

Electronic Supplementary Information

Rediscovering the Bacon's Hydrazine/Phenylhydrazine Mediated Cyclization of 2,2'-Dicarbonylbi(hetero)aryls: Construction of (5-Azo)-/Indazolo[2,3-*a*]quinolines.

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Crystal Parameters for compound 3aa, 6bb and 10

Diffraction Studies:

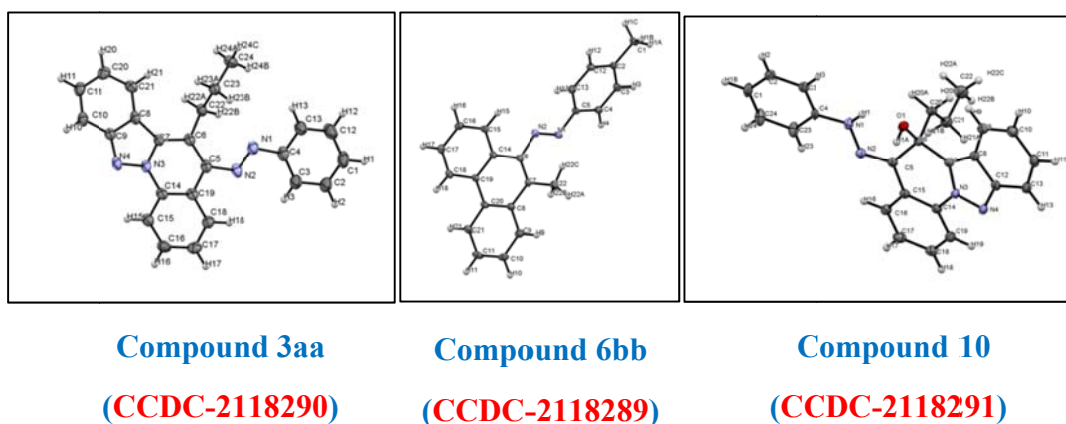
Single crystal data of **3aa**, **6bb** and **10** crystal was collected on Bruker SMART APEX II single crystal X-ray CCD diffractometer having graphite-monochromatized (Cu-K α = 1.54178 Å) radiation at 100 K. The X-ray data acquisition was monitored by APEX2 program suit.¹ The data were corrected for Lorentz-polarization and absorption effects using SAINT and SADABS programs which are integral part of APEX2 package.¹ The structures were solved by direct methods and refined by full matrix least squares, based on F^2 , using SHELXL². Crystal structures were refined using Olex2 1.0 software.³ PLATON⁴ was used to check the any higher symmetry of crystal, and Mercury software was utilized for molecular representations in ORTEP diagrams. Refinement of coordinates and anisotropic thermal parameters of non-hydrogen atoms were performed with the full-matrix least-squares method. The hydrogen atoms of CH were calculated using the riding model.

1. Bruker **2006**. *APEX2*, *SAIN*T and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
2. Sheldrick, G. M. Structure validation in chemical crystallography *Acta Cryst.* **2015**, *D65*, 148–155.
3. Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K.; Puschmann, H. A complete structure solution, refinement and analysis program *J. Appl. Cryst.* 2009, *42*, 339–341.
4. Spek, L. Crystal structure refinement with SHELXL *Acta. Cryst.* **2009**, *C71*, 3–8.

Sample preparation for crystal growth:

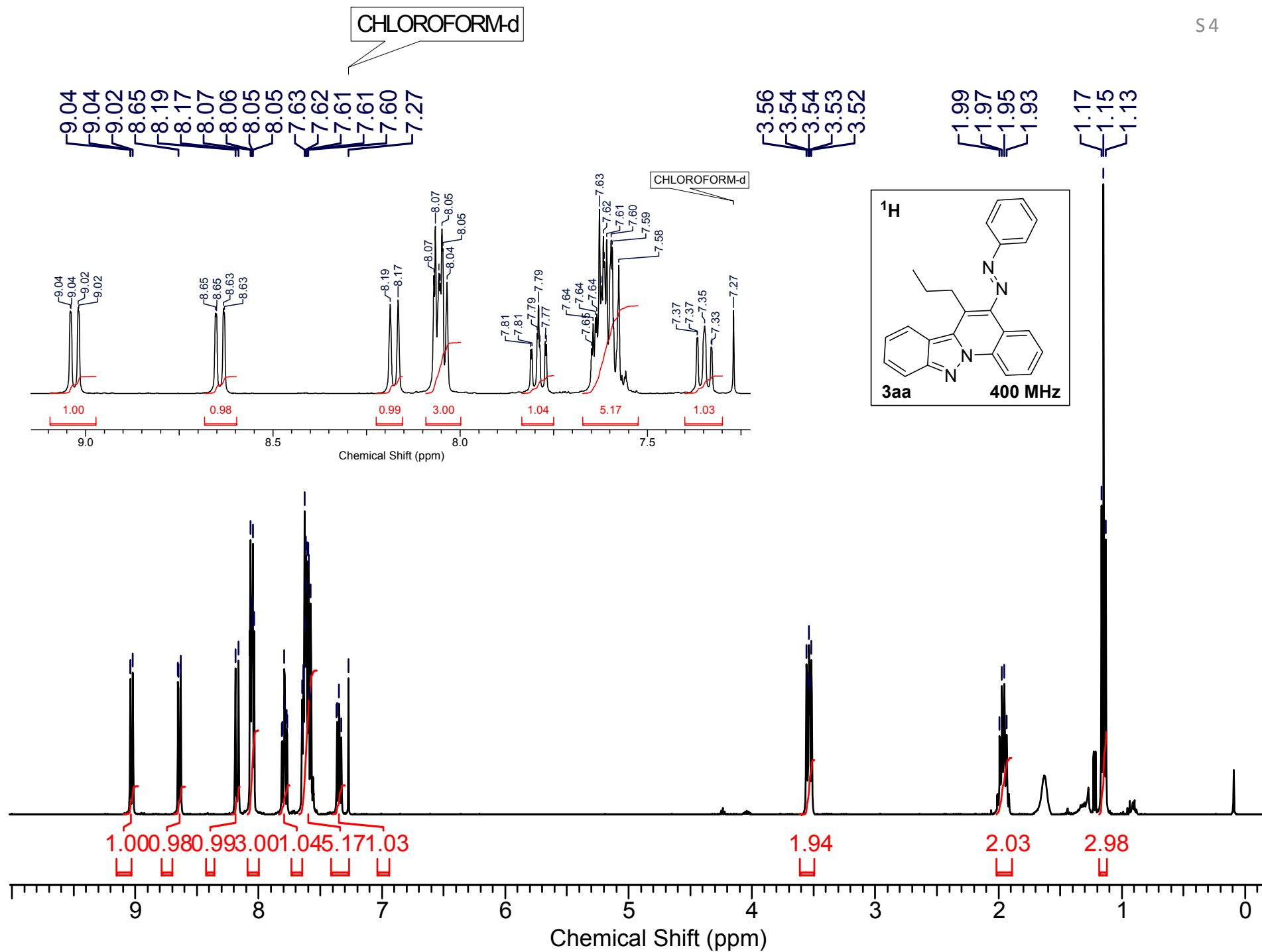
The 10 mg compound was taken in 2 ml glass vial, 1 ml acetonitrile solvent was added and the *vail* was closed with the cotton plug. Kept it for slow evaporation at room temperature in a dark place untill the crystals are formed.

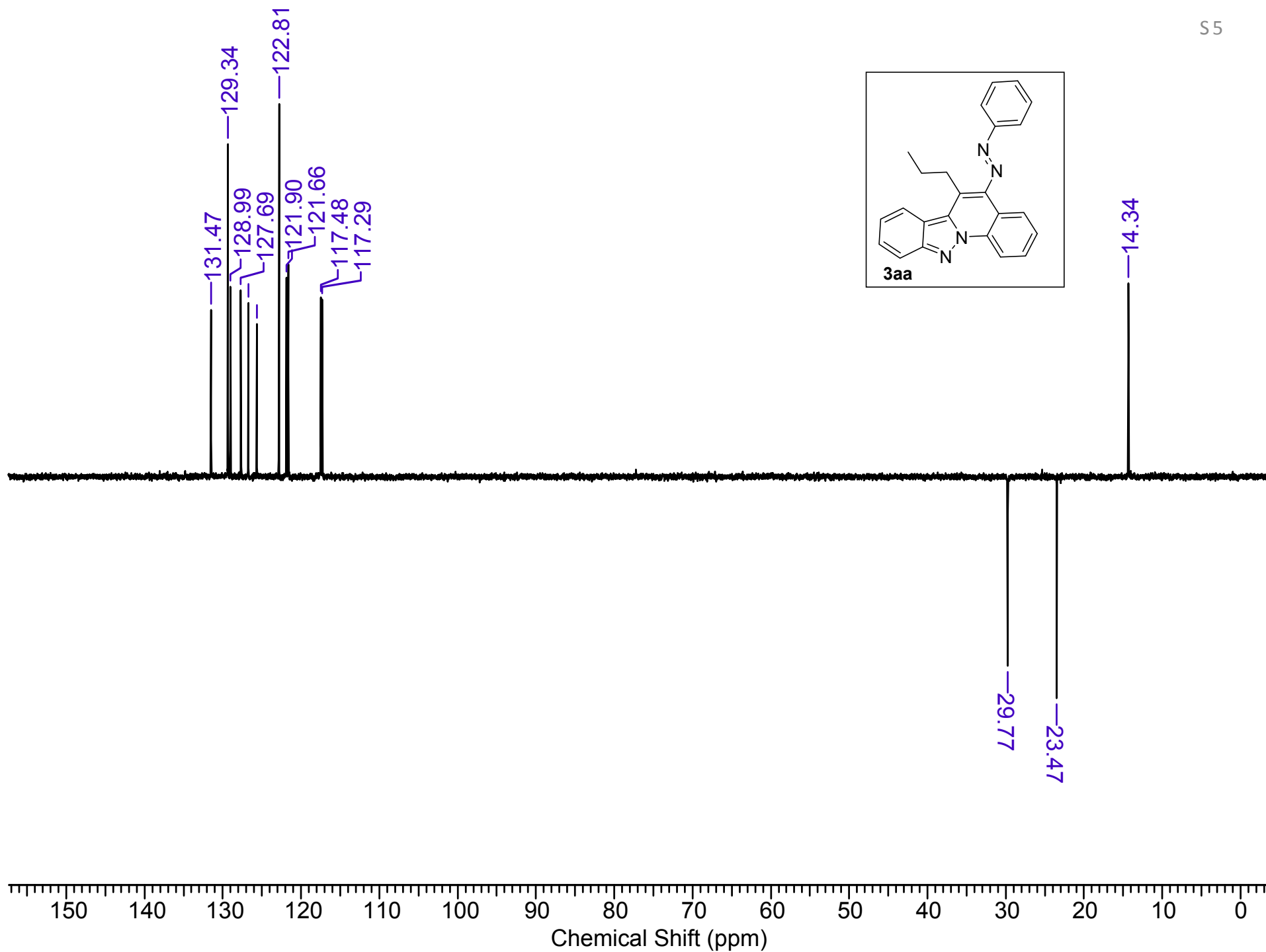
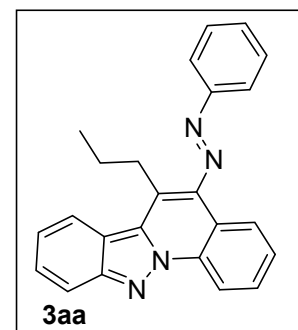
Figure S1. ORTEP diagram of **3aa**, **6bb**, **10** and Thermal ellipsoids are shown at the 50% probability level.

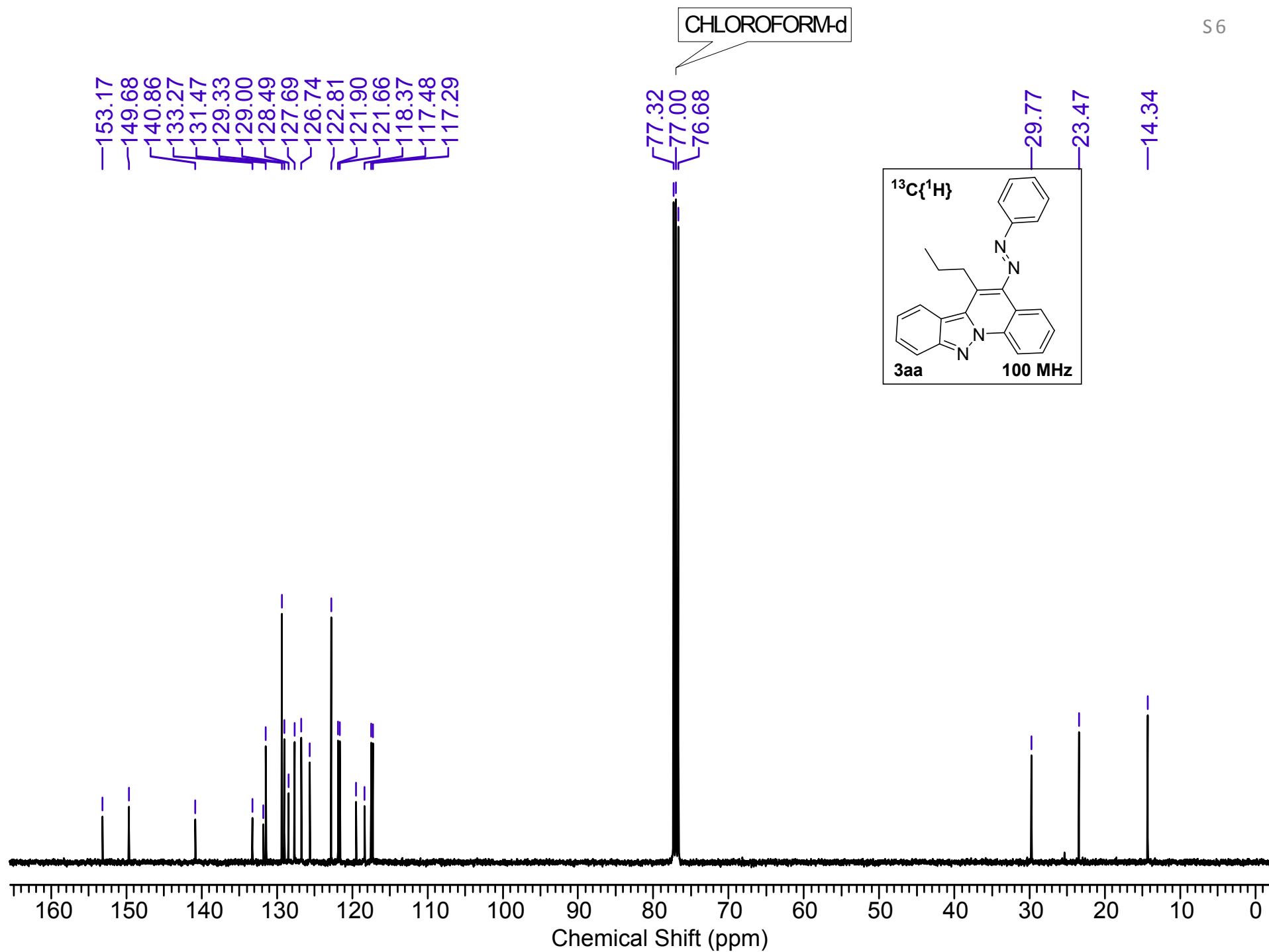


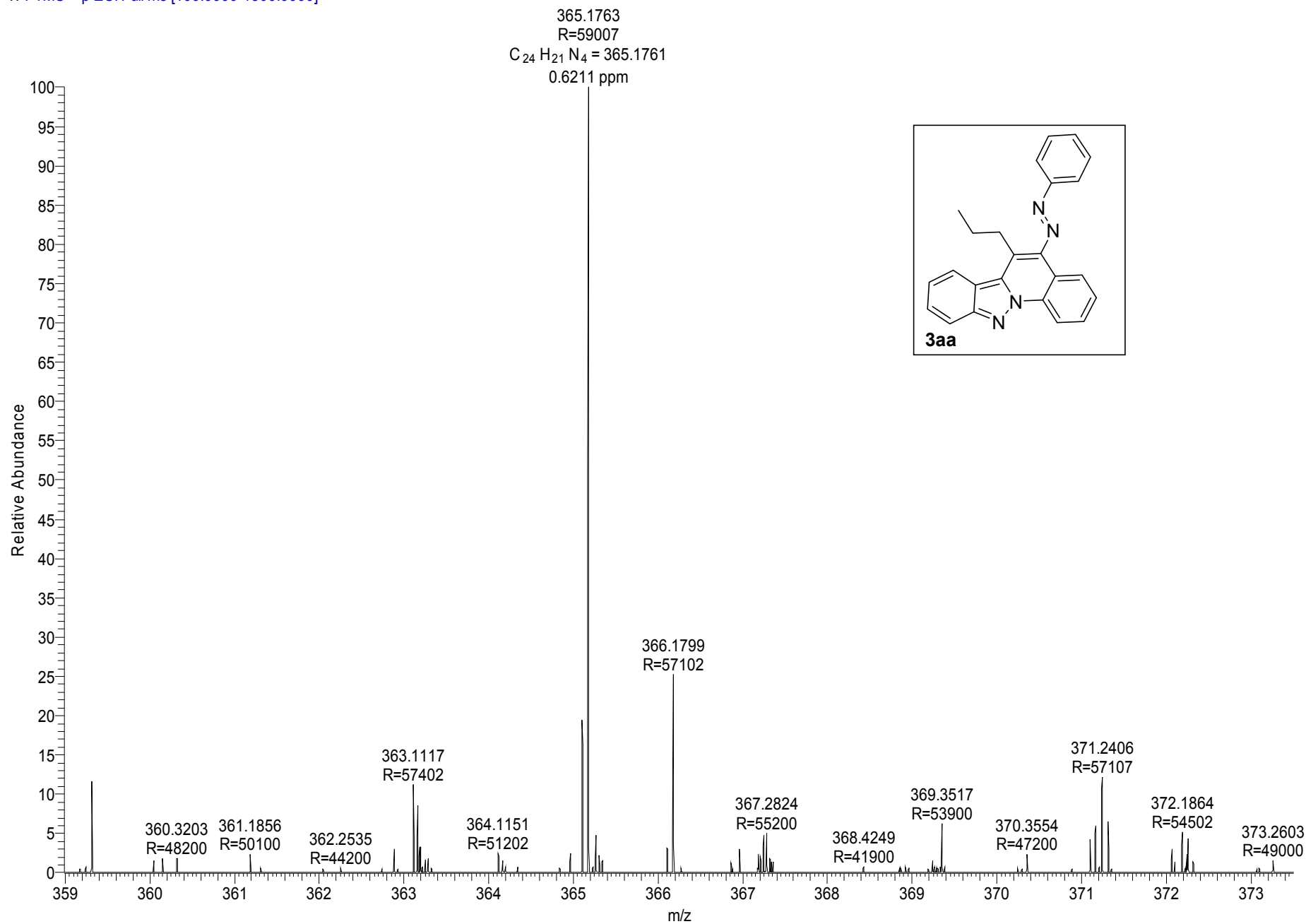
Single crystal data:

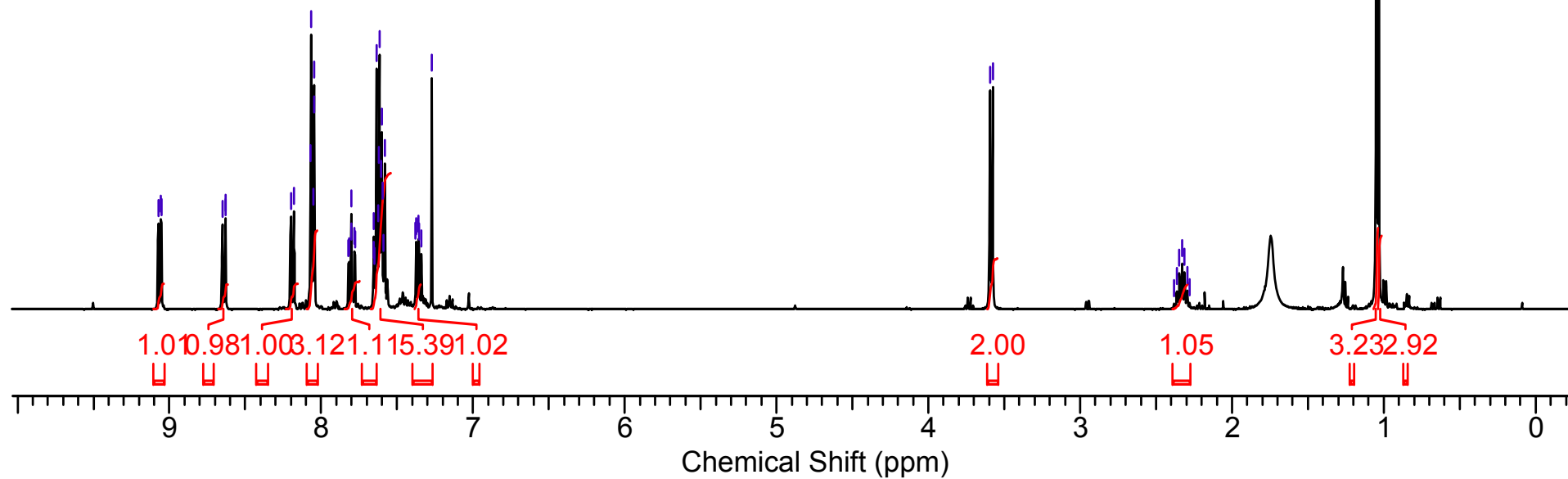
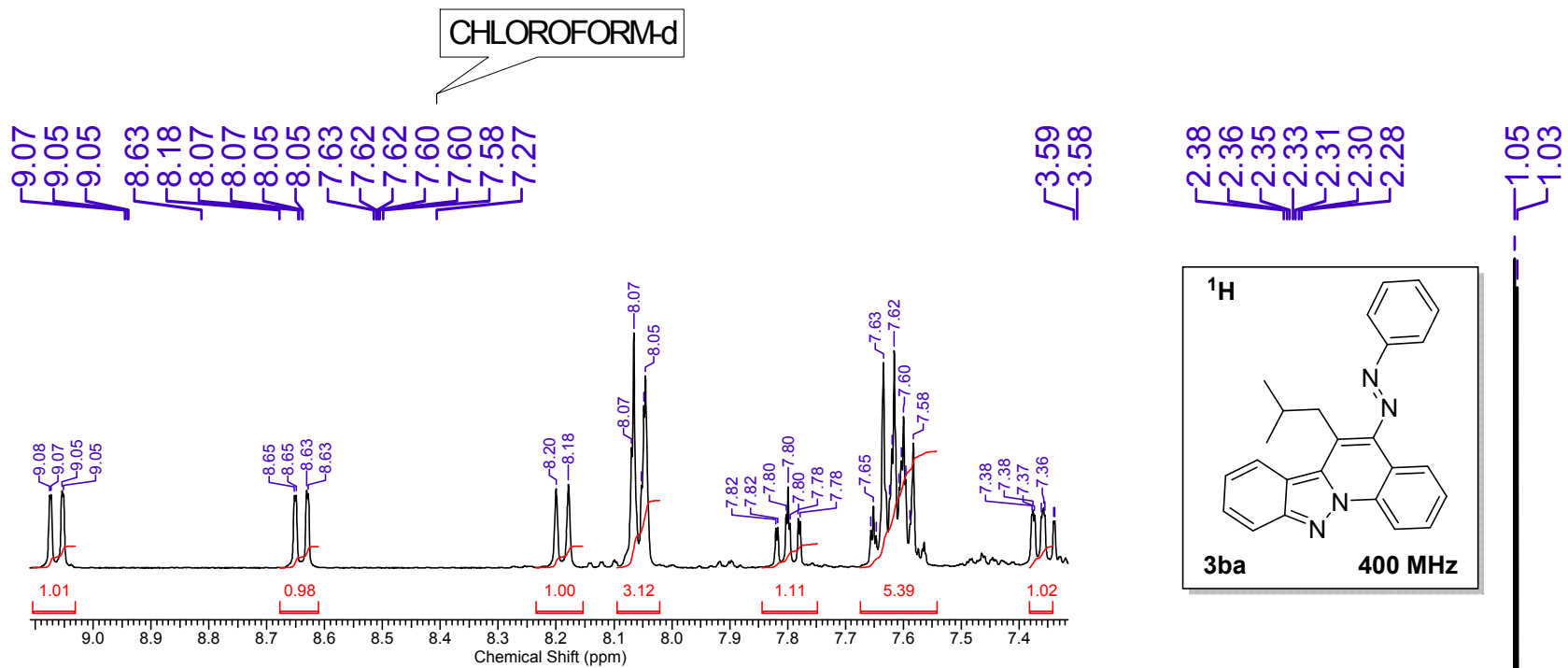
Parameters	3aa	6bb	10
Empirical Formula	C ₂₄ H ₂₀ N ₄	C ₂₂ H ₁₈ N ₂	C ₂₄ H ₂₂ N ₄ O
Formula weight	364.44	310.38	382.45
Crystal System	Monoclinic	Triclinic	Monoclinic
Space Group	<i>P2₁/n</i>	<i>P₁</i>	<i>C2/c</i>
T (K)	100	100	100
<i>a</i> (Å)	4.729 (6)	8.4314 (4)	11.4975 (6)
<i>b</i> (Å)	36.33 (4)	9.1682 (4)	19.6301 (10)
<i>c</i> (Å)	10.854 (16)	11.7324 (5)	17.6537 (9)
α (°)	90	73.137 (1)	90
β (°)	93.66 (3)	89.952 (1)	99.469 (2)
γ (°)	90	67.155 (1)	90
<i>V</i> (Å ³)	1861 (4)	793.39 (6)	3930.1 (4)
<i>D</i> _{cal} (g cm ⁻³)	1.301	1.299	1.293
<i>Z</i>	4	2	8
<i>R</i> ₁ [<i>I</i> > 2σ(<i>I</i>)]	0.097	0.041	0.040
<i>R</i> _{int}	0.207	0.034	0.192
w <i>R</i> ₂ (all)	0.238	0.1222	0.137
Measured reflections	45059	21699	26562
Independent reflections	3951	4072	4043
Reflections with <i>I</i> > 2σ(<i>I</i>)	2617	3860	3670
Goodness of fit (S)	1.039	1.05	1.14
X-ray Diffractometer	Bruker	Bruker	Bruker
CCDC number	2118290	2118289	2118291

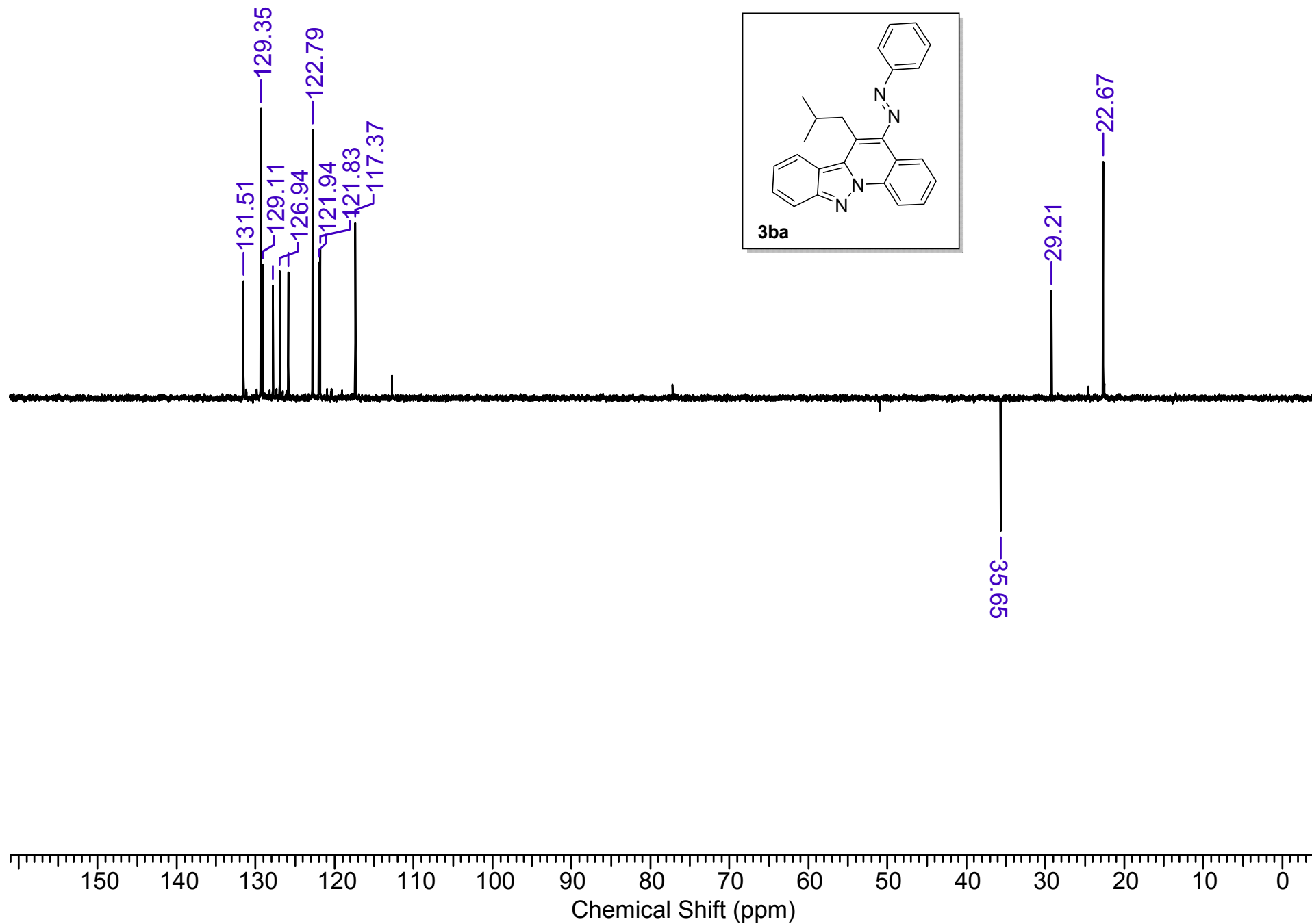


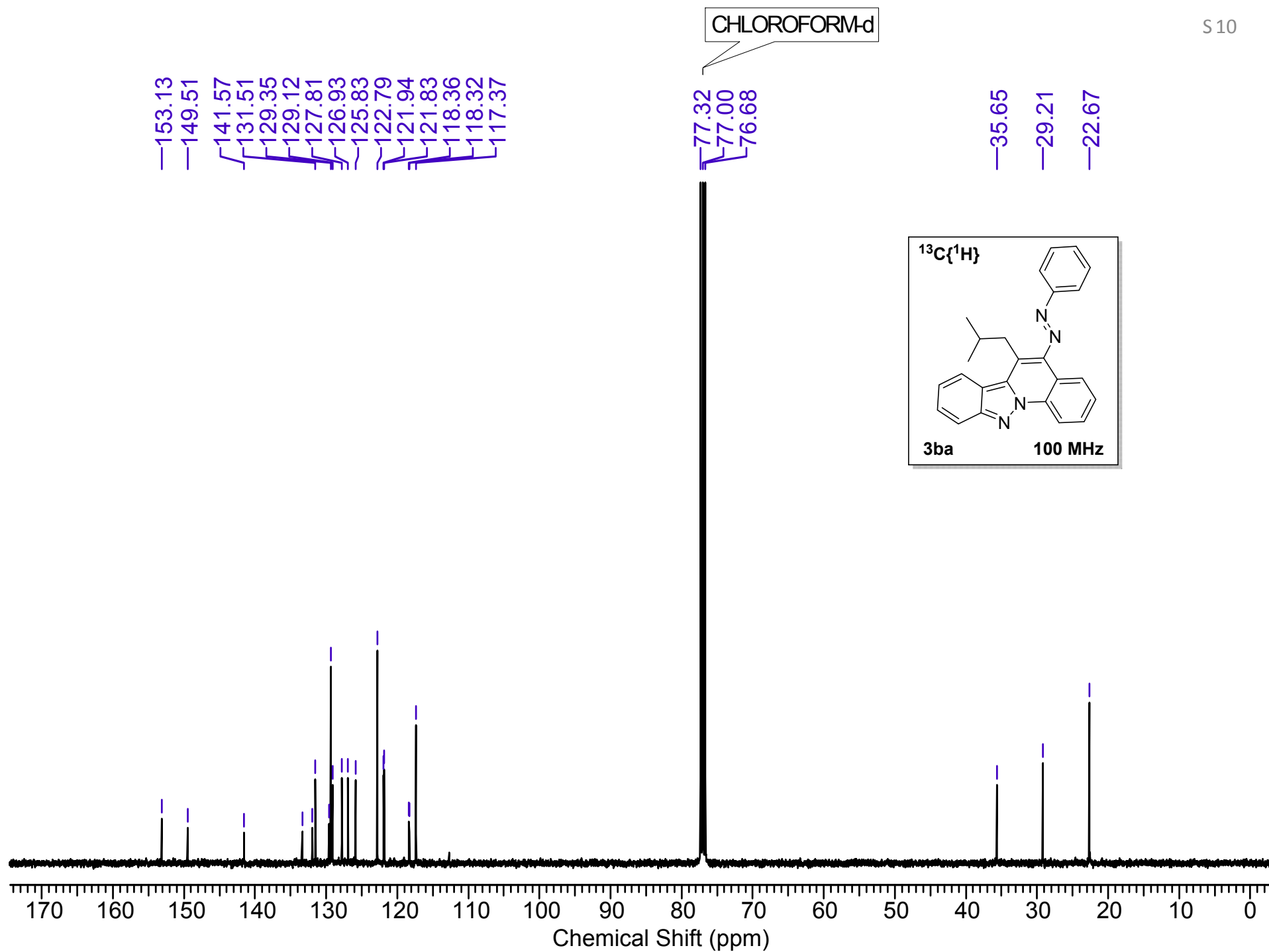




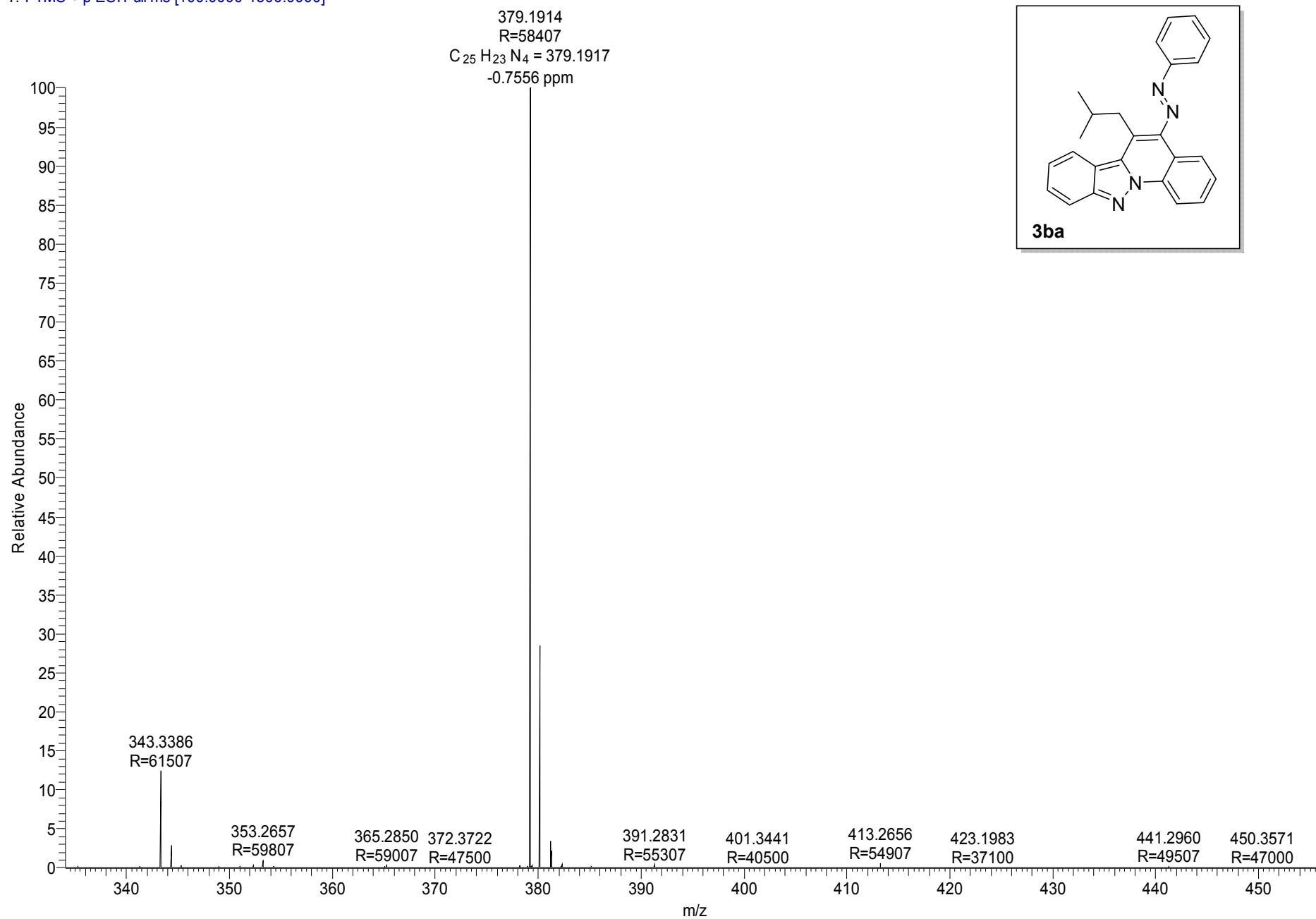


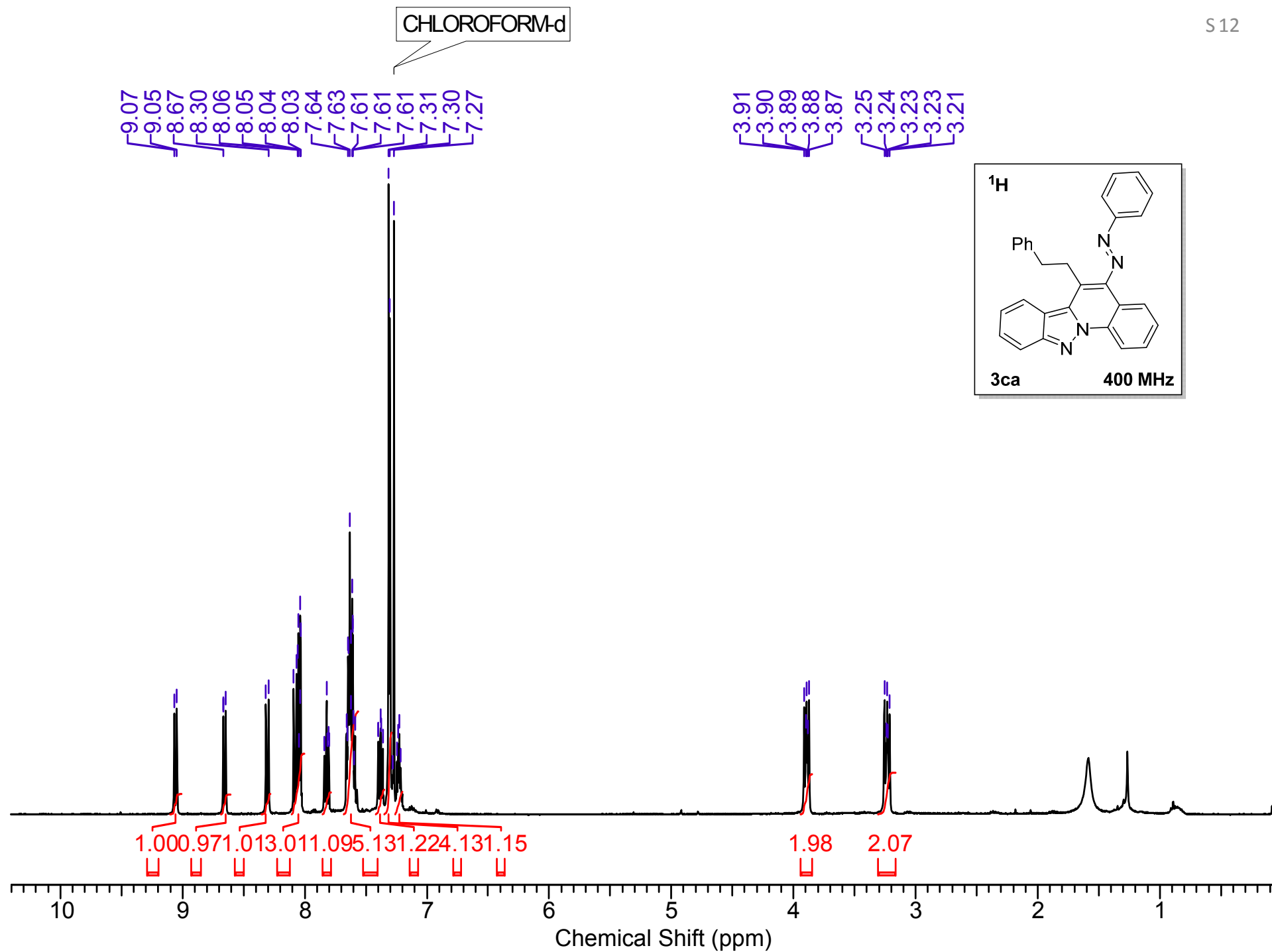


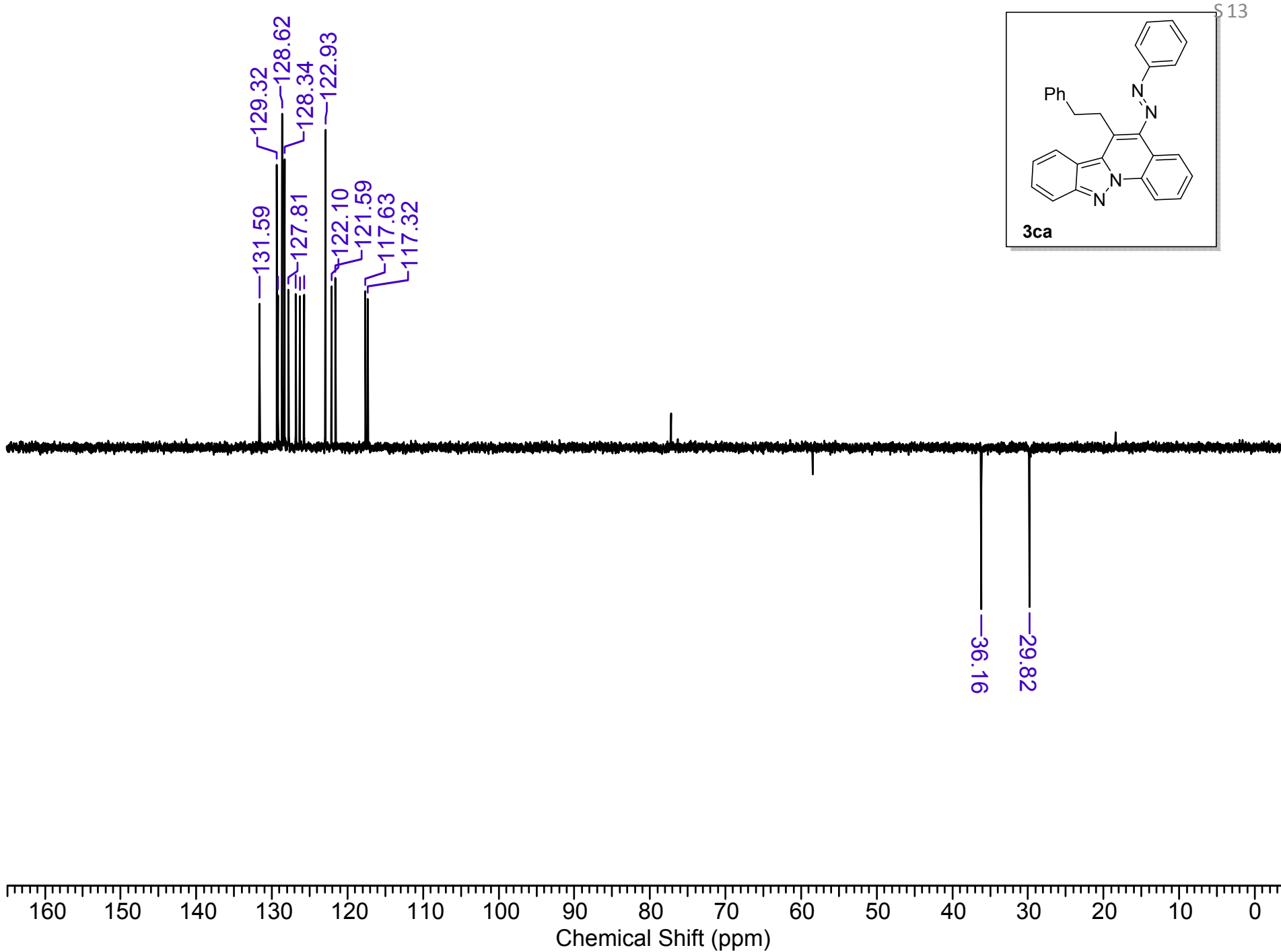


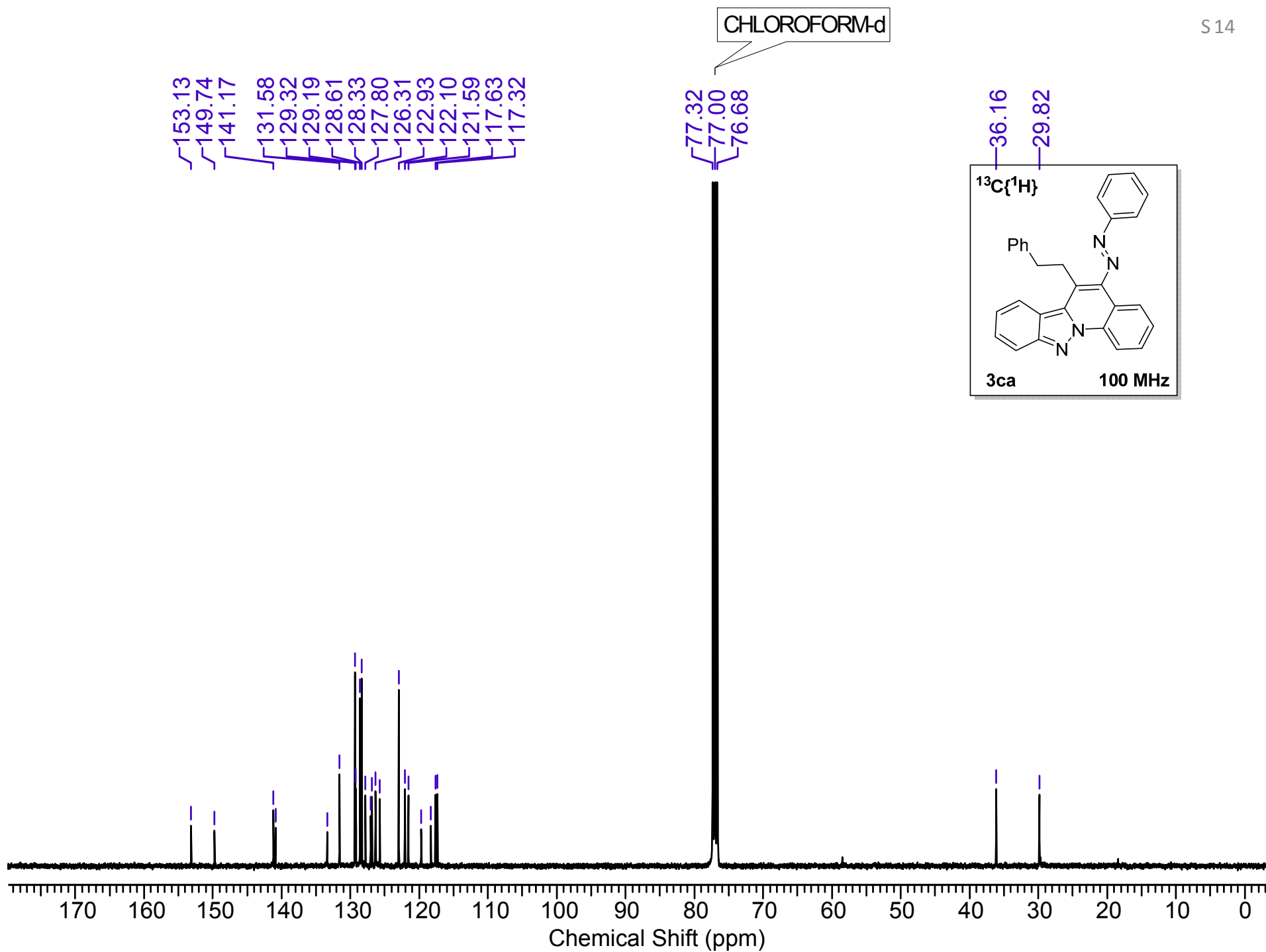


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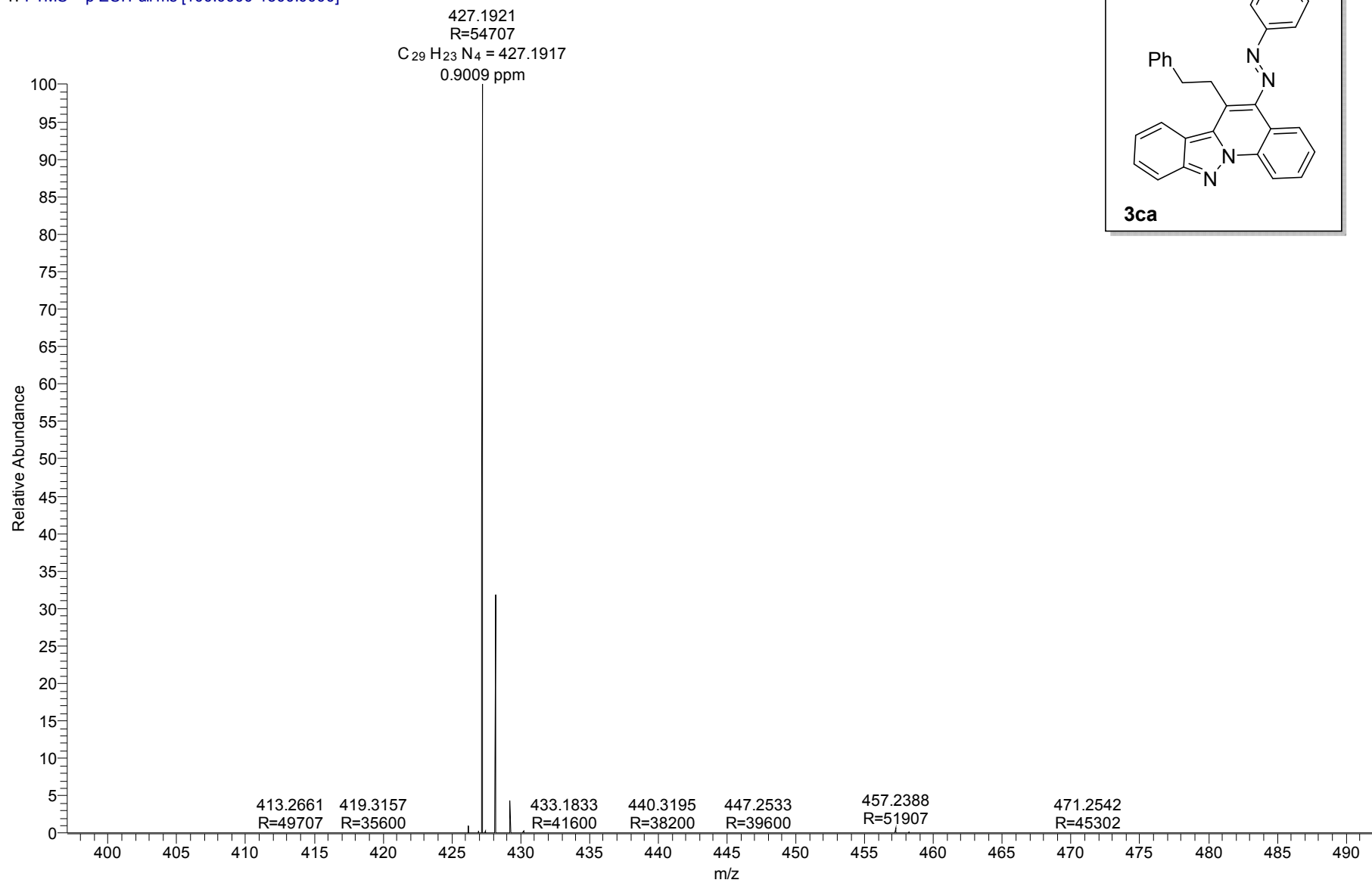


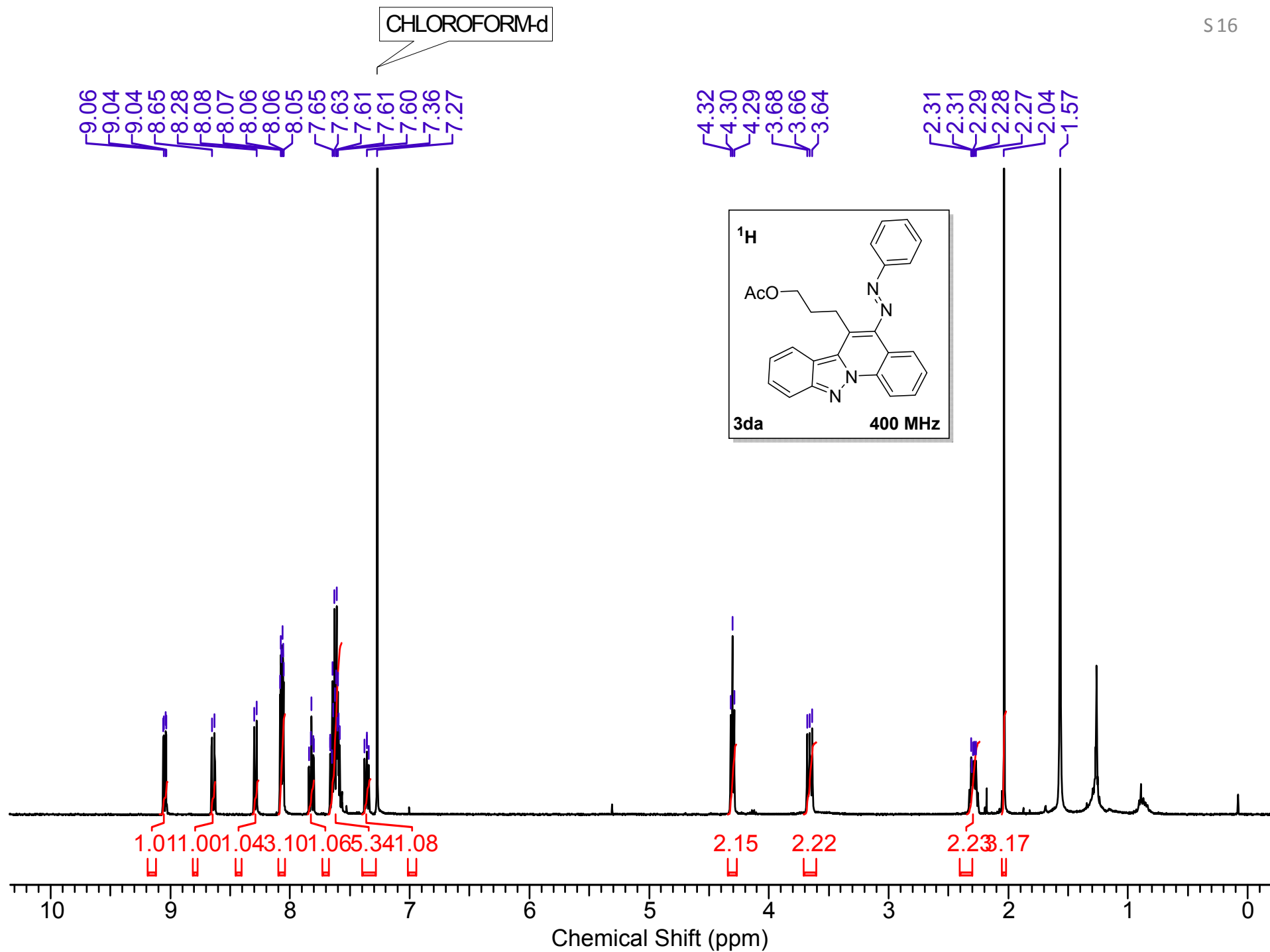


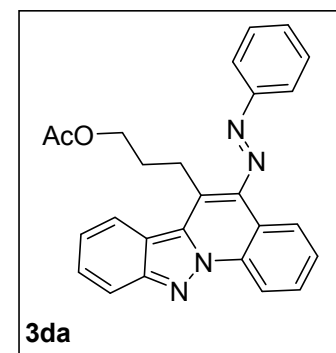




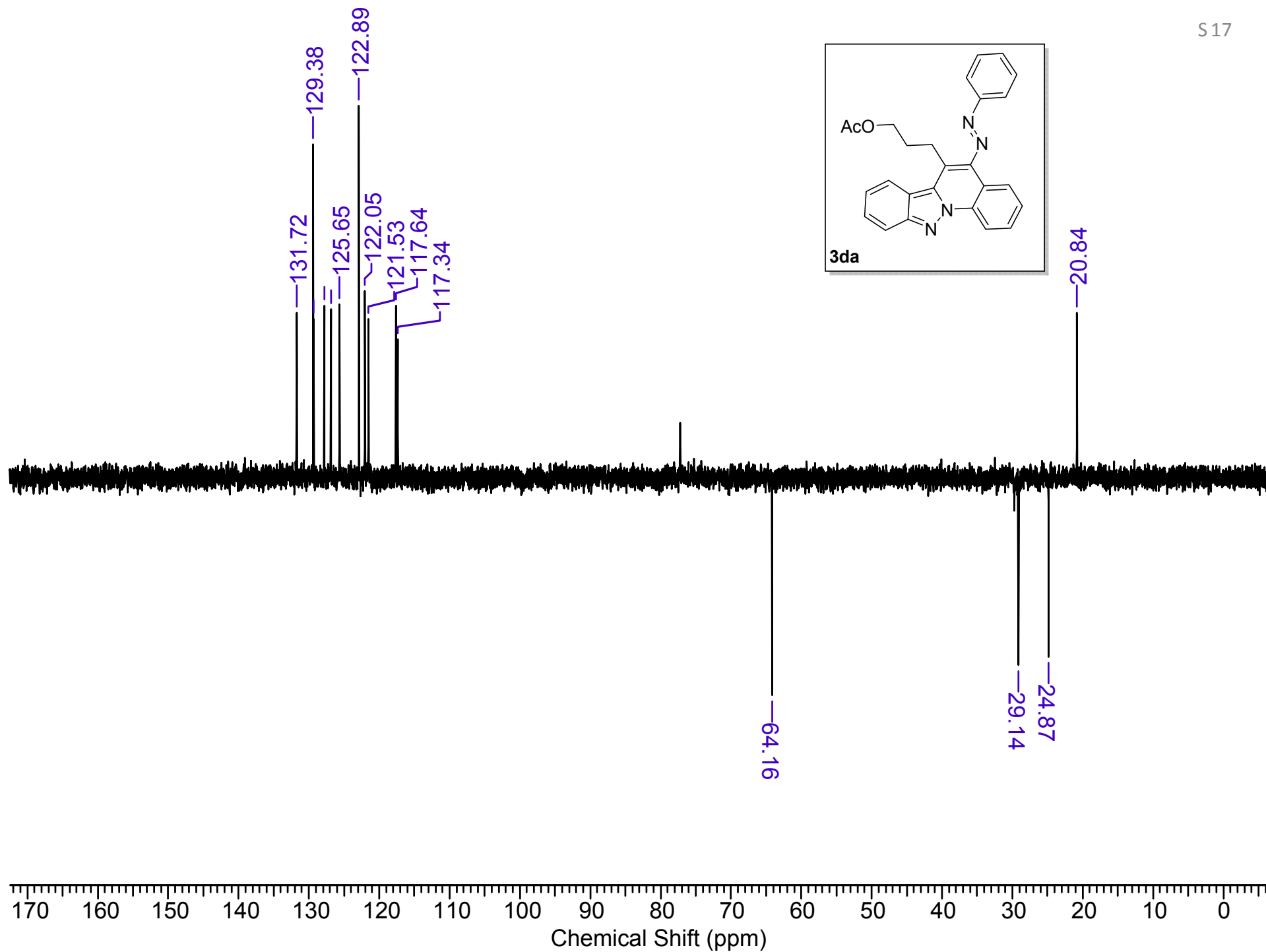
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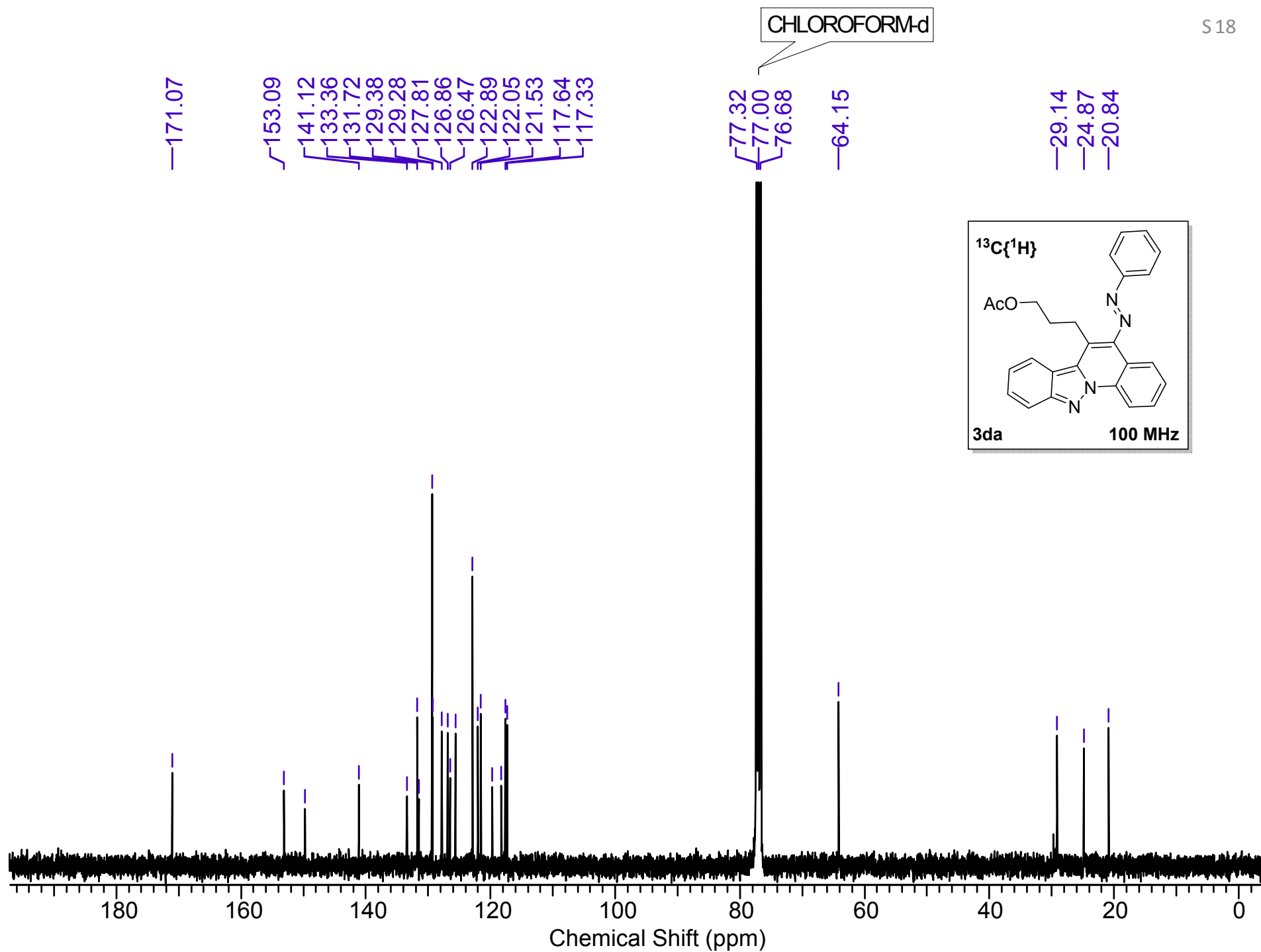




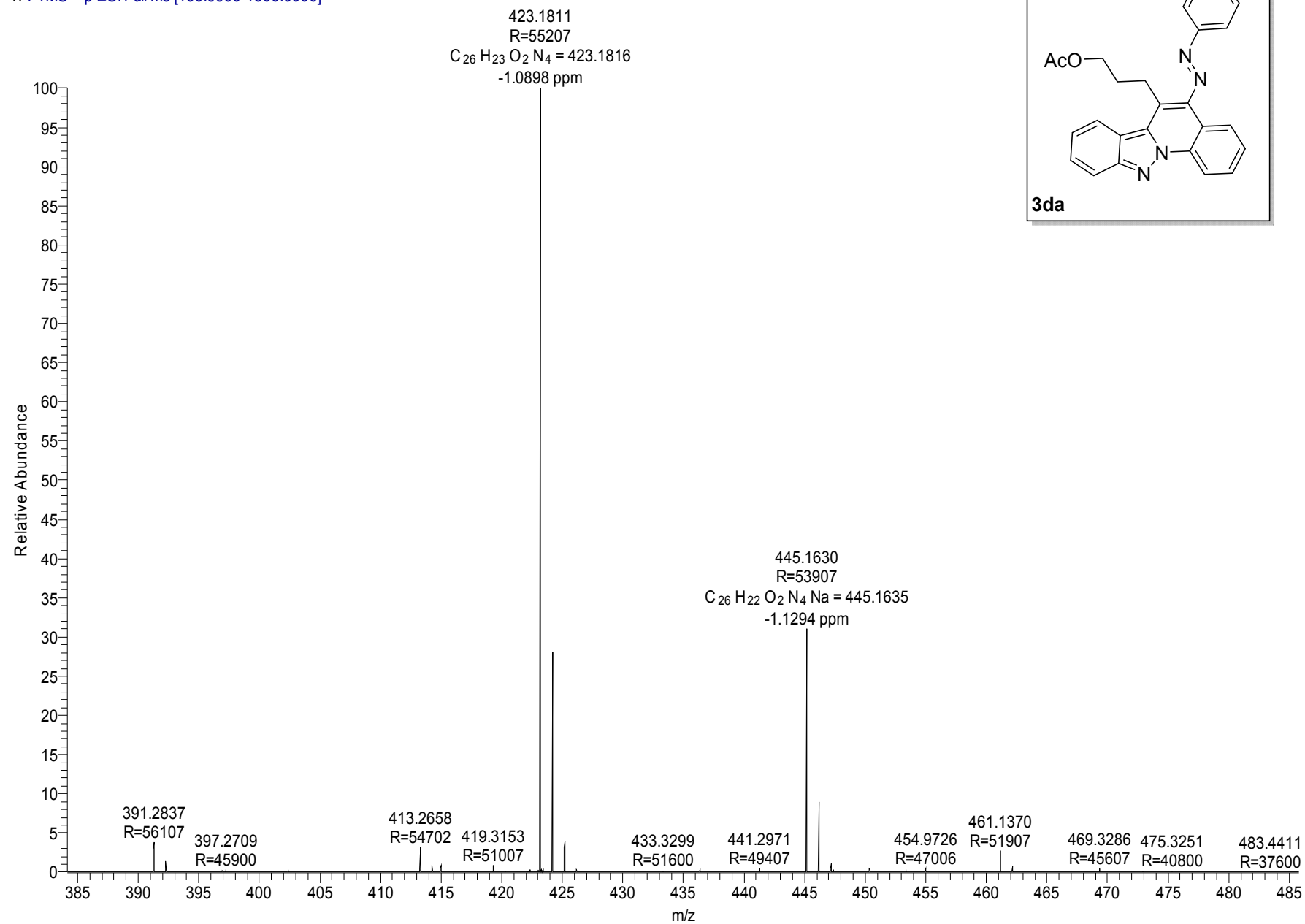


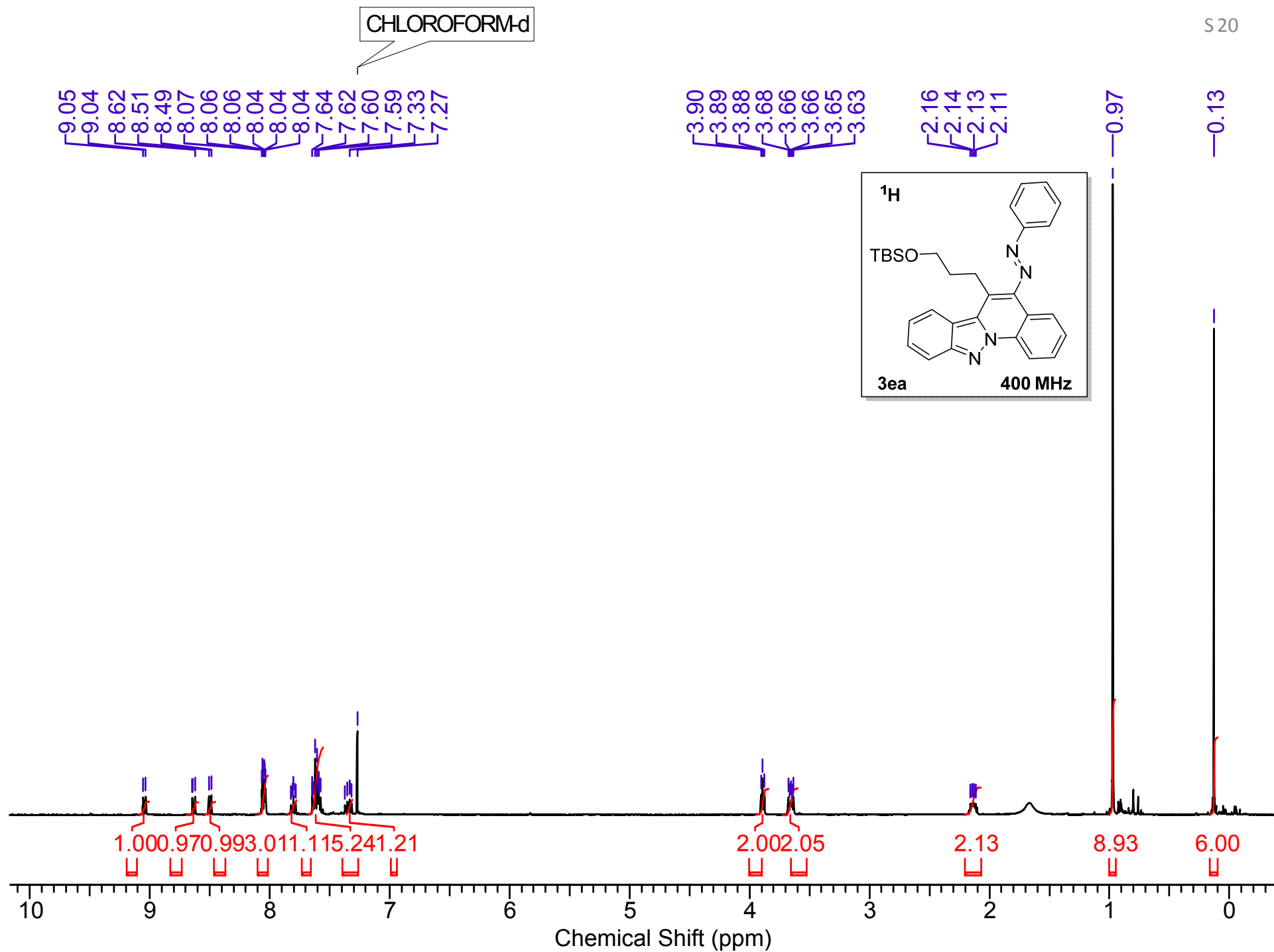
3da

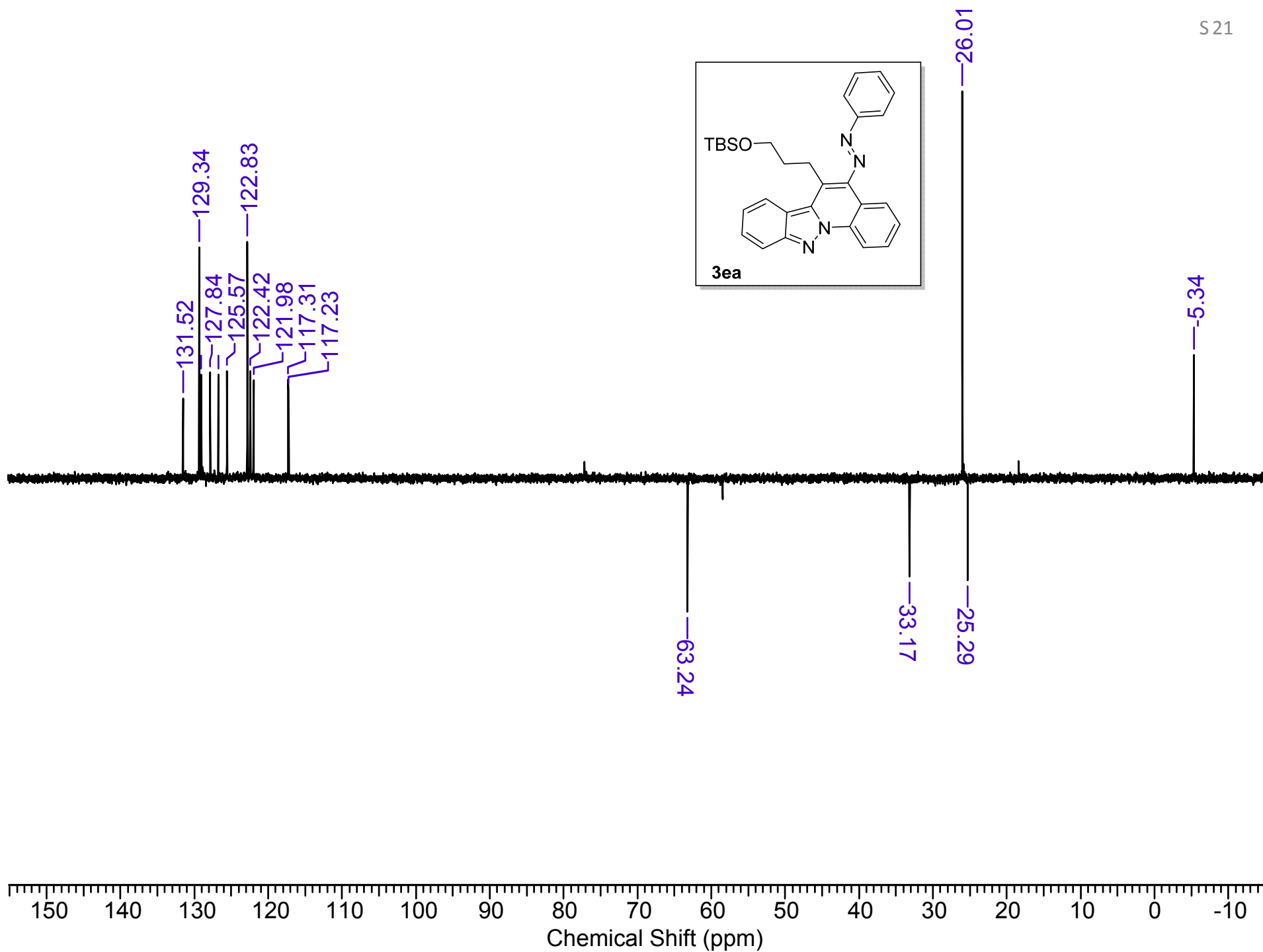


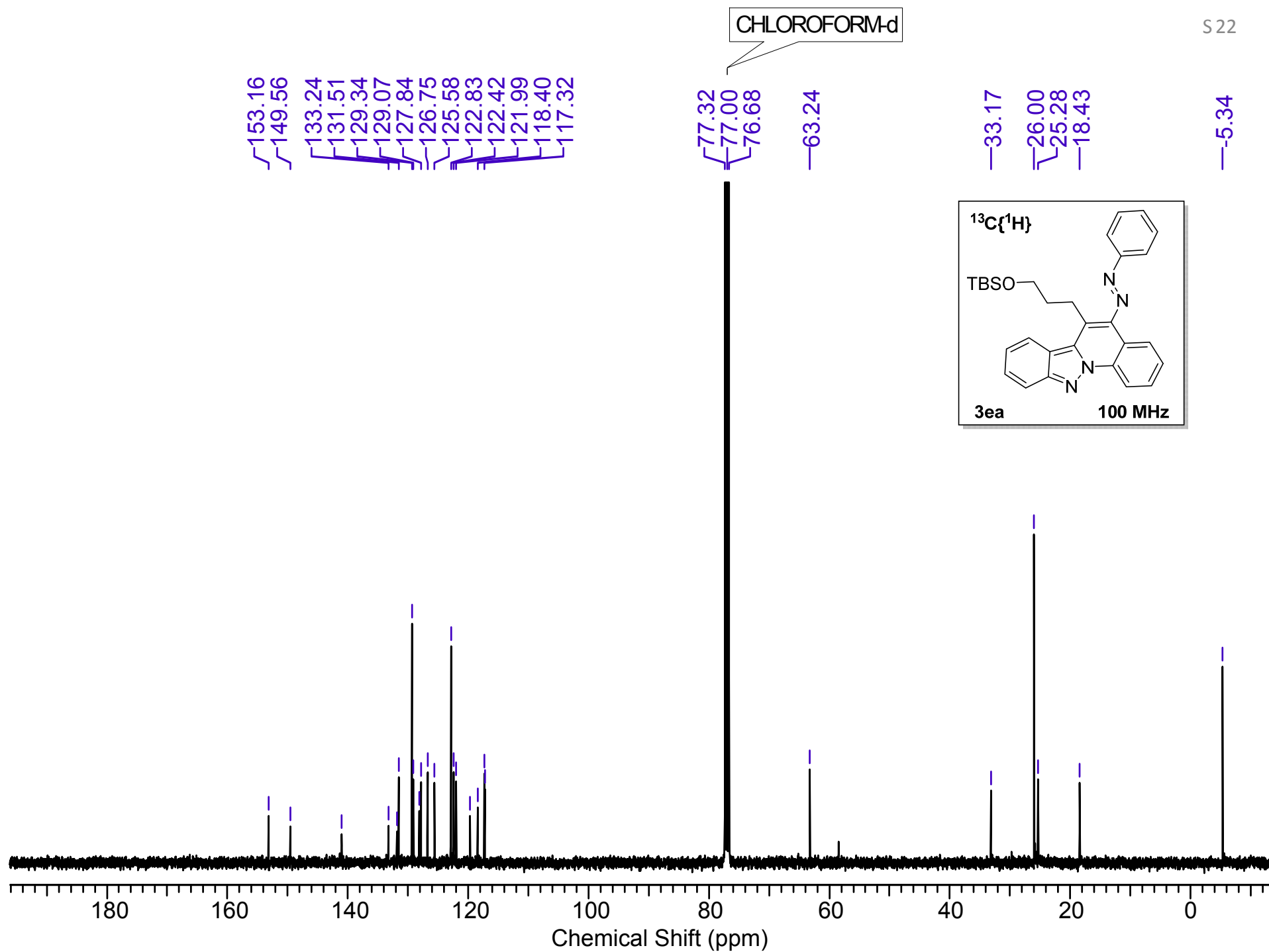


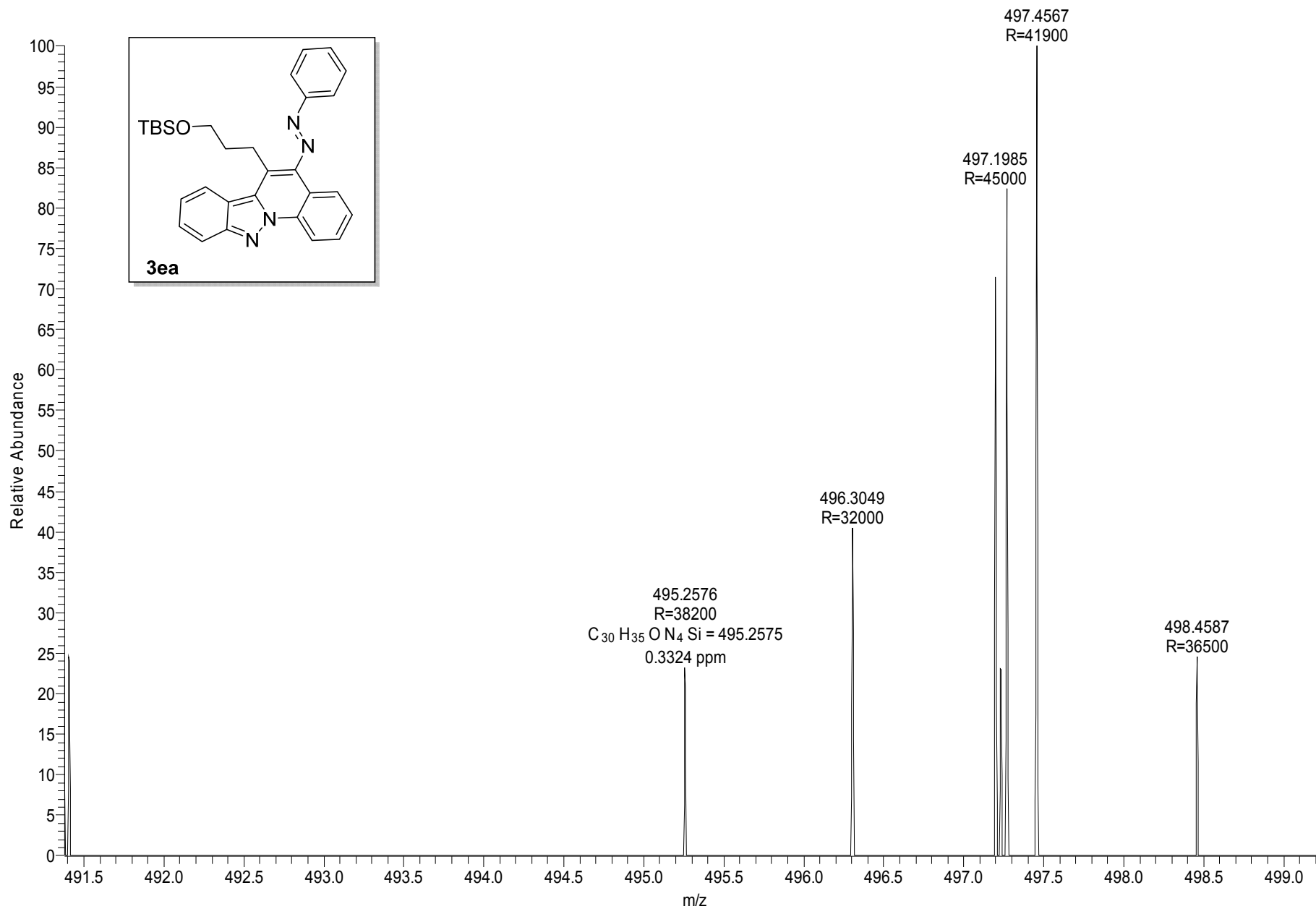
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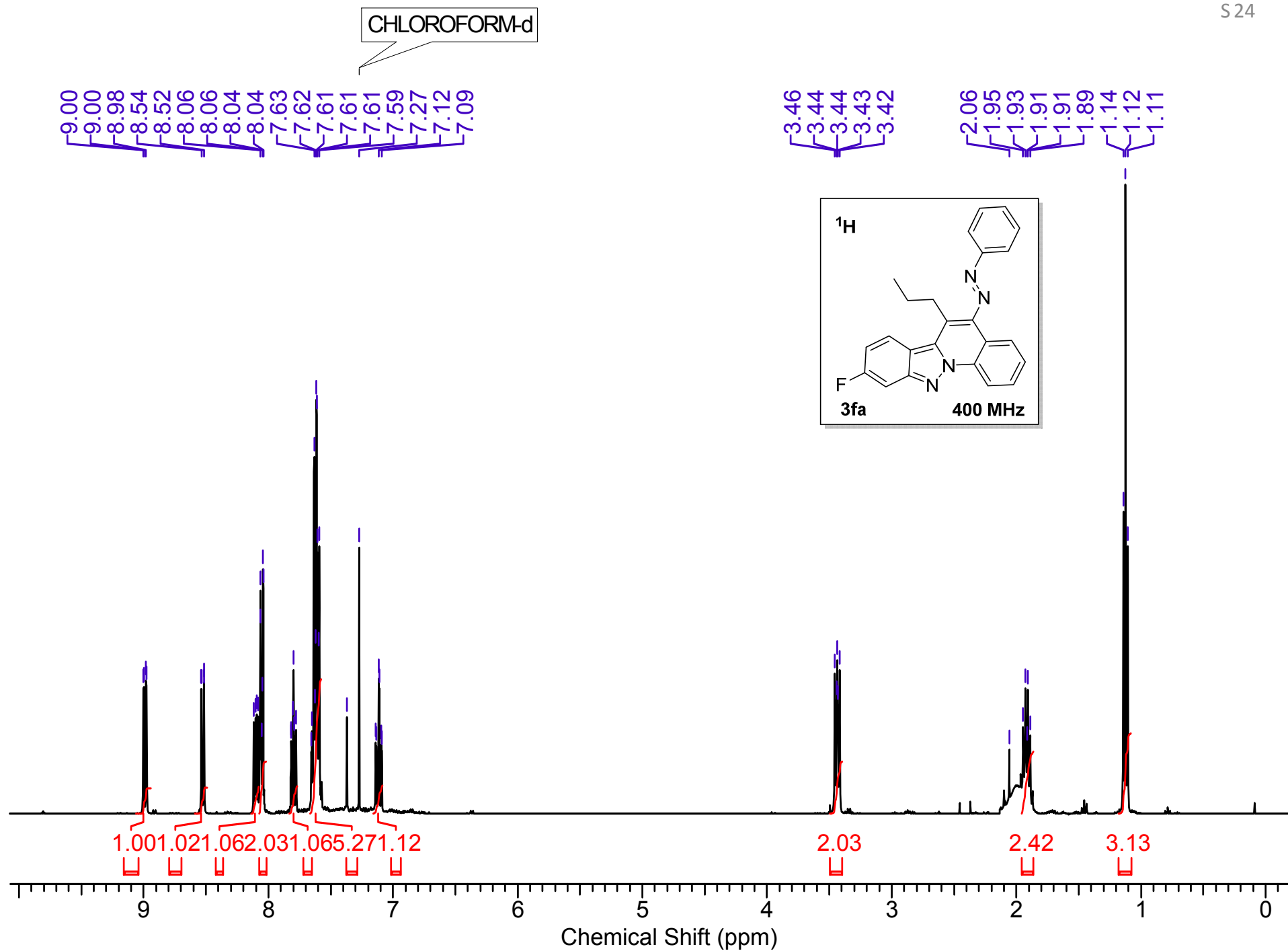


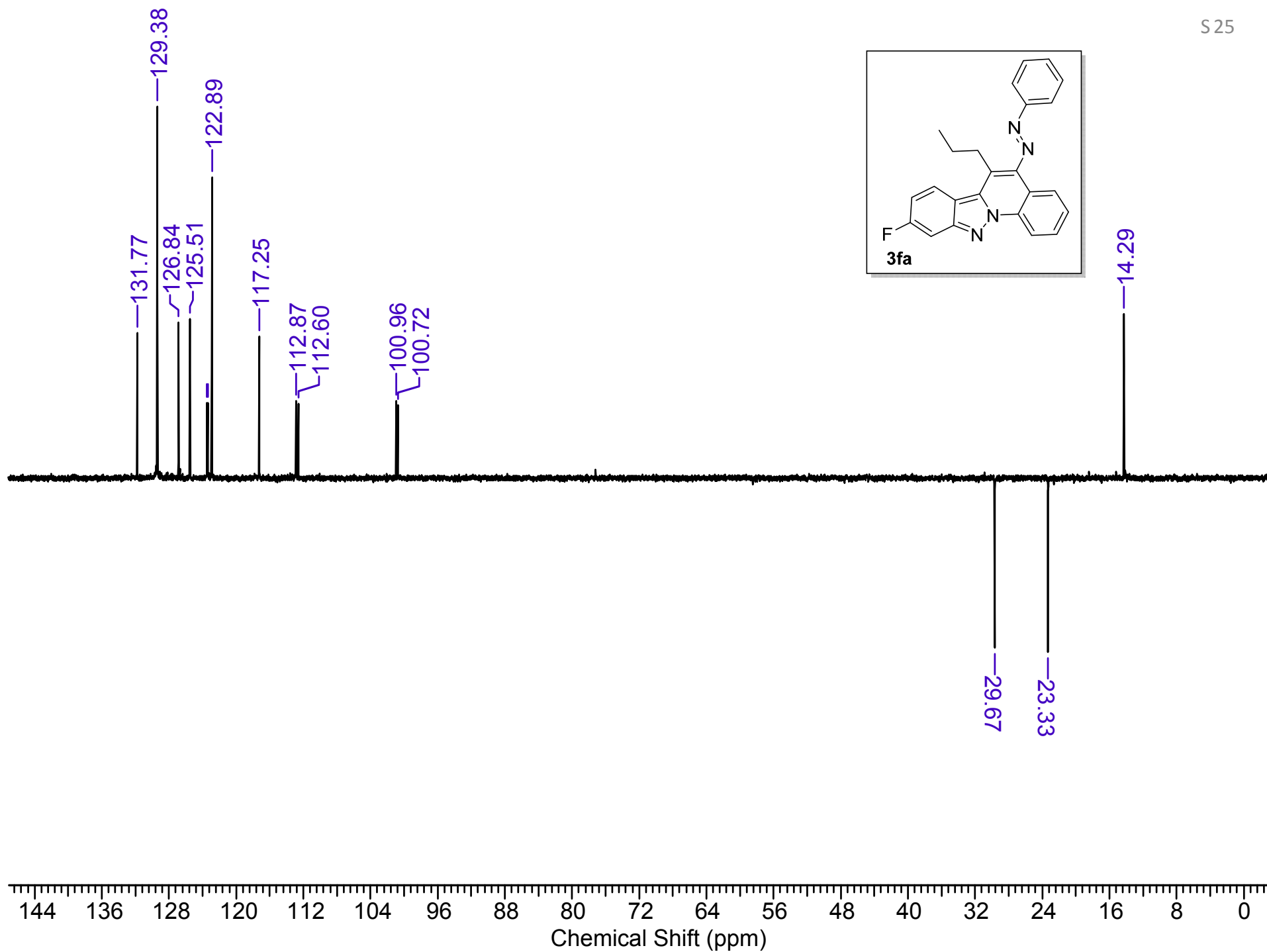
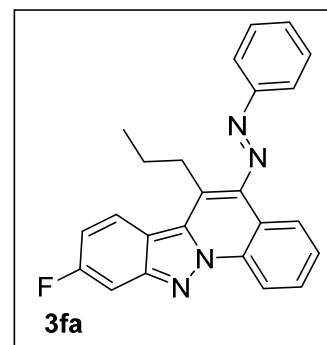


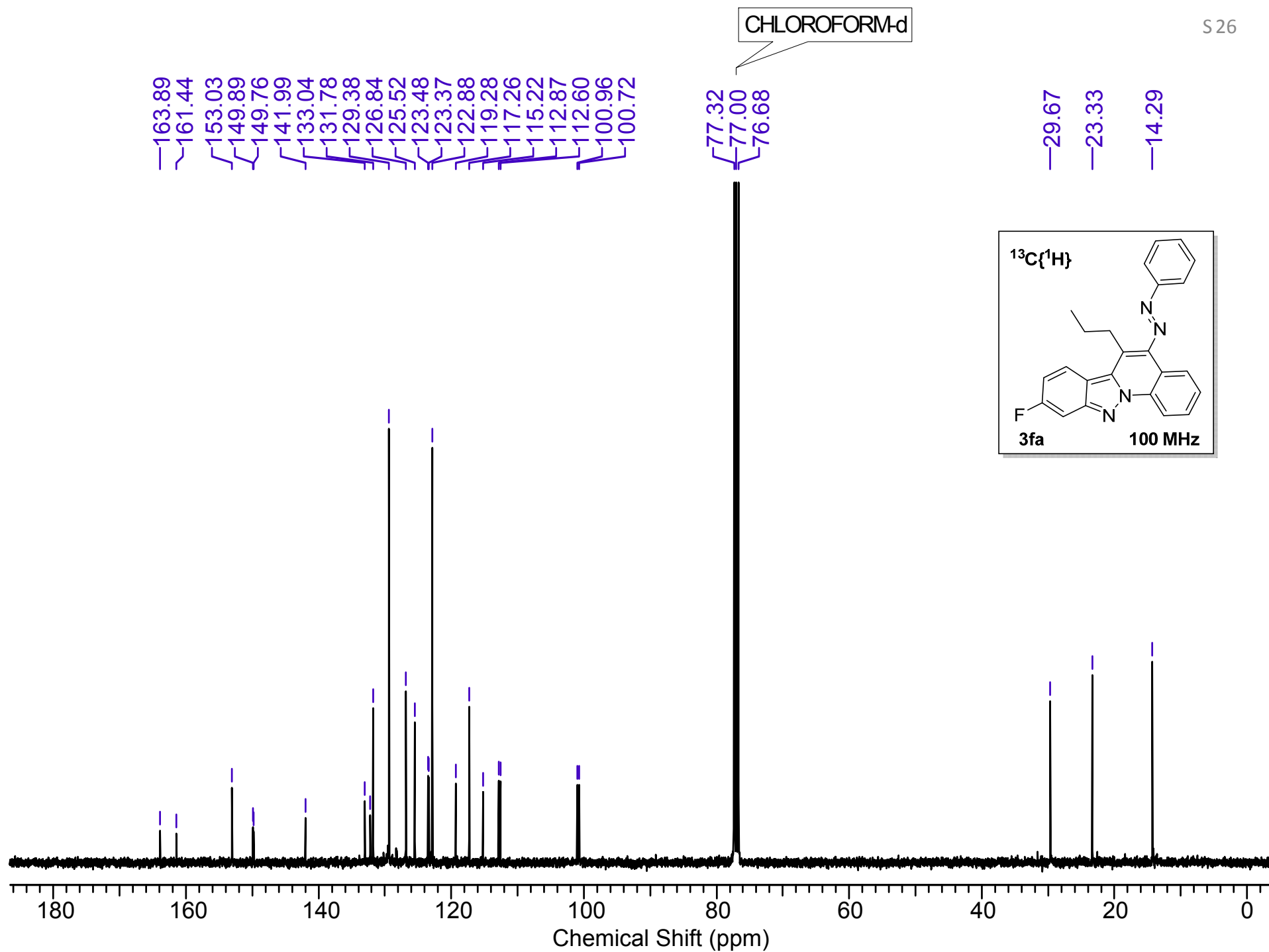




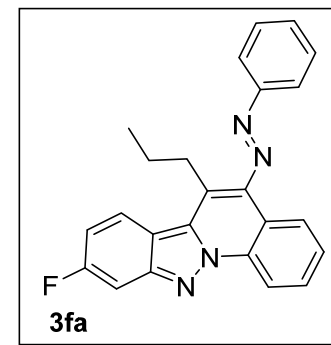
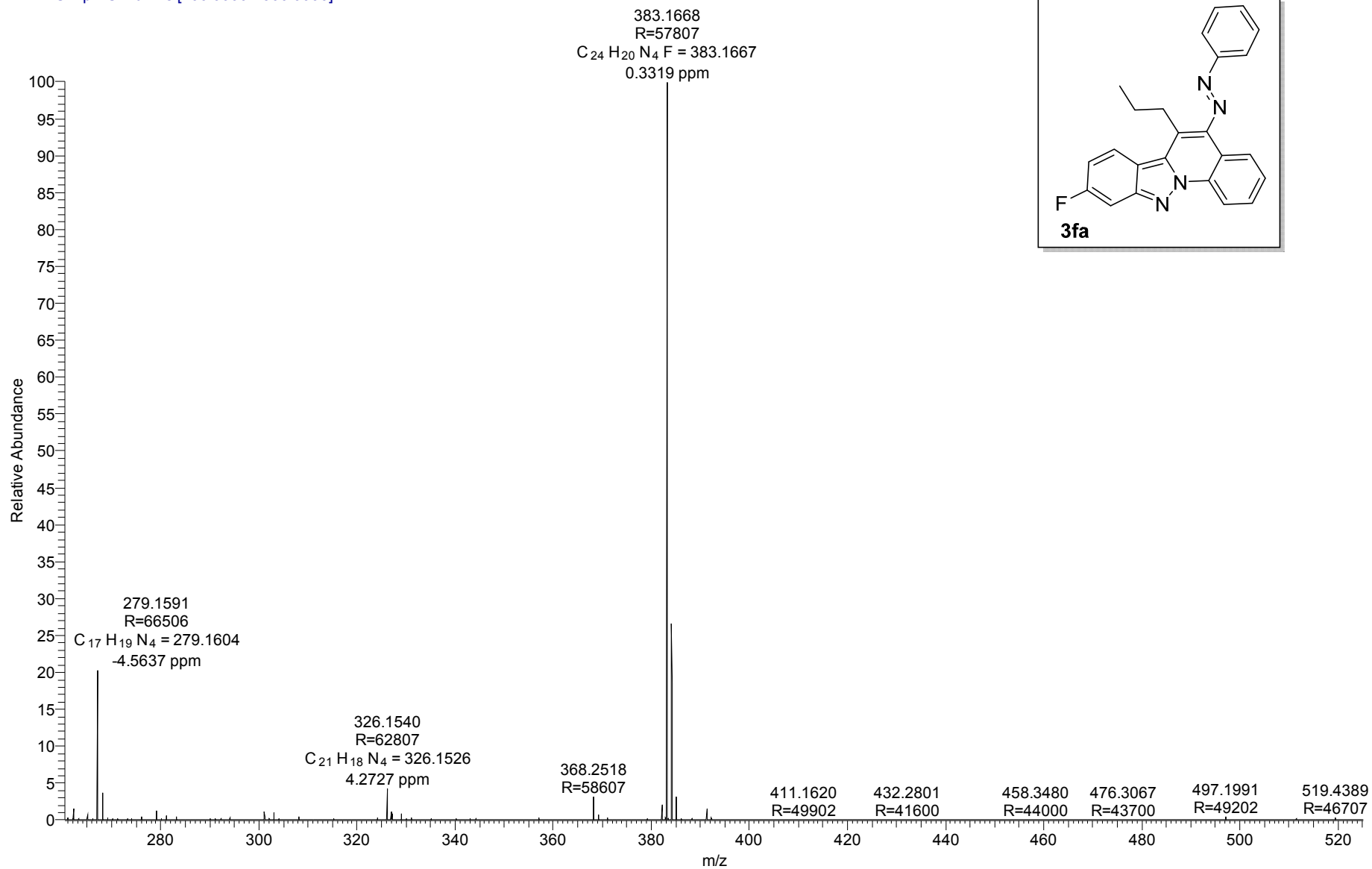


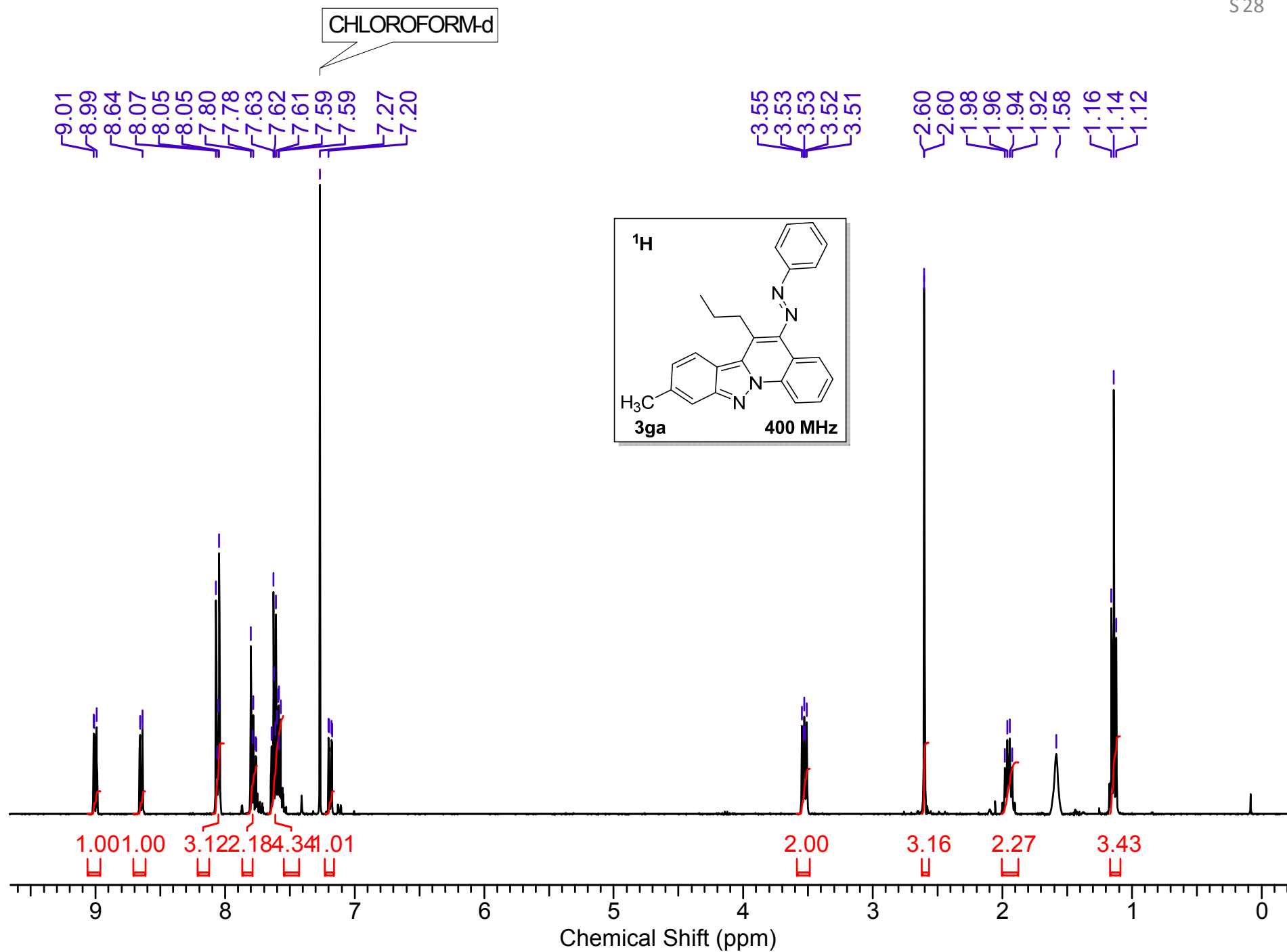


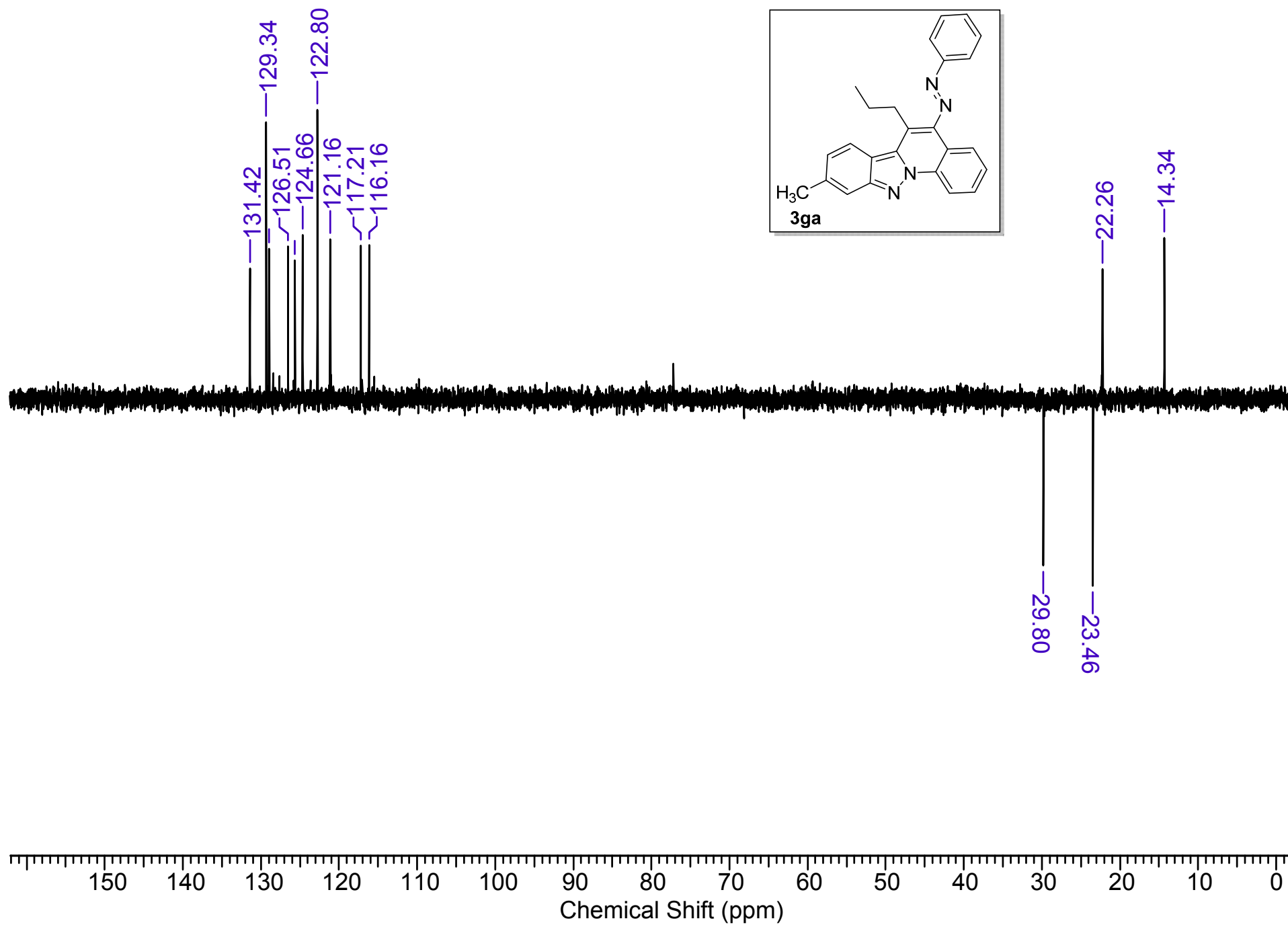


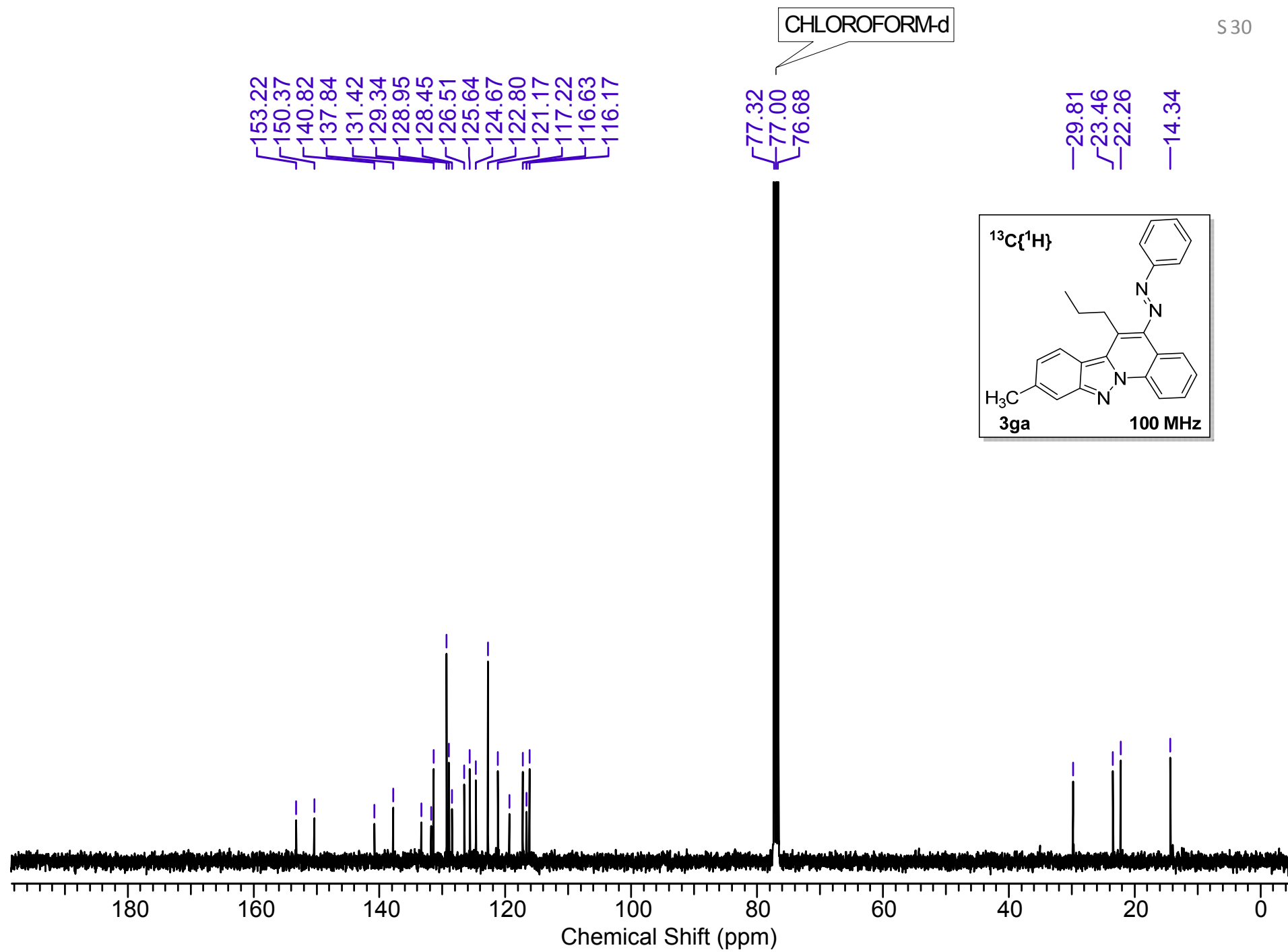


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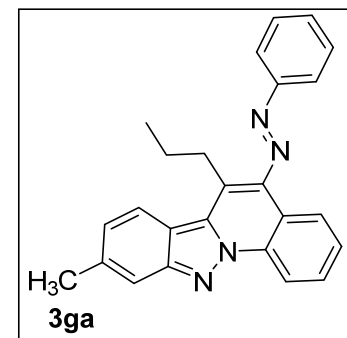
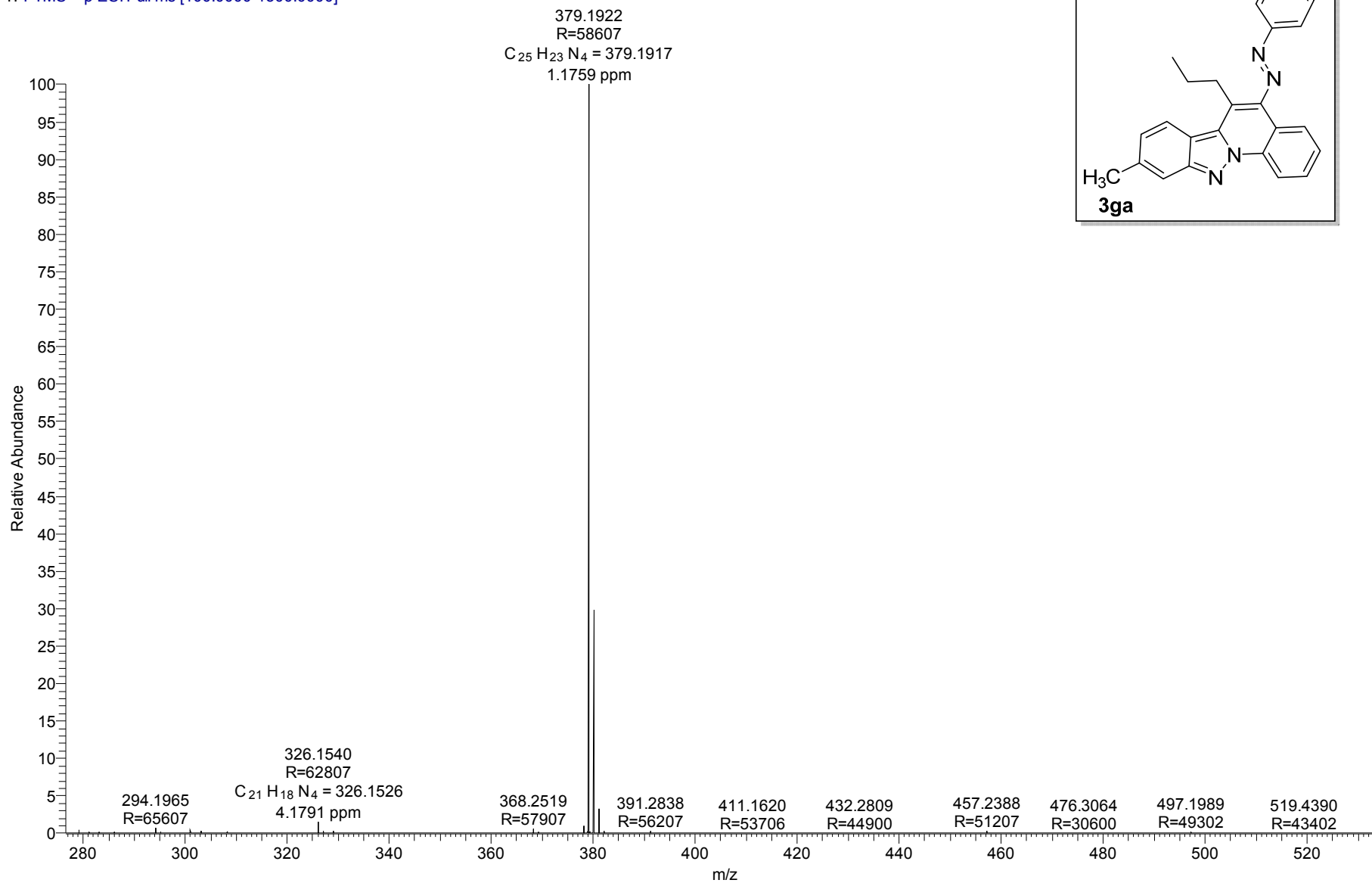


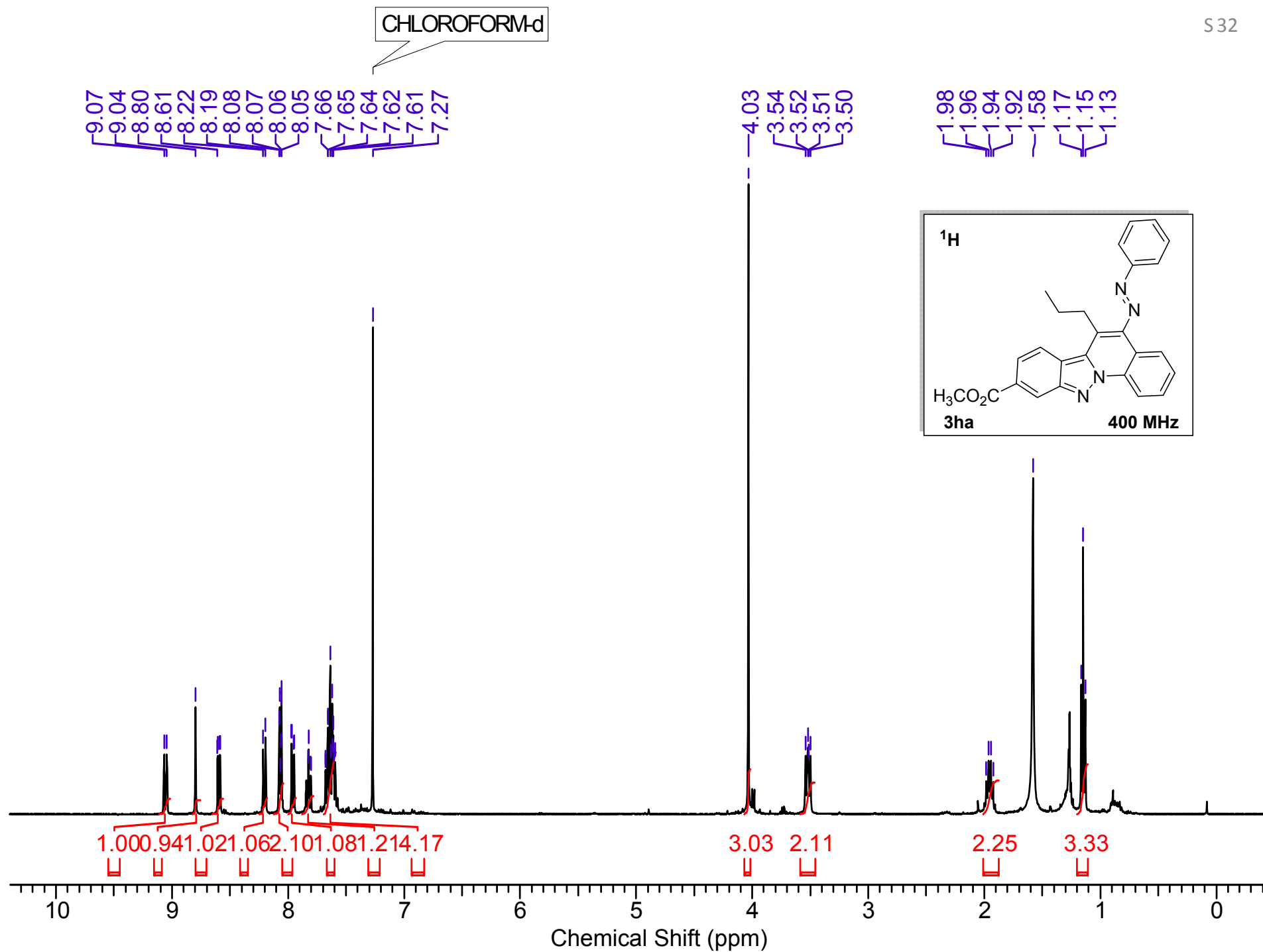


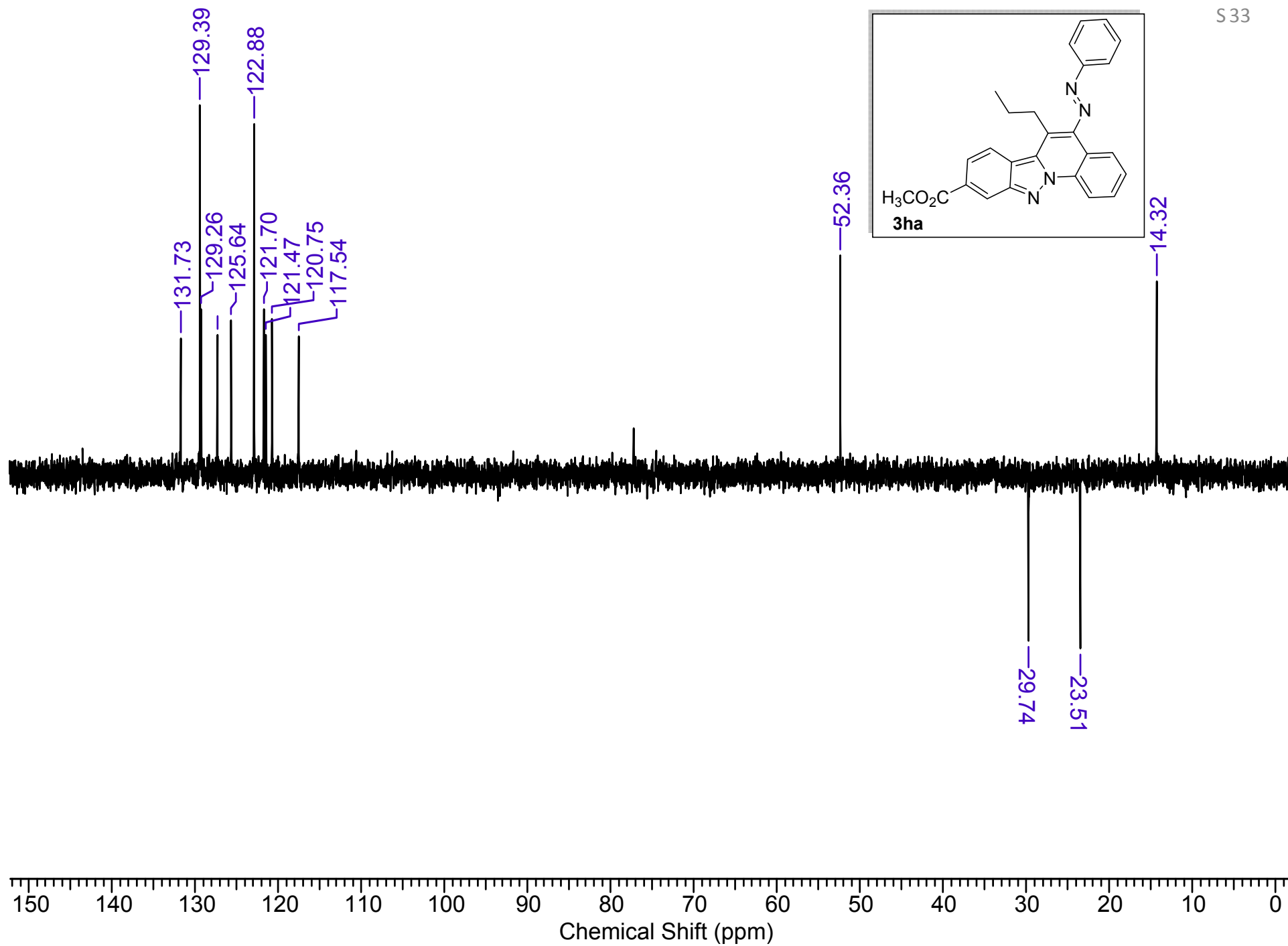
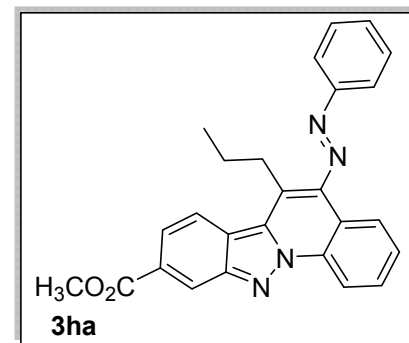


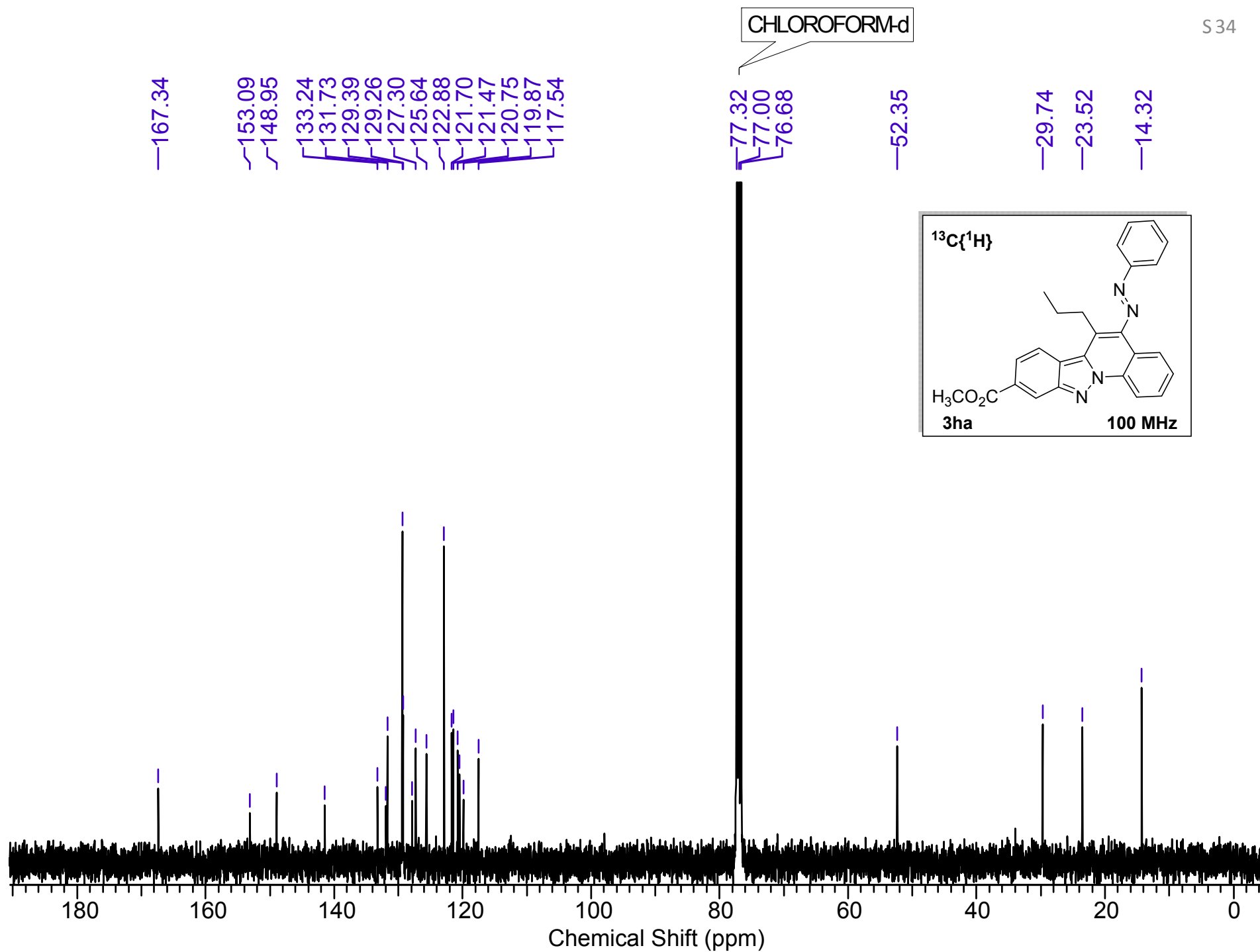


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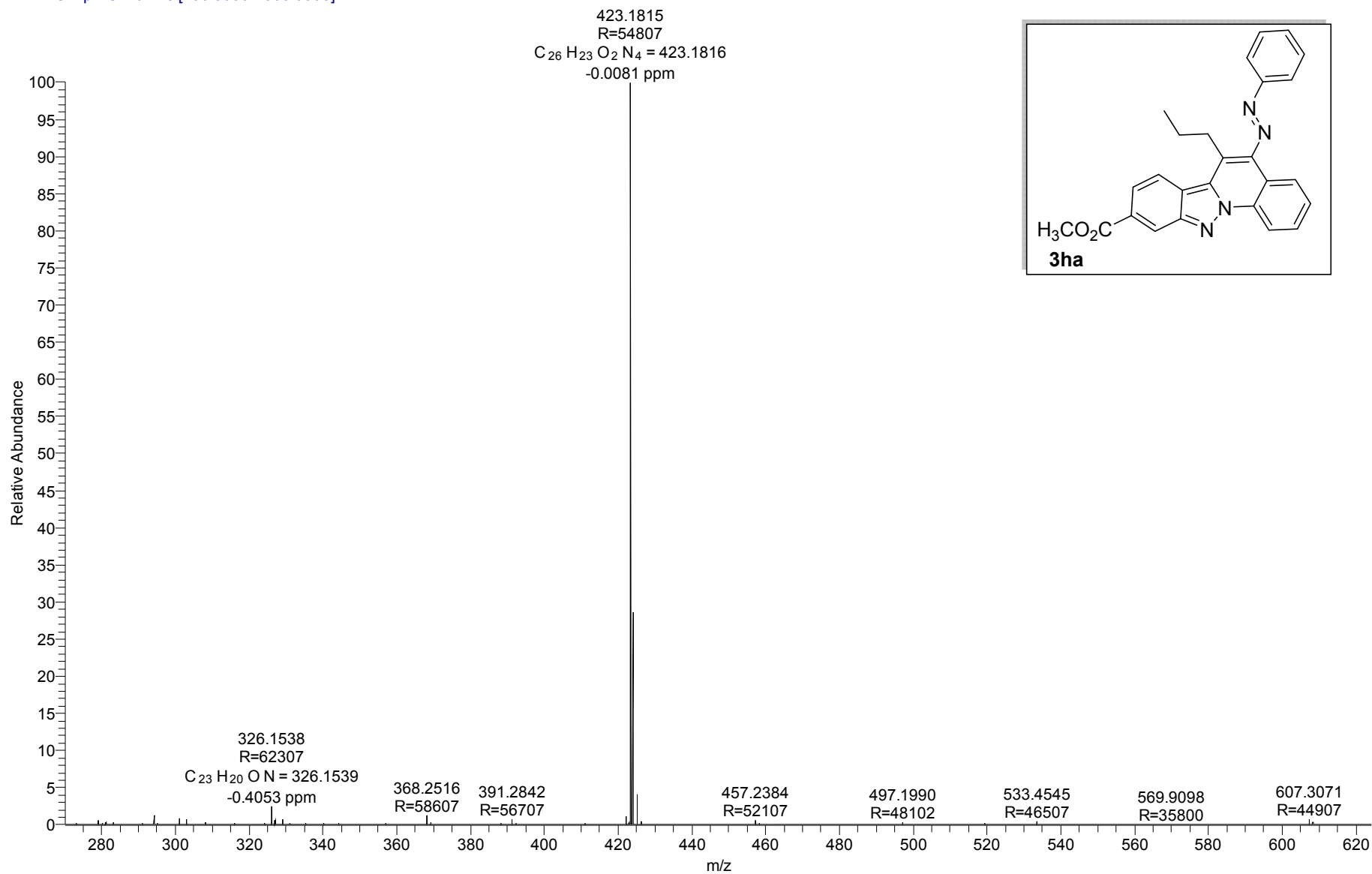


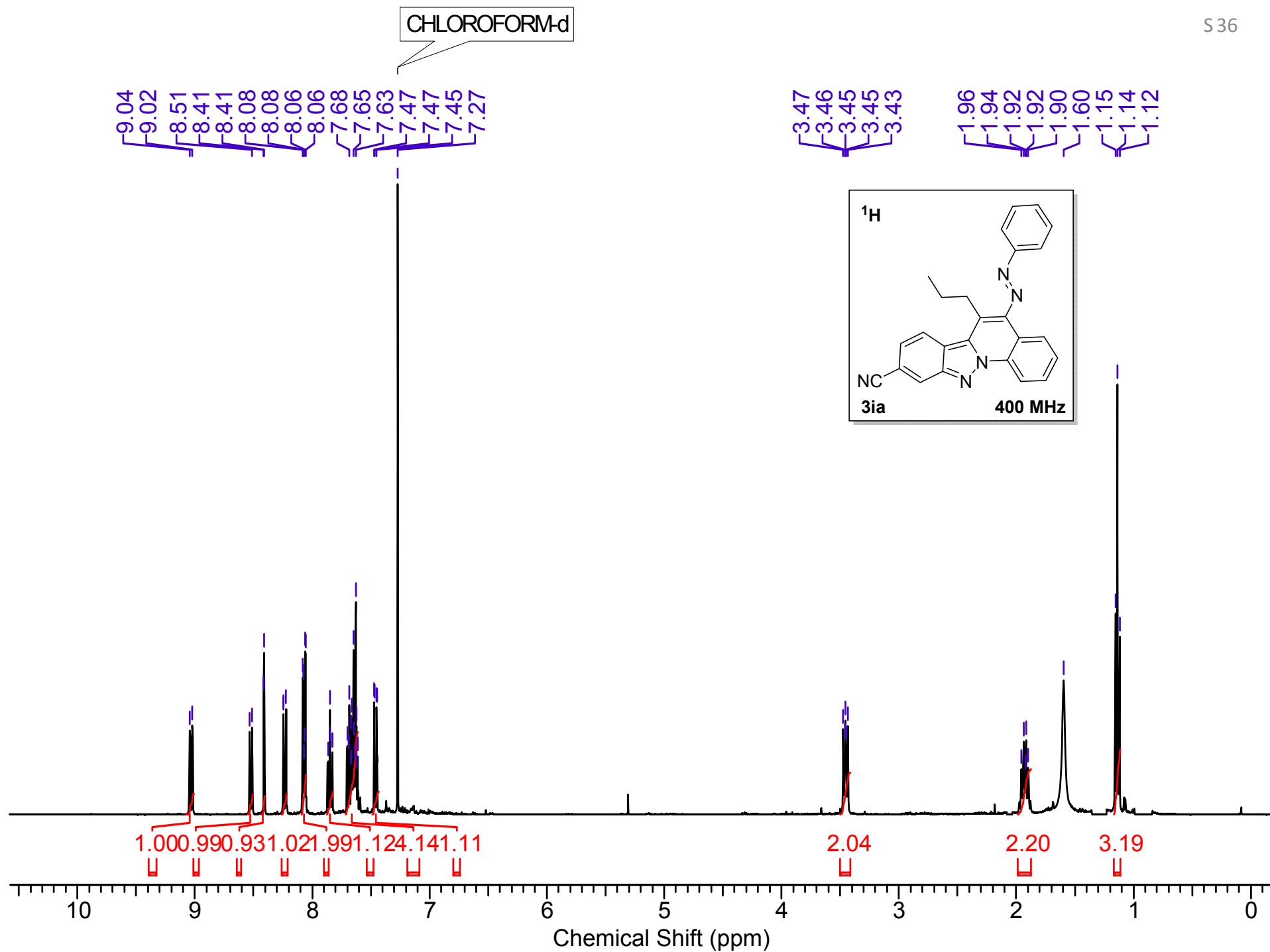


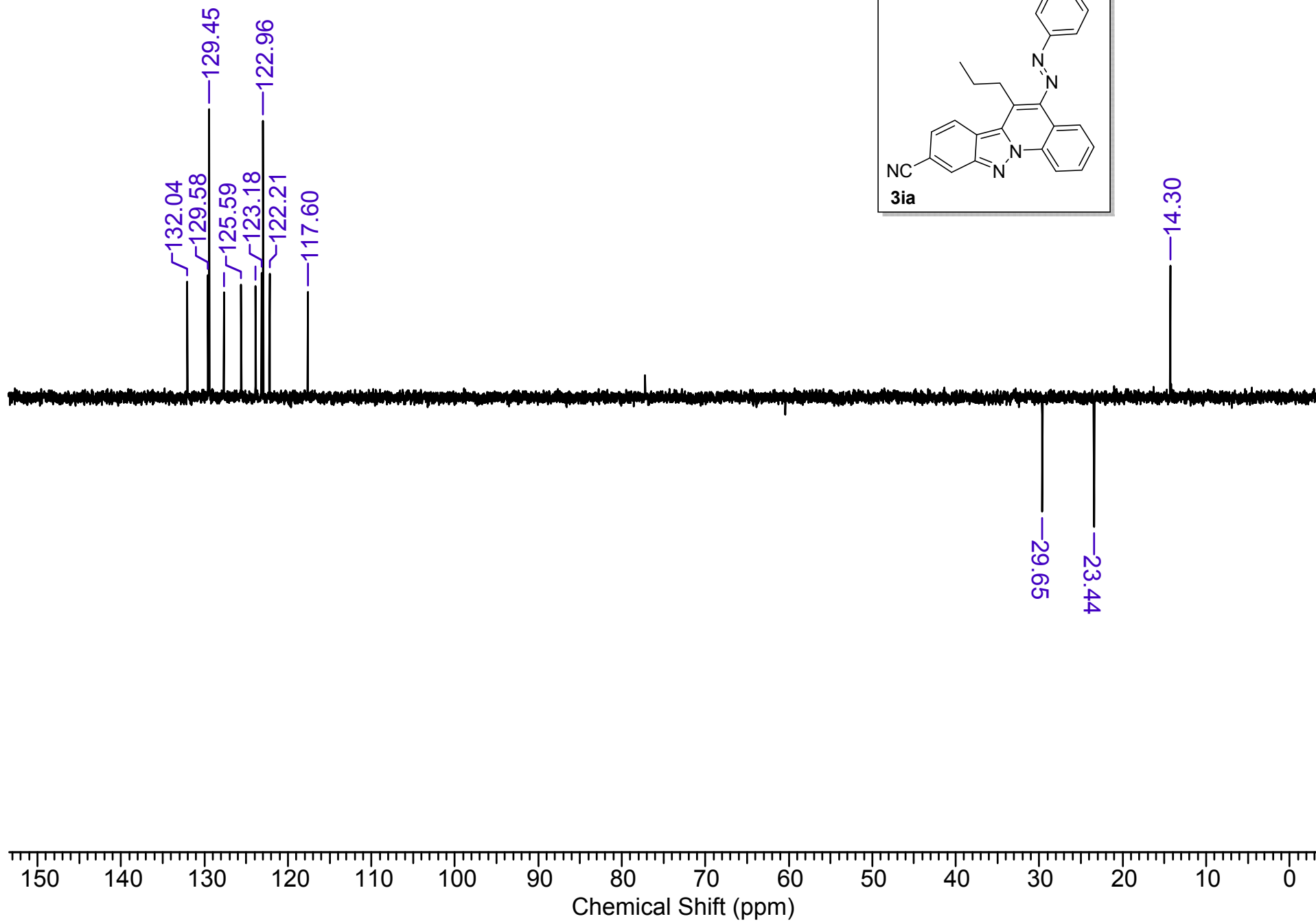
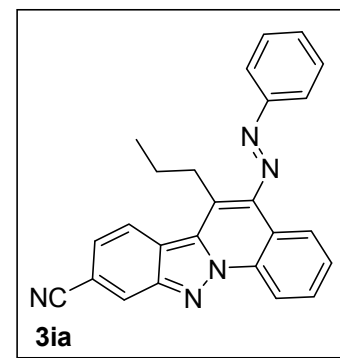


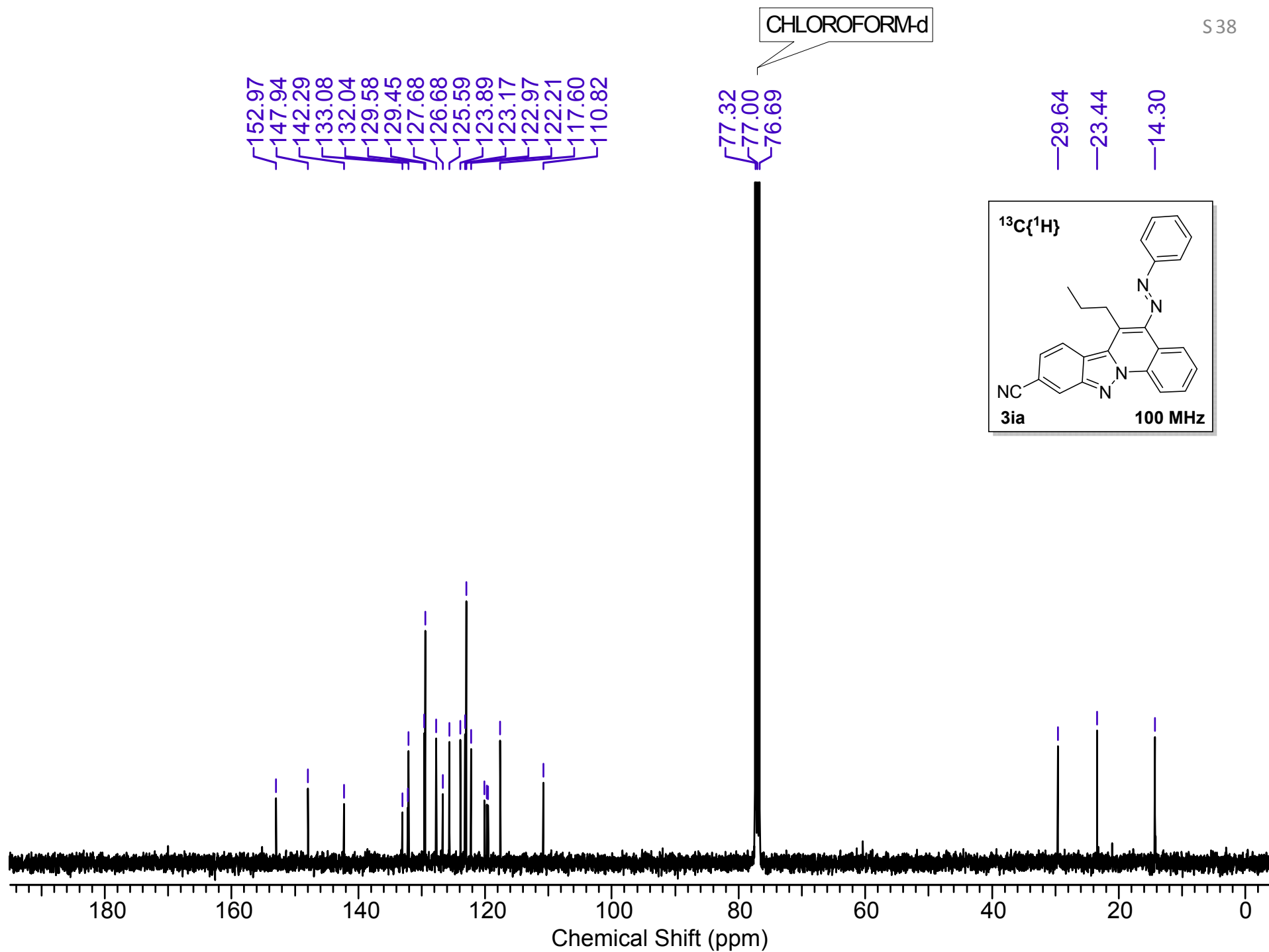


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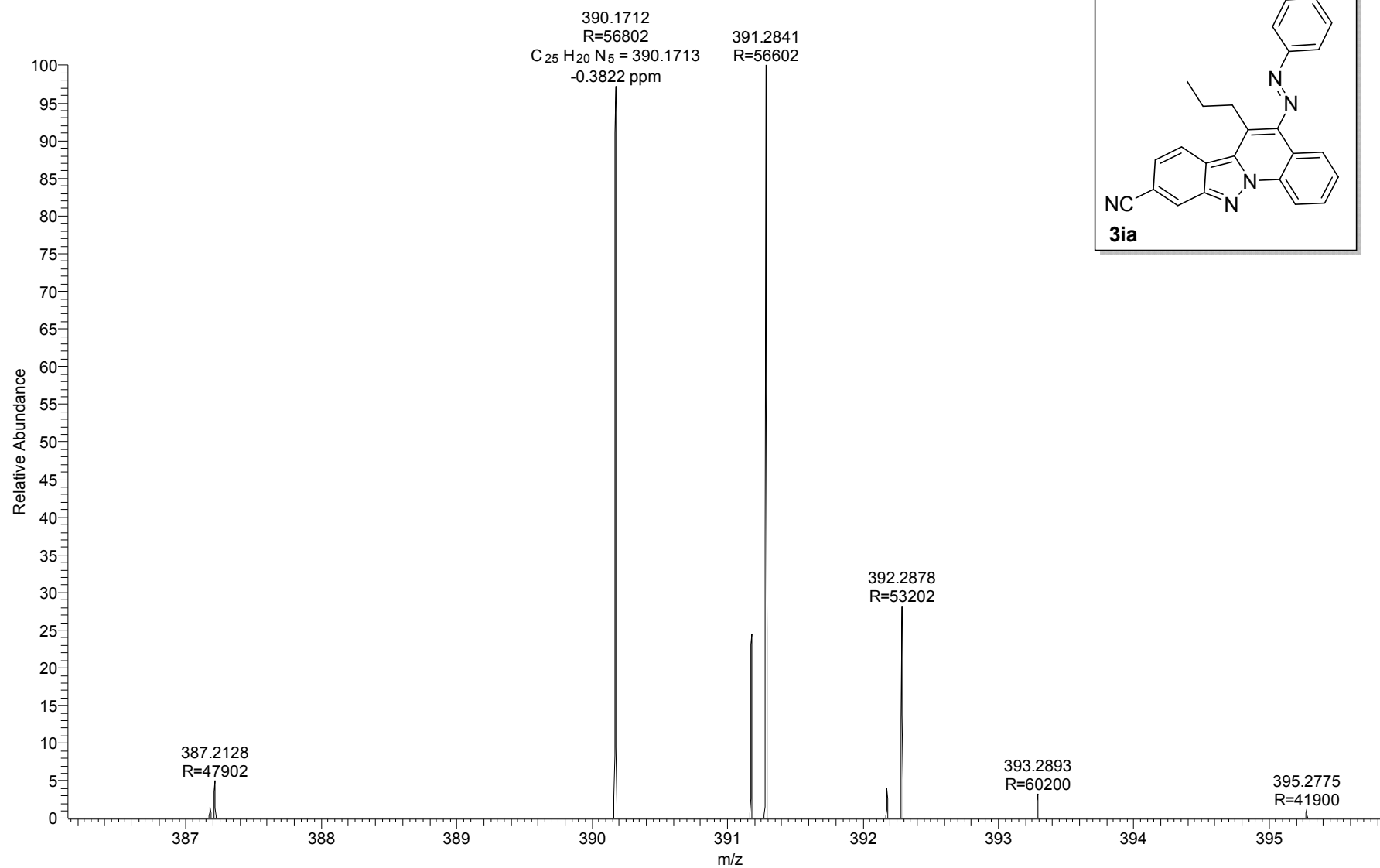


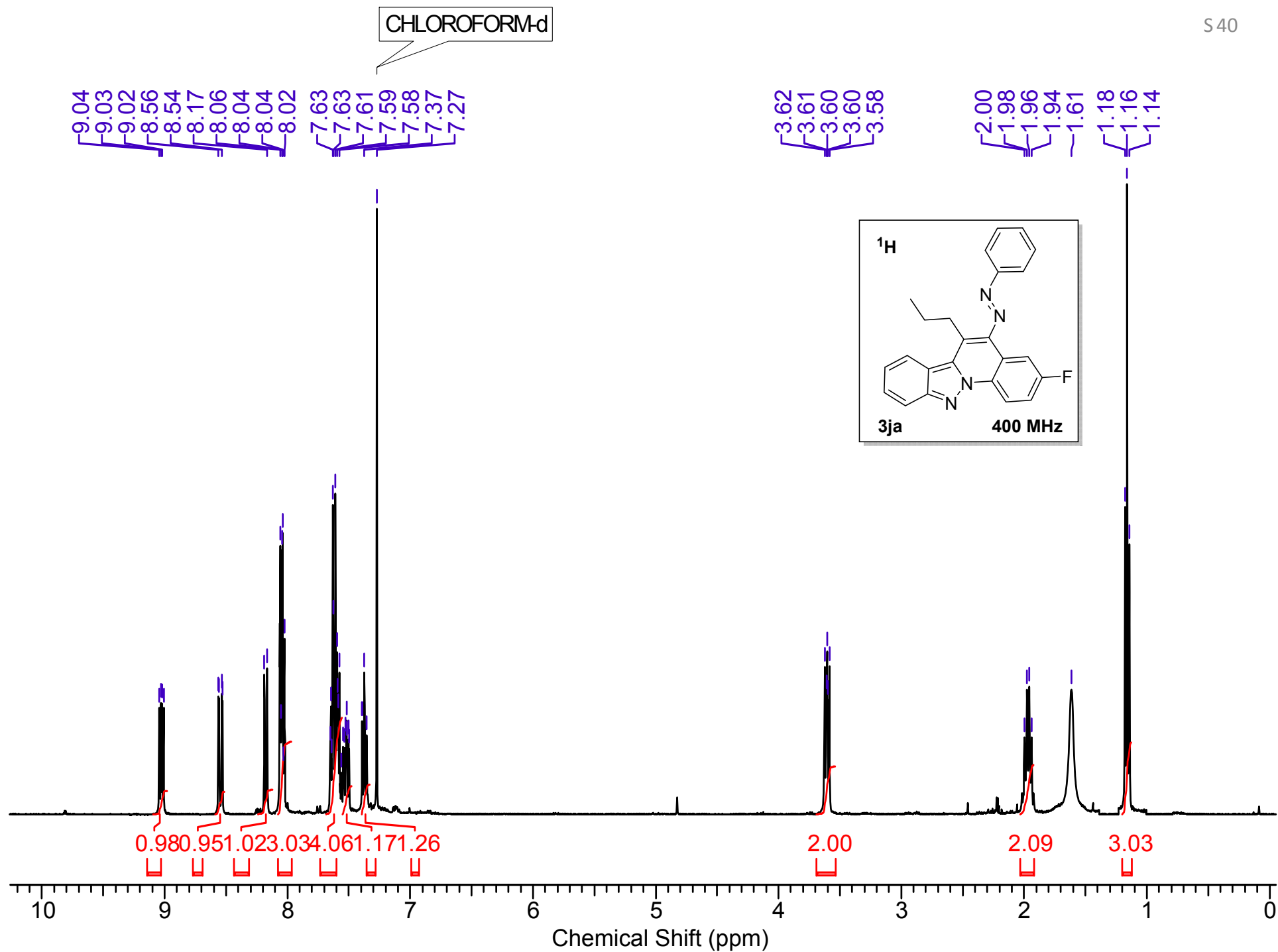


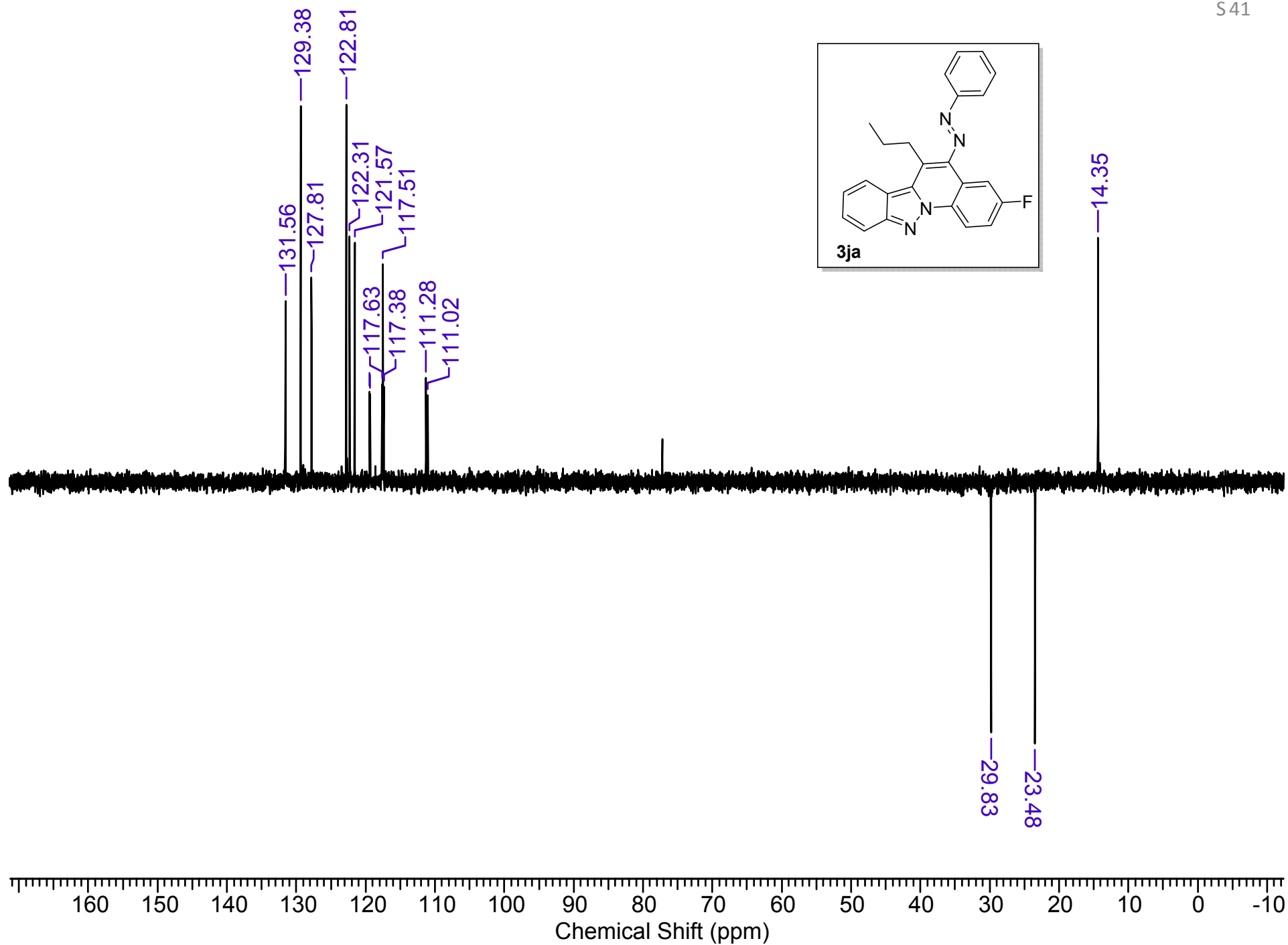
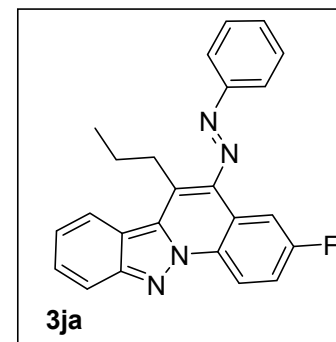


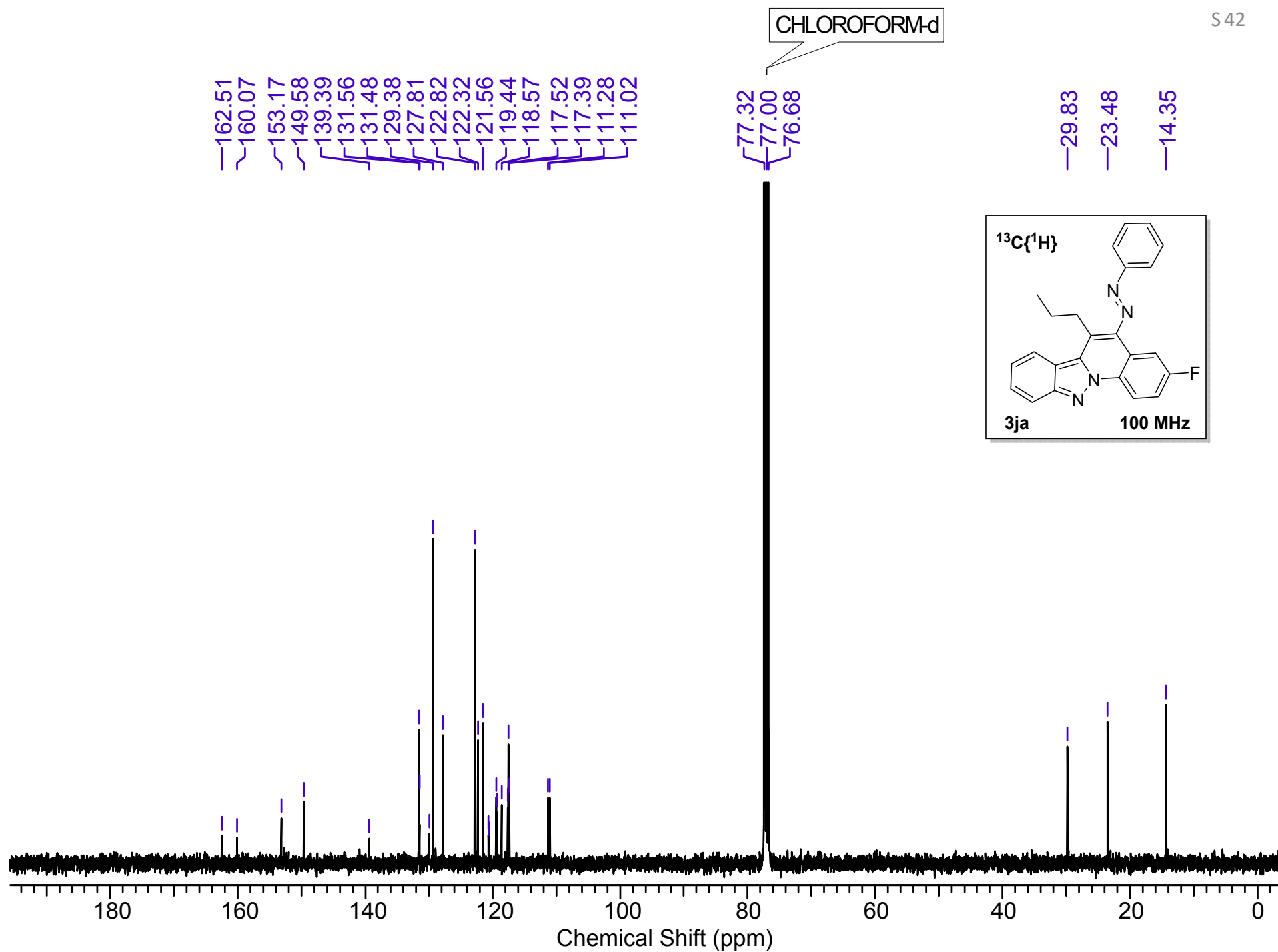


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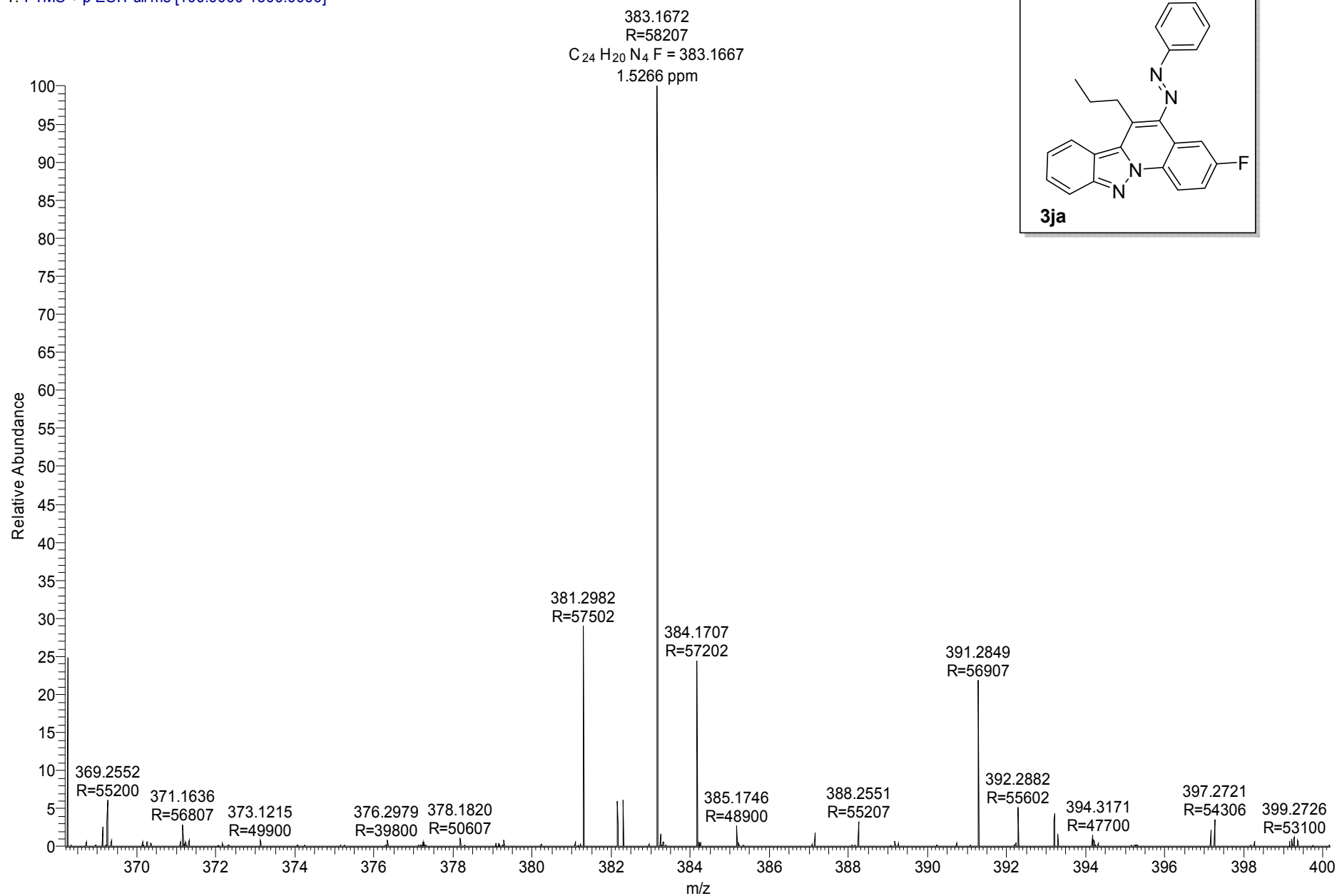


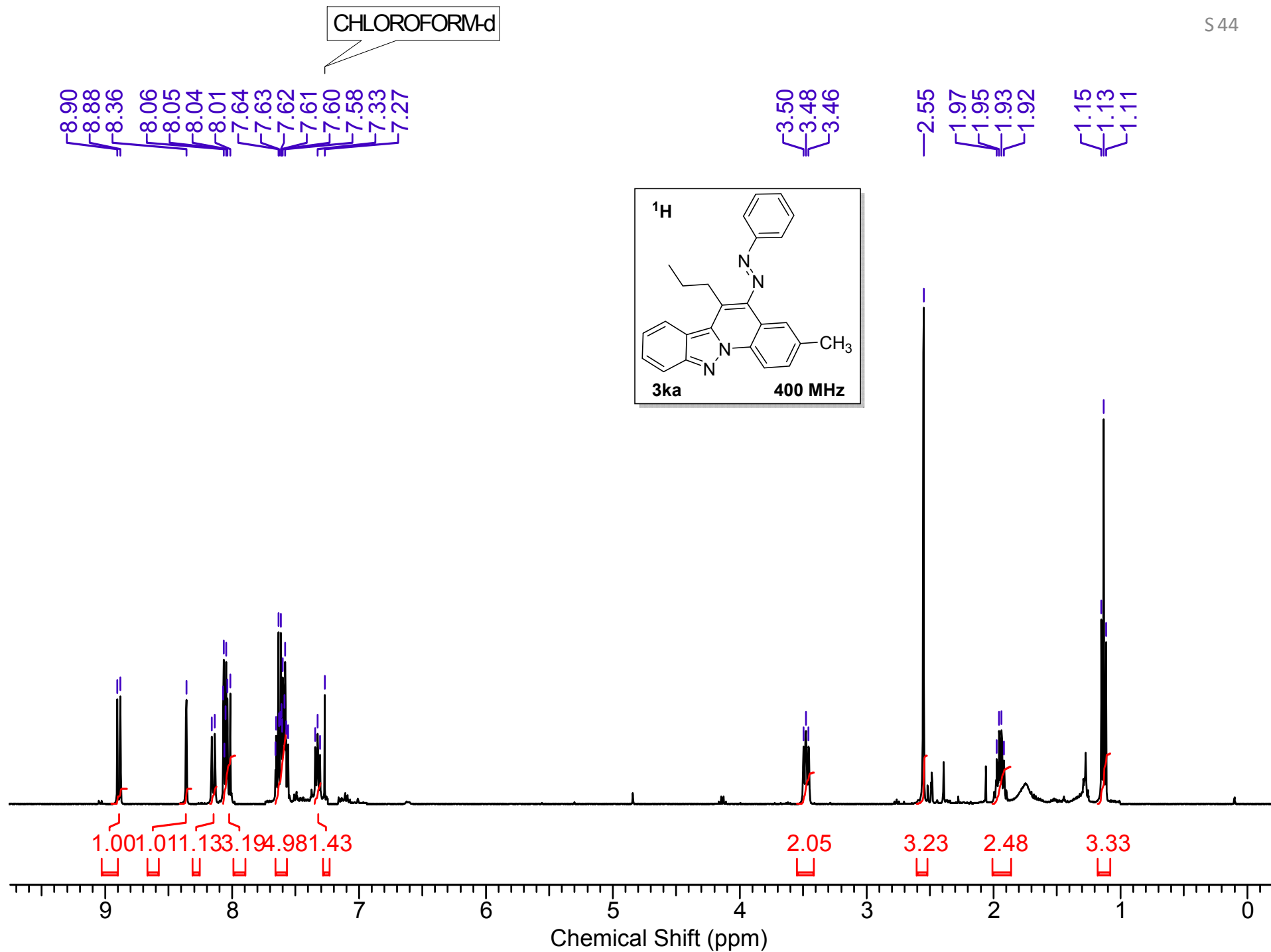


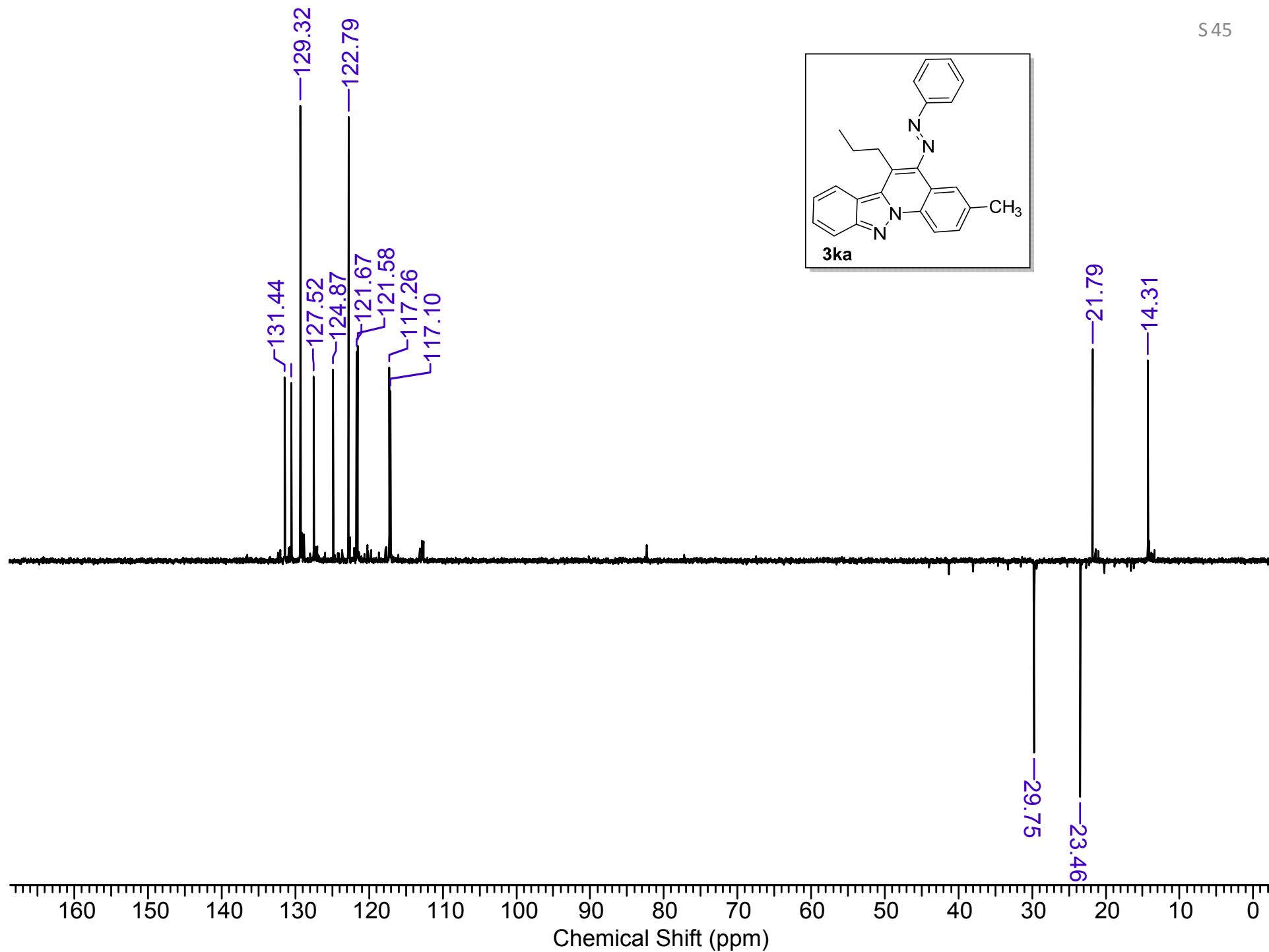
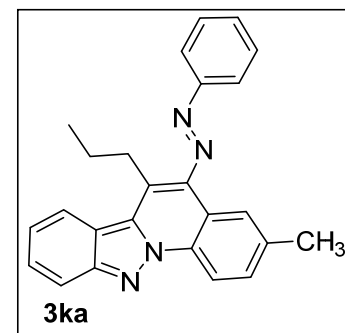


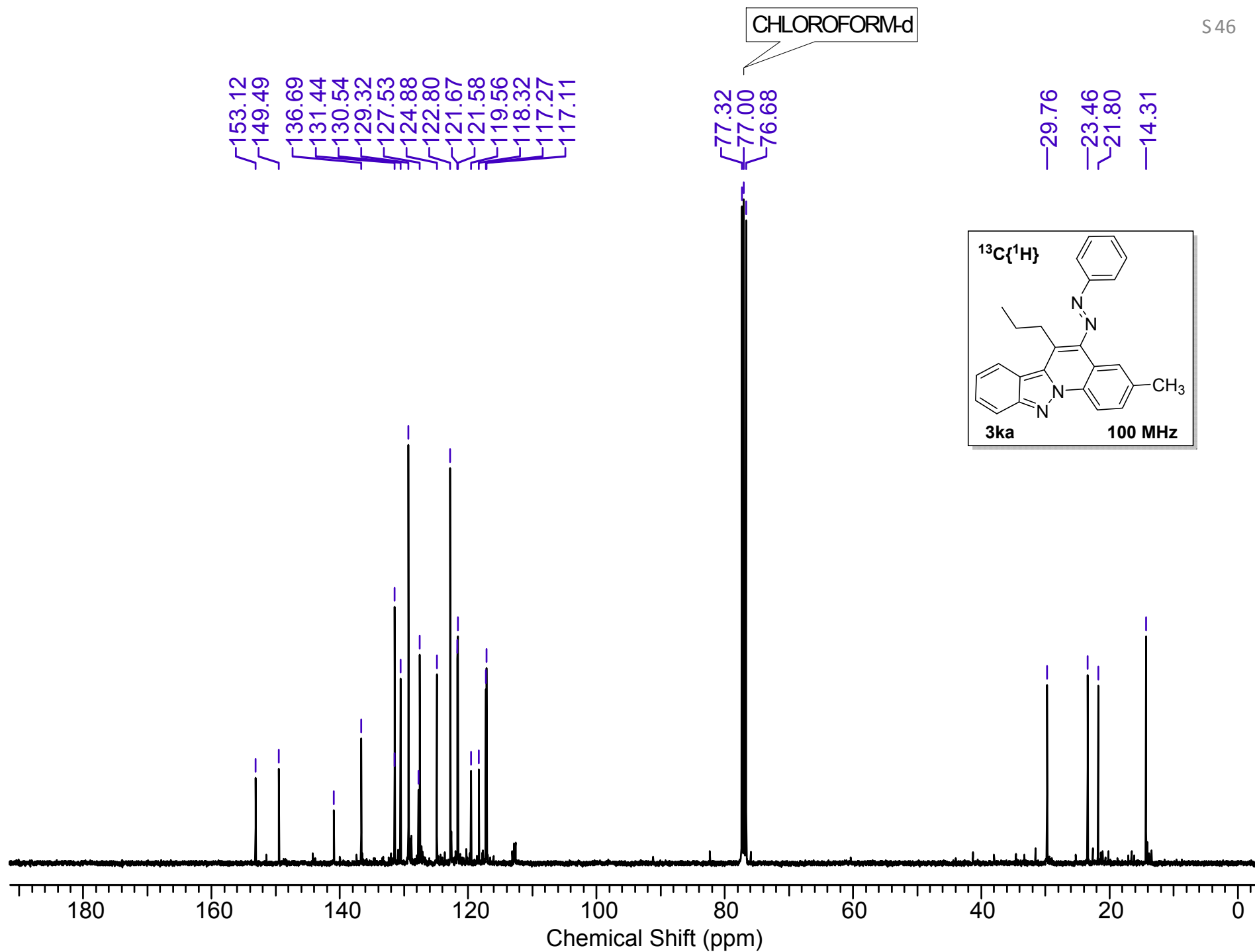


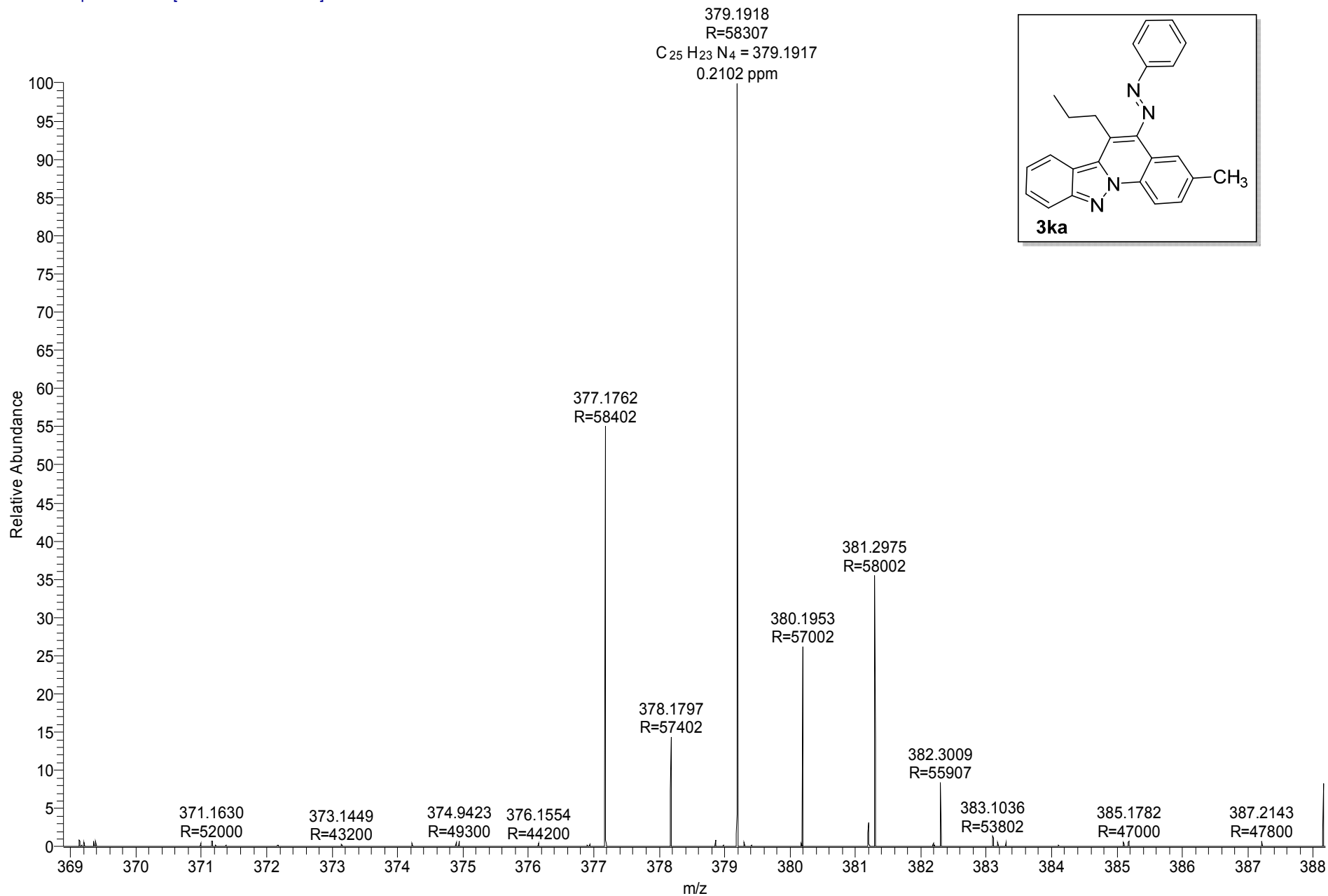
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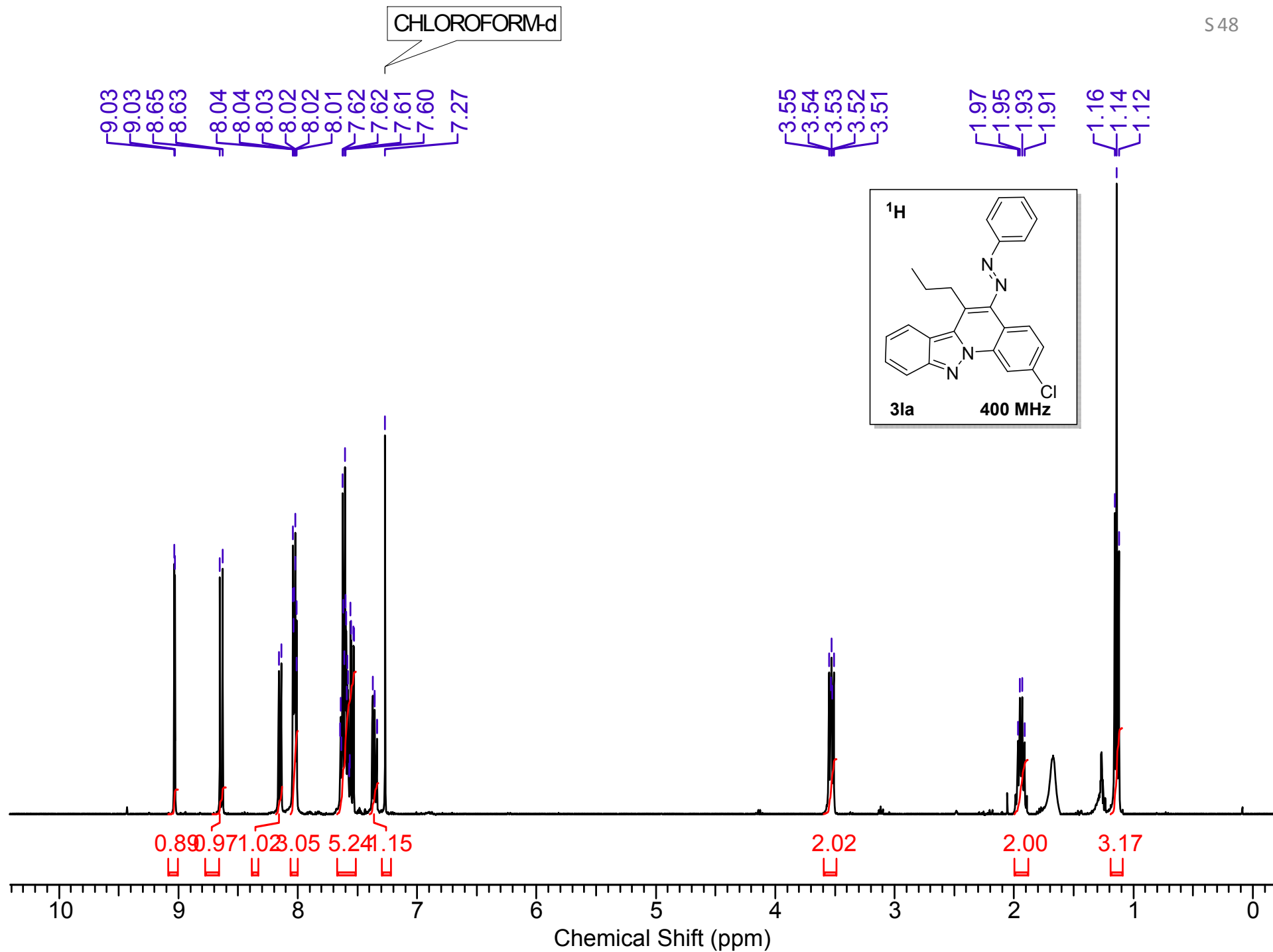


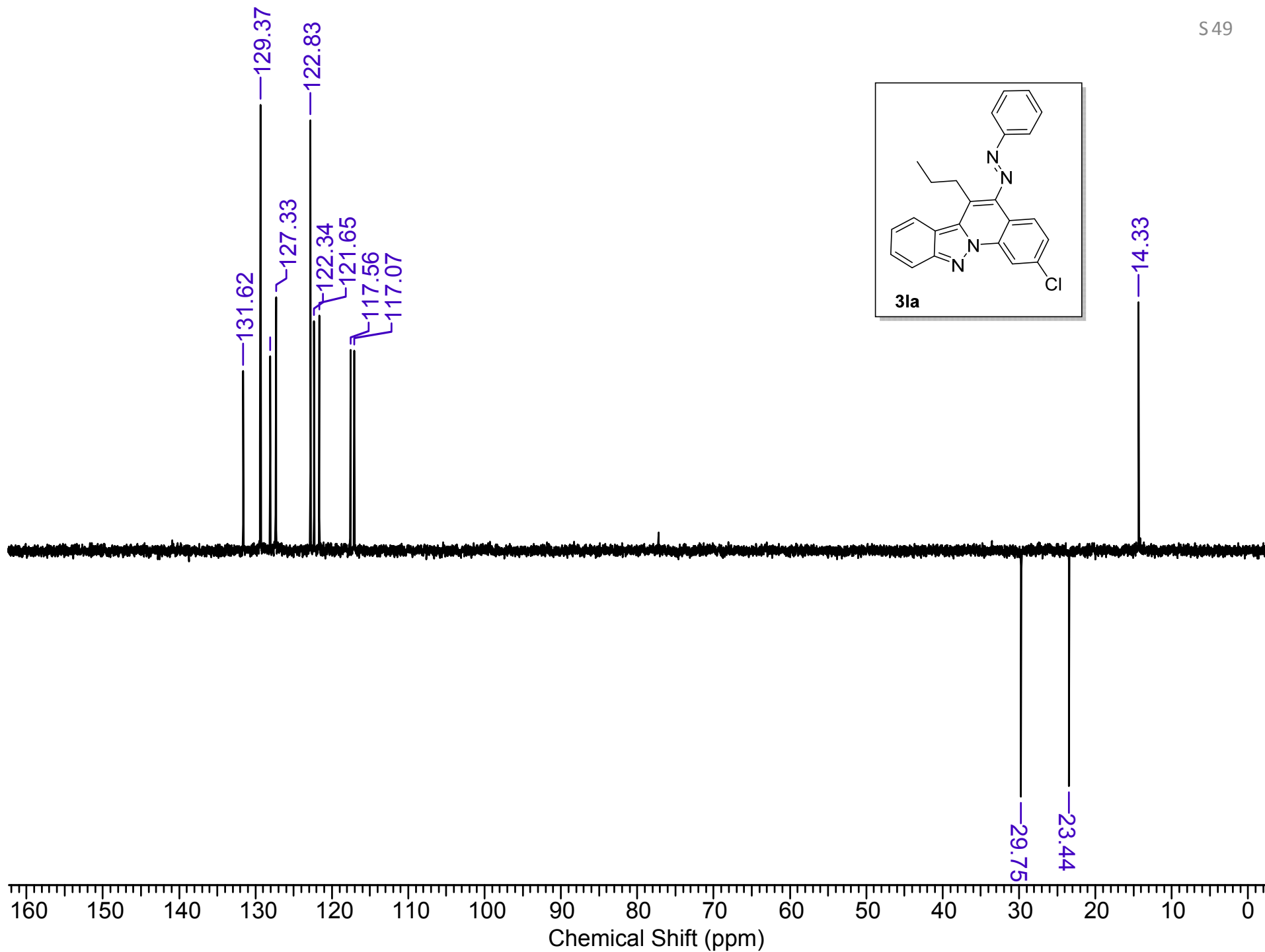
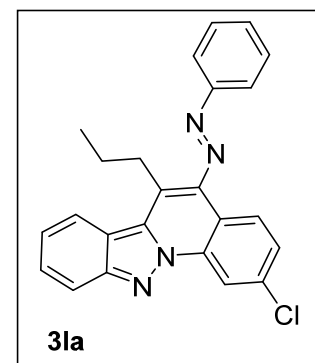


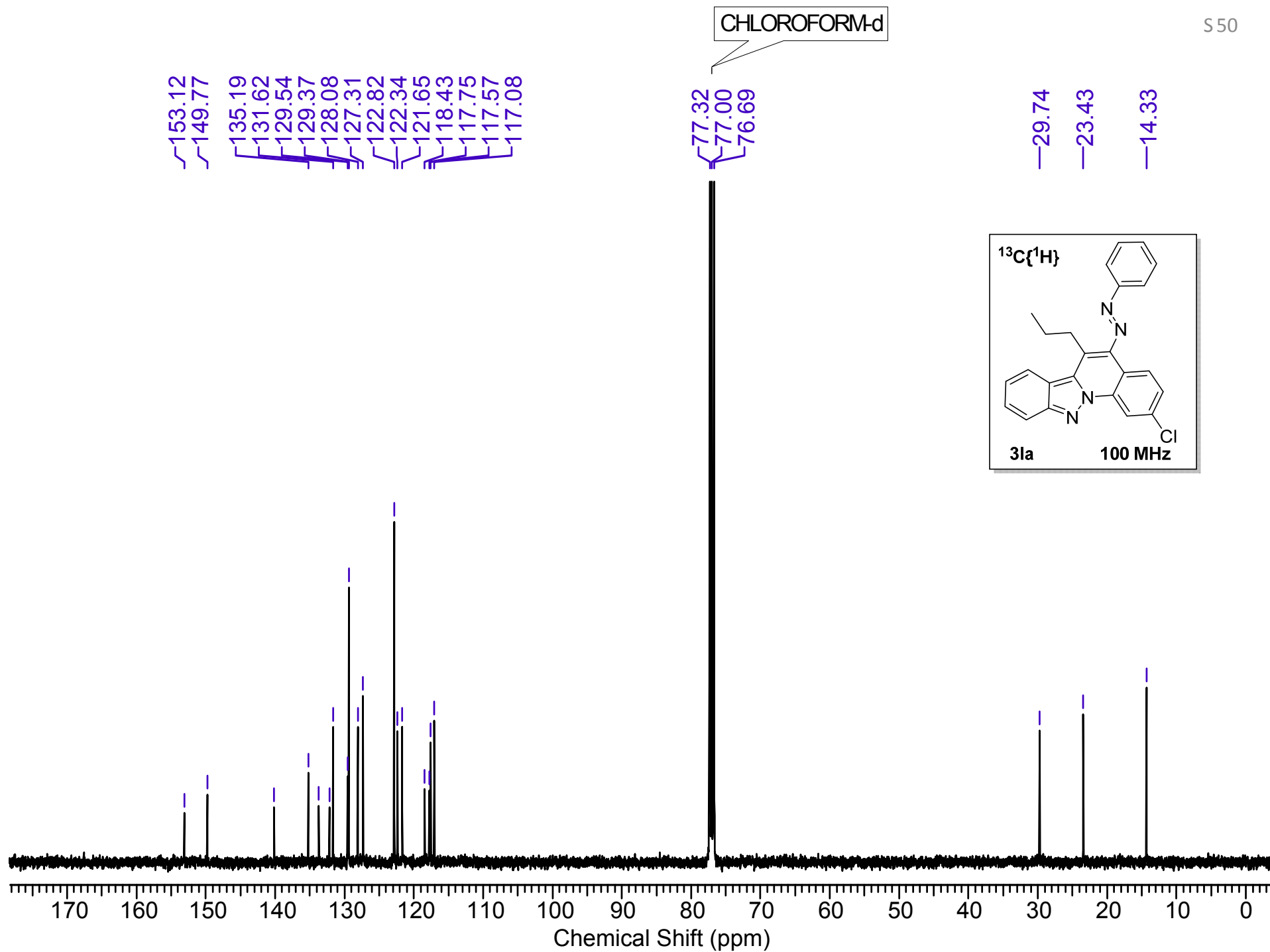




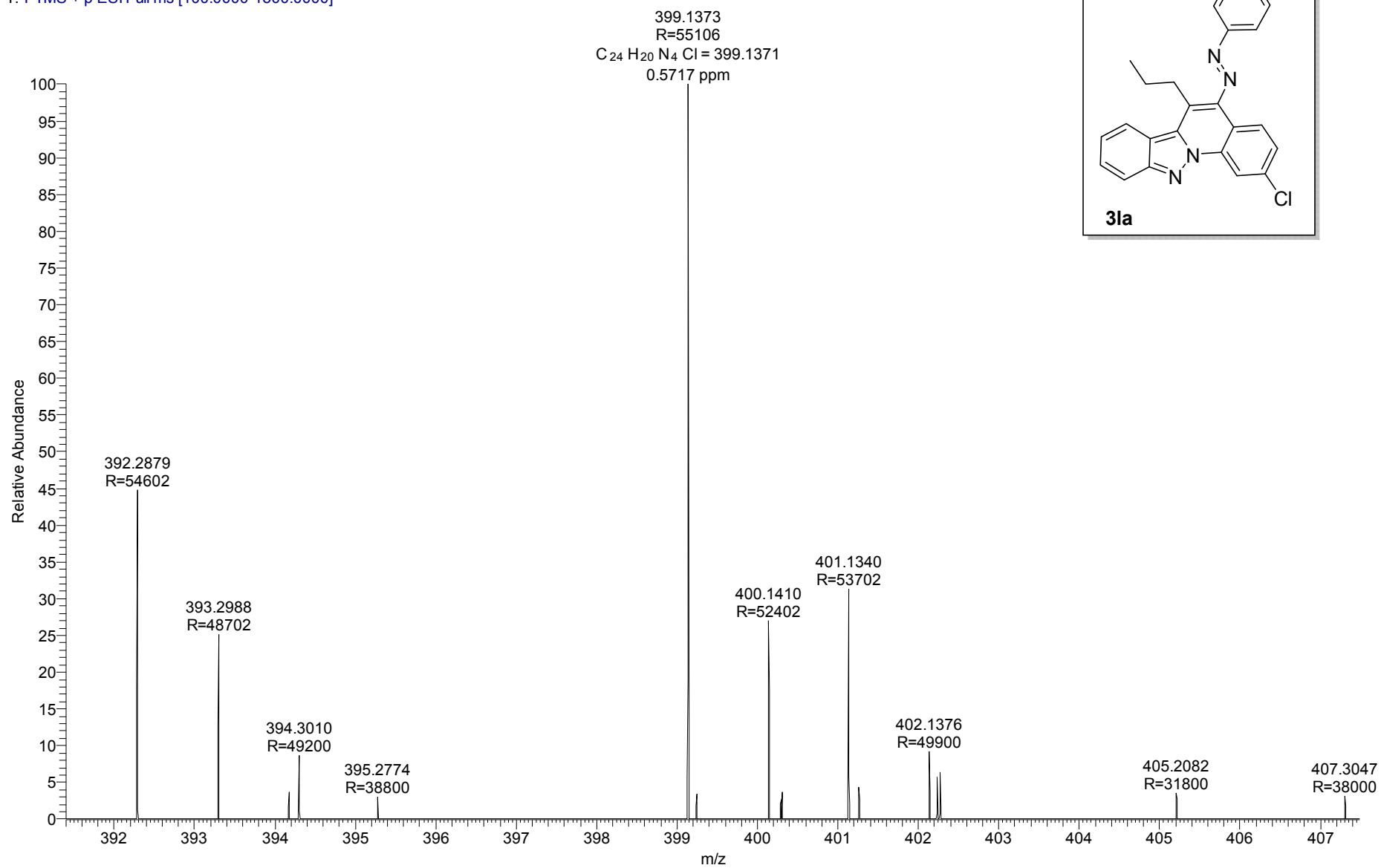


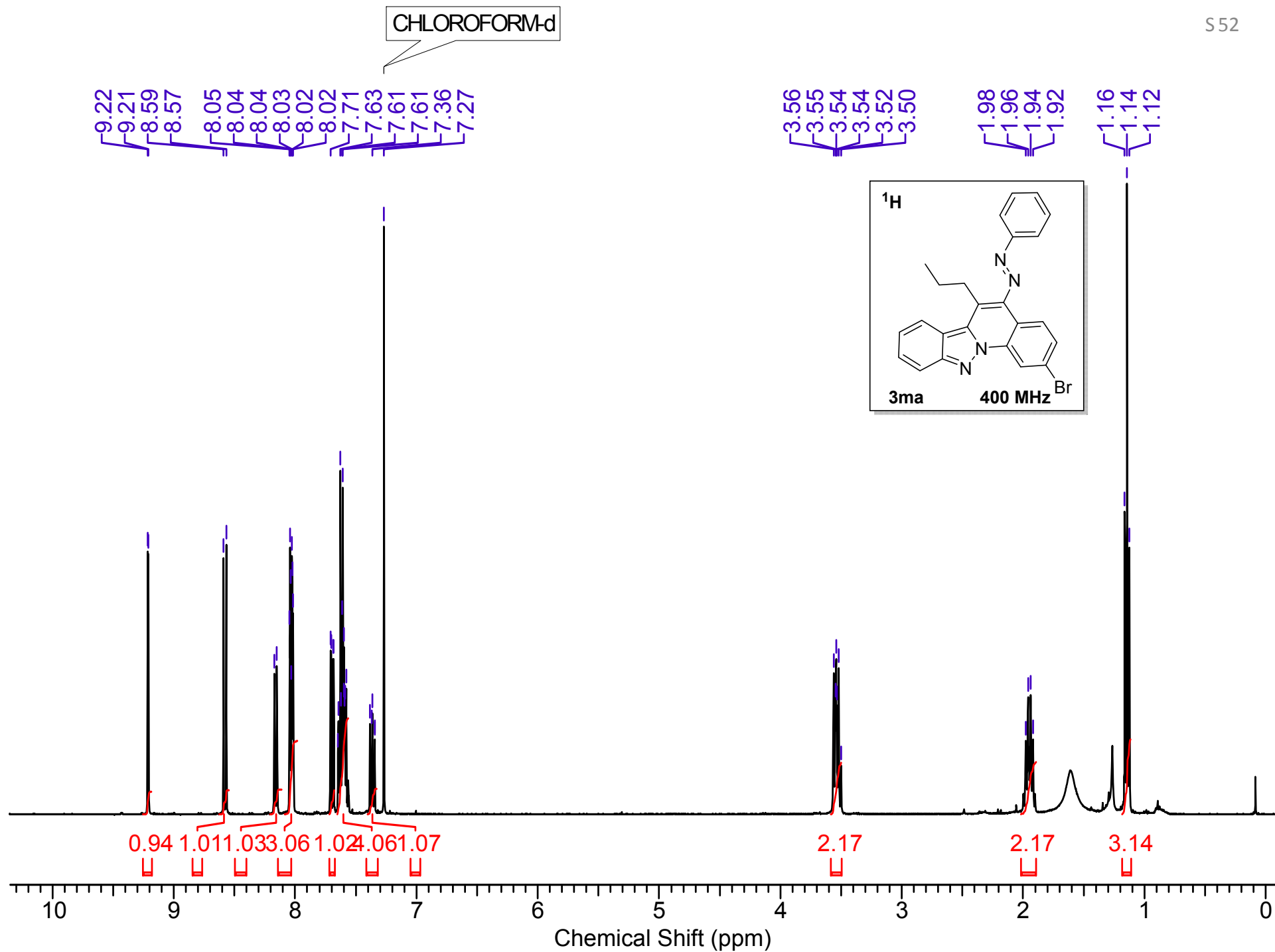


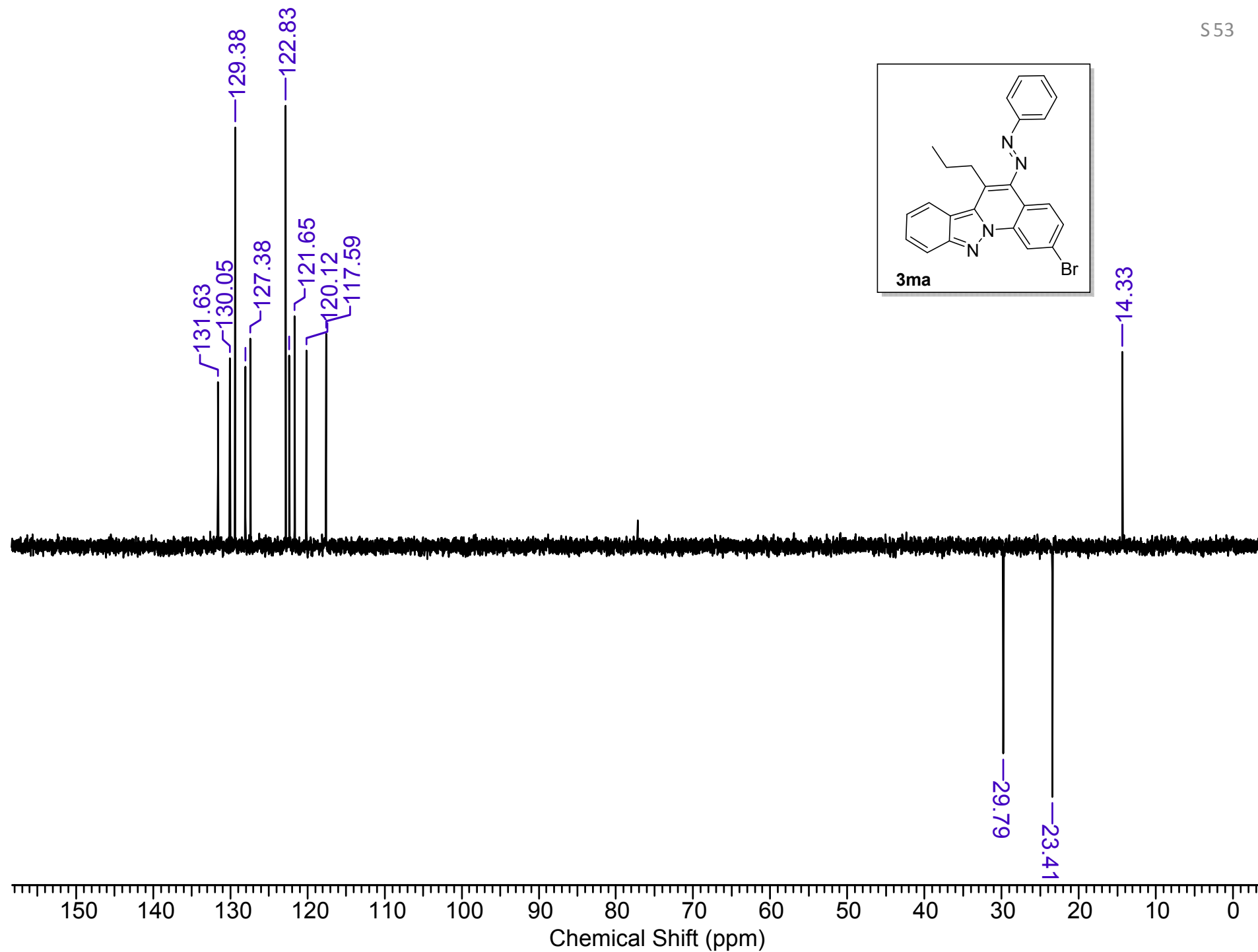
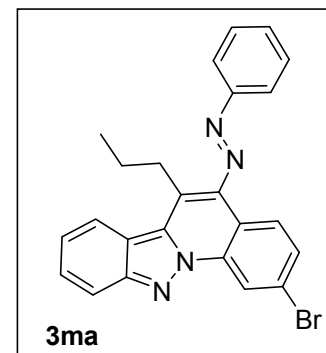


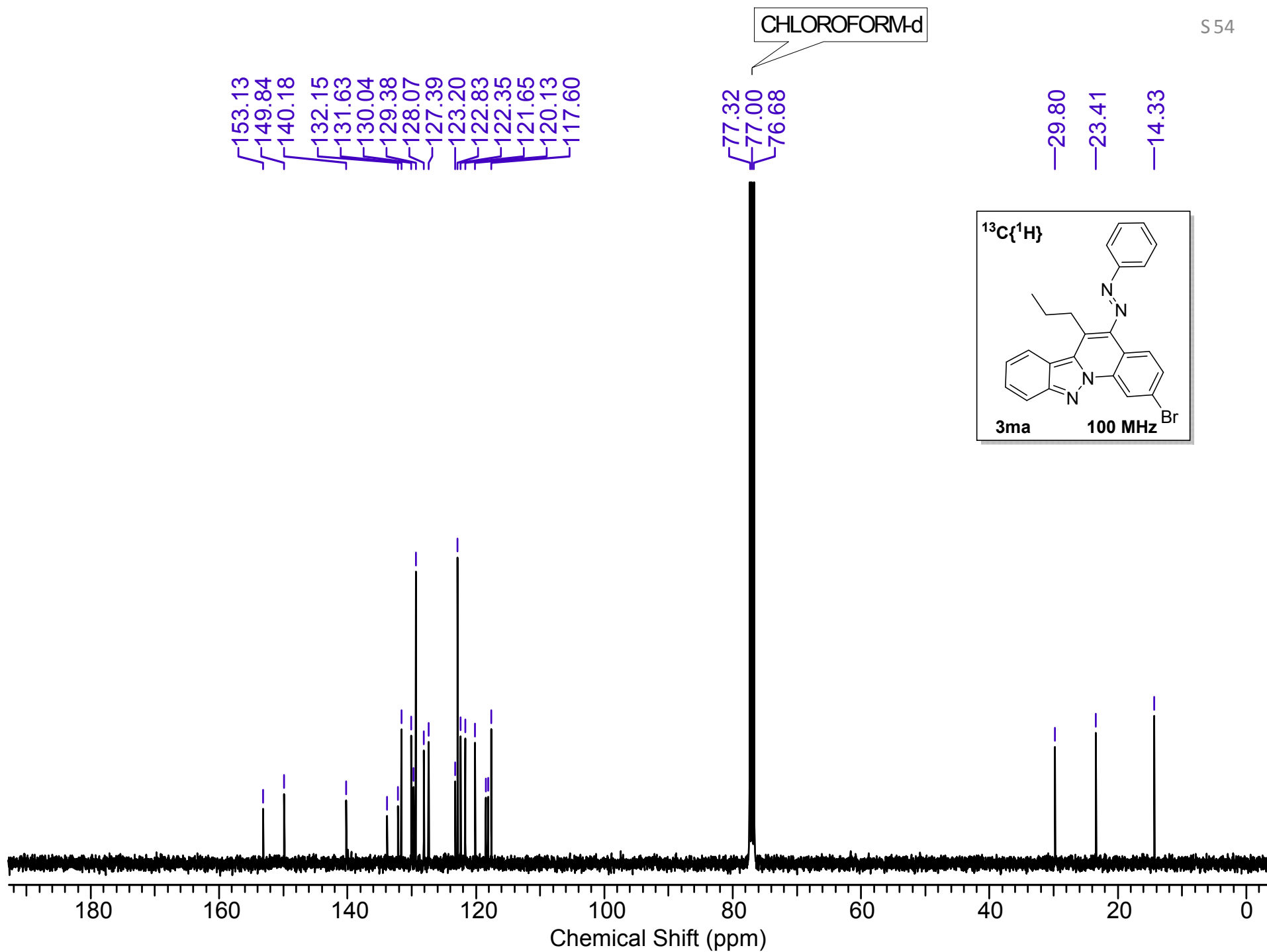


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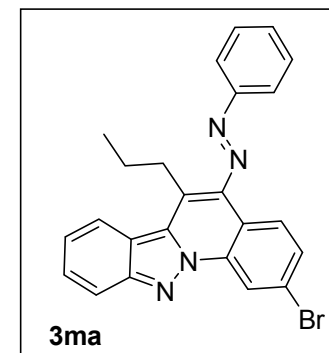
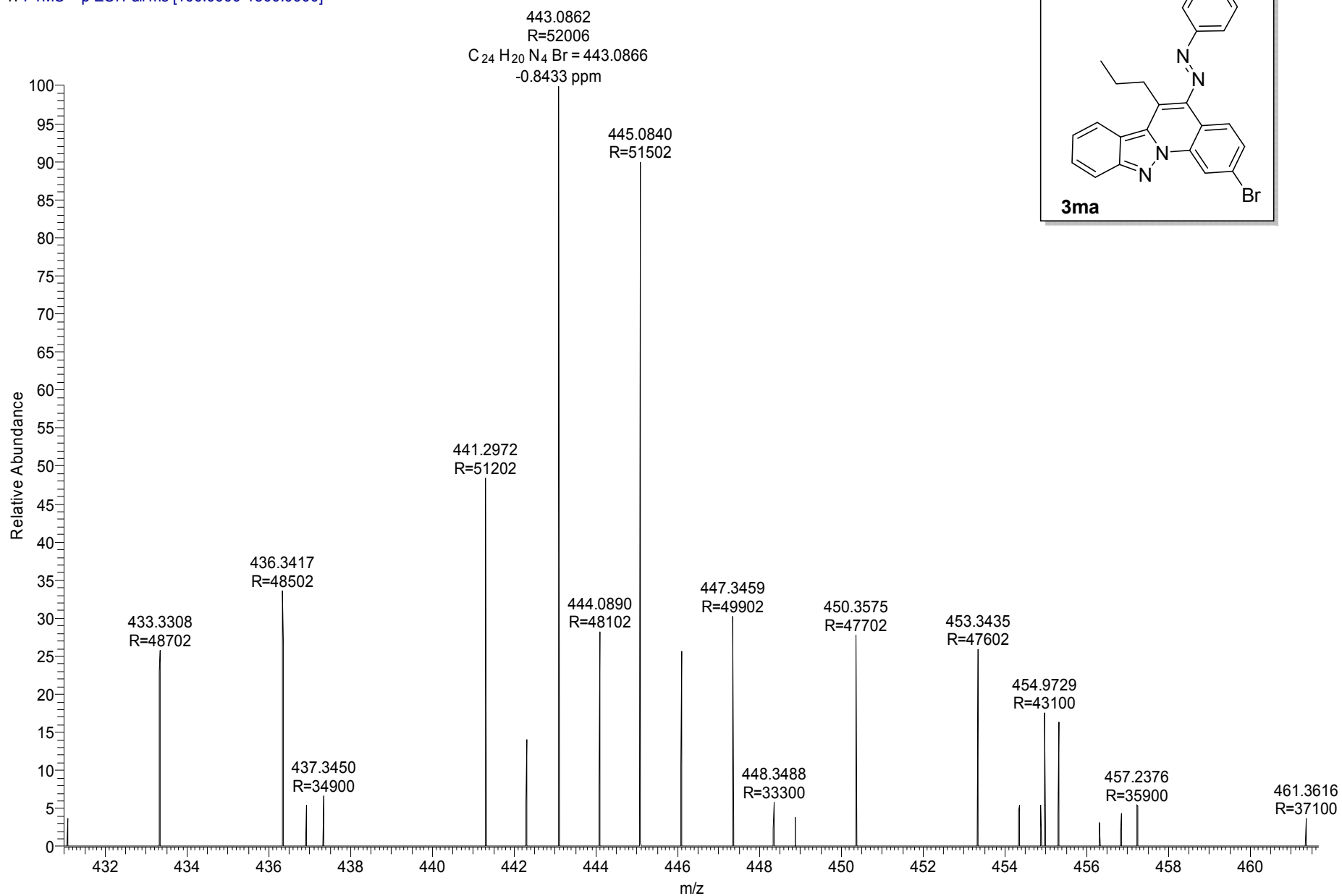


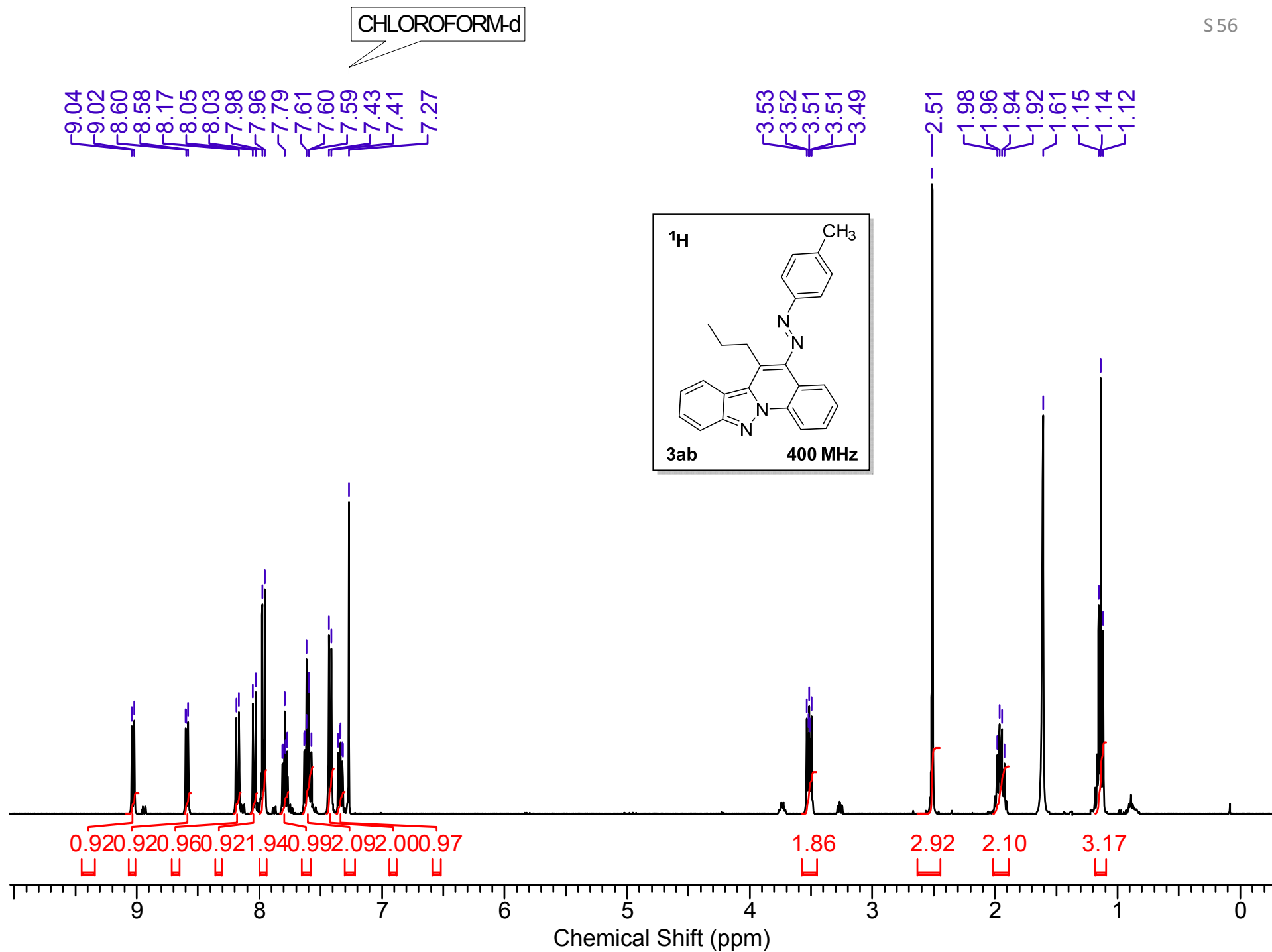


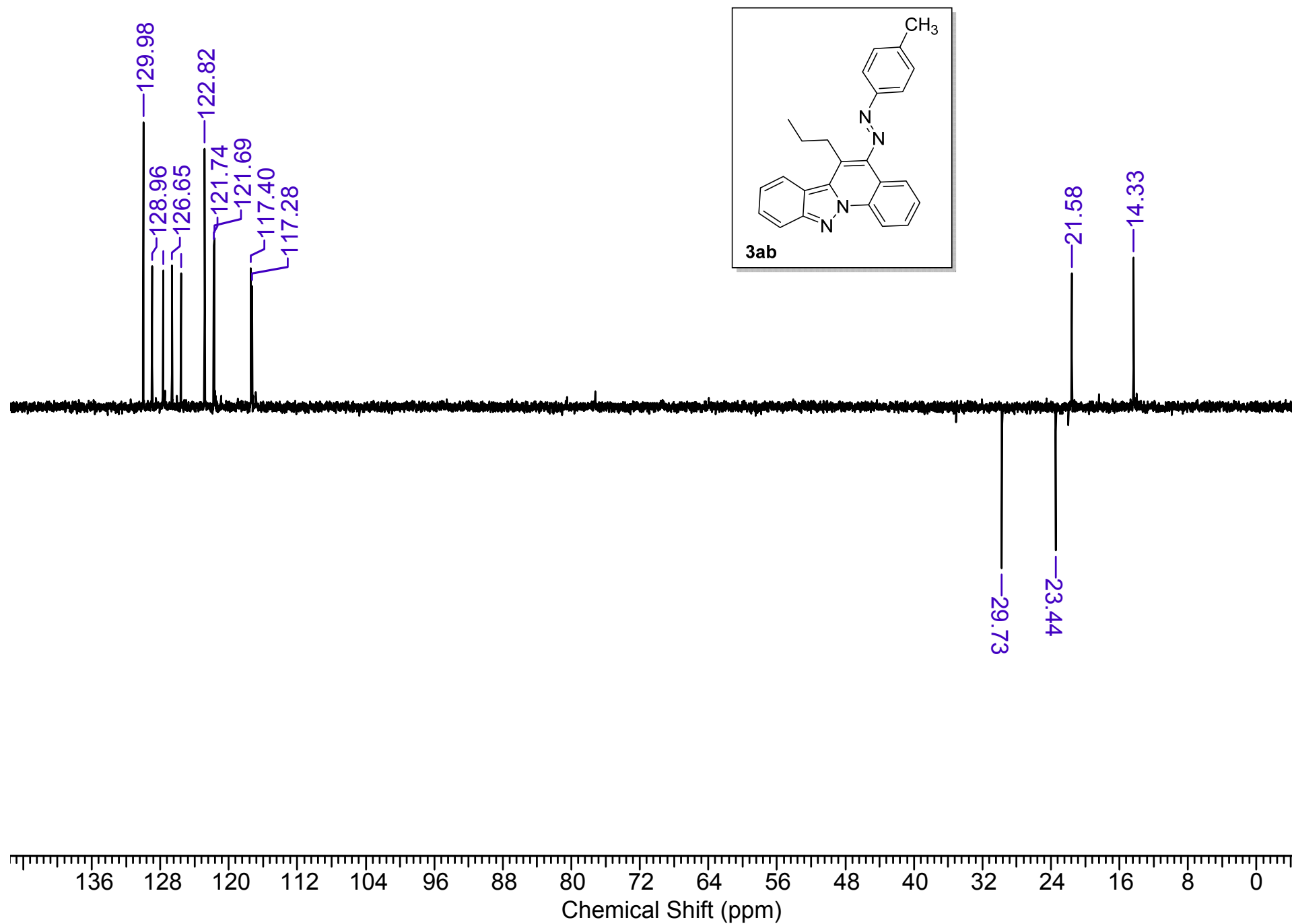


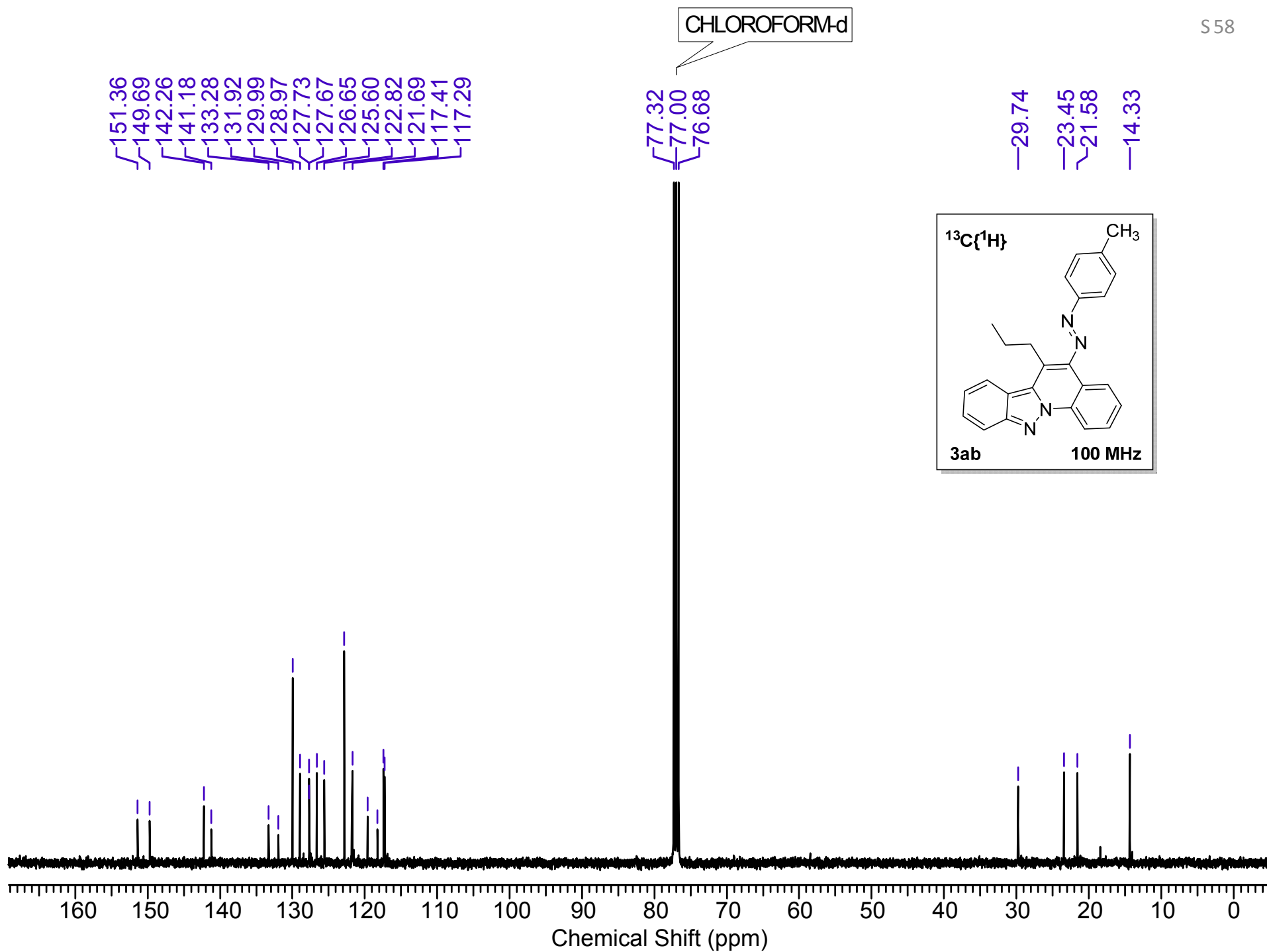


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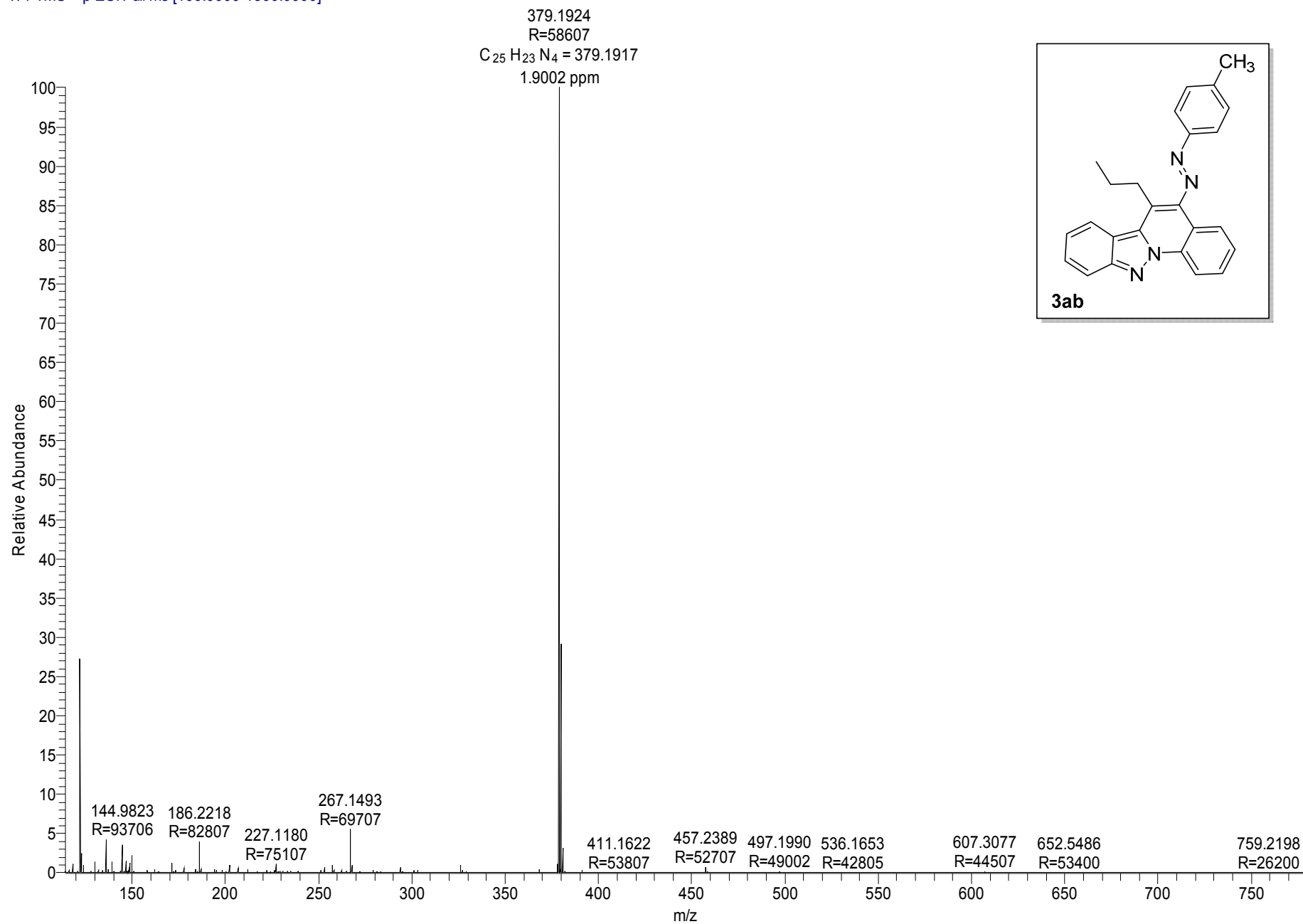


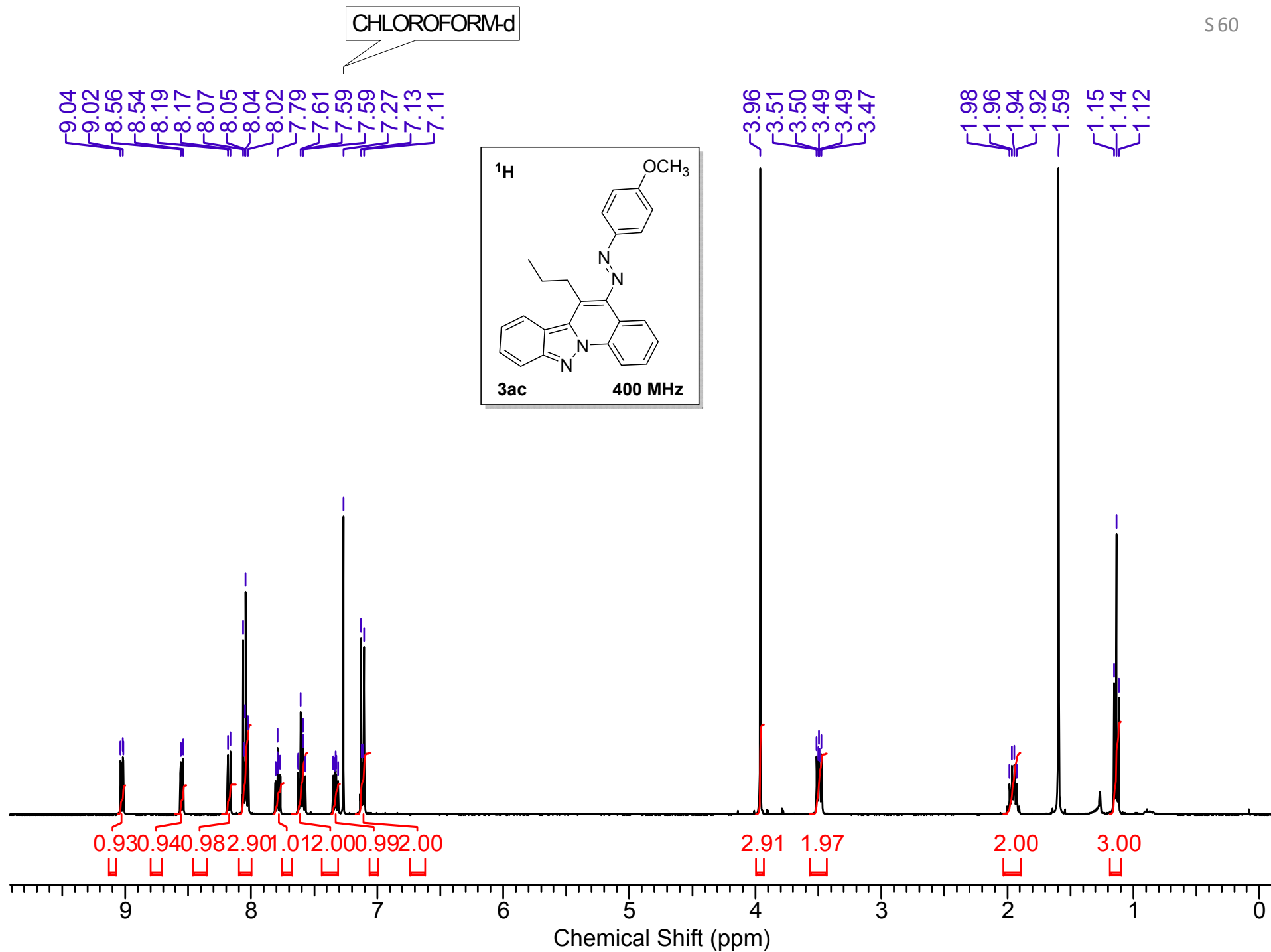


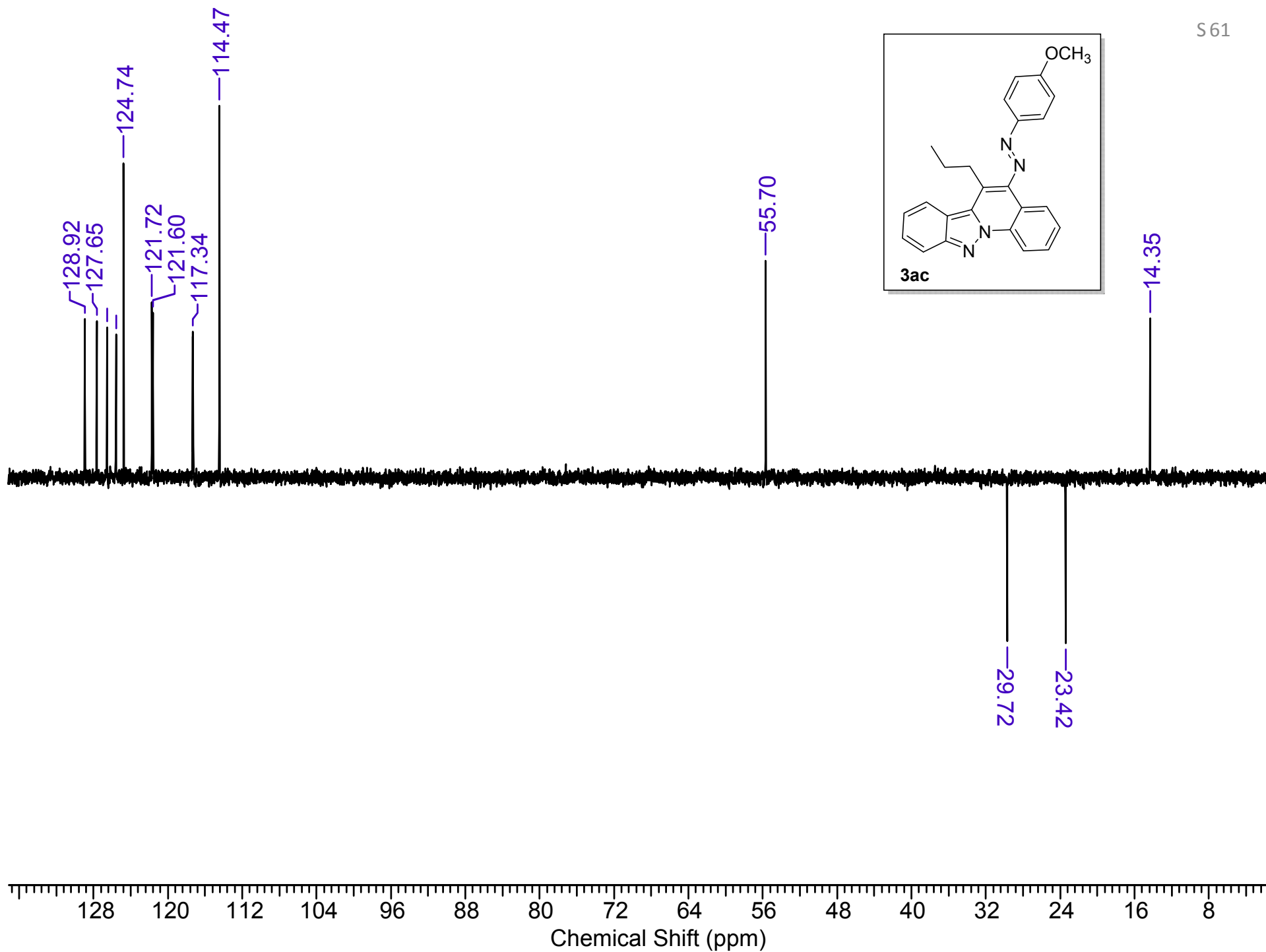
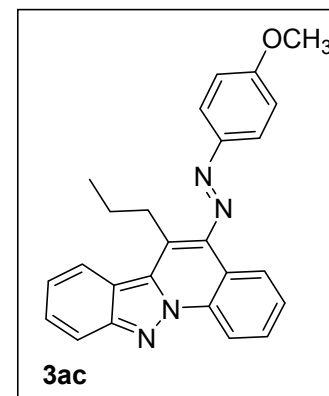


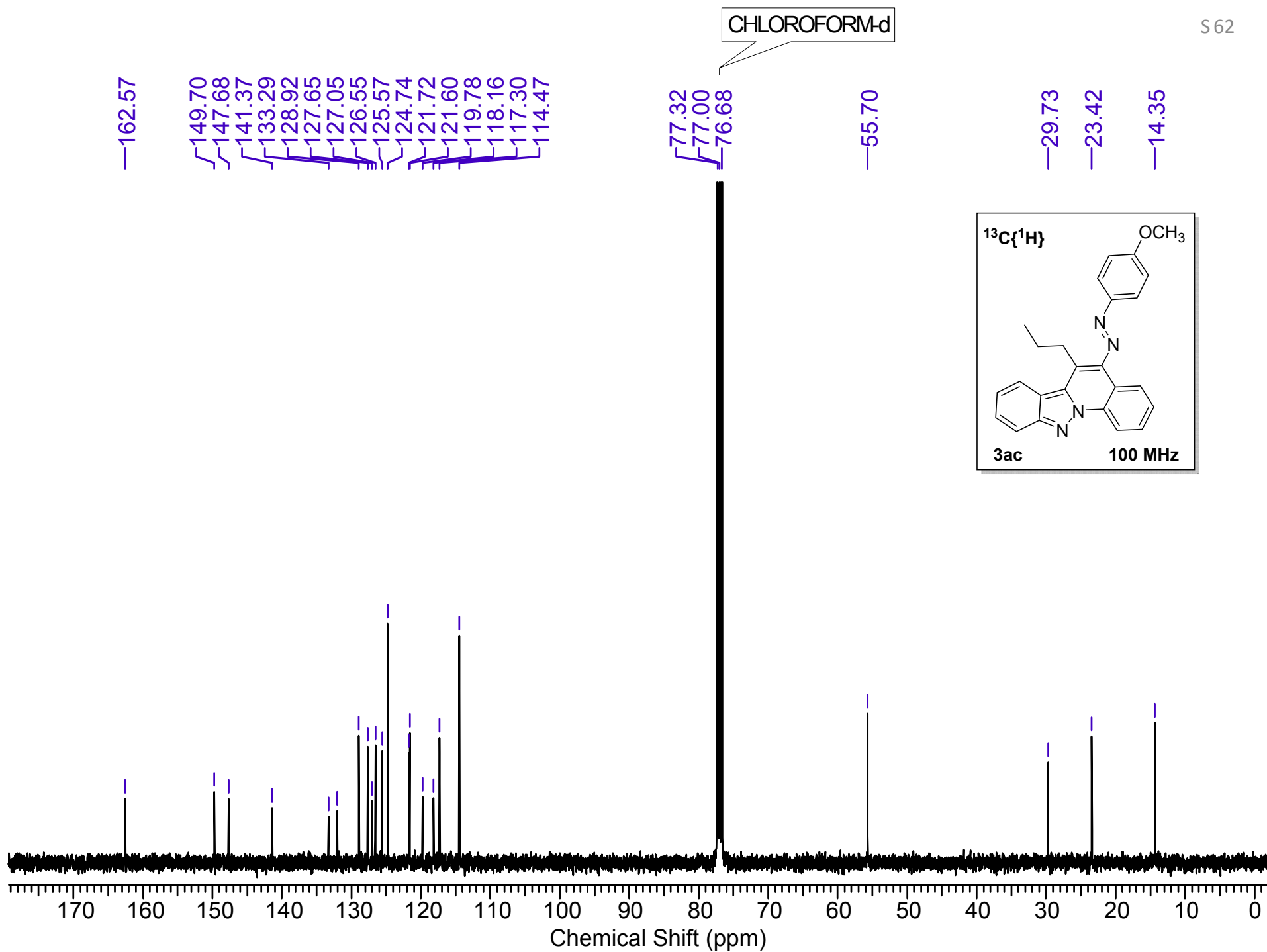


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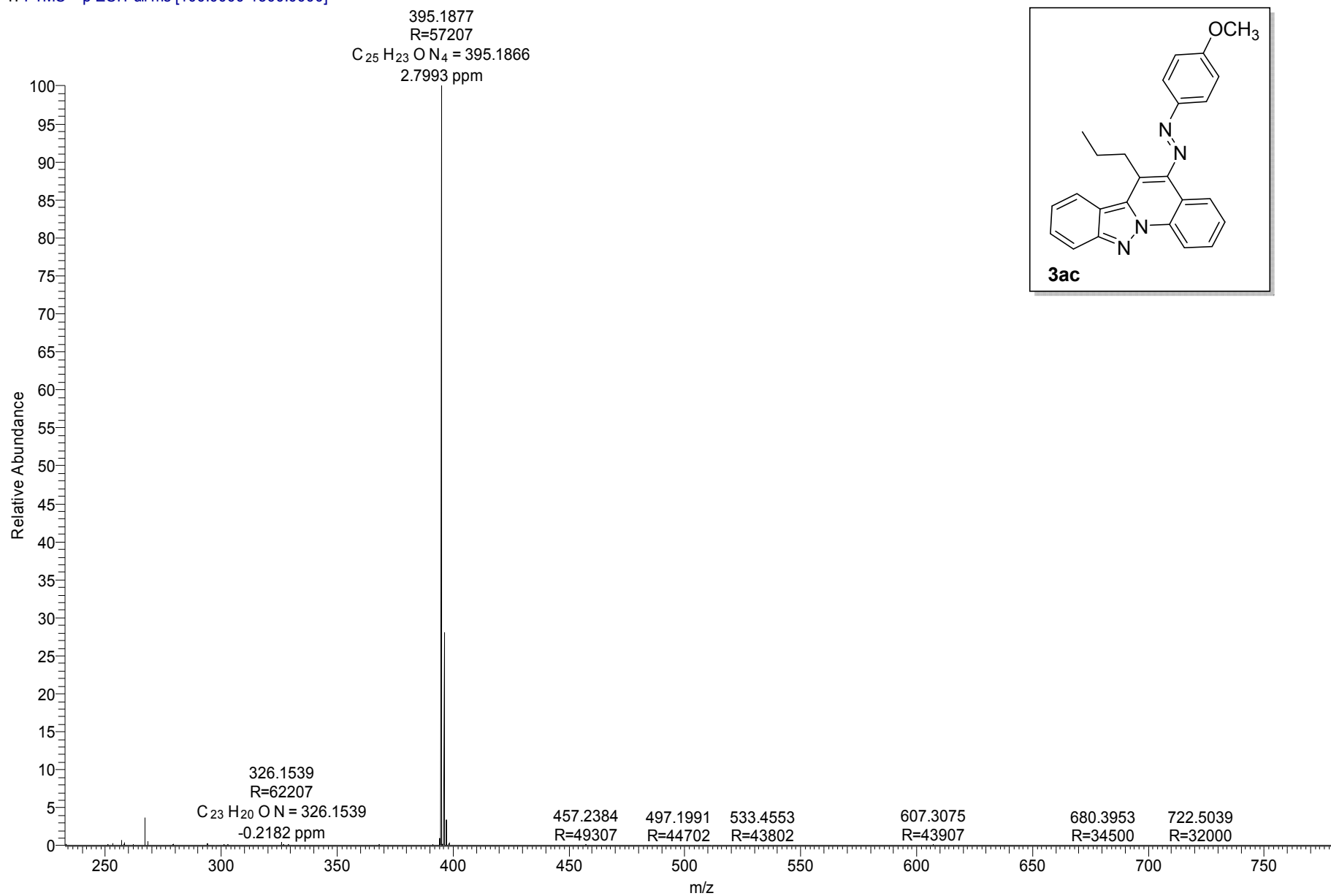


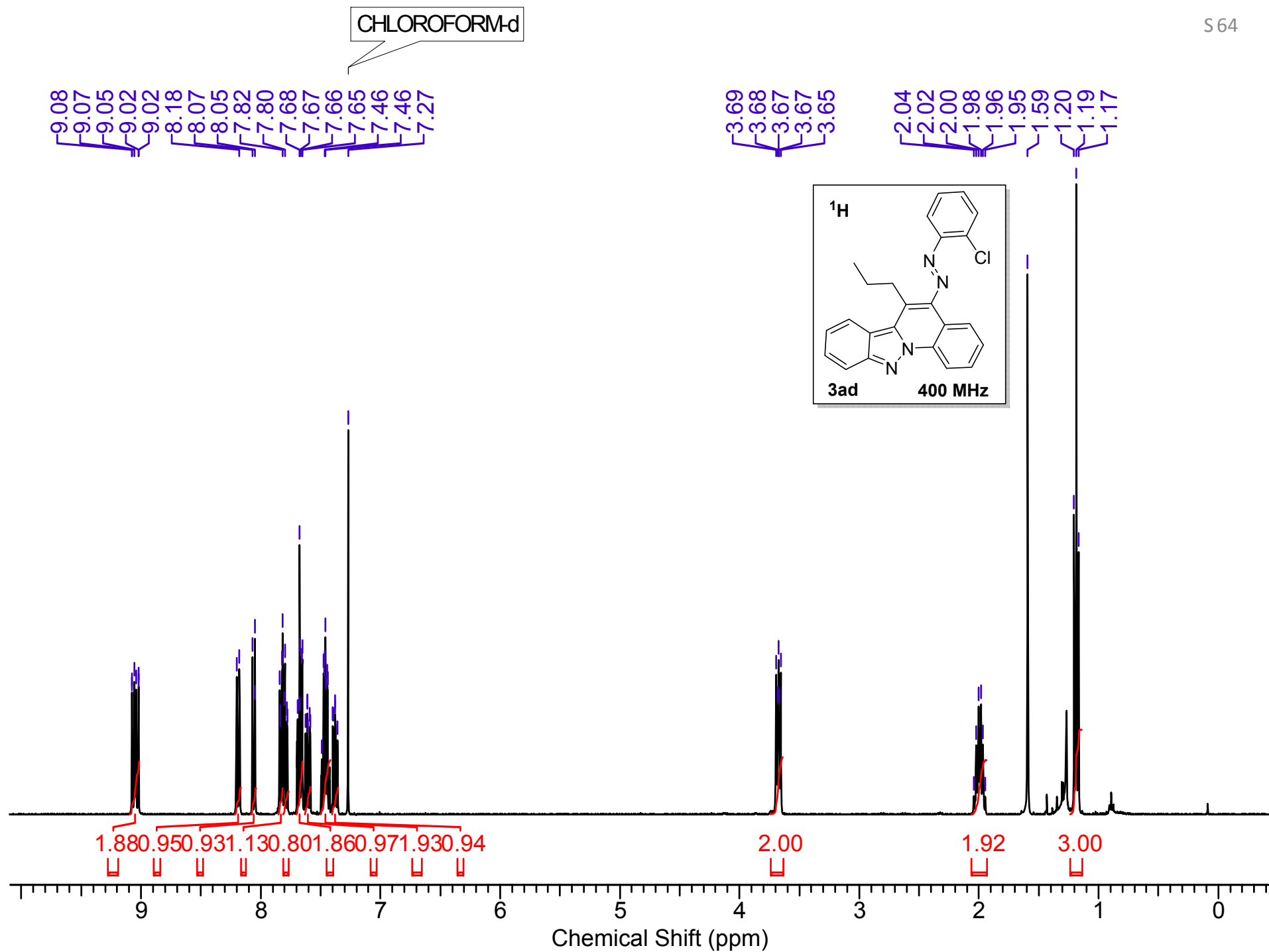


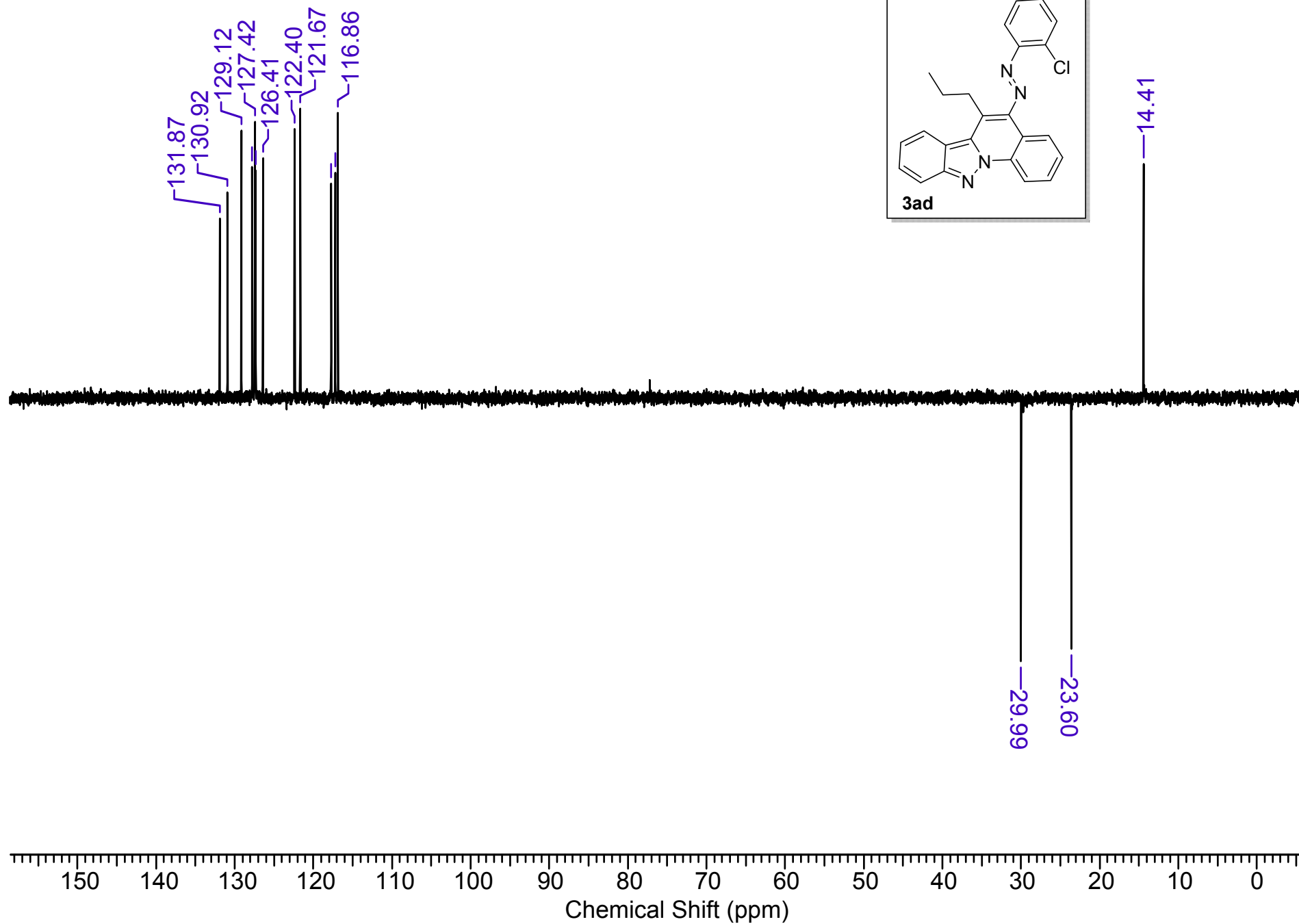
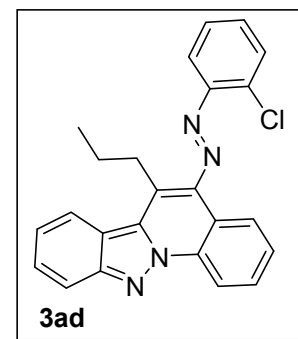


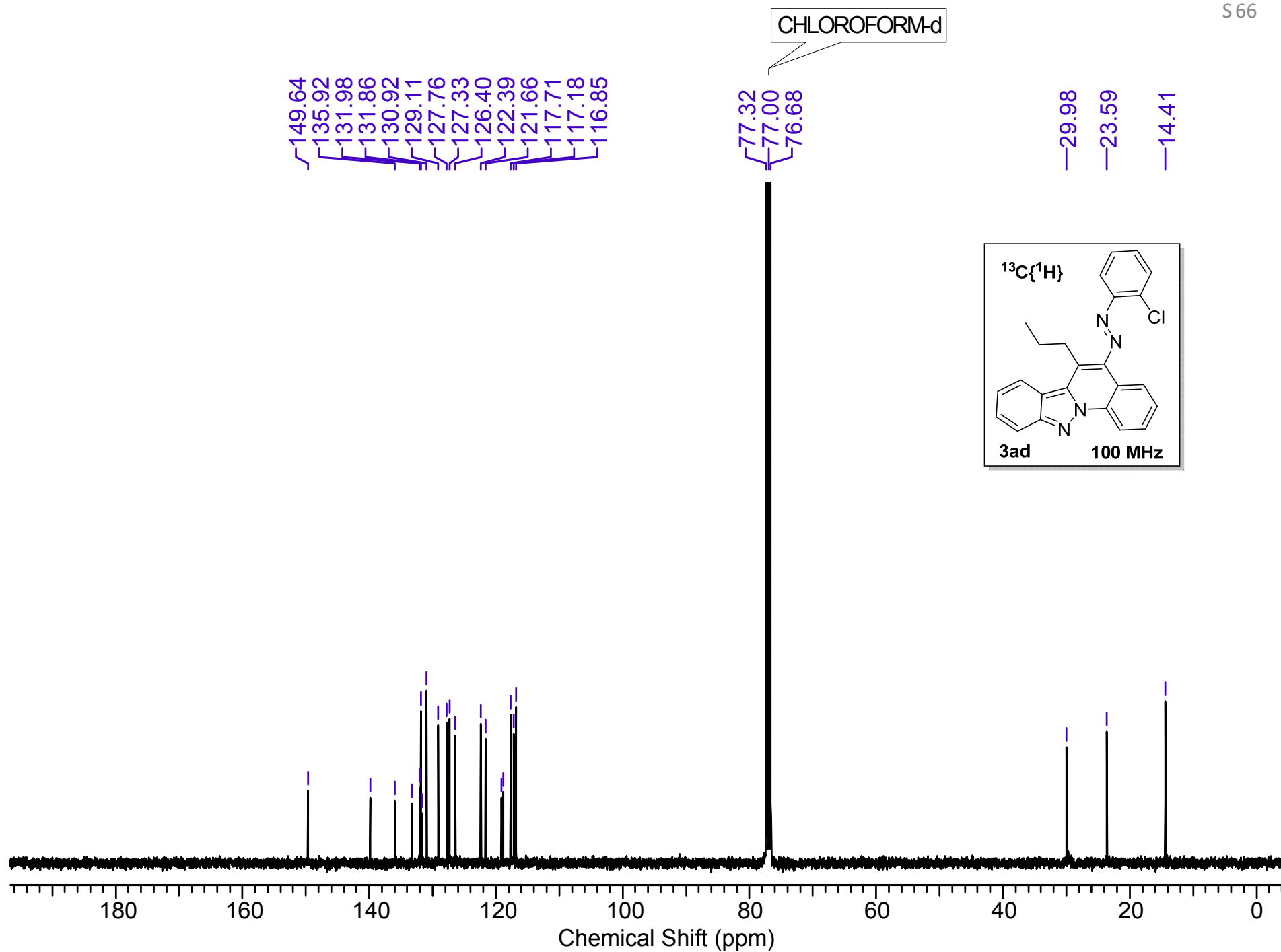


PD-696 #763 RT: 6.43 AV: 1 NL: 2.57E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

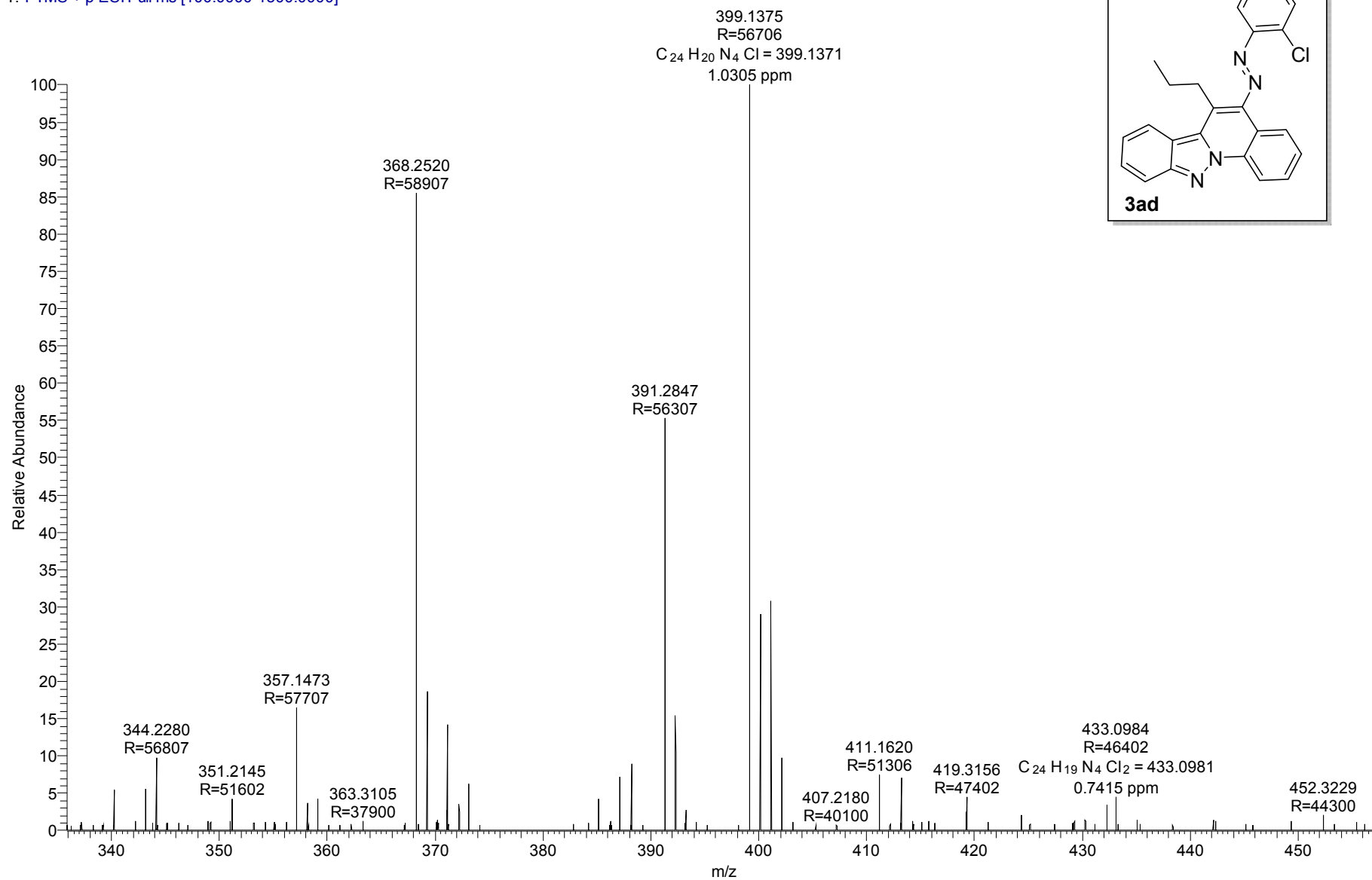
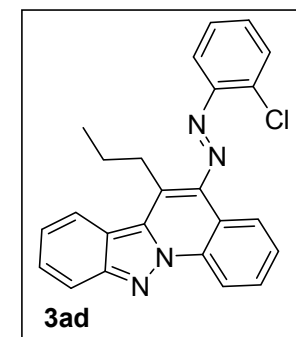


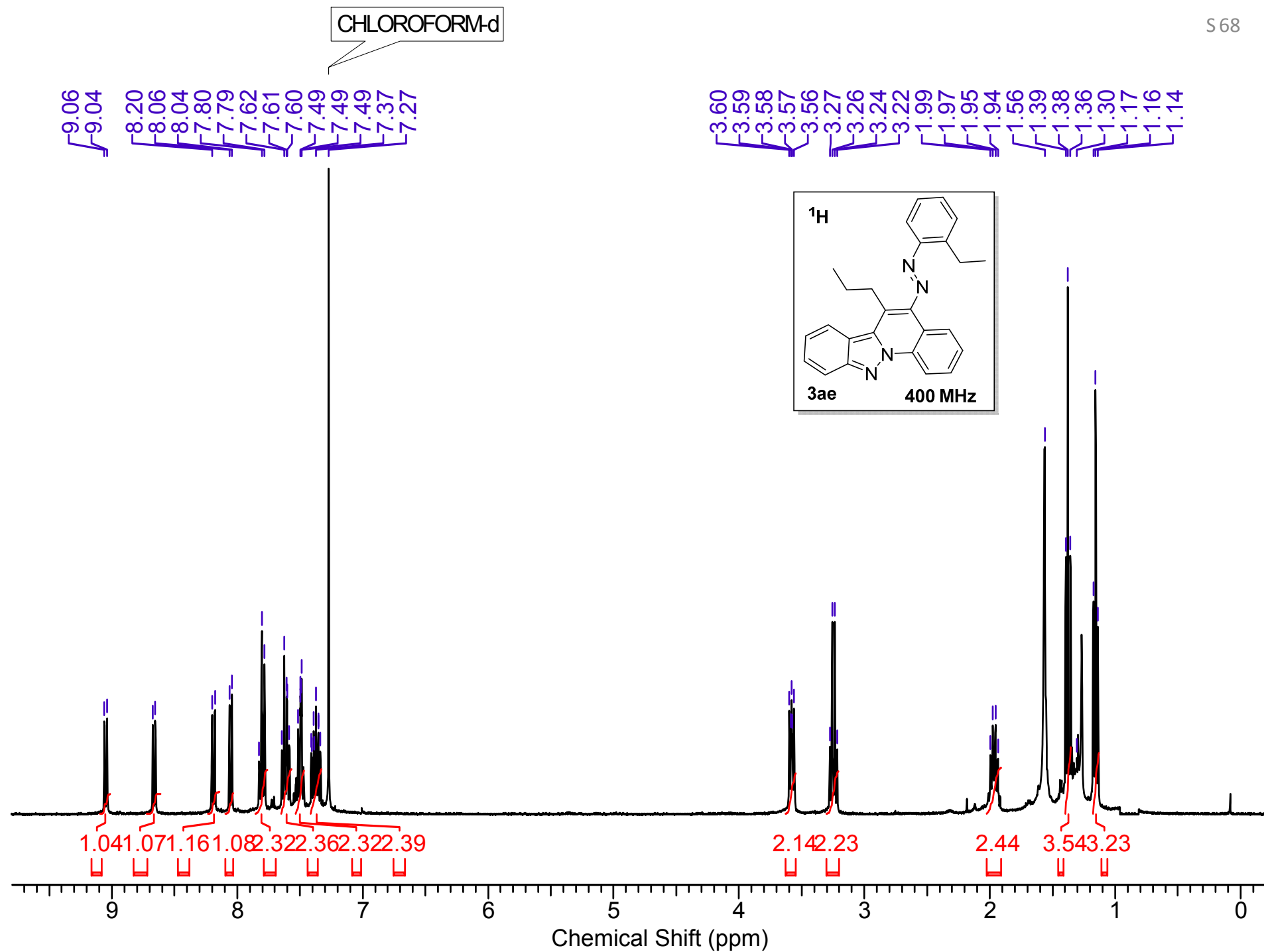


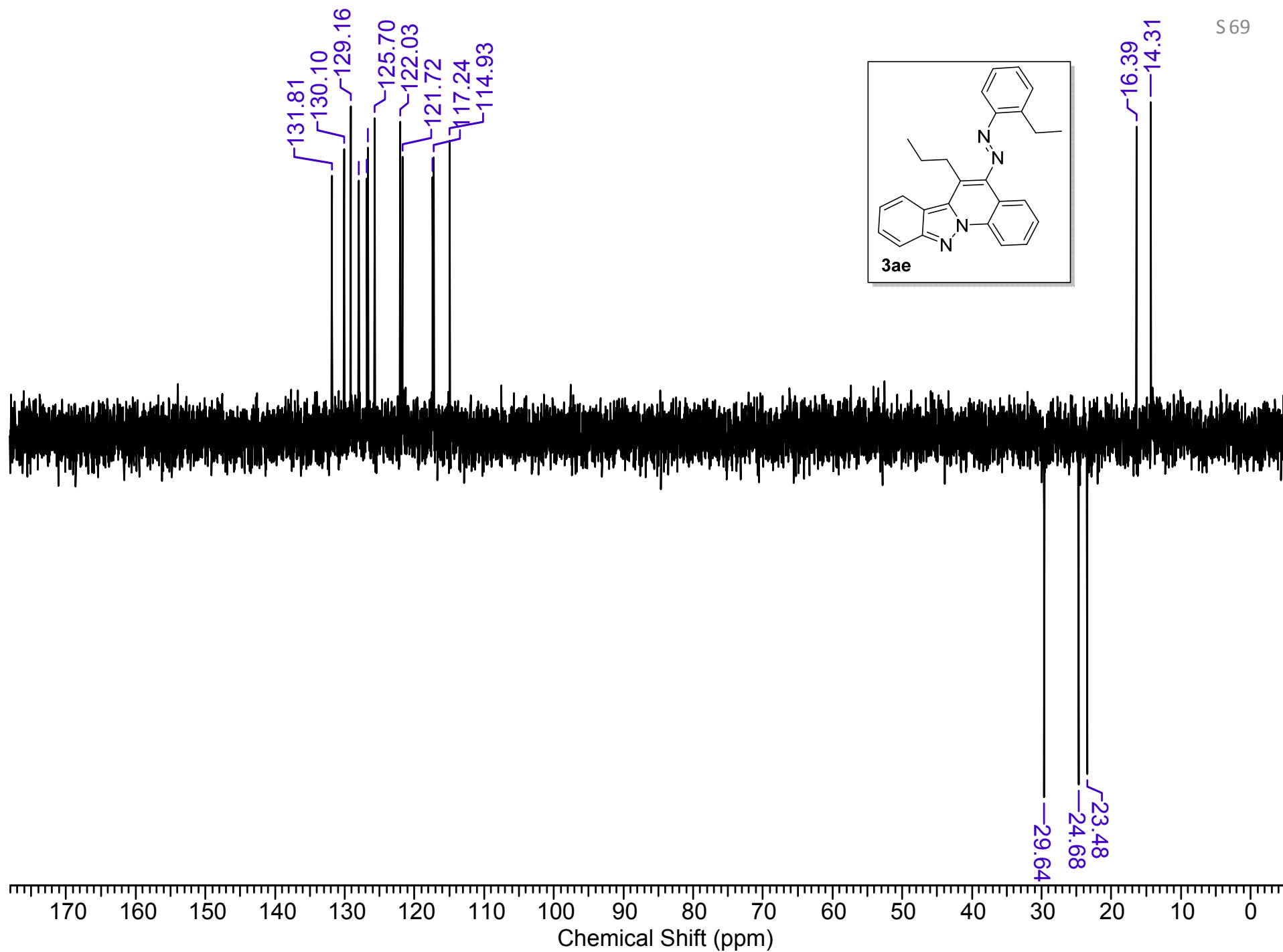
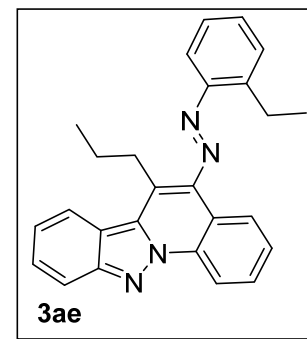


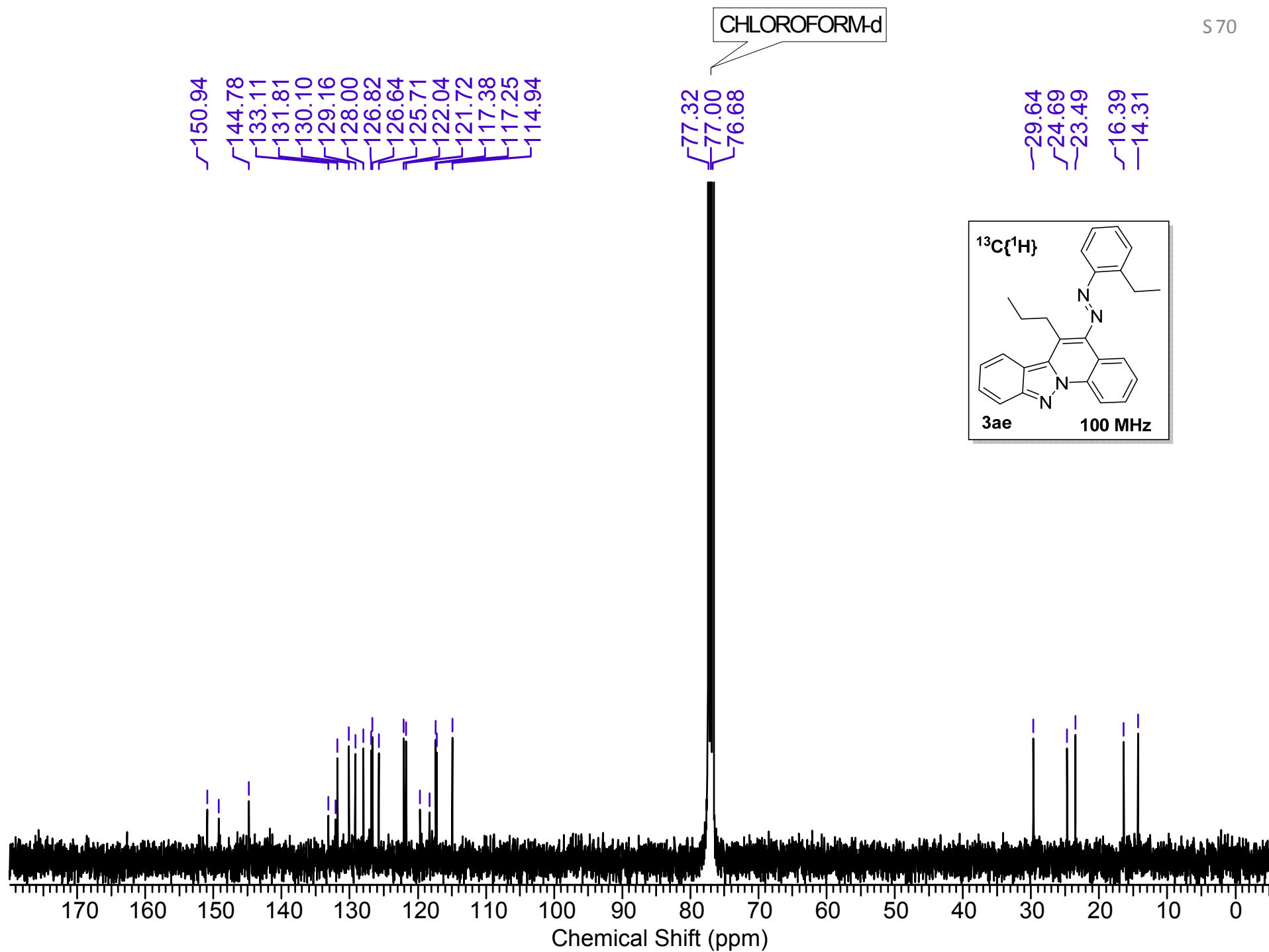


PD-703 #61 RT: 0.52 AV: 1 NL: 1.03E5
T: FTMS + p ESI Full ms [100.0000-1500.0000]

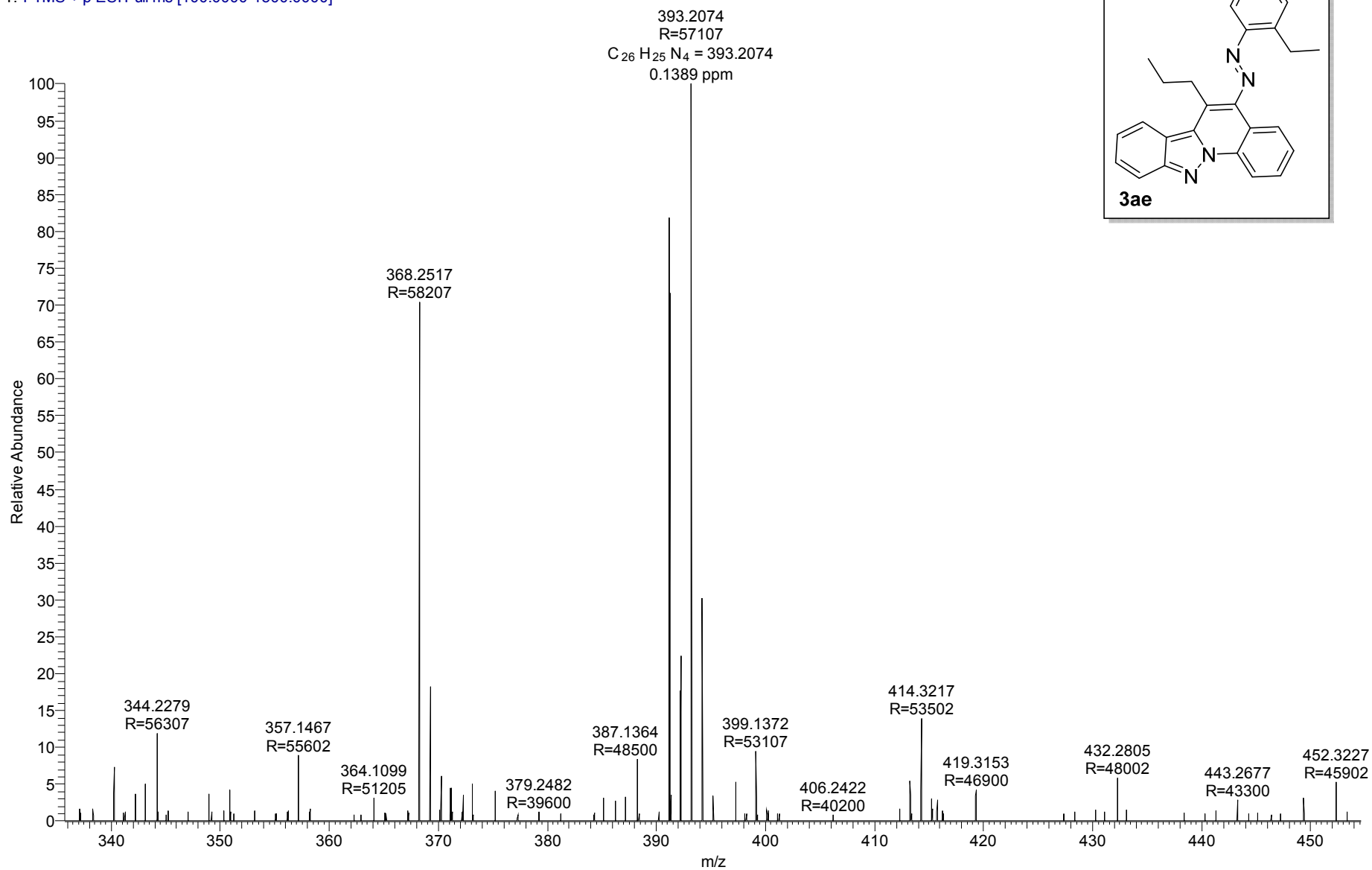


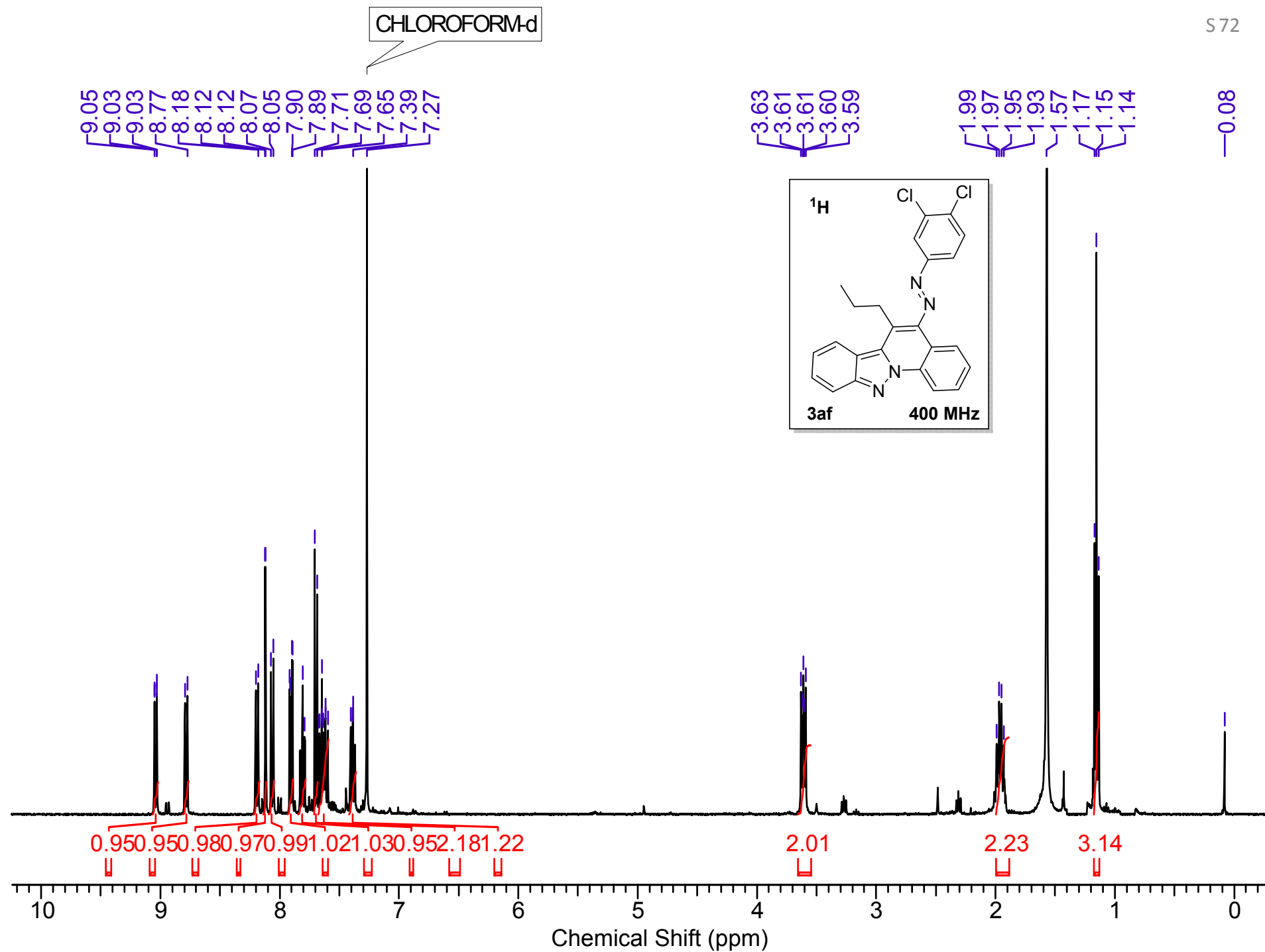


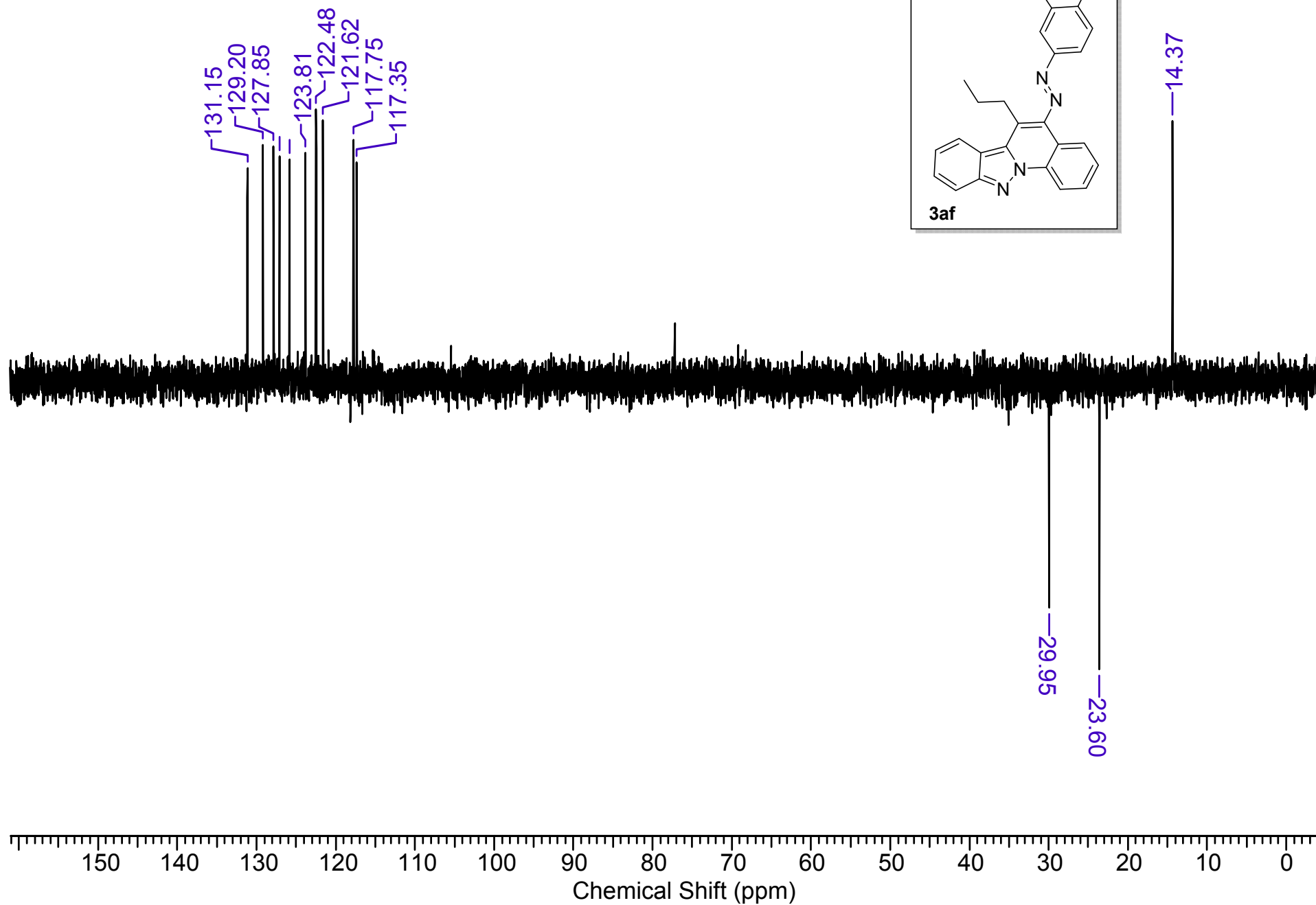
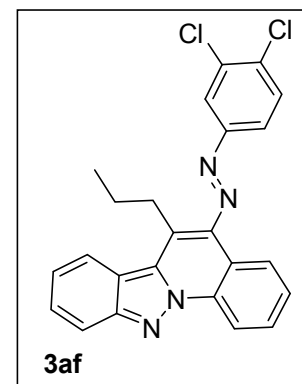


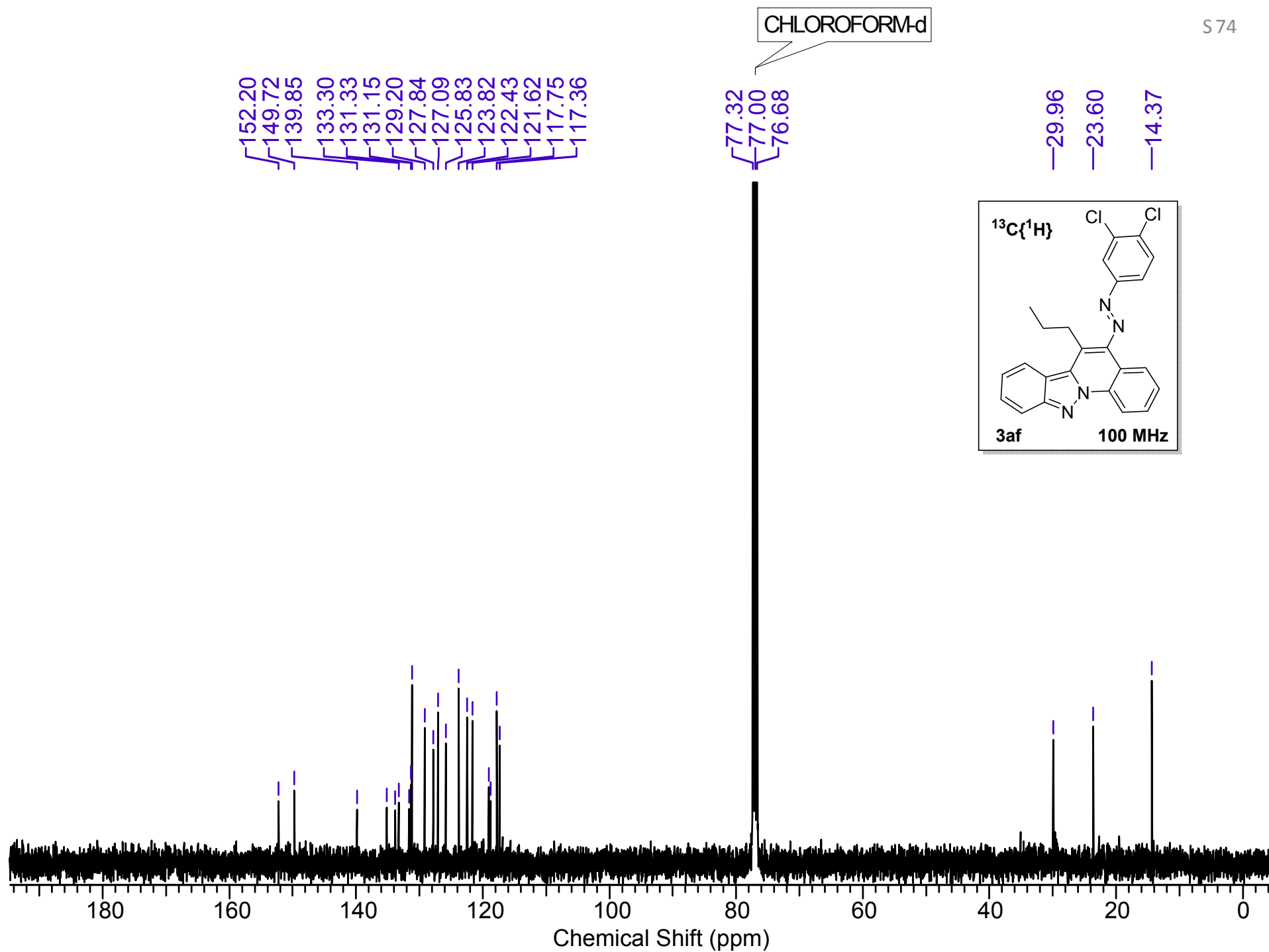


PD-708_201231152143 #566 RT: 4.80 AV: 1 NL: 8.34E4
T: FTMS + p ESI Full ms [100.0000-1500.0000]

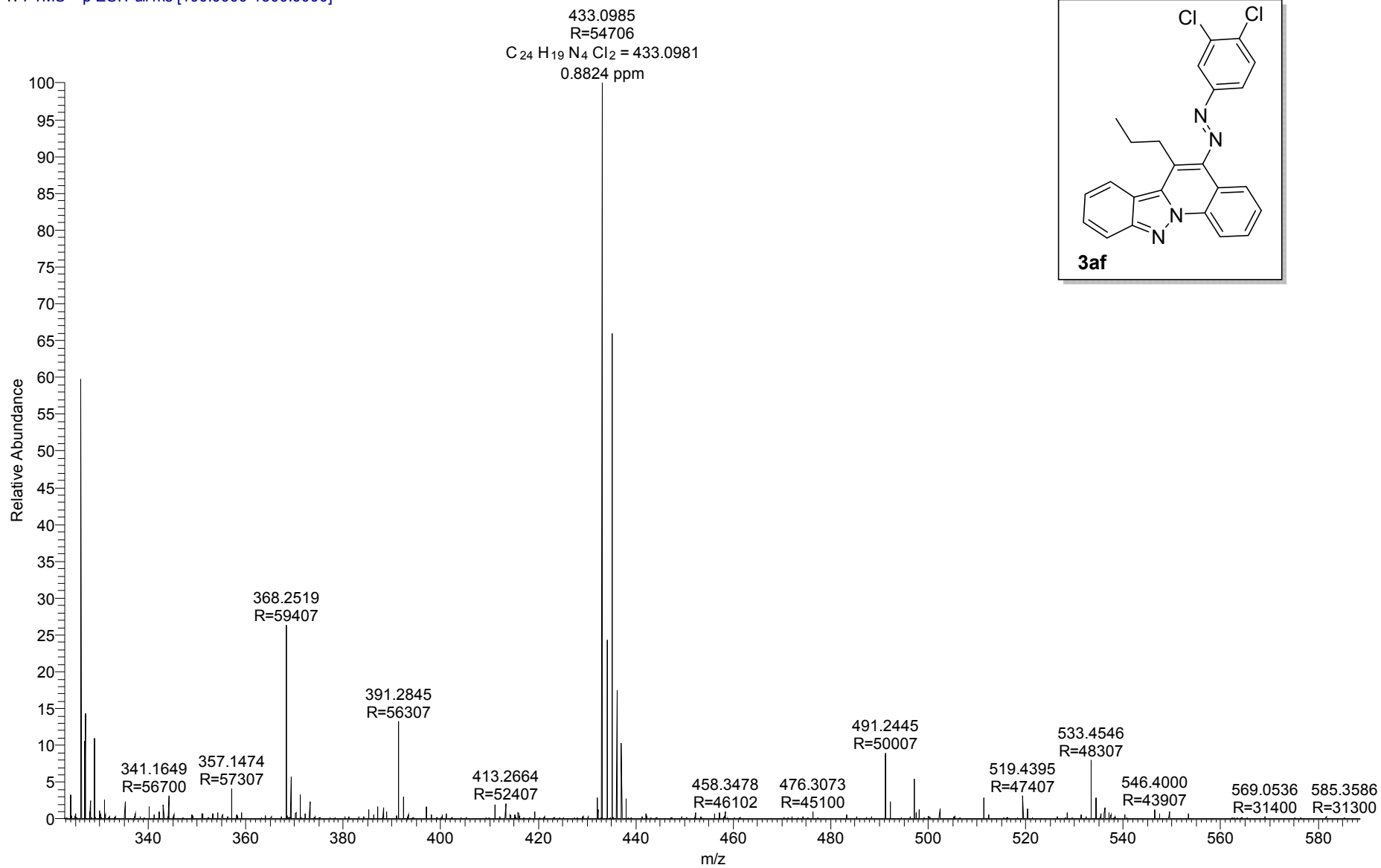




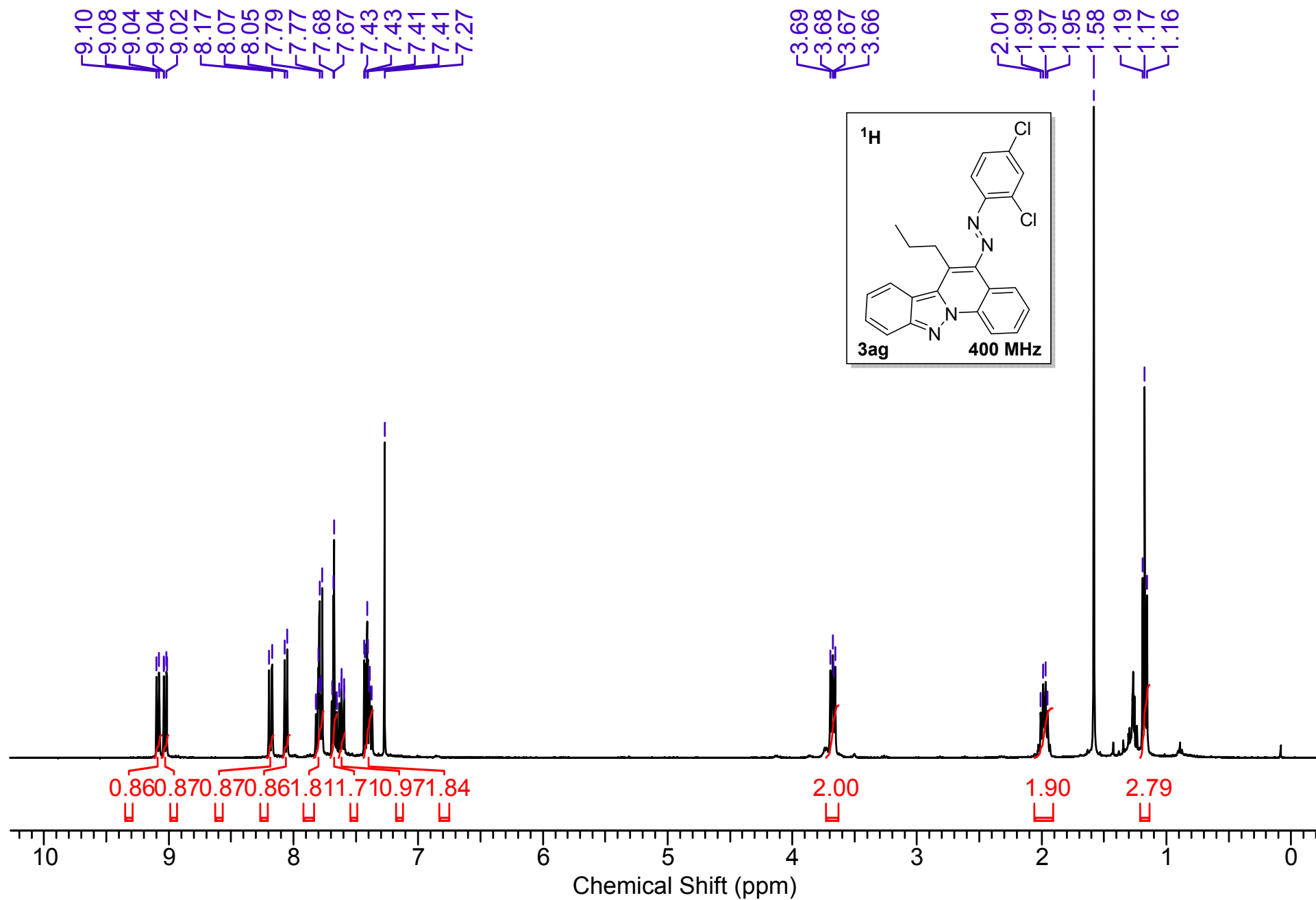


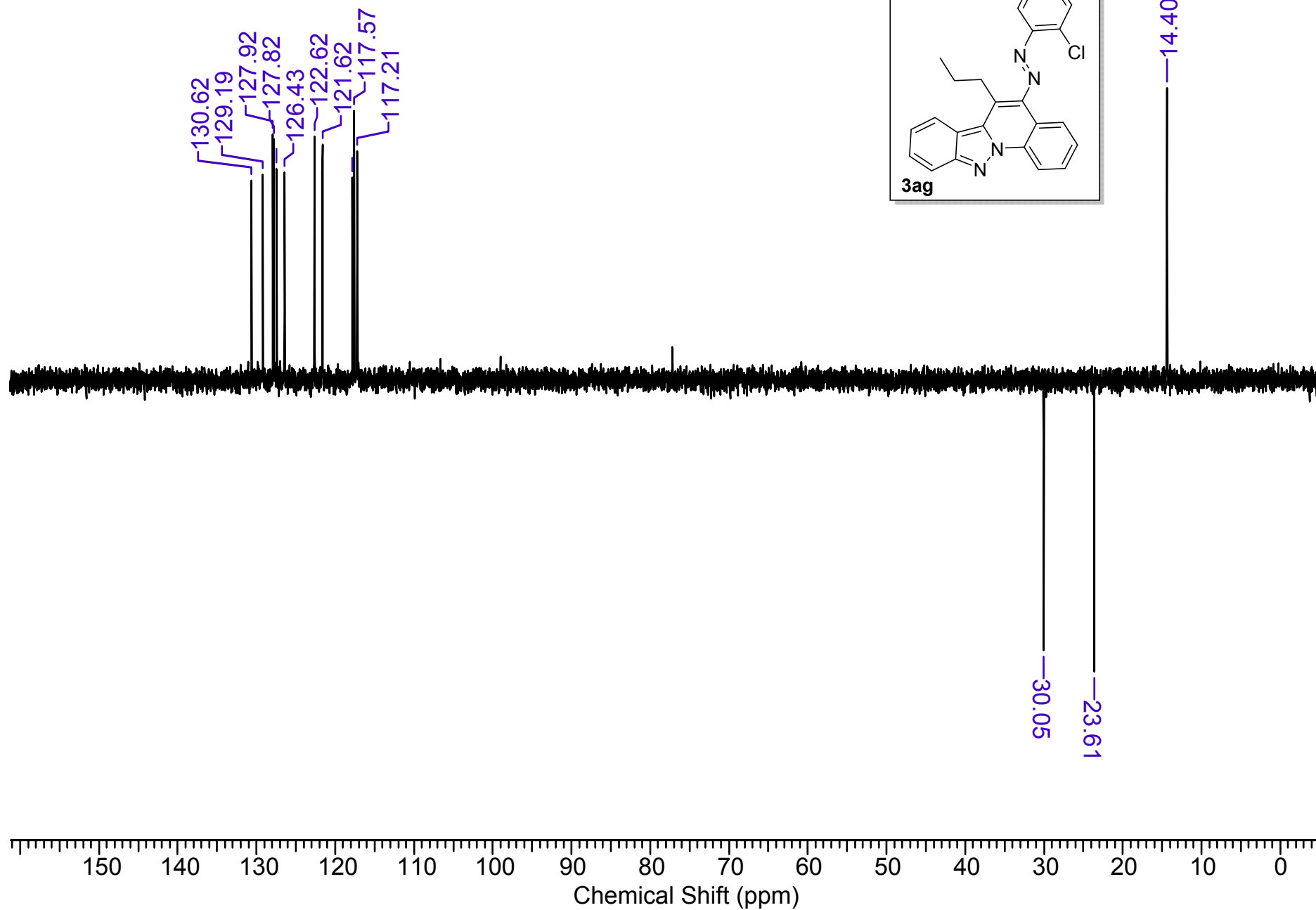
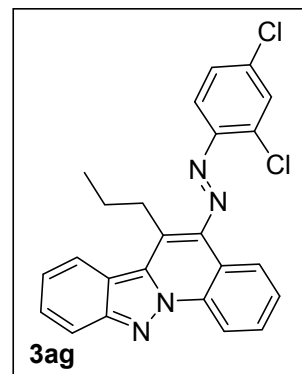


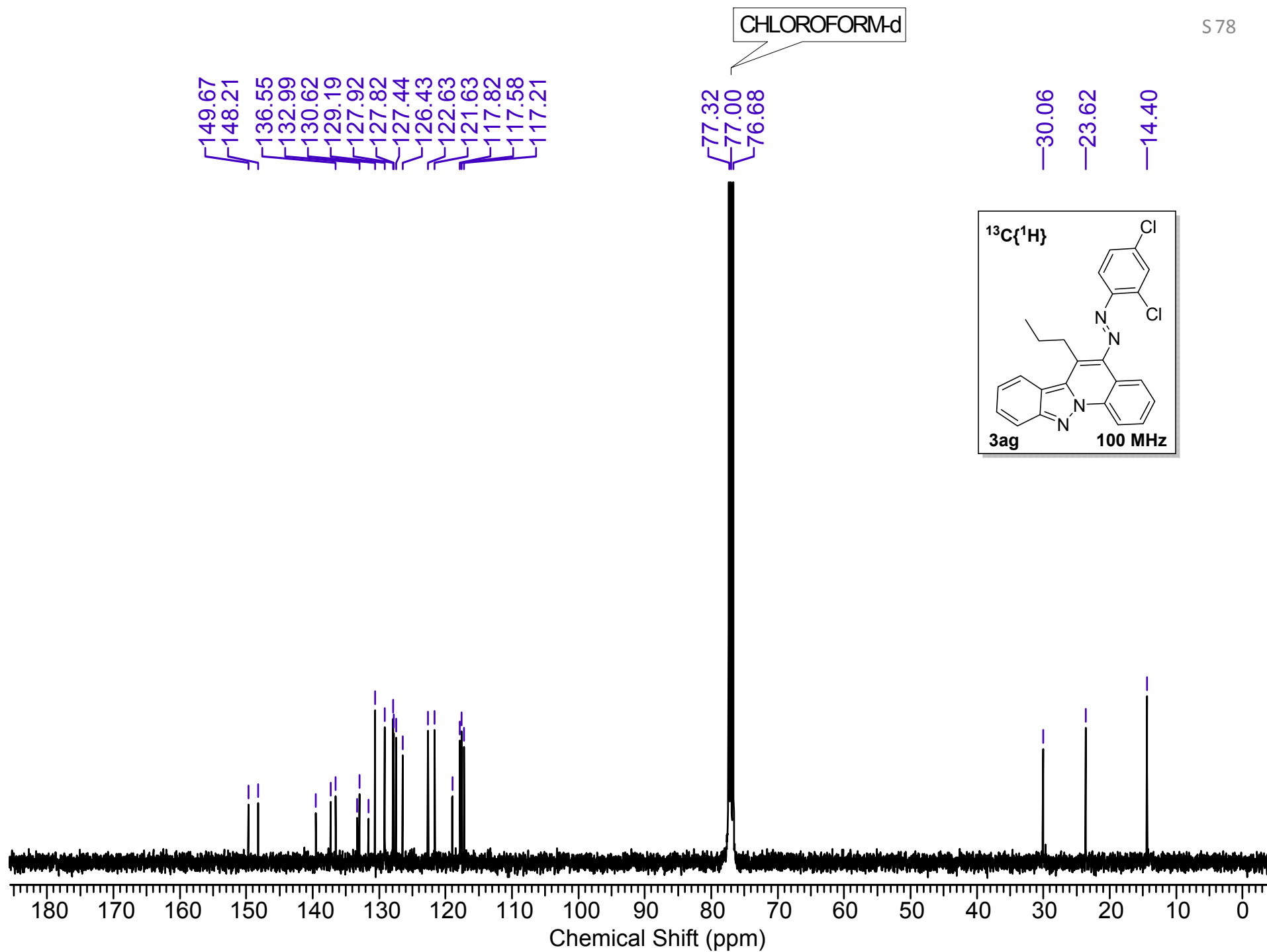
PD-711 #592 RT: 4.89 AV: 1 NL: 4.89E5
T: FTMS + p ESI Full ms [100.0000-1500.0000]



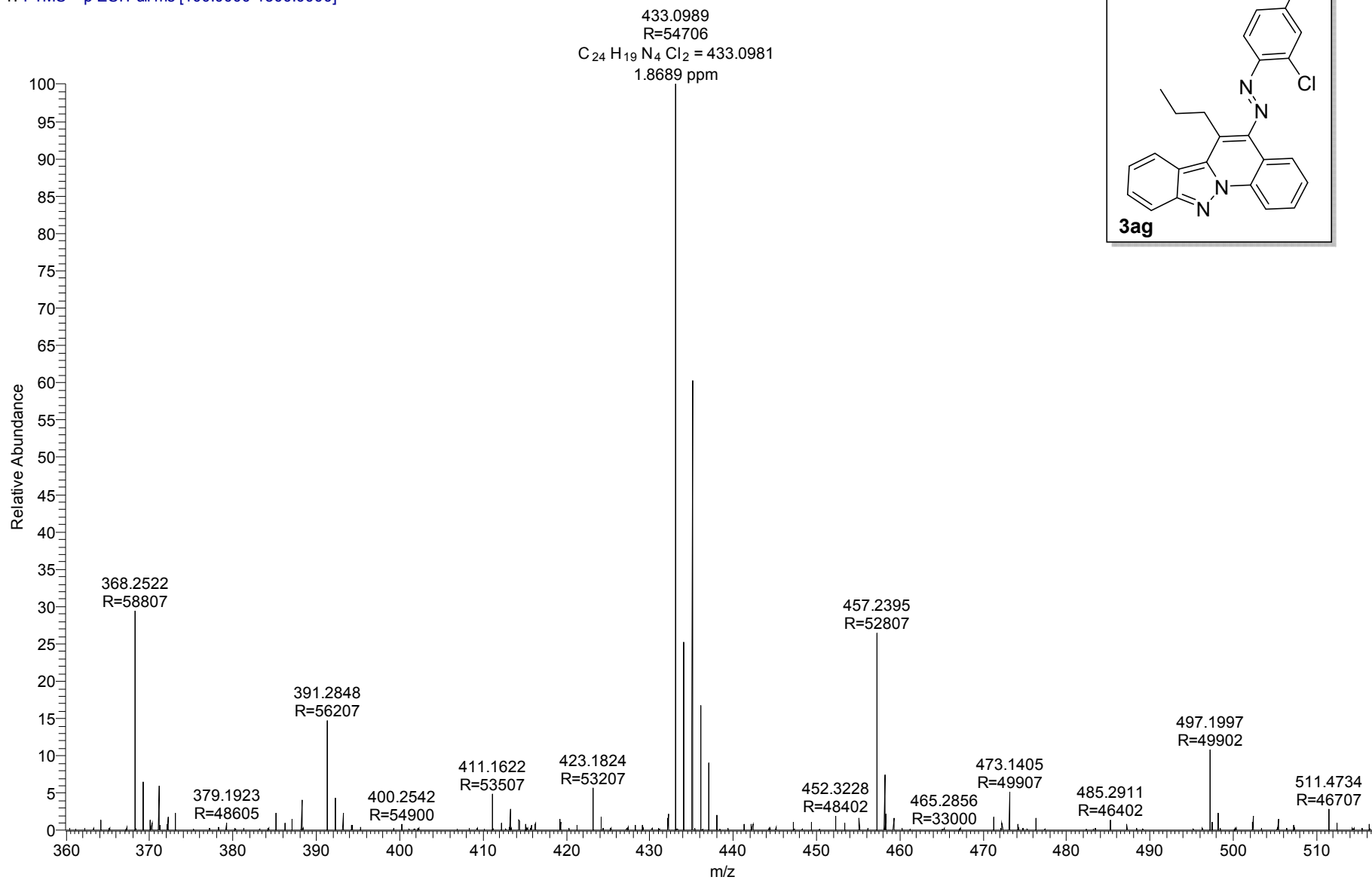
CHLOROFORM-d

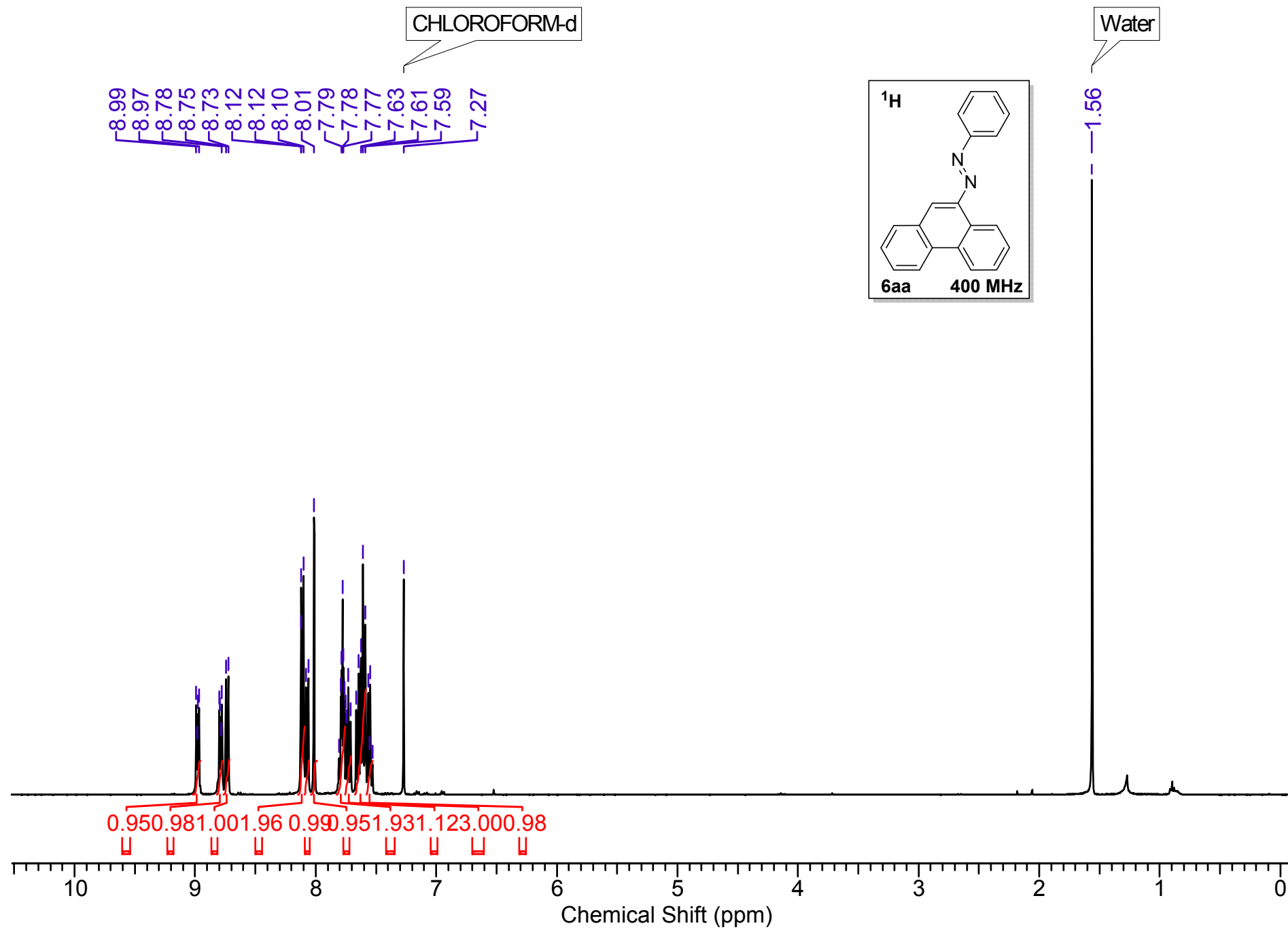


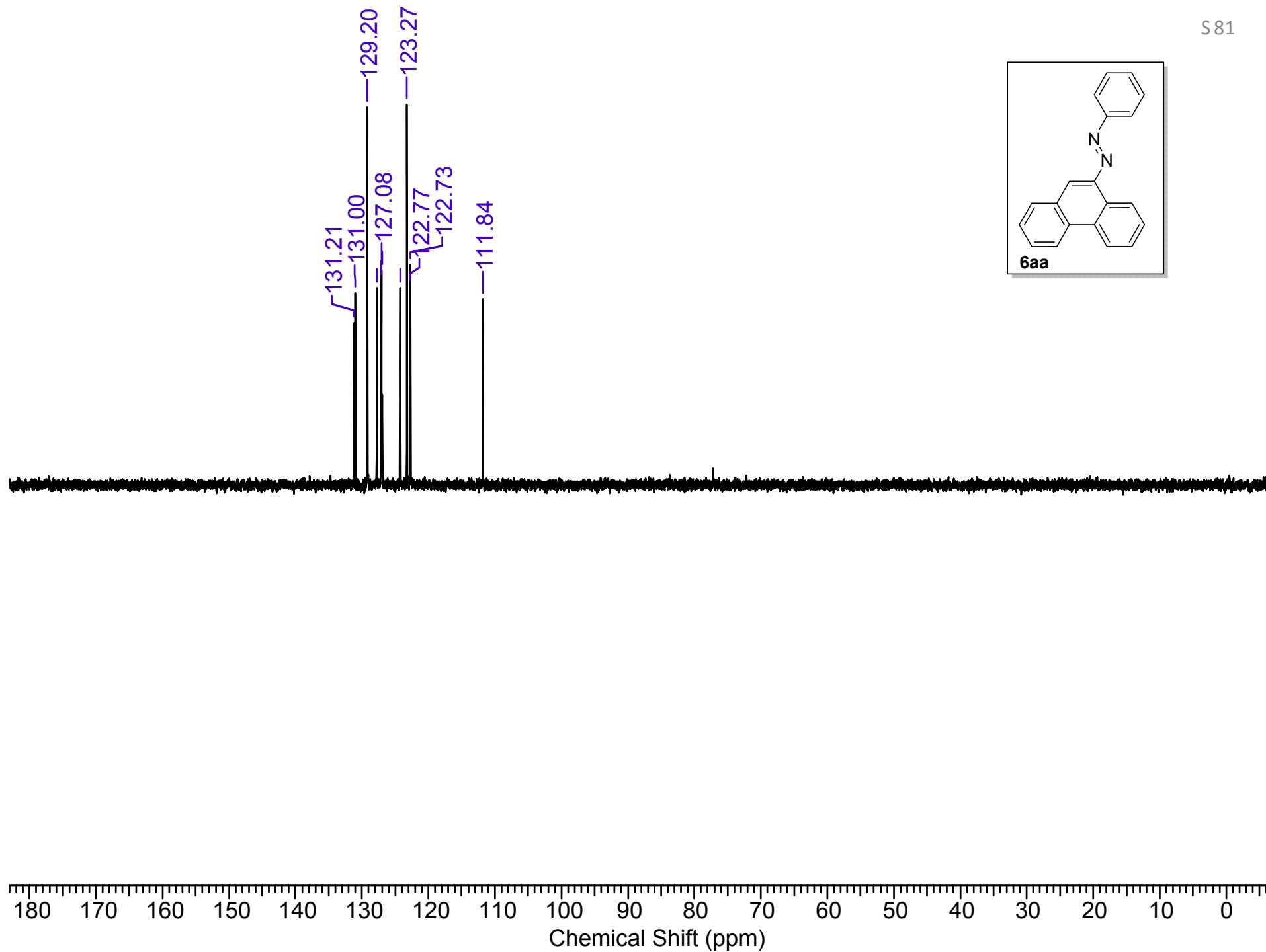


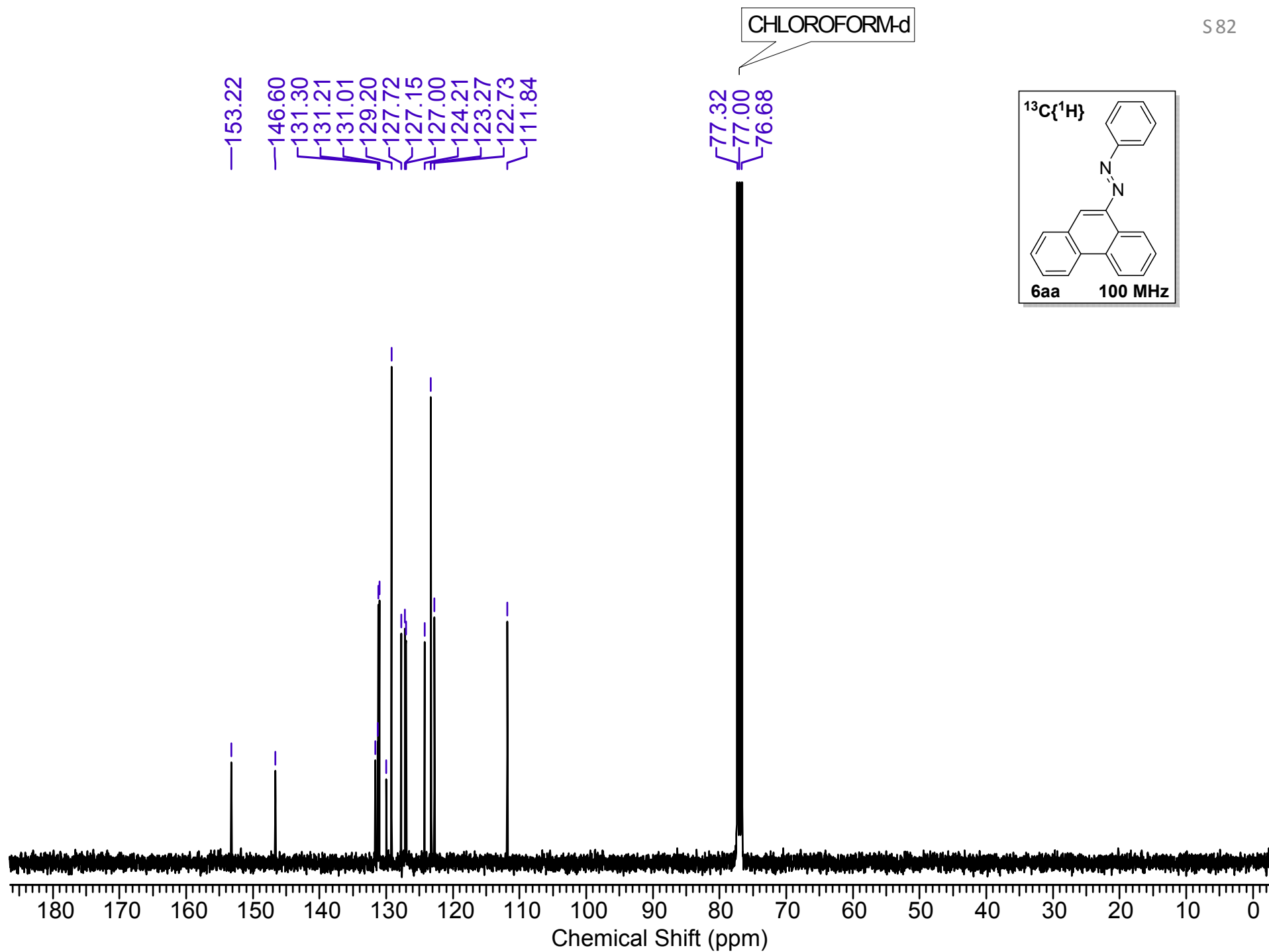


PD-720 #577 RT: 4.86 AV: 1 NL: 4.21E5
T: FTMS + p ESI Full ms [100.0000-1500.0000]

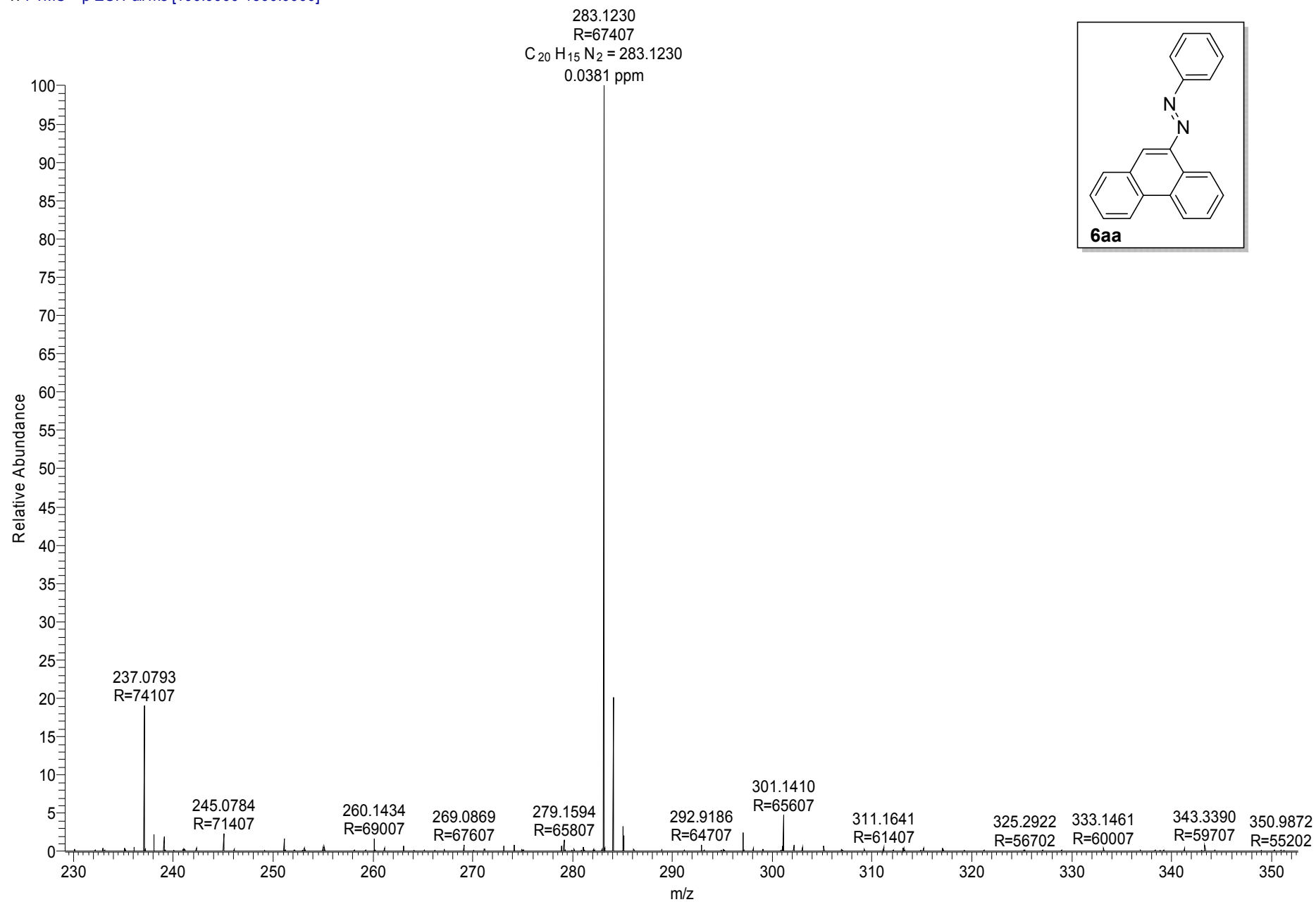


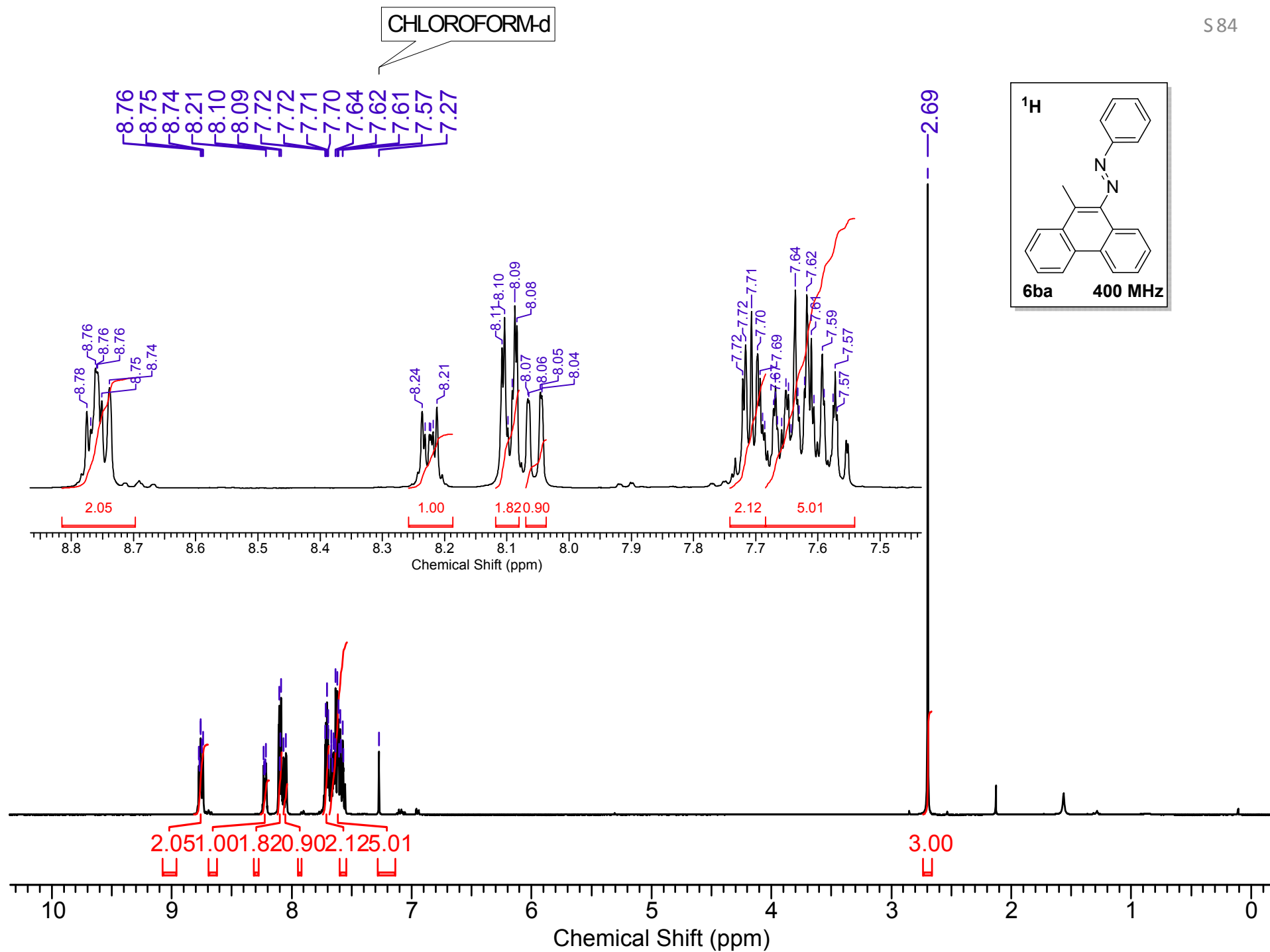


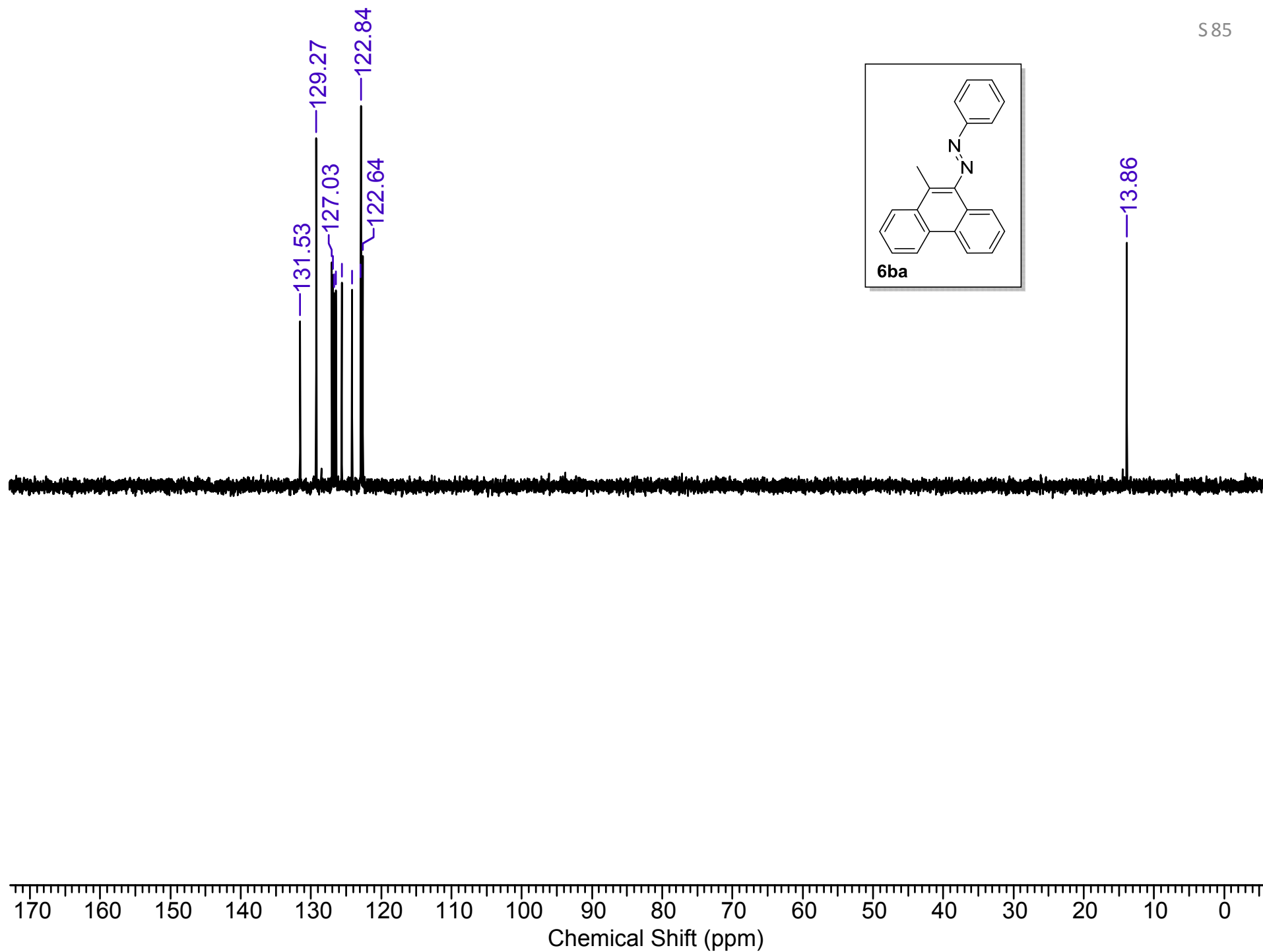
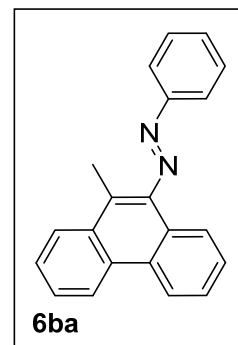


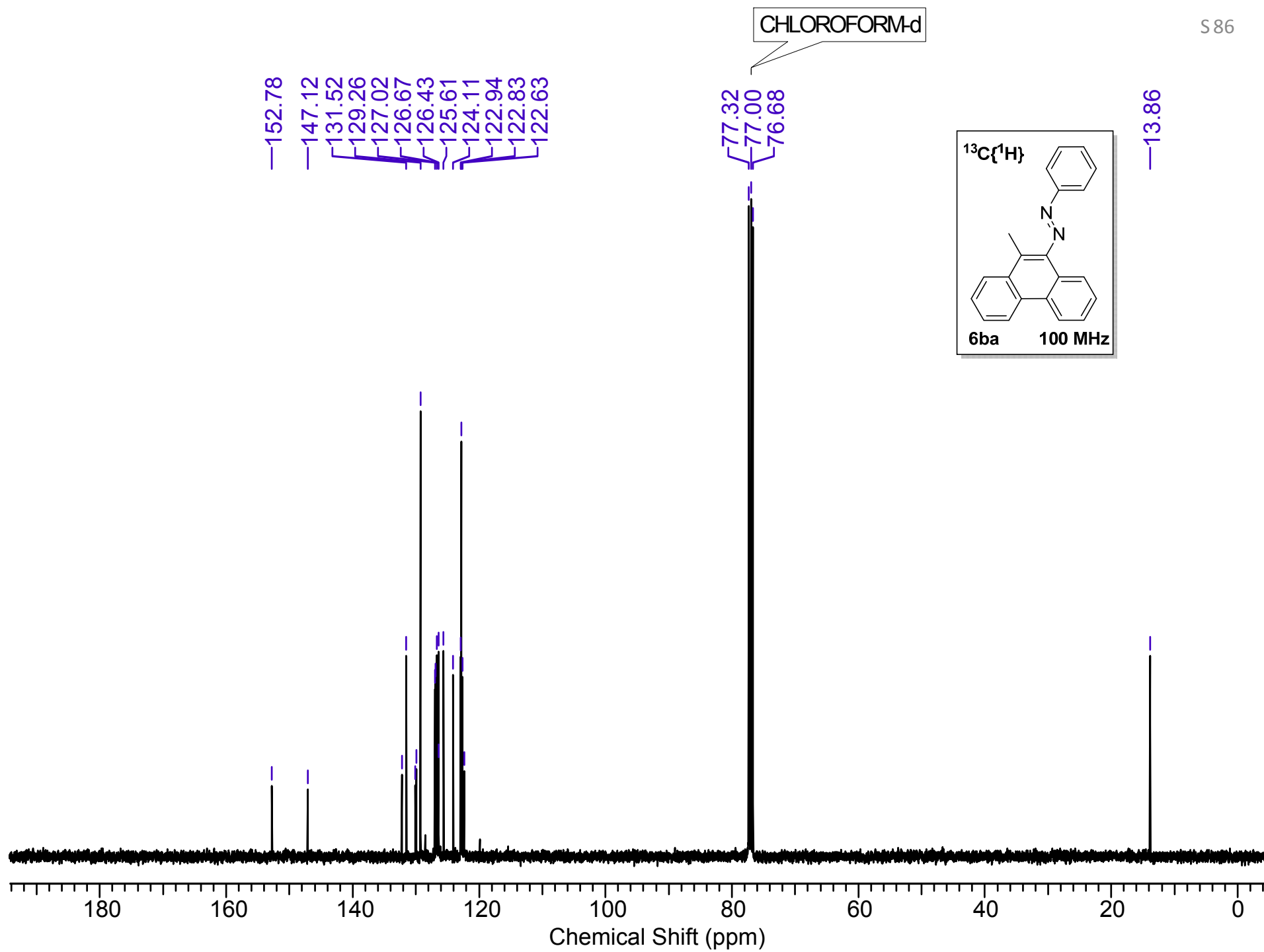


PSD-917_210505153209 #315 RT: 1.76 AV: 1 NL: 4.34E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

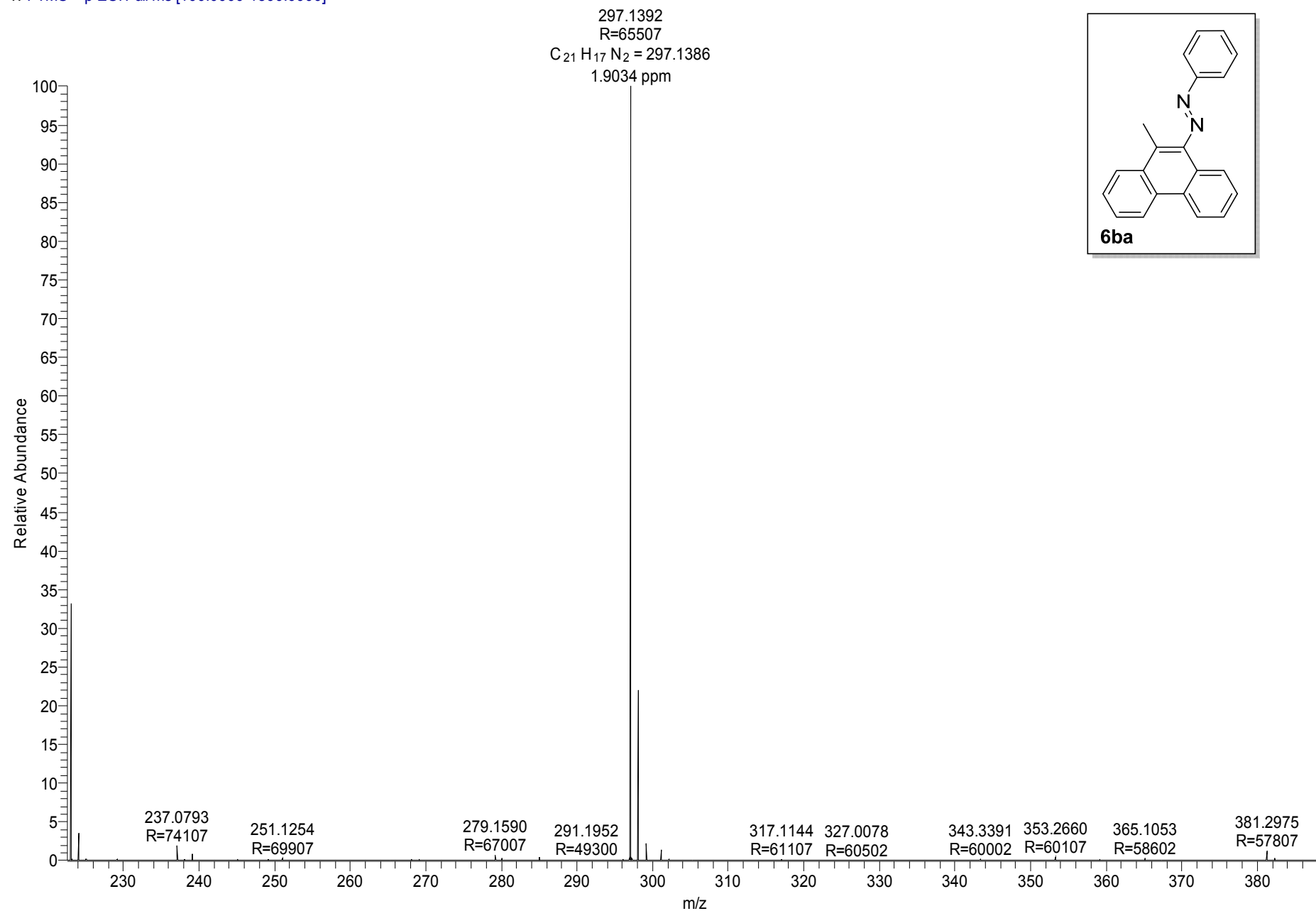


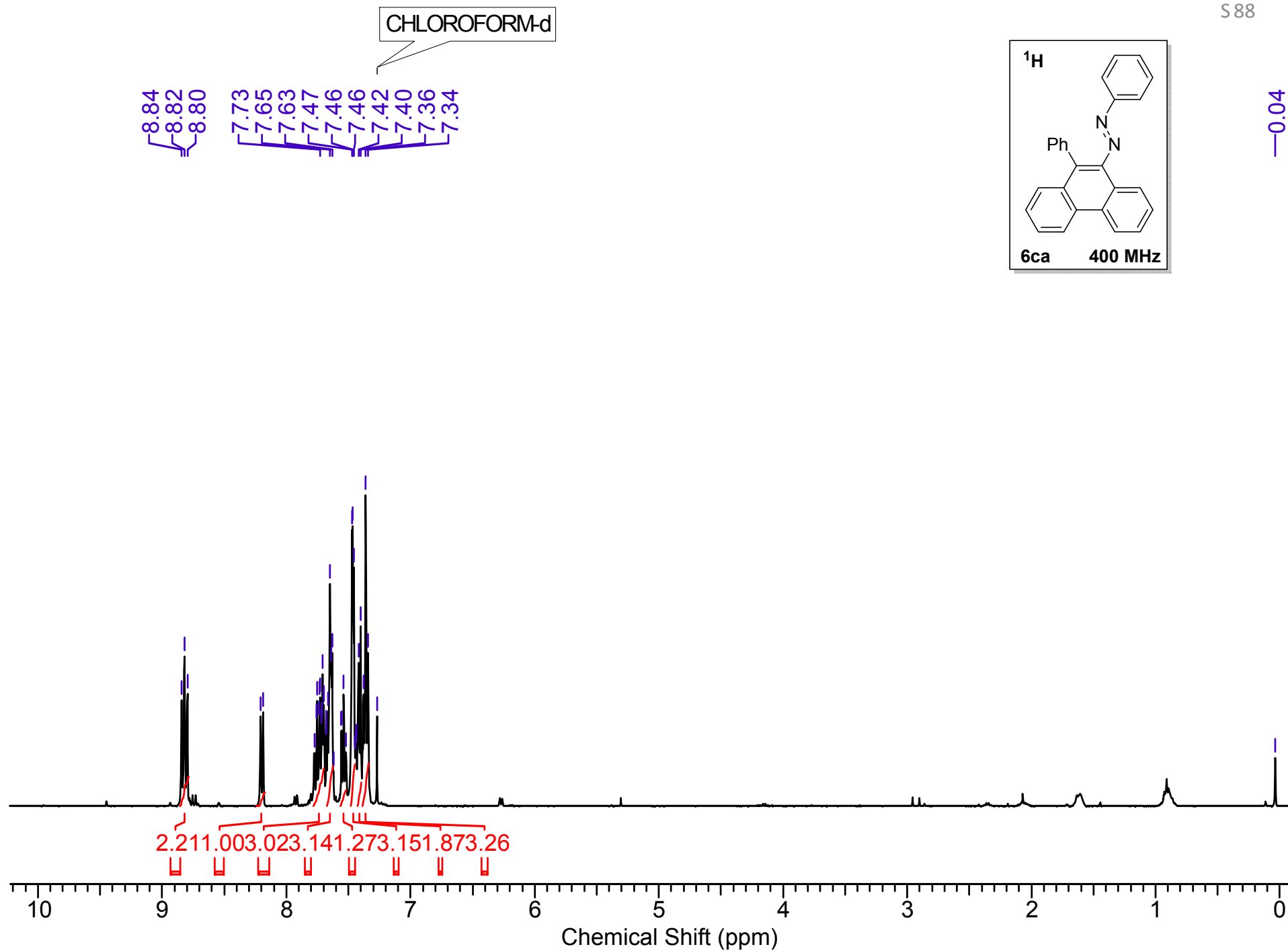


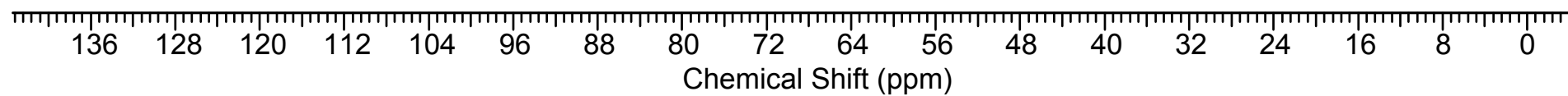
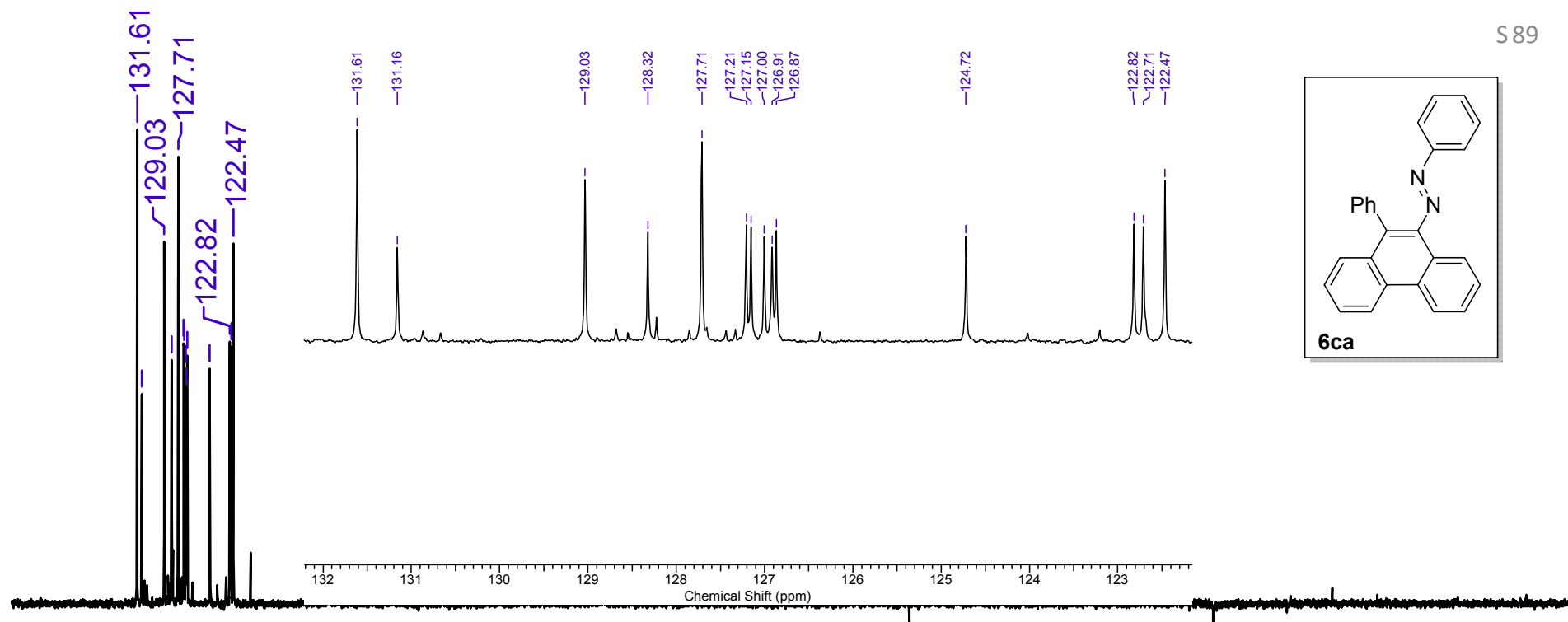


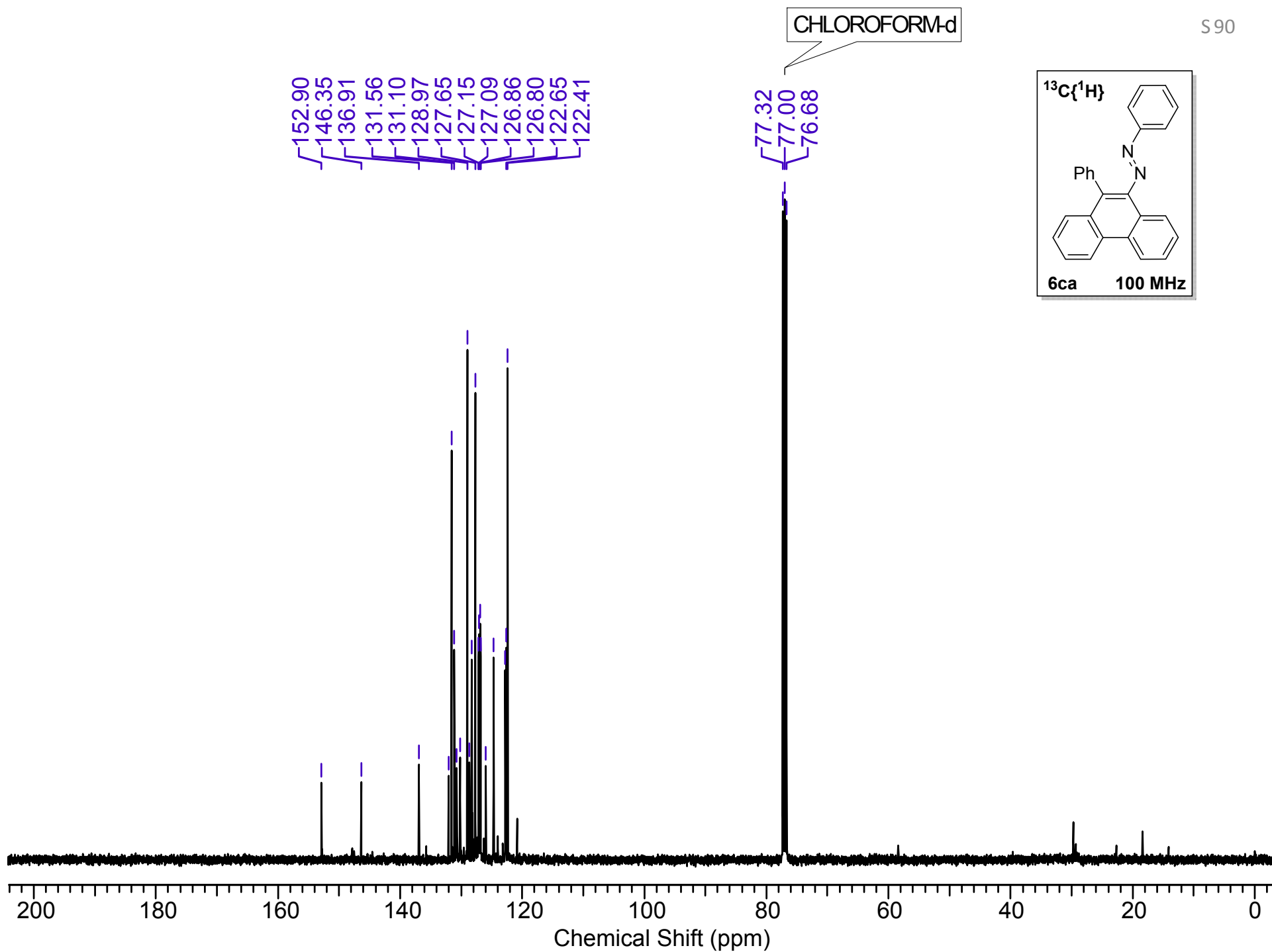


PSD-Simple #516 RT: 2.77 AV: 1 NL: 5.21E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]



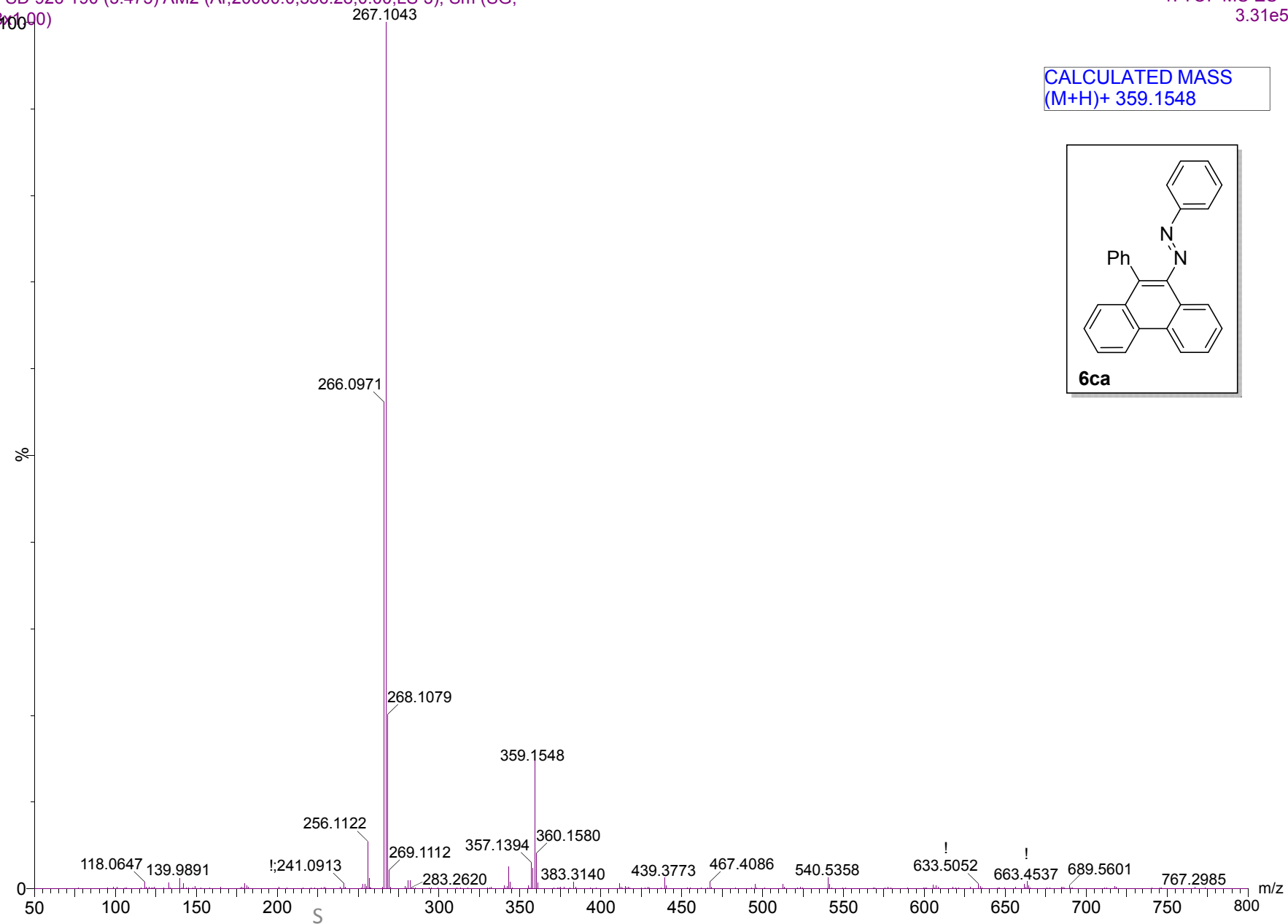
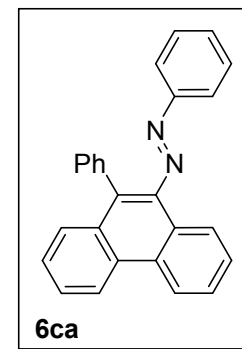


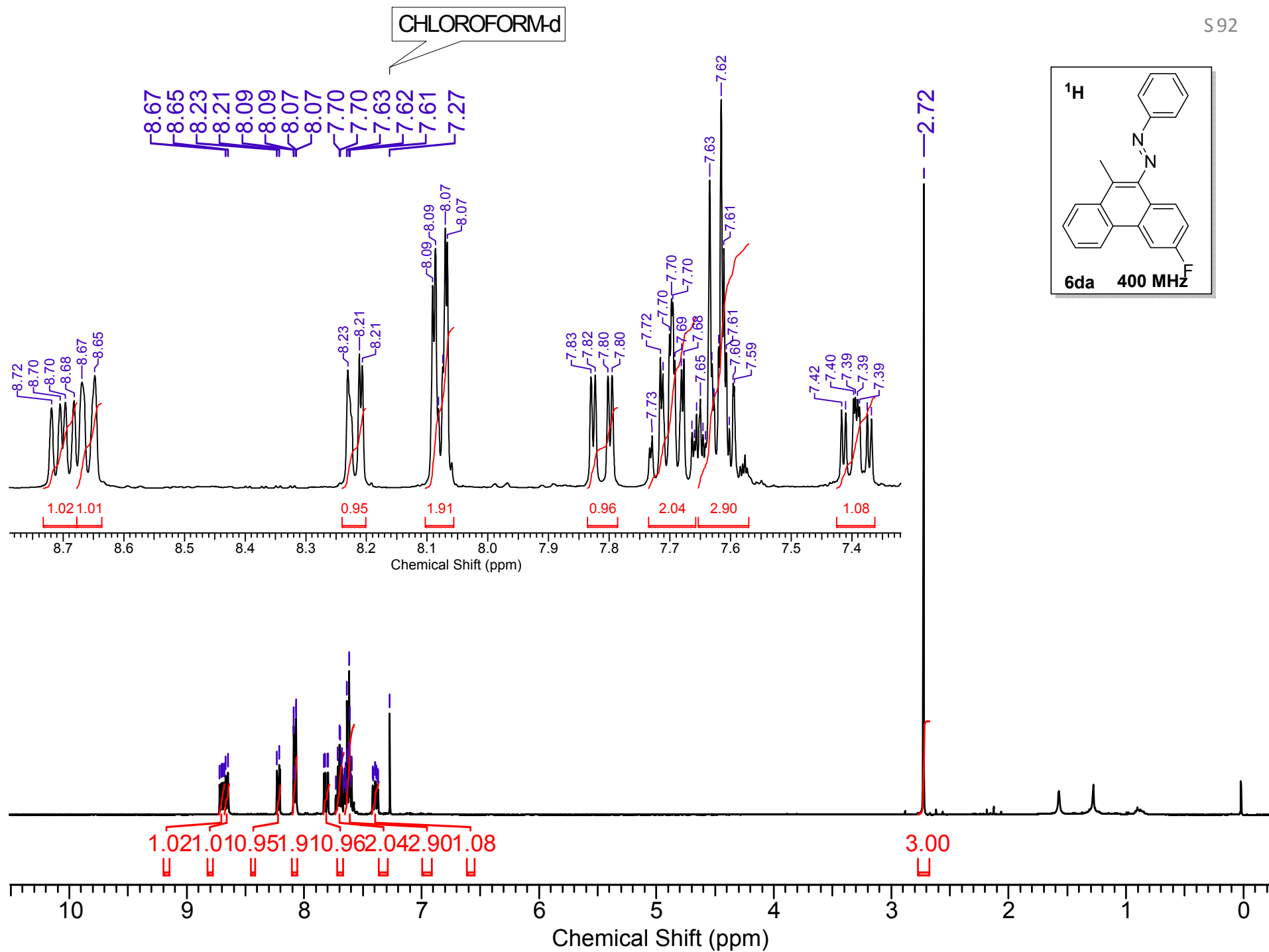


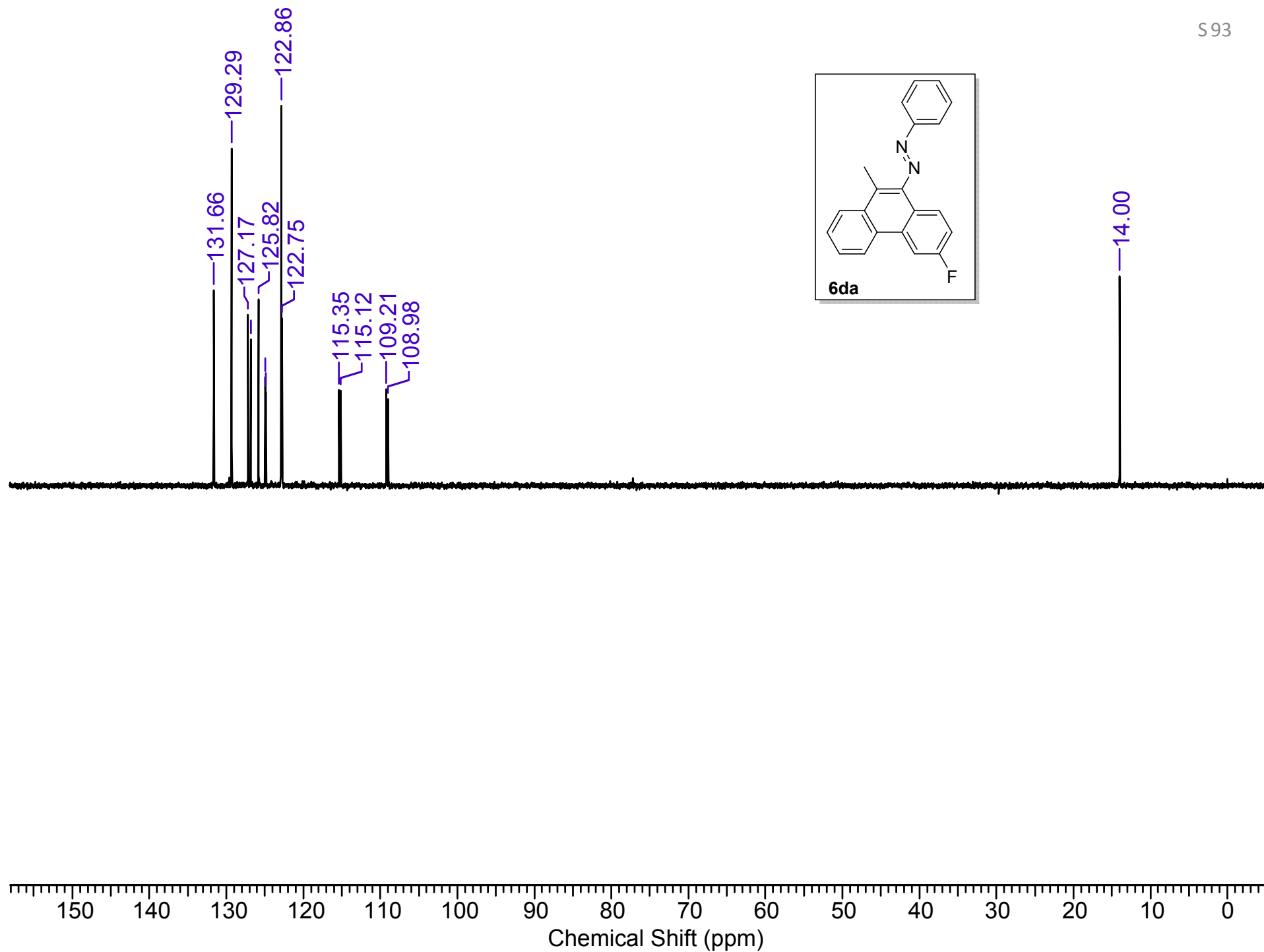
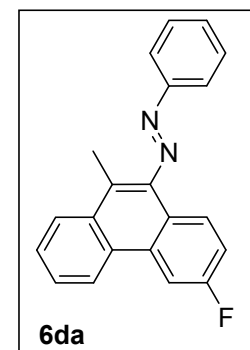


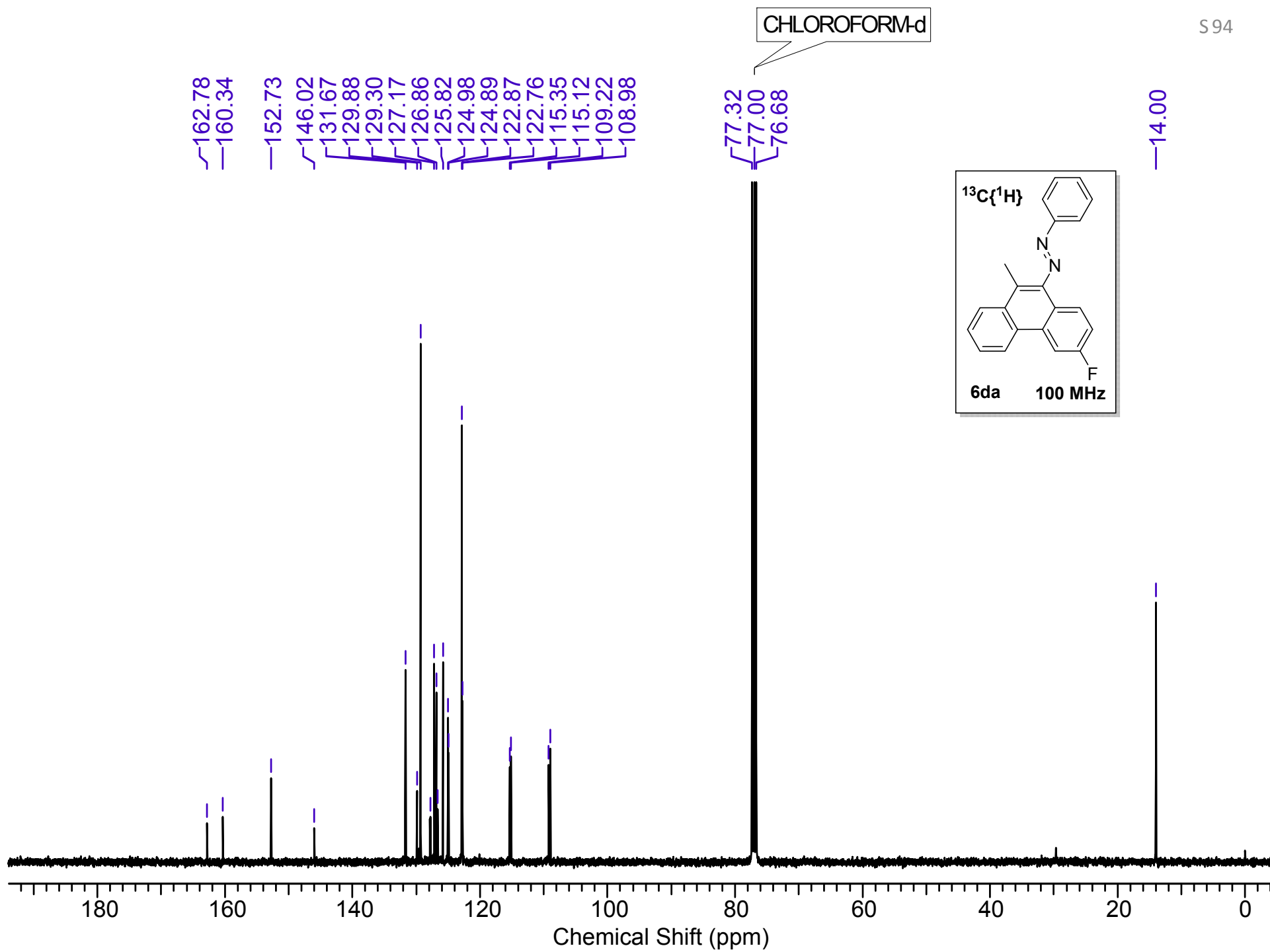
PSD 926

IISER PUNE

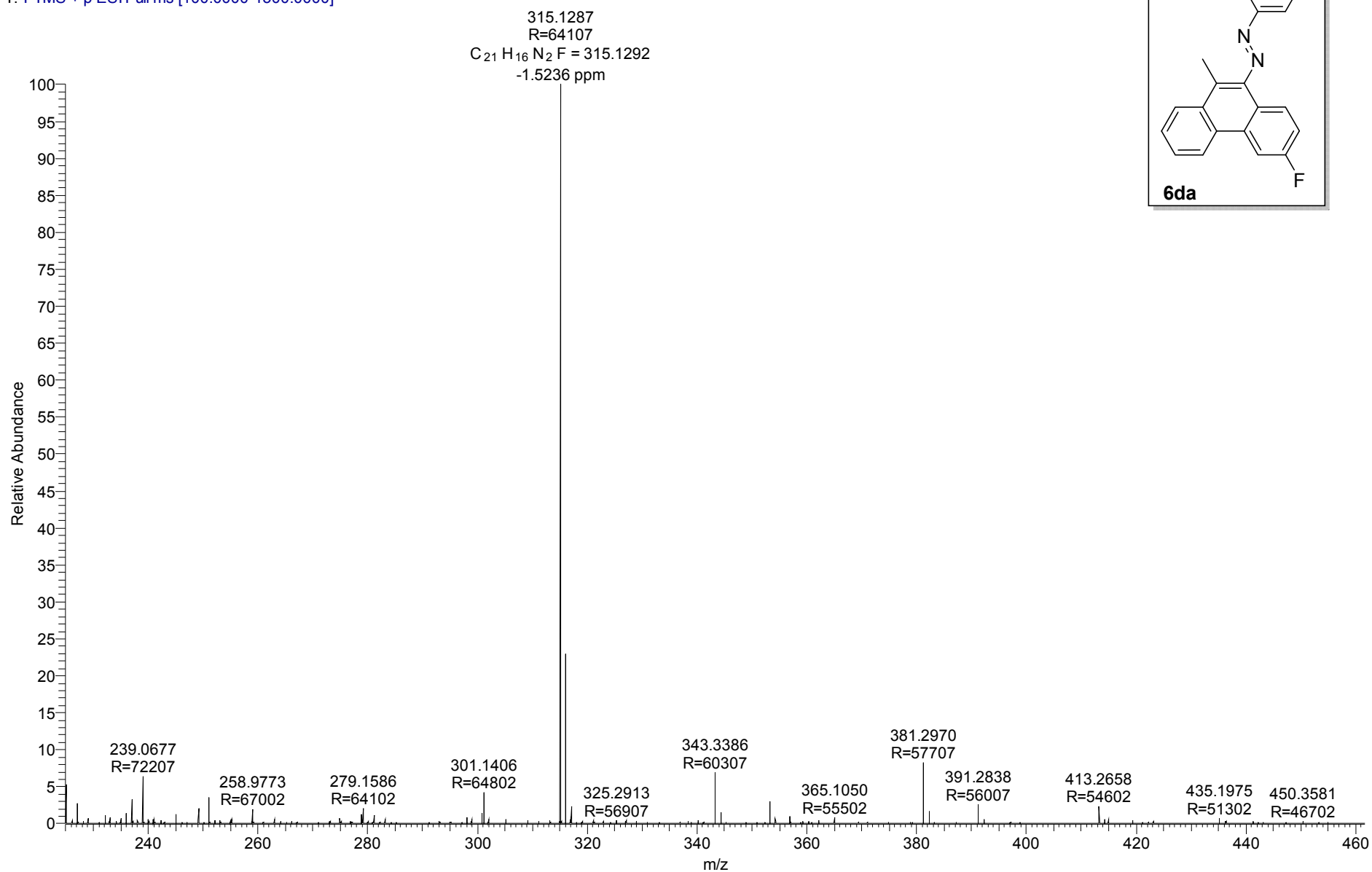
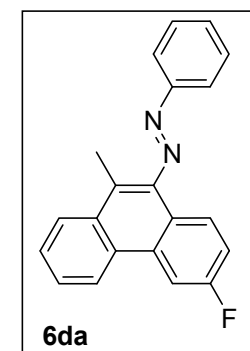
1: TOF MS ES+
3.31e5PSD 926 190 (3.475) AM2 (Ar,20000.0,556.28,0.00,LS 3); Sm (SG,
300.00)CALCULATED MASS
(M+H)+ 359.1548

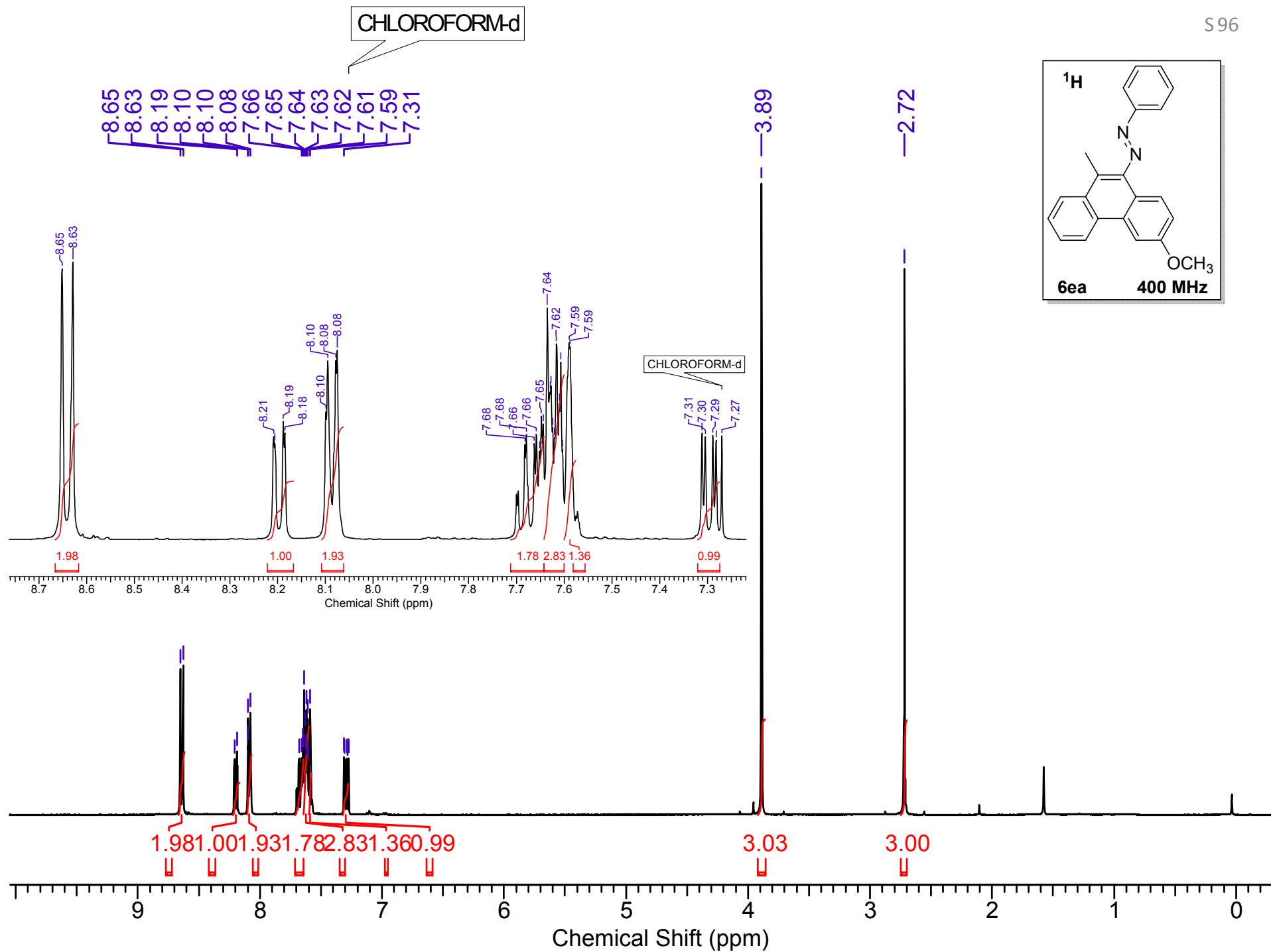


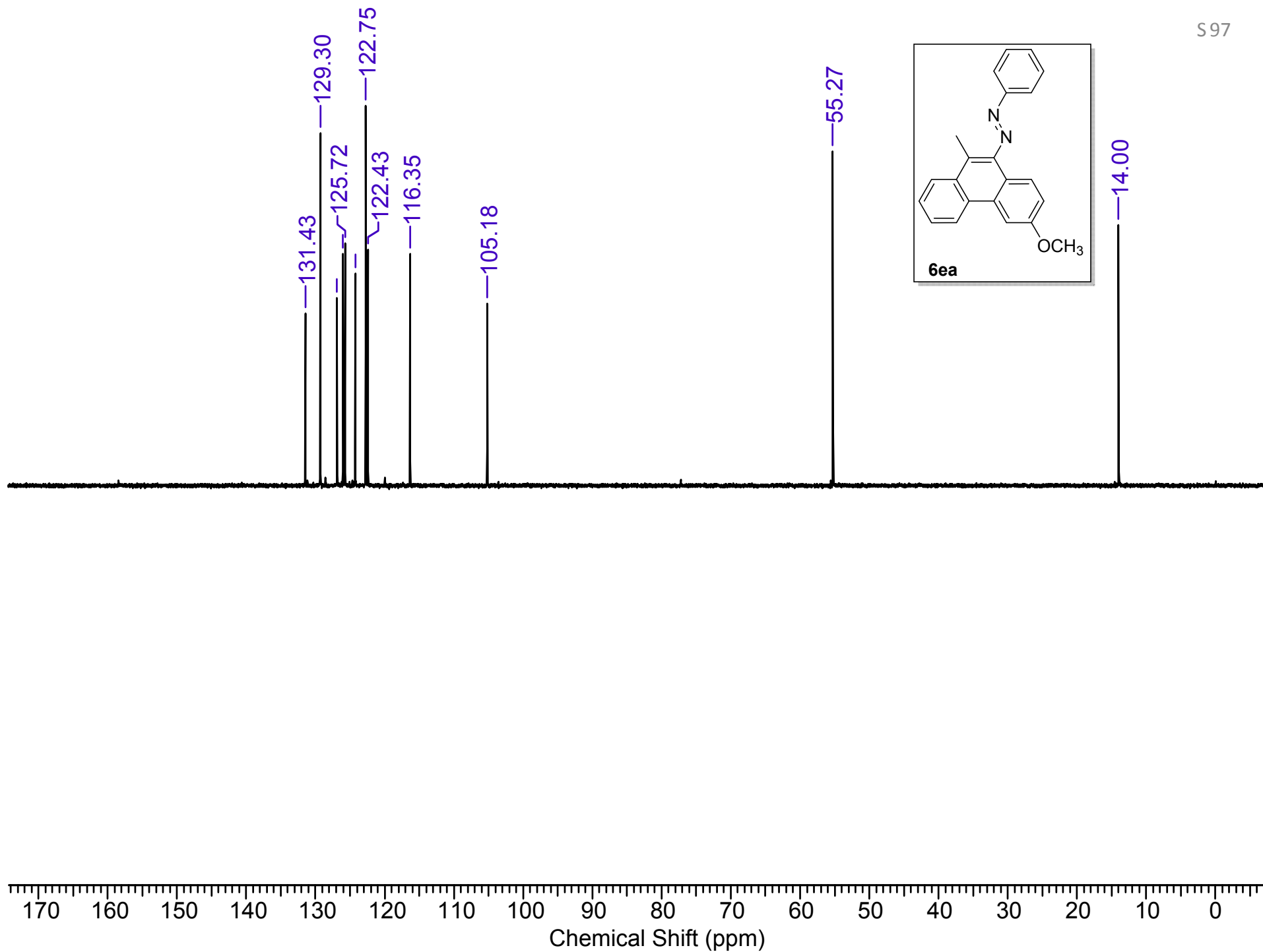
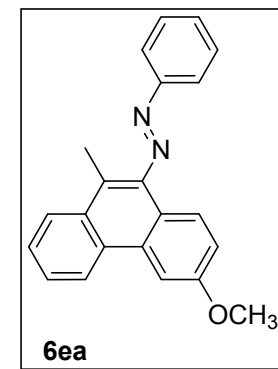


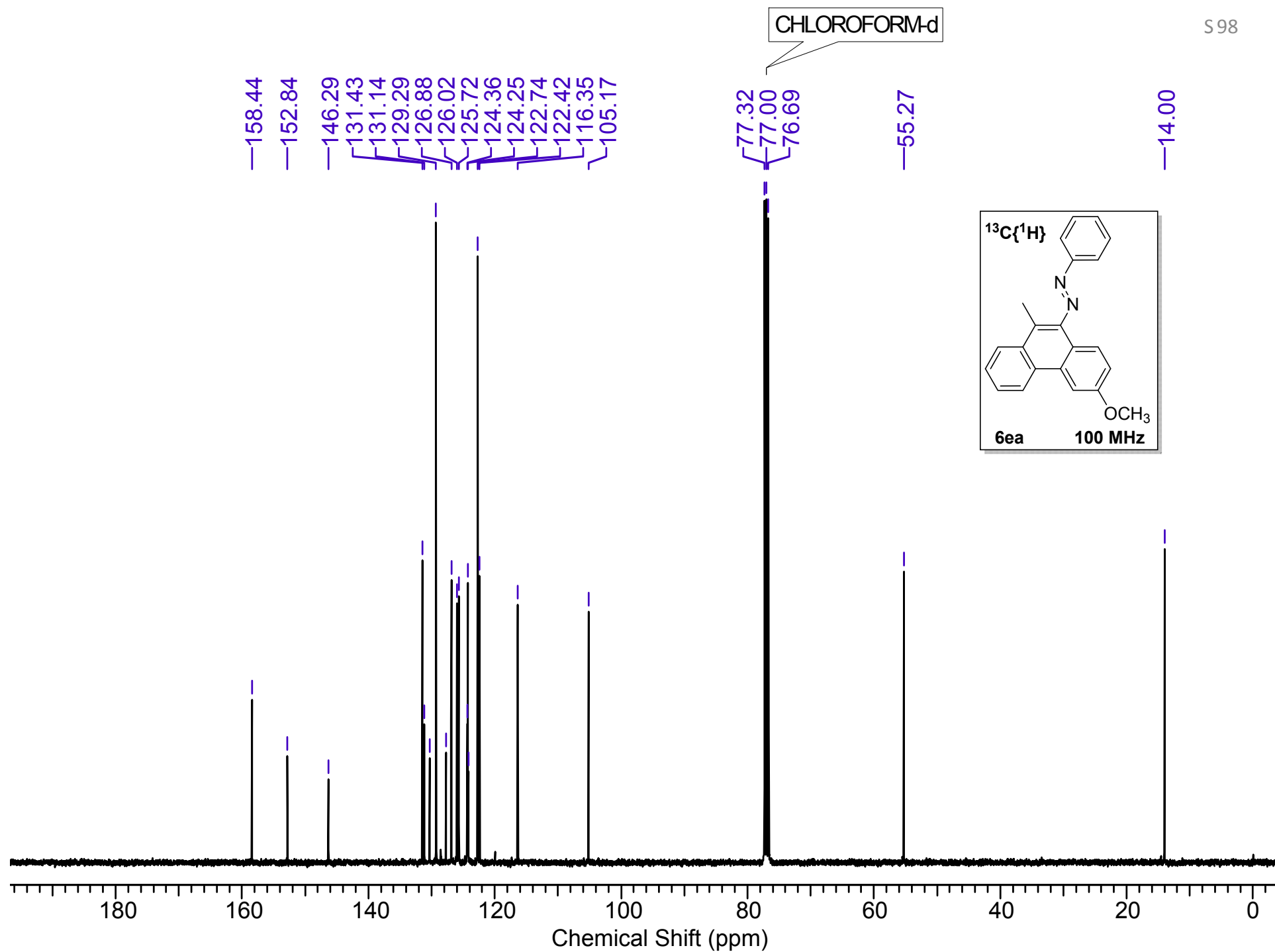


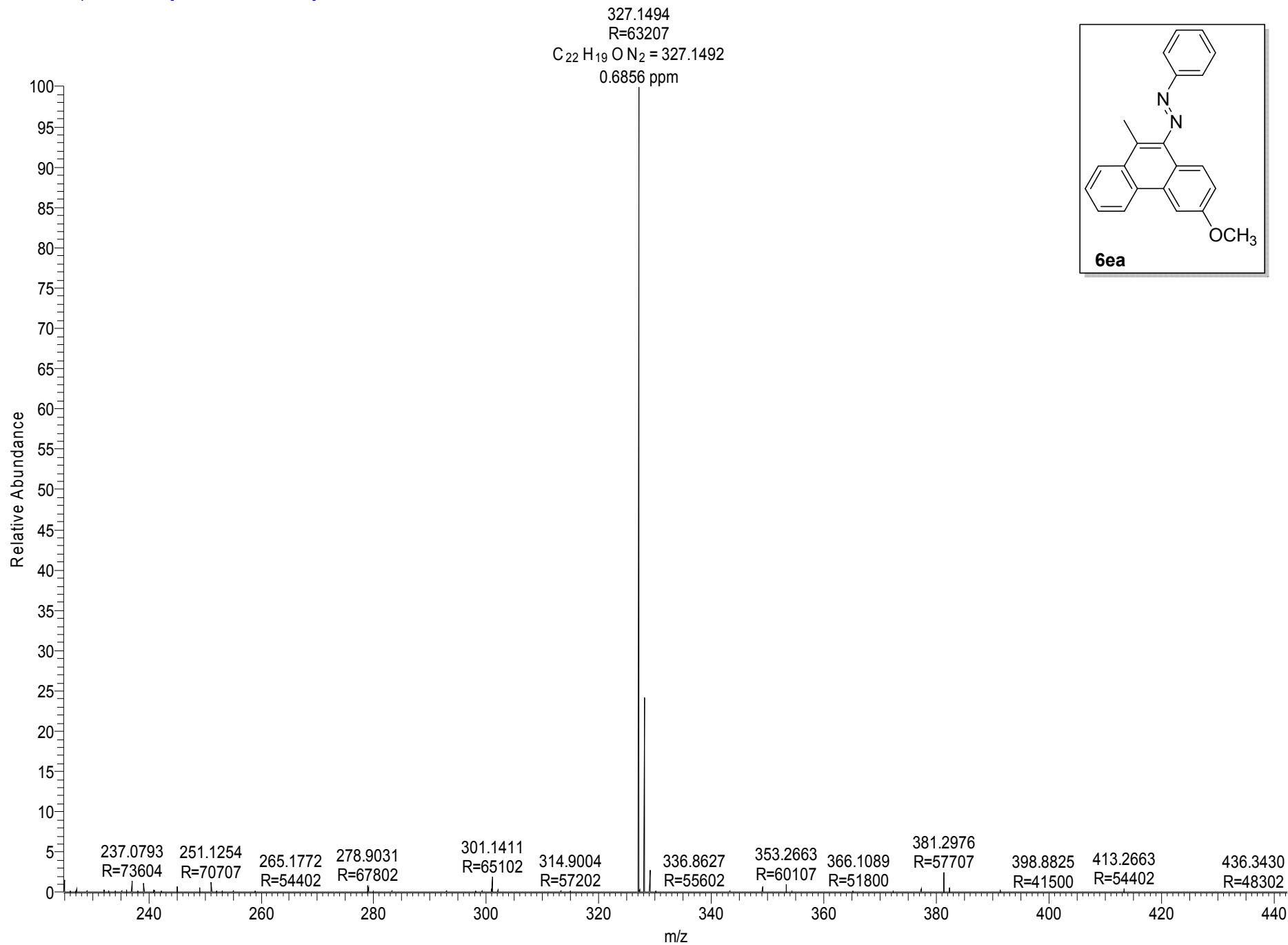
PSD-886 #515 RT: 3.12 AV: 1 NL: 9.75E6
T: FTMS + p ESI Full ms [100.0000-1500.0000]



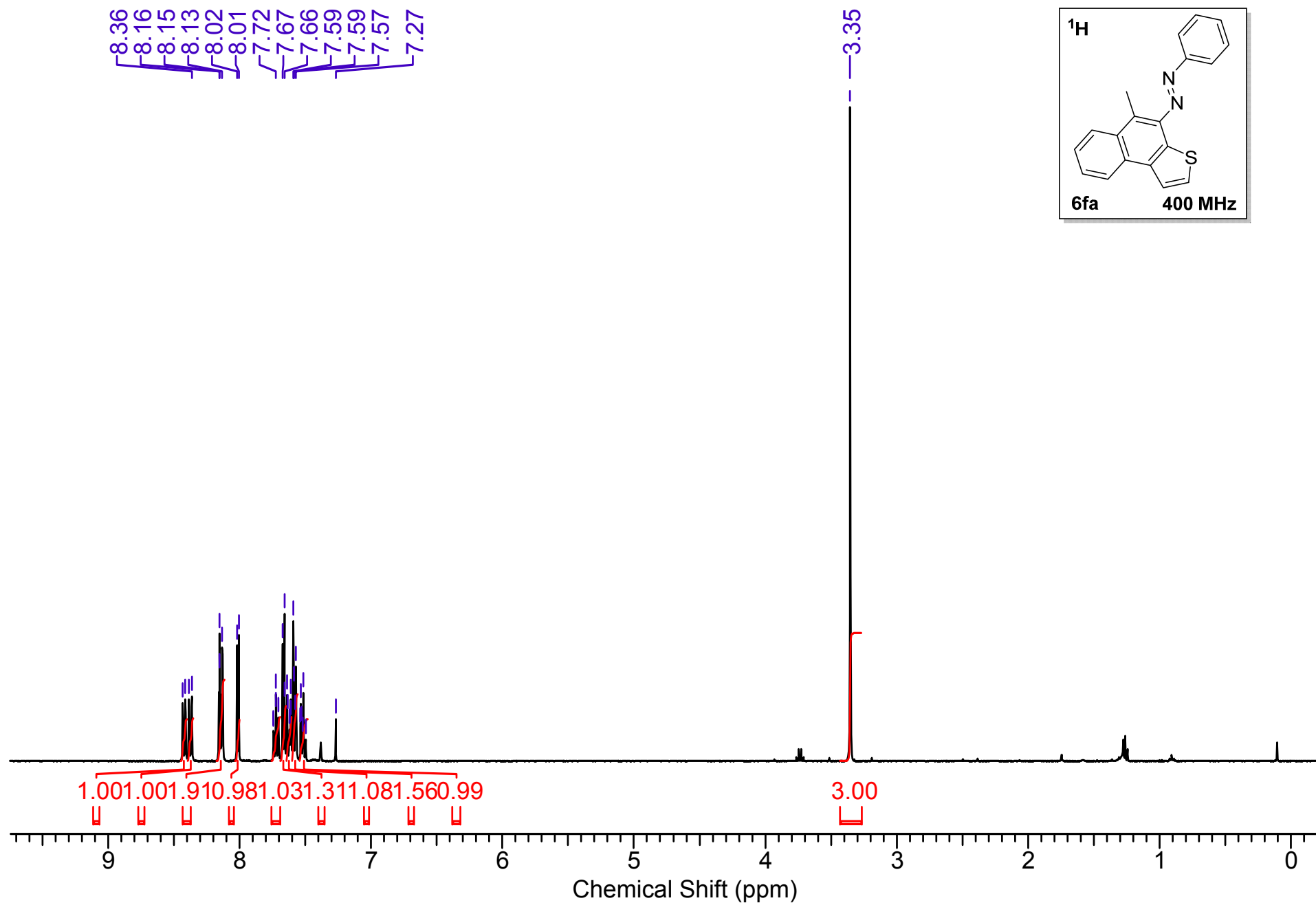


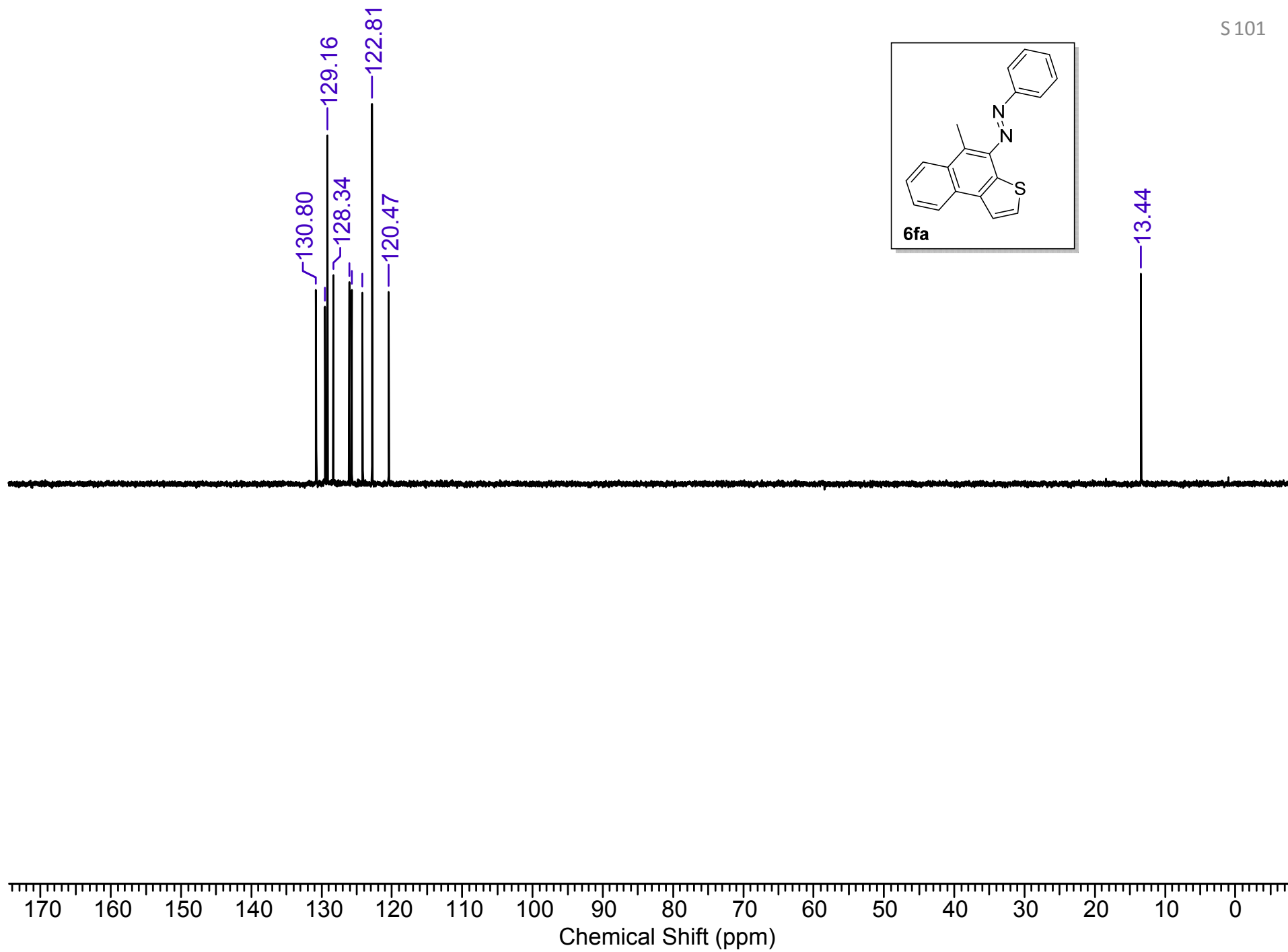
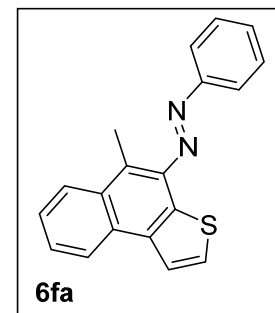


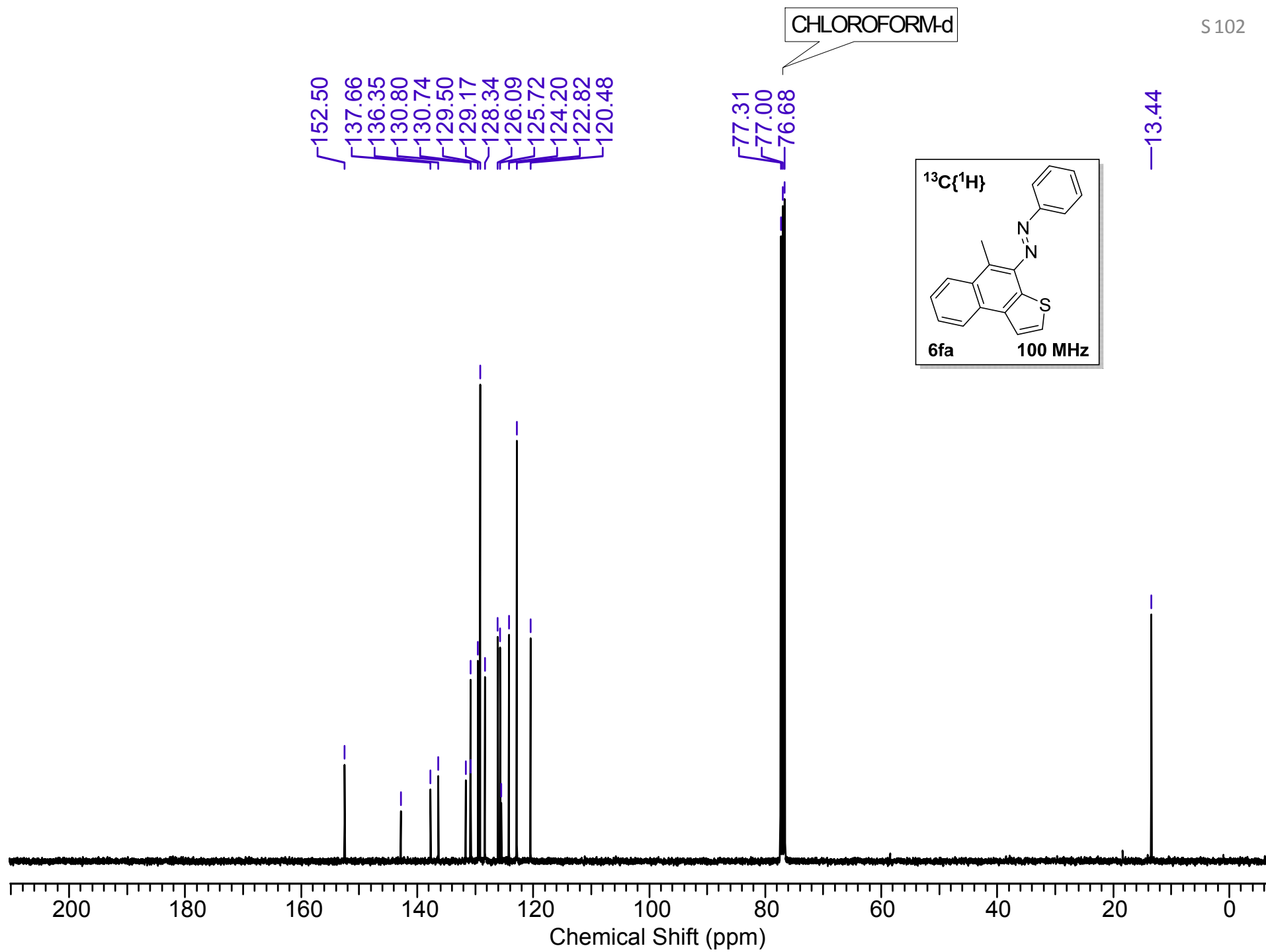




CHLOROFORM-d

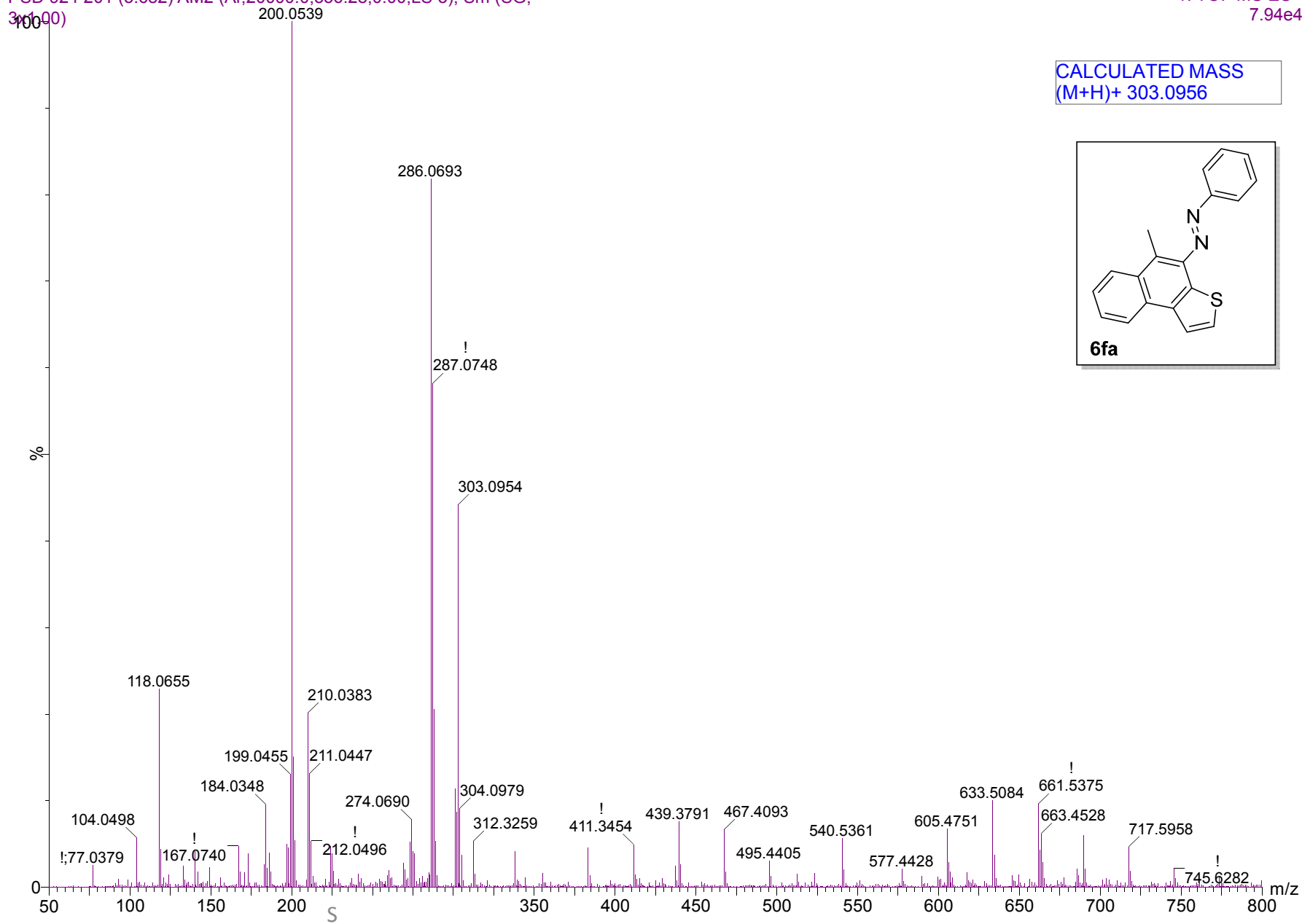
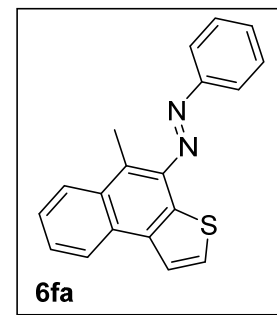


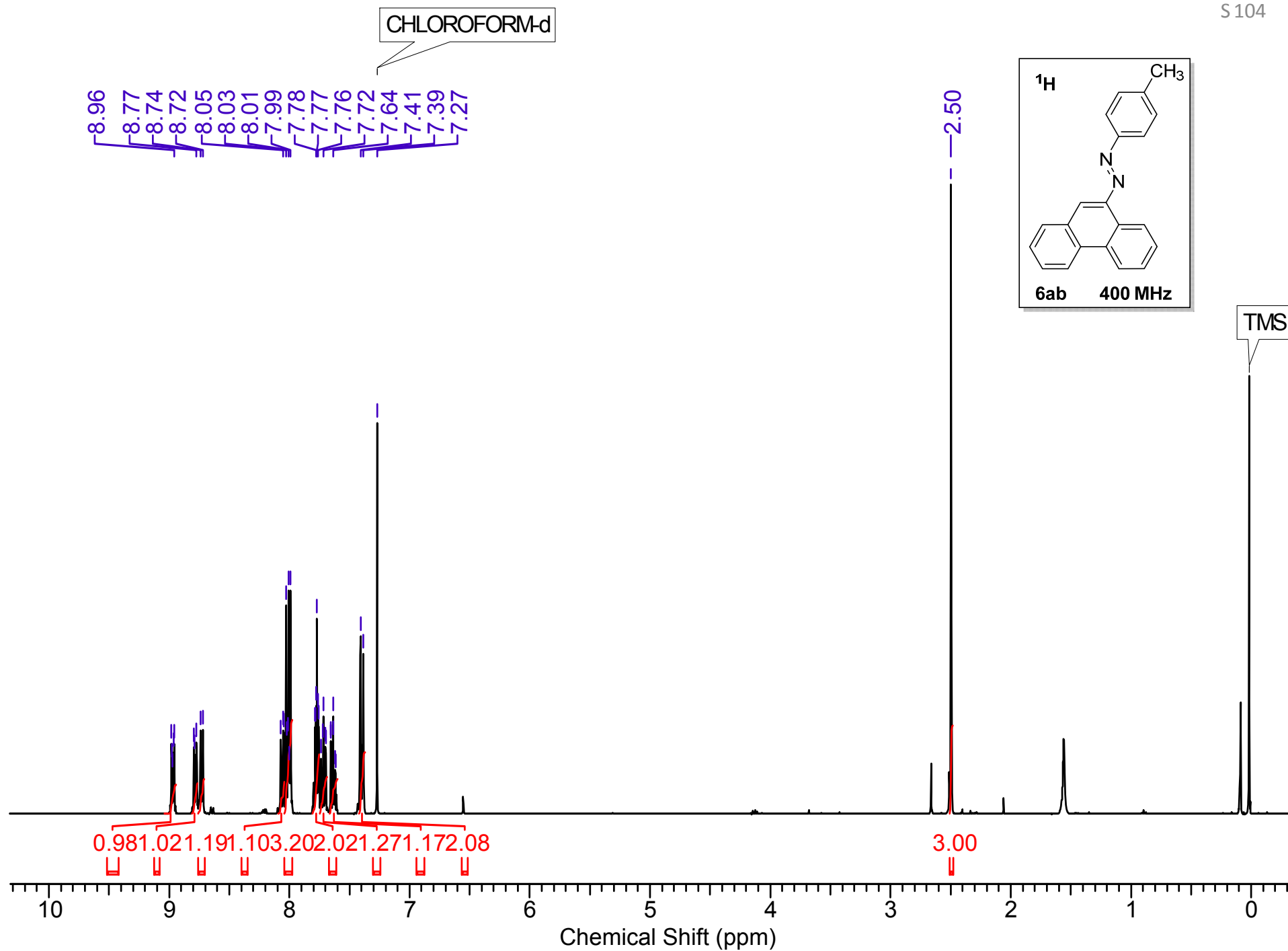


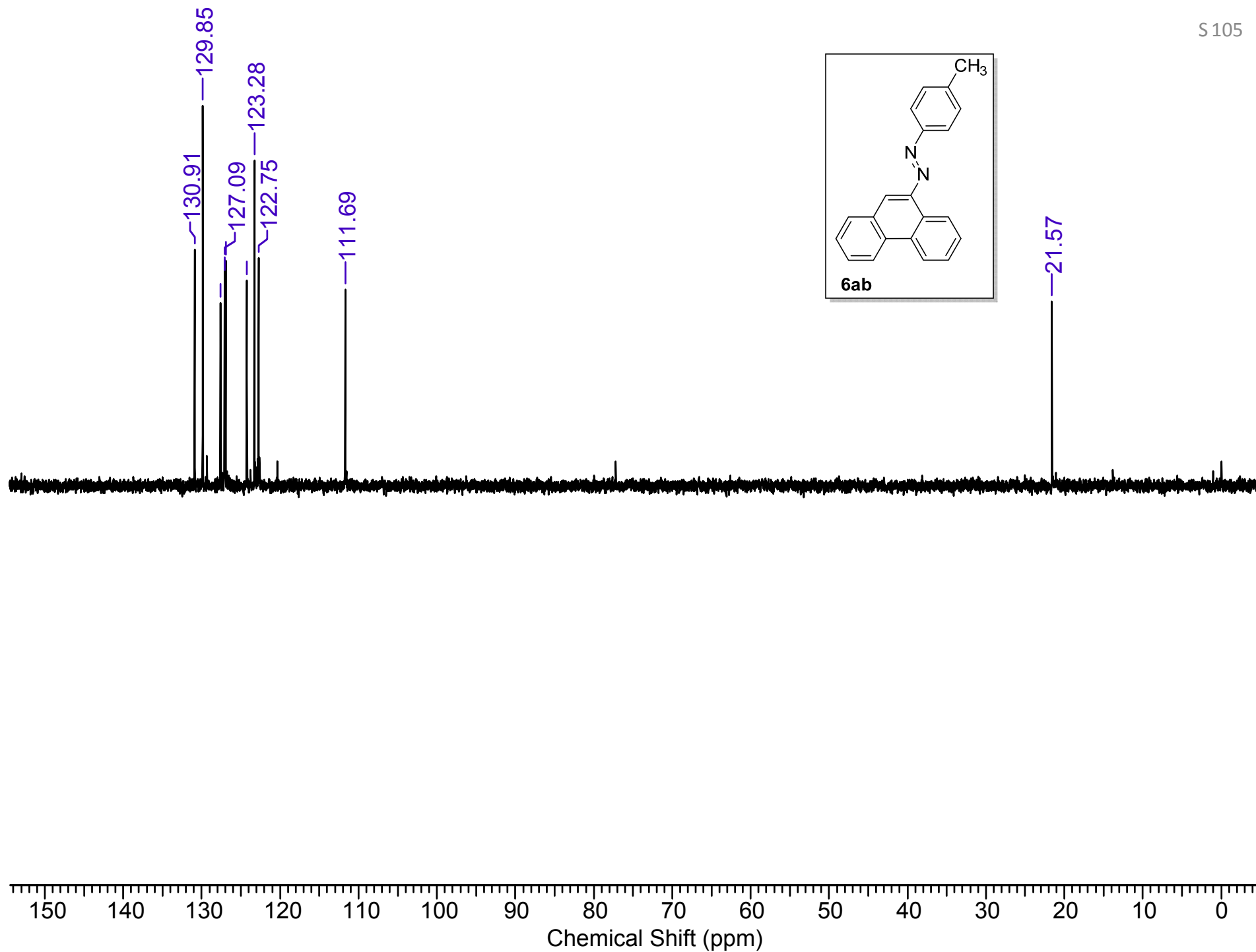
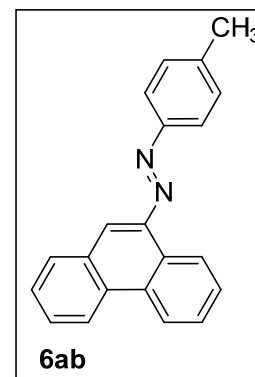


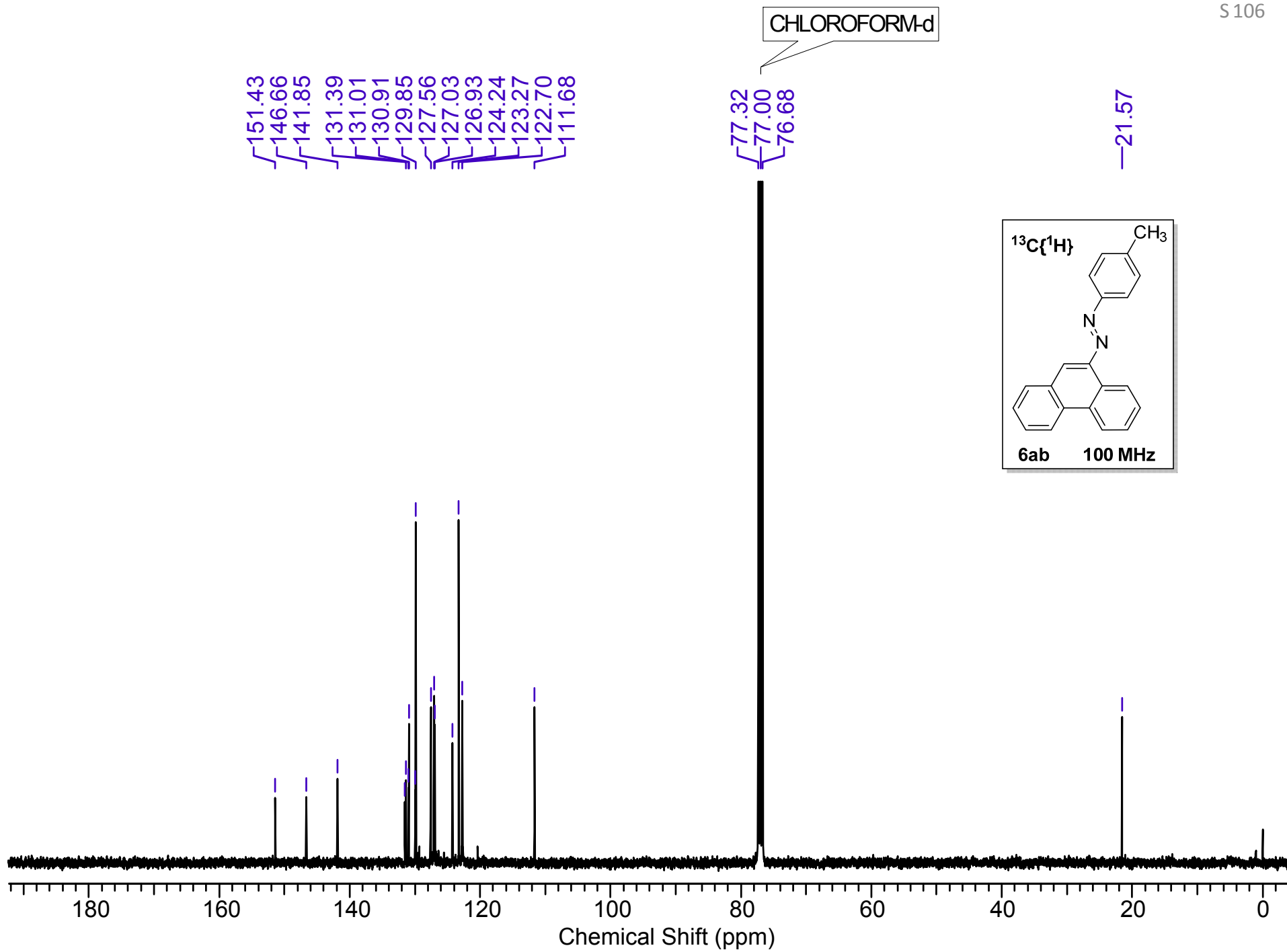
PSD 924

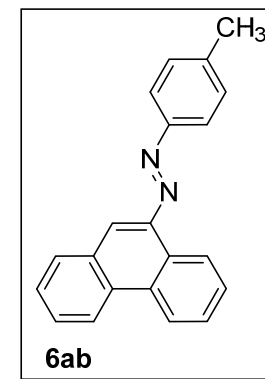
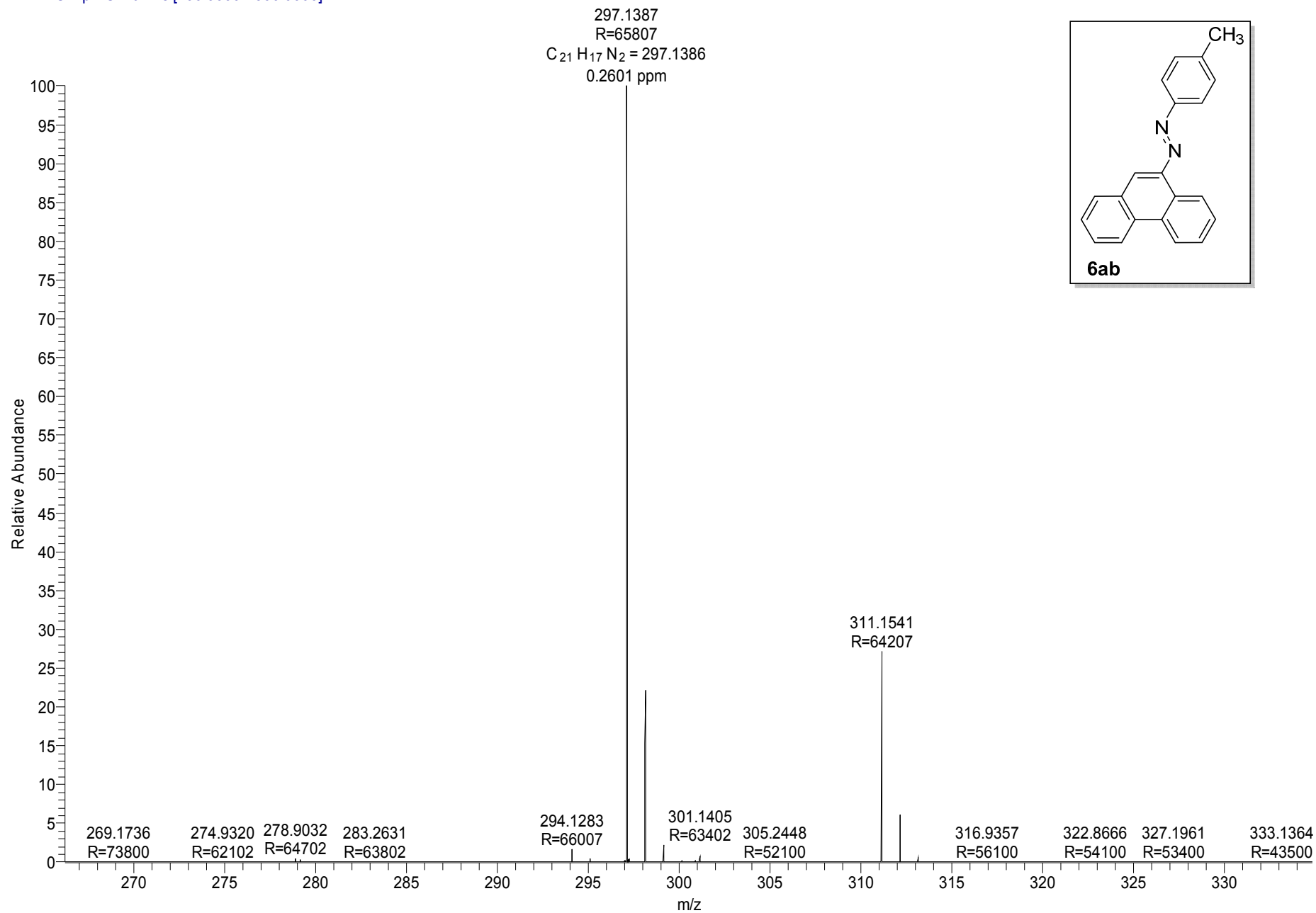
IISER PUNE

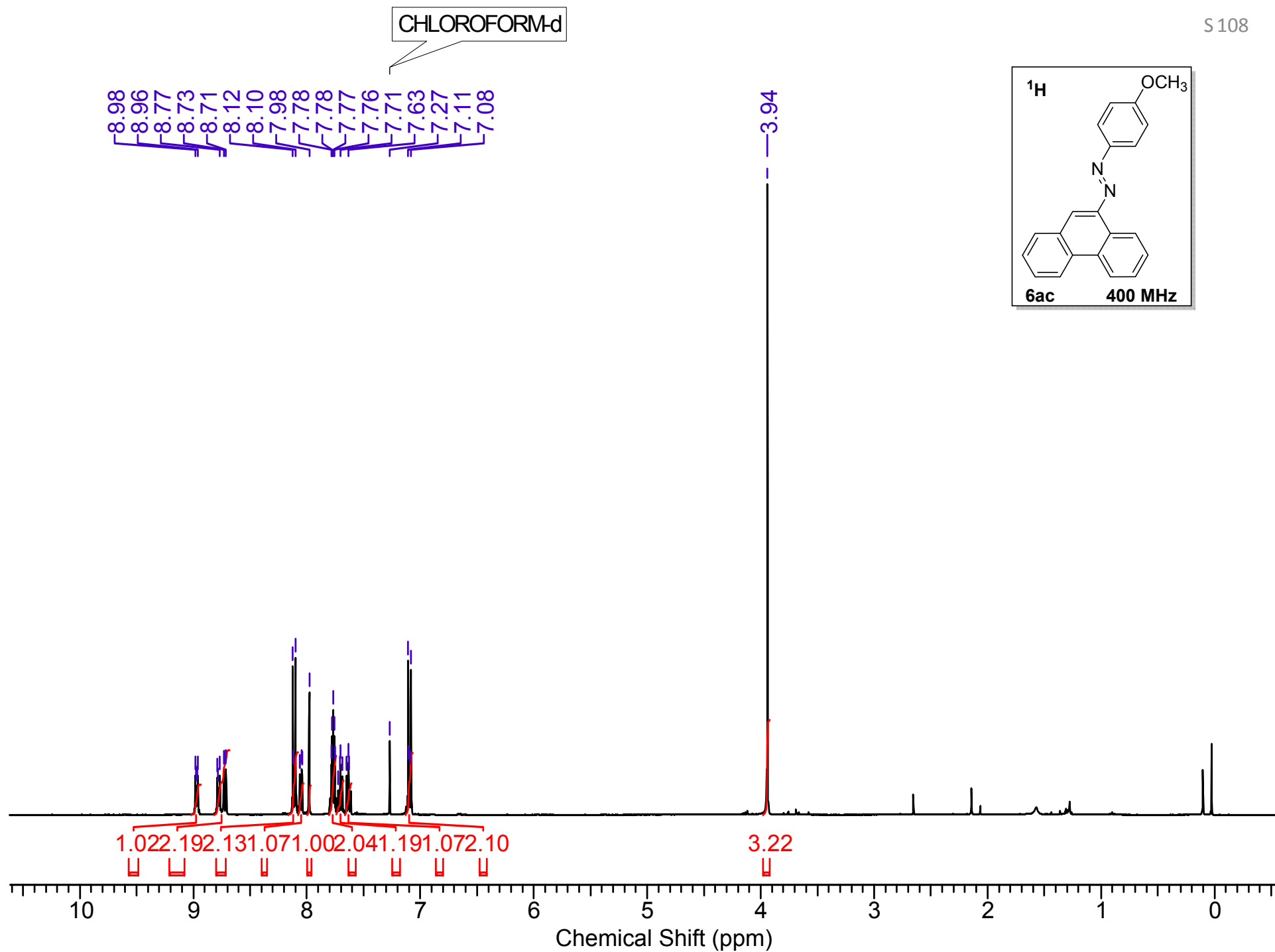
1: TOF MS ES+
7.94e4PSD 924 201 (3.682) AM2 (Ar,20000.0,556.28,0.00,LS 3); Sm (SG,
300.00)CALCULATED MASS
(M+H)+ 303.0956

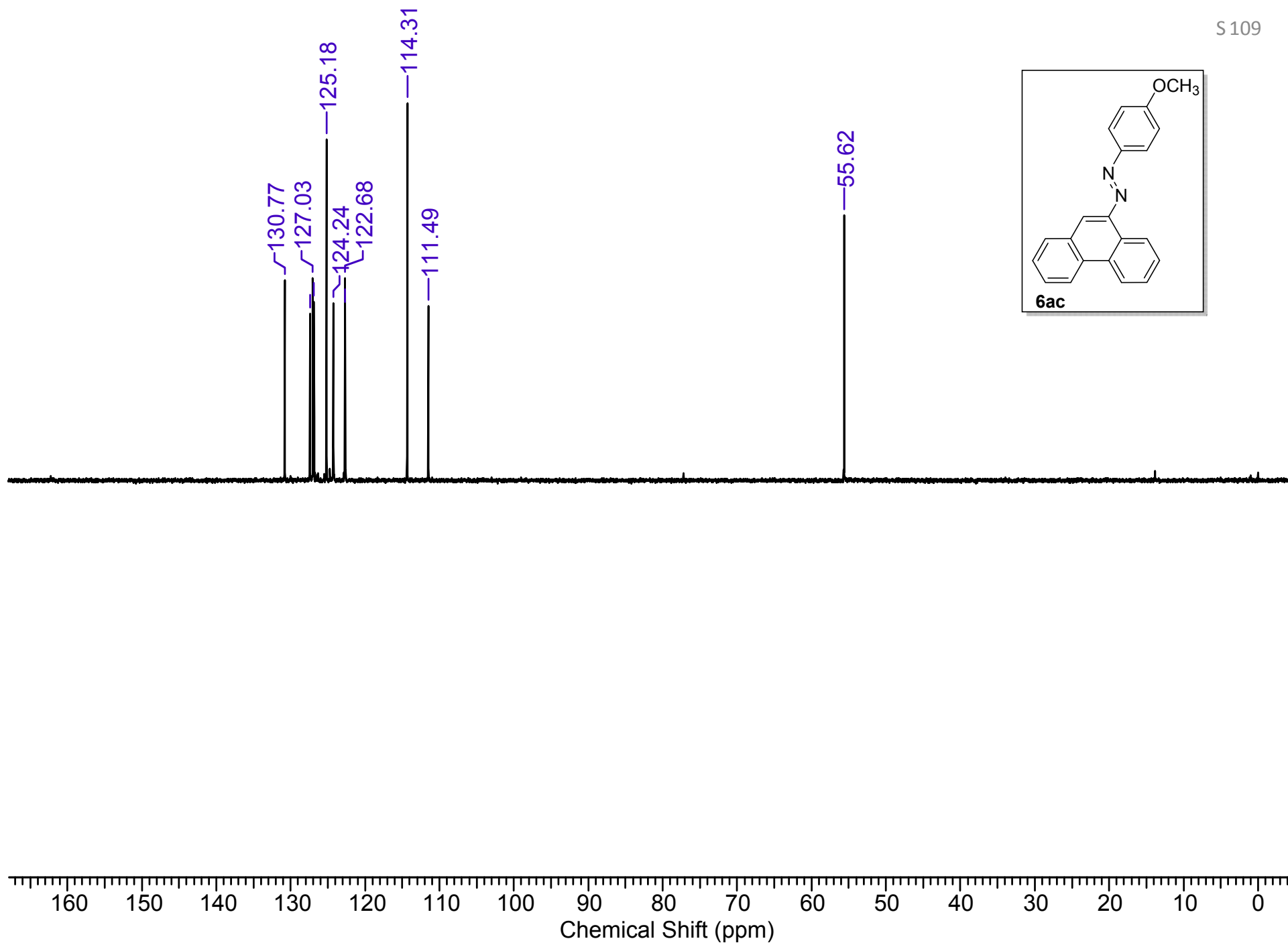


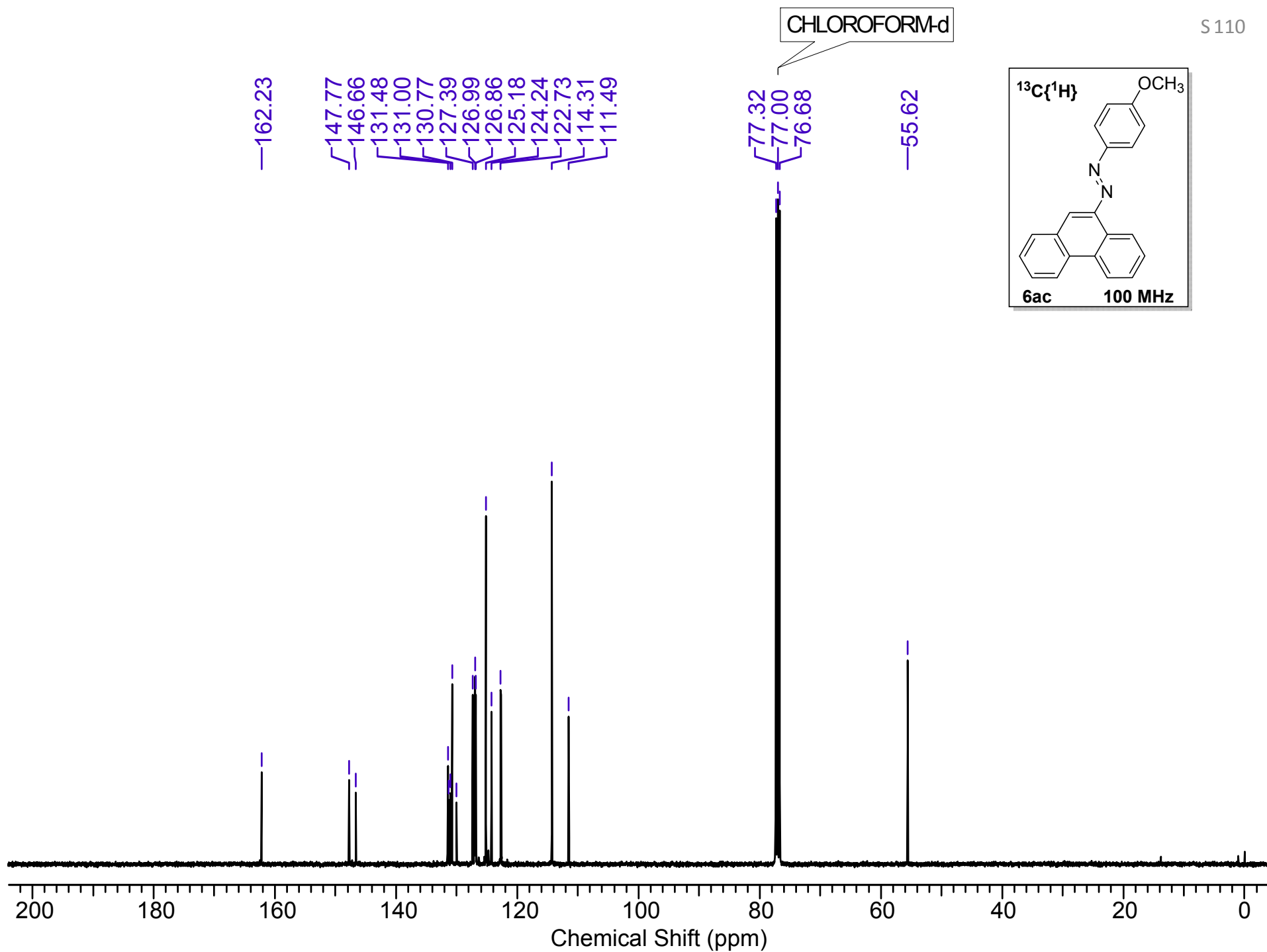


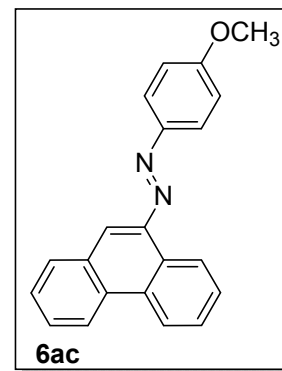
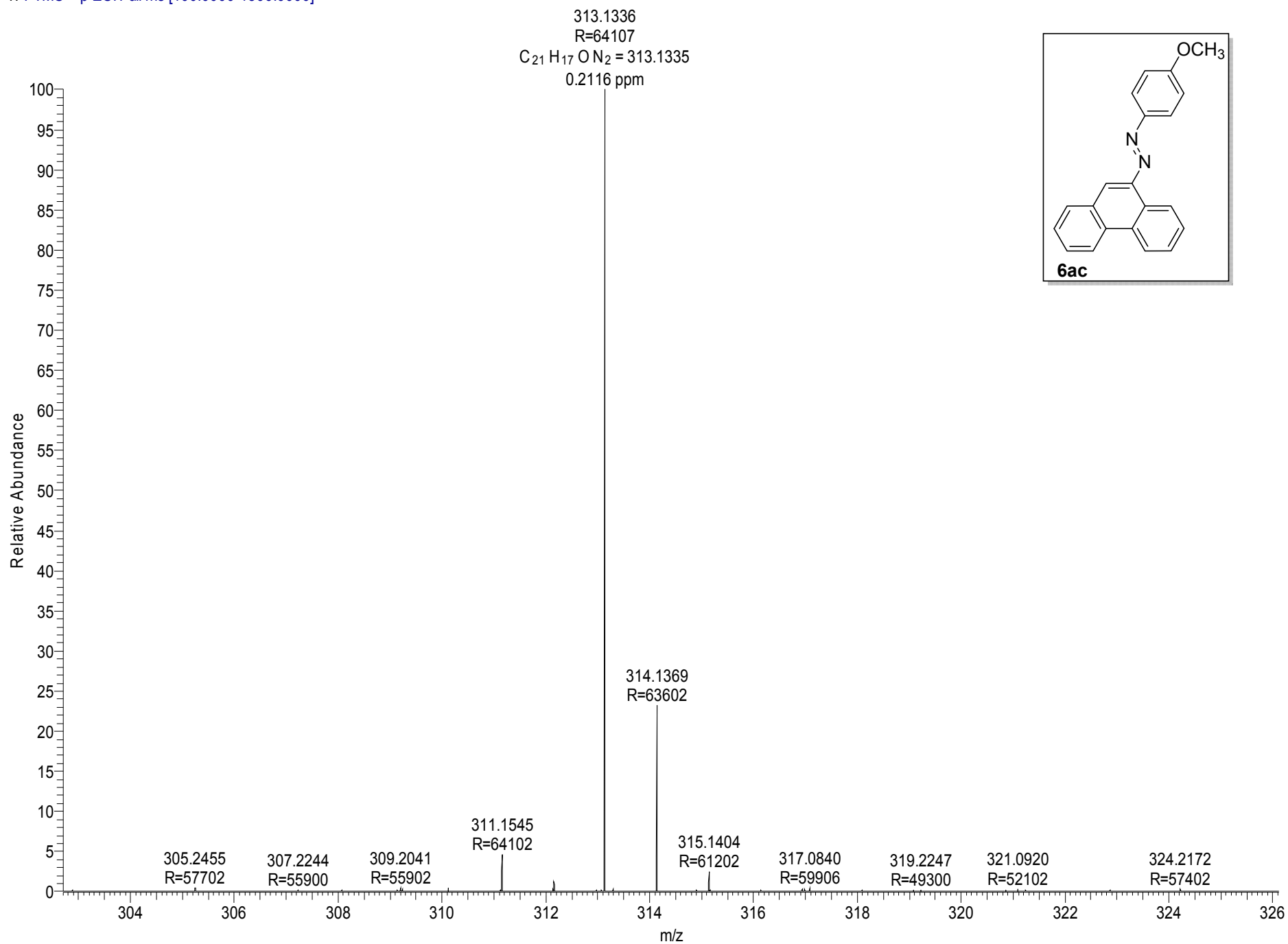


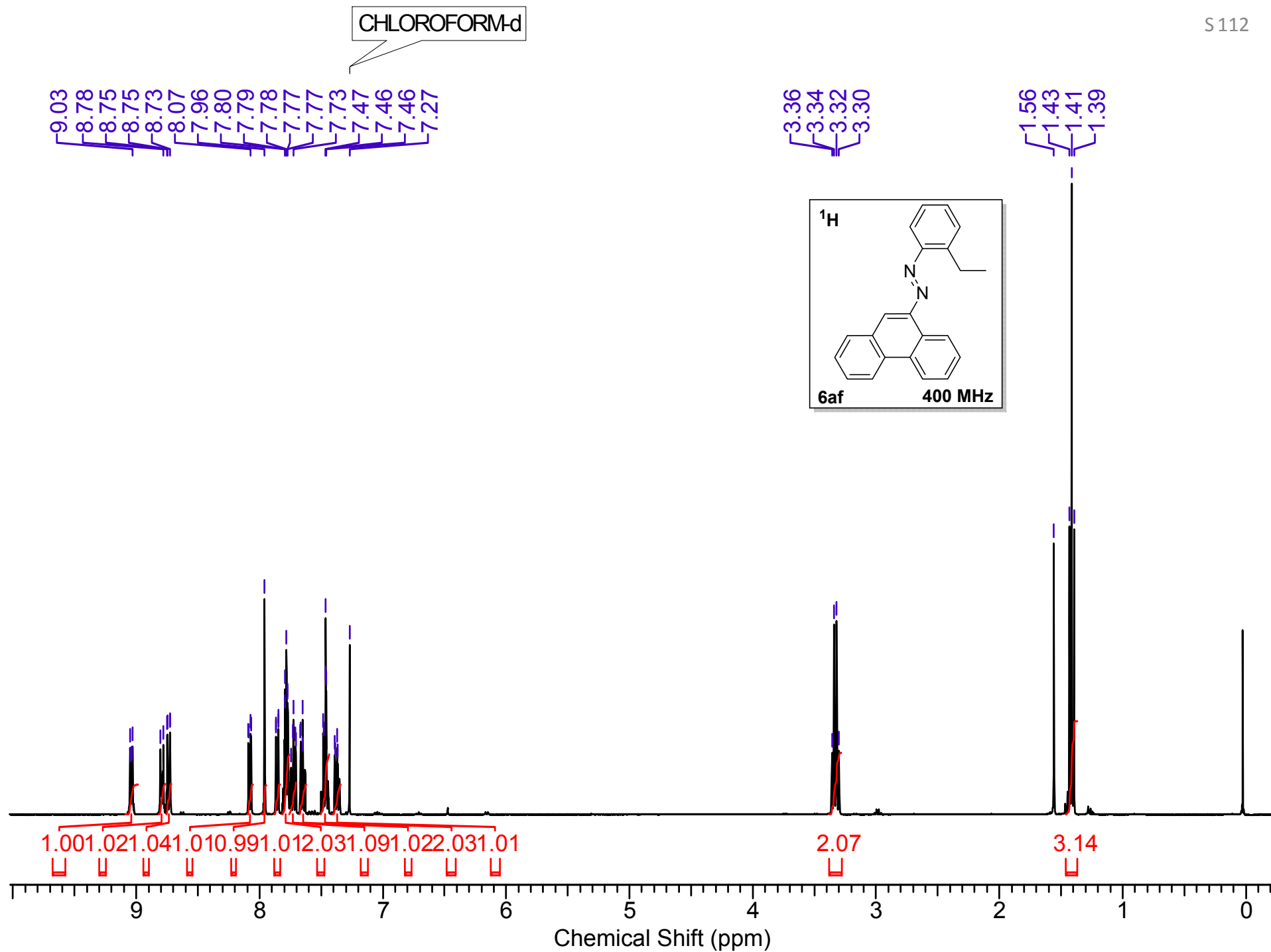


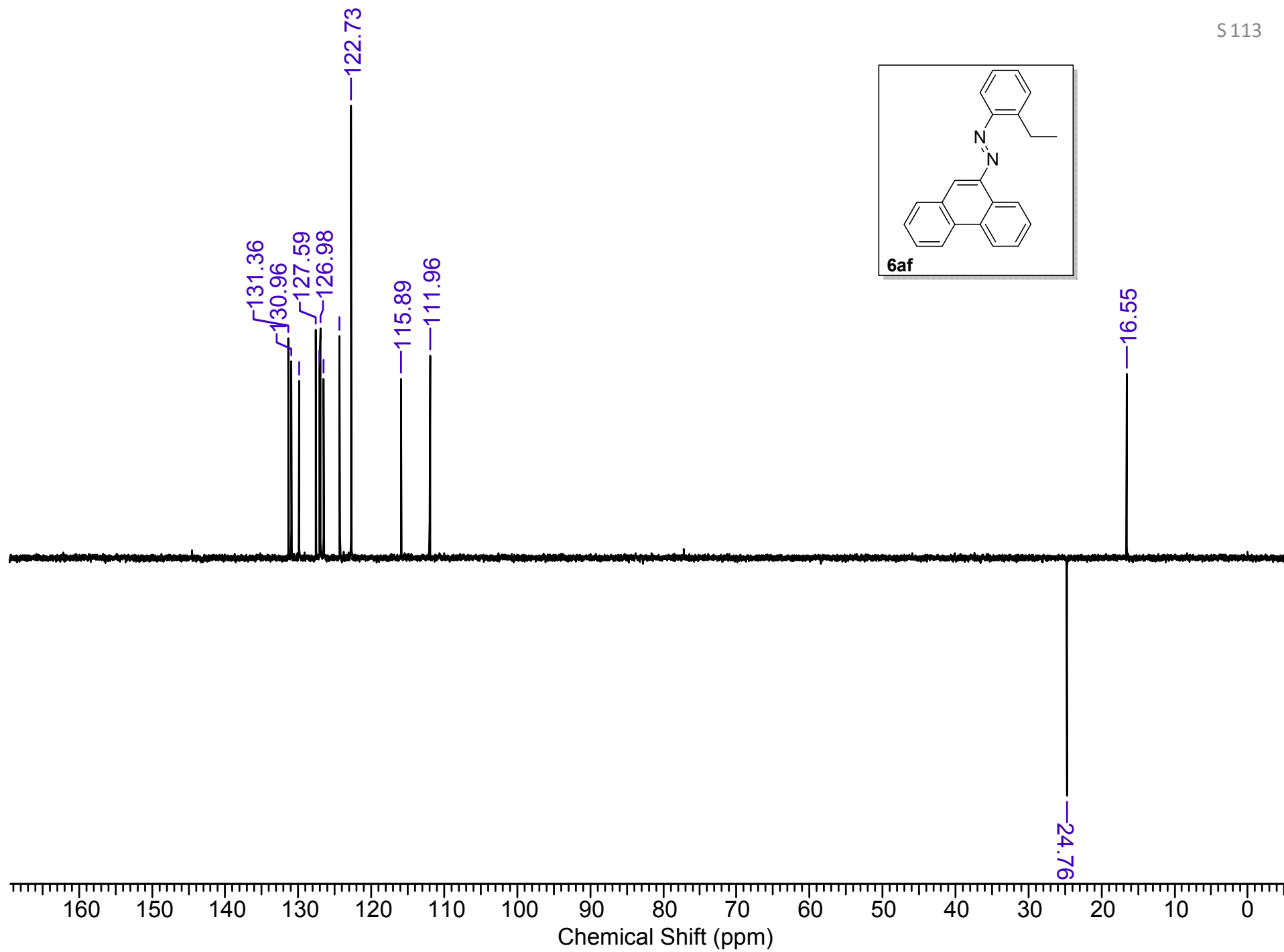
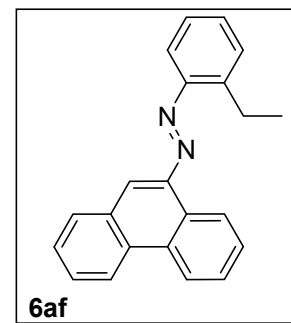


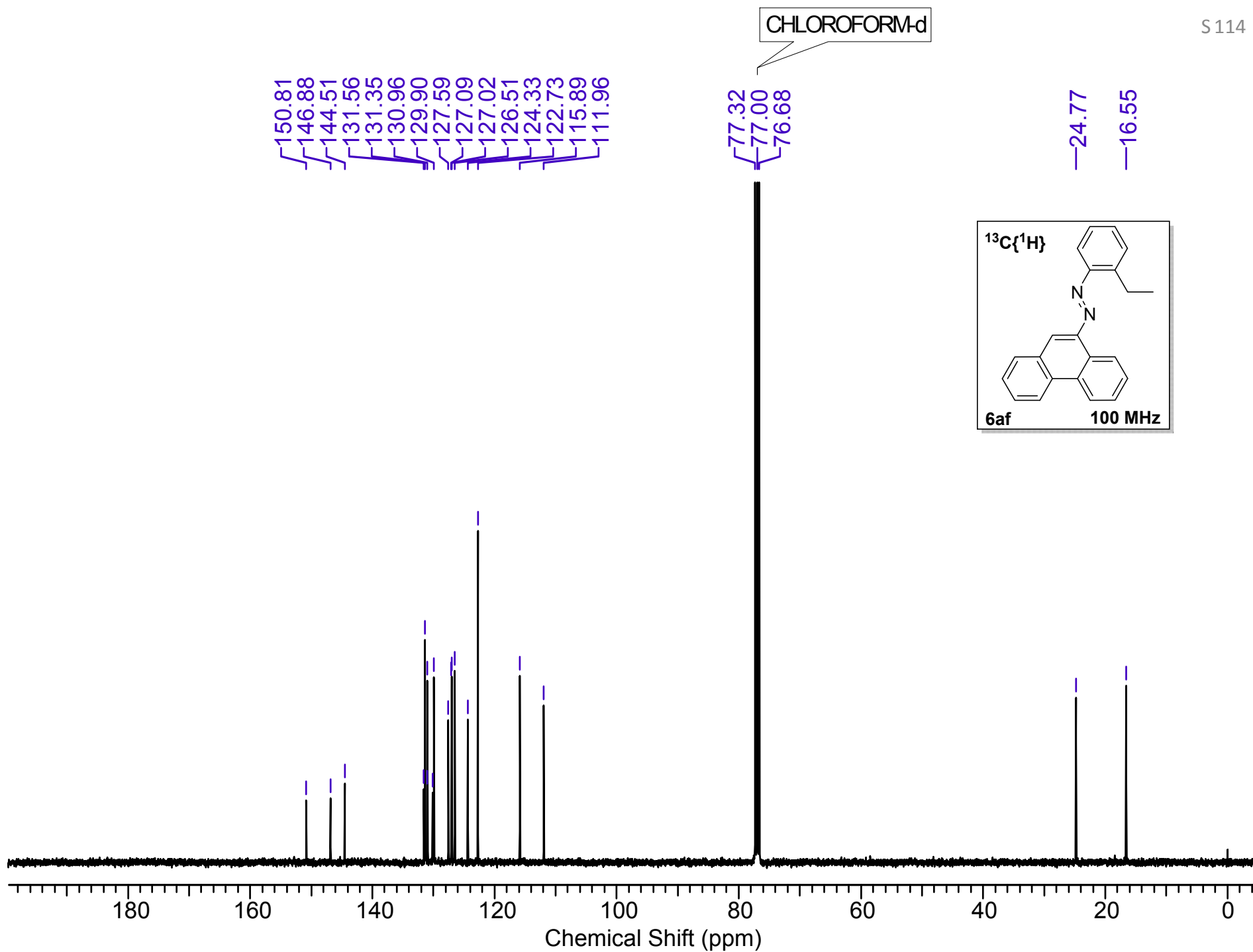




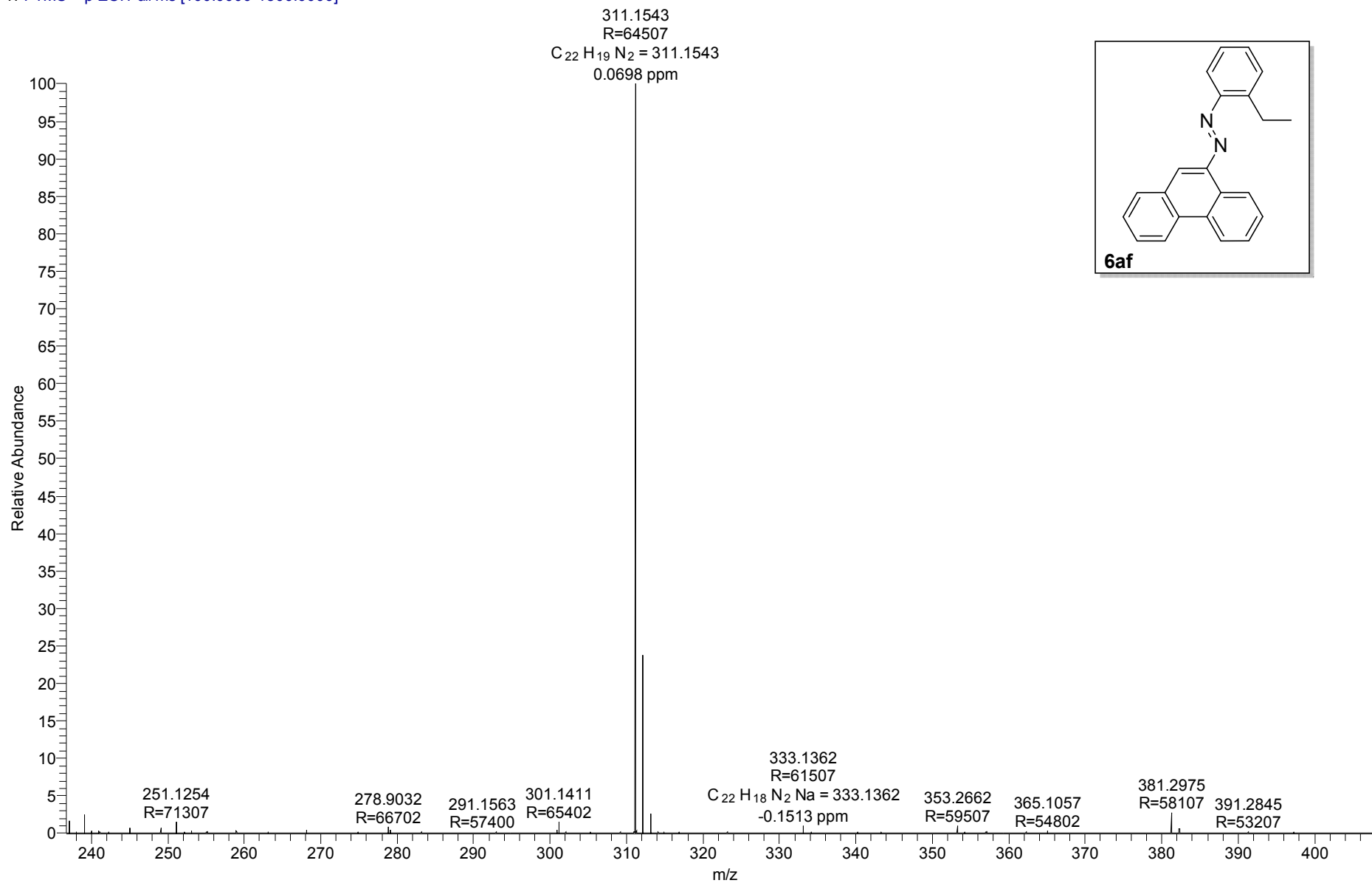


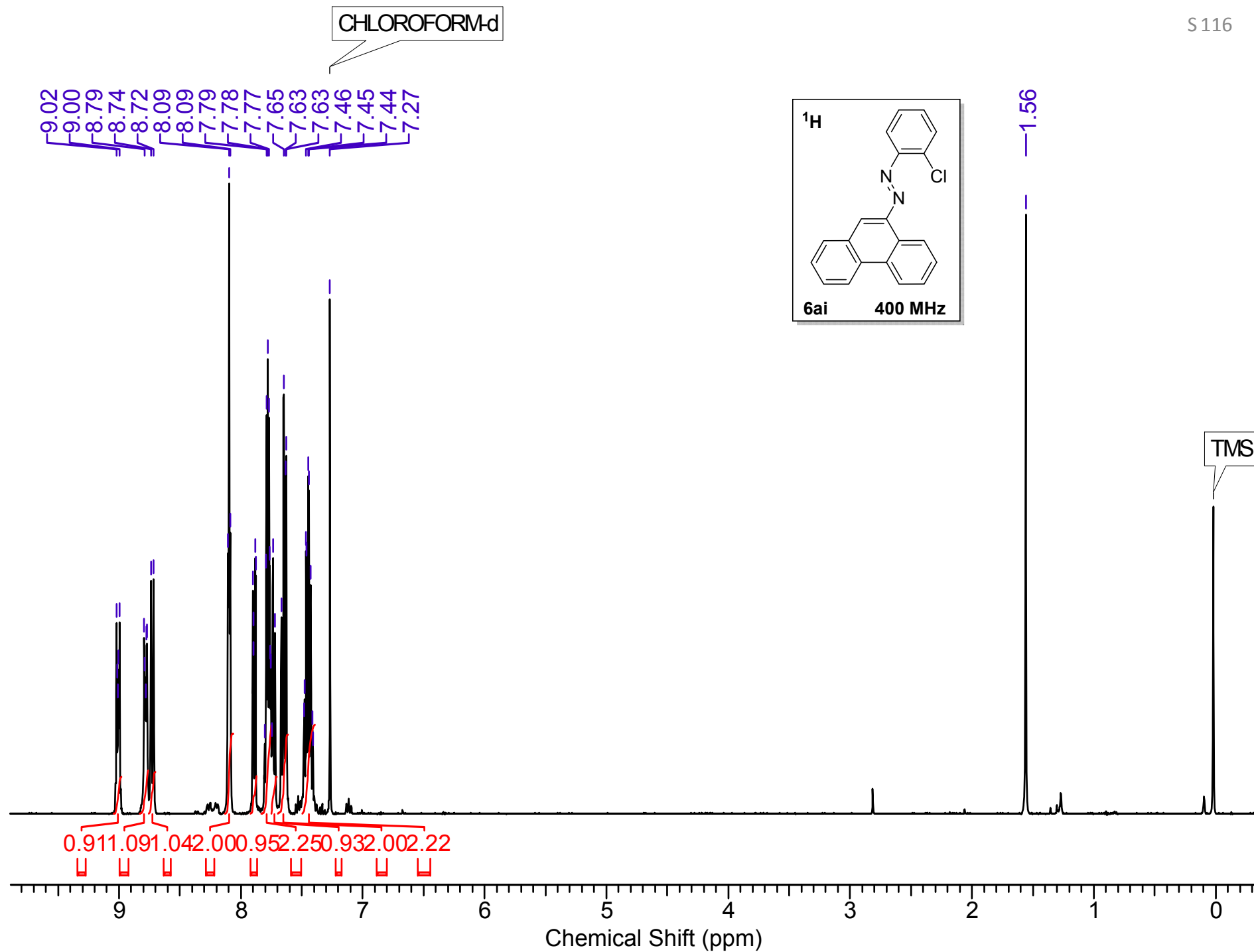


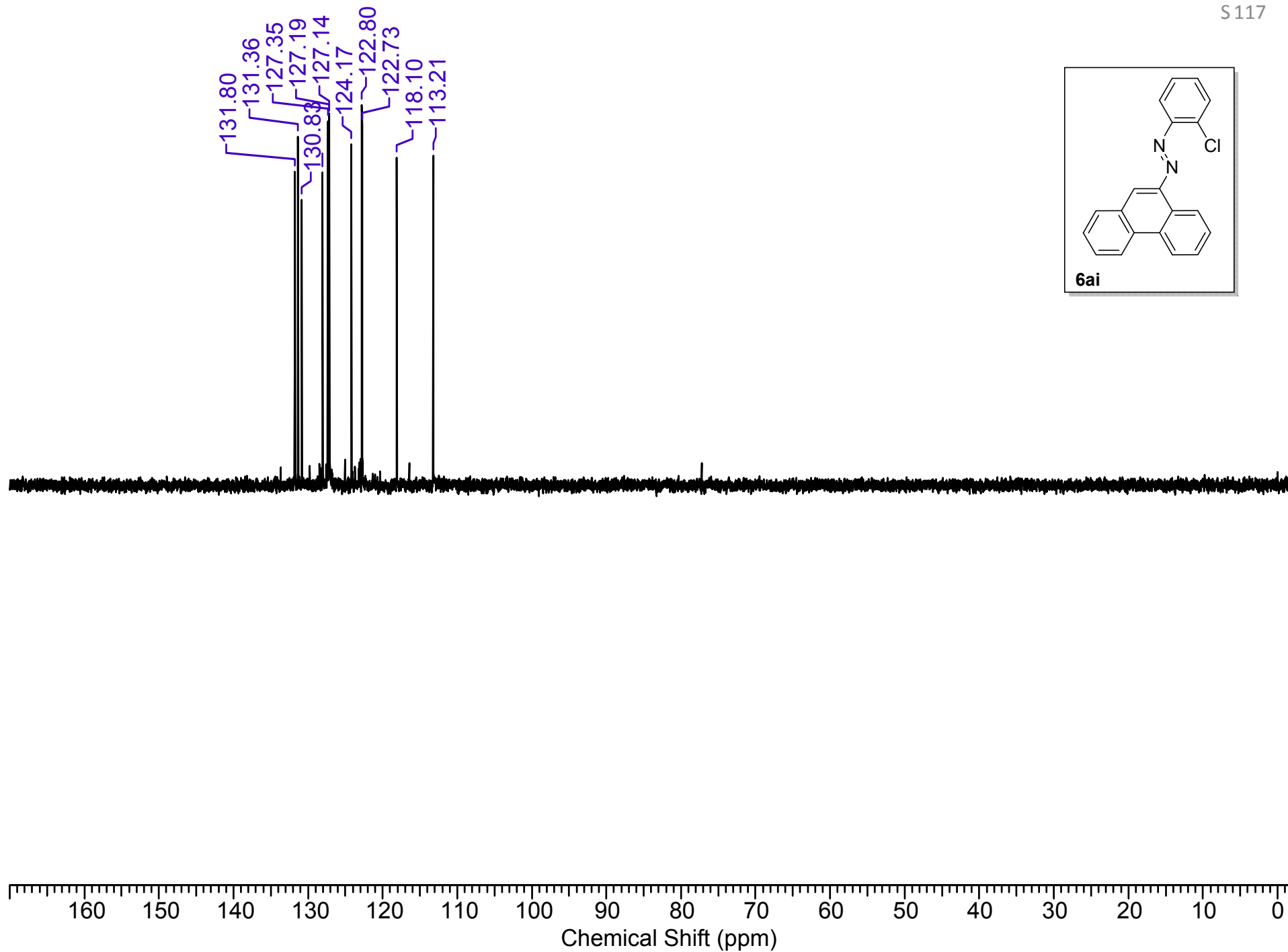


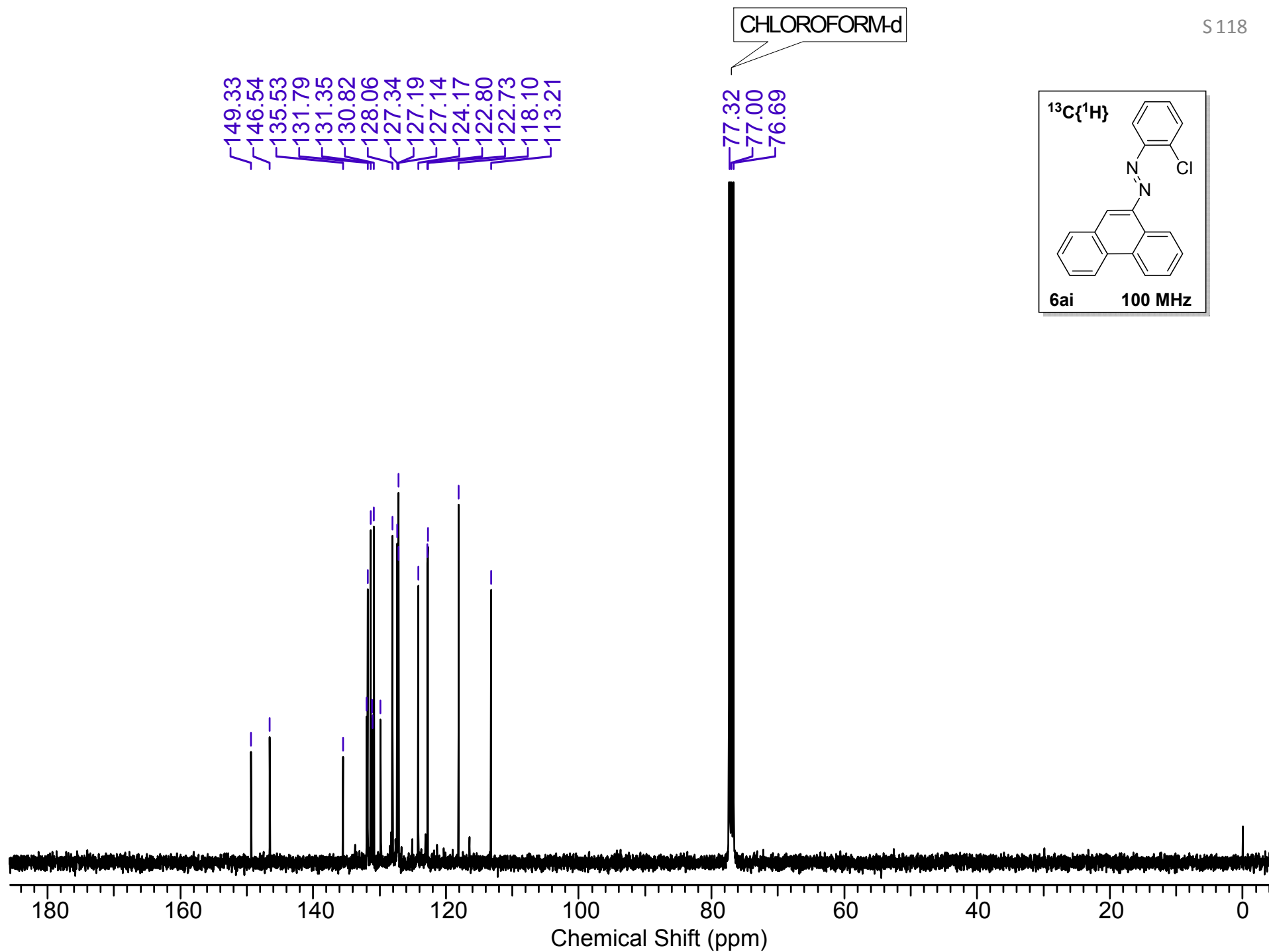


PSD-881-A #337 RT: 2.06 AV: 1 NL: 3.12E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]



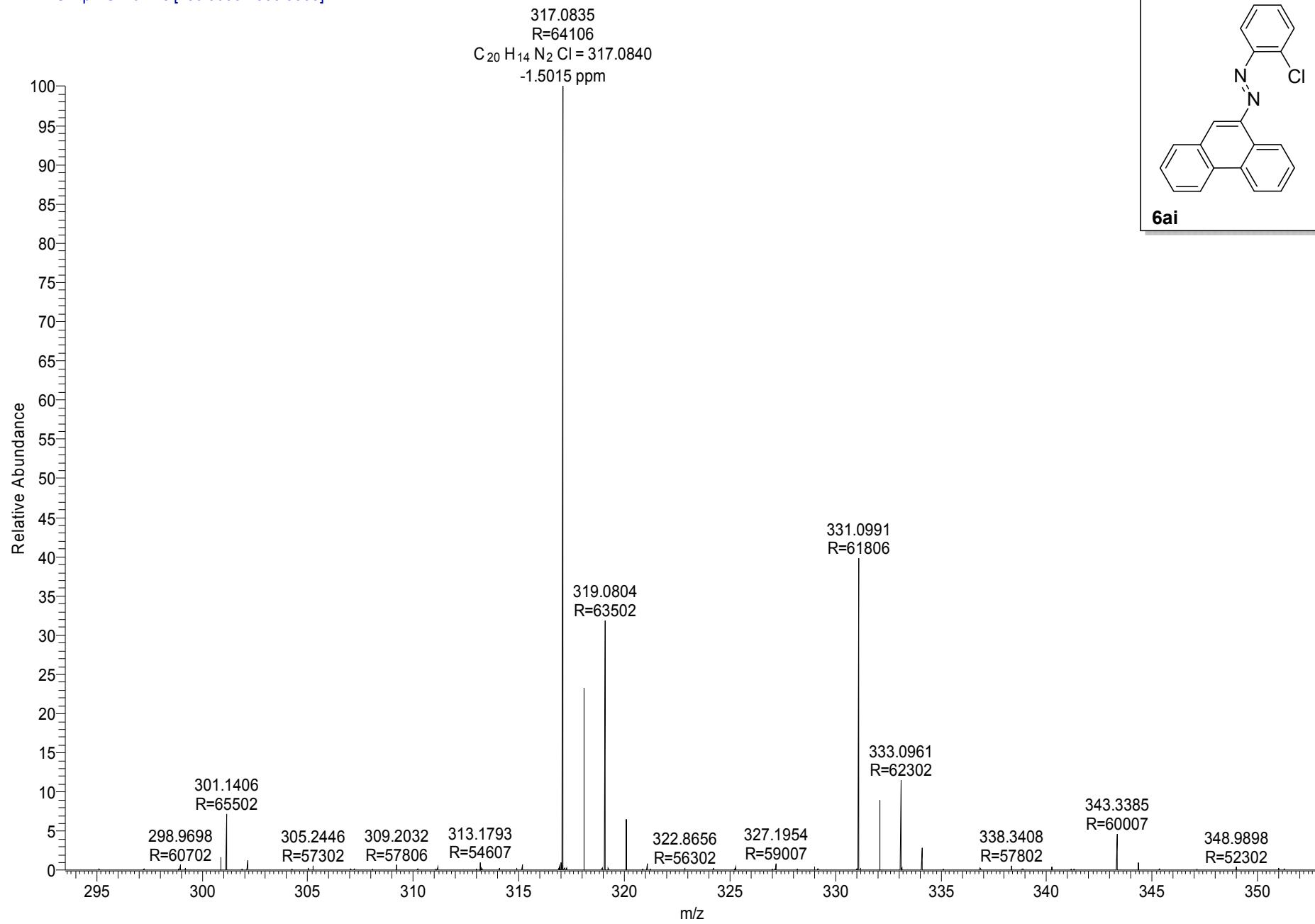


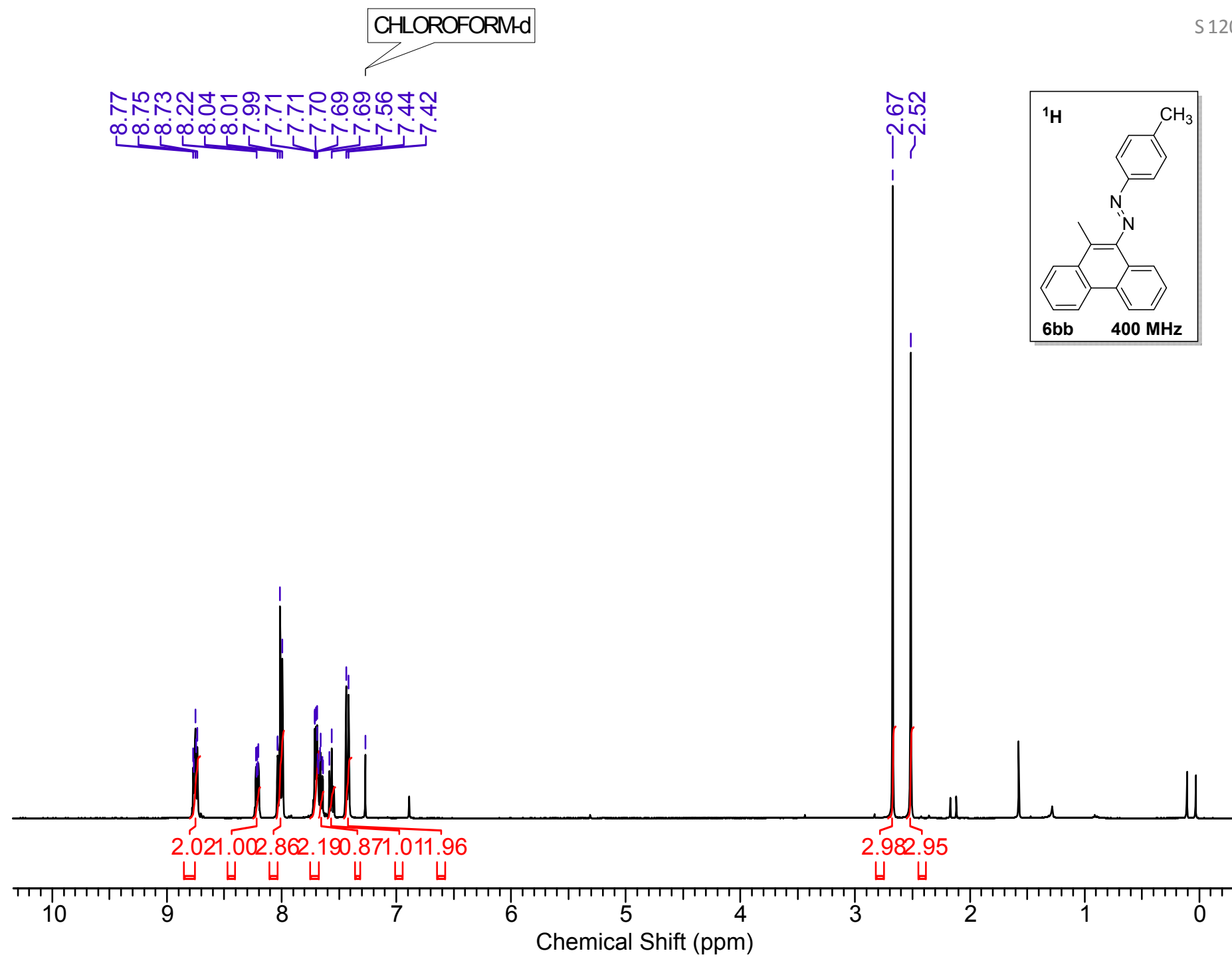


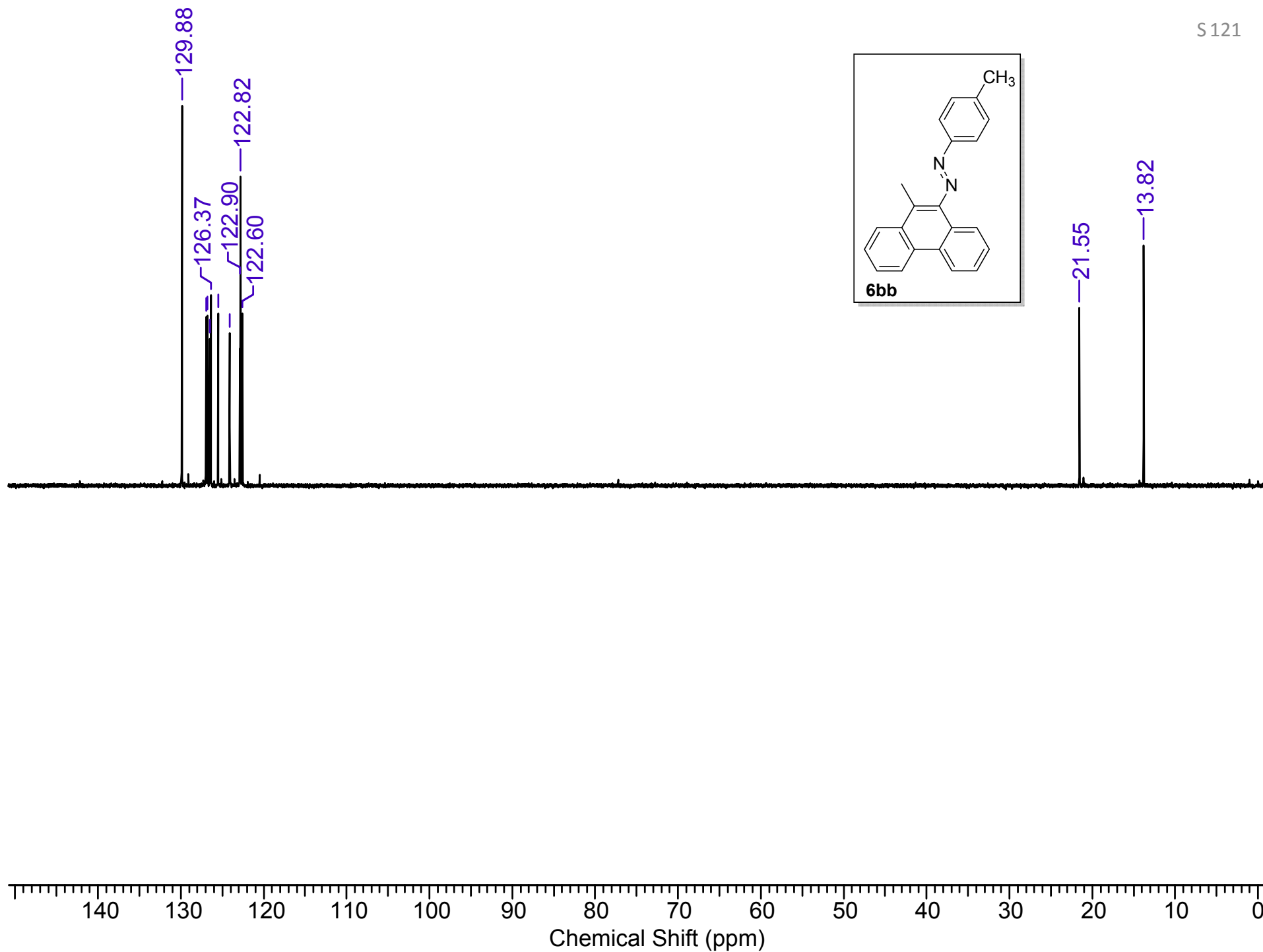
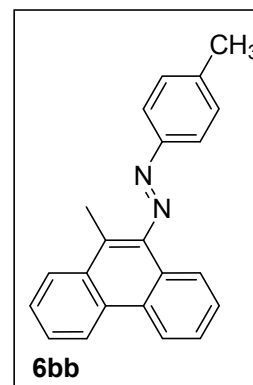


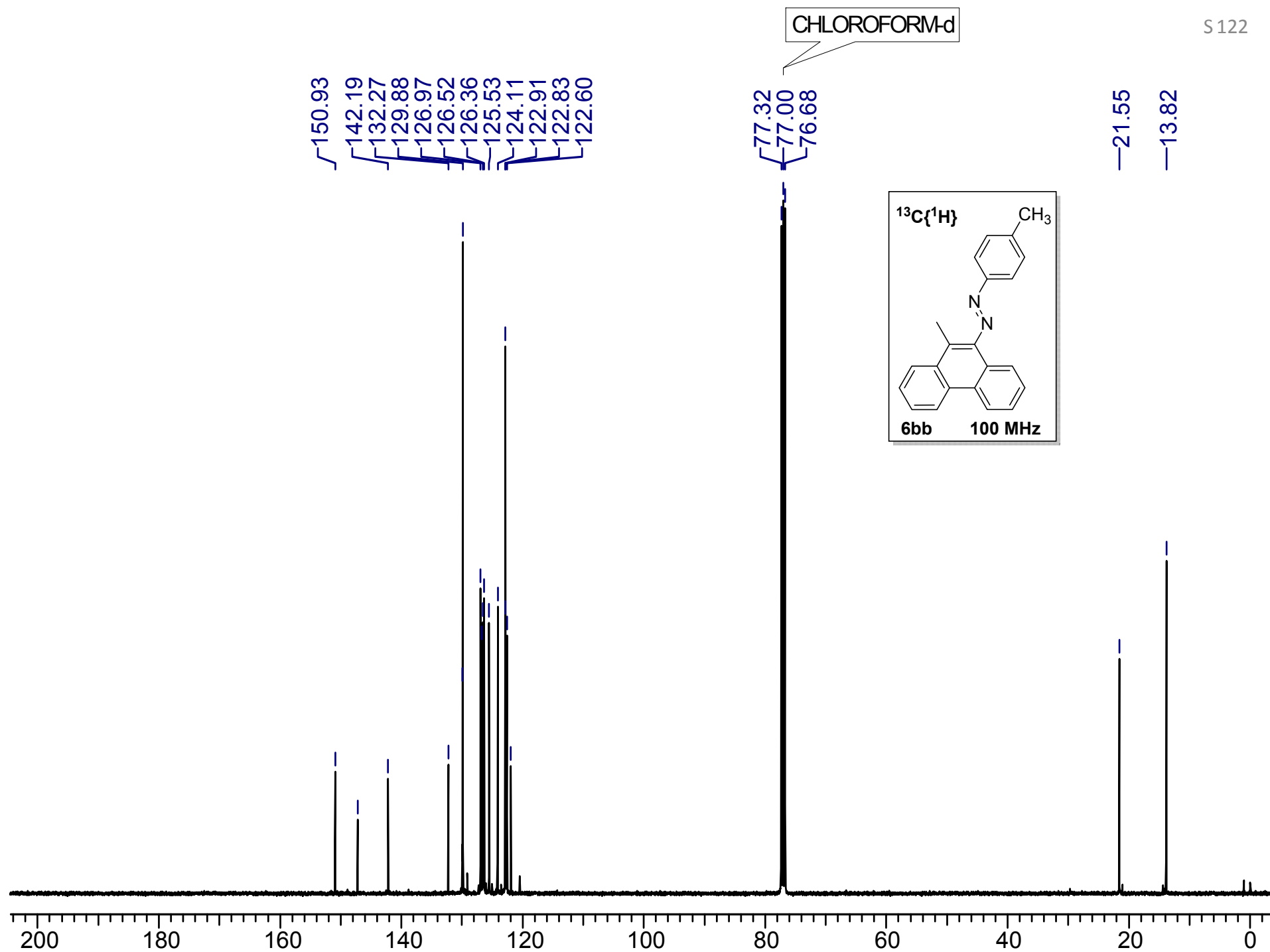
PSD-880 #599 RT: 3.65 AV: 1 NL: 1.24E7
T: FTMS + p ESI Full ms [100.0000-1500.0000]

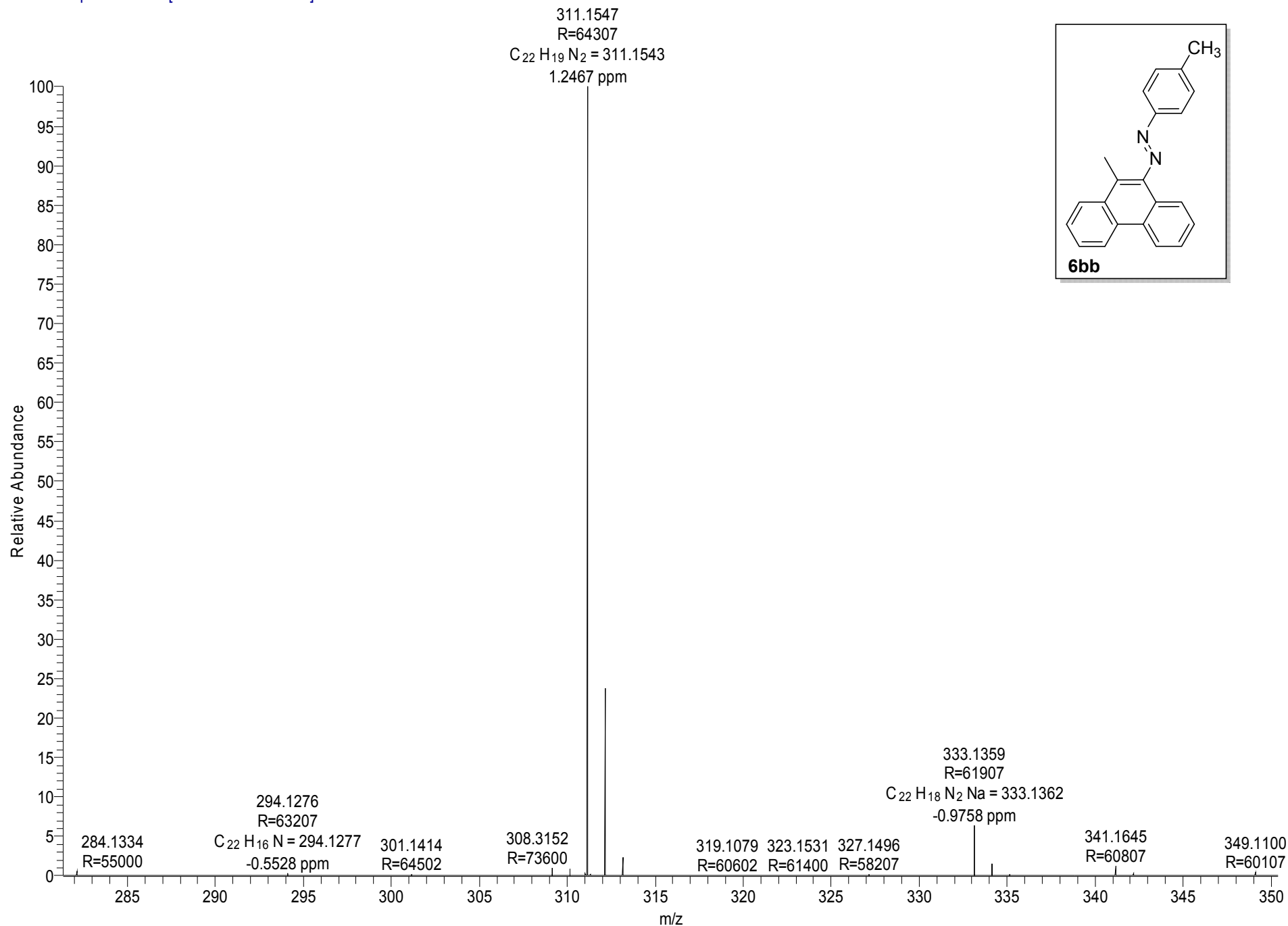
S 119

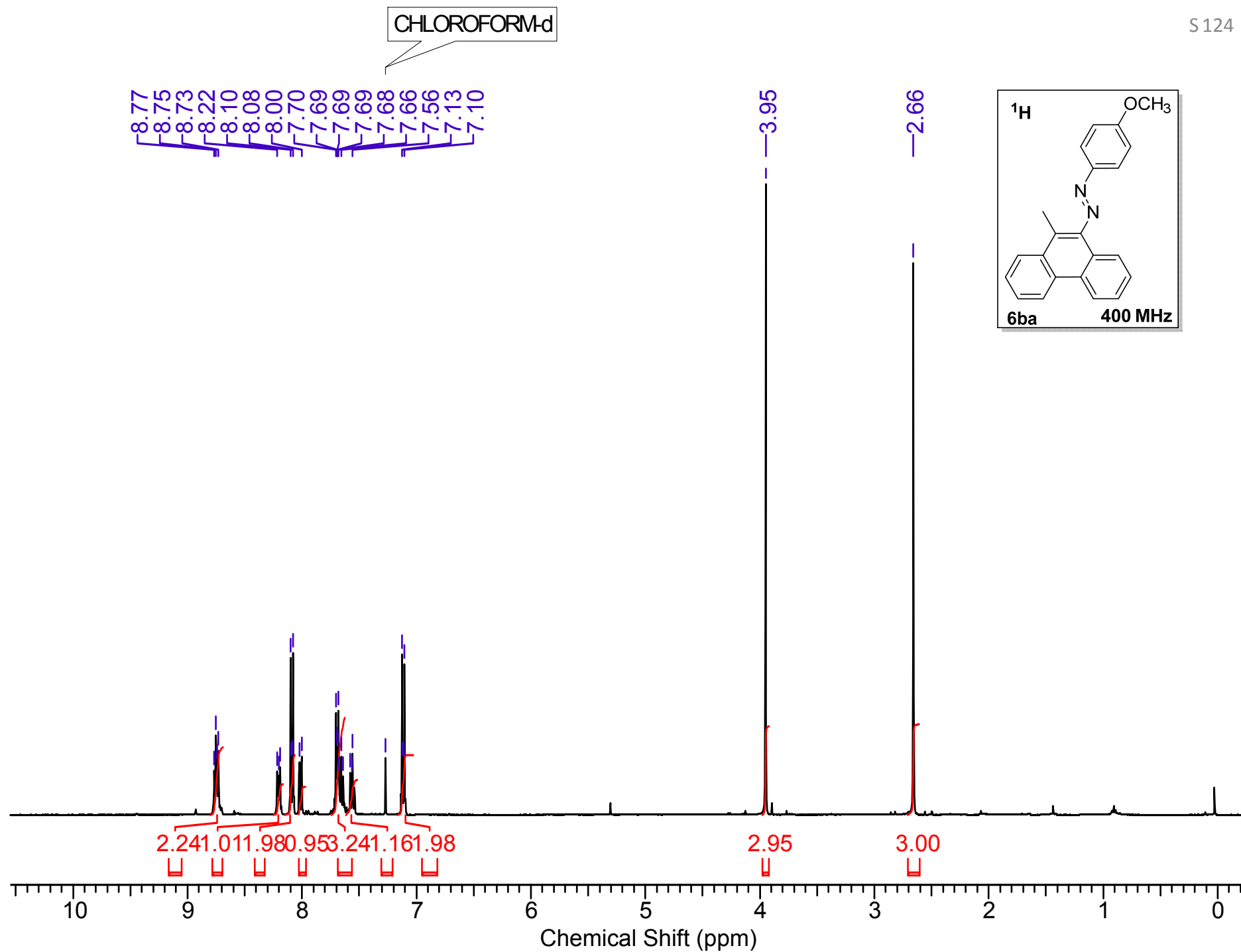


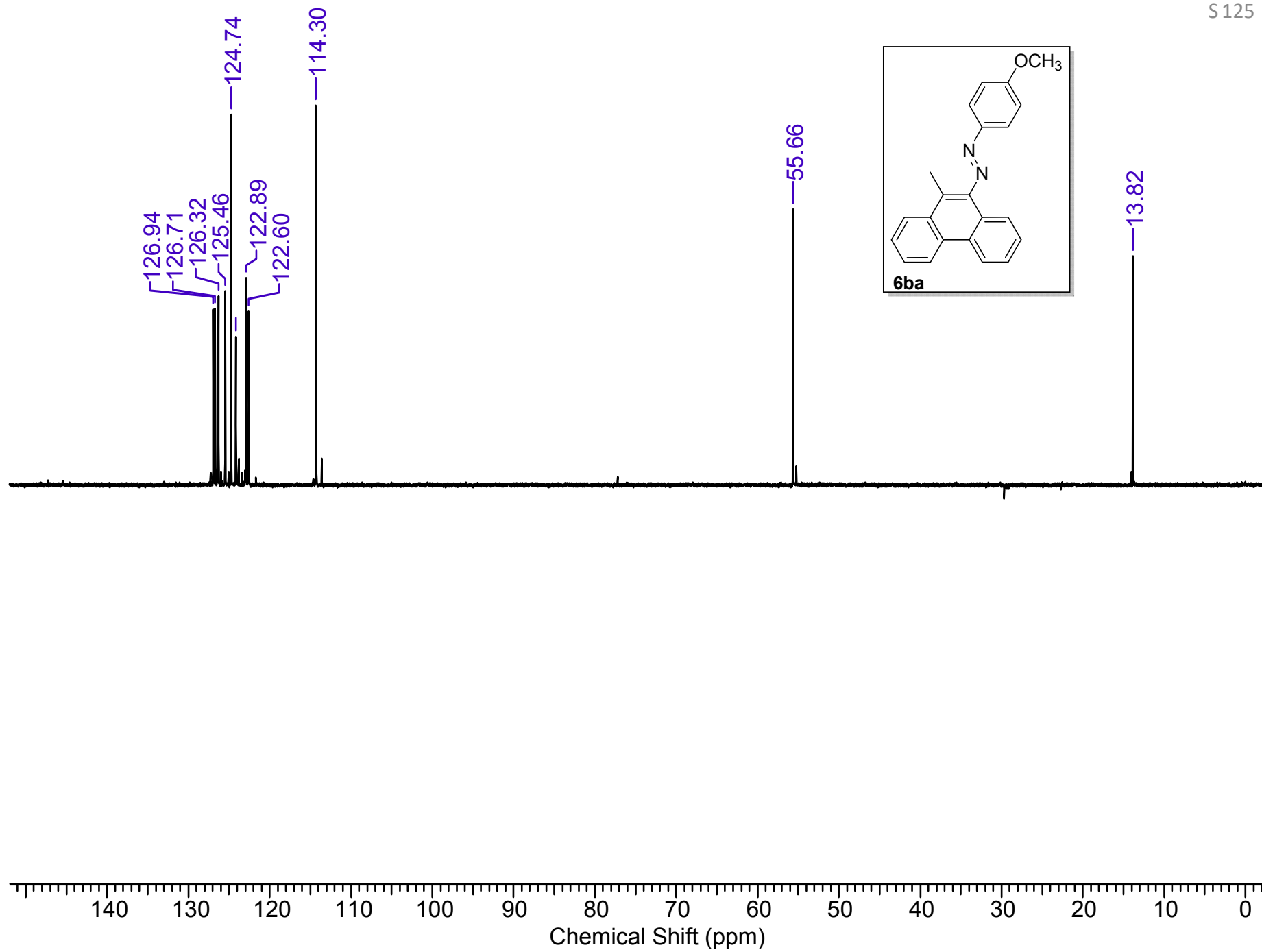


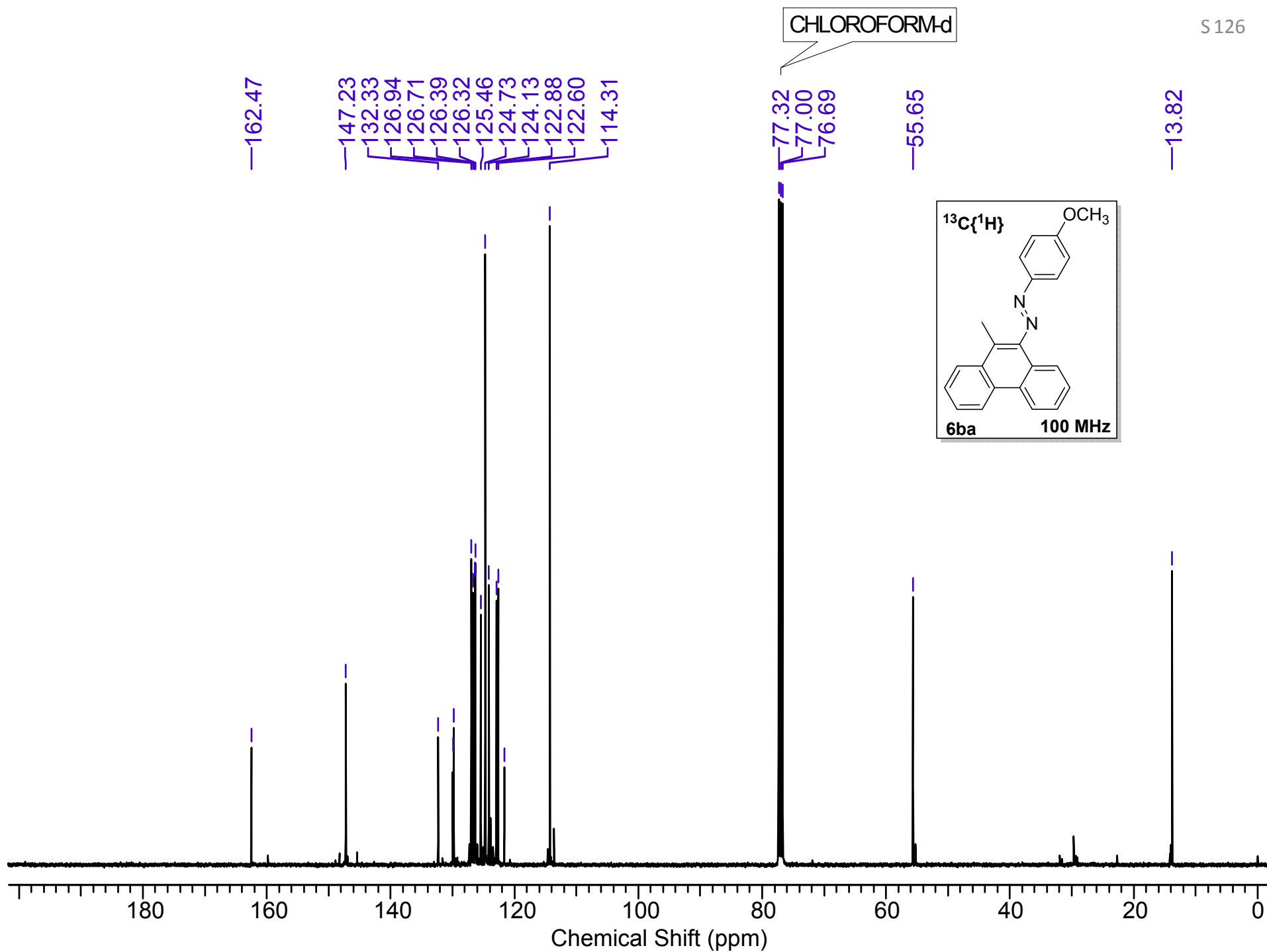


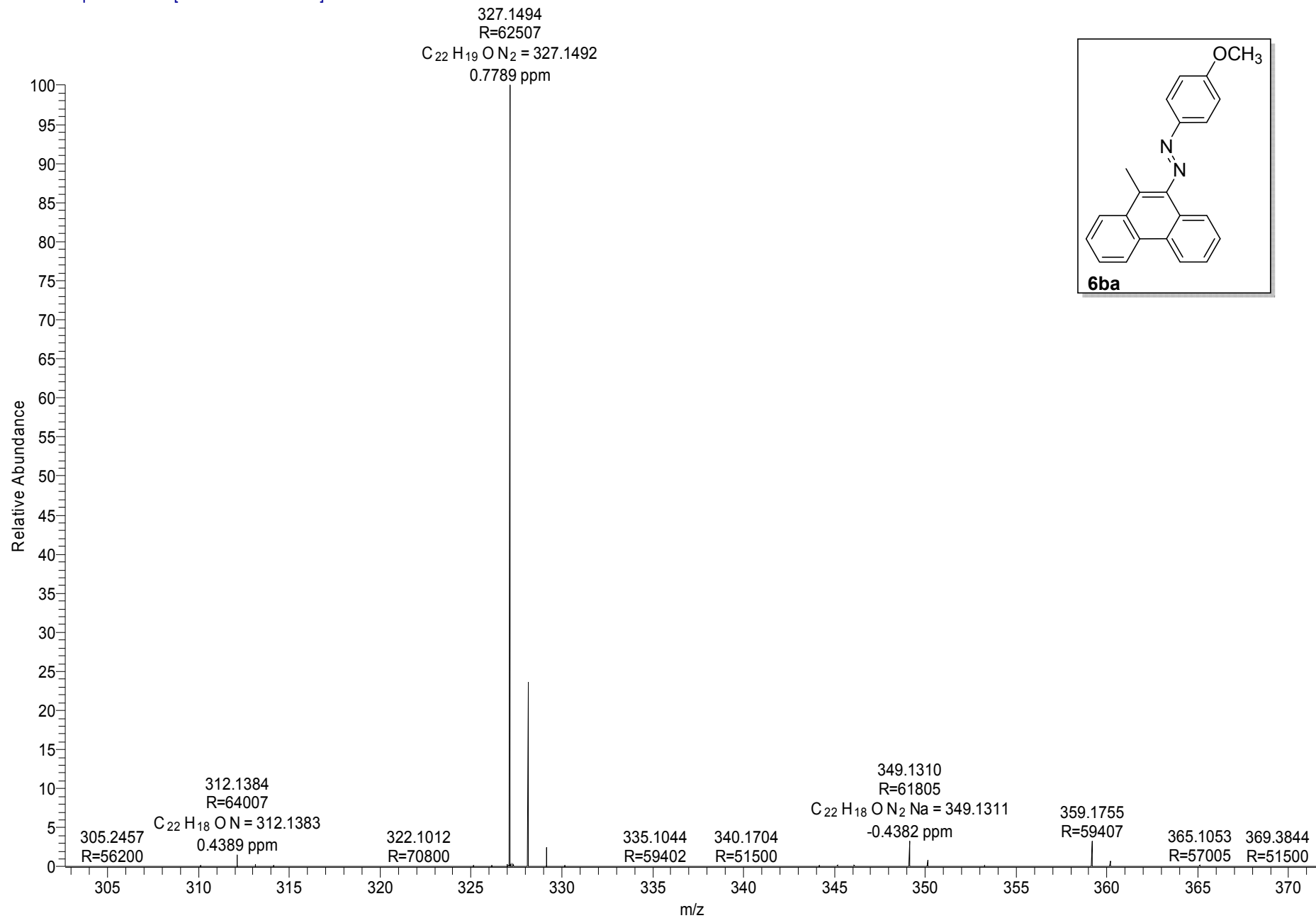


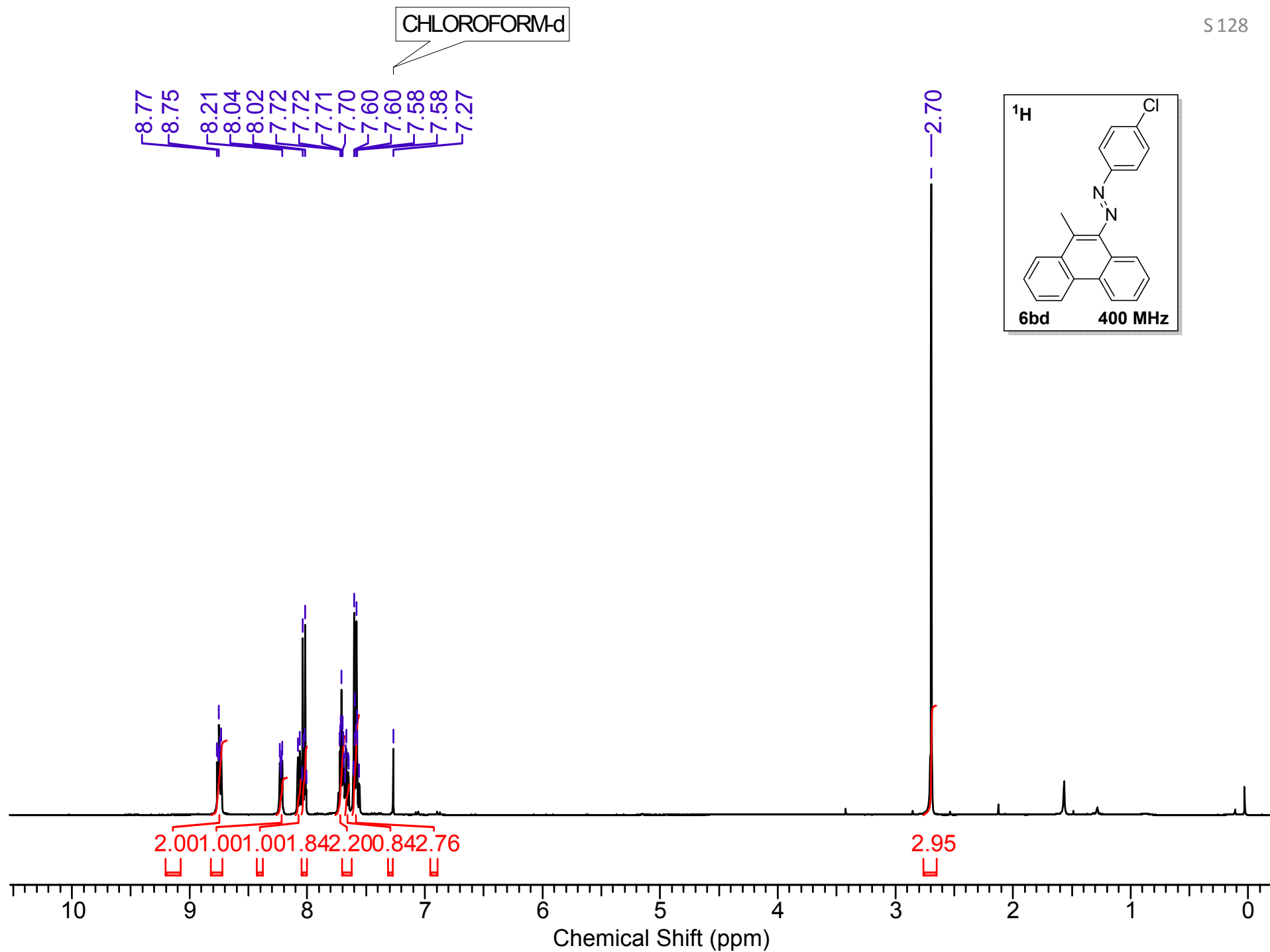


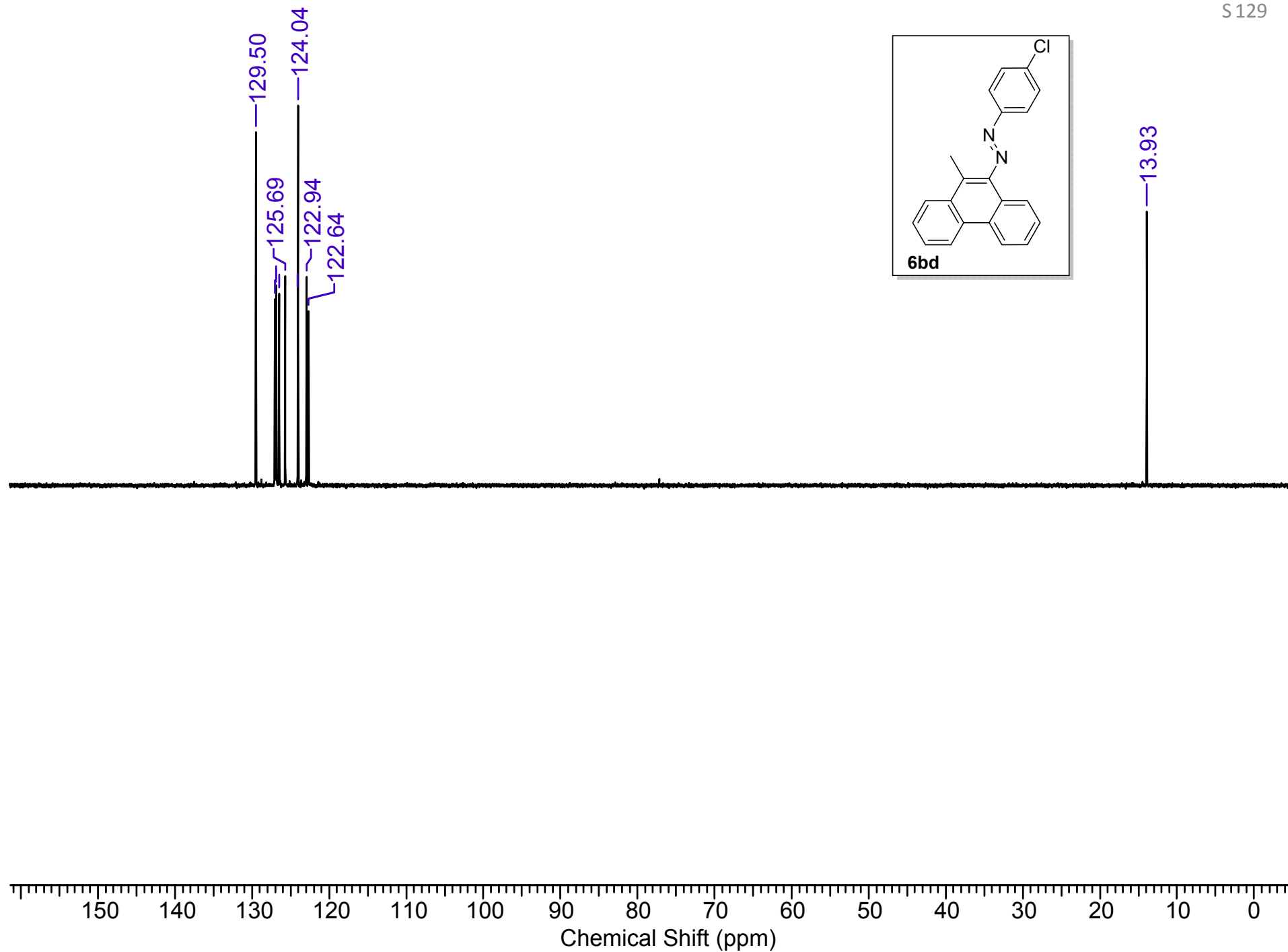
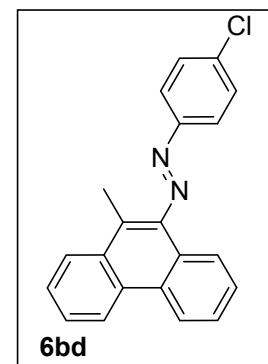


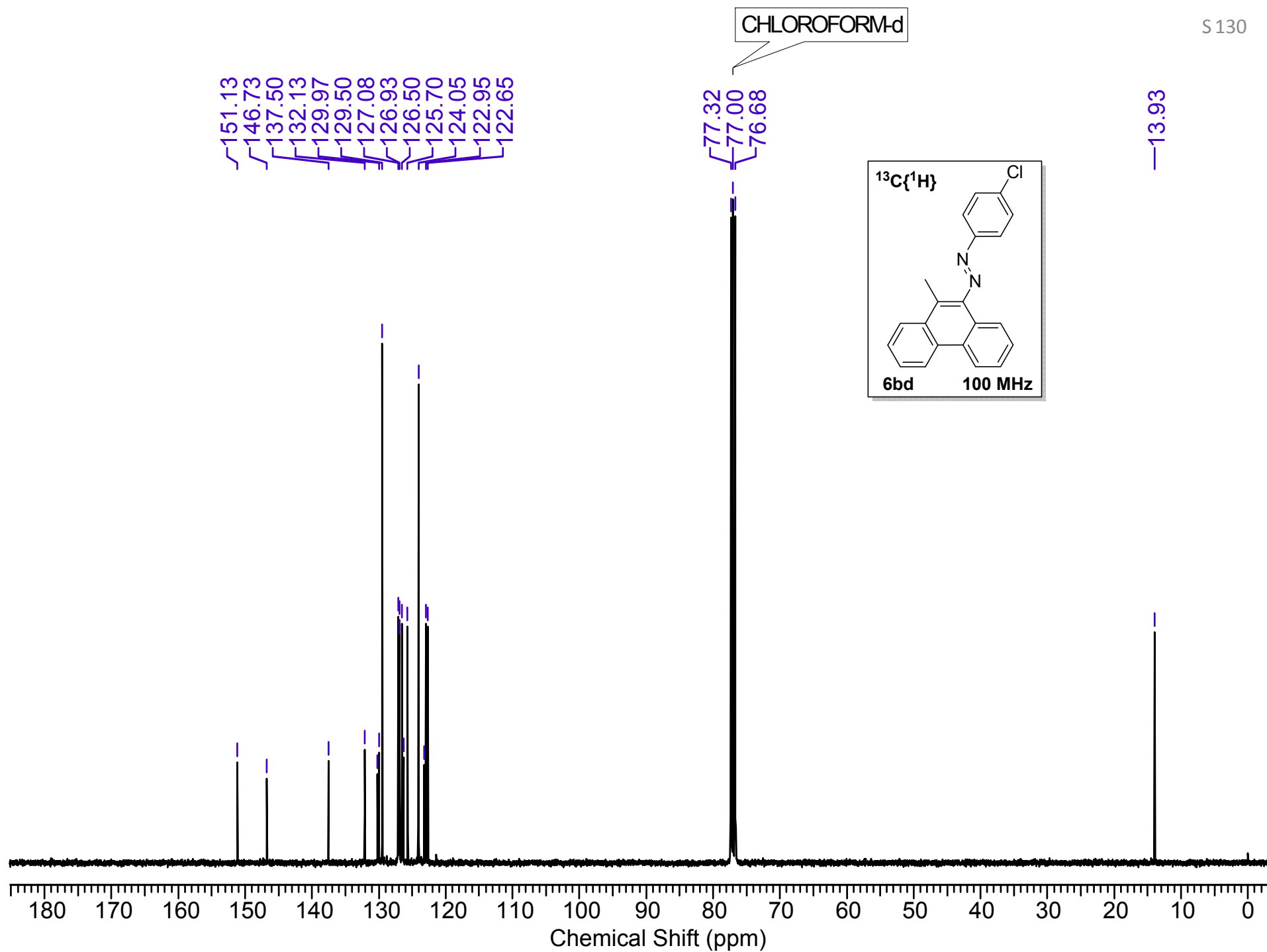




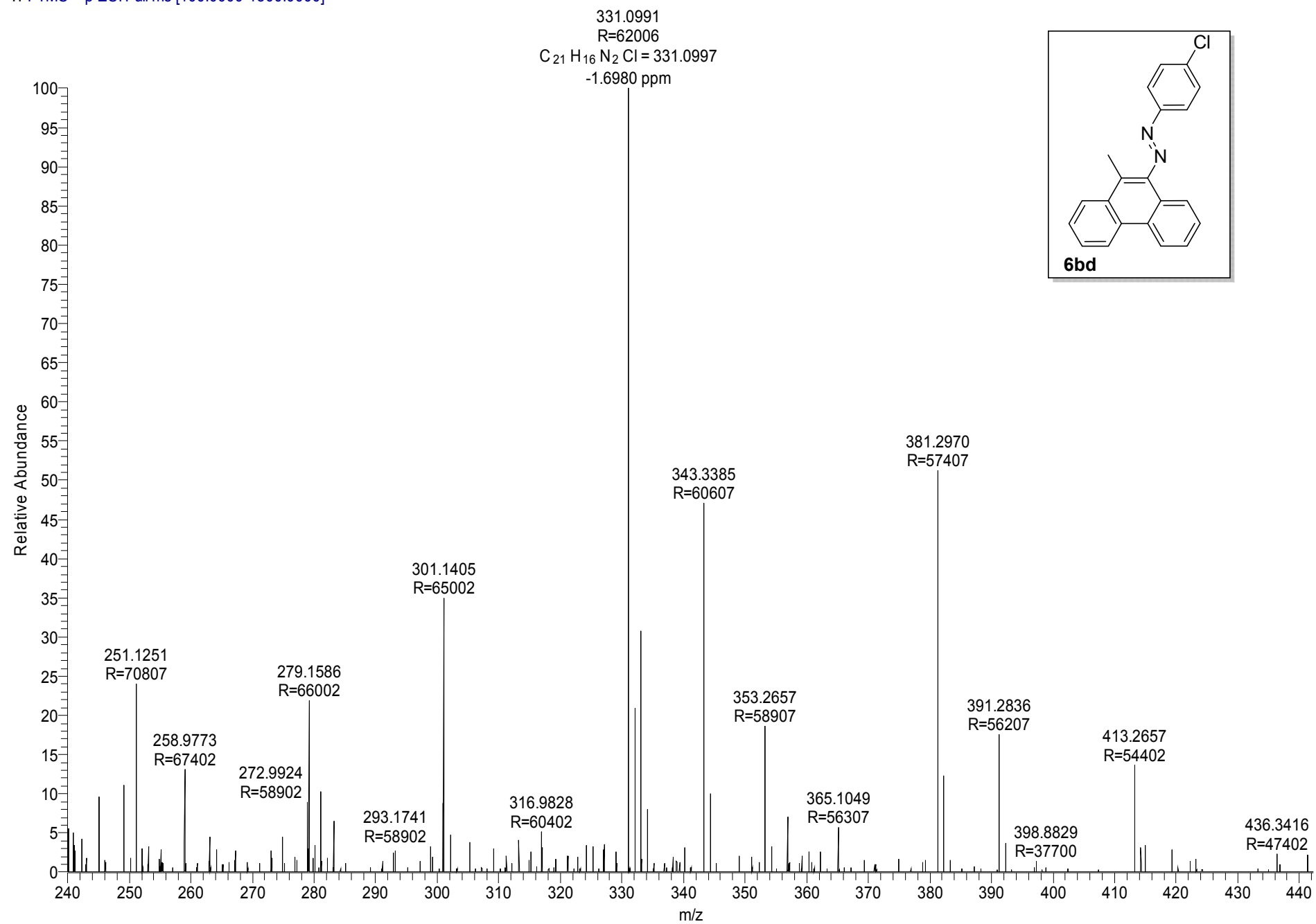


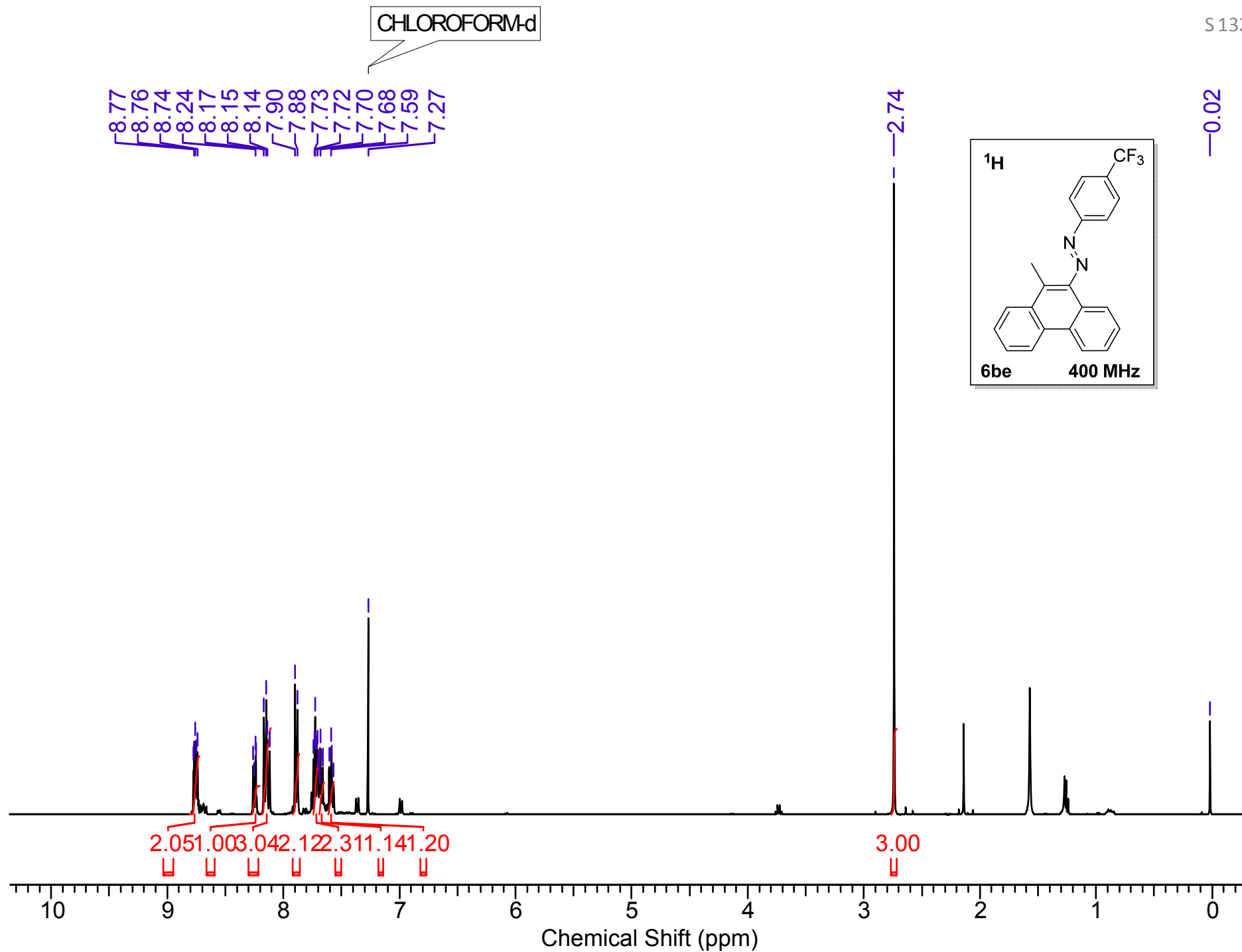


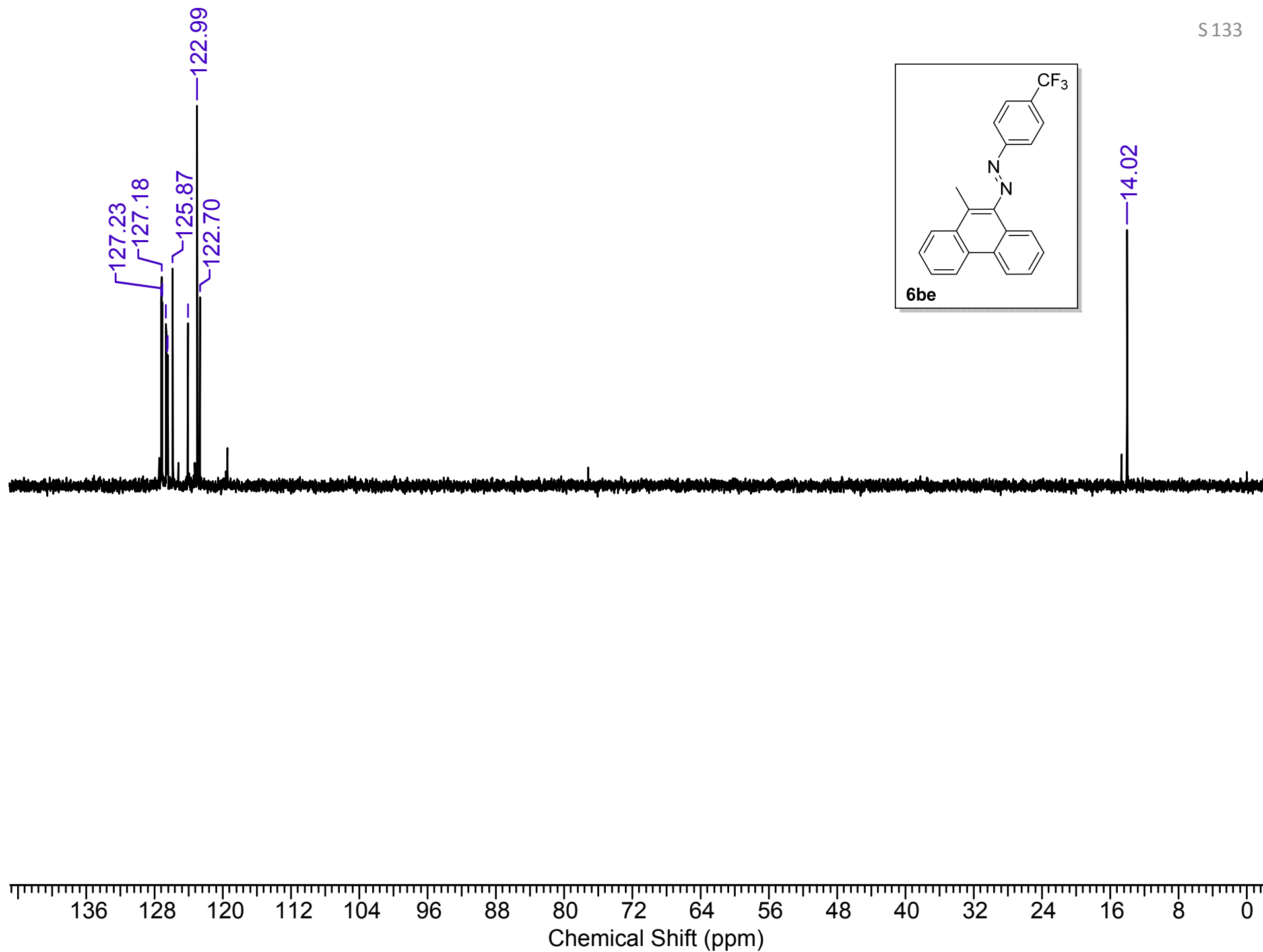
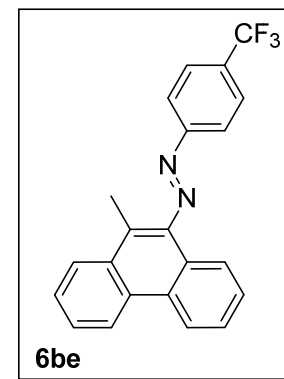


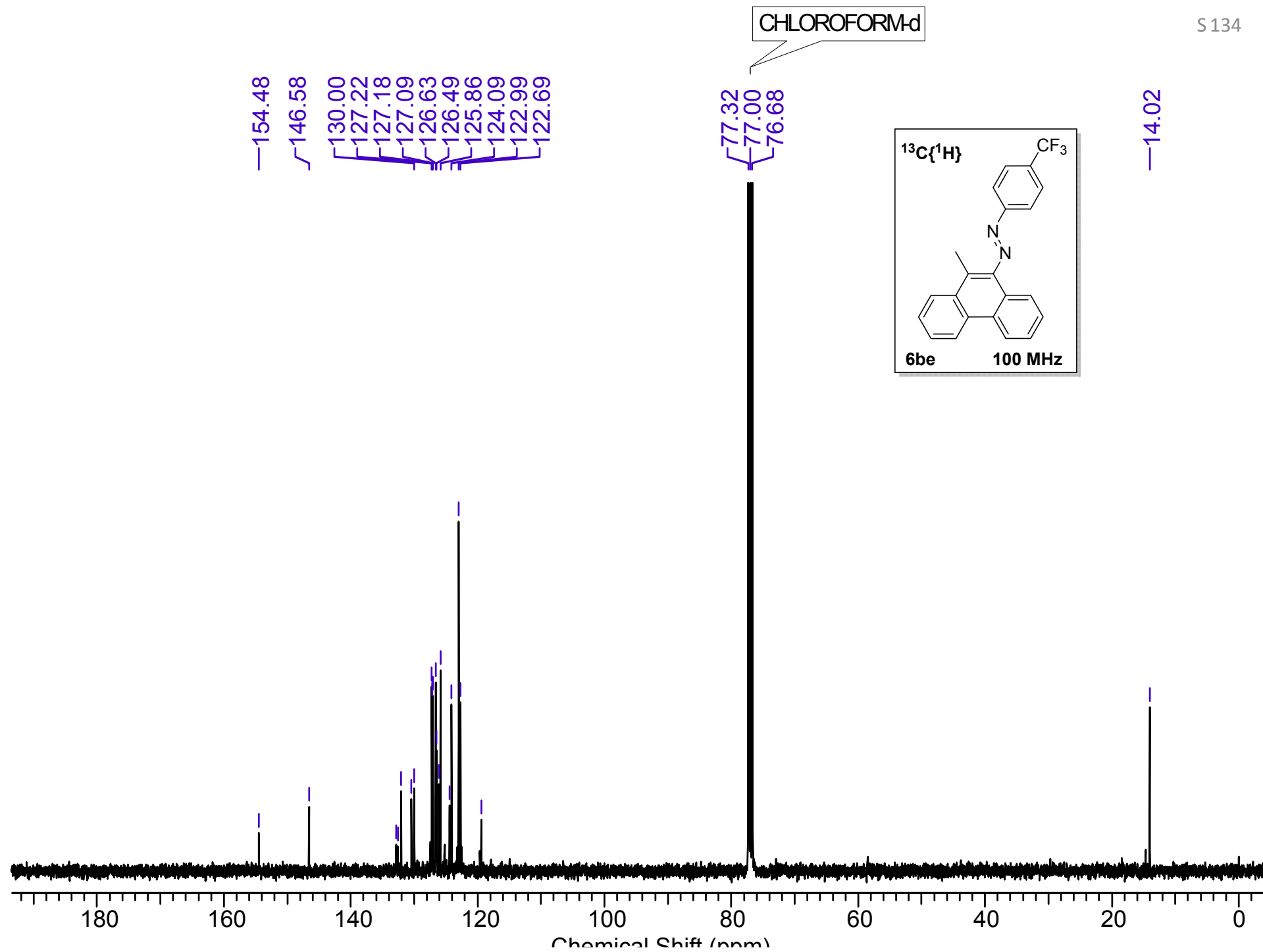


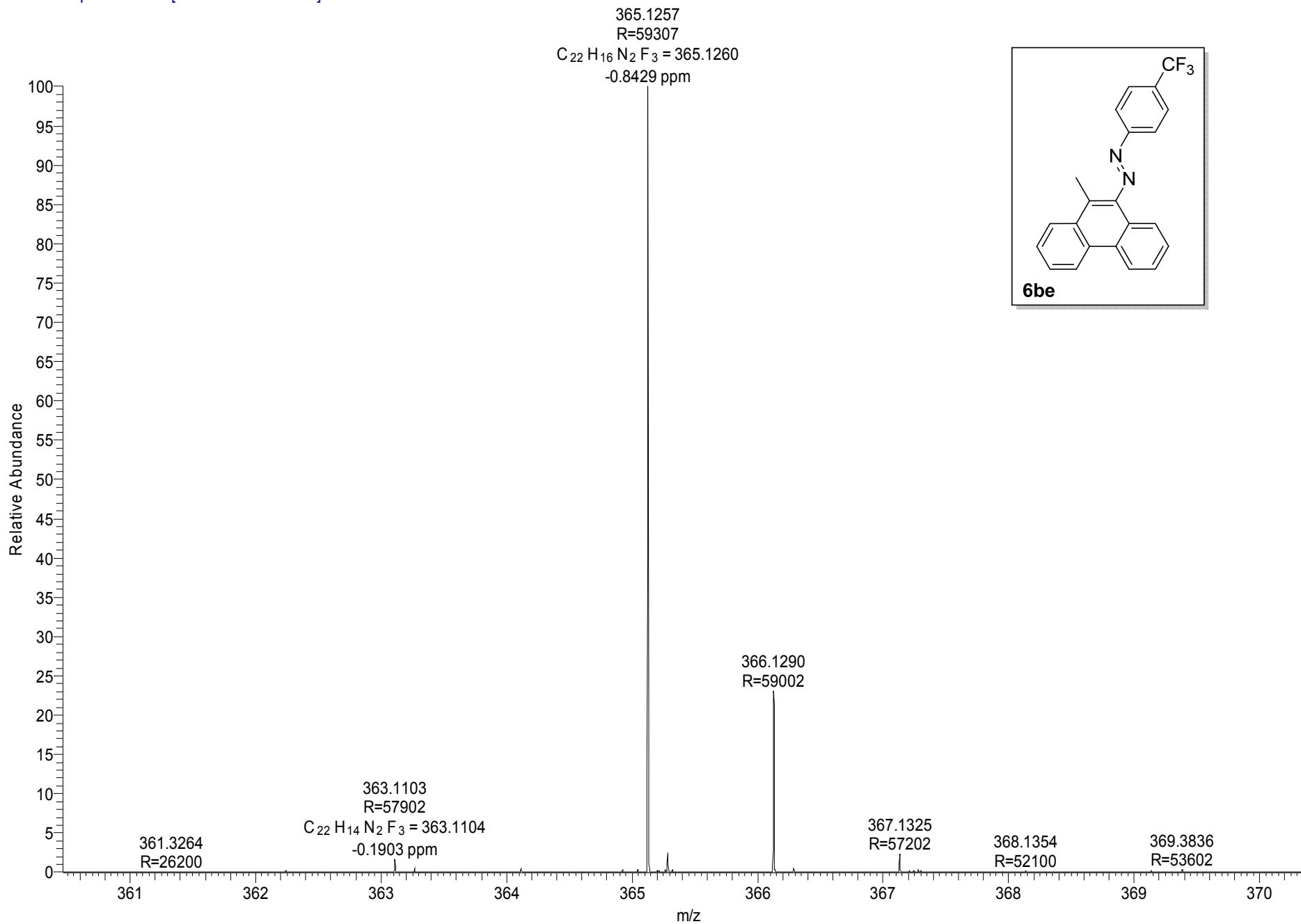
PSD-879 #346 RT: 2.12 AV: 1 NL: 1.07E6
T: FTMS + p ESI Full ms [100.0000-1500.0000]

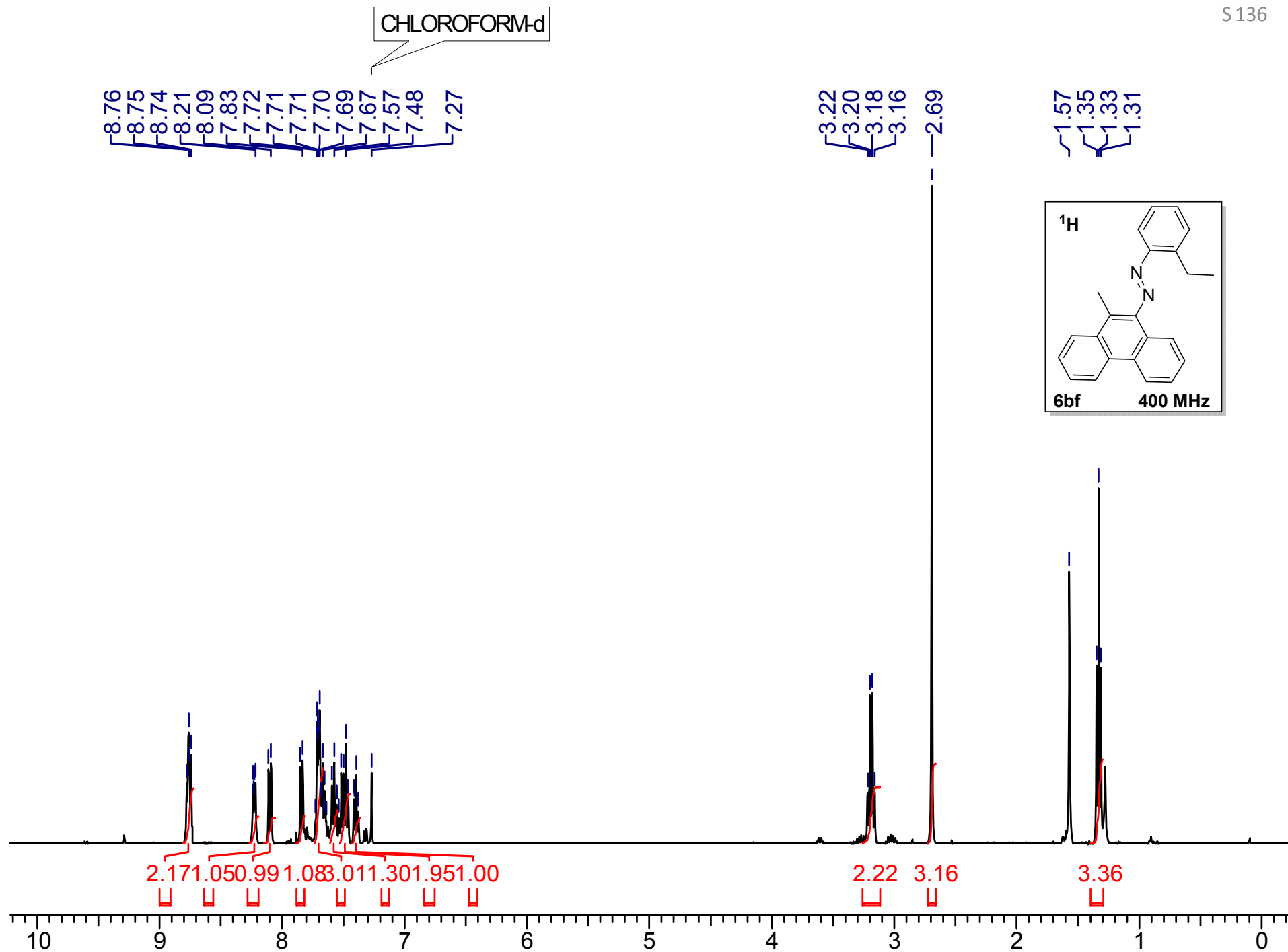


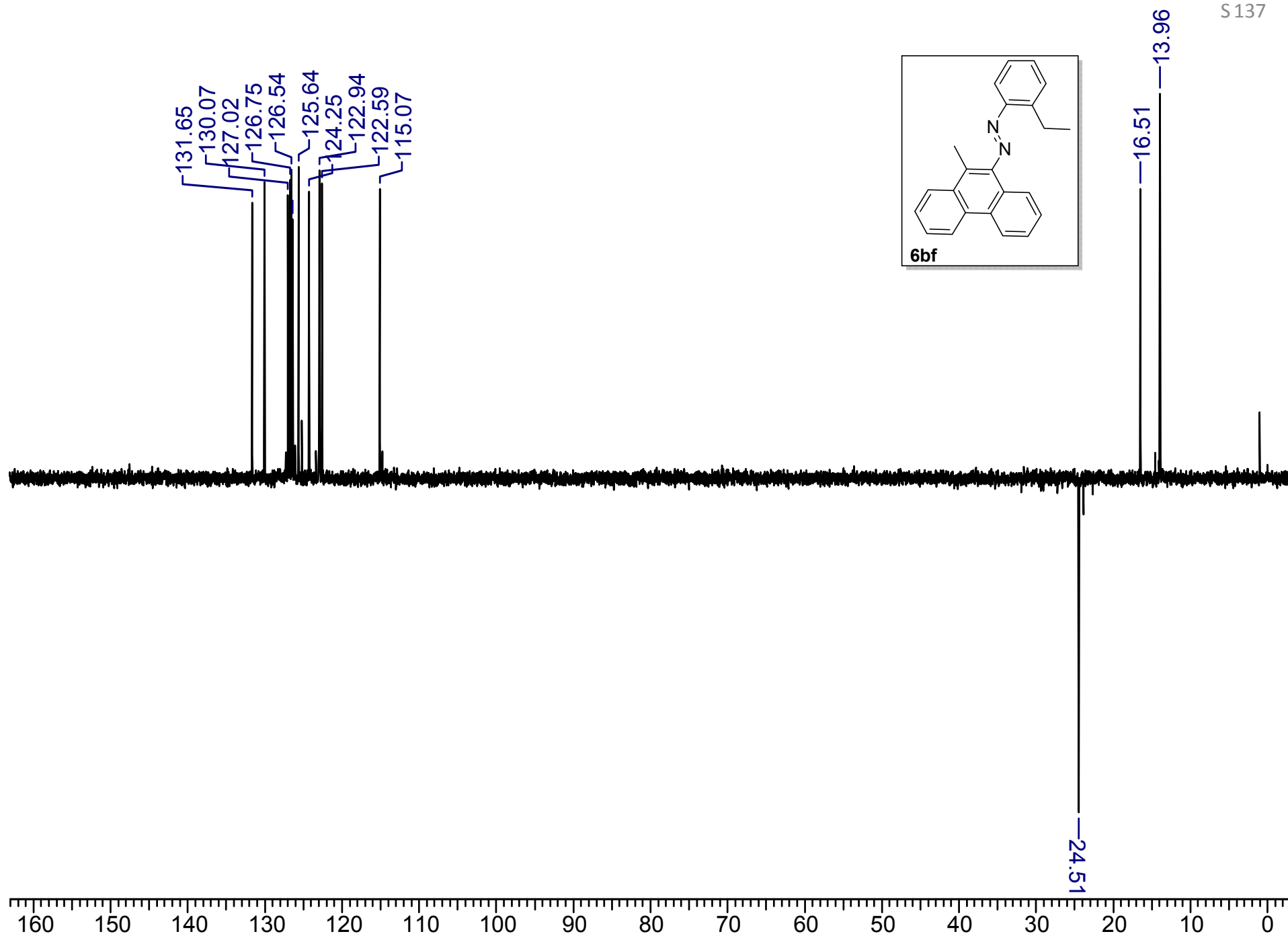
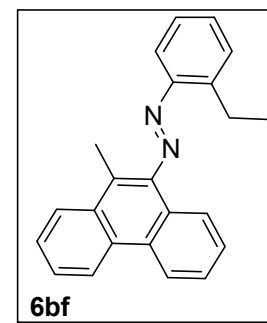












CHLOROFORM-d

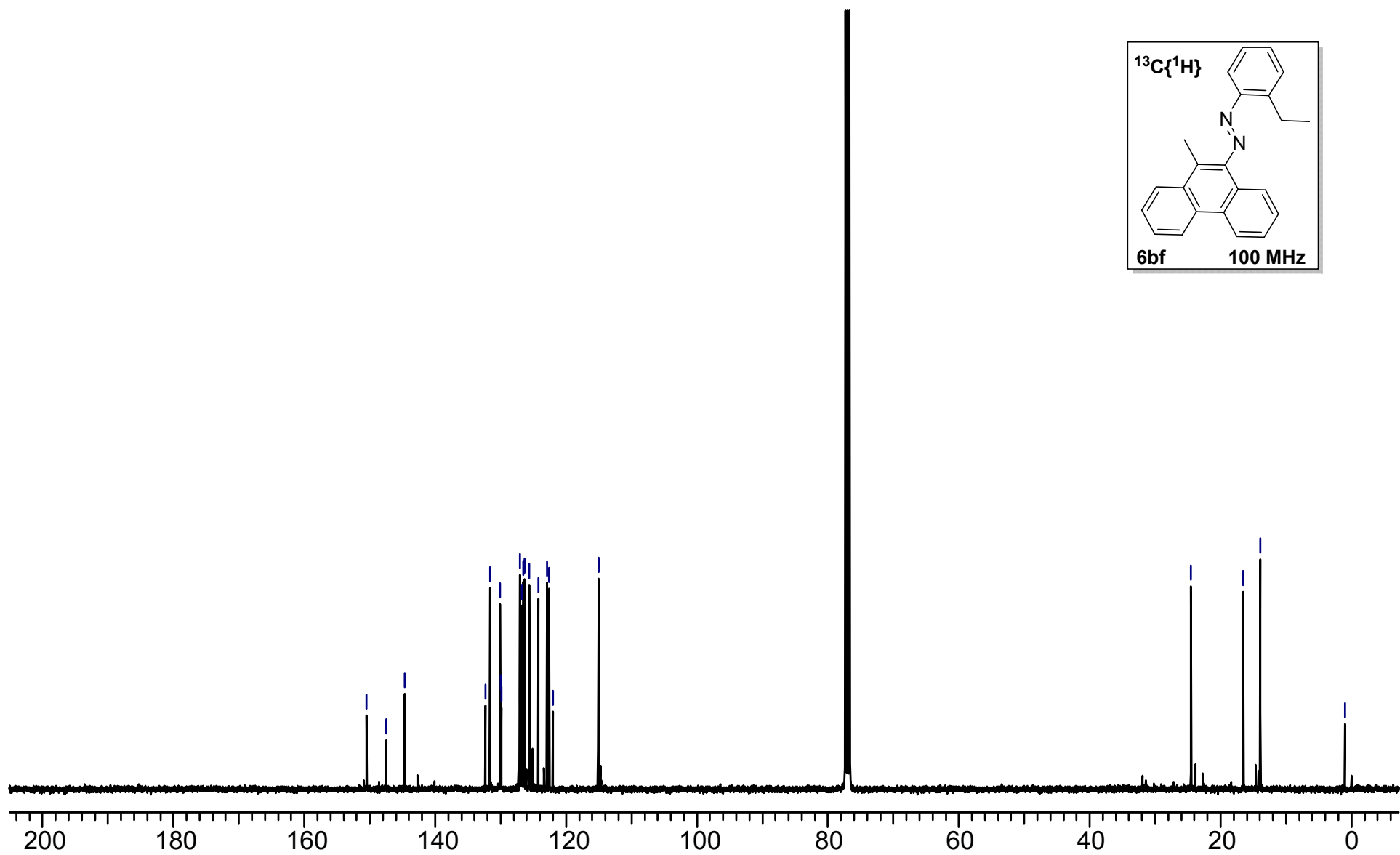
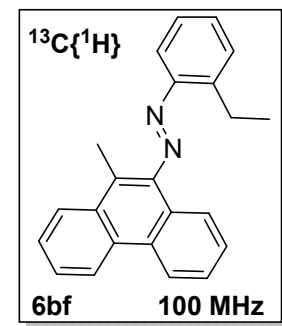
S 138

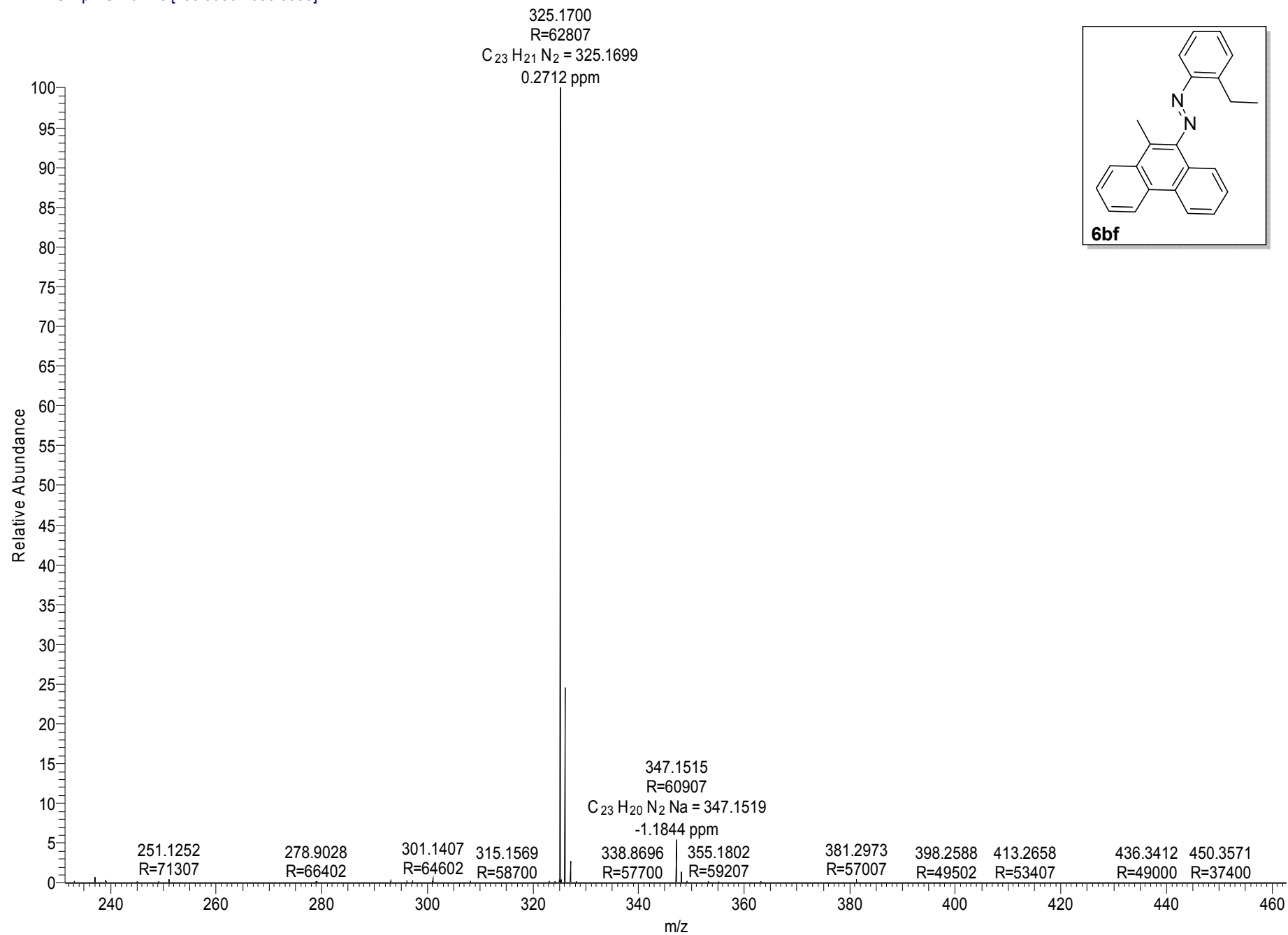
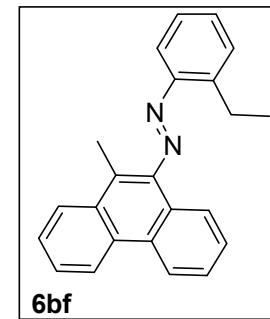
150.47
147.52
144.65
131.65
130.07
130.03
127.02
126.59
126.37
125.64
124.24
122.94
122.60
115.07

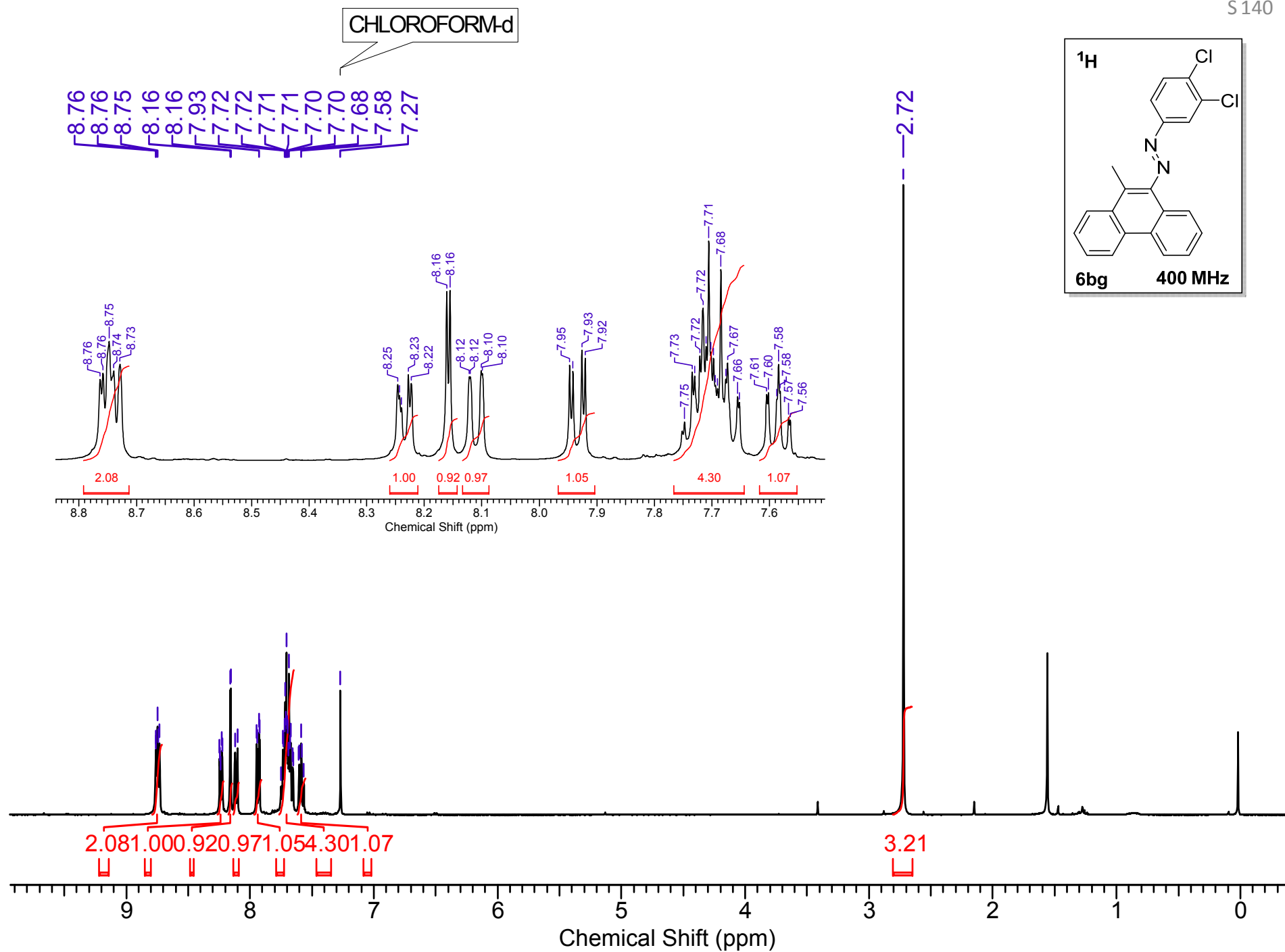
77.32
77.00
76.69

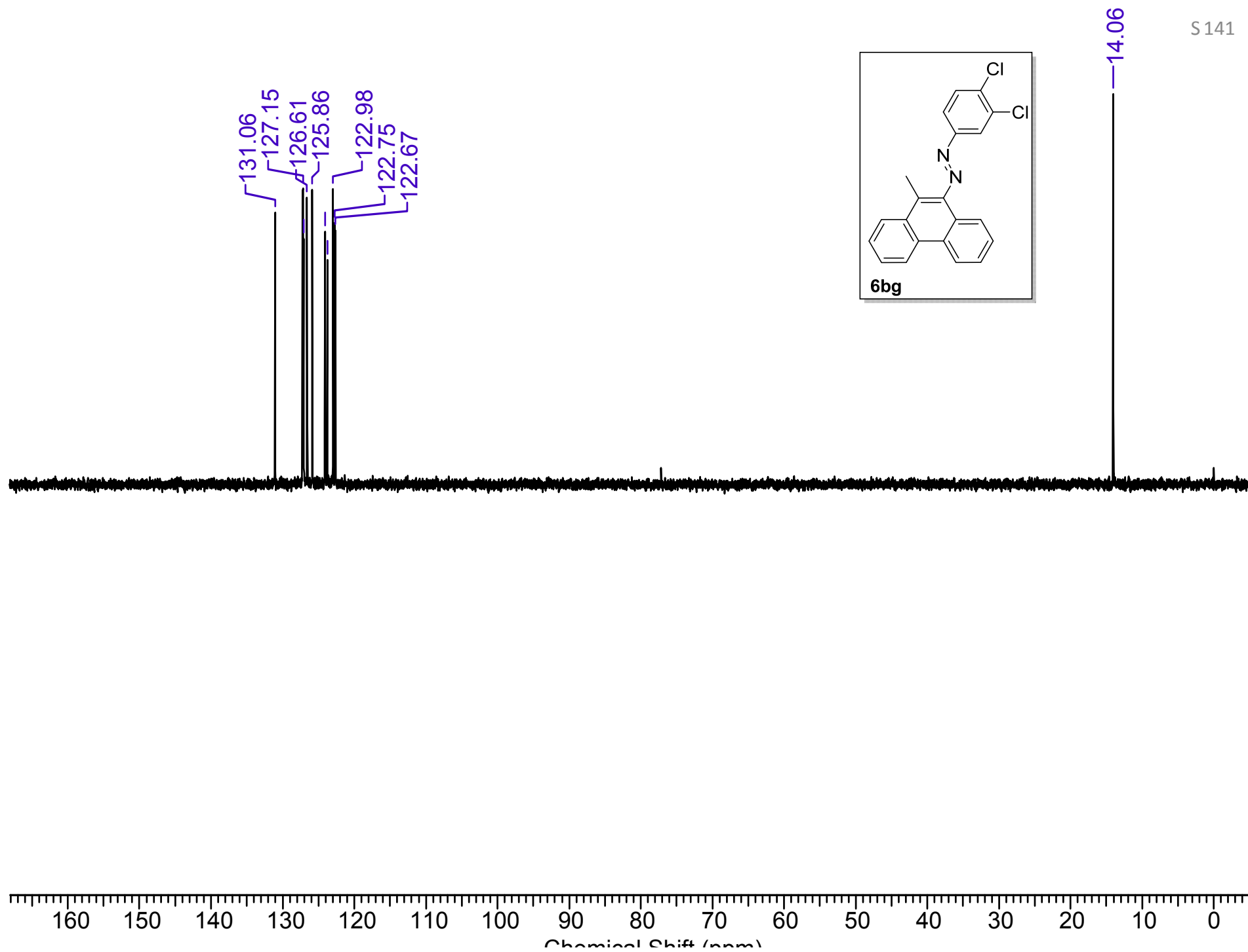
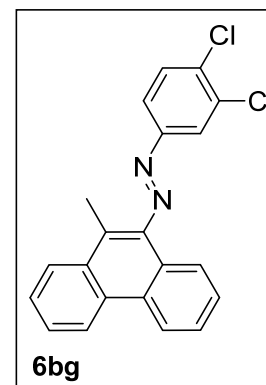
24.50
16.51
13.96

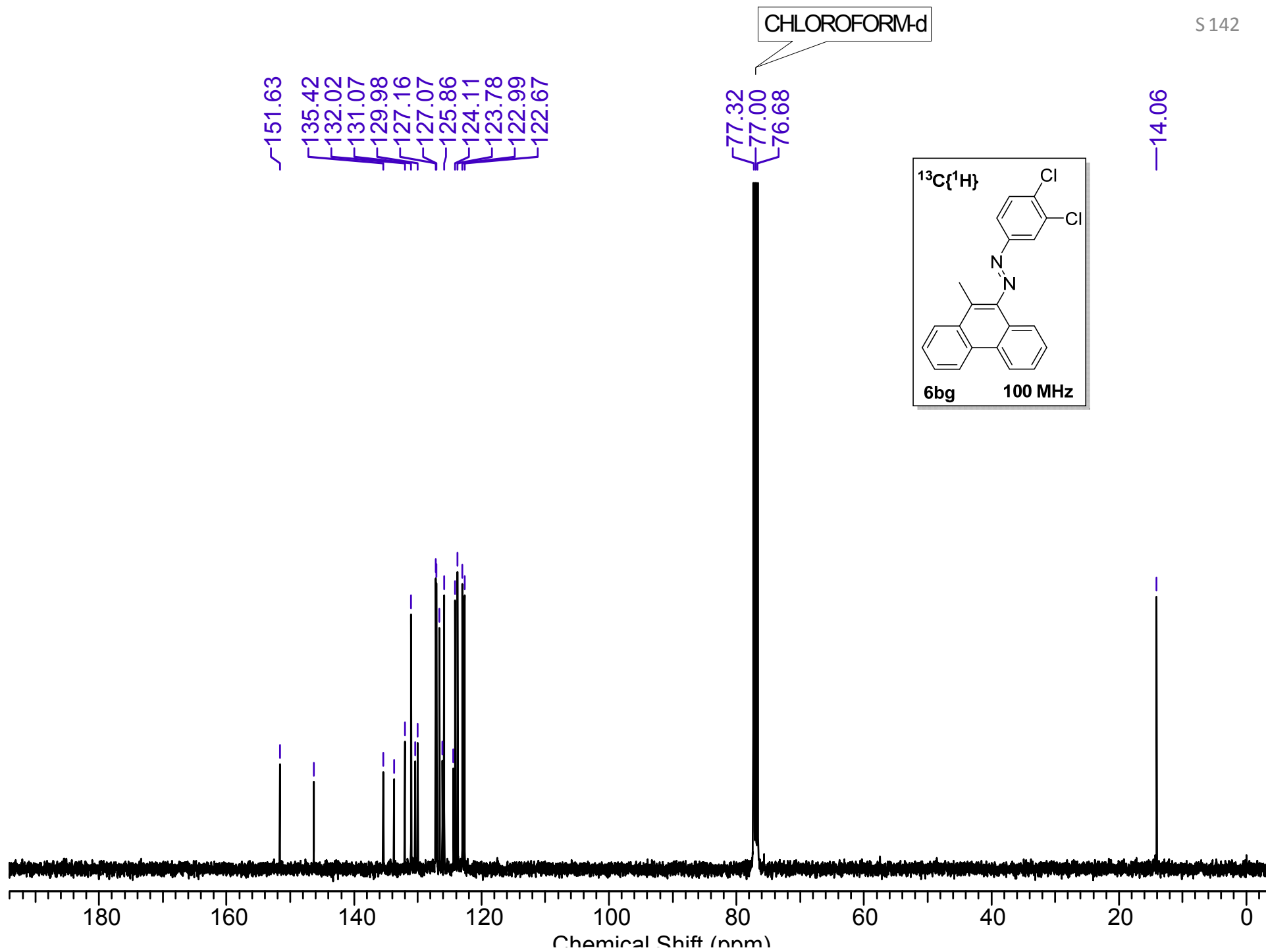
1.01

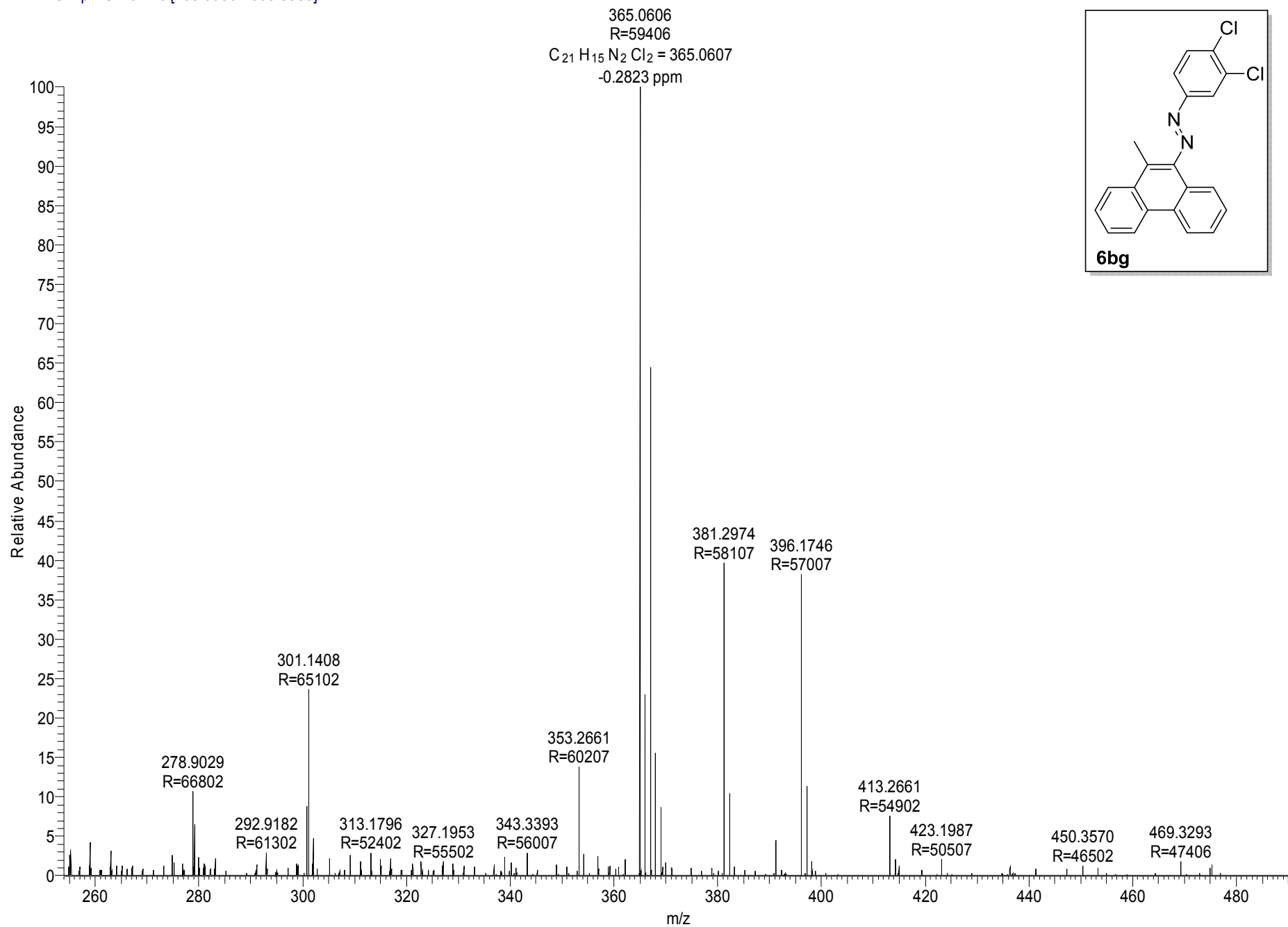
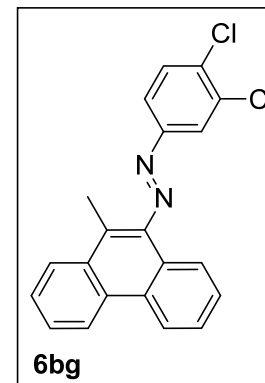


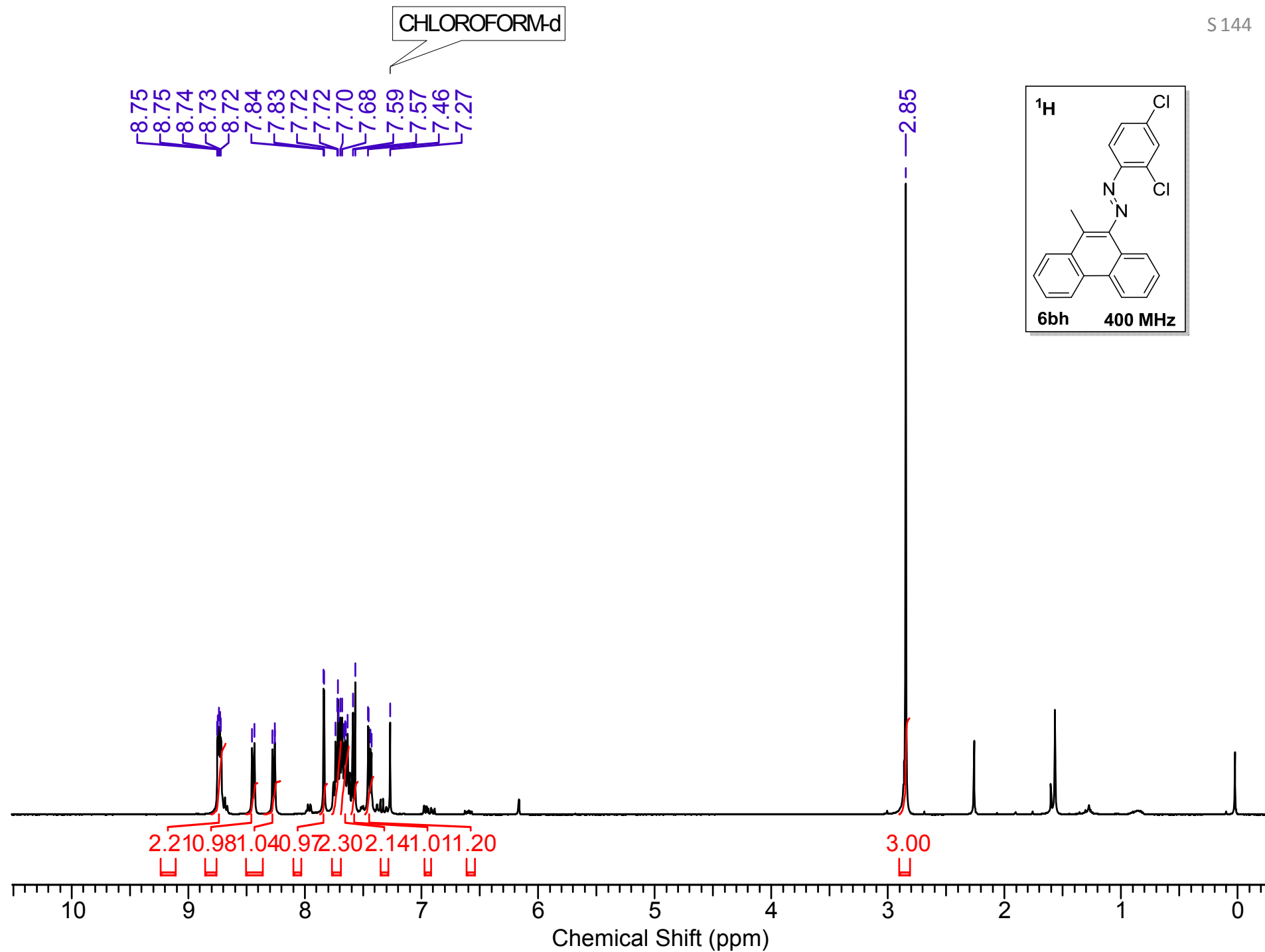


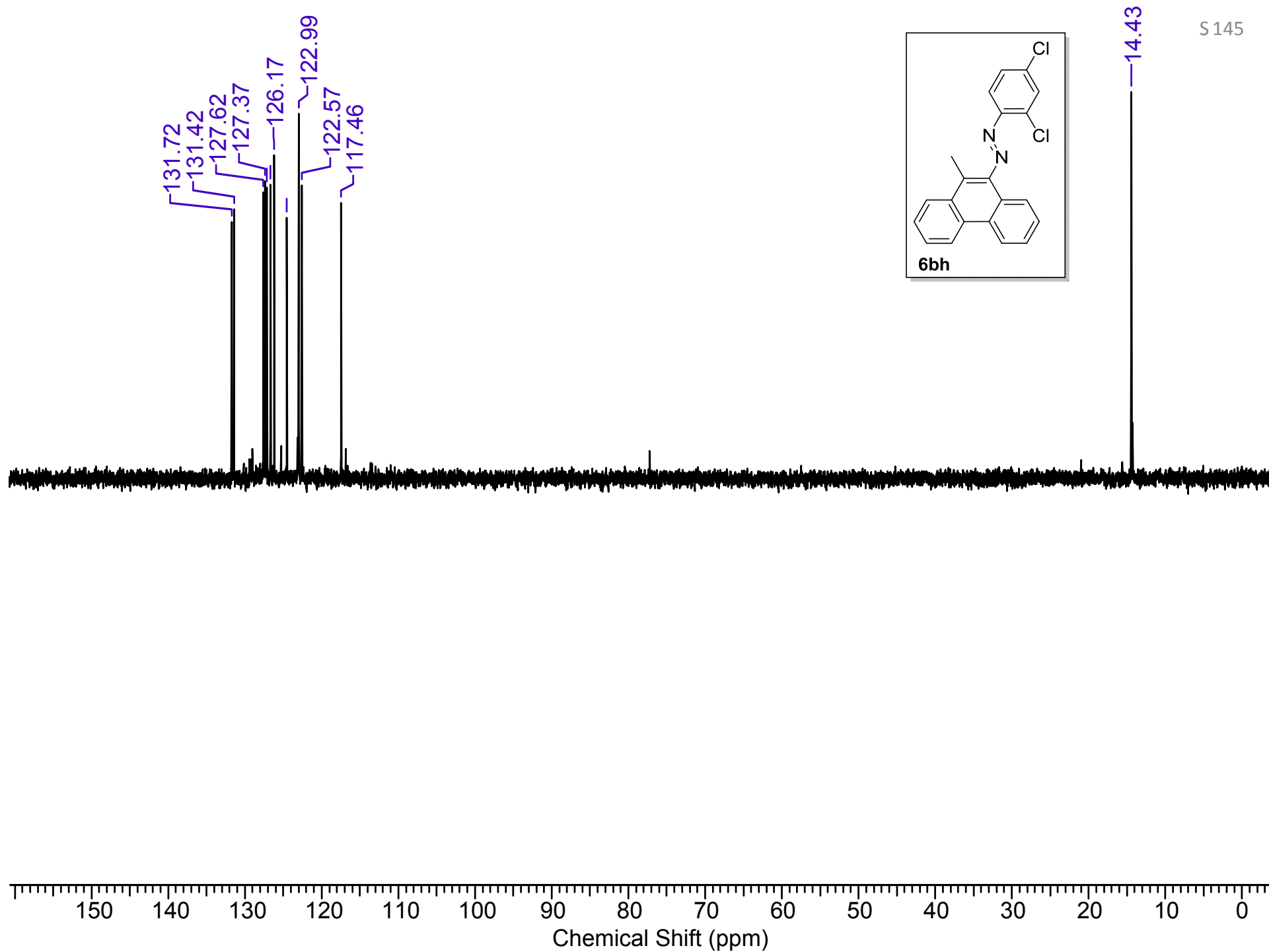
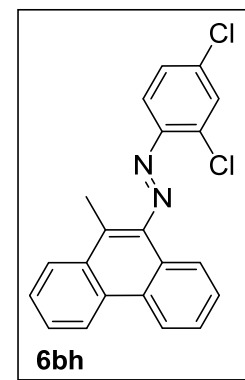


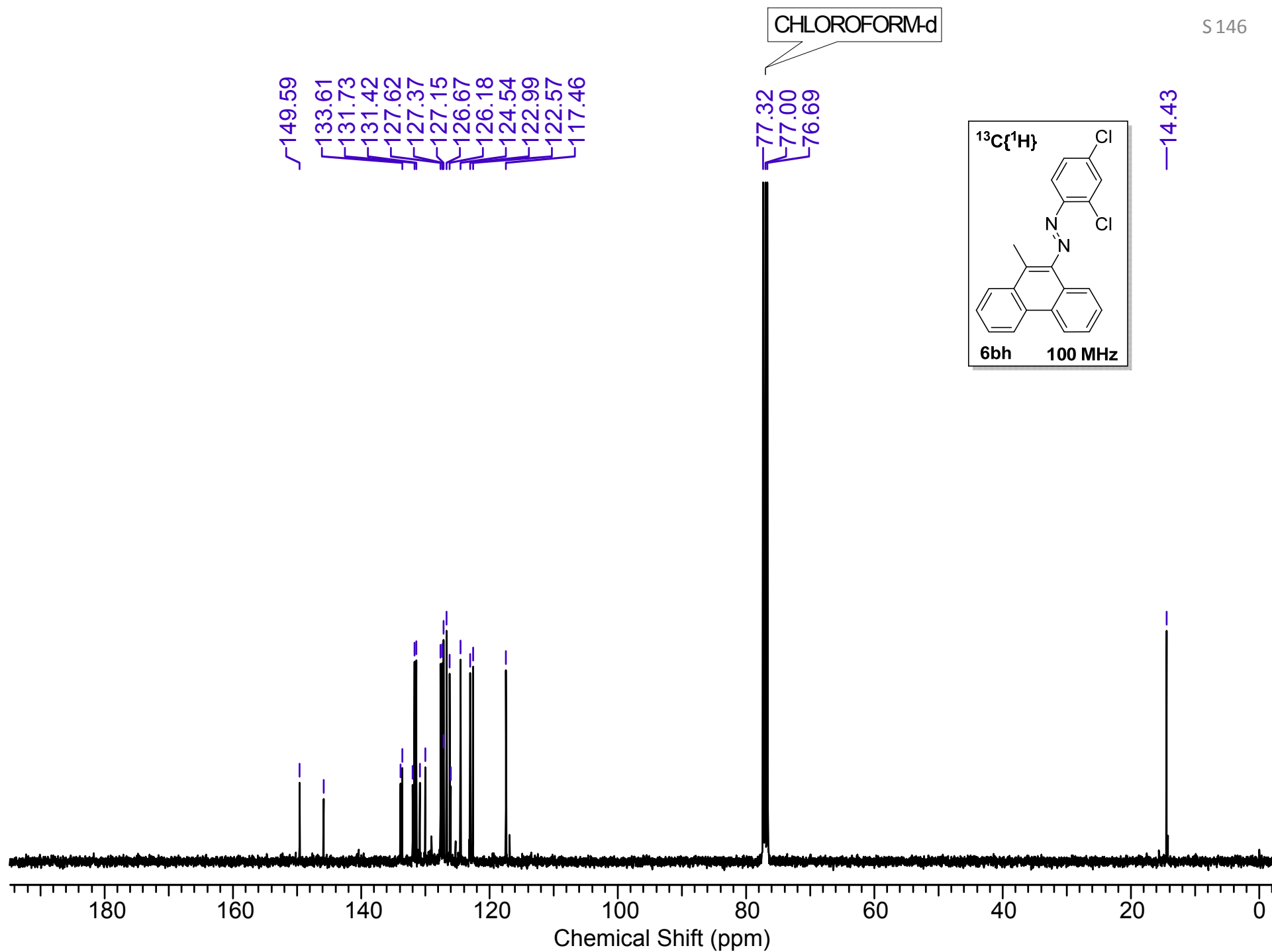


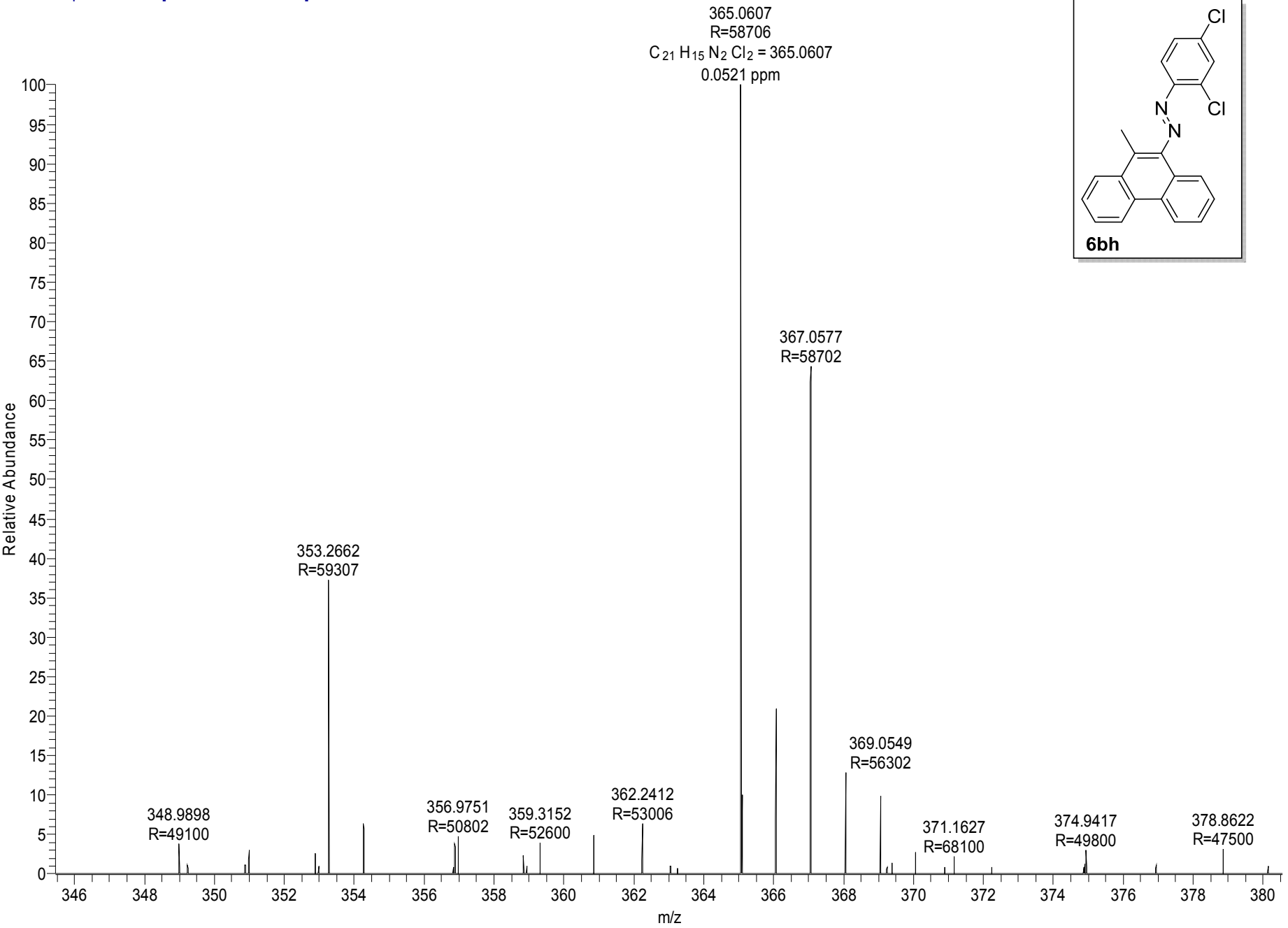


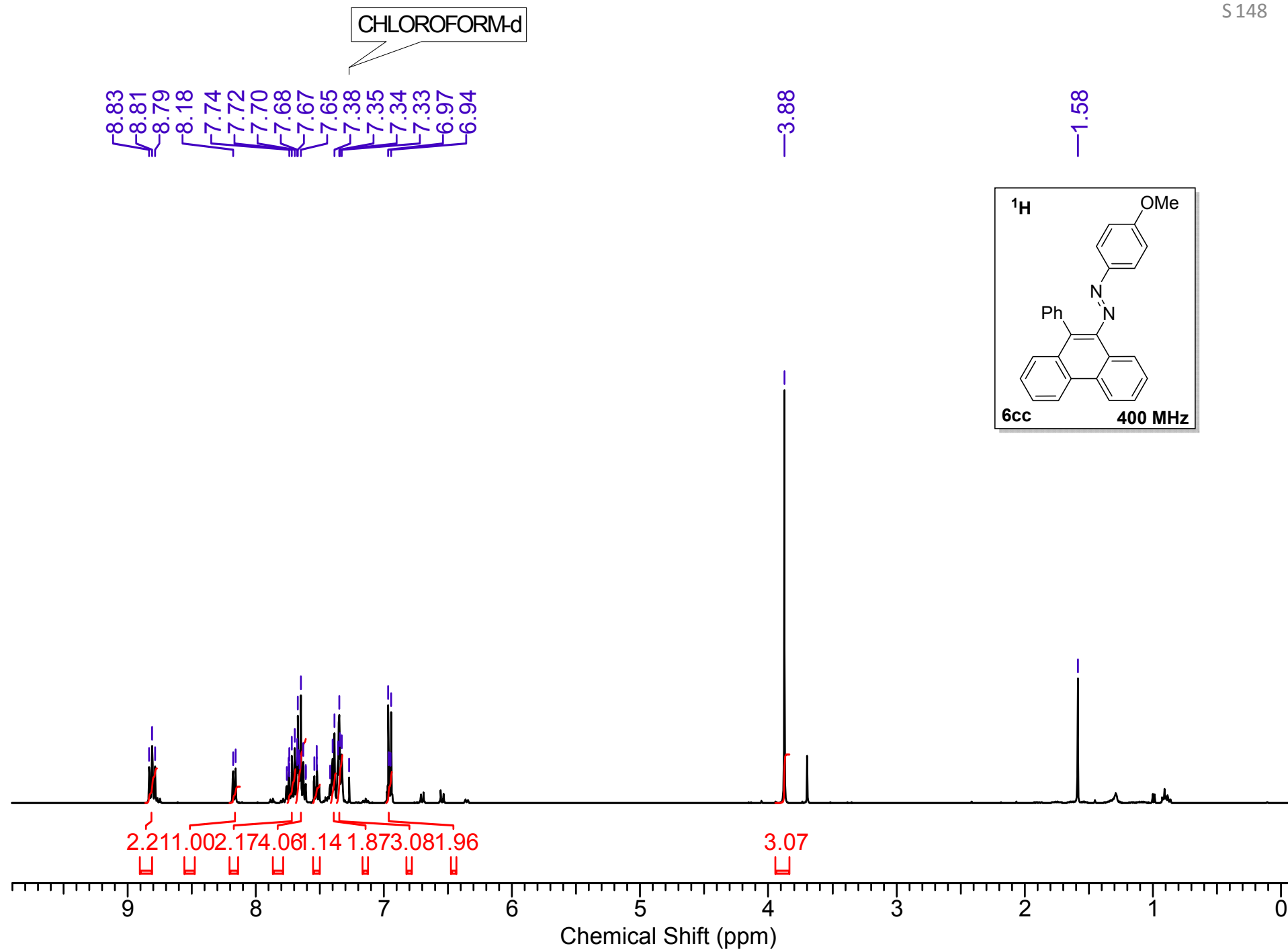


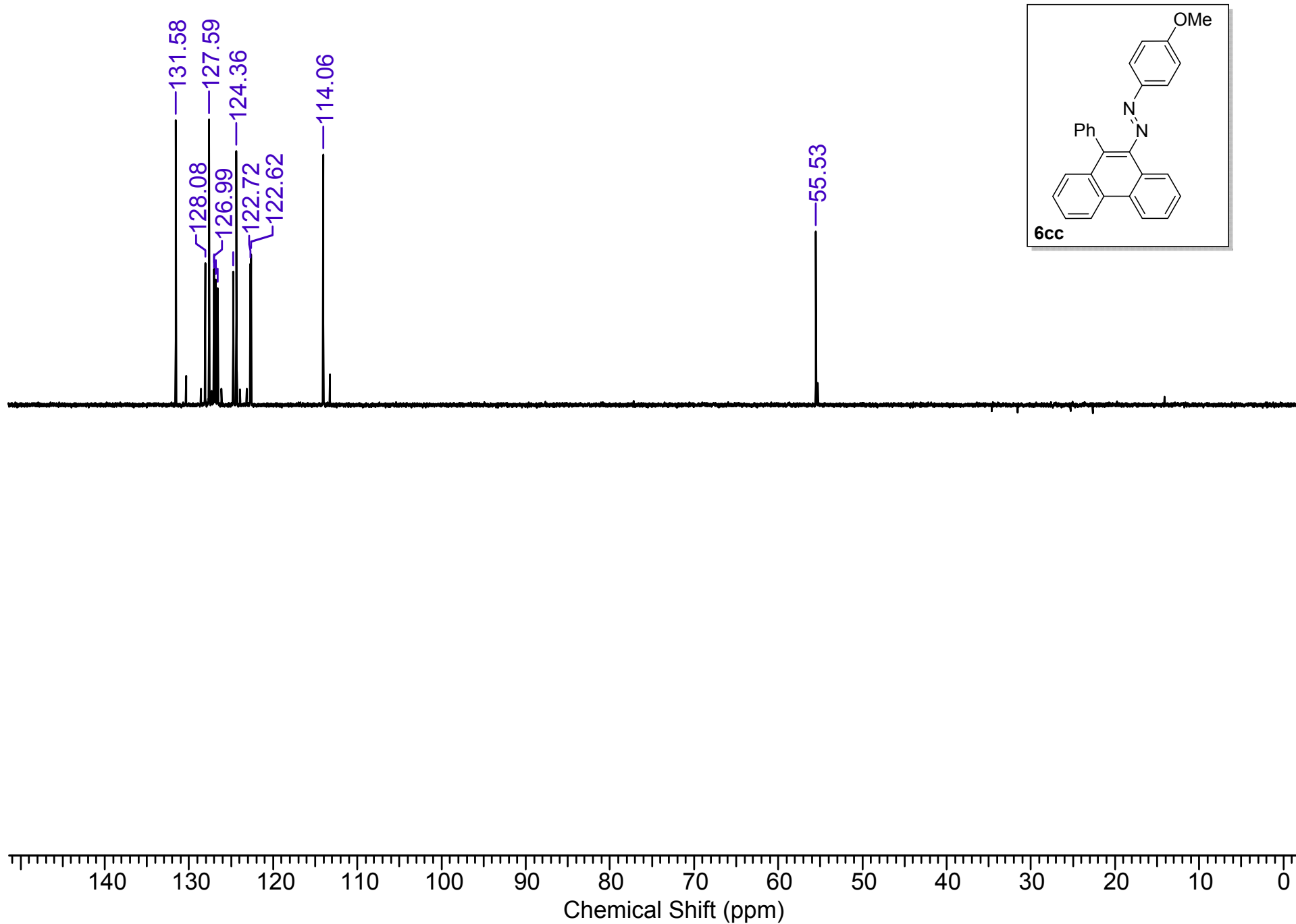


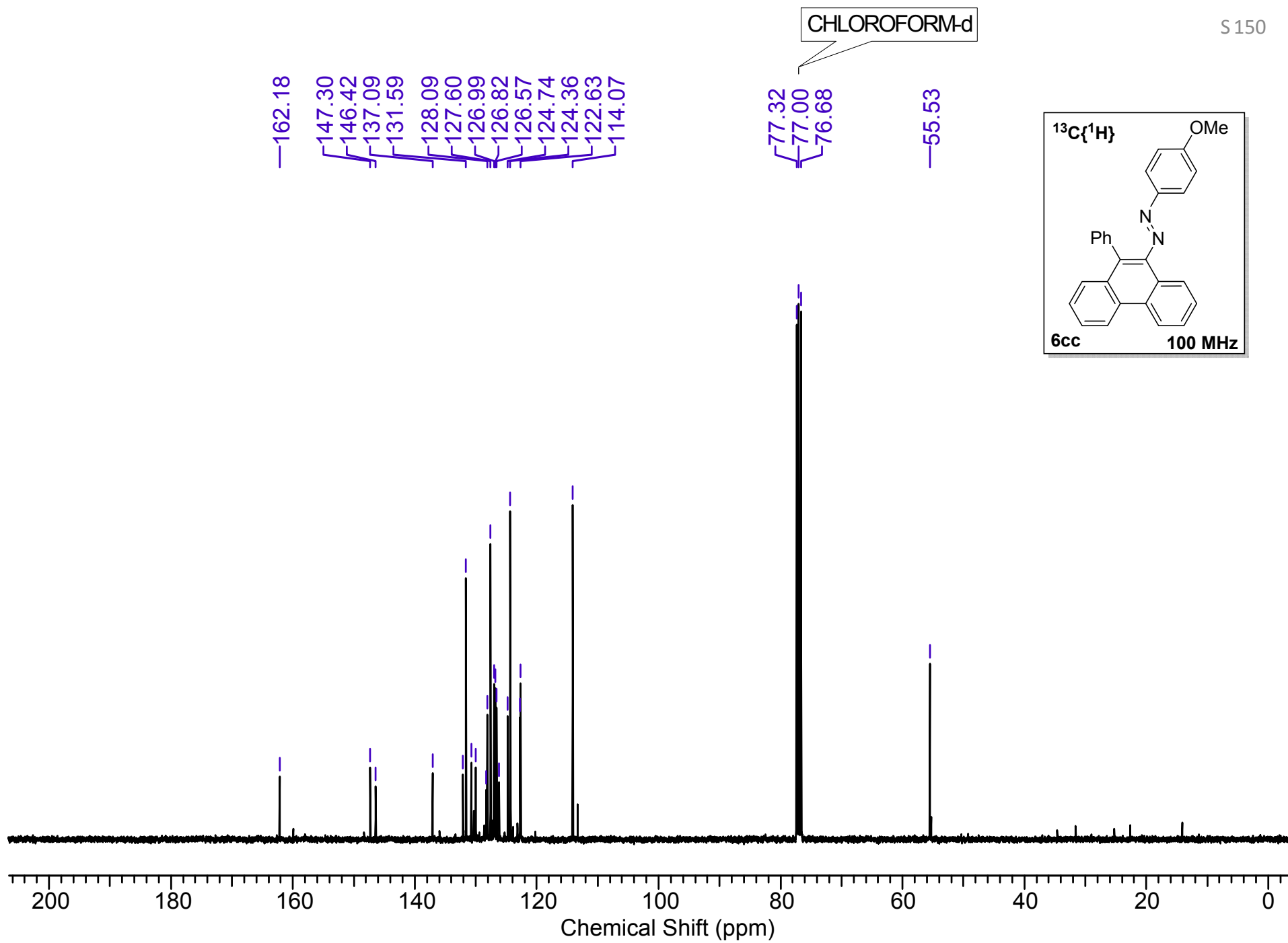








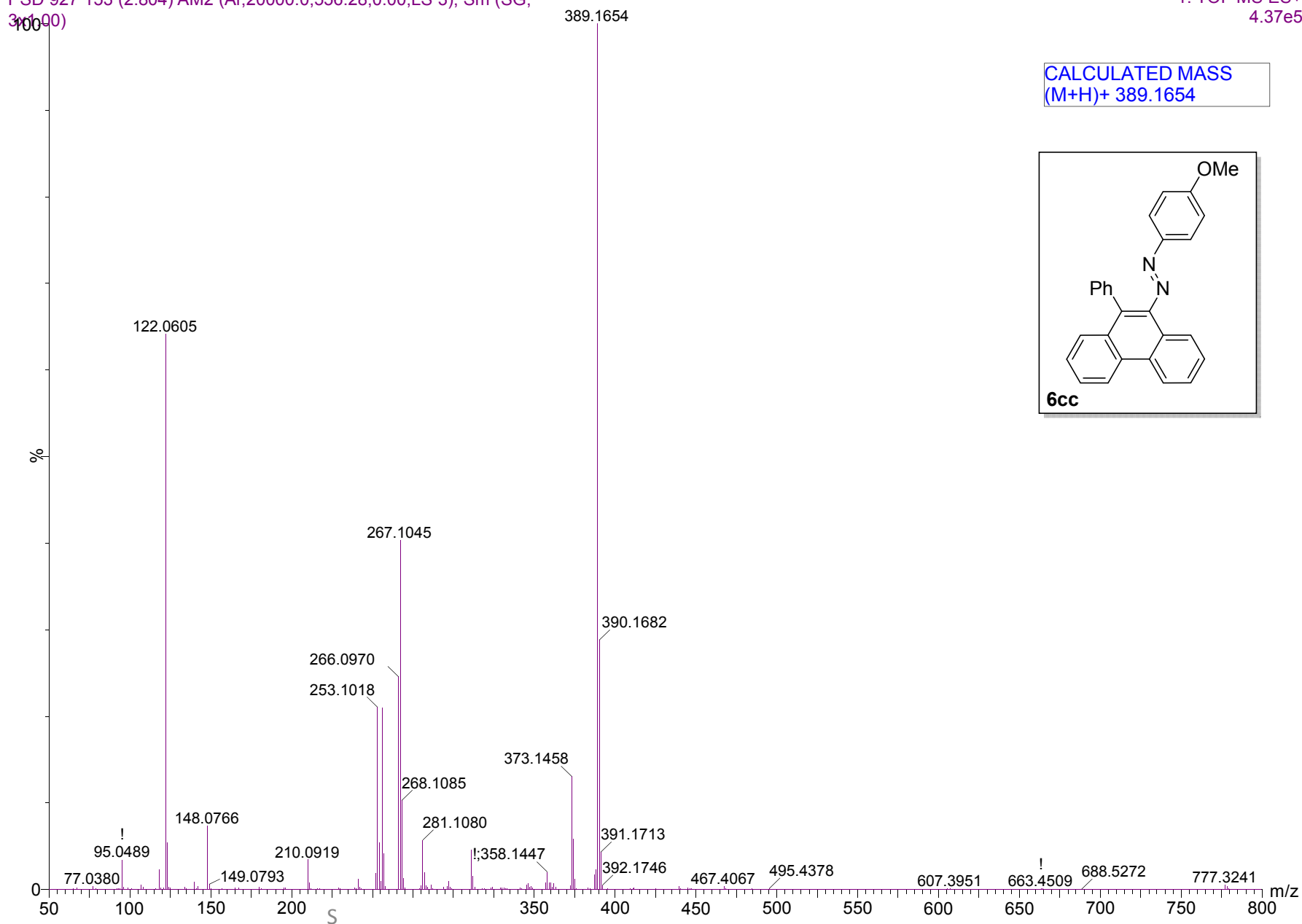
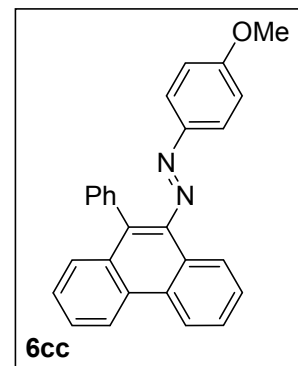




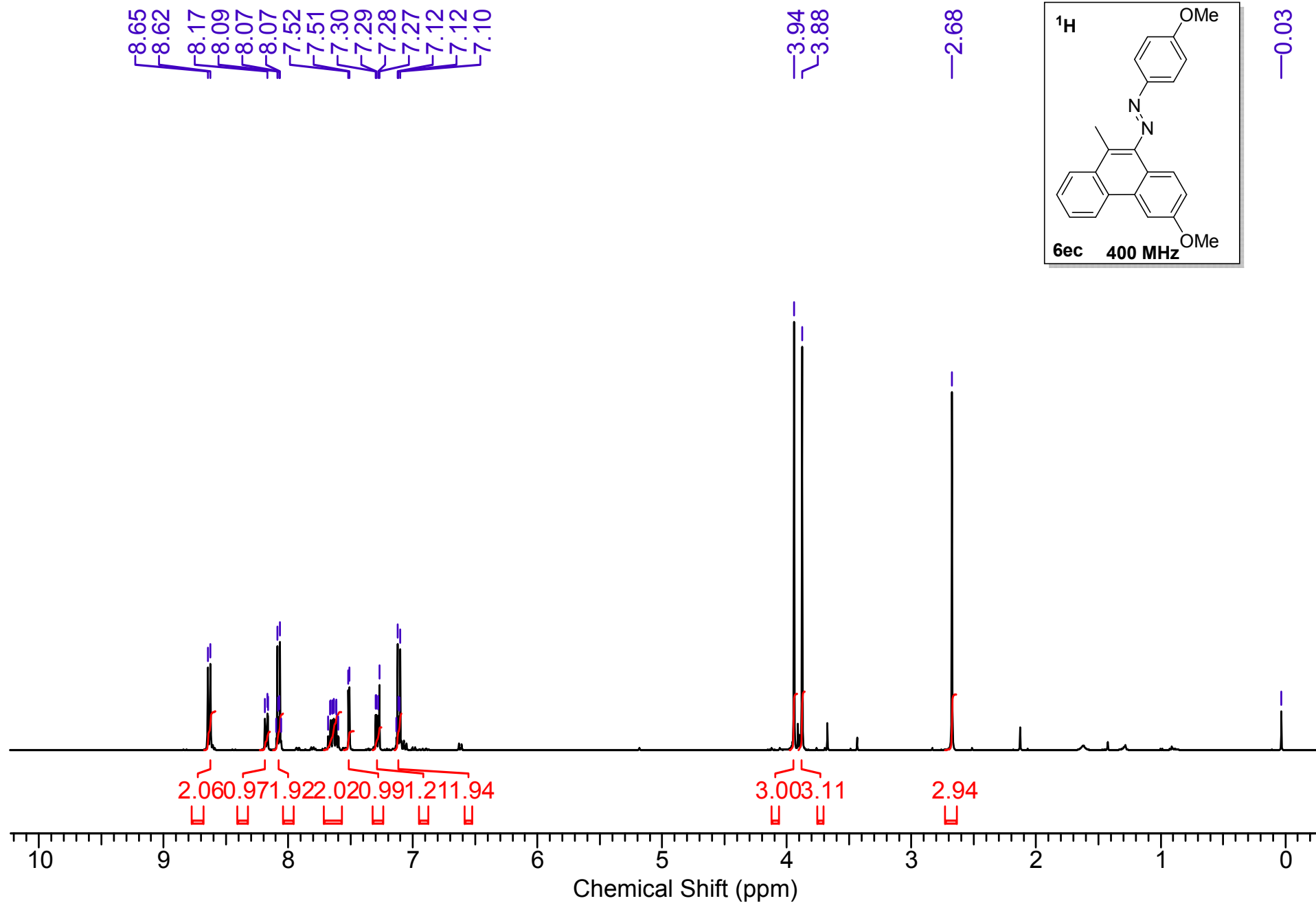
PSD 927

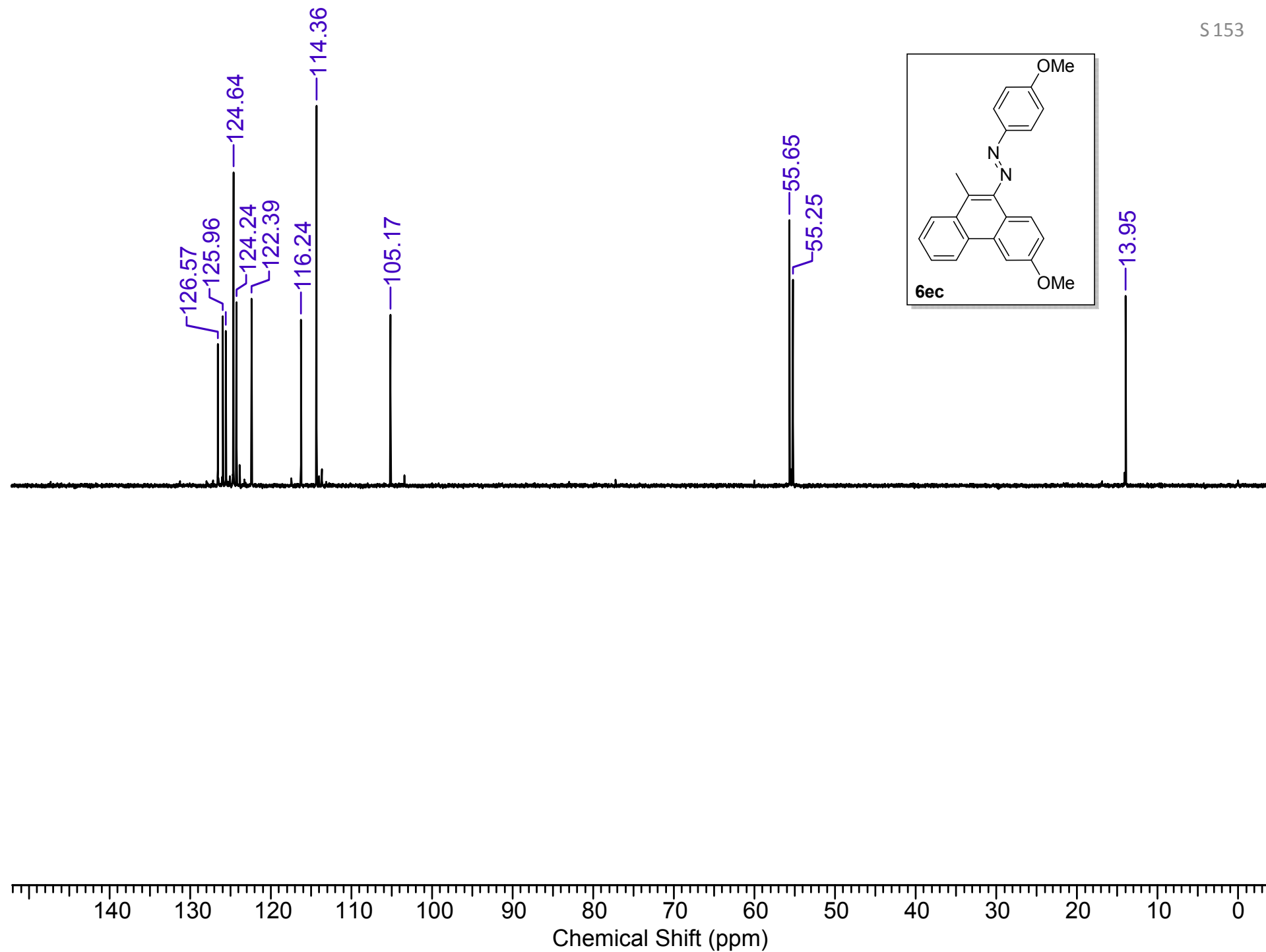
PSD 927 153 (2.804) AM2 (Ar,20000.0,556.28,0.00,LS 3); Sm (SG,
300.00)

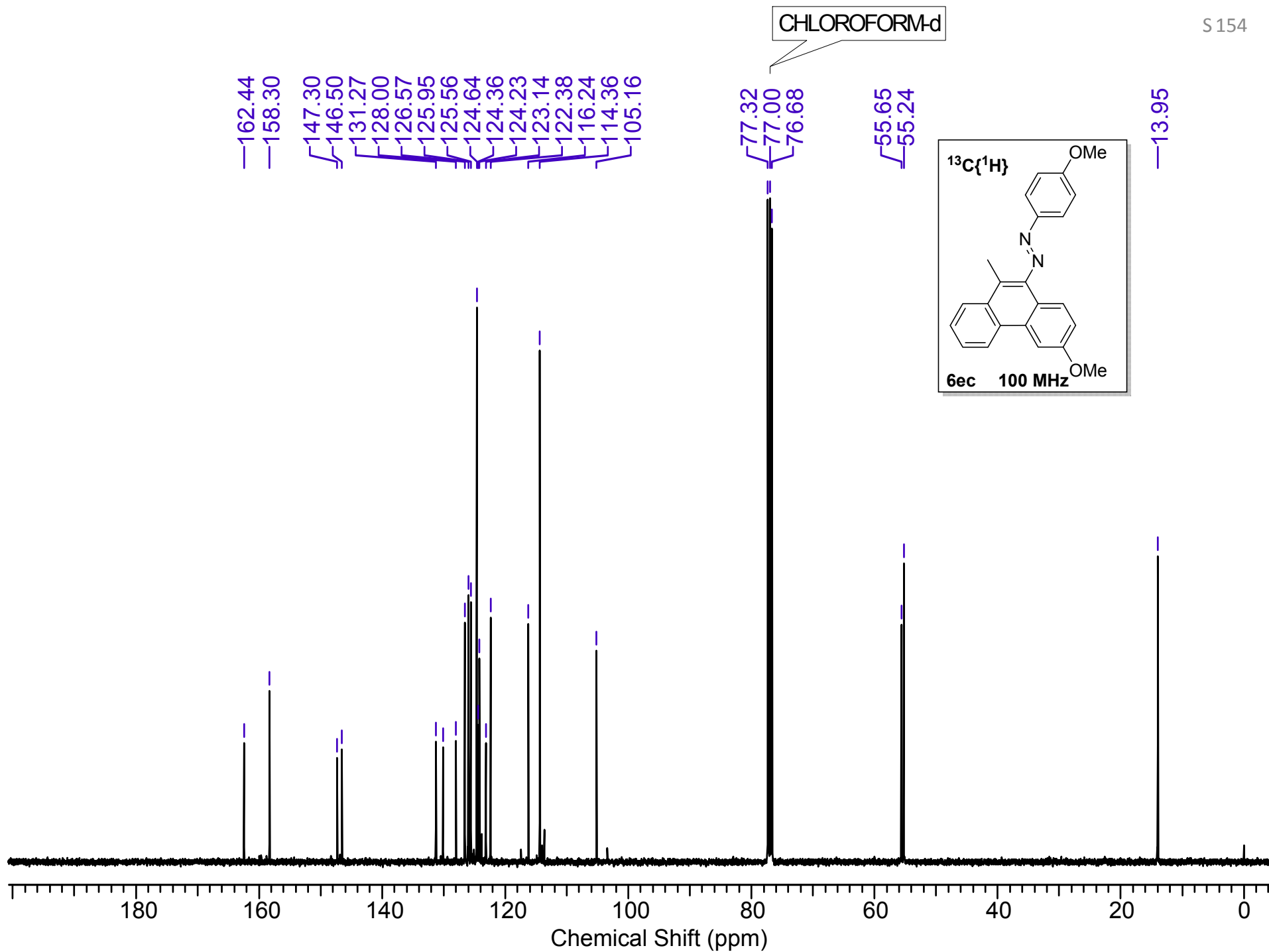
IISER PUNE

1: TOF MS ES+
4.37e5CALCULATED MASS
(M+H)+ 389.1654

CHLOROFORM-d

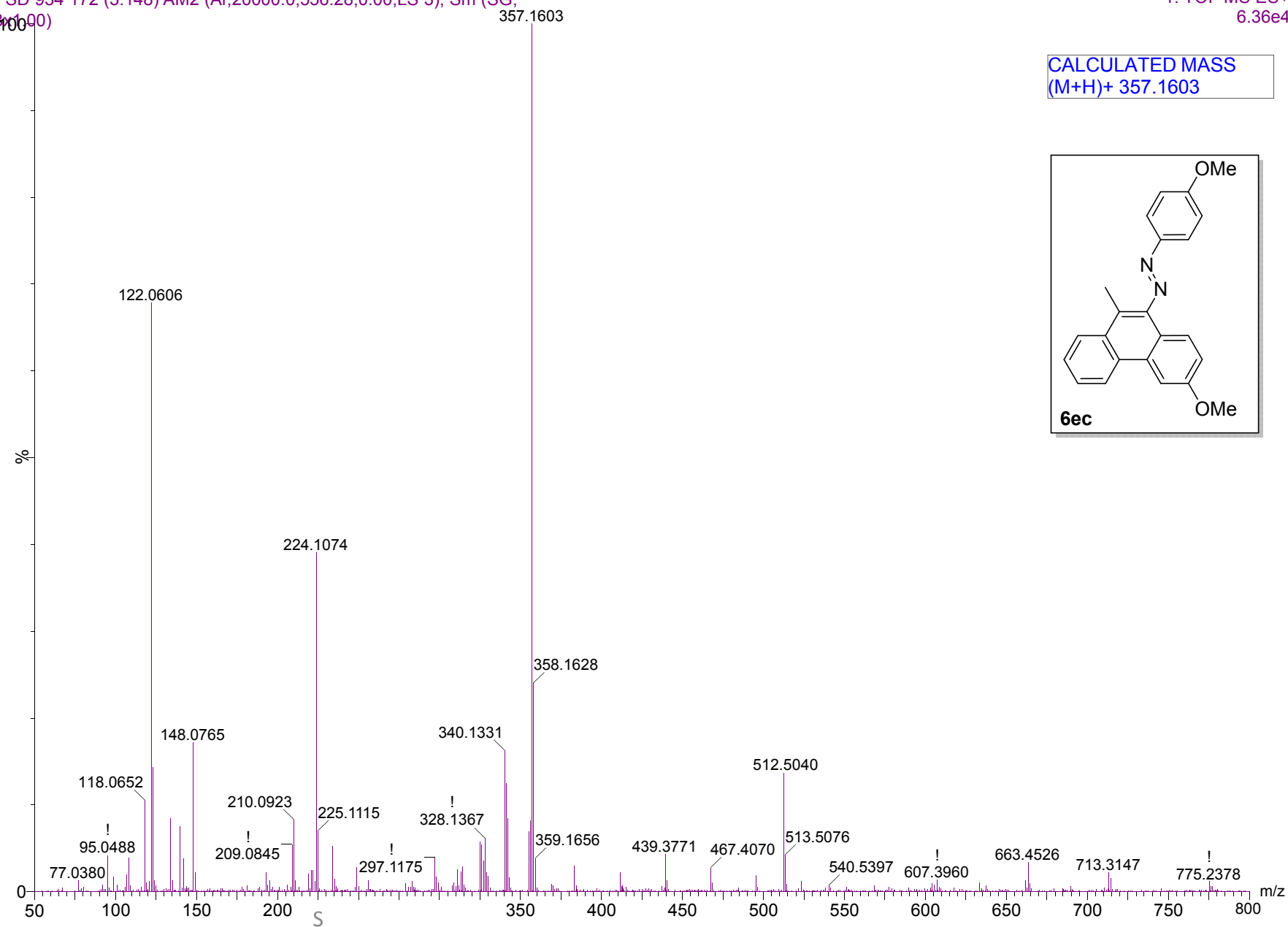
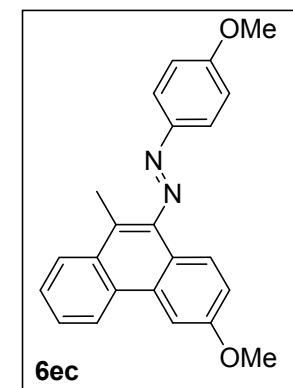




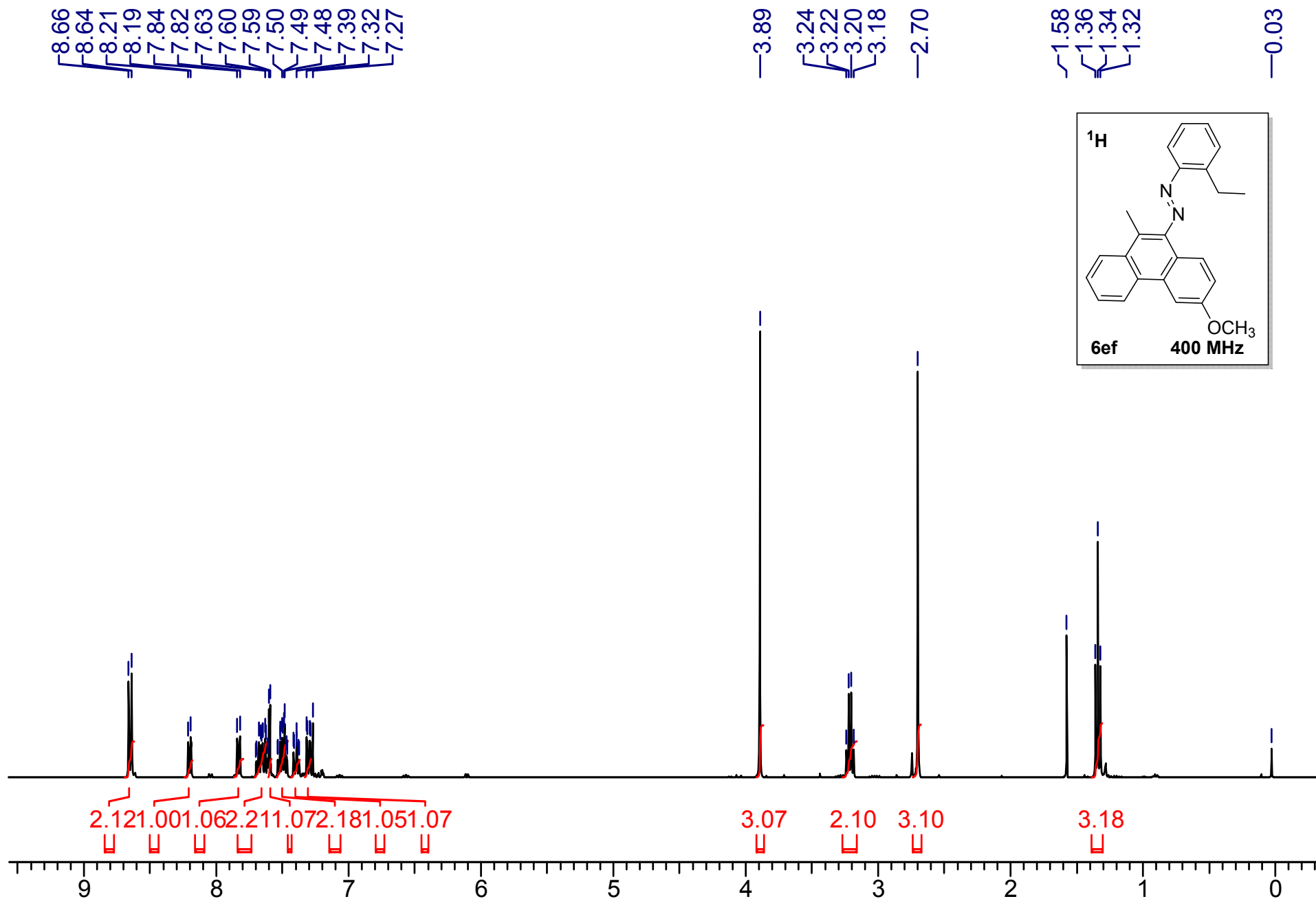


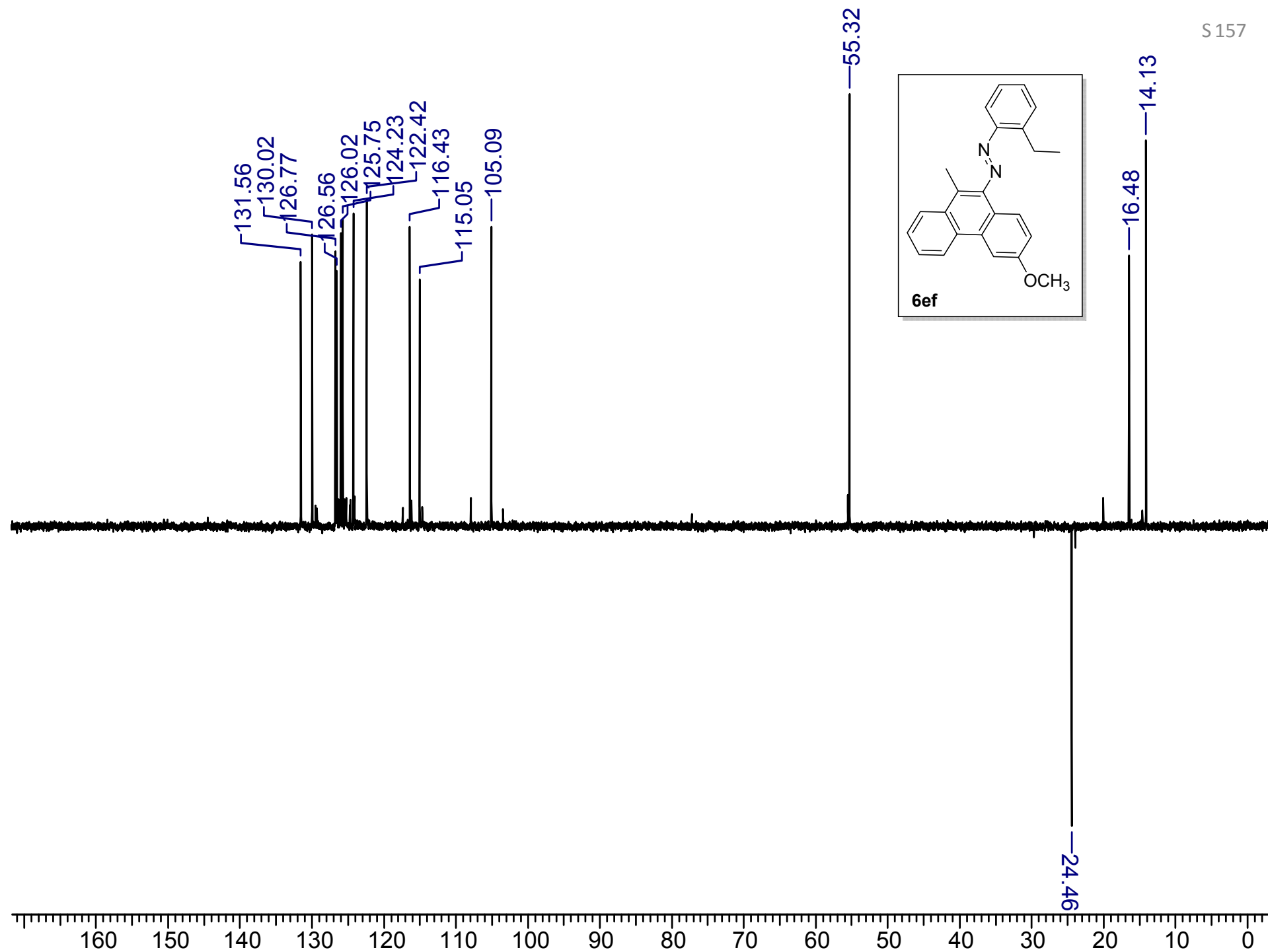
PSD 934

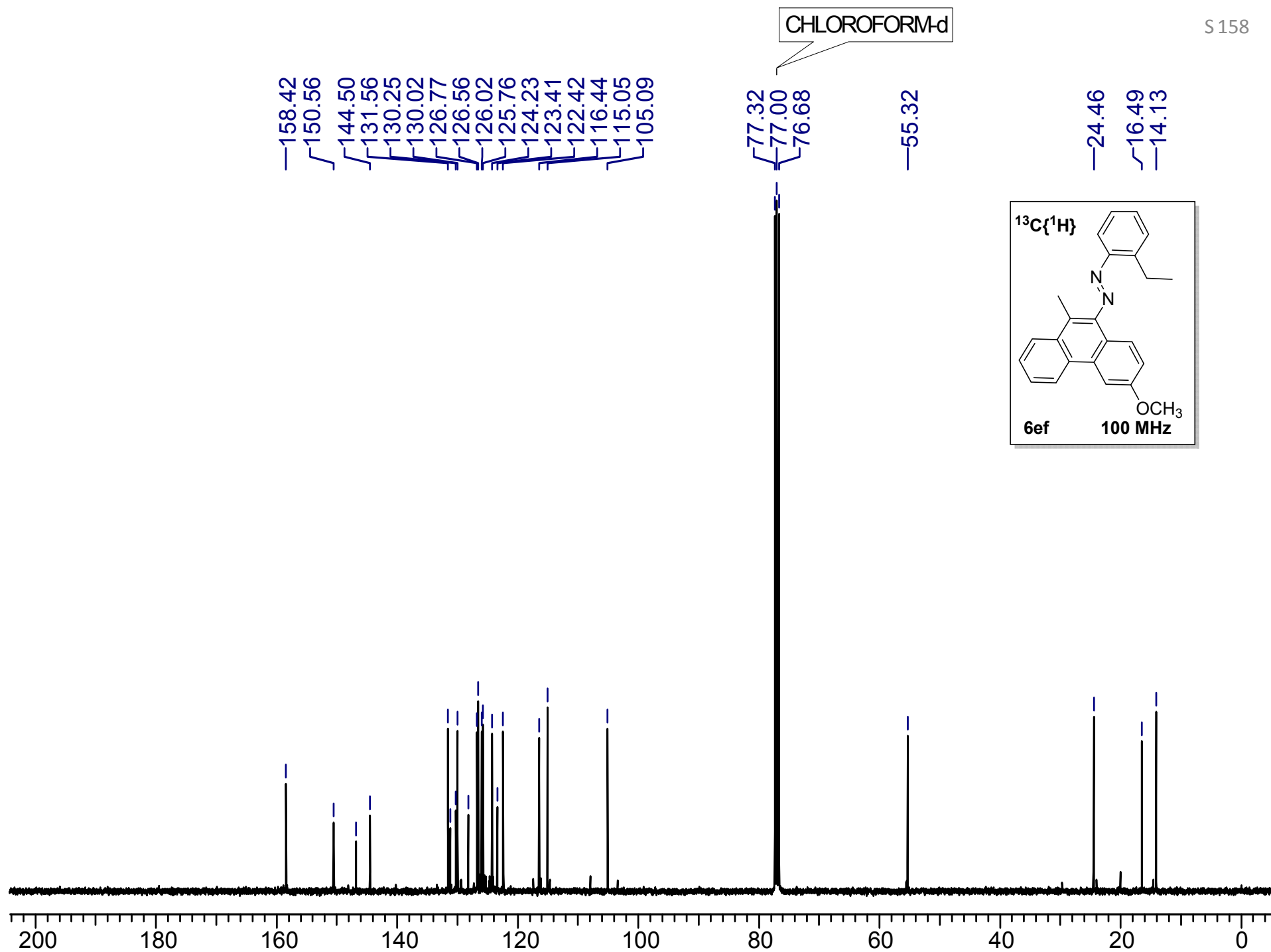
IISER PUNE

PSD 934 172 (3.148) AM2 (Ar,20000.0,556.28,0.00,LS 3); Sm (SG,
300.00)1: TOF MS ES+
6.36e4CALCULATED MASS
(M+H)⁺ 357.1603

CHLOROFORM-d

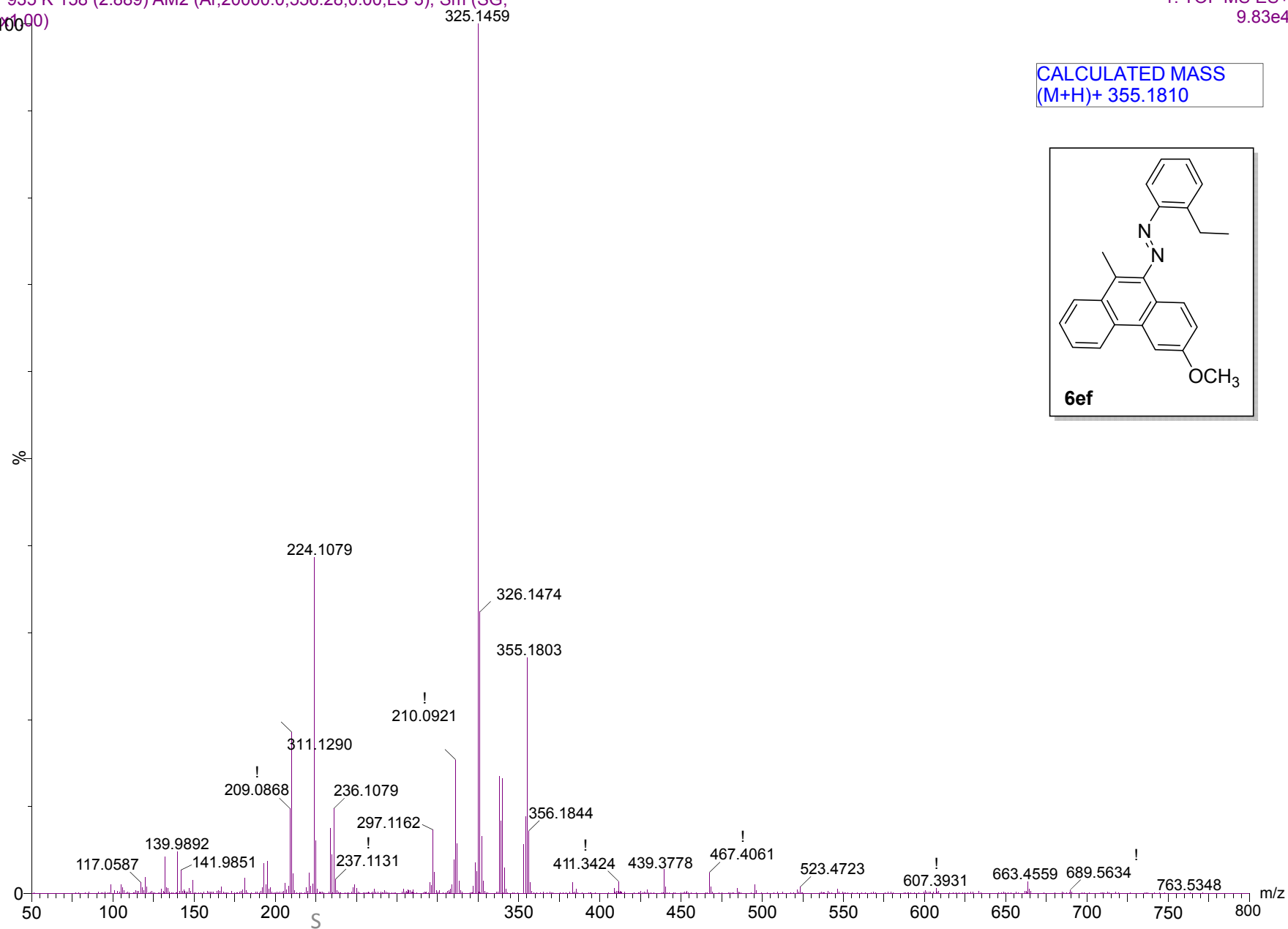
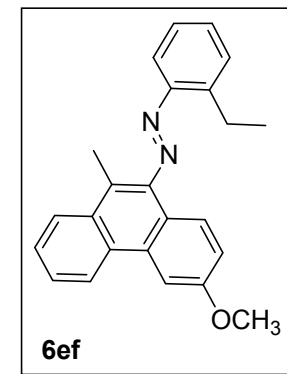


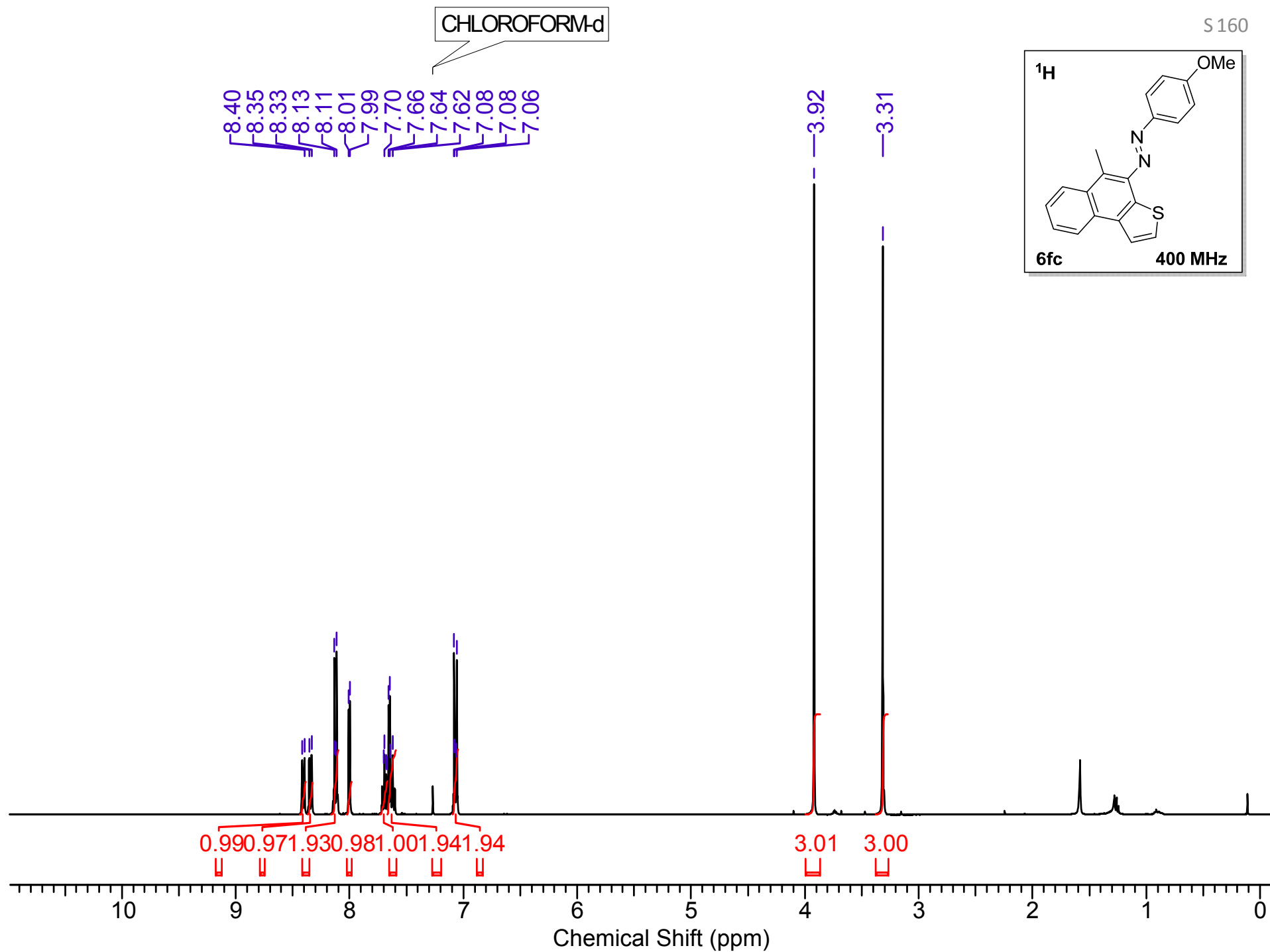


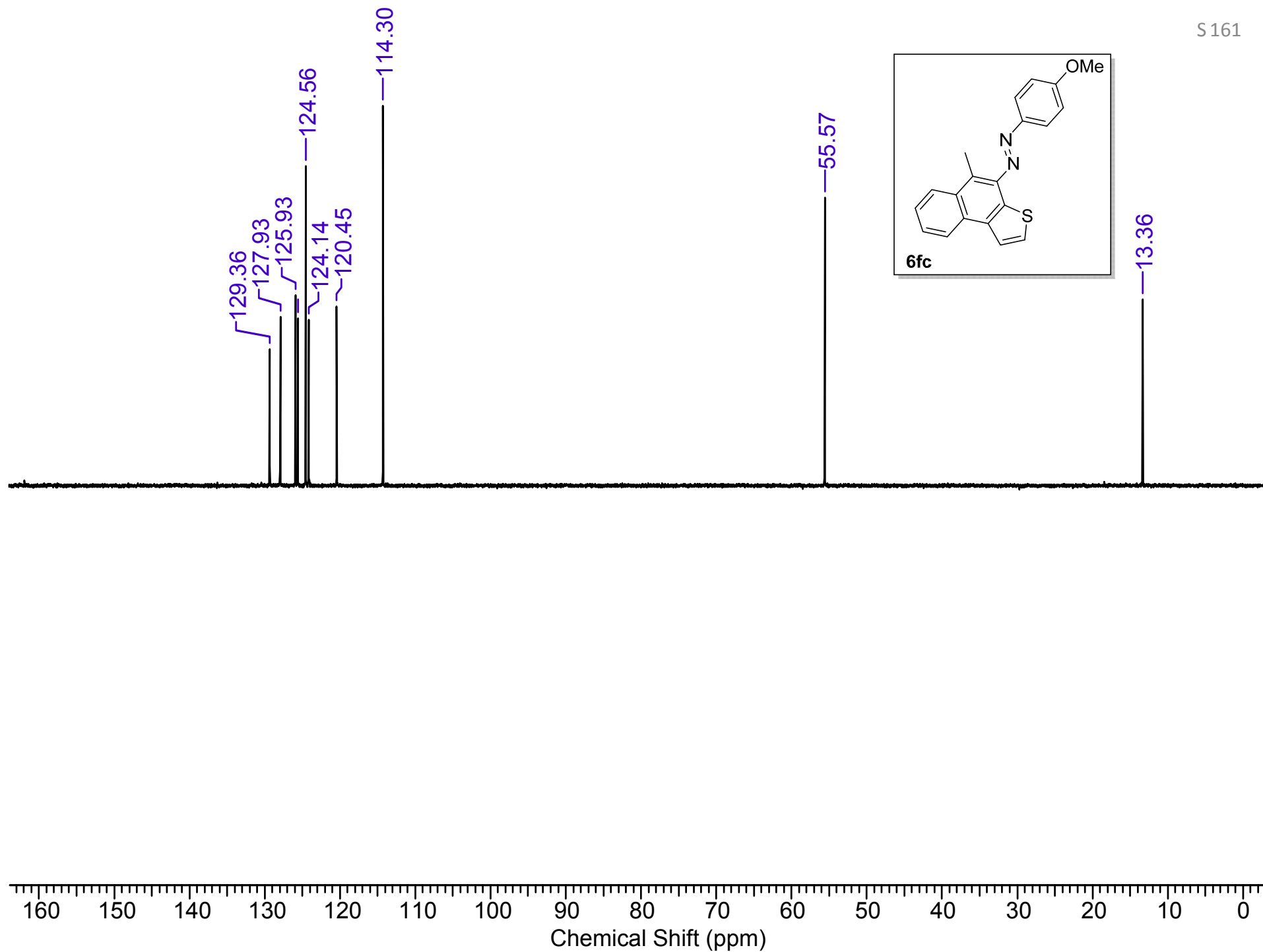
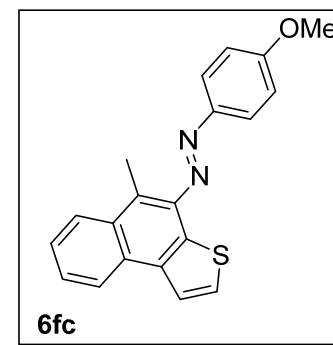


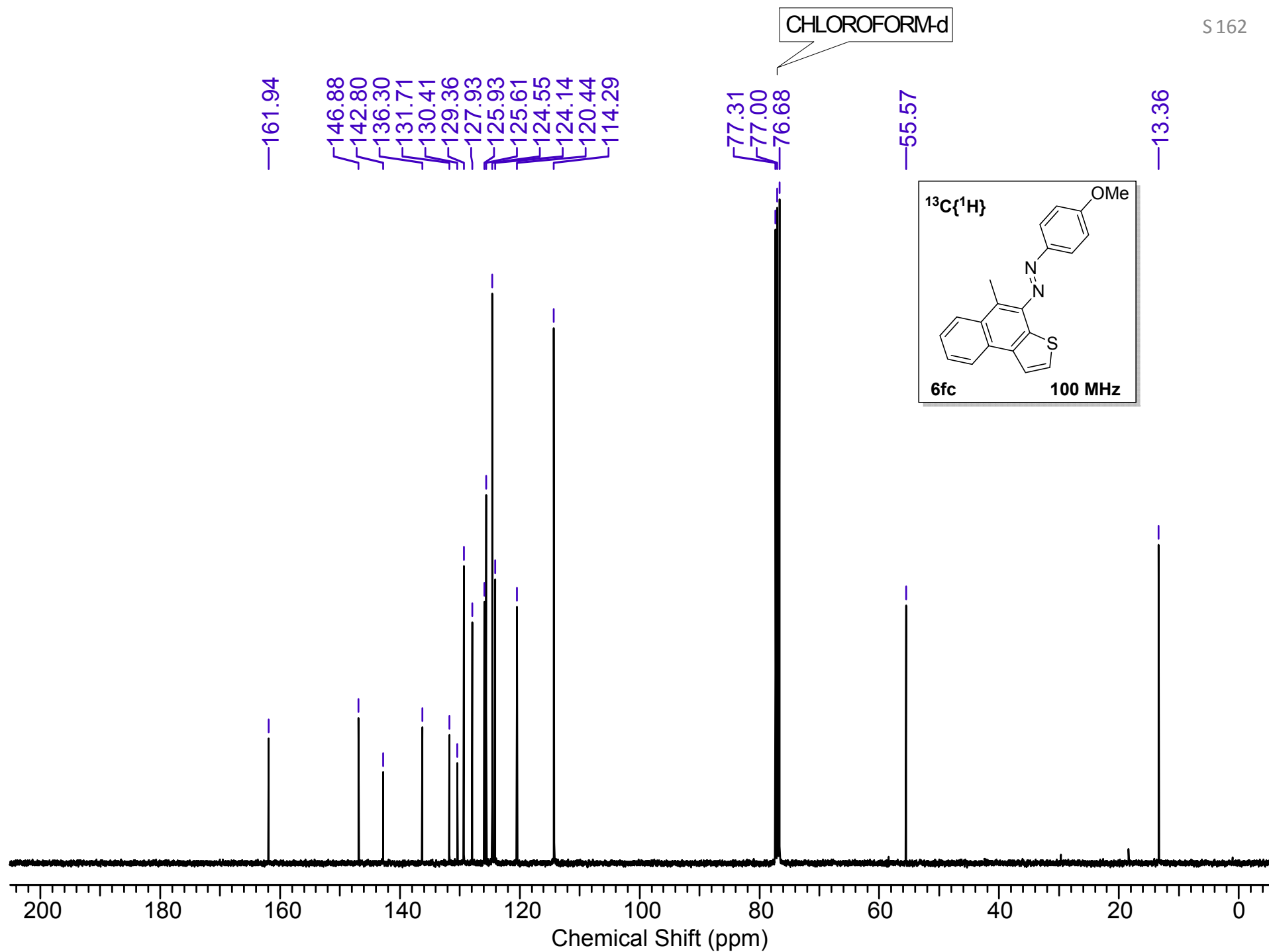
Y 935 K

IISER PUNE

1: TOF MS ES+
9.83e4Y 935 K 158 (2.889) AM2 (Ar,20000.0,556.28,0.00,LS 3); Sm (SG,
300.00)CALCULATED MASS
(M+H)⁺ 355.1810

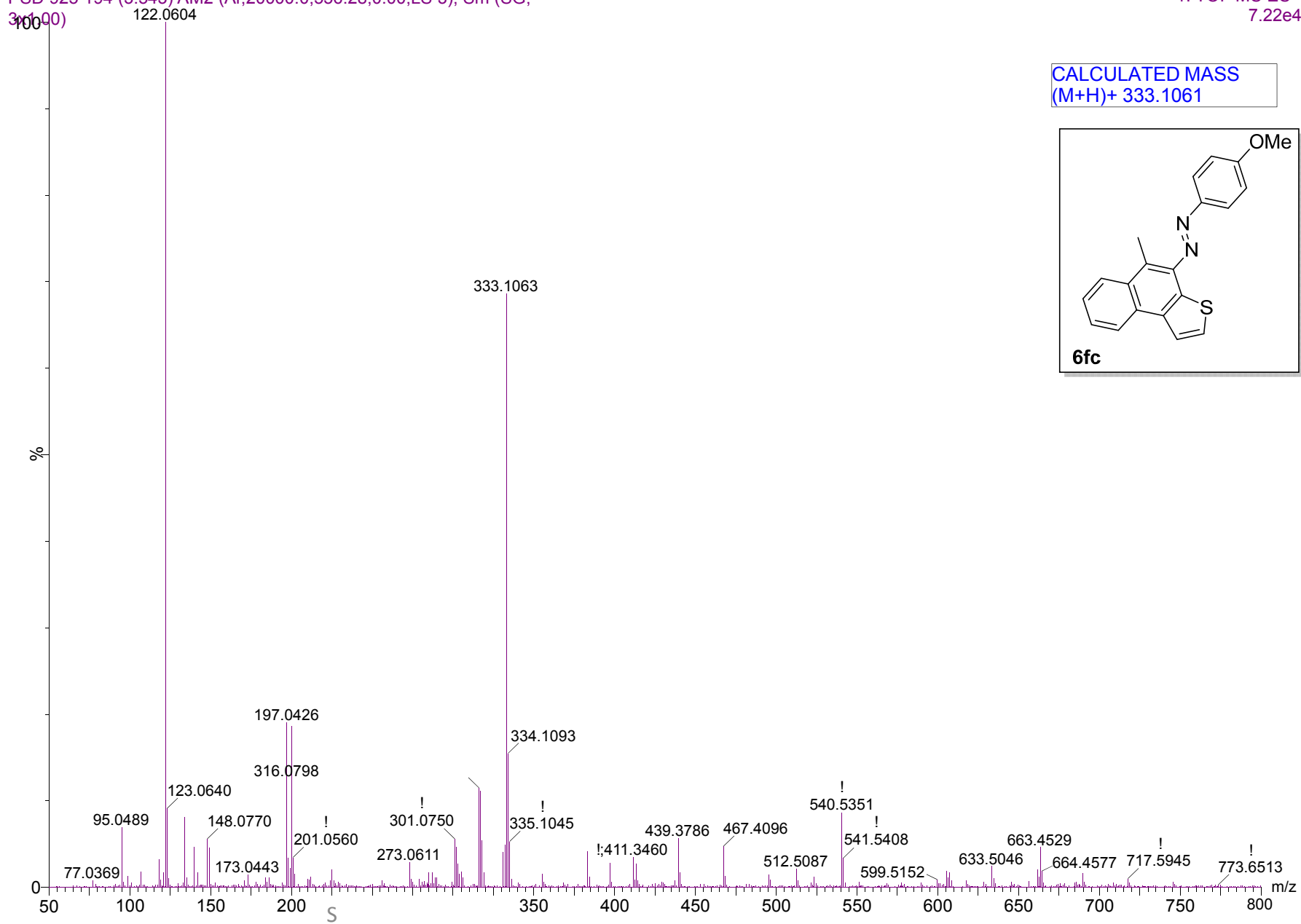
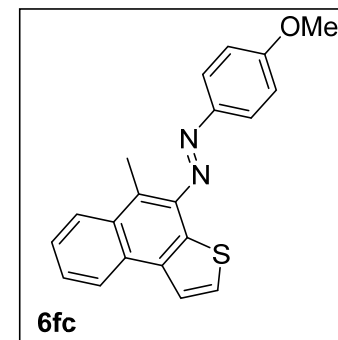


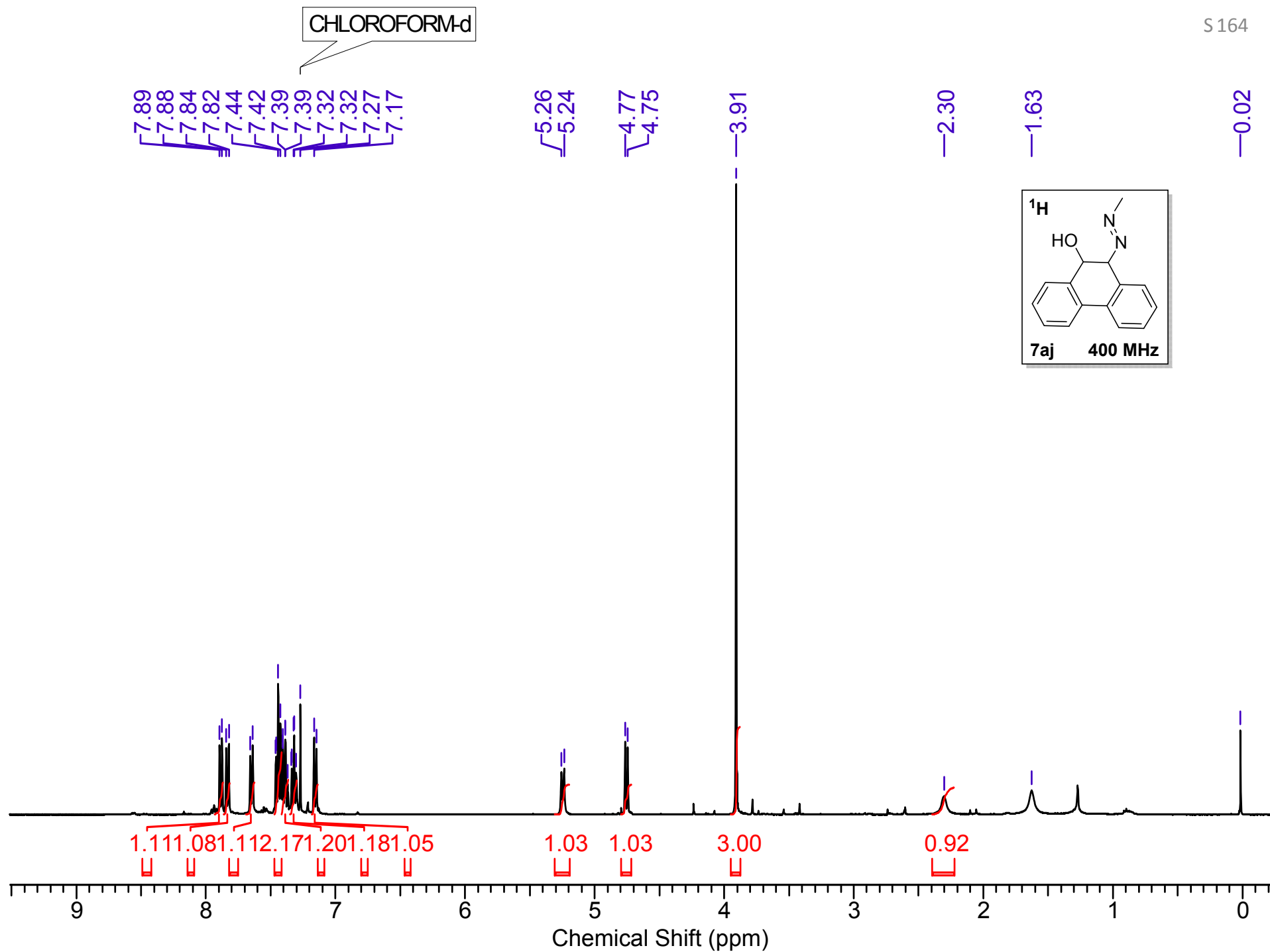


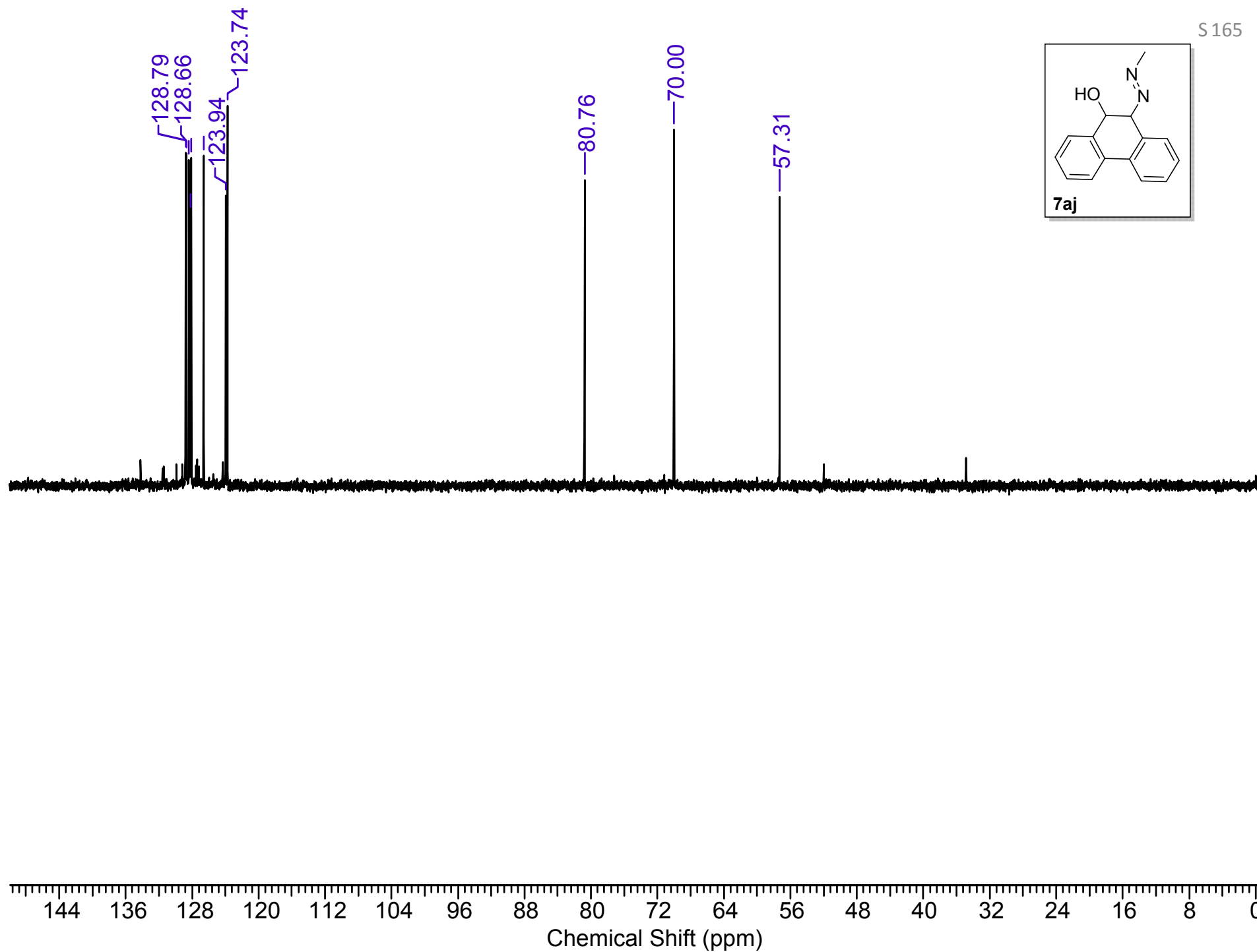


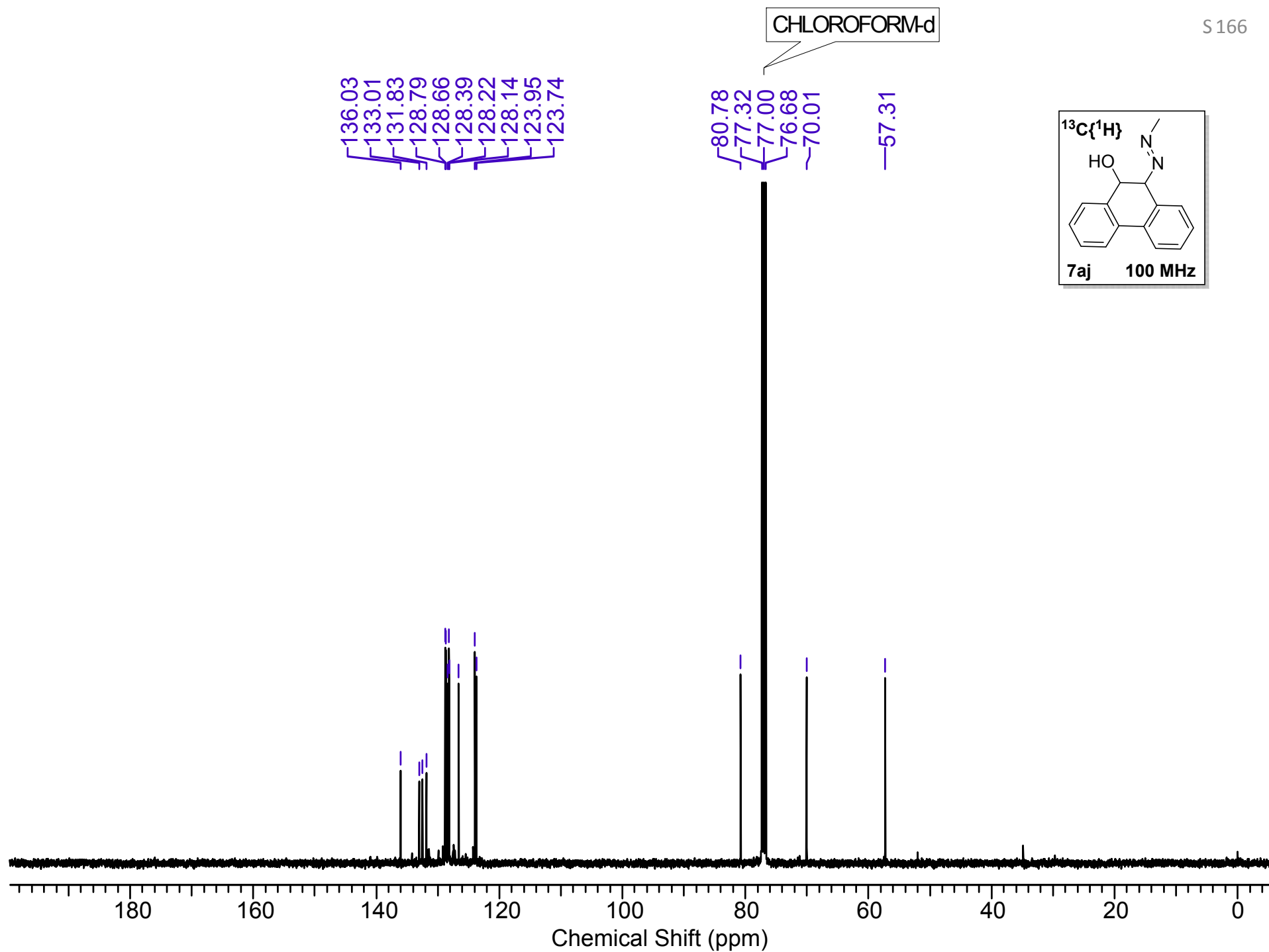
PSD 925

IISER PUNE

1: TOF MS ES+
7.22e4PSD 925 194 (3.543) AM2 (Ar,20000.0,556.28,0.00,LS 3); Sm (SG,
310.00)CALCULATED MASS
(M+H)+ 333.1061

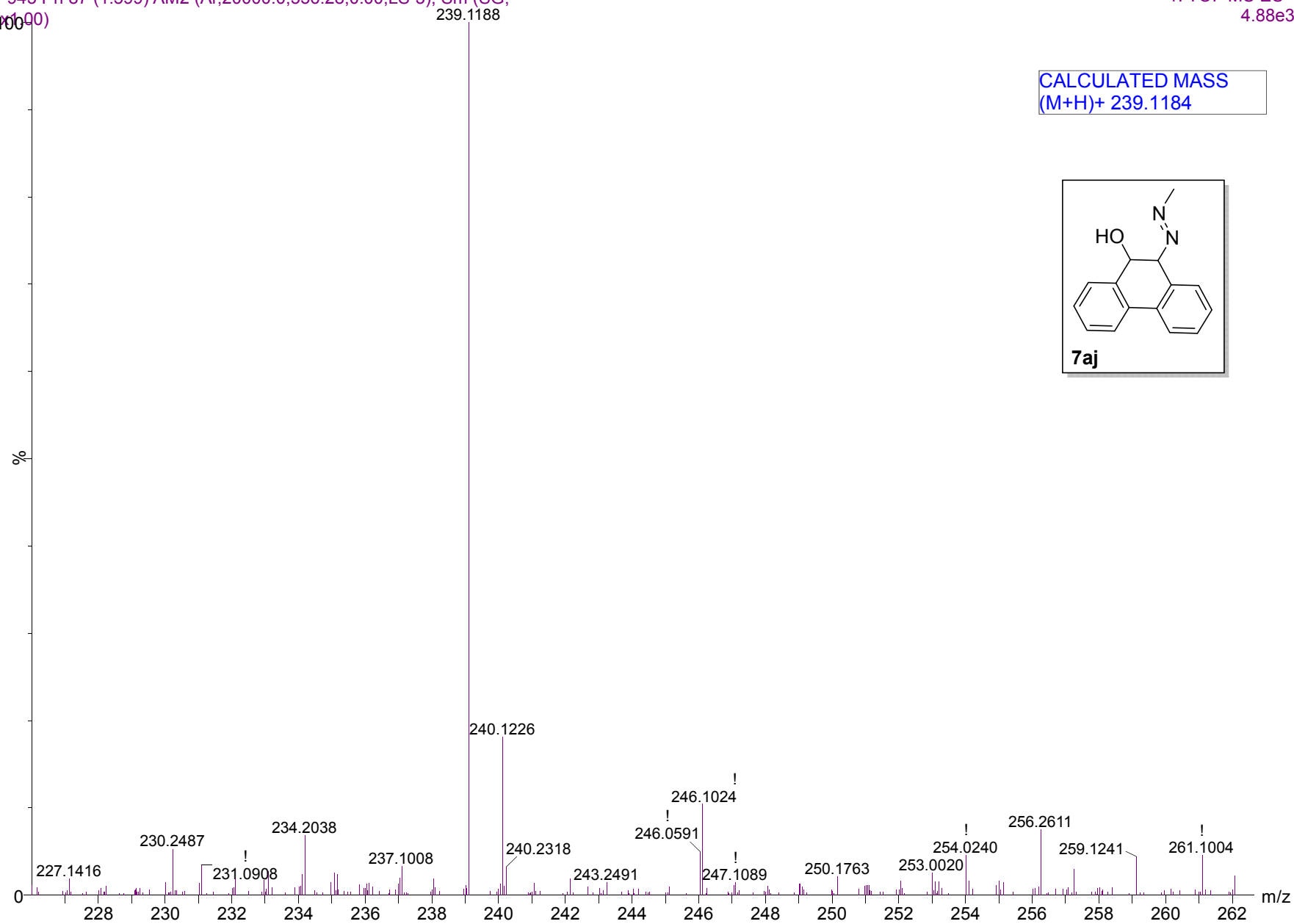
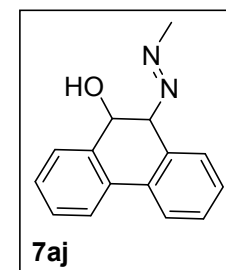


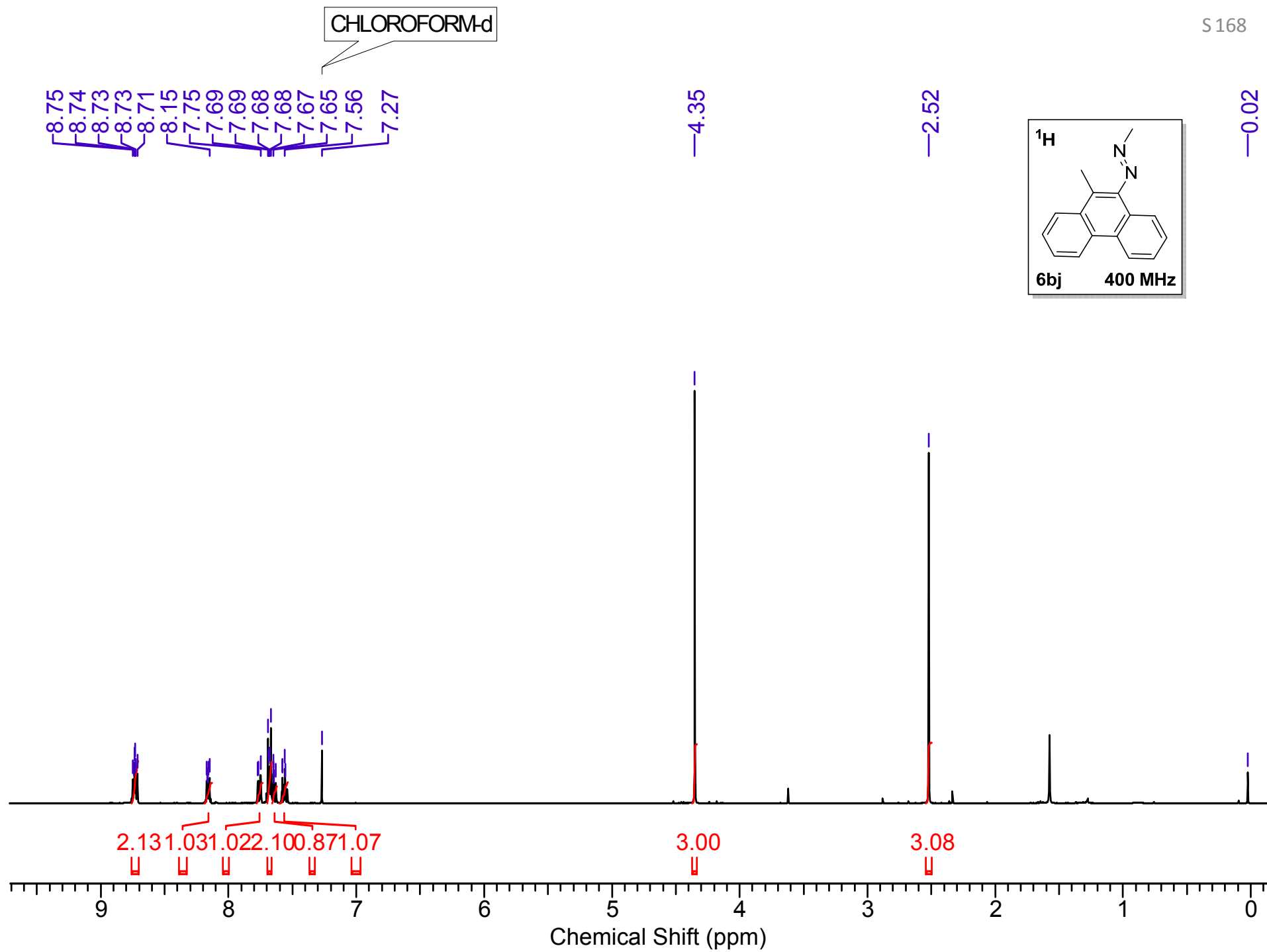


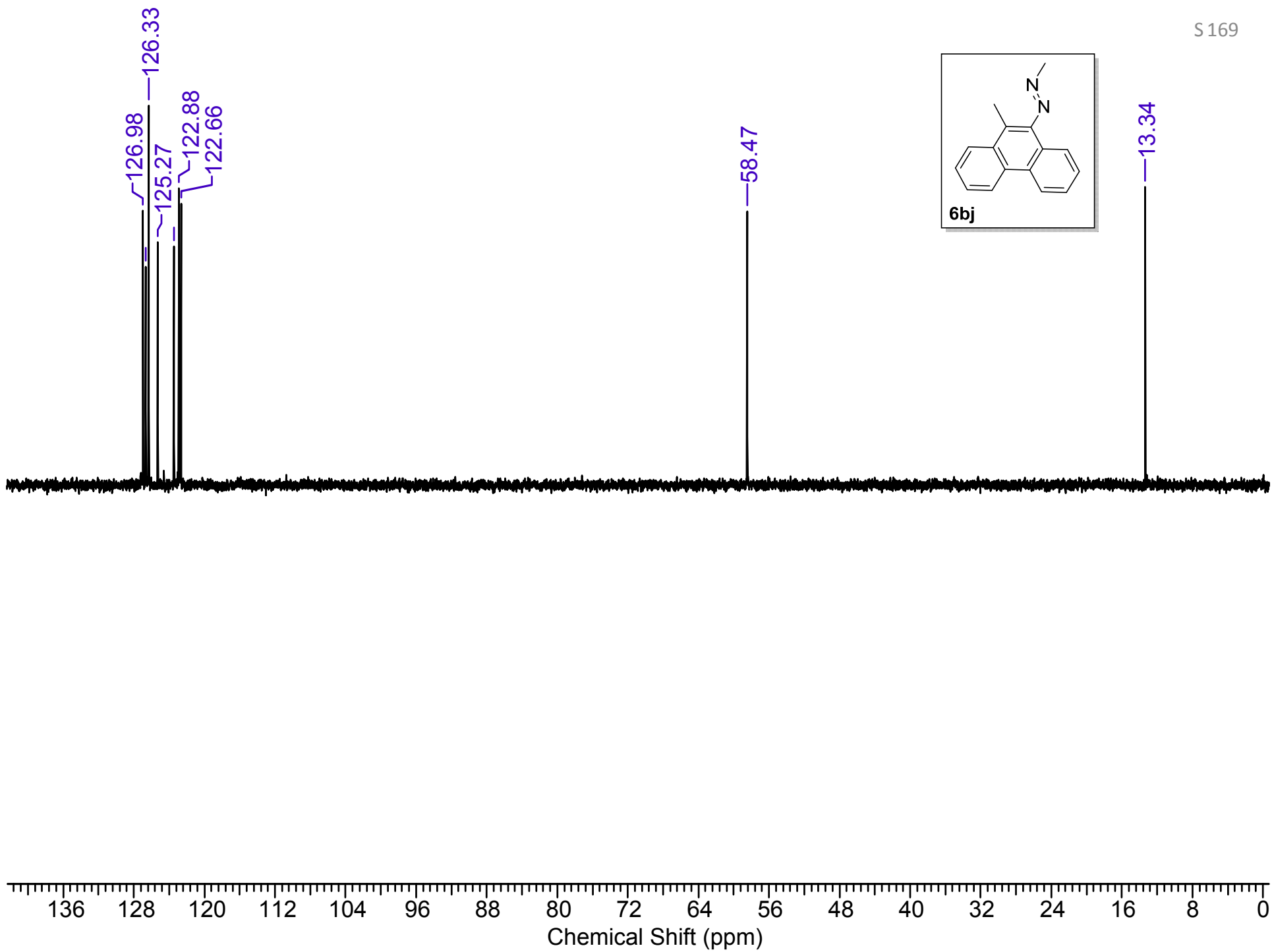


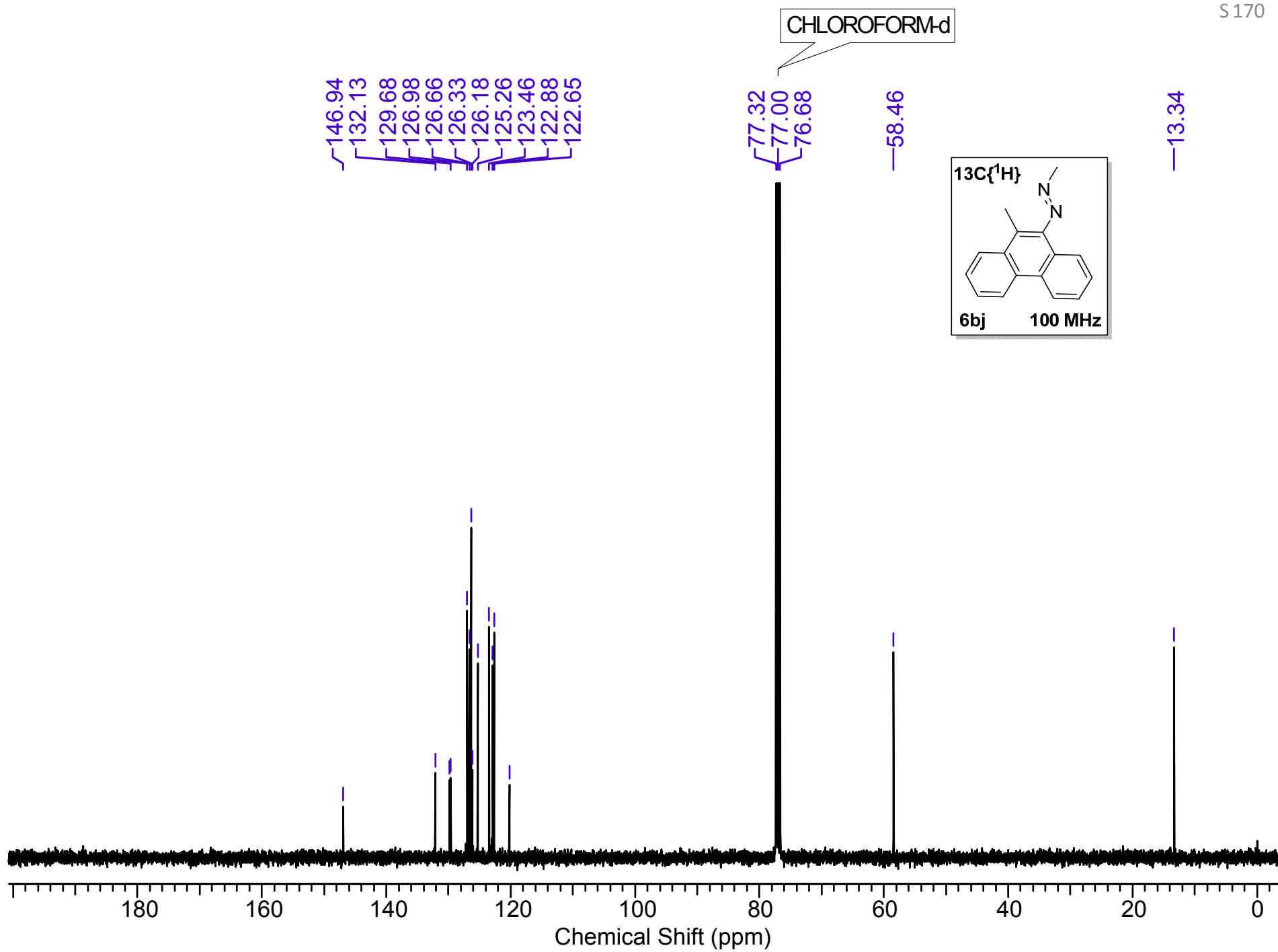
Y 943 Ph

IISER PUNE

1: TOF MS ES+
4.88e3Y 943 Ph 87 (1.599) AM2 (Ar,20000.0,556.28,0.00,LS 3); Sm (SG,
300.00)CALCULATED MASS
(M+H)⁺ 239.1184

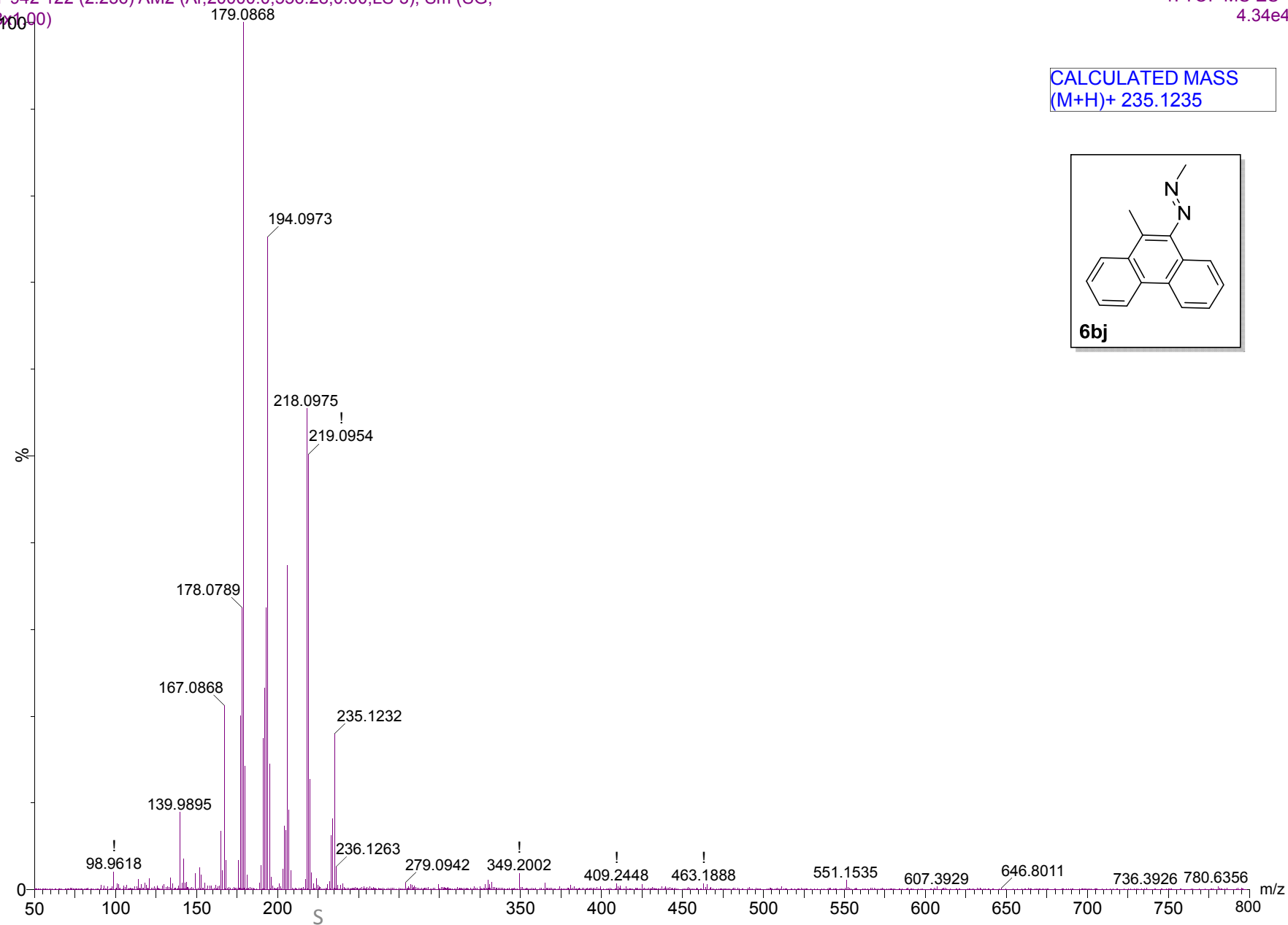
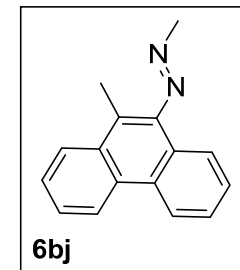






Y 942

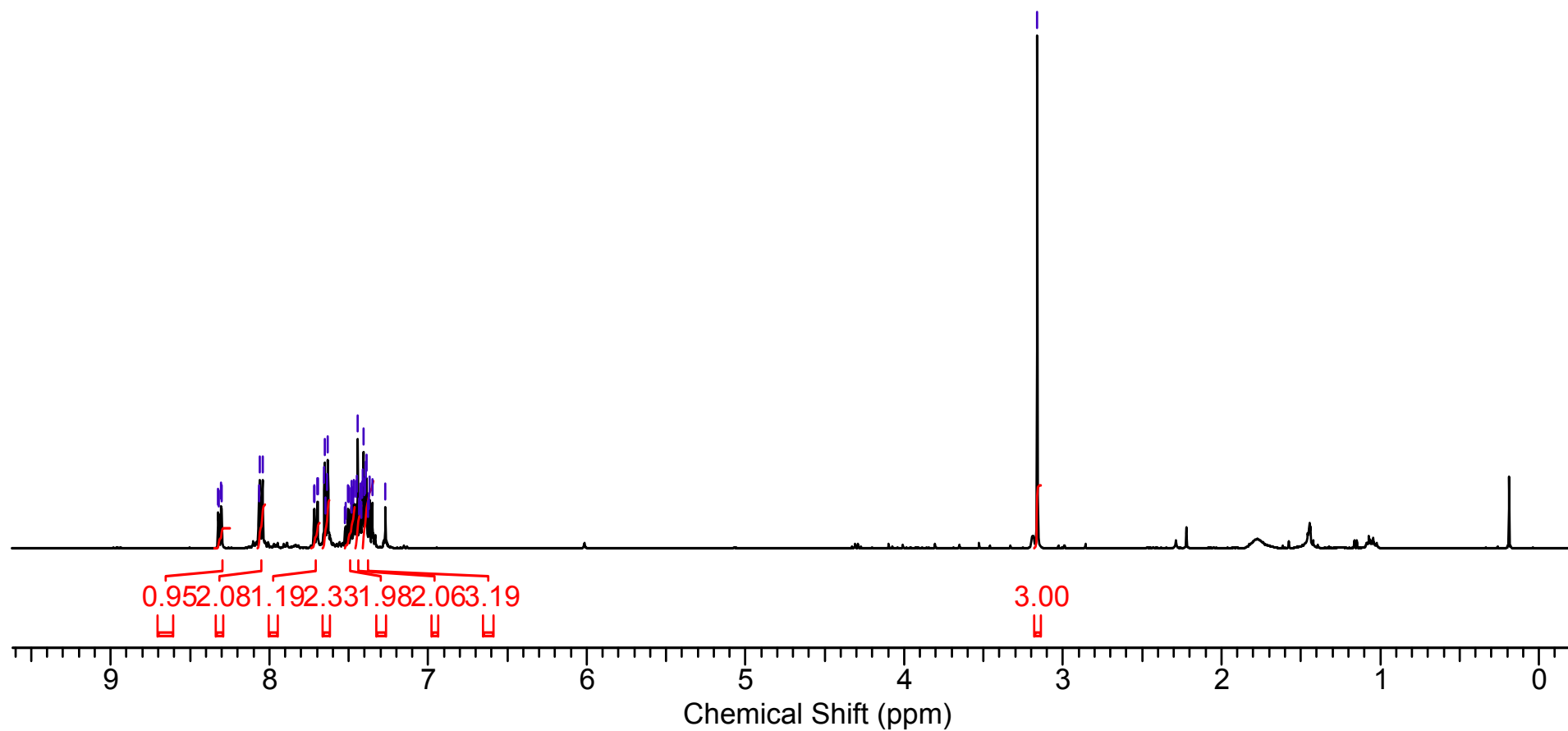
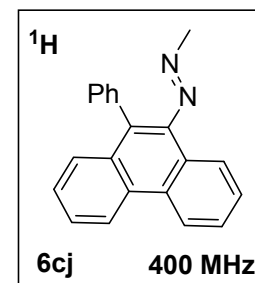
IISER PUNE

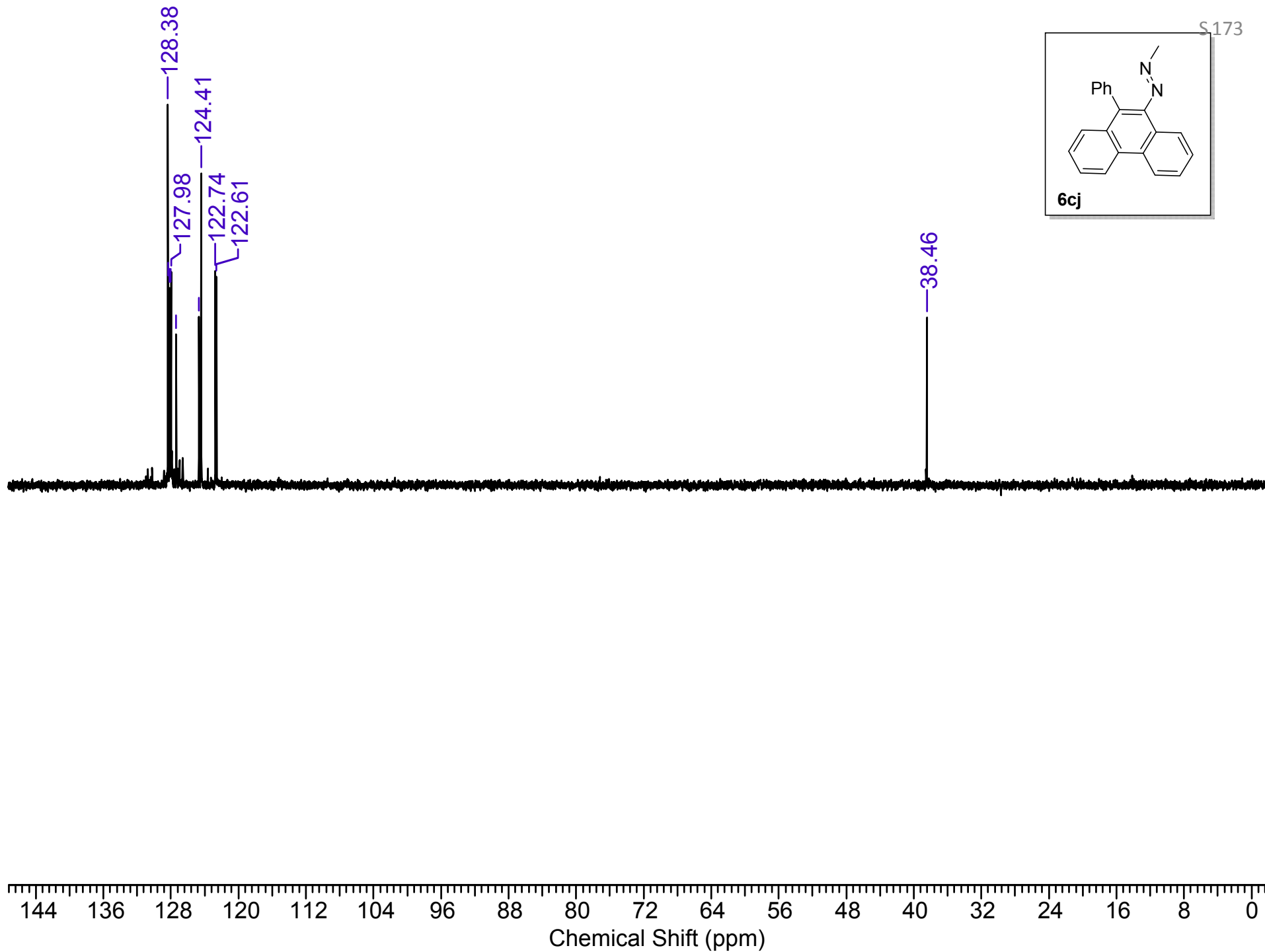
1: TOF MS ES+
4.34e4Y 942 122 (2.236) AM2 (Ar,20000.0,556.28,0.00,LS 3); Sm (SG,
300.00)CALCULATED MASS
(M+H)+ 235.1235

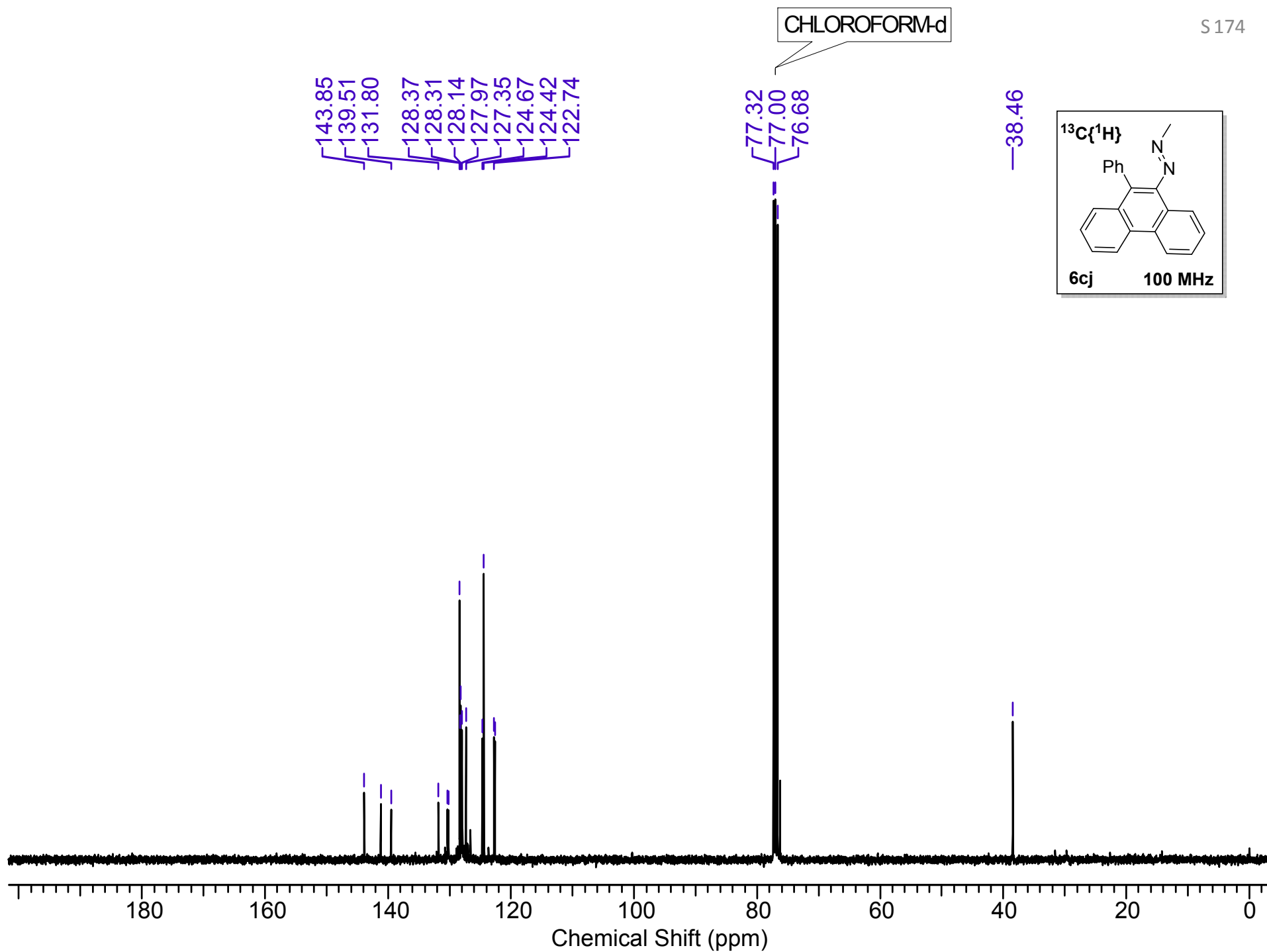
CHLOROFORM-d

8.30
8.06
8.04
7.65
7.65
7.63
7.44
7.41
7.41
7.40
7.39
7.39
7.27

—3.16



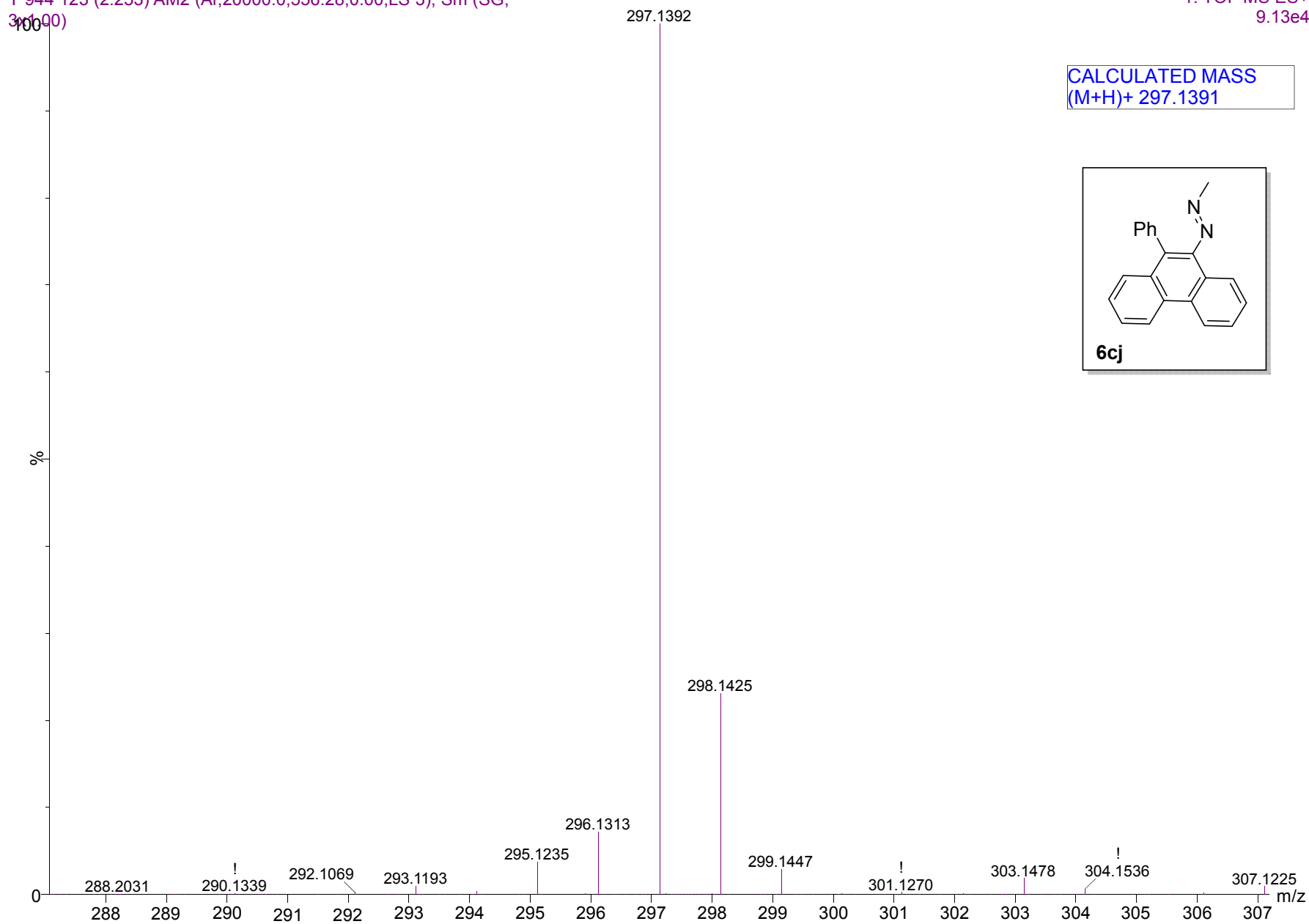




Y 944

Y 944 123 (2.253) AM2 (Ar,20000.0,556.28,0.00,LS 3); Sm (SG,
30000)

IISER PUNE

1: TOF MS ES+
9.13e4

CHLOROFORM-d

Water

S 176

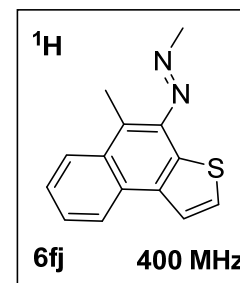
8.43
8.41
8.37
8.35
8.00
7.99
7.71
7.66
7.64
7.63
7.61
7.60
7.27

4.26

3.26

1.56

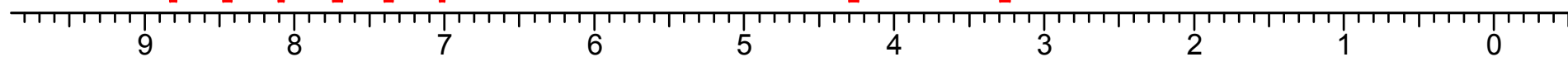
0.02



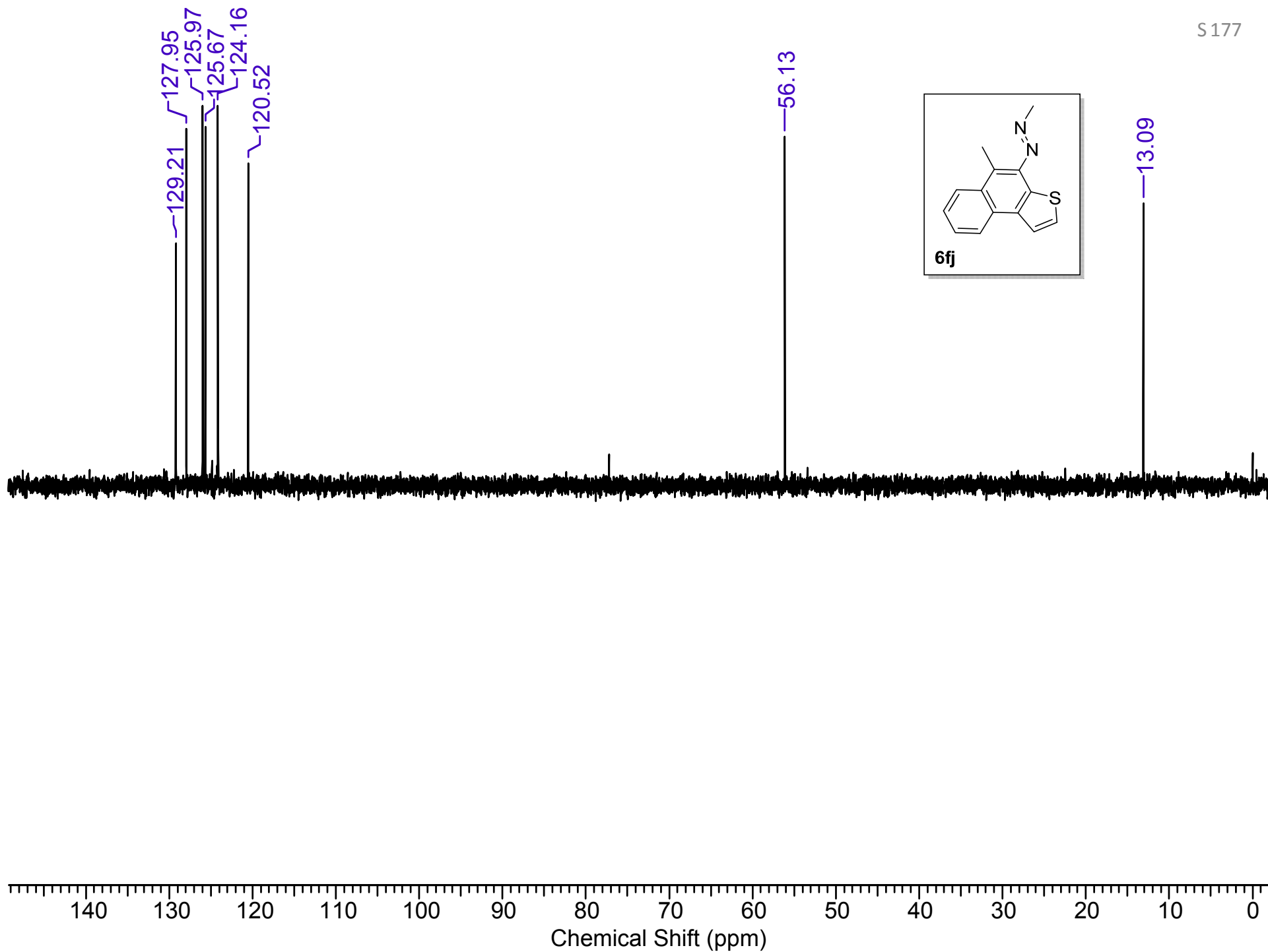
0.98 1.06 1.00 1.01 1.16 0.98

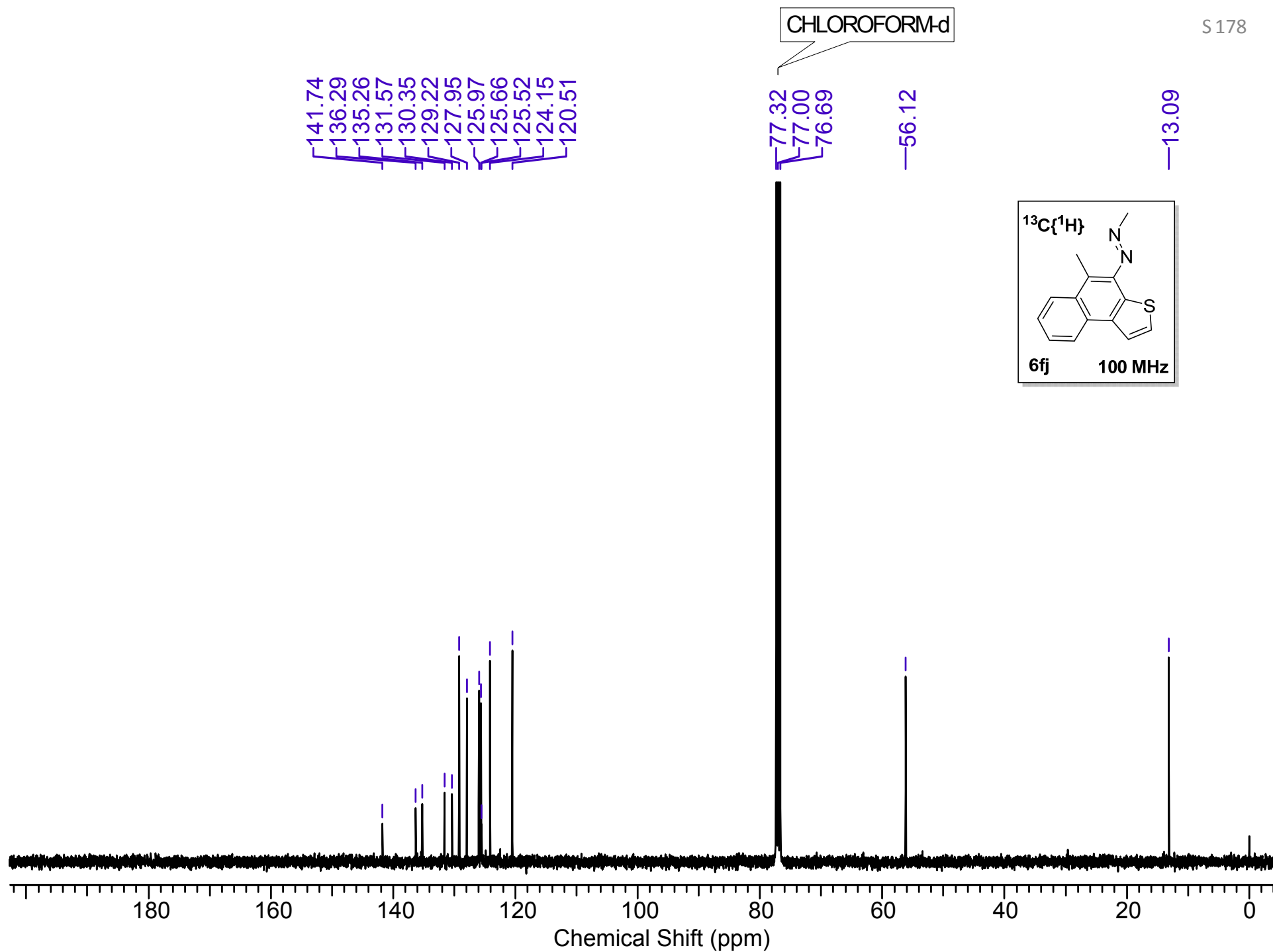
3.03

3.08



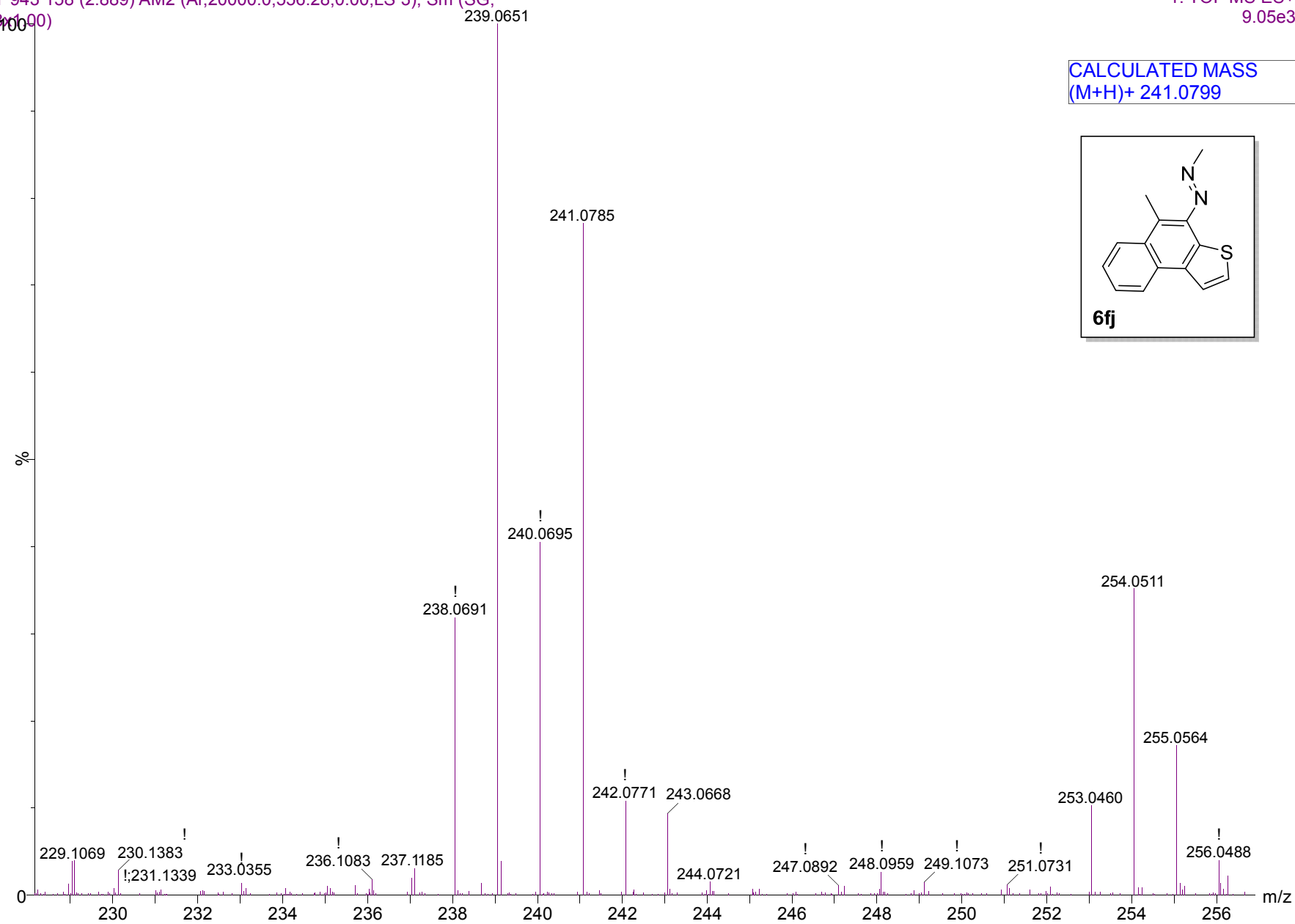
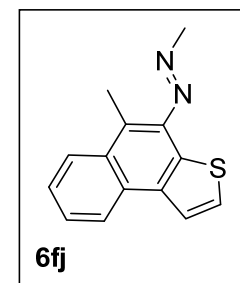
Chemical Shift (ppm)



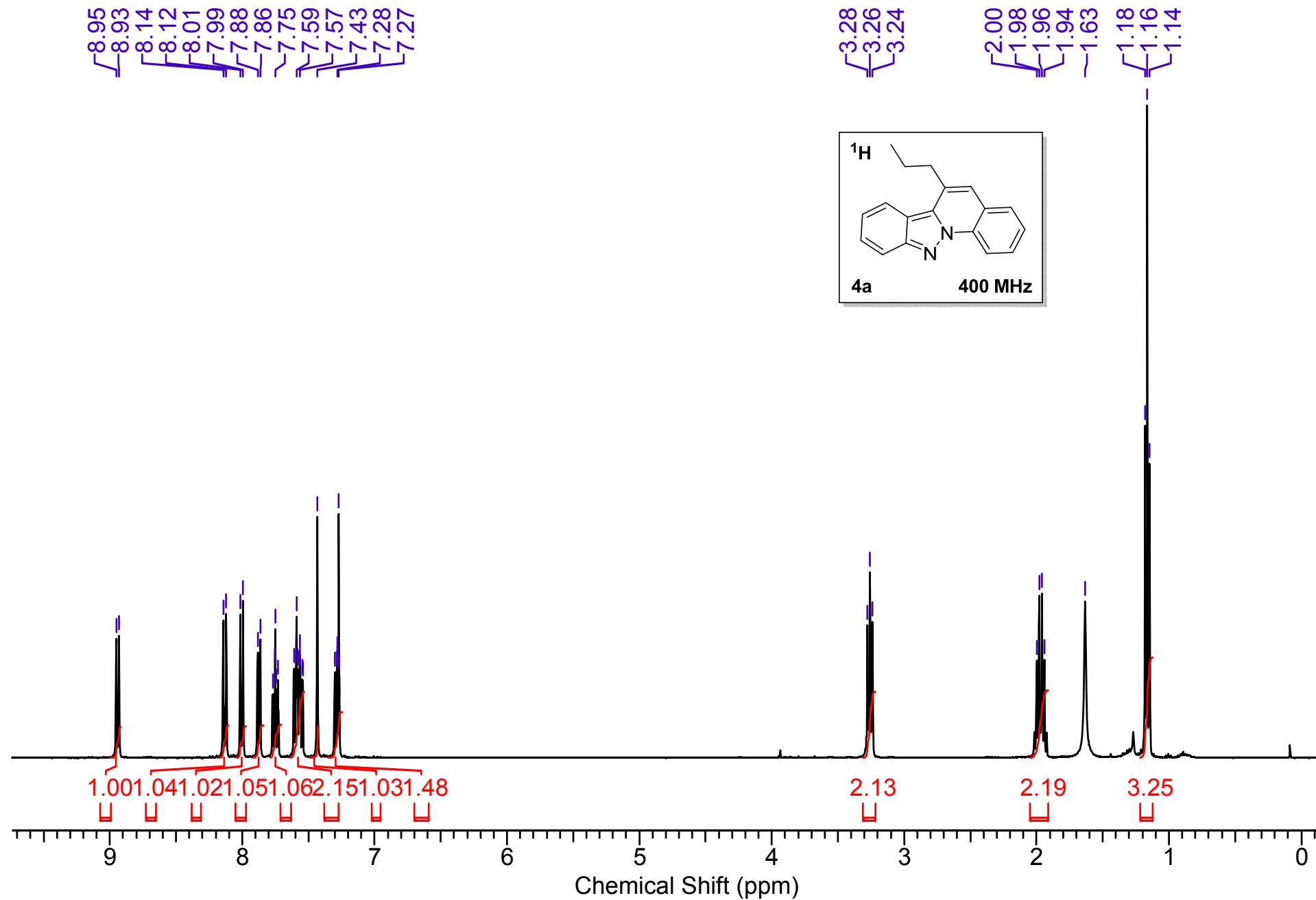


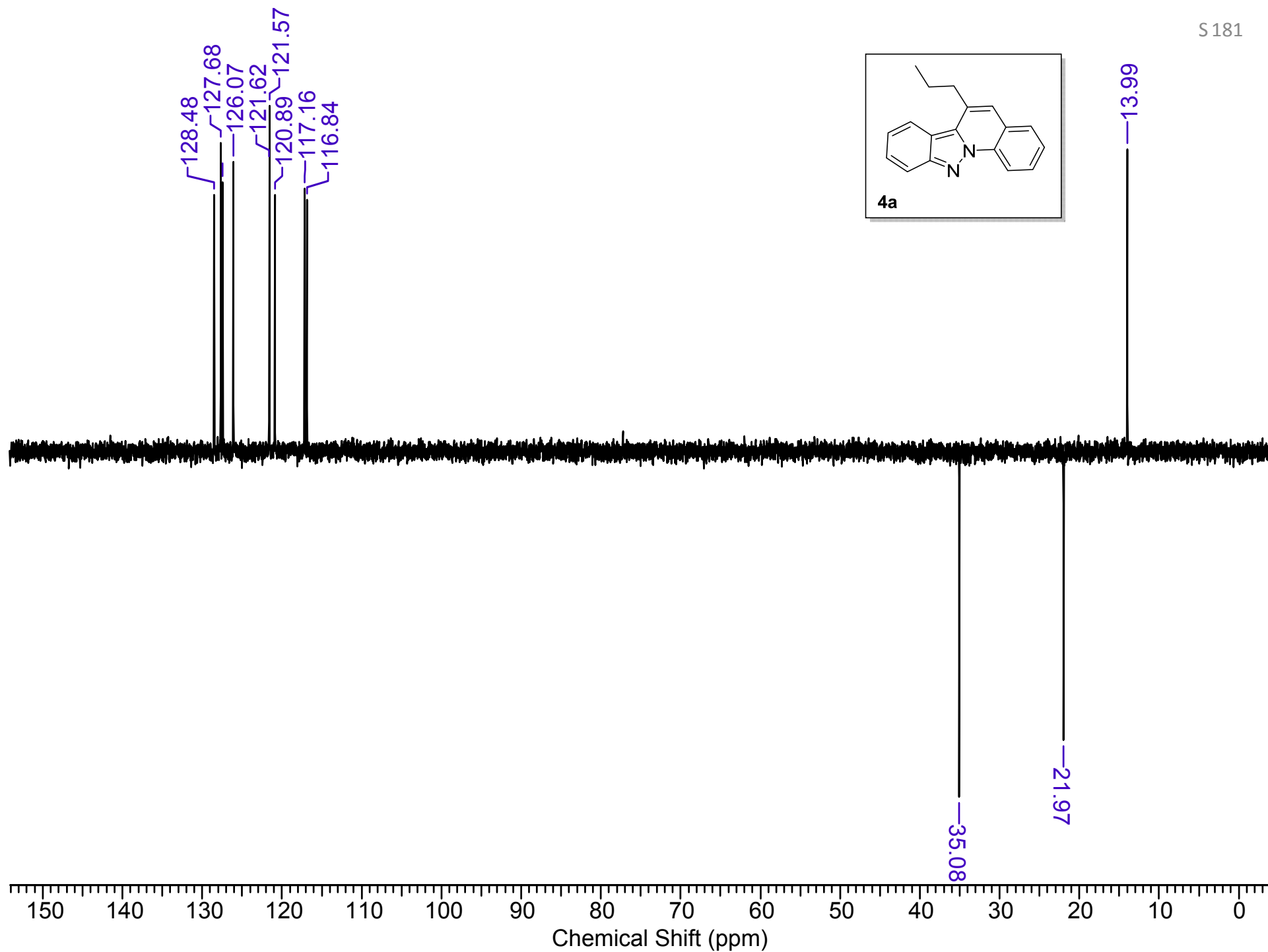
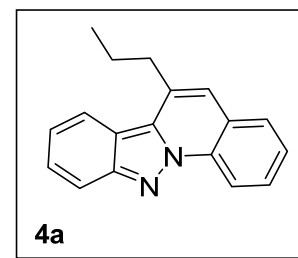
Y 943

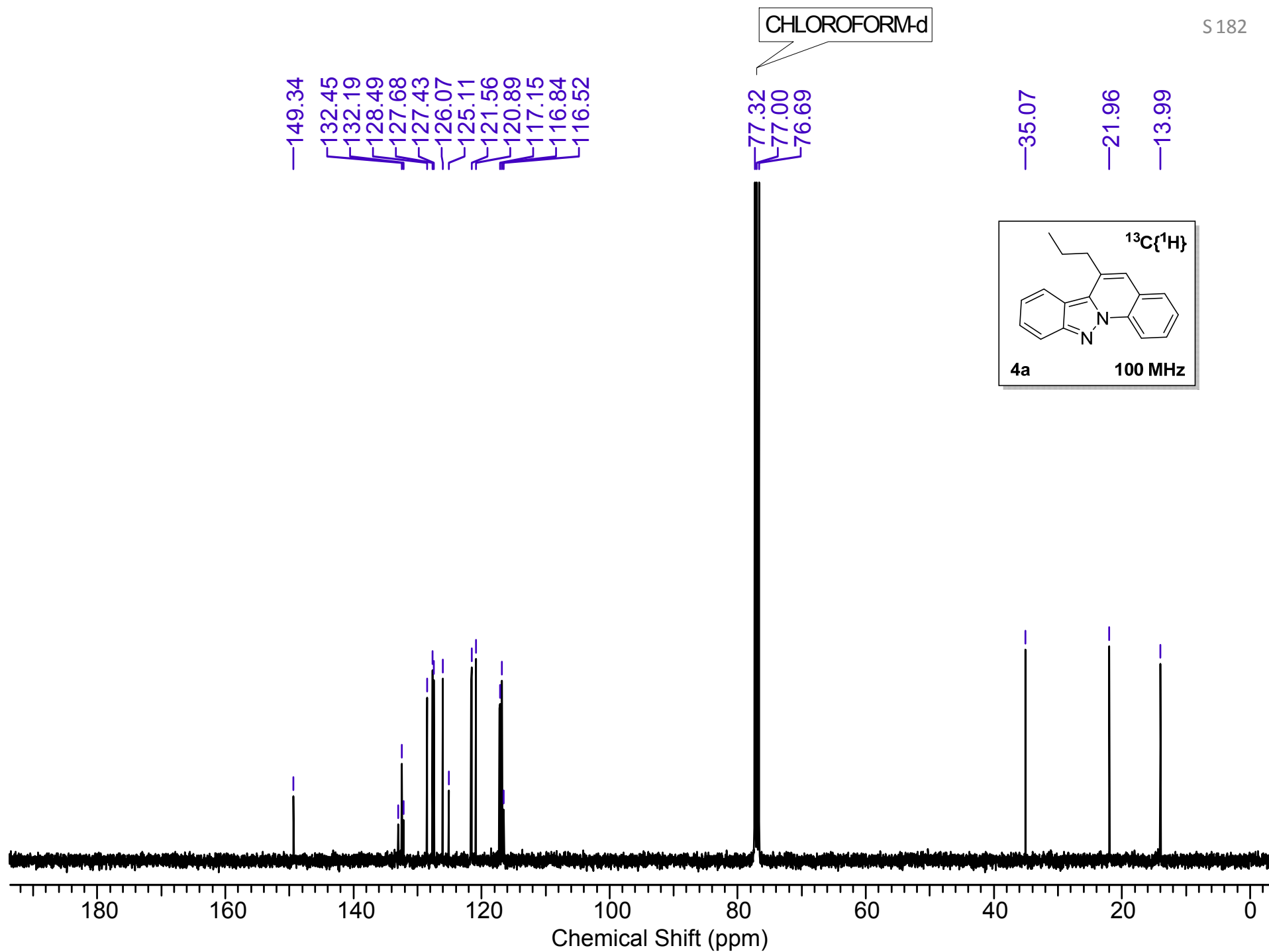
IISER PUNE

1: TOF MS ES+
9.05e3Y 943 158 (2.889) AM2 (Ar,20000.0,556.28,0.00,LS 3); Sm (SG,
300.00)CALCULATED MASS
(M+H)+ 241.0799

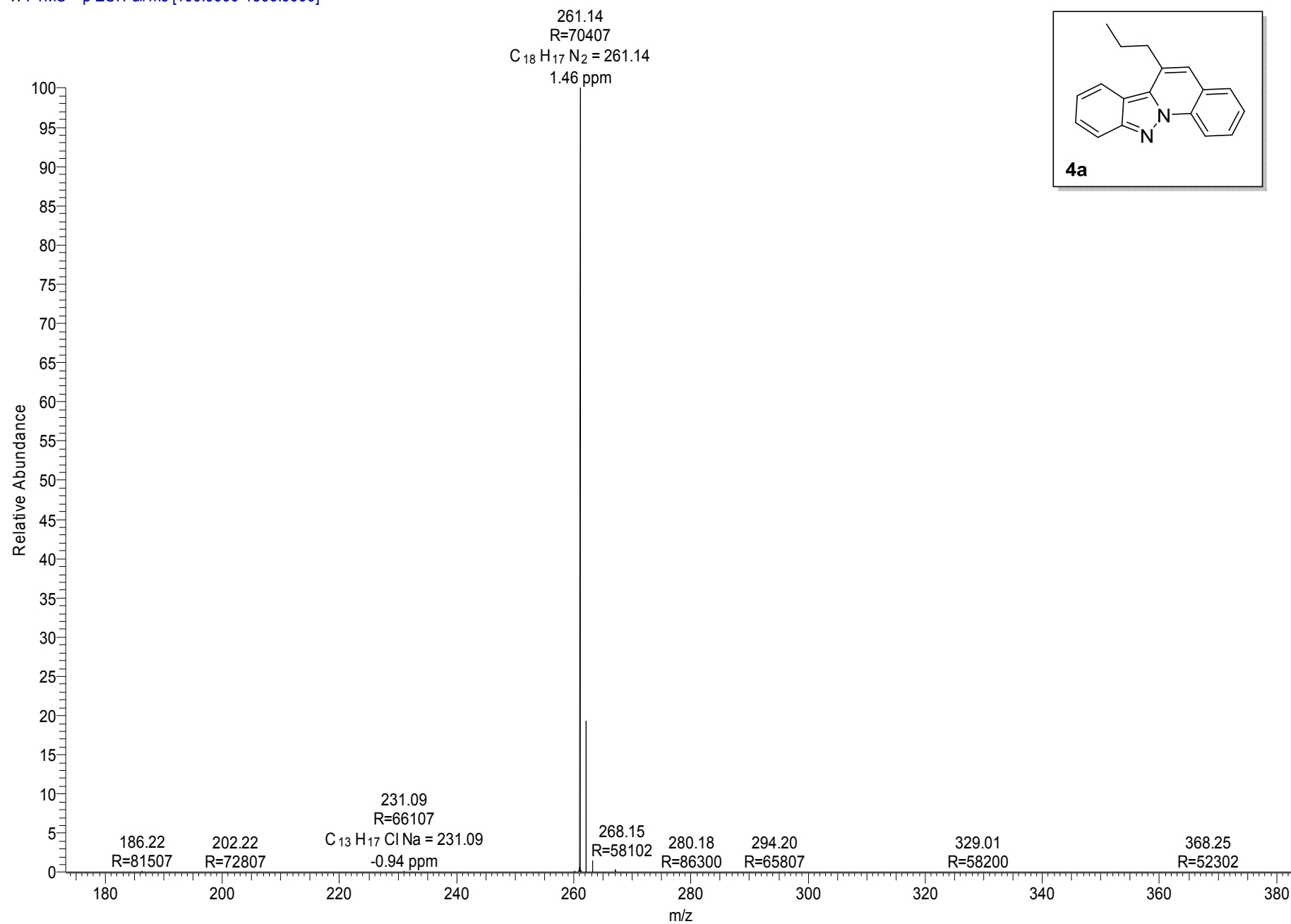
CHLOROFORM-d

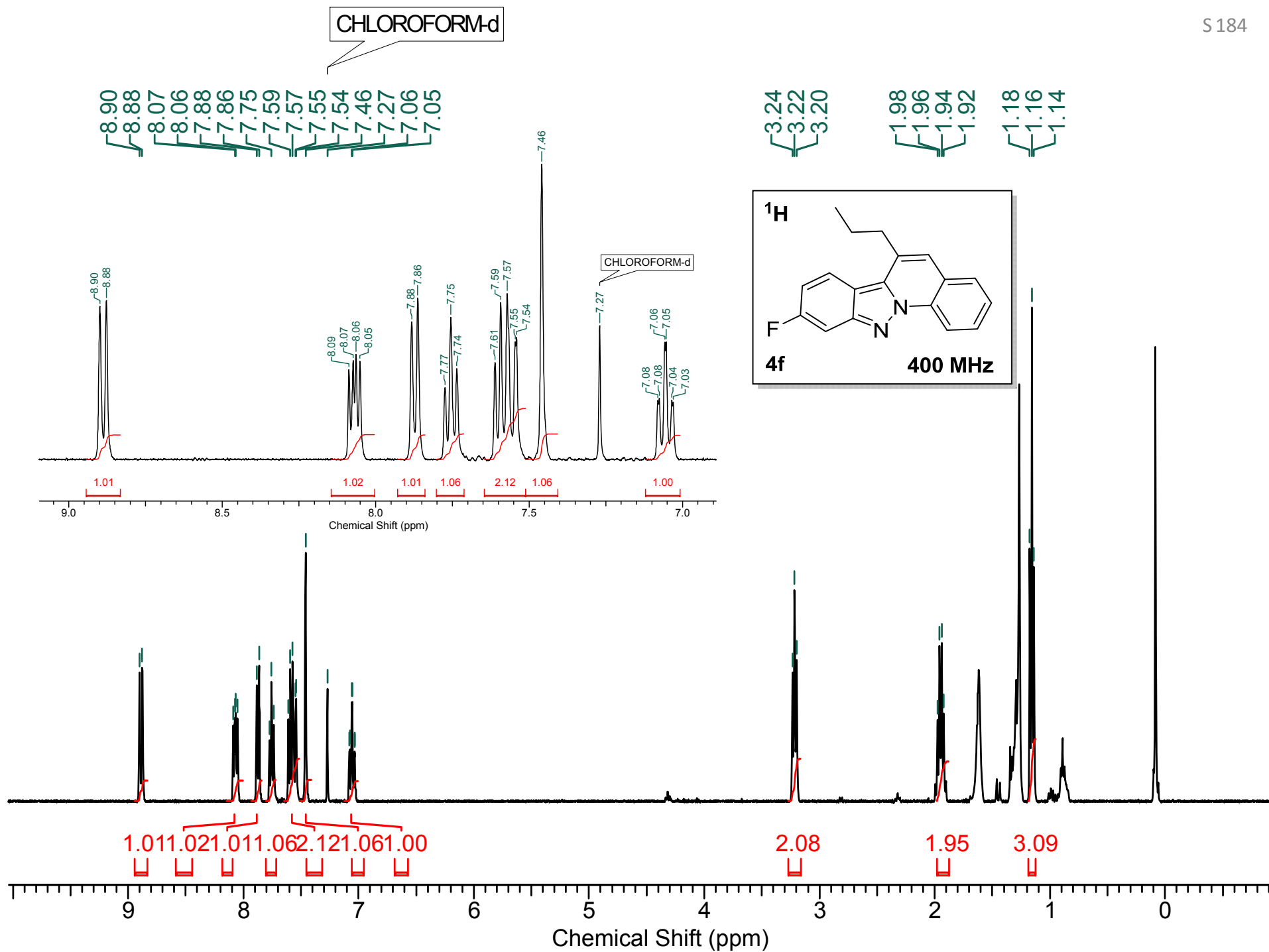


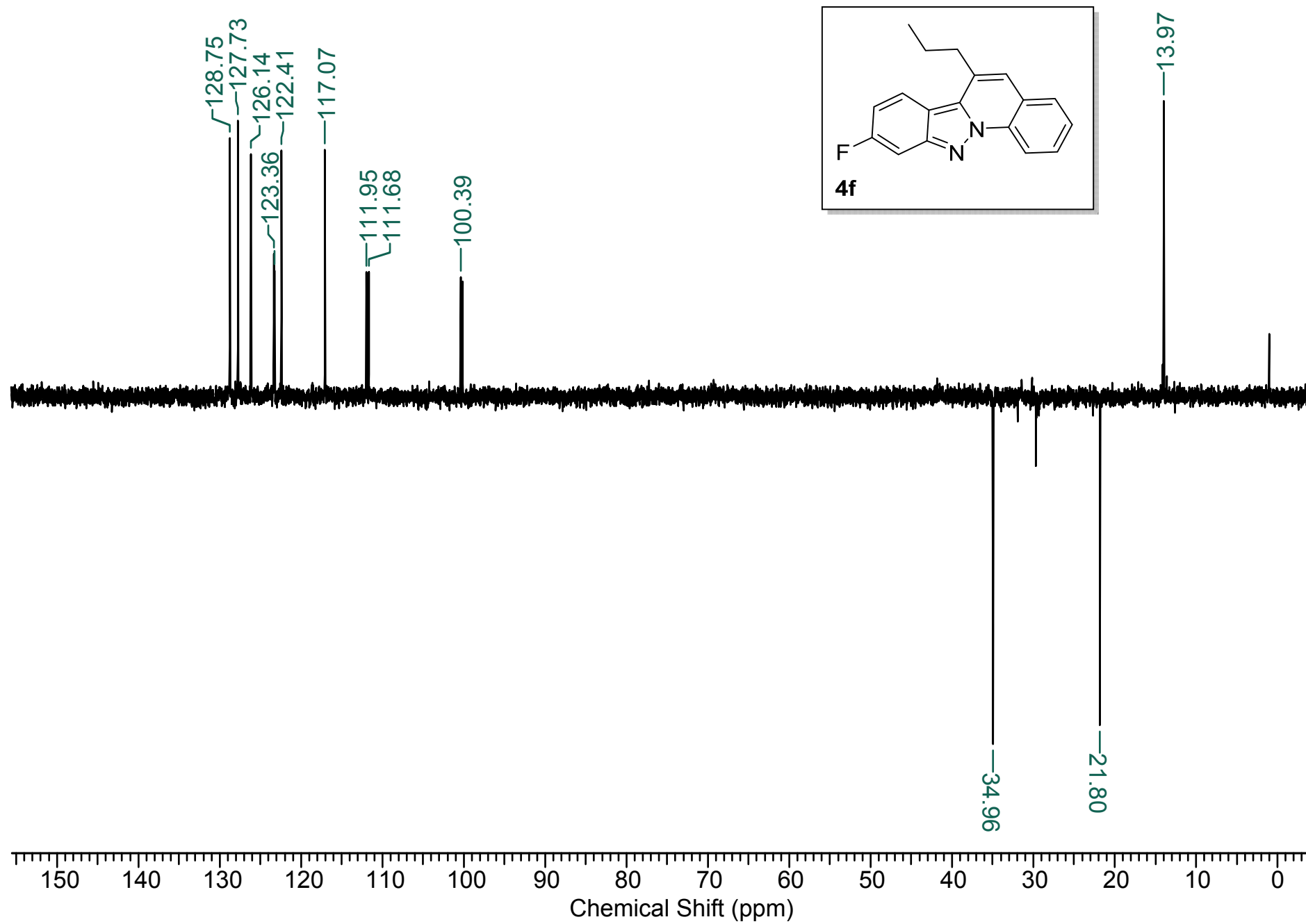


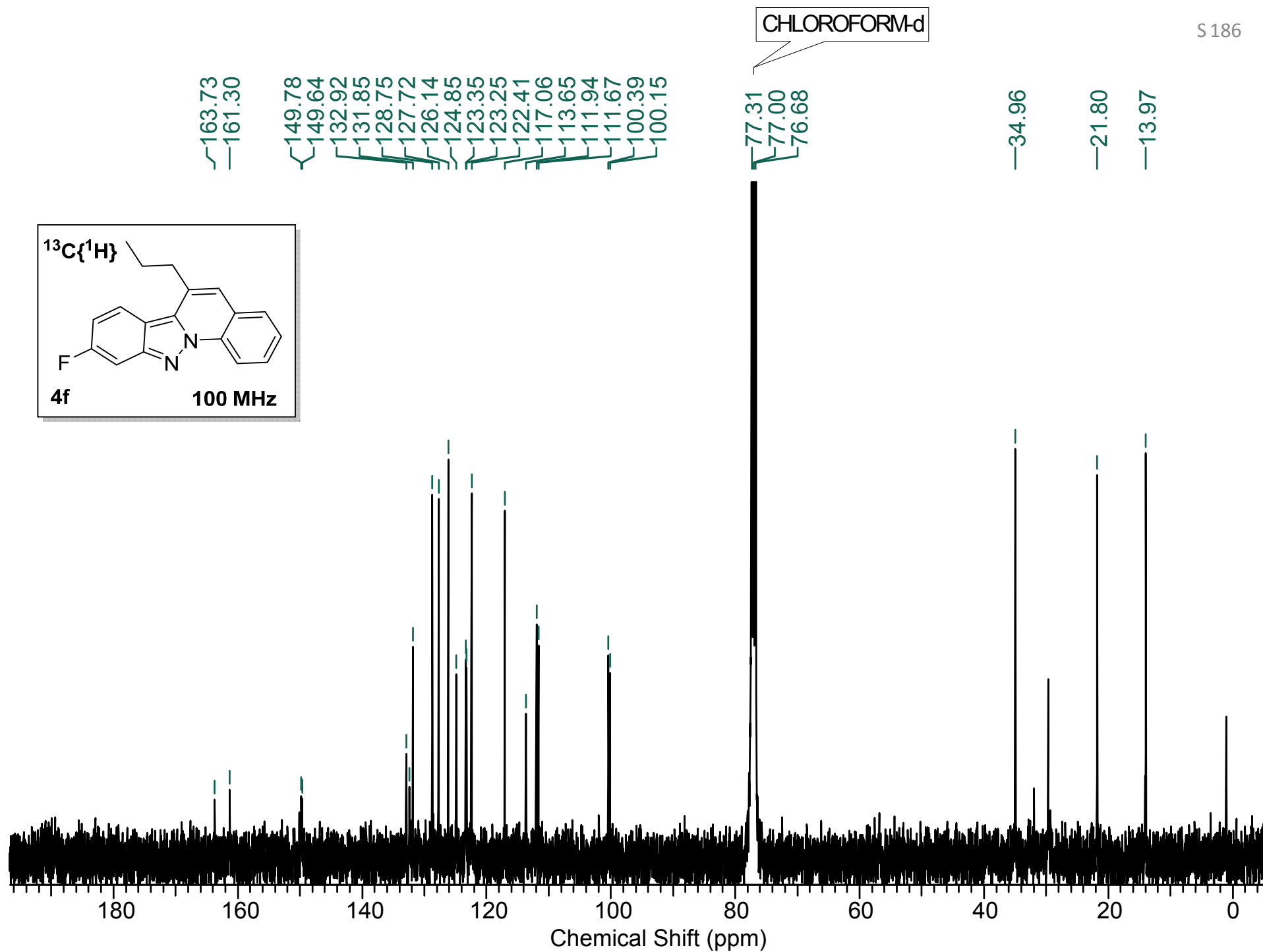


PSD-Fluorescence #491 RT: 4.02 AV: 1 NL: 3.16E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]

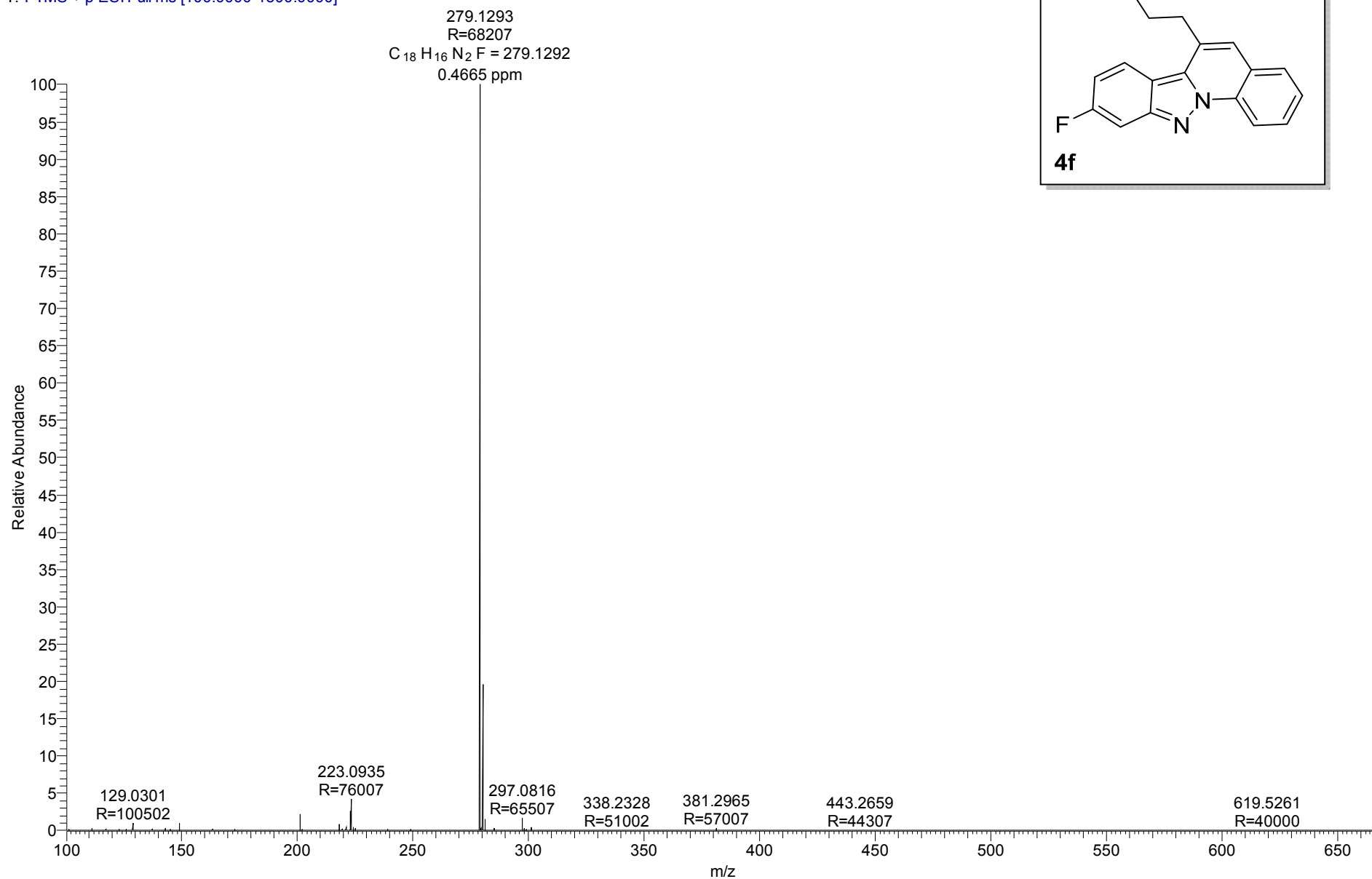


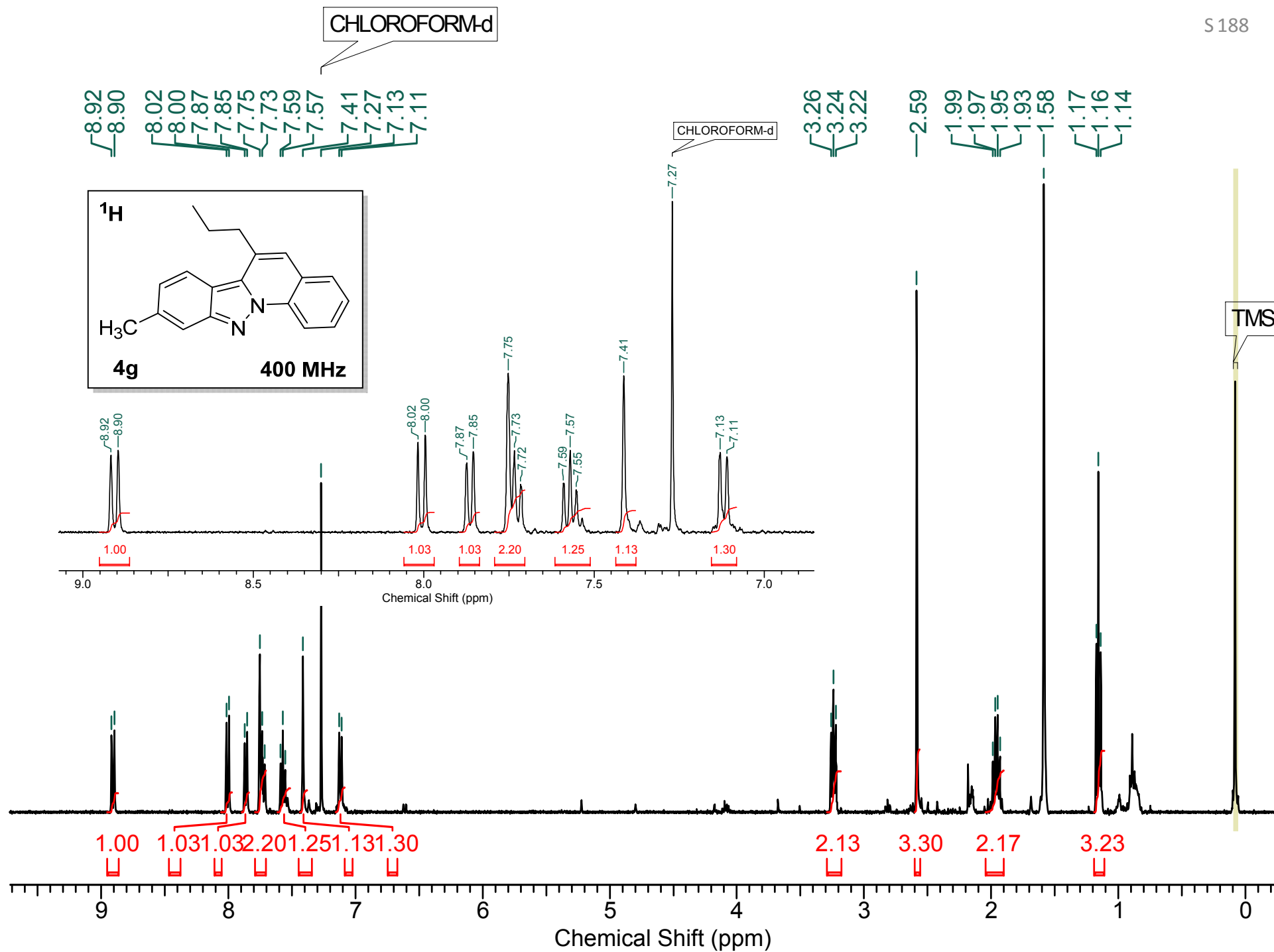


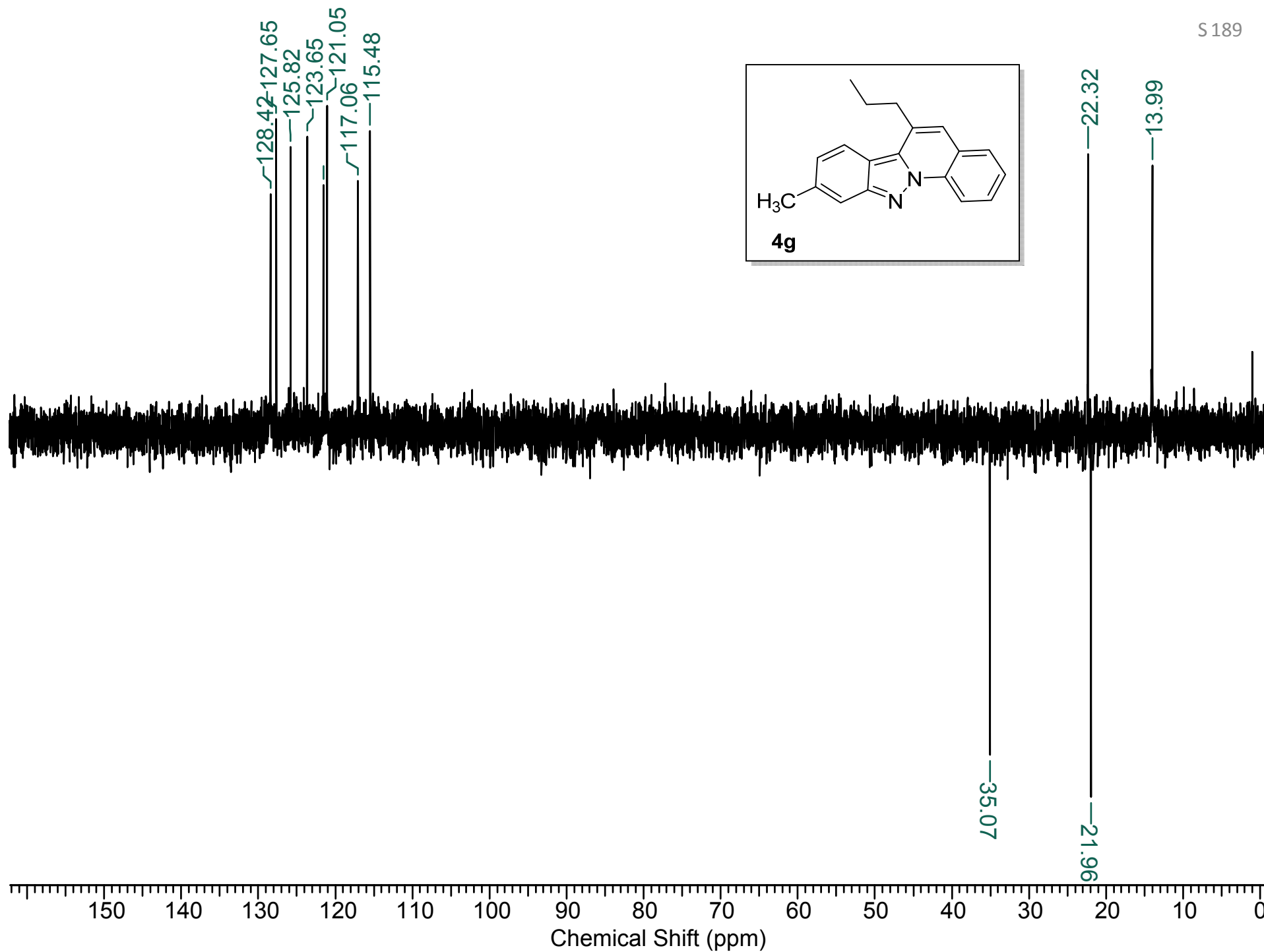


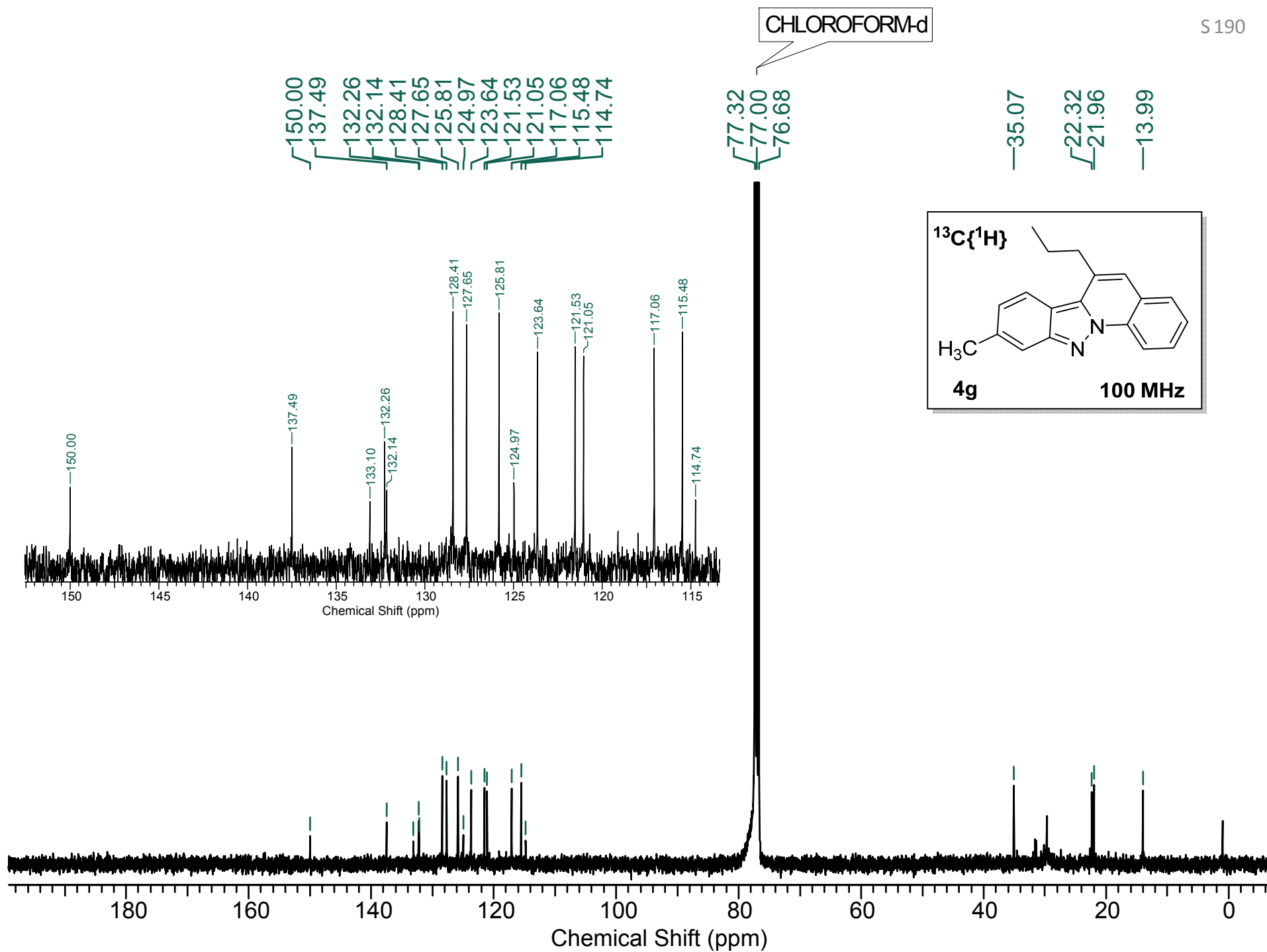


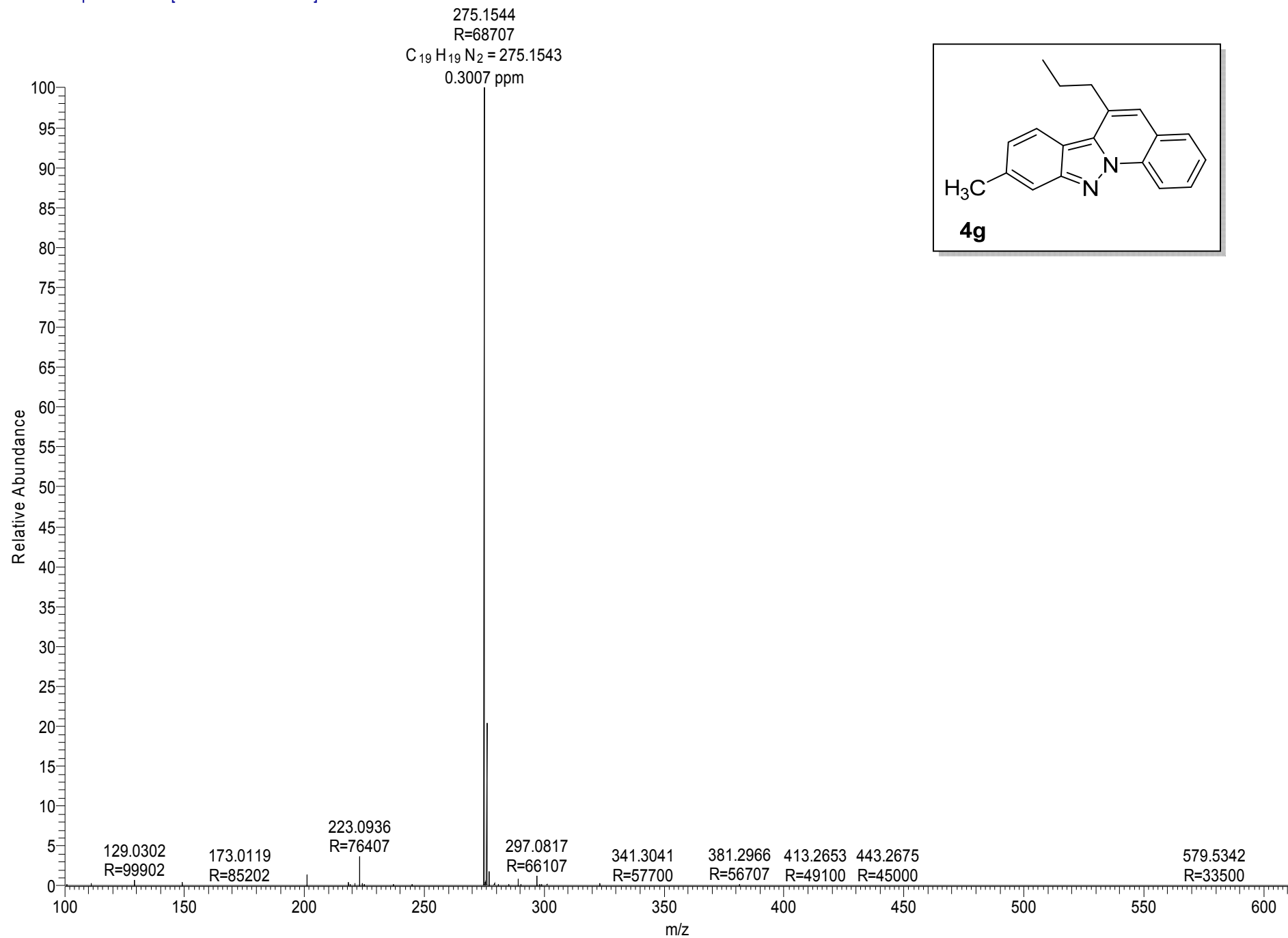
PSD-78K #562 RT: 3.08 AV: 1 NL: 1.15E9
T: FTMS + p ESI Full ms [100.0000-1500.0000]



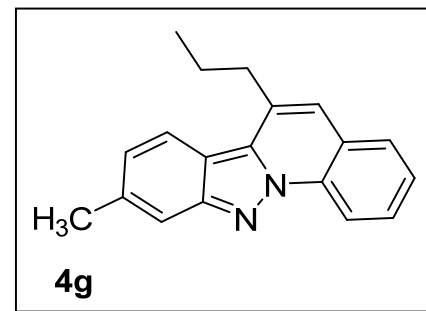


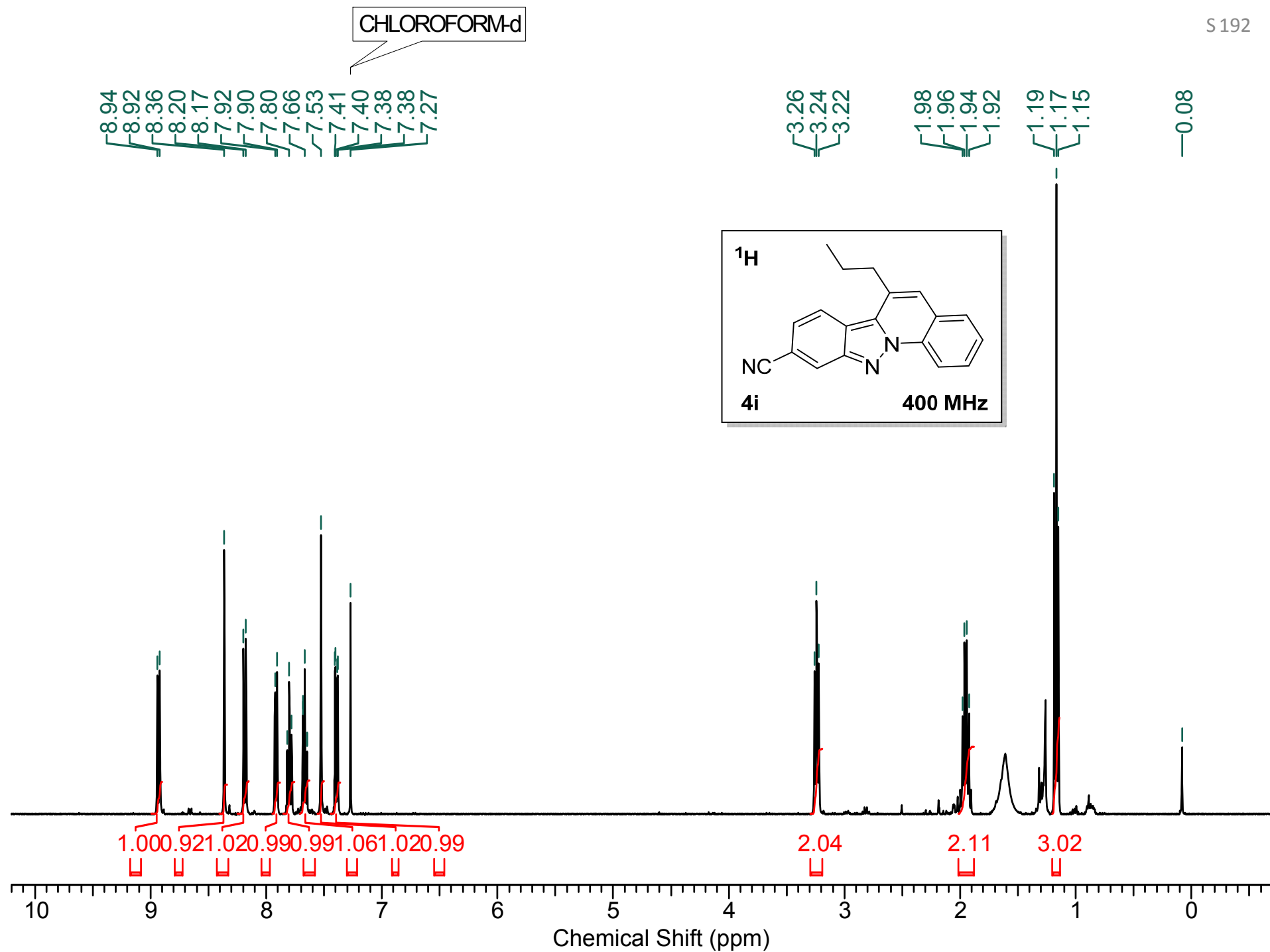


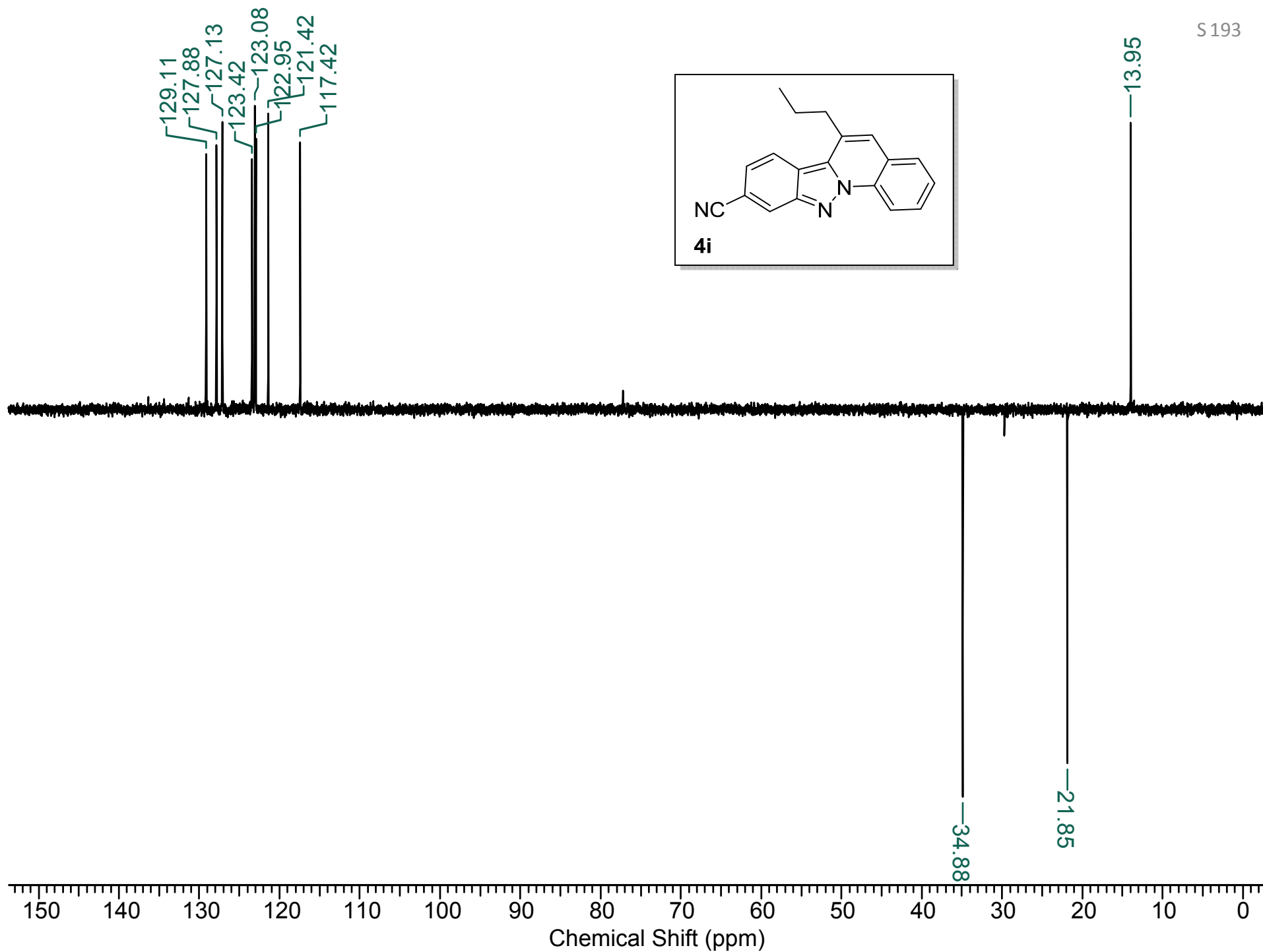




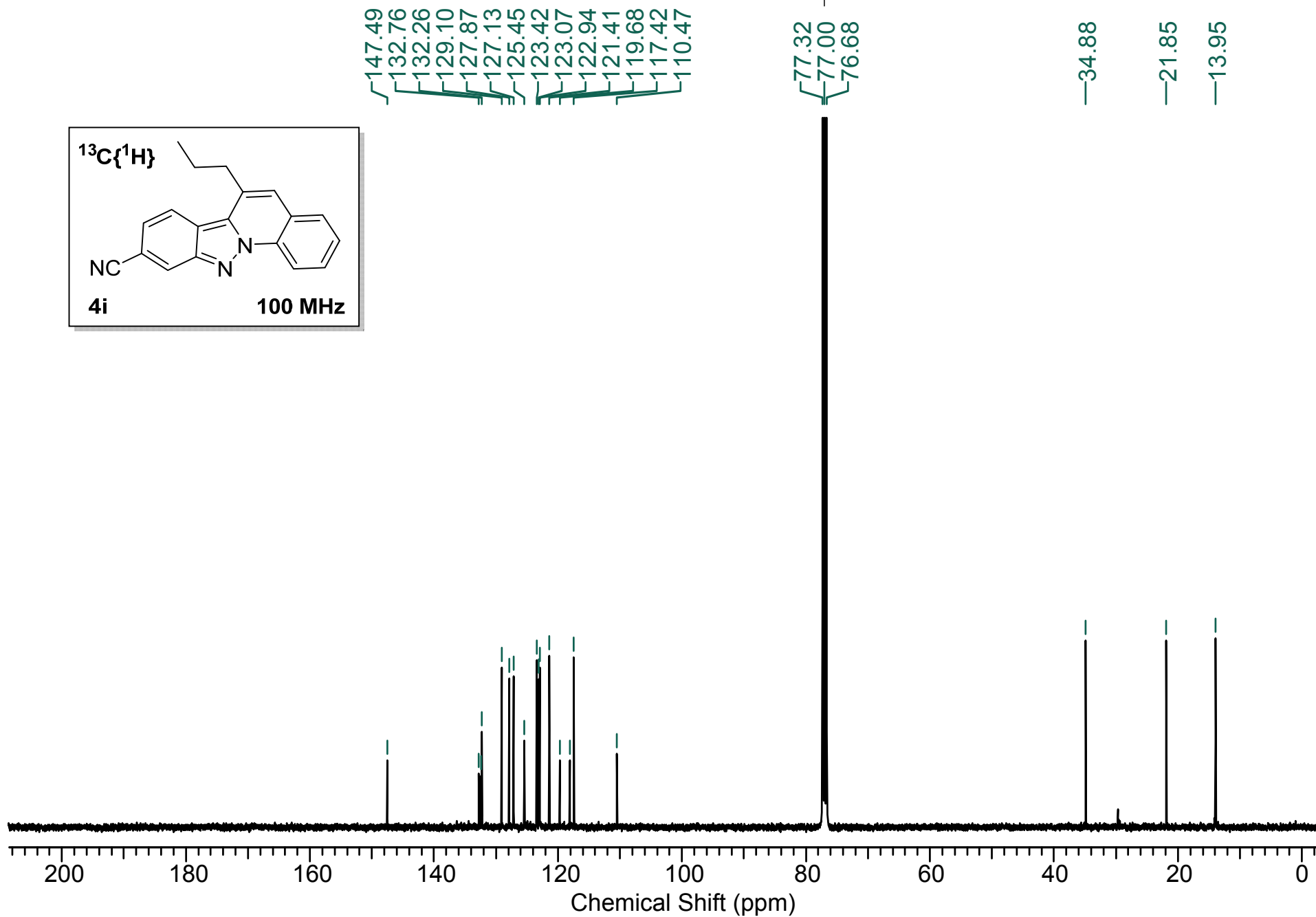
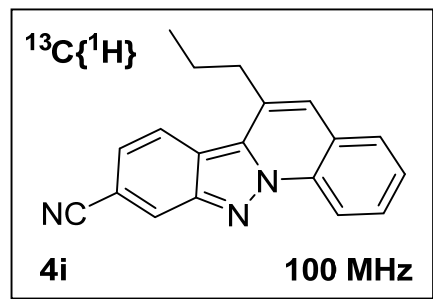
275.1544
R=68707
C₁₉ H₁₉ N₂ = 275.1543
0.3007 ppm

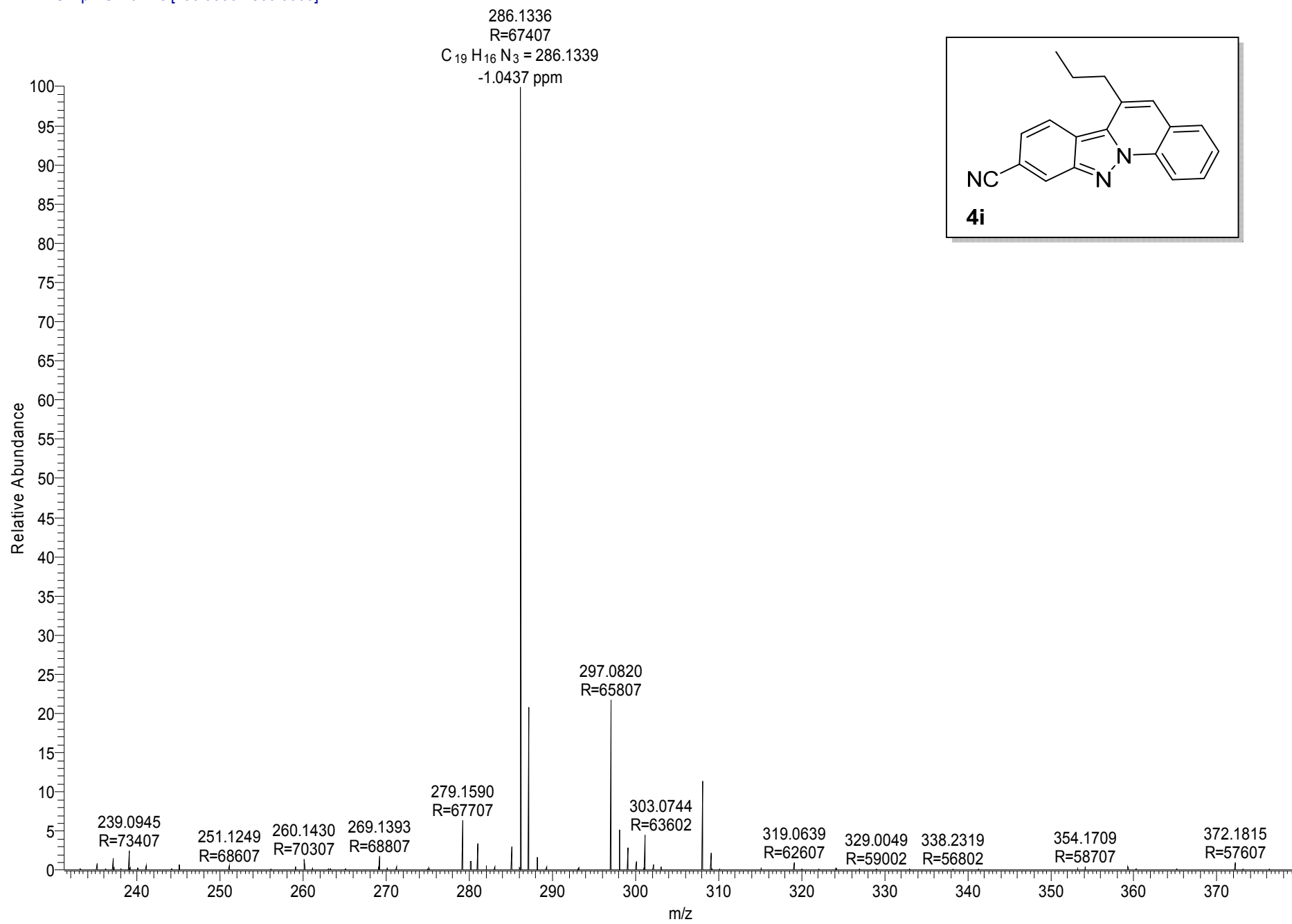
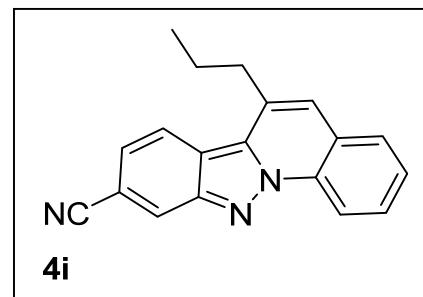


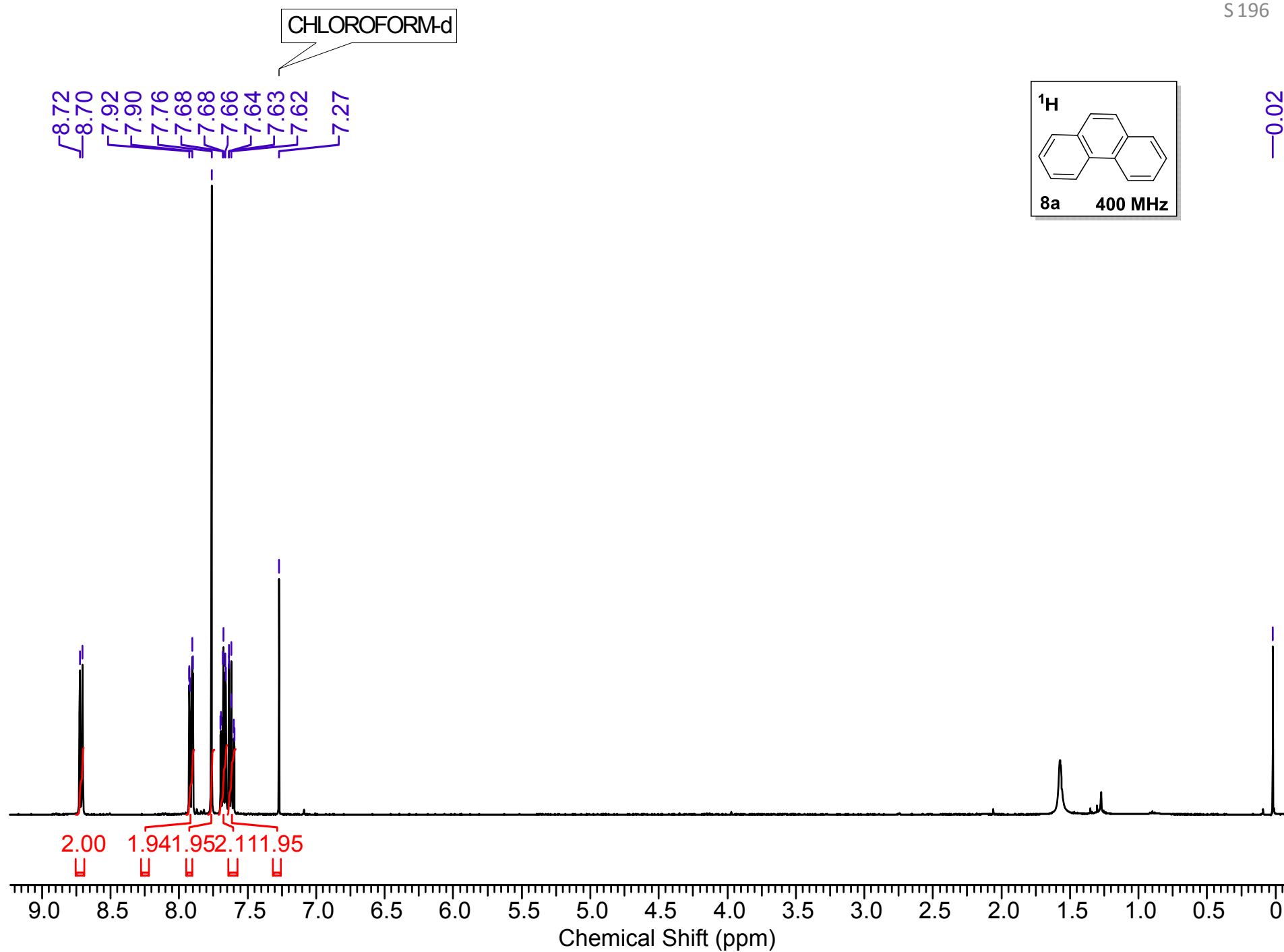


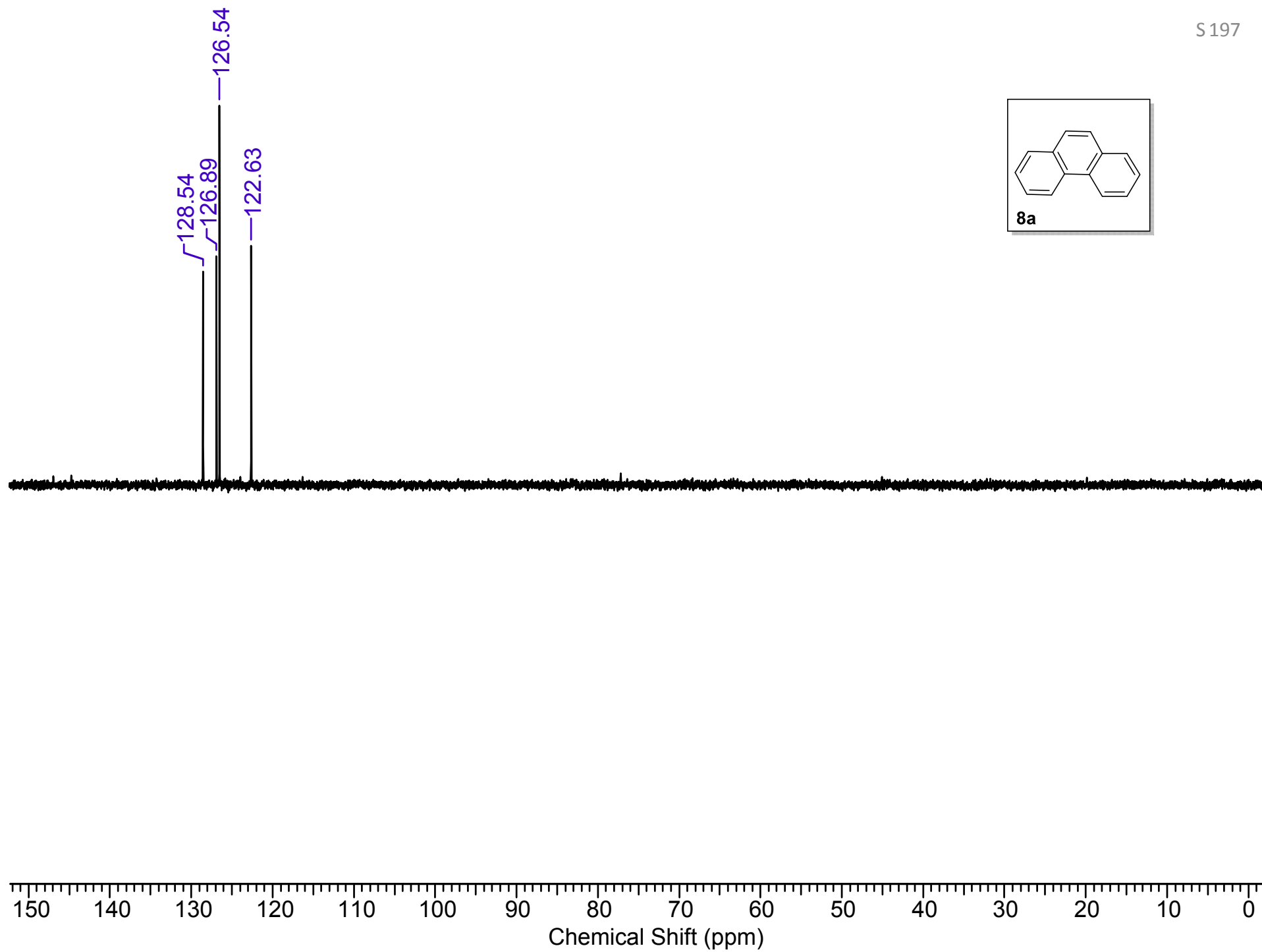


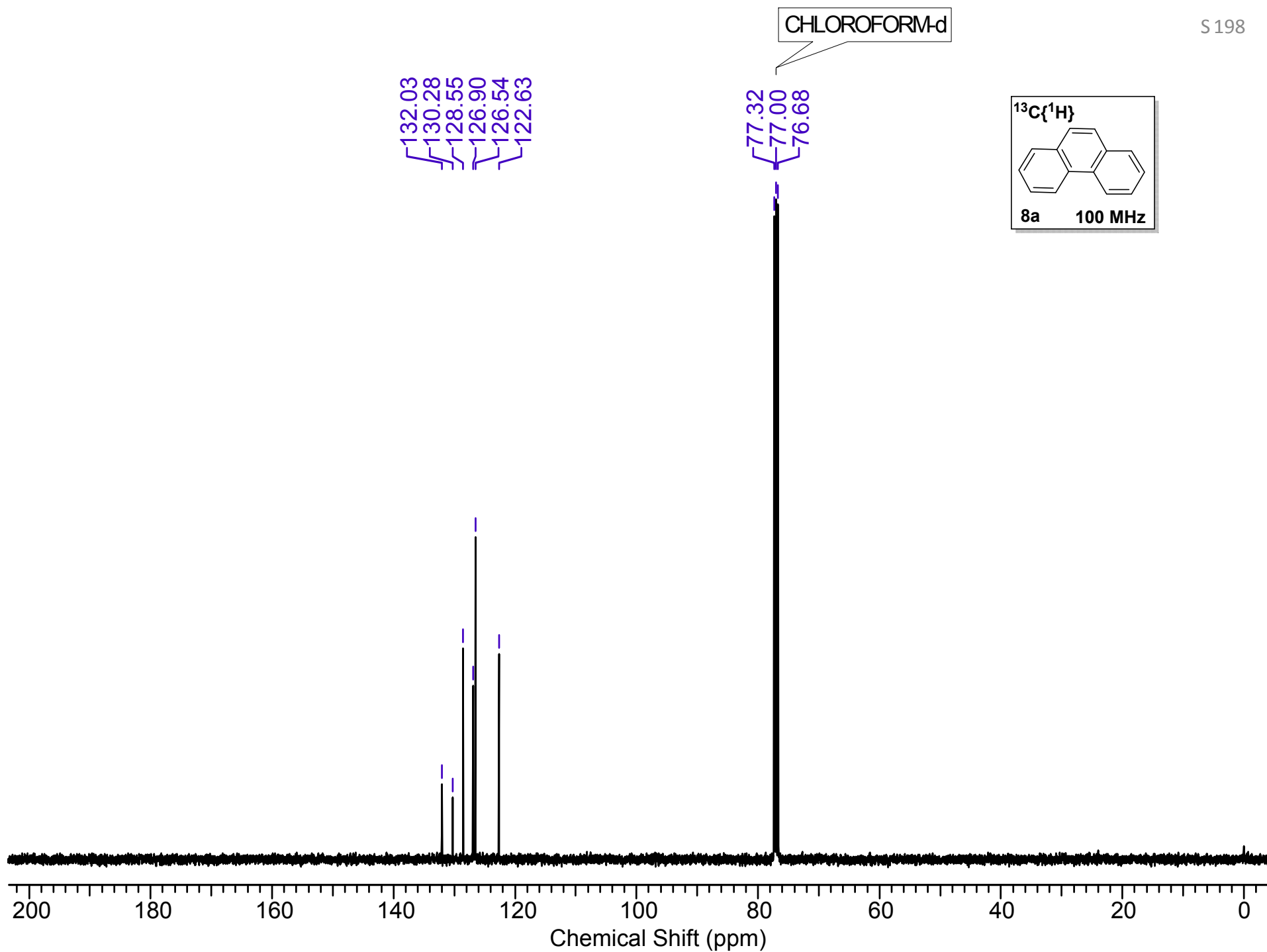
CHLOROFORM-d









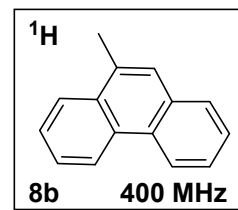


CHLOROFORM-d

Water

S 199

8.75
8.73
8.68
8.66
8.09
8.07
7.82
7.68
7.67
7.67
7.61
7.60
7.60
7.59
7.27



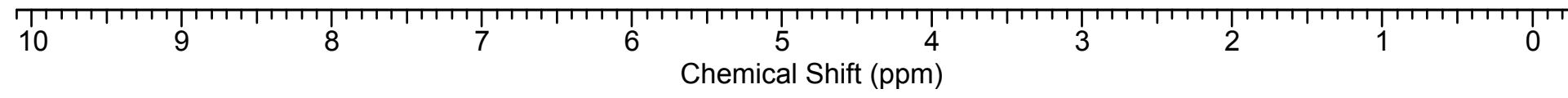
2.76

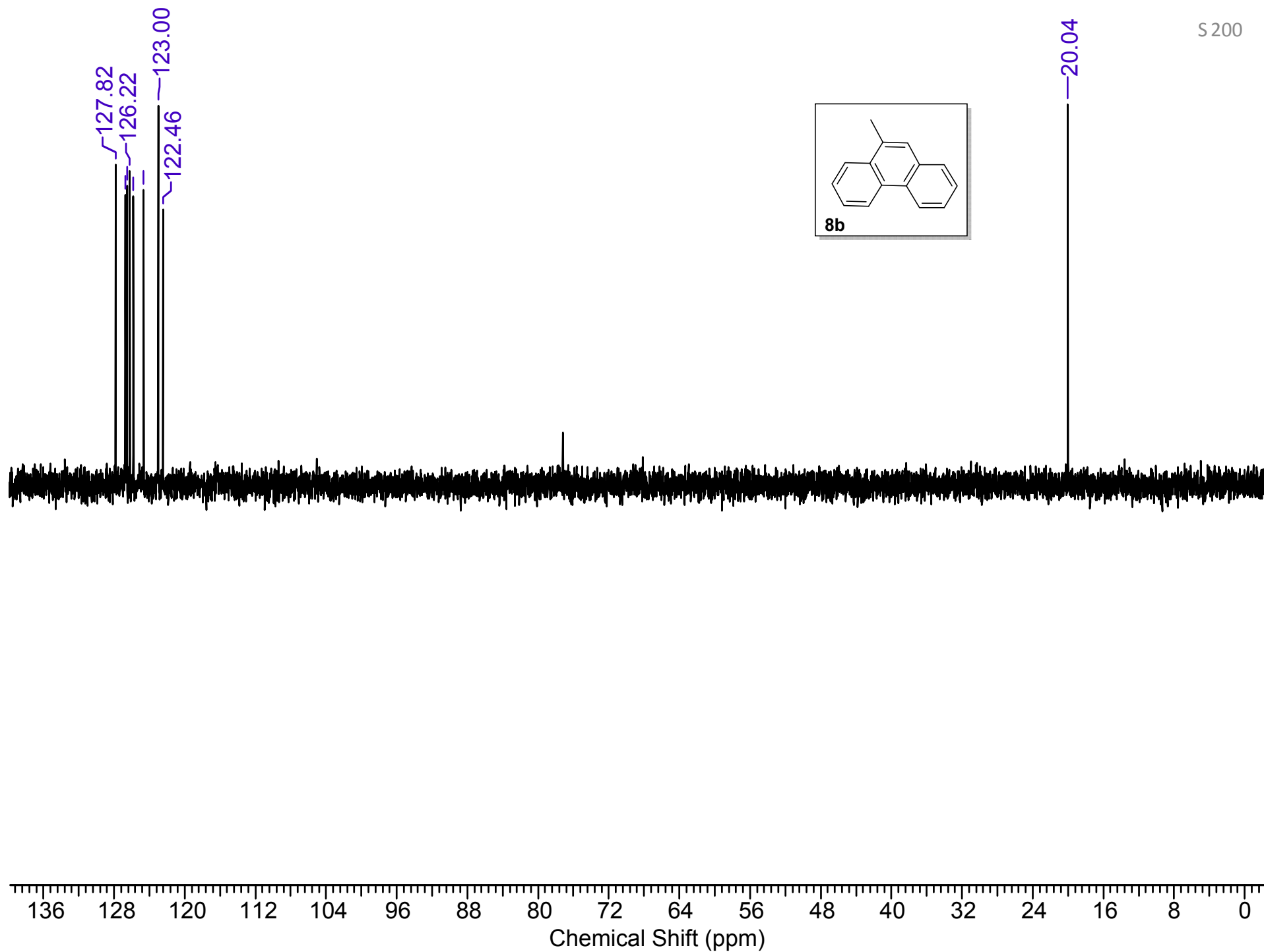
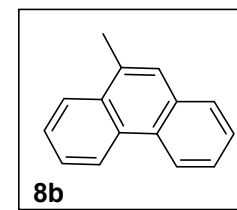
1.55

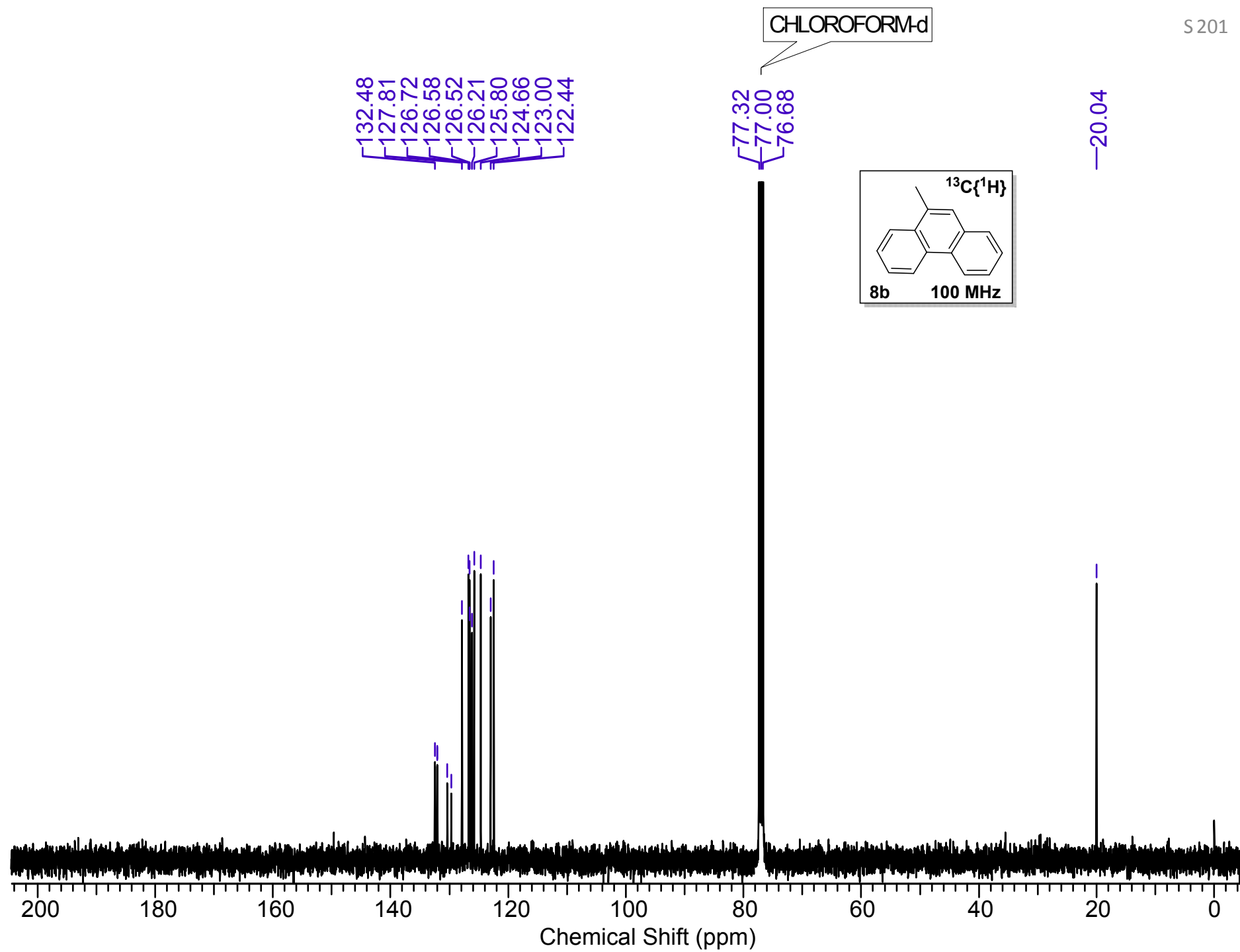
0.02

1.06
1.06
1.00
1.01
1.93
3.17

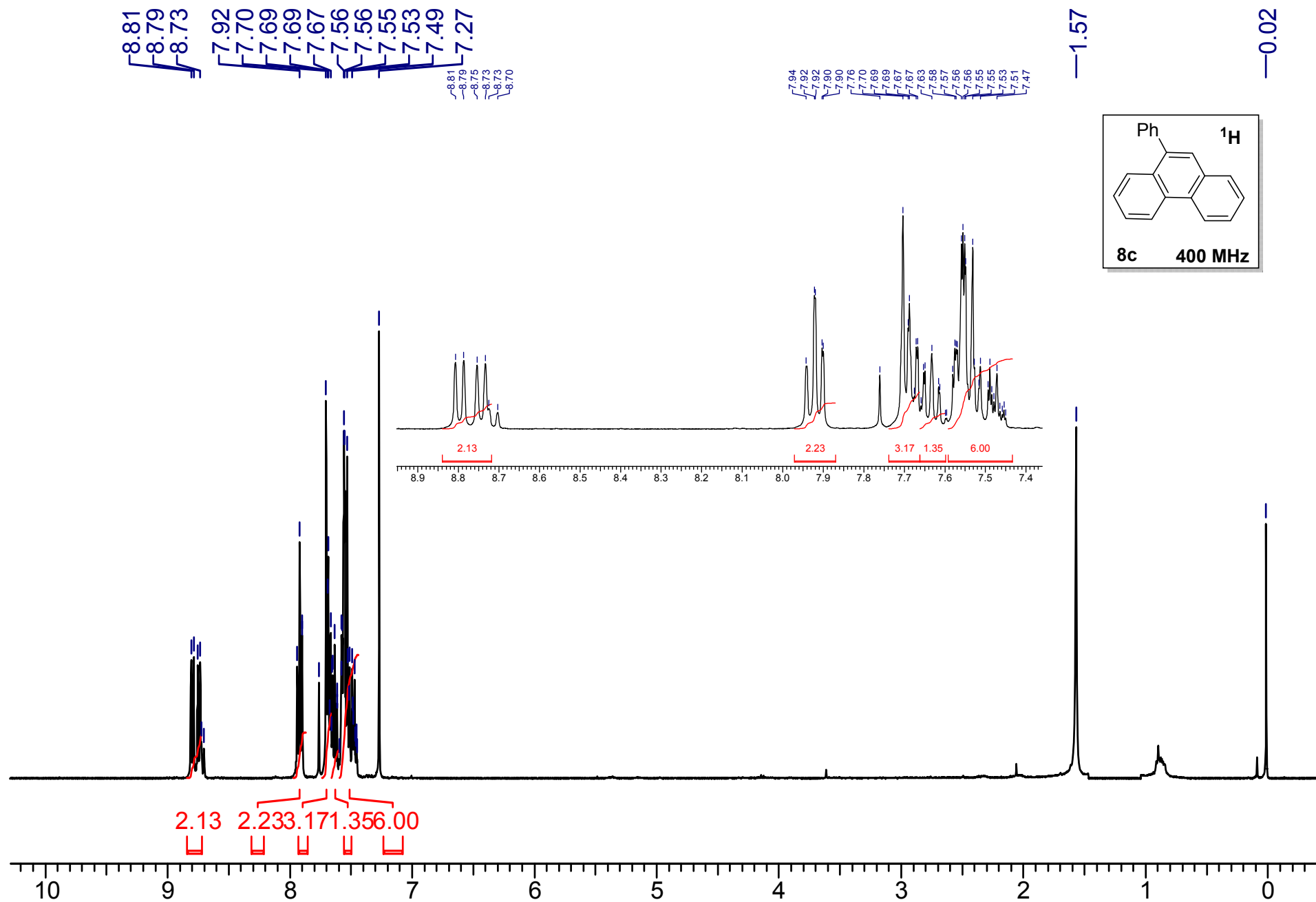
3.13

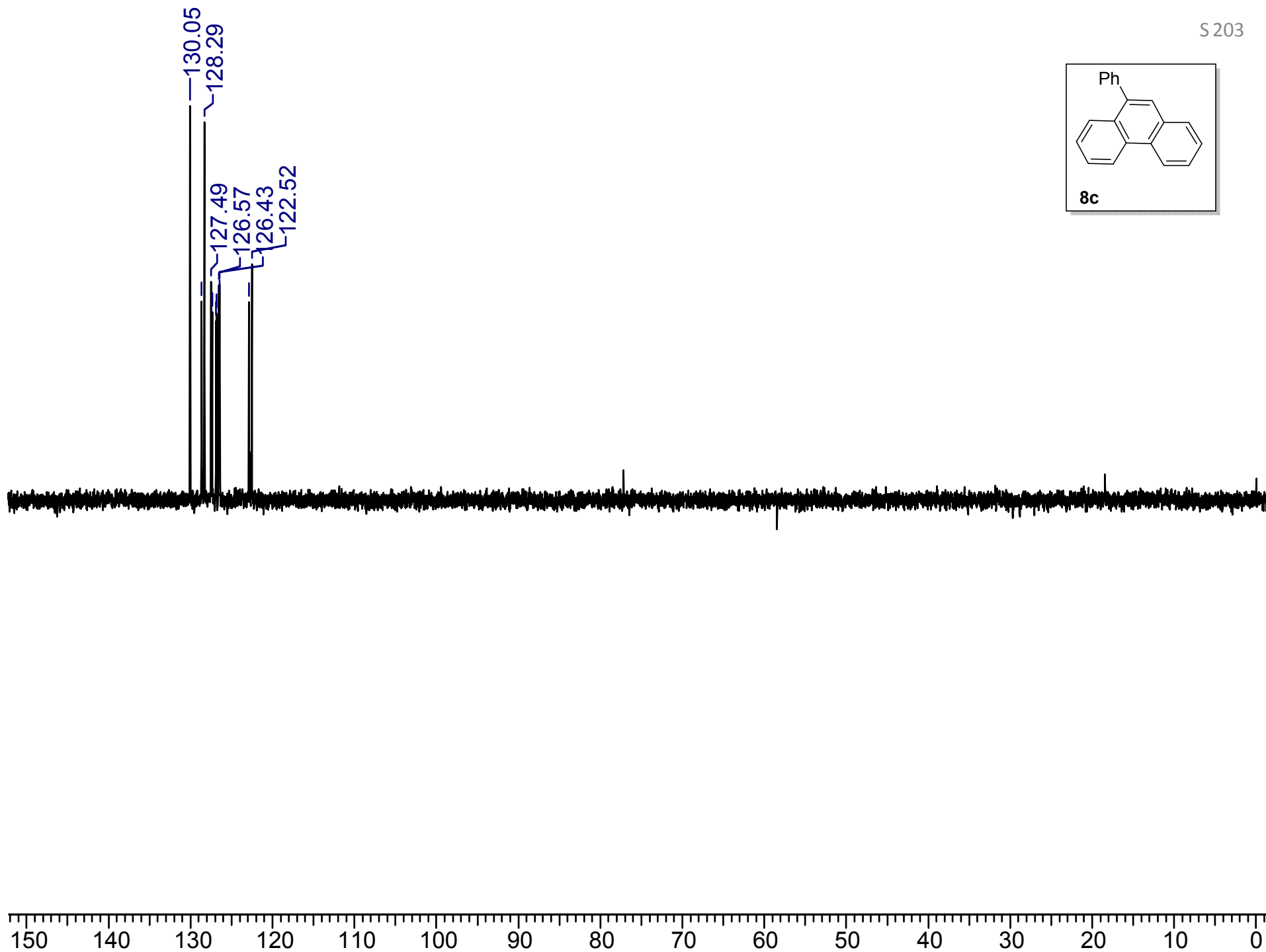
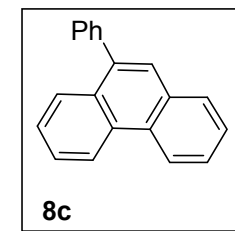


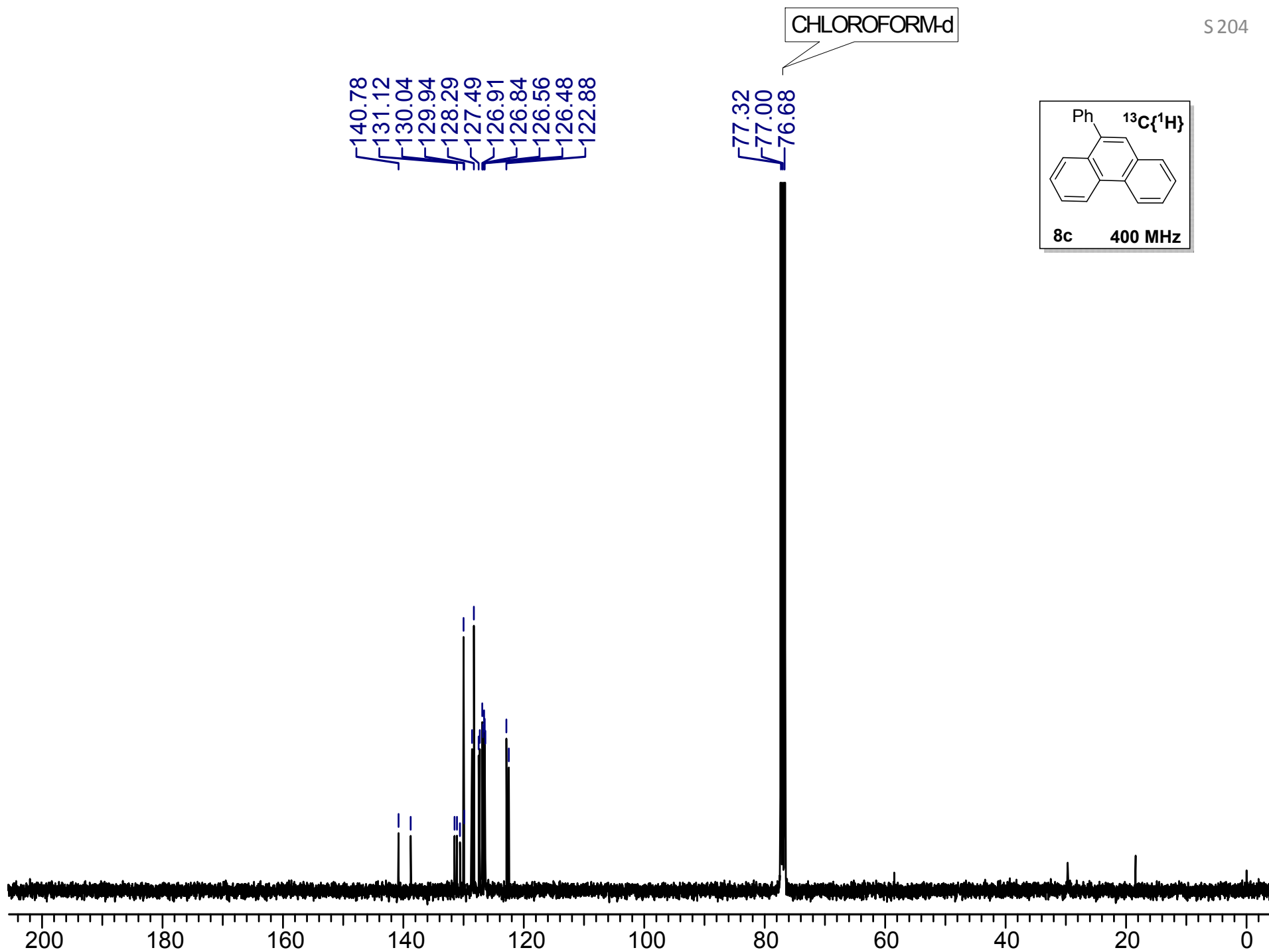




CHLOROFORM-d







CHLOROFORM-d

Water

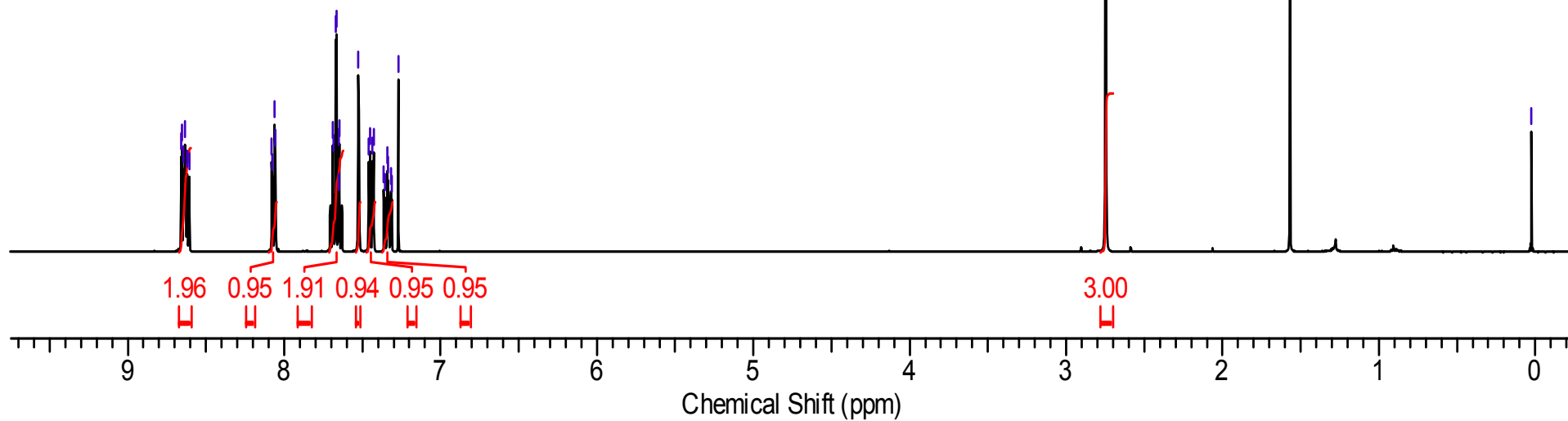
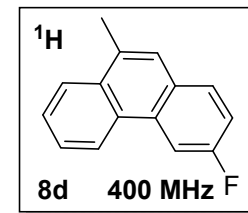
S 205

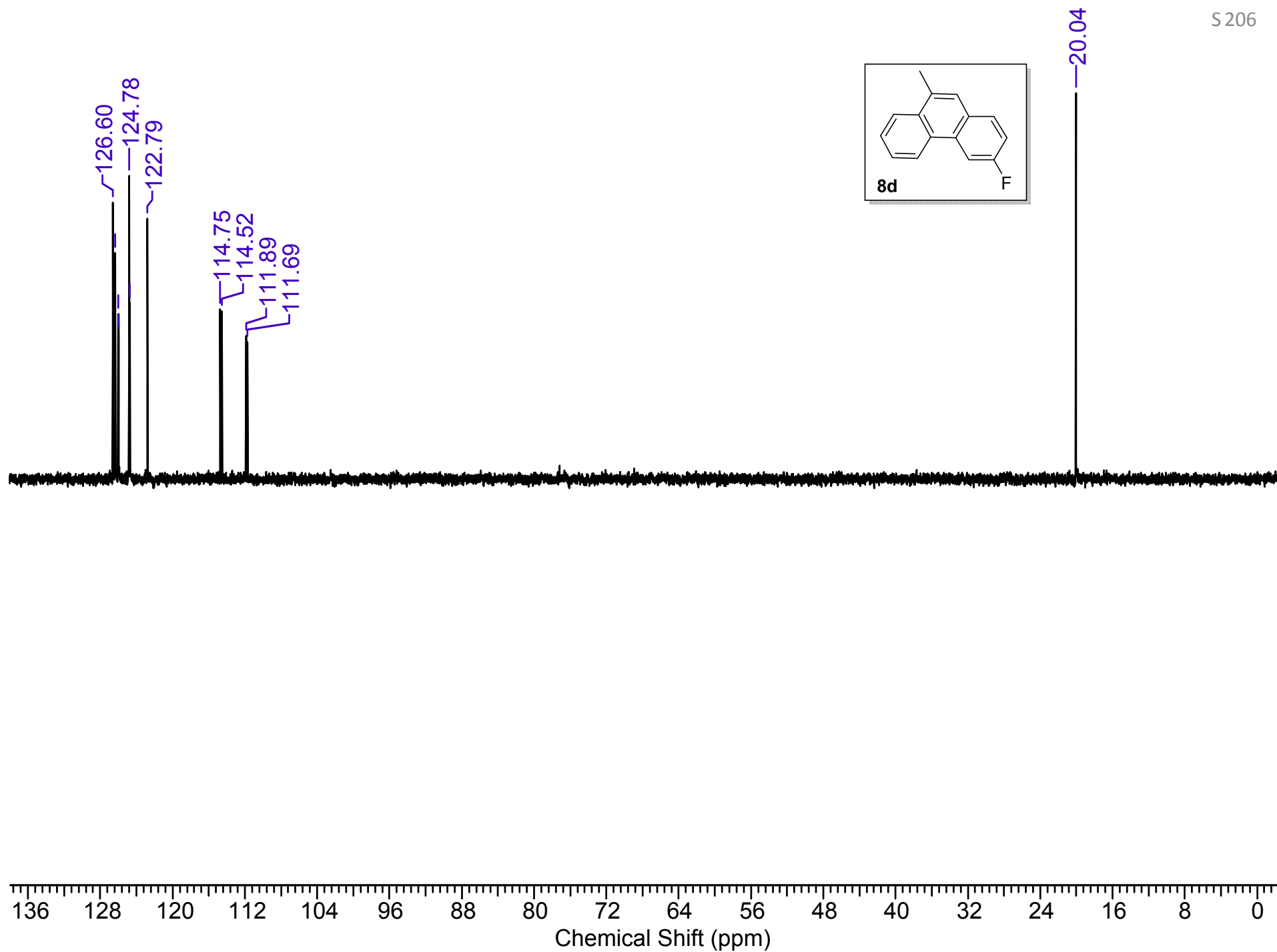
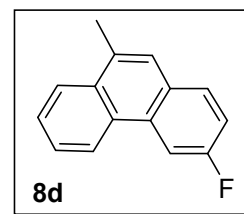
8.66
8.65
8.64
8.06
8.06
7.69
7.69
7.67
7.67
7.65
7.65
7.53
7.45
7.43
7.27

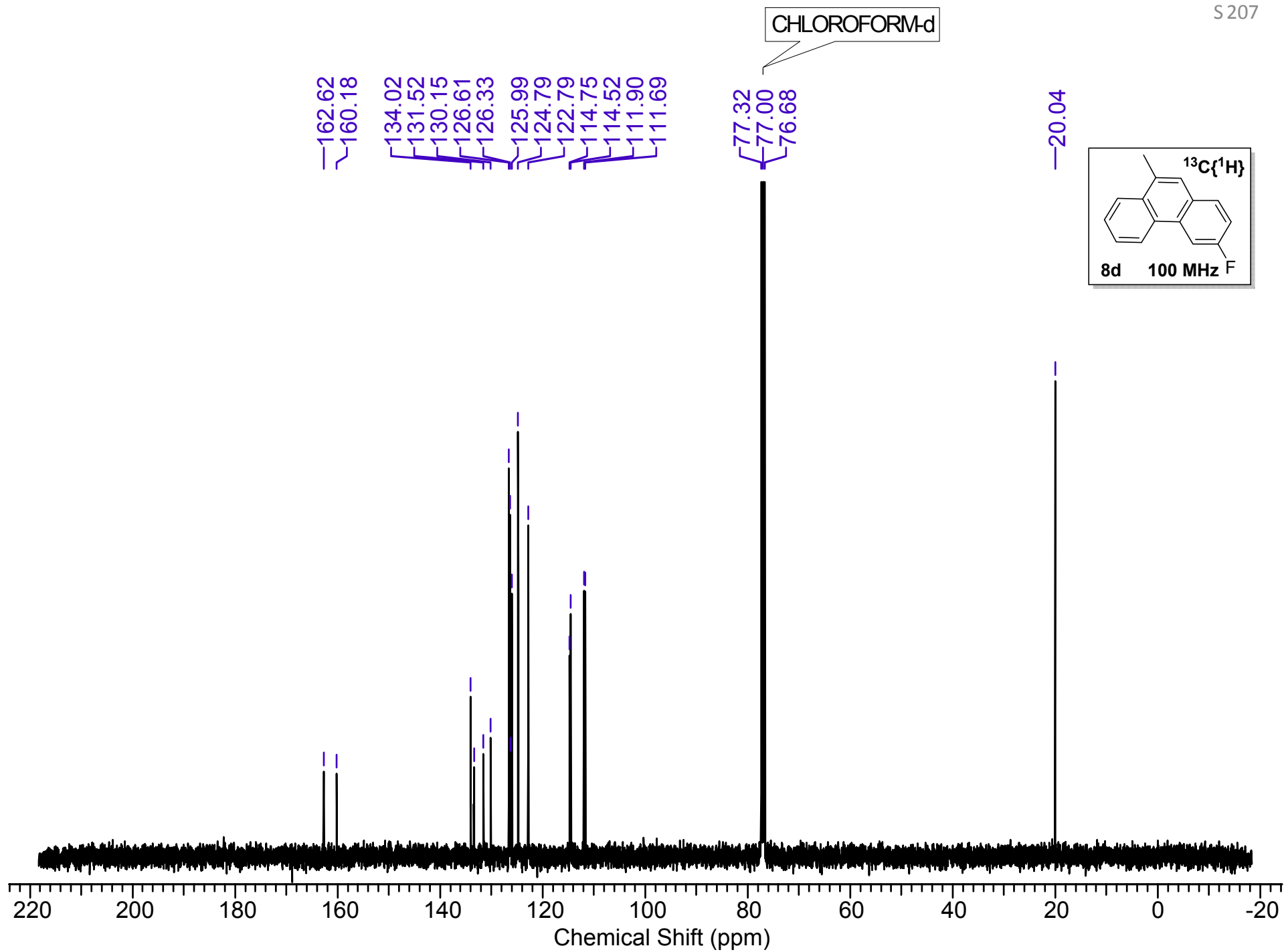
2.75
2.75

1.57

0.03







CHLOROFORM-d

Water

S 208

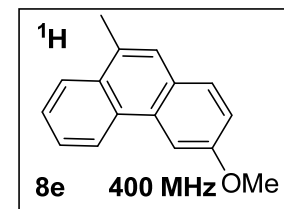
8.64
8.62
8.57
8.55
8.06
8.04
7.65
7.60
7.54
7.27
7.25
7.25
7.23
7.21
7.20

3.97

2.74

1.58

0.03

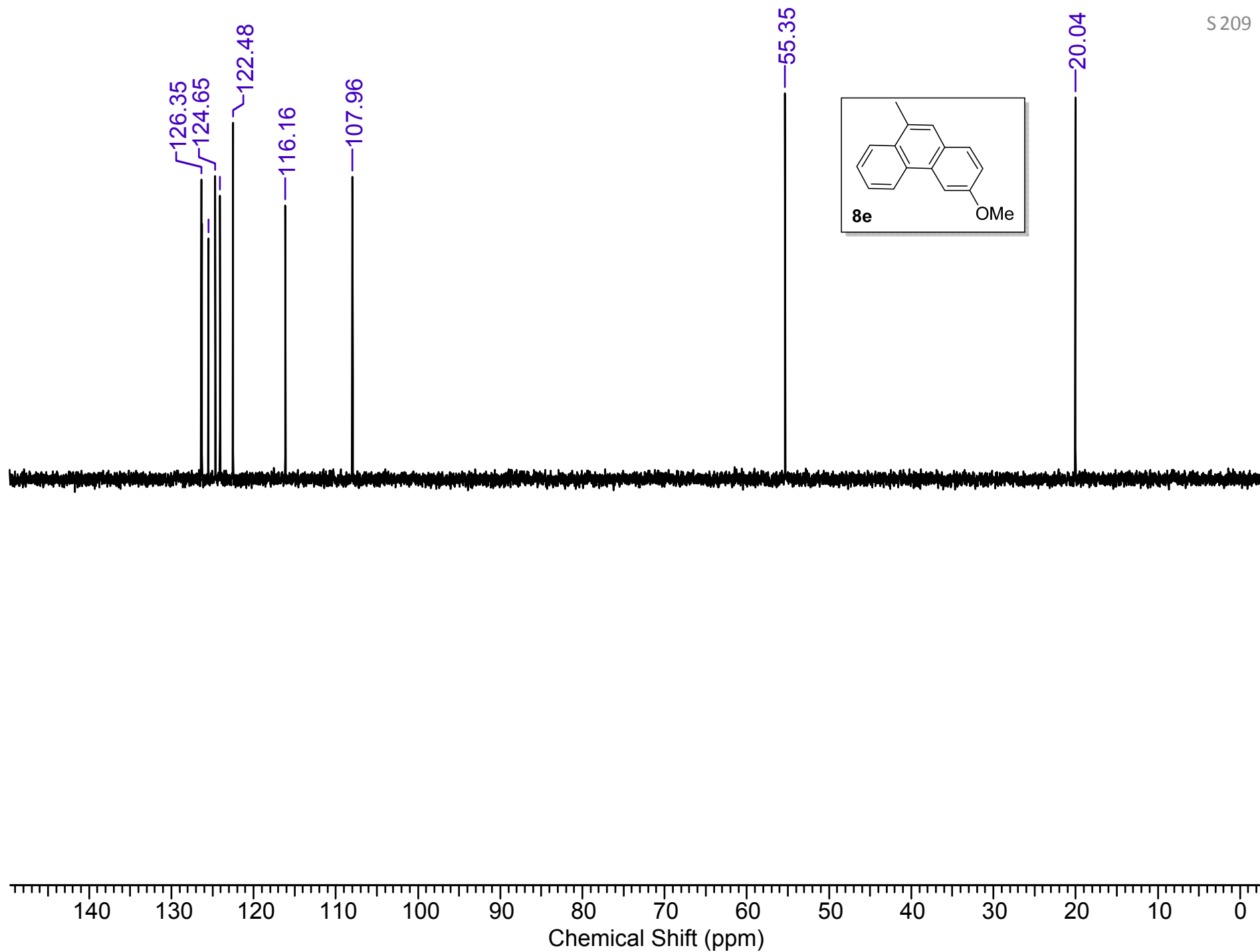


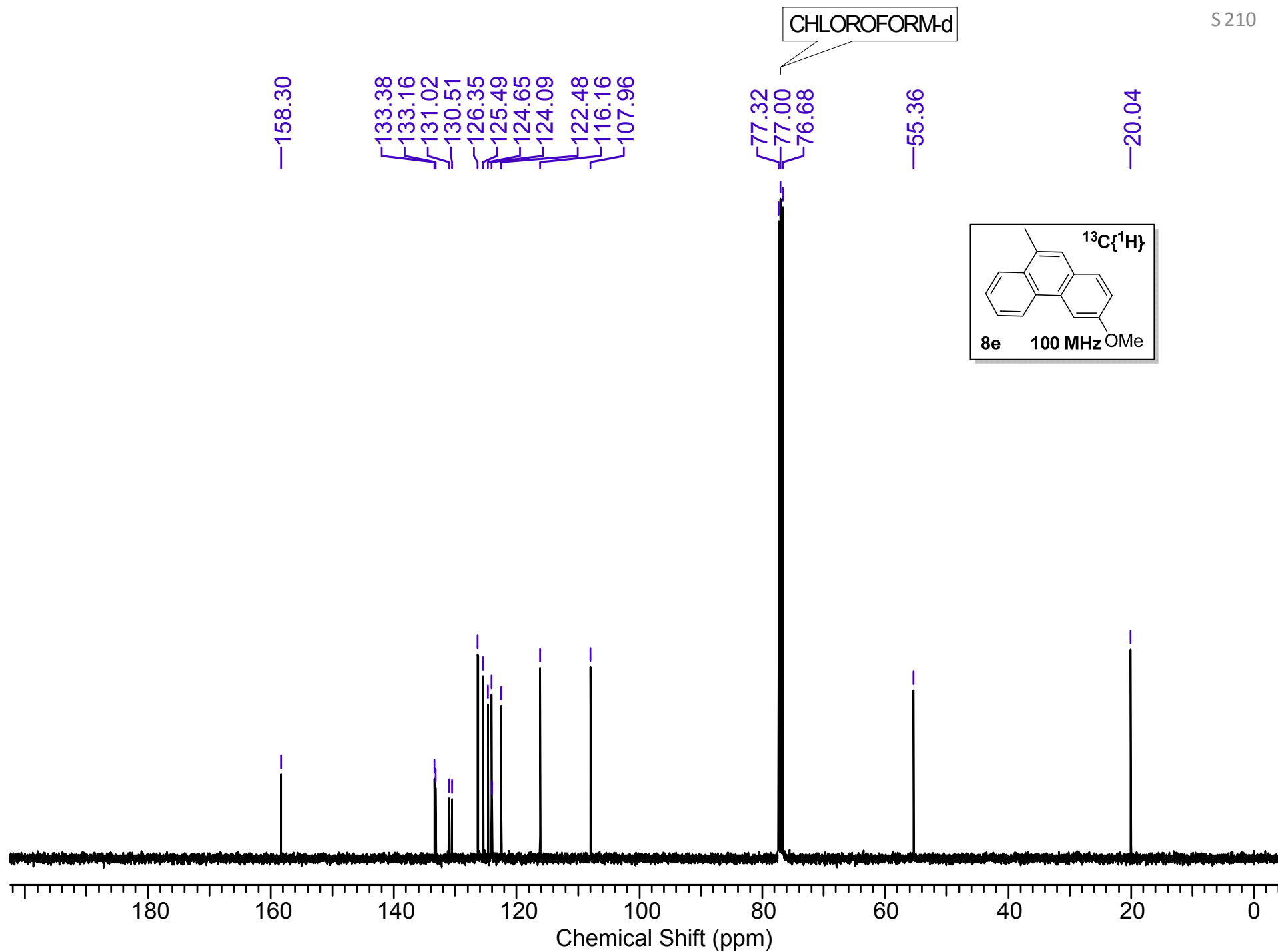
1.011.000.982.000.971.220.97

3.00

3.07

Chemical Shift (ppm)



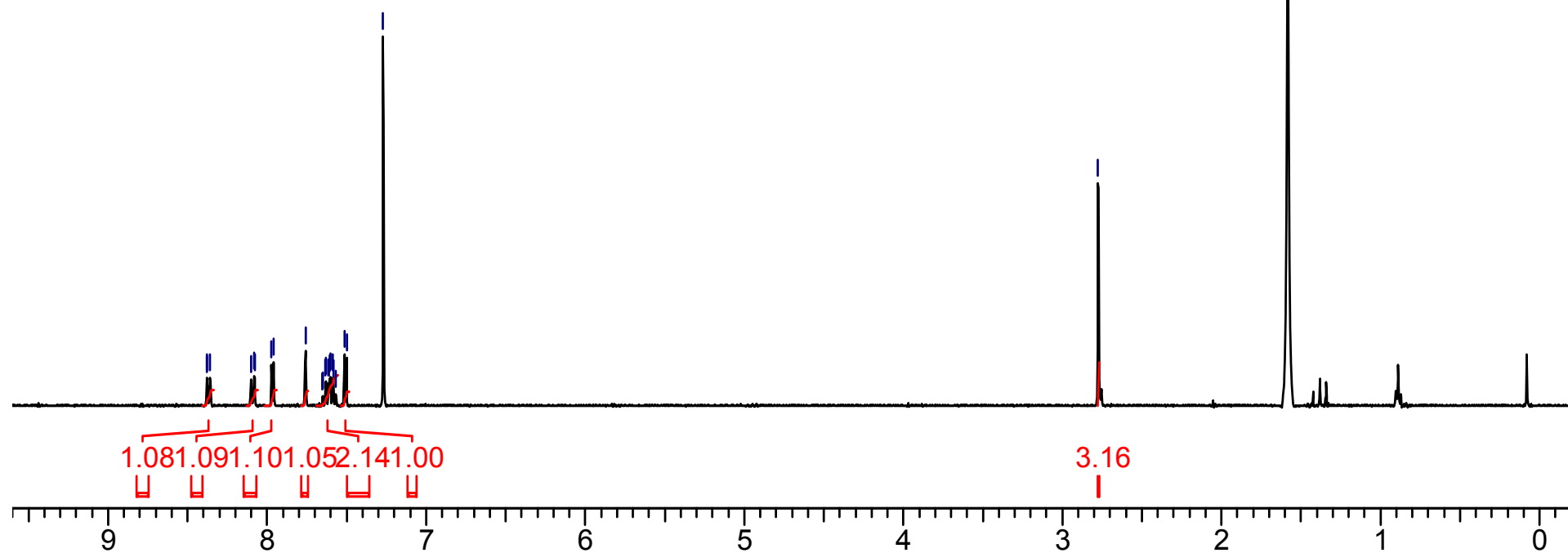
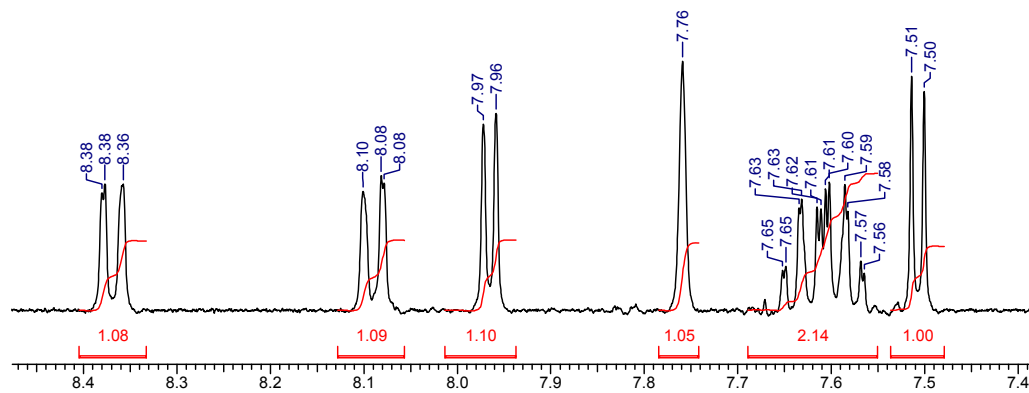
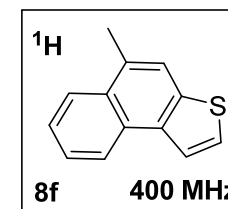


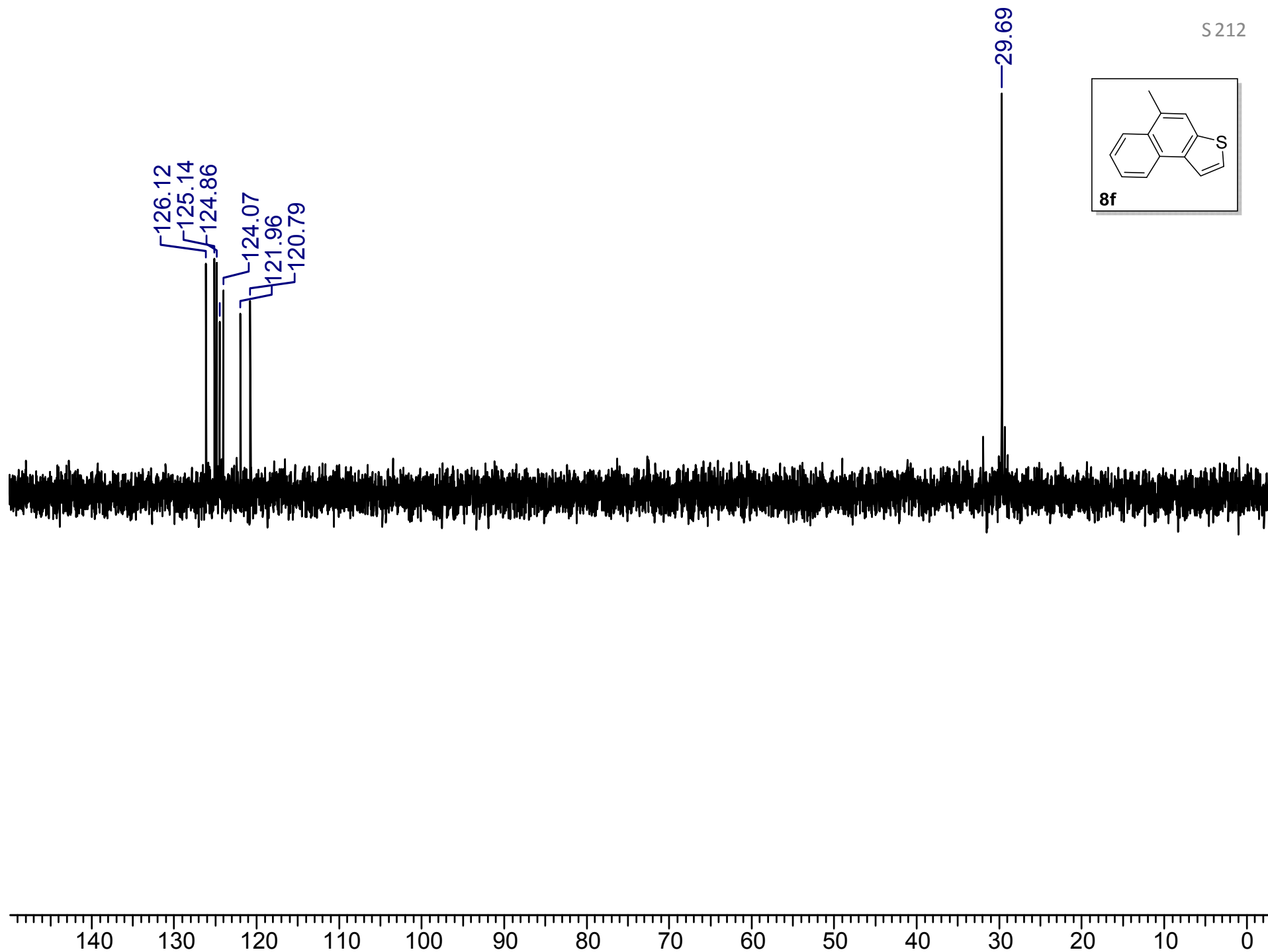
CHLOROFORM-d

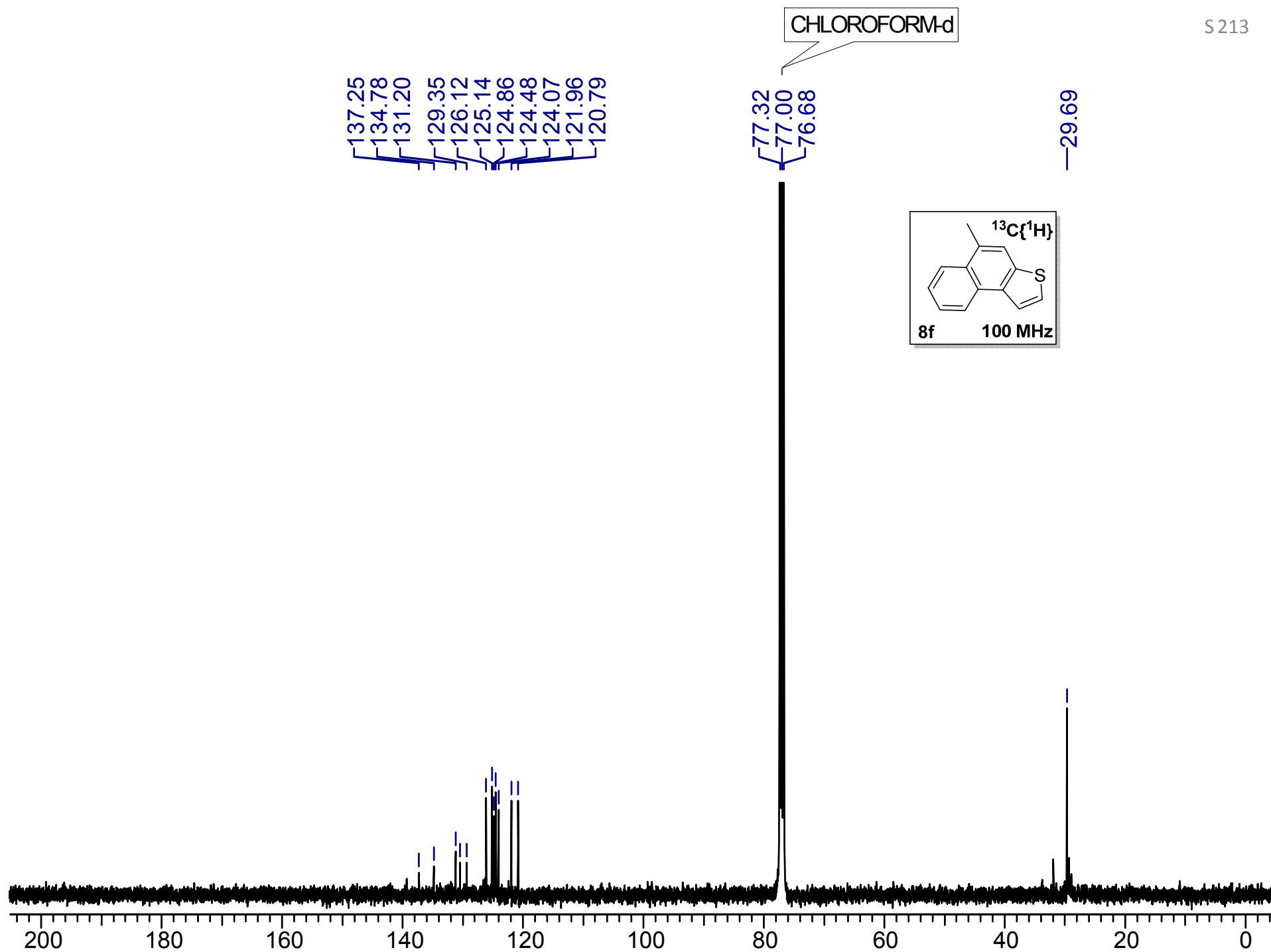
8.38
8.36
8.08
8.08
7.97
7.96
7.76
7.61
7.60
7.59
7.51
7.50
7.27

—2.78

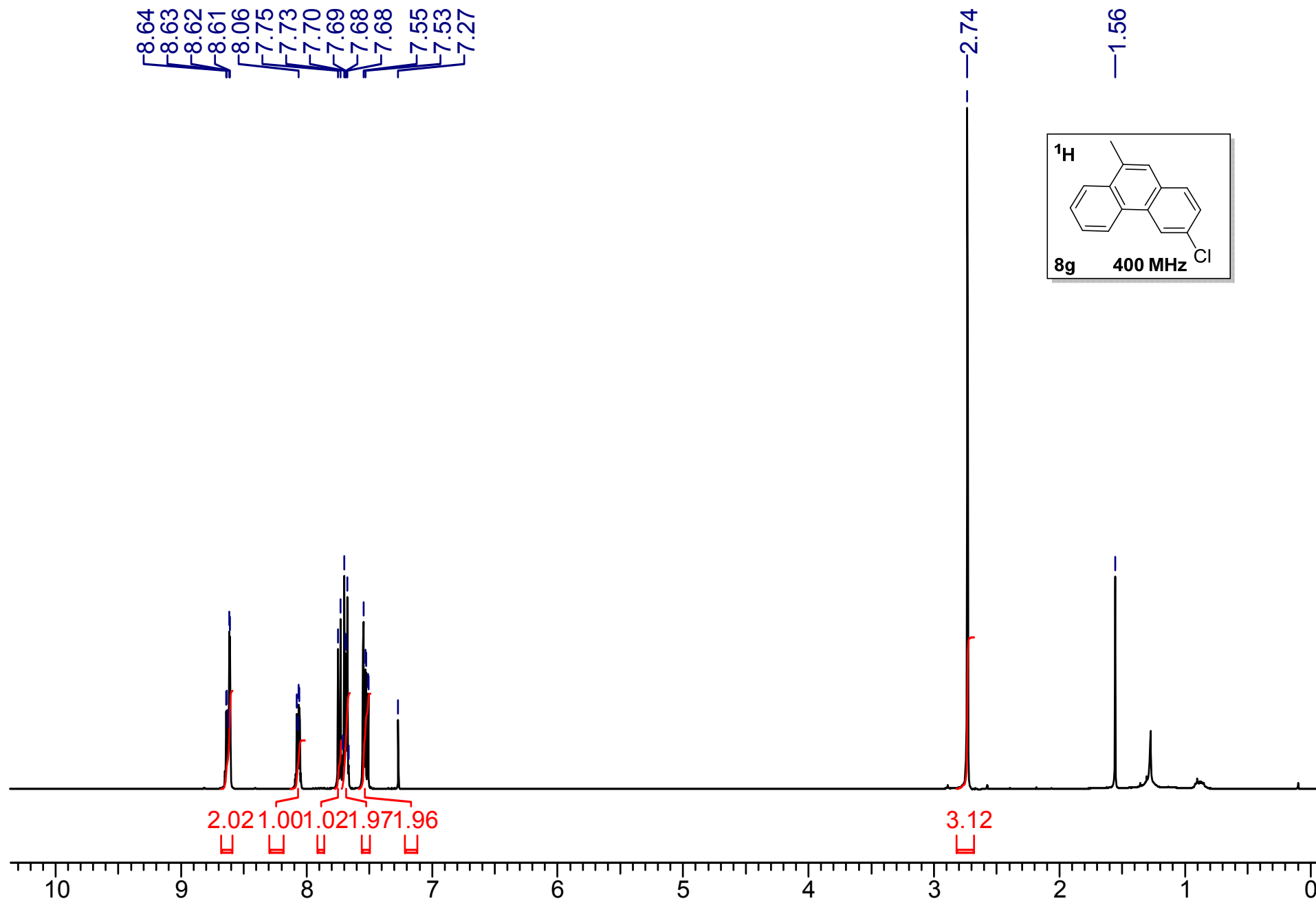
—1.58

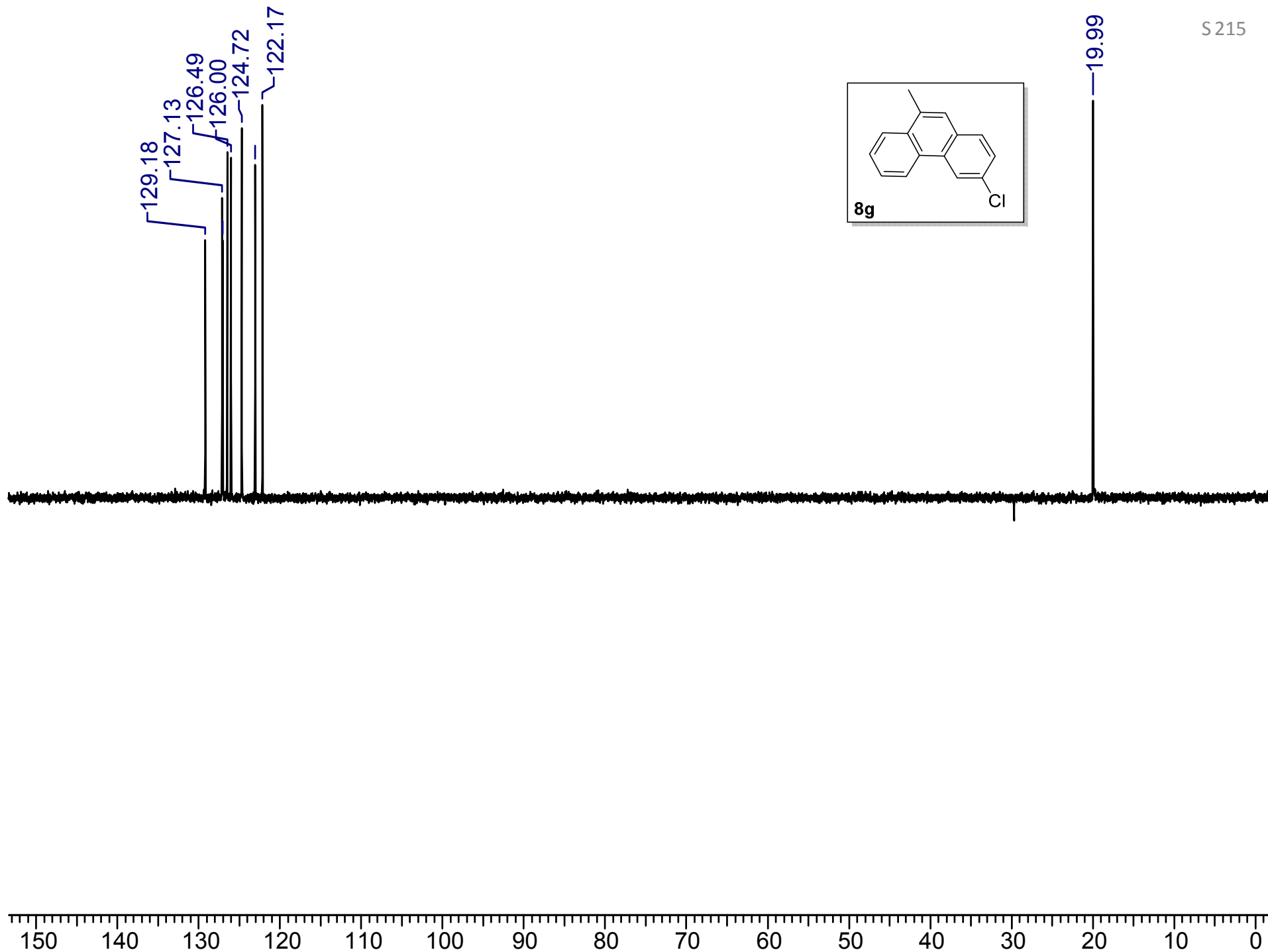
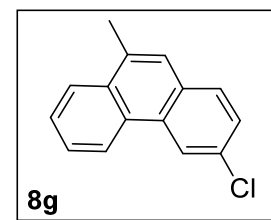


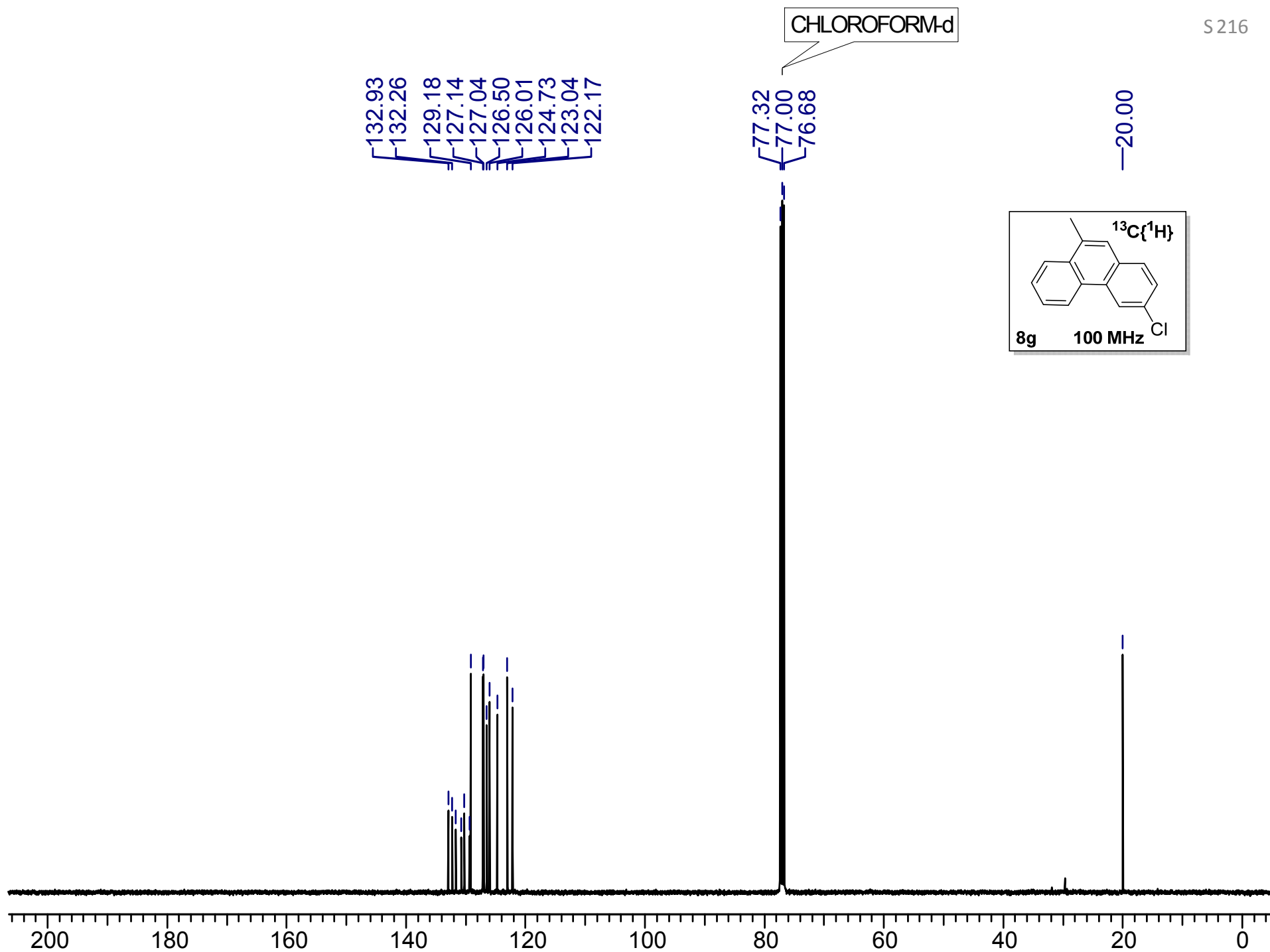




CHLOROFORM-d

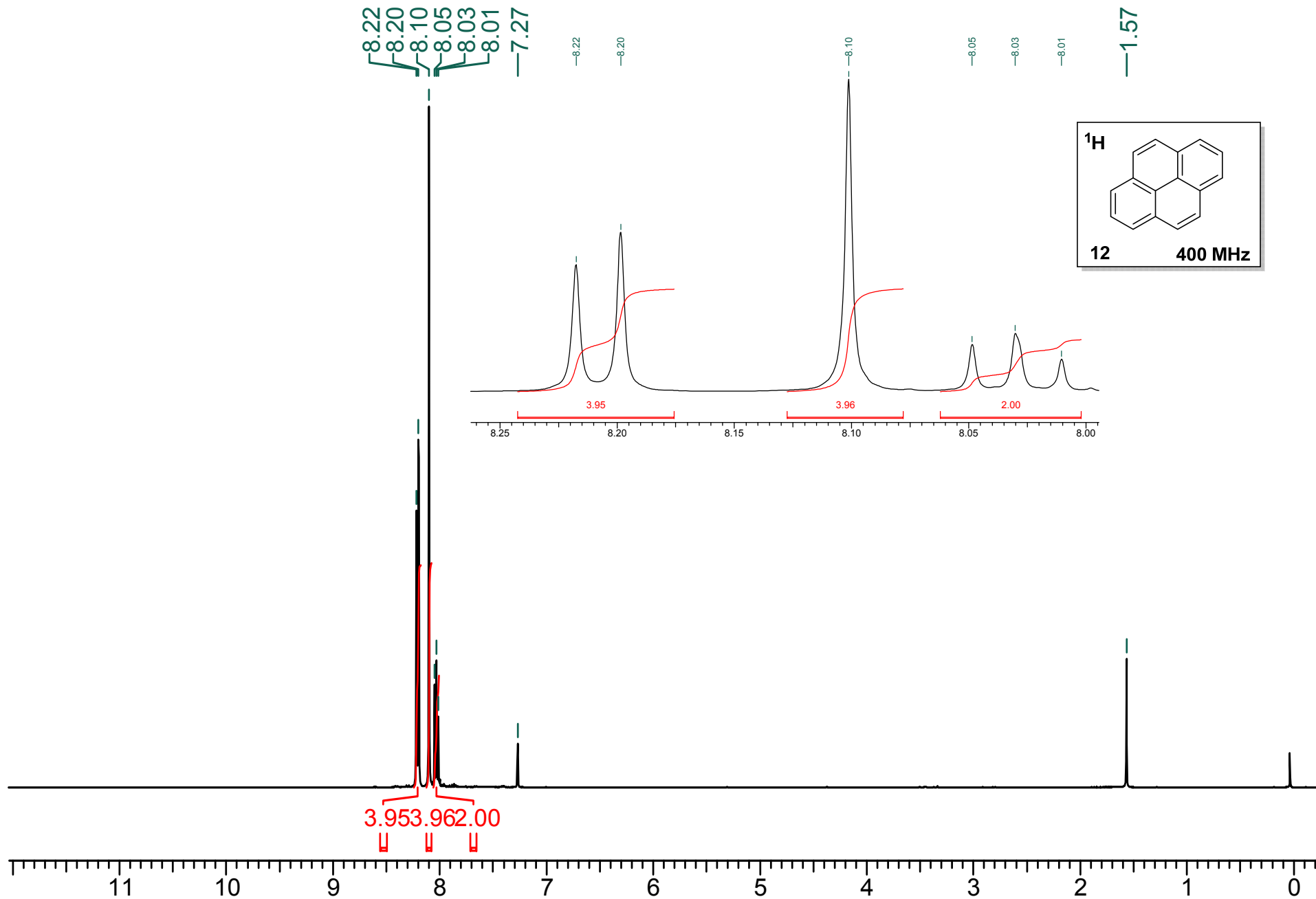


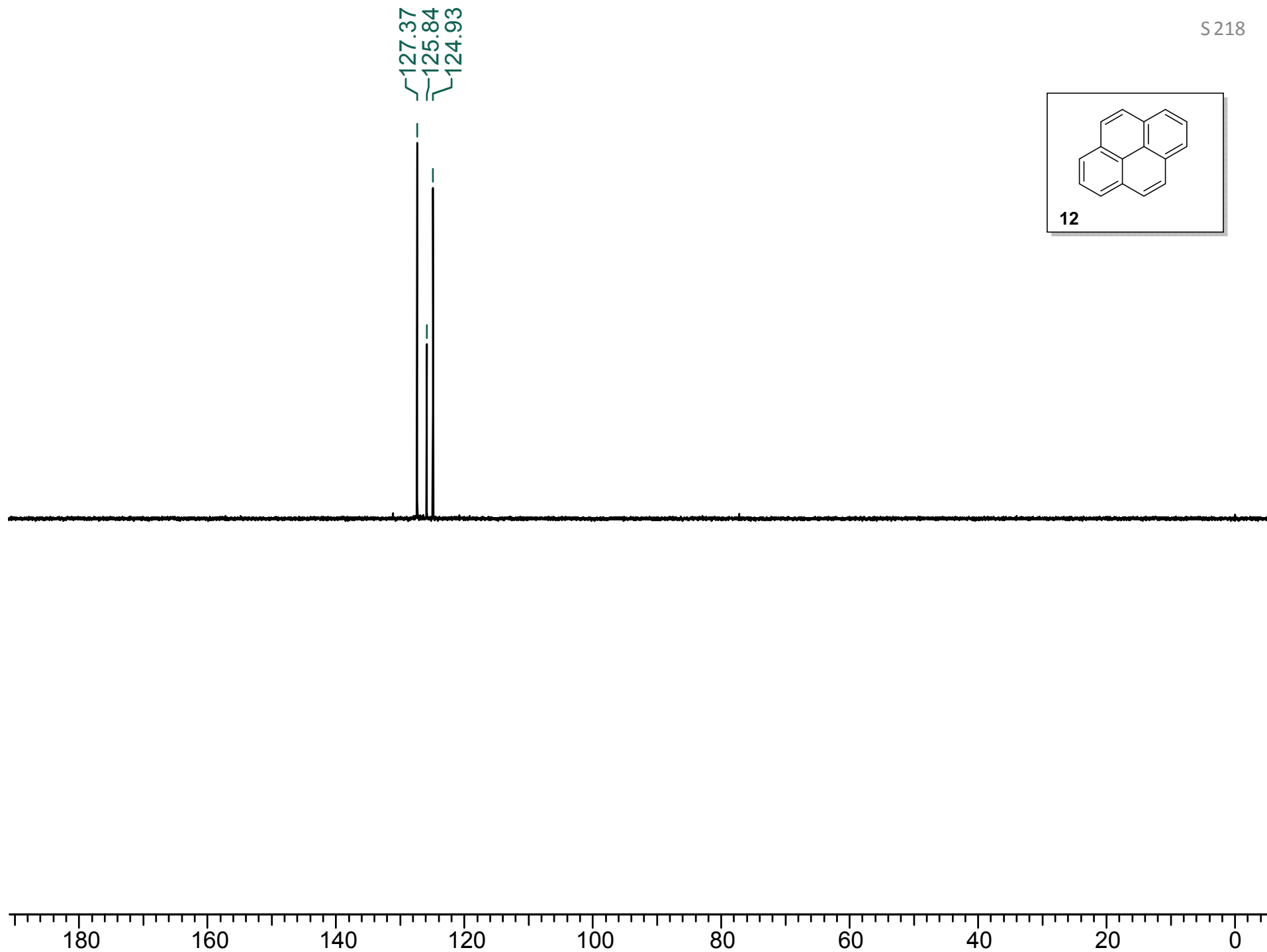
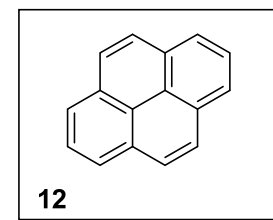


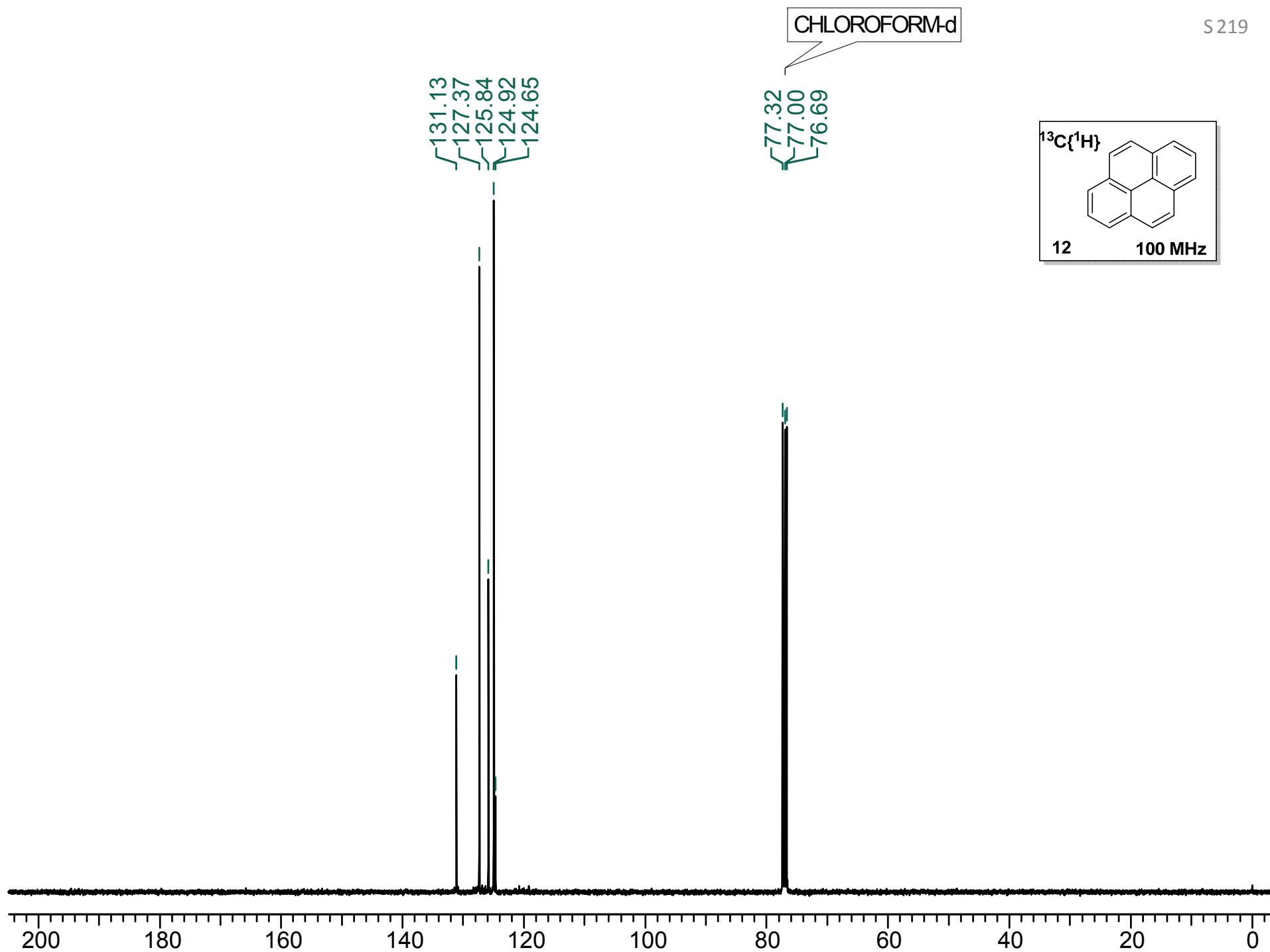


CHLOROFORM-d

S 217







CHLOROFORM-d

9.21
9.21
9.19
9.19
8.33
8.19
8.18
8.17
8.15
8.14
8.12
8.10
8.03
7.64
7.62
7.56
7.27

0.12
0.04

9.21
9.21
9.19

8.36
8.34
8.33
8.28
8.22
8.20
8.19
8.18
8.17
8.15
8.14
8.12
8.10
8.05
8.03
8.01

7.66
7.66
7.64
7.63
7.62
7.60
7.59
7.58

