Supporting Information

Electrochemical-Induced Regioselective C-3 Thiomethylation of Imidazopyridines via a Three-Component Cross-Coupling Strategy

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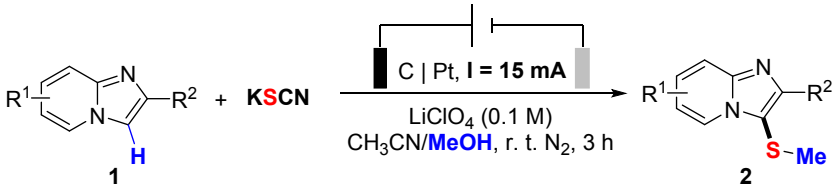
1. General information

All glassware was oven dried at 100 ^{o}C for hours and cooled down under vacuum. Imidazopyridines was prepared according to reported procedures.[1] All the reaction prepared using the solvent of CH_3CN (99.9%, Extra Dry with molecular sieves, Water≤50 ppm) was purchased from Innochem. The deoxygenation of dideionized water and CH_3CN is through Schlenk technology. Unless otherwise noted, materials were obtained from commercial suppliers and used without further purification. The instrument for electrolysis is dual display potentiostat (DJS-292B) (made in China). Thin layer chromatography (TLC) employed glass 0.25 mm silica gel plates. Flash chromatography columns were packed with 200-300 mesh silica gel in petroleum (bp. 60-90 ^{o}C). ^{1}H and ^{13}C NMR data were recorded with Bruker Advance III (500 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. All chemical shifts are reported relative to tetramethylsilane and d-solvent peaks (77.00 ppm, chloroform), respectively.

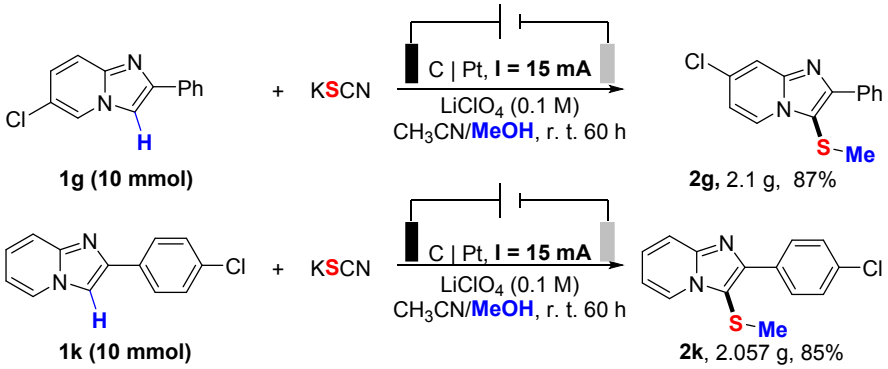
2. General procedure for electrochemical-induced regioselective C-3 thiomethylation of imidazopyridines.



1 2

In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, imidazopyridine **1** (0.25 mmol), potassium thiocyanate (0.5 mmol, 48.5 mg) and lithium perchlorate (1 mmol, 106 mg) were combined and added. The bottle was equipped with graphite rod anode and platinum plate (1 × 1 cm^2) cathode and was then charged with nitrogen. Under the protection by nitrogen, CH_3OH (0.5 mL) and CH_3CN (9.5 mL) were slowly injected into the reaction tube. The reaction mixture was stirred and electrolyzed at a constant current of 15 mA cm^{-2} under room temperature for 3 h. When the reaction was finished, the reaction mixture was washed with water and extracted with CH_2Cl_2 (10 mL x 3). The organic layers were combined, dried over Na_2SO_4, and concentrated. The pure product was obtained by flash column chromatography on silica gel (petroleum: ethyl ether = 3:1 - 5:1).

3. Procedure for gram scale synthesis



1g (10 mmol) **2g,** 2.1 g, 87%

1k (10 mmol) **2k**, 2.057 g, 85%

In an oven-dried undivided three-necked bottle (500 mL) equipped with a stir bar, 6-chloro-2-phenylimidazo[1,2-a]pyridine **1g** or 2-(4-chlorophenyl)imidazo[1,2-a]pyridine **1k** (10 mmol), potassium thiocyanate (2 equiv) and lithium perchlorate (0.1 M) were combined and added. The bottle was equipped with graphite rod anode and platinum plate (1 × 1 cm^2) cathode and was then charged with nitrogen. Under the protection by nitrogen, CH_3OH (20 mL) and CH_3CN (400 mL) were slowly injected into the reaction tube. The reaction mixture was stirred and electrolyzed at a constant current of 15 mA cm^{-2} under room temperature for 120 h. When the reaction was finished,

the reaction mixture was washed with water and extracted with CH_2Cl_2 (100 mL × 3). The organic layers were combined, dried over Na_2SO_4, and concentrated. The pure product **2e** (2.1 g) and **2h** (2.057 g) were obtained in 87% and 85% yield by flash column chromatography on silica gel, respectively (petroleum: ethyl ether = 5:1).

4. Preliminary mechanistic studies.

(1) Deuteration experiments

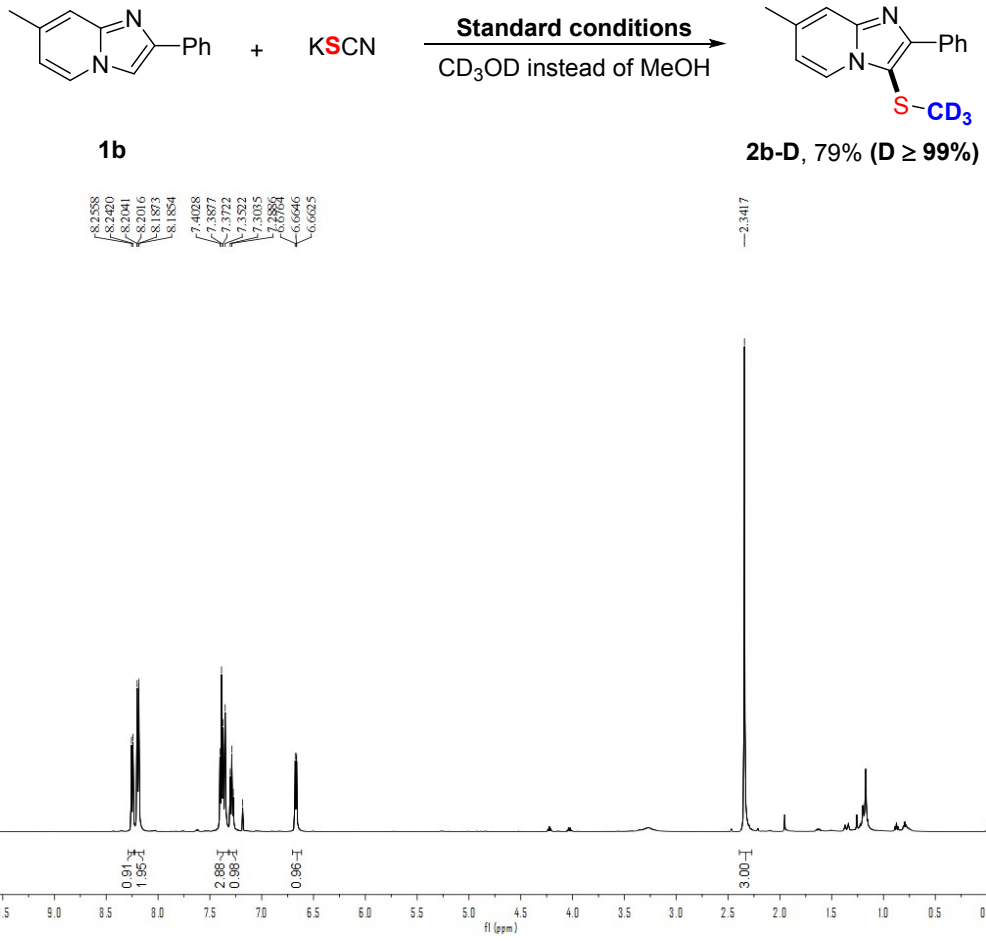
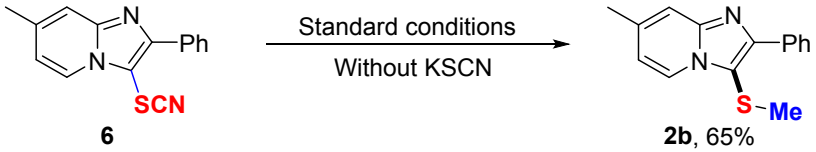


Figure S1. [1]HNMR spectrum of **2b-D**.

(2) Intermediate verification experiments

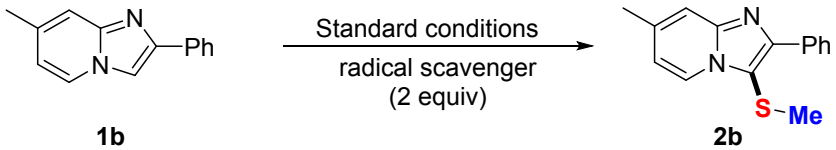


6 **2b**, 65%

In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, **6** (0.25 mmol), and lithium perchlorate (1 mmol) were combined and added. The bottle was equipped with graphite rod anode and platinum plate (1 × 1 cm^2) cathode and was then charged with nitrogen. Under the protection by nitrogen, CH_3CN (10 mL) was slowly injected into the reaction tube. The

reaction mixture was stirred and electrolyzed at a constant current of 15 mA cm^{-2} under room temperature for 3 h. When the reaction was finished, the reaction mixture was washed with water and extracted with CH_2Cl_2 (10 mL x 3). The organic layers were combined, dried over Na_2SO_4, and concentrated. The desired product **2b** was obtained in 65% yield by flash column chromatography on silica gel (petroleum: ethyl ether = 3:1).

(3) The reaction of **1b** with TEMPO or BHT under the standard conditions.



1b **2b**

TEMPO: n. d.
BHT: trace

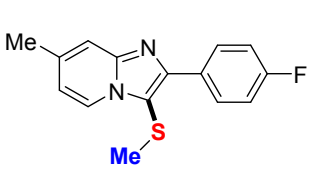
In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, ketene dithioacetals **1b** (0.25 mmol), KSCN (0.5 mmol), $LiClO_4$ (1 mmol), TEMPO or BHT (0.5 mmol) and CH_3CN / CH_3OH (9.5/0.5 = 10 mL) were combined and sealed. The bottle was equipped with graphite rod as anode and platinum electrodes (1 × 1 cm^2) as cathode, and was then charged with nitrogen. The reaction mixture was stirred and electrolyzed at a constant current of 15 mA under room temperature for 3 h. When the reaction was finished, the solution was concentrated in vacuum and the yield of **2b** was sharply decreased.

5. References

1. (a) Saldabol, N. O. and Giller, S. A., *Chem. Heterocycl. Com.,* 1976, **12**, 1155-1162; (b) Cao, H., Zhan, H., Lin, Y., Lin, X., Du, Z. and Jiang, H., *Org. Lett.*, 2012, **14**, 1688-1691; (c) Bagdi, A. K. and Hajra, A., *The Chem. Rec.*, 2016, **16**, 1868-1885; (d) Samanta, S., Mondal, S., Santra, S., Kibriya, G. and Hajra, A., *J. Org. Chem.,* 2016, **81**, 10088-10093; (e) Mondal, S., Samanta, S., Singsardar, M. and Hajra, A., *Org. Lett.*, 2017, **19**, 3751-3754.

6. Crystallographic data of 2n.

ORTEP drawing of $C_{15}H_{13}FN_2S$ with 30% probability ellipsoids, showing the atomic numbering scheme.



(**2n**)

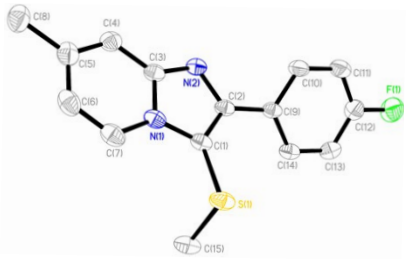


Table S1. Crystal data and structure refinement for 191116e.

Identification code	191116e
Empirical formula	C15 H13 F N2 S
Formula weight	272.33
Temperature	298(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 8.6772(8) A alpha = 110.220(4) deg. b = 9.0145(9) A beta = 107.146(3) deg. c = 9.9691(9) A gamma = 97.596(2) deg.
Volume	674.97(11) A^3
Z, Calculated density	2, 1.340 Mg/m^3
Absorption coefficient	0.238 mm^-1
F(000)	284
Crystal size	0.45 x 0.37 x 0.30 mm
Theta range for data collection	2.49 to 25.01 deg.
Limiting indices	-10<=h<=10, -10<=k<=10, -11<=l<=8
Reflections collected / unique	3379 / 2326 [R(int) = 0.0182]
Completeness to theta = 25.01	97.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9320 and 0.9004
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2326 / 0 / 174
Goodness-of-fit on F^2	1.060
Final R indices [I>2sigma(I)]	R1 = 0.0427, wR2 = 0.1159
R indices (all data)	R1 = 0.0578, wR2 = 0.1272
Largest diff. peak and hole	0.772 and -0.160 e.A^-3

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 191116e. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
F(1)	10660(2)	2474(2)	6872(2)	89(1)
N(1)	2286(3)	2937(2)	1672(2)	44(1)
N(2)	3075(2)	1823(2)	3361(2)	44(1)
S(1)	5186(1)	4446(1)	1572(1)	54(1)
C(1)	4021(3)	3405(3)	2276(2)	43(1)
C(2)	4464(3)	2686(2)	3306(2)	40(1)
C(3)	1770(3)	1989(3)	2375(3)	44(1)
C(4)	48(3)	1371(3)	1975(3)	52(1)
C(5)	-1090(3)	1649(3)	872(3)	57(1)
C(6)	-482(4)	2593(3)	164(3)	62(1)
C(7)	1157(4)	3224(3)	556(3)	56(1)
C(8)	-2931(4)	963(4)	406(4)	73(1)

C(9)	6123(3)	2671(3)	4217(2)	40(1)
C(10)	6333(3)	1247(3)	4417(3)	51(1)
C(11)	7847(4)	1168(3)	5297(3)	62(1)
C(12)	9159(3)	2531(4)	5988(3)	58(1)
C(13)	9020(3)	3967(3)	5833(3)	55(1)
C(14)	7499(3)	4028(3)	4943(3)	48(1)
C(15)	5174(4)	6547(3)	2524(3)	60(1)

Table S3. Bond lengths [A] and angles [deg] for 191116e.

F(1)-C(12)	1.359(3)
N(1)-C(7)	1.374(3)
N(1)-C(3)	1.386(3)
N(1)-C(1)	1.389(3)
N(2)-C(3)	1.327(3)
N(2)-C(2)	1.373(3)
S(1)-C(1)	1.737(2)
S(1)-C(15)	1.805(2)
C(1)-C(2)	1.384(3)
C(2)-C(9)	1.463(3)
C(3)-C(4)	1.406(4)
C(4)-C(5)	1.364(4)
C(4)-H(4)	0.9300
C(5)-C(6)	1.421(4)
C(5)-C(8)	1.500(4)
C(6)-C(7)	1.343(4)
C(6)-H(6)	0.9300
C(7)-H(7)	0.9300
C(8)-H(8A)	0.9600
C(8)-H(8B)	0.9600
C(8)-H(8C)	0.9600
C(9)-C(14)	1.392(3)
C(9)-C(10)	1.392(3)
C(10)-C(11)	1.374(4)
C(10)-H(10)	0.9300
C(11)-C(12)	1.364(4)
C(11)-H(11)	0.9300
C(12)-C(13)	1.373(4)
C(13)-C(14)	1.376(4)
C(13)-H(13)	0.9300
C(14)-H(14)	0.9300
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600

C(7)-N(1)-C(3)	121.4(2)
C(7)-N(1)-C(1)	131.2(2)
C(3)-N(1)-C(1)	107.34(19)
C(3)-N(2)-C(2)	106.03(18)
C(1)-S(1)-C(15)	103.19(11)
C(2)-C(1)-N(1)	104.85(19)
C(2)-C(1)-S(1)	132.3(2)
N(1)-C(1)-S(1)	122.34(17)
N(2)-C(2)-C(1)	111.0(2)
N(2)-C(2)-C(9)	119.37(18)
C(1)-C(2)-C(9)	129.5(2)
N(2)-C(3)-N(1)	110.8(2)
N(2)-C(3)-C(4)	130.5(2)
N(1)-C(3)-C(4)	118.7(2)
C(5)-C(4)-C(3)	120.5(2)
C(5)-C(4)-H(4)	119.7
C(3)-C(4)-H(4)	119.7
C(4)-C(5)-C(6)	118.1(3)
C(4)-C(5)-C(8)	121.1(3)
C(6)-C(5)-C(8)	120.8(3)
C(7)-C(6)-C(5)	122.3(2)
C(7)-C(6)-H(6)	118.9
C(5)-C(6)-H(6)	118.9
C(6)-C(7)-N(1)	118.9(2)
C(6)-C(7)-H(7)	120.5
N(1)-C(7)-H(7)	120.5
C(5)-C(8)-H(8A)	109.5
C(5)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(5)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(14)-C(9)-C(10)	117.9(2)
C(14)-C(9)-C(2)	123.4(2)
C(10)-C(9)-C(2)	118.6(2)
C(11)-C(10)-C(9)	121.5(2)
C(11)-C(10)-H(10)	119.2
C(9)-C(10)-H(10)	119.2
C(12)-C(11)-C(10)	118.5(2)
C(12)-C(11)-H(11)	120.8
C(10)-C(11)-H(11)	120.8
F(1)-C(12)-C(11)	119.1(2)
F(1)-C(12)-C(13)	118.5(3)
C(11)-C(12)-C(13)	122.4(3)

C(12)-C(13)-C(14)	118.5(2)
C(12)-C(13)-H(13)	120.7
C(14)-C(13)-H(13)	120.7
C(13)-C(14)-C(9)	121.1(2)
C(13)-C(14)-H(14)	119.4
C(9)-C(14)-H(14)	119.4
S(1)-C(15)-H(15A)	109.5
S(1)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
S(1)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5

Table S4. Anisotropic displacement parameters (A^2 x 10^3) for 191116e. The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
F(1)	60(1)	111(2)	98(1)	61(1)	12(1)	21(1)
N(1)	59(1)	35(1)	35(1)	13(1)	16(1)	13(1)
N(2)	52(1)	39(1)	44(1)	19(1)	18(1)	8(1)
S(1)	84(1)	43(1)	50(1)	24(1)	38(1)	18(1)
C(1)	60(2)	33(1)	37(1)	14(1)	20(1)	12(1)
C(2)	54(1)	31(1)	37(1)	12(1)	20(1)	9(1)
C(3)	56(2)	34(1)	39(1)	13(1)	17(1)	10(1)
C(4)	58(2)	40(1)	52(1)	11(1)	22(1)	11(1)
C(5)	58(2)	43(1)	50(1)	2(1)	14(1)	17(1)
C(6)	68(2)	56(2)	48(2)	16(1)	6(1)	24(1)
C(7)	75(2)	49(1)	42(1)	20(1)	14(1)	20(1)
C(8)	57(2)	64(2)	74(2)	6(2)	15(2)	20(1)
C(9)	55(1)	35(1)	36(1)	16(1)	22(1)	10(1)
C(10)	56(2)	40(1)	58(2)	25(1)	17(1)	8(1)
C(11)	67(2)	54(2)	74(2)	40(2)	23(2)	18(1)
C(12)	54(2)	74(2)	55(2)	35(1)	18(1)	18(1)
C(13)	56(2)	52(2)	50(1)	18(1)	19(1)	0(1)
C(14)	62(2)	35(1)	45(1)	16(1)	22(1)	8(1)
C(15)	92(2)	41(1)	53(2)	23(1)	31(2)	13(1)

Table S5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 191116e.

	x	y	z	U(eq)

H(4)	-318	768	2465	62
H(6)	-1245	2779	-598	74
H(7)	1522	3846	80	68
H(8A)	-3111	340	981	110
H(8B)	-3400	266	-671	110
H(8C)	-3459	1844	609	110
H(10)	5428	328	3945	61
H(11)	7974	209	5419	74
H(13)	9933	4880	6319	66
H(14)	7389	4992	4824	57
H(15A)	4045	6640	2243	90
H(15B)	5814	7232	2218	90
H(15C)	5657	6887	3616	90

Table S6. Torsion angles [deg] for 191116e.

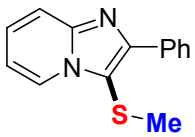
C(7)-N(1)-C(1)-C(2)	-176.9(2)
C(3)-N(1)-C(1)-C(2)	0.9(2)
C(7)-N(1)-C(1)-S(1)	-4.2(3)
C(3)-N(1)-C(1)-S(1)	173.61(15)
C(15)-S(1)-C(1)-C(2)	-107.3(2)
C(15)-S(1)-C(1)-N(1)	82.2(2)
C(3)-N(2)-C(2)-C(1)	0.2(2)
C(3)-N(2)-C(2)-C(9)	-176.84(19)
N(1)-C(1)-C(2)-N(2)	-0.7(2)
S(1)-C(1)-C(2)-N(2)	-172.37(17)
N(1)-C(1)-C(2)-C(9)	176.0(2)
S(1)-C(1)-C(2)-C(9)	4.3(4)
C(2)-N(2)-C(3)-N(1)	0.4(2)
C(2)-N(2)-C(3)-C(4)	-179.7(2)
C(7)-N(1)-C(3)-N(2)	177.27(19)
C(1)-N(1)-C(3)-N(2)	-0.8(2)
C(7)-N(1)-C(3)-C(4)	-2.7(3)
C(1)-N(1)-C(3)-C(4)	179.24(19)
N(2)-C(3)-C(4)-C(5)	-177.5(2)
N(1)-C(3)-C(4)-C(5)	2.5(3)
C(3)-C(4)-C(5)-C(6)	-0.8(3)
C(3)-C(4)-C(5)-C(8)	178.5(2)
C(4)-C(5)-C(6)-C(7)	-0.7(4)
C(8)-C(5)-C(6)-C(7)	179.9(2)
C(5)-C(6)-C(7)-N(1)	0.6(4)
C(3)-N(1)-C(7)-C(6)	1.2(3)
C(1)-N(1)-C(7)-C(6)	178.8(2)
N(2)-C(2)-C(9)-C(14)	-142.9(2)

C(1)-C(2)-C(9)-C(14)	40.7(3)
N(2)-C(2)-C(9)-C(10)	34.8(3)
C(1)-C(2)-C(9)-C(10)	-141.6(2)
C(14)-C(9)-C(10)-C(11)	-0.2(4)
C(2)-C(9)-C(10)-C(11)	-178.0(2)
C(9)-C(10)-C(11)-C(12)	0.3(4)
C(10)-C(11)-C(12)-F(1)	179.5(2)
C(10)-C(11)-C(12)-C(13)	-0.1(4)
F(1)-C(12)-C(13)-C(14)	-179.8(2)
C(11)-C(12)-C(13)-C(14)	-0.2(4)
C(12)-C(13)-C(14)-C(9)	0.3(4)
C(10)-C(9)-C(14)-C(13)	-0.1(3)
C(2)-C(9)-C(14)-C(13)	177.6(2)

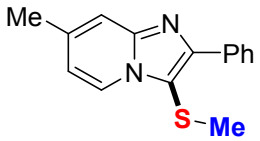
Table S7. Hydrogen bonds for 191116e [A and deg.]

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)

7. Detail descriptions for products.

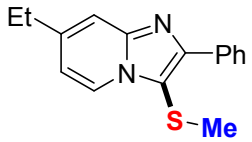


3-(methylthio)-2-phenylimidazo[1,2-a]pyridine (2a): yellow solid was obtained with 87% isolated yield (52.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40 (d, *J* = 6.8 Hz, 1H), 8.22 (d, *J* = 7.2 Hz, 2H), 7.61 (d, *J* = 9.0 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 7.22 (dd, *J* = 8.4, 7.3 Hz, 1H), 6.86 (t, *J* = 6.7 Hz, 1H), 2.18 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.60, 146.19, 133.61, 128.43, 128.37, 128.29, 126.15, 124.29, 117.56, 112.88, 111.51, 77.34, 77.08, 76.83, 18.18. HRMS (ESI) calcd for $C_{14}H_{13}N_2S$ $[M + H]^+$: 241.0794; found: 241.0793.

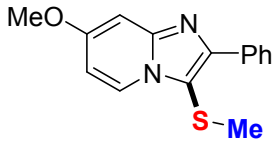


7-methyl-3-(methylthio)-2-phenylimidazo[1,2-a]pyridine (2b): yellow solid was obtained with 98% isolated yield (62.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.26 (d, *J* = 6.9 Hz, 1H), 8.19 (dd, *J* = 8.2, 1.1 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.35 (s, 1H), 7.32 – 7.26 (m, 1H), 6.68 (dd, *J* = 7.0, 1.5 Hz, 1H), 2.35 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.53, 146.66, 137.21, 133.88, 128.36, 128.21, 128.17, 123.45, 116.08, 115.40, 110.68, 21.37, 18.32. HRMS (ESI) calcd for $C_{15}H_{15}N_2S$ [M+

H]$^+$: 255.0590; found: 255.0948.

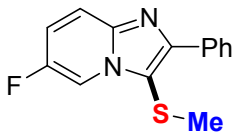


7-ethyl-3-(methylthio)-2-phenylimidazo[1,2-a]pyridine (2c): yellow solid was obtained with 99% isolated yield (66.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.37 (d, *J* = 7.0 Hz, 1H), 8.32 – 8.26 (m, 2H), 7.47 (dd, *J* = 14.5, 6.7 Hz, 3H), 7.38 (t, *J* = 7.4 Hz, 1H), 6.80 (dd, *J* = 7.0, 1.5 Hz, 1H), 2.74 (q, *J* = 7.6 Hz, 2H), 2.25 (s, 3H), 1.31 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.73, 146.85, 143.19, 134.04, 128.34, 128.18, 128.12, 123.60, 114.73, 114.35, 110.62, 28.43, 18.34, 14.45. HRMS (ESI) calcd for $C_{16}H_{17}N_2S$ [M+ H]$^+$: 269.1107; found: 269.1103.

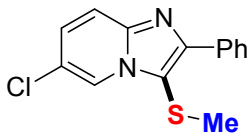


7-methoxy-3-(methylthio)-2-phenylimidazo[1,2-a]pyridine (2d): yellow solid was obtained with 81% isolated yield (54.7 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.28 (t, *J* = 6.7 Hz, 3H), 7.47 (t, *J* = 7.2 Hz, 2H), 7.37 (t, *J* = 7.4 Hz, 1H), 6.95 (s, 1H), 6.64 (dd, *J* = 7.4, 2.3 Hz, 1H), 3.89 (s, 3H), 2.25 (d, *J* = 1.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.03, 148.54, 147.76, 133.98, 128.34, 128.07, 128.00, 110.01, 107.53, 95.08, 55.66, 18.60. HRMS (ESI) calcd for $C_{15}H_{15}N_2OS$ [M+ H]$^+$: 271.0900; found: 271.0901.

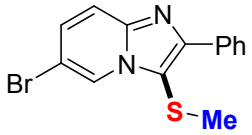
6-methyl-3-(methylthio)-2-phenylimidazo[1,2-a]pyridine (2e): yellow solid was obtained with 82% isolated yield (52.1 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24 – 8.18 (m, 2H), 8.16 (s, 1H), 7.47 (d, *J* = 9.1 Hz, 1H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.28 (t, *J* = 7.4 Hz, 1H), 7.03 (d, *J* = 9.1 Hz, 1H), 2.30 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.62, 145.38, 134.02, 129.02, 128.35, 128.17, 128.12, 122.50, 122.01, 116.96, 110.93, 18.45, 18.23. HRMS (ESI) calcd for $C_{15}H_{15}N_2S$ [M+ H]$^+$: 255.0950; found: 255.0947.



6-fluoro-3-(methylthio)-2-phenylimidazo[1,2-a]pyridine (2f):[2] yellow oil was obtained with 80% isolated yield (51.6 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.43 (s, 1H), 8.27 (d, *J* = 7.5 Hz, 2H), 7.73 (dd, *J* = 9.0, 4.5 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 2H), 7.41 (t, *J* = 7.3 Hz, 1H), 7.26 (d, *J* = 6.6 Hz, 1H), 2.27 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 154.00 (d, *J* = 238.7 Hz), 149.17, 143.28, 132.74, 128.76, 128.57, 128.16, 118.50 (d, *J* = 25.5 Hz), 117.91 (d, *J* = 8.6 Hz), 113.21, 111.29 (d, *J* = 41.8 Hz), 18.06. ^{19}F NMR (471 MHz, $CDCl_3$) δ -137.99. HRMS (ESI) calcd for $C_{14}H_{12}FN_2S$ [M+ H]$^+$: 259.0700; found: 259.0698.



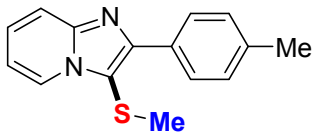
6-chloro-3-(methylthio)-2-phenylimidazo[1,2-a]pyridine (2g): white solid was obtained with 85% isolated yield (58.3 mg). ^{1}H NMR (500 MHz, DMSO) δ 8.74 (d, *J* = 1.3 Hz, 1H), 8.29 – 8.16 (m, 2H), 7.74 (d, *J* = 9.4 Hz, 1H), 7.54 – 7.46 (m, 3H), 7.42 (t, *J* = 7.3 Hz, 1H), 2.34 (s, 3H). ^{13}C NMR (126 MHz, DMSO) δ 148.84, 144.59, 133.80, 128.96, 128.91, 128.27, 127.97, 123.31, 120.86, 118.46, 112.48, 18.28. HRMS (ESI) calcd for $C_{14}H_{12}ClN_2S$ [M+ H]$^+$: 275.0404; found: 275.0403.



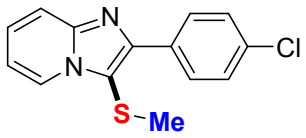
6-bromo-3-(methylthio)-2-phenylimidazo[1,2-a]pyridine (2h): white solid was obtained with 85% isolated yield (67.6 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.62 (d, *J* = 1.2 Hz, 1H), 8.30 – 8.25 (m, 2H), 7.56 (d, *J* = 9.4 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.43 – 7.38 (m, 1H), 7.36 (dd, *J* = 9.4, 1.9 Hz, 1H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 144.76, 133.39, 129.38, 128.57, 128.49, 128.41, 128.20, 124.55, 118.31, 107.71, 18.28. HRMS (ESI) calcd for $C_{14}H_{12}BrN_2S$ [M+ H]$^+$: 318.9899; found: 318.9895.

3-(methylthio)-2-phenyl-6-(trifluoromethyl)imidazo[1,2-a]pyridine (2i): yellow oil was obtained with 89% isolated yield (68.5 mg). ^{1}H NMR (500 MHz, DMSO) δ 8.95 (s, 1H), 8.32 – 8.21 (m, 2H), 7.90 (d, *J* = 9.3 Hz, 1H), 7.68 (dd, *J* = 9.4, 1.7 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 2H), 7.45 (t, *J* = 7.4 Hz, 1H), 2.37 (s, 3H). ^{13}C NMR (126 MHz, DMSO) δ 149.63, 146.01, 133.47, 129.21, 129.05,

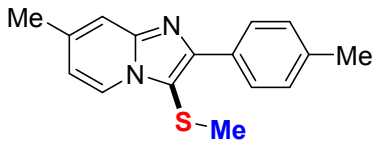
128.33, 125.54 (q, J = 5.9 Hz), 124.37 (q, J = 271.9 Hz), 122.56 (q, J = 5.7 Hz), 118.66, 116.06 (q, J = 33.6 Hz), 113.75, 18.45. ^{19}F NMR (471 MHz, $CDCl_3$) δ -55.60. HRMS (ESI) calcd for $C_{15}H_{12}F_3N_2S$ [M+ H] $^+$: 309.0668; found: 309.0666.



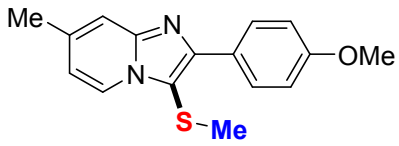
3-(methylthio)-2-(p-tolyl)imidazo[1,2-a]pyridine (2j): yellow solid was obtained with 72% isolated yield (45.7 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.51 (d, J = 6.8 Hz, 1H), 8.22 (d, J = 8.1 Hz, 2H), 7.74 (d, J = 8.9 Hz, 1H), 7.40 – 7.29 (m, 3H), 6.98 (t, J = 6.8 Hz, 1H), 2.44 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.52, 146.02, 138.37, 130.55, 129.19, 128.17, 126.20, 124.26, 117.41, 112.89, 111.74, 21.39, 18.15. HRMS (ESI) calcd for $C_{15}H_{15}N_2S$ [M+ H] $^+$: 255.0950; found: 255.0951.



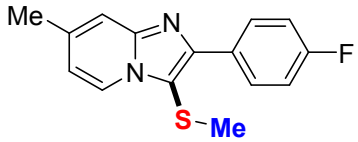
2-(4-chlorophenyl)-3-(methylthio)imidazo[1,2-a]pyridine (2k): white solid was obtained with 79% isolated yield (54.1 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.39 (d, J = 6.8 Hz, 1H), 8.19 (d, J = 8.5 Hz, 2H), 7.59 (d, J = 9.0 Hz, 1H), 7.37 (d, J = 8.5 Hz, 2H), 7.25 – 7.20 (m, 1H), 6.87 (t, J = 6.6 Hz, 1H), 2.17 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.50, 146.28, 134.24, 132.24, 129.48, 128.62, 128.43, 126.29, 124.29, 117.61, 112.98, 18.14. HRMS (ESI) calcd for $C_{14}H_{12}ClN_2S$ [M+H]$^+$: 275.0404; found: 275.0403.



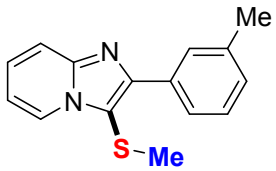
7-methyl-3-(methylthio)-2-(p-tolyl)imidazo[1,2-a]pyridine (2l): white solid was obtained with 78% isolated yield (52.3 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.27 (d, J = 6.9 Hz, 1H), 8.10 (d, J = 8.1 Hz, 2H), 7.39 (s, 1H), 7.21 (d, J = 7.9 Hz, 2H), 6.70 (d, J = 6.9 Hz, 1H), 2.37 (s, 3H), 2.34 (s, 3H), 2.17 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 145.64, 138.62, 130.13, 129.26, 129.25, 129.17, 128.17 123.60, 110.76, 21.43, 21.40, 18.26. HRMS (ESI) calcd for $C_{16}H_{17}N_2S$ [M+H]$^+$: 269.1107; found: 269.1106.



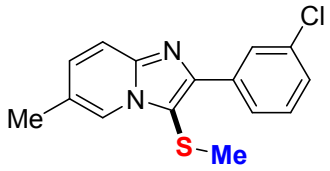
2-(4-methoxyphenyl)-7-methyl-3-(methylthio)imidazo[1,2-a]pyridine (2m): yellow solid was obtained with 95% isolated yield (67.5 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.22 (d, *J* = 6.8 Hz, 1H), 8.16 (d, *J* = 8.7 Hz, 2H), 7.33 (s, 1H), 6.92 (d, *J* = 8.7 Hz, 2H), 6.65 (d, *J* = 6.8 Hz, 1H), 3.77 (s, 3H), 2.33 (s, 3H), 2.13 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 158.71, 147.02, 145.32, 136.32, 128.45, 125.17, 122.34, 114.70, 114.32, 112.77, 108.74, 54.24, 20.31, 17.19. HRMS (ESI) calcd for $C_{16}H_{17}N_2OS$ $[M+H]^+$: 285.1056; found: 285.1053.



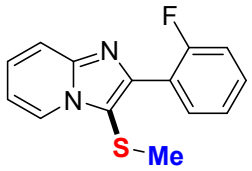
2-(4-fluorophenyl)-7-methyl-3-(methylthio)imidazo[1,2-a]pyridine (2n): yellow oil was obtained with 97% isolated yield (66.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.28 (d, *J* = 6.9 Hz, 1H), 8.25 – 8.13 (m, 2H), 7.39 (s, 1H), 7.08 (dd, *J* = 12.2, 5.3 Hz, 2H), 6.73 (dd, *J* = 6.9, 1.2 Hz, 1H), 2.38 (s, 3H), 2.17 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 160.22 (d, *J* = 249.4 Hz), 145.27, 130.30 (d, *J* = 8.3 Hz), 129.48, 124.03 (d, *J* = 3.6 Hz), 123.17, 122.08, 117.06, 115.96 (d, *J* = 22.3 Hz), 113.57, 18.47. ^{19}F NMR (471 MHz, $CDCl_3$) δ -113.18. HRMS (ESI) calcd for $C_{15}H_{14}FN_2S$ $[M+H]^+$: 273.0856; found: 273.0855.



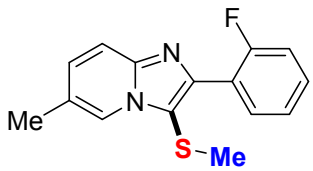
3-(methylthio)-2-(m-tolyl)imidazo[1,2-a]pyridine (2o): yellow solid was obtained with 90% isolated yield (57.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.44 (d, *J* = 6.8 Hz, 1H), 8.13 – 7.93 (m, 2H), 7.69 (d, *J* = 8.9 Hz, 1H), 7.29 (dt, *J* = 20.5, 7.9 Hz, 2H), 7.15 (d, *J* = 7.5 Hz, 1H), 6.91 (t, *J* = 6.7 Hz, 1H), 2.38 (s, 3H), 2.20 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.05, 145.71, 138.19, 132.83, 129.41, 129.01, 128.36, 126.69, 125.41, 124.35, 117.31, 113.24, 111.73, 21.57, 18.18. HRMS (ESI) calcd for $C_{15}H_{15}N_2S$ $[M+H]^+$: 255.0950; found: 255.0952.



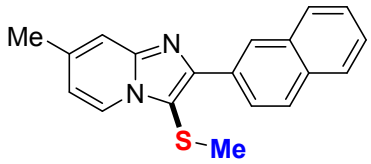
2-(3-chlorophenyl)-6-methyl-3-(methylthio)imidazo[1,2-a]pyridine (2p): yellow solid was obtained with 83% isolated yield (59.7 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.23 (d, *J* = 19.4 Hz, 2H), 8.15 (d, *J* = 7.7 Hz, 1H), 7.58 (d, *J* = 9.0 Hz, 1H), 7.34 (t, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 1H), 7.14 (d, *J* = 9.0 Hz, 1H), 2.36 (s, 3H), 2.20 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 146.44, 144.97, 134.42, 129.87, 129.70, 128.33, 128.10, 126.24, 123.23, 122.09, 116.85, 111.69, 18.48, 18.23. HRMS (ESI) calcd for $C_{15}H_{13}ClN_2S$ $[M+H]^+$: 289.0561; found: 289.0558.



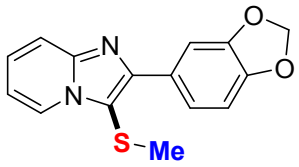
2-(2-fluorophenyl)-3-(methylthio)imidazo[1,2-a]pyridine (2q): white sloid was obtained with 79% isolated yield (50.9 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.39 (d, *J* = 6.8 Hz, 1H), 7.71 – 7.59 (m, 2H), 7.37 – 7.30 (m, 1H), 7.30 – 7.23 (m, 1H), 7.22 – 7.16 (m, 1H), 7.16 – 7.09 (m, 1H), 6.92 (t, *J* = 6.6 Hz, 1H), 2.17 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 160.23 (d, *J* = 249.7 Hz), 145.63 (d, *J* = 157.4 Hz), 132.17, 130.44 (d, *J* = 8.2 Hz), 126.44, 124.37, 124.10, 117.76, 116.09, 115.91, 113.32, 17.90. ^{19}F NMR (471 MHz, $CDCl_3$) δ -113.42. HRMS (ESI) calcd for $C_{14}H_{12}FN_2S$ $[M+H]^+$: 259.0700; found: 259.0698.



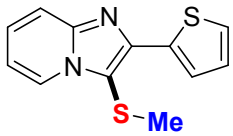
2-(2-fluorophenyl)-6-methyl-3-(methylthio)imidazo[1,2-a]pyridine (2r): yellow oil was obtained with 73% isolated yield (49.6 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.17 (s, 1H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.58 (d, *J* = 9.1 Hz, 1H), 7.33 (dd, *J* = 13.6, 6.7 Hz, 1H), 7.21 – 7.18 (m, 1H), 7.12 (t, *J* = 7.8 Hz, 2H), 2.36 (s, 3H), 2.18 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 160.22 (d, *J* = 249.4 Hz), 145.27, 144.79, 132.16 (d, *J* = 3.0 Hz), 130.30 (d, *J* = 8.3 Hz), 129.48, 124.03 (d, *J* = 3.6 Hz), 123.17, 122.08, 117.06, 116.05, 115.87, 113.57, 18.47, 17.92. ^{19}F NMR (471 MHz, $CDCl_3$) δ -113.45. HRMS (ESI) calcd for $C_{15}H_{14}FN_2S$ $[M + H]^+$: 273.0856; found: 273.0855.



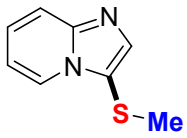
7-methyl-3-(methylthio)-2-(naphthalen-2-yl)imidazo[1,2-a]pyridine (2s): yellow solid was obtained with 95% isolated yield (72.2 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.29 (d, *J* = 6.9 Hz, 1H), 7.99 (d, *J* = 8.3 Hz, 1H), 7.82 (dd, *J* = 12.1, 8.3 Hz, 2H), 7.58 (d, *J* = 7.0 Hz, 1H), 7.51 – 7.45 (m, 1H), 7.44 – 7.33 (m, 3H), 6.74 (dd, *J* = 6.9, 1.3 Hz, 1H), 2.38 (s, 3H), 2.03 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.14, 146.68, 137.10, 133.80, 132.40, 131.52, 128.85, 128.72, 128.16, 126.45, 126.20, 125.75, 125.00, 123.67, 116.31, 115.53, 113.26, 21.41, 18.72. HRMS (ESI) calcd for $C_{20}H_{21}N_2O$ [M + H]$^+$; 305.1107; found: 305.1104.



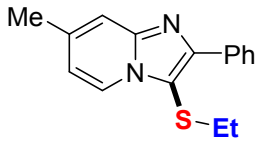
2-(benzo[d][1,3]dioxol-5-yl)-3-(methylthio)imidazo[1,2-a]pyridine (2t): yellow oil was obtained with 70% isolated yield (40.1 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.46 (d, *J* = 6.8 Hz, 1H), 7.85 (dt, *J* = 4.1, 2.0 Hz, 2H), 7.63 (d, *J* = 9.0 Hz, 1H), 7.31 – 7.25 (m, 1H), 6.92 (dd, *J* = 9.8, 5.5 Hz, 2H), 6.02 (s, 2H), 2.25 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.53, 147.73, 147.71, 146.19, 128.00, 125.89, 124.20, 122.34, 117.46, 112.63, 110.61, 108.64, 108.34, 101.13, 18.12. HRMS (EI) calcd for $C_{15}H_{13}N_2O_2S$ [M + H] $^+$: 285.0692; found: 285.0688.



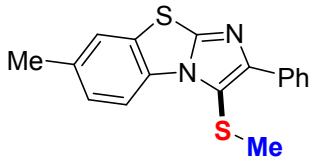
3-(methylthio)-2-(thiophen-2-yl)imidazo[1,2-a]pyridine (**2u**): yellow oil was obtained with 74% isolated yield (45.5mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.37 (d, *J* = 6.7 Hz, 1H), 8.00 (d, *J* = 3.5 Hz, 1H), 7.57 (d, *J* = 8.9 Hz, 1H), 7.31 (t, *J* = 11.2 Hz, 1H), 7.24 – 7.19 (m, 1H), 7.11 – 7.03 (m, 1H), 6.85 (t, *J* = 6.7 Hz, 1H), 2.22 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 146.35, 144.57, 136.60, 127.67, 126.50, 126.31, 126.28, 124.18, 117.33, 112.87, 110.29, 17.85. HRMS (ESI) calcd for $C_{12}H_{11}N_2S_2$ [M + H] $^+$: 247.0358; found: 247.0356.



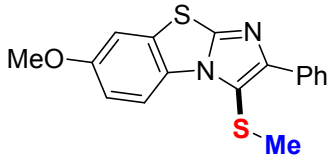
3-(methylthio)imidazo[1,2-a]pyridine (2v): yellow oil was obtained with 84% isolated yield (34.5 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.37 (d, *J* = 6.7 Hz, 1H), 7.74 (s, 1H), 7.66 (d, *J* = 8.9 Hz, 1H), 7.31 – 7.22 (m, 1H), 6.93 (t, *J* = 6.7 Hz, 1H), 2.24 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 146.80, 138.81, 126.15, 124.19, 117.79, 113.35, 19.33. HRMS (EI) calcd for $C_8H_9N_2S$ $[M+H]^+$: 165.0481; found: 165.0482.



3-(ethylthio)-7-methyl-2-phenylimidazo[1,2-a]pyridine (2w): yellow solid was obtained with 70% isolated yield (46.9 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.32 (d, *J* = 6.9 Hz, 1H), 8.24 (d, *J* = 7.3 Hz, 2H), 7.39 (t, *J* = 7.6 Hz, 3H), 7.29 (t, *J* = 7.4 Hz, 1H), 6.69 (dd, *J* = 6.9, 1.0 Hz, 1H), 2.60 (q, *J* = 7.4 Hz, 2H), 2.36 (s, 3H), 1.03 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.71, 146.37, 137.75, 133.44, 128.33, 128.31, 128.29, 123.64, 115.81, 115.59, 109.53, 29.99, 21.38, 14.75. HRMS (ESI) calcd for $C_{16}H_{17}N_2S$ $[M + H]^+$: 269.1107; found: 269.1108.

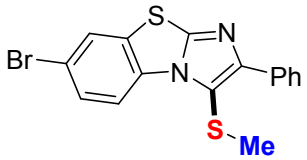


7-methyl-3-(methylthio)-2-phenylbenzo[d]imidazo[2,1-b]thiazole (4a): yellow solid was obtained with 86% isolated yield (66.6 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40 (d, *J* = 8.4 Hz, 1H), 8.09 (d, *J* = 7.6 Hz, 2H), 7.40 (s, 1H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.27 (t, *J* = 7.2 Hz, 1H), 7.20 – 7.15 (m, 1H), 2.37 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.48, 149.23, 134.96, 133.76, 131.67, 130.33, 128.33, 127.85, 127.67, 127.34, 124.17, 115.15, 113.68, 21.36, 20.34. HRMS (ESI) calcd for $C_{17}H_{15}N_2S_2$ $[M + H]^+$: 311.0671; found: 311.0670.



7-methoxy-3-(methylthio)-2-phenylbenzo[d]imidazo[2,1-b]thiazole (4b): yellow solid was obtained with 98% isolated yield (79.9 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.45 (d, *J* = 9.0 Hz, 1H),

8.09 (d, *J* = 7.6 Hz, 2H), 7.38 (t, *J* = 7.6 Hz, 2H), 7.28 (t, *J* = 7.3 Hz, 1H), 7.14 (d, *J* = 2.3 Hz, 1H), 6.97 (dd, *J* = 9.0, 2.4 Hz, 1H), 3.81 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 157.08, 150.20, 148.70, 133.77, 131.65, 128.34, 127.94, 127.81, 127.61, 114.69, 113.52, 108.47, 55.90, 20.30. HRMS (EI) calcd for $C_{17}H_{15}N_2OS_2$ [M + H] $^+$: 327.0620; found: 327.0619.

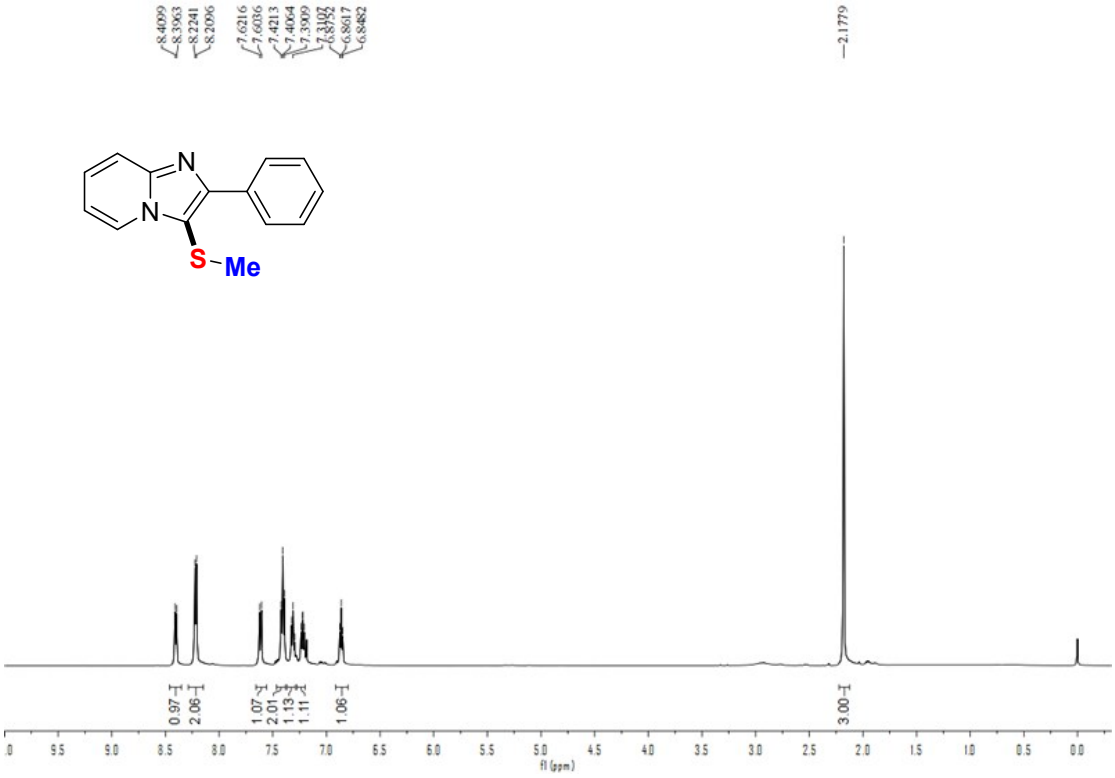


7-bromo-3-(methylthio)-2-phenylbenzo[d]imidazo[2,1-b]thiazole (4c): yellow solid was obtained with 79% isolated yield (73.7 mg). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.45 (d, *J* = 8.7 Hz, 1H), 8.09 (d, *J* = 7.4 Hz, 2H), 7.77 (d, *J* = 1.8 Hz, 1H), 7.52 (dd, *J* = 8.7, 1.9 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.29 (t, *J* = 7.3 Hz, 1H), 2.30 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 151.09, 149.01, 133.39, 132.71, 132.10, 129.54, 128.41, 128.13, 127.68, 126.68, 117.67, 115.53, 115.13, 20.29. HRMS (ESI) calcd for $C_{16}H_{12}BrN_2S_2$ [M + H]$^+$: 374.9620; found 376.9592.

8. Copies of product NMR Spectra

2a

^{1}HNMR



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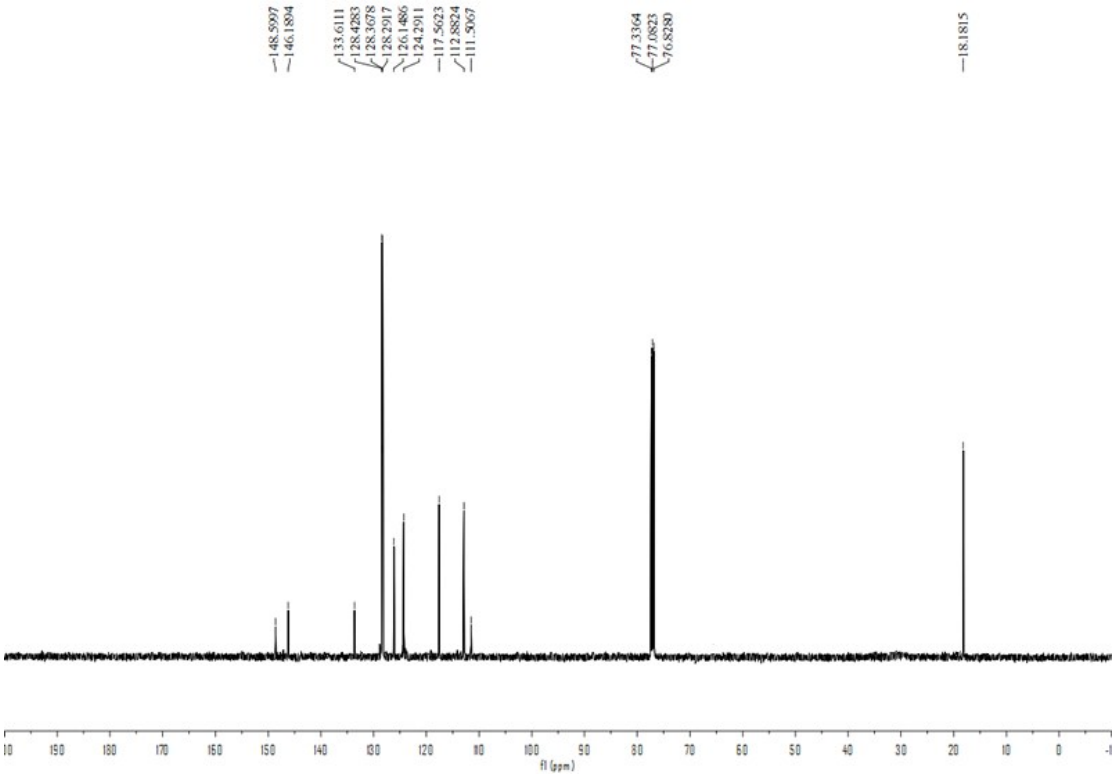
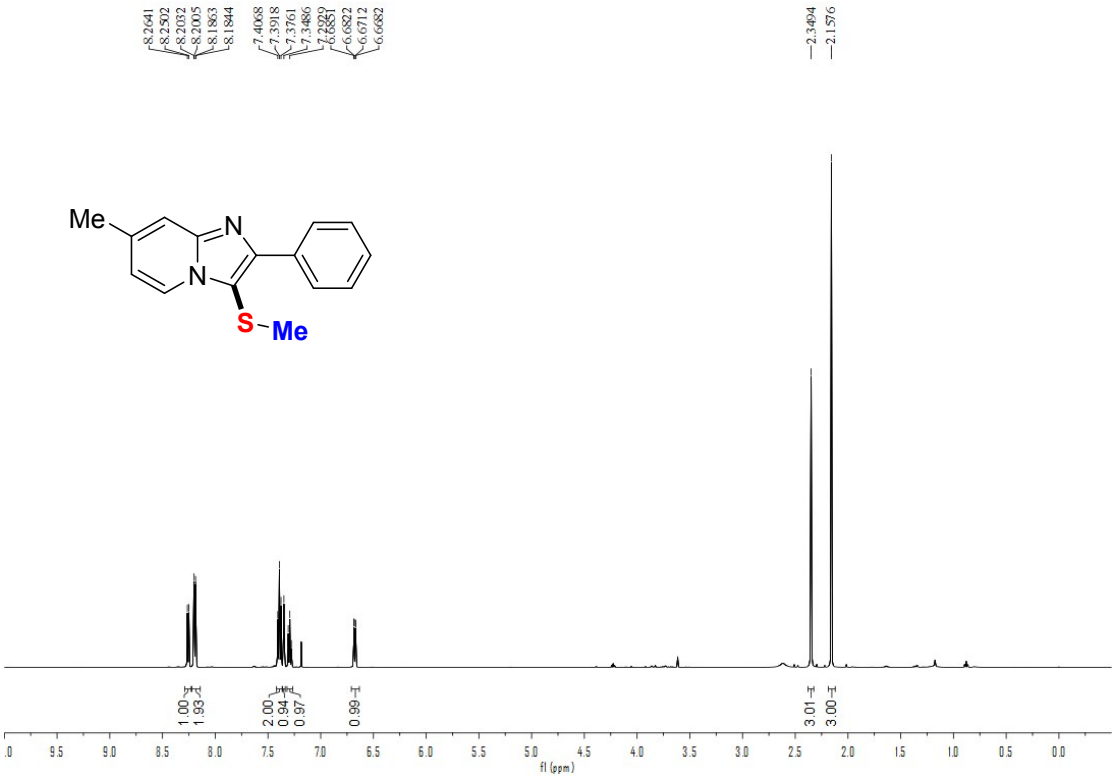


2b

[1]HNMR



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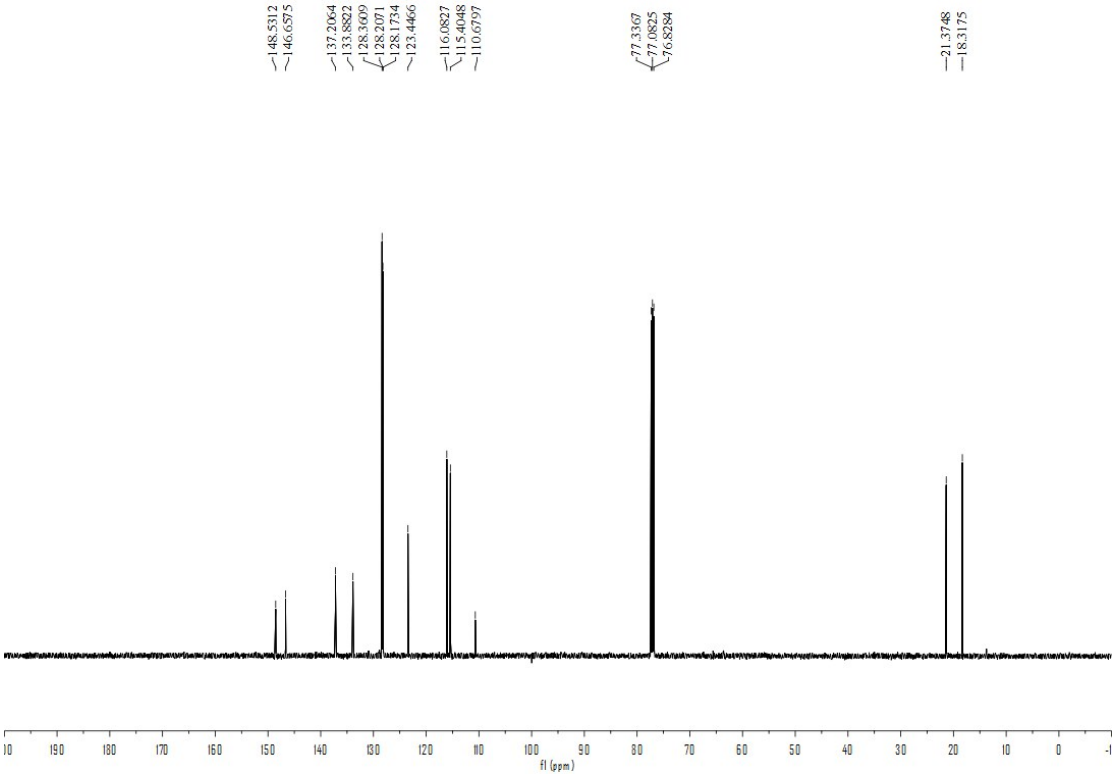
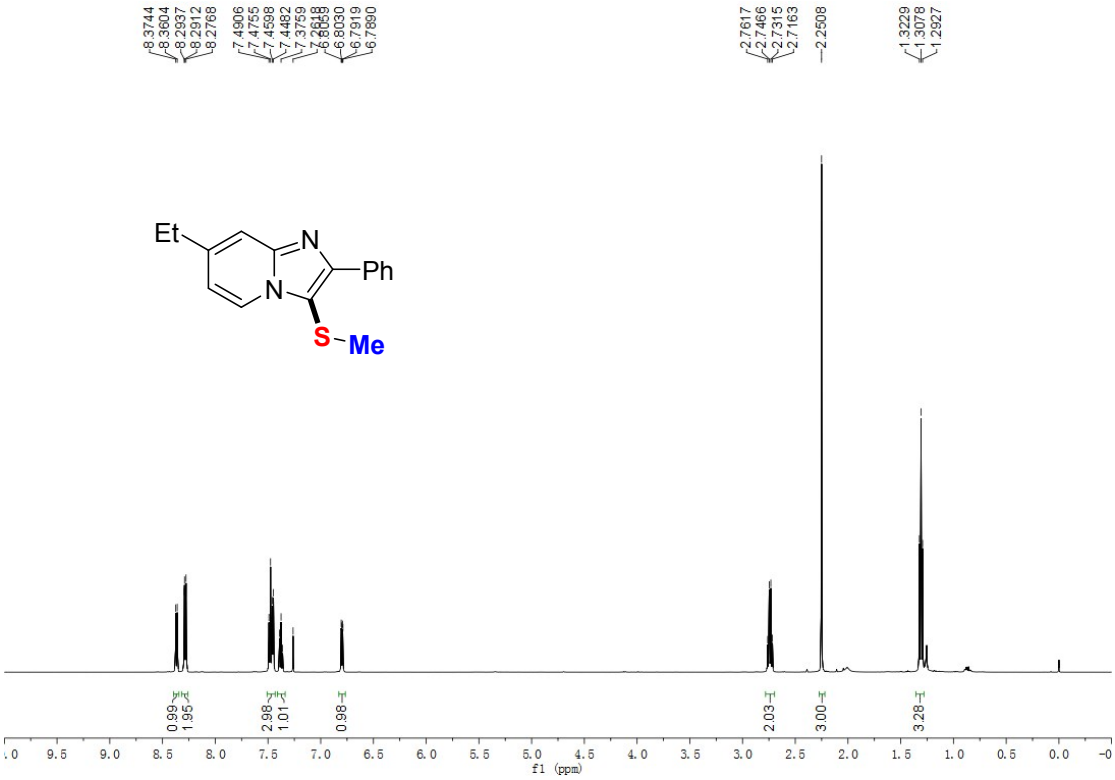


2c

[1]HNMR



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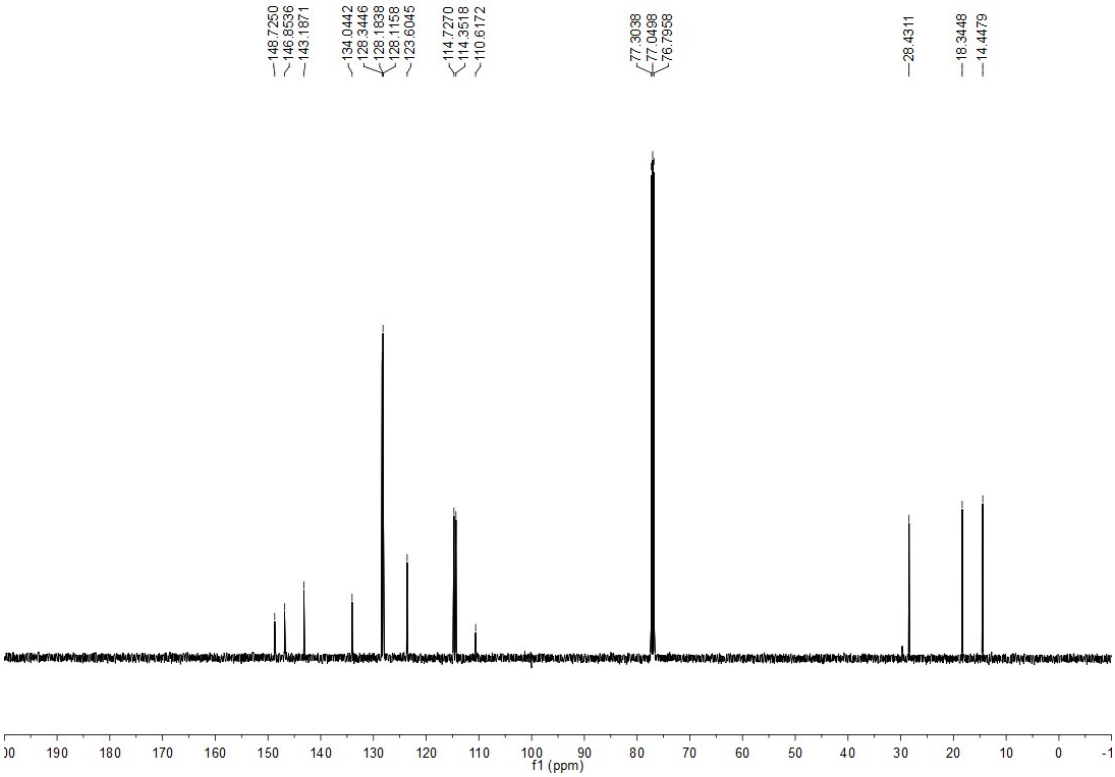

2d

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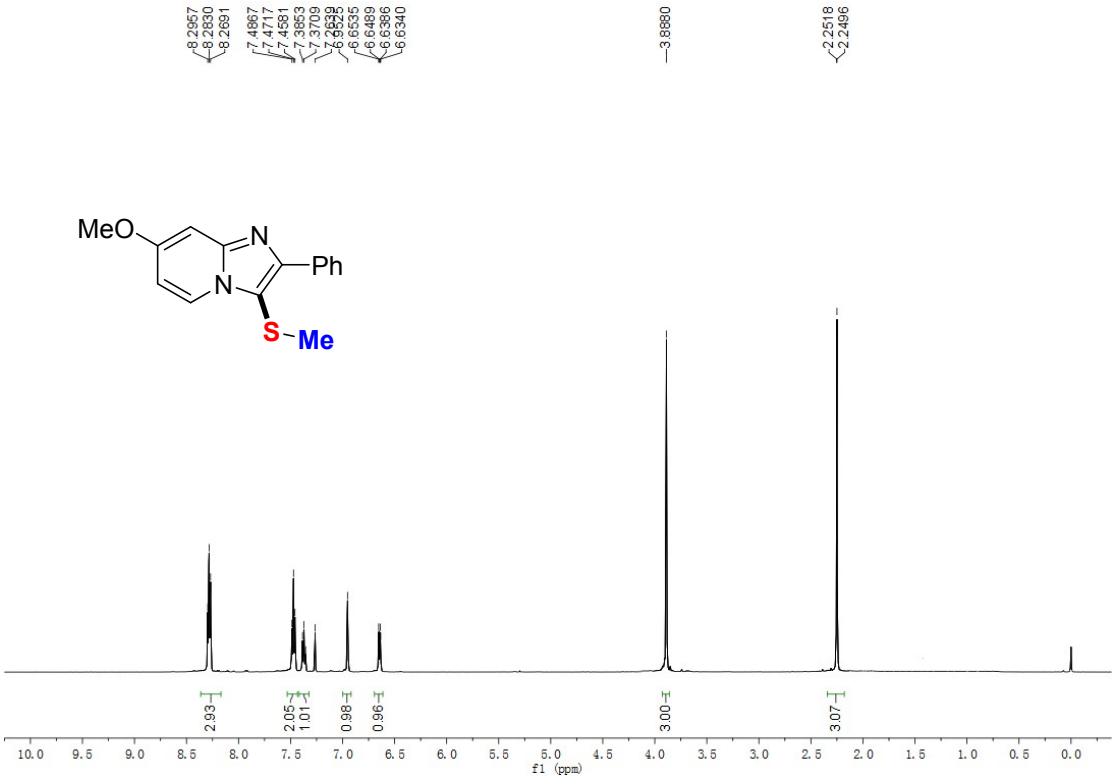

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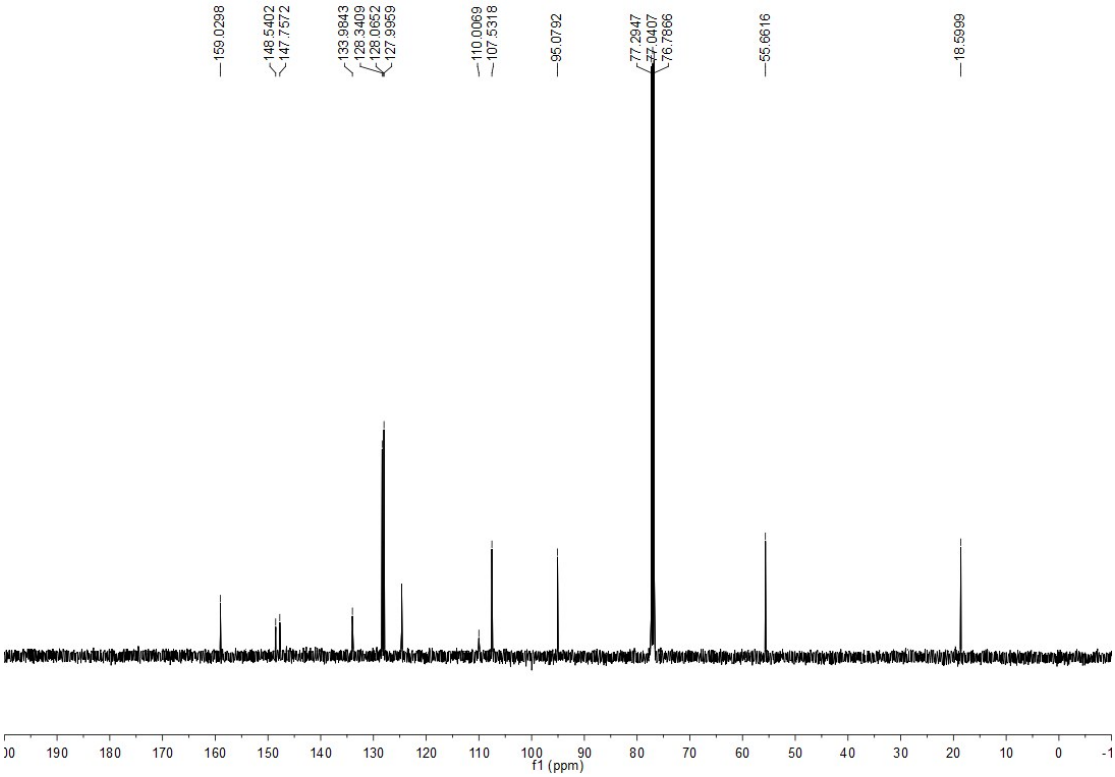

2e

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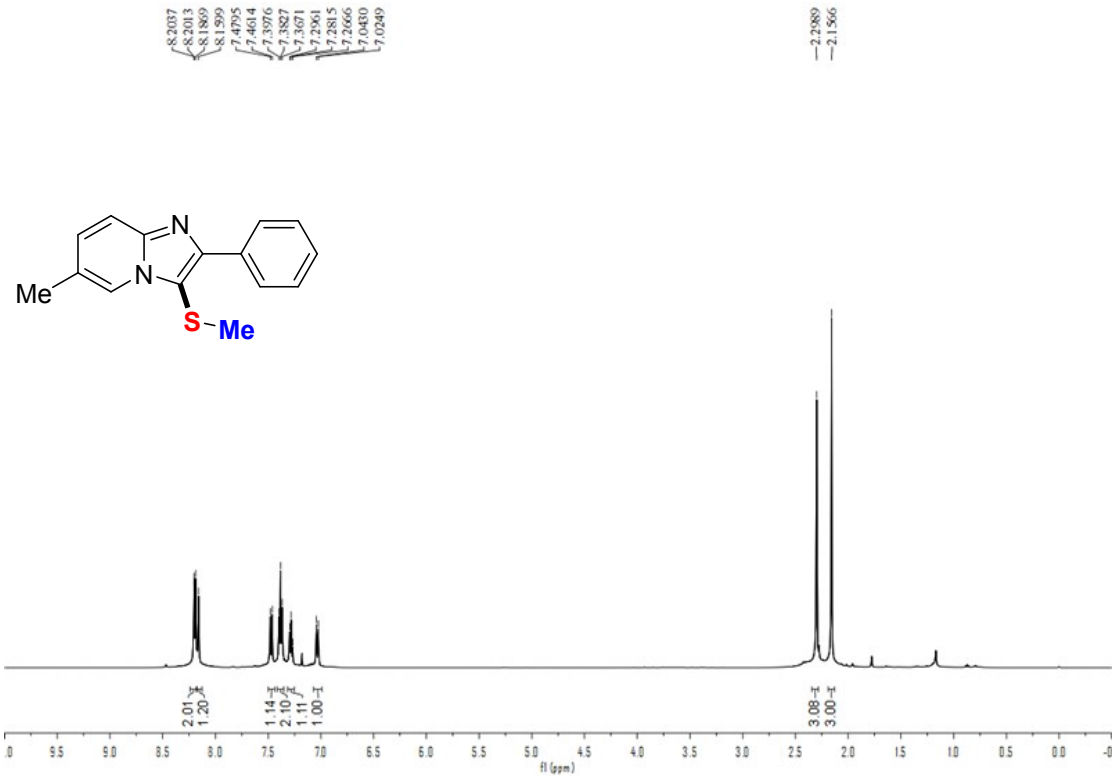

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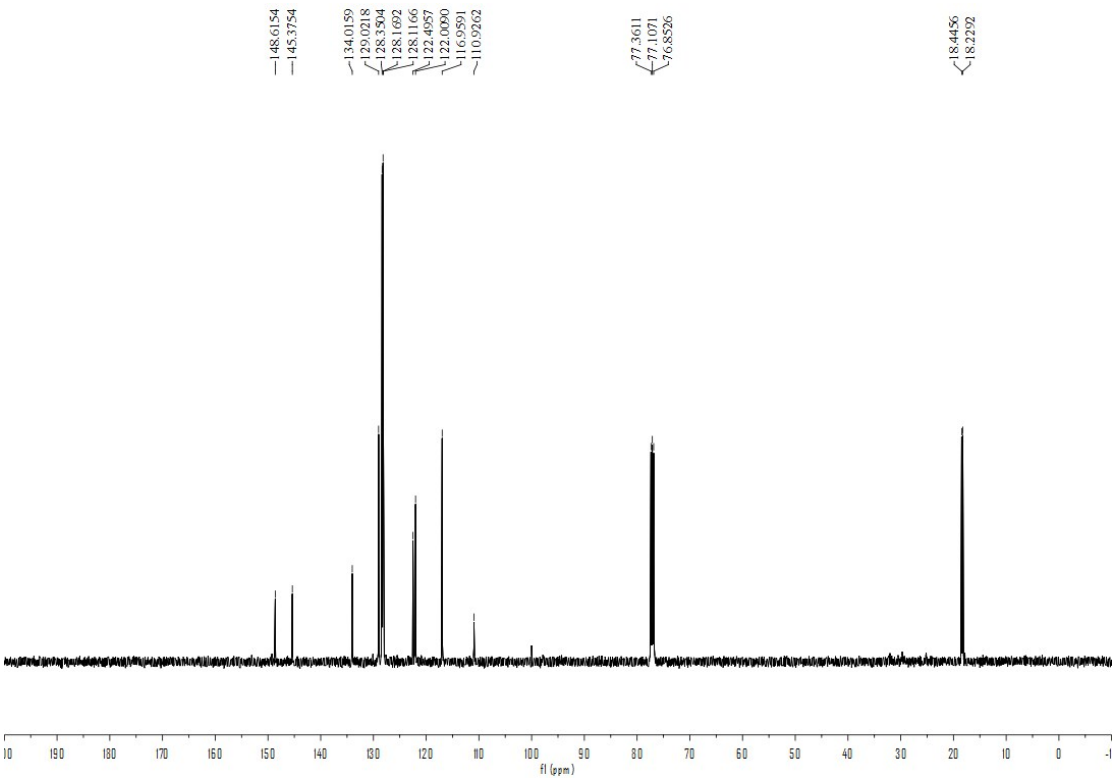
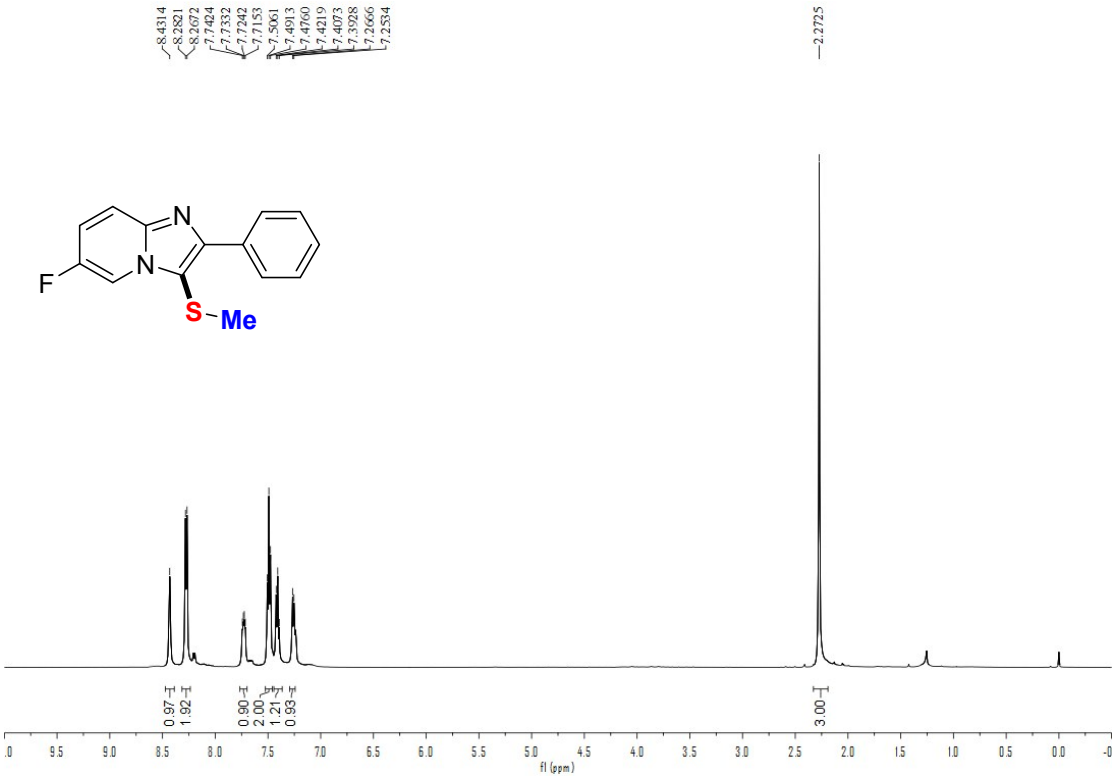


2f

[1]HNMR



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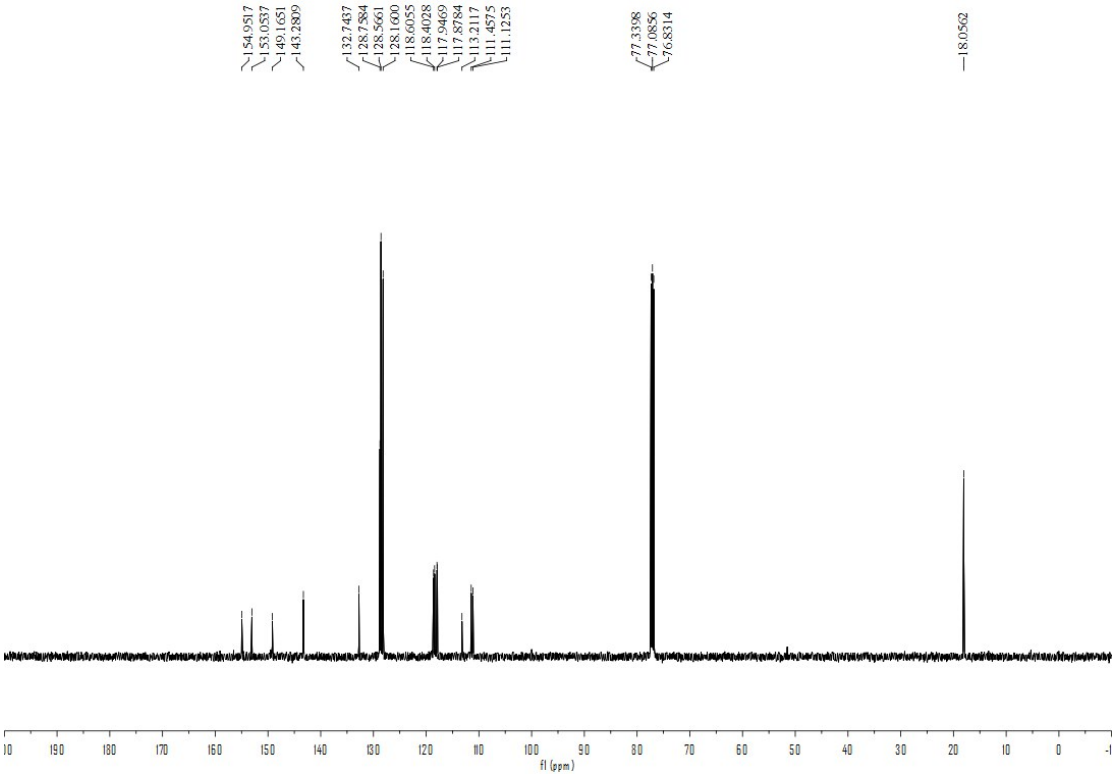

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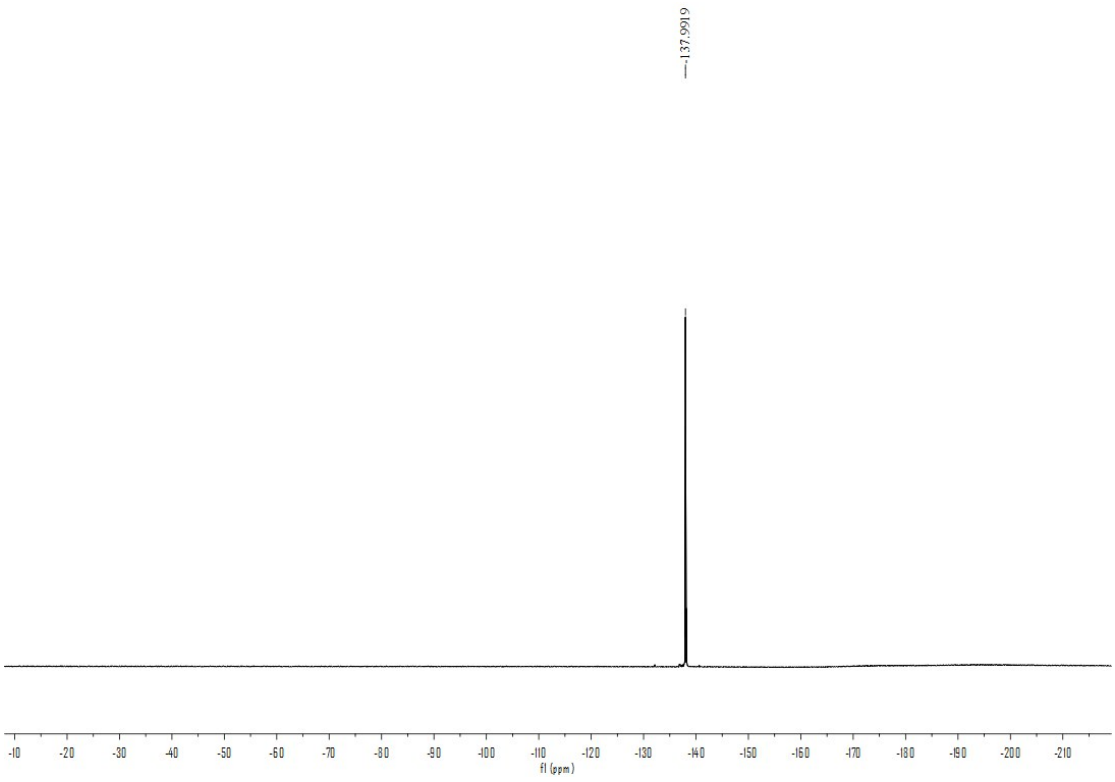
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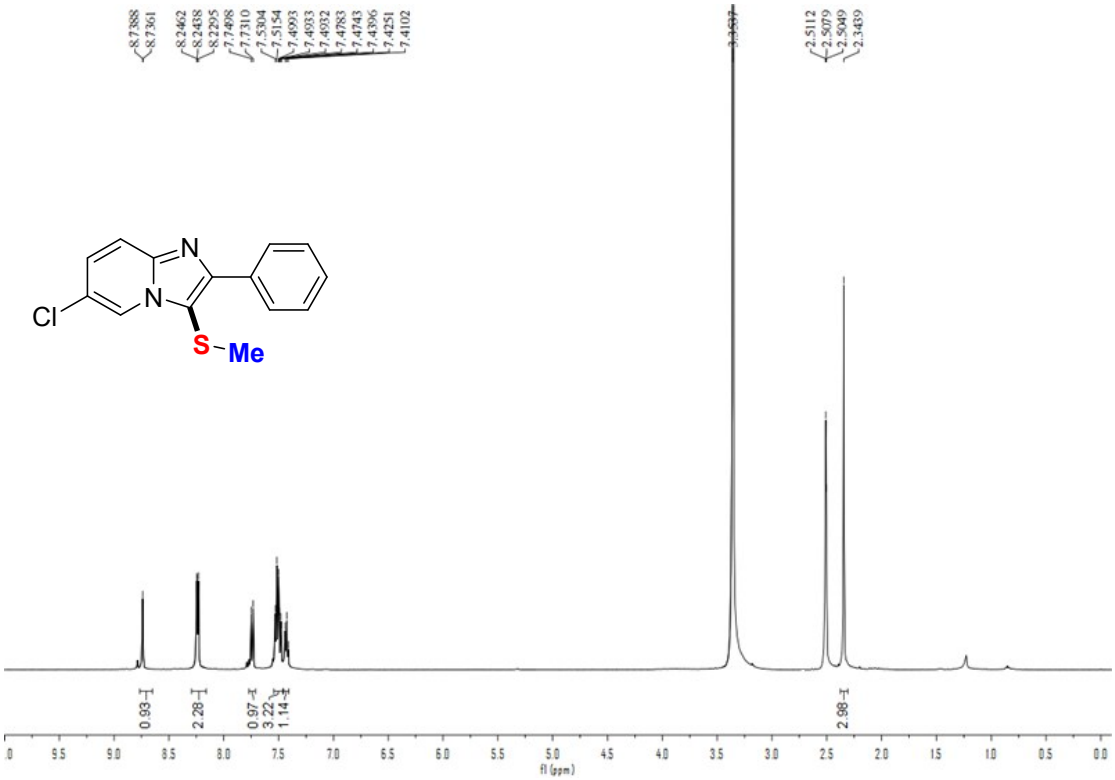
f1 (ppm)

2g

[1]HNMR



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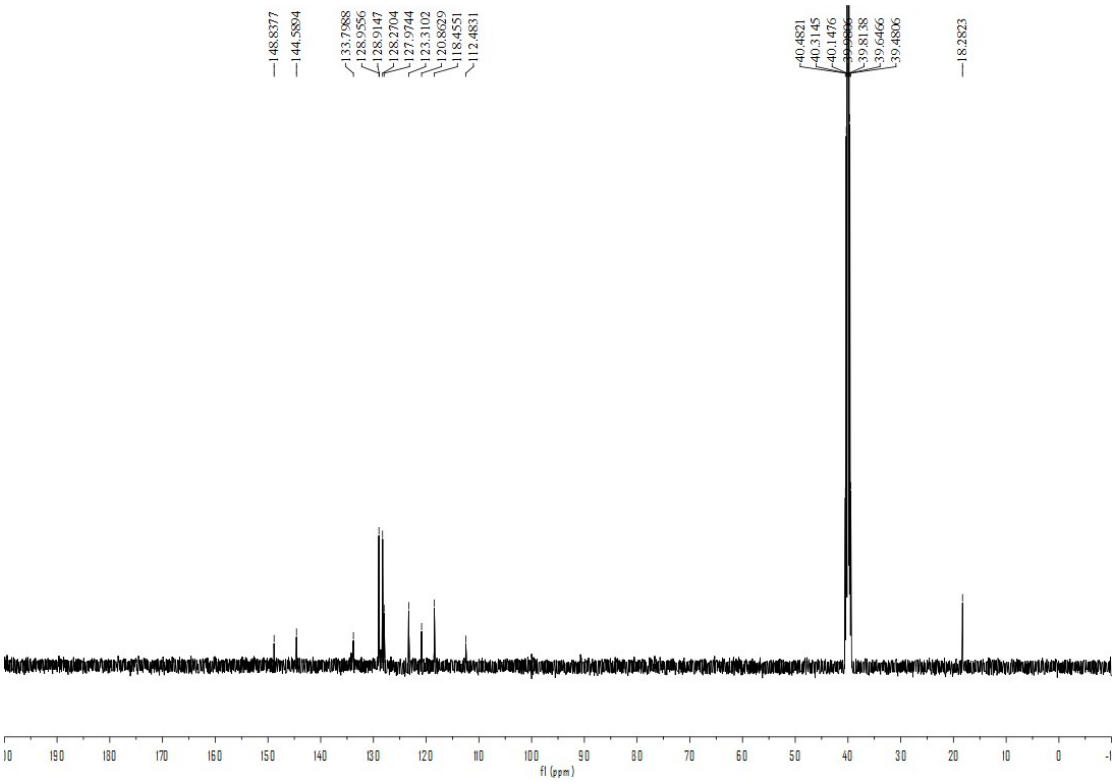

2h

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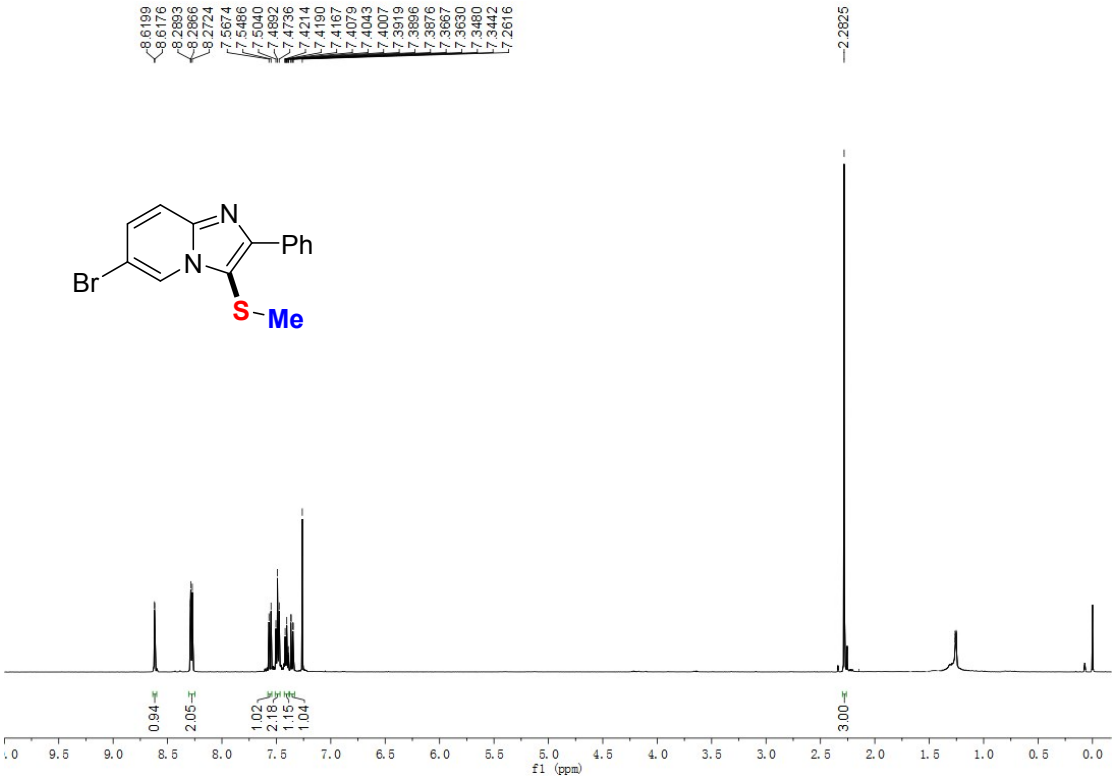

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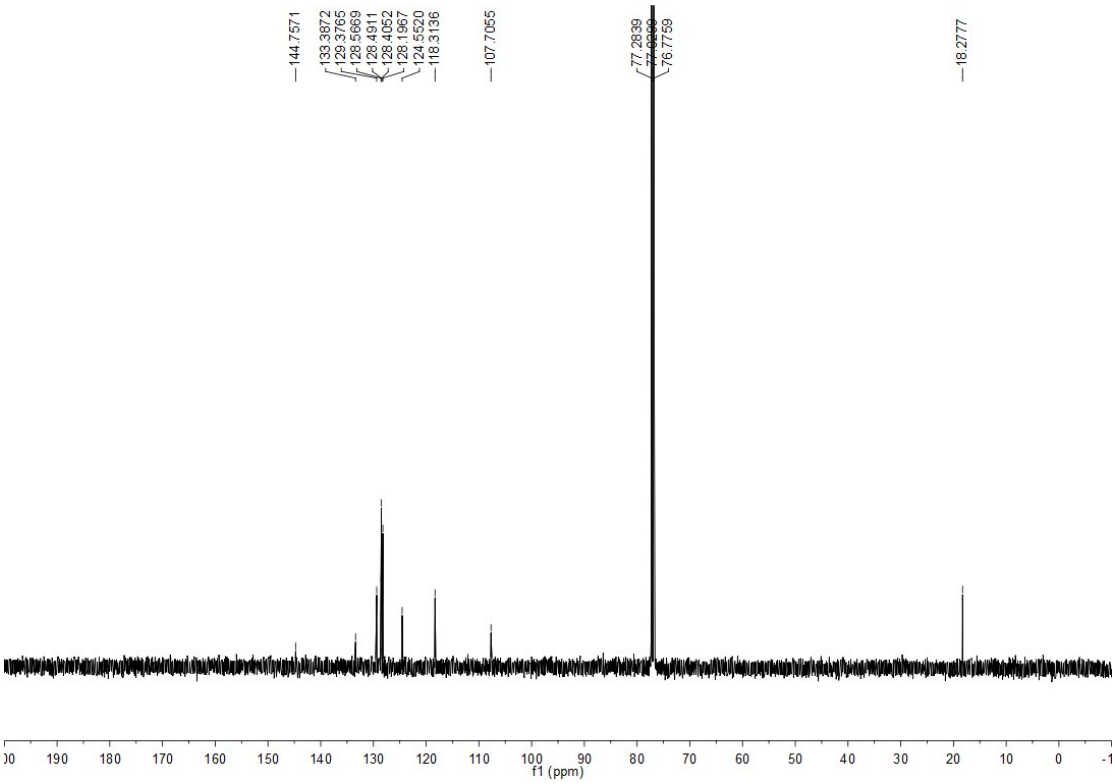

2i

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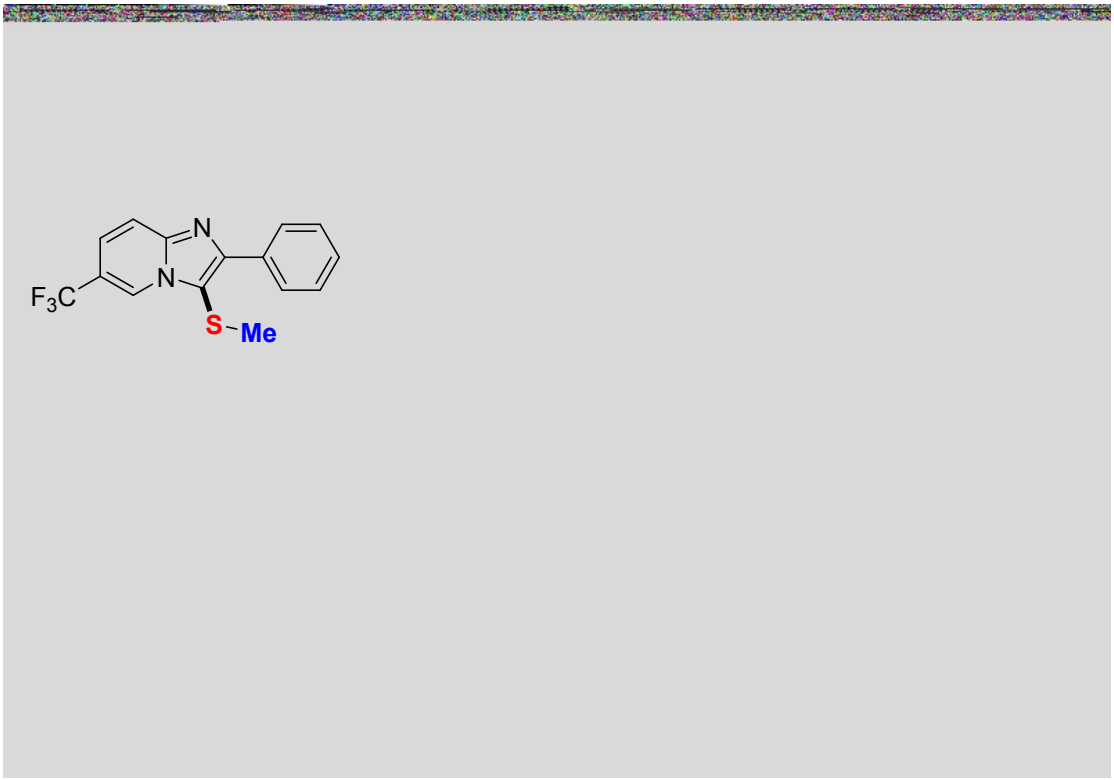

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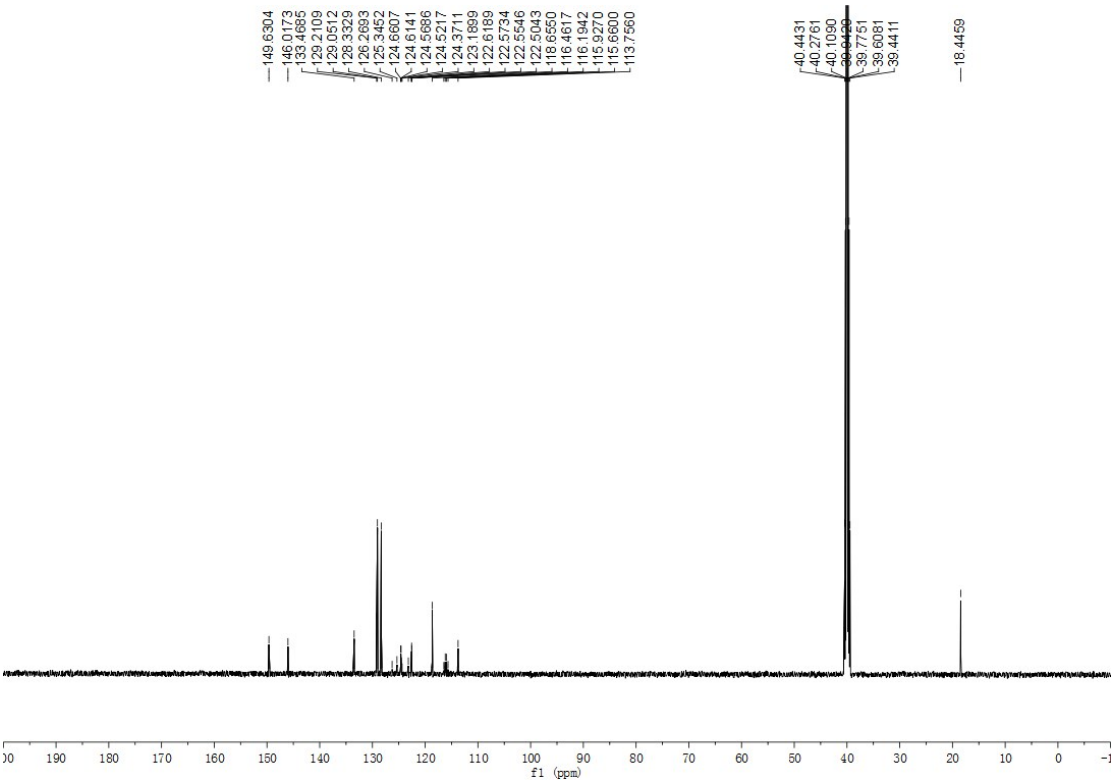

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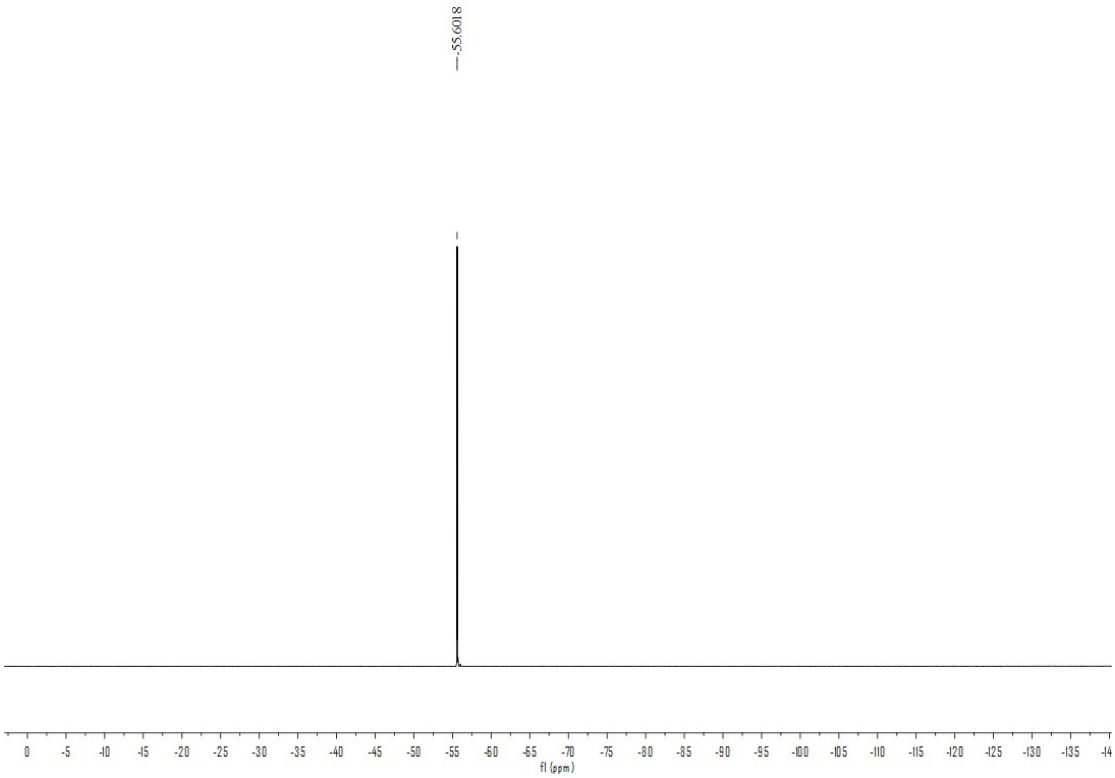
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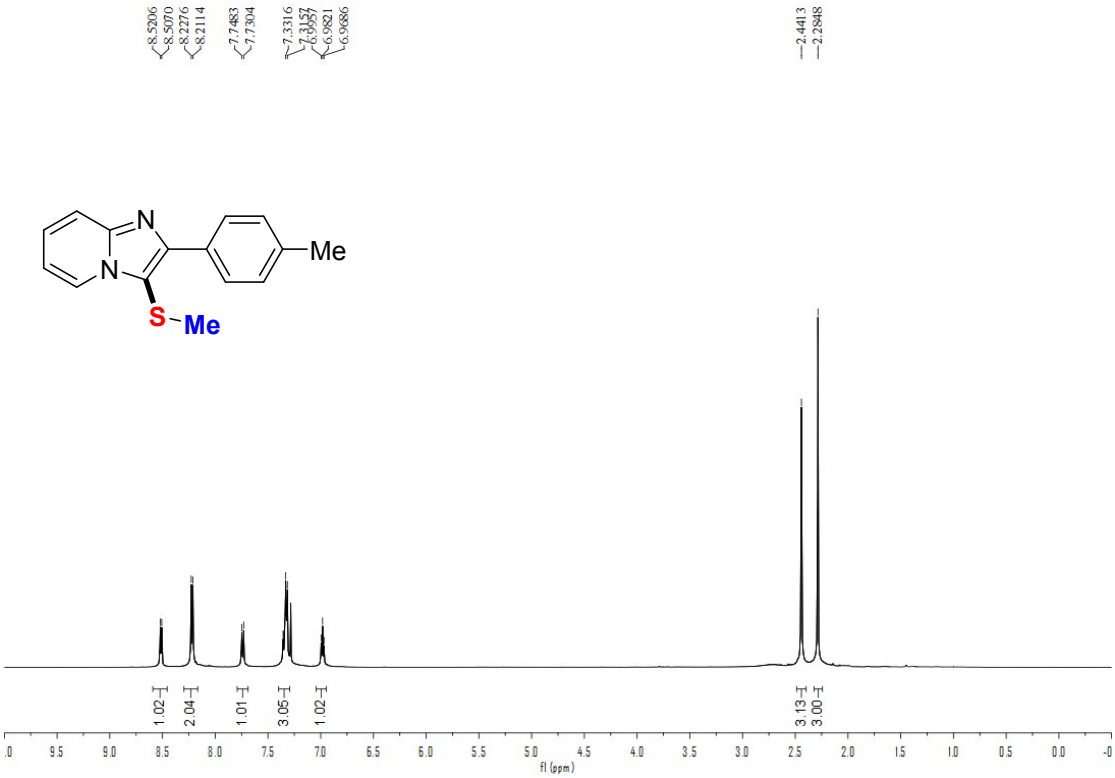
f1 (ppm)

2j

[1]HNMR



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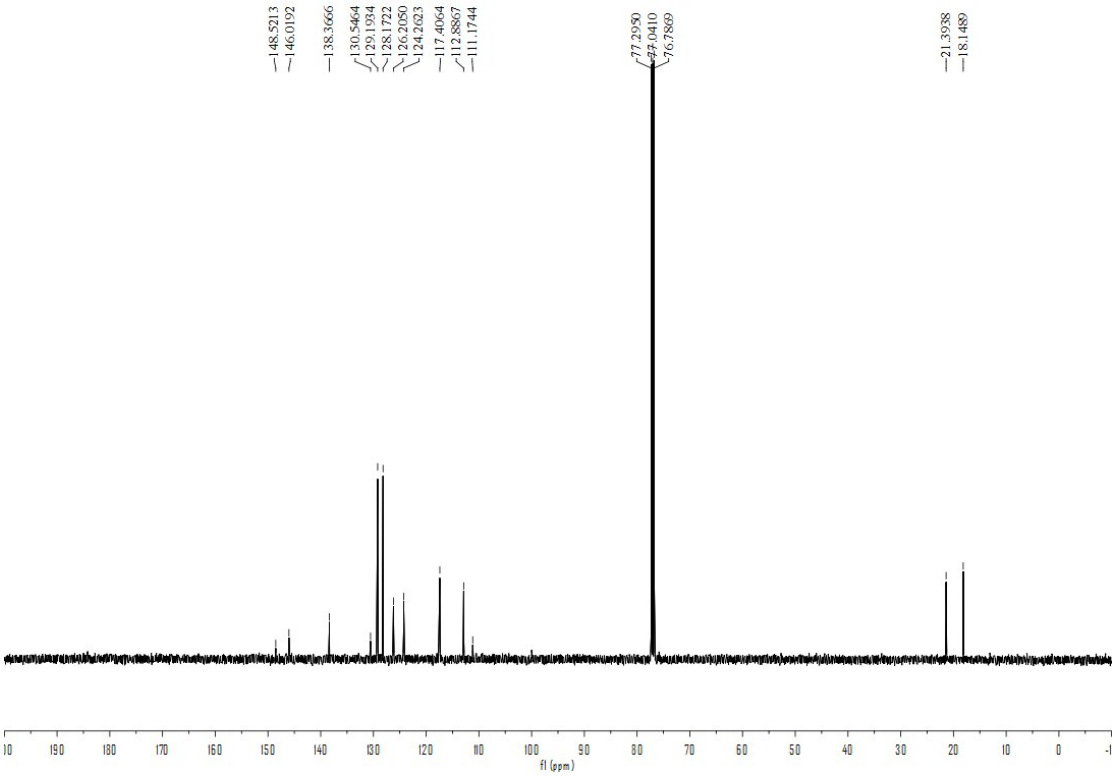

2k

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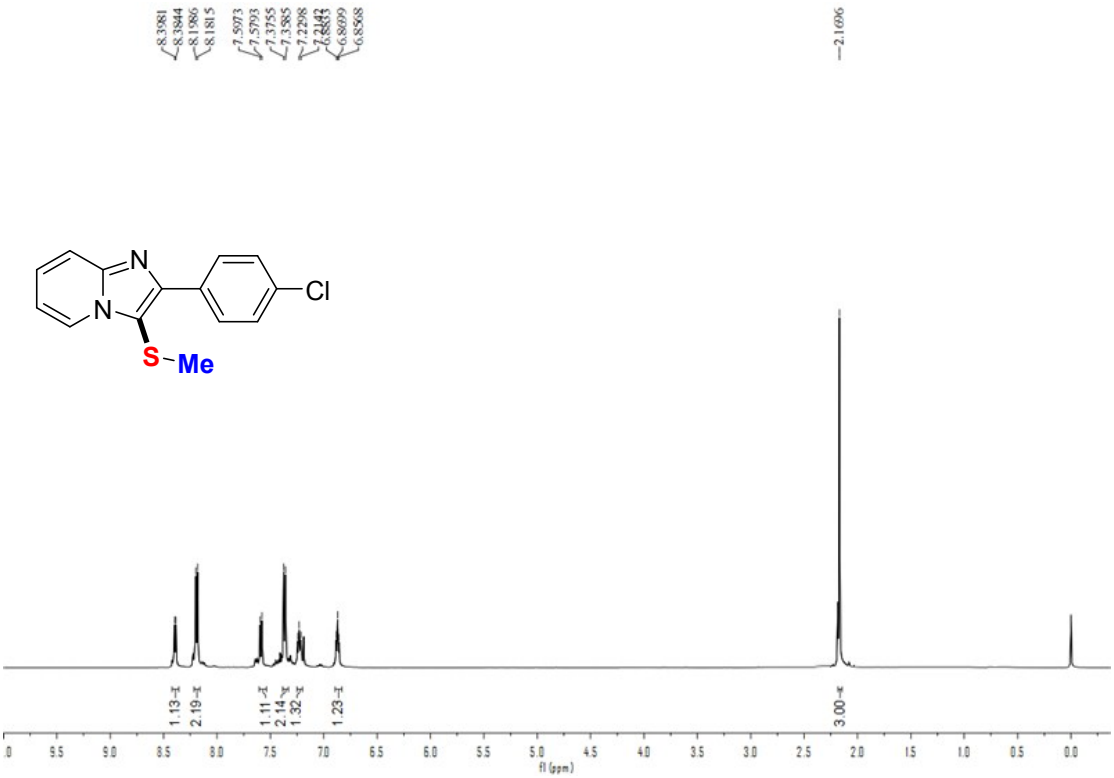

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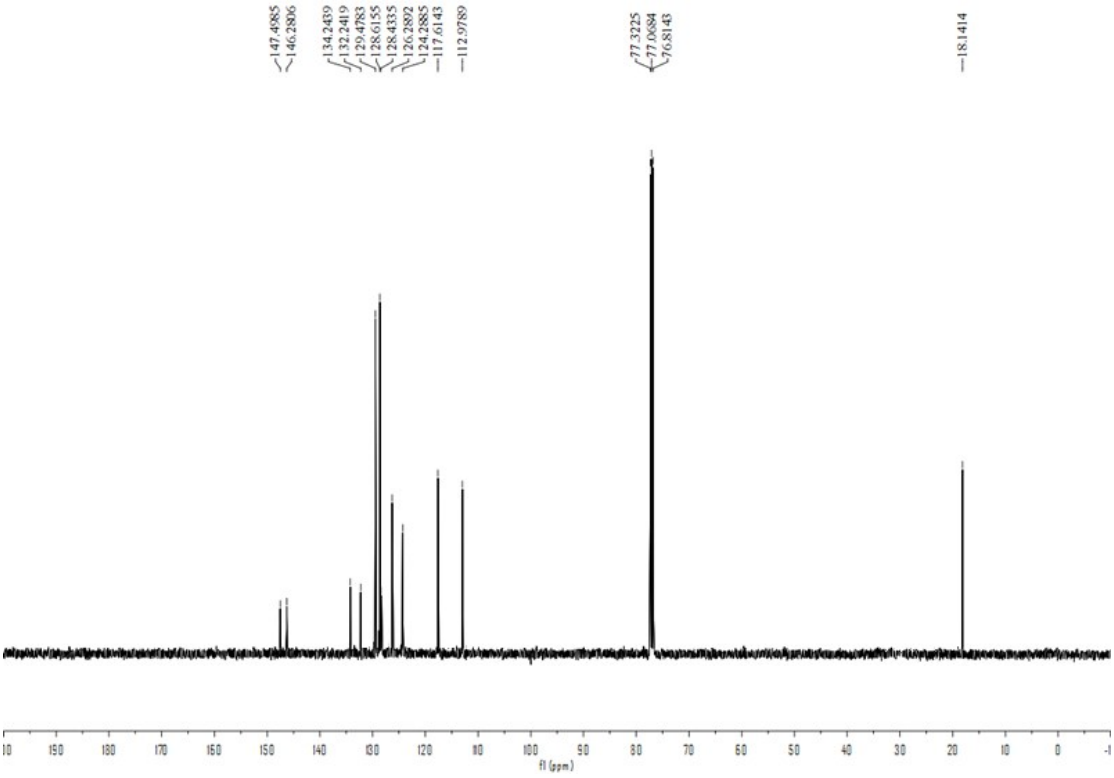

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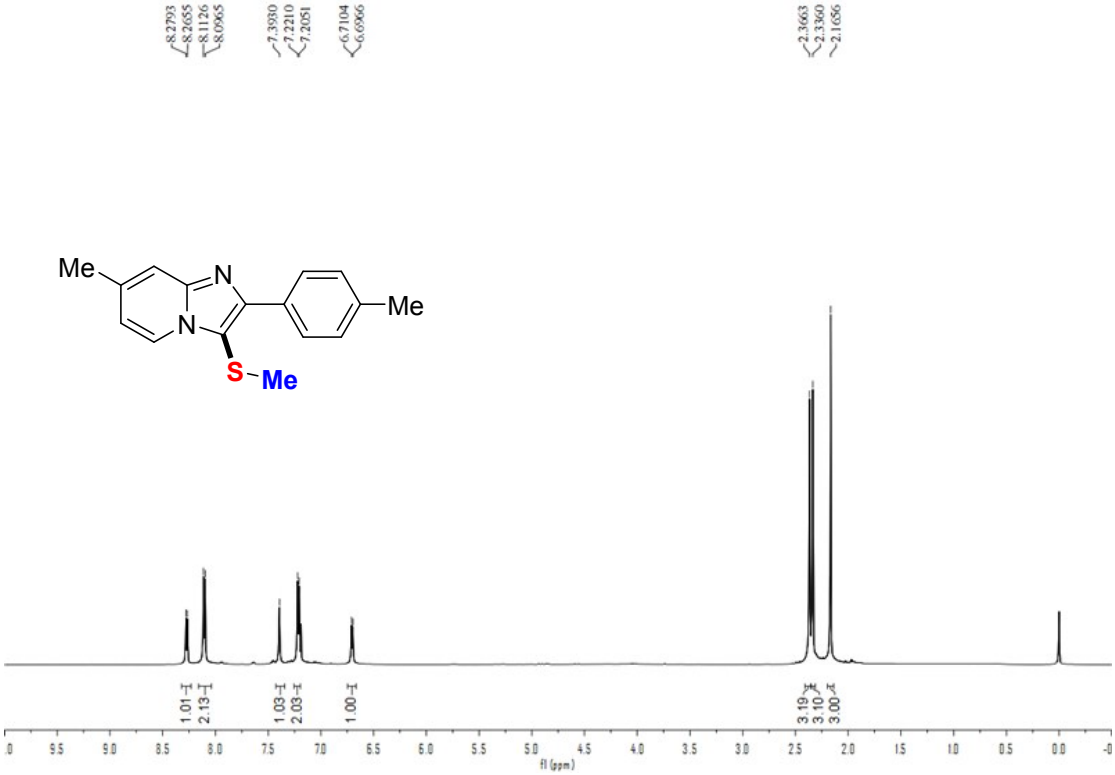

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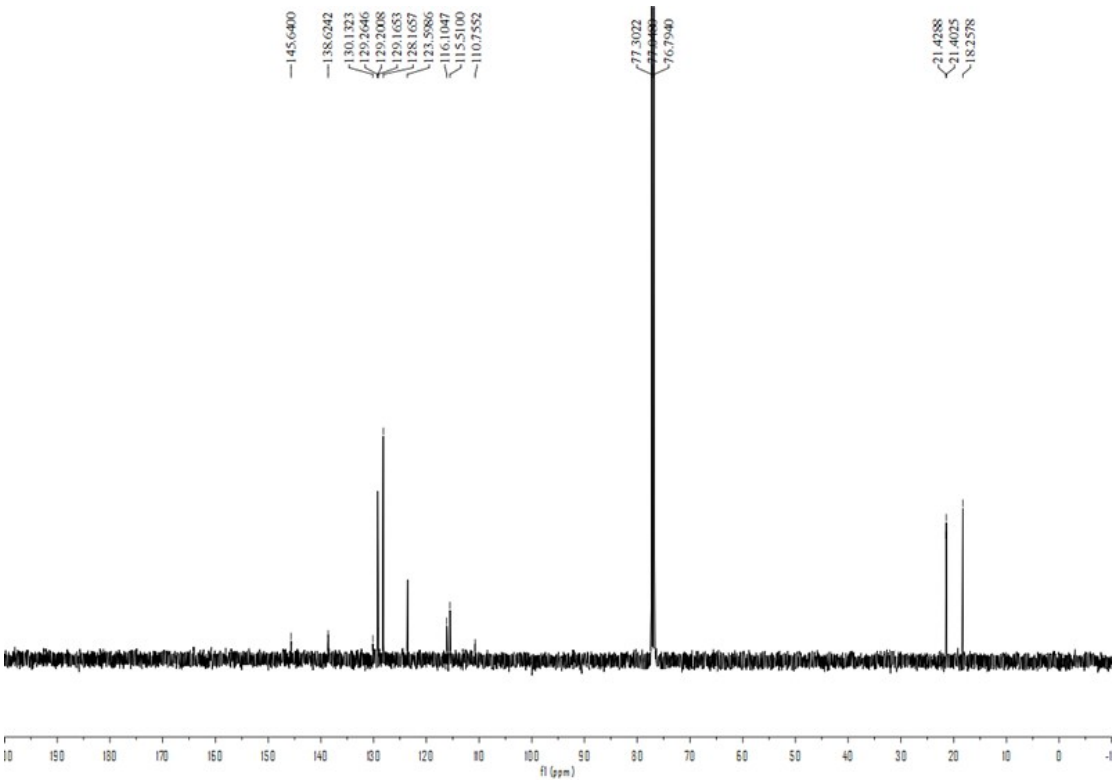
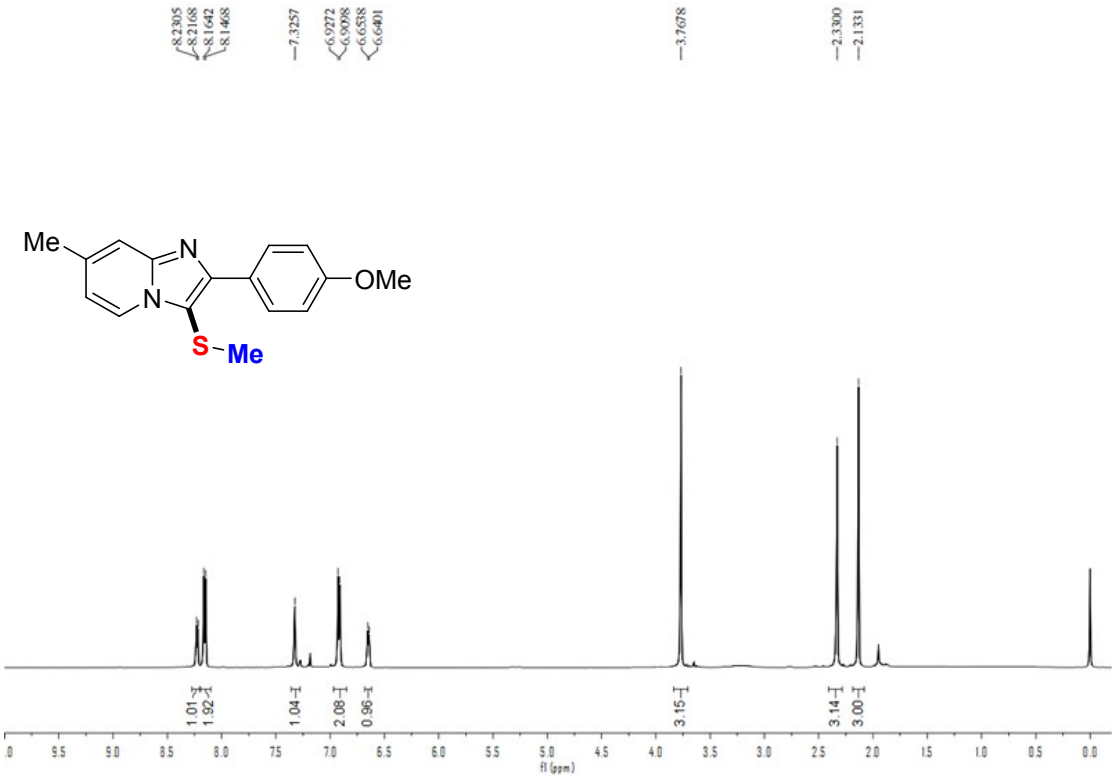


2m

[1]HNMR



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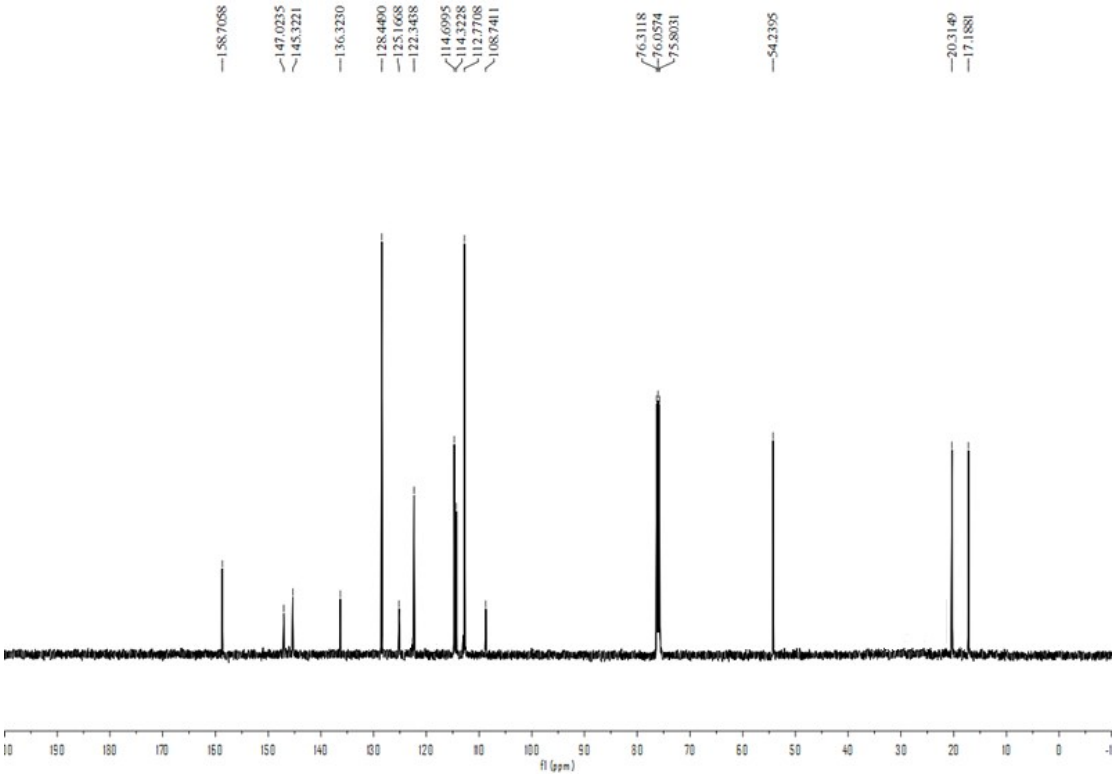

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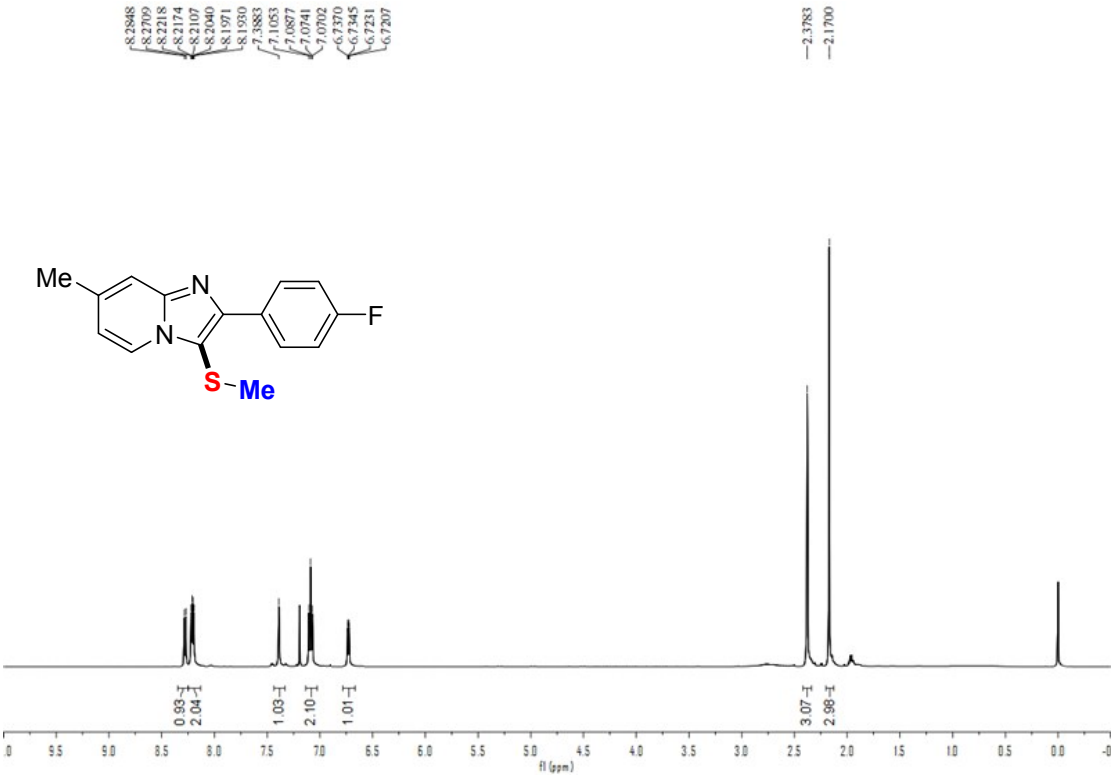

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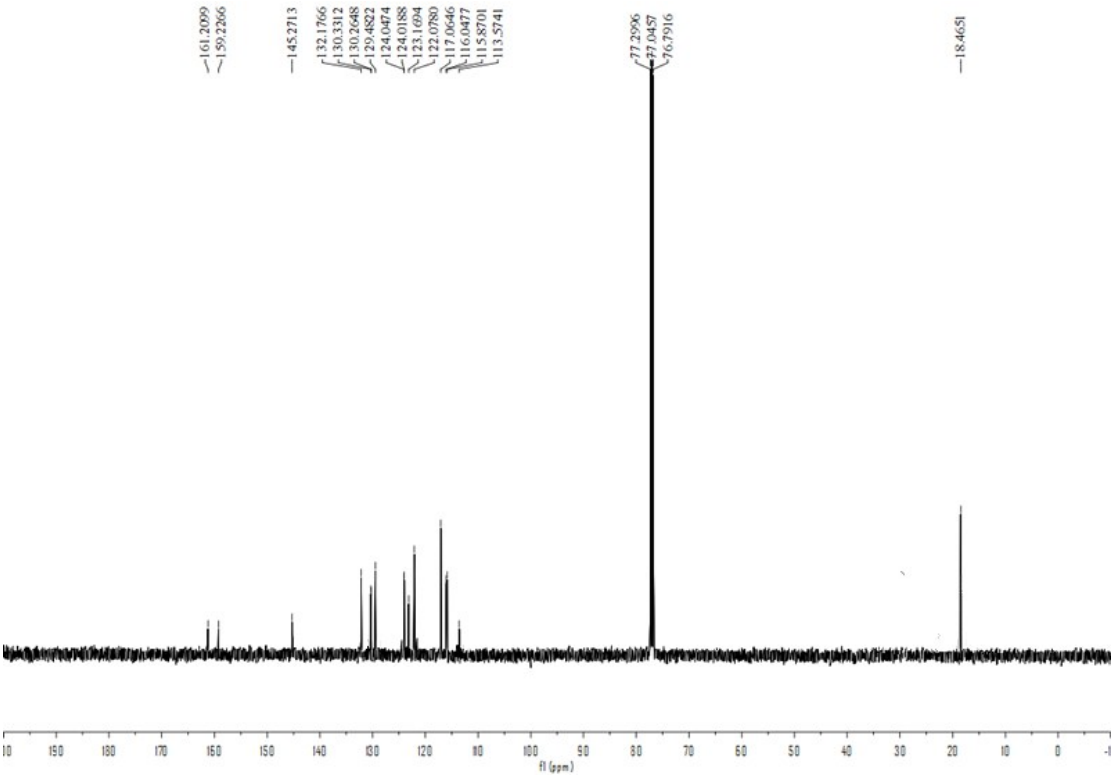

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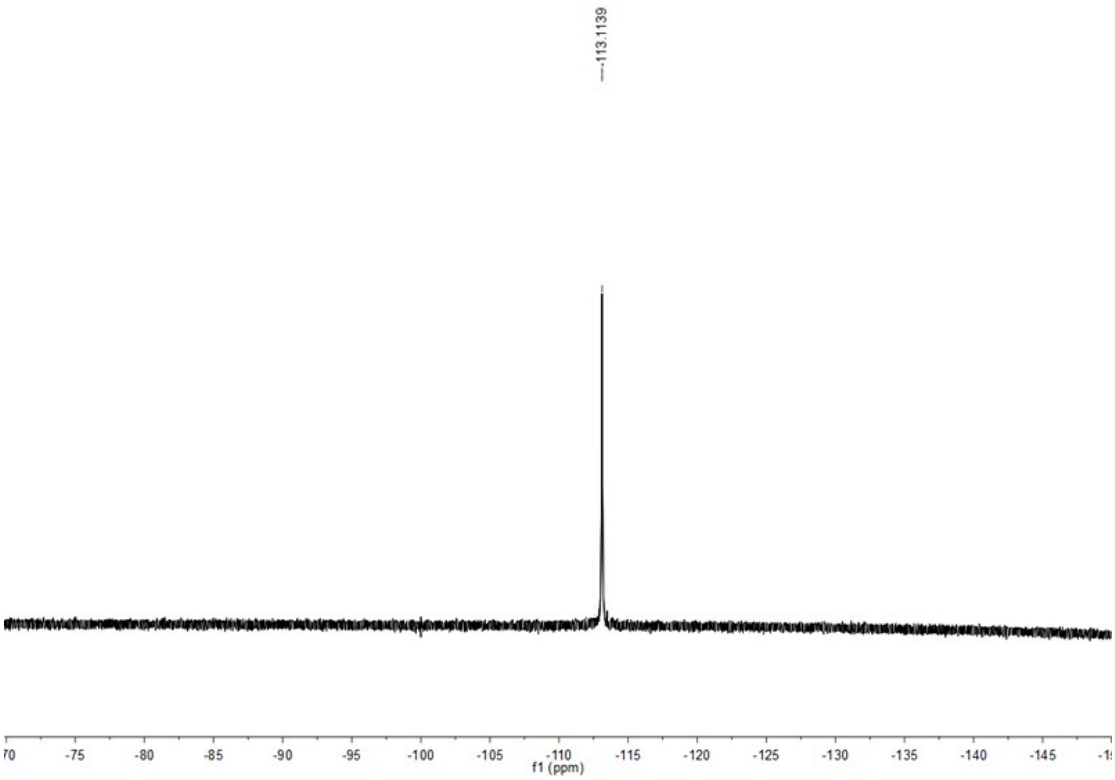
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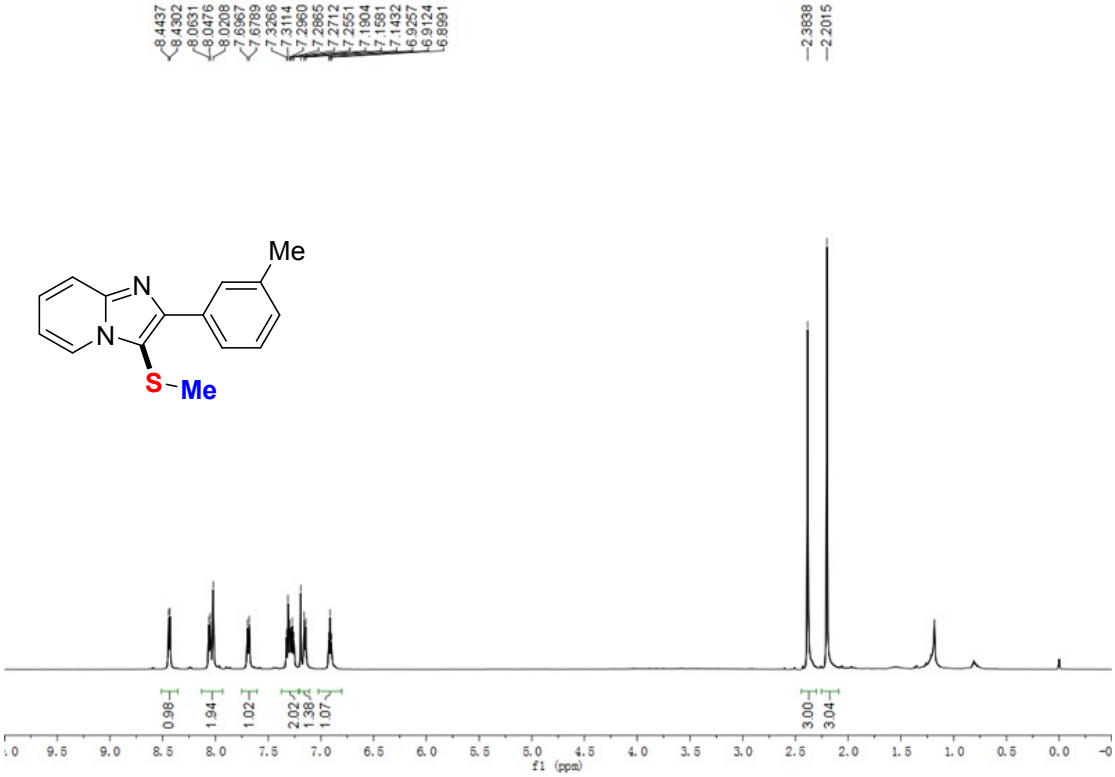
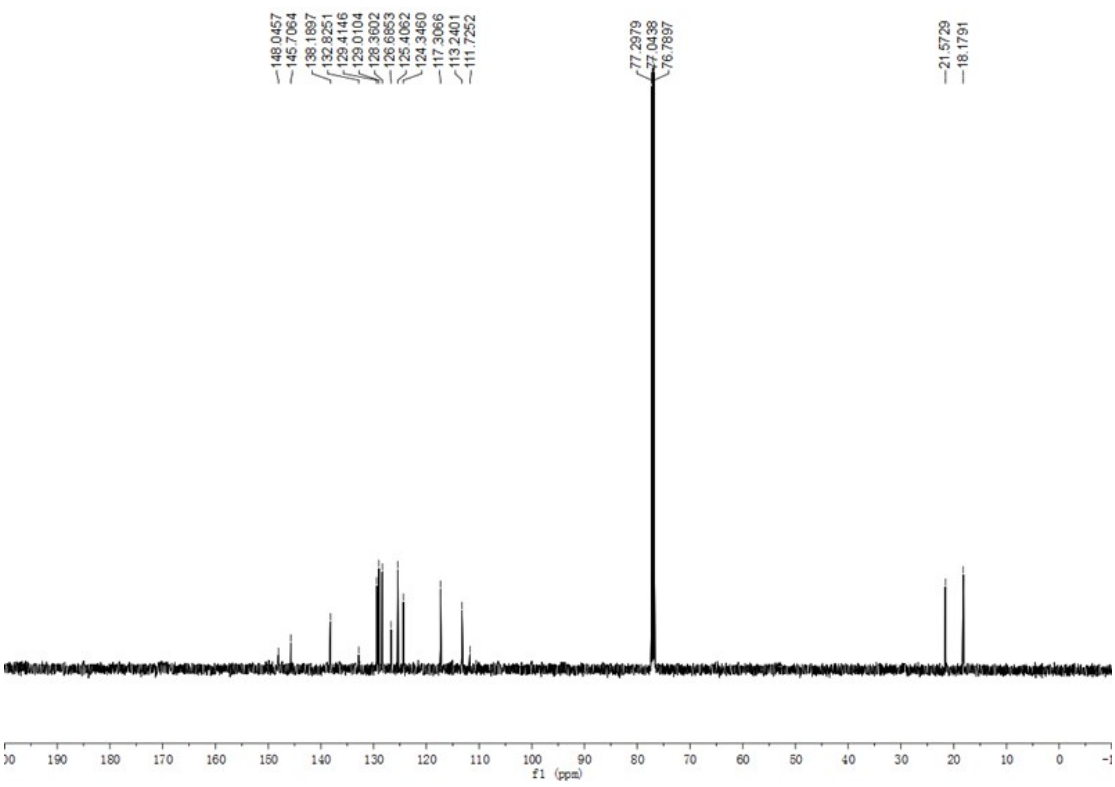
2o

[1]HNMR

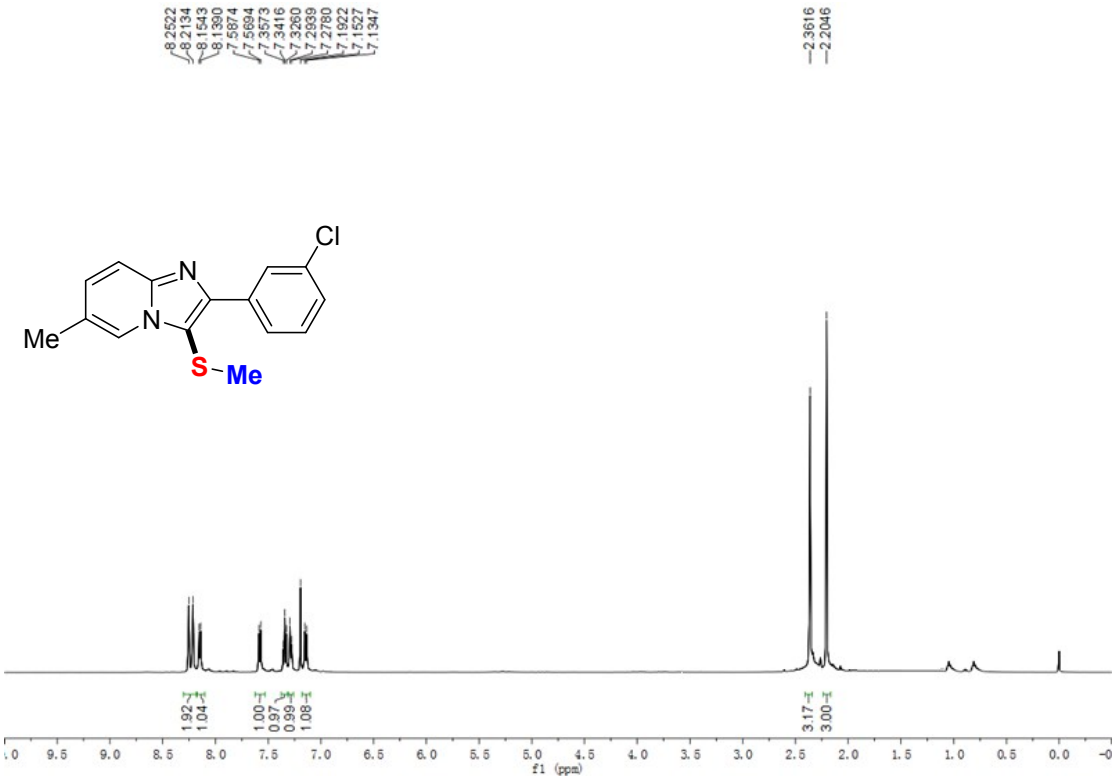


[13]CNMR

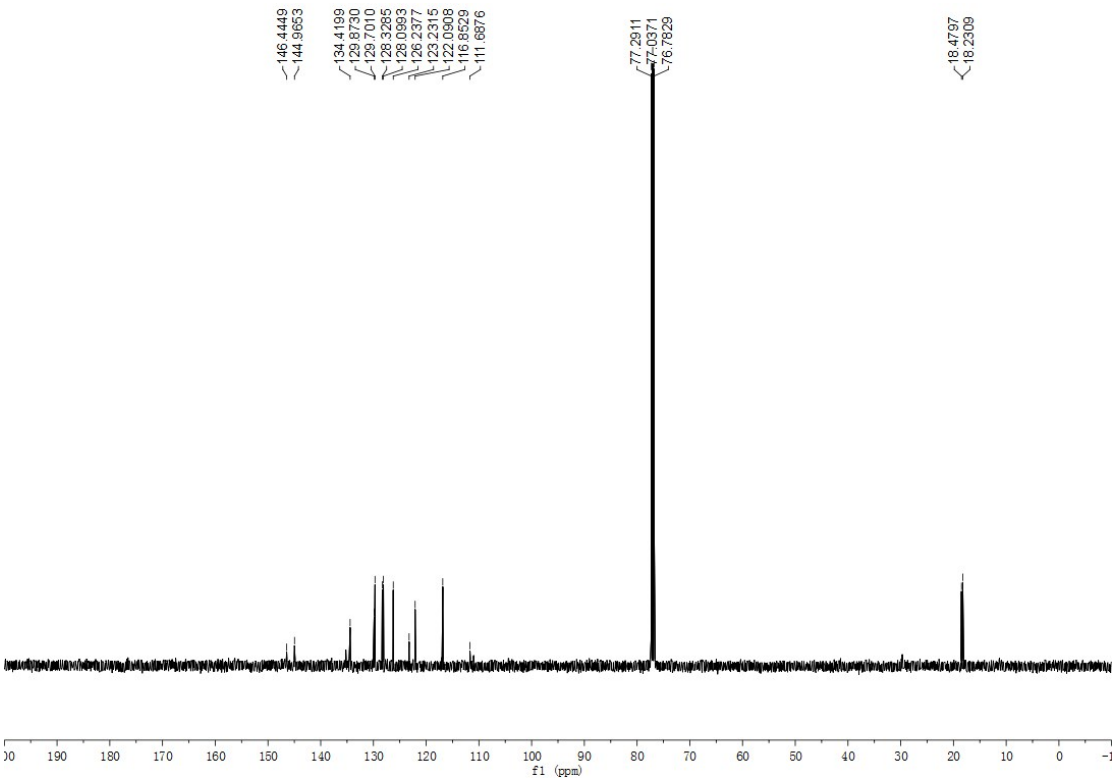


2p

[1]HNMR

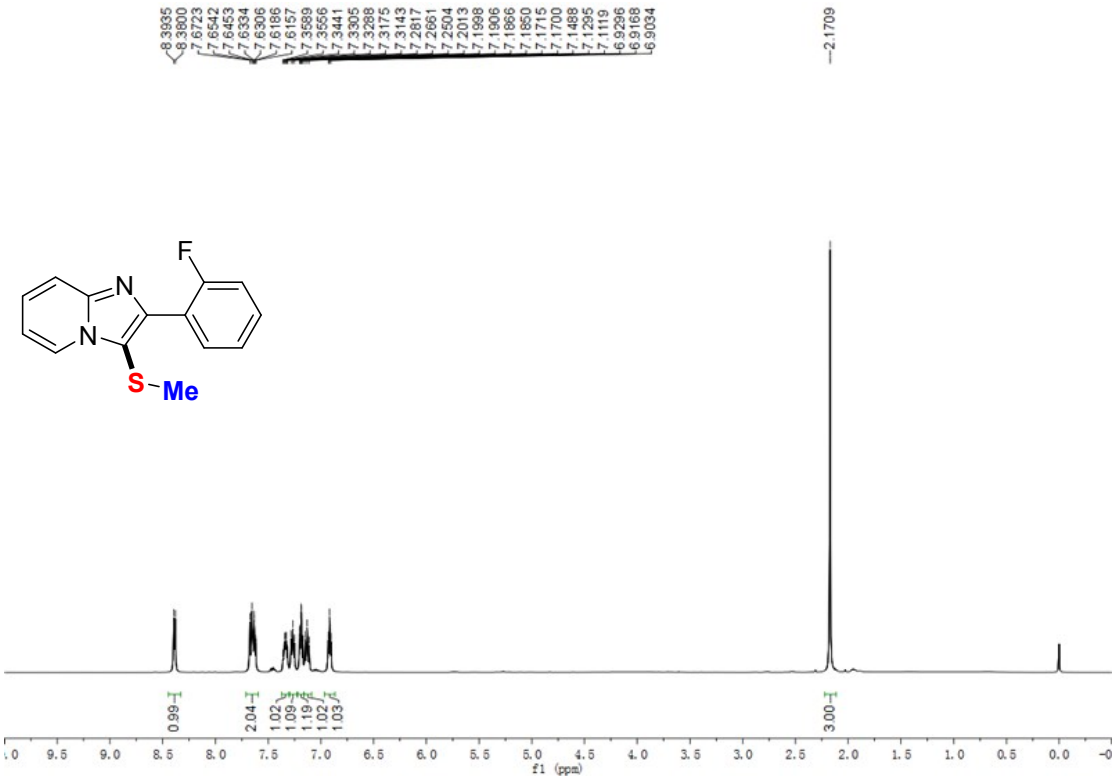


[13]CNMR



2q

[1]HNMR



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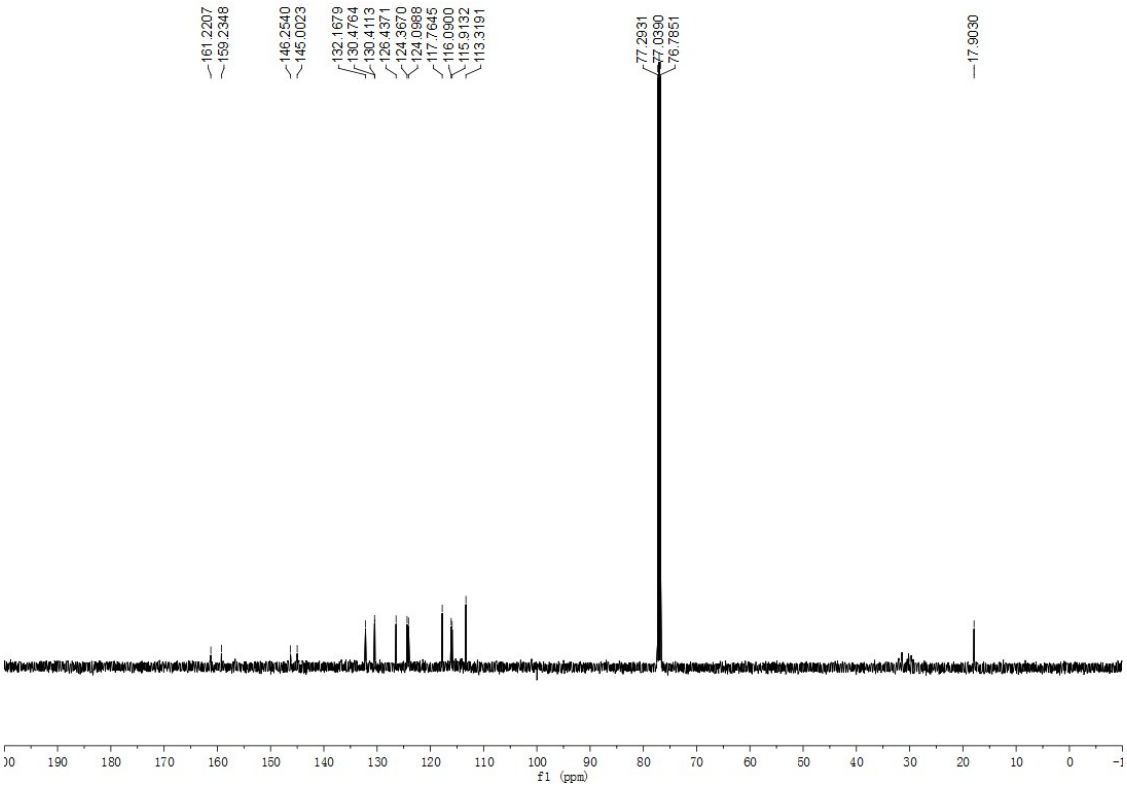
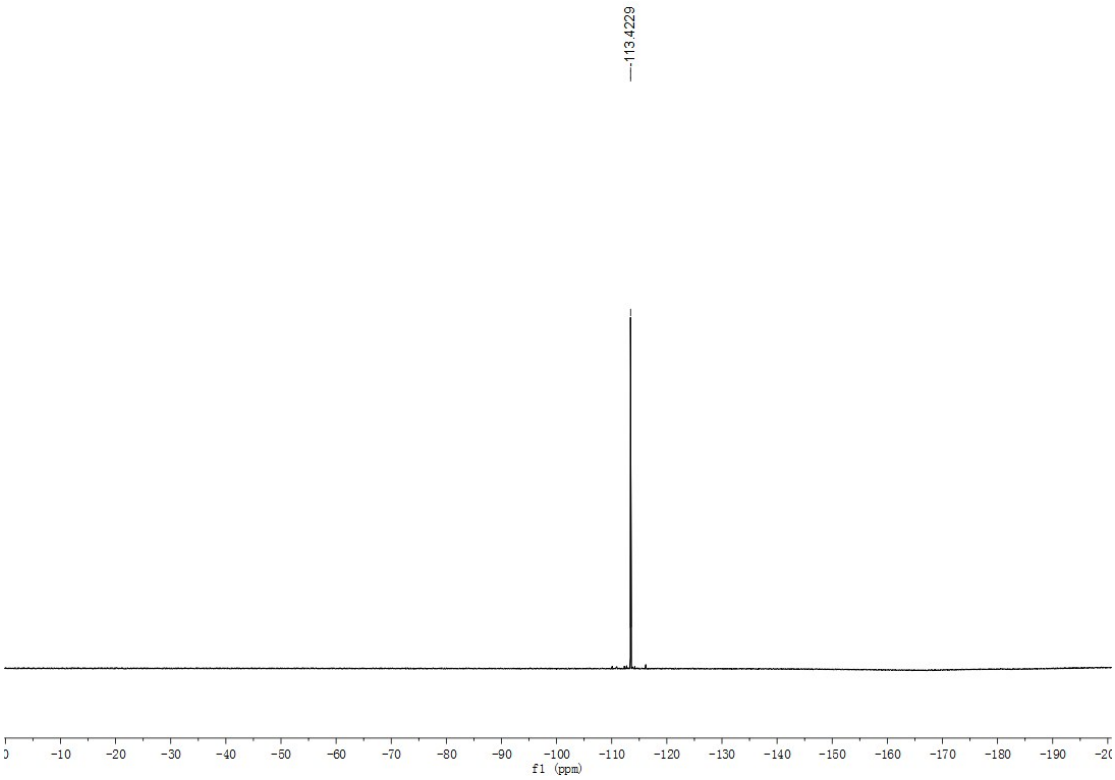


^{19}FNMR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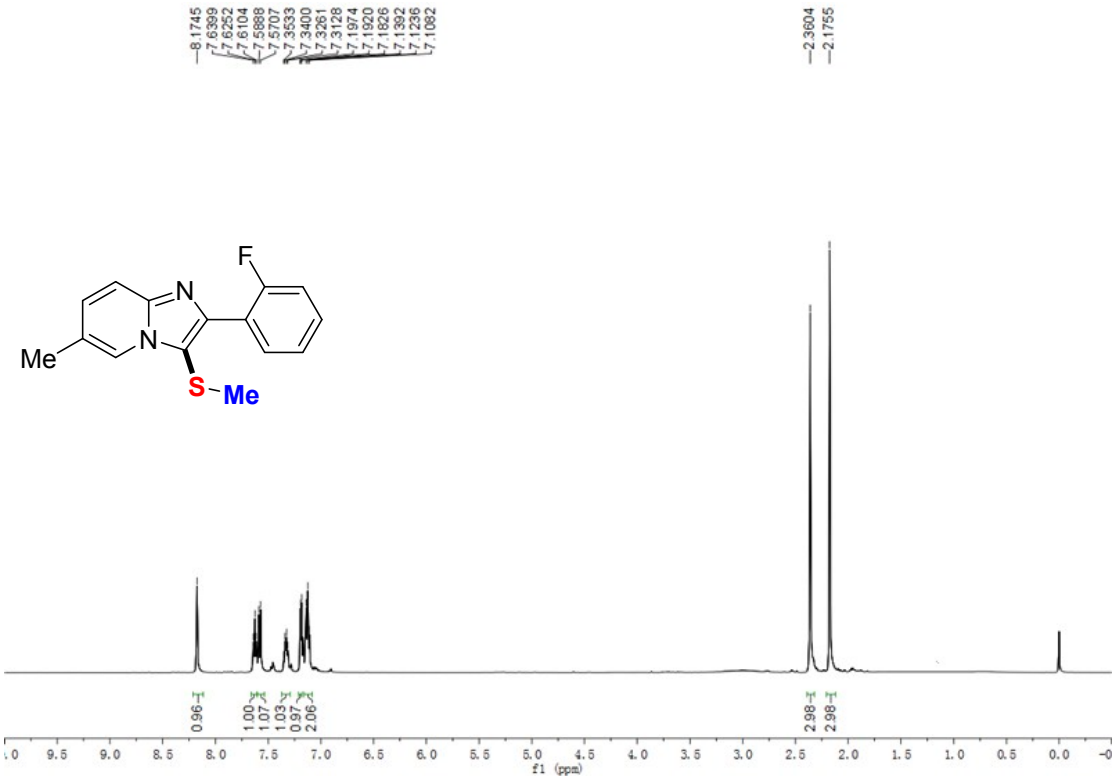
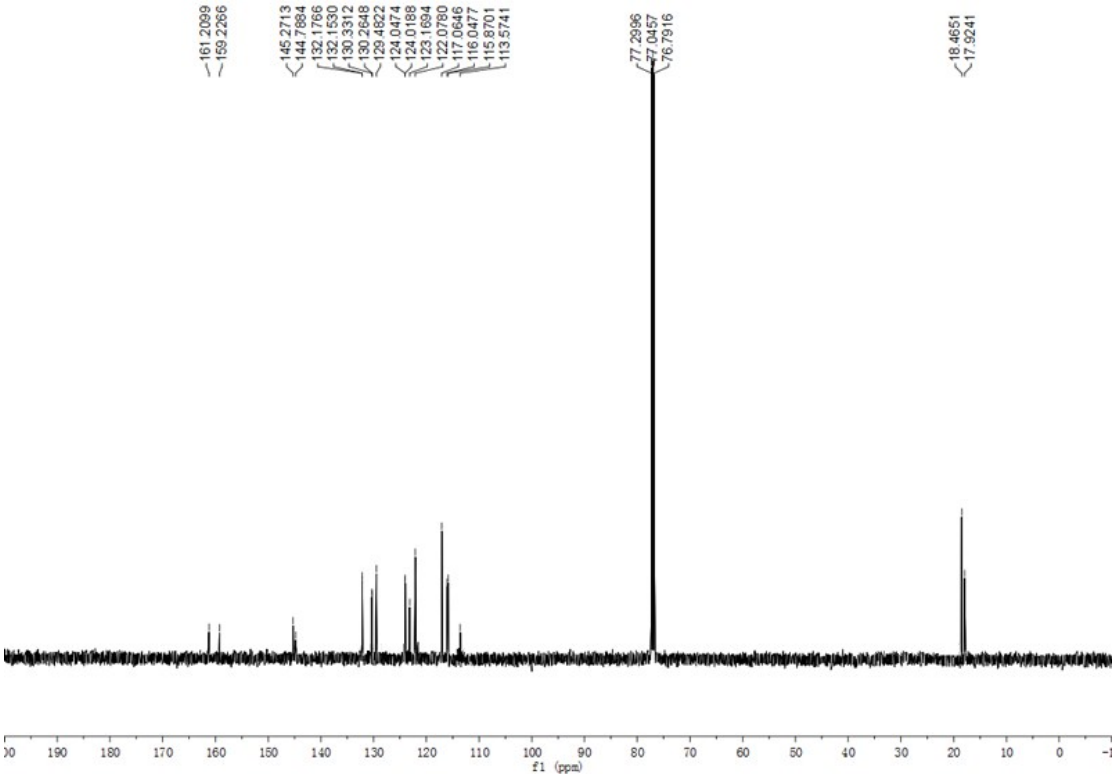
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

2r

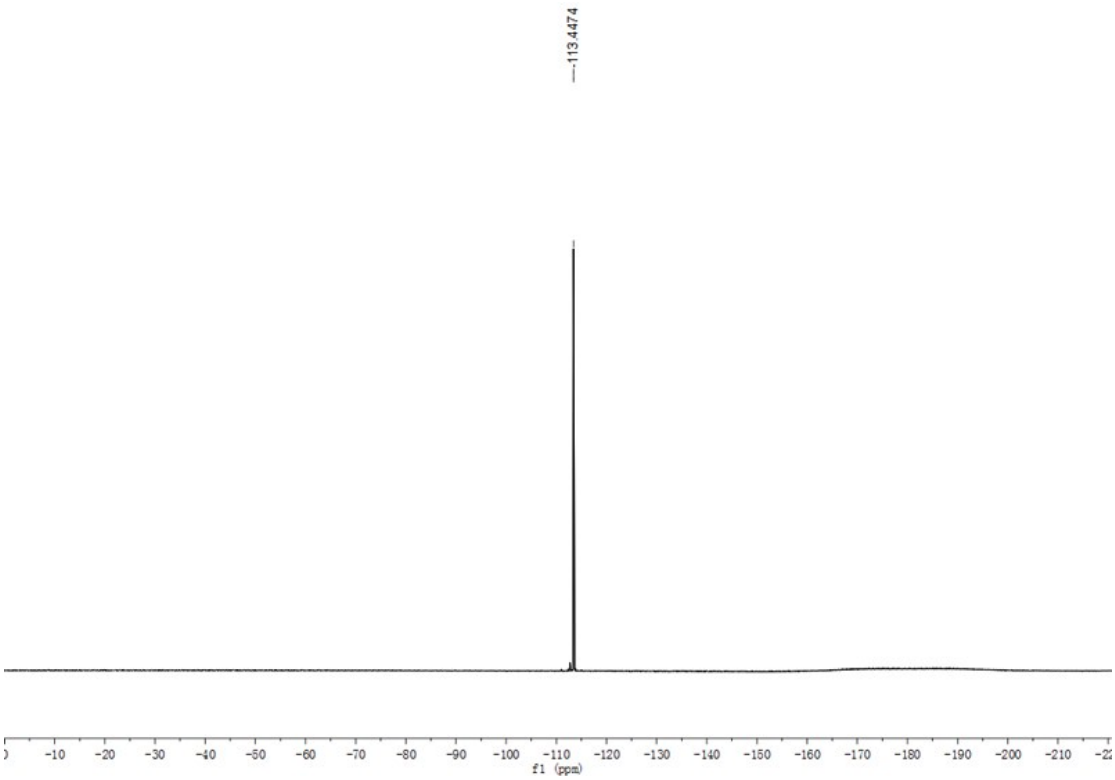
[1]HNMR



[13]CNMR



^{19}FNMR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-80
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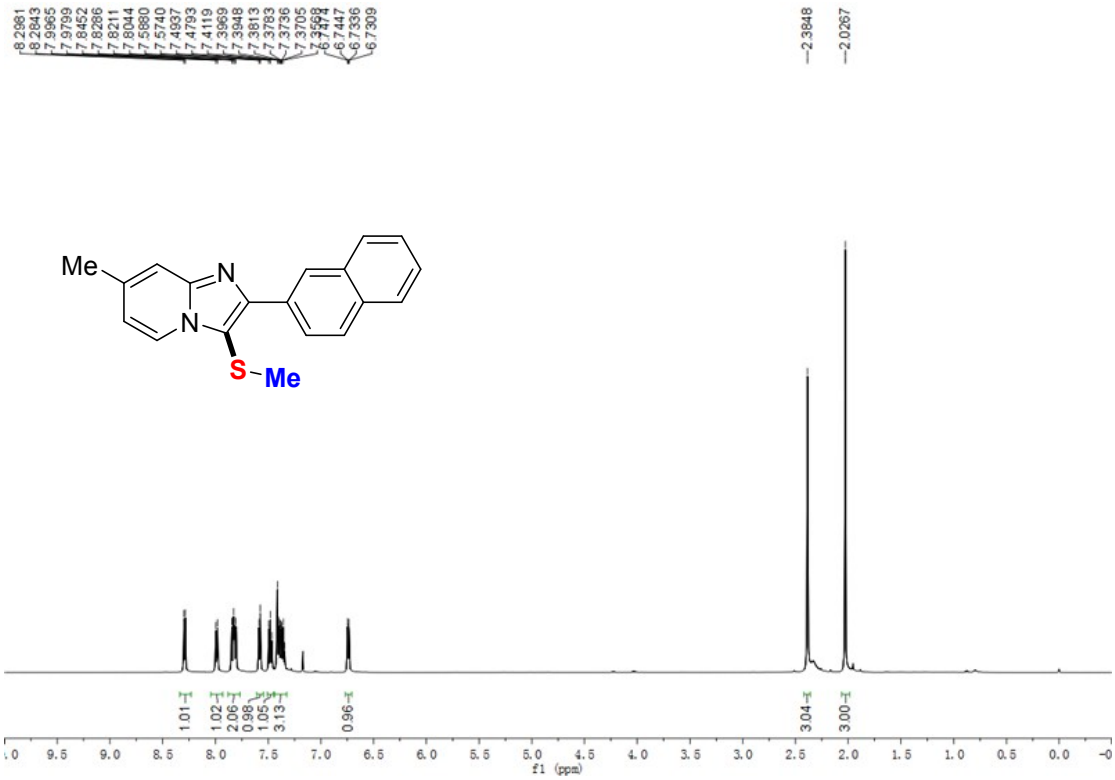
-190
-200
-210
f1 (ppm)

2s

[1]HNMR



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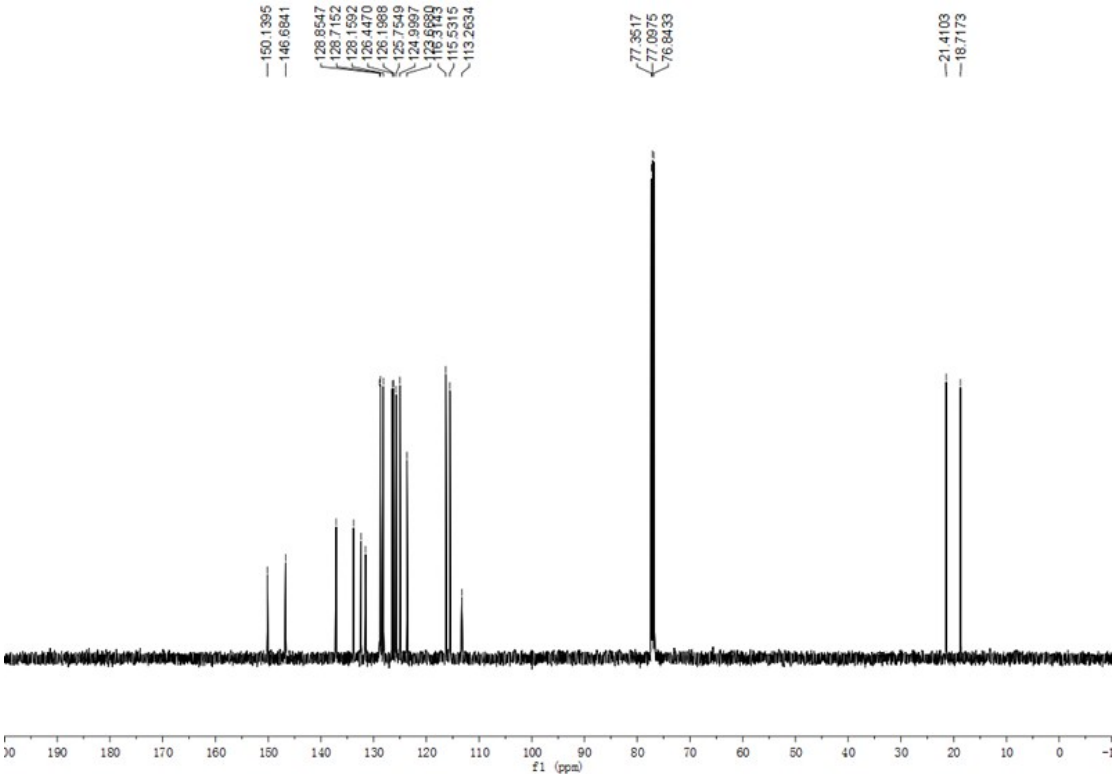
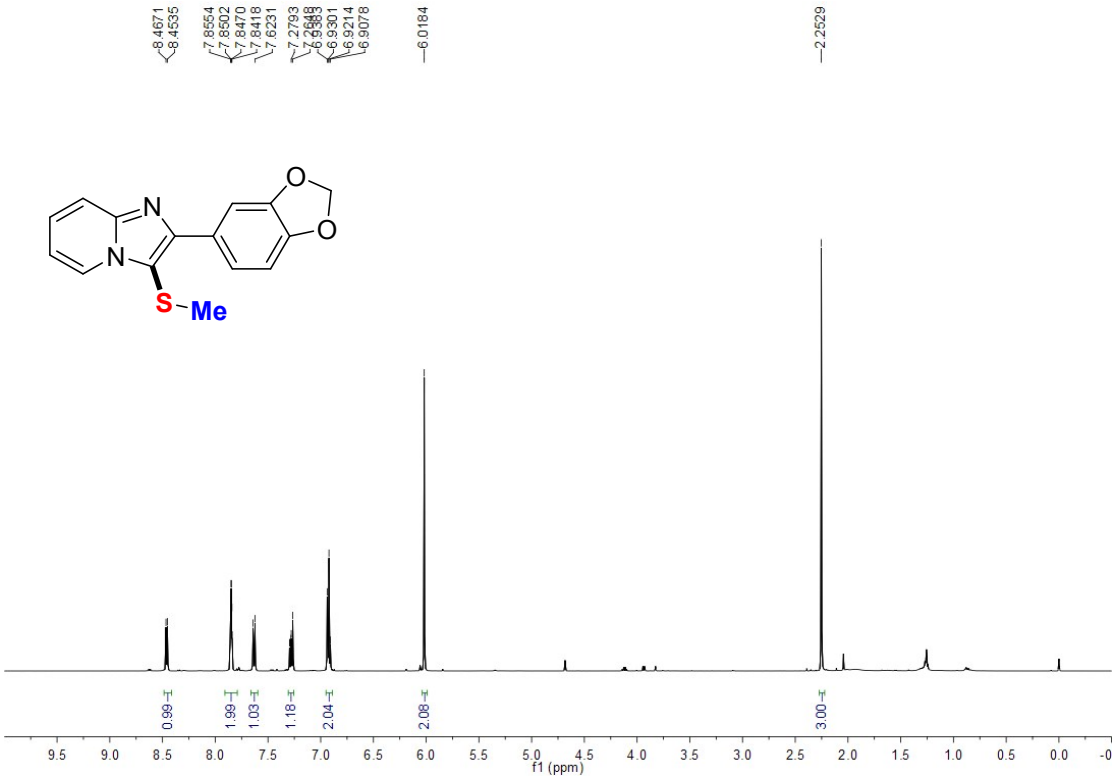


2t

[1]HNMR



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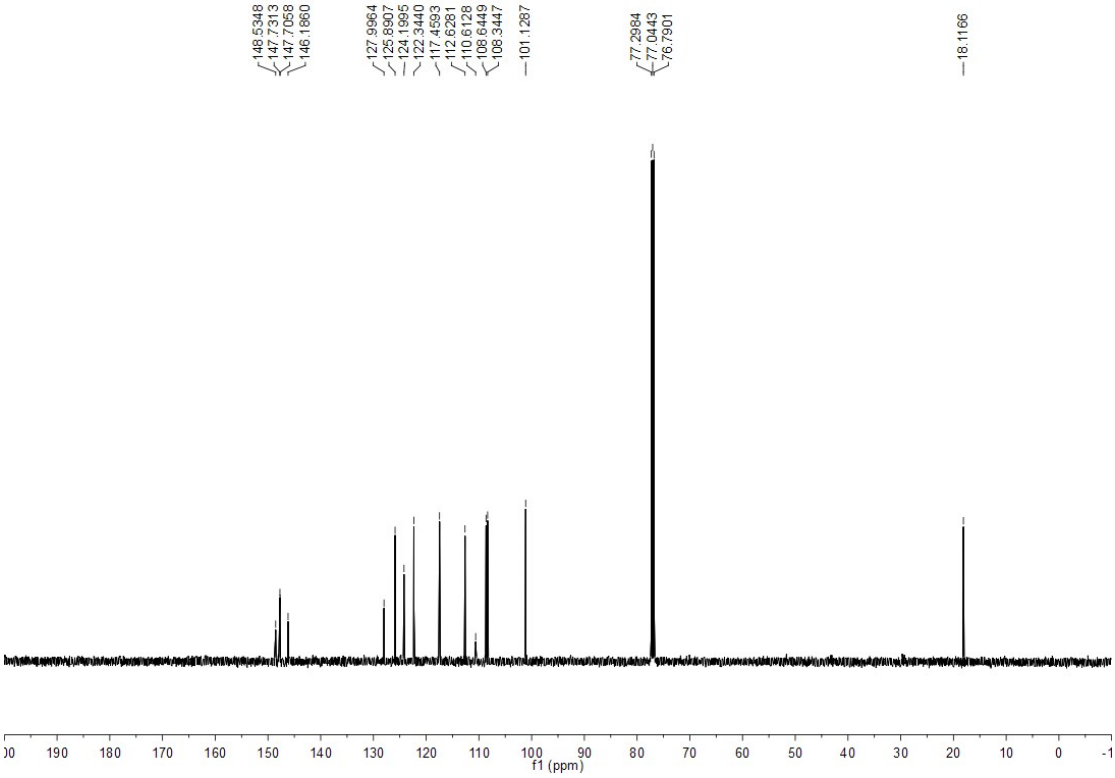
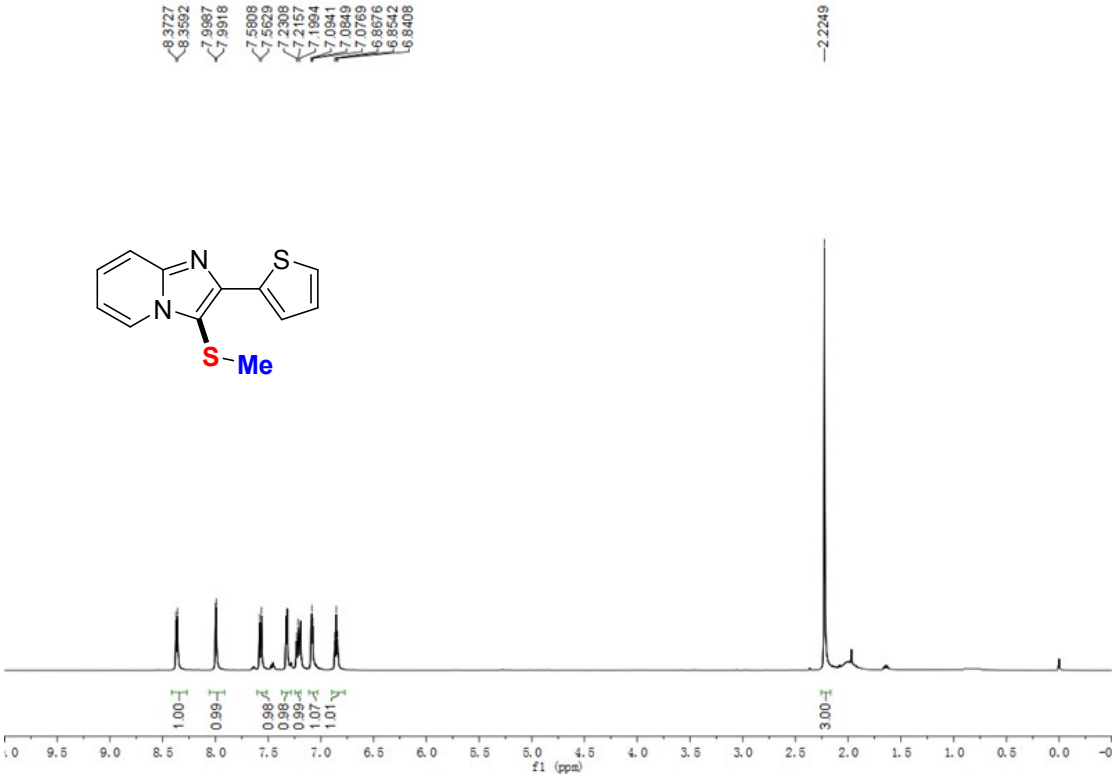
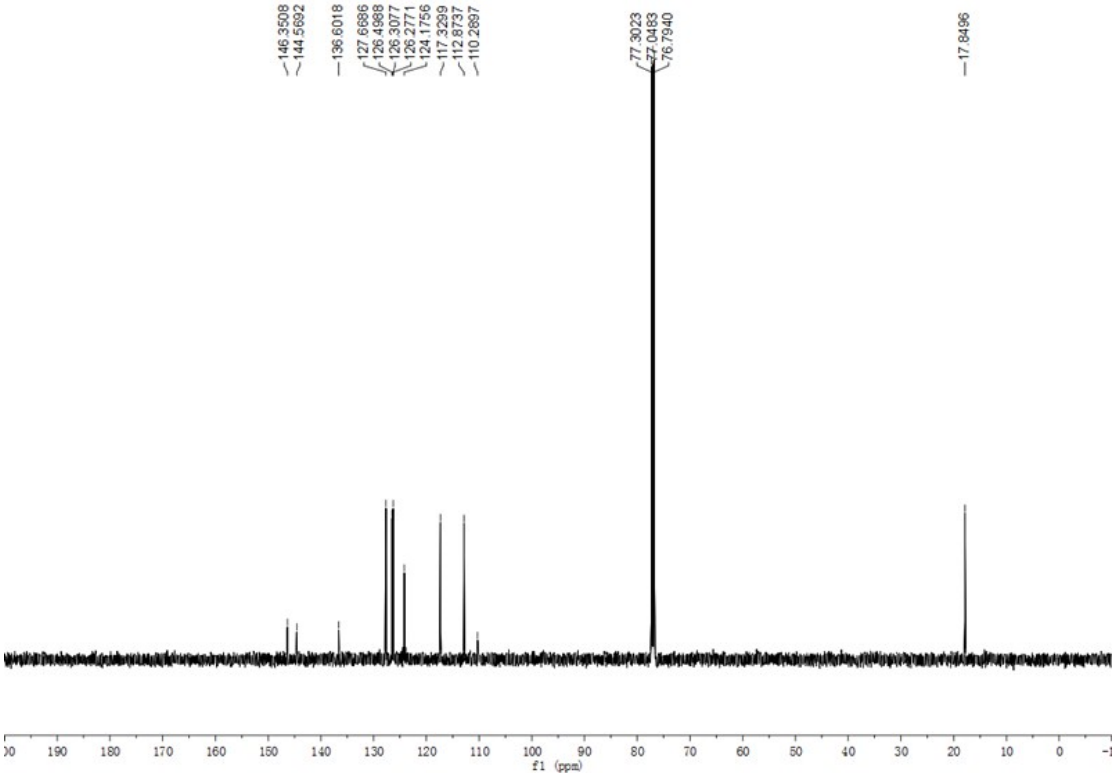


2u

[1]HNMR

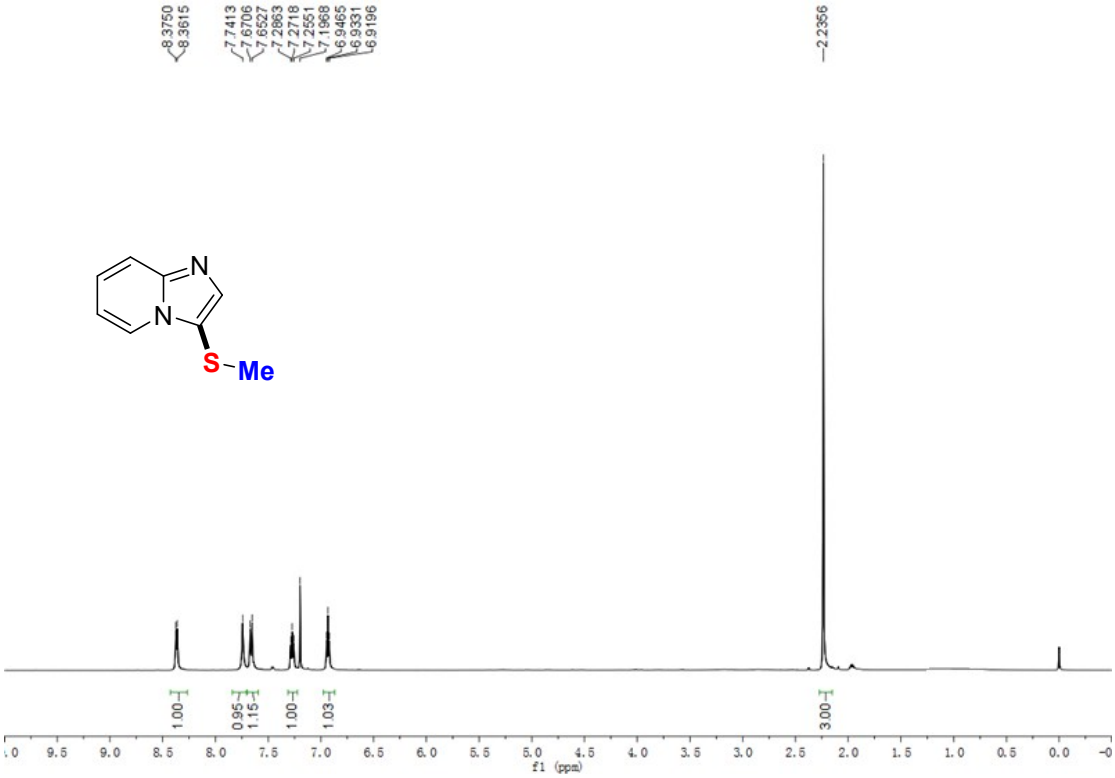


[13]CNMR



2v

[1]HNMR



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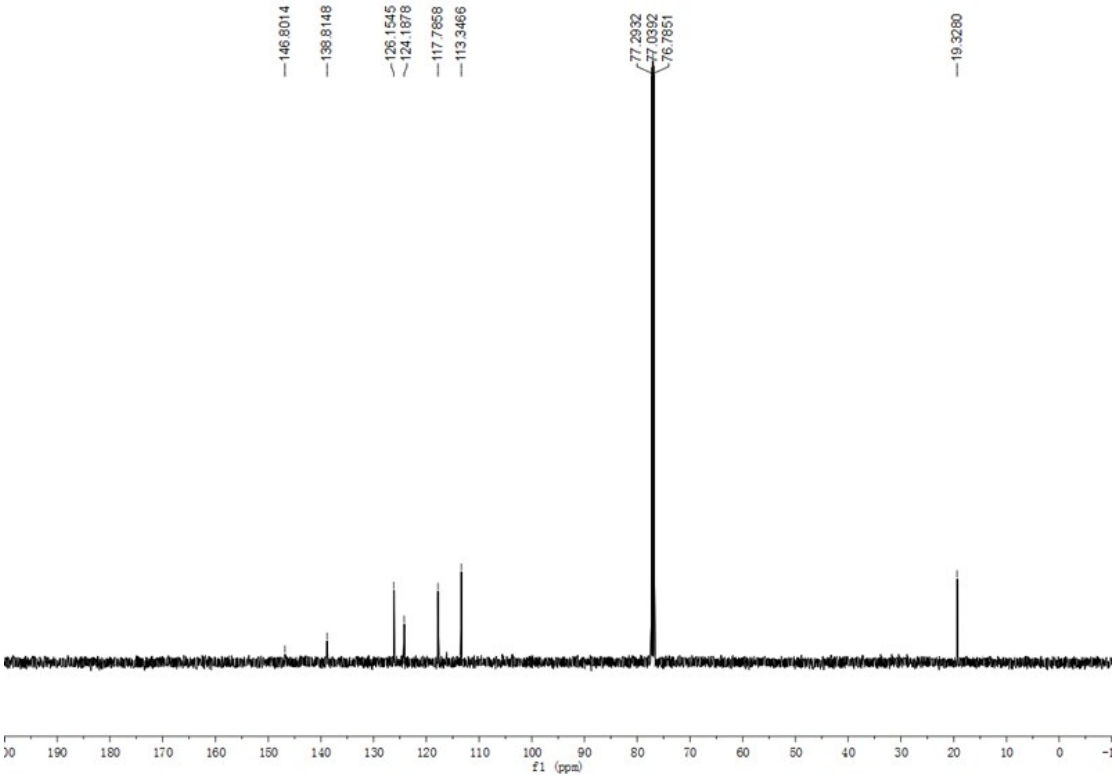

2w

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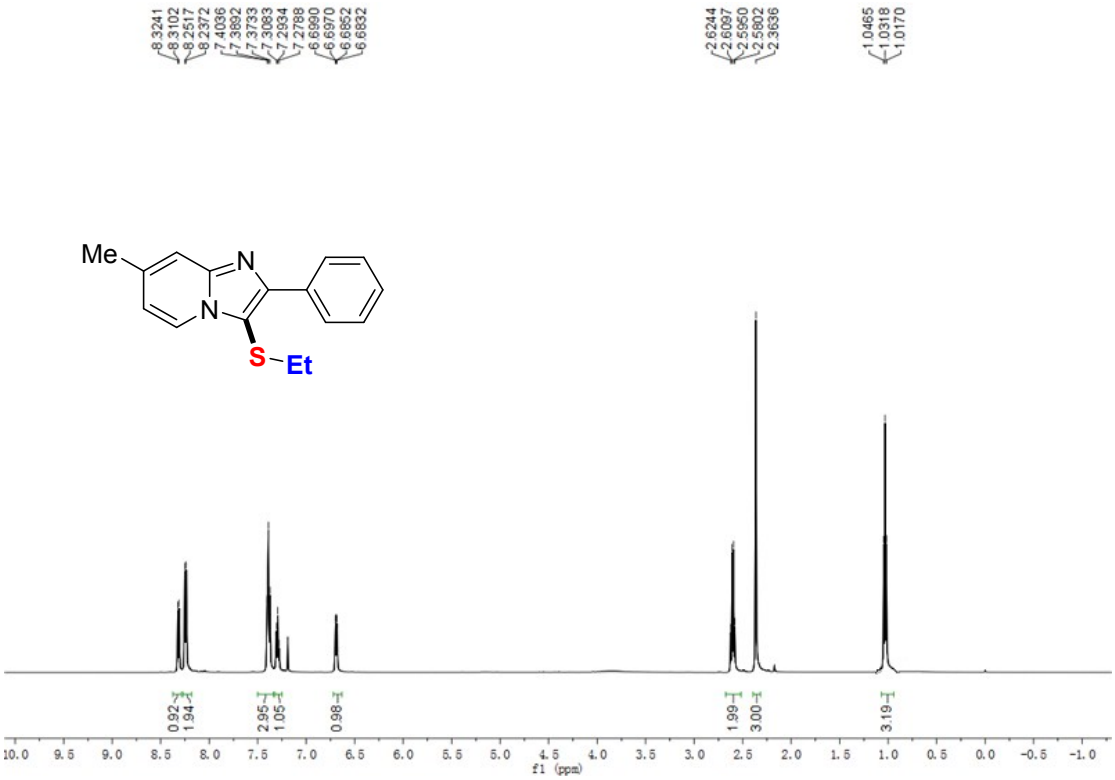

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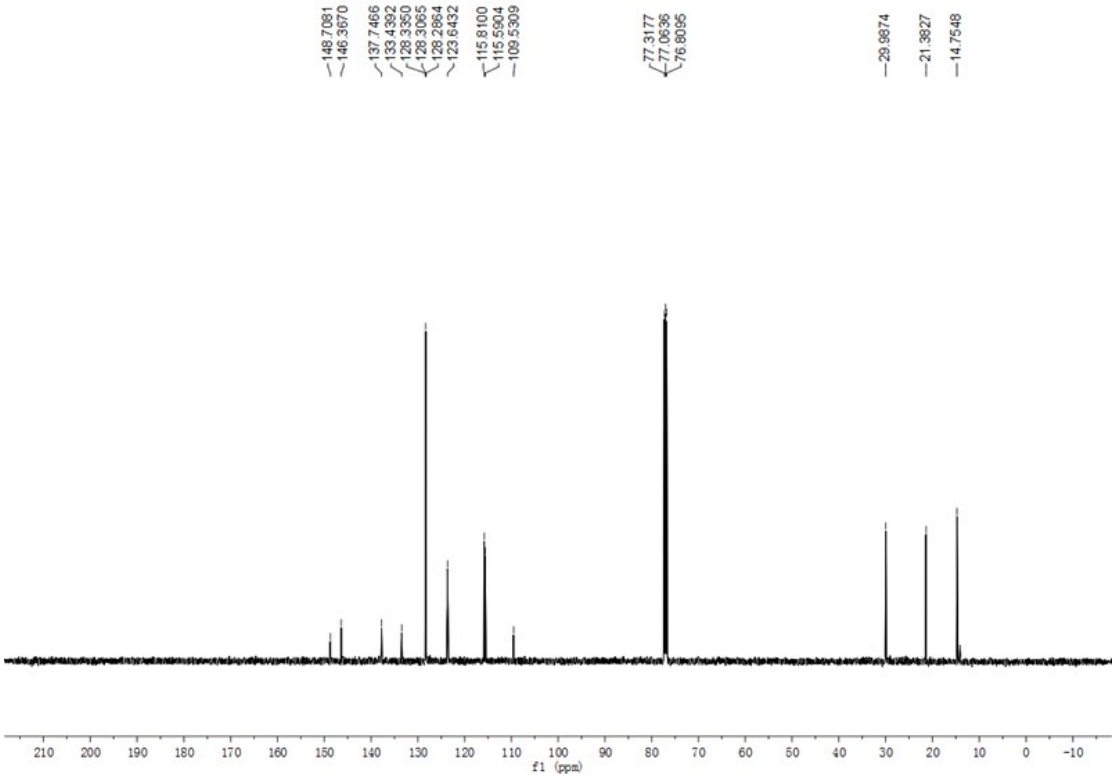
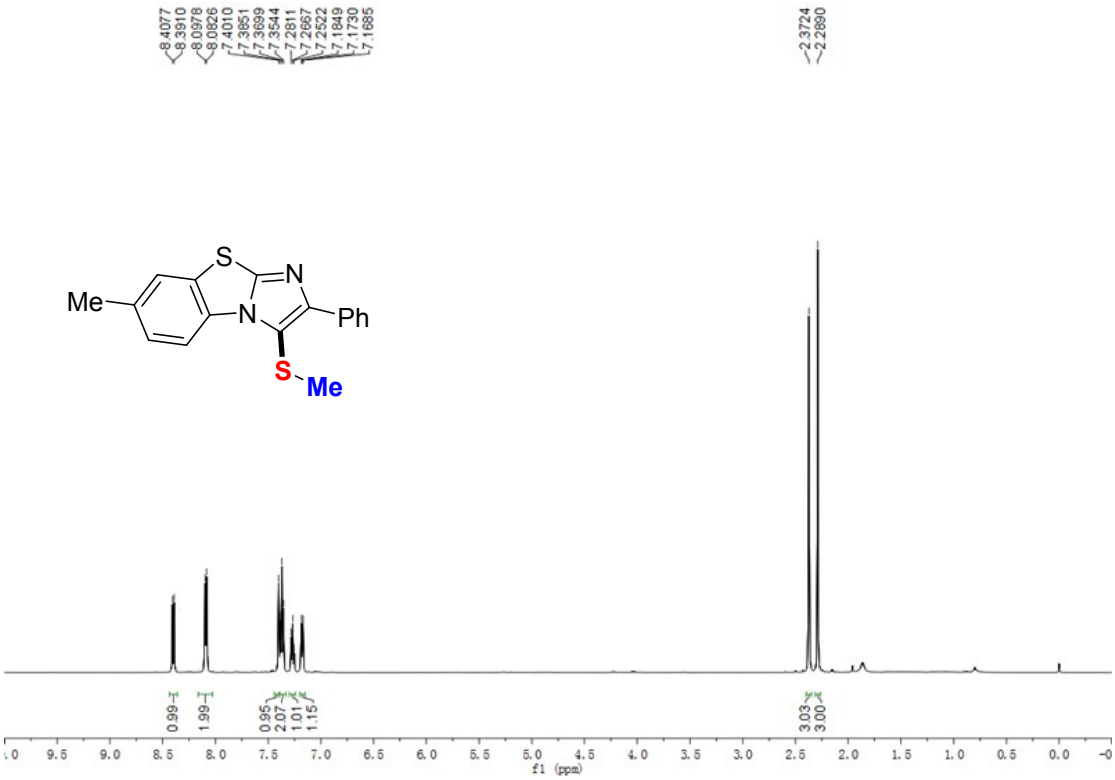
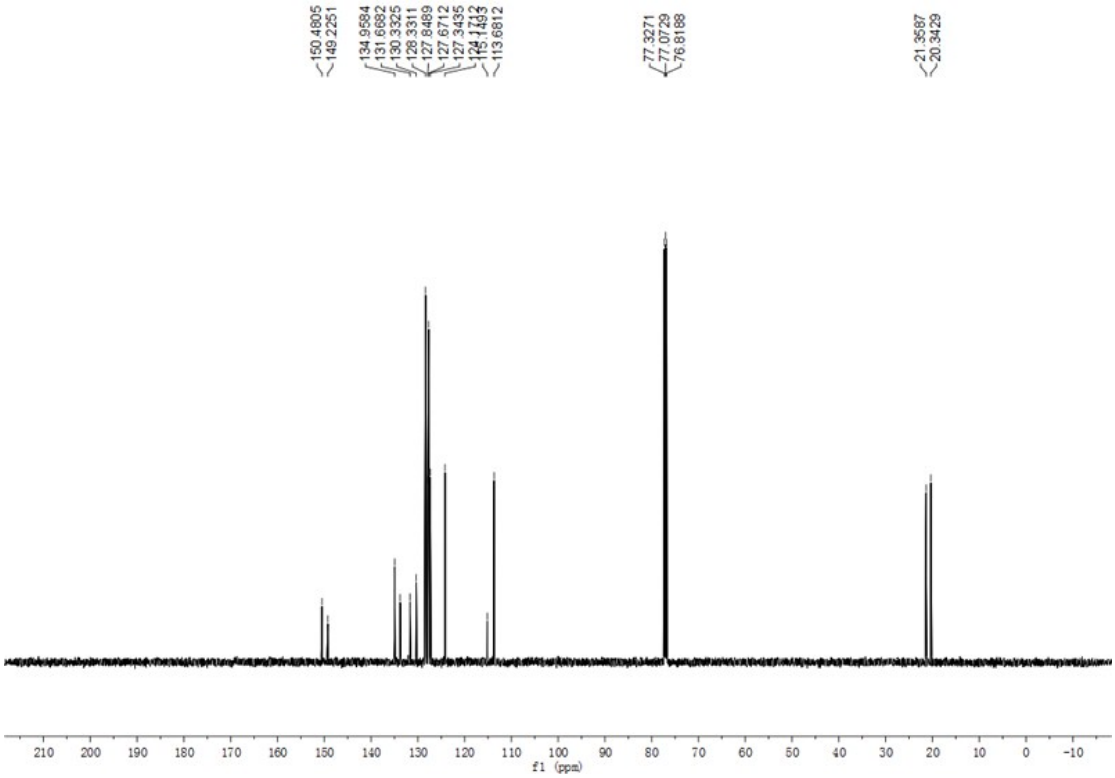


4a

[1]HNMR

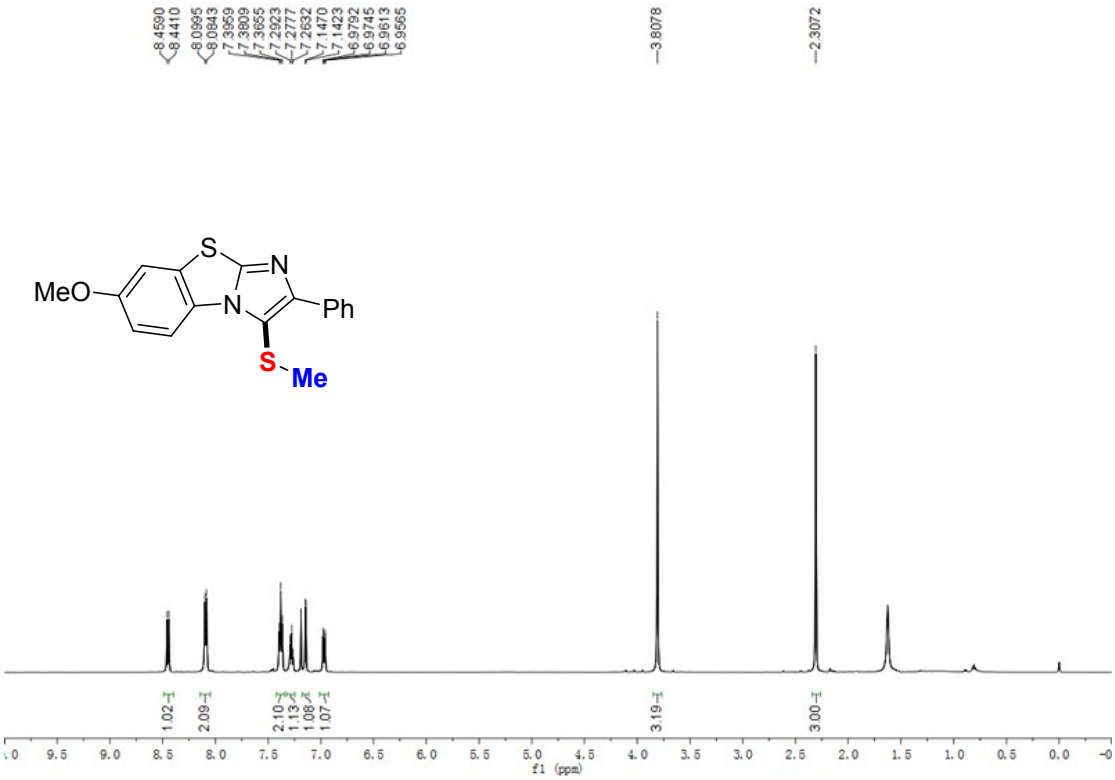


[13]CNMR



4b

[1]HNMR



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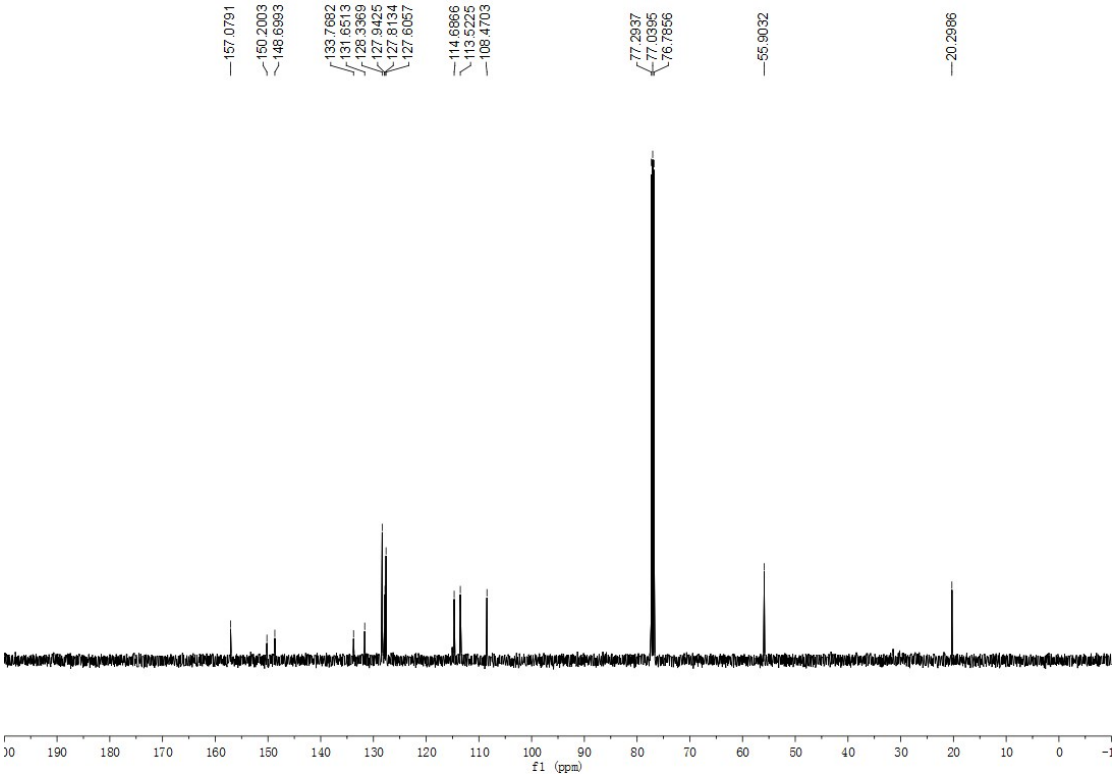
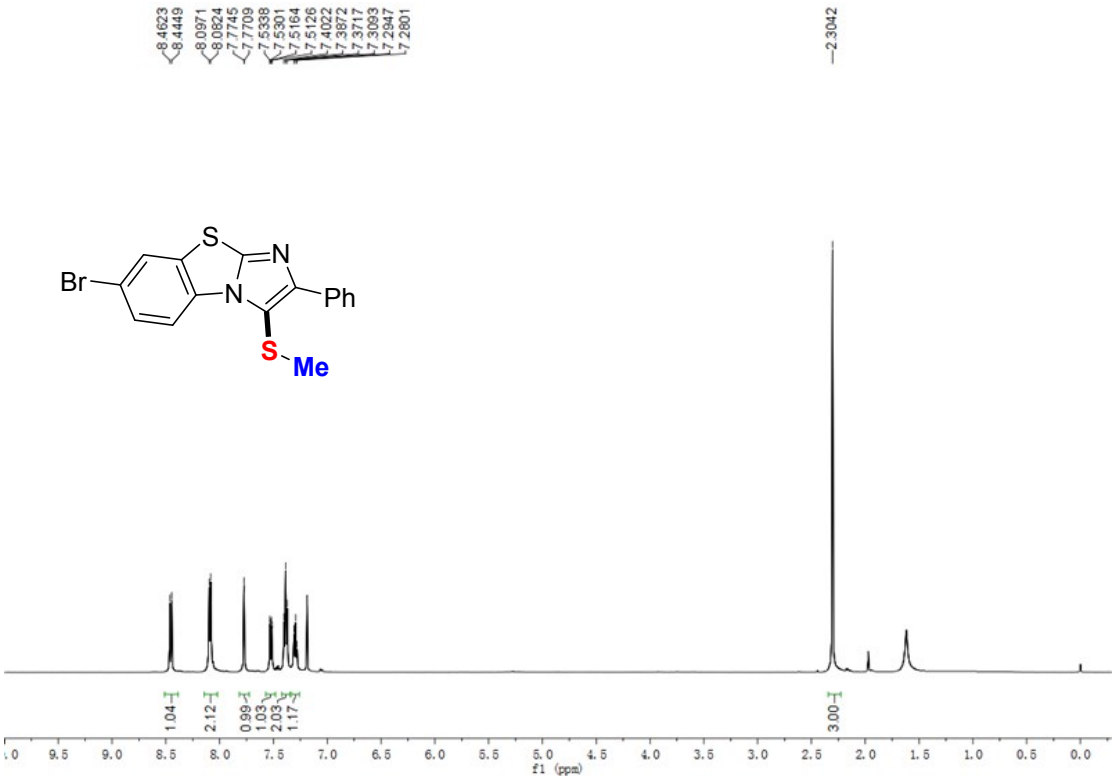


4c

[1]HNMR



[13]CNMR