Copper-Catalyzed Direct Alkylation of 1,3-Azoles with *N*-Tosylhydrazones Bearing a Ferrocenyl Group: A Novel Method for the Synthesis of Ferrocenyl-based Ligands

Supporting Information

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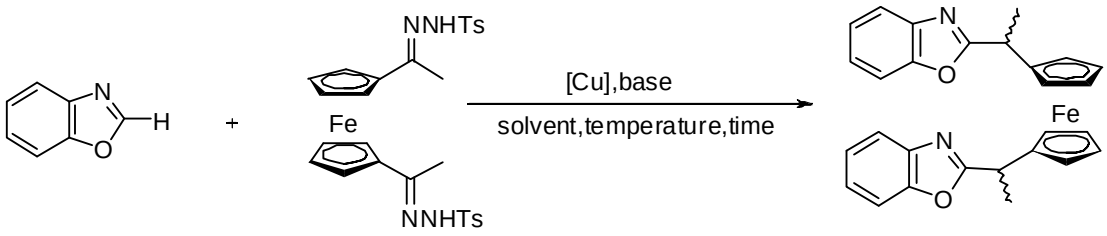
1. General Procedure for Cu-Catalyzed Reaction

The oxazoles (1.0 mmol, 4 equiv), *N*-tosylhydrazones (0.25 mmol), lithium *t*-butoxide (1.5 mmol, 6equiv), copper bromide (0.125 mmol, 50 mol%) and 10 mL toluene were mixed in a dry reaction tube. The mixture was stirred at 110 °C under nitrogen atmosphere for 12 hours. When the reaction was completed, the crude reaction mixture was allowed to reach room temperature, the solvent was eliminated. Then ethyl acetate and ammonia were added and the layers were separated. The aqueous phase was extracted three times with ethyl acetate. The combined organic layers were washed with water and then dried over $MgSO_4$ and filtered. The solvents were evaporated under reduced pressure and the residue was purified by flash chromatography on silica gel.

2. Table 1. Optimization of Reaction Conditions[a]



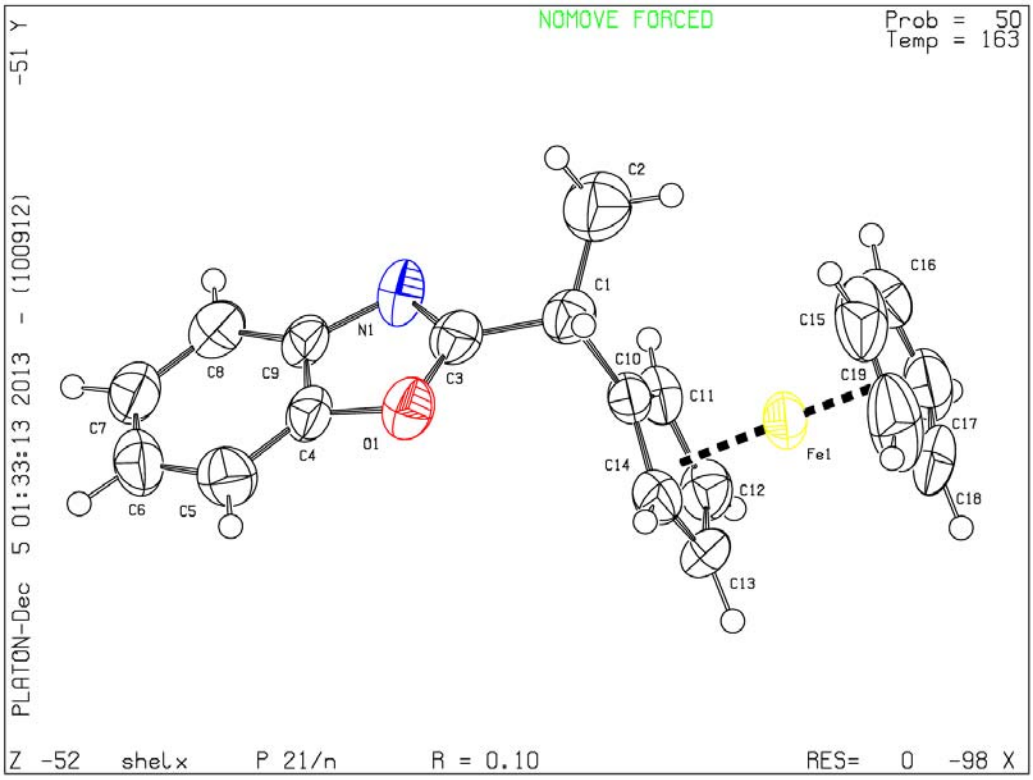
1a **4a** **5a**

entry	1a : 4a	cat.(mol%)	solvent(ml)	base (equiv)	t(^{o}C)	yield [b] (%)
1	4:1	CuI(50%)	toluene(5)	LiO^{t}Bu(6)	120	13
2	4:1	CuBr(50%)	toluene(5)	LiO^{t}Bu(6)	120	47
3	4:1	CuCl(50%)	toluene(5)	LiO^{t}Bu(6)	120	38
4	4:1	$Cu(OTf)_2$(50%)	toluene(5)	LiO^{t}Bu(6)	120	39
5	4:1	$Ni(PPh_3)_2Br_2$(50%)	toluene(5)	LiO^{t}Bu(6)	120	trace
6	4:1	CuBr(30%)	toluene(5)	LiO^{t}Bu(6)	120	20
7	4:1	CuBr(40%)	toluene(5)	LiO^{t}Bu(6)	120	42
8	3:1	CuBr(50%)	toluene(5)	LiO^{t}Bu(6)	120	44
9	7:1	CuBr(50%)	toluene(5)	LiO^{t}Bu(6)	120	48
10	4:1	CuBr(50%)	toluene(5)	LiO^{t}Bu(4)	120	42
11	4:1	CuBr(50%)	toluene(5)	LiO^{t}Bu(5)	120	44
12	4:1	CuBr(50%)	toluene(5)	LiO^{t}Bu(8)	120	48
13	4:1	CuBr(50%)	dioxane(5)	LiO^{t}Bu(6)	120	38
14	4:1	CuBr(50%)	toluene(4)+ DMF(1)	LiO^{t}Bu(6)	120	25
15	4:1	CuBr(50%)	toluene(4)+ DMF(0.2)	LiO^{t}Bu(6)	120	40
16	4:1	CuBr(50%)	toluene(10)	LiO^{t}Bu(6)	120	67
17	4:1	CuBr(50%)	toluene(20)	LiO^{t}Bu(6)	120	69
18	4:1	CuBr(50%)	toluene(10)	LiO^{t}Bu(6)	90	69
19	4:1	CuBr(50%)	toluene(10)	LiO^{t}Bu(6)	100	73
20	**4:1**	**CuBr(50%)**	**toluene(10)**	**LiO^{t}Bu(6)**	**110**	**75**
21	4:1	CuBr(50%)	toluene(10)	LiO^{t}Bu(6)	120	67

[a] Reactions were carried out with 1.0 mmol of **1a** and 0.25 mmol of **4a** in the presence of 50 mol% of catalyst and 6 equiv of base at 110℃ temperature under a N_2 atmosphere for 12 h.

[b] Yield of isolated product after chromatography.

3. Molecular structure of compound 3a



Crystal Structure Report for work

A specimen of C19H17FeNO, approximate dimensions 0.120 mm x 0.150 mm x 0.220 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured.
The integration of the data using a monoclinic unit cell yielded a total of 8372 reflections to a maximum θ angle of 25.00° (0.84 Å resolution), of which 2643 were independent (average redundancy 3.168, completeness = 98.3%, Rint = 2.70%) and 2499 (94.55%) were greater than 2σ(F2). The final cell constants of a = 6.121(5) Å, b = 30.224(5) Å, c = 8.524(5) Å, α = 90.000(5)°, β = 104.105(5)°, γ = 90.000(5)°, volume = 1529.4(16) Å3, are based upon the refinement of the XYZ-centroids of reflections above 20 σ(I). The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.8120 and 0.8910.

The final anisotropic full-matrix least-squares refinement on F2 with 199 variables converged at R1 = 9.65%, for the observed data and wR2 = 19.37% for all data. The goodness-of-fit was 1.140. The largest peak in the final difference electron density synthesis was 0.678 e-/Å3 and the largest hole was -0.860 e-/Å3 with an RMS deviation of 0.087 e-/Å3. On the basis of the final model, the calculated density was 1.438 g/cm3 and F(000), 688 e-.

Table 1. Sample and crystal data for work.

Identification code	work
Chemical formula	C19H17FeNO
Formula weight	331.18
Temperature	163(2) K
Wavelength	0.71073 Å
Crystal size	0.120 x 0.150 x 0.220 mm
Crystal system	monoclinic
Space group	P 1 21/n 1
Unit cell dimensions	a = 6.121(5) Å α = 90.000(5)°
	b = 30.224(5) Å β = 104.105(5)°
	c = 8.524(5) Å ɣ = 90.000(5)°
Volume	1529.4(16) Å3
Z	4
Density (calculated)	1.438 g/cm3
Absorption coefficient	0.986 mm-1
F(000)	688

Table 2. Data collection and structure refinement for work.

Theta range for data collection	1.35 to 25.00°
Index ranges	-7<=h<=7, -34<=k<=35, -10<=l<=9
Reflections collected	8372
Independent reflections	2643 [R(int) = 0.0270]
Max. and min. transmission	0.8910 and 0.8120
Refinement method	Full-matrix least-squares on F2
Refinement program	SHELXL-2013 (Sheldrick, 2013)
Function minimized	Σ w(Fo2 - Fc2)2
Data / restraints / parameters	2643 / 0 / 199
Goodness-of-fit on F2	1.140
Final R indices	2499 data; I>2σ(I) R1 = 0.0965, wR2 = 0.1923
	all data R1 = 0.0997, wR2 = 0.1937
Weighting scheme	w=1/[σ2(Fo2)+12.0000P] where P=(Fo2+2Fc2)/3
Largest diff. peak and hole	0.678 and -0.860 eÅ-3
R.M.S. deviation from mean	0.087 eÅ-3

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

work.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x/a	y/b	z/c	U(eq)
C1	0.2831(13)	0.1476(2)	0.0909(9)	0.0503(18)
C2	0.442(2)	0.1714(3)	0.0069(12)	0.091(3)
C3	0.1644(13)	0.1099(3)	0.9894(9)	0.0527(19)
C4	0.9143(13)	0.0596(3)	0.9060(8)	0.0490(18)
C5	0.7318(14)	0.0316(3)	0.8798(11)	0.063(2)
C6	0.7363(16)	0.9967(3)	0.7731(11)	0.067(3)
C7	0.9096(16)	0.9908(3)	0.7025(10)	0.062(2)
C8	0.0923(16)	0.0192(3)	0.7291(10)	0.062(2)
C9	0.0884(13)	0.0545(2)	0.8330(8)	0.0480(18)
C10	0.3999(12)	0.1288(2)	0.2543(8)	0.0444(17)
C11	0.6169(12)	0.1087(2)	0.2985(9)	0.0478(18)
C12	0.6510(14)	0.0927(3)	0.4573(10)	0.056(2)
C13	0.4581(16)	0.1019(3)	0.5133(9)	0.063(2)
C14	0.3025(13)	0.1235(3)	0.3884(9)	0.054(2)
C15	0.598(2)	0.2252(3)	0.4181(15)	0.093(4)
C16	0.8151(19)	0.2080(3)	0.4346(13)	0.076(3)
C17	0.8827(17)	0.1882(3)	0.5827(13)	0.076(3)
C18	0.718(2)	0.1919(4)	0.6628(12)	0.089(4)
C19	0.540(2)	0.2152(4)	0.5659(19)	0.102(4)
N1	0.2500(10)	0.0870(2)	0.8885(8)	0.0536(16)
O1	0.9650(9)	0.09683(18)	0.0058(6)	0.0568(14)
Fe1	0.59388(19)	0.15908(4)	0.45441(13)	0.0515(4)

Table 4. Bond lengths (Å) for work.

C1-C3	1.504(10)	C1-C10	1.513(10)	C1-C2
1.521(13)				
C3-N1	1.309(10)	C3-O1	1.323(9)	C4-C9
1.367(11)				
C4-C5	1.377(11)	C4-O1	1.398(9)	C5-C6
1.397(12)				
C6-C7	1.353(13)	C7-C8	1.383(12)	C8-C9
1.390(11)				
C9-N1	1.394(9)	C10-C14	1.421(11)	C10-C11
1.426(10)				
C10-Fe1	2.042(7)	C11-C12	1.404(11)	C11-Fe1

2.049(7)
C12-C13 1.404(12) C12-Fe1 2.035(8) C13-C14 1.405(11)
C13-Fe1 2.033(8) C14-Fe1 2.041(7) C15-C16 1.399(15)
C15-C19 1.422(17) C15-Fe1 2.024(9) C16-C17 1.367(14)
C16-Fe1 2.039(9) C17-C18 1.354(14) C17-Fe1 2.038(9)
C18-C19 1.387(16) C18-Fe1 2.015(8) C19-Fe1 2.011(10)

Table 5. Bond angles (°) for work.

C3-C1-C10 107.4(6) C3-C1-C2 111.3(7) C10-C1-C2 113.5(7)
N1-C3-O1 116.0(7) N1-C3-C1 124.3(7) O1-C3-C1 119.7(7)
C9-C4-C5 123.1(7) C9-C4-O1 106.7(7) C5-C4-O1 130.2(8)
C4-C5-C6 115.3(8) C7-C6-C5 122.0(9) C6-C7-C8 122.4(8)
C7-C8-C9 116.2(8) C4-C9-C8 121.0(8) C4-C9-N1 109.4(7)
C8-C9-N1 129.5(8) C14-C10-C11 106.6(7) C14-C10-C1 125.9(7) C11-C10-C1 127.2(7) C14-C10-Fe1 69.6(4)
C11-C10-Fe1 69.9(4)
C1-C10-Fe1 130.5(5) C12-C11-C10 108.1(7) C12-C11-Fe1 69.3(5)
C10-C11-Fe1 69.4(4) C11-C12-C13 108.7(7) C11-C12-Fe1 70.4(4)
C13-C12-Fe1 69.7(5) C12-C13-C14 107.8(7) C12-C13-Fe1 69.9(5)
C14-C13-Fe1 70.1(5) C13-C14-C10 108.7(7) C13-C14-Fe1 69.5(5)
C10-C14-Fe1 69.7(4) C16-C15-C19 105.6(10) C16-C15-Fe1 70.5(5)
C19-C15-Fe1 68.9(6) C17-C16-C15 108.8(10) C17-C16-Fe1 70.4(5)
C15-C16-Fe1 69.3(6) C18-C17-C16 109.5(11) C18-C17-Fe1 69.6(5)
C16-C17-Fe1 70.5(5) C17-C18-C19 108.3(11) C17-C18-Fe1 71.4(5)
C19-C18-Fe1 69.7(5) C18-C19-C15 107.8(10) C18-C19-Fe1 70.0(6)

C15-C19-Fe1	69.8(6)	C3-N1-C9	103.4(7)	C3-O1-C4	104.5(6)
C19-Fe1-C18	40.3(5)	C19-Fe1-C15	41.3(5)	C18-Fe1-C15	68.4(5)
C19-Fe1-C13	118.5(5)	C18-Fe1-C13	106.8(4)	C15-Fe1-C13	154.0(5)
C19-Fe1-C12	150.9(5)	C18-Fe1-C12	116.6(4)	C15-Fe1-C12	165.1(5)
C13-Fe1-C12	40.4(3)	C19-Fe1-C17	66.6(4)	C18-Fe1-C17	39.0(4)
C15-Fe1-C17	67.2(4)	C13-Fe1-C17	126.0(4)	C12-Fe1-C17	107.2(4)
C19-Fe1-C16	67.4(5)	C18-Fe1-C16	66.5(4)	C15-Fe1-C16	40.3(4)
C13-Fe1-C16	163.0(4)	C12-Fe1-C16	126.9(4)	C17-Fe1-C16	39.2(4)
C19-Fe1-C14	110.0(4)	C18-Fe1-C14	128.2(4)	C15-Fe1-C14	121.4(4)
C13-Fe1-C14	40.3(3)	C12-Fe1-C14	67.7(3)	C17-Fe1-C14	163.9(4)
C16-Fe1-C14	155.8(4)	C19-Fe1-C10	130.1(5)	C18-Fe1-C10	167.1(4)
C15-Fe1-C10	110.0(4)	C13-Fe1-C10	68.6(3)	C12-Fe1-C10	68.4(3)
C17-Fe1-C10	153.3(4)	C16-Fe1-C10	121.2(4)	C14-Fe1-C10	40.7(3)
C19-Fe1-C11	168.2(5)	C18-Fe1-C11	150.2(5)	C15-Fe1-C11	129.0(5)
C13-Fe1-C11	68.0(3)	C12-Fe1-C11	40.2(3)	C17-Fe1-C11	118.8(4)
C16-Fe1-C11	109.5(4)	C14-Fe1-C11	67.9(3)	C10-Fe1-C11	40.8(3)

Table 6. Anisotropic atomic displacement parameters (Å2) for work.
The anisotropic atomic displacement factor exponent takes the form: -2π2[h2 a*2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C1	0.056(5)	0.047(4)	0.045(4)	0.003(3)	0.007(3)	0.005(3)
C2	0.123(10)	0.071(6)	0.072(7)	0.010(5)	0.007(6)	0.000(6)
C3	0.047(4)	0.064(5)	0.040(4)	0.008(4)	-0.002(3)	

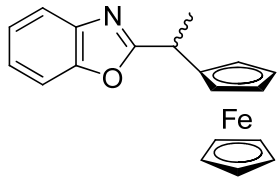
0.000(4)
C4 0.052(4) 0.053(4) 0.036(4) -0.003(3) -0.001(3)
0.000(4)
C5 0.049(5) 0.073(6) 0.065(5) 0.004(5) 0.010(4)
-0.003(4)
C6 0.073(6) 0.052(5) 0.062(5) 0.001(4) -0.012(5)
-0.010(4)
C7 0.074(6) 0.058(5) 0.047(5) -0.004(4) 0.003(4)
0.000(5)
C8 0.072(6) 0.068(6) 0.046(5) -0.002(4) 0.013(4)
-0.002(5)
C9 0.053(4) 0.053(4) 0.032(4) 0.002(3) -0.001(3)
-0.001(4)
C10 0.047(4) 0.039(4) 0.042(4) -0.001(3) 0.001(3)
-0.008(3)
C11 0.042(4) 0.042(4) 0.054(5) -0.006(3) 0.002(3)
-0.002(3)
C12 0.056(5) 0.050(4) 0.054(5) 0.003(4) -0.004(4)
0.002(4)
C13 0.092(7) 0.063(5) 0.031(4) -0.002(4) 0.013(4)
-0.020(5)
C14 0.046(4) 0.062(5) 0.055(5) -0.007(4) 0.013(4)
-0.016(4)
C15 0.124(10) 0.038(5) 0.090(8) -0.007(5) -0.025(8)
-0.003(6)
C16 0.091(8) 0.061(6) 0.076(7) -0.008(5) 0.024(6)
-0.033(6)
C17 0.067(6) 0.079(7) 0.080(7) -0.029(6) 0.013(5)
-0.026(5)
C18 0.114(9) 0.101(8) 0.049(5) -0.037(6) 0.013(6)
-0.042(7)
C19 0.077(7) 0.091(8) 0.139(12) -0.073(8) 0.030(8)
-0.010(6)
N1 0.049(4) 0.058(4) 0.049(4) -0.017(3) 0.002(3)
0.007(3)
O1 0.052(3) 0.065(4) 0.051(3) -0.011(3) 0.010(3)
0.004(3)
Fe1 0.0553(7) 0.0461(6) 0.0468(6) -0.0059(5) 0.0003(5)
-0.0103(6)

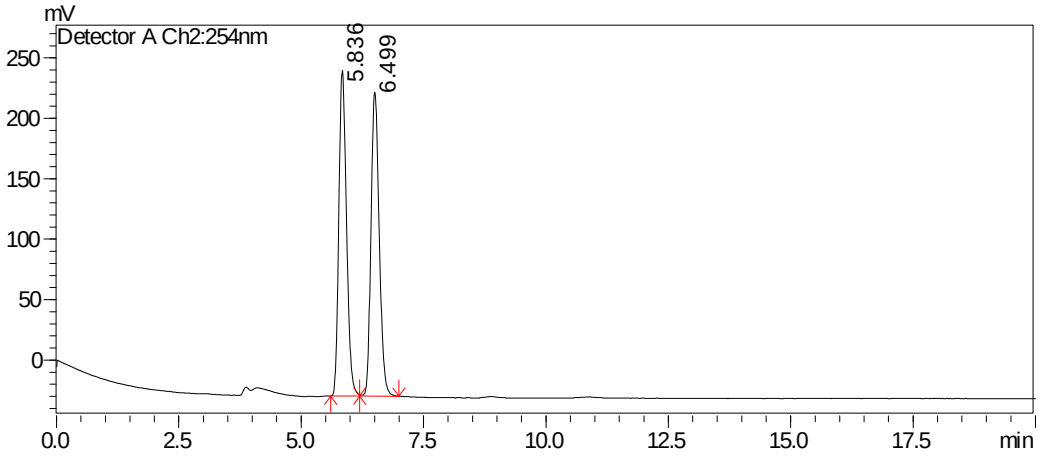
Table 7. Hydrogen atomic coordinates and isotropic atomic displacement parameters (Å2) for work.

	x/a	y/b	z/c	U(eq)
H1	0.1667	0.1692	0.1069	0.06
H2A	0.5157	0.1958	0.0753	0.137
H2B	0.3556	0.1832	-0.0970	0.137
H2C	0.5556	0.1507	-0.0117	0.137
H5	-0.3886	0.0356	-0.0693	0.076
H6	-0.3861	-0.0236	-0.2505	0.081
H7	-0.0944	-0.0336	-0.3681	0.074
H8	0.2136	0.0148	-0.3207	0.075
H11	0.7205	0.1064	0.2320	0.057
H12	0.7826	0.0781	0.5170	0.067
H13	0.4367	0.0947	0.6170	0.075
H14	0.1562	0.1331	0.3929	0.065
H15	0.5090	0.2404	0.3273	0.112
H16	0.9010	0.2098	0.3559	0.091
H17	1.0237	0.1740	0.6232	0.091
H18	0.7228	0.1805	0.7675	0.107
H19	0.4040	0.2232	0.5933	0.122

HPLC Charts of Products

(±)





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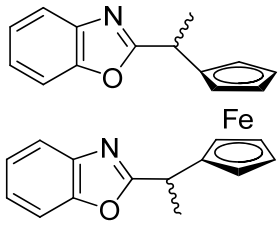
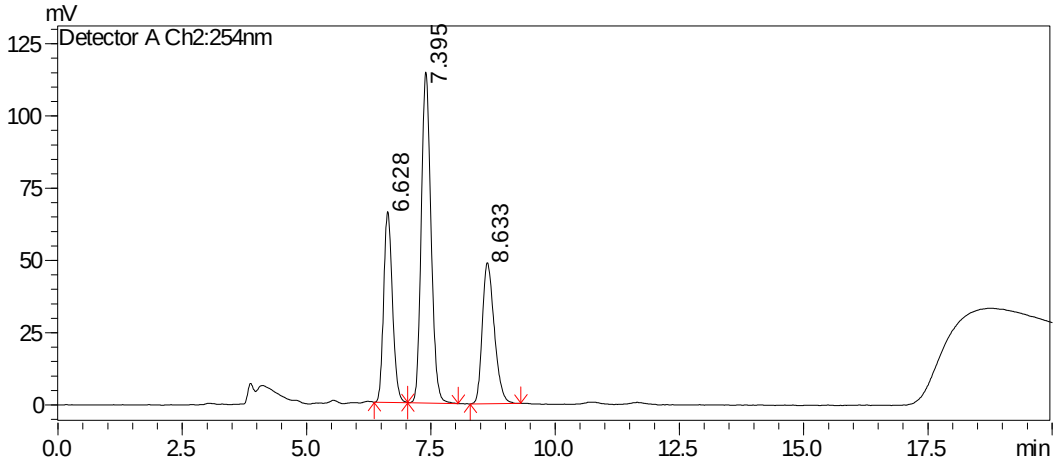
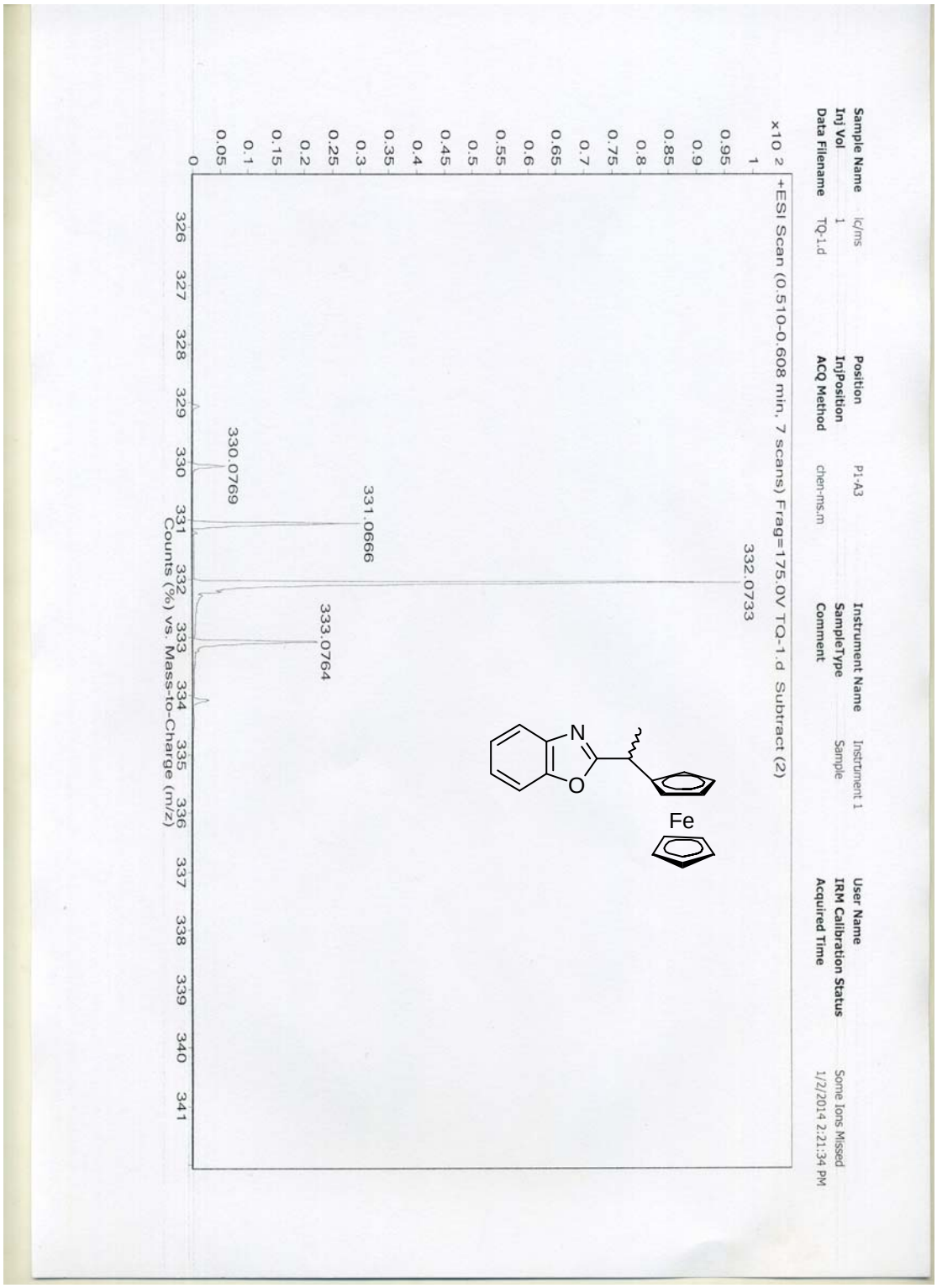
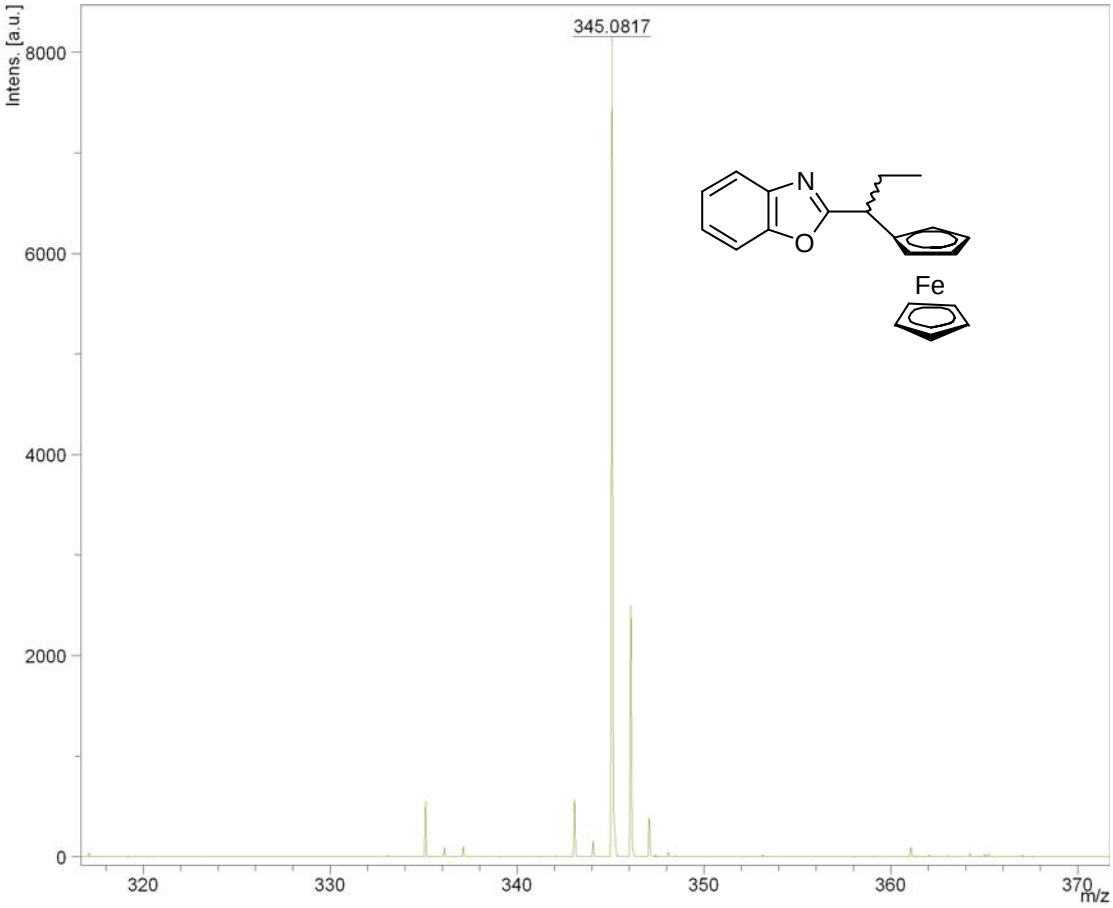




4. ^{1}H, ^{13}C NMR and HRMS (ESI-TOF) spectra for all compounds.

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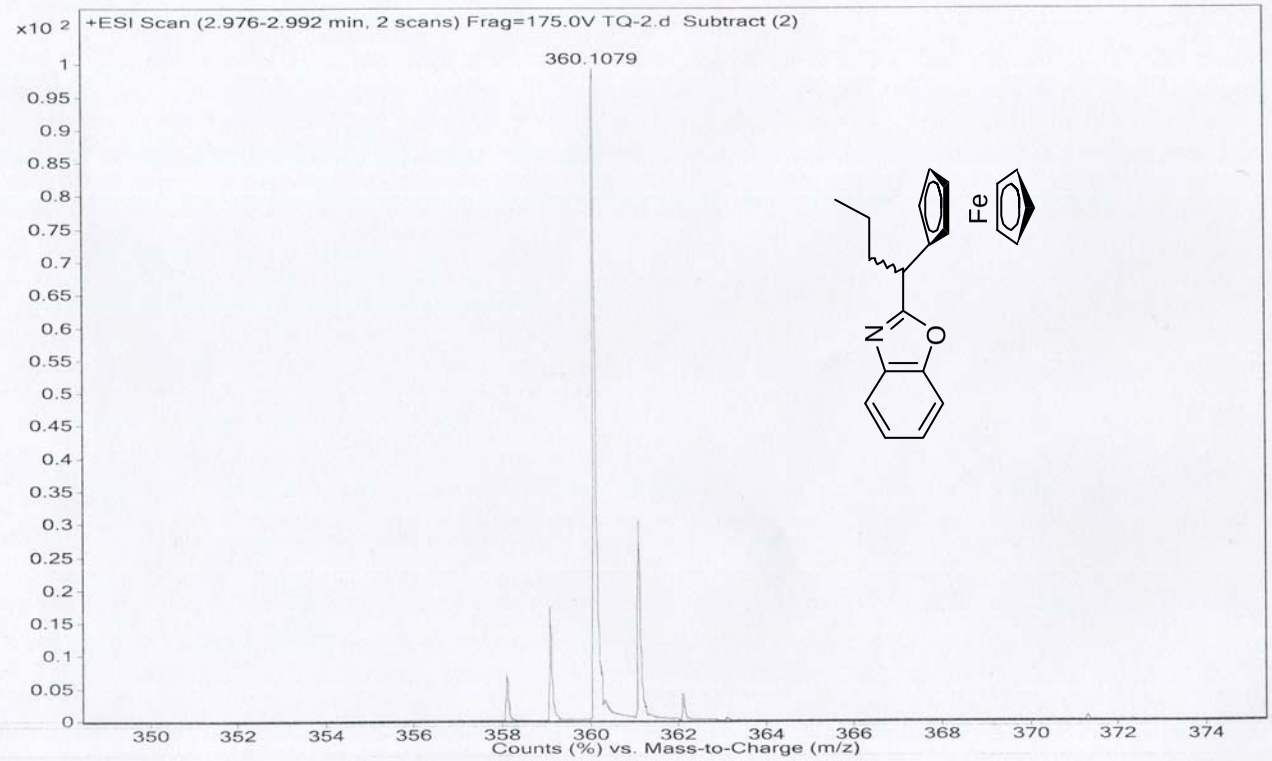

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Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
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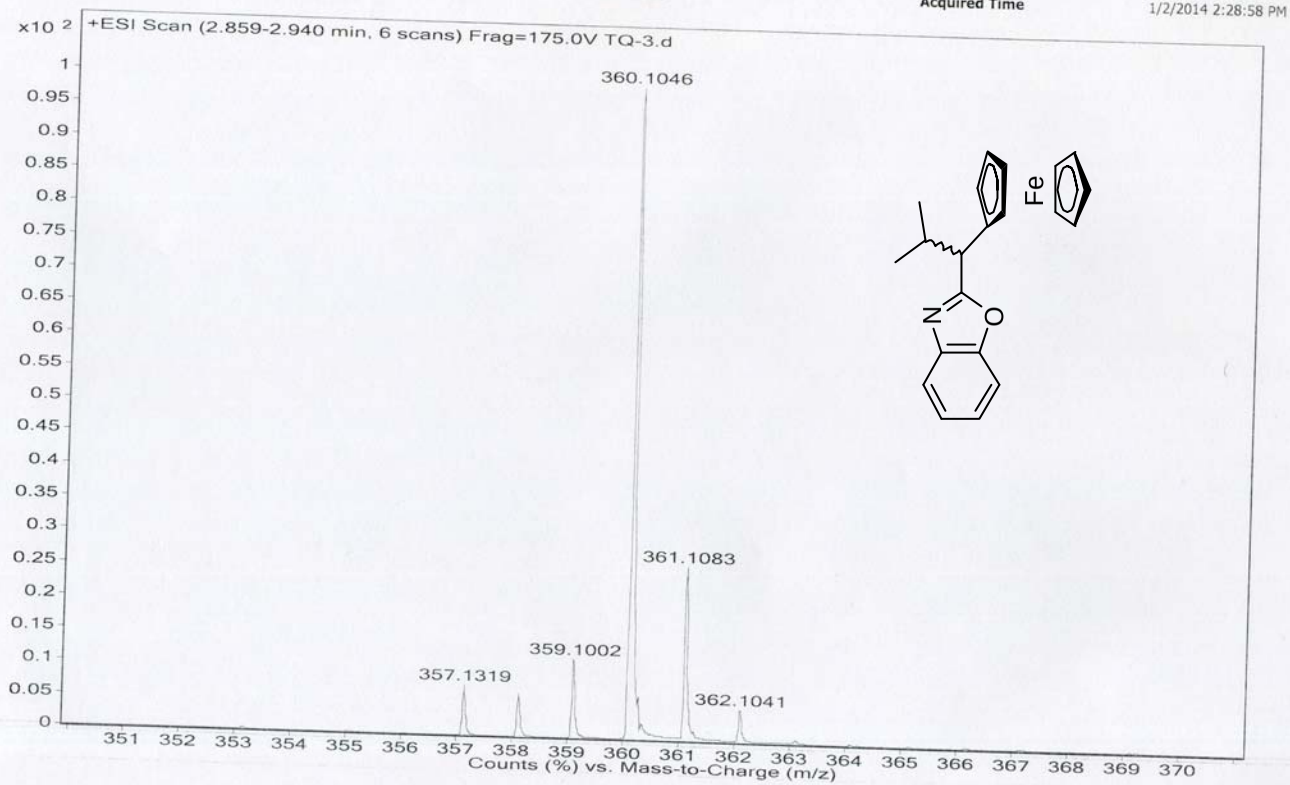
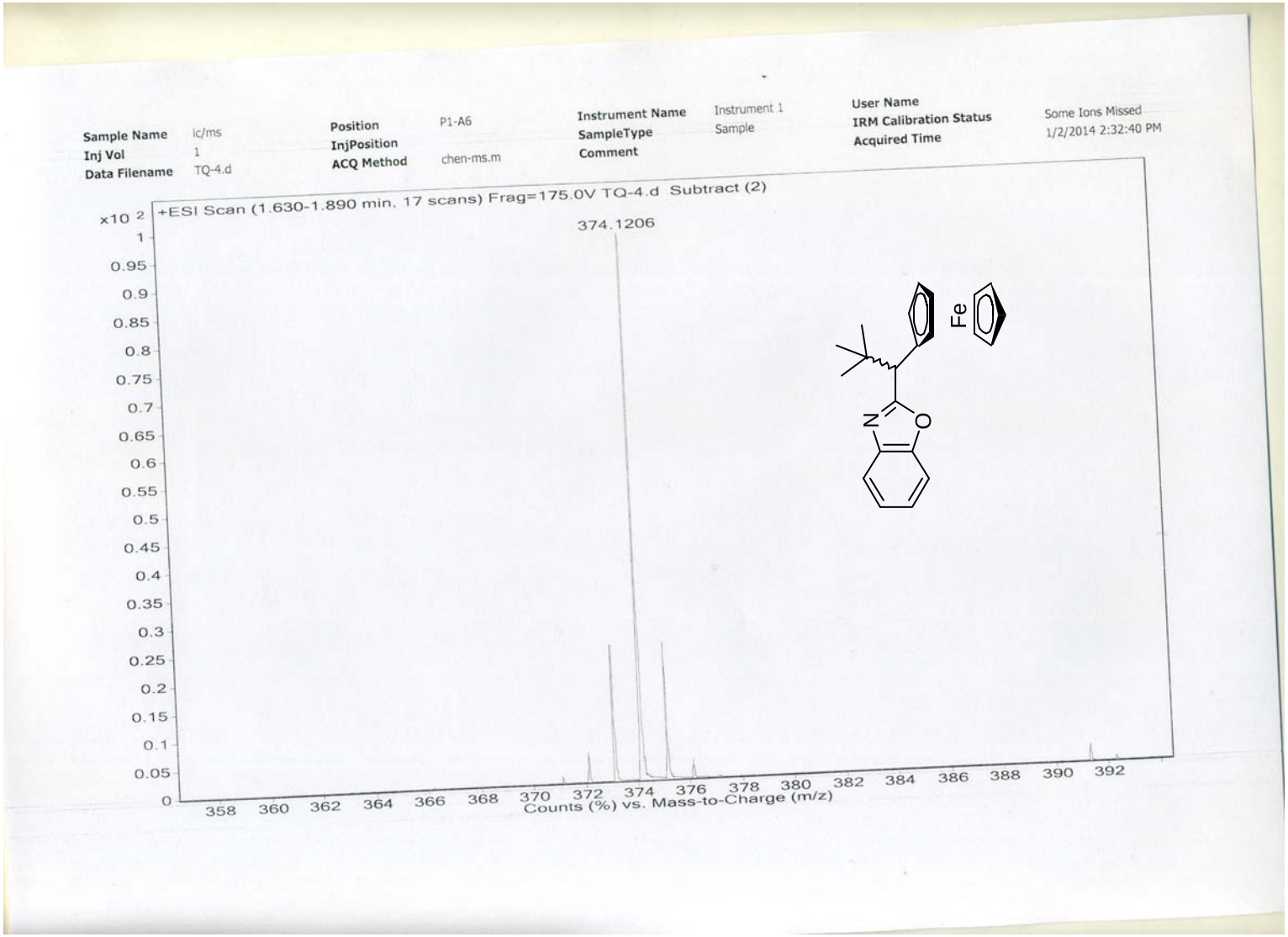
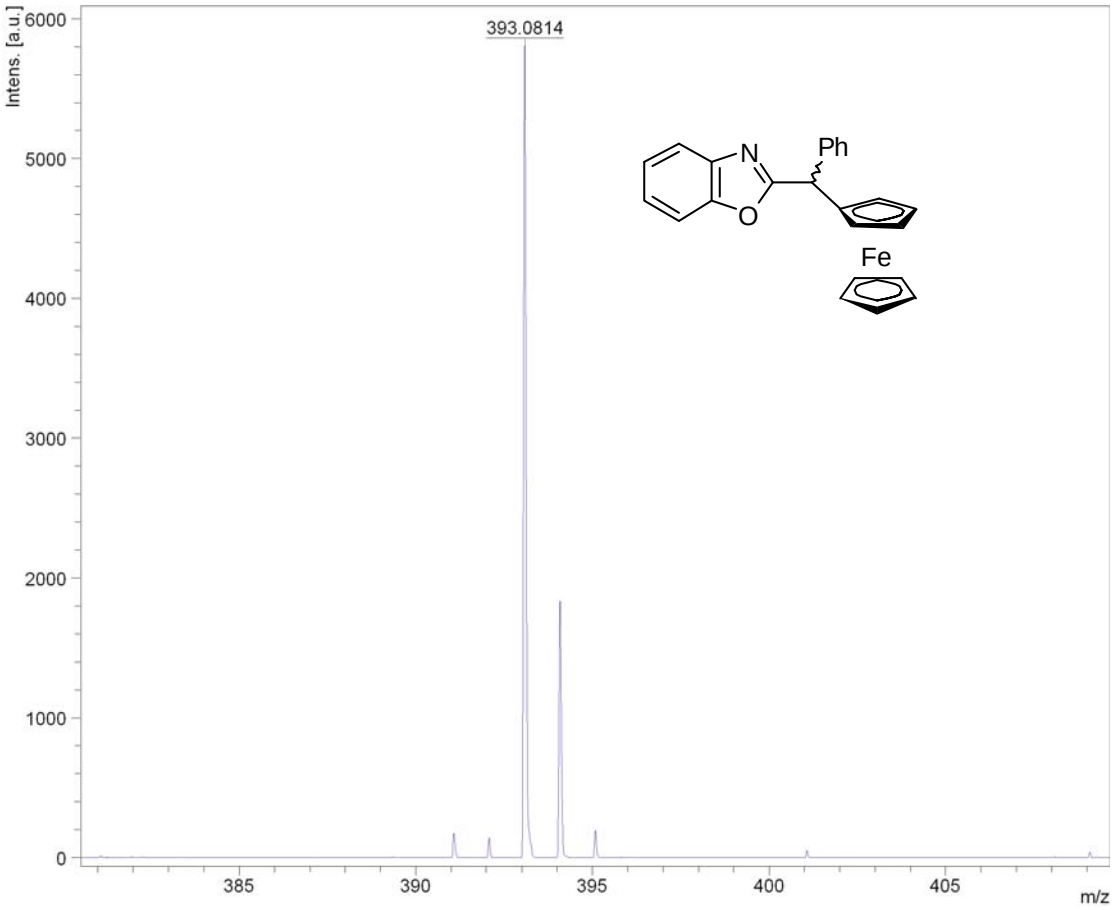
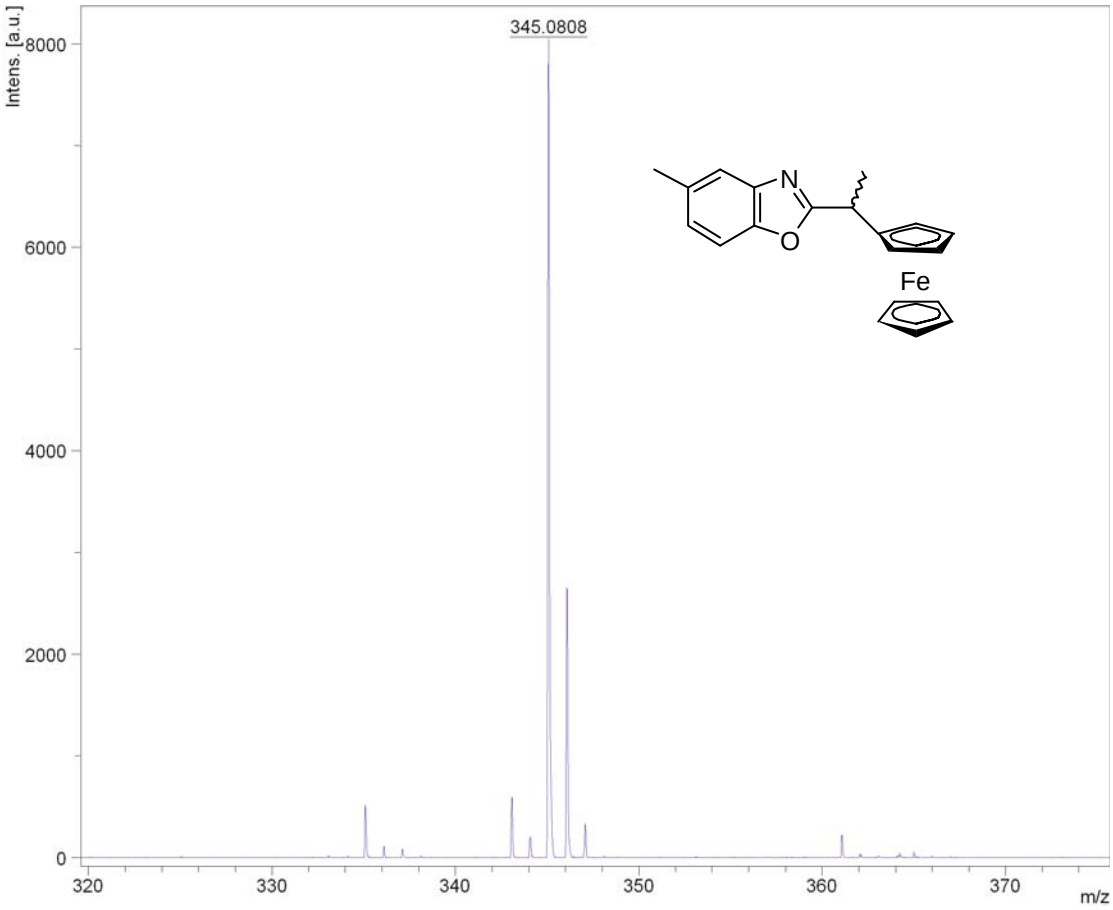
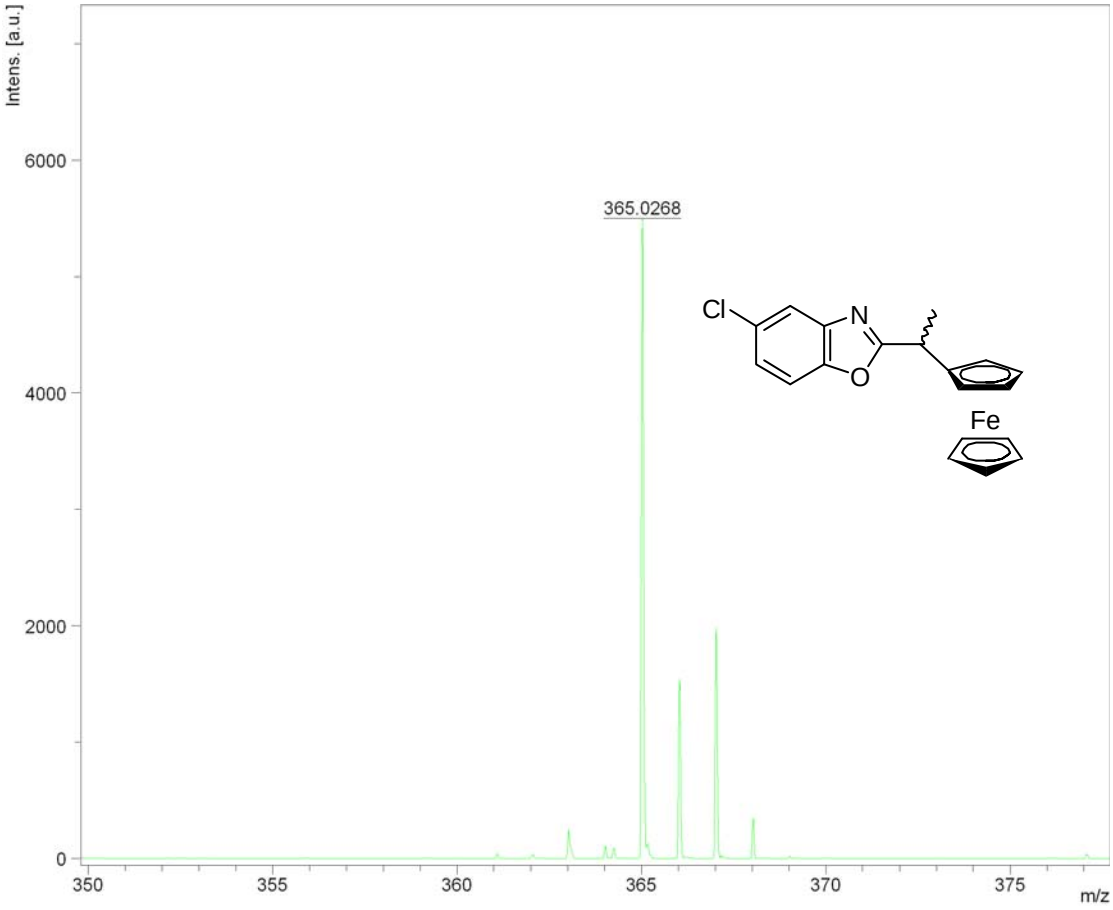


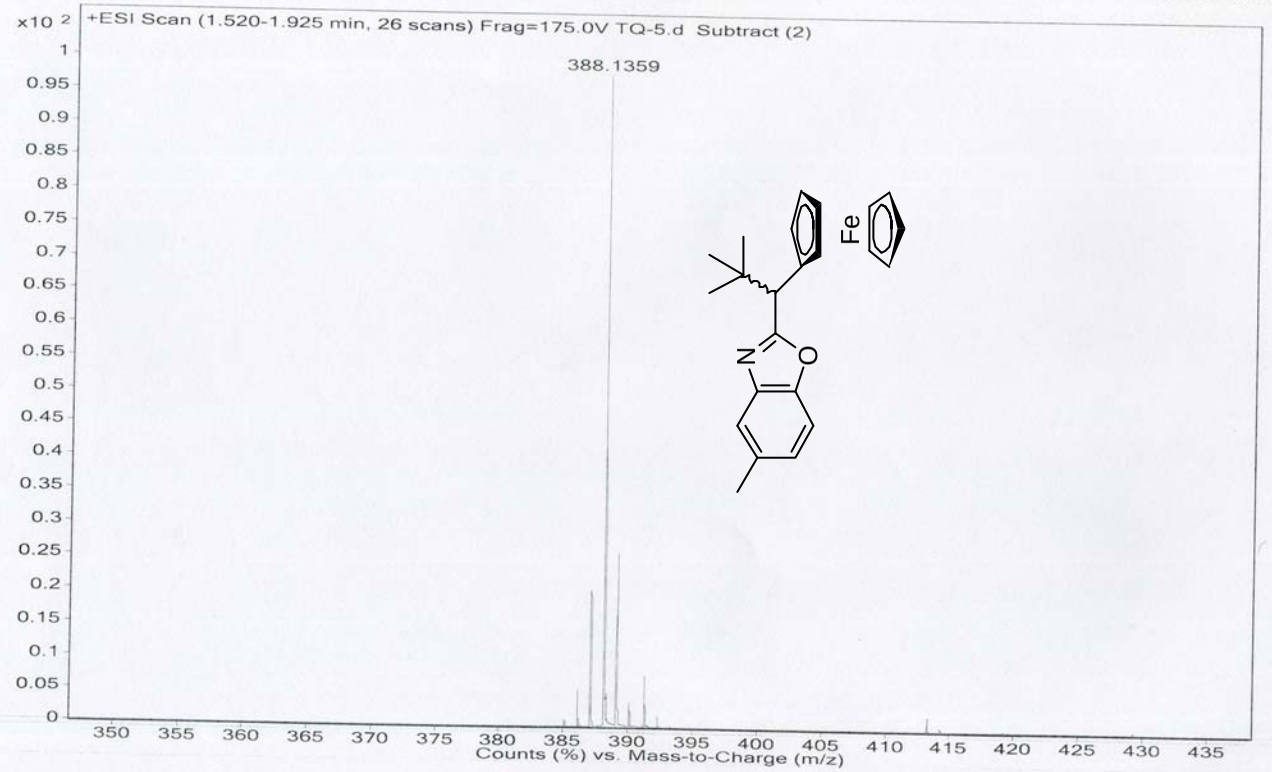

Sample Name ic/ms
Inj Vol 1
Data Filename TQ-4.d
Position P1-A6
InjPosition
ACQ Method chen-ms.m
Instrument Name Instrument 1
SampleType Sample
Comment
User Name
IRM Calibration Status
Acquired Time
Some Ions Missed
1/2/2014 2:32:40 PM
+ESI Scan (1.630-1.890 min, 17 scans) Frag=175.0V TQ-4.d Subtract (2)
374.1206
x10 2
Counts (%) vs. Mass-to-Charge (m/z)
Fe
N
O


393.0814
Intens. [a.u.]
6000
5000
4000
3000
2000
1000
0
385
390
395
400
405
m/z
N
O
Ph
Fe

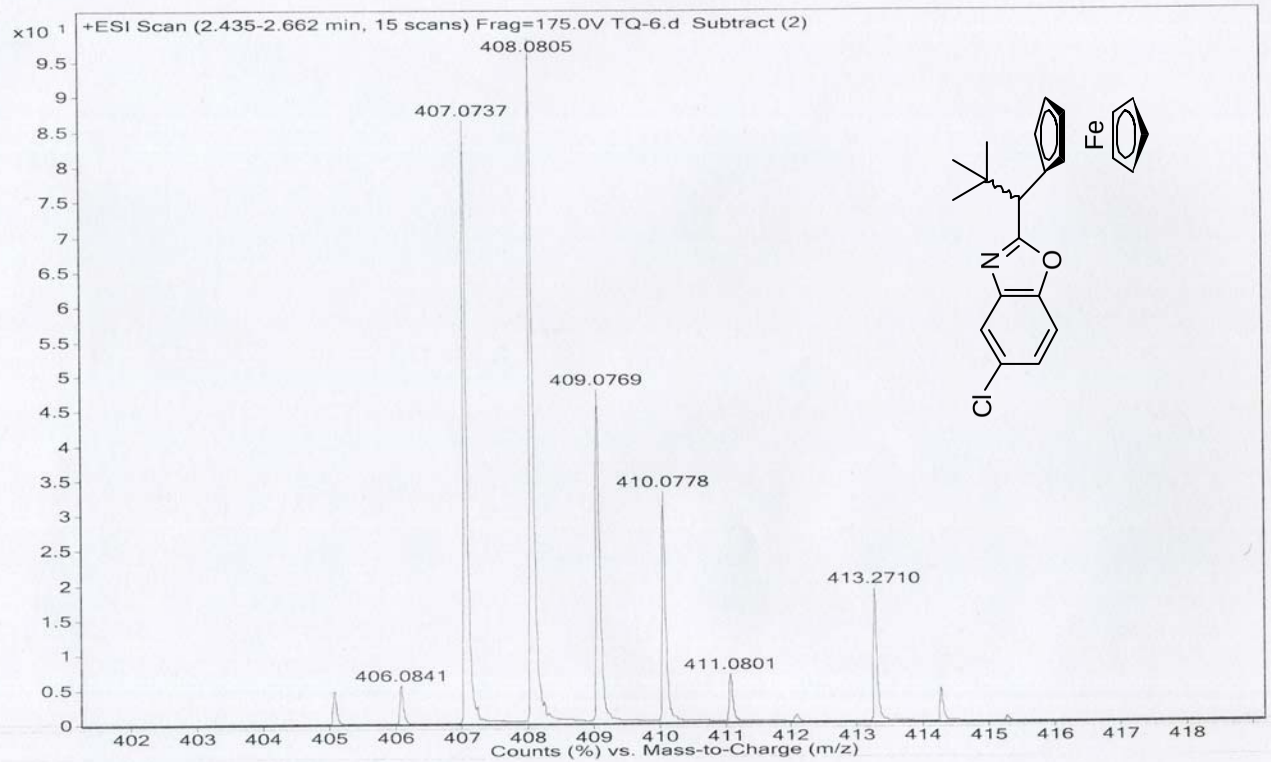

345.0808
Intens. [a.u.]
8000
6000
4000
2000
0
320
330
340
350
360
370
m/z
N
O
Fe

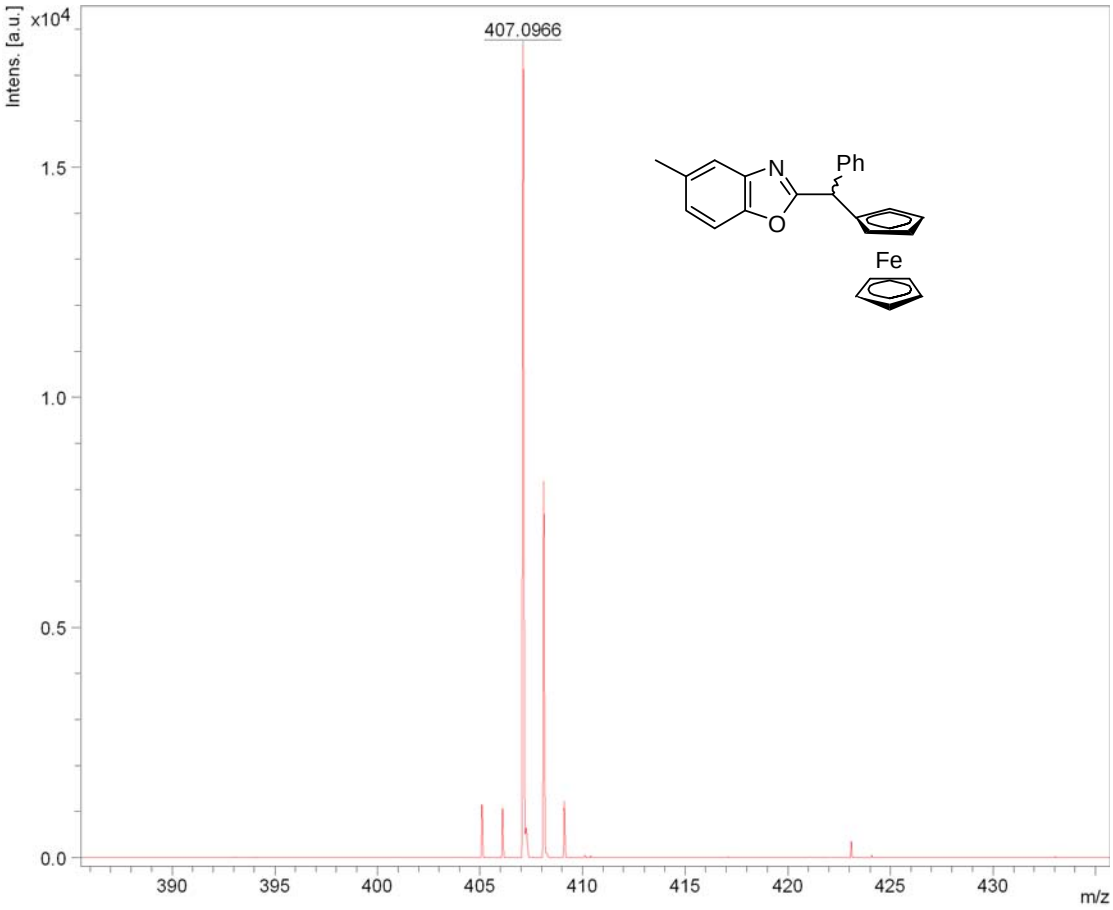

Intens. [a.u.]
6000
4000
2000
0
365.0268
Cl
N
O
Fe
350
355
360
365
370
375
m/z

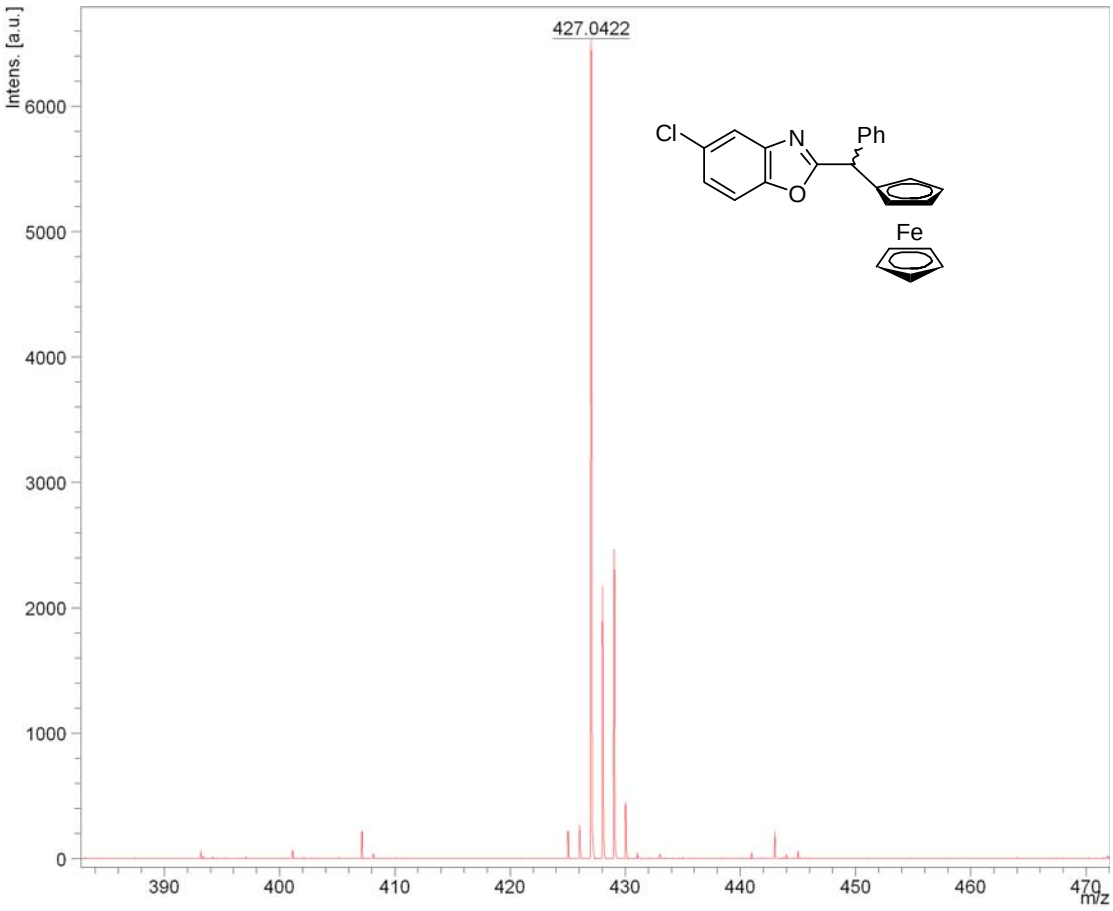
Sample Name	lc/ms	**Position**	P1-A7	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	TQ-5.d	**ACQ Method**	chen-ms.m	**Comment**		**Acquired Time**	1/2/2014 2:36:20 PM

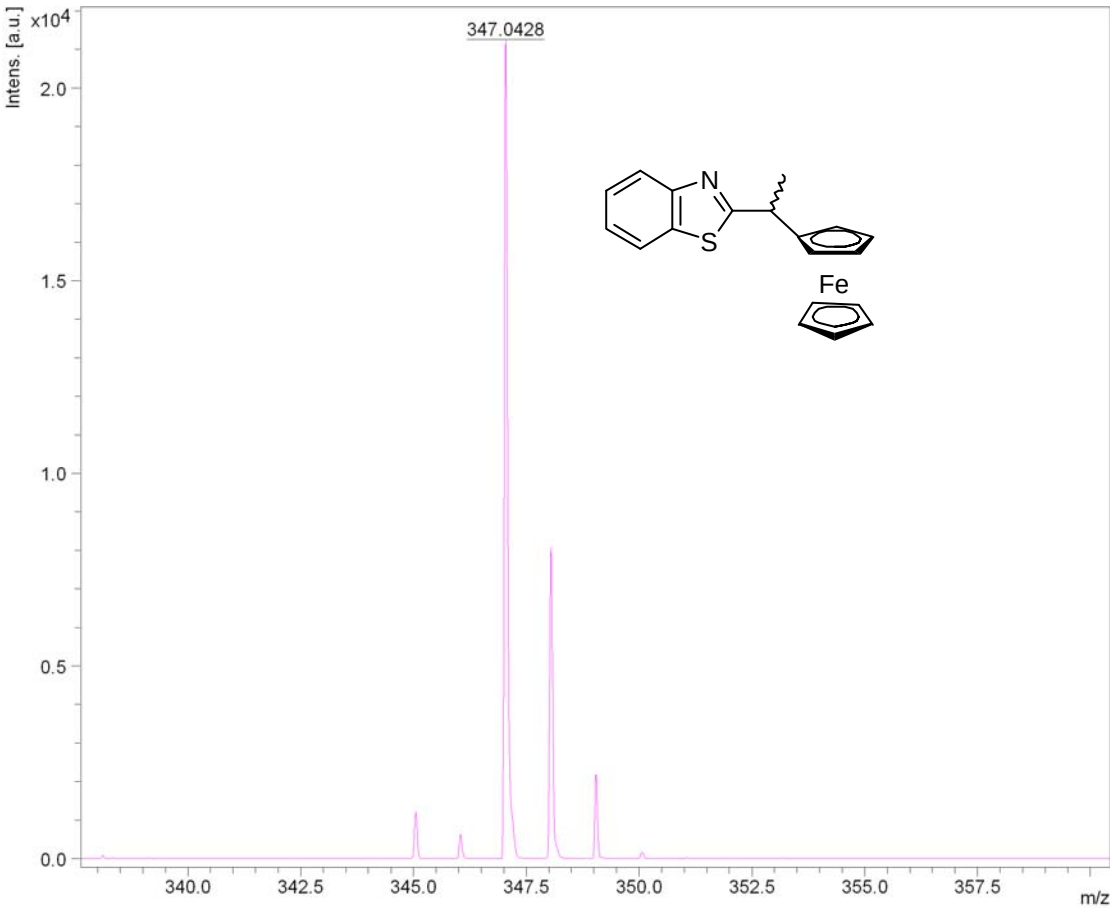


Sample Name	lc/ms	**Position**	P1-A1	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	TQ-6.d	**ACQ Method**	chen-ms.m	**Comment**		**Acquired Time**	1/2/2014 3:18:45 PM

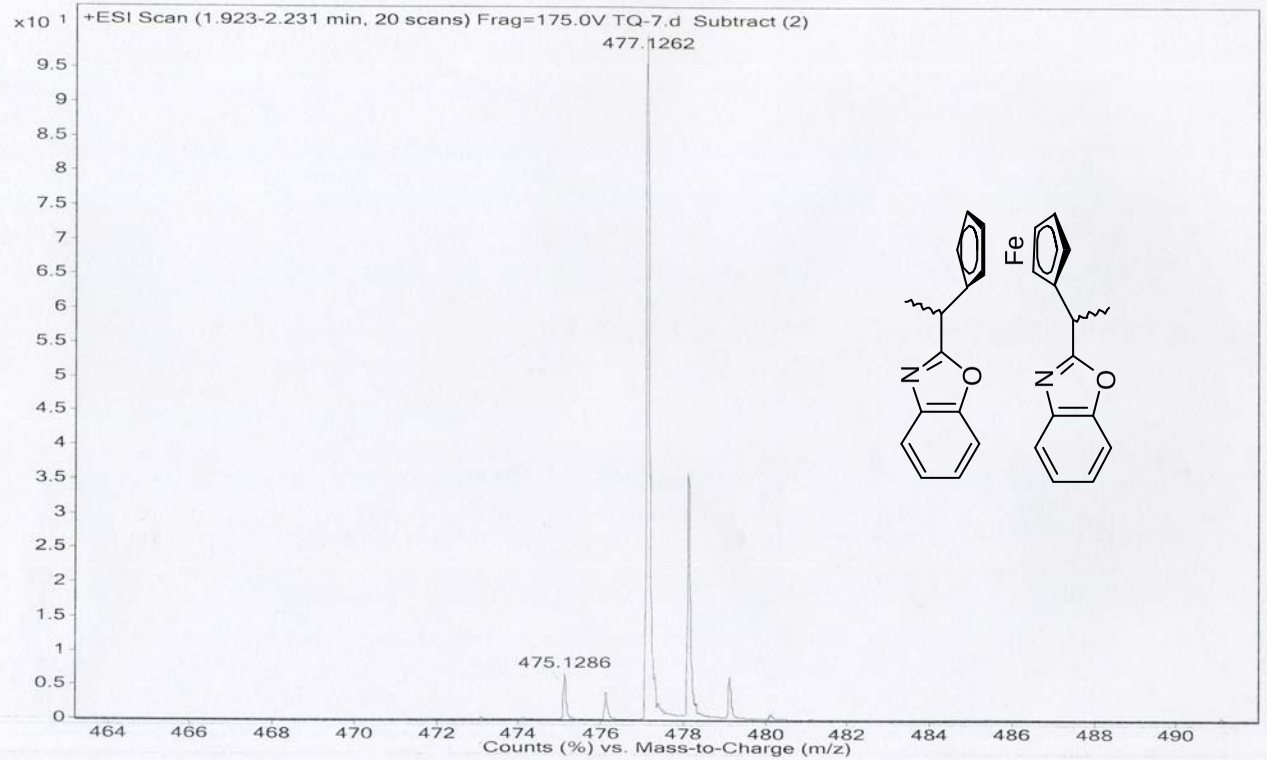



Intens. [a.u.]
x10^4
407.0966
1.5
1.0
0.5
0.0
390
395
400
405
410
415
420
425
430
m/z
N
O
Ph
Fe

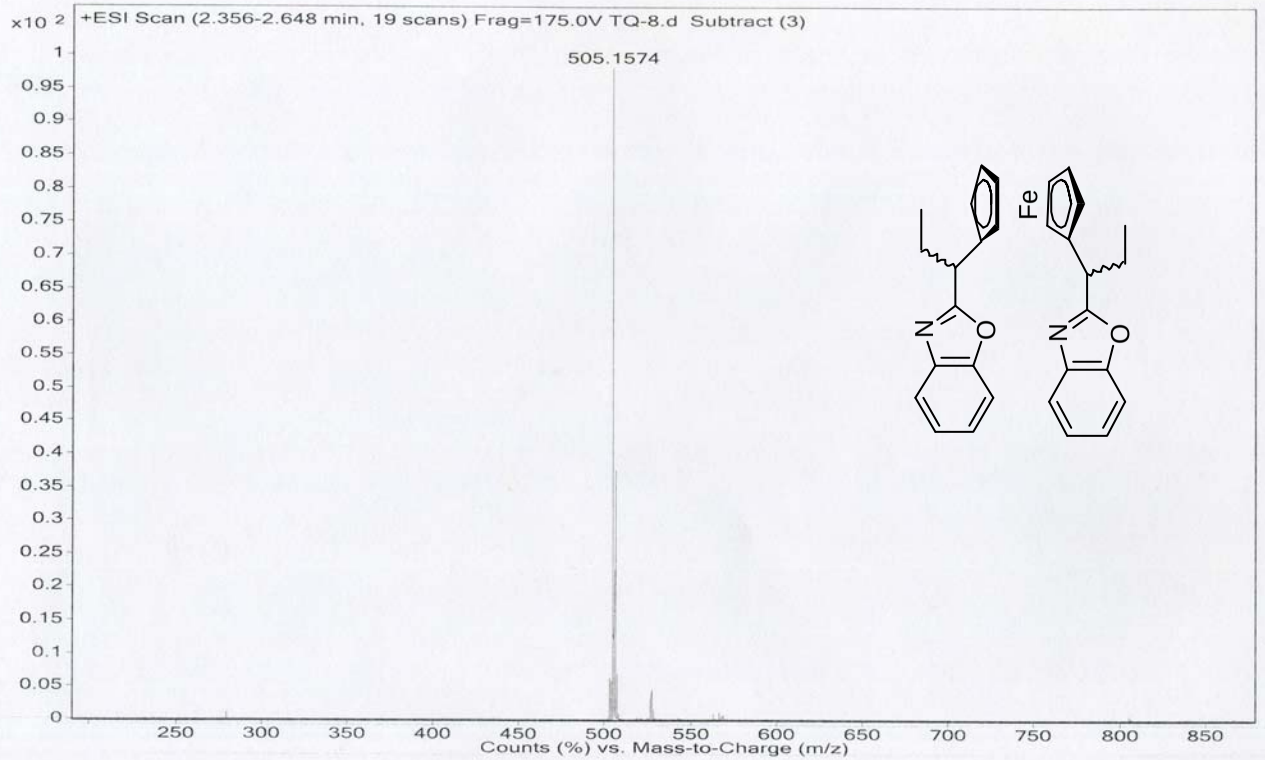

Intens. [a.u.]
427.0422
6000
5000
4000
3000
2000
1000
0
390
400
410
420
430
440
450
460
470
m/z
Cl
N
O
Ph
Fe

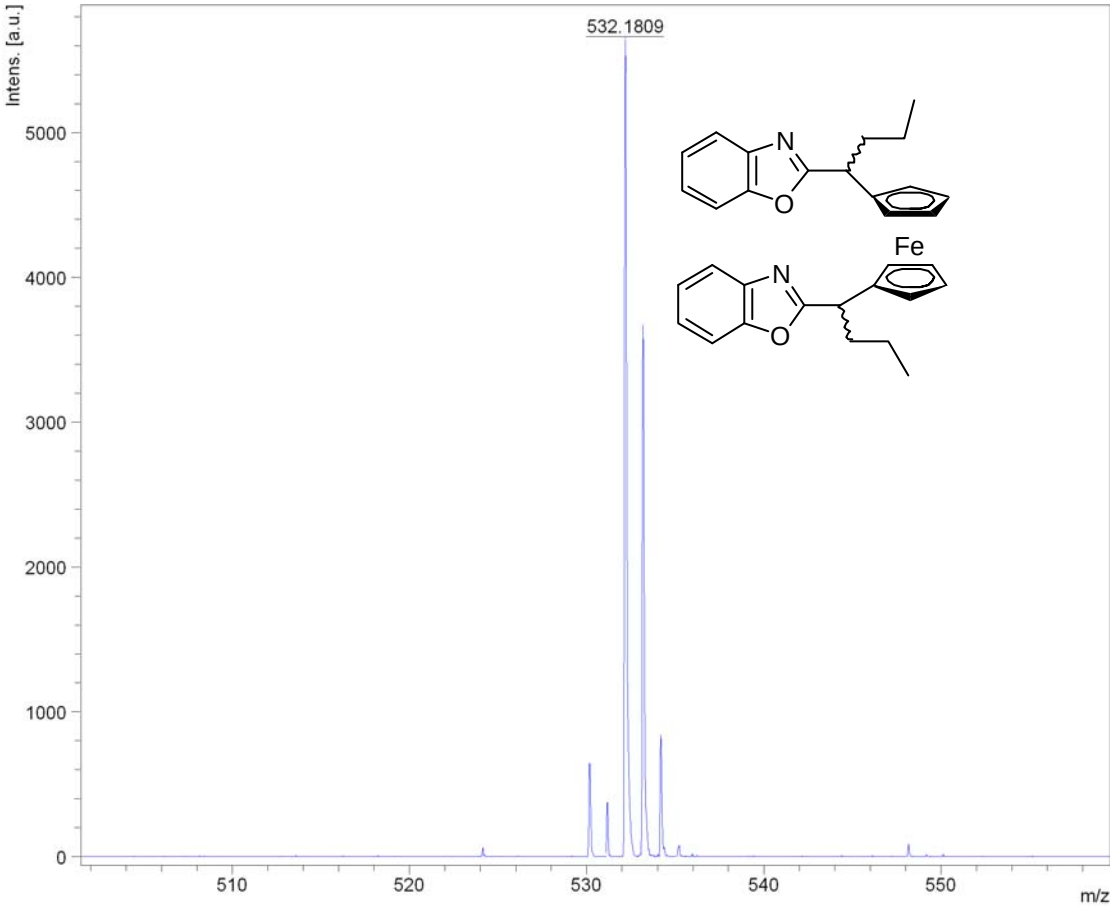

Intens. [a.u.]
x10^4
2.0
1.5
1.0
0.5
0.0
347.0428
N
S
Fe
340.0
342.5
345.0
347.5
350.0
352.5
355.0
357.5
m/z

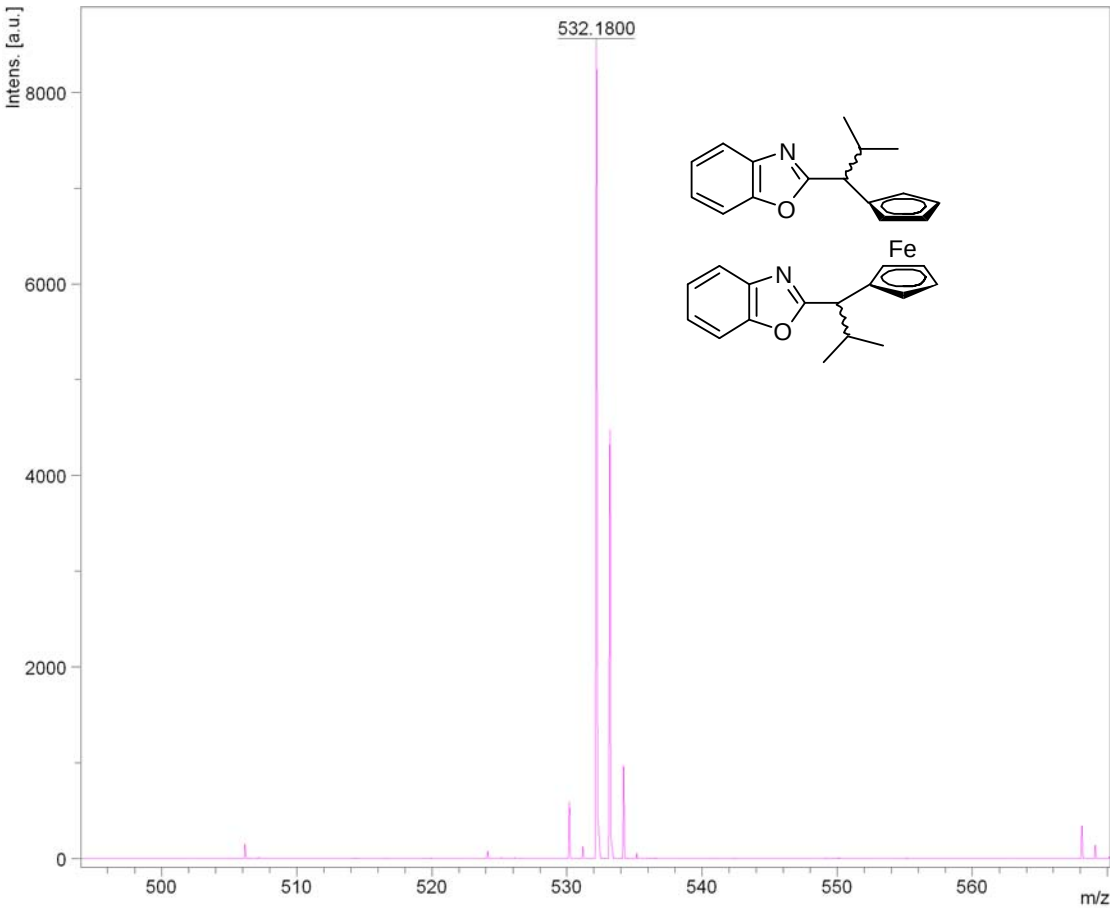
Sample Name	lc/ms	**Position**	P1-A2	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	TQ-7.d	**ACQ Method**	chen-ms.m	**Comment**		**Acquired Time**	1/2/2014 3:22:27 PM

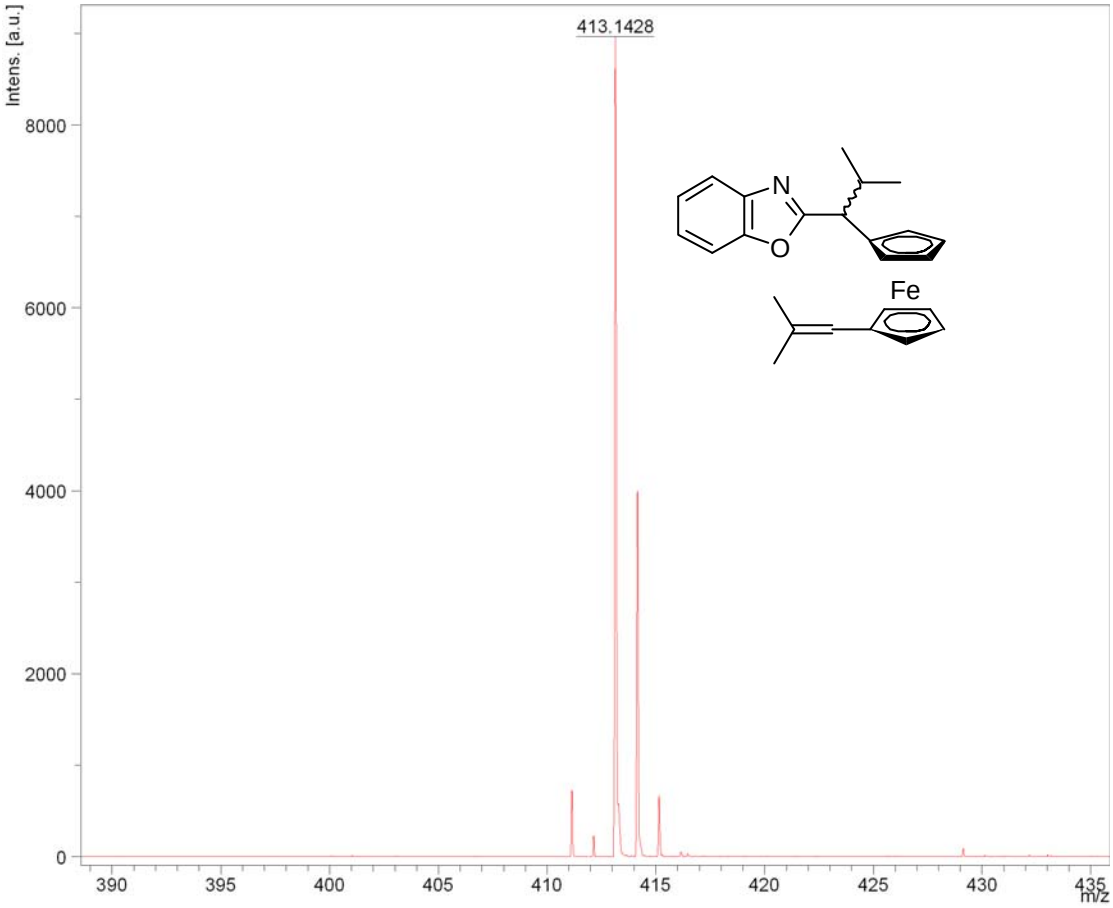


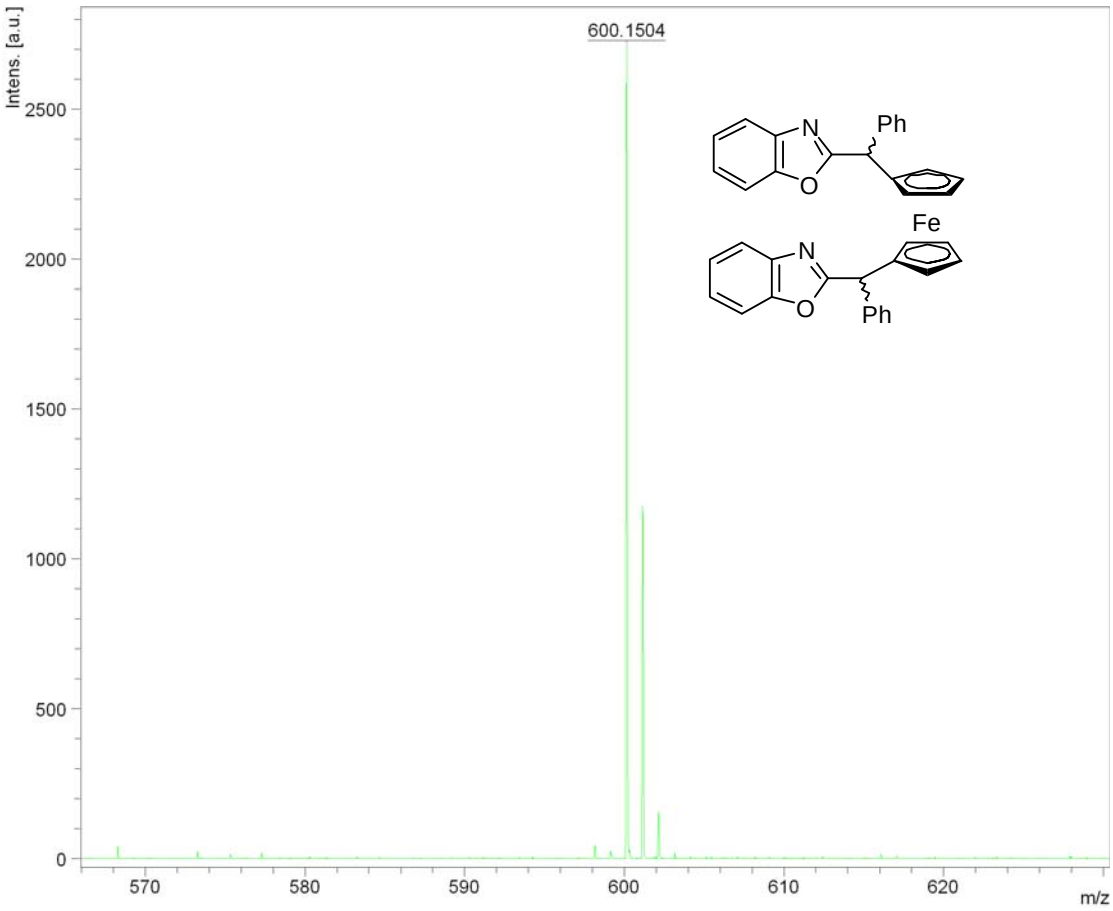
Sample Name	lc/ms	**Position**	P1-A3	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Some Ions Missed
Data Filename	TQ-8.d	**ACQ Method**	chen-ms.m	**Comment**		**Acquired Time**	1/2/2014 3:26:08 PM

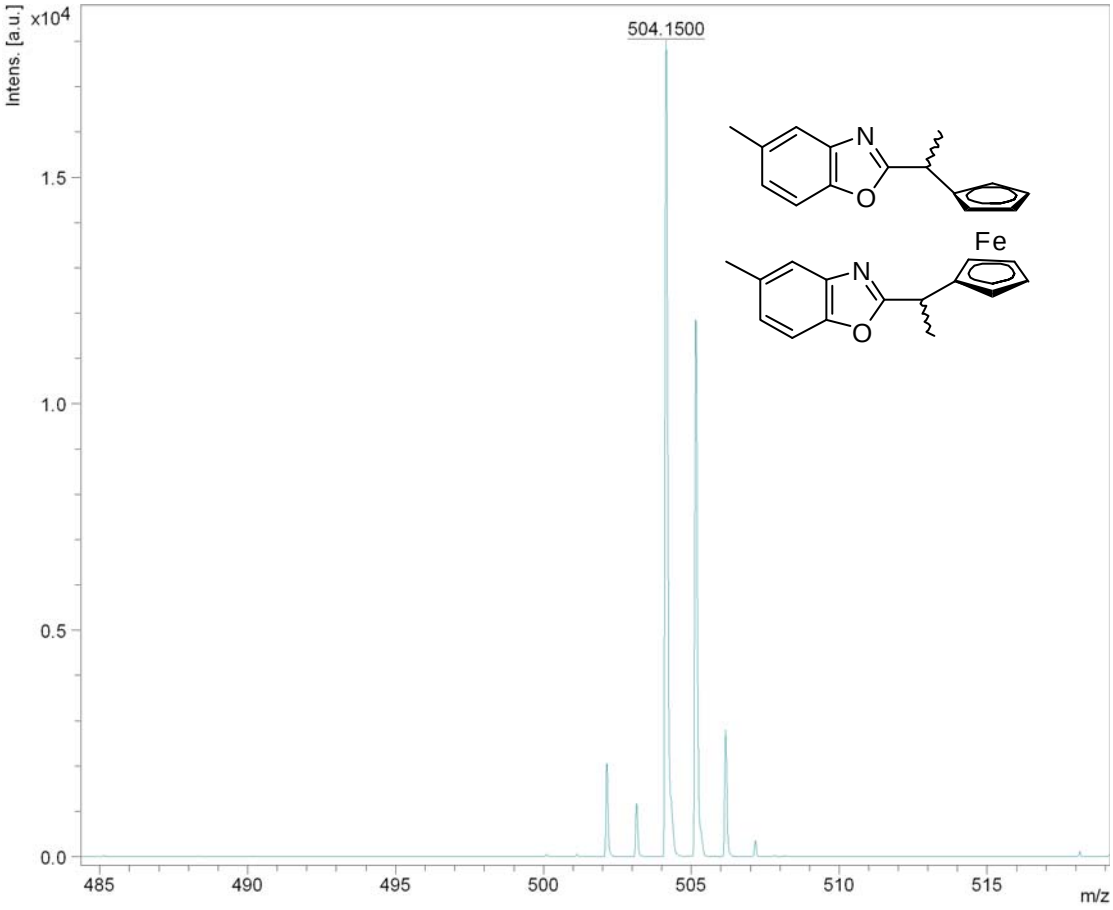


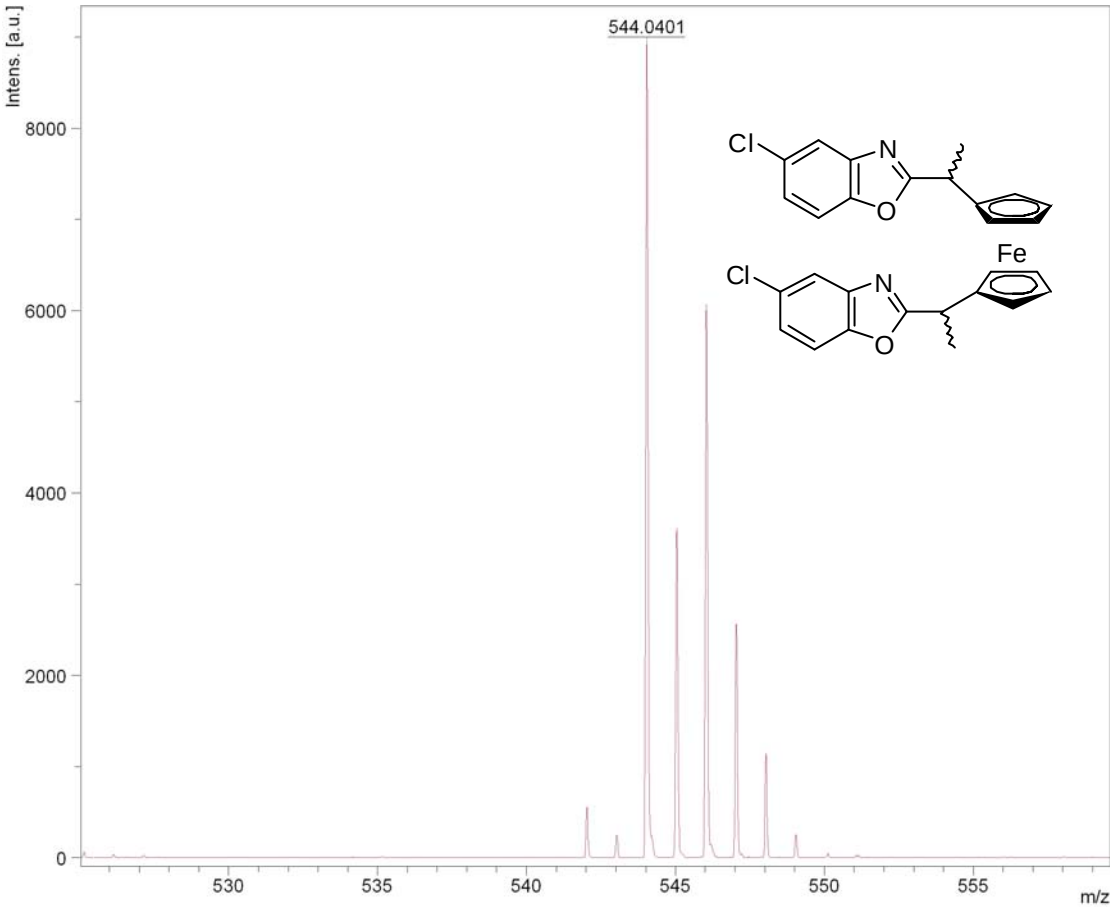

532.1809
Intens. [a.u.]
5000
4000
3000
2000
1000
0
510
520
530
540
550
m/z
N
O
Fe
N
O

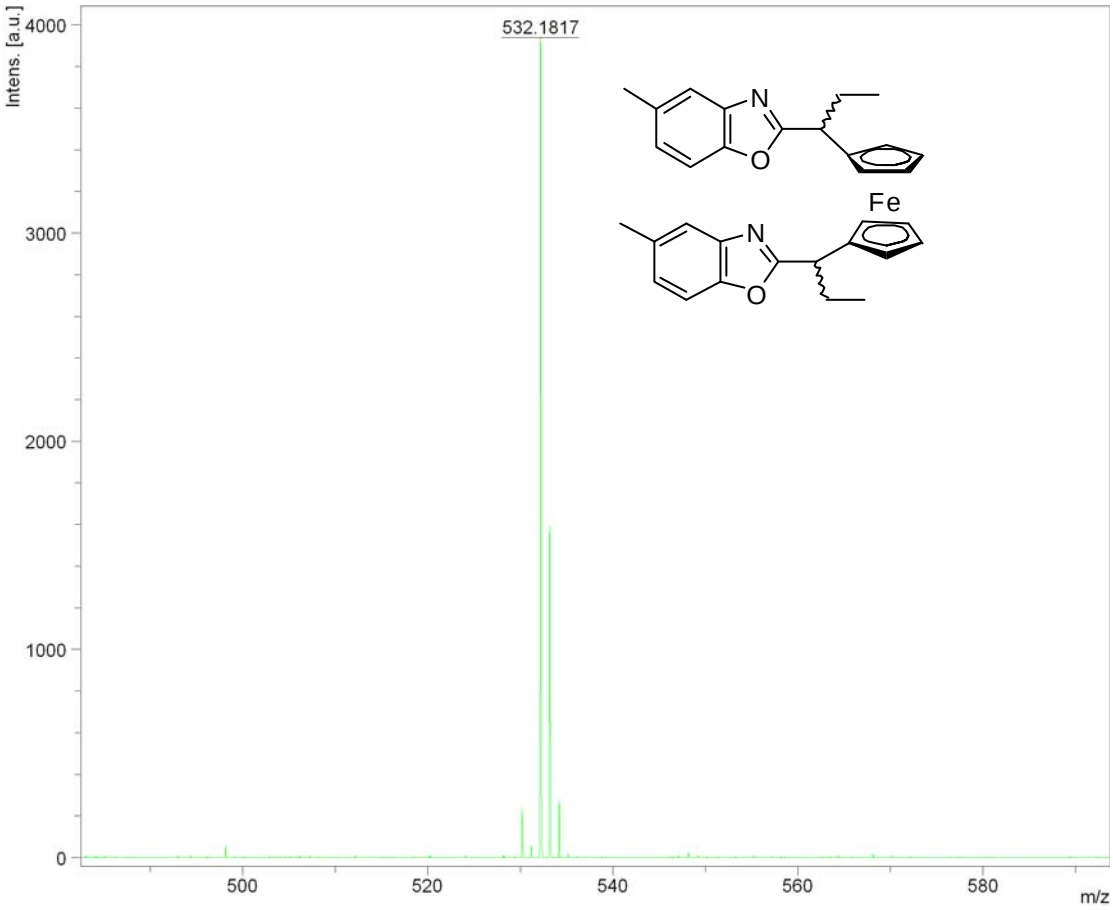

532.1800
Intens. [a.u.]
8000
6000
4000
2000
0
500
510
520
530
540
550
560
m/z
N
O
Fe
N
O

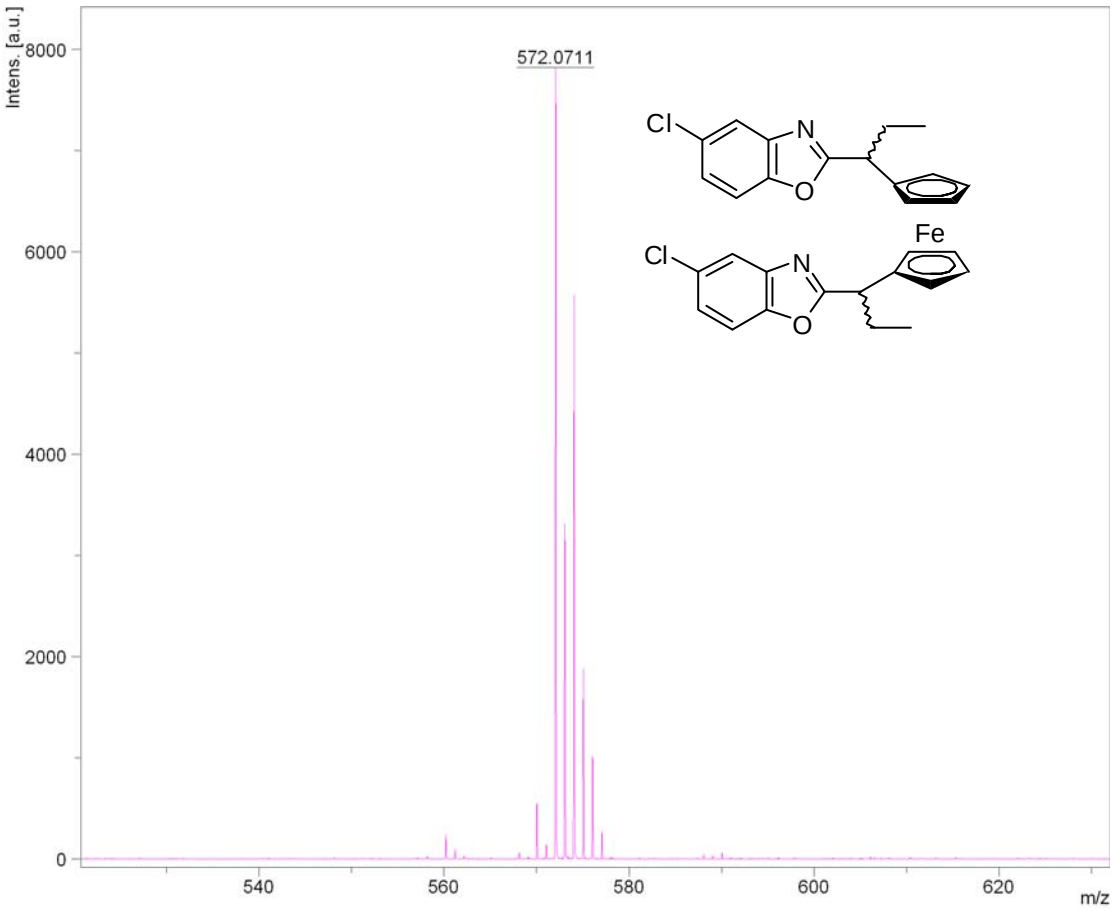

413.1428
Intens. [a.u.]
8000
6000
4000
2000
0
390
395
400
405
410
415
420
425
430
435
m/z
N
O
Fe

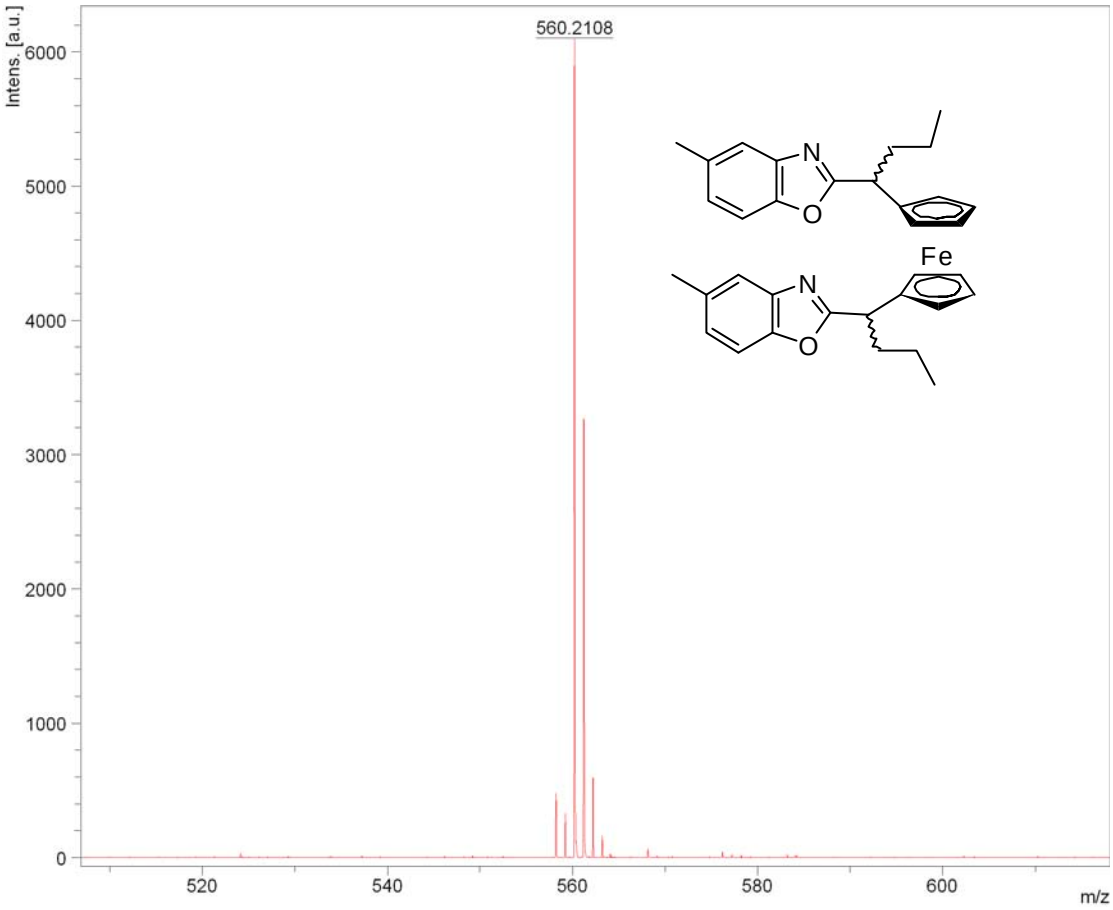

600.1504
Intens. [a.u.]
2500
2000
1500
1000
500
0
570
580
590
600
610
620
m/z
N
O
Ph
Fe
N
O
Ph

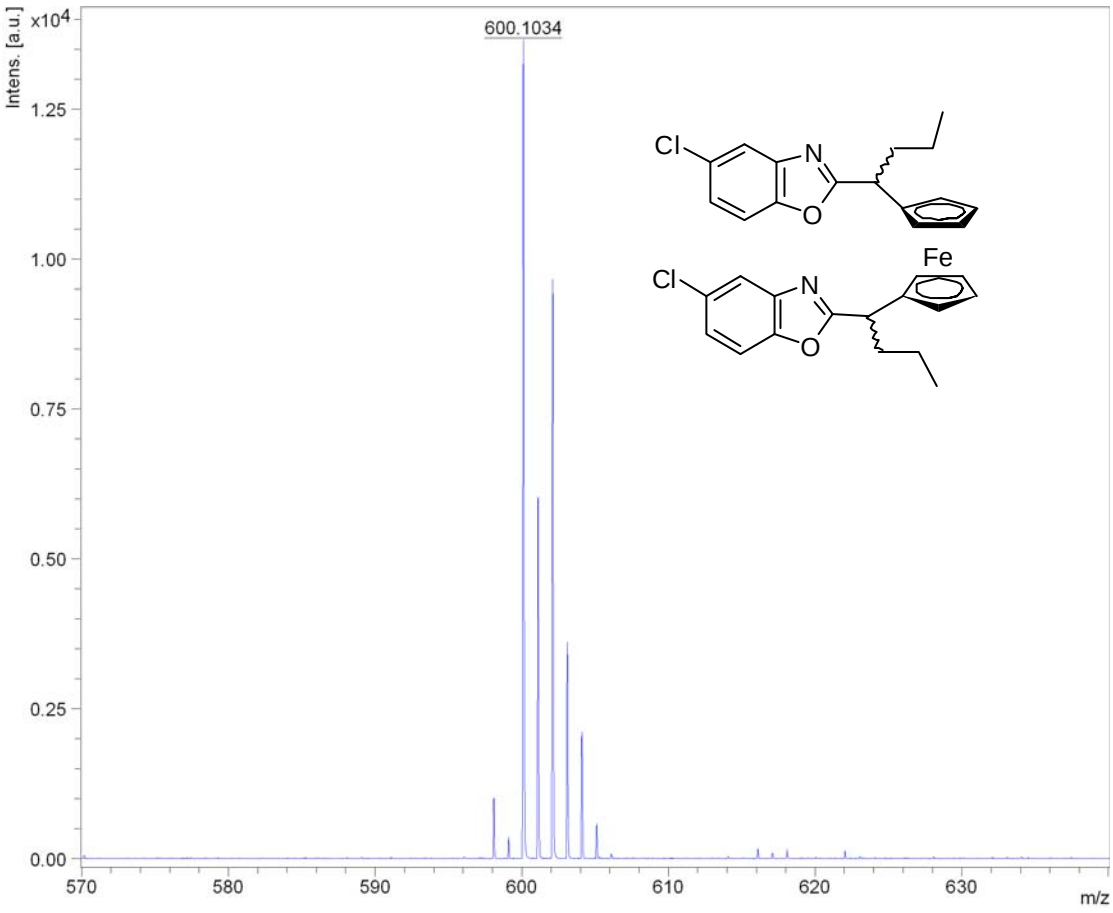

Intens. [a.u.]
x10^4
504.1500
1.5
1.0
0.5
0.0
485
490
495
500
505
510
515
m/z
N
O
Fe

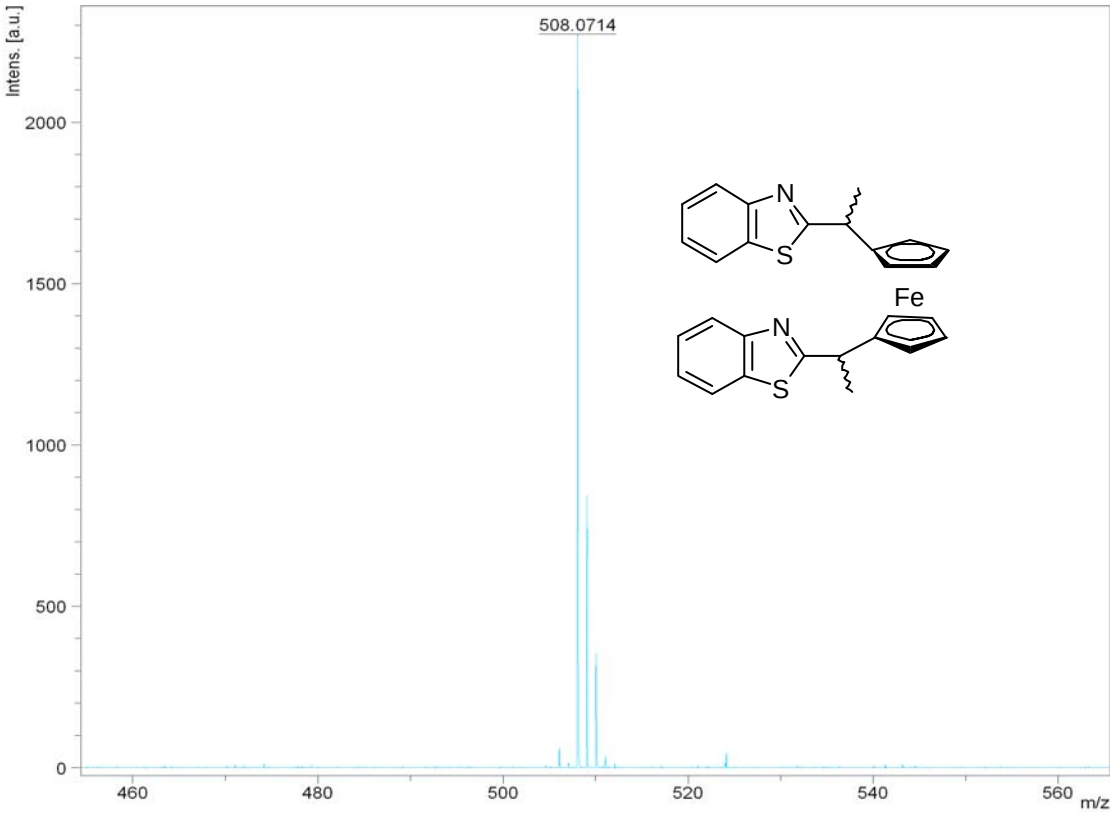

Intens. [a.u.]
544.0401
8000
6000
4000
2000
0
530
535
540
545
550
555
m/z
Cl
N
O
Fe

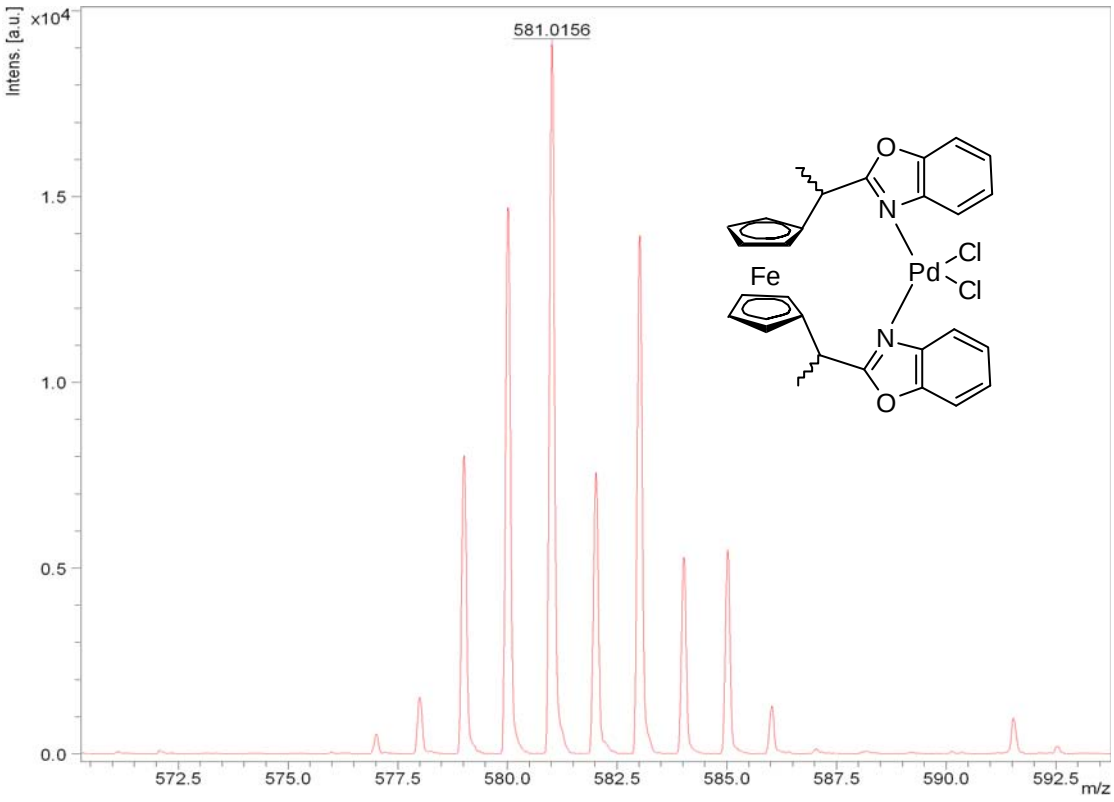

Intens. [a.u.]
4000
3000
2000
1000
0
532.1817
N
O
Fe
500
520
540
560
580
m/z

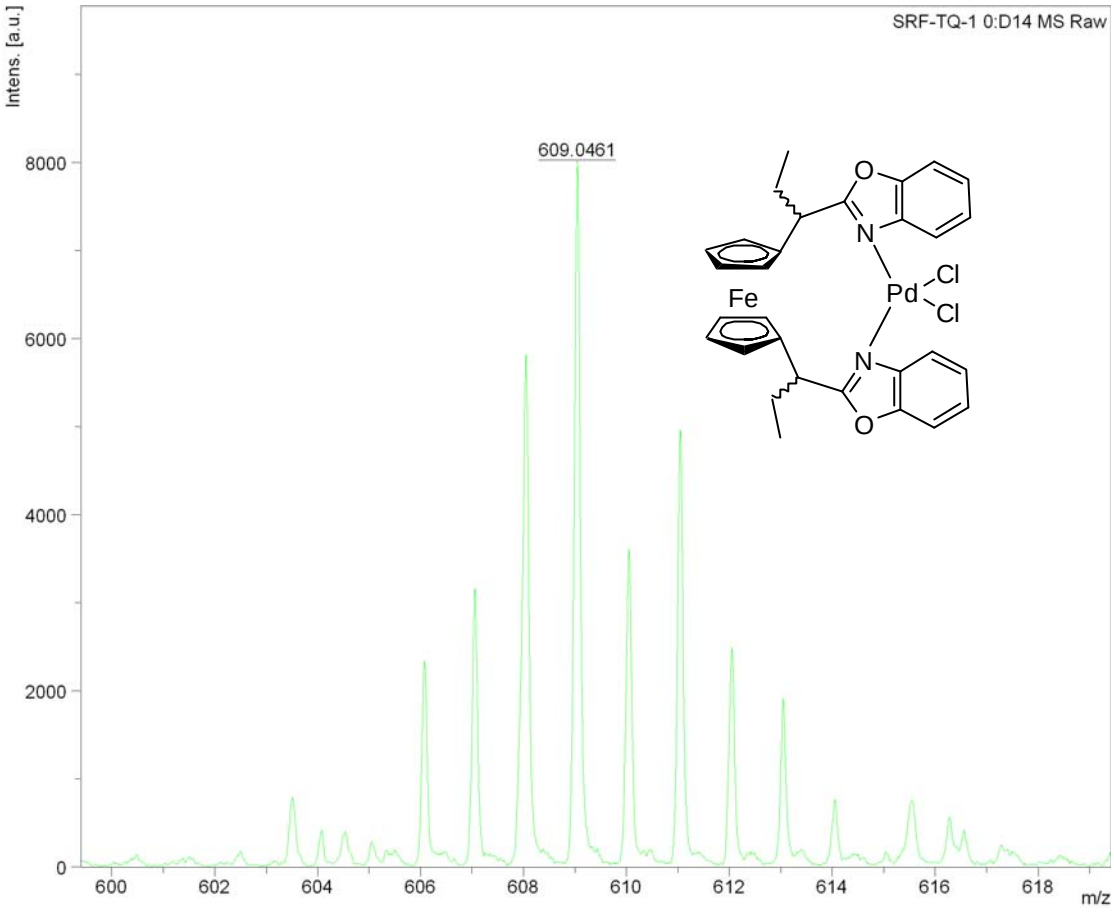

Intens. [a.u.]
8000
6000
4000
2000
0
572.0711
Cl
N
O
Fe
540
560
580
600
620
m/z

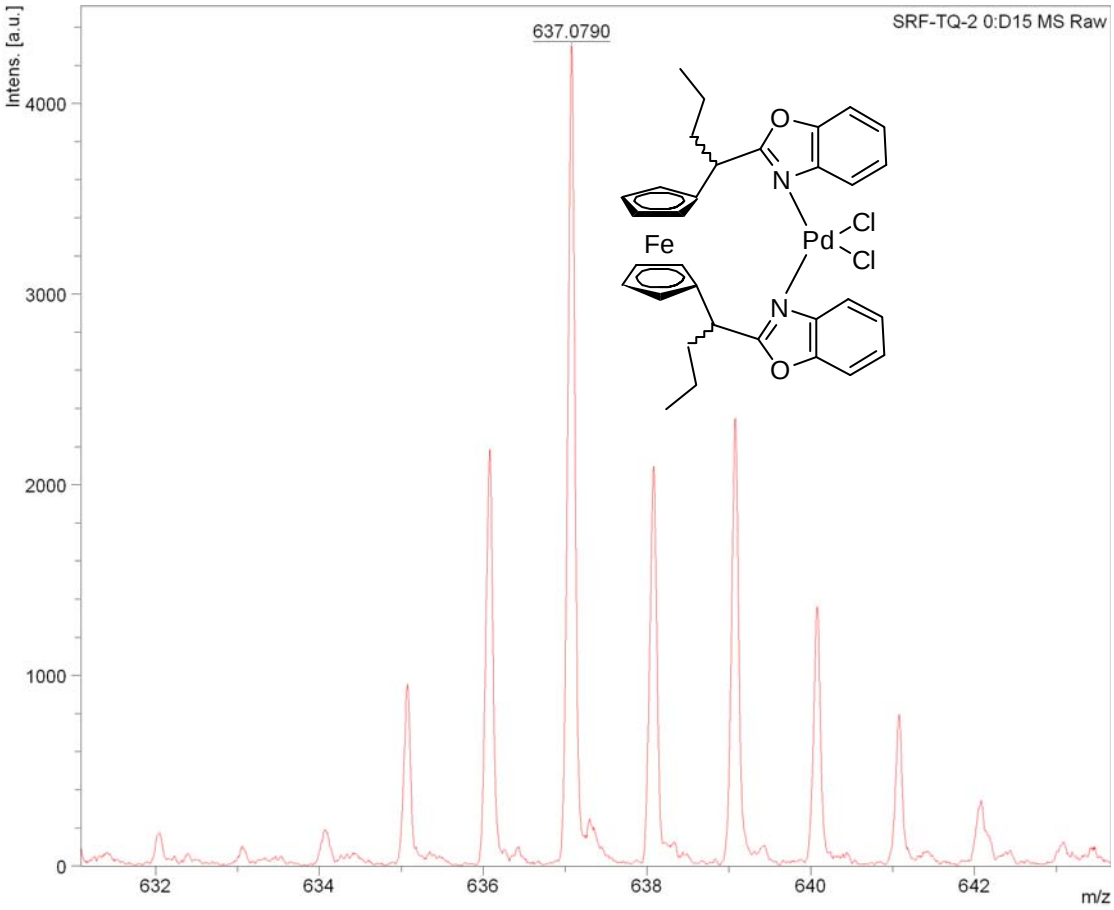

560.2108
Intens. [a.u.]
6000
5000
4000
3000
2000
1000
0
520
540
560
580
600
m/z
N
O
Fe


600.1034
Intens. [a.u.]
x10^4
1.25
1.00
0.75
0.50
0.25
0.00
570
580
590
600
610
620
630
m/z
Cl
N
O
Fe

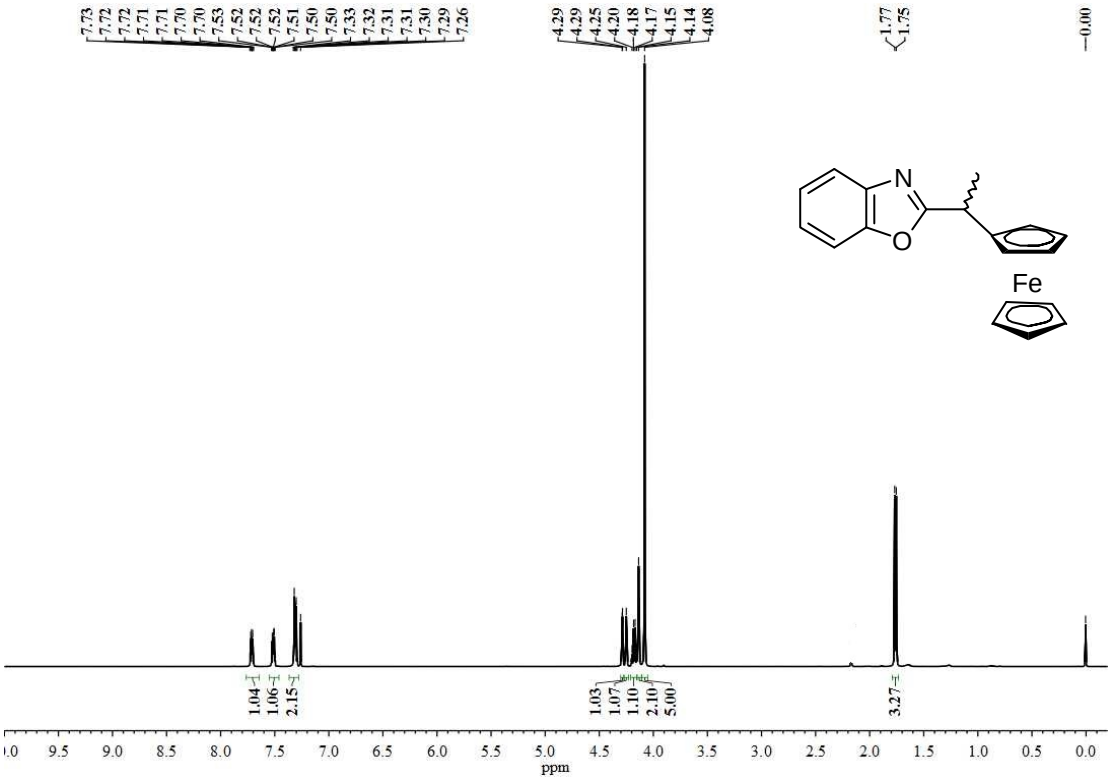

508.0714
Intens. [a.u.]
2000
1500
1000
500
0
460
480
500
520
540
560
m/z
N
S
Fe
N
S

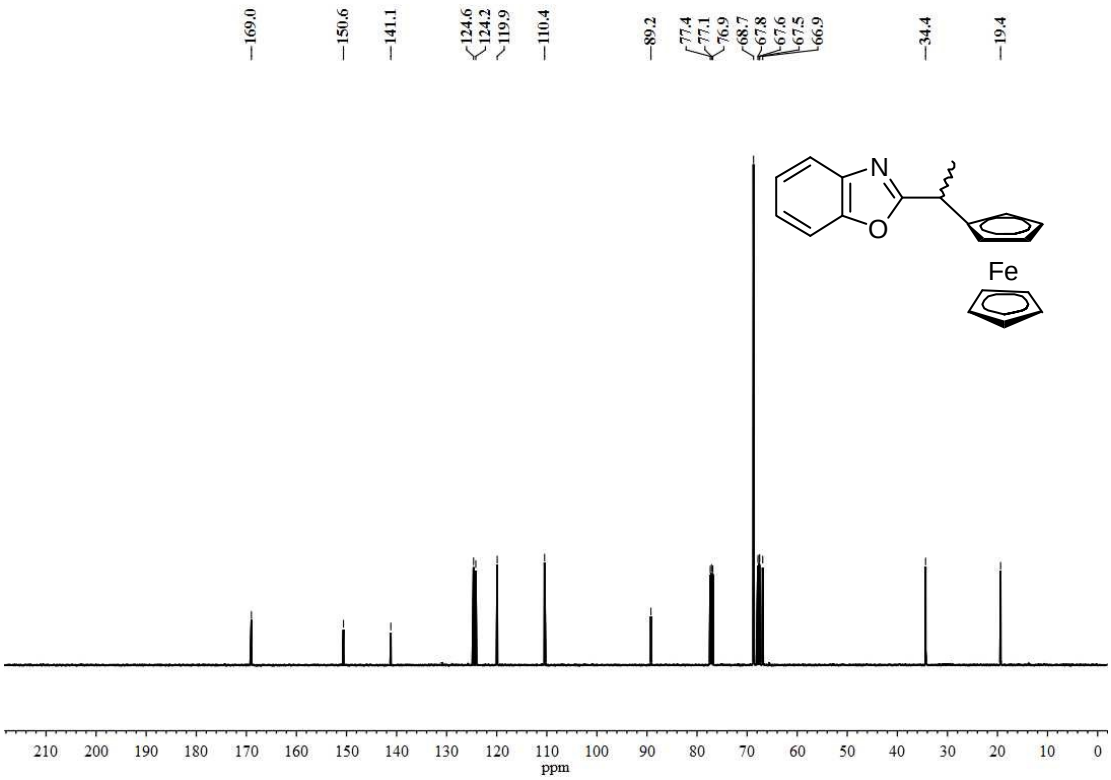

581.0156
Intens. [a.u.]
x10^4
1.5
1.0
0.5
0.0
572.5
575.0
577.5
580.0
582.5
585.0
587.5
590.0
592.5
m/z
O
N
Pd
Cl
Cl
Fe
N
O

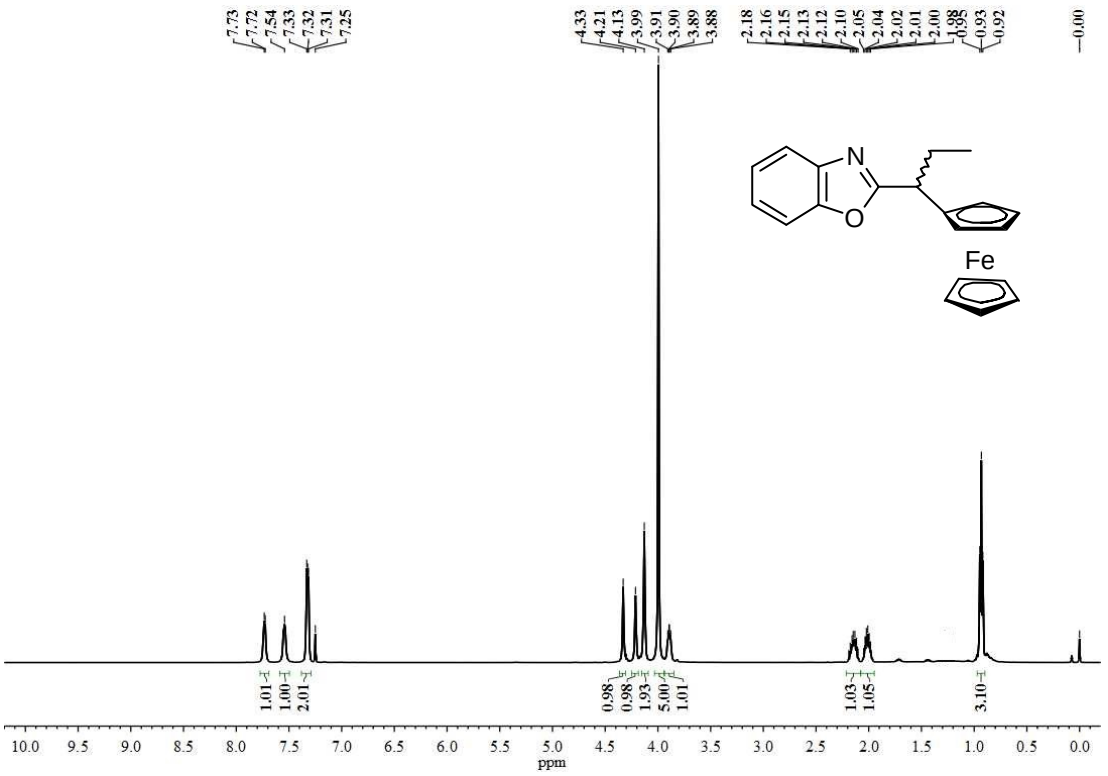

SRF-TQ-1 0:D14 MS Raw
Intens. [a.u.]
609.0461
8000
6000
4000
2000
0
600
602
604
606
608
610
612
614
616
618
m/z
O
N
Pd
Cl
Cl
Fe
N
O

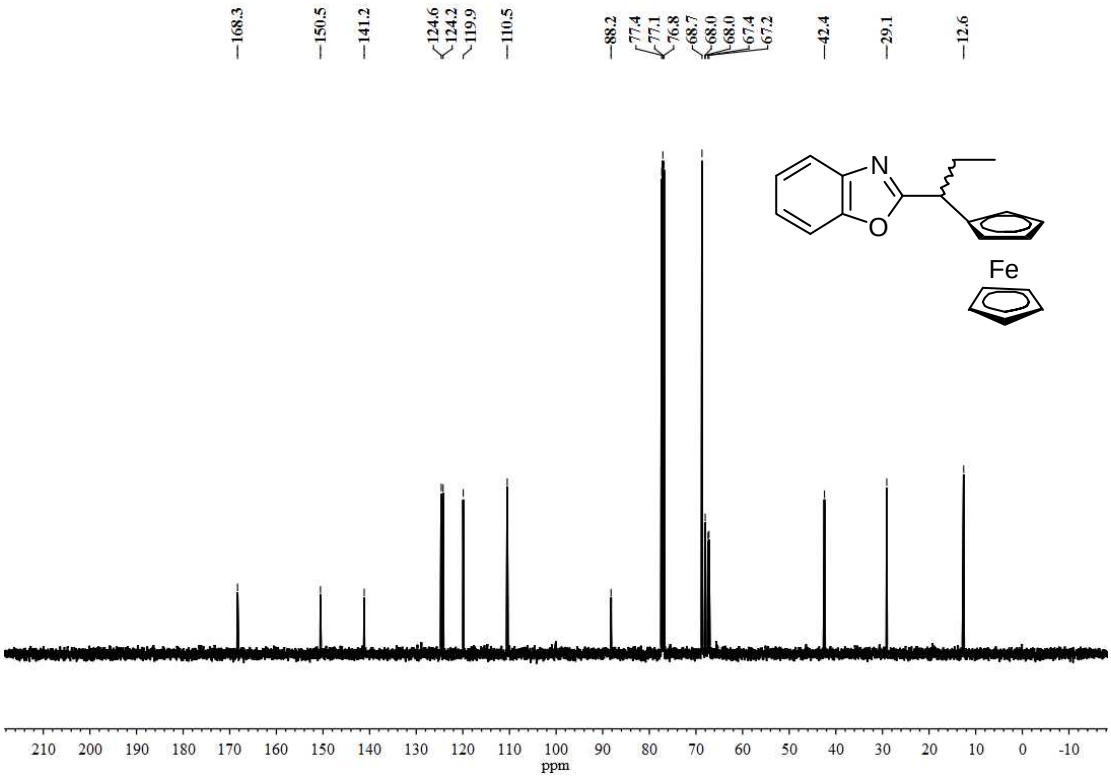

SRF-TQ-2 0:D15 MS Raw
Intens. [a.u.]
637.0790
4000
3000
2000
1000
0
632
634
636
638
640
642
m/z
O
N
Pd
Cl
Cl
Fe
N
O

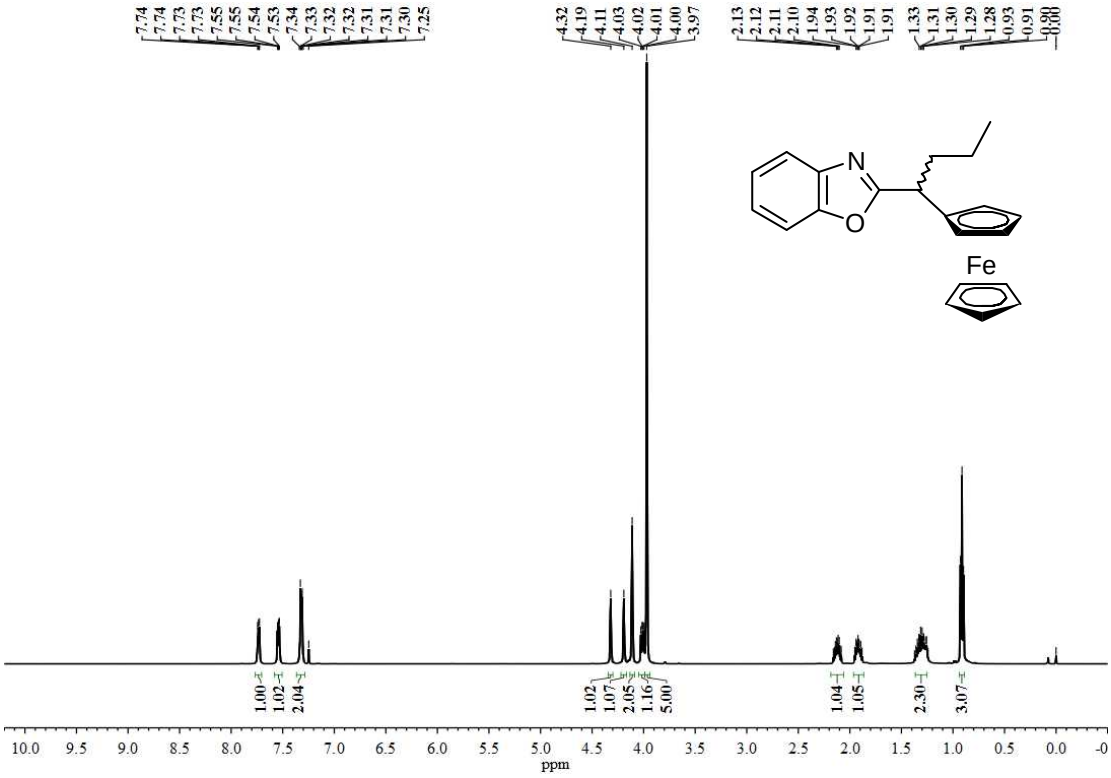
^{1}H NMR and ^{13}C NMR Spectra for the Products

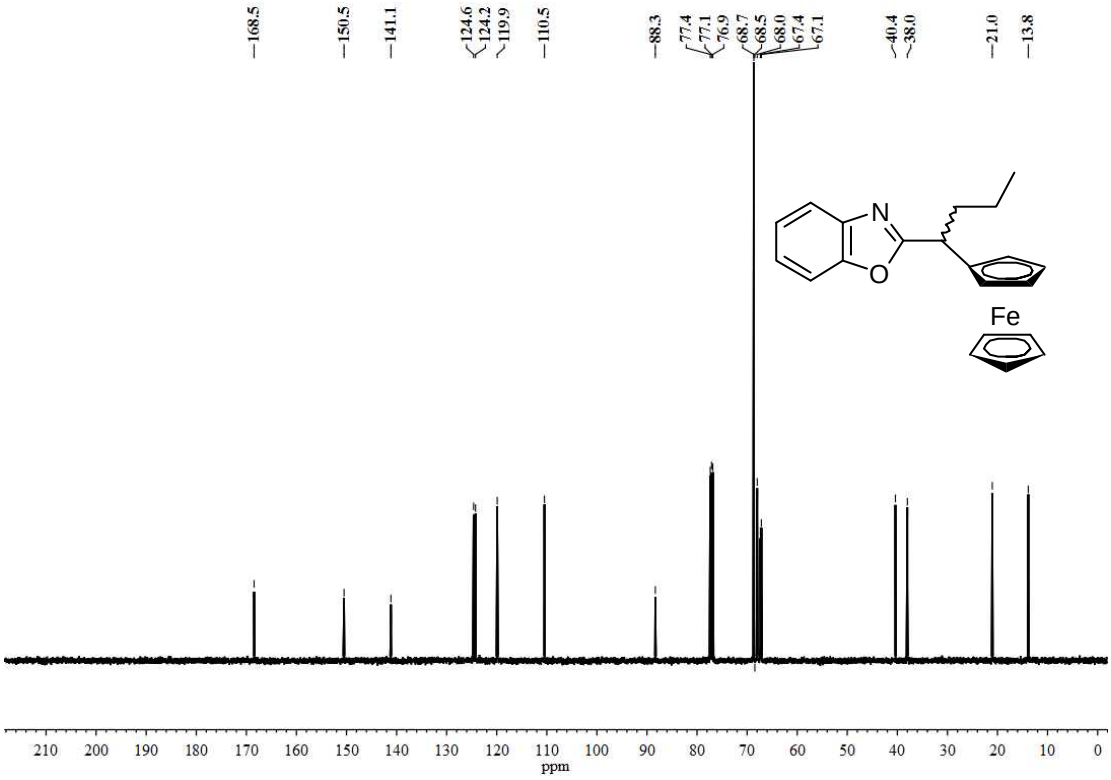


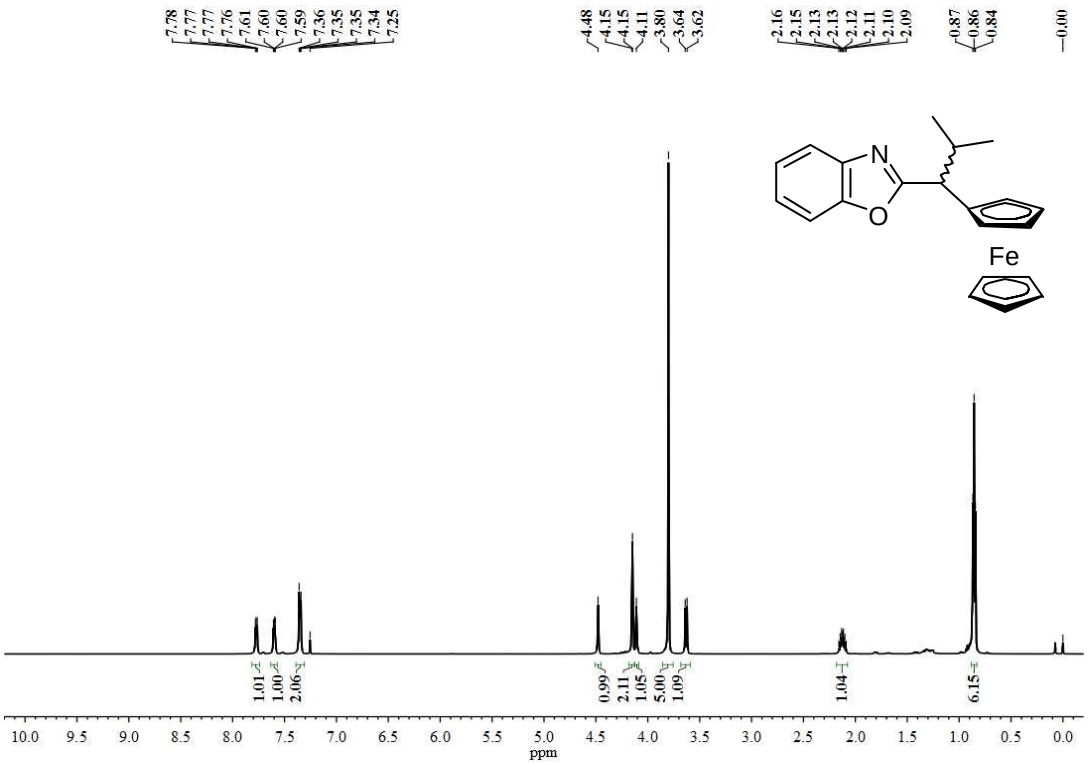



7.73
7.72
7.54
7.33
7.32
7.31
7.25
4.33
4.21
4.13
3.99
3.91
3.90
3.89
3.88
2.18
2.16
2.15
2.13
2.12
2.10
2.05
2.04
2.02
2.01
2.00
1.98
0.93
0.92
0.00
N
O
Fe
1.01
1.00
2.01
0.98
0.98
1.93
5.00
1.01
1.03
1.05
3.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm


168.3
150.5
141.2
124.6
124.2
119.9
110.5
88.2
77.4
77.1
76.8
68.7
68.0
68.0
67.4
67.2
42.4
29.1
12.6
N
O
Fe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm


7.74
7.74
7.73
7.73
7.55
7.55
7.54
7.53
7.34
7.33
7.32
7.32
7.31
7.31
7.30
7.25
4.32
4.19
4.11
4.03
4.02
4.01
4.00
3.97
2.13
2.12
2.11
2.10
1.94
1.93
1.92
1.91
1.91
1.33
1.31
1.30
1.29
1.28
0.93
0.91
0.80
0.00
N
O
Fe
1.00
1.02
2.04
1.02
1.07
2.05
1.16
5.00
1.04
1.05
2.30
3.07
10.0 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 -0
ppm


168.5
150.5
141.1
124.6
124.2
119.9
110.5
88.3
77.4
77.1
76.9
68.7
68.5
68.0
67.4
67.1
40.4
38.0
21.0
13.8
N
O
Fe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm


7.78
7.77
7.77
7.76
7.61
7.60
7.60
7.59
7.36
7.35
7.35
7.34
7.25
4.48
4.15
4.15
4.11
3.80
3.64
3.62
2.16
2.15
2.13
2.13
2.12
2.11
2.10
2.09
0.87
0.86
0.84
0.00
N
O
Fe
1.01
1.00
2.06
0.99
2.11
1.05
5.00
1.09
1.04
6.15
10.0 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
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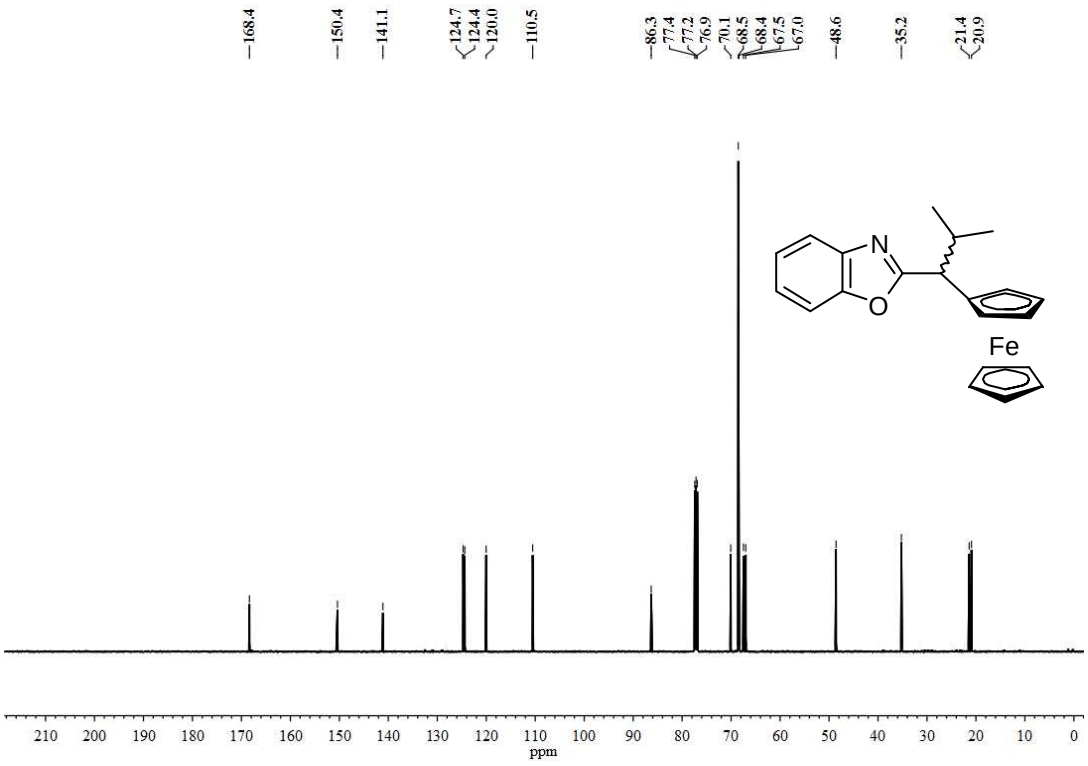
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68.4
67.5
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Fe
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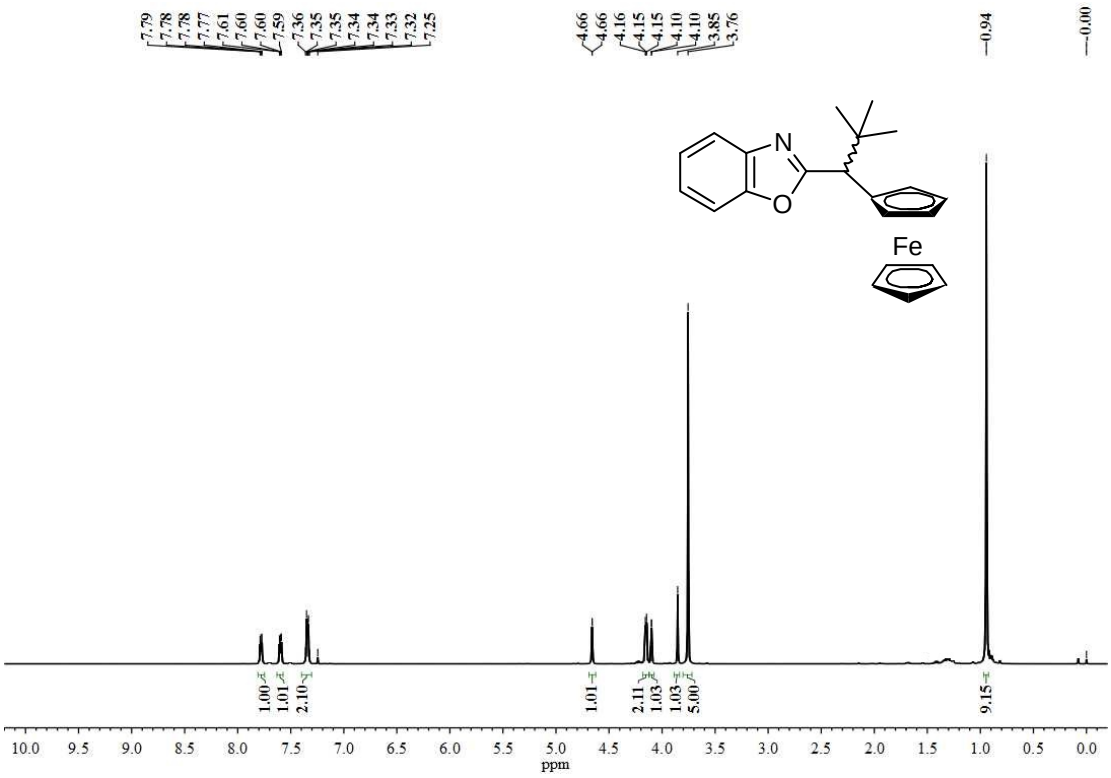
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7.34
7.33
7.32
7.25
4.66
4.66
4.16
4.15
4.15
4.10
4.10
3.85
3.76
0.94
0.00
N
O
Fe
1.00
1.01
2.10
1.01
2.11
1.03
1.03
5.00
9.15
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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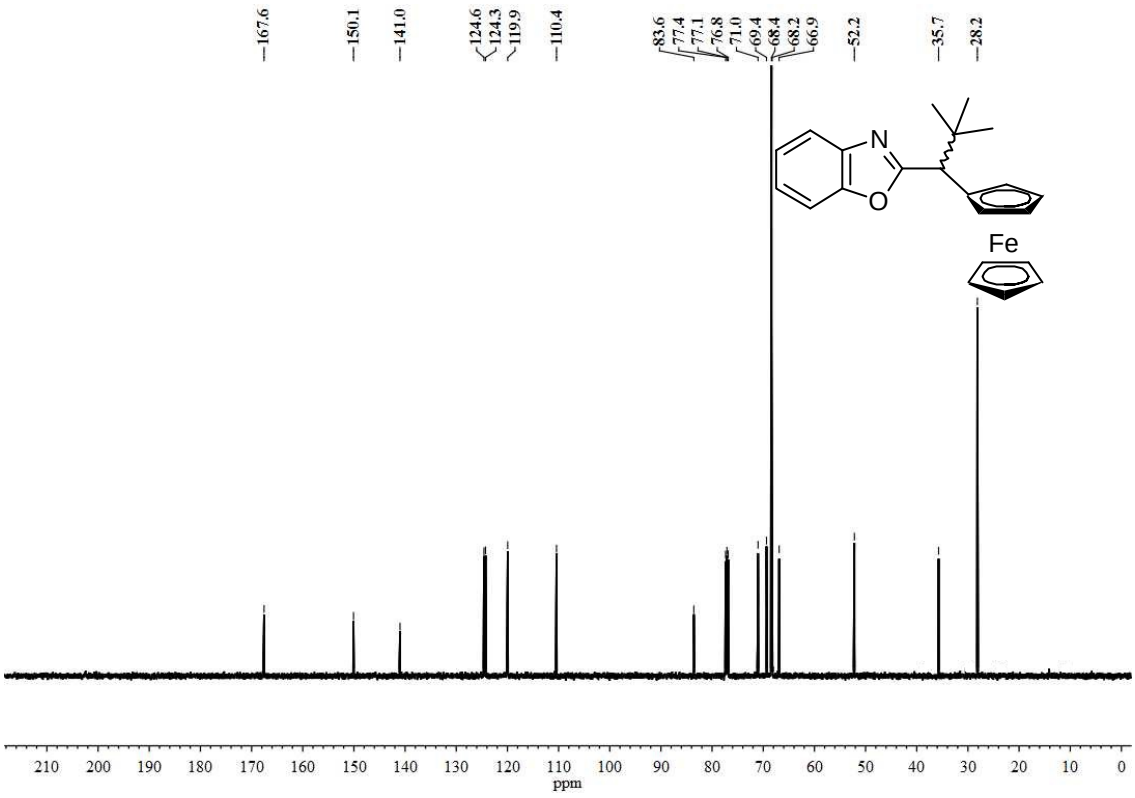
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77.1
76.8
71.0
69.4
68.4
68.2
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35.7
28.2
N
O
Fe
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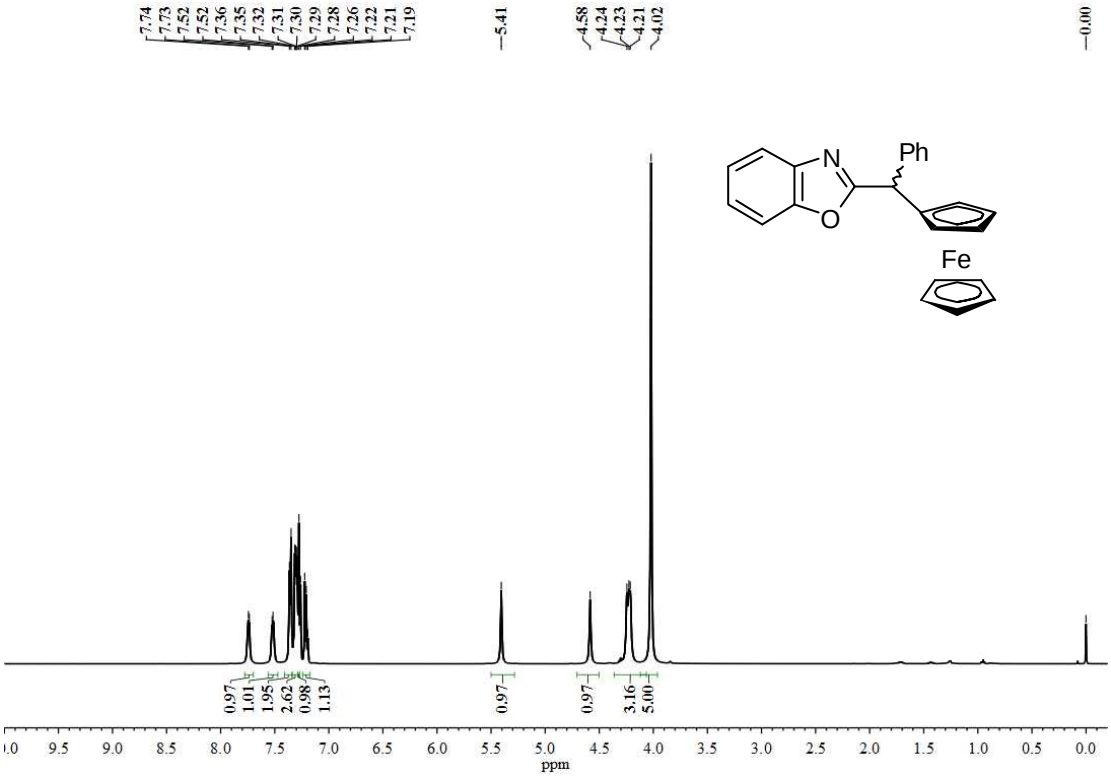
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7.21
7.19
5.41
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4.24
4.23
4.21
4.02
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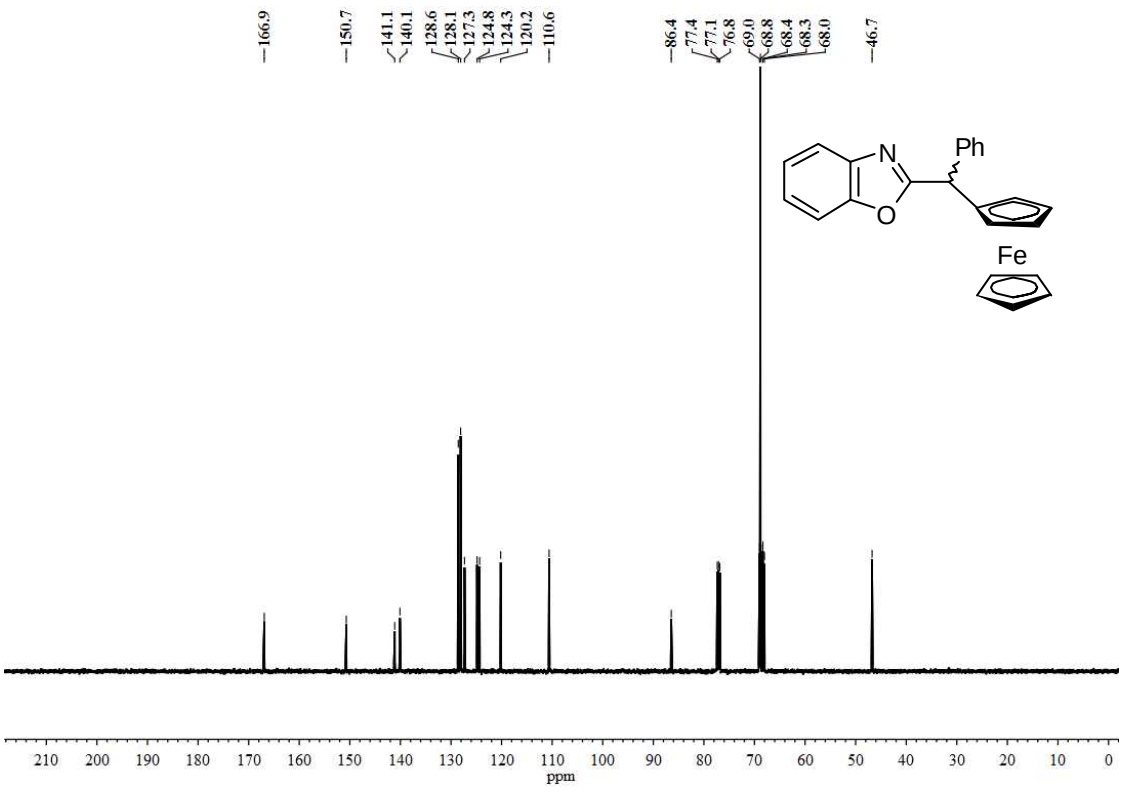
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110.6
86.4
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77.1
76.8
69.0
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68.0
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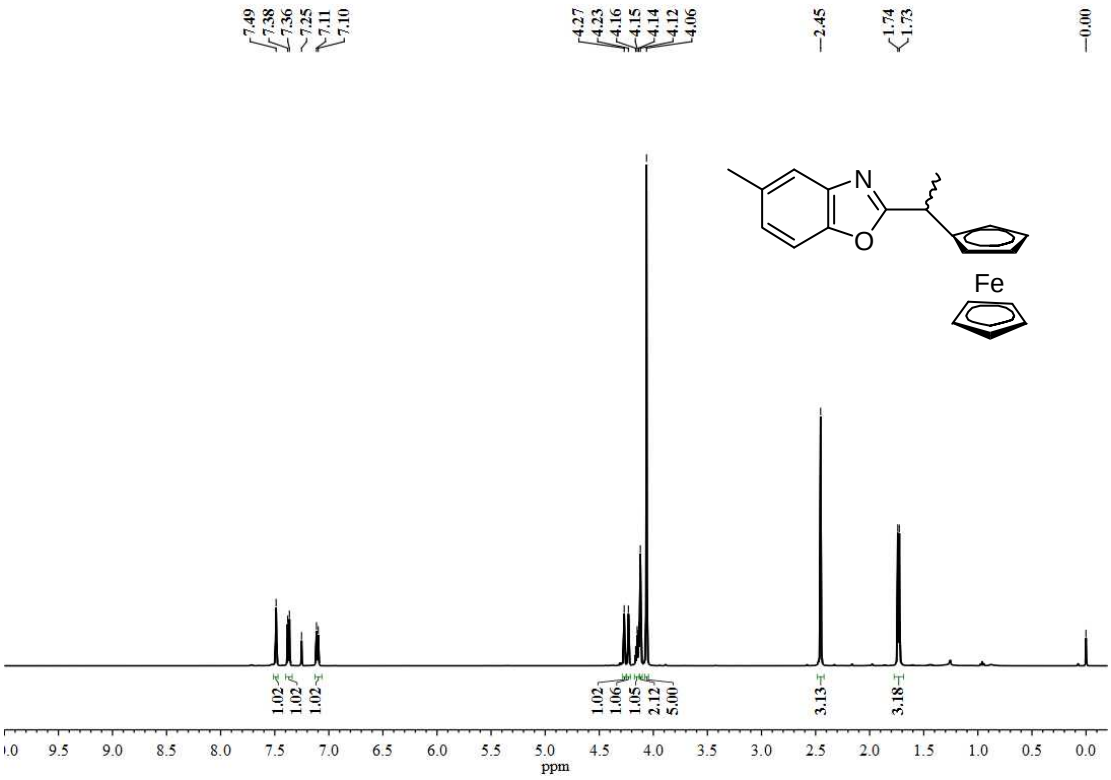
N
O
Ph
Fe
ppm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4.12
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2.45
1.74
1.73
0.00
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1.02
1.02
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2.12
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3.18
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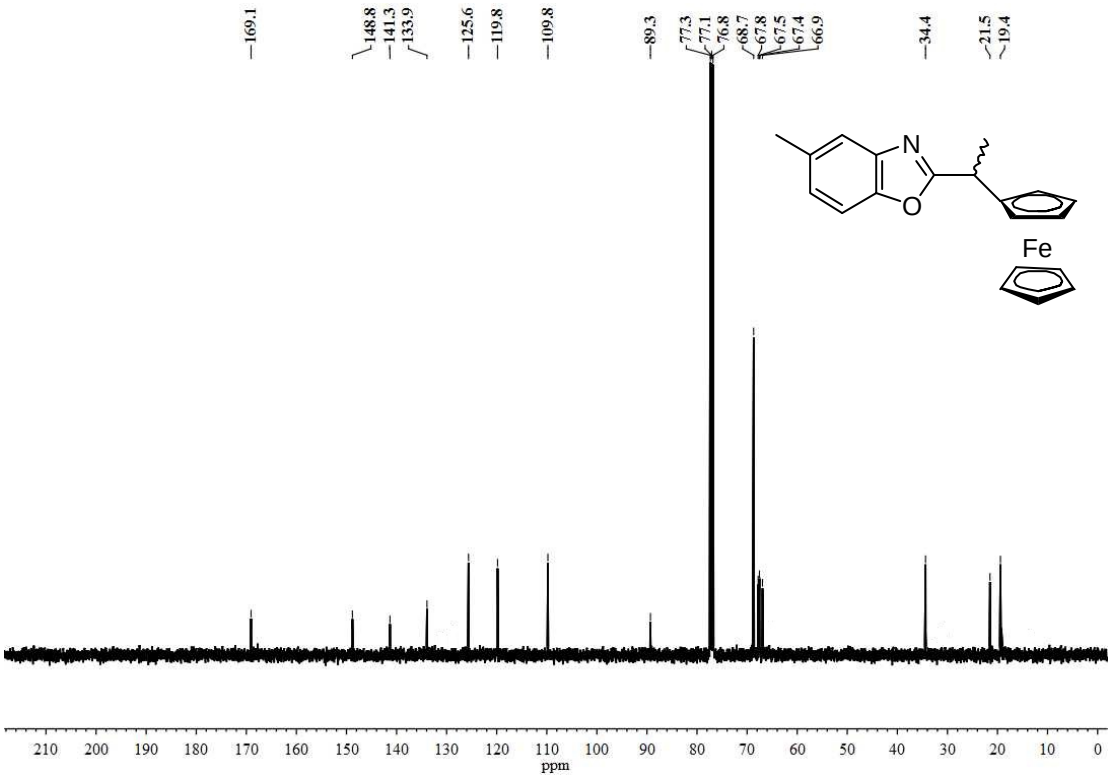
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67.5
67.4
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21.5
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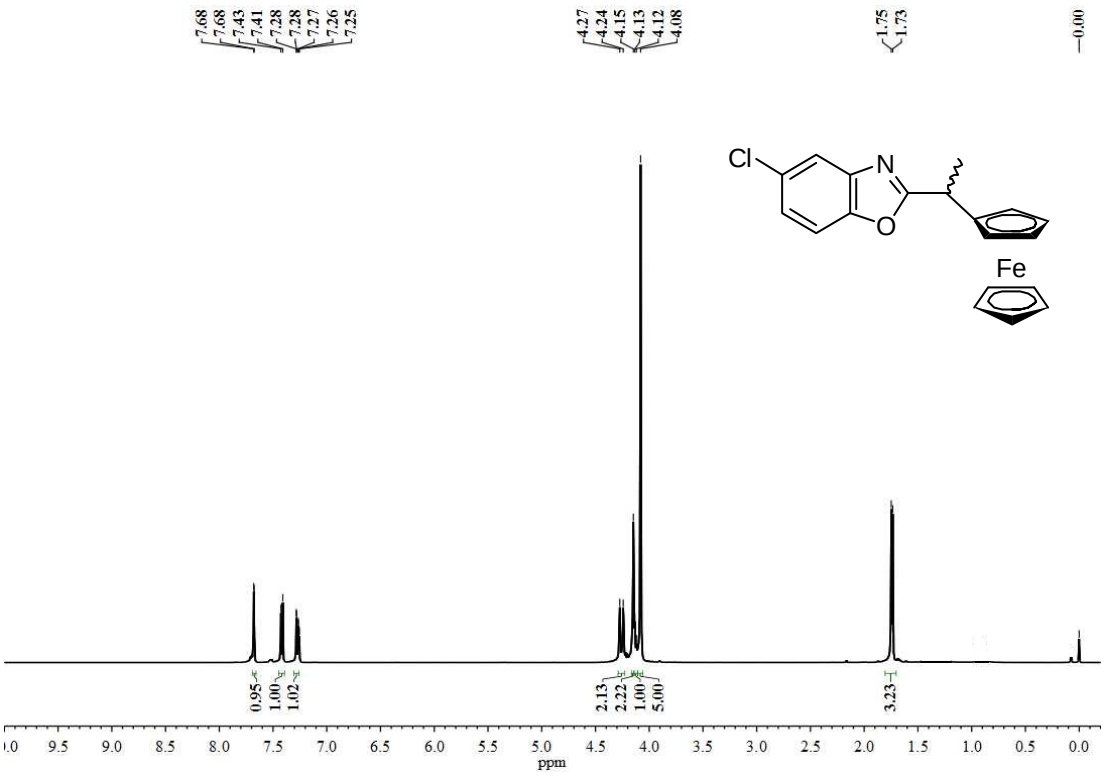
O
Fe
ppm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7.28
7.27
7.26
7.25
4.27
4.24
4.15
4.13
4.12
4.08
1.75
1.73
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O
Fe
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1.00
1.02
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2.22
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5.00
3.23
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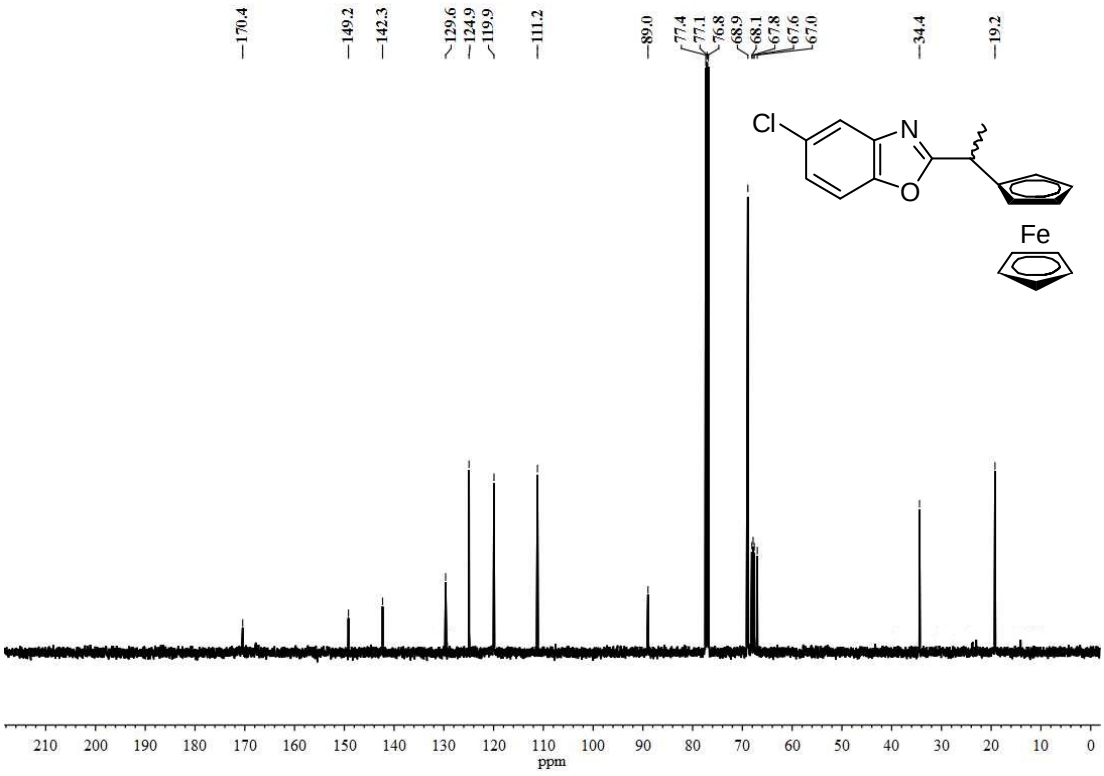
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119.9
111.2
89.0
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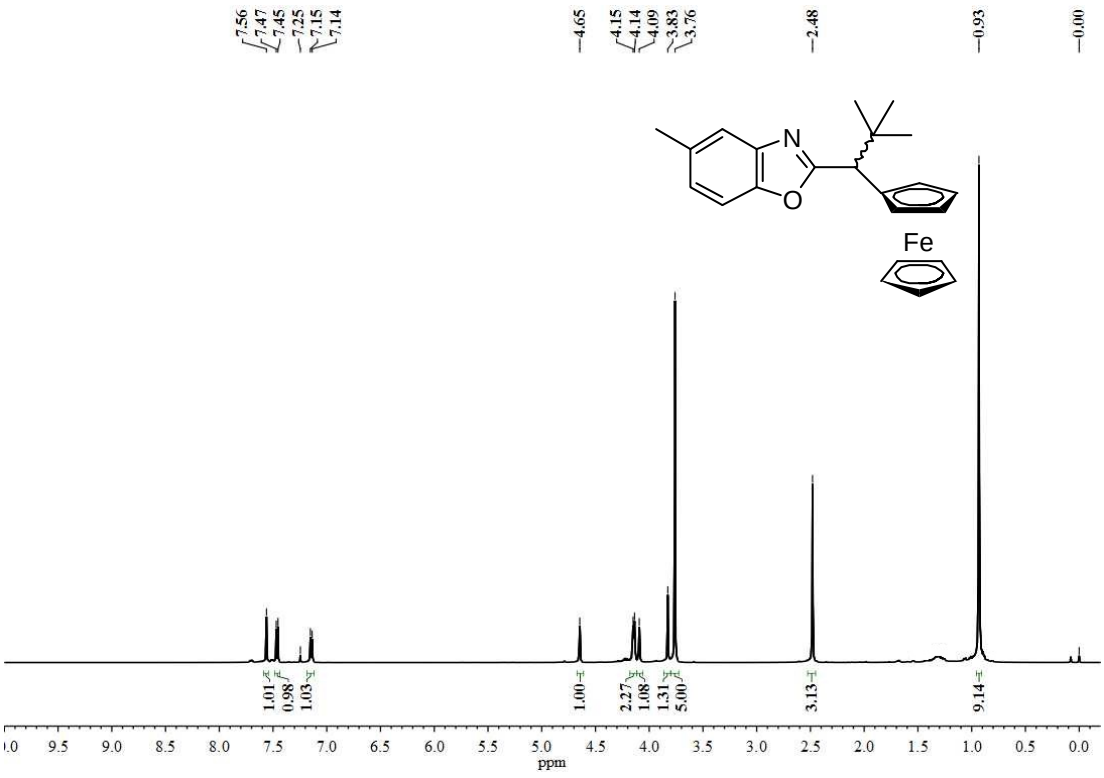
68.1
67.8
67.6
67.0
34.4
19.2
Cl
N
O
Fe
ppm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7.14
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4.15
4.14
4.09
3.83
3.76
2.48
0.93
0.00
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Fe
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1.31
5.00
3.13
9.14
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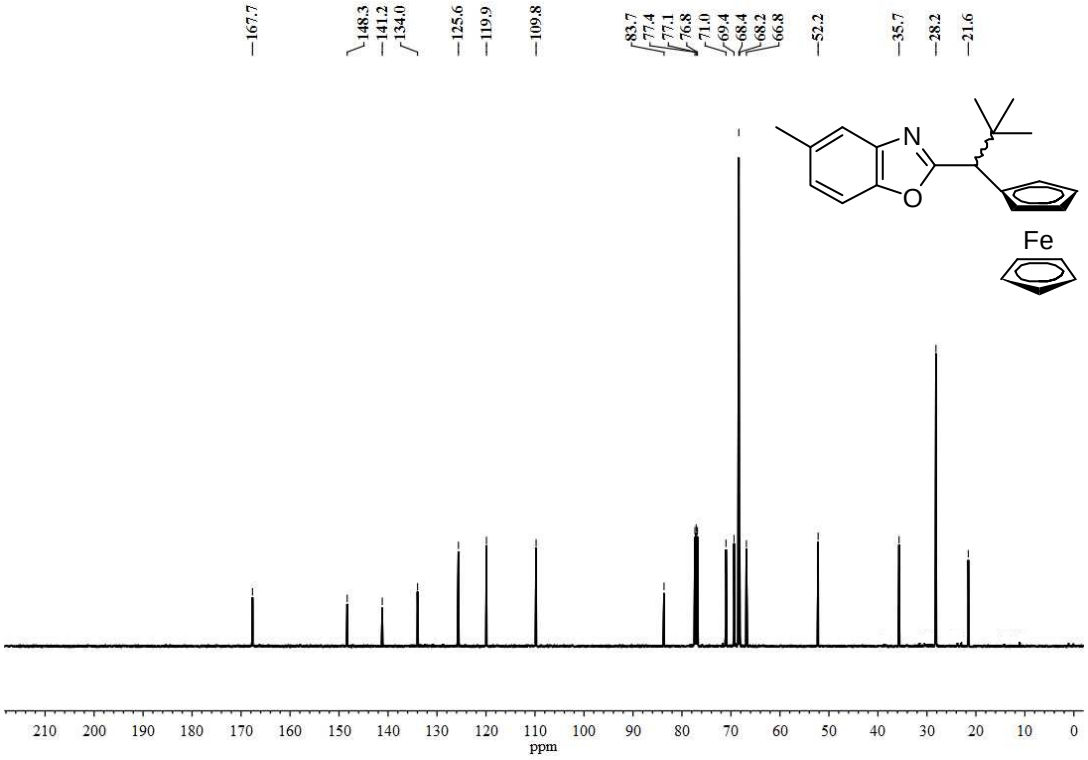
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119.9
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77.4
77.1
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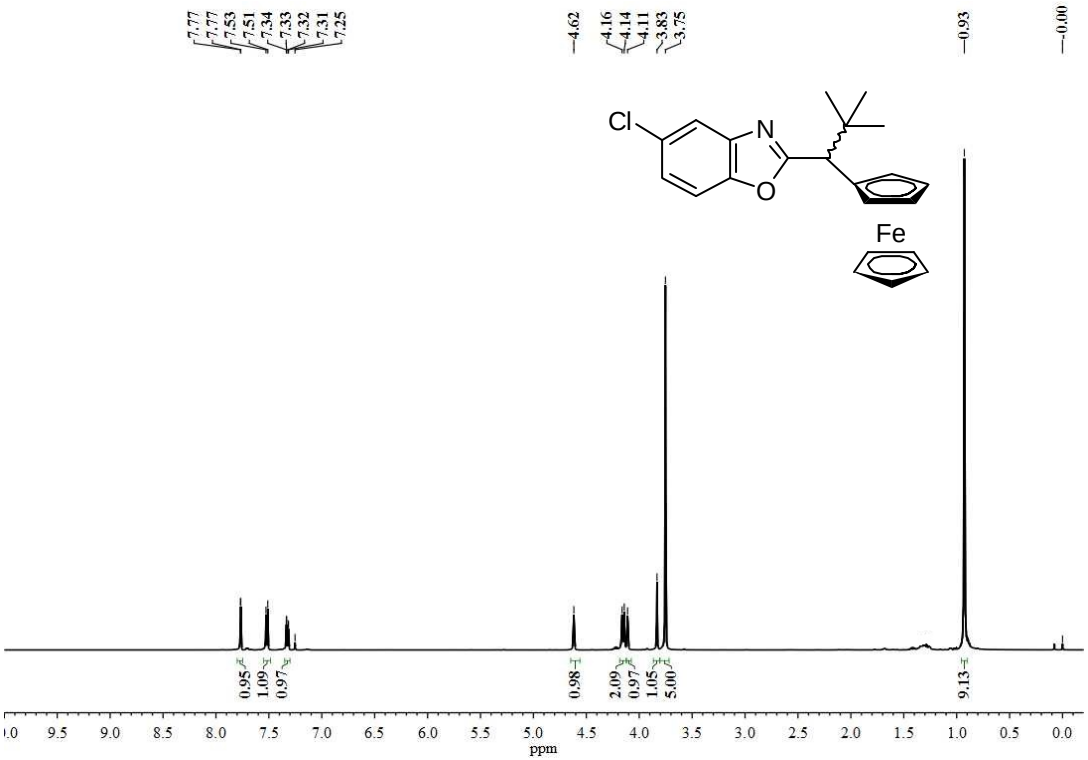
69.4
68.4
68.2
66.8
52.2
35.7
28.2
21.6
N
O
Fe
ppm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4.11
3.83
3.75
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0.97
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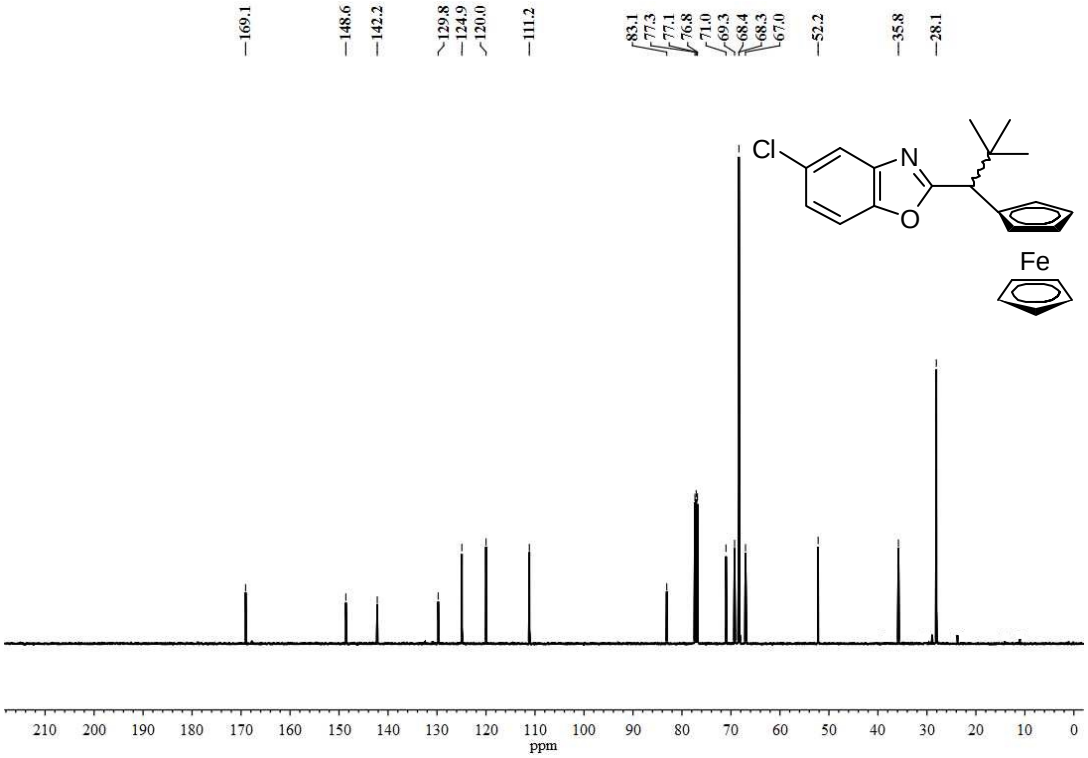
5.00
9.13
ppm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83.1
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77.1
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68.4
68.3
67.0
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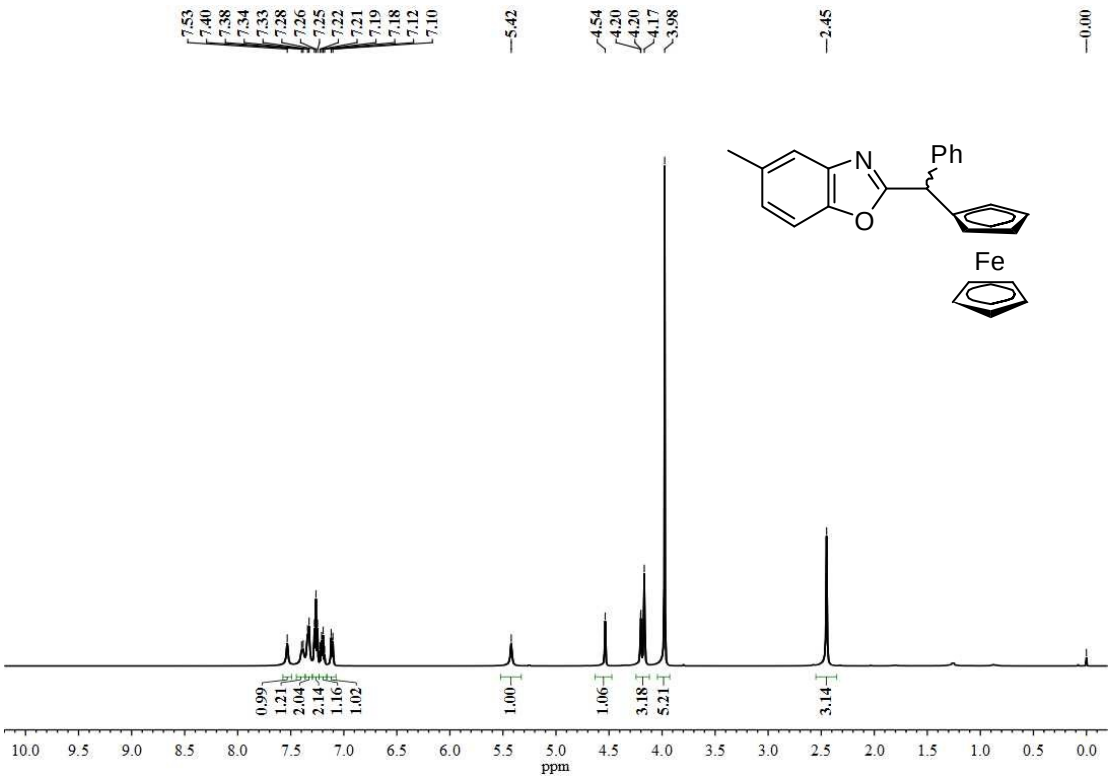
35.8
28.1
Cl
N
O
Fe
ppm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7.19
7.18
7.12
7.10
5.42
4.54
4.20
4.20
4.17
3.98
2.45
0.00
N
O
Ph
Fe
0.99
1.21
2.04
2.14
1.16
1.02
1.00
1.06
3.18
5.21
3.14
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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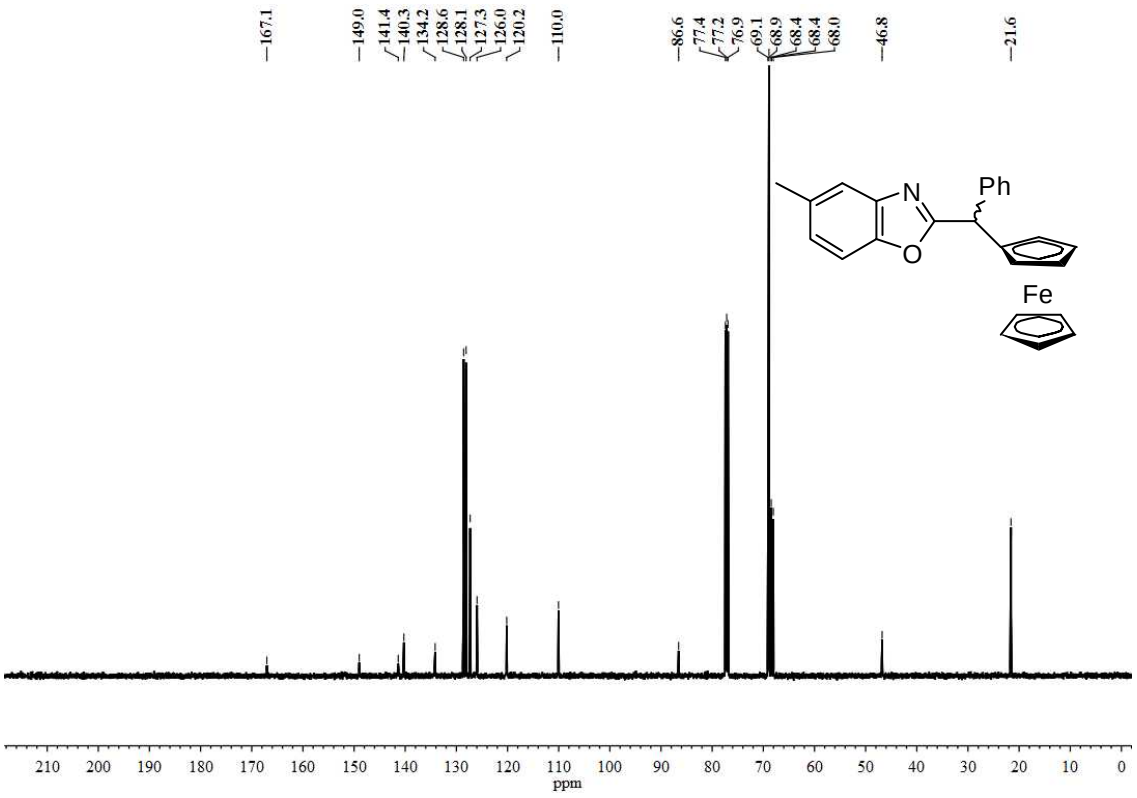
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127.3
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120.2
110.0
86.6
77.4
77.2
76.9
69.1
68.9
68.4
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46.8
21.6
N
O
Ph
Fe
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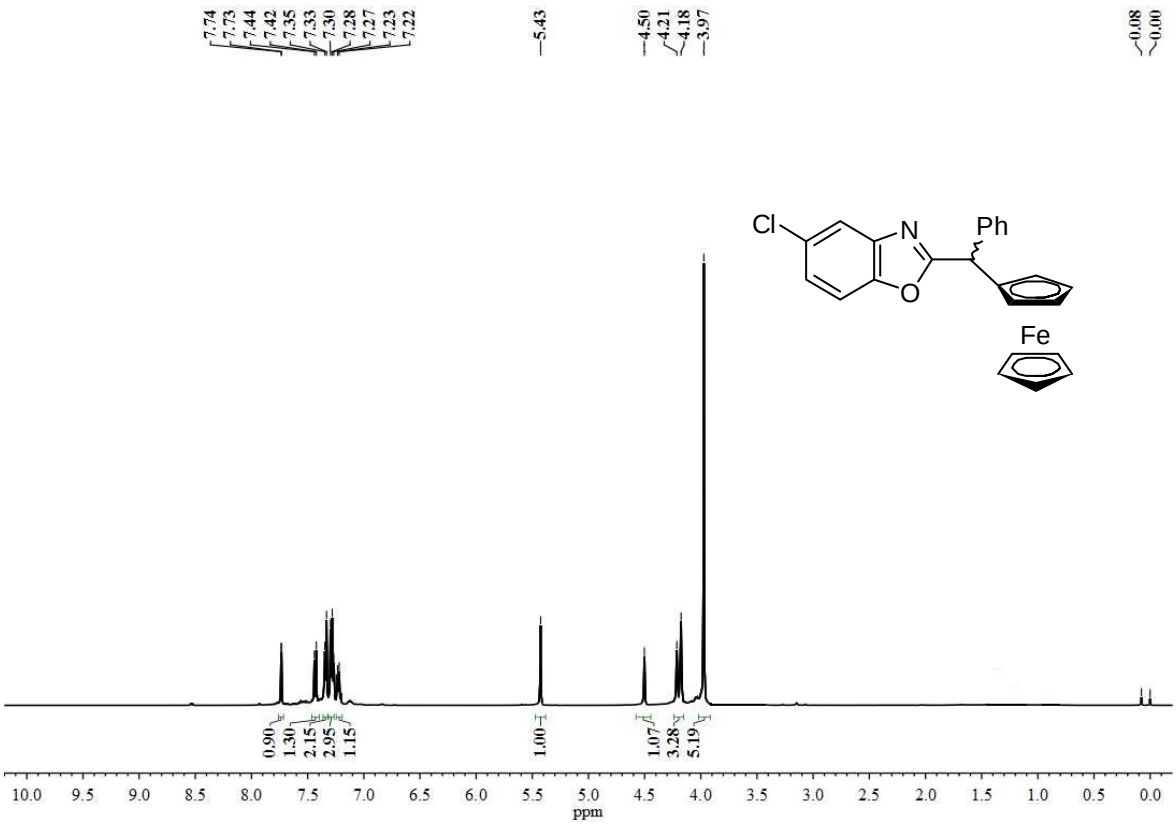
ppm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7.23
7.22
5.43
4.50
4.21
4.18
3.97
0.08
0.00
Cl
N
Ph
O
Fe
0.90
1.30
2.15
2.95
1.15
1.00
1.07
3.28
5.19
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
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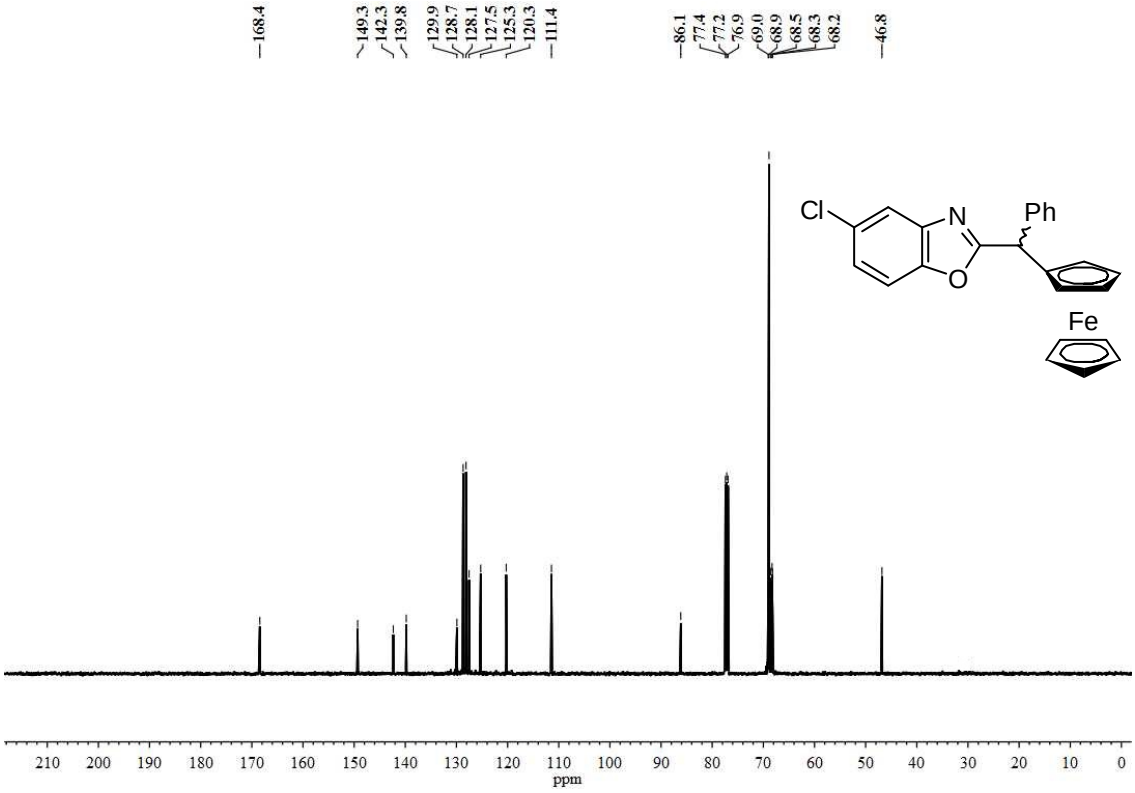
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120.3
111.4
86.1
77.4
77.2
76.9
69.0
68.9
68.5
68.3
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46.8
Cl
N
Ph
O
Fe
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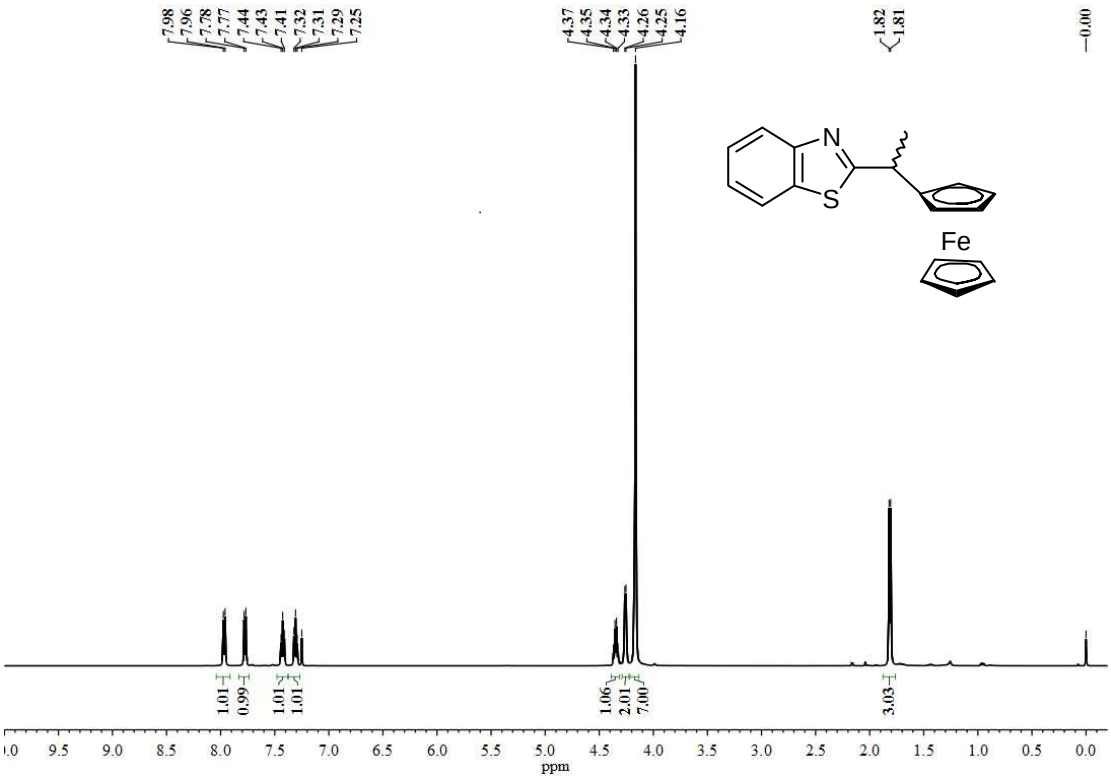
ppm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4.34
4.33
4.26
4.25
4.16
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S
Fe
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1.06
2.01
7.00
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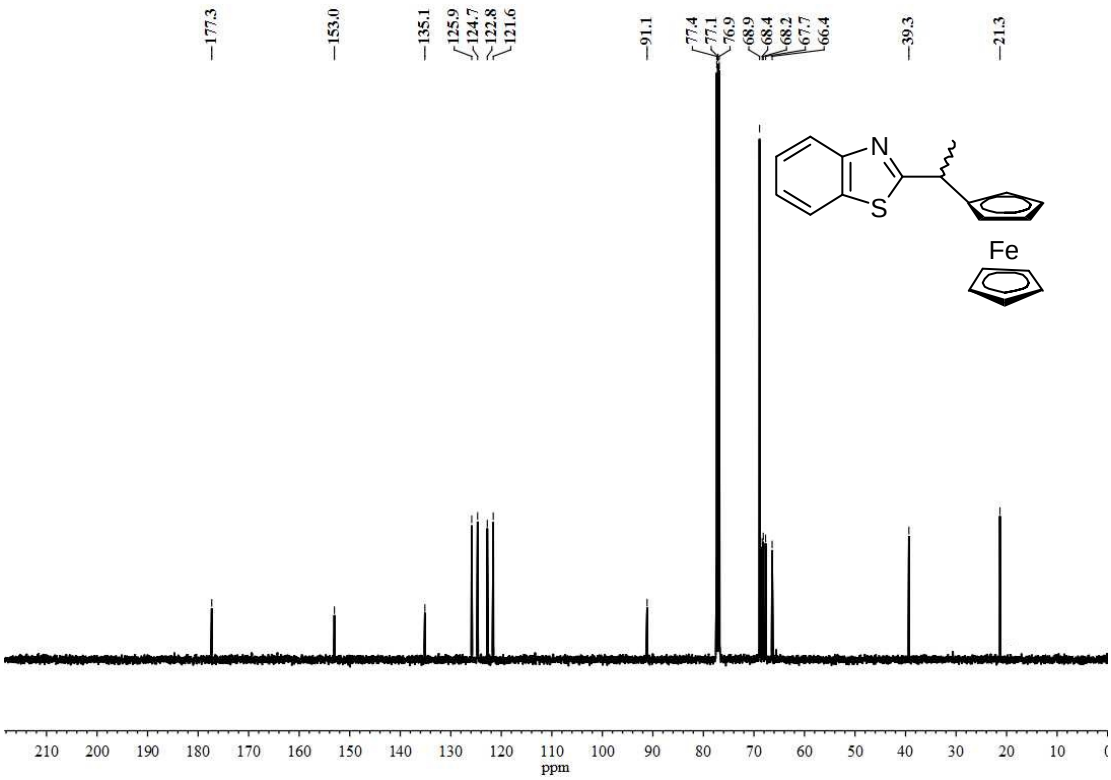
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122.8
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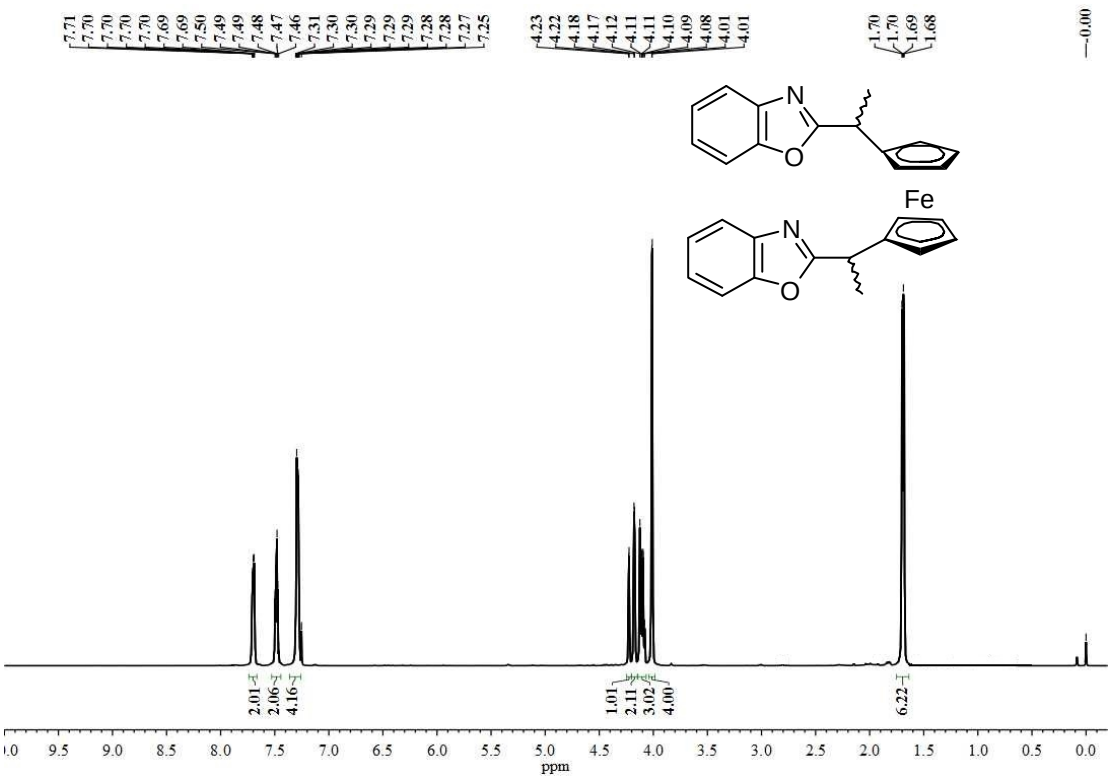
68.9
68.4
68.2
67.7
66.4
39.3
21.3
N
S
Fe
ppm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7.29
7.29
7.28
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7.27
7.25
4.23
4.22
4.18
4.17
4.12
4.11
4.11
4.10
4.09
4.08
4.01
4.01
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1.70
1.69
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O
Fe
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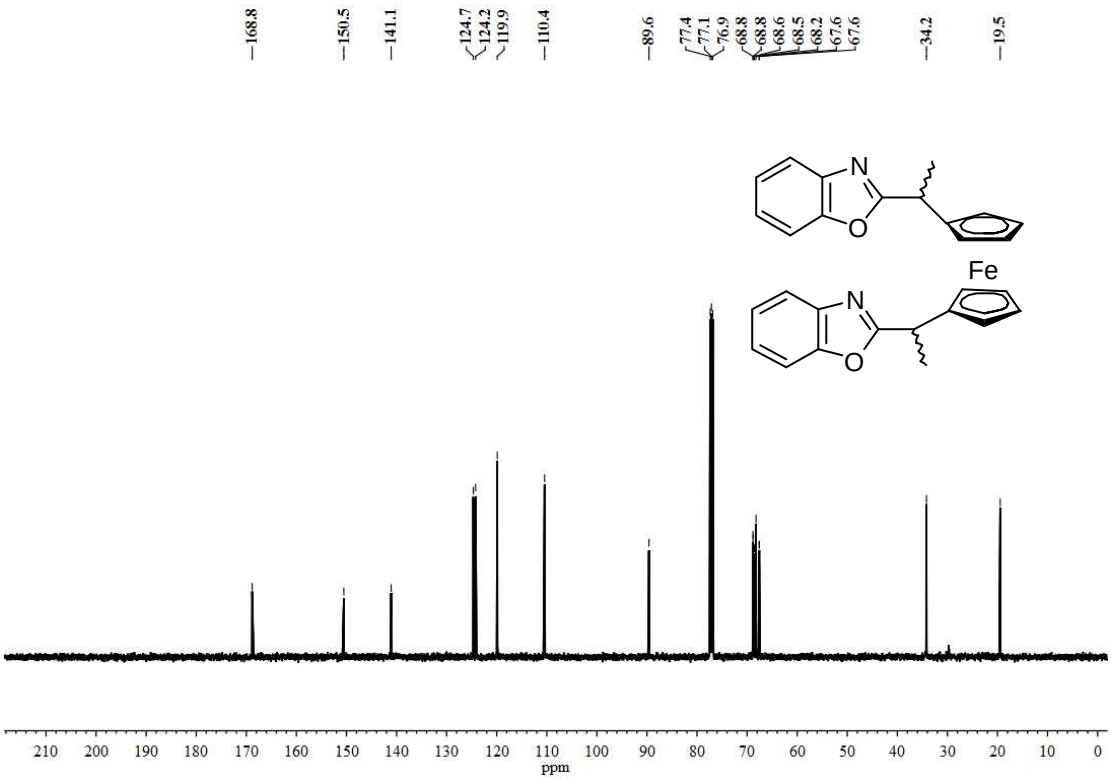
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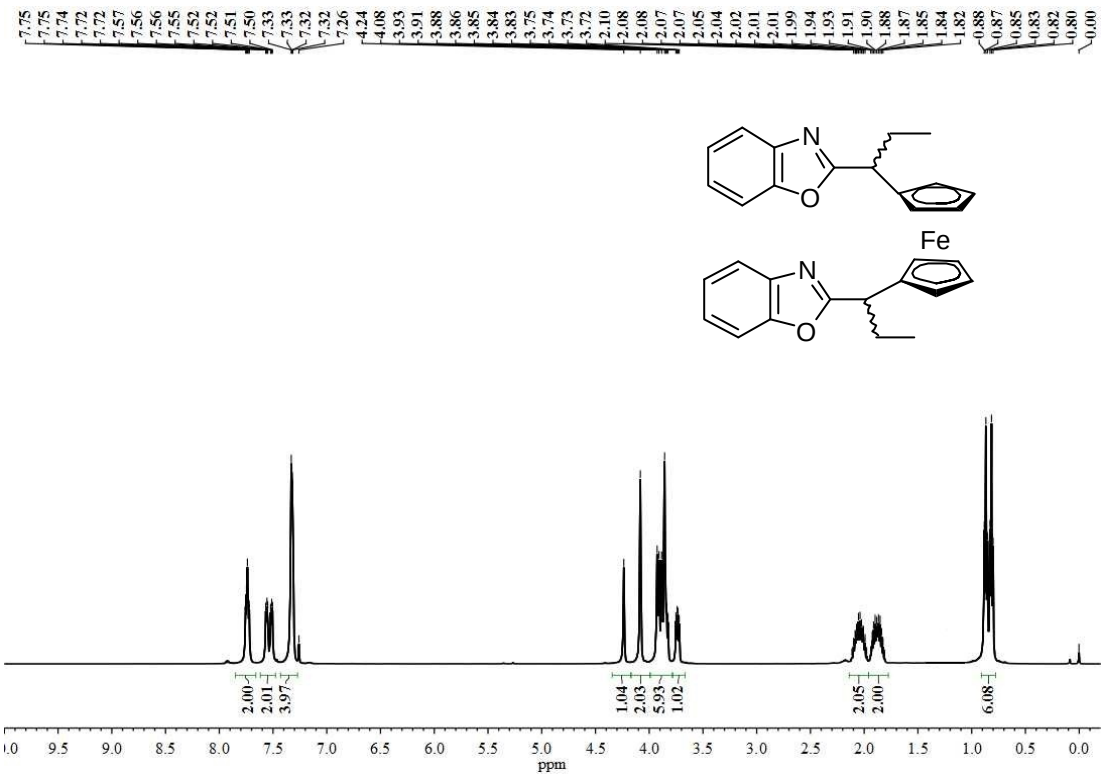
67.6
34.2
19.5
N
O
Fe
ppm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7.33
7.32
7.32
7.26
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4.08
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3.91
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3.86
3.85
3.84
3.83
3.75
3.74
3.73
3.72
2.10
2.08
2.08
2.07
2.07
2.05
2.04
2.02
2.01
2.01
1.99
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1.93
1.91
1.90
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1.87
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1.84
1.82
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0.87
0.85
0.83
0.82
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2.00
6.08
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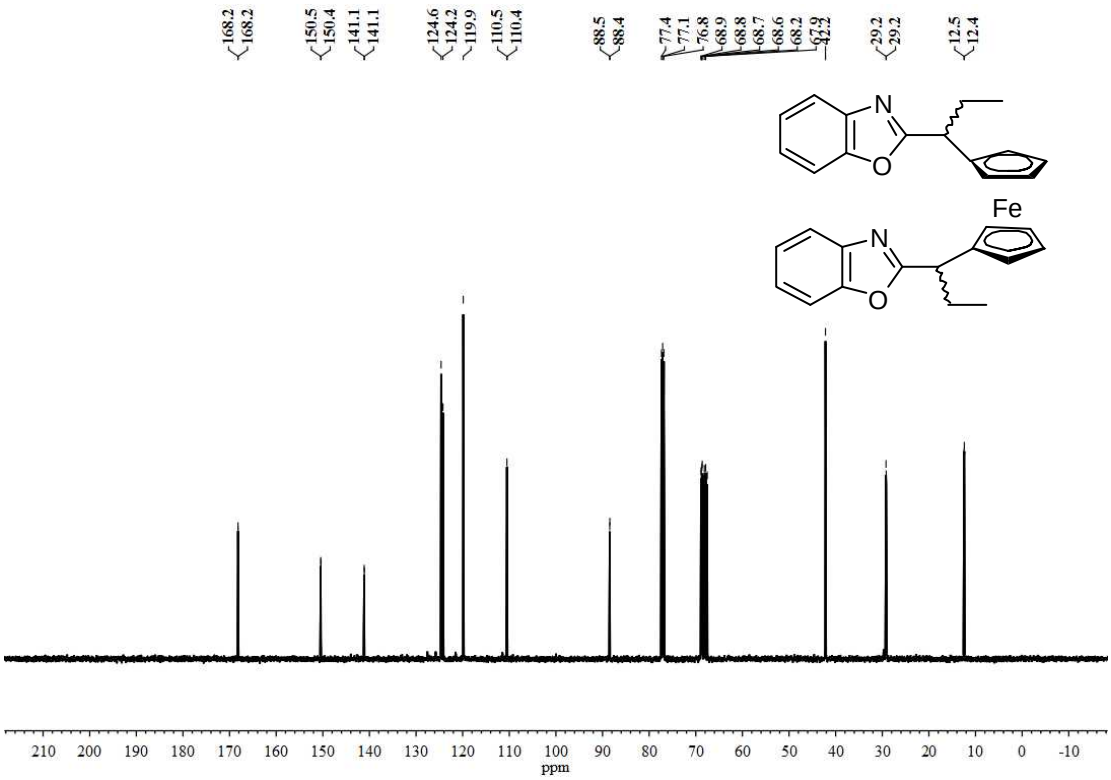
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124.2
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110.4
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88.4
77.4
77.1
76.8
68.9
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68.2
43.2
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29.2
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12.4
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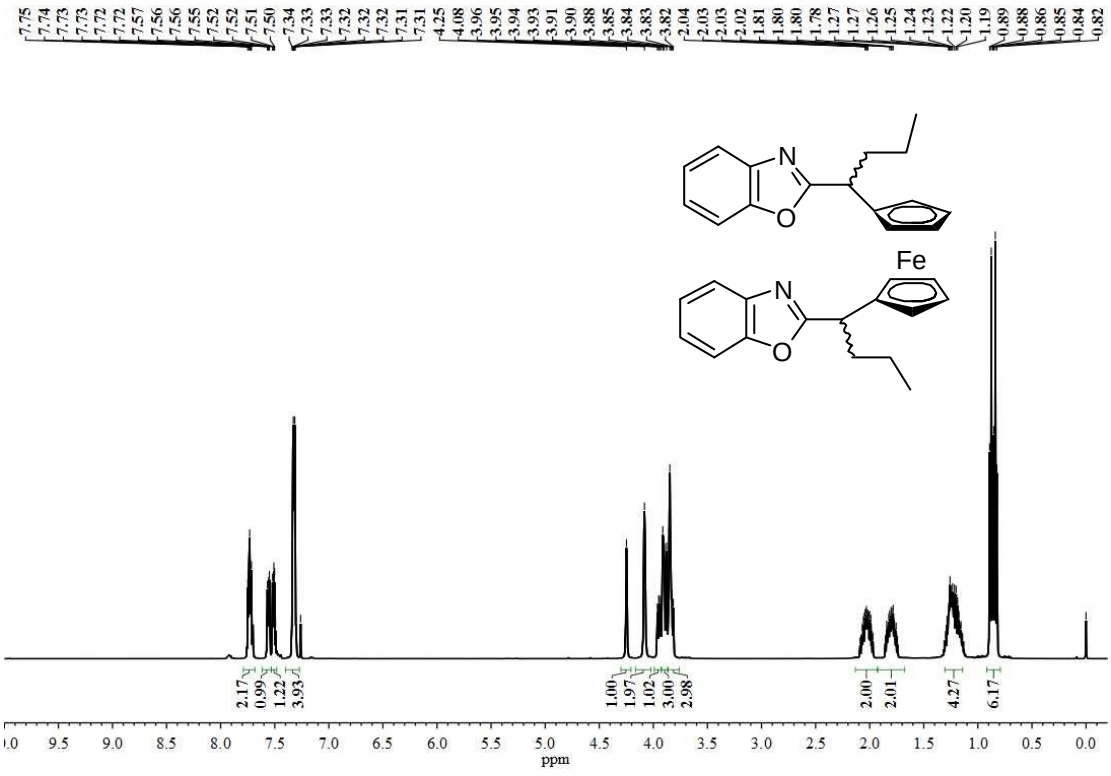
Fe
N
O
ppm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7.51
7.50
7.34
7.33
7.33
7.32
7.32
7.32
7.31
7.31
4.25
4.08
3.96
3.95
3.94
3.93
3.91
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2.03
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1.27
1.27
1.26
1.25
1.24
1.23
1.22
1.20
1.19
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0.88
0.86
0.85
0.84
0.82
N
O
Fe
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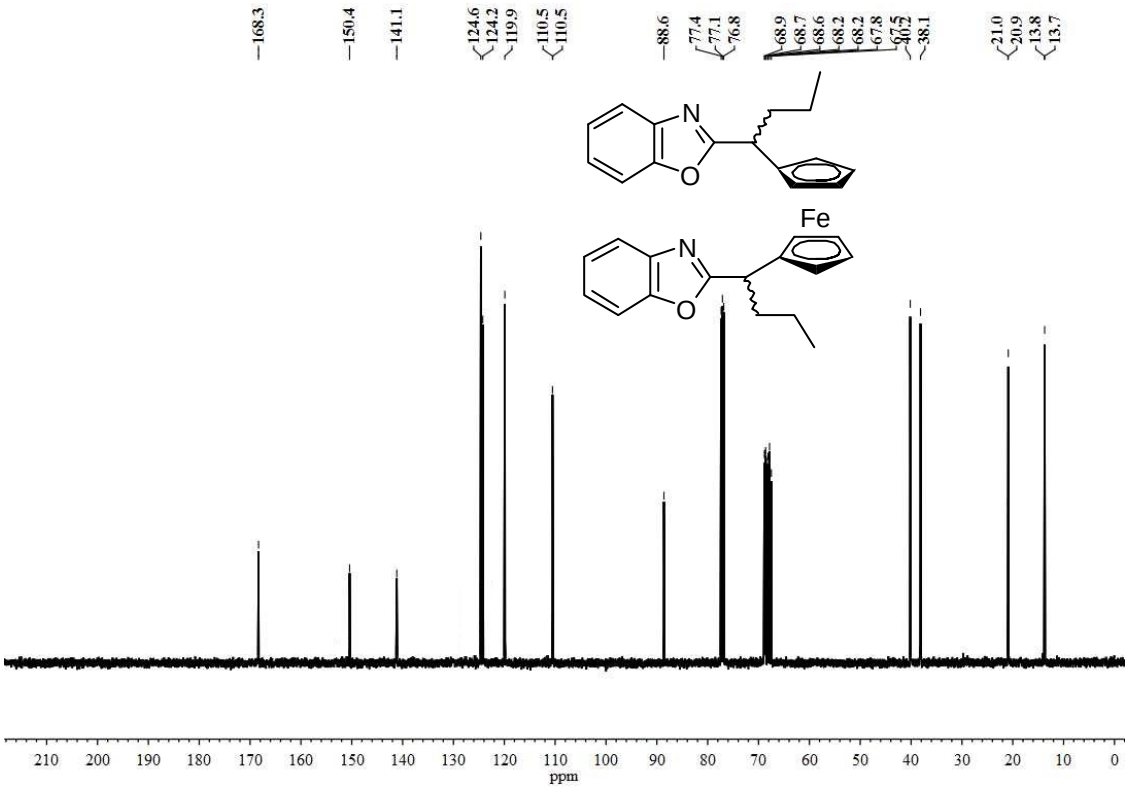
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124.6
124.2
119.9
110.5
110.5
88.6
77.4
77.1
76.8
68.9
68.7
68.6
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67.5
40.7
38.1
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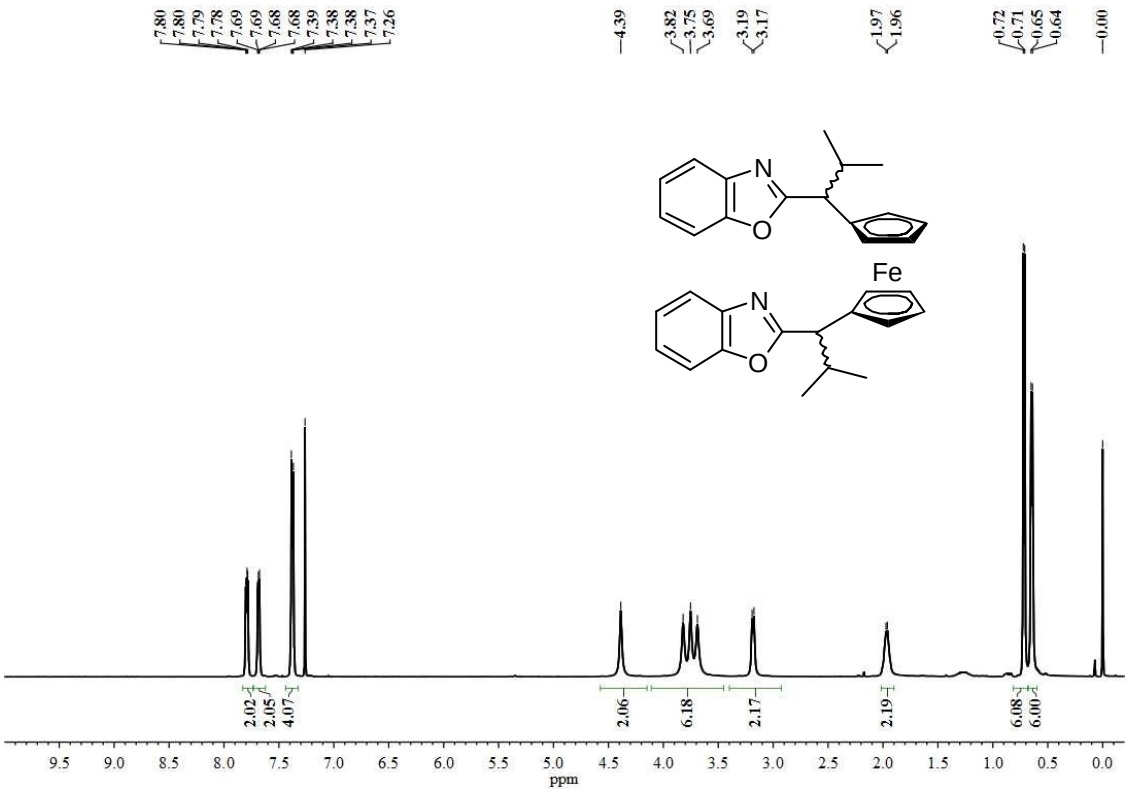
13.8
13.7
N
O
Fe
ppm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7.26
4.39
3.82
3.75
3.69
3.19
3.17
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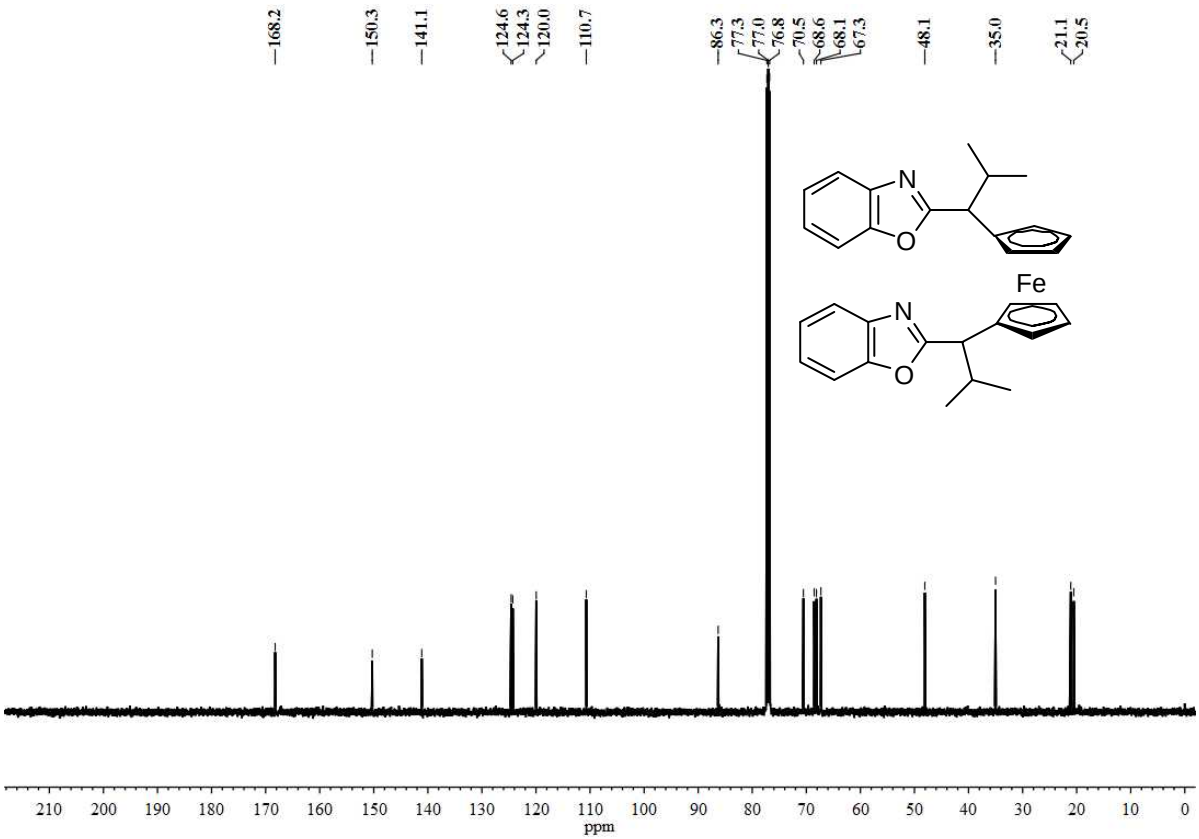
1.96
0.72
0.71
0.65
0.64
0.00
N
O
Fe
2.02
2.05
4.07
2.06
6.18
2.17
2.19
6.08
6.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm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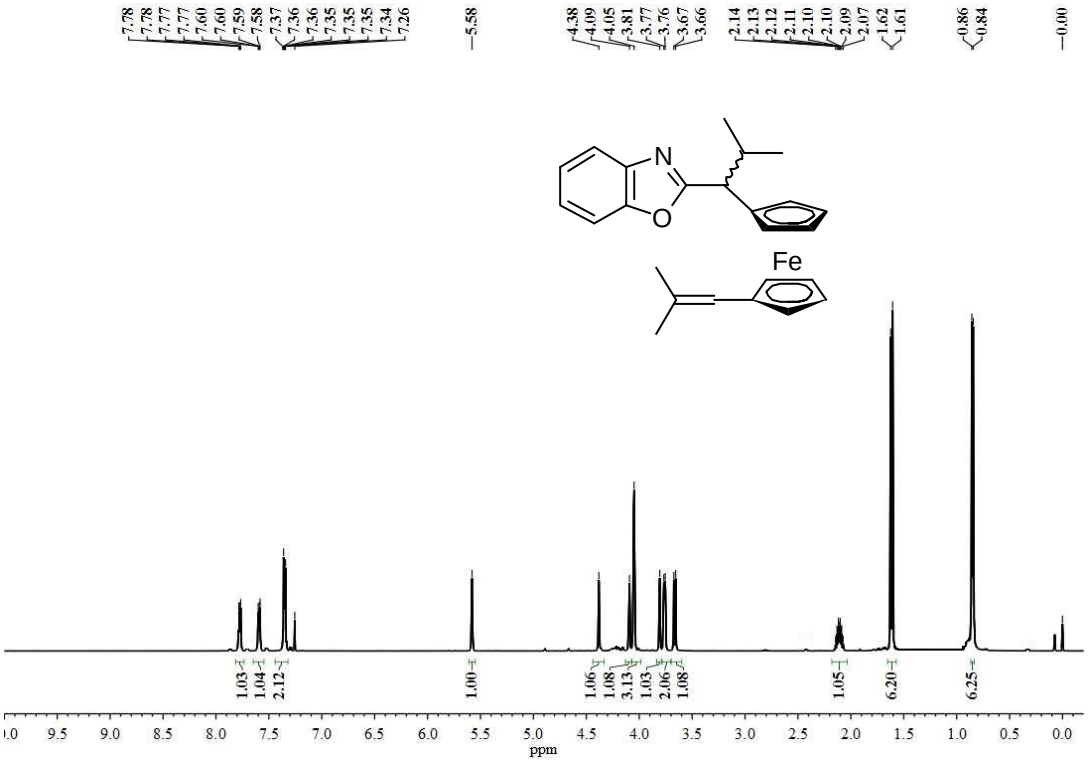
150.3
141.1
124.6
124.3
120.0
110.7
86.3
77.3
77.0
76.8
70.5
68.6
68.1
67.3
48.1
35.0
21.1
20.5
N
O
Fe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm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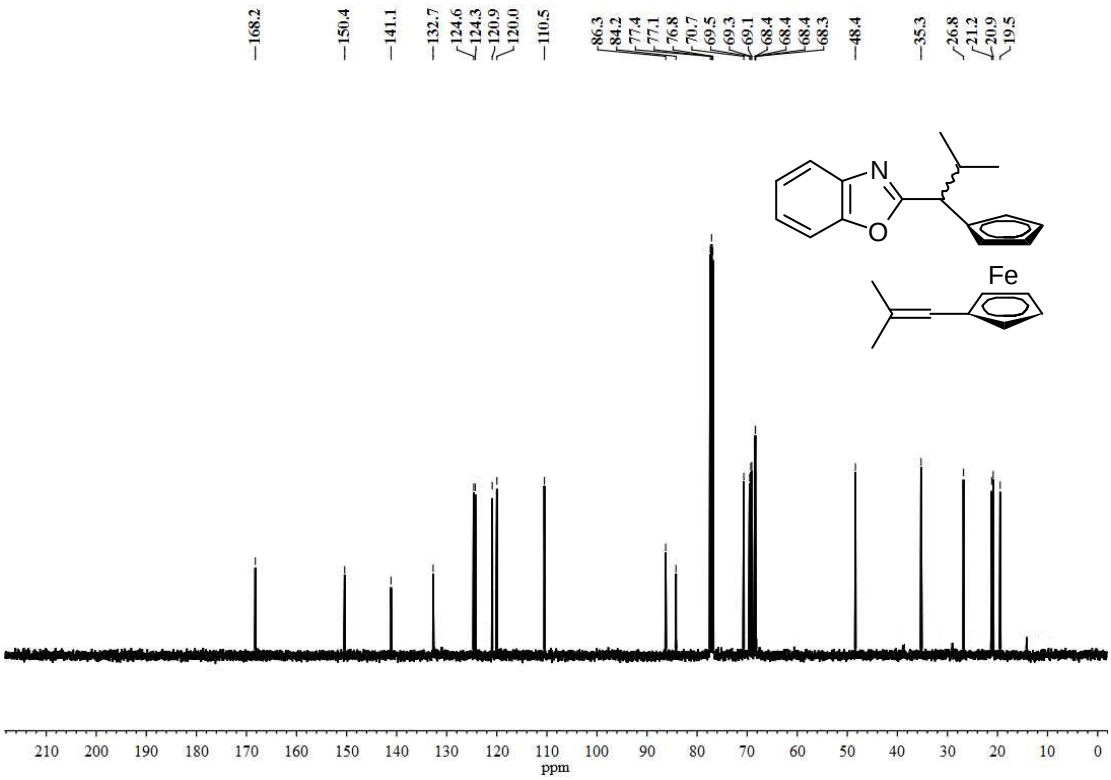
7.78
7.77
7.77
7.60
7.60
7.59
7.58
7.37
7.36
7.36
7.35
7.35
7.35
7.34
7.26
5.58
4.38
4.09
4.05
3.81
3.77
3.76
3.67
3.66
2.14
2.13
2.12
2.11
2.10
2.10
2.09
2.07
1.62
1.61
0.86
0.84
0.00
N
O
Fe
1.03
1.04
2.12
1.00
1.06
1.08
3.13
1.03
2.06
1.08
1.05
6.20
6.25
ppm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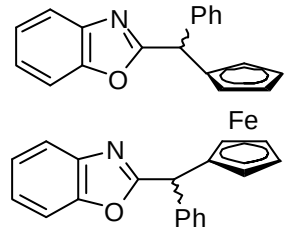
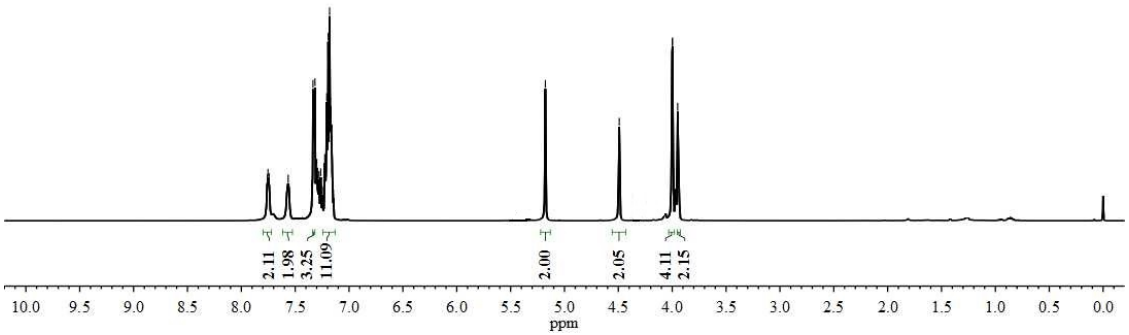
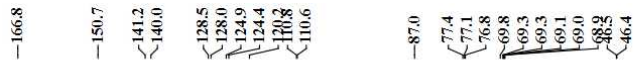
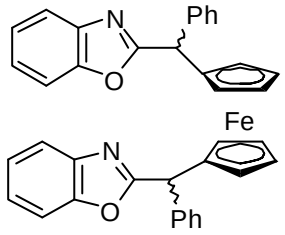
150.4
141.1
132.7
124.6
124.3
120.9
120.0
110.5
86.3
84.2
77.4
77.1
76.8
70.7
69.5
69.3
69.1
68.4
68.4
68.4
68.3
48.4
35.3
26.8
21.2
20.9
19.5
N
O
Fe
ppm


7.76
7.75
7.74
7.57
7.34
7.33
7.33
7.32
7.32
7.31
7.30
7.29
7.28
7.26
7.25
7.23
7.22
7.22
7.21
7.21
7.20
7.19
7.18
7.17
7.17
7.16
7.14
7.13
4.49
4.00
4.00
3.95

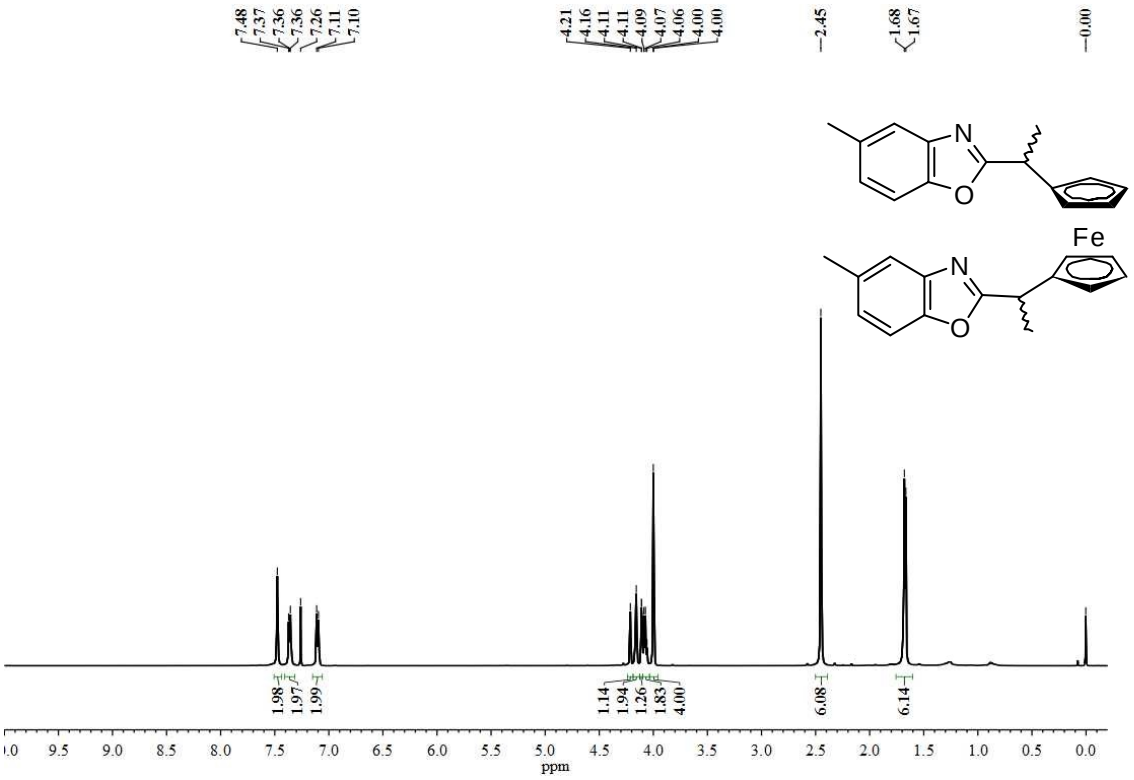

N
O
Ph
Fe
N
O
Ph

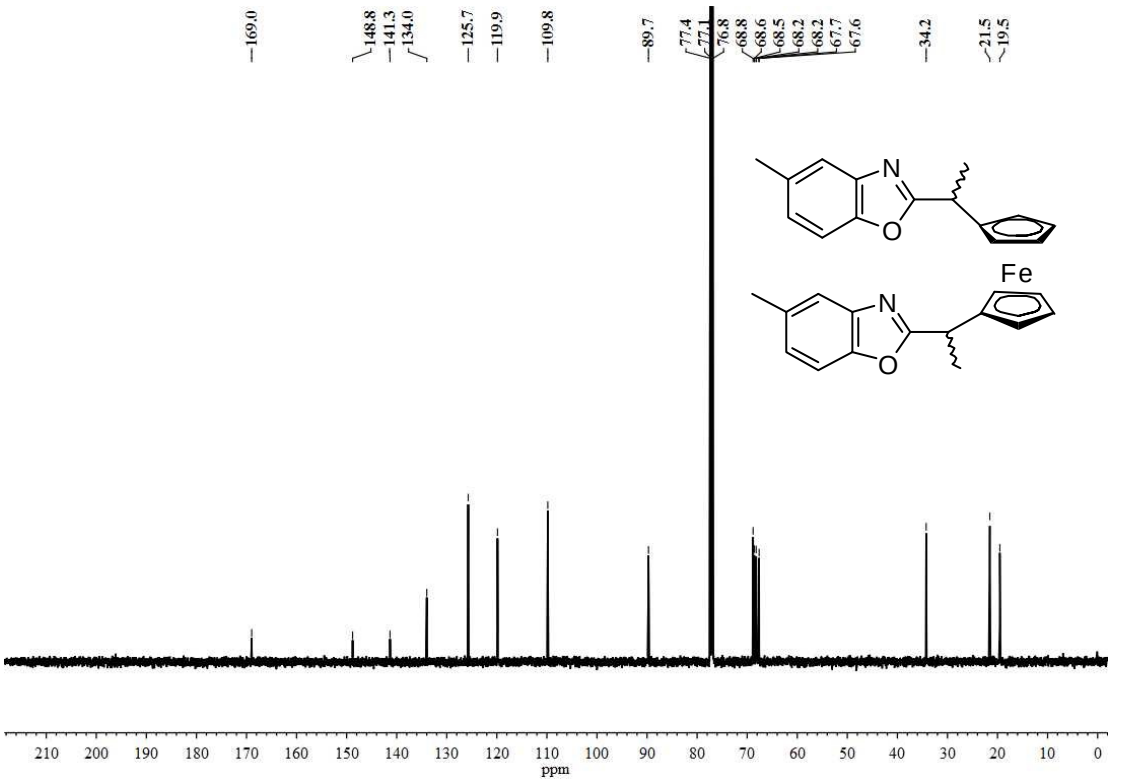

2.11
1.98
3.25
11.09
2.00
2.05
4.11
2.15
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm

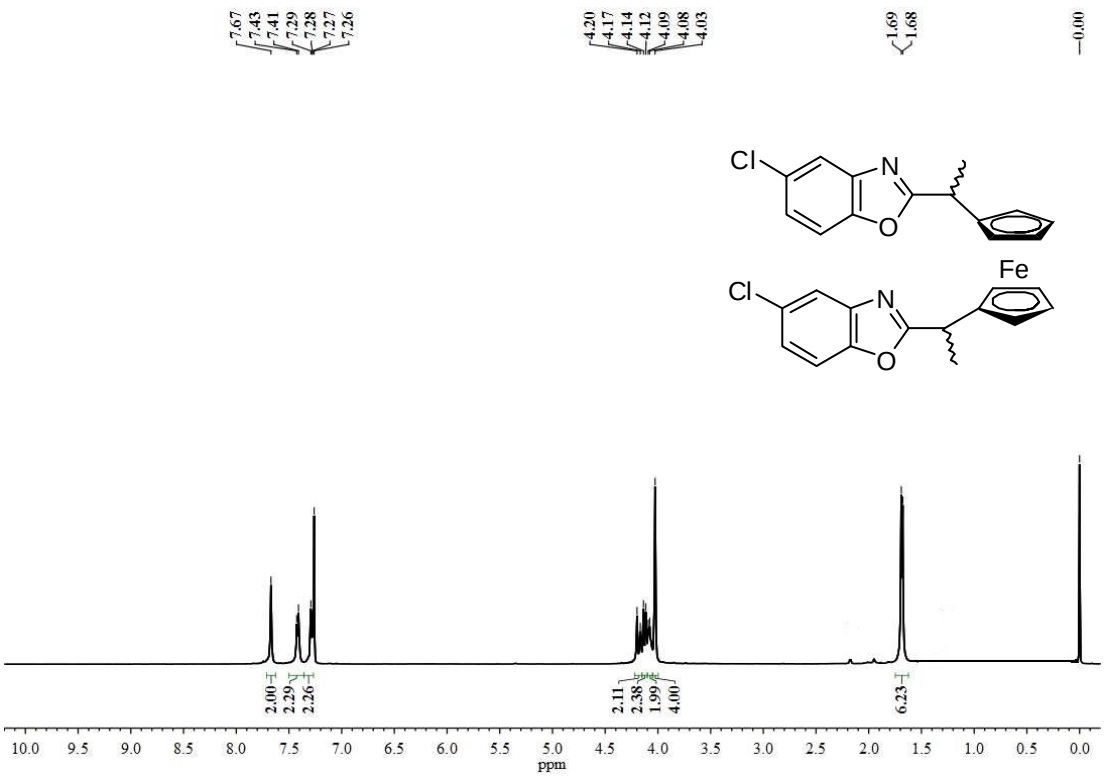

166.8
150.7
141.2
140.0
128.5
128.0
124.9
124.4
120.3
110.8
110.6
87.0
77.4
77.1
76.8
69.8
69.3
69.3
69.1
69.0
68.9
46.5
46.4

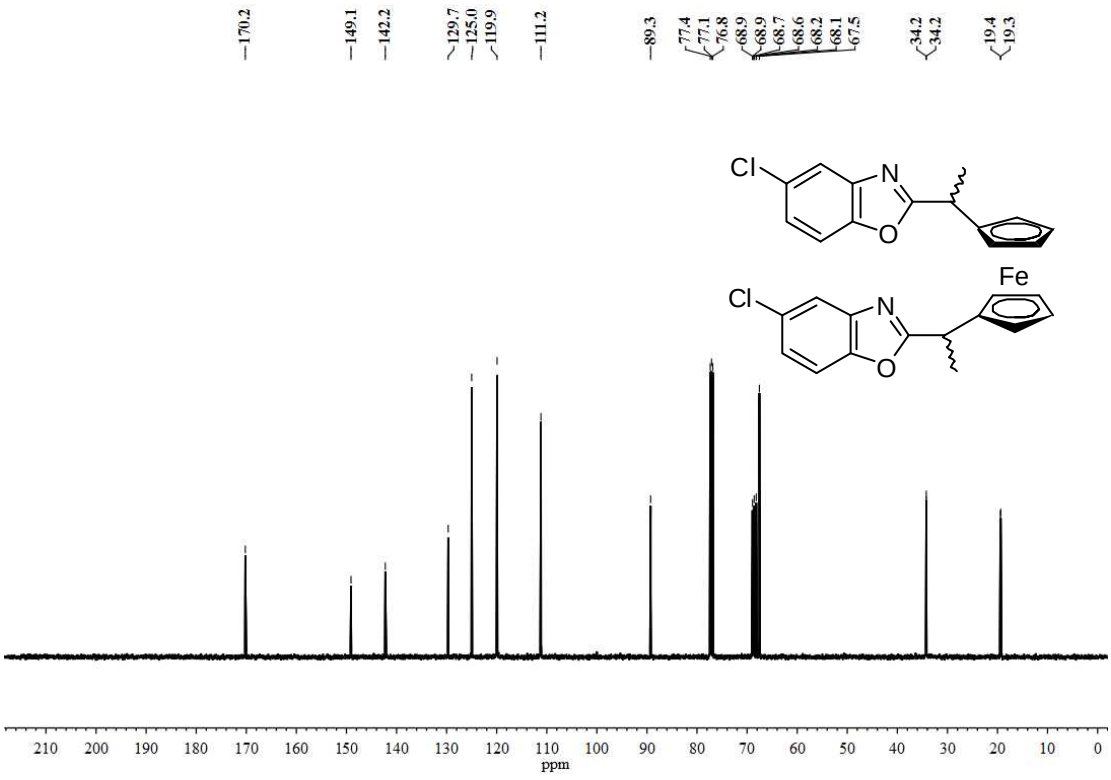

N
O
Ph
Fe
N
O
Ph

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


7.48
7.37
7.36
7.36
7.26
7.11
7.10
4.21
4.16
4.11
4.11
4.09
4.07
4.06
4.00
4.00
2.45
1.68
1.67
0.00
N
O
Fe
1.98
1.97
1.99
1.14
1.94
1.26
1.83
4.00
6.08
6.14
ppm


169.0
148.8
141.3
134.0
125.7
119.9
109.8
89.7
77.4
77.1
76.8
68.8
68.6
68.5
68.2
68.2
67.7
67.6
34.2
21.5
19.5
N
O
Fe
ppm


7.67
7.43
7.41
7.29
7.28
7.27
7.26
4.20
4.17
4.14
4.12
4.09
4.08
4.03
1.69
1.68
0.00
Cl
N
O
Fe
2.00
2.29
2.26
2.11
2.38
1.99
4.00
6.23
10.0 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
ppm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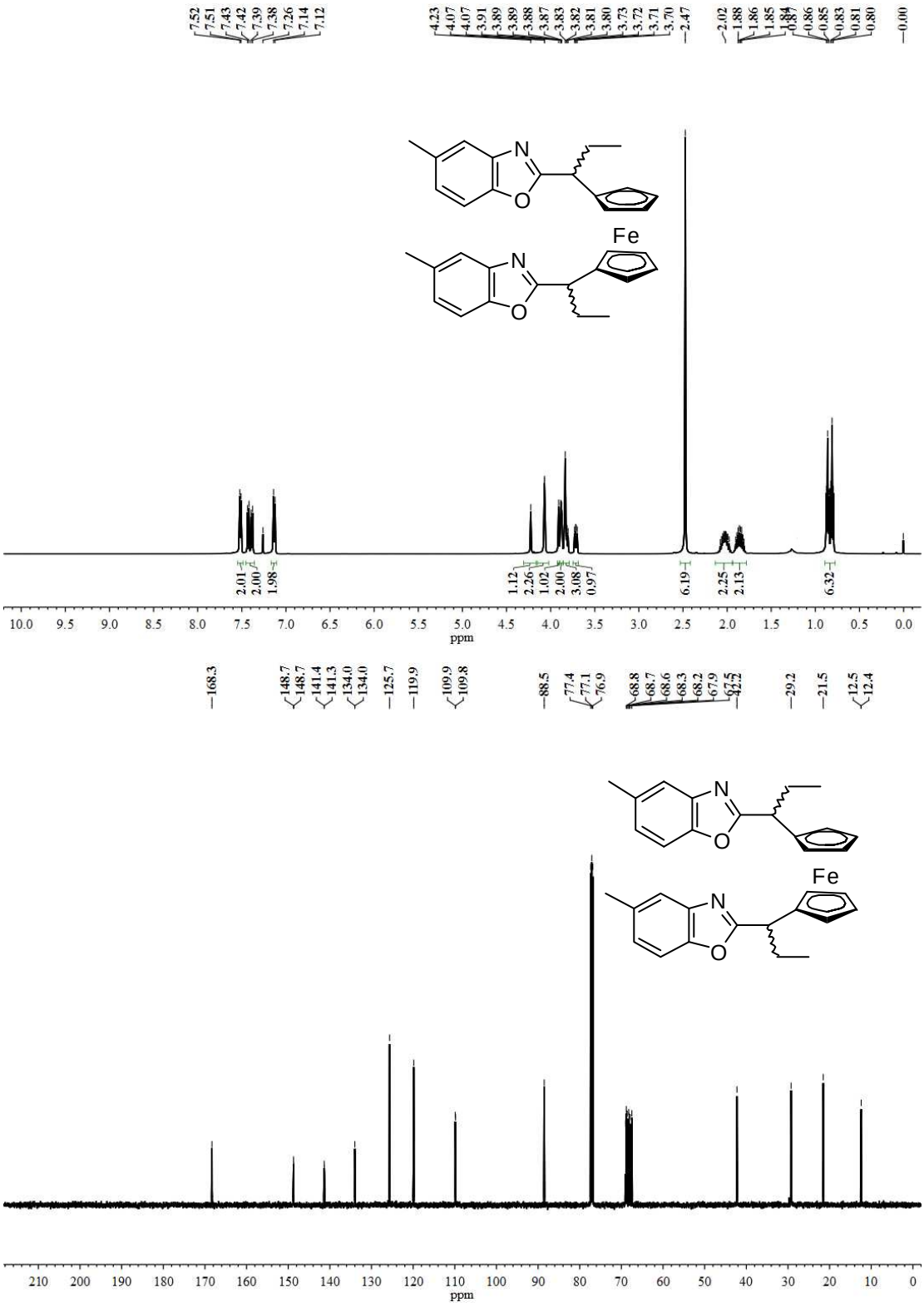
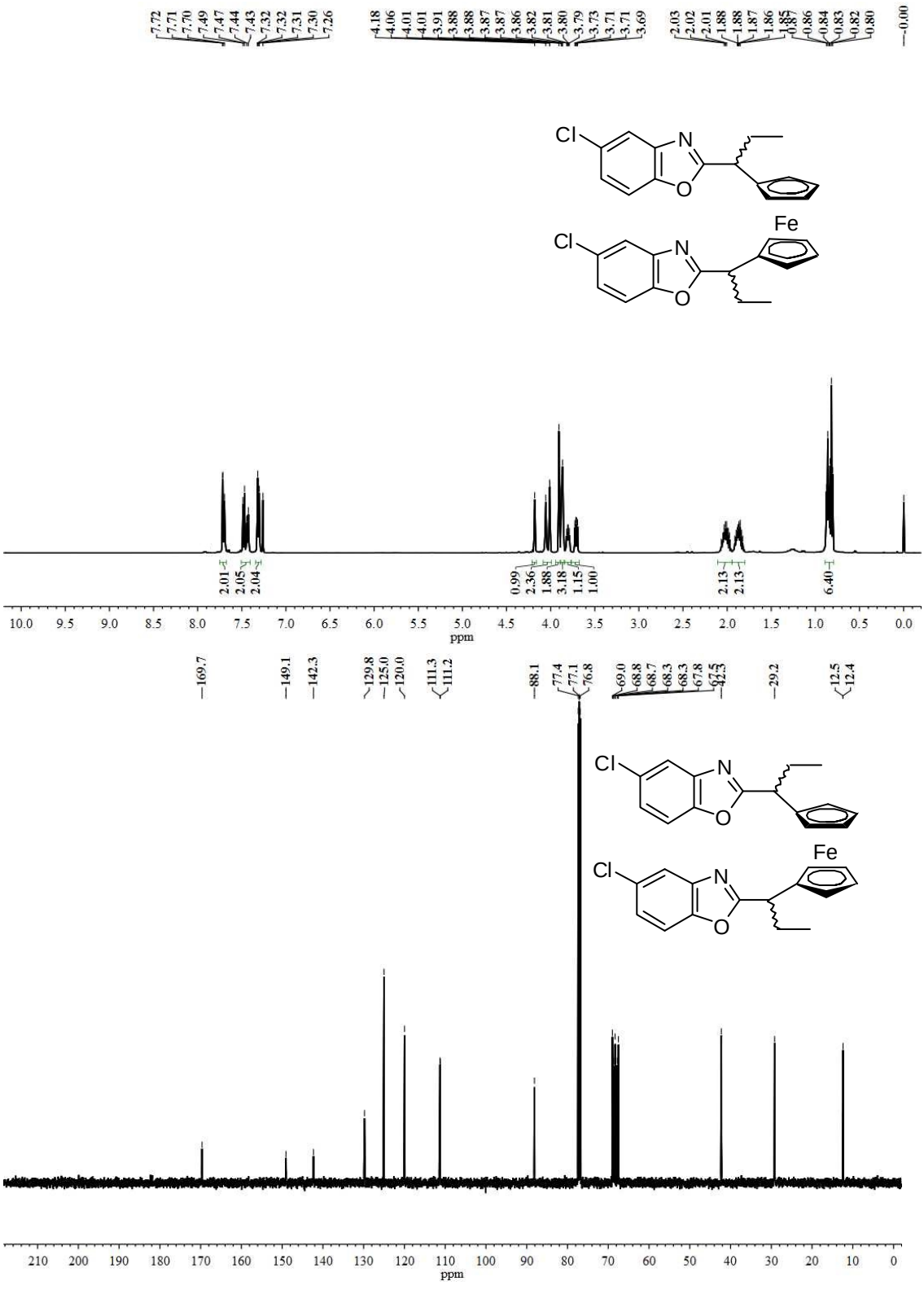
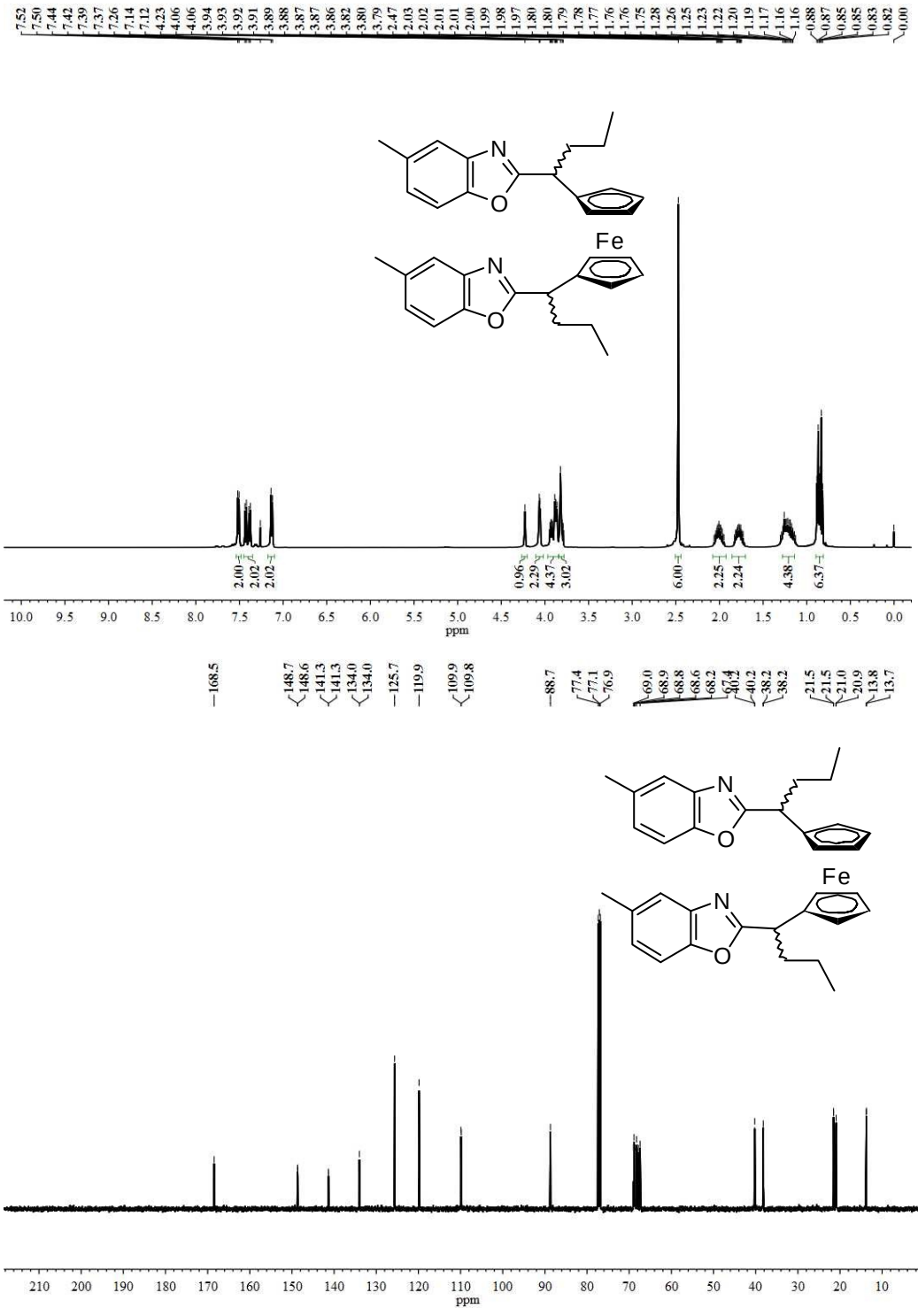
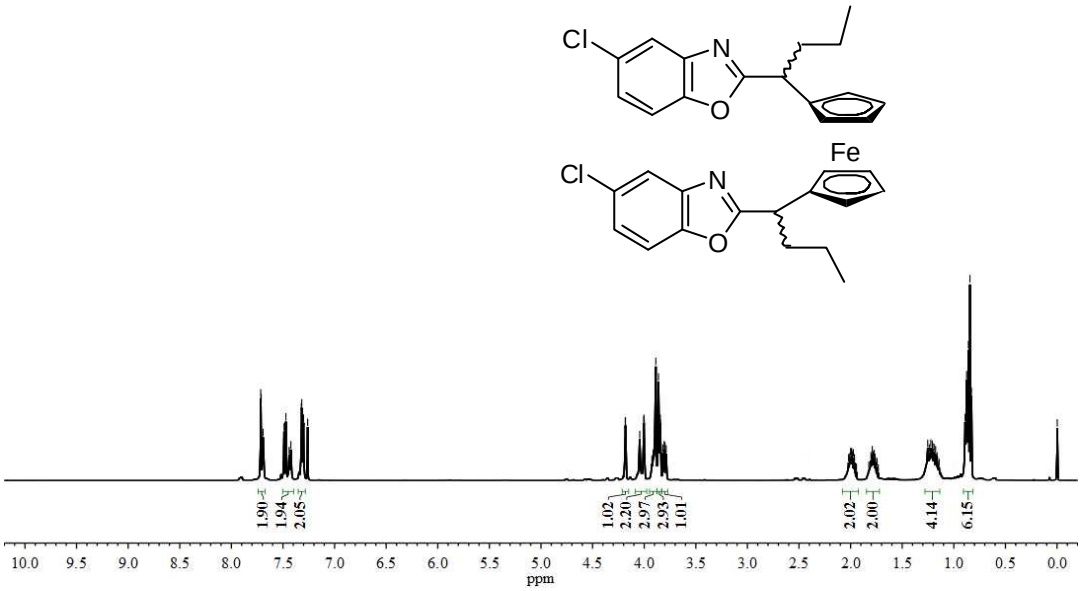
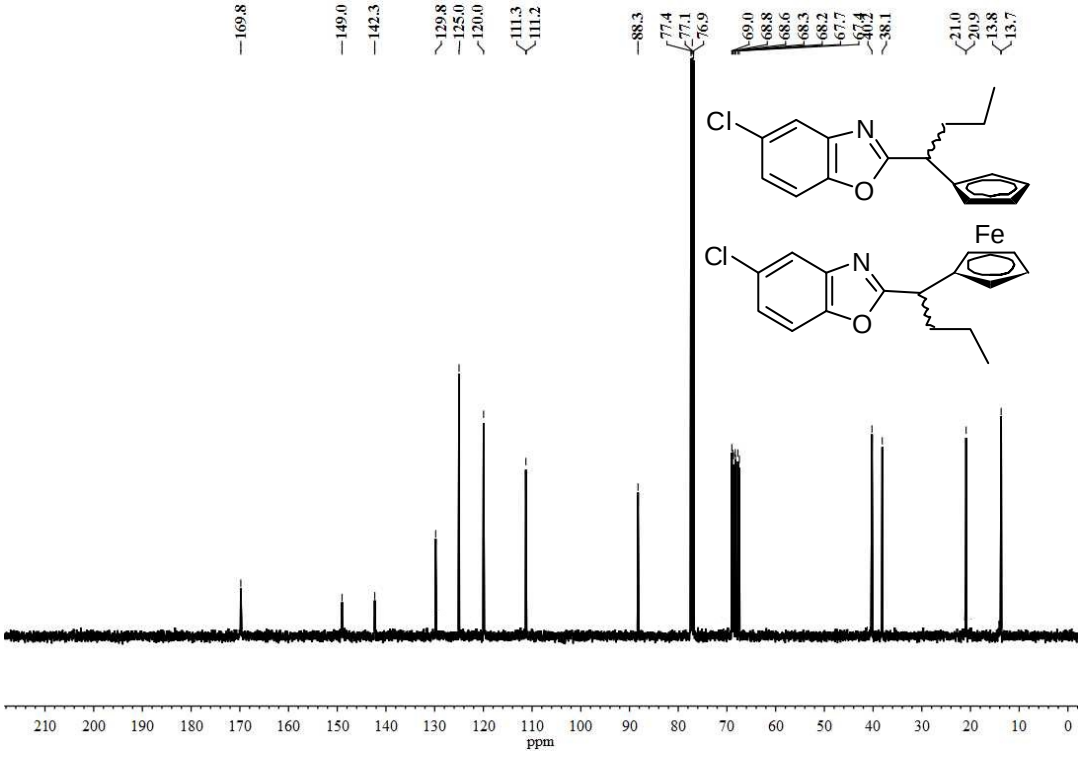
149.1
142.2
129.7
125.0
119.9
111.2
89.3
77.4
77.1
76.8
68.9
68.9
68.7
68.6
68.2
68.1
67.5
34.2
34.2
19.4
19.3
Cl
N
O
Fe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

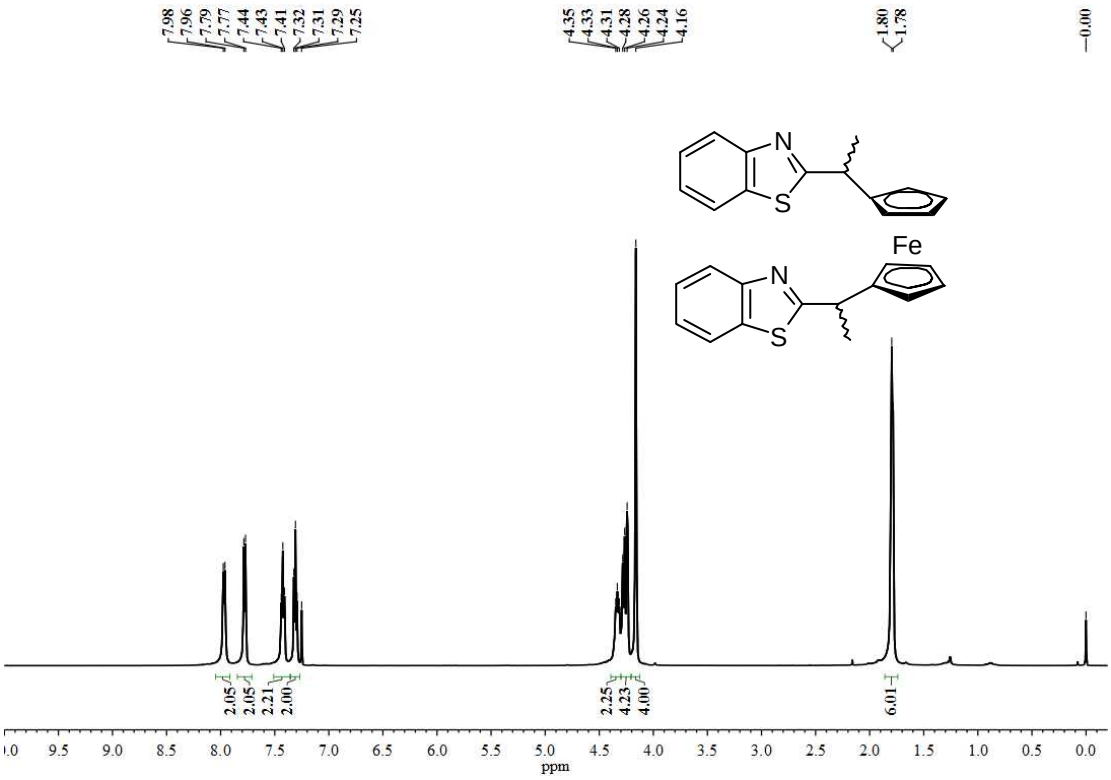

7.52
7.51
7.43
7.42
7.39
7.38
7.26
7.14
7.12
4.23
4.07
4.07
3.91
3.89
3.89
3.88
3.87
3.83
3.82
3.81
3.80
3.73
3.72
3.71
3.70
2.47
2.02
1.88
1.86
1.85
0.87
0.86
0.85
0.83
0.81
0.80
0.00
N
O
Fe
2.01
2.00
1.98
1.12
2.26
1.02
2.00
3.08
0.97
6.19
2.25
2.13
6.32
ppm
168.3
148.7
148.7
141.4
141.3
134.0
134.0
125.7
119.9
109.9
109.8
88.5
77.4
77.1
76.9
68.8
68.7
68.6
68.3
68.2
67.9
67.5
42.2
29.2
21.5
12.5
12.4
ppm


7.72 7.71 7.70 7.49 7.47 7.44 7.43 7.32 7.32 7.31 7.30 7.26
4.18 4.06 4.01 4.01 3.91 3.88 3.88 3.87 3.87 3.86 3.82 3.81 3.80 3.79 3.73 3.71 3.71 3.69
2.03 2.02 2.01 1.88 1.88 1.87 1.86 1.85 1.87 0.86 0.84 0.83 0.82 0.80
0.00
Cl
N
O
Fe
2.01 2.05 2.04 0.99 2.36 1.88 3.18 1.15 1.00 2.13 2.13 6.40
10.0 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
ppm
169.7 149.1 142.3 129.8 125.0 120.0 111.3 111.2 88.1 77.4 77.1 76.8 69.0 68.8 68.7 68.3 68.3 67.8 67.5 42.3 29.2 12.5 12.4
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm


7.52 7.50 7.44 7.42 7.39 7.37 7.26 7.14 7.12 4.23 4.06 4.06 3.94 3.93 3.92 3.91 3.89 3.88 3.87 3.87 3.86 3.82 3.80 3.79 2.47 2.03 2.02 2.01 2.01 2.00 1.99 1.98 1.97 1.80 1.80 1.79 1.78 1.77 1.76 1.76 1.75 1.28 1.26 1.25 1.23 1.22 1.20 1.19 1.17 1.16 1.16 0.88 0.87 0.85 0.85 0.83 0.82 0.00
N
O
Fe
2.00 2.02 2.02 0.96 2.29 4.37 3.02 6.00 2.25 2.24 4.38 6.37
10.0 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
ppm
168.5 148.7 148.6 141.3 141.3 134.0 134.0 125.7 119.9 109.9 109.8 88.7 77.4 77.1 76.9 69.0 68.9 68.8 68.6 68.2 67.4 40.2 40.2 38.2 38.2 21.5 21.5 21.0 20.9 13.8 13.7
N
O
Fe
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm


Cl
N
O
Fe
1.90
1.94
2.05
1.02
2.20
2.97
2.93
1.01
2.02
2.00
4.14
6.15
ppm


169.8
149.0
142.3
129.8
125.0
120.0
111.3
111.2
88.3
77.4
77.1
76.9
69.0
68.8
68.6
68.3
68.2
67.7
60.4
40.2
38.1
21.0
20.9
13.8
13.7
Cl
N
O
Fe
ppm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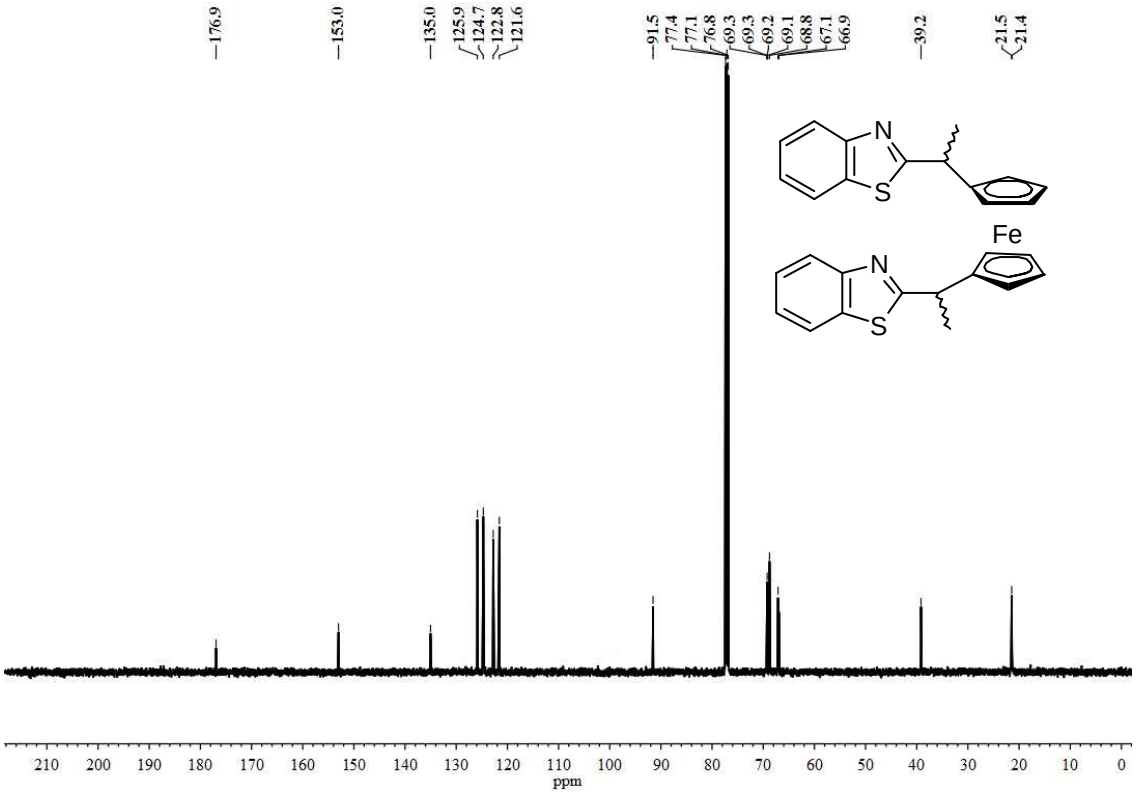
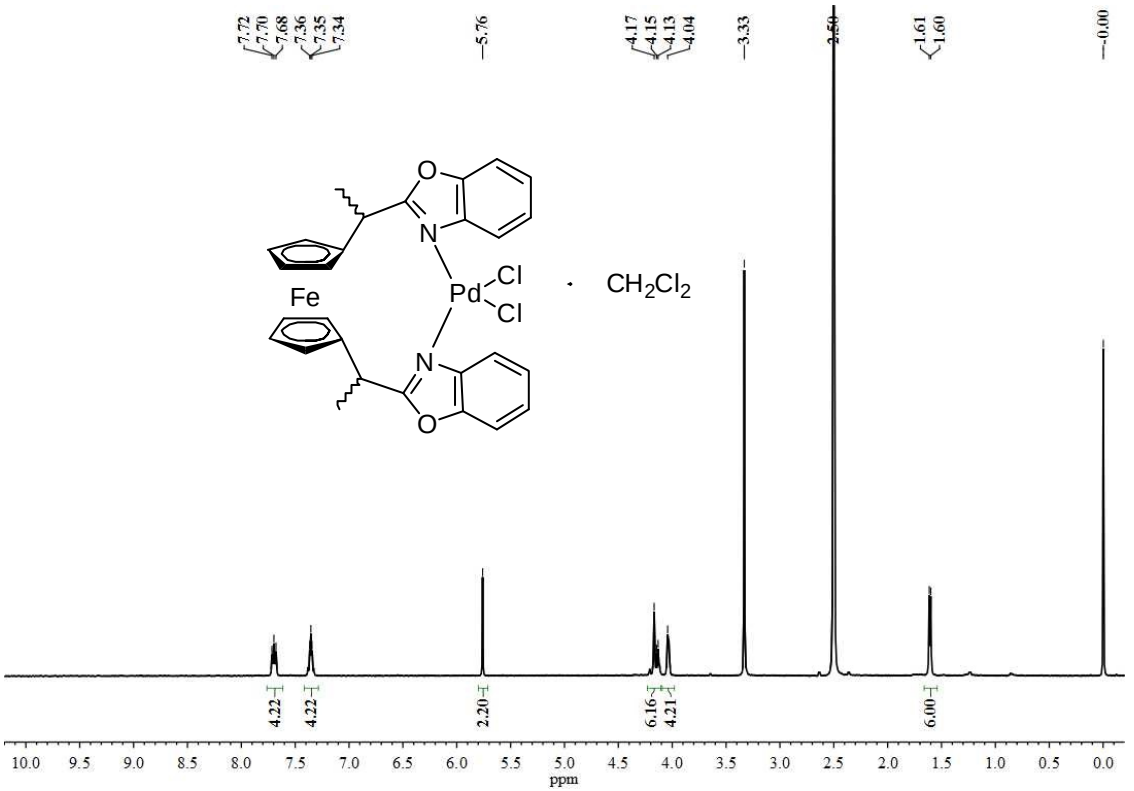
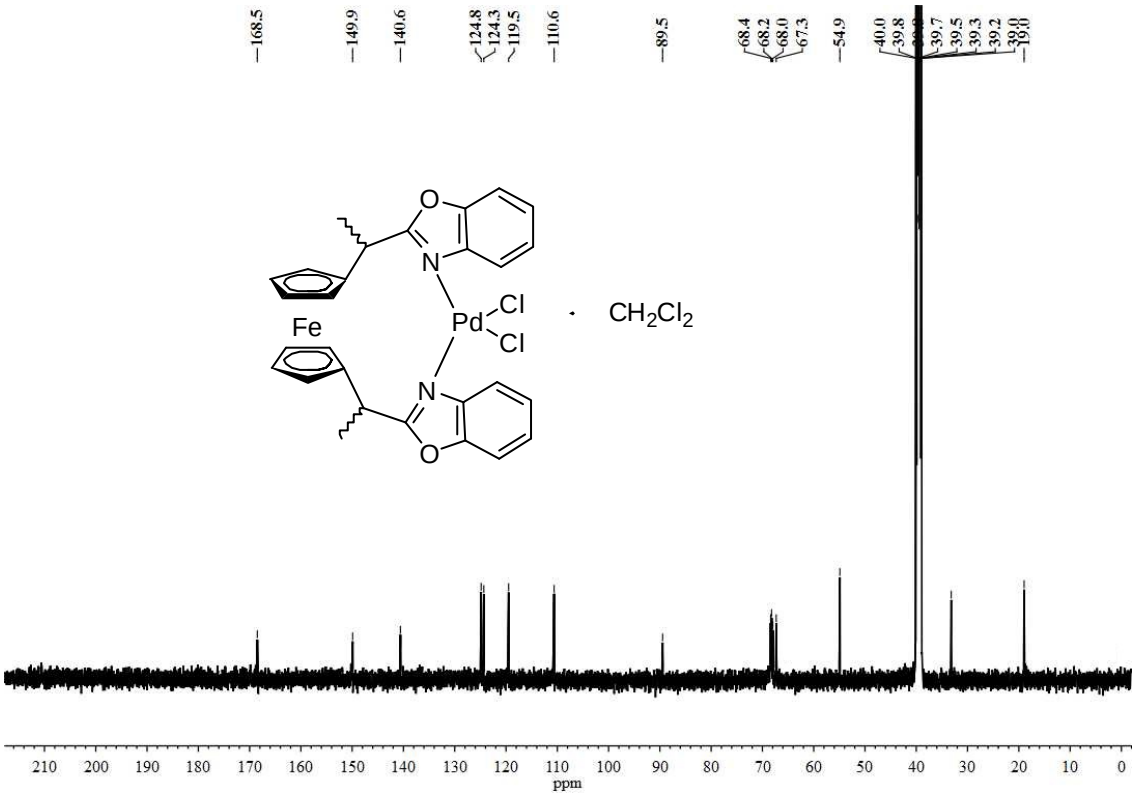
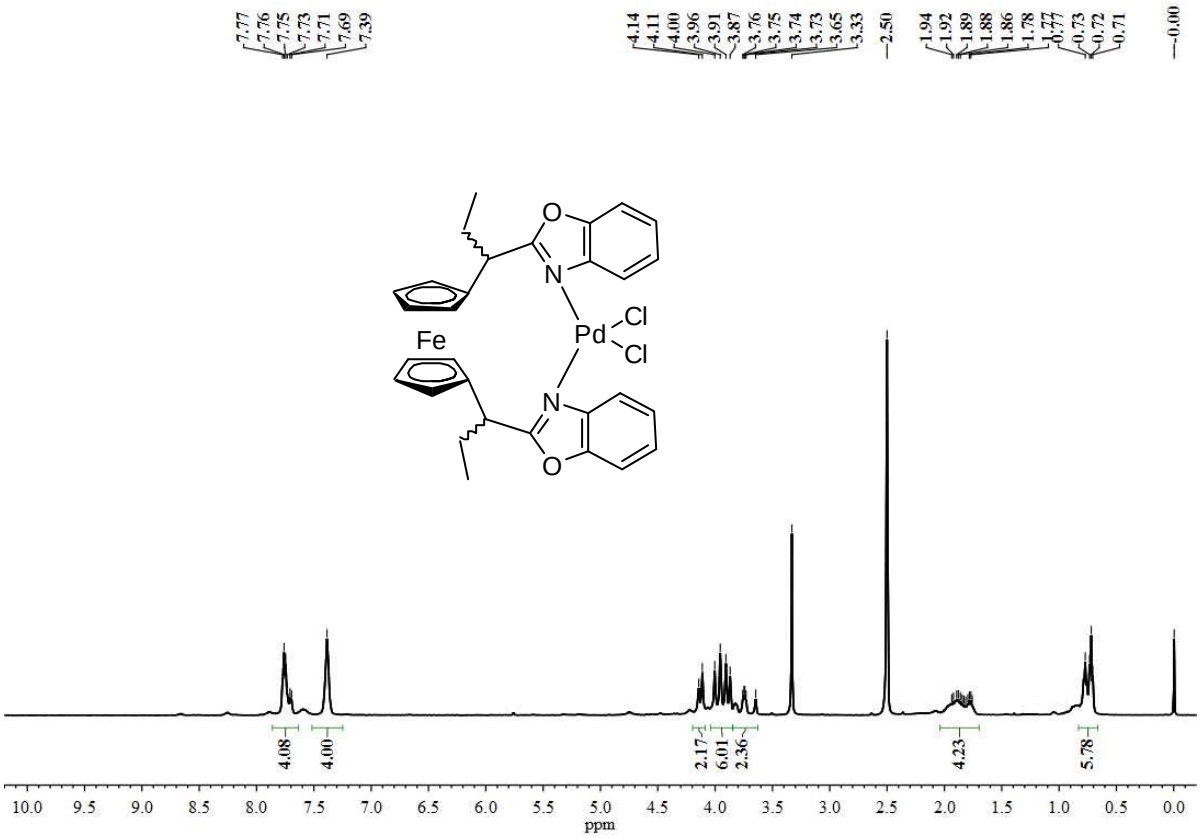
7.96
7.79
7.77
7.44
7.43
7.41
7.32
7.31
7.29
7.25
4.35
4.33
4.31
4.28
4.26
4.24
4.16
1.80
1.78
0.00
N
S
Fe
2.05
2.05
2.21
2.00
2.25
4.23
4.00
6.01
ppm


176.9
153.0
135.0
125.9
124.7
122.8
121.6
91.5
77.4
77.1
76.8
69.3
69.3
69.2
69.1
68.8
67.1
66.9
39.2
21.5
21.4
N
S
Fe
ppm


7.72
7.70
7.68
7.36
7.35
7.34
5.76
4.17
4.15
4.13
4.04
3.33
2.50
1.61
1.60
0.00
Fe
Pd
Cl
Cl
· CH2Cl2
4.22
4.22
2.20
6.16
4.21
6.00
ppm


168.5
149.9
140.6
124.8
124.3
119.5
110.6
89.5
68.4
68.2
68.0
67.3
54.9
40.0
39.8
39.7
39.5
39.3
39.2
39.0
19.0
Fe
Pd
Cl
Cl
· CH2Cl2
ppm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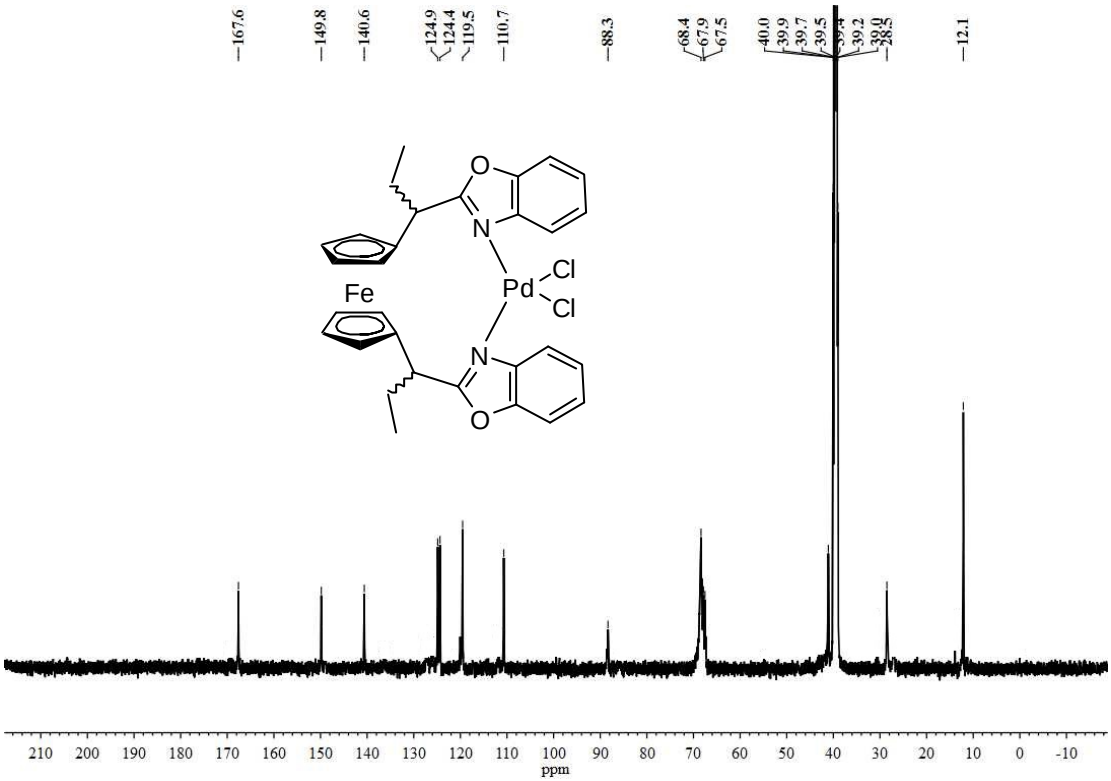
7.75
7.73
7.71
7.69
7.39
4.14
4.11
4.00
3.96
3.91
3.87
3.76
3.75
3.74
3.73
3.65
3.33
2.50
1.94
1.92
1.89
1.88
1.86
1.78
1.77
0.77
0.73
0.72
0.71
0.00
O
N
Pd
Cl
Cl
Fe
N
O
4.08
4.00
2.17
6.01
2.36
4.23
5.78
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
ppm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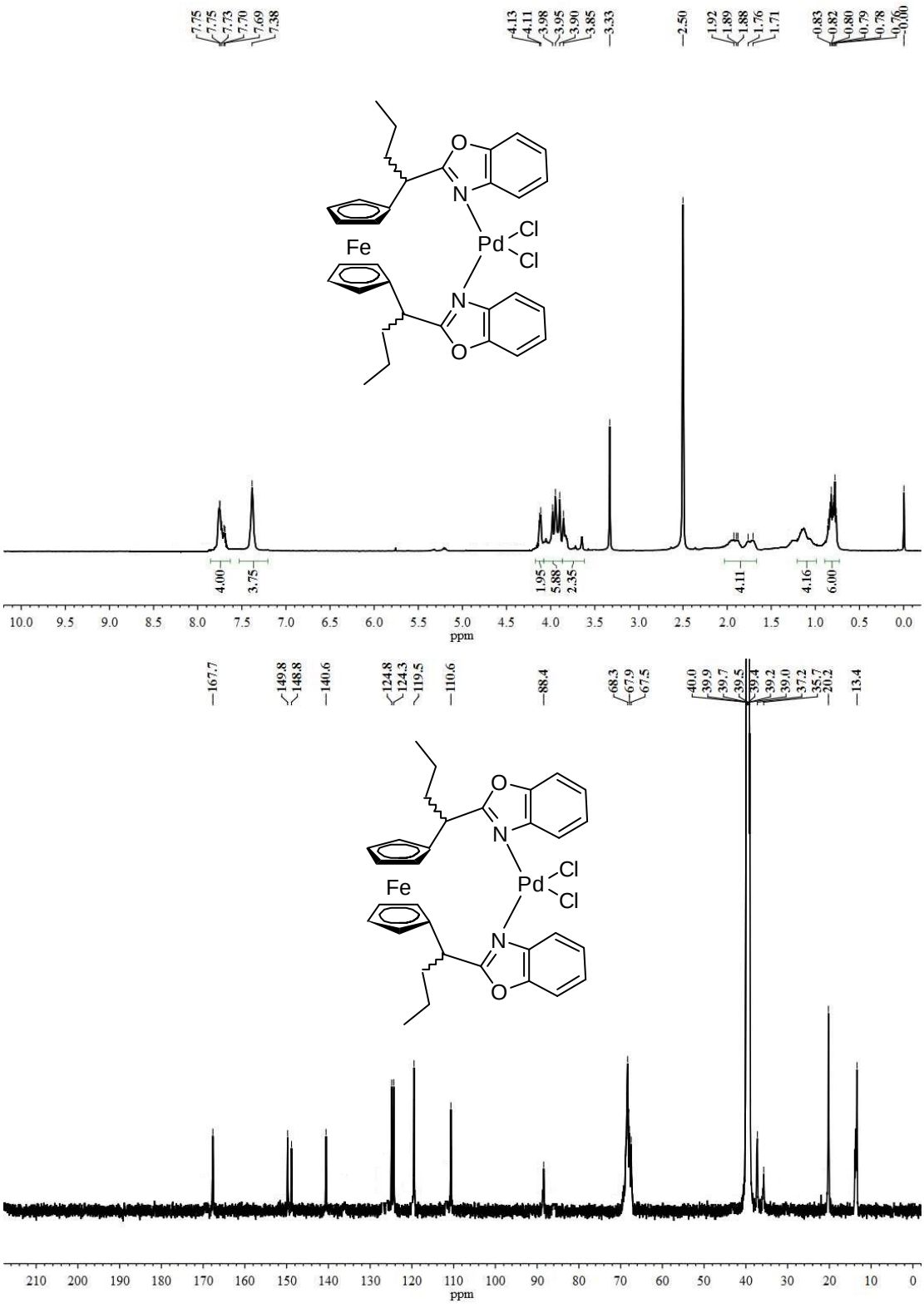
149.8
140.6
124.9
124.4
119.5
110.7
88.3
68.4
67.9
67.5
40.0
39.9
39.7
39.5
39.2
39.0
28.5
12.1
O
N
Pd
Cl
Cl
Fe
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
ppm


7.75
7.75
7.73
7.70
7.69
7.38
4.13
4.11
3.98
3.95
3.90
3.85
3.33
2.50
1.92
1.89
1.88
1.76
1.71
0.83
0.82
0.80
0.79
0.78
0.76
0.00
O
N
Pd
Cl
Cl
Fe
N
O
4.00
3.75
1.95
5.88
2.35
4.11
4.16
6.00
10.0 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
ppm
167.7
149.8
148.8
140.6
124.8
124.3
119.5
110.6
88.4
68.3
67.9
67.5
40.0
39.9
39.7
39.5
39.4
39.2
39.0
37.2
35.7
20.2
13.4
O
N
Pd
Cl
Cl
Fe
N
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm

5. Comparison of IR spectra of 5a and 6a