = 272.6 Hz), 118.6, 50.3, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{12}F_3N_5$) requires *m/z* 284.1118, found *m/z* 284.1118.

(3Ka) 4-(5-(pyrrolidin-1-yl)-1H-tetrazol-1-yl)benzonitrile



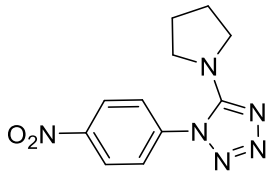
white crystal, 29.2 mg, 61%, m.p. 177-178 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.4 Hz, 2H), 7.67 (d, *J* = 8.8 Hz, 2H), 3.35 – 3.27 (m, 4H), 2.00 – 1.92 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8, 138.1, 133.2, 126.1, 117.4, 113.4, 50.5, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{12}N_6$) requires *m/z* 241.1196, found *m/z* 241.1196.

(3La) 1-(4-nitrophenyl)-5-(pyrrolidin-1-yl)-1H-tetrazole



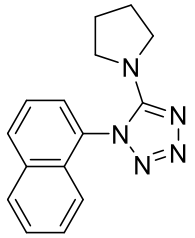
orange solid, 29.6 mg, 57%, m.p. 134-136 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.44 – 8.39 (m, 2H), 7.76 – 7.71 (m, 2H), 3.37 – 3.30 (m, 4H), 2.00 – 1.94 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.0, 147.8, 139.6, 126.1, 124.7, 50.6, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_{12}N_6O_2$) requires *m/z* 261.1095, found *m/z* 261.1097.

(3Ma) 1-(naphthalen-1-yl)-5-(pyrrolidin-1-yl)-1H-tetrazole



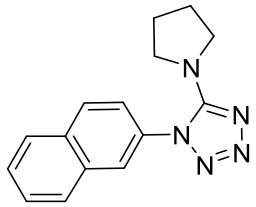
brown-red crystal, 35.5 mg, 67%, m.p. 127-128 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 – 8.02 (m, 1H), 7.98 – 7.95 (m, 1H), 7.61 – 7.53 (m, 4H), 7.45 (d, *J* = 8.0 Hz, 1H), 3.15 (s, 4H), 1.80 – 1.73 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.3, 133.8 130.9, 130.9, 130.4, 128.3, 127.2, 126.0, 124.7, 122.1, 49.0, 25.4.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{15}H_{15}N_5$) requires *m/z* 266.1400, found *m/z* 266.1400.

(3Na) 1-(naphthalen-2-yl)-5-(pyrrolidin-1-yl)-1H-tetrazole



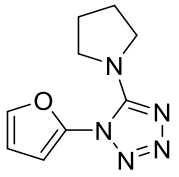
yellow crystal, 30.7 mg, 58%, m.p. 118-120 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.89 (m, 4H), 7.64 – 7.58 (m, 2H), 7.57 – 7.52 (m, 1H), 3.34 – 3.27 (m, 4H), 1.91 – 1.84 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.9, 133.2, 132.6, 132.0, 129.4, 128.2, 127.9, 127.6, 127.5, 125.1, 123.5, 50.0, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{15}H_{15}N_5$) requires *m/z* 266.1400, found *m/z* 266.1400.

(3Oa) 1-(furan-2-yl)-5-(pyrrolidin-1-yl)-1H-tetrazole



red-brown crystal, 18.9 mg, 46%, m.p. 98 °C

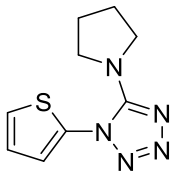
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.47 (m, 1H), 6.62 (d, *J* = 3.2 Hz, 1H), 6.59 – 6.56 (m, 1H), 3.36 – 3.30 (m, 4H), 1.97 – 1.91 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.5, 142.0, 142.0, 139.0, 111.9, 109.2, 48.5, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_9H_{11}N_5O$) requires *m/z* 206.1036, found *m/z*

206.1035.

(3Pa) 5-(pyrrolidin-1-yl)-1-(thiophen-2-yl)-1H-tetrazole



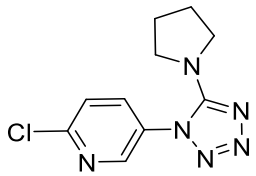
black crystal, 36.2 mg, 82%, m.p. 121-122 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.42 (m, 1H), 7.24 – 7.21 (m, 1H), 7.08 – 7.05 (m, 1H), 3.40 – 3.33 (m, 4H), 1.95 – 1.89 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.8, 134.1, 127.6, 127.0, 125.7, 49.0, 25.5.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_9H_{11}N_5S$) requires *m/z* 222.0808, found *m/z* 222.0808.

(3Qa) 2-chloro-5-(5-(pyrrolidin-1-yl)-1H-tetrazol-1-yl)pyridine



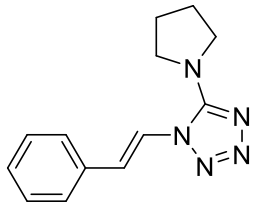
white crystal, 40.5 mg, 81%, m.p. 146-148 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (d, *J* = 2.8 Hz, 1H), 7.86 (dd, *J* = 8.8, 2.8 Hz, 1H), 7.55 (d, *J* = 8.8 Hz, 1H), 3.35 – 3.27 (m, 4H), 2.00 – 1.92 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 155.7, 152.4, 146.4, 135.9, 130.6, 124.8, 50.3, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{10}H_{11}ClN_6$) requires *m/z* 251.0826, found *m/z* 251.0811

(3Ra) (E)-5-(pyrrolidin-1-yl)-1-styryl-1H-tetrazole



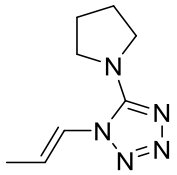
orange crystal, 37.5 mg, 78%, m.p. 99-100 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.32 (m, 6H), 7.23 (d, *J* = 14.0 Hz, 1H), 3.66 (t, *J* = 6.4 Hz, 4H), 2.06 – 2.01 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 154.9, 133.7, 128.9, 128.7, 126.6, 125.7, 118.8, 50.2, 25.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{13}H_{13}N_5$) requires *m/z* 242.1400, found *m/z* 242.1401.

(3Sa) (E)-1-(prop-1-en-1-yl)-5-(pyrrolidin-1-yl)-1H-tetrazole



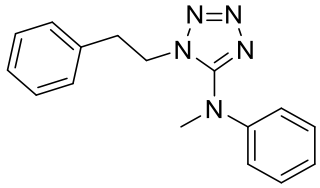
white crystal, 6.4 mg, 18%, m.p. 108-110 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.76 (dq, *J* = 13.6, 1.6 Hz, 1H), 6.31 (dq, *J* = 13.6, 6.8 Hz, 1H), 3.61 – 3.56 (m, 4H), 2.03 – 1.98 (m, 4H), 1.89 (dd, *J* = 6.8, 1.6 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 154.7, 124.7, 12.0, 50.0, 25.6, 15.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_8H_{13}N_5$) requires *m/z* 180.1244, found *m/z* 180.1246.

(5aa) N-methyl-1-phenethyl-N-phenyl-1H-tetrazol-5-amine



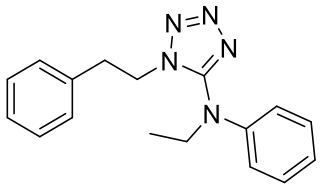
light yellow liquid, 27.9 mg, 50%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.34 (m, 2H), 7.27 – 7.17 (m, 2H), 6.95 – 6.90 (m, 2H), 6.84 – 6.80 (m, 2H), 3.90 (t, *J* = 7.6 Hz, 2H), 3.33 (s, 3H), 2.91 (t, *J* = 7.6 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.9, 145.2, 136.5, 130.0, 128.6, 127.0, 125.3, 122.2, 48.5, 42.3, 35.1.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{16}H_{17}N_5$) requires *m/z* 280.1557, found *m/z* 280.1563.

(5ba) N-ethyl-1-phenethyl-N-phenyl-1H-tetrazol-5-amine



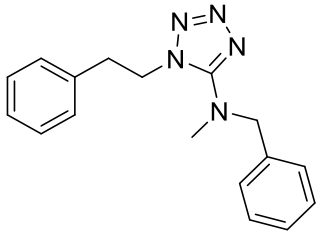
brown-red oily liquid, 18.1 mg, 31% •

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.37 (m, 2H), 7.25 – 7.17 (m, 4H), 6.99 – 6.94 (m, 2H), 6.85 – 6.77 (m, 2H), 3.87 – 3.76 (m, 4H), 2.88 (t, 2H), 1.21 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.2, 143.5, 136.5, 130.0, 128.6, 127.0, 125.8, 123.8, 49.9, 48.4, 34.9, 12.8.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{17}H_{19}N_5$) requires *m/z* 294.1713, found *m/z* 294.1712.

(5ca) N-benzyl-N-methyl-1-phenethyl-1H-tetrazol-5-amine



light yellow transparent liquid, 16.4 mg, 28%

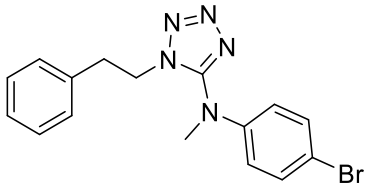
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.22 (m, 6H), 7.22 – 7.18 (m, 2H), 7.05 – 6.99 (m, 2H), 4.35 (t, *J* = 7.2 Hz, 2H), 4.27 (s, 2H), 3.21 (t, *J* = 7.2 Hz, 2H), 2.80 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 136.6, 136.1, 128.8, 128.8, 128.7, 127.8, 127.6, 127.2, 57.9, 48.5, 39.1, 35.6.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{17}H_{19}N_5$) requires *m/z* 294.1713, found *m/z*

294.1716.

(5da) N-(4-bromophenyl)-N-methyl-1-phenethyl-1H-tetrazol-5-amine



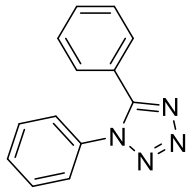
yellow liquid, 24.8 mg, 35%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.42 (m, 2H), 7.28 – 7.21 (m, 3H), 6.88 – 6.82 (m, 2H), 6.74 – 6.65 (m, 2H), 3.96 (t, *J* = 7.2 Hz, 2H), 3.23 (s, 3H), 2.98 (t, *J* = 7.2 Hz, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 156.6, 144.2, 136.3, 132.9, 128.7, 127.1, 122.7, 117.7, 48.6, 41.8, 35.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{16}H_{16}BrN_5$) requires *m/z* 358.0662, found *m/z* 358.0661.

(7aa) 1,5-diphenyl-1H-tetrazole



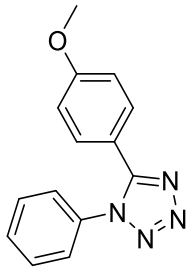
white crystal, 35.9 mg, 81%, m.p. 114-116 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.47 (m, 6H), 7.43 – 7.36 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 153.5, 134.5, 131.2, 130.3, 129.8, 128.9, 128.8, 125.2, 123.5.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{13}H_{10}N_4$) requires *m/z* 223.0978, found *m/z* 223.0978.

(7ba) 5-(4-methoxyphenyl)-1-phenyl-1H-tetrazole



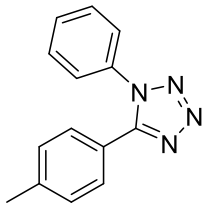
incarnadine solid, 44.8 mg, 89%, m.p. 98-99 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.48 (m, 5H), 7.43 – 7.38 (m, 2H), 6.92 – 6.87 (m, 2H), 3.83 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 161.8, 153.4, 134.7, 130.4, 129.8, 125.3, 115.6, 114.4, 55.4.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{14}H_{12}N_4$) requires *m/z* 253.1089, found *m/z* 253.1089.

(7ca) 1-phenyl-5-(p-tolyl)-1H-tetrazole



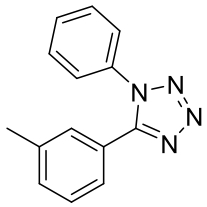
white crystal, 27.8 mg, 59%, m.p. 126-128 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.50 (m, 3H), 7.46 – 7.38 (m, 4H), 7.20 (d, *J* = 8.0 Hz, 2H), 2.38 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 153.6, 141.7, 134.7, 130.3, 129.8, 129.6, 128.8, 125.3, 120.6, 21.4.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{14}H_{12}N_4$) requires *m/z* 237.1135, found *m/z* 237.1136.

(7da) 1-phenyl-5-(m-tolyl)-1H-tetrazole



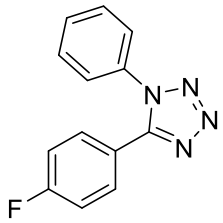
light yellow crystal, 33.5 mg, 71%, m.p. 115-117°C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.50 (m, 3H), 7.49 – 7.47 (m, 1H), 7.43 – 7.37 (m, 2H), 7.32 – 7.20 (m, 3H), 2.33 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 153.7, 139.0, 134.6, 132.0, 130.3, 129.8, 129.6, 128.7, 125.8, 125.23, 123.4, 21.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{14}H_{12}N_4$) requires *m/z* 237.1135, found *m/z* 237.1137.

(7ea) 5-(4-fluorophenyl)-1-phenyl-1H-tetrazole



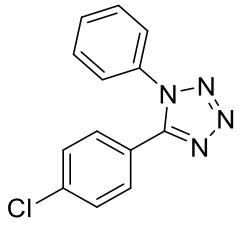
white crystal, 36.4 mg, 76%, m.p. 115-117 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 – 7.52 (m, 5H), 7.43 – 7.38 (m, 2H), 7.14 – 7.07 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 164.3 (d, *J* = 253.0 Hz), 152.8, 134.4, 131.1, 131.1, 130.5, 130.0, 125.3, 119.8, 119.7 (d, *J* = 3.0 Hz), 116.3 (d, *J* = 22.0 Hz).

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{13}H_9FN_4$) requires *m/z* 241.0884, found *m/z* 241.0883.

(7fa) 5-(4-chlorophenyl)-1-phenyl-1H-tetrazole



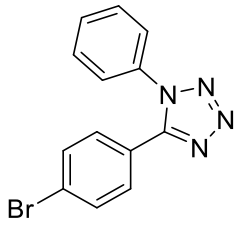
white crystal, 36.3 mg, 71%, m.p. 151-153 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 – 7.49 (m, 5H), 7.43 – 7.36 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 152.7, 137.7, 134.3, 130.6, 130.1, 130.0, 129.4, 125.3, 122.0.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{13}H_9ClN_4$) requires *m/z* 257.0589, found *m/z* 257.0591.

(7ga) 5-(4-bromophenyl)-1-phenyl-1H-tetrazole



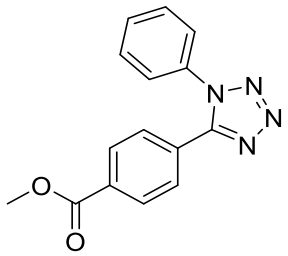
white crystal, 30.0 mg, 50%, m.p. 160-162 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 – 7.52 (m, 5H), 7.46 – 7.41 (m, 2H), 7.41 – 7.38 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 152.8, 134.3, 132.3, 130.6, 130.3, 130.0, 126.1, 125.3, 122.5.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{13}H_9BrN_4$) requires *m/z* 301.0083, found *m/z* 301.0088.

(7ha) methyl 4-(1-phenyl-1H-tetrazol-5-yl)benzoate



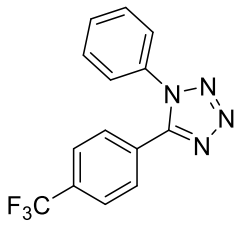
light yellow powder, 43.1 mg, 77%, m.p. 140-142 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 – 8.04 (m, 2H), 7.68 – 7.63 (m, 2H), 7.62 – 7.52 (m, 3H), 7.43 – 7.36 (m, 2H), 3.93 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 165.9, 152.8, 134.2, 132.5, 130.6, 130.1, 130.0, 128.9, 127.6, 52.4.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{15}H_{12}N_4O_2$) requires *m/z* 281.1033, found *m/z* 281.1035.

(7ia) 1-phenyl-5-(4-(trifluoromethyl)phenyl)-1H-tetrazole



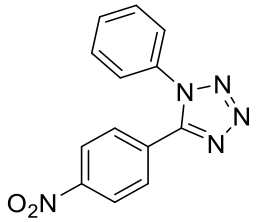
light yellow crystal, 39.4 mg, 68%, m.p. 64-66 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 – 7.66 (m, 4H), 7.62 – 7.54 (m, 3H), 7.43 – 7.38 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 152.5, 134.2, 133.1 (q, *J* = 33.3 Hz), 130.8, 130.1, 129.3, 127.1, 126.4 – 125.7 (q, *J* = 4 Hz), 125.3, 123.4 (q, *J* = 272.8 Hz).

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{14}H_9F_3N_4$) requires *m/z* 291.0852, found *m/z* 291.0854.

(7ja) 5-(4-nitrophenyl)-1-phenyl-1H-tetrazole



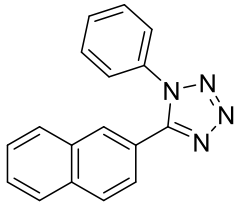
yellow crystal, 37.9 mg, 71%, m.p. 162-164 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 – 8.25 (m, 2H), 7.81 – 7.76 (m, 2H), 7.65 – 7.56 (m, 3H), 7.43 – 7.39 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 151.9, 149.3, 134.0, 131.1, 130.3, 130.0, 129.6, 125.3, 124.1.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{13}H_9N_5O_2$) requires *m/z* 268.0829, found *m/z* 268.0839.

(7ka) 5-(naphthalen-2-yl)-1-phenyl-1H-tetrazole



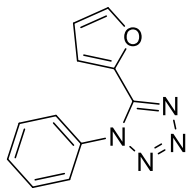
light brown-yellow solid, 36.9 mg, 68%, m.p. 111-112 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 – 8.12 (m, 1H), 7.87 – 7.82 (m, 2H), 7.80 – 7.76 (m, 1H), 7.61 – 7.50 (m, 6H), 7.46 – 7.42 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 153.7, 134.6, 134.1, 132.7, 130.4, 129.9, 129.7, 128.8, 128.7, 128.1, 127.8, 127.1, 125.3, 124.8, 120.8.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{17}H_{12}N_4$) requires *m/z* 273.1135, found *m/z* 273.1140.

(7la) 5-(furan-2-yl)-1-phenyl-1H-tetrazole



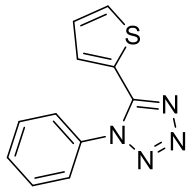
brown crystal, 35.1 mg, 83%, m.p. 100-101 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.57 (m, 3H), 7.54 – 7.53 (m, 1H), 6.84 – 6.82 (m, 1H), 6.53 – 6.50 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 146.5, 145.7, 138.9, 134.2, 130.8, 129.6, 125.8, 115.1, 112.0.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_8N_4O$) requires *m/z* 213.0771, found *m/z* 213.0775.

(7ma) 1-phenyl-5-(thiophen-2-yl)-1H-tetrazole



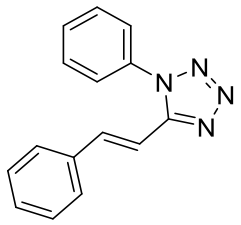
gray-white crystal, 40.1 mg, 88%, m.p. 82-84 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 – 7.59 (m, 3H), 7.54 – 7.45 (m, 3H), 7.28 – 7.23 (m, 1H), 7.08 – 7.02 (m, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 149.8, 134.0, 131.2, 130.5, 130.0, 128.0, 126.3, 124.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{11}H_8N_4S$) requires *m/z* 229.0542, found *m/z* 229.0546.

(7na) (E)-1-phenyl-5-styryl-1H-tetrazole



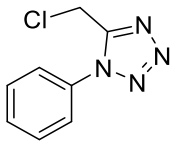
orange crystal, 35.2 mg, 71%, m.p. 160-162 °C

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 16.4 Hz, 1H), 7.67 – 7.60 (m, 3H), 7.56 – 7.47 (m, 4H), 7.41 – 7.36 (m, 3H), 6.81 (d, *J* = 16.4 Hz, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 152.2, 141.2, 134.6, 133.7, 130.4, 130.0, 128.9, 127.6, 125.1, 107.4.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{15}H_{12}N_4$) requires *m/z* 249.1135, found *m/z* 249.1138.

(7oa) 5-(chloromethyl)-1-phenyl-1H-tetrazole



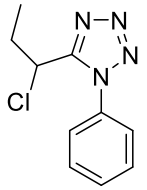
yellow liquid, 26.3 mg, 68%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 – 7.62 (m, 3H), 7.62 – 7.58 (m, 2H), 4.80 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 151.4, 133.2, 131.0, 130.1, 124.6, 31.1.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_8H_7ClN_4$) requires *m/z* 195.0432, found *m/z* 195.0433.

(7pa) 5-(1-chloropropyl)-1-phenyl-1H-tetrazole



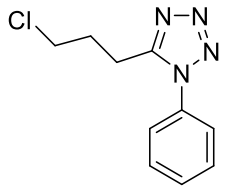
transparent liquid, 31.0 mg, 70%

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 – 7.59 (m, 3H), 7.59 – 7.53 (m, 2H), 4.85 (t, *J* = 7.2 Hz, 1H), 2.44 – 2.35 (m, 2H), 1.06 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 133.3, 131.0, 130.0, 125.3, 49.8, 29.5, 11.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{10}H_{11}ClN_4$) requires *m/z* 223.0475, found *m/z* 223.0476.

(7qa) 5-(3-chloropropyl)-1-phenyl-1H-tetrazole



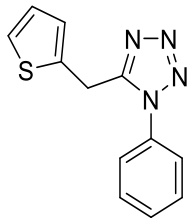
light yellow liquid, 32.4 mg, 73%

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.67 – 7.57 (m, 3H), 7.52 – 7.41 (m, 2H), 3.68 (t, *J* = 6.0 Hz, 2H), 3.08 (t, *J* = 7.2 Hz, 2H), 2.40 – 2.30 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ* 153.9, 133.5, 130.5, 129.9, 124.8, 43.5, 29.2, 20.9.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{10}H_{11}ClN_4$) requires *m/z* 223.0475, found *m/z* 223.0476.

(7ra) 1-phenyl-5-(thiophen-2-ylmethyl)-1H-tetrazole



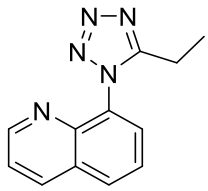
brown solid, 35.3 mg, 73%, m.p. 118-119°C

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.61 – 7.51 (m, 3H), 7.38 – 7.33 (m, 2H), 7.17 (dd, *J* = 5.2, 1.2 Hz, 1H), 6.88 (dd, *J* = 5.2, 3.6 Hz, 1H), 6.75 (dd, *J* = 3.6, 0.8 Hz, 1H), 4.47 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) *δ* 153.3, 135.5, 133.5, 130.6, 129.8, 127.1, 126.8, 125.4, 125.1, 24.2.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_{10}N_4S$) requires *m/z* 243.0699, found *m/z* 243.0699.

(7sa) 8-(5-ethyl-1H-tetrazol-1-yl)quinoline



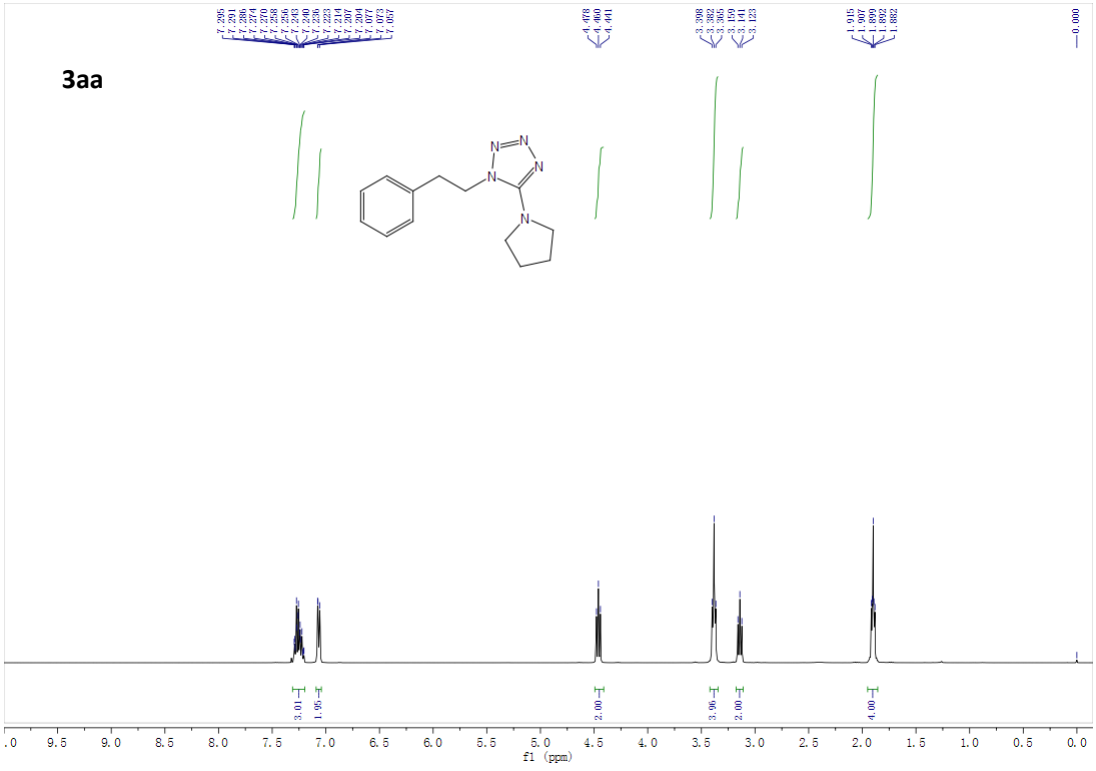
brown-red crystal, 17.1 mg, 38%, m.p. 140-141°C

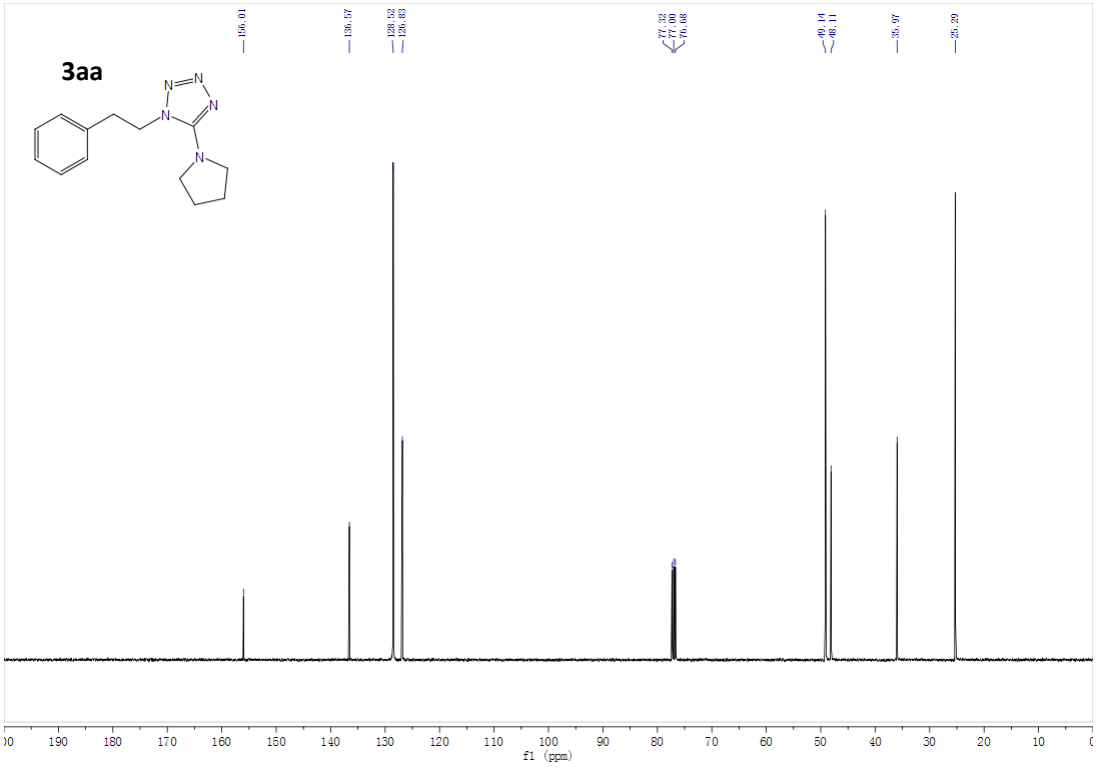
^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.89 (dd, *J* = 4.0, 1.6 Hz, 1H), 8.32 (dd, *J* = 8.4, 1.6 Hz, 1H), 8.10 (dd, *J* = 8.4, 1.2 Hz, 1H), 7.86 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.77 – 7.71 (m, 1H), 7.55 (dd, *J* = 8.4, 4.4 Hz, 1H), 2.76 (q, *J* = 7.6 Hz, 2H), 1.31 (t, *J* = 7.6 Hz, 3H).

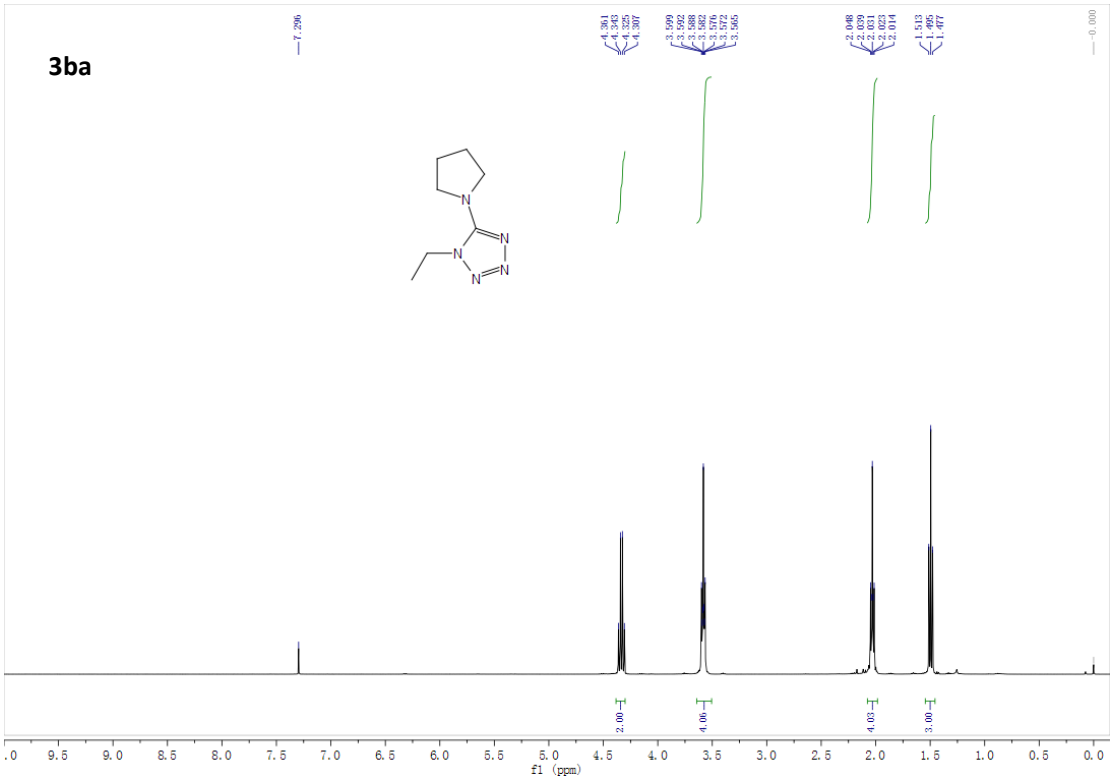
^{13}C NMR (100 MHz, $CDCl_3$) *δ* 158.2, 151.9, 142.5, 136.2, 131.3, 131.1, 129.0, 128.1, 126.0, 122.60, 17.6, 11.3.

HRMS (ESI+): exact mass calculated for $[M+H]^+$ ($C_{12}H_1N_5$) requires *m/z* 226.1087, found *m/z* 226.1091.

Copies of NMR Spectra



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3.588
3.582
3.576
3.572
3.565
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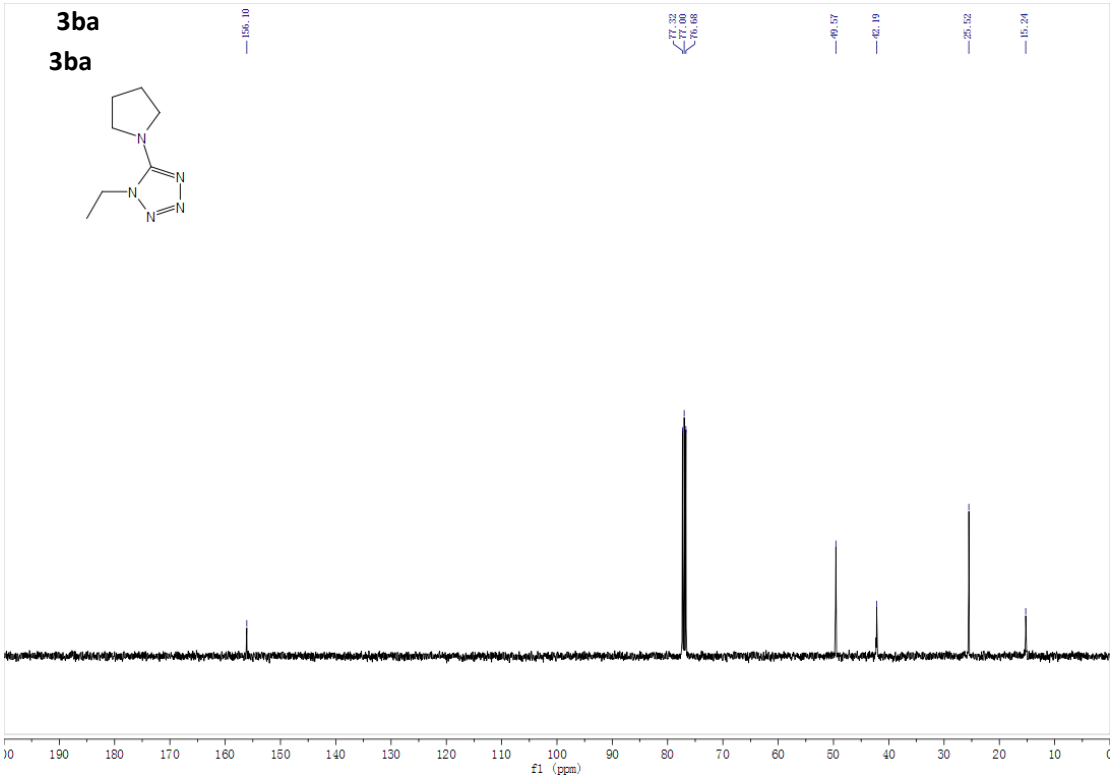
2.039
2.031
2.023
2.014
1.513
1.495
1.477
0.000
2.00
4.06
4.03
3.00
f1 (ppm)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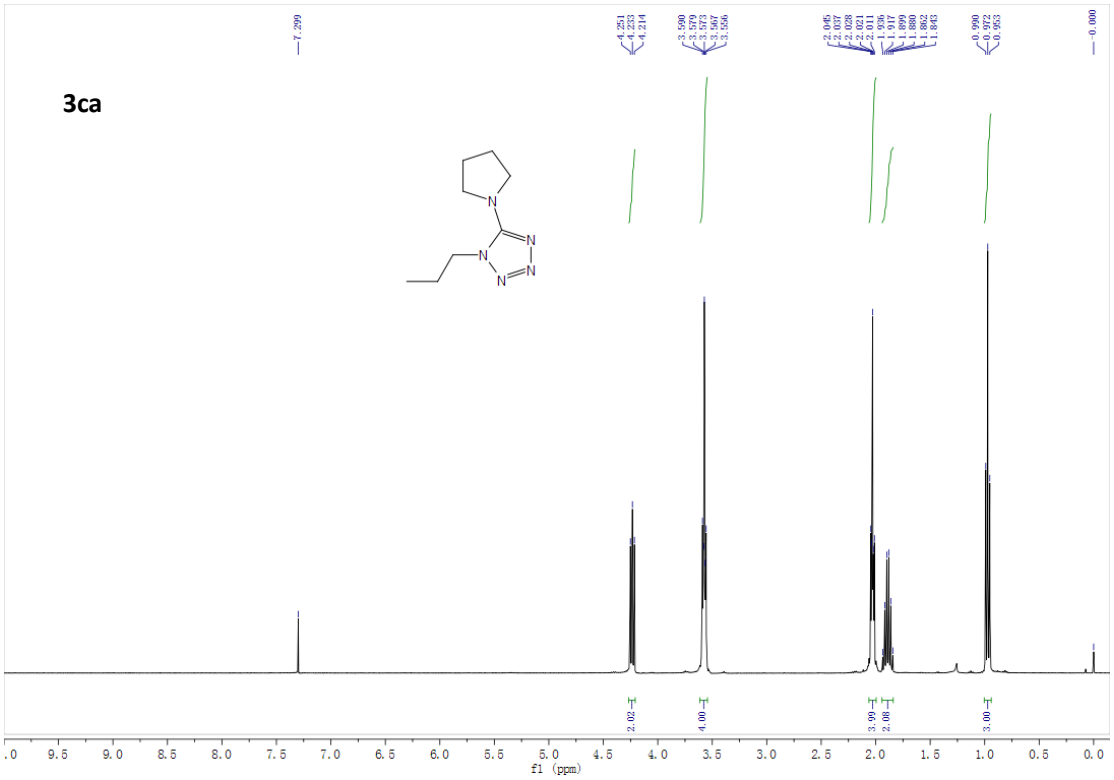
15.24
f1 (ppm)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2.021
2.011
1.936
1.917
1.899
1.890
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0.000
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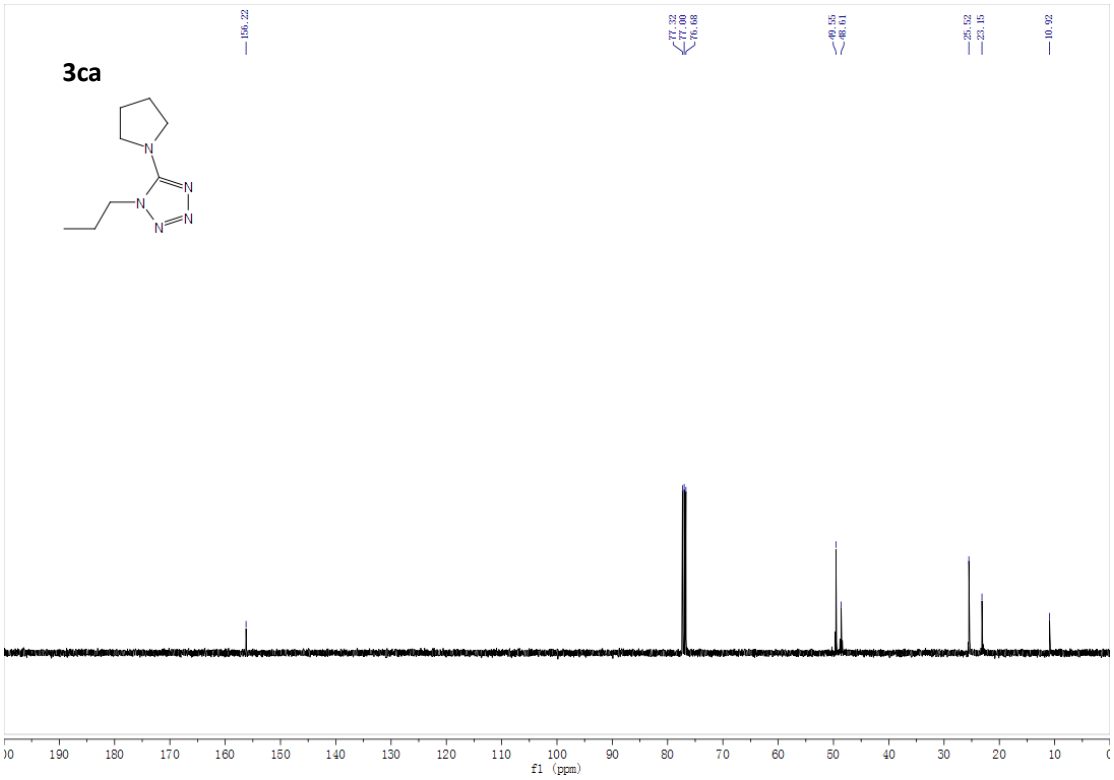
3.00
f1 (ppm)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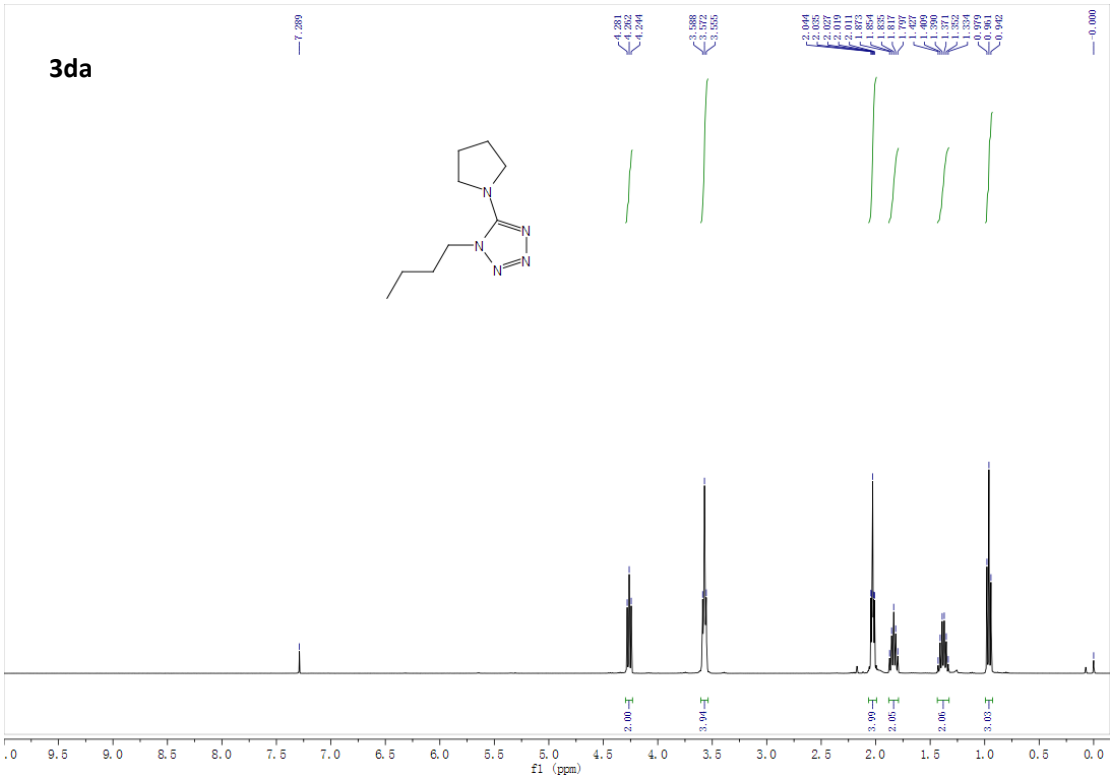
23.15
10.92
f1 (ppm)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1.854
1.835
1.817
1.797
1.487
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1.390
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1.352
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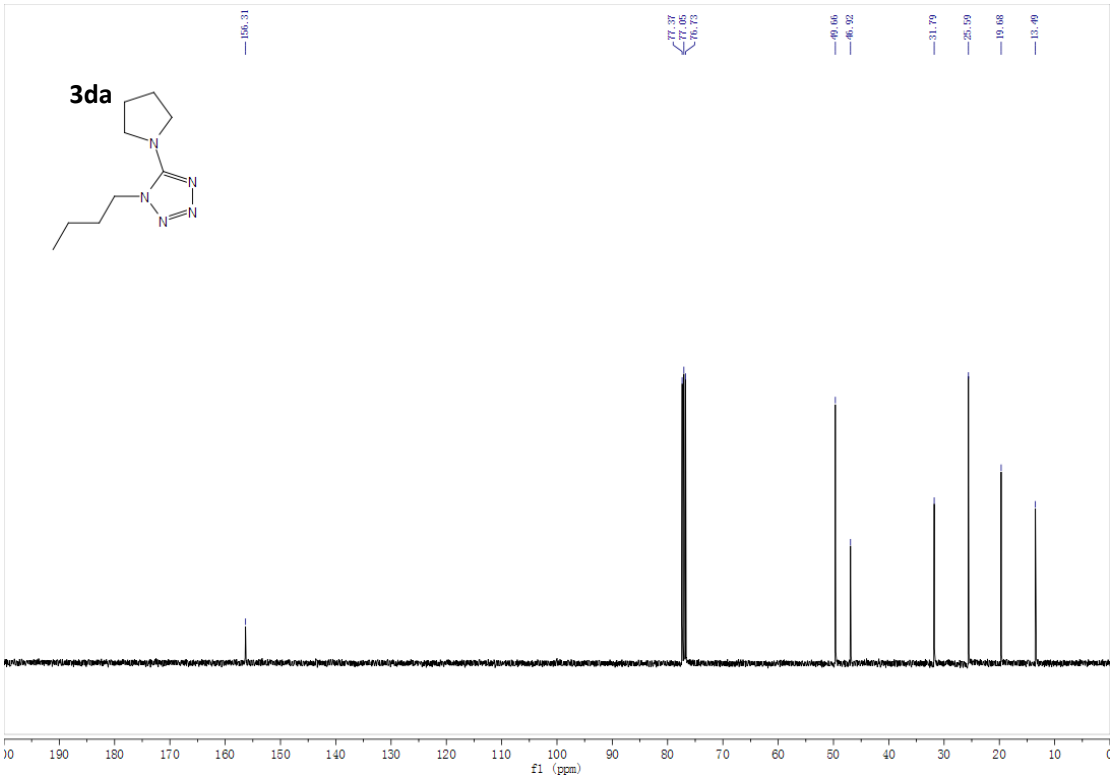
f1 (ppm)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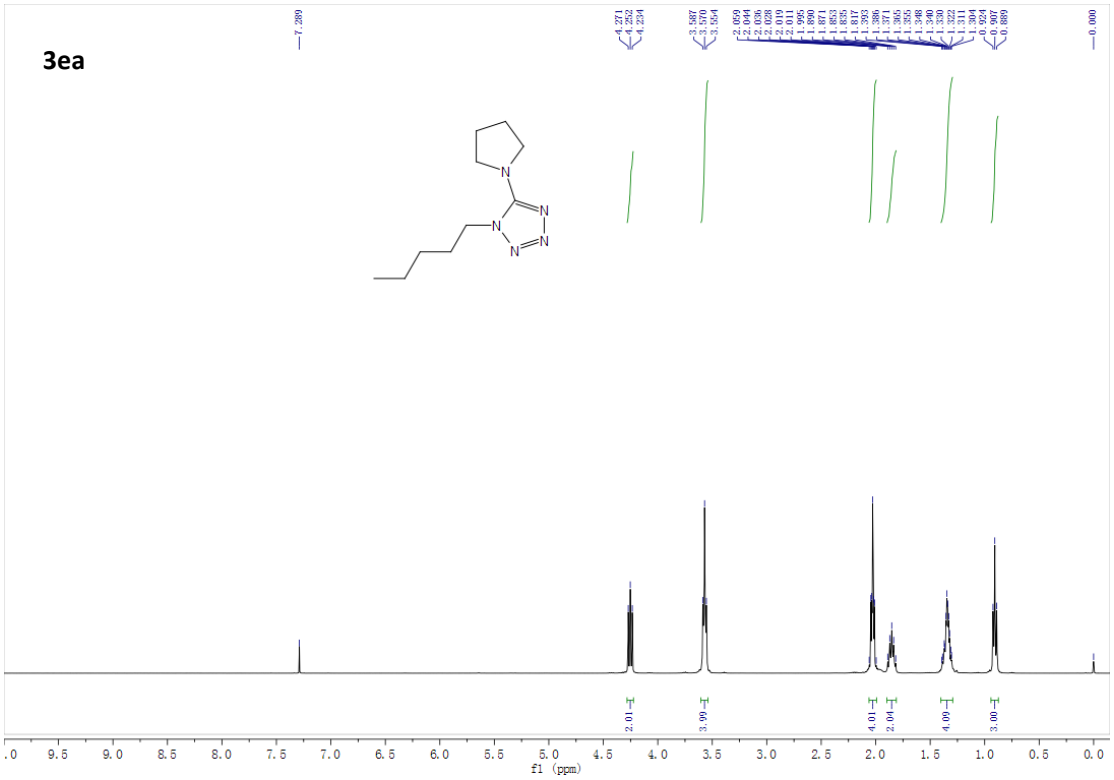
19.68
13.49
f1 (ppm)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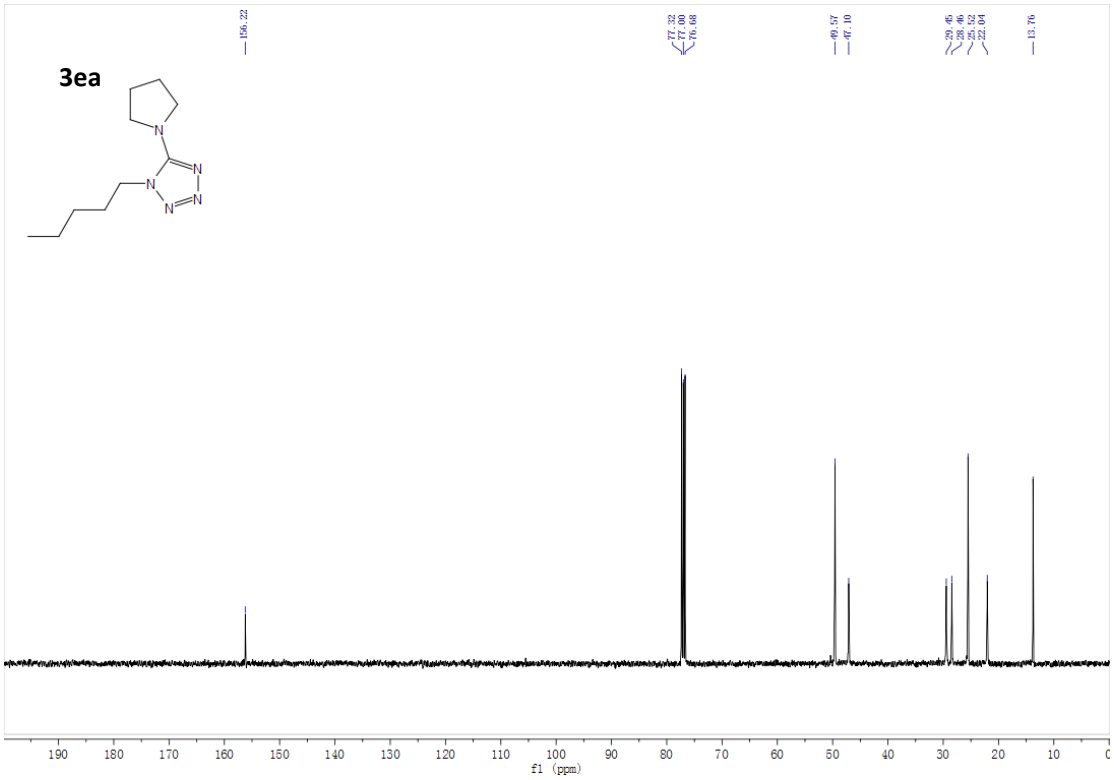
f1 (ppm)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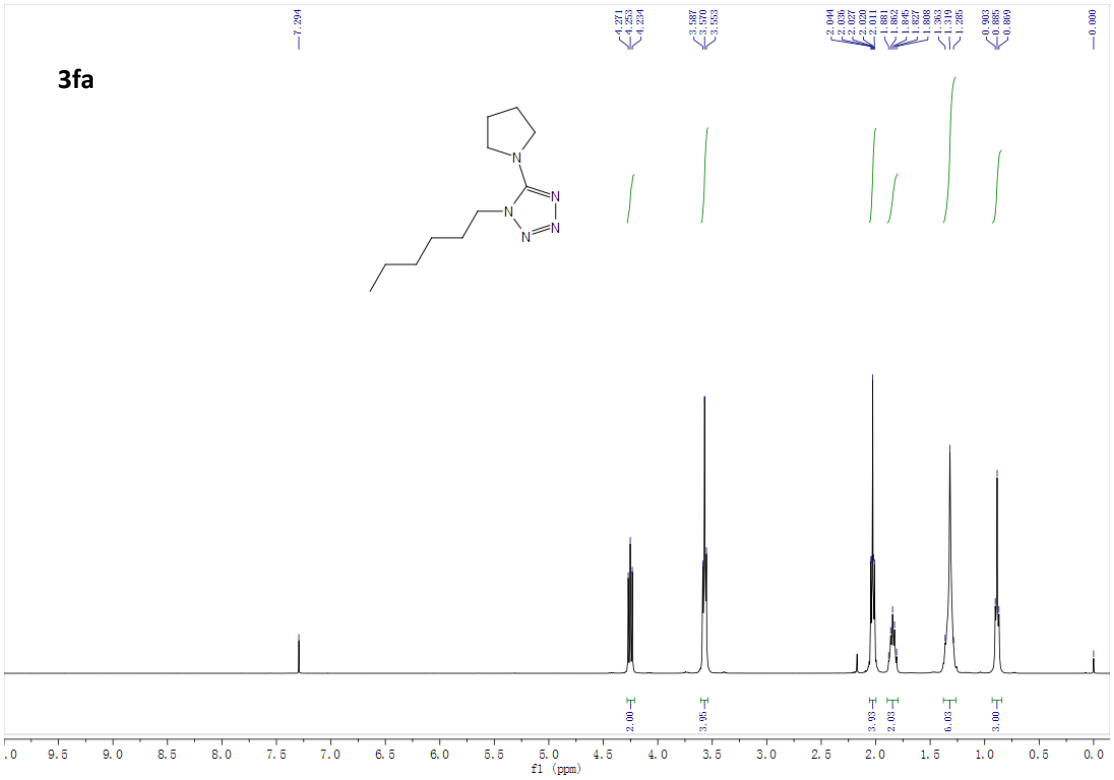
28.46
25.52
22.04
13.76
f1 (ppm)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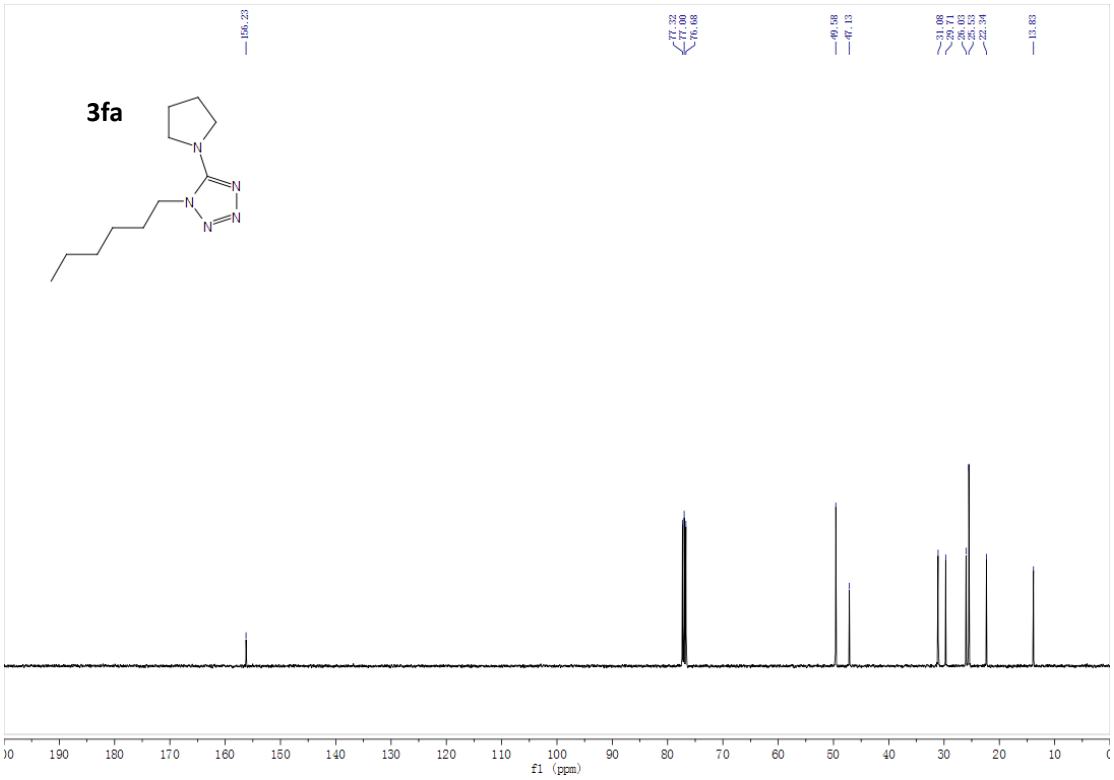
f1 (ppm)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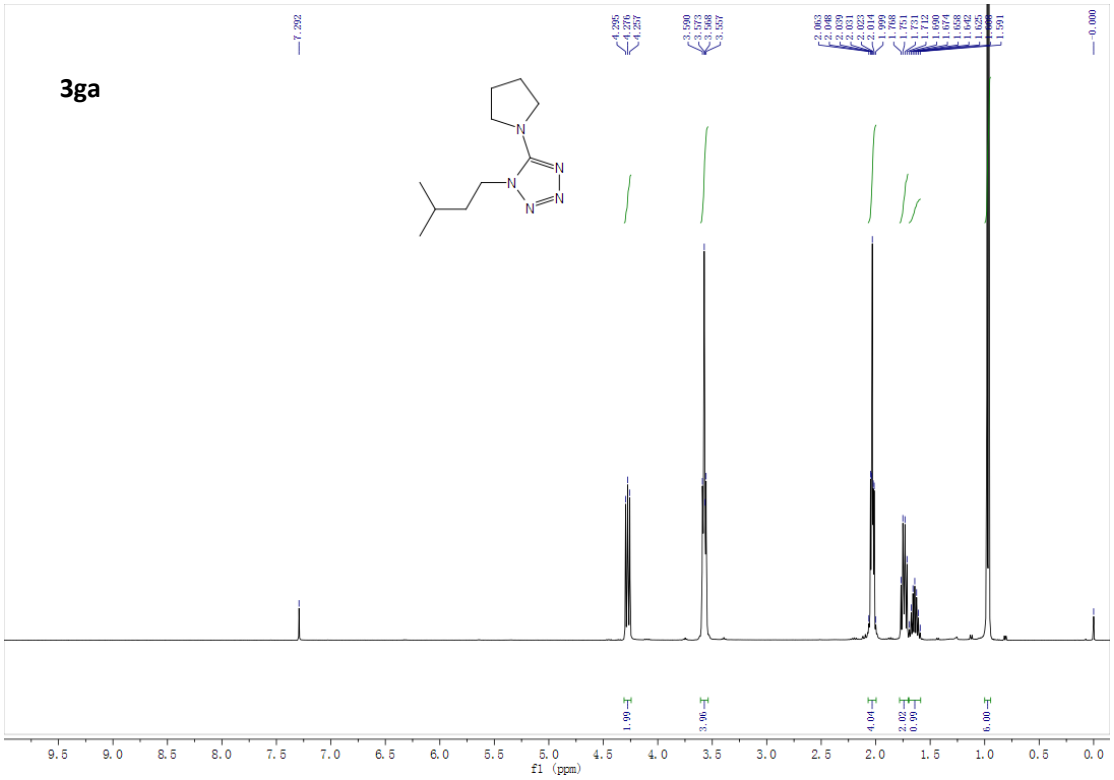
f1 (ppm)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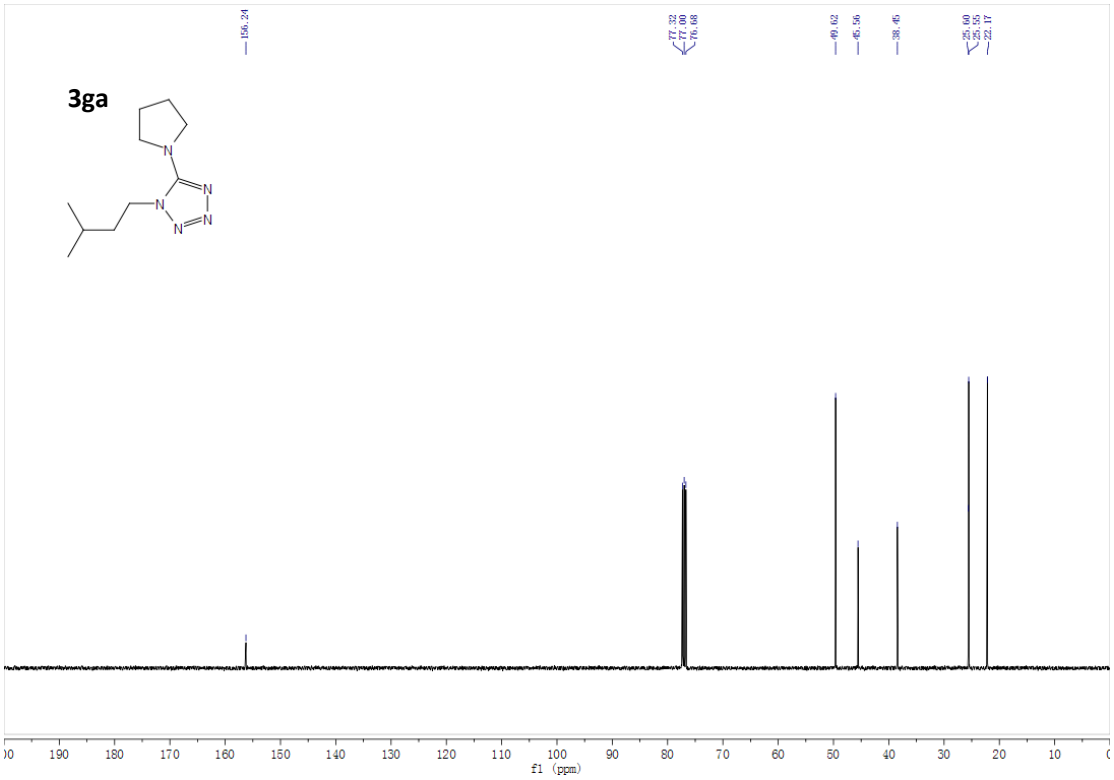
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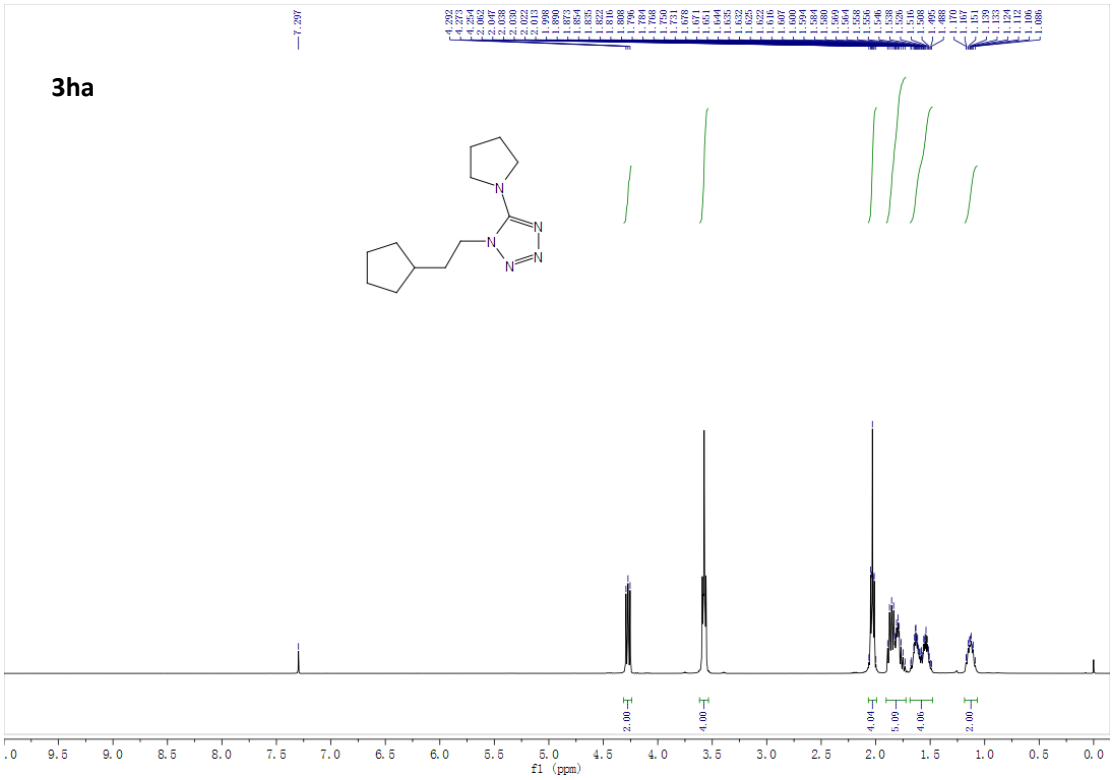
f1 (ppm)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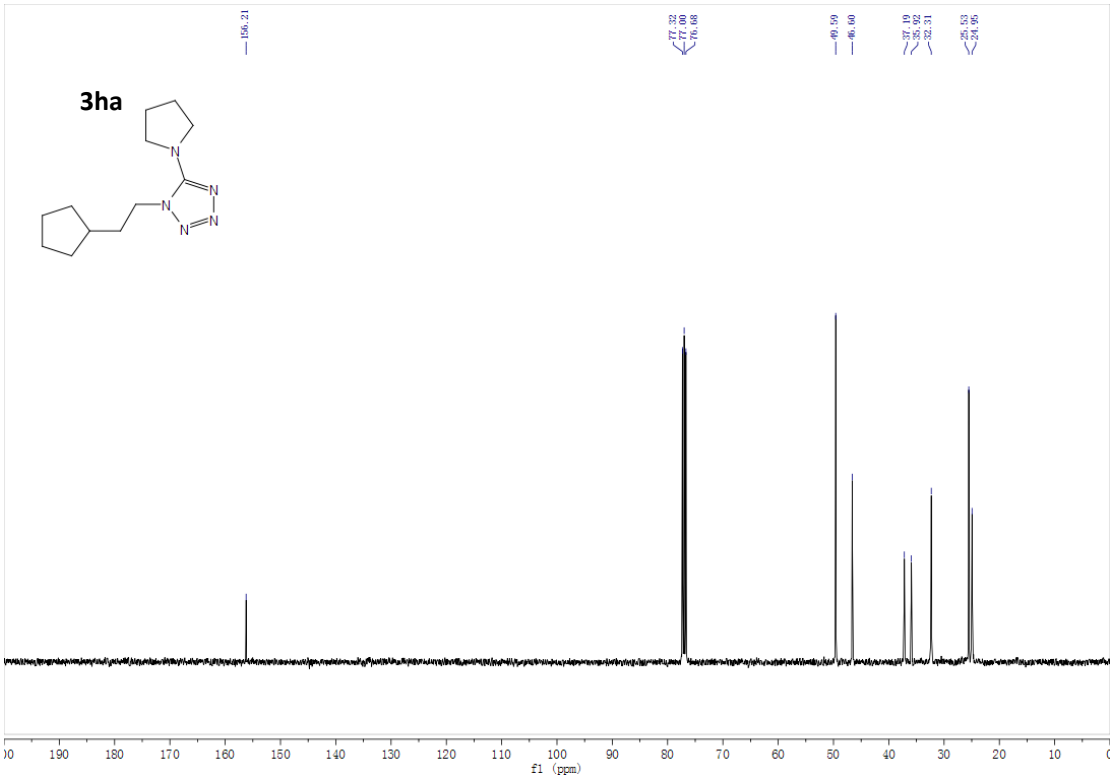
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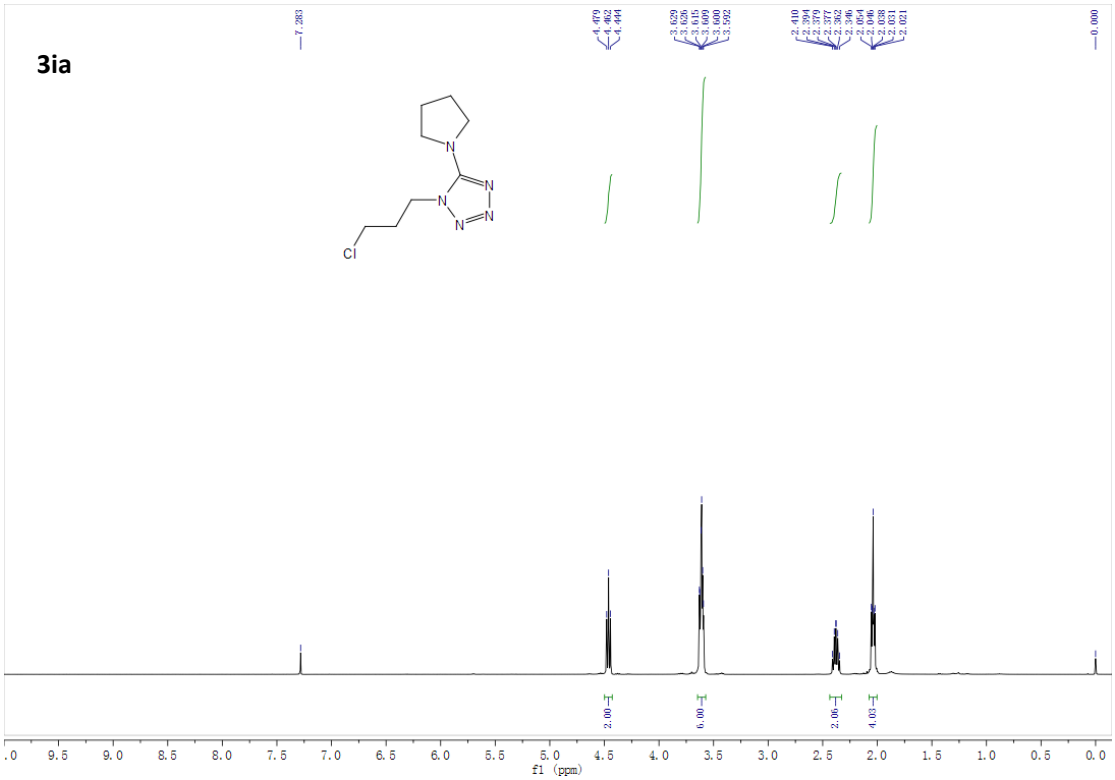
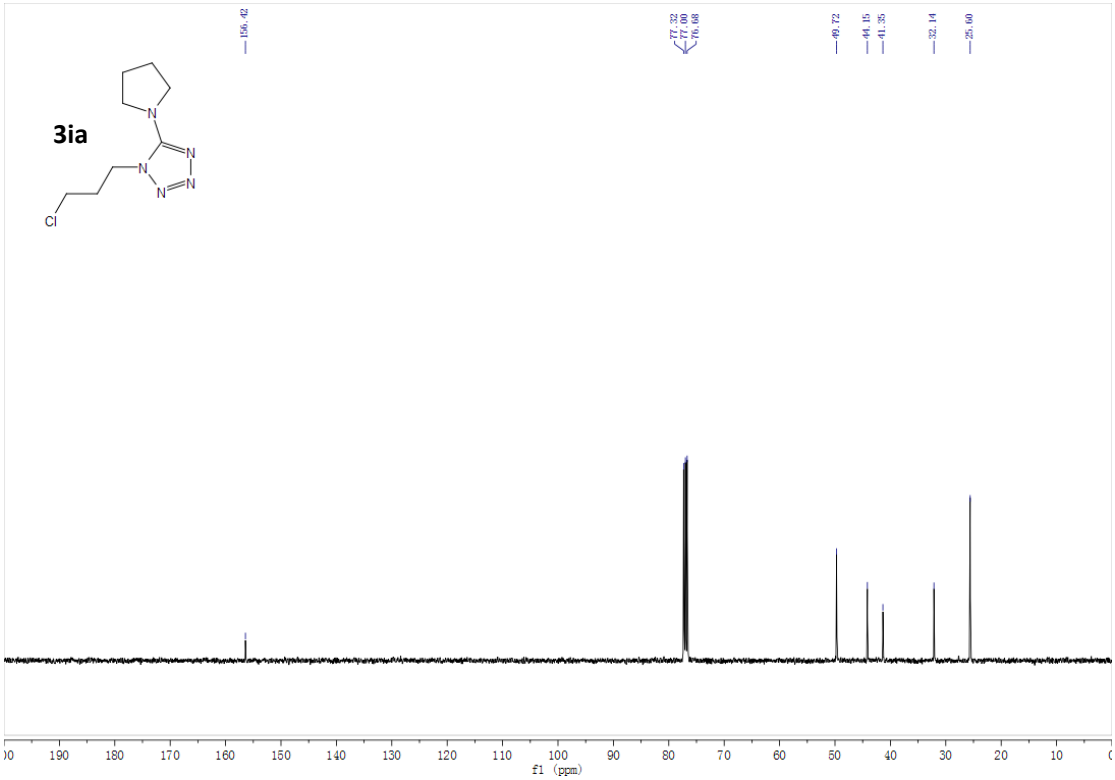
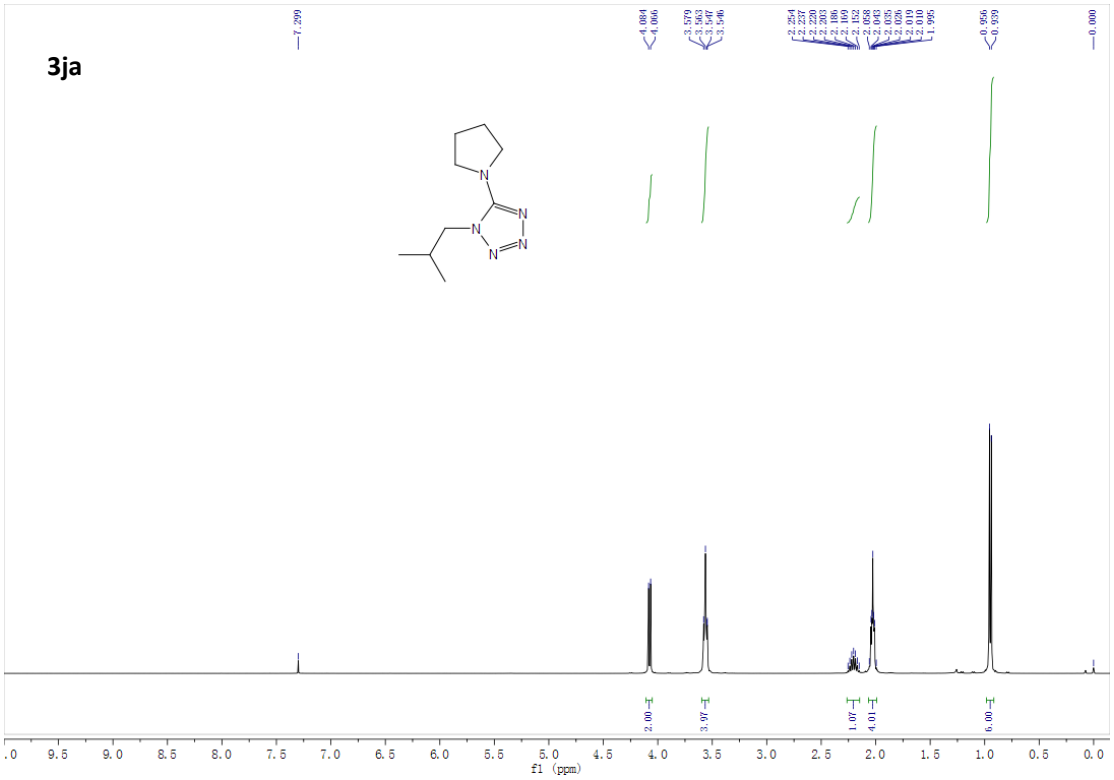
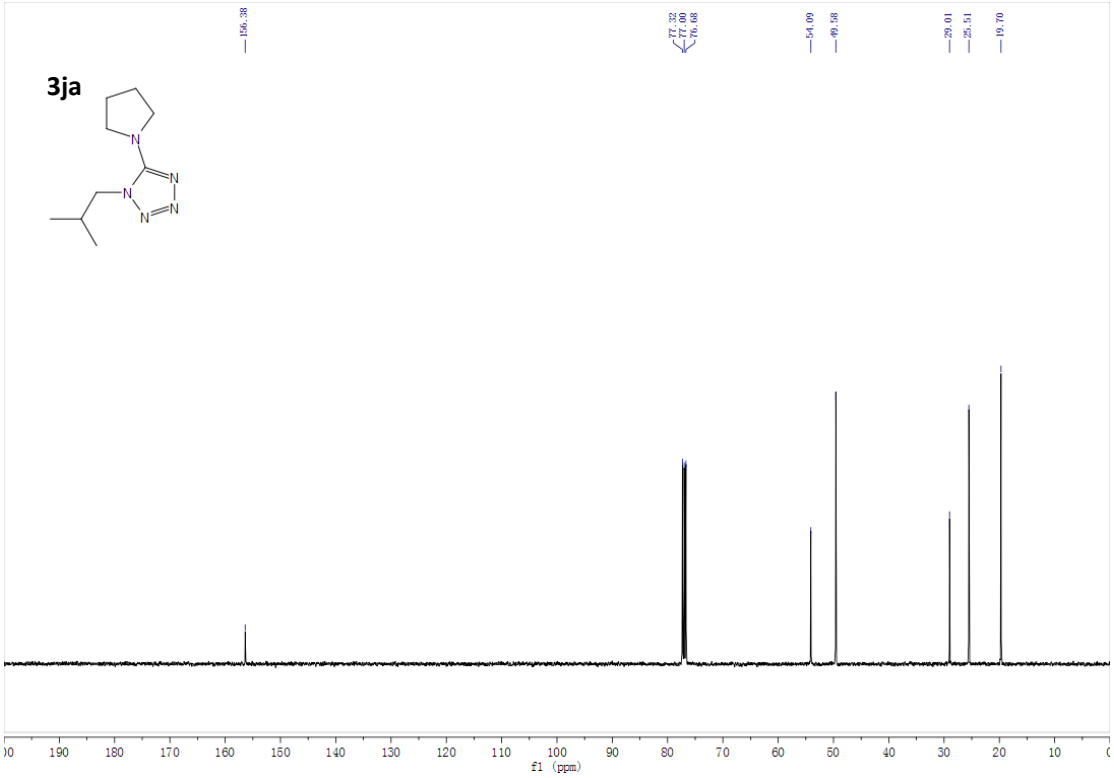

3ia
Cl
f1 (ppm)

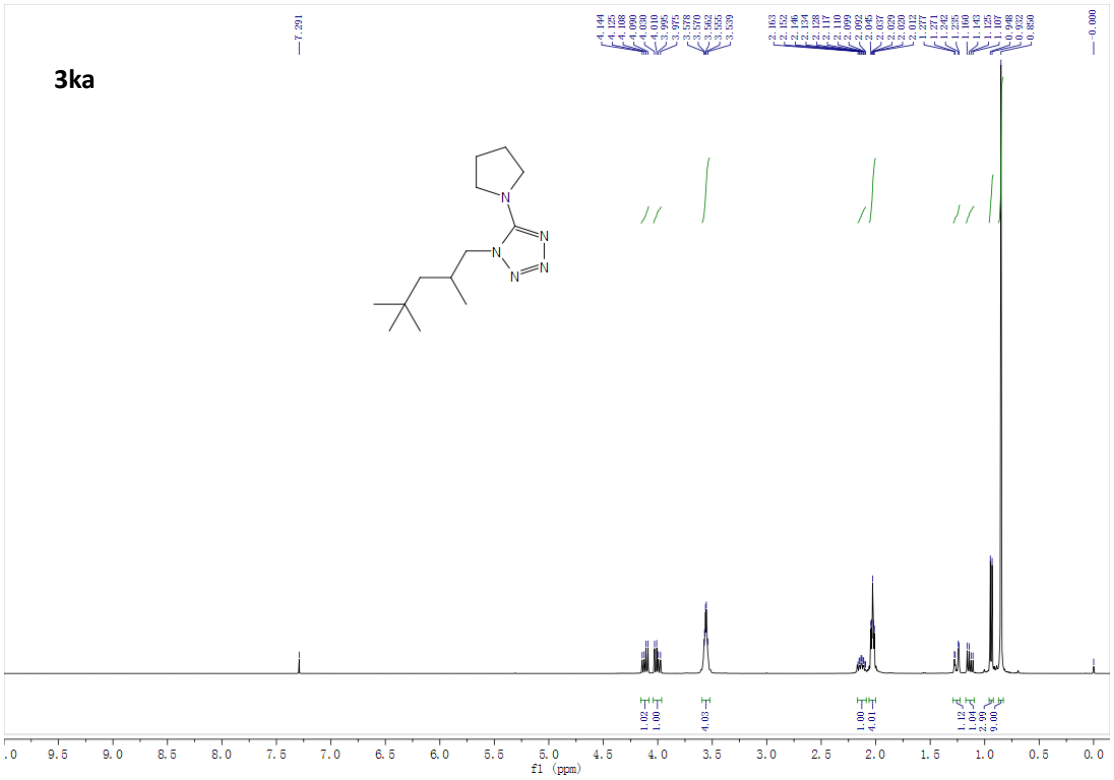

3ia
Cl
f1 (ppm)

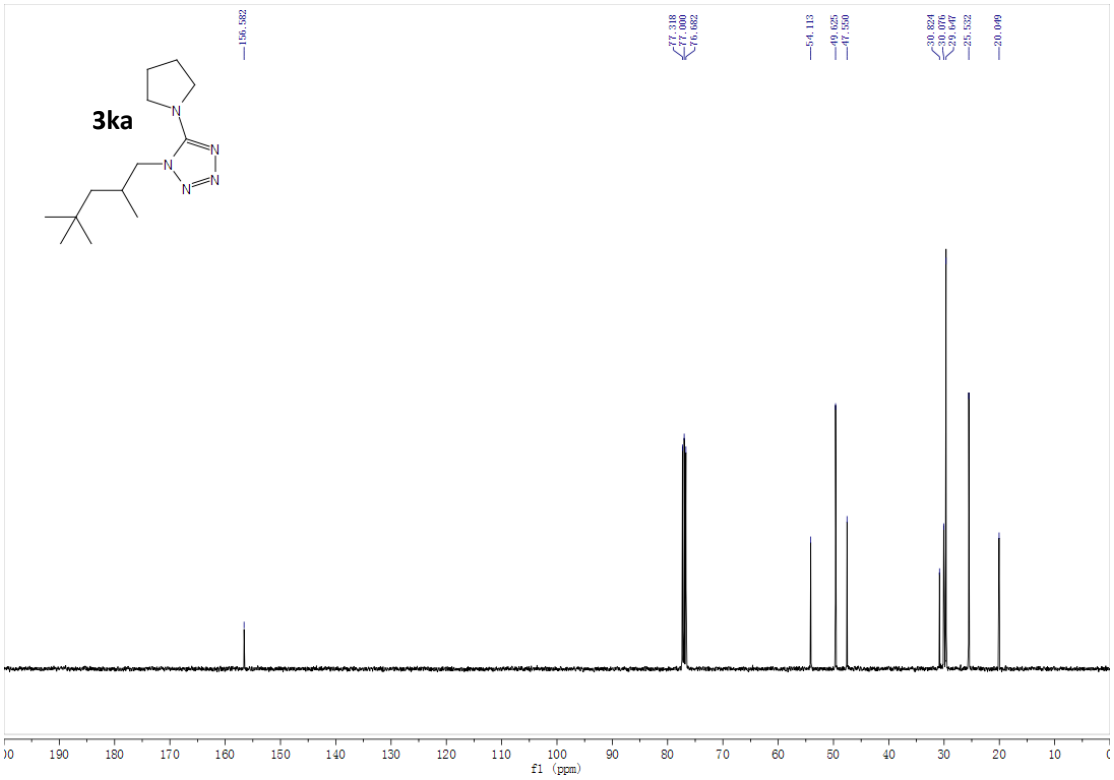
3ja

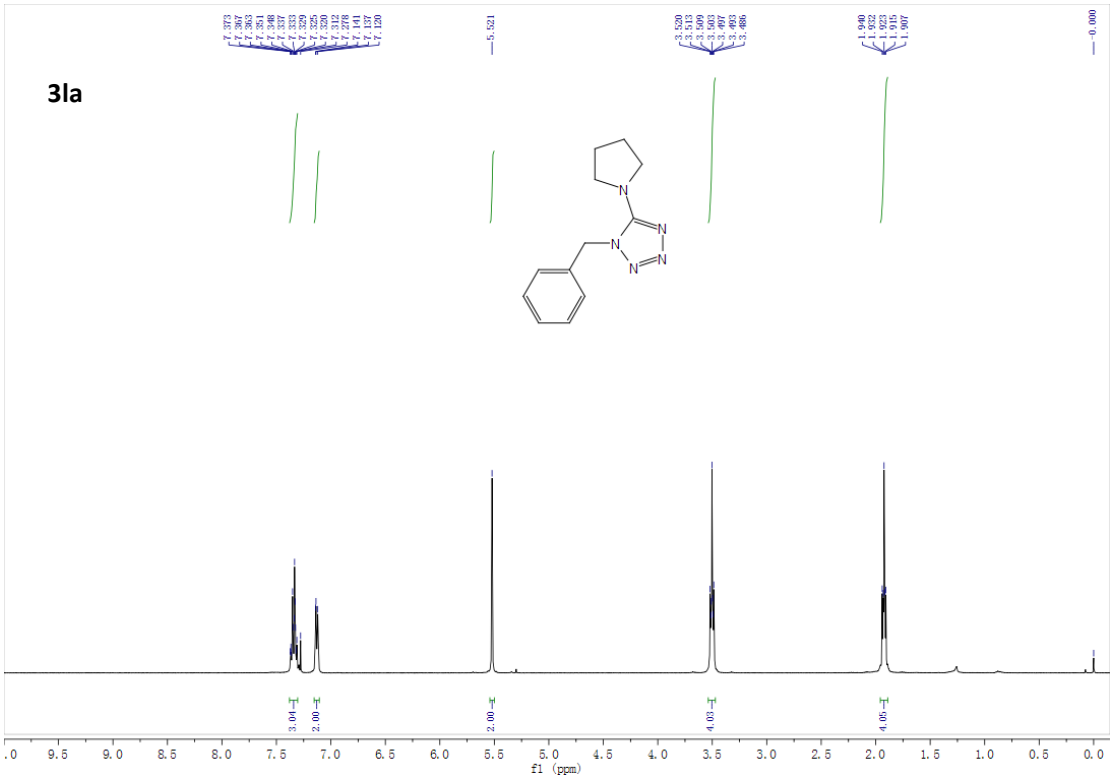

7.299
4.084
4.066
3.579
3.563
3.547
3.546
2.254
2.237
2.220
2.203
2.186
2.169
2.152
2.058
2.043
2.035
2.026
2.010
1.995
0.956
0.939
0.000
2.00
3.97
1.07
4.01
6.00
f1 (ppm)

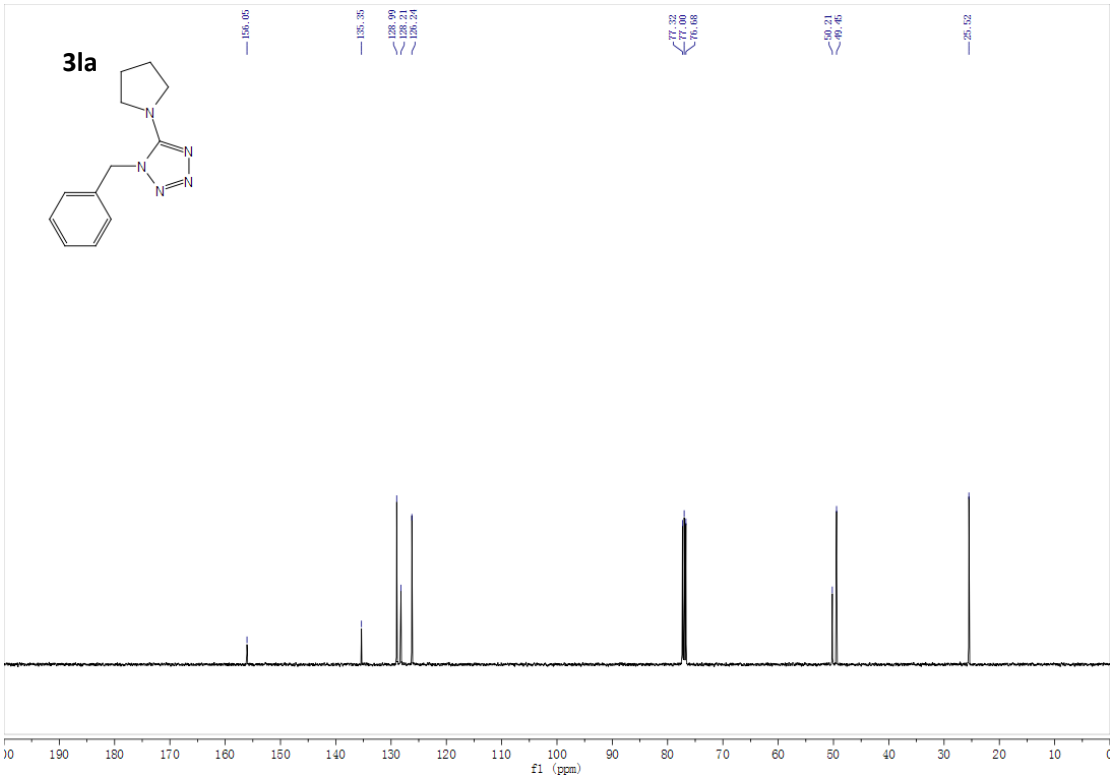
3ja

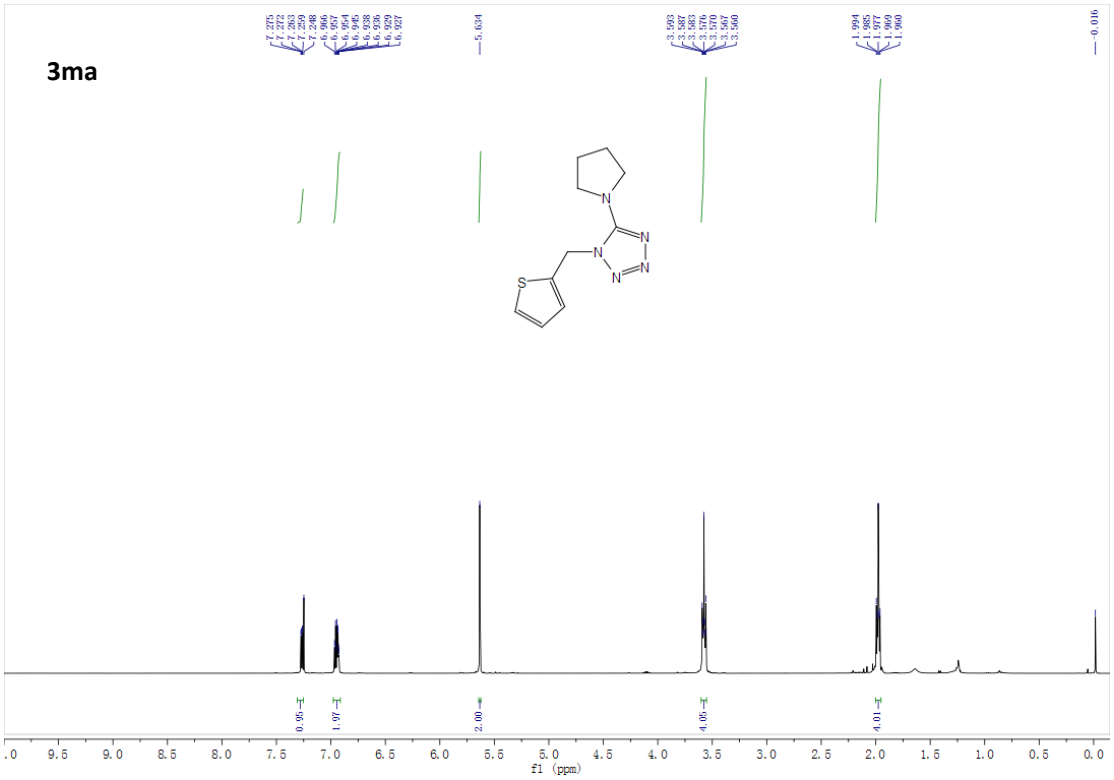

156.38
77.32
77.00
76.68
54.09
49.58
29.01
25.51
19.70
f1 (ppm)

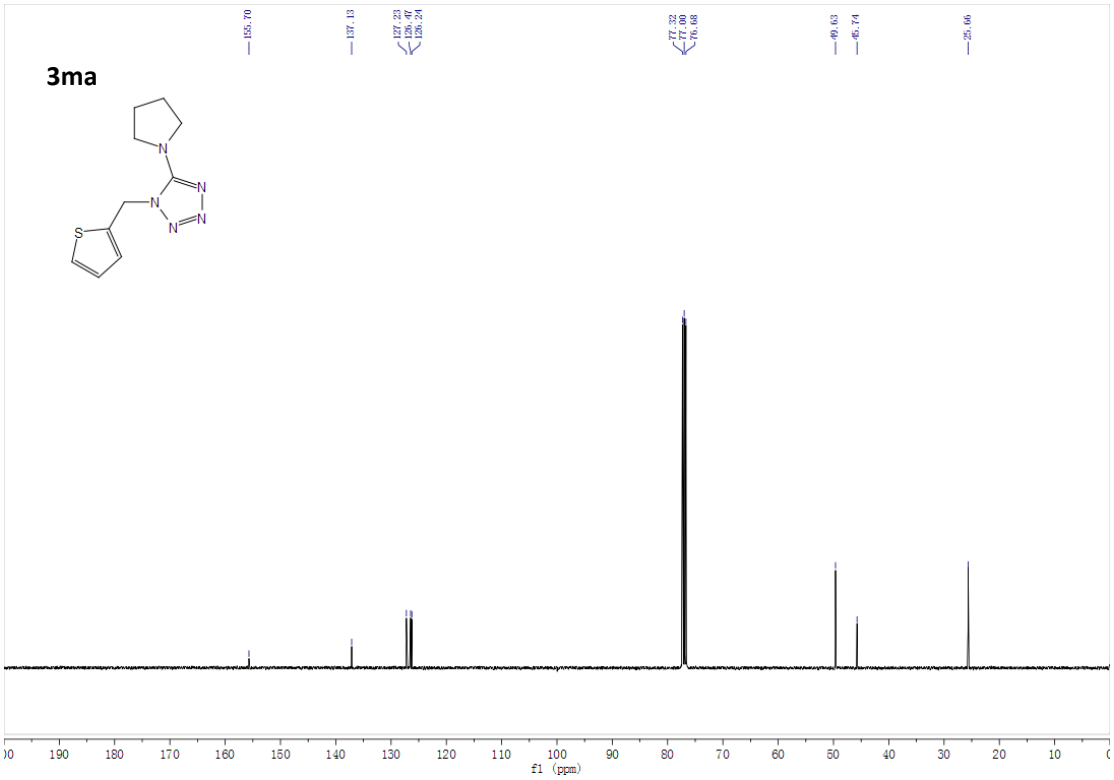

3ka
7.291
4.144
4.125
4.108
4.099
4.089
4.010
3.995
3.975
3.958
3.570
3.562
3.555
3.539
2.163
2.152
2.146
2.134
2.128
2.117
2.110
2.099
2.092
2.046
2.037
2.029
2.020
2.012
1.277
1.271
1.242
1.235
1.160
1.143
1.125
1.107
0.986
0.932
0.850
0.000
1.02
1.00
4.03
1.00
4.01
1.12
1.04
2.99
9.00
f1 (ppm)

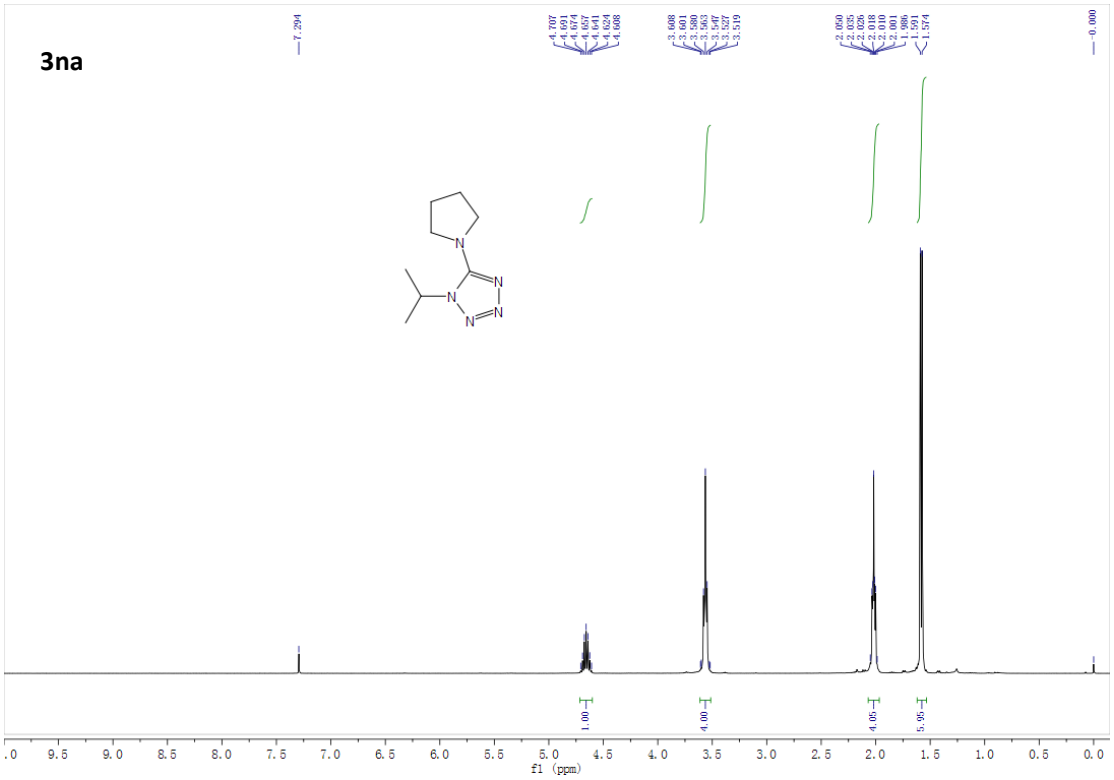

3ka
156.582
77.318
77.000
76.682
54.113
49.625
47.550
30.824
30.076
29.647
25.532
20.049
f1 (ppm)

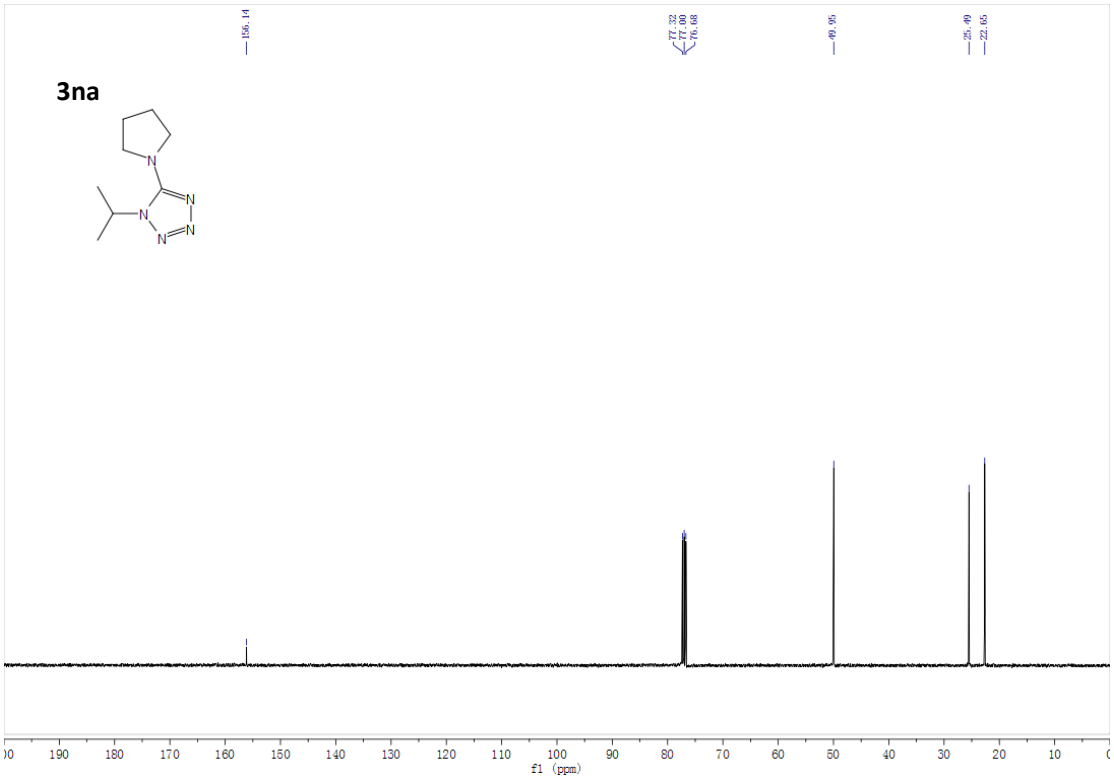

3la
7.373
7.367
7.363
7.351
7.348
7.337
7.333
7.329
7.325
7.320
7.312
7.278
7.141
7.137
7.129
5.521
3.520
3.515
3.509
3.503
3.497
3.493
3.486
1.940
1.932
1.923
1.915
1.907
0.000
3.04
2.00
2.00
4.03
4.05
f1 (ppm)

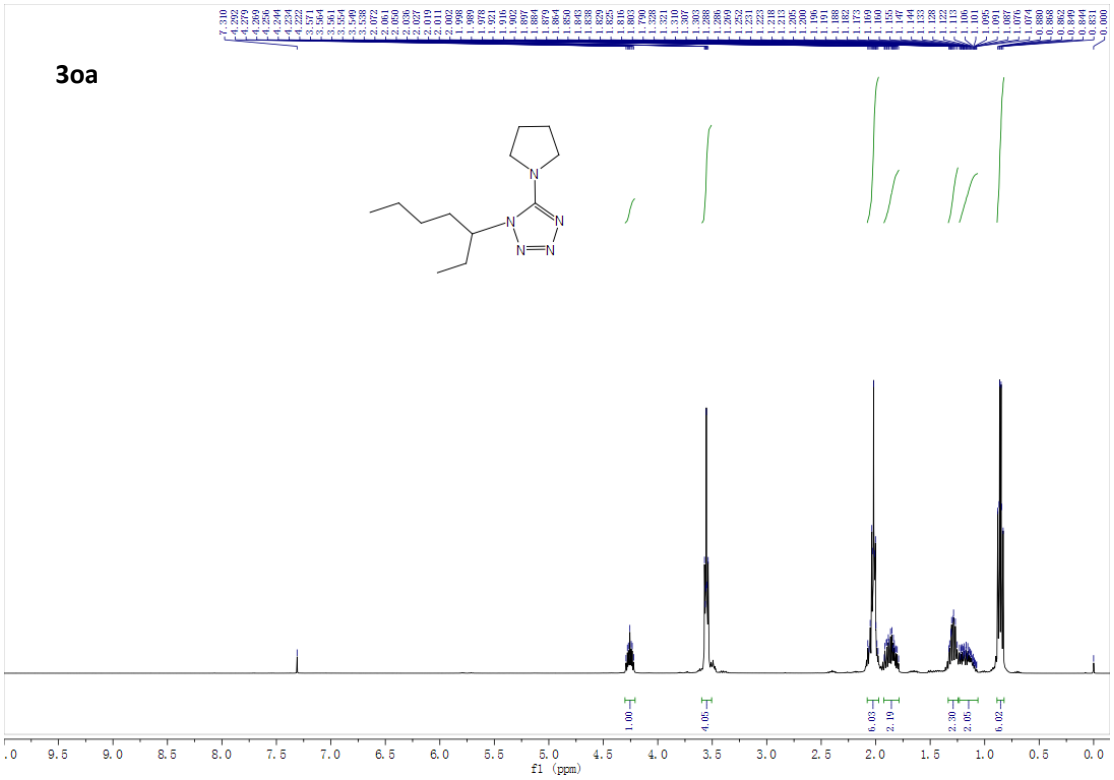

3la
156.05
135.35
128.99
128.21
126.24
77.32
77.00
76.68
50.21
49.45
25.52
f1 (ppm)

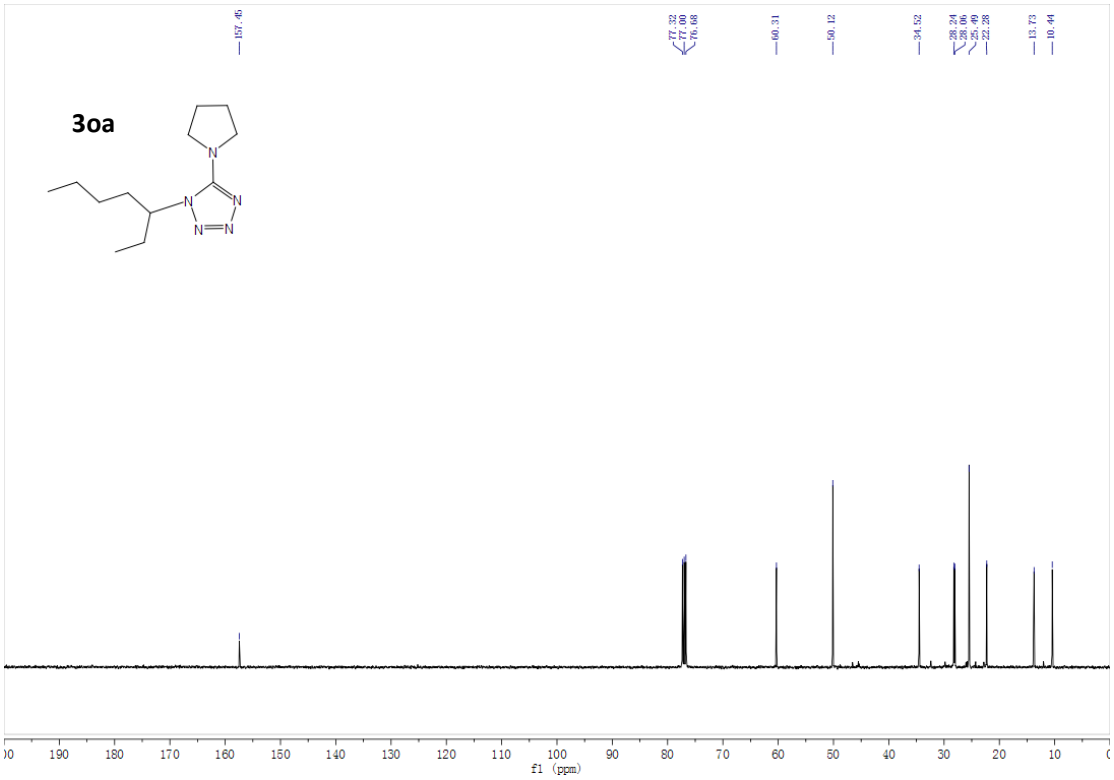

3ma
7.275
7.272
7.263
7.259
7.248
6.966
6.957
6.954
6.946
6.938
6.938
6.929
6.927
5.634
3.593
3.587
3.583
3.576
3.570
3.567
3.560
1.994
1.985
1.977
1.969
1.960
-0.016
0.95
1.97
2.00
4.01
4.01
f1 (ppm)

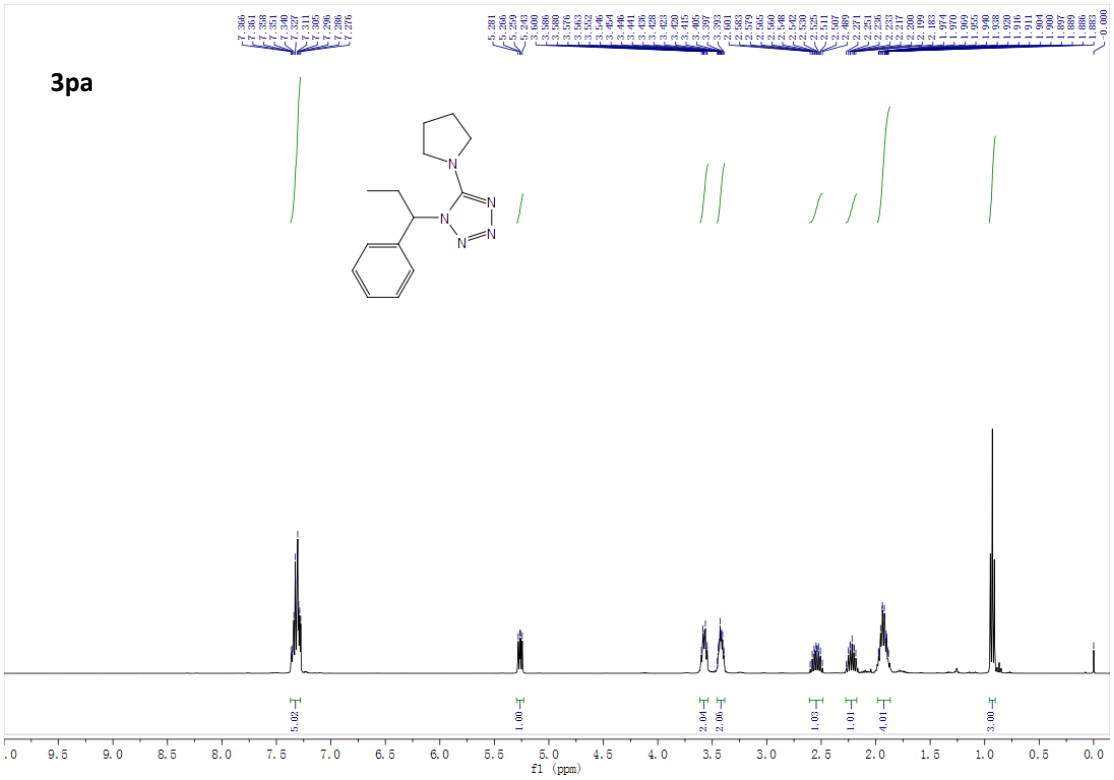

3ma
155.70
137.13
127.23
126.47
126.24
77.32
77.00
76.68
49.63
45.74
25.66
f1 (ppm)

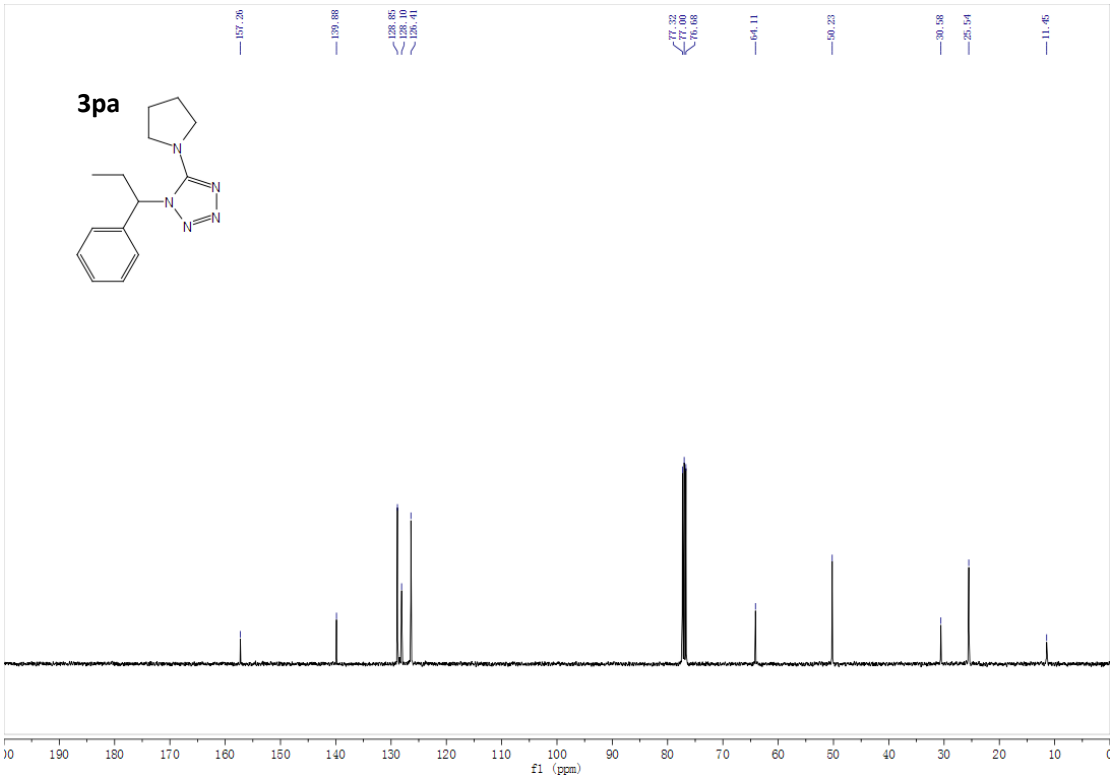

3na
7.294
4.707
4.691
4.674
4.657
4.641
4.624
4.608
3.608
3.601
3.580
3.563
3.547
3.527
3.519
2.050
2.035
2.026
2.018
2.010
2.001
1.986
1.991
1.974
0.000
1.00
4.00
4.05
5.95
f1 (ppm)

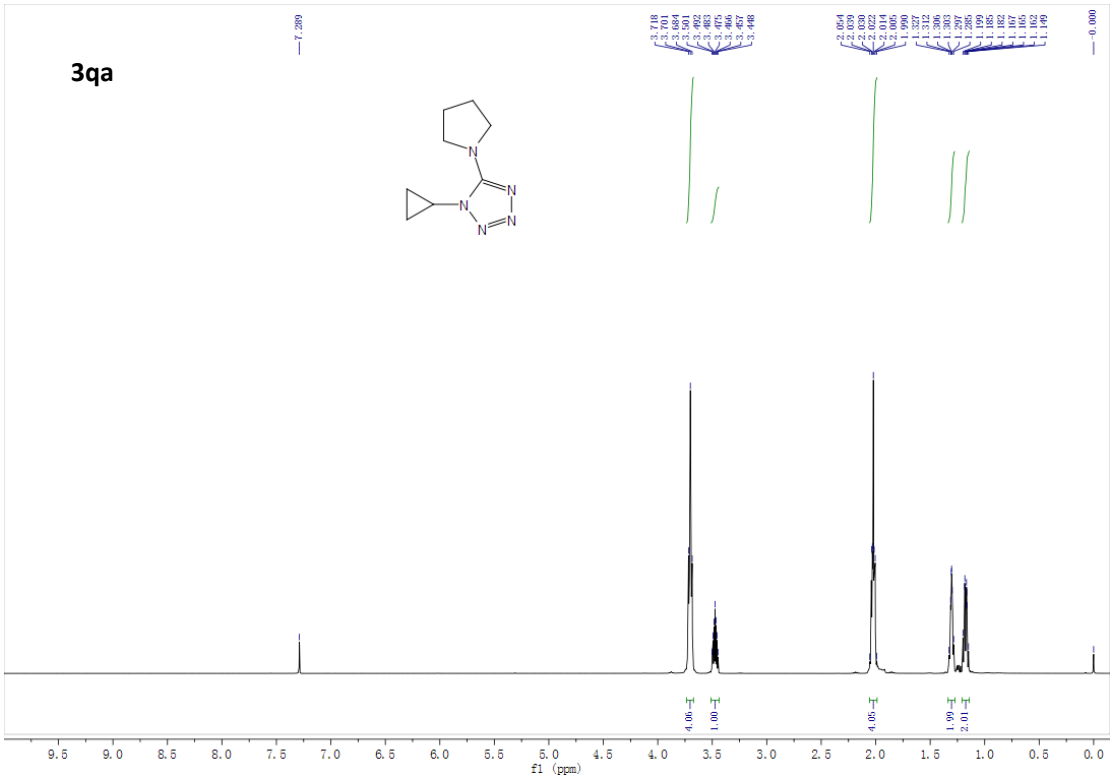

3na
156.14
77.32
77.00
76.68
49.95
25.49
22.65
f1 (ppm)

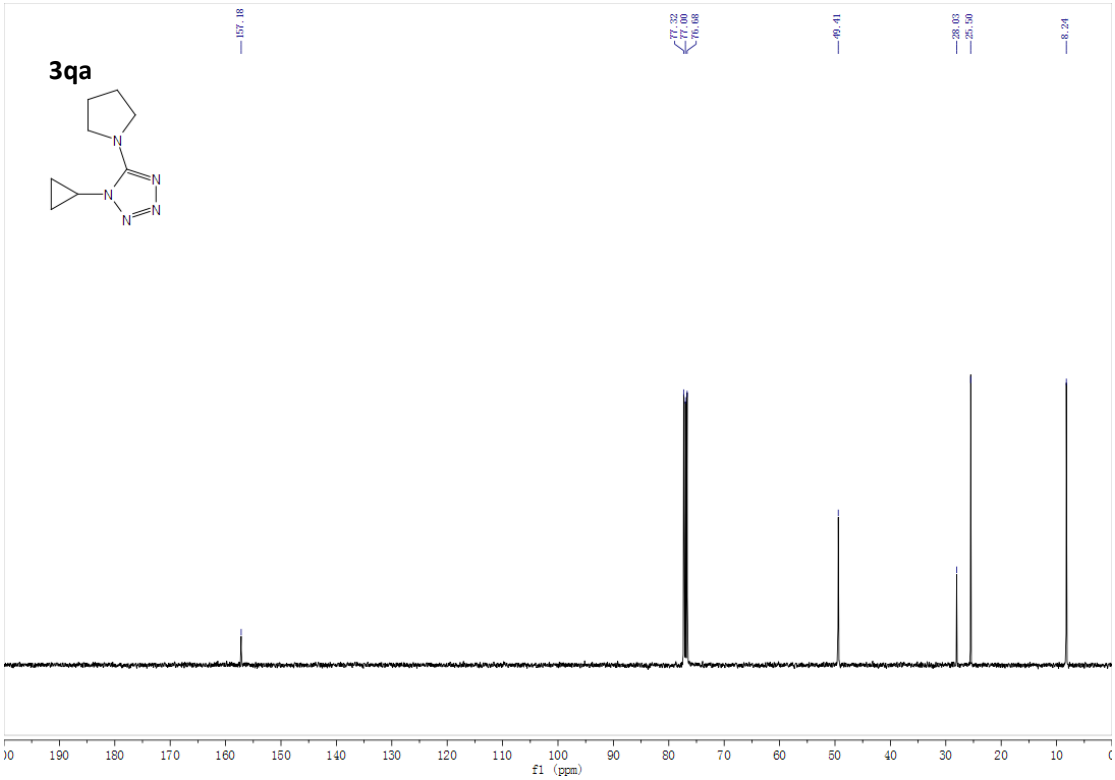

3oa
f1 (ppm)


3oa
f1 (ppm)

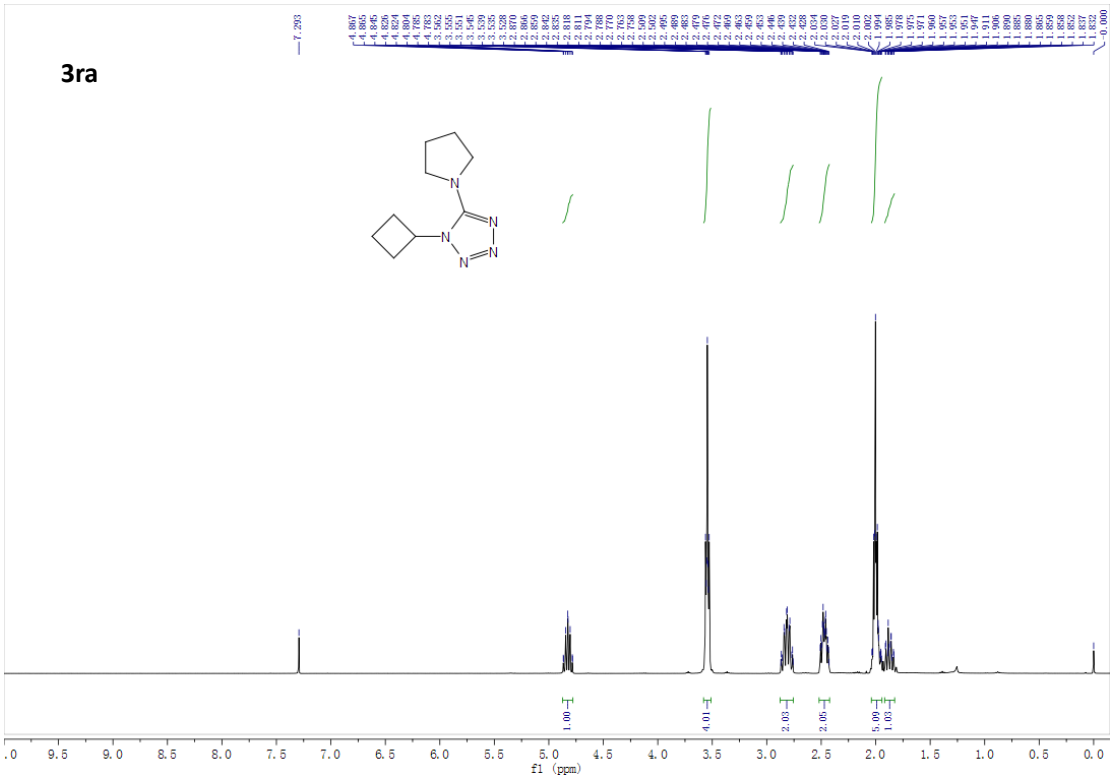

3pa
f1 (ppm)


3pa
f1 (ppm)


3qa
7.289
3.718
3.701
3.684
3.501
3.492
3.483
3.475
3.466
3.457
3.448
2.054
2.039
2.030
2.022
2.014
2.005
1.990
1.327
1.312
1.306
1.303
1.297
1.295
1.199
1.185
1.182
1.167
1.165
1.162
1.149
0.000
4.06
1.00
4.05
1.99
2.01
f1 (ppm)


3qa
157.18
77.32
77.00
76.68
49.41
28.03
25.50
8.24
f1 (ppm)

3ra



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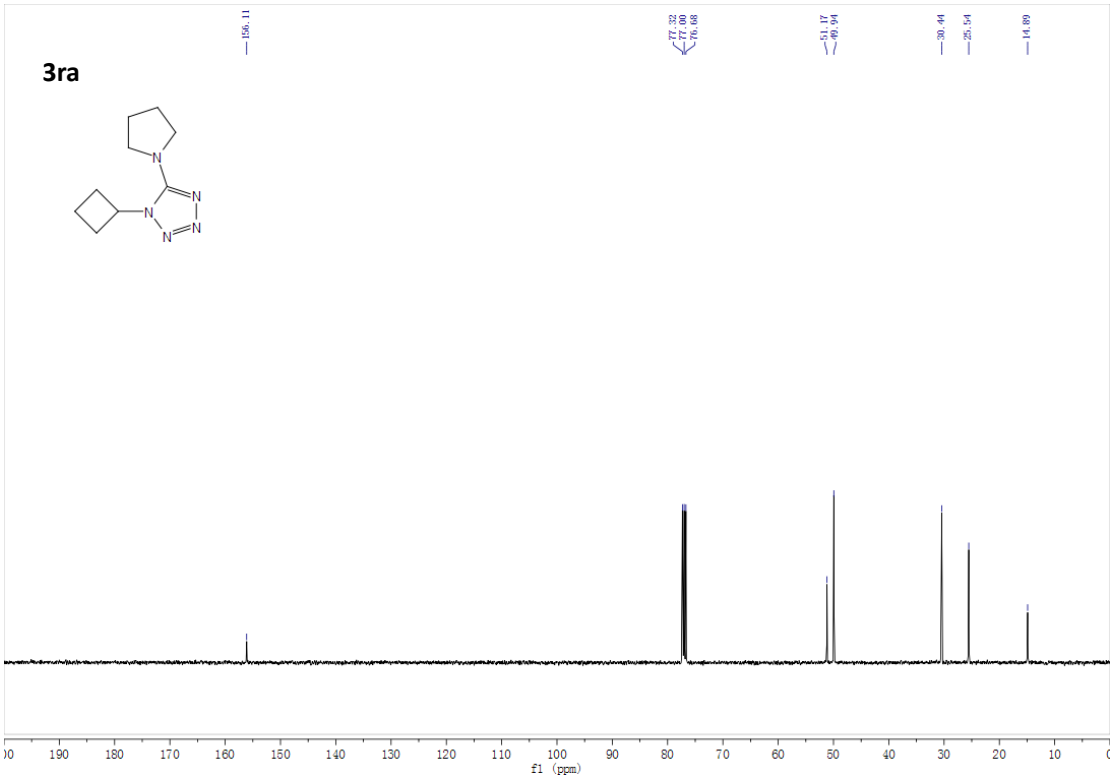
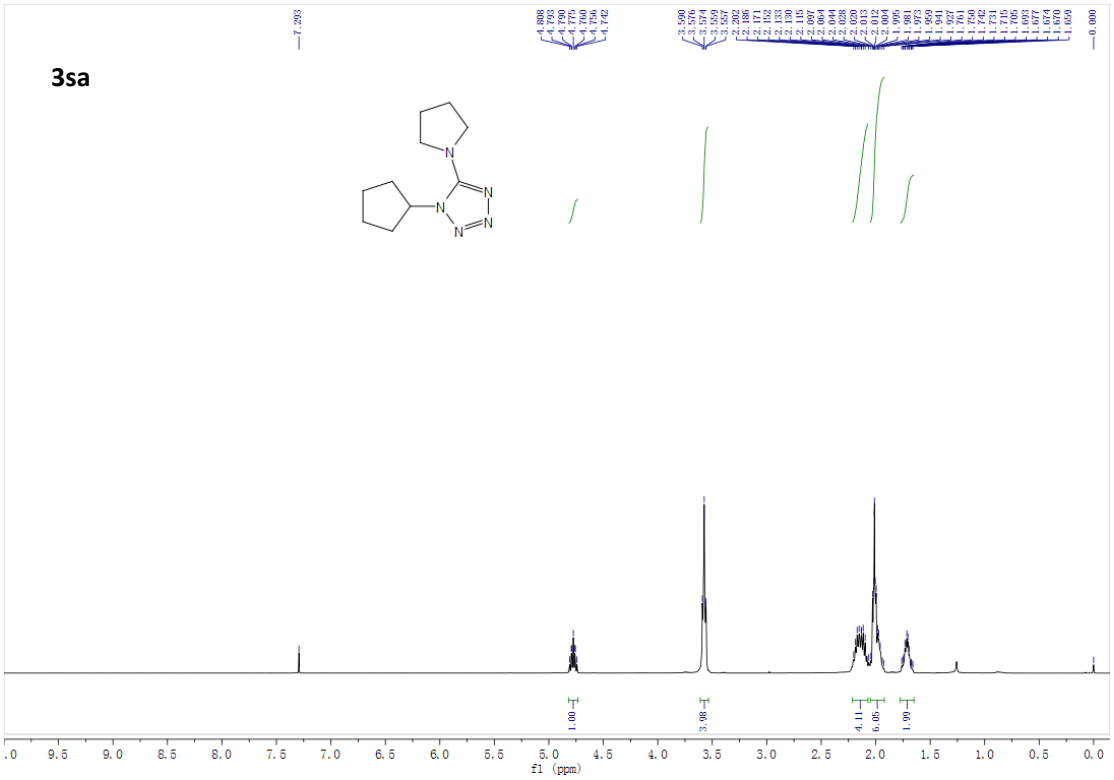
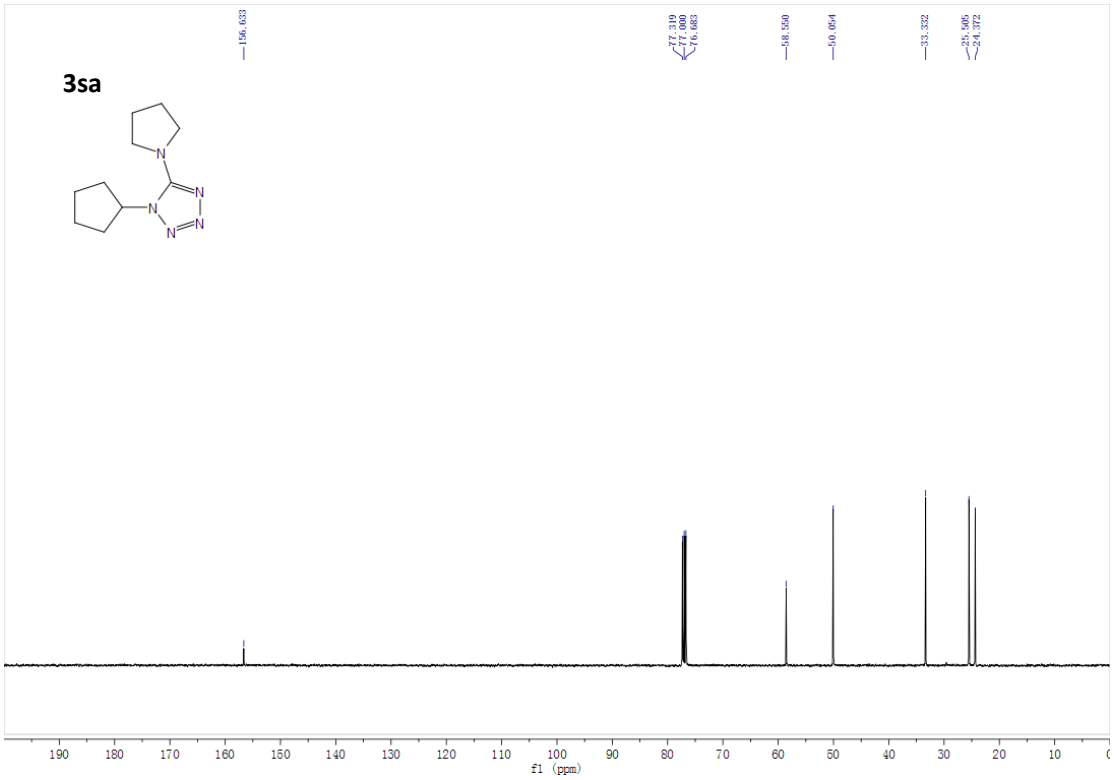
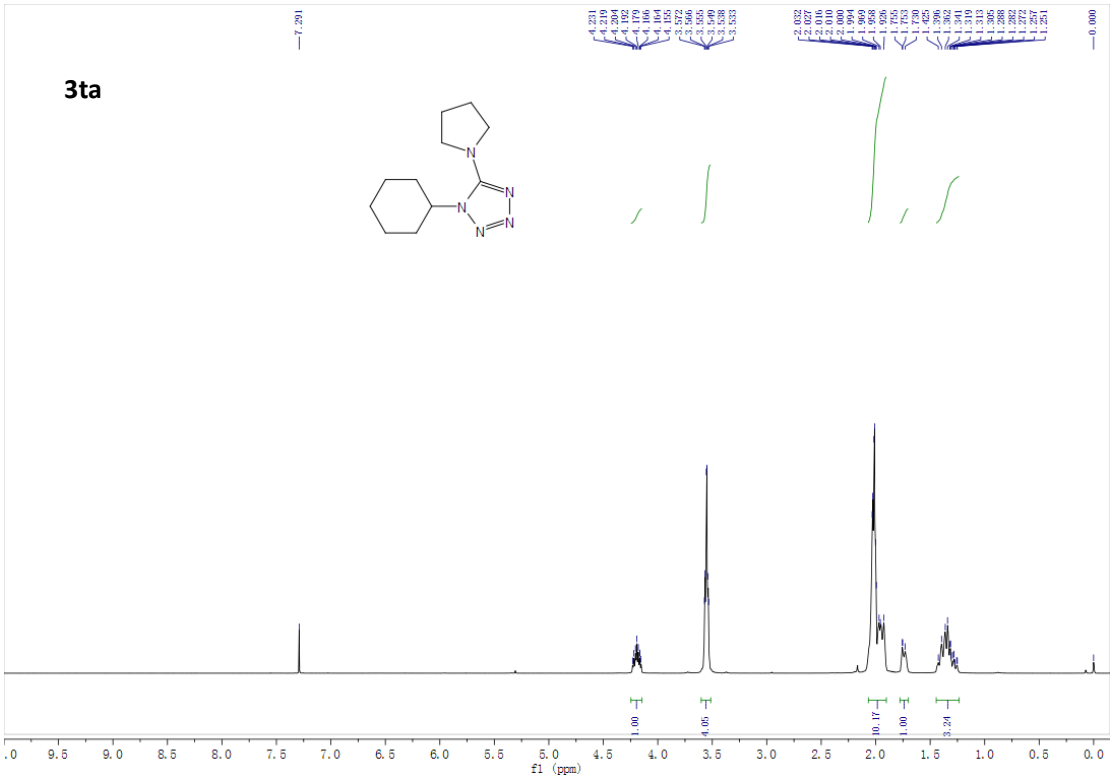


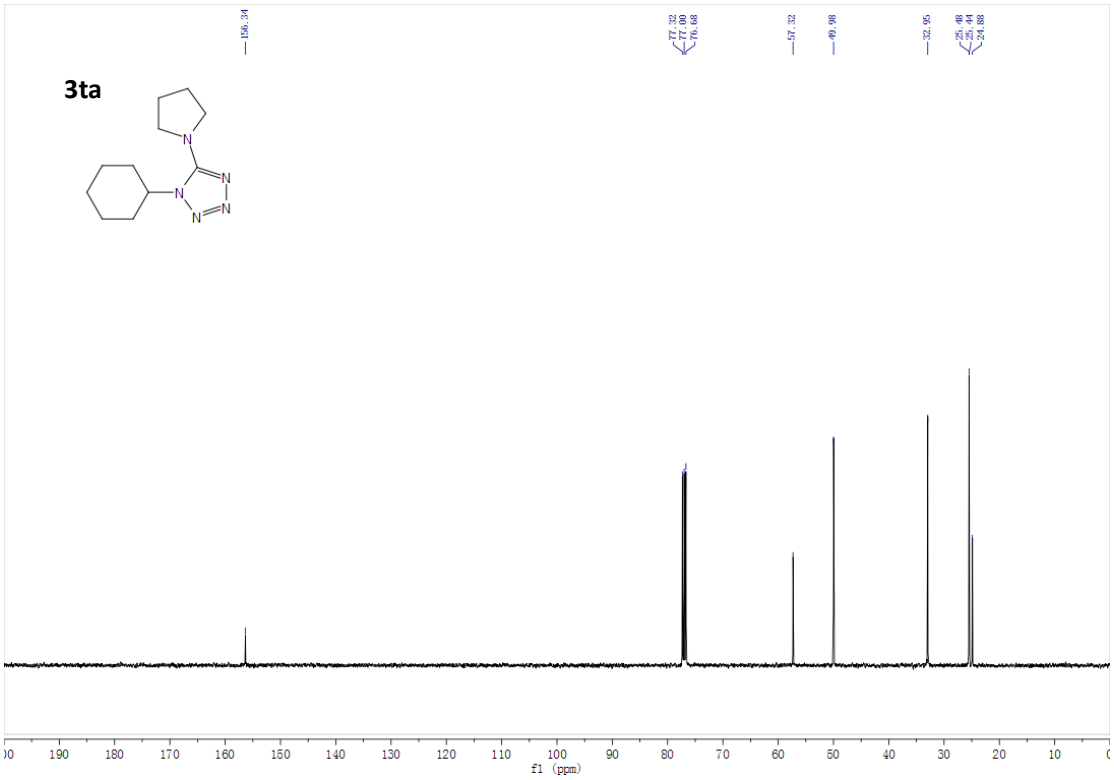
3sa

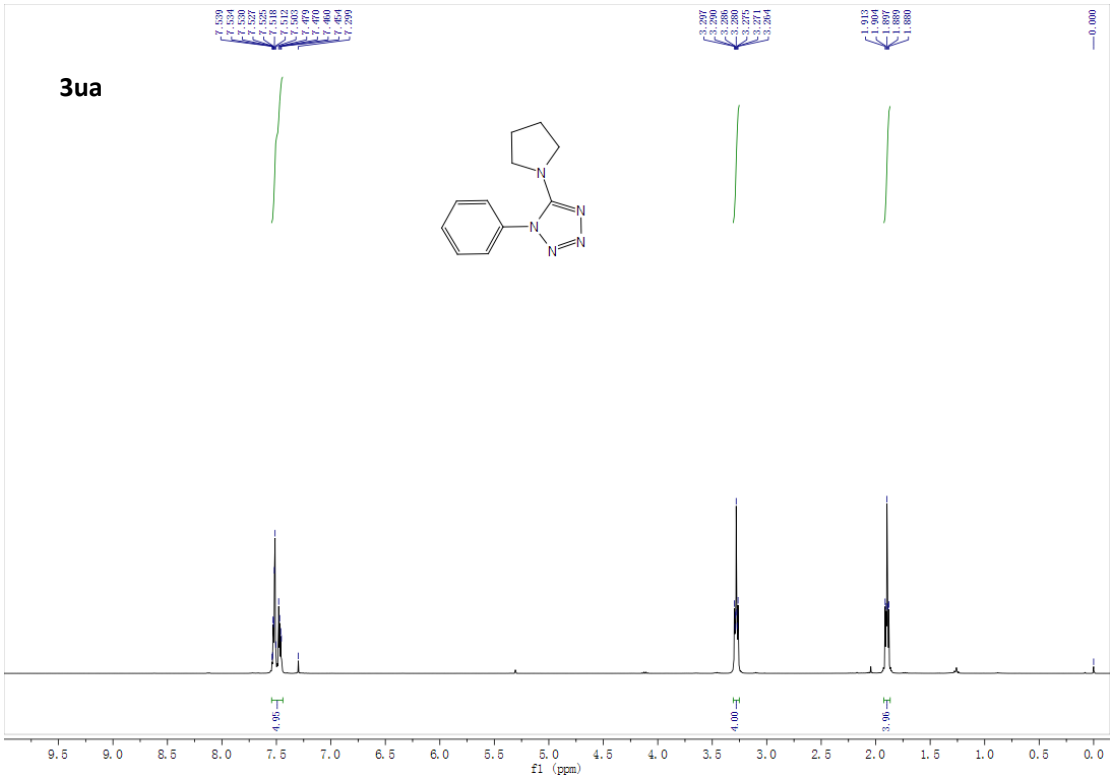


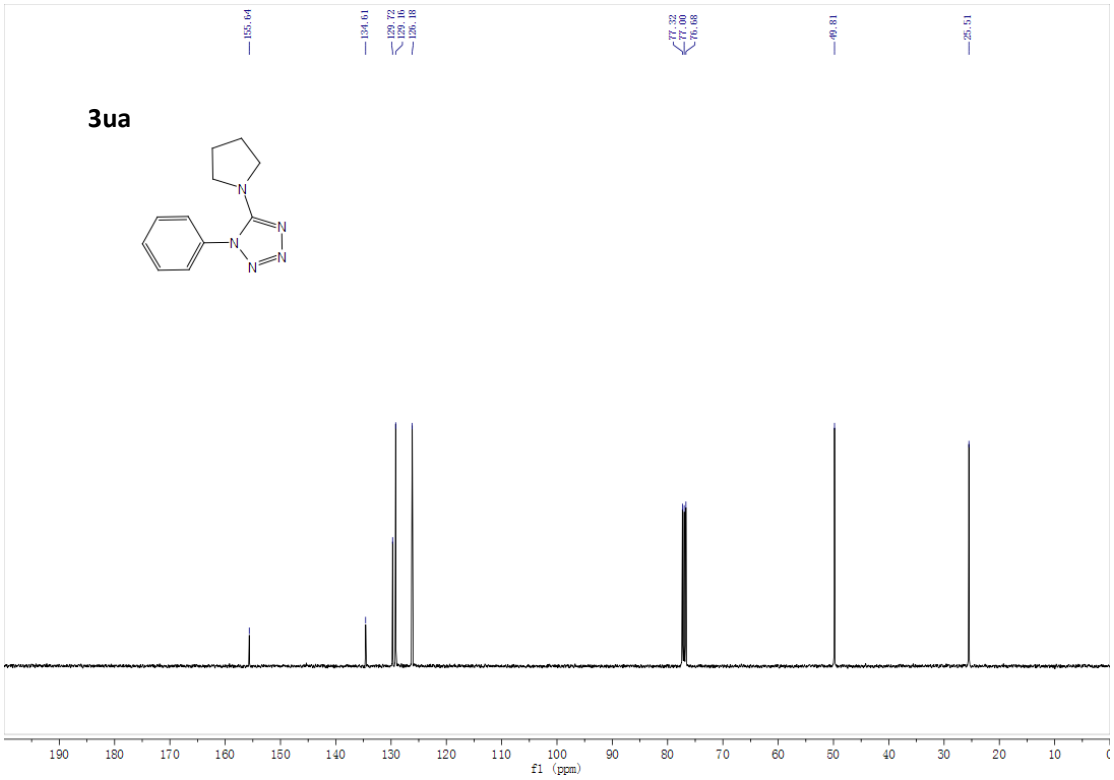
3sa

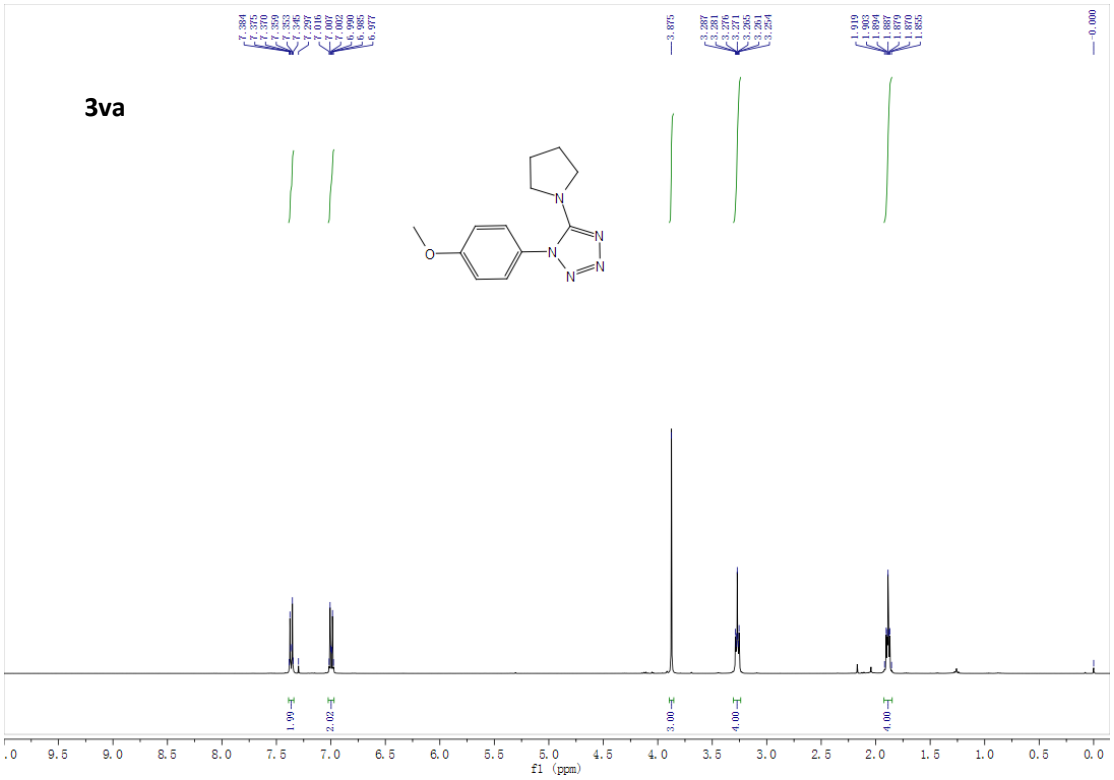


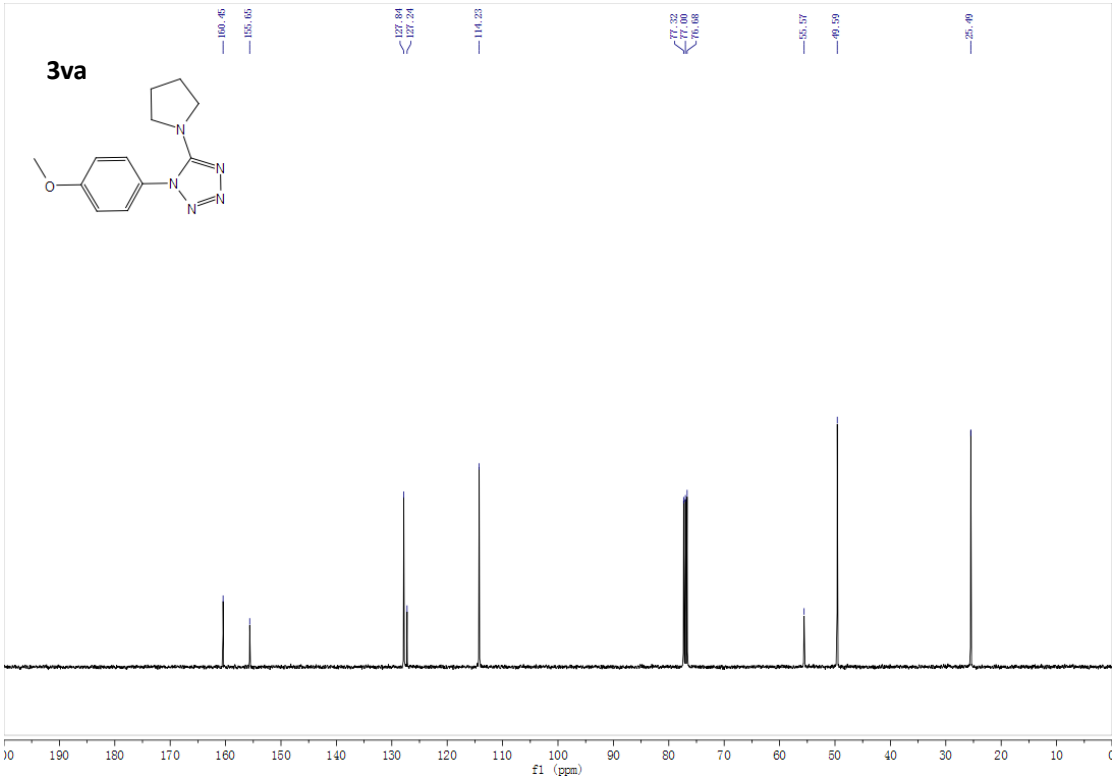

3ta
f1 (ppm)

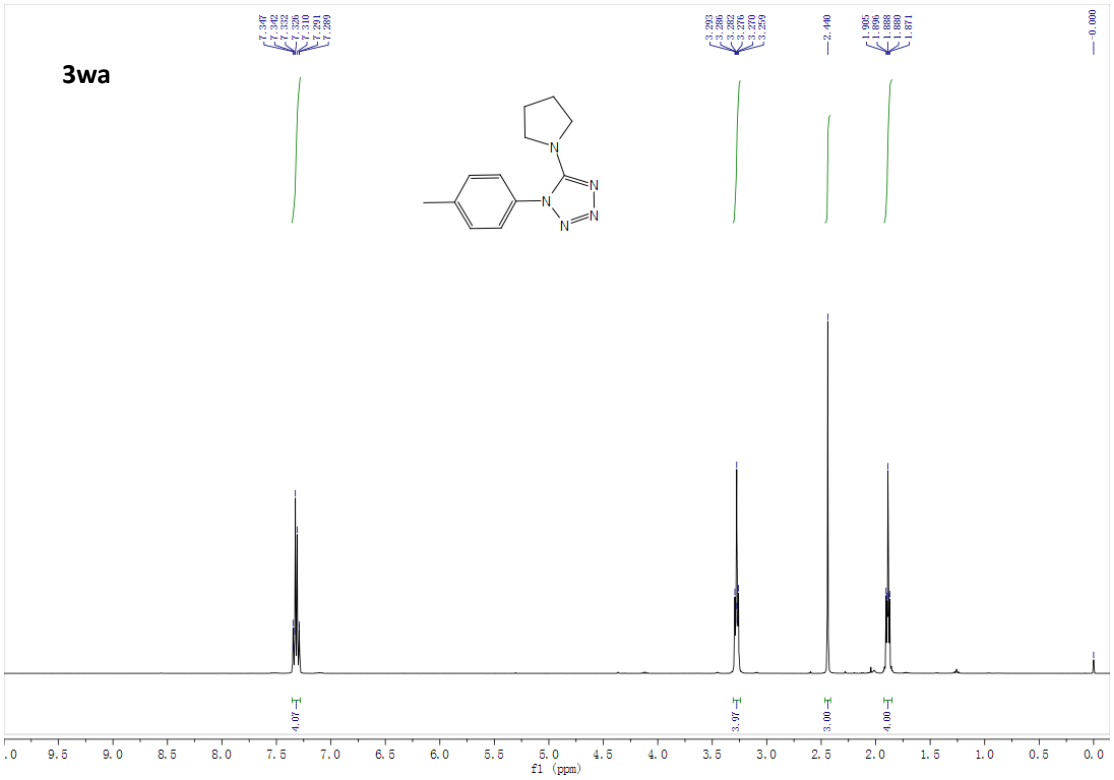

3ta
f1 (ppm)

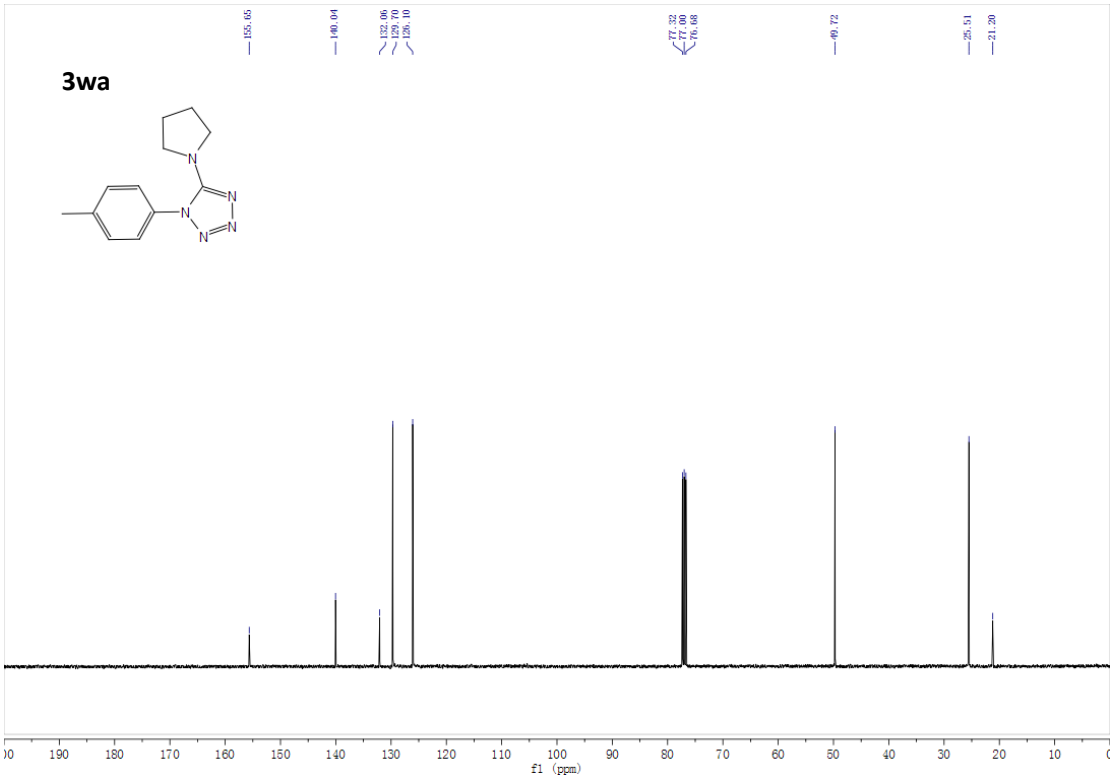

3ua
7.539
7.534
7.530
7.527
7.525
7.518
7.512
7.505
7.479
7.470
7.460
7.454
7.299
3.297
3.290
3.286
3.280
3.275
3.271
3.264
1.913
1.904
1.897
1.889
1.880
0.000
4.95
4.00
3.96
f1 (ppm)


3ua
155.64
134.61
129.72
129.16
126.18
77.32
77.00
76.68
49.81
25.51
f1 (ppm)


3va
7.384
7.375
7.370
7.359
7.353
7.345
7.297
7.016
7.007
7.002
6.990
6.985
6.977
3.875
3.287
3.281
3.276
3.271
3.265
3.261
3.254
1.919
1.903
1.894
1.887
1.879
1.870
1.855
0.000
1.99
2.02
3.00
4.00
4.00
f1 (ppm)


3va
160.45
155.65
127.84
127.24
114.23
77.32
77.00
76.68
55.57
49.59
25.49
f1 (ppm)


3wa
7.347
7.342
7.332
7.326
7.310
7.291
7.289
3.293
3.286
3.282
3.276
3.270
3.259
2.440
1.905
1.896
1.888
1.880
1.871
0.000
4.07
3.97
3.00
4.00
f1 (ppm)


3wa
155.65
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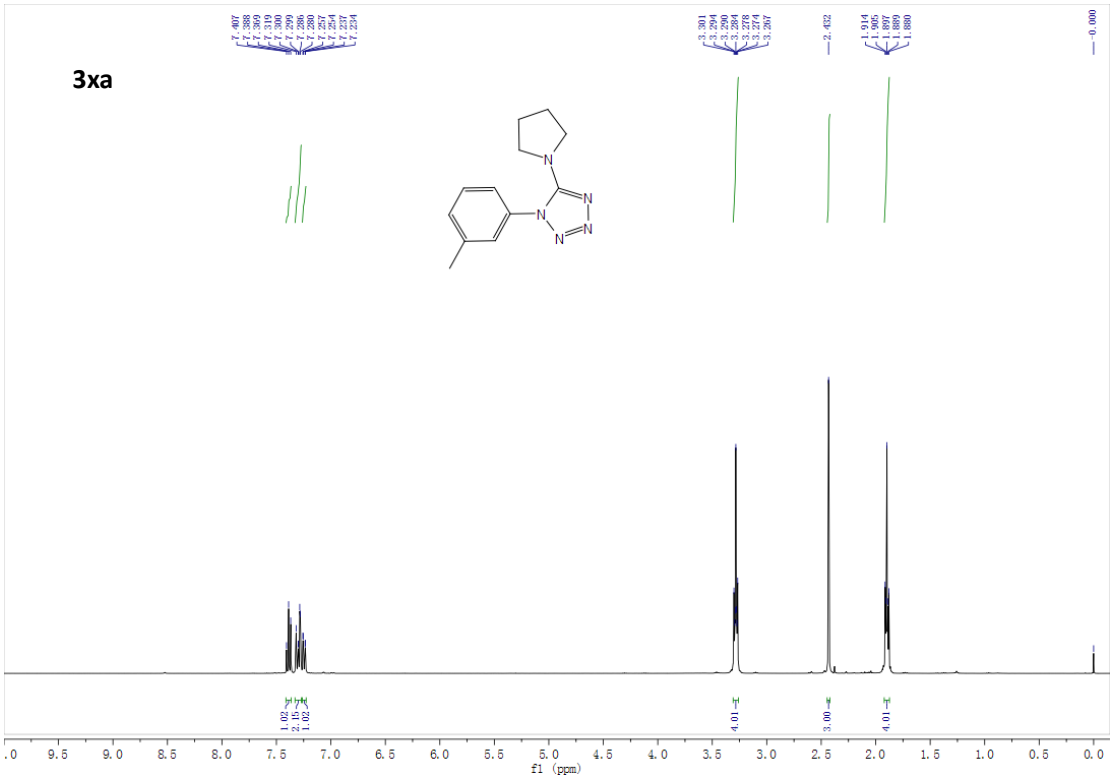
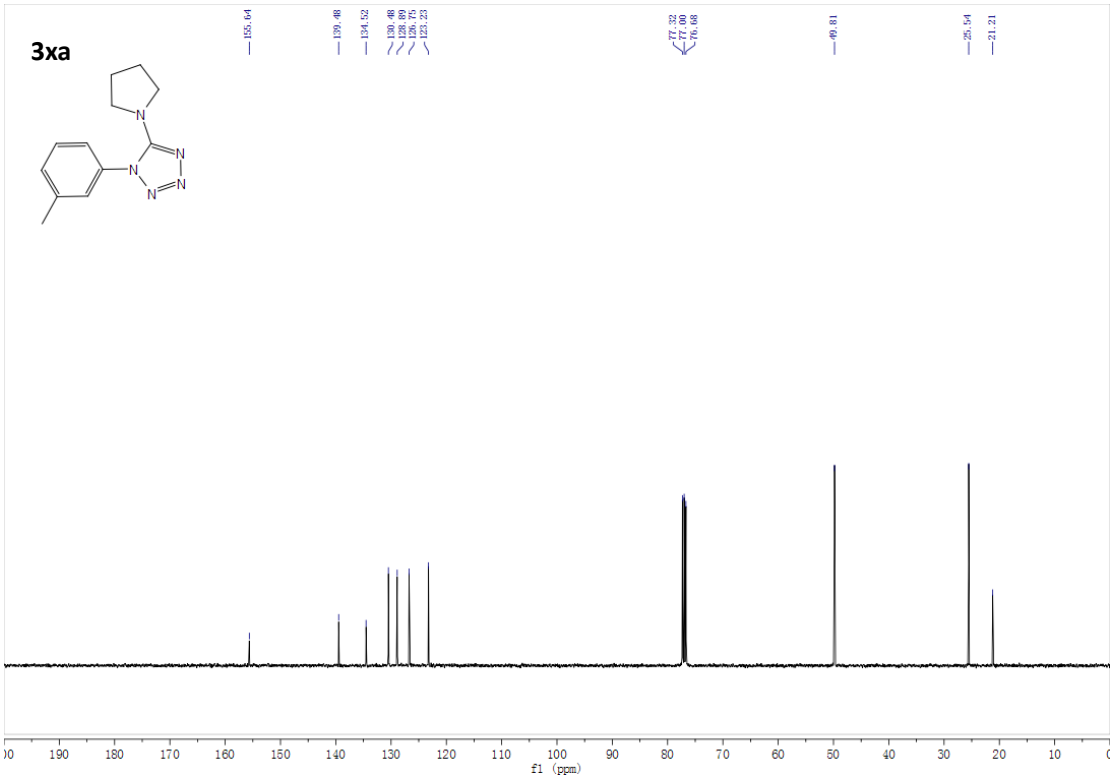
132.06
129.70
128.10
77.32
77.00
76.68
49.72
25.51
21.20
f1 (ppm)


3xa
7.407
7.388
7.369
7.319
7.300
7.299
7.286
7.269
7.254
7.237
7.234
3.301
3.294
3.290
3.284
3.278
3.274
3.267
2.432
1.914
1.905
1.897
1.889
1.880
0.000
1.02
1.15
1.02
4.01
3.00
4.01
f1 (ppm)


3xa
155.64
139.48
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130.48
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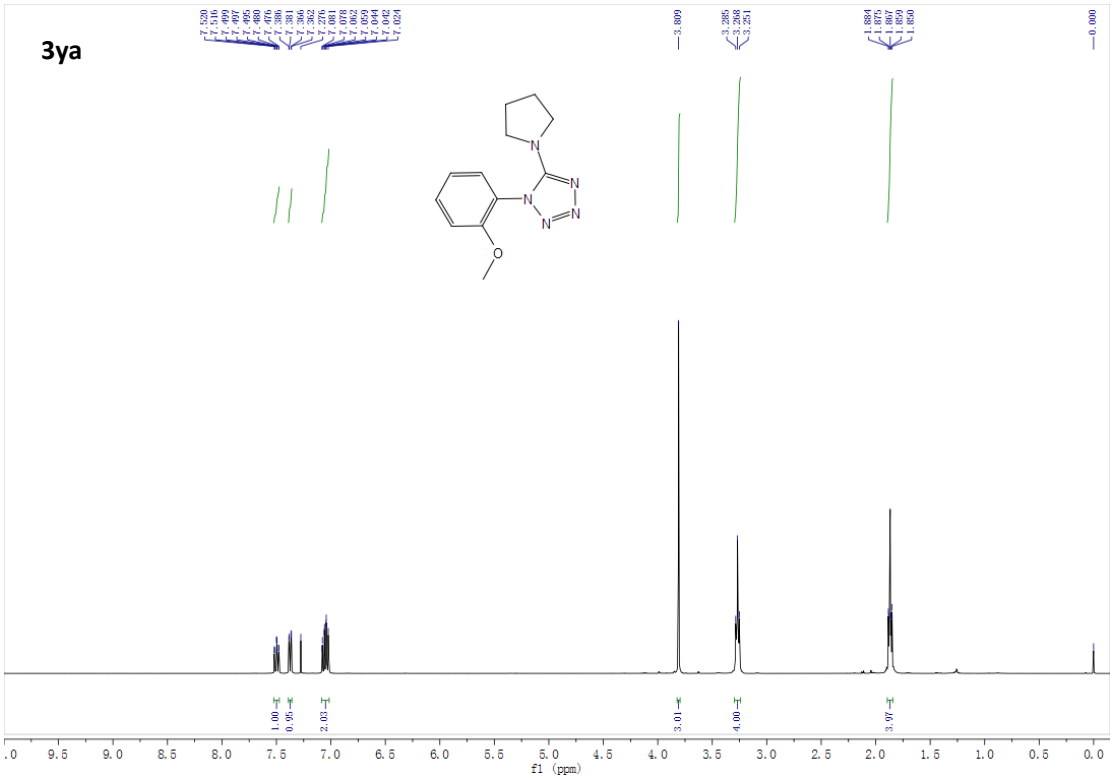
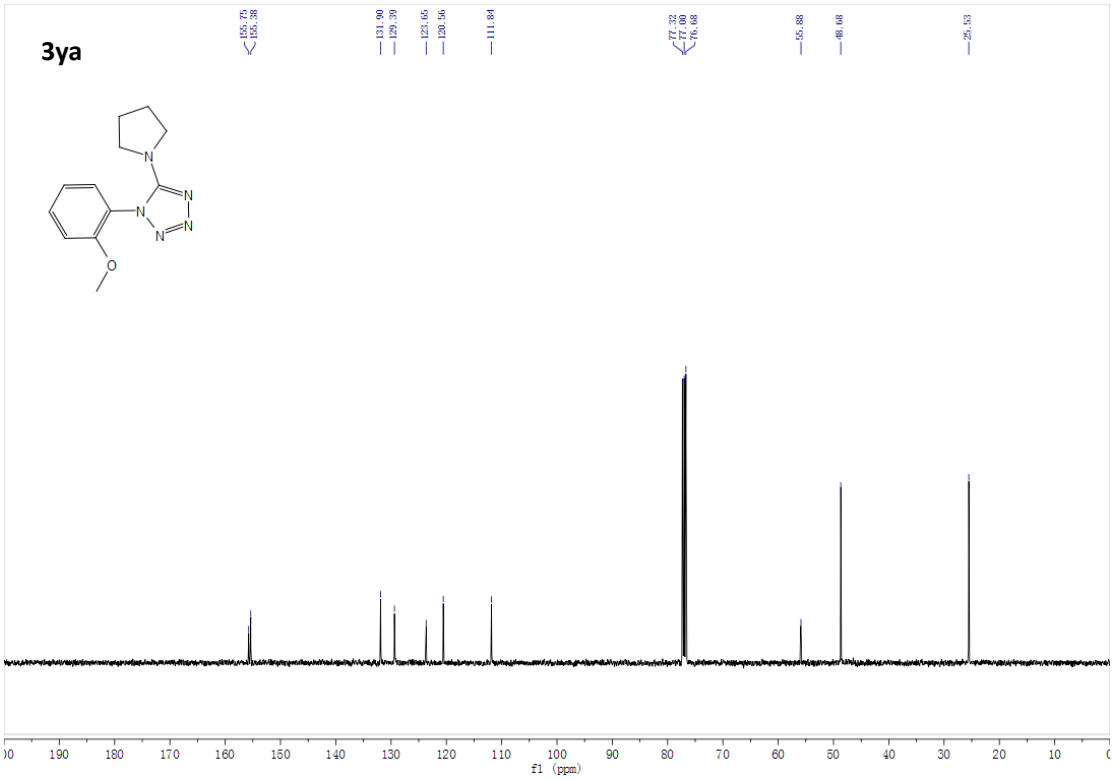
126.75
123.23
77.32
77.00
76.68
49.81
25.54
21.21
f1 (ppm)


3ya
7.520
7.516
7.499
7.497
7.495
7.480
7.466
7.398
7.381
7.366
7.362
7.356
7.081
7.078
7.062
7.060
7.044
7.042
7.024
3.899
3.285
3.268
3.251
1.884
1.875
1.867
1.850
1.840
0.000
1.00
0.95
2.03
3.01
4.00
3.97
f1 (ppm)


3ya
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155.38
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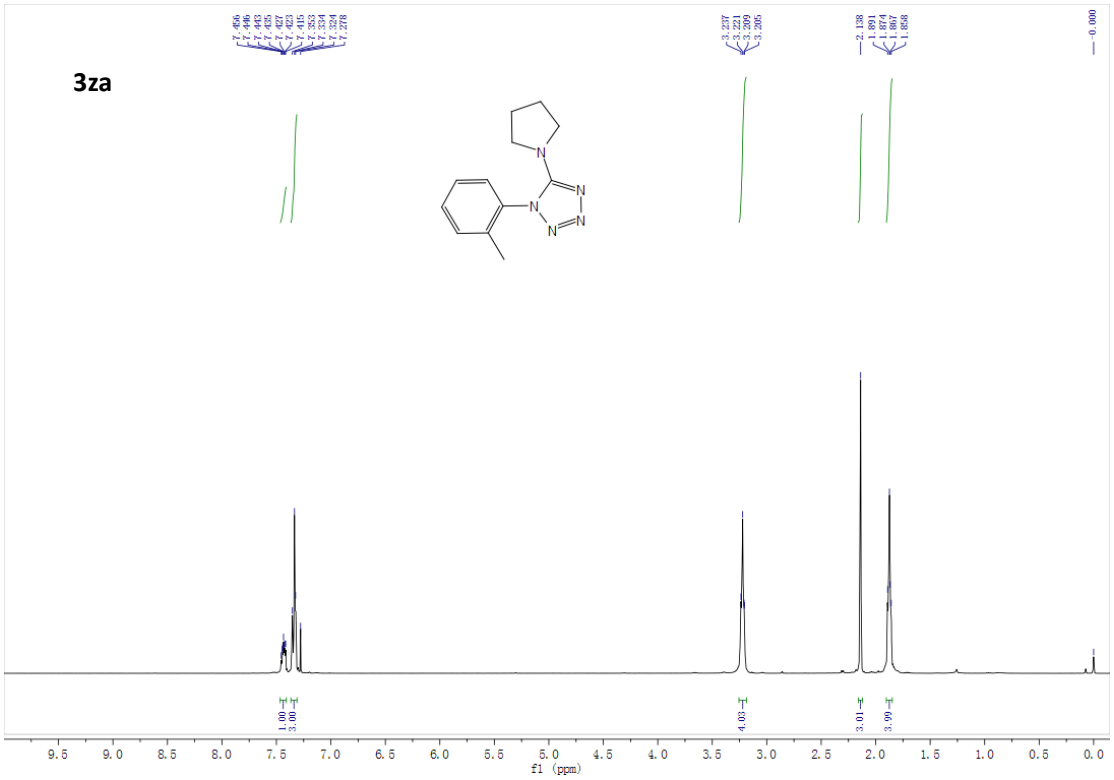
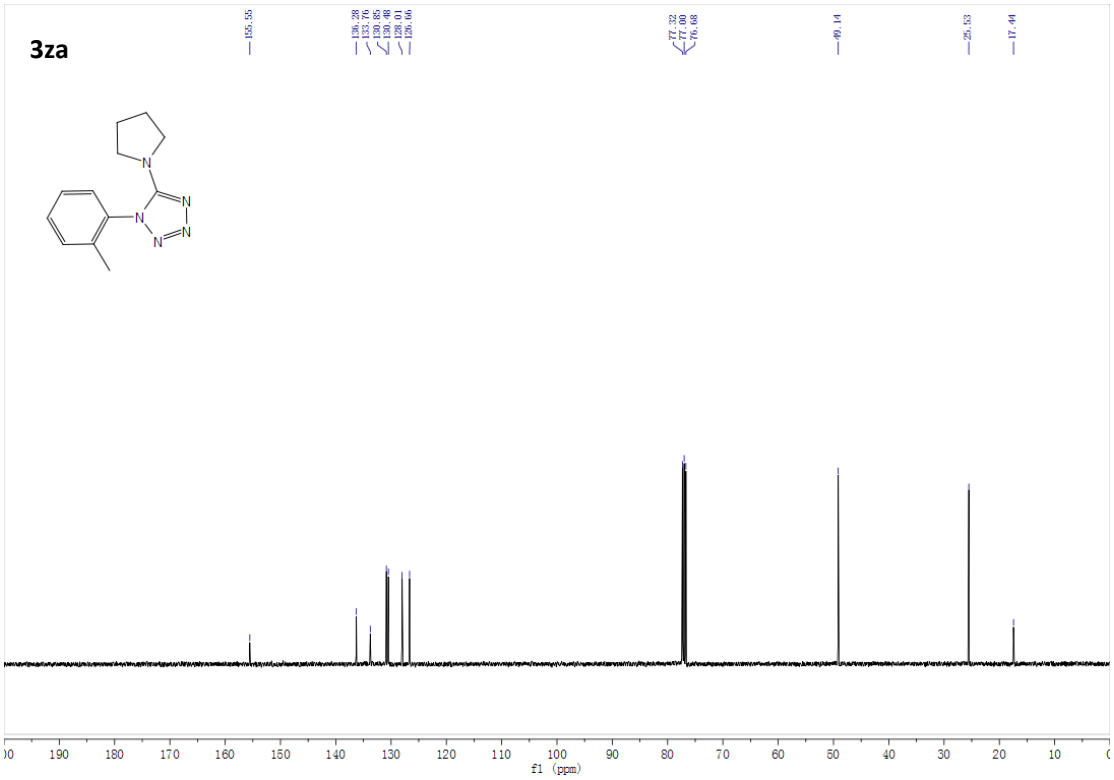
129.39
123.65
120.56
111.84
77.32
77.00
76.68
55.88
48.68
25.53
f1 (ppm)


3za
7.456
7.446
7.443
7.435
7.427
7.423
7.415
7.353
7.334
7.324
7.278
3.237
3.221
3.209
3.205
2.138
1.891
1.874
1.867
1.858
0.000
1.00
3.00
4.03
3.01
3.99
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)


3za
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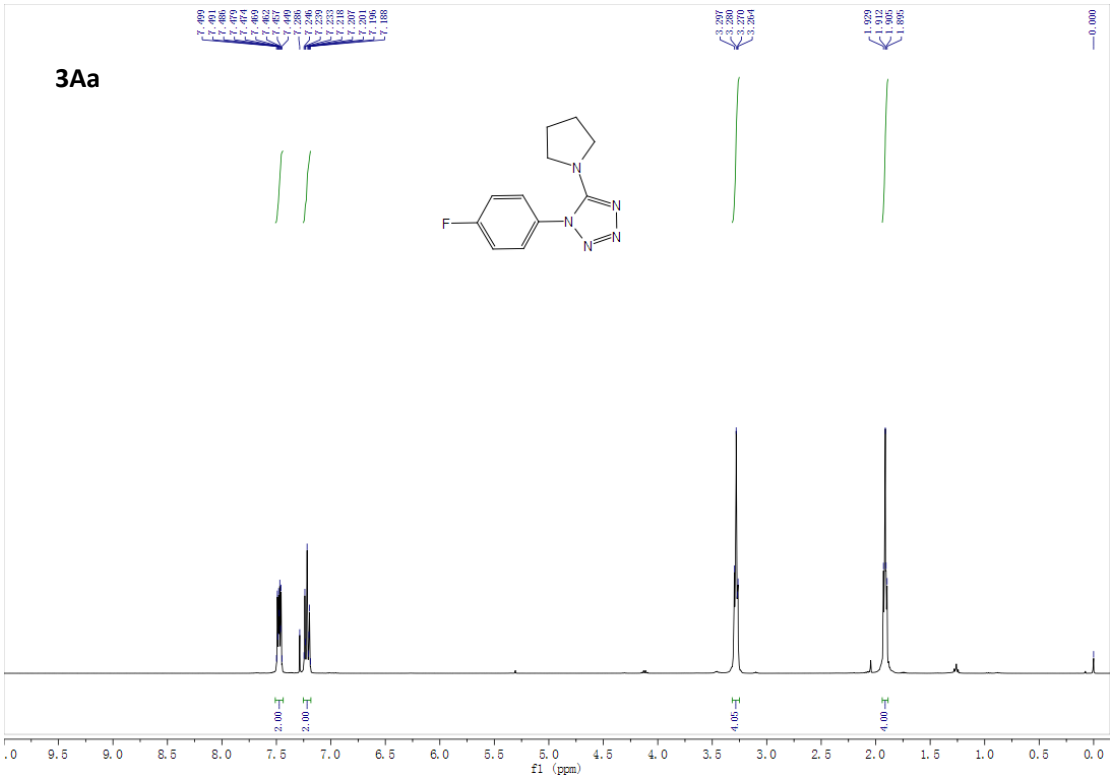
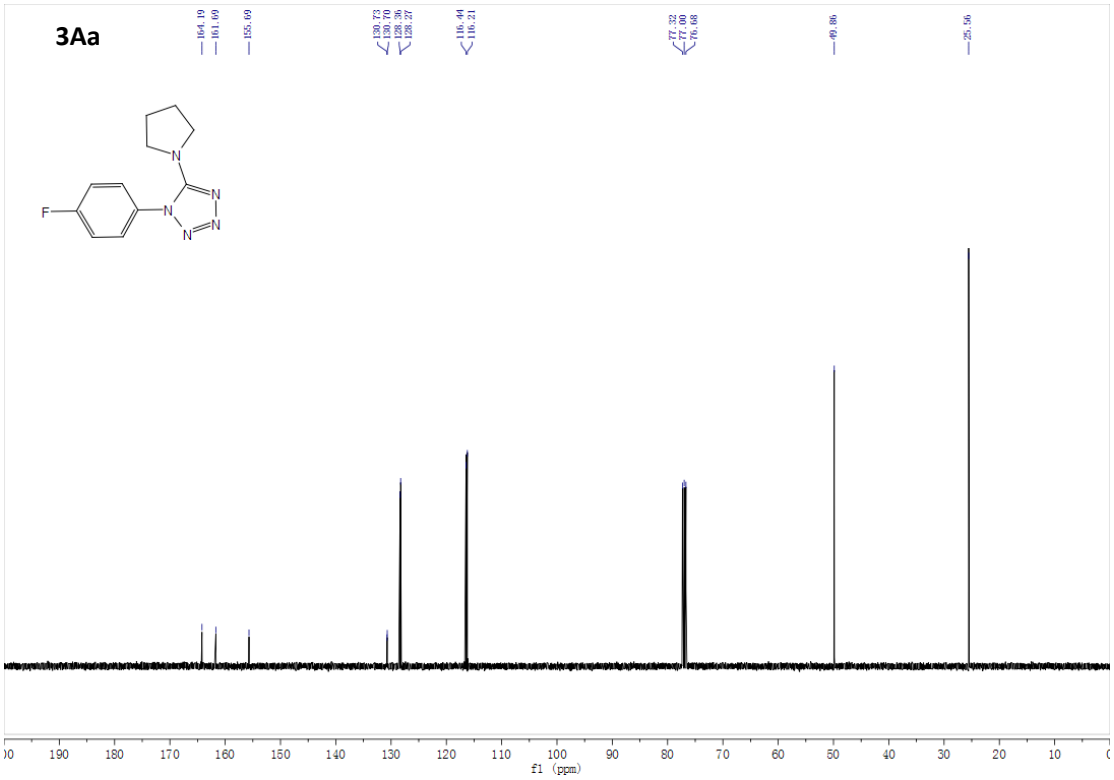
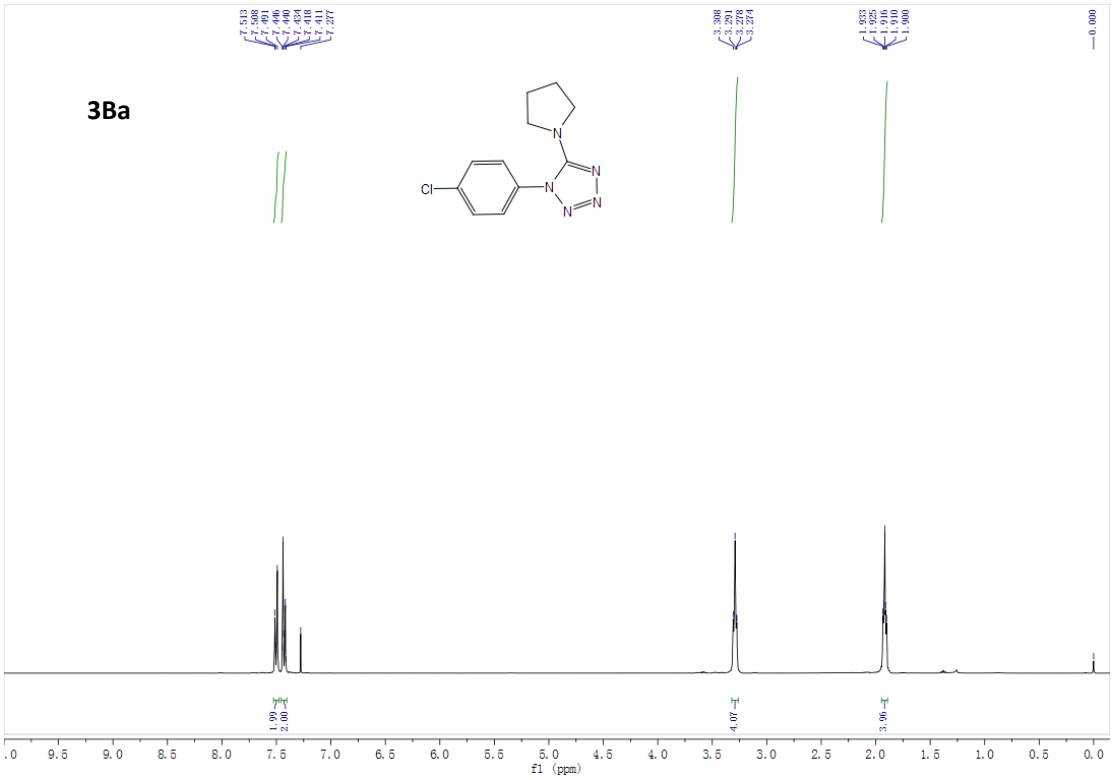
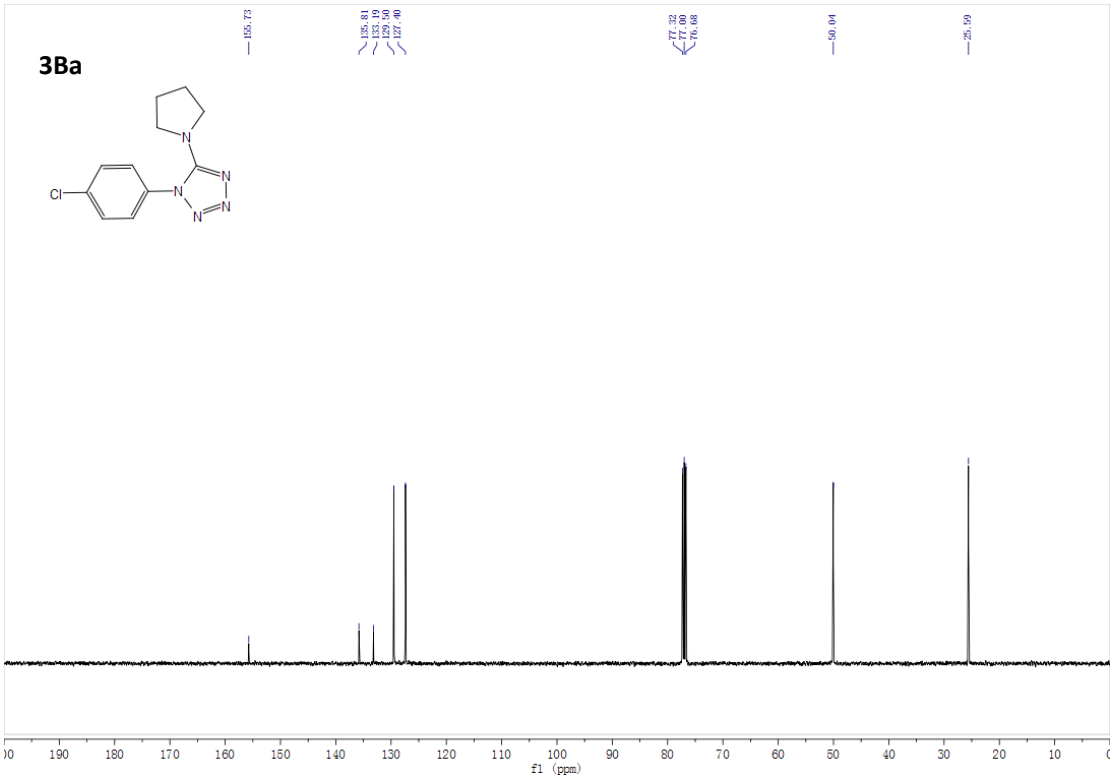
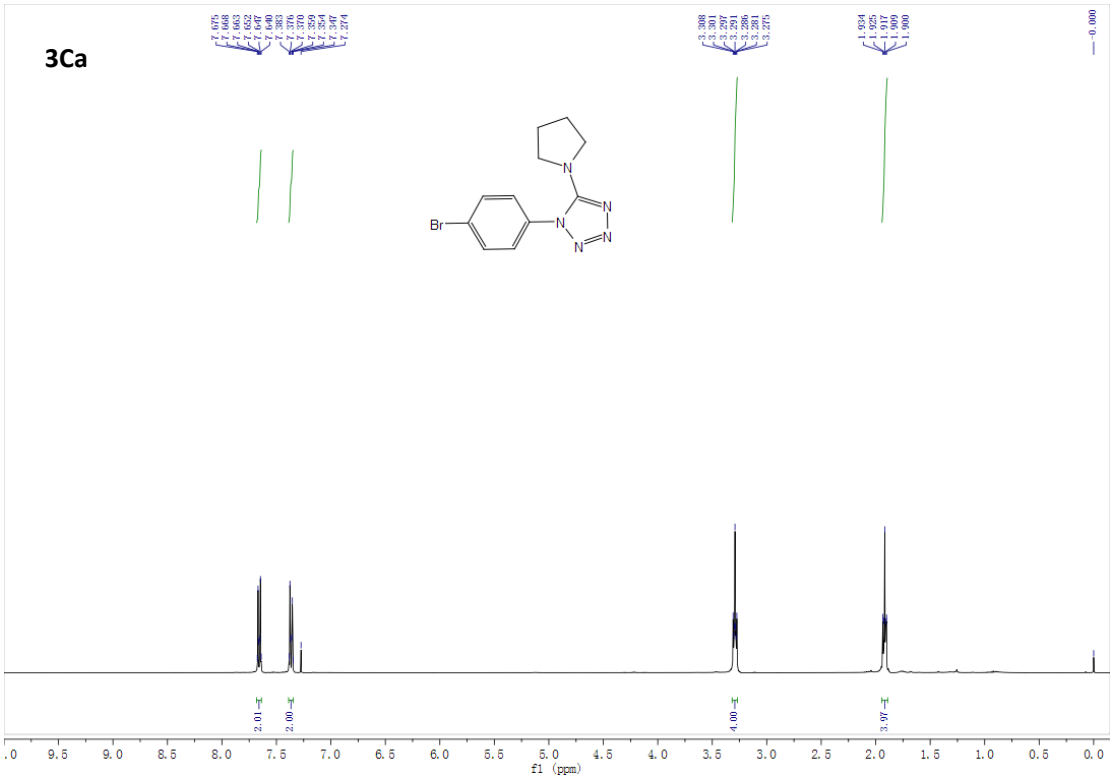
136.28
133.76
130.85
130.48
128.01
125.66
77.32
77.00
76.68
49.14
25.53
17.44
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

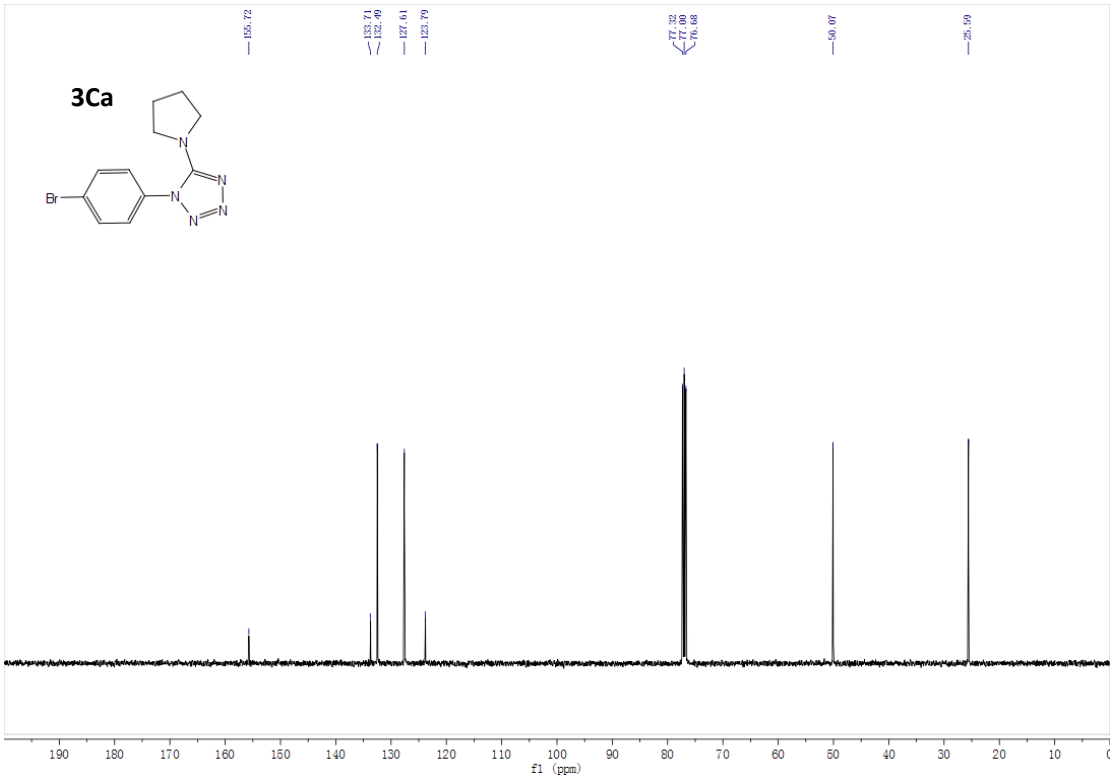

3Aa
7.499
7.491
7.488
7.479
7.474
7.469
7.462
7.457
7.449
7.296
7.286
7.239
7.233
7.218
7.207
7.201
7.196
7.188
3.297
3.290
3.270
3.264
1.929
1.912
1.905
1.895
0.000
2.00
2.00
4.05
4.00
f1 (ppm)

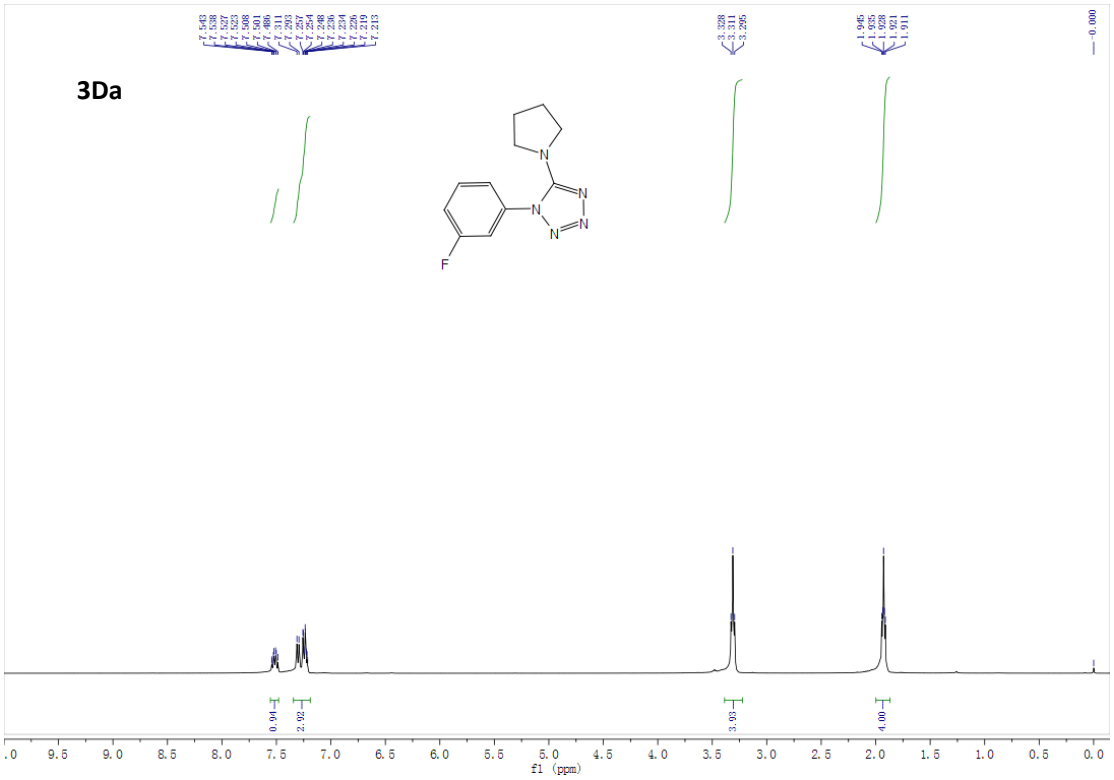

3Aa
164.19
161.69
155.69
130.73
130.70
128.36
128.27
116.44
116.21
77.32
77.00
76.68
49.86
25.56
f1 (ppm)

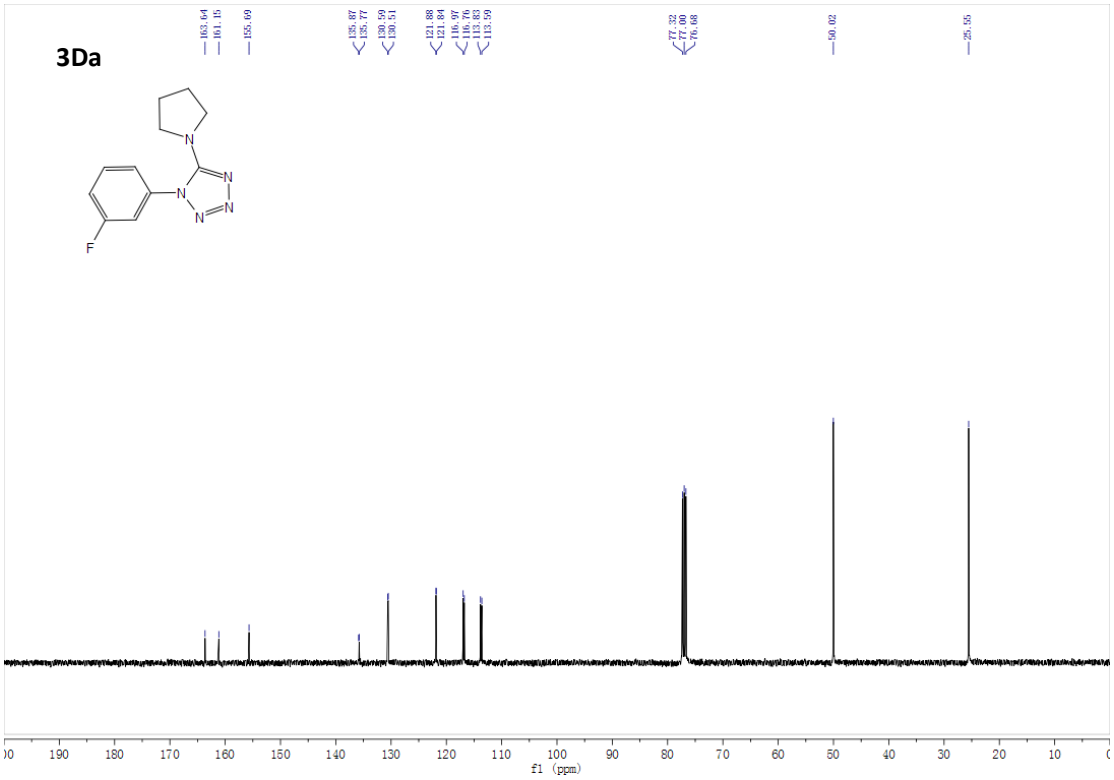

3Ba
7.513
7.508
7.501
7.496
7.440
7.434
7.418
7.411
7.277
3.308
3.291
3.278
3.274
1.933
1.925
1.916
1.910
1.900
0.000
Cl
N
N
N
N
1.99
2.00
4.07
3.96
f1 (ppm)

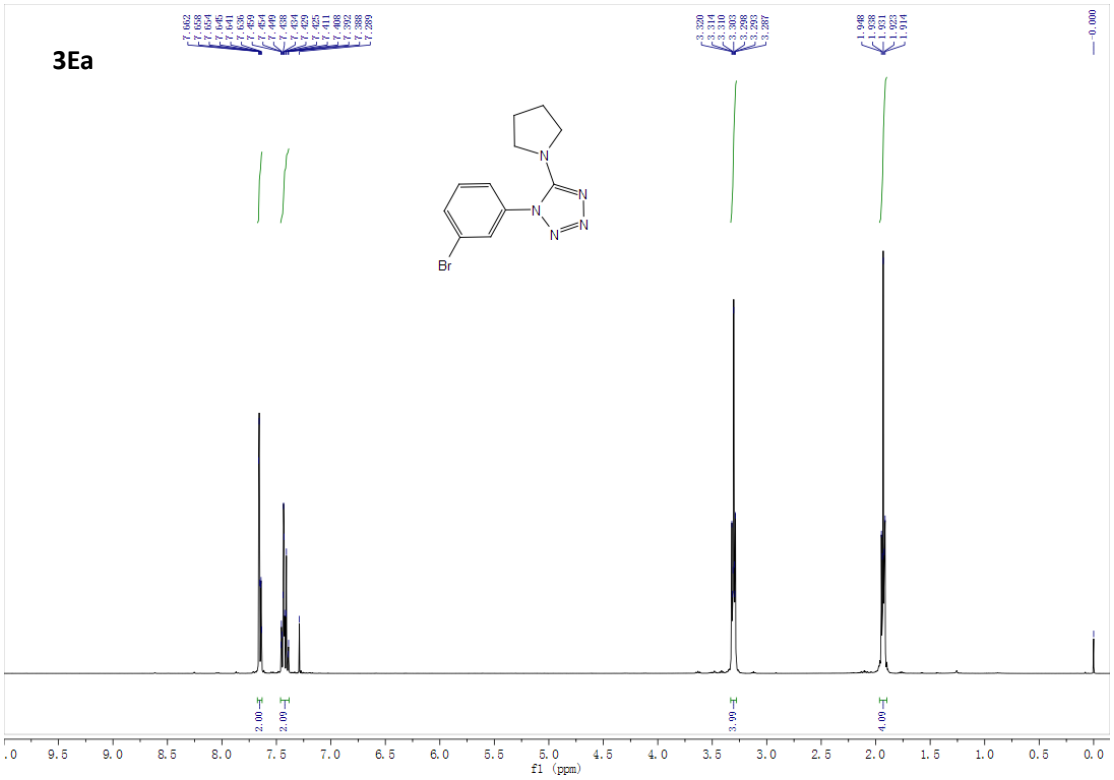

3Ba
155.73
135.81
133.19
129.50
127.40
77.32
77.00
76.68
50.04
25.59
Cl
N
N
N
N
f1 (ppm)

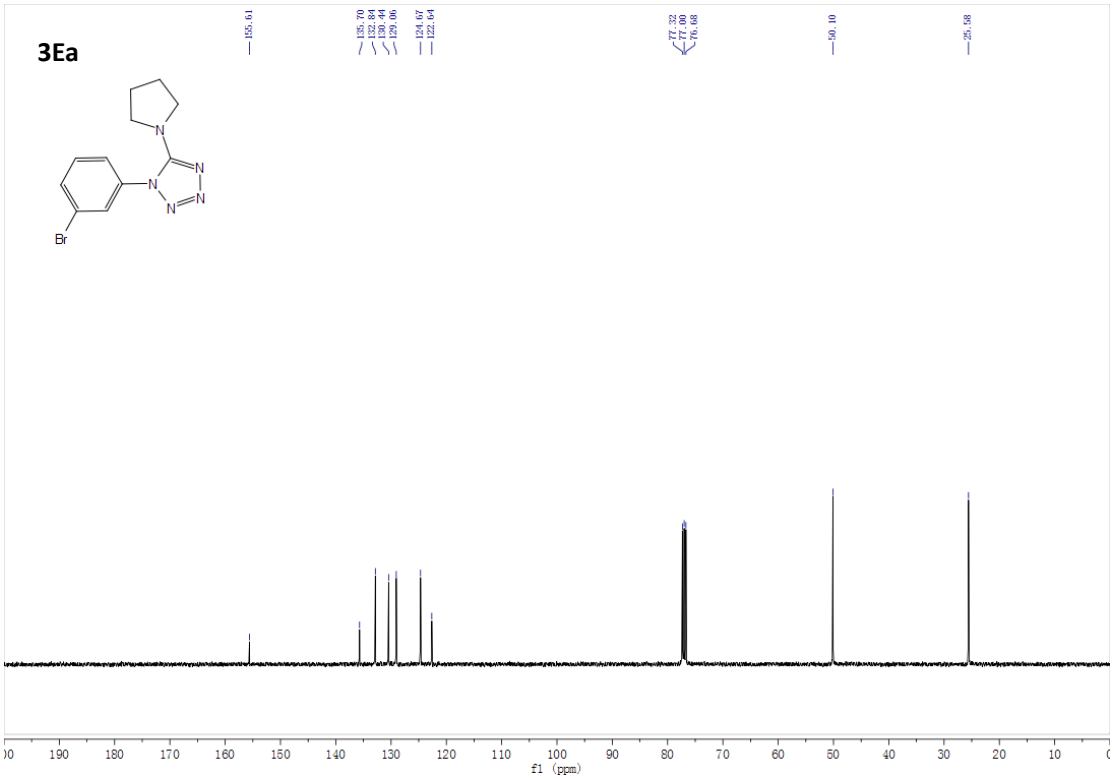

3Ca
7.675
7.668
7.663
7.652
7.647
7.640
7.376
7.370
7.359
7.354
7.347
7.274
3.308
3.301
3.297
3.291
3.286
3.281
3.275
1.934
1.925
1.917
1.909
1.900
0.000
Br
N
N
N
N
2.01
2.00
4.00
3.97
f1 (ppm)

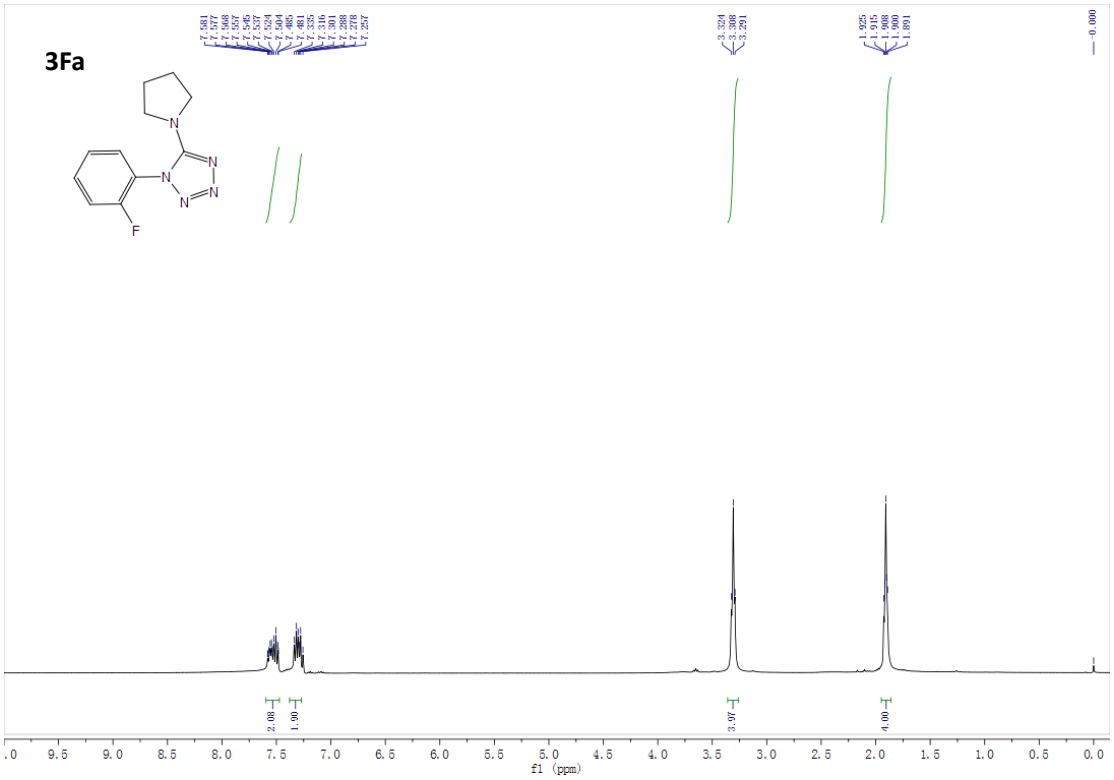

155.72
133.71
132.40
127.61
123.79
77.32
77.00
76.68
50.07
25.59
3Ca
Br
N
N
N
N
f1 (ppm)

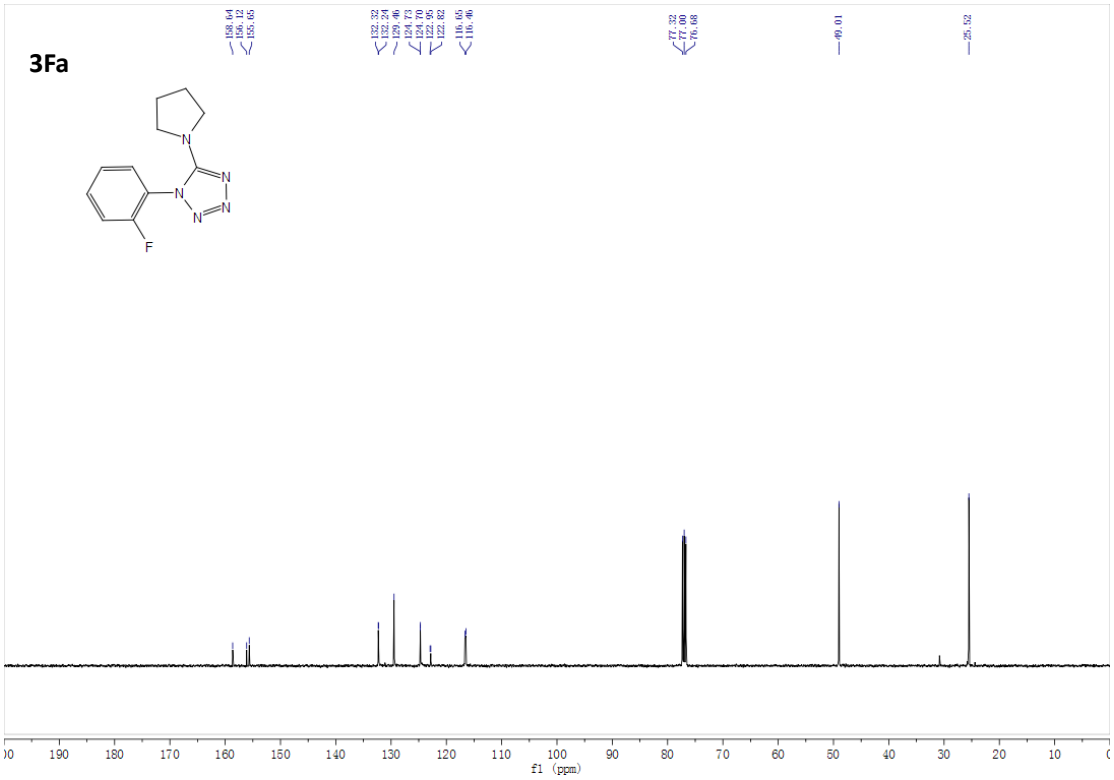

3Da
7.543
7.538
7.527
7.523
7.508
7.501
7.486
7.311
7.293
7.257
7.254
7.248
7.236
7.234
7.226
7.219
7.213
3.328
3.311
3.295
1.945
1.935
1.928
1.921
1.911
0.000
0.94
2.92
3.93
4.00
f1 (ppm)

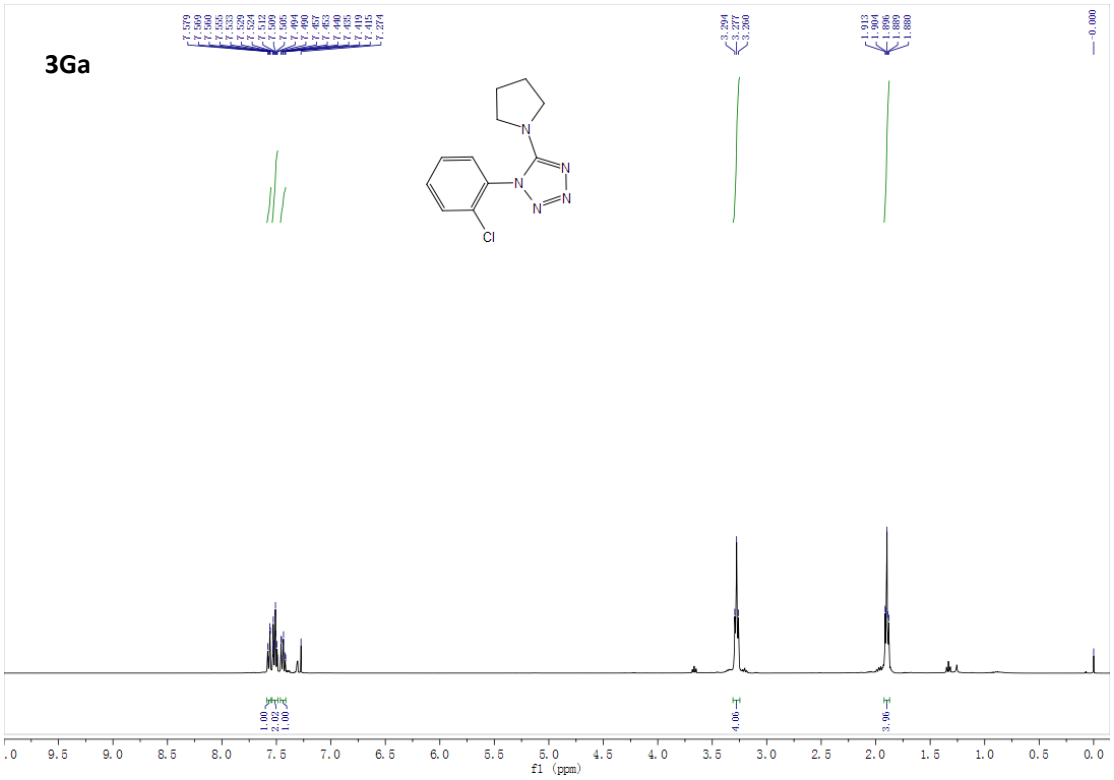

3Da
163.64
161.15
155.69
135.87
135.77
130.59
130.51
121.88
121.84
116.97
116.76
113.85
113.59
77.32
77.00
76.68
50.02
25.55
f1 (ppm)

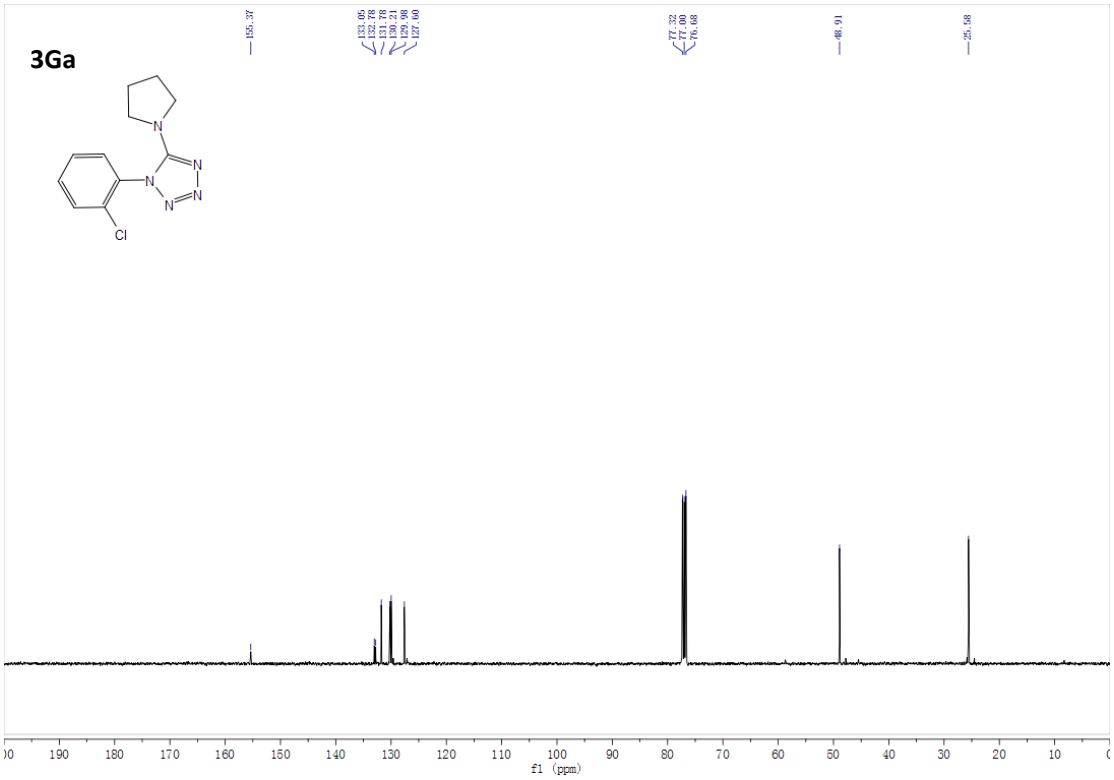

3Ea
7.662
7.658
7.654
7.645
7.641
7.635
7.459
7.454
7.449
7.438
7.434
7.429
7.425
7.411
7.406
7.392
7.388
7.289
3.320
3.314
3.310
3.303
3.298
3.293
3.287
1.948
1.938
1.931
1.923
1.914
0.000
Br
2.00
2.09
3.99
4.09
f1 (ppm)

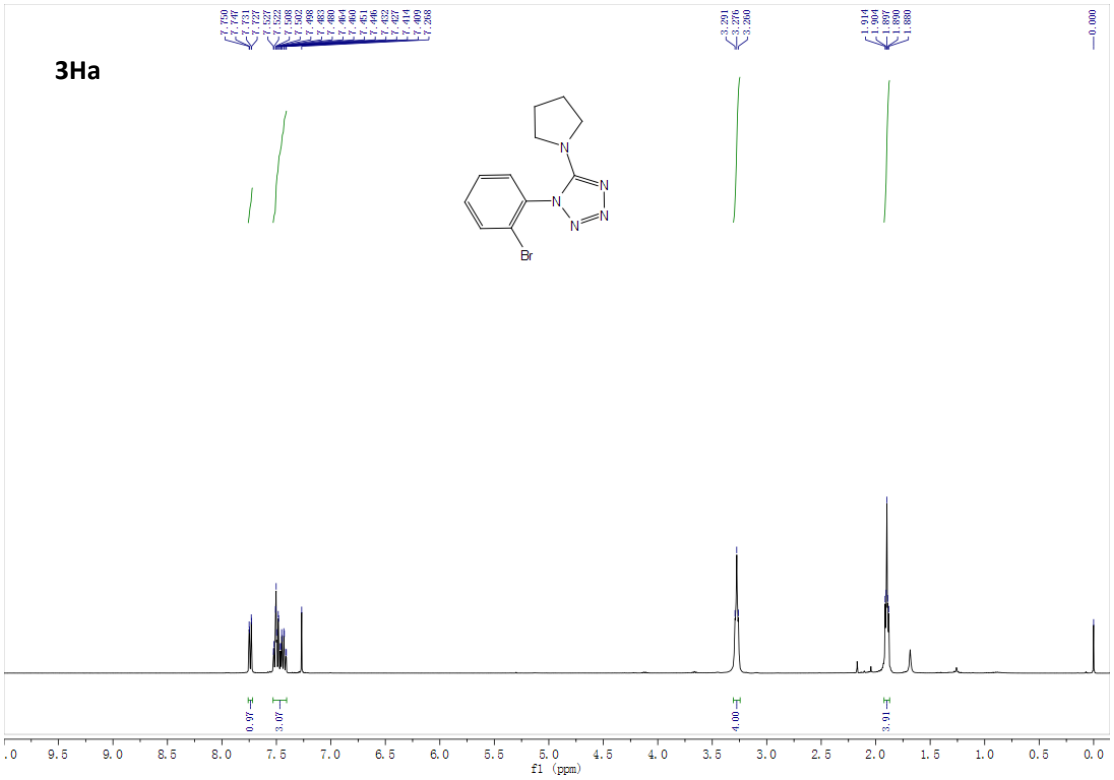

3Ea
155.61
135.70
132.84
130.44
129.06
124.67
122.64
77.32
77.00
76.68
50.10
25.58
Br
f1 (ppm)

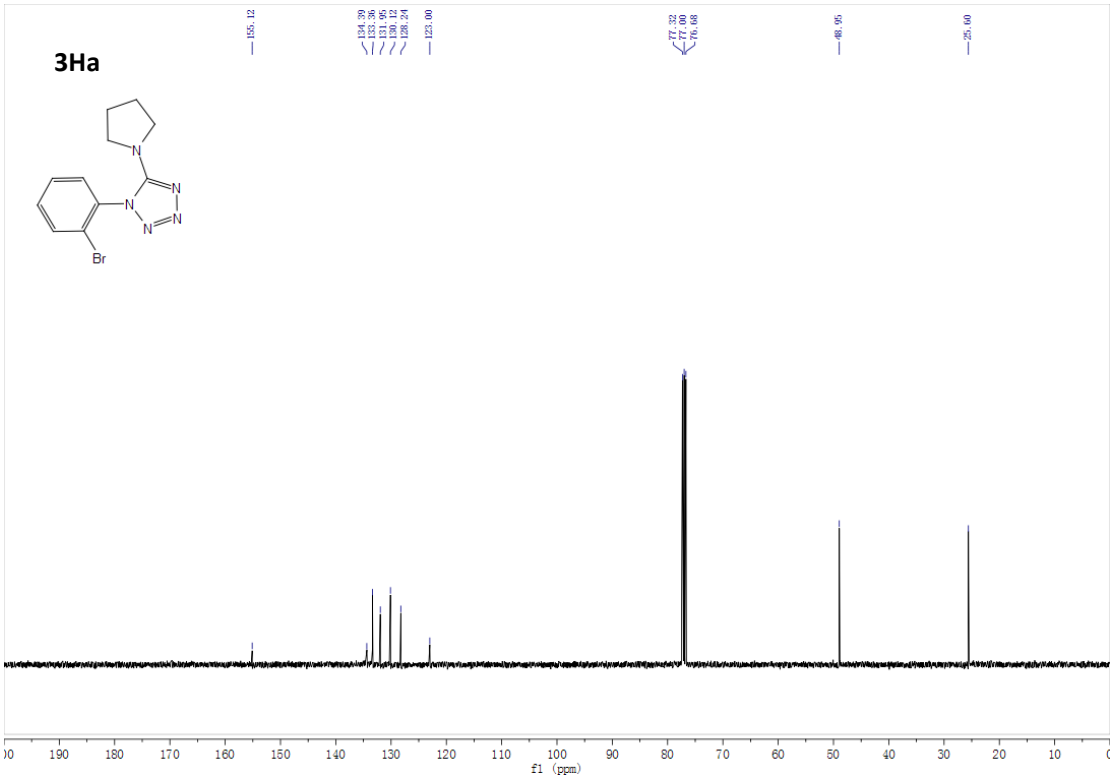

3Fa
7.591
7.577
7.568
7.557
7.545
7.537
7.504
7.494
7.485
7.481
7.335
7.316
7.301
7.288
7.278
7.257
3.324
3.308
3.291
1.925
1.915
1.908
1.900
1.891
0.000
2.08
1.90
3.97
4.00
f1 (ppm)

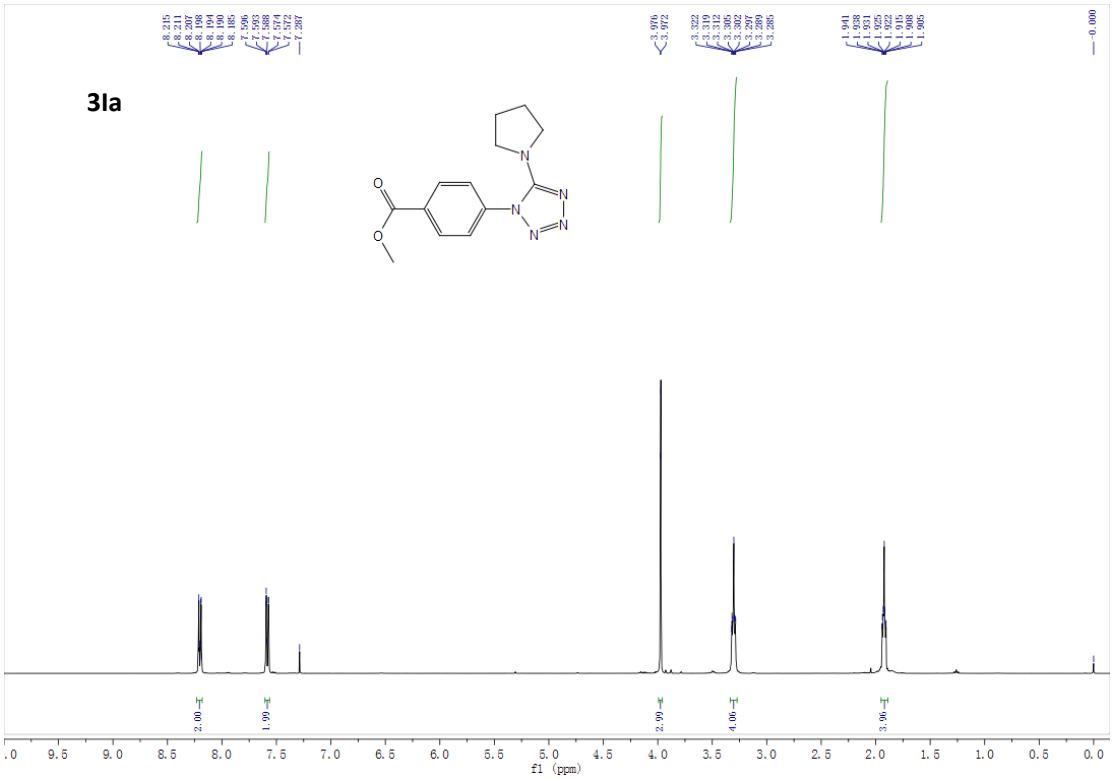

3Fa
158.04
156.12
155.65
132.32
132.24
129.46
124.73
124.70
122.95
122.82
116.65
116.46
77.32
77.00
76.68
49.01
25.52
f1 (ppm)

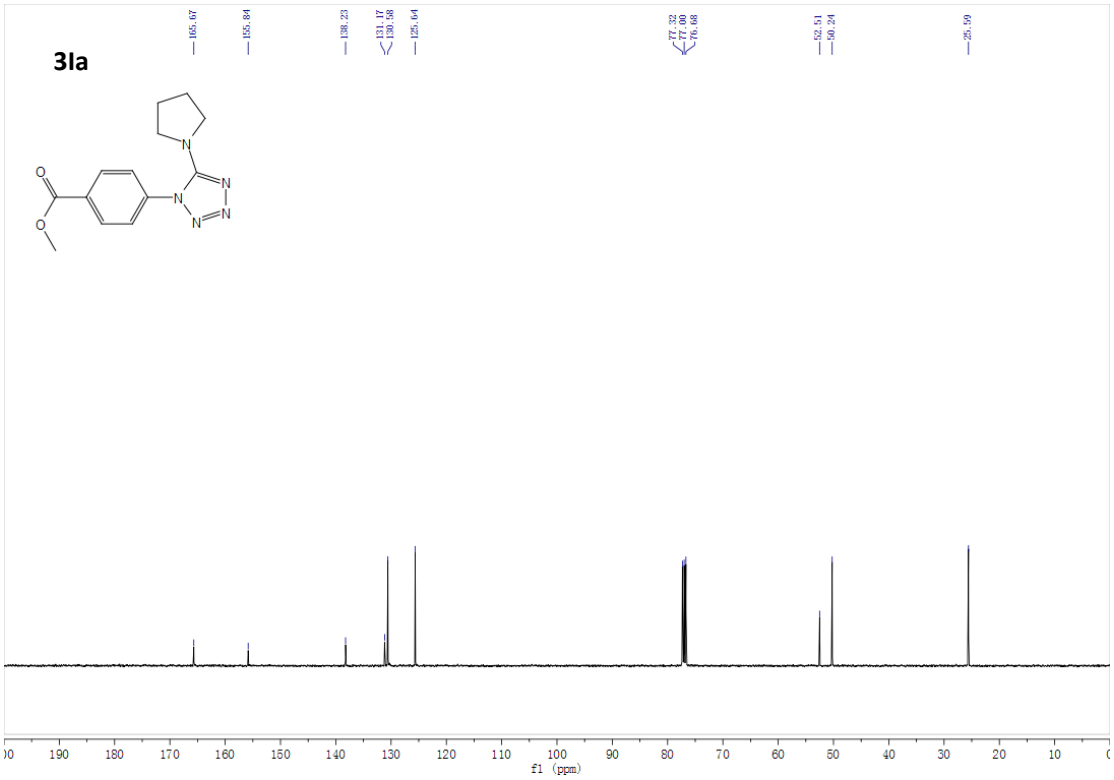

3Ga
7.579
7.566
7.560
7.555
7.533
7.529
7.524
7.512
7.509
7.495
7.494
7.490
7.467
7.463
7.460
7.435
7.419
7.415
7.274
3.294
3.277
3.260
1.913
1.904
1.896
1.888
1.880
0.000
N
N
N
N
Cl
1.00
2.02
1.00
4.06
3.90
f1 (ppm)

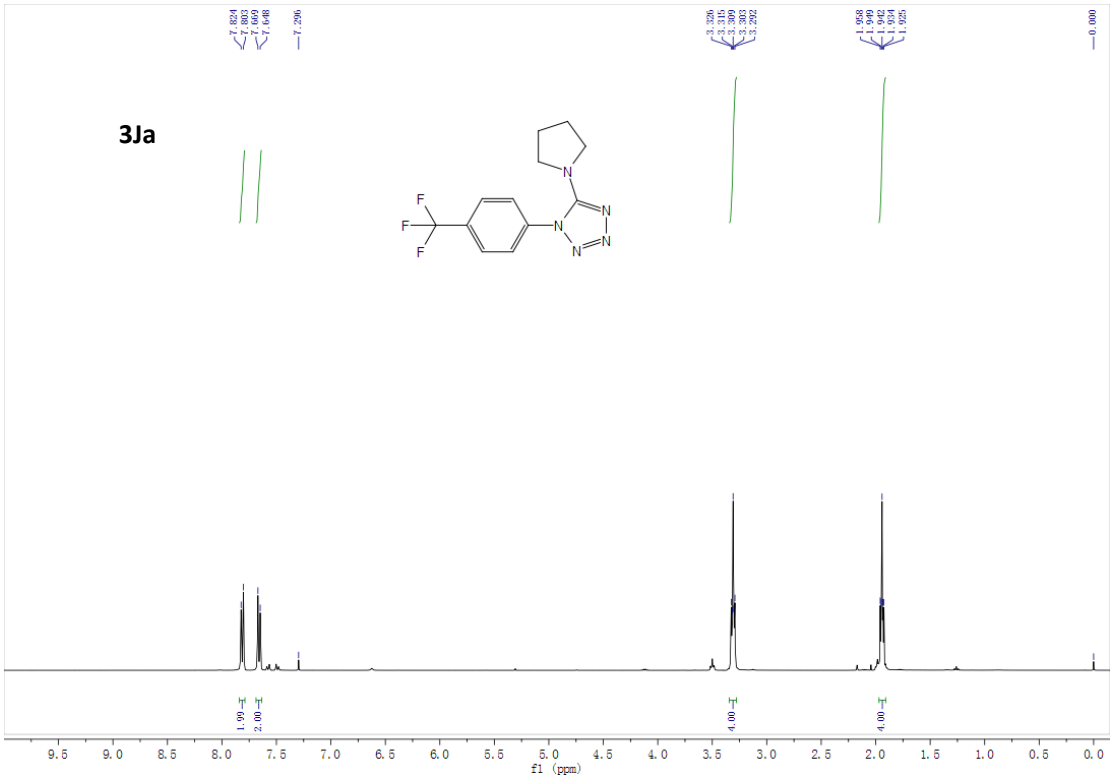

3Ga
155.37
133.05
132.78
131.78
130.71
129.98
127.60
77.32
77.00
76.68
48.91
25.58
N
N
N
N
Cl
f1 (ppm)

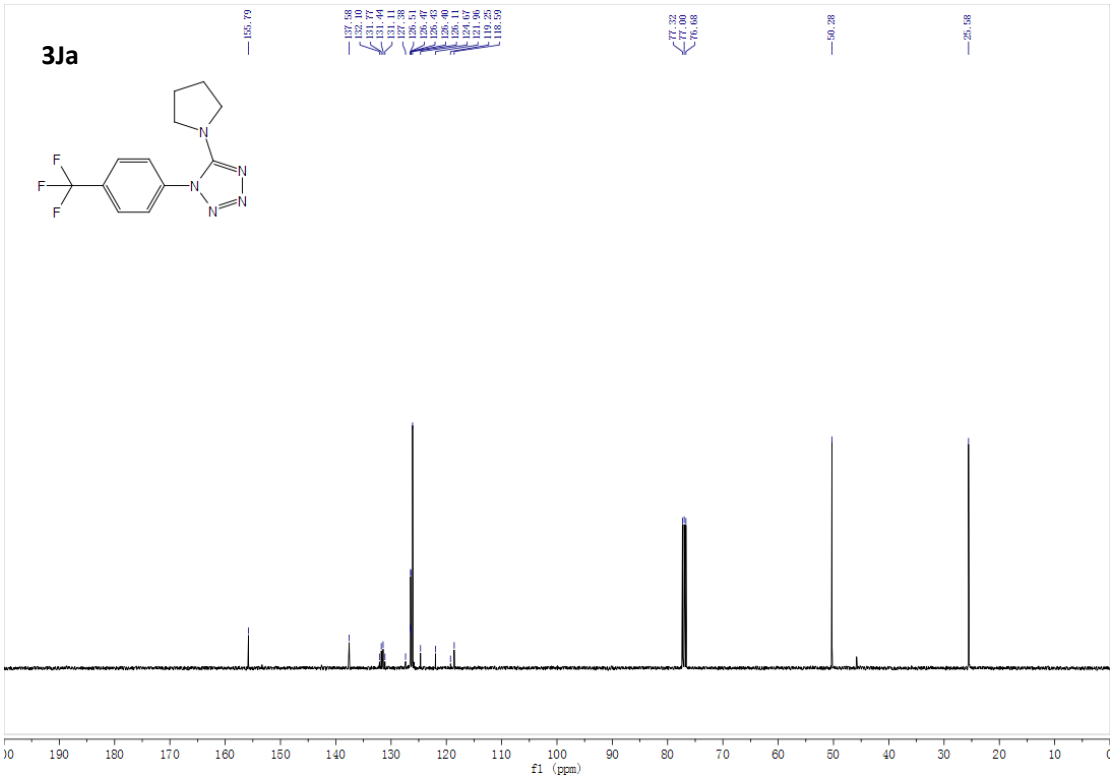

3Ha
7.750
7.747
7.731
7.727
7.522
7.508
7.502
7.493
7.483
7.480
7.464
7.460
7.451
7.446
7.432
7.427
7.414
7.409
7.268
3.291
3.276
3.260
1.914
1.904
1.897
1.890
1.880
0.000
Br
0.97
3.07
4.00
3.91
f1 (ppm)


3Ha
155.12
134.39
133.36
131.95
130.12
128.24
123.00
77.32
77.00
76.68
48.95
25.60
Br
f1 (ppm)


3la
8.215
8.211
8.207
8.198
8.194
8.191
8.185
7.596
7.593
7.588
7.574
7.572
7.287
3.976
3.972
3.322
3.319
3.312
3.305
3.302
3.297
3.289
3.285
1.941
1.938
1.931
1.925
1.922
1.915
1.908
1.905
0.000
2.00
1.99
2.99
4.06
3.96
f1 (ppm)


3la
165.67
155.84
138.23
131.17
130.58
125.64
77.32
77.00
76.68
52.51
50.24
25.59
f1 (ppm)


3Ja
7.824
7.803
7.669
7.649
7.648
7.296
3.326
3.315
3.309
3.303
3.292
1.958
1.949
1.942
1.934
1.925
0.000
1.99
2.00
4.00
4.00
f1 (ppm)


3Ja
155.79
137.58
132.10
131.77
131.44
131.11
127.38
126.51
126.47
126.45
126.40
126.11
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118.59
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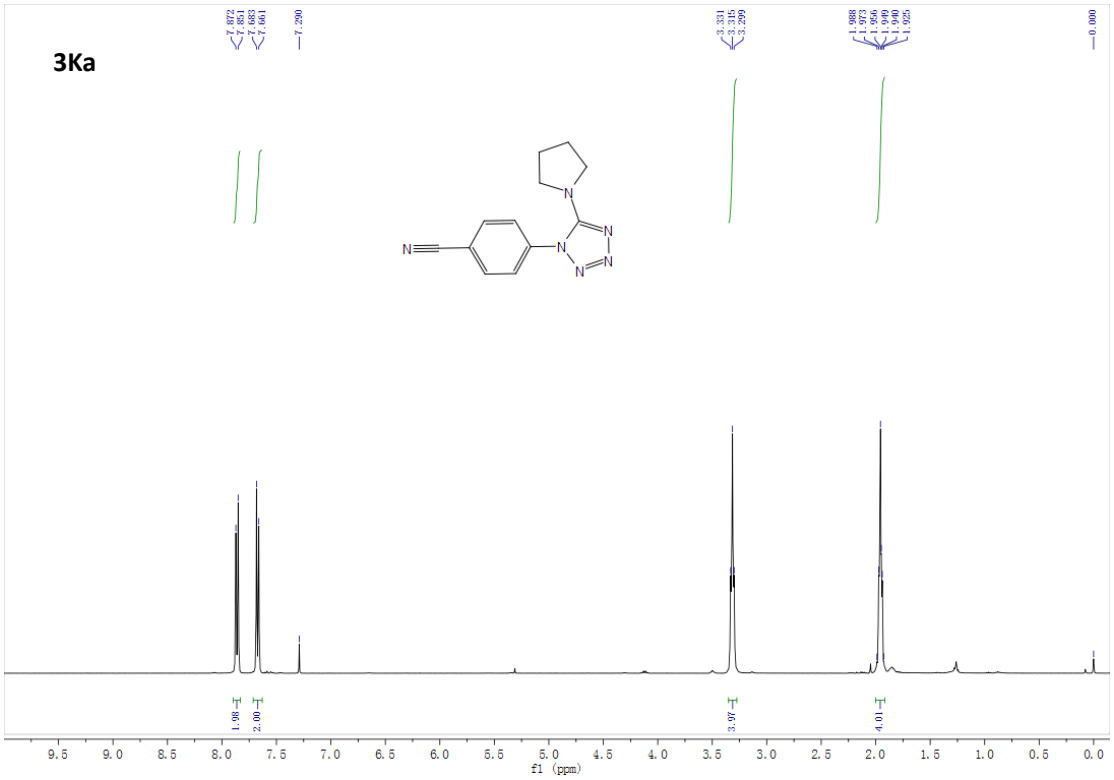
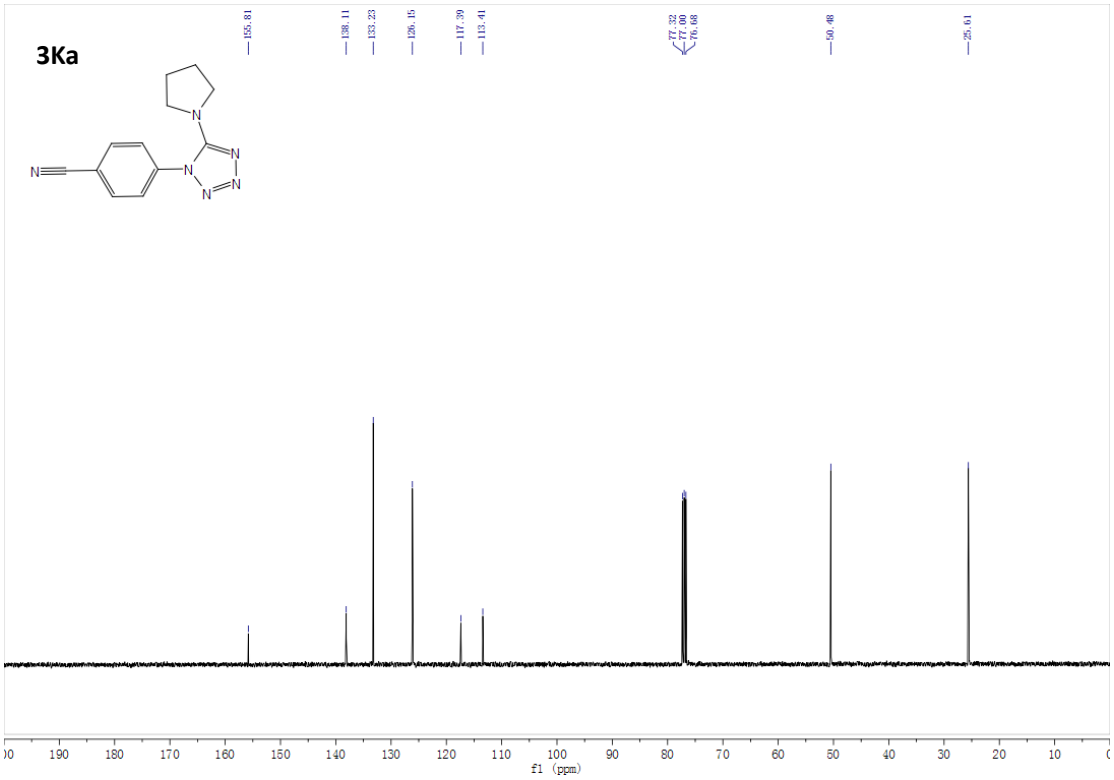
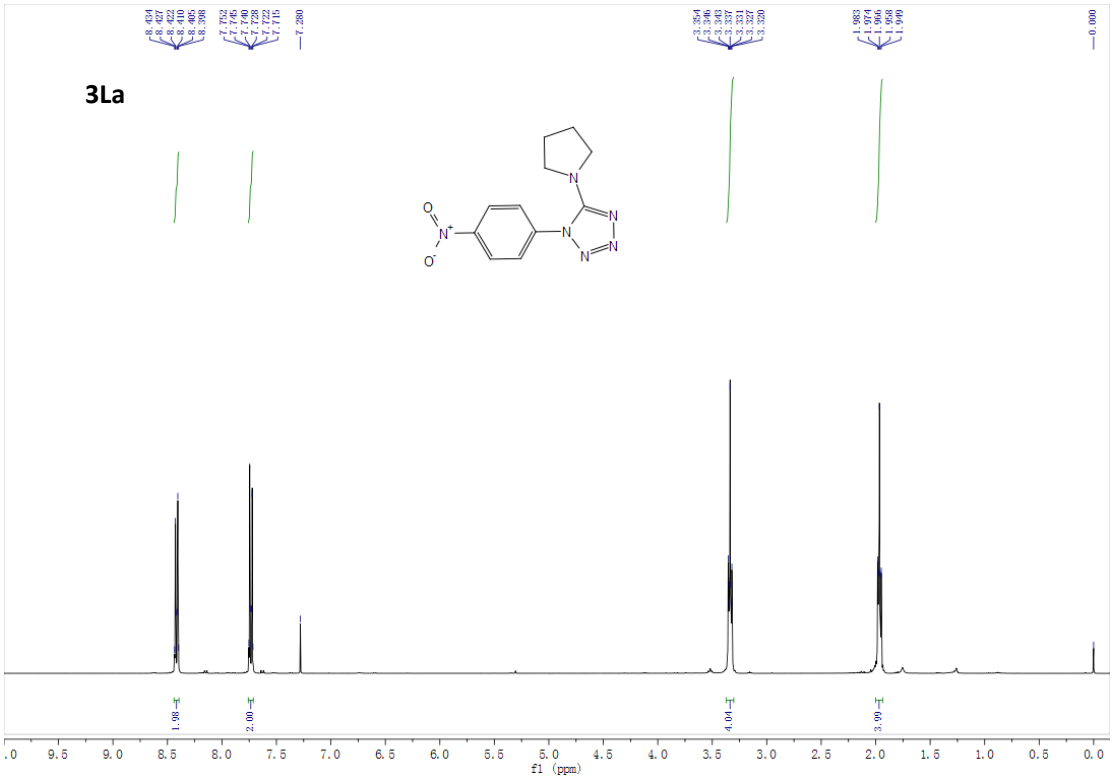
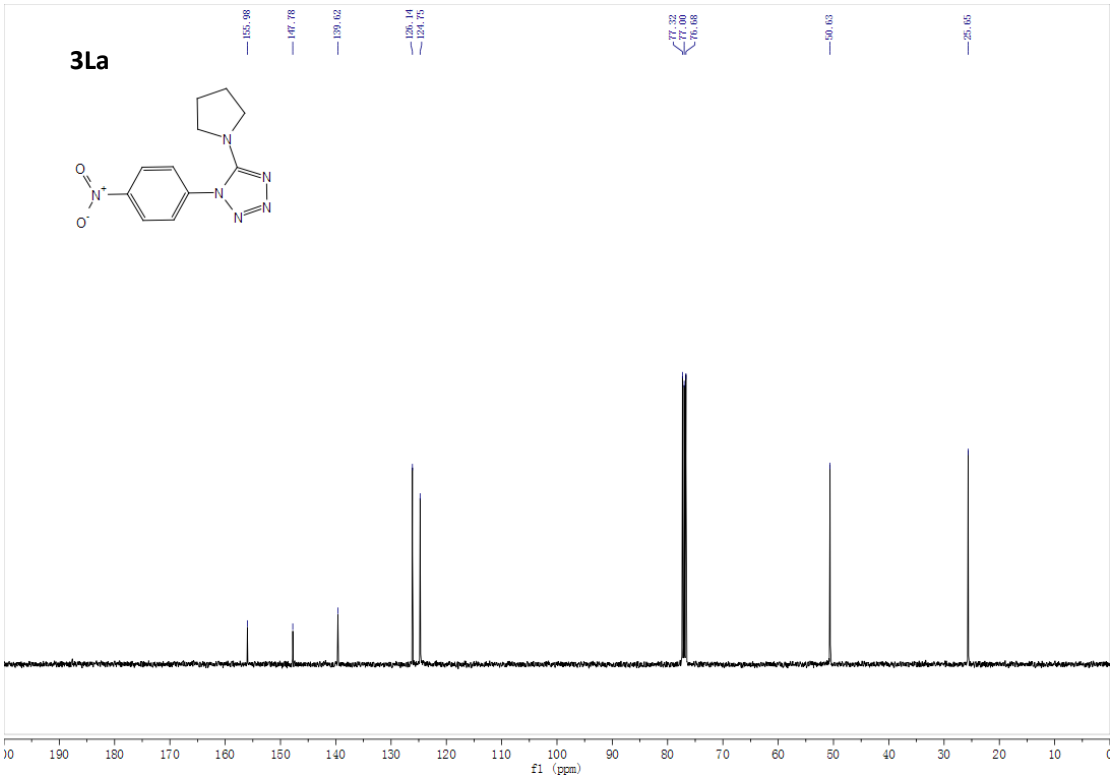
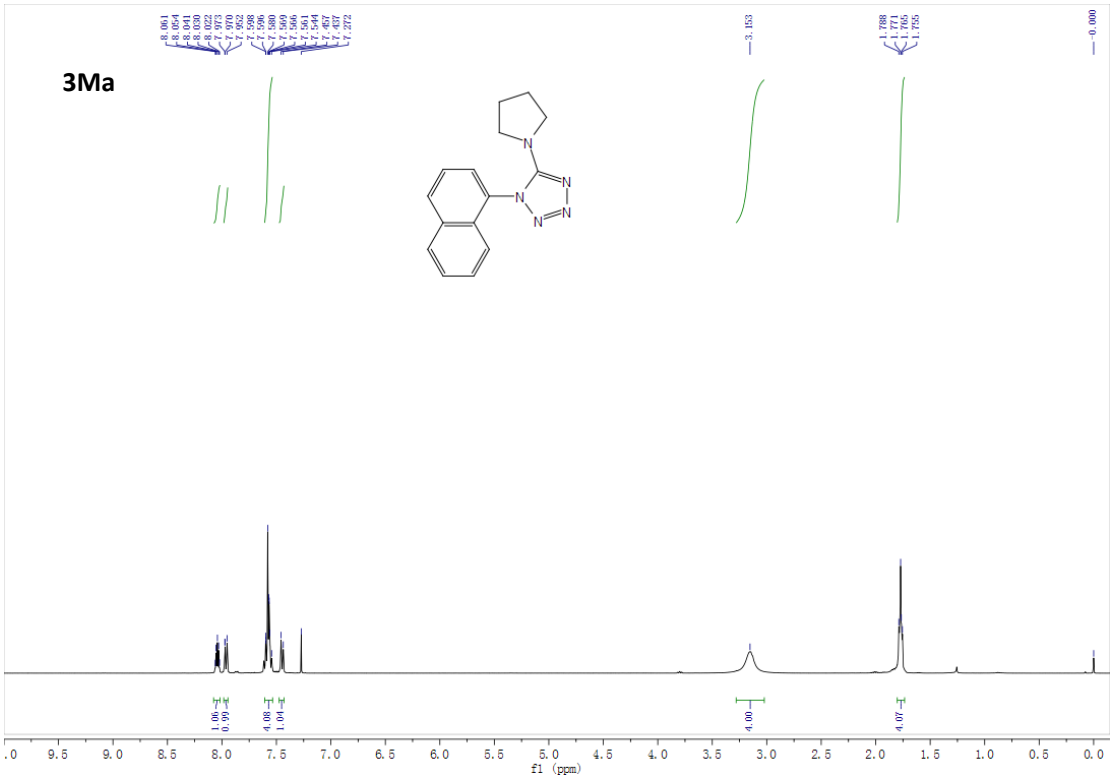
77.00
76.68
50.28
25.58
f1 (ppm)

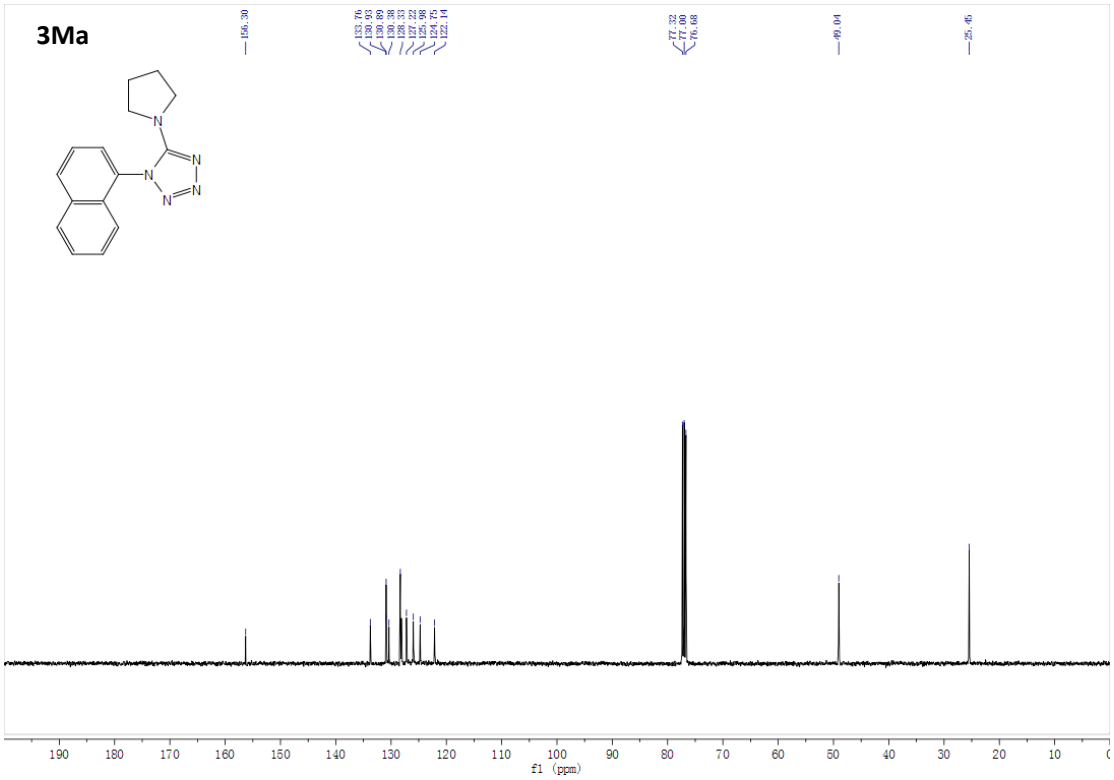

3Ka
7.872
7.851
7.683
7.661
7.290
3.331
3.315
3.299
1.988
1.973
1.956
1.940
1.908
1.925
0.000
1.98
2.00
3.97
4.01
f1 (ppm)

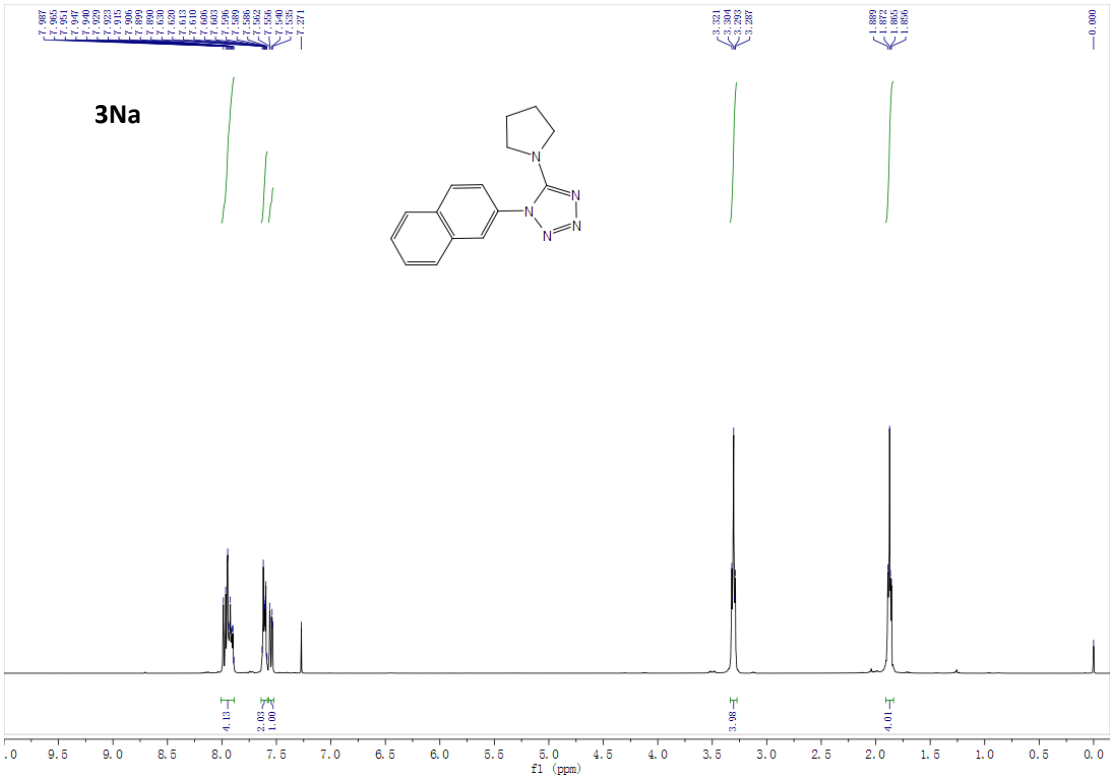

3Ka
155.81
138.11
133.23
126.15
117.39
113.41
77.32
77.00
76.68
50.48
25.61
f1 (ppm)

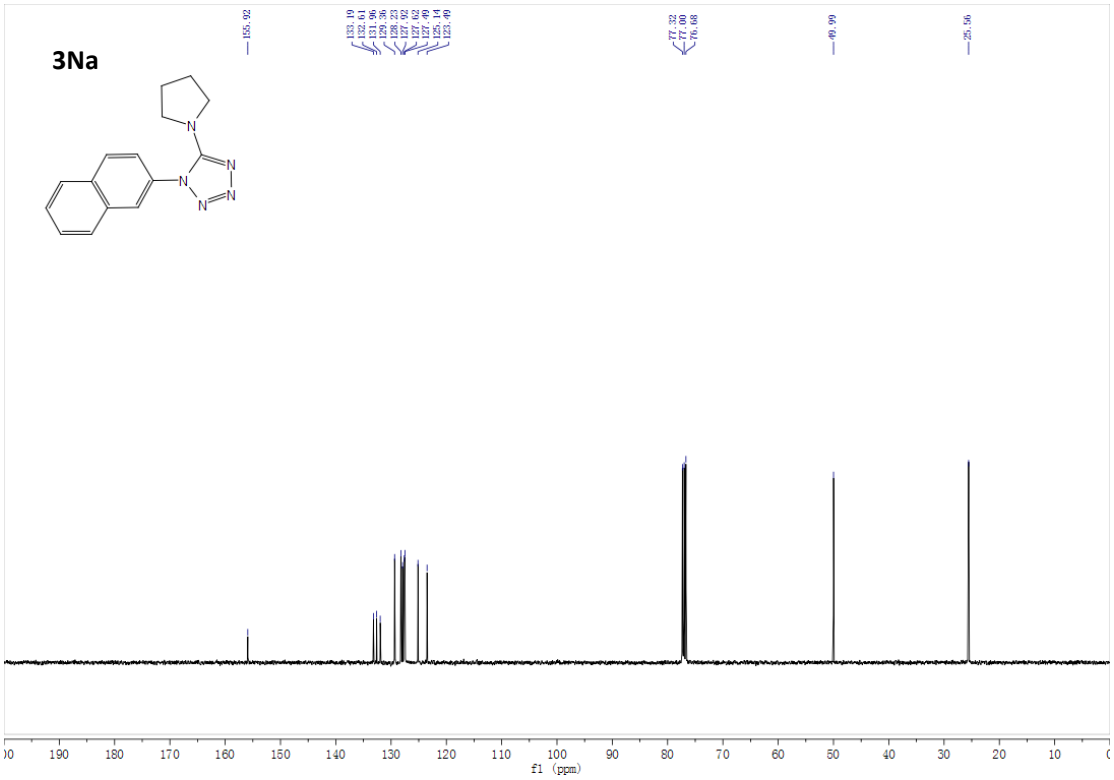

3La
8.434
8.427
8.422
8.410
8.405
8.398
7.752
7.745
7.740
7.728
7.722
7.715
7.280
3.354
3.346
3.343
3.337
3.331
3.327
3.320
1.983
1.974
1.966
1.958
1.949
0.000
1.98
2.00
4.04
3.99
f1 (ppm)

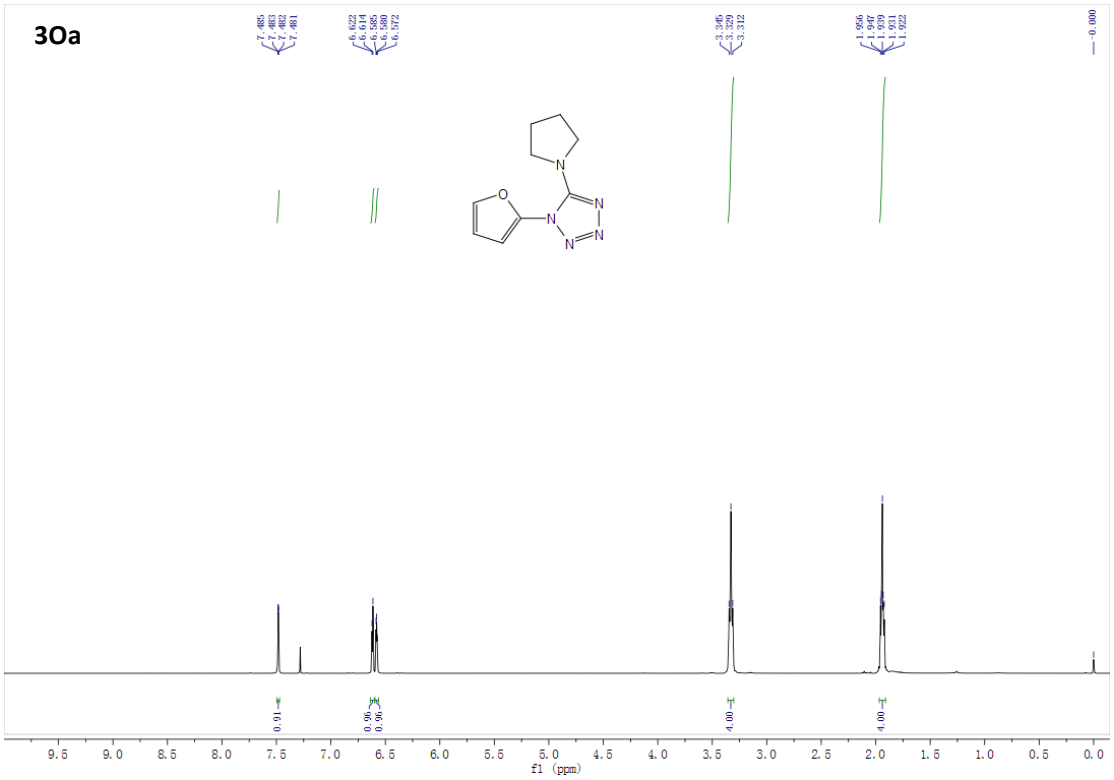

3La
155.98
147.78
139.62
126.14
124.75
77.32
77.00
76.68
50.63
25.65
f1 (ppm)

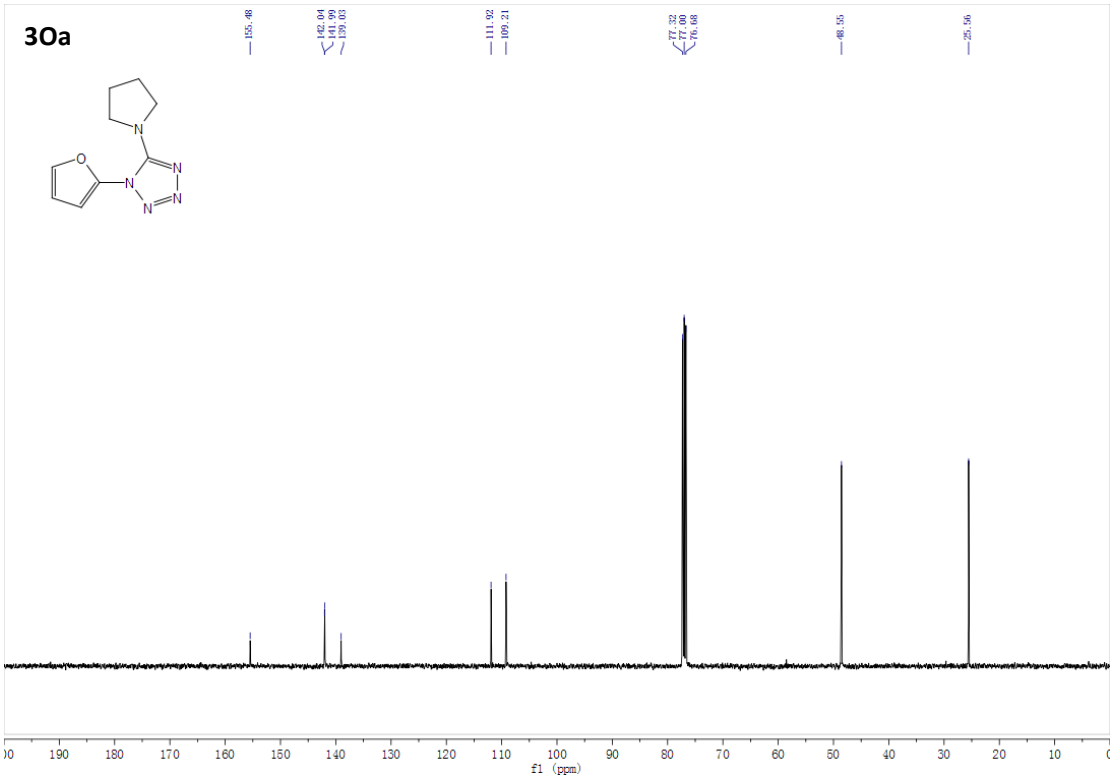

3Ma
8.061
8.054
8.041
8.030
8.022
7.973
7.970
7.952
7.596
7.580
7.569
7.564
7.561
7.554
7.457
7.437
7.272
3.153
1.788
1.771
1.765
1.755
0.000
1.06
0.99
4.08
1.04
4.00
4.07
f1 (ppm)

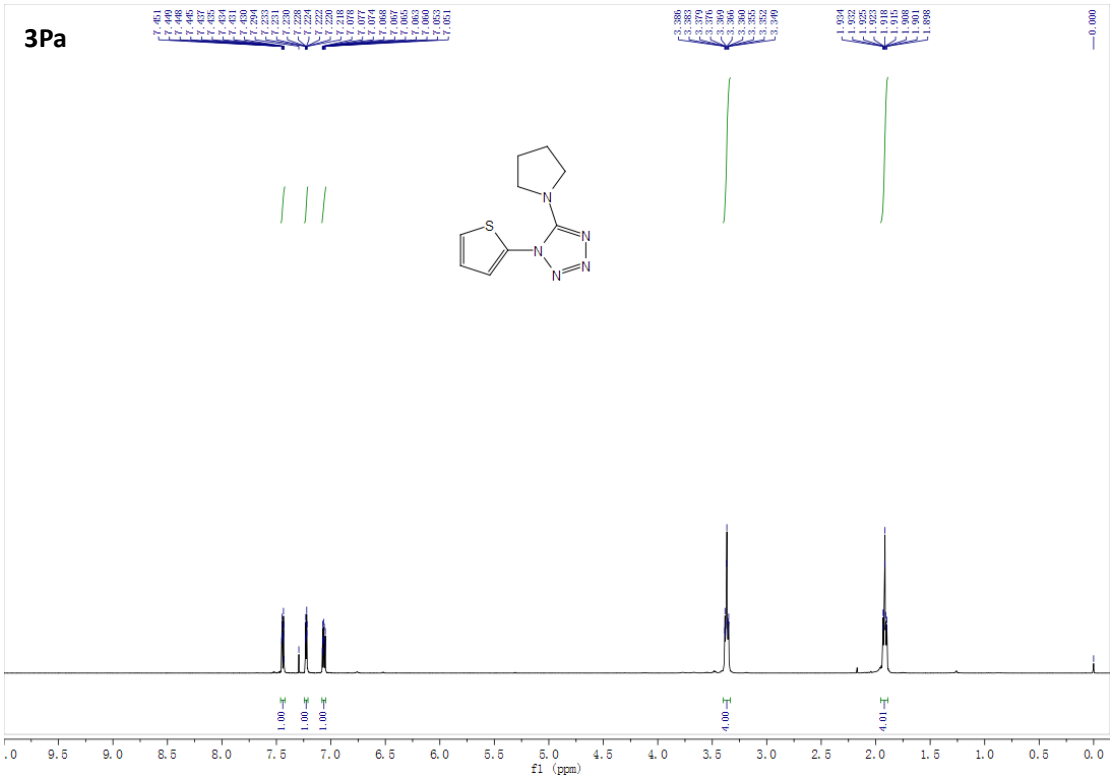

3Ma
156.30
133.76
130.93
130.89
130.38
128.33
127.22
125.96
124.75
122.14
77.32
77.00
76.68
49.04
25.45
f1 (ppm)

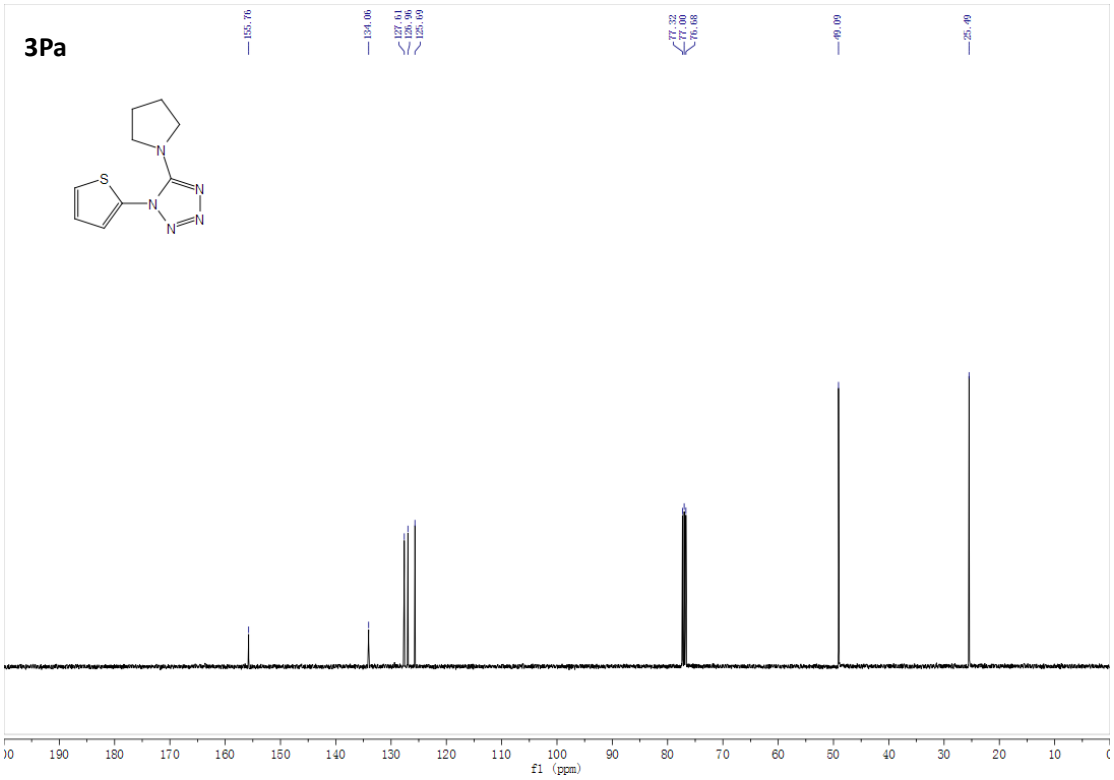

3Na
f1 (ppm)

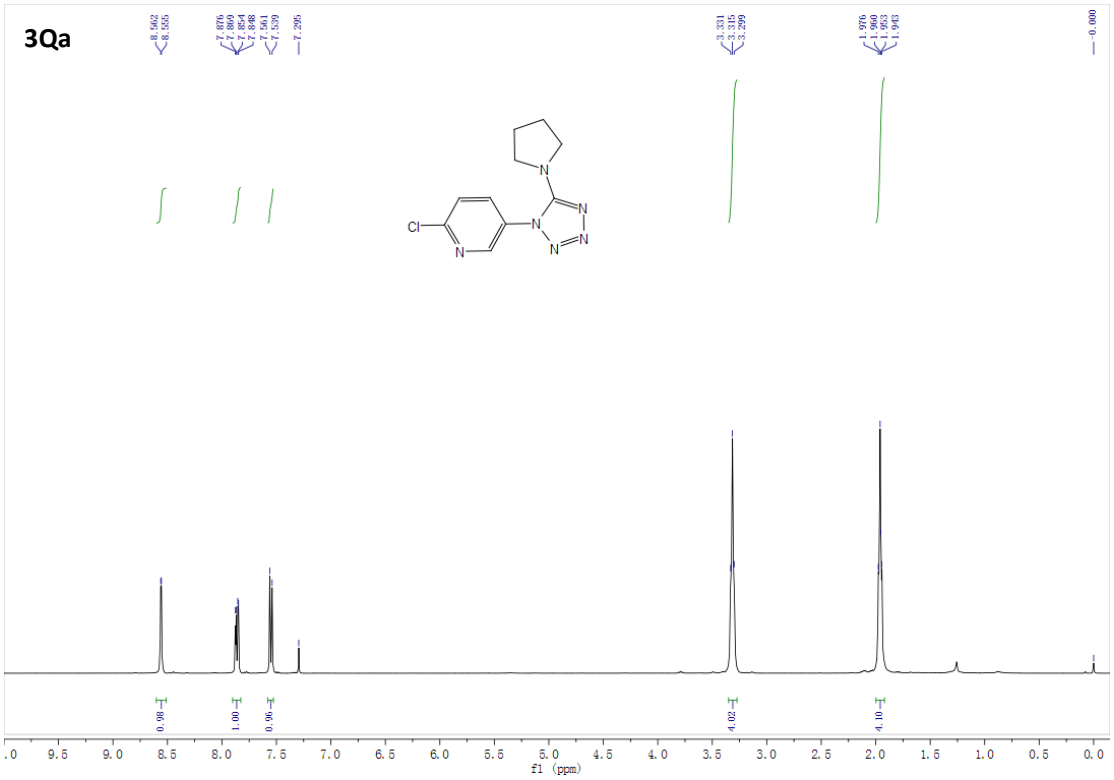

3Na
f1 (ppm)

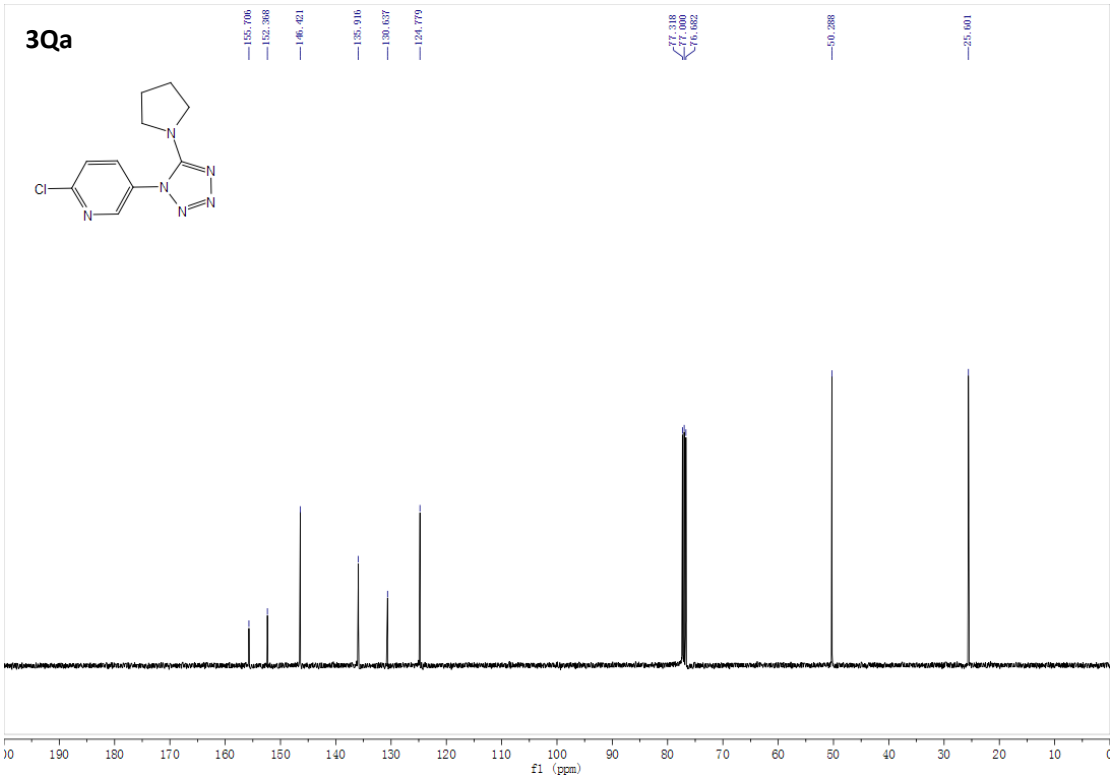

3Oa
7.485
7.483
7.482
7.481
6.622
6.614
6.585
6.580
6.572
3.395
3.369
3.312
1.956
1.947
1.939
1.931
1.922
0.000
0.91
0.96
0.96
4.00
4.00
f1 (ppm)


3Oa
155.48
142.04
141.99
139.03
111.92
109.21
77.32
77.00
76.68
68.55
25.56
f1 (ppm)

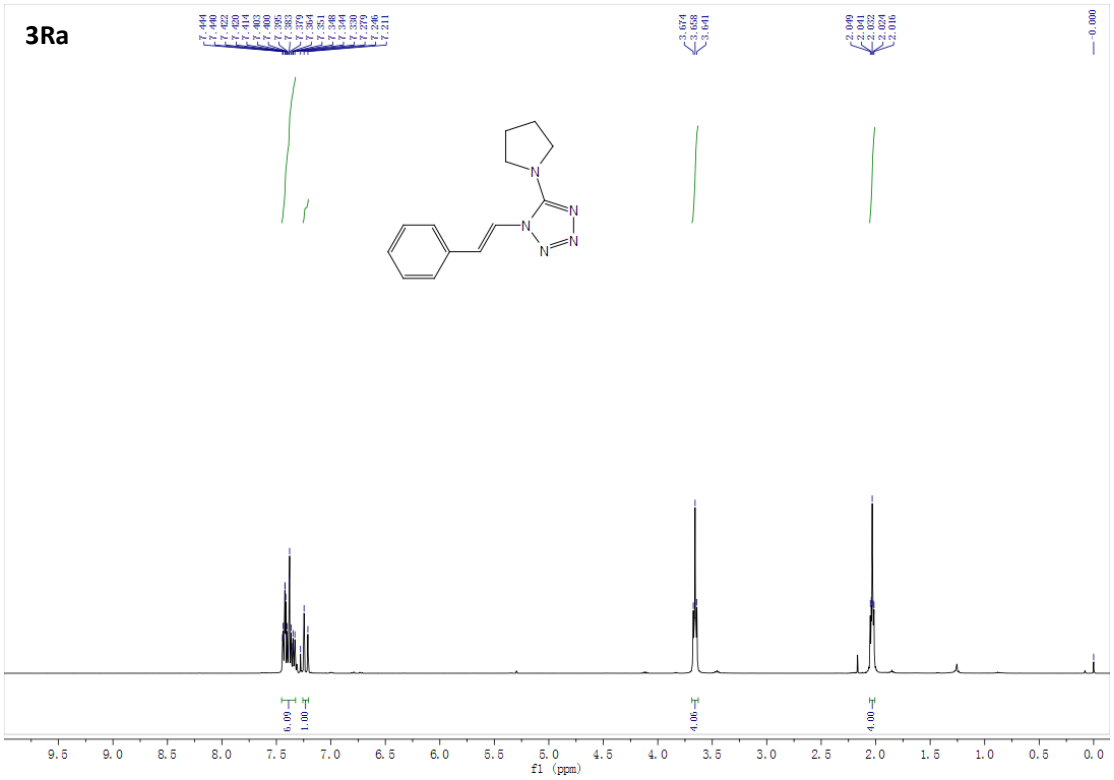

3Pa
7.491
7.490
7.448
7.445
7.437
7.435
7.431
7.430
7.294
7.233
7.231
7.230
7.228
7.224
7.222
7.220
7.218
7.078
7.077
7.074
7.068
7.067
7.065
7.063
7.060
7.053
7.051
3.386
3.383
3.379
3.376
3.369
3.366
3.359
3.355
3.352
3.349
1.934
1.932
1.925
1.923
1.918
1.911
1.908
1.901
1.898
0.000
1.00
1.00
1.00
4.00
4.01
f1 (ppm)


3Pa
155.76
134.06
127.61
126.96
125.69
77.32
77.00
76.68
49.09
25.49
f1 (ppm)


3Qa
8.562
8.555
7.876
7.869
7.854
7.848
7.561
7.539
7.295
3.331
3.315
3.299
1.976
1.960
1.955
1.943
0.000
Cl
N
N
N
N
0.98
1.00
0.96
4.02
4.10
f1 (ppm)


3Qa
155.706
152.368
146.421
135.916
130.637
124.779
77.318
77.000
76.682
50.288
25.601
Cl
N
N
N
N
f1 (ppm)

3Ra


7.444
7.440
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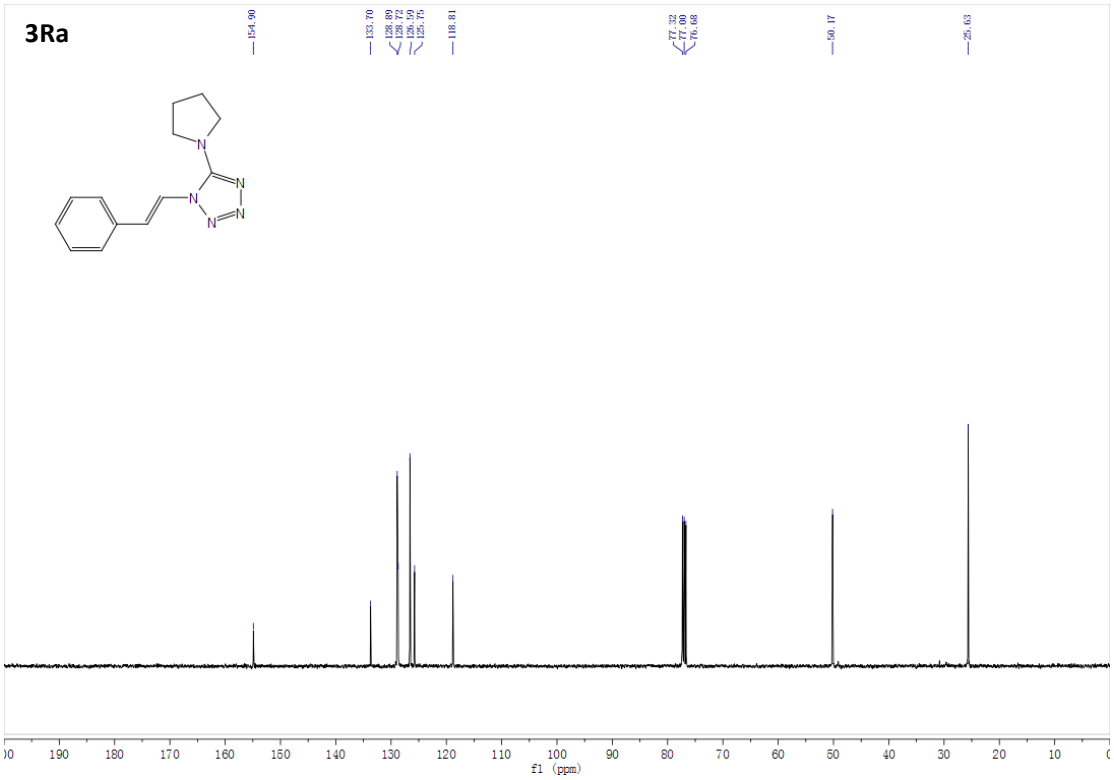
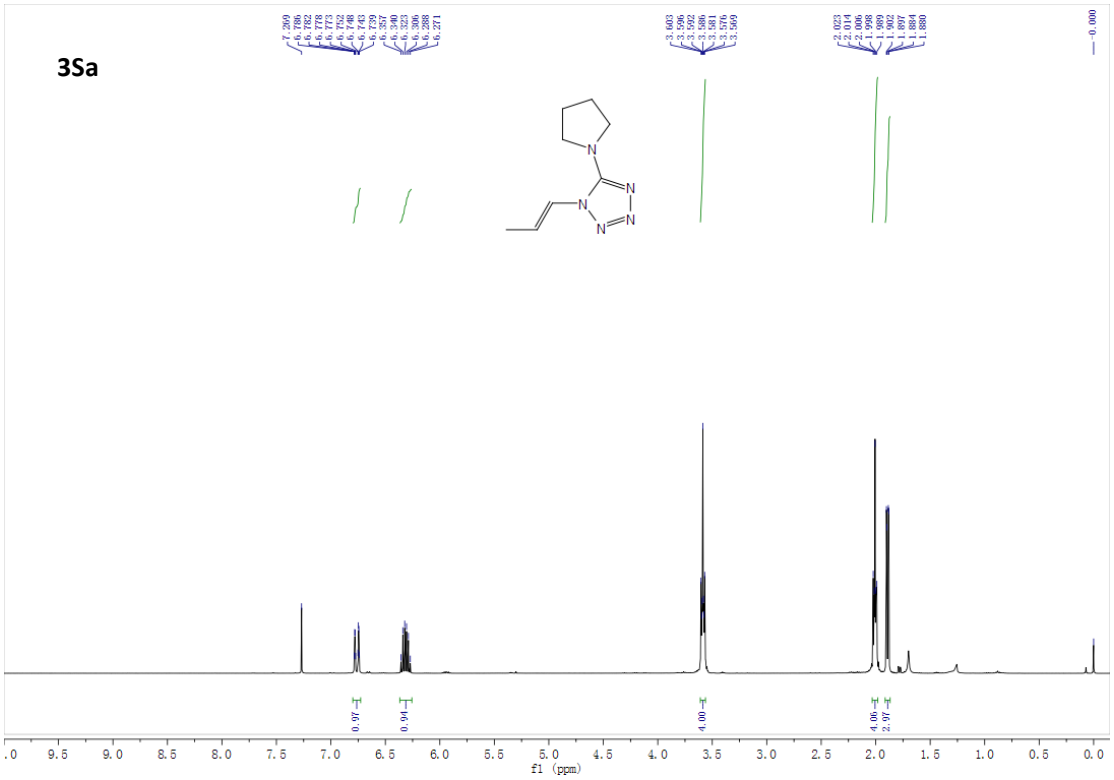
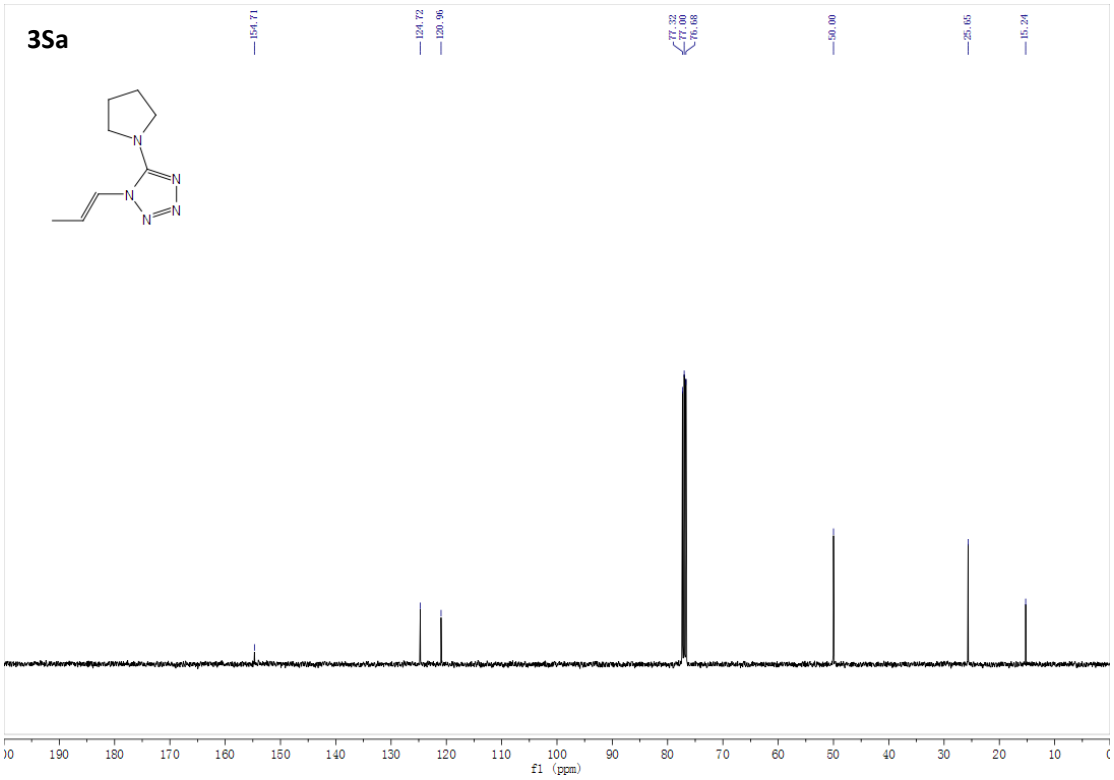
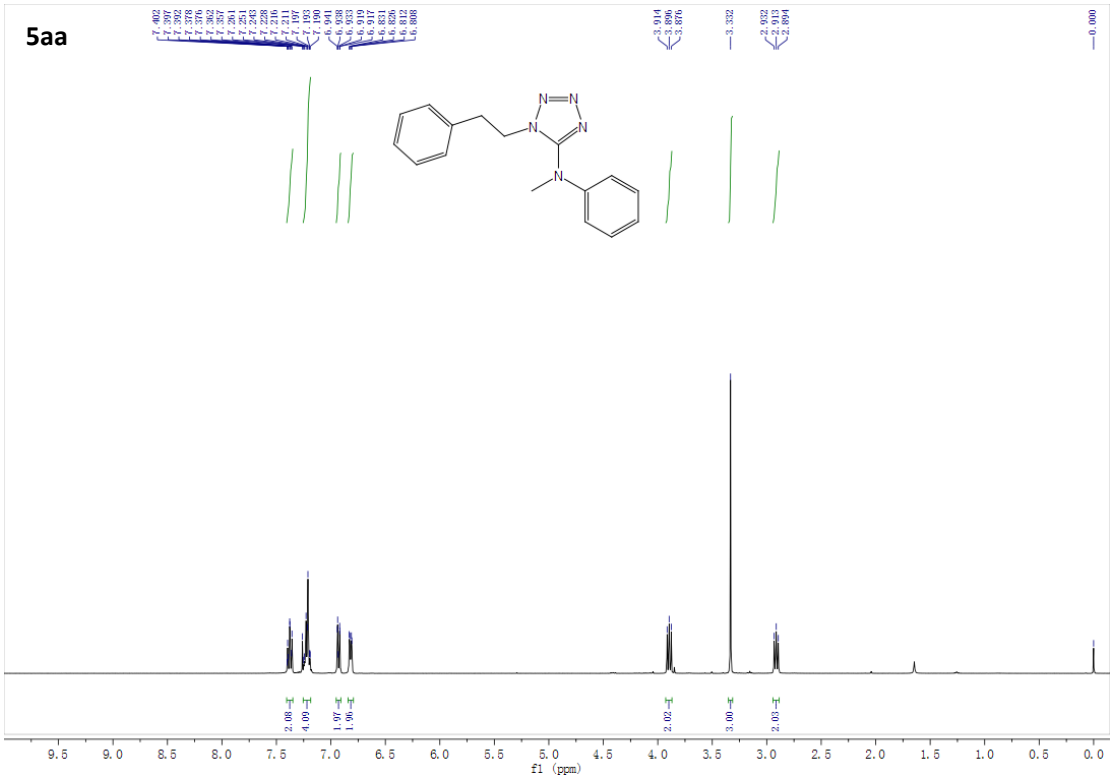
7.422
7.420
7.414
7.403
7.400
7.395
7.383
7.379
7.364
7.351
7.348
7.344
7.339
7.279
7.246
7.211
3.674
3.658
3.641
2.099
2.041
2.032
2.024
2.016
0.000
6.09
1.00
4.06
4.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

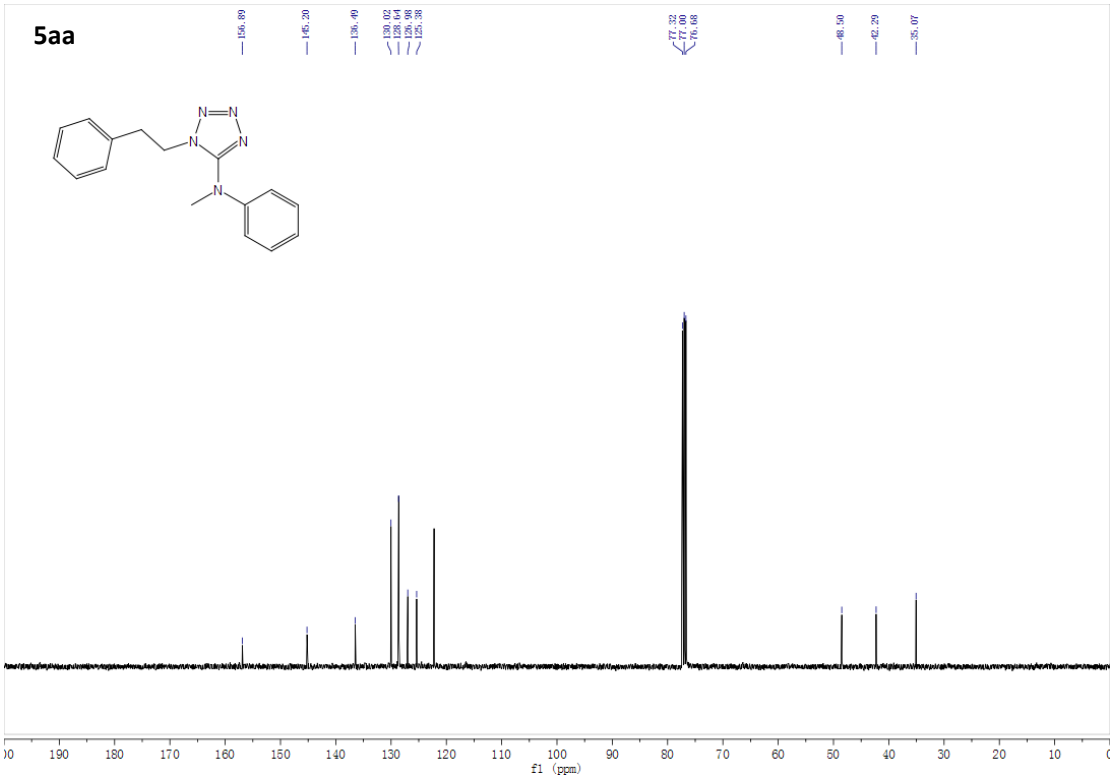
3Ra

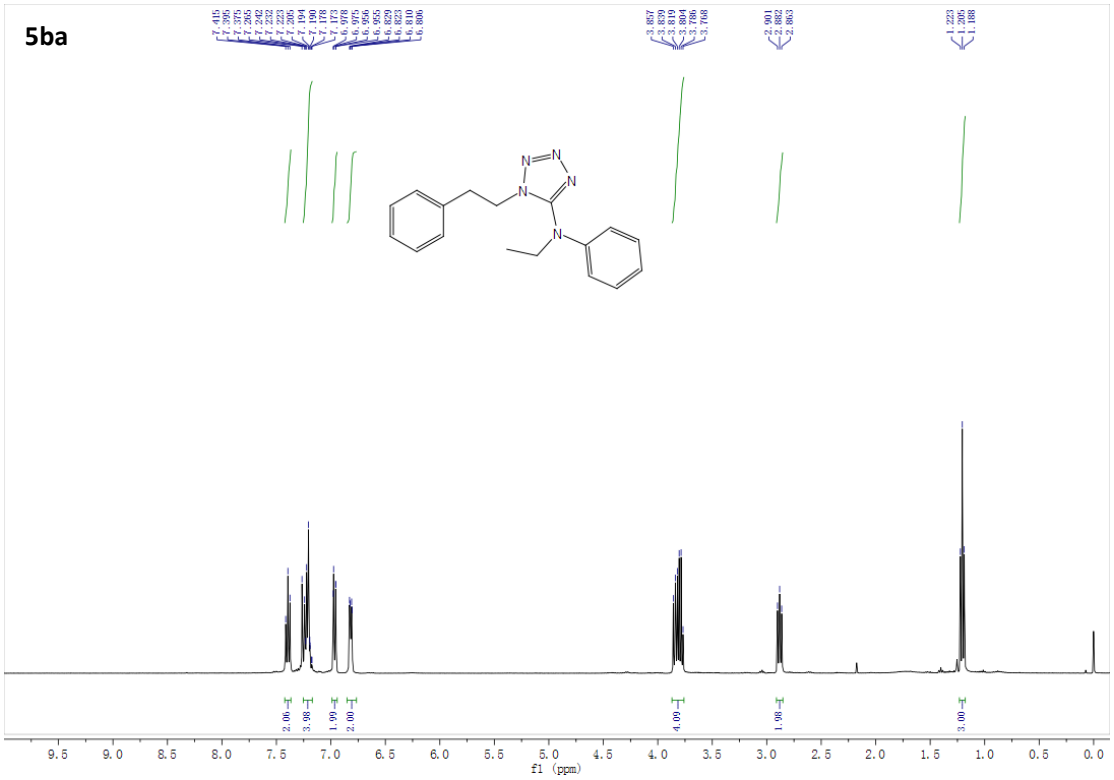

154.90
133.70
128.89
128.72
126.59
125.75
118.81
77.32
77.00
76.68
50.17
25.63
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

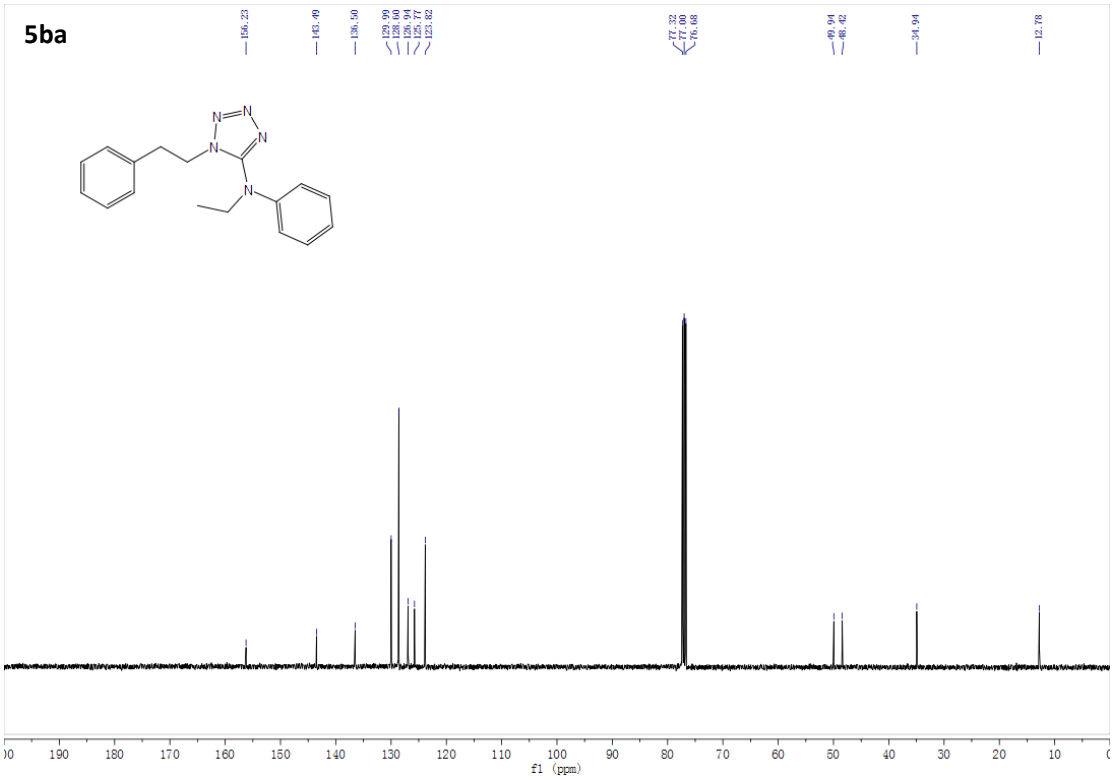

3Sa
7.399
6.786
6.782
6.778
6.773
6.732
6.728
6.743
6.739
6.397
6.393
6.323
6.306
6.288
6.271
3.603
3.596
3.592
3.586
3.581
3.576
3.569
2.023
2.014
2.006
1.998
1.989
1.902
1.897
1.894
1.889
0.000
0.97
0.94
4.00
4.00
2.97
f1 (ppm)

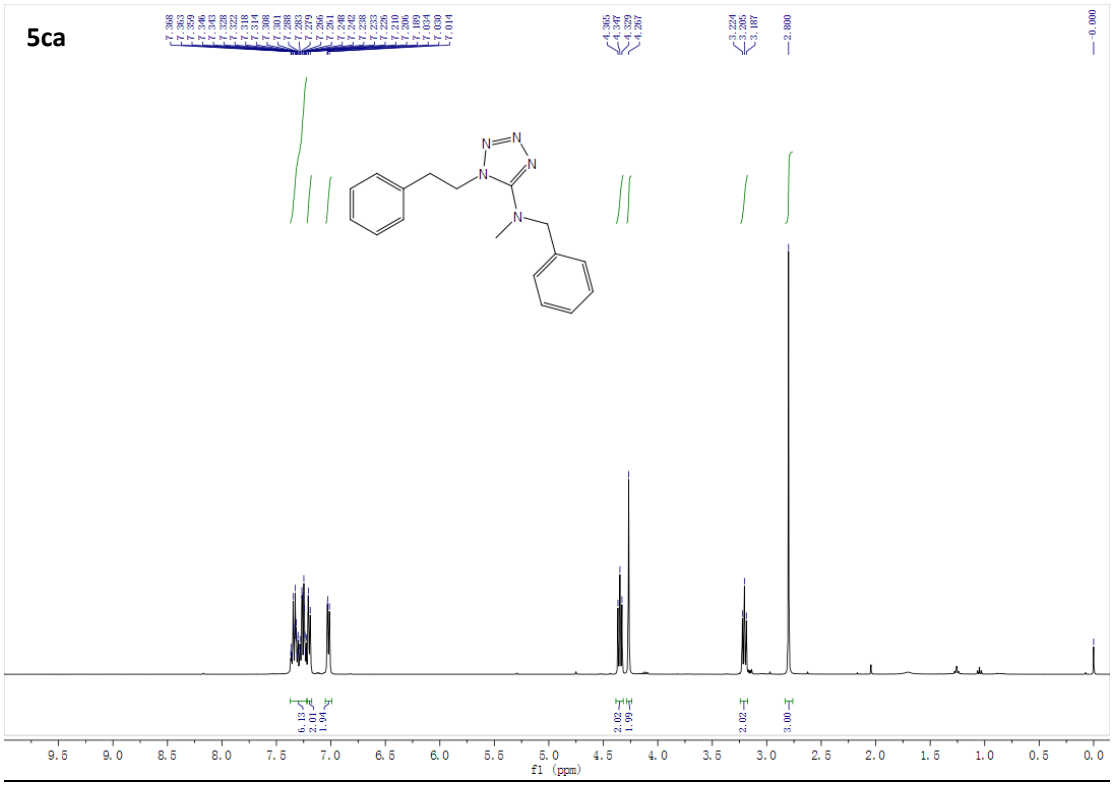

3Sa
154.71
124.72
120.96
77.32
77.00
76.68
50.00
25.65
15.24
f1 (ppm)

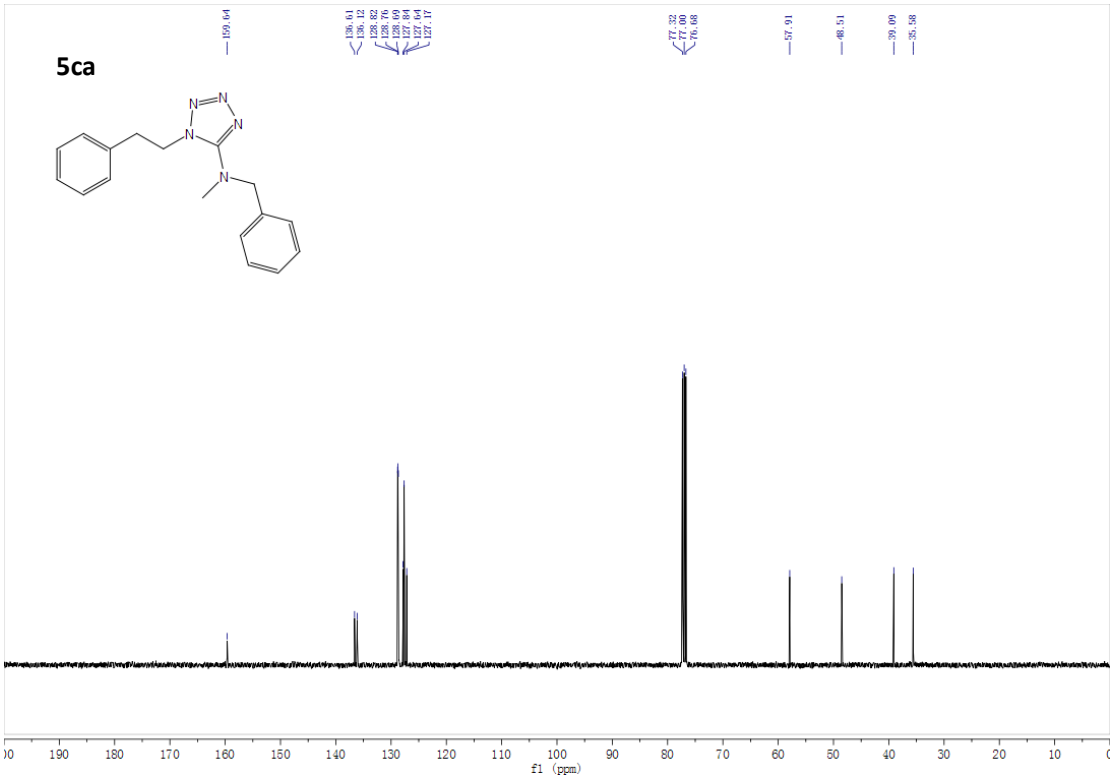

5aa
7.402
7.397
7.392
7.378
7.376
7.372
7.367
7.261
7.251
7.245
7.228
7.216
7.211
7.197
7.193
7.190
6.941
6.938
6.933
6.919
6.917
6.831
6.829
6.812
6.808
3.914
3.896
3.876
3.332
2.932
2.913
2.894
0.000
2.08
4.09
1.97
1.96
2.02
3.00
2.03
f1 (ppm)

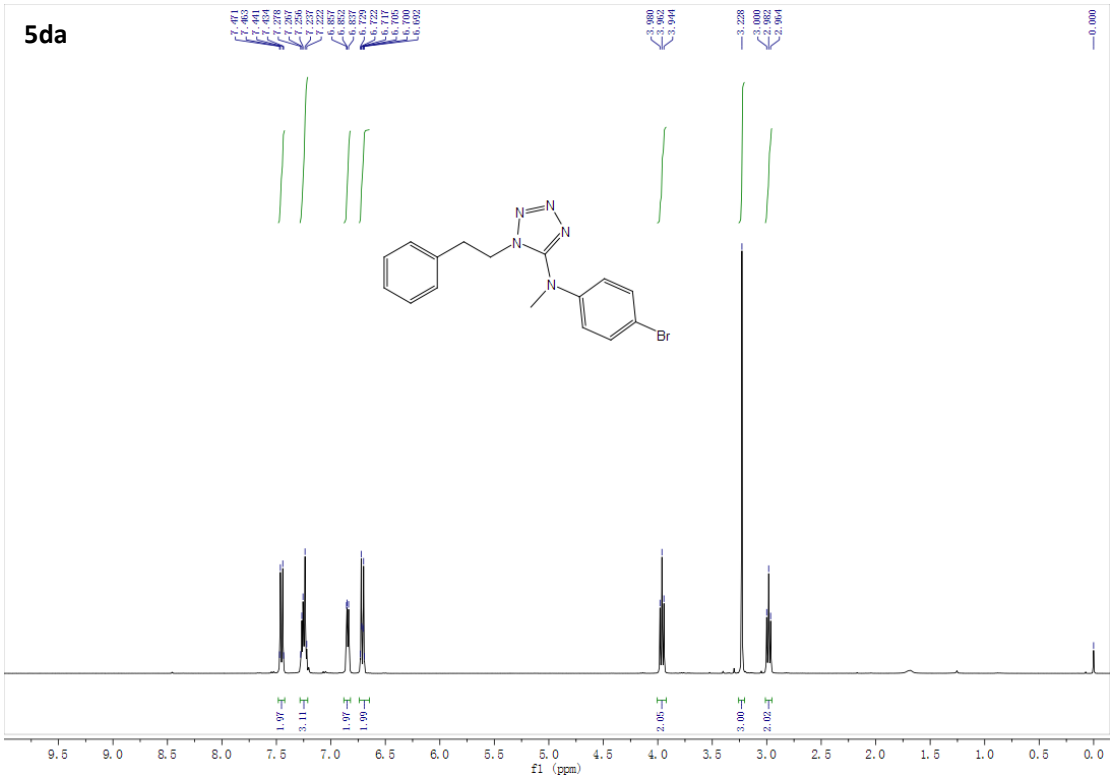

5aa
156.89
145.20
136.49
130.02
128.64
126.98
125.38
77.32
77.00
76.68
48.50
42.29
35.07
f1 (ppm)

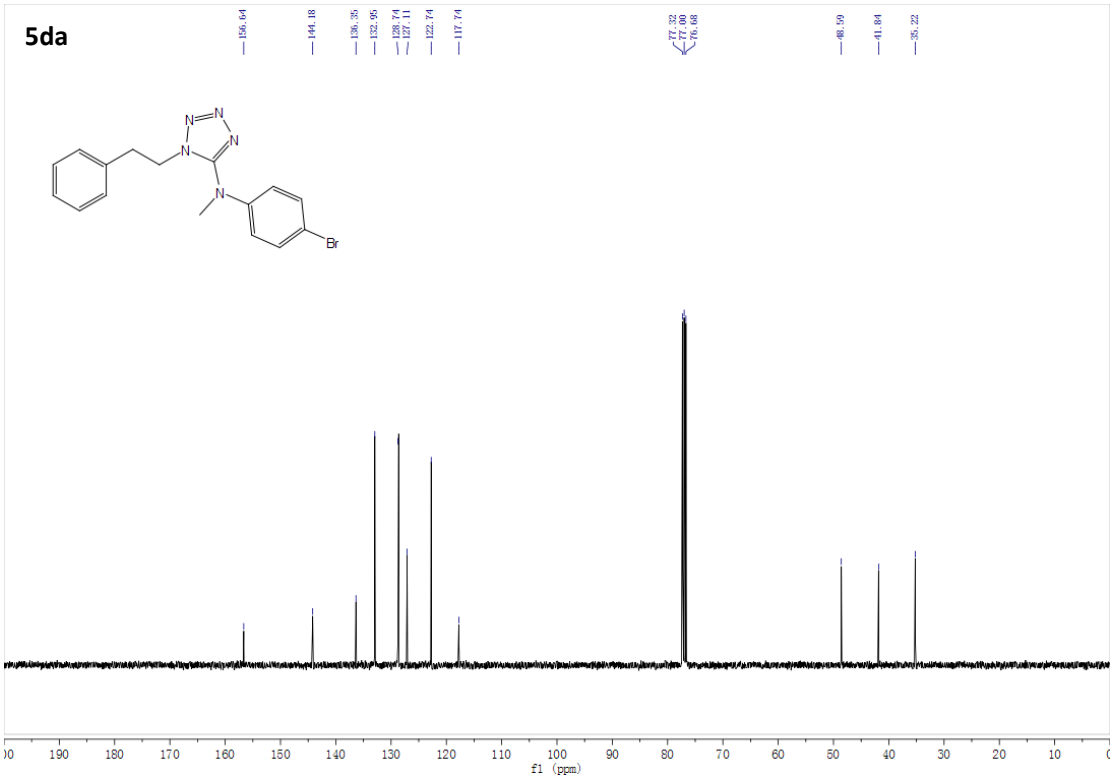

5ba
7.415
7.396
7.375
7.265
7.242
7.232
7.223
7.205
7.194
7.190
7.178
7.173
6.978
6.975
6.966
6.955
6.829
6.823
6.810
6.806
3.857
3.839
3.819
3.804
3.786
3.768
2.901
2.882
2.863
1.223
1.205
1.188
2.06
3.98
1.99
2.00
4.09
1.98
3.00
f1 (ppm)

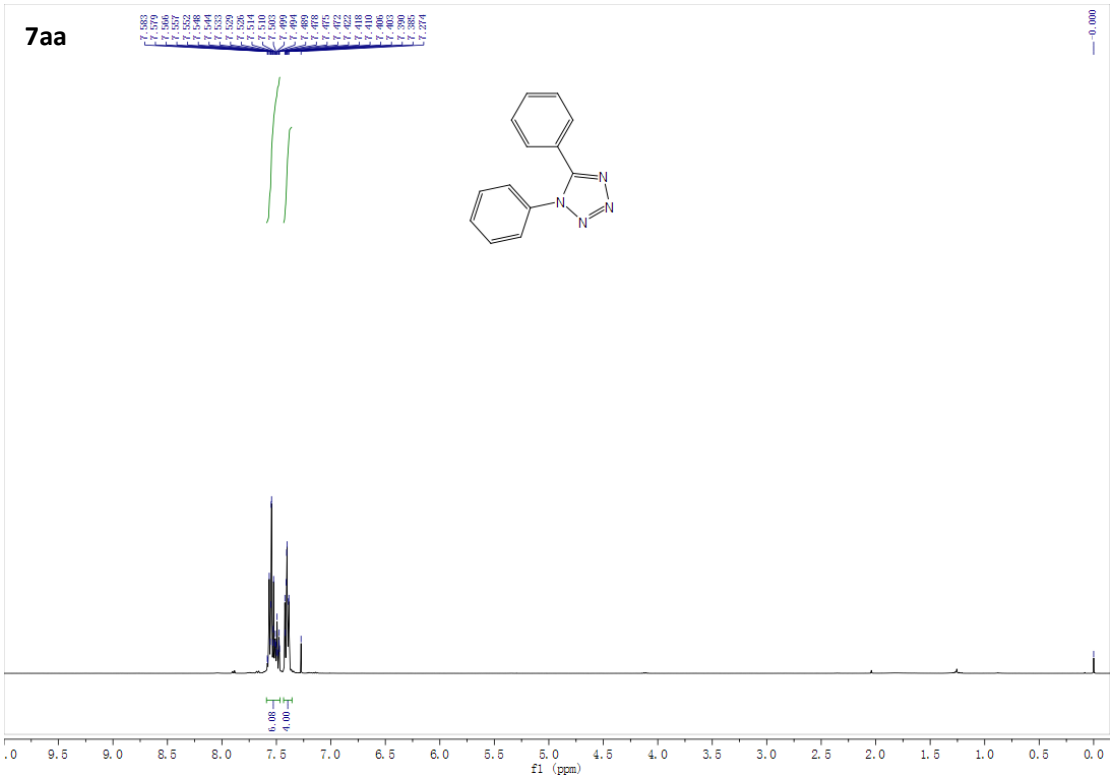

5ba
156.23
143.49
136.50
129.99
128.60
128.04
125.77
123.82
77.32
77.00
76.68
49.94
46.42
34.94
12.78
f1 (ppm)

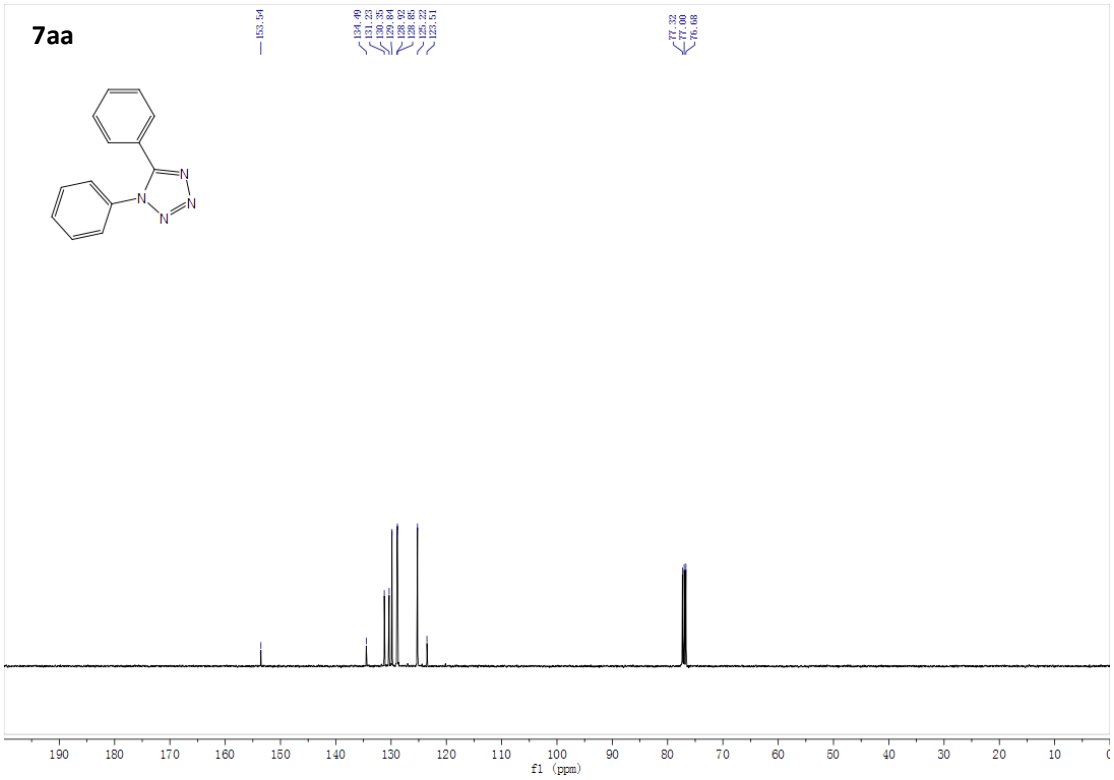

5ca
f1 (ppm)


5ca
f1 (ppm)

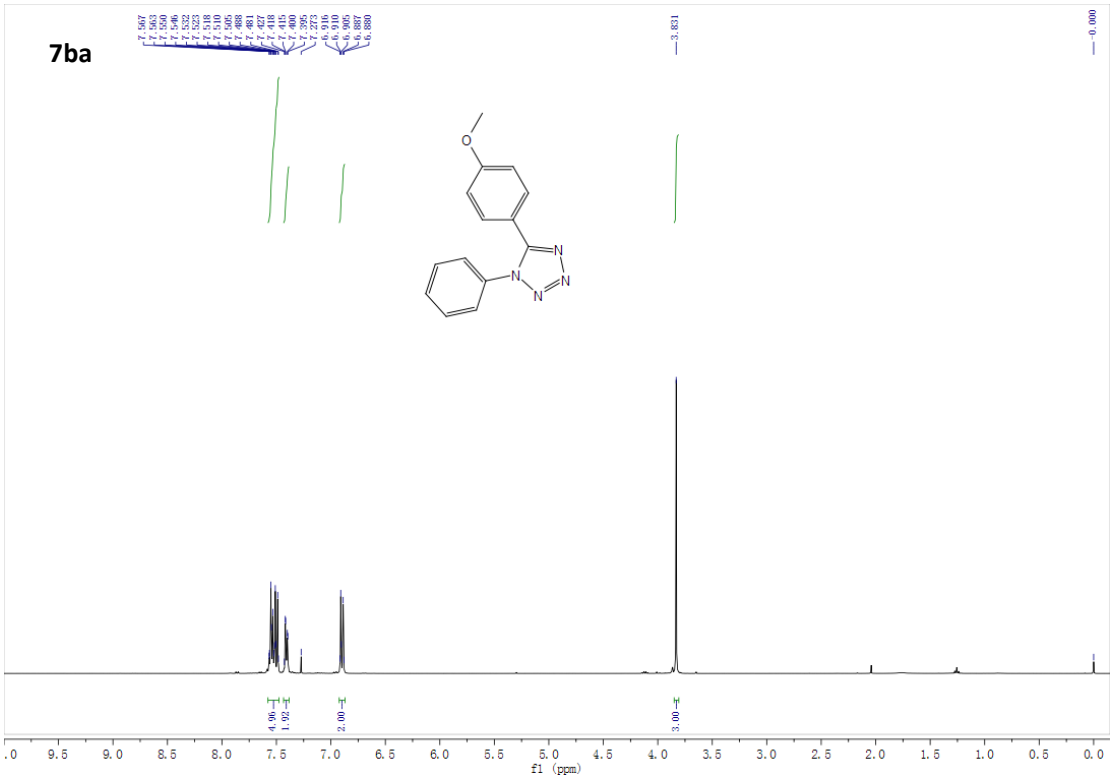

5da
7.471
7.463
7.441
7.434
7.278
7.267
7.256
7.227
7.222
6.857
6.852
6.737
6.729
6.722
6.717
6.705
6.700
6.692
3.980
3.962
3.944
3.228
3.000
2.982
2.964
0.000
N
N
N
N
N
Br
1.97
3.11
1.97
1.99
2.05
3.00
2.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


5da
156.64
144.18
136.35
132.95
128.74
127.11
122.74
117.74
77.32
77.00
76.68
48.59
41.84
35.22
N
N
N
N
N
Br
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

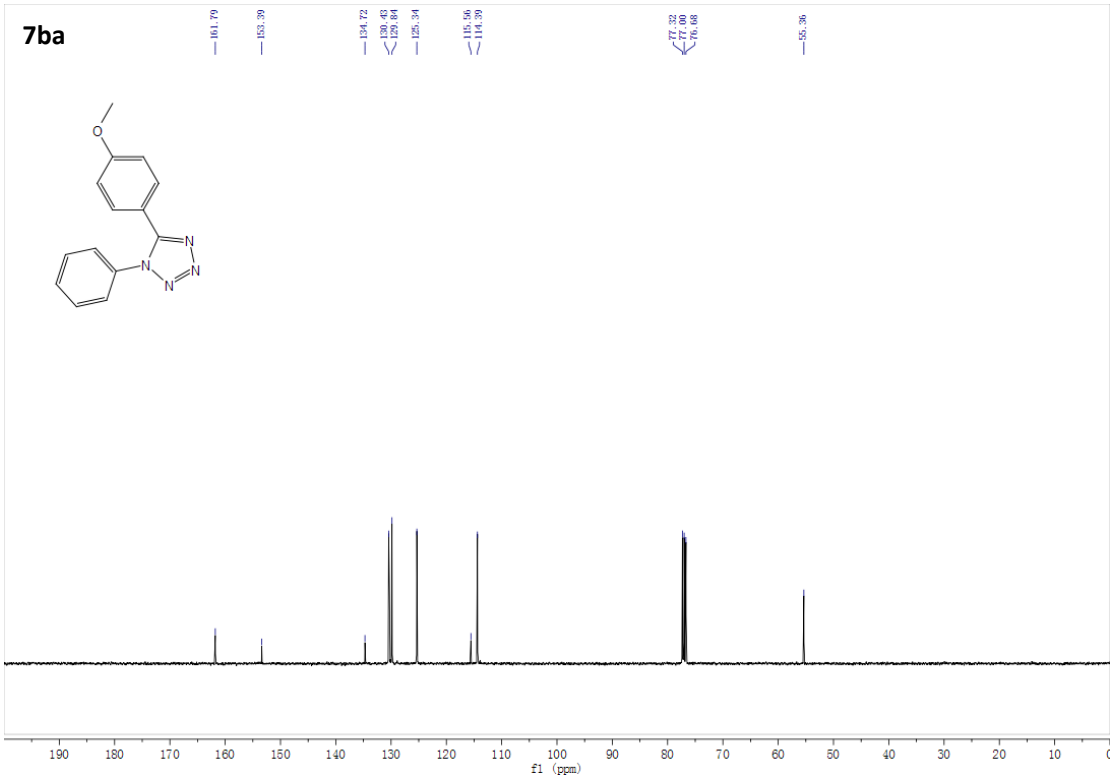

7aa
7.583
7.579
7.566
7.557
7.552
7.548
7.544
7.533
7.529
7.526
7.514
7.510
7.503
7.499
7.494
7.489
7.478
7.475
7.472
7.422
7.418
7.410
7.406
7.403
7.390
7.385
7.274
-0.000
6.08
4.00
f1 (ppm)

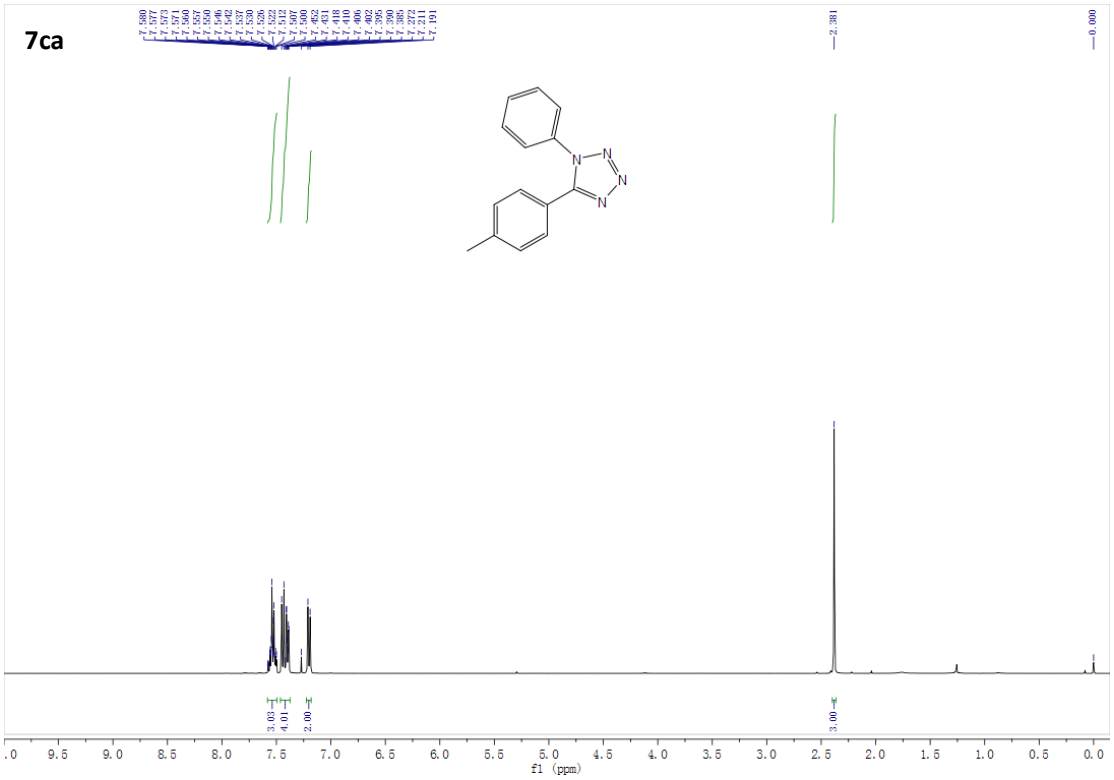

7aa
153.54
134.49
131.23
130.35
129.84
128.92
128.85
125.22
123.51
77.32
77.00
76.68
f1 (ppm)

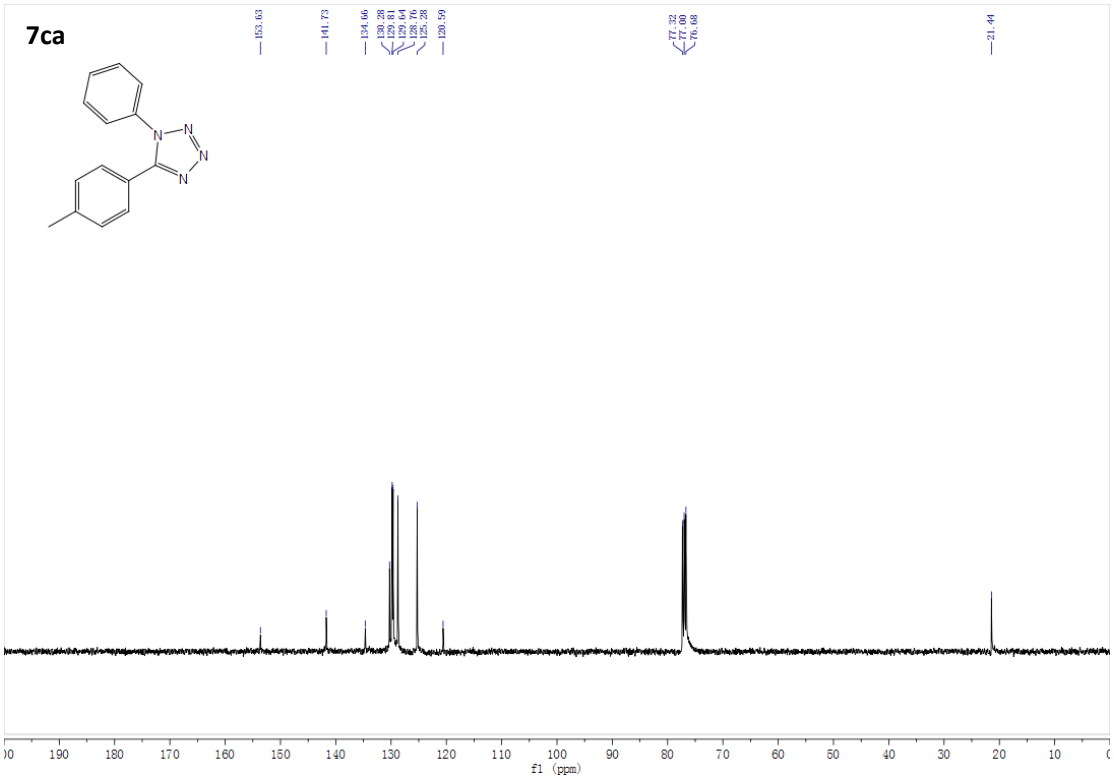
7ba

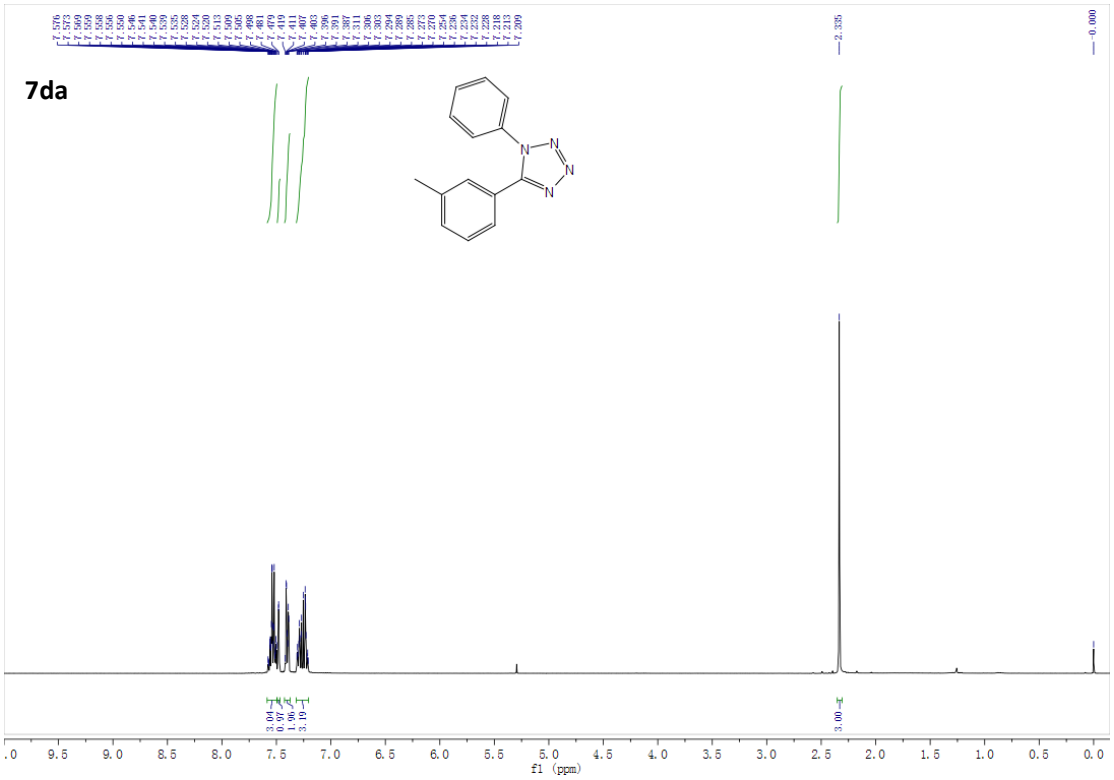

7.567
7.563
7.550
7.546
7.532
7.529
7.518
7.510
7.505
7.488
7.481
7.427
7.418
7.415
7.400
7.395
7.273
6.916
6.910
6.905
6.887
6.880
3.831
0.000
4.90
1.92
2.00
3.00
f1 (ppm)

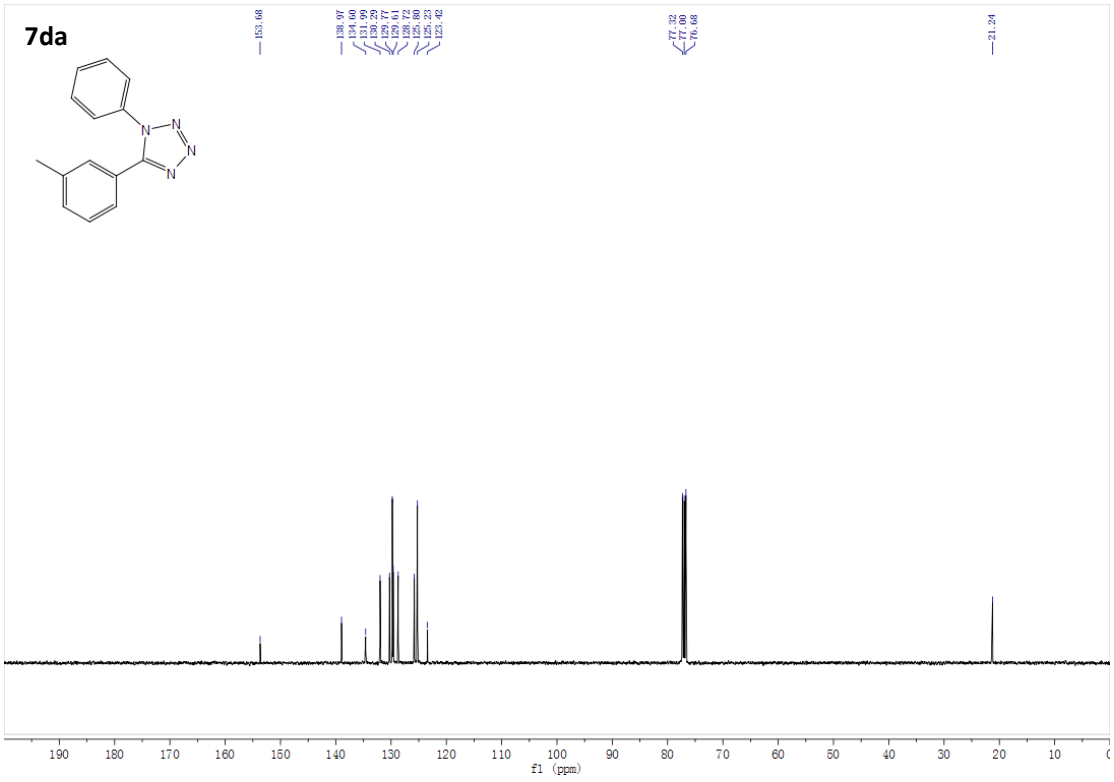
7ba

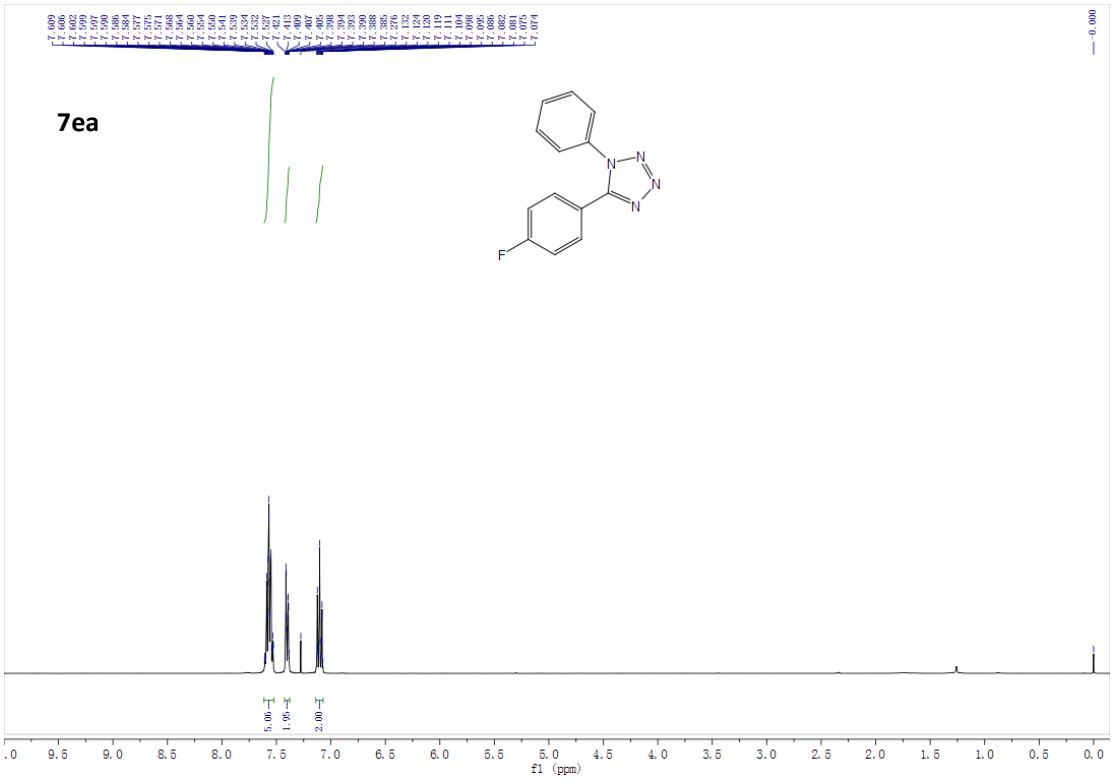

161.79
153.39
134.72
130.43
129.84
125.34
115.56
114.39
77.32
77.00
76.68
55.36
f1 (ppm)

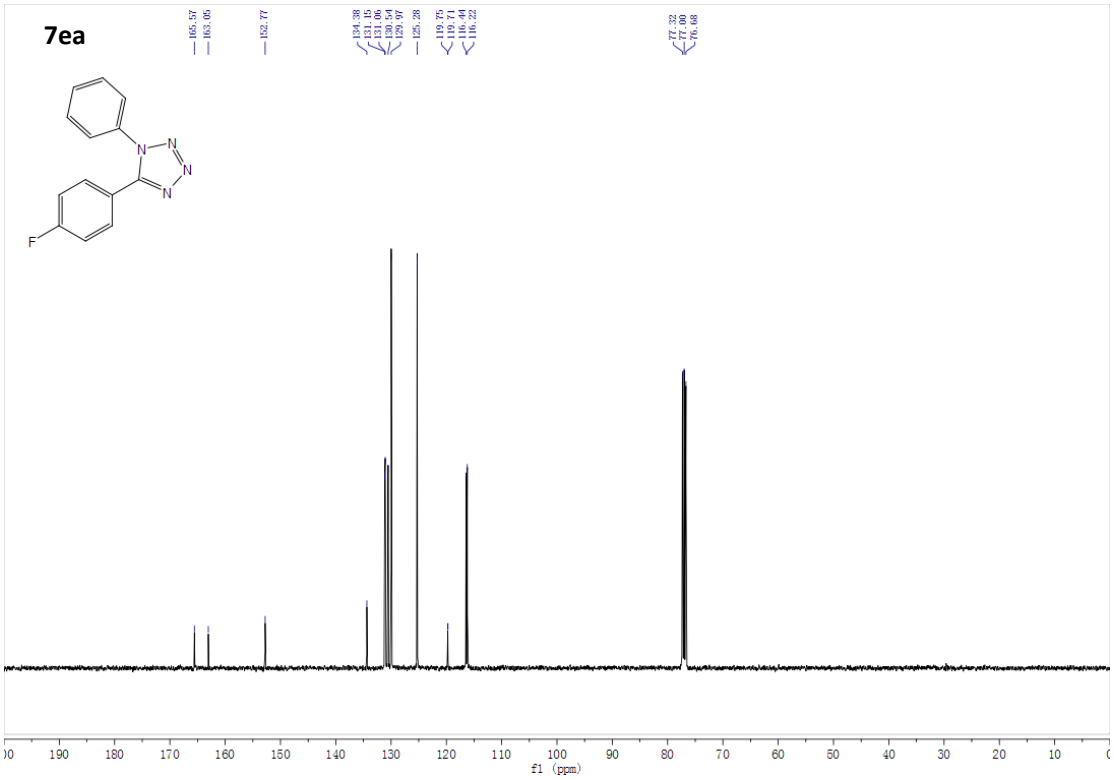

7ca
7.580
7.577
7.573
7.571
7.560
7.557
7.550
7.546
7.542
7.537
7.530
7.526
7.522
7.512
7.507
7.500
7.452
7.451
7.448
7.410
7.406
7.402
7.395
7.395
7.385
7.272
7.211
7.191
2.381
0.000
3.03
4.01
2.00
3.00
f1 (ppm)

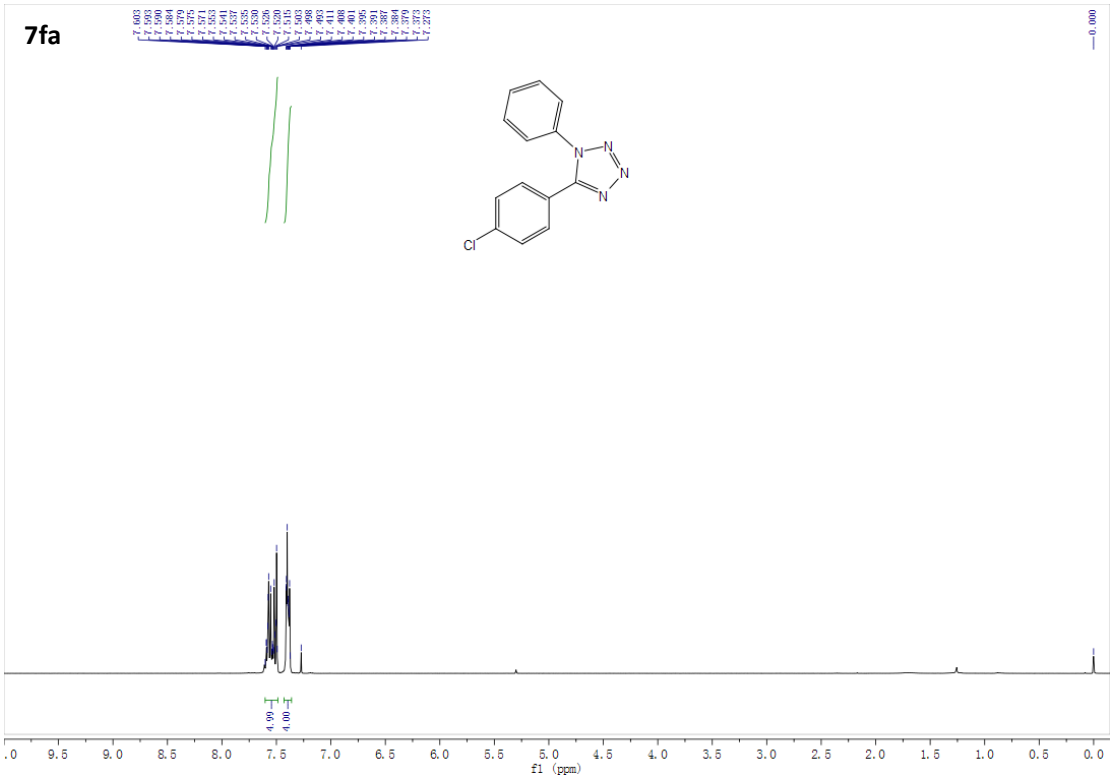

7ca
153.63
141.73
134.66
130.28
130.01
129.64
128.76
125.28
120.59
77.32
77.00
76.68
21.44
f1 (ppm)

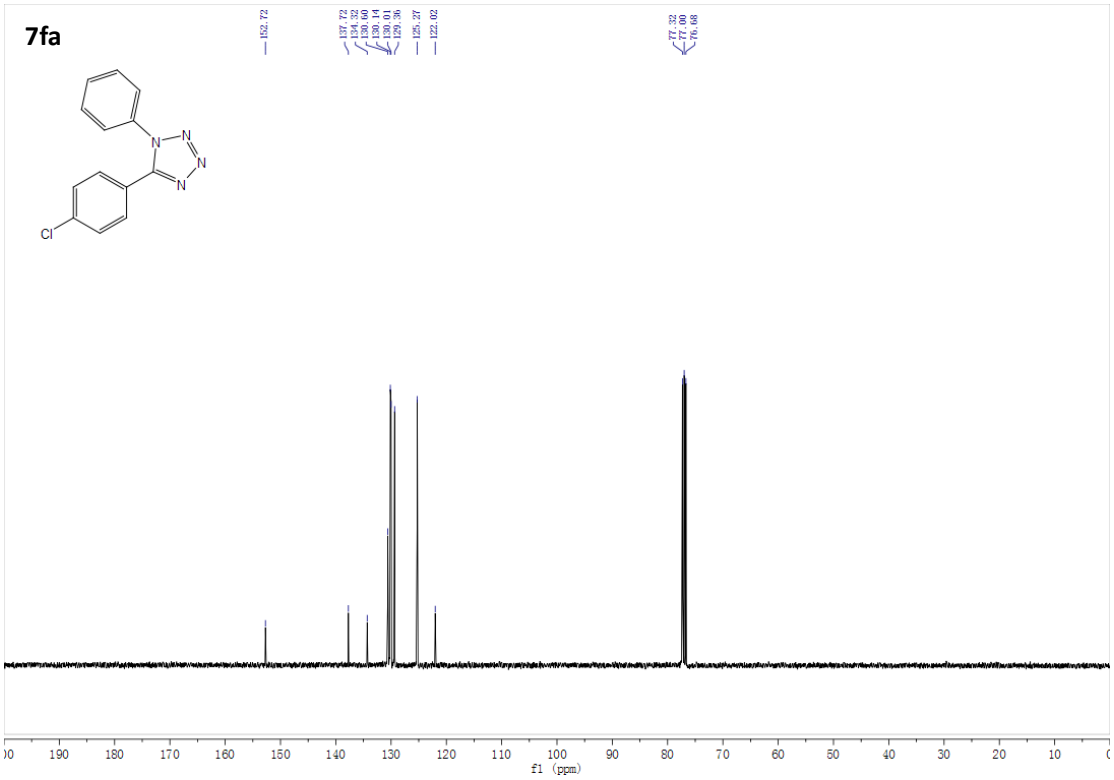

7da
f1 (ppm)

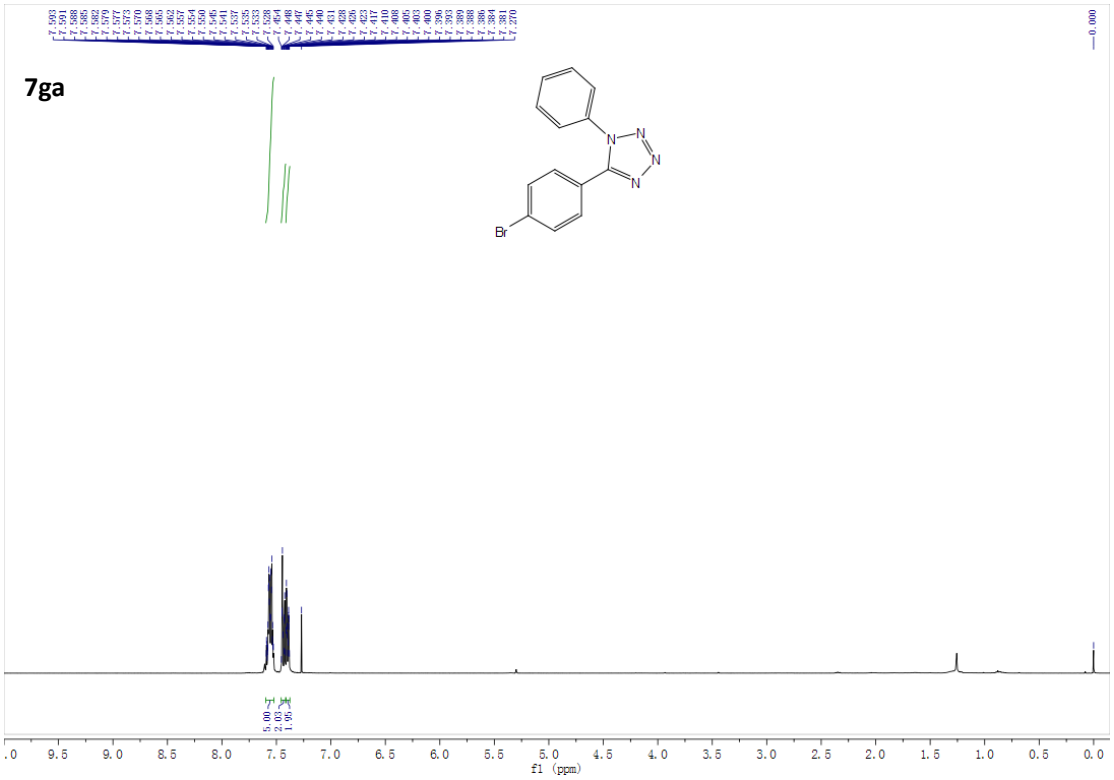

7da
f1 (ppm)

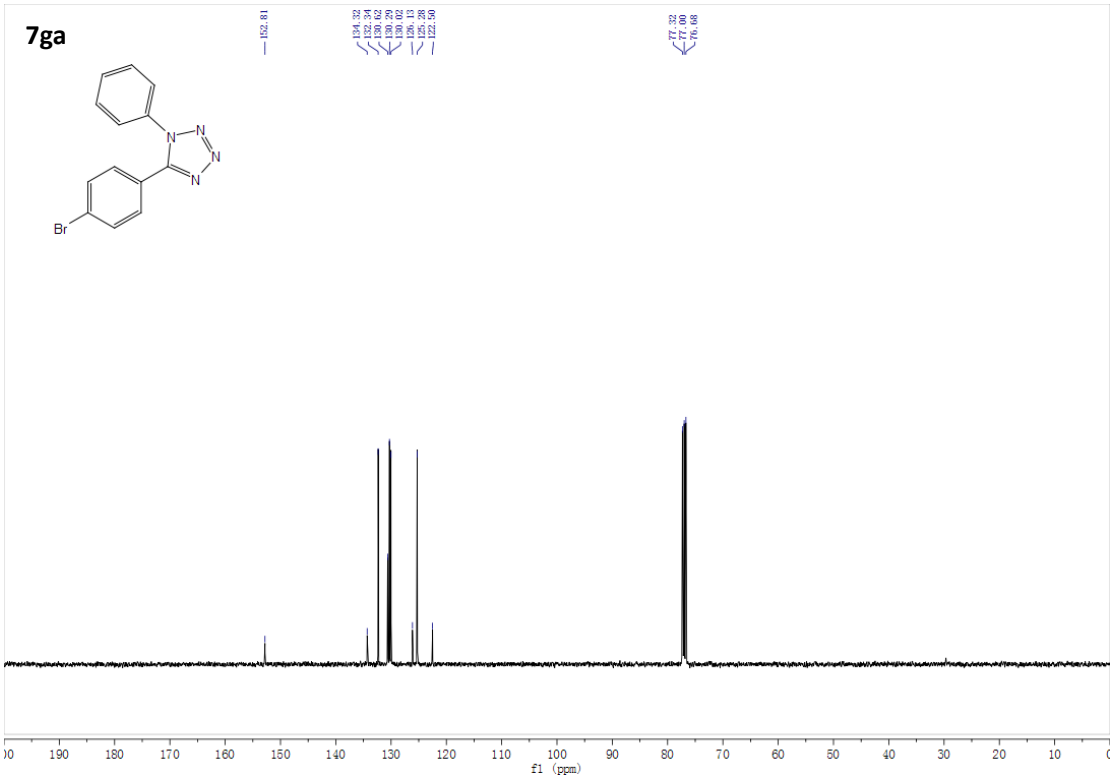

7ea
5.06
1.95
2.00
f1 (ppm)

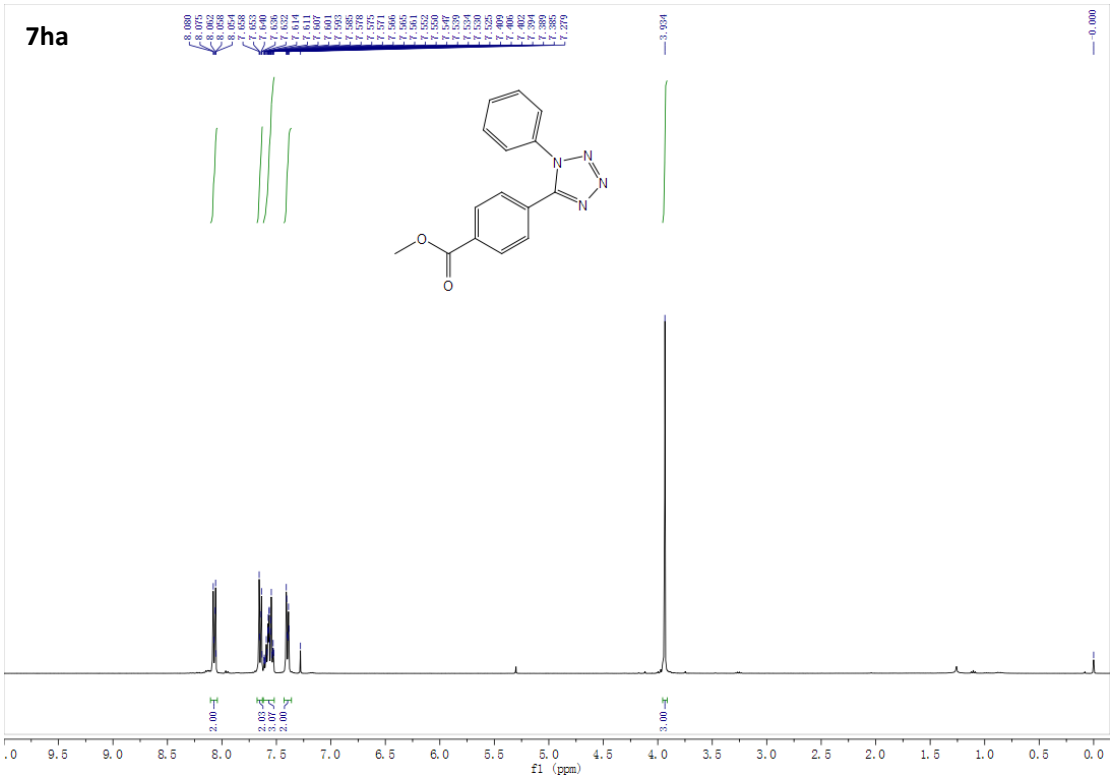

7ea
f1 (ppm)

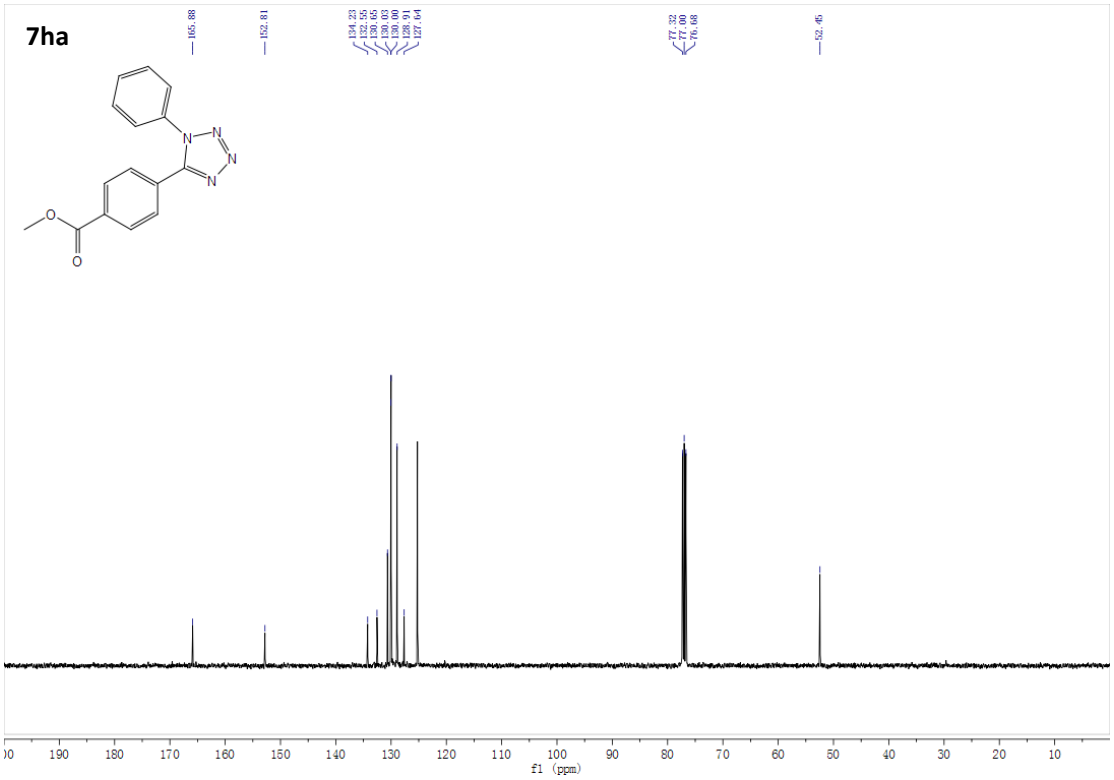

7fa
7.603
7.593
7.590
7.584
7.579
7.575
7.571
7.553
7.541
7.537
7.535
7.530
7.526
7.515
7.503
7.498
7.493
7.411
7.408
7.401
7.395
7.391
7.387
7.384
7.379
7.373
7.273
0.000
4.99
4.00
f1 (ppm)

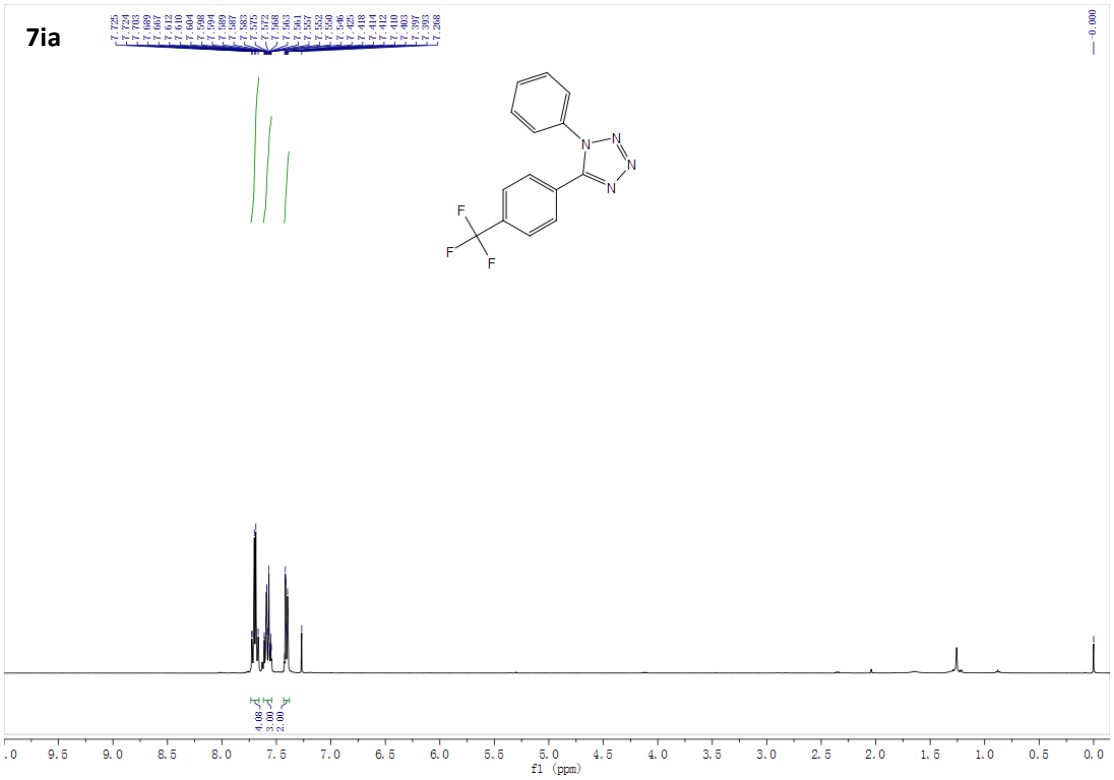

7fa
152.72
137.72
134.32
130.59
130.14
130.01
129.36
125.27
122.02
77.32
77.00
76.68
f1 (ppm)

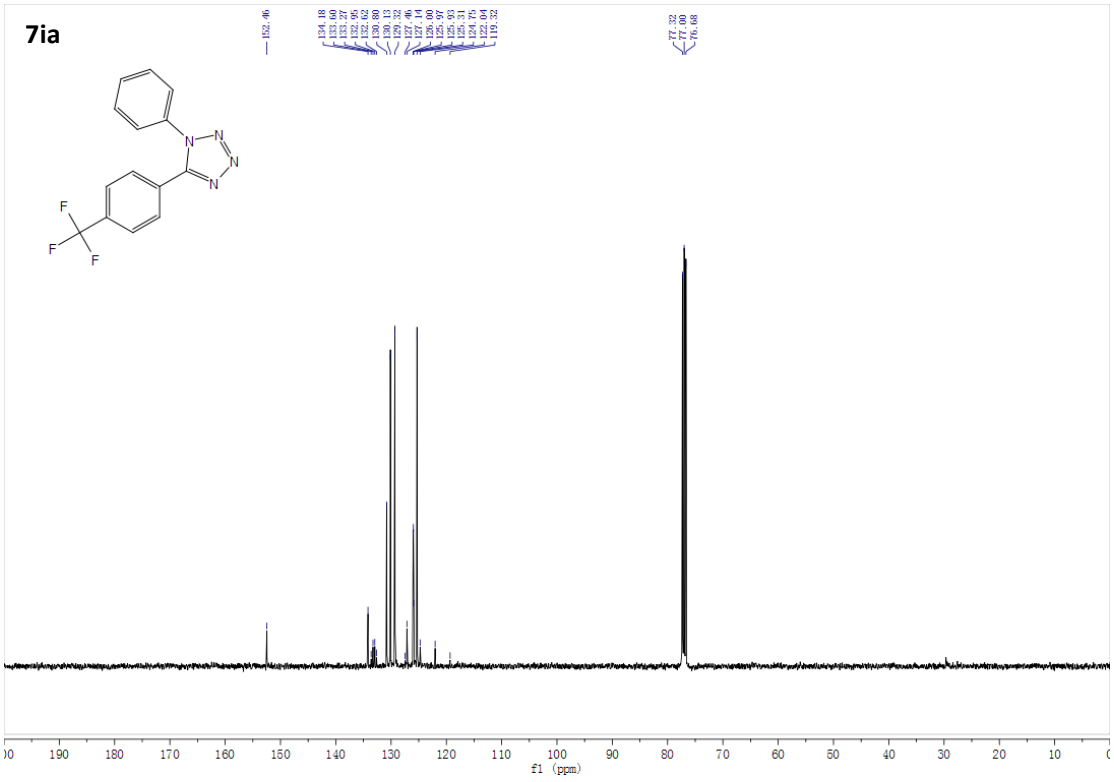

7ga
Br
f1 (ppm)

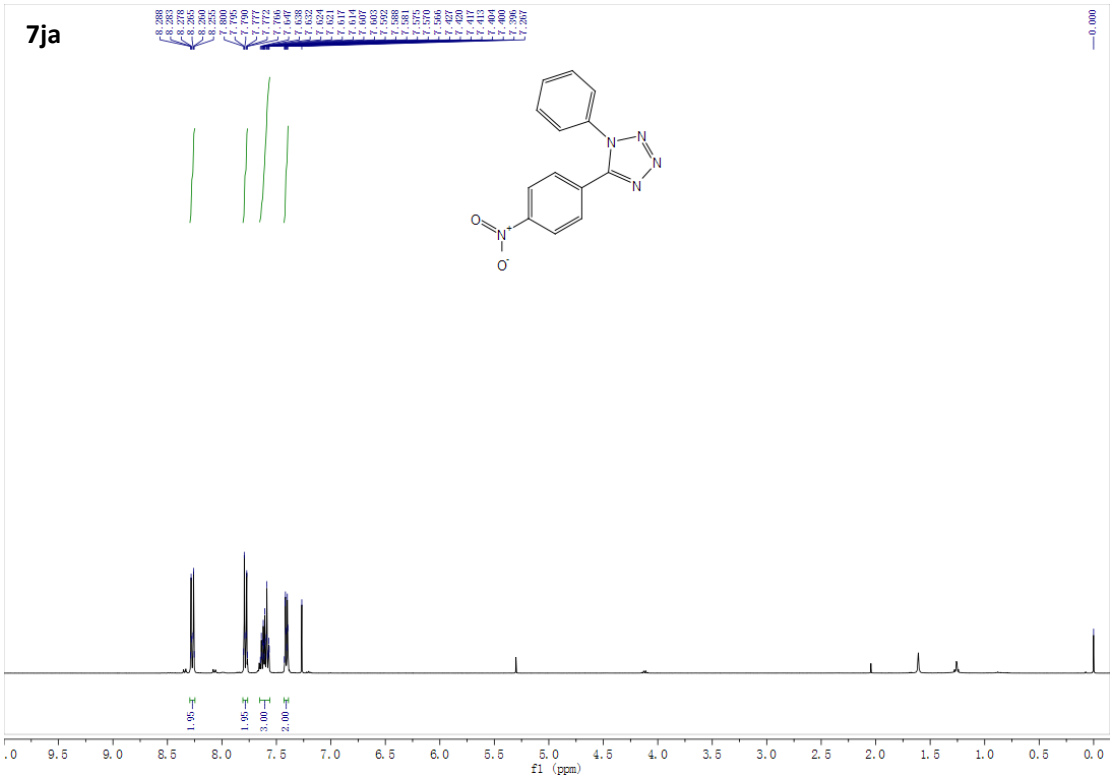

7ga
Br
f1 (ppm)


7ha
8.080
8.075
8.062
8.058
8.054
7.658
7.655
7.640
7.636
7.632
7.614
7.611
7.607
7.601
7.595
7.585
7.578
7.575
7.571
7.566
7.565
7.561
7.552
7.550
7.547
7.539
7.534
7.530
7.525
7.409
7.406
7.402
7.399
7.389
7.385
7.279
3.934
0.000
2.00
2.03
3.07
2.00
3.00
f1 (ppm)


7ha
165.88
152.81
134.23
132.55
130.65
130.03
130.00
128.71
127.64
77.32
77.00
76.68
52.45
f1 (ppm)


7ia
f1 (ppm)


7ia
f1 (ppm)


7ja
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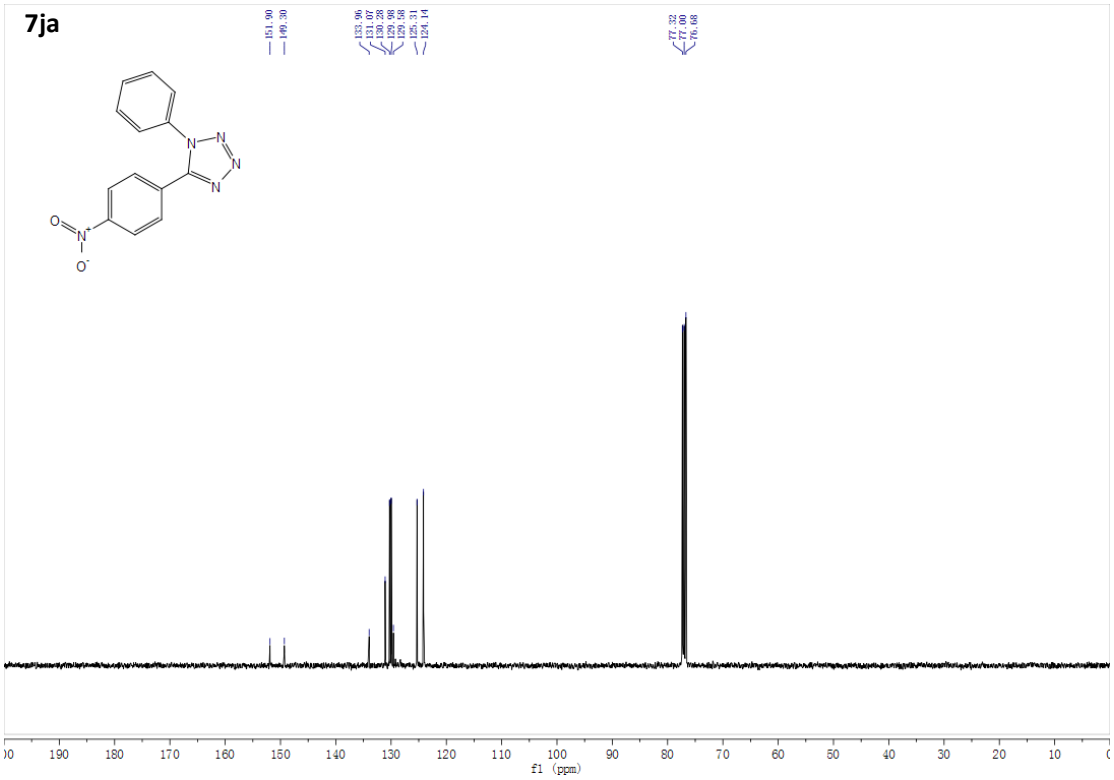
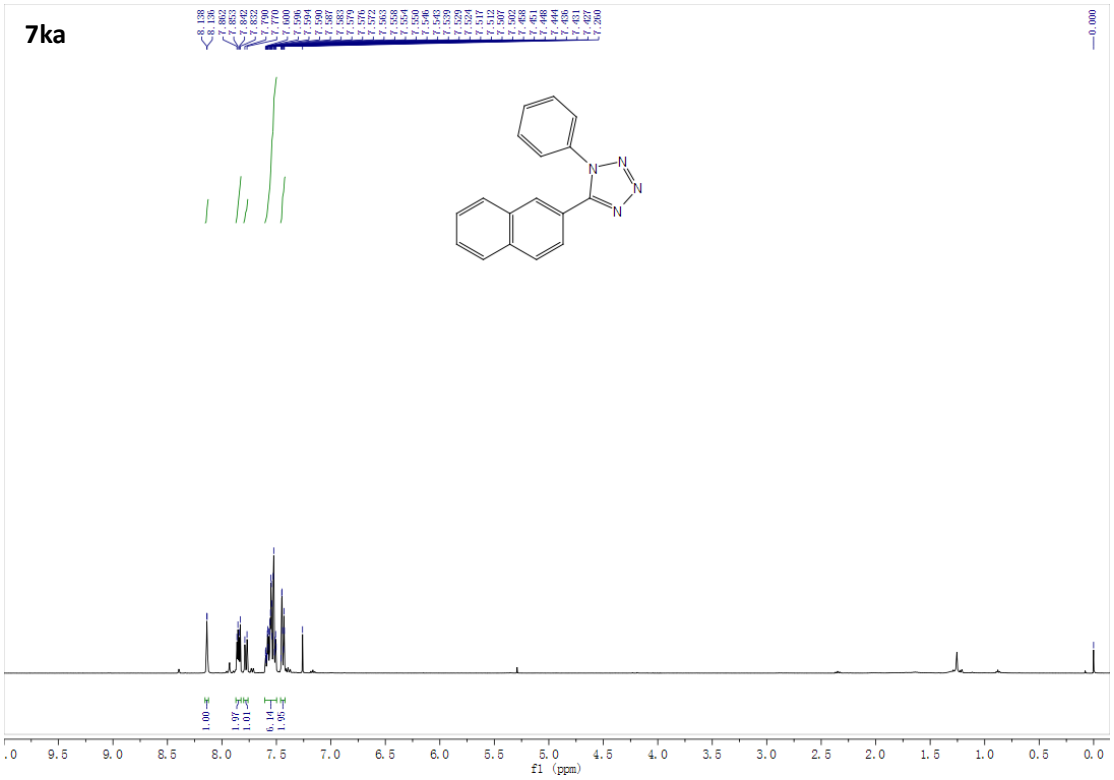
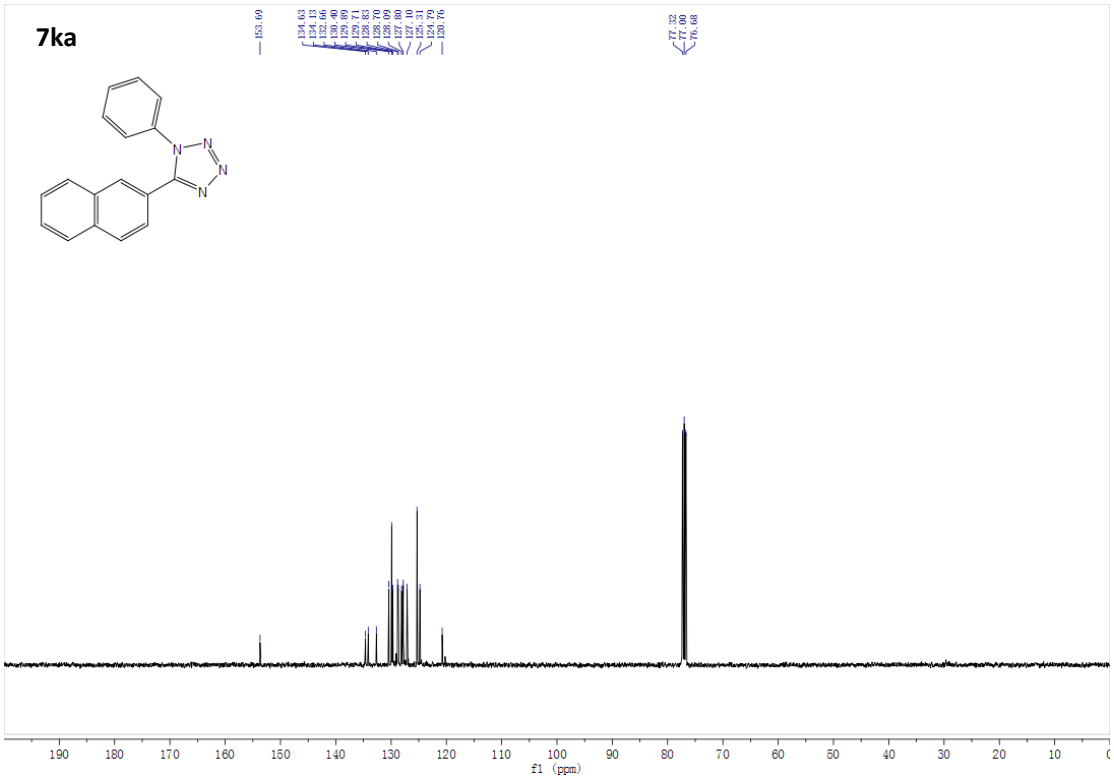
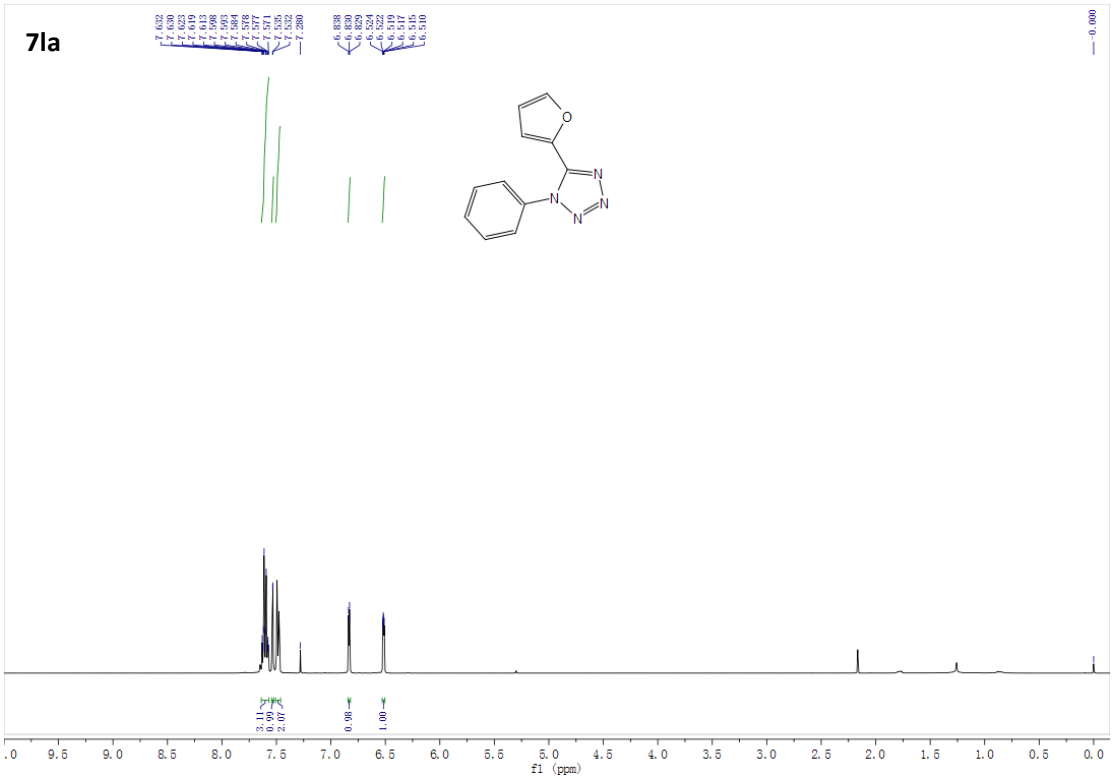
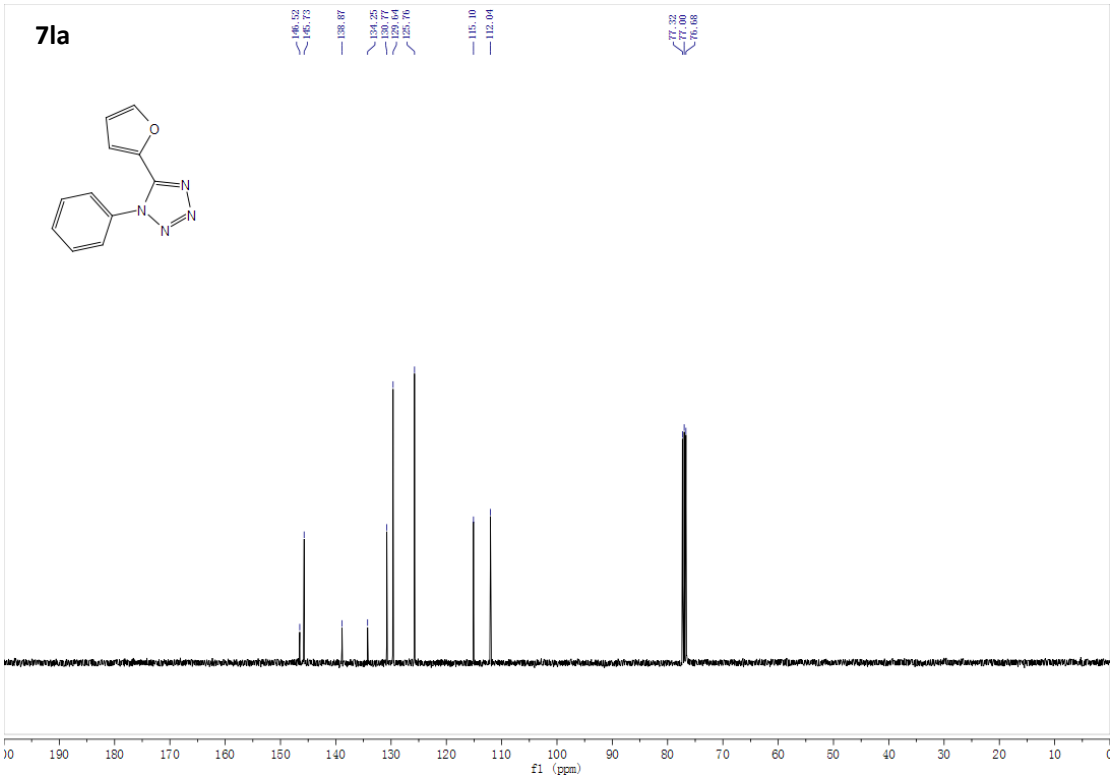
8.278
8.265
8.260
8.255
8.800
7.795
7.790
7.777
7.772
7.766
7.647
7.638
7.632
7.624
7.621
7.617
7.614
7.607
7.603
7.592
7.588
7.584
7.575
7.570
7.566
7.557
7.420
7.417
7.413
7.406
7.400
7.396
7.267
0.000
1.95
1.95
3.00
2.00
f1 (ppm)

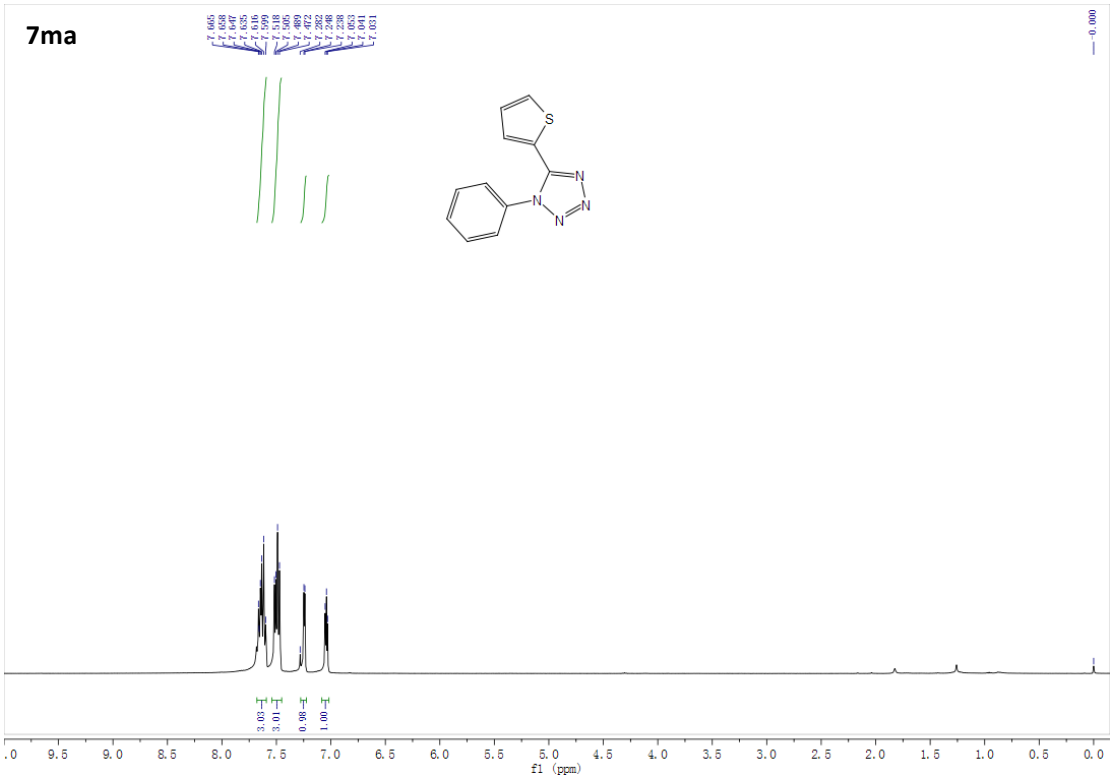

7ja
151.90
149.30
133.96
131.07
130.38
129.98
129.58
125.31
124.14
77.32
77.00
76.68
f1 (ppm)

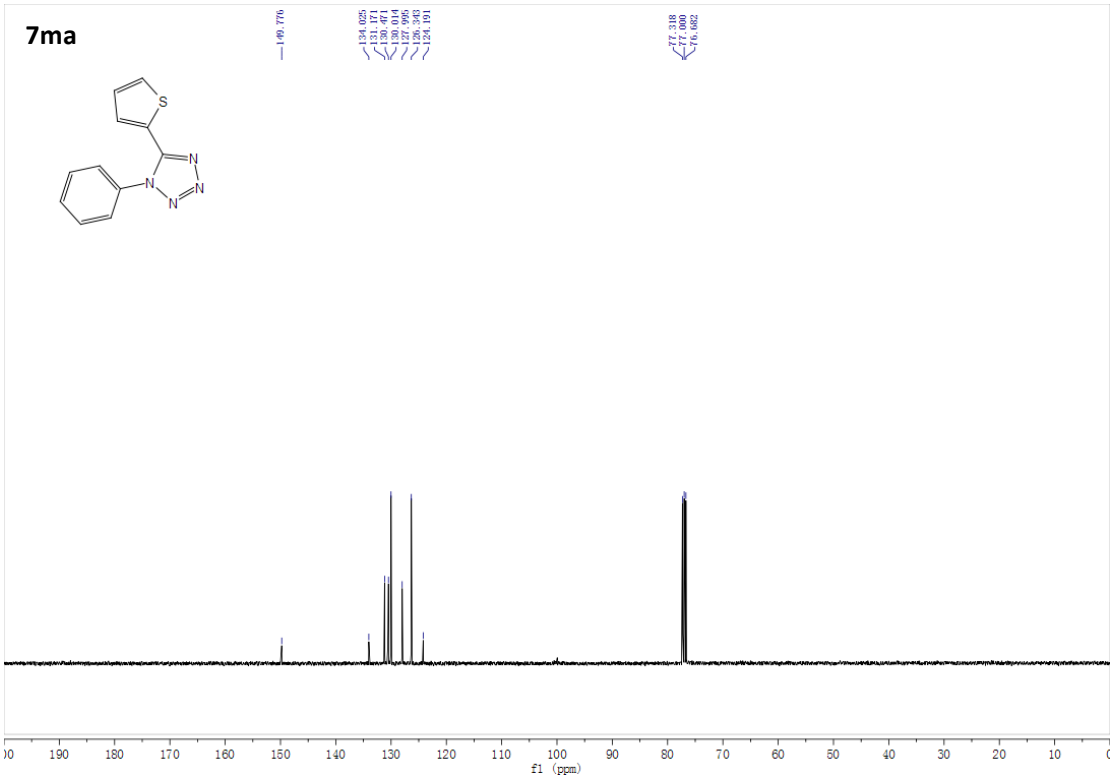

7ka
8.138
8.136
7.862
7.853
7.842
7.832
7.790
7.770
7.600
7.596
7.594
7.590
7.587
7.583
7.579
7.576
7.572
7.563
7.558
7.554
7.550
7.546
7.543
7.539
7.529
7.524
7.517
7.512
7.507
7.502
7.458
7.451
7.448
7.444
7.436
7.431
7.427
7.260
0.000
1.00
1.97
1.01
6.14
1.90
f1 (ppm)

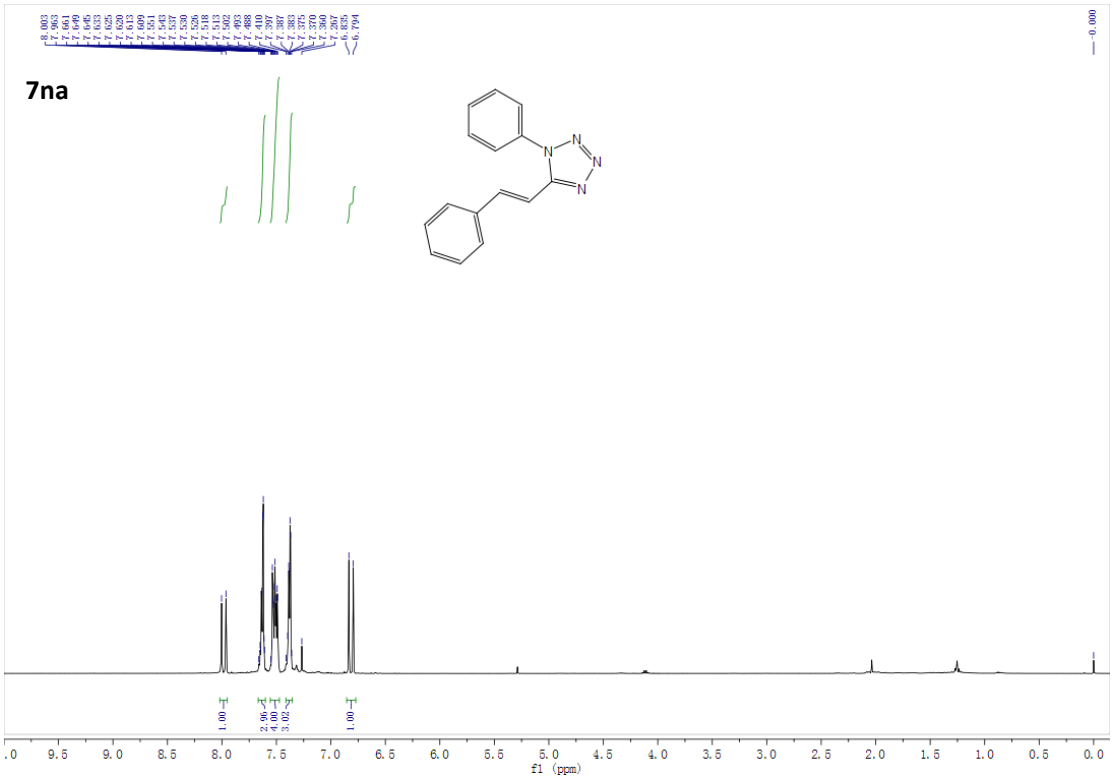

7ka
153.69
134.63
134.13
132.46
130.40
129.89
129.71
128.95
128.70
128.09
127.80
127.10
125.31
124.79
120.76
77.32
77.00
76.68
f1 (ppm)

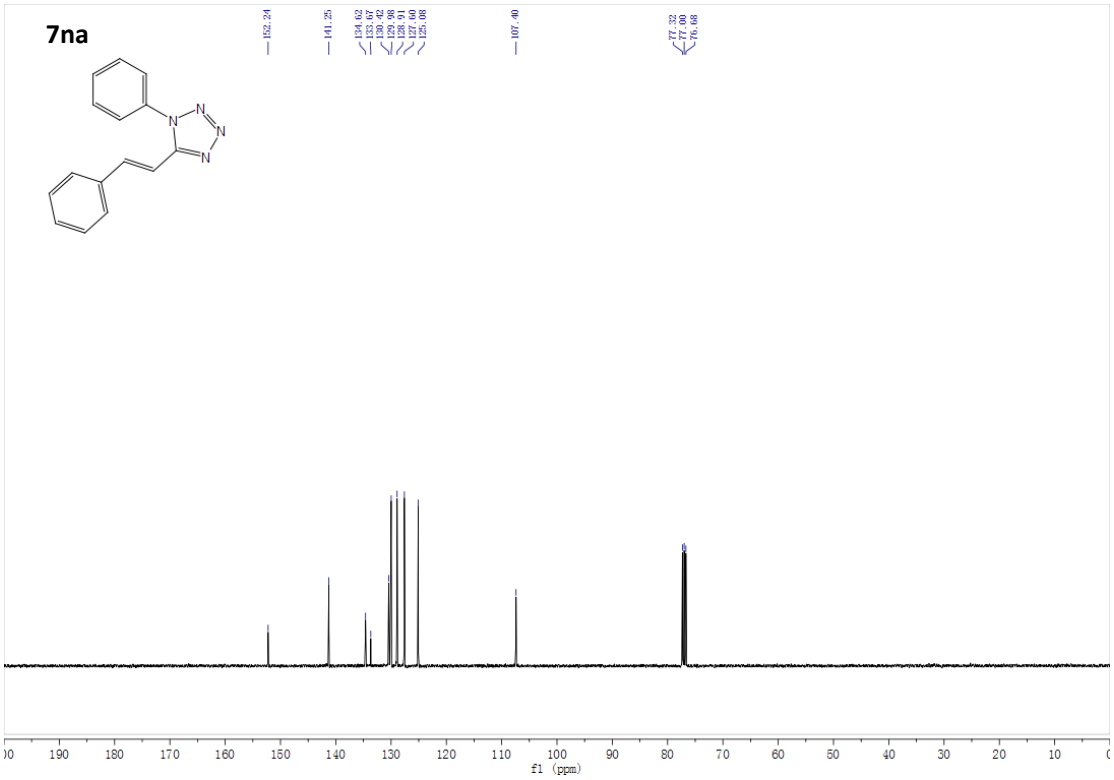

7la
7.632
7.630
7.625
7.619
7.613
7.598
7.595
7.584
7.578
7.577
7.571
7.535
7.532
7.280
6.838
6.830
6.829
6.524
6.522
6.519
6.517
6.515
6.510
-0.000
O
N
N
N
N
3.11
0.99
2.07
0.98
1.00
f1 (ppm)

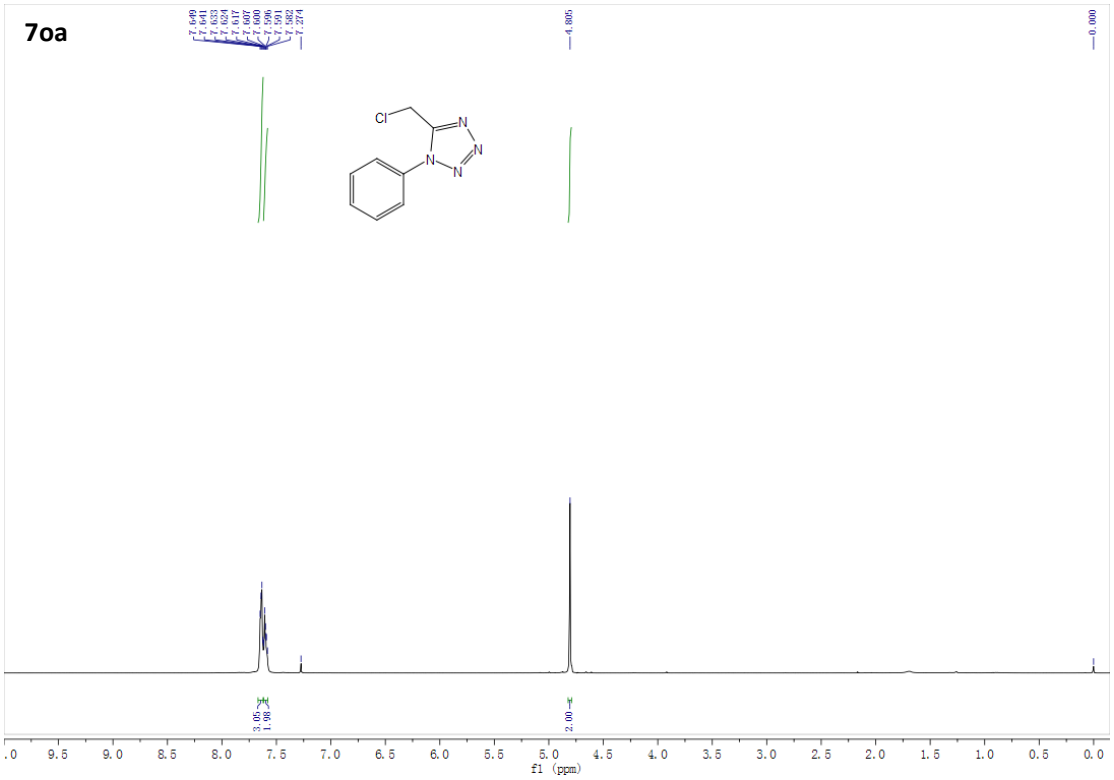

7la
146.52
145.73
138.87
134.25
130.77
129.64
125.76
115.10
112.04
77.32
77.00
76.68
O
N
N
N
N
f1 (ppm)

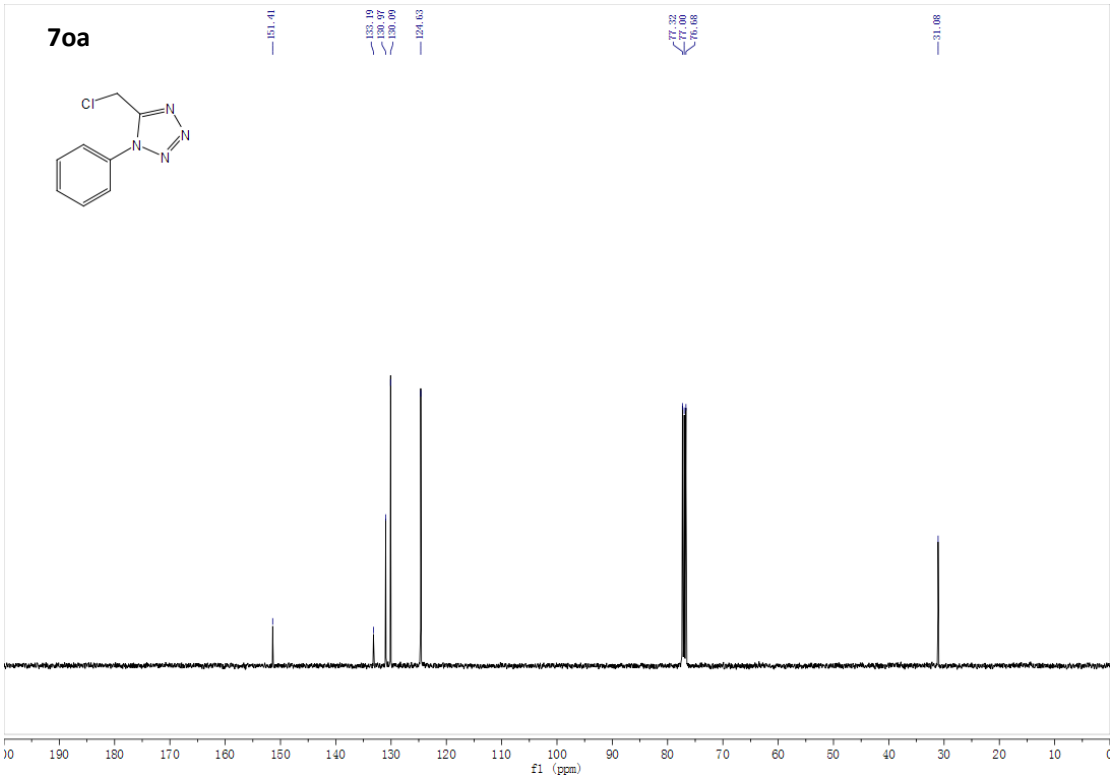

7ma
7.665
7.658
7.647
7.635
7.616
7.599
7.518
7.505
7.489
7.472
7.382
7.298
7.238
7.055
7.041
7.031
-0.000
S
N
N
N
3.03
3.01
0.98
1.00
f1 (ppm)

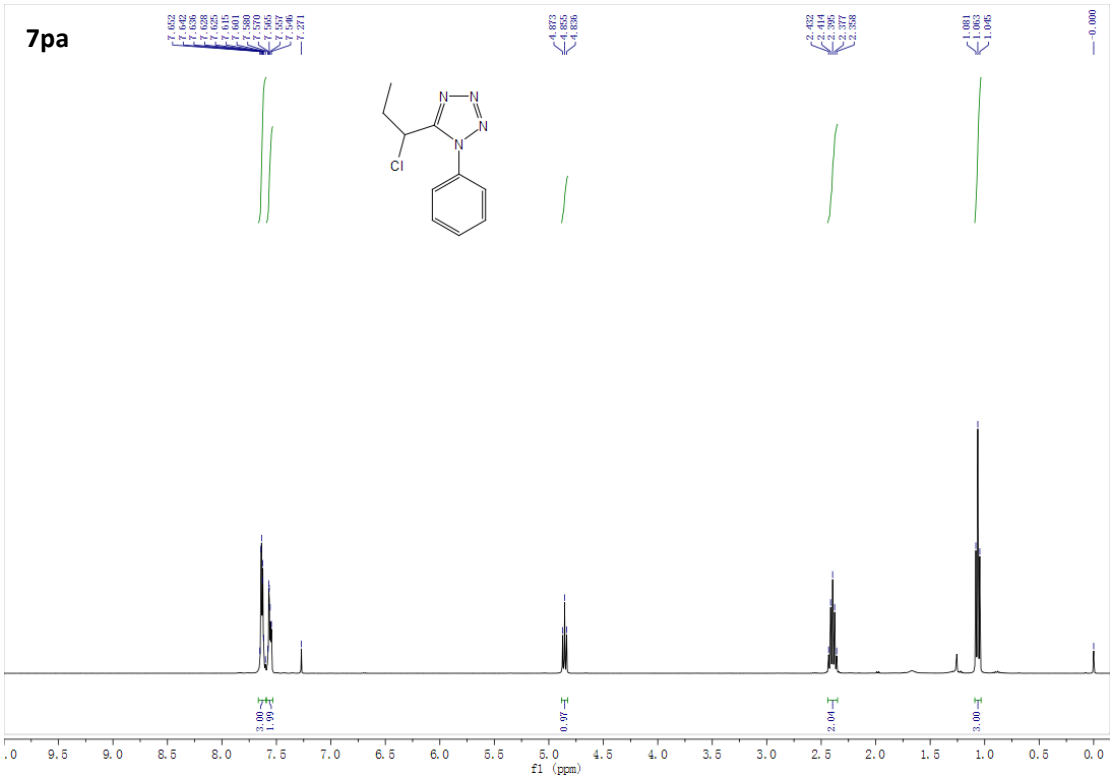

7ma
149.776
134.025
131.171
130.471
129.014
127.995
126.343
124.191
77.318
77.000
76.682
S
N
N
N
f1 (ppm)

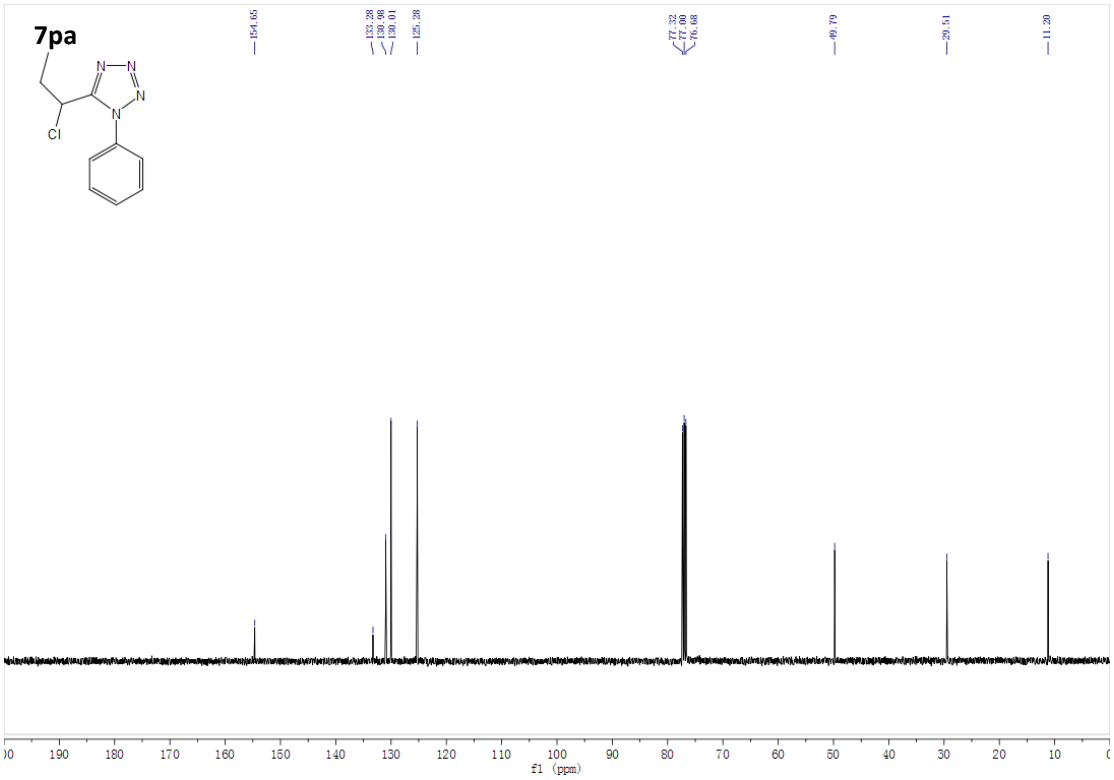

7na
8.002
7.963
7.661
7.649
7.645
7.635
7.625
7.620
7.613
7.609
7.551
7.545
7.537
7.530
7.526
7.518
7.513
7.507
7.495
7.488
7.410
7.397
7.387
7.383
7.375
7.370
7.369
7.297
6.835
6.794
0.000
1.00
2.96
4.00
3.02
1.00
f1 (ppm)

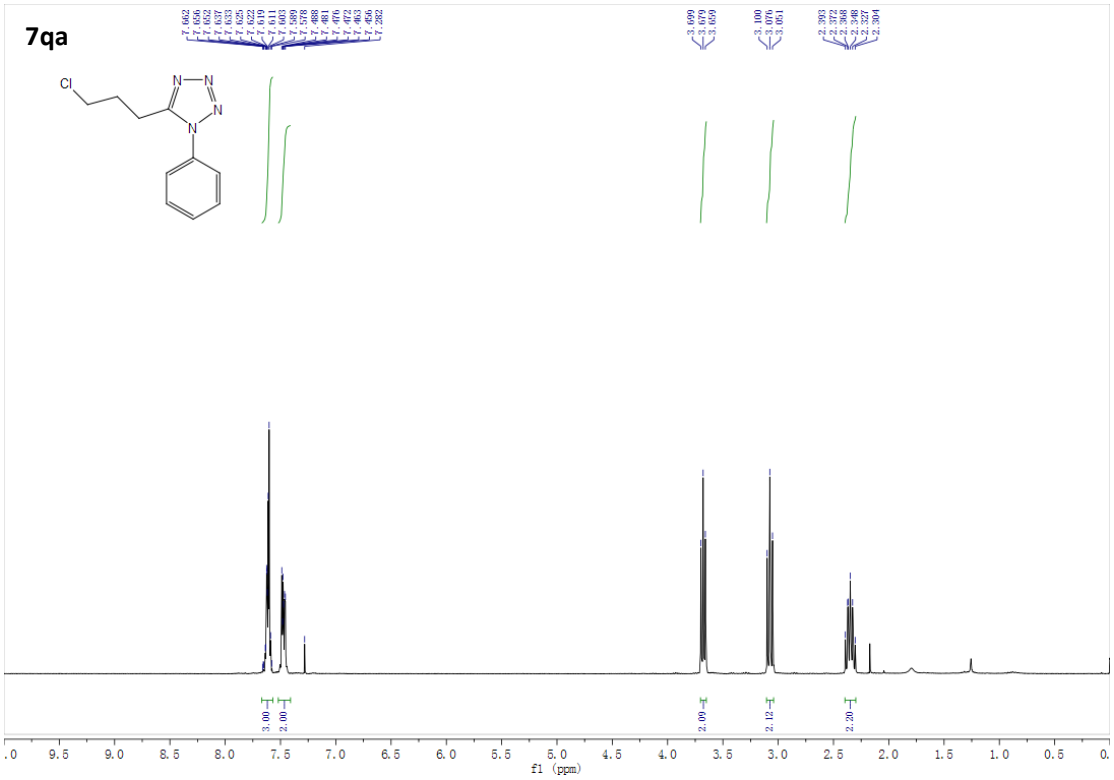

7na
152.24
141.25
134.62
133.67
130.42
129.98
128.91
127.60
125.08
107.40
77.32
77.00
76.68
f1 (ppm)

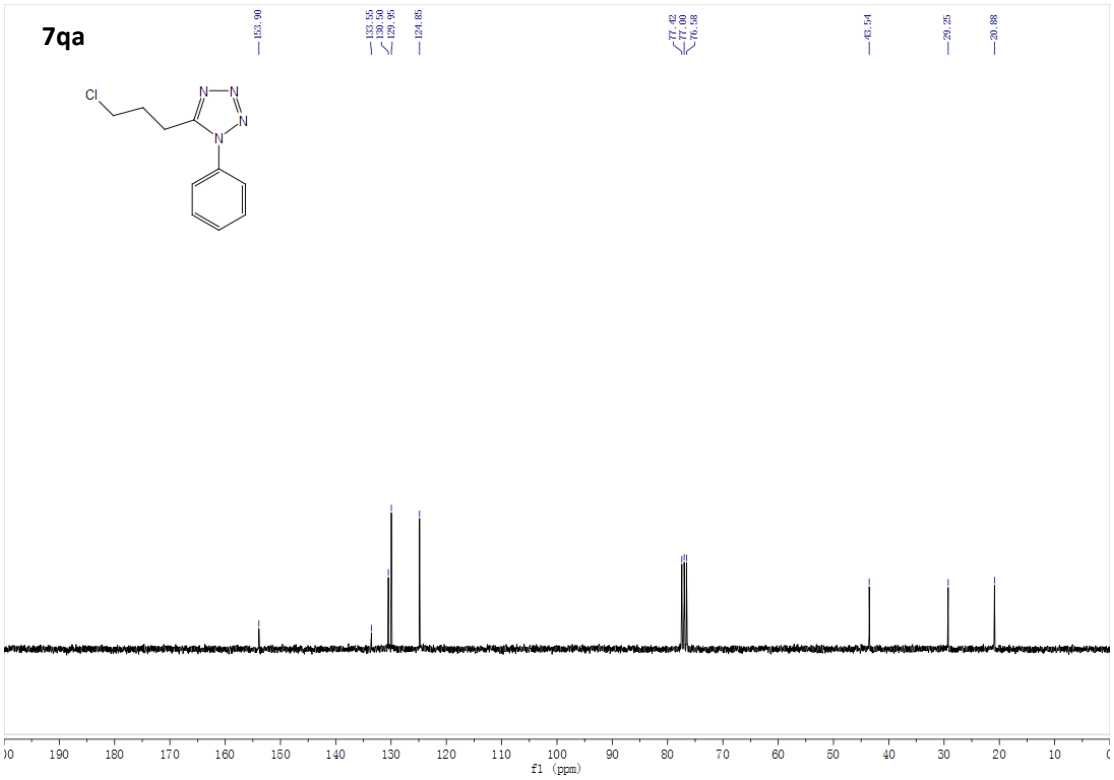

7oa
7.649
7.641
7.633
7.624
7.617
7.607
7.600
7.595
7.591
7.582
7.274
4.805
0.000
Cl
N
N
N
3.05
1.90
2.00
f1 (ppm)

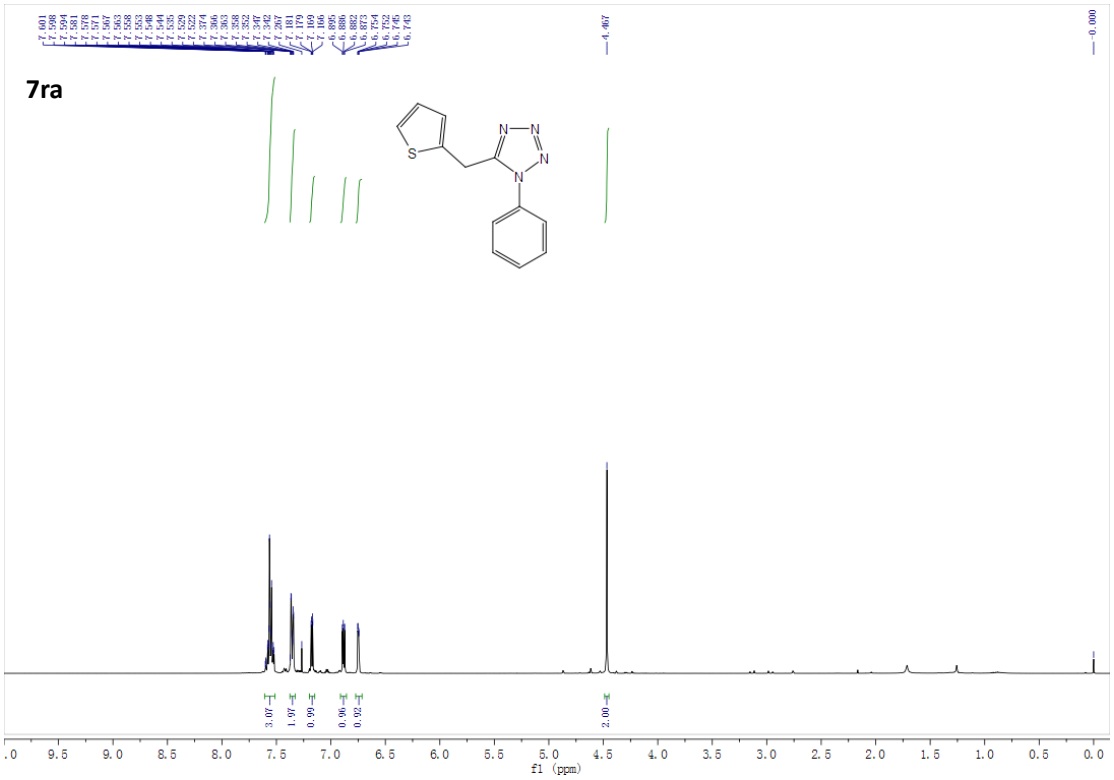

7oa
151.41
133.19
130.97
130.09
124.63
77.32
77.00
76.68
31.08
Cl
N
N
N
f1 (ppm)

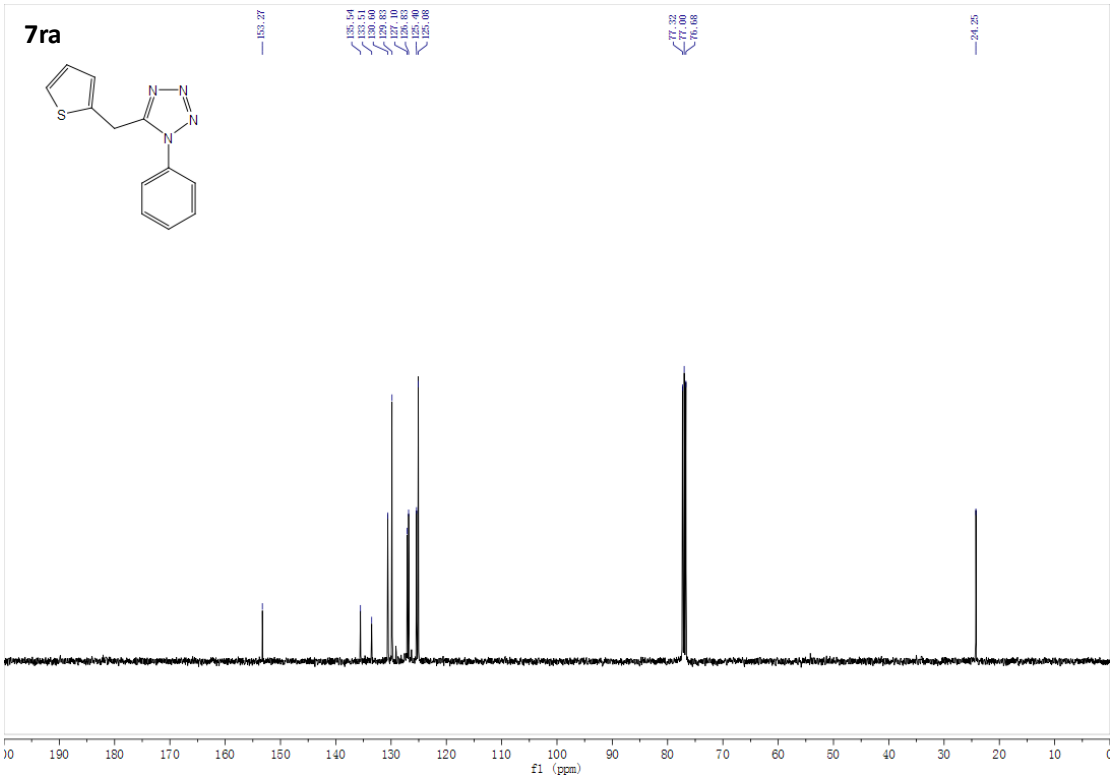

7pa
7.652
7.642
7.636
7.628
7.625
7.615
7.601
7.580
7.570
7.566
7.557
7.546
7.271
4.873
4.855
4.838
2.432
2.414
2.395
2.377
2.358
1.081
1.063
1.045
0.000
Cl
3.00
1.99
0.97
2.04
3.00
f1 (ppm)

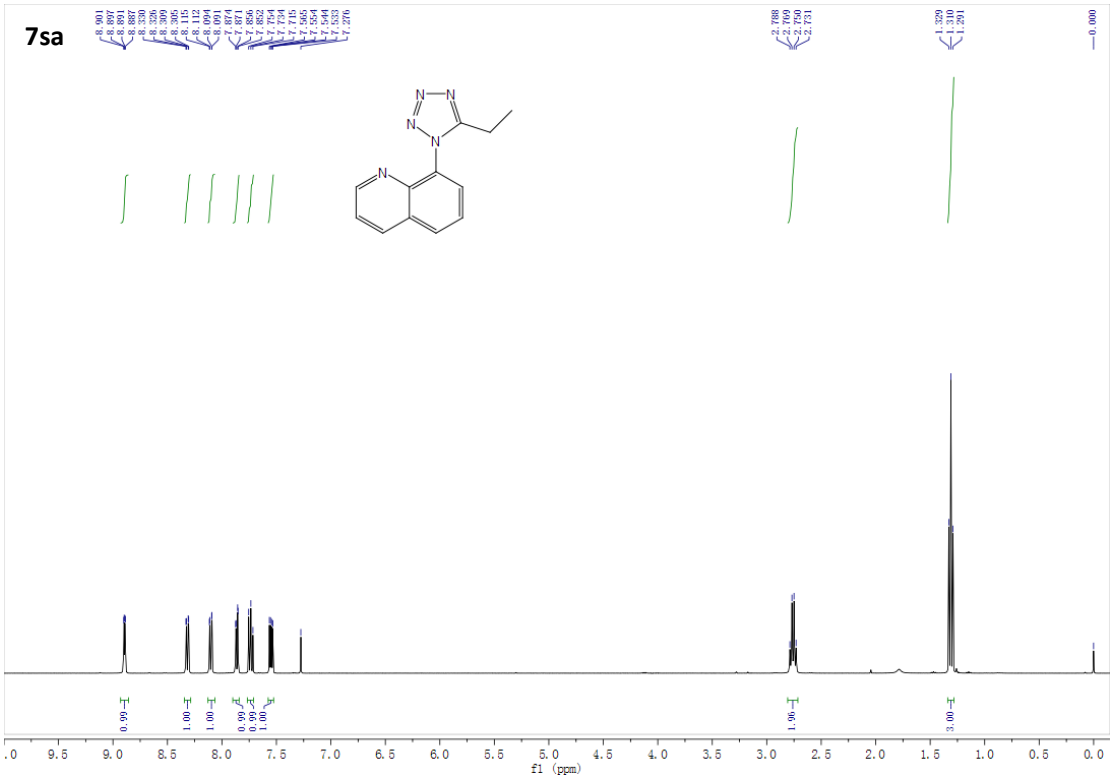

7pa
154.65
133.28
130.98
130.01
125.28
77.32
77.00
76.68
49.79
29.51
11.20
Cl
f1 (ppm)

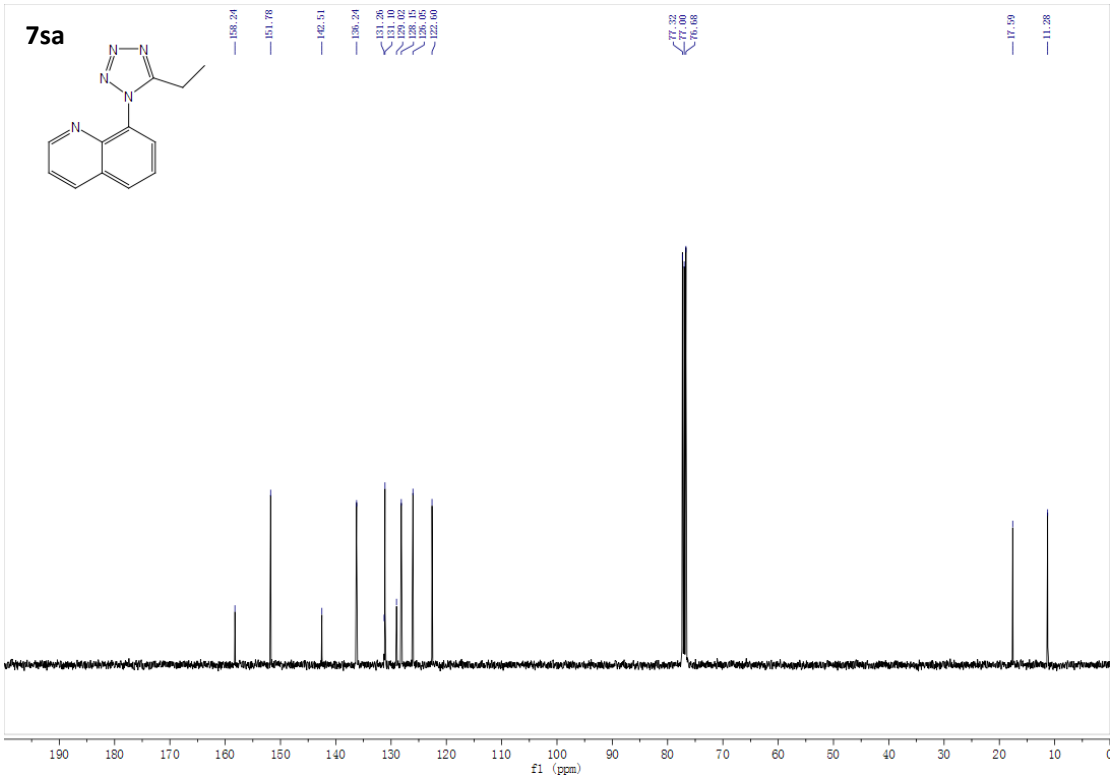

7qa
Cl
N
N
N
7.662
7.656
7.652
7.637
7.633
7.625
7.622
7.619
7.611
7.603
7.599
7.578
7.488
7.481
7.476
7.472
7.463
7.456
7.282
3.699
3.693
3.659
3.100
3.076
3.051
2.393
2.372
2.368
2.348
2.327
2.304
3.00
2.00
2.09
2.12
2.20
f1 (ppm)


7qa
Cl
N
N
N
153.90
133.55
130.50
129.95
124.85
77.42
77.00
76.58
63.54
29.25
20.88
f1 (ppm)


7ra
7.661
7.598
7.594
7.581
7.578
7.571
7.567
7.563
7.558
7.553
7.548
7.544
7.535
7.529
7.522
7.374
7.366
7.363
7.358
7.352
7.347
7.342
7.297
7.181
7.179
7.169
7.166
6.895
6.886
6.882
6.877
6.754
6.752
6.745
6.743
4.467
0.000
3.07
1.97
0.99
0.96
0.92
2.00
f1 (ppm)


7ra
153.27
135.54
133.51
130.60
129.83
127.10
126.83
125.40
125.08
77.32
77.00
76.68
24.25
f1 (ppm)


7sa
8.901
8.897
8.891
8.887
8.339
8.330
8.300
8.305
8.115
8.112
8.094
8.091
7.874
7.867
7.856
7.852
7.754
7.734
7.715
7.565
7.554
7.544
7.535
7.276
2.788
2.769
2.750
2.731
1.329
1.310
1.291
0.000
0.99
1.00
1.00
0.99
0.99
1.00
1.96
3.00
f1 (ppm)


7sa
158.24
151.78
142.51
136.24
131.26
131.10
129.02
128.45
126.95
122.60
77.32
77.00
76.68
17.59
11.28
f1 (ppm)

References

[1] J. Deruiter, B. E. Swearingen, V. Wandrekar and C. A. Mayfield, *J. Med. Chem.*, **1989**, 32, 1033.

[2] Jesus M. Aizpurua and Claudio Palomo, *Tetrahedron Letters*., **1985**, 26, 6113.