Supporting Information

Metal-free cascade reactions of aziridines with arylalkynes and aryldiazoniums: facial access to arylazopyrrolines

Jing Rong, Hao Jiang, Sijing Wang, Zhenni Su, Huiyan Wang and Chuanzhou Tao*

School of Chemical Engineering, Jiangsu Ocean University, Lianyungang 222000, P. R. China

E-mail: taocz@jou.edu.cn

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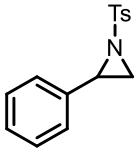
1. Preparation of aziridines[1]

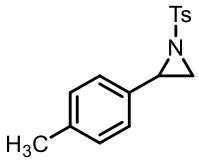
To a mixture of an olefin (3 mmol) and $TsNClNa \cdot 3H_2O$ (0.93 g, 3.3 mmol) in MeCN (15 mL), was added phenyltrimethyl-ammonium tribromide (PTAB) (0.113 g, 0.3 mmol) at room temperature. After vigorous stirring for 12 h, the reaction mixture was diluted with ethyl acetate (30 mL) and water (20 mL). The organic layer was separated, washed with brine, and dried over anhydrous sodium sulfate. After evaporation of solvent, the resultant solid was purified by flash column chromatography to yield the corresponding aziridine.

2-Phenyl-1-tosylaziridine (1a):[2]



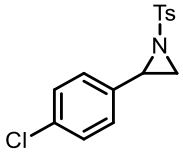
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.9 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.30 – 7.24 (m, 3H), 7.23-7.18 (m, 2H), 3.77 (dd, *J* = 7.0, 4.6 Hz, 1H), 2.98 (d, *J* = 7.2 Hz, 1H), 2.42 (s, 3H), 2.38 (d, *J* = 4.4 Hz, 1H).

2-(p-Tolyl)-1-tosylaziridine (1b):[3]



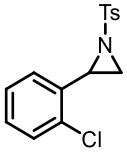
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.09 (s, 4H), 3.74 (dd, *J* = 7.2, 4.5 Hz, 1H), 2.96 (d, *J* = 7.2 Hz, 1H), 2.42 (s, 3H), 2.37 (d, *J* = 4.5 Hz, 1H), 2.30 (s, 3H).

2-(4-Chlorophenyl)-1-tosylaziridine (1c):[2]



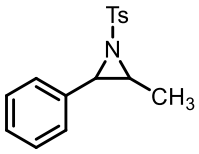
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89 – 7.83 (m, 2H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.28 – 7.25 (m, 2H), 7.18 – 7.13 (m, 2H), 3.73 (dd, *J* = 7.1, 4.4 Hz, 1H), 2.98 (d, *J* = 7.2 Hz, 1H), 2.44 (s, 3H), 2.34 (d, *J* = 4.4 Hz, 1H).

2-(2-Chlorophenyl)-1-tosylaziridine (1d):[3]



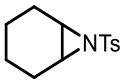
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 – 7.87 (m, 2H), 7.38 – 7.31 (m, 3H), 7.23 – 7.15 (m, 3H), 4.04 (dd, *J* = 7.2, 4.4 Hz, 1H), 3.03 (d, *J* = 7.3 Hz, 1H), 2.45 (s, 3H), 2.29 (d, *J* = 4.4 Hz, 1H).

***(trans)*-2-Methyl-3-phenyl-1-tosylaziridine (1e):**[2]



^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 – 7.79 (m, 2H), 7.28 – 7.22 (m, 5H), 7.17 – 7.11 (m, 2H), 3.79 (d, *J* = 4.3 Hz, 1H), 2.91 (qd, *J* = 6.0, 4.4 Hz, 1H), 2.38 (s, 3H), 1.84 (d, *J* = 6.0 Hz, 3H).

7-tosyl-7-azabicyclo [4.1.0] heptane (1f):[2]



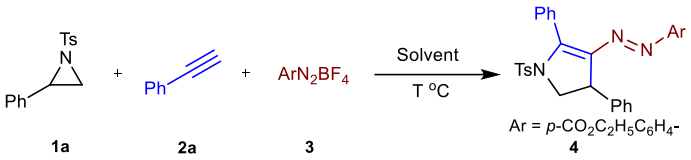
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.2 Hz, 2H), 7.32 (d, *J* = 8.1 Hz, 2H), 2.96 (s, 2H), 2.43 (s, 3H), 1.78 (t, *J* = 5.8 Hz, 4H), 1.45 – 1.34 (m, 2H), 1.25 – 1.17 (m, 2H).

2. Preparation of aryldiazonium tetrafluoroborates[4]

Arylamine (50 mmol) was dissolved in a mixture of hydrofluoroboric acid (48 wt.%, 17 mL) and distilled water (20 mL). After cooling the reaction mixture to 0°C, the solution of sodium nitrite (3.4 g in 7.5 mL distilled water) was added dropwise into the reaction system (5 min interval of time). The resulting mixture was stirred for 1 h and the precipitate was collected by filtration and redissolved in minimum amount of acetone. Diethyl ether was added until precipitation of aryldiazonium tetrafluoroborate, which is filtered, washed several times with diethyl ether and dried under vacuum.

3. Supplementary notes on the optimization of conditions for the synthesizing arylazopyrrolines[a]

To an oven-dried 25 mL Schlenk tube equipped with a magnetic stir bar, aziridine (0.2 mmol, 1.0 equiv.), alkyne (0.5 mmol, 2.5 equiv., if the alkyne is solid) and aryl diazonium tetrafluoroborate (0.3 mmol, 1.5 equiv.) were added. The Schlenk tube was evacuated and backfilled with argon for three times. Under argon atmosphere, alkyne (0.5 mmol, 2.5 equiv., if the alkyne is liquid) and solvent (2.0 mL, anhydrous and degassed) were added into the tube and the mixture was stirred for the given time. After cooling to room temperature, Et_3N (0.5 mL) was used to quench the reaction. The volatiles were evaporated under vacuum and the residue was purified with column chromatography on silica gel using ethyl acetate/petroleum ether as the eluent to give azo products. All the silica gel was dealt with 5% Et_3N (the solution in petroleum ether) overnight.



Entry[a]	Substrate	Solvent	T (°C)	t (h)	Yield[b] (%)
1	*p*-$CO_2EtC_6H_4N_2BF_4$	DCM	40	48	60
2	*p*-$ClC_6H_4N_2BF_4$	DCM	40	48	48
3	*p*-$ClC_6H_4N_2BF_4$	DCM	40	60	65
4	*p*-$CO_2EtC_6H_4N_2BF_4$	DCE	60	24	50

[a] Reaction conditions: **1a** 0.2 mmol, **2a** 0.5 mmol, **3** 0.3 mmol, Solvent 2.0 mL. [b] The yields are isolated yields with flash chromatography.

4. X-ray crystal structure and crystal data

4.1 X-ray crystal structure and crystal data of 4aaa (CCDC: 1975117)

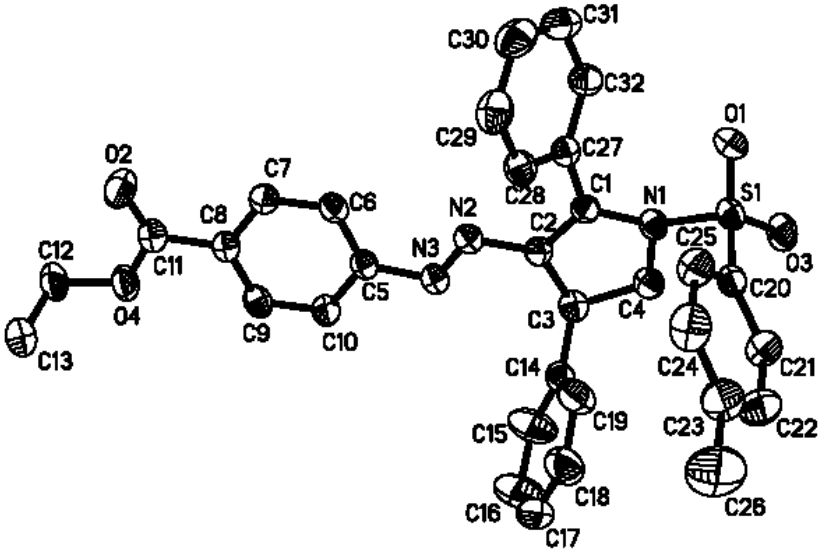


Figure 1.X-Ray crystal structure of 4aaa

Table 1-1 Crystal data and structure refinement for 4aaa

Crystal data	Compound **4aaa**
Identification code	rj_0m
Empirical formula	$C_{32}H_{29}N_3O_4S$
Formula weight	551.64
Temperature	296(2) K
Wavelength	71.073 pm
Crystal system, space group	Monoclinic, $P2_1/c$
Unit cell dimensions	a = 1481.8(6) pm alpha= 90° b = 1192.0(5) pm beta= 91.828(7)°

	c = 1655.7(7) pm gamma= 90°
Volume	2.923(2) nm^3
Z, Calculated density	4, 1.254 Mg/m^3
Absorption coefficient	0.151 mm^{-1}
F(000)	1160
Theta range for data collection	4.212 to 55.792°.
Limiting indices	-18<=h<=19, -15<=k<=14, -21<=l<=19
Reflections collected / unique	18161, 6834 [R(int) = 0.0469]
Completeness to theta = 25.242°	99.5 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6834 / 0 / 363
Goodness-of-fit on F^2	0.987
Final R indices [I>2sigma(I)]	R1 = 0.0496, wR2 = 0.1137
R indices (all data)	R1 = 0.1153, wR2 = 0.1434
Largest diff. peak and hole	0.167 and -0.345 $e.Å^{-3}$

Table 1-2 Bond lengths [pm] and angles [°] for 4aaa

_____

S(1)-O(1)	142.05(16)
S(1)-O(3)	142.82(16)
S(1)-N(1)	166.82(18)
S(1)-C(20)	175.4(2)
O(2)-C(11)	120.3(3)
O(4)-C(11)	133.0(3)
O(4)-C(12)	145.0(3)
N(1)-C(1)	140.2(2)
N(1)-C(4)	150.6(2)
N(2)-N(3)	127.7(2)
N(2)-C(2)	137.9(2)
N(3)-C(5)	142.9(3)
C(1)-C(2)	136.3(3)
C(1)-C(27)	148.2(3)
C(2)-C(3)	150.6(3)
C(3)-C(14)	152.3(3)
C(3)-C(4)	153.9(3)
C(3)-H(3)	98.00
C(4)-H(4A)	97.00
C(4)-H(4B)	97.00
C(5)-C(6)	138.3(3)
C(5)-C(10)	138.7(3)

C(6)-C(7)	137.5(3)
C(6)-H(6)	93.00
C(7)-C(8)	139.1(3)
C(7)-H(7)	93.00
C(8)-C(9)	138.6(3)
C(8)-C(11)	148.4(3)
C(9)-C(10)	139.0(3)
C(9)-H(9)	93.00
C(10)-H(10)	93.00
C(12)-C(13)	147.1(4)
C(12)-H(12A)	97.00
C(12)-H(12B)	97.00
C(13)-H(13A)	96.00
C(13)-H(13B)	96.00
C(13)-H(13C)	96.00
C(14)-C(19)	135.9(3)
C(14)-C(15)	136.3(3)
C(15)-C(16)	138.9(4)
C(15)-H(15)	93.00
C(16)-C(17)	134.1(5)
C(16)-H(16)	93.00
C(17)-C(18)	135.5(4)
C(17)-H(17)	93.00
C(18)-C(19)	139.3(4)
C(18)-H(18)	93.00
C(19)-H(19)	93.00
C(20)-C(25)	137.5(3)
C(20)-C(21)	138.3(3)
C(21)-C(22)	137.4(3)
C(21)-H(21)	93.00
C(22)-C(23)	137.3(4)
C(22)-H(22)	93.00
C(23)-C(24)	139.2(4)
C(23)-C(26)	152.5(4)
C(24)-C(25)	138.1(3)
C(24)-H(24)	93.00
C(25)-H(25)	93.00
C(26)-H(26A)	96.00

C(26)-H(26B)	96.00
C(26)-H(26C)	96.00
C(27)-C(28)	138.4(3)
C(27)-C(32)	139.1(3)
C(28)-C(29)	138.2(3)
C(28)-H(28)	93.00
C(29)-C(30)	137.0(4)
C(29)-H(29)	93.00
C(30)-C(31)	137.3(4)
C(30)-H(30)	93.00
C(31)-C(32)	137.4(3)
C(31)-H(31)	93.00
C(32)-H(32)	93.00

O(1)-S(1)-O(3)	120.40(10)
O(1)-S(1)-N(1)	107.37(9)
O(3)-S(1)-N(1)	104.18(10)
O(1)-S(1)-C(20)	109.27(10)
O(3)-S(1)-C(20)	108.82(10)
N(1)-S(1)-C(20)	105.77(9)
C(11)-O(4)-C(12)	118.7(2)
C(1)-N(1)-C(4)	108.87(15)
C(1)-N(1)-S(1)	126.53(14)
C(4)-N(1)-S(1)	117.26(13)
N(3)-N(2)-C(2)	113.49(17)
N(2)-N(3)-C(5)	112.29(17)
C(2)-C(1)-N(1)	110.30(17)
C(2)-C(1)-C(27)	124.32(19)
N(1)-C(1)-C(27)	125.37(17)
C(1)-C(2)-N(2)	120.91(18)
C(1)-C(2)-C(3)	112.10(18)
N(2)-C(2)-C(3)	126.97(18)
C(2)-C(3)-C(14)	111.16(18)
C(2)-C(3)-C(4)	102.01(16)
C(14)-C(3)-C(4)	114.69(18)
C(2)-C(3)-H(3)	109.6
C(14)-C(3)-H(3)	109.6
C(4)-C(3)-H(3)	109.6

N(1)-C(4)-C(3)	105.57(16)
N(1)-C(4)-H(4A)	110.6
C(3)-C(4)-H(4A)	110.6
N(1)-C(4)-H(4B)	110.6
C(3)-C(4)-H(4B)	110.6
H(4A)-C(4)-H(4B)	108.8
C(6)-C(5)-C(10)	119.8(2)
C(6)-C(5)-N(3)	124.73(18)
C(10)-C(5)-N(3)	115.49(19)
C(7)-C(6)-C(5)	120.05(19)
C(7)-C(6)-H(6)	120.0
C(5)-C(6)-H(6)	120.0
C(6)-C(7)-C(8)	120.9(2)
C(6)-C(7)-H(7)	119.5
C(8)-C(7)-H(7)	119.5
C(9)-C(8)-C(7)	119.0(2)
C(9)-C(8)-C(11)	121.9(2)
C(7)-C(8)-C(11)	119.1(2)
C(8)-C(9)-C(10)	120.23(19)
C(8)-C(9)-H(9)	119.9
C(10)-C(9)-H(9)	119.9
C(5)-C(10)-C(9)	120.0(2)
C(5)-C(10)-H(10)	120.0
C(9)-C(10)-H(10)	120.0
O(2)-C(11)-O(4)	123.3(2)
O(2)-C(11)-C(8)	124.3(2)
O(4)-C(11)-C(8)	112.4(2)
O(4)-C(12)-C(13)	108.7(2)
O(4)-C(12)-H(12A)	109.9
C(13)-C(12)-H(12A)	109.9
O(4)-C(12)-H(12B)	109.9
C(13)-C(12)-H(12B)	109.9
H(12A)-C(12)-H(12B)	108.3
C(12)-C(13)-H(13A)	109.5
C(12)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(12)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5

H(13B)-C(13)-H(13C)	109.5
C(19)-C(14)-C(15)	116.6(3)
C(19)-C(14)-C(3)	121.7(2)
C(15)-C(14)-C(3)	121.8(3)
C(14)-C(15)-C(16)	121.2(3)
C(14)-C(15)-H(15)	119.4
C(16)-C(15)-H(15)	119.4
C(17)-C(16)-C(15)	121.2(3)
C(17)-C(16)-H(16)	119.4
C(15)-C(16)-H(16)	119.4
C(16)-C(17)-C(18)	119.1(3)
C(16)-C(17)-H(17)	120.5
C(18)-C(17)-H(17)	120.5
C(17)-C(18)-C(19)	119.3(3)
C(17)-C(18)-H(18)	120.3
C(19)-C(18)-H(18)	120.3
C(14)-C(19)-C(18)	122.6(3)
C(14)-C(19)-H(19)	118.7
C(18)-C(19)-H(19)	118.7
C(25)-C(20)-C(21)	120.6(2)
C(25)-C(20)-S(1)	119.32(18)
C(21)-C(20)-S(1)	119.99(18)
C(22)-C(21)-C(20)	119.6(2)
C(22)-C(21)-H(21)	120.2
C(20)-C(21)-H(21)	120.2
C(23)-C(22)-C(21)	121.1(3)
C(23)-C(22)-H(22)	119.4
C(21)-C(22)-H(22)	119.4
C(22)-C(23)-C(24)	118.5(3)
C(22)-C(23)-C(26)	121.0(3)
C(24)-C(23)-C(26)	120.5(3)
C(25)-C(24)-C(23)	121.1(3)
C(25)-C(24)-H(24)	119.4
C(23)-C(24)-H(24)	119.4
C(20)-C(25)-C(24)	119.0(2)
C(20)-C(25)-H(25)	120.5
C(24)-C(25)-H(25)	120.5
C(23)-C(26)-H(26A)	109.5

C(23)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
C(23)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
C(28)-C(27)-C(32)	118.8(2)
C(28)-C(27)-C(1)	118.51(19)
C(32)-C(27)-C(1)	122.57(19)
C(29)-C(28)-C(27)	120.8(2)
C(29)-C(28)-H(28)	119.6
C(27)-C(28)-H(28)	119.6
C(30)-C(29)-C(28)	119.6(3)
C(30)-C(29)-H(29)	120.2
C(28)-C(29)-H(29)	120.2
C(29)-C(30)-C(31)	120.3(2)
C(29)-C(30)-H(30)	119.8
C(31)-C(30)-H(30)	119.8
C(30)-C(31)-C(32)	120.4(3)
C(30)-C(31)-H(31)	119.8
C(32)-C(31)-H(31)	119.8
C(31)-C(32)-C(27)	120.0(2)
C(31)-C(32)-H(32)	120.0
C(27)-C(32)-H(32)	120.0

4.2 X-ray crystal structure and crystal data of 7 (CCDC: 1975115)

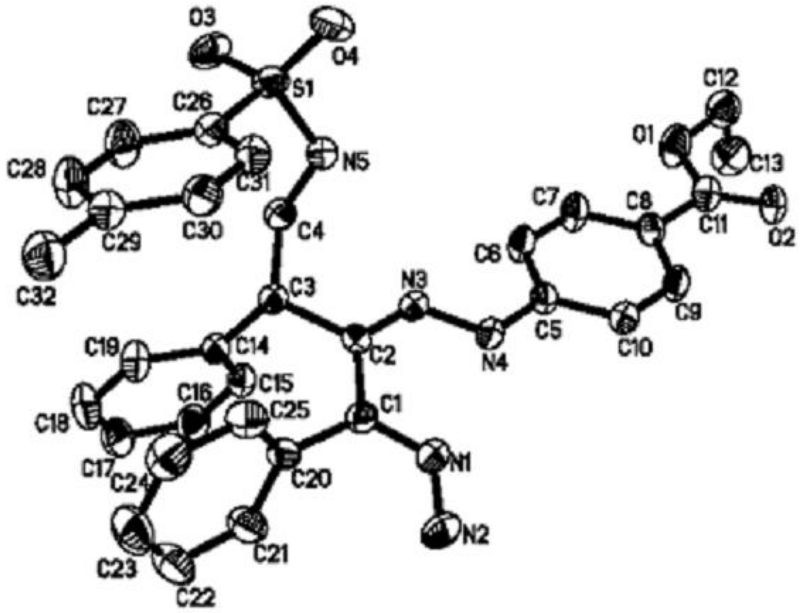


Figure S1.X-Ray crystal structure of 7

Table 2-1 Crystal data and structure refinement for 7

Crystal data	Compound **7**
Identification code	rj_1_0m
Empirical formula	$C_{32}H_{33}N_5O_4S$
Formula weight	583.69
Temperature	296(2) K
Wavelength	71.073 pm
Crystal system, space group	Monoclinic, $P2_1/c$
Unit cell dimensions	a = 831.54(14) pm alpha = 90° b = 2034.0(4) pm beta = 101.759(3)° c = 1796.2(3) pm gamma = 90°
Volume	2.9743(9) nm^3
Z, Calculated density	4, 1.303 Mg/m^3
Absorption coefficient	0.154 mm^{-1}
F(000)	1232
Theta range for data collection	1.531 to 27.567°.
Limiting indices	-10<=h<=9, -26<=k<=26, -23<=l<=20
Reflections collected / unique	18482, 6830 [R(int) = 0.0720]
Completeness to theta = 25.242º	99.9 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6830 / 0 / 389
Goodness-of-fit on F^2	0.983
Final R indices [I>2sigma(I)]	R1 = 0.0592, wR2 = 0.1285
R indices (all data)	R1 = 0.1548, wR2 = 0.1675
Largest diff. peak and hole	0.40 and -0.30 e.$Å^{-3}$

Table 2-2. Bond lengths [pm] and angles [°] for 7

S(1)-O(4)	142.9(2)
S(1)-O(3)	143.3(2)
S(1)-N(5)	160.6(2)
S(1)-C(26)	175.5(3)
O(1)-C(11)	135.1(4)
O(1)-C(12)	148.3(4)
O(2)-C(11)	120.5(3)
N(1)-C(1)	129.2(3)
N(1)-N(2)	138.2(4)
N(2)-H(2C)	86(4)
N(2)-H(2D)	110(5)
N(3)-C(2)	129.2(3)
N(3)-N(4)	135.6(3)
N(4)-C(5)	138.0(3)
N(4)-H(4)	86.00
N(5)-C(4)	146.3(3)
N(5)-H(5)	86.00
C(1)-C(2)	148.0(4)
C(1)-C(20)	149.8(4)
C(2)-C(3)	151.8(4)
C(3)-C(14)	151.8(4)
C(3)-C(4)	153.0(4)
C(3)-H(3)	98.00
C(4)-H(4A)	97.00
C(4)-H(4B)	97.00
C(5)-C(10)	139.2(4)
C(5)-C(6)	140.0(4)
C(6)-C(7)	136.5(4)
C(6)-H(6)	93.00
C(7)-C(8)	138.8(4)
C(7)-H(7)	93.00
C(8)-C(9)	139.2(4)
C(8)-C(11)	147.6(4)
C(9)-C(10)	136.6(4)
C(9)-H(9)	93.00
C(10)-H(10)	93.00

C(12)-C(13)	145.0(5)
C(12)-H(12A)	97.00
C(12)-H(12B)	97.00
C(13)-H(13A)	96.00
C(13)-H(13B)	96.00
C(13)-H(13C)	96.00
C(14)-C(19)	137.4(4)
C(14)-C(15)	138.1(4)
C(15)-C(16)	137.9(4)
C(15)-H(15)	93.00
C(16)-C(17)	136.9(4)
C(16)-H(16)	93.00
C(17)-C(18)	137.1(5)
C(17)-H(17)	93.00
C(18)-C(19)	139.1(4)
C(18)-H(18)	93.00
C(19)-H(19)	93.00
C(20)-C(25)	138.2(4)
C(20)-C(21)	138.2(4)
C(21)-C(22)	138.1(5)
C(21)-H(21)	93.00
C(22)-C(23)	136.4(6)
C(22)-H(22)	93.00
C(23)-C(24)	136.5(6)
C(23)-H(23)	93.00
C(24)-C(25)	137.8(5)
C(24)-H(24)	93.00
C(25)-H(25)	93.00
C(26)-C(27)	137.1(4)
C(26)-C(31)	138.9(4)
C(27)-C(28)	138.4(5)
C(27)-H(27)	93.00
C(28)-C(29)	137.4(4)
C(28)-H(28)	93.00
C(29)-C(30)	138.7(4)
C(29)-C(32)	150.7(4)
C(30)-C(31)	137.9(4)
C(30)-H(30)	93.00

C(31)-H(31)	93.00
C(32)-H(32A)	96.00
C(32)-H(32B)	96.00
C(32)-H(32C)	96.00

O(4)-S(1)-O(3)	119.48(14)
O(4)-S(1)-N(5)	106.77(14)
O(3)-S(1)-N(5)	107.23(14)
O(4)-S(1)-C(26)	108.00(14)
O(3)-S(1)-C(26)	106.25(15)
N(5)-S(1)-C(26)	108.77(14)
C(11)-O(1)-C(12)	117.1(3)
C(1)-N(1)-N(2)	118.1(3)
N(1)-N(2)-H(2C)	113(3)
N(1)-N(2)-H(2D)	112(2)
H(2C)-N(2)-H(2D)	115(3)
C(2)-N(3)-N(4)	120.9(2)
N(3)-N(4)-C(5)	119.2(2)
N(3)-N(4)-H(4)	120.4
C(5)-N(4)-H(4)	120.4
C(4)-N(5)-S(1)	120.9(2)
C(4)-N(5)-H(5)	119.6
S(1)-N(5)-H(5)	119.6
N(1)-C(1)-C(2)	118.4(3)
N(1)-C(1)-C(20)	121.8(3)
C(2)-C(1)-C(20)	119.7(3)
N(3)-C(2)-C(1)	127.3(3)
N(3)-C(2)-C(3)	115.1(2)
C(1)-C(2)-C(3)	117.6(2)
C(14)-C(3)-C(2)	112.9(2)
C(14)-C(3)-C(4)	109.1(2)
C(2)-C(3)-C(4)	112.3(2)
C(14)-C(3)-H(3)	107.4
C(2)-C(3)-H(3)	107.4
C(4)-C(3)-H(3)	107.4
N(5)-C(4)-C(3)	113.5(2)
N(5)-C(4)-H(4A)	108.9
C(3)-C(4)-H(4A)	108.9

N(5)-C(4)-H(4B)	108.9
C(3)-C(4)-H(4B)	108.9
H(4A)-C(4)-H(4B)	107.7
N(4)-C(5)-C(10)	120.0(3)
N(4)-C(5)-C(6)	122.0(3)
C(10)-C(5)-C(6)	118.0(3)
C(7)-C(6)-C(5)	120.4(3)
C(7)-C(6)-H(6)	119.8
C(5)-C(6)-H(6)	119.8
C(6)-C(7)-C(8)	121.5(3)
C(6)-C(7)-H(7)	119.3
C(8)-C(7)-H(7)	119.3
C(7)-C(8)-C(9)	118.2(3)
C(7)-C(8)-C(11)	123.0(3)
C(9)-C(8)-C(11)	118.8(3)
C(10)-C(9)-C(8)	120.6(3)
C(10)-C(9)-H(9)	119.7
C(8)-C(9)-H(9)	119.7
C(9)-C(10)-C(5)	121.2(3)
C(9)-C(10)-H(10)	119.4
C(5)-C(10)-H(10)	119.4
O(2)-C(11)-O(1)	122.4(3)
O(2)-C(11)-C(8)	125.2(3)
O(1)-C(11)-C(8)	112.3(3)
C(13)-C(12)-O(1)	107.5(3)
C(13)-C(12)-H(12A)	110.2
O(1)-C(12)-H(12A)	110.2
C(13)-C(12)-H(12B)	110.2
O(1)-C(12)-H(12B)	110.2
H(12A)-C(12)-H(12B)	108.5
C(12)-C(13)-H(13A)	109.5
C(12)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(12)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(19)-C(14)-C(15)	117.8(3)
C(19)-C(14)-C(3)	121.2(3)

C(15)-C(14)-C(3)	120.9(3)
C(16)-C(15)-C(14)	121.4(3)
C(16)-C(15)-H(15)	119.3
C(14)-C(15)-H(15)	119.3
C(17)-C(16)-C(15)	120.2(3)
C(17)-C(16)-H(16)	119.9
C(15)-C(16)-H(16)	119.9
C(16)-C(17)-C(18)	119.3(3)
C(16)-C(17)-H(17)	120.3
C(18)-C(17)-H(17)	120.3
C(17)-C(18)-C(19)	120.2(3)
C(17)-C(18)-H(18)	119.9
C(19)-C(18)-H(18)	119.9
C(14)-C(19)-C(18)	120.9(3)
C(14)-C(19)-H(19)	119.5
C(18)-C(19)-H(19)	119.5
C(25)-C(20)-C(21)	119.1(3)
C(25)-C(20)-C(1)	121.4(3)
C(21)-C(20)-C(1)	119.4(3)
C(22)-C(21)-C(20)	120.4(4)
C(22)-C(21)-H(21)	119.8
C(20)-C(21)-H(21)	119.8
C(23)-C(22)-C(21)	119.7(4)
C(23)-C(22)-H(22)	120.1
C(21)-C(22)-H(22)	120.1
C(22)-C(23)-C(24)	120.4(4)
C(22)-C(23)-H(23)	119.8
C(24)-C(23)-H(23)	119.8
C(23)-C(24)-C(25)	120.5(4)
C(23)-C(24)-H(24)	119.8
C(25)-C(24)-H(24)	119.8
C(24)-C(25)-C(20)	119.8(3)
C(24)-C(25)-H(25)	120.1
C(20)-C(25)-H(25)	120.1
C(27)-C(26)-C(31)	119.1(3)
C(27)-C(26)-S(1)	120.5(3)
C(31)-C(26)-S(1)	120.4(2)
C(26)-C(27)-C(28)	120.0(3)

C(26)-C(27)-H(27)	120.0
C(28)-C(27)-H(27)	120.0
C(29)-C(28)-C(27)	122.2(3)
C(29)-C(28)-H(28)	118.9
C(27)-C(28)-H(28)	118.9
C(28)-C(29)-C(30)	117.1(3)
C(28)-C(29)-C(32)	121.9(3)
C(30)-C(29)-C(32)	121.0(3)
C(31)-C(30)-C(29)	121.8(3)
C(31)-C(30)-H(30)	119.1
C(29)-C(30)-H(30)	119.1
C(30)-C(31)-C(26)	119.9(3)
C(30)-C(31)-H(31)	120.1
C(26)-C(31)-H(31)	120.1
C(29)-C(32)-H(32A)	109.5
C(29)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
C(29)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5

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5. References

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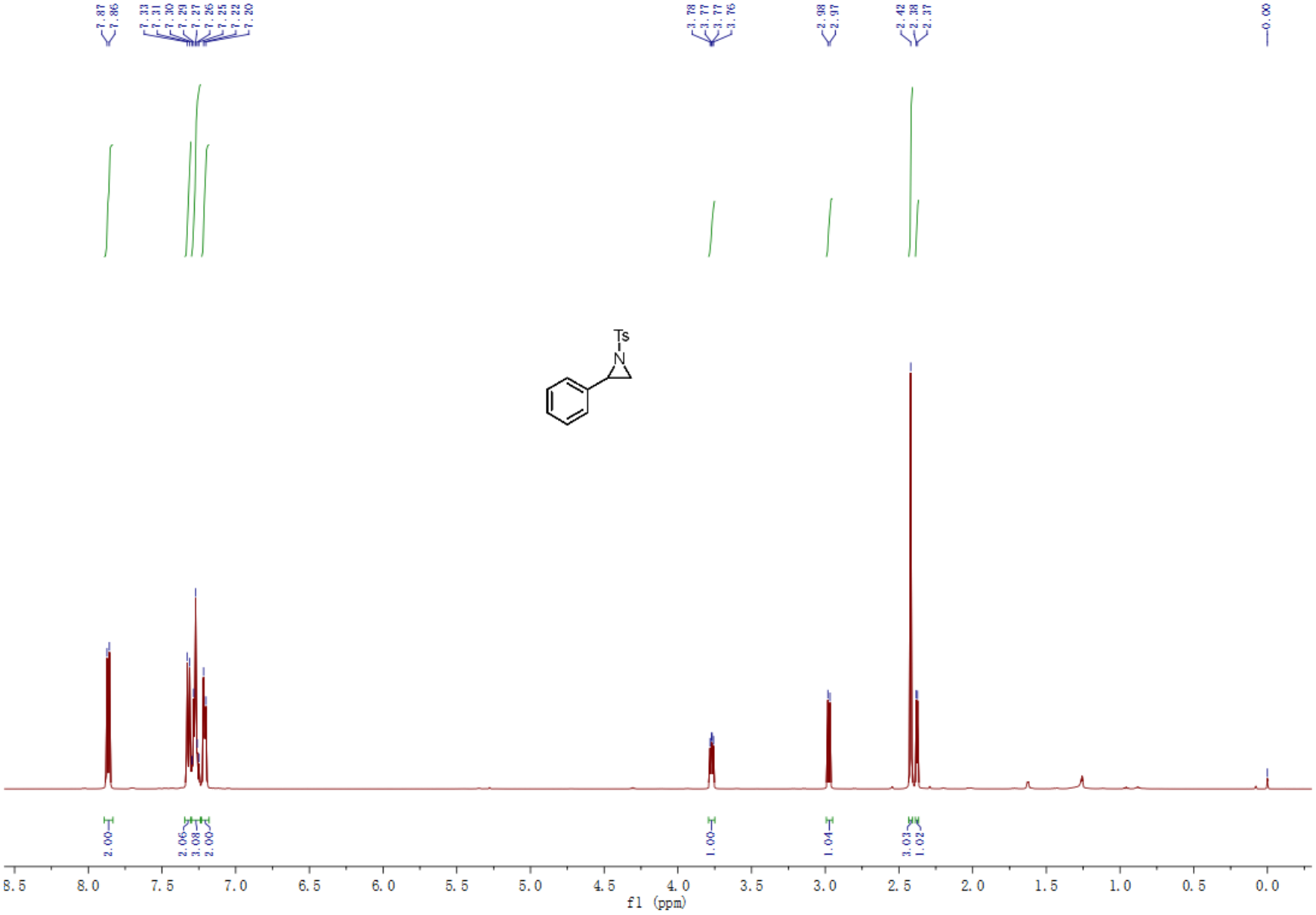
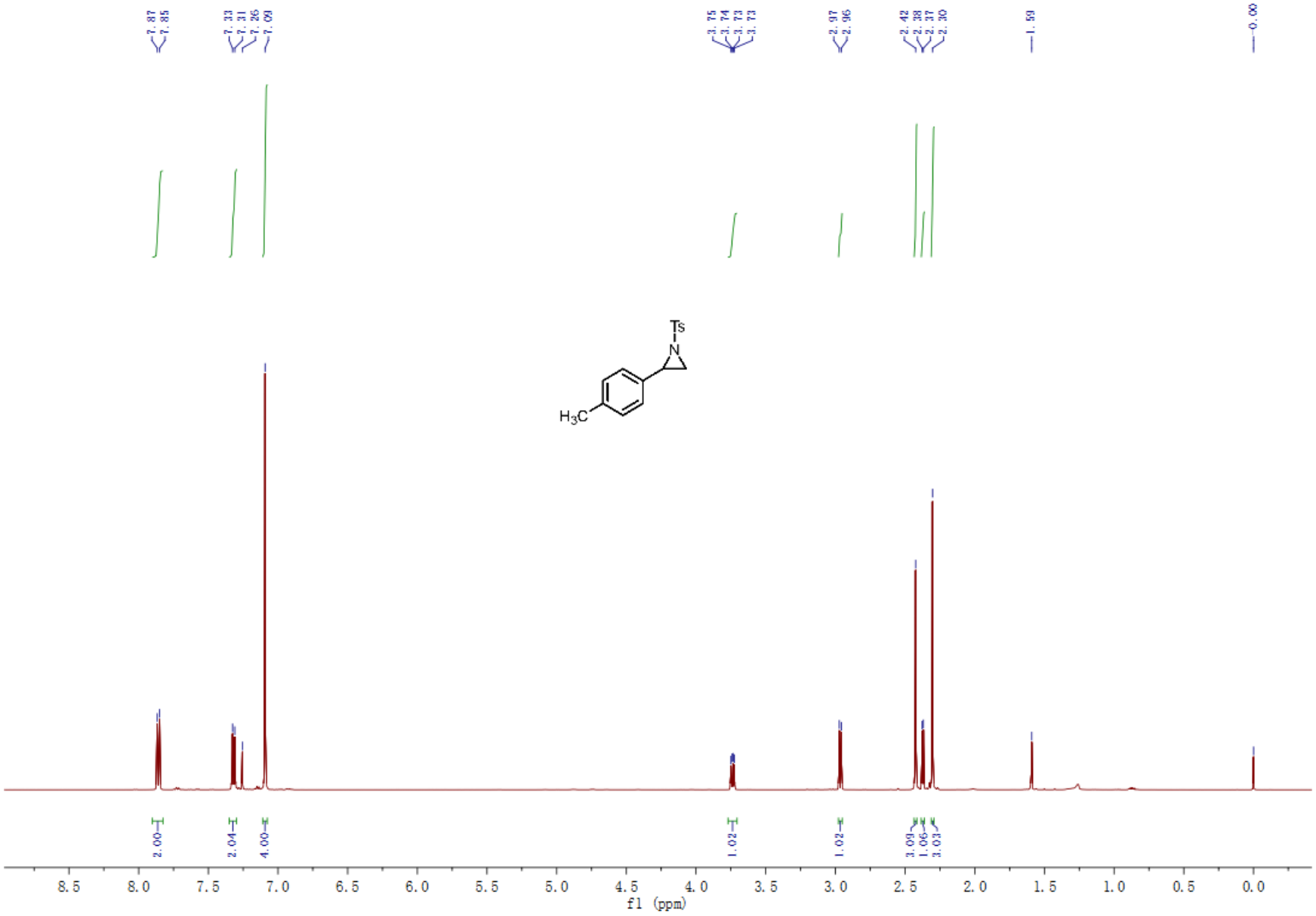
[2]. I. Saikia, B. Kashyap, P. Phukan, *Chem. Commun*. **2011**, *47*, 2967-2969.

[3]. H. N. Sun, B. B. Huang, R. Lin, C. Yang, W. J. Xia, *Beilstein J. Org. Chem.* **2015**, *11*, 524-529.

[4]. P. Hanson, J. R. Jones, A. B. Taylor, P. H. Walton, A. W. Timmsb, *J. Chem. Soc., Perkin Trans. 2*. **2002**, 1135-1150.

6. Spectral data

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7.09
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3.74
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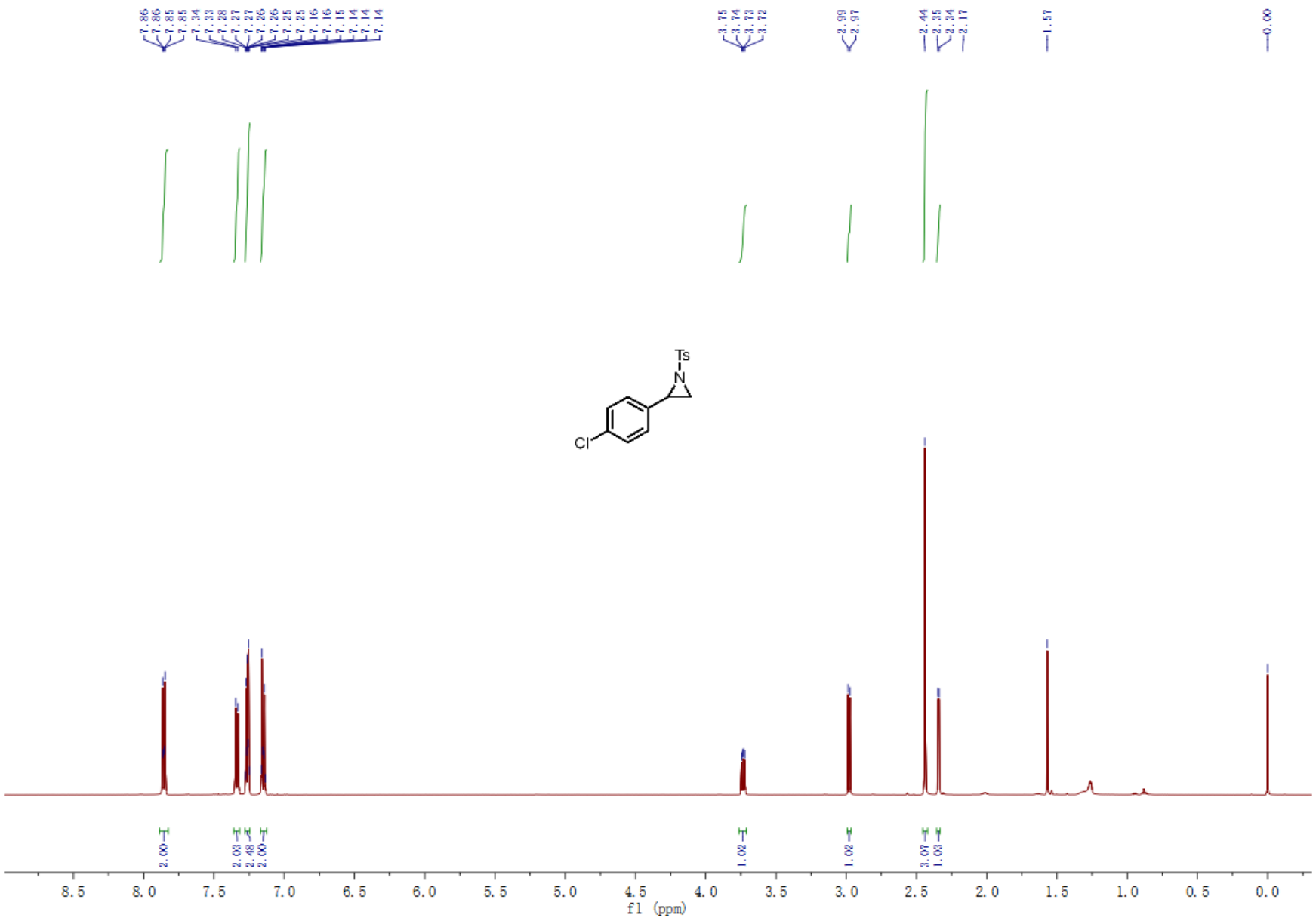
3.73
2.97
2.96
2.42
2.38
2.37
2.30
1.59
0.00
Ts
N
H3C
2.00
2.04
4.00
1.02
1.02
3.09
1.06
3.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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7.85
7.85
7.34
7.33
7.28
7.27
7.27
7.26
7.26
7.25
7.25
7.16
7.16
7.15
7.14
7.14
7.14
3.75
3.74
3.73
3.72
2.99
2.97
2.44
2.35
2.34
2.17
1.57
0.00
Ts
N
Cl
2.00
2.03
2.48
2.00
1.02
1.02
3.07
1.03
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
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2.5
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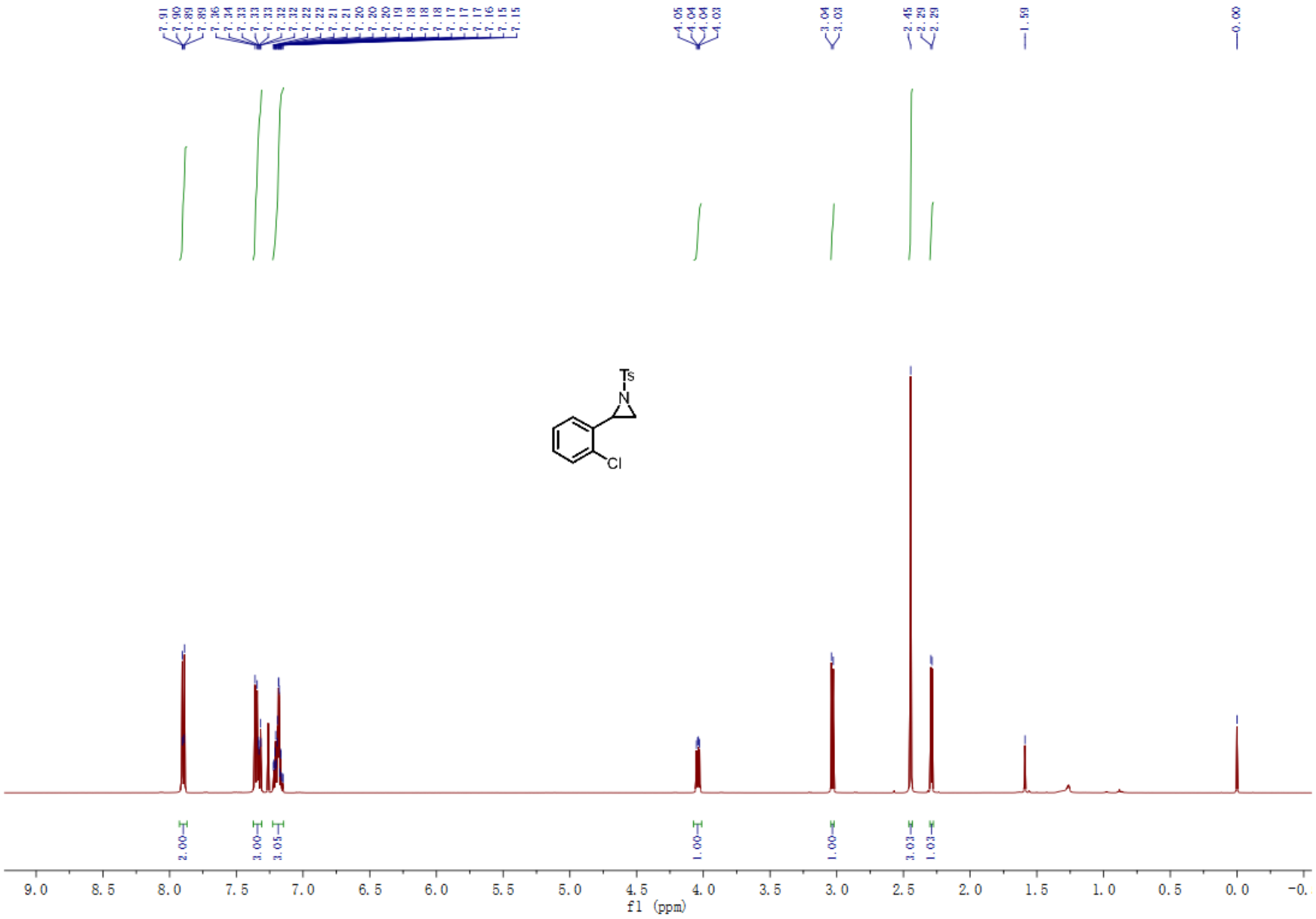
f1 (ppm)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7.90
7.89
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7.33
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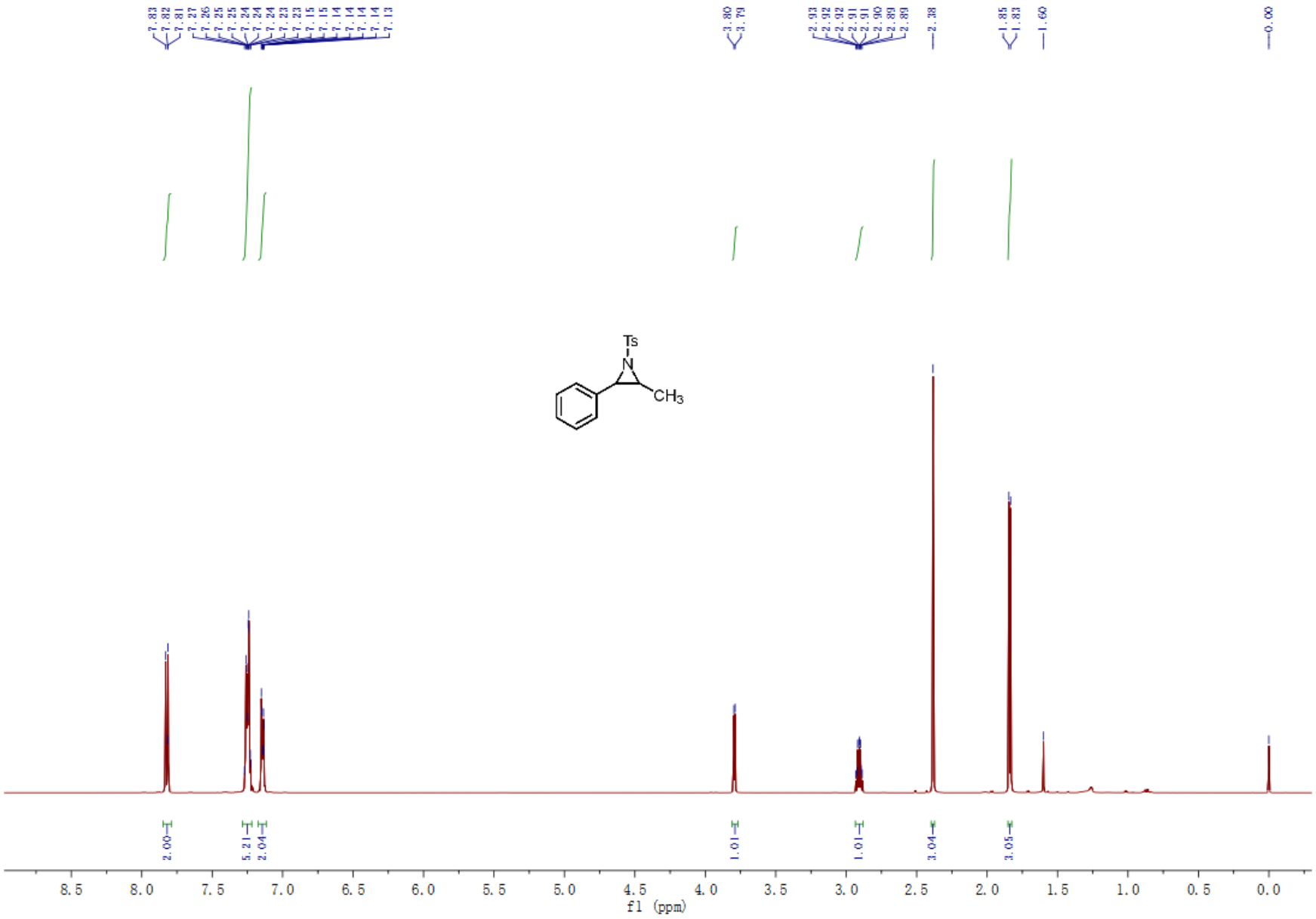
7.32
7.32
7.22
7.22
7.21
7.21
7.20
7.20
7.20
7.19
7.18
7.18
7.18
7.17
7.17
7.17
7.16
7.15
7.15
4.05
4.04
4.04
4.03
3.04
3.03
2.45
2.29
2.29
1.59
0.00
Ts
N
Cl
2.00
3.00
3.05
1.00
1.00
3.03
1.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)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7.24
7.23
7.23
7.15
7.14
7.14
7.14
7.13
3.80
3.79
2.93
2.92
2.92
2.91
2.91
2.90
2.89
2.89
2.38
1.85
1.83
1.60
0.00
Ts
N
CH3
2.00
5.21
2.04
1.01
1.01
3.04
3.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.0
2.5
2.0
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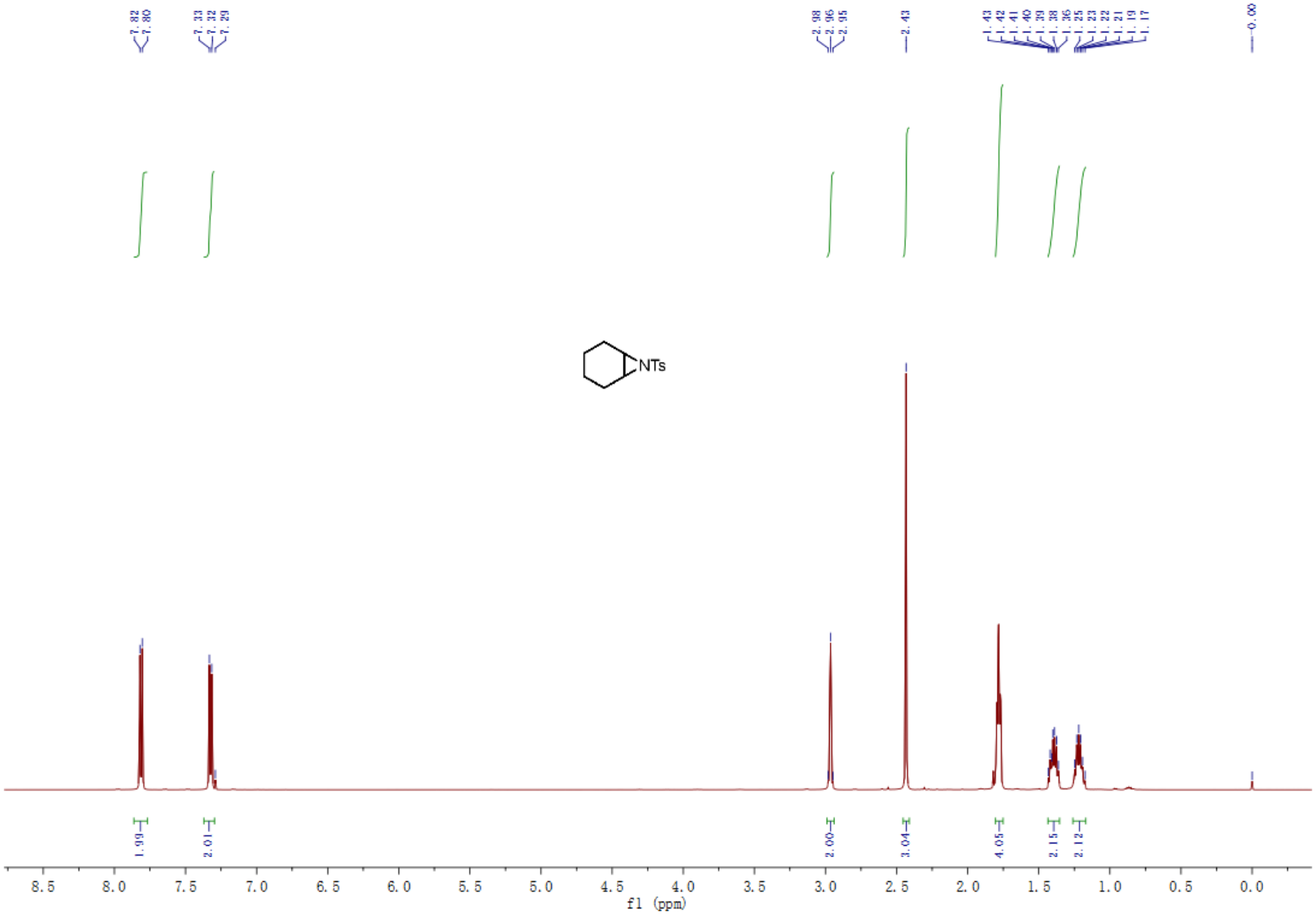
f1 (ppm)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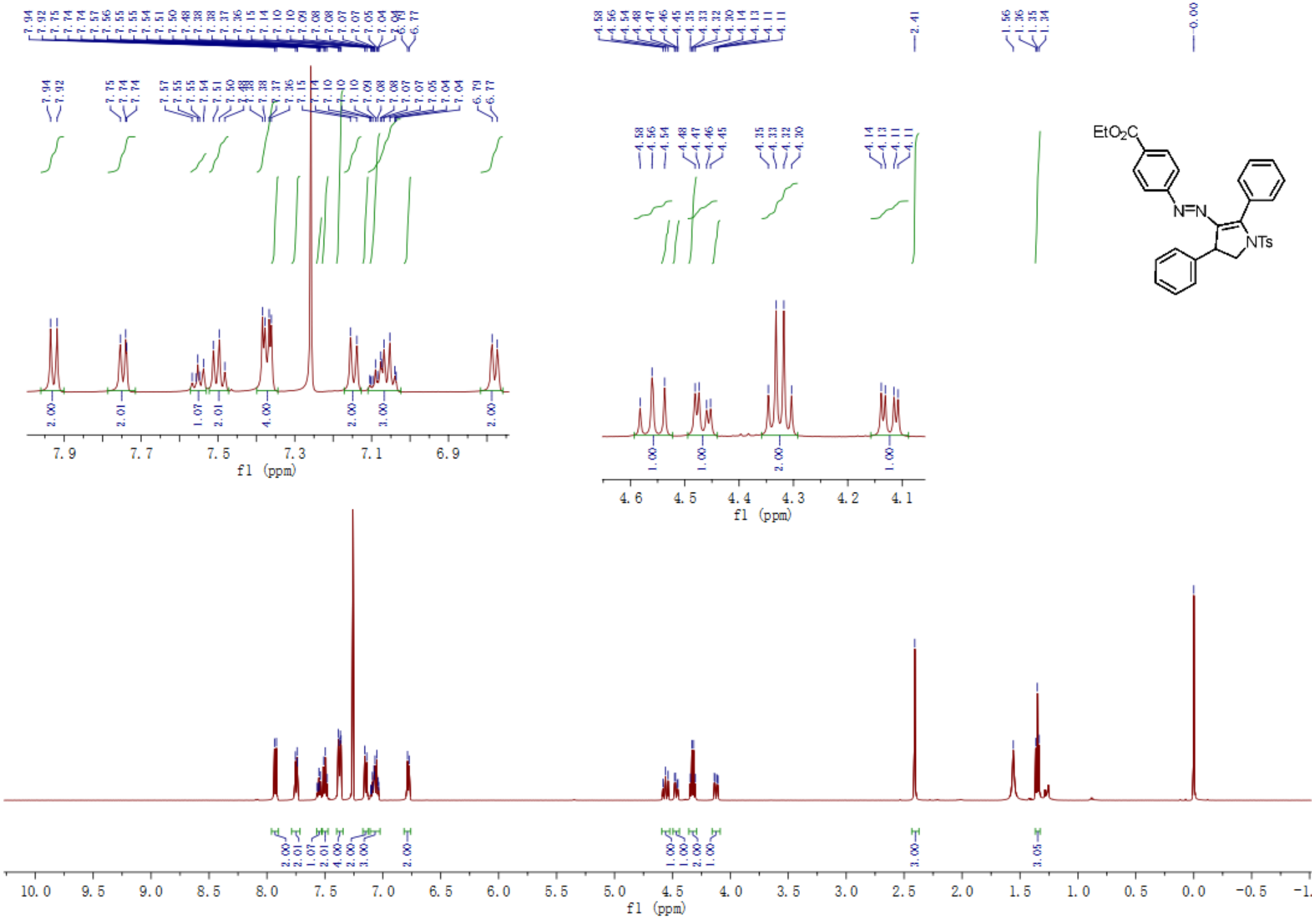
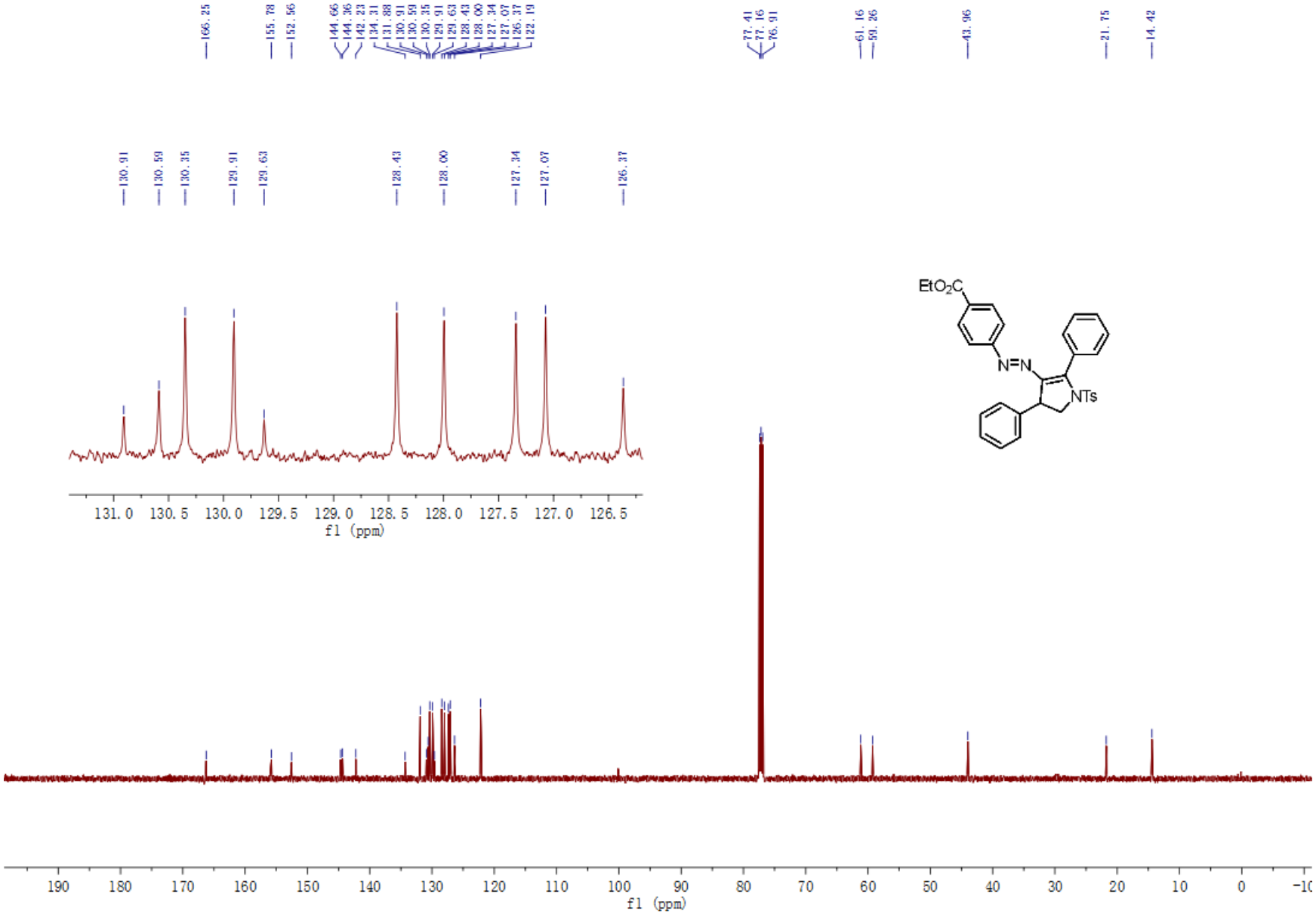
1.40
1.39
1.38
1.36
1.25
1.23
1.22
1.21
1.19
1.17
0.00
NTs
1.99
2.01
2.00
3.04
4.05
2.15
2.12
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


EtO2C
N=N
NTs
f1 (ppm)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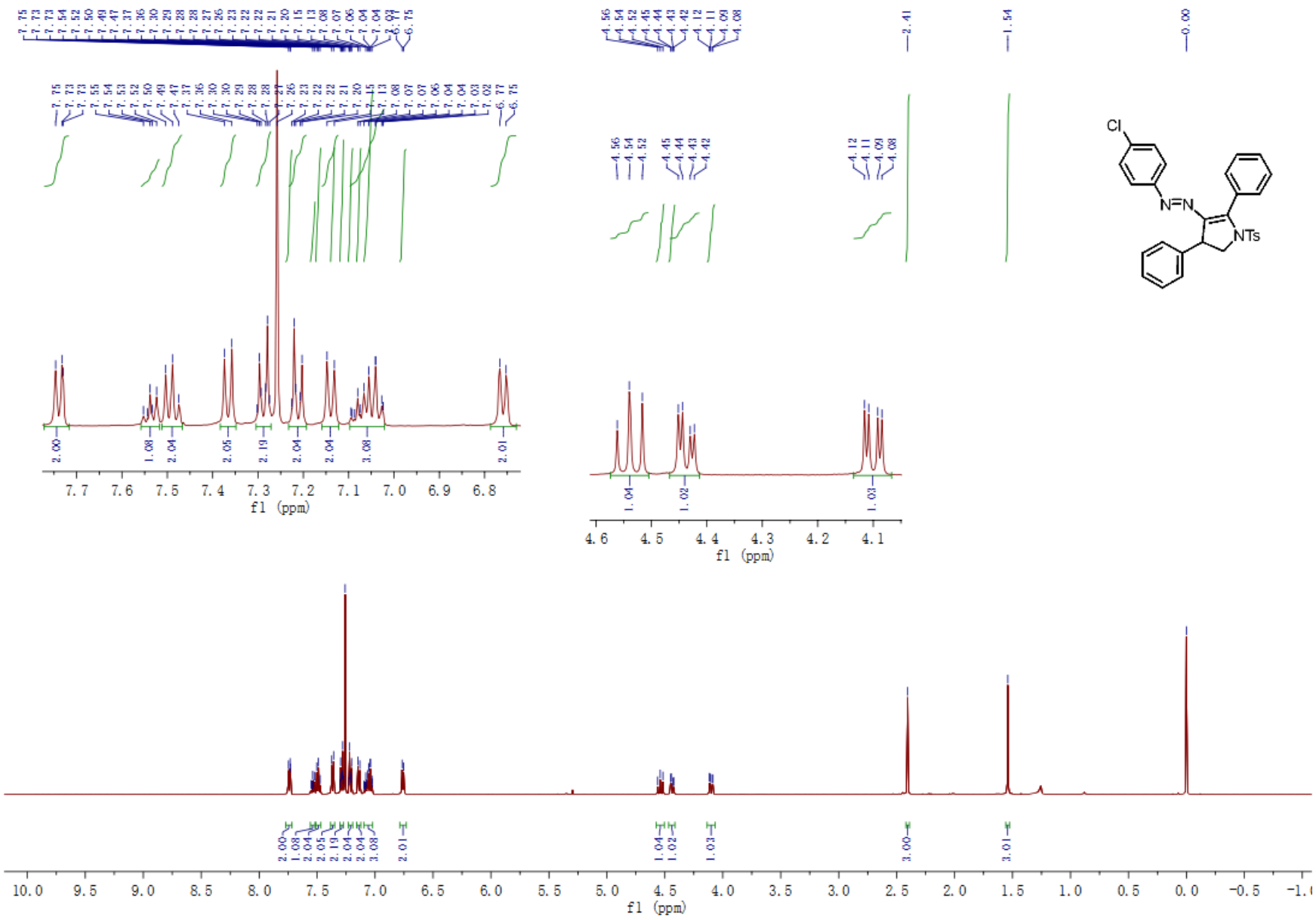
128.43
128.00
127.34
127.07
126.37
122.19
77.41
77.16
76.91
61.16
59.26
43.96
21.75
14.42
130.91
130.59
130.35
129.91
129.63
128.43
128.00
127.34
127.07
126.37
EtO2C
N=N
NTs
131.0 130.5 130.0 129.5 129.0 128.5 128.0 127.5 127.0 126.5
f1 (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


Cl
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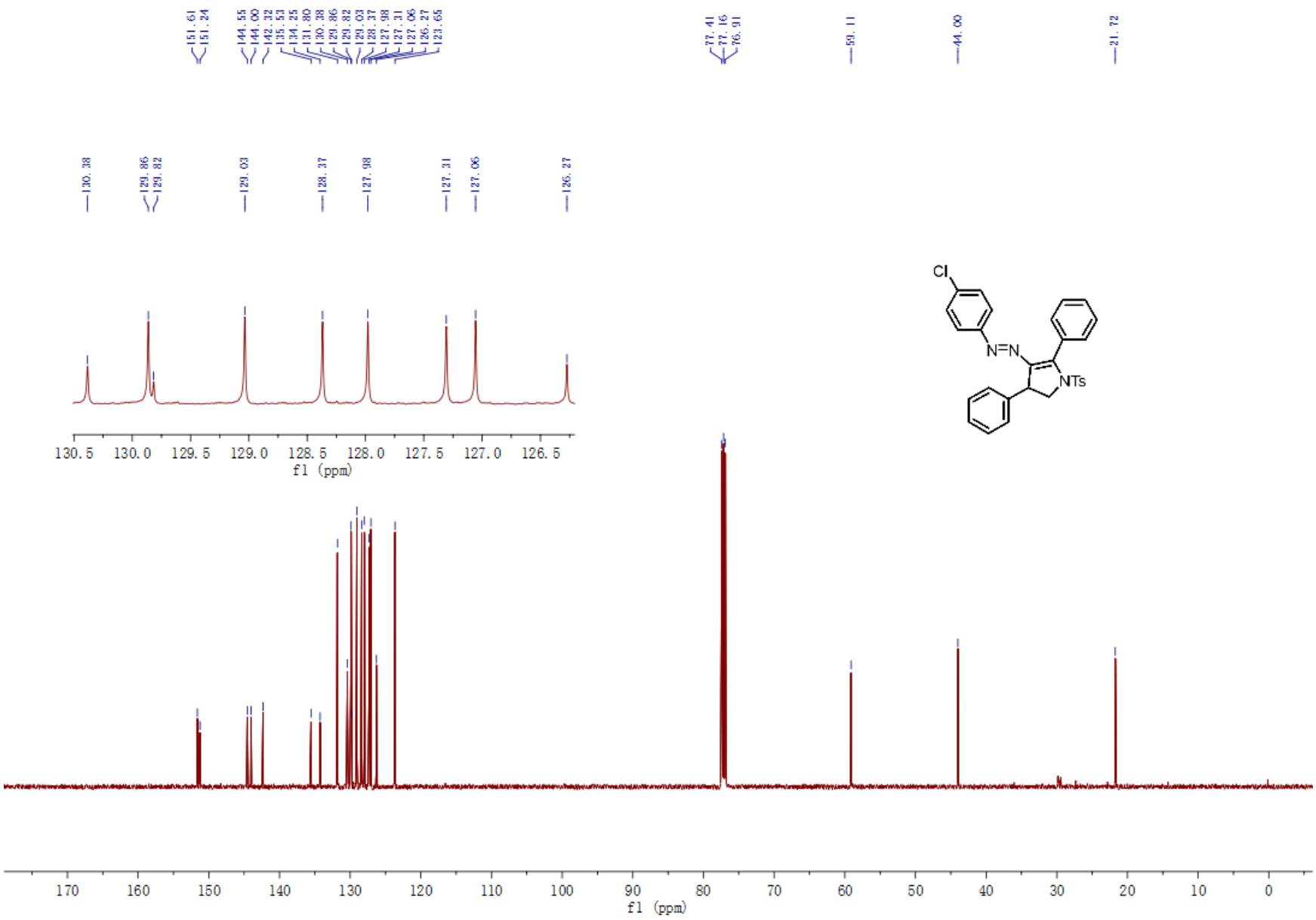
NTs
f1 (ppm)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128.37
127.98
127.31
127.06
126.27
123.65
77.41
77.16
76.91
59.11
44.00
21.72
130.38
129.86
129.82
129.03
128.37
127.98
127.31
127.06
126.27
Cl
N=N
NTs
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130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
f1 (ppm)
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160
150
140
130
120
110
100
90
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70
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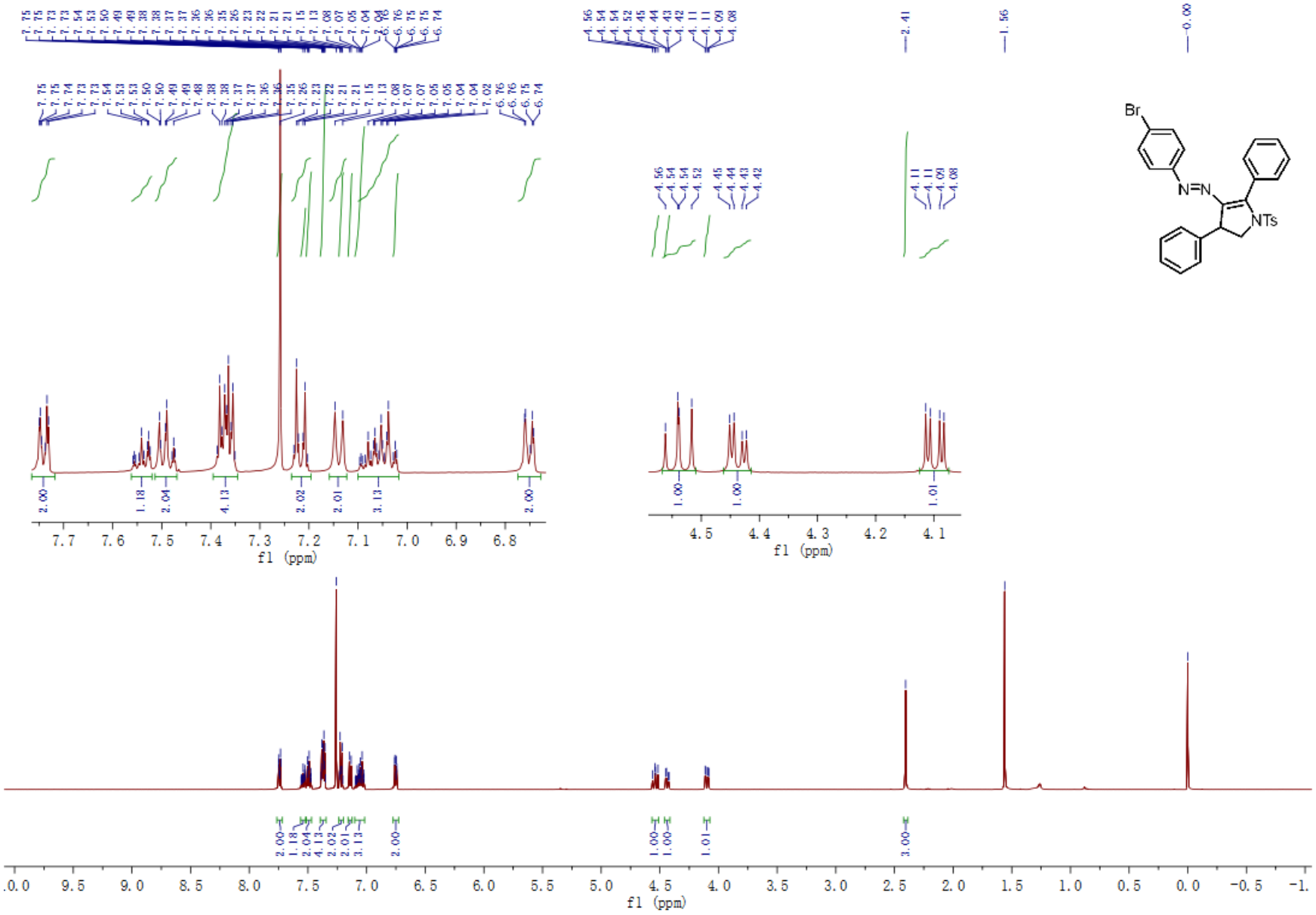
50
40
30
20
10
0
f1 (ppm)


Br
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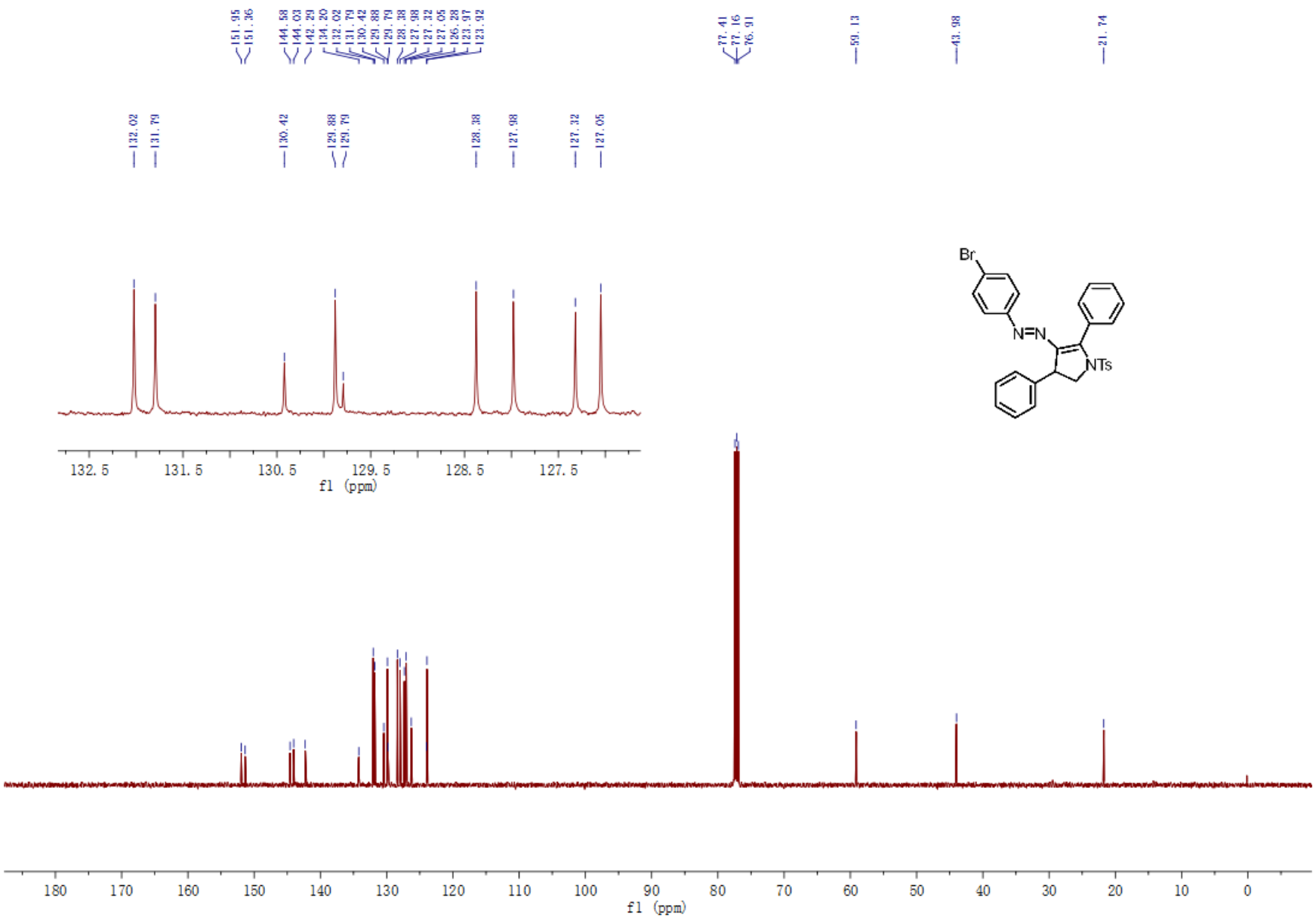
NTs
f1 (ppm)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127.05
126.28
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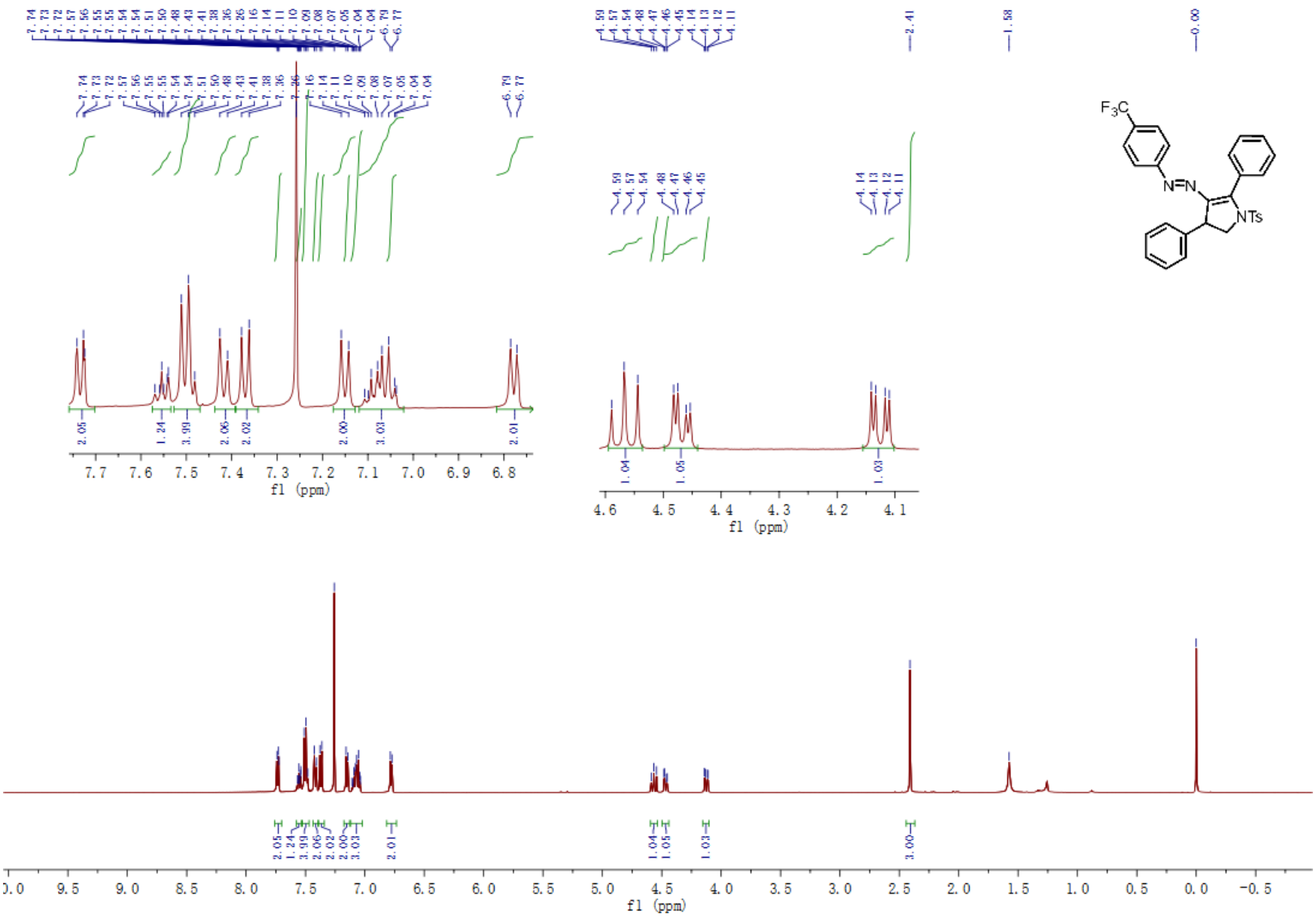
123.92
77.41
77.16
76.91
59.13
43.98
21.74
132.62
131.79
130.42
129.88
129.79
128.38
127.98
127.32
127.05
Br
N=N
NTs
132.5
131.5
130.5
129.5
128.5
127.5
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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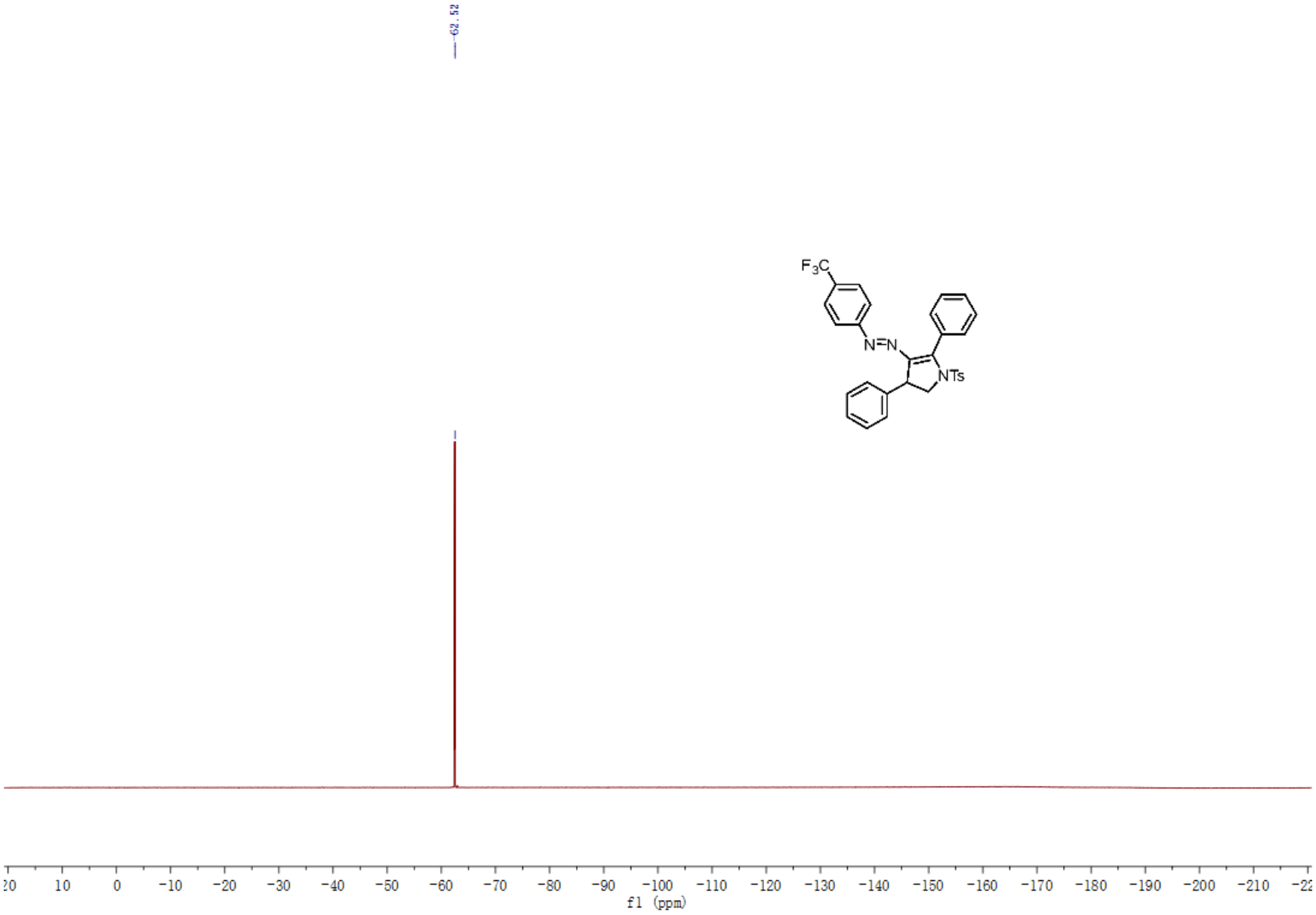
N=N
NTs
f1 (ppm)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F3C
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-50
-60
-70
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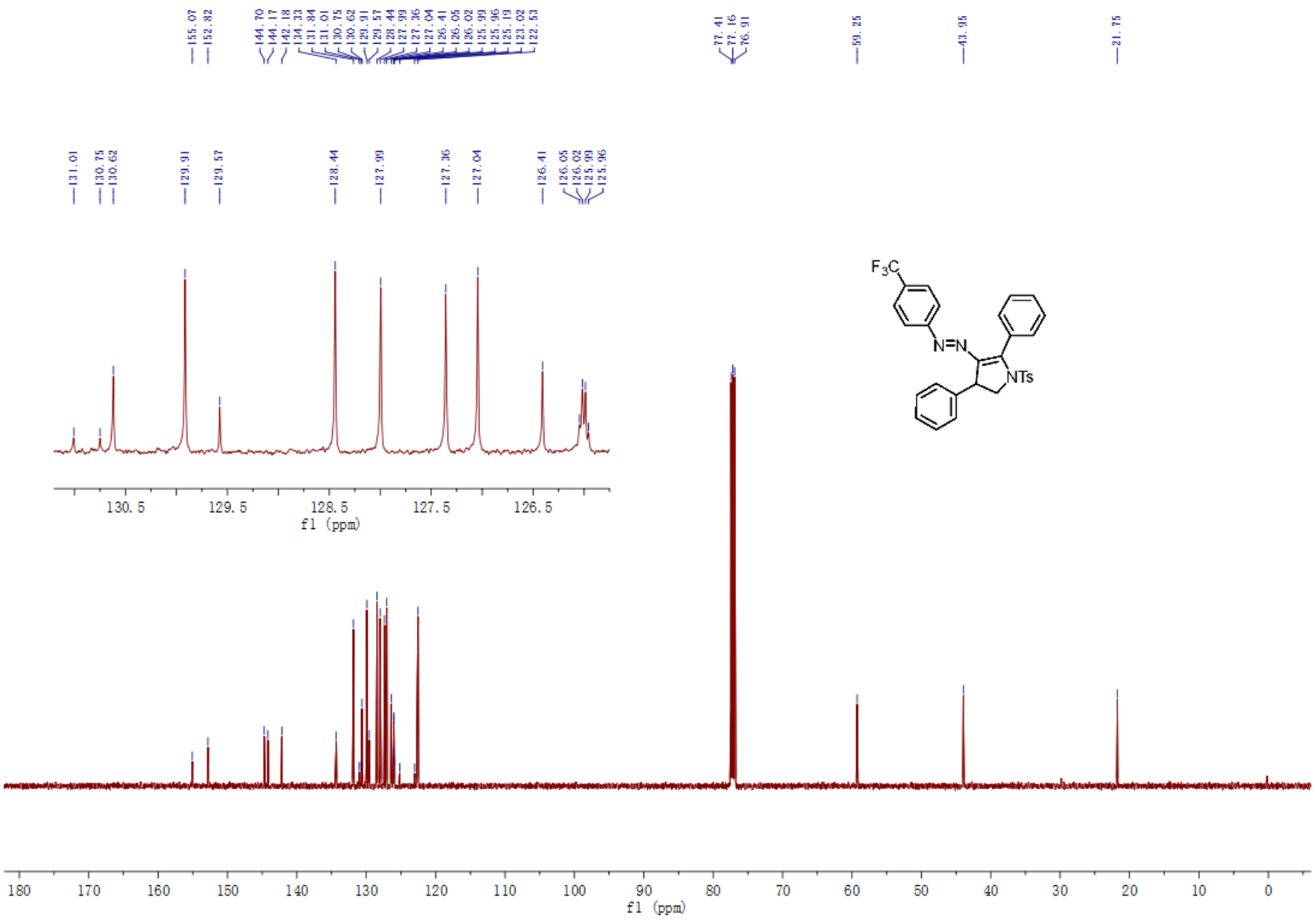
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)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134.33
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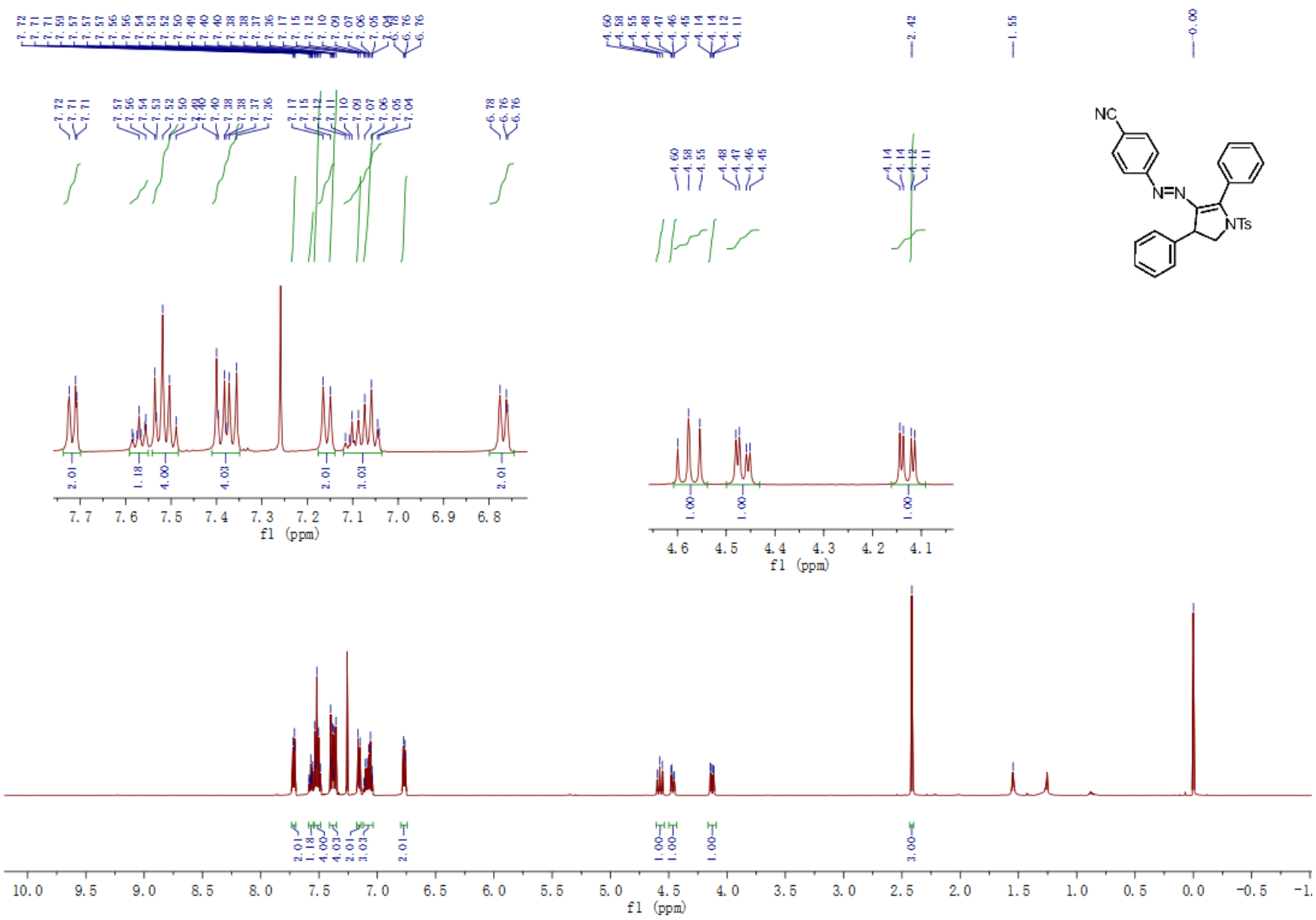
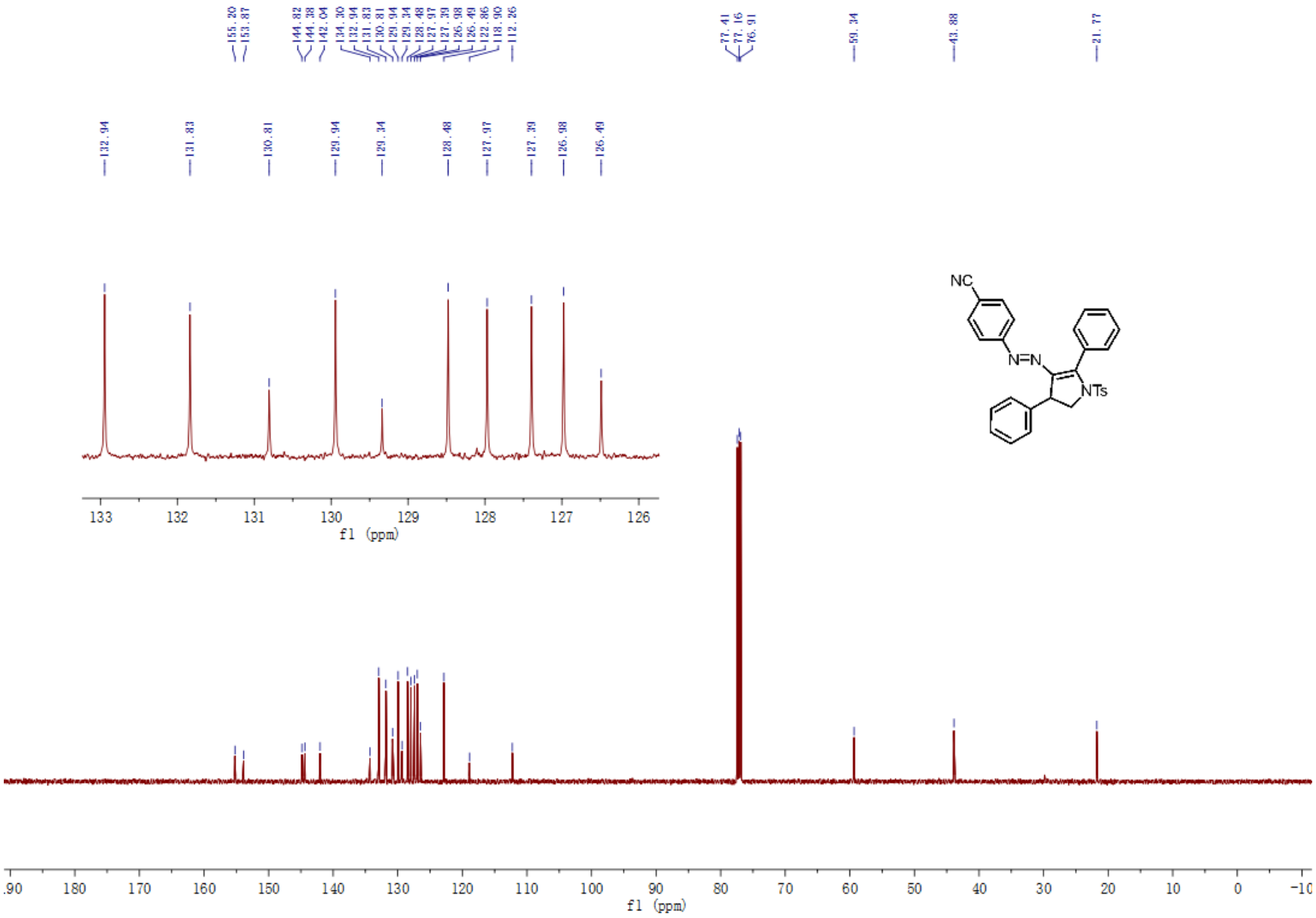
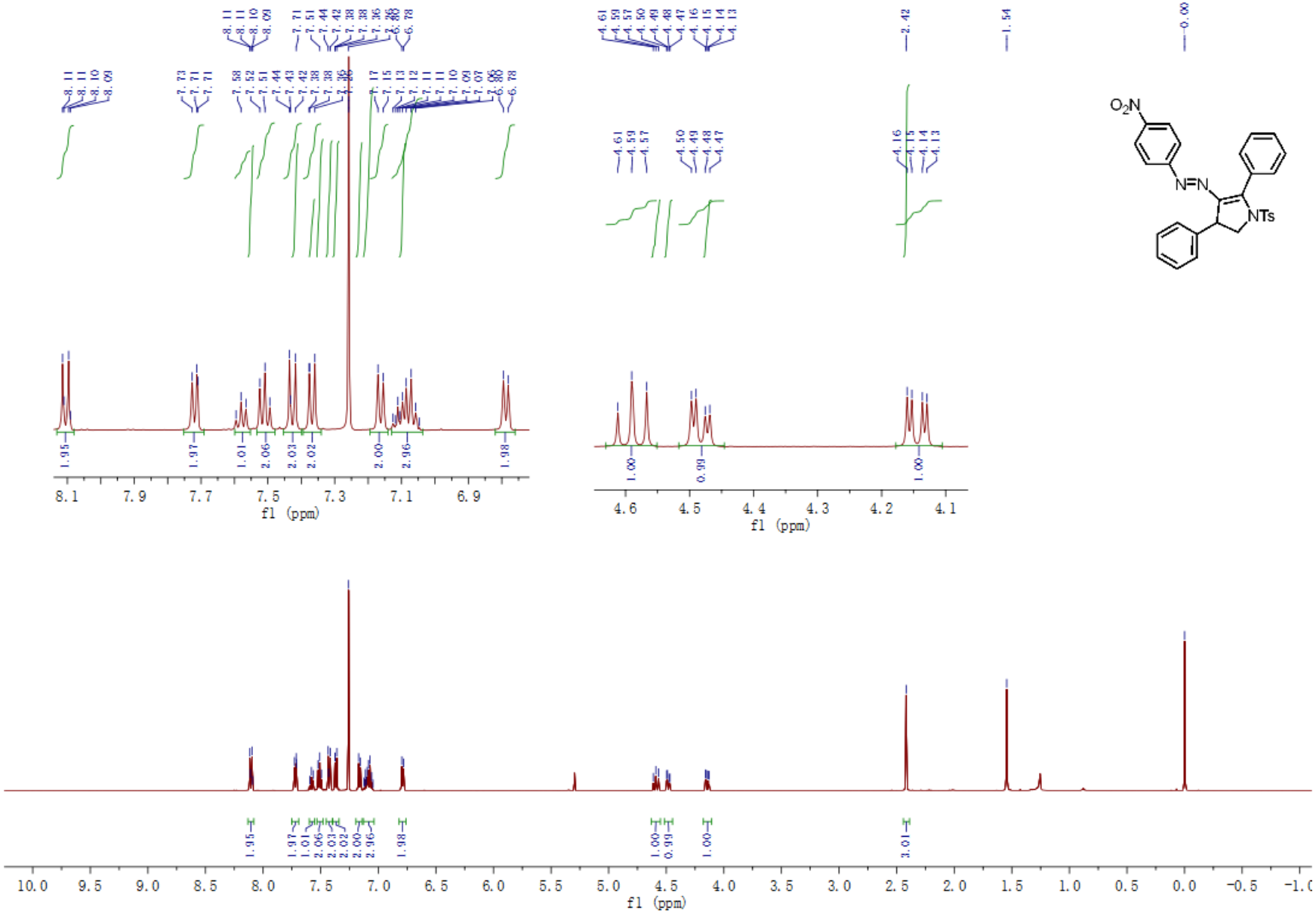
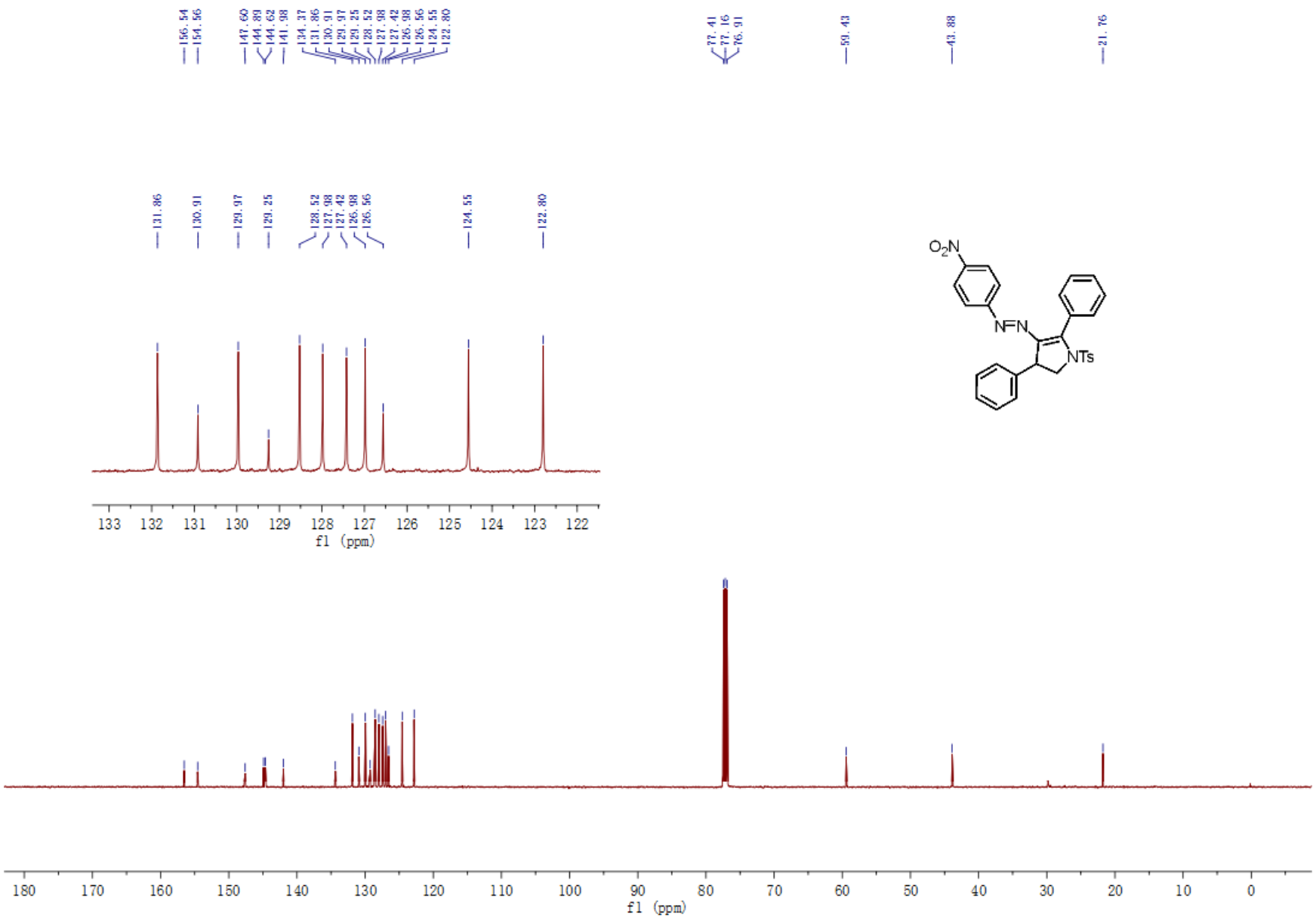
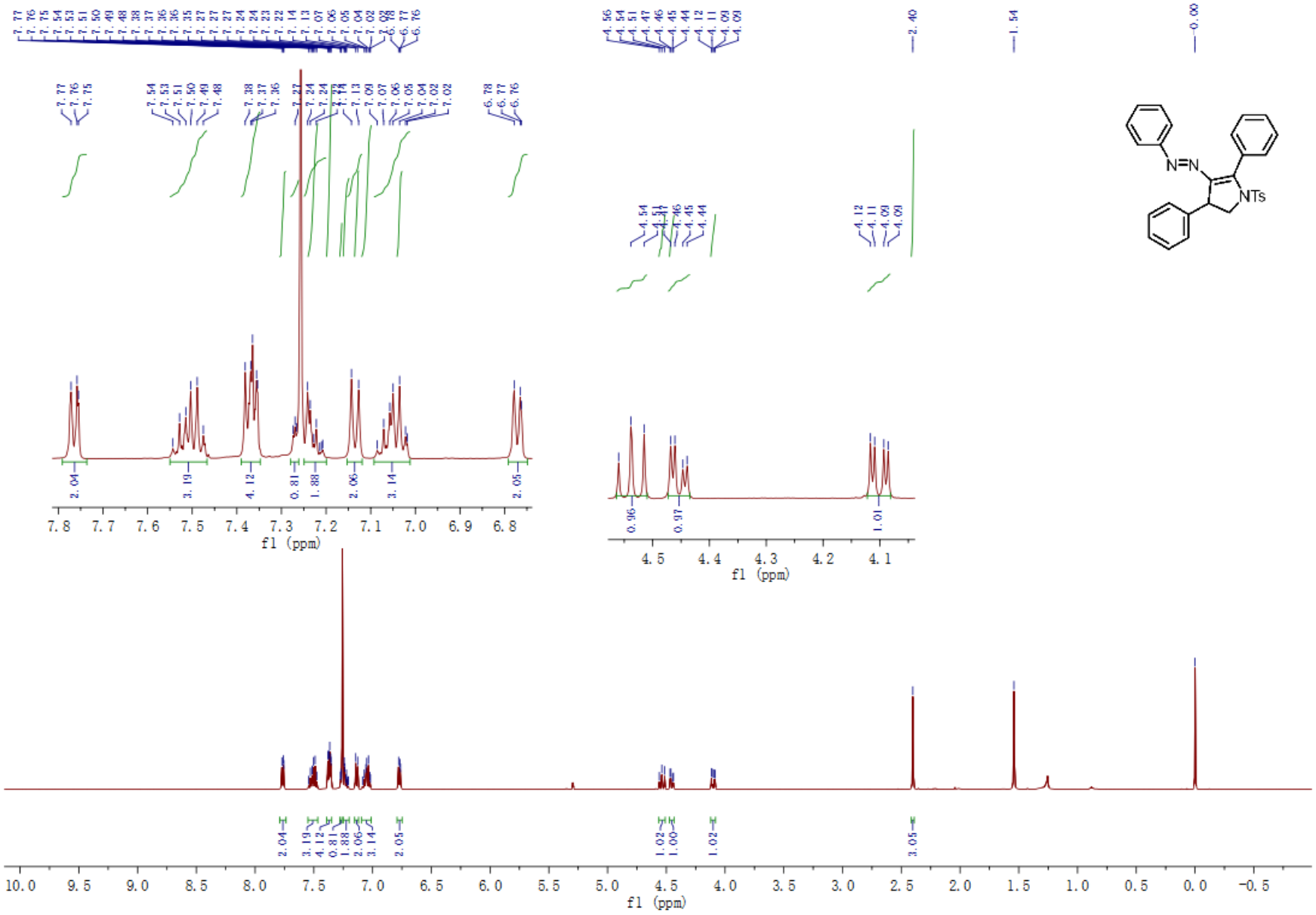
130.62
129.91
129.57
128.44
127.99
127.36
127.04
126.41
126.05
126.02
125.99
125.96
125.19
123.02
122.53
77.41
77.16
76.91
59.25
43.95
21.75
F3C
N=N
NTs
130.5
129.5
128.5
127.5
126.5
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

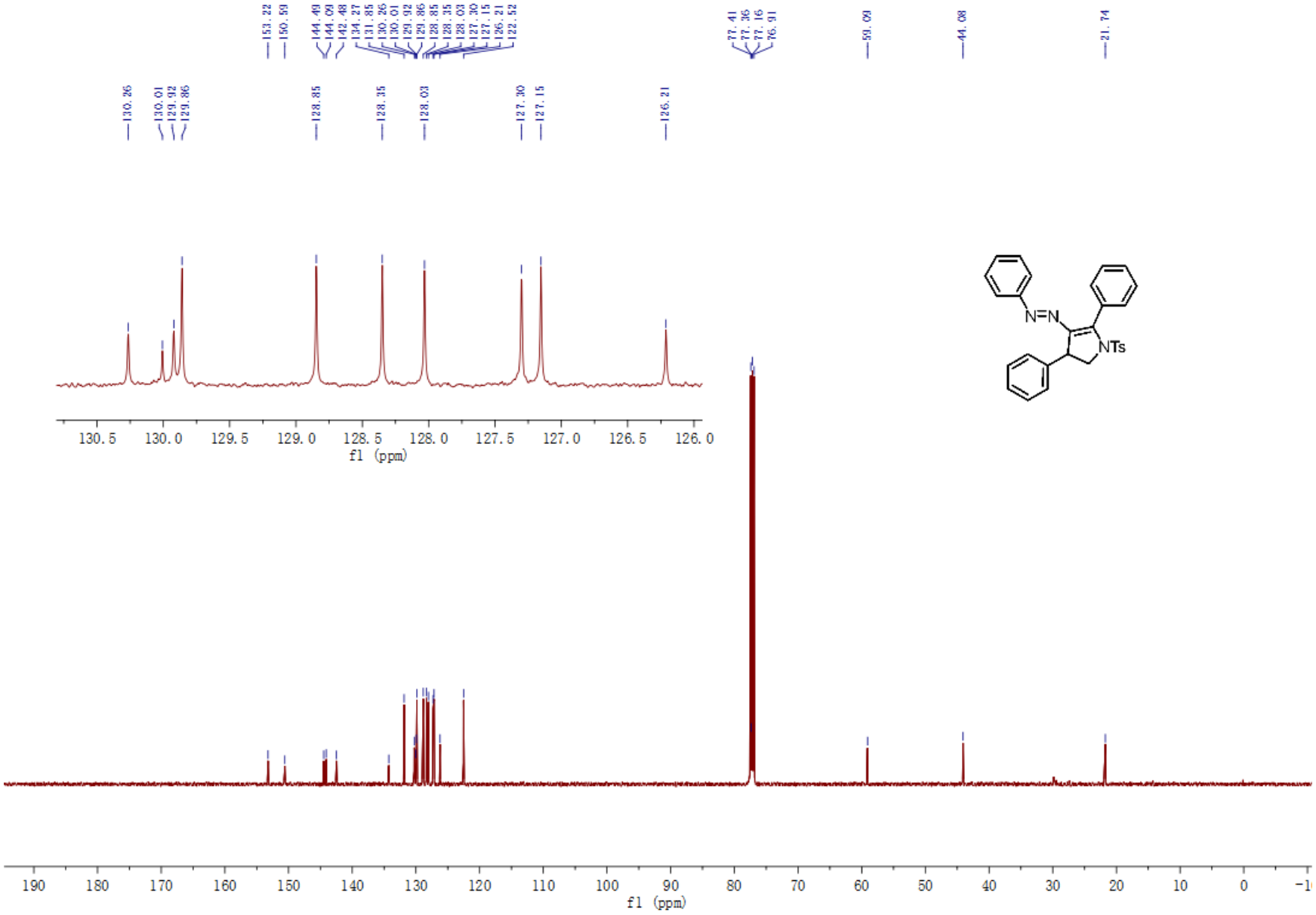

NC
N=N
NTs
f1 (ppm)

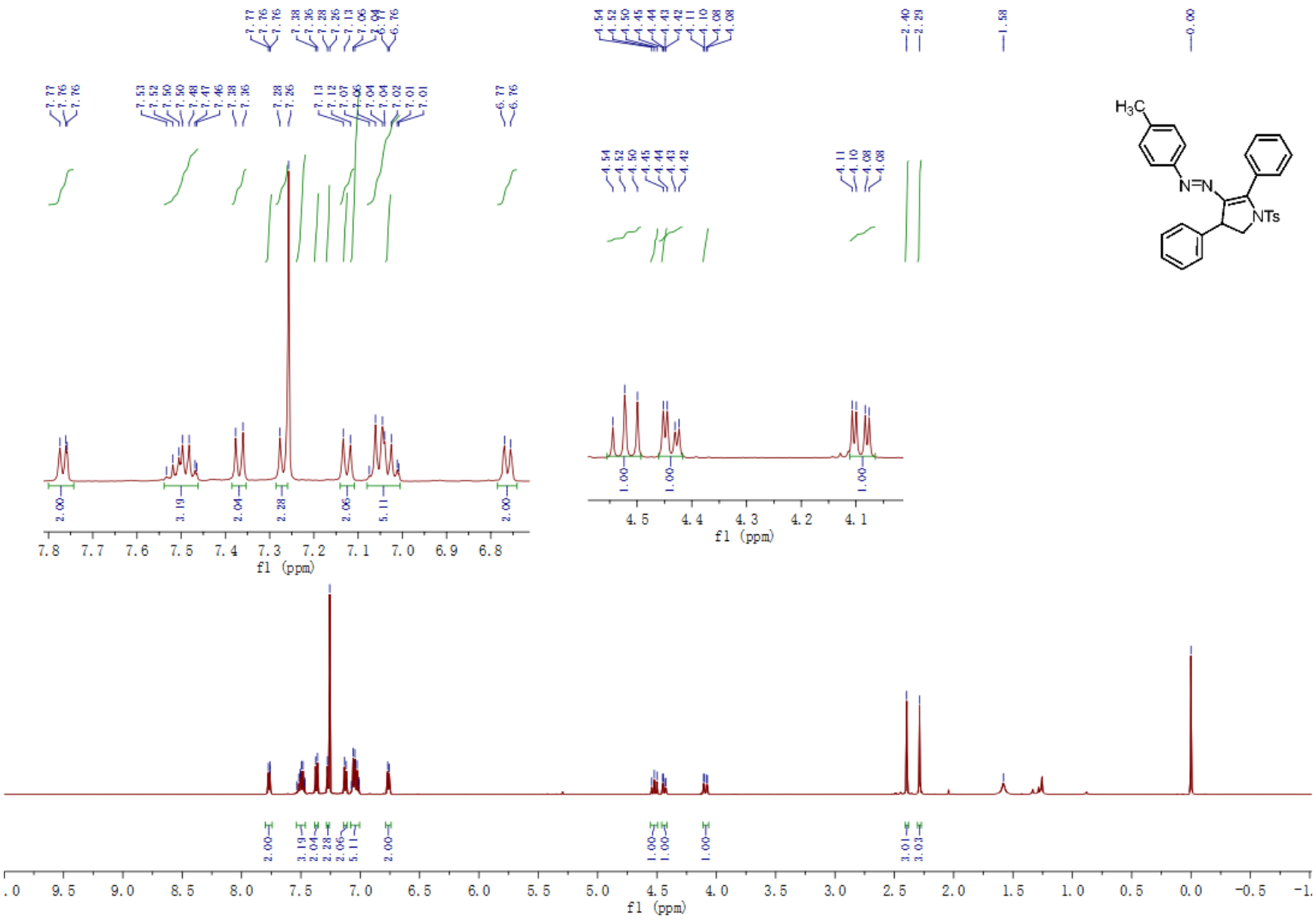

155.20
153.87
144.82
144.38
142.04
134.30
132.94
131.83
130.81
129.94
129.34
128.48
127.97
127.39
126.98
126.49
122.86
118.90
112.26
77.41
77.16
76.91
59.34
43.88
21.77
132.94
131.83
130.81
129.94
129.34
128.48
127.97
127.39
126.98
126.49
NC
N=N
NTs
133
132
131
130
129
128
127
126
f1 (ppm)
.90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

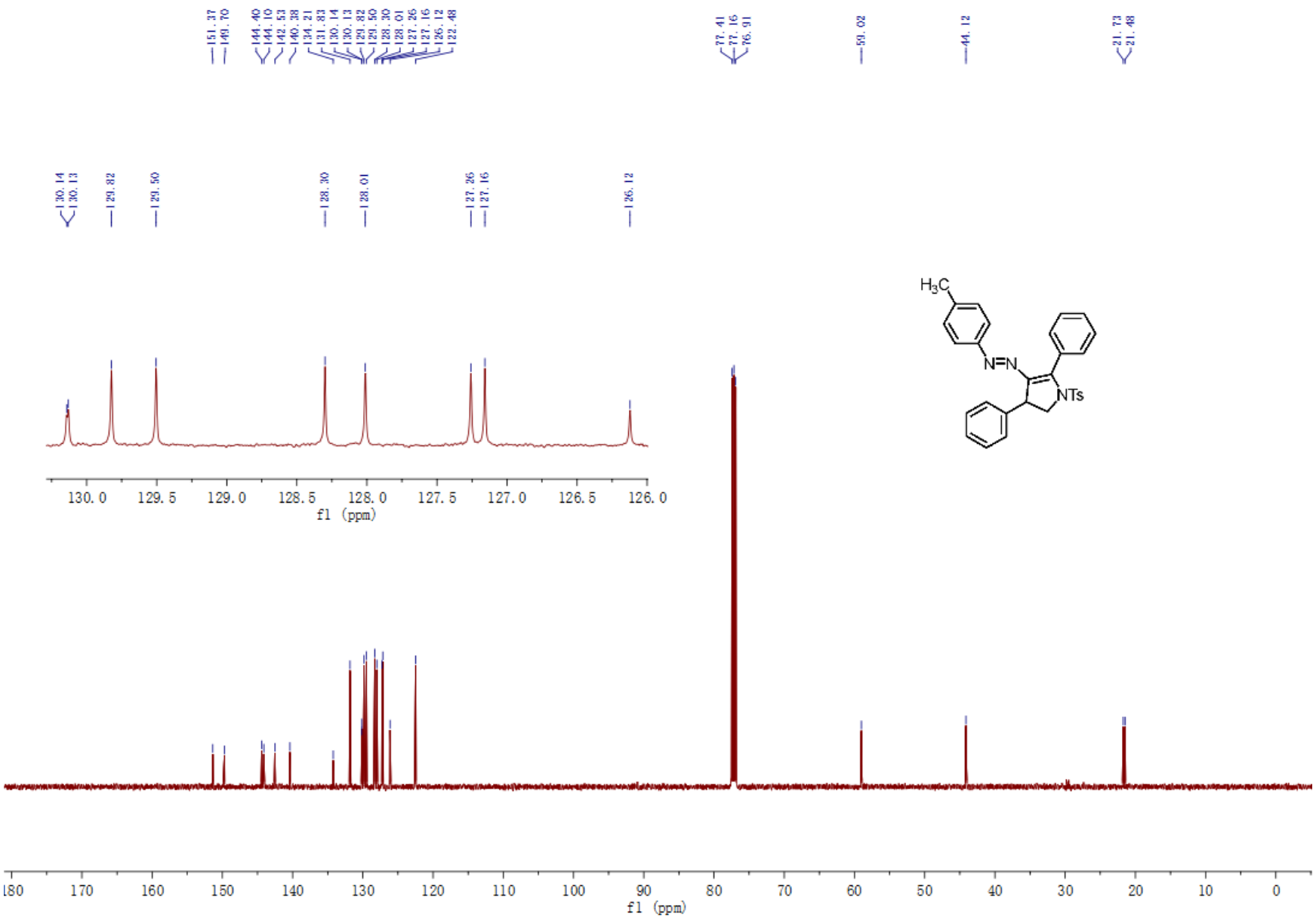

8.11
8.11
8.10
8.09
7.71
7.51
7.44
7.42
7.38
7.36
6.78
4.61
4.59
4.57
4.50
4.49
4.48
4.47
4.16
4.15
4.14
4.13
2.42
1.54
0.00
O2N
N=N
NTs
1.95
1.97
1.01
2.06
2.03
2.02
2.00
2.96
1.98
1.00
0.99
1.00
3.01
f1 (ppm)

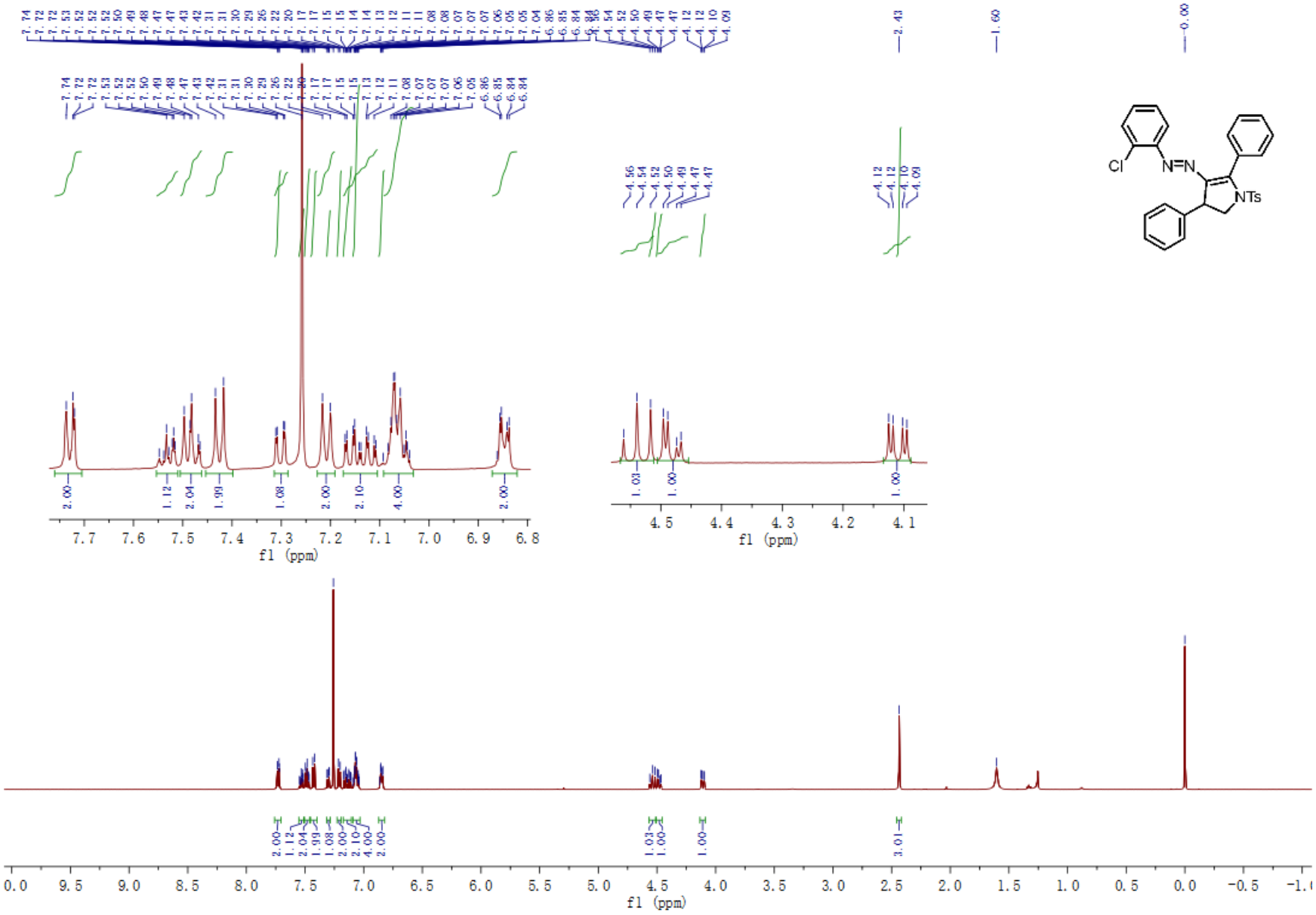

156.54
154.56
147.60
144.89
144.62
141.98
134.37
131.86
130.91
129.97
129.25
128.52
127.98
127.42
126.98
126.56
124.55
122.80
77.41
77.16
76.91
59.43
43.88
21.76
O2N
N=N
NTs
f1 (ppm)
f1 (ppm)

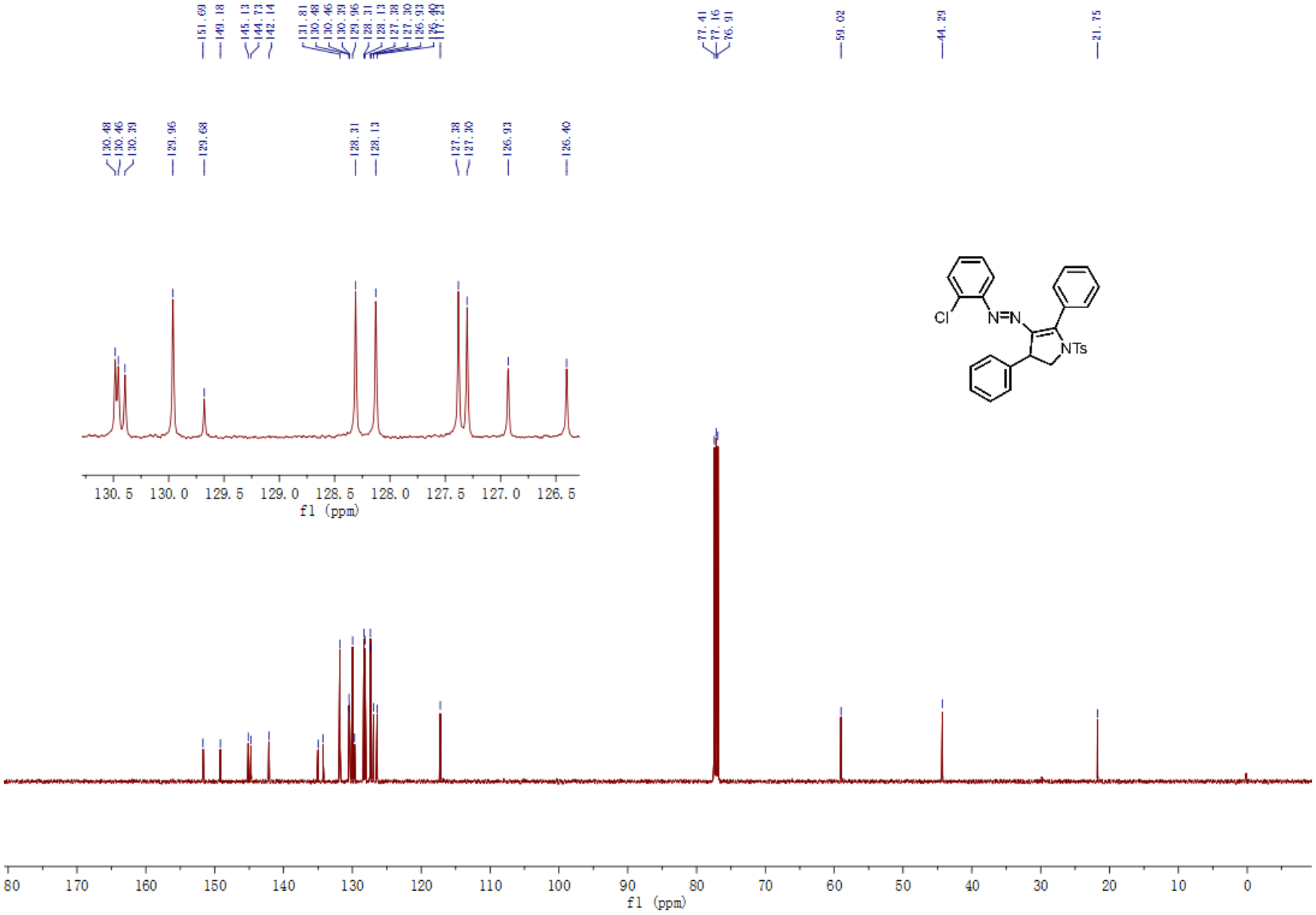

N=N
NTs
f1 (ppm)

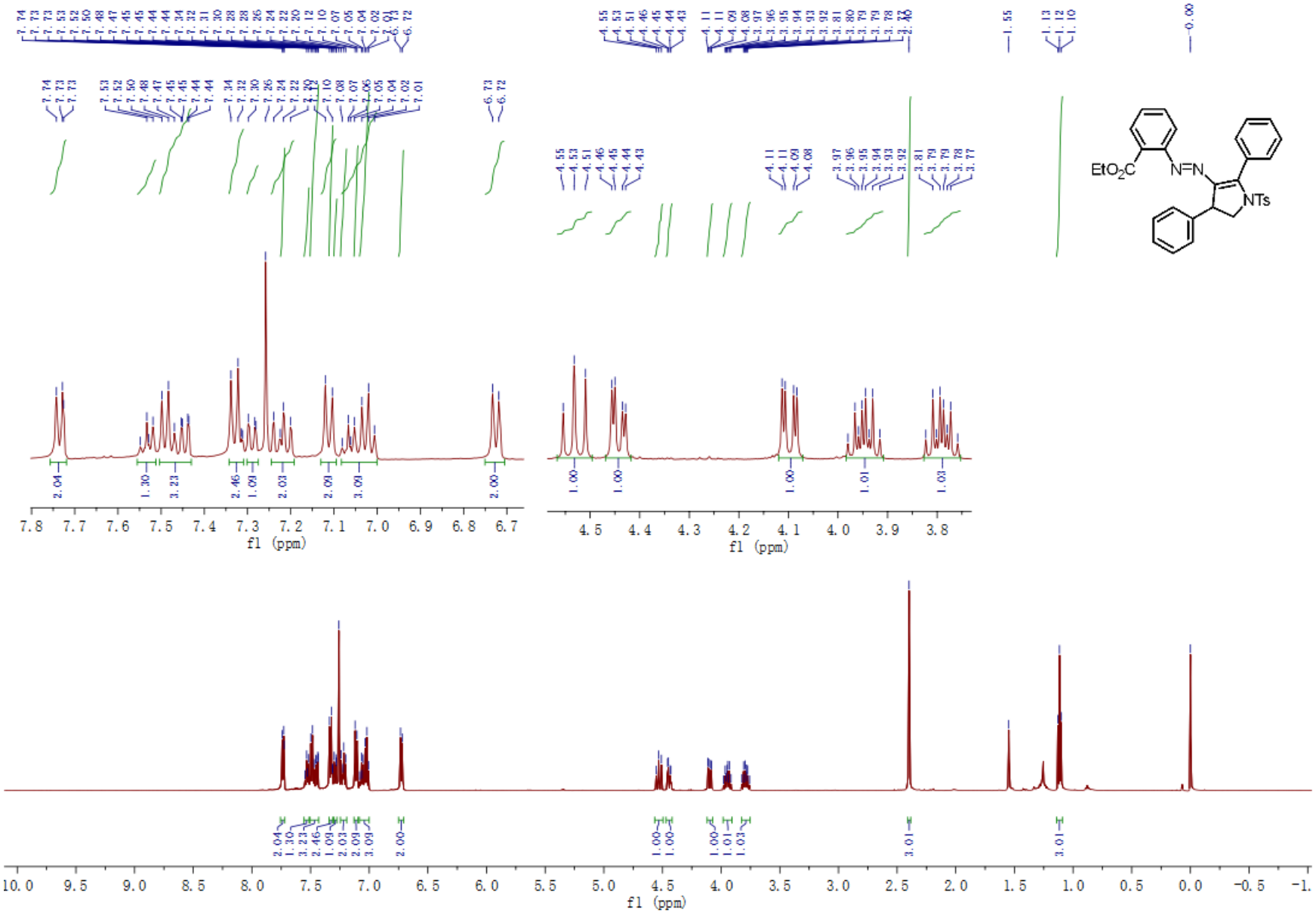

153.22
150.59
144.49
144.09
142.48
134.27
131.85
130.26
130.01
129.92
129.86
128.85
128.35
128.03
127.30
127.15
126.21
122.52
77.41
77.36
77.16
76.91
59.09
44.08
21.74
130.26
130.01
129.92
129.86
128.85
128.35
128.03
127.30
127.15
126.21
N=N
NTs
130.5
130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

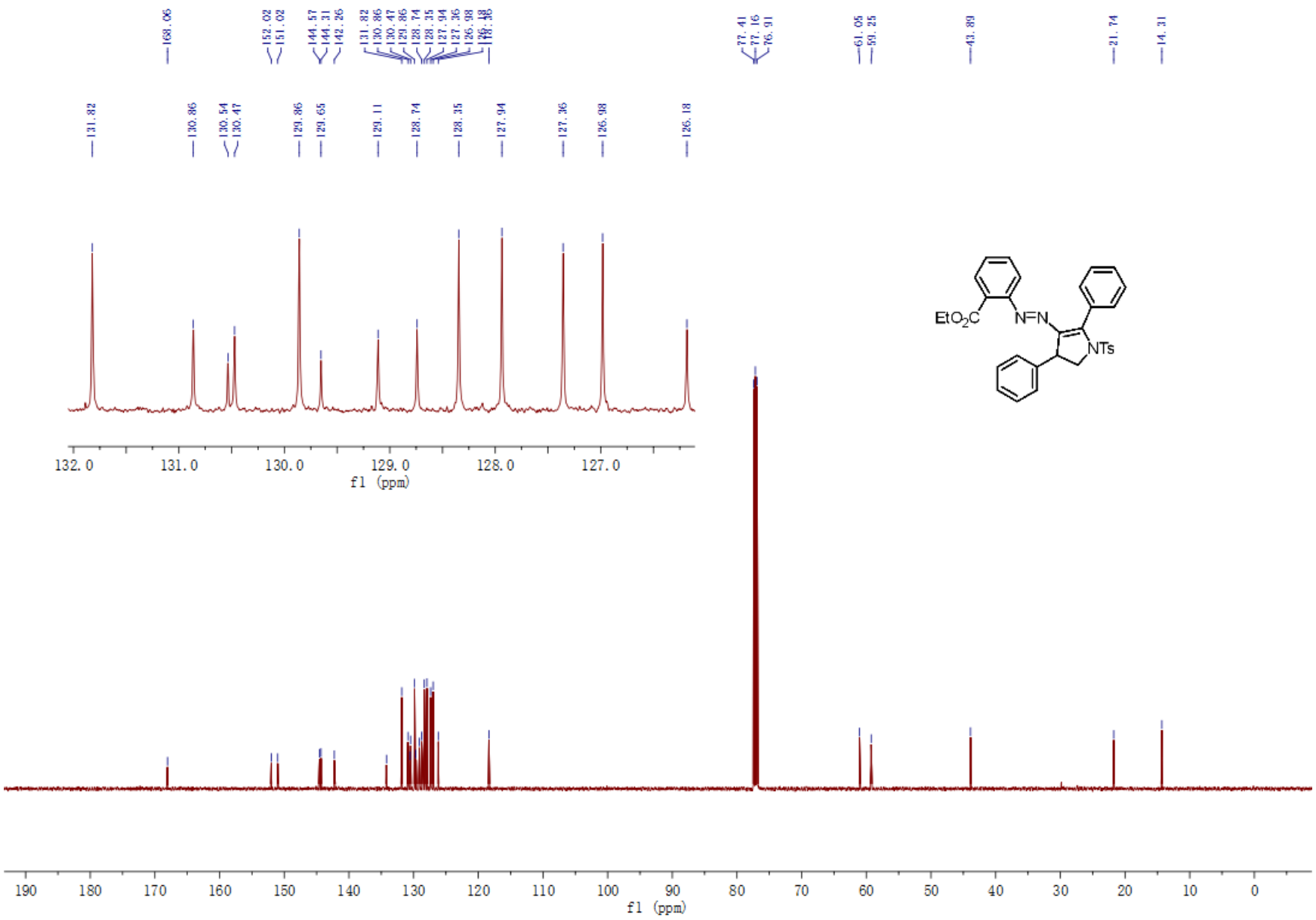

7.77
7.76
7.76
7.38
7.36
7.28
7.25
7.13
7.06
7.04
6.77
6.76
4.54
4.52
4.50
4.45
4.44
4.43
4.42
4.11
4.10
4.08
4.08
2.40
2.29
1.58
0.00
H3C
N=N
NTs
2.00
3.19
2.04
2.28
2.06
5.11
2.00
1.00
1.00
1.00
3.01
3.03
f1 (ppm)

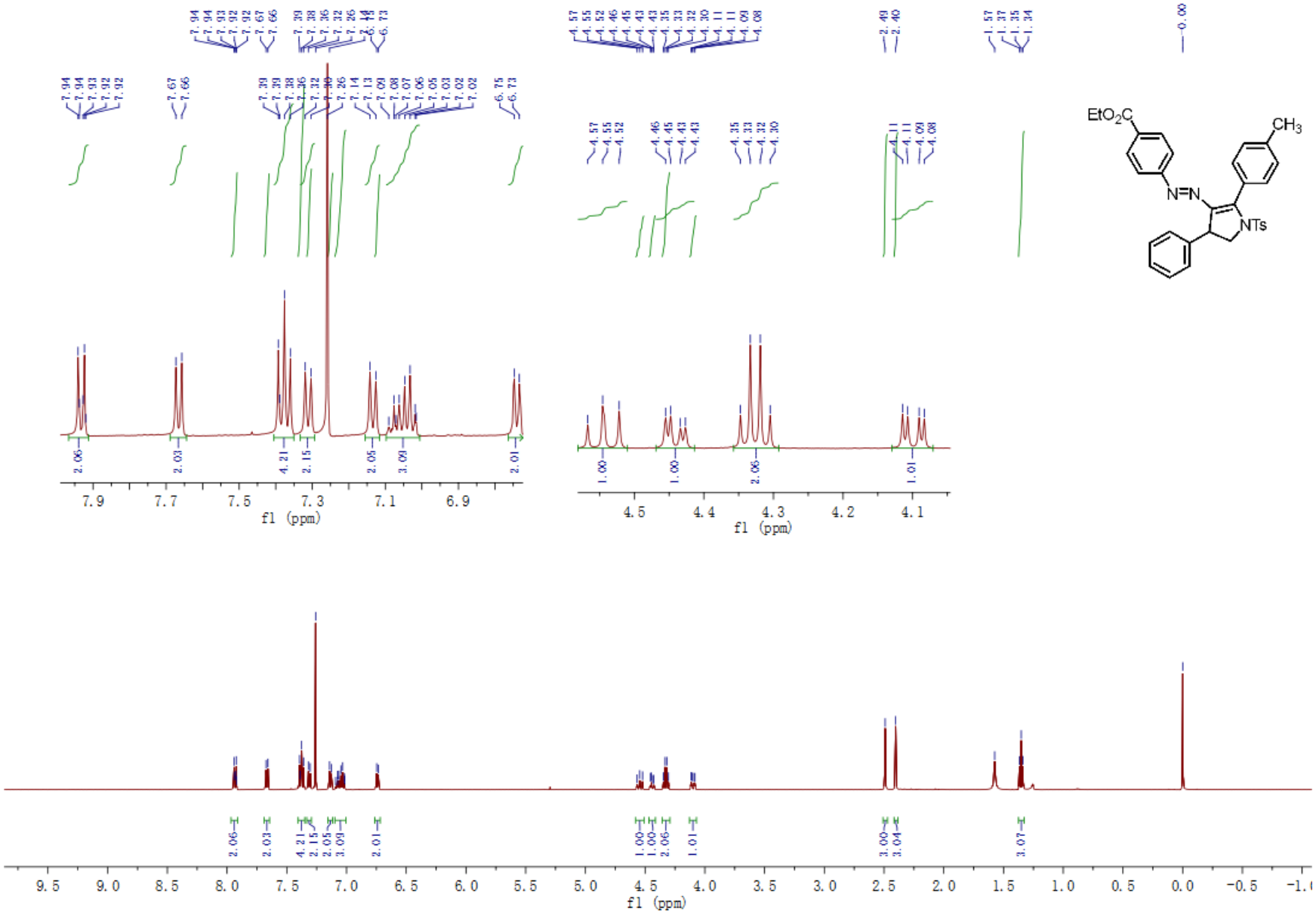

151.37
149.70
144.40
144.10
142.53
140.38
134.21
131.83
130.14
130.13
129.82
129.50
128.30
128.01
127.26
127.16
126.12
122.48
77.41
77.16
76.91
59.02
44.12
21.73
21.48
130.14
130.13
129.82
129.50
128.30
128.01
127.26
127.16
126.12
H3C
N=N
NTs
130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
126.0
f1 (ppm)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

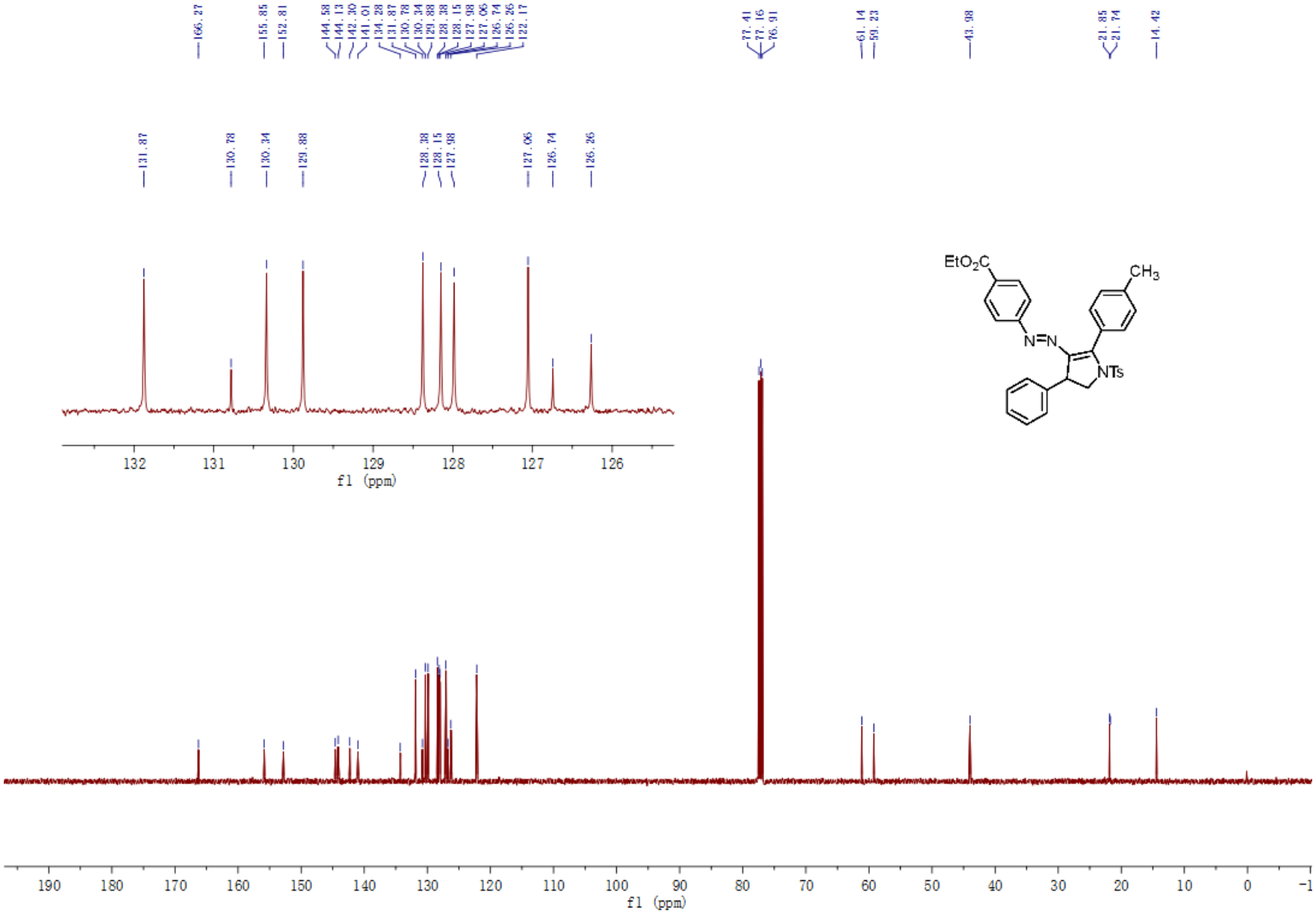

Cl
N=N
NTs
f1 (ppm)

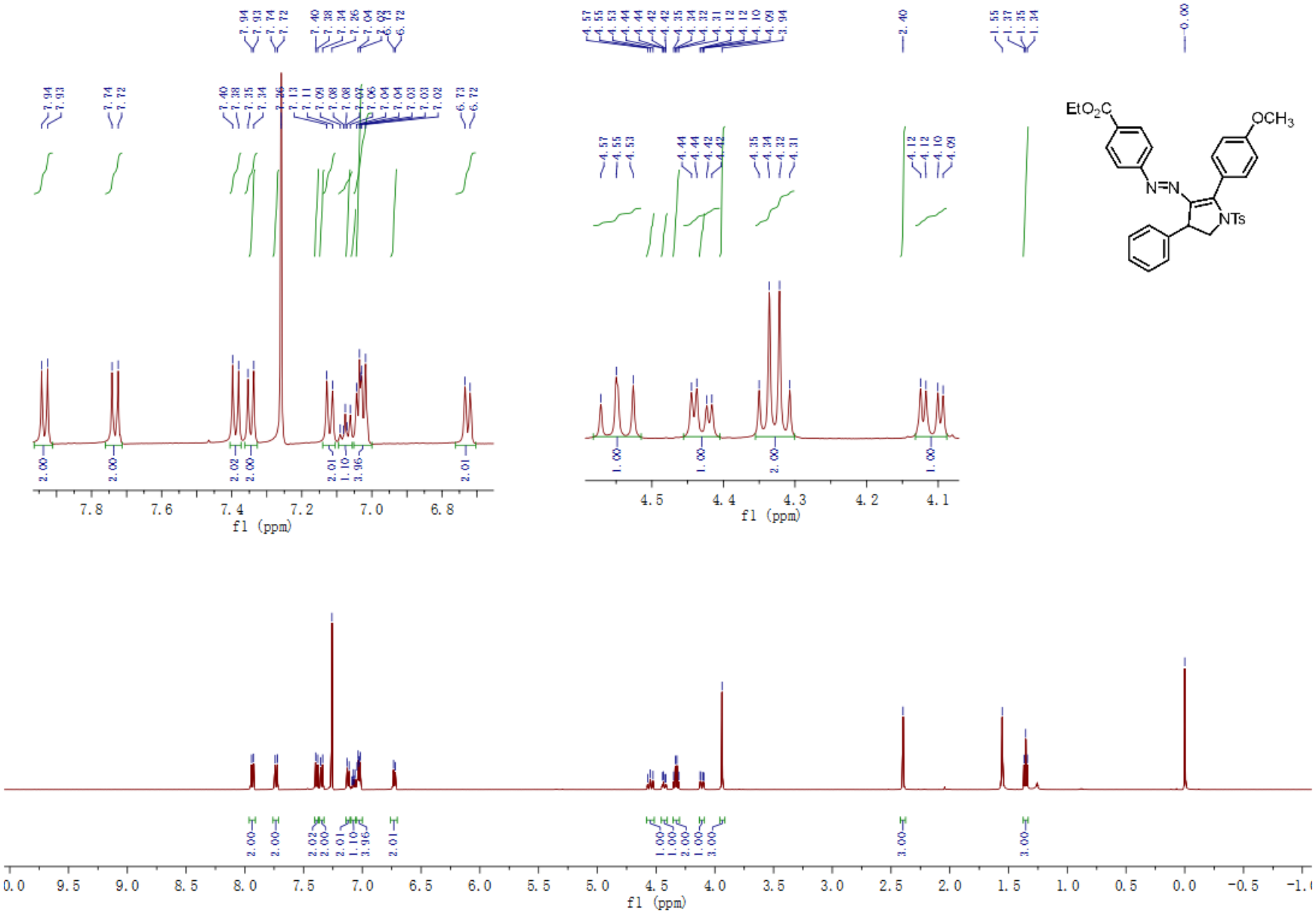

151.69
149.18
145.13
144.73
142.14
131.81
130.48
130.46
130.39
129.96
128.31
128.13
127.38
127.30
126.93
126.40
77.41
77.16
76.91
59.02
44.29
21.75
Cl
N=N
NTs
130.5
130.0
129.5
129.0
128.5
128.0
127.5
127.0
126.5
f1 (ppm)
80
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

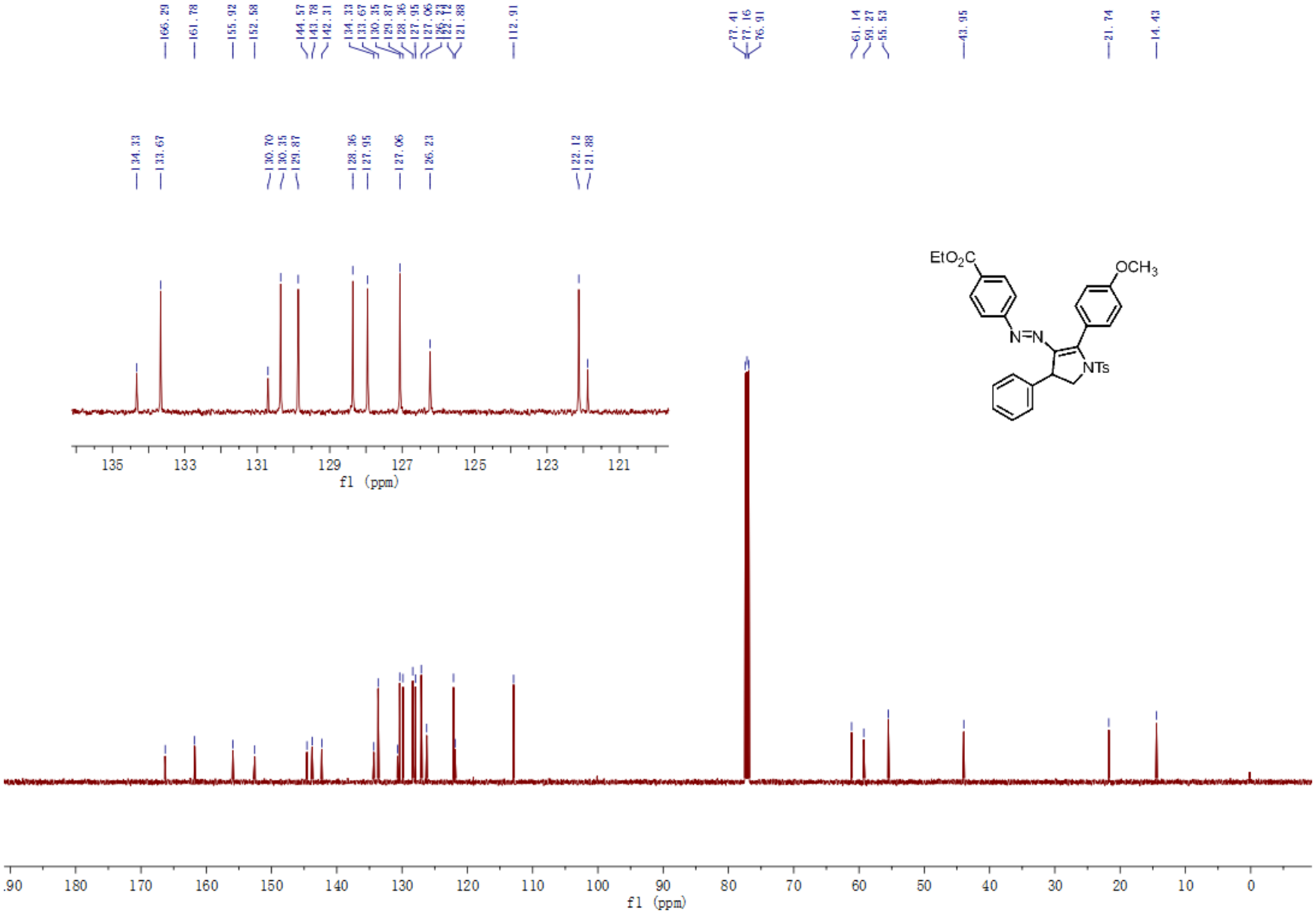

EtO2C
N=N
NTs
f1 (ppm)

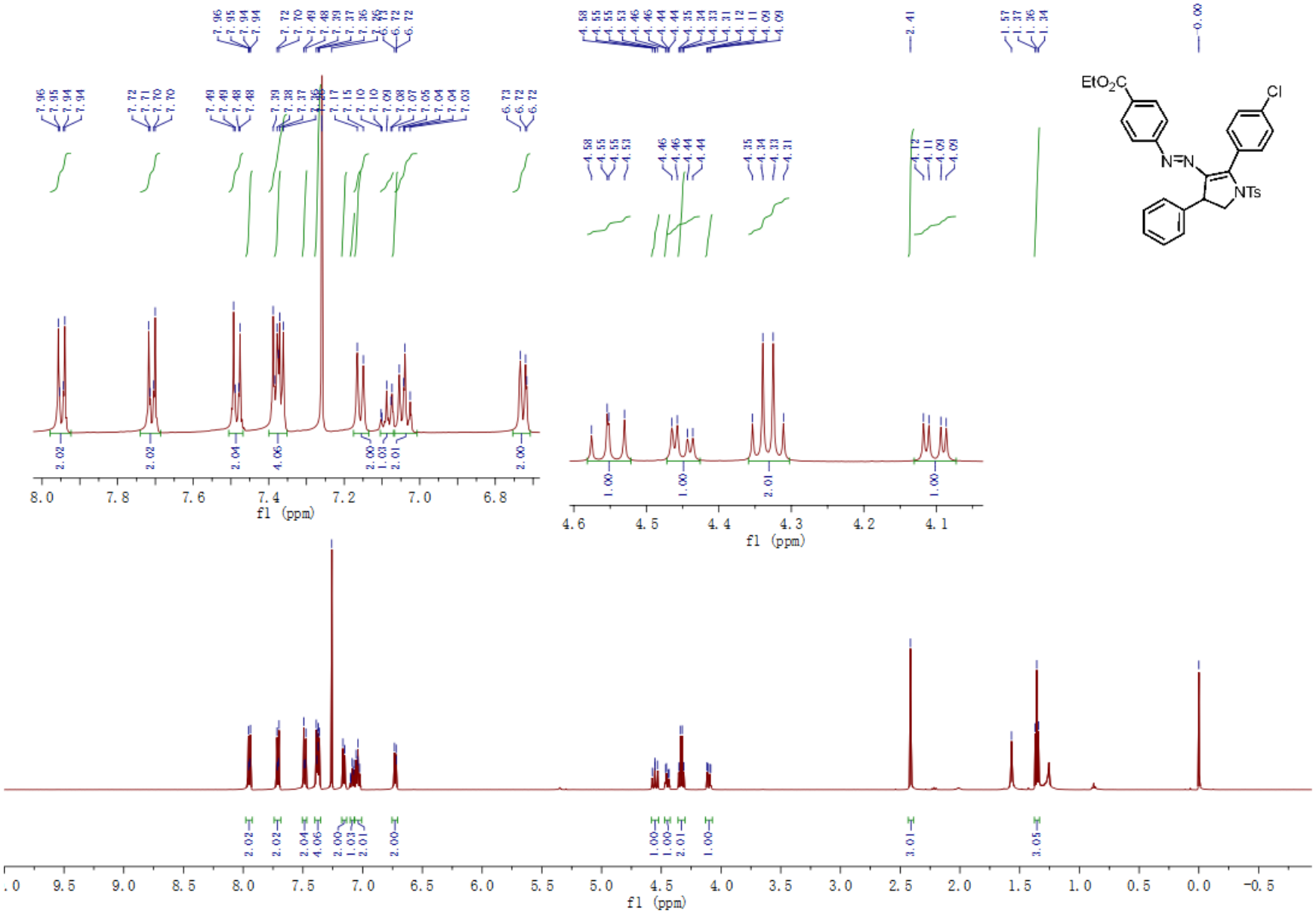

168.06
152.02
151.02
144.57
144.31
142.26
131.82
130.86
130.47
129.86
128.74
128.35
127.94
127.36
126.98
77.41
77.16
76.91
61.05
59.25
43.89
21.74
14.31
131.82
130.86
130.54
130.47
129.86
129.65
129.11
128.74
128.35
127.94
127.36
126.98
126.18
132.0
131.0
130.0
129.0
128.0
127.0
f1 (ppm)
EtO2C
N=N
NTs
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

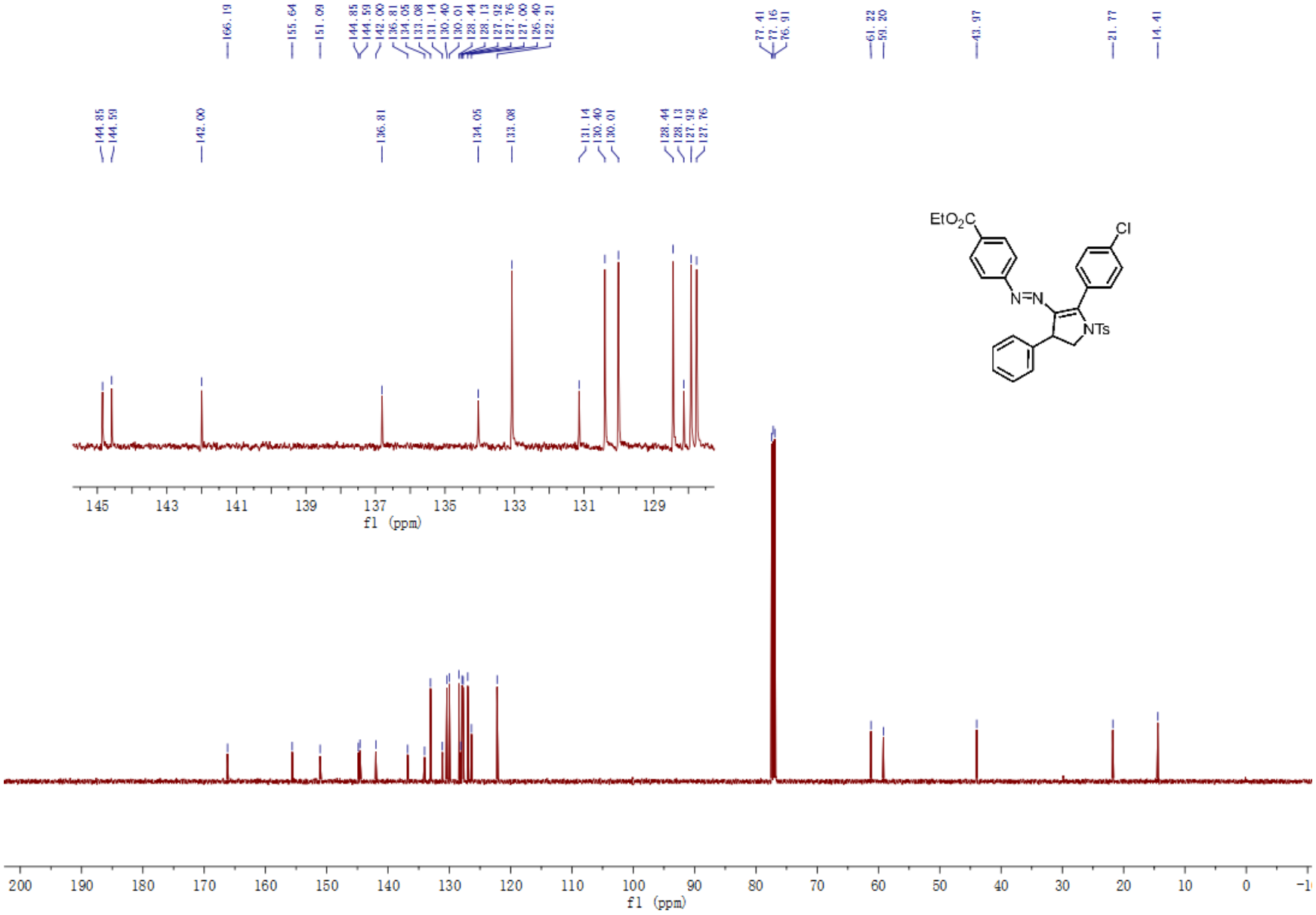

EtO2C
CH3
N=N
NTs
f1 (ppm)

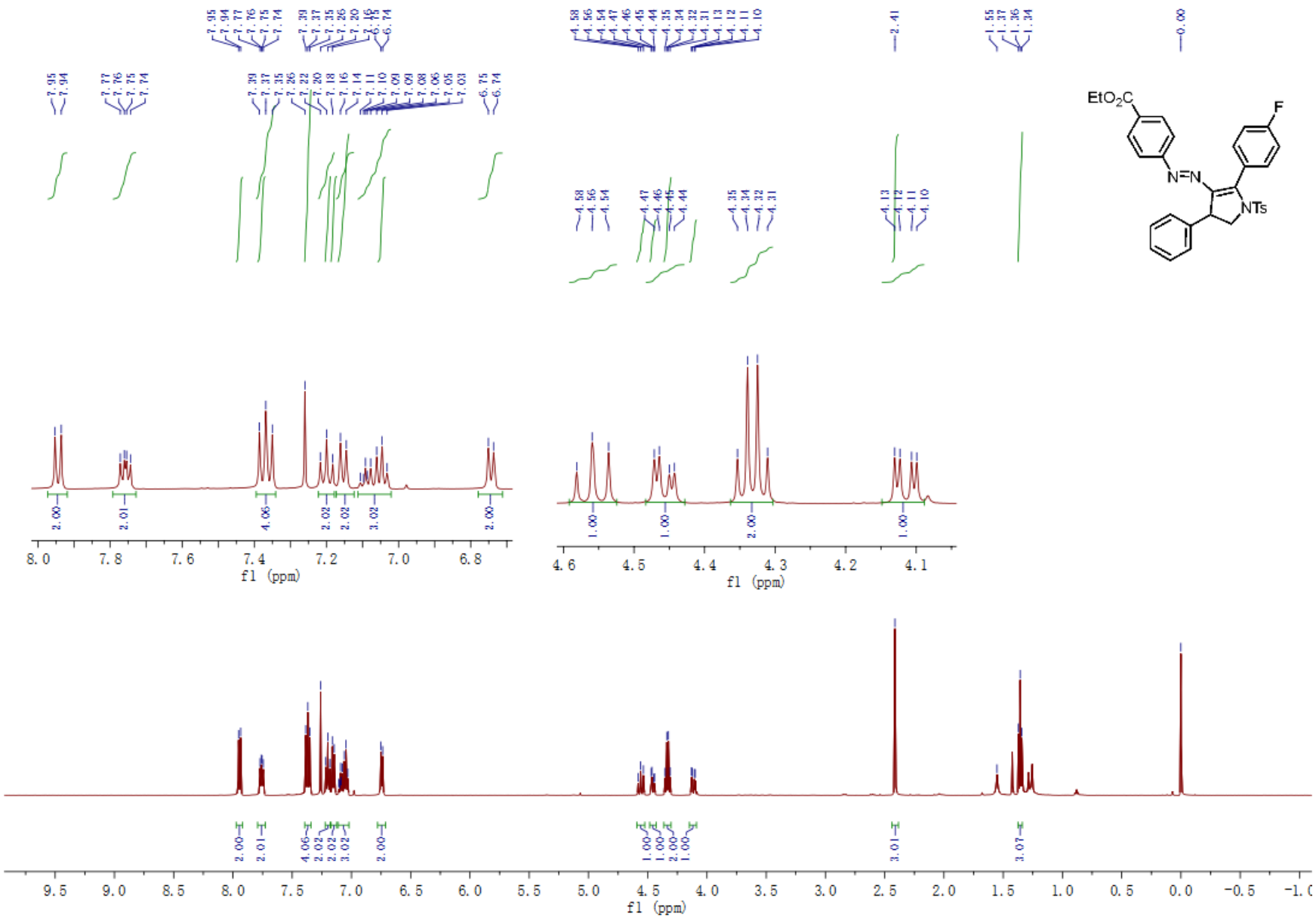

165.27
155.85
152.81
144.58
144.13
142.30
141.01
134.28
131.87
130.78
130.34
129.88
128.38
128.15
127.98
127.06
126.74
126.26
122.17
77.41
77.16
76.91
61.14
59.23
43.98
21.85
21.74
14.42
131.87
130.78
130.34
129.88
128.38
128.15
127.98
127.06
126.74
126.26
132
131
130
129
128
127
126
f1 (ppm)
EtO2C
CH3
N=N
NTs
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1
f1 (ppm)

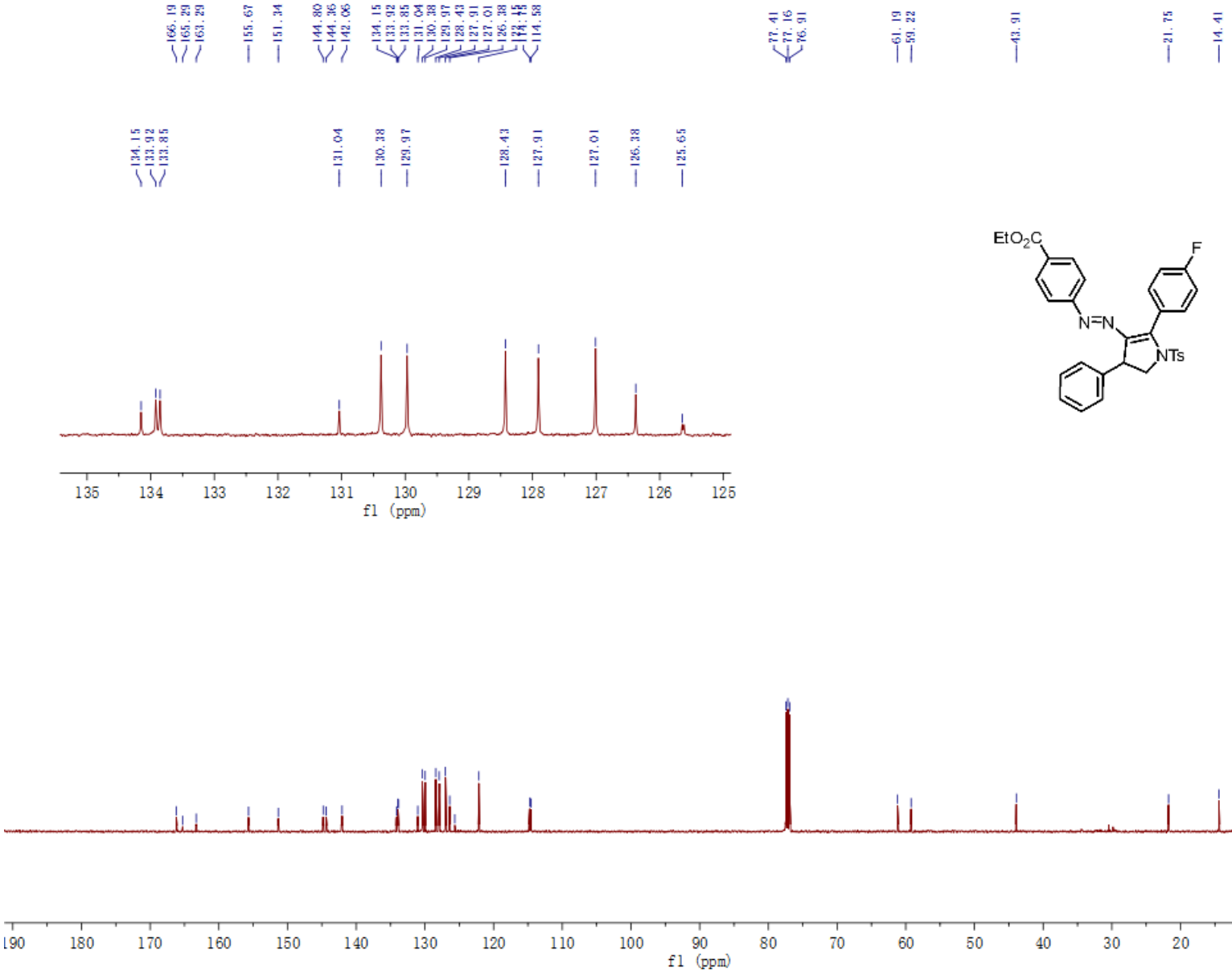

EtO2C
OCH3
N=N
NTs
f1 (ppm)

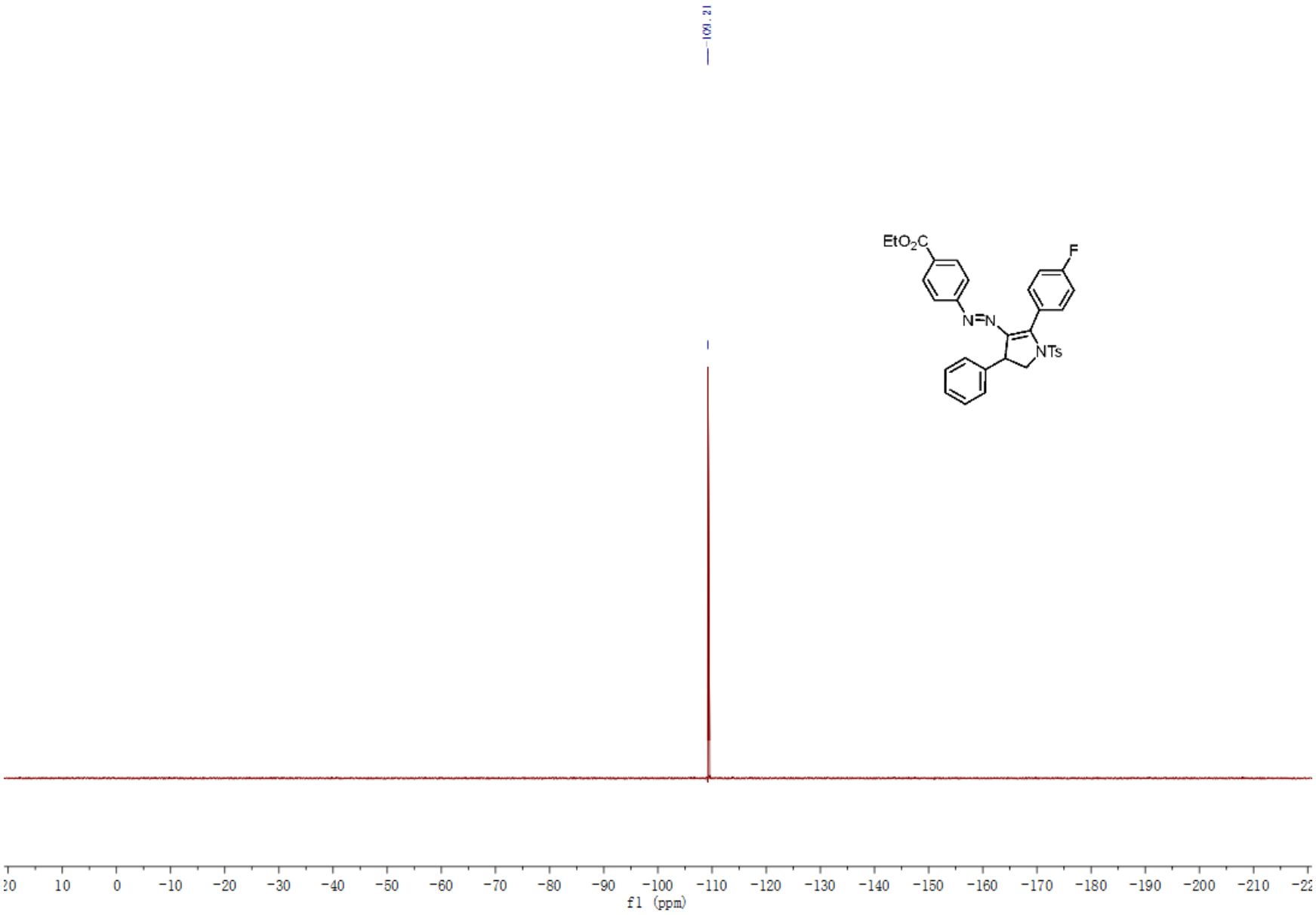

166.29
161.78
155.92
152.58
144.57
143.78
142.31
134.33
133.67
130.35
129.87
128.36
127.95
127.06
126.23
122.12
121.88
112.91
77.41
77.16
76.91
61.14
59.27
55.53
43.95
21.74
14.43
EtO2C
OCH3
N=N
NTs
f1 (ppm)
f1 (ppm)

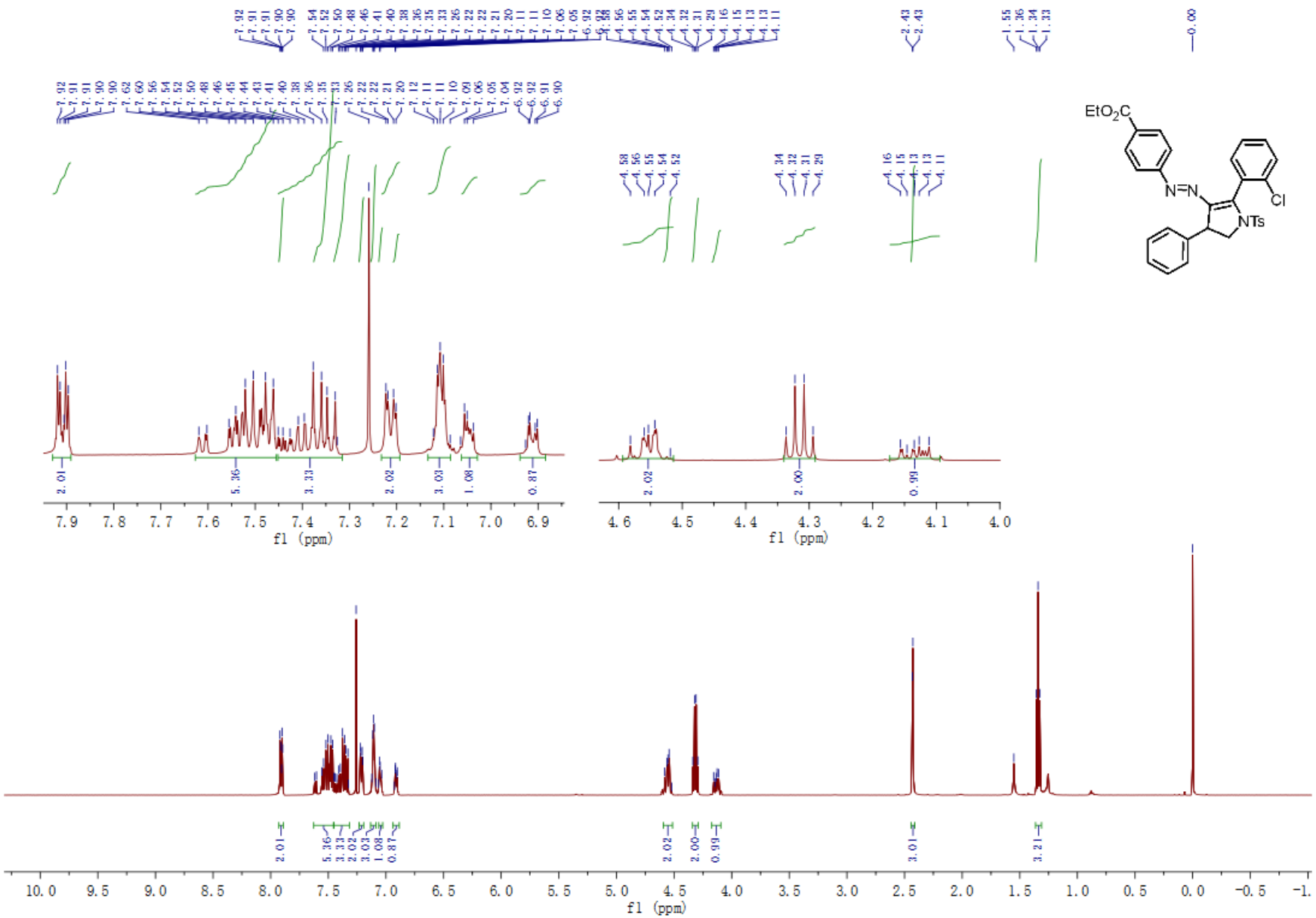

EtO2C
Cl
N=N
NTs
f1 (ppm)

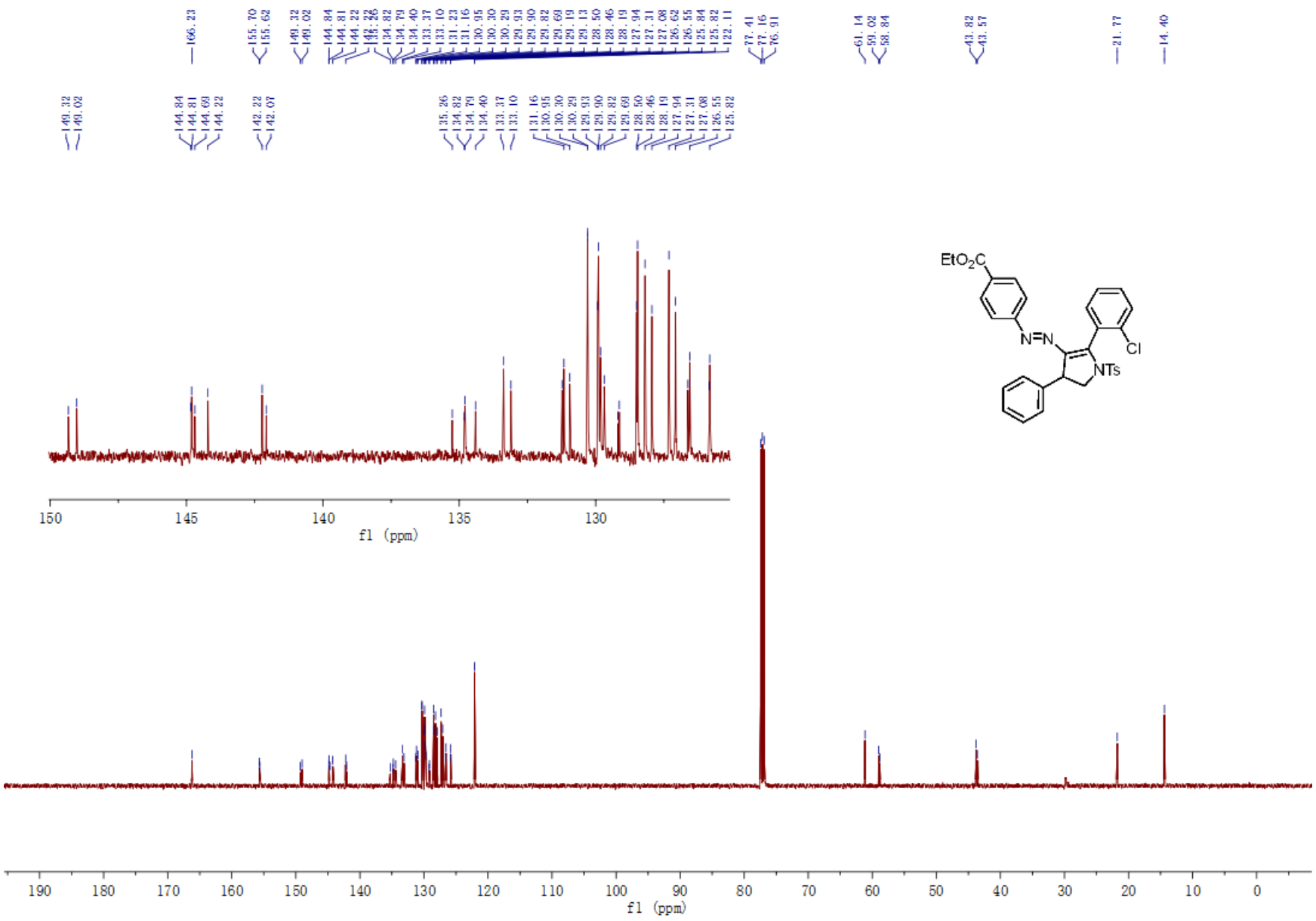

166.19
155.64
151.09
144.85
144.59
142.00
136.81
134.05
133.08
131.14
130.40
130.01
128.44
128.13
127.92
127.76
127.00
126.40
122.21
77.41
77.16
76.91
61.22
59.20
43.97
21.77
14.41
EtO2C
Cl
N=N
NTs
f1 (ppm)
f1 (ppm)


EtO2C
F
N=N
NTs
f1 (ppm)
f1 (ppm)
f1 (ppm)


166.19
165.29
163.29
155.67
151.34
144.80
144.36
142.06
134.15
133.92
133.85
131.04
130.38
129.97
128.43
127.91
127.01
126.38
125.65
114.58
77.41
77.16
76.91
61.19
59.22
43.91
21.75
14.41
EtO2C
F
N=N
NTs
135
134
133
132
131
130
129
128
127
126
125
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


-109.21
EtO2C
F
N=N
NTs
f1 (ppm)


EtO2C
N=N
Cl
NTs
f1 (ppm)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155.62
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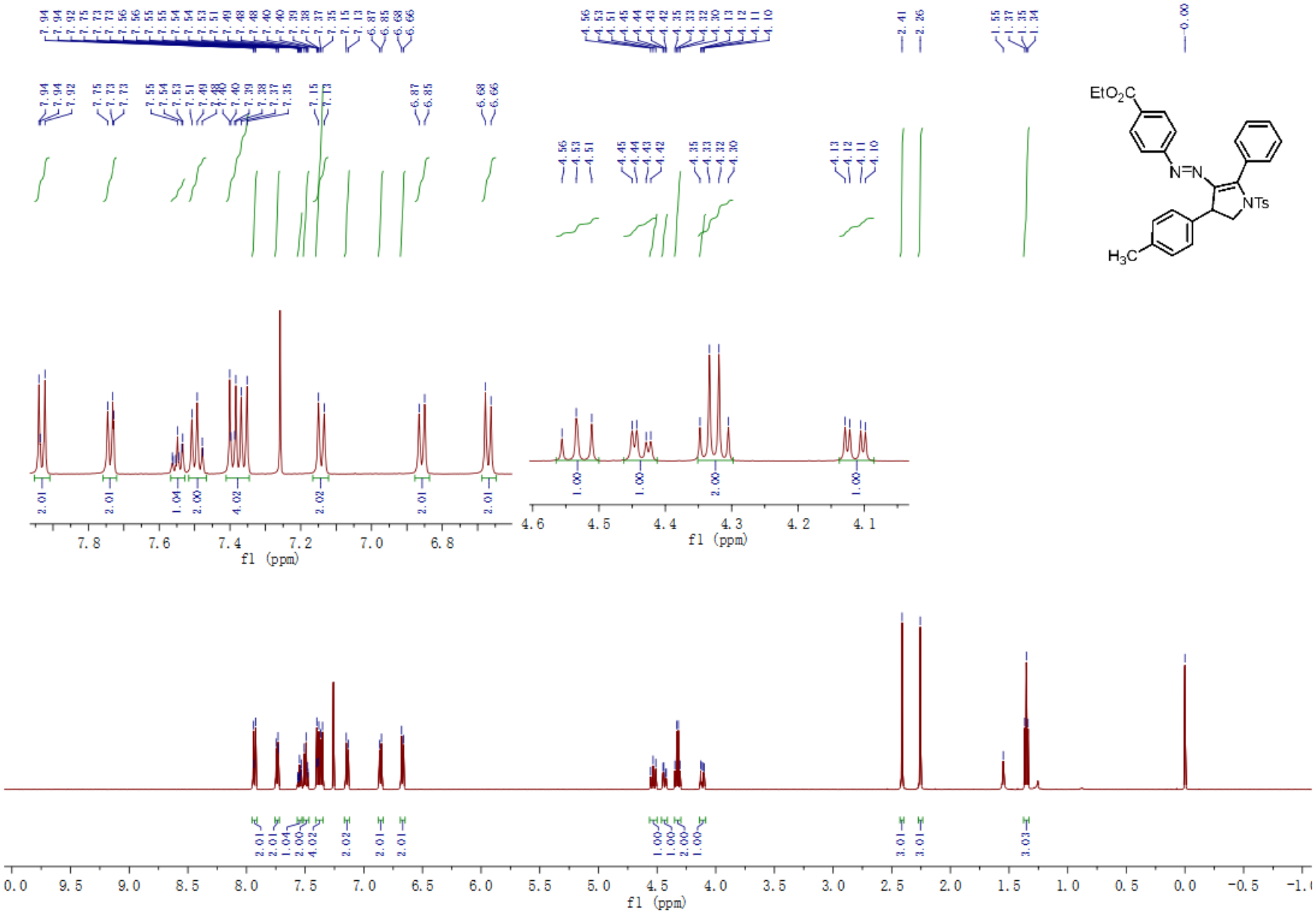
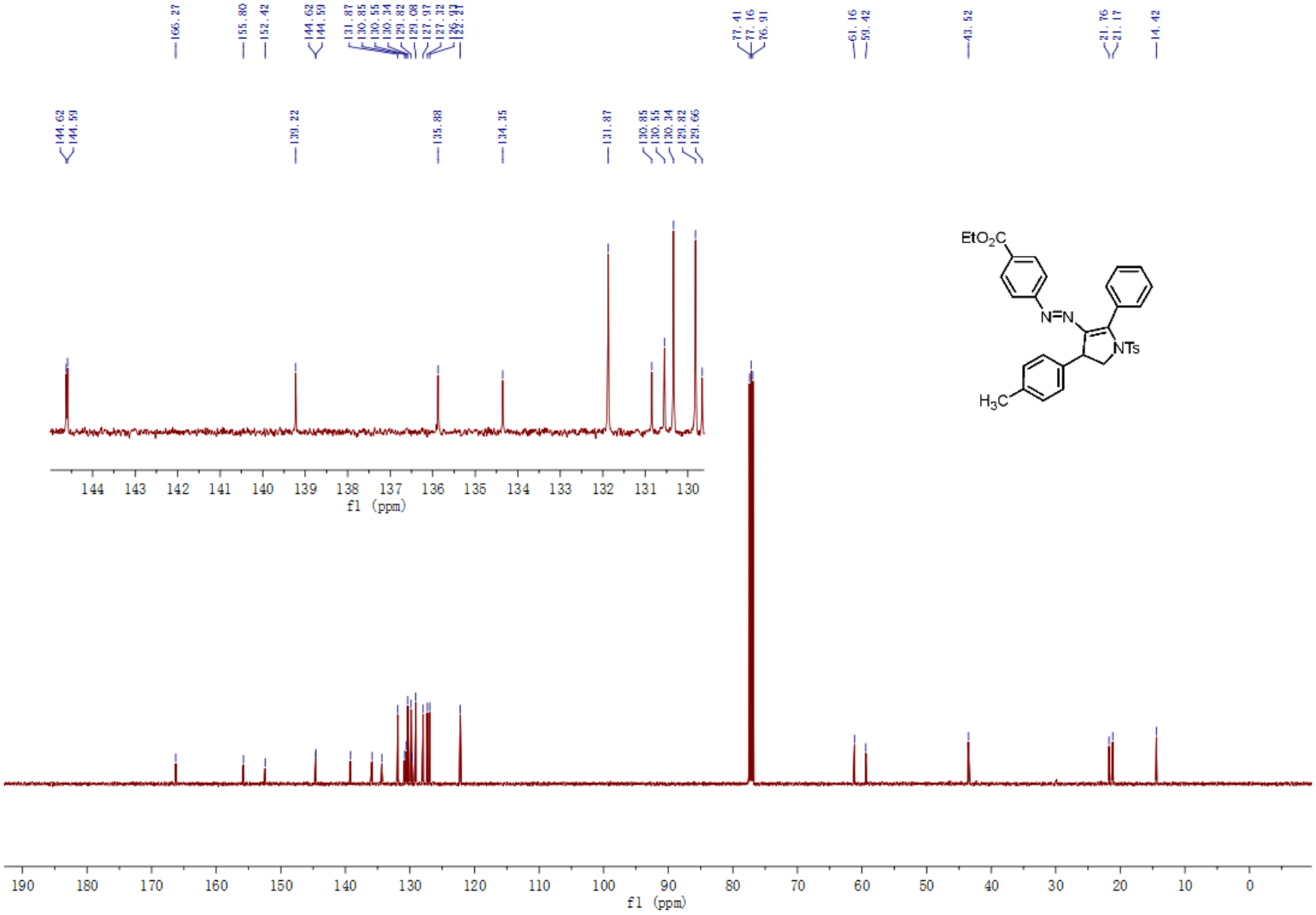
144.22
142.22
135.26
134.82
134.79
134.40
133.37
133.10
131.23
131.16
130.95
130.30
130.29
129.93
129.90
129.82
129.69
129.19
129.13
128.50
128.46
128.19
127.94
127.31
127.08
126.52
126.55
125.84
125.82
122.11
77.41
77.16
76.91
61.14
59.02
58.84
43.82
43.57
21.77
14.40
EtO2C
N=N
Cl
NTs
f1 (ppm)
f1 (ppm)


EtO2C
N=N
NTs
H3C
f1 (ppm)


166.27
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144.59
131.87
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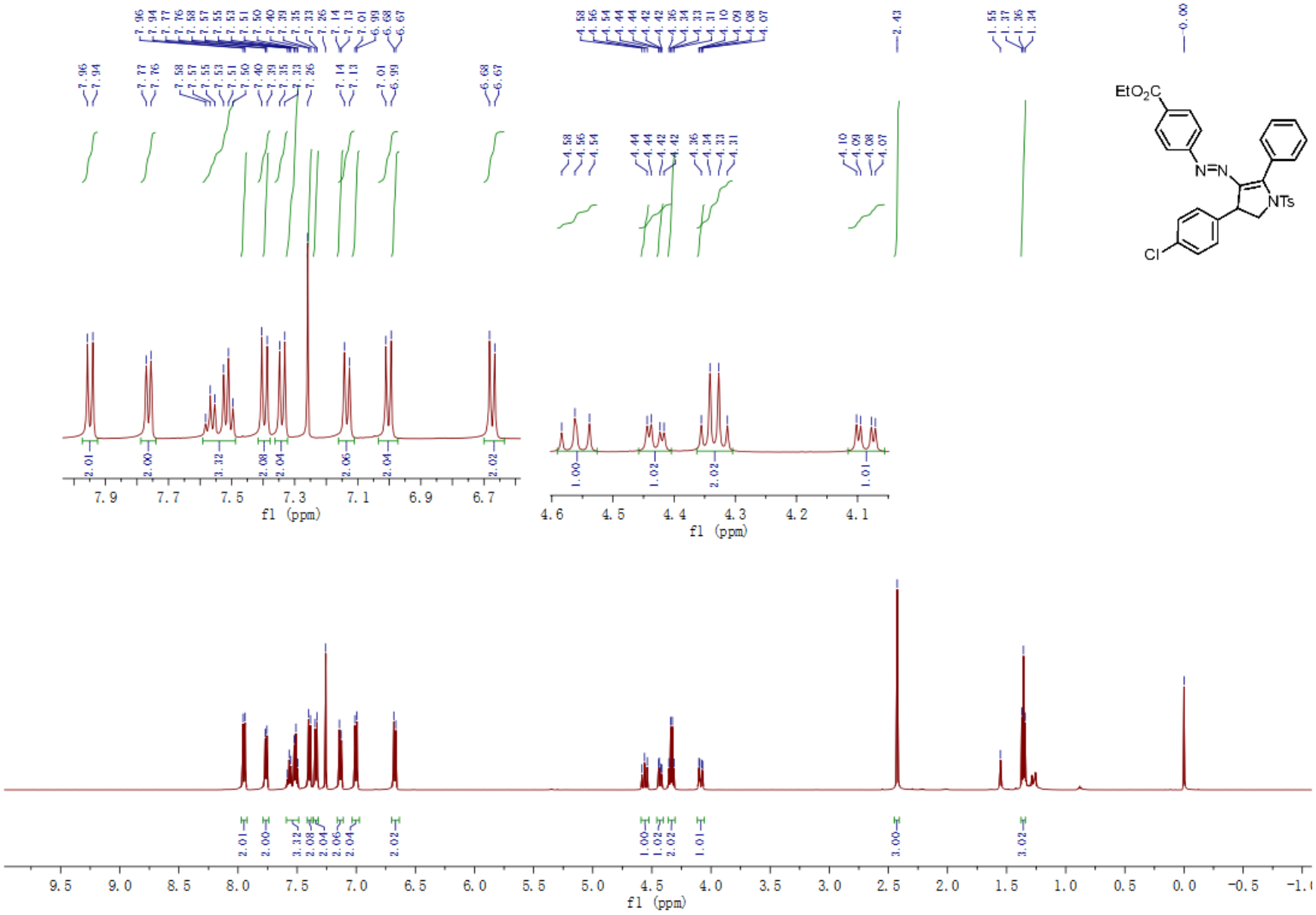
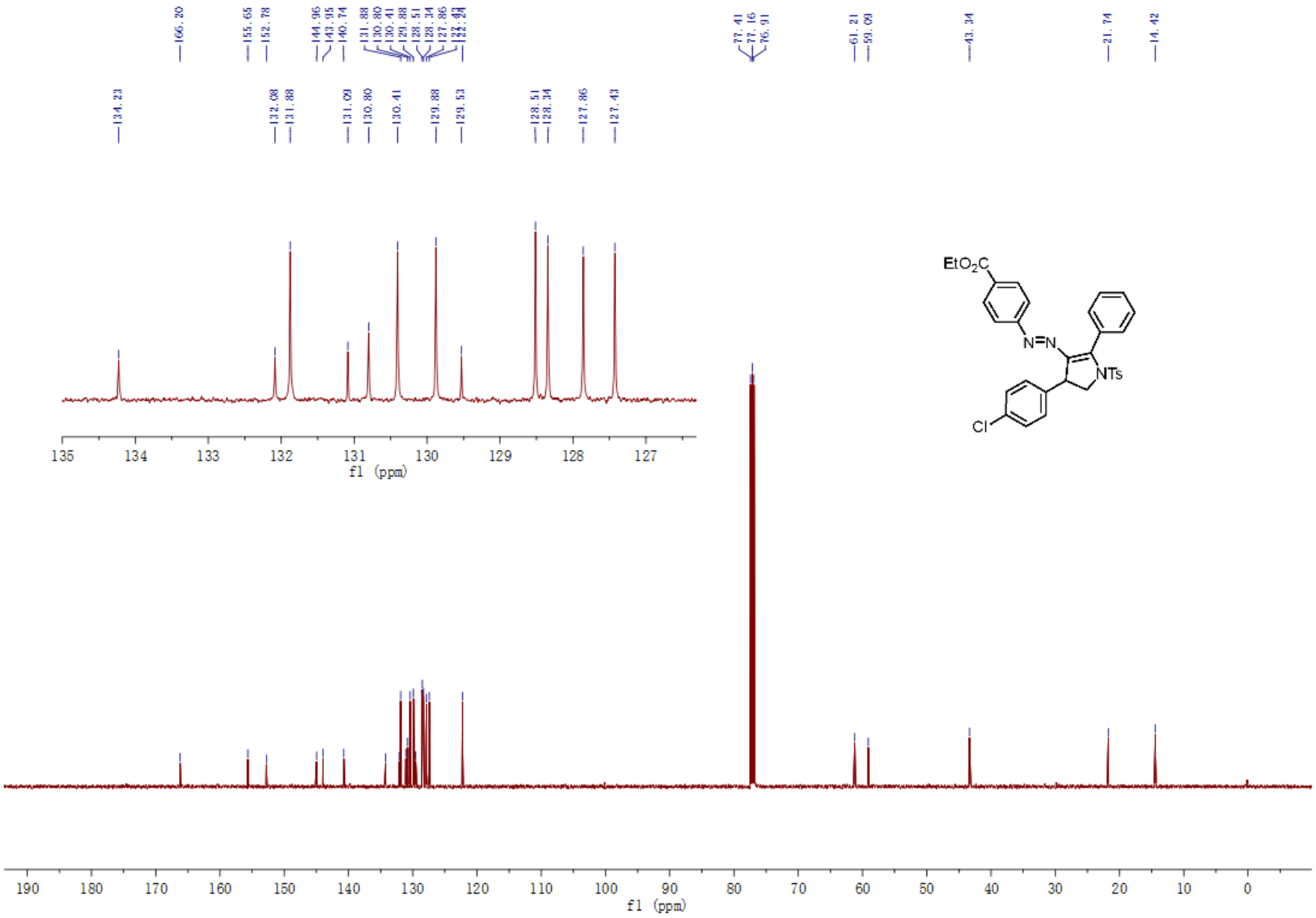
130.55
130.34
129.82
129.08
127.97
127.32
126.93
126.27
77.41
77.16
76.91
61.16
59.42
43.52
21.76
21.17
14.42
144.62
144.59
139.22
135.88
134.35
131.87
130.85
130.55
130.34
129.82
129.66
EtO2C
N=N
NTs
H3C
144 143 142 141 140 139 138 137 136 135 134 133 132 131 130
f1 (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


EtO2C
N=N
NTs
Cl
f1 (ppm)


166.20
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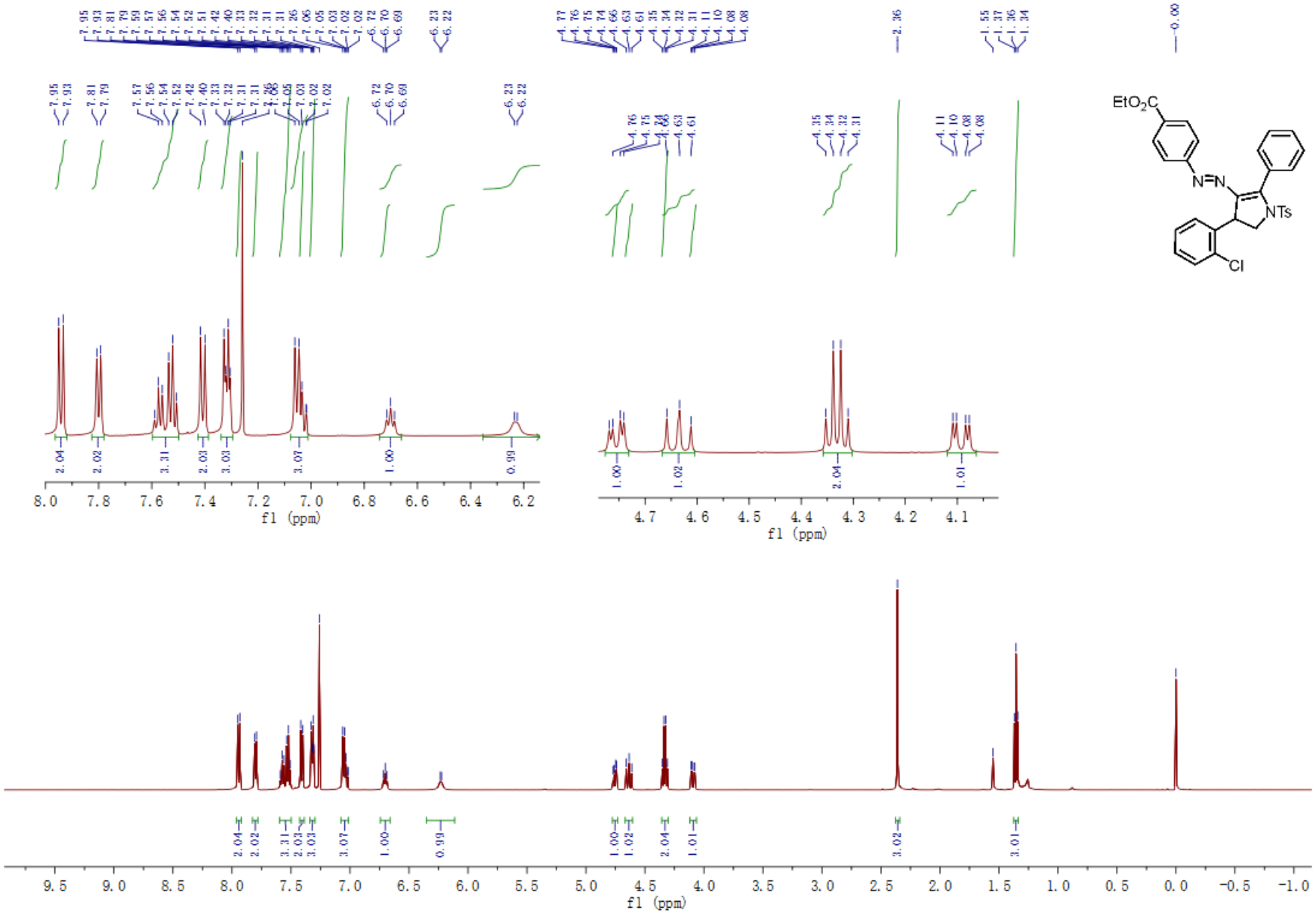
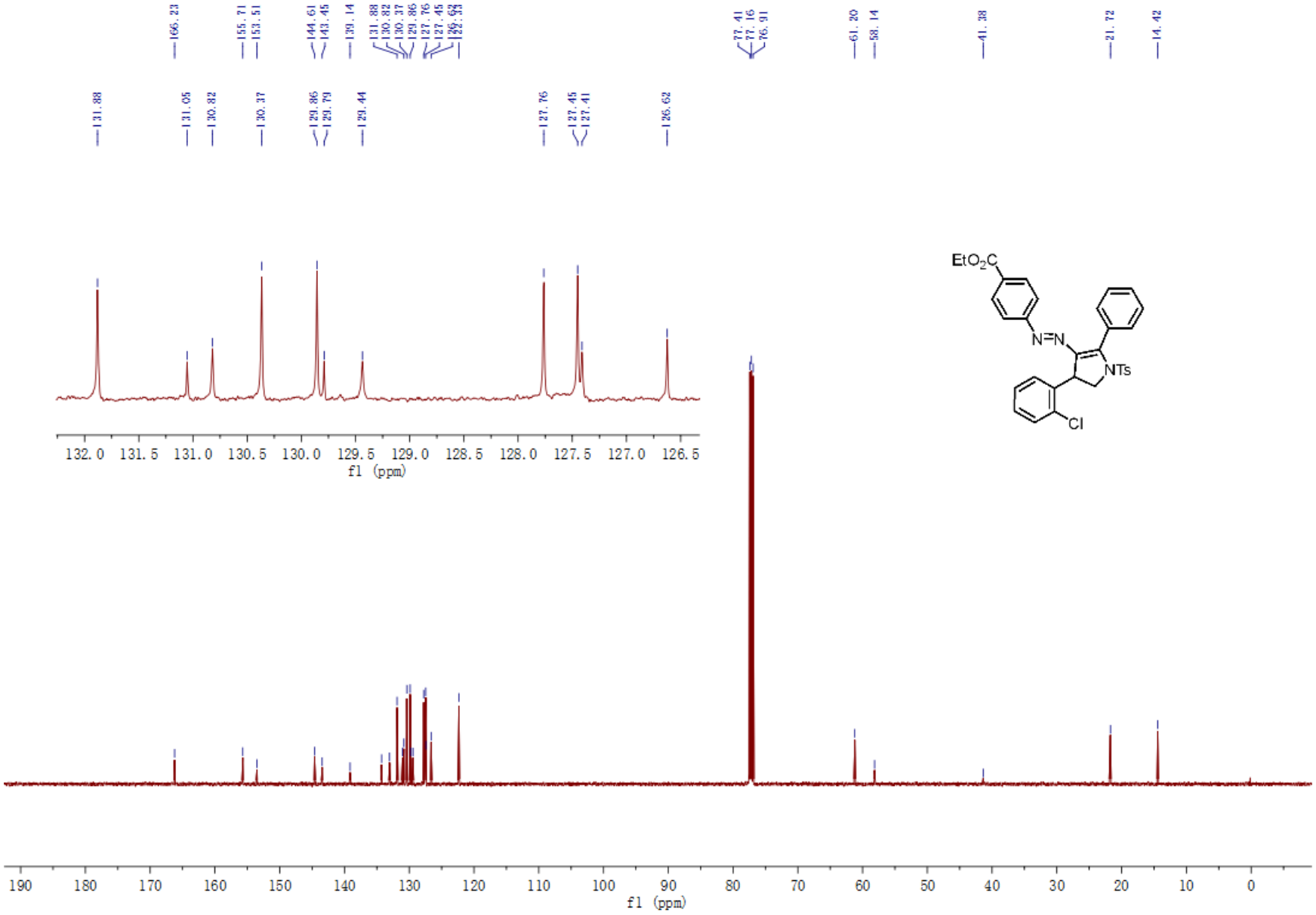
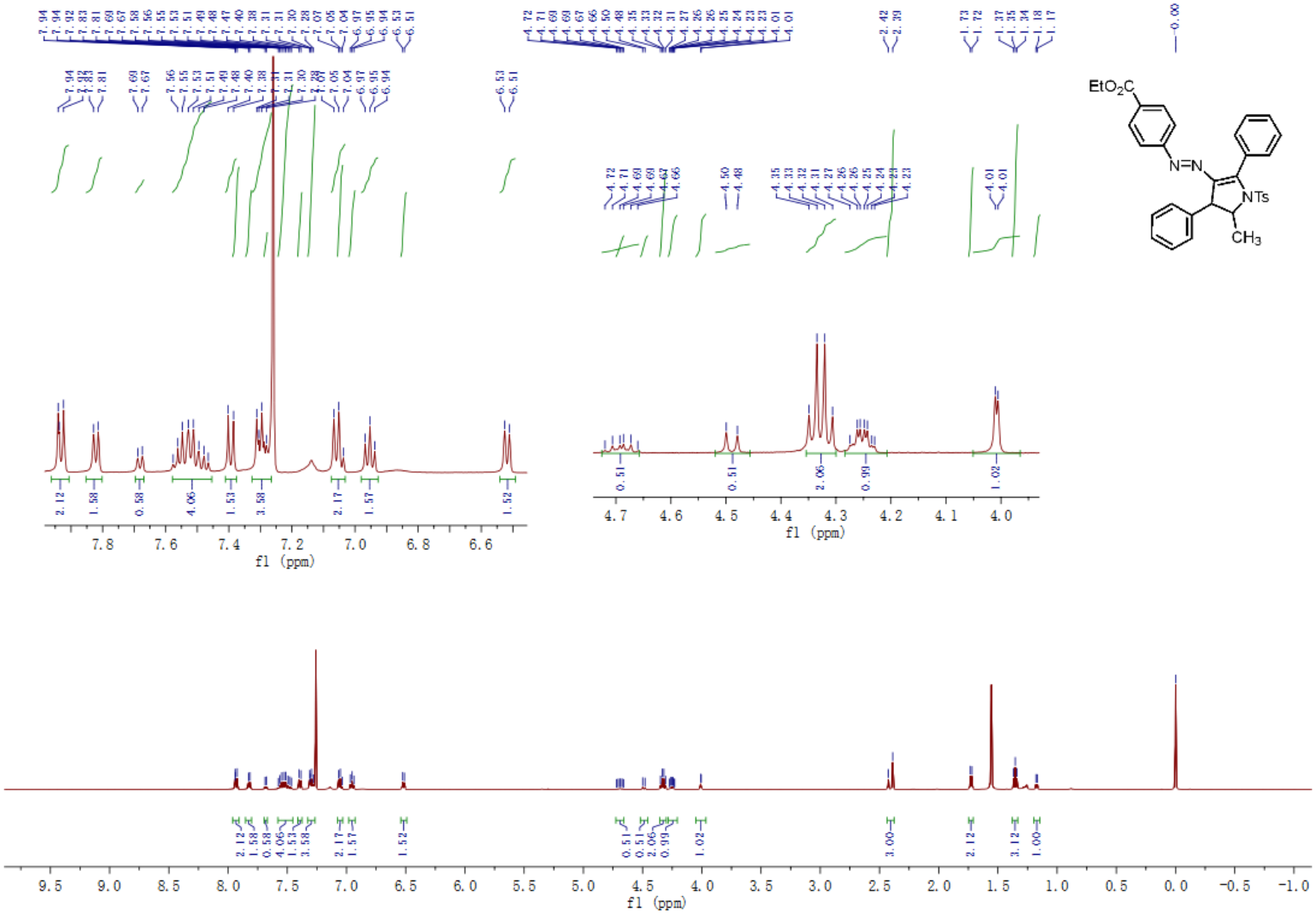
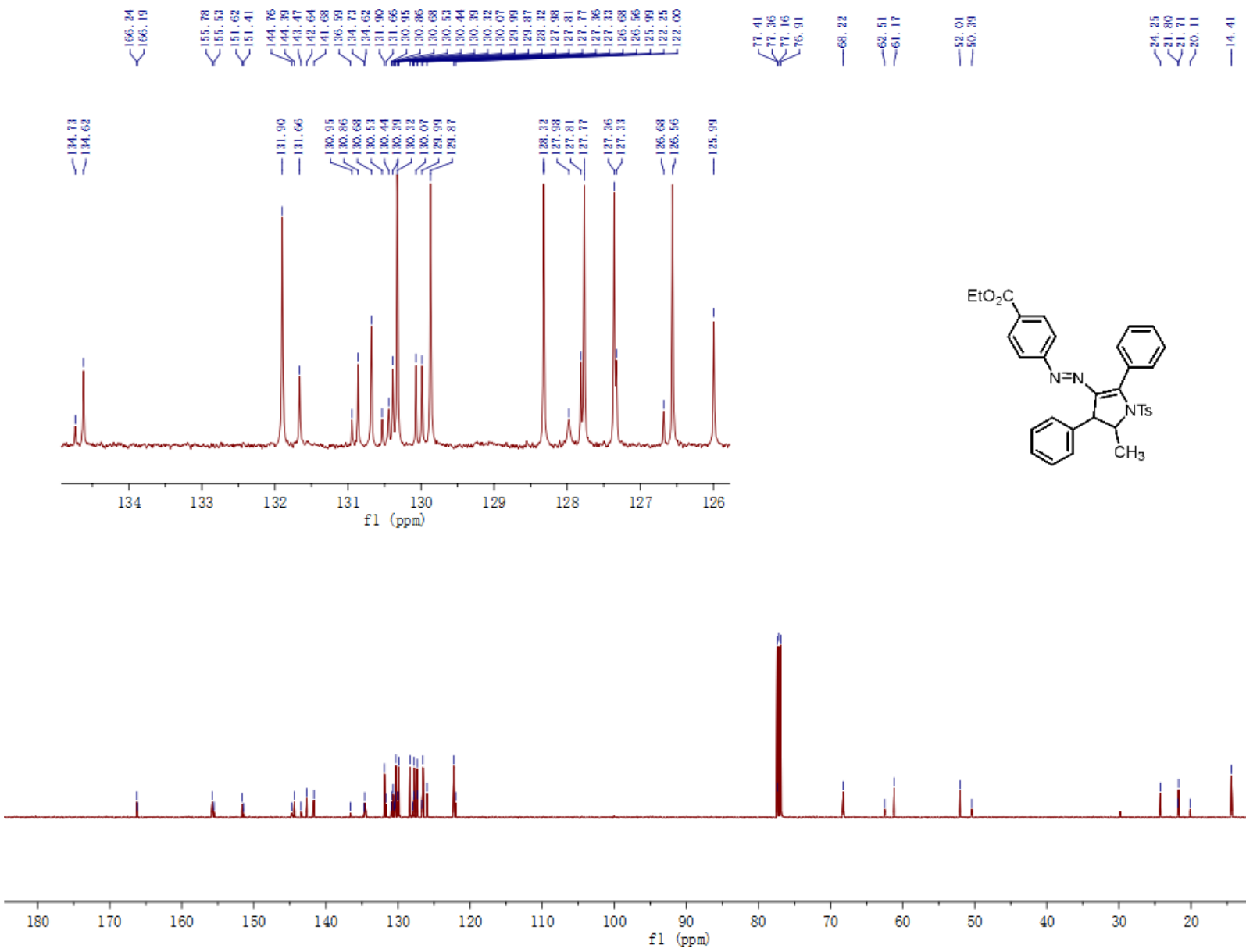
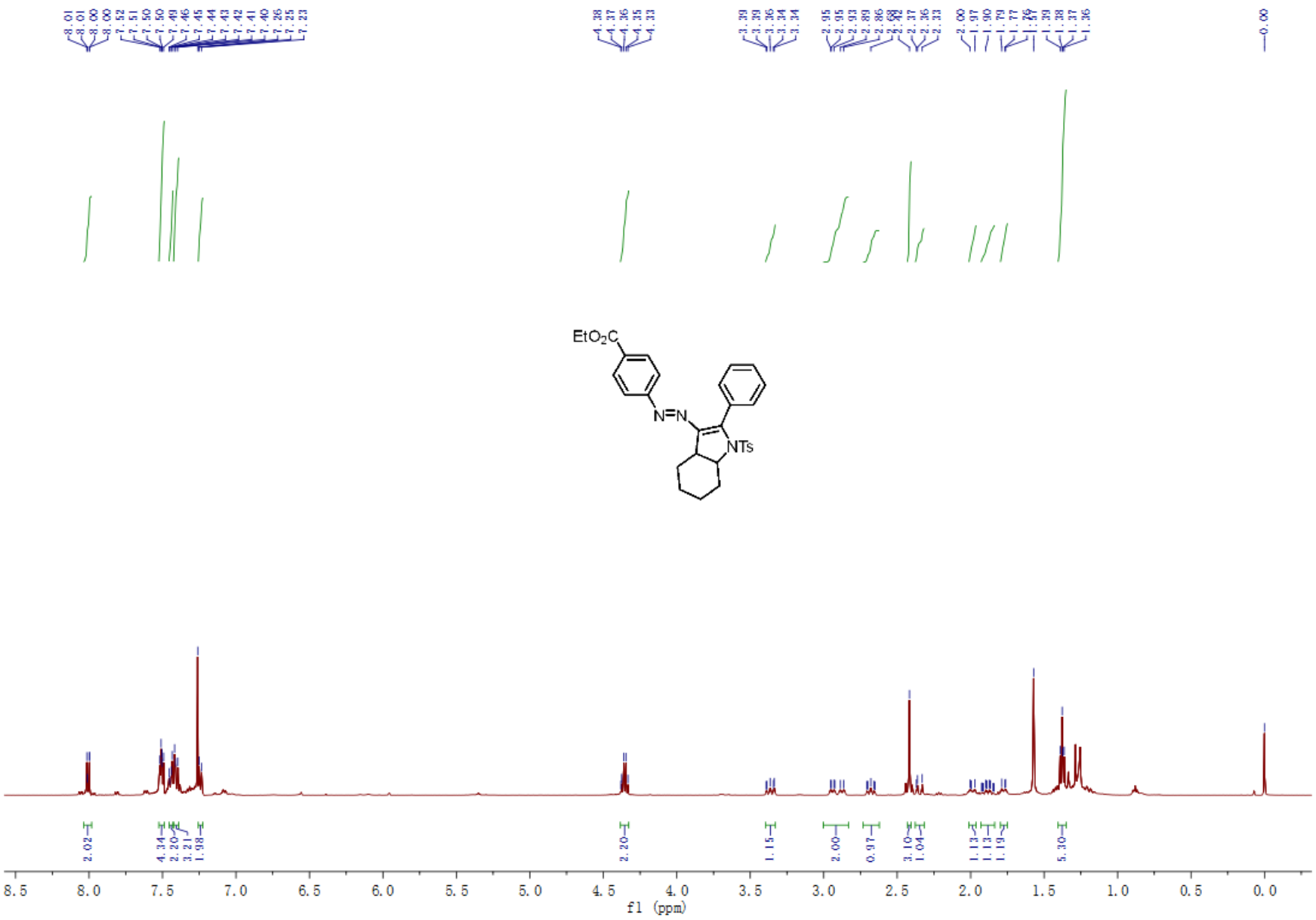
144.96
143.95
140.74
131.88
130.80
130.41
129.88
128.51
128.34
127.86
77.41
77.16
76.91
61.21
59.09
43.34
21.74
14.42
134.23
132.08
131.88
131.09
130.80
130.41
129.88
129.53
128.51
128.34
127.86
127.43
EtO2C
N=N
NTs
Cl
135
134
133
132
131
130
129
128
127
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

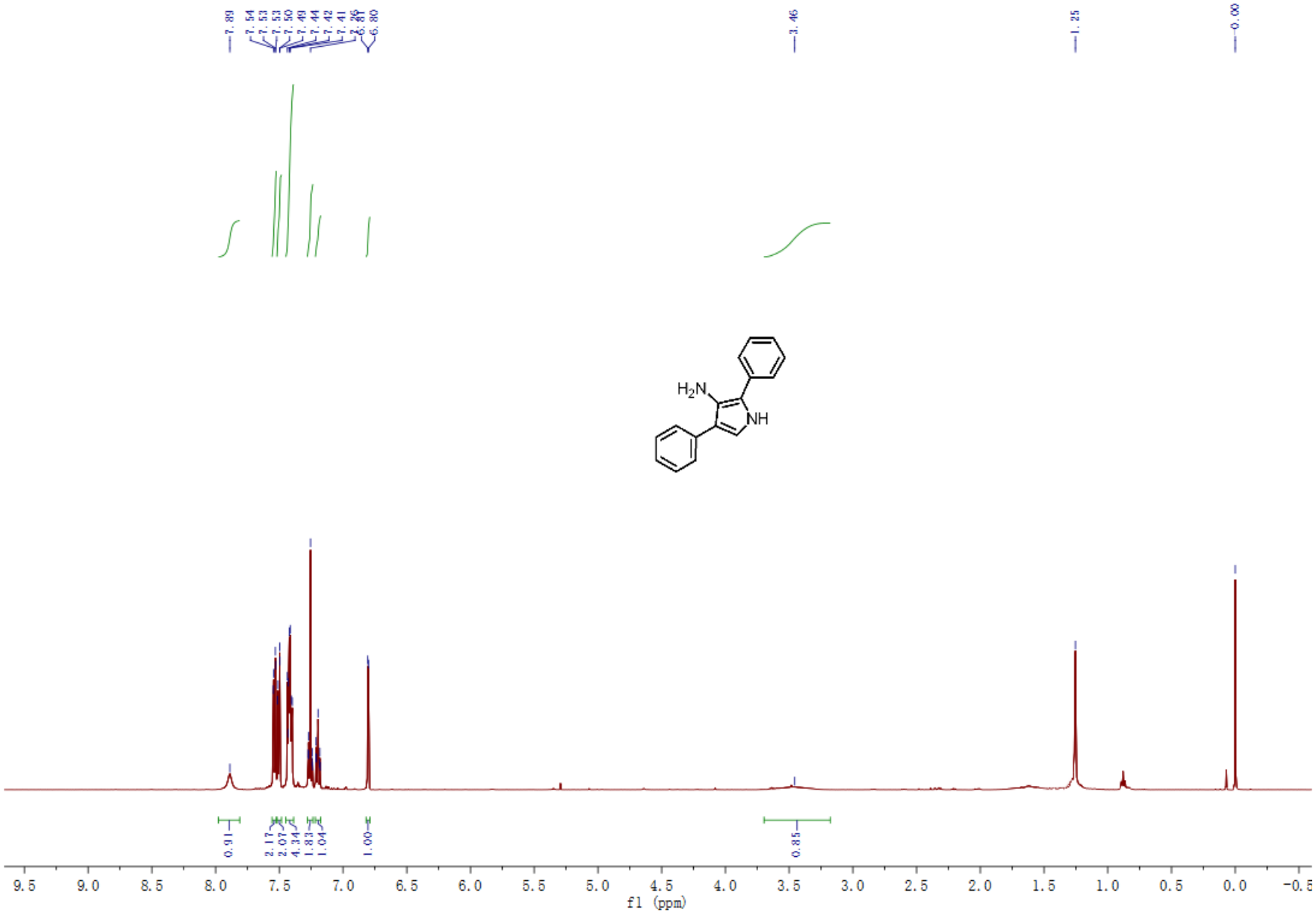

7.95 7.93 7.81 7.79 7.59 7.57 7.56 7.54 7.52 7.51 7.42 7.40 7.33 7.32 7.31 7.31 7.26 7.05 7.03 7.02 7.02 6.72 6.70 6.69 6.23 6.22
4.77 4.76 4.75 4.74 4.66 4.63 4.61 4.35 4.34 4.32 4.31 4.11 4.10 4.08 4.08
2.36
1.55 1.37 1.36 1.34
-0.00
EtO2C
N=N
NTs
Cl
2.04 2.02 3.31 2.03 3.03 3.07 1.00 0.99
f1 (ppm)
1.00 1.02 2.04 1.01
f1 (ppm)
2.04 2.02 3.31 2.03 3.03 3.07 1.00 0.99 1.00 1.02 2.04 1.01 3.02 3.01
f1 (ppm)

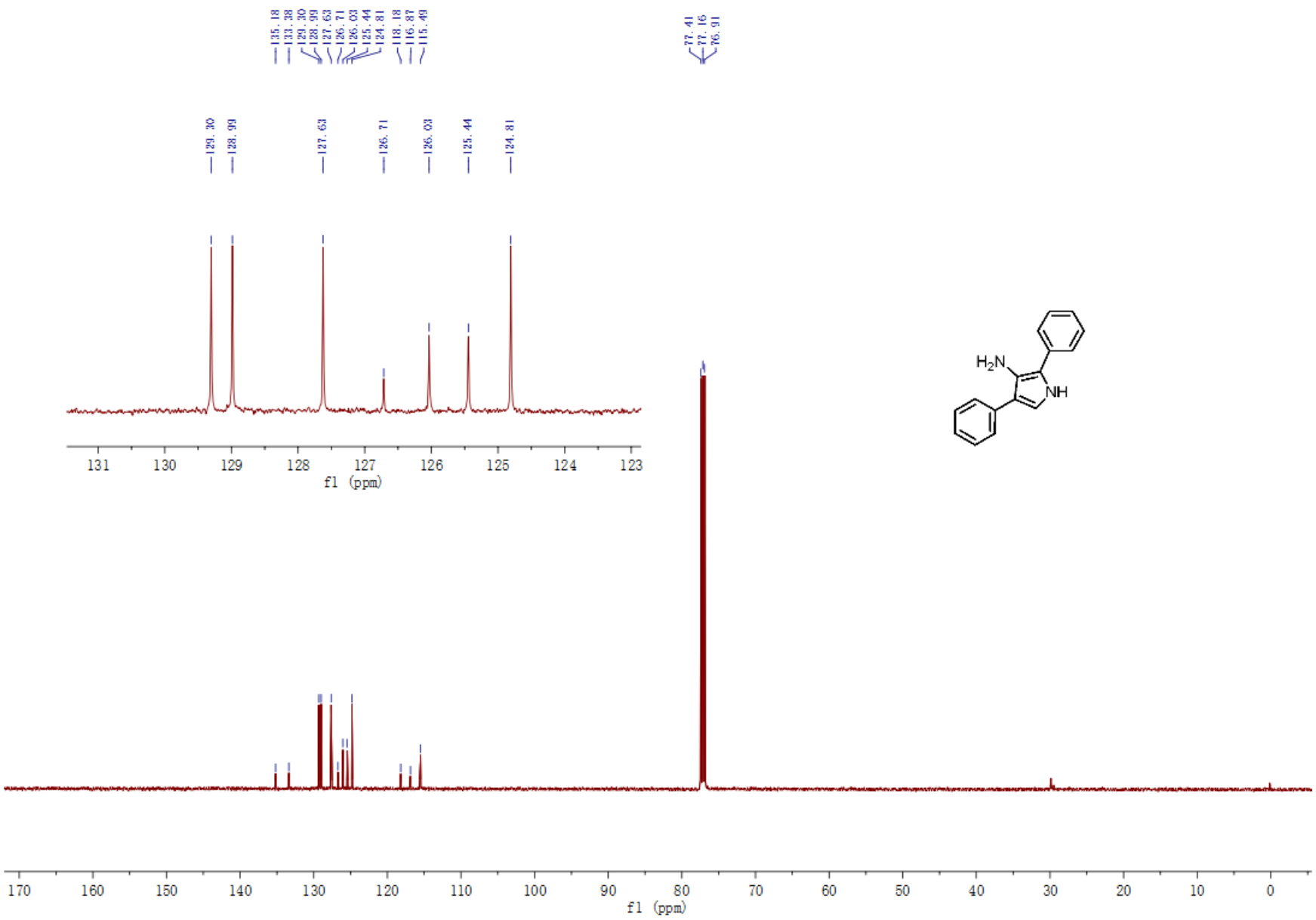

166.23
155.71
153.51
144.61
143.45
139.14
131.88
130.82
130.37
129.86
127.76
127.45
126.62
77.41
77.16
76.91
61.20
58.14
41.38
21.72
14.42
131.88
131.05
130.82
130.37
129.86
129.79
129.44
127.76
127.45
127.41
126.62
EtO2C
N=N
NTs
Cl
132.0 131.5 131.0 130.5 130.0 129.5 129.0 128.5 128.0 127.5 127.0 126.5
f1 (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

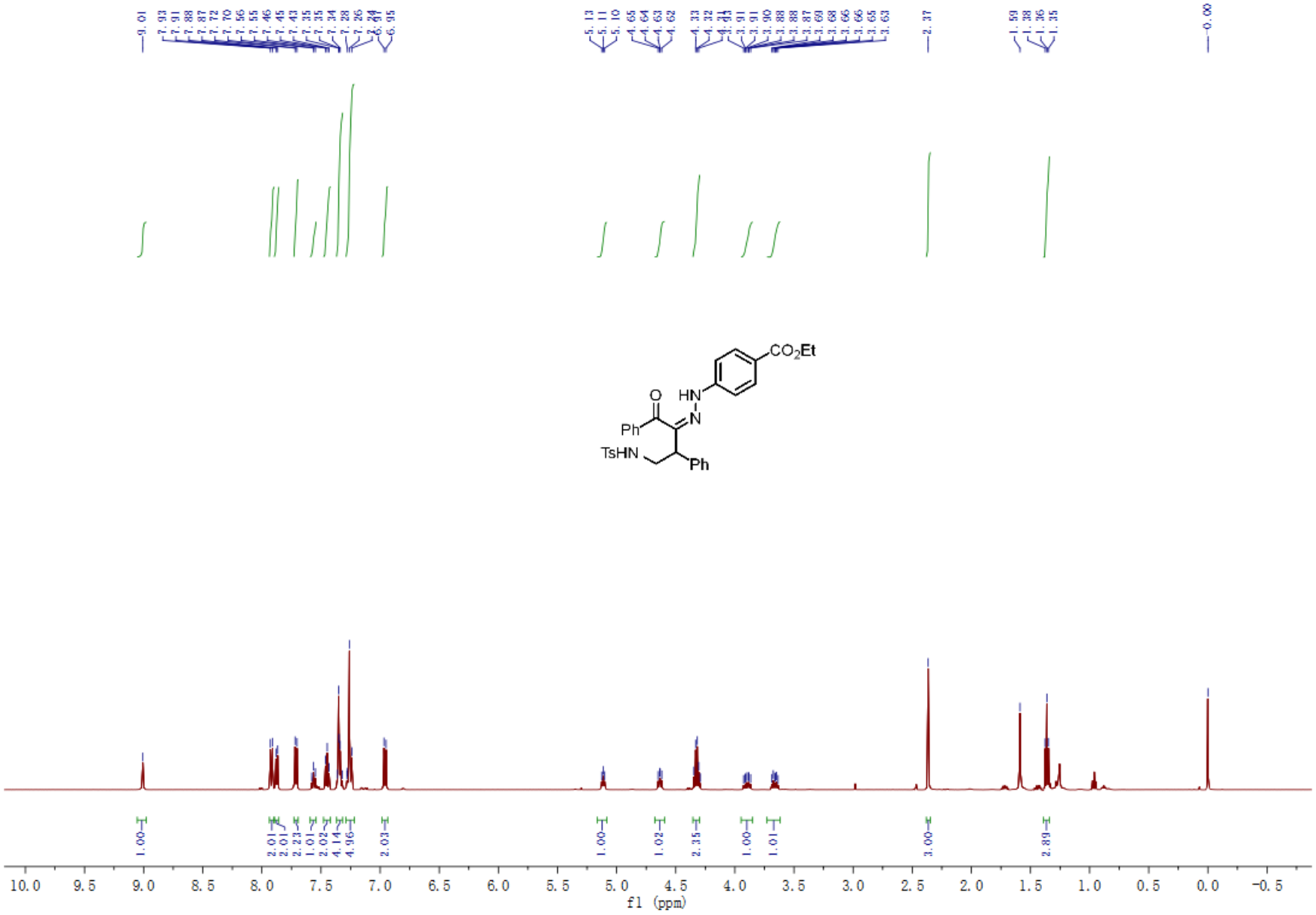

EtO2C
N=N
NTs
CH3
f1 (ppm)

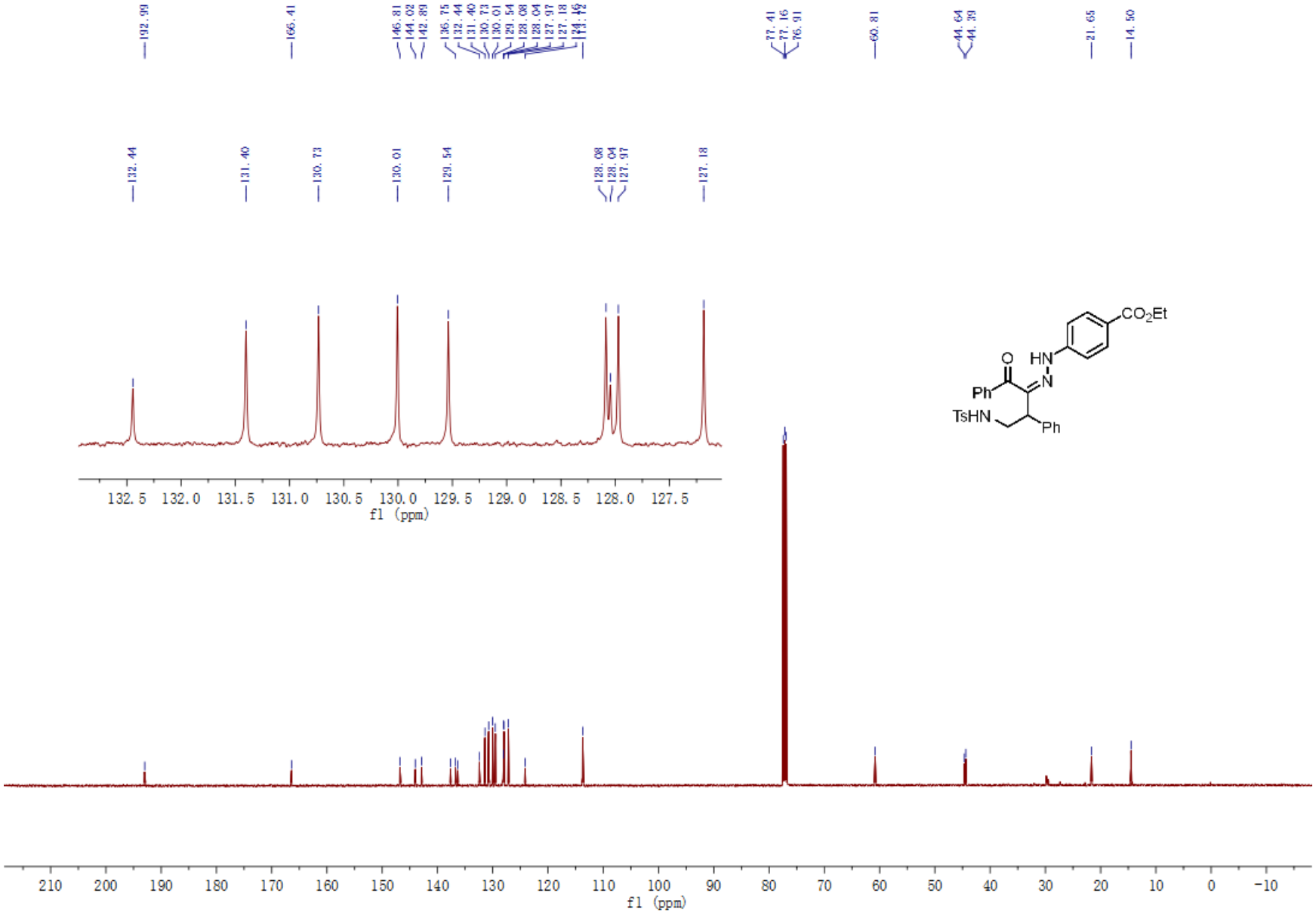

166.24
166.19
155.78
155.53
151.62
151.41
144.76
144.39
143.47
142.64
141.68
136.59
134.73
134.62
131.90
131.66
130.95
130.86
130.68
130.53
130.44
130.39
130.32
130.07
129.99
129.87
128.32
127.98
127.81
127.77
127.36
127.33
126.68
126.56
125.99
122.25
122.09
77.41
77.36
77.16
76.91
68.22
62.51
61.17
52.01
50.39
24.25
21.80
21.71
20.11
14.41
EtO2C
N=N
NTs
CH3
f1 (ppm)
f1 (ppm)

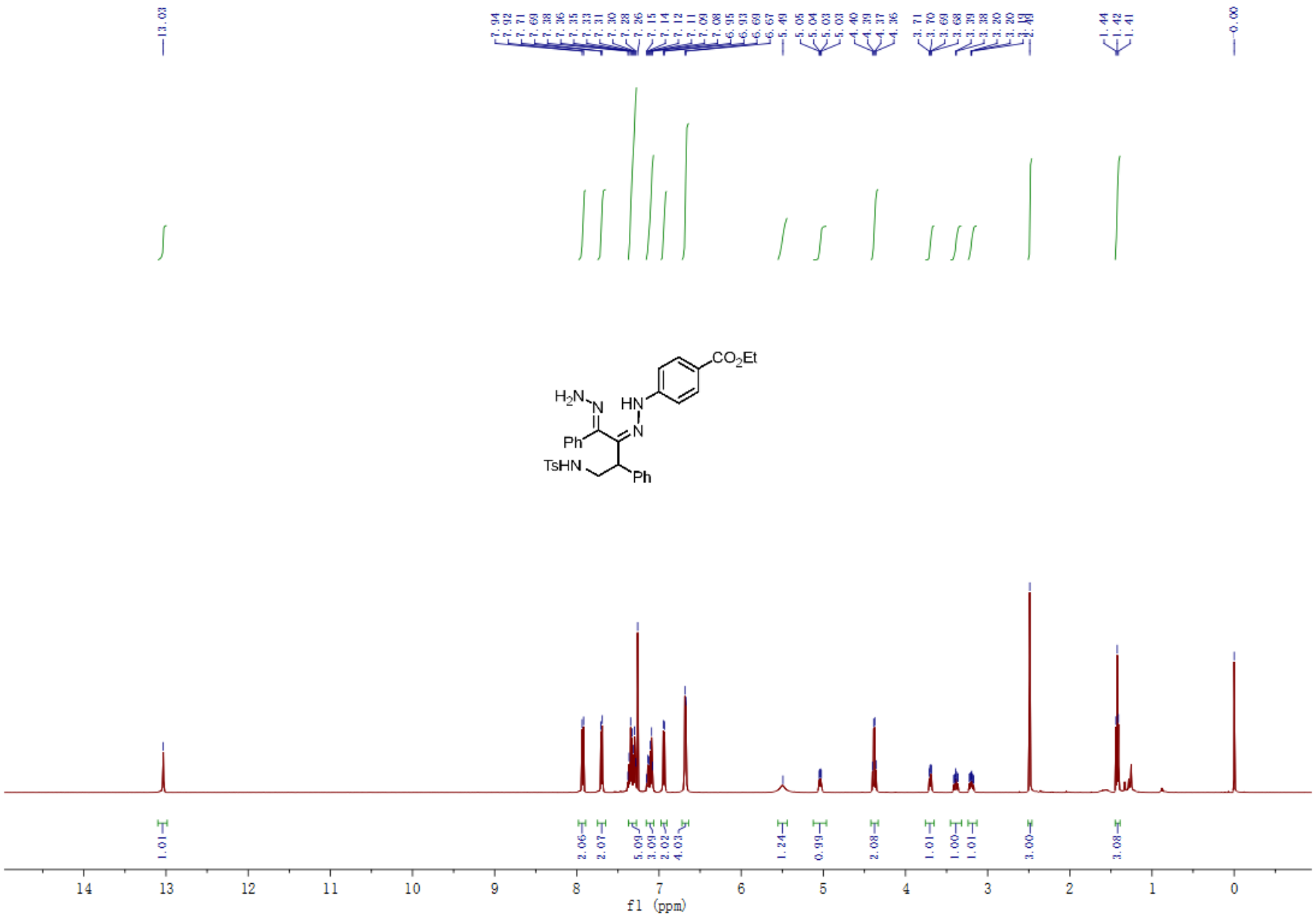

8.01
8.01
8.00
8.00
7.52
7.51
7.50
7.50
7.49
7.46
7.45
7.44
7.43
7.42
7.41
7.40
7.26
7.25
7.23
4.38
4.37
4.36
4.35
4.33
3.39
3.39
3.36
3.34
3.34
2.95
2.95
2.93
2.89
2.86
2.37
2.36
2.33
2.00
1.97
1.90
1.79
1.77
1.39
1.38
1.37
1.36
0.00
EtO2C
N=N
NTs
2.02
4.34
2.20
3.21
1.98
2.20
1.15
2.00
0.97
3.10
1.04
1.13
1.13
1.19
5.30
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

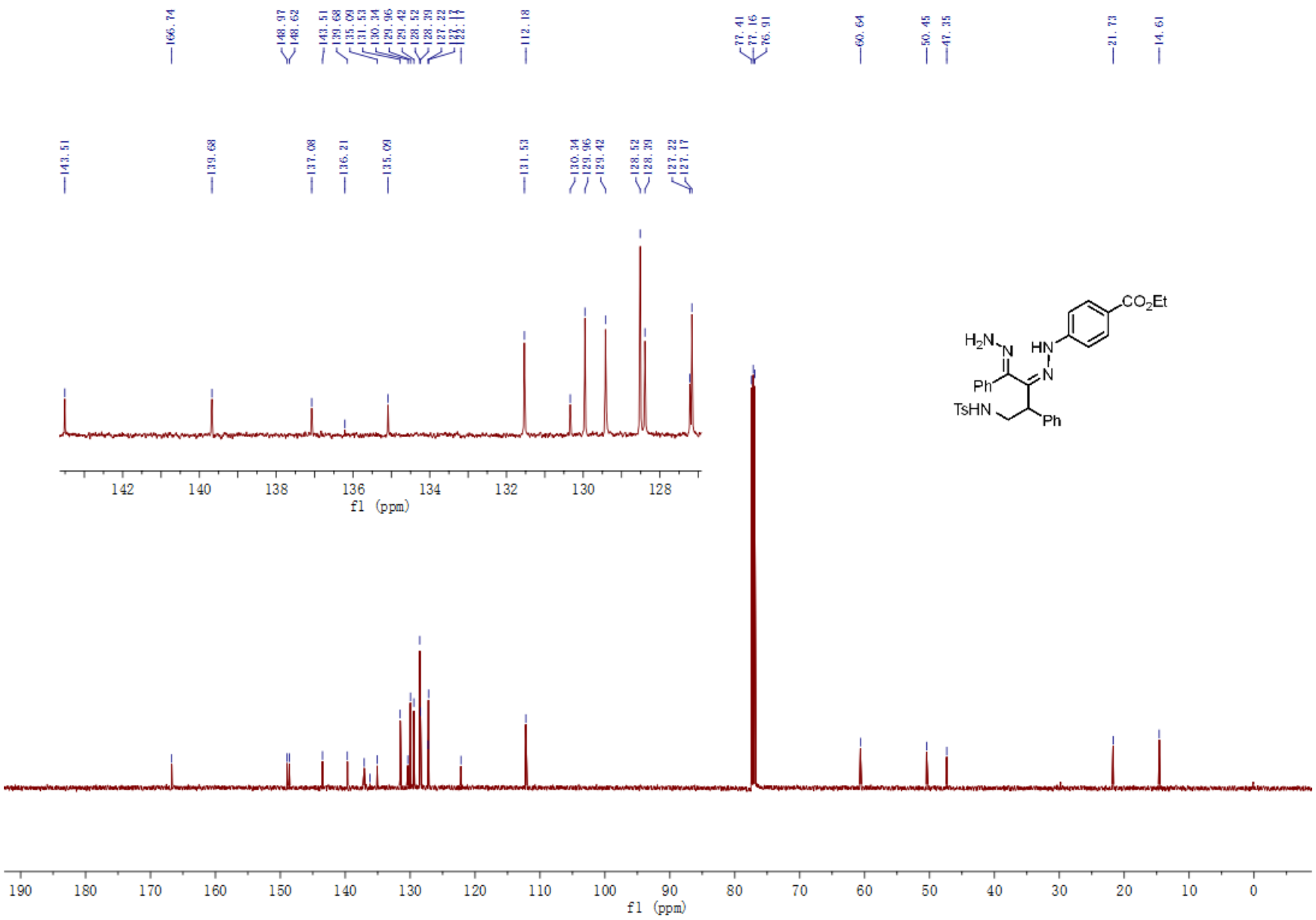

7.89
7.54
7.53
7.53
7.50
7.49
7.44
7.42
7.41
6.81
6.80
3.46
1.25
0.00
H2N
NH
0.91
2.17
2.07
4.34
1.83
1.04
1.00
0.85
f1 (ppm)

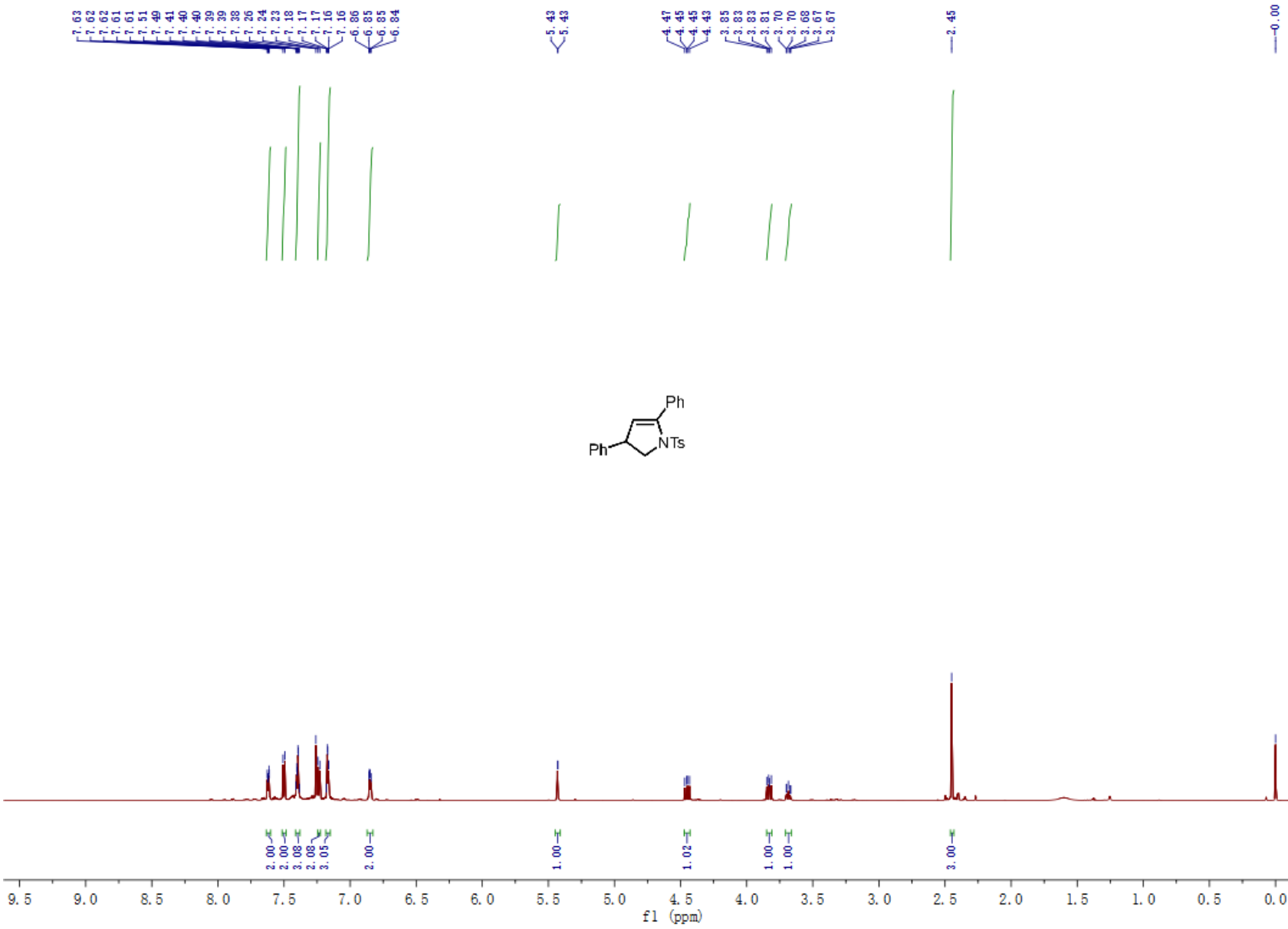

135.18
133.38
129.30
128.99
127.63
126.71
126.03
125.44
124.81
118.18
116.87
115.49
77.41
77.16
76.91
129.30
128.99
127.63
126.71
126.03
125.44
124.81
131
130
129
128
127
126
125
124
123
f1 (ppm)
H2N
NH
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


9.01
7.93
7.91
7.88
7.87
7.72
7.70
7.56
7.55
7.46
7.45
7.43
7.35
7.35
7.34
7.28
7.26
6.95
5.13
5.11
5.10
4.65
4.64
4.63
4.62
4.33
4.32
3.91
3.91
3.90
3.88
3.88
3.87
3.68
3.66
3.66
3.65
3.63
2.37
1.59
1.38
1.36
1.35
0.00
CO2Et
HN
O
N
Ph
TsHN
Ph
1.00
2.01
2.01
2.23
1.01
2.02
4.14
4.96
2.03
1.00
1.02
2.35
1.00
1.01
3.00
2.89
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
-0.5
f1 (ppm)


192.99
166.41
146.81
144.02
142.89
136.75
132.44
131.40
130.73
130.01
129.54
128.08
128.04
127.97
127.18
77.41
77.16
76.91
60.81
44.64
44.39
21.65
14.50
132.44
131.40
130.73
130.01
129.54
128.08
128.04
127.97
127.18
132.5 132.0 131.5 131.0 130.5 130.0 129.5 129.0 128.5 128.0 127.5
f1 (ppm)
CO2Et
HN
N
O
Ph
TsHN
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


13.03
7.94
7.92
7.71
7.69
7.38
7.36
7.35
7.33
7.31
7.30
7.28
7.26
7.15
7.14
7.12
7.11
7.09
7.08
6.95
6.93
6.69
6.67
5.49
5.05
5.04
5.03
4.40
4.39
4.37
4.36
3.71
3.70
3.69
3.68
3.39
3.38
3.20
3.19
2.48
1.44
1.42
1.41
0.00
CO2Et
H2N
N
HN
N
Ph
TsHN
Ph
1.01
2.06
2.07
5.09
3.09
2.02
4.03
1.24
0.99
2.08
1.01
1.00
1.01
3.00
3.08
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


166.74
148.97
148.62
143.51
139.68
135.09
131.53
130.34
129.96
129.42
128.52
128.39
127.22
127.17
112.18
77.41
77.16
76.91
60.64
50.45
47.35
21.73
14.61
143.51
139.68
137.08
136.21
135.09
131.53
130.34
129.96
129.42
128.52
128.39
127.22
127.17
142
140
138
136
134
132
130
128
f1 (ppm)
CO2Et
H2N
N
HN
N
Ph
TsHN
Ph
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.63
7.62
7.62
7.61
7.61
7.51
7.49
7.41
7.40
7.40
7.39
7.39
7.38
7.26
7.24
7.23
7.18
7.17
7.17
7.16
7.16
6.86
6.85
6.85
6.84
5.43
5.43
4.47
4.45
4.45
4.43
3.85
3.83
3.83
3.81
3.70
3.70
3.68
3.67
3.67
2.45
0.00
Ph
NTs
Ph
2.00
2.00
3.08
2.08
3.05
2.00
1.00
1.02
1.00
1.00
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)