Pyridine-Phosphinimine Ligands-Accelerated Cu(I)-Catalyzed Azide-Alkyne Cycloaddition for Preparation of 1-(Pyridin-2-yl)-1,2,3-Triazole Derivatives

Supporting Information

Ranfeng Sun, Huangdong Wang, Jianfeng Hu, Jiudong Zhao and Hao Zhang*

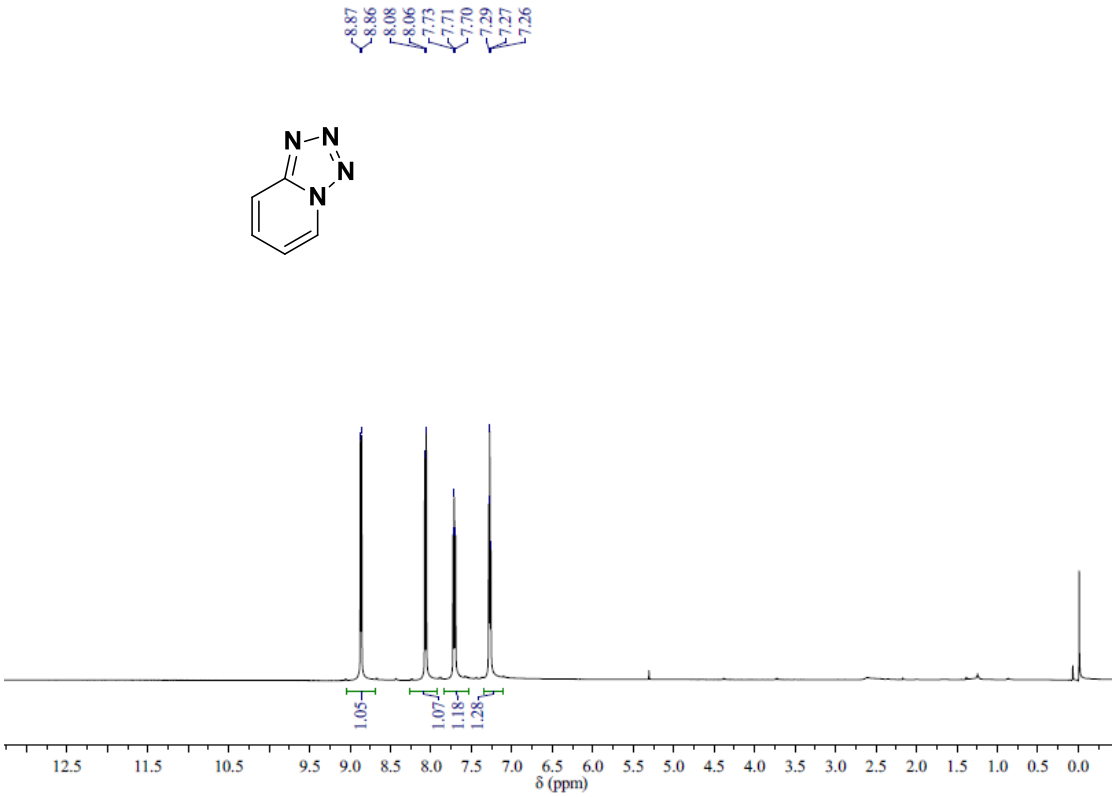
College of Chemistry and Chemical Engineering, Inner Mongolia University, Hohhot 010021, P. R. China

* Corresponding author. haozhang@imu.edu.cn; zh_hjf@hotmail.com.

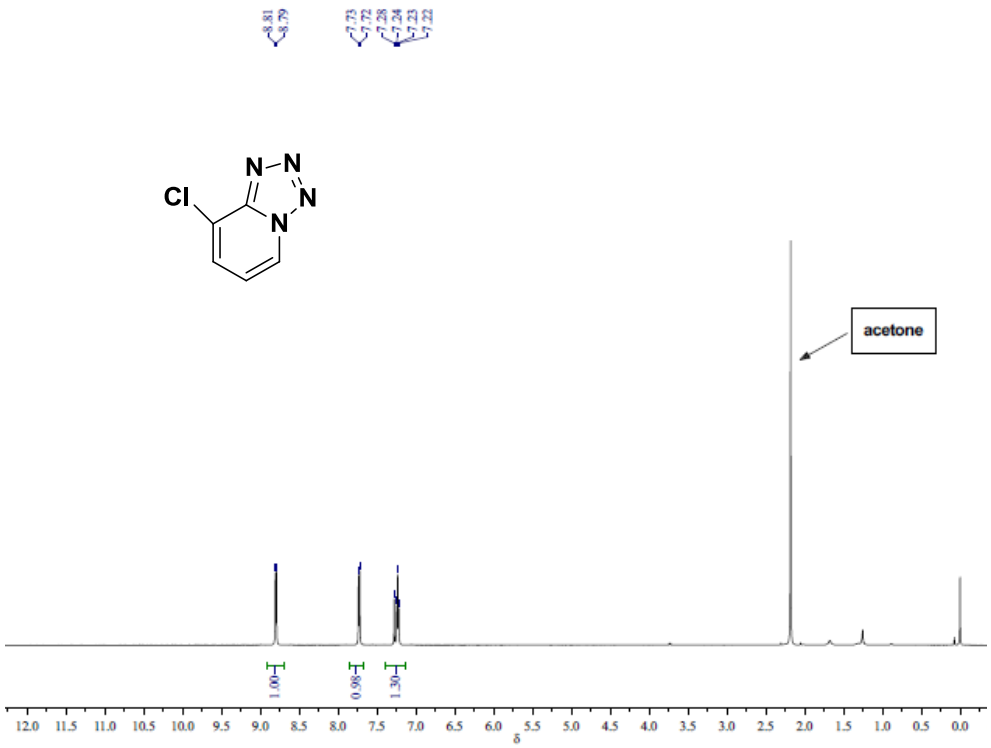
Table of Contents

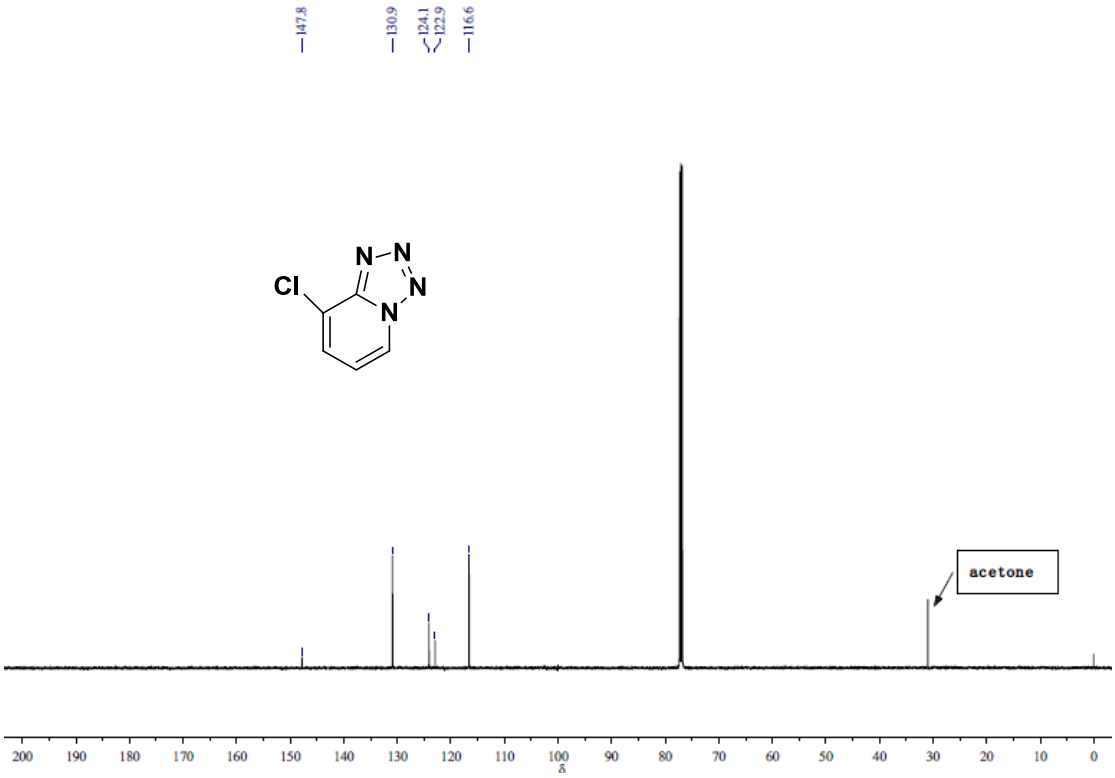
^{1}H, ^{13}C and ^{31}P NMR spectra for all compounds.

Tetrazolo[1,5-*a*]pyridine (2a)

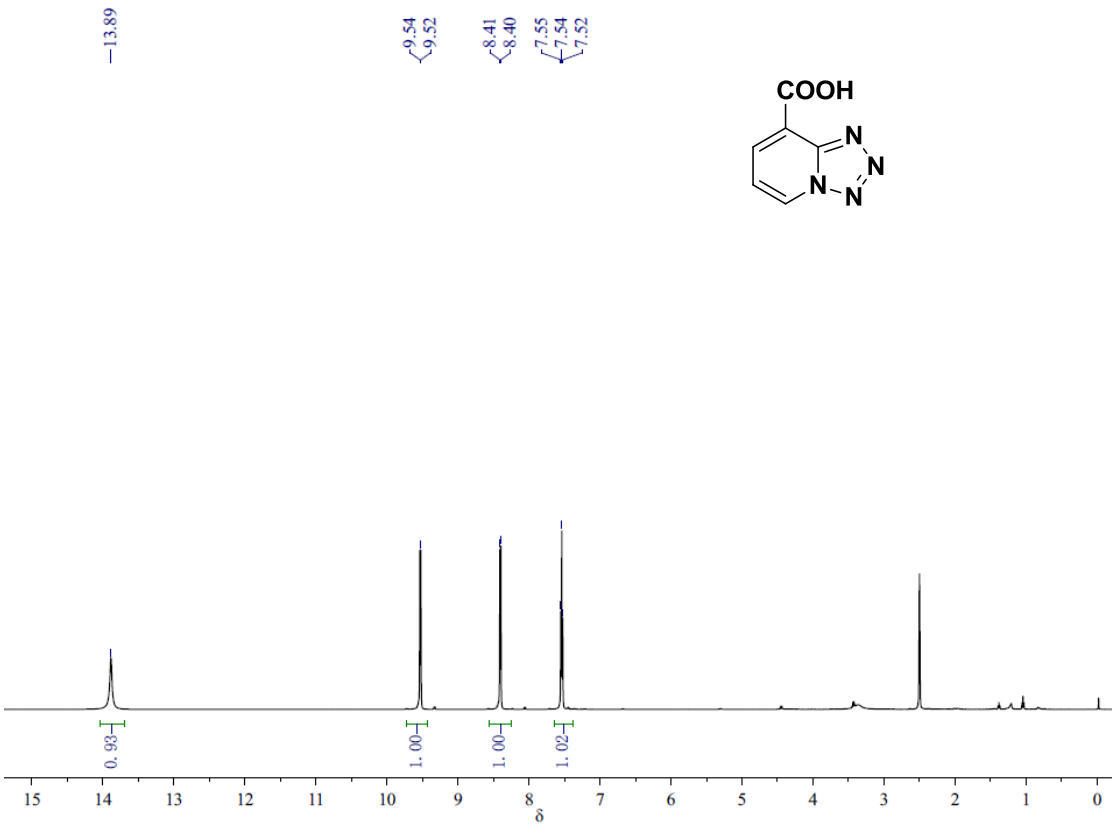


8-Chlorotetrazolo[1,5-*a*]pyridine (2b).

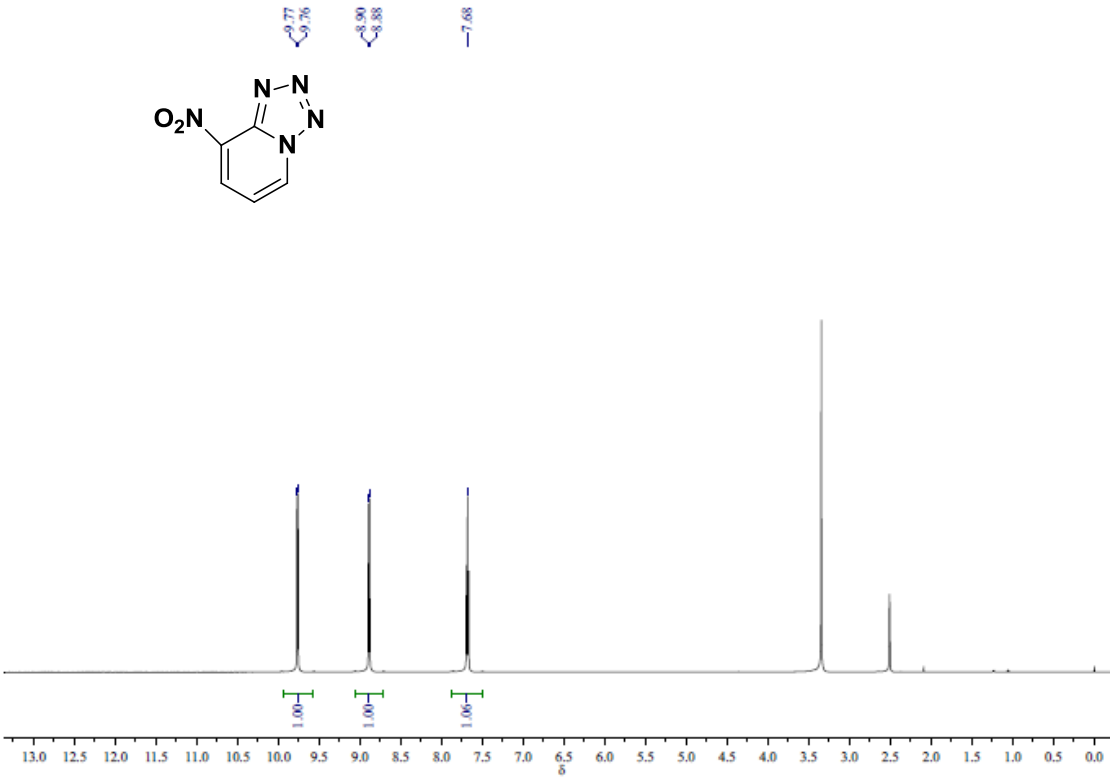


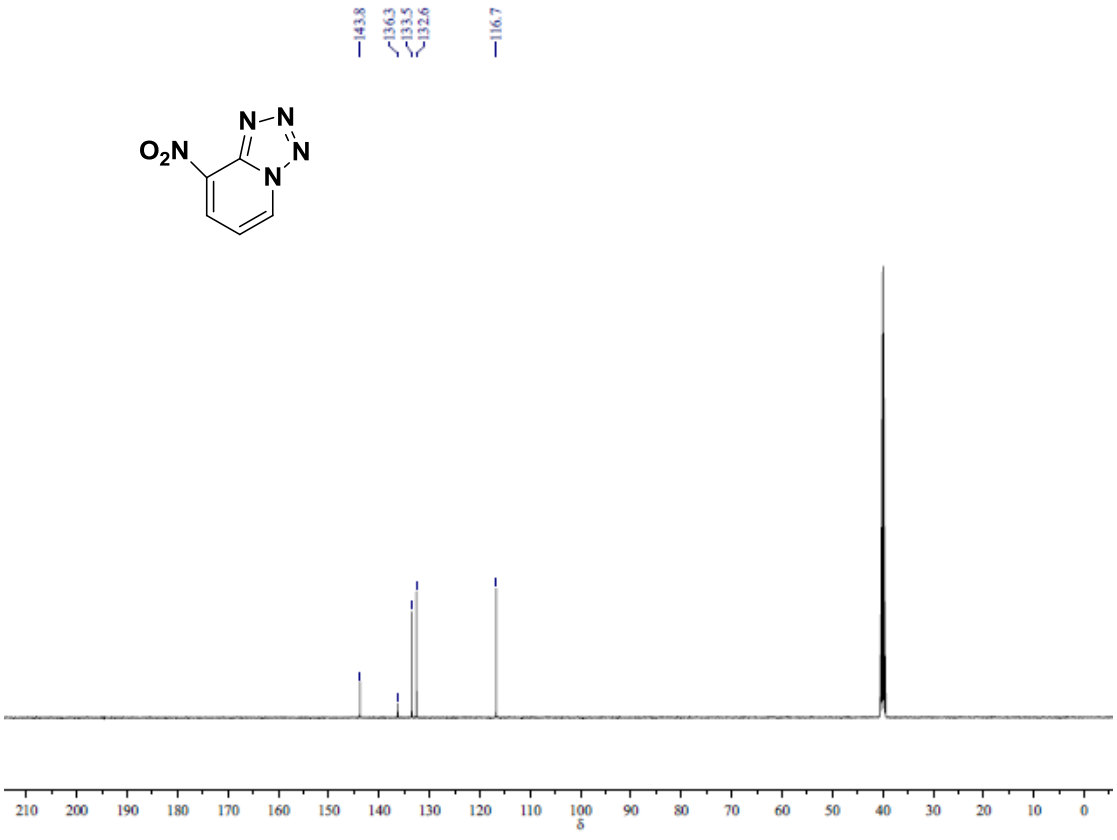


Tetrazolo[1,5-*a*]pyridine-8-carboxylic acid (2c).

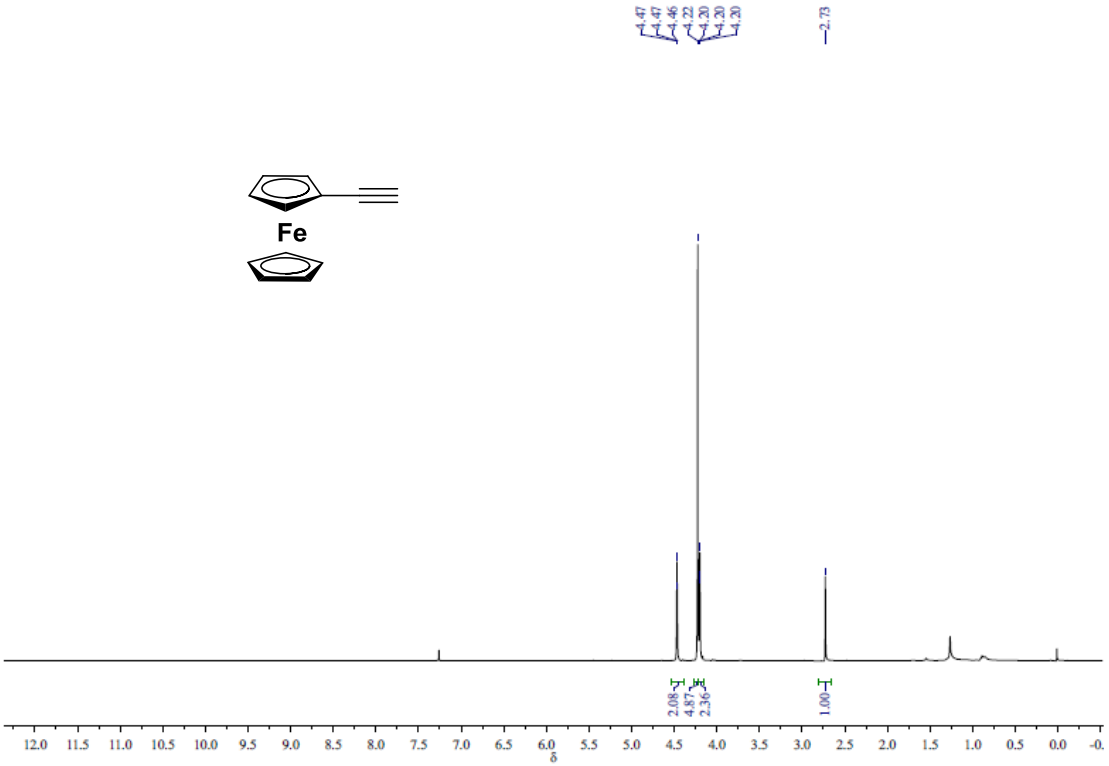


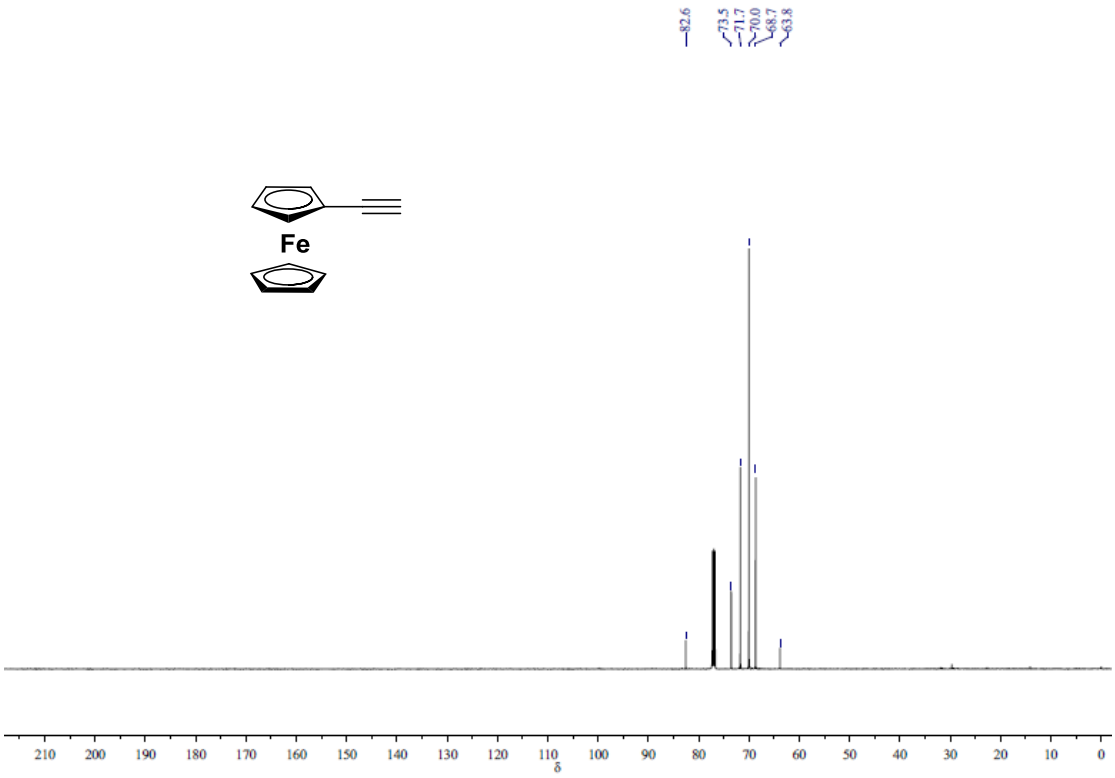
8-Nitrotetrazolo[1,5-*a*]pyridine (2d).



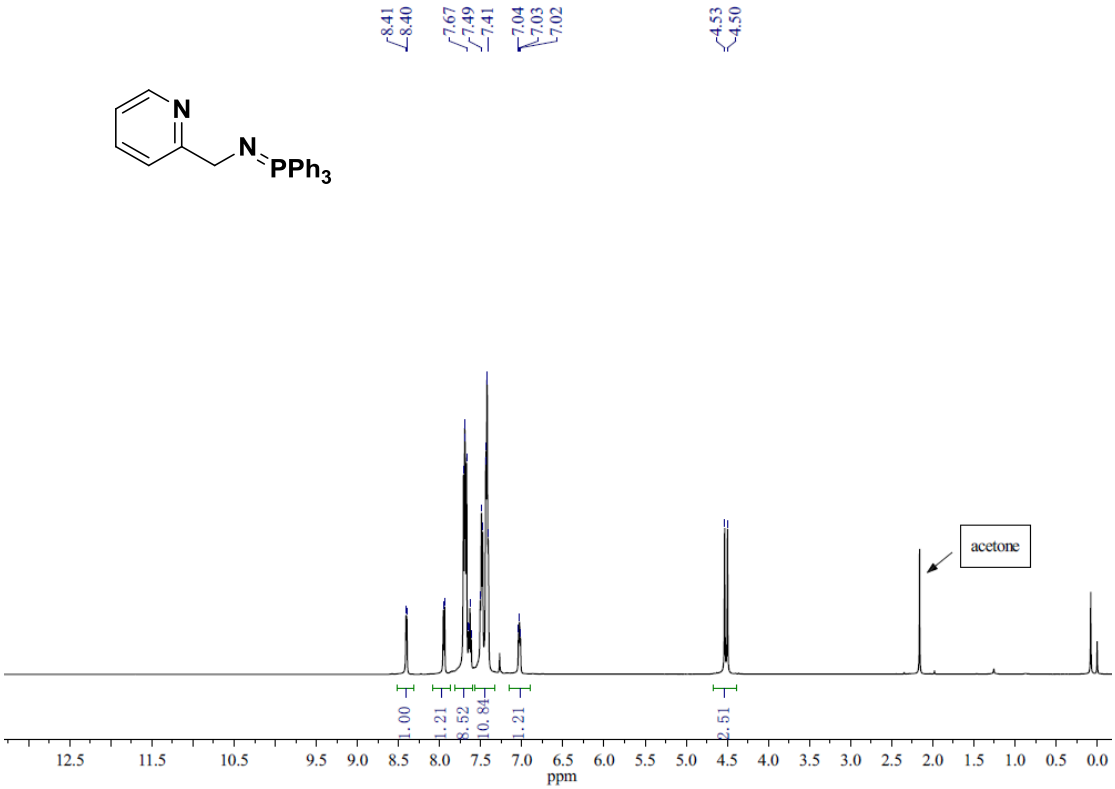


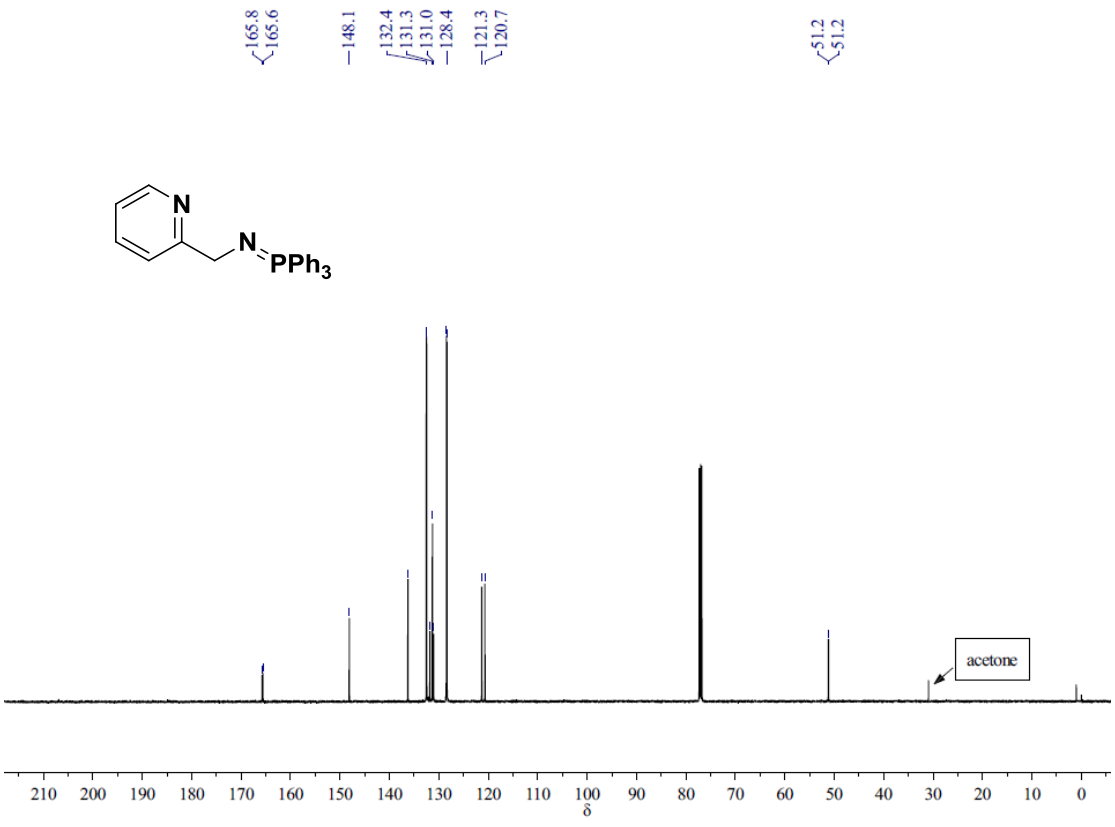
Ferrocenyl acetylene (1a).

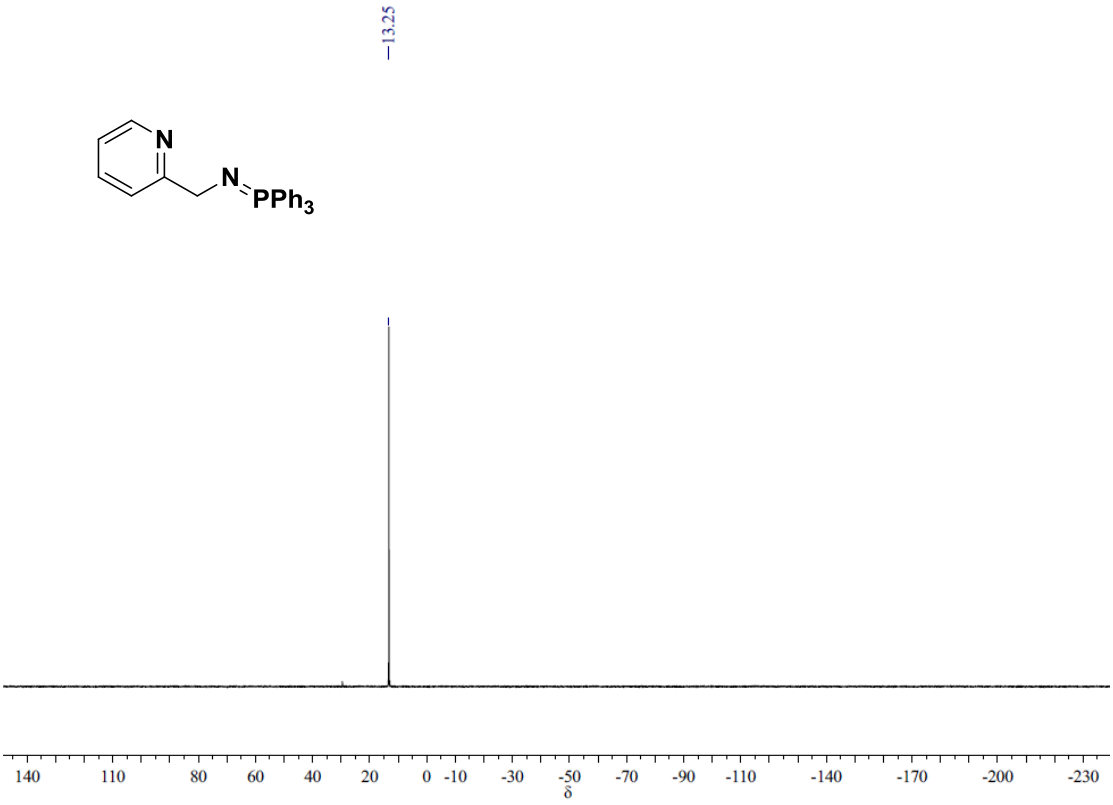




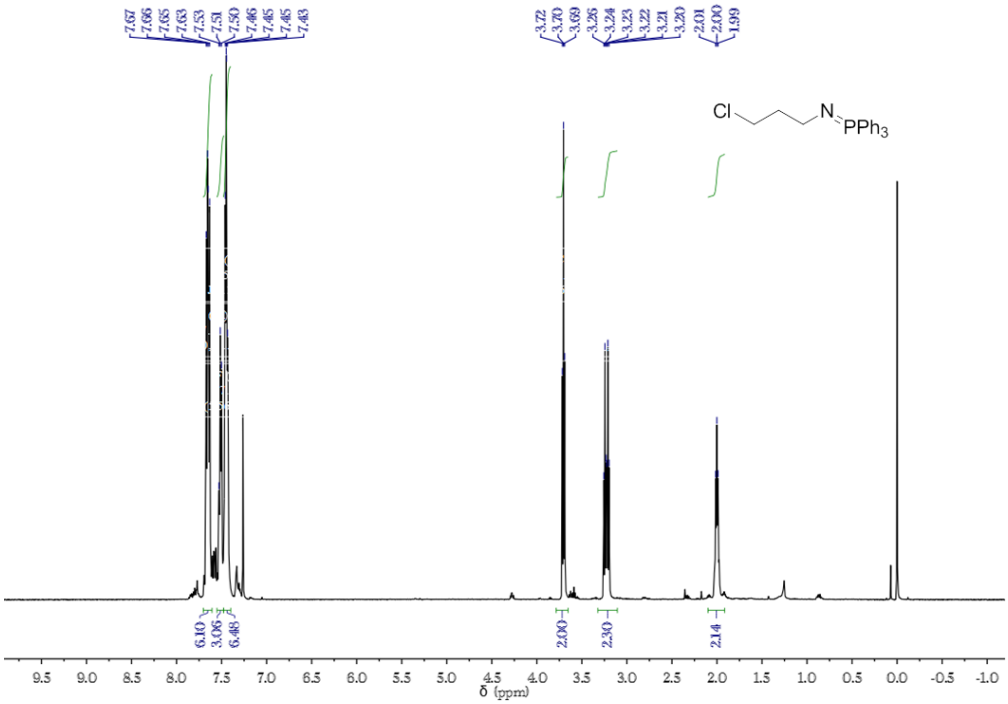
(2-Pyridyl)-CH_2-N=PPh_3 (L_1).







3-Chloro-*N*-(triphenylphosphoranylidene)propan-1-amine (L_2).



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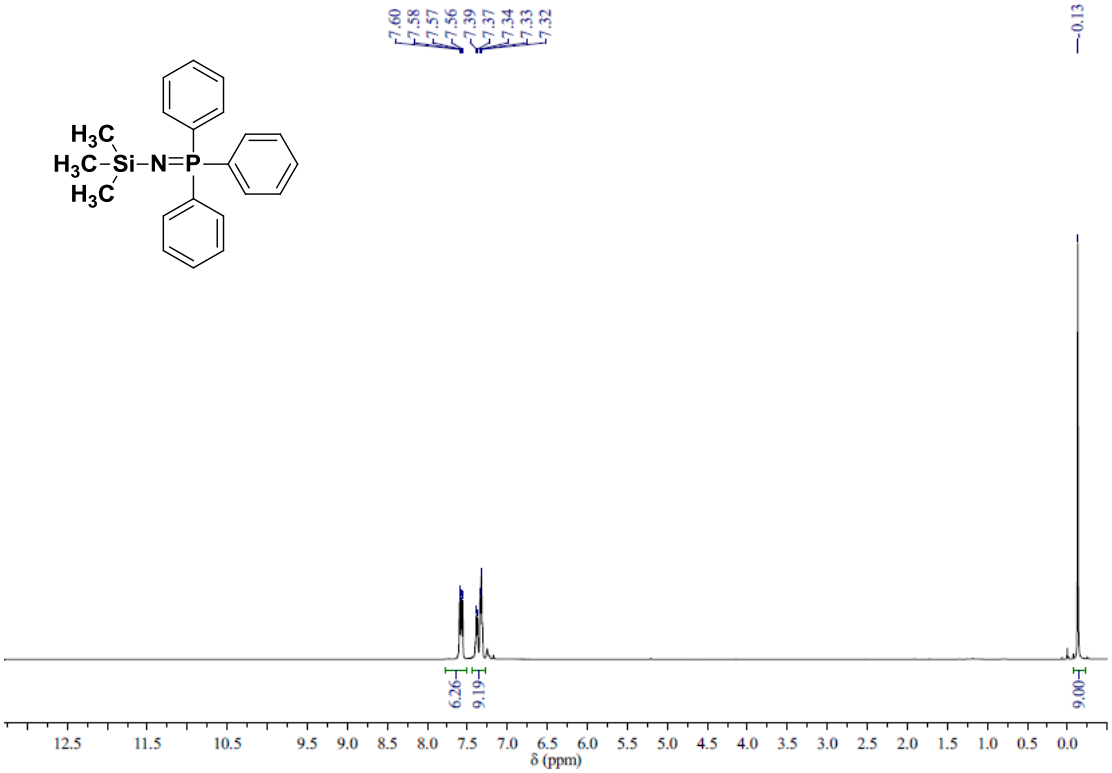
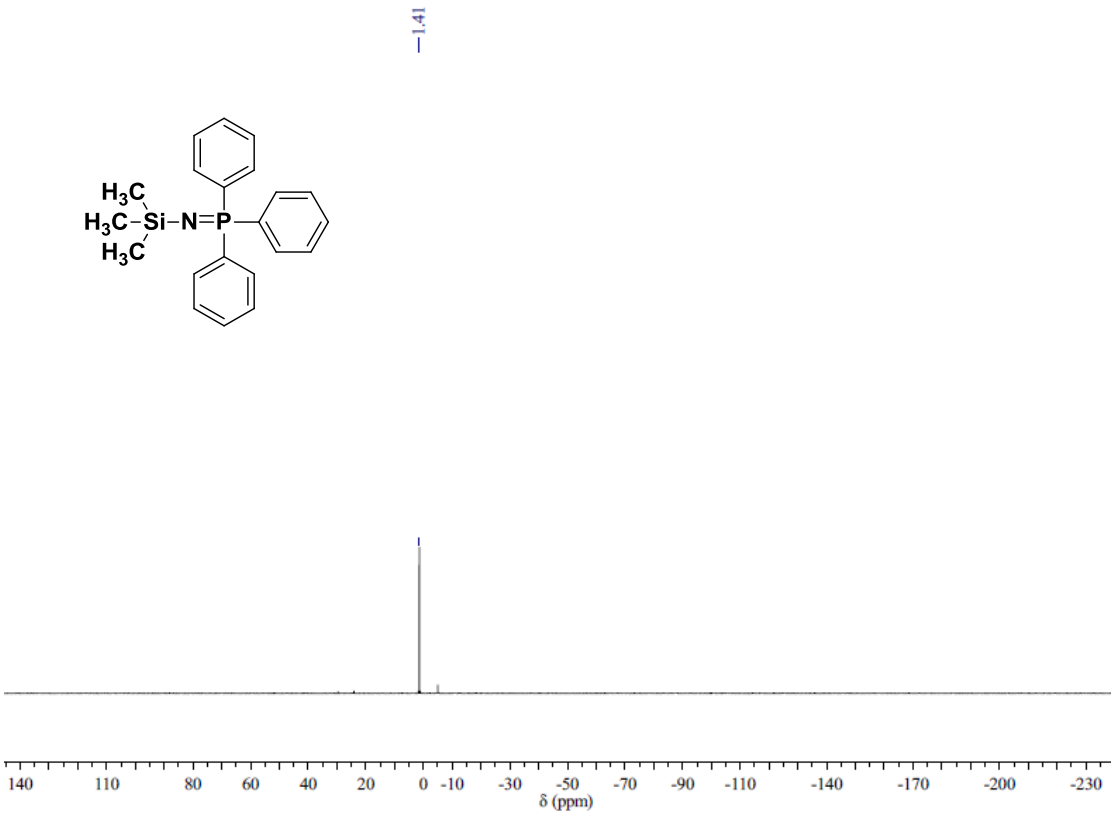
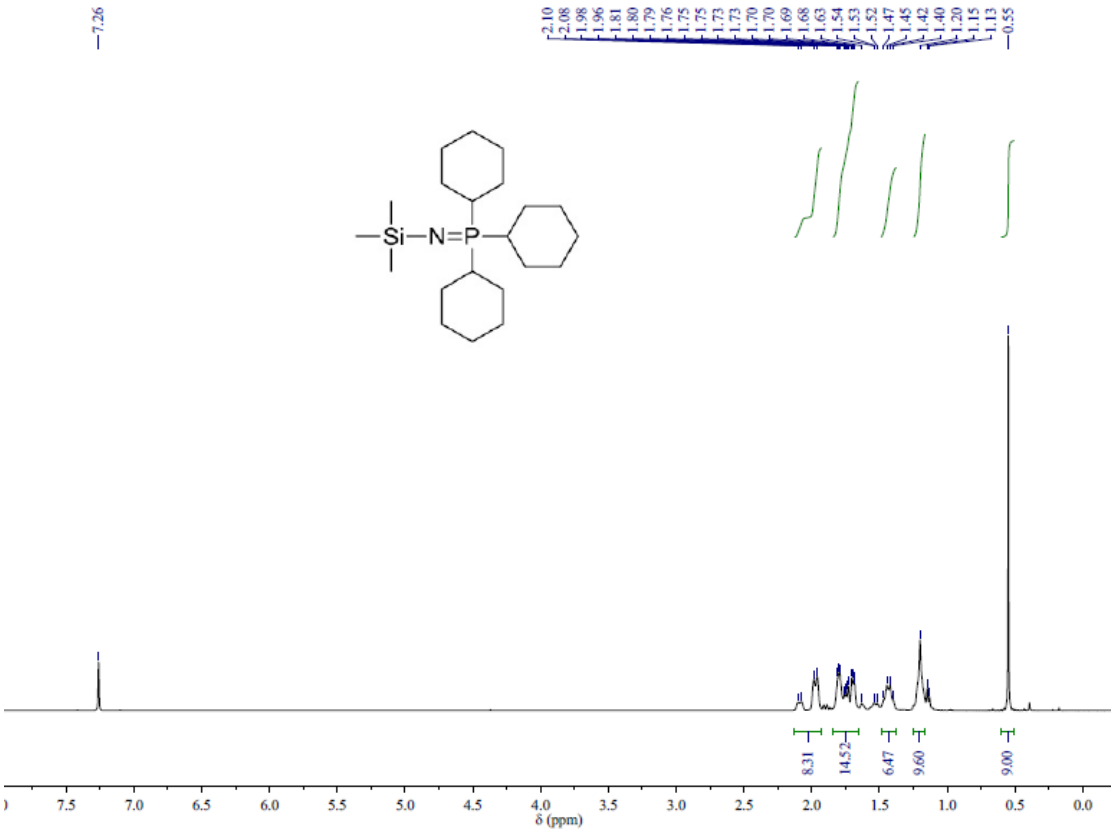
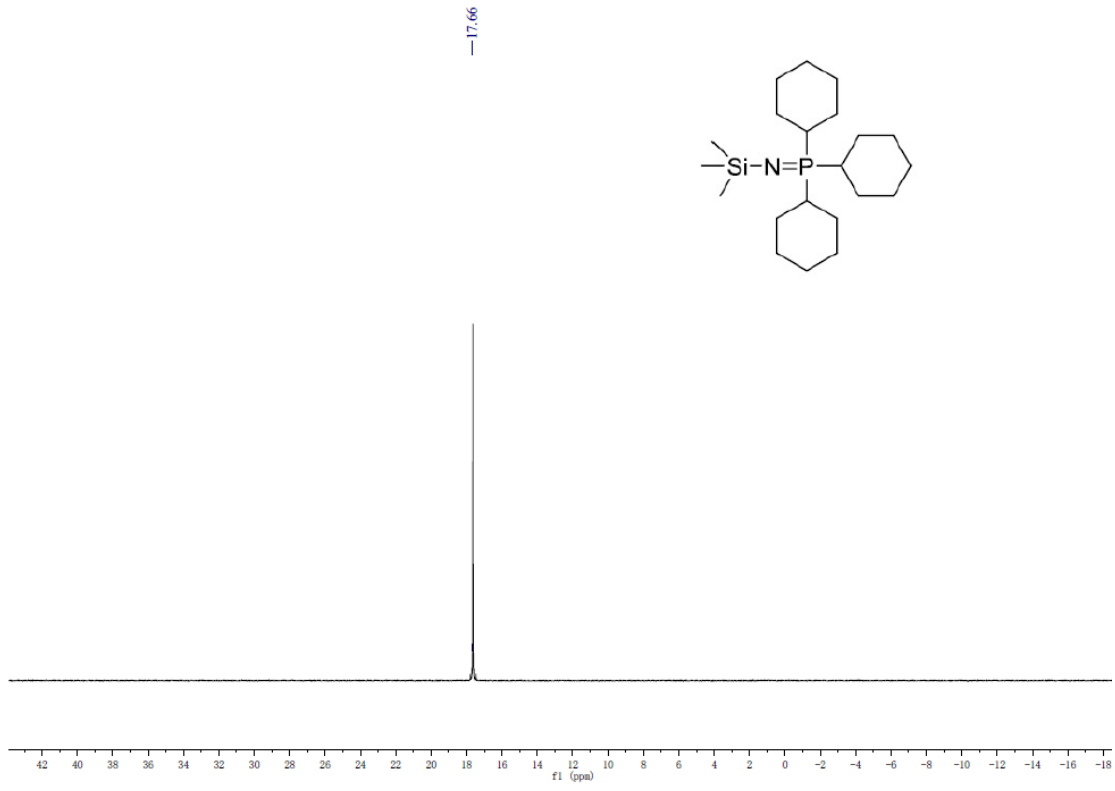


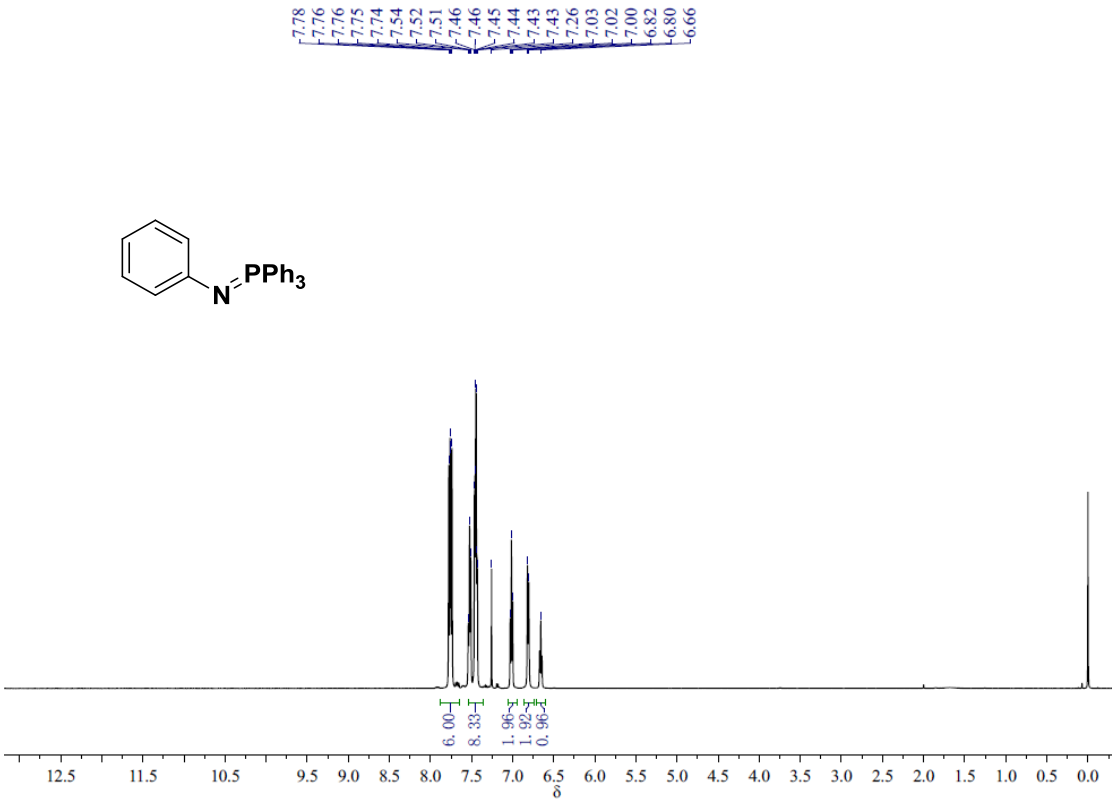


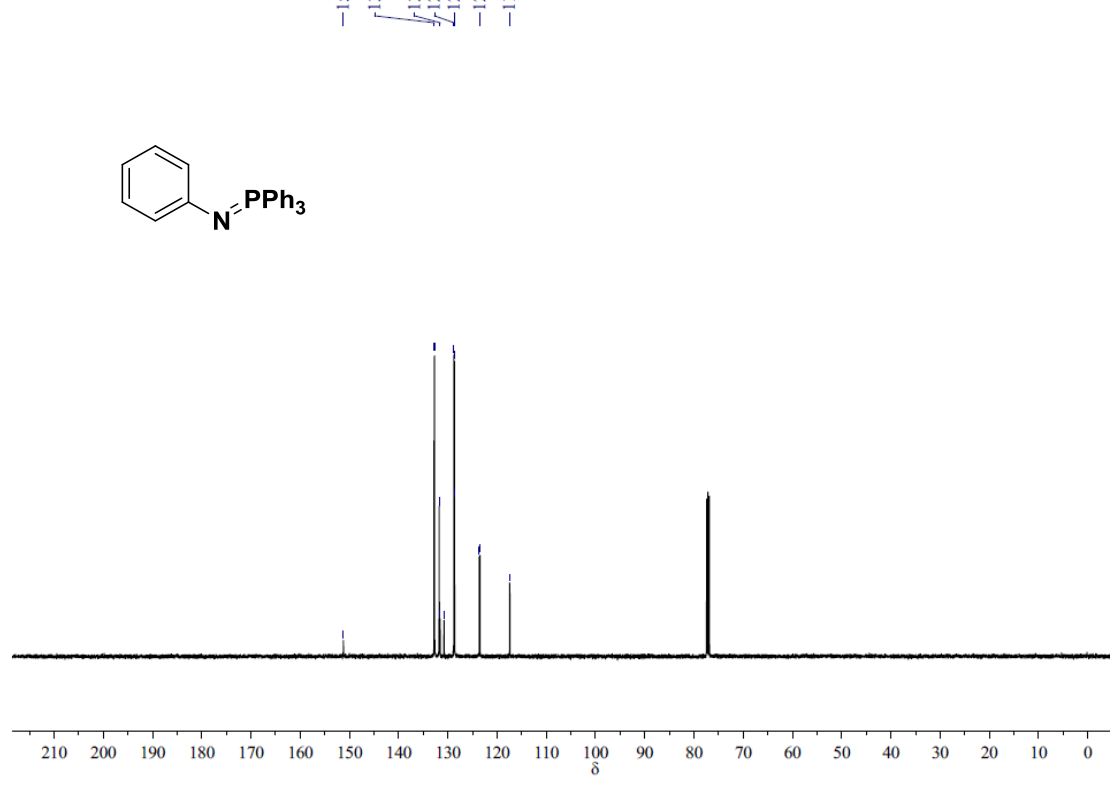
1,1,1-Trimethyl-*N*-(tricyclohexylphosphoranylidene)silanamine (L_4).



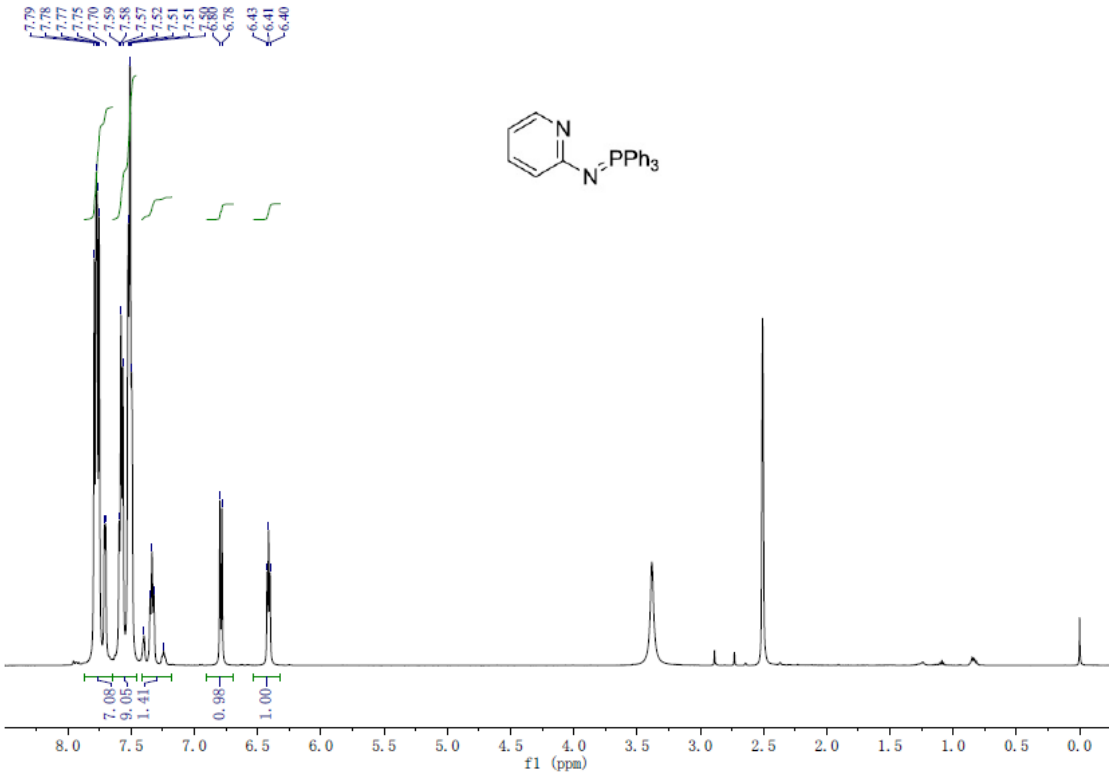


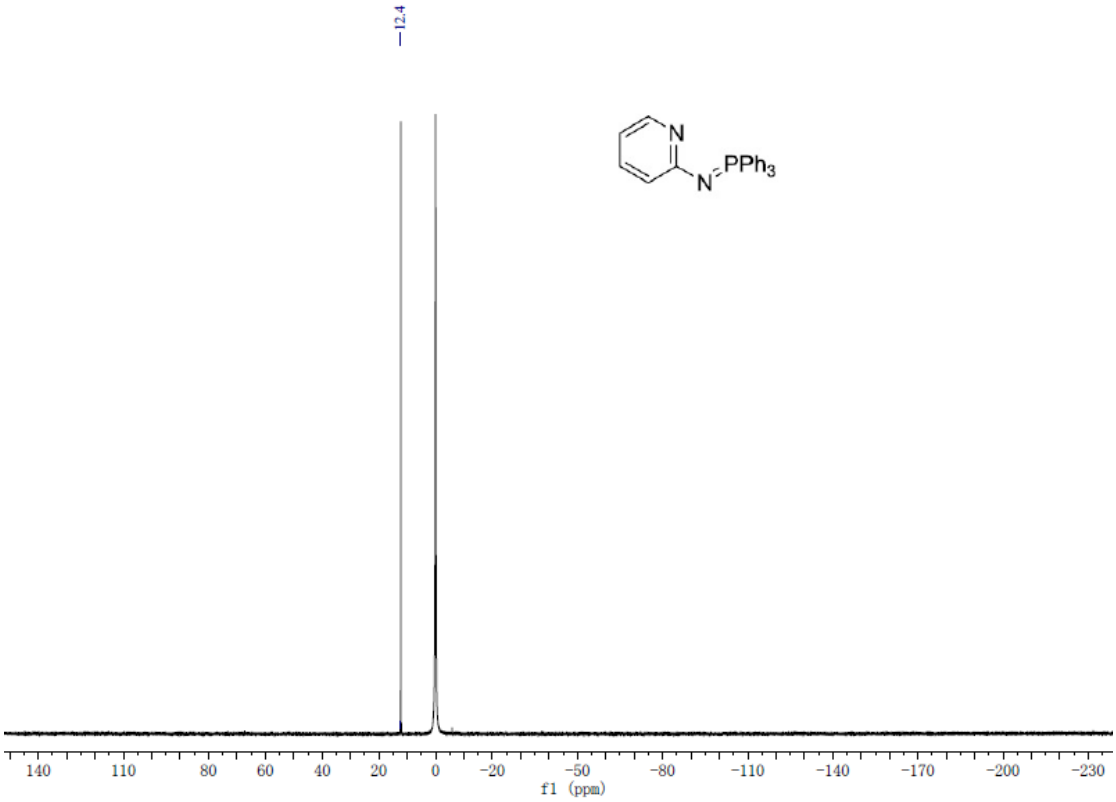
***N*-(triphenylphosphoranylidene)aniline (L_5).**



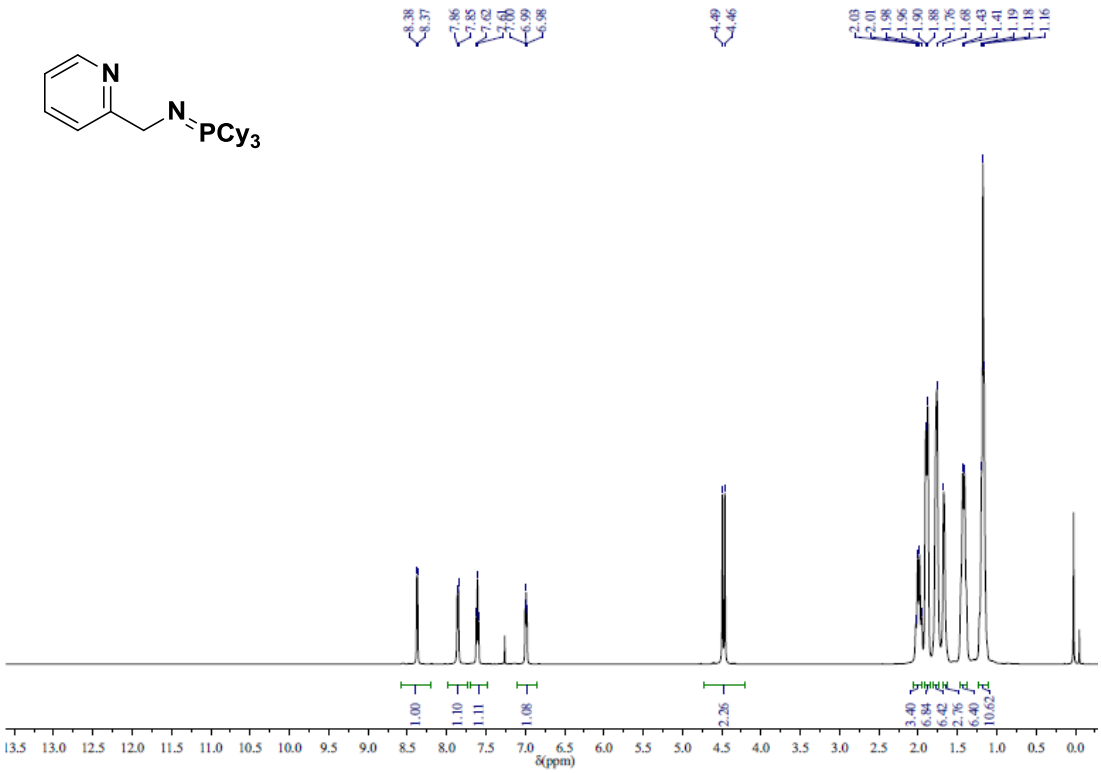


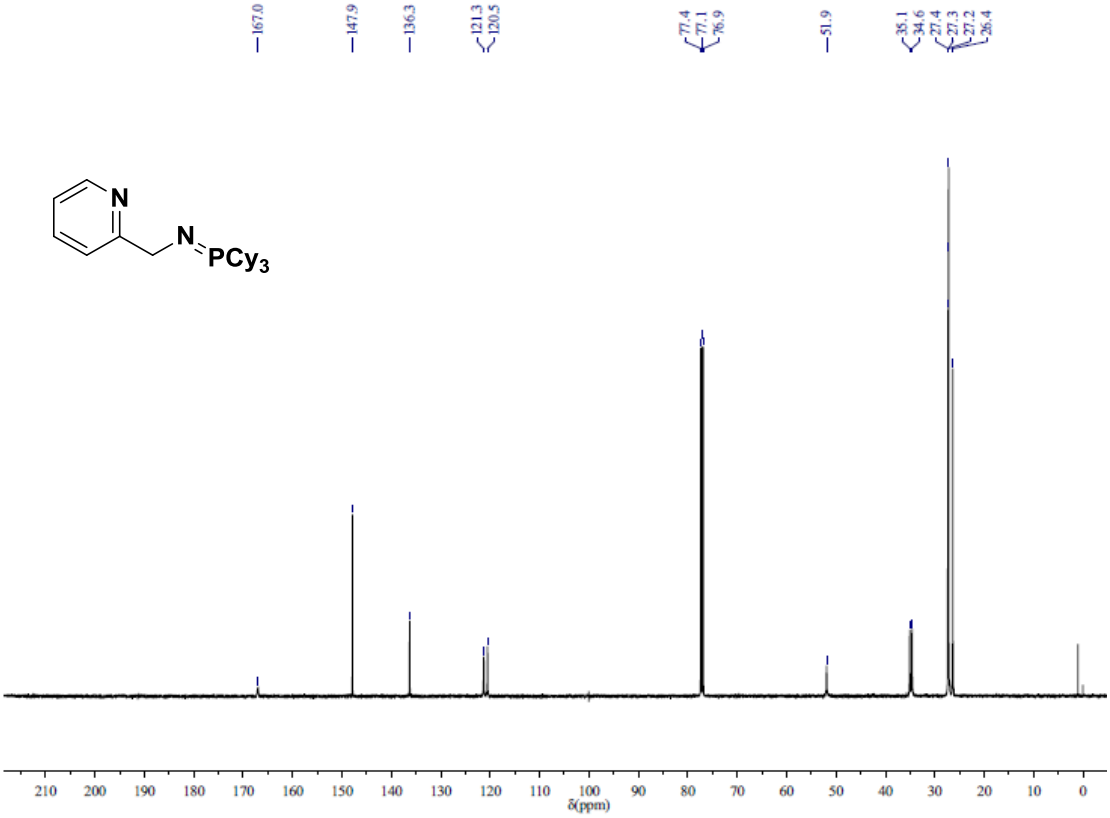
***N*-(triphenylphosphoranylidene)pyridin-2-amine (L_6).**

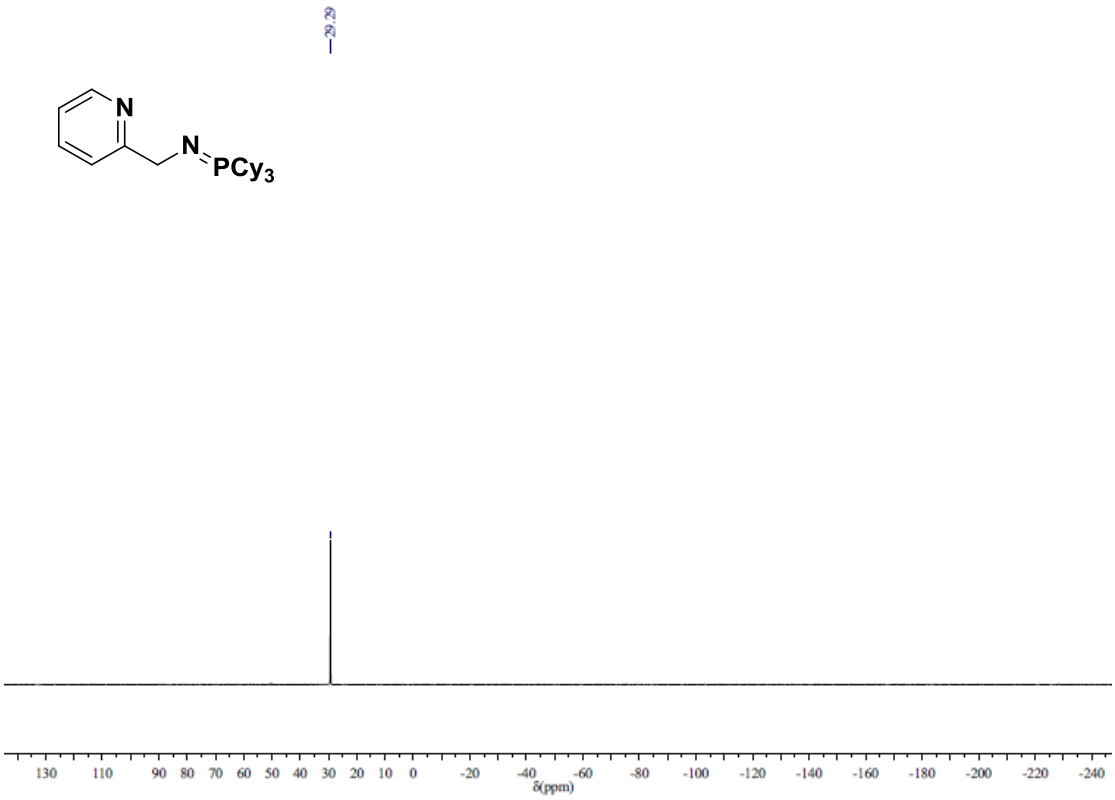




(2-Pyridyl)-CH_2-N=PCy_3 (L_7).







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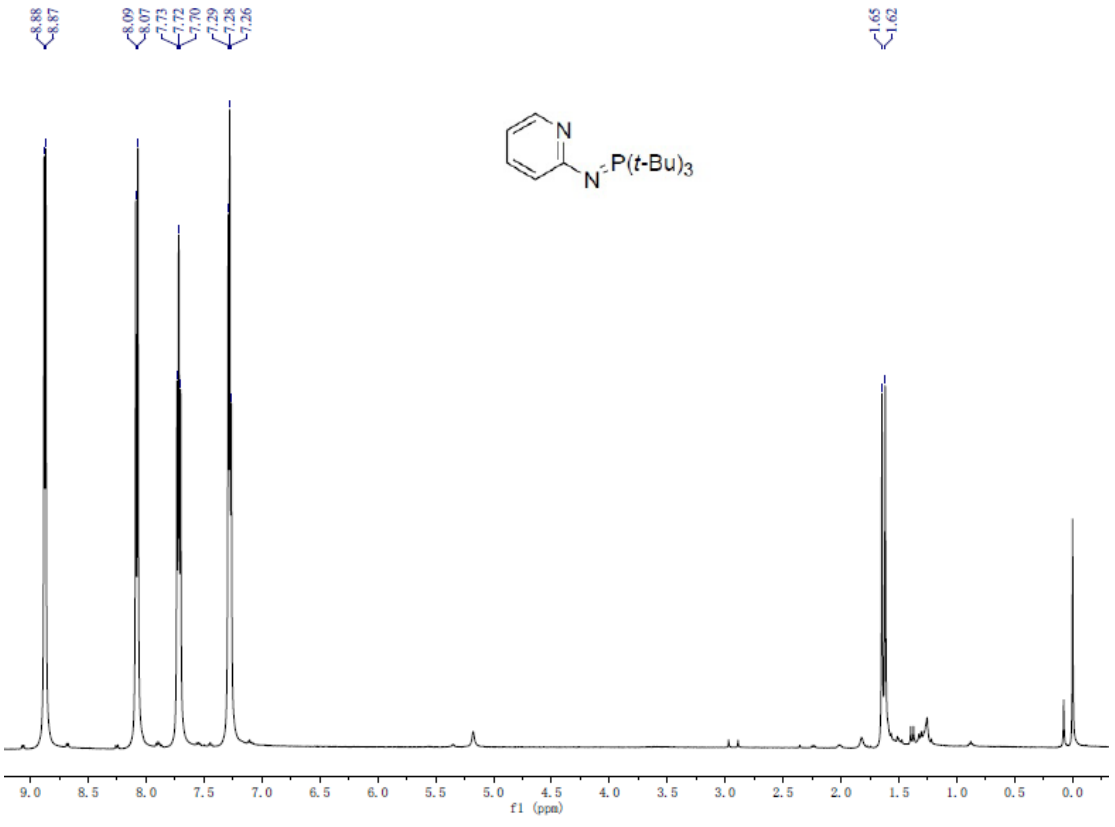
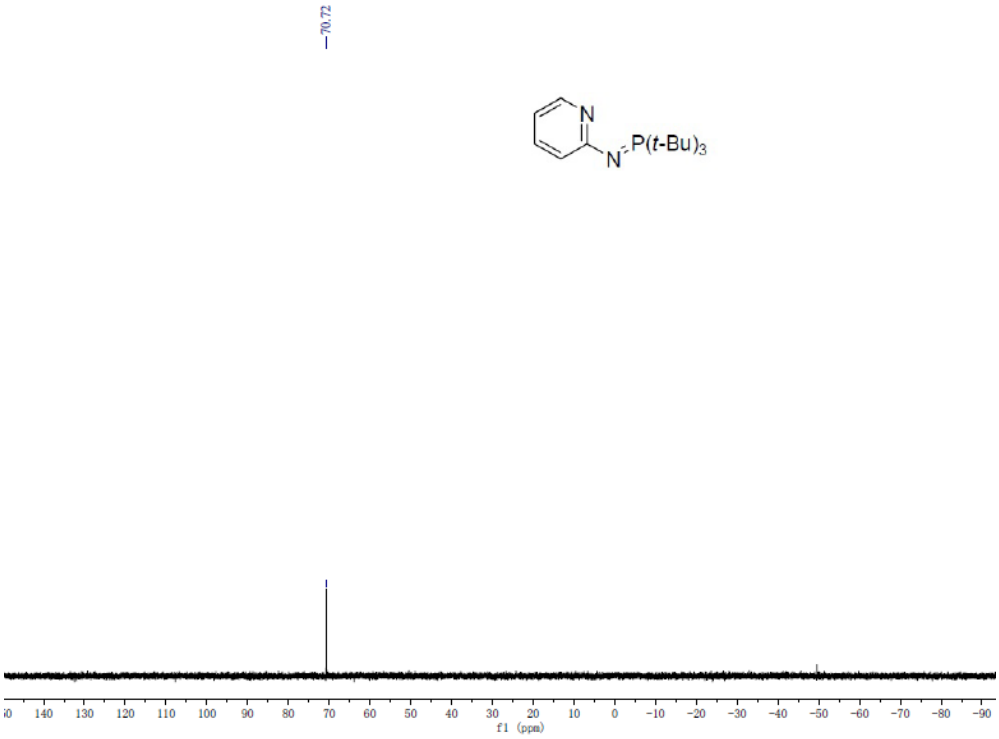
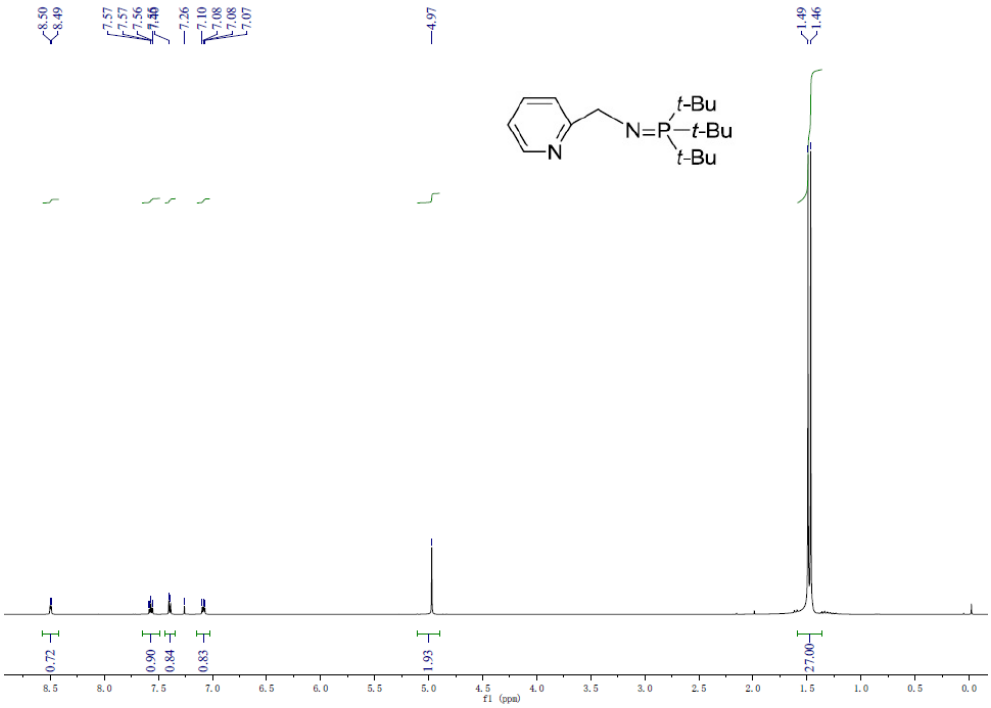
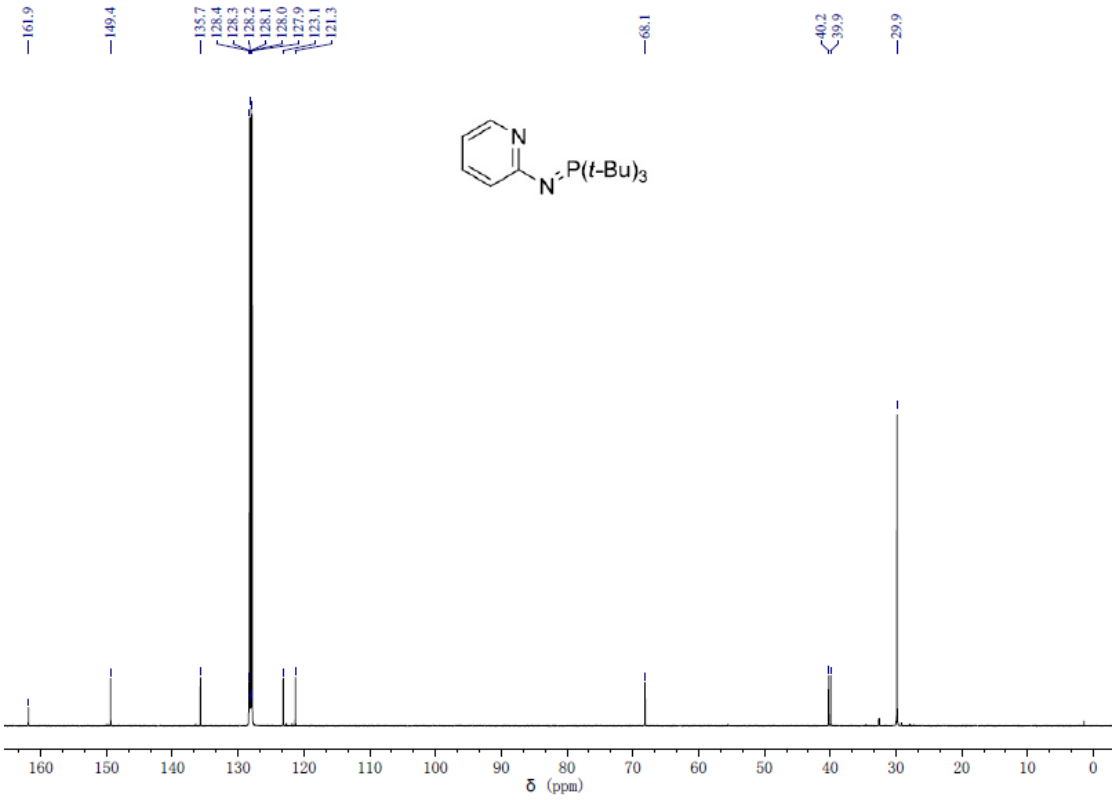




(2-Pyridyl)-CH_2-N=P^tBu_3 (L_9).

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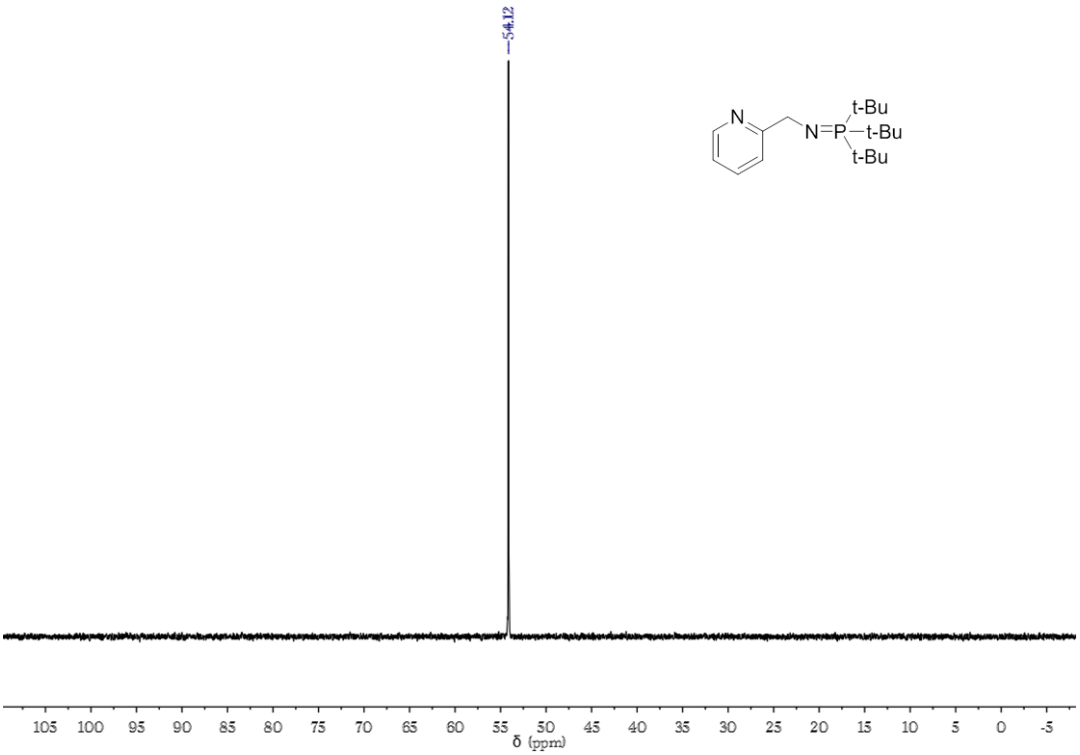
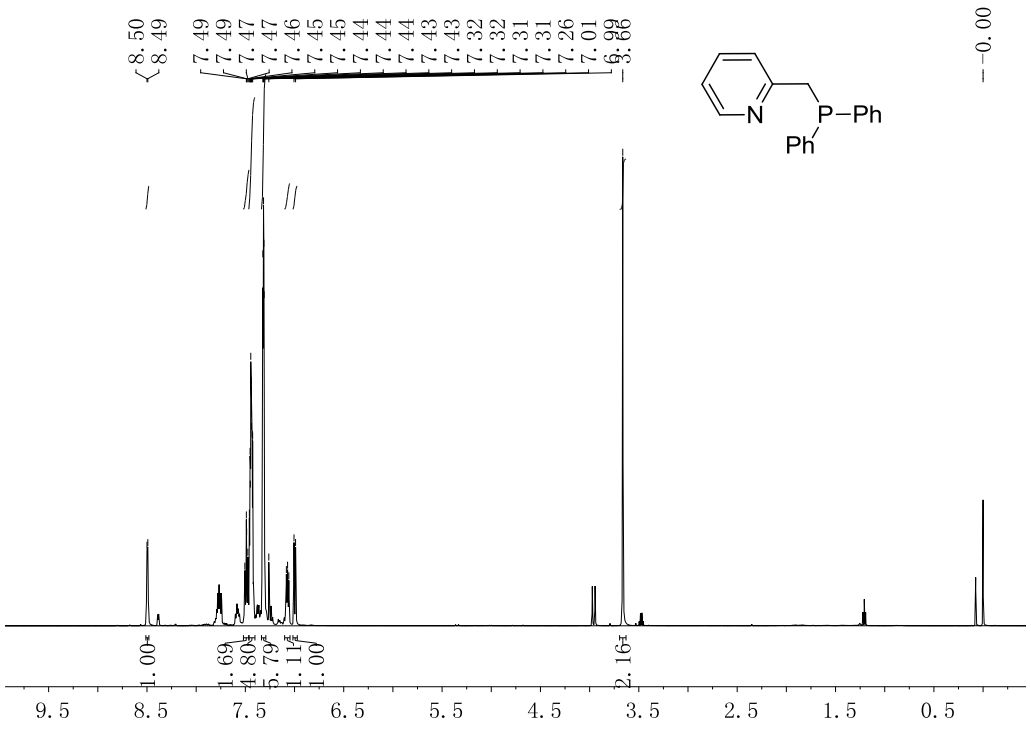
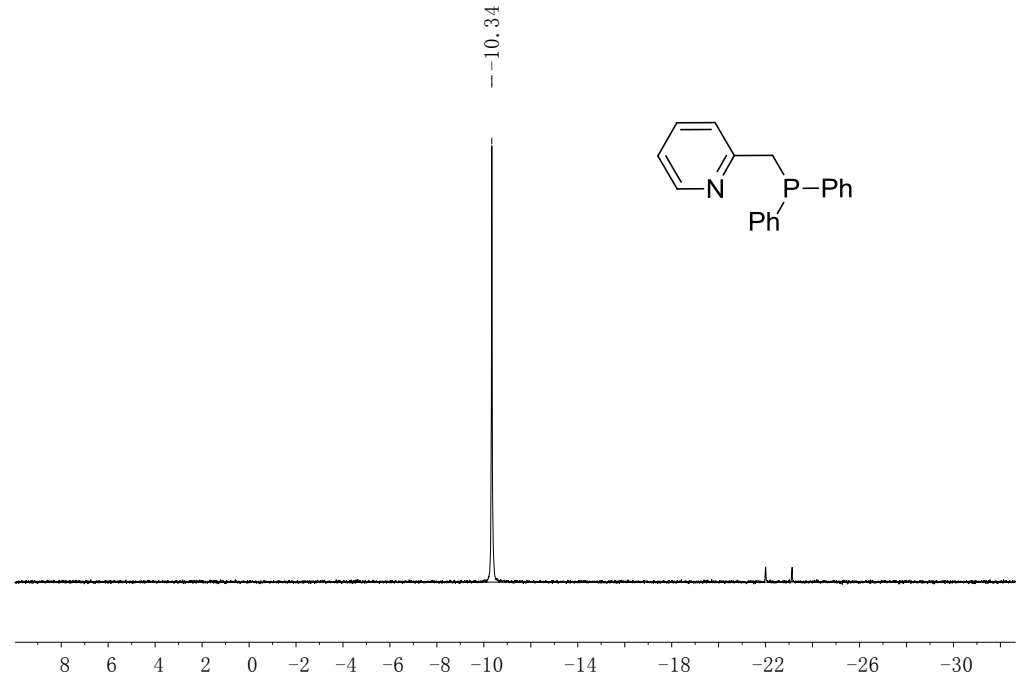
149.4
135.7
128.4
128.3
128.2
128.1
128.0
127.9
123.1
121.3
68.1
40.2
39.9
29.9
N
N
P(t-Bu)3
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)


54.12
N
N=P
t-Bu
t-Bu
t-Bu
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
δ (ppm)

2-((Diphenylphosphino)methyl)pyridine (L_{10}).





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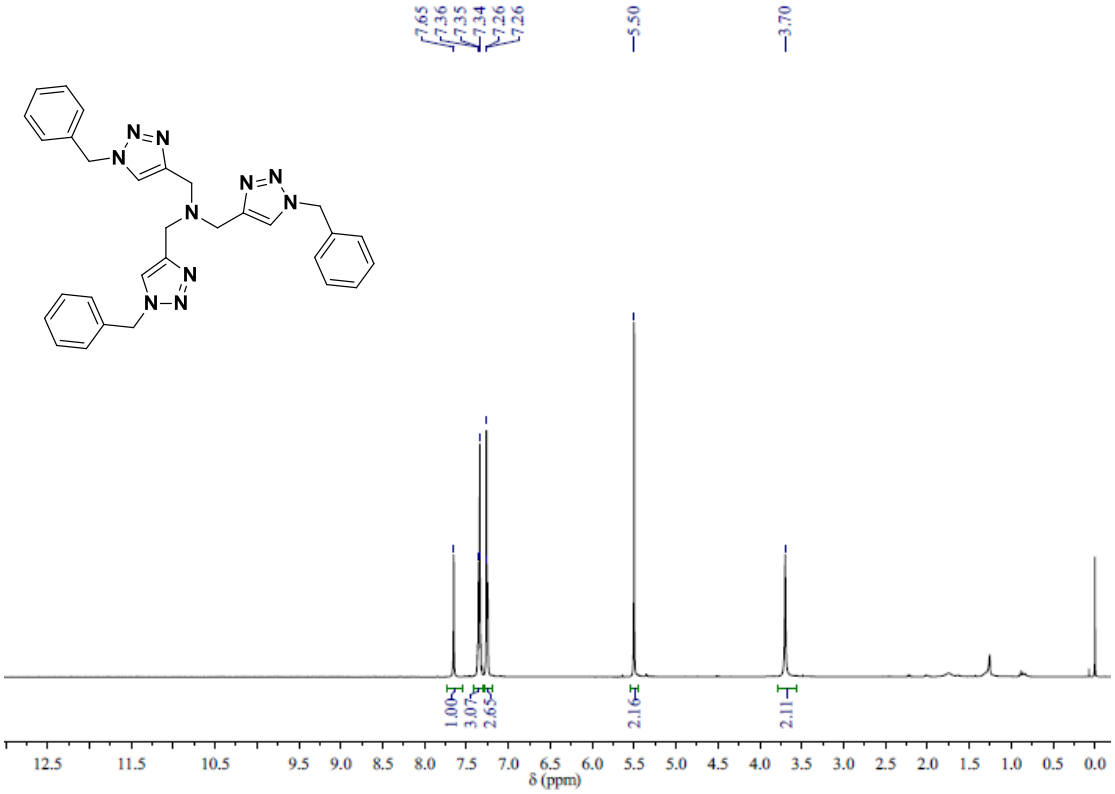
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7.26
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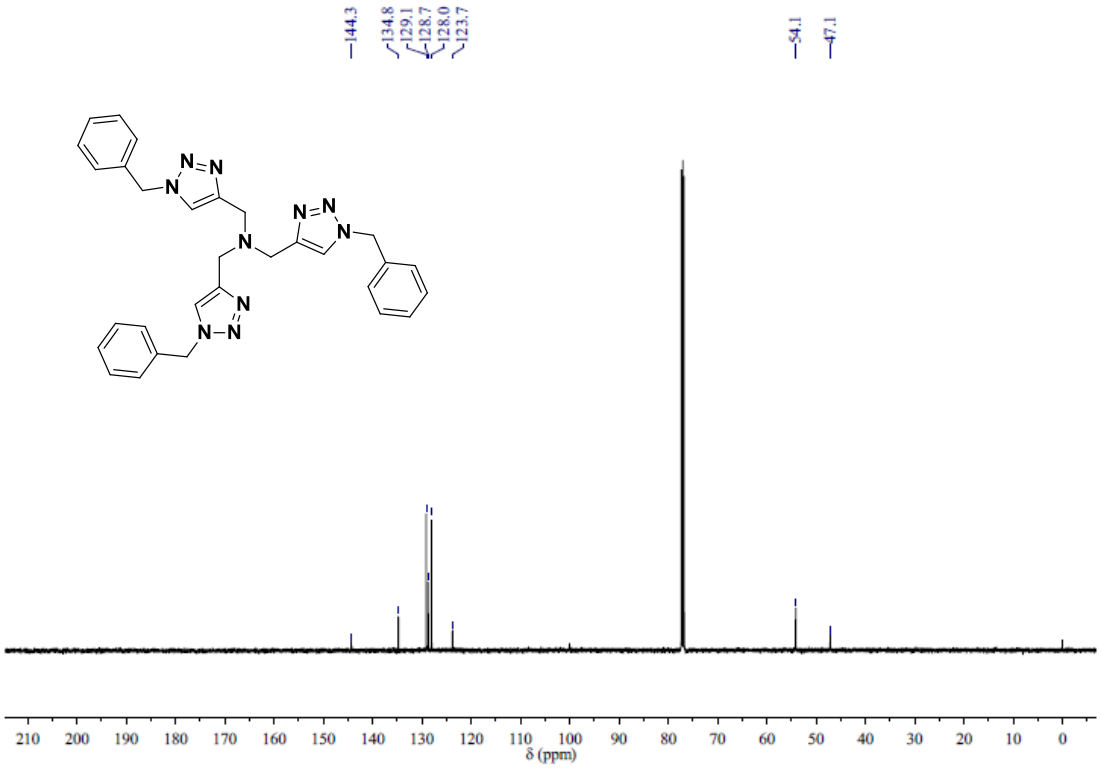
1.00
3.07
2.65
2.16
2.11
δ (ppm)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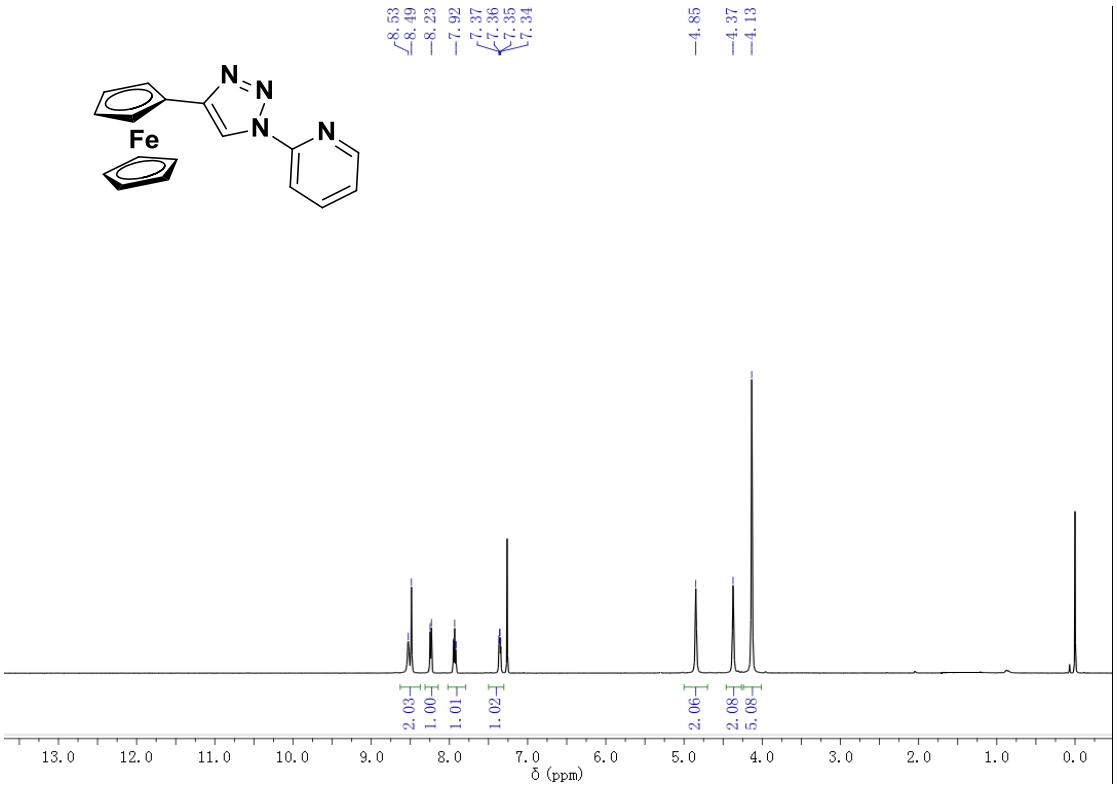
134.8
129.1
128.7
128.0
123.7
54.1
47.1
δ (ppm)

Compound 3a.


8.53
8.49
8.23
7.92
7.37
7.36
7.35
7.34
4.85
4.37
4.13
Fe
N
N
N
2.03
1.00
1.01
1.02
2.06
2.08
5.08
13.0
12.0
11.0
10.0
9.0
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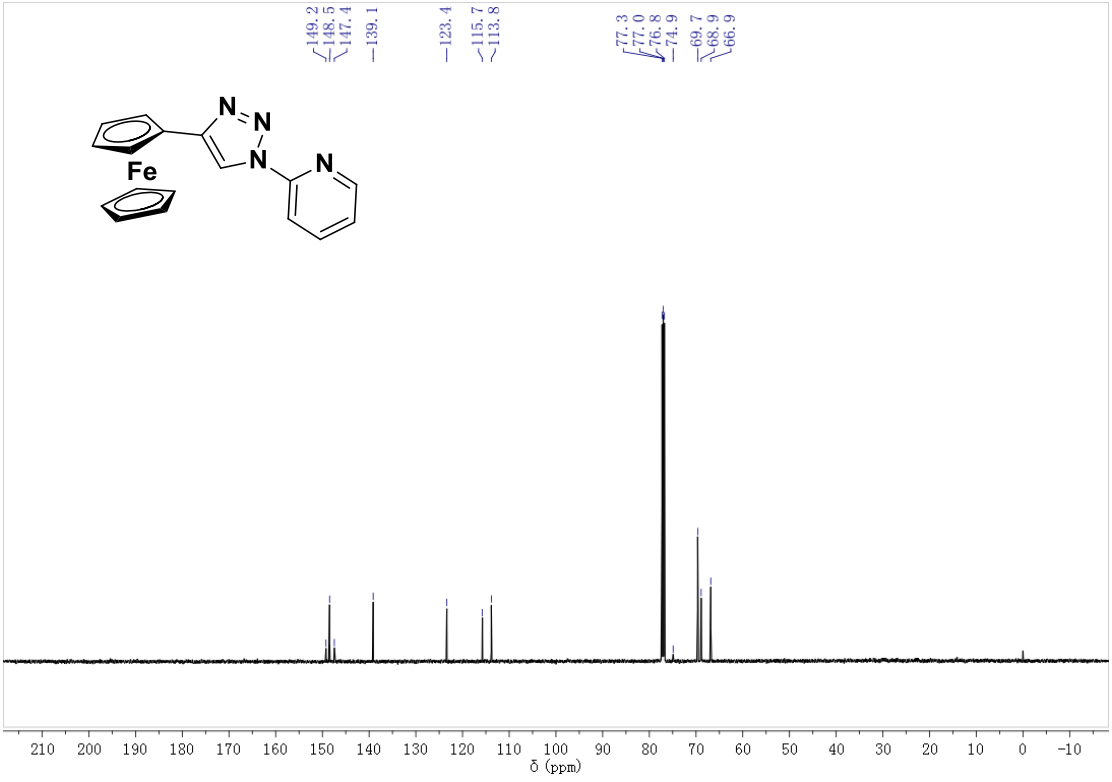
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
δ (ppm)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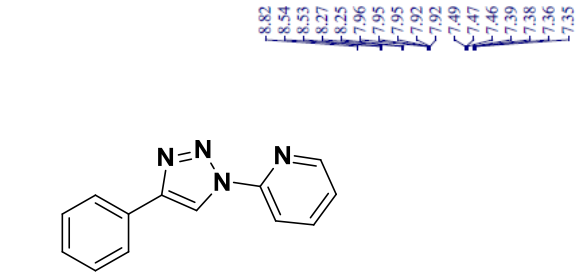
139.1
123.4
115.7
113.8
77.3
77.0
76.8
74.9
69.7
68.9
66.9
Fe
N
N
N
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
δ (ppm)

Compound 3b.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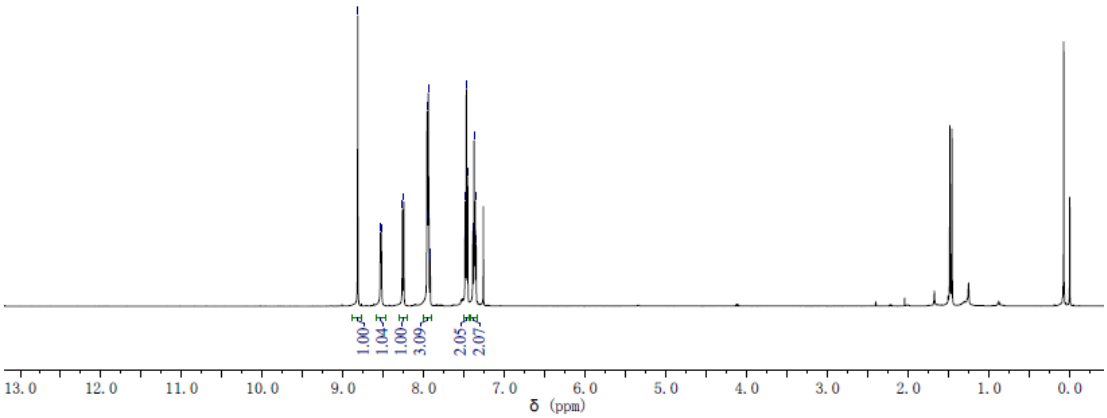
7.36
7.35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1.04
1.00
3.09
2.05
2.07
13.0
12.0
11.0
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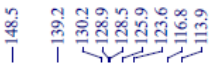
4.0
3.0
2.0
1.0
0.0
δ (ppm)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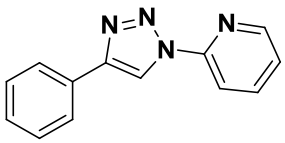
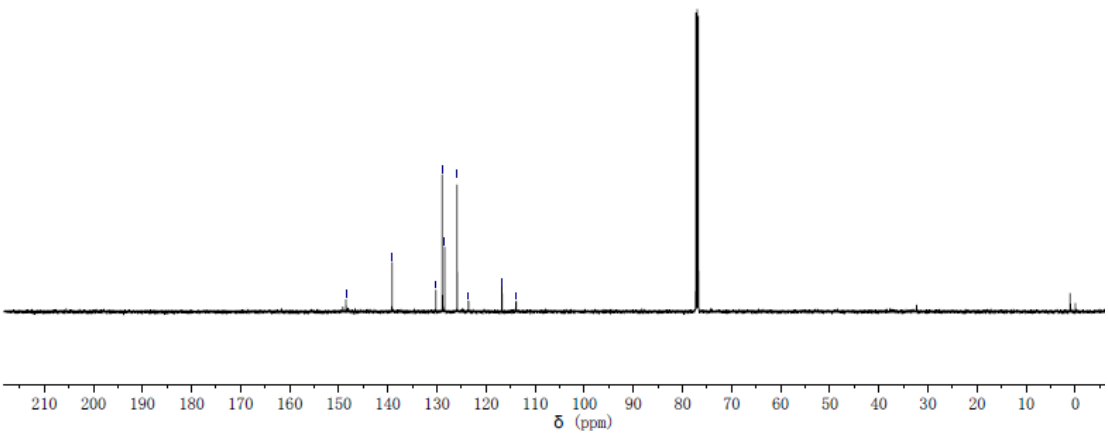
128.9
128.5
125.9
123.6
116.8
113.9

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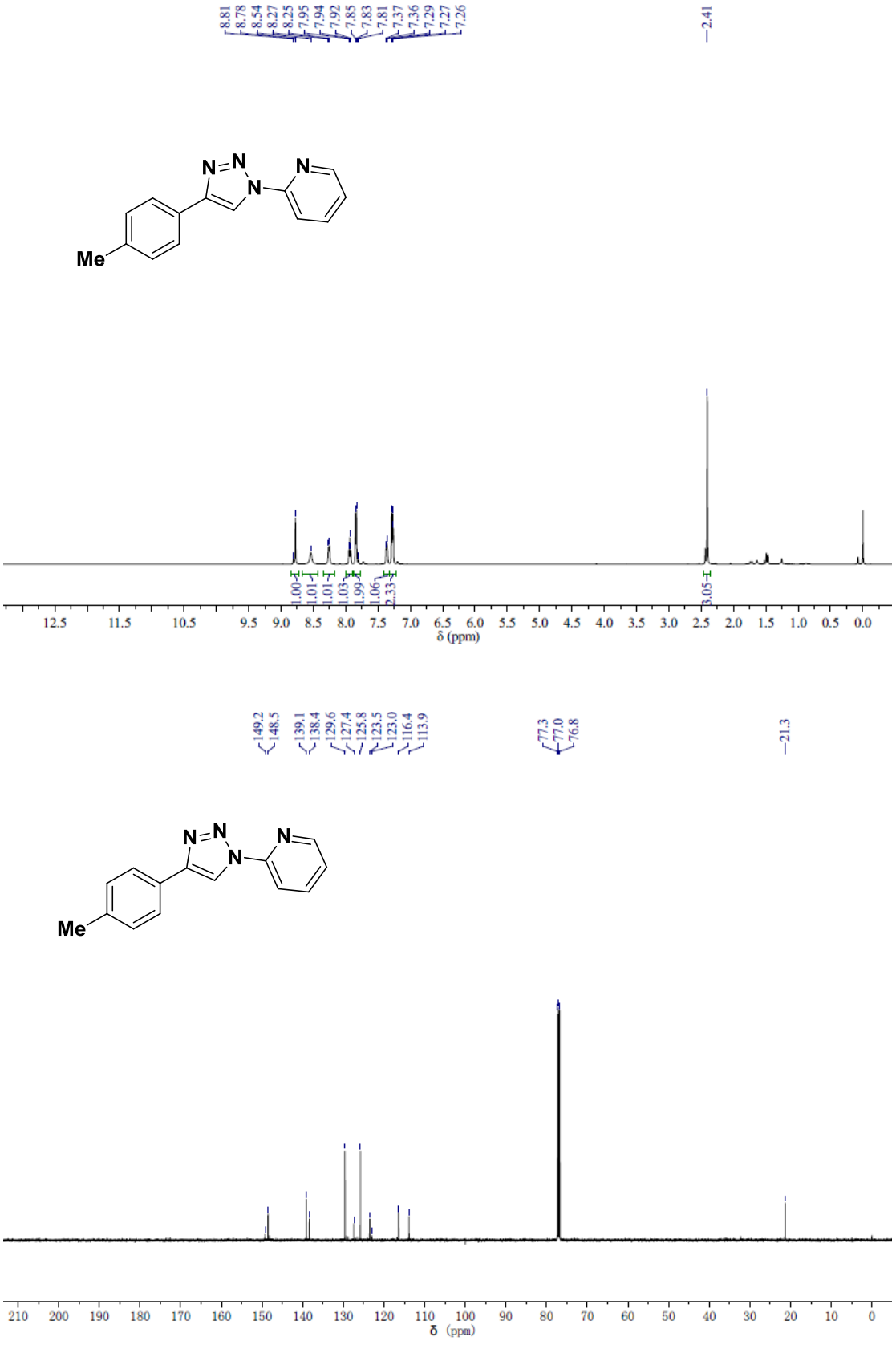
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

Compound 3c.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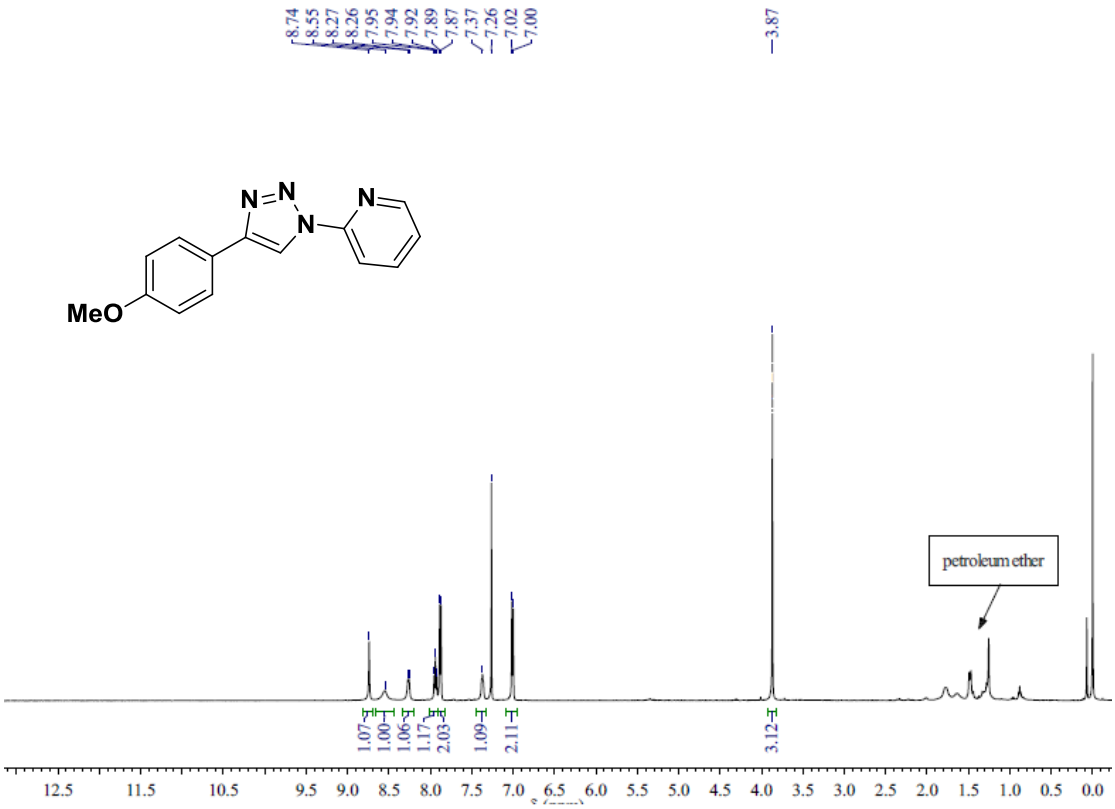
8.78
8.54
8.27
8.25
7.95
7.94
7.92
7.85
7.83
7.81
7.37
7.36
7.29
7.27
7.26
2.41
N
N
N
Me
1.00
1.01
1.01
1.03
1.99
1.06
2.33
3.05
δ (ppm)
149.2
148.5
139.1
138.4
129.6
127.4
125.8
123.5
123.0
116.4
113.9
77.3
77.0
76.8
21.3
δ (ppm)

Compound 3d.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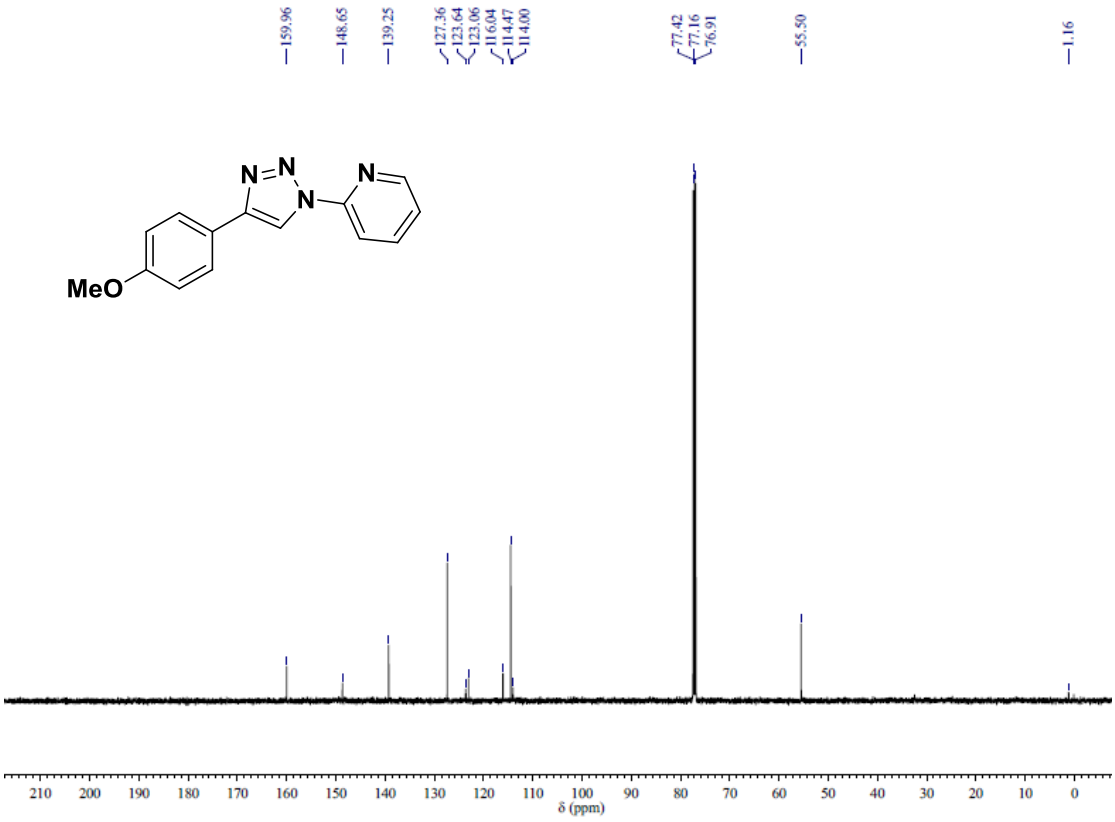
7.94
7.92
7.89
7.87
7.37
7.26
7.02
7.00
3.87
MeO
petroleum ether
1.07
1.00
1.06
1.17
2.03
1.09
2.11
3.12
12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


159.96
148.65
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127.36
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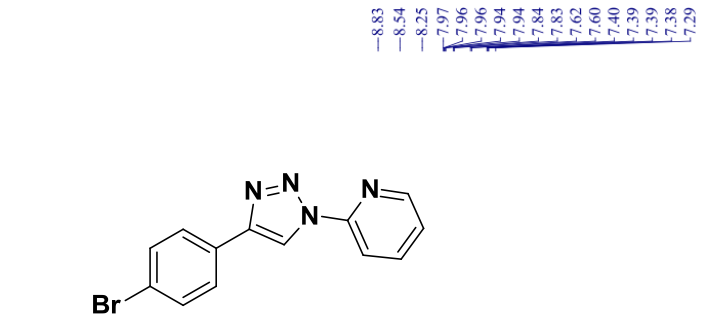
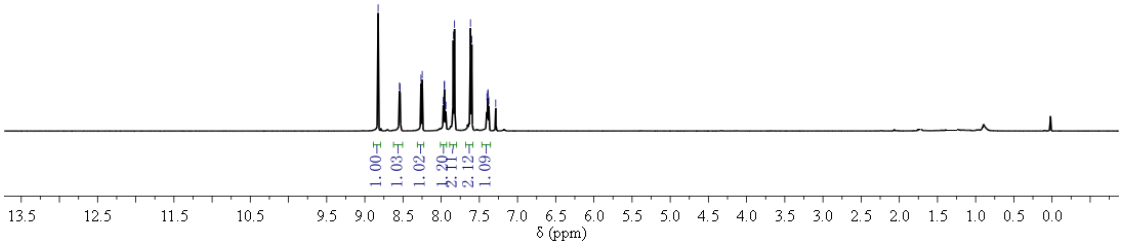
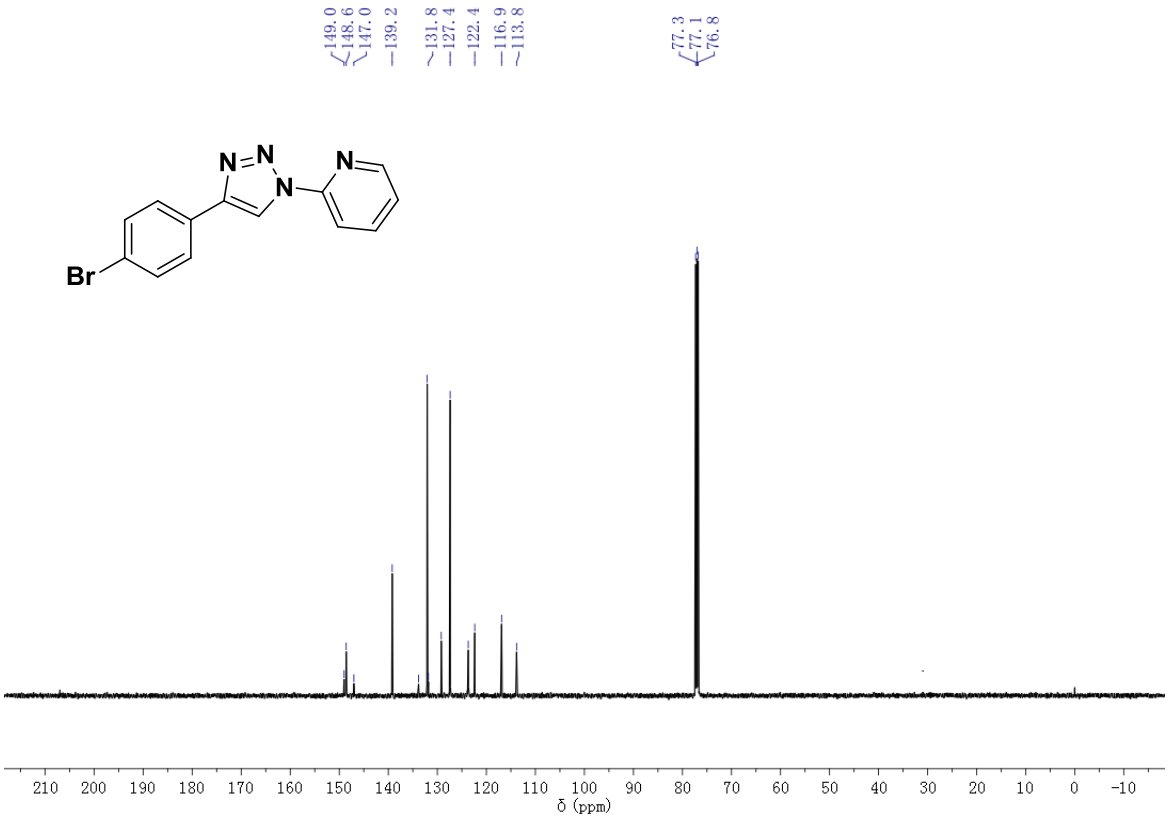
114.47
114.00
77.42
77.16
76.91
55.50
1.16
MeO
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
δ (ppm)

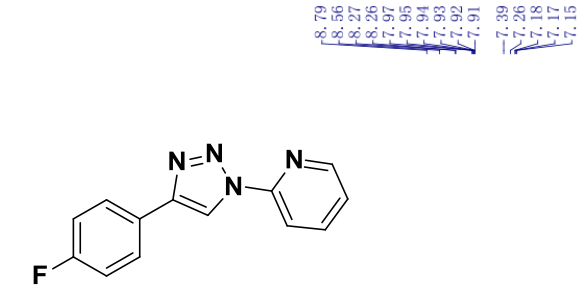
Compound 3e.

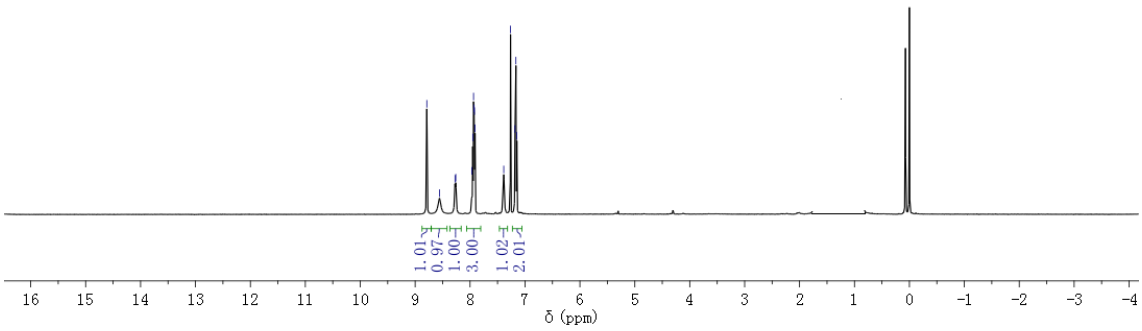

8.83
8.54
8.25
7.97
7.96
7.96
7.94
7.94
7.84
7.83
7.62
7.60
7.40
7.39
7.39
7.38
7.29
N
N
N
Br

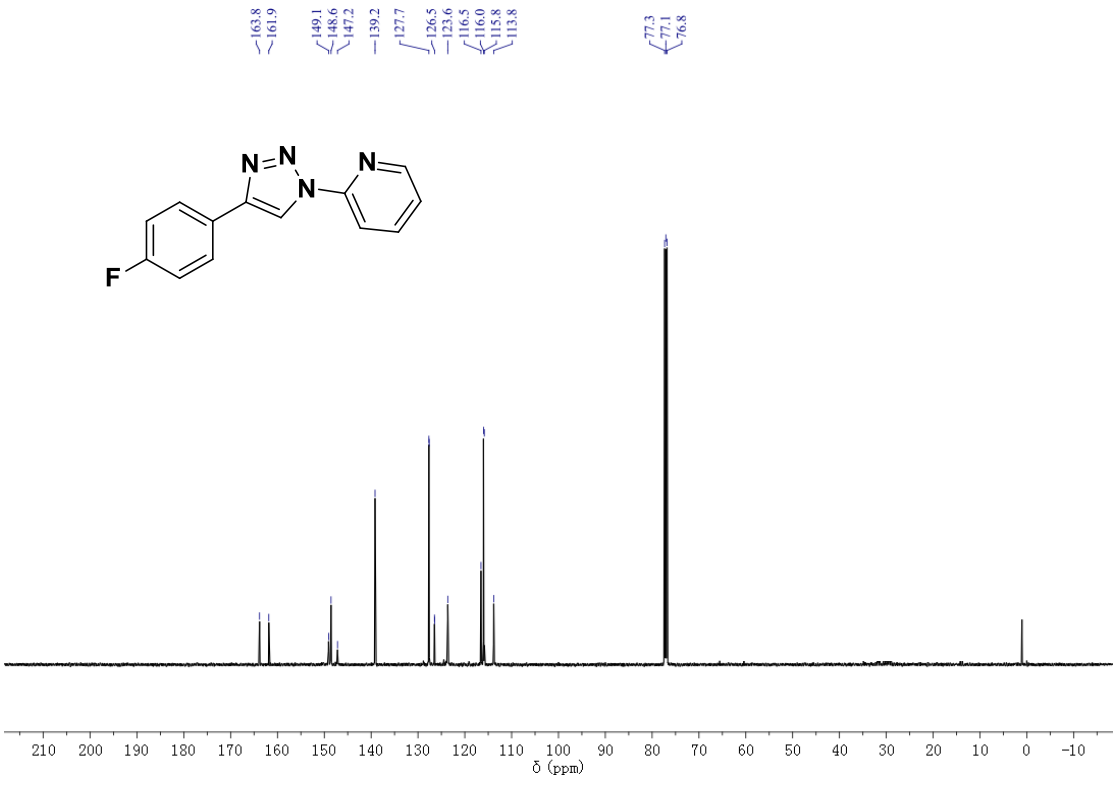

1.00
1.03
1.02
1.20
1.11
2.12
1.09
13.5 12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
δ (ppm)


149.0
148.6
147.0
139.2
131.8
127.4
122.4
116.9
113.8
77.3
77.1
76.8
N
N
N
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
δ (ppm)

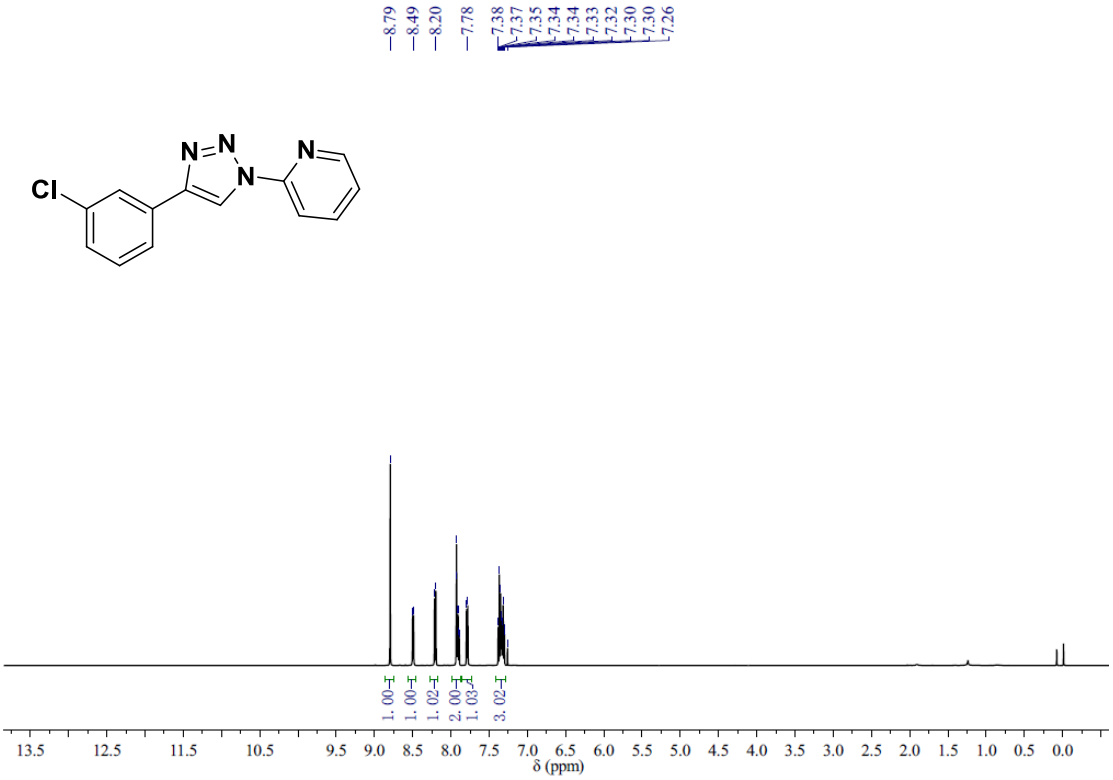
Compound 3f.

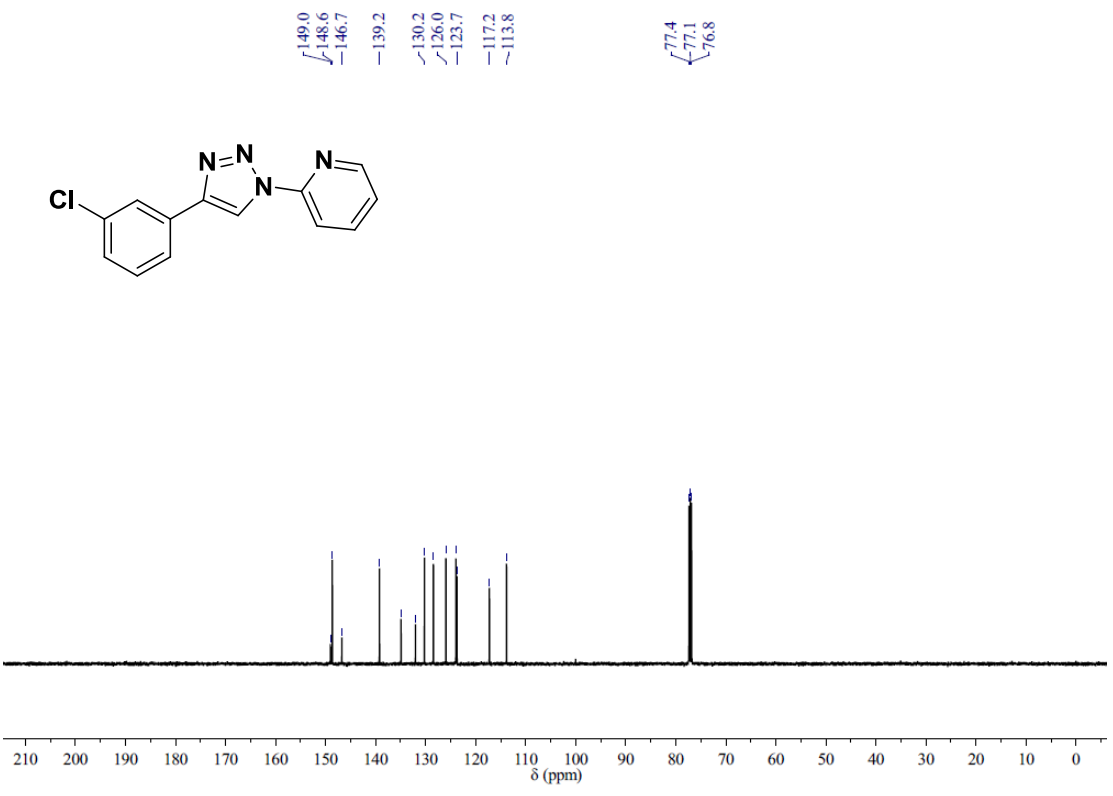

8.79
8.56
8.27
8.26
7.97
7.95
7.94
7.93
7.92
7.91
7.39
7.26
7.18
7.17
7.15
N
N
N
F


1.01
0.97
1.00
3.00
1.02
2.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
δ (ppm)

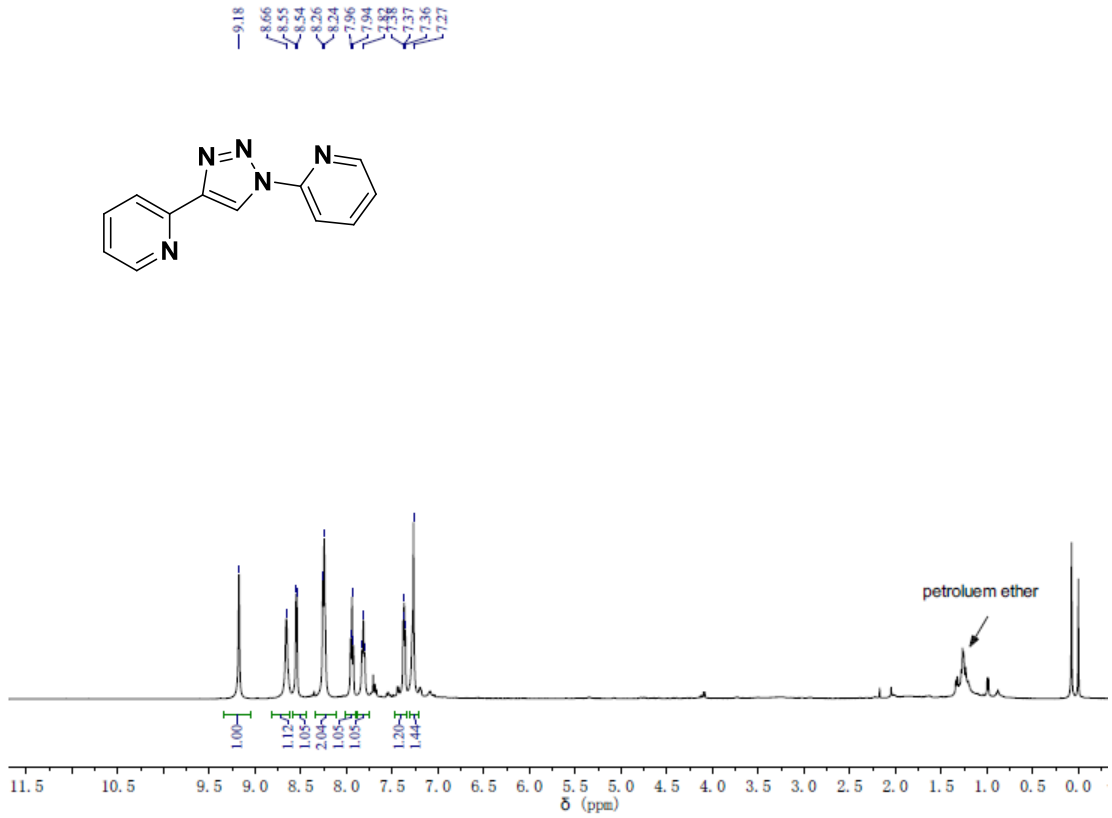

163.8
161.9
149.1
148.6
147.2
139.2
127.7
126.5
123.6
116.5
116.0
115.8
113.8
77.3
77.1
76.8
N
N
N
F
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
δ (ppm)

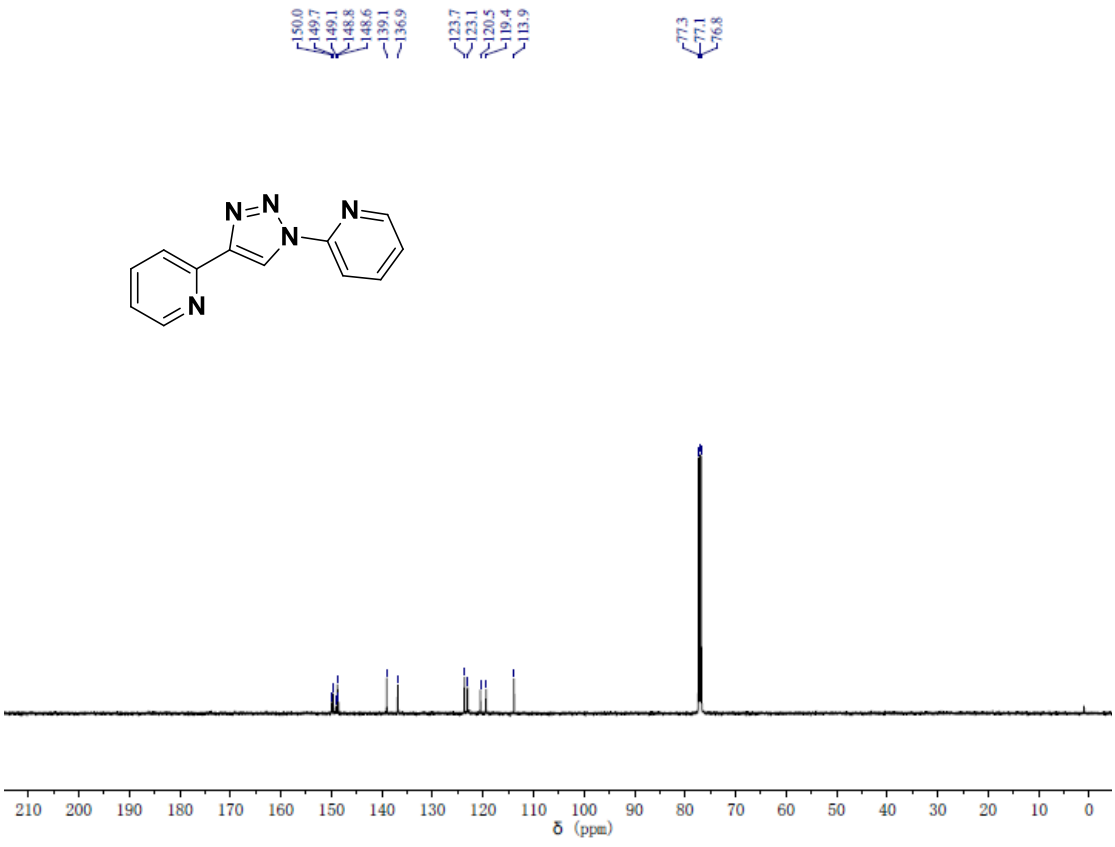
Compound 3g.


8.79
8.49
8.20
7.78
7.38
7.37
7.35
7.34
7.34
7.33
7.32
7.30
7.30
7.26
Cl
N
N
N
1.00
1.00
1.02
2.00
1.03
3.02
δ (ppm)

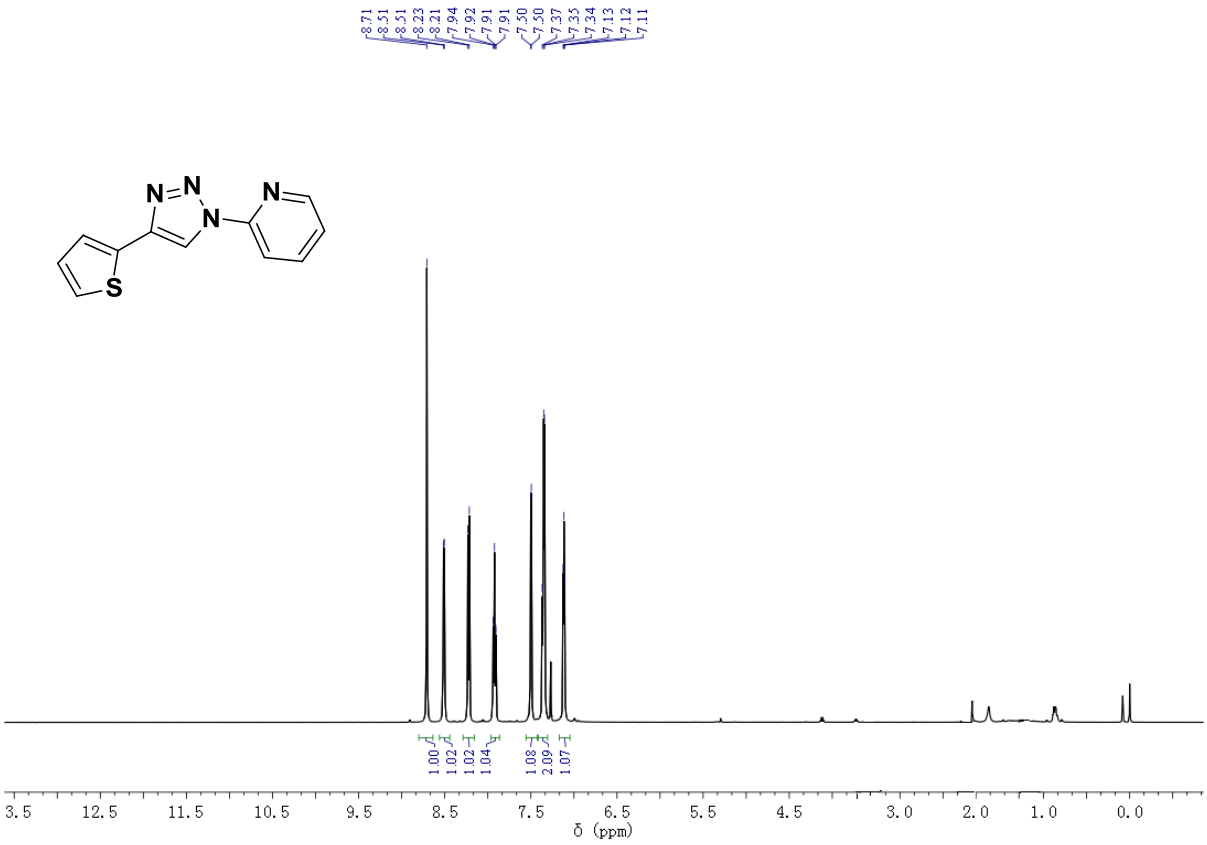

149.0
148.6
146.7
139.2
130.2
126.0
123.7
117.2
113.8
77.4
77.1
76.8
Cl
N
N
N
δ (ppm)

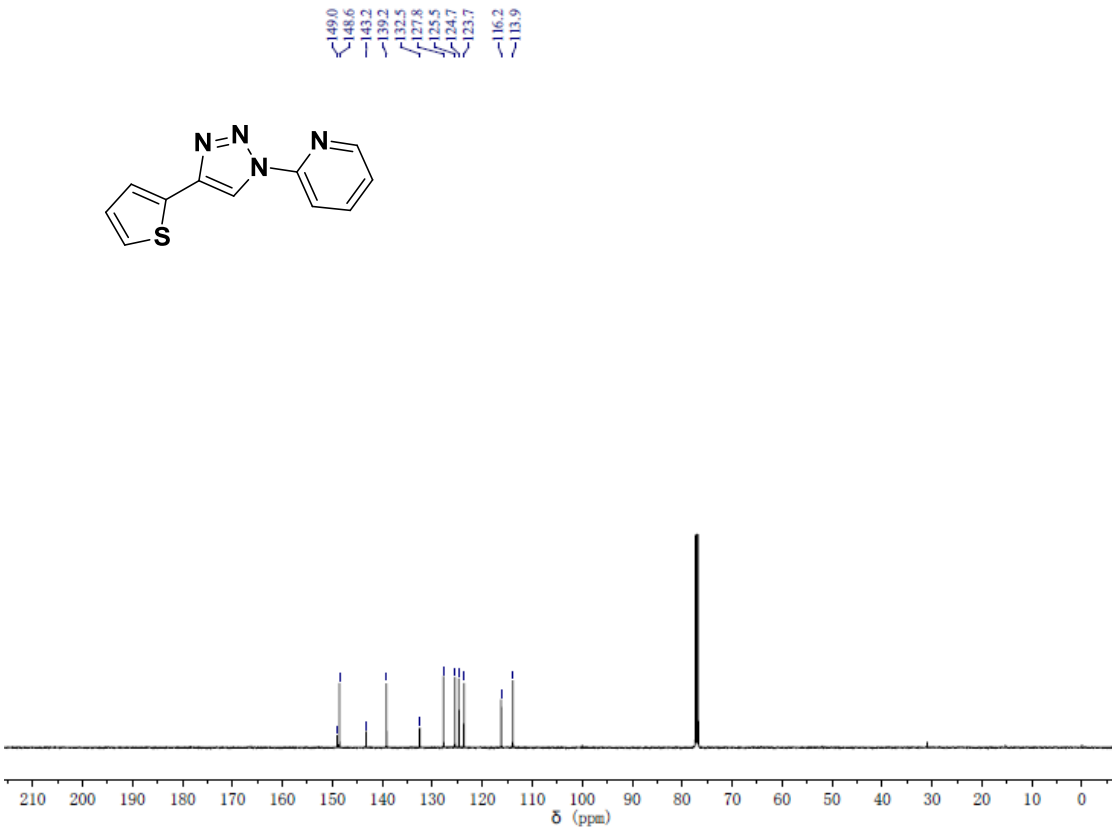
Compound 3h.


9.18
8.66
8.55
8.54
8.26
8.24
7.96
7.94
7.82
7.38
7.37
7.36
7.27
petroluem ether
1.00
1.12
1.05
2.04
1.05
1.05
1.20
1.44
δ (ppm)

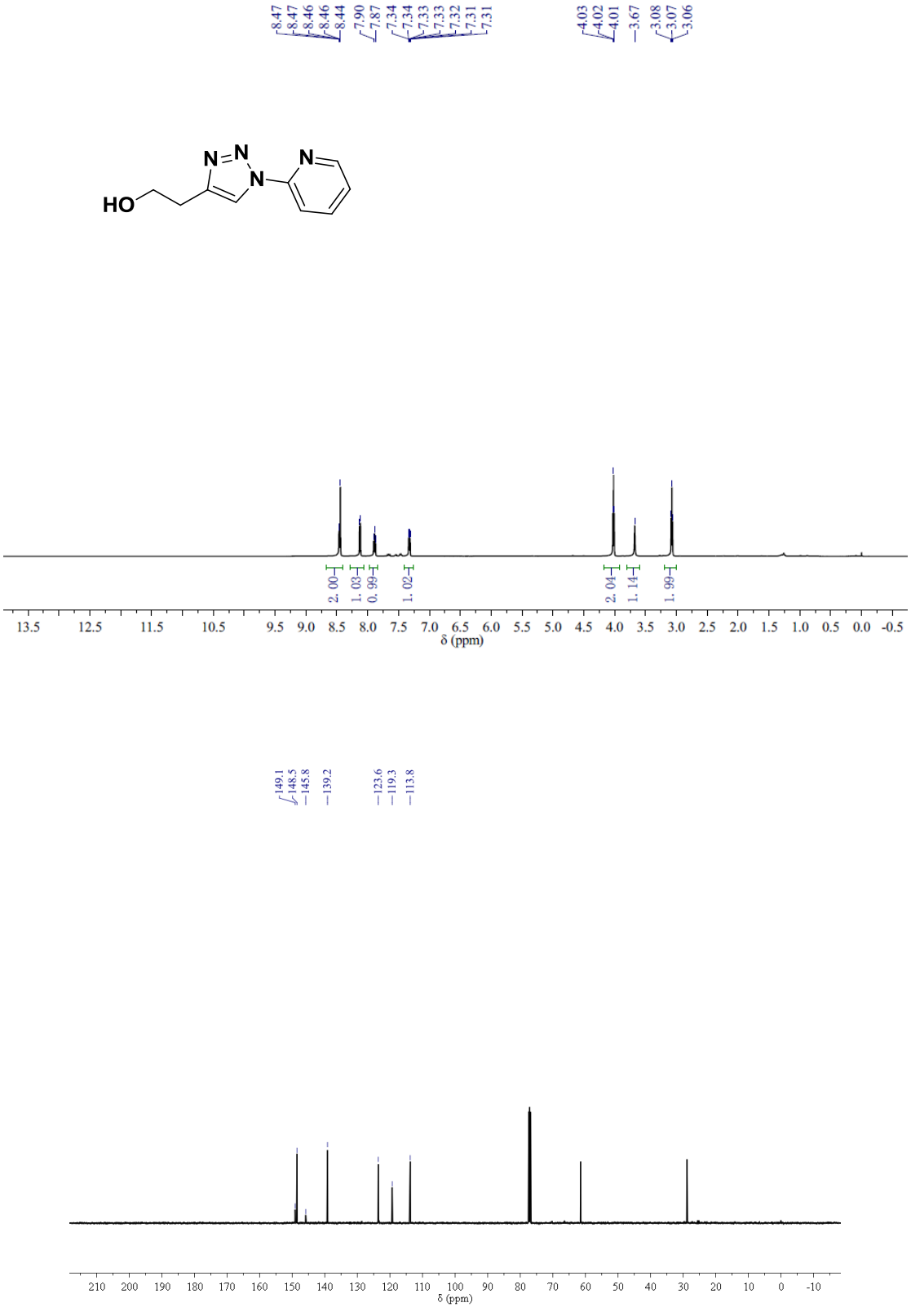

150.0
149.7
149.1
148.8
148.6
139.1
136.9
123.7
123.1
120.5
119.4
113.9
77.3
77.1
76.8
δ (ppm)

Compound 3i.


8.71
8.51
8.51
8.23
8.21
7.94
7.92
7.91
7.91
7.50
7.50
7.37
7.35
7.34
7.13
7.12
7.11
1.00
1.02
1.02
1.04
1.08
2.09
1.07
3.5
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.0
2.0
1.0
0.0
δ (ppm)

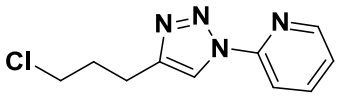

149.0
148.6
143.2
139.2
132.5
127.8
125.5
124.7
123.7
116.2
113.9
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

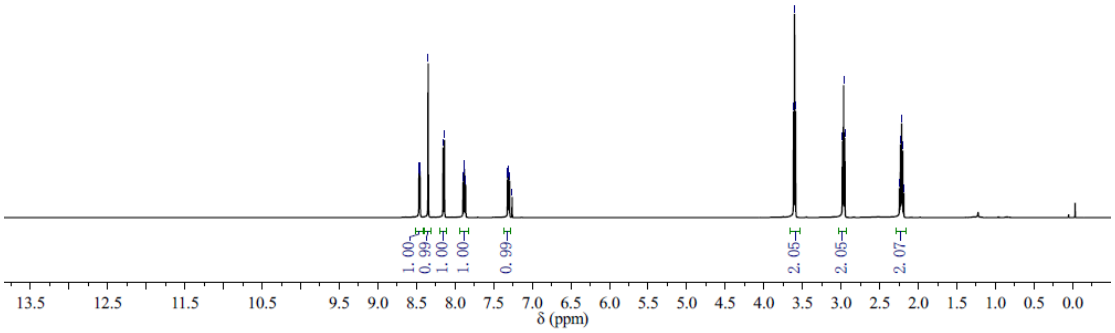
Compound 3j.


8.47
8.47
8.46
8.46
8.44
7.90
7.87
7.34
7.34
7.33
7.33
7.32
7.31
7.31
4.03
4.02
4.01
3.67
3.08
3.07
3.06
HO
N
N
N
2.00
1.03
0.99
1.02
2.04
1.14
1.99
δ (ppm)
149.1
148.5
145.8
139.2
123.6
119.3
113.8
δ (ppm)

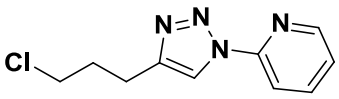
Compound 3k.

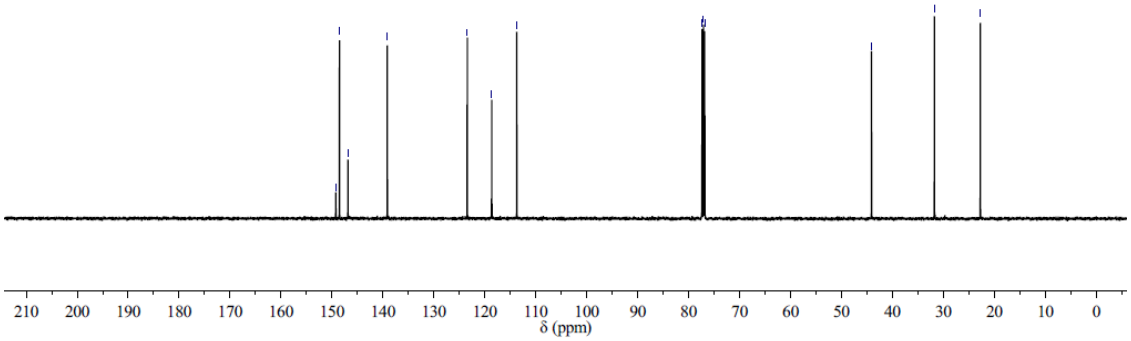

8.47
8.46
8.46
8.46
8.46
8.45
8.45
8.35
8.15
8.14
7.90
7.89
7.88
7.88
7.88
7.88
7.87
7.86
7.32
7.32
7.31
7.31
7.31
7.30
7.30
7.29
7.26
3.61
3.60
3.59
3.97
2.95
2.24
2.23
2.22
2.20
2.19


N
N
N
Cl

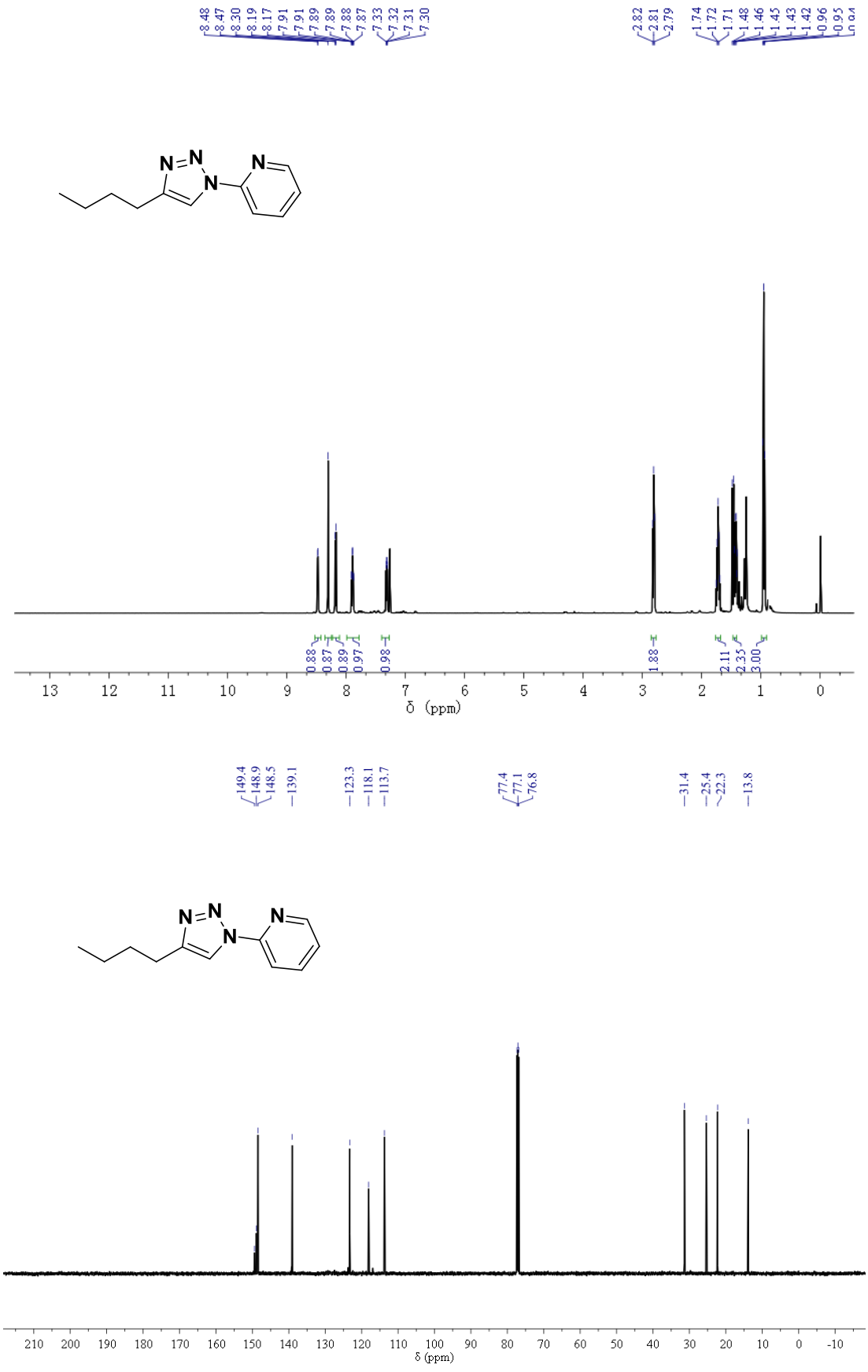

1.00
0.99
1.00
1.00
0.99
2.05
2.05
2.07
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
δ (ppm)


149.3
148.5
146.9
139.1
123.4
118.6
113.7
77.4
77.1
76.8
44.1
31.8
22.7

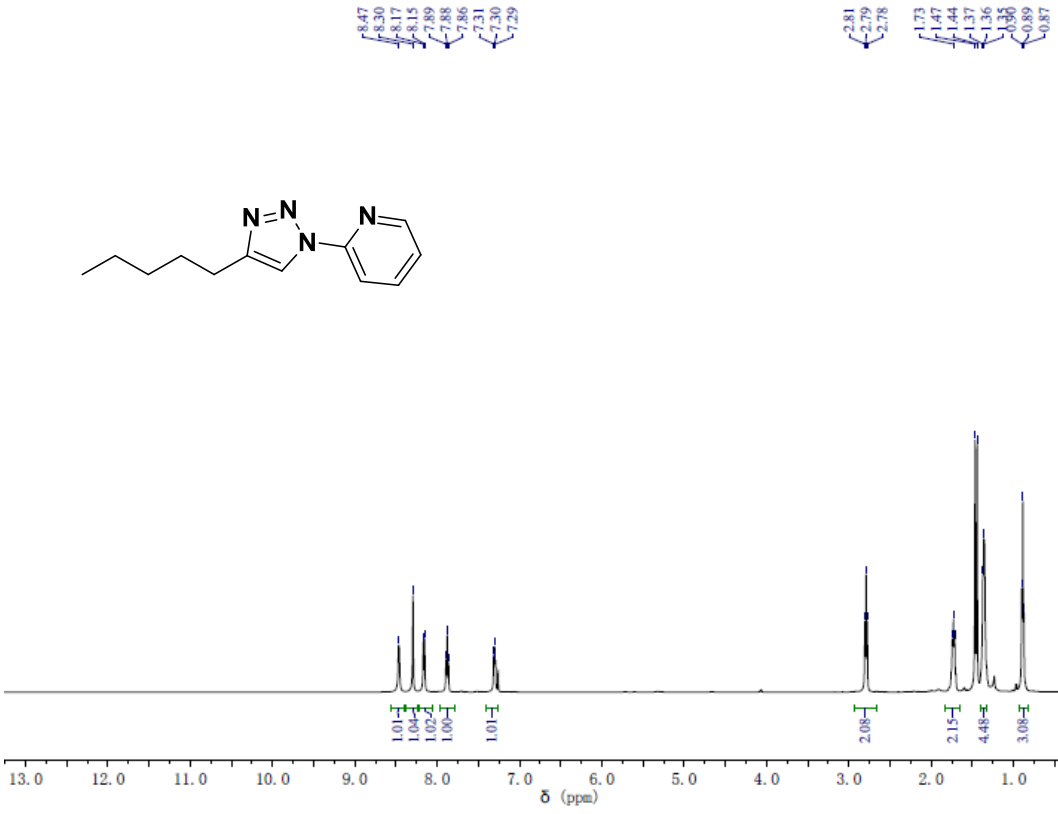

N
N
N
Cl

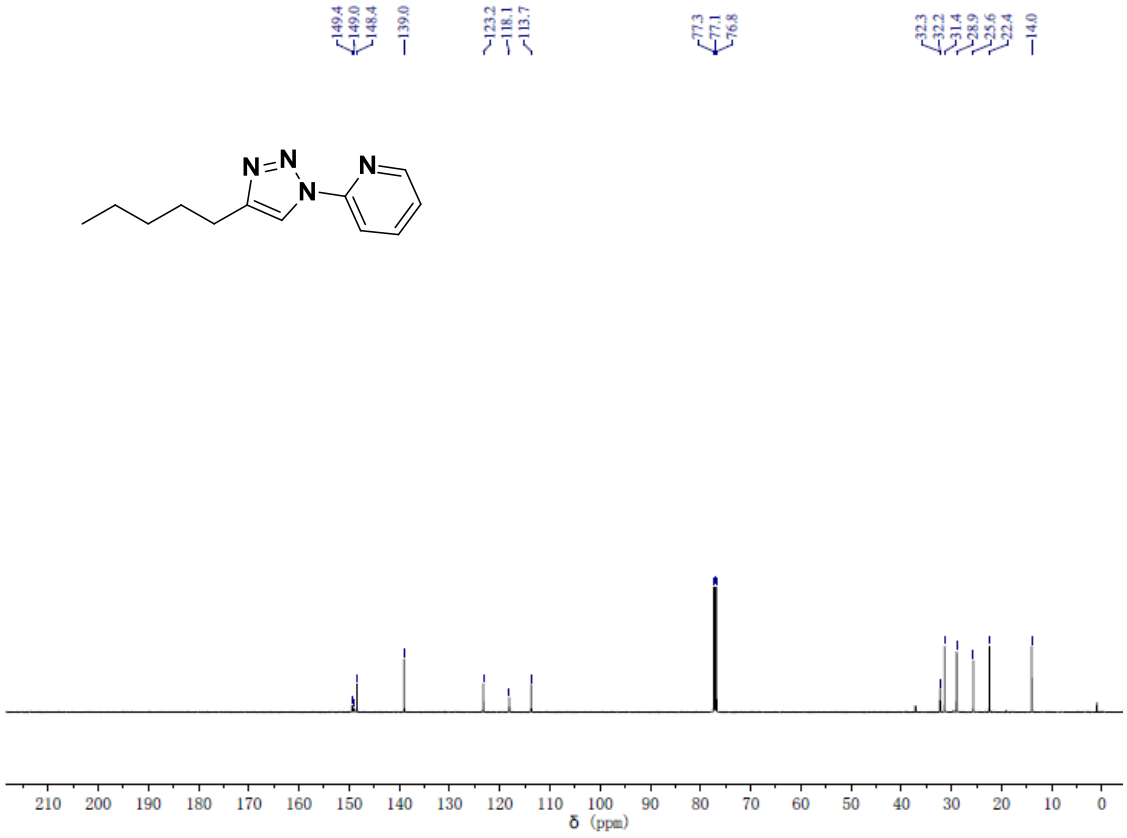

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

Compound 3l.

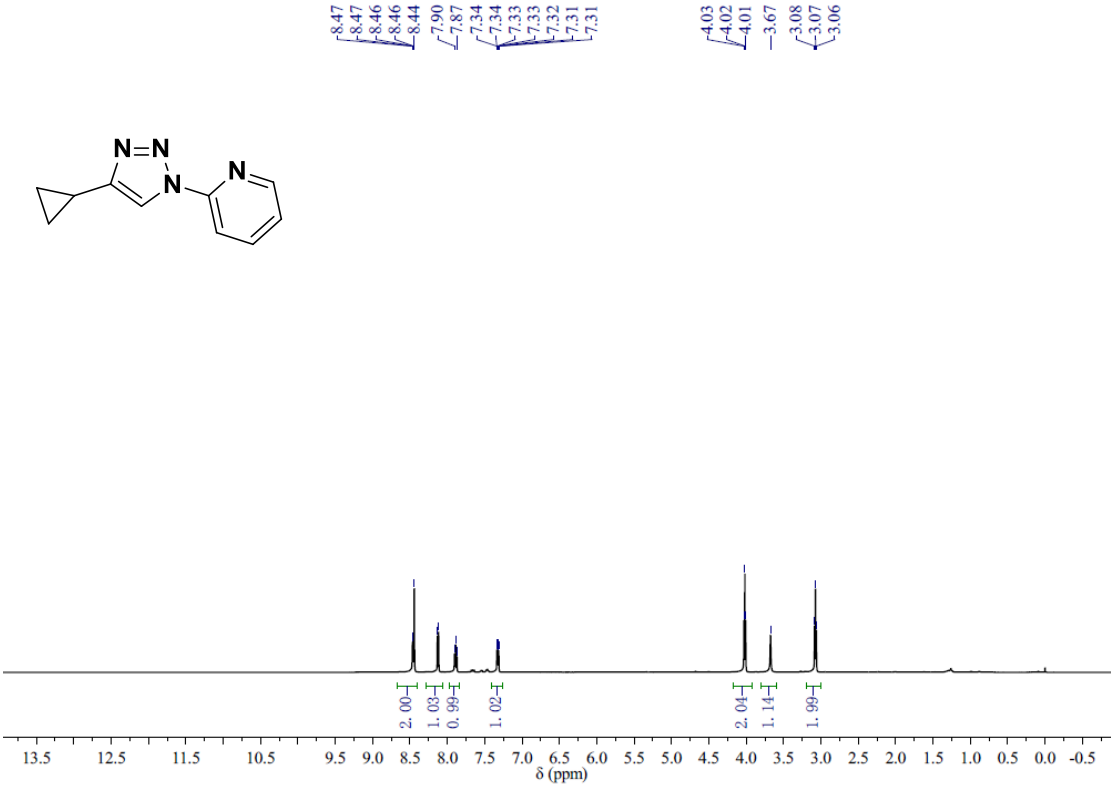

8.48
8.47
8.30
8.19
8.17
7.91
7.91
7.89
7.89
7.88
7.87
7.33
7.32
7.31
7.30
2.82
2.81
2.79
1.74
1.72
1.71
1.48
1.46
1.45
1.43
1.42
0.96
0.95
0.88
0.87
0.89
0.97
0.98
1.88
2.11
2.35
3.00
δ (ppm)
149.4
148.9
148.5
139.1
123.3
118.1
113.7
77.4
77.1
76.8
31.4
25.4
22.3
13.8
δ (ppm)

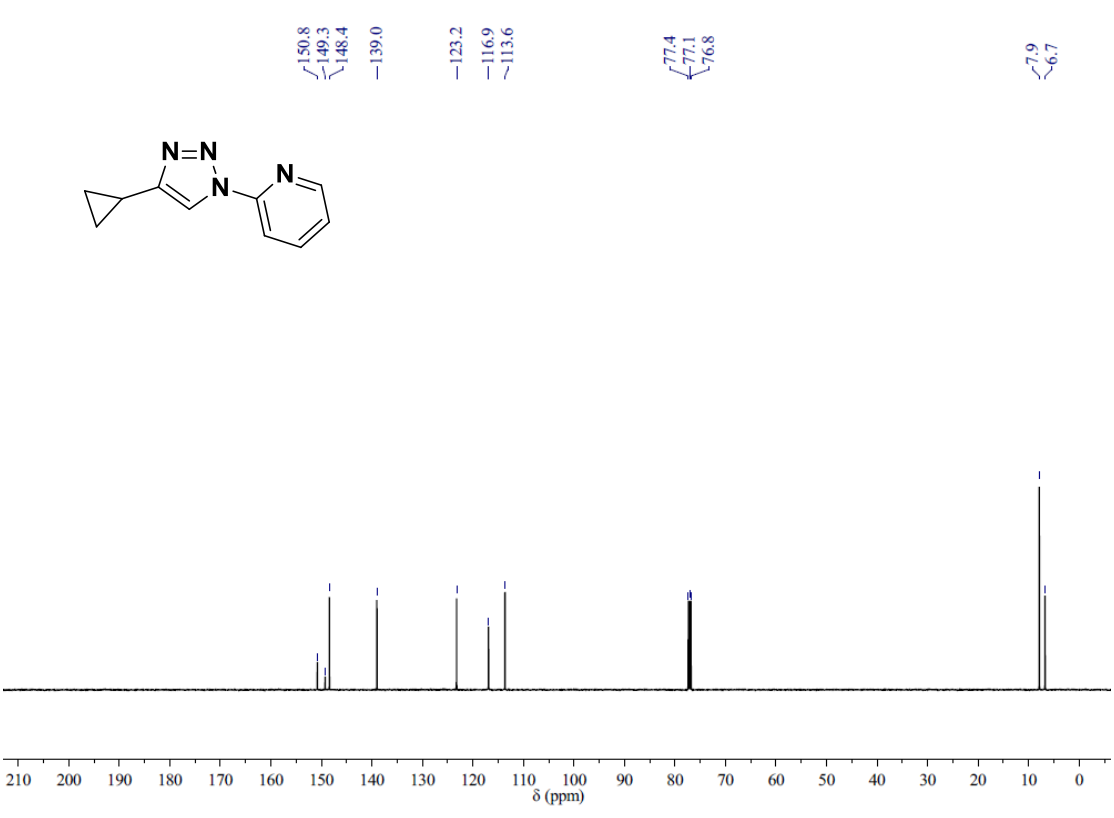
Compound 3m.


8.47
8.30
8.17
8.15
7.89
7.88
7.86
7.31
7.30
7.29
2.81
2.79
2.78
1.73
1.47
1.44
1.37
1.36
0.90
0.89
0.87
1.01
1.04
1.02
1.00
1.01
2.08
2.15
4.48
3.08
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
δ (ppm)

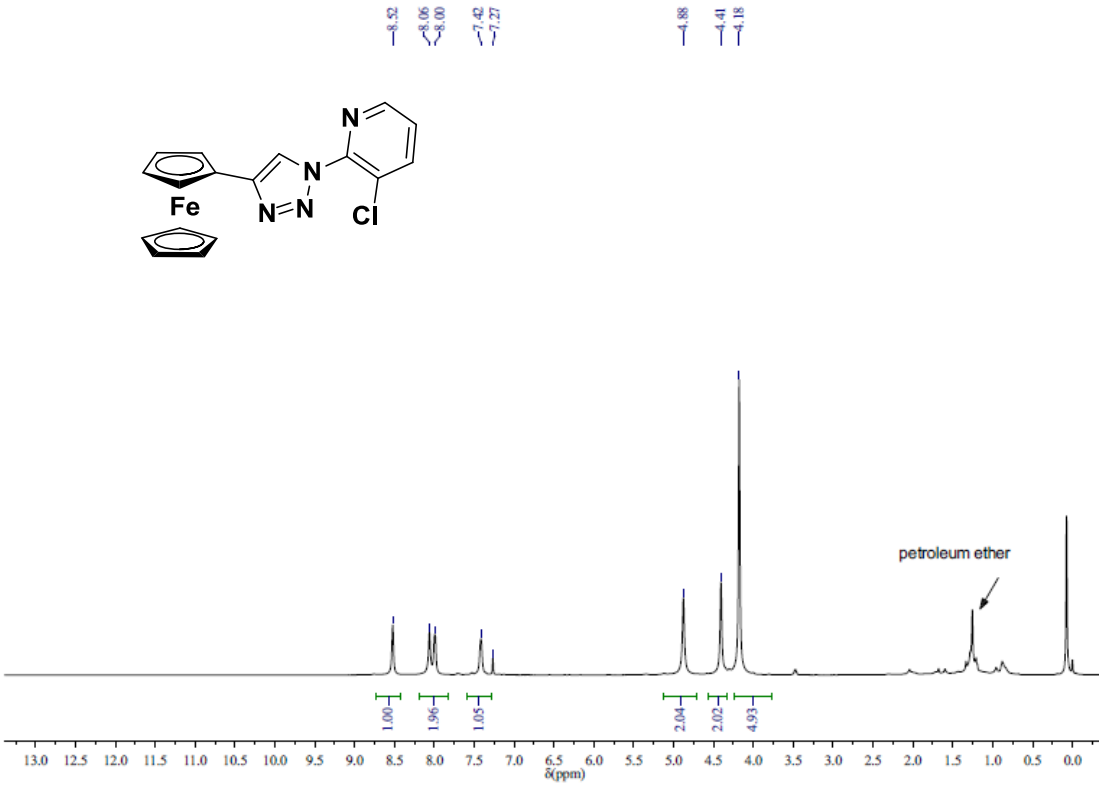

149.4
149.0
148.4
139.0
123.2
118.1
113.7
77.3
77.1
76.8
32.3
32.2
31.4
28.9
25.6
22.4
14.0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
δ (ppm)

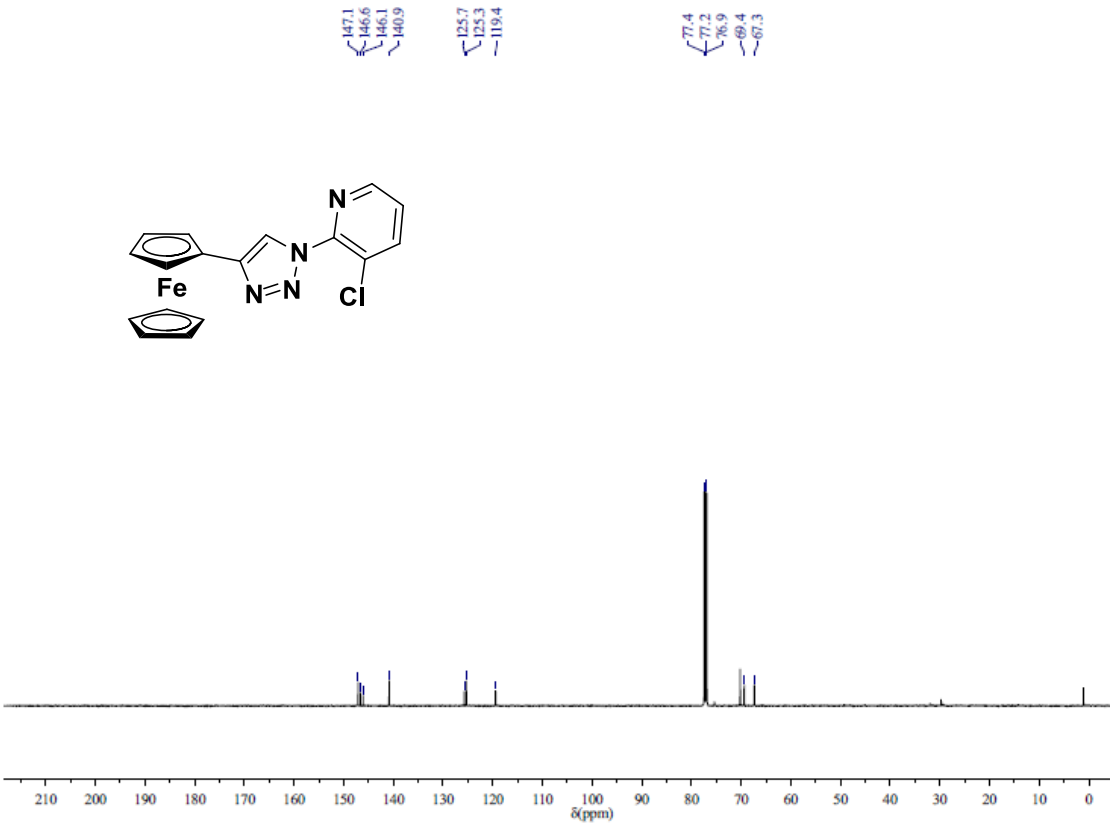
Compound 3n.


8.47
8.47
8.46
8.46
8.44
7.90
7.87
7.34
7.34
7.33
7.33
7.32
7.31
7.31
4.03
4.02
4.01
3.67
3.08
3.07
3.06
N=N
N
N
2.00
1.03
0.99
1.02
2.04
1.14
1.99
δ (ppm)

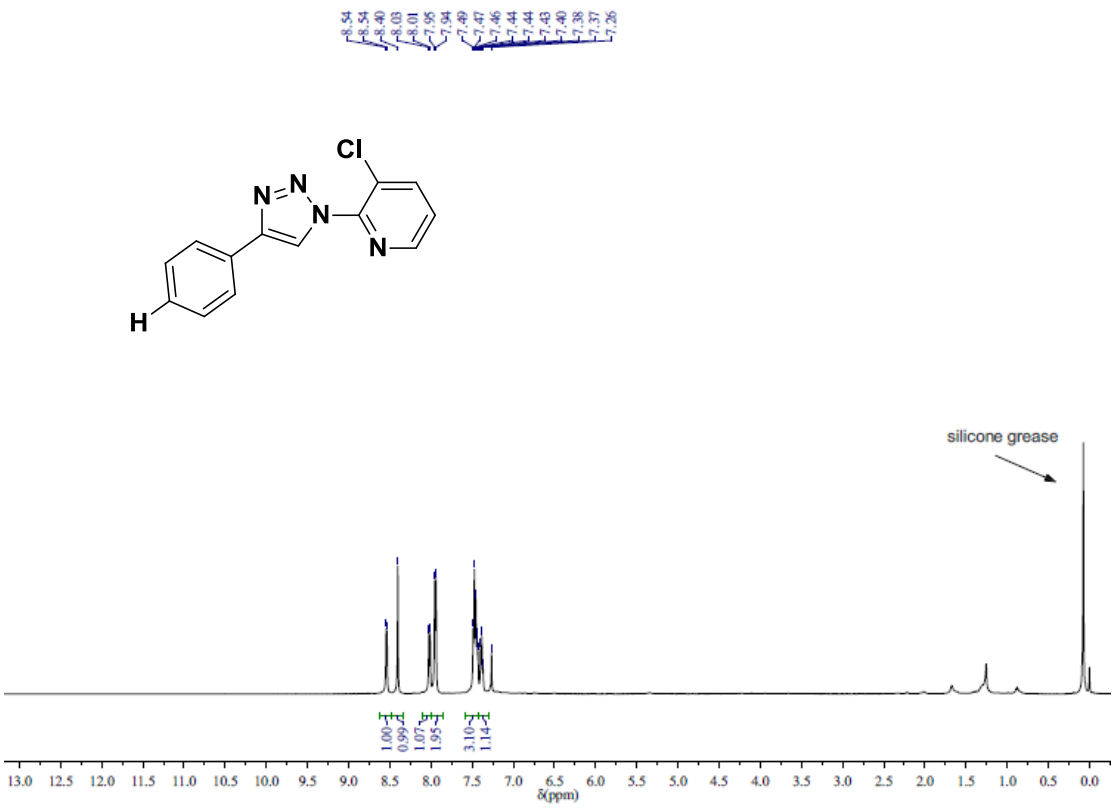

150.8
149.3
148.4
139.0
123.2
116.9
113.6
77.4
77.1
76.8
7.9
6.7
N=N
N
N
δ (ppm)

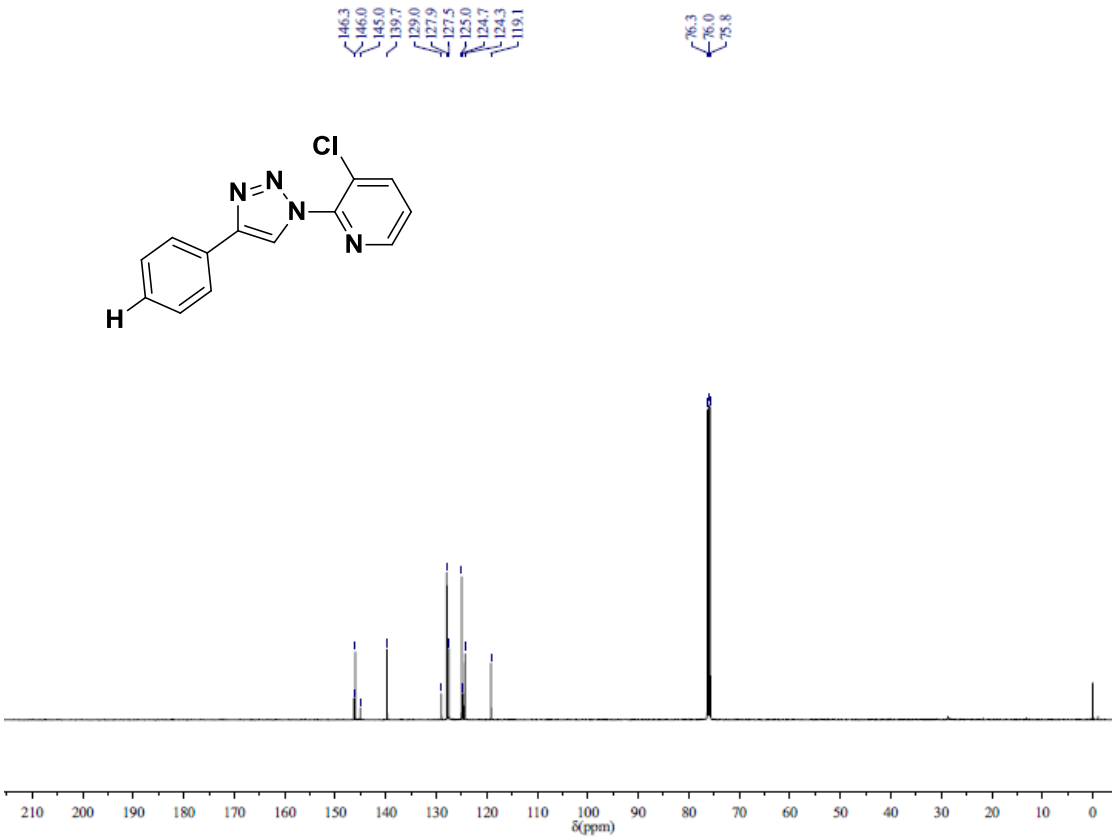
Compound 3o.


8.52
8.06
8.00
7.42
7.27
4.88
4.41
4.18
N
N
N
Fe
Cl
petroleum ether
1.00
1.96
1.05
2.04
2.02
4.93
δ(ppm)


147.1
146.6
146.1
140.9
125.7
125.3
119.4
77.4
77.2
76.9
69.4
67.3
N
N
N
Fe
Cl
δ(ppm)

Compound 3p.


8.54
8.54
8.40
8.03
8.01
7.95
7.94
7.49
7.47
7.46
7.44
7.44
7.43
7.40
7.38
7.37
7.26
Cl
N
N
N
H
silicone grease
1.00
0.99
1.07
1.95
3.10
1.14
δ(ppm)


146.3
146.0
145.0
139.7
129.0
127.9
127.5
125.0
124.7
124.3
119.1
76.3
76.0
75.8
Cl
N
N
N
H
δ(ppm)

Crystal data for compound 3a.

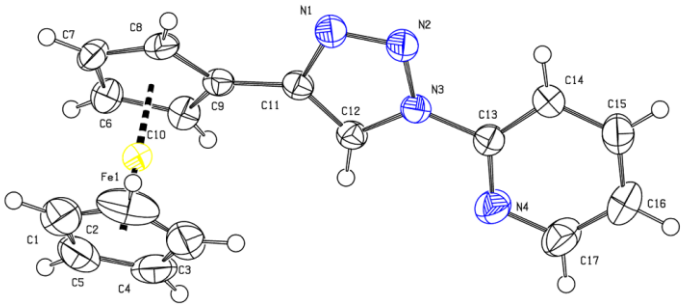


FIGURE S1. Molecular structure of compound **3a**.

ORTEP (50% ellipsoids) diagrams of [$C_{17}H_{14}FeN_4$] Selected distances (Å): [C(9)-C(11) 1.459(3), C(11)-N(1) 1.369(3), N(1)-N(2) 1.309(3), N(3)-N(2) 1.357(2), N(3)-C(12) 1.357(2), N(3)-C(13) 1.427(3)].

Table 1. Sample and crystal data for compound **3a**

Identification code	compound 3a	
Chemical formula	$C_{17}H_{15}FeN_4$	
Formula weight	331.18	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 5.8931(13) Å	α = 90 °
	b = 20.199(4) Å	β = 92.215(4) °
	c = 11.950(3) Å	γ = 90 °
Volume	1421.4(5) Å3	
Z	4	
Density (calculated)	1.548 g/cm^3	
Absorption coefficient	1.062 mm^{-1}	
F(000)	684	
Theta range for data collection	1.98 to 28.28 °	
Index ranges	-7<=h<=7, -26<=k<=26, -15<=l<=15	
Reflections collected	19469	
Independent reflections	3511 [R(int) = 0.0321]	
Absorption correction	multi-scan	
Structure solution technique	direct methods	
Structure solution program	SHELXS-97 (Sheldrick, 2008)	
Refinement method	Full-matrix least-squares on F2	

Refinement program	SHELXL-97 (Sheldrick, 2008)
Function minimized	$\Sigma\ w(Fo_2 - Fc_2)_2$
Data / restraints / parameters	3511 / 1 / 199
Goodness-of-fit on F2	1.034
Δ/σmax	0.002
Final R indices	2959 data; I>2σ(I)
	R1 = 0.0344, wR2 = 0.0848
all data	R1 = 0.0436, wR2 = 0.0897
Weighting scheme	w=1/[σ2(Fo2)+(0.0438P)2+0.6794P]
	where P=(Fo2+2Fc2)/3
Largest diff. peak and hole	0.381 and -0.272 eÅ-3
R.M.S. deviation from mean	0.054 eÅ-3

Table 2 Selected bond lengths (Å) and angles (°) for compound **3a**

Fe(1)-C(1)	2.043(3)	Fe(1)-C(2)	2.031(3)
Fe(1)-C(3)	2.032(3)	Fe(1)-C(4)	2.035(3)
Fe(1)-C(5)	2.033(3)	Fe(1)-C(6)	2.042(2)
Fe(1)-C(7)	2.049(2)	Fe(1)-C(8)	2.053(2)
Fe(1)-C(9)	2.052(2)	Fe(1)-C(10)	2.046(2)
C(11)-N(1)	1.369(3)	N(1)-N(2)	1.309(3)
N(3)-N(2)	1.357(2)	N(3)-C(12)	1.357(3)
N(3)-C(13)	1.427(3)	C(13)-N(4)	1.320(3)
N(4)-C(17)	1.345(3)	C(11)-C(9)	1.459(3)
C(2)-Fe(1)-C(1)	40.73(13)	C(3)-Fe(1)-C(1)	67.88(12)
C(4)-Fe(1)-C(1)	66.93(12)	C(5)-Fe(1)-C(1)	39.54(12)
C(6)-Fe(1)-C(1)	121.72(12)	C(1)-Fe(1)-C(7)	108.98(11)
C(1)-Fe(1)-C(8)	126.20(11)	C(1)-Fe(1)-C(9)	162.49(12)
C(1)-Fe(1)-C(10)	155.87(12)	C(2)-Fe(1)-C(3)	40.80(14)
C(2)-Fe(1)-C(4)	67.73(13)	C(2)-Fe(1)-C(5)	67.37(12)
C(2)-Fe(1)-C(6)	158.41(14)	C(2)-Fe(1)-C(7)	123.95(12)
C(2)-Fe(1)-C(8)	110.17(11)	C(2)-Fe(1)-C(9)	125.21(12)
C(2)-Fe(1)-C(10)	160.53(13)	C(3)-Fe(1)-C(4)	39.96(13)
C(3)-Fe(1)-C(5)	66.89(12)	C(3)-Fe(1)-C(6)	158.27(13)
C(3)-Fe(1)-C(7)	160.19(14)	C(3)-Fe(1)-C(8)	124.55(12)
C(3)-Fe(1)-C(9)	108.28(10)	C(3)-Fe(1)-C(10)	122.84(12)
C(5)-Fe(1)-C(4)	39.43(12)	C(4)-Fe(1)-C(6)	122.16(12)
C(4)-Fe(1)-C(7)	158.60(12)	C(4)-Fe(1)-C(8)	158.90(11)
C(4)-Fe(1)-C(9)	121.92(10)	C(4)-Fe(1)-C(10)	106.31(11)
C(5)-Fe(1)-C(6)	107.06(11)	C(5)-Fe(1)-C(7)	124.04(11)
C(5)-Fe(1)-C(8)	160.98(11)	C(5)-Fe(1)-C(9)	156.38(11)

C(5)-Fe(1)-C(10)	120.62(11)	C(6)-Fe(1)-C(7)	40.47(9)
C(6)-Fe(1)-C(8)	67.87(9)	C(6)-Fe(1)-C(9)	68.38(9)
C(6)-Fe(1)-C(10)	40.57(9)	C(7)-Fe(1)-C(8)	40.32(9)
C(7)-Fe(1)-C(9)	68.47(9)	C(10)-Fe(1)-C(7)	68.36(9)
C(9)-Fe(1)-C(8)	40.75(8)	C(10)-Fe(1)-C(8)	68.31(9)
C(10)-Fe(1)-C(9)	40.84(8)	C(12)-C(11)-N(1)	108.13(19)

Table 3. Atomic coordinates and equivalent isotropic atomic displacement parameters (Å2) for compound **3a**

x/a	y/b	z/c	U(eq)	
Fe1	0.60764(4)	0.819163(13)	0.89503(2)	0.02902(10)
N1	0.1668(3)	0.65836(9)	0.92115(15)	0.0380(4)
N2	0.1455(3)	0.60866(9)	0.99021(15)	0.0383(4)
N3	0.3562(3)	0.59507(8)	0.03411(13)	0.0296(3)
C15	0.3880(3)	0.54418(9)	0.11584(15)	0.0309(4)
C1	0.8522(5)	0.82542(13)	0.0197(2)	0.0576(7)
C2	0.8574(5)	0.88096(13)	0.9534(2)	0.0563(6)
C3	0.6513(5)	0.91311(13)	0.9552(2)	0.0631(7)
C4	0.5087(5)、	0.87590(17)	0.0247(2)	0.0712(8)
C5	0.6385(6)	0.82123(15)	0.0650(2)	0.0672(9)
C6	0.4627(4)	0.82389(10)	0.73646(17)	0.0376(4)
C7	0.6815(4)	0.79487(11)	0.73453(17)	0.0374(4)
C8	0.6843(3)	0.73734(10)	0.80281(16)	0.0333(4)
C9	0.4643(3)	0.73045(9)	0.84788(15)	0.0294(4)
C10	0.3899(3)	0.67711(9)	0.92021(15)	0.0292(4)
C11	0.2114(4)	0.50201(10)	0.13935(18)	0.0400(5)
C12	0.2546(4)	0.45316(11)	0.2180(2)	0.0471(5)
C13	0.4669(4)	0.44904(13)	0.2694(2)	0.0513(6)
C14	0.6286(4)	0.49374(13)	0.2400(2)	0.0537(6)
N4	0.5939(3)	0.54174(10)	0.16311(16)	0.0454(4)
C16	0.5120(3)	0.63705(9)	0.99253(16)	0.0304(4)
C17	0.3279(3)	0.78421(10)	0.80562(16)	0.0339(4)

Table 4. Torsion angles (°) for compound **3a**

C10-N1-N2-N3	-0.1(2)	N1-N2-N3-C16	0.3(2)	N1-N2-N3-C15	177.98(16)
N2-N3-C15-N4	-171.17(18)	C16-N3-C15-N4	6.0(3)	N2-N3-C15-C11	9.3(3)
C16-N3-C15-C11	-173.53(19)	C4-Fe1-C1-C2	-81.0(2)	C5-Fe1-C1-C2	-119.3(2)
C3-Fe1-C1-C2	-36.70(18)	C7-Fe1-C1-C2	77.33(19)	C8-Fe1-C1-C2	118.68(17)

C6-Fe1-C1-C2	46.5(3)	C9-Fe1-C1-C2	160.39(15)	C17-Fe1-C1-C2	-169.4(2)
C4-Fe1-C1-C5	38.22(18)	C2-Fe1-C1-C5	119.3(2)	C3-Fe1-C1-C5	82.57(19)
C7-Fe1-C1-C5	-163.41(16)	C8-Fe1-C1-C5	-122.05(17)	C6-Fe1-C1-C5	165.8(2)
C9-Fe1-C1-C5	-80.34(19)	C17-Fe1-C1-C5	-50.1(3)	C5-C1-C2-C3	0.2(3)
Fe1-C1-C2-C3	59.88(18)	C5-C1-C2-Fe1	-59.65(18)	C4-Fe1-C2-C1	82.0(2)
C5-Fe1-C2-C1	37.56(18)	C3-Fe1-C2-C1	120.3(3)	C7-Fe1-C2-C1	-120.21(17)
C8-Fe1-C2-C1	-78.06(19)	C6-Fe1-C2-C1	-161.38(16)	C9-Fe1-C2-C1	-45.3(3)
C17-Fe1-C2-C1	168.3(3)	C4-Fe1-C2-C3	-38.27(19)	C5-Fe1-C2-C3	-82.7(2)
C1-Fe1-C2-C3	-120.3(3)	C7-Fe1-C2-C3	119.52(17)	C8-Fe1-C2-C3	161.68(16)
C6-Fe1-C2-C3	78.4(2)	C9-Fe1-C2-C3	-165.6(2)	C17-Fe1-C2-C3	48.0(4)
C1-C2-C3-C4	-0.6(3)	Fe1-C2-C3-C4	59.16(17)	C1-C2-C3-Fe1	-59.76(18)
C4-Fe1-C3-C2	118.9(2)	C5-Fe1-C3-C2	80.12(19)	C1-Fe1-C3-C2	36.63(17)
C7-Fe1-C3-C2	-77.96(19)	C8-Fe1-C3-C2	-41.5(3)	C6-Fe1-C3-C2	-120.84(17)
C9-Fe1-C3-C2	160.6(3)	C17-Fe1-C3-C2	-162.53(15)	C2-Fe1-C3-C4	-118.9(2)
C5-Fe1-C3-C4	-38.74(18)	C1-Fe1-C3-C4	-82.23(19)	C7-Fe1-C3-C4	163.18(16)
C8-Fe1-C3-C4	-160.3(2)	C6-Fe1-C3-C4	120.30(18)	C9-Fe1-C3-C4	41.7(4)
C17-Fe1-C3-C4	78.6(2)	C2-C3-C4-C5	0.7(3)	Fe1-C3-C4-C5	60.32(17)
C2-C3-C4-Fe1	-59.59(18)	C2-Fe1-C4-C5	-80.33(18)	C1-Fe1-C4-C5	-37.51(17)
C3-Fe1-C4-C5	-117.5(2)	C7-Fe1-C4-C5	-159.5(2)	C8-Fe1-C4-C5	38.2(4)
C6-Fe1-C4-C5	162.90(16)	C9-Fe1-C4-C5	76.70(19)	C17-Fe1-C4-C5	119.94(17)
C2-Fe1-C4-C3	37.13(16)	C5-Fe1-C4-C3	117.5(2)	C1-Fe1-C4-C3	79.96(18)
C7-Fe1-C4-C3	-42.0(3)	C8-Fe1-C4-C3	155.7(3)	C6-Fe1-C4-C3	-79.64(19)
C9-Fe1-C4-C3	-165.83(14)	C17-Fe1-C4-C3	-122.60(16)	C2-C1-C5-C4	0.2(3)
Fe1-C1-C5-C4	-59.58(18)	C2-C1-C5-Fe1	59.81(18)	C3-C4-C5-C1	-0.6(3)
Fe1-C4-C5-C1	59.95(18)	C3-C4-C5-Fe1	-60.54(17)	C4-Fe1-C5-C1	-118.8(2)
C2-Fe1-C5-C1	-37.06(16)	C3-Fe1-C5-C1	-80.03(18)	C7-Fe1-C5-C1	40.8(3)
C8-Fe1-C5-C1	75.50(18)	C6-Fe1-C5-C1	-164.7(2)	C9-Fe1-C5-C1	118.17(16)
C17-Fe1-C5-C1	160.39(14)	C2-Fe1-C5-C4	81.70(19)	C1-Fe1-C5-C4	118.8(2)
C3-Fe1-C5-C4	38.73(17)	C7-Fe1-C5-C4	159.6(2)	C8-Fe1-C5-C4	-165.75(16)
C6-Fe1-C5-C4	-45.9(4)	C9-Fe1-C5-C4	-123.07(17)	C17-Fe1-C5-C4	-80.86(19)
C4-Fe1-C6-C17	-81.22(17)	C2-Fe1-C6-C17	-165.26(14)	C5-Fe1-C6-C17	-46.8(3)
C1-Fe1-C6-C17	161.0(2)	C3-Fe1-C6-C17	-124.02(14)	C7-Fe1-C6-C17	119.02(17)
C8-Fe1-C6-C17	81.58(12)	C9-Fe1-C6-C17	37.51(11)	C4-Fe1-C6-C7	159.76(15)
C2-Fe1-C6-C7	75.72(16)	C5-Fe1-C6-C7	-165.8(3)	C1-Fe1-C6-C7	41.9(3)
C3-Fe1-C6-C7	116.97(15)	C8-Fe1-C6-C7	-37.44(12)	C9-Fe1-C6-C7	-81.51(13)
C17-Fe1-C6-C7	-119.02(17)	C17-C6-C7-C8	-0.3(2)	Fe1-C6-C7-C8	59.35(14)
C17-C6-C7-Fe1	-59.67(14)	C4-Fe1-C7-C6	-51.3(3)	C2-Fe1-C7-C6	-122.83(14)
C5-Fe1-C7-C6	167.0(2)	C1-Fe1-C7-C6	-163.26(14)	C3-Fe1-C7-C6	-82.21(16)
C8-Fe1-C7-C6	119.73(17)	C9-Fe1-C7-C6	81.77(13)	C17-Fe1-C7-C6	37.67(12)
C4-Fe1-C7-C8	-171.0(2)	C2-Fe1-C7-C8	117.44(14)	C5-Fe1-C7-C8	47.3(3)
C1-Fe1-C7-C8	77.01(15)	C3-Fe1-C7-C8	158.06(14)	C6-Fe1-C7-C8	-119.73(17)
C9-Fe1-C7-C8	-37.95(11)	C17-Fe1-C7-C8	-82.06(13)	C6-C7-C8-C9	0.0(2)
Fe1-C7-C8-C9	59.47(13)	C6-C7-C8-Fe1	-59.47(15)	C4-Fe1-C8-C7	170.1(3)
C2-Fe1-C8-C7	-80.35(15)	C5-Fe1-C8-C7	-161.14(15)	C1-Fe1-C8-C7	-120.72(15)

C3-Fe1-C8-C7	-51.0(3)	C6-Fe1-C8-C7	37.34(12)	C9-Fe1-C8-C7	119.03(17)
C17-Fe1-C8-C7	80.91(13)	C4-Fe1-C8-C9	51.1(3)	C2-Fe1-C8-C9	160.62(13)
C5-Fe1-C8-C9	79.83(16)	C1-Fe1-C8-C9	120.25(14)	C3-Fe1-C8-C9	-170.1(2)
C7-Fe1-C8-C9	-119.03(17)	C6-Fe1-C8-C9	-81.69(12)	C17-Fe1-C8-C9	-38.12(11)
C7-C8-C9-C17	0.3(2)	Fe1-C8-C9-C17	59.68(13)	C7-C8-C9-C10	178.25(18)
Fe1-C8-C9-C10	-122.40(19)	C7-C8-C9-Fe1	-59.36(14)	C4-Fe1-C9-C8	-161.45(15)
C2-Fe1-C9-C8	-45.4(3)	C5-Fe1-C9-C8	-119.42(15)	C1-Fe1-C9-C8	-77.55(16)
C3-Fe1-C9-C8	166.4(3)	C7-Fe1-C9-C8	37.74(11)	C6-Fe1-C9-C8	81.43(12)
C17-Fe1-C9-C8	118.56(16)	C4-Fe1-C9-C17	79.99(17)	C2-Fe1-C9-C17	-164.0(2)
C5-Fe1-C9-C17	122.02(15)	C1-Fe1-C9-C17	163.89(14)	C3-Fe1-C9-C17	47.9(4)
C7-Fe1-C9-C17	-80.82(12)	C8-Fe1-C9-C17	-118.56(16)	C6-Fe1-C9-C17	-37.13(12)
C4-Fe1-C9-C10	-40.8(2)	C2-Fe1-C9-C10	75.2(3)	C5-Fe1-C9-C10	1.2(2)
C1-Fe1-C9-C10	43.1(2)	C3-Fe1-C9-C10	-72.9(4)	C7-Fe1-C9-C10	158.37(19)
C8-Fe1-C9-C10	120.6(2)	C6-Fe1-C9-C10	-157.93(19)	C17-Fe1-C9-C10	-120.8(2)
N2-N1-C10-C16	-0.2(2)	N2-N1-C10-C9	178.25(17)	C8-C9-C10-C16	26.1(3)
C17-C9-C10-C16	-156.4(2)	Fe1-C9-C10-C16	-65.0(3)	C8-C9-C10-N1	-151.96(19)
C17-C9-C10-N1	25.6(3)	Fe1-C9-C10-N1	117.03(18)	N4-C15-C11-C12	-0.5(3)
N3-C15-C11-C12	178.95(19)	C15-C11-C12-C13	0.7(3)	C11-C12-C13-C14	-0.3(4)
C12-C13-C14-N4	-0.3(4)	C11-C15-N4-C14	0.0(3)	N3-C15-N4-C14	-179.5(2)
C13-C14-N4-C15	0.5(4)	N2-N3-C16-C10	-0.4(2)	C15-N3-C16-C10	-177.80(17)
N1-C10-C16-N3	0.3(2)	C9-C10-C16-N3	-177.87(18)	C7-C6-C17-C9	0.5(2)
Fe1-C6-C17-C9	-58.83(13)	C7-C6-C17-Fe1	59.35(14)	C8-C9-C17-C6	-0.5(2)
C10-C9-C17-C6	-178.45(18)	Fe1-C9-C17-C6	58.95(14)	C8-C9-C17-Fe1	-59.47(13)
C10-C9-C17-Fe1	122.60(19)	C4-Fe1-C17-C6	119.15(16)	C2-Fe1-C17-C6	40.3(3)
C5-Fe1-C17-C6	162.52(15)	C1-Fe1-C17-C6	-160.7(3)	C3-Fe1-C17-C6	76.18(17)
C7-Fe1-C17-C6	-37.79(12)	C8-Fe1-C17-C6	-81.70(13)	C9-Fe1-C17-C6	-119.87(17)
C4-Fe1-C17-C9	-120.98(16)	C2-Fe1-C17-C9	160.2(3)	C5-Fe1-C17-C9	-77.61(17)
C1-Fe1-C17-C9	-40.8(3)	C3-Fe1-C17-C9	-163.95(14)	C7-Fe1-C17-C9	82.08(12)
C8-Fe1-C17-C9	38.17(11)	C6-Fe1-C17-C9	119.87(17)		

Table 5. Anisotropic atomic displacement parameters (Å2) for compound **3a**

	U11	U22	U33	U23	U13	U12
Fe1	0.03052(15)	0.02916(15)	0.02717(15)	0.00129(10)	-0.00173(10)	-0.00032(10)
N1	0.0296(8)	0.0360(9)	0.0480(10)	0.0076(8)	-0.0040(7)	-0.0020(7)
N2	0.0295(8)	0.0381(9)	0.0470(10)	0.0078(8)	-0.0045(7)	-0.0030(7)
N3	0.0283(7)	0.0293(8)	0.0309(8)	-0.0006(6)	-0.0010(6)	-0.0013(6)
C15	0.0353(9)	0.0295(9)	0.0280(9)	-0.0022(7)	0.0025(7)	0.0015(7)
C1	0.0599(16)	0.0560(15)	0.0545(15)	-0.0102(12)	-0.0274(13)	0.0026(12)
C2	0.0542(14)	0.0577(16)	0.0566(15)	-0.0107(12)	-0.0011(12)	-0.0207(12)
C3	0.092(2)	0.0344(12)	0.0607(16)	-0.0118(10)	-0.0228(12)	0.0056(12)
C4	0.0460(14)	0.105(2)	0.0632(17)	-0.0496(15)	0.0056(11)	-0.0008(14)
C5	0.104(2)	0.0678(18)	0.0297(12)	0.0002(11)	-0.0044(13)	-0.0361(17)

C6	0.0457(11)	0.0350(10)	0.0314(10)	0.0056(8)	-0.0073(8)	0.0038(8)
C7	0.0416(11)	0.0410(11)	0.0298(10)	0.0046(8)	0.0057(8)	-0.0009(9)
C8	0.0356(10)	0.0325(10)	0.0321(10)	-0.0008(8)	0.0046(8)	0.0041(8)
C9	0.0296(9)	0.0299(9)	0.0283(9)	-0.0020(7)	-0.0024(7)	0.0002(7)
C10	0.0281(8)	0.0284(9)	0.0310(9)	-0.0028(7)	0.0005(7)	0.0000(7)
C11	0.0416(11)	0.0362(11)	0.0417(11)	0.0033(9)	-0.0029(9)	-0.0065(8)
C12	0.0571(14)	0.0372(11)	0.0471(13)	0.0067(10)	0.0051(10)	-0.0047(10)
C13	0.0632(15)	0.0488(14)	0.0422(12)	0.0141(10)	0.0052(11)	0.0122(11)
C14	0.0443(13)	0.0649(16)	0.0513(14)	0.0170(12)	-0.0043(10)	0.0073(11)
N4	0.0359(9)	0.0544(11)	0.0454(10)	0.0118(9)	-0.0038(8)	-0.0022(8)
C16	0.0264(8)	0.0332(9)	0.0314(9)	0.0021(7)	0.0002(7)	-0.0025(7)
C17	0.0298(9)	0.0373(10)	0.0339(10)	-0.0012(8)	-0.0072(7)	0.0030(8)

Table 6. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å2) for compound **3a**

	x/a	y/b	z/c	U(eq)
H1	0.9710	0.7957	1.0321	0.069
H2	0.9813	0.8948	0.9135	0.068
H3	0.6126	0.9521	0.9177	0.076
H4	0.3591	0.8855	1.0407	0.085
H5	0.5897	0.7884	1.1131	0.081
H6	0.4160	0.8622	0.6990	0.045
H7	0.8030	0.8109	0.6951	0.045
H8	0.8073	0.7092	0.8159	0.04
H11	0.0692	0.5064	1.1036	0.048
H12	0.1414	0.4233	1.2360	0.056
H13	0.4998	0.4166	1.3229	0.062
H14	0.7716	0.4908	1.2755	0.064
H16	0.6673	0.6383	1.0095	0.036
H17	0.1692	0.7926	0.8227	0.041