

## Supporting Information

### Brain-penetrant, Orally Bioavailable Microtubule-Stabilizing Small Molecules are Potential Candidates Therapeutics for Alzheimer's Disease and Related Tauopathies

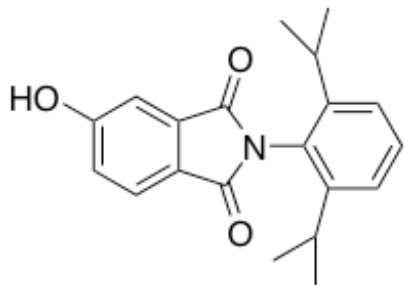
Kevin Lou,<sup>a</sup> Yuemang Yao,<sup>b</sup> Adam Hoye,<sup>a</sup> Michael James,<sup>b</sup> Anne-Sophie Cornec,<sup>a</sup> Edward Hyde,<sup>b</sup> Bryant Gay,<sup>a</sup> Virginia M-Y Lee,<sup>b</sup> John Q. Trojanowski,<sup>b</sup> Amos B. Smith III,<sup>a</sup> Kurt R. Brunden,<sup>b,\*</sup> and Carlo Ballatore<sup>a,b,\*</sup>

<sup>a</sup>Department of Chemistry, School of Arts and Sciences, University of Pennsylvania, 231 South 34th St., Philadelphia, PA 19104-6323; <sup>b</sup>Center for Neurodegenerative Diseases Research, Institute on Aging, University of Pennsylvania, 3600 Spruce Street, Philadelphia, PA 19104-6323

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**S2-144.** NMR spectra of test compounds.

**S145-174.** X-ray crystal structures of compounds **23**, **36**, and **43**.



7.89  
7.80  
7.79  
7.49  
7.47  
7.46  
7.37  
7.37  
7.32  
7.31  
7.30  
7.30  
7.27  
7.27  
7.07  
7.07  
7.06  
7.05

2.77  
2.75  
2.74  
2.73  
2.71  
2.25  
2.25  
2.16

1.19  
1.19  
1.18  
1.17

ss sds s

s s

C (d) 7.80  
D (d) 7.37  
B (s) 7.89  
A (t) 7.47  
F (dd) 7.06  
E (d) 7.31

G (p) 2.74  
H (s) 2.16

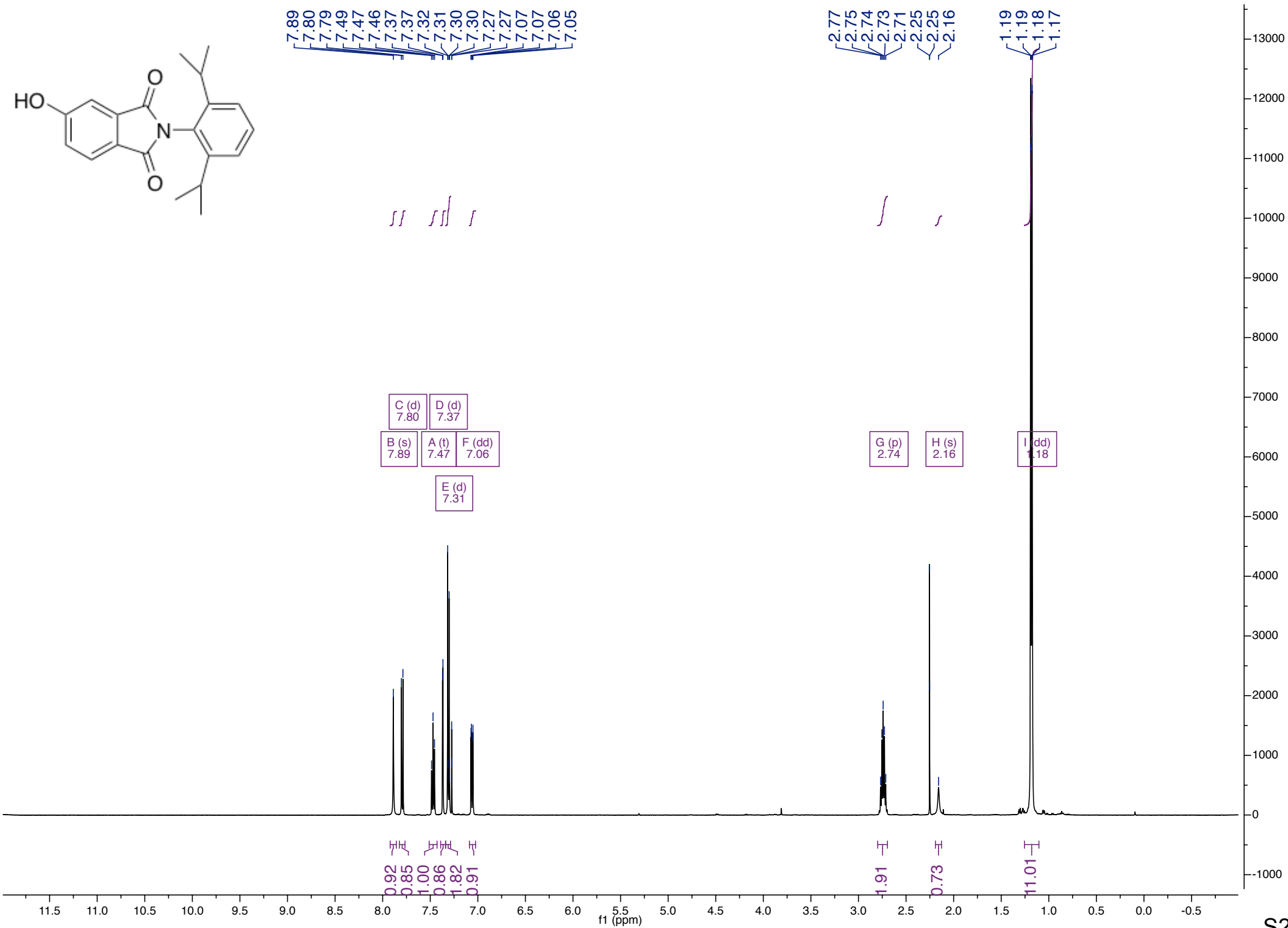
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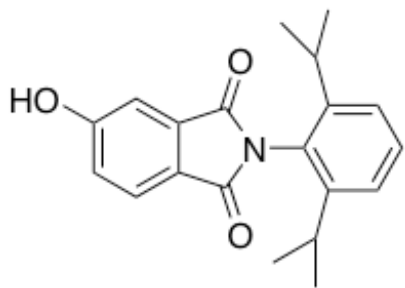
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1.82  
0.91

1.91

0.73

11.01





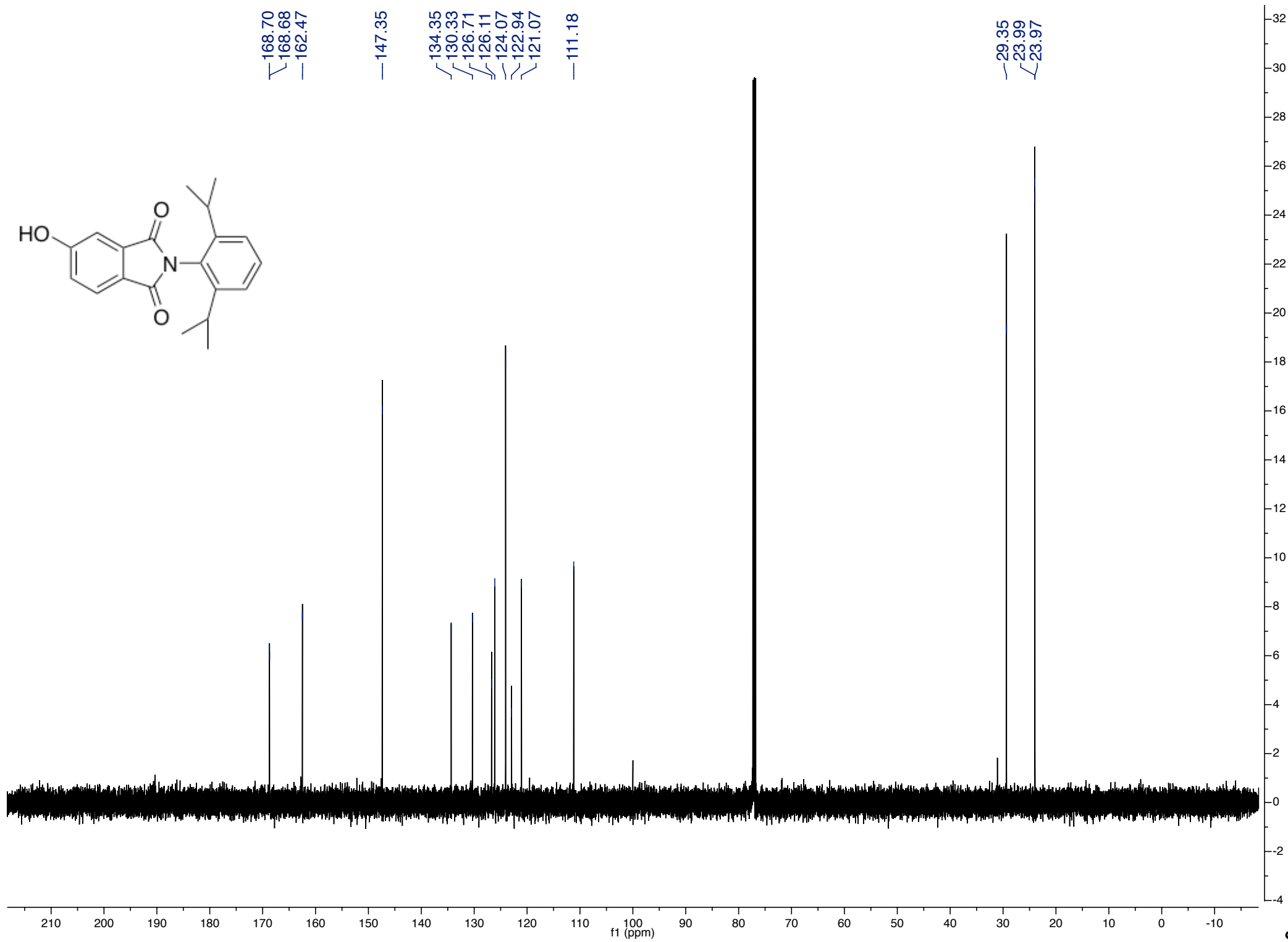
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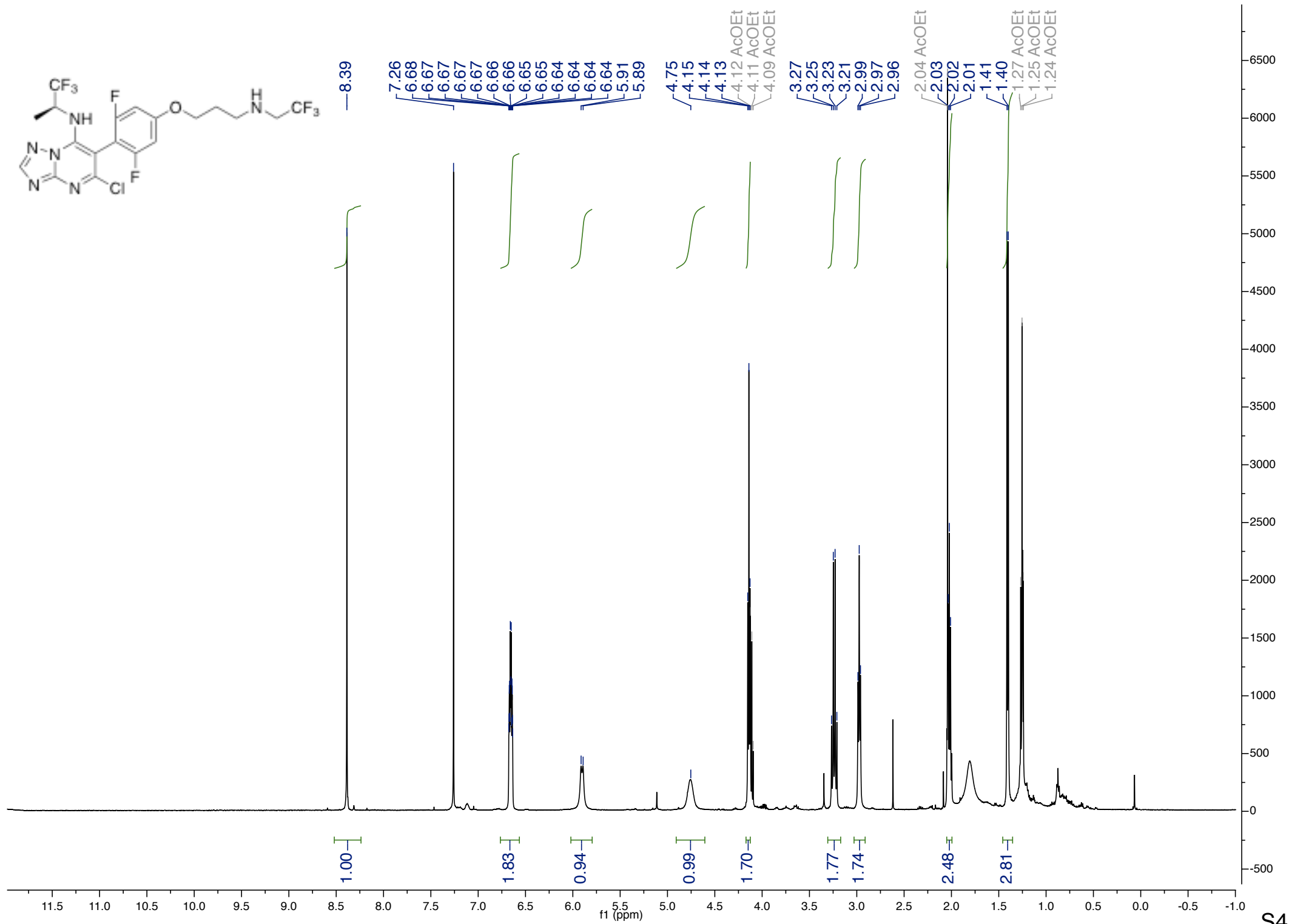
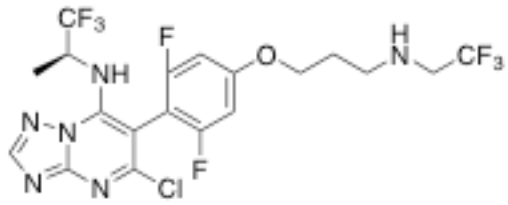
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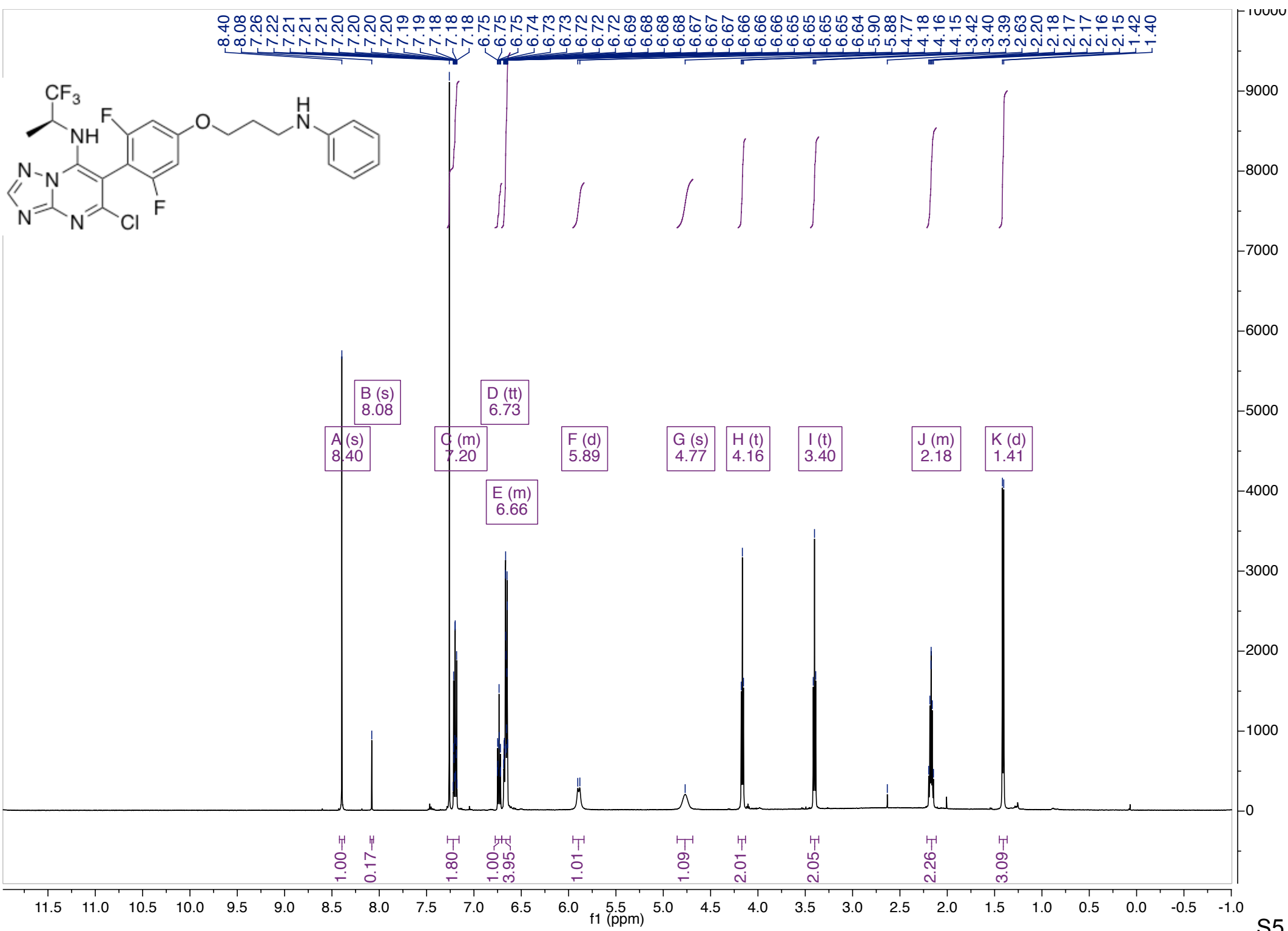
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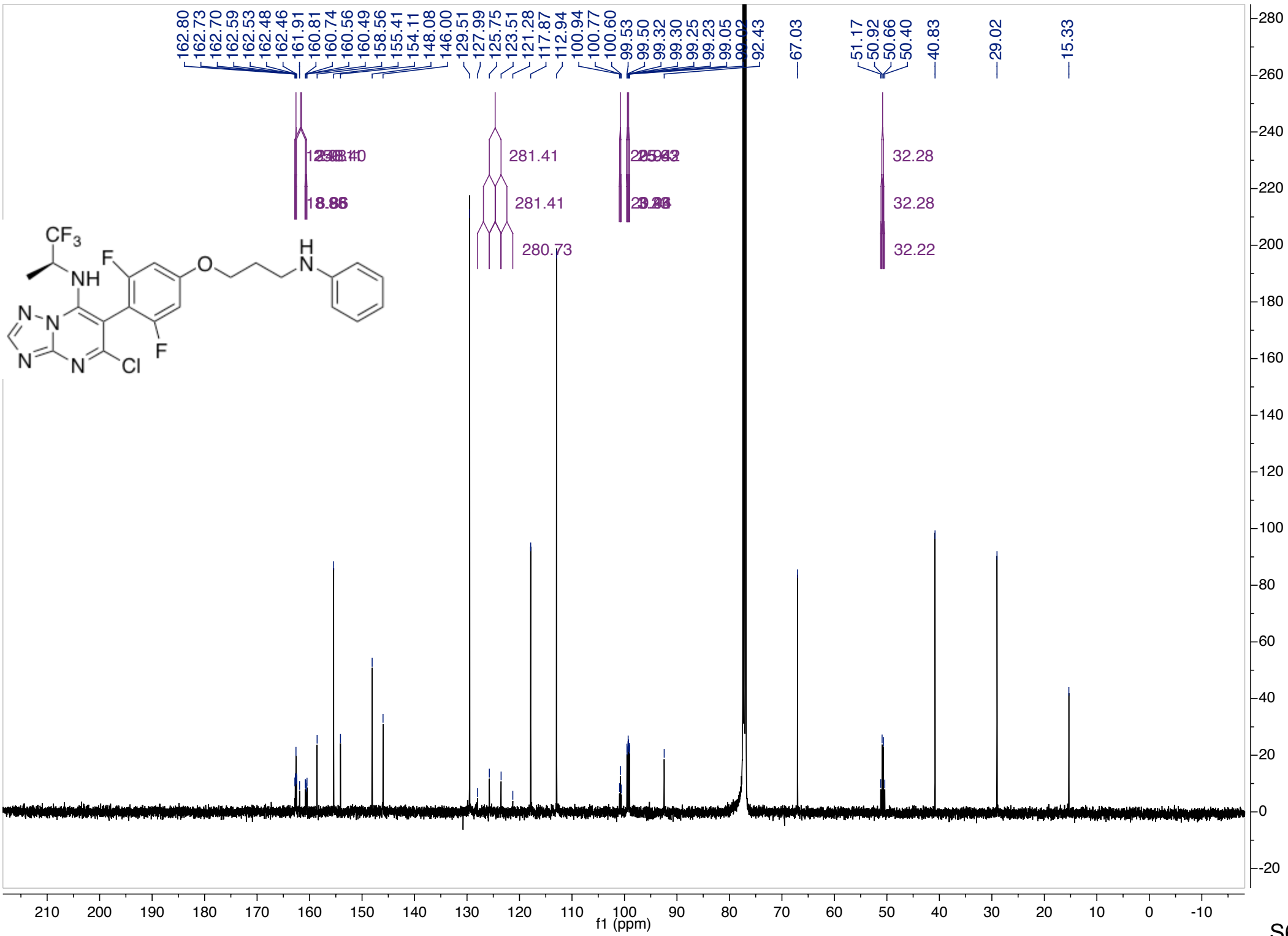
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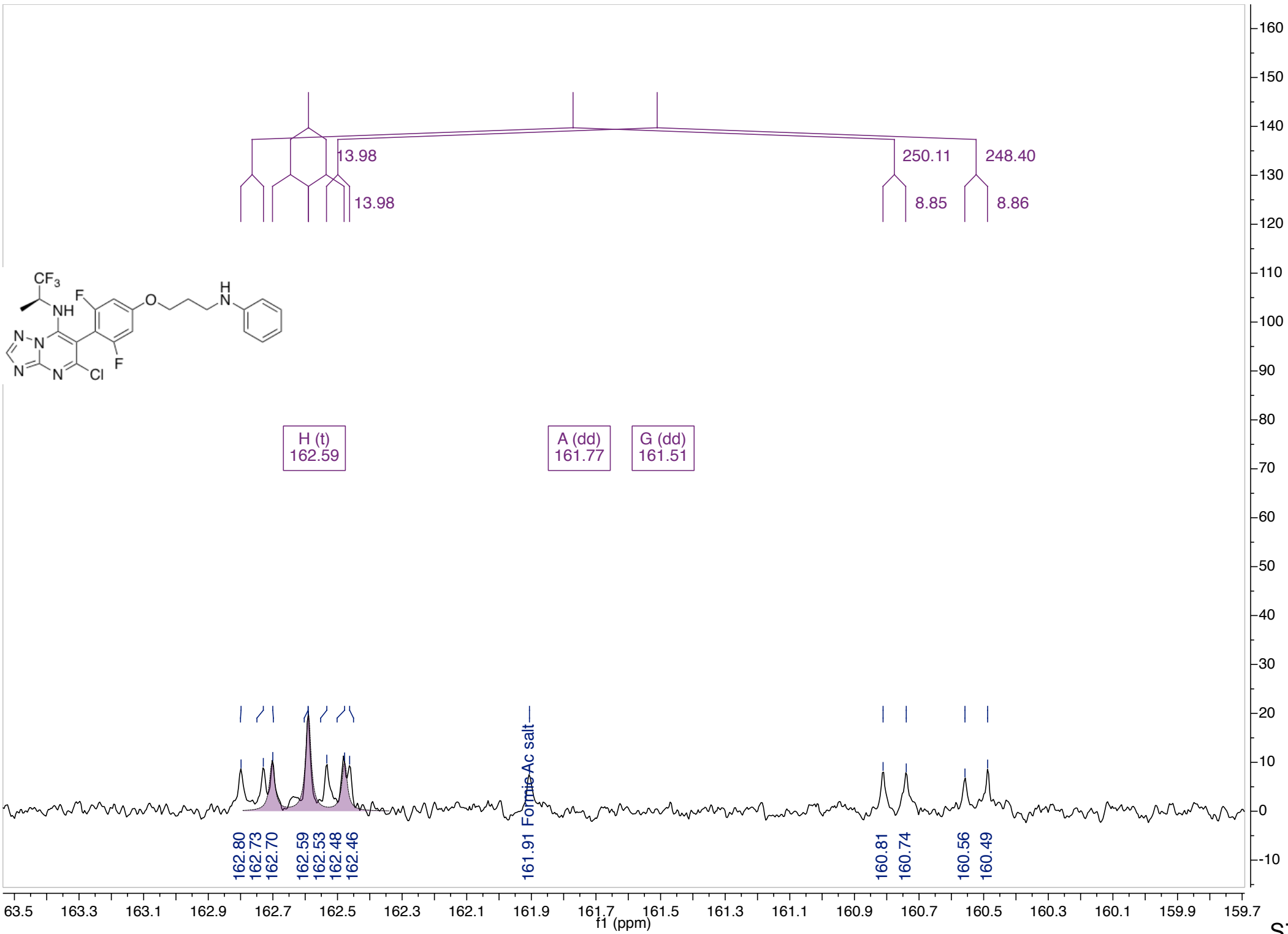
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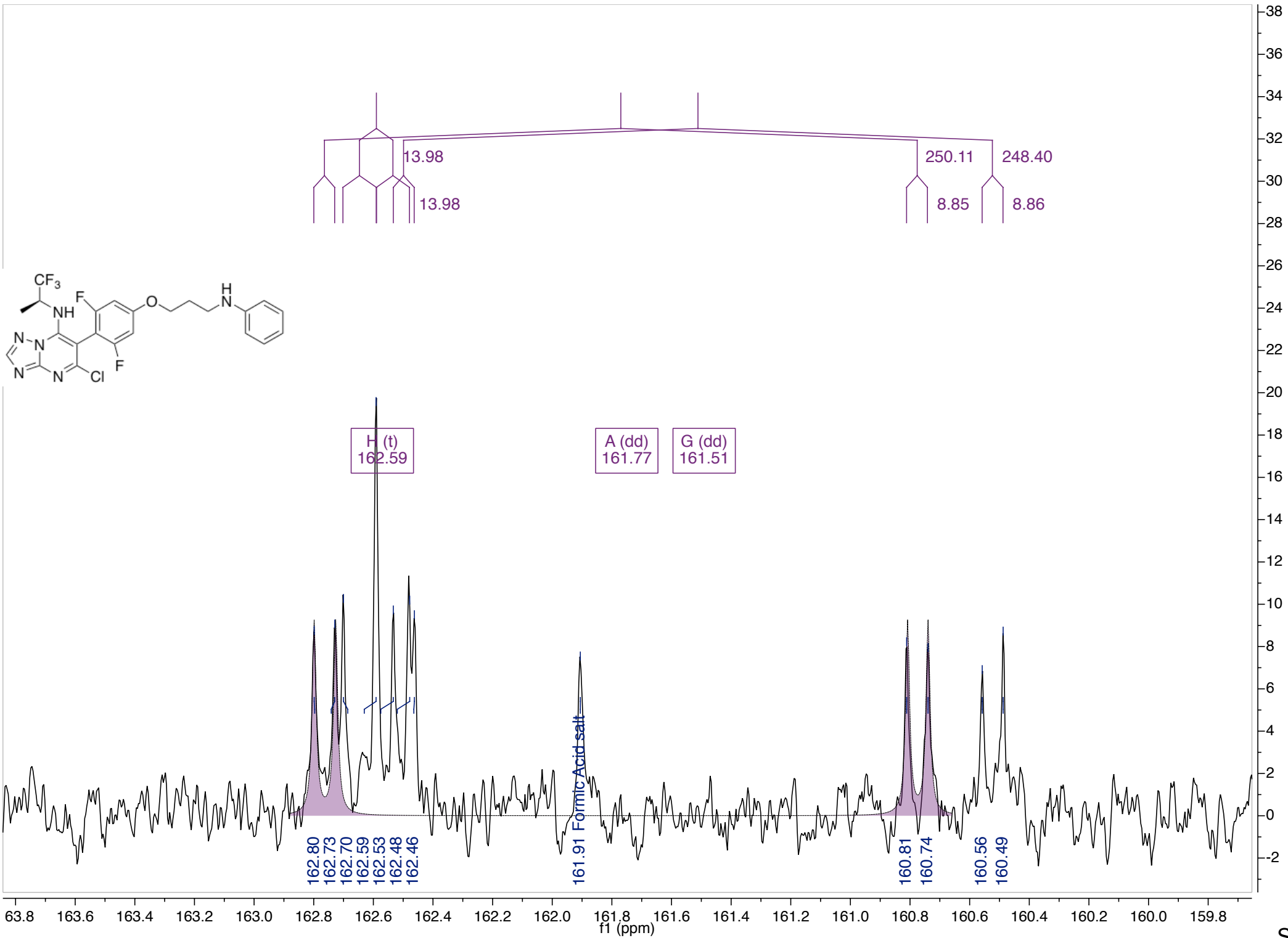




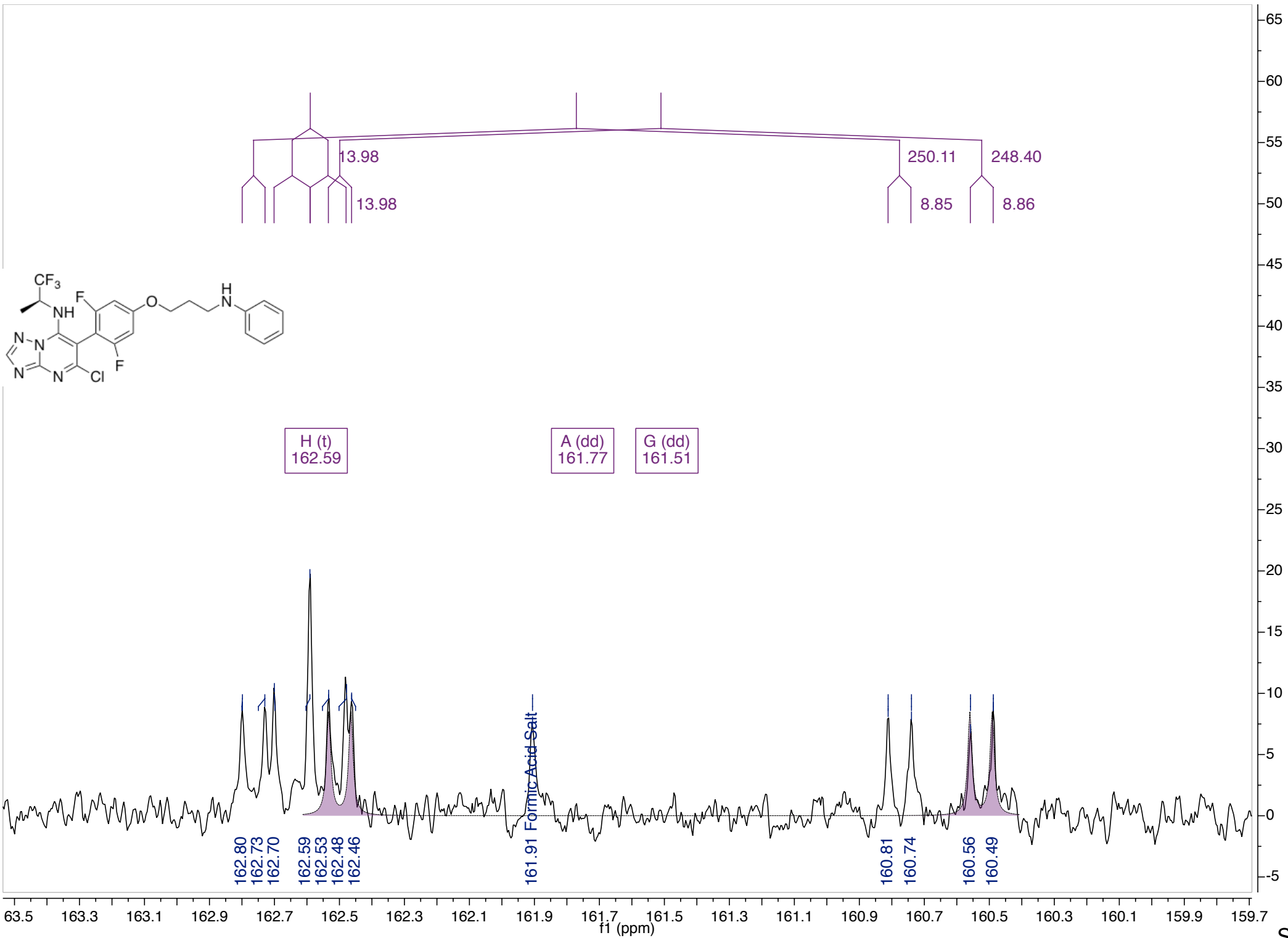


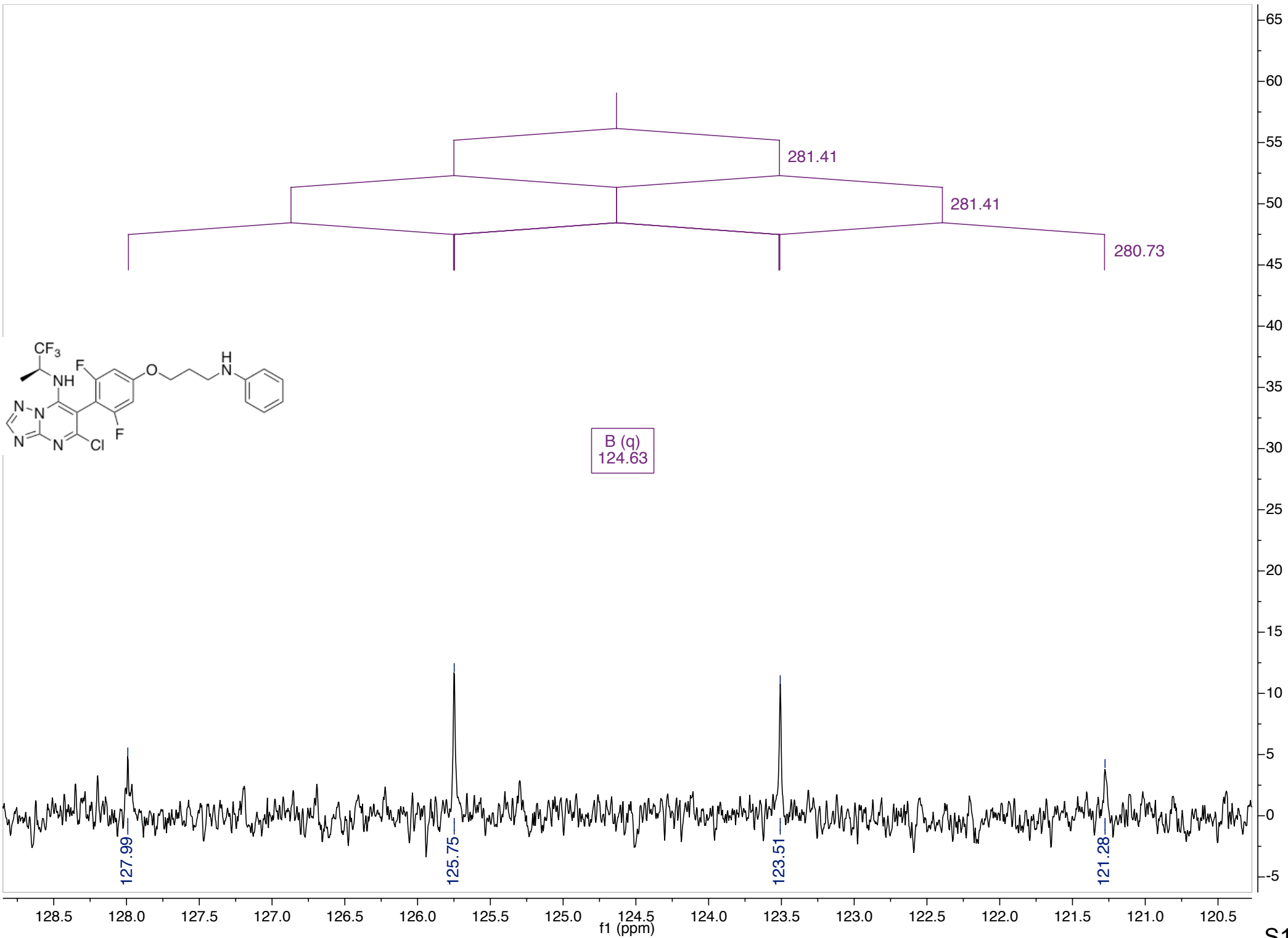


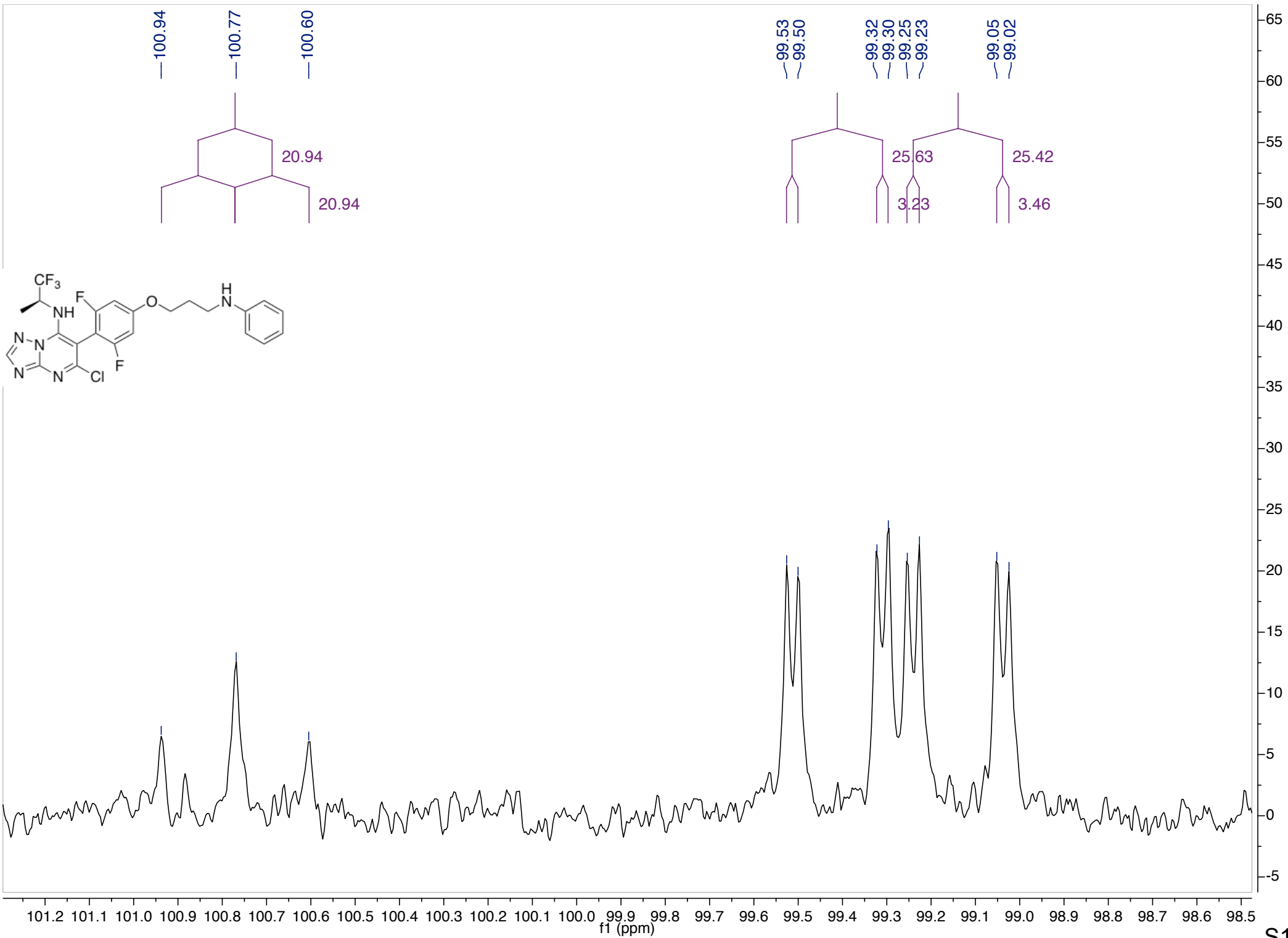


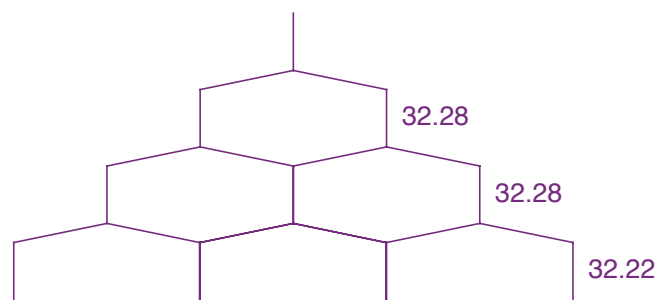
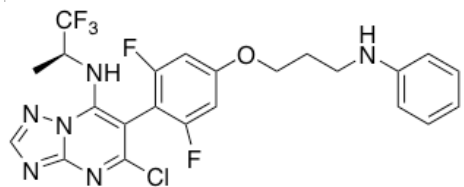




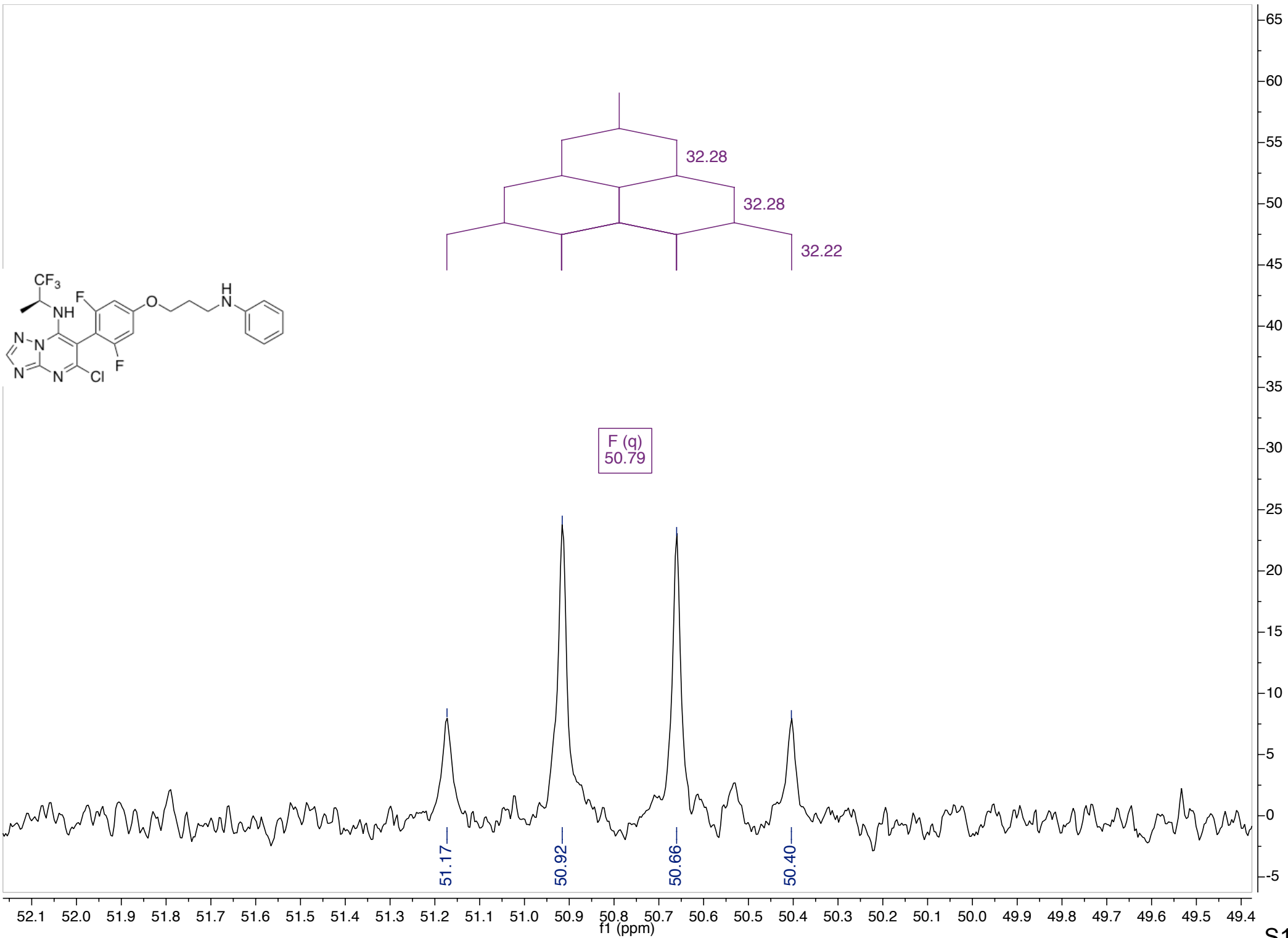


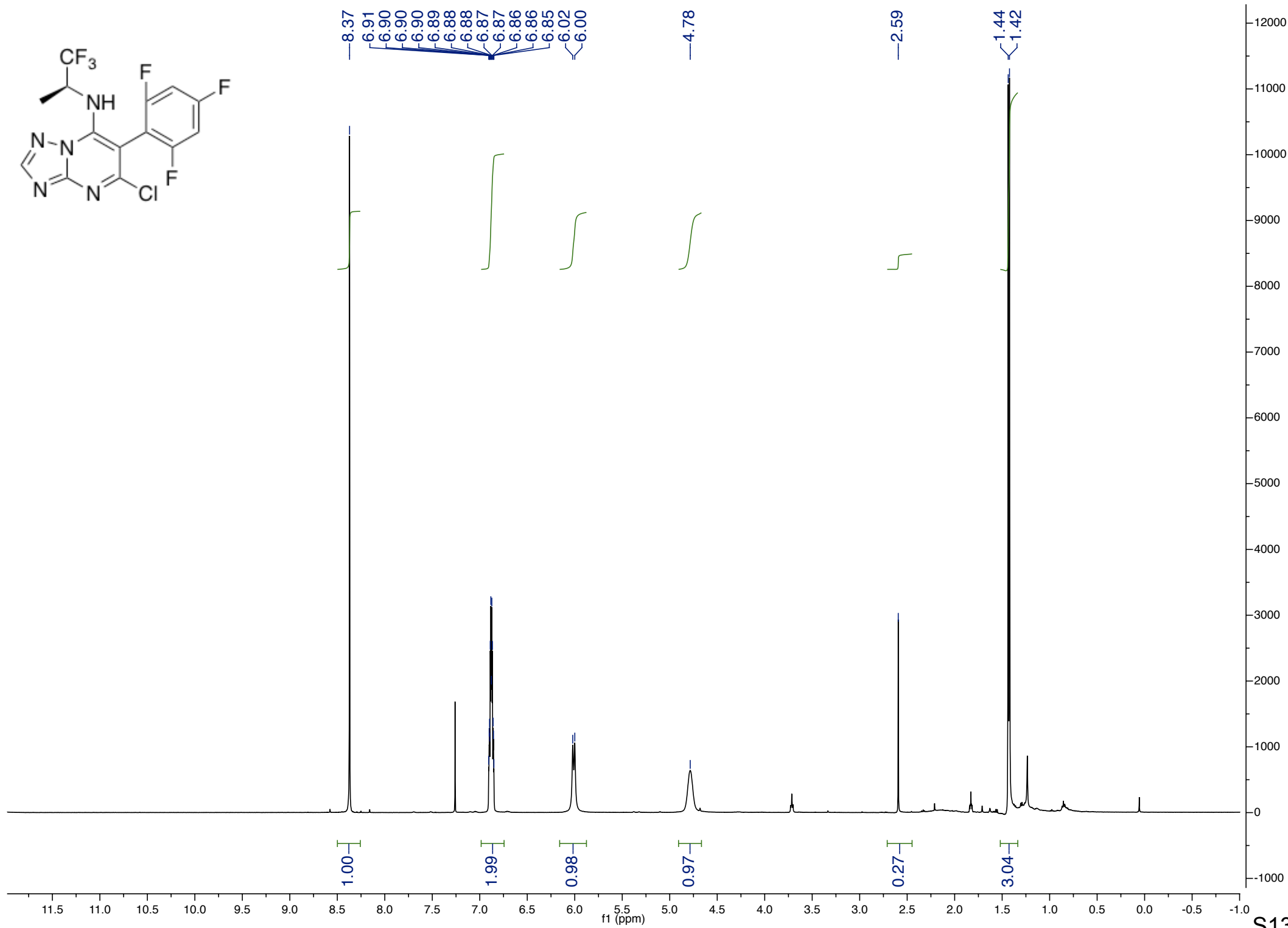
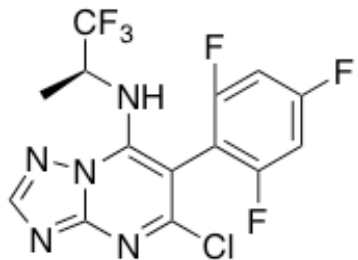


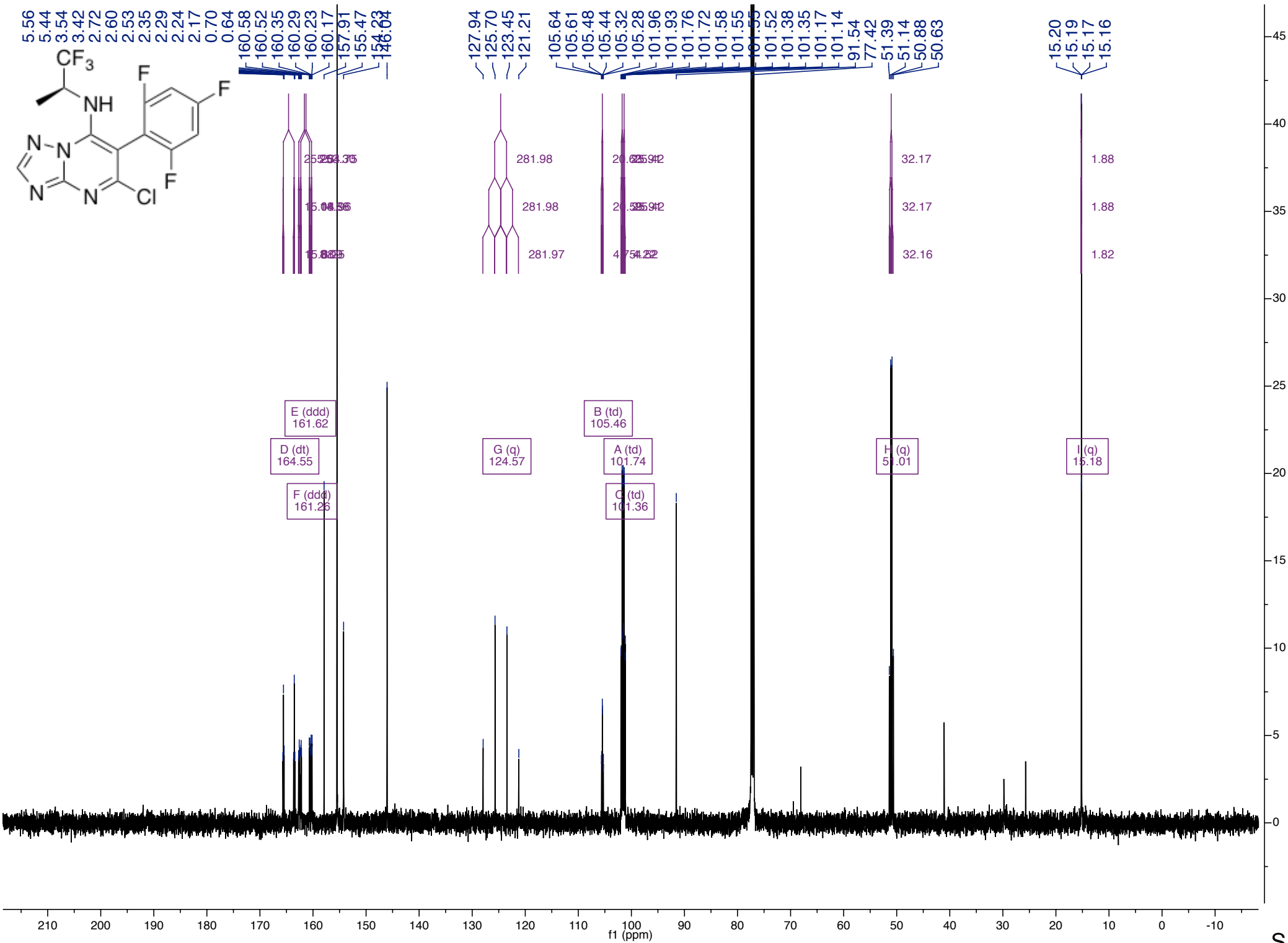
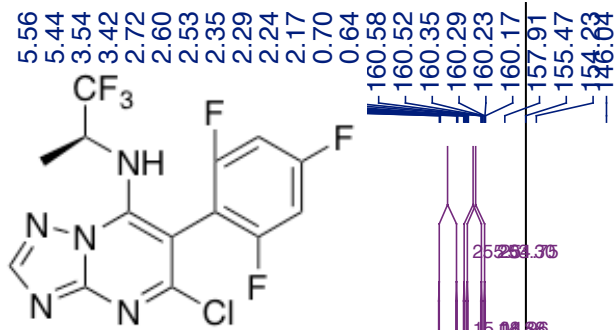


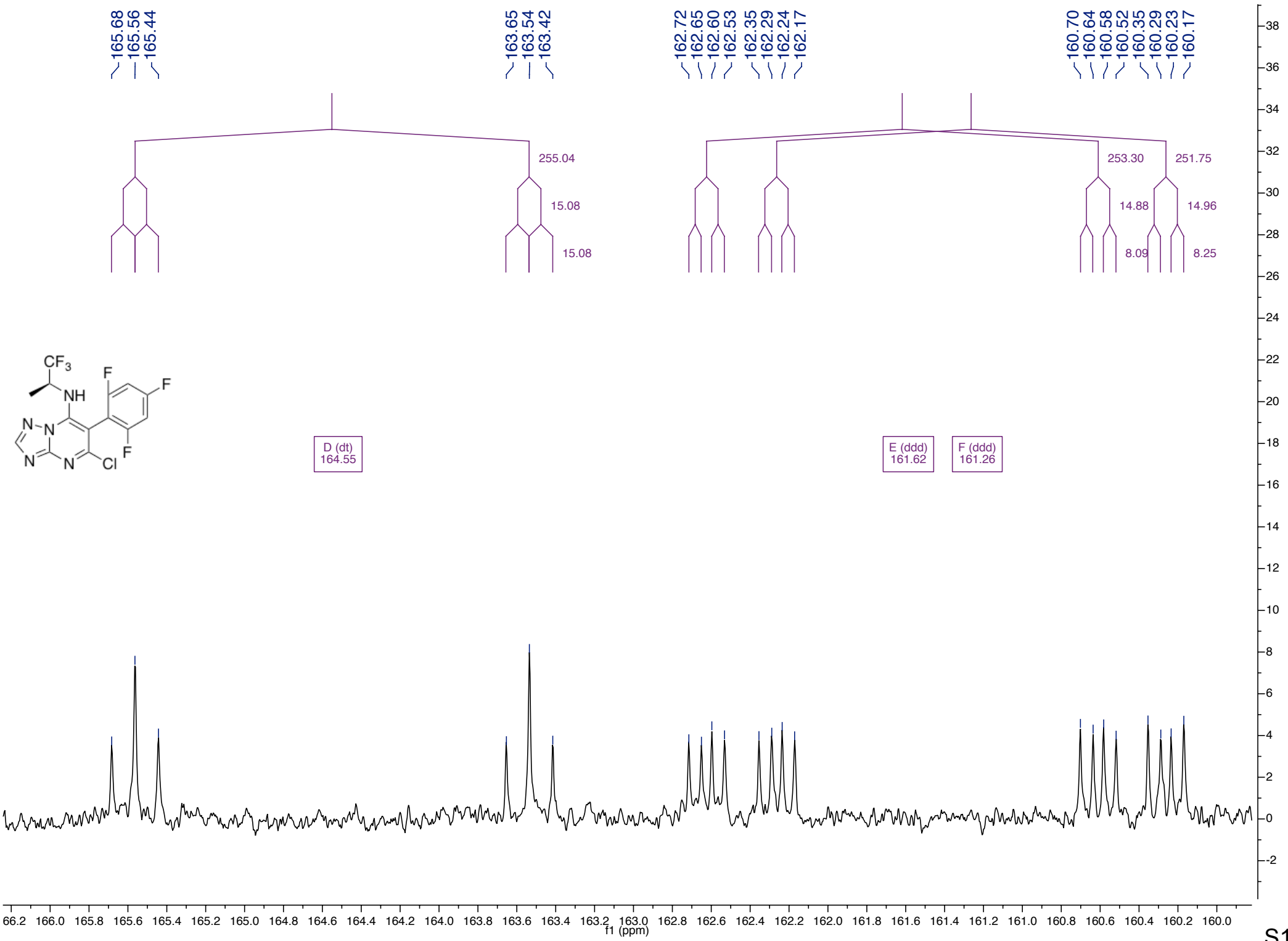


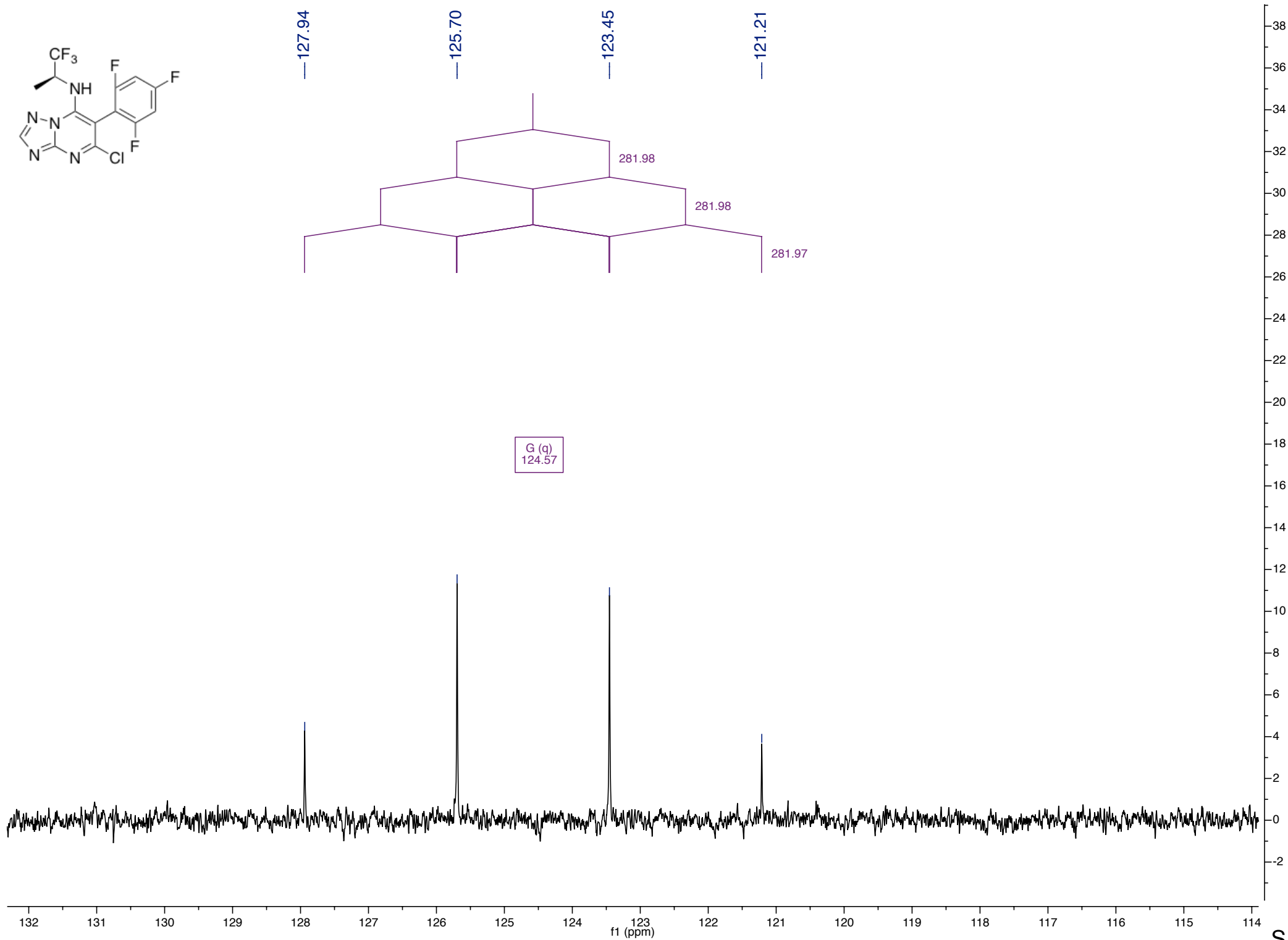
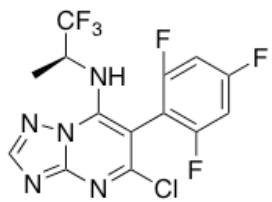
F (q)  
50.79





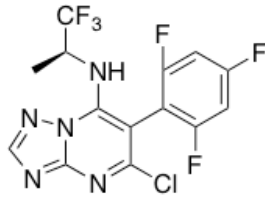
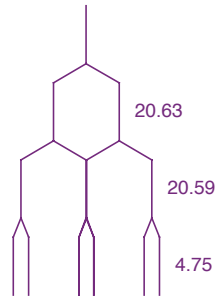




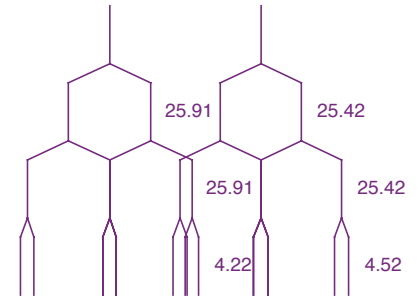




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105.61  
105.48  
105.44  
105.32  
105.28



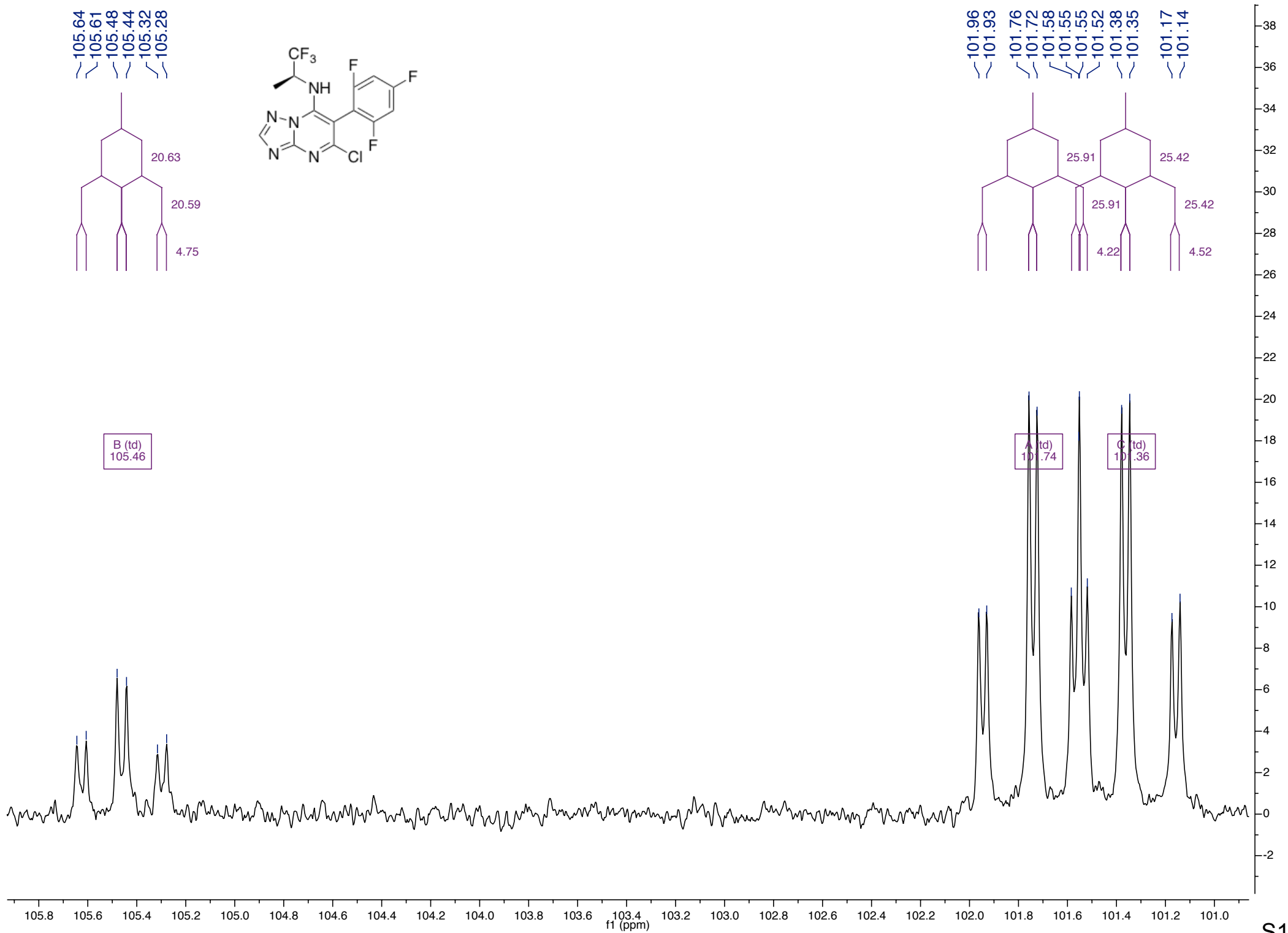
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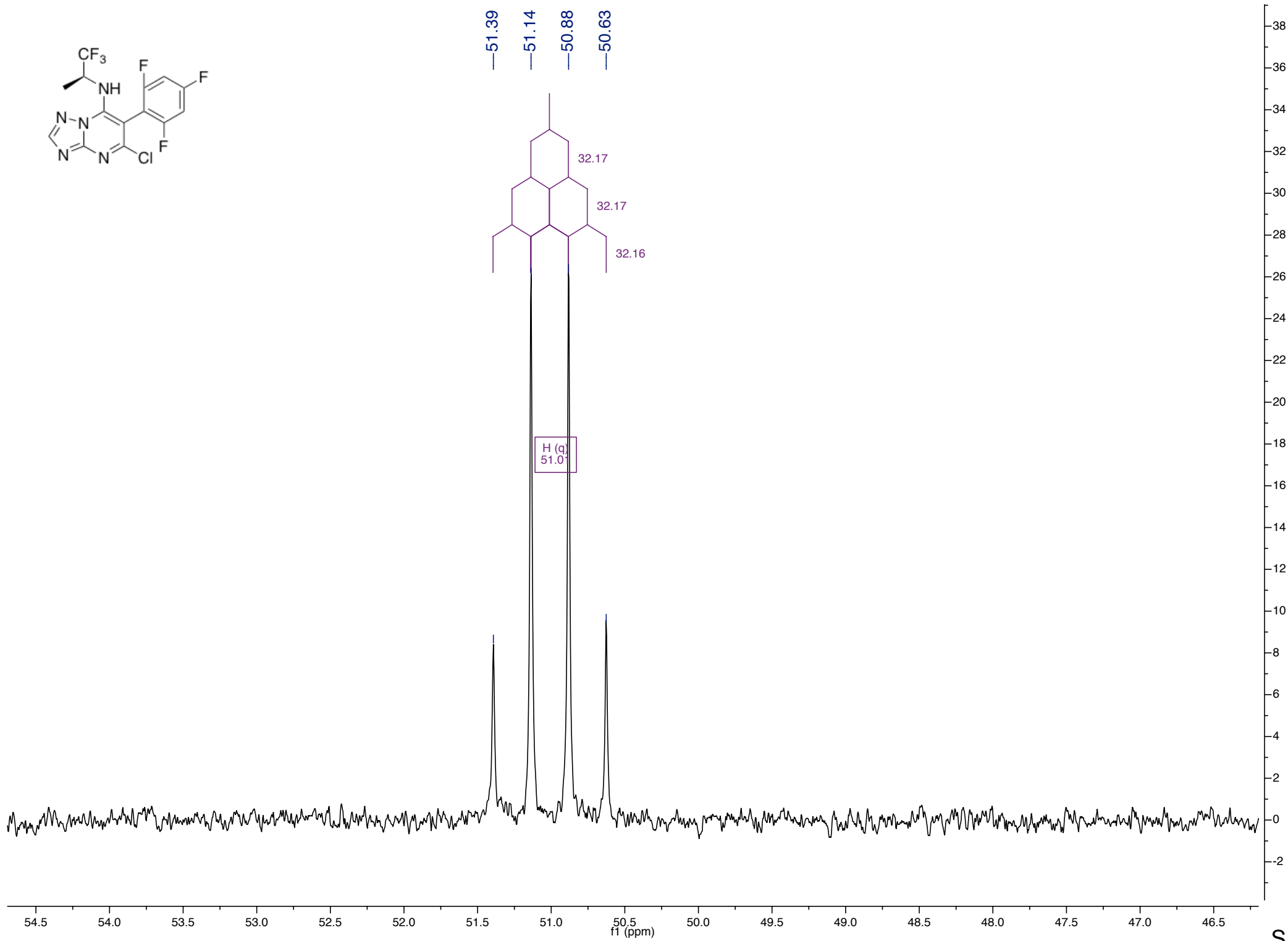
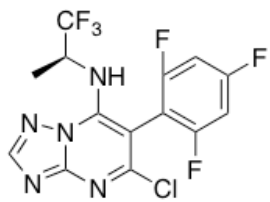


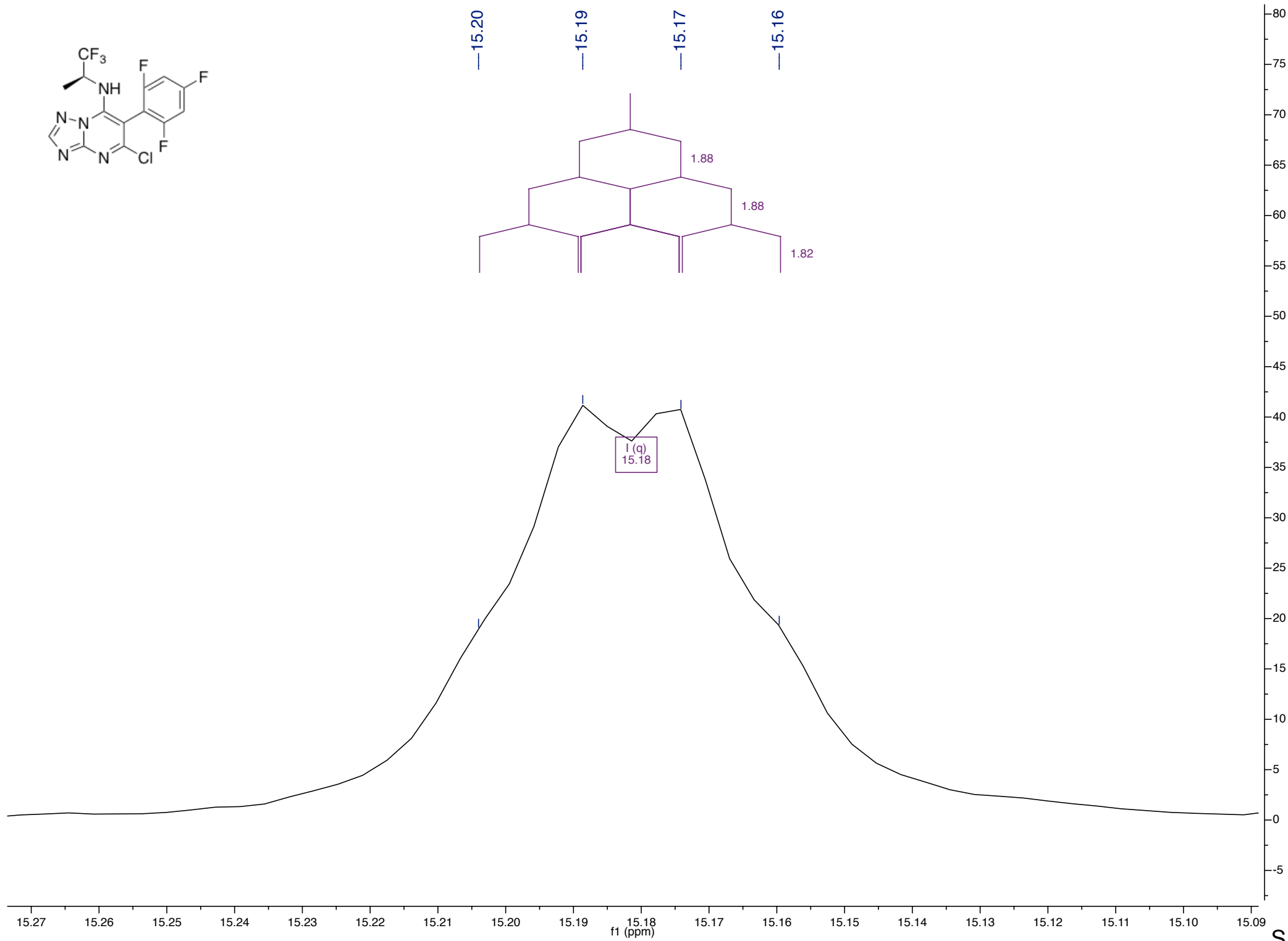
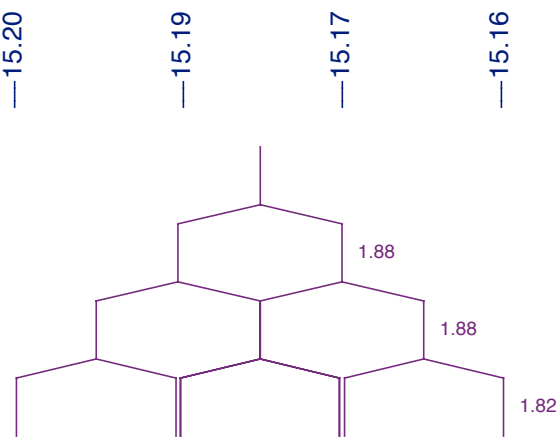
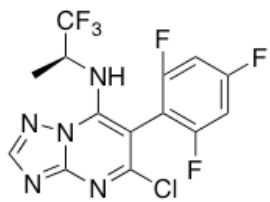
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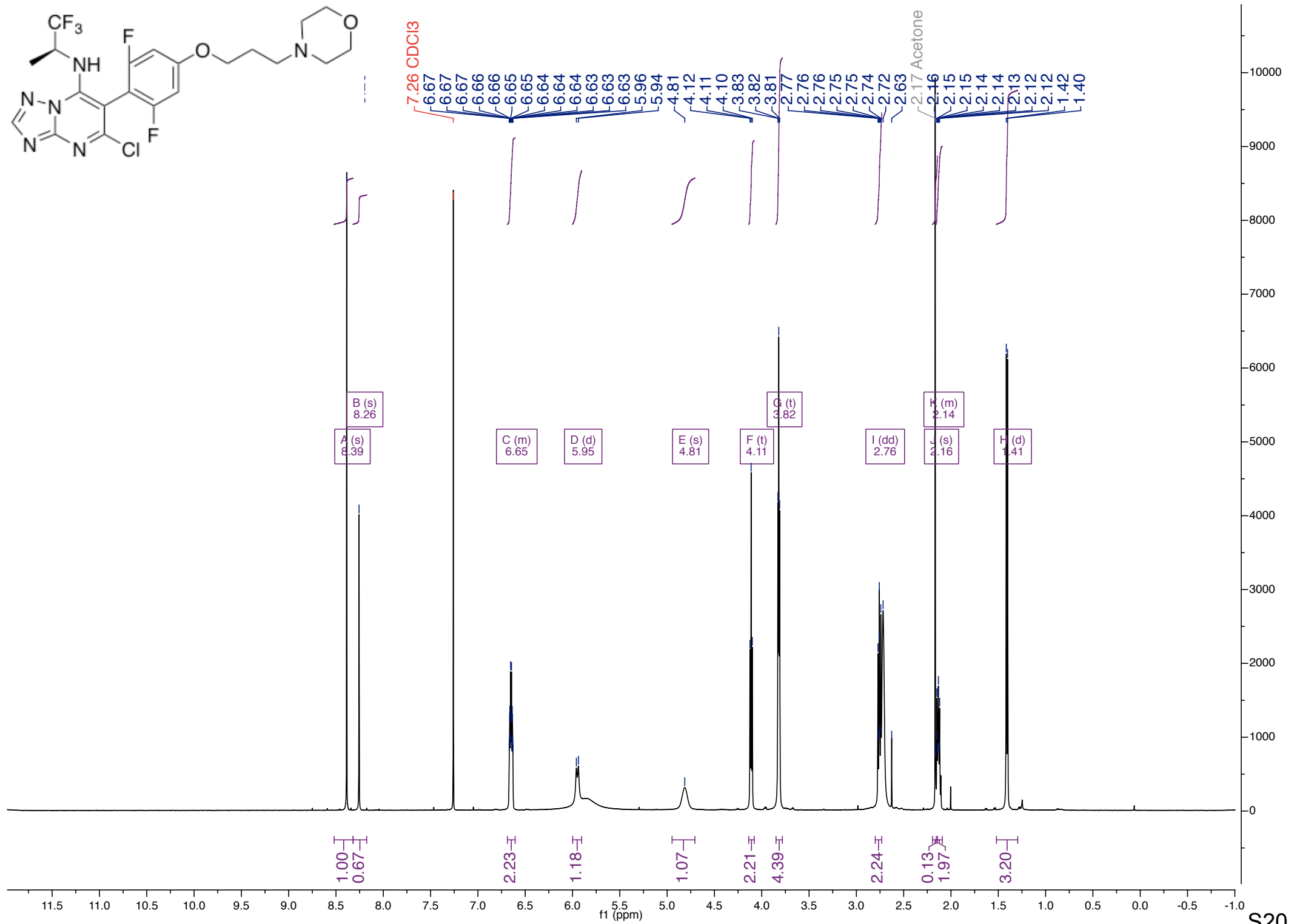
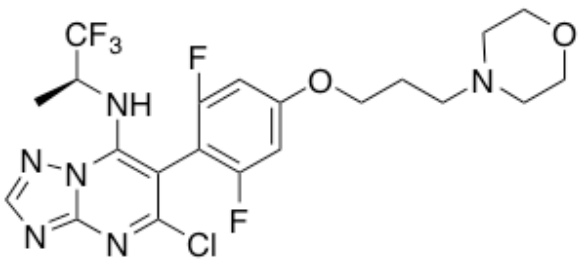
A (td)  
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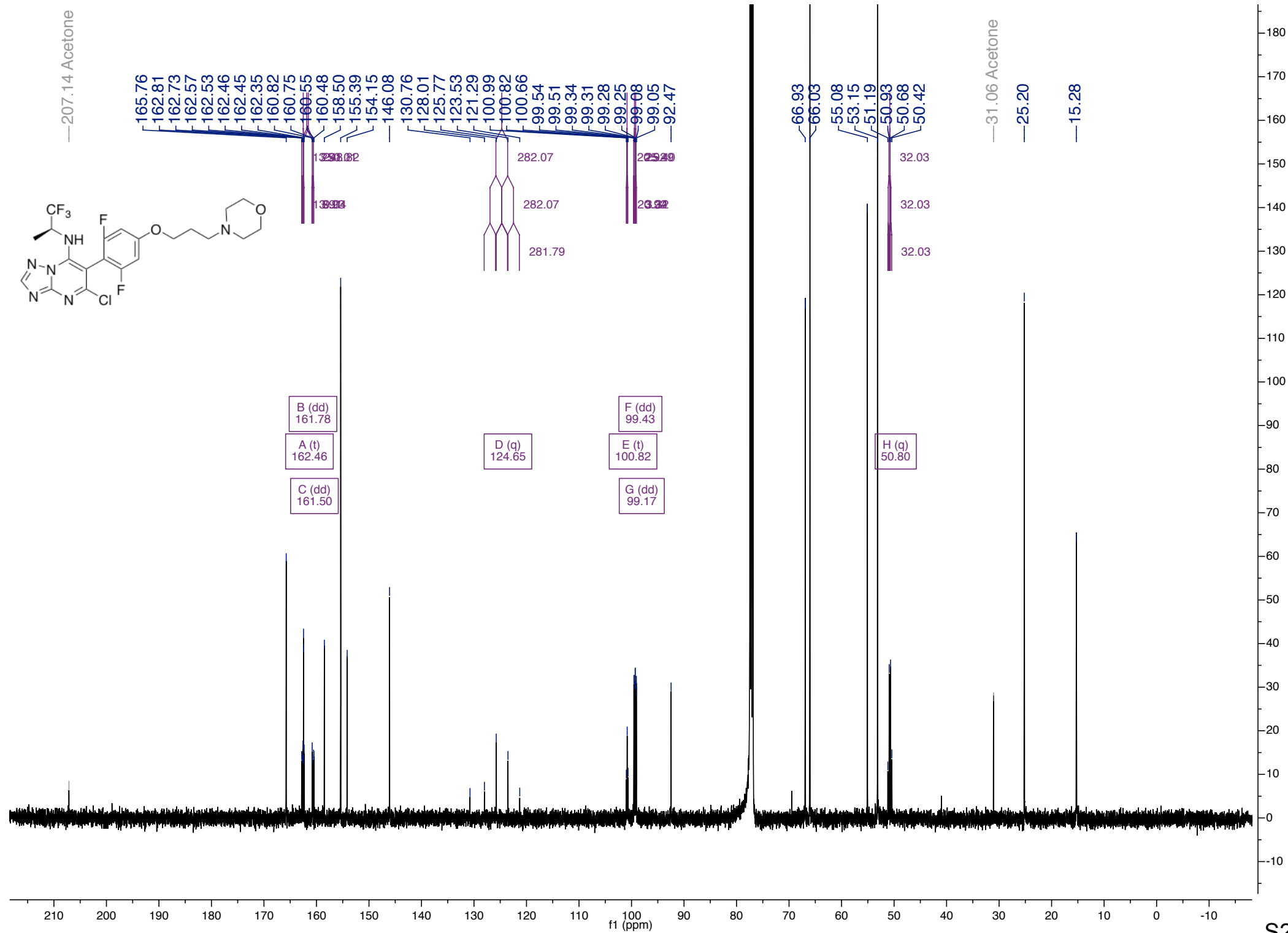
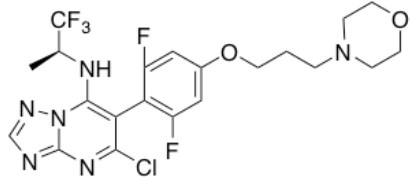
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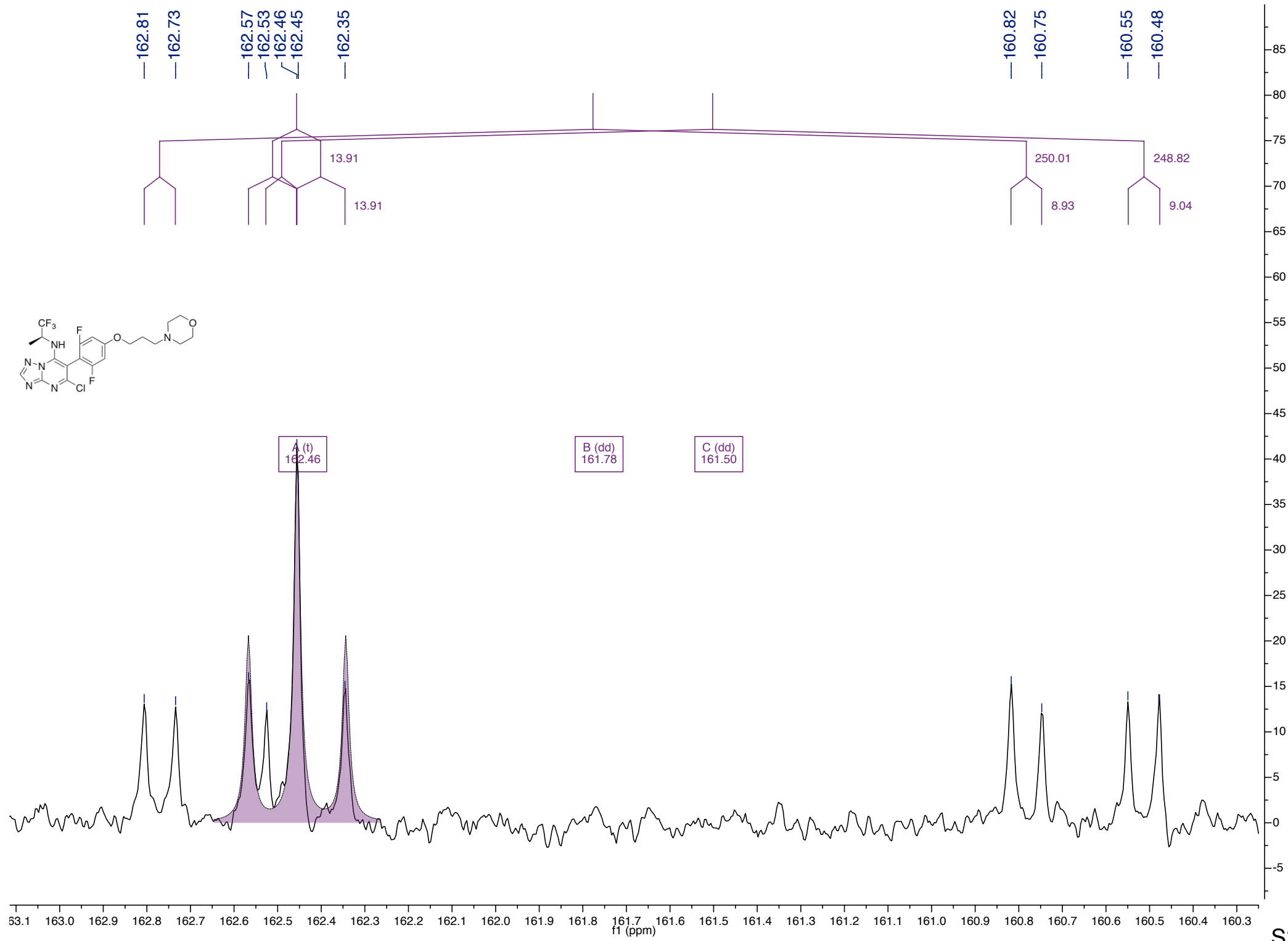


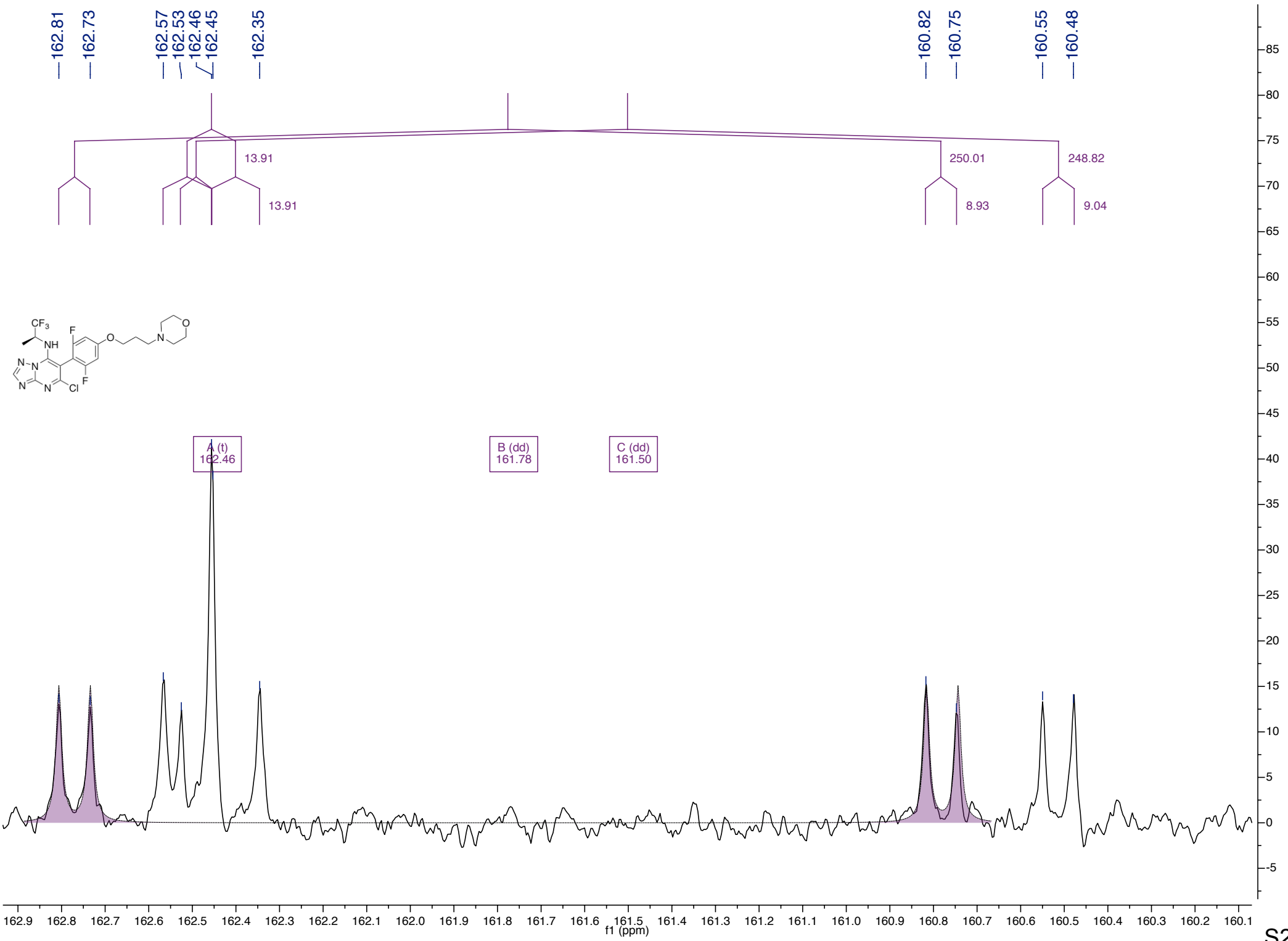








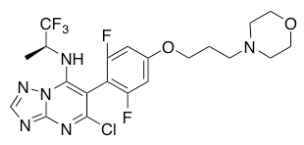
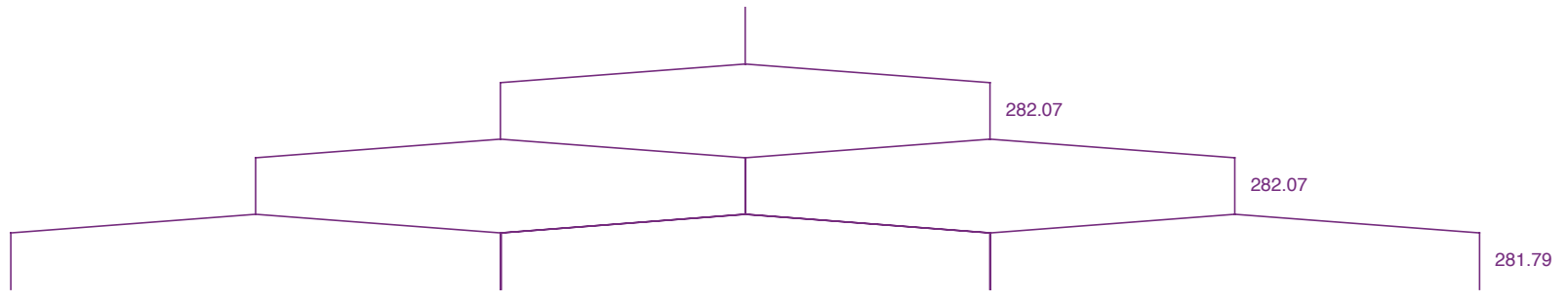








—128.01 —125.77 —123.53 —121.29

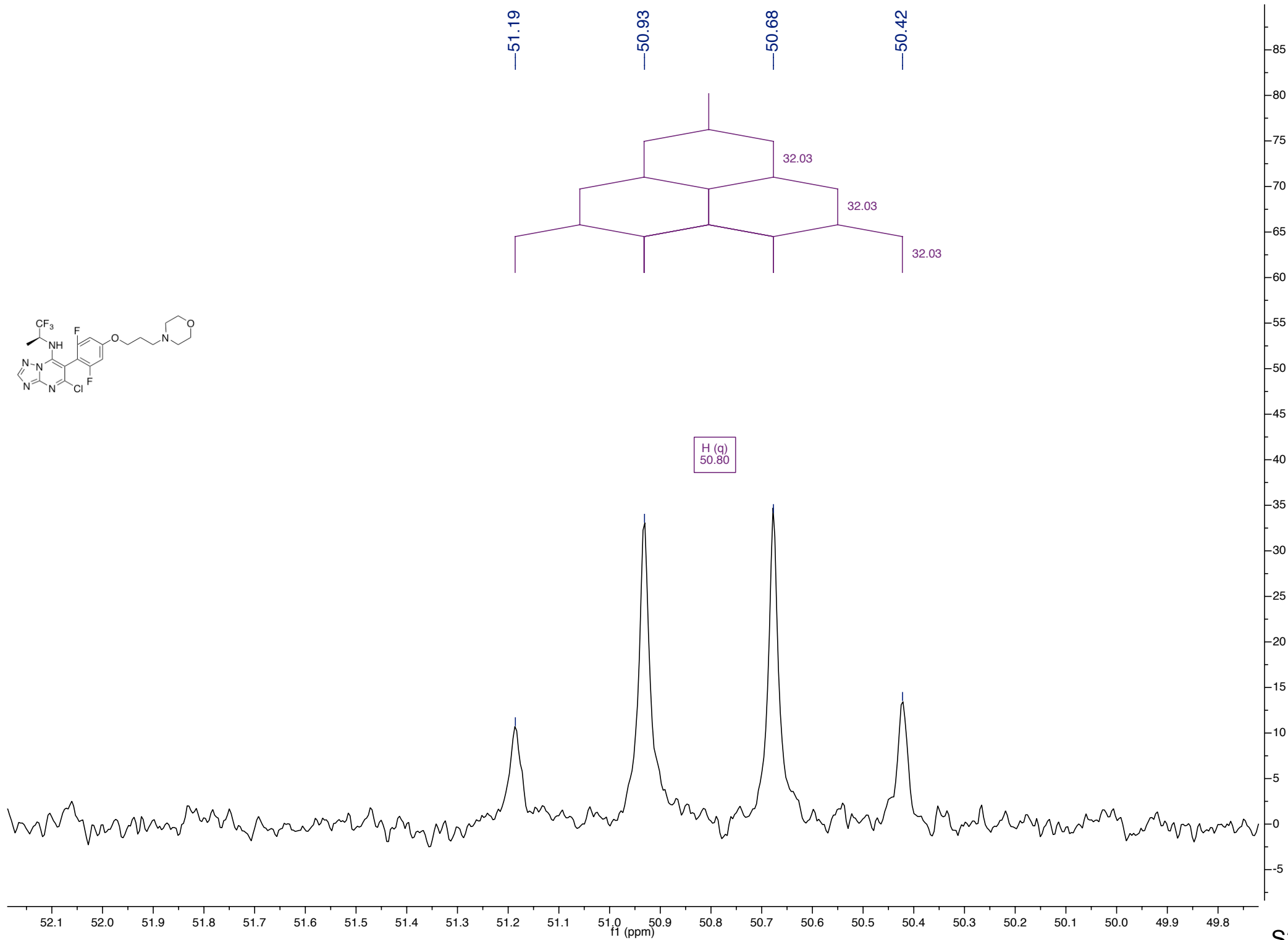
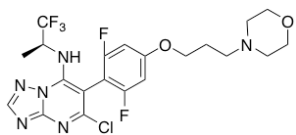


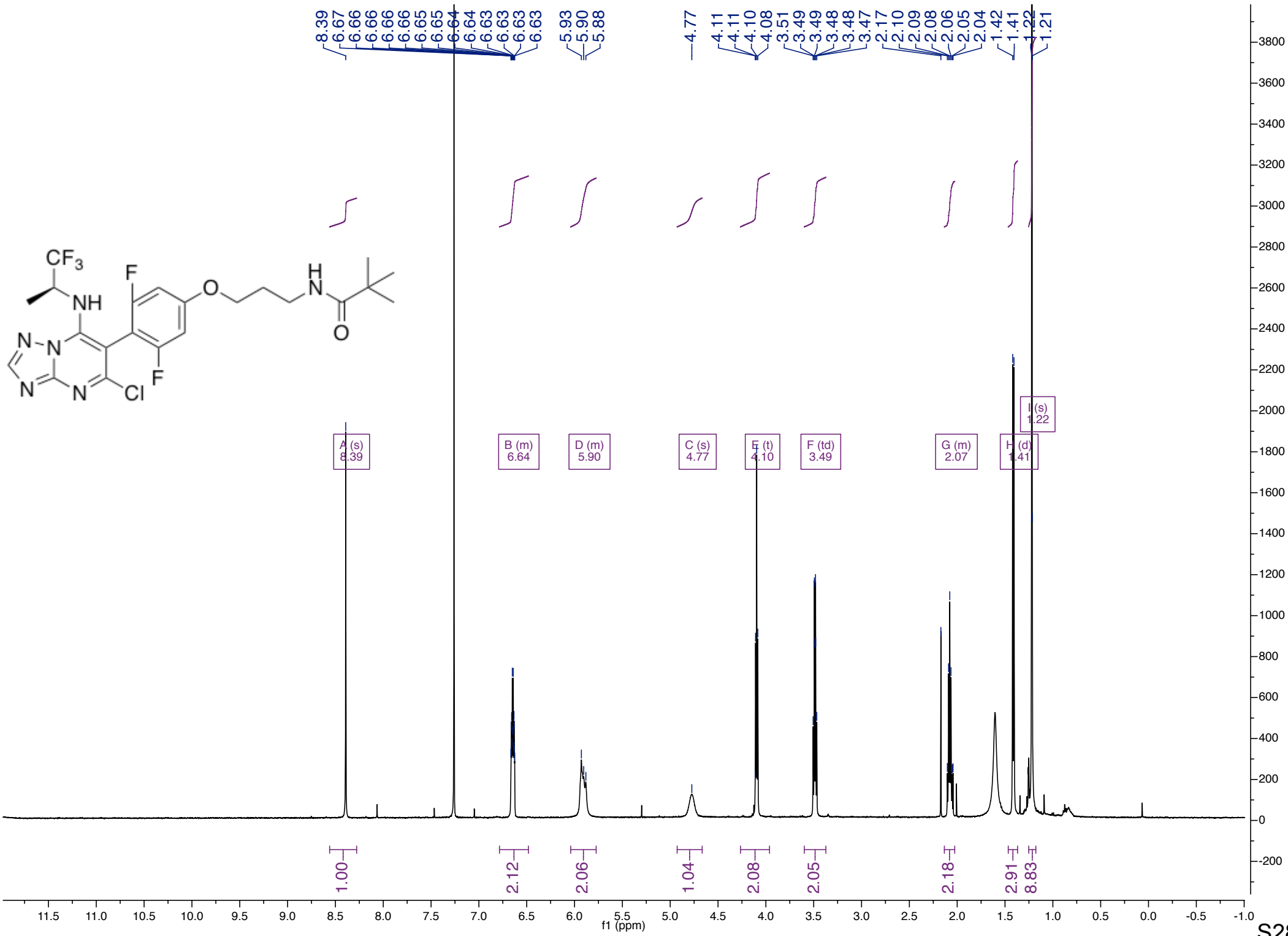
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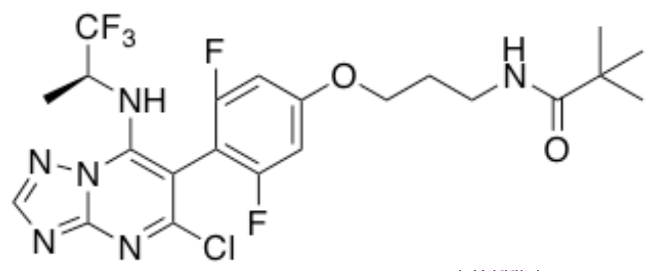
129.0 128.5 128.0 127.5 127.0 126.5 126.0 125.5 125.0 124.5 124.0 123.5 123.0 122.5 122.0 121.5 121.0 120.5  
f1 (ppm)

85  
80  
75  
70  
65  
60  
55  
50  
45  
40  
35  
30  
25  
20  
15  
10  
5  
0  
-5



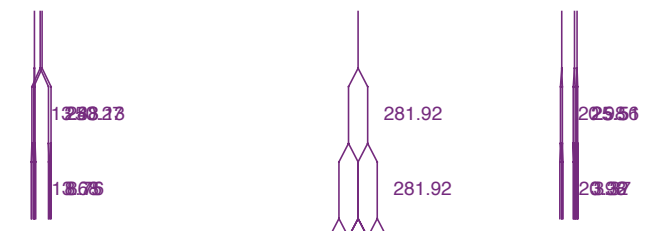






178.92  
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160.90  
160.83  
160.64  
160.57  
158.55  
155.52  
154.20  
146.08

128.05  
125.81  
123.57  
121.33  
101.14  
100.97  
100.80  
99.57  
99.55  
99.37  
99.34  
99.27  
99.24  
99.07  
99.04  
92.42



B (dd)  
161.86

A (t)  
162.51

C (dd)  
161.59

D (q)  
124.69

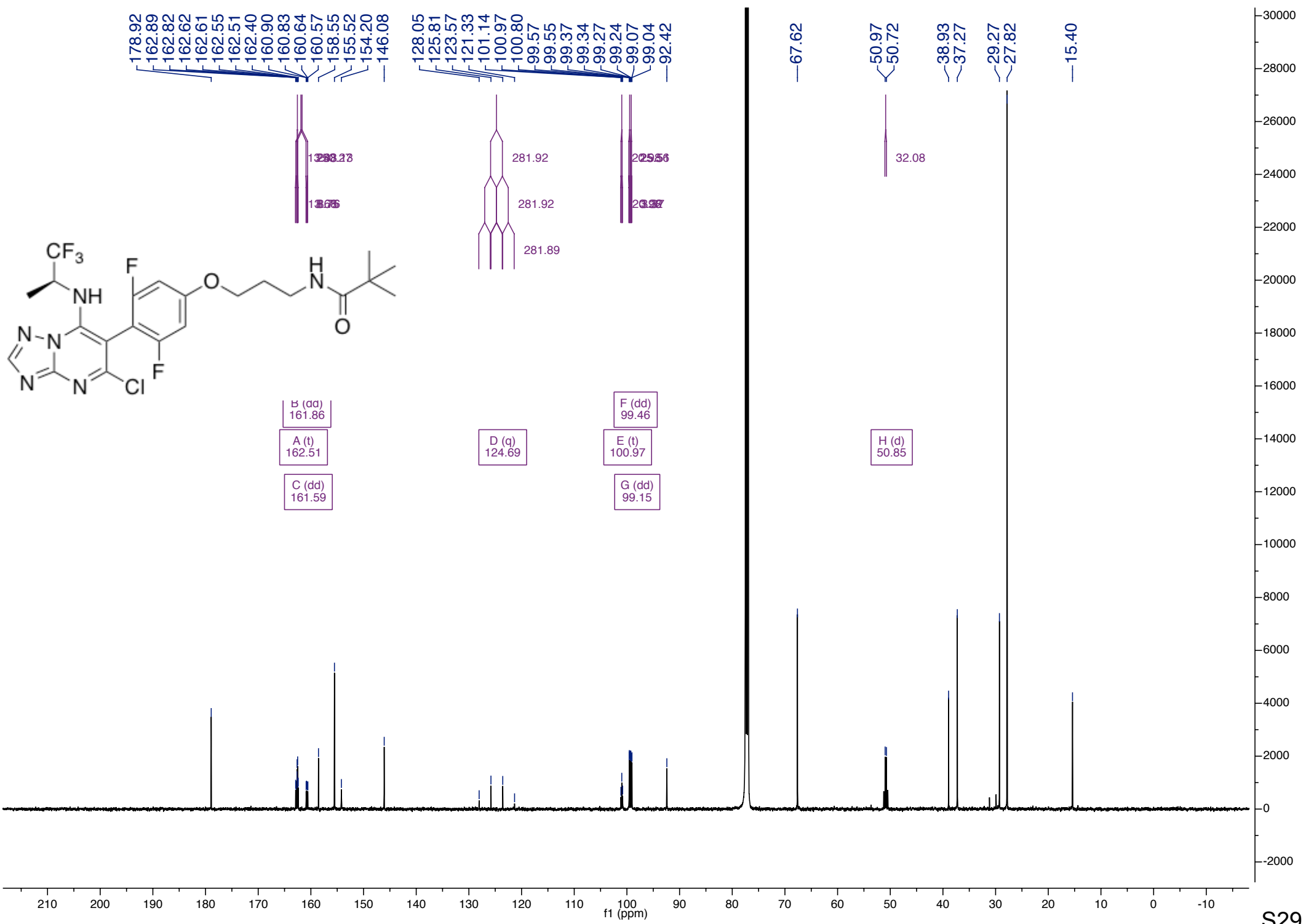
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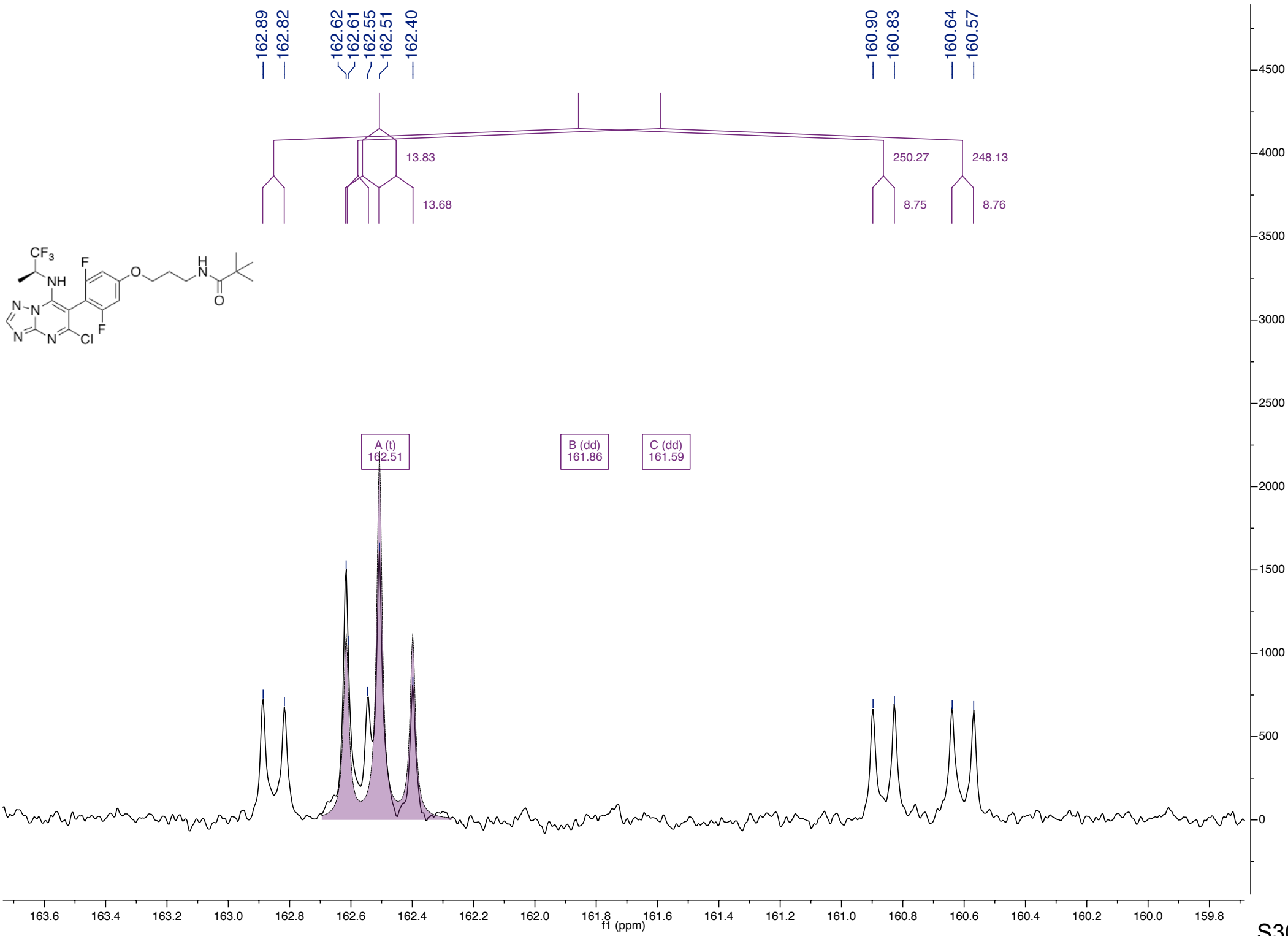
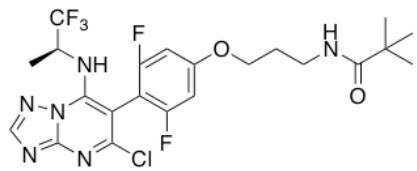
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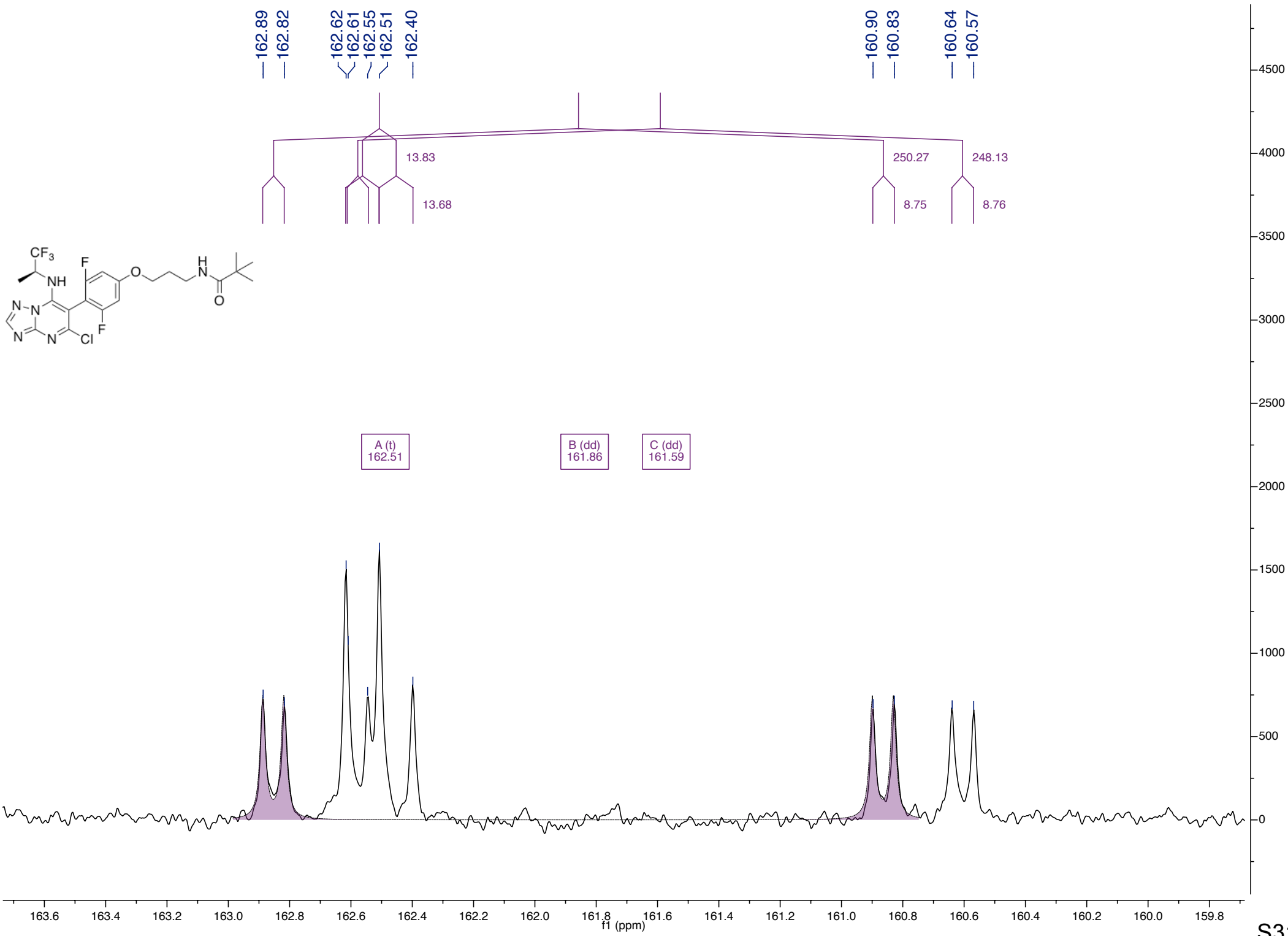
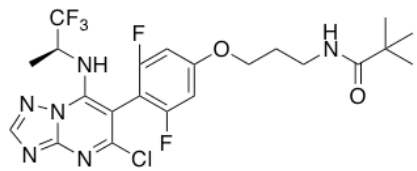
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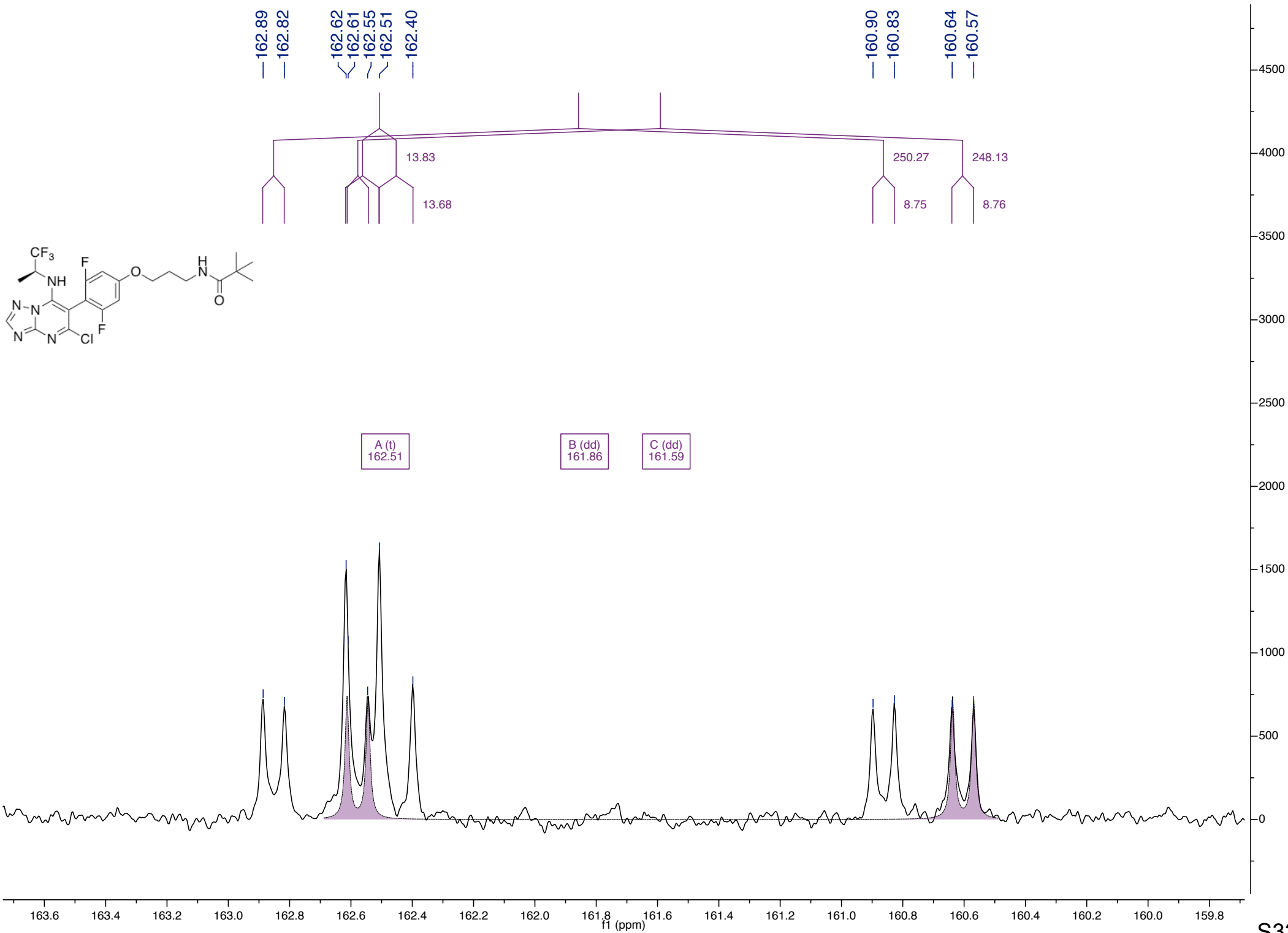
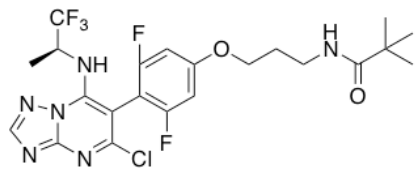
H (d)  
50.85

67.62  
50.97  
50.72  
38.93  
37.27  
29.27  
27.82  
15.40

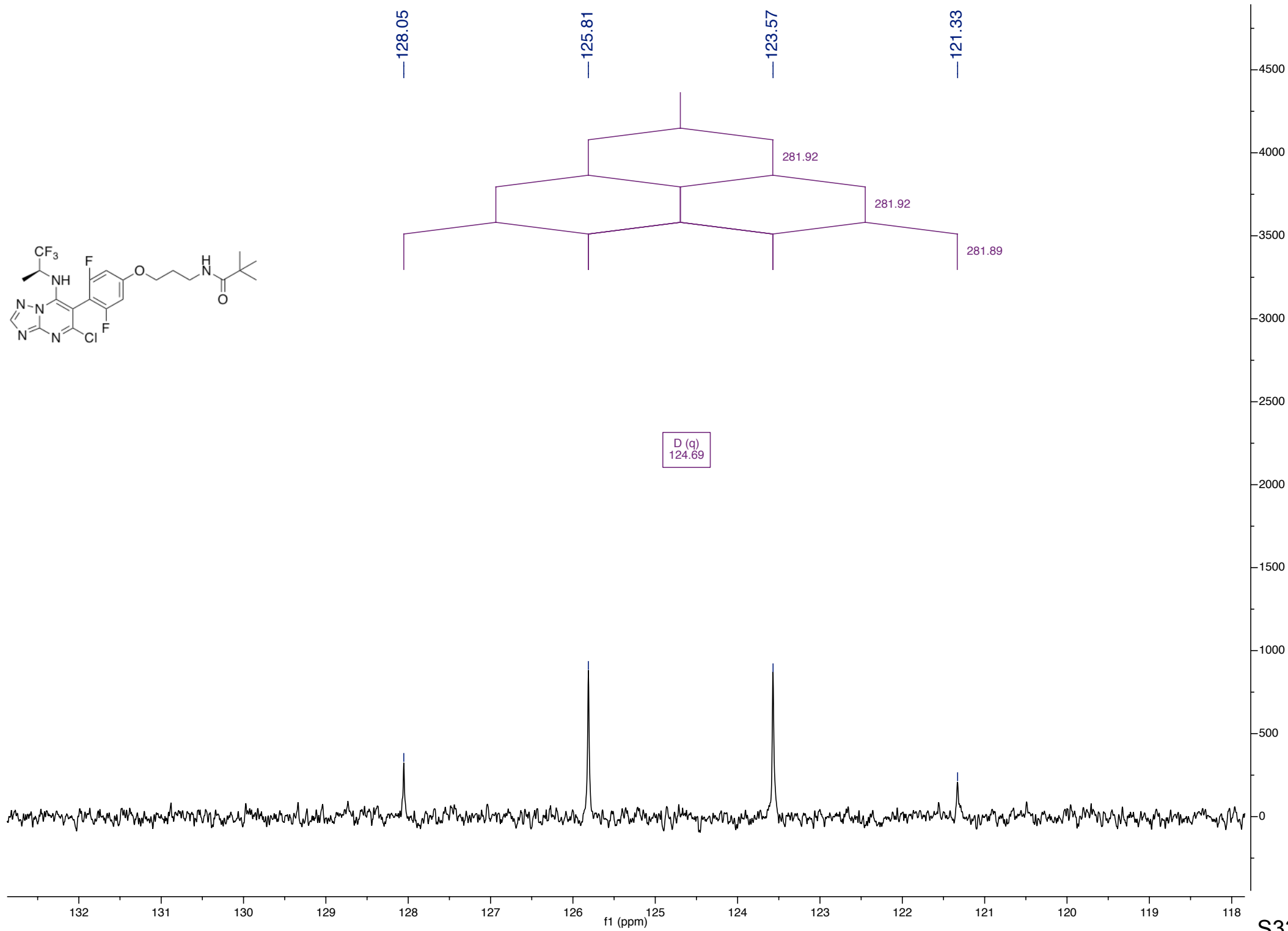
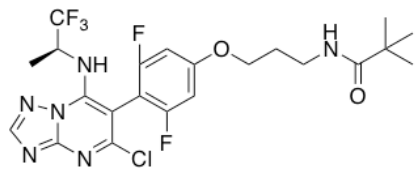


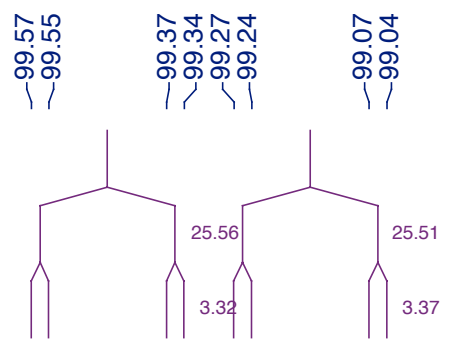
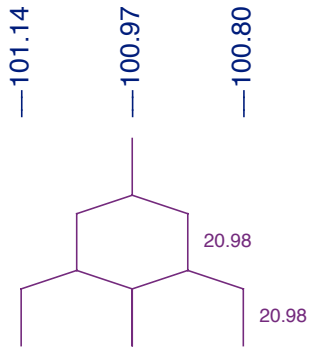
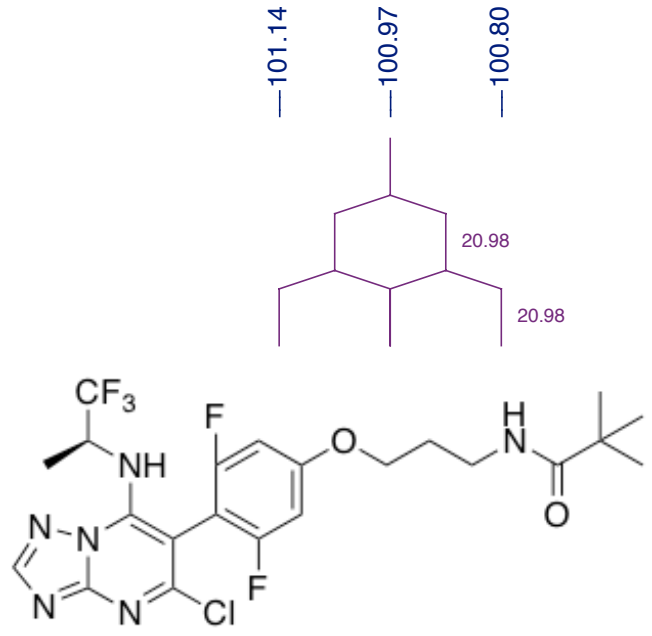








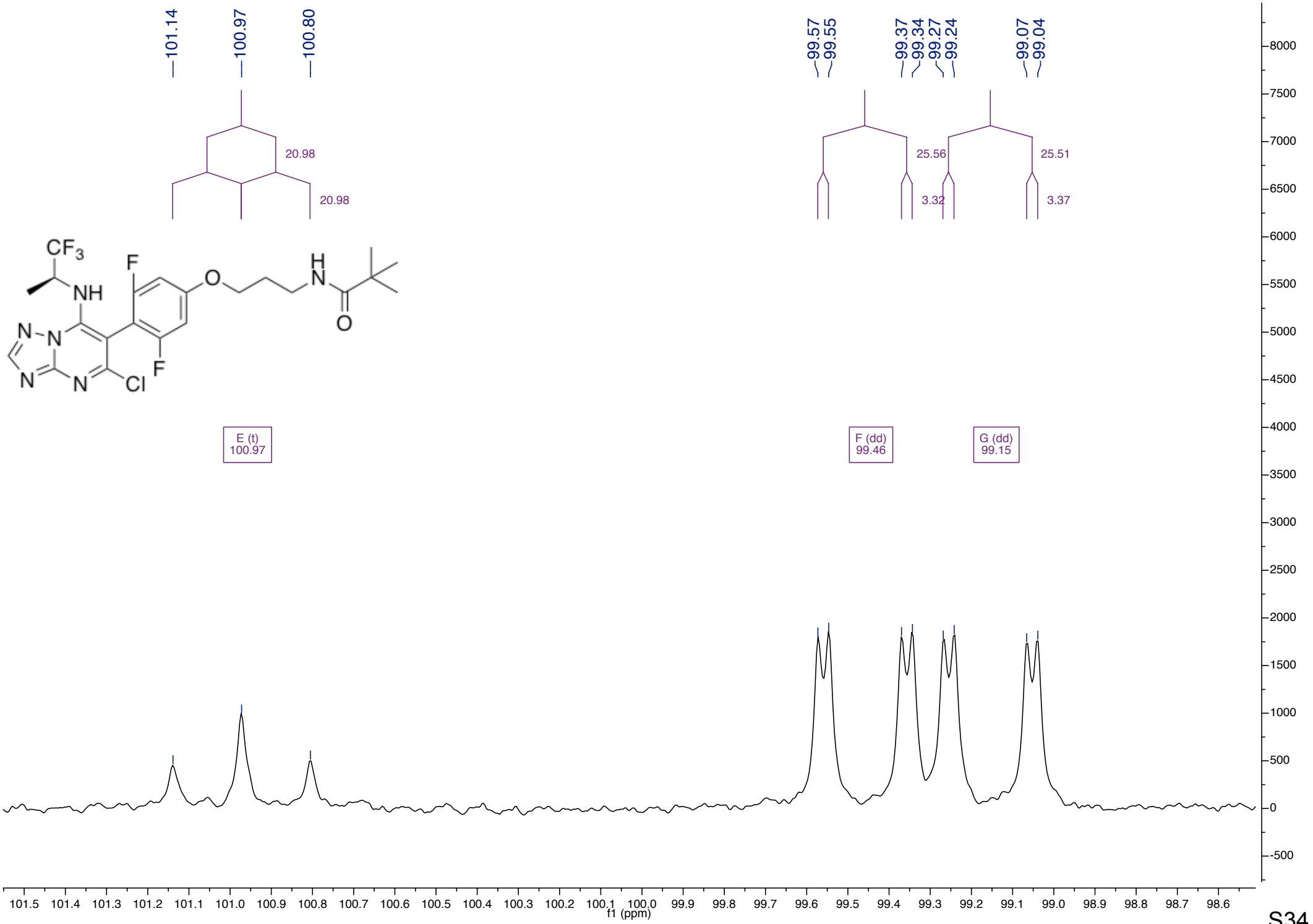


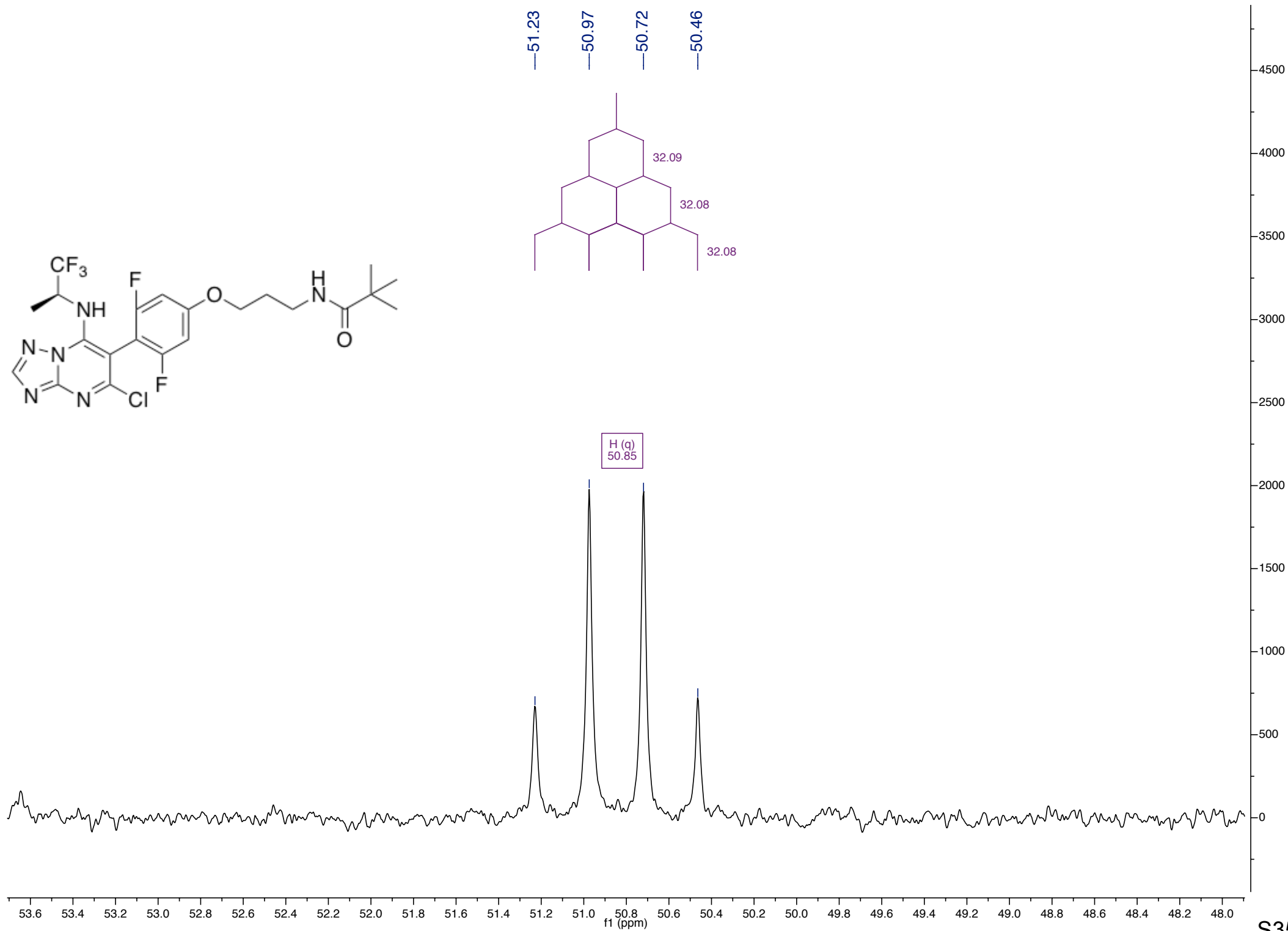
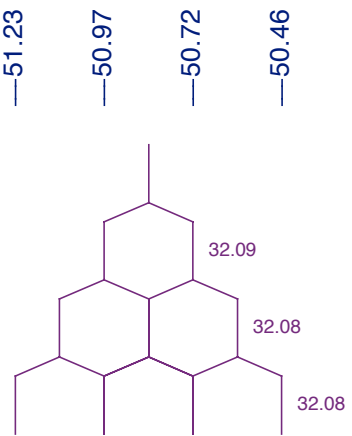
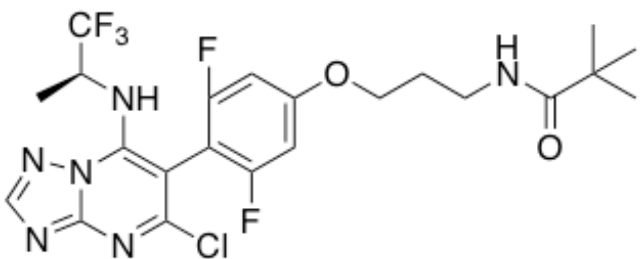


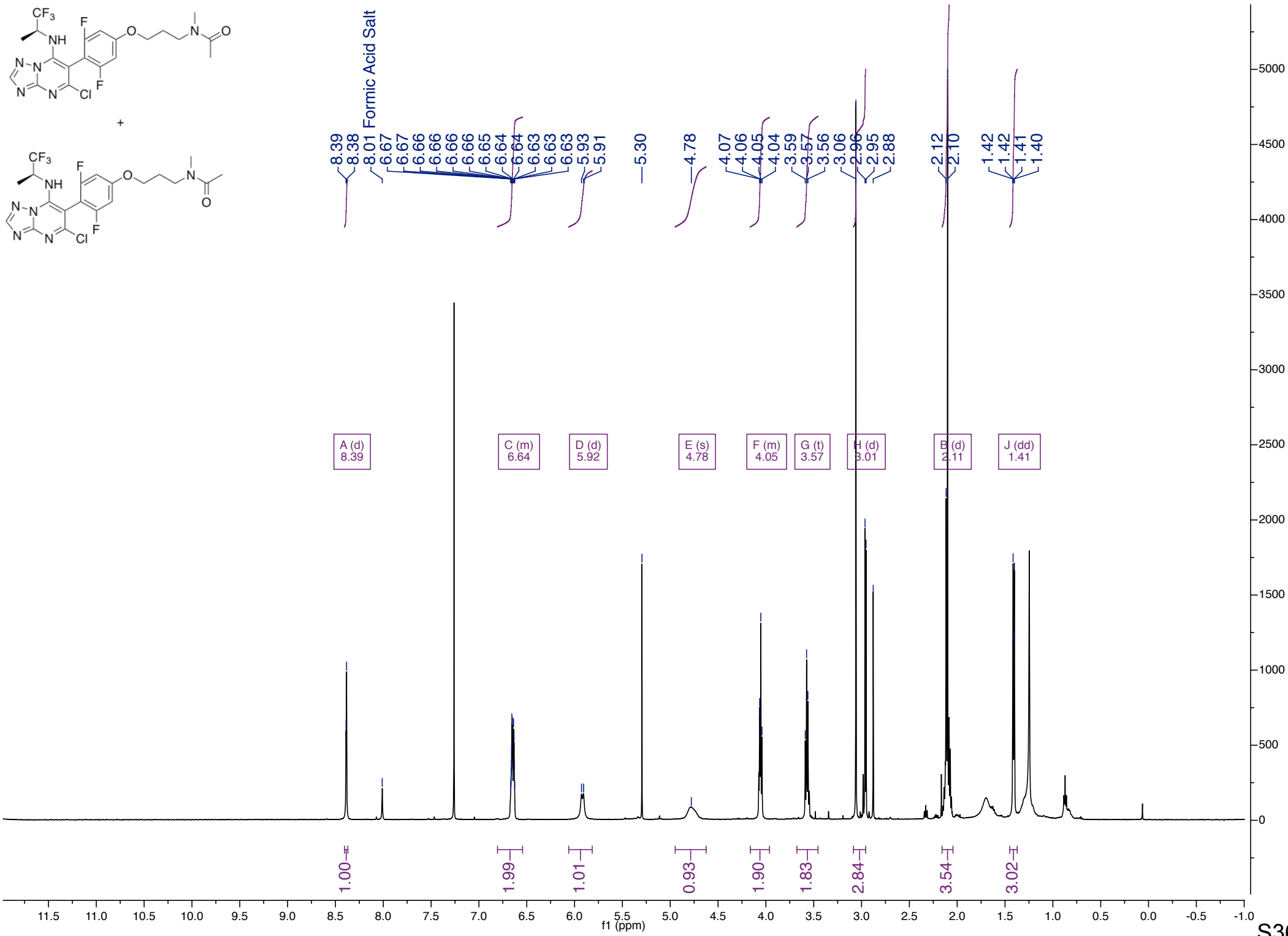
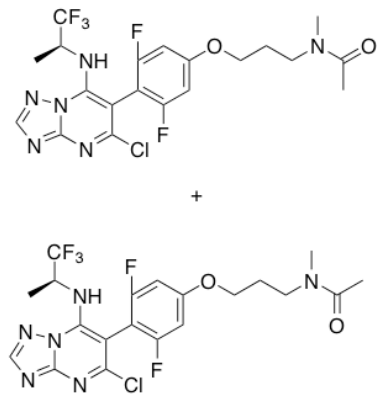
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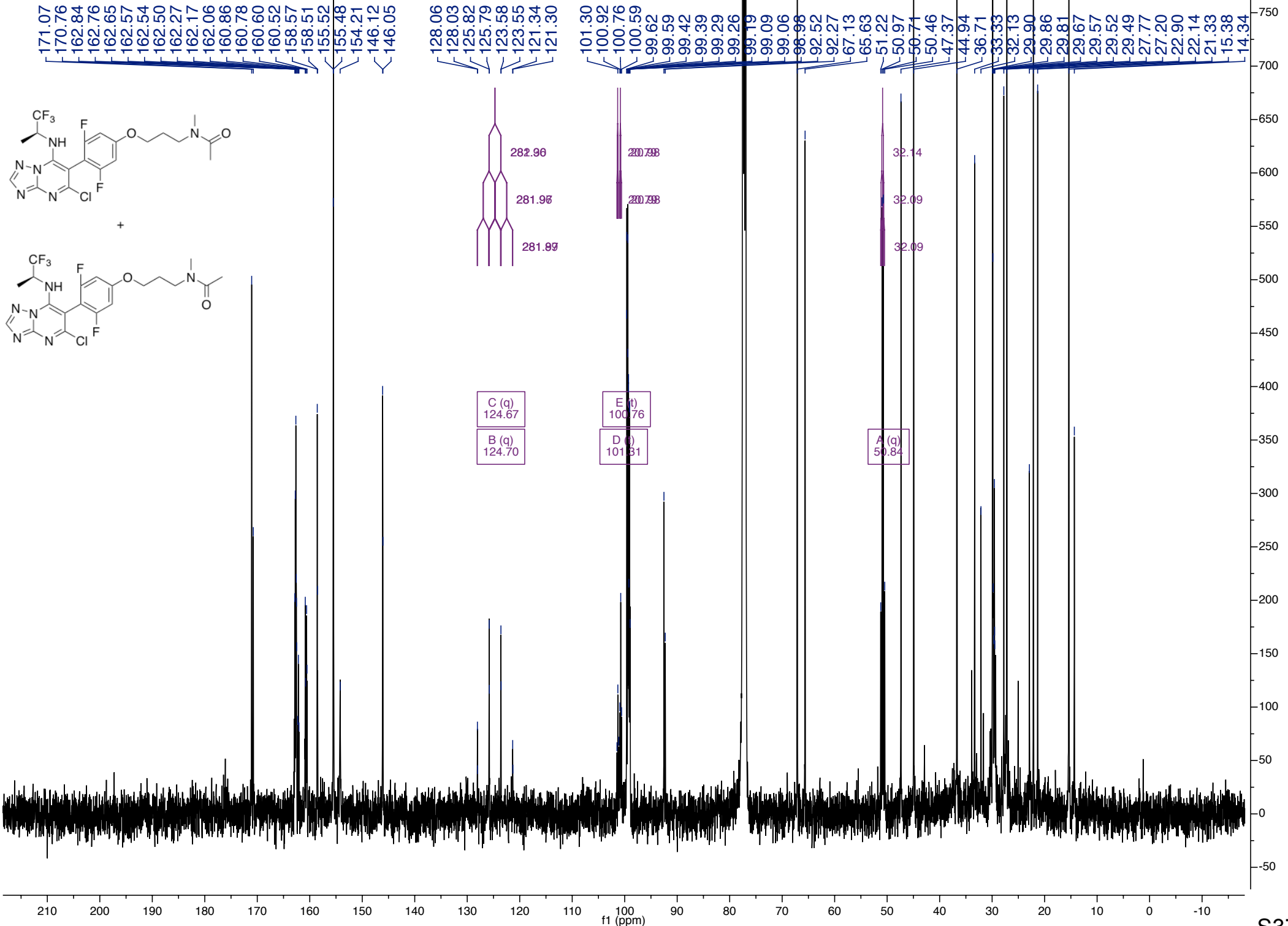
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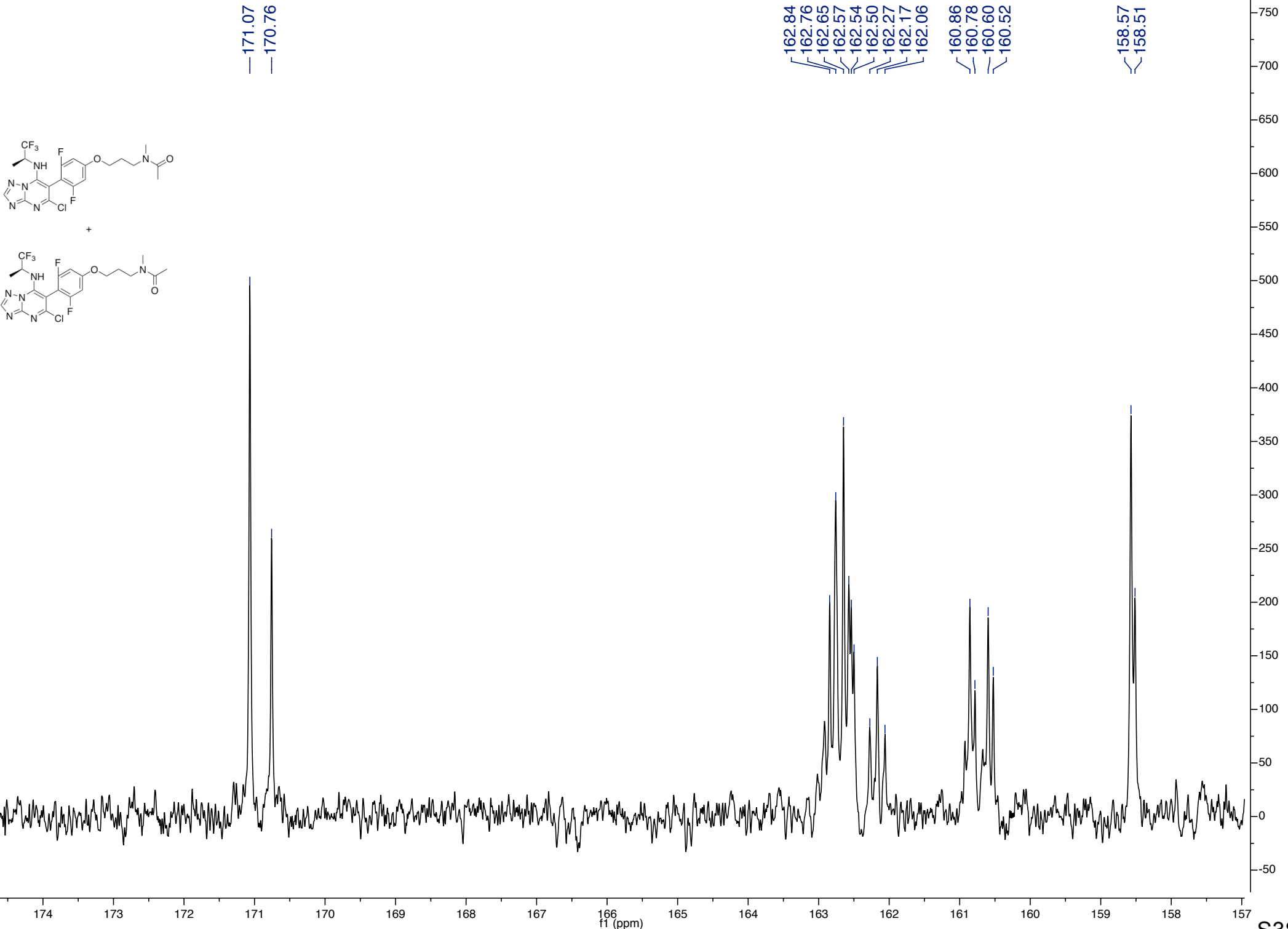
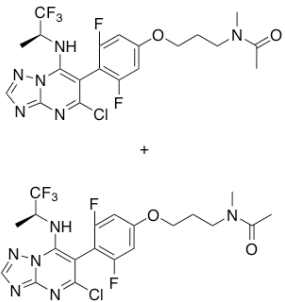
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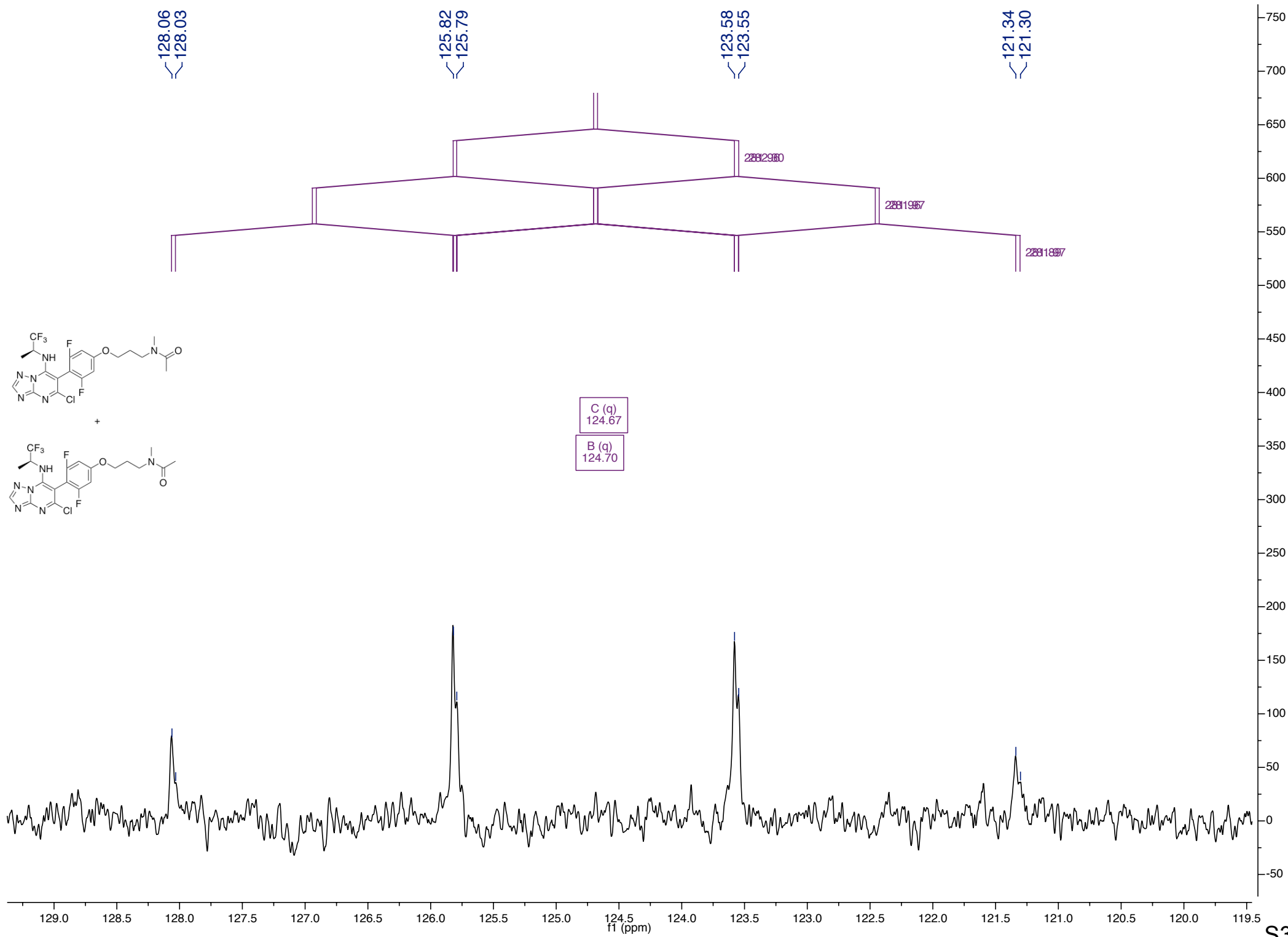


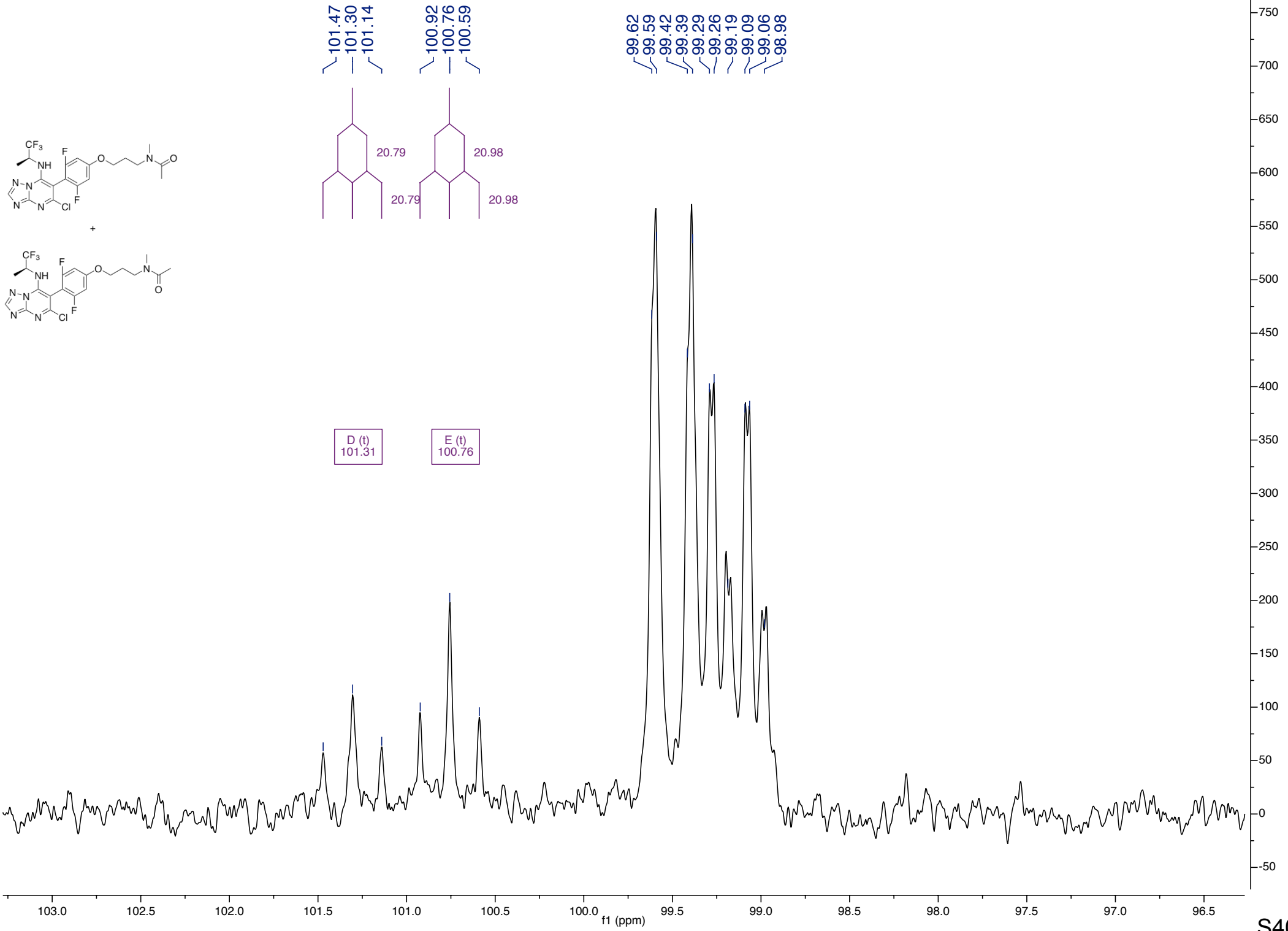
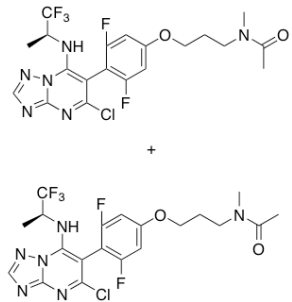




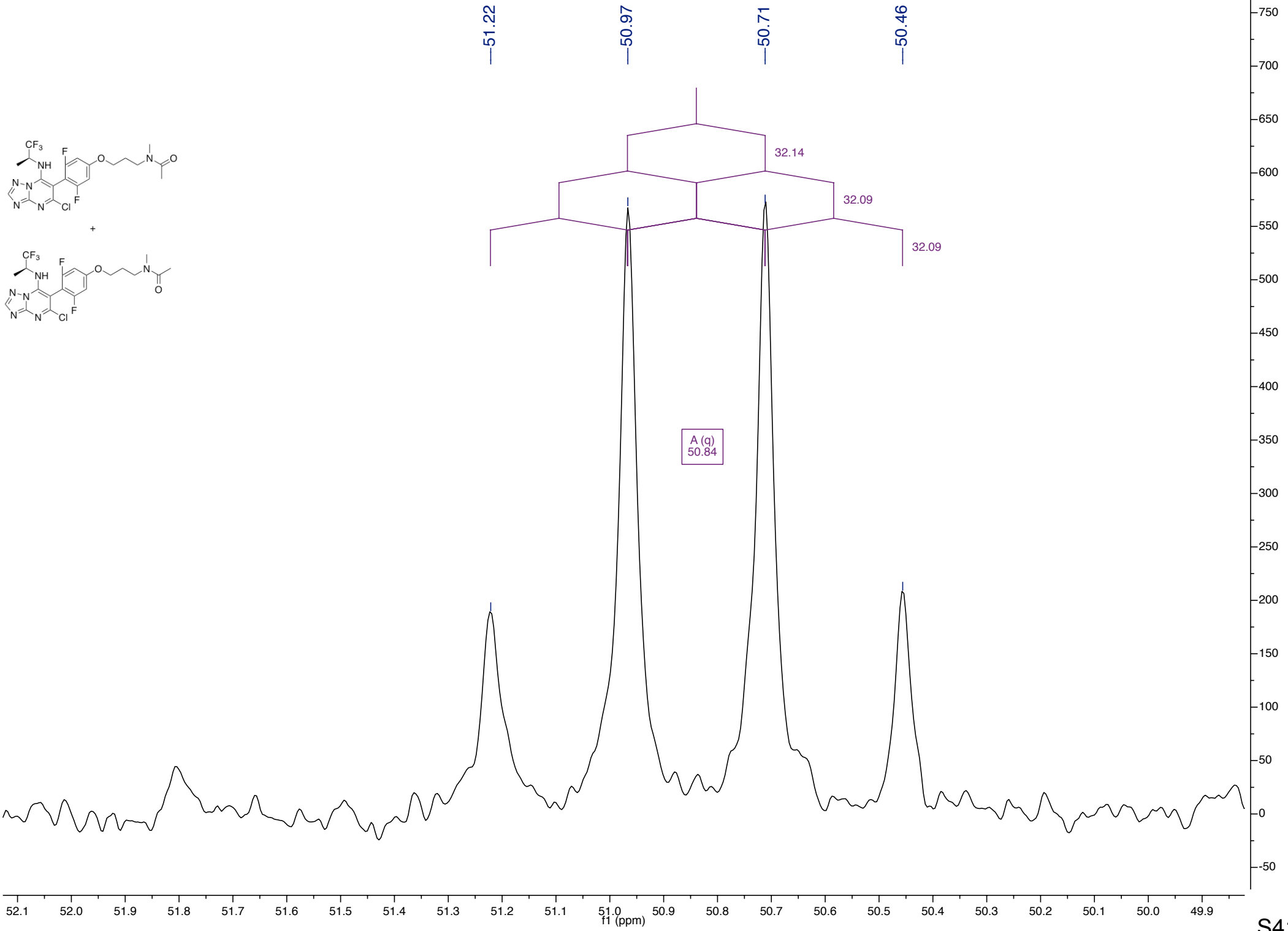
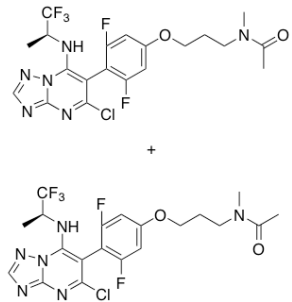




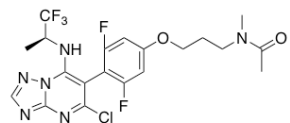




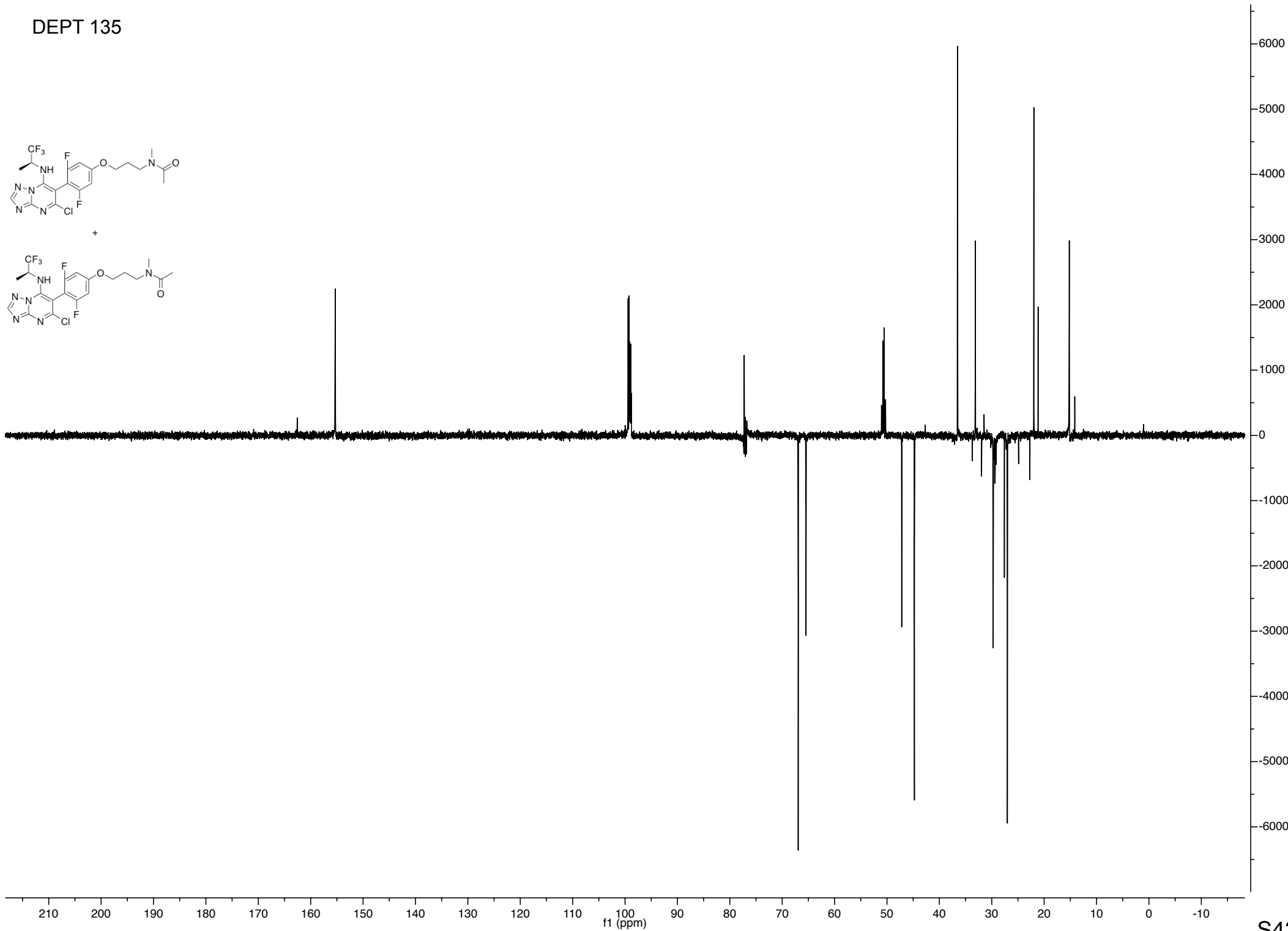
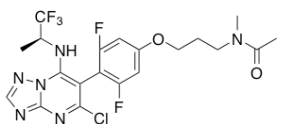




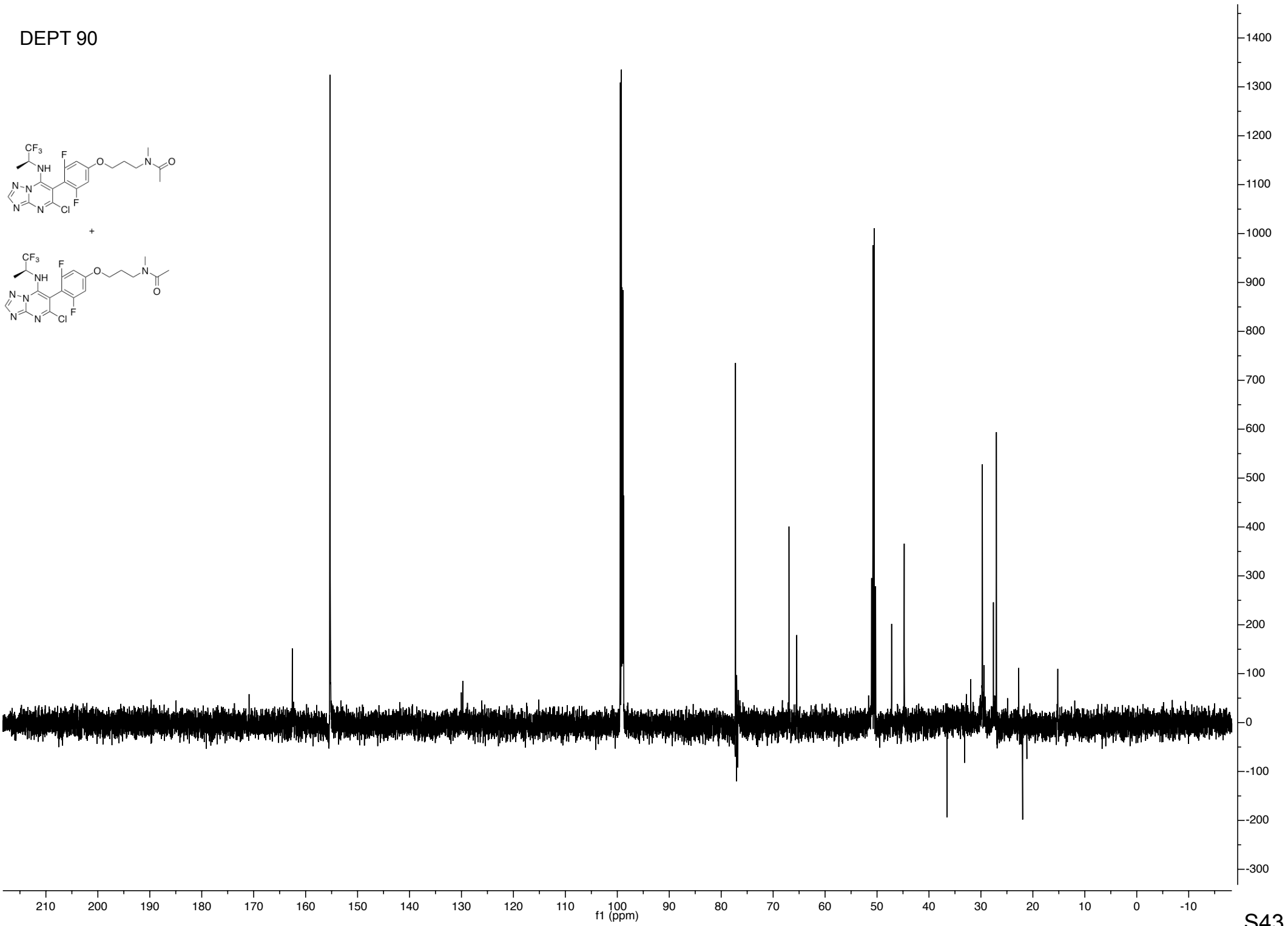
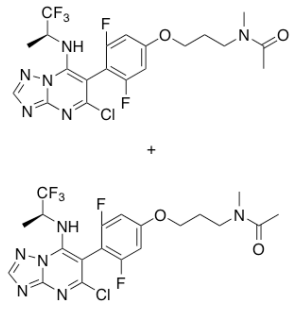
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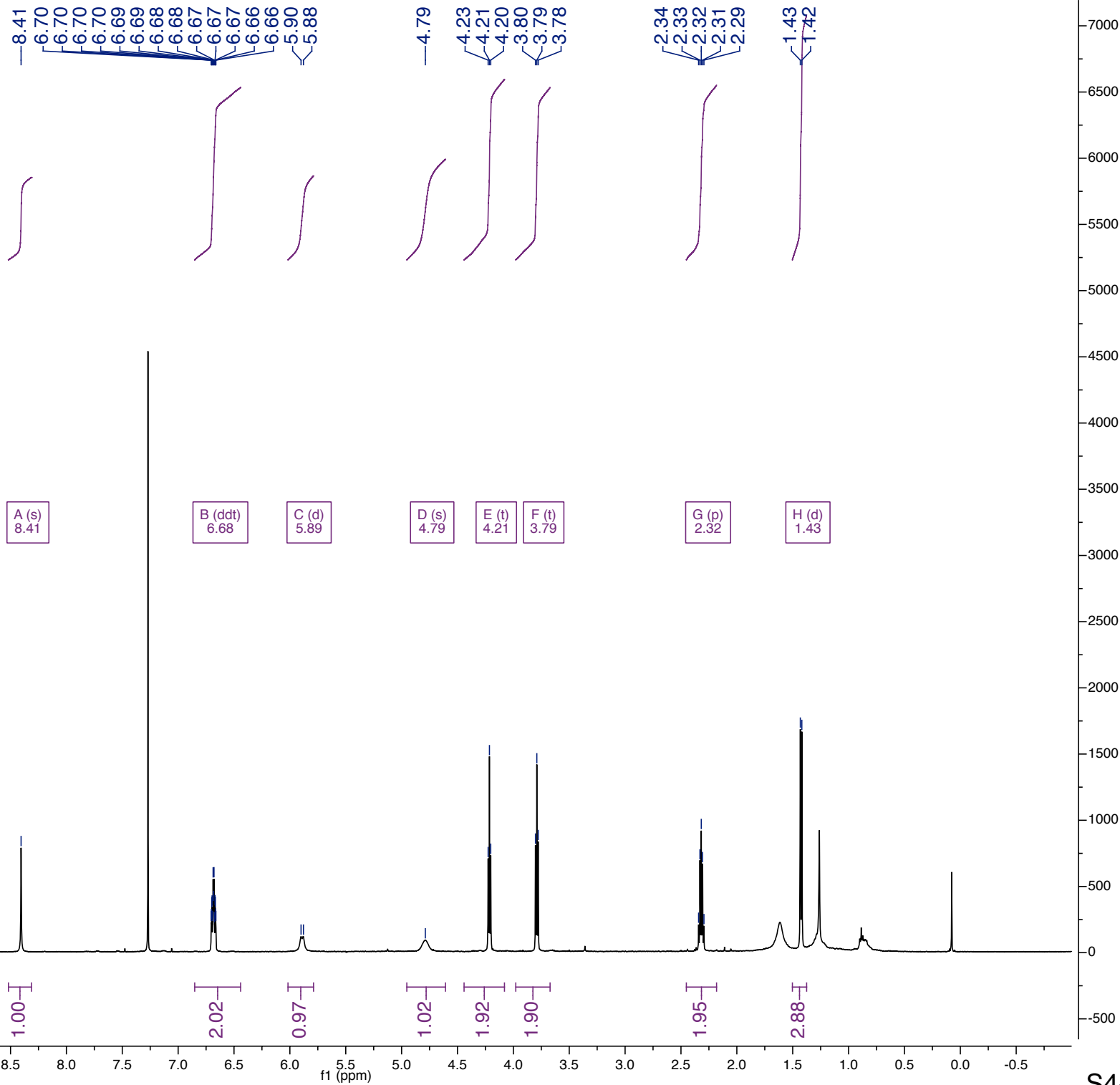
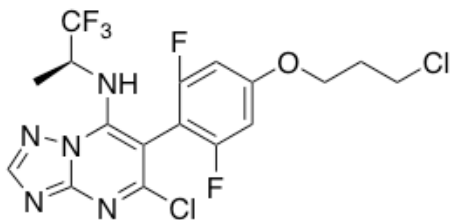


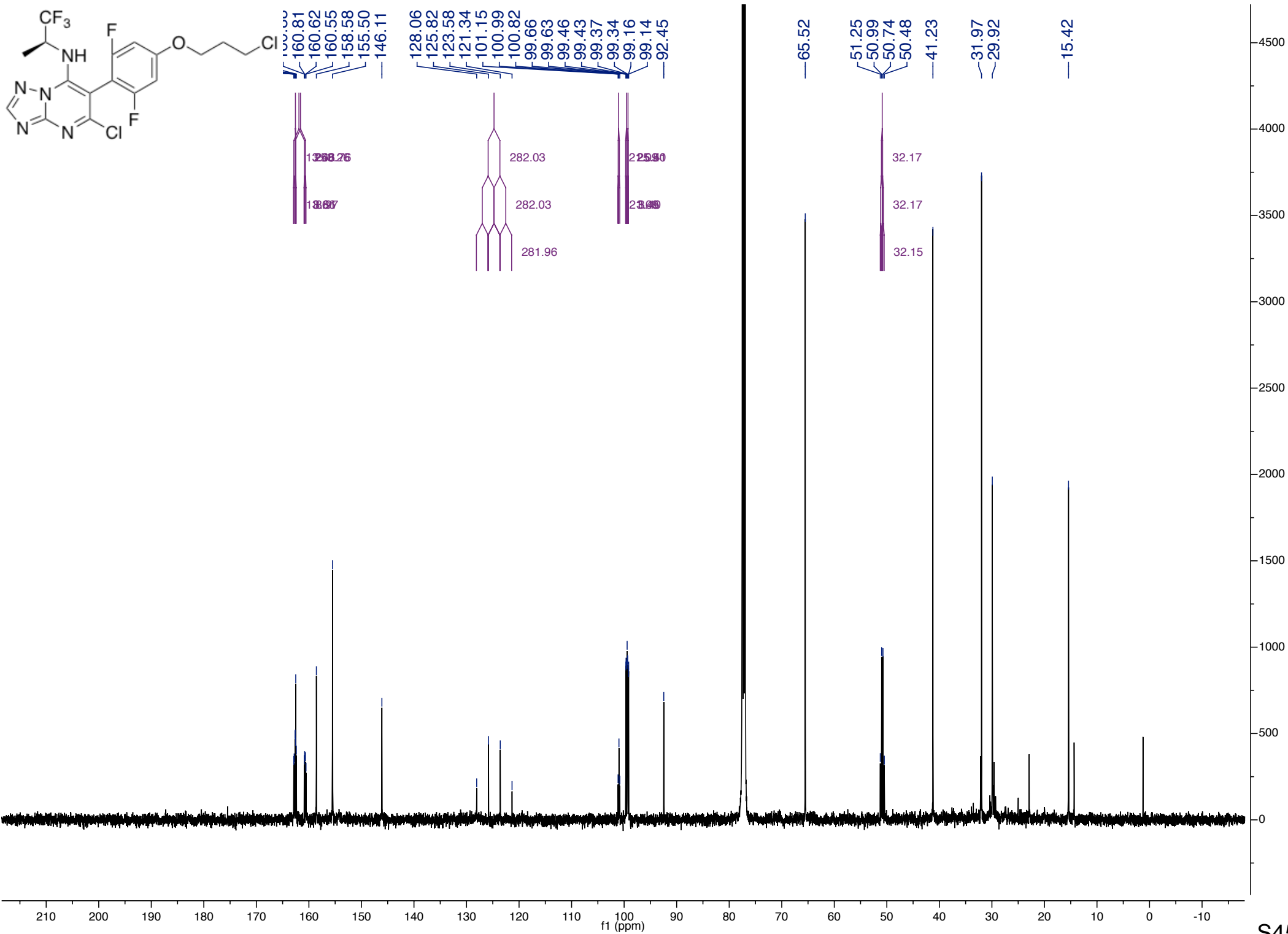
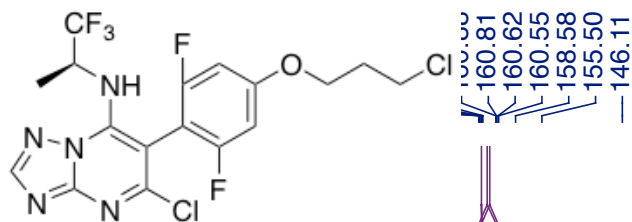
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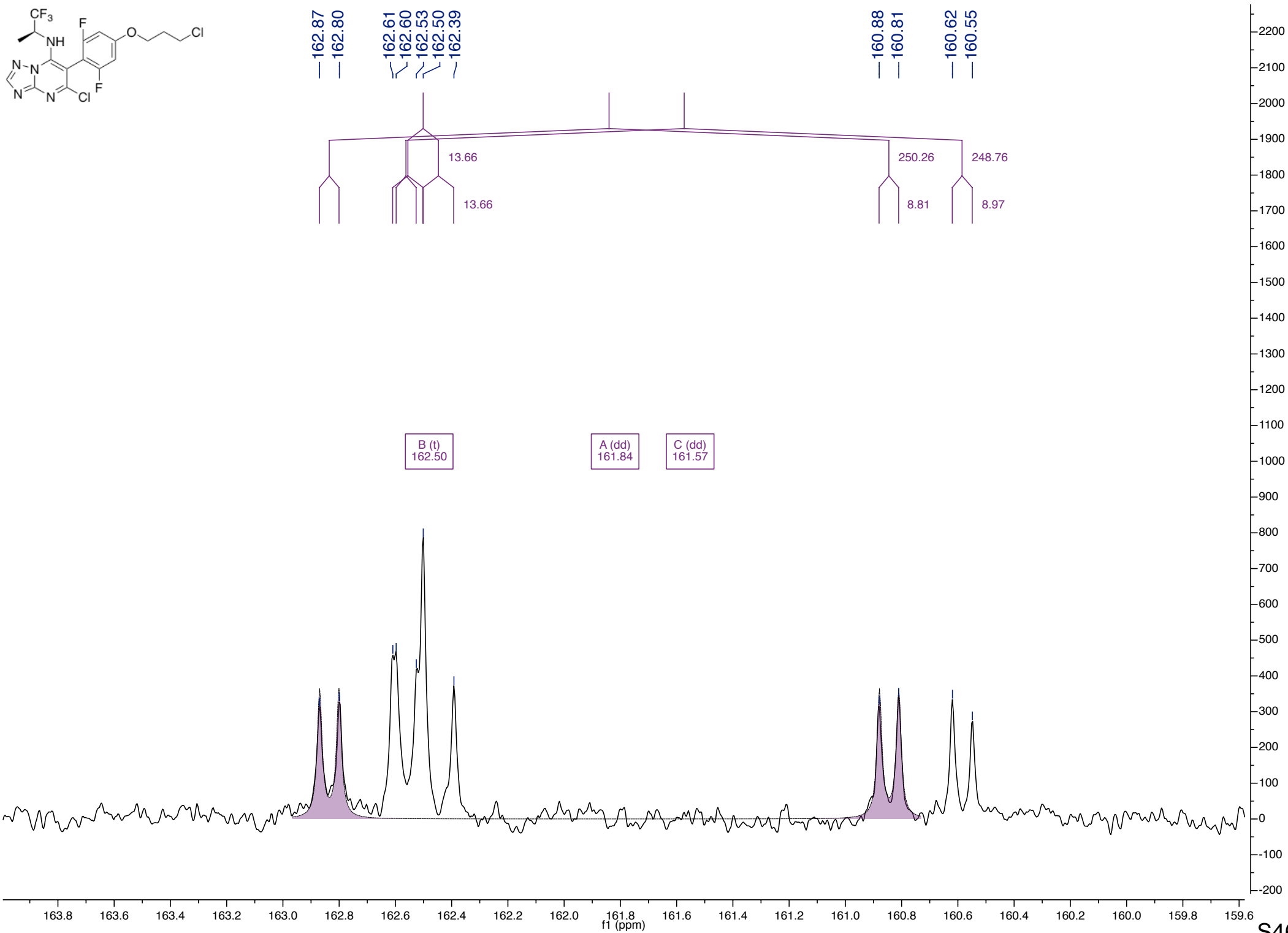
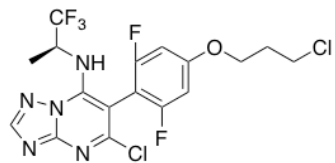


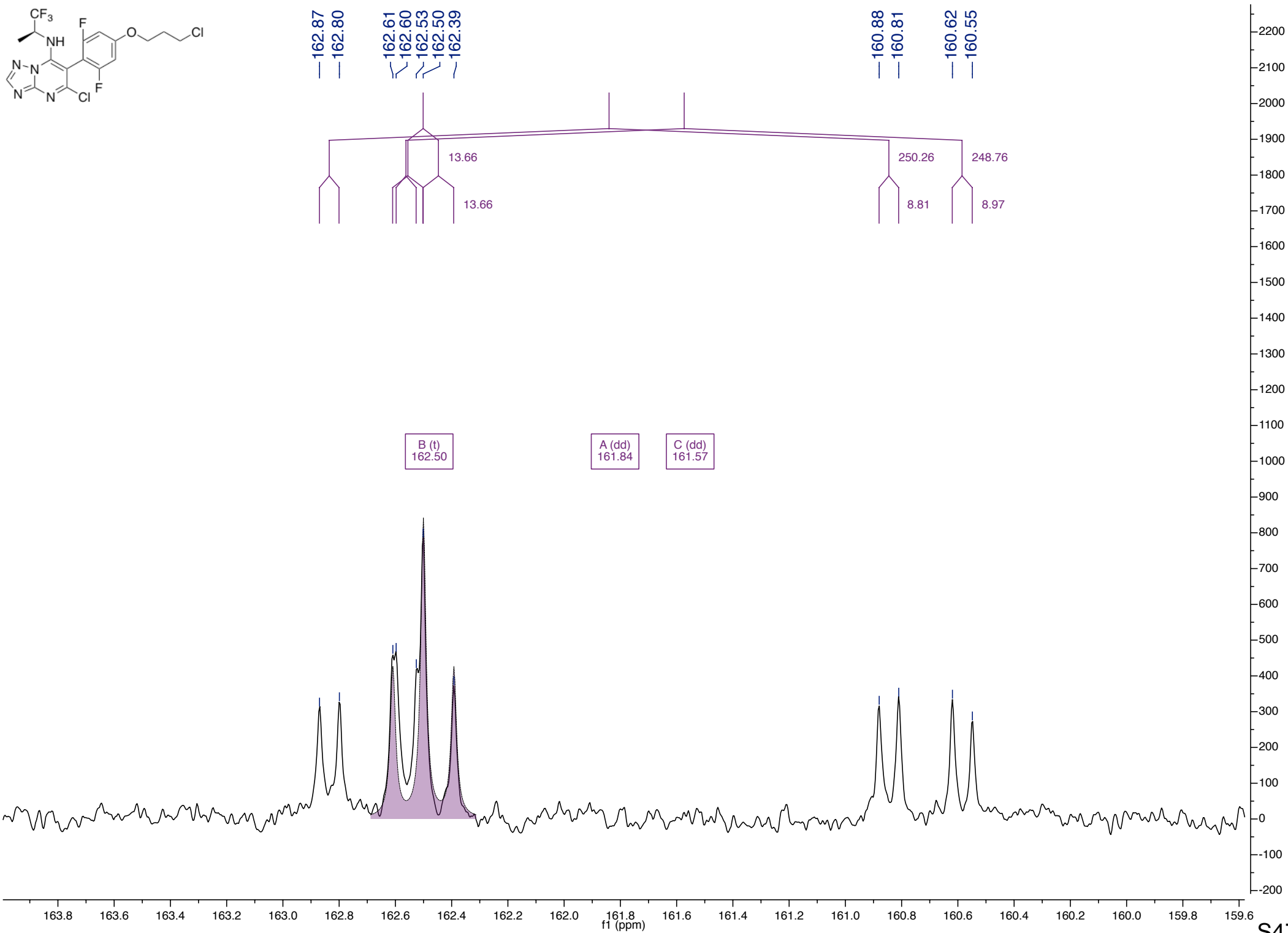
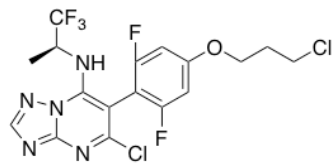
DEPT 90

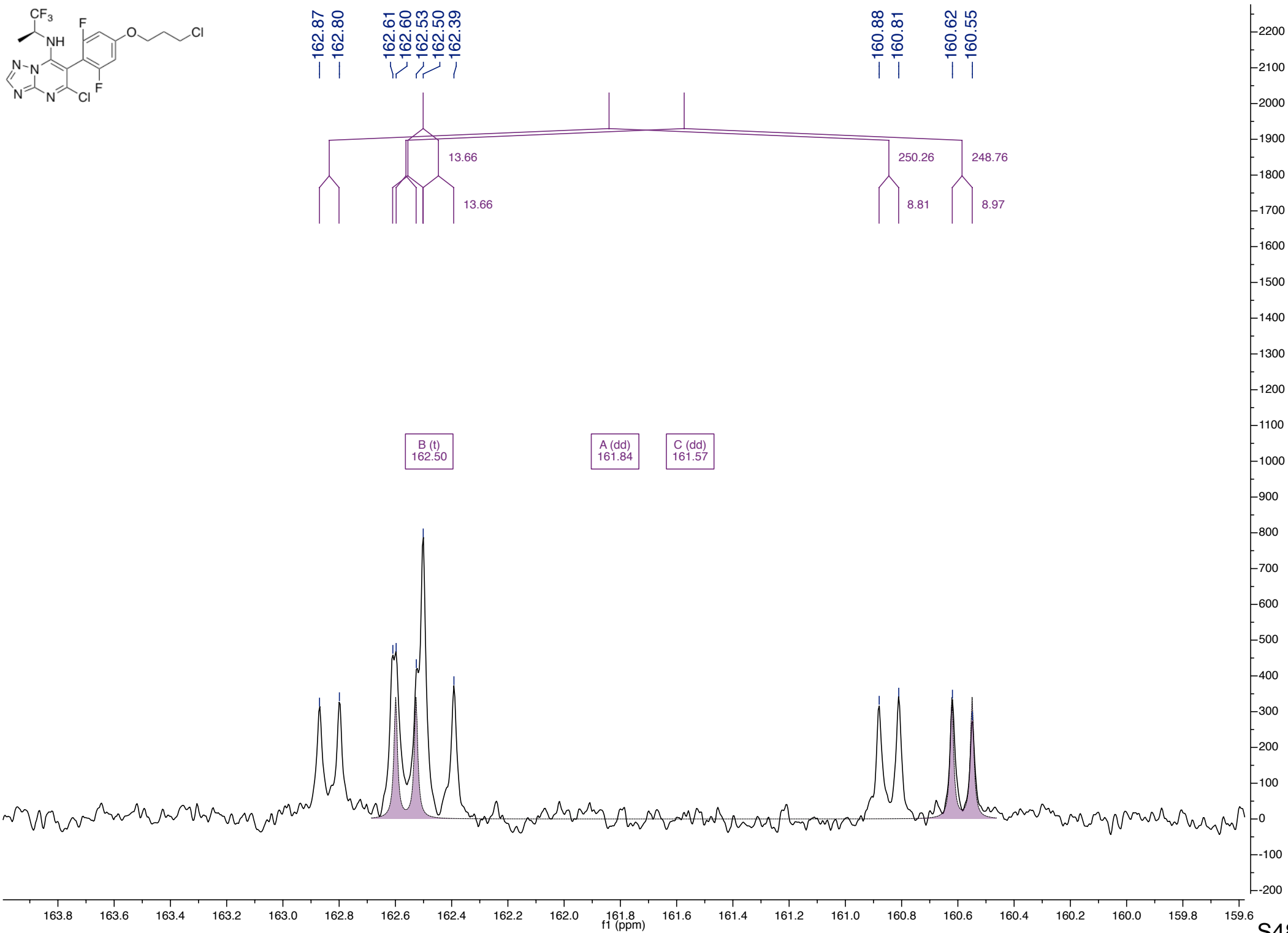
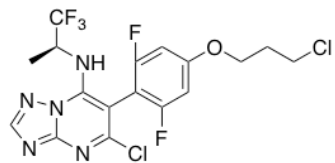




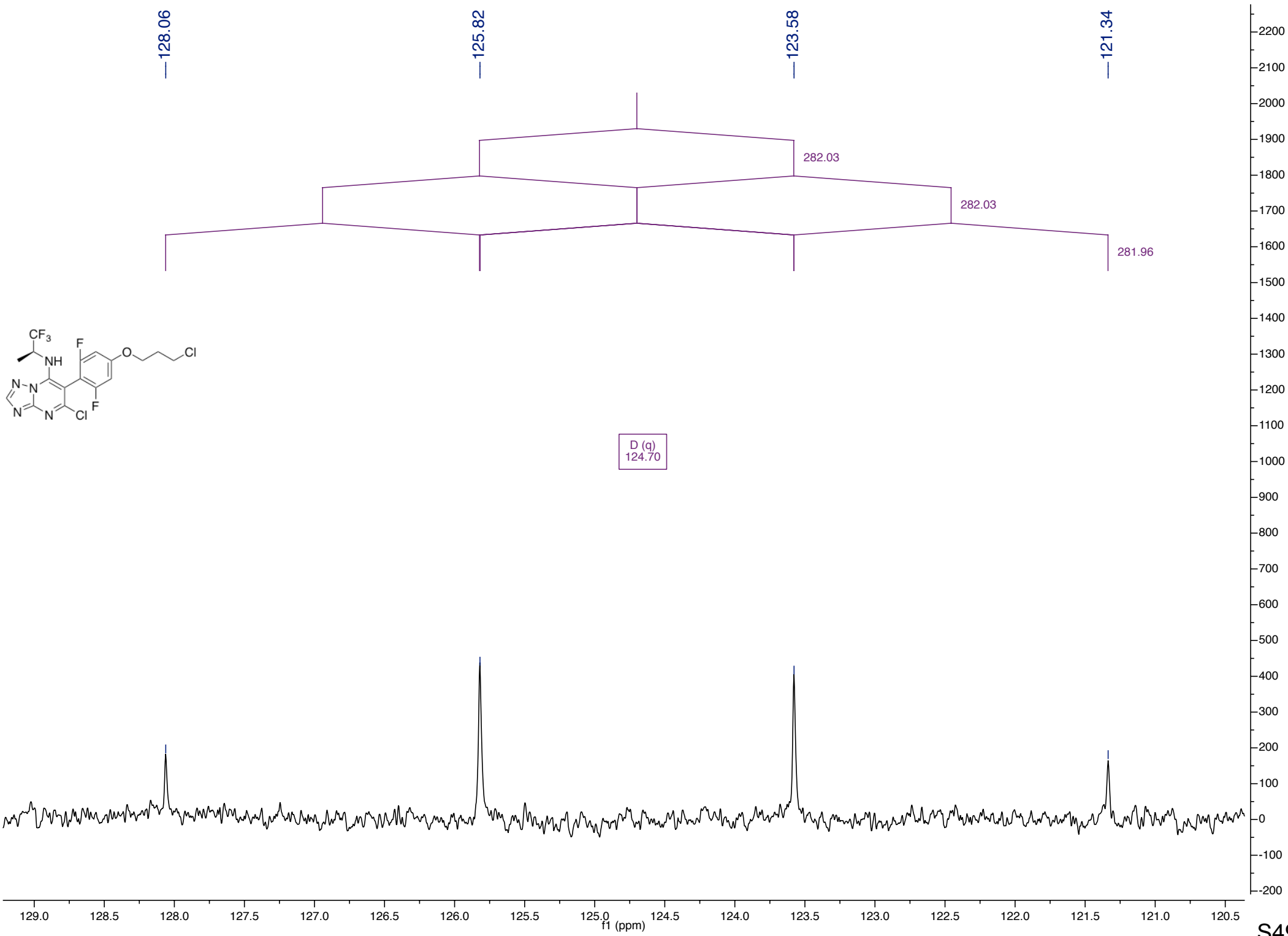


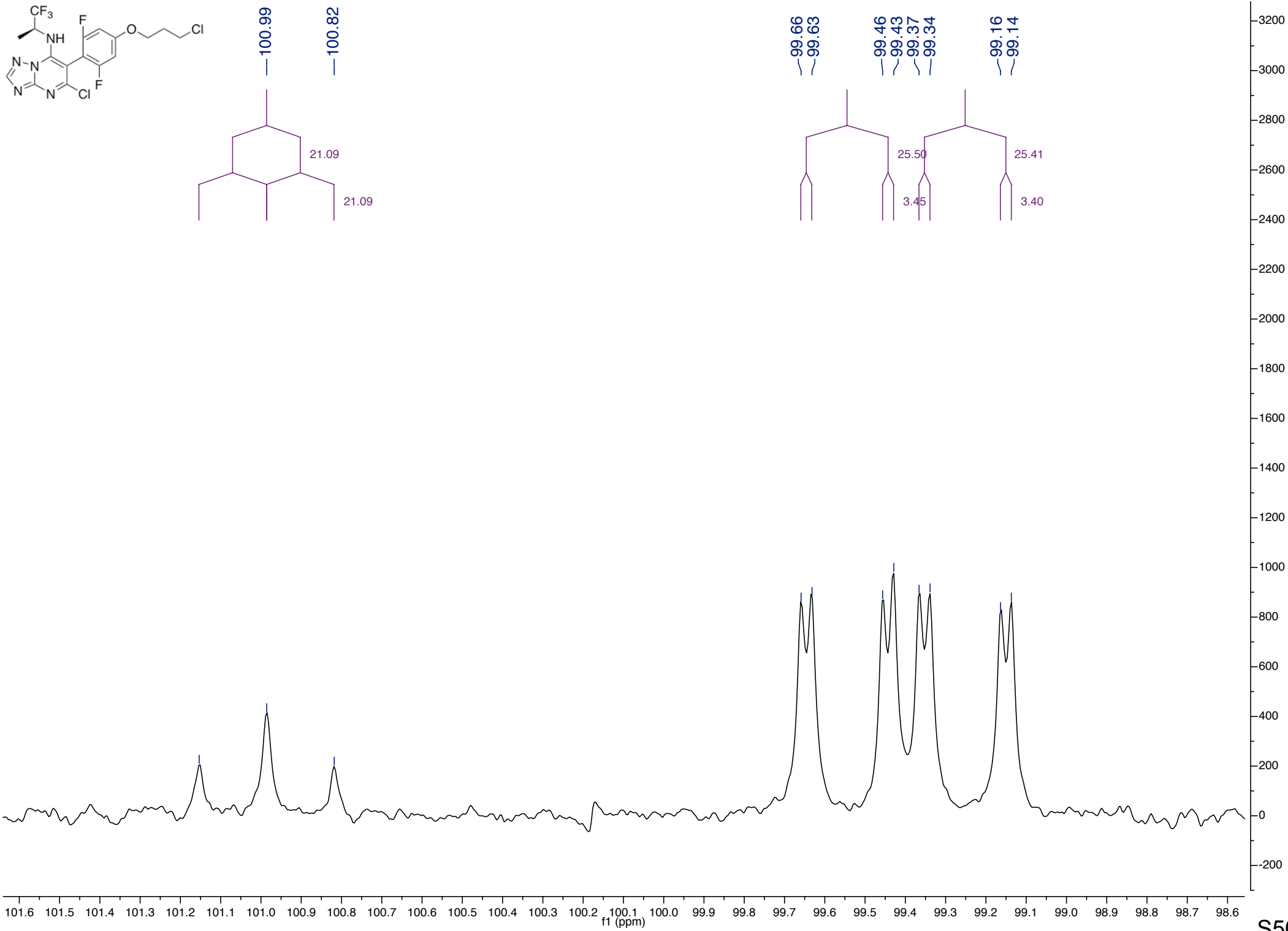
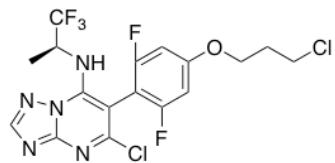


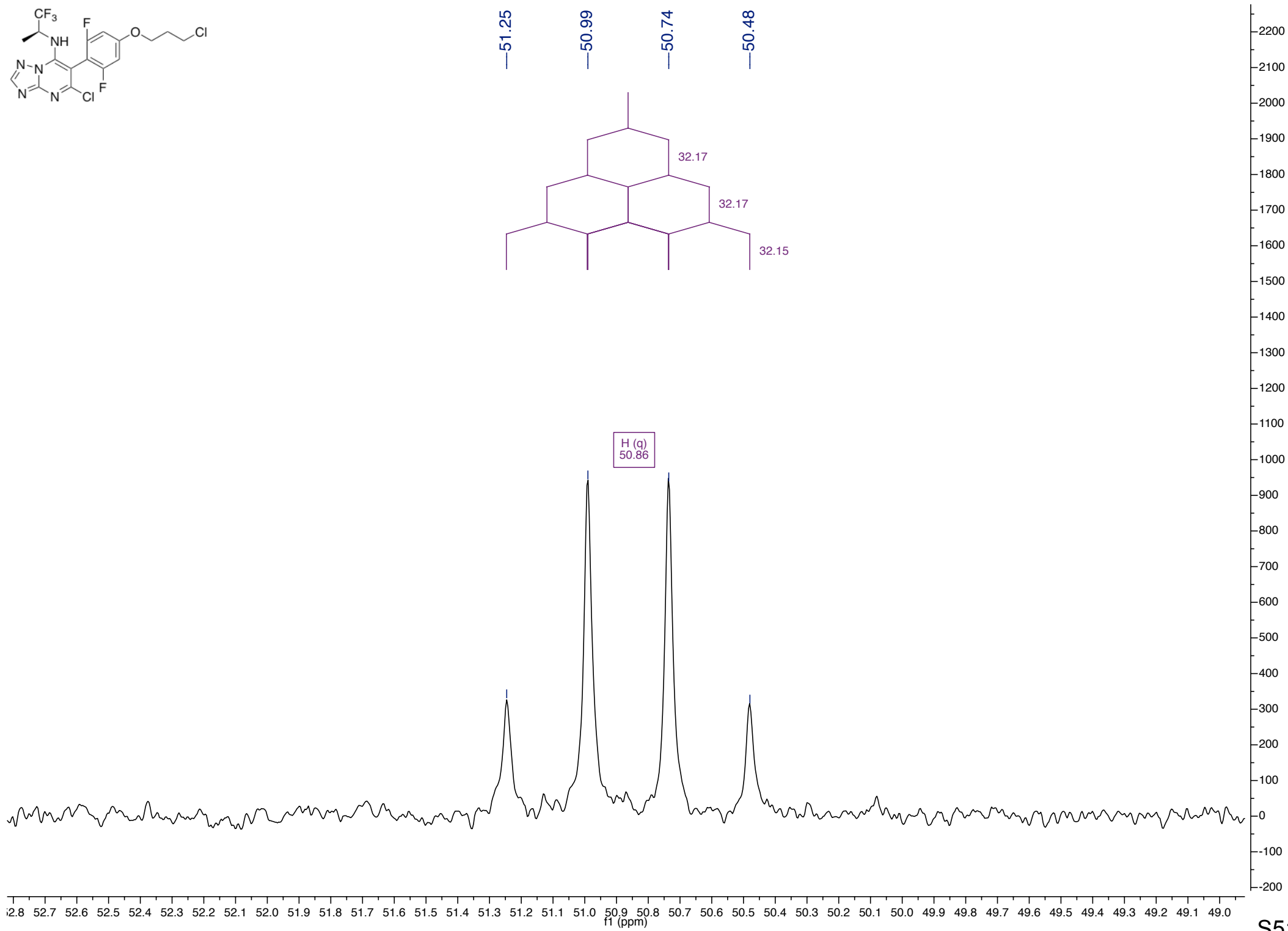
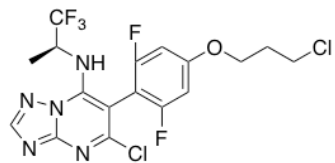






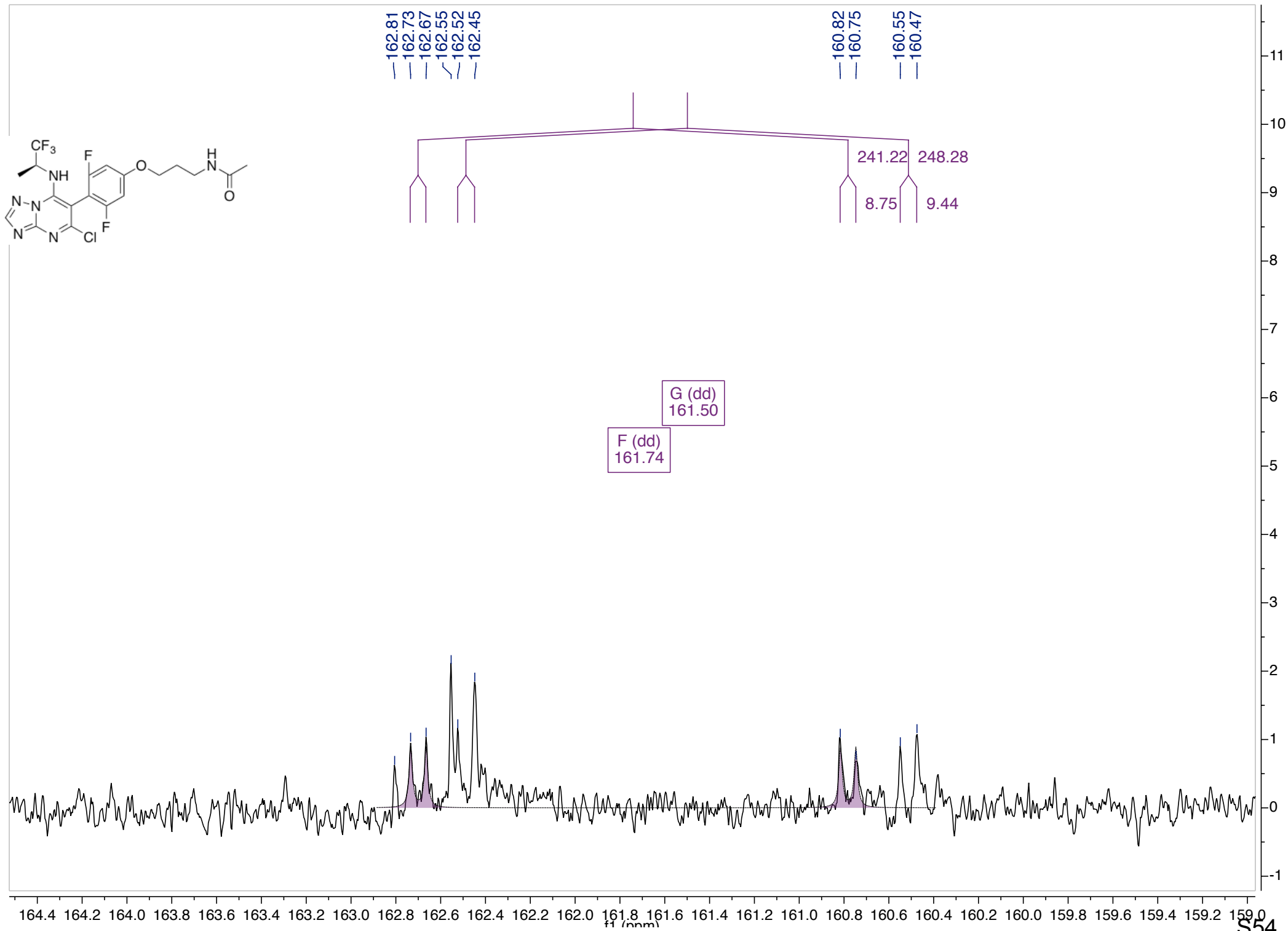


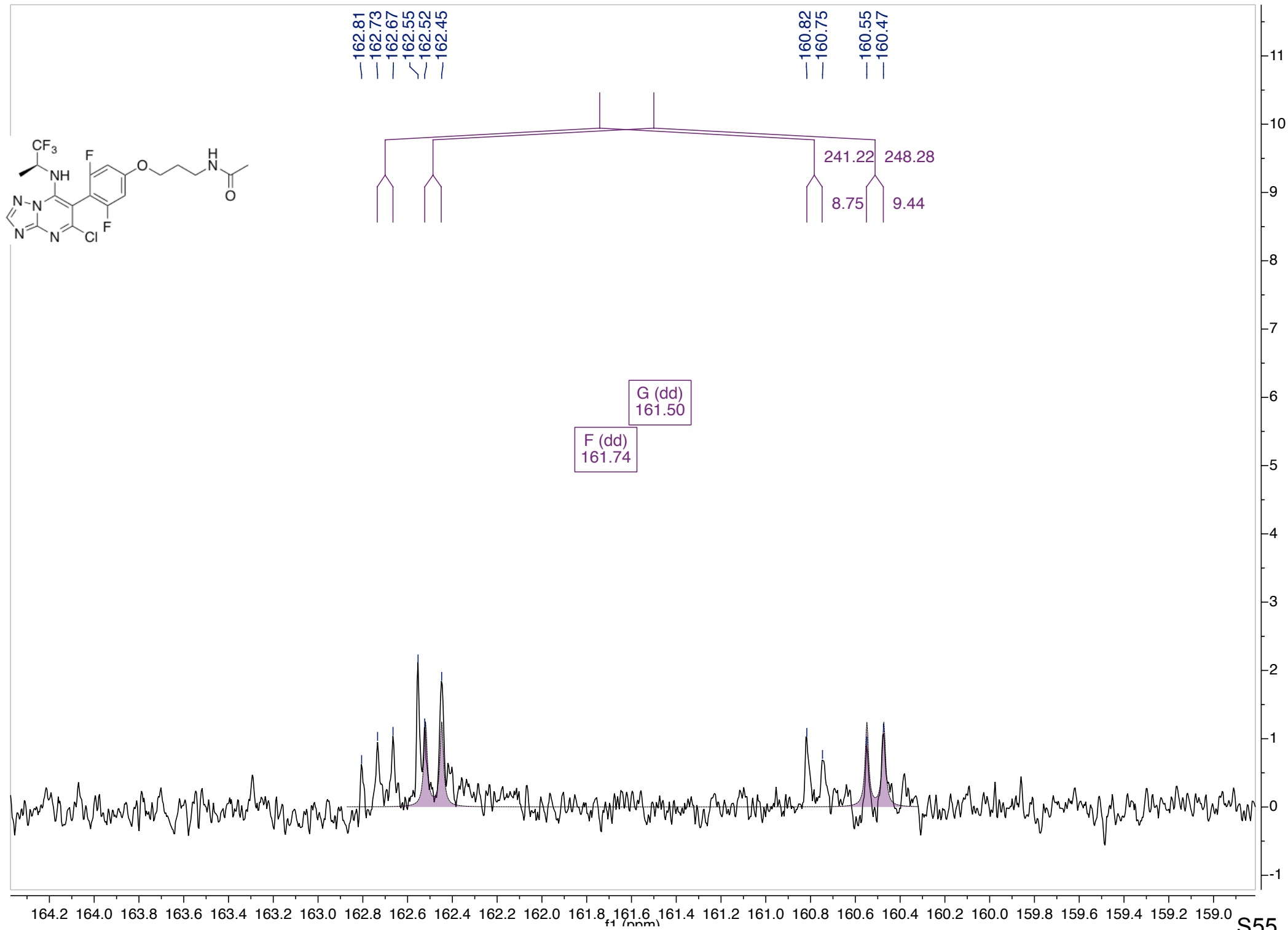


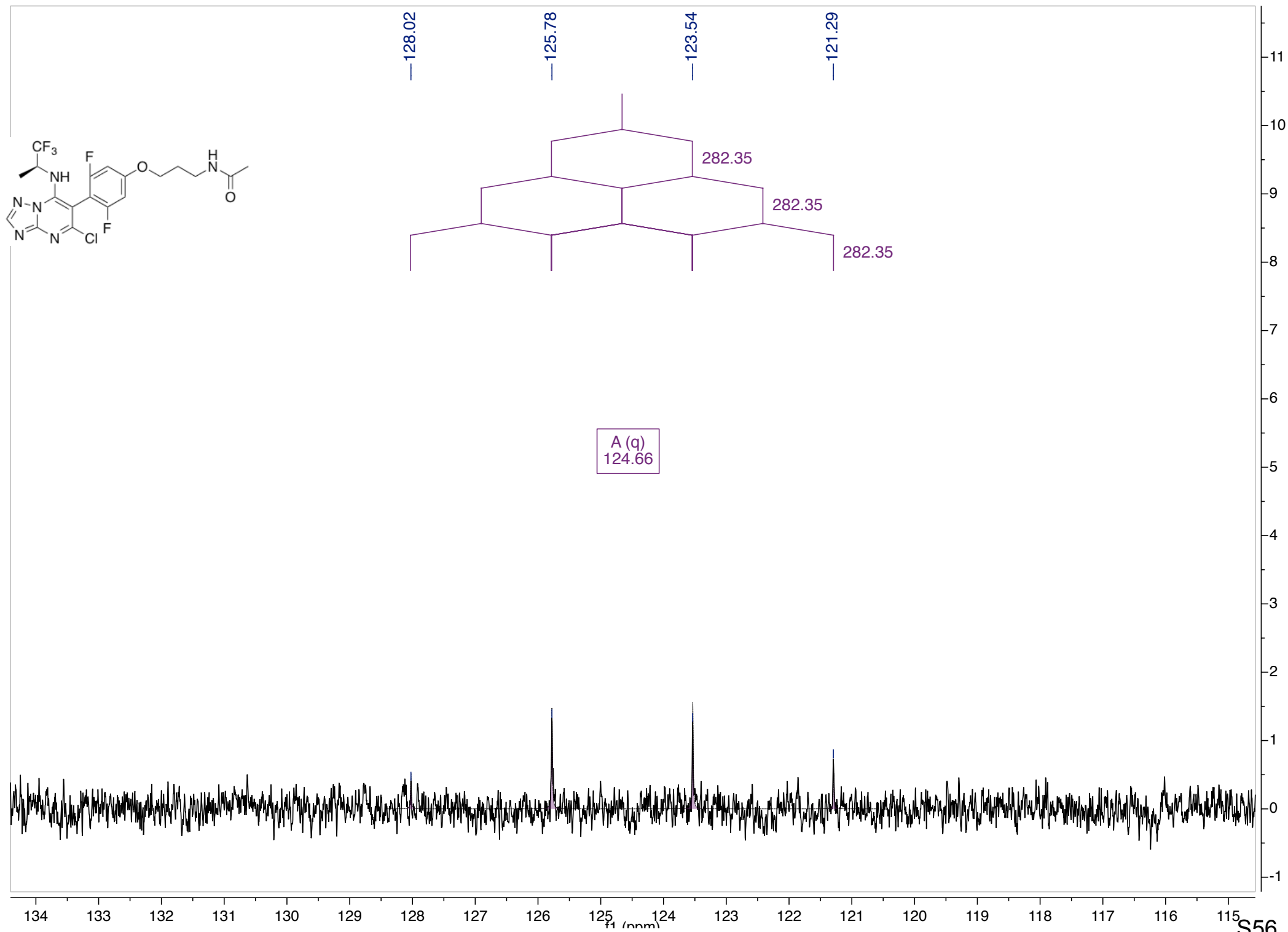




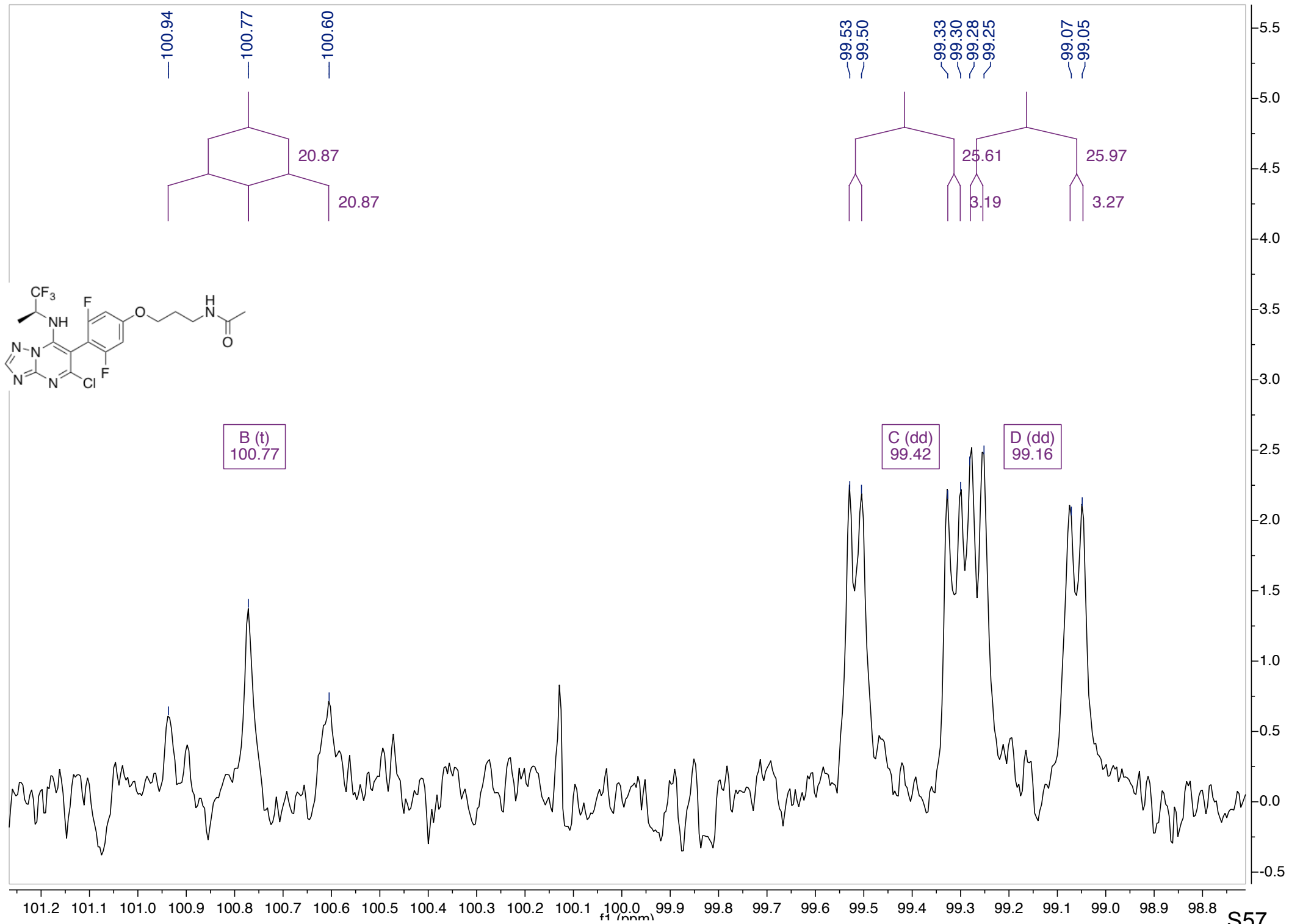


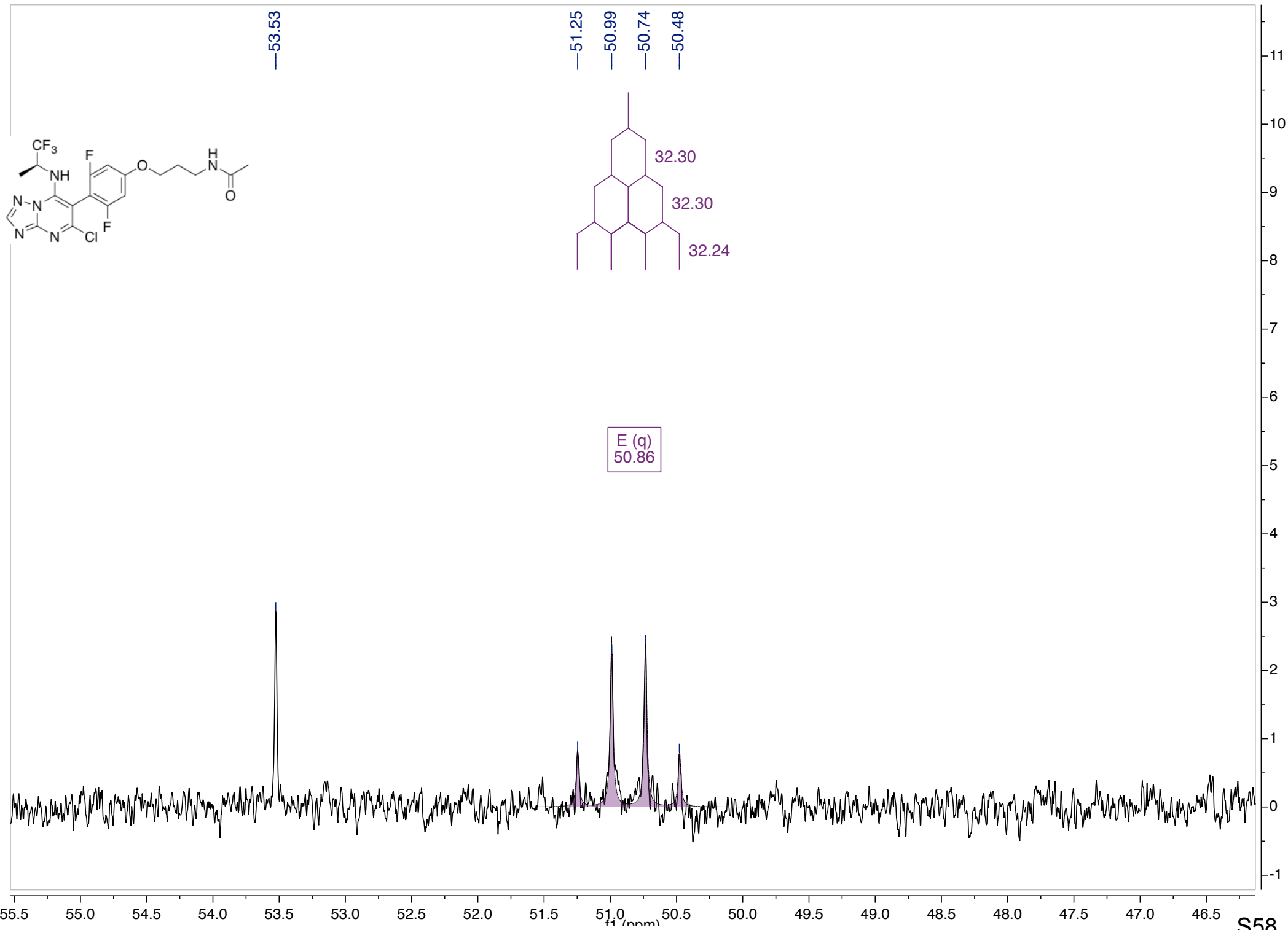


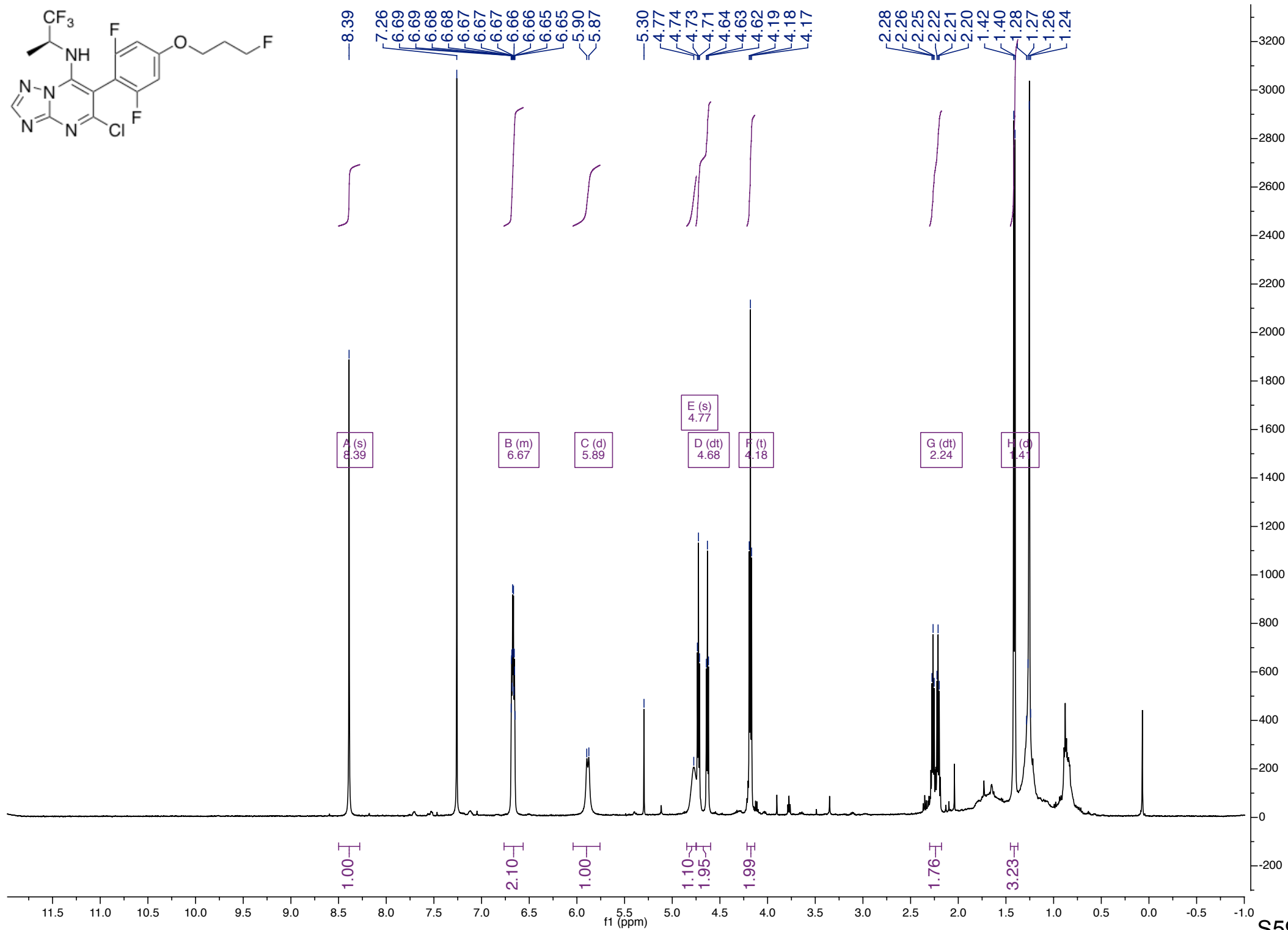
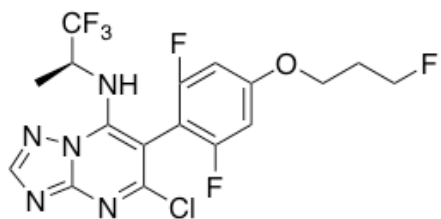


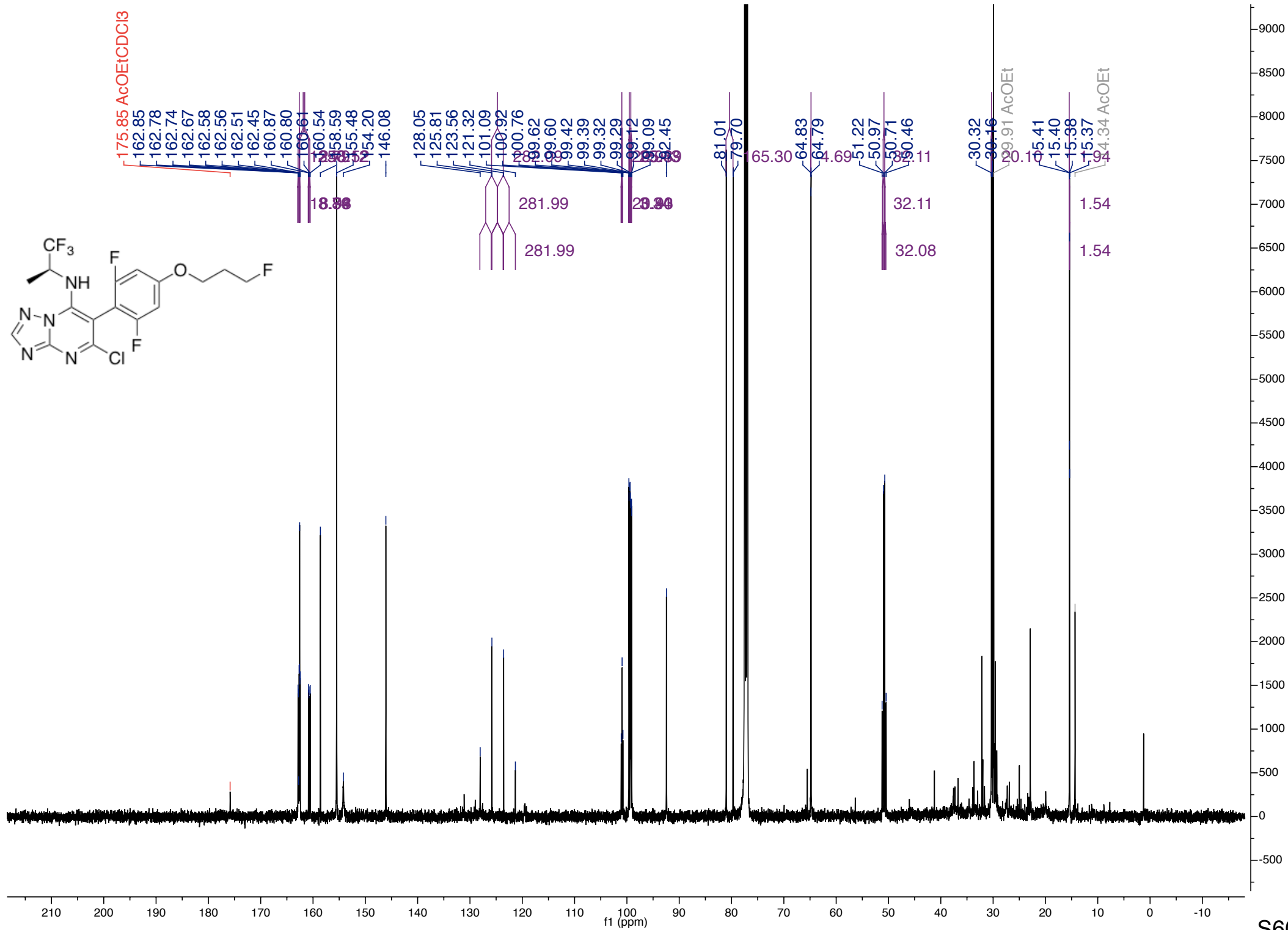
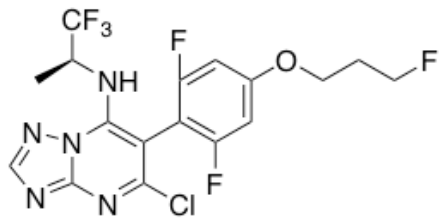


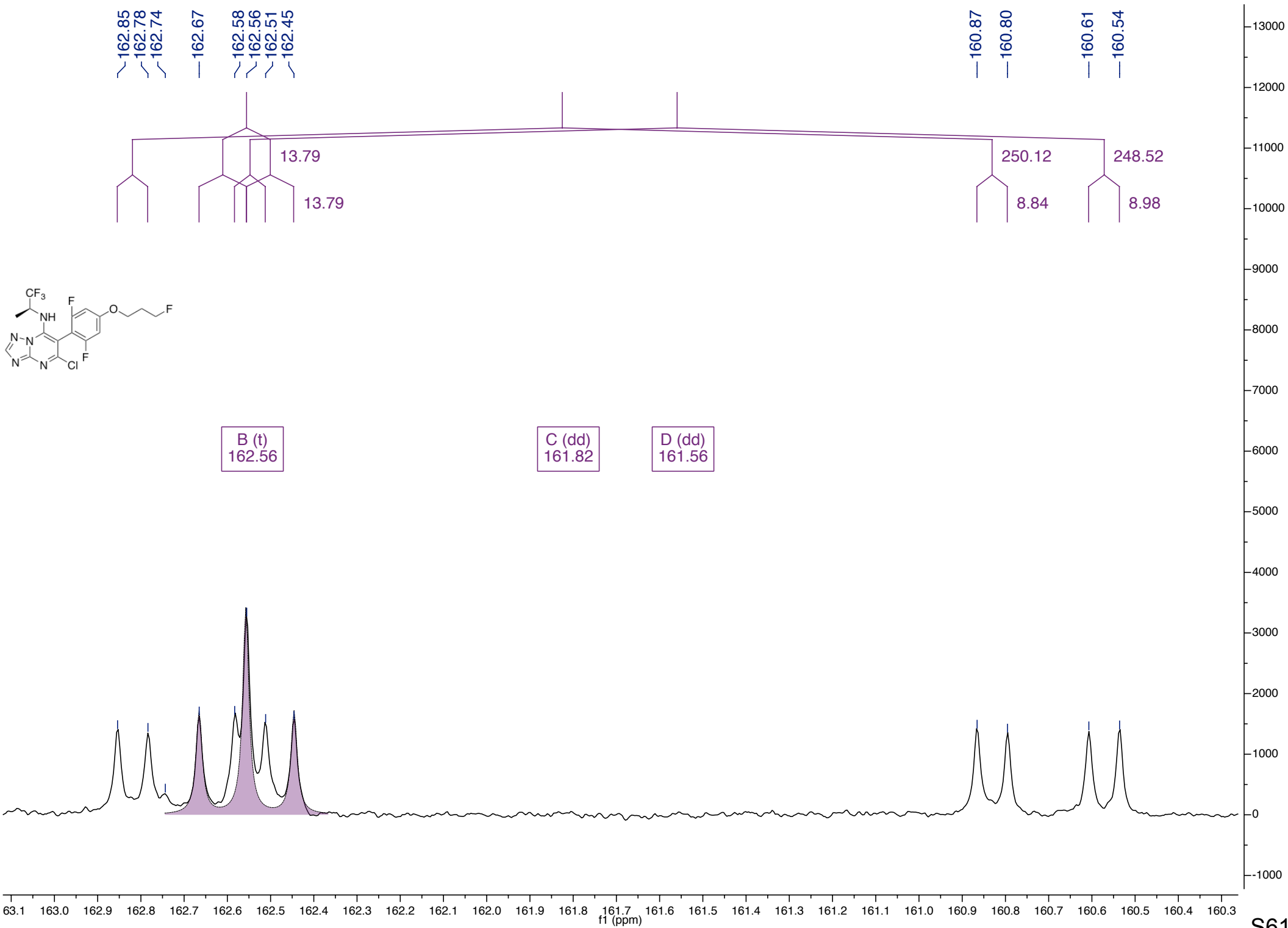


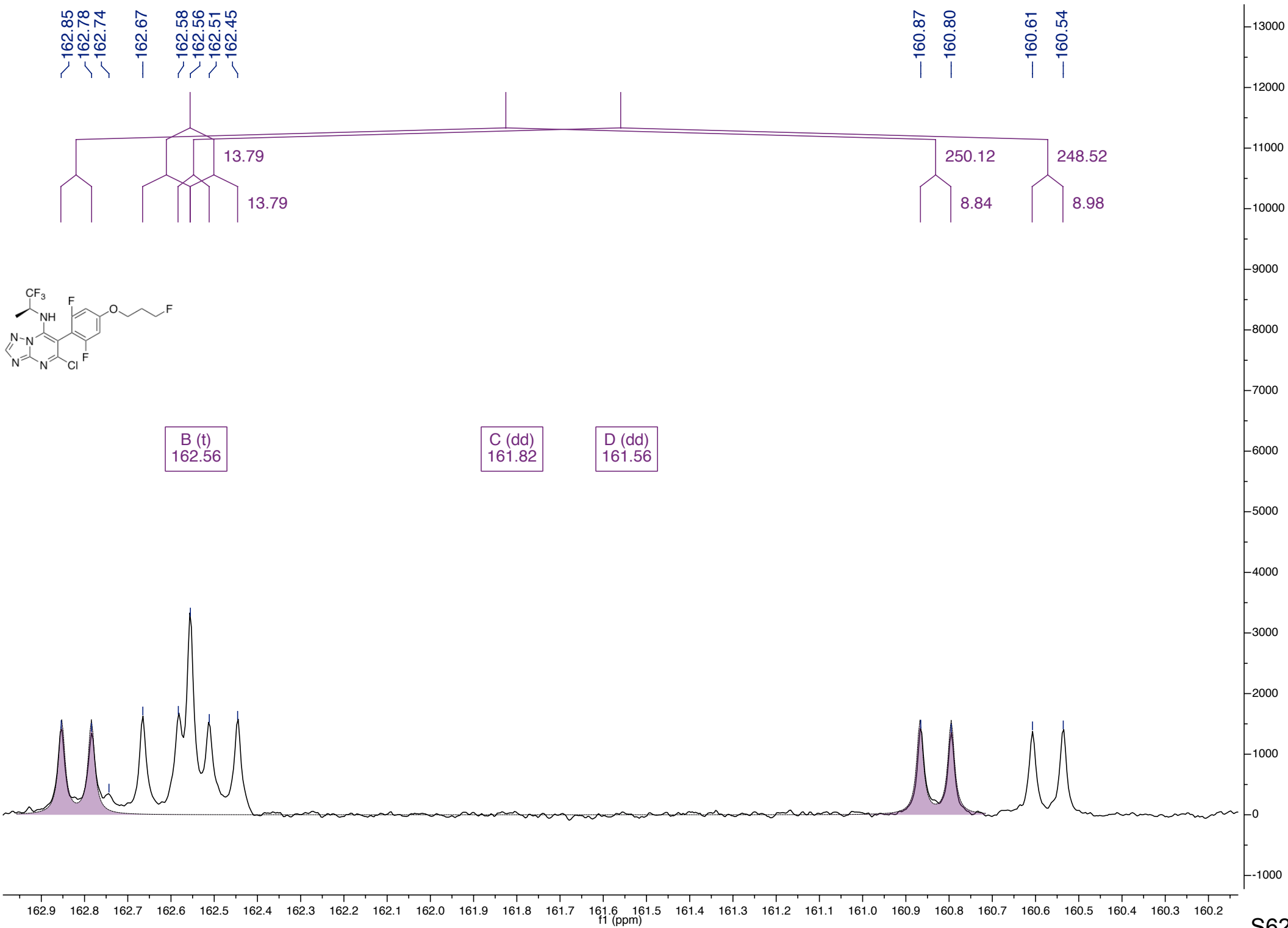


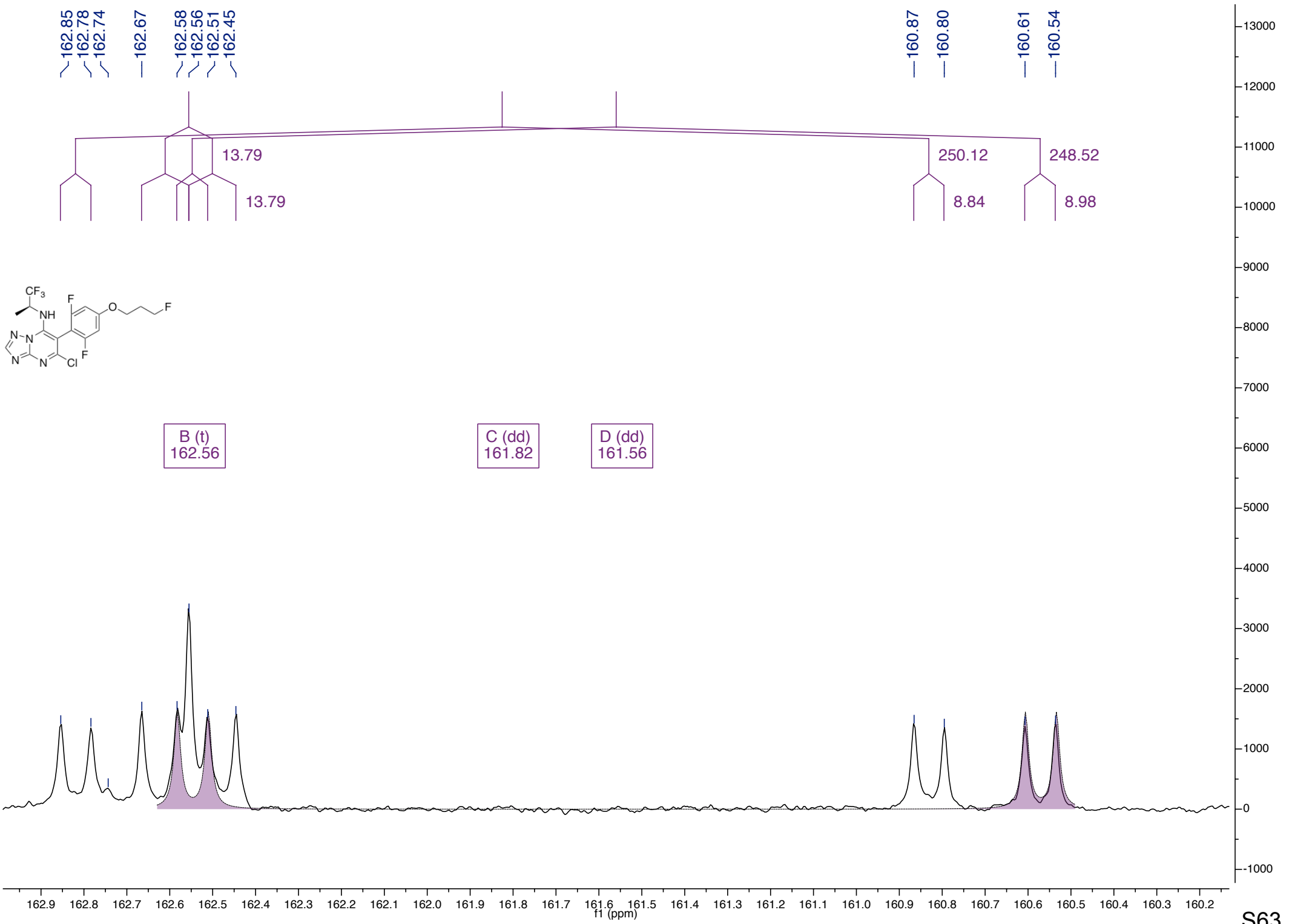


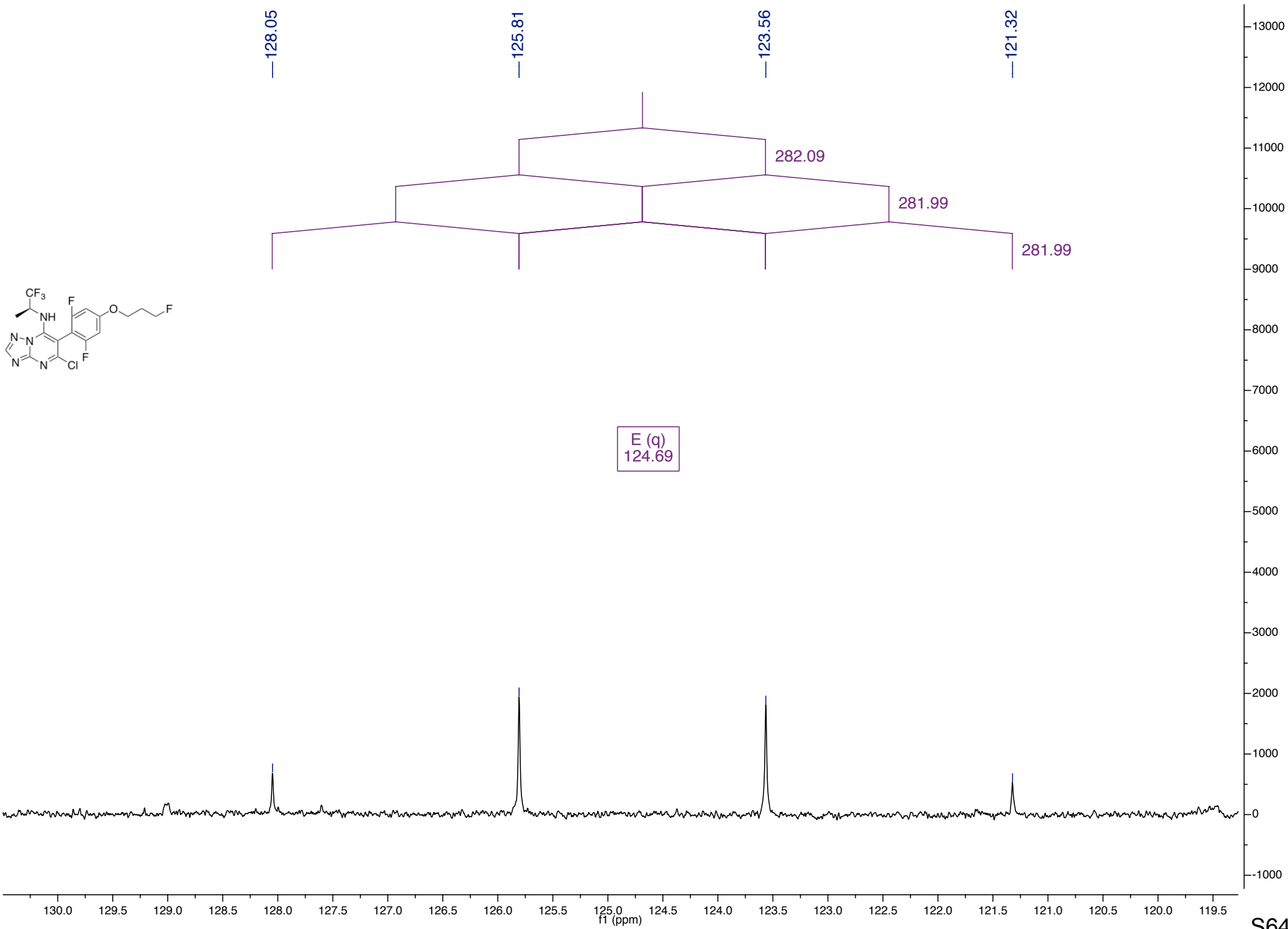
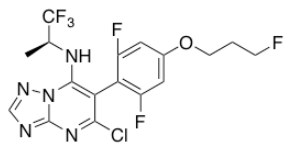




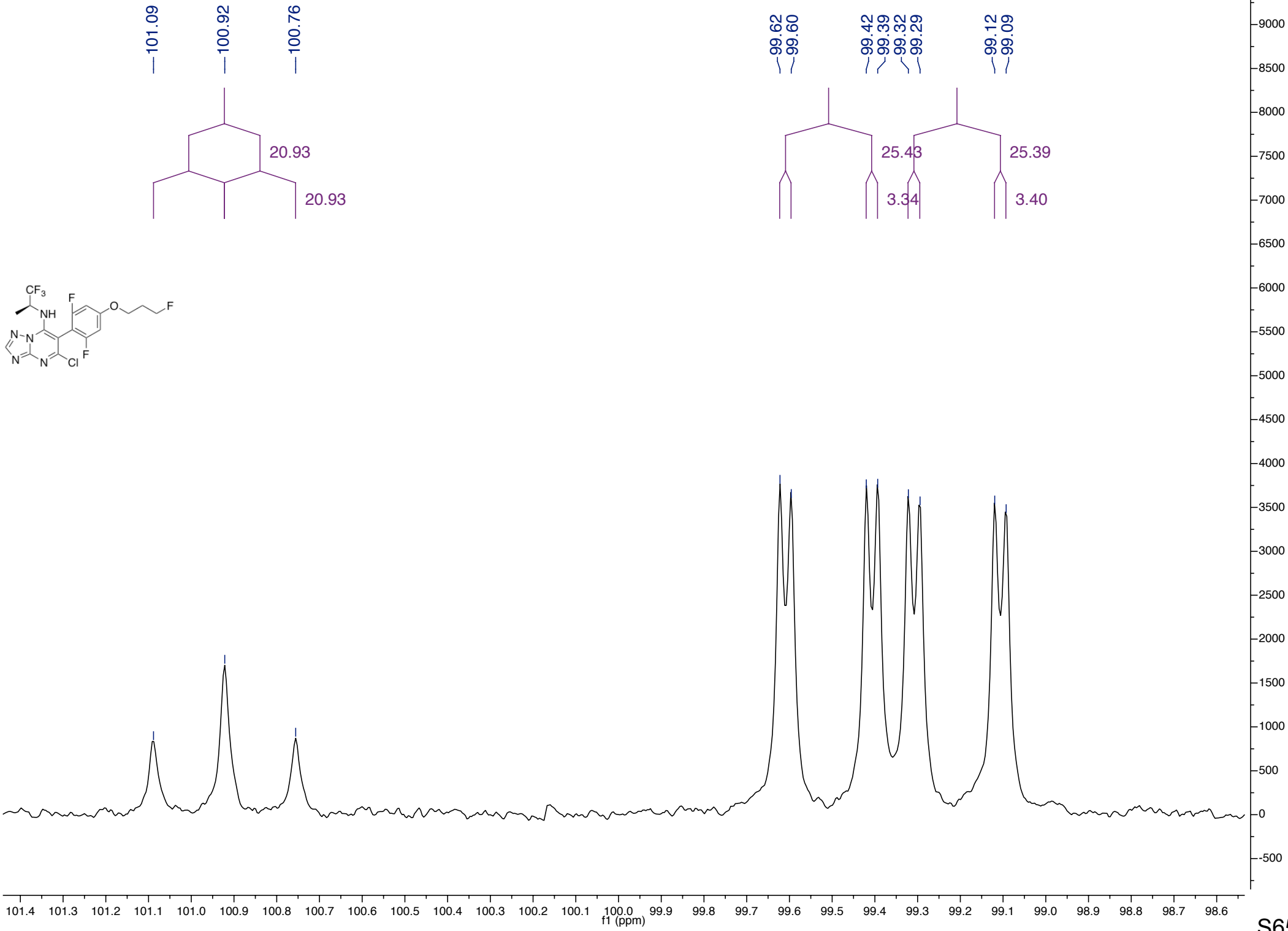


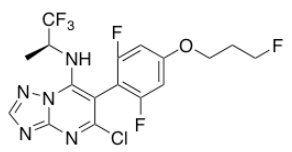
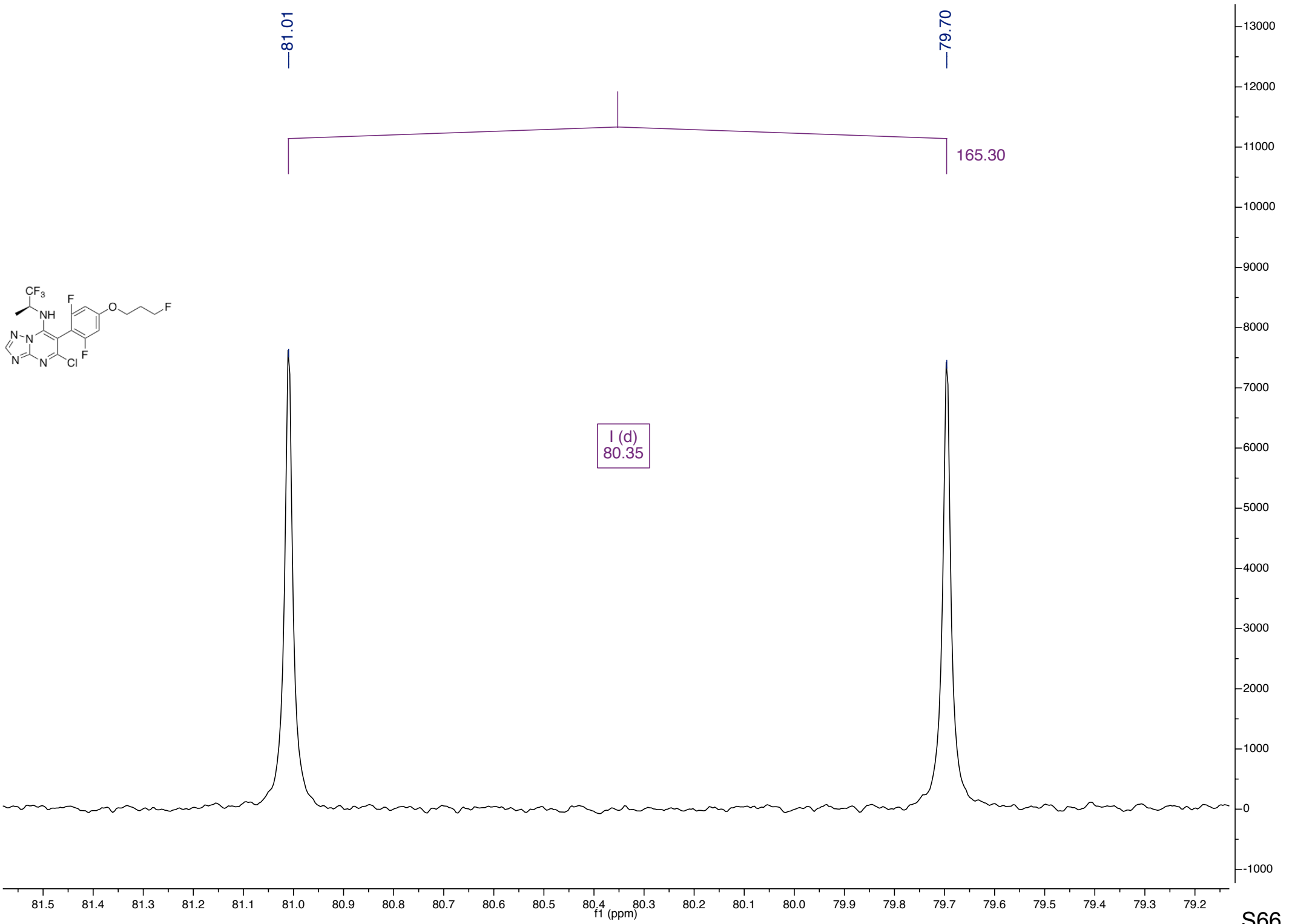


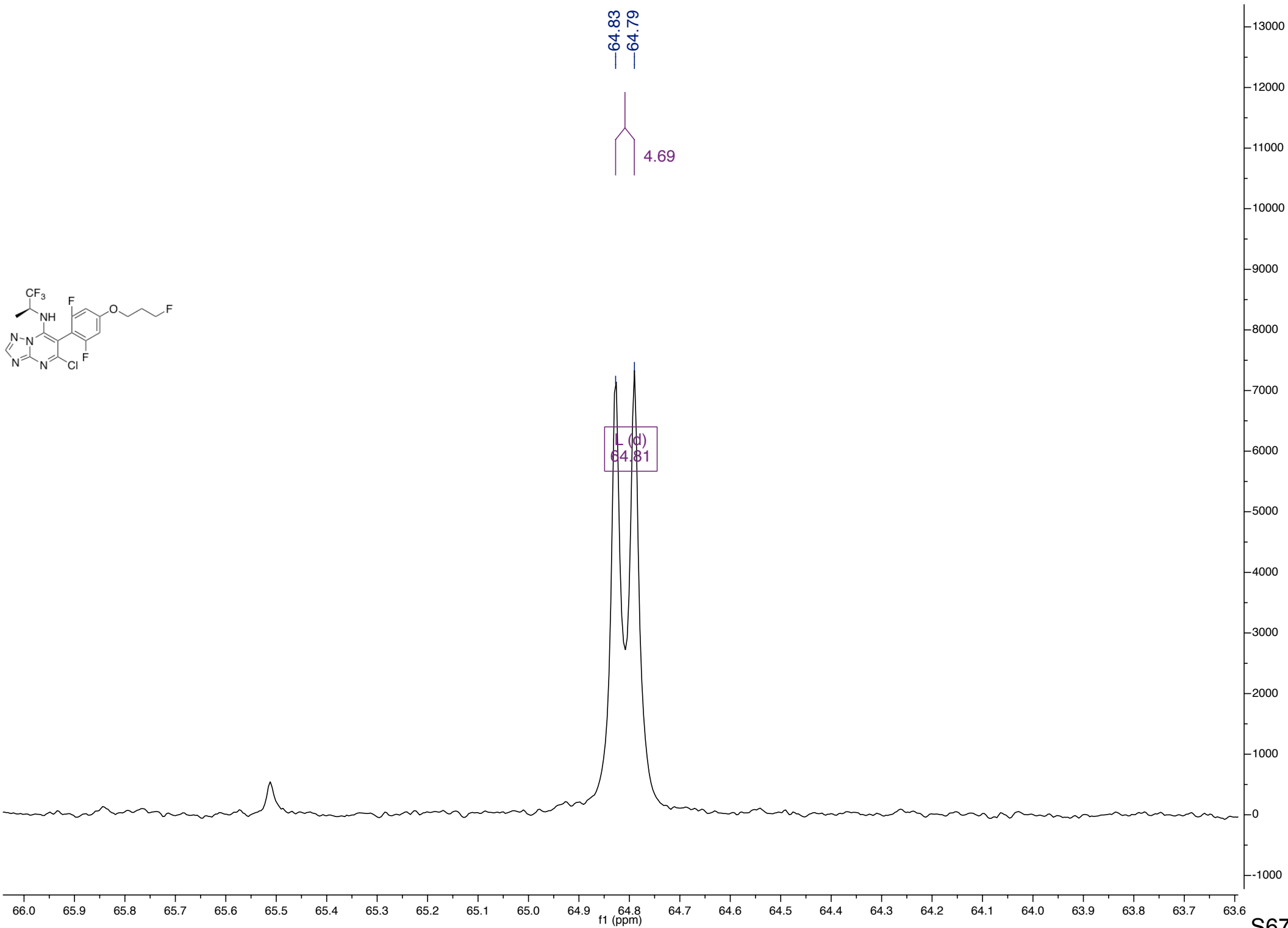
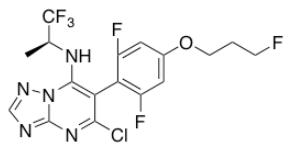


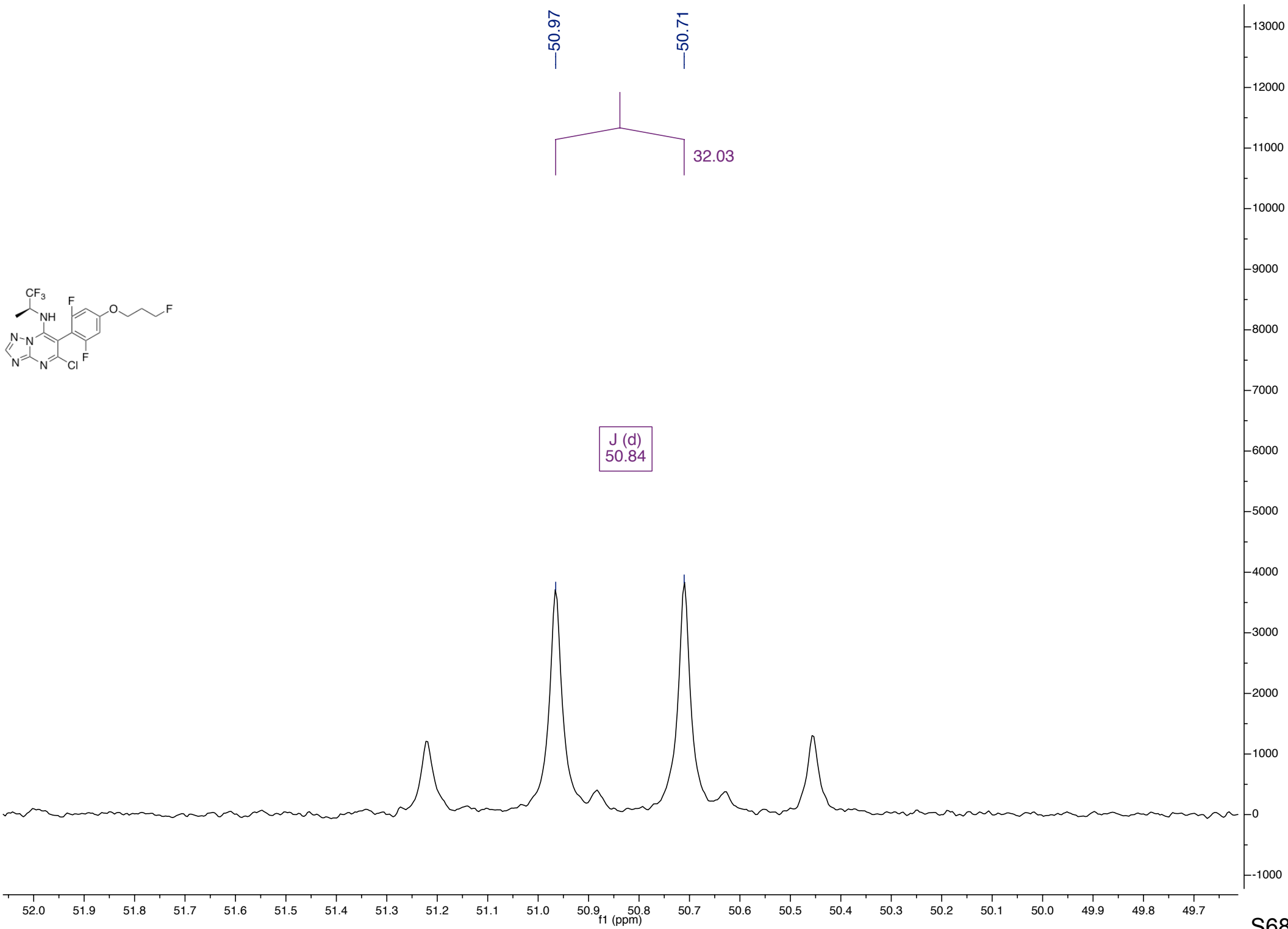
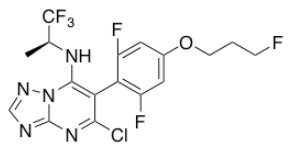


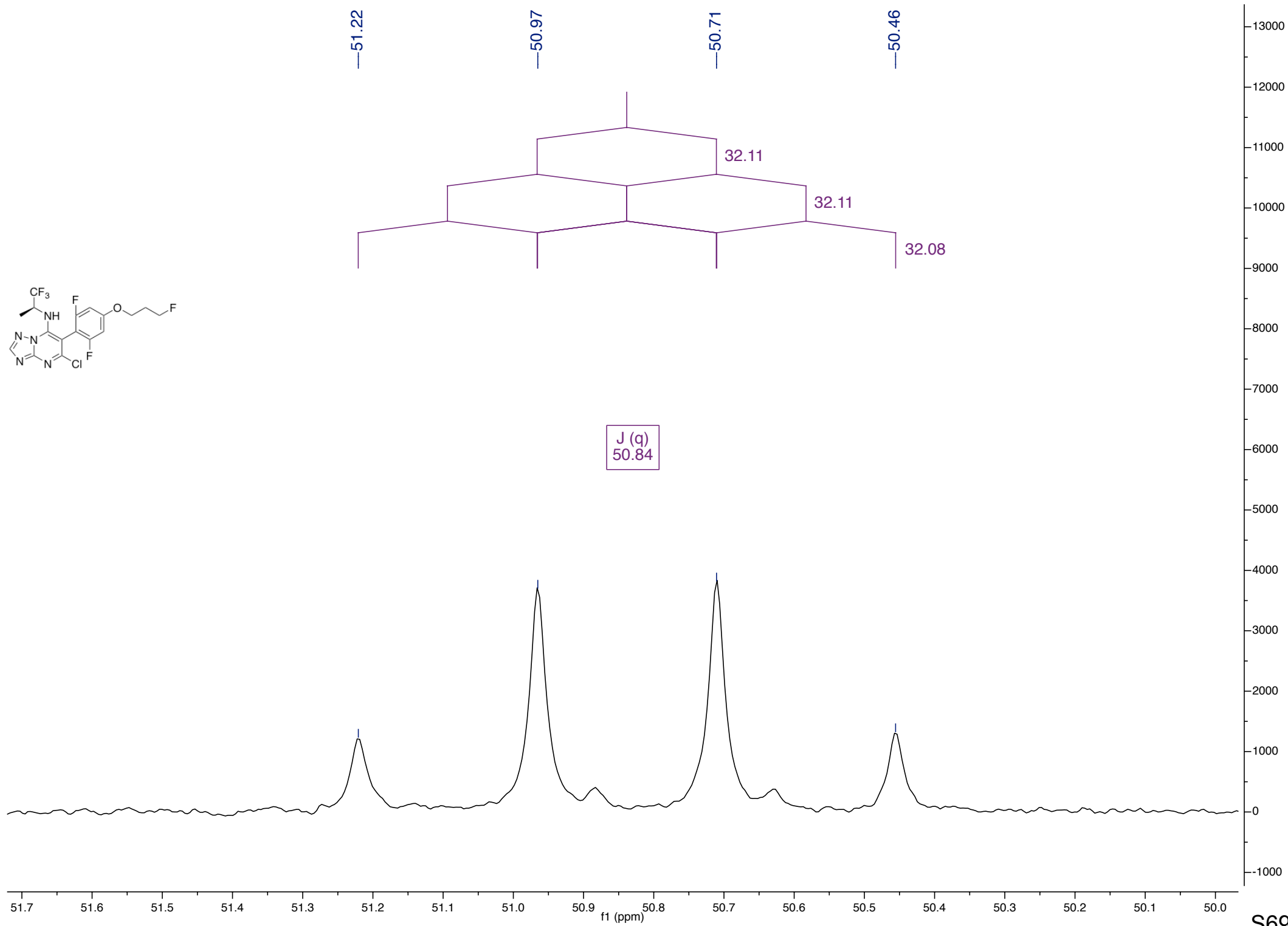
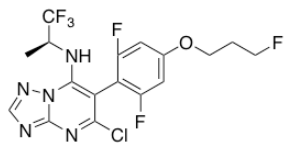


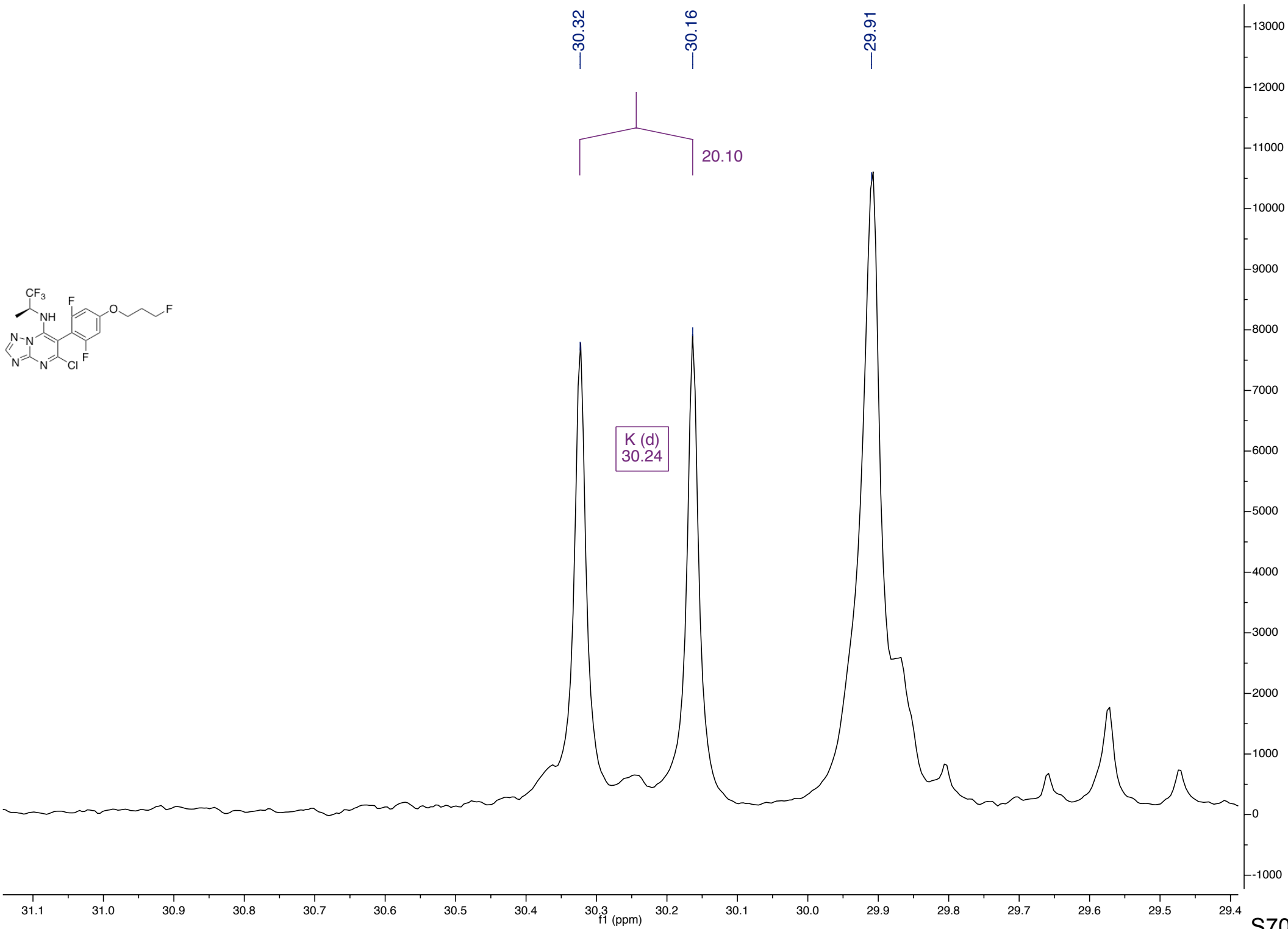
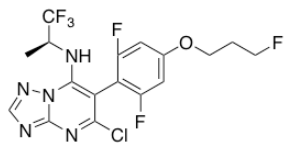


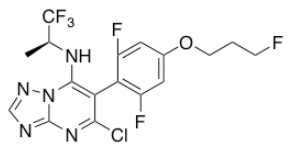




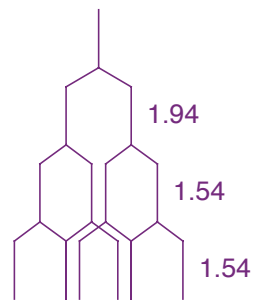




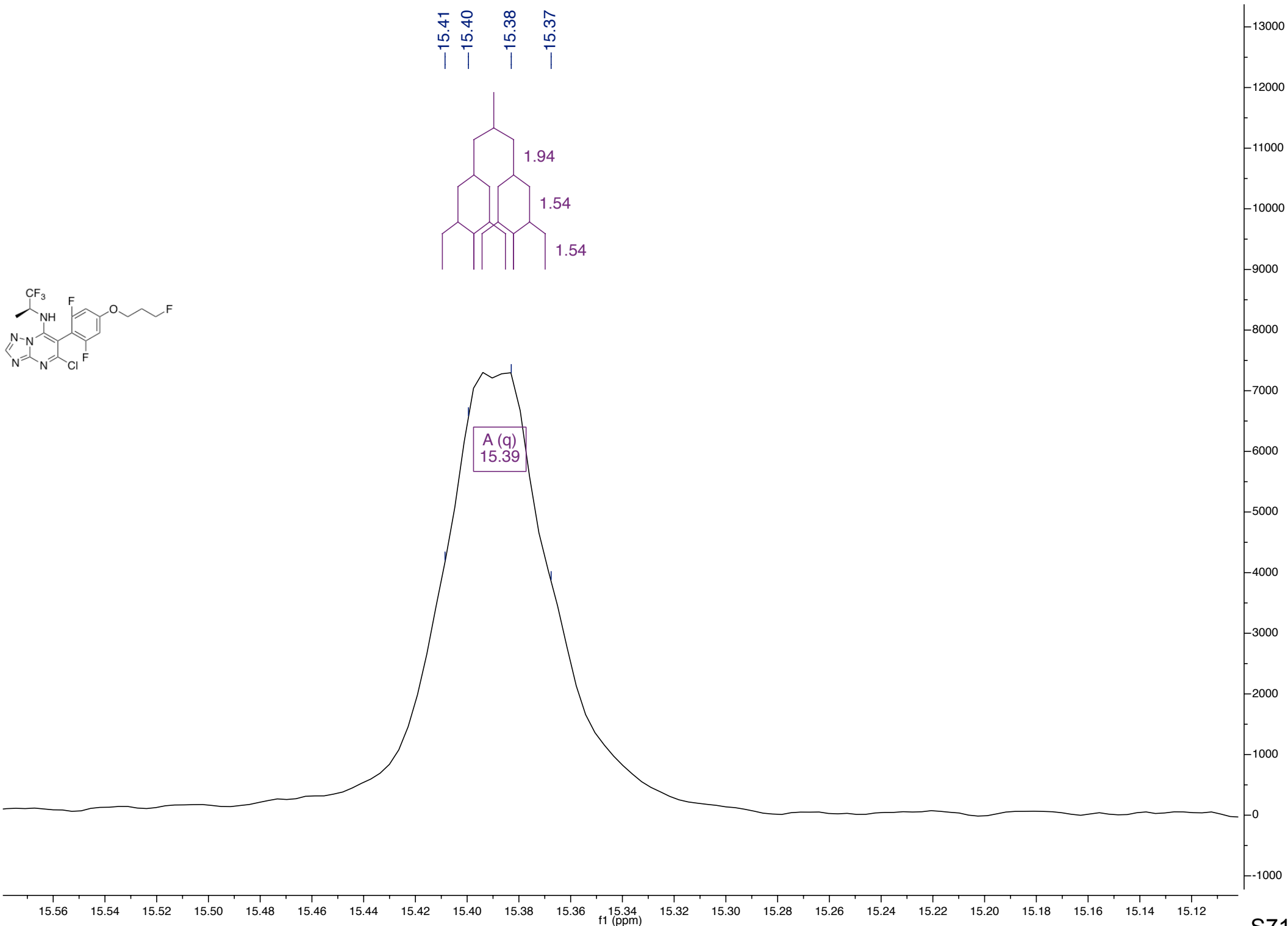


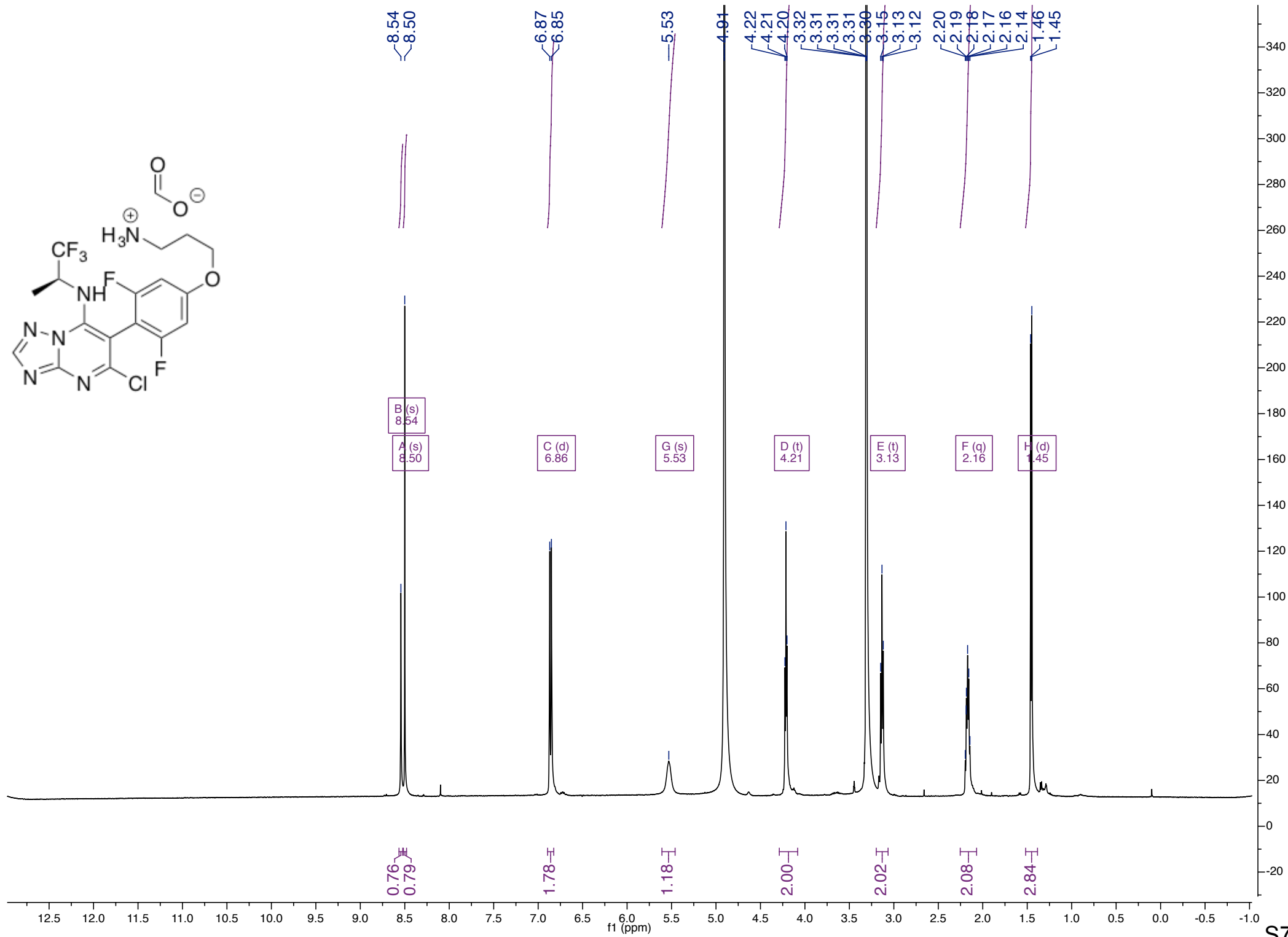
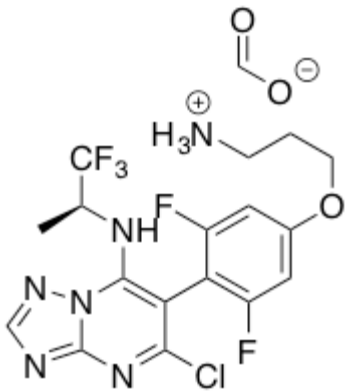


—15.41  
—15.40  
—15.38  
—15.37

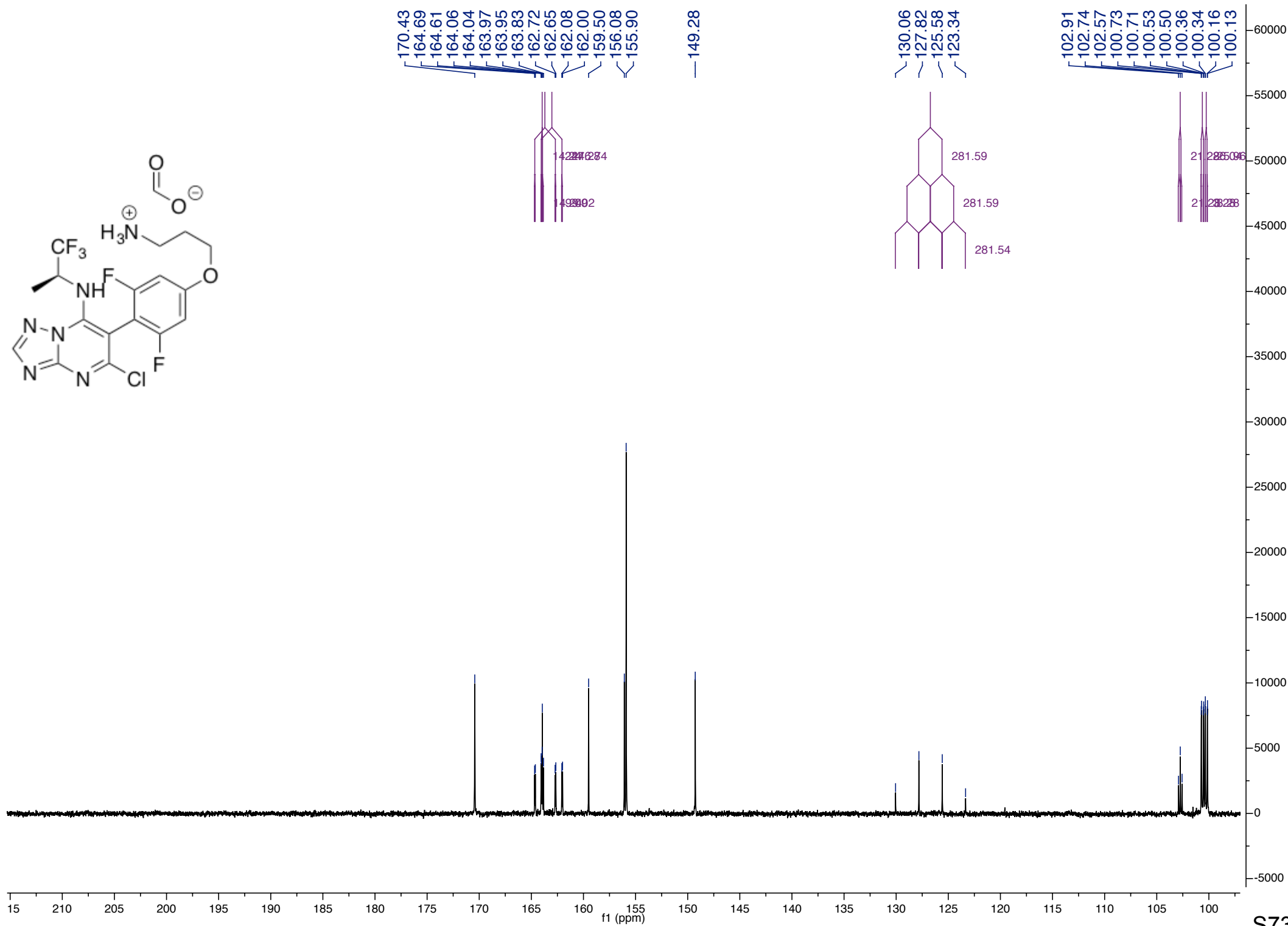
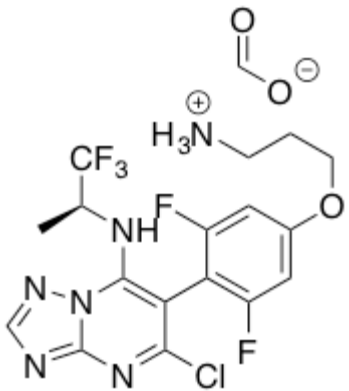


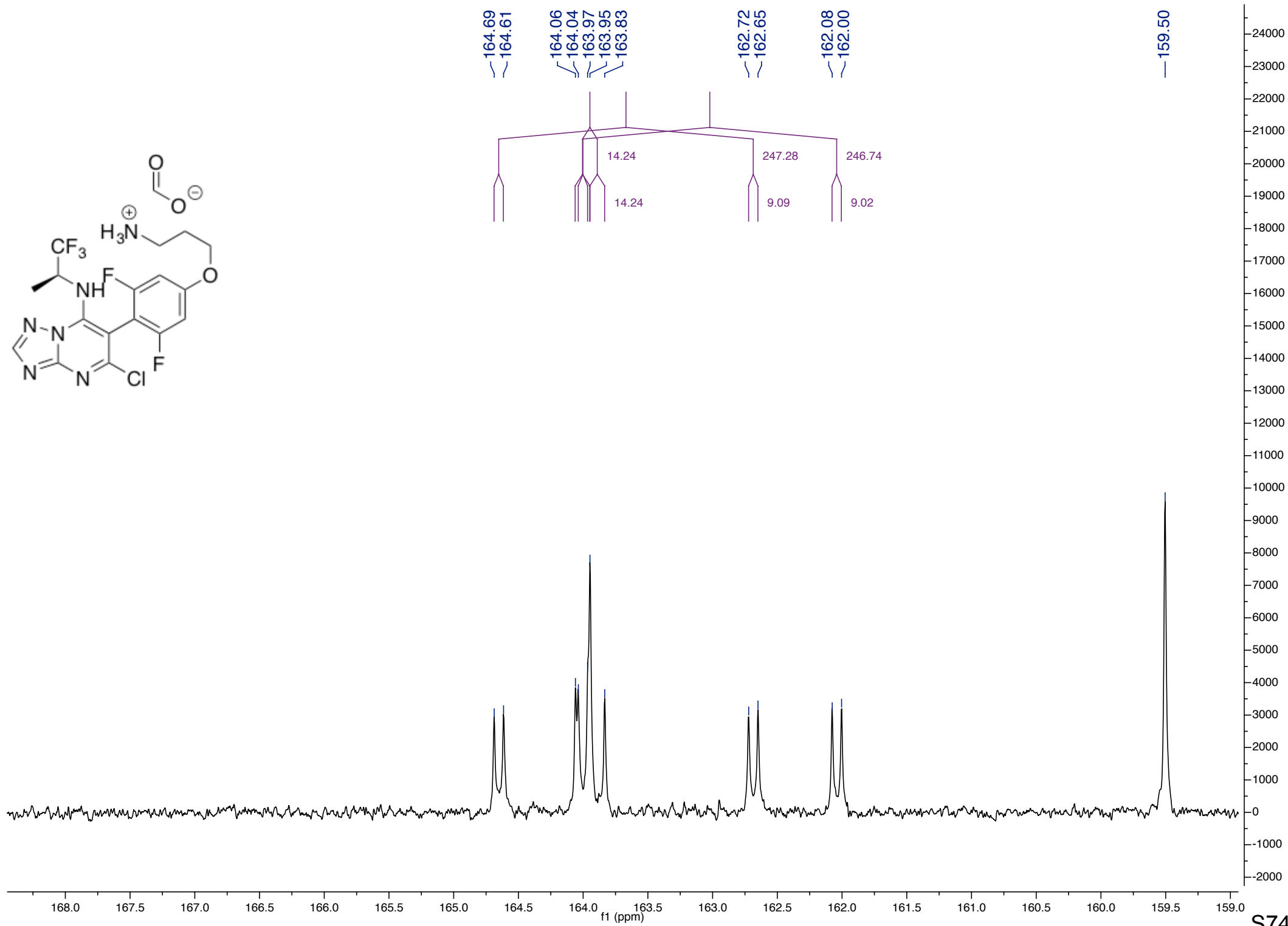
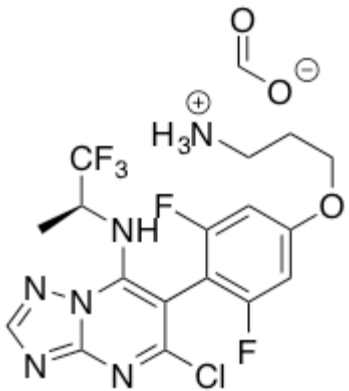
A (q)  
15.39

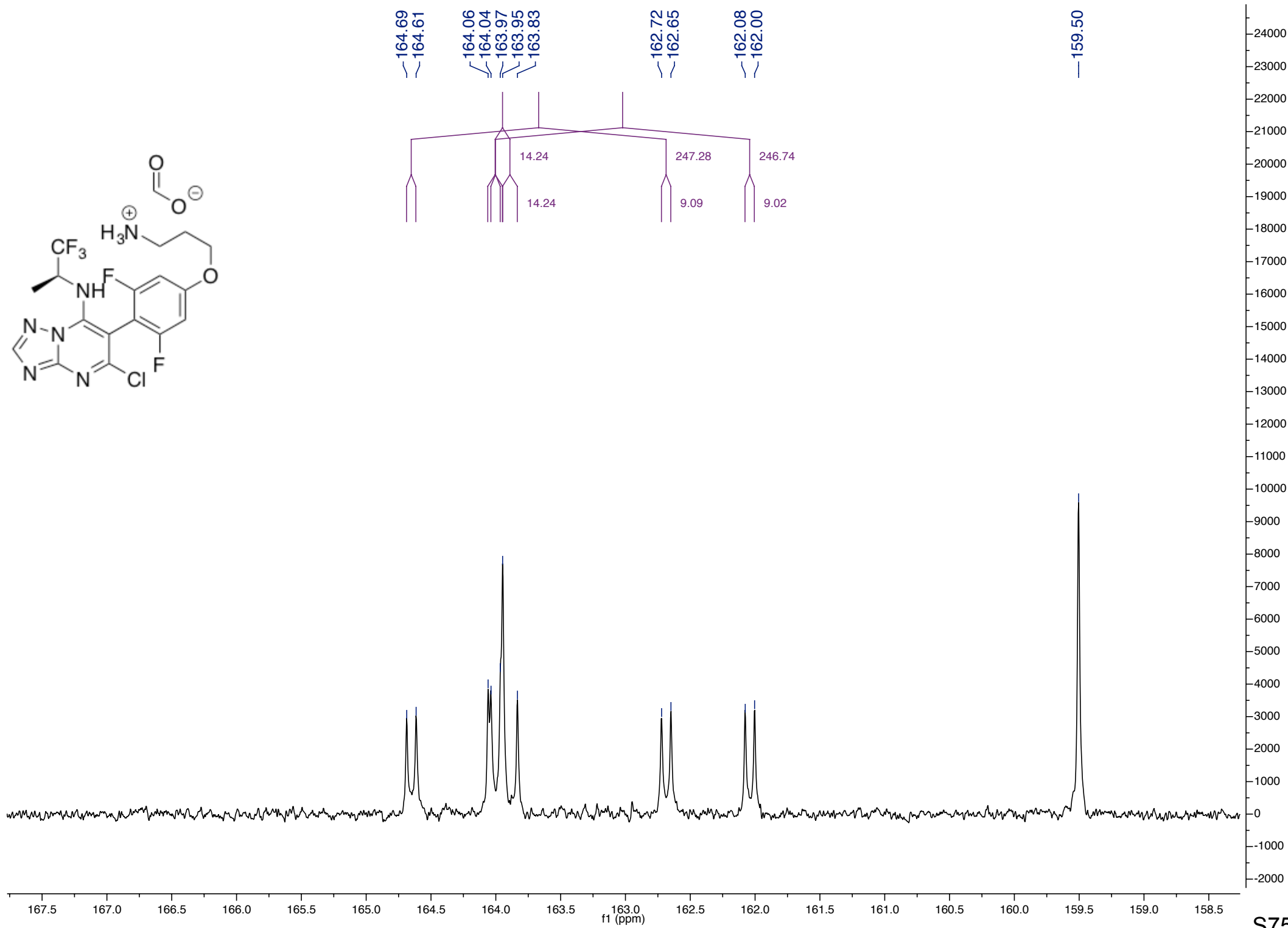
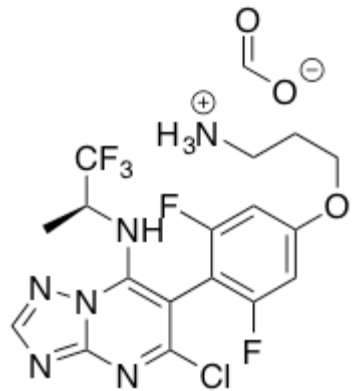


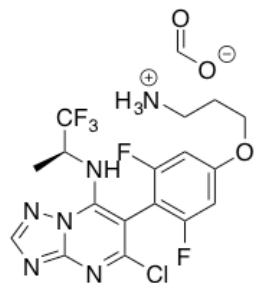










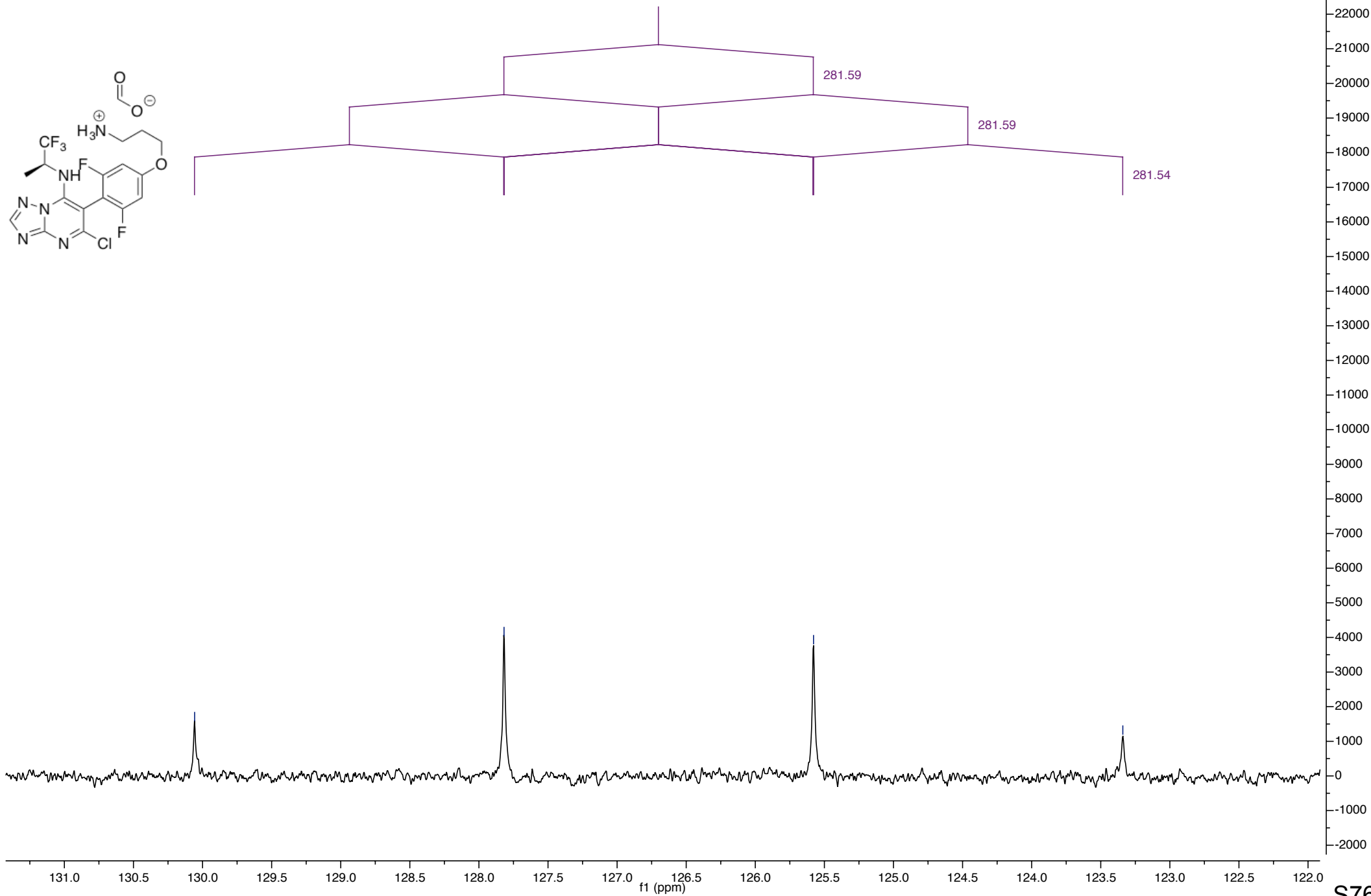


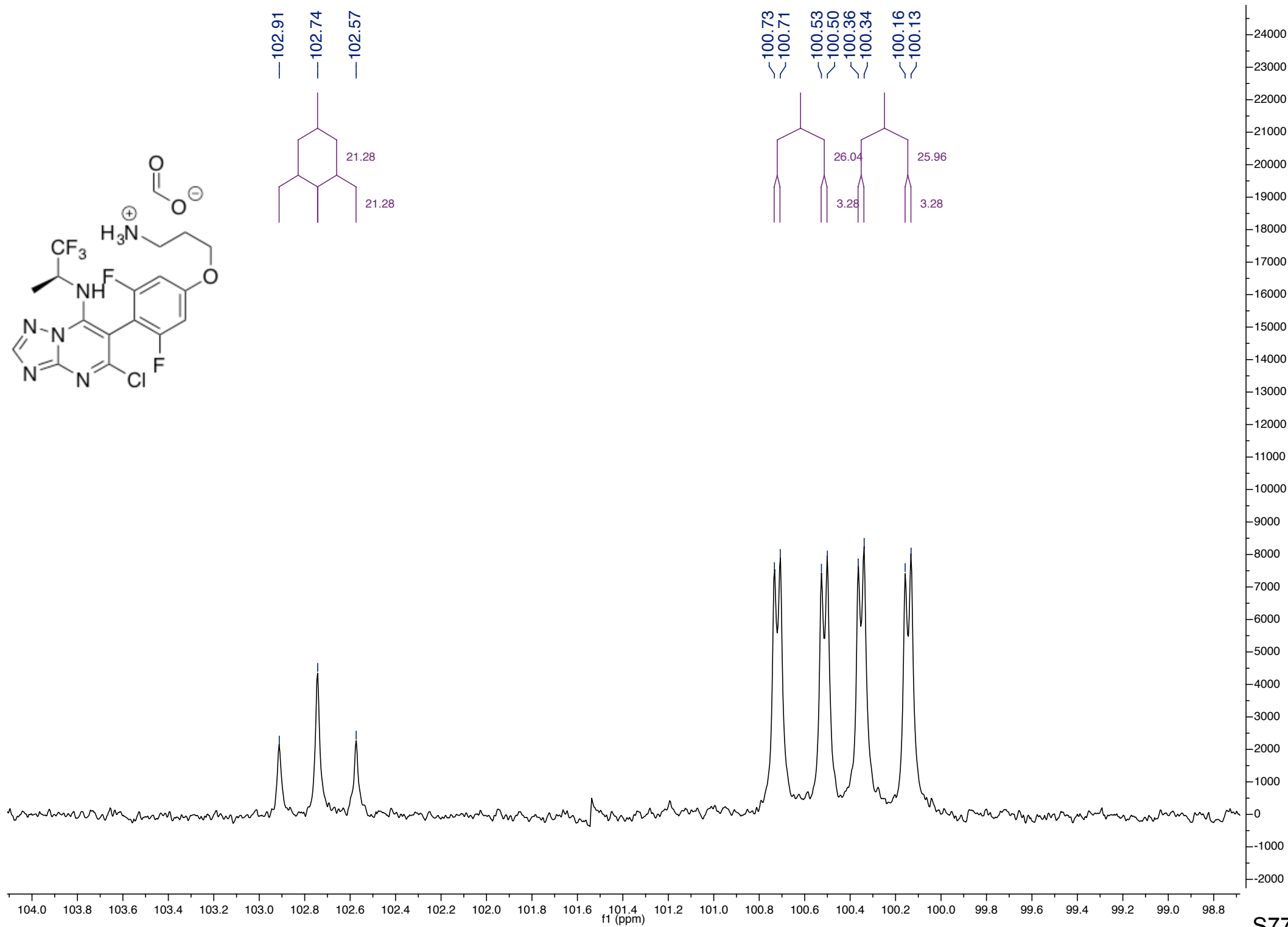
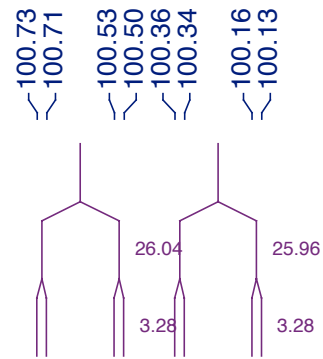
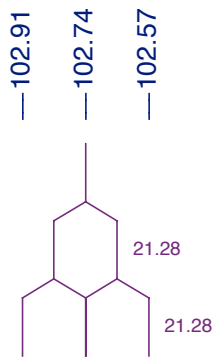
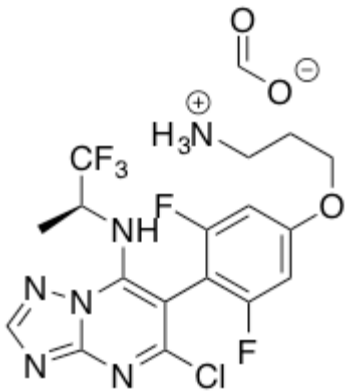
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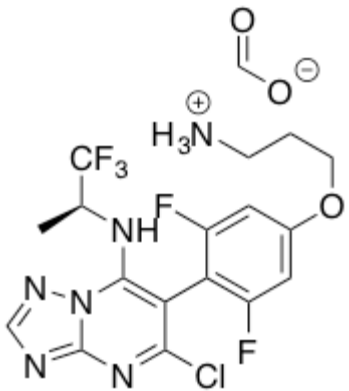
—127.82

—125.58

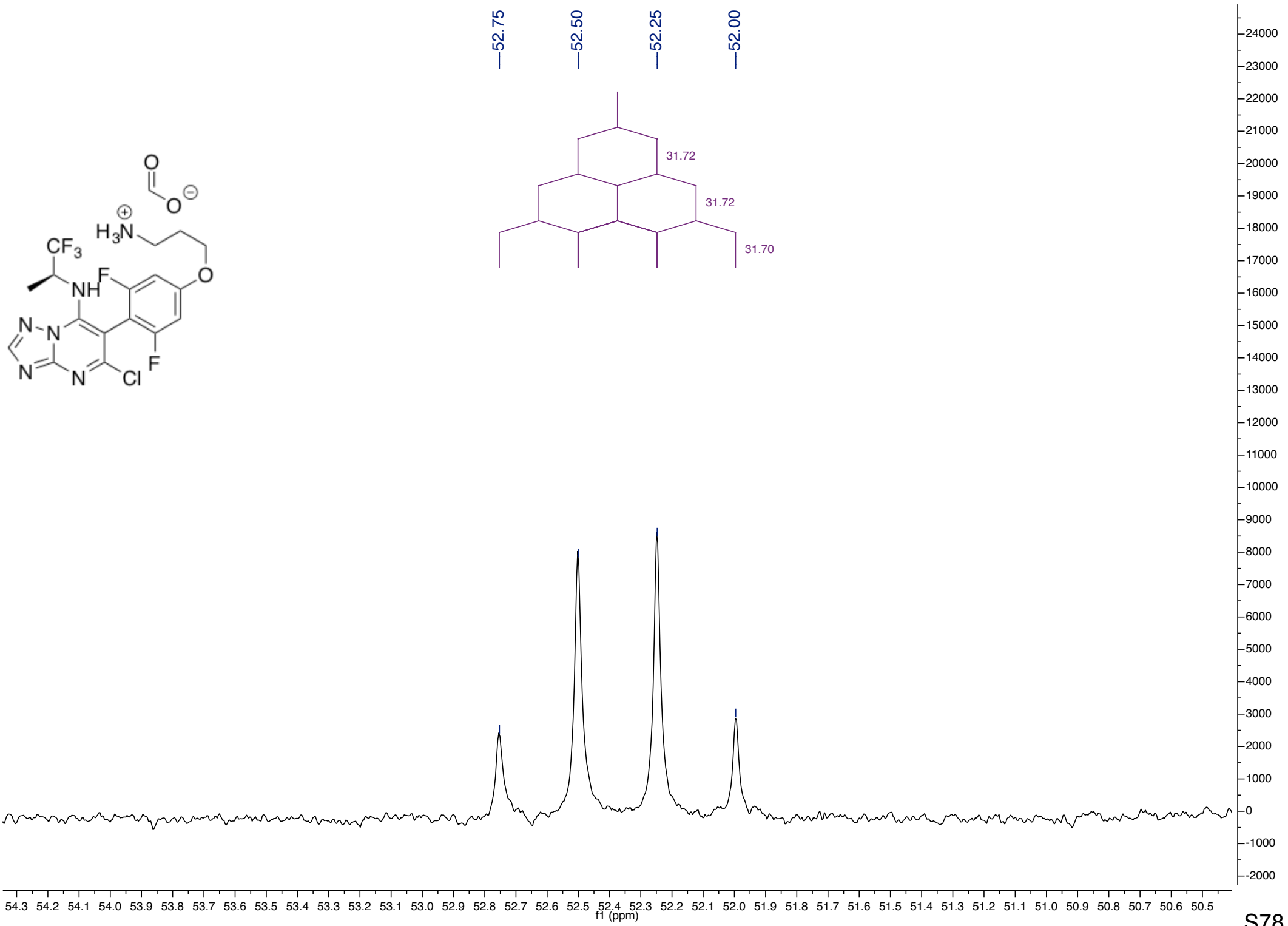
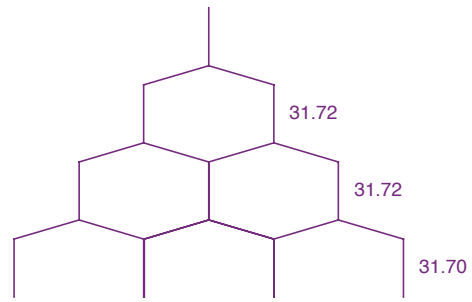
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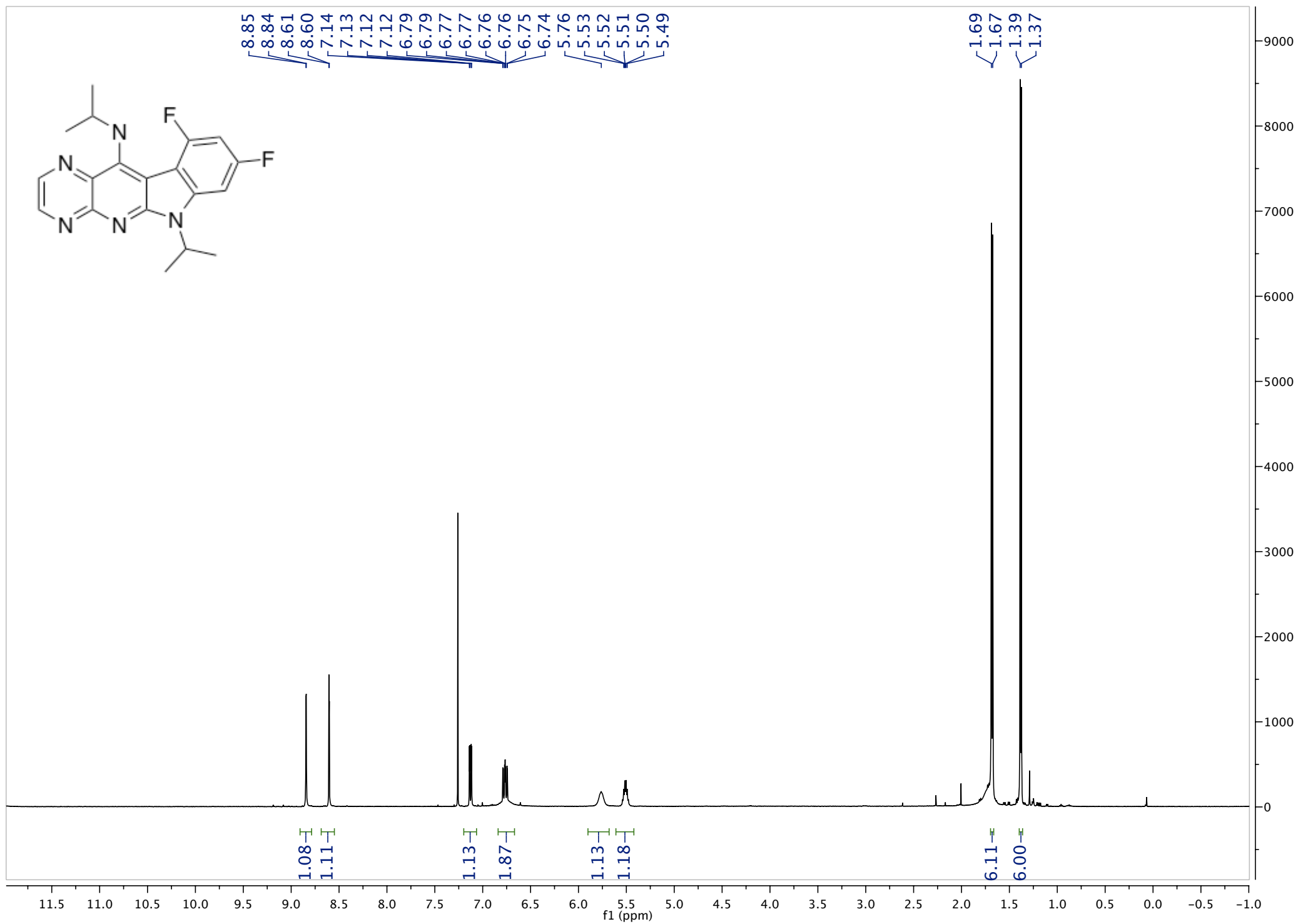


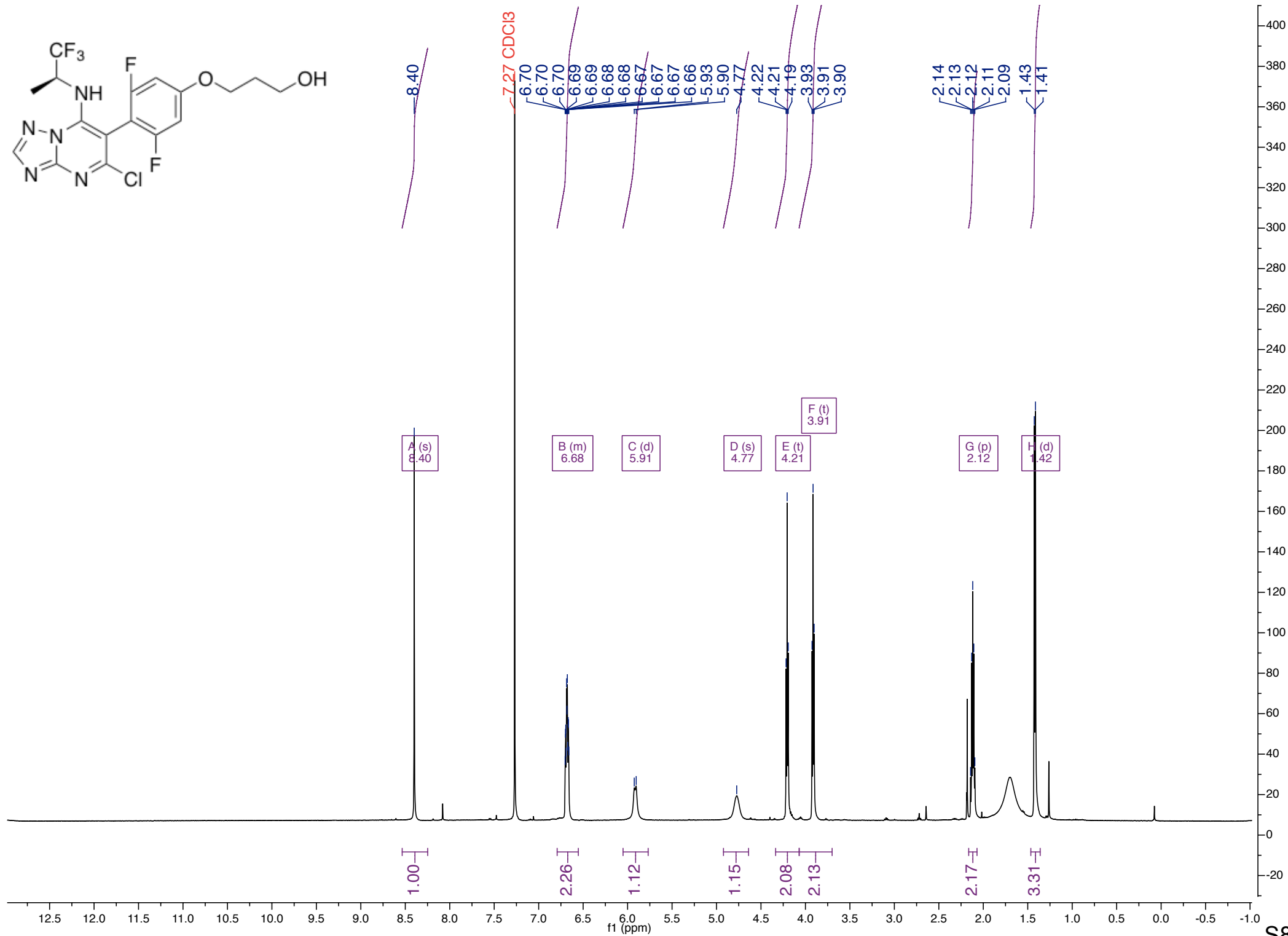
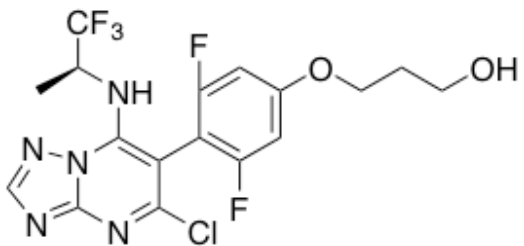




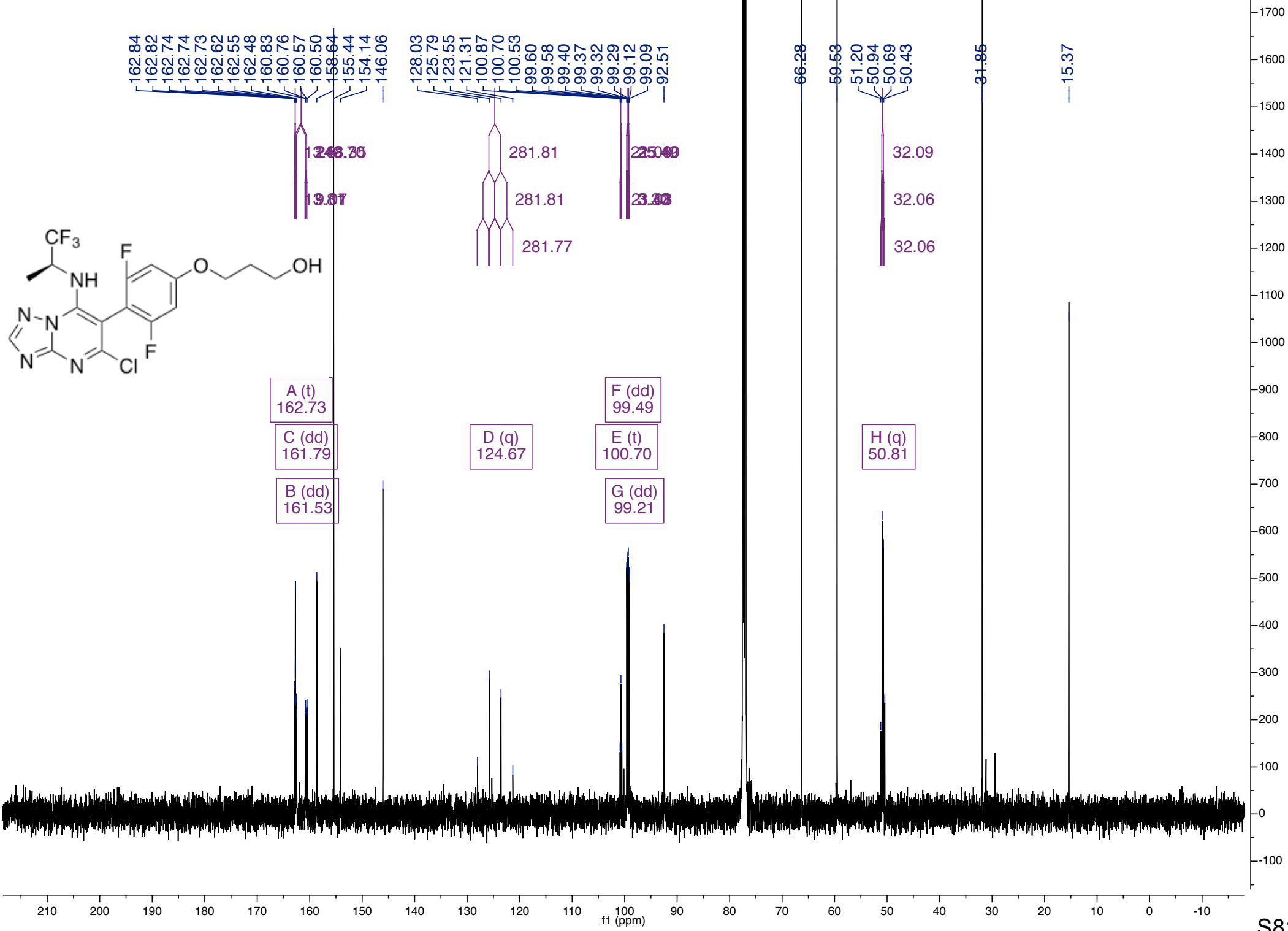
—52.75      —52.50      —52.25      —52.00

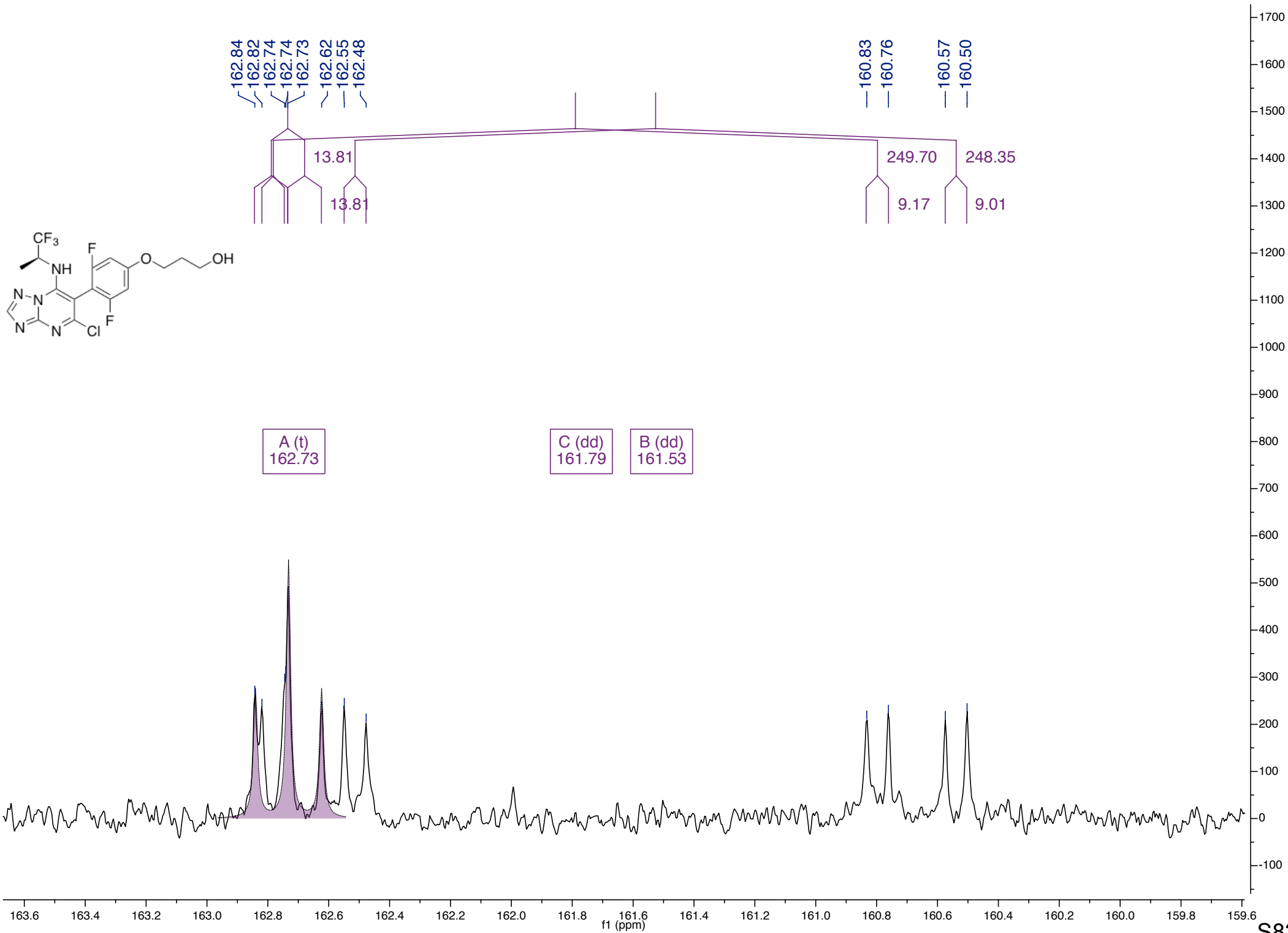
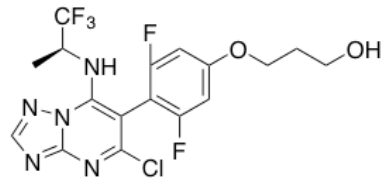


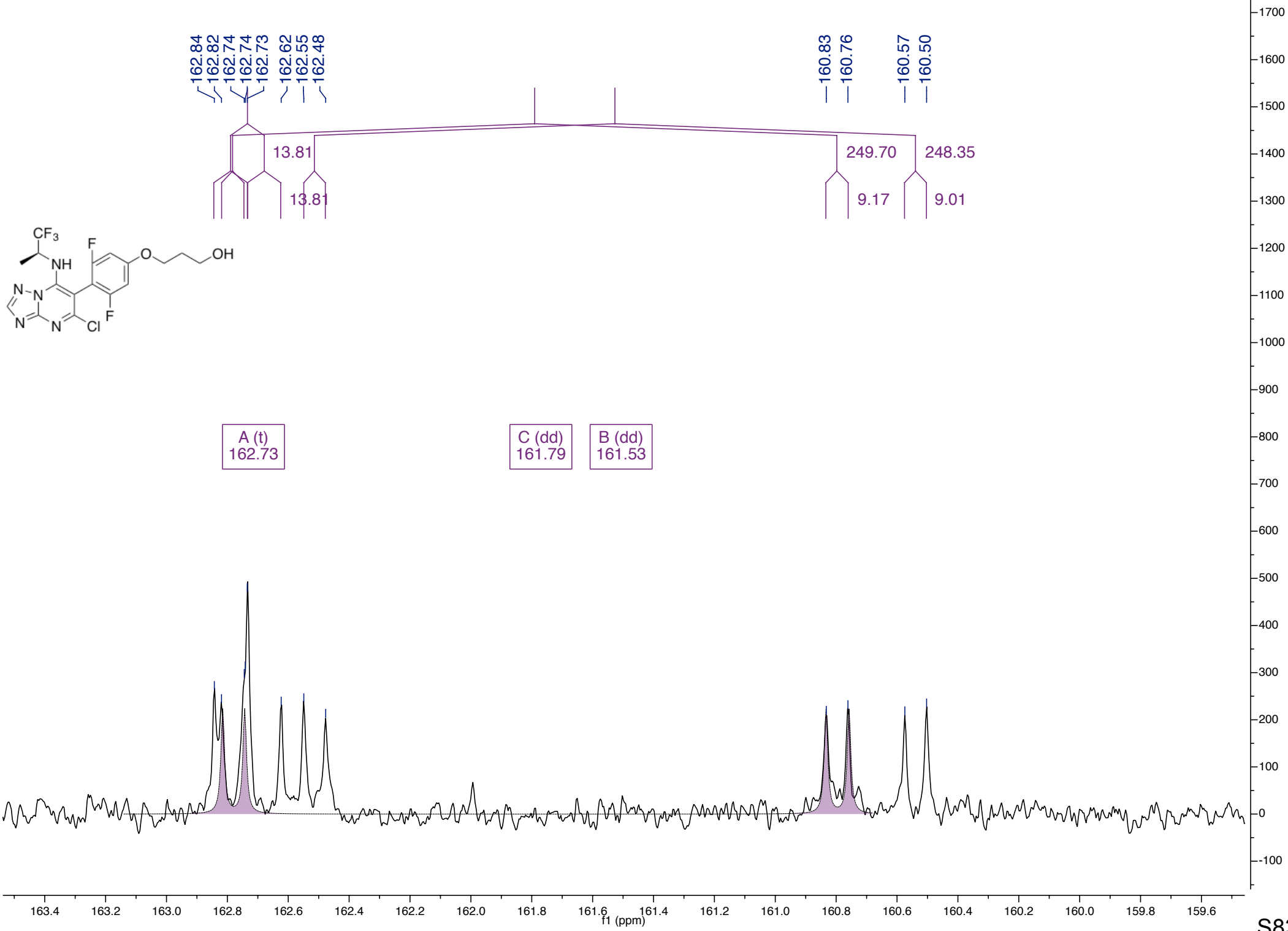


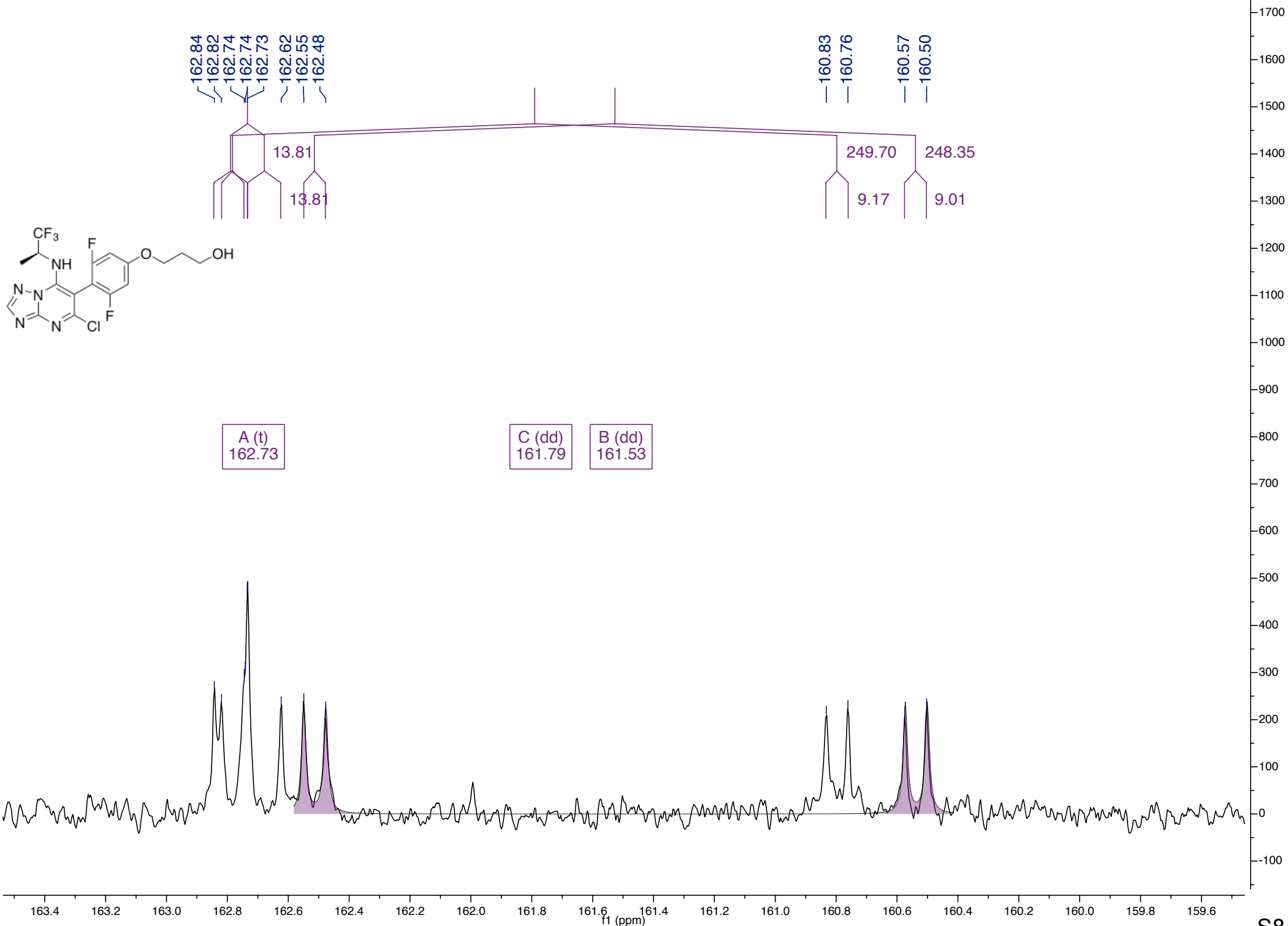


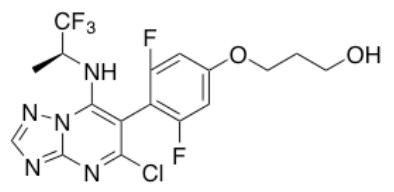
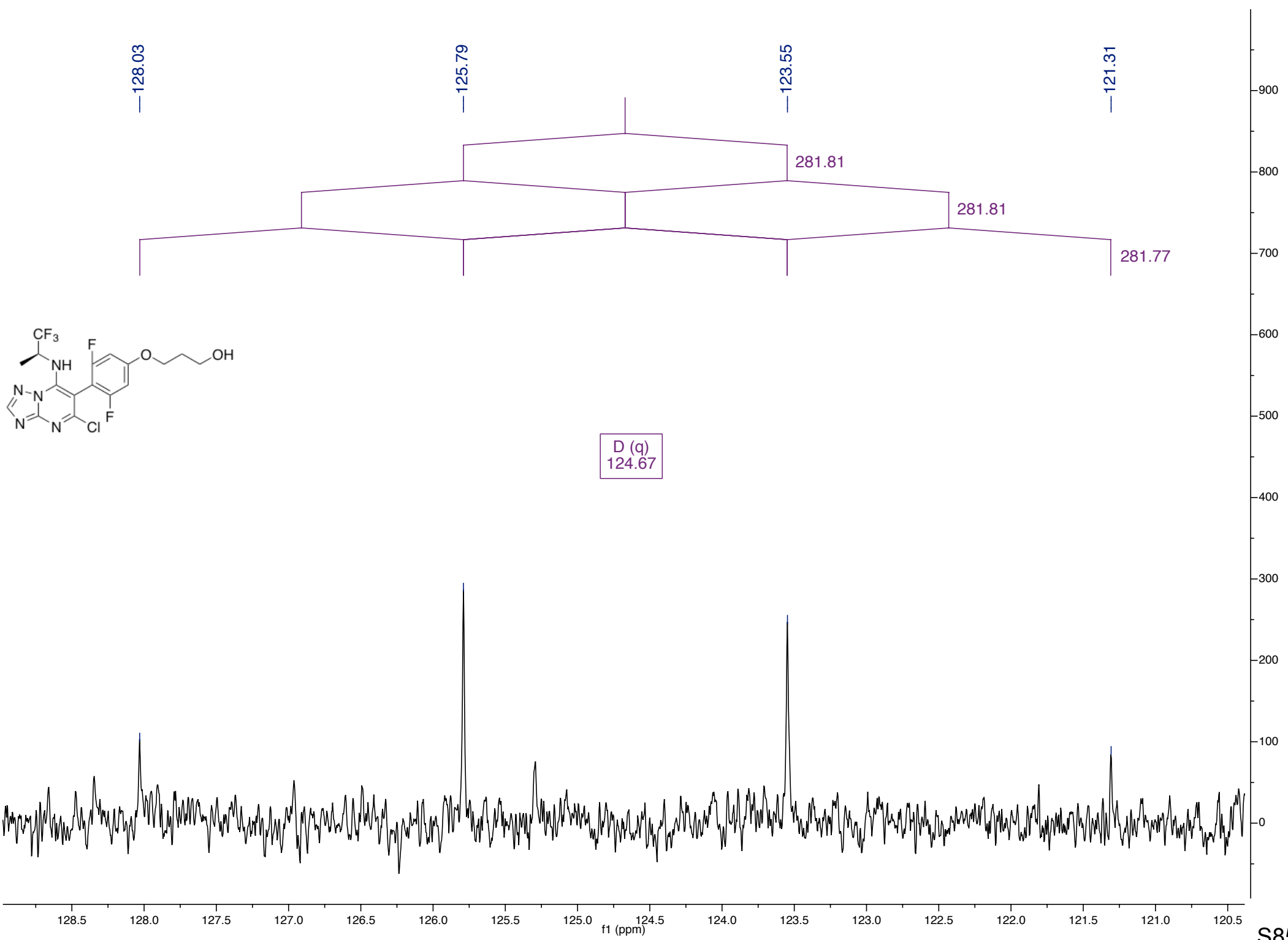


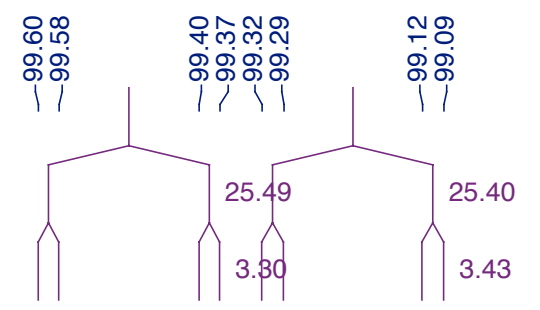
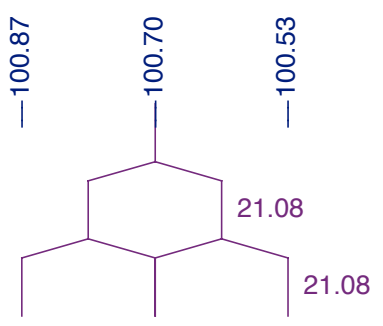
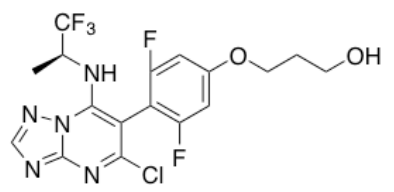








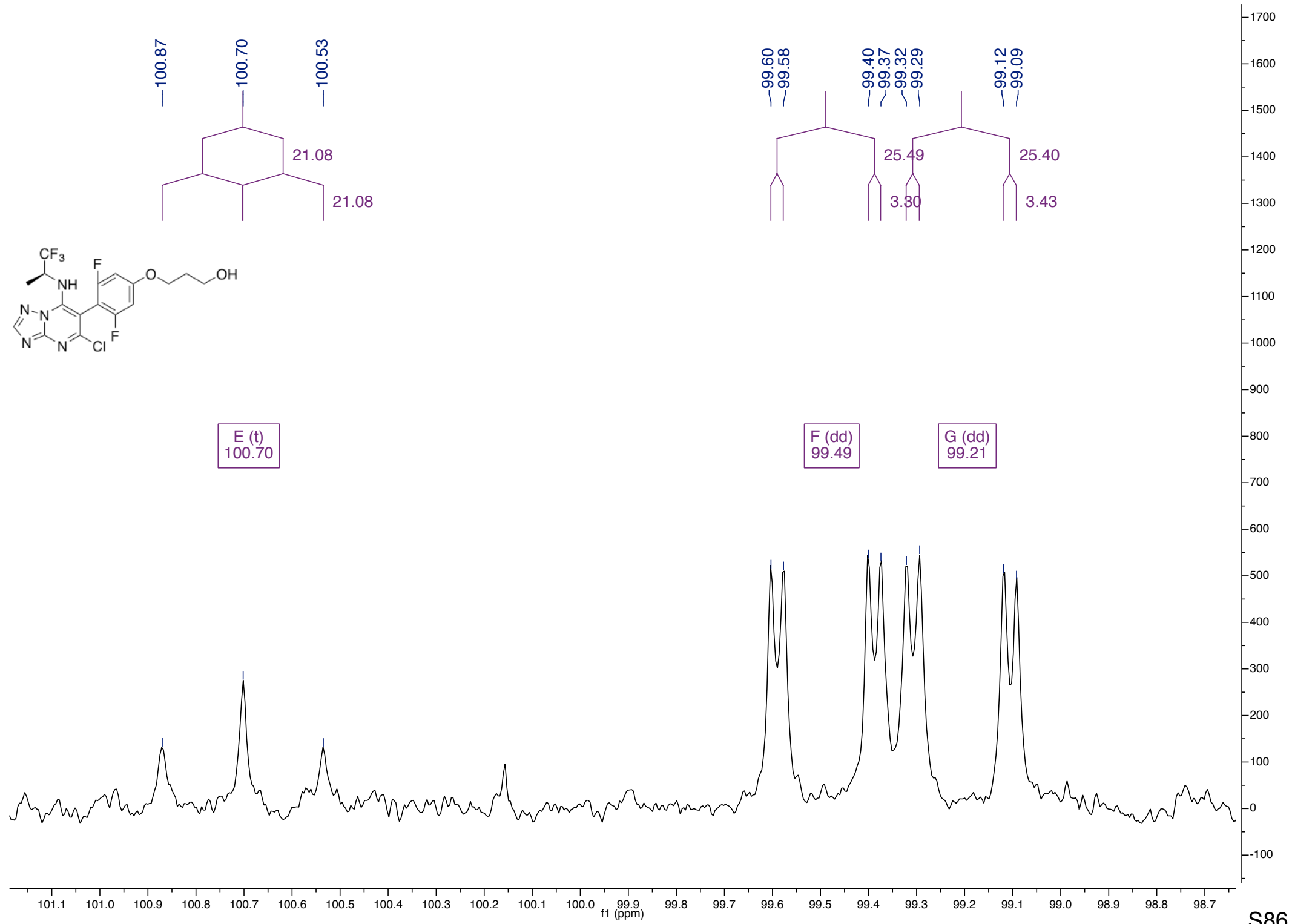


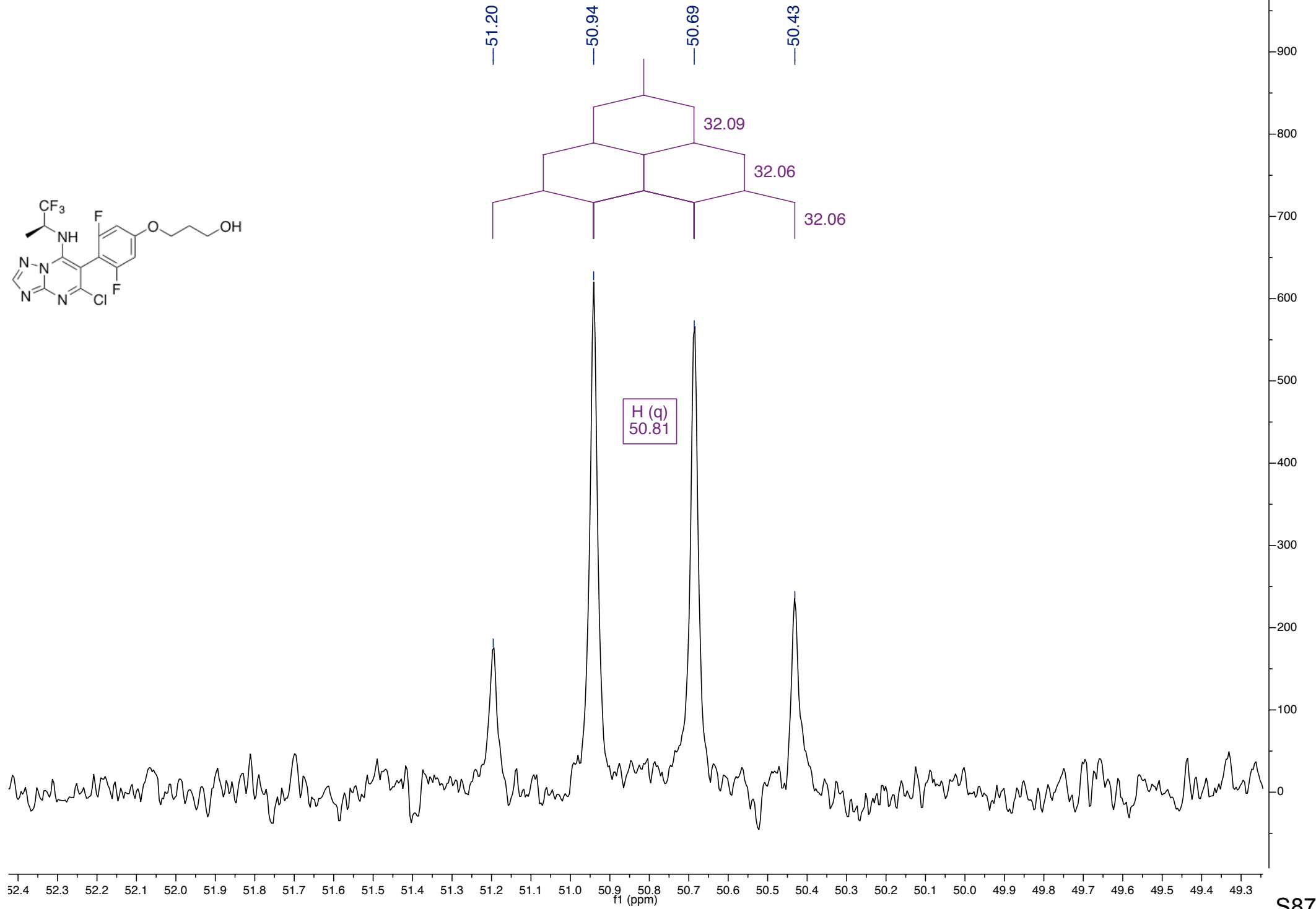
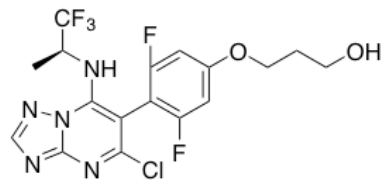


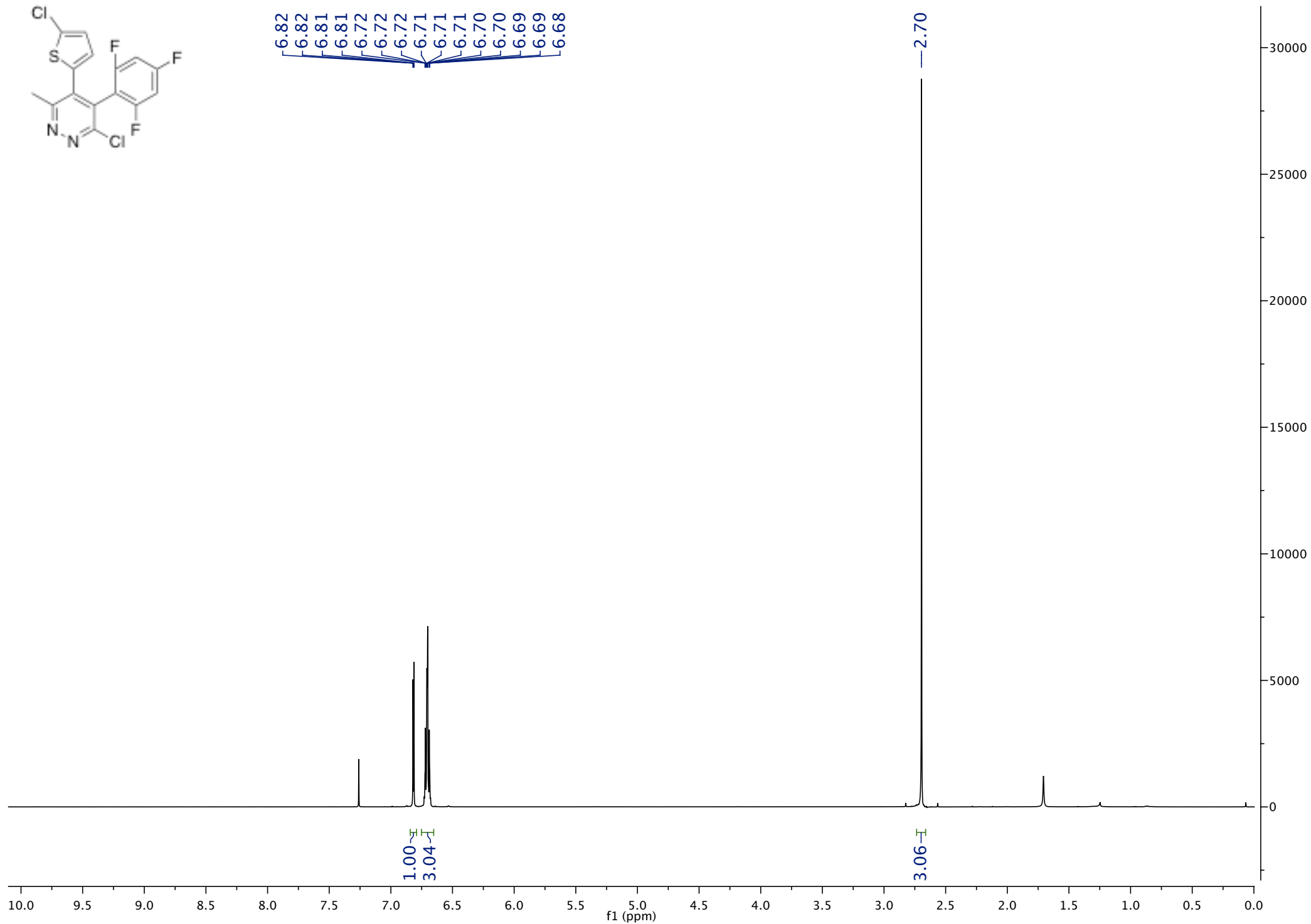
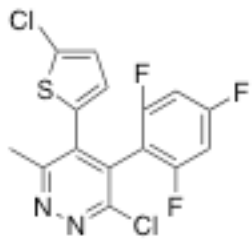
E (t)  
100.70

F (dd)  
99.49

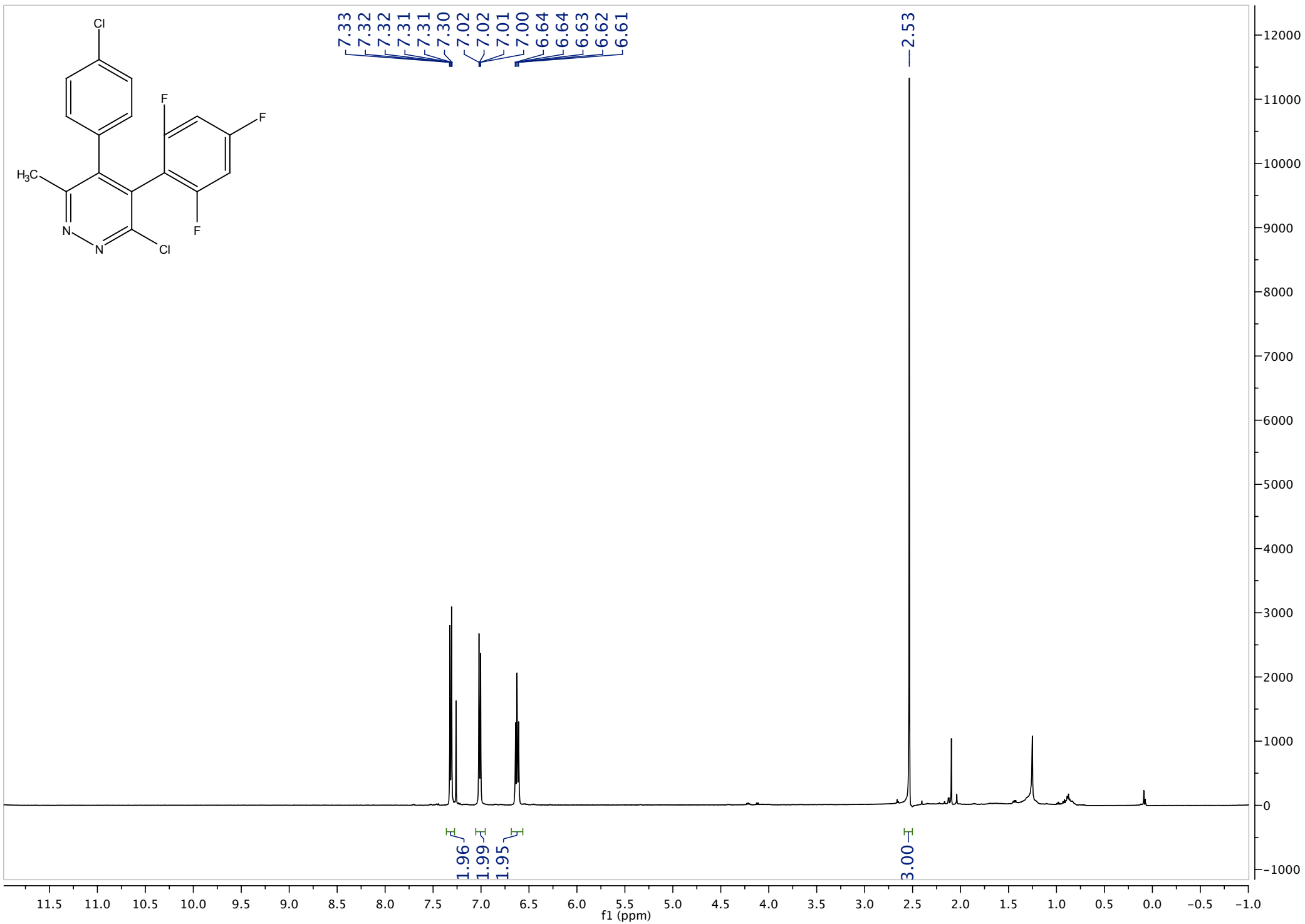
G (dd)  
99.21

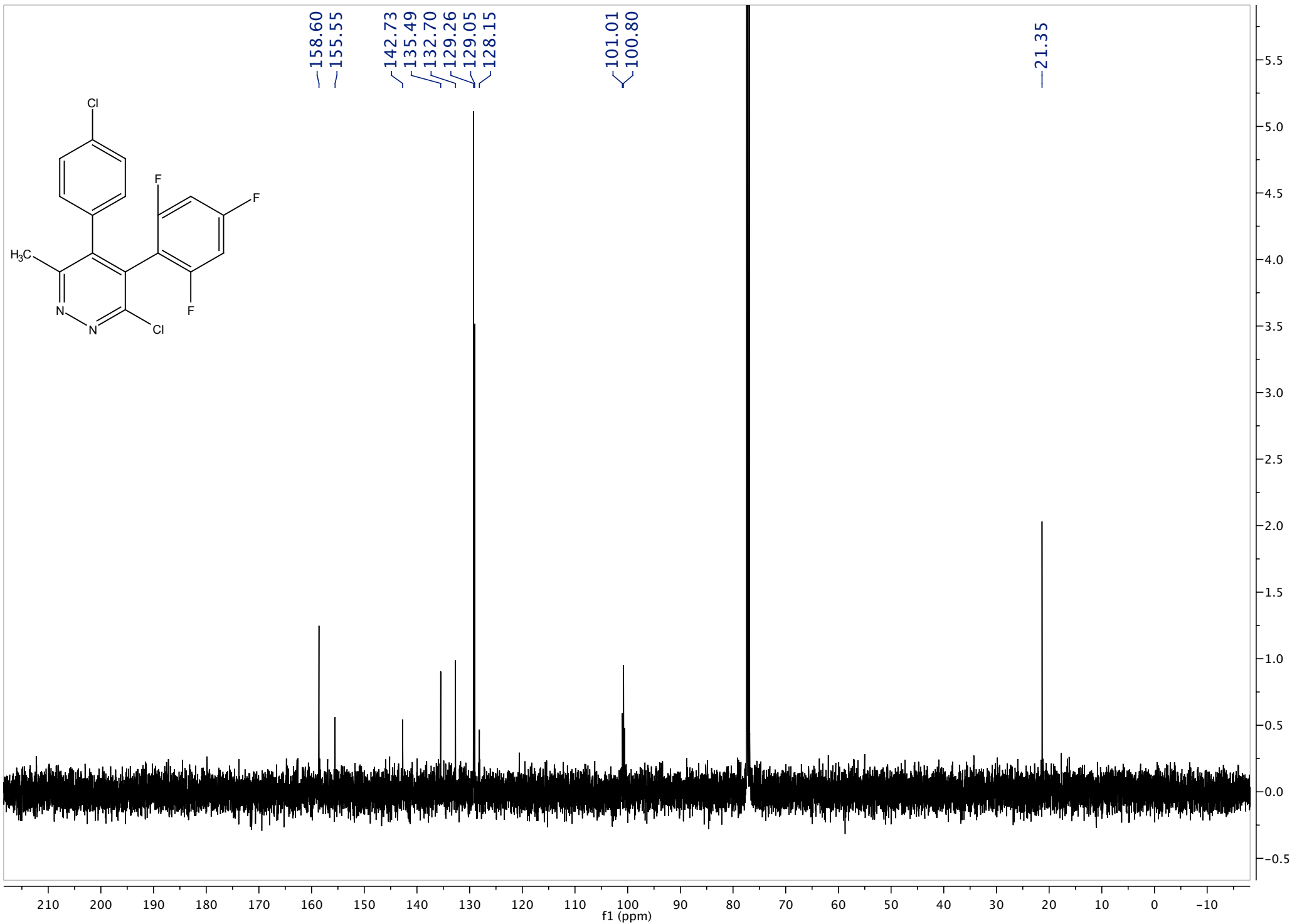


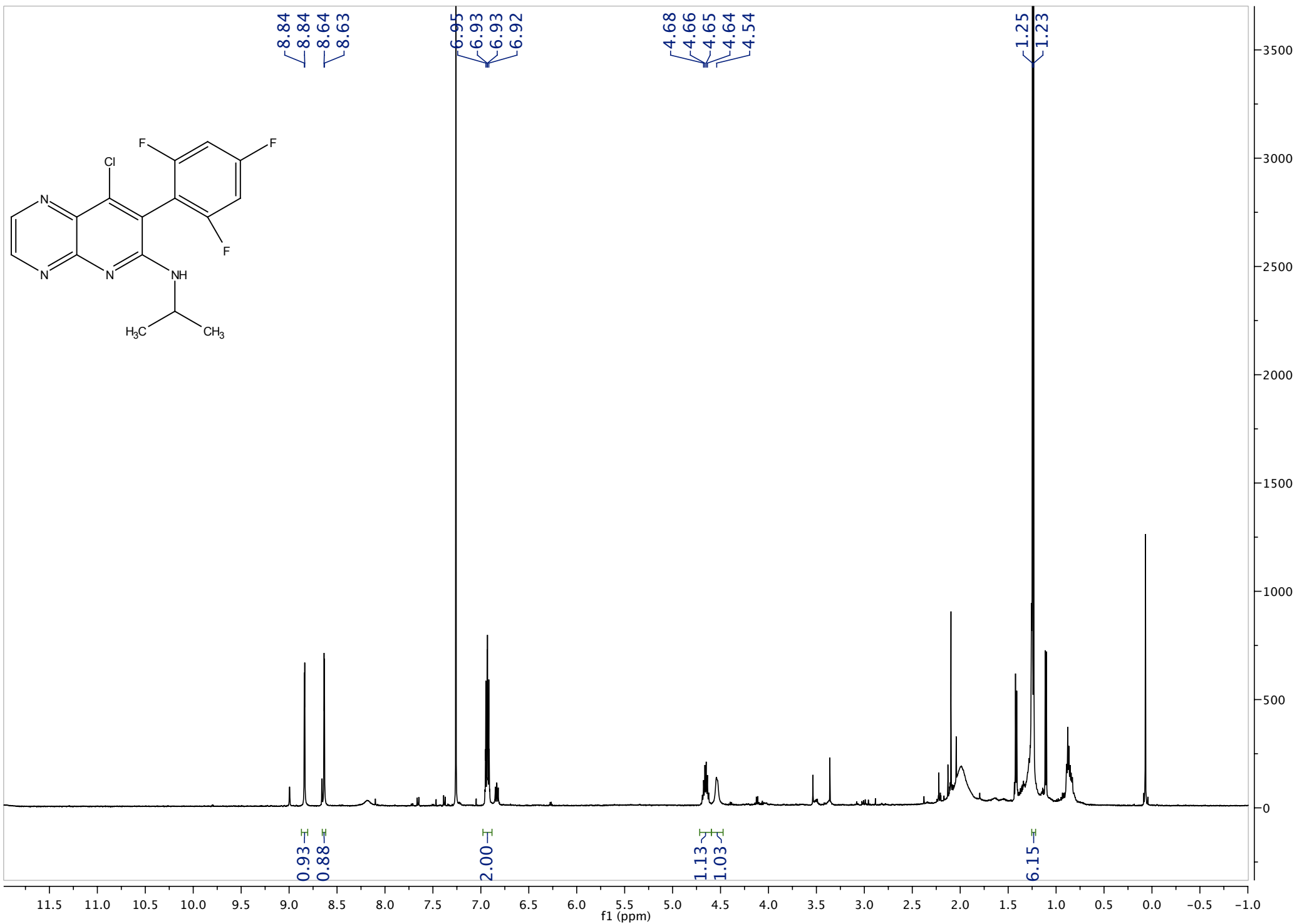


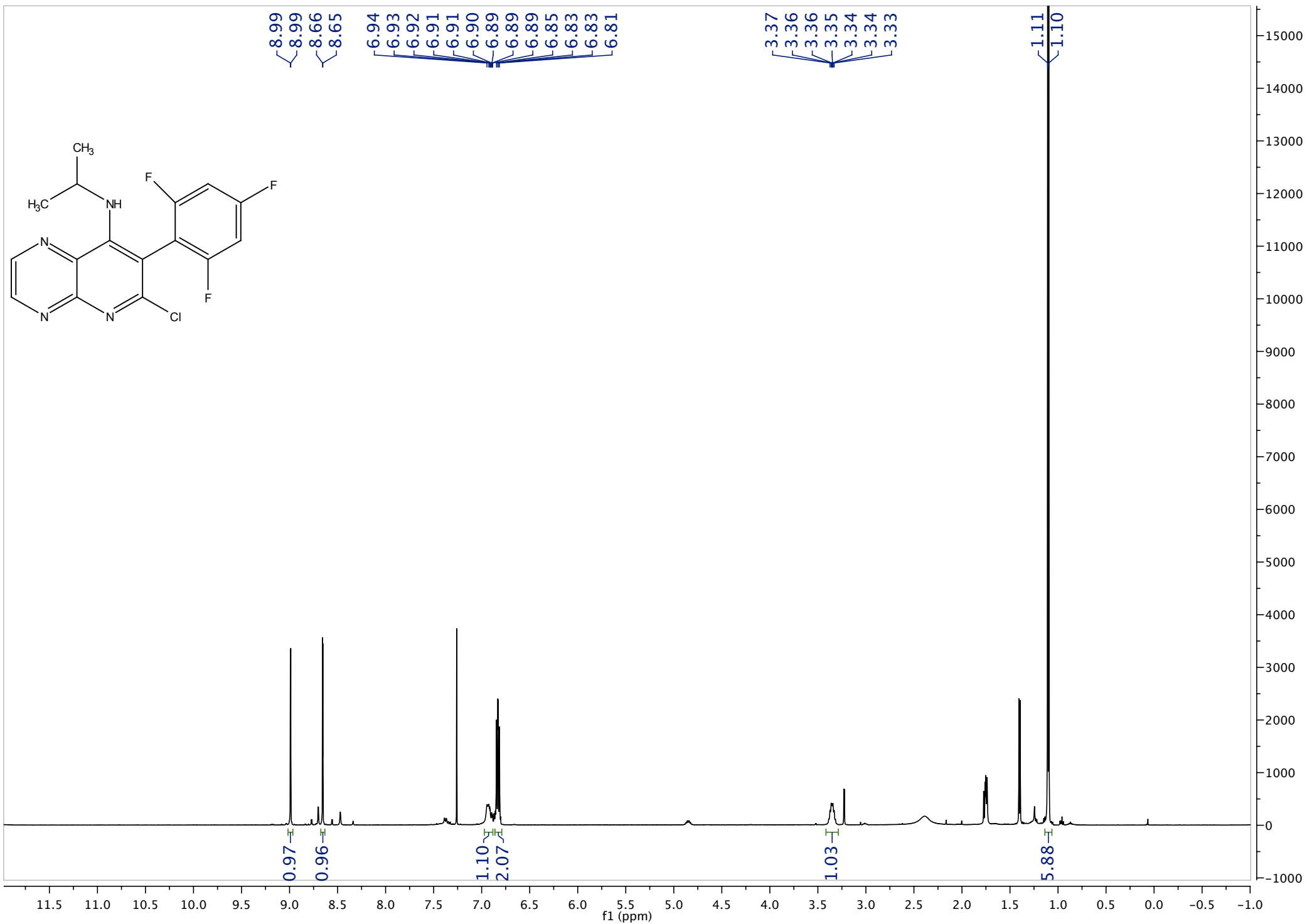


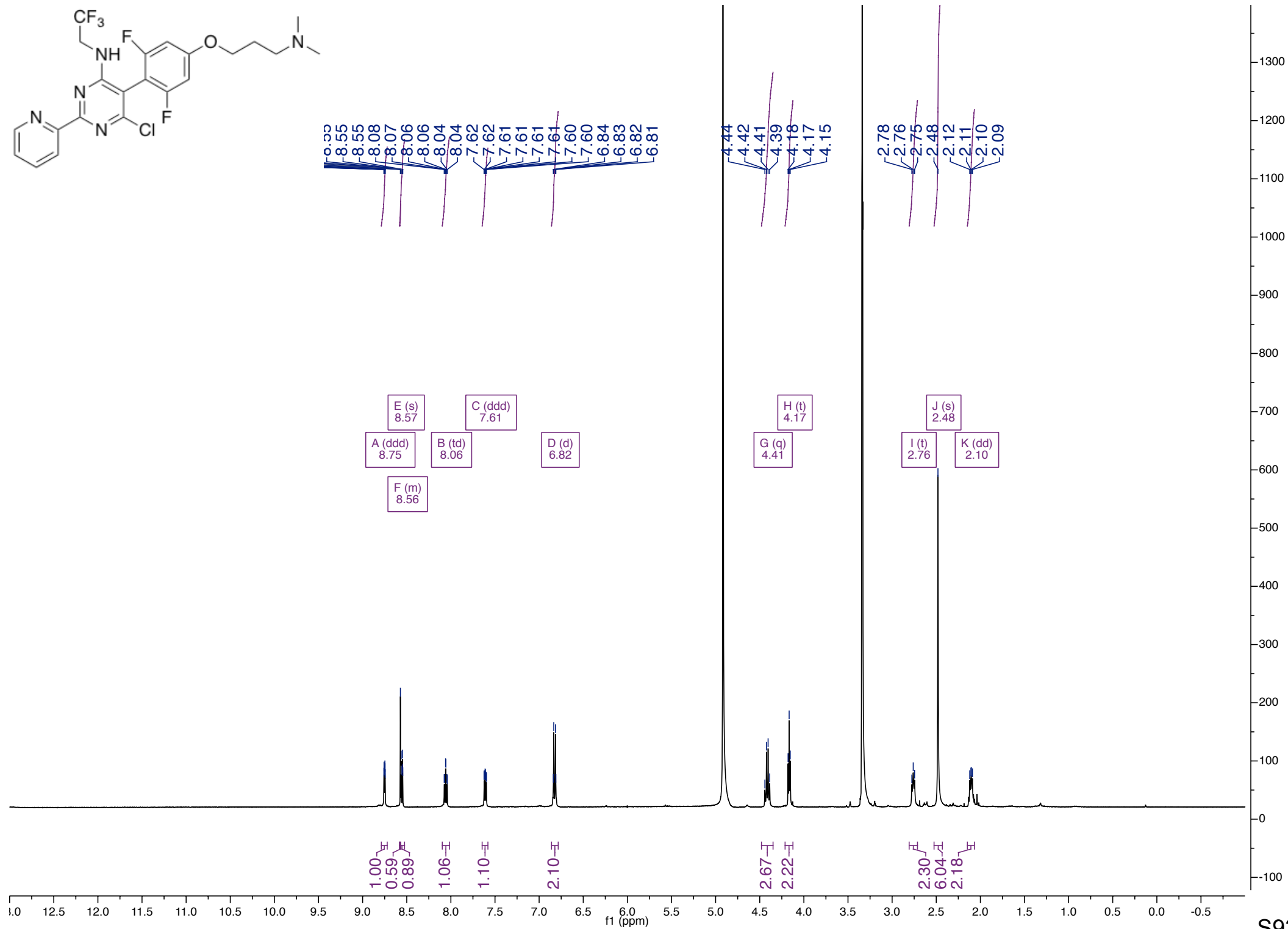
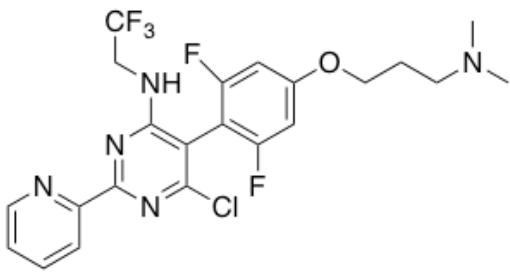


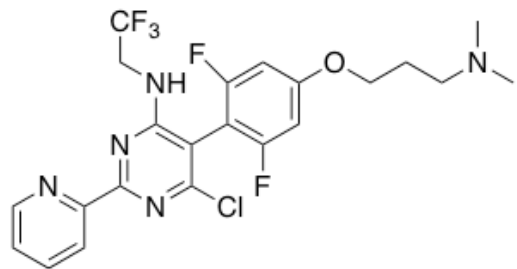












170.36  
164.16  
163.84  
163.81  
163.76  
163.70  
163.67  
163.59  
161.93  
161.88  
161.80  
154.88  
150.44

139.20  
129.67  
127.45  
127.18  
125.50  
125.23  
123.01  
106.57  
102.51  
102.34  
102.16  
100.48  
100.47  
100.44  
100.30  
100.26  
100.25

68.10

57.15

45.20  
43.32  
43.04  
42.77  
42.49

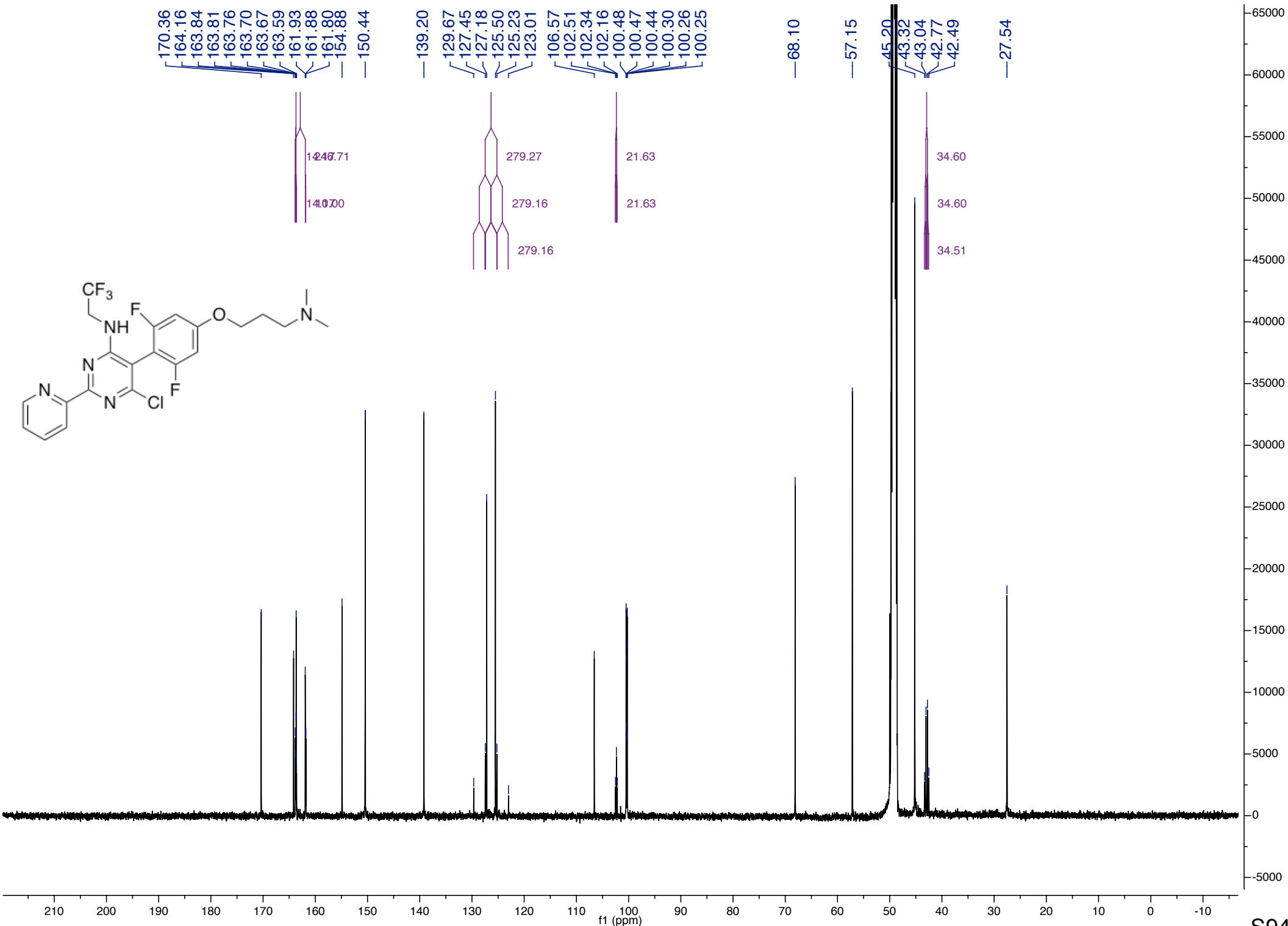
27.54

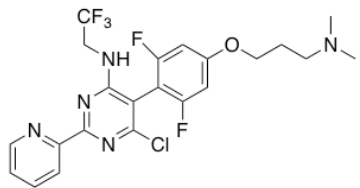
146.71  
140.70

279.27  
279.16  
279.16

21.63  
21.63

34.60  
34.60  
34.51





164.16

163.84  
163.81  
163.76  
163.70  
163.67  
163.59

161.93  
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161.80

14.17

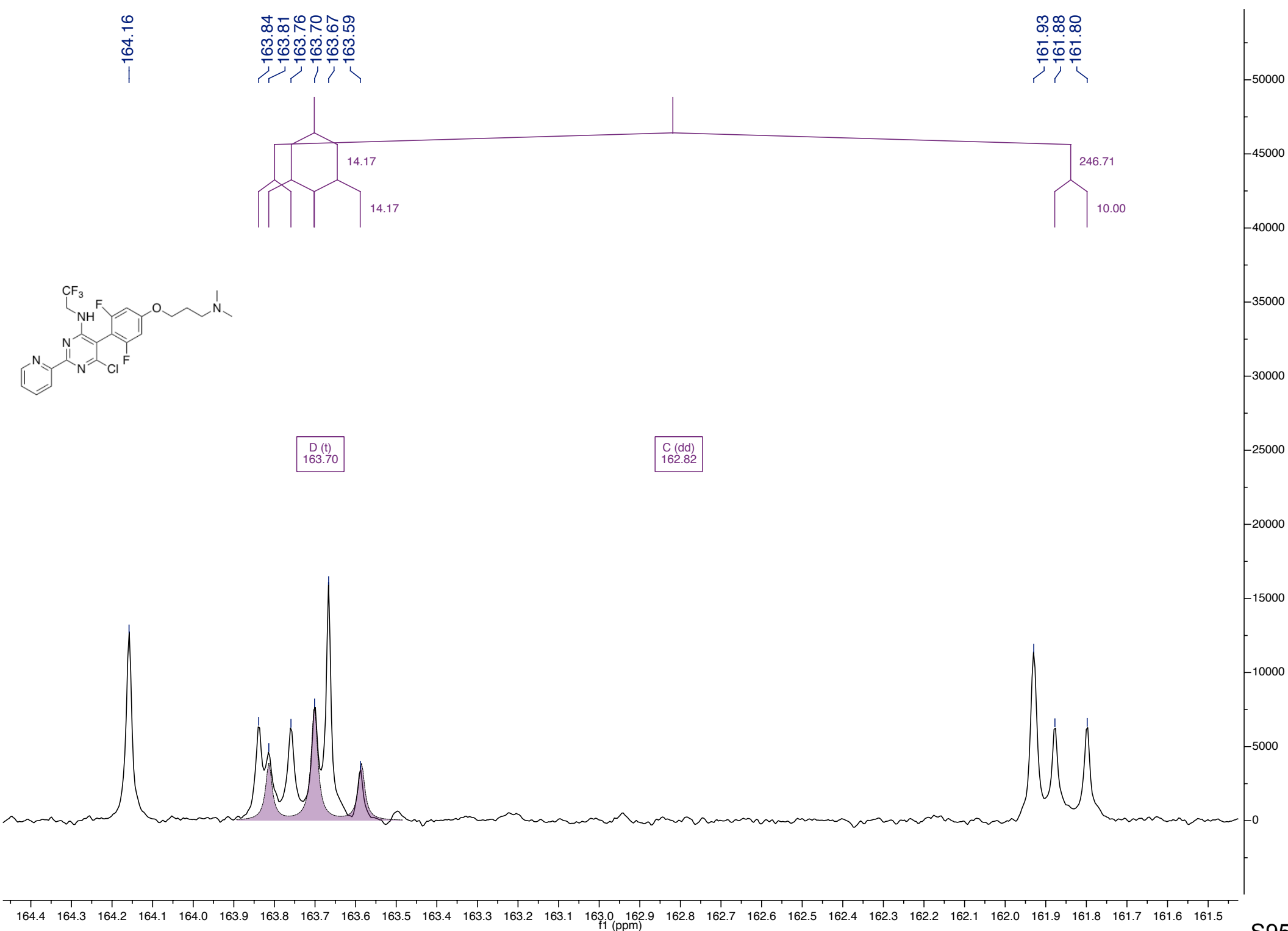
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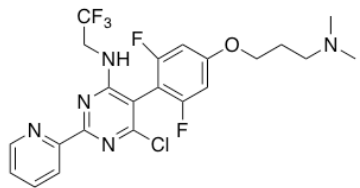
246.71

10.00

D (t)  
163.70

C (dd)  
162.82





164.16

163.84  
163.81  
163.76  
163.70  
163.67  
163.59

161.93  
161.88  
161.80

14.17

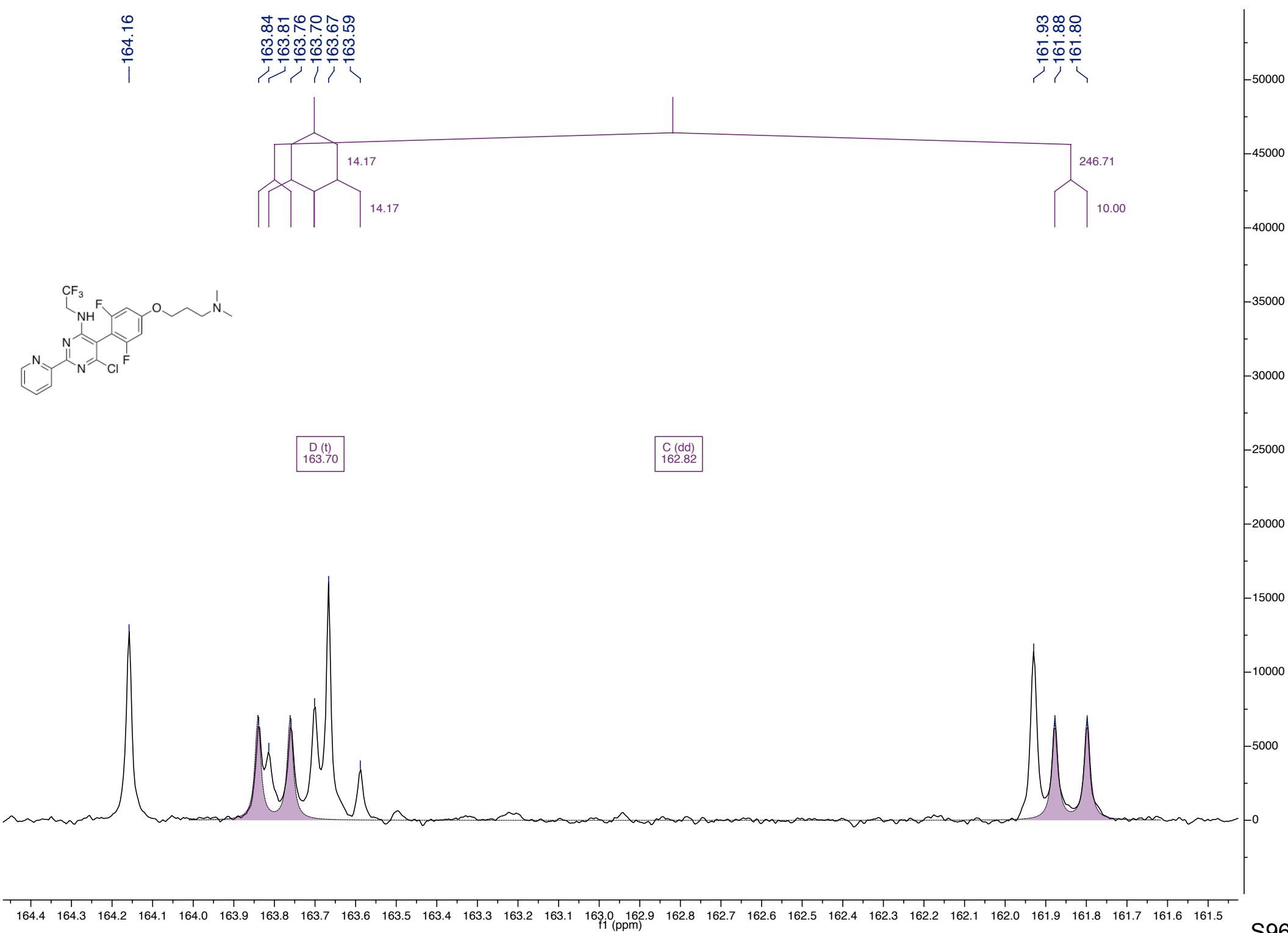
14.17

246.71

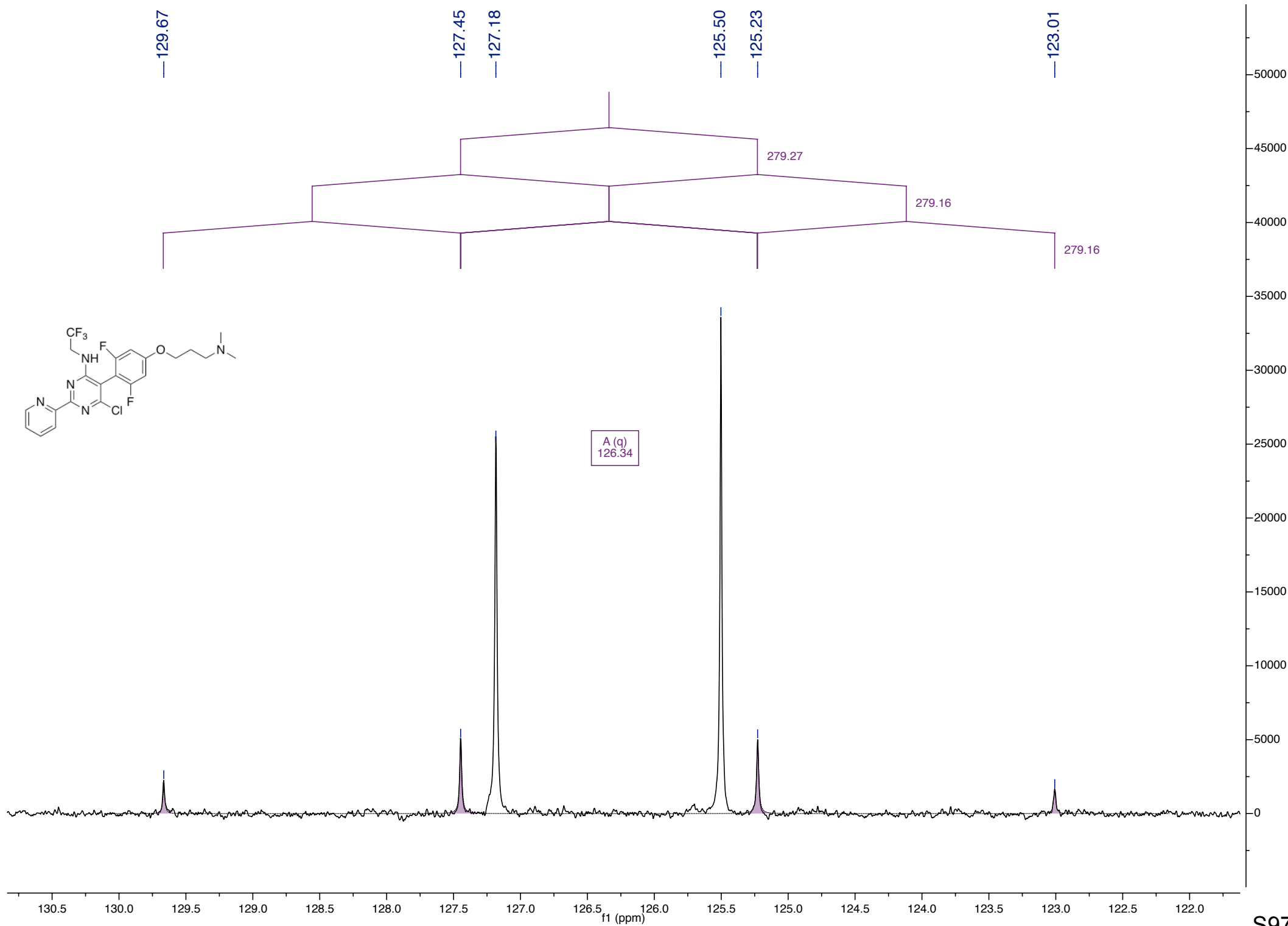
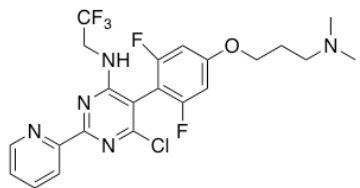
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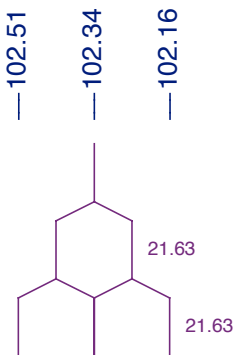
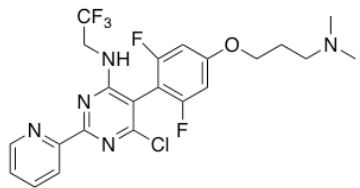
D (t)  
163.70

C (dd)  
162.82

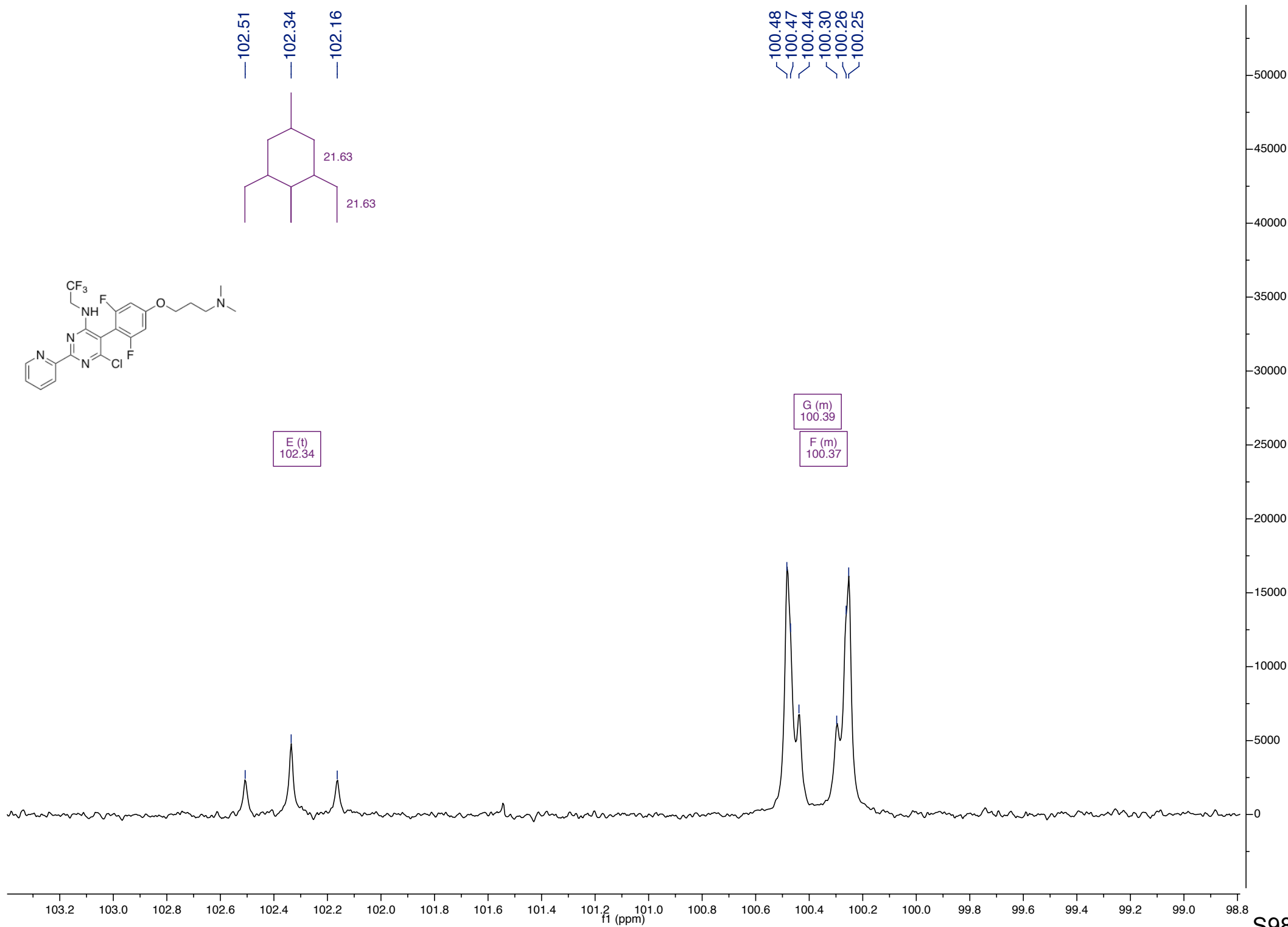


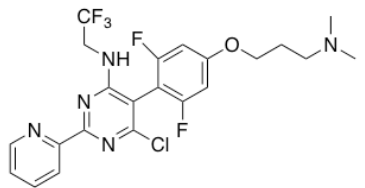






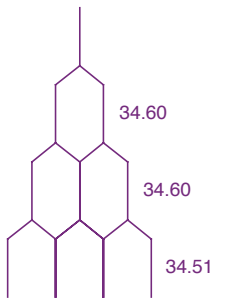
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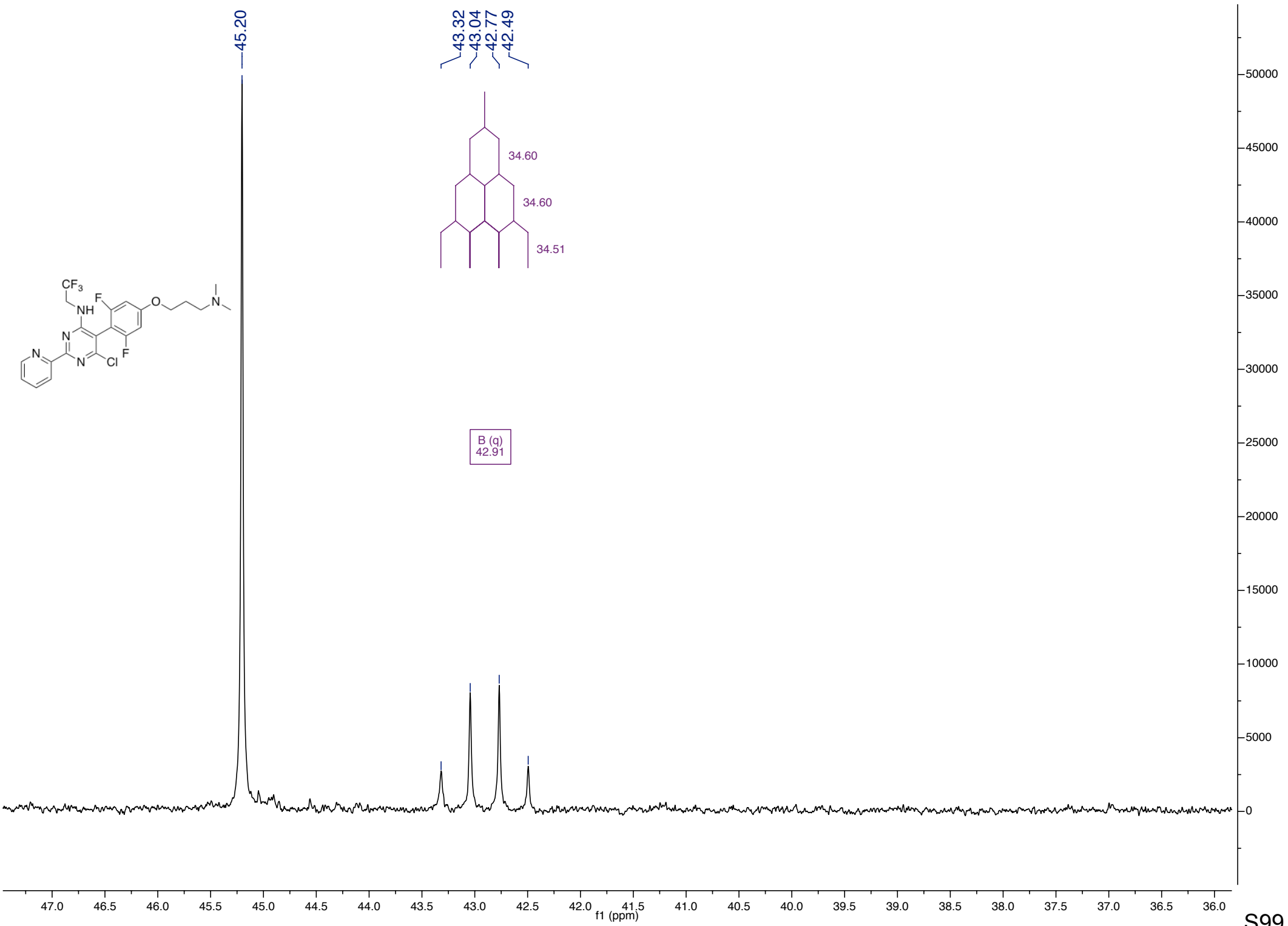


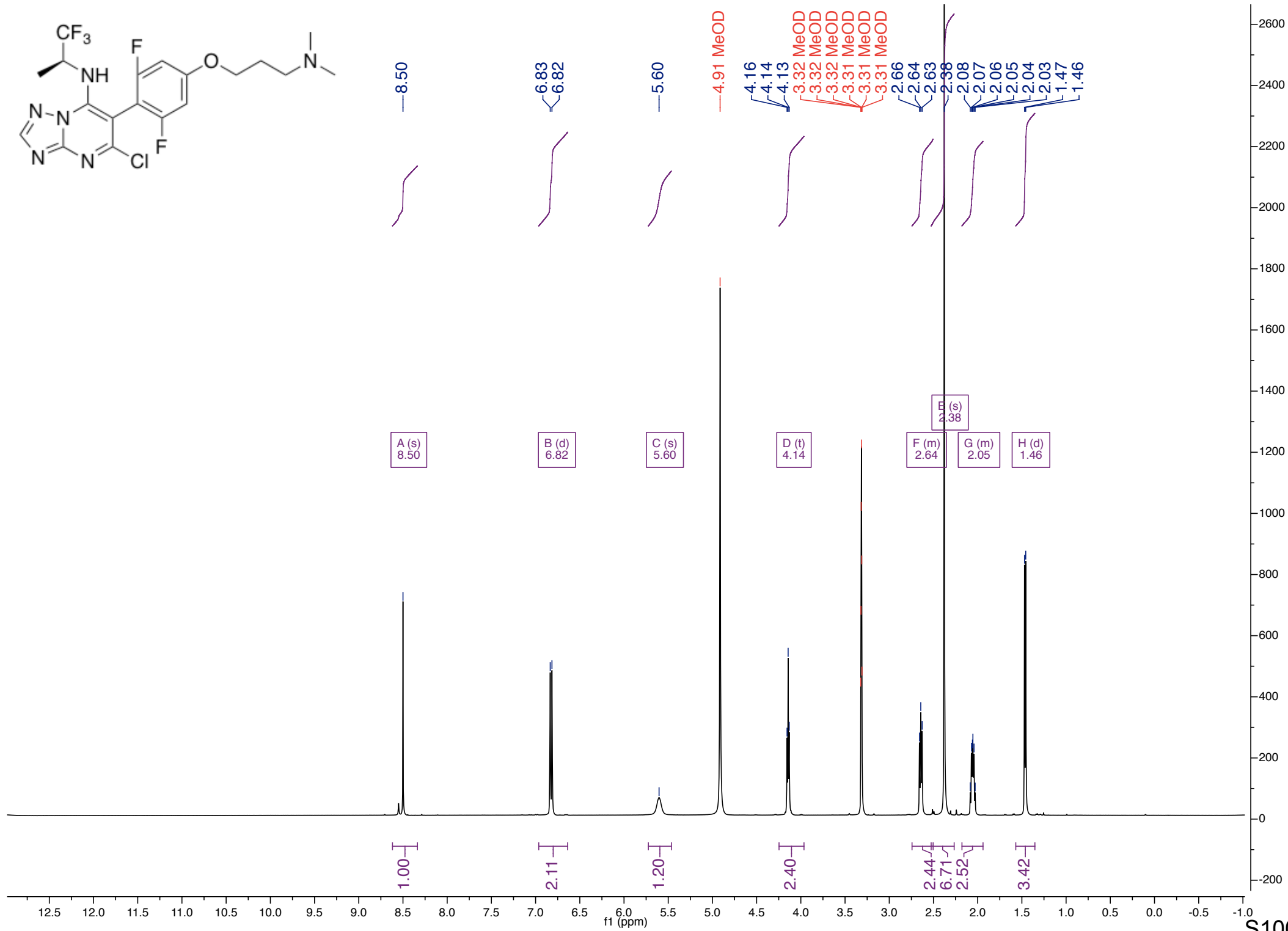
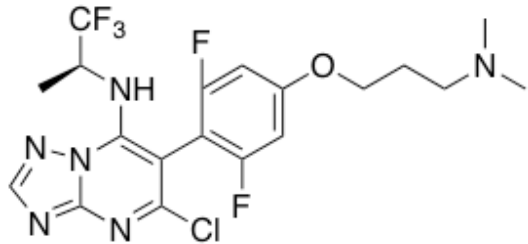
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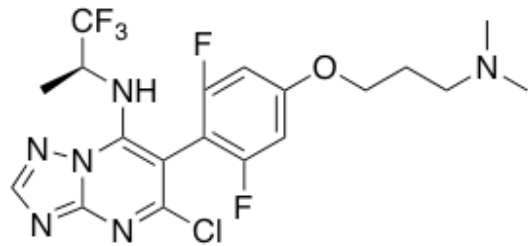
43.32  
43.04  
42.77  
42.49



B (q)  
42.91







170.42 formic acid  
 164.68  
 164.60  
 164.43  
 164.32  
 164.21  
 164.02  
 163.95  
 162.71  
 162.64  
 162.06  
 161.99  
 159.57  
 156.12  
 155.87  
 149.34

130.07  
 127.83  
 125.59  
 123.35  
 102.44  
 102.28  
 102.10  
 100.64  
 100.61  
 100.43  
 100.41  
 100.29  
 100.27  
 100.09  
 100.06  
 94.91

68.32

57.18  
 52.75  
 52.50  
 52.25  
 52.00  
 45.40

27.77

14.05

B (t)  
 164.32  
 A (dd)  
 163.66  
 C (dd)  
 163.01

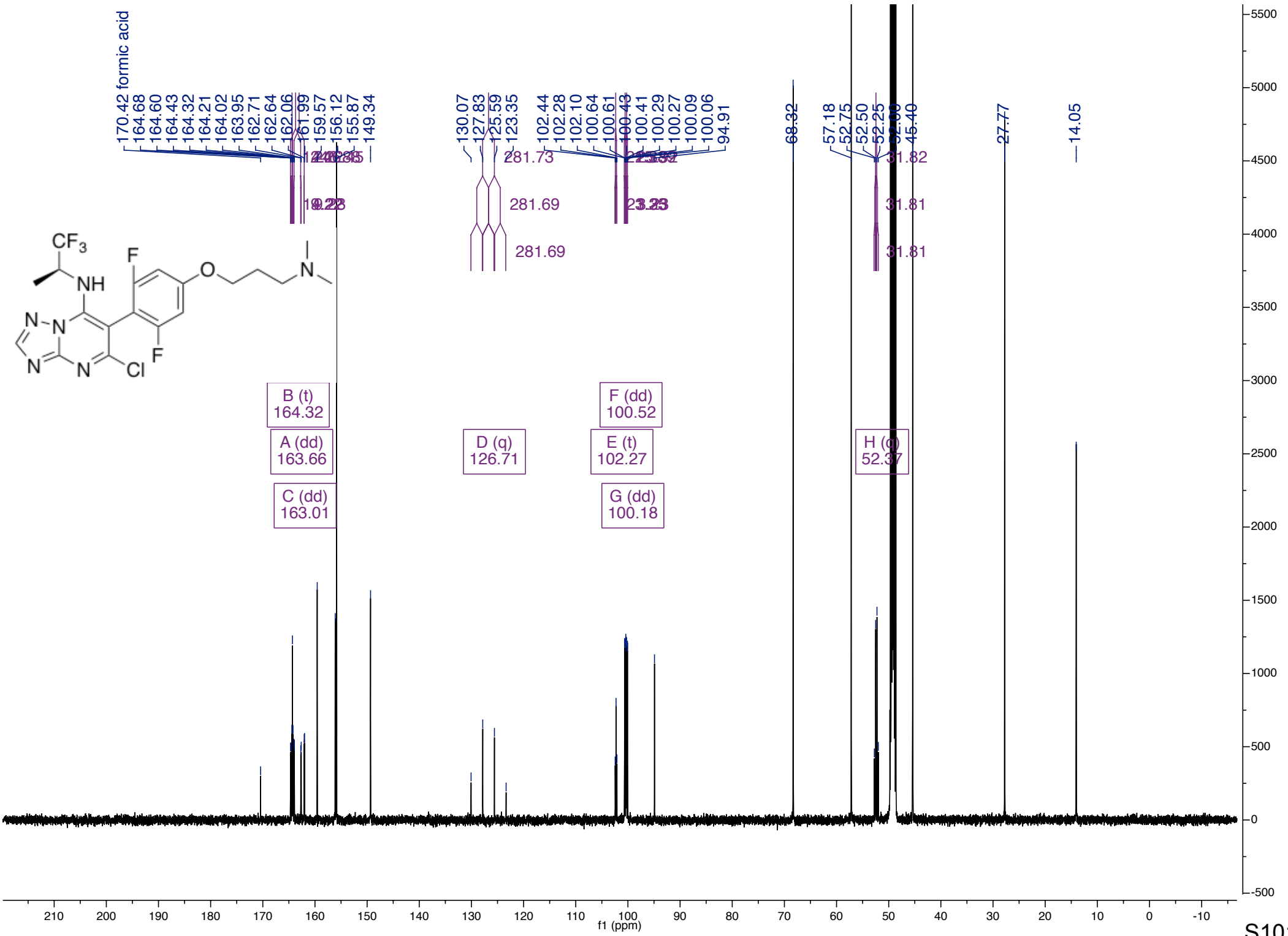
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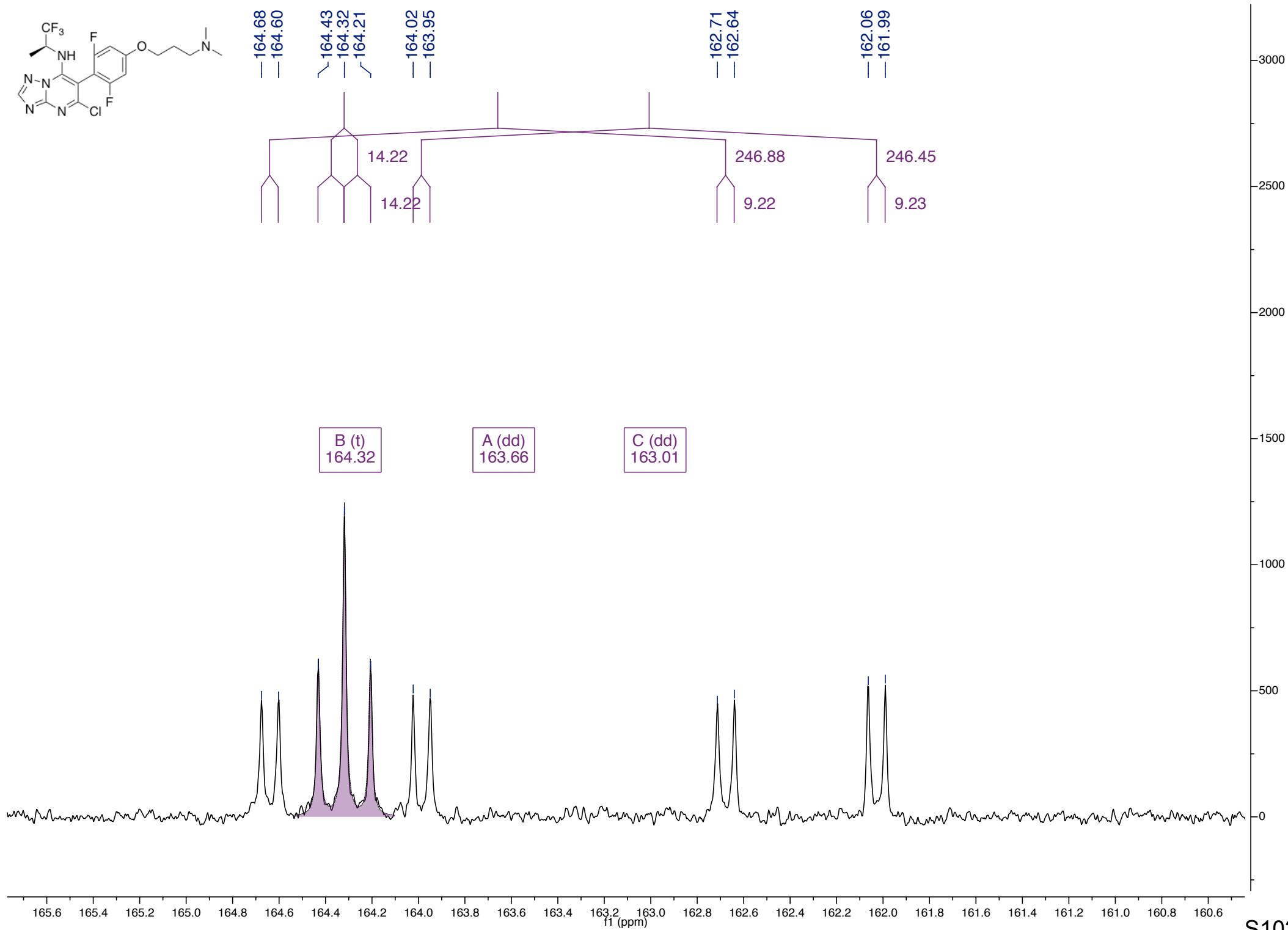
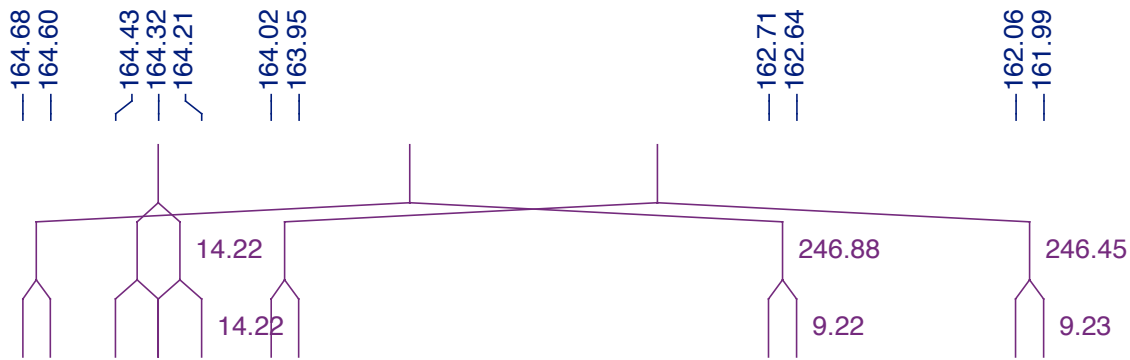
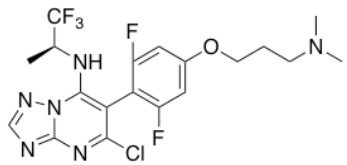
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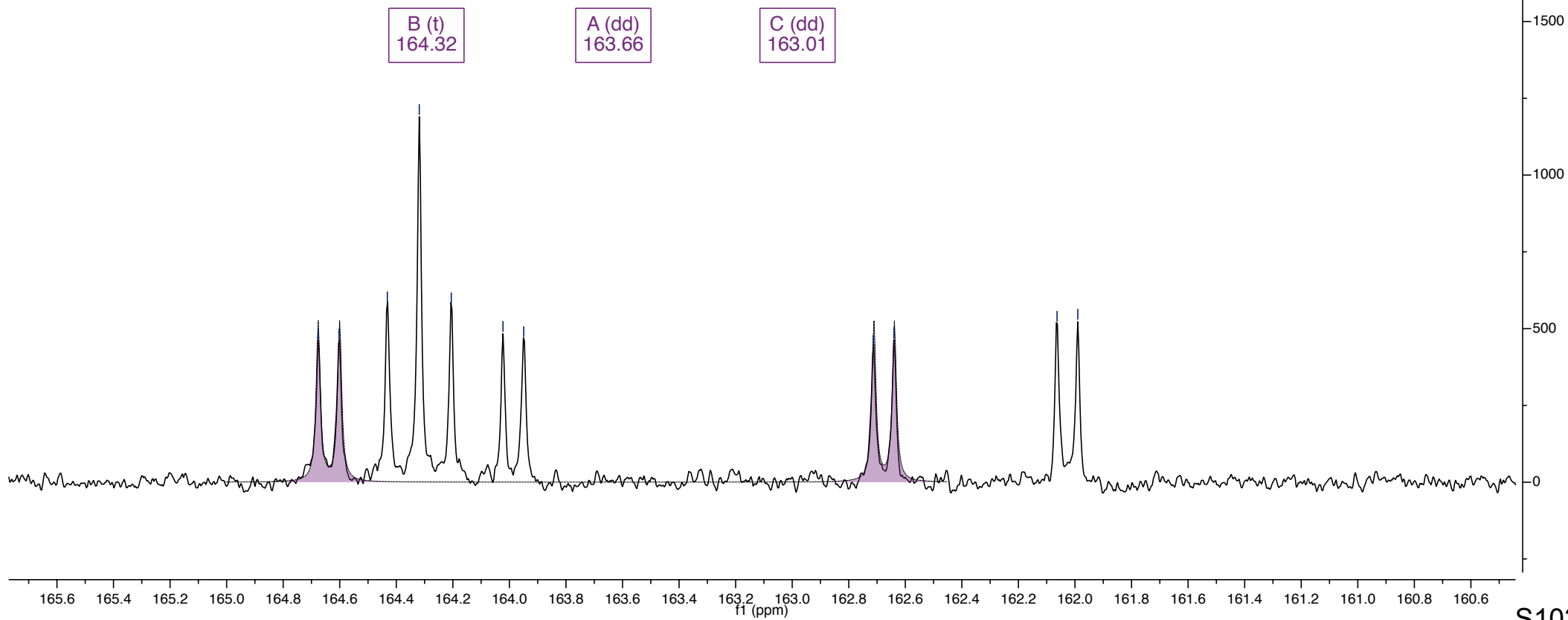
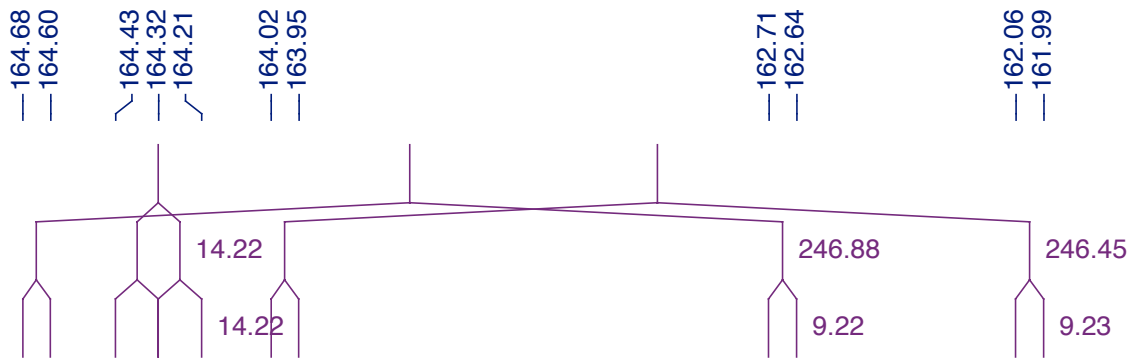
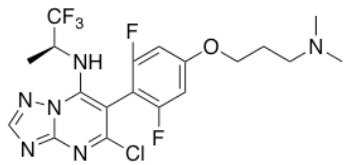
G (dd)  
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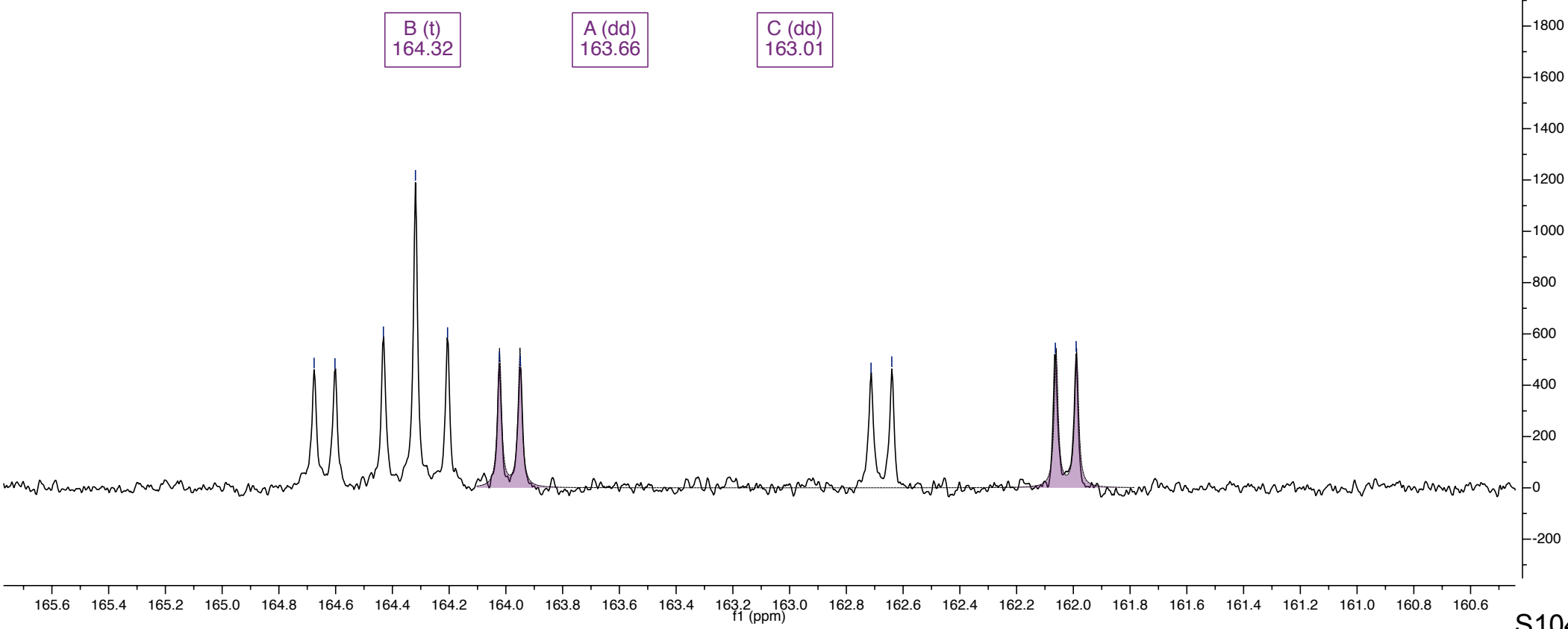
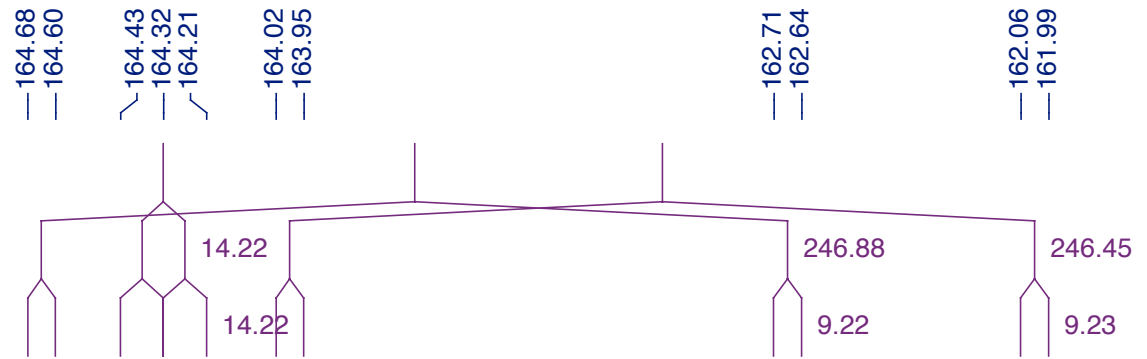
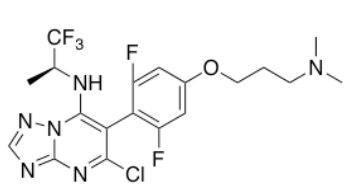
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H (q)  
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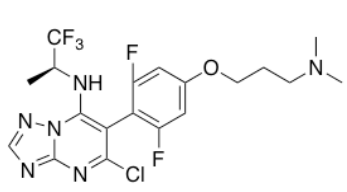












— 130.07

— 127.83

— 125.59

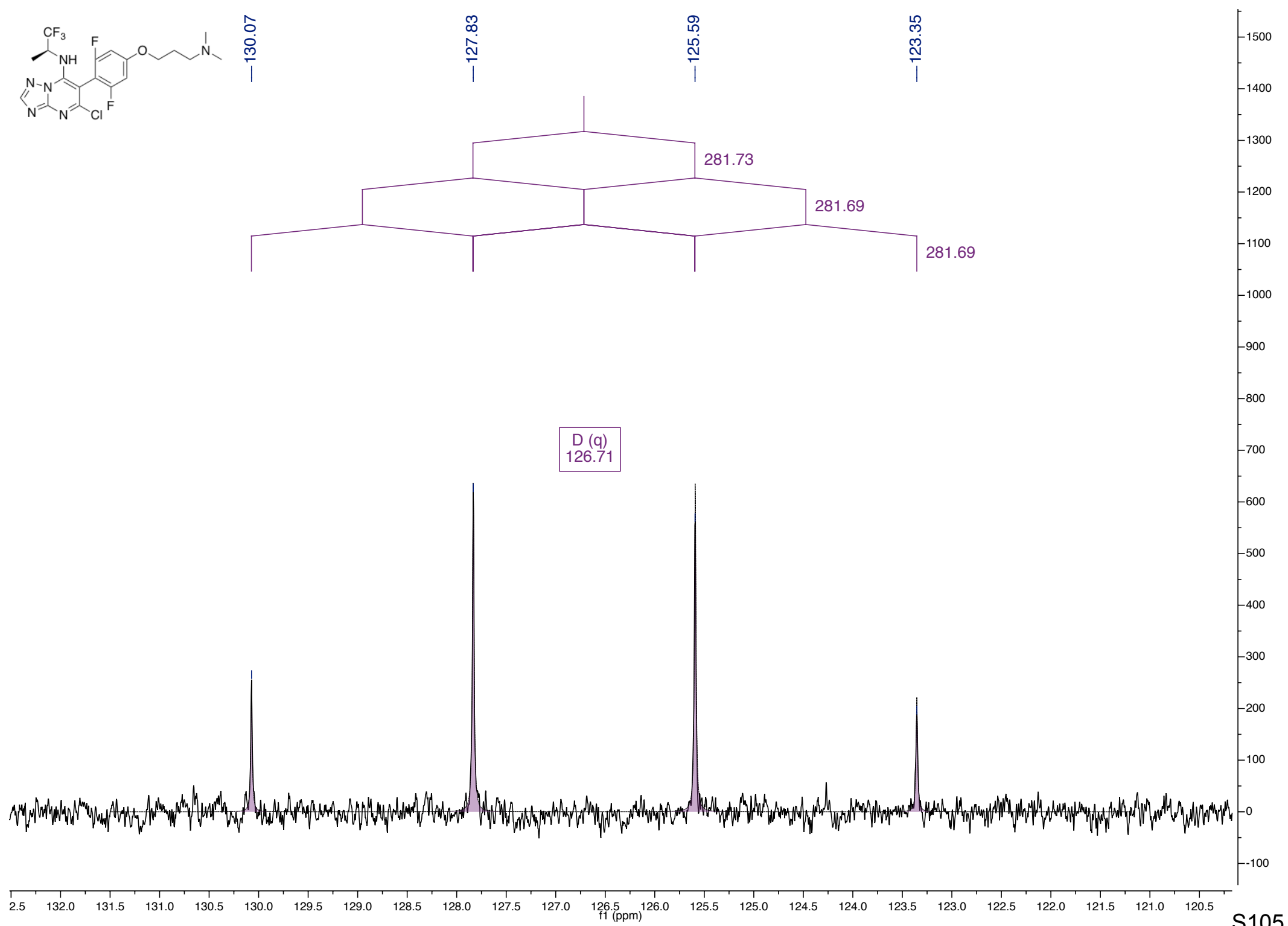
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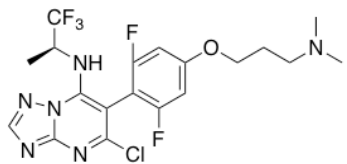
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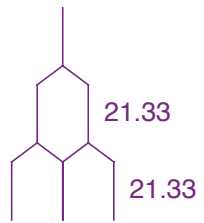
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D (q)  
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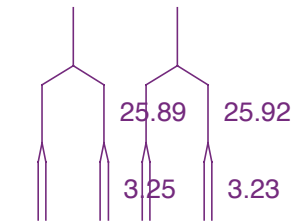




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102.10



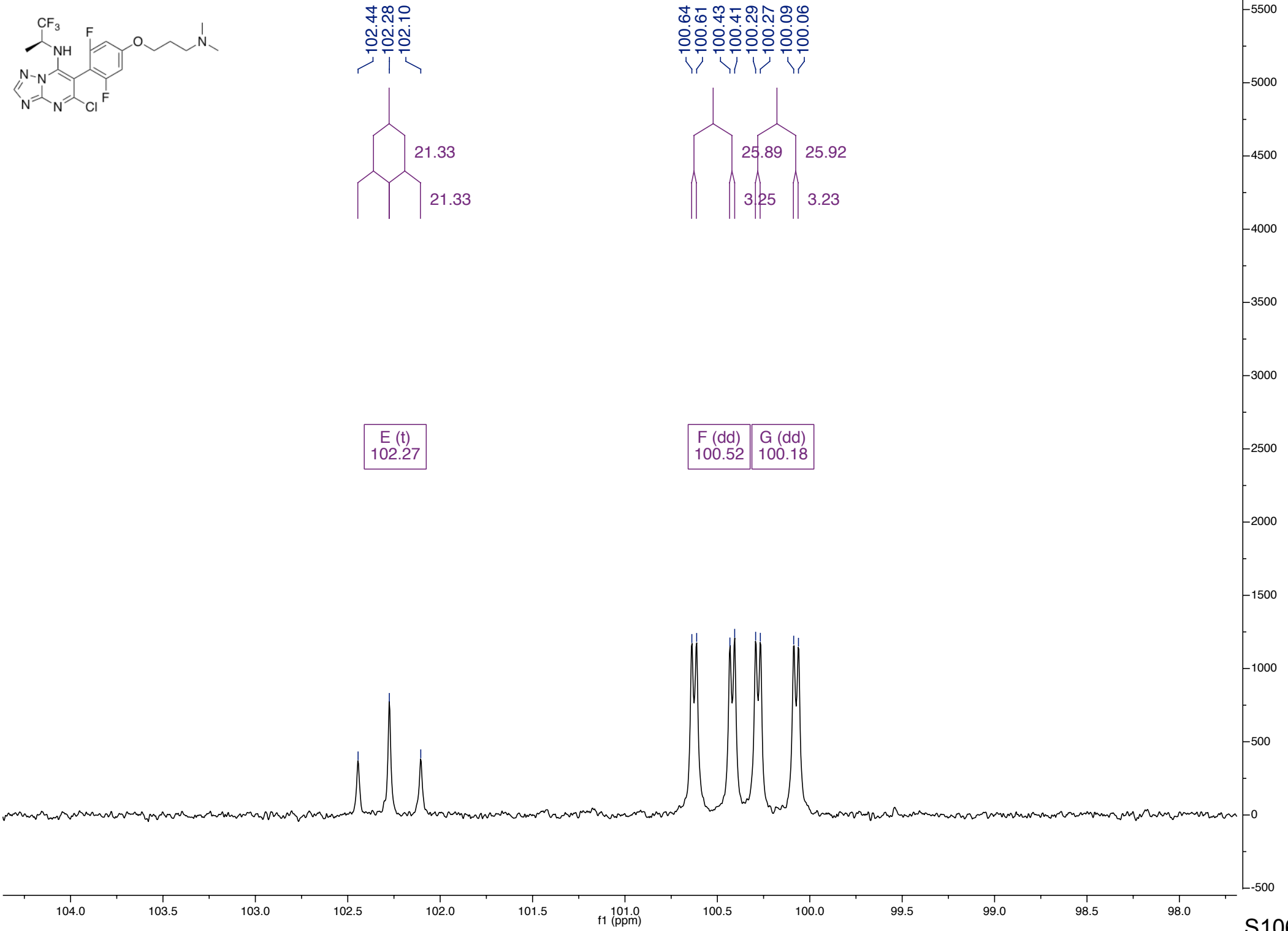
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100.06

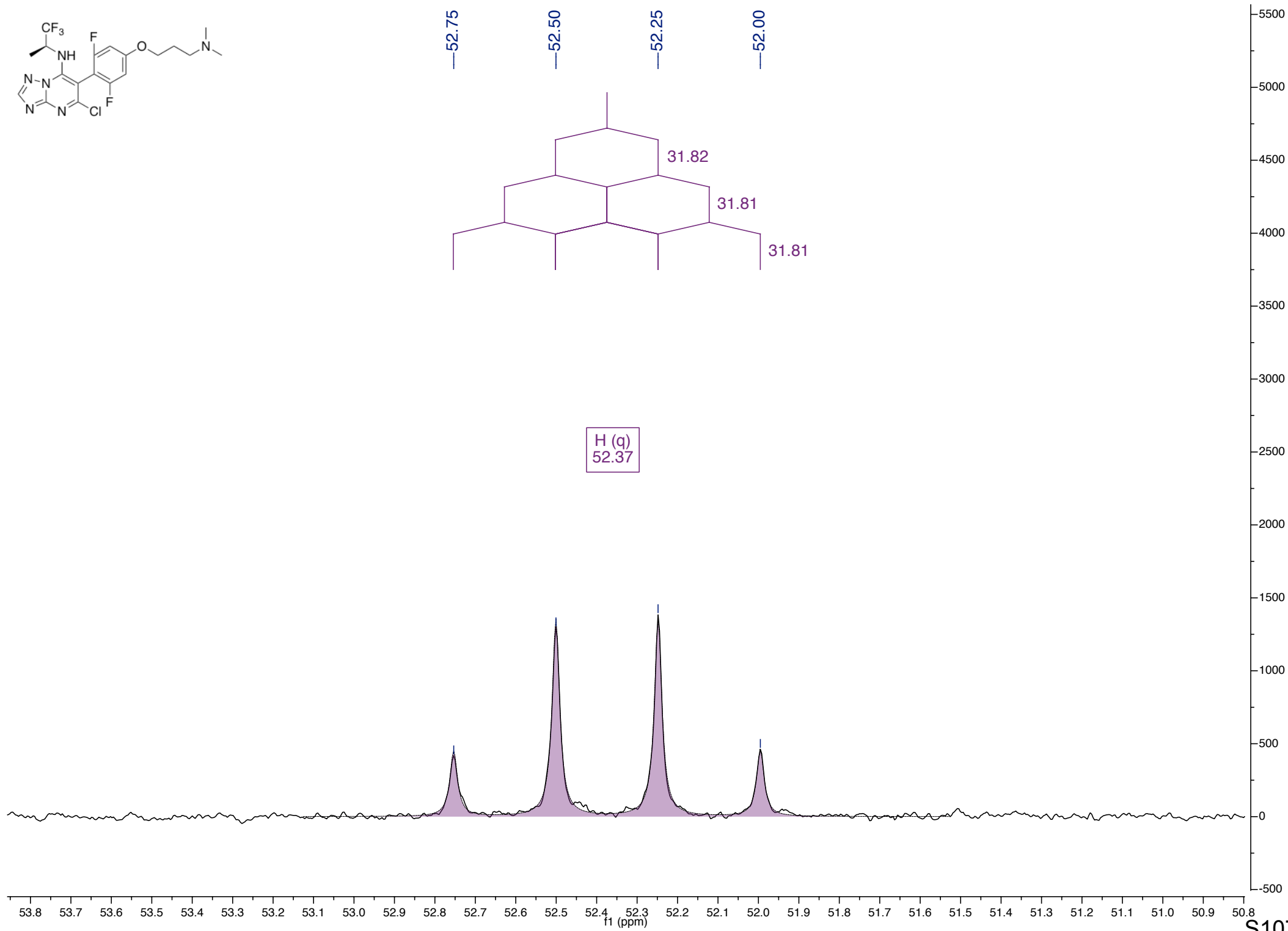
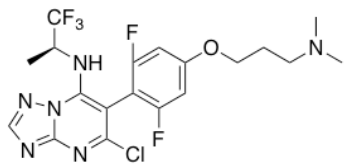


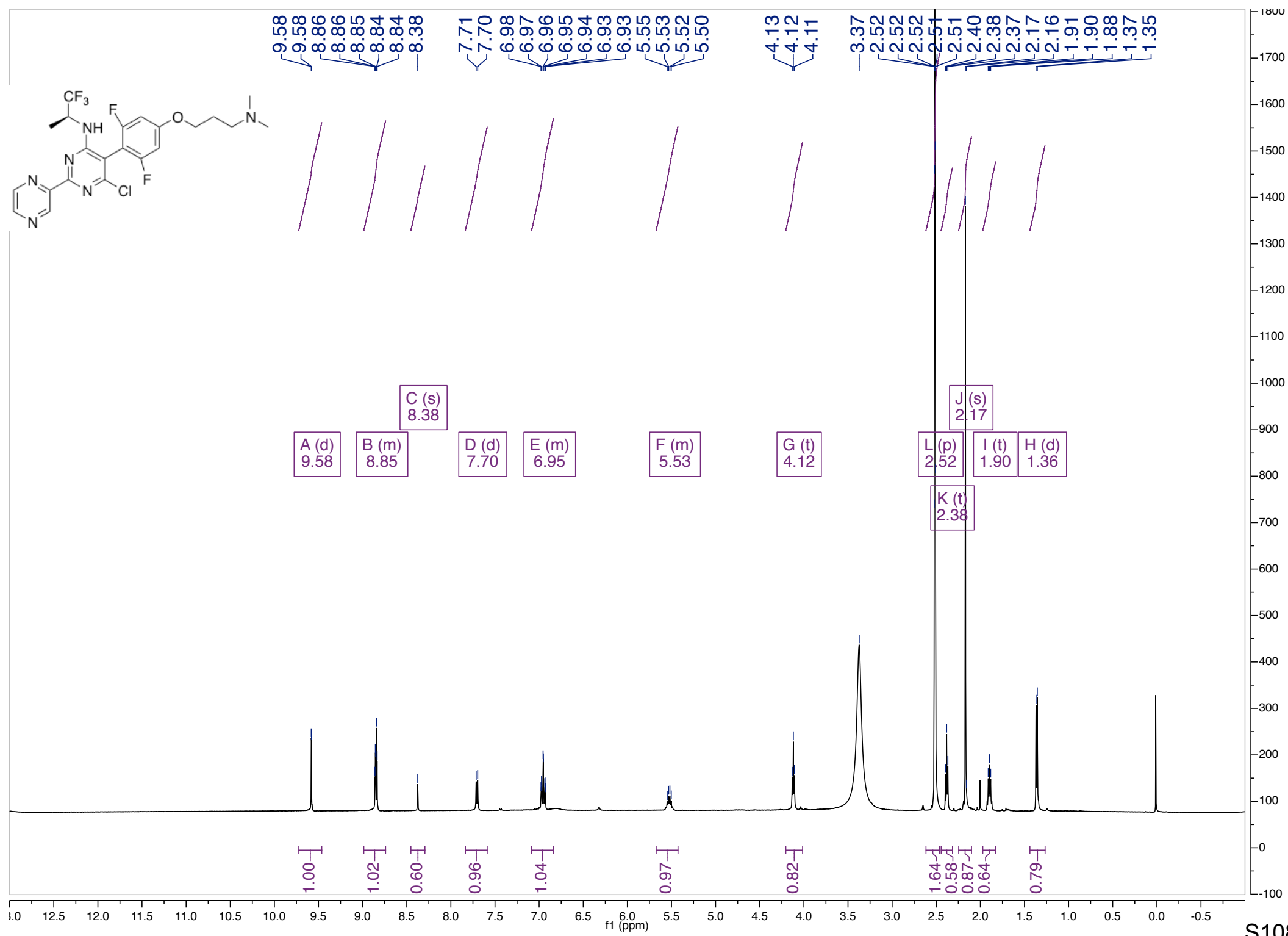
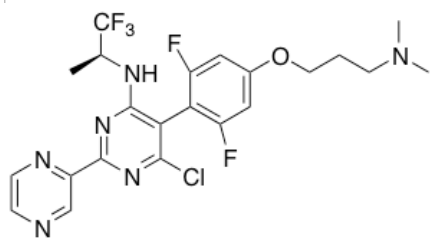
E (t)  
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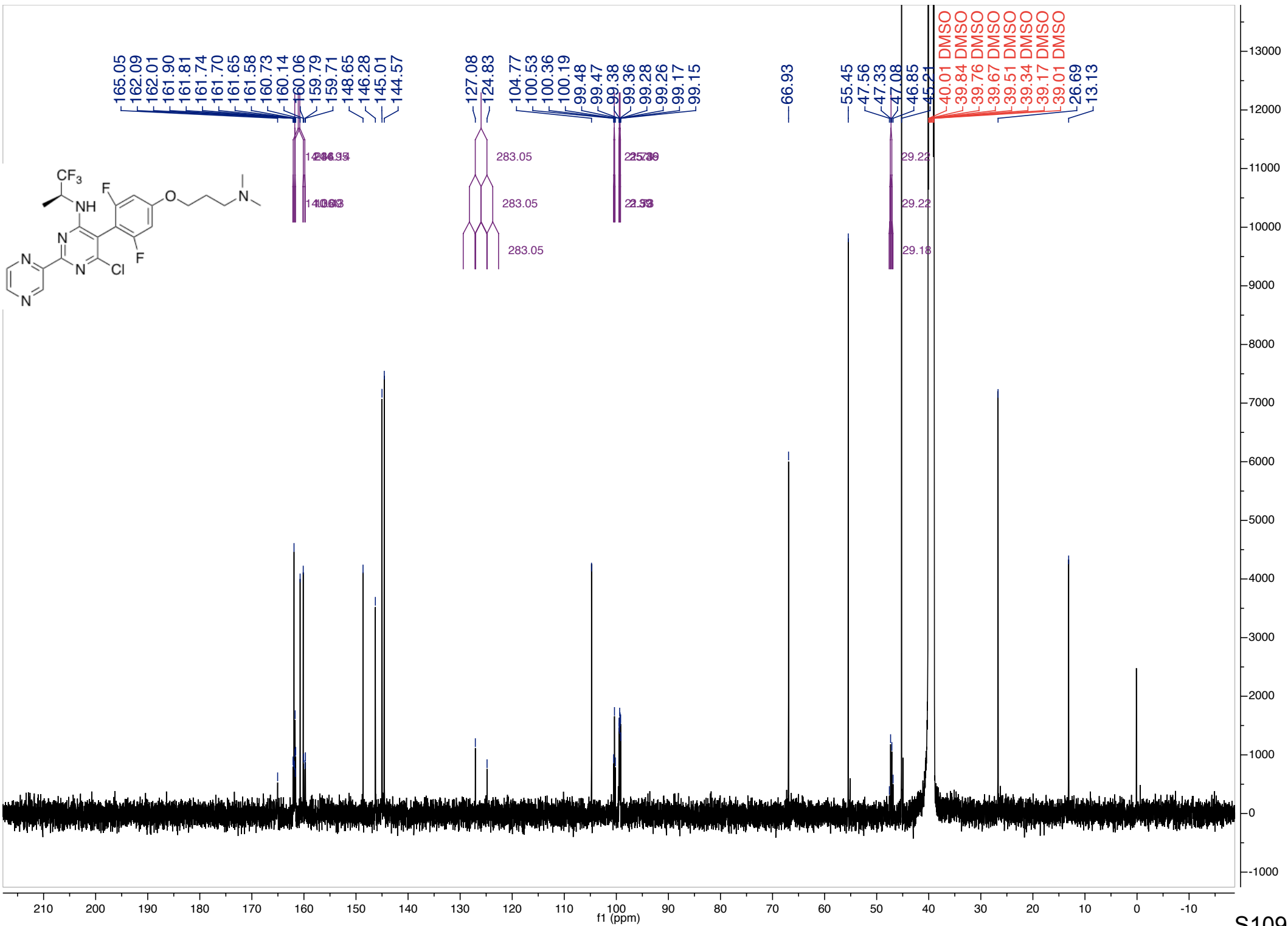
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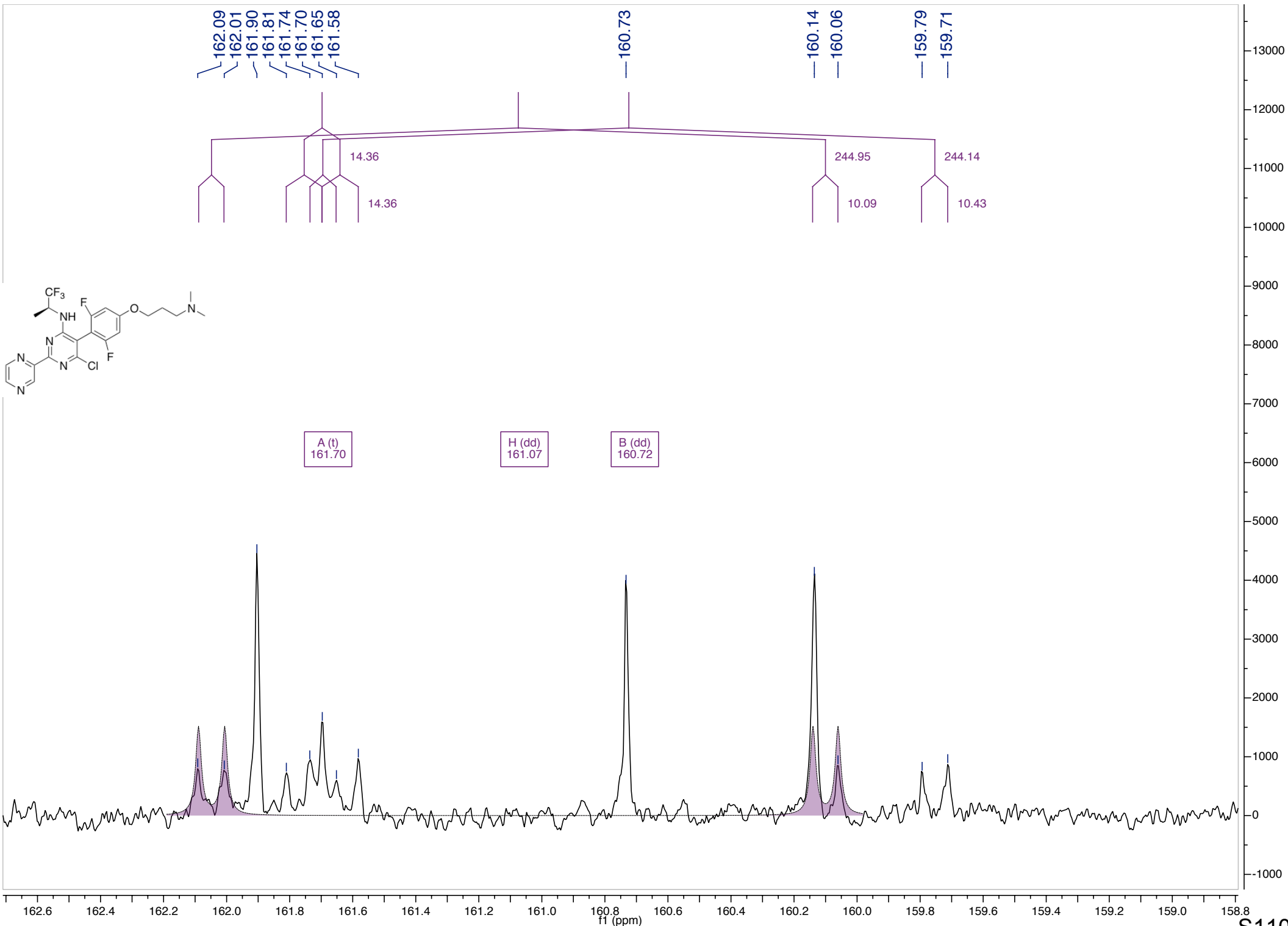
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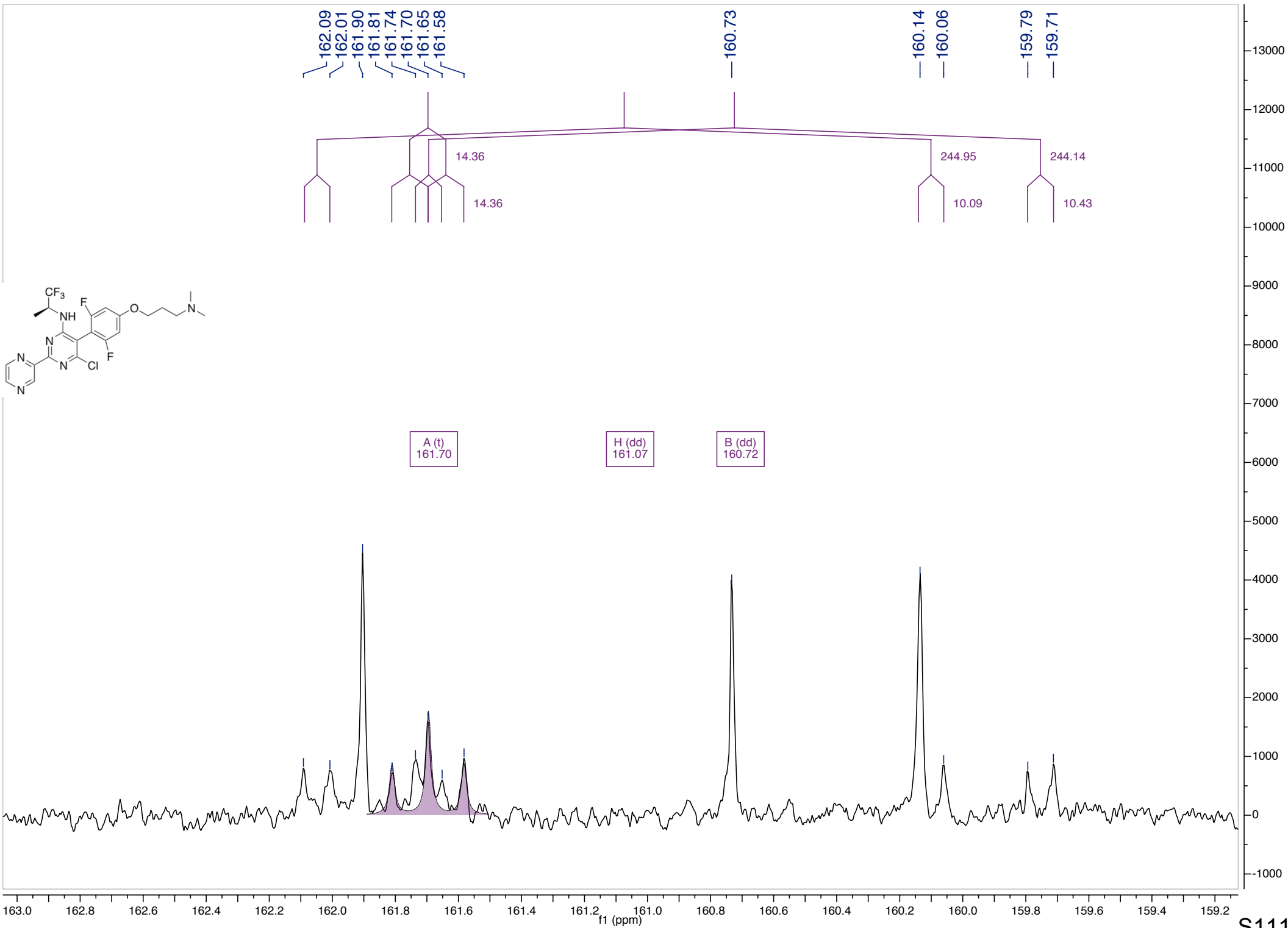


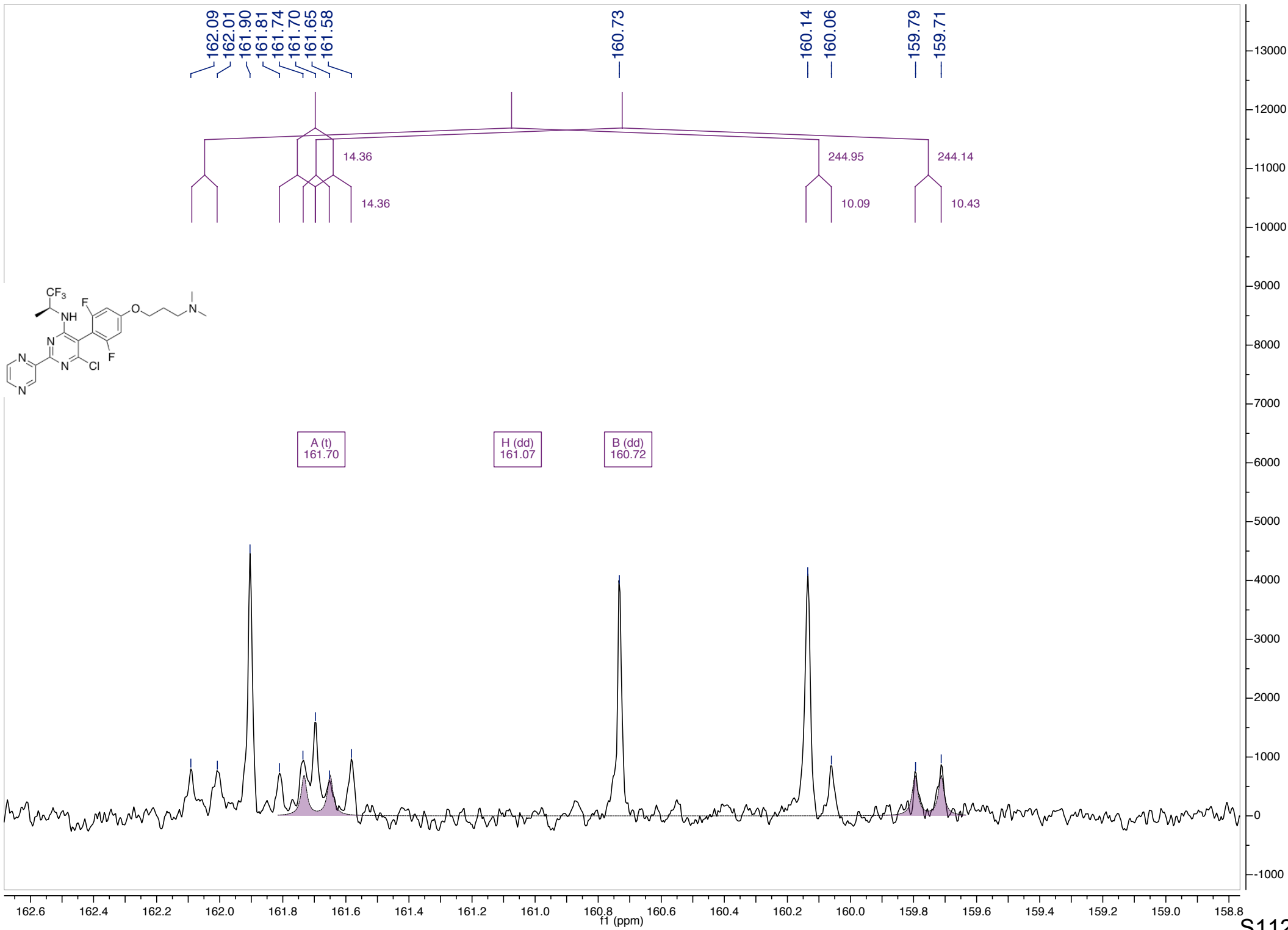




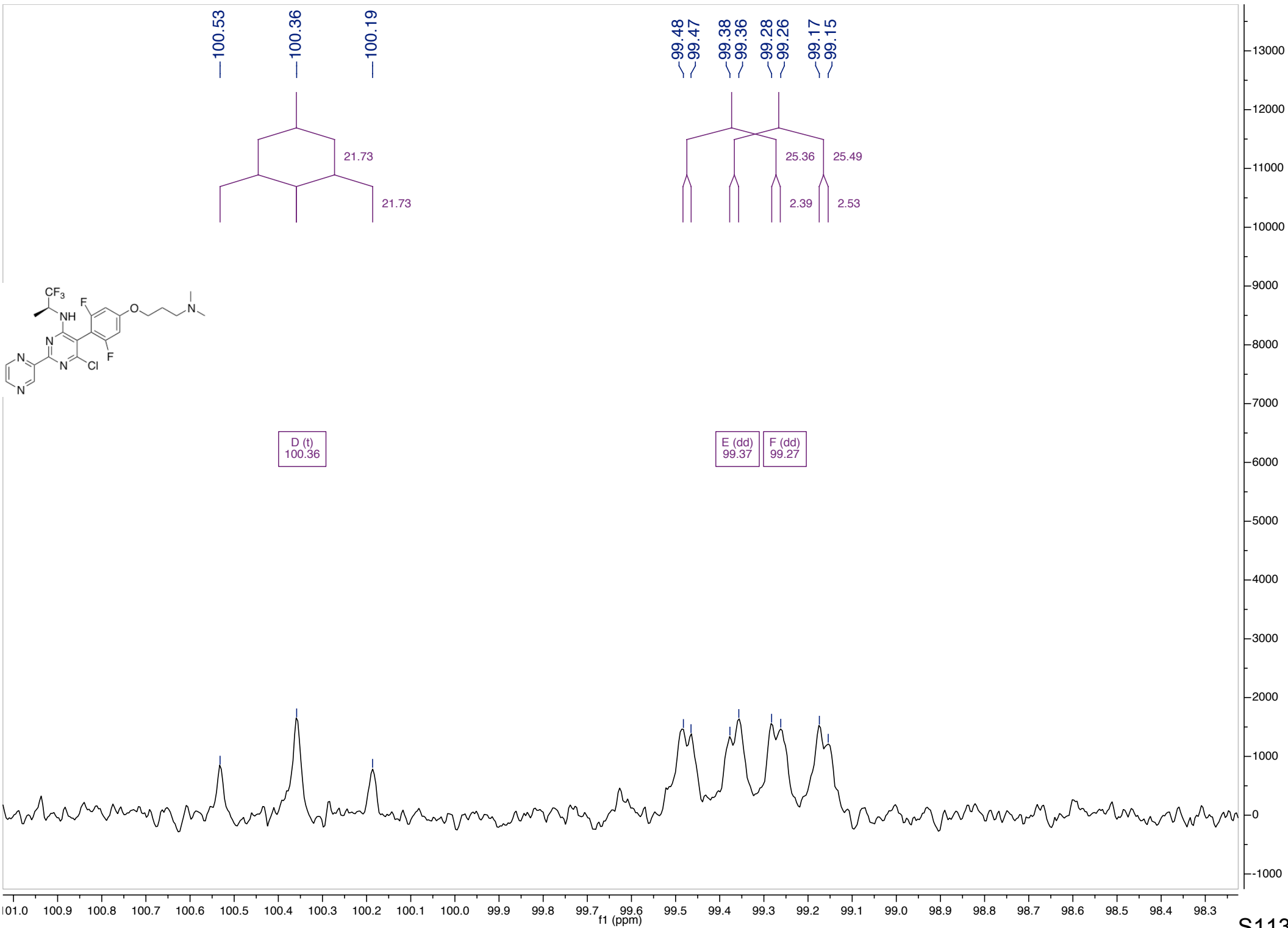


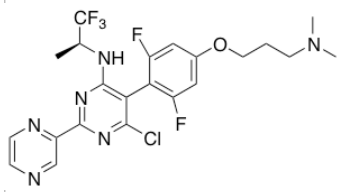
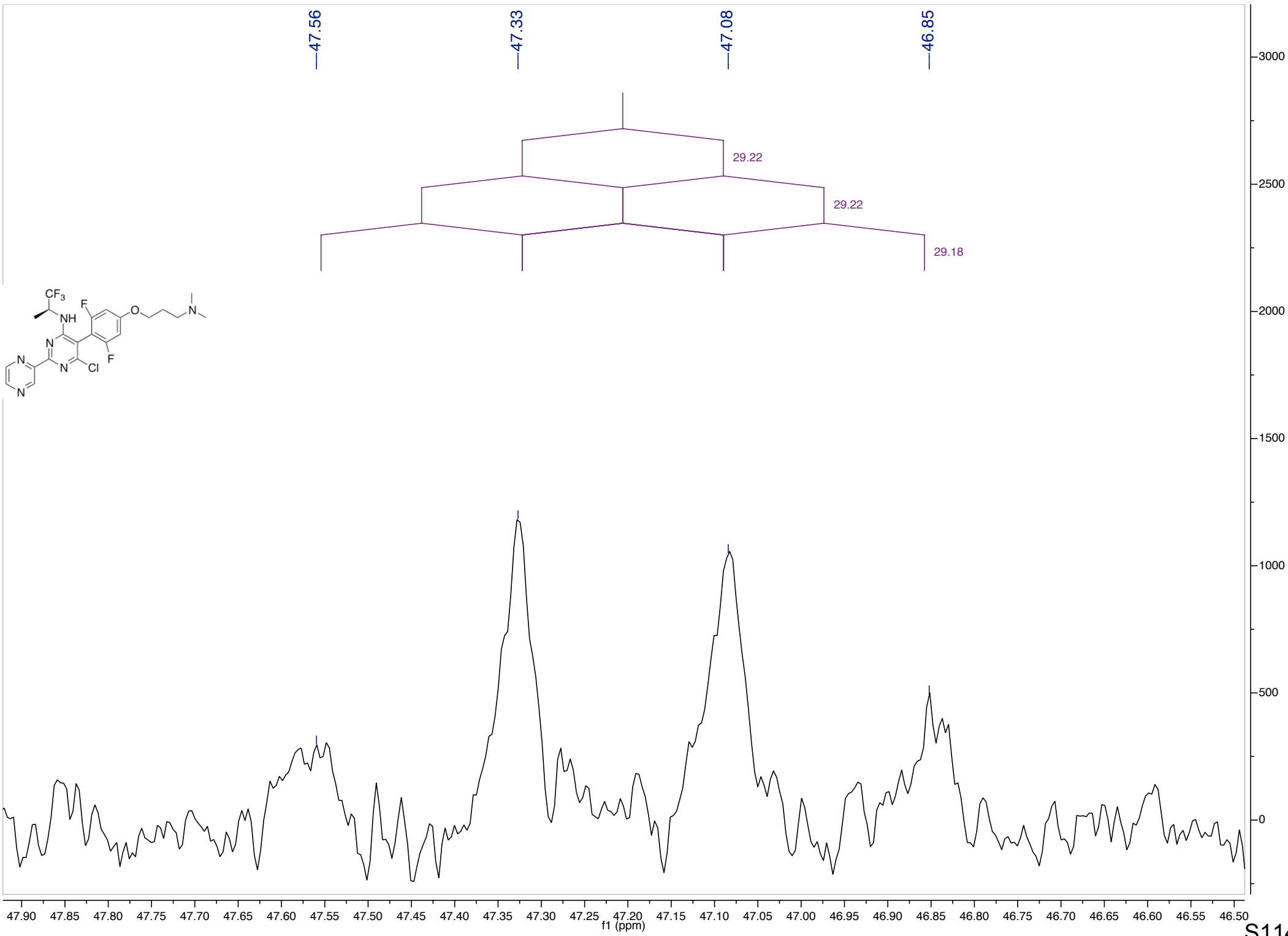


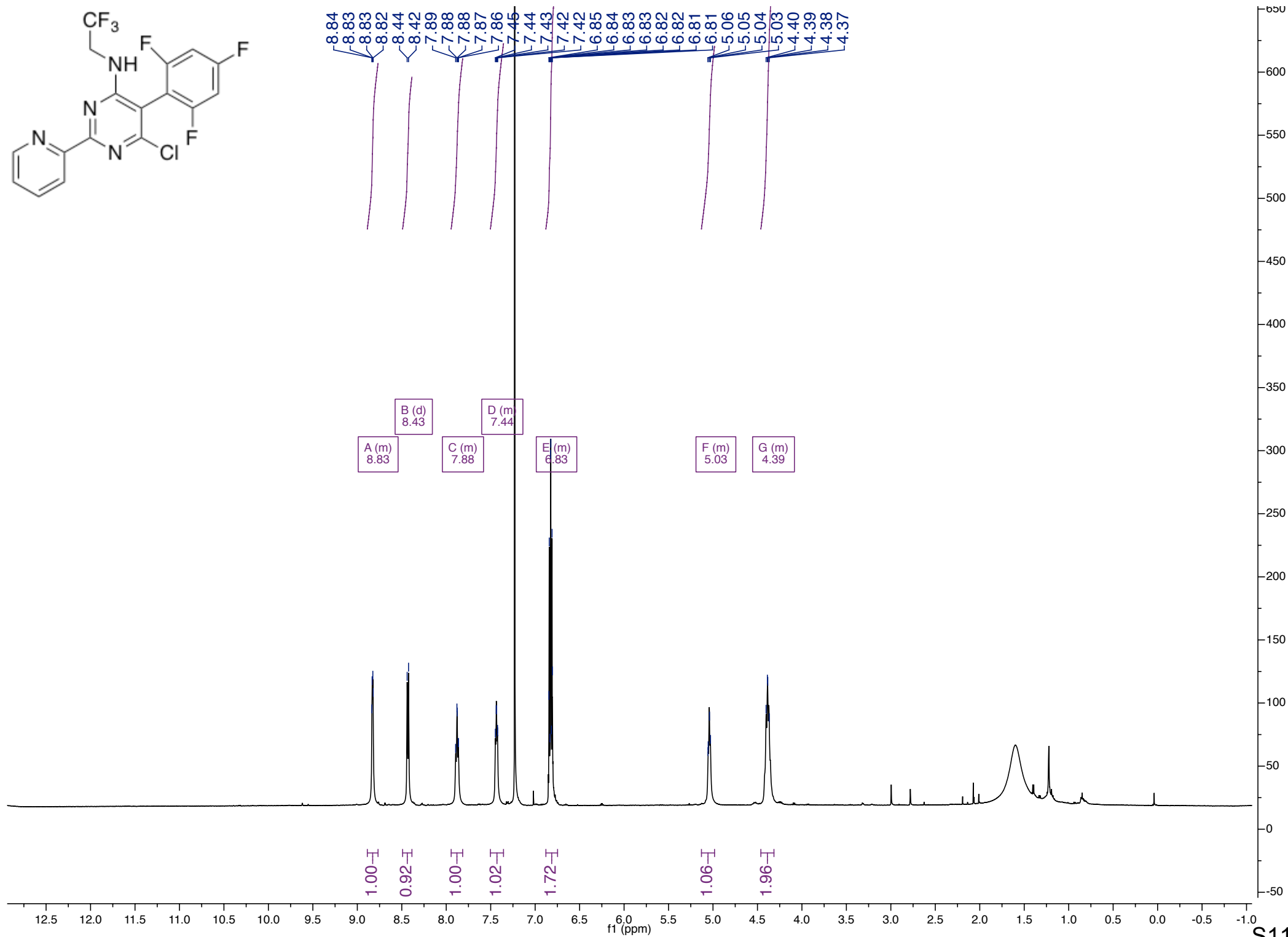
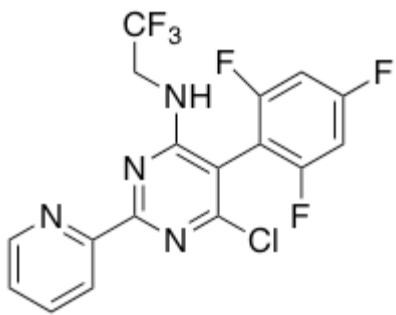


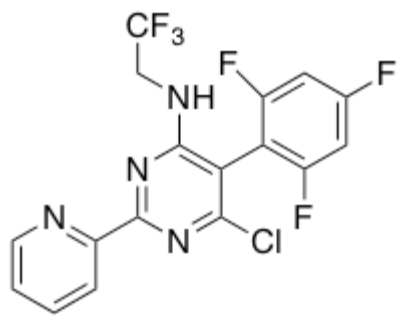












165.44  
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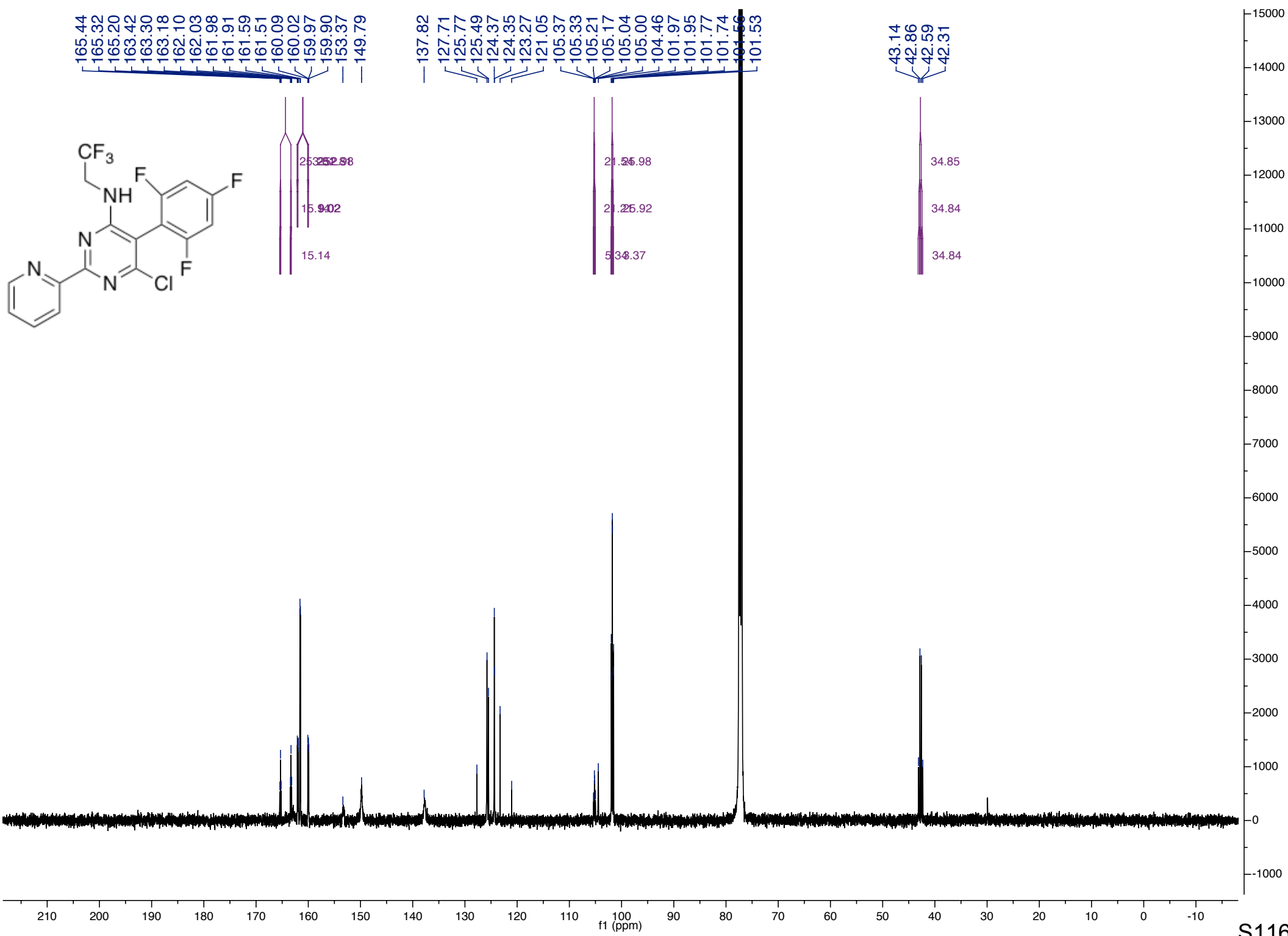
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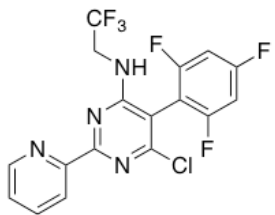
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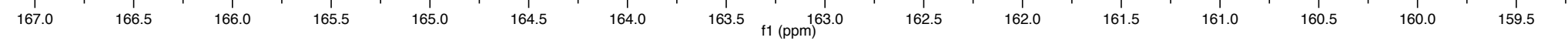
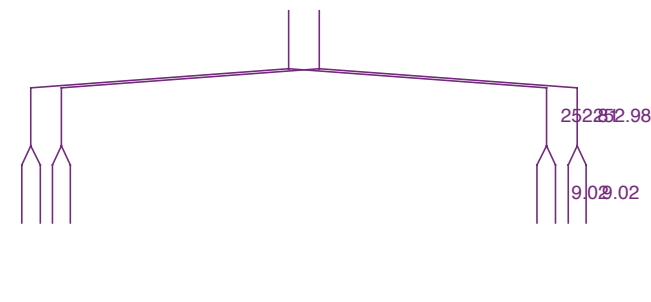
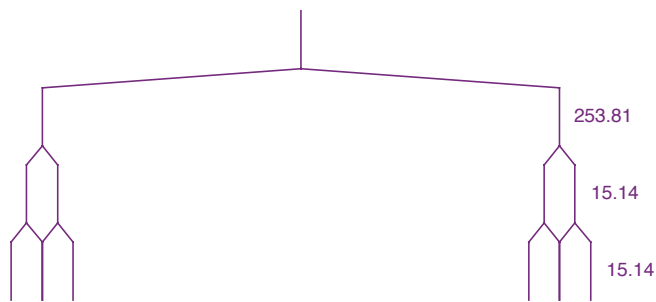
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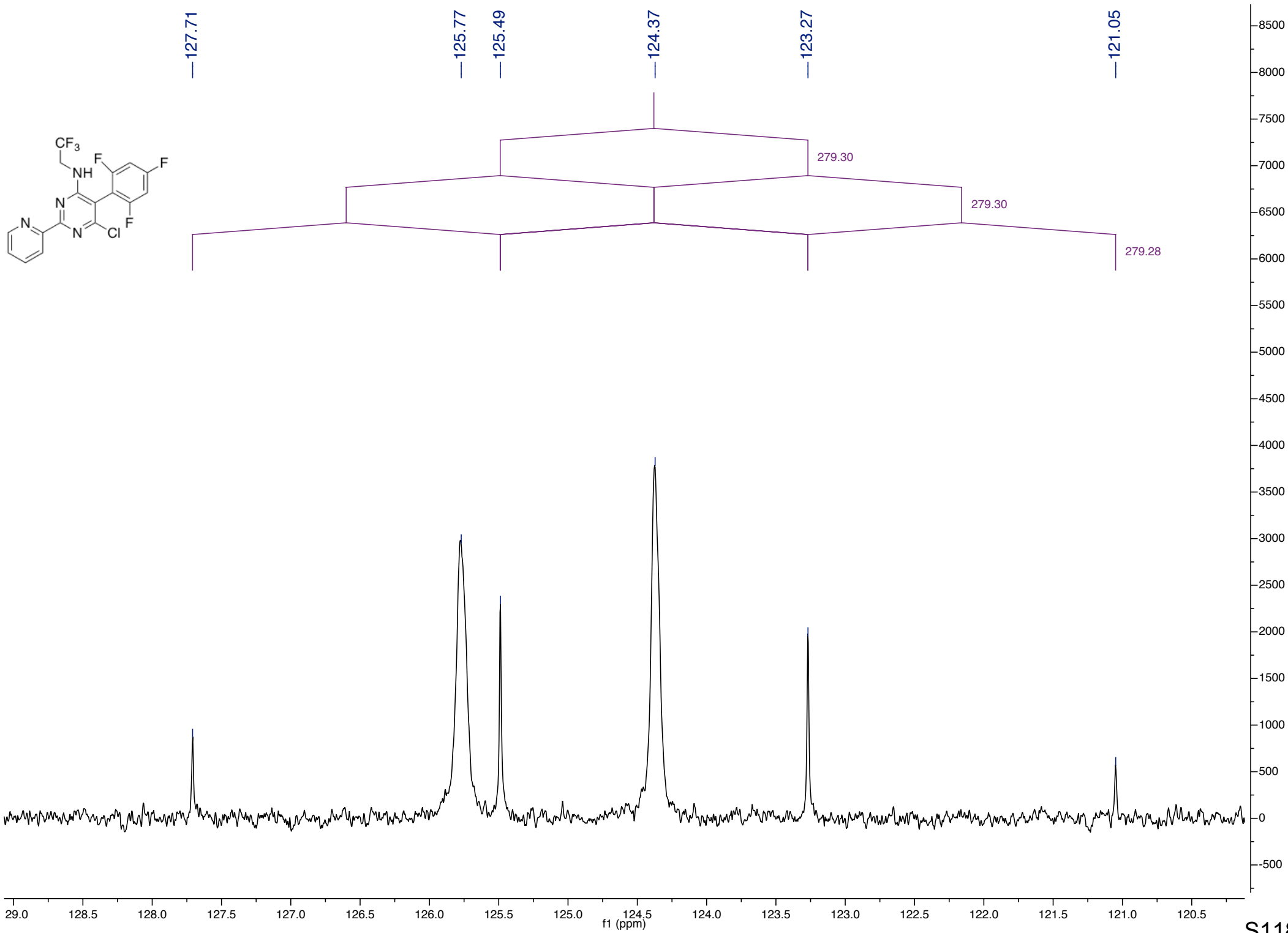
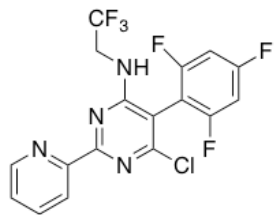
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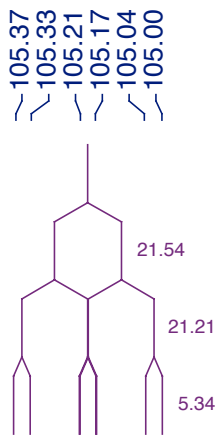
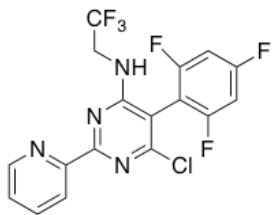
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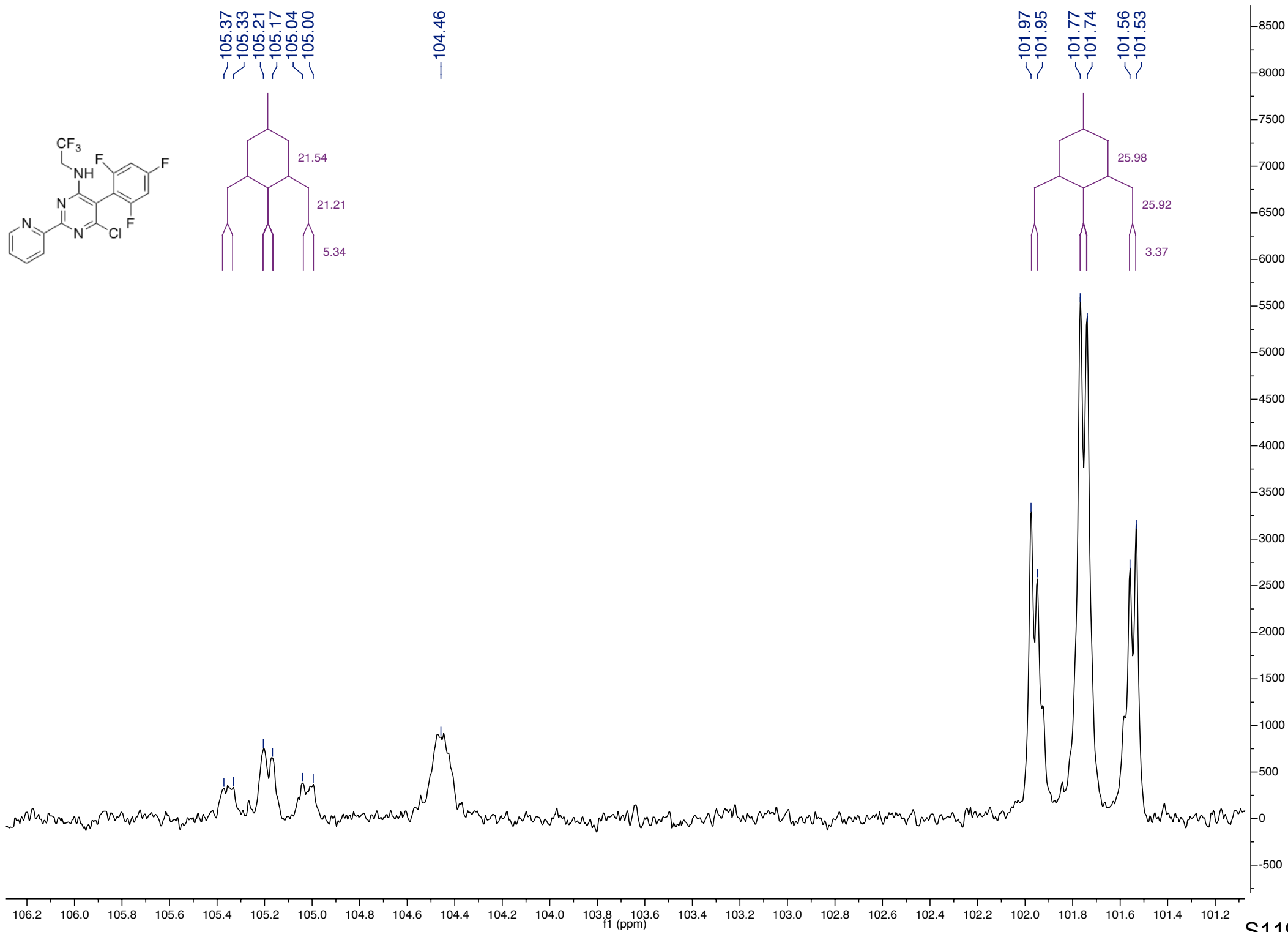
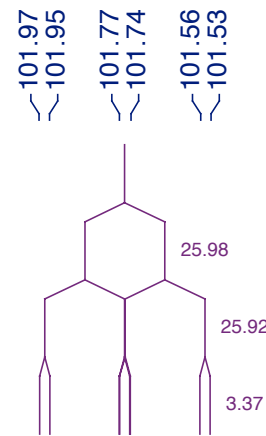
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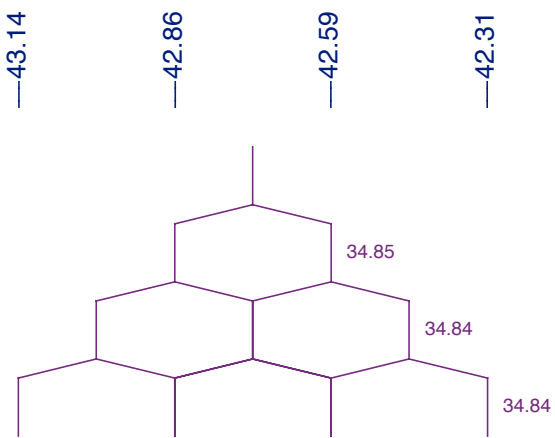
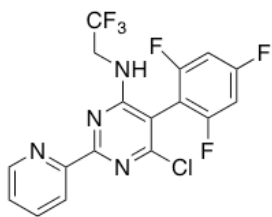






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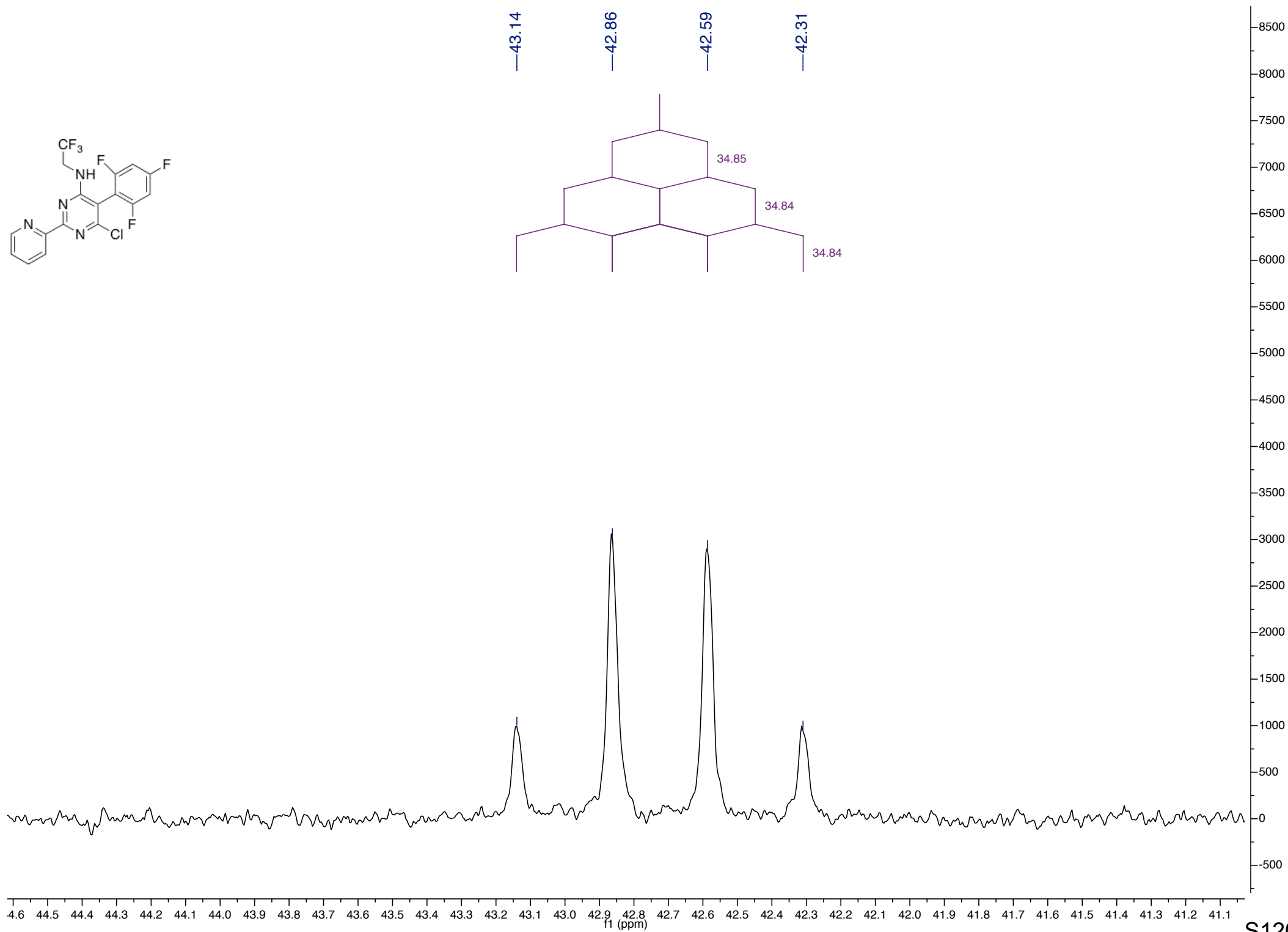


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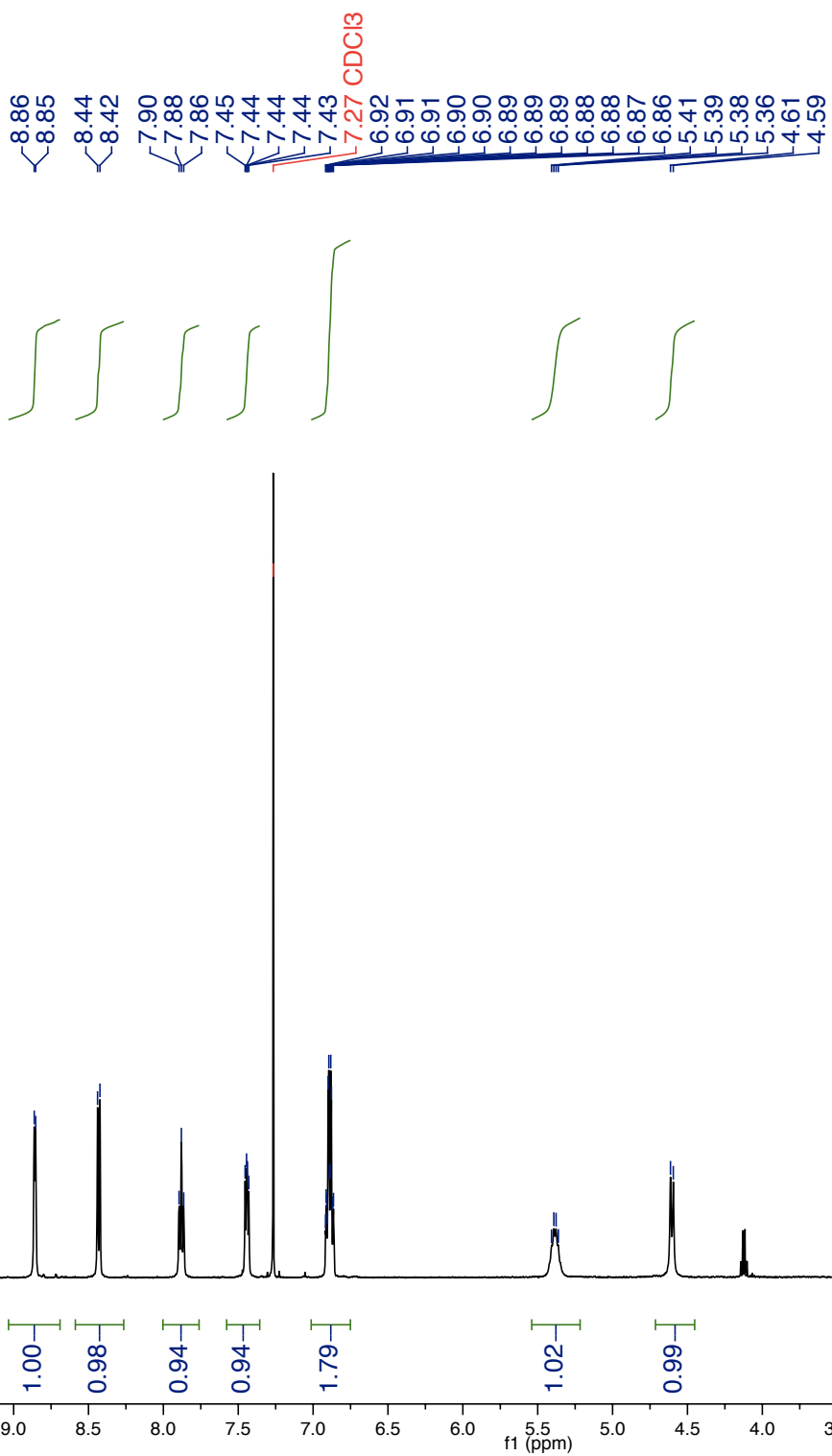
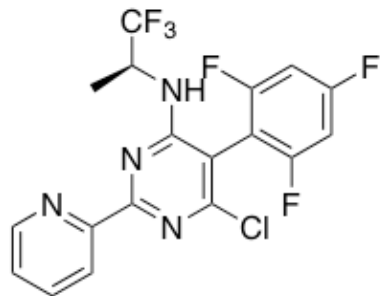
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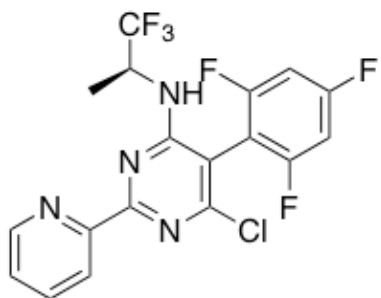
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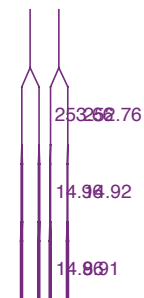




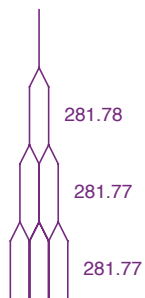




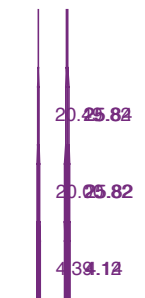
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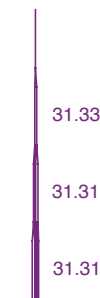
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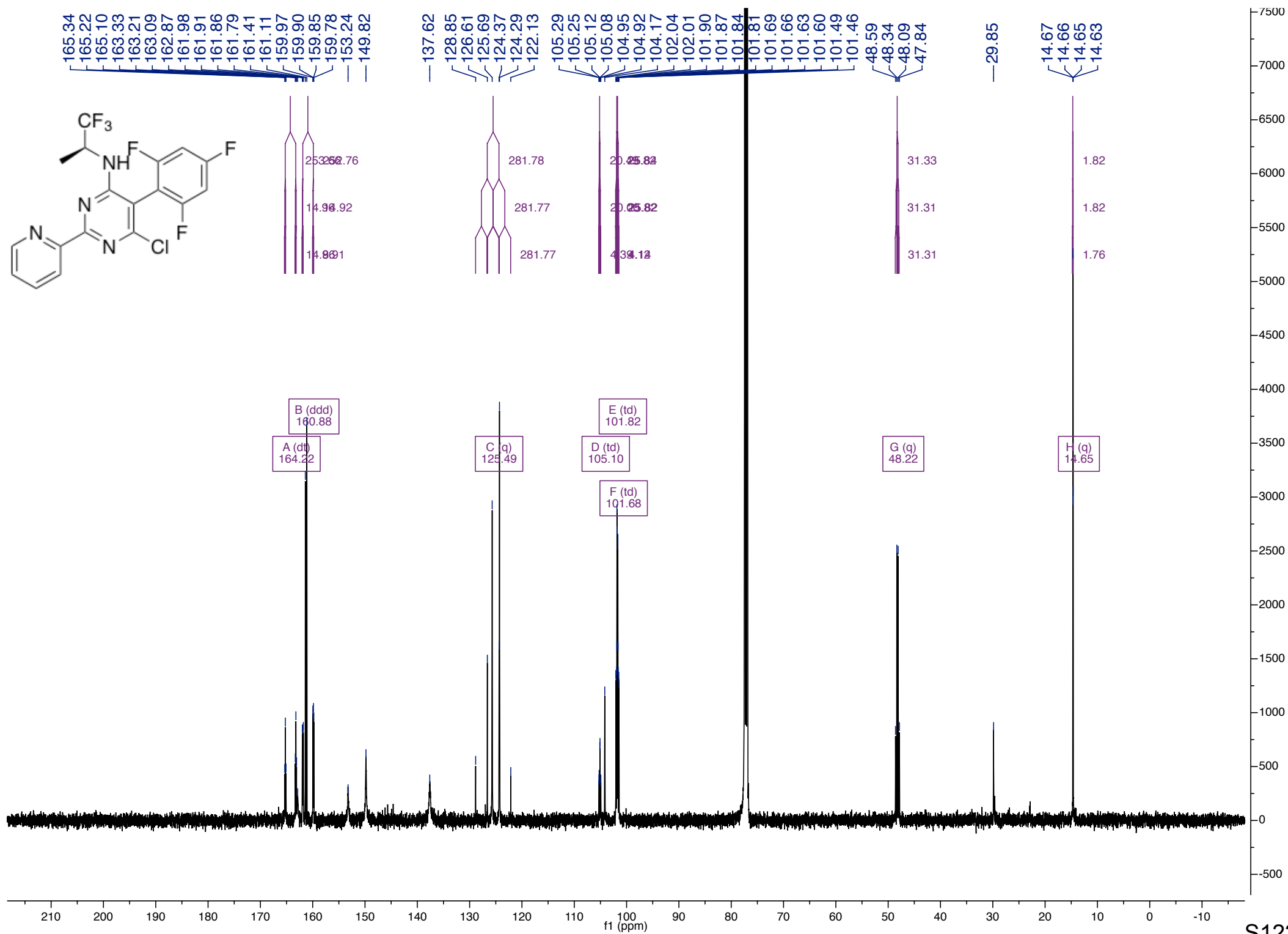


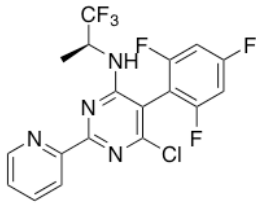
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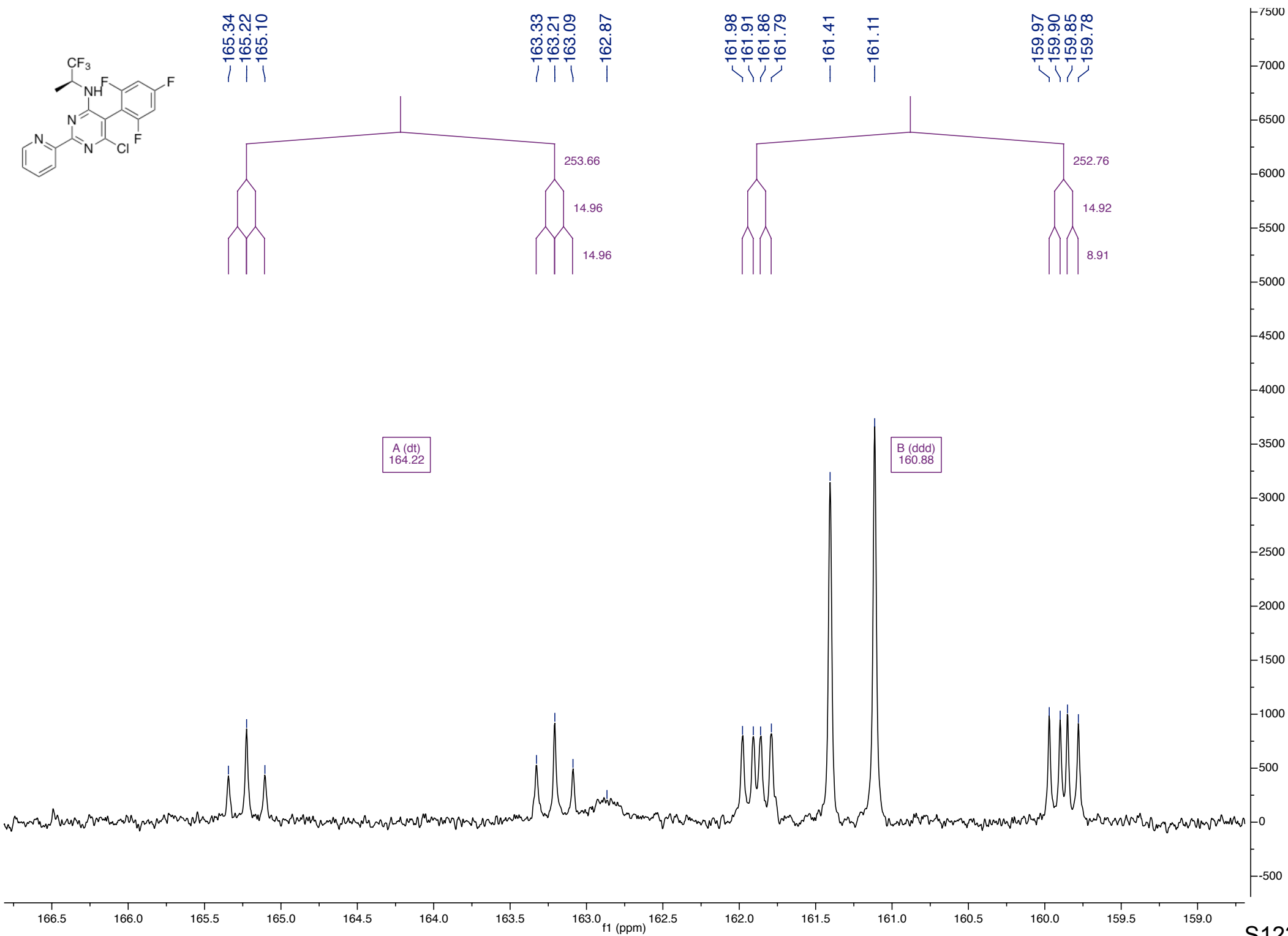
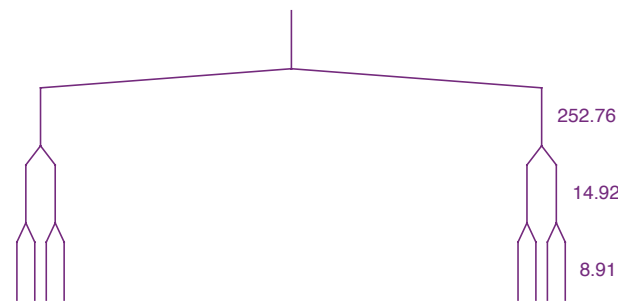
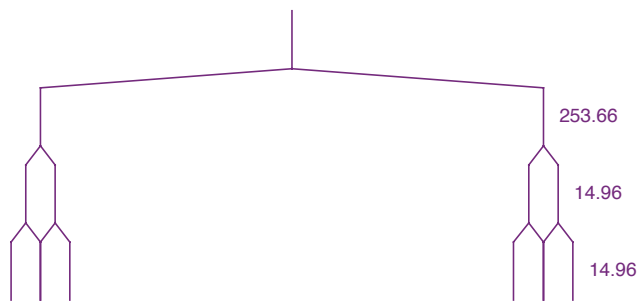


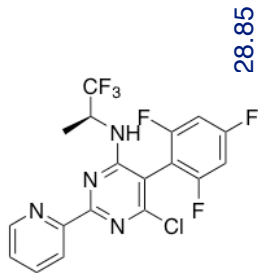
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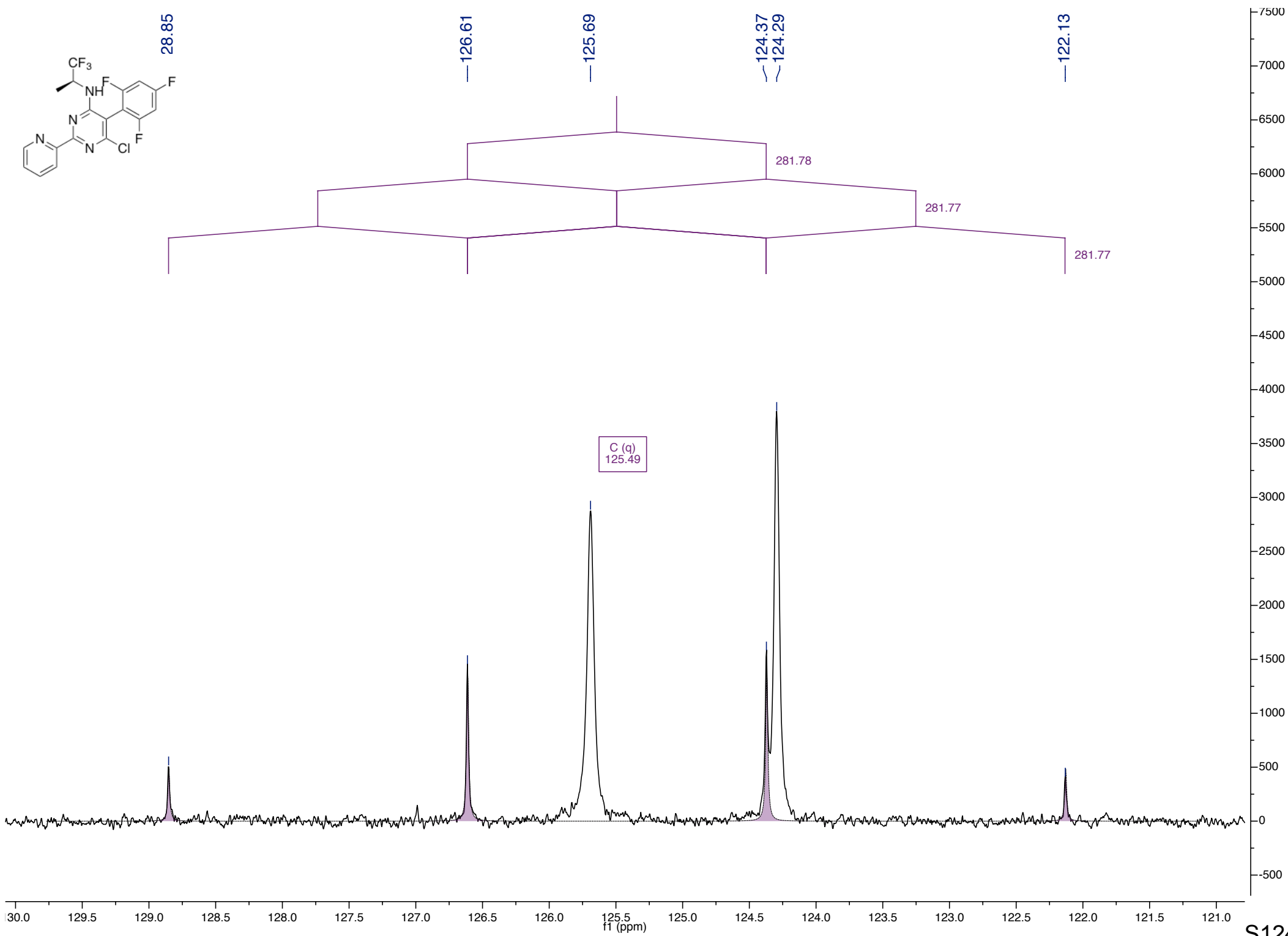
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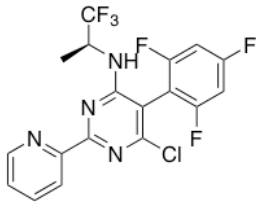
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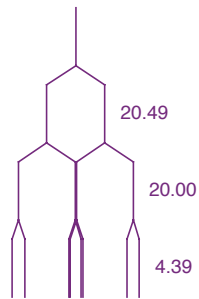


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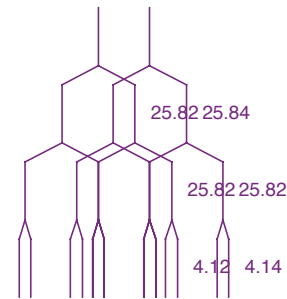


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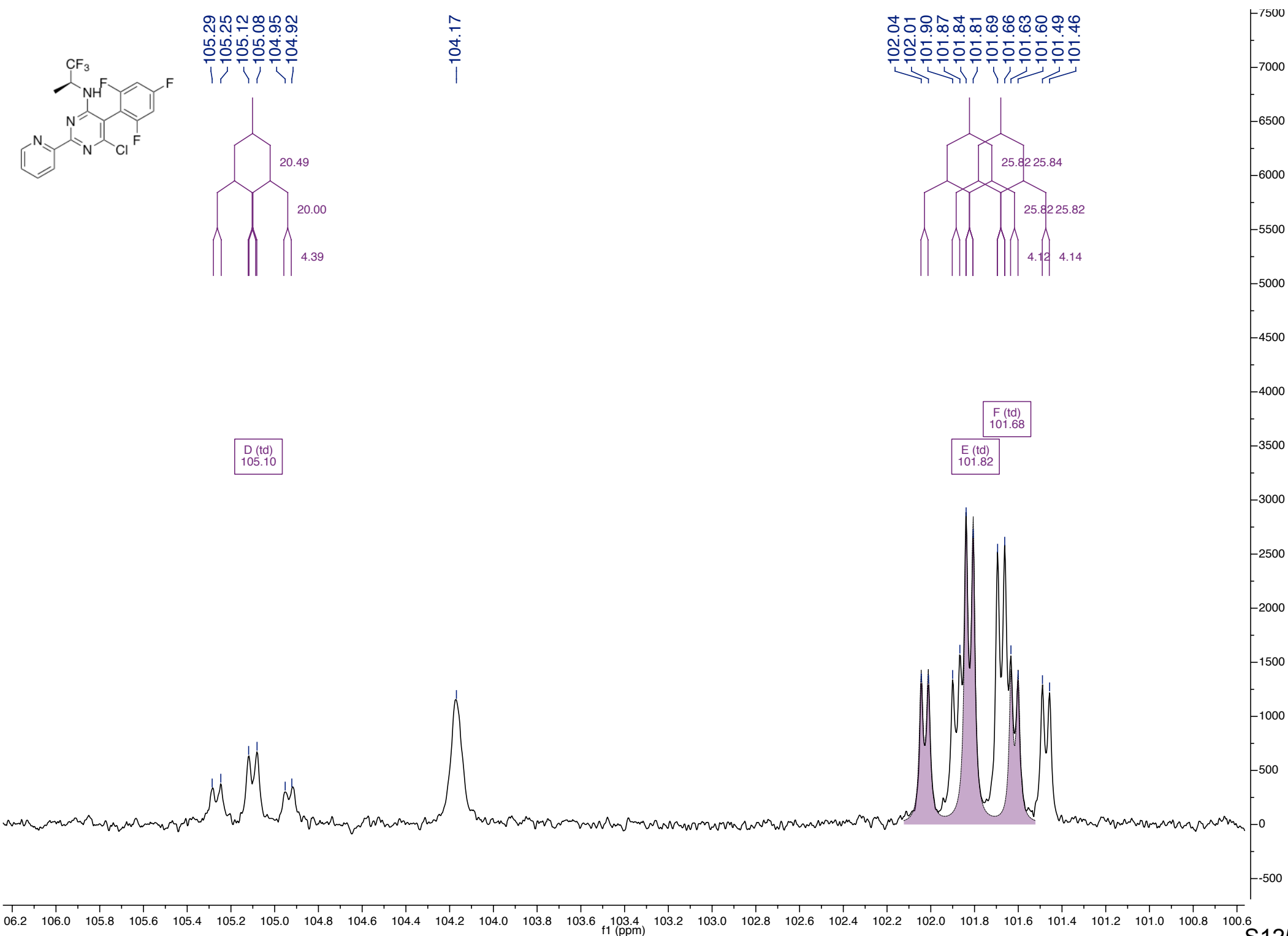
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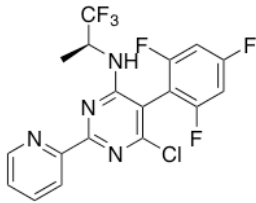


D (td)  
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E (td)  
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F (td)  
101.68

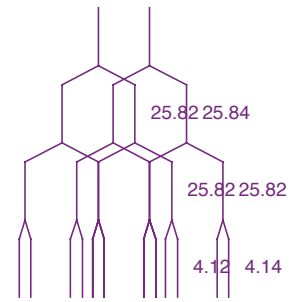
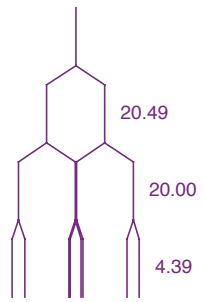




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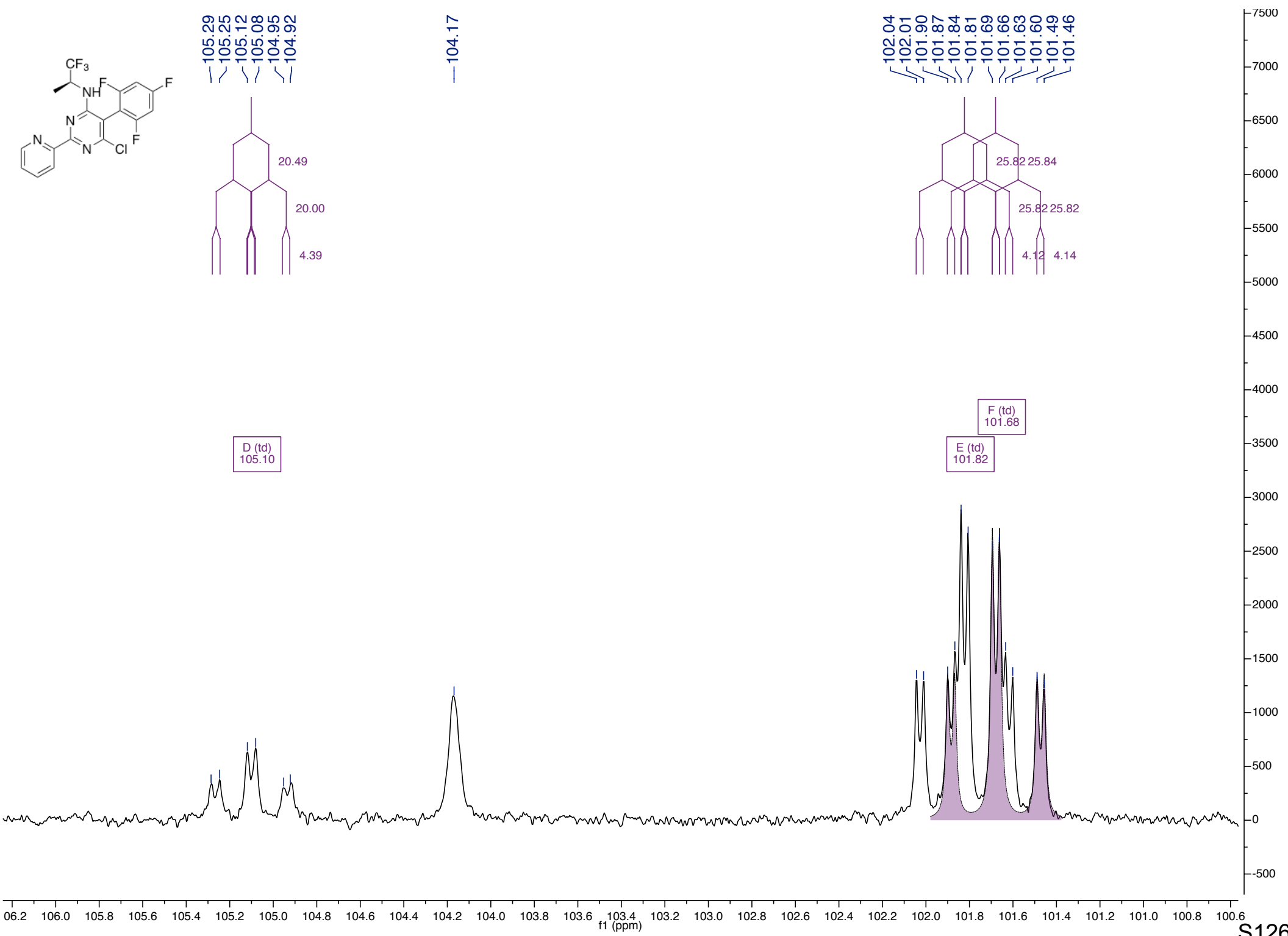
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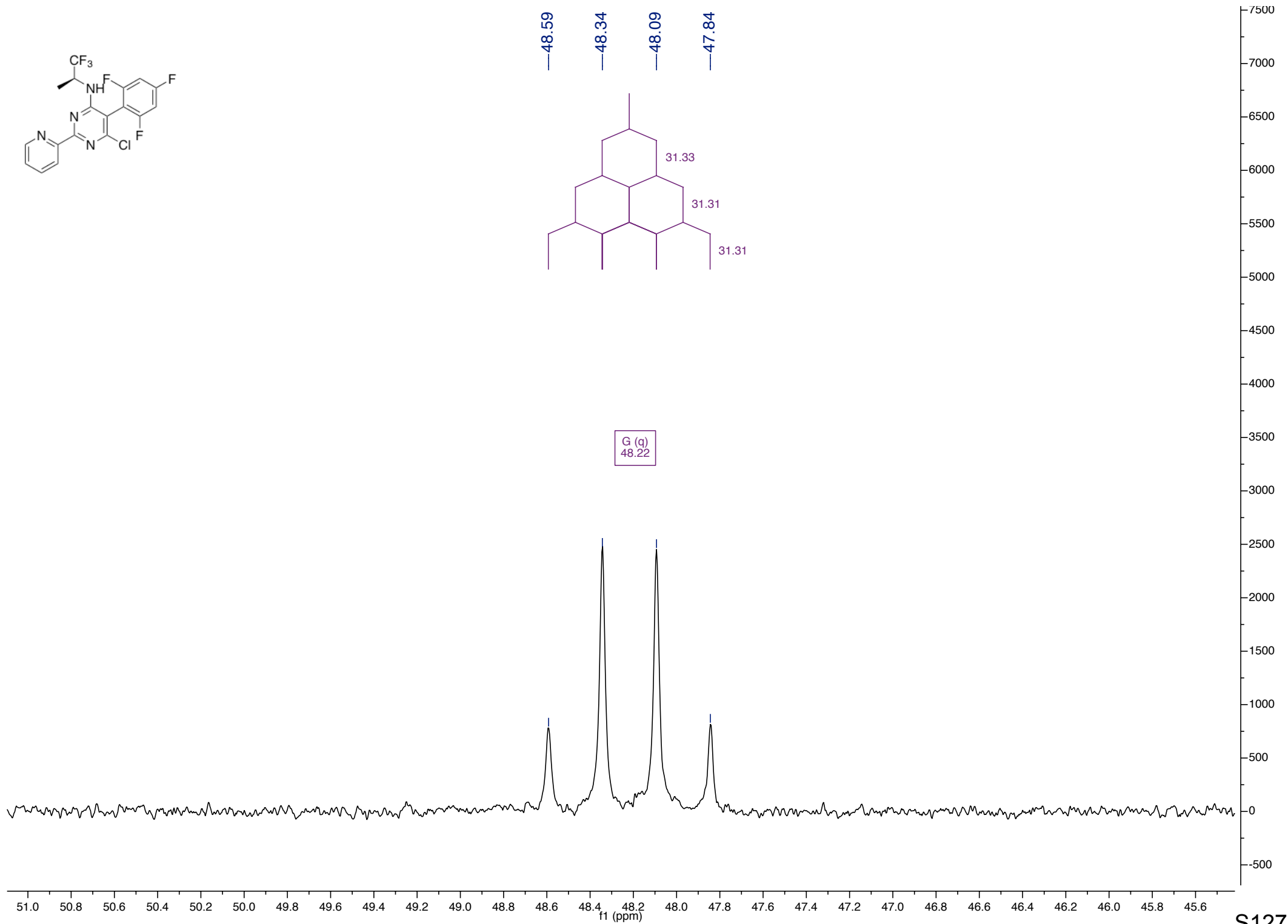
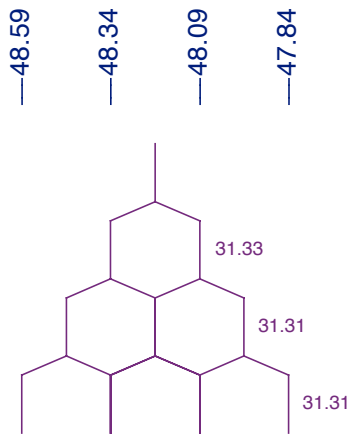
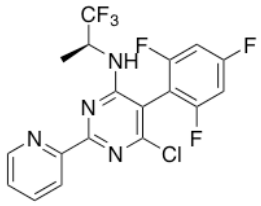


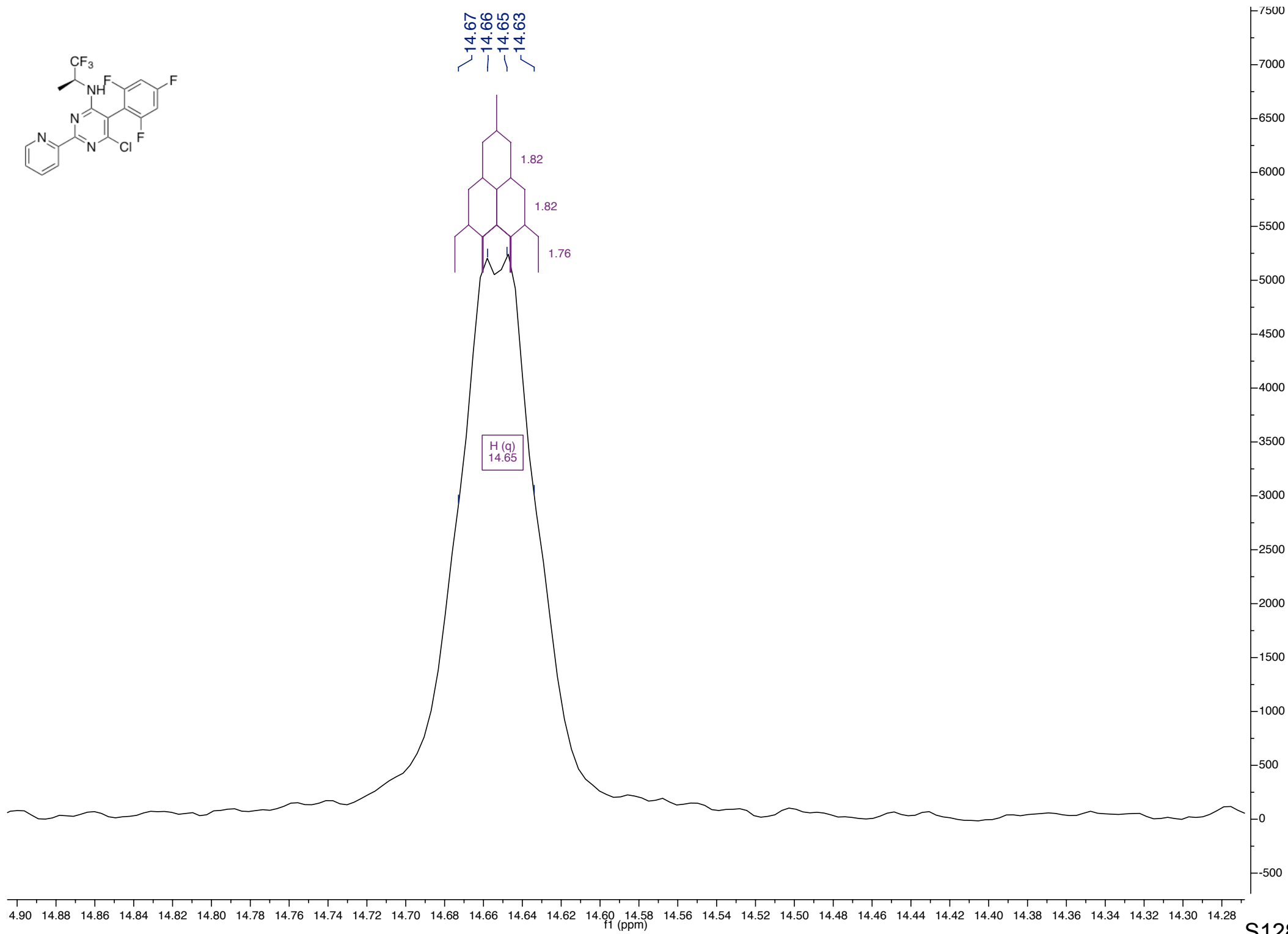
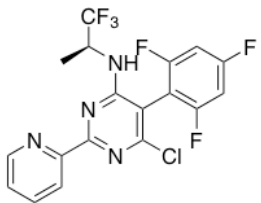
D (td)  
105.10

F (td)  
101.68

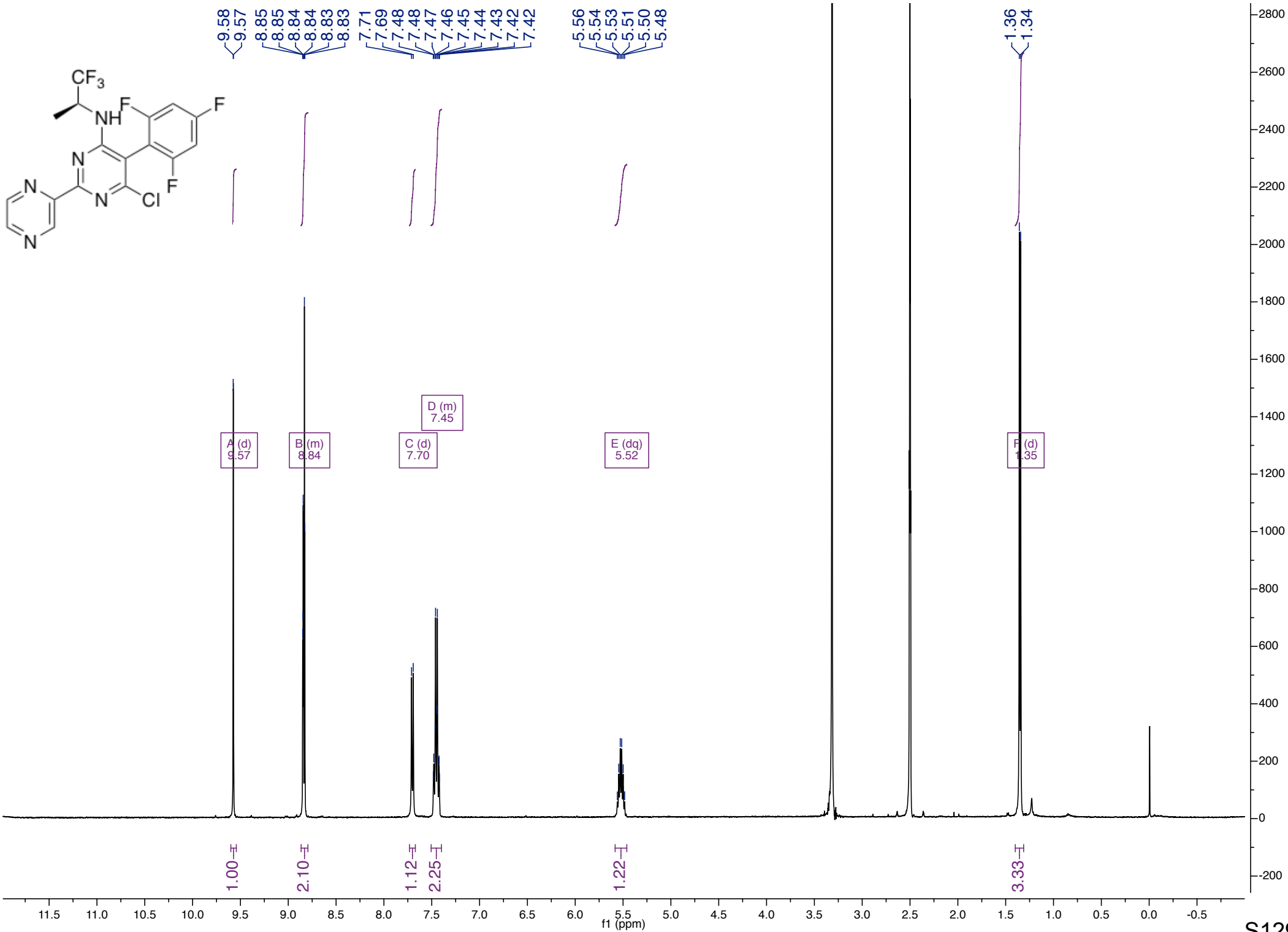
E (td)  
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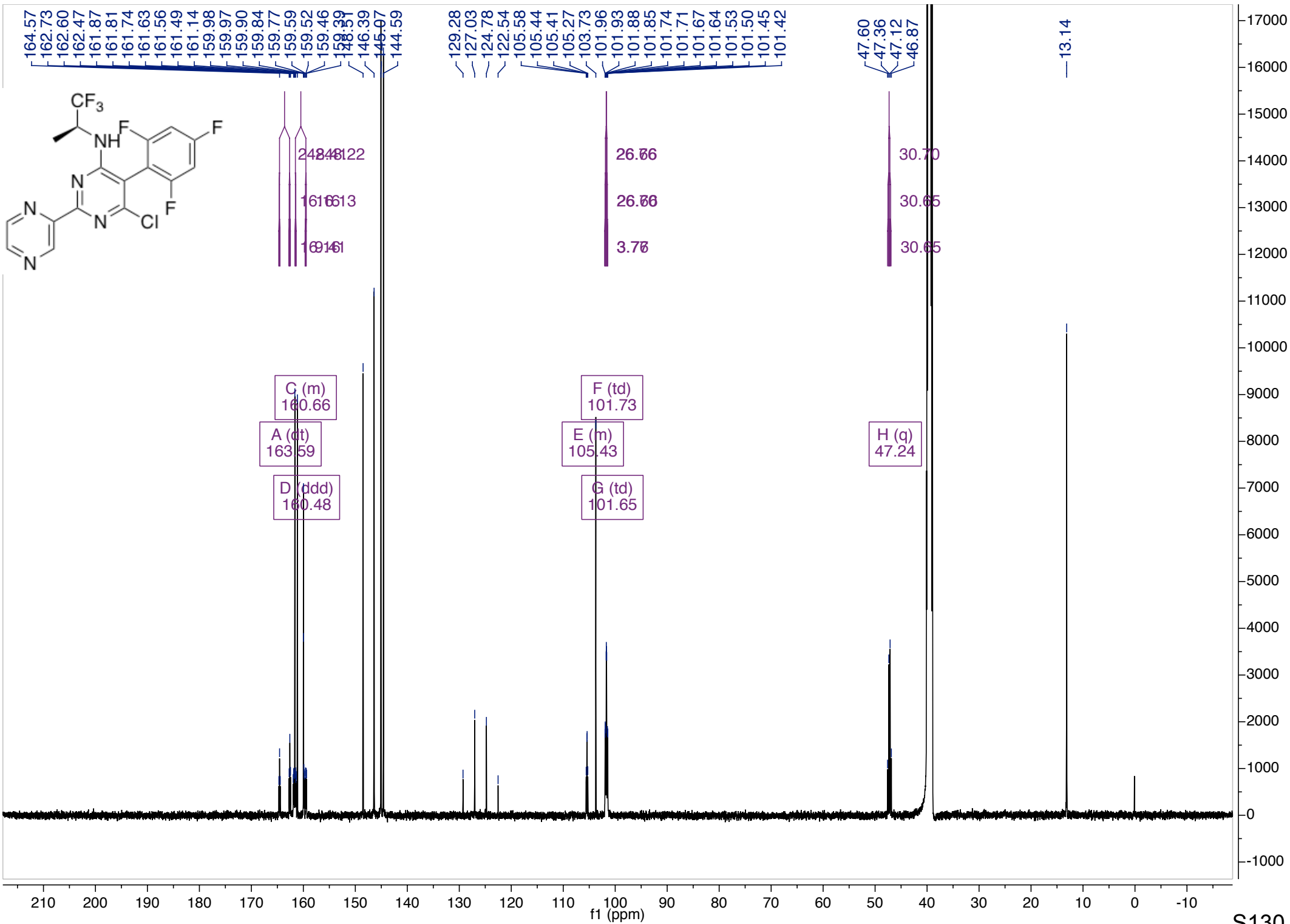


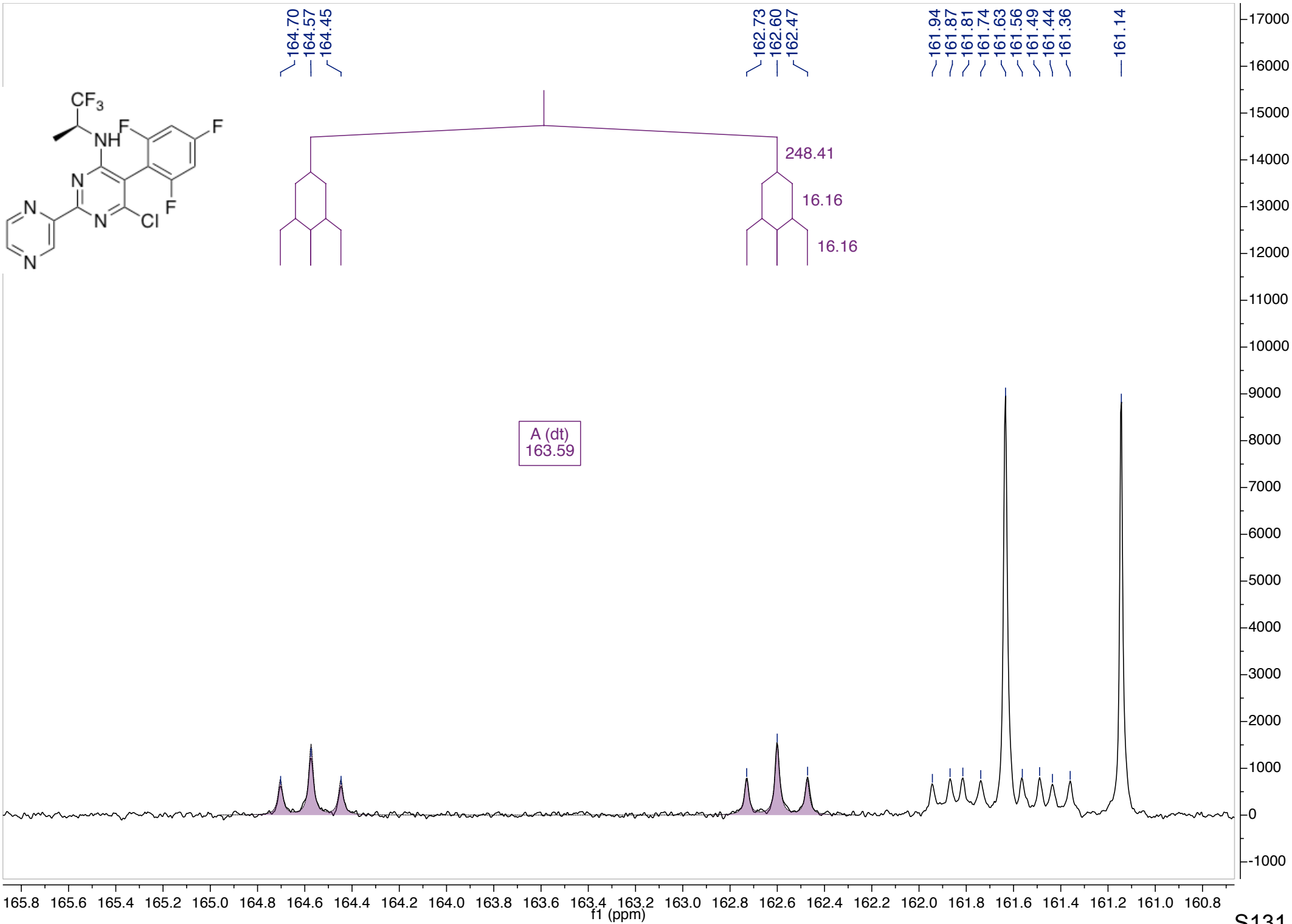


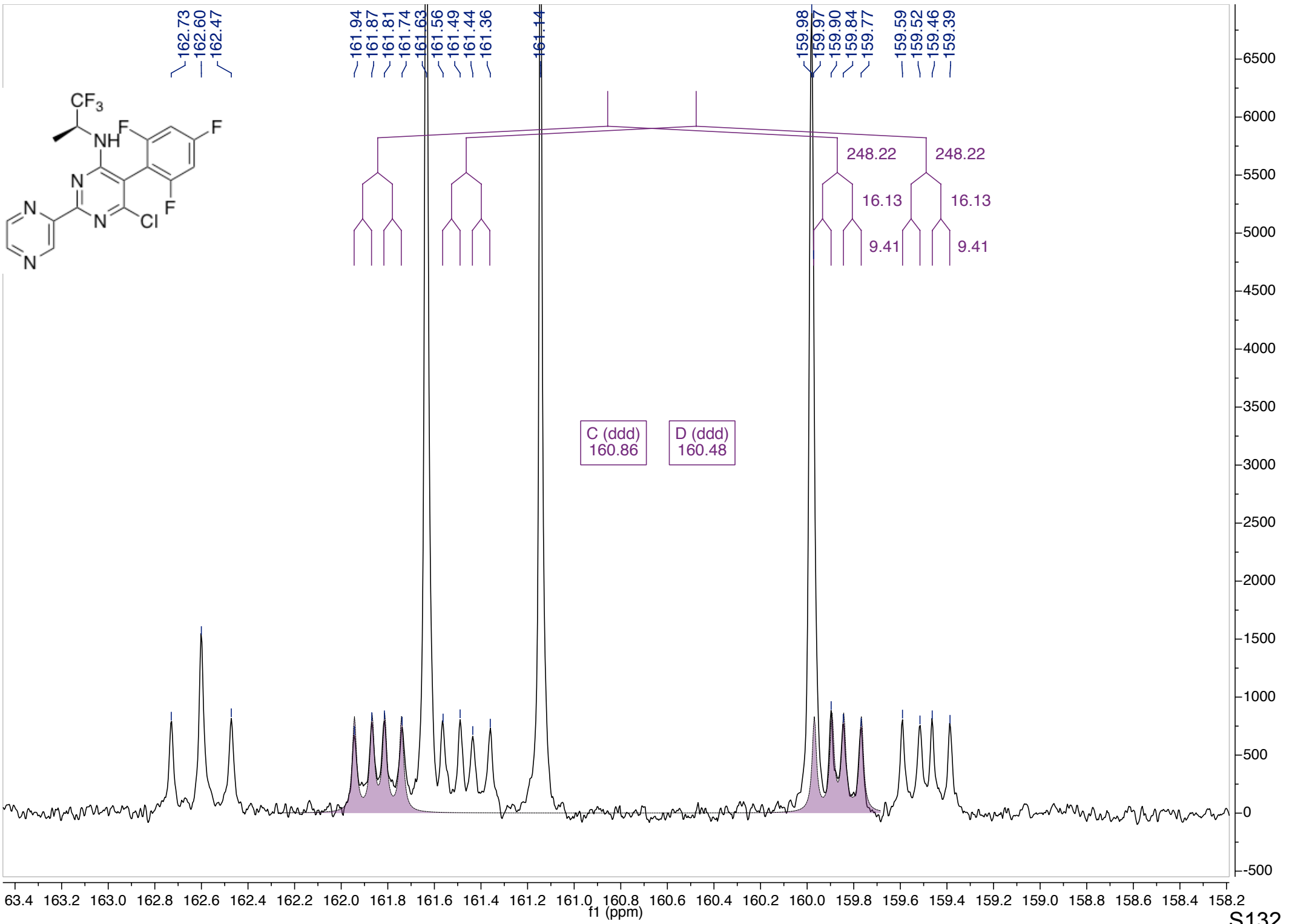


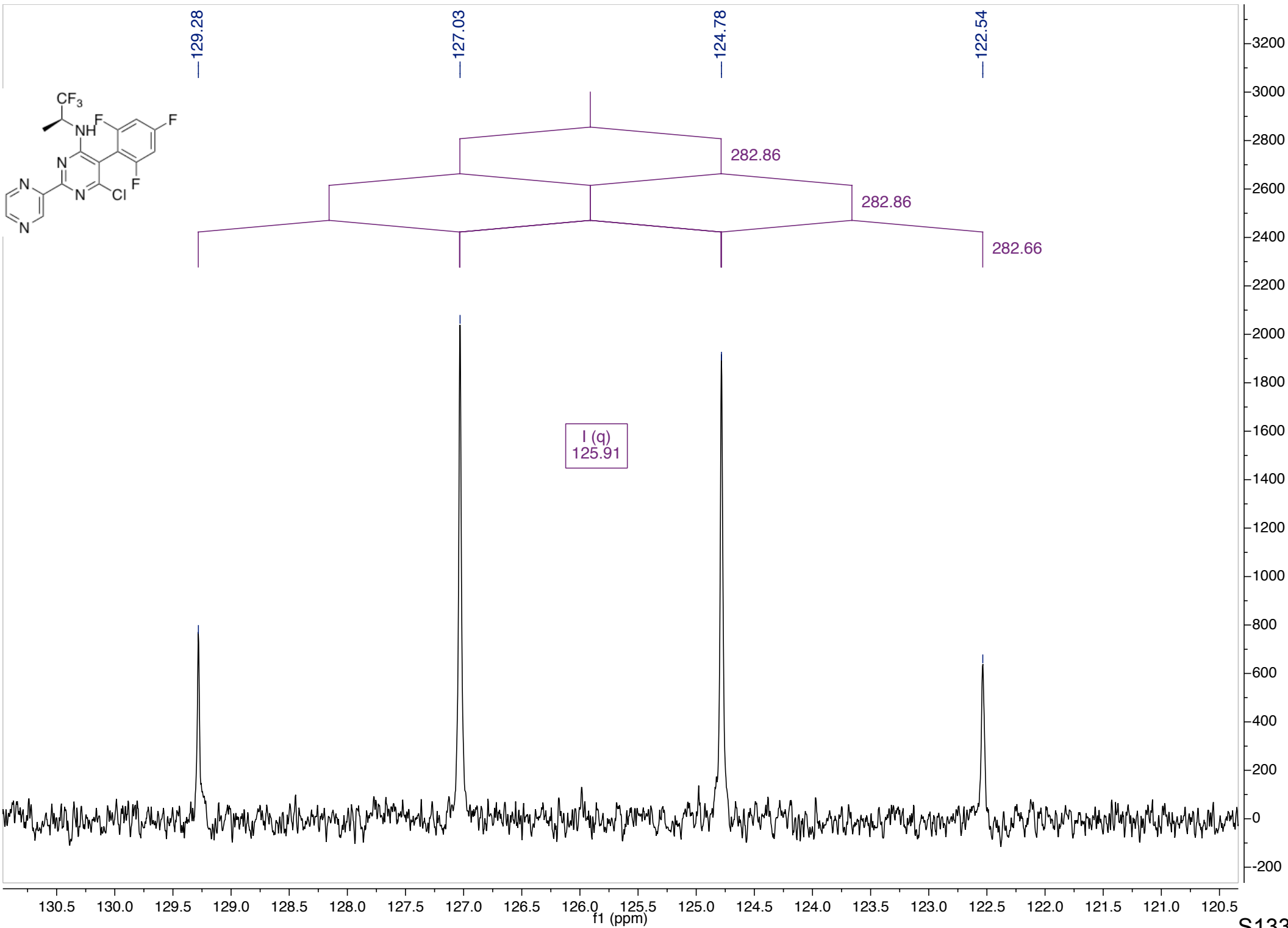


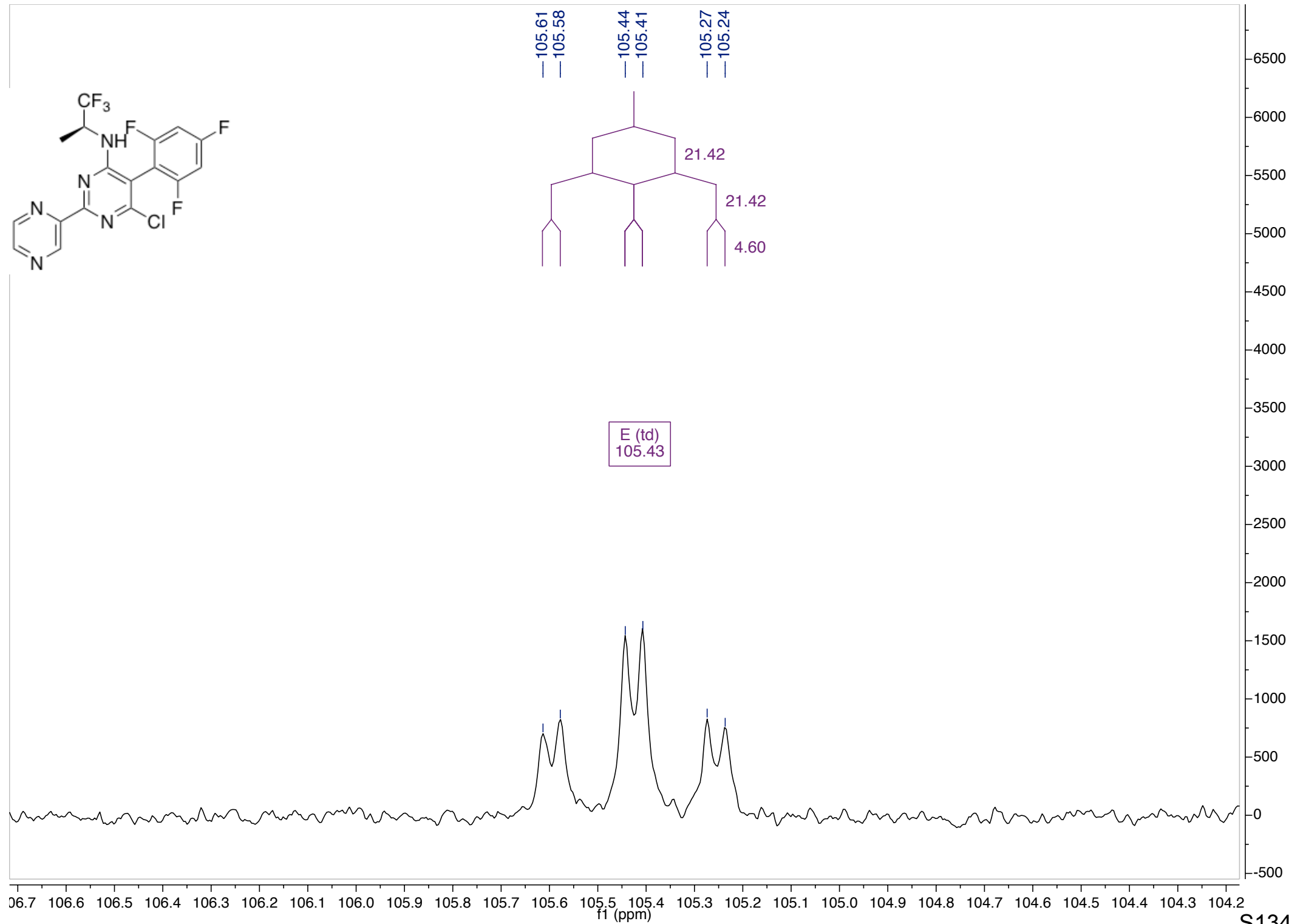
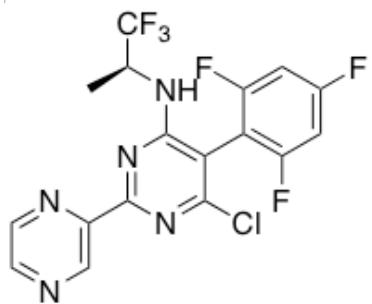


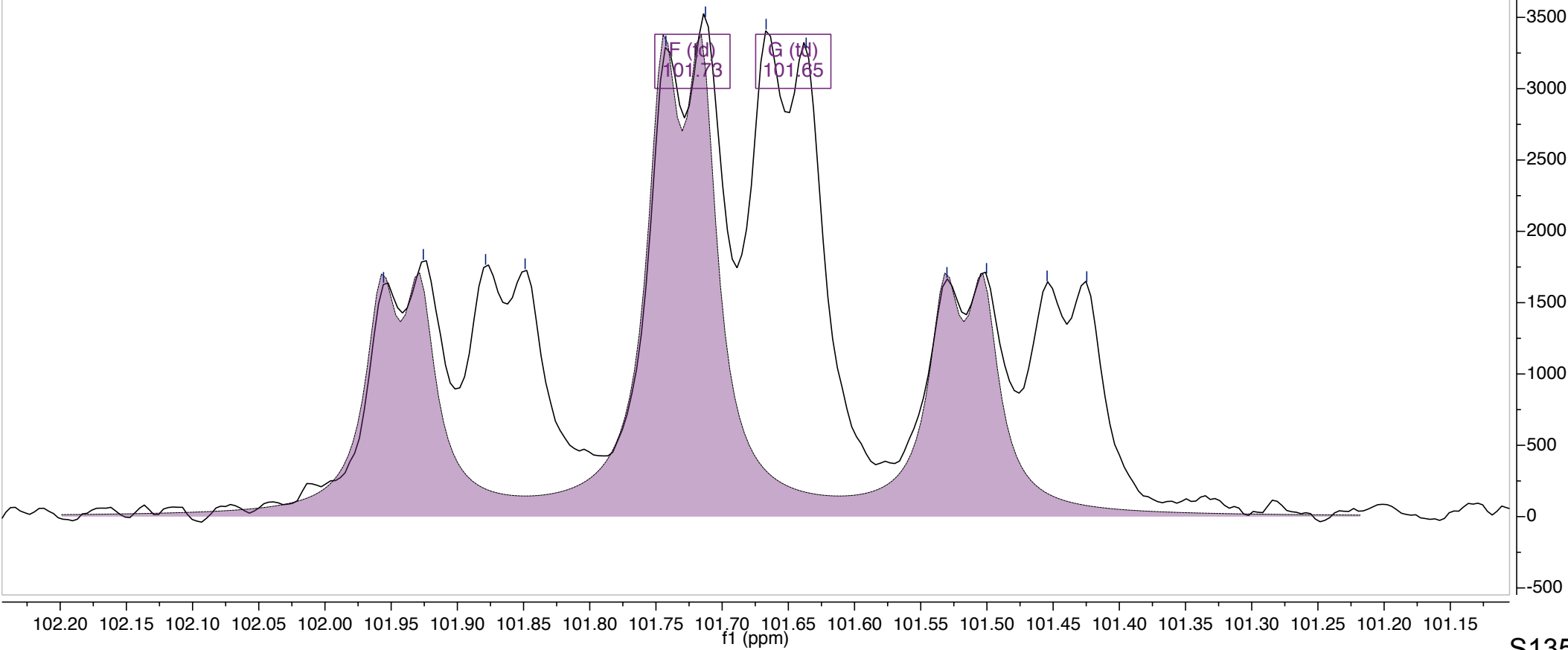
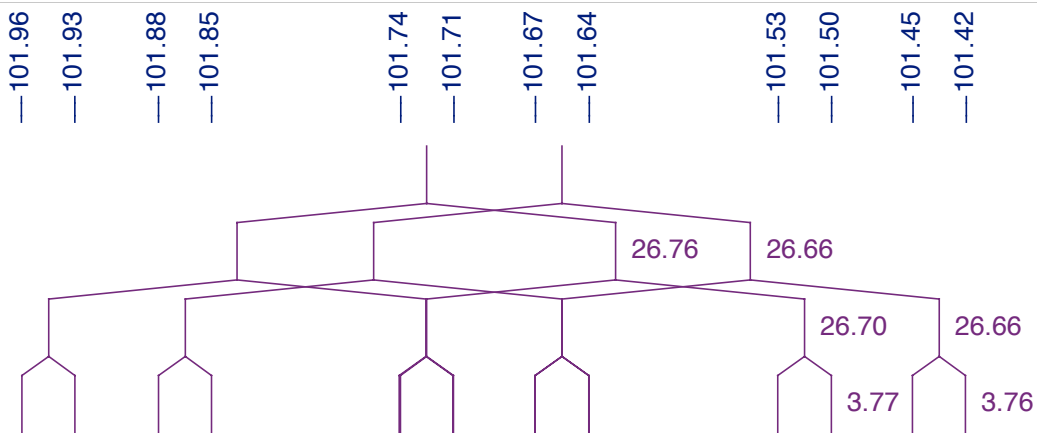
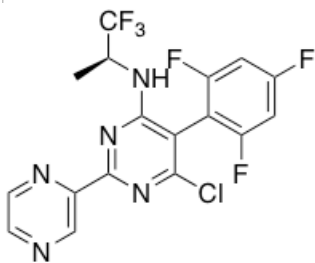


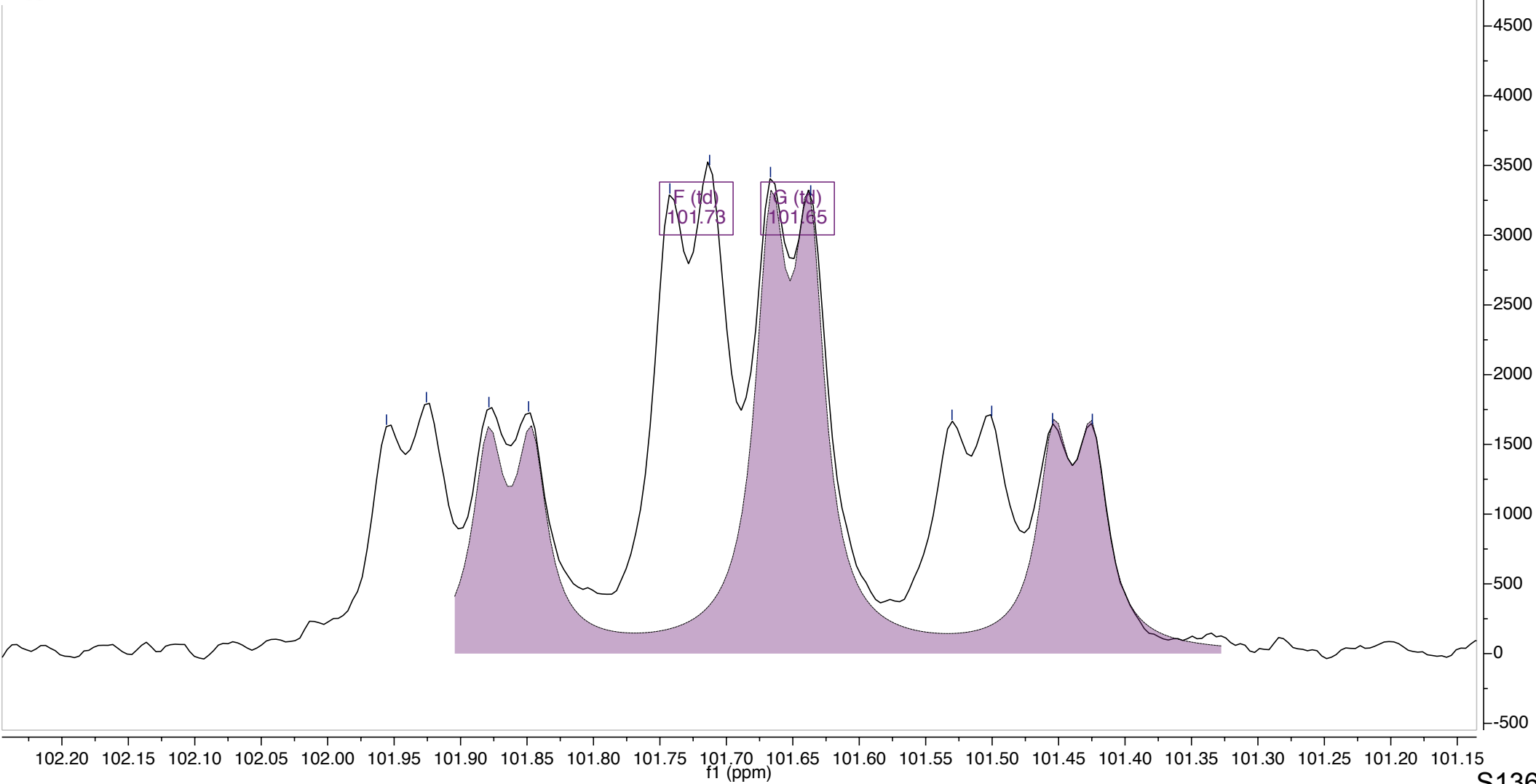
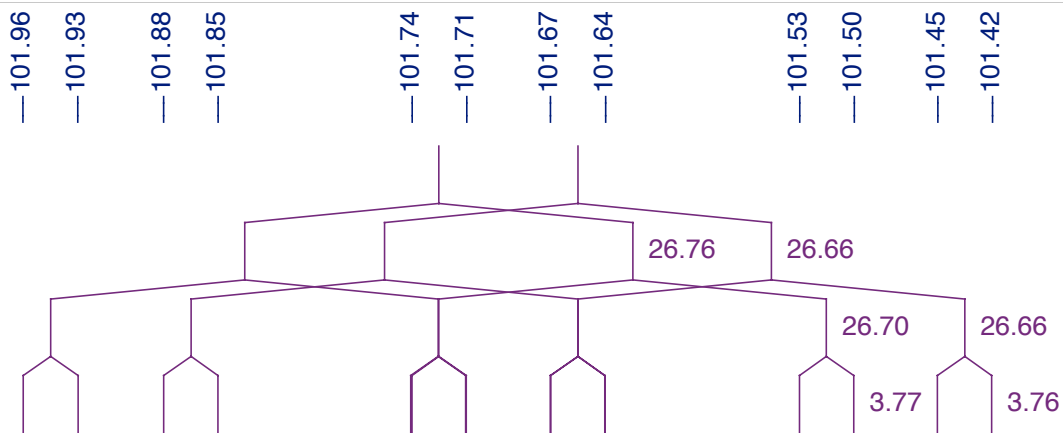
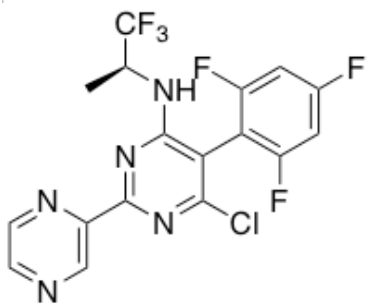




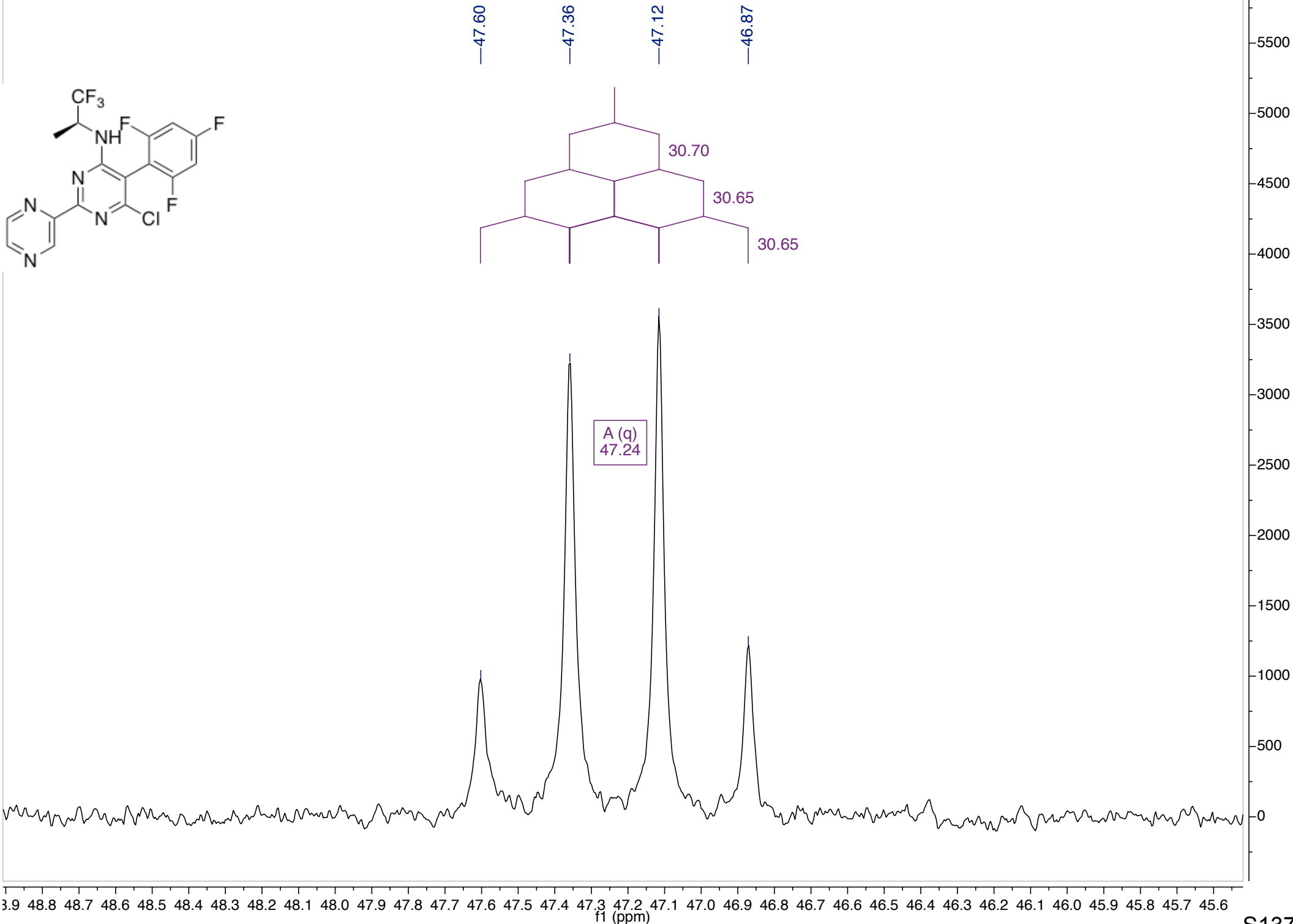
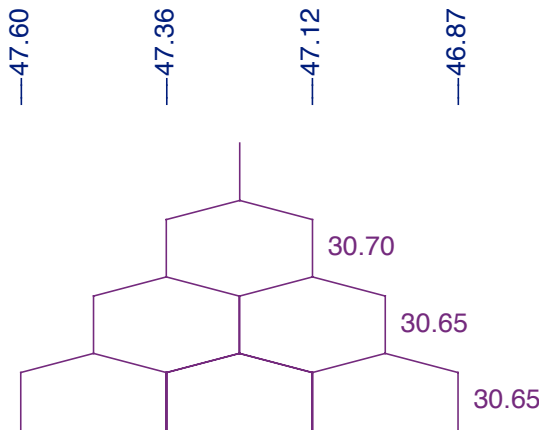
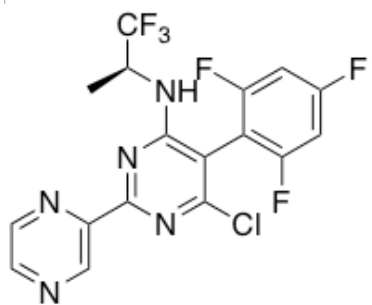


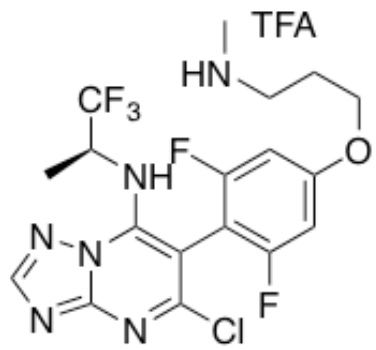












164.67  
164.60  
164.02  
163.94  
163.93  
163.81  
163.70  
162.96  
162.70  
162.68  
162.63  
162.06  
161.99  
159.51  
156.07  
155.91  
149.28

130.04  
127.80  
125.56  
123.32  
119.35  
117.03  
102.97  
102.80  
102.63  
100.77  
100.75  
100.56  
100.54  
100.41  
100.38  
100.20  
100.18  
94.67

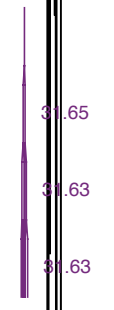
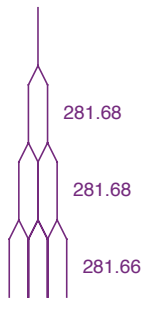
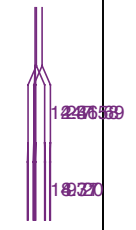
67.48

52.74  
52.49  
52.24  
51.98

34.01

27.05

14.06



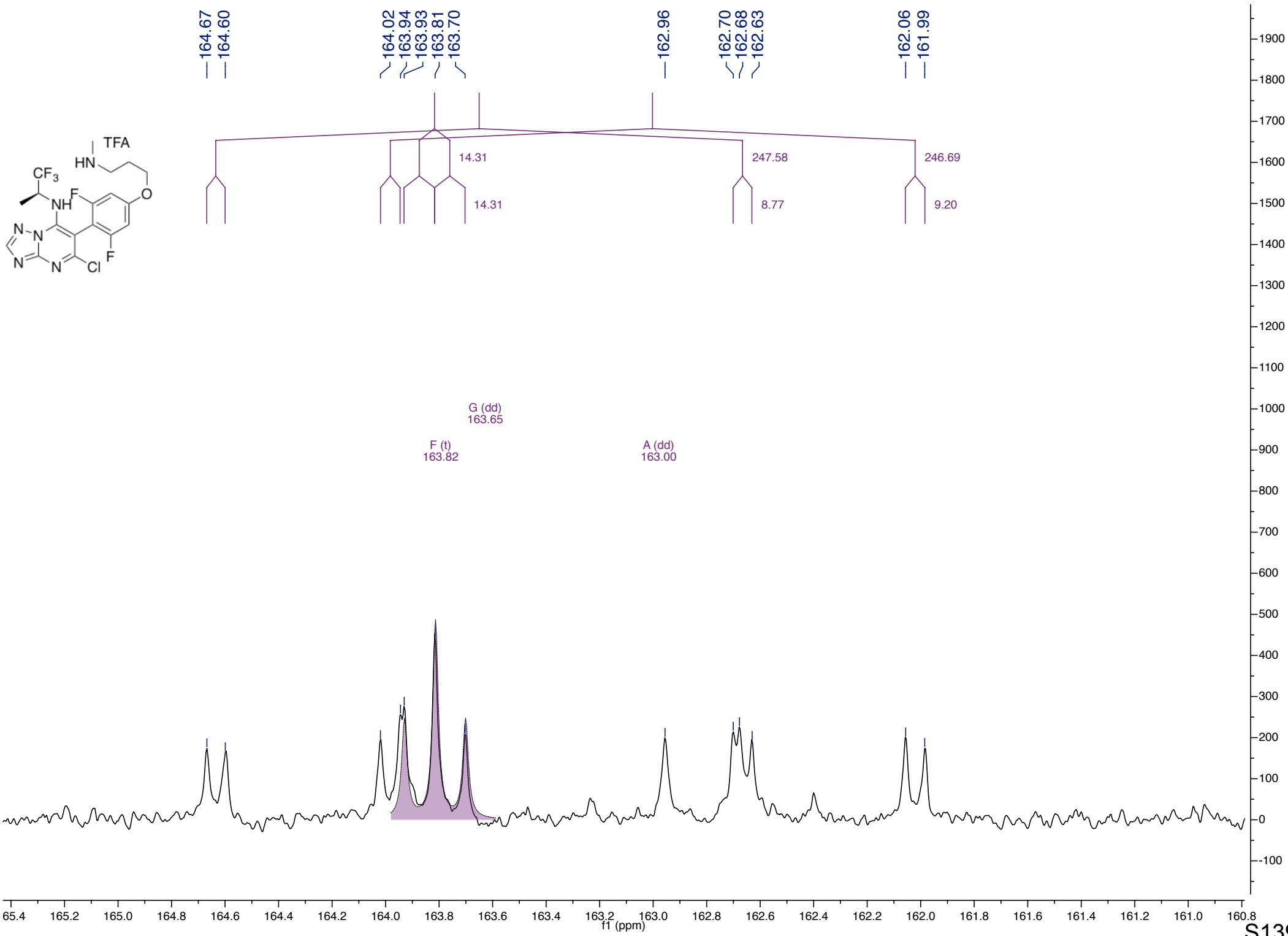
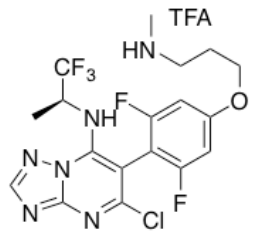
F (t)  
163.82  
A (dd)  
163.00  
G (dd)  
163.63

H (q)  
126.68

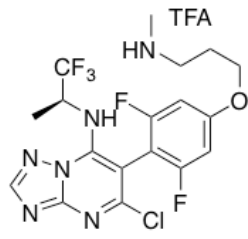
C (dd)  
100.66  
B (t)  
102.80  
D (dd)  
100.29

E (d)  
52.36

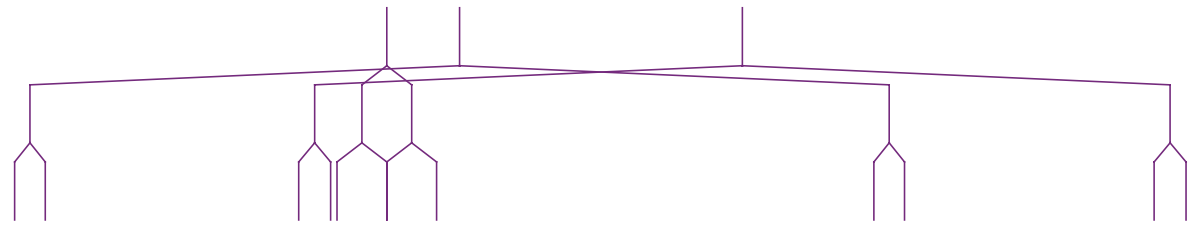
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10  
f1 (ppm)



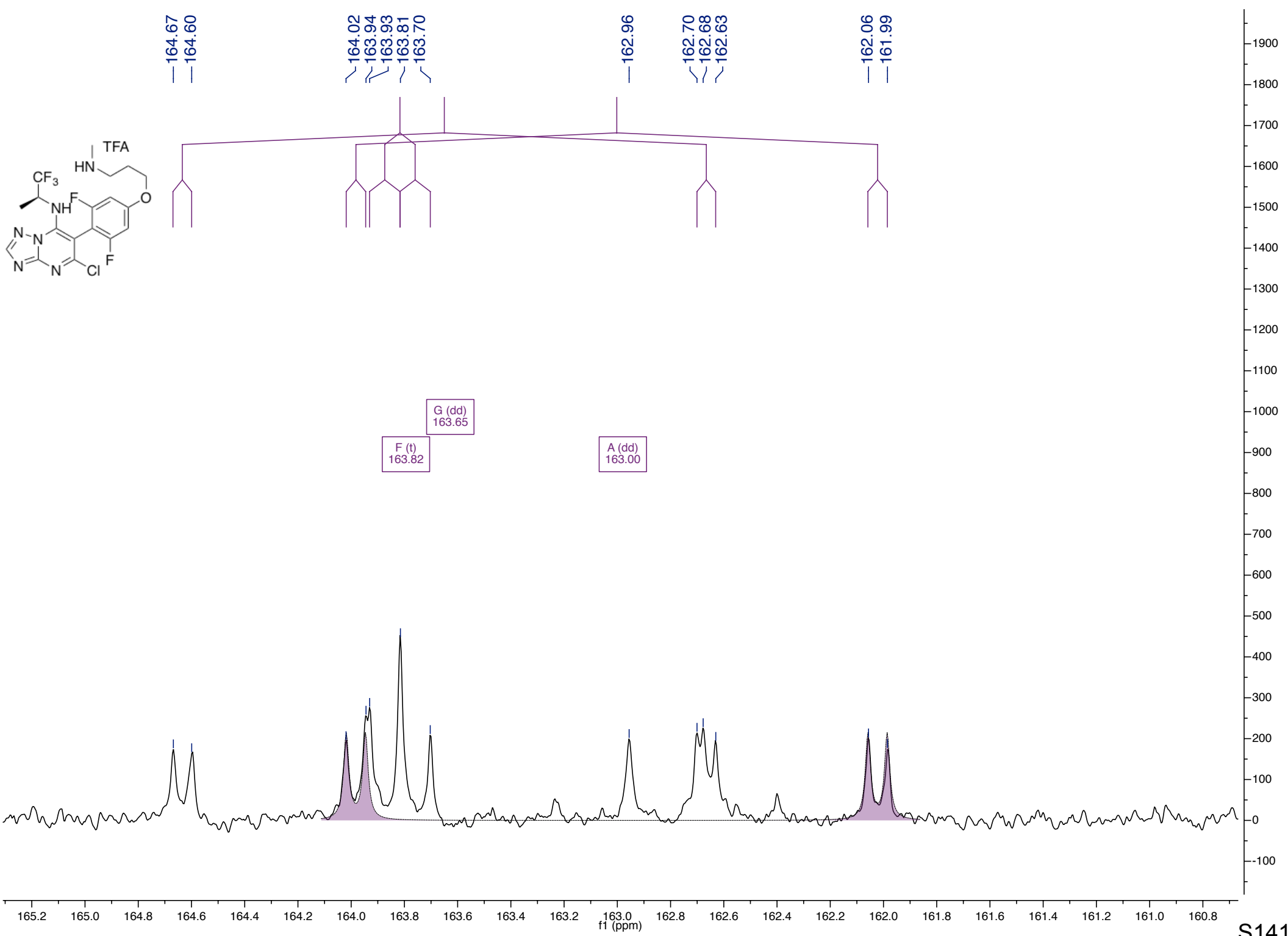


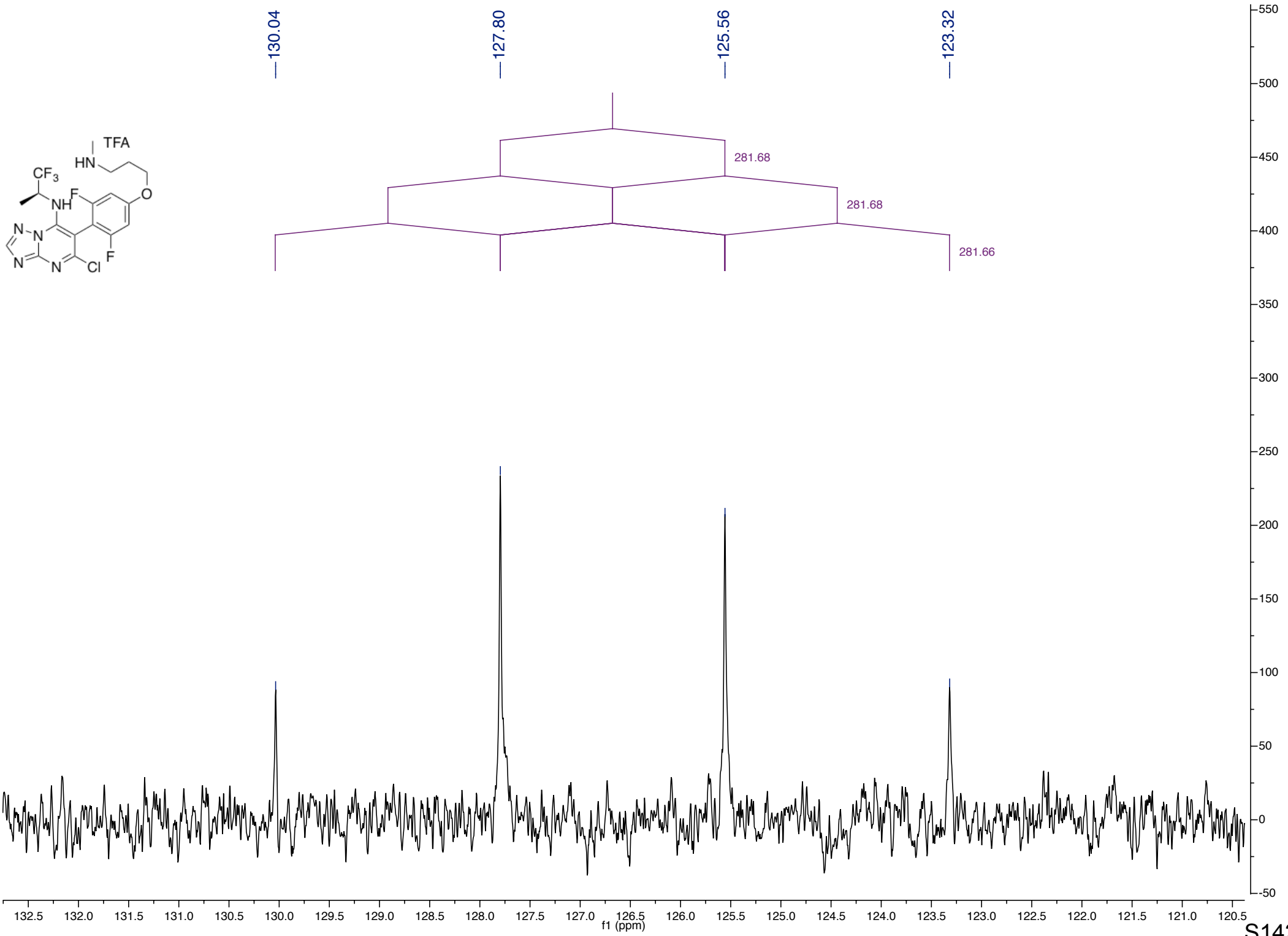
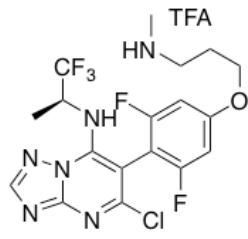


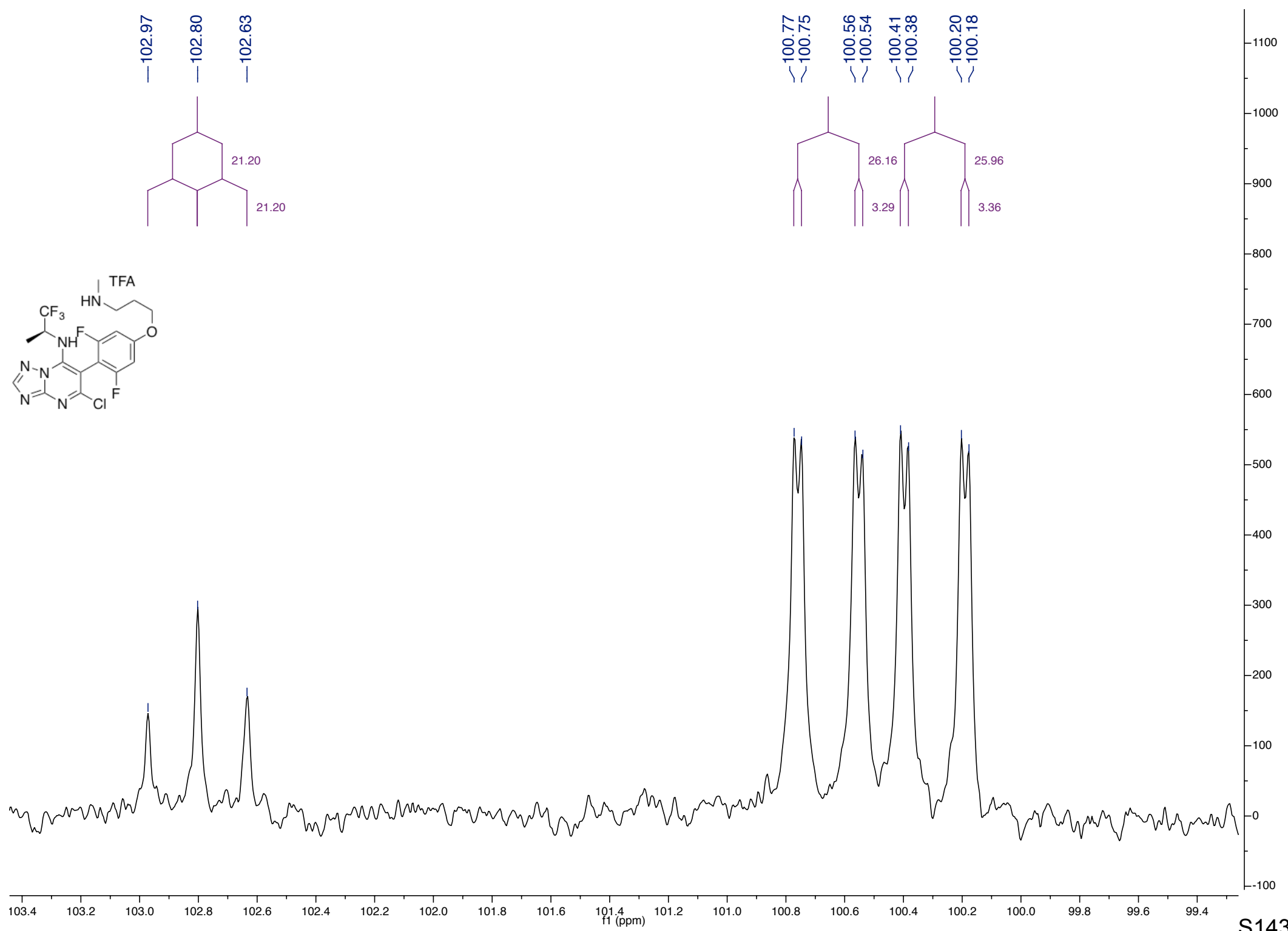
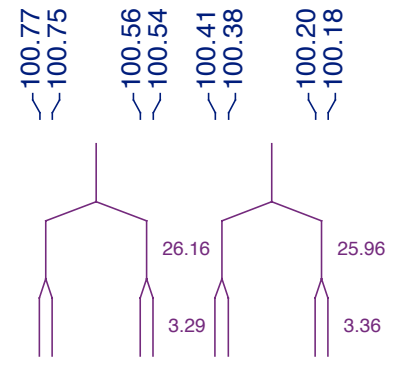
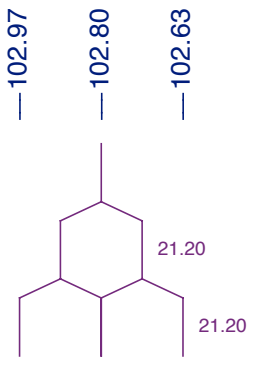
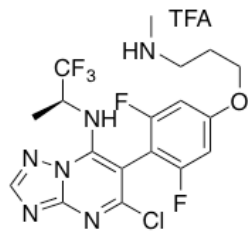
164.67  
164.60  
164.02  
163.94  
163.93  
163.81  
163.70  
162.96  
162.70  
162.68  
162.63  
162.06  
161.99

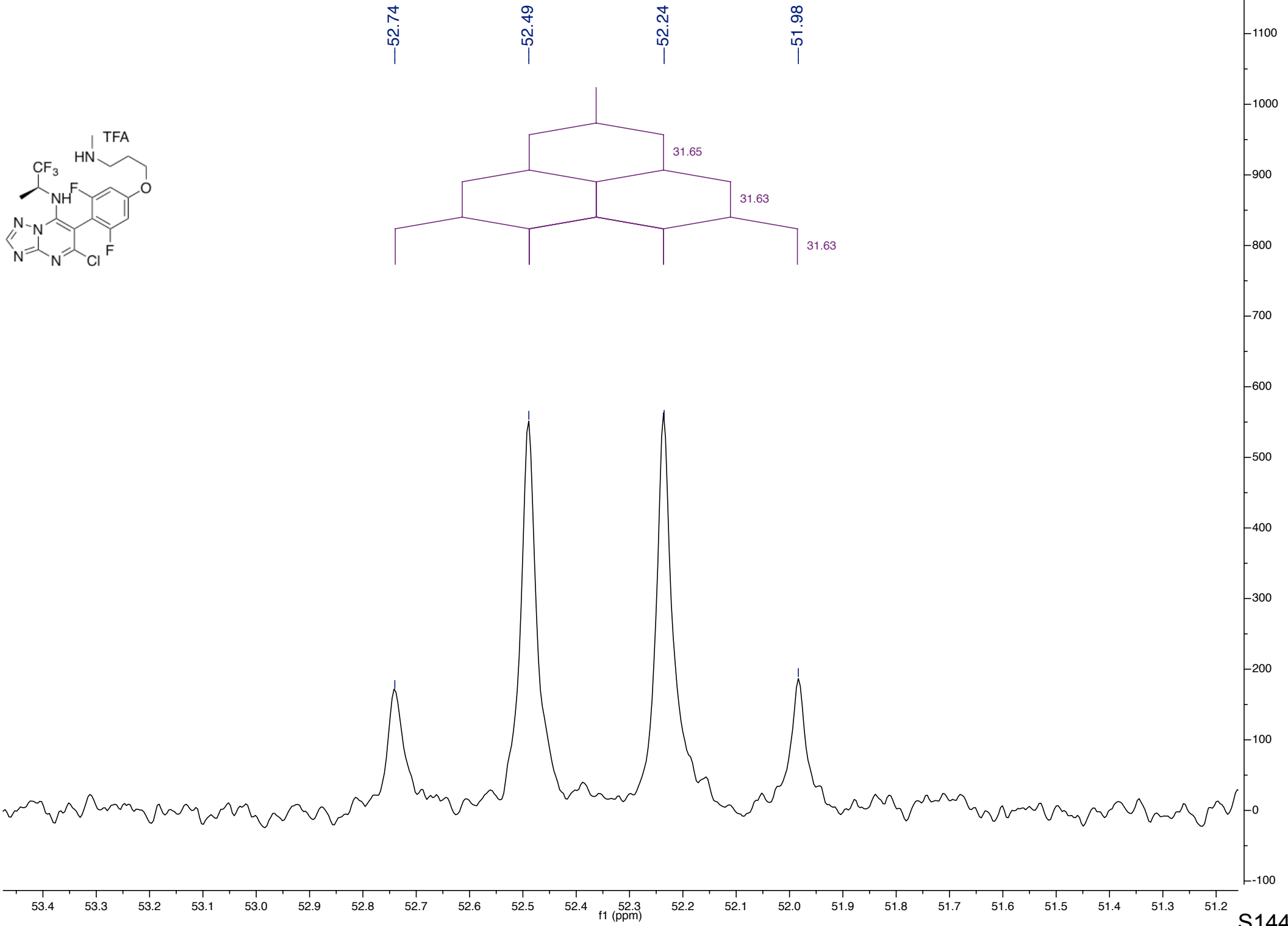
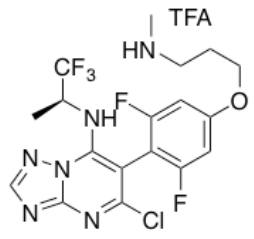


F (t)  
163.82  
G (dd)  
163.65  
A (dd)  
163.00



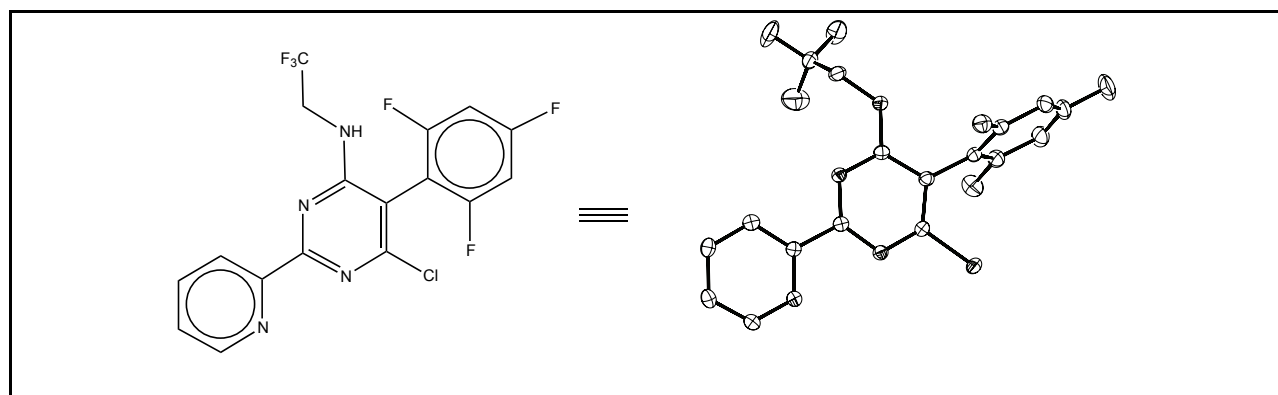








### X-ray Structure Determination of Compound 23 (CCDC 992821)



Compound 1342,  $C_{51}H_{27}N_{12}F_{18}Cl_3$ , crystallizes in the triclinic space group  $P\bar{1}$  with  $a=9.6735(9)\text{\AA}$ ,  $b=17.3727(16)\text{\AA}$ ,  $c=18.984(3)\text{\AA}$ ,  $\alpha=116.369(6)^\circ$ ,  $\beta=94.290(6)^\circ$ ,  $\gamma=102.067(4)^\circ$ ,  $V=2743.6(6)\text{\AA}^3$ ,  $Z=2$ , and  $d_{\text{calc}}=1.521\text{ g/cm}^3$ . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-K $\alpha$  radiation ( $\lambda=0.71073\text{ \AA}$ ) at a temperature of 143(1)K. Preliminary indexing was performed from a series of thirty-six  $0.5^\circ$  rotation frames with exposures of 10 seconds. A total of 2378 frames were collected with a crystal to detector distance of 37.646 mm, rotation widths of  $0.5^\circ$  and exposures of 5 seconds:

scan type	$2\theta$	$\omega$	$\phi$	$\chi$	frames
$\phi$	19.50	59.55	348.71	-26.26	739
$\phi$	-15.50	349.33	93.82	-77.44	161
$\phi$	-23.00	334.21	38.95	73.66	739
$\phi$	-23.00	315.83	12.48	28.88	739

Rotation frames were integrated using SAINT<sup>i</sup>, producing a listing of unaveraged  $F^2$  and  $\sigma(F^2)$  values which were then passed to the SHELXTL<sup>ii</sup> program package for further processing and structure solution. A total of 54588 reflections were measured over the ranges  $2.16 \leq \theta \leq 25.46^\circ$ ,  $-11 \leq h \leq 11$ ,  $-20 \leq k \leq 20$ ,  $-22 \leq l \leq 22$  yielding 9973 unique reflections ( $R_{\text{int}} = 0.0221$ ). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS<sup>iii</sup> (minimum and maximum transmission 0.6979, 0.7452).

The structure was solved by direct methods (SHELXS-97<sup>iv</sup>). There was a region of disordered dichloromethane solvent for which a reliable disorder model could not be devised; the X-ray data were

corrected for the presence of disordered solvent using SQUEEZE<sup>v</sup>. Refinement was by full-matrix least squares based on  $F^2$  using SHELXL-97.<sup>vi</sup> All reflections were used during refinement. The weighting scheme used was  $w=1/[\sigma^2(F_o^2) + (0.0523P)^2 + 1.7350P]$  where  $P = (F_o^2 + 2F_c^2)/3$ . Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to  $R1=0.0418$  and  $wR2=0.1095$  for 8420 observed reflections for which  $F > 4\sigma(F)$  and  $R1=0.0490$  and  $wR2=0.1152$  and  $GOF = 1.100$  for all 9973 unique, non-zero reflections and 758 variables.<sup>vii</sup> The maximum  $\Delta/\sigma$  in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were  $+0.541$  and  $-0.558 e/\text{\AA}^3$ .

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figures 1., 2., and 3. are ORTEP<sup>viii</sup> representations of the molecule with 30% probability thermal ellipsoids displayed.

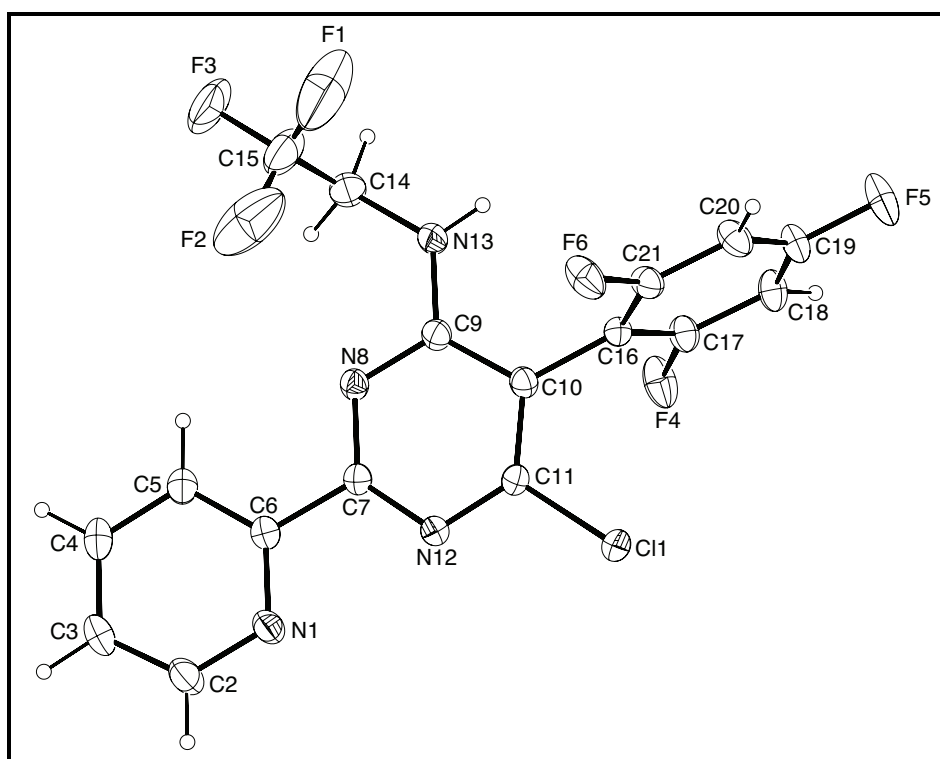


Figure 1. ORTEP drawing of molecule no. 1 of the asymmetric unit with 30% probability thermal ellipsoids.

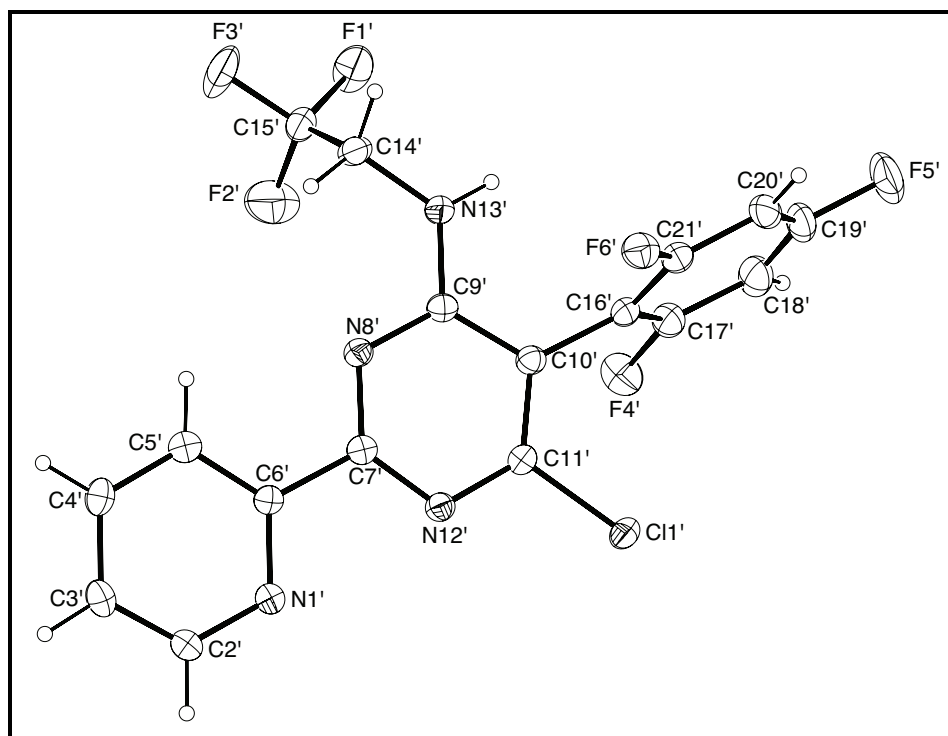


Figure 2. ORTEP drawing of molecule no. 2 of the asymmetric unit with 30% probability thermal ellipsoids.



Figure 3. ORTEP drawing of molecule no. 3 of the asymmetric unit with 30% probability thermal ellipsoids.

**Table 1. Summary of Structure Determination of Compound 1342**

Empirical formula	C <sub>51</sub> H <sub>27</sub> N <sub>12</sub> F <sub>18</sub> Cl <sub>3</sub>
Formula weight	1256.20
Temperature	143(1) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	P $\bar{1}$
Cell constants:	
a	9.6735(9) Å
b	17.3727(16) Å
c	18.984(3) Å
$\alpha$	116.369(6)°
$\beta$	94.290(6)°
$\gamma$	102.067(4)°
Volume	2743.6(6) Å <sup>3</sup>
Z	2
Density (calculated)	1.521 Mg/m <sup>3</sup>
Absorption coefficient	0.277 mm <sup>-1</sup>
F(000)	1260
Crystal size	0.42 x 0.30 x 0.08 mm <sup>3</sup>
Theta range for data collection	2.16 to 25.46°
Index ranges	-11 ≤ h ≤ 11, -20 ≤ k ≤ 20, -22 ≤ l ≤ 22
Reflections collected	54588
Independent reflections	9973 [R(int) = 0.0221]
Completeness to theta = 25.46°	98.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.6979
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	9973 / 0 / 758
Goodness-of-fit on F <sup>2</sup>	1.100
Final R indices [I > 2σ(I)]	R1 = 0.0418, wR2 = 0.1095
R indices (all data)	R1 = 0.0490, wR2 = 0.1152
Largest diff. peak and hole	0.541 and -0.558 e.Å <sup>-3</sup>

Table 2. Refined Positional Parameters for Compound 1342

Atom	x	y	z	$U_{eq}, \text{Å}^2$
N1	0.8166(2)	0.89569(12)	0.38599(11)	0.0409(4)
C2	0.8011(3)	0.93399(16)	0.33918(15)	0.0483(6)
C3	0.7854(3)	1.01934(16)	0.36733(15)	0.0457(5)
C4	0.7898(3)	1.06860(16)	0.44768(15)	0.0457(5)
C5	0.8032(2)	1.02975(15)	0.49690(14)	0.0400(5)
C6	0.8150(2)	0.94320(13)	0.46355(12)	0.0303(4)
C7	0.8264(2)	0.89718(12)	0.51339(11)	0.0285(4)
N8	0.81890(18)	0.94255(11)	0.59020(10)	0.0309(4)
C9	0.8289(2)	0.90249(13)	0.63616(12)	0.0318(4)
C10	0.8515(2)	0.81631(13)	0.60421(12)	0.0313(4)
C11	0.8536(2)	0.77707(13)	0.52360(12)	0.0305(4)
N12	0.84032(18)	0.81383(11)	0.47587(10)	0.0303(3)
N13	0.8165(2)	0.94720(12)	0.71315(10)	0.0425(4)
C14	0.8111(3)	1.03908(15)	0.74971(14)	0.0485(6)
C15	0.6633(4)	1.05108(19)	0.7495(2)	0.0719(9)
C16	0.8730(2)	0.77124(13)	0.65359(11)	0.0309(4)
C17	1.0088(2)	0.76949(14)	0.68087(13)	0.0358(4)
C18	1.0345(2)	0.72719(15)	0.72481(14)	0.0411(5)
C19	0.9155(2)	0.68345(15)	0.74097(14)	0.0399(5)
C20	0.7775(2)	0.68088(17)	0.71585(14)	0.0430(5)
C21	0.7606(2)	0.72562(15)	0.67307(13)	0.0374(5)
Cl1	0.87222(7)	0.66915(4)	0.47631(3)	0.04536(15)
F1	0.5900(4)	1.0181(2)	0.7877(3)	0.187(2)
F2	0.5880(3)	1.0162(2)	0.67598(19)	0.1486(14)
F3	0.6676(3)	1.13676(12)	0.78584(14)	0.0994(7)
F4	1.12268(14)	0.81173(10)	0.66266(10)	0.0559(4)
F5	0.93741(15)	0.64187(11)	0.78470(10)	0.0603(4)
F6	0.62692(14)	0.72591(12)	0.64871(9)	0.0570(4)
N1'	0.54045(18)	0.37955(11)	-0.01543(10)	0.0325(4)
C2'	0.5645(2)	0.32887(14)	-0.08834(12)	0.0378(5)
C3'	0.6854(2)	0.35081(15)	-0.11664(13)	0.0383(5)
C4'	0.7897(2)	0.42990(14)	-0.06701(13)	0.0359(5)
C5'	0.7676(2)	0.48358(13)	0.00865(12)	0.0311(4)
C6'	0.64195(19)	0.45678(12)	0.03224(11)	0.0262(4)
C7'	0.6129(2)	0.51204(12)	0.11282(11)	0.0260(4)
N8'	0.72069(16)	0.58213(10)	0.16479(9)	0.0272(3)
C9'	0.6960(2)	0.63067(12)	0.23855(11)	0.0279(4)
C10'	0.5607(2)	0.61044(13)	0.25908(11)	0.0289(4)
C11'	0.4616(2)	0.53591(13)	0.19943(11)	0.0288(4)
N12'	0.48341(17)	0.48499(10)	0.12654(9)	0.0284(3)
N13'	0.80556(17)	0.69949(11)	0.29317(10)	0.0329(4)
C14'	0.9406(2)	0.72864(15)	0.27441(13)	0.0370(5)
C15'	1.0581(2)	0.69916(16)	0.30206(13)	0.0435(5)
C16'	0.5287(2)	0.66480(13)	0.33895(12)	0.0323(4)
C17'	0.5164(2)	0.63512(15)	0.39572(13)	0.0406(5)
C18'	0.4873(3)	0.68387(18)	0.47030(14)	0.0501(6)
C19'	0.4684(3)	0.76582(18)	0.48741(13)	0.0509(6)
C20'	0.4752(2)	0.80025(16)	0.43472(14)	0.0469(6)
C21'	0.5050(2)	0.74783(14)	0.36081(12)	0.0359(5)
Cl1'	0.29230(5)	0.50227(4)	0.21704(3)	0.03918(13)
F1'	1.08979(16)	0.73367(12)	0.38125(8)	0.0630(4)
F2'	1.0280(2)	0.61123(12)	0.27177(14)	0.0909(7)
F3'	1.18043(15)	0.72622(15)	0.28099(10)	0.0789(6)
F4'	0.53497(17)	0.55384(9)	0.37651(8)	0.0521(3)
F5'	0.43998(19)	0.81555(13)	0.56035(9)	0.0747(5)
F6'	0.50961(14)	0.77812(8)	0.30661(8)	0.0441(3)
N1''	1.00721(19)	0.95184(12)	0.84609(10)	0.0365(4)
C2''	1.1399(3)	0.99464(15)	0.84799(14)	0.0434(5)
C3''	1.2609(3)	1.00298(15)	0.89662(15)	0.0476(6)

C4"	1.2446(2)	0.96462(16)	0.94636(15)	0.0466(5)
C5"	1.1090(2)	0.91914(15)	0.94515(13)	0.0386(5)
C6"	0.9931(2)	0.91395(13)	0.89424(11)	0.0307(4)
C7"	0.8440(2)	0.86536(13)	0.89082(11)	0.0294(4)
N8"	0.83392(17)	0.81398(11)	0.92667(9)	0.0291(3)
C9"	0.7012(2)	0.77097(12)	0.92622(11)	0.0281(4)
C10"	0.5774(2)	0.77924(13)	0.88853(12)	0.0308(4)
C11"	0.6053(2)	0.83274(14)	0.85235(13)	0.0347(4)
N12"	0.73525(18)	0.87725(11)	0.85229(10)	0.0347(4)
N13"	0.68902(17)	0.71877(11)	0.96228(10)	0.0314(4)
C14"	0.8069(2)	0.68915(13)	0.98229(12)	0.0319(4)
C15"	0.8860(2)	0.74706(14)	1.06721(13)	0.0369(5)
C16"	0.4314(2)	0.72922(14)	0.88618(12)	0.0328(4)
C17"	0.3602(2)	0.64716(14)	0.82165(13)	0.0352(4)
C18"	0.2281(2)	0.59612(15)	0.81874(15)	0.0410(5)
C19"	0.1664(2)	0.62981(16)	0.88491(15)	0.0409(5)
C20"	0.2277(2)	0.71073(17)	0.95069(15)	0.0433(5)
C21"	0.3595(2)	0.75839(15)	0.94914(13)	0.0391(5)
Cl1"	0.46135(6)	0.84401(4)	0.80065(4)	0.05298(17)
F1"	0.80103(17)	0.75734(12)	1.12090(8)	0.0637(4)
F2"	0.95687(19)	0.82886(9)	1.08157(9)	0.0651(4)
F3"	0.98544(14)	0.71166(10)	1.08524(8)	0.0482(3)
F4"	0.42598(15)	0.61492(9)	0.75865(8)	0.0518(3)
F5"	0.03932(13)	0.58019(10)	0.88504(10)	0.0543(4)
F6"	0.42356(16)	0.83819(10)	1.01384(9)	0.0596(4)
$U_{eq} = \frac{1}{3}[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha]$				

**Table 3. Positional Parameters for Hydrogens in Compound 1342**

Atom	x	y	z	$U_{iso}, \text{Å}^2$
H2	0.8010	0.9013	0.2849	0.064
H3	0.7722	1.0428	0.3326	0.061
H4	0.7839	1.1272	0.4689	0.061
H5	0.8042	1.0614	0.5514	0.053
H13	0.8116	0.9201	0.7417	0.056
H14a	0.8609	1.0680	0.7217	0.065
H14b	0.8629	1.0691	0.8047	0.065
H18	1.1273	0.7281	0.7426	0.055
H20	0.6989	0.6503	0.7272	0.057
H2'	0.4952	0.2756	-0.1219	0.050
H3'	0.6970	0.3135	-0.1679	0.051
H4'	0.8729	0.4466	-0.0842	0.048
H5'	0.8361	0.5369	0.0432	0.041
H13'	0.7933	0.7271	0.3417	0.044
H14a'	0.9683	0.7933	0.2991	0.049
H14b'	0.9292	0.7057	0.2169	0.049
H18'	0.4807	0.6622	0.5071	0.067
H20'	0.4604	0.8559	0.4480	0.062
H2''	1.1515	1.0205	0.8144	0.058
H3''	1.3514	1.0338	0.8959	0.063
H4''	1.3242	0.9694	0.9803	0.062
H5''	1.0955	0.8923	0.9779	0.051
H13''	0.6072	0.7026	0.9738	0.042
H14a''	0.8739	0.6881	0.9466	0.042
H14b''	0.7703	0.6286	0.9737	0.042
H18''	0.1830	0.5415	0.7742	0.055
H20''	0.1825	0.7325	0.9943	0.058

Table 4. Refined Thermal Parameters (U's) for Compound 1342

Atom	U <sub>11</sub>	U <sub>22</sub>	U <sub>33</sub>	U <sub>23</sub>	U <sub>13</sub>	U <sub>12</sub>
N1	0.0545(11)	0.0365(10)	0.0345(10)	0.0205(8)	0.0055(8)	0.0102(8)
C2	0.0644(16)	0.0455(13)	0.0402(12)	0.0273(11)	0.0064(11)	0.0100(11)
C3	0.0487(13)	0.0497(13)	0.0519(14)	0.0371(12)	0.0050(11)	0.0104(11)
C4	0.0476(13)	0.0404(12)	0.0607(15)	0.0319(12)	0.0091(11)	0.0166(10)
C5	0.0447(12)	0.0376(11)	0.0423(12)	0.0211(10)	0.0075(10)	0.0153(10)
C6	0.0242(9)	0.0309(10)	0.0346(10)	0.0168(9)	0.0009(8)	0.0039(8)
C7	0.0255(9)	0.0273(9)	0.0309(10)	0.0137(8)	0.0012(7)	0.0052(7)
N8	0.0324(9)	0.0285(8)	0.0292(9)	0.0126(7)	-0.0004(7)	0.0078(7)
C9	0.0320(10)	0.0314(10)	0.0286(10)	0.0123(8)	-0.0005(8)	0.0085(8)
C10	0.0314(10)	0.0342(10)	0.0316(10)	0.0178(9)	0.0042(8)	0.0110(8)
C11	0.0311(10)	0.0301(10)	0.0334(10)	0.0157(8)	0.0069(8)	0.0124(8)
N12	0.0324(9)	0.0318(9)	0.0294(8)	0.0158(7)	0.0060(7)	0.0106(7)
N13	0.0652(13)	0.0380(10)	0.0263(9)	0.0140(8)	0.0038(8)	0.0226(9)
C14	0.0677(16)	0.0346(12)	0.0316(11)	0.0073(10)	0.0009(11)	0.0129(11)
C15	0.089(2)	0.0415(15)	0.092(2)	0.0284(16)	0.040(2)	0.0307(15)
C16	0.0356(10)	0.0311(10)	0.0283(10)	0.0140(8)	0.0058(8)	0.0135(8)
C17	0.0323(10)	0.0353(11)	0.0439(12)	0.0236(10)	0.0053(9)	0.0069(8)
C18	0.0321(11)	0.0441(12)	0.0545(14)	0.0306(11)	0.0000(9)	0.0108(9)
C19	0.0390(12)	0.0467(12)	0.0463(13)	0.0332(11)	0.0034(9)	0.0118(10)
C20	0.0350(11)	0.0592(14)	0.0474(13)	0.0361(12)	0.0101(10)	0.0110(10)
C21	0.0308(10)	0.0534(13)	0.0354(11)	0.0246(10)	0.0070(8)	0.0171(9)
Cl1	0.0714(4)	0.0368(3)	0.0401(3)	0.0208(2)	0.0205(3)	0.0291(3)
F1	0.207(3)	0.134(2)	0.356(5)	0.175(3)	0.220(4)	0.116(2)
F2	0.0784(16)	0.142(2)	0.140(2)	-0.0139(19)	-0.0185(15)	0.0613(15)
F3	0.1354(19)	0.0487(10)	0.1261(18)	0.0352(11)	0.0538(15)	0.0527(11)
F4	0.0342(7)	0.0655(9)	0.0889(11)	0.0592(9)	0.0055(7)	0.0039(6)
F5	0.0452(8)	0.0819(11)	0.0852(11)	0.0698(10)	0.0033(7)	0.0121(7)
F6	0.0323(7)	0.1012(12)	0.0652(9)	0.0596(9)	0.0115(6)	0.0250(7)
N1'	0.0316(9)	0.0319(9)	0.0302(9)	0.0114(7)	0.0075(7)	0.0078(7)
C2'	0.0372(11)	0.0343(11)	0.0312(11)	0.0076(9)	0.0065(9)	0.0069(9)
C3'	0.0447(12)	0.0416(12)	0.0293(10)	0.0142(9)	0.0124(9)	0.0174(10)
C4'	0.0356(11)	0.0442(12)	0.0386(11)	0.0254(10)	0.0149(9)	0.0155(9)
C5'	0.0327(10)	0.0311(10)	0.0330(10)	0.0186(9)	0.0039(8)	0.0084(8)
C6'	0.0259(9)	0.0276(9)	0.0272(9)	0.0145(8)	0.0023(7)	0.0089(7)
C7'	0.0274(9)	0.0262(9)	0.0286(9)	0.0161(8)	0.0030(7)	0.0088(7)
N8'	0.0254(8)	0.0284(8)	0.0275(8)	0.0135(7)	0.0040(6)	0.0064(6)
C9'	0.0273(9)	0.0276(9)	0.0284(10)	0.0138(8)	0.0019(7)	0.0066(8)
C10'	0.0267(9)	0.0293(10)	0.0292(10)	0.0132(8)	0.0033(8)	0.0068(8)
C11'	0.0254(9)	0.0311(10)	0.0309(10)	0.0156(8)	0.0062(8)	0.0072(8)
N12'	0.0266(8)	0.0289(8)	0.0285(8)	0.0133(7)	0.0043(6)	0.0060(6)
N13'	0.0271(8)	0.0344(9)	0.0275(8)	0.0085(7)	0.0047(7)	0.0032(7)
C14'	0.0303(10)	0.0402(11)	0.0316(11)	0.0147(9)	0.0031(8)	-0.0017(9)
C15'	0.0289(11)	0.0532(14)	0.0377(12)	0.0138(10)	0.0091(9)	0.0068(10)
C16'	0.0226(9)	0.0362(11)	0.0298(10)	0.0098(9)	0.0035(8)	0.0053(8)
C17'	0.0364(11)	0.0462(13)	0.0334(11)	0.0147(10)	0.0065(9)	0.0094(10)
C18'	0.0453(13)	0.0682(17)	0.0330(12)	0.0204(12)	0.0096(10)	0.0149(12)
C19'	0.0389(12)	0.0681(17)	0.0279(11)	0.0059(11)	0.0082(9)	0.0175(11)
C20'	0.0322(11)	0.0443(13)	0.0449(13)	0.0031(11)	0.0030(9)	0.0154(10)
C21'	0.0252(10)	0.0399(11)	0.0346(11)	0.0119(9)	0.0016(8)	0.0075(8)
Cl1'	0.0274(2)	0.0450(3)	0.0355(3)	0.0140(2)	0.0092(2)	0.0013(2)
F1'	0.0436(8)	0.1033(13)	0.0410(8)	0.0351(8)	0.0047(6)	0.0167(8)
F2'	0.0617(11)	0.0548(10)	0.1255(17)	0.0152(10)	-0.0079(10)	0.0276(8)
F3'	0.0296(7)	0.1422(17)	0.0597(10)	0.0458(11)	0.0177(7)	0.0140(9)
F4'	0.0698(10)	0.0501(8)	0.0431(8)	0.0262(6)	0.0116(7)	0.0190(7)
F5'	0.0735(11)	0.0965(13)	0.0352(8)	0.0062(8)	0.0188(7)	0.0403(10)
F6'	0.0432(7)	0.0393(7)	0.0493(8)	0.0202(6)	0.0055(6)	0.0129(6)
N1''	0.0401(10)	0.0348(9)	0.0347(9)	0.0184(8)	0.0094(7)	0.0047(8)
C2''	0.0457(13)	0.0413(12)	0.0475(13)	0.0260(11)	0.0166(10)	0.0055(10)
C3''	0.0360(12)	0.0396(12)	0.0605(15)	0.0210(11)	0.0142(11)	0.0013(10)



C4"	0.0331(12)	0.0469(13)	0.0547(14)	0.0232(12)	0.0027(10)	0.0047(10)
C5"	0.0343(11)	0.0416(12)	0.0389(12)	0.0206(10)	0.0051(9)	0.0053(9)
C6"	0.0336(10)	0.0282(10)	0.0272(10)	0.0115(8)	0.0062(8)	0.0057(8)
C7"	0.0331(10)	0.0294(10)	0.0237(9)	0.0123(8)	0.0041(8)	0.0056(8)
N8"	0.0275(8)	0.0307(8)	0.0276(8)	0.0144(7)	0.0026(6)	0.0043(7)
C9"	0.0282(9)	0.0292(9)	0.0265(9)	0.0138(8)	0.0050(7)	0.0057(8)
C10"	0.0276(10)	0.0321(10)	0.0324(10)	0.0163(9)	0.0030(8)	0.0059(8)
C11"	0.0309(10)	0.0378(11)	0.0382(11)	0.0214(9)	0.0014(8)	0.0085(8)
N12"	0.0328(9)	0.0375(9)	0.0378(9)	0.0236(8)	0.0028(7)	0.0056(7)
N13"	0.0245(8)	0.0367(9)	0.0382(9)	0.0238(8)	0.0041(7)	0.0048(7)
C14"	0.0313(10)	0.0319(10)	0.0348(11)	0.0189(9)	0.0030(8)	0.0071(8)
C15"	0.0363(11)	0.0415(12)	0.0350(11)	0.0213(10)	0.0028(9)	0.0088(9)
C16"	0.0269(10)	0.0374(11)	0.0408(11)	0.0248(9)	0.0037(8)	0.0082(8)
C17"	0.0307(10)	0.0365(11)	0.0432(12)	0.0217(10)	0.0069(9)	0.0116(9)
C18"	0.0312(11)	0.0351(11)	0.0574(14)	0.0253(11)	-0.0023(10)	0.0059(9)
C19"	0.0227(10)	0.0514(13)	0.0648(15)	0.0423(12)	0.0048(10)	0.0083(9)
C20"	0.0306(11)	0.0604(15)	0.0513(14)	0.0344(12)	0.0129(10)	0.0162(10)
C21"	0.0328(11)	0.0429(12)	0.0411(12)	0.0208(10)	0.0049(9)	0.0078(9)
Cl1"	0.0342(3)	0.0675(4)	0.0771(4)	0.0548(4)	-0.0008(3)	0.0091(3)
F1"	0.0584(9)	0.1033(13)	0.0373(8)	0.0329(8)	0.0143(7)	0.0360(9)
F2"	0.0834(11)	0.0404(8)	0.0500(8)	0.0195(7)	-0.0239(8)	-0.0087(7)
F3"	0.0397(7)	0.0668(9)	0.0466(8)	0.0345(7)	0.0000(6)	0.0164(6)
F4"	0.0457(8)	0.0454(8)	0.0503(8)	0.0124(6)	0.0131(6)	0.0069(6)
F5"	0.0252(6)	0.0685(9)	0.0866(11)	0.0560(9)	0.0058(6)	0.0040(6)
F6"	0.0484(8)	0.0604(9)	0.0468(8)	0.0102(7)	0.0132(6)	0.0023(7)

The form of the anisotropic displacement parameter is:

$$\exp[-2\pi(a^*{}^2U_{11}h^2+b^*{}^2U_{22}k^2+c^*{}^2U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$$

**Table 5. Bond Distances in Compound 1342, Å**

N1-C6	1.334(3)	N1-C2	1.342(3)	C2-C3	1.382(3)
C3-C4	1.370(4)	C4-C5	1.387(3)	C5-C6	1.383(3)
C6-C7	1.497(3)	C7-N8	1.331(3)	C7-N12	1.342(2)
N8-C9	1.345(3)	C9-N13	1.346(3)	C9-C10	1.417(3)
C10-C11	1.375(3)	C10-C16	1.492(3)	C11-N12	1.333(3)
C11-C11	1.7387(19)	N13-C14	1.445(3)	C14-C15	1.487(4)
C15-F1	1.277(4)	C15-F3	1.323(3)	C15-F2	1.327(4)
C16-C21	1.381(3)	C16-C17	1.387(3)	C17-F4	1.347(2)
C17-C18	1.374(3)	C18-C19	1.375(3)	C19-F5	1.351(2)
C19-C20	1.369(3)	C20-C21	1.373(3)	C21-F6	1.342(2)
N1'-C2'	1.340(3)	N1'-C6'	1.349(2)	C2'-C3'	1.375(3)
C3'-C4'	1.386(3)	C4'-C5'	1.385(3)	C5'-C6'	1.387(3)
C6'-C7'	1.488(3)	C7'-N12'	1.330(2)	C7'-N8'	1.338(2)
N8'-C9'	1.347(2)	C9'-N13'	1.354(2)	C9'-C10'	1.412(3)
C10'-C11'	1.376(3)	C10'-C16'	1.486(3)	C11'-N12'	1.333(2)
C11'-C11'	1.7297(19)	N13'-C14'	1.429(3)	C14'-C15'	1.491(3)
C15'-F2'	1.326(3)	C15'-F1'	1.329(3)	C15'-F3'	1.337(3)
C16'-C21'	1.387(3)	C16'-C17'	1.389(3)	C17'-F4'	1.348(3)
C17'-C18'	1.377(3)	C18'-C19'	1.368(4)	C19'-F5'	1.354(3)
C19'-C20'	1.374(4)	C20'-C21'	1.385(3)	C21'-F6'	1.348(3)
N1''-C2''	1.331(3)	N1''-C6''	1.343(3)	C2''-C3''	1.375(4)
C3''-C4''	1.378(4)	C4''-C5''	1.375(3)	C5''-C6''	1.385(3)
C6''-C7''	1.492(3)	C7''-N8''	1.335(2)	C7''-N12''	1.340(3)
N8''-C9''	1.341(2)	C9''-N13''	1.350(2)	C9''-C10''	1.415(3)
C10''-C11''	1.378(3)	C10''-C16''	1.482(3)	C11''-N12''	1.331(3)
C11''-C11''	1.738(2)	N13''-C14''	1.436(2)	C14''-C15''	1.500(3)
C15''-F1''	1.331(3)	C15''-F2''	1.336(3)	C15''-F3''	1.343(2)
C16''-C21''	1.379(3)	C16''-C17''	1.385(3)	C17''-F4''	1.348(3)
C17''-C18''	1.376(3)	C18''-C19''	1.372(3)	C19''-F5''	1.347(2)
C19''-C20''	1.367(4)	C20''-C21''	1.378(3)	C21''-F6''	1.352(3)

Table 6. Bond Angles in Compound 1342, °

C6-N1-C2	117.37(19)	N1-C2-C3	123.5(2)	C4-C3-C2	118.4(2)
C3-C4-C5	119.0(2)	C6-C5-C4	118.9(2)	N1-C6-C5	122.75(19)
N1-C6-C7	115.98(17)	C5-C6-C7	121.26(18)	N8-C7-N12	126.60(17)
N8-C7-C6	116.55(16)	N12-C7-C6	116.83(17)	C7-N8-C9	117.60(16)
N8-C9-N13	117.58(18)	N8-C9-C10	120.90(18)	N13-C9-C10	121.52(18)
C11-C10-C9	114.74(17)	C11-C10-C16	122.08(17)	C9-C10-C16	123.17(17)
N12-C11-C10	125.87(18)	N12-C11-Cl1	114.96(15)	C10-C11-Cl1	119.16(15)
C11-N12-C7	114.16(16)	C9-N13-C14	122.52(18)	N13-C14-C15	114.7(2)
F1-C15-F3	104.7(3)	F1-C15-F2	107.9(4)	F3-C15-F2	106.9(3)
F1-C15-C14	113.6(3)	F3-C15-C14	110.9(3)	F2-C15-C14	112.3(3)
C21-C16-C17	114.99(18)	C21-C16-C10	123.13(18)	C17-C16-C10	121.84(18)
F4-C17-C18	118.02(18)	F4-C17-C16	117.70(18)	C18-C17-C16	124.28(19)
C17-C18-C19	116.35(19)	F5-C19-C20	118.84(19)	F5-C19-C18	117.74(19)
C20-C19-C18	123.42(19)	C19-C20-C21	116.8(2)	F6-C21-C20	118.46(19)
F6-C21-C16	117.40(18)	C20-C21-C16	124.14(19)	C2'-N1'-C6'	117.15(17)
N1'-C2'-C3'	124.0(2)	C2'-C3'-C4'	118.42(19)	C5'-C4'-C3'	118.78(19)
C4'-C5'-C6'	119.08(19)	N1'-C6'-C5'	122.53(17)	N1'-C6'-C7'	116.24(16)
C5'-C6'-C7'	121.22(17)	N12'-C7'-N8'	126.66(17)	N12'-C7'-C6'	116.40(16)
N8'-C7'-C6'	116.92(16)	C7'-N8'-C9'	116.98(16)	N8'-C9'-N13'	117.98(17)
N8'-C9'-C10'	121.43(17)	N13'-C9'-C10'	120.59(17)	C11'-C10'-C9'	114.54(17)
C11'-C10'-C16'	122.84(17)	C9'-C10'-C16'	122.61(17)	N12'-C11'-C10'	125.65(18)
N12'-C11'-Cl1'	114.90(14)	C10'-C11'-Cl1'	119.45(15)	C7'-N12'-C11'	114.66(16)
C9'-N13'-C14'	123.39(17)	N13'-C14'-C15'	113.16(18)	F2'-C15'-F1'	107.0(2)
F2'-C15'-F3'	107.6(2)	F1'-C15'-F3'	105.22(18)	F2'-C15'-C14'	112.83(19)
F1'-C15'-C14'	113.27(19)	F3'-C15'-C14'	110.5(2)	C21'-C16'-C17'	115.58(19)
C21'-C16'-C10'	121.95(19)	C17'-C16'-C10'	122.46(19)	F4'-C17'-C18'	118.3(2)
F4'-C17'-C16'	117.79(18)	C18'-C17'-C16'	123.9(2)	C19'-C18'-C17'	116.6(2)
F5'-C19'-C18'	118.0(2)	F5'-C19'-C20'	118.1(2)	C18'-C19'-C20'	123.9(2)
C19'-C20'-C21'	116.5(2)	F6'-C21'-C20'	118.6(2)	F6'-C21'-C16'	117.97(18)
C20'-C21'-C16'	123.4(2)	C2''-N1''-C6''	117.05(19)	N1''-C2''-C3''	124.0(2)
C2''-C3''-C4''	118.4(2)	C5''-C4''-C3''	119.0(2)	C4''-C5''-C6''	118.8(2)
N1''-C6''-C5''	122.76(19)	N1''-C6''-C7''	116.48(17)	C5''-C6''-C7''	120.76(18)
N8''-C7''-N12''	127.00(18)	N8''-C7''-C6''	115.78(17)	N12''-C7''-C6''	117.23(17)
C7''-N8''-C9''	117.24(16)	N8''-C9''-N13''	118.03(17)	N8''-C9''-C10''	121.14(17)
N13''-C9''-C10''	120.82(17)	C11''-C10''-C9''	114.82(17)	C11''-C10''-C16''	124.39(18)
C9''-C10''-C16''	120.72(17)	N12''-C11''-C10''	125.76(18)	N12''-C11''-Cl1''	115.47(15)
C10''-C11''-Cl1''	118.77(16)	C11''-N12''-C7''	114.01(17)	C9''-N13''-C14''	122.78(16)
N13''-C14''-C15''	113.23(17)	F1''-C15''-F2''	106.78(19)	F1''-C15''-F3''	106.05(17)
F2''-C15''-F3''	106.12(17)	F1''-C15''-C14''	113.45(18)	F2''-C15''-C14''	113.06(17)
F3''-C15''-C14''	110.85(18)	C21''-C16''-C17''	115.15(19)	C21''-C16''-C10''	122.79(19)
C17''-C16''-C10''	121.98(19)	F4''-C17''-C18''	118.3(2)	F4''-C17''-C16''	117.73(18)
C18''-C17''-C16''	124.0(2)	C19''-C18''-C17''	116.6(2)	F5''-C19''-C20''	118.5(2)
F5''-C19''-C18''	118.1(2)	C20''-C19''-C18''	123.4(2)	C19''-C20''-C21''	116.7(2)
F6''-C21''-C20''	118.0(2)	F6''-C21''-C16''	117.88(19)	C20''-C21''-C16''	124.1(2)

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<sup>i</sup> Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

<sup>ii</sup> Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

<sup>iii</sup> Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

<sup>iv</sup> Sheldrick, G.M. (2008) *Acta Cryst.* A64,112-122.

<sup>v</sup> v.d. Sluis, P. & A.L. Spek (1990). *Acta. Cryst.*, **A46**, 194.

<sup>vi</sup> Sheldrick, G.M. (2008) *Acta Cryst.* A64,112-122.

<sup>vii</sup>  $R1 = \sum |F_o| - |F_c| / \sum |F_o|$

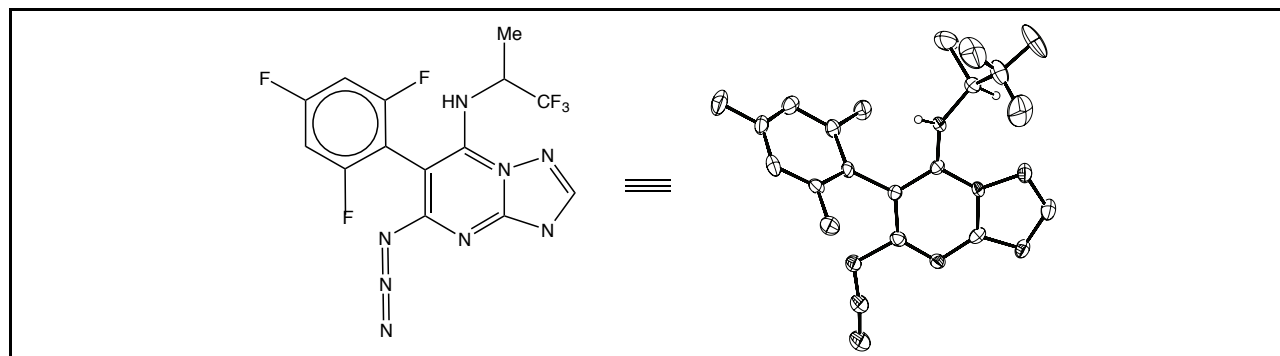
$wR2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{1/2}$

$GOF = [\sum w(F_o^2 - F_c^2)^2 / (n - p)]^{1/2}$

where n = the number of reflections and p = the number of parameters refined.

<sup>viii</sup> "ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.

### X-ray Structure Determination of Compound 36 (CCDC 992820)



Compound 1384,  $C_{14}H_8N_8F_6$ , crystallizes in the monoclinic space group  $P2_1$  (systematic absences  $0k0: k=\text{odd}$ ) with  $a=12.7869(7)\text{\AA}$ ,  $b=9.6365(5)\text{\AA}$ ,  $c=13.9992(7)\text{\AA}$ ,  $\beta=107.054(2)^\circ$ ,  $V=1649.14(15)\text{\AA}^3$ ,  $Z=4$ , and  $d_{\text{calc}}=1.620\text{ g/cm}^3$ . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo- $K\alpha$  radiation ( $\lambda=0.71073\text{ \AA}$ ) at a temperature of  $100(1)\text{K}$ . Preliminary indexing was performed from a series of thirty-six  $0.5^\circ$  rotation frames with exposures of 10 seconds. A total of 1498 frames were collected with a crystal to detector distance of 37.4 mm, rotation widths of  $0.5^\circ$  and exposures of 10 seconds:

scan type	$2\theta$	$\omega$	$\phi$	$\chi$	frames
$\phi$	19.50	59.55	348.71	-26.26	739
$\omega$	14.50	283.75	54.11	21.36	197
$\phi$	-23.00	334.21	158.53	73.66	496
$\omega$	-8.00	320.74	277.32	84.61	66

Rotation frames were integrated using SAINT<sup>i</sup>, producing a listing of unaveraged  $F^2$  and  $\sigma(F^2)$  values which were then passed to the SHELXTL<sup>ii</sup> program package for further processing and structure solution. A total of 19505 reflections were measured over the ranges  $1.67 \leq \theta \leq 25.40^\circ$ ,  $-15 \leq h \leq 15$ ,  $-11 \leq k \leq 11$ ,  $-16 \leq l \leq 16$  yielding 5913 unique reflections ( $R_{\text{int}} = 0.0380$ ). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS<sup>iii</sup> (minimum and maximum transmission 0.5973, 0.7452).

The structure was solved by direct methods (SHELXS-97<sup>iv</sup>). The asymmetric unit consists of two

molecules of the title compound. Refinement was by full-matrix least squares based on  $F^2$  using SHELXL-97.<sup>v</sup> All reflections were used during refinement. The weighting scheme used was  $w=1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.2595P]$  where  $P = (F_o^2 + 2F_c^2)/3$ . Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to  $R1=0.0426$  and  $wR2=0.1027$  for 4714 observed reflections for which  $F > 4\sigma(F)$  and  $R1=0.0619$  and  $wR2=0.1129$  and  $GOF = 1.035$  for all 5913 unique, non-zero reflections and 508 variables.<sup>vi</sup> The maximum  $\Delta/\sigma$  in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were  $+0.262$  and  $-0.269 \text{ e}/\text{\AA}^3$ .

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figures 1. and 2. are ORTEP<sup>vii</sup> representations of the molecule with 50% probability thermal ellipsoids displayed.

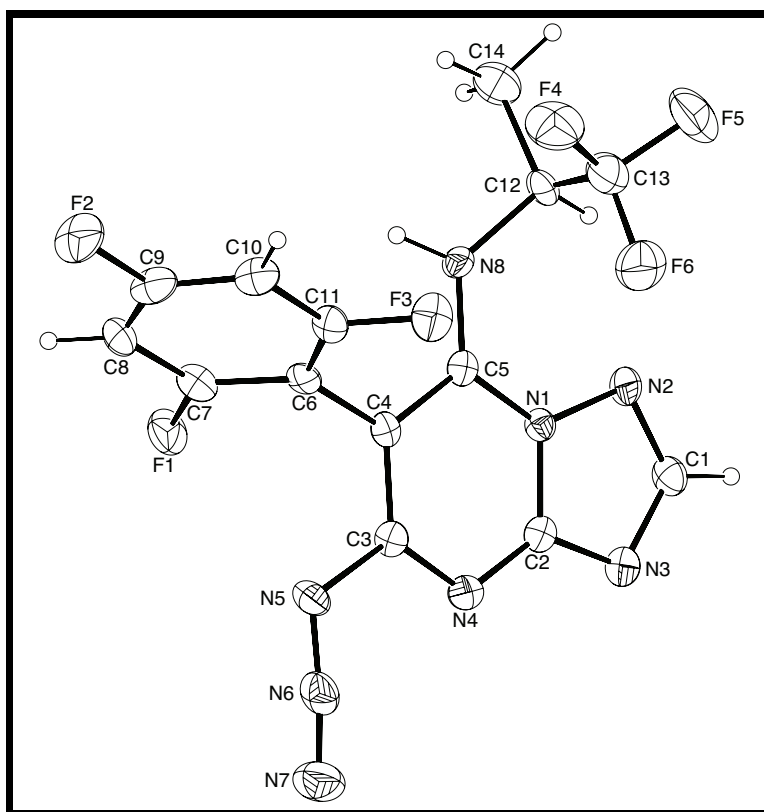


Figure 1. ORTEP drawing of molecule no. 1 of the asymmetric unit with 50% probability thermal ellipsoids.

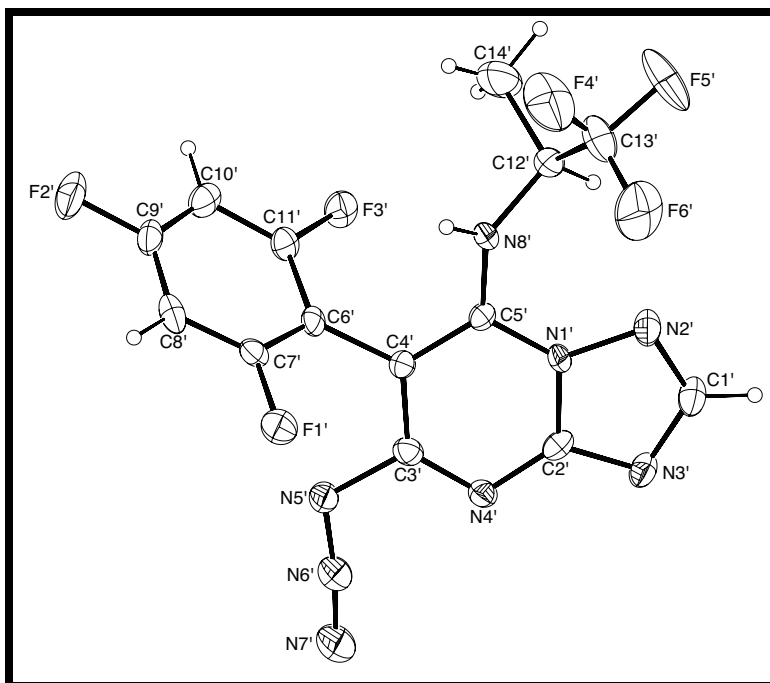


Figure 2. ORTEP drawing of molecule no. 2 of the asymmetric unit with 50% probability thermal ellipsoids.

**Table 1. Summary of Structure Determination of Compound 1384**

Empirical formula	C <sub>14</sub> H <sub>8</sub> N <sub>8</sub> F <sub>6</sub>
Formula weight	402.28
Temperature	100(1) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	P2 <sub>1</sub>
Cell constants:	
a	12.7869(7) Å
b	9.6365(5) Å
c	13.9992(7) Å
β	107.054(2)°
Volume	1649.14(15) Å <sup>3</sup>
Z	4
Density (calculated)	1.620 Mg/m <sup>3</sup>
Absorption coefficient	0.153 mm <sup>-1</sup>
F(000)	808
Crystal size	0.32 x 0.18 x 0.04 mm <sup>3</sup>
Theta range for data collection	1.67 to 25.40°
Index ranges	-15 ≤ h ≤ 15, -11 ≤ k ≤ 11, -16 ≤ l ≤ 16
Reflections collected	19505
Independent reflections	5913 [R(int) = 0.0380]
Completeness to theta = 25.40°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.5973
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	5913 / 1 / 508
Goodness-of-fit on F <sup>2</sup>	1.035
Final R indices [I > 2σ(I)]	R1 = 0.0426, wR2 = 0.1027
R indices (all data)	R1 = 0.0619, wR2 = 0.1129
Absolute structure parameter	0.5(7)
Largest diff. peak and hole	0.262 and -0.269 e.Å <sup>-3</sup>



Table 2. Refined Positional Parameters for Compound 1384

Atom	x	y	z	$U_{eq}, \text{Å}^2$
C1	0.0765(3)	0.4464(4)	0.2853(2)	0.0230(8)
C2	0.1868(2)	0.5887(3)	0.2565(2)	0.0182(7)
C3	0.2952(2)	0.6980(3)	0.1796(2)	0.0179(7)
C4	0.2702(2)	0.6051(3)	0.0995(2)	0.0165(7)
C5	0.1957(2)	0.4974(3)	0.0976(2)	0.0172(7)
C6	0.3269(2)	0.6137(3)	0.0211(2)	0.0188(7)
C7	0.3141(3)	0.7241(3)	-0.0449(2)	0.0246(8)
C8	0.3675(3)	0.7346(4)	-0.1177(2)	0.0284(8)
C9	0.4370(3)	0.6283(4)	-0.1217(2)	0.0298(9)
C10	0.4546(3)	0.5149(4)	-0.0607(2)	0.0268(8)
C11	0.3987(2)	0.5110(4)	0.0102(2)	0.0213(7)
C12	0.1105(3)	0.2734(3)	0.0166(2)	0.0234(7)
C13	0.2001(3)	0.1676(4)	0.0588(3)	0.0359(8)
C14	0.0466(3)	0.2371(4)	-0.0899(3)	0.0423(10)
N1	0.15816(19)	0.4919(3)	0.17972(18)	0.0169(6)
N2	0.0833(2)	0.3983(3)	0.19832(18)	0.0221(6)
N3	0.1365(2)	0.5593(3)	0.32387(19)	0.0218(6)
N4	0.2546(2)	0.6937(3)	0.25719(18)	0.0202(6)
N5	0.3724(2)	0.8056(3)	0.1786(2)	0.0234(6)
N6	0.3828(2)	0.8874(3)	0.2487(2)	0.0288(7)
N7	0.3985(3)	0.9690(4)	0.3093(2)	0.0393(8)
N8	0.15876(19)	0.4106(3)	0.01930(18)	0.0194(6)
F1	0.24295(17)	0.8246(2)	-0.03960(14)	0.0331(5)
F2	0.48918(16)	0.6347(3)	-0.19400(15)	0.0417(6)
F3	0.41485(15)	0.4020(2)	0.07159(13)	0.0302(5)
F4	0.26574(18)	0.1488(2)	0.00208(17)	0.0472(6)
F5	0.1568(2)	0.0436(2)	0.06882(19)	0.0582(7)
F6	0.26334(18)	0.2044(2)	0.14953(14)	0.0459(6)
C1'	0.0883(3)	0.6406(4)	0.7847(2)	0.0270(8)
C2'	0.1902(2)	0.4855(4)	0.7540(2)	0.0188(7)
C3'	0.2911(3)	0.3674(3)	0.6766(2)	0.0202(7)
C4'	0.2747(2)	0.4645(3)	0.5985(2)	0.0180(7)
C5'	0.2084(2)	0.5784(3)	0.5995(2)	0.0162(7)
C6'	0.3314(2)	0.4521(3)	0.5195(2)	0.0181(7)
C7'	0.3120(2)	0.3454(3)	0.4497(2)	0.0198(7)
C8'	0.3634(3)	0.3329(4)	0.3773(2)	0.0257(8)
C9'	0.4371(3)	0.4357(4)	0.3739(2)	0.0254(8)
C10'	0.4604(3)	0.5463(4)	0.4382(2)	0.0264(8)
C11'	0.4068(2)	0.5507(4)	0.5099(2)	0.0212(7)
C12'	0.1573(3)	0.8188(4)	0.5324(2)	0.0248(7)
C13'	0.0452(3)	0.8545(4)	0.4686(3)	0.0410(9)
C14'	0.2423(3)	0.9067(5)	0.5026(4)	0.0536(12)
N1'	0.1705(2)	0.5885(3)	0.68155(19)	0.0179(6)
N2'	0.1026(2)	0.6878(3)	0.70146(19)	0.0252(6)
N3'	0.1379(2)	0.5188(3)	0.82068(19)	0.0236(7)
N4'	0.2500(2)	0.3731(3)	0.75252(18)	0.0196(6)
N5'	0.3627(2)	0.2537(3)	0.6741(2)	0.0263(7)
N6'	0.3721(2)	0.1729(3)	0.7448(2)	0.0357(8)
N7'	0.3874(3)	0.0924(4)	0.8050(3)	0.0514(10)
N8'	0.1796(2)	0.6720(3)	0.52512(18)	0.0178(6)
F1'	0.23763(16)	0.2482(2)	0.45463(14)	0.0324(5)
F2'	0.48880(16)	0.4290(3)	0.30222(14)	0.0417(6)
F3'	0.42695(14)	0.6581(2)	0.57457(13)	0.0275(5)
F4'	0.0317(2)	0.8447(3)	0.37181(15)	0.0646(8)
F5'	0.0202(2)	0.9851(3)	0.4865(2)	0.0711(8)
F6'	-0.03139(16)	0.7729(3)	0.48670(19)	0.0630(7)

$U_{eq} = \frac{1}{3}[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha]$

**Table 3. Positional Parameters for Hydrogens in Compound 1384**

Atom	x	y	z	$U_{iso}, \text{Å}^2$
H1	0.0318	0.4038	0.3184	0.031
H8	0.3567	0.8097	-0.1612	0.038
H10	0.5017	0.4440	-0.0665	0.036
H12	0.0605	0.2740	0.0578	0.031
H14a	-0.0150	0.2983	-0.1123	0.063
H14b	0.0214	0.1430	-0.0925	0.063
H14c	0.0930	0.2472	-0.1324	0.063
H8a	0.1645	0.4406	-0.0368	0.026
H1'	0.0460	0.6885	0.8177	0.036
H8'	0.3493	0.2590	0.3326	0.034
H10'	0.5100	0.6147	0.4335	0.035
H12'	0.1619	0.8399	0.6020	0.033
H14a'	0.3119	0.8977	0.5524	0.080
H14b'	0.2200	1.0023	0.4974	0.080
H14c'	0.2483	0.8755	0.4393	0.080
H8a'	0.1737	0.6412	0.4661	0.024

**Table 4. Refined Thermal Parameters (U's) for Compound 1384**

Atom	U <sub>11</sub>	U <sub>22</sub>	U <sub>33</sub>	U <sub>23</sub>	U <sub>13</sub>	U <sub>12</sub>
C1	0.0214(17)	0.0227(19)	0.0278(18)	0.0010(15)	0.0119(14)	-0.0027(14)
C2	0.0159(16)	0.0201(18)	0.0191(16)	0.0026(14)	0.0057(13)	0.0042(14)
C3	0.0117(15)	0.0220(18)	0.0204(15)	0.0011(15)	0.0052(12)	-0.0009(13)
C4	0.0122(15)	0.0164(17)	0.0197(16)	0.0037(14)	0.0027(12)	0.0014(13)
C5	0.0153(15)	0.0184(17)	0.0185(16)	0.0030(14)	0.0057(12)	0.0034(13)
C6	0.0146(15)	0.0207(18)	0.0213(16)	-0.0039(15)	0.0059(13)	-0.0091(14)
C7	0.0216(18)	0.020(2)	0.0321(19)	-0.0028(16)	0.0083(15)	-0.0014(15)
C8	0.034(2)	0.022(2)	0.0281(18)	0.0007(16)	0.0081(16)	-0.0130(16)
C9	0.0241(18)	0.045(2)	0.0253(18)	-0.0100(18)	0.0142(15)	-0.0121(17)
C10	0.0209(17)	0.034(2)	0.0271(18)	-0.0057(17)	0.0095(14)	-0.0016(15)
C11	0.0150(16)	0.024(2)	0.0251(17)	0.0004(15)	0.0063(13)	-0.0041(14)
C12	0.0268(17)	0.0185(17)	0.0273(16)	-0.0012(13)	0.0116(14)	-0.0105(15)
C13	0.050(2)	0.025(2)	0.038(2)	-0.0036(16)	0.0210(18)	-0.0033(17)
C14	0.044(2)	0.039(2)	0.040(2)	-0.0056(19)	0.0064(18)	-0.0224(19)
N1	0.0164(13)	0.0170(14)	0.0189(13)	0.0006(11)	0.0075(10)	-0.0001(11)
N2	0.0215(13)	0.0247(16)	0.0235(13)	0.0018(12)	0.0118(11)	-0.0066(12)
N3	0.0195(14)	0.0238(17)	0.0234(14)	0.0029(13)	0.0081(12)	0.0024(12)
N4	0.0192(13)	0.0207(16)	0.0202(13)	-0.0003(12)	0.0047(11)	0.0022(12)
N5	0.0260(15)	0.0170(16)	0.0301(15)	-0.0038(13)	0.0125(12)	-0.0034(12)
N6	0.0236(15)	0.0280(18)	0.0347(17)	0.0018(16)	0.0083(13)	-0.0086(13)
N7	0.0391(18)	0.038(2)	0.0400(18)	-0.0115(17)	0.0108(15)	-0.0125(16)
N8	0.0220(14)	0.0223(15)	0.0153(13)	-0.0014(11)	0.0077(10)	-0.0053(12)
F1	0.0430(12)	0.0205(11)	0.0389(11)	0.0042(9)	0.0169(9)	0.0064(10)
F2	0.0412(12)	0.0572(16)	0.0369(12)	-0.0049(11)	0.0272(10)	-0.0111(11)
F3	0.0268(10)	0.0318(12)	0.0342(10)	0.0065(9)	0.0122(8)	0.0100(9)
F4	0.0509(13)	0.0418(14)	0.0562(14)	-0.0121(12)	0.0271(11)	0.0053(11)
F5	0.0875(18)	0.0226(12)	0.0716(16)	-0.0001(11)	0.0343(14)	-0.0064(12)
F6	0.0558(14)	0.0394(13)	0.0360(11)	0.0045(10)	0.0033(10)	0.0126(11)
C1'	0.0250(17)	0.037(2)	0.0215(17)	-0.0015(16)	0.0101(14)	0.0063(17)
C2'	0.0151(15)	0.0257(19)	0.0150(15)	0.0000(15)	0.0034(13)	-0.0050(15)
C3'	0.0191(16)	0.0164(18)	0.0247(17)	-0.0003(15)	0.0058(13)	-0.0013(14)
C4'	0.0155(15)	0.0179(19)	0.0214(16)	0.0007(14)	0.0070(13)	0.0005(14)
C5'	0.0116(15)	0.0189(18)	0.0181(16)	-0.0001(13)	0.0043(12)	-0.0019(13)
C6'	0.0149(15)	0.0217(19)	0.0178(15)	0.0042(14)	0.0048(12)	0.0085(13)
C7'	0.0181(16)	0.0150(18)	0.0252(16)	0.0011(15)	0.0048(13)	0.0028(14)
C8'	0.0269(18)	0.027(2)	0.0214(16)	-0.0049(15)	0.0046(14)	0.0113(16)
C9'	0.0232(17)	0.033(2)	0.0230(18)	0.0063(16)	0.0117(14)	0.0118(16)
C10'	0.0155(16)	0.035(2)	0.0292(18)	0.0084(17)	0.0072(14)	0.0039(15)
C11'	0.0141(15)	0.0240(19)	0.0245(17)	-0.0002(15)	0.0040(13)	0.0030(14)
C12'	0.0240(17)	0.0218(18)	0.0311(17)	0.0062(15)	0.0119(14)	0.0047(14)
C13'	0.034(2)	0.038(2)	0.050(2)	0.0000(19)	0.0106(17)	0.0175(18)
C14'	0.047(2)	0.027(2)	0.095(3)	0.009(2)	0.035(2)	-0.0015(19)
N1'	0.0169(13)	0.0170(15)	0.0208(13)	0.0020(11)	0.0071(11)	0.0061(11)
N2'	0.0245(15)	0.0299(17)	0.0233(14)	-0.0003(13)	0.0101(11)	0.0073(13)
N3'	0.0227(15)	0.0317(18)	0.0190(14)	0.0008(13)	0.0100(12)	0.0025(12)
N4'	0.0180(13)	0.0169(16)	0.0241(14)	0.0022(12)	0.0065(11)	0.0015(11)
N5'	0.0288(15)	0.0272(17)	0.0264(15)	0.0095(13)	0.0134(12)	0.0109(13)
N6'	0.0394(18)	0.0294(19)	0.0436(19)	0.0103(17)	0.0203(15)	0.0123(15)
N7'	0.062(2)	0.040(2)	0.061(2)	0.024(2)	0.034(2)	0.0269(19)
N8'	0.0193(13)	0.0147(14)	0.0201(13)	0.0004(12)	0.0071(10)	0.0038(11)
F1'	0.0374(11)	0.0230(12)	0.0396(11)	-0.0058(9)	0.0155(9)	-0.0046(9)
F2'	0.0411(12)	0.0580(16)	0.0349(12)	0.0002(11)	0.0249(10)	0.0085(11)
F3'	0.0198(9)	0.0282(12)	0.0352(10)	-0.0053(9)	0.0091(8)	-0.0053(8)
F4'	0.0749(18)	0.0729(18)	0.0357(13)	0.0083(12)	0.0001(12)	0.0352(15)
F5'	0.0649(17)	0.0479(16)	0.0900(19)	-0.0040(14)	0.0063(14)	0.0420(14)
F6'	0.0211(12)	0.0843(19)	0.0816(17)	0.0049(14)	0.0119(11)	0.0061(12)

The form of the anisotropic displacement parameter is:

$$\exp[-2\pi(a^*U_{11}h^2+b^*U_{22}k^2+c^*U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$$

**Table 5. Bond Distances in Compound 1384, Å**

C1-N2	1.329(4)	C1-N3	1.350(4)	C2-N3	1.319(4)
C2-N4	1.330(4)	C2-N1	1.389(4)	C3-N4	1.335(4)
C3-C4	1.397(4)	C3-N5	1.434(4)	C4-C5	1.404(4)
C4-C6	1.484(4)	C5-N8	1.349(4)	C5-N1	1.371(4)
C6-C7	1.387(5)	C6-C11	1.389(5)	C7-F1	1.345(4)
C7-C8	1.386(5)	C8-C9	1.369(5)	C9-C10	1.364(5)
C9-F2	1.367(3)	C10-C11	1.384(4)	C11-F3	1.334(4)
C12-N8	1.455(4)	C12-C14	1.516(5)	C12-C13	1.518(5)
C13-F4	1.326(4)	C13-F6	1.338(4)	C13-F5	1.342(4)
N1-N2	1.394(4)	N5-N6	1.236(4)	N6-N7	1.131(4)
C1'-N2'	1.313(4)	C1'-N3'	1.359(5)	C2'-N4'	1.331(4)
C2'-N3'	1.336(4)	C2'-N1'	1.388(4)	C3'-N4'	1.318(4)
C3'-C4'	1.407(4)	C3'-N5'	1.435(4)	C4'-C5'	1.389(4)
C4'-C6'	1.494(4)	C5'-N8'	1.345(4)	C5'-N1'	1.375(4)
C6'-C11'	1.388(5)	C6'-C7'	1.389(4)	C7'-F1'	1.352(4)
C7'-C8'	1.366(4)	C8'-C9'	1.378(5)	C9'-F2'	1.355(3)
C9'-C10'	1.370(5)	C10'-C11'	1.372(4)	C11'-F3'	1.350(4)
C12'-N8'	1.453(4)	C12'-C13'	1.489(5)	C12'-C14'	1.529(5)
C13'-F4'	1.318(4)	C13'-F6'	1.336(5)	C13'-F5'	1.341(4)
N1'-N2'	1.375(4)	N5'-N6'	1.236(4)	N6'-N7'	1.120(4)

**Table 6. Bond Angles in Compound 1384, °**

N2-C1-N3	117.7(3)	N3-C2-N4	127.7(3)	N3-C2-N1	109.6(3)
N4-C2-N1	122.7(3)	N4-C3-C4	125.5(3)	N4-C3-N5	117.6(3)
C4-C3-N5	116.8(3)	C3-C4-C5	118.9(3)	C3-C4-C6	120.7(3)
C5-C4-C6	120.2(3)	N8-C5-N1	122.2(3)	N8-C5-C4	123.2(3)
N1-C5-C4	114.5(3)	C7-C6-C11	115.2(3)	C7-C6-C4	123.2(3)
C11-C6-C4	121.5(3)	F1-C7-C8	118.3(3)	F1-C7-C6	117.7(3)
C8-C7-C6	124.0(3)	C9-C8-C7	116.2(3)	C10-C9-F2	118.2(3)
C10-C9-C8	124.3(3)	F2-C9-C8	117.4(3)	C9-C10-C11	116.5(3)
F3-C11-C10	118.0(3)	F3-C11-C6	118.2(3)	C10-C11-C6	123.8(3)
N8-C12-C14	109.7(3)	N8-C12-C13	109.6(3)	C14-C12-C13	110.8(3)
F4-C13-F6	107.0(3)	F4-C13-F5	107.1(3)	F6-C13-F5	106.8(3)
F4-C13-C12	113.3(3)	F6-C13-C12	111.6(3)	F5-C13-C12	110.6(3)
C5-N1-C2	123.0(3)	C5-N1-N2	127.6(3)	C2-N1-N2	109.3(2)
C1-N2-N1	100.3(3)	C2-N3-C1	103.1(3)	C2-N4-C3	115.2(3)
N6-N5-C3	111.5(3)	N7-N6-N5	173.7(3)	C5-N8-C12	129.0(3)
N2'-C1'-N3'	117.2(3)	N4'-C2'-N3'	127.9(3)	N4'-C2'-N1'	123.5(3)
N3'-C2'-N1'	108.6(3)	N4'-C3'-C4'	126.5(3)	N4'-C3'-N5'	117.3(3)
C4'-C3'-N5'	116.1(3)	C5'-C4'-C3'	118.0(3)	C5'-C4'-C6'	120.0(3)
C3'-C4'-C6'	121.8(3)	N8'-C5'-N1'	121.0(3)	N8'-C5'-C4'	123.5(3)
N1'-C5'-C4'	115.4(3)	C11'-C6'-C7'	115.0(3)	C11'-C6'-C4'	121.3(3)
C7'-C6'-C4'	123.7(3)	F1'-C7'-C8'	118.4(3)	F1'-C7'-C6'	117.4(3)
C8'-C7'-C6'	124.2(3)	C7'-C8'-C9'	116.4(3)	F2'-C9'-C10'	117.6(3)
F2'-C9'-C8'	118.6(3)	C10'-C9'-C8'	123.8(3)	C9'-C10'-C11'	116.4(3)
F3'-C11'-C10'	118.4(3)	F3'-C11'-C6'	117.5(3)	C10'-C11'-C6'	124.1(3)
N8'-C12'-C13'	110.9(3)	N8'-C12'-C14'	110.5(3)	C13'-C12'-C14'	110.2(3)
F4'-C13'-F6'	105.7(3)	F4'-C13'-F5'	107.1(3)	F6'-C13'-F5'	106.8(3)
F4'-C13'-C12'	114.3(3)	F6'-C13'-C12'	112.4(3)	F5'-C13'-C12'	110.2(3)
N2'-N1'-C5'	128.1(3)	N2'-N1'-C2'	109.8(2)	C5'-N1'-C2'	121.8(3)
C1'-N2'-N1'	101.5(3)	C2'-N3'-C1'	102.9(3)	C3'-N4'-C2'	114.4(3)
N6'-N5'-C3'	112.0(3)	N7'-N6'-N5'	173.1(4)	C5'-N8'-C12'	127.9(3)

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<sup>i</sup>Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

<sup>ii</sup>Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

<sup>iii</sup>Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

<sup>iv</sup>Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

<sup>v</sup>Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

<sup>vi</sup> $R1 = \sum |F_o| - |F_c| / \sum |F_o|$

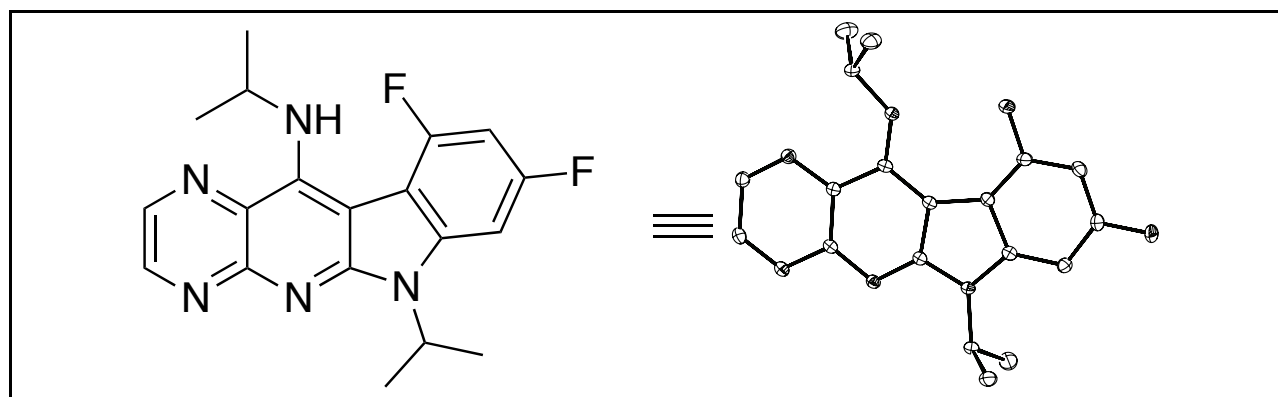
$wR2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{1/2}$

$GOF = [\sum w(F_o^2 - F_c^2)^2 / (n - p)]^{1/2}$

where n = the number of reflections and p = the number of parameters refined.

<sup>vii</sup>“ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations”. C.K. Johnson (1976) ORNL-5138.

### X-ray Structure Determination of Compound 43 (CCDC 992823)



Compound 1346,  $C_{19}H_{19}N_5F_2$ , crystallizes in the triclinic space group  $P\bar{1}$  with  $a=6.7843(6)\text{\AA}$ ,  $b=10.9039(8)\text{\AA}$ ,  $c=12.5675(10)\text{\AA}$ ,  $\alpha=67.575(4)^\circ$ ,  $\beta=87.517(4)^\circ$ ,  $\gamma=84.897(4)^\circ$ ,  $V=855.93(12)\text{\AA}^3$ ,  $Z=2$ , and  $d_{\text{calc}}=1.379\text{ g/cm}^3$ . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-K $\alpha$  radiation ( $\lambda=0.71073\text{ \AA}$ ) at a temperature of 143(1)K. Preliminary indexing was performed from a series of thirty-six  $0.5^\circ$  rotation frames with exposures of 10 seconds. A total of 2916 frames were collected with a crystal to detector distance of 37.6 mm, rotation widths of  $0.5^\circ$  and exposures of 20 seconds:

scan type	$2\theta$	$\omega$	$\phi$	$\chi$	frames
$\phi$	19.50	59.55	348.71	-26.26	739
$\omega$	7.00	0.92	261.23	-20.60	192
$\phi$	-23.00	315.83	257.18	28.88	219
$\phi$	-10.50	336.23	38.95	73.66	531
$\phi$	-10.50	300.13	17.77	39.97	739
$\phi$	22.00	14.84	145.80	97.50	496

Rotation frames were integrated using SAINT<sup>i</sup>, producing a listing of unaveraged  $F^2$  and  $\sigma(F^2)$  values which were then passed to the SHELXTL<sup>ii</sup> program package for further processing and structure solution. A total of 39187 reflections were measured over the ranges  $1.75 \leq \theta \leq 25.39^\circ$ ,  $-8 \leq h \leq 8$ ,  $-11 \leq k \leq 13$ ,  $0 \leq l \leq 15$  yielding 3103 unique reflections ( $R_{\text{int}} = 0.0268$ ). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS<sup>iii</sup> (minimum and maximum transmission 0.7123, 0.7452).

The structure was solved by direct methods (SHELXS-97<sup>iv</sup>). Refinement was by full-matrix least

squares based on  $F^2$  using SHELXL-97.<sup>v</sup> All reflections were used during refinement. The weighting scheme used was  $w=1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.2795P]$  where  $P = (F_o^2 + 2F_c^2)/3$ . Non-hydrogen atoms were refined anisotropically and hydrogen atoms were refined using a riding model. Refinement converged to  $R1=0.0342$  and  $wR2=0.0883$  for 2637 observed reflections for which  $F > 4\sigma(F)$  and  $R1=0.0424$  and  $wR2=0.0917$  and  $GOF = 1.035$  for all 3103 unique, non-zero reflections and 240 variables.<sup>vi</sup> The maximum  $\Delta/\sigma$  in the final cycle of least squares was 0.001 and the two most prominent peaks in the final difference Fourier were  $+0.191$  and  $-0.211 e/\text{\AA}^3$ .

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP<sup>vii</sup> representation of the molecule with 30% probability thermal ellipsoids displayed.

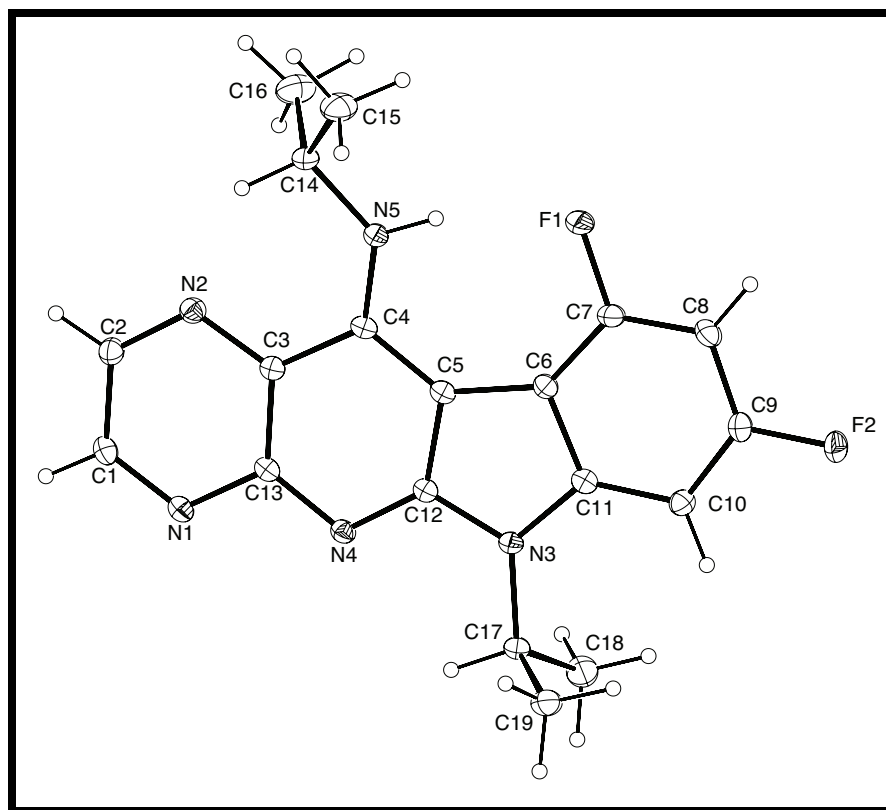


Figure 1. ORTEP drawing of the title compound with 30% probability thermal ellipsoids.

**Table 1. Summary of Structure Determination of Compound 1346**

Empirical formula	C <sub>19</sub> H <sub>19</sub> N <sub>5</sub> F <sub>2</sub>
Formula weight	355.39
Temperature	143(1) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	P $\bar{1}$
Cell constants:	
a	6.7843(6) Å
b	10.9039(8) Å
c	12.5675(10) Å
$\alpha$	67.575(4)°
$\beta$	87.517(4)°
$\gamma$	84.897(4)°
Volume	855.93(12) Å <sup>3</sup>
Z	2
Density (calculated)	1.379 Mg/m <sup>3</sup>
Absorption coefficient	0.101 mm <sup>-1</sup>
F(000)	372
Crystal size	0.38 x 0.08 x 0.03 mm <sup>3</sup>
Theta range for data collection	1.75 to 25.39°
Index ranges	-8 ≤ h ≤ 8, -11 ≤ k ≤ 13, 0 ≤ l ≤ 15
Reflections collected	39187
Independent reflections	3103 [R(int) = 0.0268]
Completeness to theta = 25.39°	98.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7452 and 0.7123
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	3103 / 0 / 240
Goodness-of-fit on F <sup>2</sup>	1.035
Final R indices [I > 2σ(I)]	R1 = 0.0342, wR2 = 0.0883
R indices (all data)	R1 = 0.0424, wR2 = 0.0917
Largest diff. peak and hole	0.191 and -0.211 e.Å <sup>-3</sup>



**Table 2. Refined Positional Parameters for Compound 1346**

Atom	x	y	z	$U_{eq}, \text{\AA}^2$
C1	0.2579(2)	0.20417(14)	0.57814(12)	0.0238(3)
C2	0.2484(2)	0.23618(13)	0.67632(12)	0.0239(3)
C3	0.25158(18)	0.45642(13)	0.56282(11)	0.0185(3)
C4	0.25143(18)	0.59421(13)	0.55015(11)	0.0187(3)
C5	0.24492(18)	0.68663(13)	0.43598(11)	0.0188(3)
C6	0.24031(19)	0.83054(13)	0.38033(11)	0.0195(3)
C7	0.2317(2)	0.93879(14)	0.41333(12)	0.0240(3)
C8	0.2289(2)	1.06883(14)	0.33710(12)	0.0265(3)
C9	0.2332(2)	1.09044(13)	0.22160(12)	0.0240(3)
C10	0.2409(2)	0.99140(13)	0.17891(12)	0.0227(3)
C11	0.24497(19)	0.86152(13)	0.25946(11)	0.0200(3)
C12	0.25131(18)	0.63860(13)	0.34557(11)	0.0181(3)
C13	0.25689(18)	0.42410(13)	0.46225(11)	0.0186(3)
C14	0.2964(2)	0.56170(14)	0.76111(11)	0.0243(3)
C15	0.4326(3)	0.63943(17)	0.79986(14)	0.0371(4)
C16	0.1053(3)	0.53960(18)	0.82919(14)	0.0406(4)
C17	0.2675(2)	0.72811(14)	0.12946(11)	0.0228(3)
C18	0.0817(2)	0.78776(16)	0.05784(13)	0.0347(4)
C19	0.4550(2)	0.78083(15)	0.06379(13)	0.0314(3)
N1	0.26116(17)	0.29462(11)	0.47308(10)	0.0226(3)
N2	0.24485(16)	0.35927(11)	0.66958(10)	0.0222(3)
N3	0.25252(16)	0.74459(11)	0.24054(9)	0.0197(3)
N4	0.25857(16)	0.51450(11)	0.35295(9)	0.0198(3)
N5	0.25866(18)	0.63514(11)	0.63810(10)	0.0252(3)
F1	0.22582(15)	0.91794(8)	0.52761(7)	0.0375(2)
F2	0.22848(13)	1.21800(8)	0.14399(7)	0.0309(2)

$U_{eq} = \frac{1}{3}[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta + 2U_{23}bb^*cc^*\cos\alpha]$

**Table 3. Positional Parameters for Hydrogens in Compound 1346**

Atom	x	y	z	$U_{iso}, \text{\AA}^2$
H1	0.2622	0.1151	0.5878	0.032
H2	0.2446	0.1679	0.7485	0.032
H8	0.2243	1.1389	0.3623	0.035
H10	0.2432	1.0102	0.1002	0.030
H14	0.3645	0.4748	0.7715	0.032
H15a	0.5544	0.6479	0.7567	0.056
H15b	0.4596	0.5933	0.8804	0.056
H15c	0.3700	0.7263	0.7870	0.056
H16a	0.0414	0.6238	0.8241	0.061
H16b	0.1328	0.4852	0.9083	0.061
H16c	0.0201	0.4957	0.7985	0.061
H17	0.2764	0.6322	0.1468	0.030
H18a	-0.0329	0.7534	0.1033	0.052
H18b	0.0881	0.7646	-0.0087	0.052
H18c	0.0731	0.8829	0.0338	0.052
H19a	0.4462	0.8761	0.0384	0.047
H19b	0.4704	0.7559	-0.0017	0.047
H19c	0.5671	0.7438	0.1132	0.047
H5	0.2372	0.7198	0.6187	0.034

**Table 4. Refined Thermal Parameters (U's) for Compound 1346**

Atom	U <sub>11</sub>	U <sub>22</sub>	U <sub>33</sub>	U <sub>23</sub>	U <sub>13</sub>	U <sub>12</sub>
C1	0.0254(7)	0.0185(7)	0.0285(8)	-0.0099(6)	-0.0016(6)	-0.0017(5)
C2	0.0269(8)	0.0206(7)	0.0231(7)	-0.0069(6)	-0.0010(6)	-0.0028(5)
C3	0.0146(6)	0.0213(7)	0.0204(7)	-0.0089(6)	-0.0008(5)	-0.0009(5)
C4	0.0157(6)	0.0225(7)	0.0212(7)	-0.0118(6)	0.0006(5)	-0.0022(5)
C5	0.0170(7)	0.0204(7)	0.0212(7)	-0.0106(6)	0.0005(5)	-0.0019(5)
C6	0.0178(7)	0.0201(7)	0.0216(7)	-0.0089(6)	0.0001(5)	-0.0013(5)
C7	0.0300(8)	0.0249(7)	0.0198(7)	-0.0113(6)	0.0017(6)	-0.0035(6)
C8	0.0319(8)	0.0210(7)	0.0302(8)	-0.0136(6)	0.0007(6)	-0.0035(6)
C9	0.0243(7)	0.0189(7)	0.0270(8)	-0.0063(6)	-0.0004(6)	-0.0028(5)
C10	0.0234(7)	0.0233(7)	0.0209(7)	-0.0078(6)	-0.0011(5)	-0.0022(5)
C11	0.0173(7)	0.0222(7)	0.0229(7)	-0.0109(6)	-0.0005(5)	-0.0022(5)
C12	0.0152(6)	0.0201(7)	0.0202(7)	-0.0092(5)	-0.0010(5)	-0.0008(5)
C13	0.0152(6)	0.0203(7)	0.0227(7)	-0.0110(6)	-0.0008(5)	-0.0010(5)
C14	0.0325(8)	0.0239(7)	0.0186(7)	-0.0104(6)	-0.0016(6)	-0.0020(6)
C15	0.0461(10)	0.0423(10)	0.0301(9)	-0.0199(7)	-0.0021(7)	-0.0120(7)
C16	0.0425(10)	0.0510(10)	0.0322(9)	-0.0190(8)	0.0093(7)	-0.0134(8)
C17	0.0301(8)	0.0222(7)	0.0187(7)	-0.0106(6)	-0.0016(6)	-0.0012(6)
C18	0.0415(9)	0.0345(9)	0.0309(8)	-0.0152(7)	-0.0132(7)	0.0022(7)
C19	0.0396(9)	0.0308(8)	0.0256(8)	-0.0127(6)	0.0069(6)	-0.0061(6)
N1	0.0240(6)	0.0207(6)	0.0254(6)	-0.0115(5)	-0.0004(5)	-0.0012(5)
N2	0.0231(6)	0.0224(6)	0.0222(6)	-0.0092(5)	-0.0006(5)	-0.0029(5)
N3	0.0234(6)	0.0197(6)	0.0177(6)	-0.0091(5)	-0.0005(4)	-0.0018(4)
N4	0.0211(6)	0.0200(6)	0.0207(6)	-0.0103(5)	-0.0012(4)	-0.0014(4)
N5	0.0395(7)	0.0187(6)	0.0194(6)	-0.0094(5)	-0.0012(5)	-0.0019(5)
F1	0.0703(7)	0.0236(5)	0.0226(5)	-0.0132(4)	0.0032(4)	-0.0058(4)
F2	0.0441(5)	0.0169(4)	0.0289(5)	-0.0052(3)	-0.0018(4)	-0.0034(3)

The form of the anisotropic displacement parameter is:

$$\exp[-2\pi(a^2U_{11}h^2+b^2U_{22}k^2+c^2U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$$

**Table 5. Bond Distances in Compound 1346, Å**

C1-N1	1.3131(18)	C1-C2	1.402(2)	C2-N2	1.3104(18)
C3-N2	1.3577(17)	C3-C13	1.4341(18)	C3-C4	1.4491(18)
C4-N5	1.3447(17)	C4-C5	1.4039(18)	C5-C12	1.4187(18)
C5-C6	1.4519(18)	C6-C7	1.3885(19)	C6-C11	1.4244(19)
C7-F1	1.3651(16)	C7-C8	1.374(2)	C8-C9	1.378(2)
C9-F2	1.3590(16)	C9-C10	1.373(2)	C10-C11	1.3903(19)
C11-N3	1.3796(17)	C12-N4	1.3172(17)	C12-N3	1.3838(17)
C13-N4	1.3514(17)	C13-N1	1.3635(17)	C14-N5	1.4664(17)
C14-C16	1.508(2)	C14-C15	1.517(2)	C17-N3	1.4722(17)
C17-C19	1.519(2)	C17-C18	1.5255(19)		

**Table 6. Bond Angles in Compound 1346, °**

N1-C1-C2	122.82(13)	N2-C2-C1	122.10(13)	N2-C3-C13	120.60(12)
N2-C3-C4	119.79(12)	C13-C3-C4	119.61(12)	N5-C4-C5	120.57(12)
N5-C4-C3	124.62(12)	C5-C4-C3	114.80(11)	C4-C5-C12	118.68(12)
C4-C5-C6	135.46(12)	C12-C5-C6	105.81(11)	C7-C6-C11	115.70(12)
C7-C6-C5	137.53(13)	C11-C6-C5	106.77(11)	F1-C7-C8	116.66(12)
F1-C7-C6	119.48(12)	C8-C7-C6	123.86(13)	C7-C8-C9	116.87(13)
F2-C9-C10	117.28(12)	F2-C9-C8	118.31(12)	C10-C9-C8	124.41(13)
C9-C10-C11	116.53(13)	N3-C11-C10	128.53(12)	N3-C11-C6	108.84(11)
C10-C11-C6	122.63(12)	N4-C12-N3	121.87(11)	N4-C12-C5	128.52(12)
N3-C12-C5	109.61(11)	N4-C13-N1	115.32(11)	N4-C13-C3	124.53(12)
N1-C13-C3	120.15(12)	N5-C14-C16	110.91(12)	N5-C14-C15	107.87(12)
C16-C14-C15	112.20(12)	N3-C17-C19	111.85(11)	N3-C17-C18	111.57(11)
C19-C17-C18	112.43(12)	C1-N1-C13	116.93(12)	C2-N2-C3	117.36(12)
C11-N3-C12	108.97(10)	C11-N3-C17	127.81(11)	C12-N3-C17	123.18(11)
C12-N4-C13	113.74(11)	C4-N5-C14	131.70(12)		

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<sup>i</sup>Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

<sup>ii</sup>Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

<sup>iii</sup>Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

<sup>iv</sup>Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

<sup>v</sup>Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

<sup>vi</sup> $R1 = \sum |F_o| - |F_c| / \sum |F_o|$

$wR2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)^2]^{1/2}$

$GOF = [\sum w(F_o^2 - F_c^2)^2 / (n - p)]^{1/2}$

where n = the number of reflections and p = the number of parameters refined.

<sup>vii</sup>“ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations”. C.K. Johnson (1976) ORNL-5138.