Supporting Information for

Rhodium-catalyzed cycloadditions between 3-diazoindolin-2-imines and 1,3-dienes

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Content

General Information

^{1}H NMR spectra were obtained on 400, 500 or 600 MHz in $CDCl_3$. The chemical shifts were quoted in parts per million (ppm) referenced to 0.0 ppm for tetramethylsilane as an internal standard. ^{13}C NMR spectra were recorded on 100, 125 or 150 MHz in $CDCl_3$. The chemical shifts were reported in ppm referenced to the internal solvent signals (77.0 ppm for $CDCl_3$). The following abbreviations were used to describe peak patterns where appropriate: b = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants J were reported in hertz unit (Hz). Infrared spectra were obtained on an FTIR spectrometer. High-resolution mass spectra (HRMS) data were obtained by using EI ionization on time-of-flight (TOF) mass spectrometer. Melting points were measured with SGW X-4 micro melting point apparatus. Flash column chromatography was performed employing 300-400 mesh silica gel. Thin layer chromatography (TLC) was performed on silica gel HSGF254.

CH_2Cl_2, MeCN and 1, 2-dichloroethane (DCE) were dried by distillation over CaH_2. THF, toluene and chlorobenzene was distilled from Na. $Rh_2(Oct)_4$, $Rh_2(OAc)_4$, Rh_2(s-ptad)$_4$, Rh_2(s-dosp)$_4$, AgoTf, isoprene, (*E*)-penta-1, 3-diene, myrcene, TMSO-diene, cyclo- hexa-1, 3-diene and cyclopentadiene were used as received from the commercial sources. The (*E*)-1-phenyl-buta-1, 3-diene, 3-diazoindolin-2-imines and its analogues were prepared according to the published methods.[1-3]

General Procedure for the Synthesis of 3

To an over-dried sealed tube with a magnetic stirring bar were added sequentially **1a** (0.2 mmol), **2** (1 mmol), Rh_2 (oct)$_4$ (1.56 mg, 0.002 mmol), AgOTf (2.57 mg, 0.01 mmol) and dry toluene (2 mL) in N_2 atmosphere. The reaction vessel was heated to 110 ^{o}C in oil bath for 12 hours. Upon completion, the reaction mixture was cooled to room temperature and then the solvent was evaporated in vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/ ethyl acetate = 6:1, v/v) to give pure product **3**.

General Procedure for the Synthesis of 5

To an over-dried sealed tube with a magnetic stirring bar were added sequentially **1a** (0.2 mmol), **4** (1 mmol), Rh_2(oct)$_4$ (1.56 mg, 0.002 mmol), AgOTf (2.57 mg, 0.01 mmol) and dry toluene (2 mL) in N_2 atmosphere. The reaction vessel was heated to 110 ^{o}C in oil bath for 12 hours. Upon completion, the reaction mixture was cooled to room temperature and then the solvent was evaporated in vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/ ethyl acetate = 6:1) to give pure product **5**.

General Procedure for the Synthesis of 7

To an over-dried sealed tube with a magnetic stirring bar were added sequentially **1a** (0.2 mmol), **6** (1 mmol), $Rh_2(oct)_4$ (1.56 mg, 0.002 mmol), AgOTf (2.57 mg, 0.01 mmol) and dry toluene (2 mL) in N_2 atmosphere. The reaction vessel was heated to 110 ^oC in oil bath for 12 hours. Upon completion, the reaction mixture was cooled to room temperature and then the solvent was evaporated in vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/ ethyl acetate = 6:1) to give pure product **7**.

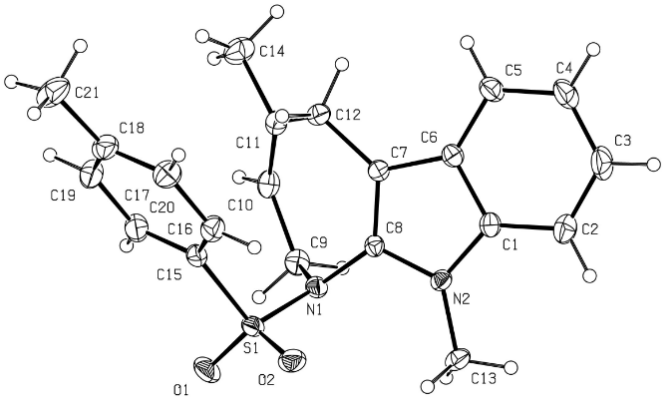
General Procedure for the Synthesis of 9

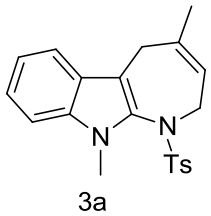
To an over-dried sealed tube with a magnetic stirring bar were added sequentially **1a** (0.2 mmol), **8** (1 mmol), $Rh_2(oct)_4$ (1.56 mg, 0.002 mmol), AgOTf (2.57 mg, 0.01 mmol) and dry toluene (2 mL) in N_2 atmosphere. The reaction vessel was heated to 110 ^oC in oil bath for 12-24 hours. Upon completion, the reaction mixture was cooled to room temperature and then the solvent was evaporated in vacuum. The residue was purified by column chromatography on silica gel (petroleum ether/ ethyl acetate = 5:1) to give pure product **9**.

Reference

1. Lishchynskyi, A.; Muñiz, K. *Chem. Eur. J.* **2012**, *18*, 2212.
2. Xing, Y. P.; Sheng, G. R.; Wang, J.; Lu, P.; Wang, Y. G. *Org. Lett.* **2014**, *16*, 1244.
3. Sheng, G. R.; Huang, K.; Chi, Z. H.; Ding, H. L.; Xing, Y. P.; Lu, P.; Wang, Y. G. *Org. Lett.* **2014**, *16*, 5096.

The ORTEP and Crystal Parameters of 3a





3a

Bond precision: C-C = 0.0029 A Wavelength=0.71073

Cell: a=14.3337(6) b=6.8499(4) c=18.7839(8)
alpha=90 beta=95.751(4) gamma=90
Temperature: 175 K

	Calculated	Reported
Volume	1835.00(15)	1835.02(14)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C21 H22 N2 O2 S	C21 H22 N2 O2 S
Sum formula	C21 H22 N2 O2 S	C21 H22 N2 O2 S
Mr	366.47	366.47
Dx,g cm-3	1.327	1.326
Z	4	4
Mu (mm-1)	0.194	0.194
F000	776.0	776.0
F000'	776.79	
h,k,lmax	17,8,22	17,8,22
Nref	3360	3351
Tmin,Tmax	0.943,0.956	0.966,1.000
Tmin'	0.943	

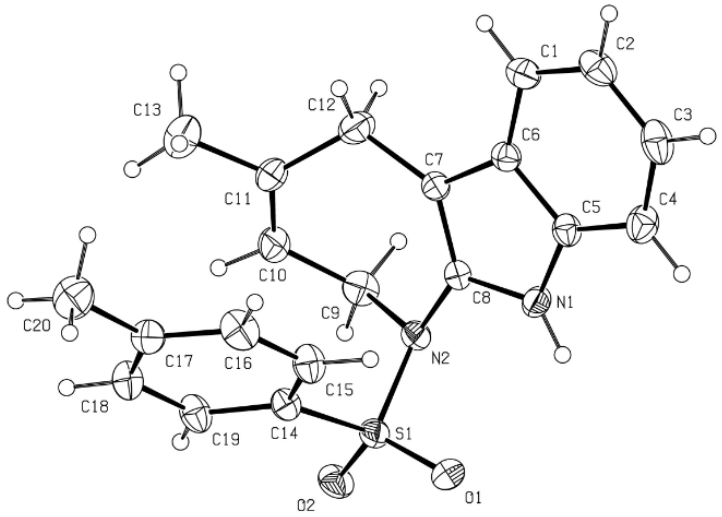
Correction method= MULTI-SCAN

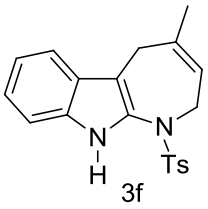
Data completeness= 0.997 Theta(max)= 25.350

R(reflections)= 0.0388(2761) wR2(reflections)= 0.1036(3351)

S = 1.048 Npar= 238

The ORTEP and Crystal Parameters of 3f





3f

Bond precision: C-C = 0.0043 A Wavelength=0.71073

Cell: a=10.9681(6) b=12.3253(5) c=12.9362(5)
alpha=90 beta=90 gamma=90
Temperature: 293 K

	Calculated	Reported
Volume	1748.78(14)	1748.78(14)
Space group	P 21 21 21	P 21 21 21
Hall group	P 2ac 2ab	P 2ac 2ab
Moiety formula	C20 H20 N2 O2 S	C20 H20 N2 O2 S
Sum formula	C20 H20 N2 O2 S	C20 H20 N2 O2 S
Mr	352.44	352.44
Dx,g cm-3	1.339	1.339
Z	4	4
Mu (mm-1)	0.201	0.201
F000	744.0	744.0
F000'	744.78	
h,k,lmax	13,14,15	13,14,15
Nref	3196[1836]	1836
Tmin,Tmax	0.908,0.919	0.898,1.000
Tmin'	0.908	

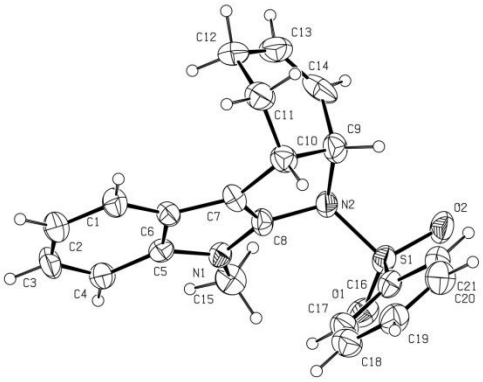
Correction method= # Reported T Limits: Tmin=0.898 Tmax=1.000
AbsCorr = MULTI-SCAN

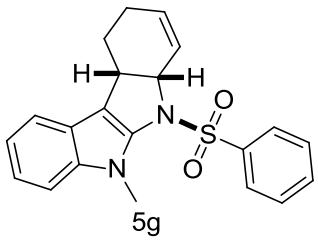
Data completeness= 1.00/0.57 Theta(max)= 25.340

R(reflections)= 0.0306(1642) wR2(reflections)= 0.0743(1836)

S = 1.056 Npar= 228

The ORTEP and Crystal Parameters of 5g





Bond precision: C-C = 0.0124 A Wavelength=0.71073

Cell: a=13.1455(12) b=7.7198(11) c=21.857(2)
alpha=90 beta=126.178(6) gamma=90
Temperature: 293 K

	Calculated	Reported
Volume	1790.4(4)	1790.4(3)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc
Moiety formula	C21 H20 N2 O2 S	C21 H20 N2 O2 S
Sum formula	C21 H20 N2 O2 S	C21 H20 N2 O2 S
Mr	364.45	364.45
Dx,g cm-3	1.352	1.352
Z	4	4
Mu (mm-1)	0.199	0.199
F000	768.0	768.0
F000'	768.79	
h,k,lmax	15,9,26	15,9,26
Nref	3282	3219
Tmin,Tmax	0.931,0.944	0.908,1.000
Tmin'	0.923	

Correction method= # Reported T Limits: Tmin=0.908 Tmax=1.000
AbsCorr = MULTI-SCAN

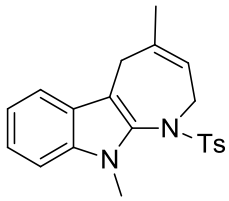
Data completeness= 0.981 Theta(max)= 25.350

R(reflections)= 0.1193(2330) wR2(reflections)= 0.3597(3219)

S = 1.099 Npar= 236

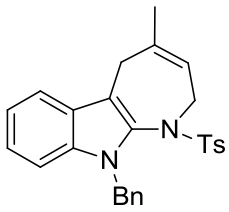
Characterization Data for Products

4, 10-Dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (**3a**)



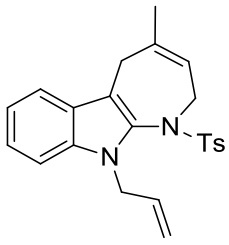
Yellow solid; Yield 40% (29 mg); m.p. 69.5-71.2 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.27 (td, *J* = 6.8 Hz, 1.2 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.12-7.08 (m, 1H), 5.14 (d, *J* = 6.4 Hz, 1H), 4.60 (dd, *J* = 18.0 Hz, 6.4 Hz, 1H), 4.01 (dt, *J* = 18.4 Hz, 2.4 Hz, 1H), 3.81 (s, 3H), 3.12 (d, *J* = 21.2 Hz, 1H), 2.41(s, 3H), 2.25 (d, *J* = 21.2 Hz, 1H), 1.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.5, 136.3, 135.1, 134.9, 133.2, 129.0, 128.7, 125.4, 122.4, 119.1, 118.8,118.1, 109.8, 106.9, 49.1, 29.8, 29.6, 26.3, 21.5; IR(film): 3057, 2968, 2925, 1613, 1471, 1353, 1164, 1090, 748,666 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{22}N_2O_2S$: 366.1402; found: 366.1405.

10-Benzyl-4-methyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3b)



Yellow solid; Yield 63% (56 mg); m.p. 157.8-159.5 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.28 (d, *J* = 8.4 Hz, 1H), 7.25-7.17 (m, 6H), 7.12-7.08 (m, 1H),7.02-7.00 (m, 2H), 5.81 (d, *J* = 16.4 Hz, 1H), 5.37 (d, *J* = 16.4 Hz, 1H), 5.10 (d, *J* = 6.4Hz, 1H), 4.42 (dd, *J* = 18.0 Hz, 5.2 Hz, 1H), 3.65-3.59(m, 1H), 3.09 (d, *J* = 21.2 Hz, 1H), 2.42(s, 3H), 2.16(d, *J* = 20.4 Hz, 1H), 1.41(s, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.6, 138.5, 136.2, 134.9, 134.6, 133.1, 129.0, 128.8, 128.5, 127.0, 126.7, 125.8, 122.7, 119.4, 119.0, 118.3, 110.6, 108.4, 48.9, 46.5, 29.6, 26.4, 21.6; IR(film): 3060, 3031, 2970, 2912, 1597, 1463, 1353, 1163, 1089, 909, 761, 740 cm^{-1}; HRMS (EI) calcd for $C_{27}H_{26}N_2O_2S$: 442.1715; found: 442.1714.

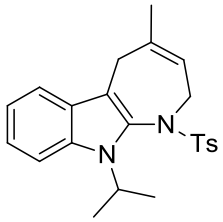
10-Allyl-4-methyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3c)



Yellow oil; Yield 49% (38 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.26-7.22(m, 1H), 7.17(d, *J* = 8.4 Hz, 2H), 7.12-7.08 (m, 1H), 6.01-5.94 (m, 1H), 5.18-5.12 (m, 2H), 5.09 (q, *J* = 1.2 Hz, 1H), 4.92 (dd, *J* = 17.2 Hz, 1.6 Hz, 1H),

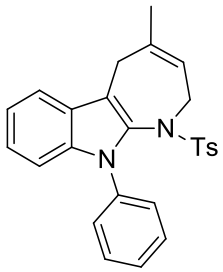
4.95-4.80 (m, 1H), 4.59 (dd, *J* = 18.0 Hz, 6.0 Hz, 1H), 3.98 (d, *J* = 18.0 Hz, 1H), 3.08 (d, *J* = 16.8 Hz, 1H), 2.42 (s, 3H), 2.14 (d, *J* = 21.0 Hz, 1H), 1.42 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$) δ 143.6, 136.2, 134.8, 134.7, 134.5, 132.8, 129.0, 128.8, 125.7, 122.5, 119.3, 118.9, 118.2, 116.1, 110.6, 107.9, 49.1, 45.4, 29.6, 26.4, 21.6; IR(film): 3057, 2971, 1914, 1596, 1465, 1352, 1168, 1093, 1012, 909, 745, 665 cm^{-1}; HRMS(EI) calcd for $C_{23}H_{24}N_2O_2S$: 392.1558; found: 392.1552.

10-Isopropyl-4-methyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3d)



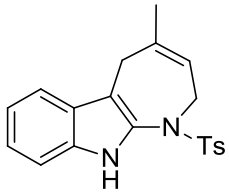
White solid; Yield 51% (40 mg); m.p. 168.2-169.4 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 8.4 Hz, 1H), 7.56 (d, *J* = 8.0 Hz, 2H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.26-7.16 (m, 3H), 7.10-7.06 (m, 1H), 5.18-5.09 (m, 2H), 4.65 (dd, *J* = 20.4 Hz, 4.8 Hz, 1H), 3.97 (d, *J* = 18.8 Hz, 1H), 3.04 (d, *J* = 20.8 Hz, 1H), 2.42 (s, 3H), 2.18 (d, *J* = 20.0 Hz,1H), 1.82 (d, *J* = 7.2 Hz, 3H), 1.50 (d, *J* =7.2 Hz, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.5, 136.3, 135.0, 132.7, 132.5, 128.97, 128.87, 126.2, 121.8, 118.8, 118.7, 118.4, 112.3, 107.2, 49.4, 46.3, 29.4, 26.4, 21.6, 21.5, 21.1; IR (film): 2971, 2929, 1596, 1457, 1349, 1164, 1091, 908, 740, 664 cm^{-1}; HRMS (EI) calcd for $C_{23}H_{26}N_2O_2S$:394.1715; found: 394.1713.

4-Methyl-10-phenyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3e)



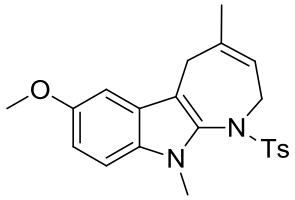
White solid; Yield 56% (48 mg); m.p. 168.2-169.6 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52 (t, *J* = 7.6 Hz, 2H), 7.47-7.41 (m, 4H), 7.40-7.35 (m, 2H), 7.28-7.12 (m, 5H), 5.22 (d, *J* = 3.6 Hz, 1H), 4.51 (dd, *J* = 17.6, 5.2 Hz, 1H), 4.12 (d, *J* = 18.0 Hz, 1H), 3.22 (d, *J* = 20.4 Hz, 1H), 2.55 (d, *J* = 20.8 Hz, 1H), 2.40 (s, 3H), 1.52 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.4, 136.9, 136.4, 135.7, 134.2, 132.8, 129.04, 128.97, 128.6, 127.9, 127.3, 125.7, 123.1, 120.0, 119.2, 118.2, 110.8, 109.8, 48.9, 29.5, 26.5, 21.5; IR(film): 3055, 2971, 2910, 2857, 1598, 1500, 1455, 1379, 1358, 1164, 1090, 909, 766, 748, 664 cm^{-1}; HRMS (EI) calcd for $C_{26}H_{24}N_2O_2S$: 428.1558; found: 428.1558.

4-Methyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3f)



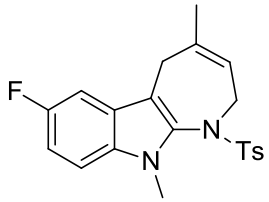
White solid; Yield 57% (40 mg); m.p. 159.0- 160.0 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.63 (s, 1H), 7.42 (d, *J* = 8.0 Hz, 2H), 7.36 (dd, *J* = 12.4, 7.6 Hz, 2H), 7.22 (td, *J* =7.2 Hz, 1.2 Hz, 1H), 7.15-7.09 (m, 3H), 5.29-5.24 (m, 1H), 4.32 (dd, *J* = 6.4 Hz, 0.8 Hz, 2H), 3.15 (s, 2H), 2.37 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 139.6, 135.6, 132.7, 132.3, 129.2, 127.8, 127.0, 122.3, 119.4, 119.1, 117.7, 110.8, 102.8, 47.9, 29.4, 25.6, 21.5; IR(film): 3390, 3060, 2971, 2917, 1597, 1459, 1355, 1161, 1088, 743, 663 cm^{-1}; HRMS (EI) calcd for $C_{20}H_{20}N_2O_2S$: 352.1245; found: 352.1249.

7-Methoxy-4, 10-dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3g)



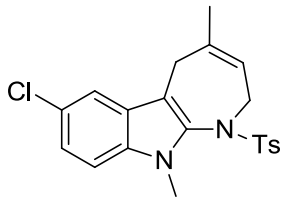
White solid; Yield 53% (42 mg); m.p. 156.0-157.0 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53 (d, *J* = 8.4 Hz, 2H), 7.22 (d, *J* = 8.8 Hz, 1H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.93 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 6.81 (d, *J* = 2.4 Hz, 1H), 5.15 (d, *J* = 6.0 Hz, 1H), 4.59 (dd, *J* = 18.0 Hz, 6.4 Hz, 1H), 4.00 (d, *J* = 18.0 Hz, 1H), 3.84 (s, 3H), 3.77 (s, 3H), 3.07 (d, *J* = 20.8 Hz, 1H), 2.42 (s, 3H), 2.23 (d, *J* = 20.8 Hz, 1H), 1.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.9, 143.5, 136.3, 135.0, 133.5, 130.4, 129.0, 128.7, 125.6, 118.8, 112.6, 110.7, 106.5, 100.1, 55.9, 49.1, 29.9, 29.7, 26.3, 21.5; IR(film): 2928, 2855, 1594, 1488, 1353, 1163, 1090, 1034, 910, 770, 748, 668 cm^{-1}; HRMS (EI) calcd for $C_{22}H_{24}N_2O_3S$: 396.1508; found: 396.1512.

7-Fluoro-4, 10-dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3h)



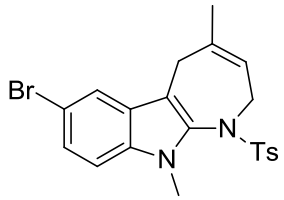
White solid; Yield 72% (55 mg); m.p. 87.8-89.1 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ7.52 (d, *J* = 8.4 Hz, 2H), 7.25-7.22 (m, 1H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.02 (d, *J* = 9.2 Hz, 2H), 5.15 (d, *J* = 6.4 Hz, 1H), 4.60 (dd, *J* =18.0 Hz, 6.4 Hz, 1H), 4.01(dt, *J* = 18.0 Hz, 2.4 Hz, 1H), 3.79(s, 3H), 3.02(d, *J* = 20.8 Hz, 1H), 2.42 (s, 3H), 2.20 (d, *J* = 21.2 Hz, 1H), 1.40 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ157.4 (d, *J* = 233.5 Hz), 143.7, 136.1, 134.8, 134.4, 131.7, 129.0, 128.8, 125.5(d, *J* = 9.6 Hz), 118.8, 110.9(d, *J* = 35.2 Hz), 110.6, 107.0 (d, *J* = 4.8 Hz), 103.3 (d, *J* = 23.3 Hz) , 49.0, 29.84, 29.77, 26.3, 21.5; IR(film): 2974, 2926, 2856, 1594, 1488, 1355, 1164, 1090, 911, 797, 696, 668 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{21}FN_2O_2S$: 384.1308; found: 384.1303.

7-Chloro-4, 10-dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3i)



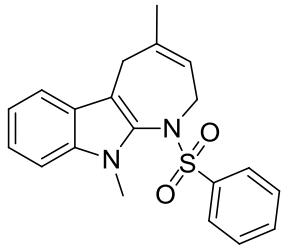
White oil; Yield 60% (48 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.0 Hz, 2H), 7.34 (m, 1H), 7.23-7.21 (m, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 5.15 (d, *J* = 6.4 Hz, 1H), 4.60 (dd, *J* = 17.6 Hz, 6.4 Hz, 1H), 4.02 (d, *J* = 17.6 Hz, 1H), 3.79 (s, 3H), 3.04 (d, *J* = 20.8 Hz, 1H), 2.42 (s, 3H), 2.20 (d, *J* = 20.8 Hz, 1H), 1.40 (s, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ 143.8, 136.1, 134.7, 134.3, 133.5, 129.1, 128.7, 126.4, 125.0, 122.7, 118.8, 117.7, 110.9, 106.7, 49.0, 29.9, 29.7, 26.3, 21.6; IR(film): 2971, 2924, 2852, 1594, 1474, 1352, 1165, 1091, 1034, 909, 764, 666 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{21}ClN_2O_2S$: 400.1012; found: 400.1015.

7-Bromo-4, 10-dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3j)



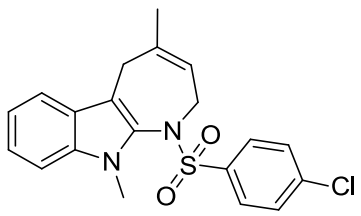
White solid; Yield 74% (66 mg); m.p. 76.2-77.8 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52-7.50 (m, 3H), 7.34 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.20-7.17 (m, 3H), 5.15 (d, *J* = 5.6 Hz, 1H), 4.60 (dd, *J* = 17.6 Hz, 6.4 Hz, 1H), 4.02 (d, *J* = 18.0 Hz, 1H), 3.79 (s, 3H), 3.04 (d, *J* = 20.8 Hz, 1H), 2.43 (s, 3H), 2.19 (d, *J* = 20.8 Hz, 1H),1.40 (s, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ 143.8, 136.1, 134.7, 134.1, 133.8, 129.1, 128.7, 127.0, 125.2, 120.8, 118.8, 112.5, 111.3, 106.7, 49.0, 29.8, 29.7, 26.3, 21.6; IR(film): 2974, 2920, 2855, 1597 ,1471, 1164, 1089, 909, 794, 666 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{21}BrN_2O_2S$: 444.0507; found: 444.0502.

4, 10-Dimethyl-1-(phenylsulfonyl)-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3k)



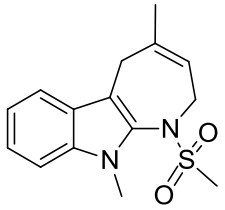
White solid; Yield 67% (47 mg); m.p. 157.0-158. 0 ^{o}C; 1H NMR (400 MHz, $CDCl_3$): δ 7.66 (d, *J* = 7.2 Hz, 2H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.40-7.32 (m, 5H), 7.11 (t, *J* = 6.8 Hz, 1H), 5.12 (d, *J* = 4.0 Hz, 1H), 4.62 (dd, *J* =17.6 Hz, 6.0 Hz, 1H), 4.02 (d, *J* = 18.0 Hz, 1H), 3.82 (s, 3H), 3.11 (d, *J* = 20.8 Hz, 1H), 2.19 (d, *J* = 20.8 Hz,1H), 1.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ139.1, 135.14, 135.09, 133.0, 132.7, 128,7, 128.4, 125.4, 122.4, 119.2, 118.6, 118.2, 109.8, 107.0, 49.1, 29.7, 29.6, 26.4; IR(film): 3057, 2974, 2913, 2852, 1612, 1471, 1446, 1408, 1378, 1167, 1121, 1013, 908, 746, 687 cm^{-1}; HRMS (EI) calcd for $C_{20}H_{20}N_2O_2S$: 352.1245; found: 352.1248.

1-((4-Chlorophenyl) sulfonyl)-4, 10-dimethyl-1, 2, 5, 10- tetrahydroazepino [2,3 -b]indole (3l)



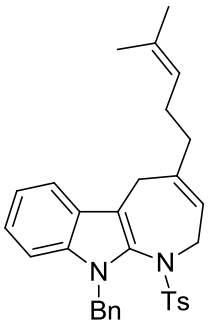
White oil; Yield 66% (51 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 8.4 Hz, 2H), 7.40-7.24 (m, 5H),, 7.14-7.10 (m, 1H), 5.18 (d, *J* = 5.6 Hz, 1H), 4.60 (dd, *J* = 18.0 Hz, 6.4 Hz, 1H), 4.03 (d, *J* = 18.0 Hz, 1H), 3.80 (s, 3H), 3.19 (d, *J* = 21.2 Hz, 1H), 2.29 (d, *J* = 21,2 Hz, 1H), 1.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.4, 137.7, 135.3, 135.1, 132.7, 130.2, 128.6, 125.3, 122.6, 119.3, 118.8, 118.2, 109.8, 106.8, 49.2, 30.0, 29.7, 26.3; IR(film): 3057, 2974, 2914, 2858, 1617, 1585, 1471, 1357, 1167, 1092, 1012, 908, 768, 643 cm^{-1}; HRMS (EI) calcd for $C_{20}H_{19}ClN_2O_2S$: 386.0856; found: 386.0860.

4, 10-Dimethyl-1-(methylsulfonyl)-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3m)



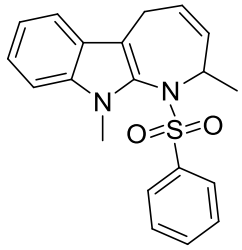
White solid 49% (28 mg); m.p. 176.9-178.0 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.0 Hz, 1H), 7.28 (dd, *J* = 2.0 Hz, 1.2 Hz, 2H), 7.16-7.14 (m, 1H), 5.56 (d, *J* = 5.6 Hz, 1H), 4.47 (dd, *J* = 17.6, 6.4 Hz, 1H), 4.03 (d, *J* =18.0 Hz, 1H), 3.77-3.71 (m, 4H), 3.53 (d, *J* = 21.2 Hz, 1H), 2.98 (s, 3H), 1.92 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ135.3, 135.1, 133.1, 125.5, 122.6, 120.3, 119.4, 118.1, 109.8, 105.8, 48.7, 38.8, 30.8, 29.6, 26.6; IR(film): 3012, 2974, 2926, 2867, 1597, 1470, 1350, 1167, 1090, 989, 909, 791, 671 cm^{-1}; HRMS (EI) calcd for $C_{15}H_{18}N_2O_2S$: 290.1089; found: 290.1093.

10-Benzyl-4-(4-methylpent-3-en-1-yl)-1-tosyl-1, 2, 5, 10-tetrahydroazepino[2,3 -b]indole (3n)



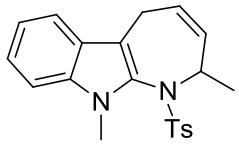
Yellow oil; Yield 35% (36 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.41 (d, *J* = 8.0 Hz, 1H), 7.28 (d, *J* = 8.4 Hz, 1H), 7.25-7.16 (m, 6H), 7.12-7.08 (m, 1H), 7.02 (d, *J* = 6.8 Hz, 2H), 5.81 (d, *J* = 16.4 Hz, 1H), 5.38 (d, *J* = 16.4 Hz, 1H), 5.10 (d, *J* = 5.2 Hz, 1H), 5.02-4.98 (m, 1H), 4.48-4.43 (m, 1H) , 3.62(d, *J* = 18.0 Hz, 1H), 3.12(d, *J* = 20.4 Hz, 1H), 2.40(s, 3H), 2.22(dd, *J* = 20.4 Hz, 2.4 Hz,1H), 1.92-1.78 (m, 2H), 1.71-1.66 (m, 5H), 1.59 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 138.4, 137.7, 136.5, 134.9, 132.8, 131.8, 129.1, 128.6, 128.5, 127.0, 126.7, 125.8, 123.7, 122.7, 119.3, 118.7, 118.3, 110.5, 108.8, 48.6, 46.5, 40.3, 27.9, 26.7, 25.6, 21.5, 17.7; IR (film): 3062, 3036, 2920, 2848, 1593, 1495, 1465, 1346, 1162, 1093, 906, 808, 736, 668 cm^{-1}; HRMS (EI) calcd for $C_{32}H_{34}N_2O_2S$ 510.2341; found:510.2340.

2, 10-Dimethyl-1-(phenylsulfonyl)-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3o)



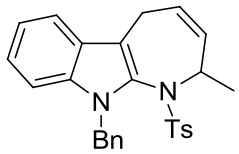
Yellow solid; Yield 71% (50 mg); m.p. 128.9-129.7 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68 (dd, *J* = 8.4 Hz, 1.2 Hz, 2H), 7.58-7.54 (m, 1H), 7.40 (s, 1H), 7.38-7.33 (m, 3H), 7.27 (dt, *J* = 6.8, 1.2 Hz, 1H), 7.13-7.09 (m, 1H), 5.39-5.34 (m, 1H), 5.26-5.21 (m, 1H), 5.03-4.96 (m, 1H), 3.80 (s, 3H), 3.15 (dd, *J* = 20.8, 6.0 Hz, 1H), 2.21-2.13 (m, 1H), 1.14 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.0, 135.4, 132.9, 130.0, 129.3, 128.6, 128.51, 125.47, 125.3, 122.3, 119.1, 118.3, 109.8, 109.4, 54.3, 29.6, 24.1, 21.4; IR(film):3060, 2974, 2926, 1618, 1471,1351, 1168, 1097, 909, 752, 689 cm^{-1}; HRMS (EI) calcd for $C_{20}H_{20}N_2O_2S$ 352.1245; found:352.1242.

2, 10-Dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3p)



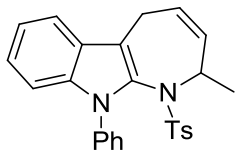
White solid; Yield 50% (37 mg); m.p. 180.1-181.2 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.34 (d, *J* = 8.4 Hz, 1H), 7.28(d, *J* = 7.6Hz, 1H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.11 (t, *J* = 8.0 Hz, 1H), 5.39-5.35 (m, 1H), 5.28-5.23 (m, 1H),4.98 (m, 1H), 3.80 (s, 3H), 3.18 (dd, *J* = 21, 6.0 Hz, 1H), 2.42 (s, 3H), 2.22 (d, *J* = 21 Hz, 1H), 1.14 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 136.2, 135.4, 130.1, 129.4, 129.1, 128.6, 125.5, 125.3, 122.3, 119.1, 118.3, 109.8, 109.4, 54.2, 29.5, 24.2, 21.6, 21.4; IR(film): 3048, 2962, 2924, 2824, 1620, 1435, 1157, 964, 847, 744, 699 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{22}N_2O_2S$ 366.1402; found: 366.1402.

10-Benzyl-2-methyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3q)



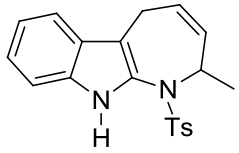
Yellow solid; Yield 65% (57 mg); m.p. 83.5-84.4 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (d, *J* = 8.0 Hz, 2H), 7.42-7.40 (m, 1H), 7.27-7.25 (m, 1H), 7.22-7.20 (m, 2H), 7.18-7.14 (m, 3H), 7.13-7.07 (m, 4H) , 5.74 (d, *J* = 8.8 Hz, 1H), 5.44 (d, *J* = 8.8Hz, 1H), 5.37-5.31 (m, 1H), 5.25-5.20 (m, 1H), 4.90-4.86 (m, 1H), 3.22 (dd, *J* = 21.6, 6.0Hz, 1H), 2.41 (s, 3H), 2.30-2.23 (m, 1H), 0.87 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.8, 137.8, 135.9, 135.3, 129.6, 129.1, 129.2, 128.8, 128.3, 127.0, 126.8, 125.6, 125.2, 122.5, 119.3, 118.3, 111.1, 110.0, 54.3, 46.7, 24.5, 21.6, 21.5; IR(film): 3059, 2975, 2923, 2869, 1597, 1500, 1455, 1166, 1089, 908, 751, 698 cm^{-1}; HRMS (EI) calcd for $C_{27}H_{26}N_2O_2S$ 442.1715; found: 442.1714.

2-Methyl-10-phenyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3r)



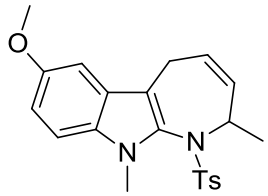
White solid; Yield 52% (45 mg); m.p.222.5-223.3^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.53-7.50 (m, 4H), 7.48-7.45 (m, 4H), 7.21-7.19 (m, 1H), 7.16 (dd, *J* = 11.4, 0.6 Hz, 1H), 7.14 (d, *J* = 6.4 Hz, 2H), 7.07 (d, *J* = 6.4 Hz, 1H), 5.39-5.33 (m, 2H), 4.67-4.65 (m, 1H), 3.36 (dd, *J* = 20.4, 4.8 Hz, 1H), 2.75 (dd, *J* = 21.0, 3 Hz, 3H), 2.40 (s, 3H), 1.18 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (125MHz, $CDCl_3$) δ 143.6, 137.1, 137.0, 136.4, 130.7, 130.1,129.6, 129.1, 128.8, 128.7,128.0, 125.8, 124.7, 122.9, 119.8, 118.2, 111.5, 111.2, 54.1, 24.5, 21.6, 21.4; IR(film): 3060, 2976, 2923, 2869, 1597, 1500, 1456, 1166, 909, 751, 698 cm^{-1}; HRMS (EI) calcd for $C_{26}H_{24}N_2O_2S$ 428.1558; found: 428.1554.

2-Methyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3s)



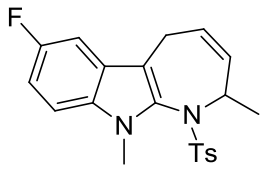
White solid; Yield 46% (32 mg); m.p. 180.7-181.5 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.41(s, 1H), 7.47(d, *J* = 7.8 Hz, 2H), 7.36 (dd, *J* = 9.0, 8.4 Hz, 2H), 7.23 (dd, *J* = 13.8 Hz, 3.6 Hz, 1H), 7.14 (d, *J* = 6.4 Hz, 2H), 7.10 (t, *J* = 7.8 Hz, 1H), 5.48-5.45 (m, 1H), 5.32-5.28 (m, 1H), 4.97-4.94 (m, 1H), 3.32 (dd, *J* = 21.6 Hz, 4.8 Hz, 1H), 2.53-2.49 (m, 1H), 2.38 (s, 3H), 1.25 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.6, 135.7, 134.1, 129.6, 129.2, 128.7, 127.9, 126.6, 125.4, 122.6, 119.4, 118.1, 111.1, 108.3, 53.8, 24.7, 21.59, 21.56; IR(film): 3387, 3062, 3018, 2926, 2855, 1597, 1353, 1168, 1040, 909, 812, 706, 669 cm^{-1}; HRMS (EI) calcd for $C_{20}H_{20}N_2O_2S$352.1245; found: 352.1248.

7-Methoxy-2, 10-dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3t)



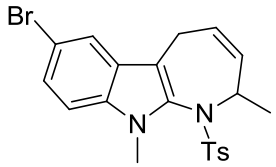
White solid 51% (40 mg); m.p. 157.8-158.3 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.56 (d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 9.0 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 6.93 (dd , *J* = 9.0 Hz, 2.4 Hz, 1H), 6.83 (d, *J* = 8.4 Hz, 1H), 5.38-5.35 (m, 1H), 5.27-5.24 (m, 1H), 4.98-4.97 (m, 1H), 3.84 (s, 3H), 3.76 (s, 3H), 3.11 (dd, *J* = 21.0 Hz, 6.0 Hz, 1H), 2.41 (s, 3H), 2.20 (dd, *J* = 21.0 Hz, 3.0 Hz, 1H), 1.13 (d, *J* = 7.2Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ153.8, 143.6, 136.2, 130.7, 130.4, 129.4, 129.1, 128.6, 125.5, 125.4, 112.5, 110.6, 109.0, 100.2,55.9, 54.2, 29.6, 24.3 21.6, 21.4; IR(film): 2959, 2926, 1590, 1434,1348, 1166, 1090, 1041, 950, 736, 672 cm^{-1}; HRMS (EI) calcd for $C_{22}H_{24}N_2O_3S$ 396.1508; found:396.1510.

7-Fluoro-2, 10-dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3u)



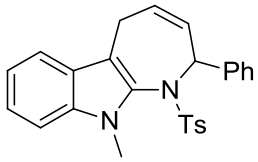
White solid; Yield 72% (55 mg); m.p. 200.6-201.4 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.55 (d, *J* = 8.4 Hz, 2H), 7.24 (dd, *J* = 9.0 Hz, 4.2 Hz, 1H), 7.18 (d, *J* = 6.4 Hz, 2H), 7.03-7.01 (m, 2H), 5.39-5.35 (m, 1H), 5.27-5.23(m, 1H), 4.98-4.96(m, 1H), 3.78(s, 3H), 3.07(dd, *J* = 20.4 Hz, 6.0 Hz, 1H), 2.42(s, 3H), 2.19 (dd, *J* = 21.0 Hz, 3.0 Hz, 1H), 1.14 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (125MHz, $CDCl_3$) δ157.2 (d, *J* = 194.5 Hz), 143.8, 136.1, 132.0, 131.4, 129.5, 129.2, 128.6, 125.35(d, *J* = 8.0Hz), 125.26, 110.7(d, *J* = 21.9 Hz), 110.6 (d, *J* = 7.8 Hz), 109.5 (d, *J* = 3.9Hz), 103.2 (d, *J* = 19.5Hz), 54.3, 29.8, 24.2, 21.6, 21.5; IR(film): 2959, 2926, 1582, 1489,1349, 1163, 1131, 950, 896, 706, 672cm^{-1}; HRMS (EI) calcd for $C_{21}H_{21}FN_2O_2S$ 384.1308; found: 384.1308.

7-Bromo-2, 10-dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3v)



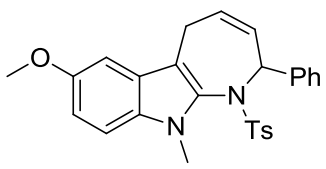
White solid; Yield 74% (66 mg); m.p. 74.5-75.2 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.53 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 1.8 Hz, 1H), 7.33 (dd, *J* = 9.0 Hz, 1.8 Hz, 1H), 7.20 (d, , *J* =9.0 Hz,1H), 7.17 (d, *J* = 7.8 Hz, 2H), 5.38-5.35 (m, 1H), 5.25-5.22 (m, 1H), 4.98-4.95 (m, 1H), 3.77 (s, 3H), 3.08 (dd, *J* = 21.0 Hz, 6.0 Hz, 1H), 2.41(s, 3H), 2.16 (dd, *J* = 21.0 Hz, 3.0 Hz, 1H), 1.13 (d, *J* = 7.2Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.9, 135.9, 134.0, 131.2, 129.4, 129.2, 128.5, 126.8, 125.10,125.09, 120.9, 112.4, 111.3, 109.1, 54.2, 29.7, 24.0, 21.6, 21.5; IR(film): 2965, 2926, 2855, 1599, 1471,1348, 1167, 1145, 948, 793, 705, 674 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{21}BrN_2O_2S$ 444.0507; found:444.0499.

10-Methyl-2-phenyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3w)



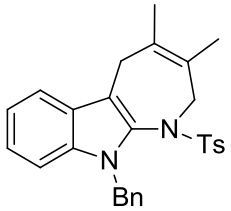
White solid; Yield 71% (61 mg); m.p. 162.1-163.2 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.18 (td, *J* = 7.2 Hz, 1.2 Hz, 2H), 7.15 (d, *J* = 3.2 Hz, 3H), 7.13-7.05 (m, 3H), 6.09 (t, *J* = 4.4 Hz, 1H), 5.71-5.66 (m, 1H), 5.52-5.47 (m, 1H) , 3.31 (d, *J* = 6.8 Hz, 1H), 3.12 (s, 3H), 2.45 (s, 3H), 2.29-2.22 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.8, 138.0, 136.4, 135.1, 129.9, 129.3, 128.7, 128.5, 128.2,128.1, 127.9, 125.8, 125.1, 122.1, 118.9, 118.2, 109.9, 109.8, 61.6, 28.8, 23.9, 21.7; IR (film): 3060, 2924, 1597, 1471, 1350, 1164, 1090, 813, 751, 670cm^{-1}; HRMS (EI) calcd for $C_{26}H_{24}N_2O_2S$ 428.1558; found: 428.1563.

7-Methoxy-10-methyl-2-phenyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3x)



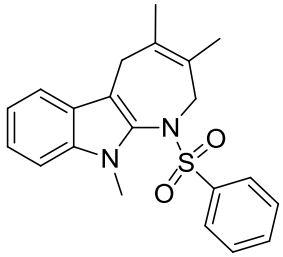
White solid; Yield 66% (60 mg); m.p. 149.3-150.7 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.17-7.13 (m, 5H), 7.01 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 6.86-6.83 (m, 2H), 6.08 (t, *J* = 5.2 Hz, 1H), 5.67-5.65 (m, 1H), 5.51-5.50 (m, 1H) , 3.83 (s, 3H), 3.22 (d, *J* = 20.4Hz, 6.8 Hz, 1H), 3.08 (s, 3H), 2.45 (s,3H), 2.23 (dd, *J* =20.4 Hz, 2.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.7,143.8, 138.1, 136.5, 130.5, 130.2, 129.3, 128.7, 128.5, 128.2, 128.1,127.9, 125.9, 125.2, 112.3, 110.7, 109.4, 100.1, 61.6, 55.8, 28.8, 23.9, 21.6; IR (film): 3030, 2929, 2828, 1492, 1348, 1164, 1091, 802, 758, 672 cm^{-1}; HRMS (EI) calcd for $C_{27}H_{26}N_2O_3S$ 458.1664; found: 458.1661.

10-Benzyl-3, 4-dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3y)



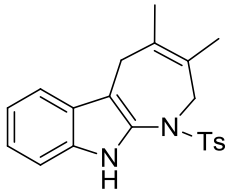
Pink solid; Yield 50% (46 mg); m.p. 67.0-68.0 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.26 (d, *J* = 8.4 Hz, 1H), 7.23 (t, *J* = 2.8 Hz, 1H), 7.21 (s, 1H), 7.20-7.19 (m, 1H), 7.17 (d, *J* = 8.0 Hz, 3H), 7.10-7.06 (m, 1H), 7.01 (d, *J* = 6.4 Hz, 2H), 5.80 (d, *J* = 16.4 Hz, 1H), 5.37 (d, *J* = 16.8 Hz, 1H), 4.28 (d, *J* = 17.6 Hz, 1H), 3.70 (d, *J* = 17.6 Hz, 1H), 3.13 (d, *J* = 20.8 Hz , 1H), 2.40 (s, 3H), 2.20 (d, *J* = 20.8 Hz, 1H), 1.54 (s, 3H), 1.30 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ143.6, 138.5, 136.4, 135.0, 133.0, 129.0, 128.5, 128.1, 127.0, 126.7, 126.4, 125.8, 122.6, 122.5, 119.3, 118.2, 110.5, 109.0, 54.3, 46.4, 31.4, 21.6, 21.5, 20.9; IR(film): 3054, 3027, 2917, 2852, 1617, 1597, 1462, 1353, 1163, 1089, 908, 666 cm^{-1}; HRMS (EI) calcd for $C_{28}H_{28}N_2O_2S$: 456.1871; found: 456.1867.

3, 4, 10-Trimethyl-1-(phenylsulfonyl)-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3z)



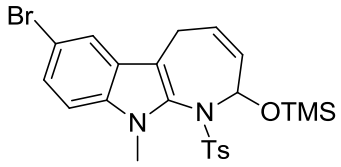
White solid; Yield 55% (40 mg); m.p. 181.6-182.4 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61 (dd, *J* = 8.4 Hz, 1.2 Hz, 2H), 7.56-7.51 (m, 1H), 7.39 (s, 1H), 7.35 (d, *J* = 8.4 Hz, 2H), 7.32 (d, *J* = 8.4 Hz, 1H), 7.28-7.25 (m, 1H), 7.12-7.08(m, 1H), 4.48 (d, *J* = 16.8 Hz, 1H), 4.07 (d, *J* = 17.6 Hz, 1H), 3.81 (s, 3H), 3.15 (d, *J* = 23.2 Hz, 1H), 2.23 (d, *J* = 20.8 Hz, 1H), 1.61 (s, 3H), 1.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.3, 135.3, 132.9, 132.7, 128.3, 128.0, 126.9, 125.4, 122.4, 122.2, 119.1, 118.1, 109.7, 107.7, 54.7, 31.5, 29.6, 21.7, 21.1; IR(film): 3057, 2916, 2852, 1620, 1597, 1447, 1353, 1163, 1088, 910, 783, 658 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{22}N_2O_2S$: 366.1402; found: 366.1404.

3, 4-Dimethyl-1-tosyl-1, 2, 5, 10- tetrahydroazepino[2,3-b]indole (3A)



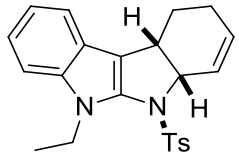
Yellow oil; Yield 52% (38 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.79 (s, 1H), 7.34 (dd, *J* = 6.8 Hz, *J* = 1.6 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 1H), 7.24 (d, *J* = 8.0Hz, 1H), 7.13-7.09 (m, 1H), 7.06-7.01 (m, 3H), 4.25 (s, 1H), 3.19 (s, 2H), 2.27 (s, 3H), 1.55 (s, 3H), 1.39 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 135.8, 133.3, 132.5, 132.4, 129.1, 127.1, 124.3, 121.9, 119.5, 117.4, 110.7, 101.7, 54.1, 30.6, 21.5, 21.2, 20.8.; IR(film): 3400, 3057, 2922, 2861, 1620, 1597, 1462, 1354, 1160, 1087, 909, 741, 666 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{22}N_2O_2S$: 366.1402; found: 366.1403.

7-Bromo-10-methyl-1-tosyl-2- ((trimethylsilyl)oxy)-1,2,5,10-tetrahydroazepino[2,3-b]indole (3B)



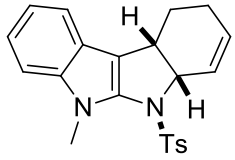
White solid; Yield 84% (87 mg); m. p. 67.4-68.6 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.51 (d, *J* = 7.2 Hz, 2H), 7.47 (s, 1H), 7.31 (d, *J* = 8.4 Hz, 1H), 7.19 (d, *J* = 6.0Hz, 3H), 6.17 (s, 1H), 5.55-5.52 (m, 1H), 5.40 (d, *J* = 11.4 Hz, 1H), 3.77 (s, 3H), 3.00 (dd, *J* = 20.4 Hz, *J* = 6.6 Hz, 1H), 2.42 (s, 3H), 1.90 (s, *J* = 20.4 Hz, 1H), 0.21(s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.1, 135.9, 134.2, 130.6, 129.3, 128.6, 128.0, 127.6, 126.6, 125.0, 120.9, 112.3, 111.3, 109.8, 79.4, 29.8, 23.0, 21.6, 0.13.; IR (film): 3011, 2956, 2856, 1596, 1472, 1351, 1253, 1168, 874, 705, 675 cm^{-1}; HRMS (EI) calcd for $C_{23}H_{27}BrN_2O_3SSi$: 518.0695; found: 518.0691.

6-Ethyl-5-tosyl-1, 2, 4a, 5, 6, 10c- hexahydroindolo[2,3-b]indole (5a)



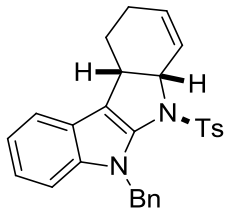
Yellow oil; Yield 50% (39 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.45 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 1H), 7.30 (d, *J* = 7.6 Hz, 1H), 7.19-7.14 (m, 3H), 7.09-7.05(m, 1H), 5.80-5.76 (m, 1H), 5.69-5.65(m, 1H), 5.00-4.97 (m, 1H), 4.54-4.47 (m, 1H), 4.45-4.33 (m, 1H), 2.80-2.77(m, 1H), 2.37(s, 3H), 2.18-2.10 (m, 1H), 1.75-1.71 (m, 2H), 1.68-1.61 (m, 1H), 1.43 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ144.2, 142.0,139.2,133.5, 132.1, 129.6, 127.7, 124.9, 123.3, 120.5, 119.8, 117.8, 111.0, 110.8, 71.3, 39.7, 34.7, 23.2, 21.6, 20.1, 14.8; IR(film):3030, 2927, 2872, 1597, 1447, 1356, 1167, 1089, 990, 910, 660 cm^{-1}; HRMS (EI) calcd for $C_{23}H_{24}N_2O_2S$:392.1558; found: 392.1552.

6-Methyl-5-tosyl-1, 2, 4a, 5, 6, 10c- hexahydroindolo[2,3-b]indole (5b)



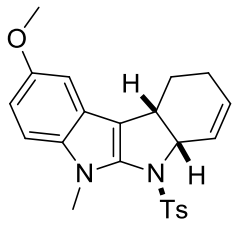
Yellow oil; Yield 45% (34 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 8.4 Hz, 1H), 7.31 (d, *J* = 7.6 Hz, 1H), 7.20-7.14(m, 3H), 7.10-7.06(m, 1H), 5.83-5.80 (m, 1H), 5.70-5.67 (m, 1H), 4.98-4.96 (m, 1H), 3.90 (s, 3H), 2.82-2.79 (m, 1H), 2.37 (s, 3H), 2.12(dd, *J* = 12.4 Hz, 3.2 Hz,1H), 1.77-1.70 (m, 2H), 1.70-1.62 (m,1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ144.3, 142.5, 140.1, 133.5, 132.3, 129.6, 127.8, 125.1, 122.9, 120.6, 119.9, 117.8, 110.40, 110.36, 71.1, 35.0, 31.6, 23.4, 21.6, 20.2; IR(film):3030,2926, 2866,1597, 1481,1353, 1167, 1089, 986, 730,661 cm^{-1}; HRMS (EI) calcd for $C_{22}H_{22}N_2O_2S$:378.1402; found: 378.1407.

6-Benzyl-5-tosyl-1, 2, 4a, 5, 6, 10c- hexahydroindolo[2,3-b]indole (5c)



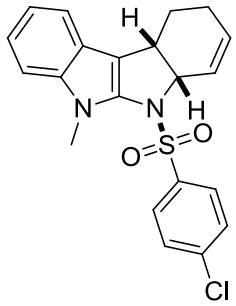
White oil; Yield 50% (46 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.4 Hz, 2H), 7.31 (dd, *J* = 6.4 Hz, 2.4 Hz, 1H), 7.23-7.21 (m, 2H), 7.19-7.16 (m, 4H), 7.10-7.05 (m, 2H),7.02 (d, *J* = 6.8 Hz, 2H) 5.89 (d, *J* = 16.8 Hz, 1H), 5.83 (d, *J* = 10.4 Hz, 1H), 5.43 (d, *J* = 16.8 Hz, 1H), 5.01 (d, *J* = 2.8 Hz, 1H), 2.86 (dd, *J* = 6.8 Hz, 4.0 Hz, 1H), 2.39 (s, 3H), 2.15 (d, *J* = 12.4 Hz, 1H), 1.77-1.71 (m, 2H), 1.71-1.64 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.4, 142.7, 139.8, 137.7, 133.6, 132.1, 129.6, 128.3, 127.9, 127.0, 126.6, 125.0, 123.6, 120.9, 120.2, 117.9, 111.70, 111.66, 71.4, 48.1, 34.9, 23.3, 21.7, 20.2; IR(film): 3029, 2926, 2875, 1597, 1467, 1450, 1353, 1160, 1089, 909, 739, 661 cm^{-1}; HRMS (MALDI-TOF) calcd for $C_{26}H_{24}N_2O_3S + H^+$ ($[M+H]^+$): 455.1788; found: 455.1790.

9-Methoxy-6-methyl-5-tosyl-1, 2, 4a, 5, 6, 10c- hexahydroindolo[2,3 -b]indole (5d)



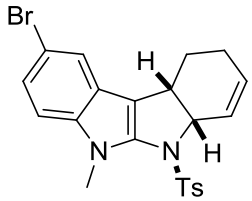
Yellow oil; Yield 46% (38 mg); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.42 (d, *J* = 7.8 Hz, 2H), 7.22 (d, *J* = 9.0 Hz, 1H), 7.15 (d, *J* = 7.8 Hz, 2H), 6.83 (dd, *J* = 9.0 Hz, 1.8 Hz, 1H), 6.76 (s, 1H), 5.82 (d, *J* =10.2 Hz, 1H), 5.68 (d, *J* = 10.2 Hz, 1H), 4.95 (s, 1H), 3.86 (s, 3H), 3.82 (s, 3H), 2.78 (t, *J* = 3.0 Hz, 1H), 2.38 (s, 3H), 2.10 (dd, *J* = 13.2 Hz, 2.4 Hz, 1H), 1.76 (s, 2H), 1.69-1.64 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.2, 144.3, 143.0, 135.4, 133.5, 132.3, 129.6, 127.8, 125.1, 123.2, 110.9, 109.9, 109.5, 100.7, 70.9, 55.9, 34.9, 31.6, 23.3, 21.6, 20.2; IR(film):3036, 2926, 2863, 1574, 1494, 1353, 1166, 1089, 907, 728 cm^{-1}; HRMS (EI) calcd for $C_{23}H_{24}N_2O_3S$: 408.1508; found: 408.1508 .

5-((4-Chlorophenyl) sulfonyl)-6-methyl-1, 2, 4a, 5, 6, 10c- hexahy droindolo [2, 3-b] indole (5e)



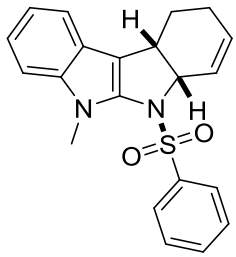
White solid; Yield 55% (44 mg); m.p. 192.6-193.6 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (d, *J* = 8.4 Hz, 2H), 7.35-7.32 (m, 4H), 7.21 (t, *J* = 8.0 Hz, 1H), 7.10 (t, *J* = 7.6 Hz, 1H), 5.83 (d, *J* = 11.6 Hz, 1H), 5.67 (d, *J* = 10.0 Hz, 1H), 4.96 (s, 1H), 3.89(s, 3H), 2.84 (dd, *J* = 6.8 Hz, 4.0 Hz, 1H), 2.15(d, *J* = 12.4 Hz, 1H), 1.77-1.74 (m, 2H), 1.71-1.64 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.1, 140.15, 140.10, 134.9, 132.7, 129.3. 129.2, 124.7, 122.8, 121.0, 120.1, 117.9, 110.6, 110.4, 71.2, 35.1, 31.6, 23.3, 20.1; IR(film):3033, 2926, 2869, 1571, 1480, 1357, 1171, 1092, 911, 820, 669 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{19}ClN_2O_2S$: 398.0856; found: 398.0858.

9-Bromo-6-methyl-5-tosyl-1, 2, 4a, 5, 6, 10c- hexahydroindolo[2,3 -b]indole (5f)



Yellow oil; Yield 40% (36 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40-7.39 (m, 3H), 7.27-7.24 (m, 1H), 7.20-7.14 (m, 3H), 5.80-5.76 (m, 1H), 5.84-5.81 (m, 1H), 5.67(d, *J* =10.0 Hz, 1H), 3.88(s, 3H), 2.74 (d, *J* = 4.4 Hz, 1H), 2.38(s, 3H), 2.06(dd, *J* = 12.0 Hz, 1H), 1.80-1,72 (m, 2H), 1.68-1.62 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 143.5, 138.7, 133.3, 132.4, 129.7, 127.7, 124.9, 124.3, 123.3, 120.2, 113.4, 111.8, 109.9, 71.0, 34.9, 31.7, 23.2, 21.6, 20.1; IR(film):3036, 2926, 2872, 1561, 1463, 1167, 1089, 909, 790, 731 cm^{-1}; HRMS (EI) calcd for $C_{22}H_{21}BrN_2O_2S$: 456.0507; found: 456.0513.

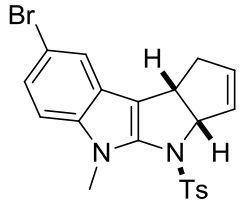
6-Methyl-5-(phenylsulfonyl)-1, 2, 5, 6- tetrahydroindolo[2,3-b]indole (5g)



Yellow solid; Yield 50% (36 mg); m.p. 164.0-165.0 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57-7.53 (m, 3H), 7.38-7.33 (m, 3H), 7.29 (d, *J* = 3.6 Hz, 1H), 7.19 (dt, *J* = 8.4 Hz, 1.2 Hz, 1H), 7.10-7.06 (m, 1H), 5.83-5.79 (m, 1H), 5.70-5.67 (m, 1H), 4.98 (dd, *J* = 3.2 Hz, 1.2 Hz, 1H), 3.91 (s, 3H), 2.77-2.74

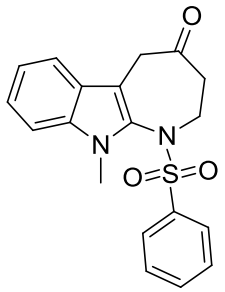
(m, 1H), 2.13-2.08 (m, 1H), 2.06 (dd, *J* = 12.0 Hz, 1H), 1.76-1.71 (m, 2H), 1.70-1.61 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ142.4, 140.1, 136.3, 133.4, 132.5, 128.9, 127.8, 125.0, 122.9, 120.7, 120.0, 117.8, 110.5, 110.4, 71.1, 35.0, 31.6, 23.3, 20.1; IR(film): 3030, 2927, 2870, 1572, 1446, 1354, 1170, 1089, 910, 821, 609 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{20}N_2O_2S$: 364.1245; found: 364.1248 .

(4aS, 10cS)-7-Bromo-10-methyl-1-tosyl-1, 2, 5, 10-tetrahydro-2, 5-methanoazepino [2, 3-b] indole (7)



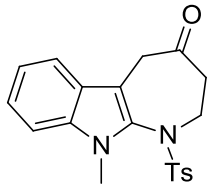
Yellow oil; Yield 34% (30 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40 (d, *J* = 1.6 Hz, 1H), 7.35 (d, *J* = 8.0 Hz, 2H), 7.26 (dd, *J* = 8.8 Hz, 2.0 Hz, 1H), 7.20-7.15 (m, 3H), 5.80-5.77 (m, 2H), 5.42 (dd, *J* = 4.0, 2.4Hz, 1H), 3.89 (s, 3H), 3.10-3.06 (m, 1H), 2.51-2.43 (m, 2H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.6, 142.2, 139.2, 133.3, 131.9, 129.6, 128.5, 128.0, 123.8, 123.6, 120.4, 113.3, 112.3, 111.9, 82.6, 37.4, 36.2, 32.0, 21.6; IR(film):3057, 2921, 2855, 1596, 1462, 1355, 1169, 1089, 815, 734, 661 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{19}BrN_2O_2S$: 442.0351; found: 442.0351.

10-Methyl-1-(phenylsulfonyl)-2, 3, 5, 10- tetrahydroazepino[2,3-b]indol-4(1H)-one (9a)



Yellow oil; Yield 58% (41 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, *J* = 7.6 Hz, 2H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.51 (t, *J* =7 .6 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.33 (t, *J* = 7.6 Hz, 1H), 7.17 (t, *J* = 7.6 Hz, 1H), 4.48-4.41 (m, 1H), 3.84-3.78 (m, 4H), 3.35 (d, *J* = 16.8 Hz, 1H), 2.64 (d, *J* = 16.8 Hz, 1H), 2.55 (t, *J* = 6.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.0, 138.6, 135.2, 133.8, 131.7, 129.4, 127.9, 124.8, 123.2, 120.0, 118.6, 110.1, 103.0, 50.2, 41.6, 38.7, 30.1; IR(film): 3069, 2928, 1710, 1472, 1356, 1167, 1066, 909, 746 cm^{-1}; HRMS (EI) calcd for $C_{19}H_{18}N_2O_3S$ 354.1038; found: 354.1039.

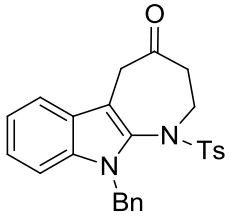
10-Methyl-1-tosyl-2, 3, 5, 10- tetrahydroazepino[2,3-b]indol-4(1H)-one (9b)



Yellow oil; Yield 50% (37 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.61(d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.37 (d, *J* = 8.4 Hz, 1H), 7.32 (d, *J* = 6.8 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.18-7.14 (m,

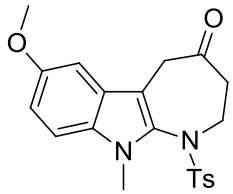
1H), 4.43 (m, 1H), 3.81-3.75 (m, 4H), 3.35 (d, *J* = 16.4 Hz, 1H), 2.69 (d, *J* = 16.4 Hz, 1H), 2.63-2.56 (m, 1H), 2.54-2.46 (m, 1H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ205.9, 144.9, 135.8, 135.2, 131.9, 130.0, 127.9, 124.8, 123.1, 119.9, 118.6, 110.1, 102.8, 50.2, 41.8, 38.7, 30.1, 21.7 ; IR(film): 3070, 2927, 1712, 1471, 1356, 1166, 1090, 911, 817, 746, 669 cm^{-1}; HRMS (EI) calcd for $C_{20}H_{20}N_2O_3S$ 368.1195; found: 368.1199.

10-Benzyl-1-tosyl-2, 3, 5, 10- tetrahydroazepino[2,3-b]indol-4(1H)-one (9c)



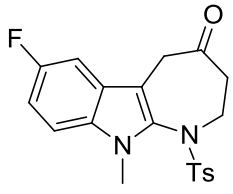
Yellow oil; Yield 62% (55 mg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.29-7.26 (m, 3H), 7.26- 7.20 (m, 3H), 7.16 (m, 1H), 7.02-7.00 (m, 2H), 5.76 (d, *J* = 16.4 Hz, 1H), 5.43 (d, *J* = 16.4 Hz, 1H), 4.22-4.15 (m, 1H), 3.34 (d, *J* = 16.8 Hz, 1H), 3.17-3.11 (m, 1H), 2.62 (d, *J*= 17.2 Hz, 1H), 2.48-2.33 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.1, 144.8, 137.8, 135.8, 135.1, 131.7, 130.0, 128.7, 127.8, 127.3, 126.6, 125.0, 123.4, 120.1, 118.7, 110.7, 104.6, 49.8, 46.7, 41.6, 38.6, 21.7; IR(film): 3066, 2925, 1712, 1597, 1462, 1352, 1165, 1089, 908, 745, 666 cm^{-1}; HRMS (MALDI-TOF) calcd for $C_{26}H_{24}N_2O_3S + H^+$ ($[M+H]^+$): 445.1580; found: 445.1580.

7-Methoxy-10-methyl-1-tosyl-2, 3, 5, 10- tetrahydroazepino[2,3-b]indol-4(1H)-one (9d)



White solid; Yield 59% (46 mg); m.p. 67.4-68.9 ^oC; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, *J* = 8.4 Hz, 2H), 7.31-7.25 (m, 3H), 6.97 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 6.86 (d, *J* = 2.4 Hz, 1H), 4.46-4.40 (m, 1H), 3.84 (s, 3H), 3.80-3.73 (m, 4H), 3.30 (d, *J* = 16.0 Hz, 1H), 2.68 (d, *J* = 16.4 Hz, 1H), 2.65-2.58 (m, 1H); 2.54-2.50 (m, 1H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.0,154.4, 144.8, 135.9, 132.1, 130.4, 130.0, 127.9, 124.9, 113.6, 111.0, 102.2, 100.0, 55.8, 50.1, 41.9, 38.8, 30.1, 21.7; IR(film): 2927, 2828, 1711, 1625, 1597, 1492, 1356, 1165, 1090, 1037, 911, 801, 732, 672 cm^{-1}; HRMS (EI) calcd for $C_{21}H_{22}N_2O_4S$: 398.1300; found: 398.1298 .

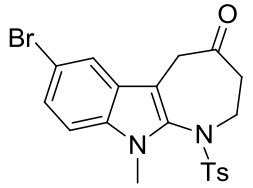
7-Fluoro-10-methyl-1-tosyl-2, 3, 5, 10- tetrahydroazepino[2,3-b]indol-4(1H)-one (9e)



White solid; Yield 70% (54 mg); m.p. 131.4-132.7 ^oC; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 8.4 Hz, 2H), 7.31-7.26 (m, 3H), 7.10-7.02 (m, 2H), 4.42 (m, 1H), 3.79-3.74 (m, 4H), 3.25 (d, *J* = 16.4

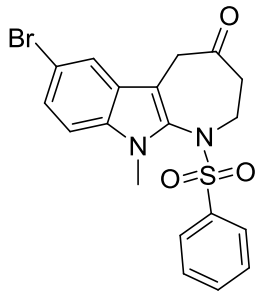
Hz, 1H), 2.68 (d, *J* = 16.4 Hz, 1H), 2.63-2.57 (m, 1H), 2.53-2.49 (m, 1H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.5, 158.0 (d, *J* = 234.8 Hz), 145.0, 135.7, 133.2, 131.7, 130.1, 127.9, 124.9 (d, *J* = 9.8 Hz), 111.6 (d, *J* = 26.1 Hz), 111.1(d, *J* = 9.4 Hz), 103.5 (d, *J* = 23.7 Hz), 102.8 (d, *J* = 4.9 Hz) , 50.0, 41.7, 38.7, 30.3, 21.7; IR(film): 3063, 2927, 1712, 1597, 1406, 1359, 1164, 1090, 892, 733, 706 cm^{-1}; HRMS (EI) calcd for $C_{20}H_{19}FN_2O_3S$: 386.1100; found: 386.1102 .

7-Bromo-10-methyl-1-tosyl-2, 3, 5, 10- tetrahydroazepino[2,3-b]indol-4(1H)-one (9f)



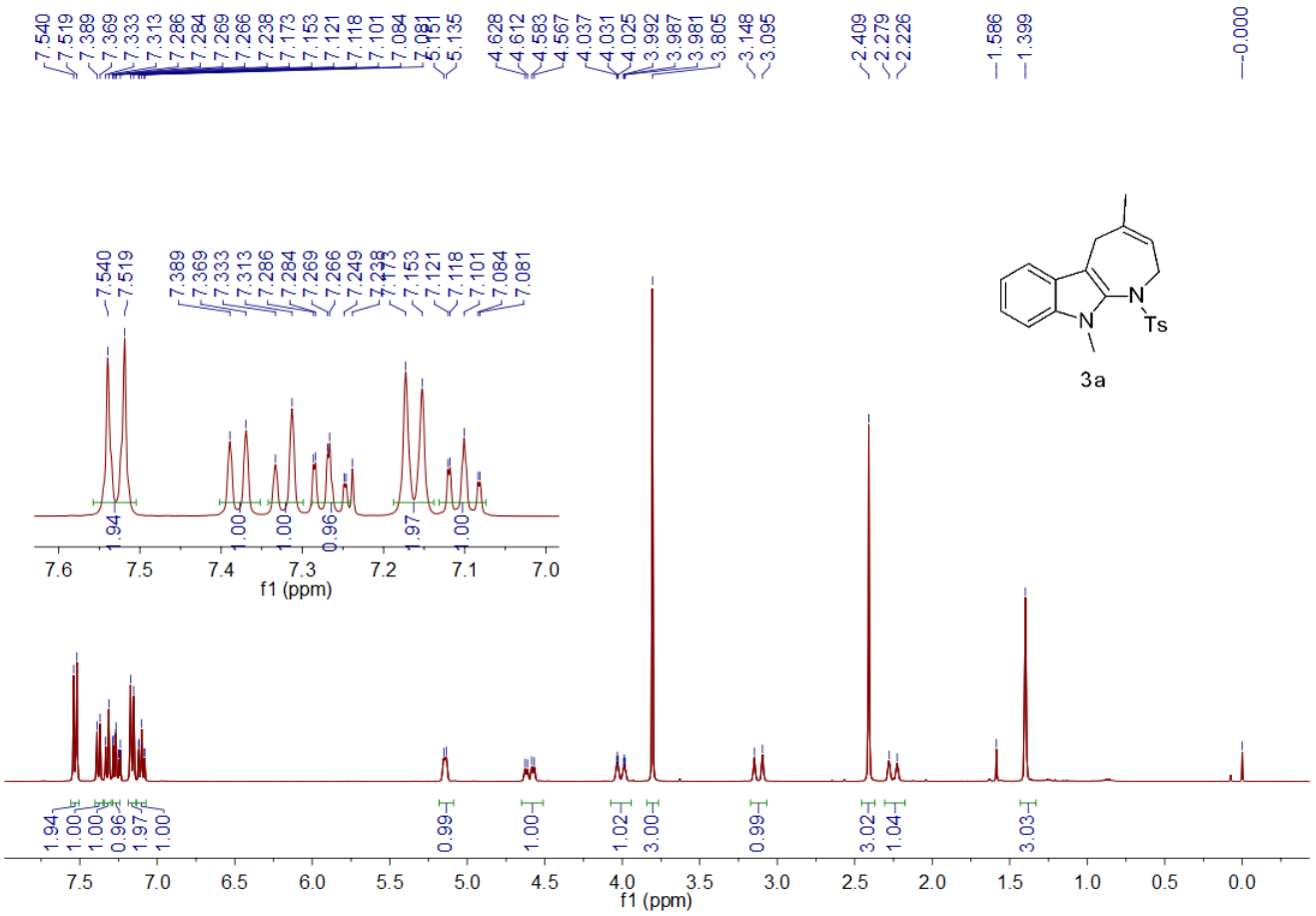
White solid; Yield 85% (79 mg); m.p. 93.2- 94.5 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59-7.56(m, 3H), 7.36 (dd, *J* = 8.8 Hz, 2.0 Hz, 1H), 7.29(d, *J* = 8.0 Hz, 1H), 7.22(d, *J* = 8.8 Hz, 1H), 4.45-4.38(m, 1H), 3.80-3.74 (m, 4H), 3.26 (d, *J* = 16.4 Hz, 1H), 2.64 (d, *J* = 16.4 Hz, 1H), 2.59-2.56 (m, 1H); 2.52-2.49 (m, 1H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.3, 145.0, 135.6, 133.7, 132.9, 130.1, 127.8, 126.2, 125.9, 121.2, 113.2, 111.7, 102.4, 50.1, 41.7, 38.5, 30.2, 21.7; IR (film): 3063, 2945, 1710, 1625, 1570, 1473, 1302, 1167, 1090, 911, 792, 725, 689 cm^{-1}; HRMS (EI) calcd for $C_{20}H_{19}BrN_2O_3S$ 446.0300; found: 446.0305.

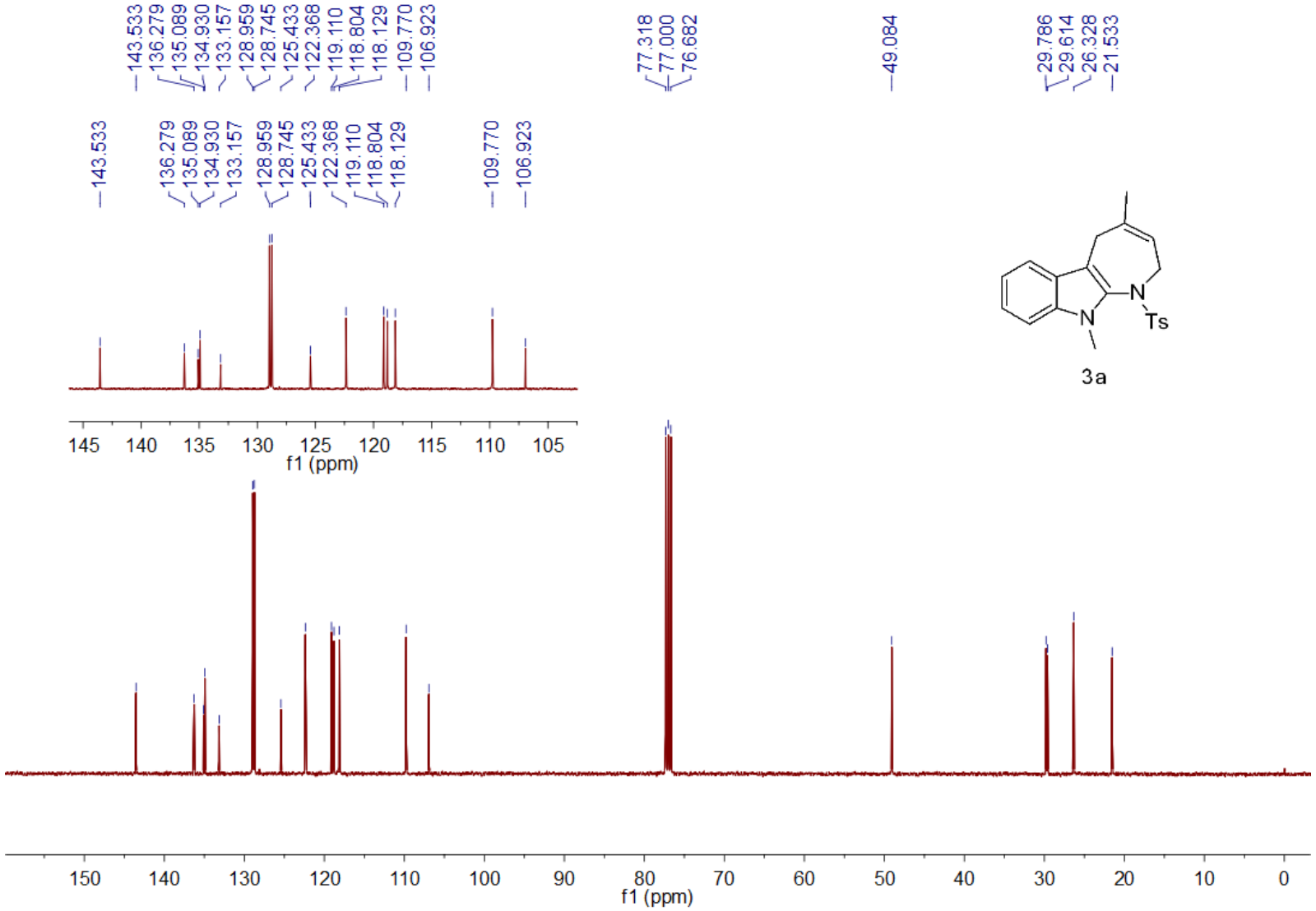
7-Bromo-10-methyl-1-(phenylsulfonyl)-2, 3, 5, 10-tetrahydroazepino[2,3-b]indol -4(1H)-one (9g)



White solid; Yield 80% (69 mg); m.p. 85.2-86.3 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71-7.67 (m, 3H), 7.56 (d, *J* = 1.6 Hz, 1H), 7.51 (t, *J* = 8.0 Hz, 2H), 7.38 (dd, *J* = 8.8 Hz, 1.6 Hz, 1H), 7.24 (d, *J* = 8.8 Hz, 1H), 4.46-4.40 (m, 1H), 3.83-3.77 (m, 4H), 3.26 (d, *J* = 16.4 Hz, 1H), 2.61-2.52 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.3, 138.4, 133.9, 133.8, 132.6, 129.5, 127.8, 126.2, 126.0, 121.2, 113.3, 111.7, 102.7, 50.1, 41.5, 38.5, 30.3; IR (film): 3063, 2945, 1710, 1473, 1447, 1365, 1167, 1090, 911, 792, 725, 689 cm^{-1}; HRMS (EI) calcd for $C_{19}H_{17}BrN_2O_3S$: 432.0143; found: 432.0146.

Copies of NMR Spectra

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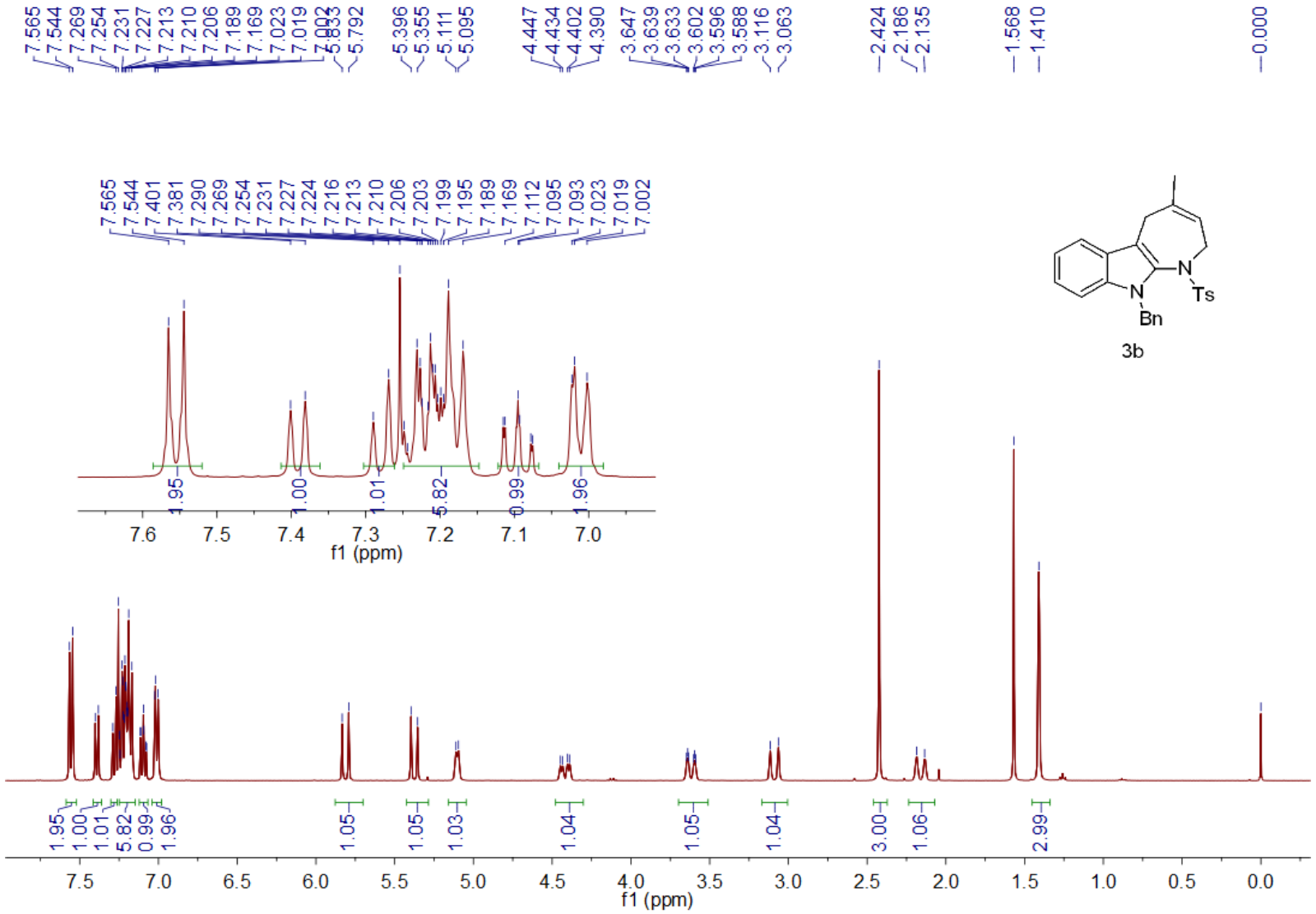
135.089
134.930
133.157
128.959
128.745
125.433
122.368
119.110
118.804
118.129
109.770
106.923
77.318
77.000
76.682
49.084
29.786
29.614
26.328
21.533
N
N
Ts
3a
f1 (ppm)
f1 (ppm)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7.210
7.206
7.189
7.169
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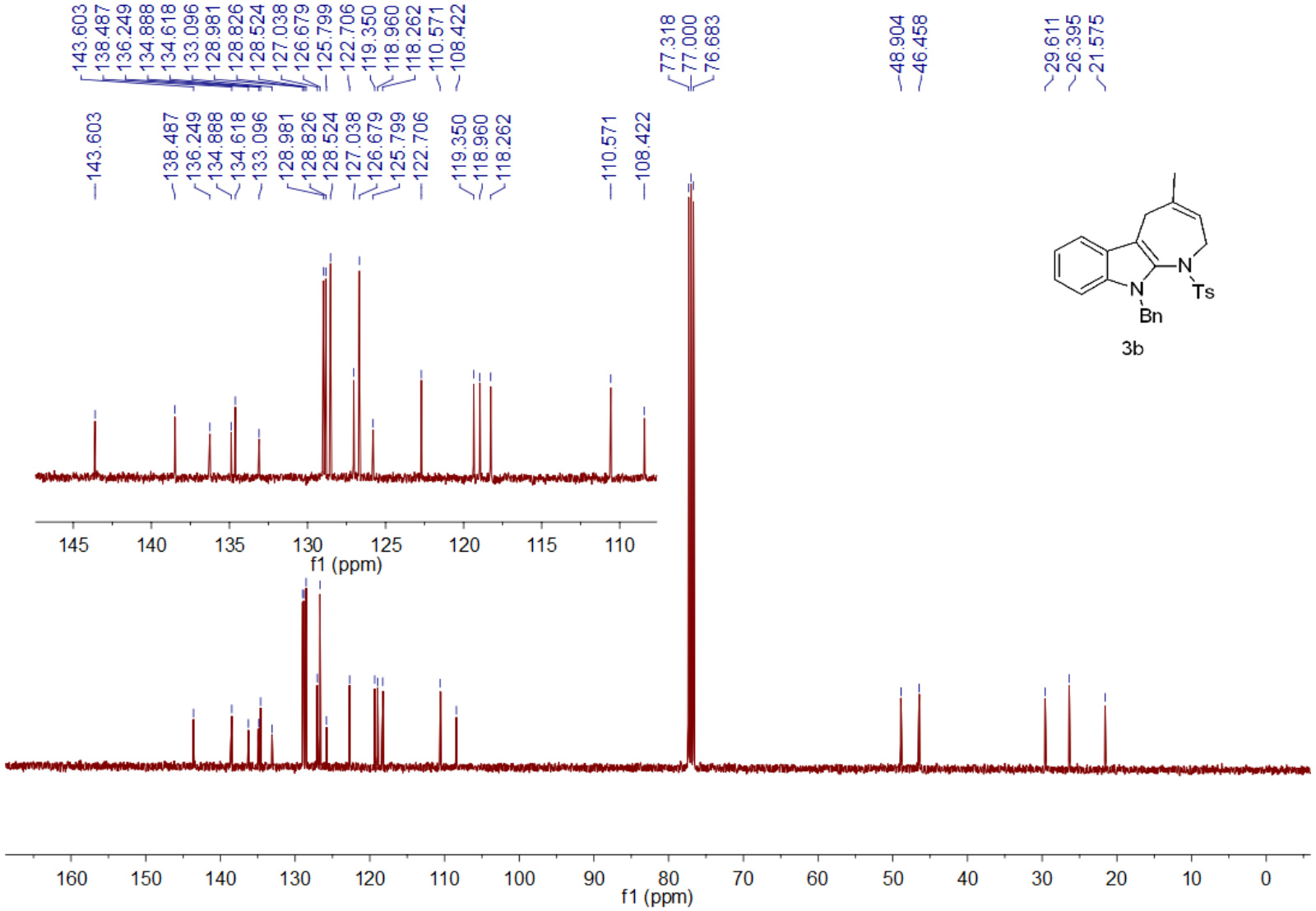
7.019
5.792
5.396
5.355
5.111
5.095
4.447
4.434
4.402
4.390
3.647
3.639
3.633
3.602
3.596
3.588
3.116
3.063
2.424
2.186
2.135
1.568
1.410
0.000
7.565
7.544
7.401
7.381
7.290
7.269
7.254
7.231
7.227
7.224
7.216
7.213
7.210
7.206
7.203
7.199
7.195
7.189
7.169
7.112
7.095
7.093
7.023
7.019
7.002
1.95
1.00
1.01
5.82
0.99
1.96
7.6
7.5
7.4
7.3
7.2
7.1
7.0
f1 (ppm)
N
N
Ts
Bn
3b
1.95
1.00
1.01
5.82
0.99
1.96
1.05
1.05
1.03
1.04
1.05
1.04
3.00
1.06
2.99
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)


143.603
138.487
136.249
134.888
134.618
133.096
128.981
128.826
128.524
127.038
126.679
125.799
122.706
119.350
118.960
118.262
110.571
108.422
77.318
77.000
76.683
48.904
46.458
29.611
26.395
21.575
145
140
135
130
125
120
115
110
f1 (ppm)
160
150
140
130
120
110
100
90
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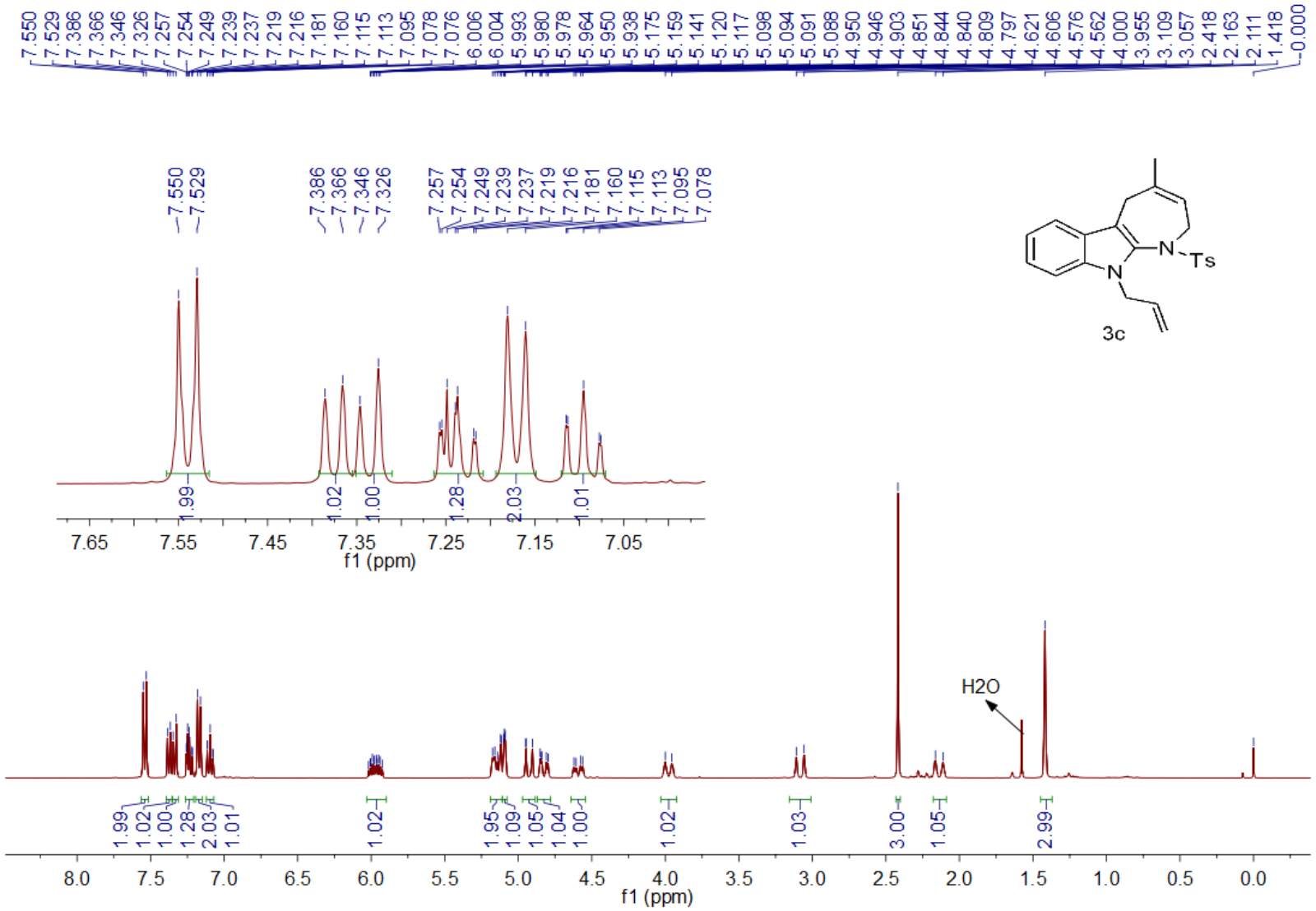
70
60
50
40
30
20
10
0
f1 (ppm)
N
N
Ts
Bn
3b


7.550
7.529
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7.366
7.346
7.326
7.257
7.254
7.249
7.239
7.237
7.219
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4.621
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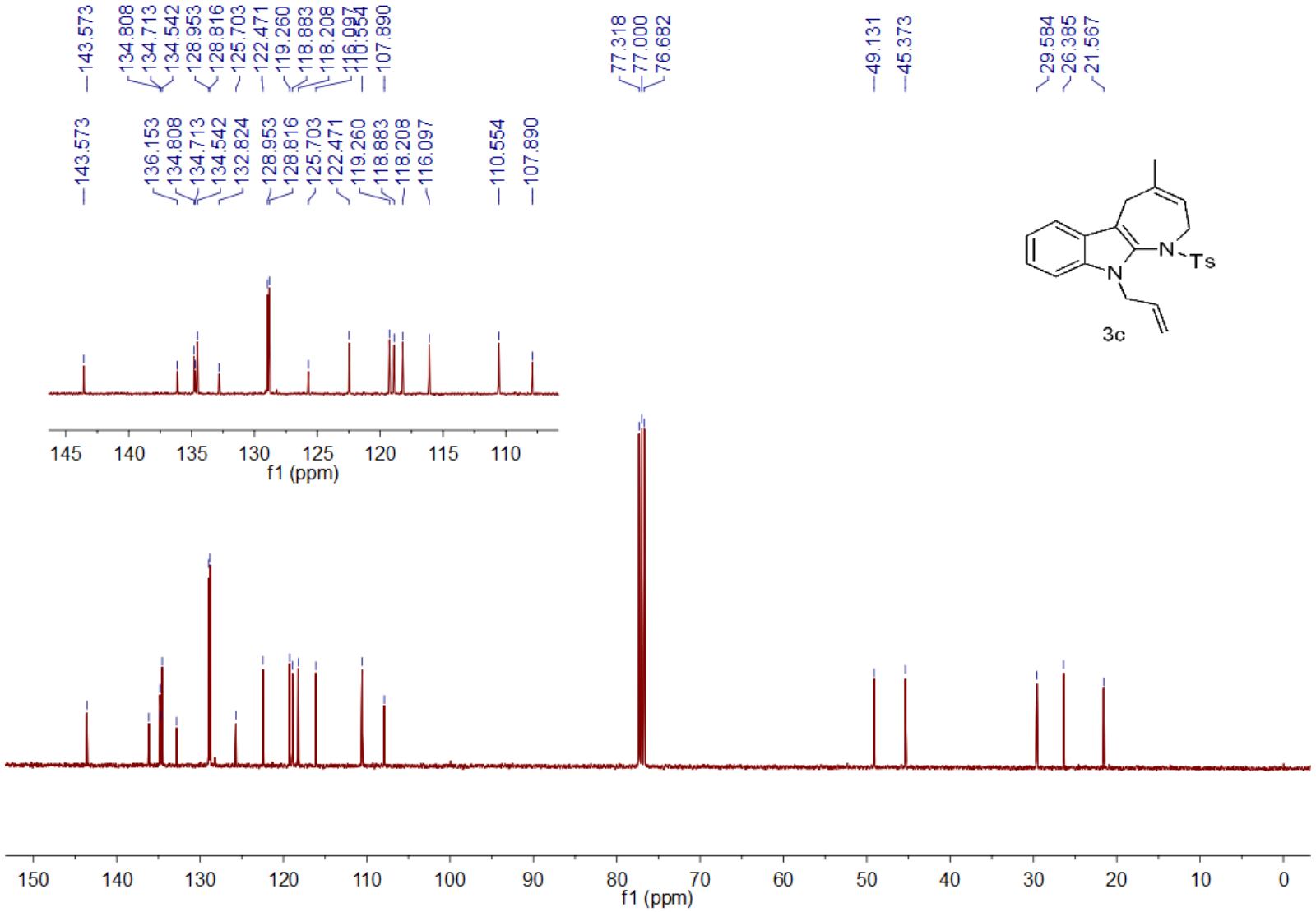
4.000
3.955
3.109
3.057
2.418
2.163
2.111
1.418
0.000
1.99
1.02
1.00
1.28
2.03
1.01
7.65
7.55
7.45
7.35
7.25
7.15
7.05
f1 (ppm)
N
Ts
N
3c
H2O
1.99
1.02
1.00
1.28
2.03
1.01
1.02
1.95
1.09
1.05
1.04
1.00
1.02
1.03
3.00
1.05
2.99
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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128.816
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122.471
119.260
118.883
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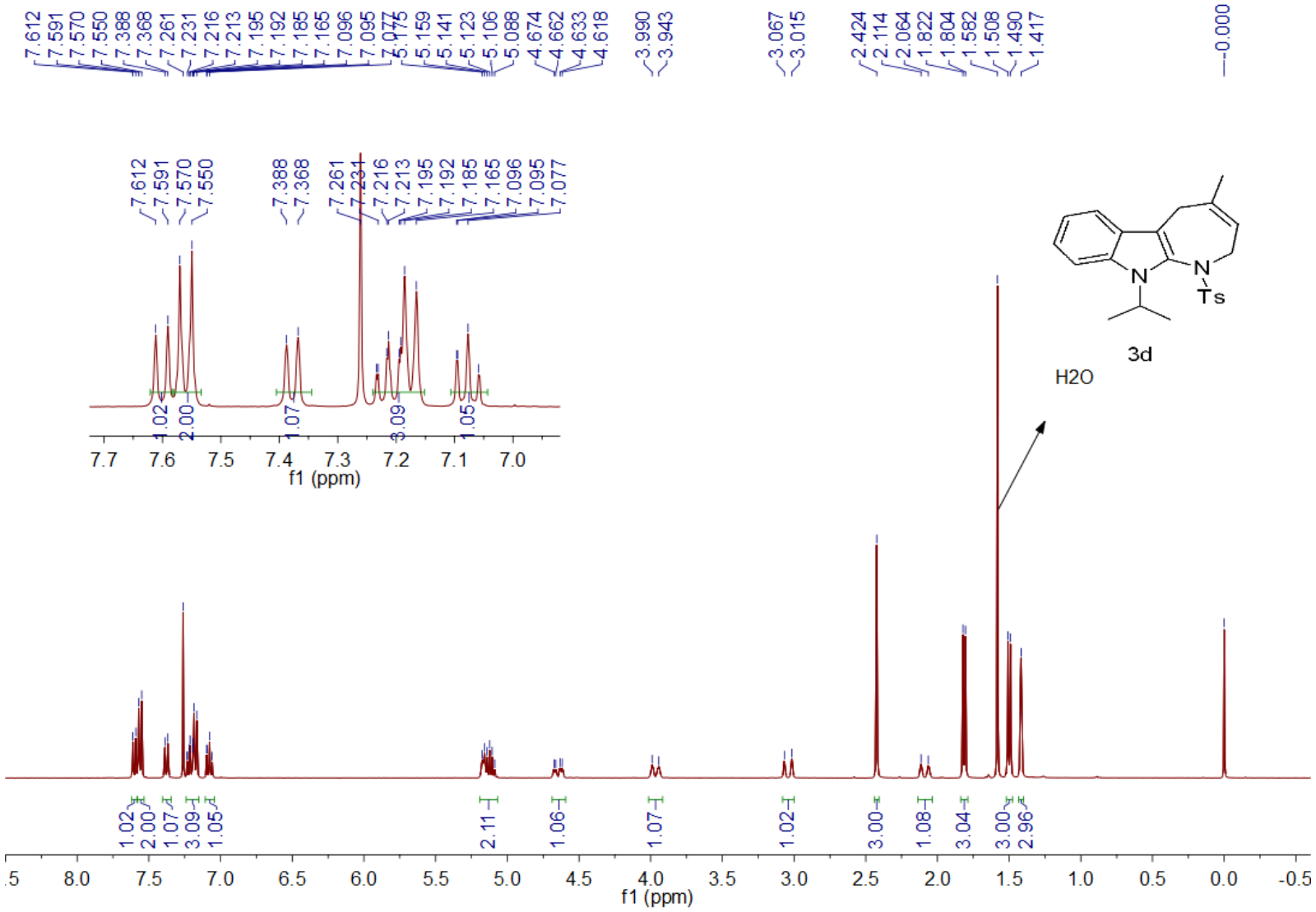
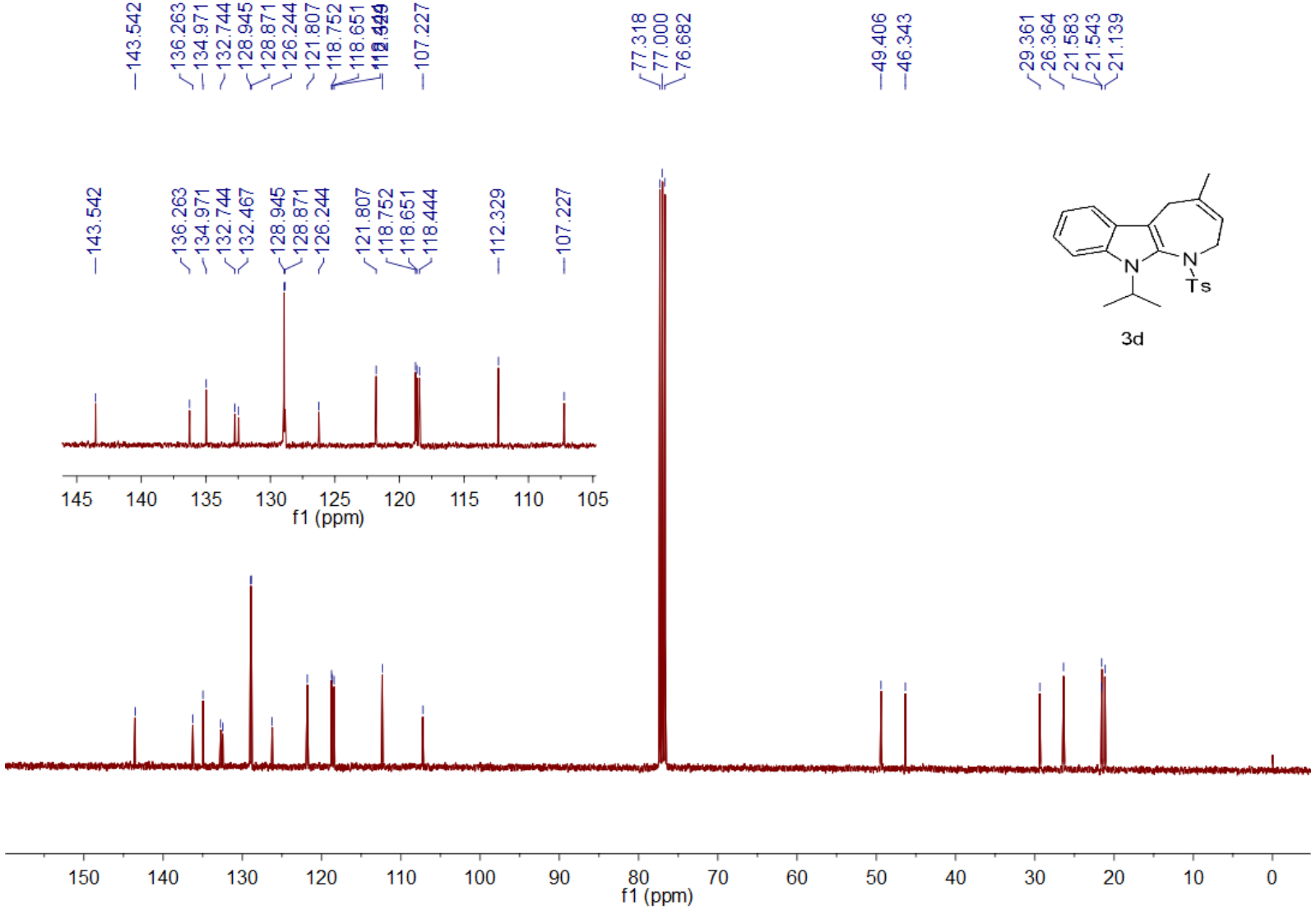
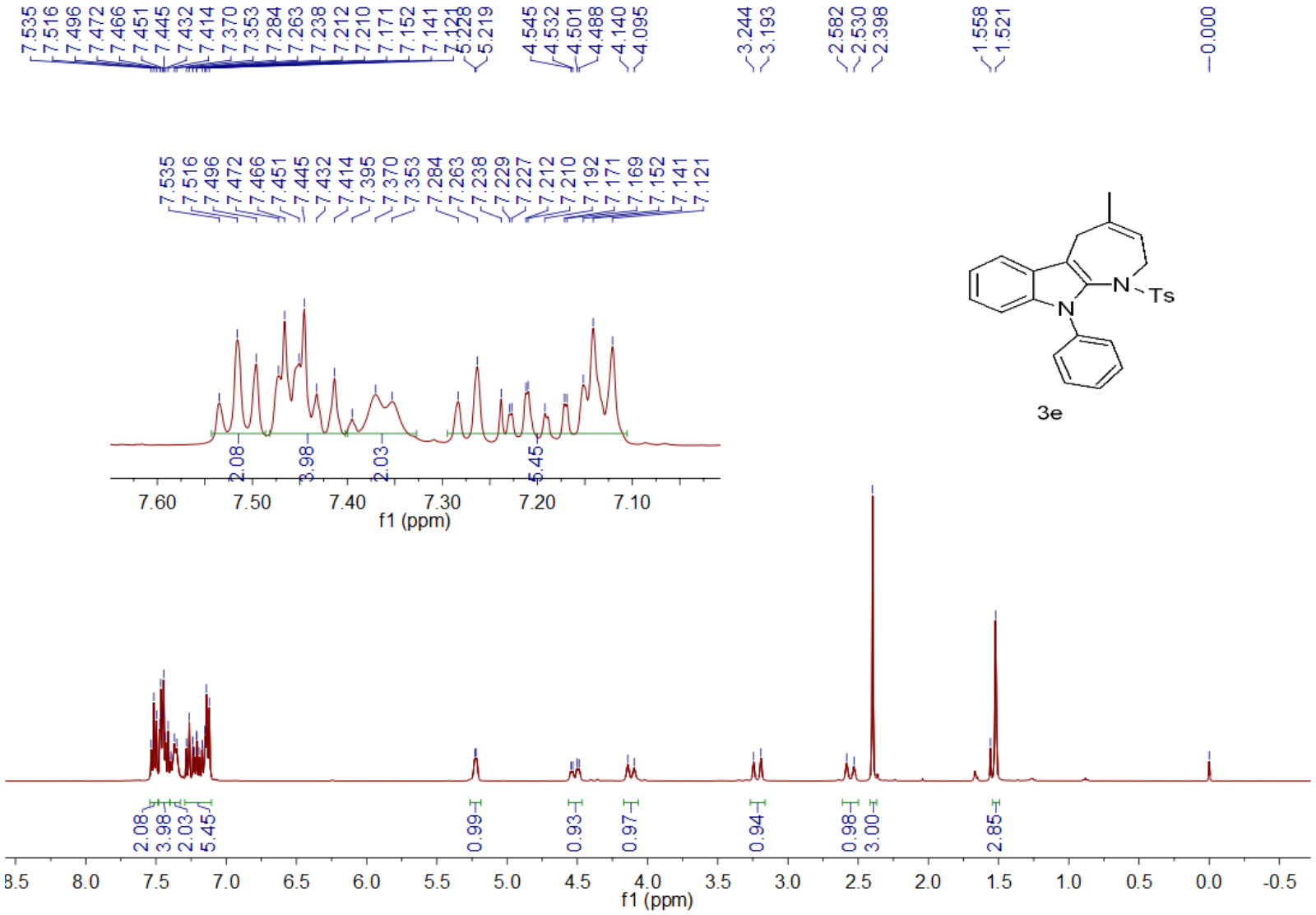
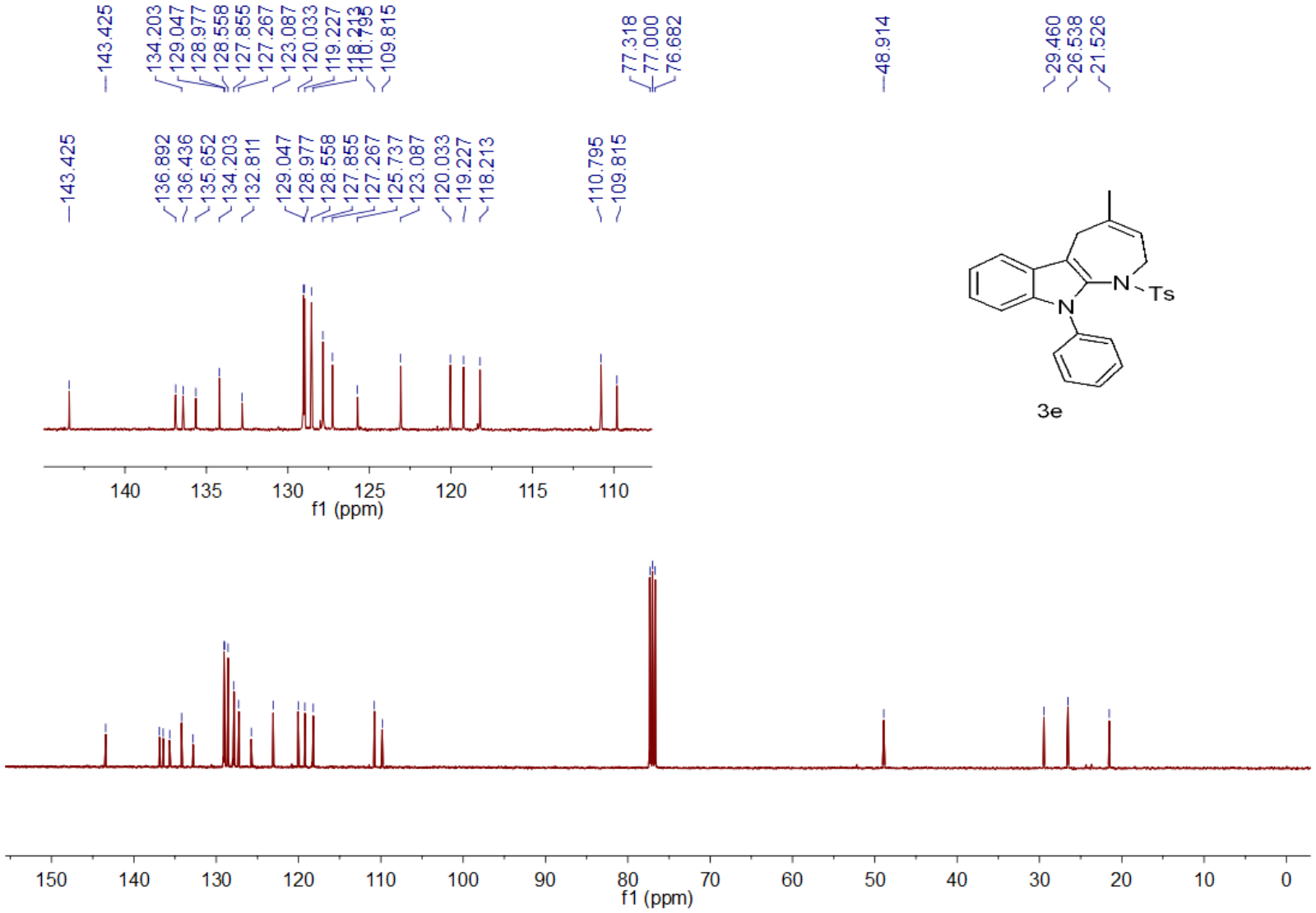
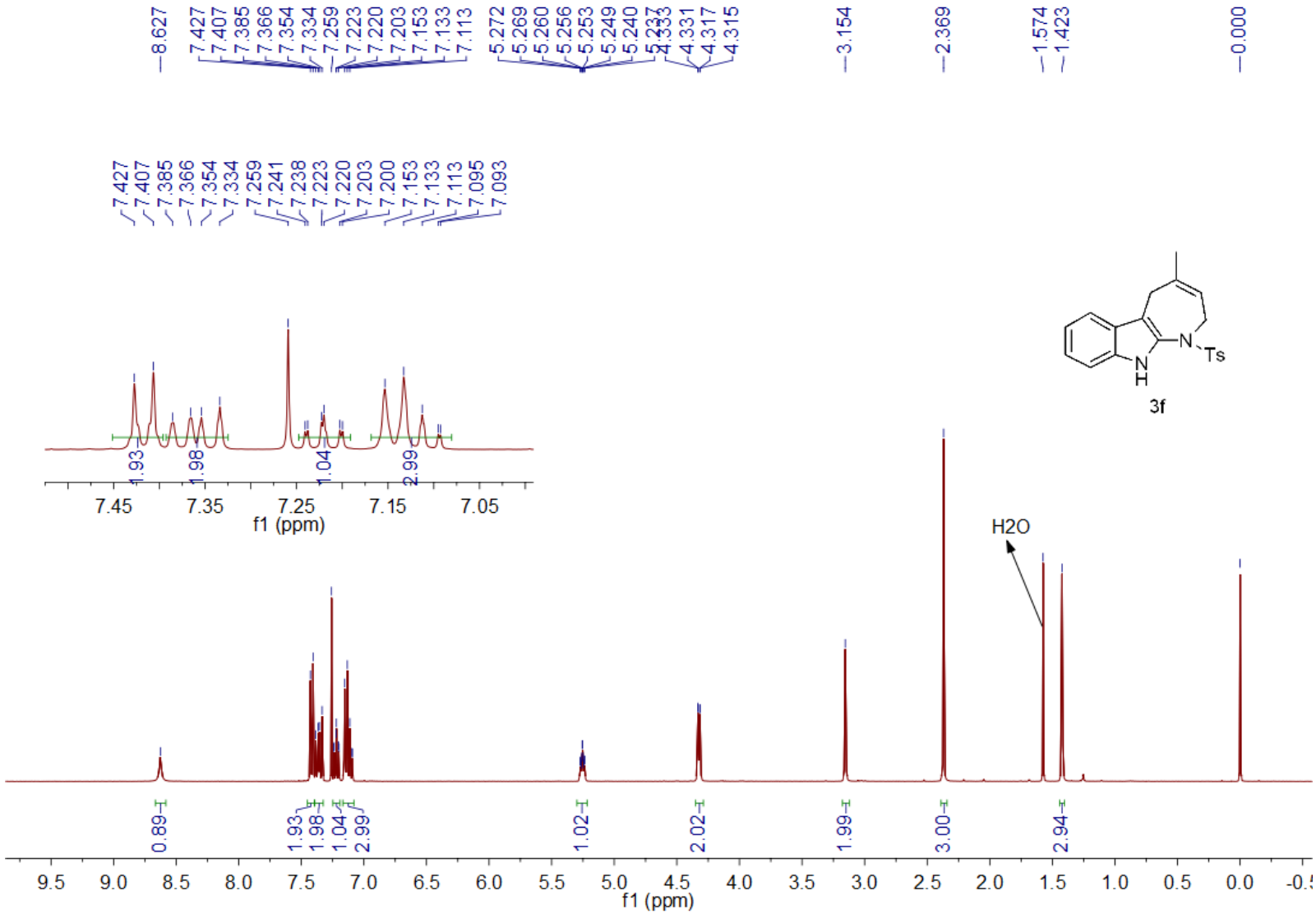
107.890
77.318
77.000
76.682
49.131
45.373
29.584
26.385
21.567
136.153
132.824
Ts
3c
f1 (ppm)

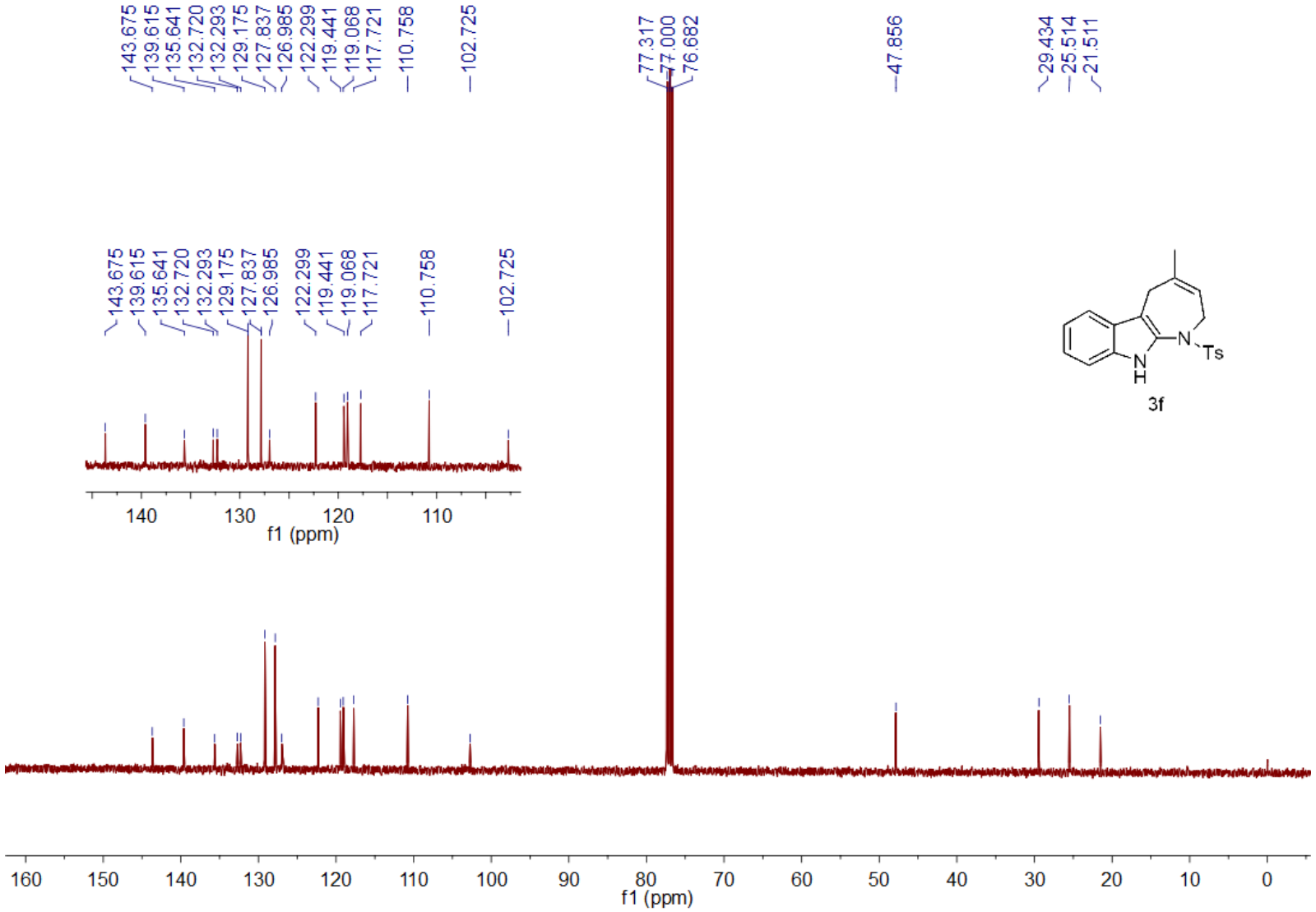

7.612
7.591
7.570
7.550
7.388
7.368
7.261
7.231
7.216
7.213
7.195
7.192
7.185
7.165
7.096
7.095
7.077
5.159
5.141
5.123
5.106
5.088
4.674
4.662
4.633
4.618
3.990
3.943
3.067
3.015
2.424
2.114
2.064
1.822
1.804
1.582
1.508
1.490
1.417
0.000
1.02
2.00
1.07
3.09
1.05
f1 (ppm)
N
N
Ts
3d
H2O
2.11
1.06
1.07
1.02
3.00
1.08
3.04
3.00
2.96

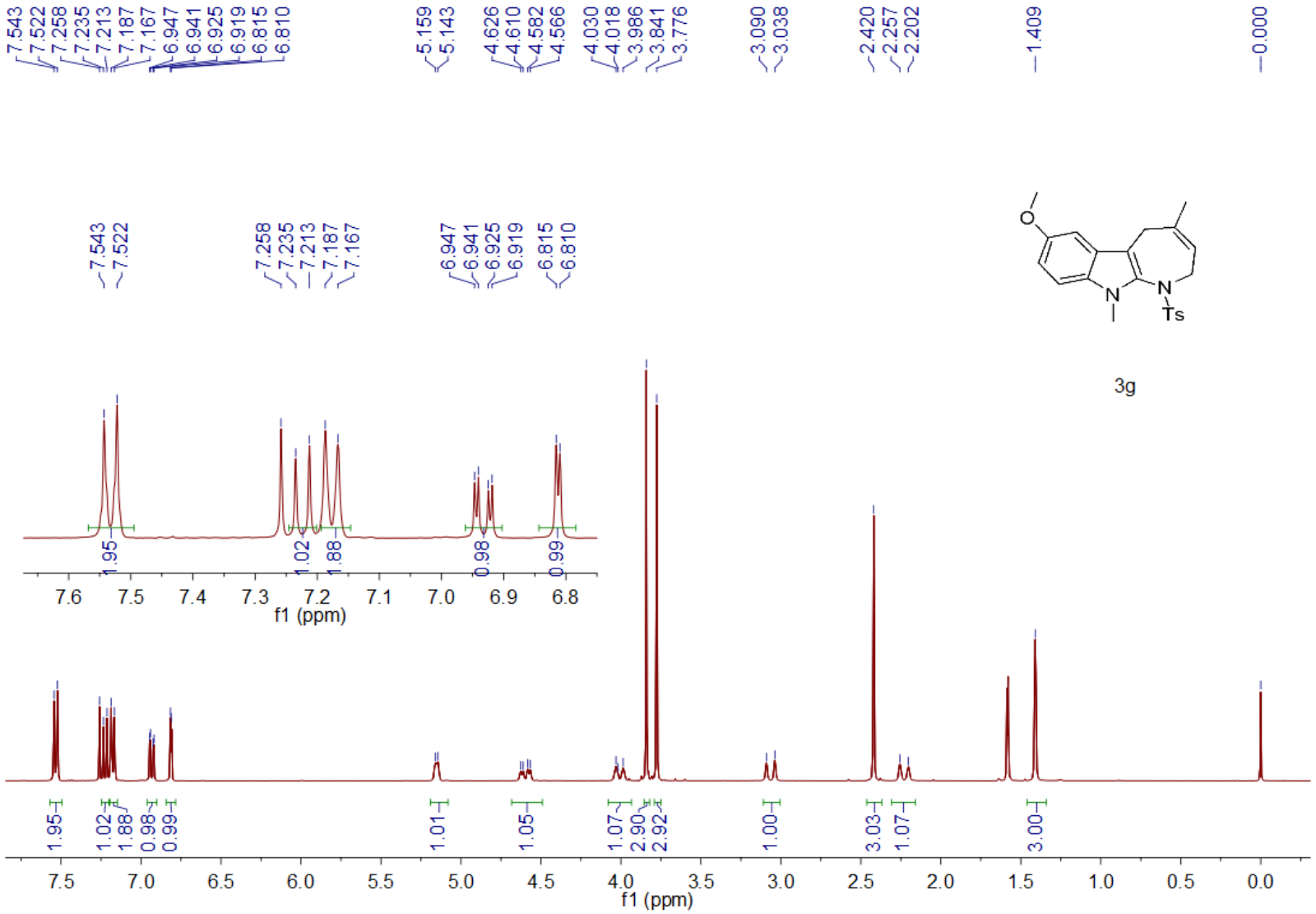

143.542
136.263
134.971
132.744
128.945
128.871
126.244
121.807
118.752
118.651
107.227
77.318
77.000
76.682
49.406
46.343
29.361
26.364
21.583
21.543
21.139
143.542
136.263
134.971
132.744
132.467
128.945
128.871
126.244
121.807
118.752
118.651
118.444
112.329
107.227
145 140 135 130 125 120 115 110 105
f1 (ppm)
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
N
N
Ts
3d

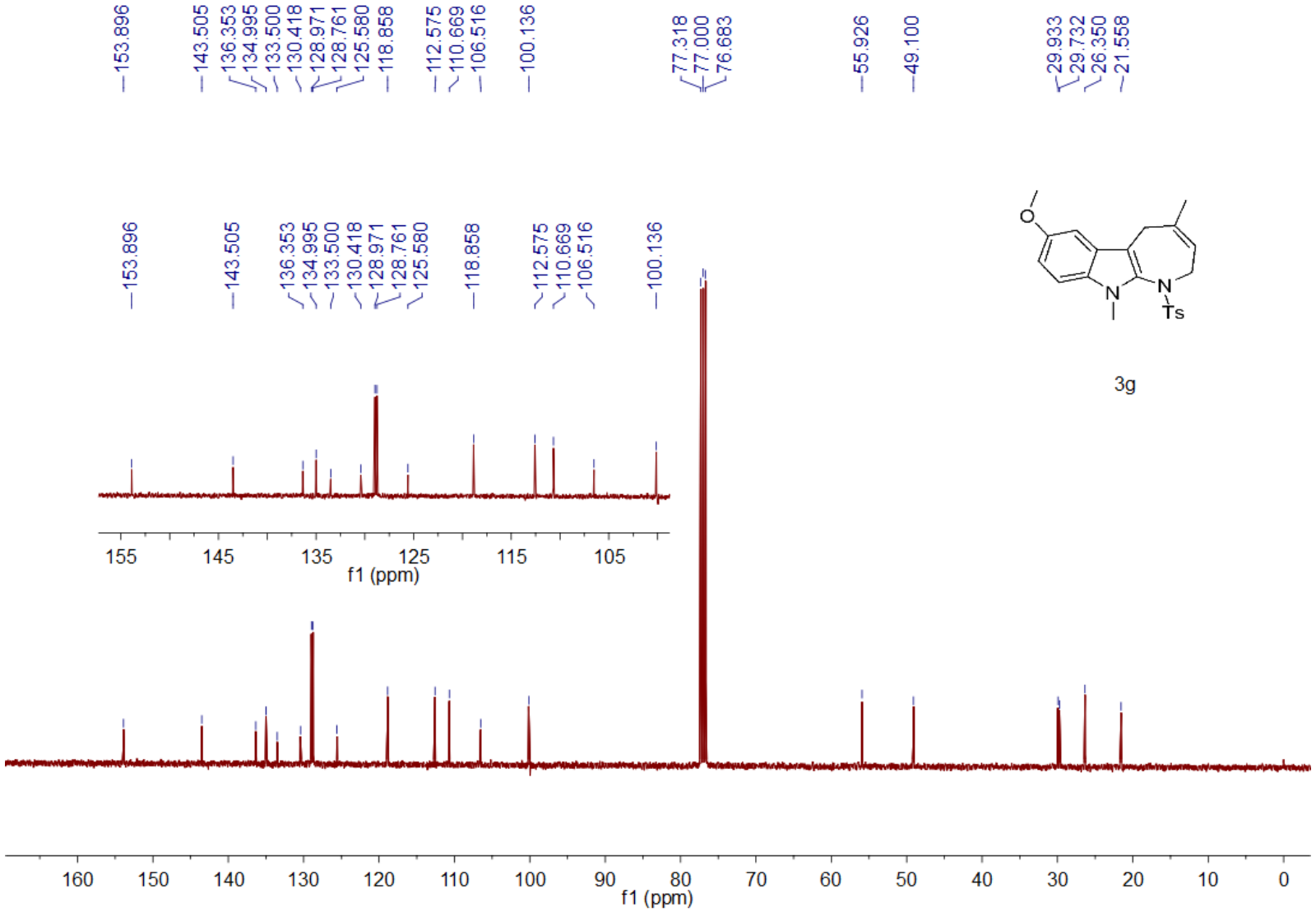

7.535
7.516
7.496
7.472
7.466
7.451
7.445
7.432
7.414
7.370
7.353
7.284
7.263
7.238
7.212
7.210
7.171
7.152
7.141
7.121
5.228
5.219
4.545
4.532
4.501
4.488
4.140
4.095
3.244
3.193
2.582
2.530
2.398
1.558
1.521
0.000
N
Ts
N
3e
2.08
3.98
2.03
5.45
7.60
7.50
7.40
7.30
7.20
7.10
f1 (ppm)
0.99
0.93
0.97
0.94
0.98
3.00
2.85
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)

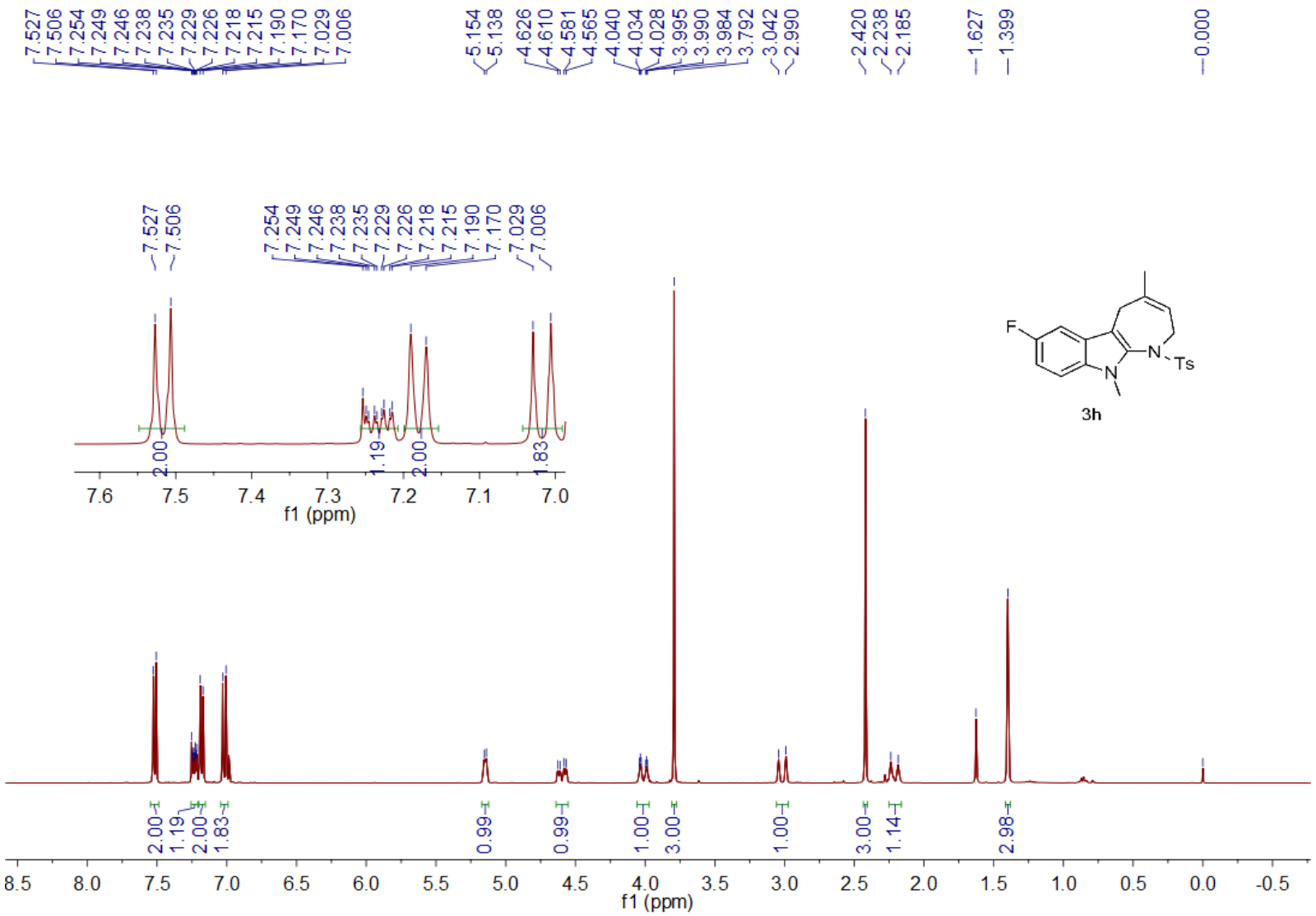

143.425
134.203
129.047
128.977
128.558
127.855
127.267
123.087
120.033
119.227
118.213
109.815
77.318
77.000
76.682
48.914
29.460
26.538
21.526
143.425
136.892
136.436
135.652
134.203
132.811
129.047
128.977
128.558
127.855
127.267
125.737
123.087
120.033
119.227
118.213
110.795
109.815
N
Ts
N
3e
140
135
130
125
120
115
110
f1 (ppm)
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

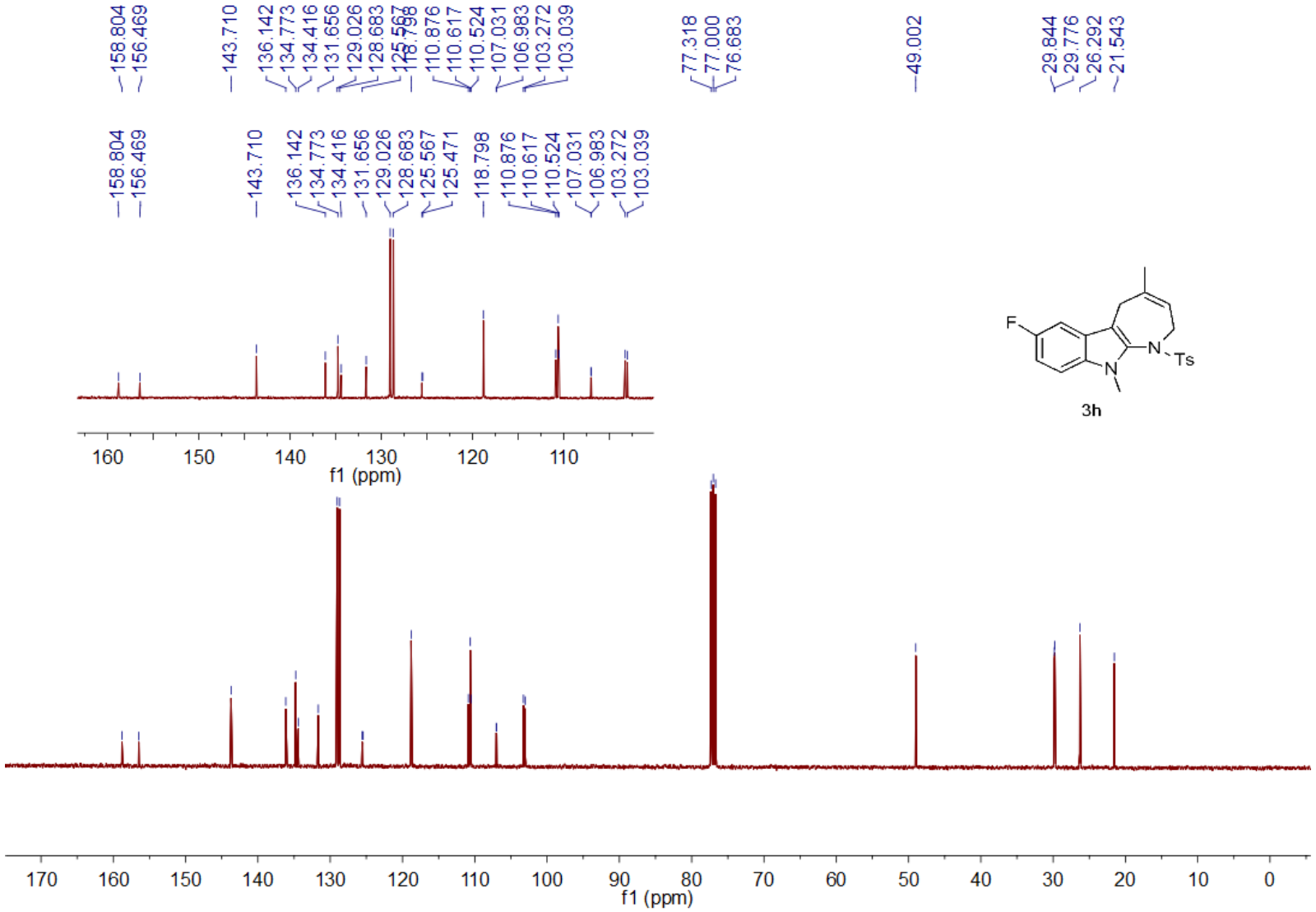

8.627
7.427
7.407
7.385
7.366
7.354
7.334
7.259
7.223
7.220
7.203
7.153
7.133
7.113
5.272
5.269
5.260
5.256
5.253
5.249
5.240
4.331
4.317
4.315
3.154
2.369
1.574
1.423
0.000
7.427
7.407
7.385
7.366
7.354
7.334
7.259
7.241
7.238
7.223
7.220
7.203
7.200
7.153
7.133
7.113
7.095
7.093
1.93
1.98
1.04
2.99
7.45
7.35
7.25
7.15
7.05
f1 (ppm)
N
Ts
H
3f
H2O
0.89
1.93
1.98
1.04
2.99
1.02
2.02
1.99
3.00
2.94
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)

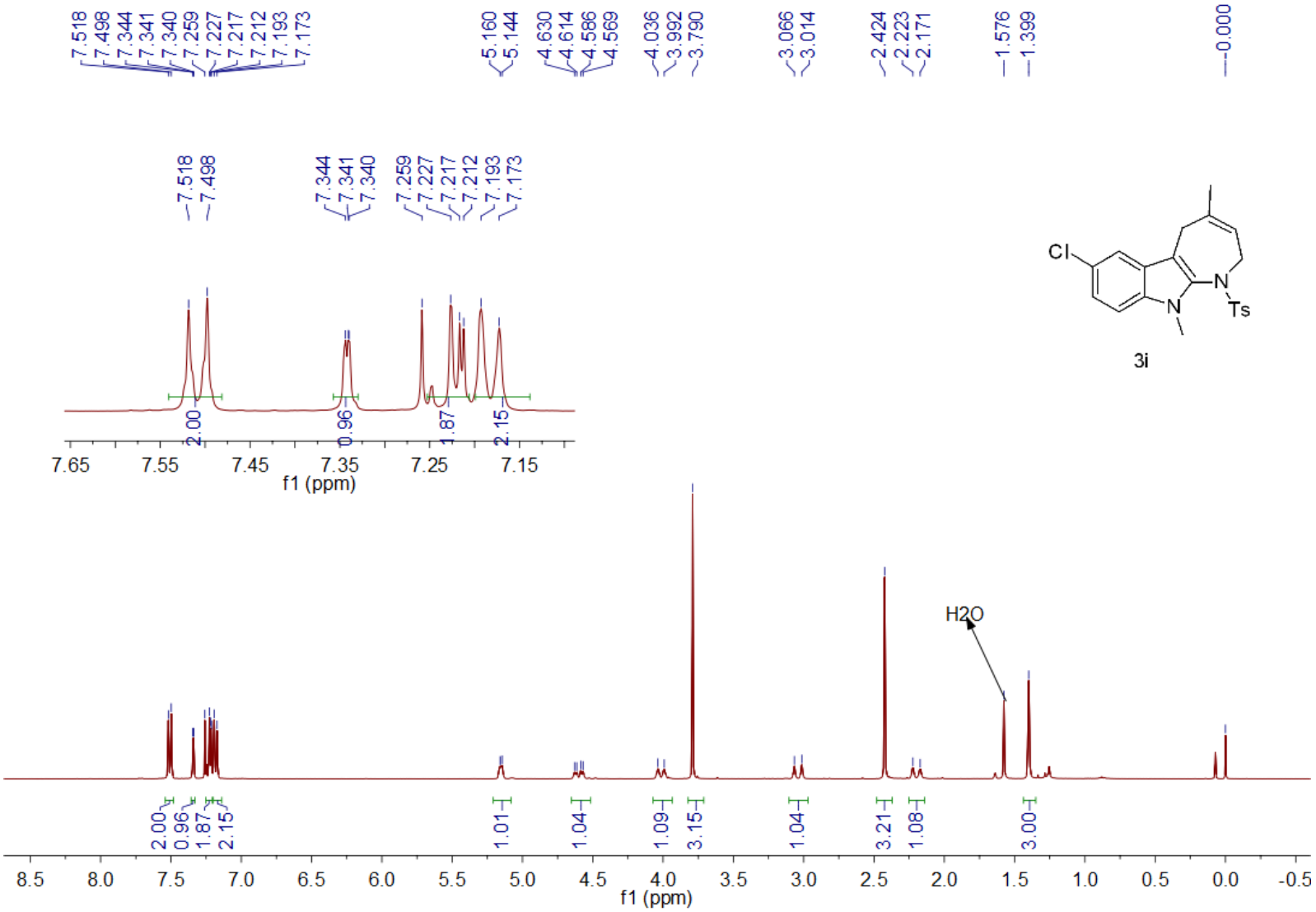

143.675
139.615
135.641
132.720
132.293
129.175
127.837
126.985
122.299
119.441
119.068
117.721
110.758
102.725
77.317
77.000
76.682
47.856
29.434
25.514
21.511
f1 (ppm)
Ts
N
H
3f

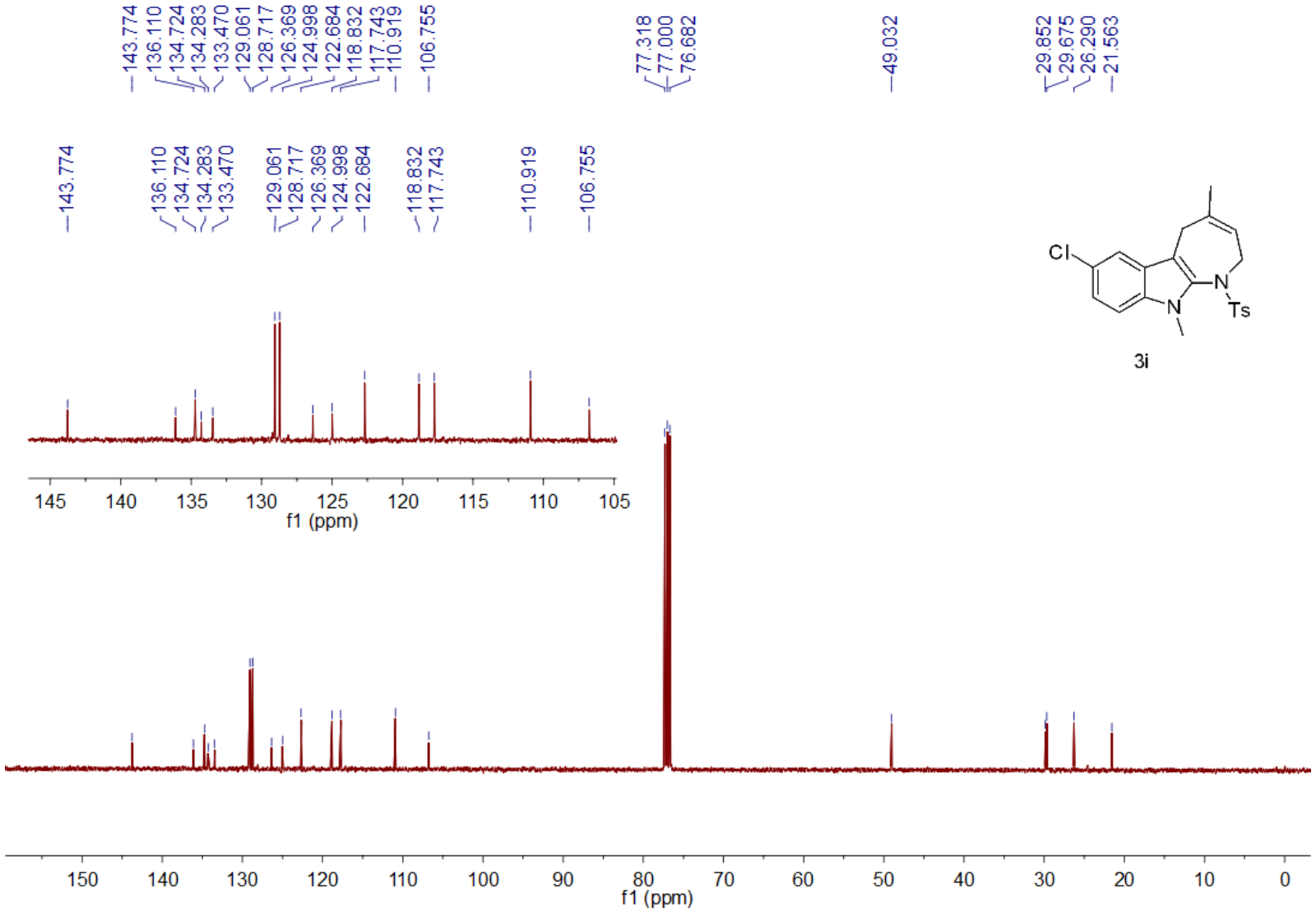

7.543
7.522
7.258
7.235
7.213
7.187
7.167
6.947
6.941
6.925
6.919
6.815
6.810
5.159
5.143
4.626
4.610
4.582
4.566
4.030
4.018
3.986
3.841
3.776
3.090
3.038
2.420
2.257
2.202
1.409
0.000
O
N
N
Ts
3g
1.95
1.02
1.88
0.98
0.99
1.01
1.05
1.07
2.90
2.92
1.00
3.03
1.07
3.00
f1 (ppm)

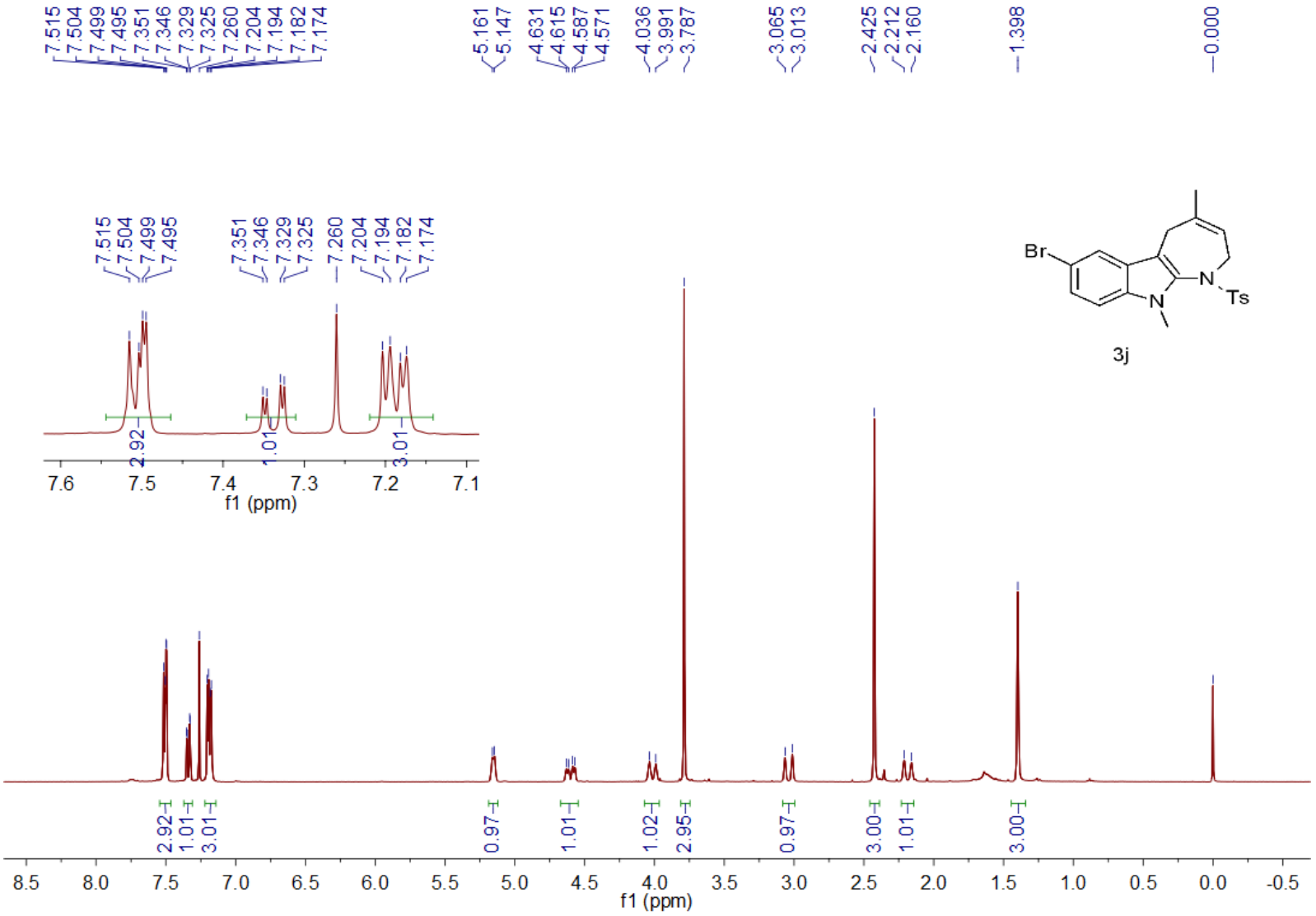

153.896
143.505
136.353
134.995
133.500
130.418
128.971
128.761
125.580
118.858
112.575
110.669
106.516
100.136
77.318
77.000
76.683
55.926
49.100
29.933
29.732
26.350
21.558
O
N
N
Ts
3g
155
145
135
125
115
105
f1 (ppm)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

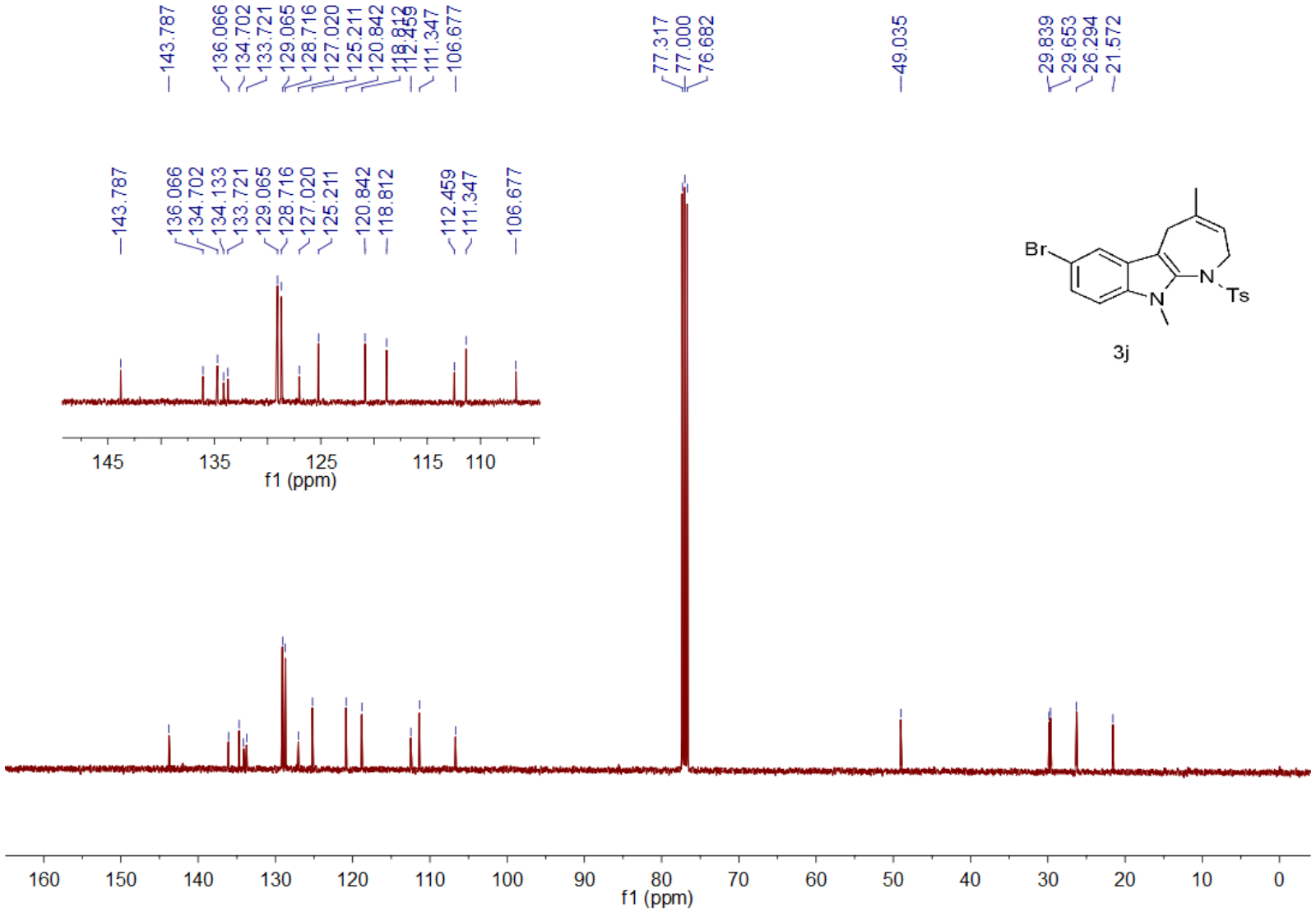

7.527
7.506
7.254
7.249
7.246
7.238
7.235
7.229
7.226
7.218
7.215
7.190
7.170
7.029
7.006
5.154
5.138
4.626
4.610
4.581
4.565
4.040
4.034
4.028
3.995
3.990
3.984
3.792
3.042
2.990
2.420
2.238
2.185
1.627
1.399
0.000
2.00
1.19
2.00
1.83
f1 (ppm)
F
N
Ts
N
3h
0.99
0.99
1.00
3.00
1.00
3.00
1.14
2.98
f1 (ppm)

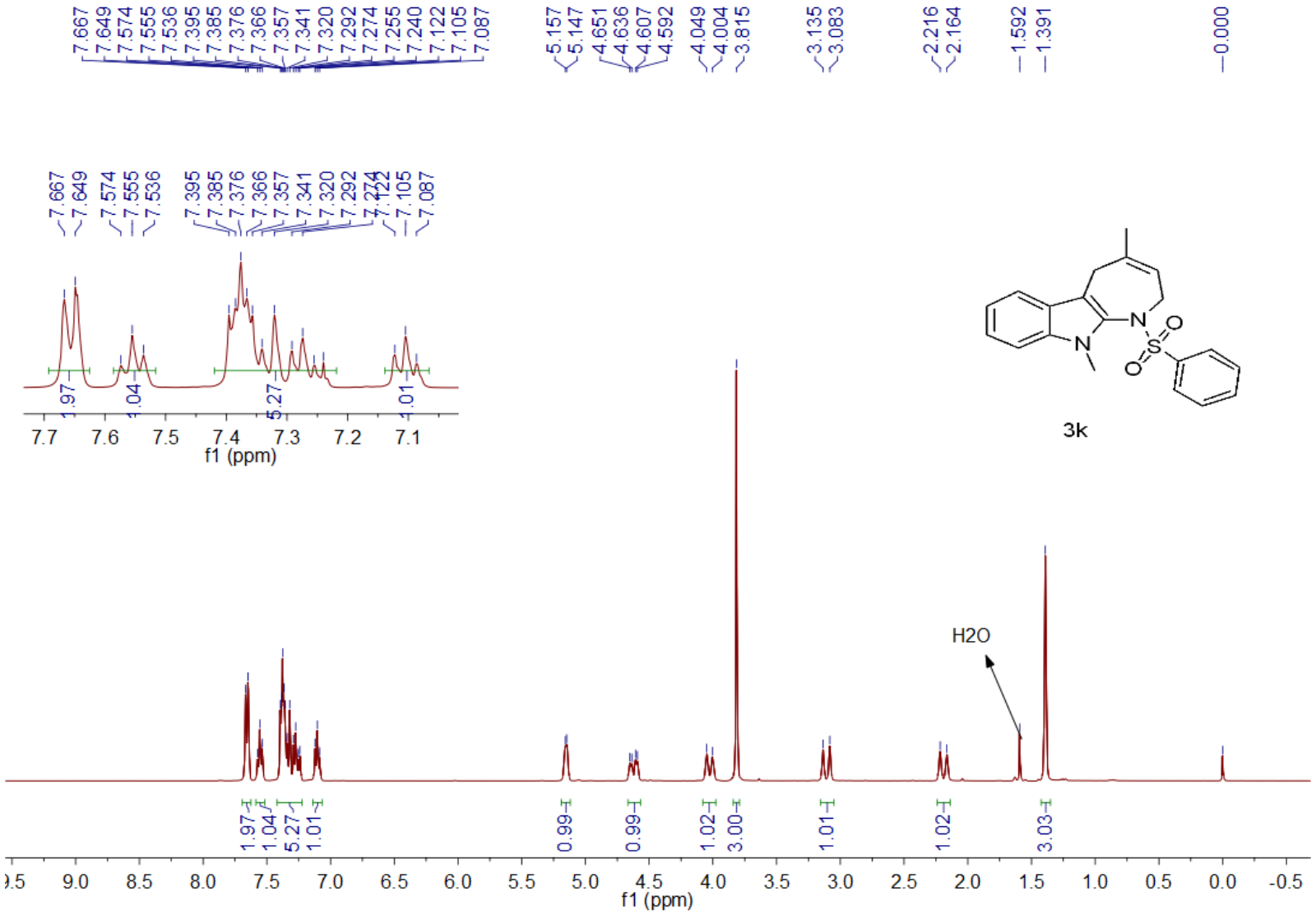

158.804
156.469
143.710
136.142
134.773
134.416
131.656
129.026
128.683
125.567
118.798
110.876
110.617
110.524
107.031
106.983
103.272
103.039
77.318
77.000
76.683
49.002
29.844
29.776
26.292
21.543
F
N
Ts
N
3h
f1 (ppm)
f1 (ppm)


7.518
7.498
7.344
7.341
7.340
7.259
7.227
7.217
7.212
7.193
7.173
5.160
5.144
4.630
4.614
4.586
4.569
4.036
3.992
3.790
3.066
3.014
2.424
2.223
2.171
1.576
1.399
0.000
2.00
0.96
1.87
2.15
7.65
7.55
7.45
7.35
7.25
7.15
f1 (ppm)
Cl
N
N
Ts
3i
H2O
1.01
1.04
1.09
3.15
1.04
3.21
1.08
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


143.774
136.110
134.724
134.283
133.470
129.061
128.717
126.369
124.998
122.684
118.832
117.743
110.919
106.755
77.318
77.000
76.682
49.032
29.852
29.675
26.290
21.563
Cl
N
N
Ts
3i
f1 (ppm)
f1 (ppm)


7.515
7.504
7.499
7.495
7.351
7.346
7.329
7.325
7.260
7.204
7.194
7.182
7.174
5.161
5.147
4.631
4.615
4.587
4.571
4.036
3.991
3.787
3.065
3.013
2.425
2.212
2.160
1.398
0.000
Br
N
N
Ts
3j
2.92
1.01
3.01
f1 (ppm)
0.97
1.01
1.02
2.95
0.97
3.00
1.01
3.00
f1 (ppm)


143.787
136.066
134.702
134.133
133.721
129.065
128.716
127.020
125.211
120.842
118.812
112.459
111.347
106.677
77.317
77.000
76.682
49.035
29.839
29.653
26.294
21.572
145
135
125
115
110
f1 (ppm)
Br
N
N
Ts
3j
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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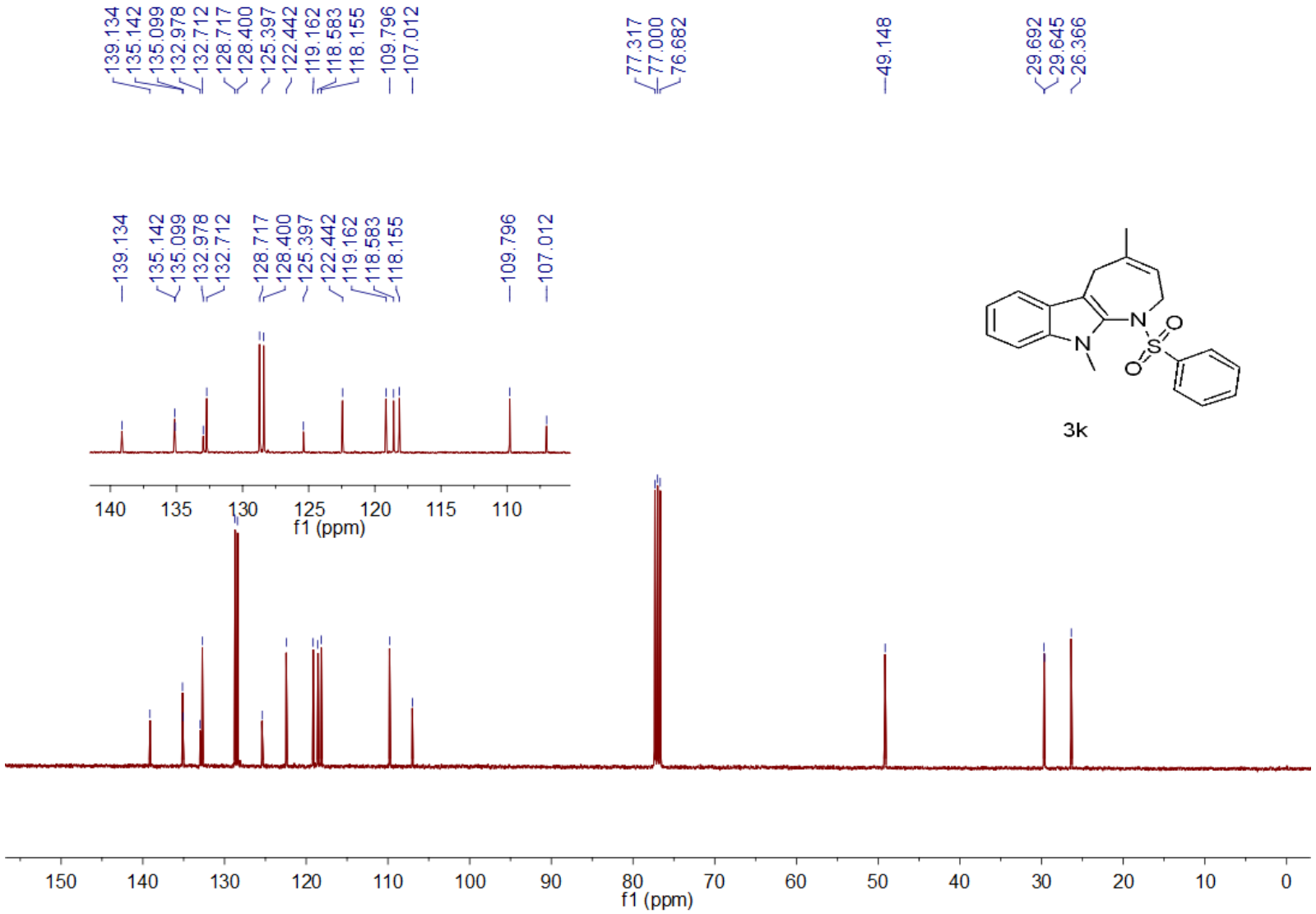
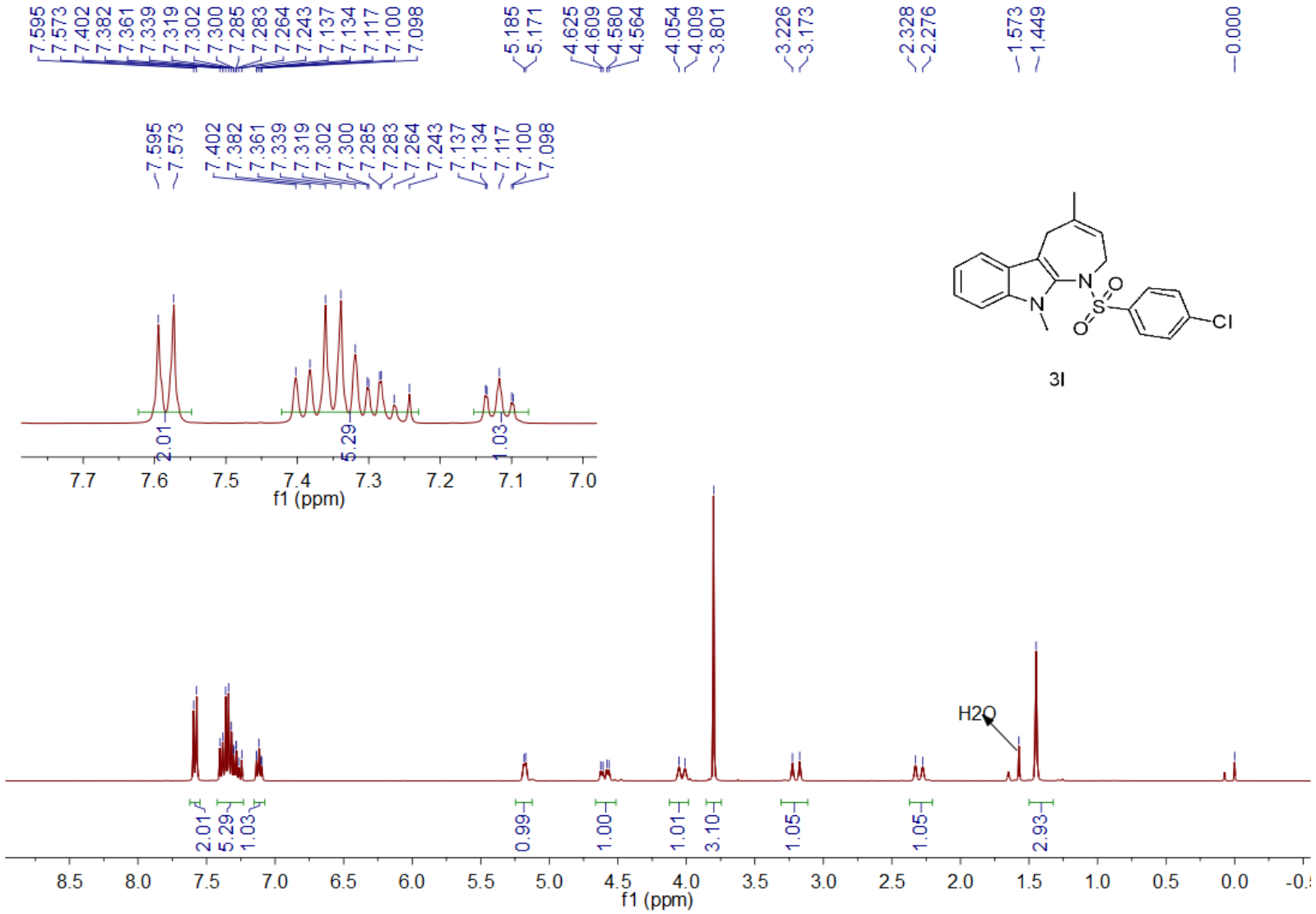
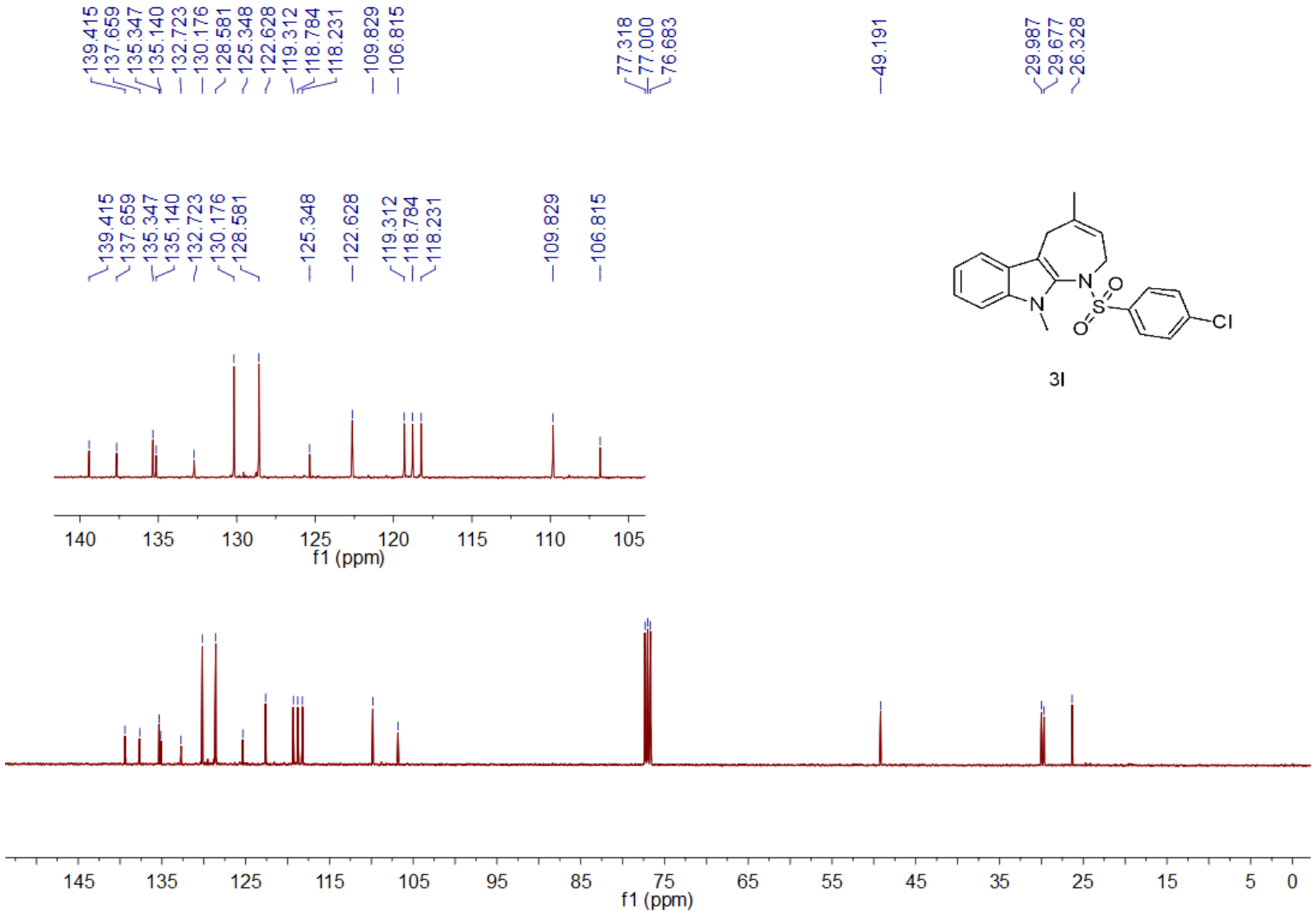
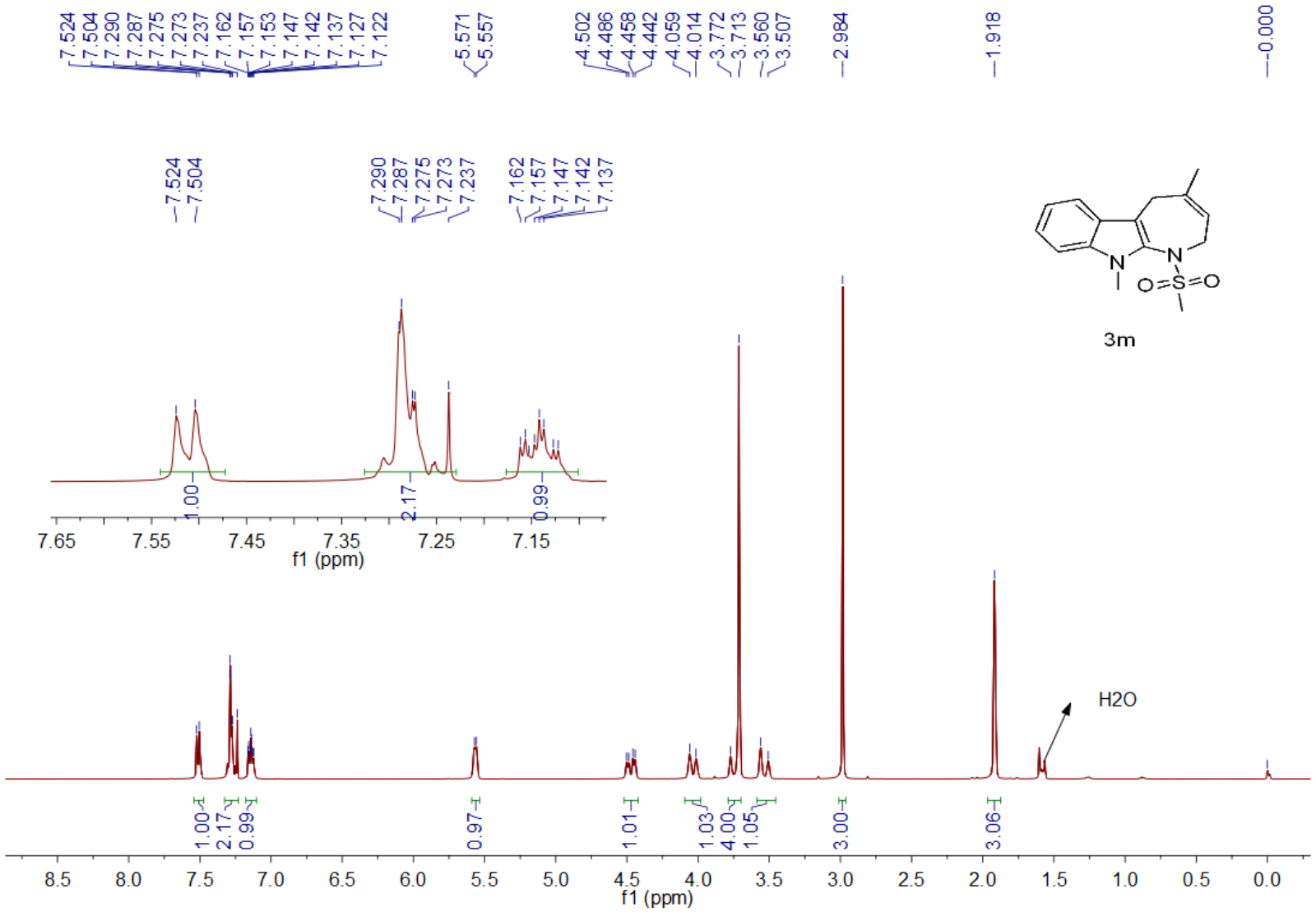
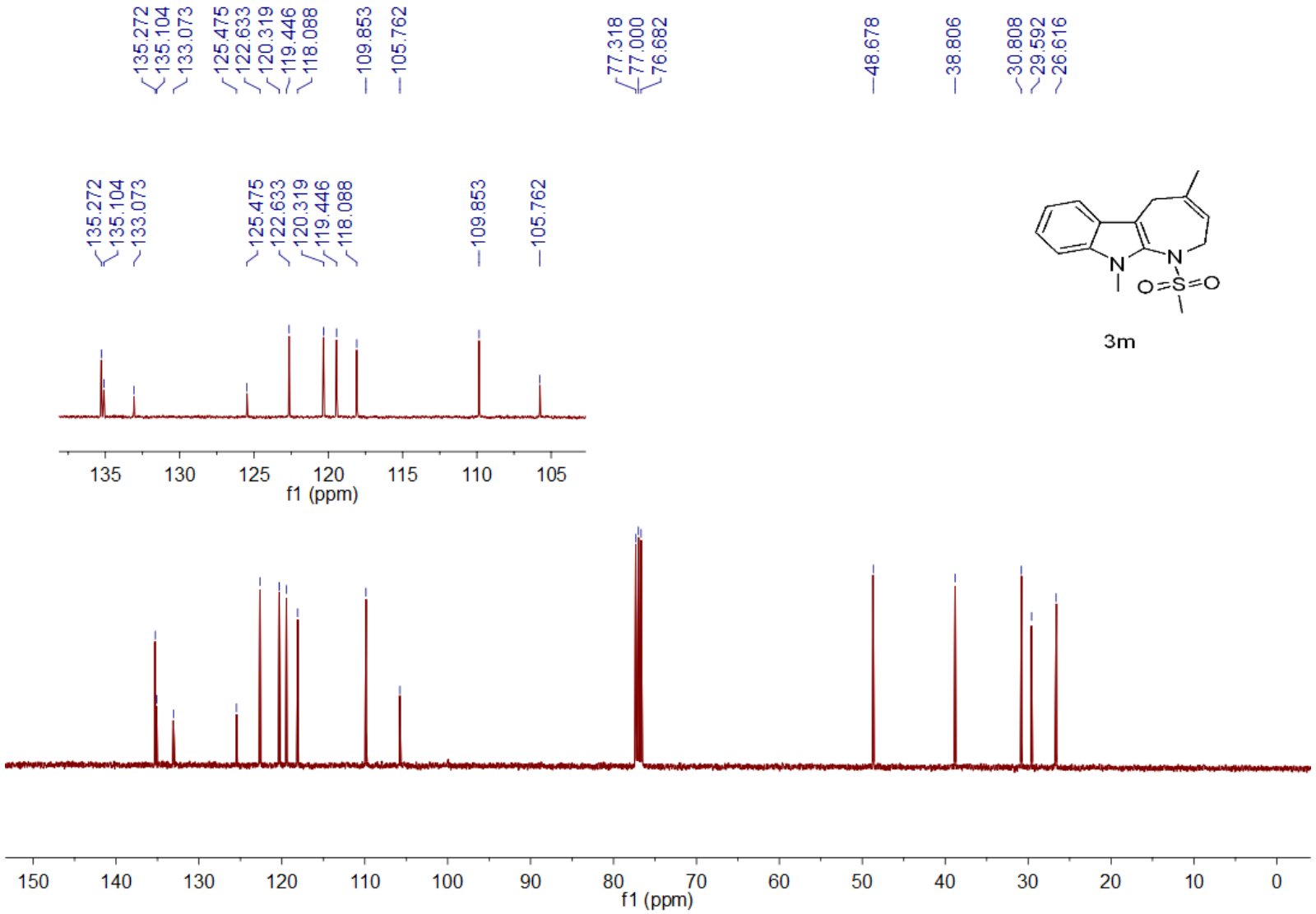
7.574
7.555
7.536
7.395
7.385
7.376
7.366
7.357
7.341
7.320
7.292
7.274
7.255
7.240
7.122
7.105
7.087
5.157
5.147
4.651
4.636
4.607
4.592
4.049
4.004
3.815
3.135
3.083
2.216
2.164
1.592
1.391
0.000
1.97
1.04
5.27
1.01
f1 (ppm)
3k
H2O
0.99
0.99
1.02
3.00
1.01
1.02
3.03
f1 (ppm)

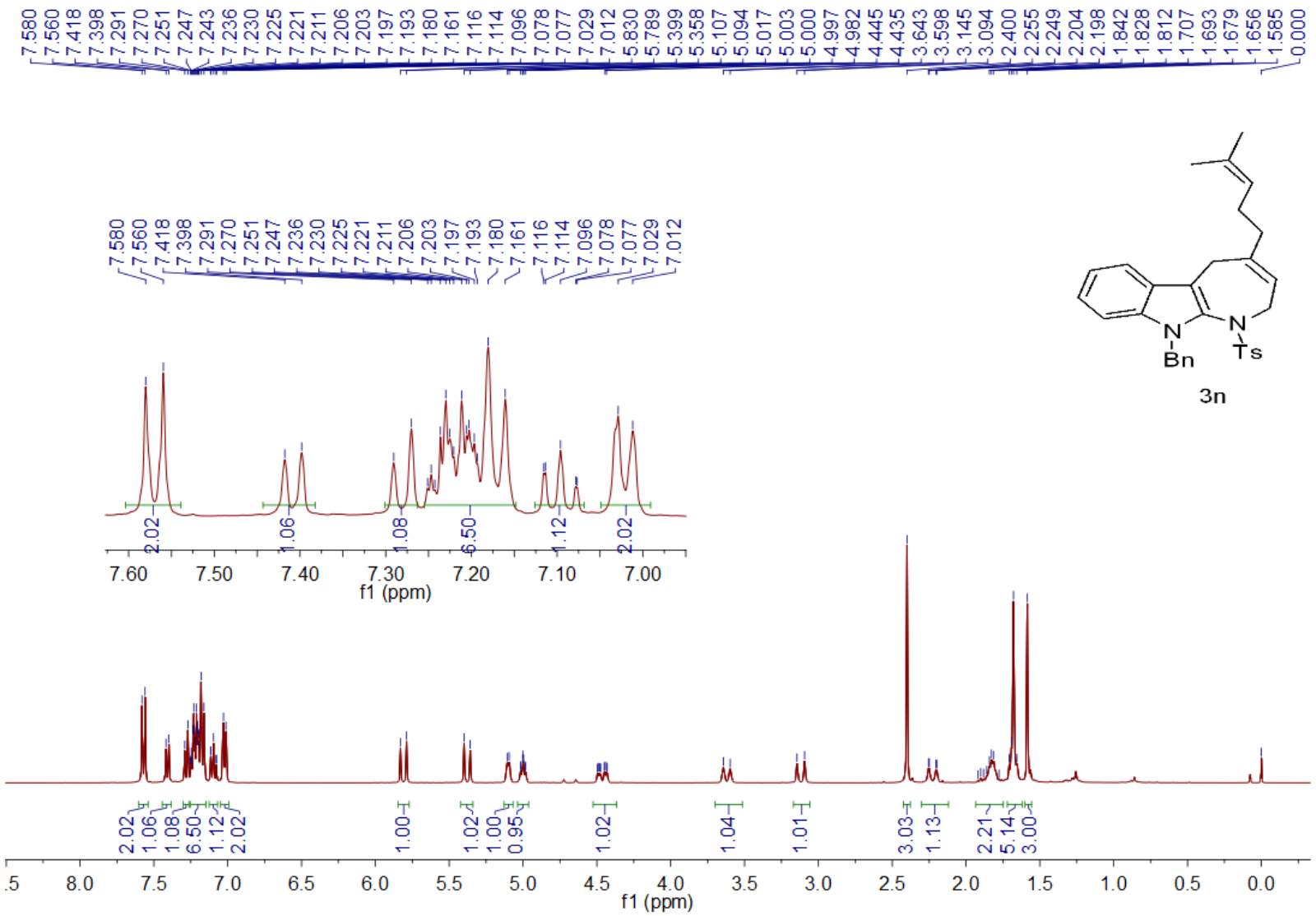

139.134
135.142
135.099
132.978
132.712
128.717
128.400
125.397
122.442
119.162
118.583
118.155
109.796
107.012
77.317
77.000
76.682
49.148
29.692
29.645
26.366
f1 (ppm)
3k

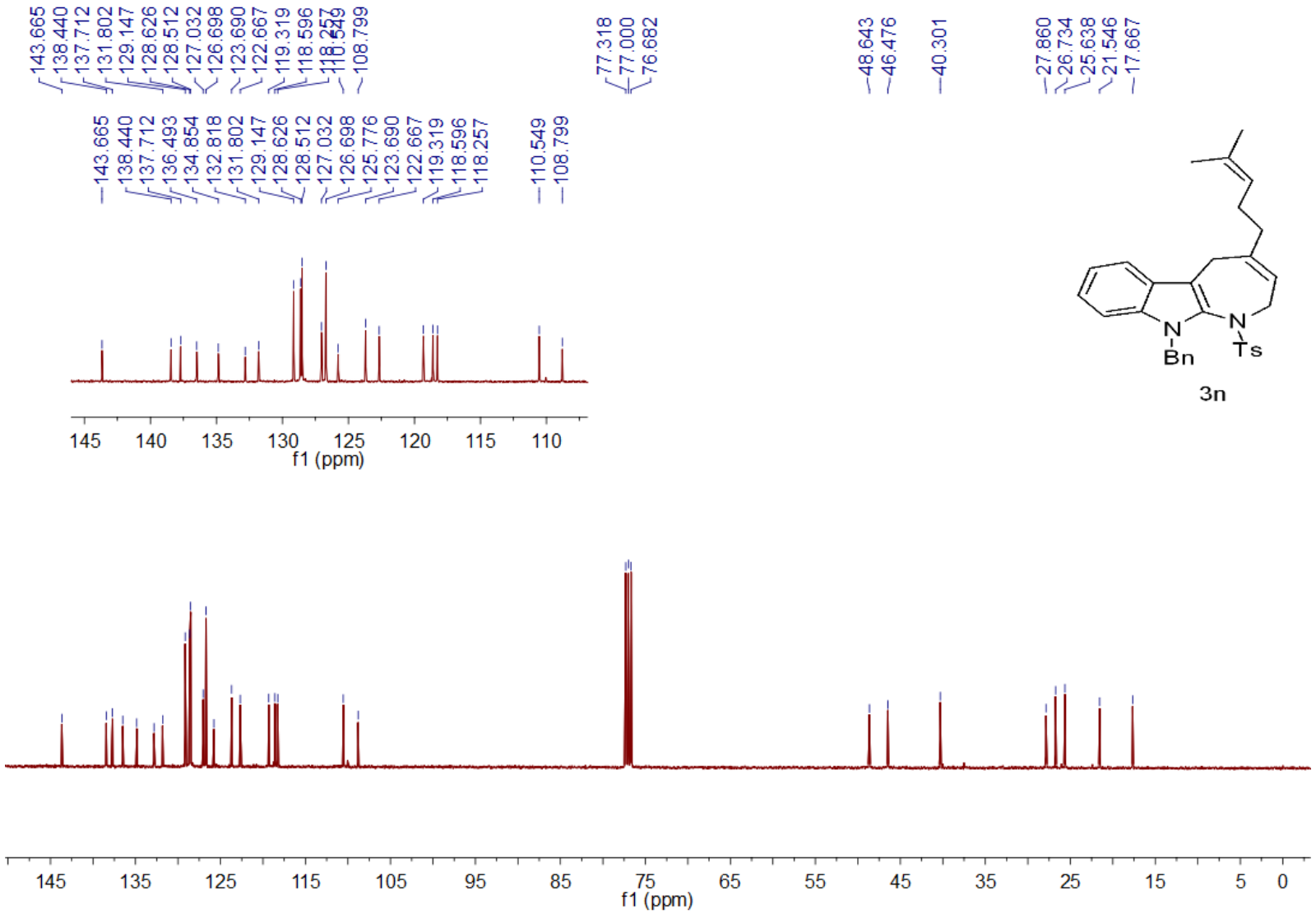

7.595
7.573
7.402
7.382
7.361
7.339
7.319
7.302
7.300
7.285
7.283
7.264
7.243
7.137
7.134
7.117
7.100
7.098
5.185
5.171
4.625
4.609
4.580
4.564
4.054
4.009
3.801
3.226
3.173
2.328
2.276
1.573
1.449
0.000
2.01
5.29
1.03
f1 (ppm)
H2O
0.99
1.00
1.01
3.10
1.05
1.05
2.93
f1 (ppm)
N
O
S
O
Cl
3l

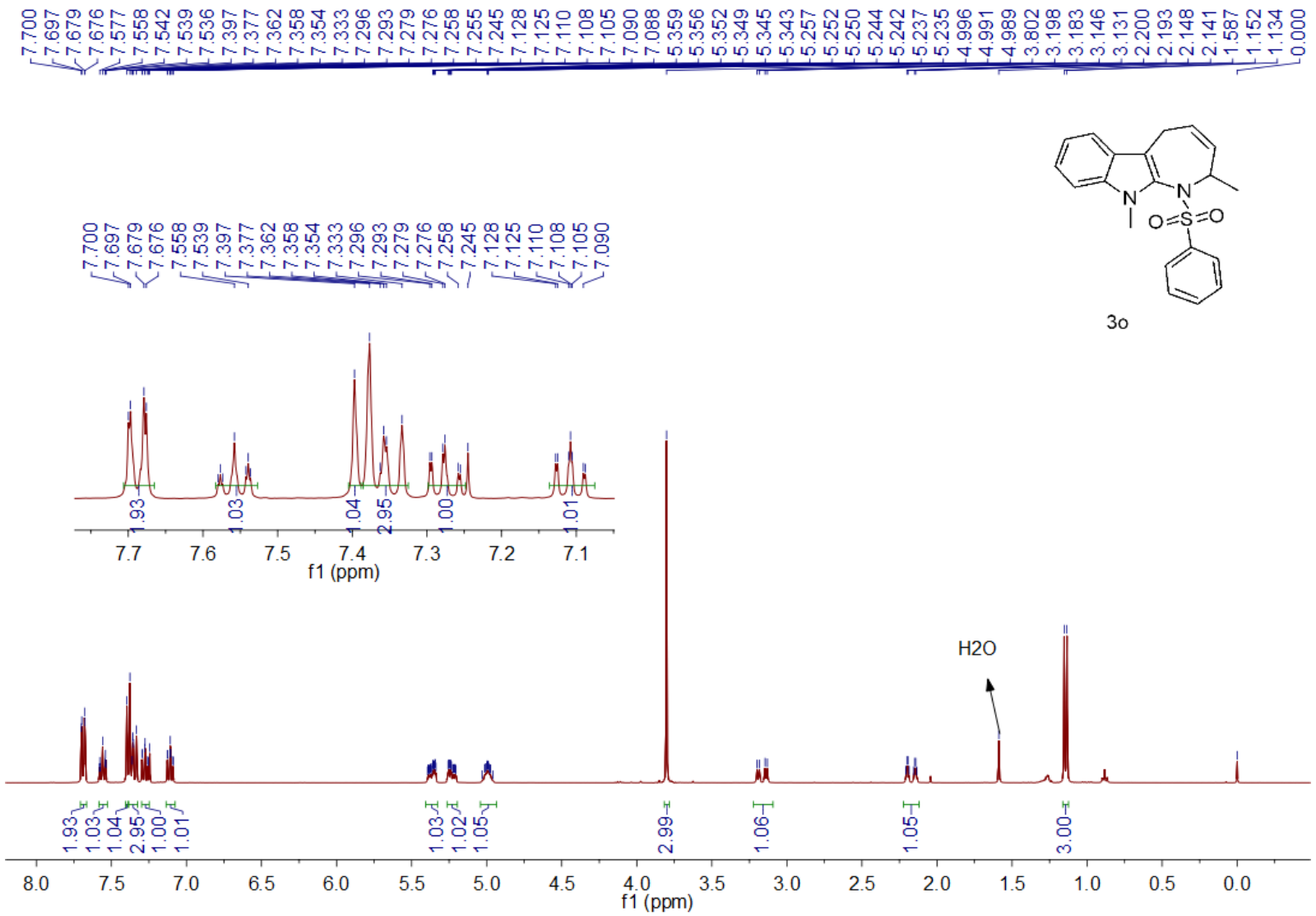

139.415
137.659
135.347
135.140
132.723
130.176
128.581
125.348
122.628
119.312
118.784
118.231
109.829
106.815
77.318
77.000
76.683
49.191
29.987
29.677
26.328
f1 (ppm)
f1 (ppm)
Cl
3l


7.524
7.504
7.290
7.287
7.275
7.273
7.237
7.162
7.157
7.153
7.147
7.142
7.137
7.127
7.122
5.571
5.557
4.502
4.486
4.458
4.442
4.059
4.014
3.772
3.713
3.560
3.507
2.984
1.918
0.000
1.00
2.17
0.99
0.97
1.01
1.03
4.00
1.05
3.00
3.06
H2O
f1 (ppm)
3m


135.272
135.104
133.073
125.475
122.633
120.319
119.446
118.088
109.853
105.762
77.318
77.000
76.682
48.678
38.806
30.808
29.592
26.616
f1 (ppm)
3m


7.580 7.560 7.418 7.398 7.291 7.270 7.251 7.247 7.243 7.236 7.230 7.225 7.221 7.211 7.206 7.203 7.197 7.193 7.180 7.161 7.116 7.114 7.096 7.078 7.077 7.029 7.012 5.830 5.789 5.399 5.358 5.107 5.094 5.017 5.003 5.000 4.997 4.982 4.445 4.435 3.643 3.598 3.145 3.094 2.400 2.255 2.249 2.204 2.198 1.842 1.828 1.812 1.707 1.693 1.679 1.656 1.585 0.000
N
Bn
N
Ts
3n
2.02 1.06 1.08 6.50 1.12 2.02
7.60 7.50 7.40 7.30 7.20 7.10 7.00
f1 (ppm)
2.02 1.06 1.08 6.50 1.12 2.02 1.00 1.02 1.00 0.95 1.02 1.04 1.01 3.03 1.13 2.21 5.14 3.00
.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)


143.665
138.440
137.712
131.802
129.147
128.626
128.512
127.032
126.698
123.690
122.667
119.319
118.596
118.257
110.549
108.799
77.318
77.000
76.682
48.643
46.476
40.301
27.860
26.734
25.638
21.546
17.667
143.665
138.440
137.712
136.493
134.854
132.818
131.802
129.147
128.626
128.512
127.032
126.698
125.776
123.690
122.667
119.319
118.596
118.257
110.549
108.799
145 140 135 130 125 120 115 110
f1 (ppm)
N
Bn
N
Ts
3n
145 135 125 115 105 95 85 75 65 55 45 35 25 15 5 0
f1 (ppm)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7.539
7.536
7.397
7.377
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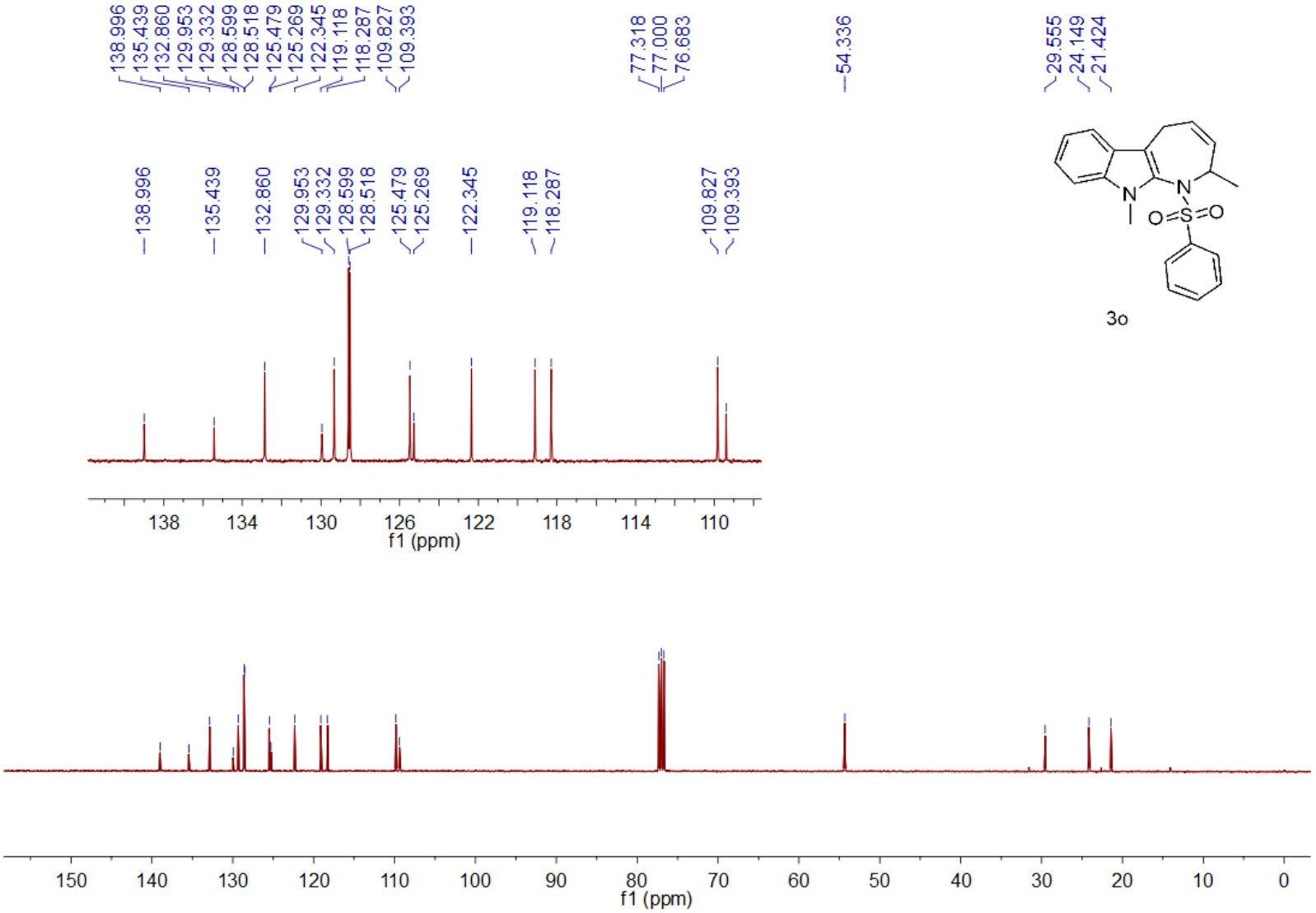
7.358
7.354
7.333
7.296
7.293
7.279
7.276
7.258
7.255
7.245
7.128
7.125
7.110
7.108
7.105
7.090
7.088
5.359
5.356
5.352
5.349
5.345
5.343
5.257
5.252
5.250
5.244
5.242
5.237
5.235
4.996
4.991
4.989
3.802
3.198
3.183
3.146
3.131
2.200
2.193
2.148
2.141
1.587
1.152
1.134
0.000
3o
1.93
1.03
1.04
2.95
1.00
1.01
f1 (ppm)
H2O
1.03
1.02
1.05
2.99
1.06
1.05
3.00
f1 (ppm)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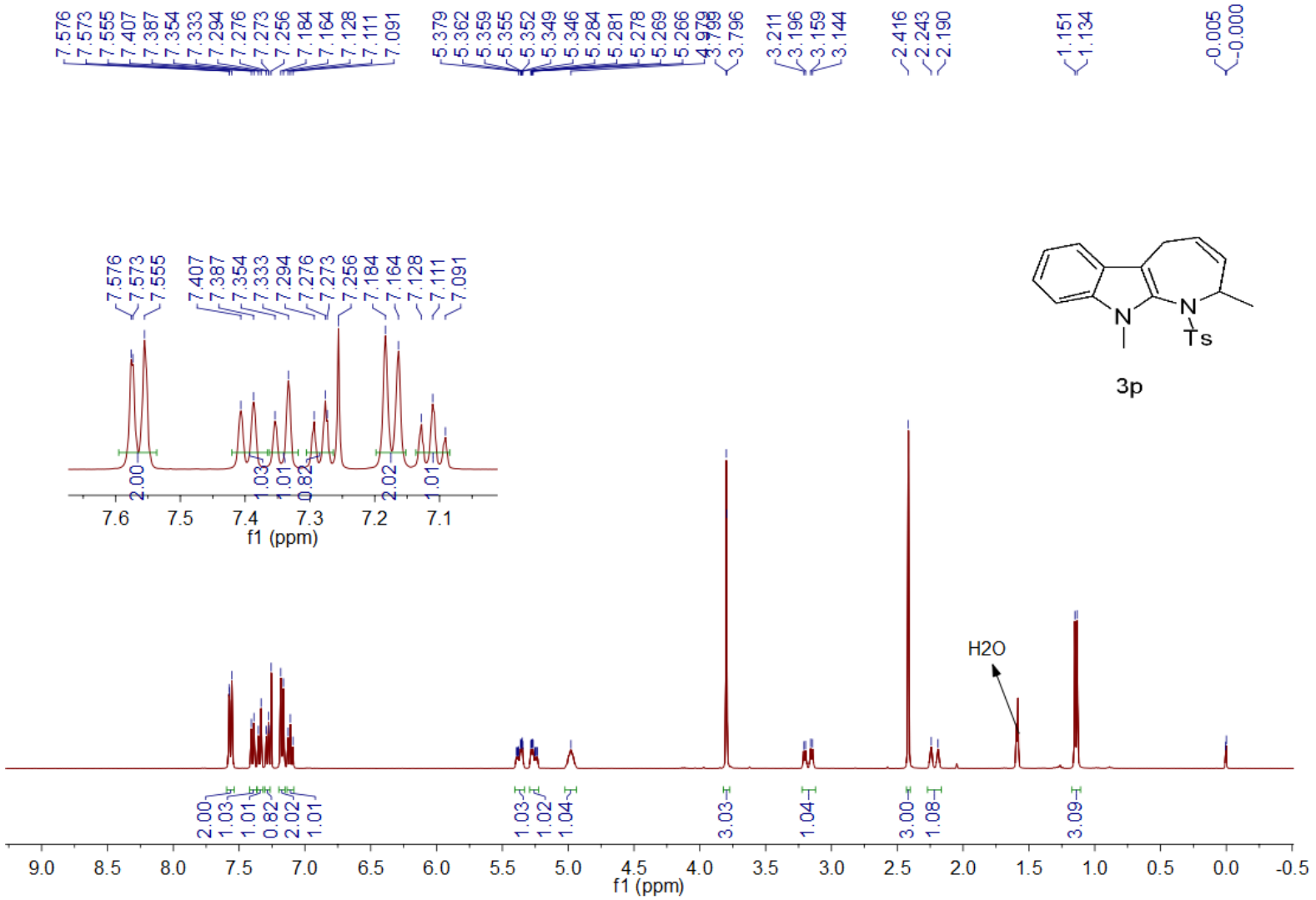
135.439
132.860
129.953
129.332
128.599
128.518
125.479
125.269
122.345
119.118
118.287
109.827
109.393
77.318
77.000
76.683
54.336
29.555
24.149
21.424
138
134
130
126
122
118
114
110
f1 (ppm)
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
3o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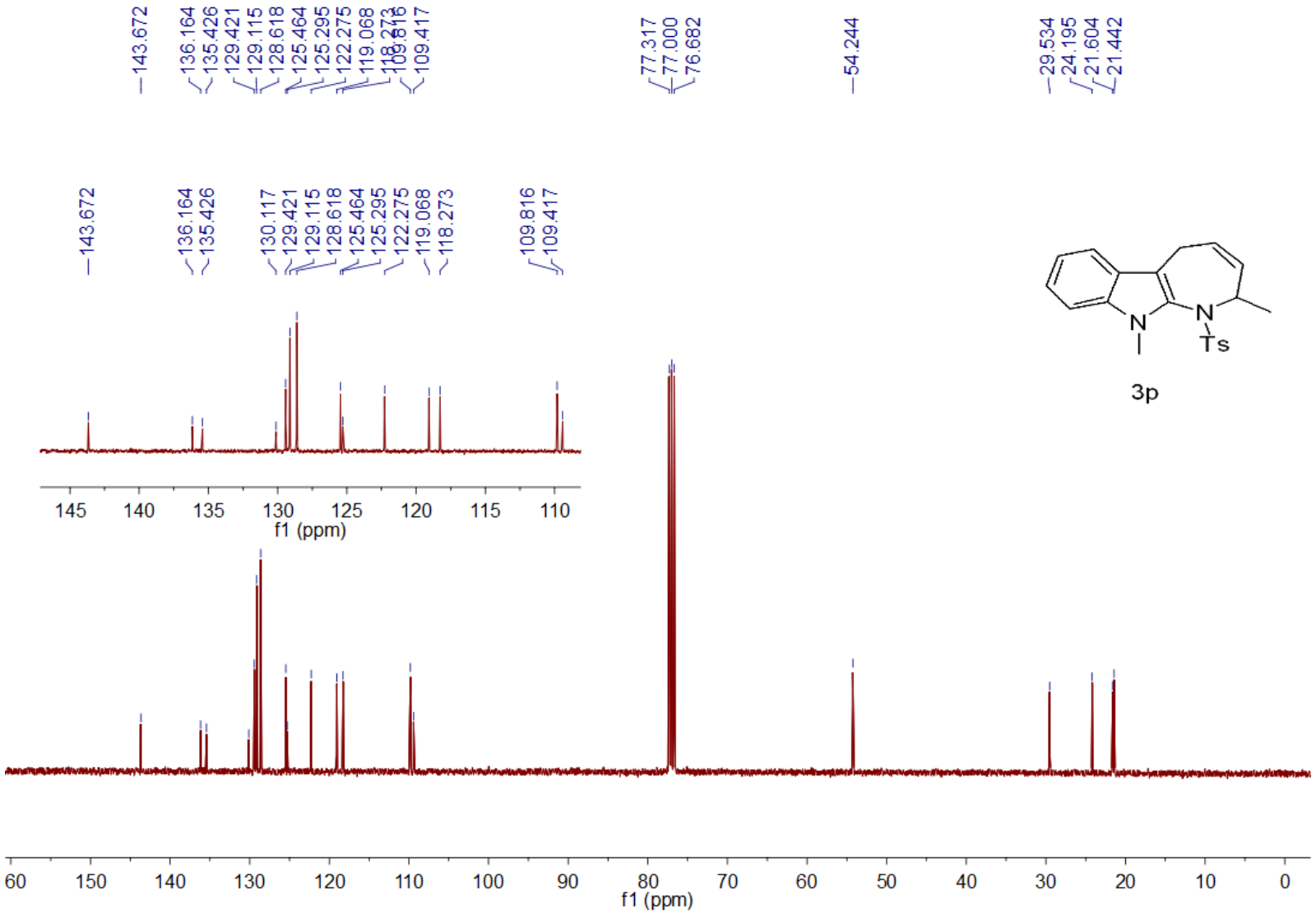
7.555
7.407
7.387
7.354
7.333
7.294
7.276
7.273
7.256
7.184
7.164
7.128
7.111
7.091
5.379
5.362
5.359
5.355
5.352
5.349
5.346
5.284
5.281
5.278
5.269
5.266
3.796
3.211
3.196
3.159
3.144
2.416
2.243
2.190
1.151
1.134
0.005
0.000
3p
H2O
f1 (ppm)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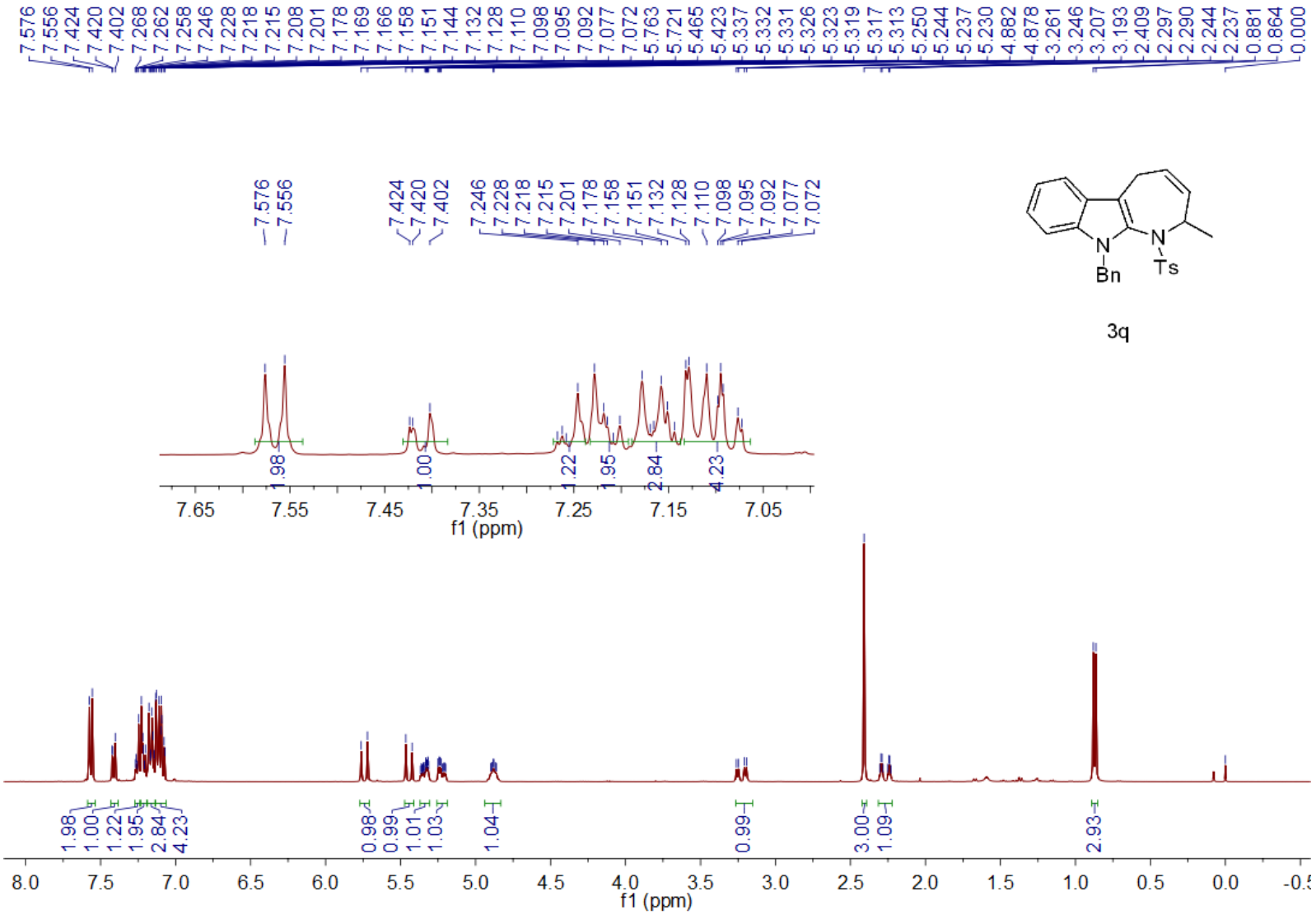
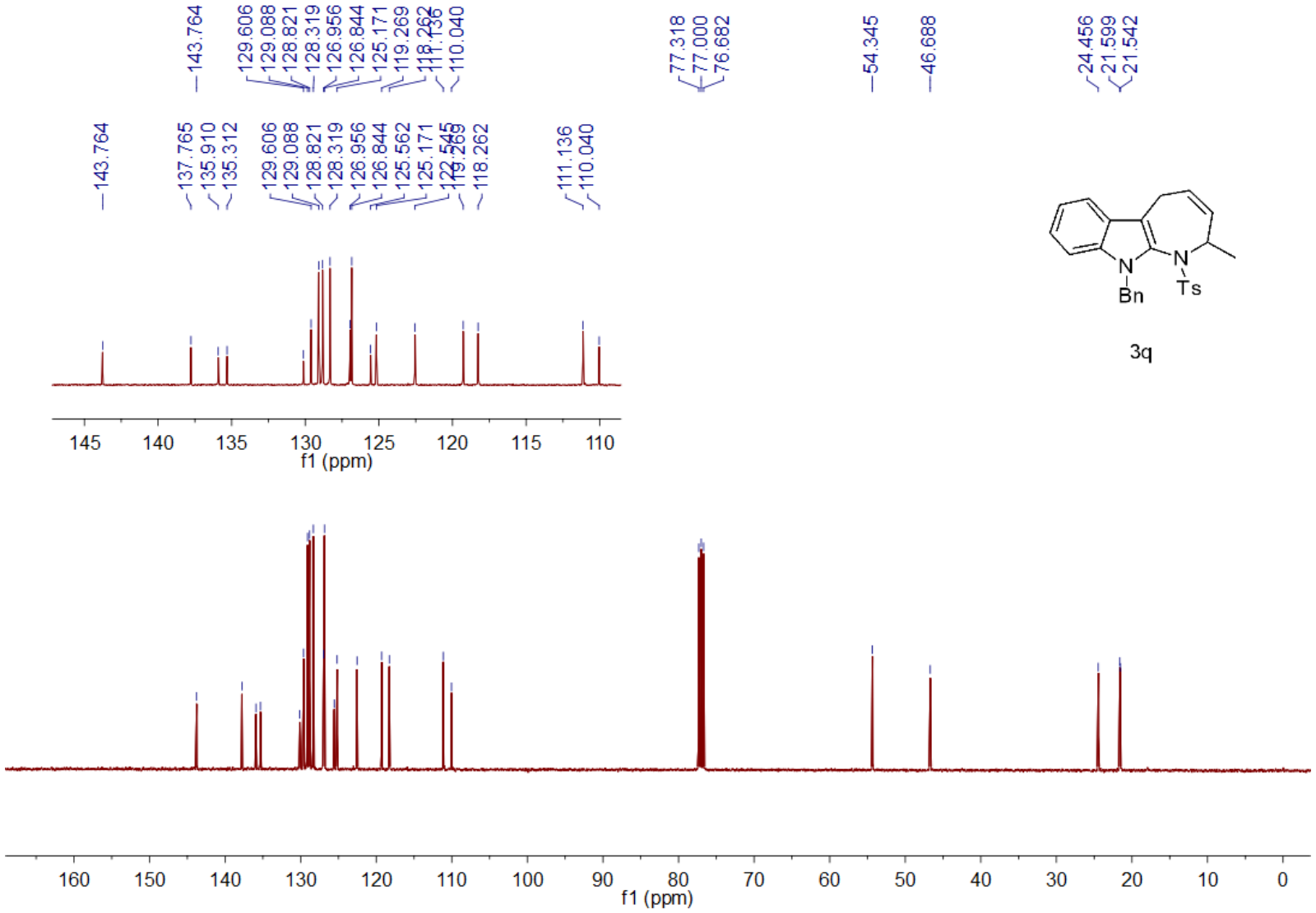
136.164
135.426
129.421
129.115
128.618
125.464
125.295
122.275
119.068
118.273
109.816
109.417
77.317
77.000
76.682
54.244
29.534
24.195
21.604
21.442
130.117
N
Ts
3p
f1 (ppm)


7.576
7.556
7.424
7.420
7.402
7.268
7.262
7.258
7.246
7.228
7.218
7.215
7.208
7.201
7.178
7.169
7.166
7.158
7.151
7.144
7.132
7.128
7.110
7.098
7.095
7.092
7.077
7.072
5.763
5.721
5.465
5.423
5.337
5.332
5.331
5.326
5.323
5.319
5.317
5.313
5.250
5.244
5.237
5.230
4.882
4.878
3.261
3.246
3.207
3.193
2.409
2.297
2.290
2.244
2.237
0.881
0.864
0.000
N
Bn
N
Ts
3q
1.98
1.00
1.22
1.95
2.84
4.23
0.98
0.99
1.01
1.03
1.04
0.99
3.00
1.09
2.93
f1 (ppm)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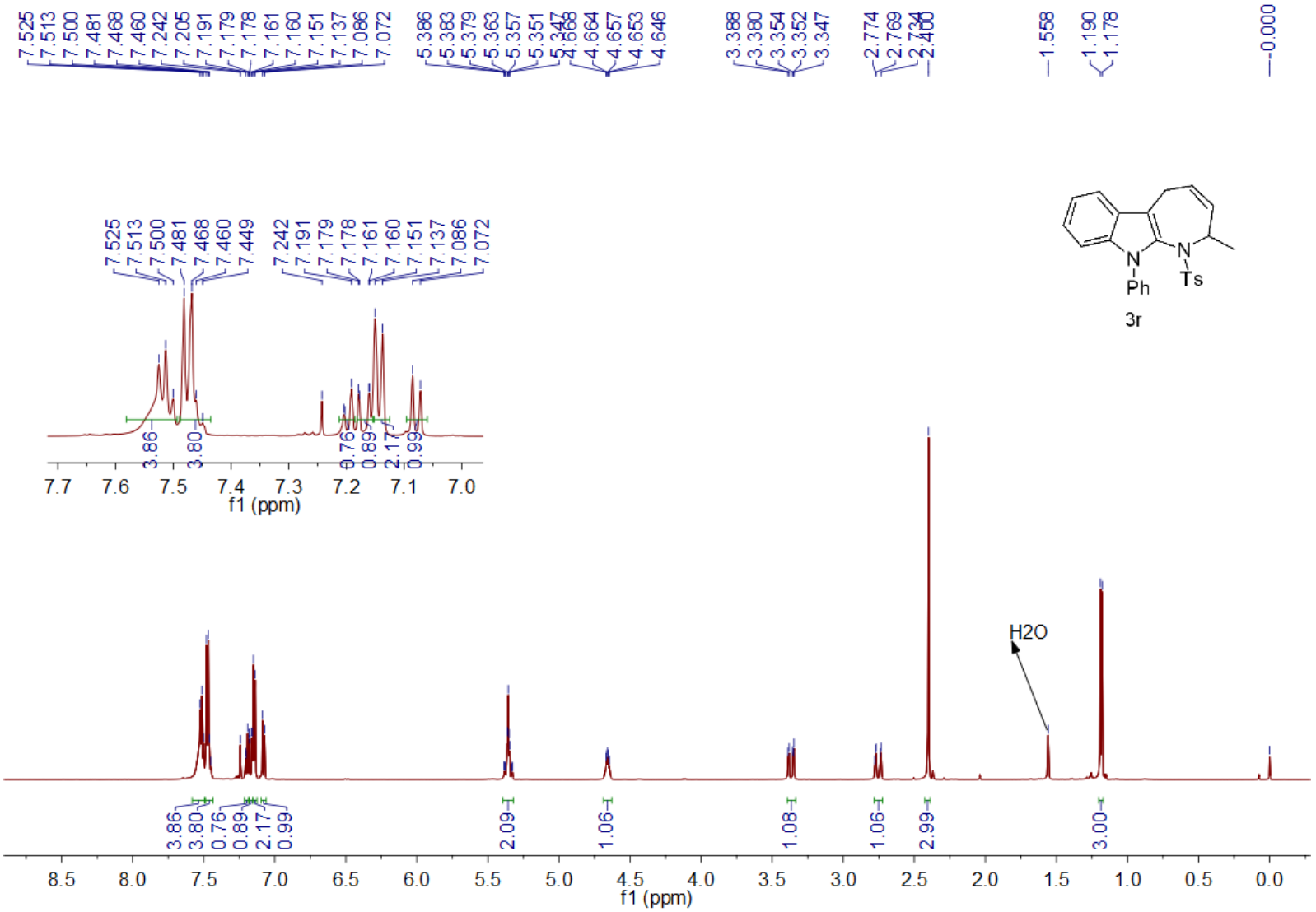
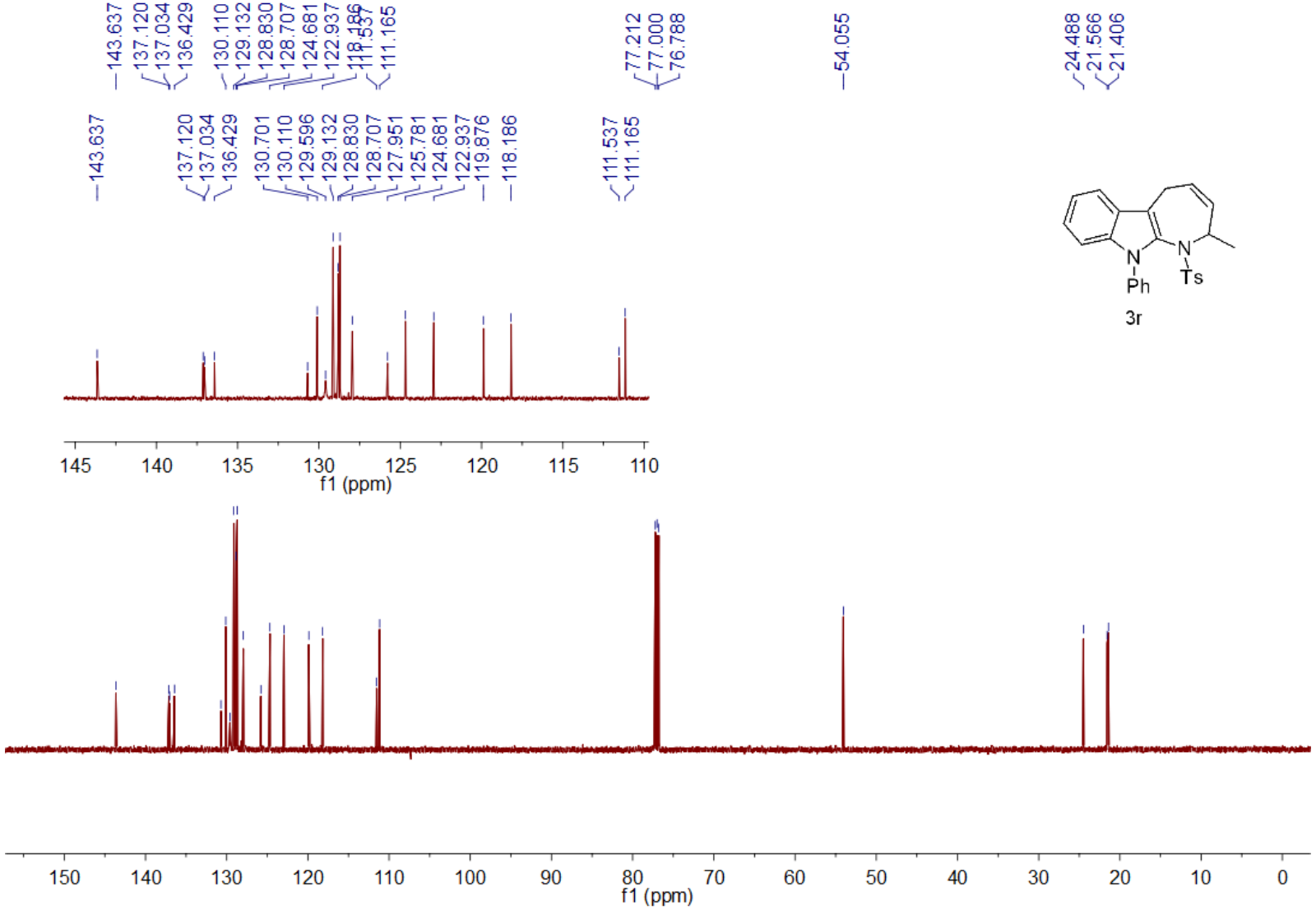
129.088
128.821
128.319
126.956
126.844
125.171
119.269
118.262
111.136
110.040
77.318
77.000
76.682
54.345
46.688
24.456
21.599
21.542
143.764
137.765
135.910
135.312
129.606
129.088
128.821
128.319
126.956
126.844
125.562
125.171
118.262
111.136
110.040
145 140 135 130 125 120 115 110
f1 (ppm)
N
Bn
N
Ts
3q
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.525
7.513
7.500
7.481
7.468
7.460
7.242
7.205
7.191
7.179
7.178
7.161
7.160
7.151
7.137
7.086
7.072
5.386
5.383
5.379
5.363
5.357
5.351
4.668
4.664
4.657
4.653
4.646
3.388
3.380
3.354
3.352
3.347
2.774
2.769
2.734
2.400
1.558
1.190
1.178
0.000
N
Ph
N
Ts
3r
3.86
3.80
0.76
0.89
2.17
0.99
f1 (ppm)
H2O
2.09
1.06
1.08
1.06
2.99
3.00
f1 (ppm)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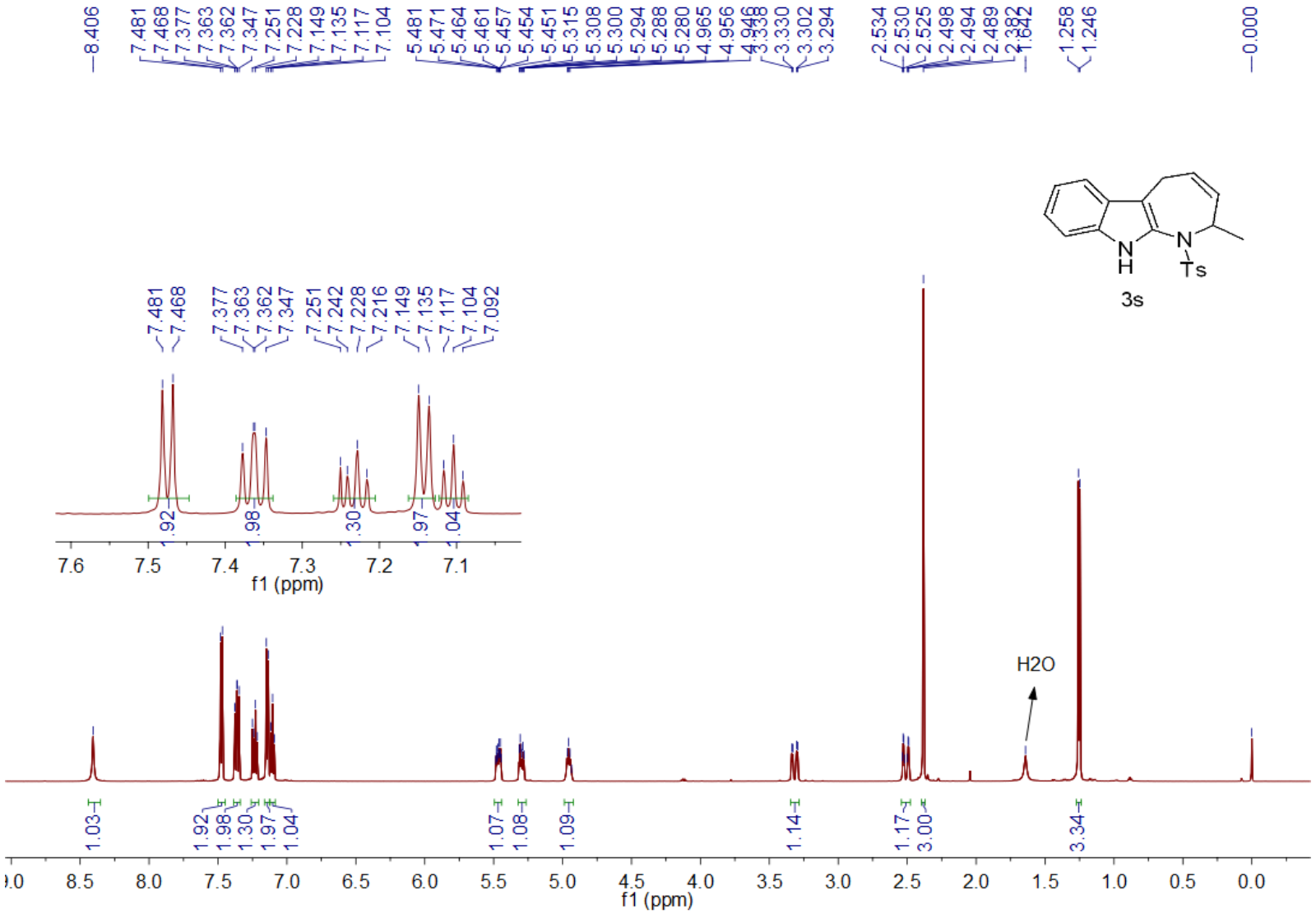
137.120
137.034
136.429
130.110
129.132
128.830
128.707
124.681
122.937
118.186
111.537
111.165
77.212
77.000
76.788
54.055
24.488
21.566
21.406
143.637
137.120
137.034
136.429
130.701
130.110
129.596
129.132
128.830
128.707
127.951
125.781
124.681
122.937
119.876
118.186
111.537
111.165
145 140 135 130 125 120 115 110
f1 (ppm)
N
Ph
N
Ts
3r
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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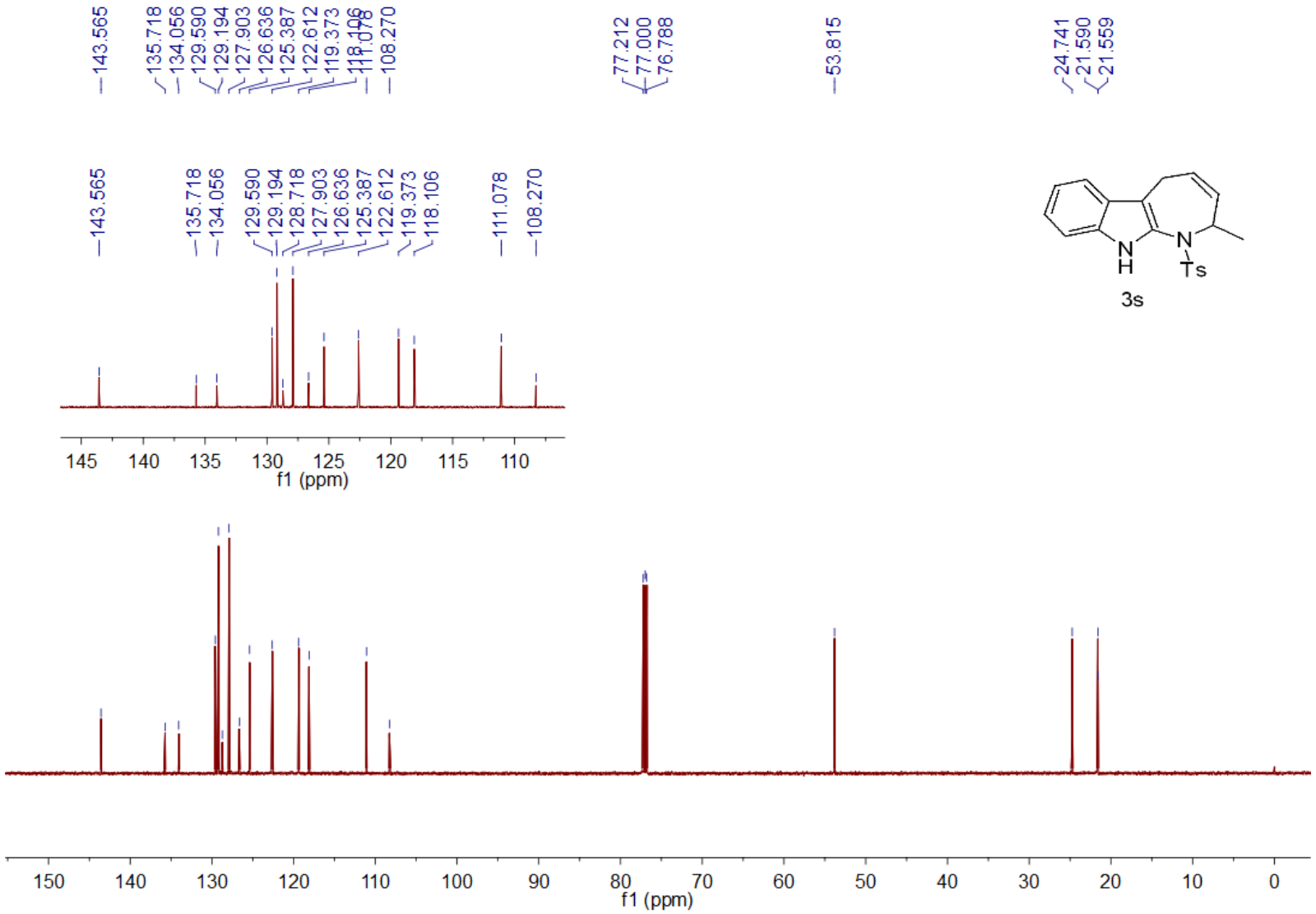
7.481
7.468
7.377
7.363
7.362
7.347
7.251
7.228
7.149
7.135
7.117
7.104
5.481
5.471
5.464
5.461
5.457
5.454
5.451
5.315
5.308
5.300
5.294
5.288
5.280
4.965
4.956
4.946
3.338
3.330
3.302
3.294
2.534
2.530
2.525
2.498
2.494
2.489
2.382
1.642
1.258
1.246
0.000
7.481
7.468
7.377
7.363
7.362
7.347
7.251
7.242
7.228
7.216
7.149
7.135
7.117
7.104
7.092
1.92
1.98
1.30
1.97
1.04
7.6
7.5
7.4
7.3
7.2
7.1
f1 (ppm)
N
H
N
Ts
3s
H2O
1.03
1.92
1.98
1.30
1.97
1.04
1.07
1.08
1.09
1.14
1.17
3.00
3.34
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)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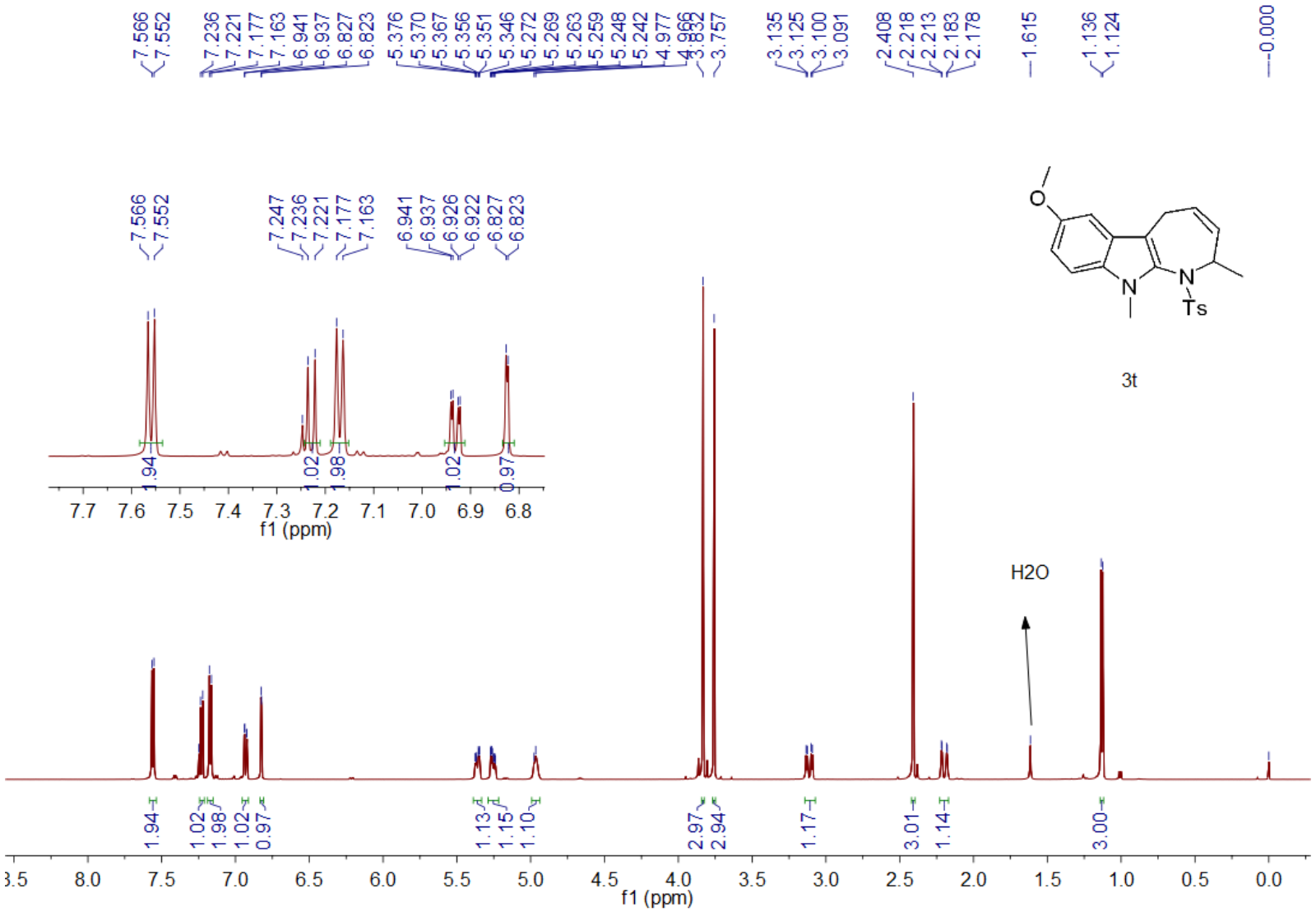
134.056
129.590
129.194
127.903
126.636
125.387
122.612
119.373
118.106
111.078
108.270
77.212
77.000
76.788
53.815
24.741
21.590
21.559
143.565
135.718
134.056
129.590
129.194
128.718
127.903
126.636
125.387
122.612
119.373
118.106
111.078
108.270
145 140 135 130 125 120 115 110
f1 (ppm)
N
H
N
Ts
3s
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.566
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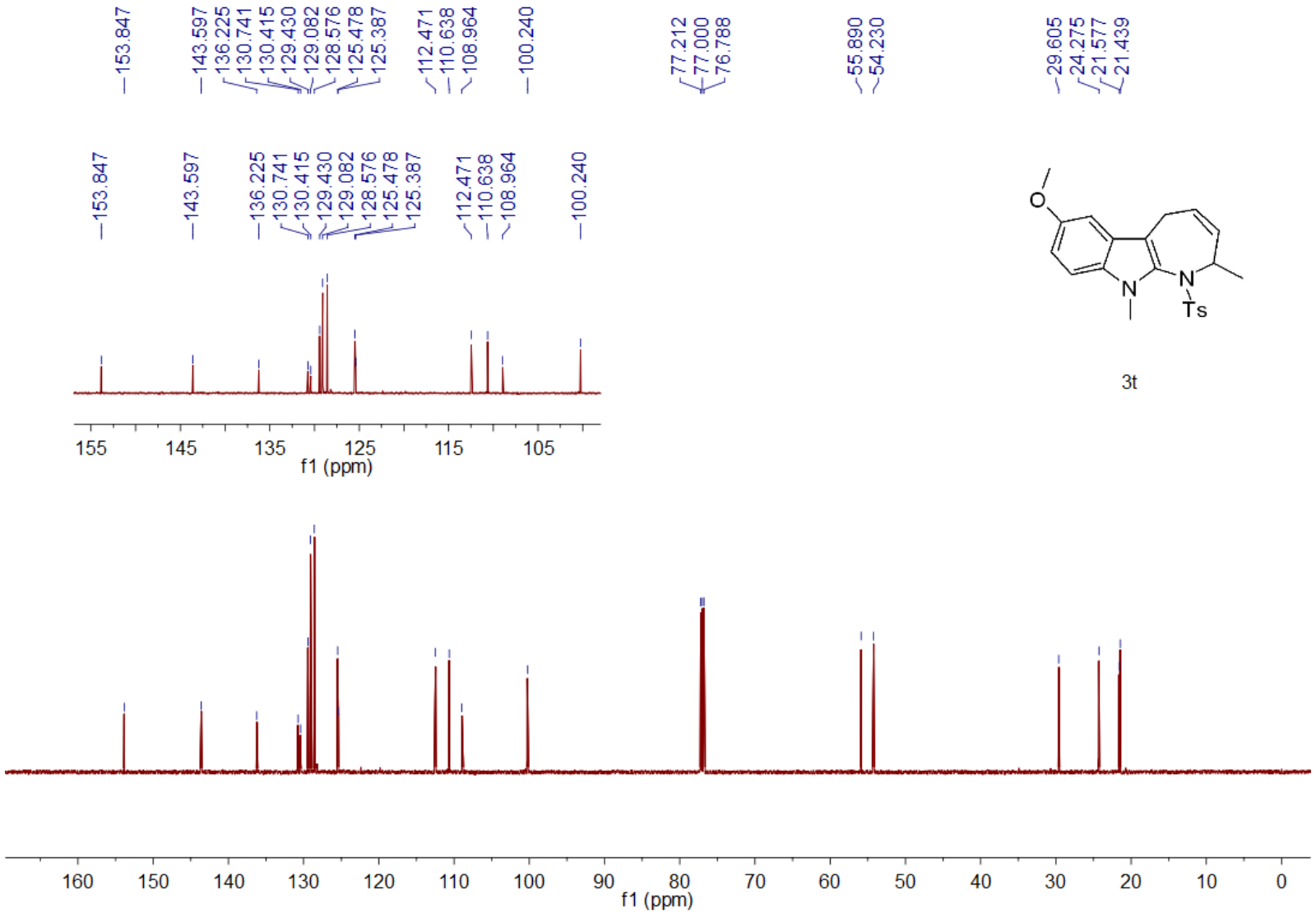
7.221
7.177
7.163
6.941
6.937
6.827
6.823
5.376
5.370
5.367
5.356
5.351
5.346
5.272
5.269
5.263
5.259
5.248
5.242
4.977
3.757
3.135
3.125
3.100
3.091
2.408
2.218
2.213
2.183
2.178
1.615
1.136
1.124
0.000
7.247
6.926
6.922
1.94
1.02
1.98
1.02
0.97
1.13
1.15
1.10
2.97
2.94
1.17
3.01
1.14
3.00
f1 (ppm)
H2O
O
N
N
Ts

3t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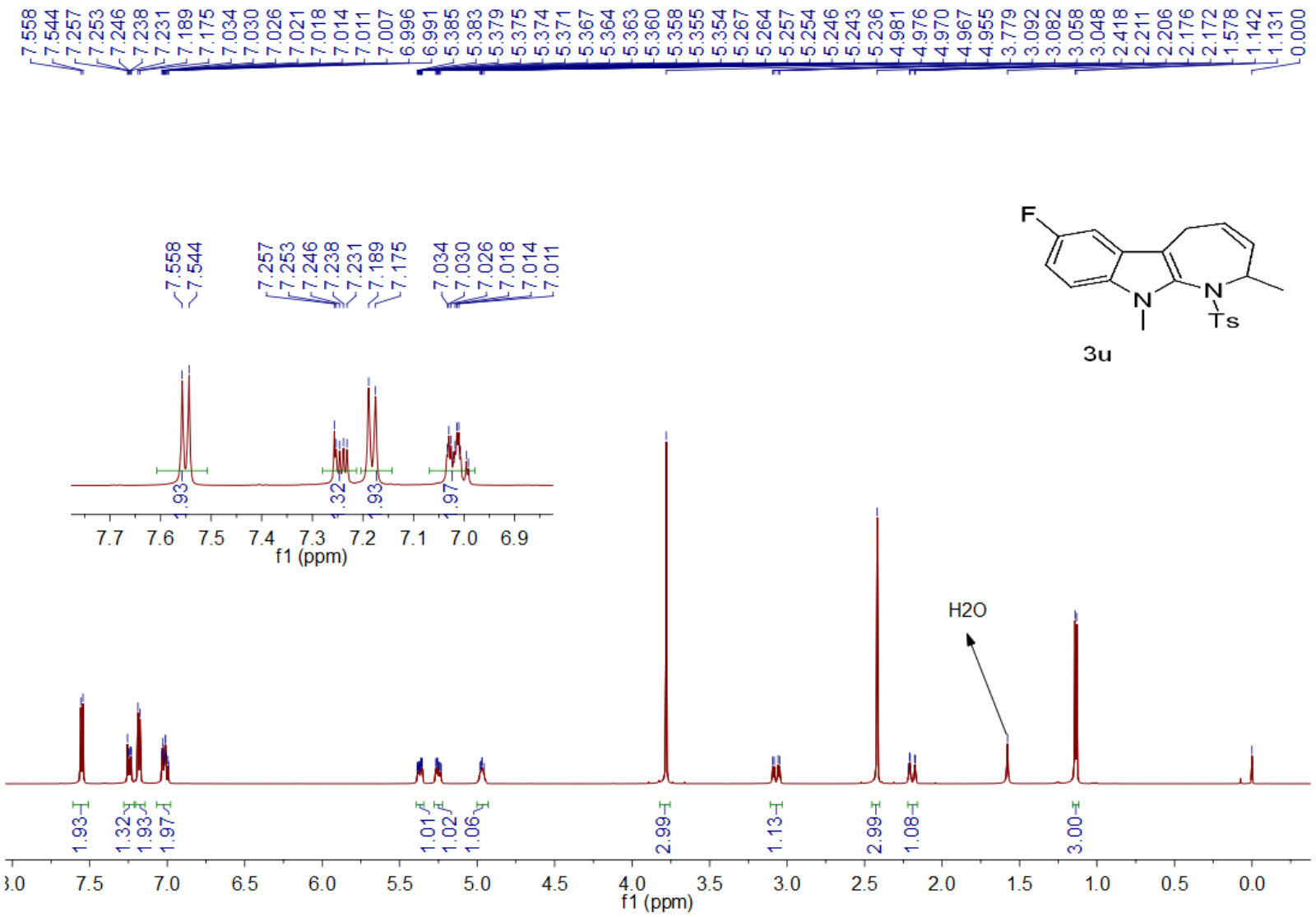
129.082
128.576
125.478
125.387
112.471
110.638
108.964
100.240
77.212
77.000
76.788
55.890
54.230
29.605
24.275
21.577
21.439
O
N
N
Ts
3t
155
145
135
125
115
105
f1 (ppm)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.253
7.246
7.238
7.231
7.189
7.175
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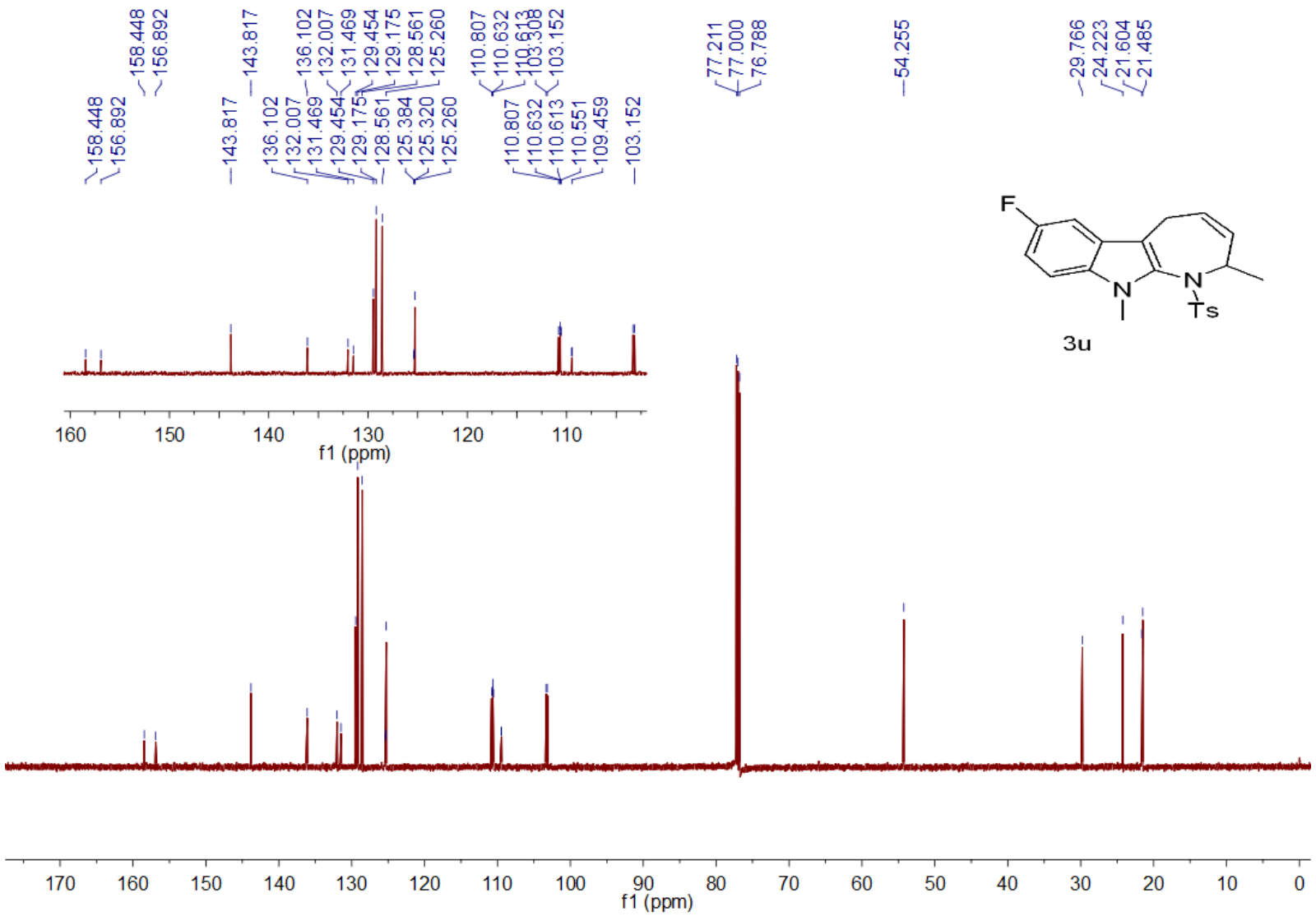
7.026
7.021
7.018
7.014
7.011
7.007
6.996
6.991
5.385
5.383
5.379
5.375
5.374
5.371
5.367
5.364
5.363
5.360
5.358
5.355
5.354
5.267
5.264
5.257
5.254
5.246
5.243
5.236
4.981
4.976
4.970
4.967
4.955
3.779
3.092
3.082
3.058
3.048
2.418
2.211
2.206
2.176
2.172
1.578
1.142
1.131
0.000
F
N
N
Ts
3u
1.93
1.32
1.93
1.97
f1 (ppm)
H2O
1.93
1.32
1.93
1.97
1.01
1.02
1.06
2.99
1.13
2.99
1.08
3.00
f1 (ppm)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129.175
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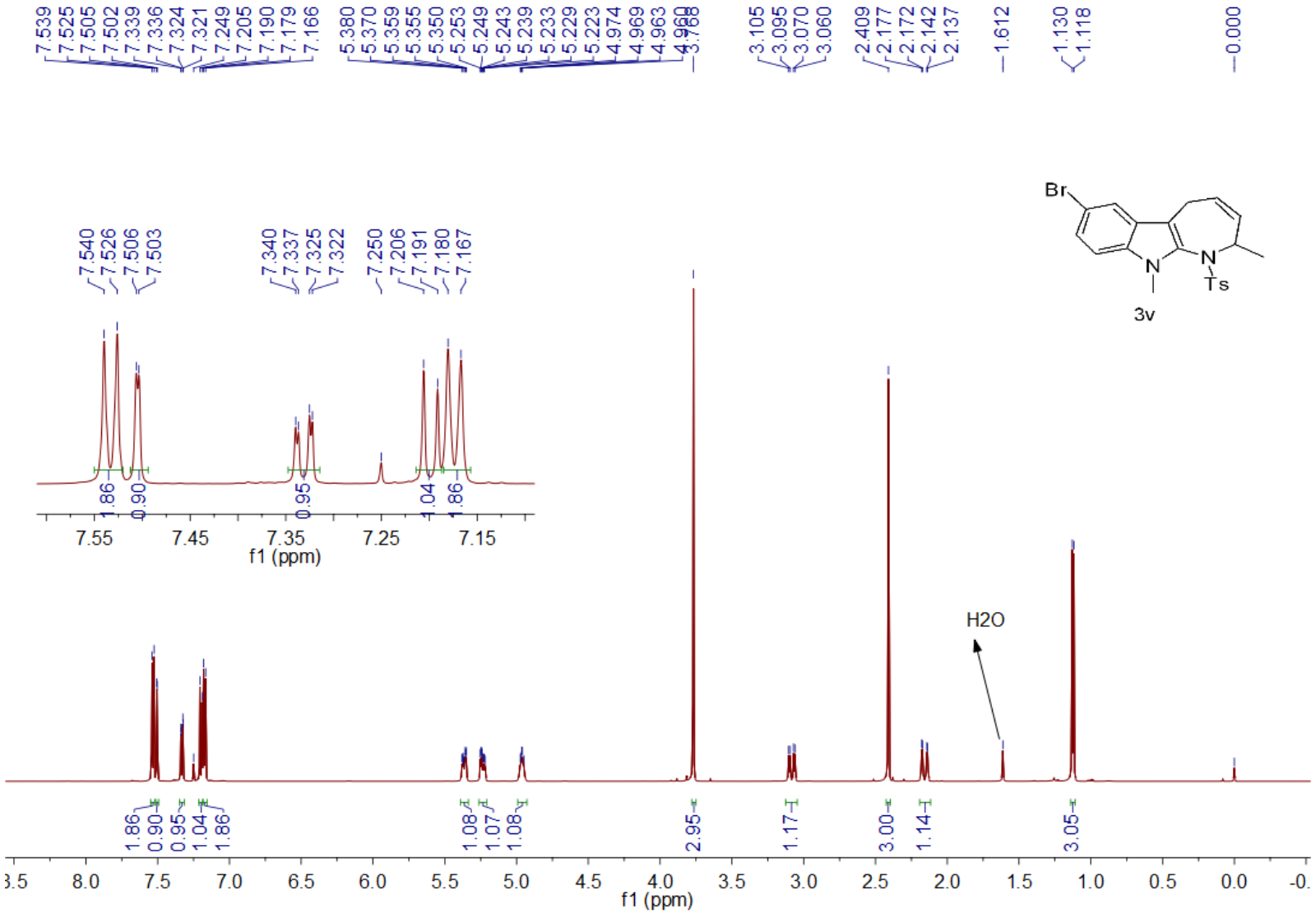
125.384
125.320
125.260
110.807
110.632
110.613
110.551
109.459
103.152
77.211
77.000
76.788
54.255
29.766
24.223
21.604
21.485
F
N
N
Ts
3u
160
150
140
130
120
110
f1 (ppm)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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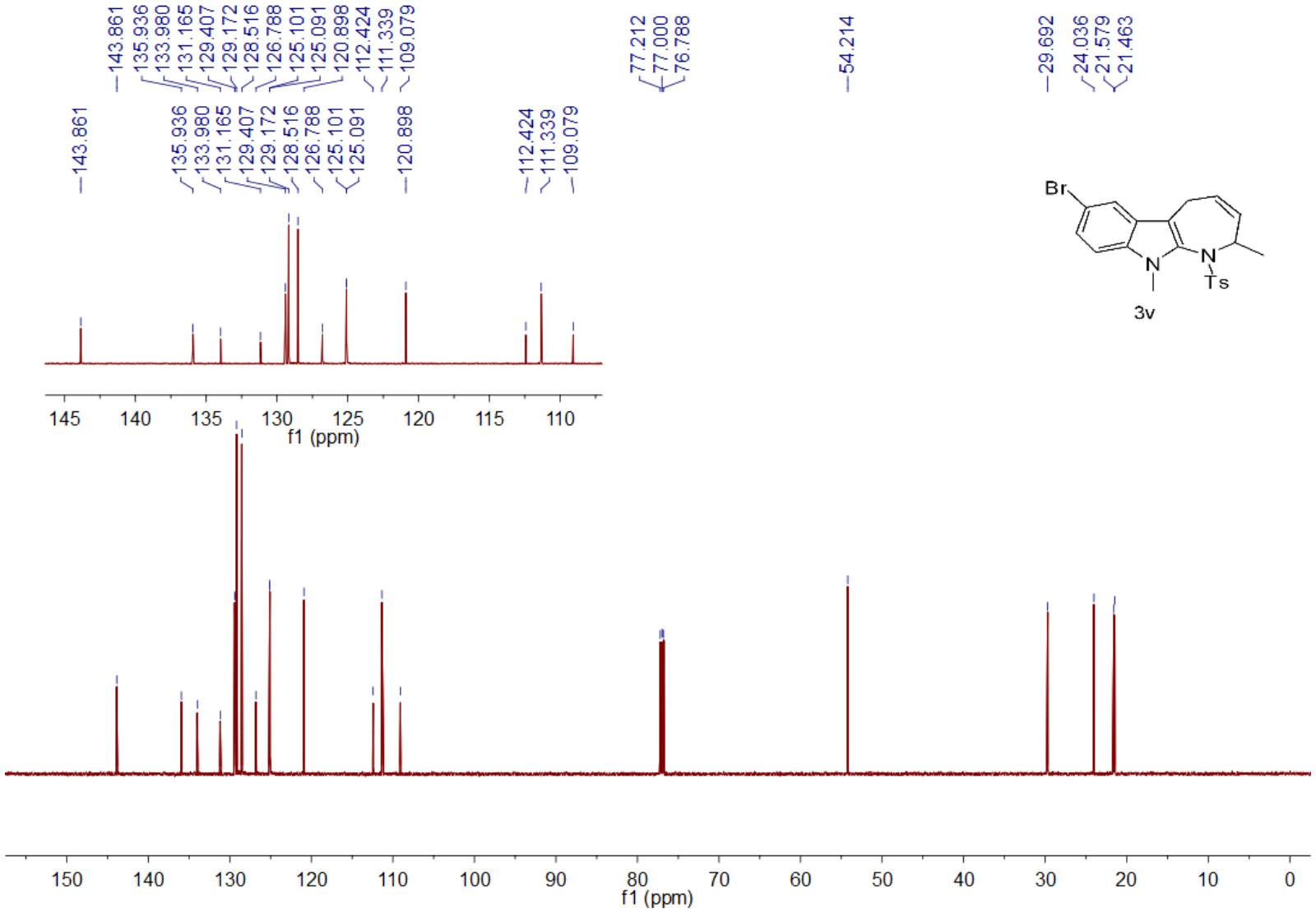
7.321
7.249
7.205
7.190
7.179
7.166
5.380
5.370
5.359
5.355
5.350
5.253
5.249
5.243
5.239
5.233
5.229
5.223
4.974
4.969
4.963
3.105
3.095
3.070
3.060
2.409
2.177
2.172
2.142
2.137
1.612
1.130
1.118
0.000
Br
N
N
Ts
3v
7.540
7.526
7.506
7.503
7.340
7.337
7.325
7.322
7.250
7.206
7.191
7.180
7.167
1.86
0.90
0.95
1.04
1.86
7.55
7.45
7.35
7.25
7.15
f1 (ppm)
H2O
1.86
0.90
0.95
1.04
1.86
1.08
1.07
1.08
2.95
1.17
3.00
1.14
3.05
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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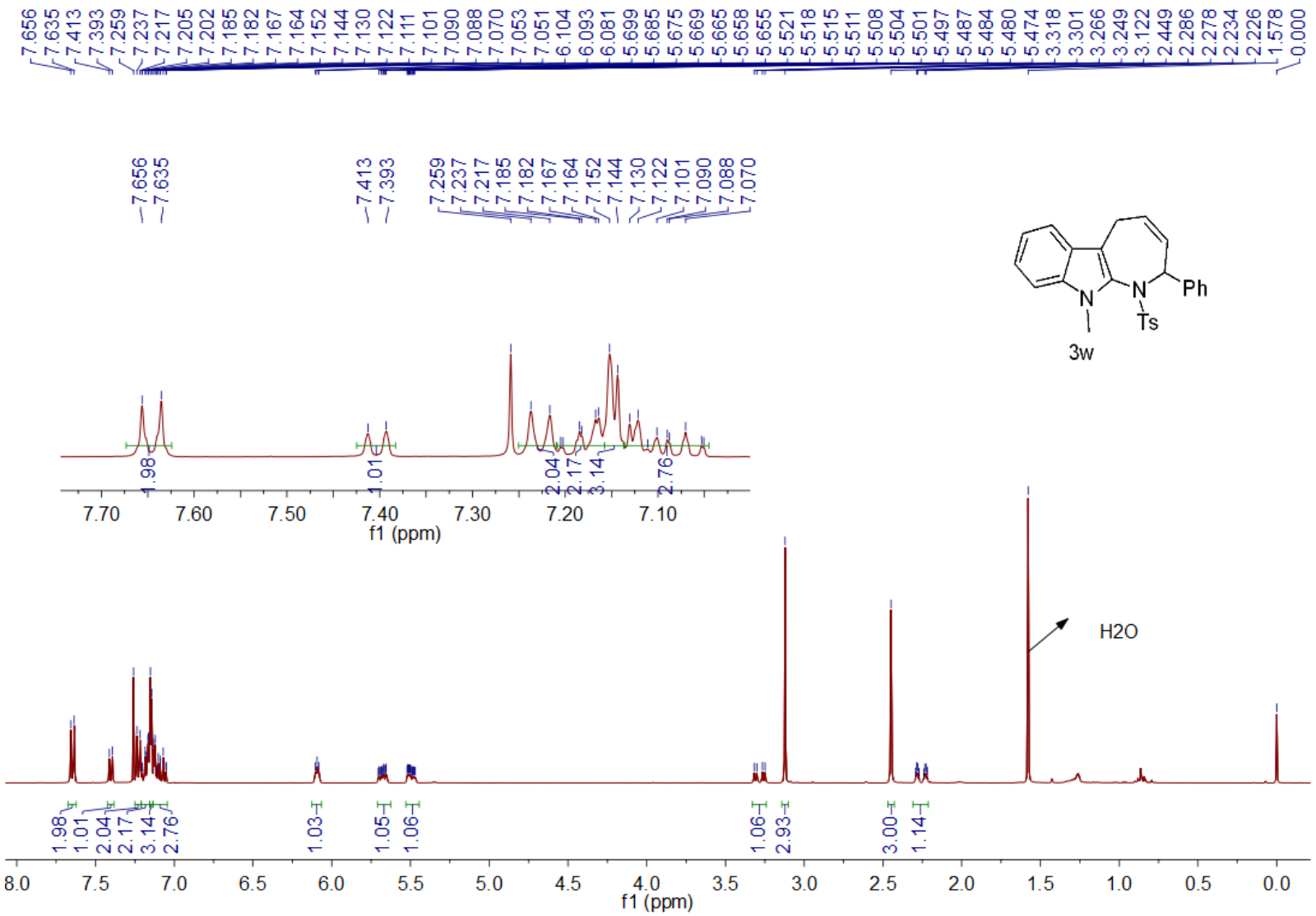
129.172
128.516
126.788
125.101
125.091
120.898
112.424
111.339
109.079
77.212
77.000
76.788
54.214
29.692
24.036
21.579
21.463
Br
N
N
Ts
3v
f1 (ppm)
f1 (ppm)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7.237
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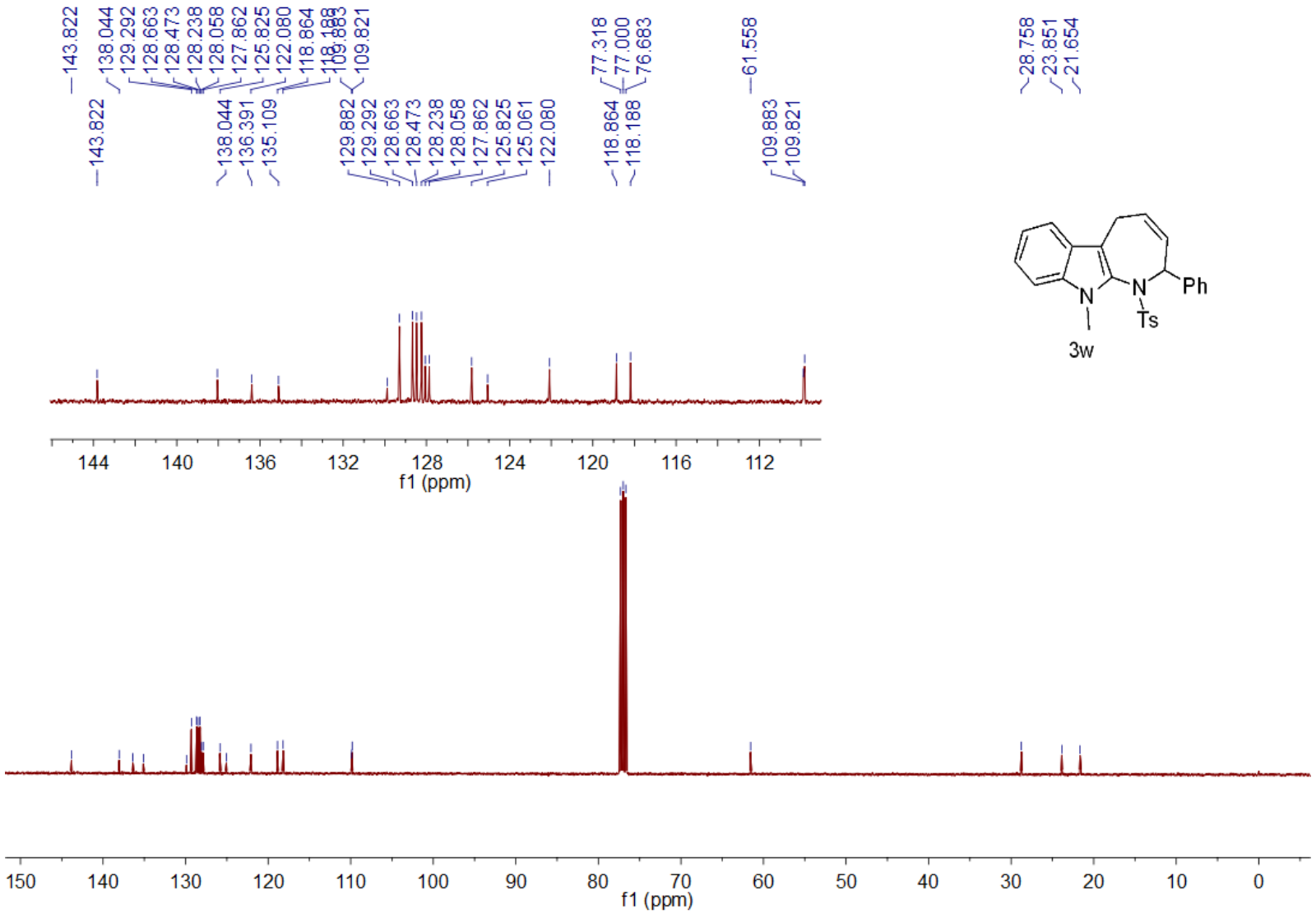
7.202
7.185
7.182
7.167
7.164
7.152
7.144
7.130
7.122
7.111
7.101
7.090
7.088
7.070
7.053
7.051
6.104
6.093
6.081
5.699
5.685
5.675
5.669
5.665
5.658
5.655
5.521
5.518
5.515
5.511
5.508
5.504
5.501
5.497
5.487
5.484
5.480
5.474
3.318
3.301
3.266
3.249
3.122
2.449
2.286
2.278
2.234
2.226
1.578
0.000
N
N
Ph
Ts
3w
1.98
1.01
2.04
2.17
3.14
2.76
7.70
7.60
7.50
7.40
7.30
7.20
7.10
f1 (ppm)
H2O
1.03
1.05
1.06
1.06
2.93
3.00
1.14
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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128.238
128.058
127.862
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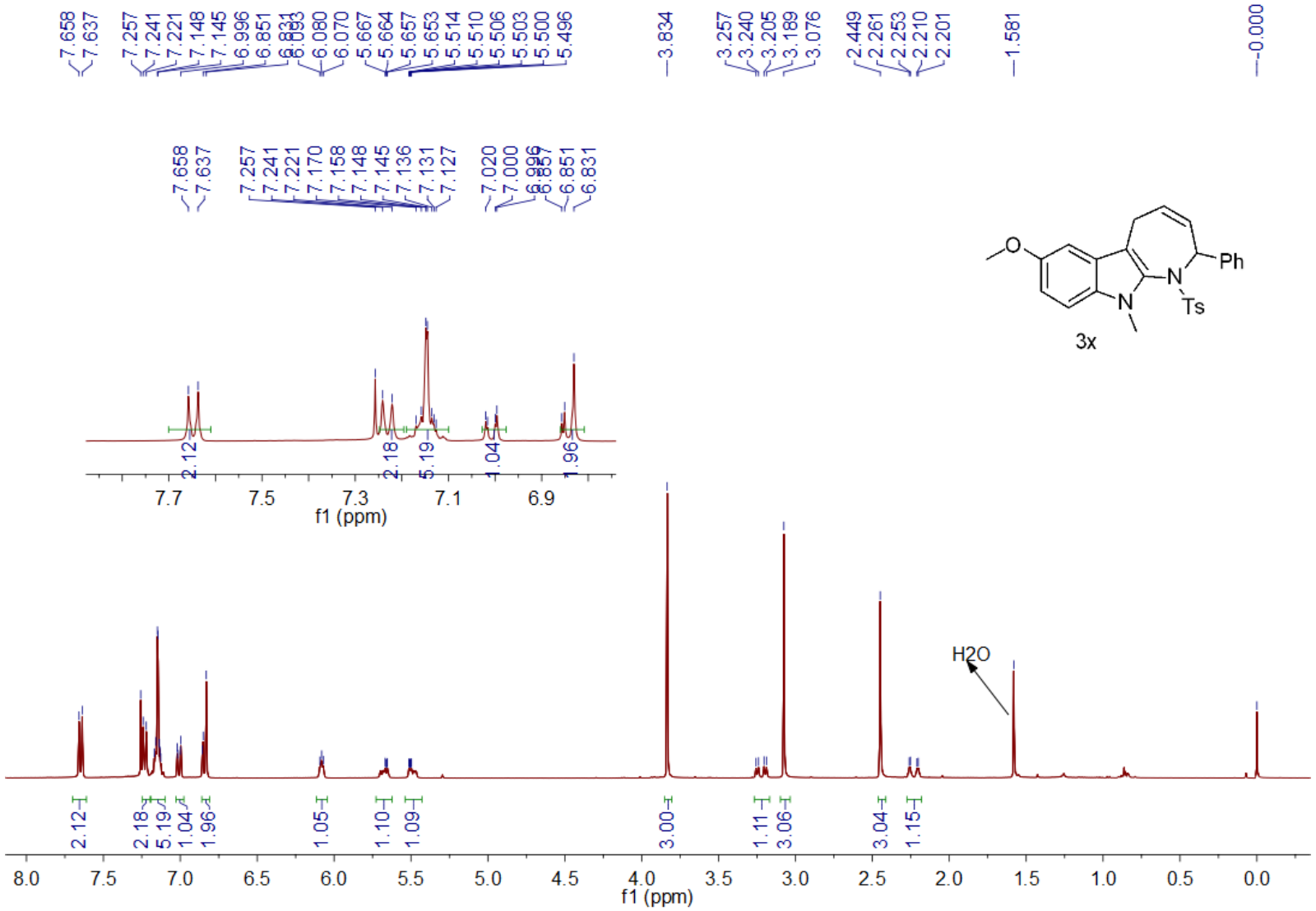
122.080
118.864
118.188
109.883
109.821
77.318
77.000
76.683
61.558
28.758
23.851
21.654
143.822
138.044
136.391
135.109
129.882
129.292
128.663
128.473
128.238
128.058
127.862
125.825
125.061
122.080
118.864
118.188
109.883
109.821
Ph
N
N
Ts
3w
144
140
136
132
128
124
120
116
112
f1 (ppm)
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.241
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7.145
6.996
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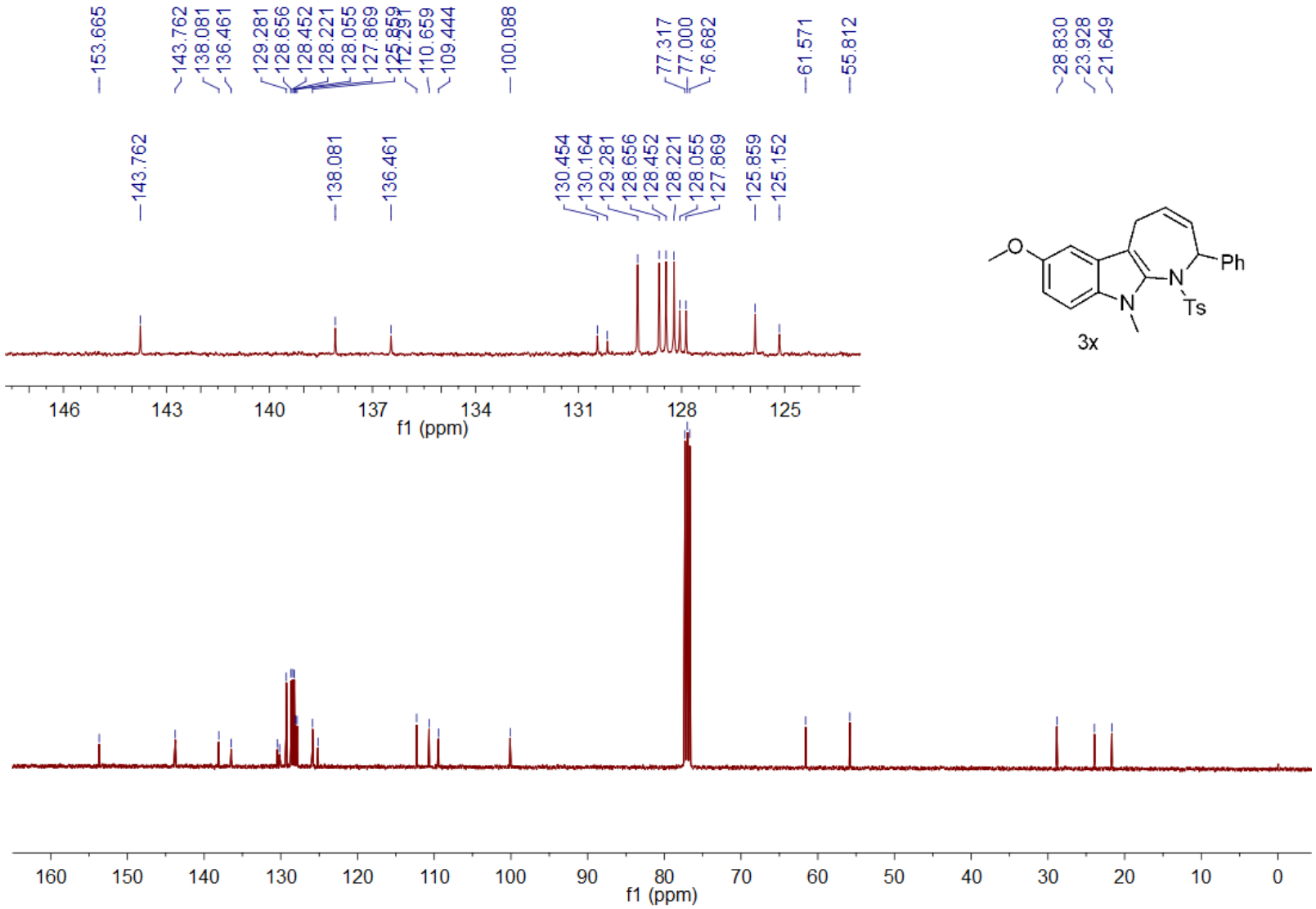
6.833
6.080
6.070
5.667
5.664
5.657
5.653
5.514
5.510
5.506
5.503
5.500
5.496
3.834
3.257
3.240
3.205
3.189
3.076
2.449
2.261
2.253
2.210
2.201
1.581
0.000
7.658
7.637
7.257
7.241
7.221
7.170
7.158
7.148
7.145
7.136
7.131
7.127
7.020
7.000
6.851
6.831
O
Ph
N
Ts
N
3x
2.12
2.18
5.19
1.04
1.96
7.7
7.5
7.3
7.1
6.9
f1 (ppm)
H2O
2.12
2.18
5.19
1.04
1.96
1.05
1.10
1.09
3.00
1.11
3.06
3.04
1.15
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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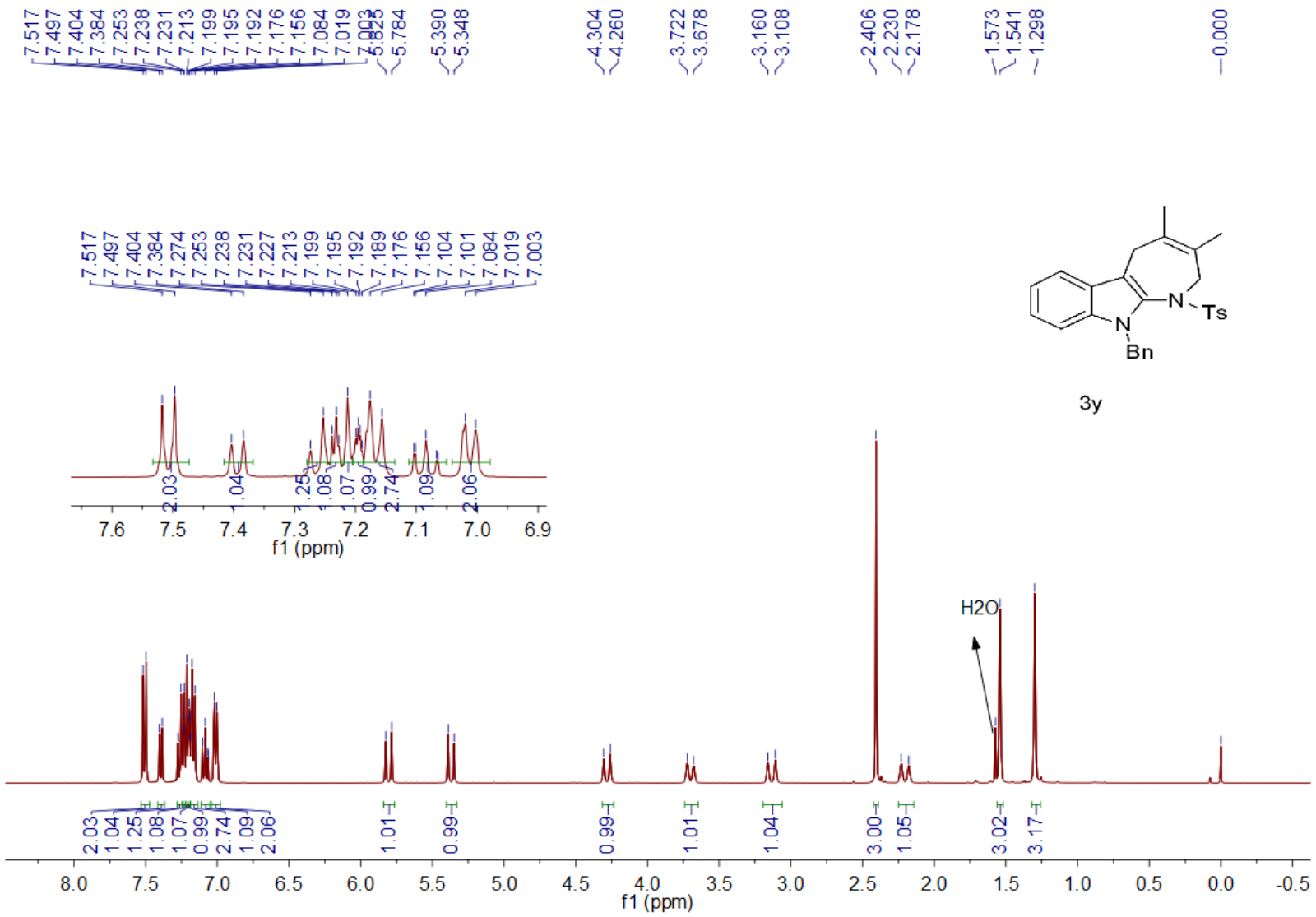
143.762
138.081
136.461
129.281
128.656
128.452
128.221
128.055
127.869
125.859
125.152
110.659
109.444
100.088
77.317
77.000
76.682
61.571
55.812
28.830
23.928
21.649
143.762
138.081
136.461
130.454
130.164
129.281
128.656
128.452
128.221
128.055
127.869
125.859
125.152
146
143
140
137
134
131
128
125
f1 (ppm)
O
N
N
Ph
Ts
3x
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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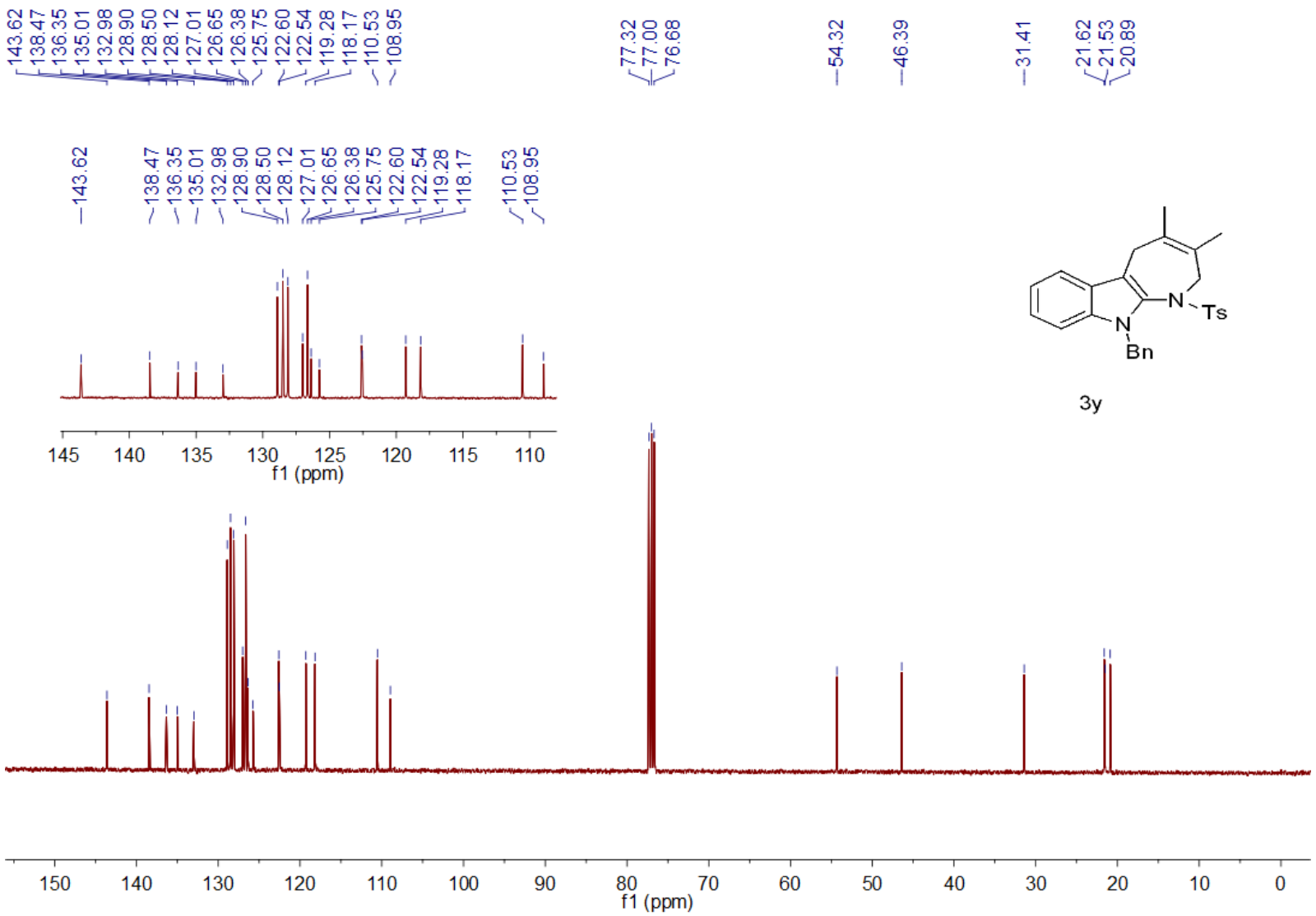
7.497
7.404
7.384
7.253
7.238
7.231
7.213
7.199
7.195
7.192
7.176
7.156
7.084
7.019
5.825
5.784
5.390
5.348
4.304
4.260
3.722
3.678
3.160
3.108
2.406
2.230
2.178
1.573
1.541
1.298
0.000
N
Ts
N
Bn
3y
H2O
f1 (ppm)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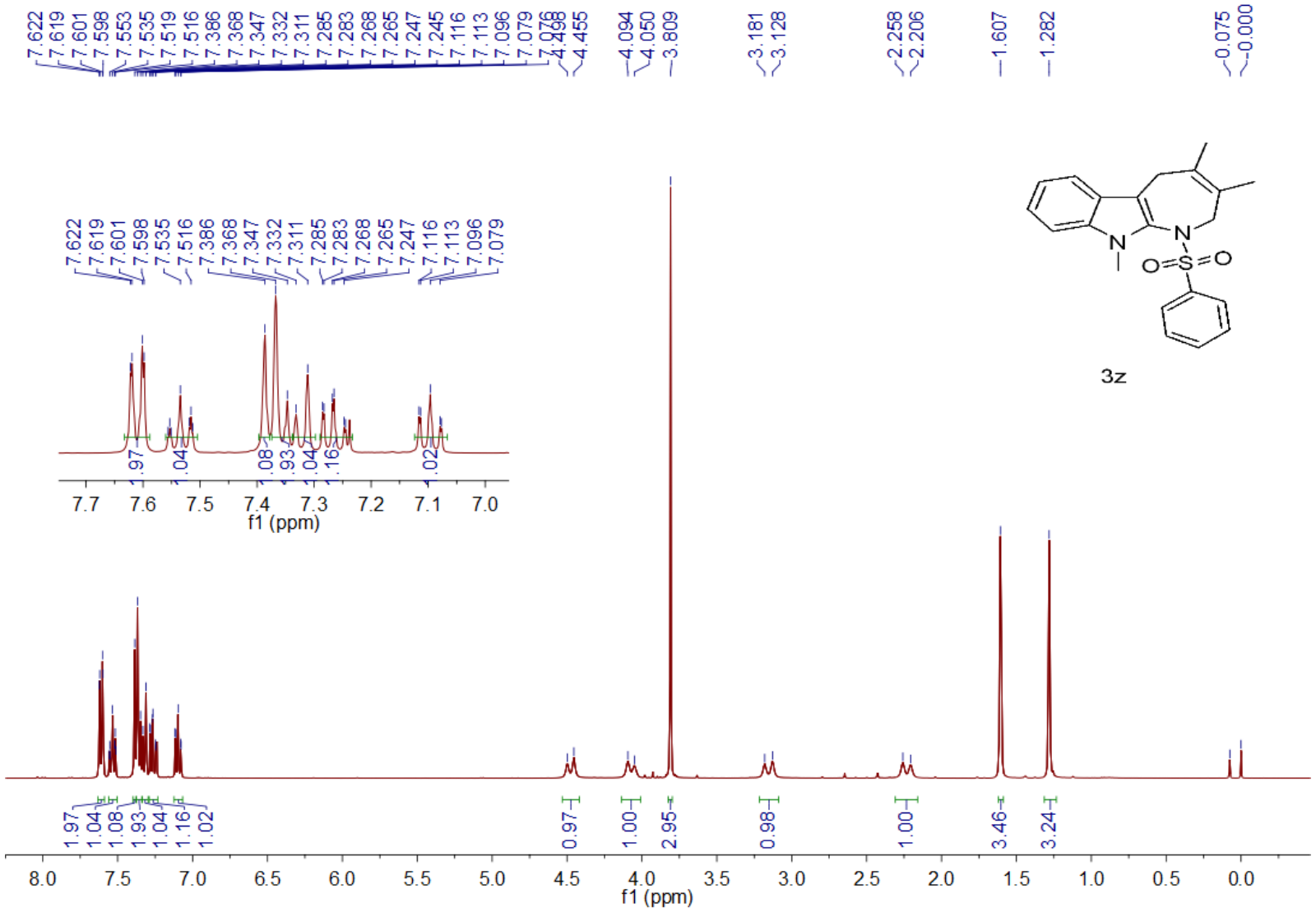
138.47
136.35
135.01
132.98
128.90
128.50
128.12
127.01
126.65
126.38
125.75
122.60
122.54
119.28
118.17
110.53
108.95
77.32
77.00
76.68
54.32
46.39
31.41
21.62
21.53
20.89
N
Ts
N
Bn
3y
f1 (ppm)
f1 (ppm)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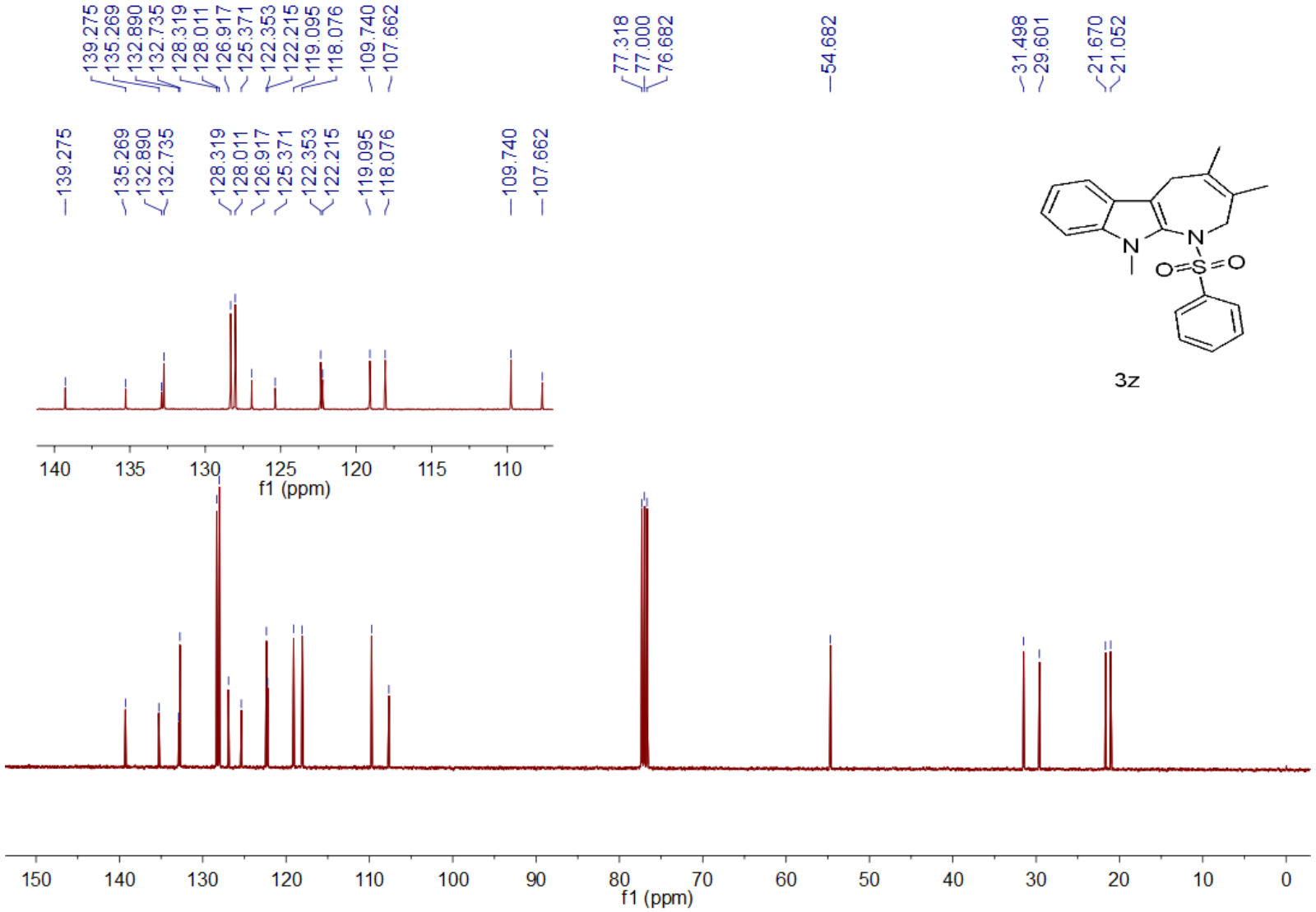
7.619
7.601
7.598
7.553
7.535
7.519
7.516
7.386
7.368
7.347
7.332
7.311
7.285
7.283
7.268
7.265
7.247
7.245
7.116
7.113
7.096
7.079
4.455
4.094
4.050
3.809
3.181
3.128
2.258
2.206
1.607
1.282
0.075
0.000
1.97
1.04
1.08
1.93
1.04
1.16
1.02
0.97
1.00
2.95
0.98
1.00
3.46
3.24
f1 (ppm)
3z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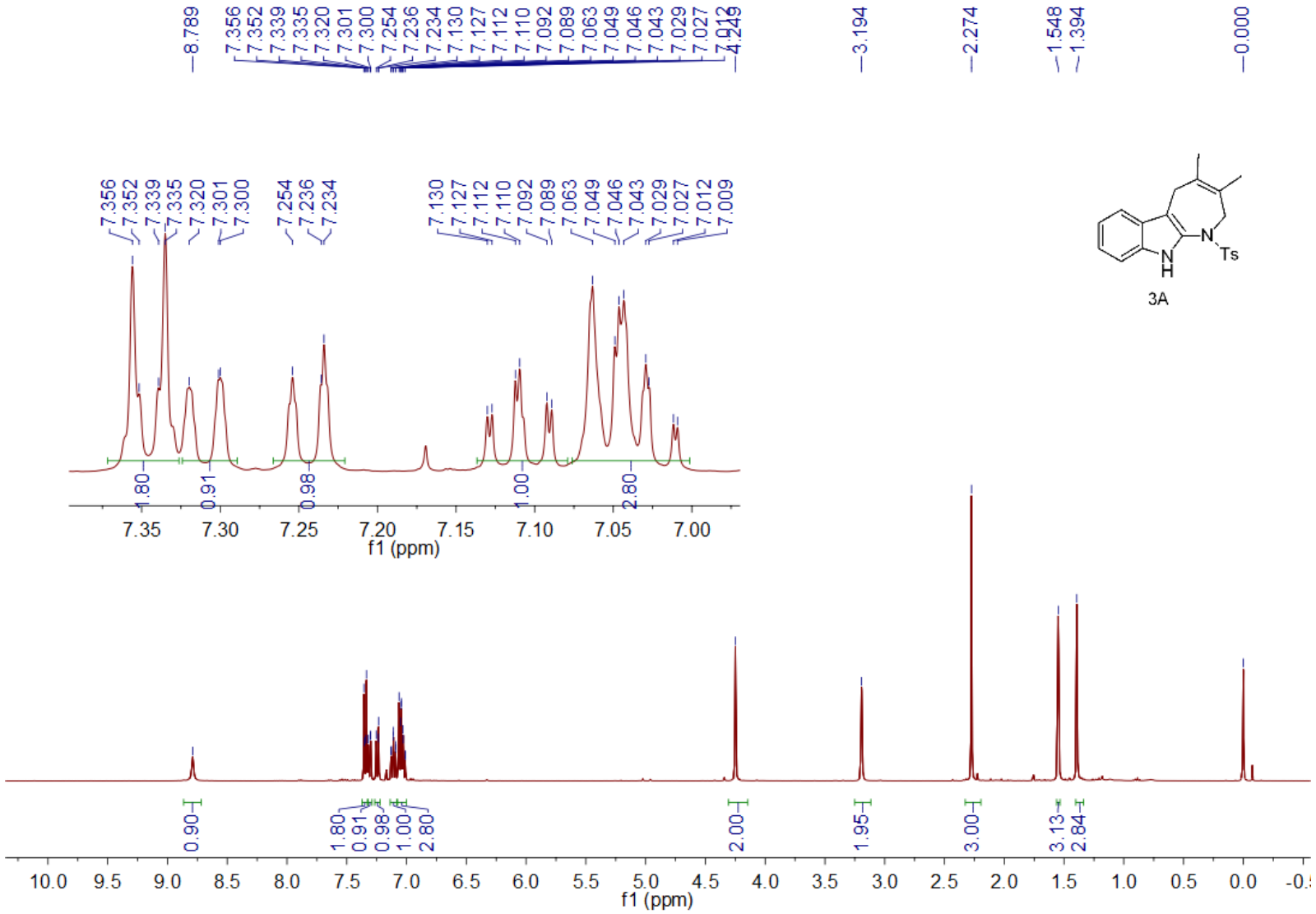
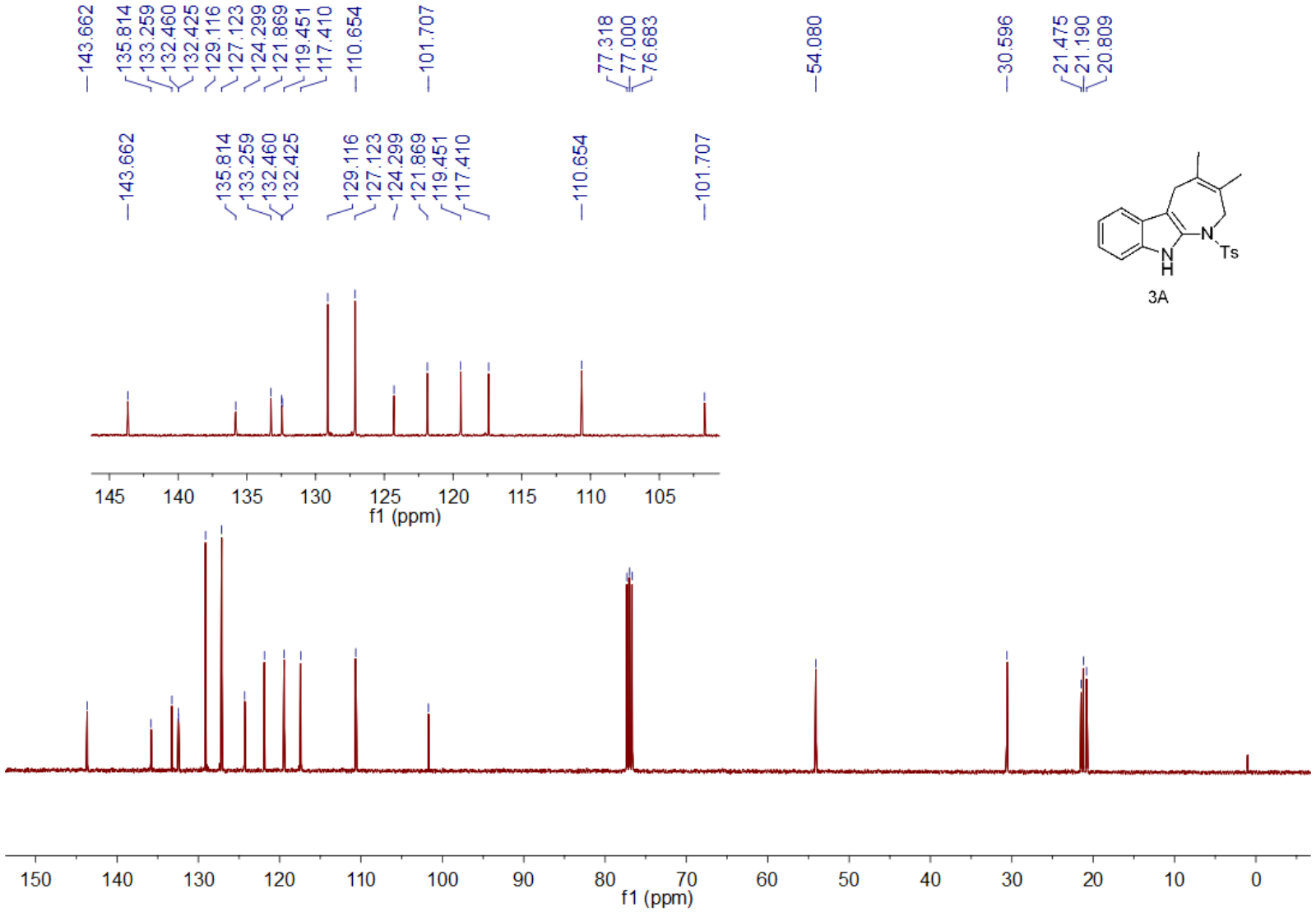
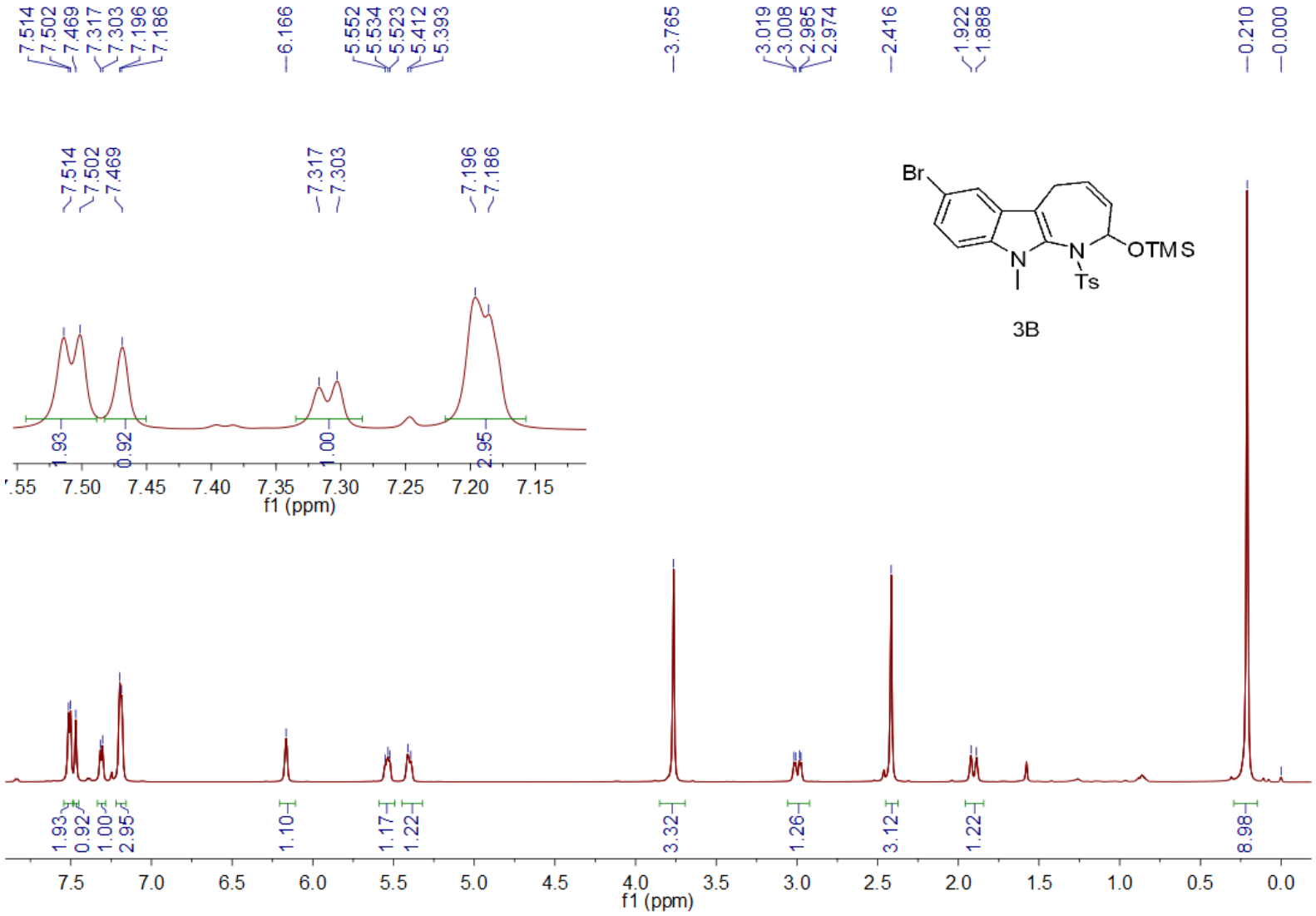
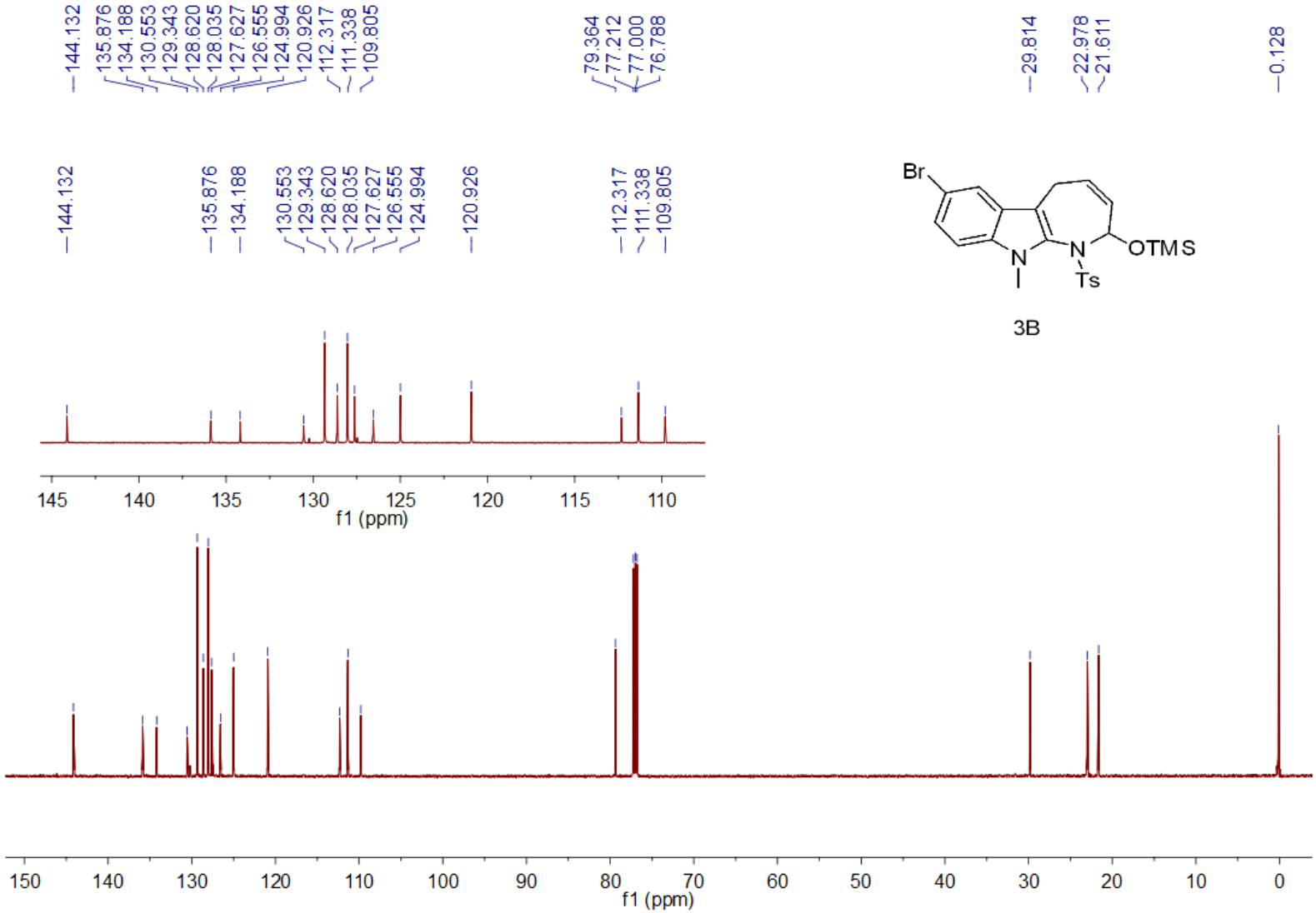
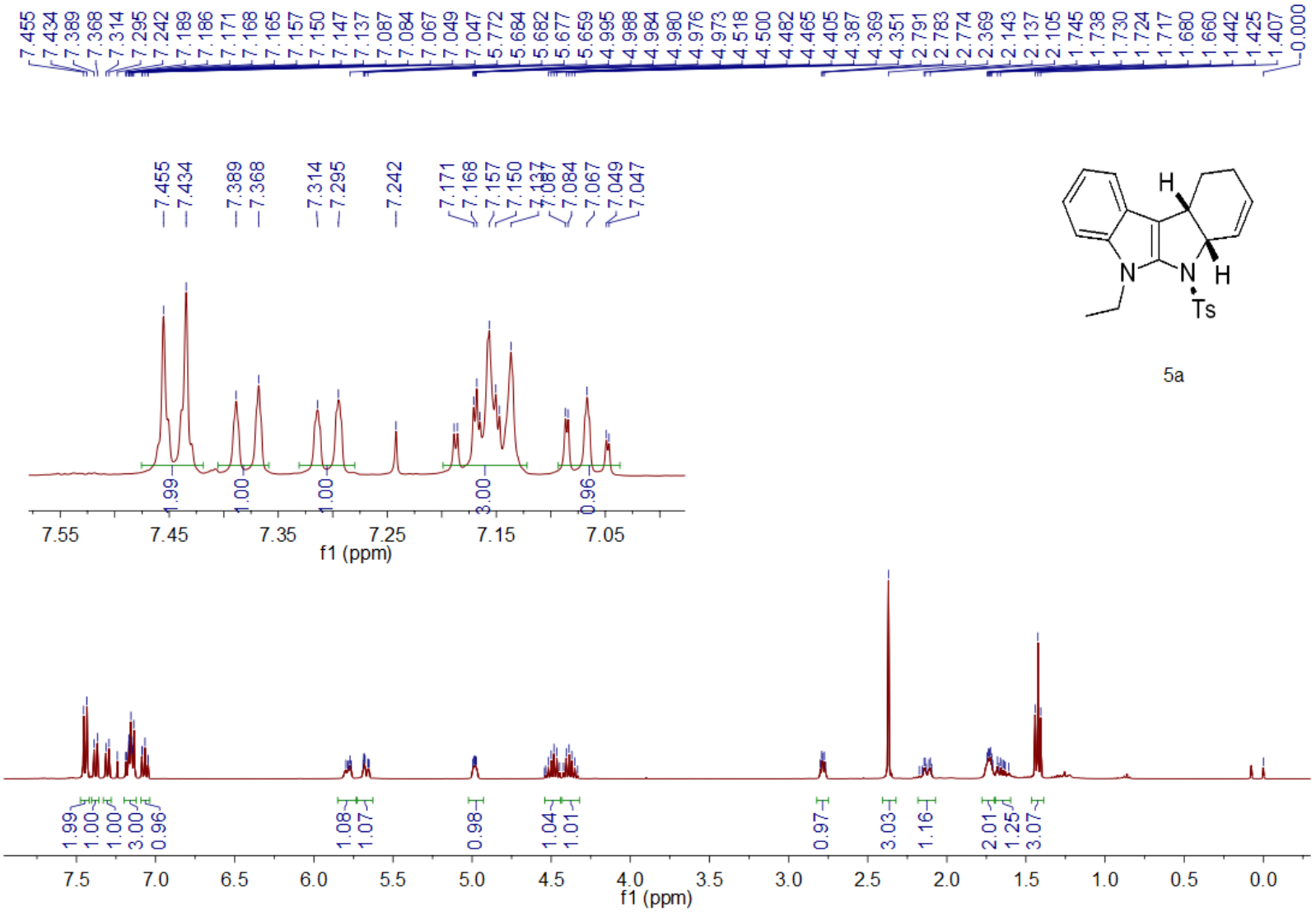
135.269
132.890
132.735
128.319
128.011
126.917
125.371
122.353
122.215
119.095
118.076
109.740
107.662
77.318
77.000
76.682
54.682
31.498
29.601
21.670
21.052
f1 (ppm)
f1 (ppm)
3z


8.789
7.356
7.352
7.339
7.335
7.320
7.301
7.300
7.254
7.236
7.234
7.130
7.127
7.112
7.110
7.092
7.089
7.063
7.049
7.046
7.043
7.029
7.027
7.012
7.009
4.249
3.194
2.274
1.548
1.394
0.000
1.80
0.91
0.98
1.00
2.80
f1 (ppm)
0.90
2.00
1.95
3.00
3.13
2.84
Ts
3A


143.662
135.814
133.259
132.460
132.425
129.116
127.123
124.299
121.869
119.451
117.410
110.654
101.707
77.318
77.000
76.683
54.080
30.596
21.475
21.190
20.809
N
Ts
N
H
3A
f1 (ppm)
f1 (ppm)


7.514
7.502
7.469
7.317
7.303
7.196
7.186
6.166
5.552
5.534
5.523
5.412
5.393
3.765
3.019
3.008
2.985
2.974
2.416
1.922
1.888
0.210
0.000
Br
N
N
OTMS
Ts
3B
1.93
0.92
1.00
2.95
1.10
1.17
1.22
3.32
1.26
3.12
1.22
8.98
f1 (ppm)


144.132
135.876
134.188
130.553
129.343
128.620
128.035
127.627
126.555
124.994
120.926
112.317
111.338
109.805
79.364
77.212
77.000
76.788
29.814
22.978
21.611
0.128
Br
N
N
OTMS
Ts
3B
f1 (ppm)
f1 (ppm)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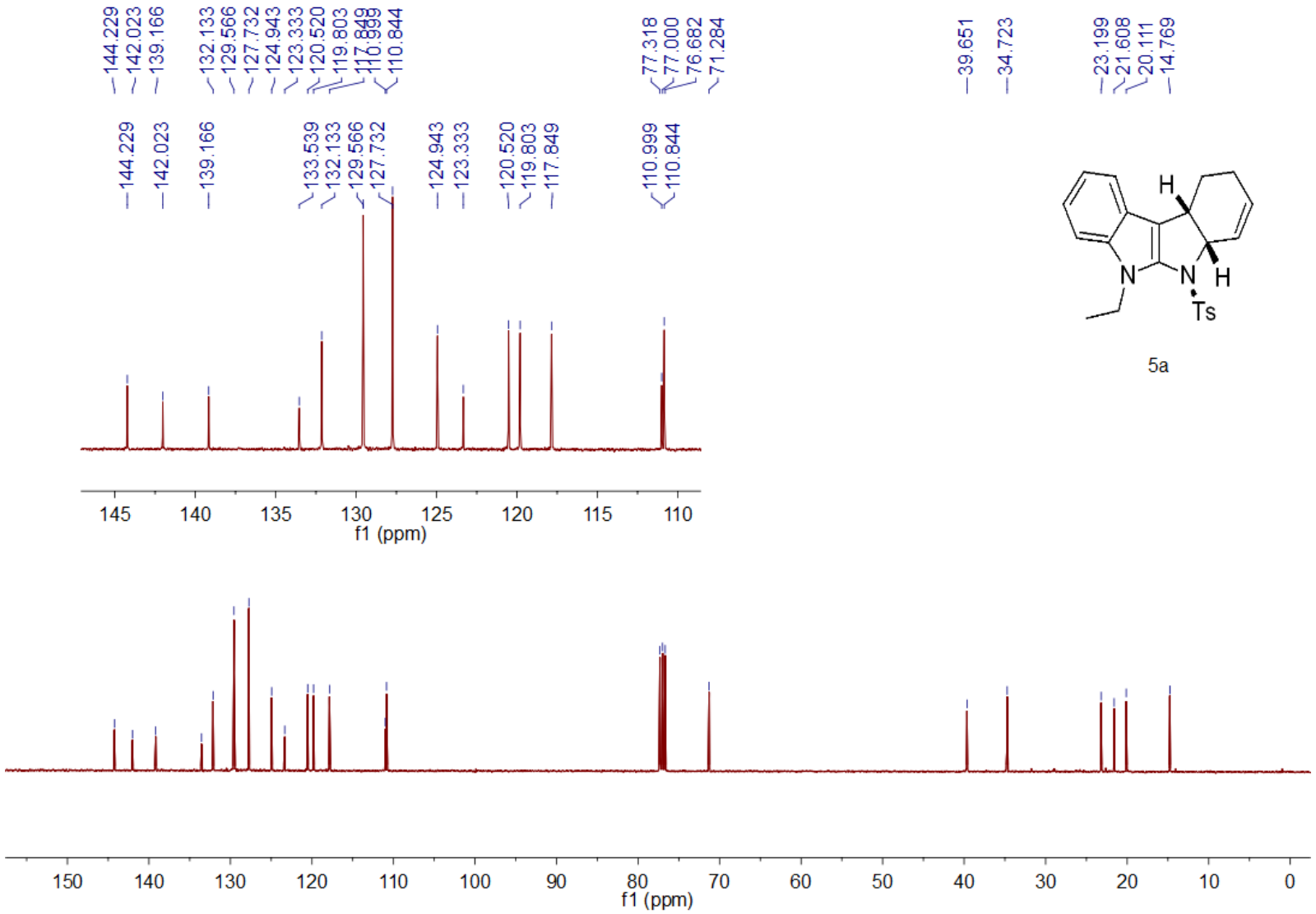
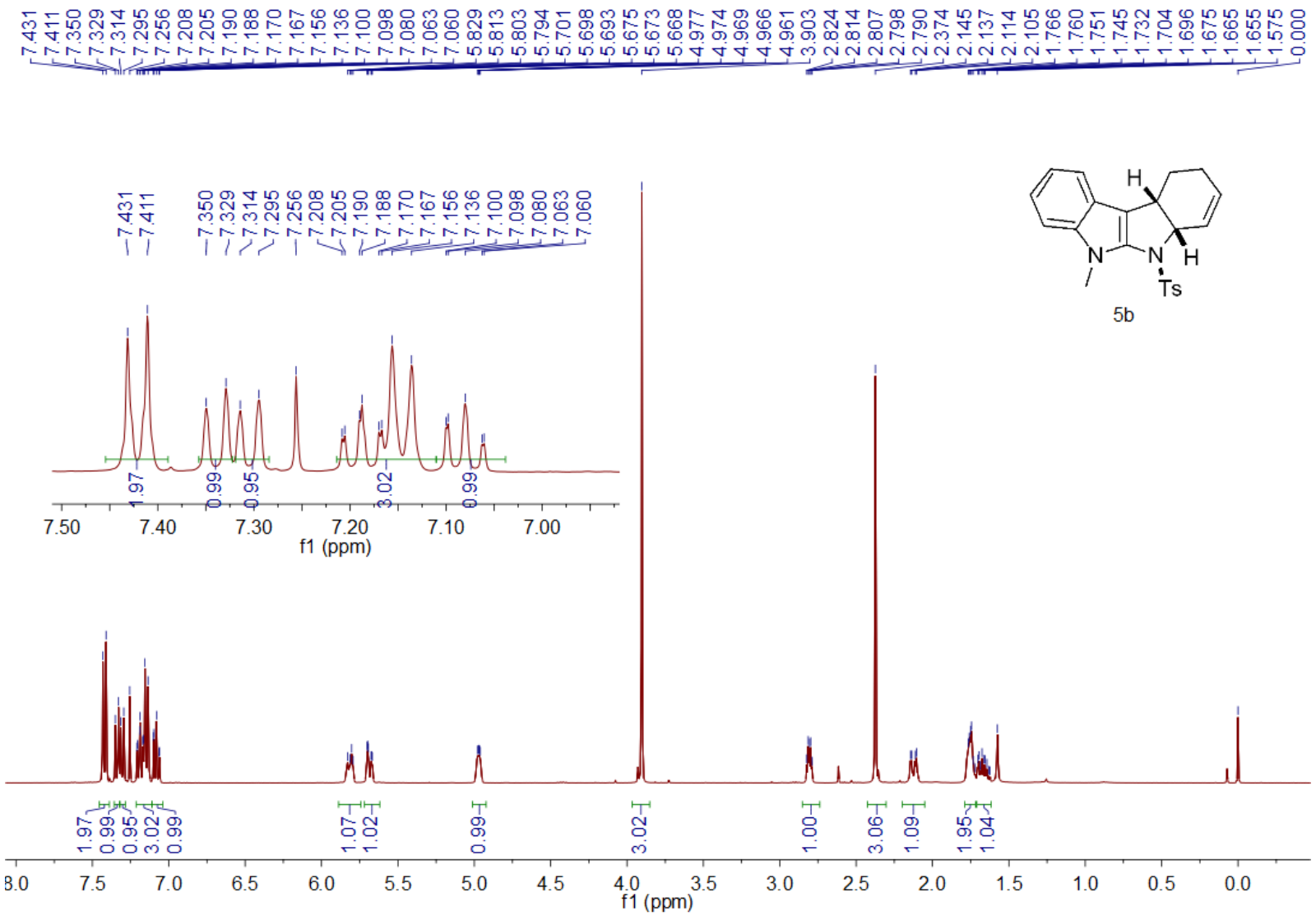
7.434
7.389
7.368
7.314
7.295
7.242
7.189
7.186
7.171
7.168
7.165
7.157
7.150
7.147
7.137
7.087
7.084
7.067
7.049
7.047
5.772
5.684
5.682
5.677
5.659
4.995
4.988
4.984
4.980
4.976
4.973
4.518
4.500
4.482
4.465
4.405
4.387
4.369
4.351
2.791
2.783
2.774
2.369
2.143
2.137
2.105
1.745
1.738
1.730
1.724
1.717
1.680
1.660
1.442
1.425
1.407
0.000
1.99
1.00
1.00
3.00
0.96
7.55
7.45
7.35
7.25
7.15
7.05
f1 (ppm)
1.08
1.07
0.98
1.04
1.01
0.97
3.03
1.16
2.01
1.25
3.07
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)
H
N
N
H
Ts
5a


144.229
142.023
139.166
132.133
129.566
127.732
124.943
123.333
120.520
119.803
117.849
110.999
110.844
77.318
77.000
76.682
71.284
39.651
34.723
23.199
21.608
20.111
14.769
f1 (ppm)
H
N
N
H
Ts

5a


7.431
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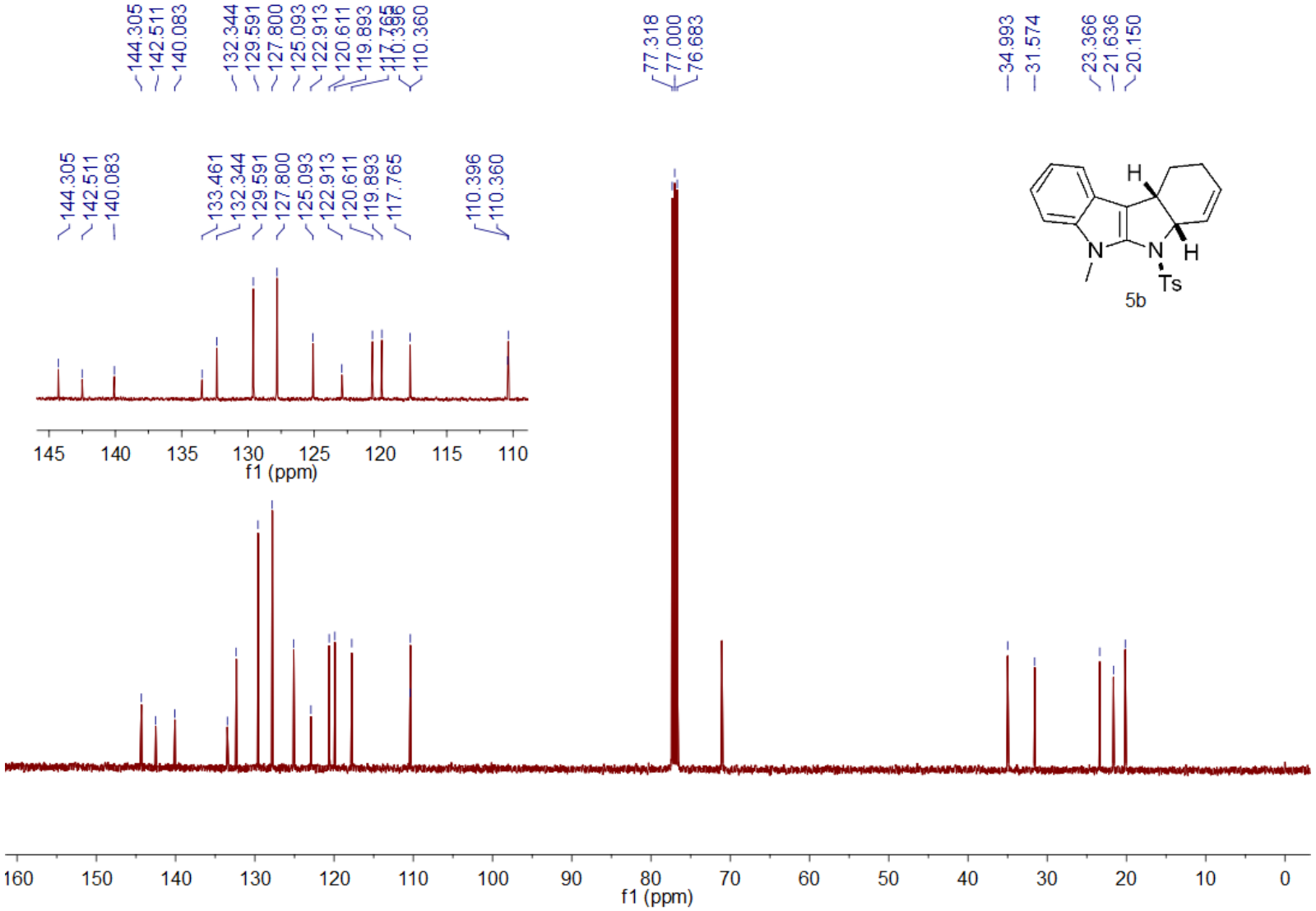
7.350
7.329
7.314
7.295
7.256
7.208
7.205
7.190
7.188
7.170
7.167
7.156
7.136
7.100
7.098
7.080
7.063
7.060
5.829
5.813
5.803
5.794
5.701
5.698
5.693
5.675
5.673
5.668
4.977
4.974
4.969
4.966
4.961
3.903
2.824
2.814
2.807
2.798
2.790
2.374
2.145
2.137
2.114
2.105
1.766
1.760
1.751
1.745
1.732
1.704
1.696
1.675
1.665
1.655
1.575
0.000
H
N
N
H
Ts
5b
1.97
0.99
0.95
3.02
0.99
1.07
1.02
0.99
3.02
1.00
3.06
1.09
1.95
1.04
f1 (ppm)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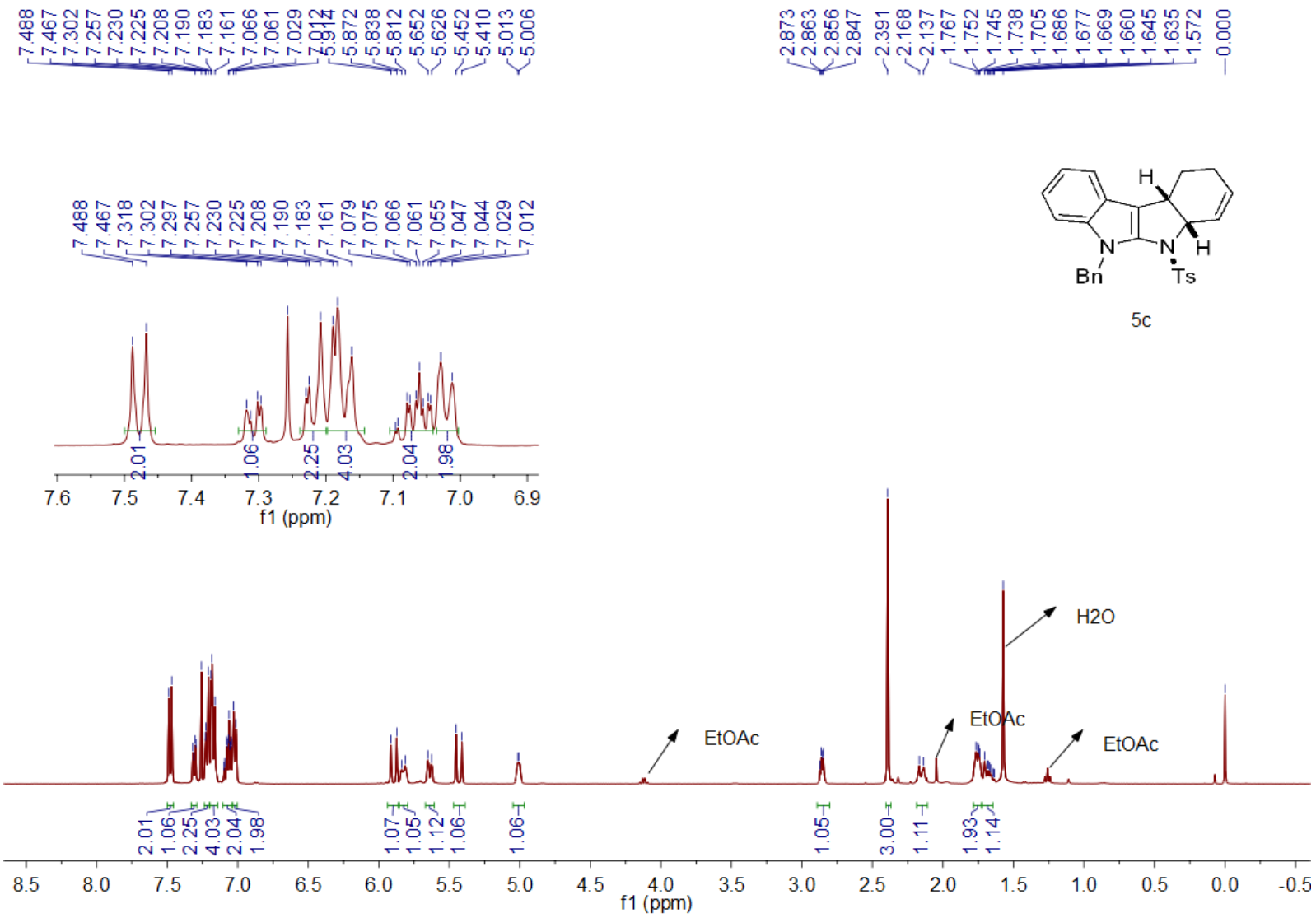
127.800
125.093
122.913
120.611
119.893
117.765
110.396
110.360
77.318
77.000
76.683
34.993
31.574
23.366
21.636
20.150
133.461
f1 (ppm)
H
N
N
H
Ts
5b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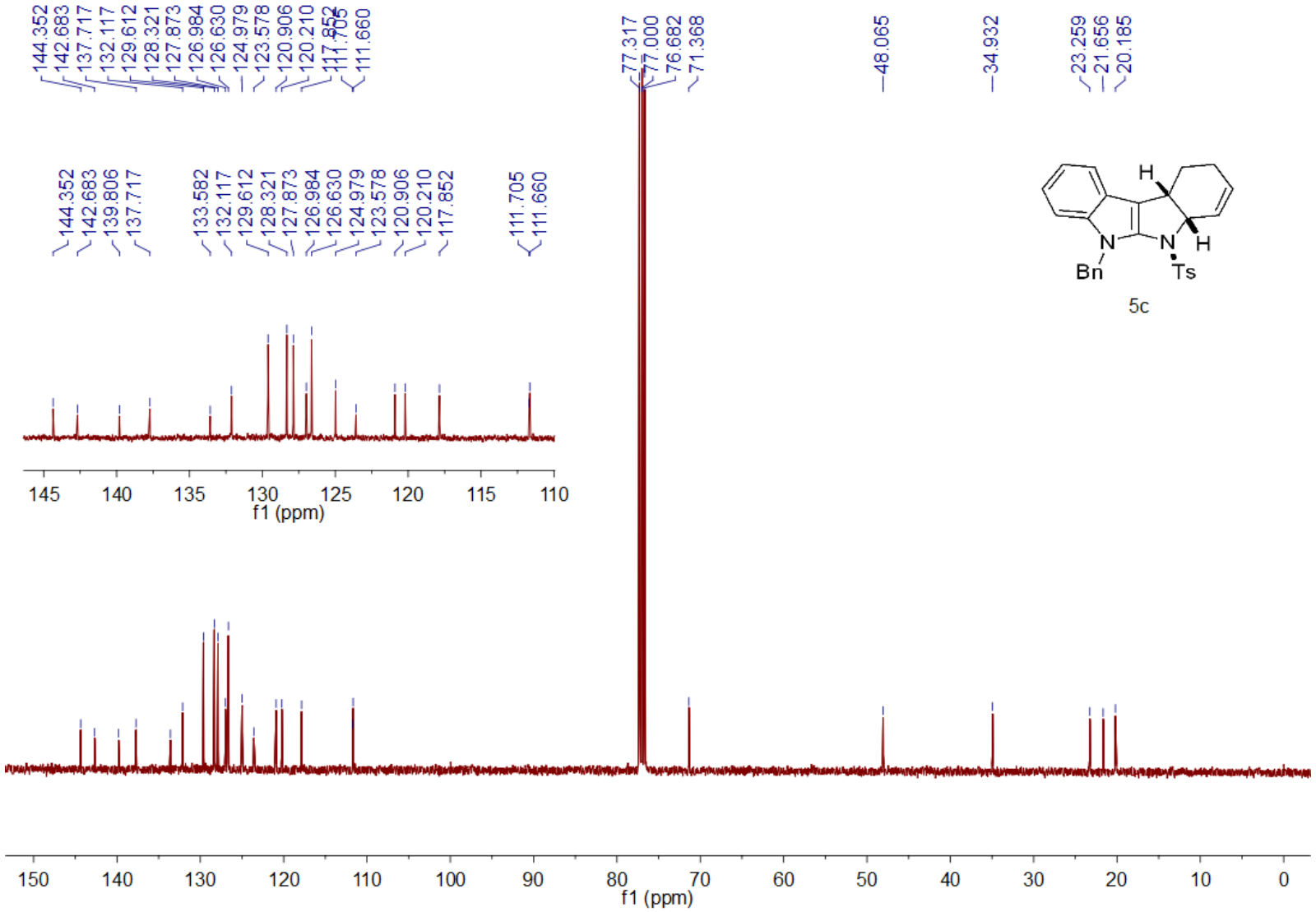
7.257
7.230
7.225
7.208
7.190
7.183
7.161
7.066
7.061
7.029
7.012
5.872
5.838
5.812
5.652
5.626
5.452
5.410
5.013
5.006
2.873
2.863
2.856
2.847
2.391
2.168
2.137
1.767
1.752
1.745
1.738
1.705
1.686
1.677
1.669
1.660
1.645
1.635
1.572
0.000
H
N
N
H
Bn
Ts
5c
2.01
1.06
2.25
4.03
2.04
1.98
f1 (ppm)
H2O
EtOAc
EtOAc
EtOAc
1.07
1.05
1.12
1.06
1.06
1.05
3.00
1.11
1.93
1.14
f1 (ppm)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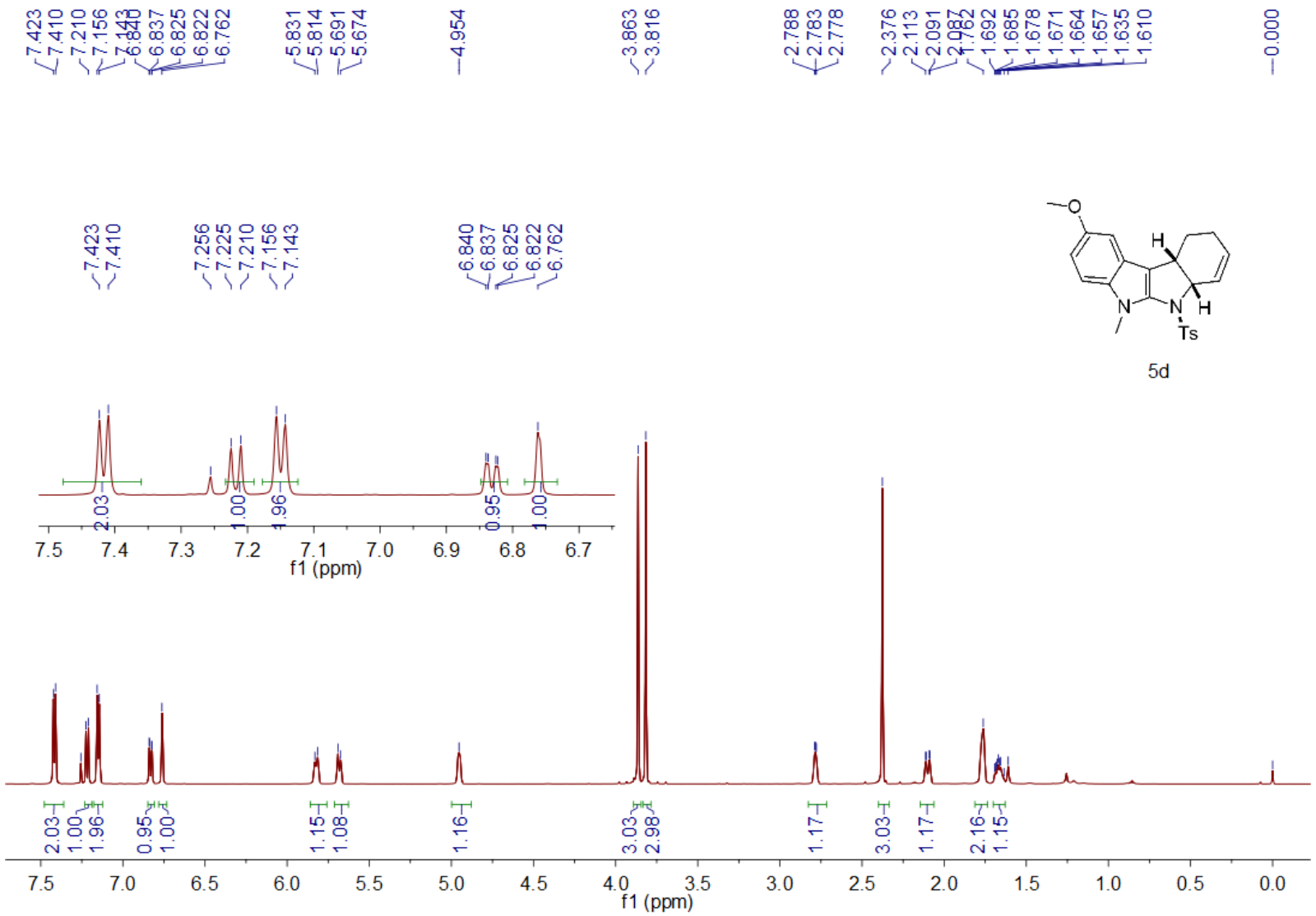
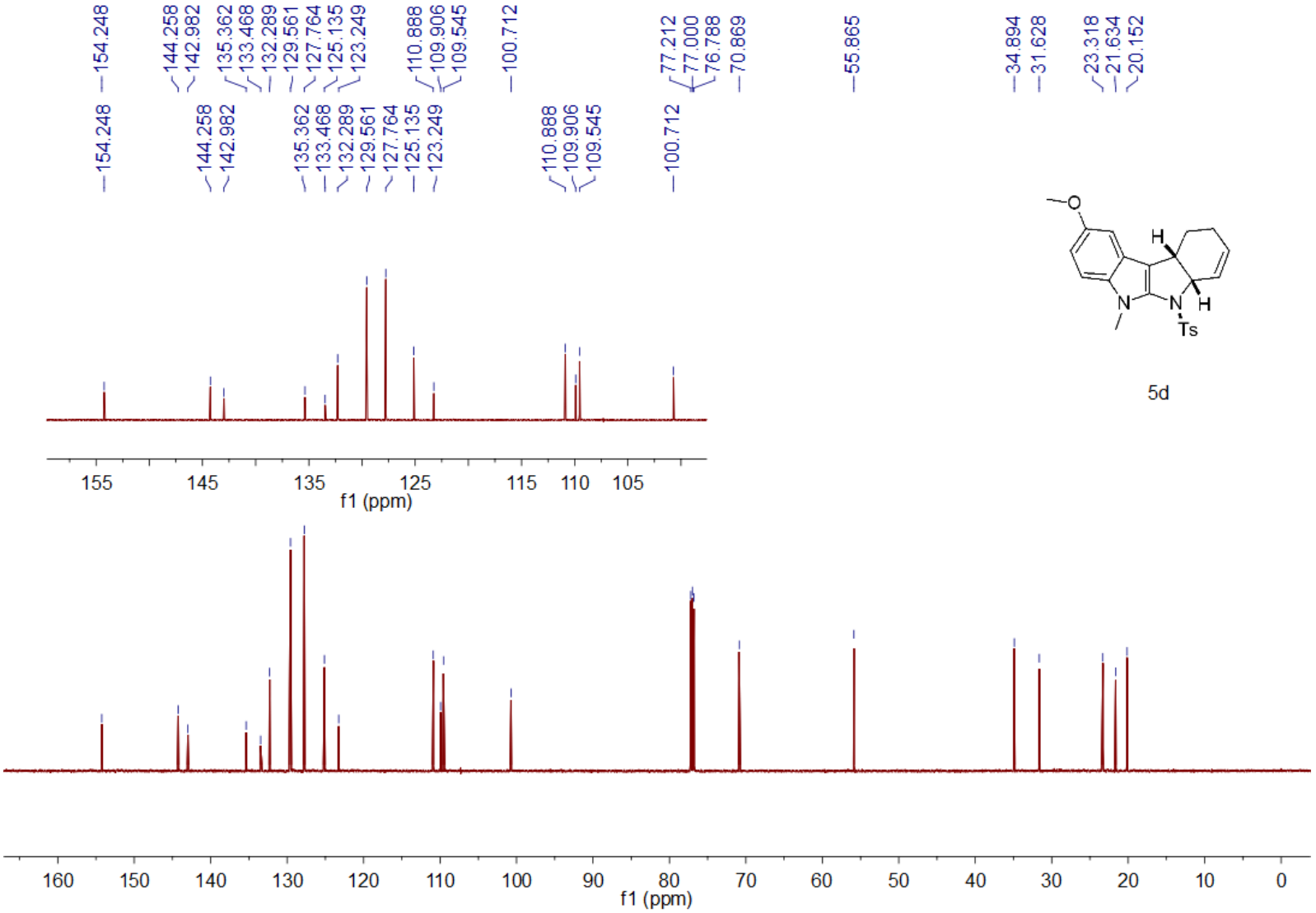
128.321
127.873
126.984
126.630
124.979
123.578
120.906
120.210
117.852
111.705
111.660
77.317
77.000
76.682
71.368
48.065
34.932
23.259
21.656
20.185
144.352
142.683
139.806
137.717
133.582
132.117
129.612
128.321
127.873
126.984
126.630
124.979
123.578
120.906
120.210
117.852
111.705
111.660
145 140 135 130 125 120 115 110
f1 (ppm)
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
H
N
N
H
Bn
Ts
5c


7.423
7.410
7.210
7.156
7.143
6.840
6.837
6.825
6.822
6.762
5.831
5.814
5.691
5.674
4.954
3.863
3.816
2.788
2.783
2.778
2.376
2.113
2.091
2.087
1.692
1.685
1.678
1.671
1.664
1.657
1.635
1.610
0.000
7.423
7.410
7.256
7.225
7.210
7.156
7.143
6.840
6.837
6.825
6.822
6.762
O
H
N
N
H
Ts
5d
2.03
1.00
1.96
0.95
1.00
1.15
1.08
1.16
3.03
2.98
1.17
3.03
1.17
2.16
1.15
f1 (ppm)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154.248
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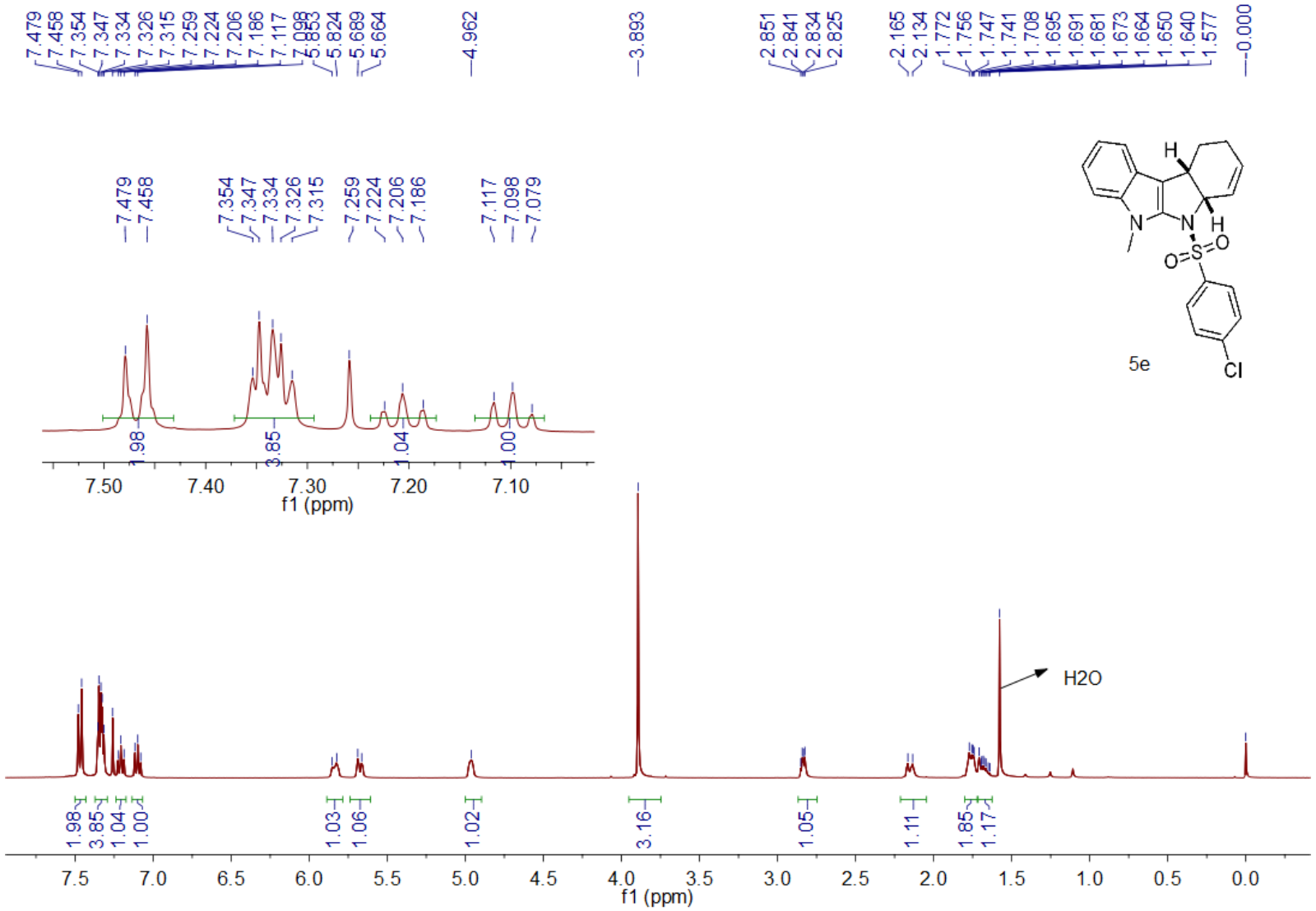
144.258
142.982
135.362
133.468
132.289
129.561
127.764
125.135
123.249
135.362
133.468
132.289
129.561
127.764
125.135
123.249
110.888
109.906
109.545
110.888
109.906
109.545
100.712
100.712
77.212
77.000
76.788
70.869
55.865
34.894
31.628
23.318
21.634
20.152
O
H
N
N
H
Ts
155
145
135
125
115
110
105
f1 (ppm)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

5d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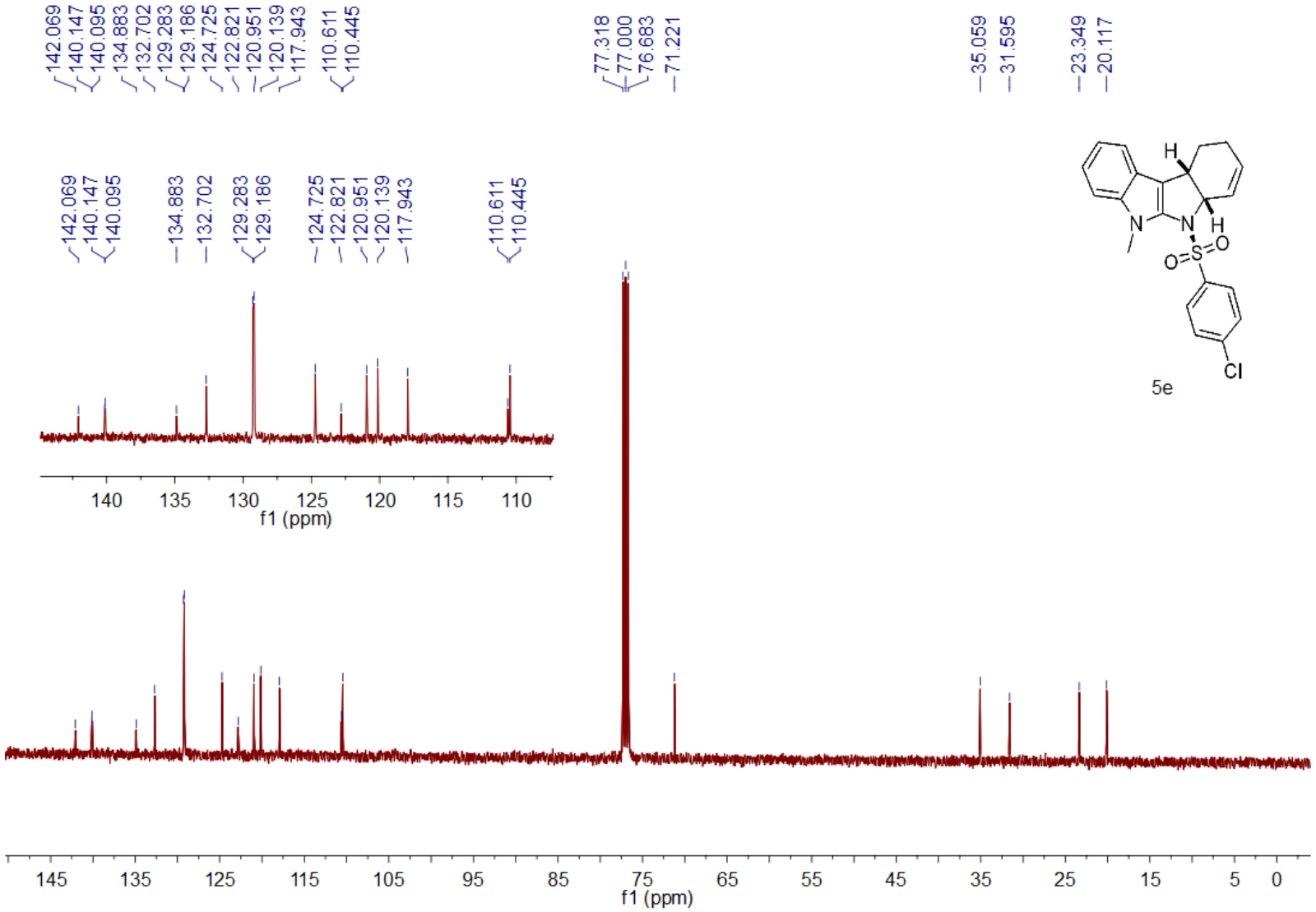
7.326
7.315
7.259
7.224
7.206
7.186
7.117
7.098
5.853
5.824
5.689
5.664
4.962
3.893
2.851
2.841
2.834
2.825
2.165
2.134
1.772
1.756
1.747
1.741
1.708
1.695
1.691
1.681
1.673
1.664
1.650
1.640
1.577
0.000
H
N
N
H
O=S=O
Cl
5e
1.98
3.85
1.04
1.00
7.50
7.40
7.30
7.20
7.10
f1 (ppm)
H2O
1.03
1.06
1.02
3.16
1.05
1.11
1.85
1.17
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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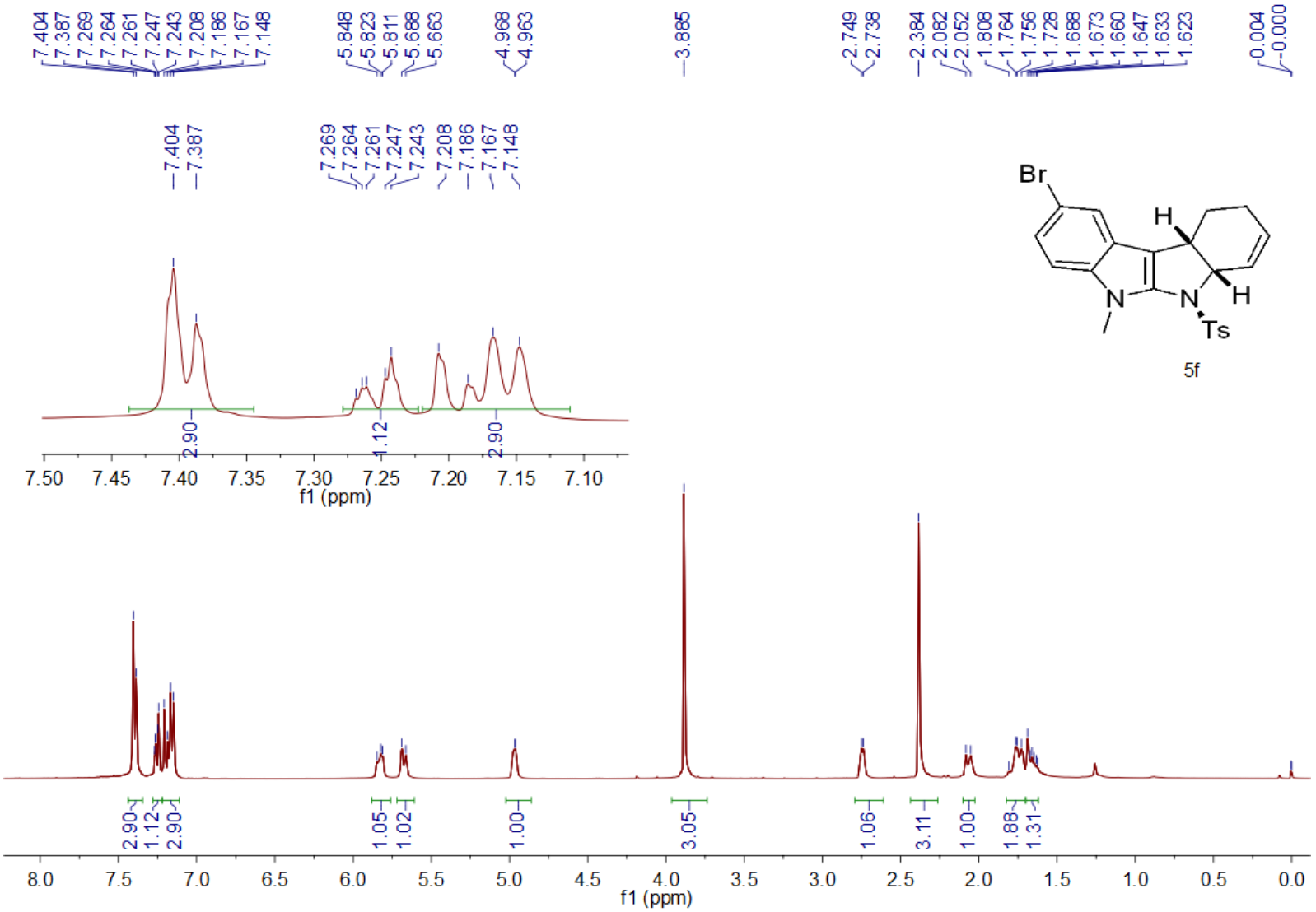
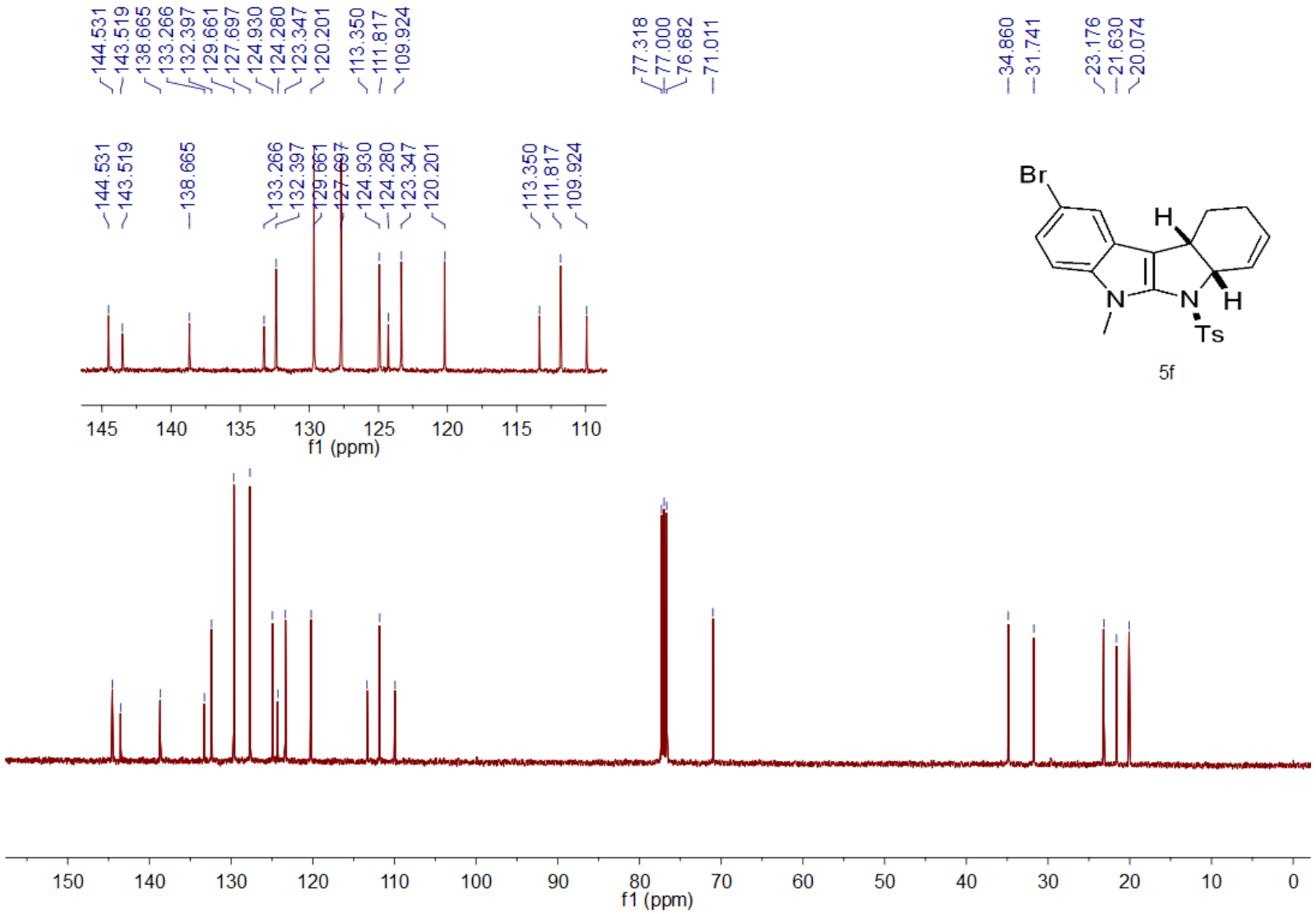
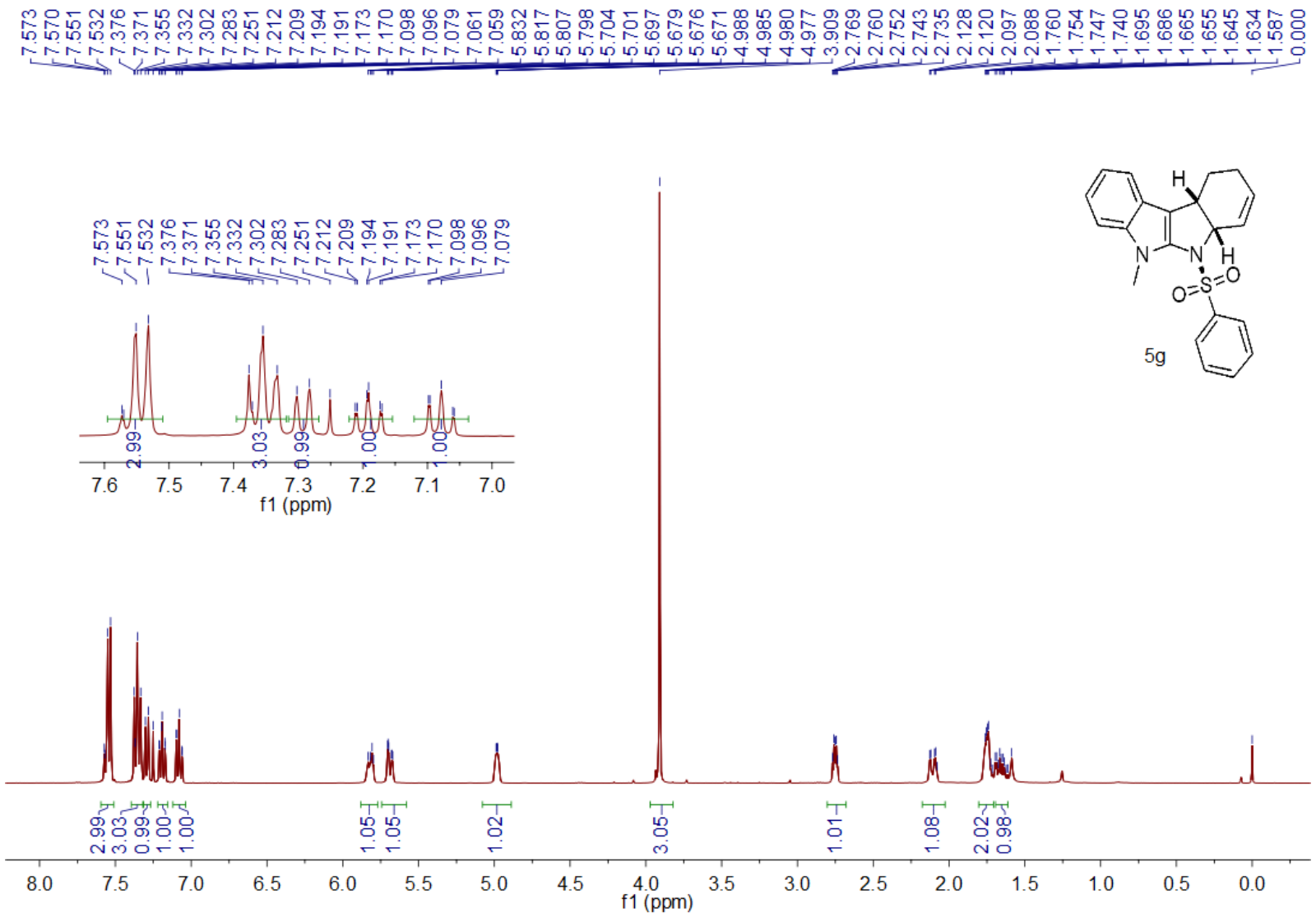
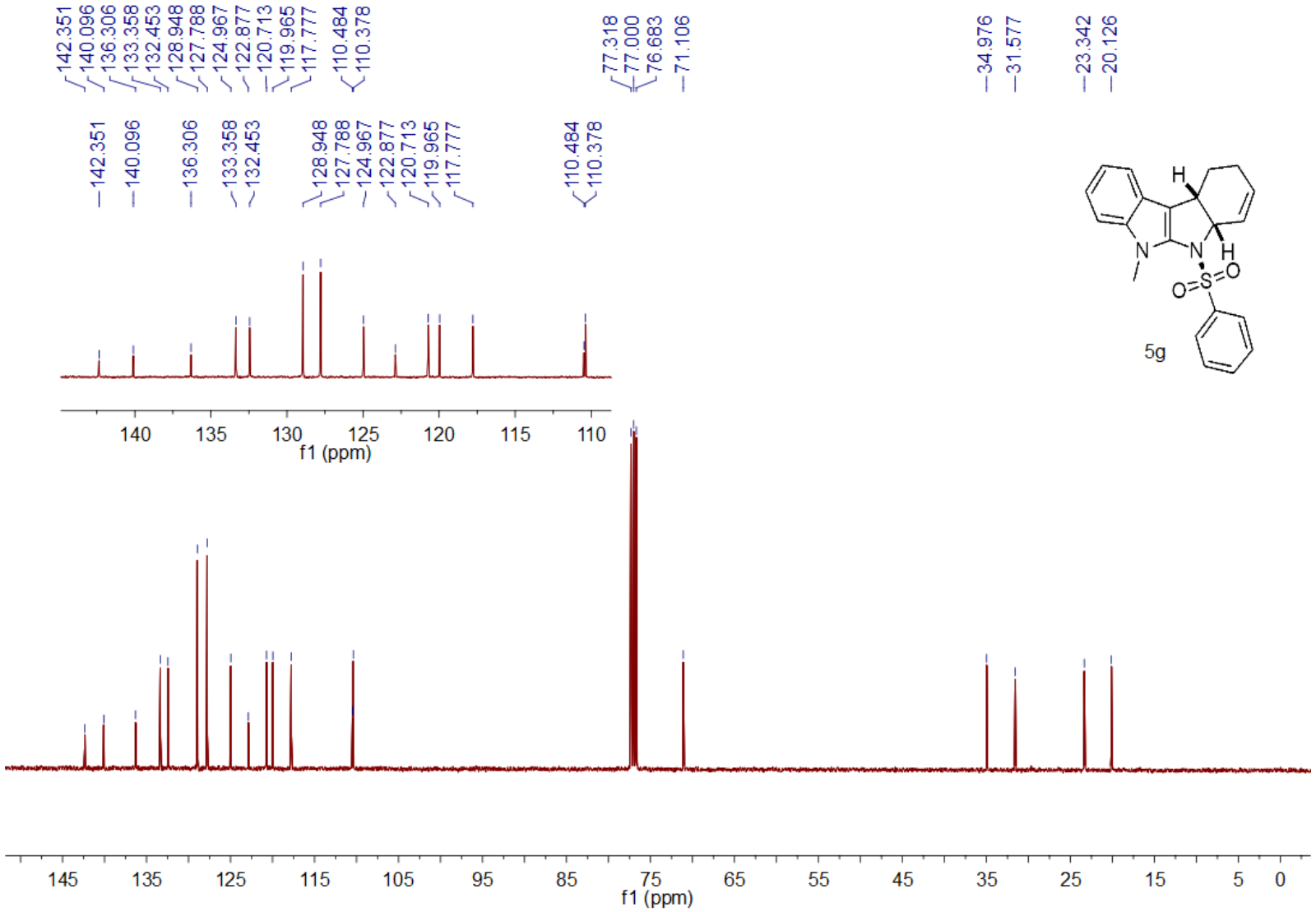
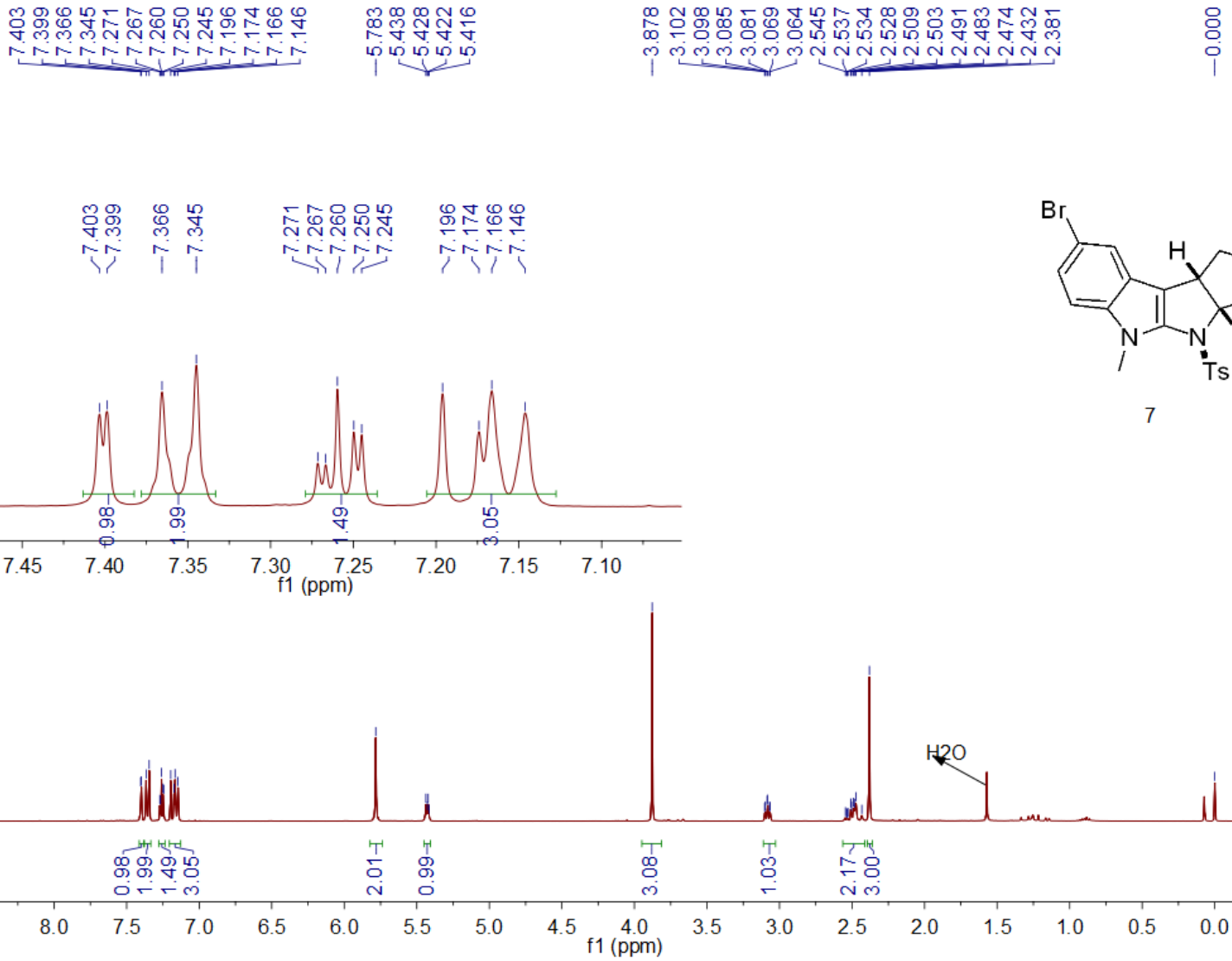
140.095
134.883
132.702
129.283
129.186
124.725
122.821
120.951
120.139
117.943
110.611
110.445
77.318
77.000
76.683
71.221
35.059
31.595
23.349
20.117
f1 (ppm)
5e

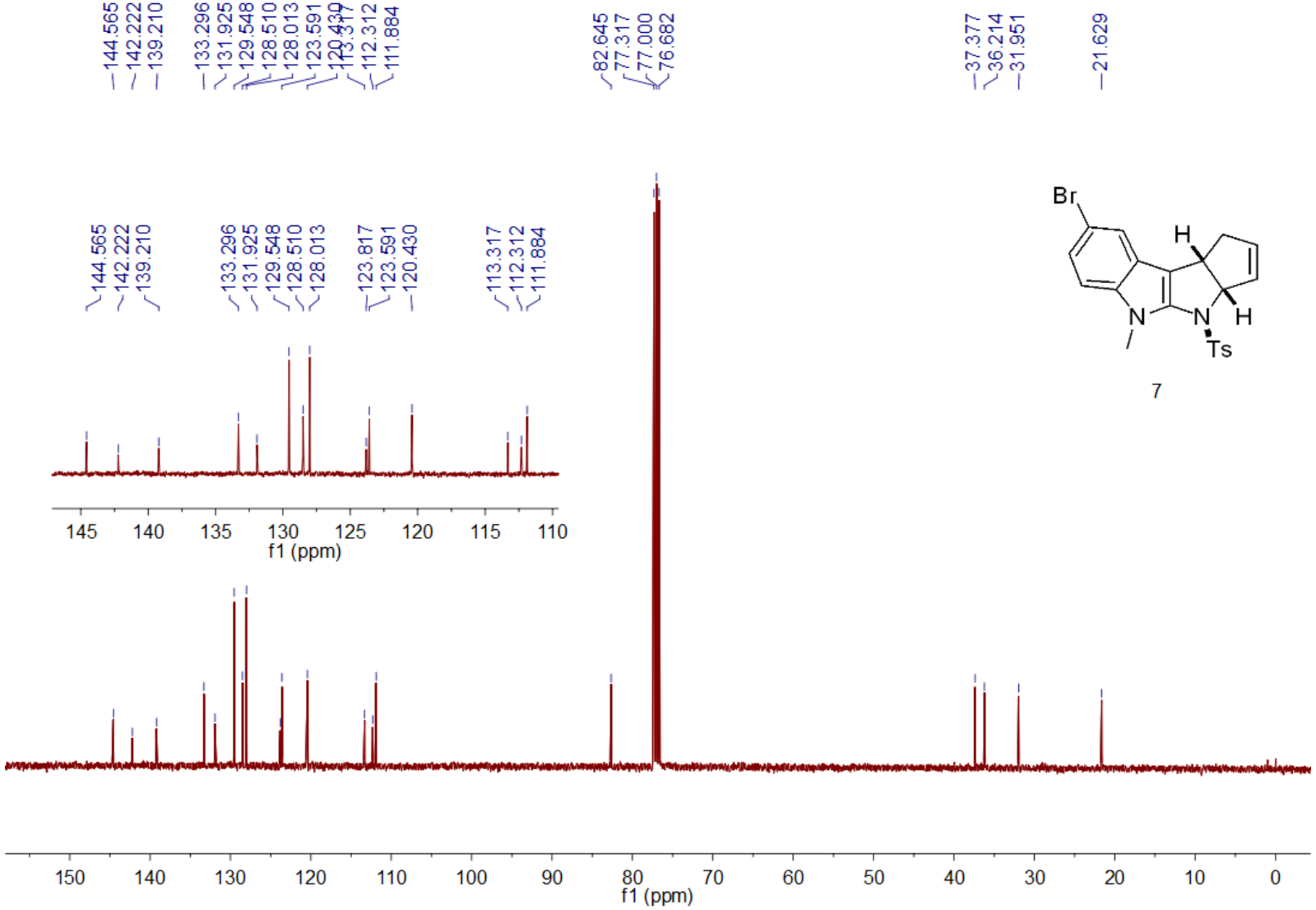

7.404
7.387
7.269
7.264
7.261
7.247
7.243
7.208
7.186
7.167
7.148
5.848
5.823
5.811
5.688
5.663
4.968
4.963
3.885
2.749
2.738
2.384
2.082
2.052
1.808
1.764
1.756
1.728
1.688
1.673
1.660
1.647
1.633
1.623
0.004
-0.000
2.90
1.12
2.90
1.05
1.02
1.00
3.05
1.06
3.11
1.00
1.88
1.31
f1 (ppm)
Br
H
N
N
H
Ts
5f


144.531
143.519
138.665
133.266
132.397
129.661
127.697
124.930
124.280
123.347
120.201
113.350
111.817
109.924
77.318
77.000
76.682
71.011
34.860
31.741
23.176
21.630
20.074
f1 (ppm)
Br
H
N
N
H
Ts
5f

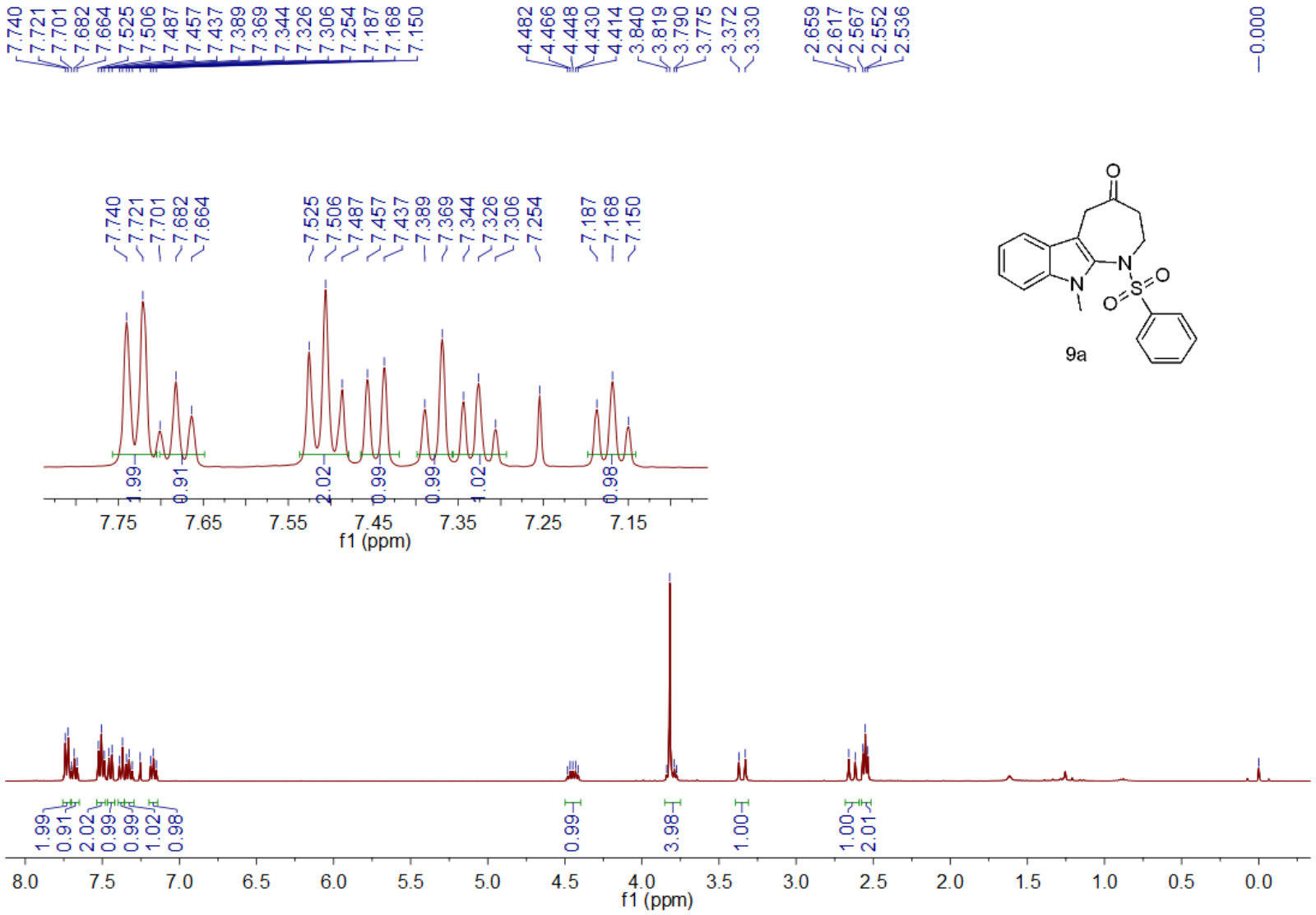

7.573
7.570
7.551
7.532
7.376
7.371
7.355
7.332
7.302
7.283
7.251
7.212
7.209
7.194
7.191
7.173
7.170
7.098
7.096
7.079
7.061
7.059
5.832
5.817
5.807
5.798
5.704
5.701
5.697
5.679
5.676
5.671
4.988
4.985
4.980
4.977
3.909
2.769
2.760
2.752
2.743
2.735
2.128
2.120
2.097
2.088
1.760
1.754
1.747
1.740
1.695
1.686
1.665
1.655
1.645
1.634
1.587
0.000
2.99
3.03
0.99
1.00
1.00
1.05
1.05
1.02
3.05
1.01
1.08
2.02
0.98
f1 (ppm)
5g

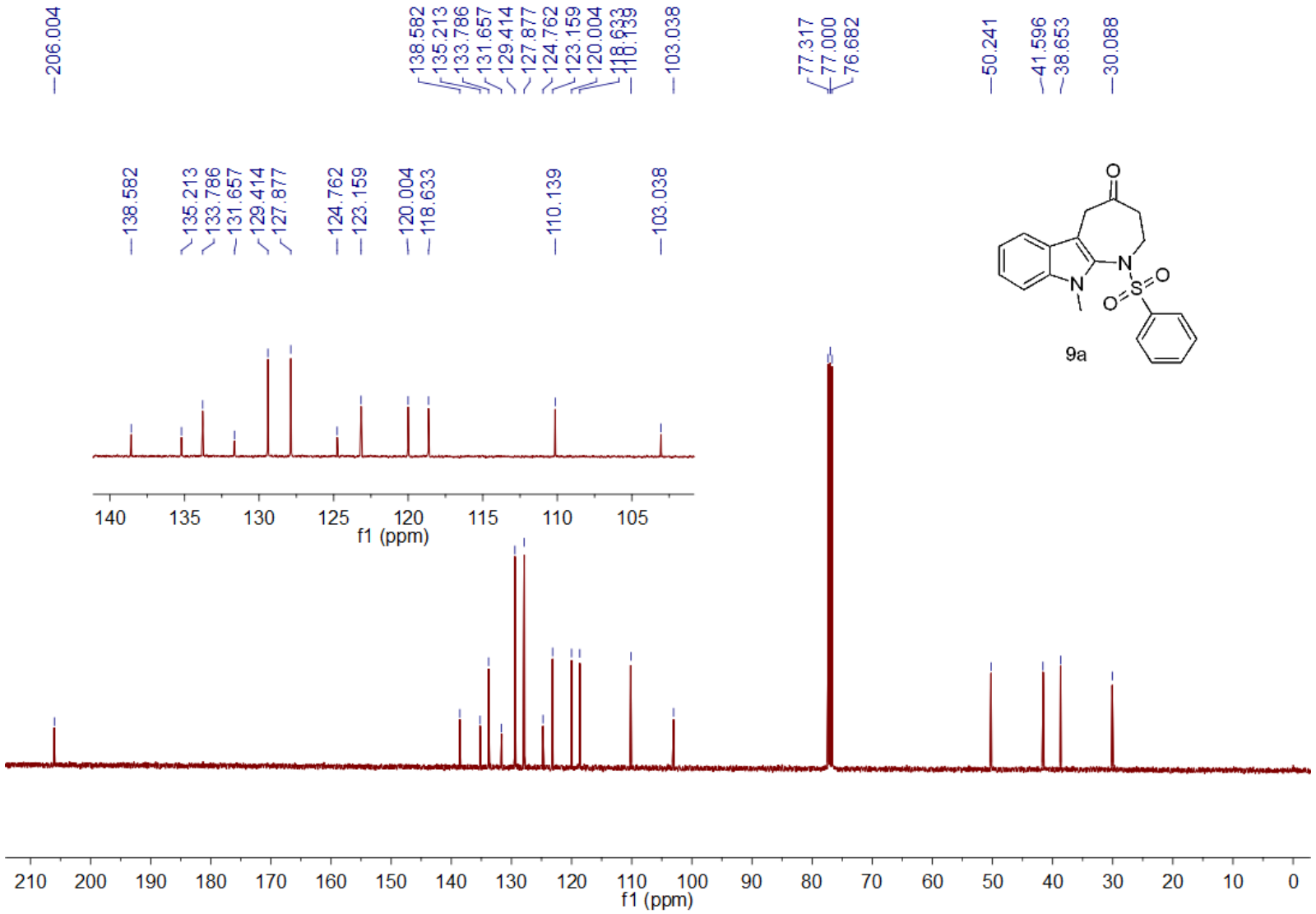

142.351
140.096
136.306
133.358
132.453
128.948
127.788
124.967
122.877
120.713
119.965
117.777
110.484
110.378
77.318
77.000
76.683
71.106
34.976
31.577
23.342
20.126
f1 (ppm)
5g

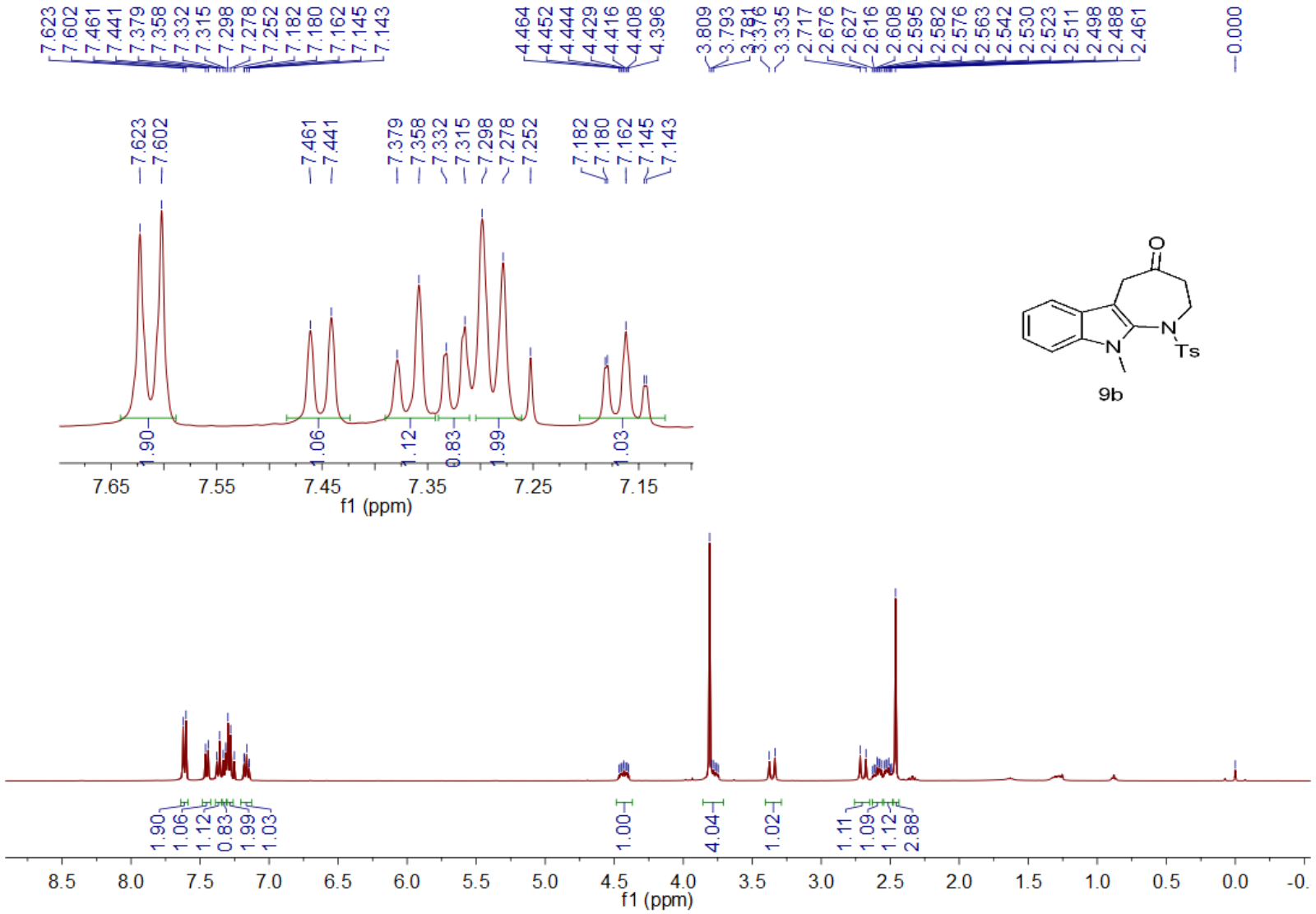

7.403
7.399
7.366
7.345
7.271
7.267
7.260
7.250
7.245
7.196
7.174
7.166
7.146
5.783
5.438
5.428
5.422
5.416
3.878
3.102
3.098
3.085
3.081
3.069
3.064
2.545
2.537
2.534
2.528
2.509
2.503
2.491
2.483
2.474
2.432
2.381
0.000
Br
H
N
N
H
Ts
7
0.98
1.99
1.49
3.05
f1 (ppm)
H2O
2.01
0.99
3.08
1.03
2.17
3.00
f1 (ppm)

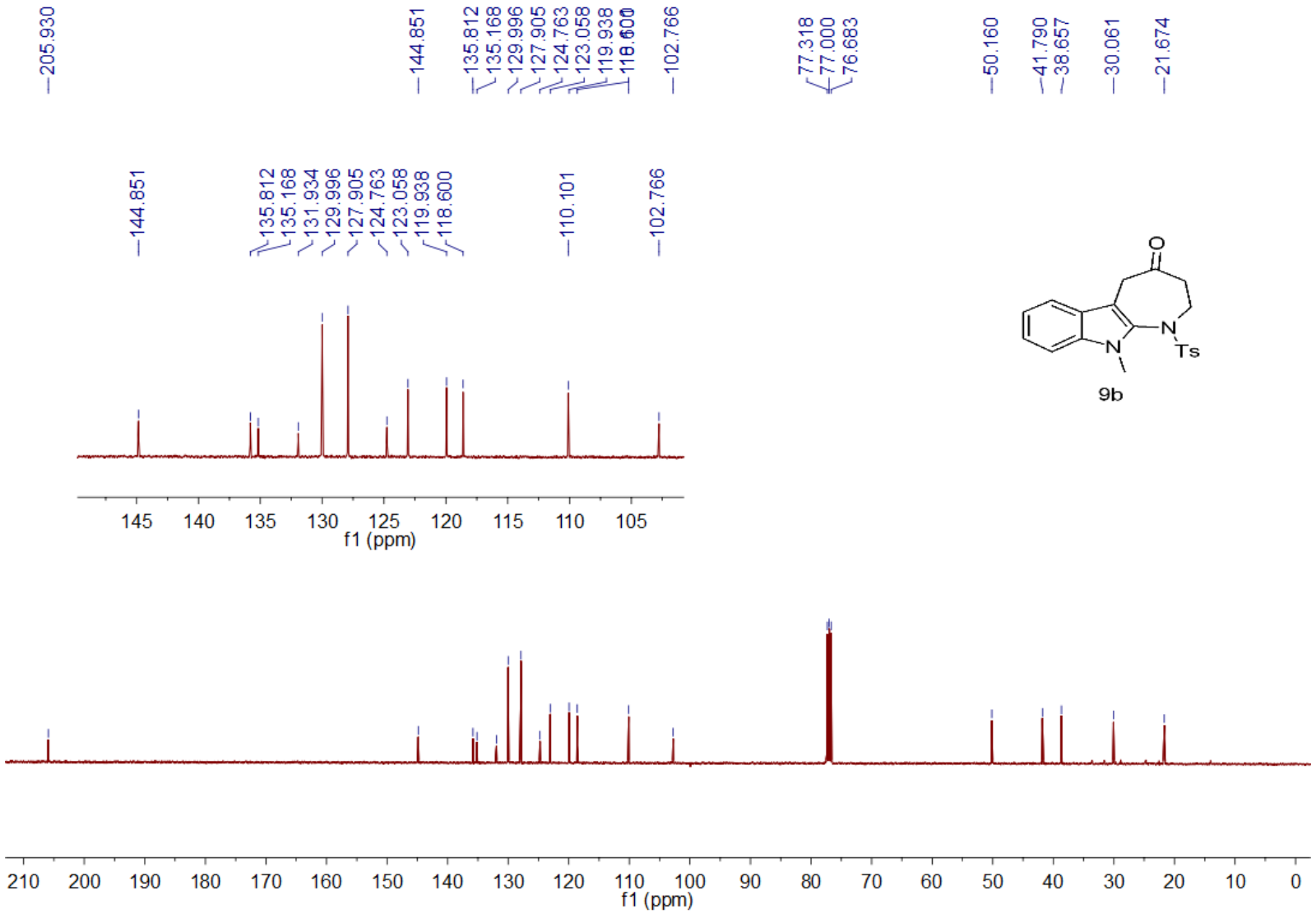

144.565
142.222
139.210
133.296
131.925
129.548
128.510
128.013
123.591
120.430
113.317
112.312
111.884
82.645
77.317
77.000
76.682
37.377
36.214
31.951
21.629
f1 (ppm)
Br
H
N
N
H
Ts

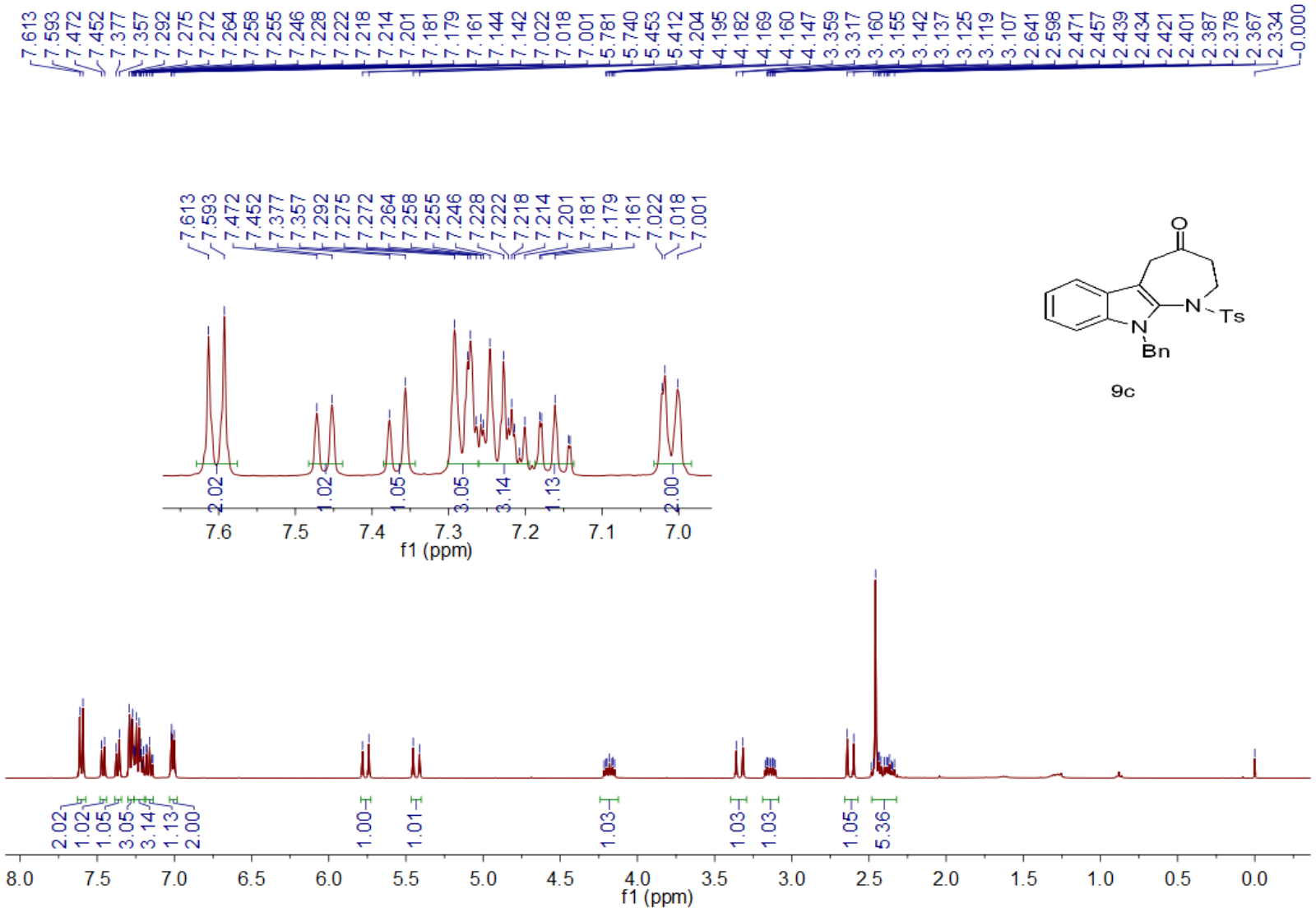
7

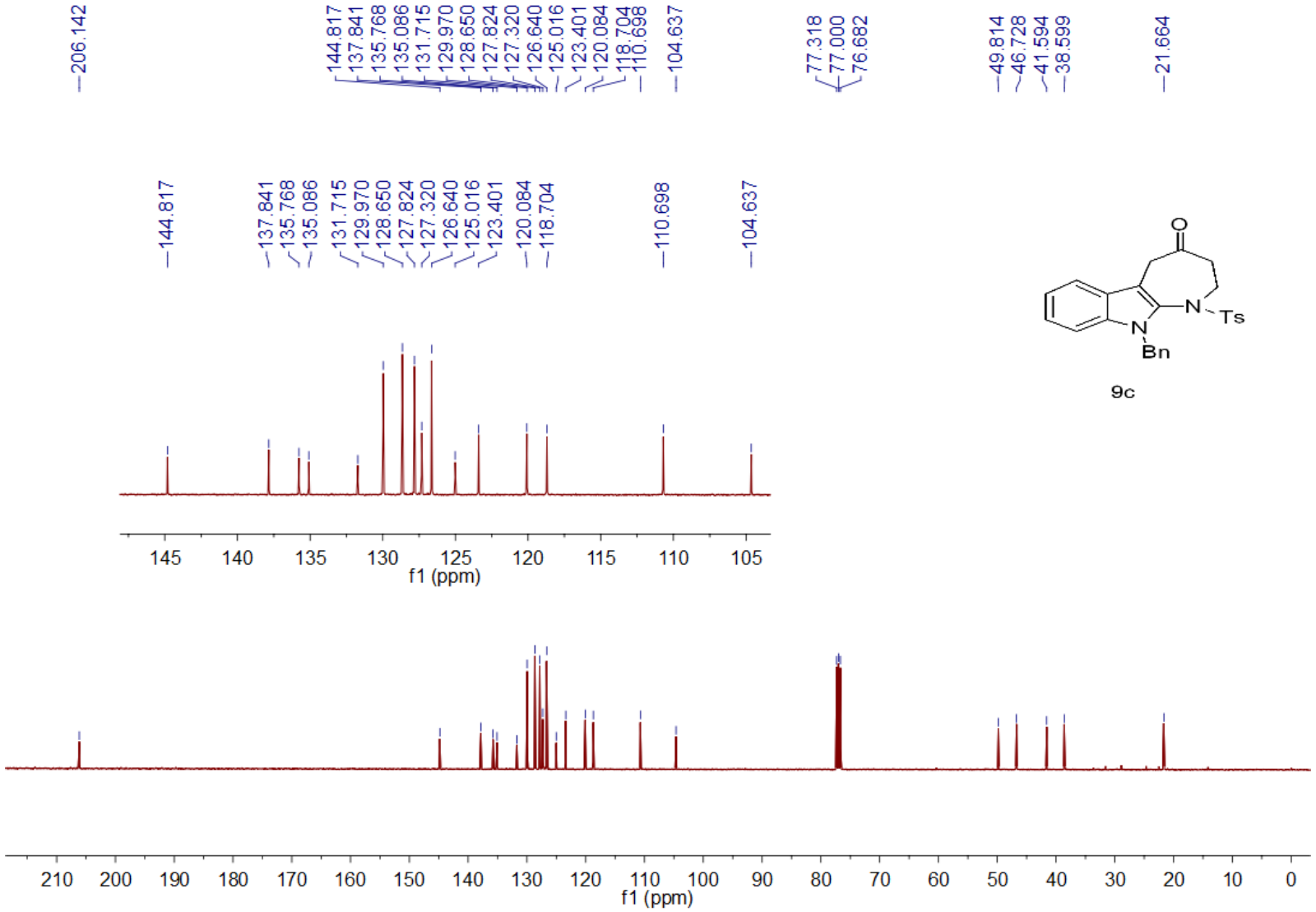

7.740
7.721
7.701
7.682
7.664
7.525
7.506
7.487
7.457
7.437
7.389
7.369
7.344
7.326
7.306
7.254
7.187
7.168
7.150
4.482
4.466
4.448
4.430
4.414
3.840
3.819
3.790
3.775
3.372
3.330
2.659
2.617
2.567
2.552
2.536
0.000
1.99
0.91
2.02
0.99
0.99
1.02
0.98
7.75
7.65
7.55
7.45
7.35
7.25
7.15
f1 (ppm)
0.99
3.98
1.00
1.00
2.01
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)
O
N
N
S
O
O
9a

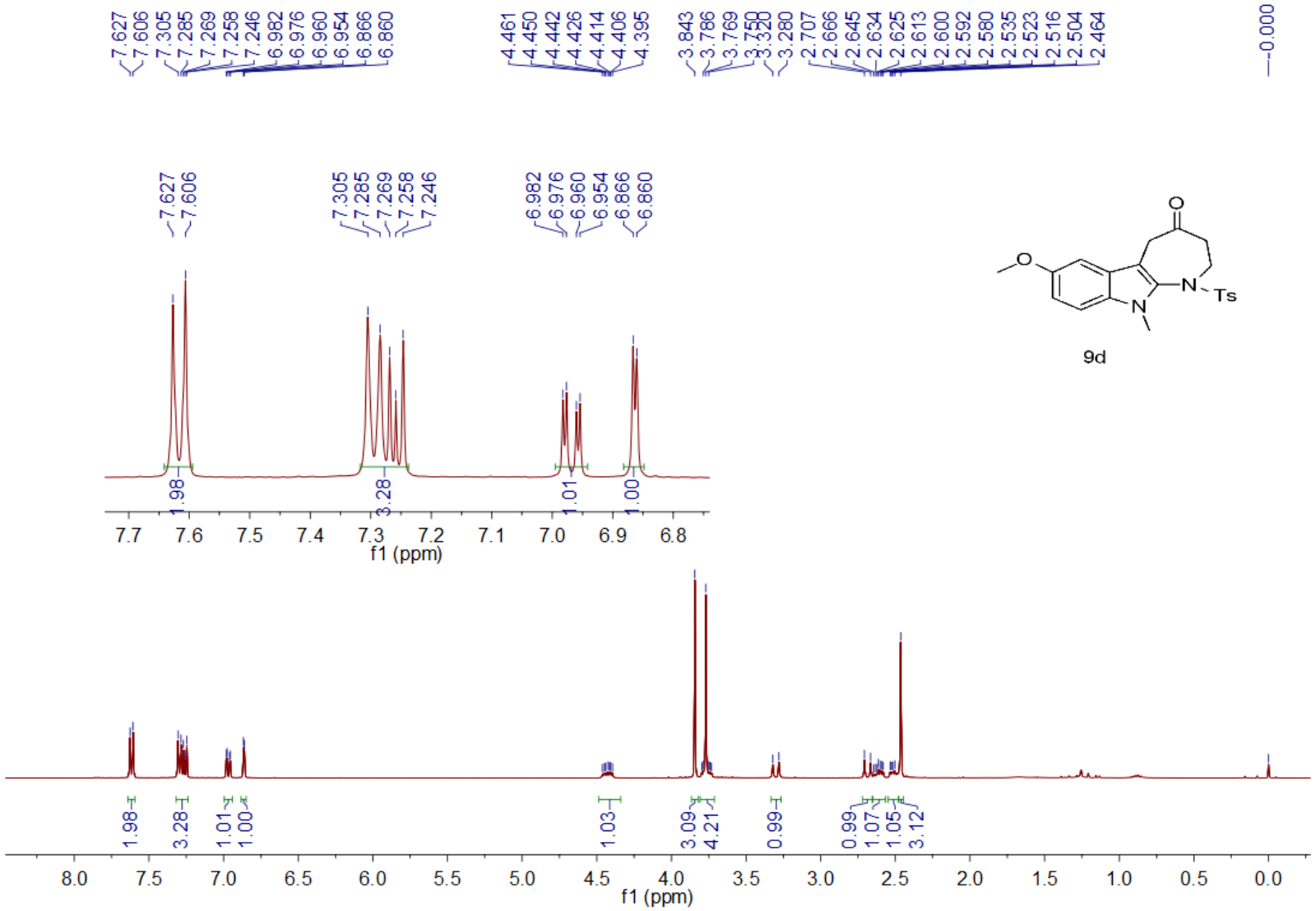

206.004
138.582
135.213
133.786
131.657
129.414
127.877
124.762
123.159
120.004
118.633
110.139
103.038
77.317
77.000
76.682
50.241
41.596
38.653
30.088
f1 (ppm)
9a

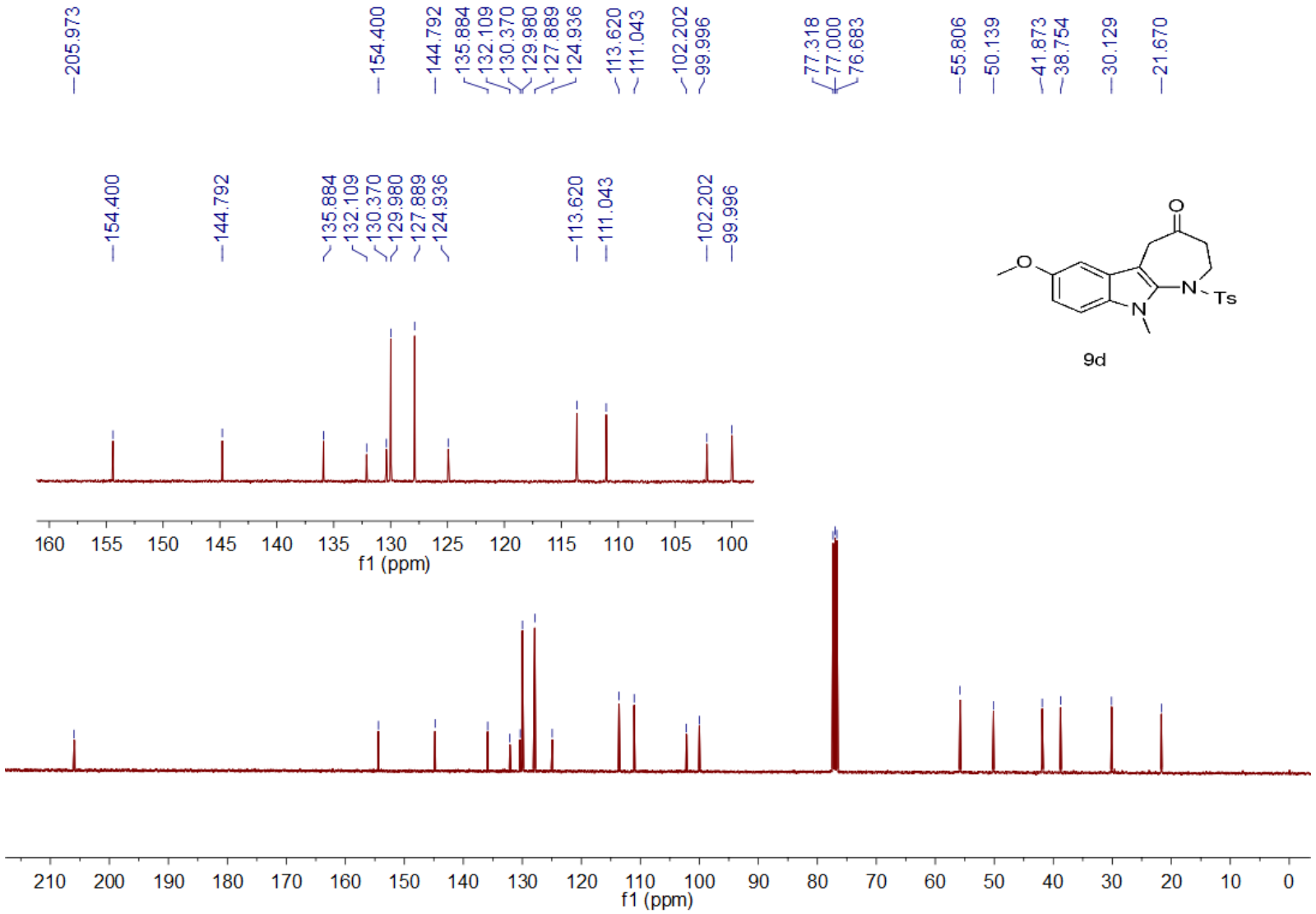

7.623
7.602
7.461
7.441
7.379
7.358
7.332
7.315
7.298
7.278
7.252
7.182
7.180
7.162
7.145
7.143
4.464
4.452
4.444
4.429
4.416
4.408
4.396
3.809
3.793
3.781
3.776
3.335
2.717
2.676
2.627
2.616
2.608
2.595
2.582
2.576
2.563
2.542
2.530
2.523
2.511
2.498
2.488
2.461
0.000
1.90
1.06
1.12
0.83
1.99
1.03
7.65
7.55
7.45
7.35
7.25
7.15
f1 (ppm)
O
N
N
Ts
9b
1.00
4.04
1.02
1.11
1.09
1.12
2.88
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)

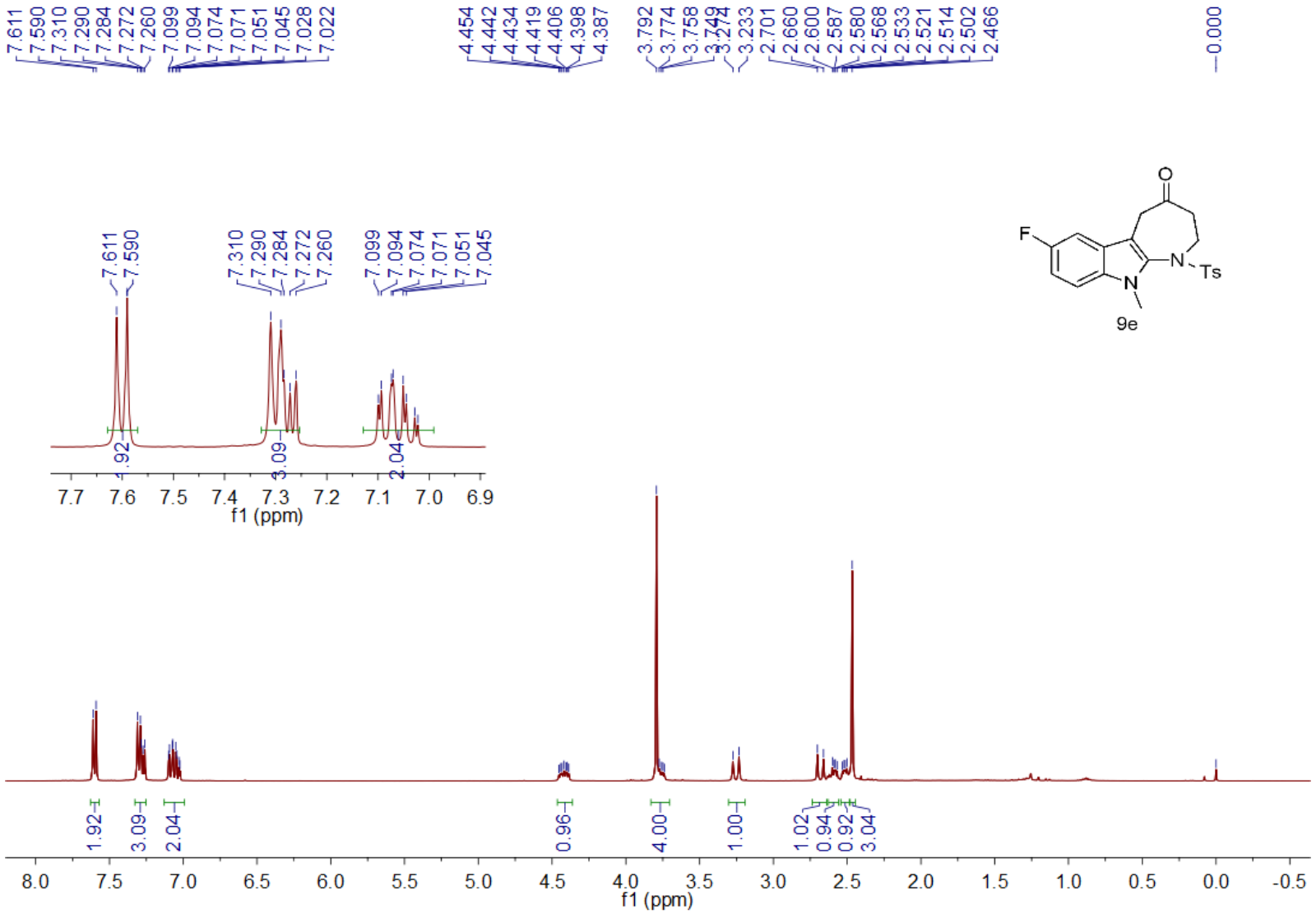

205.930
144.851
135.812
135.168
129.996
127.905
124.763
123.058
119.938
118.600
102.766
77.318
77.000
76.683
50.160
41.790
38.657
30.061
21.674
144.851
135.812
135.168
131.934
129.996
127.905
124.763
123.058
119.938
118.600
110.101
102.766
145 140 135 130 125 120 115 110 105
f1 (ppm)
O
N
N
Ts
9b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

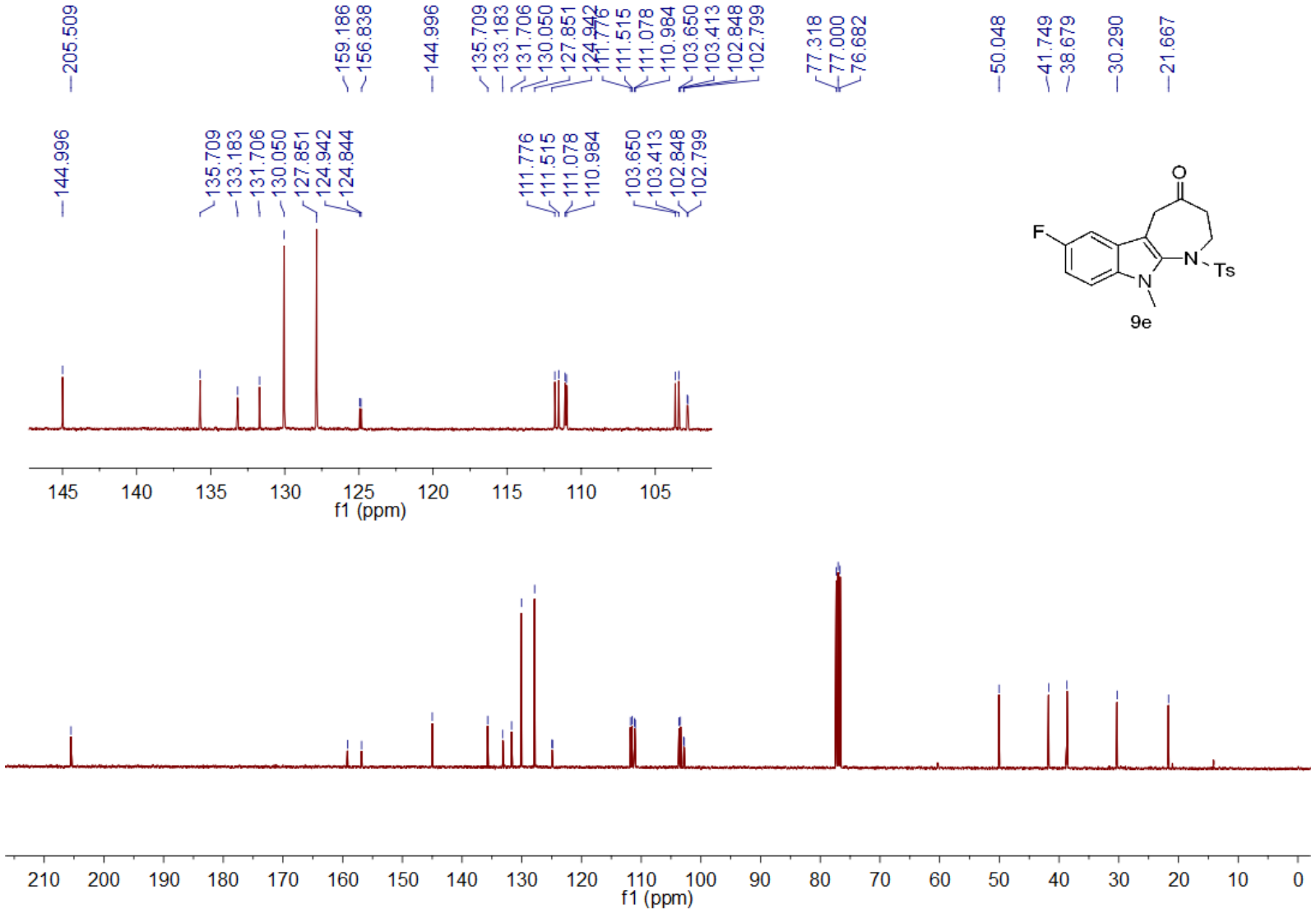

7.613 7.593 7.472 7.452 7.377 7.357 7.292 7.275 7.272 7.264 7.258 7.255 7.246 7.228 7.222 7.218 7.214 7.201 7.181 7.179 7.161 7.144 7.142 7.022 7.018 7.001 5.781 5.740 5.453 5.412 4.204 4.195 4.182 4.169 4.160 4.147 3.359 3.317 3.160 3.155 3.142 3.137 3.125 3.119 3.107 2.641 2.598 2.471 2.457 2.439 2.434 2.421 2.401 2.387 2.378 2.367 2.334 0.000
7.613 7.593 7.472 7.452 7.377 7.357 7.292 7.275 7.272 7.264 7.258 7.255 7.246 7.228 7.222 7.218 7.214 7.201 7.181 7.179 7.161 7.022 7.018 7.001
2.02 1.02 1.05 3.05 3.14 1.13 2.00
7.6 7.5 7.4 7.3 7.2 7.1 7.0
f1 (ppm)
O
N
Ts
N
Bn
9c
2.02 1.02 1.05 3.05 3.14 1.13 2.00 1.00 1.01 1.03 1.03 1.03 1.05 5.36
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)

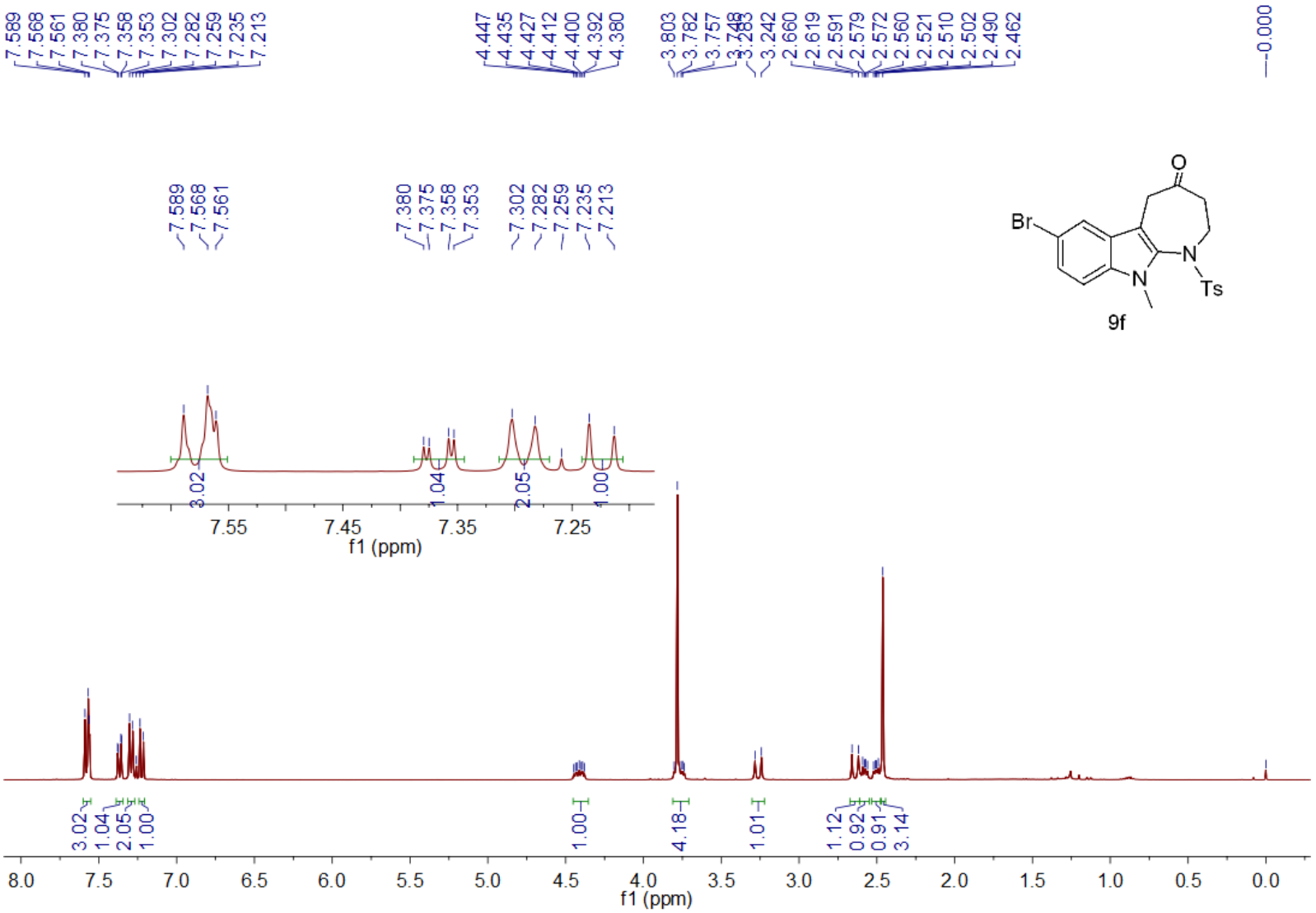

206.142
144.817
137.841
135.768
135.086
131.715
129.970
128.650
127.824
127.320
126.640
125.016
123.401
120.084
118.704
110.698
104.637
77.318
77.000
76.682
49.814
46.728
41.594
38.599
21.664
f1 (ppm)
O
N
Ts
N
Bn
9c

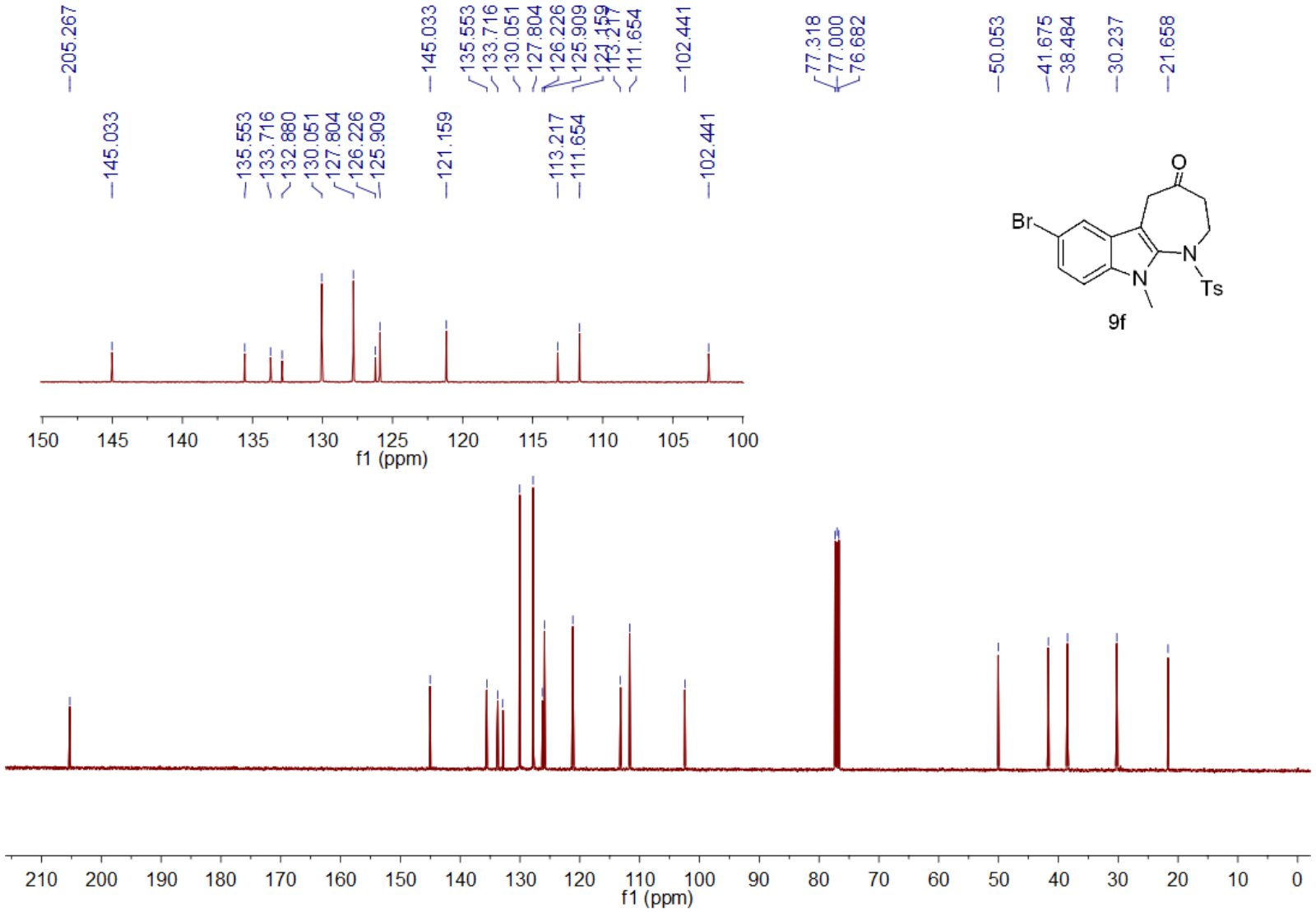

7.627
7.606
7.305
7.285
7.269
7.258
7.246
6.982
6.976
6.960
6.954
6.866
6.860
4.461
4.450
4.442
4.426
4.414
4.406
4.395
3.843
3.786
3.769
3.750
3.320
3.280
2.707
2.666
2.645
2.634
2.625
2.613
2.600
2.592
2.580
2.535
2.523
2.516
2.504
2.464
0.000
1.98
3.28
1.01
1.00
f1 (ppm)
O
O
N
Ts
N
9d
1.98
3.28
1.01
1.00
1.03
3.09
4.21
0.99
0.99
1.07
1.05
3.12
f1 (ppm)


205.973
154.400
144.792
135.884
132.109
130.370
129.980
127.889
124.936
113.620
111.043
102.202
99.996
77.318
77.000
76.683
55.806
50.139
41.873
38.754
30.129
21.670
O
O
N
Ts
N
9d
f1 (ppm)
f1 (ppm)


7.611
7.590
7.310
7.290
7.284
7.272
7.260
7.099
7.094
7.074
7.071
7.051
7.045
7.028
7.022
4.454
4.442
4.434
4.419
4.406
4.398
4.387
3.792
3.774
3.758
3.749
3.274
3.233
2.701
2.660
2.600
2.587
2.580
2.568
2.533
2.521
2.514
2.502
2.466
0.000
F
O
N
Ts
N
9e
1.92
3.09
2.04
0.96
4.00
1.00
1.02
0.94
0.92
3.04
f1 (ppm)


205.509
159.186
156.838
144.996
135.709
133.183
131.706
130.050
127.851
124.942
124.844
111.776
111.515
111.078
110.984
103.650
103.413
102.848
102.799
77.318
77.000
76.682
50.048
41.749
38.679
30.290
21.667
f1 (ppm)
F
N
Ts
O
9e


7.589
7.568
7.561
7.380
7.375
7.358
7.353
7.302
7.282
7.259
7.235
7.213
4.447
4.435
4.427
4.412
4.400
4.392
4.380
3.803
3.782
3.757
3.242
2.660
2.619
2.591
2.579
2.572
2.560
2.521
2.510
2.502
2.490
2.462
0.000
Br
O
N
N
Ts
9f
3.02
1.04
2.05
1.00
7.55
7.45
7.35
7.25
f1 (ppm)
1.00
4.18
1.01
1.12
0.92
0.91
3.14
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)


205.267
145.033
135.553
133.716
132.880
130.051
127.804
126.226
125.909
121.159
113.217
111.654
102.441
77.318
77.000
76.682
50.053
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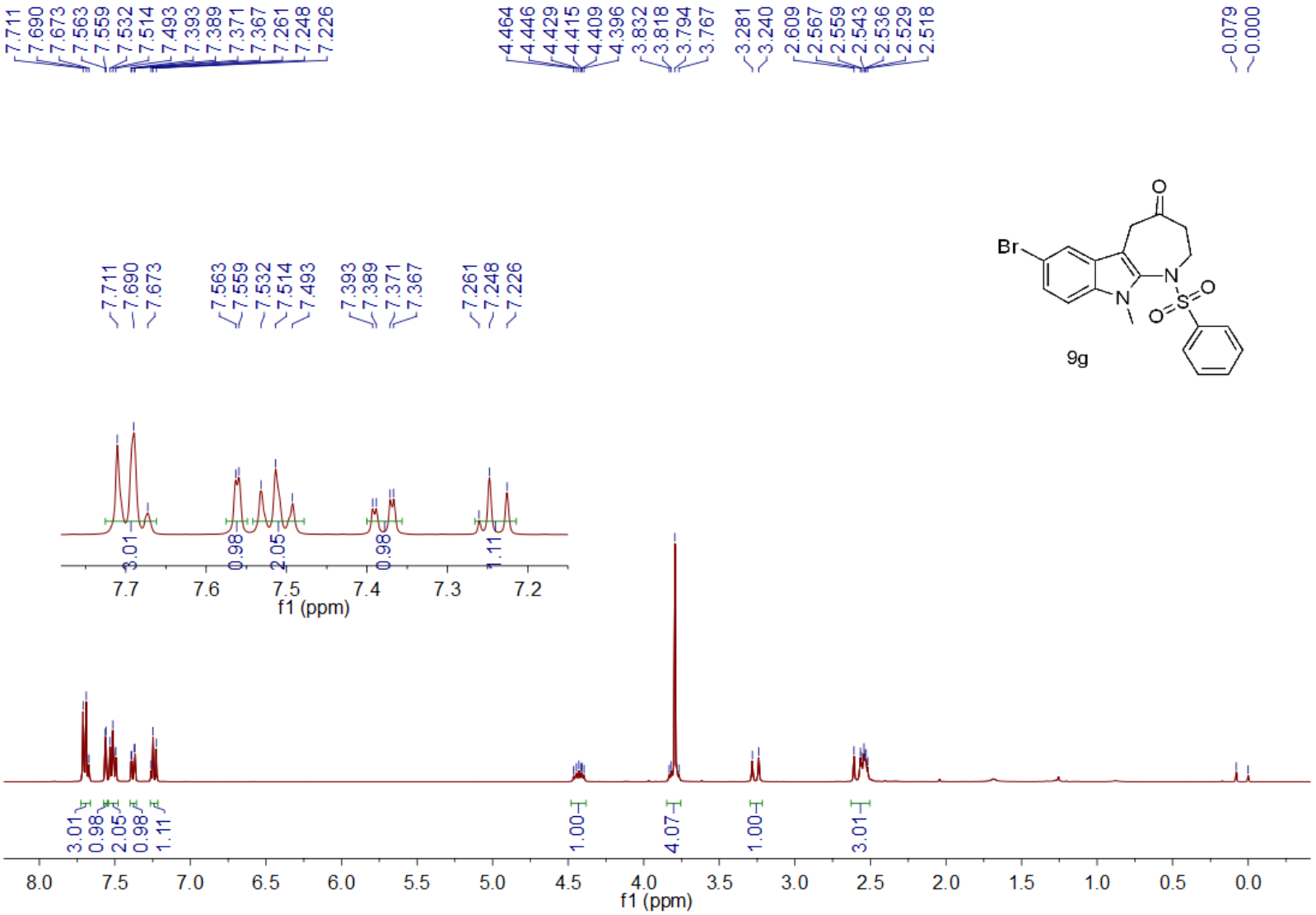
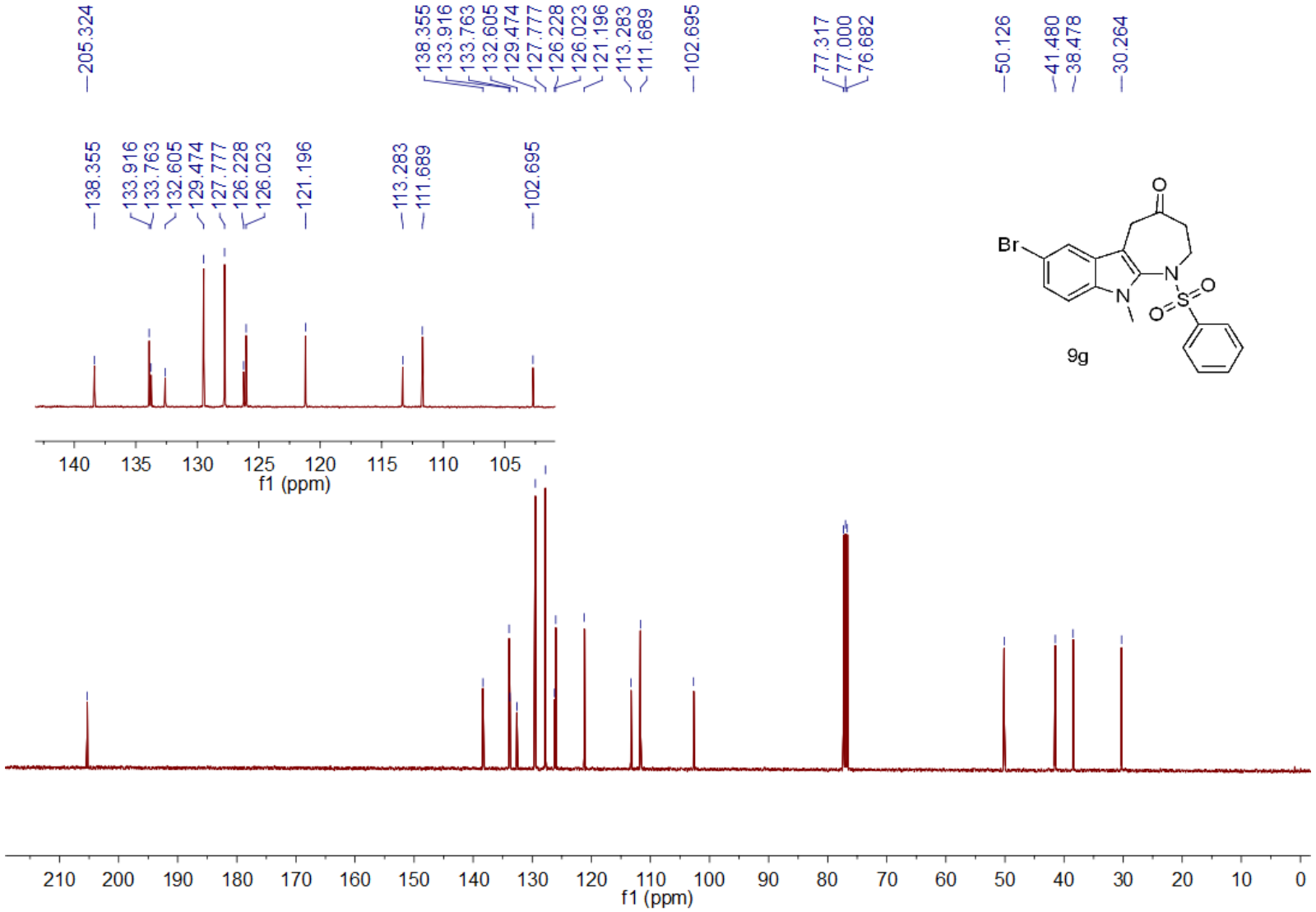
38.484
30.237
21.658
Br
N
N
Ts
O
9f
f1 (ppm)
f1 (ppm)


7.711
7.690
7.673
7.563
7.559
7.532
7.514
7.493
7.393
7.389
7.371
7.367
7.261
7.248
7.226
4.464
4.446
4.429
4.415
4.409
4.396
3.832
3.818
3.794
3.767
3.281
3.240
2.609
2.567
2.559
2.543
2.536
2.529
2.518
0.079
0.000
Br
O
N
N
S
O
O
9g
3.01
0.98
2.05
0.98
1.11
1.00
4.07
1.00
3.01
f1 (ppm)


205.324
138.355
133.916
133.763
132.605
129.474
127.777
126.228
126.023
121.196
113.283
111.689
102.695
77.317
77.000
76.682
50.126
41.480
38.478
30.264
Br
O
N
N
O
S
O
9g
f1 (ppm)
f1 (ppm)

The NOESY of 3p