

SUPPORTING INFORMATION

INDEX

I.-GENERAL CONSIDERATIONS

II.-TABLES

III.-FIGURES

IV.-EXPERIMENTAL PROCEDURES AND CHARACTERIZATION DATA

IV.1.-PREPARATION OF PRECURSORS AND SUBSTRATES

IV.2.-GENERAL PROCEDURE FOR THE (TMEDA).I3-INITIATED REACTIONS

IV.3.-CIS/TRANS ISOMERIZATION OF OLEIC ACID METHYL ESTER

IV.4.-ISOLATION OF 2,4-DINITROPHENYLHYDRAZONE FROM CARBONYL COMPOUND

IV.5. CHARACTERIZATION OF COMPOUNDS

I.-GENERAL CONSIDERATIONS

All reactions were carried out in an argon atmosphere under anhydrous conditions or otherwise noted.

Reaction solvents such as acetonitrile, were chromatography quality and were not further purified. Chromatography and extraction solvents dichloromethane, chloroform, *iso*-octane, *n*-hexane, *n*-heptane, ethyl acetate, acetone, and, ethanol were purchased from commercial suppliers.

N,N,N',N'-tetramethylethylene diamine TMEDA was 99% pure and used as received from the supplier. Fluorinated reagents 1,1,1,2,2,3,3,4,4-nonafluoro-4-iodobutane (perfluorobutyl iodide), 1,1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-heptadecafluoro-8-iodooctane were used as received from the supplier without further purification, except when traces of iodine had to be removed, in which case, the neat liquid was passed through a neutral alumina column. Sulfides were commercially available and purified by column chromatography or vacuum-distilled; the synthesis of sugar thioaldoses 2,3,4-tri-*O*-acetyl-1-thio- β -D-glucuronic acid methyl ester **5** and 2,3,4,6-tetra-*O*-acetyl-1-thio- β -D-galactopyranose **7** and disaccharide 2',3',4',6',2,3,6-hepta-*O*-acetyl-1-thio- β -D-cellobiose **9** have been accomplished by standard techniques and are given in section IV.5.-. 2,2,6,6-Tetramethyl-1-piperidinyloxy (TEMPO) and 1,4-dinitrobenzene were Ultra-pure grade. Dye Rose Bengal (4,5,6,7-Tetrachloro-3',6'-dihydroxy-2',4',5',7'-tetraiodo-3*H*-spiro[isobenzofuran-1,9'-xanthen]-3-one), was 99.9% pure and used as received from the supplier. Yields were referred to as isolated yields of analytically pure material unless otherwise noted, as the case of yields calculated from ^{19}F NMR and ^1H NMR spectral integration.

Reactions were magnetically stirred and monitored by thin-layer chromatography (TLC) using Silica gel 60 F254 pre-coated plates (0.25 mm, Merk), and revealed by UV-light or CAN solution. Purification of the reaction products was carried out by flash column chromatography using Ultra Pure Silica Gel (230–400 mesh) or standard silica-gel for column chromatography (60 mesh).

The light source was a commercially available household 60-watt fluorescent light bulb or a 50 Watt black light ($\lambda_{\text{max}} = 370 \text{ nm}$).

^1H NMR spectra were recorded on a Bruker Avance 600 (600 MHz) spectrometers, and are reported in ppm using the solvent residual peak resonance as the internal standard (dimethylsulfoxide-*d*6 at 2.54 ppm, CDCl_3 at 7.26 ppm). ^1H NMR data are reported as follows: chemical shift; multiplicity; coupling constants (Hz); number of hydrogen.

Multiplicity is abbreviated as follows: s = singlet, d = doublet, t = triplet, dd = double doublet, m = multiplet, br = broad. Proton-decoupled ^{13}C NMR spectra were recorded on a Bruker Avance 500 (at 125.758 MHz), or on a Bruker Avance 600 (at 150.903 MHz) spectrometers and are reported in ppm using the C resonance signal from the solvent as the internal standard (acetone-*d*6 at 29.8 ppm, CDCl_3 at 77.00 ppm). ^{19}F NMR spectra were recorded on a Bruker Avance 500 (at 470.592 MHz), or a Bruker Avance 600 (at 564.686 MHz) spectrometers and are reported in ppm using the internal standard signal from the spectrometer. High-resolution mass spectra (HRMS) were obtained using JEOL-DX 700 mass spectrometer.

II.-TABLES

Table S1. Averaged ^{19}F NMR chemical shift (ppm) values observed for the CF_2 group in $\text{ICF}_2\text{-C}_3\text{F}_7$ (1 equiv, 35 μL neat) in mixtures with additives and substrates in a given solvent

entry	additive	$-\delta$ (ppm) δ I- $\text{CF}_2\text{-C}_3\text{F}_7$	$\Delta \delta$ (ppm) [δ (I- $\text{CF}_2\text{-C}_3\text{F}_7$) - δ (I- $\text{CF}_2\text{-C}_3\text{F}_7$) _{additive}]
1	-	64.16 ^a	0
2	-	66.98 ^b	0
3	1 equiv TMEDA	68.08 ^a	3.92
4	0.25 equiv TMEDA	68.14 ^a	3.98
5	0.4 equiv TMEDA	68.57 ^a	4.41
6	1 equiv TMEDA + 1 equiv PhSH	64.35 ^d	0.20
7	0.5 equiv TMEDA + 1 equiv PhSH	62.17 ^d	2.00
8	0.25 equiv TMEDA + 1 equiv PhSH	61.20 ^d	2.96

a.-in CD_3CN

b.-in DMSO-d_6

c.-in DMF

d.-in CDCl_3

III.-FIGURES

Figure S1. UV-visible Spectrum of C_4F_9I [0,6 M] in (MeCN at the working concentration. $\lambda_{max}= 462$ nm ($\epsilon= 0.677 \cdot 10^3 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$)

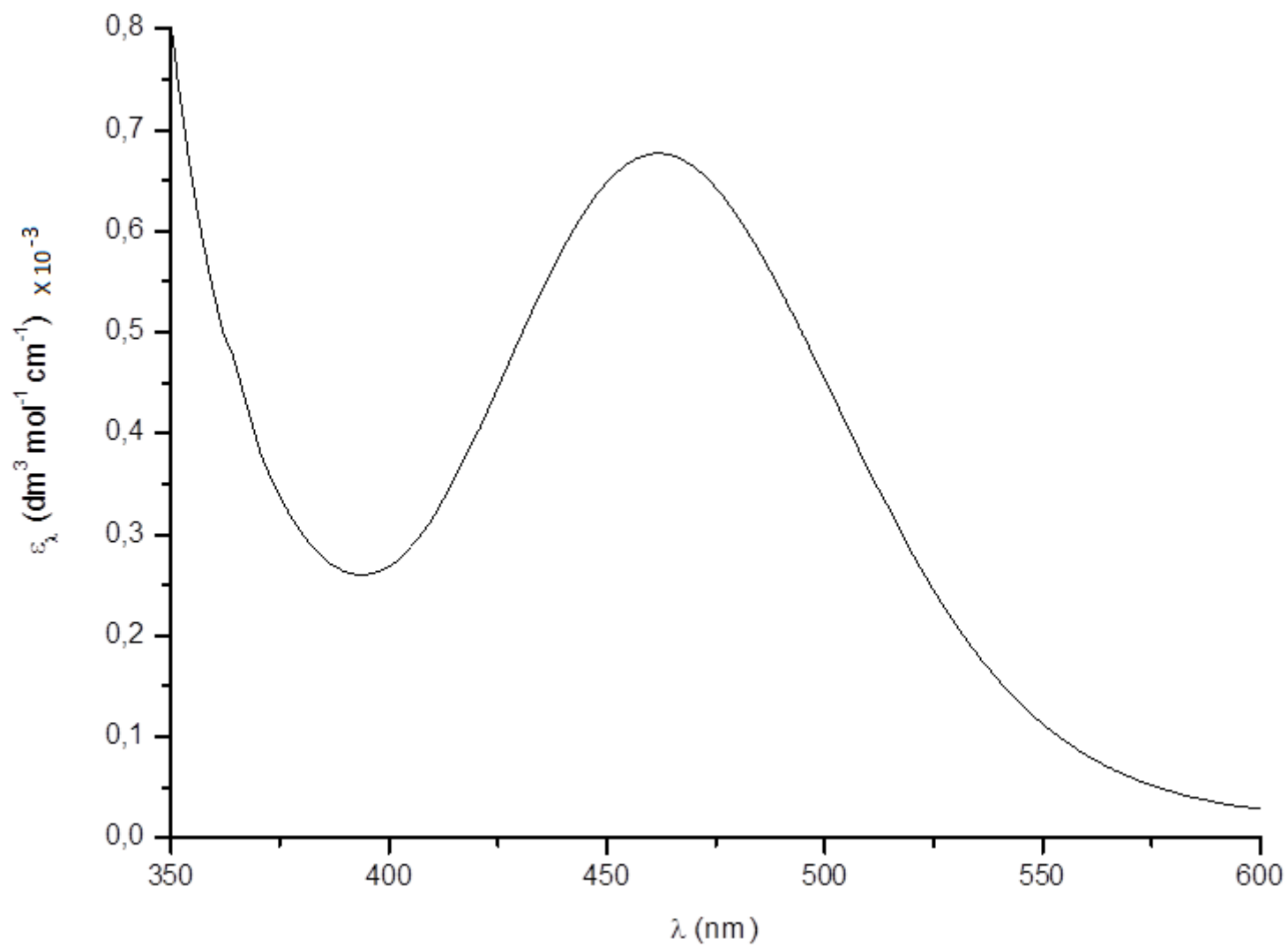


Figure S2. Uv-visible Spectrum of I₂ [0,55 mM] in (MeCN)

$$\lambda_{\max} = 457 \text{ nm } (\epsilon = 0.699 \text{ dm}^3 \text{ 10}^3 \text{ mol}^{-1} \text{ cm}^{-1})$$

$$\lambda_{\max} = 364 \text{ nm } (\epsilon = 0.741 \text{ dm}^3 \text{ 10}^3 \text{ mol}^{-1} \text{ cm}^{-1})$$

$$\lambda_{\max} = 291 \text{ nm } (\epsilon = 1.459 \text{ dm}^3 \text{ 10}^3 \text{ mol}^{-1} \text{ cm}^{-1})$$

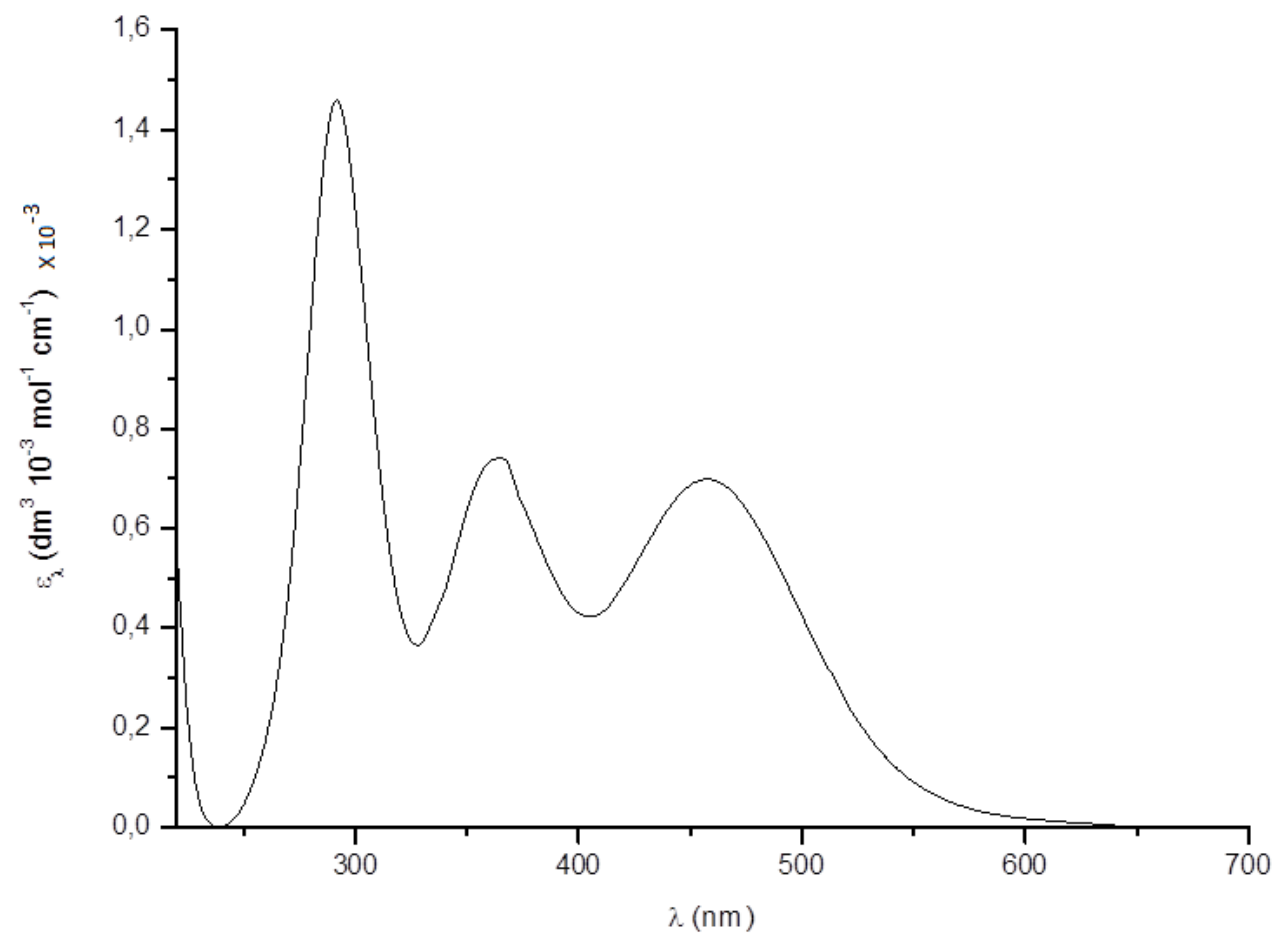


Figure S3. Uv-visible Spectrum of the mixture TMEDA [0.03 mM] + I₂ [0.06 mM] in (MeCN)

$$\lambda_{\text{max}} = 365 \text{ nm } (\epsilon = 7267 \text{ dm}^3 \text{ 10}^3 \text{ mol}^{-1} \text{ cm}^{-1})$$

$$\lambda_{\text{max}} = 292 \text{ nm } (\epsilon = 13330 \text{ dm}^3 \text{ 10}^3 \text{ mol}^{-1} \text{ cm}^{-1})$$

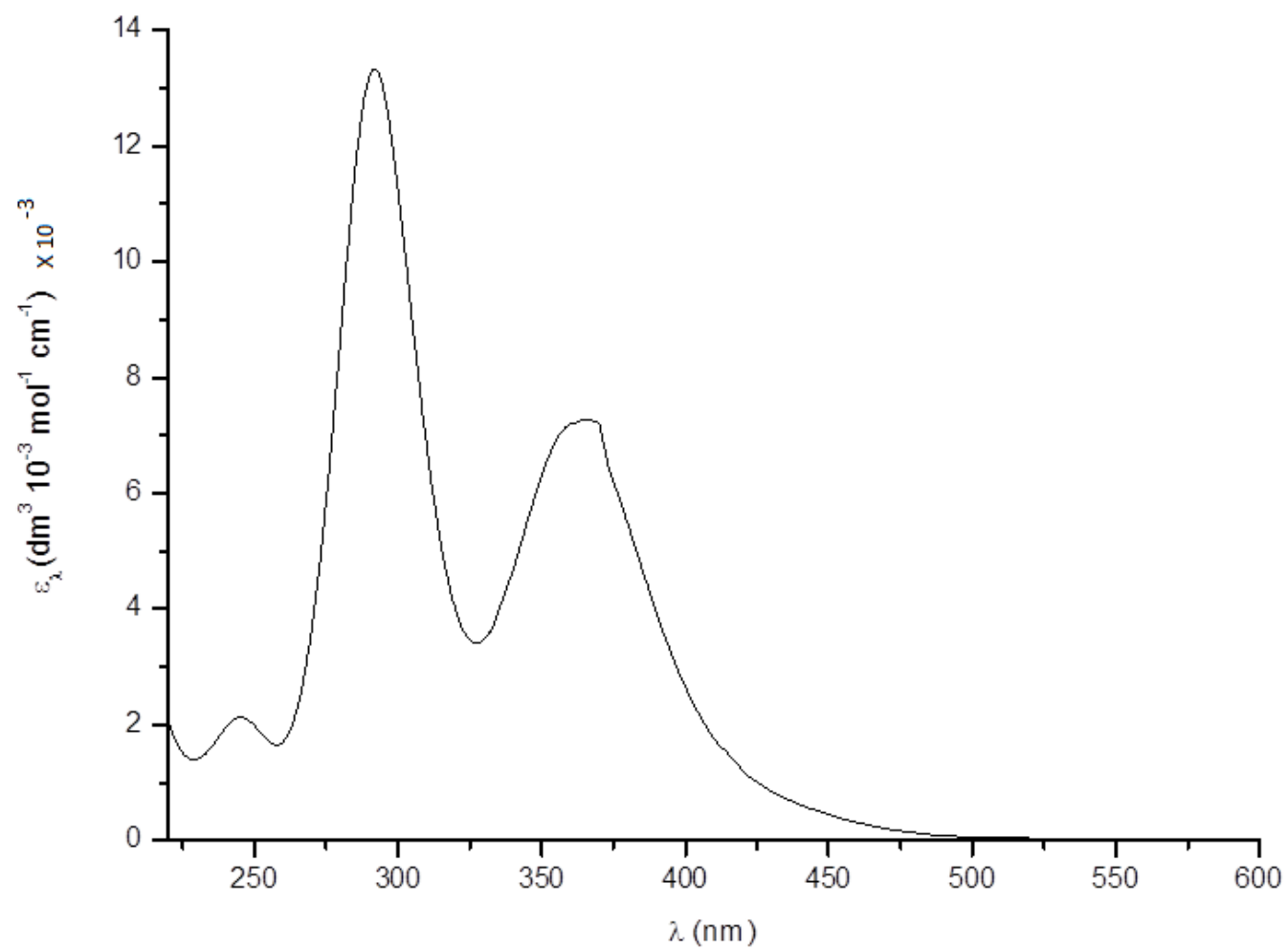
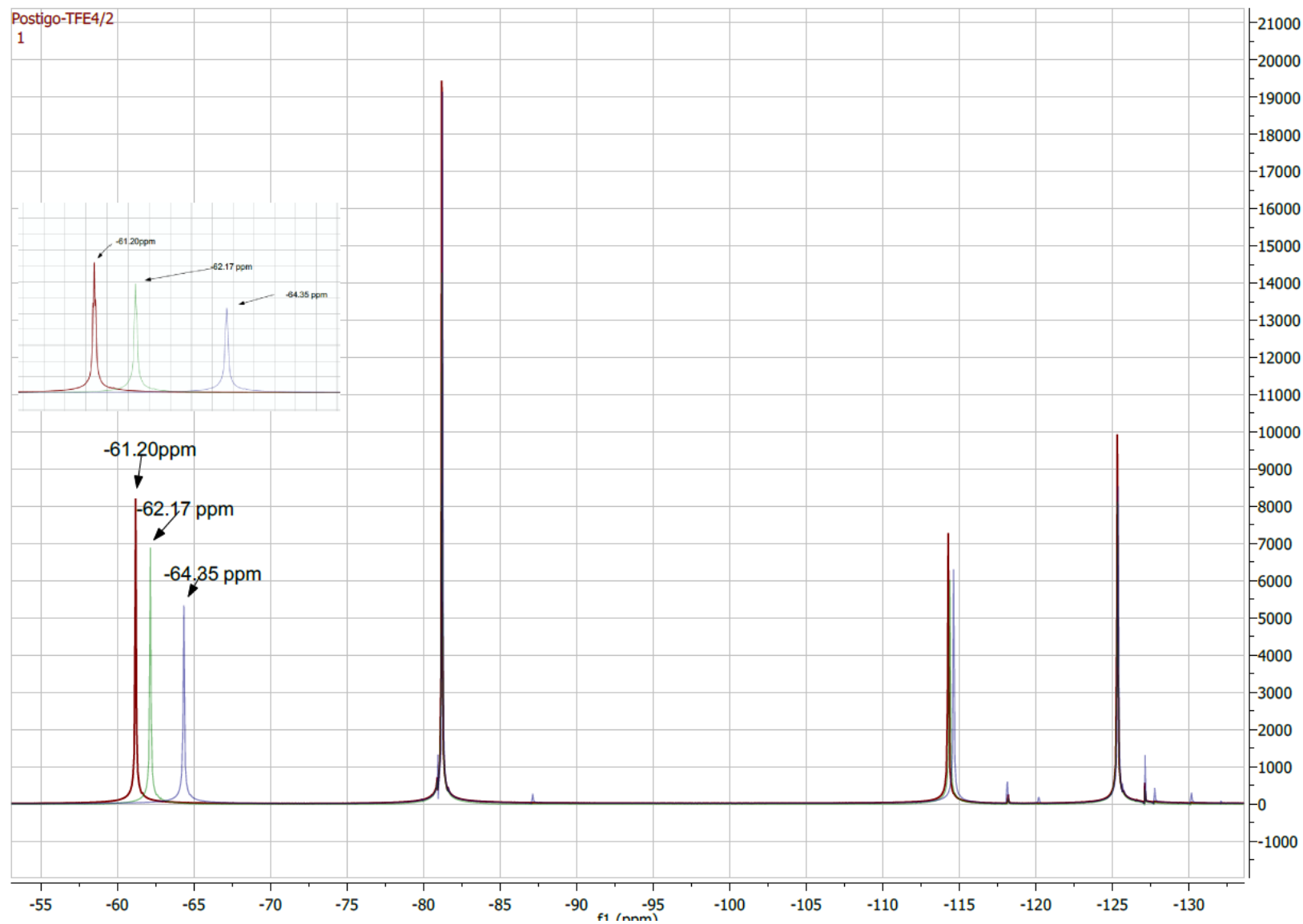


Figure S4: ^{19}F NMR overlaid spectra of $n\text{-C}_4\text{F}_9\text{I}$ in the presence of RSH, RSH and TMEDA



IV EXPERIMENTAL PROCEDURES AND CHARACTERIZATION DATA

IV.1.-PREPARATION OF PRECURSORS AND SUSBTATES

Sugar thioaldoses 2,3,4-tri-*O*-acetyl-1-thio- β -D-glucuronic acid methyl ester **5** and 2,3,4,6-tetra-*O*-acetyl-1-thio- β -D-galactopyranose were prepared by standard techniques. The disaccharide 2',3',4',6',2,3,6-hepta-*O*-acetyl-1-thio- β -D-cellobiose was also prepared by conventional techniques (see characterization, for preparation procedures).

IV.2.-GENERAL PROCEDURE FOR THE (TMEDA).I.I3)-INITIATED REACTIONS

In a 4 mL screw-cap vial provided with a micro stir bar, (TMEDA) I.I₃ complex, ca. 0.25 mM, substrate (0.6 mmol thiol), photocatalyst Rose Bengal where needed (0.05 equiv) and 3 mL of acetonitrile were introduced. The mixture was de-oxygenated with a stream of Ar for 15 min. C₄F₉I or other R_FI (3 equiv) was introduced by microliter syringe, and the vial sealed. The closed reaction vessel was placed in front of a 60 Watt household fluorescent light bulb (or 20 Watt fluorescent black light, $\lambda_{\text{max}} = 370 \text{ nm}$) and illuminated, under constant vigorous stirring, for 24 hrs or otherwise noted. After the reaction time was completed, the mixture was extracted thrice with CH₂Cl₂ / water / brine. The organic layers were gathered and dried over anhydrous Na₂SO₄, filtered and evaporated under vacuo. The crude reaction mixture was purified by silica-gel (60 mesh) column chromatography, with the eluants indicated in the TLC conditions (*vide infra*, spectral data). The polarity of the dye did not introduce any particular difficulty in the separation and purification protocol, as the several CH₂Cl₂ extractions eliminated the PC. The eluants employed are referred to in the TLC conditions of each compound.

IV.3.-TRIAL EXPERIMENT FOR THE CIS/TRANS ISOMERIZATION OF OLEIC ACID METHYL ESTER

Experiment 1:

Methyl oleate (0.5 mmol) was added to degassed MeCN (3 mL) along with TMEDA (1 equiv) and I₂ (2 equiv) in a 3 mL glass vial capped with a screw-cap provided with septum and a stirring bar. The set-up was placed on a stir plate, and vigorously stirred throughout the irradiation with a 60 Watt CFL bulb (commercial fluorescent light bulb). Aliquots were taken at 30min intervals, added an internal standard (palmitic acid methyl ester), and analyzed through capillary GC/MS with an appropriate capillary column (DB-5, 30 m). There was no conversion of oleic acid methyl ester into elaidic acid methyl ester. This experiment rules out the production of I atoms through irradiation of the complex [(TMEDA) I.I₃], or else are produced in sufficiently low quantities to enable the cis/trans isomerization. Conducting the irradiation with a black fluorescent light bulb (20 Watt), $\lambda_{\text{max}} = 370 \text{ nm}$ from a black fluorescent light bulb), the same results were obtained.

Experiment 2:

Methyl oleate (0.5 mmol) was added to degassed MeCN (3 mL) along with TMEDA (1 equiv) and C₄F₉I (3 equiv) in a 3 mL glass vial equipped with a screw-cap provided with a septum containing a stirring bar. The set-up was placed on a stir plate, sealed, and vigorously stirred throughout the irradiation with a 60 Watt CFL bulb (commercial fluorescent light bulb). Aliquots were taken at 30min intervals, added a standard, and analyzed through capillary GC/MS with a DB-5 capillary column and an internal standard (palmitic acid methyl ester). There was no conversion of oleic acid methyl ester into elaidic acid methyl ester

noticed through irradiation time. This experiment rules out that I₂ present in neat C₄F₉I, or else I₂ produced through photolytic cleavage of C₄F₉I complexes efficiently with TMEDA but do not produce I atoms on irradiation capable of the cis/trans isomerization of oleic acid methyl ester.

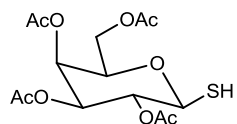
IV.4.-ISOLATION OF A 2,4-DINITROPHENYLHYDRAZONE FROM A CARBONYL COMPOUND DERIVED FROM TMEDA

To the crude reaction mixture from the irradiation of a thiol in the presence of TMEDA/n-C₄F₉I, 2,4-dinitrophenyl hydrazine was added, and left stirring at 10 °C for 24 hrs. The reaction was followed by TLC, and a deep yellow stain formed after a few hours of reaction. The hydrazine was separated by column chromatography, and the hydrazone of a carbonyl compound was detected by IR spectroscopy but not fully characterized. The yield of the hydrazine based on starting TMEDA was less than 5%.

IV.5. CHARACTERIZATION OF COMPOUNDS

All compounds are unknown chemicals, unless otherwise noted, and are reported as % yields obtained by weight or NMR integration (from ¹H and ¹⁹F NMR integration) of the crude reaction mixtures. Isolated purified mass of compounds are expressed in Gram units. Characterizations employ ¹H, ¹³C, ¹⁹F 1D-NMR techniques, and 2D NMR spectroscopic techniques (HSQC, HMBC, COSY experiments)

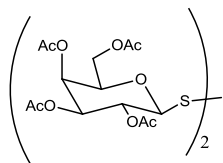
2,3,4,6-tetra-O-acetyl-1-thio-β-D-galactopyranose 7



2,3,4,6-tetra-*O*-acetyl-1-thio- β -D-galactopyranose was obtained as previously described^[39] and showed the same properties as reported. Briefly, the thioaldose was obtained by hydrolysis of the corresponding isothiuronium salt promoted by sodium carbonate. The isothiuronium salt was in turn obtained by reaction of acetobromogalactose with thiourea in boiling acetone.

^1H NMR (200 MHz, Cl_3CD) δ = 5.45 (dd, 1H, $J_{3,4}$ = 3.4, $J_{4,5}$ = 1.0 Hz, H-4), 5.20 (t, 1H, $J_{2,3}$ = $J_{1,2}$ = 10.0 Hz, H-2), 5.00 (dd, $J_{3,4}$ = 3.4, $J_{2,3}$ = 10.0 Hz, H-3), 4.55 (t, 1H, $J_{1,2}$ = $J_{1,\text{SH}}$ = 10.0 Hz, H-1), 4.20 (m, 2H, H-6a, H-6b), 3.95 (t, 1H, $J_{5,6a}$ = $J_{5,6b}$, H-5), 2.40 (d, 1H, $J_{1,\text{SH}}$ = 10.0 Hz, SH), 2.09, 2.02, 1.97, 1.91 (4s, 12H, 4 CH_3CO). ^{13}C NMR (50.3 MHz, Cl_3CD) δ = 170.3, 170.1, 169.9, 169.7 (4 COCH_3), 79.2 (C-1), 74.9, 71.6, 70.8, 67.2 (C-5, C-3, C-2, C-4), 61.5 (C-6), 20.8, 20.7 (2 $\times$), 20.6 (4 COCH_3).

Bis(2,3,4,6-tetra-*O*-acetyl-1-deoxy-1-thio- β -D-galactopyranosyl) 1,1'-disulfide **7S**

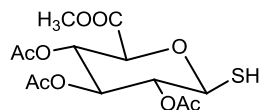


The disulfide was obtained by oxidation of the corresponding 1-thio- β -D-galactopyranose.^[40]

^1H NMR (200 MHz, Cl_3CD) δ = 5.43 (d, 1H, $J_{3,4}$ = 2.8 Hz, H-4), 5.35 (t, 1H, $J_{2,3}$ = $J_{1,2}$ = 9.9 Hz, H-2), 5.07 (dd, $J_{2,3}$ = 9.9, $J_{3,4}$ = 3.4 Hz, H-3), 4.56 (d, 1H, $J_{1,2}$ = 9.9 Hz, H-1), 4.27-3.96 (m, 3H, H-5, H-6a, H-6b), 2.17, 2.09, 2.04, 1.98 (4s, 12H, 4 CH_3CO).

^{13}C NMR (50.3 MHz, Cl_3CD) δ = 170.7, 170.3, 70.2, 170.0 (4 COCH_3), 88.7 (C-1), 74.9, 72.0, 67.8, 67.2 (C-5, C-3, C-2, C-4), 61.0 (C-6), 20.8, 20.7, 20.6, 20.5 (4 COCH_3).

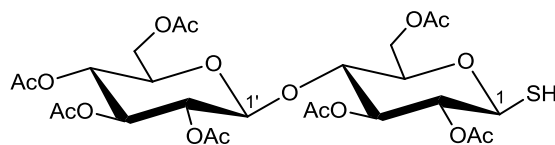
Methyl 2,3,4-tri-*O*-acetyl-1-thio- β -D-glucopyranosyluronate, **5**



Methyl 2,3,4-tri-*O*-acetyl-1-thio- β -D-glucopyranosyluronate was obtained as previously described^[41] and showed the same properties as reported. Briefly, methyl (2,3,4-tri-*O*-acetyl-D-glucopyranosyl) uronate bromide was first reacted with potassium thioacetate in acetone to yield the 1-thioacetyl derivative which upon treatment with sodium methoxide, at $-45\text{ }^\circ\text{C}$, provided the corresponding glucuronyl 1-thiol.

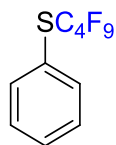
$^1\text{H-NMR}$ (200 MHz, Cl_3CD) δ = 5.28-5.16 (m, 2H, H-3, H-4), 4.98 (t, 1H, $J_{2,3} = J_{1,2} = 9.6$ Hz, H-2), 4.57 (t, 1H, $J_{1,2} = J_{1,\text{SH}} = 9.9$ Hz, H-1), 4.04 (d, 1H, $J_{4,5} = 9.8$ Hz, H-5), 3.75 (s, 3H, OCH_3), 2.38 (d, 1H, $J_{1,\text{SH}} = 9.9$ Hz, SH), 2.07, 2.01 (2 $\times$) (3s, 9H, 3 CH_3CO). ^{13}C NMR (50.3 MHz, Cl_3CD) δ = 169.7, 169.5, 169.3 (3 COCH_3), 166.7 (COOCH_3), 79.0 (C-1), 76.6, 73.3, 72.7, 69.3 (C-5, C-3, C-2, C-4), 53.0 (OCH_3), 20.7, 20.6, 20.5 (3 COCH_3).

2,2',3,3',4',6,6'-hepta-O-acetyl-1-thio- β -D-cellobiose **9**



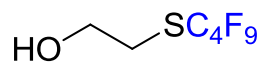
$^1\text{H-NMR}$ (500 MHz, Cl_3CD) δ = 5.15 (t, 1H, $J_{2,3} = J_{3,4} = 9.3$ Hz, H-3), 5.13 (t, 1H, $J_{2',3'} = J_{3',4'} = 9.4$ Hz, H-3'), 5.05 (dd, 1H, $J_{3',4'} = 9.4$, $J_{4',5'} = 9.9$ Hz, H-4'), 4.91 (dd, 1H, $J_{1',2'} = 8.1$, $J_{2',3'} = 9.4$ Hz, H-2'), 4.88 (dd, 1H, $J_{2,3} = 9.3$, $J_{1,2} = 9.9$ Hz, H-2), 4.51 (d, 1H, $J_{1,2} = 9.9$ Hz, H-1), 4.49 (d, 1H, $J_{1',2'} = 8.1$ Hz, H-1'), 4.47 (dd, 1H, $J_{5,6a} = 2.0$, $J_{6a,6b} = 12.1$ Hz, H-6a), 4.36 (dd, 1H, $J_{5,6a'} = 4.4$, $J_{6a',6b'} = 12.5$ Hz, H-6a'), 4.08 (dd, 1H, $J_{5,6b} = 5.3$, $J_{6a,6b} = 12.1$ Hz, H-6b), 4.03 (dd, 1H, $J_{5,6b'} = 2.2$, $J_{6a',6b'} = 12.5$ Hz, H-6b'), 3.77 (dd, 1H, $J_{3,4} = 9.3$, $J_{4,5} = 9.9$ Hz, H-4), 3.66-3.60 (m, 2H, H-5, H-5'), 2.13, 2.08, 2.06, 2.02, 2.01, 2.00, 1.97 (7s, 21H, 7 CH_3CO). ^{13}C NMR (125 MHz, Cl_3CD) δ = 170.6, 170.4, 170.3, 170.0, 169.8, 169.4, 169.2 (7 COCH_3), 100.9 (C-1'), 78.6 (C-1), 77.3 (C-5), 76.4 (C-4), 73.9 (C-2), 73.3 (C-3), 73.0 (C-3'), 72.1 (C-5'), 71.7 (C-2'), 67.9 (C-4'), 62.2 (C-6), 61.7 (C-6'), 21.0, 20.9, 20.8, 20.7 (4 $\times$) (7 COCH_3).

5H-[1,2,4]triazino[5,6-b]indole-3-thiol **16** was prepared according to literature procedures^[42] and the spectroscopic data match well with the reported values.



Perfluorobutyl(phenyl)sulfane **1**^[14]: Yield: 80 %. Isolated and purified mass obtained: 10 mg. TLC (Hexane): R_f = 0.75.

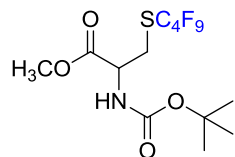
¹H-NMR (600 MHz, Cl₃CD) δ : 7.66 (d, 2H, J = 7.4 Hz), 7.51 (t, 1H, J = 7.5 Hz), 7.43 (t, 2H, J = 7.5 Hz). **¹³C NMR** (150 MHz, Cl₃CD) δ : 137.6, 131.2, 129.6, 122.9. **¹⁹F NMR** (564.603 MHz, Cl₃CD) δ : -81.0 (t, 3F), -87.1 (m, 2F), -120.1 (m, 2F), -125.5 (m, 2F)



2-((perfluorobutyl)thio)ethanol **2**^[9]: Yield: 75 %. Isolated and purified mass obtained: 12 mg. TLC (AcOEt:

Hexane 1:1 v/v): R_f = 0.7.

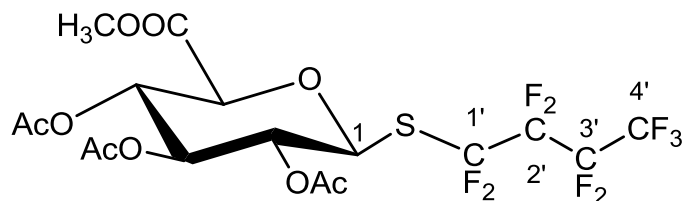
¹H-NMR (600 MHz, Cl₃CD) δ : 3.89 (t, 2H, J = 6.1 Hz), 3.14 (t, 2H, J = 6.1 Hz), 1.97 (bs, 1H). **¹³C NMR** (150 MHz, Cl₃CD) δ : 61.6, 31.7. **¹⁹F NMR** (564.603 MHz, Cl₃CD) δ : -81.0 (t, 3F), -87.0 (m, 2F), -120.7 (m, 2F), -125.5 (m, 2F).



Methyl 2-((*tert*-butoxycarbonyl)amino)-3-((perfluorobutyl)thio)propanoate,^[6] 4: Yield: 25 %. Isolated and purified mass obtained: 10 mg. TLC (AcOEt: Hexane 4:6 v/v): R_f = 0.8.

¹H-NMR (500 MHz, Cl₃CD) δ : 5.37 (d, 1H, J = 6.2 Hz), 4.63 (d, 1H, J = 5.5 Hz), 3.79 (bs, 3H), 3.53 (dd, 1H, J = 13.6 Hz, 4 Hz), 3.36 (dd, 1H, J = 13.7 Hz, 4.5 Hz), 1.44 (bs, 9H). **¹³C NMR** (150 MHz, Cl₃CD) δ : 170.2, 164.4, 155.0, 131.4, 80.8, 53.2, 53.1, 31.1, 28.3. **¹⁹F NMR** (564.603 MHz, Cl₃CD) δ : -81.0 (t, 3F), -86.7 (m, 2F), -120.5 (m, 2F), -125.5 (m, 2F).

Perfluorobutyl 2,3,4-tri-*O*-acetyl-1-thio-β-*D*-methylglucopyranosyluronate, **6**

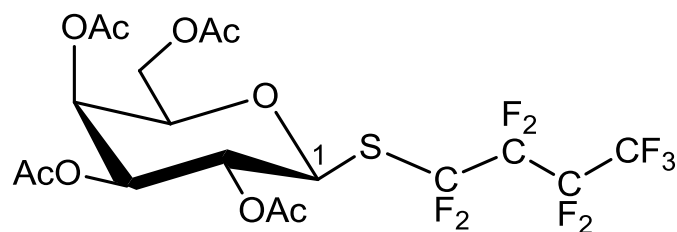


Yield: 75 %. Isolated and purified mass obtained: 21 mg. **TLC** (AcOEt: Hexane 4:6 v/v): R_f = 0.7.

¹H-NMR (600 MHz, Cl₃CD) δ = 5.33, 5.22 (2dd, 2H, J = 9.9, J = 8.9 Hz, H-3, H-4), 5.08 (dd, 1H, J_{2,3} = 9.9, J_{1,2} = 10.2 Hz, H-2), 5.04 (d, 1H, J_{1,2} = 10.2 Hz, H-1), 4.09 (d, 1H, J_{4,5} = 9.9 Hz, H-5), 3.77 (s, 3H, OCH₃), 2.06, 2.03 (2×) (3s, 9H, 3 CH₃CO). **¹³C NMR** (150.9 MHz, Cl₃CD) δ = 170.0, 169.4, 169.3 (3 COCH₃), 166.4 (COOCH₃), 80.8 (C-1), 76.4, 72.7, 69.1, 69.0 (C-5, C-3, C-

2, C-4), 53.2 (OCH₃), 20.7, 20.6, 20.5 (3 COCH₃). **¹⁹F NMR** δ = 470.592 (Cl₃CD) δ = -80.9 (t, *J* = 9.4 Hz, 3H, CF₃), -85.6, -88.2 (2 brd, 2F, *J* = 240.0 Hz, C^{1'}F₂), -120.4, -121.0 (2 dt, 2F, *J* = 8.1, *J* = 294.4 Hz, C^{2'}F₂), -125.5 (t, 2F, *J* = 12.8 Hz, C^{3'}F₂). HRMS (ESI): *m/z* [M+H]⁺ calcd for C₁₇H₁₇F₉NaO₉S: 591.03418, found: 591.03342.

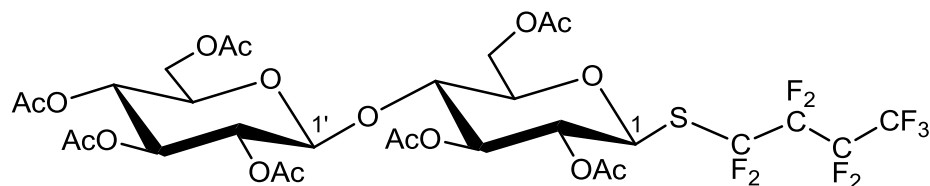
Perfluorobutyl 2,3,4,6-tetra-*O*-acetyl-1-thio-β-D-galactopyranoside, **8**



Yield: 83 %. Isolated and purified mass obtained: 30 mg. **TLC** (AcOEt: Hexane 1:1 v/v): R_f = 0.6

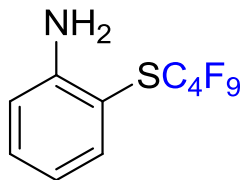
¹H NMR (600 MHz, Cl₃CD) δ = 5.44 (d, 1H, *J*_{3,4} = 2.8 Hz, H-4), 5.25 (t, 1H, *J*_{2,3} = *J*_{1,2} = 10.0 Hz, H-2), 5.10 (dd, *J*_{3,4} = 3.3, *J*_{2,3} = 10.0 Hz, H-3), 4.99 (d, 1H, *J*_{1,2} = 10.0 Hz, H-1), 4.16 (dd, 1H, *J*_{5,6a} = 7.2, *J*_{6a,6b} = 11.4 Hz, H-6a), 4.10 (dd, 1H, *J*_{5,6b} = 5.9, *J*_{6a,6b} = 11.4 Hz, H-6b), 4.00 (dd, 1H, *J*_{5,6a} = 7.2, *J*_{5,6b} = 5.9 Hz, H-5), 2.16, 2.07, 2.03, 1.99 (4s, 12H, 4 CH₃CO). **¹³C NMR** (150.9 MHz, Cl₃CD) δ = 170.5, 170.2, 169.9, 169.6 (4 COCH₃), 81.3 (C-1), 75.2, 71.7, 67.0, 66.5 (C-5, C-3, C-2, C-4), 61.4 (C-6), 20.7, 20.6 (3×) (4 COCH₃). **¹⁹F NMR** (470.592 MHz, Cl₃CD) δ = -81.0 (t, *J* = 9.5 Hz, 3H, CF₃), -85.8, -88.9 (2 brd, 2F, *J* = 241.0 Hz, C^{1'}F₂), -120.3, -121.2 (2 dt, 2F, *J* = 8.4, *J* = 294.3 Hz, C^{2'}F₂), -125.5 (t, 2F, *J* = 13.2 Hz, C^{3'}F₂). HRMS (ESI): *m/z* [M+H]⁺ calcd for C₁₈H₁₉F₉NaO₉S: 605.04983, found: 605.04978.

Perfluorobutyl 2,2',3,3',4',6,6'-hepta-O-acetyl-1-thio- β -D-cellobioside, **10**



Yield: 50 %. Isolated and purified mass obtained: 20 mg. **TLC** (AcOEt: Hexane 1:1 v/v): R_f = 0.6

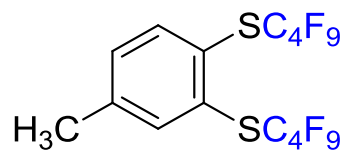
^1H NMR (600 MHz, Cl_3CD) δ = 5.23 (t, 1H, $J_{2,3} = J_{3,4} = 9.0$ Hz, H-3), 5.14 (t, 1H, $J_{2',3'} = J_{3',4'} = 9.4$ Hz, H-3'), 5.05 (dd, 1H, $J_{3',4'} = 9.4$ Hz, $J_{4',5'} = 10.0$ Hz, H-4'), 4.98 (dd, 1H, $J_{2,3} = 9.0$, $J_{1,2} = 10.0$ Hz, H-2), 4.95 (d, 1H, $J_{1,2} = 10.0$ Hz, H-1), 4.92 (dd, 1H, $J_{2',3'} = 9.4$ Hz, $J_{1',2'} = 8.0$ Hz, H-2'), 4.49 (d, 1H, $J_{1',2'} = 8.0$ Hz, H-1'), 4.46 (dd, 1H, $J_{5,6a} = 2.0$ Hz, $J_{6a,6b} = 12.0$ Hz, H-6a), 4.35 (dd, 1H, $J_{5',6a'} = 4.5$ Hz, $J_{6a',6b'} = 12.4$ Hz, H-6a'), 4.12 (dd, 1H, $J_{5,6b} = 6.1$ Hz, $J_{6a,6b} = 12.0$ Hz, H-6b), 4.03 (dd, 1H, $J_{5',6b'} = 2.0$ Hz, $J_{6a',6b'} = 12.4$ Hz, H-6b'), 3.76 (dd, 1H, $J_{3,4} = 9.0$ Hz, $J_{4,5} = 10.0$ Hz, H-4), 3.70-3.65 (m, 2H, H-5, H-5'), 2.10, 2.08, 2.06, 2.03, 2.02, 2.01, 1.98 (7s, 21H, 7 CH_3CO). **^{13}C NMR** (150.9 MHz, Cl_3CD) δ = 170.5, 170.3, 170.2, 169.7, 169.6, 169.4, 169.2 (7 COCH_3), 100.9 (C-1'), 80.7 (C-1), 77.3 (C-5), 76.2 (C-4), 73.2 (C-3), 73.0 (C-3'), 72.2 (C-5'), 71.7 (C-2'), 69.6 (C-2), 67.9 (C-4'), 62.0 (C-6), 61.7 (C-6'), 20.8, 20.7, 20.6 (7 COCH_3). **^{19}F NMR** 470.592 Cl_3CD) δ = -80.9 (t, $J = 9.7$ Hz, 3H, CF_3), -85.8, -88.8 (2 brd, 2F, $J = 242.0$ Hz, C^1F_2), -120.0, -121.2 (2 dt, 2F, $J = 8.5$, $J = 294.6$ Hz, C^2F_2), -125.5 (t, 2F, $J = 13.0$ Hz, C^3F_2). **HRMS** (ESI): m/z $[M+\text{H}]^+$ calcd for $\text{C}_{30}\text{H}_{36}\text{F}_9\text{O}_{17}\text{S}$: 871.1451, found: 871.1456.



2-((perfluorobutyl)thio)aniline **11**^[22]: Yield: 90 %. Isolated and purified mass obtained: 15 mg. TLC (CH₂Cl₂:

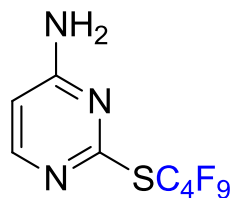
iso-octane 1:1 v/v): R_f = 0.6

¹H-NMR (600 MHz, Cl₃CD) δ : 7.47 (d, 1H, *J* = 8.6 Hz), 7.28 (t, 1H, *J* = 8.2 Hz), 6.78 (d, 1H, *J* = 8.1 Hz), 6.74 (t, 1H, *J* = 7.8 Hz), 4.45 (s, 2H). ¹³C NMR (150 MHz, Cl₃CD) δ : 151.1, 140.0, 133.4, 118.8, 115.8, 104.5. ¹⁹F NMR (564.603 MHz, Cl₃CD) δ : -81.1 (t, 3F), -87.1 (t, 2F), -120.4 (m, 2F), -125.5 (m, 2F).



(4-methyl-1,2-phenylene)bis((perfluorobutyl)sulfane) **13**: Yield: 50 %. Isolated and purified mass obtained: 8 mg. TLC (Hexane): R_f = 0.6

¹H-NMR (500 MHz, Cl₃CD) δ : 7.78 (d, 1H, *J* = 8.0 Hz), 7.72 (bs, 1H), 7.37 (cplx d, 1H, *J*_o = 8 Hz, *J*_m = 2Hz, *J*_{H-F5} = 0.7 Hz), 2.44 (s, 3H). ¹³C NMR (125.721 MHz, Cl₃CD) δ : 143.2, 140.3, 139.6, 133.0, 130.8, 127.3, 21.3. ¹⁹F NMR (470.585 MHz, Cl₃CD) δ : -81.0 (c, 6F), -86.3 (dt, 4F), -120.4 (m, 4F), -125.6 (m, 4F). HRMS (ESI): *m/z* [*M*+H]⁺ calcd for C₁₅H₇F₁₈S₂: 592.9623, found: 592.9641.

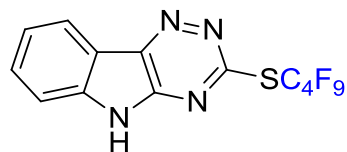


2-((perfluorobutyl)thio)pyrimidin-4-amine, **15**: Yield: 48 %. Isolated and purified mass obtained: 38 mg. TLC

(AcOEt: MeOH + I Acetic Acid 9:1 v/v): R_f = 0.8

¹H-NMR (600 MHz, Cl₃CD) δ : 8.00 (d, 1H, *J* = 5.9 Hz), 7.34 (bs, 2H), 6.37 (d, 1H, *J* = 5.9 Hz). **¹³C NMR** (150 MHz, Cl₃CD) δ : 163.7, 161.8, 155.6, 104.4. **¹⁹F NMR** (564.603 MHz, Cl₃CD) δ : -80.7 (t, 3F), -87.6 (t, 2F), -120.1 (m, 2F), -125.3 (m, 2F).

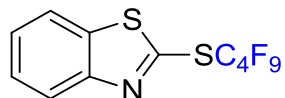
HRMS (ESI): *m/z* [*M*+H]⁺ calcd for C₈H₅F₉N₃S: 345.9982, found: 345.9896.



3-((perfluorobutyl)thio)-5H-[1,2,4]triazino[5,6-b]indole **17**: Yield: 50 %. Isolated and purified mass

obtained: 10 mg. TLC (AcOEt: Hexane 1:1 v/v): R_f = 0.8

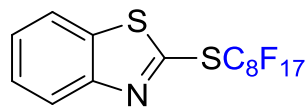
¹H-NMR (600 MHz, Cl₃CD) δ : 8.42 (d, 1H, *J* = 7.8 Hz), 7.80 (t, 1H, *J* = 8.3 Hz), 7.66 (d, 1H, *J* = 8.2 Hz), 7.52 (t, 1H, *J* = 7.9 Hz). **¹³C NMR** (150 MHz, Cl₃CD) δ : 158.2, 146.5, 143.1, 141.4, 132.3, 123.1, 122.4, 116.9, 113.1. **¹⁹F NMR** (564.603 MHz, Cl₃CD) δ : -80.4 (t, 3F), -86.3 (m, 2F), -119.8 (m, 2F), -125.1 (m, 2F). HRMS (ESI): *m/z* [*M*+H]⁺ calcd for C₁₃H₆F₉N₄S: 421.0091, found: 421.0045.



2-((perfluorobutyl)thio)benzo[d]thiazole **19**^[29]: Yield: 99 %. Isolated and purified mass obtained: 23 mg. TLC

(CHCl₃: iso-octane: MeOH 0.8:1: 0.2 v/v): R_f = 0.85

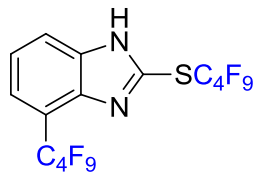
¹H-NMR (500 MHz, Cl₃CD) δ : 8.17 (cplx d, 1H, *J* = 8.2 Hz, 0.7 Hz), 7.92 (cplx d, 1H, *J* = 8.1 Hz, 0.7 Hz), 7.58 (m, 1H, *J* = 8.3 Hz, 1.2 Hz), 7.52 (m, 1H, *J* = 7.2 Hz, 1.3 Hz). **¹³C NMR** (125.721 MHz, Cl₃CD) δ : 153.4, 150.0, 138.7, 127.2, 127.1, 124.6, 121.5. **¹⁹F NMR** (470.585 MHz, Cl₃CD) δ : -80.9 (t, 3F), -85.2 (t, 2F), -119.8 (m, 2F), -125.5 (m, 2F). HRMS (ESI):*m/z* [*M*+H]⁺ calcd for C₁₁H₅F₉NS₂: 385.9641, found: 385.9625.



2-((perfluorooctyl)thio)benzo[d]thiazole **20**: Yield: 50 %. Isolated and purified mass obtained: 10 mg. TLC

(CHCl₃: iso-octane: MeOH 0.8:1: 0.2 v/v): R_f = 0.75

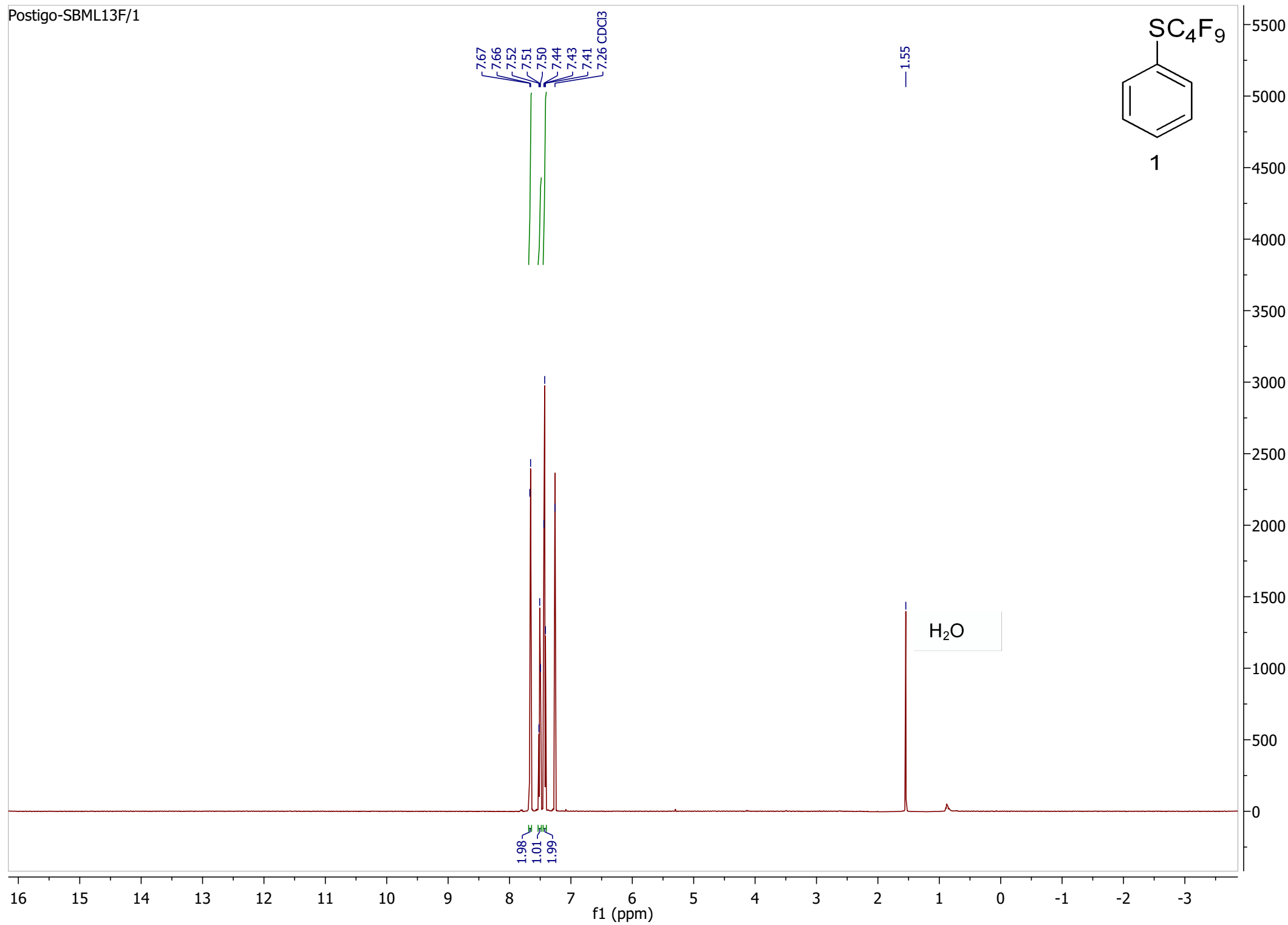
¹H-NMR (600 MHz, Cl₃CD) δ : 8.18 (d, 1H, *J* = 8.2 Hz), 7.92 (d, 1H, *J* = 8.0 Hz), 7.58 (t, 1H, *J* = 7.2 Hz), 7.52 (t, 1H, *J* = 8.01 Hz). **¹³C NMR** (150 MHz, Cl₃CD) δ : 157.7, 153.4, 138.7, 127.2, 127.1, 124.6, 121.5. **¹⁹F NMR** (564.603 MHz, Cl₃CD) δ : -80.7 (t, 3F), -84.9 (t, 2F), -118.8 (m, 2F), -121.1 (m, 2F), -121.7 (m, 2F), -121.9 (m, 2F), -122.7 (m, 2F), -126.1 (m, 2F). HRMS (ESI):*m/z* [*M*+H]⁺ calcd for C₁₅H₅F₁₇NS₂: 585.9514, found: 585.9515.

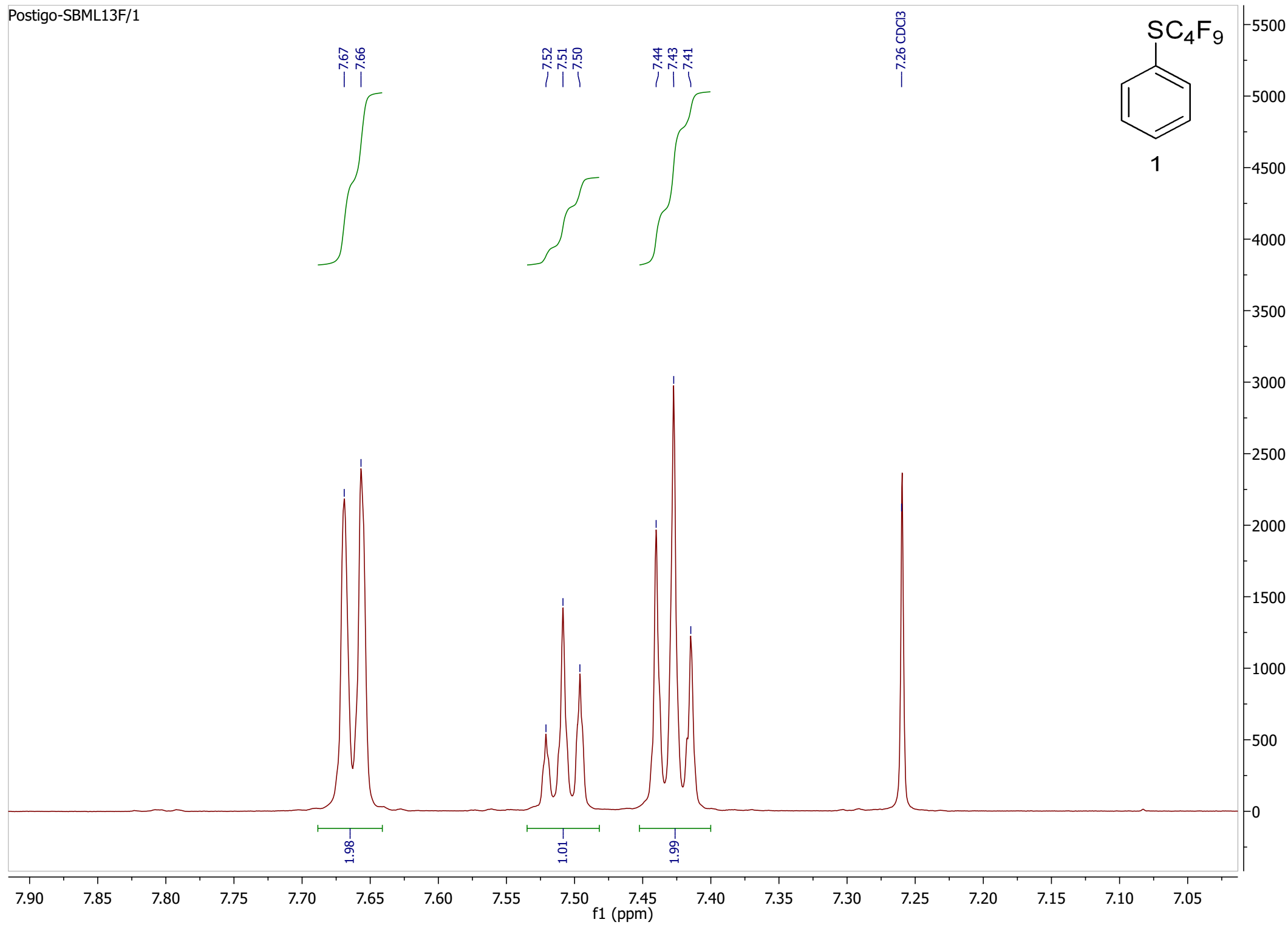


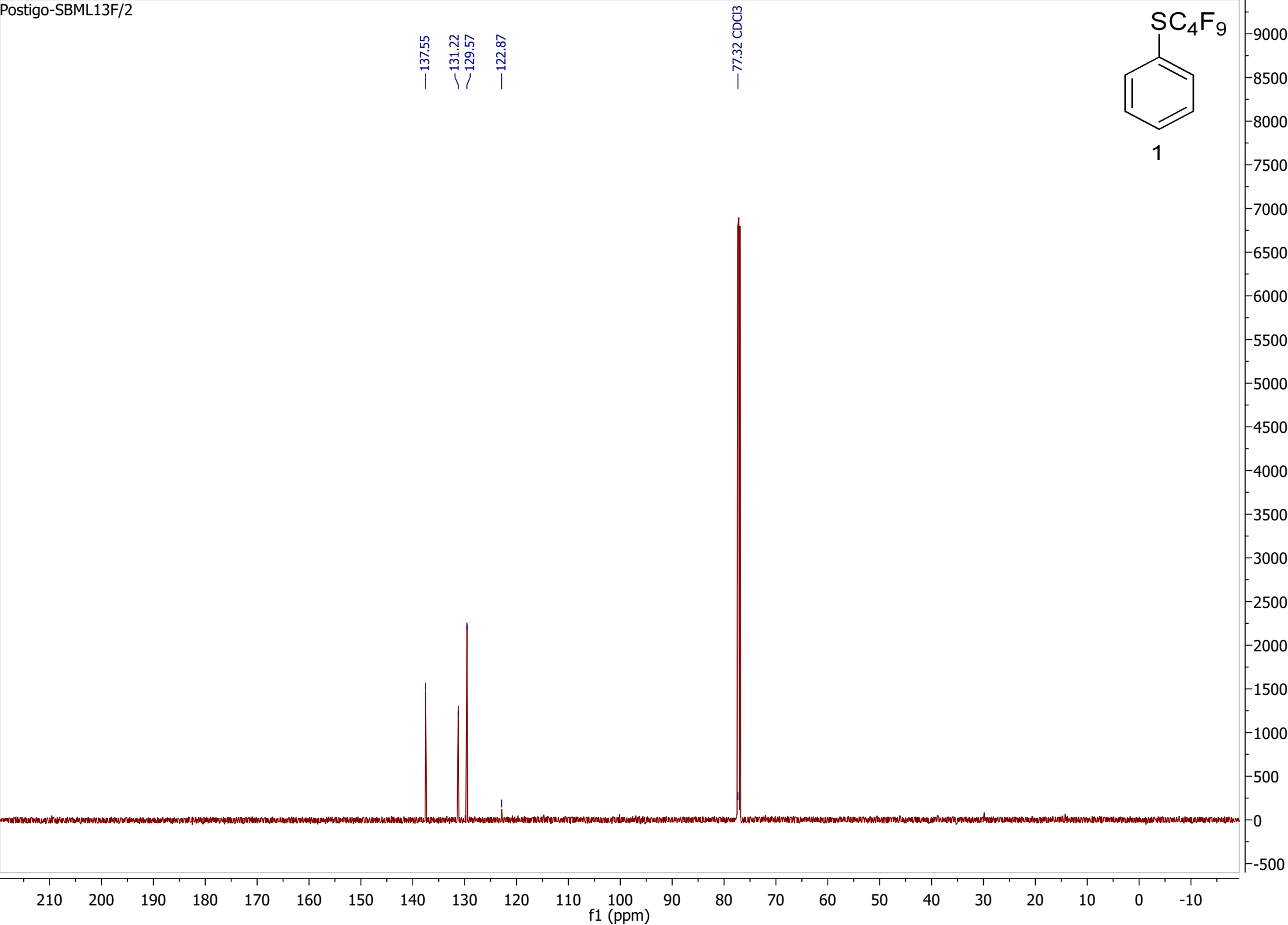
4-(perfluorobutyl)-2-((perfluorobutyl)thio)-1H-benzo[d]imidazole **22**: Yield: 50 %. Isolated and purified mass

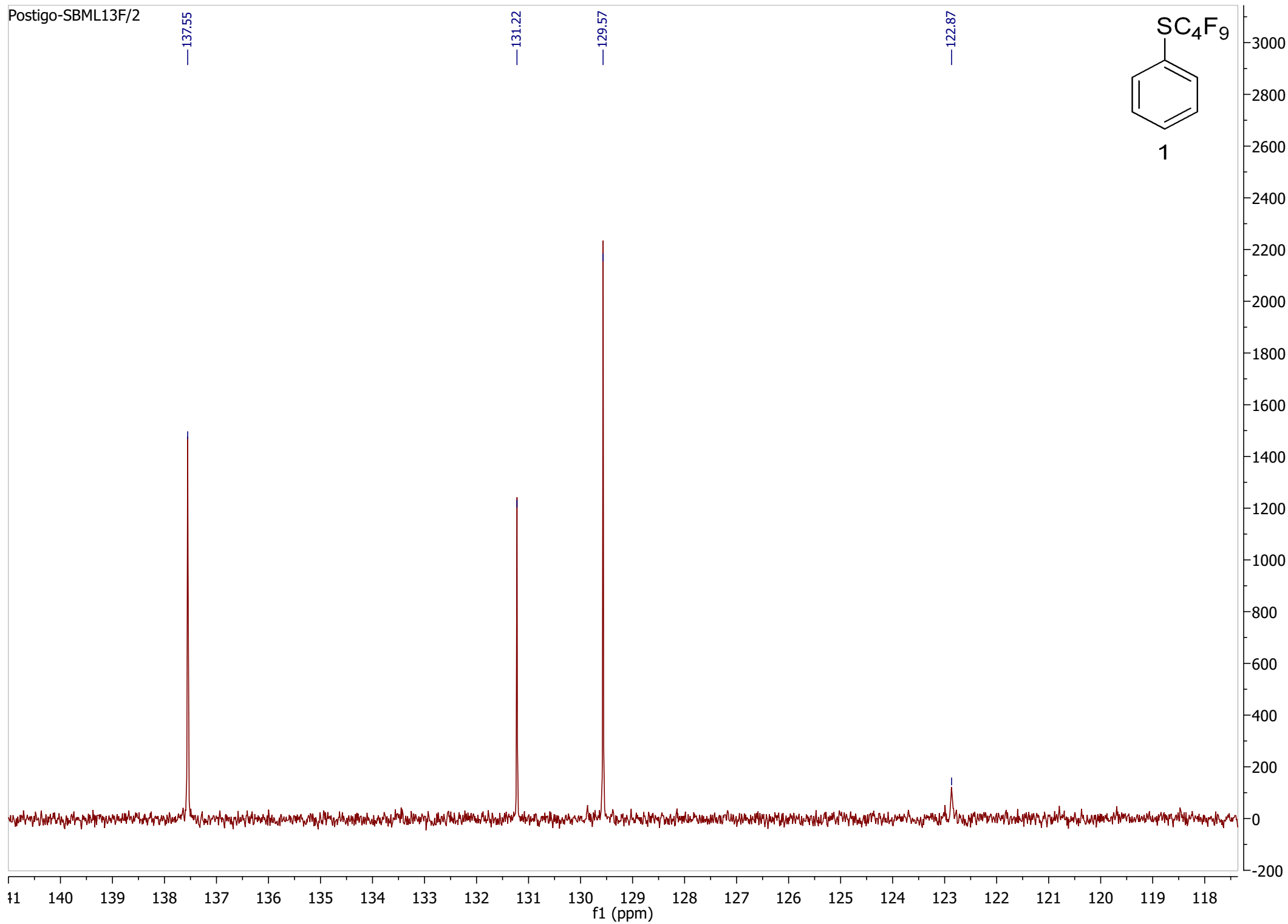
obtained: 15 mg. TLC (CHCl₃: hexane 7:3 v/v): R_f = 0.5

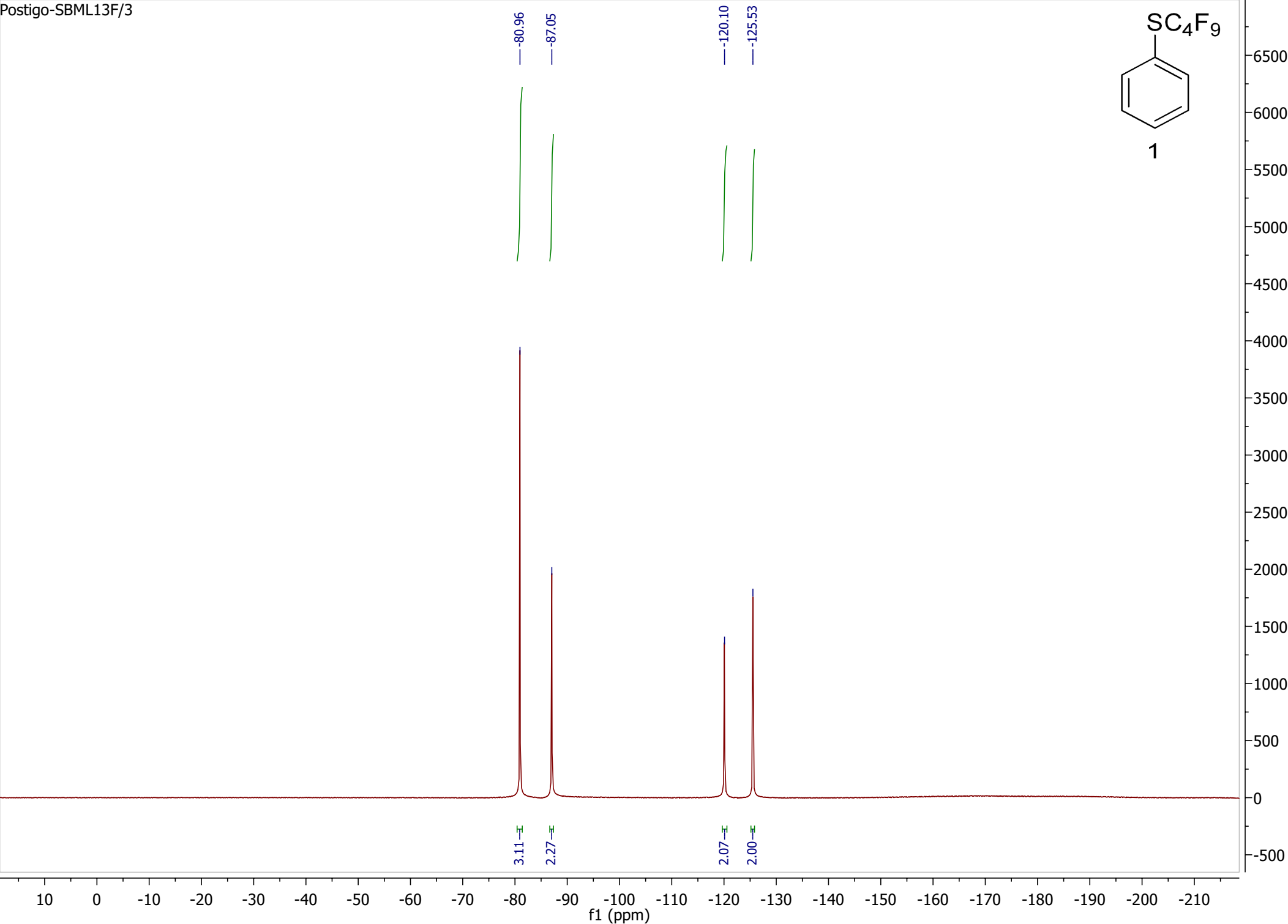
¹H-NMR (600 MHz, Cl₃CD) δ : 9.87 (bs, 1H), 8.08 (d, 1H, *J* = 8.1 Hz), 7.61 (d, 1H, *J* = 7.7 Hz), 7.49 (t, 1H, *J* = 8.0 Hz). **¹³C NMR** (150 MHz, Cl₃CD) δ : 145.3, 137.3, 132.6, 125.5, 125.1, 123.6, 122.9. **¹⁹F NMR** (564.603 MHz, Cl₃CD) δ : -81.0 (m, 6F), -84.7 (t, 2F), -109.9 (t, 2F), -120.0 (m, 2F), -123.1 (m, 2F), -125.6 (m, 4F). HRMS (ESI): *m/z* [*M*+H]⁺ calcd for C₁₅H₅F₁₈N₂S: 586.9808, found: 586.9815.

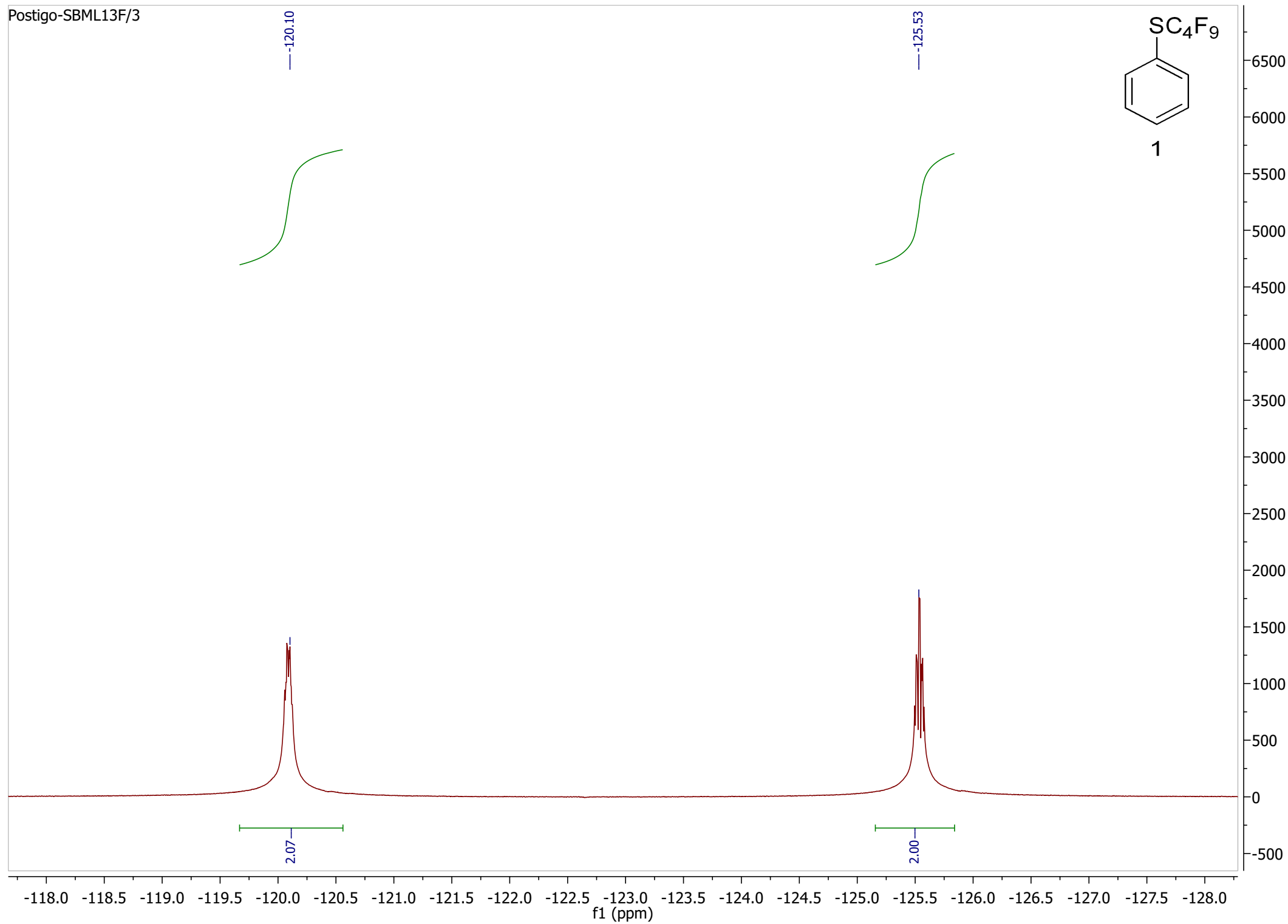


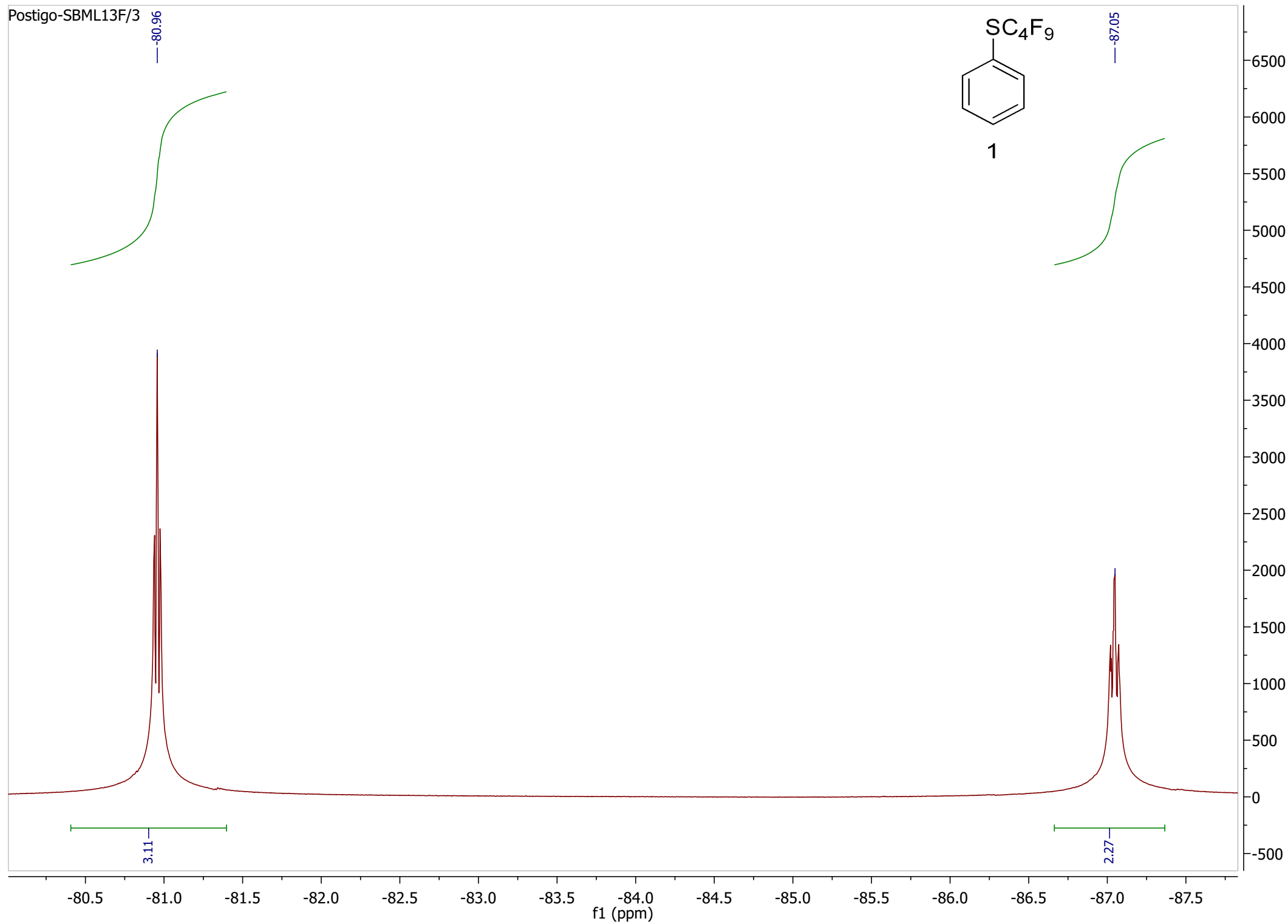


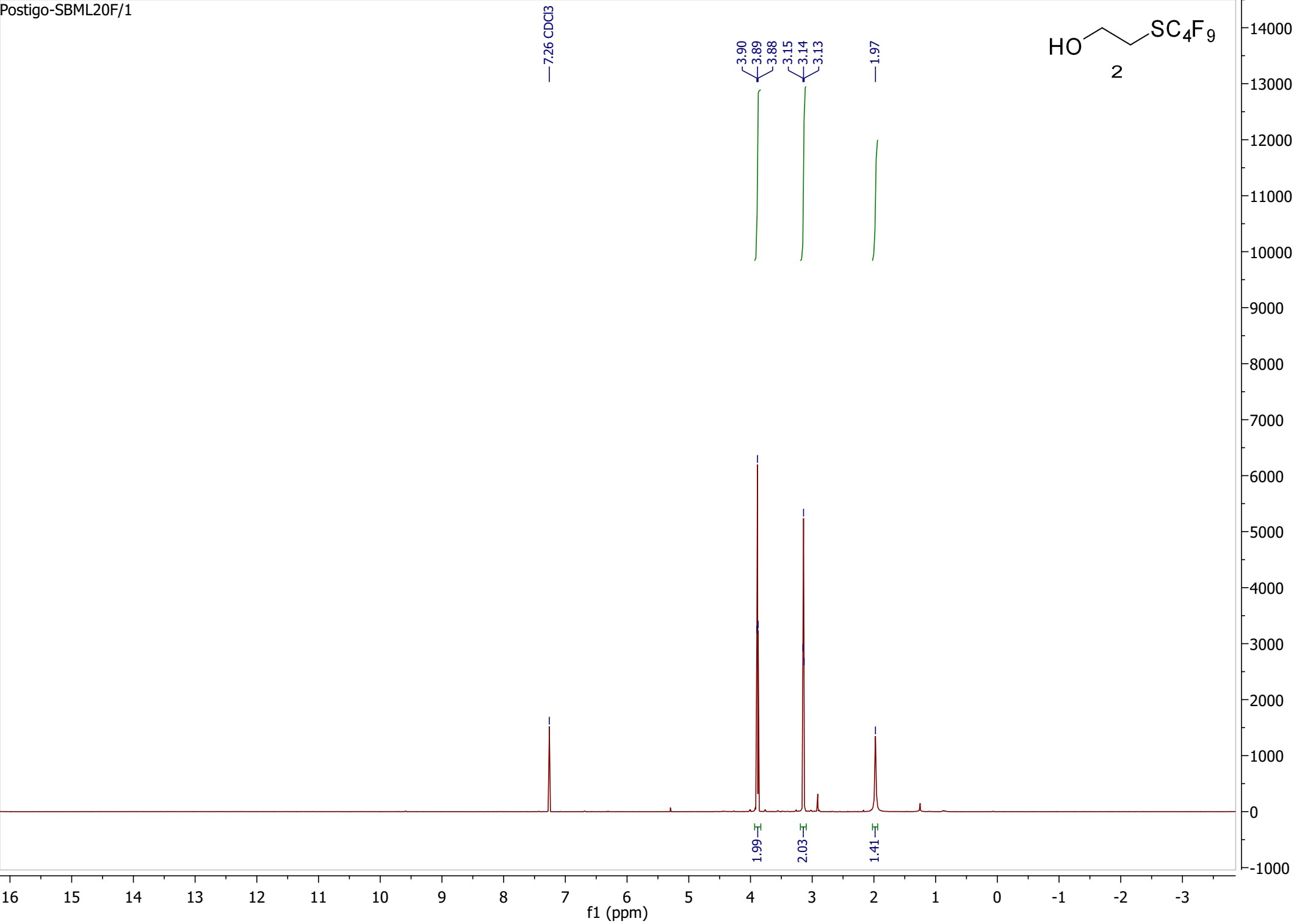


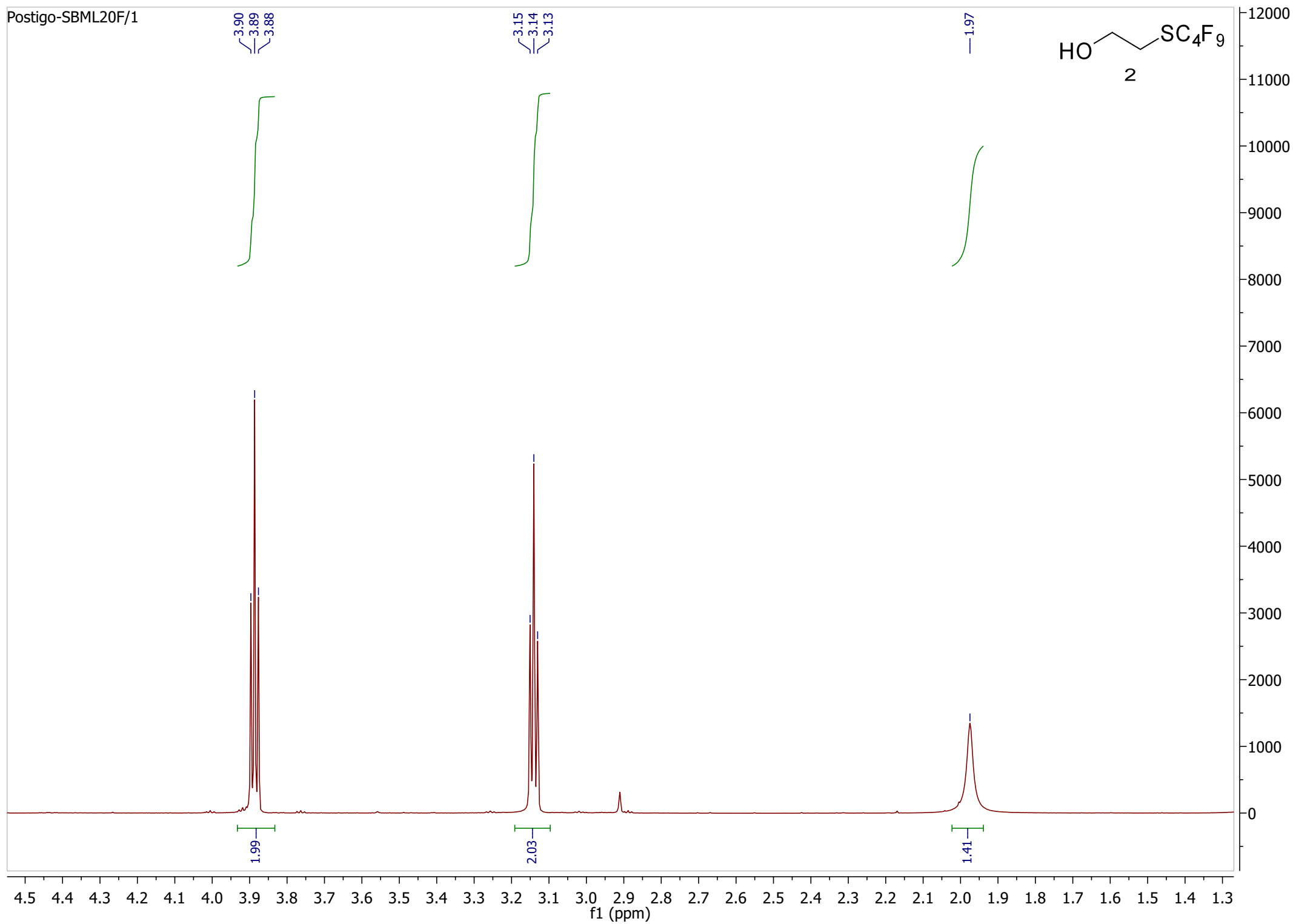


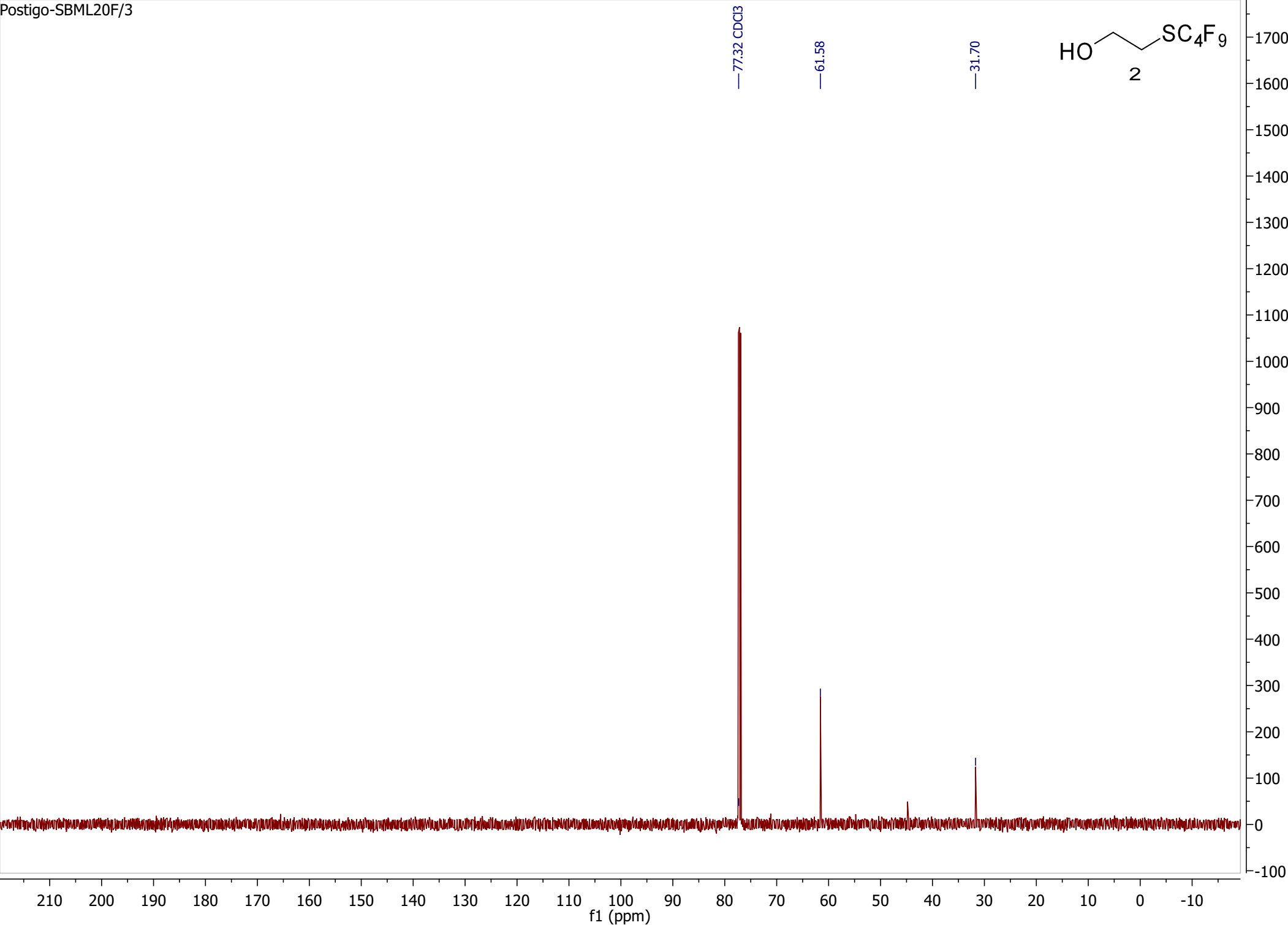


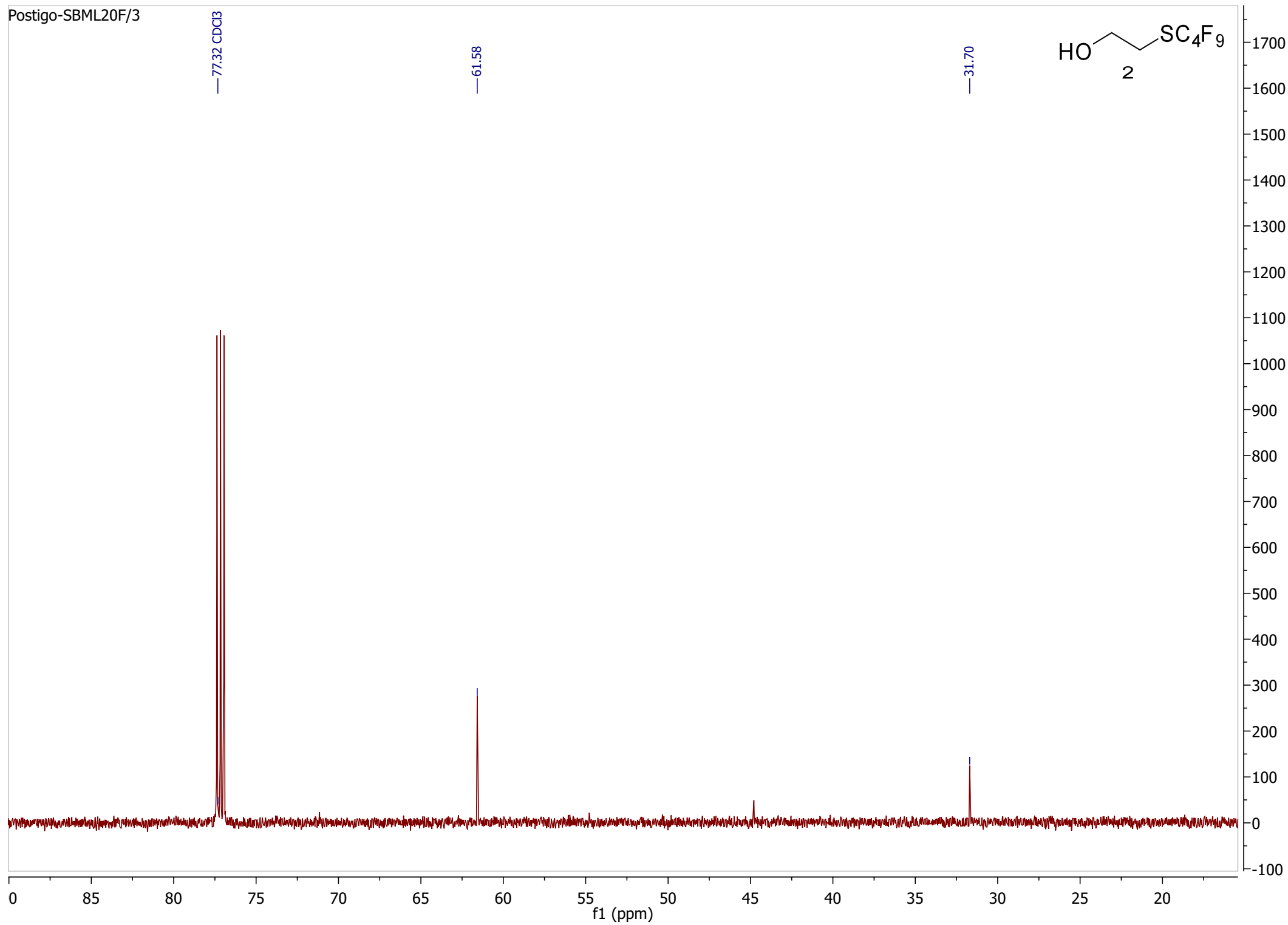


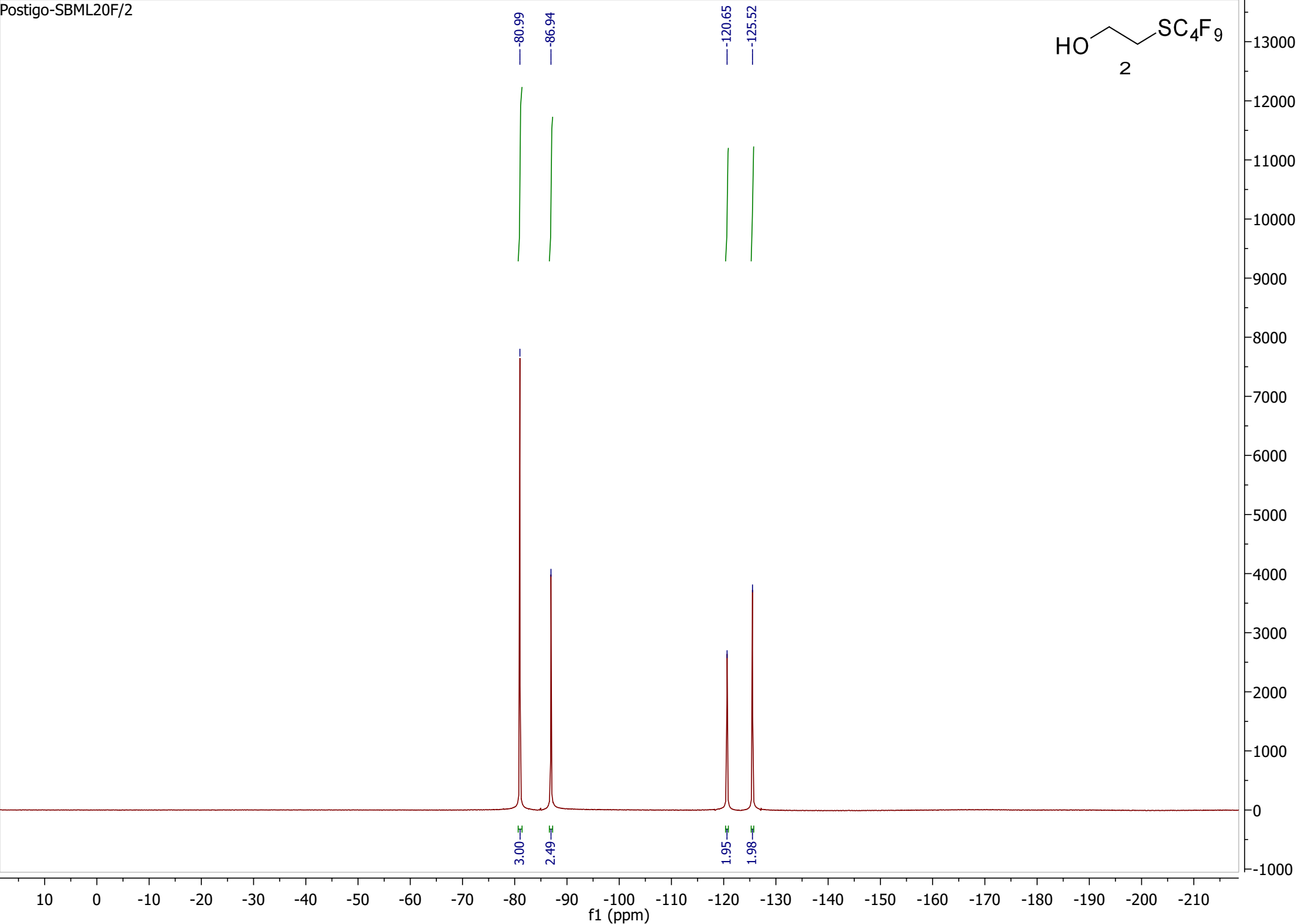


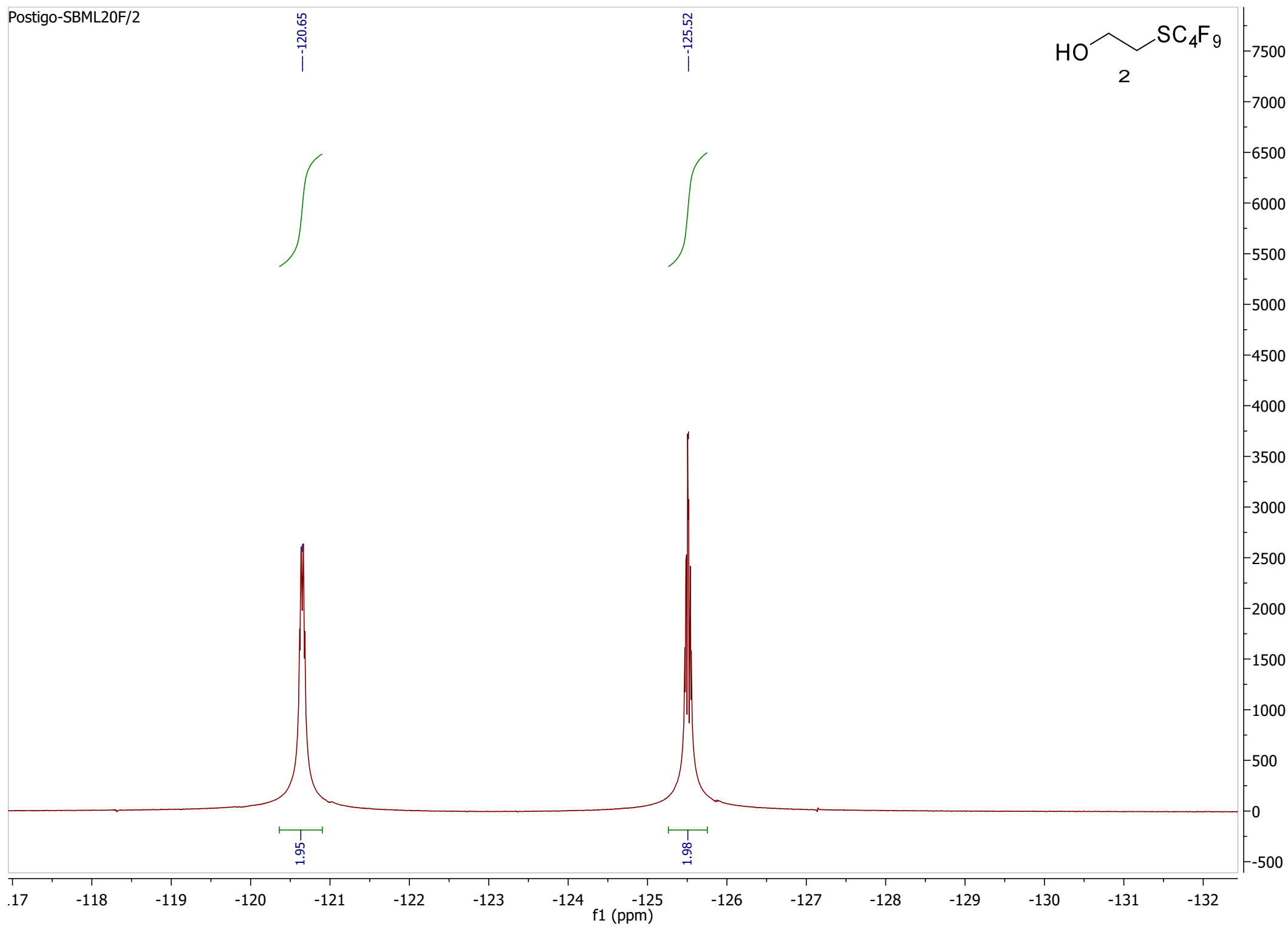


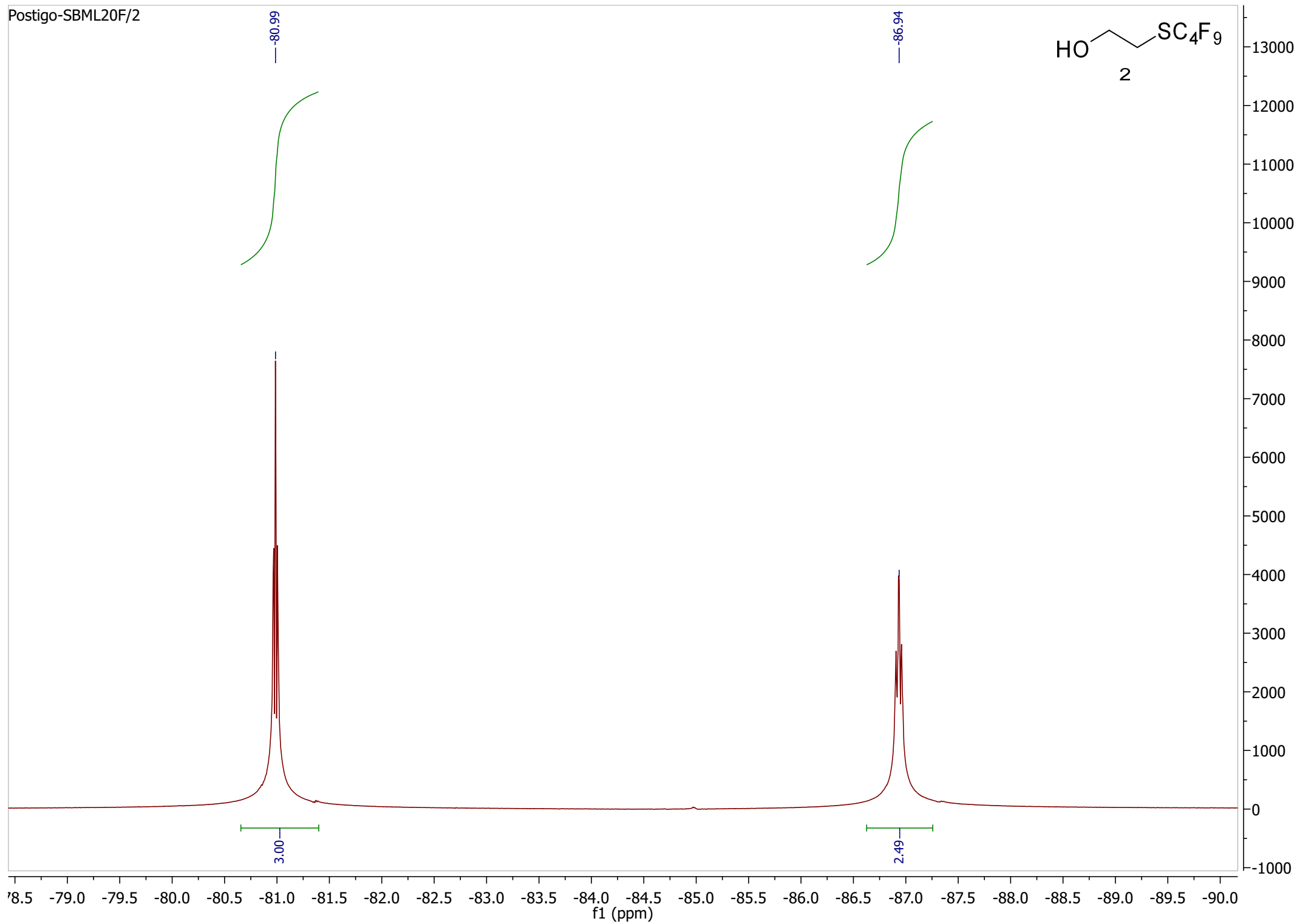




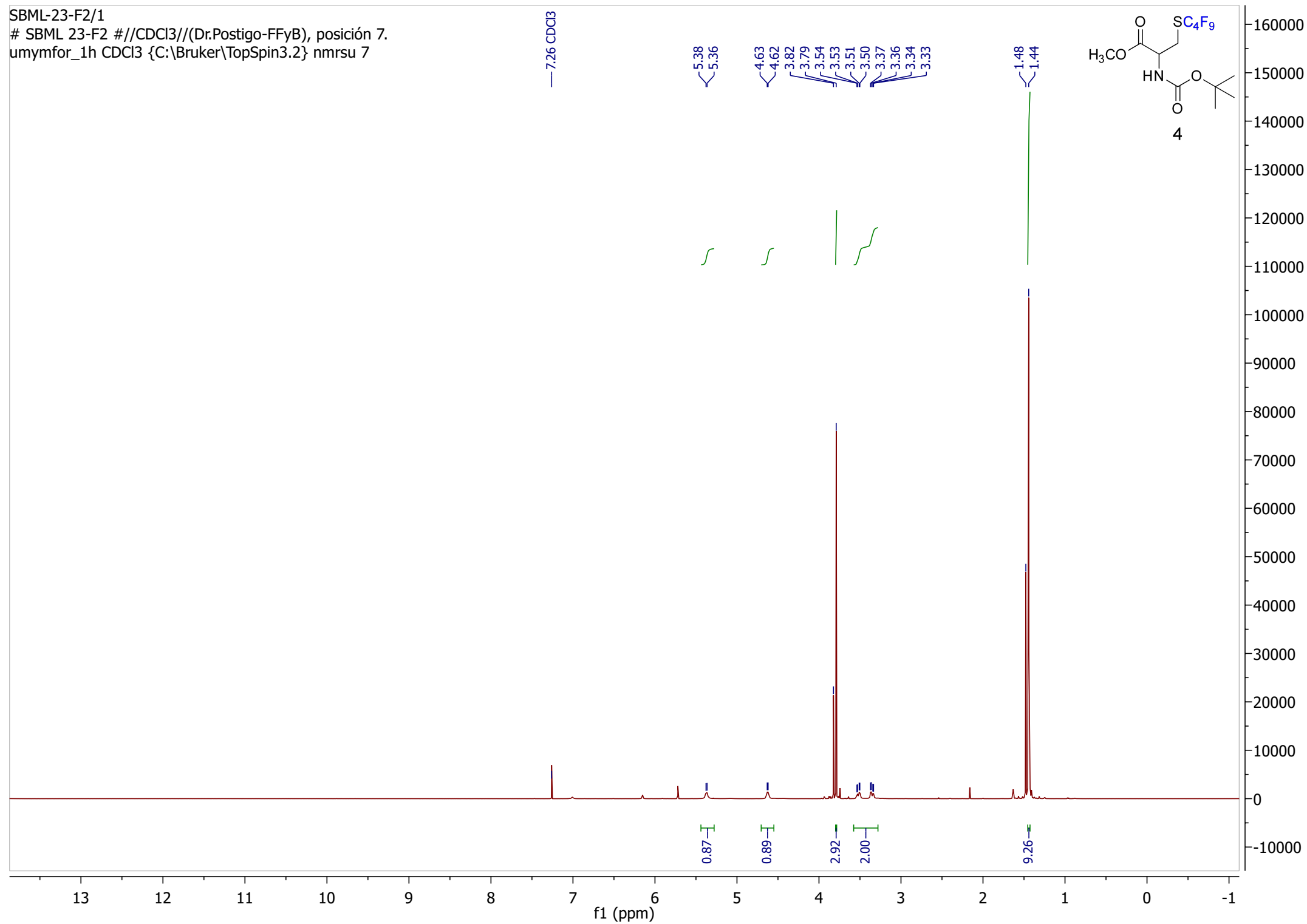




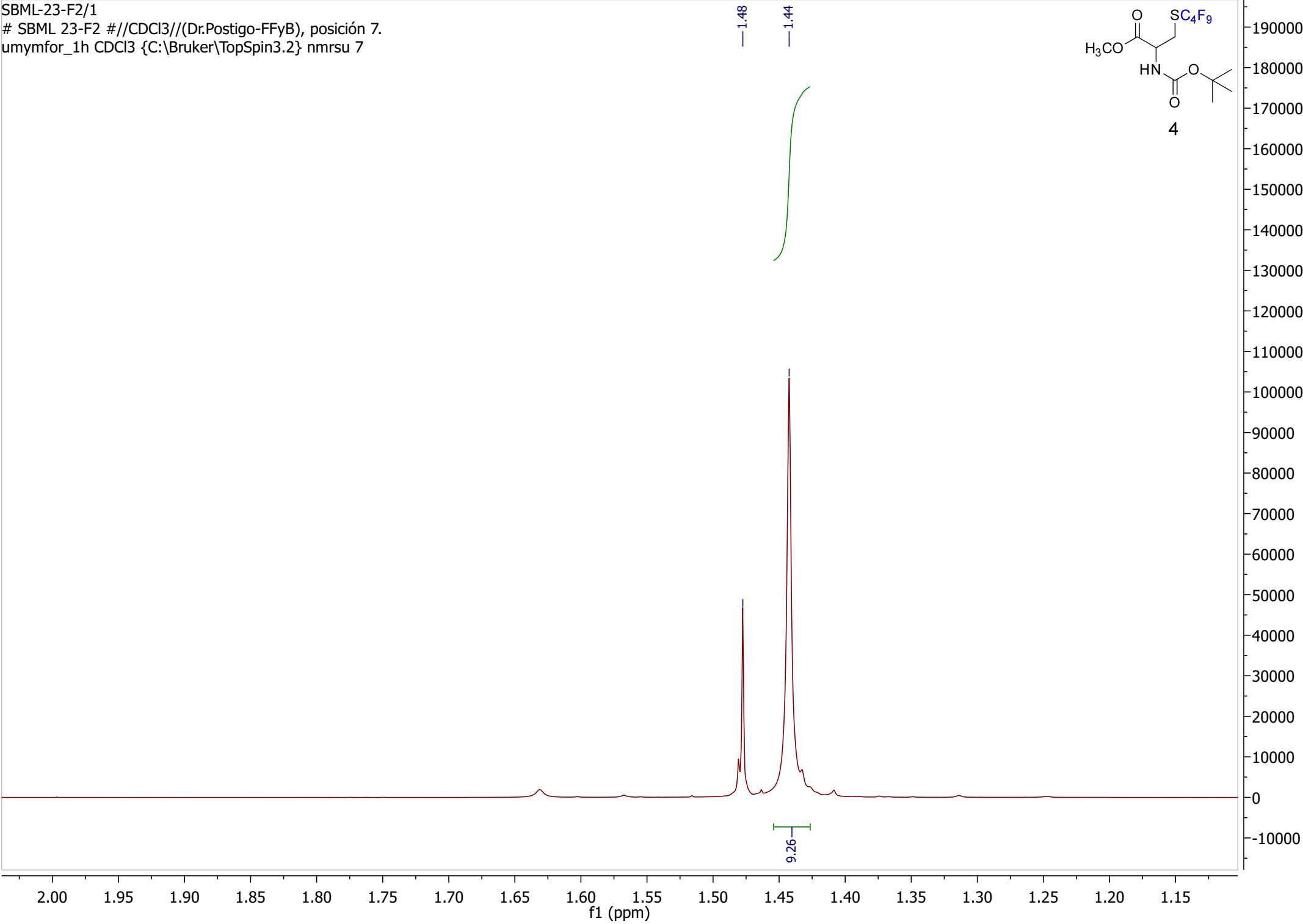
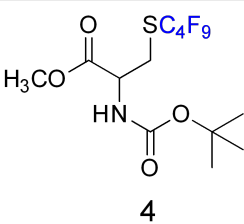




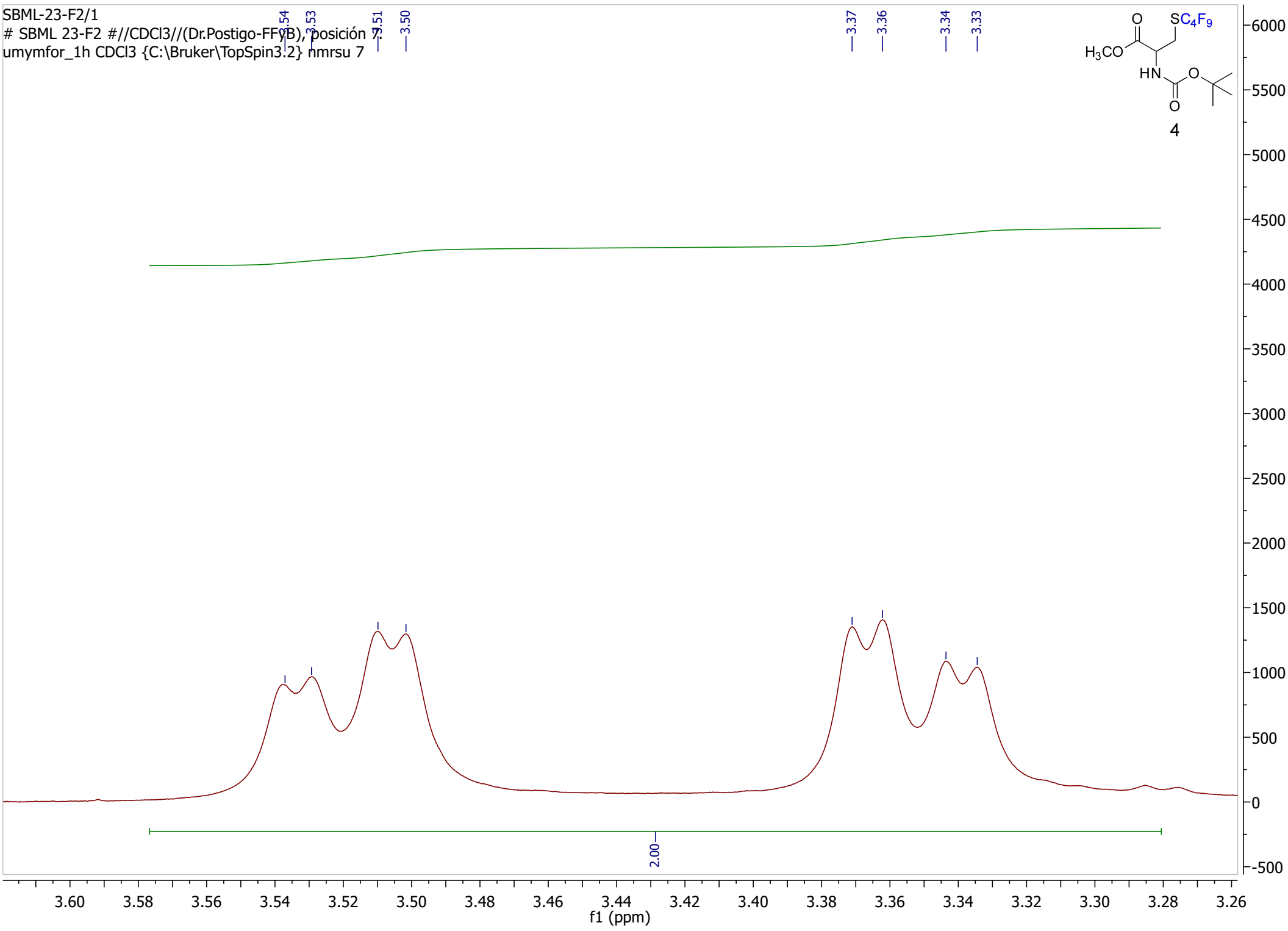
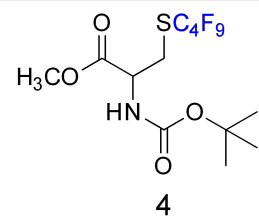
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umymfor_1h CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 7



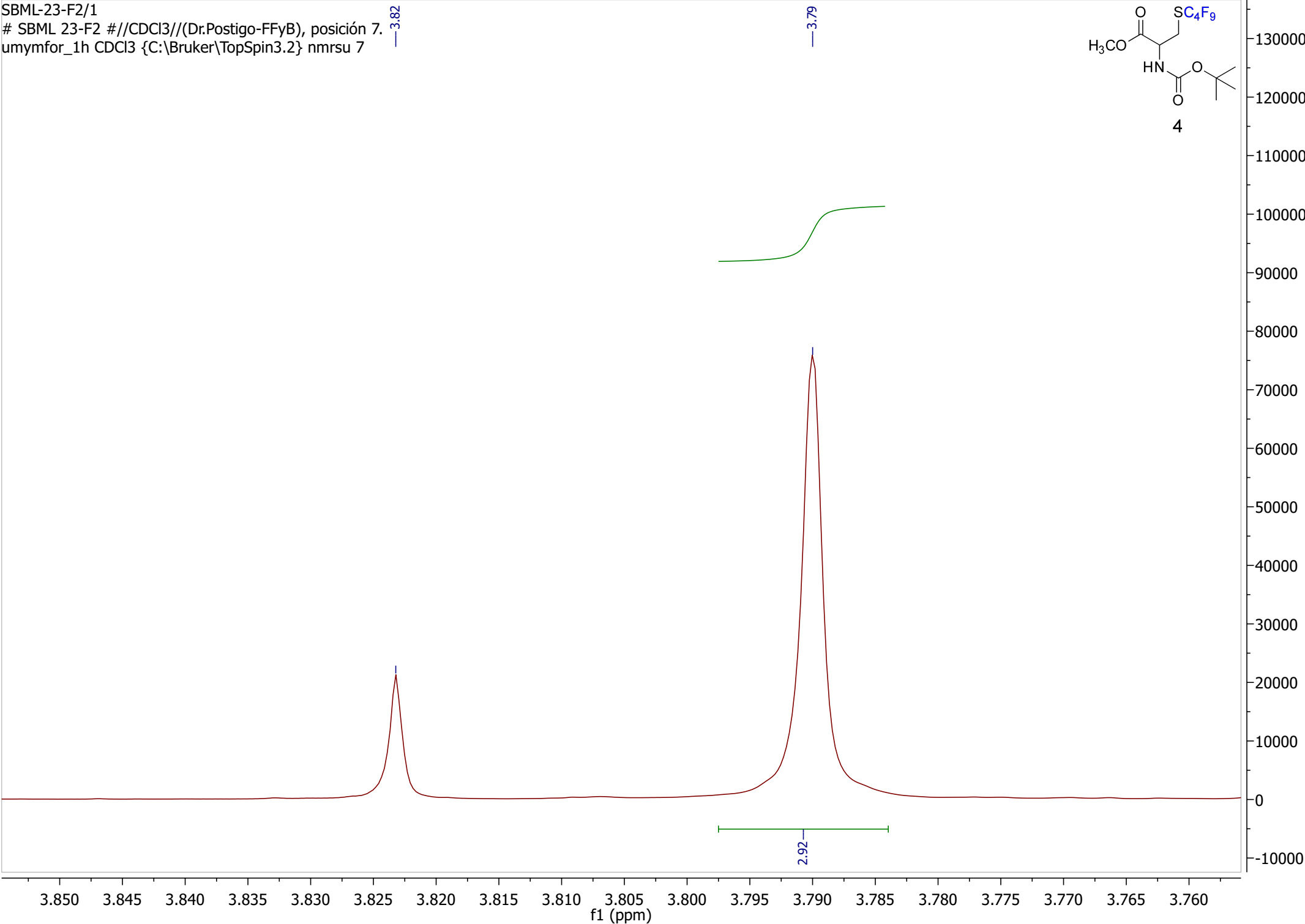
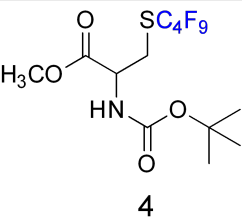
SBML-23-F2/1
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umymfor_1h CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 7



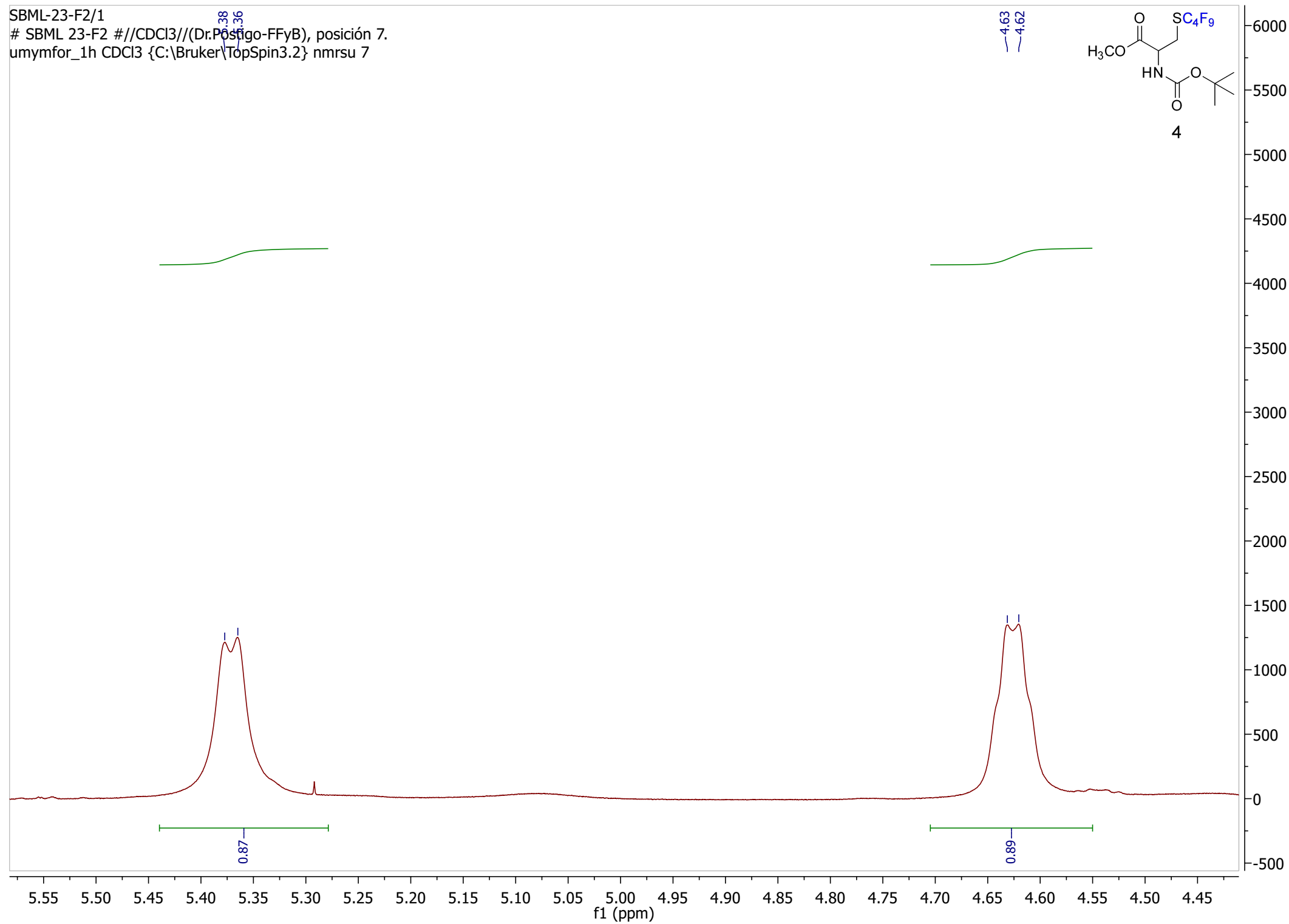
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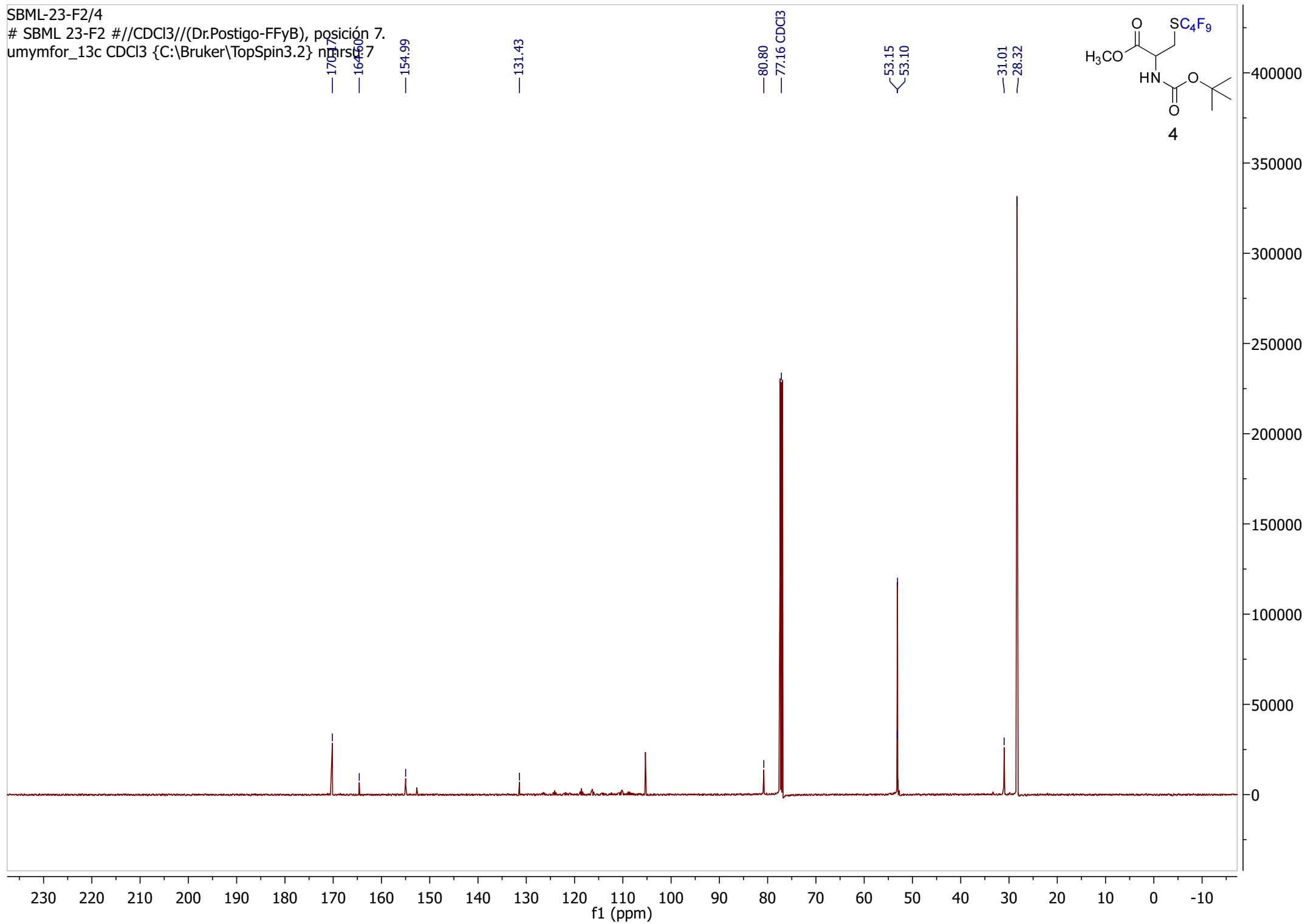
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umymfor_1h CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 7



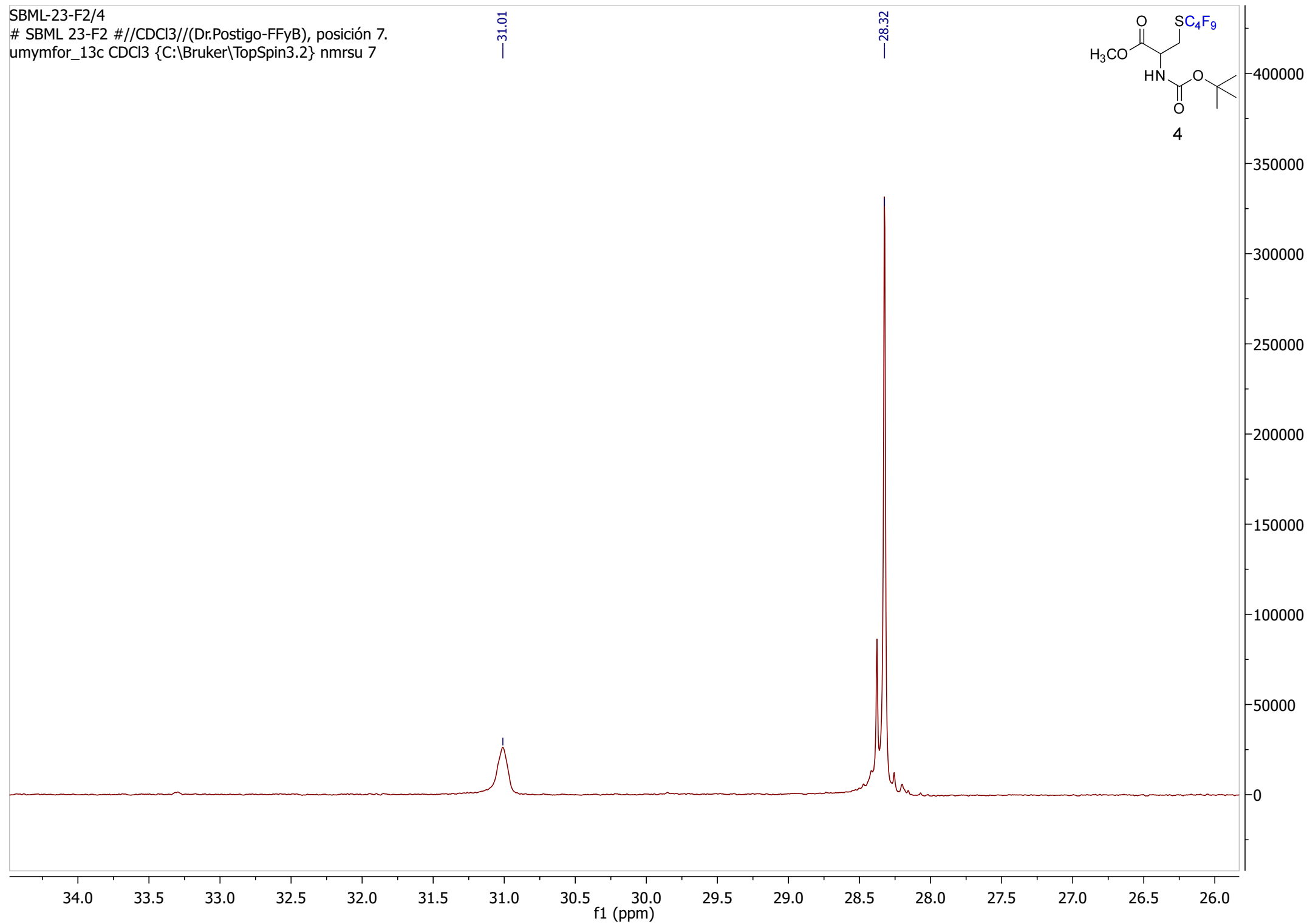
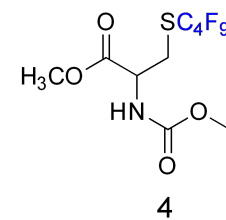
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umymfor_1h CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 7



SBML-23-F2/4
SBML 23-F2 #//CDCl3//((Dr.Postigo-FFyB), posición 7.
umymfor_13c CDCl3 {C:\Bruker\TopSpin3.2} nmrs117

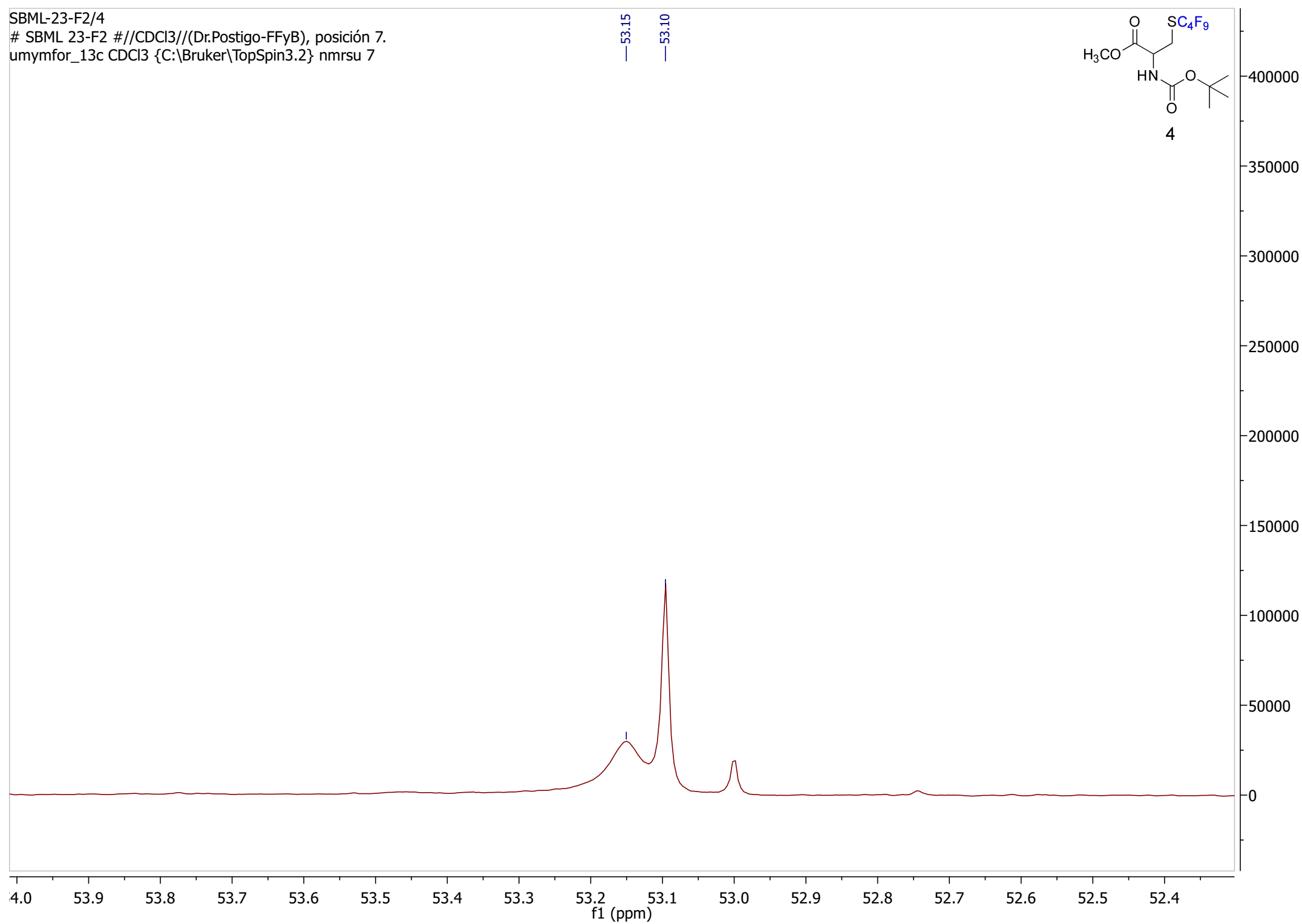
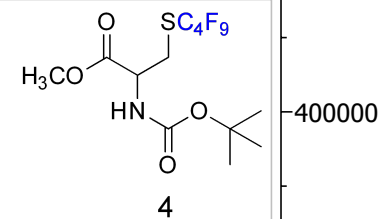


SBML-23-F2/4
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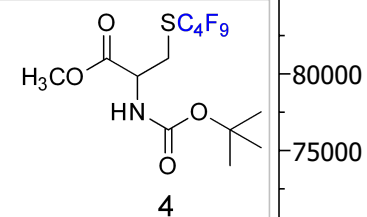
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— 53.15
— 53.10



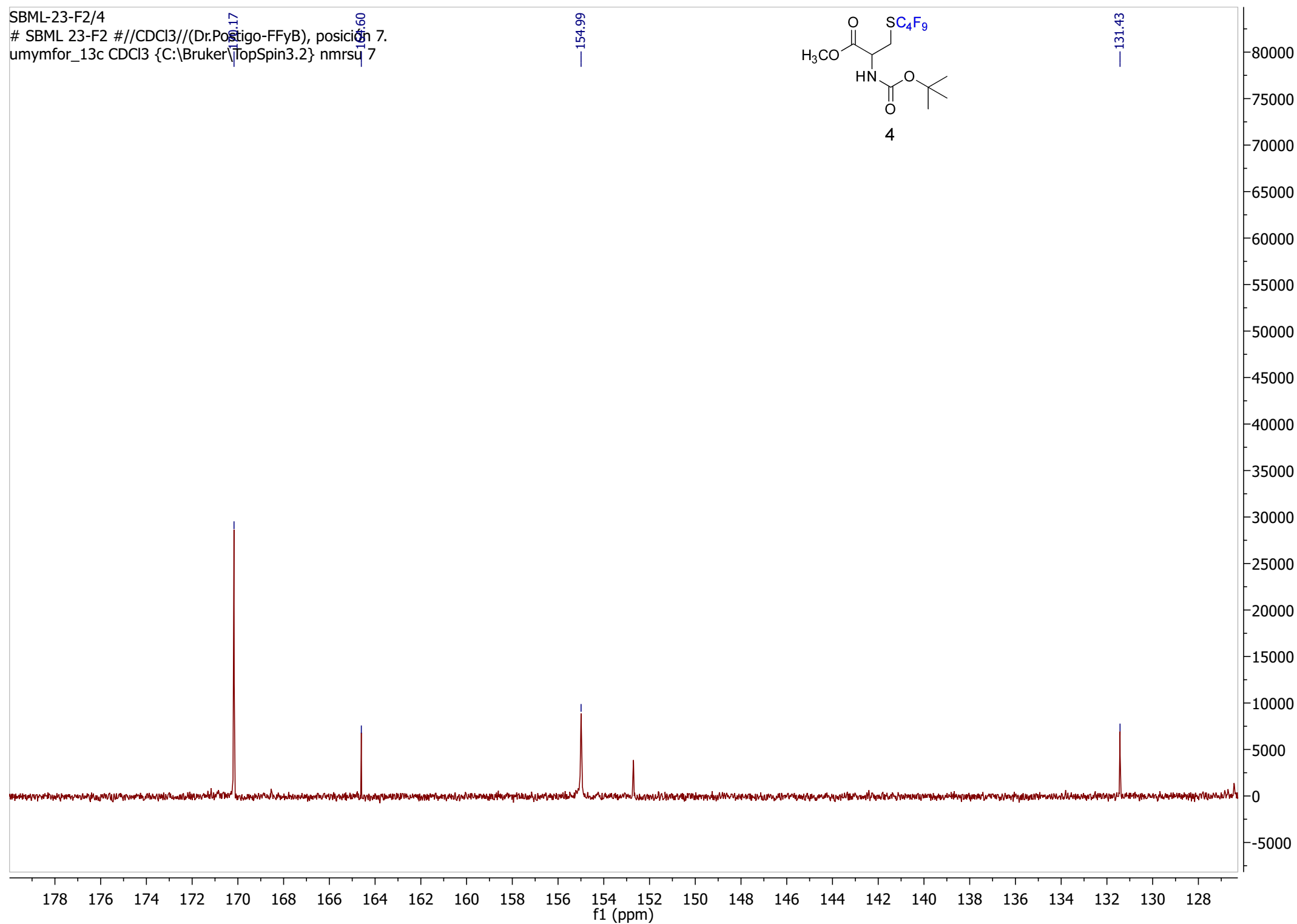
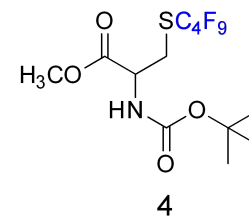
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80.08

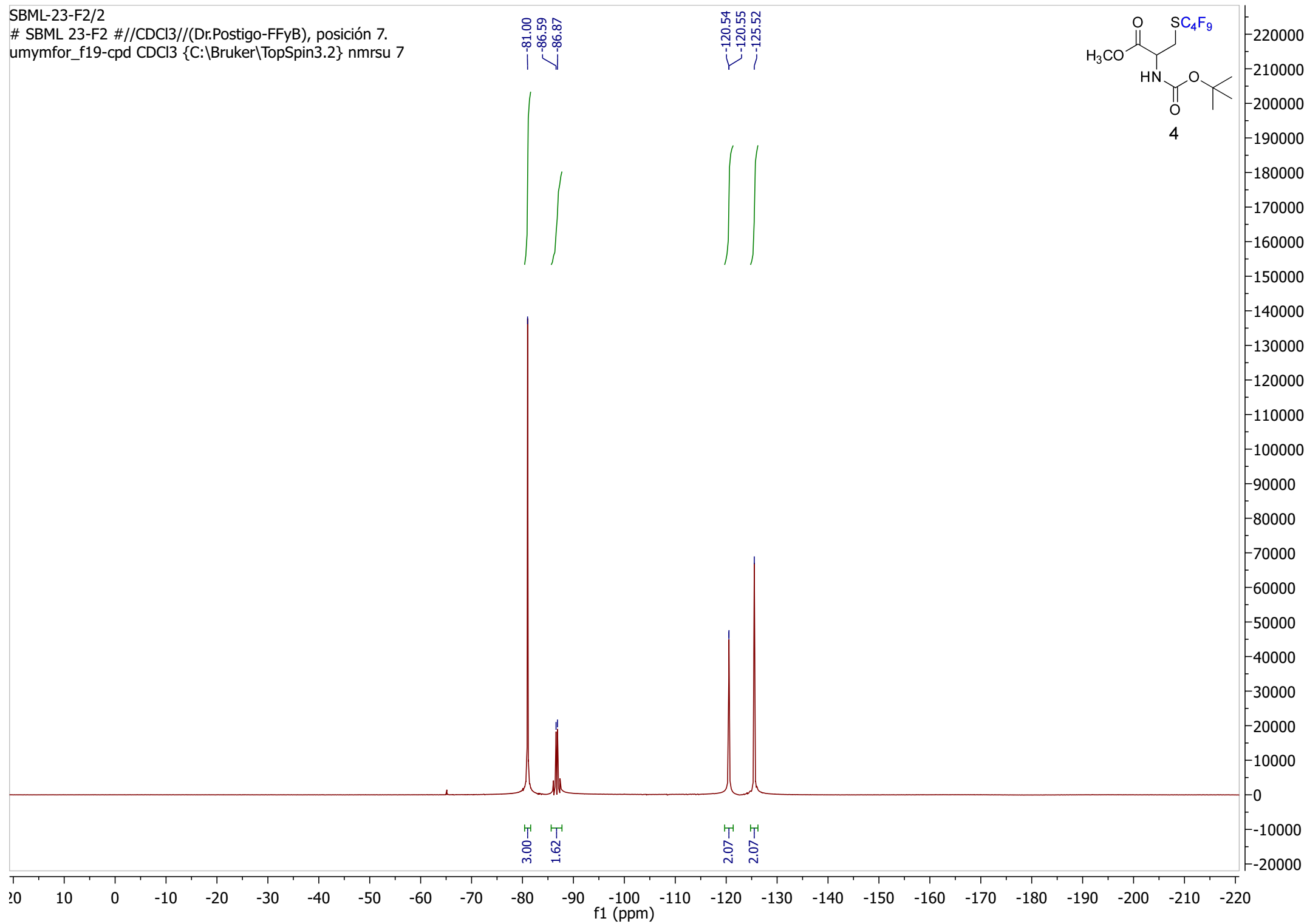


3.6 83.4 83.2 83.0 82.8 82.6 82.4 82.2 82.0 81.8 81.6 81.4 81.2 81.0 80.8 80.6 80.4 80.2 80.0 79.8 79.6 79.4 79.2 79.0 78.8 78.6 78.4 78.2 78.0 77.8
f1 (ppm)

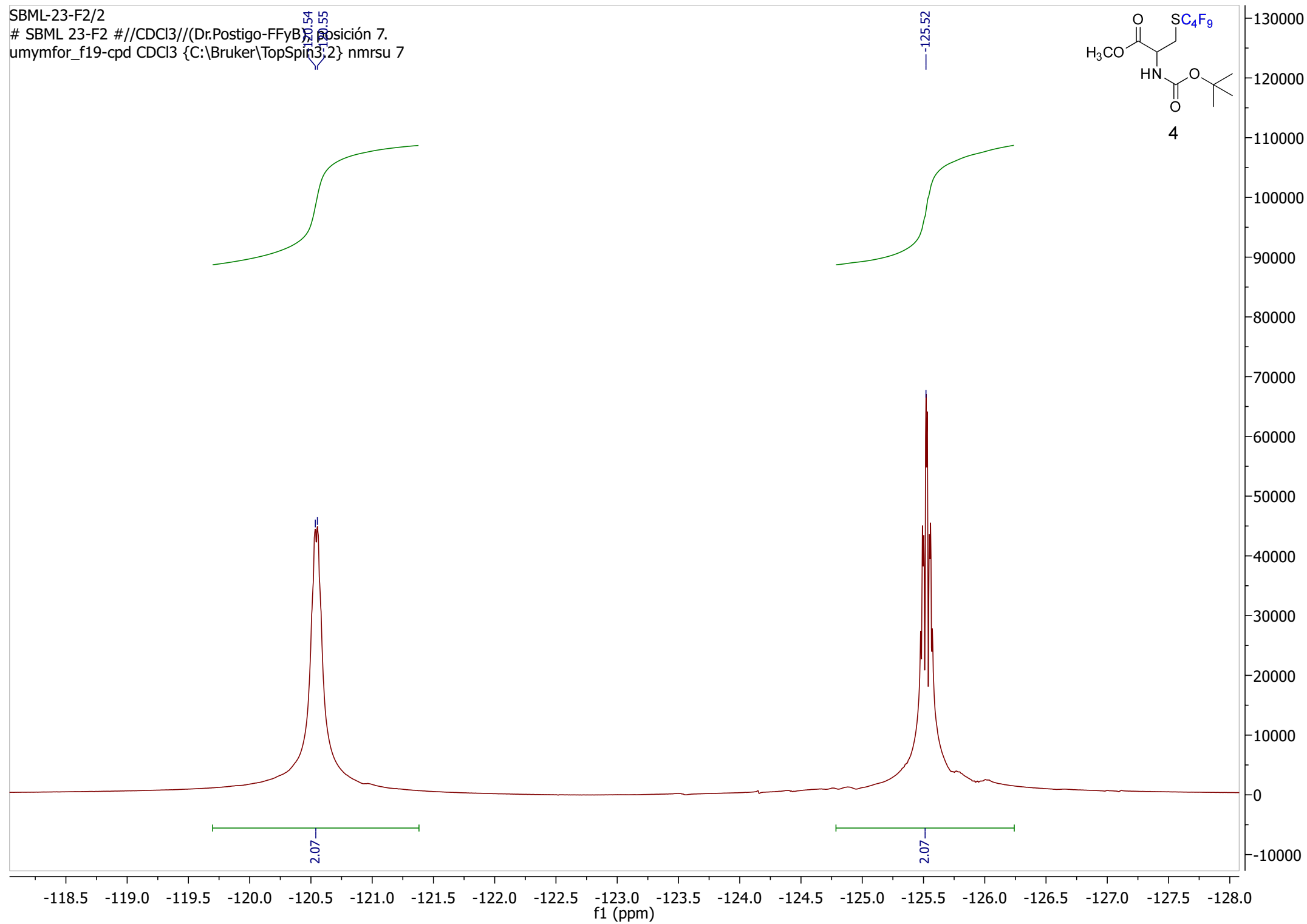
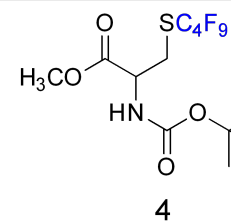
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umymfor_13c CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 7



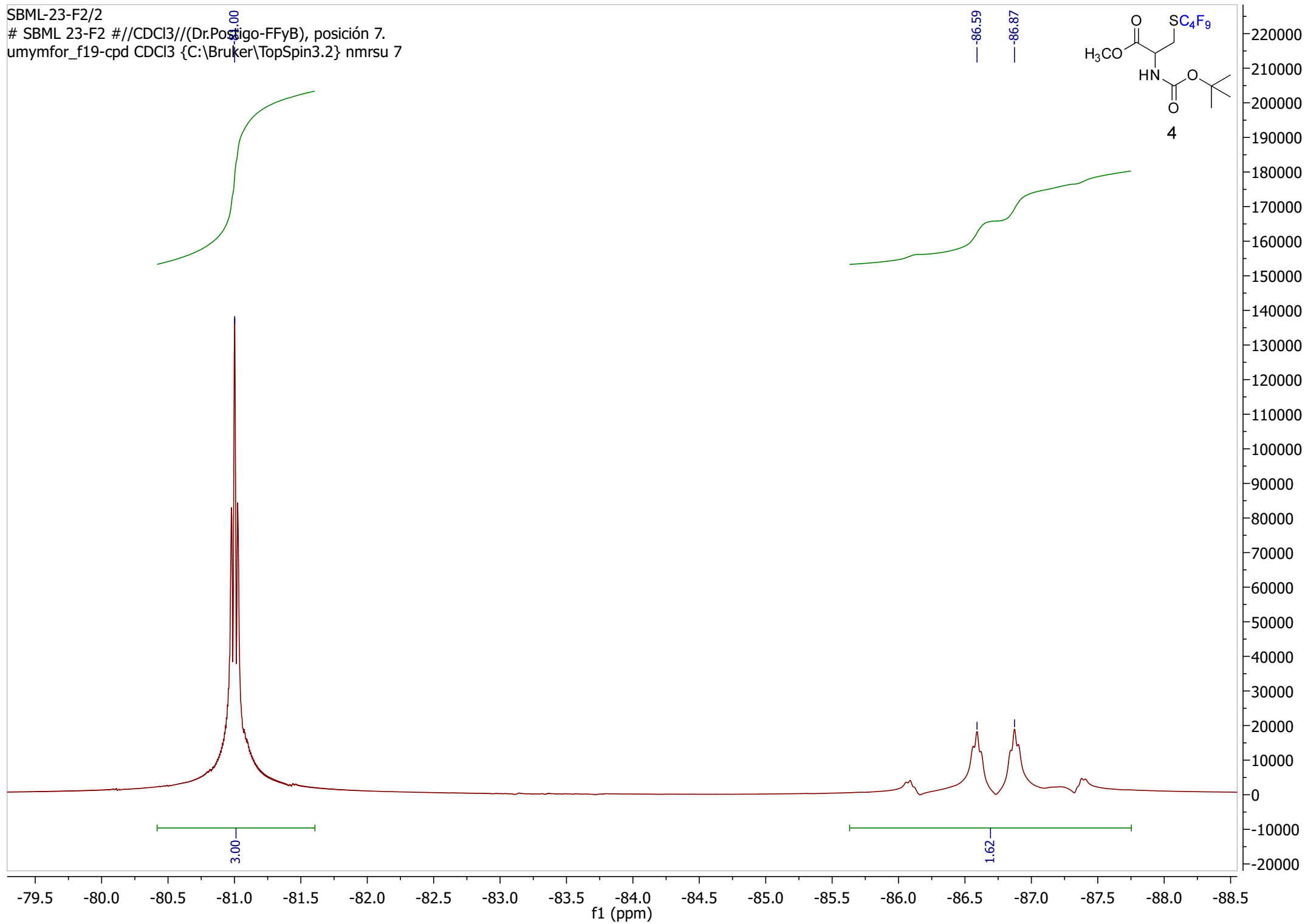
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umymfor_f19-cpd CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 7

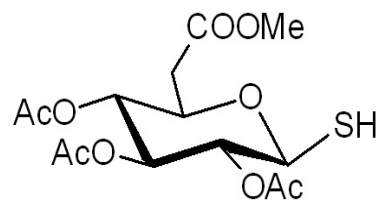


SBML-23-F2/2
SBML 23-F2 #//CDCl3//
(Dr.Postigo-FFyB) posición 7.
umymfor_f19-cpd CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 7

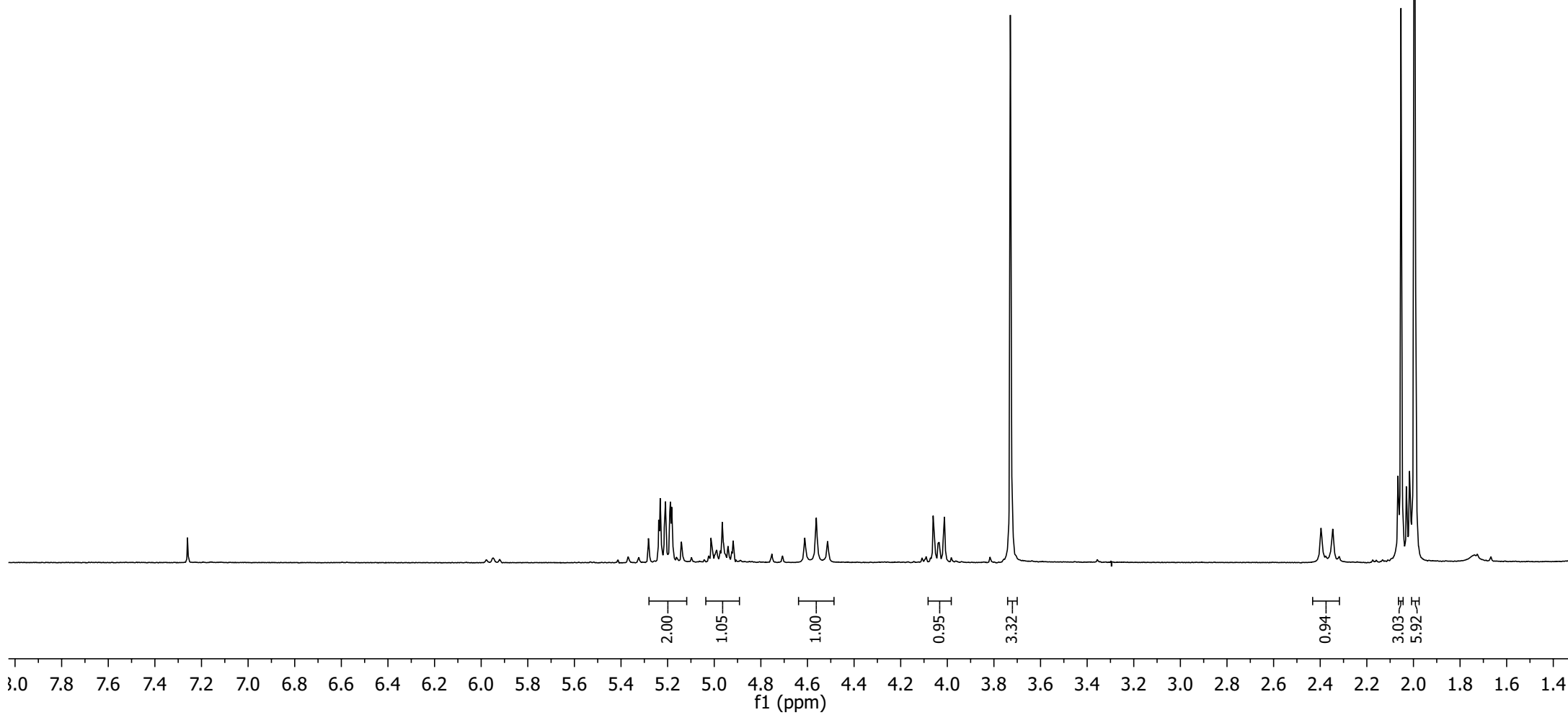


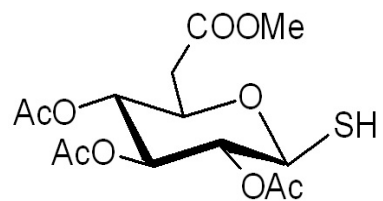
SBML-23-F2/2
SBML 23-F2 #//CDCl3//(Dr.Posigo-FFyB), posición 7.
umymfor_f19-cpd CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 7



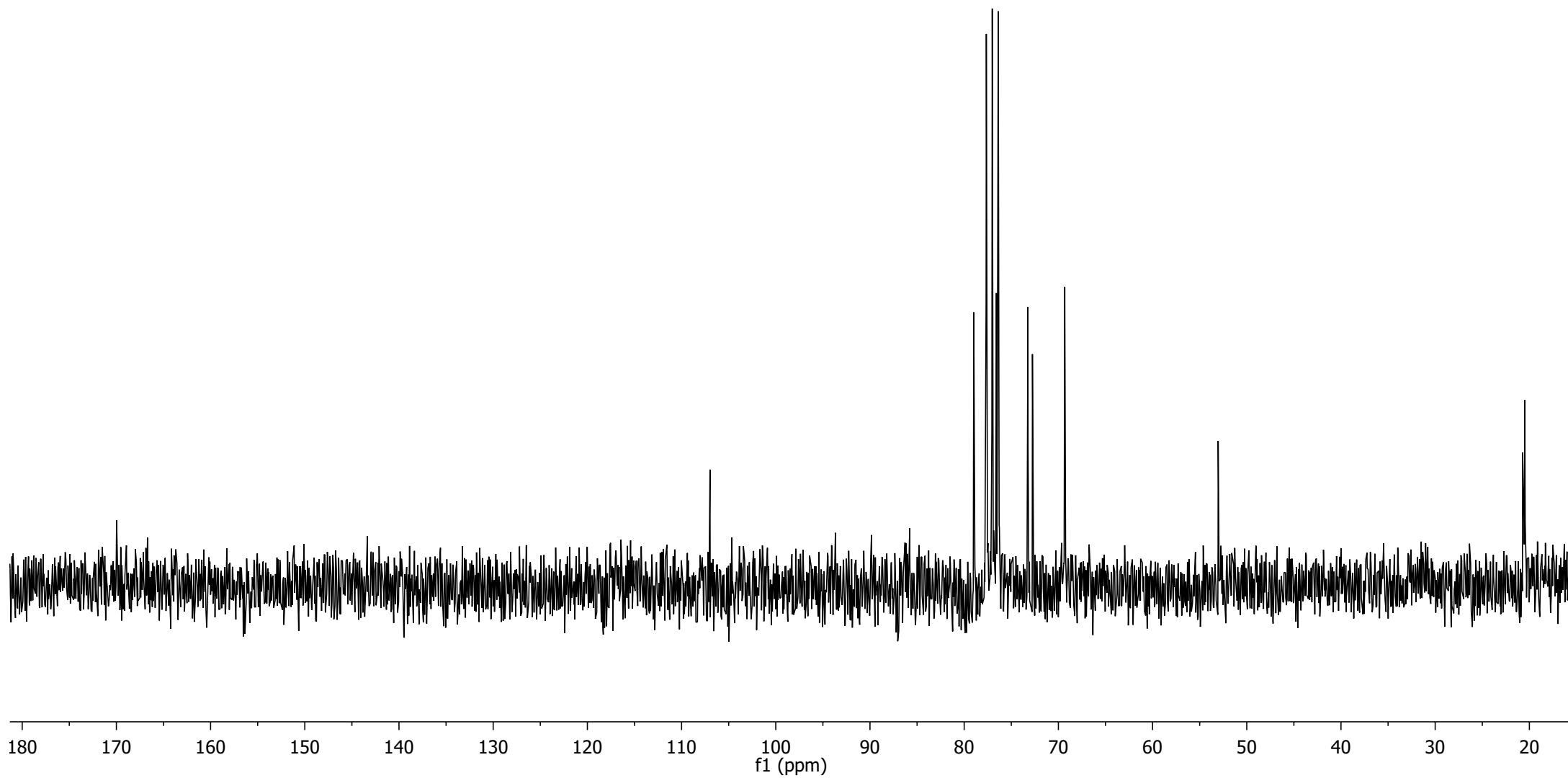


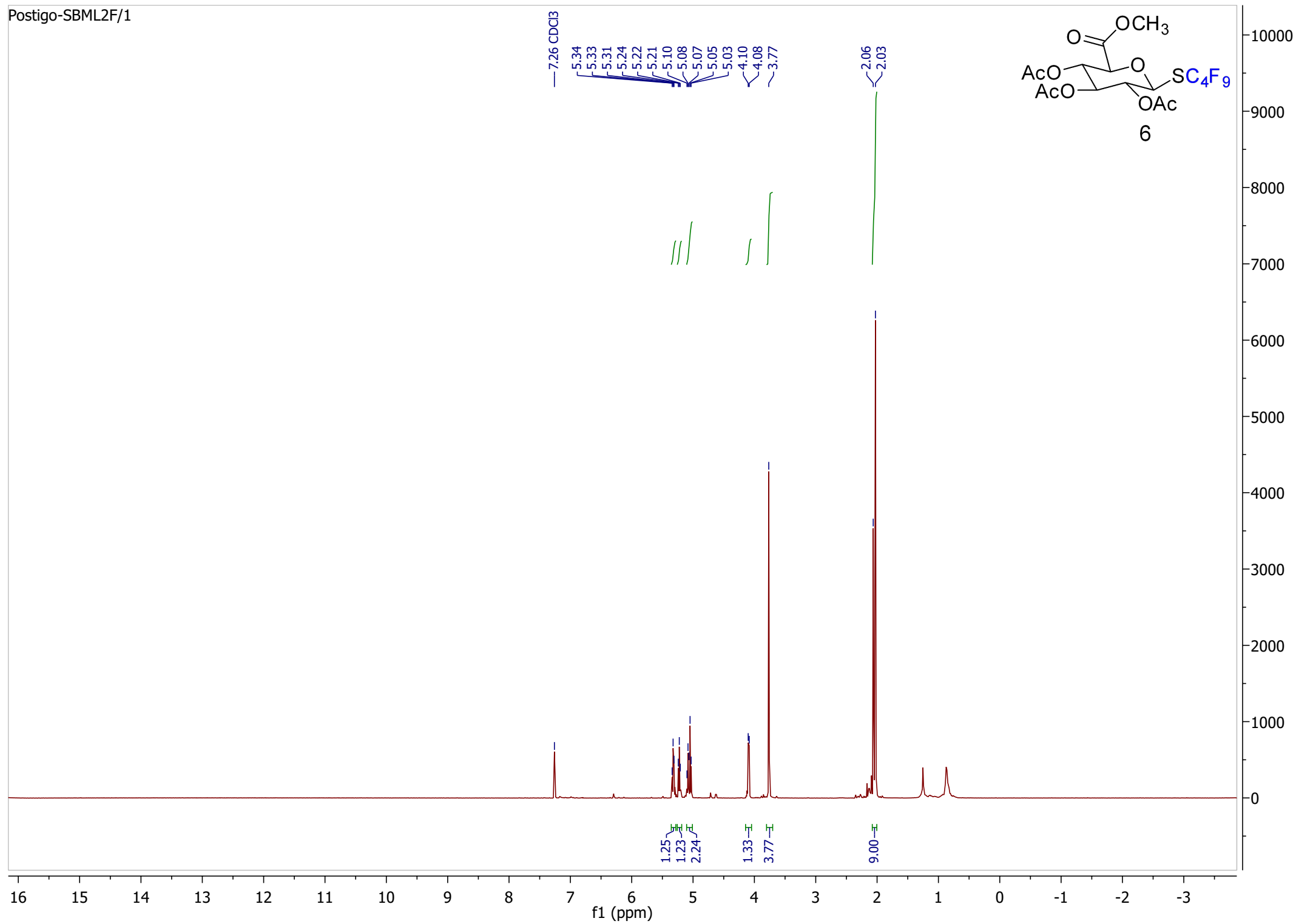
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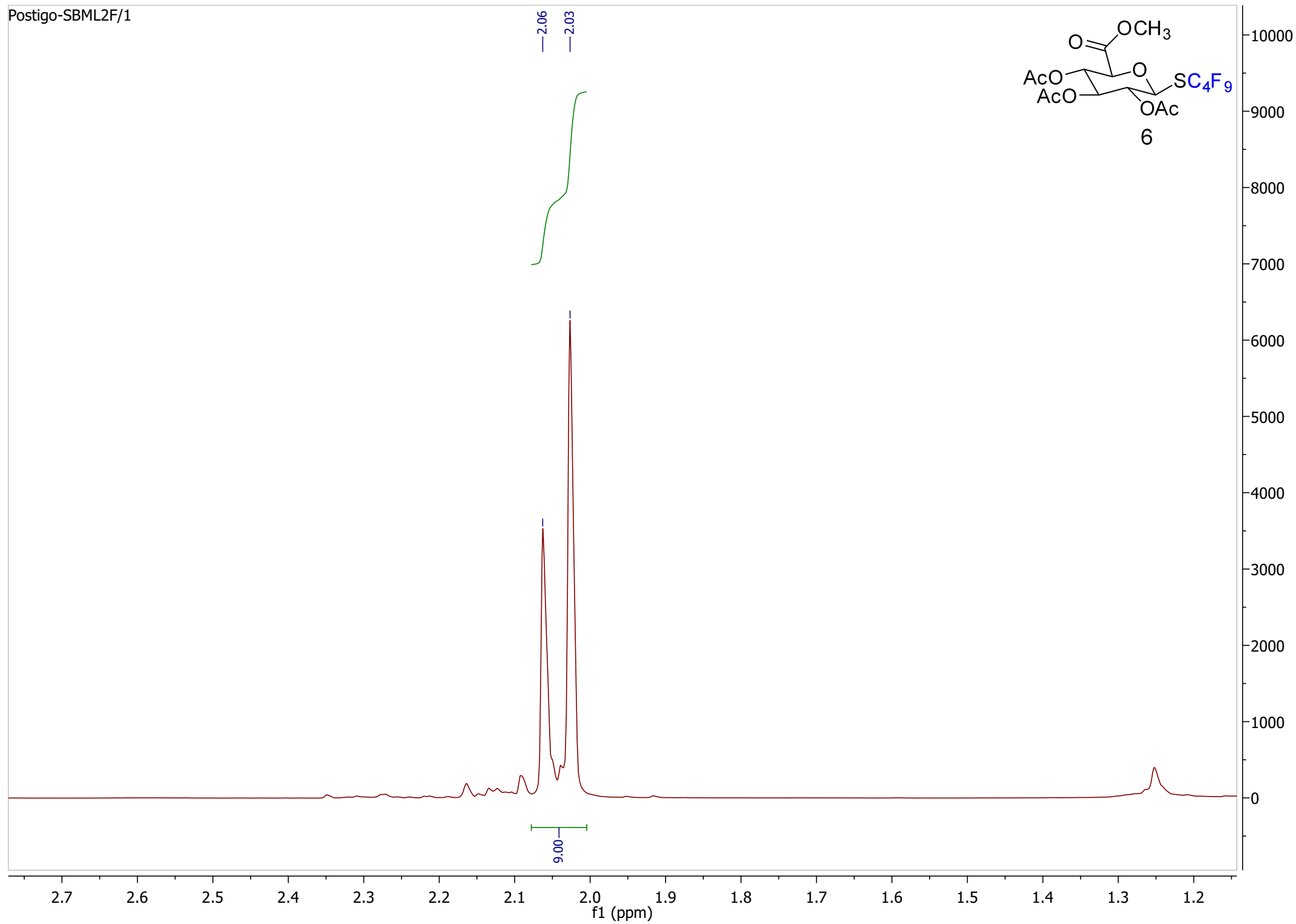


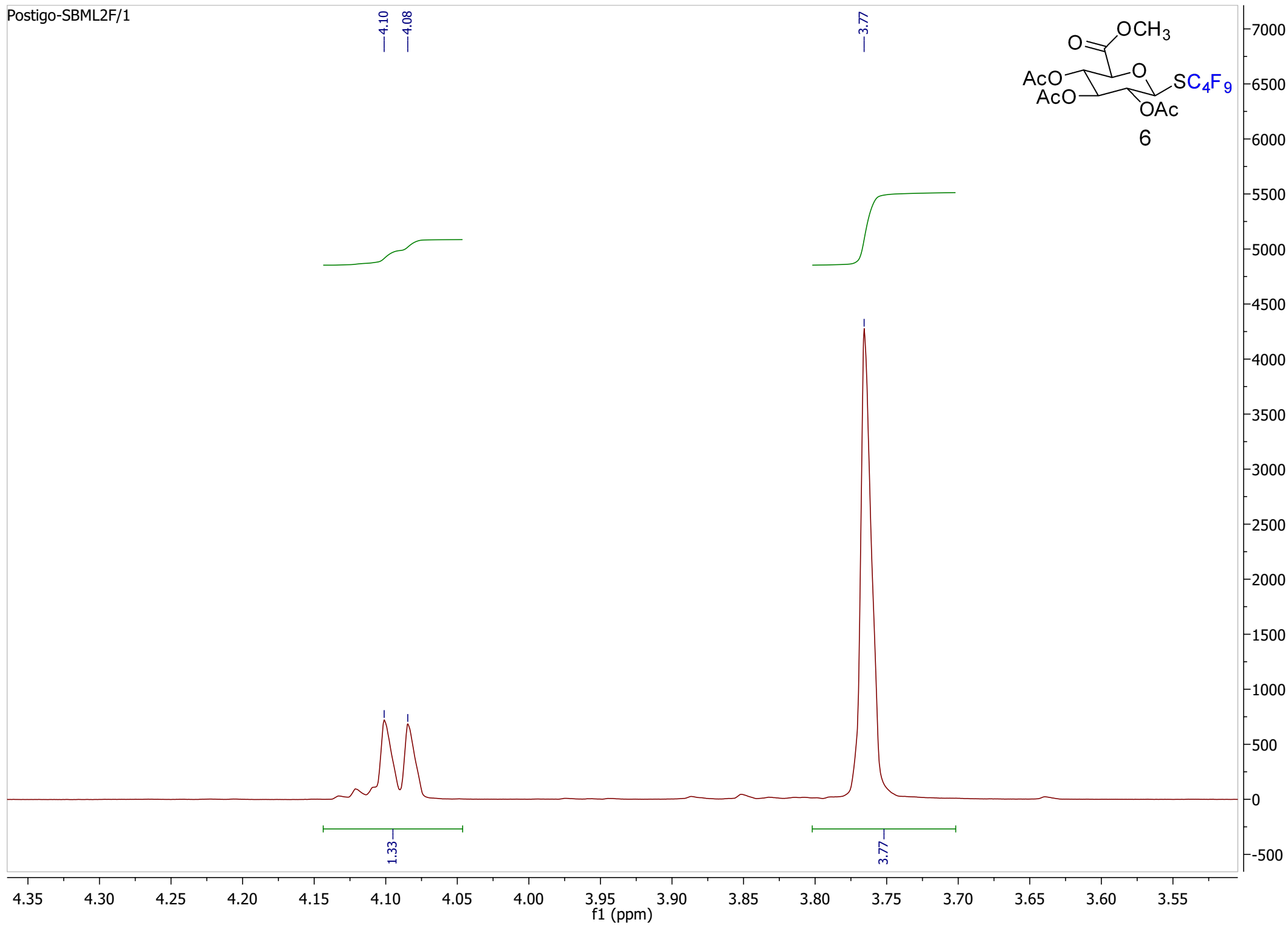


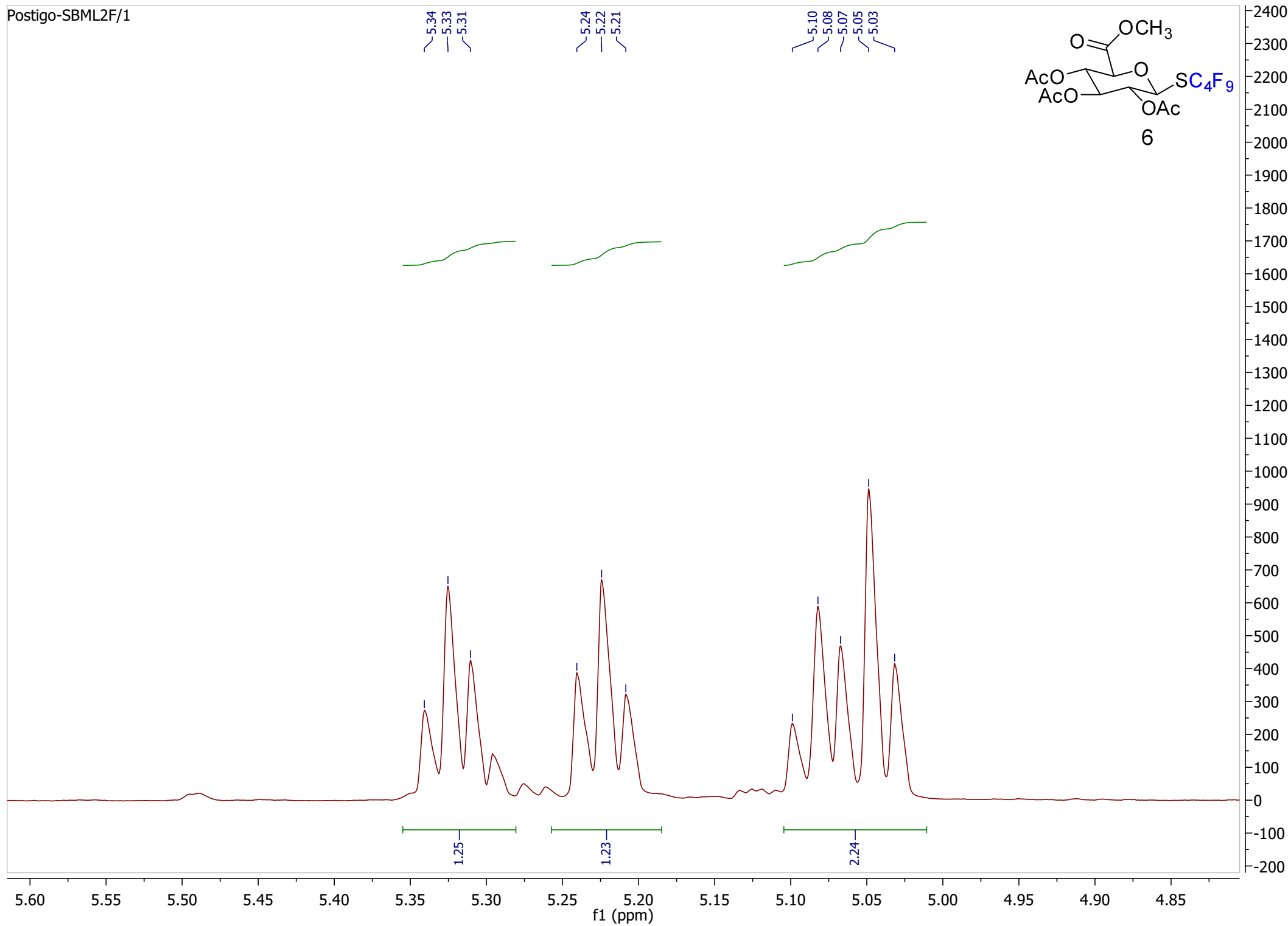
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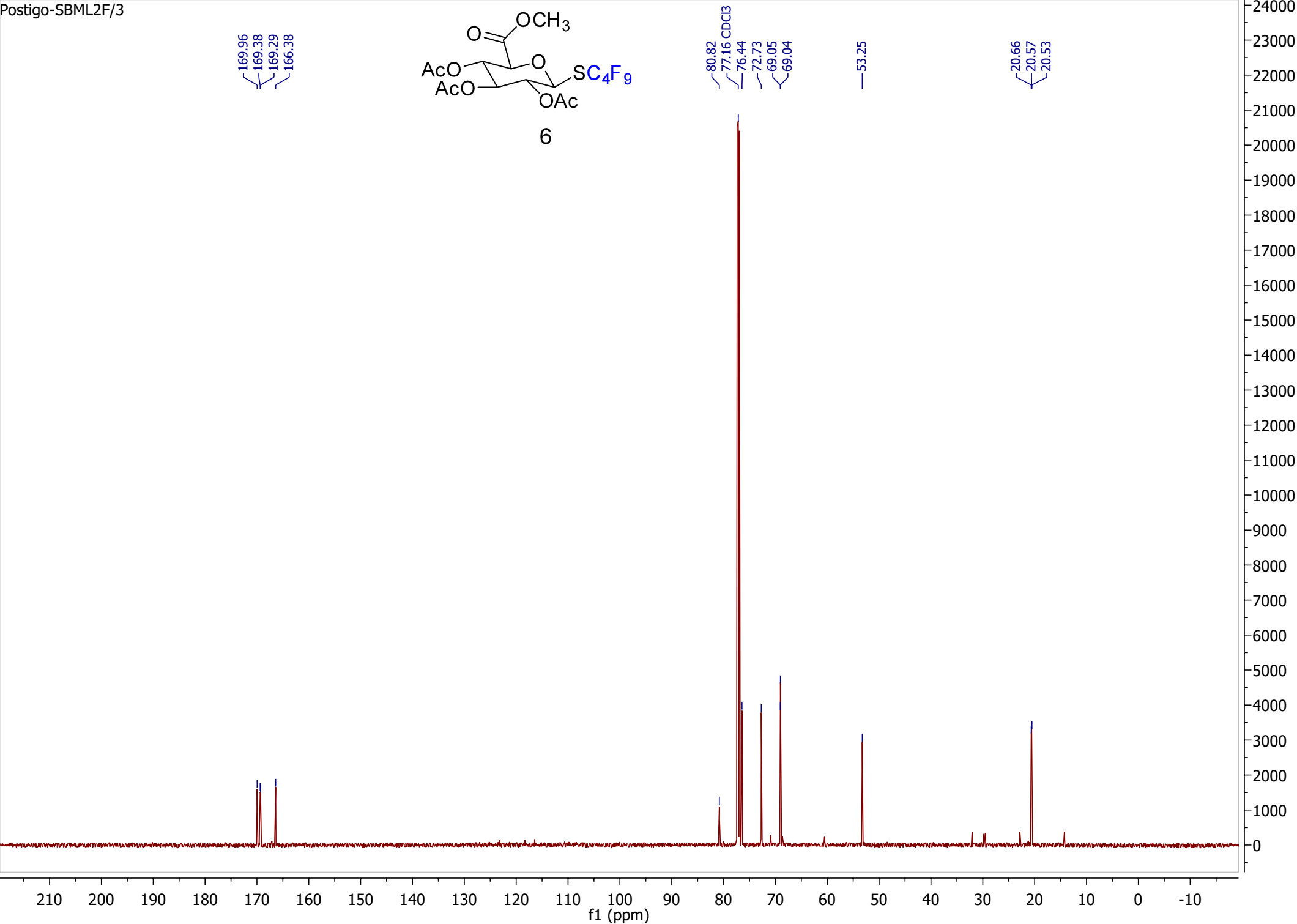


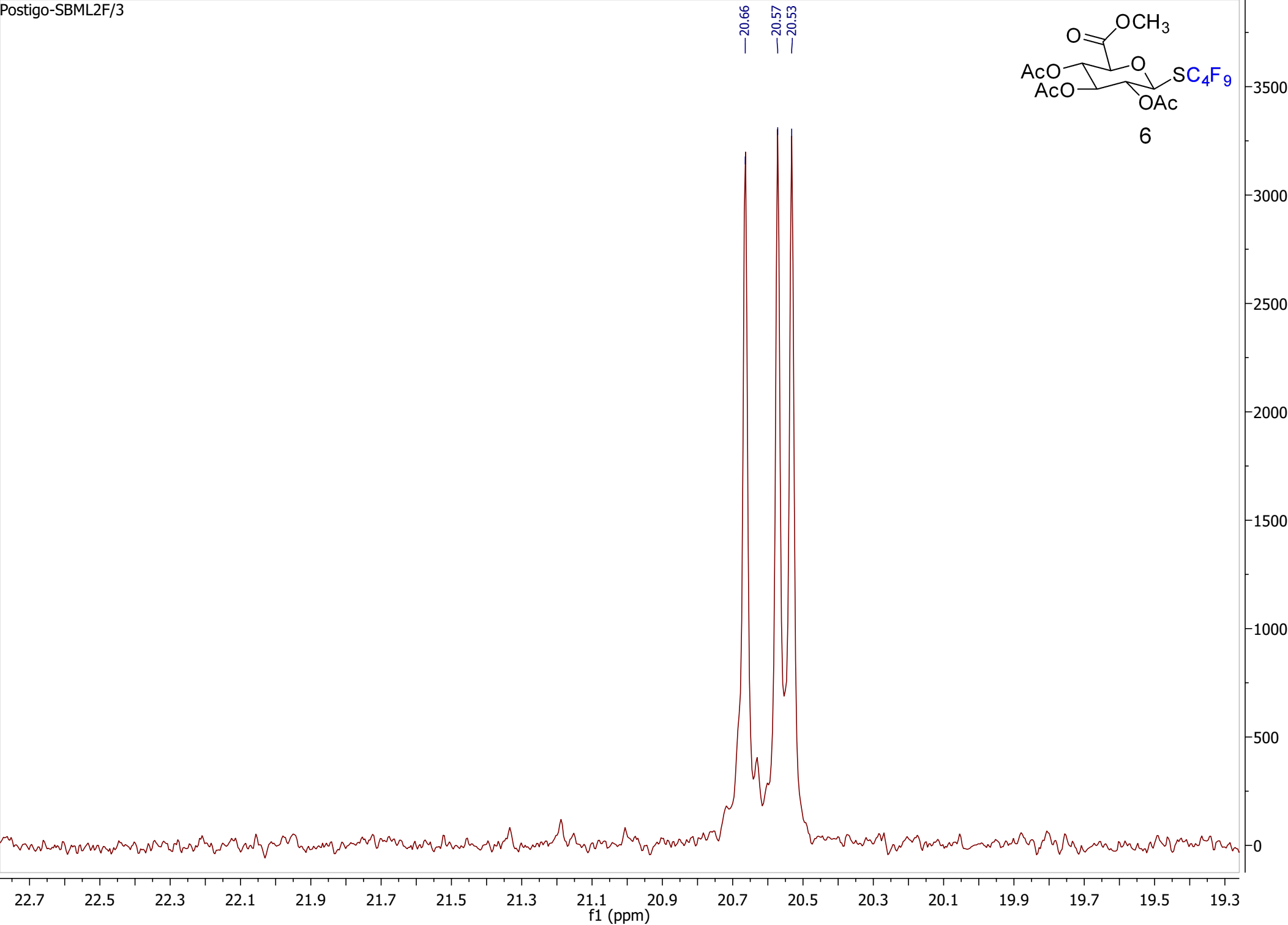


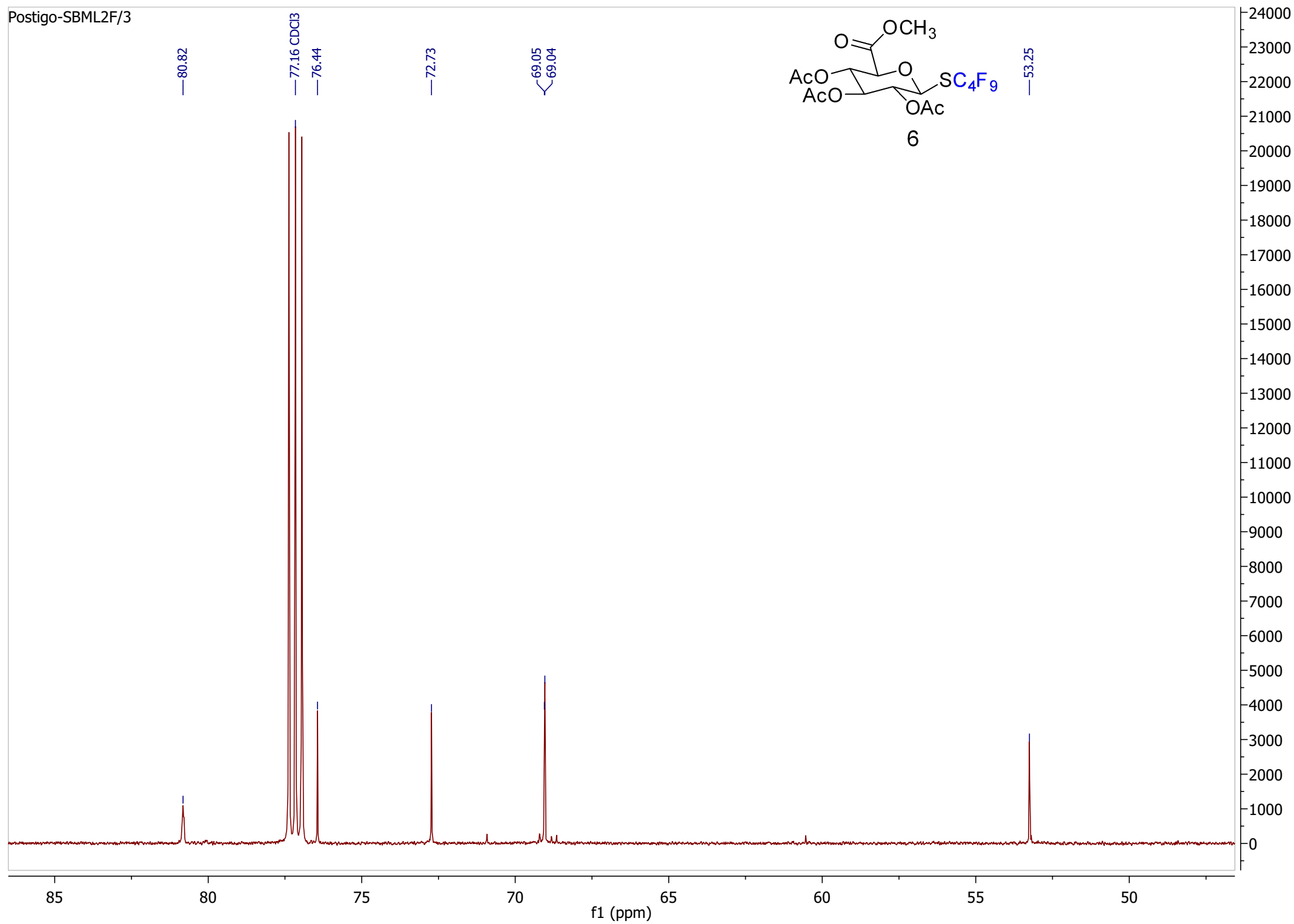


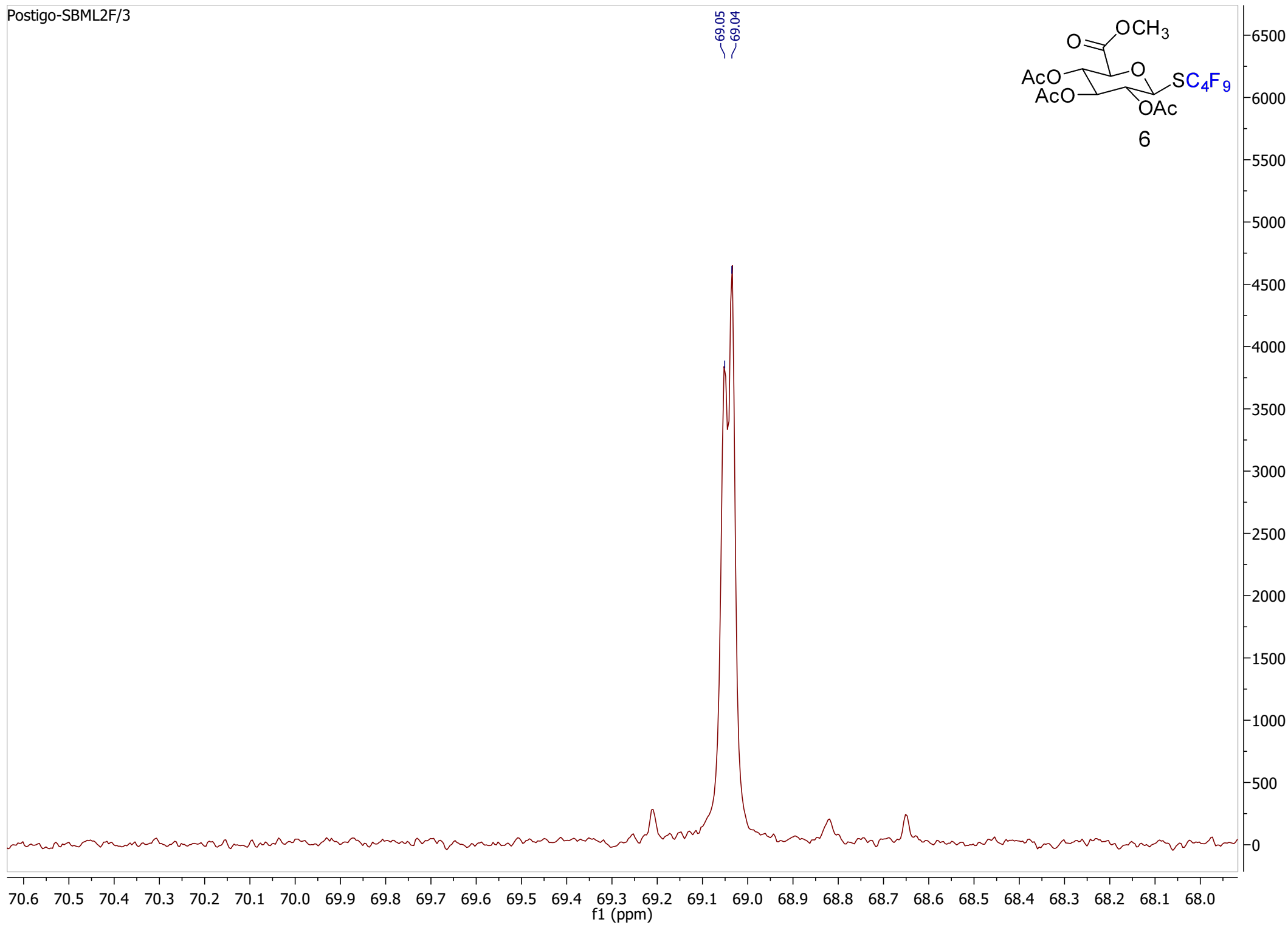


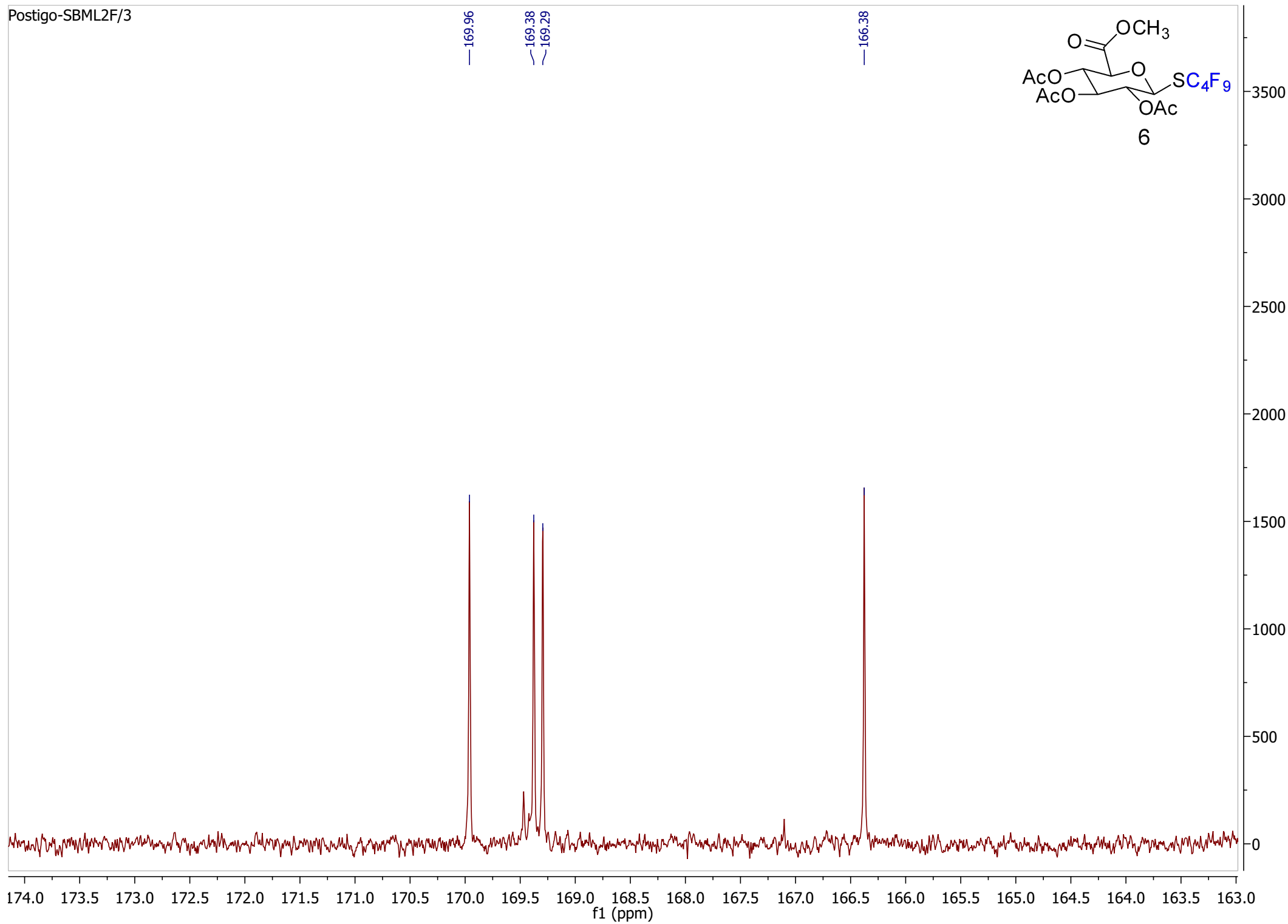


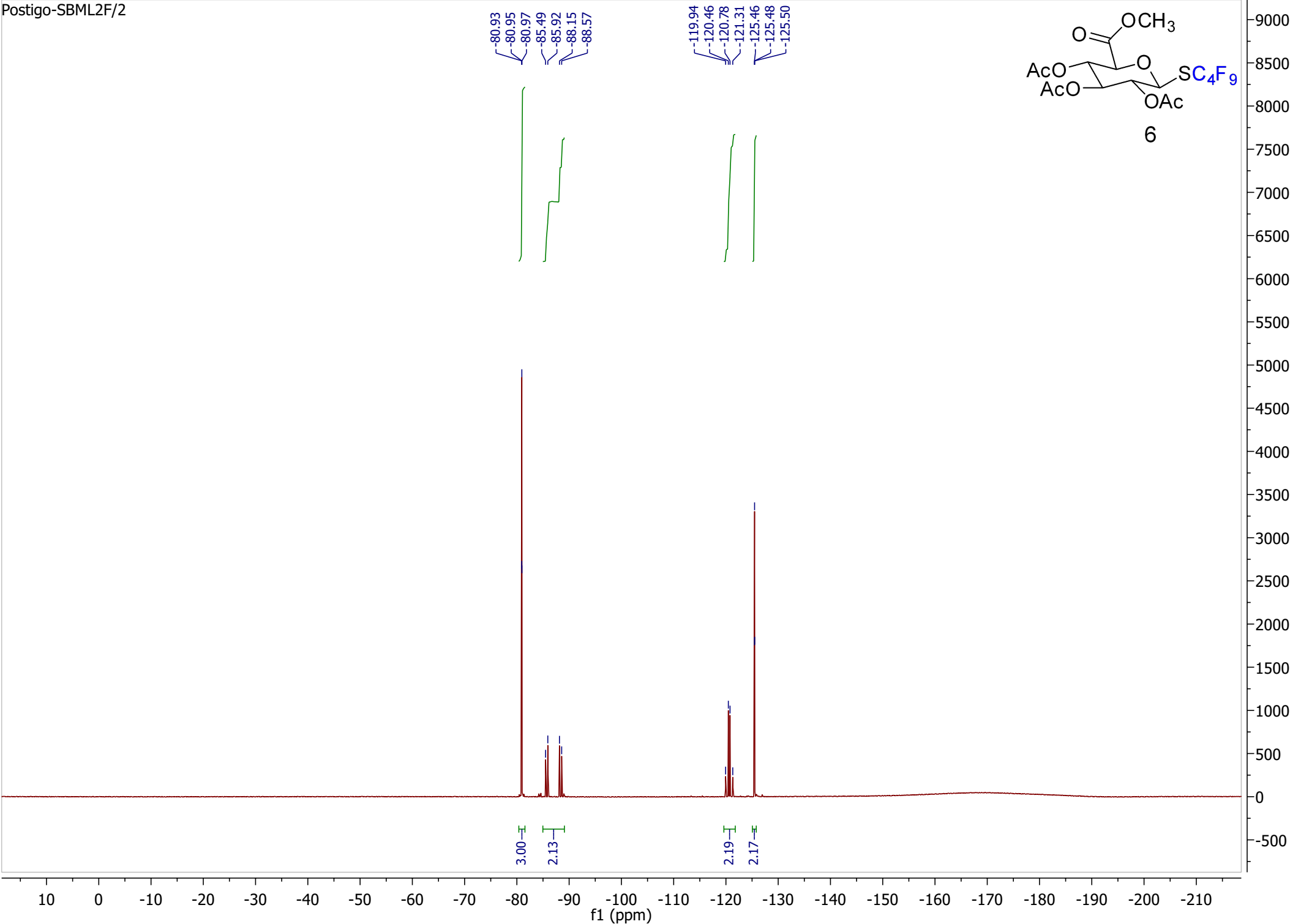


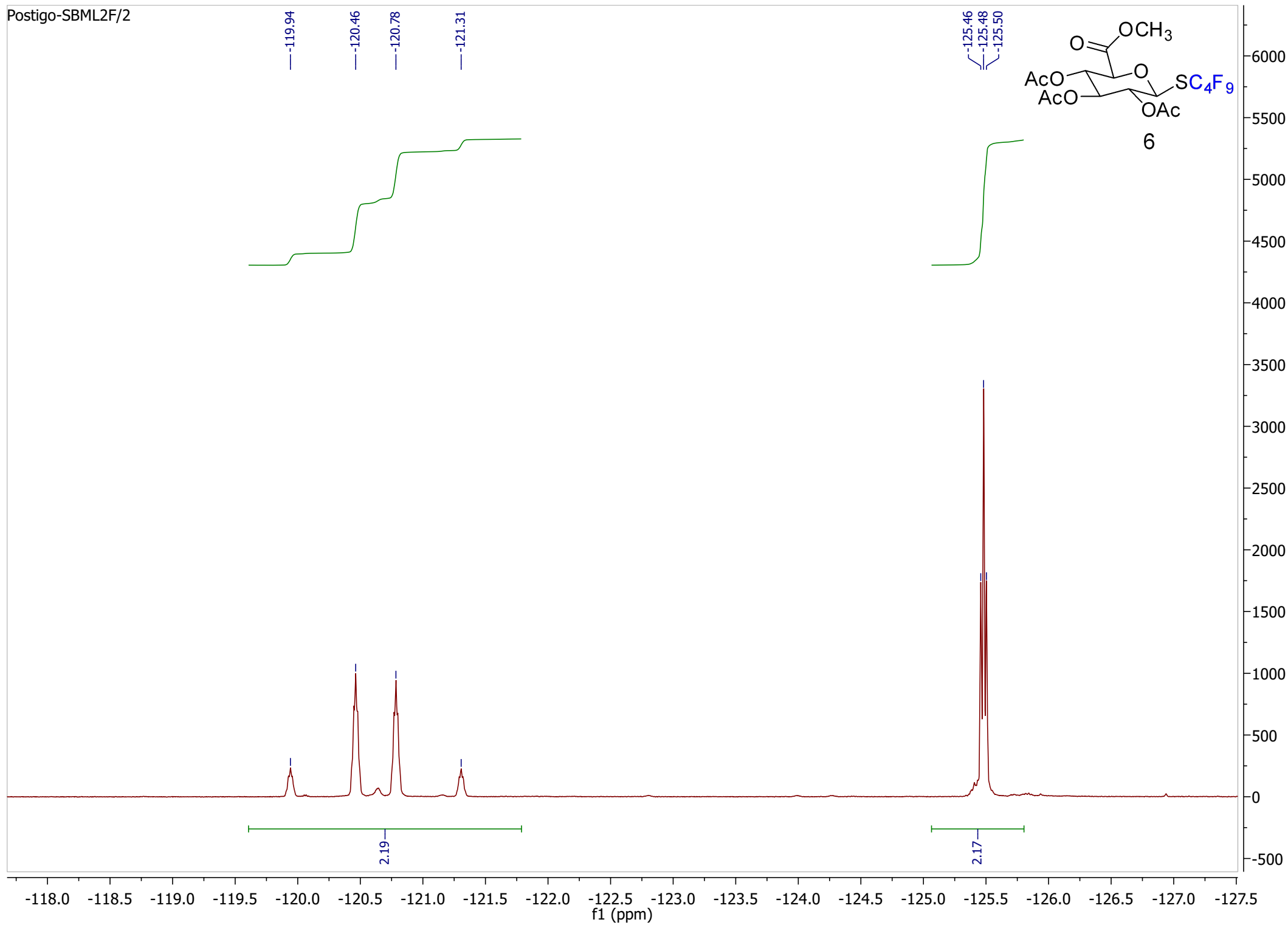


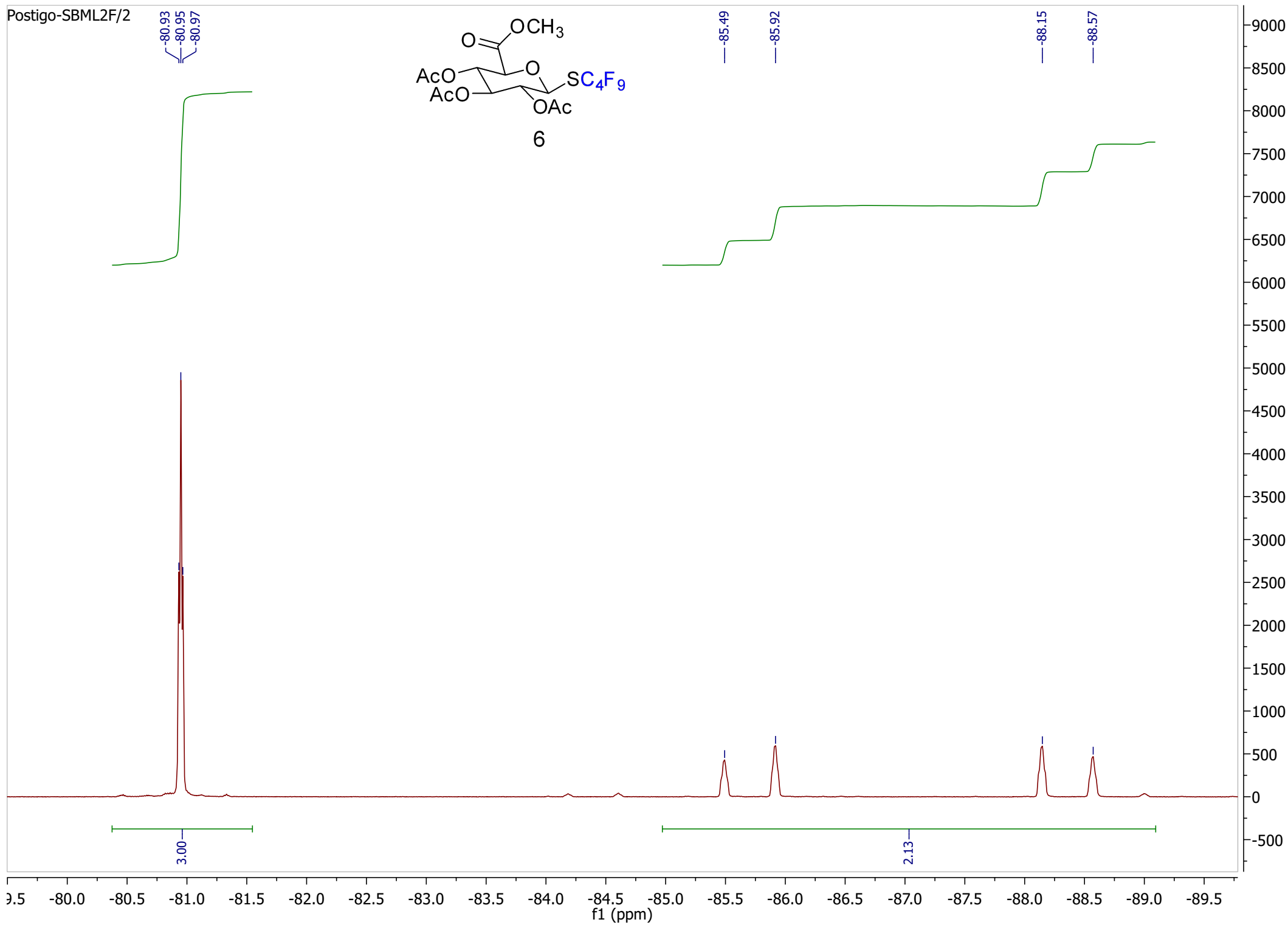


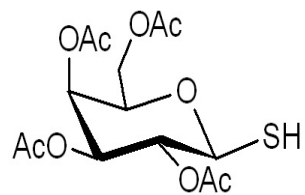




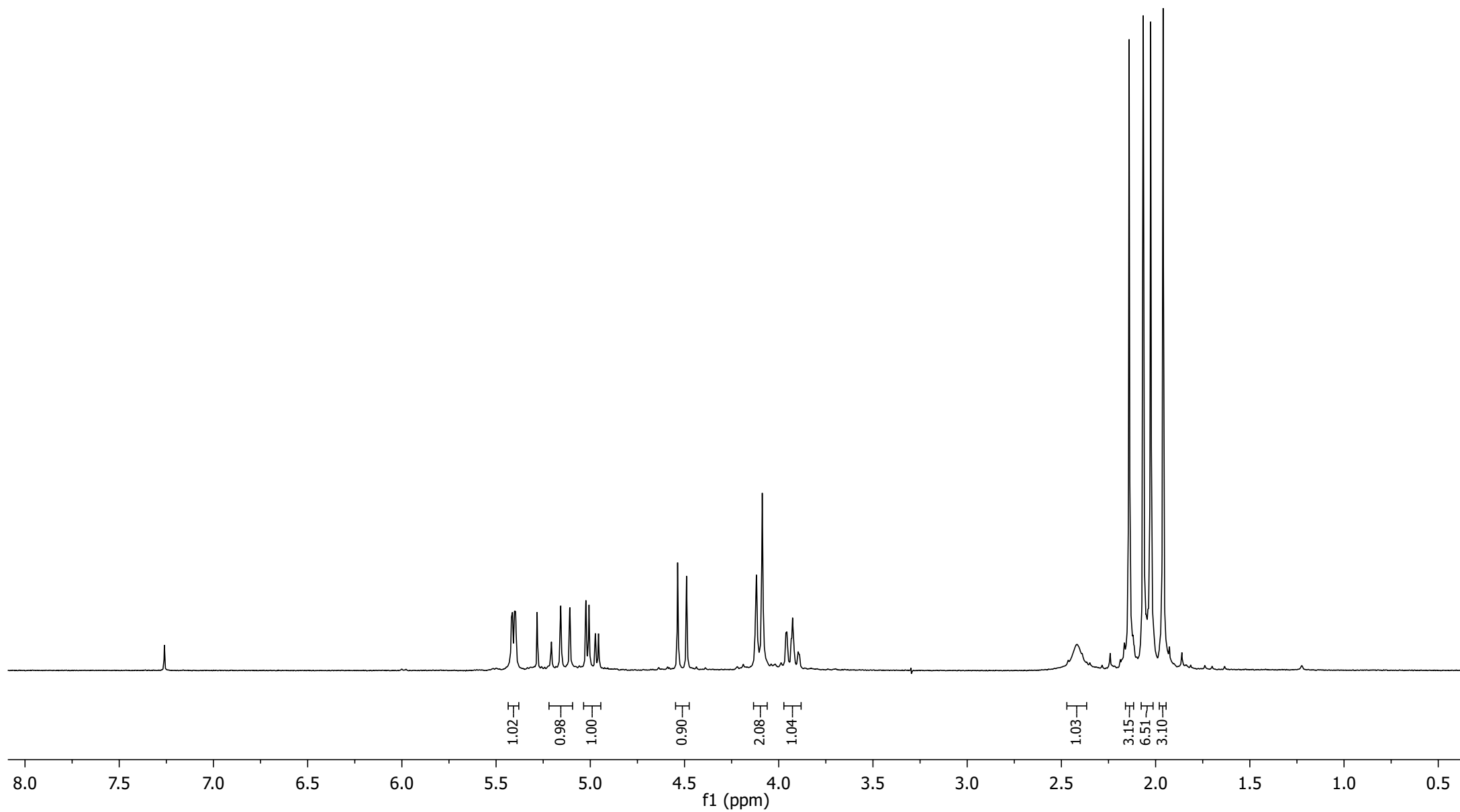


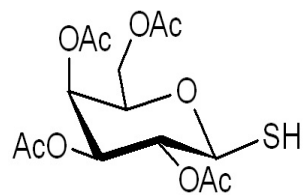




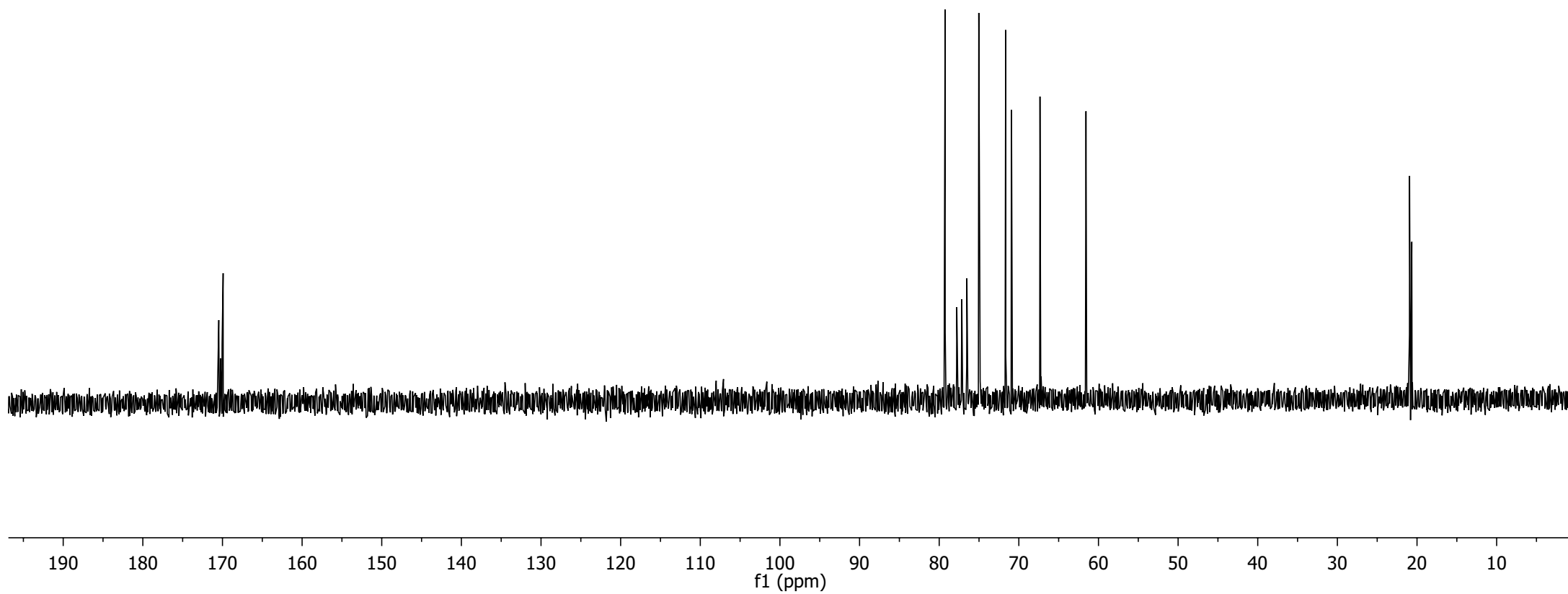


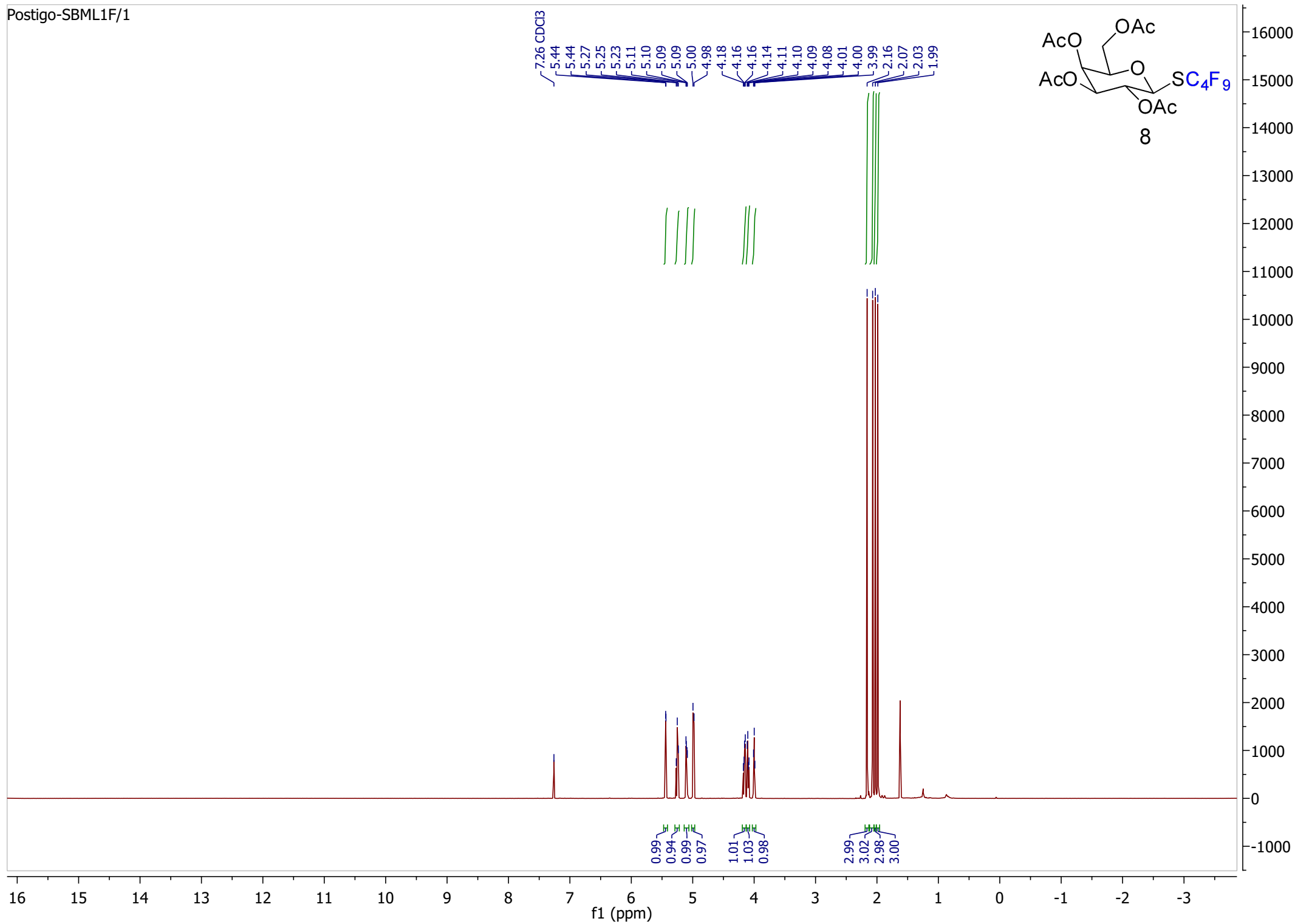
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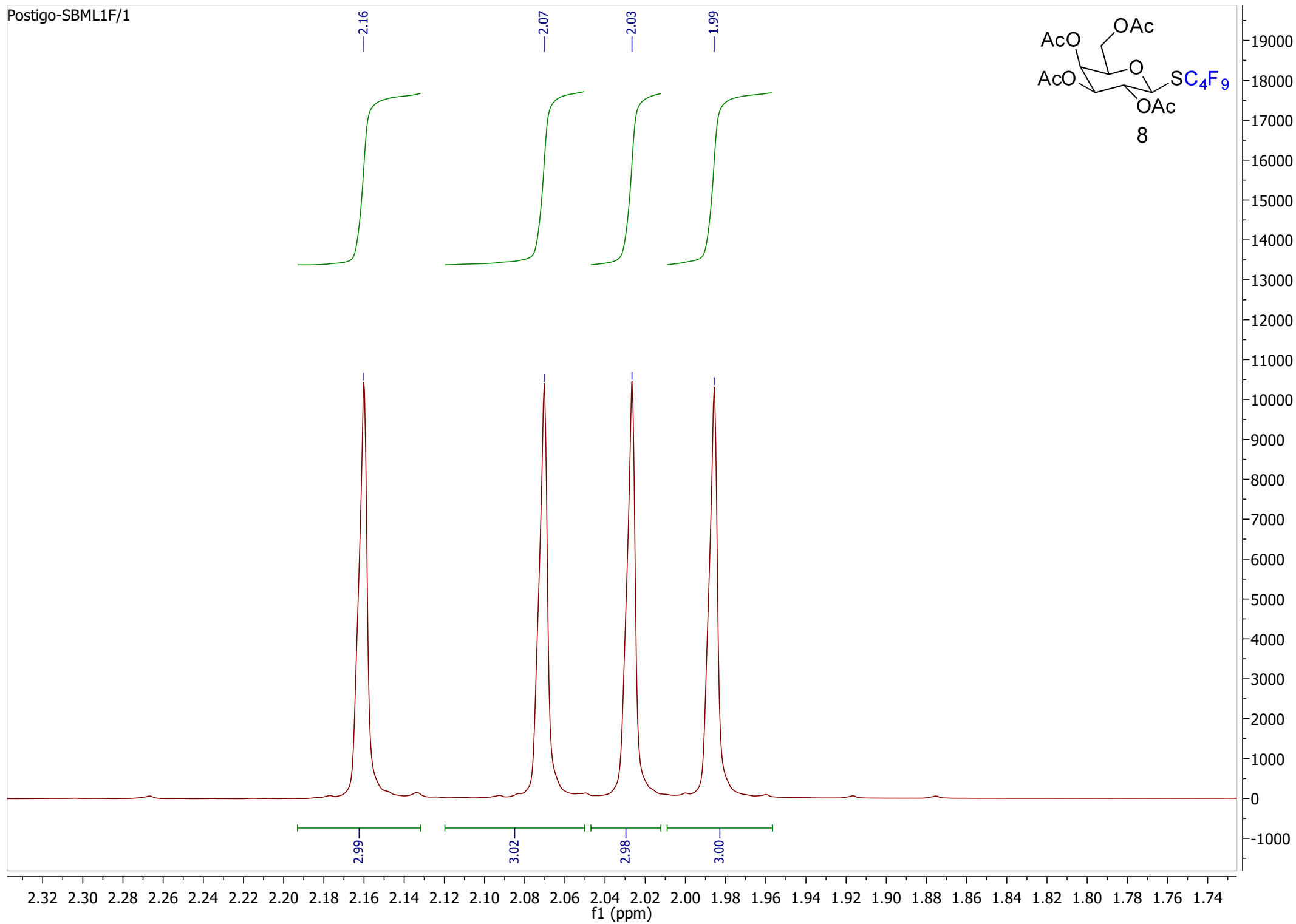


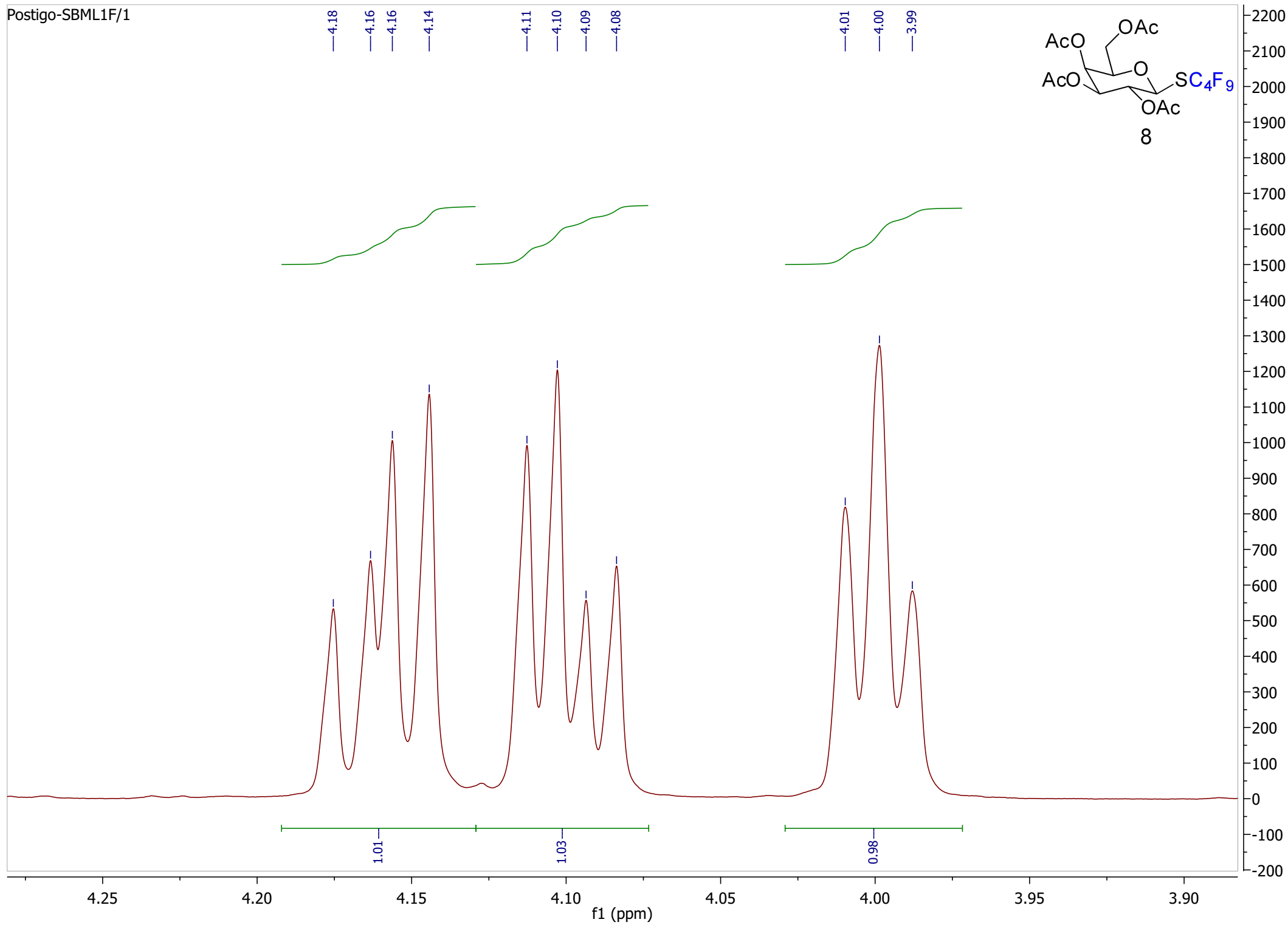


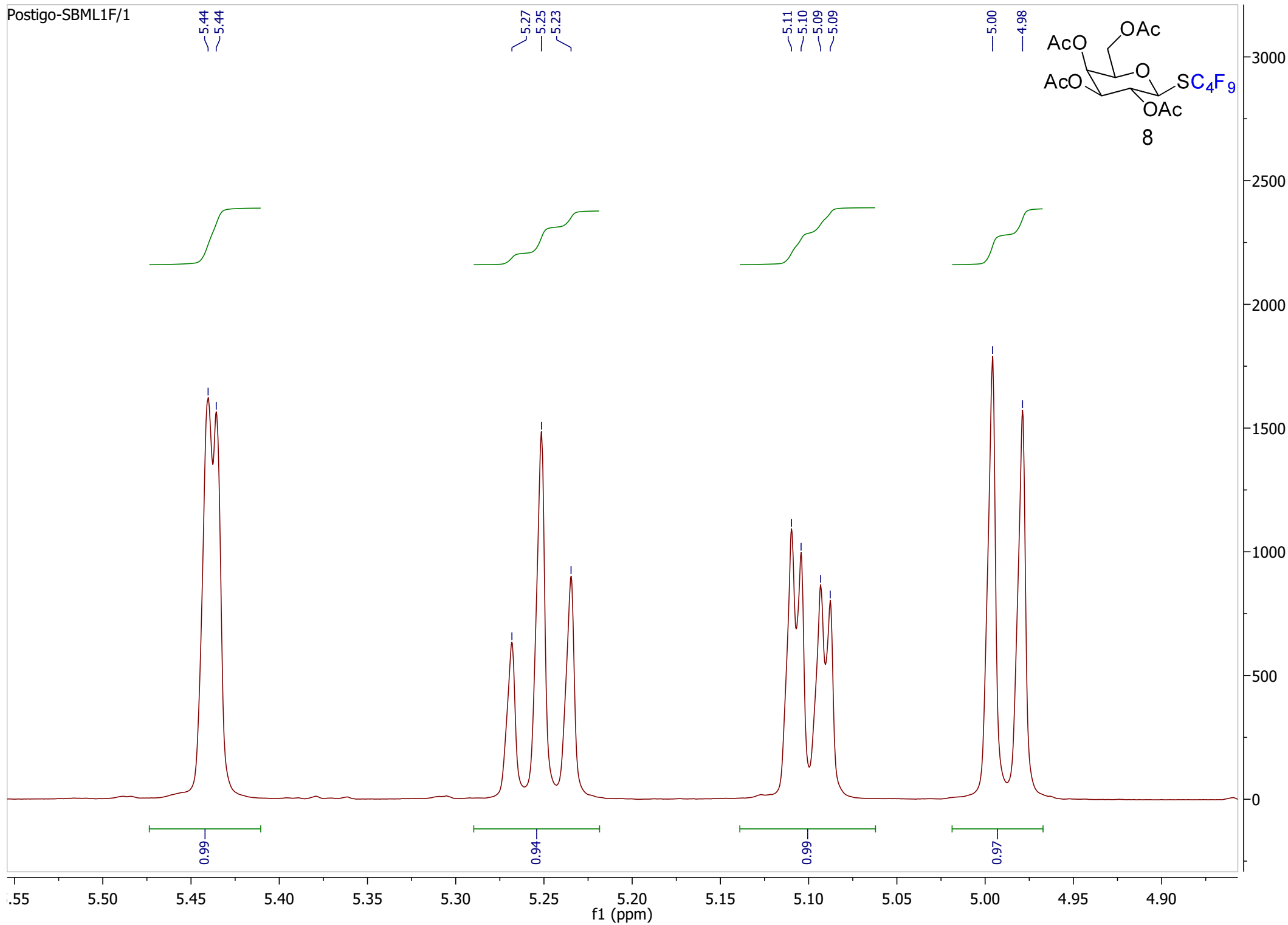
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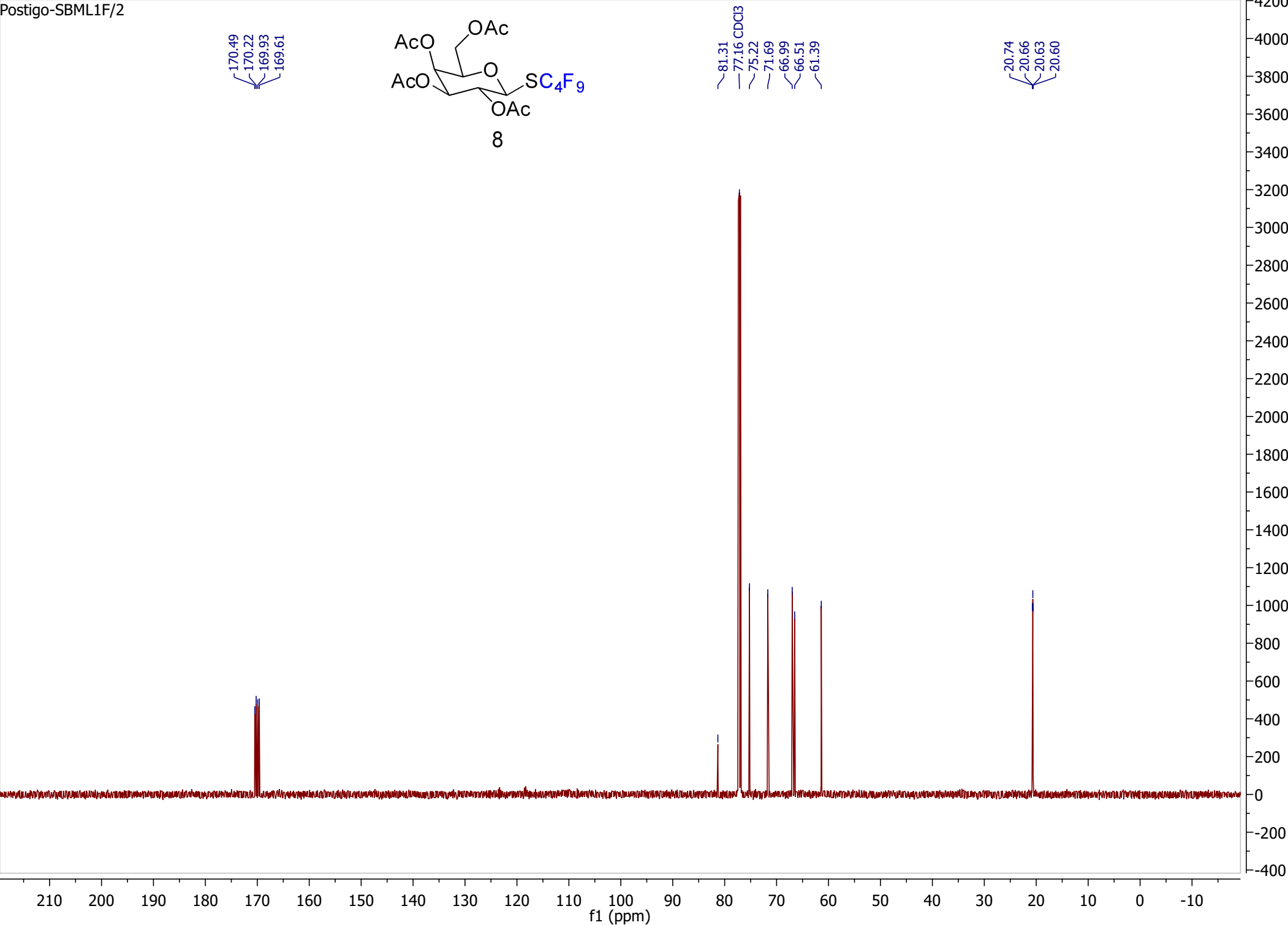


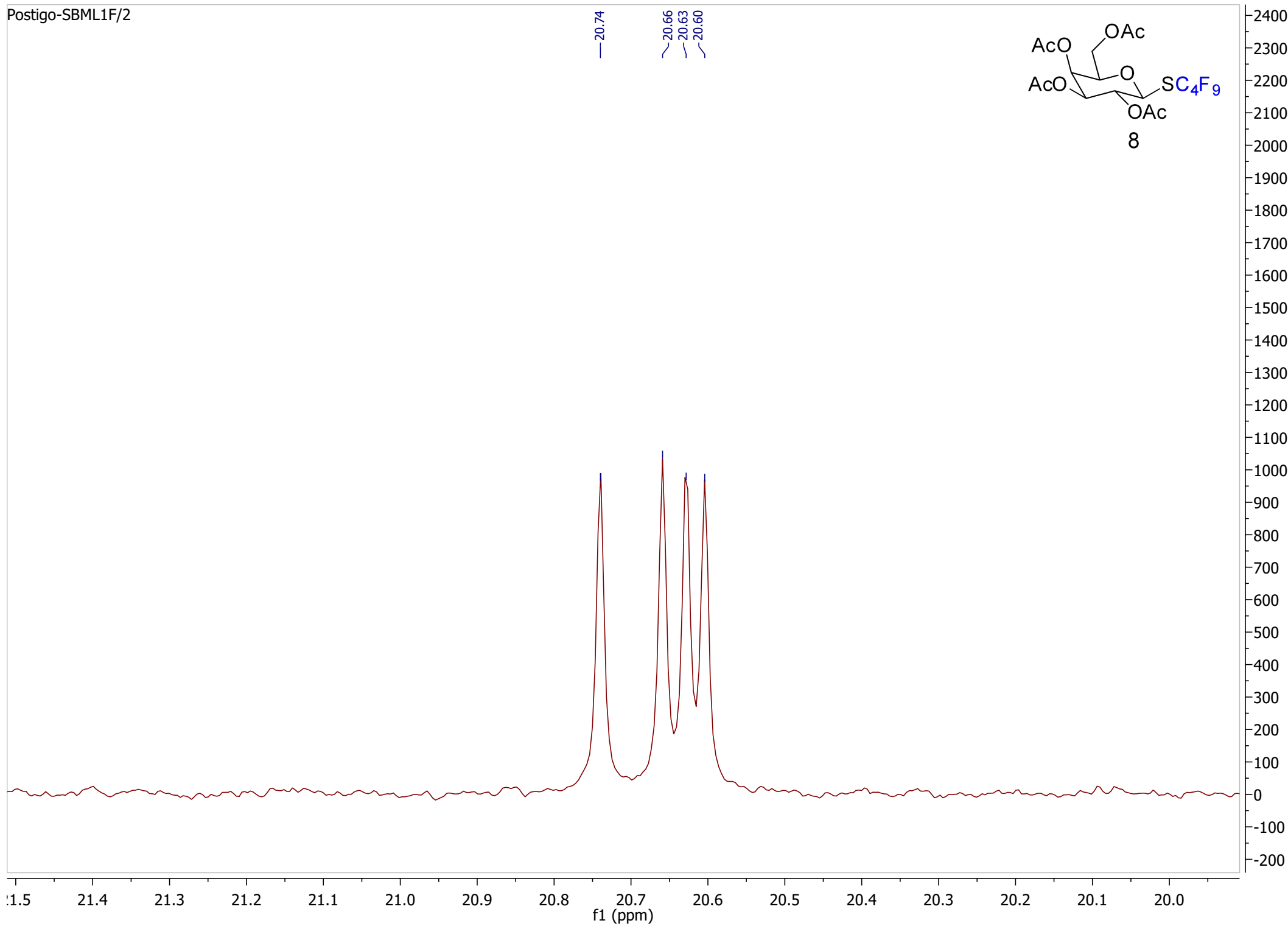


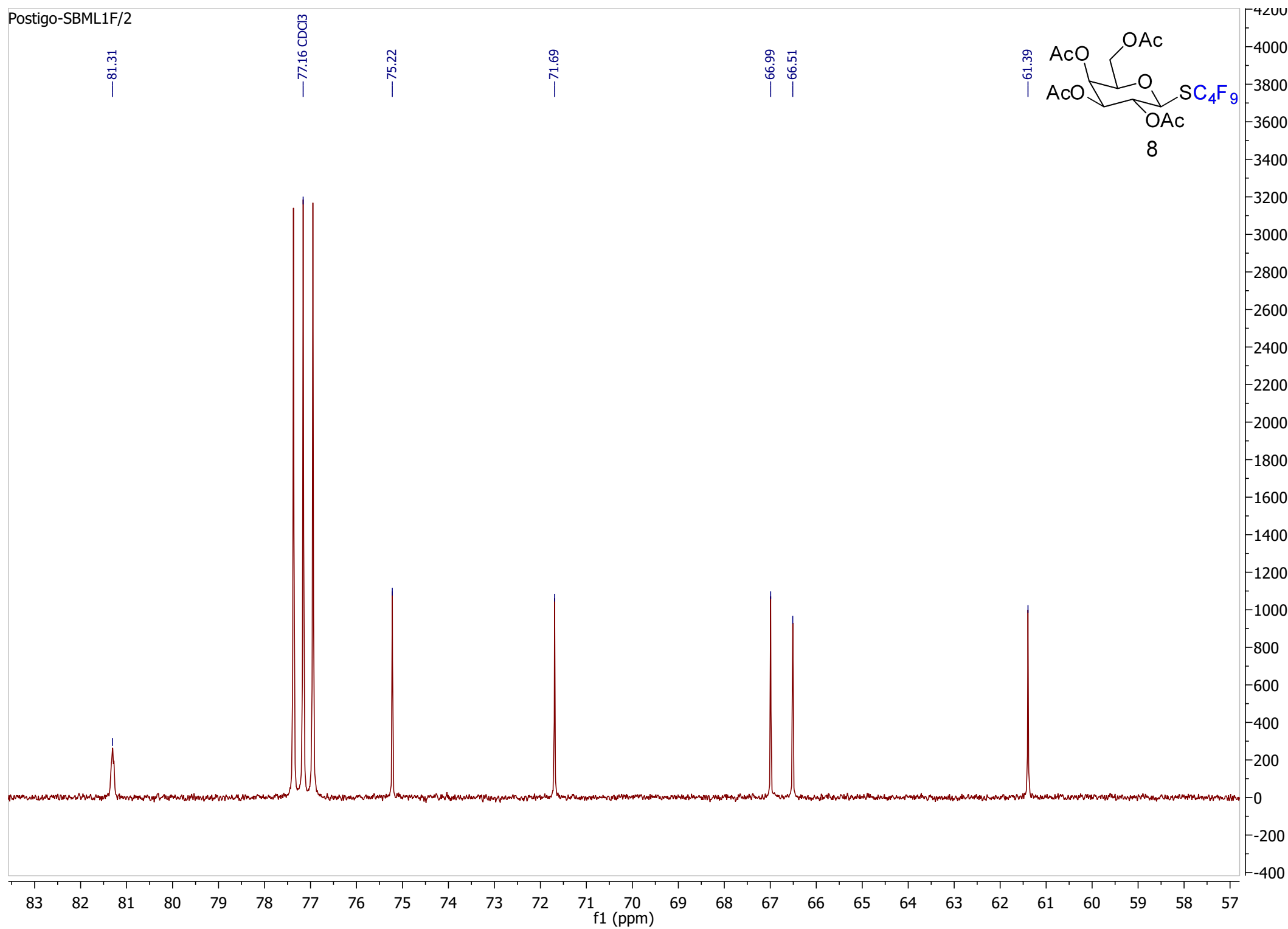


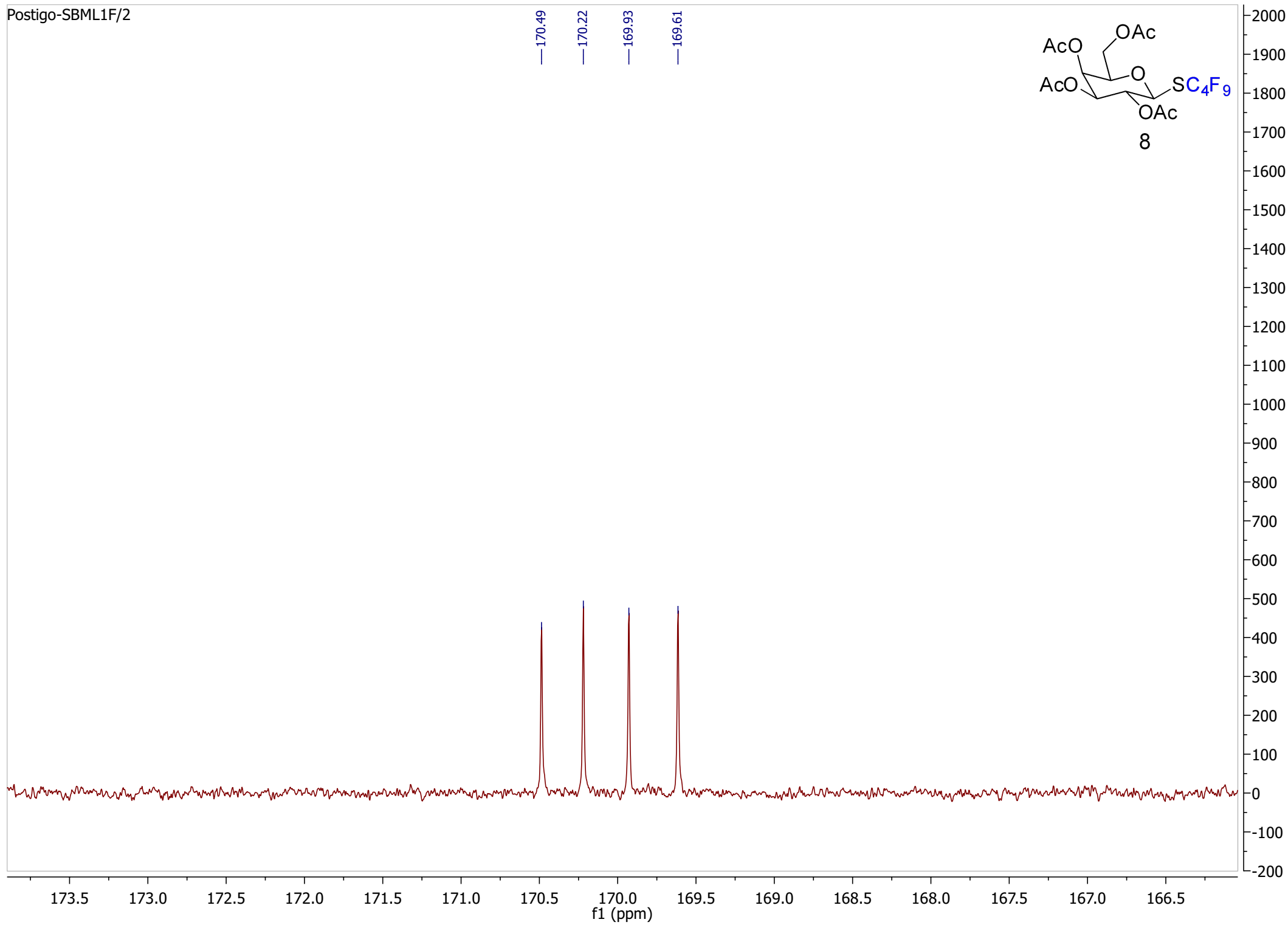


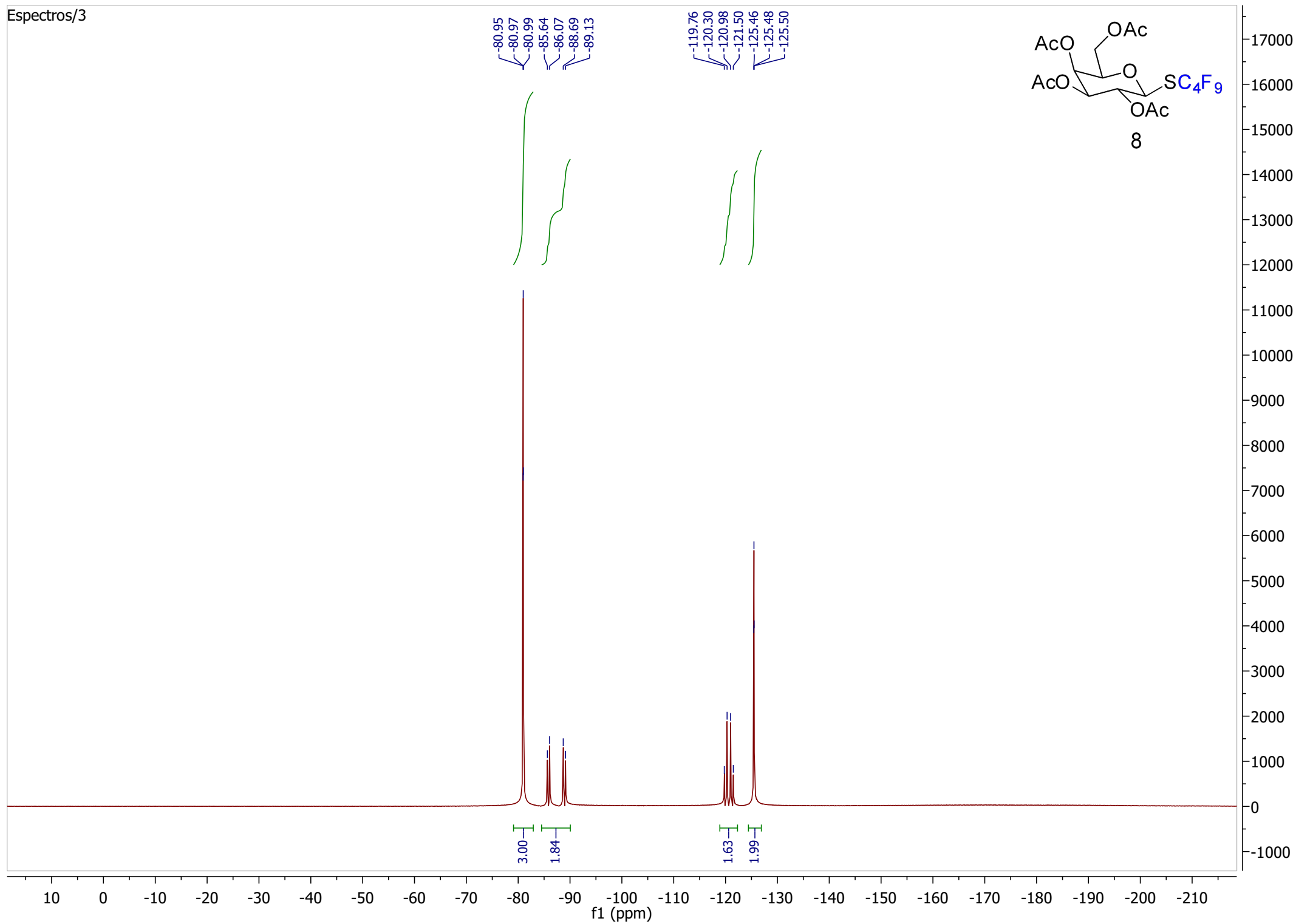


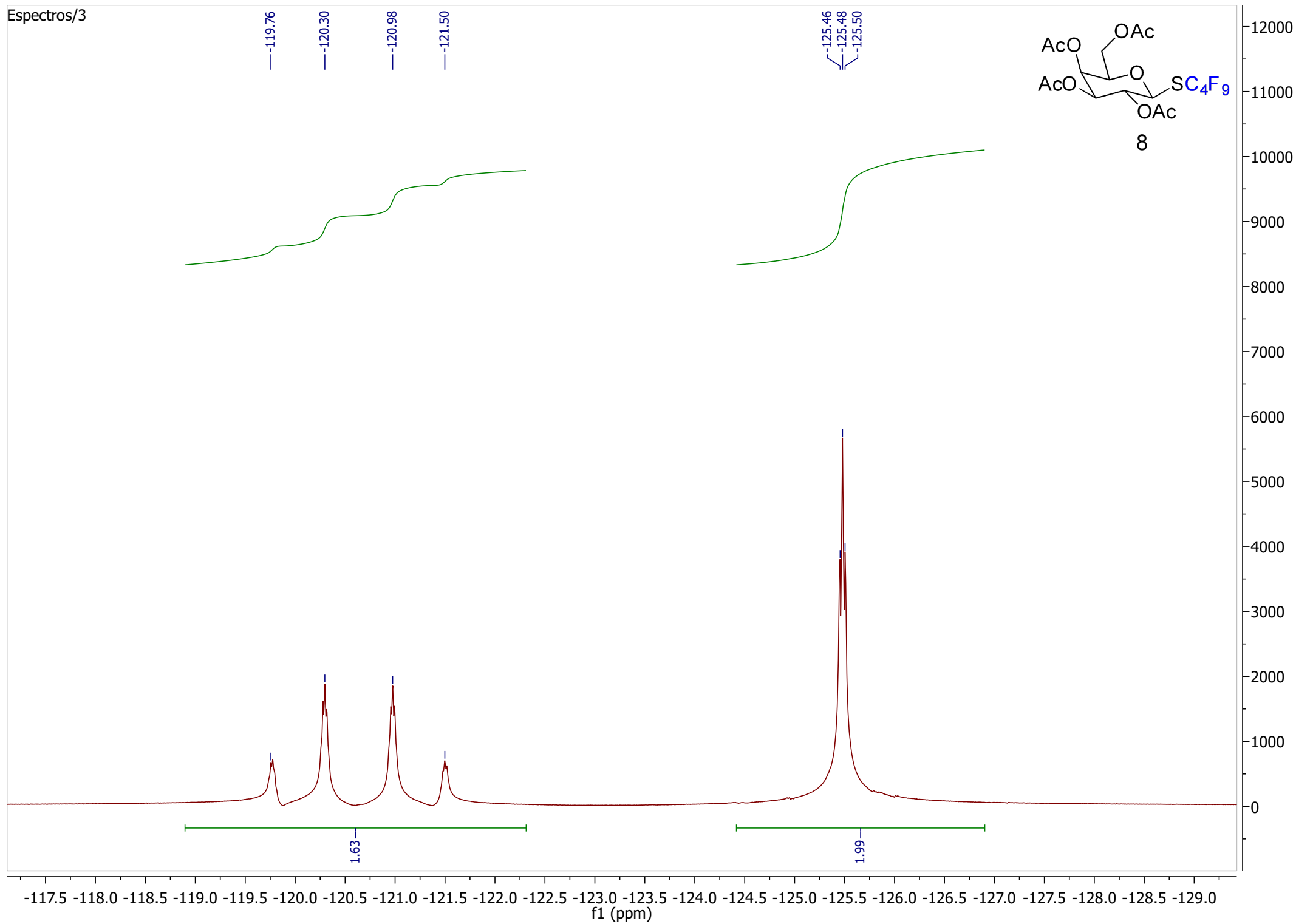


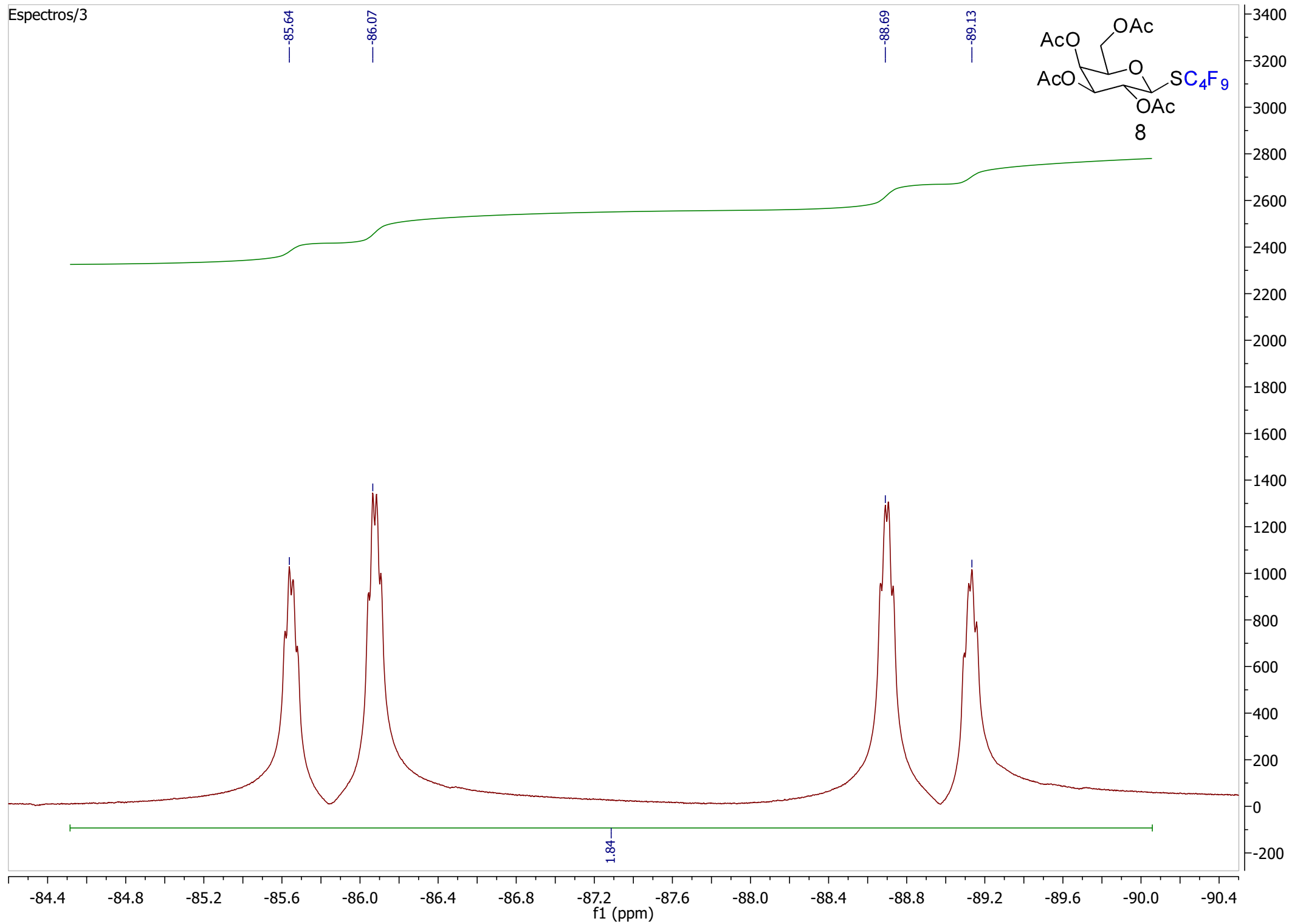


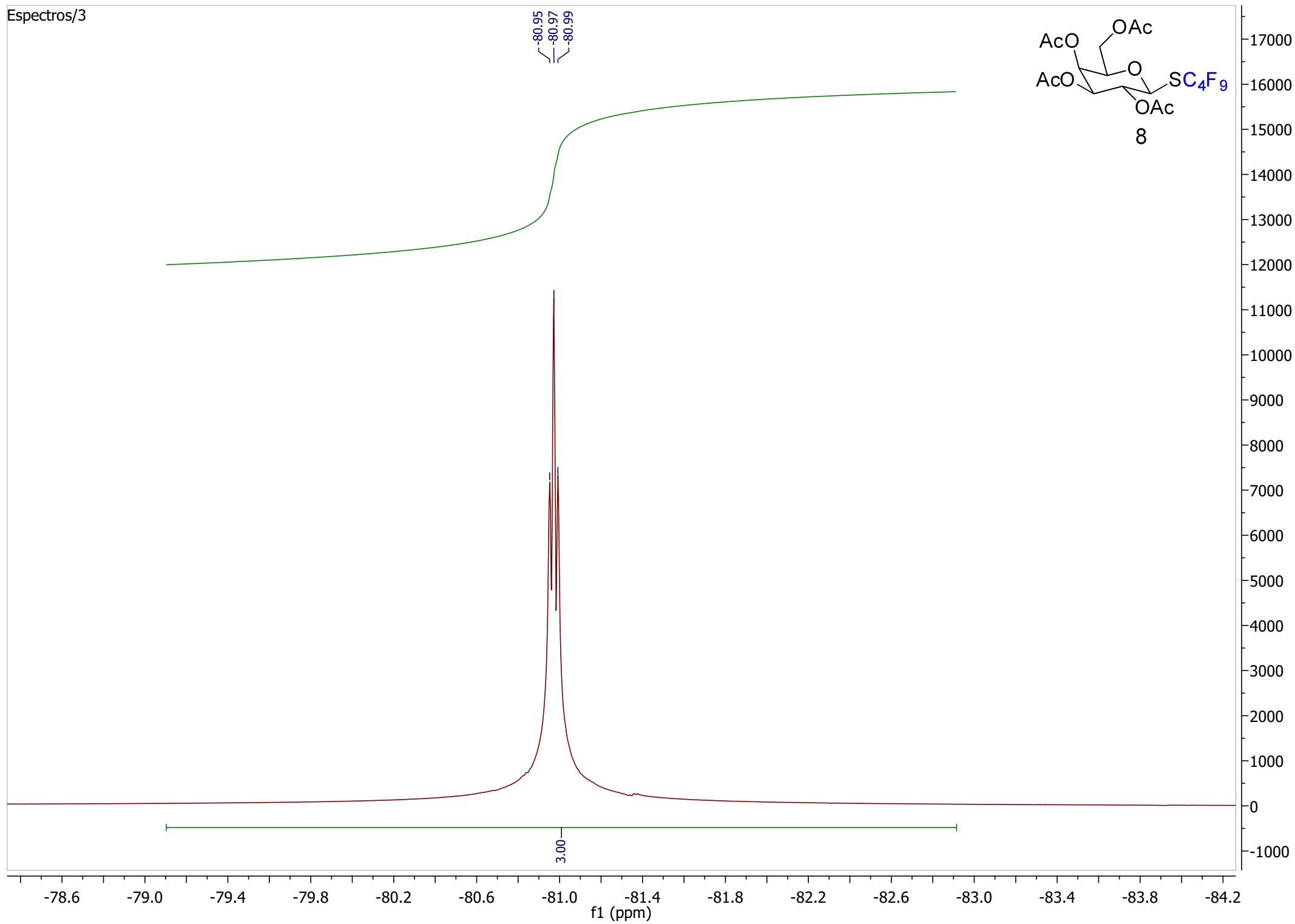


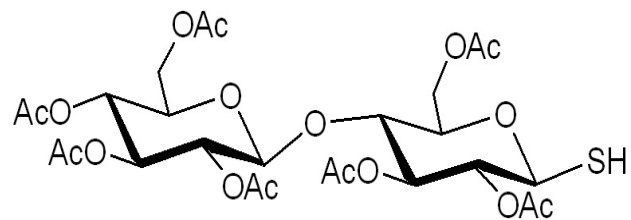




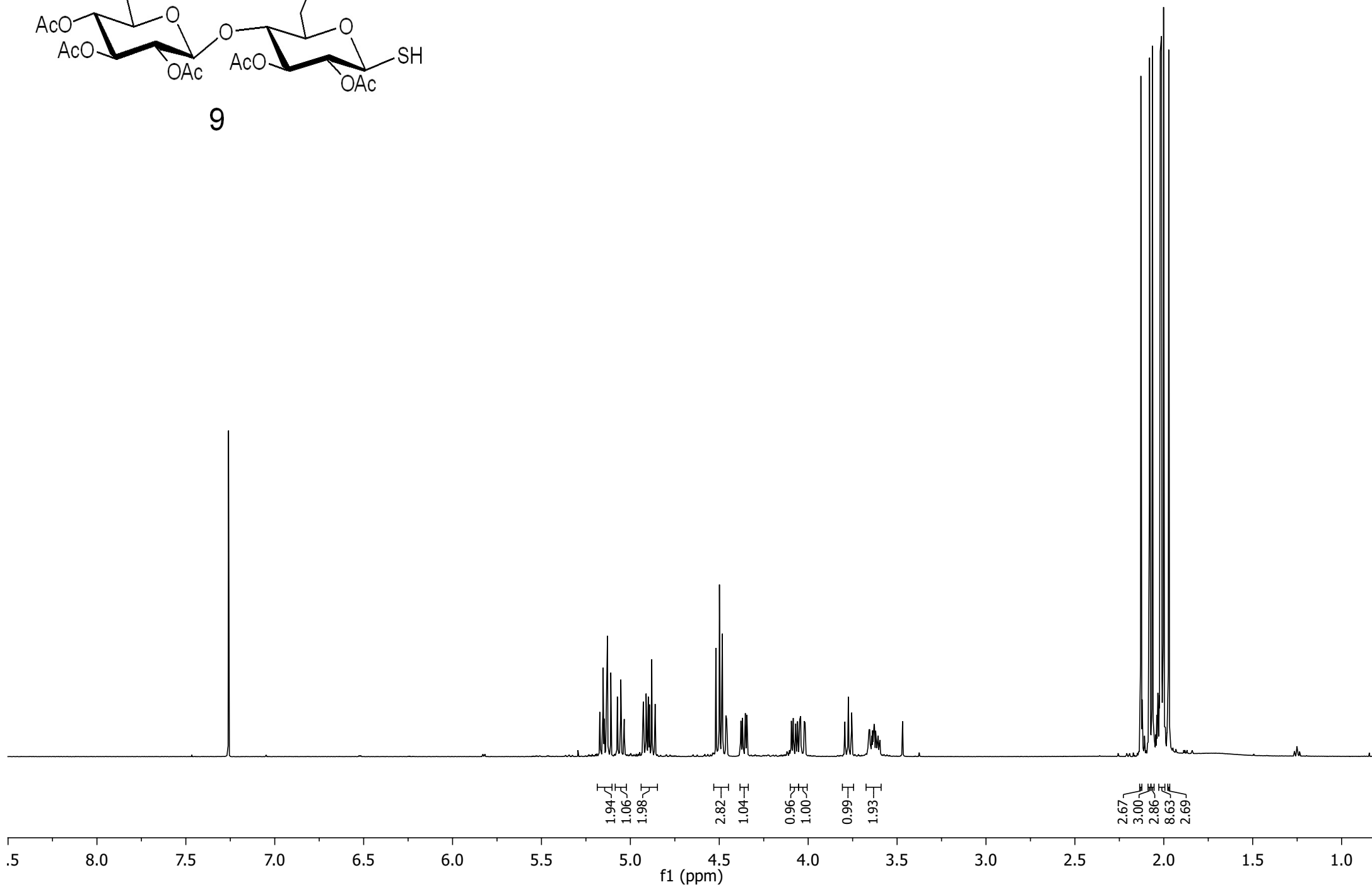


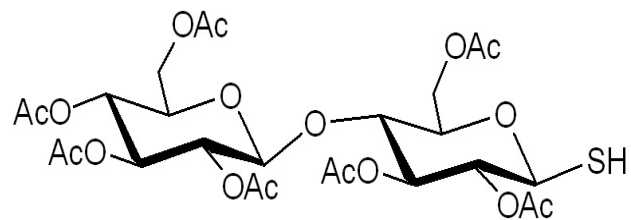




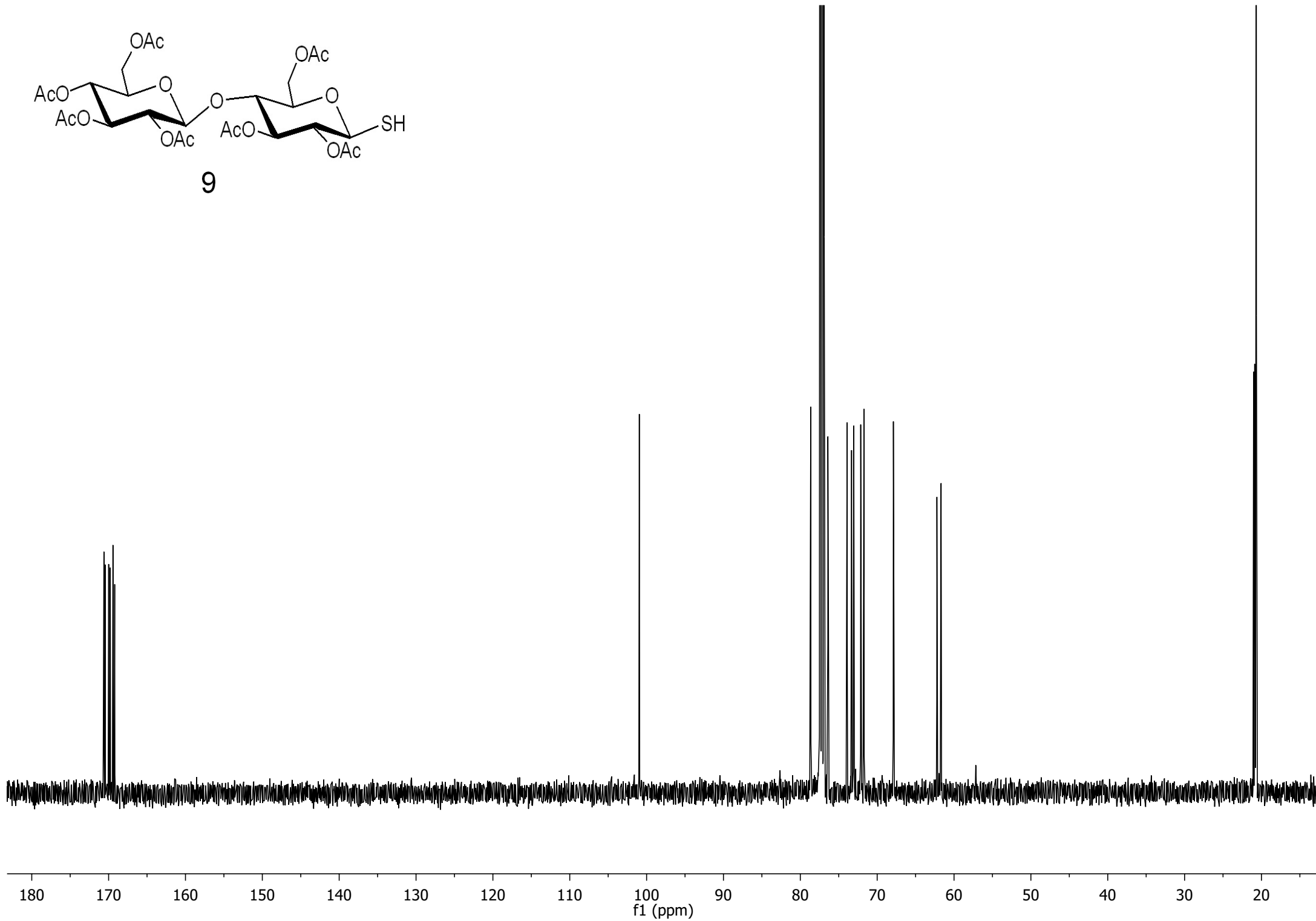


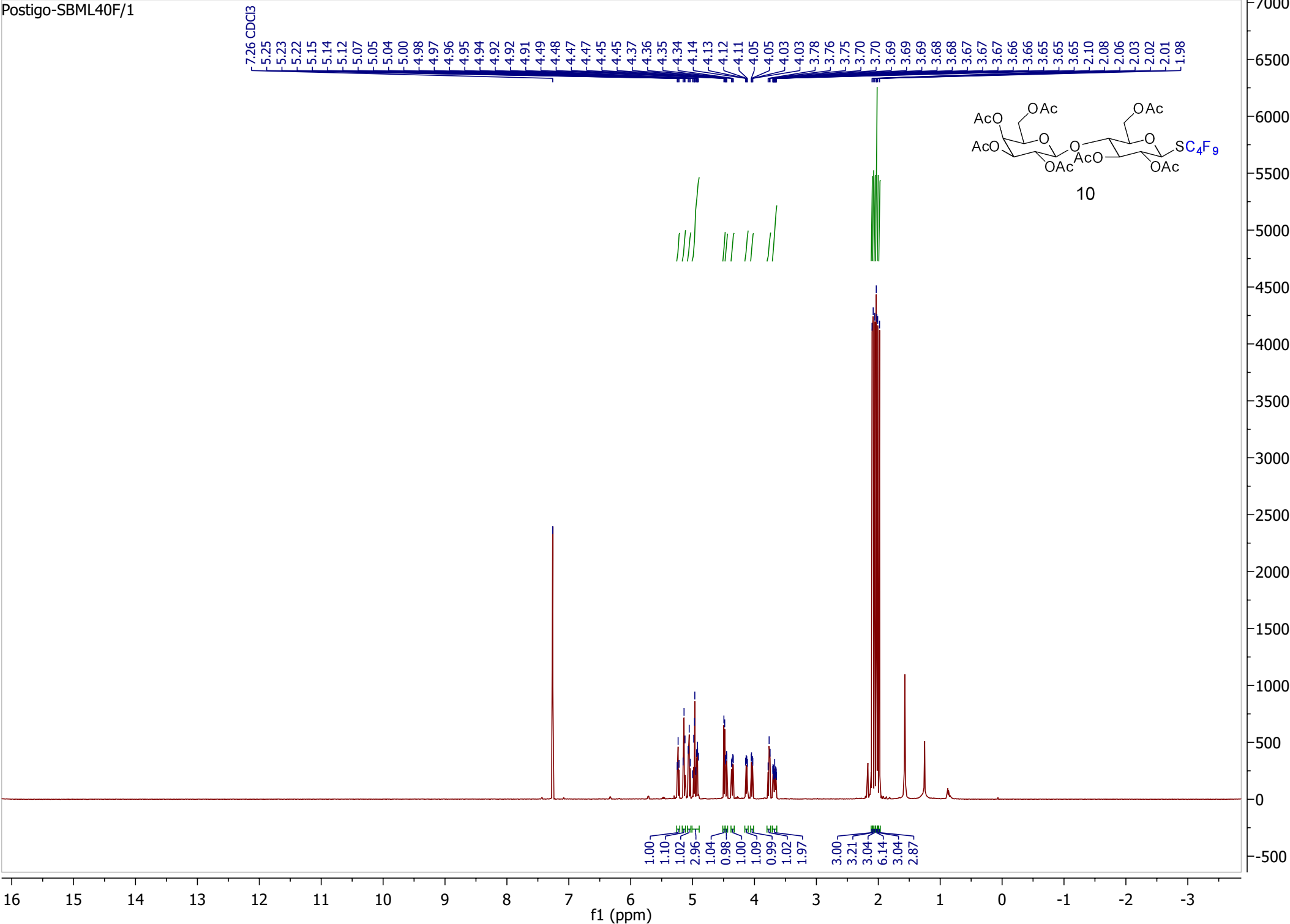
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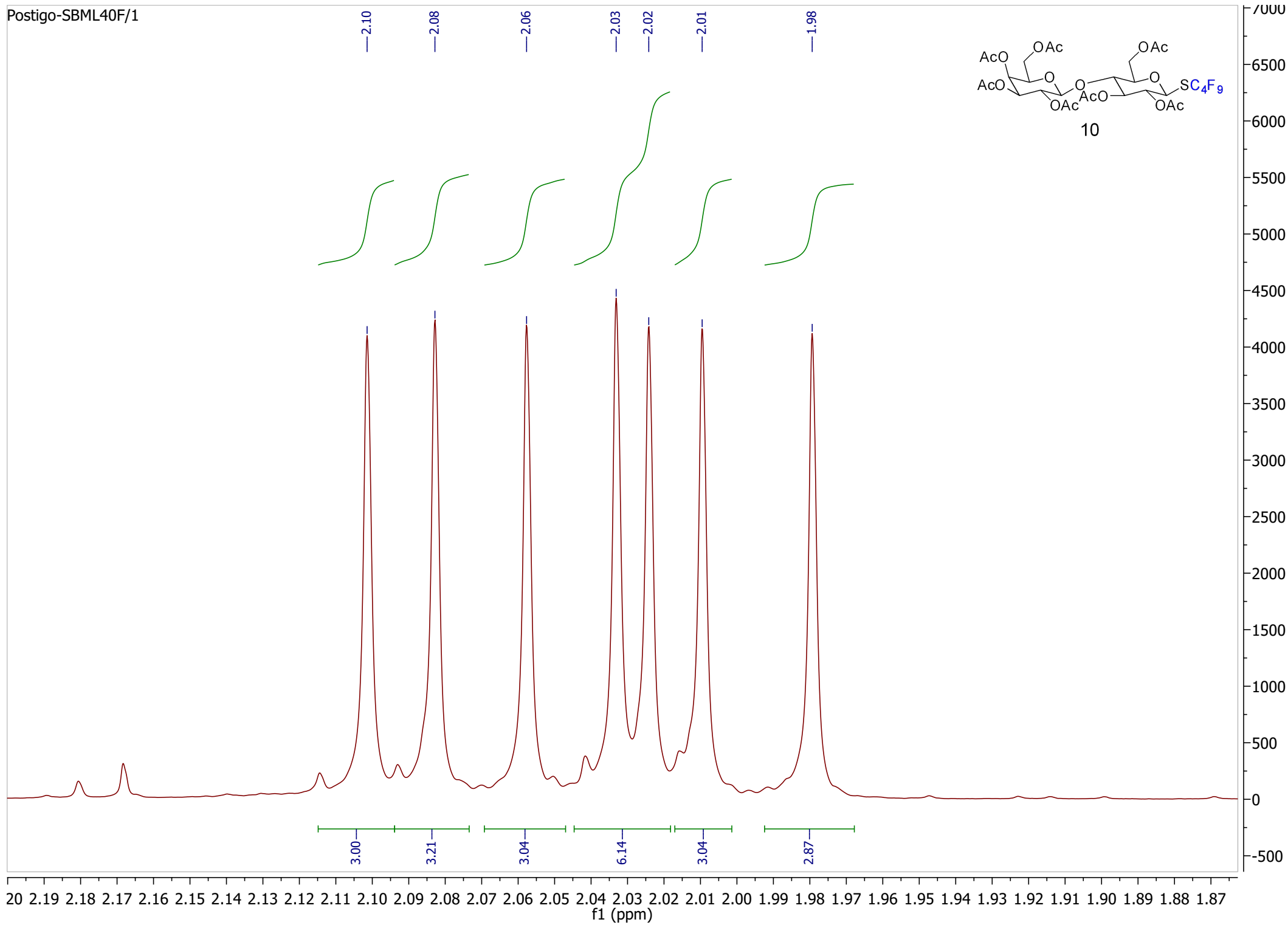


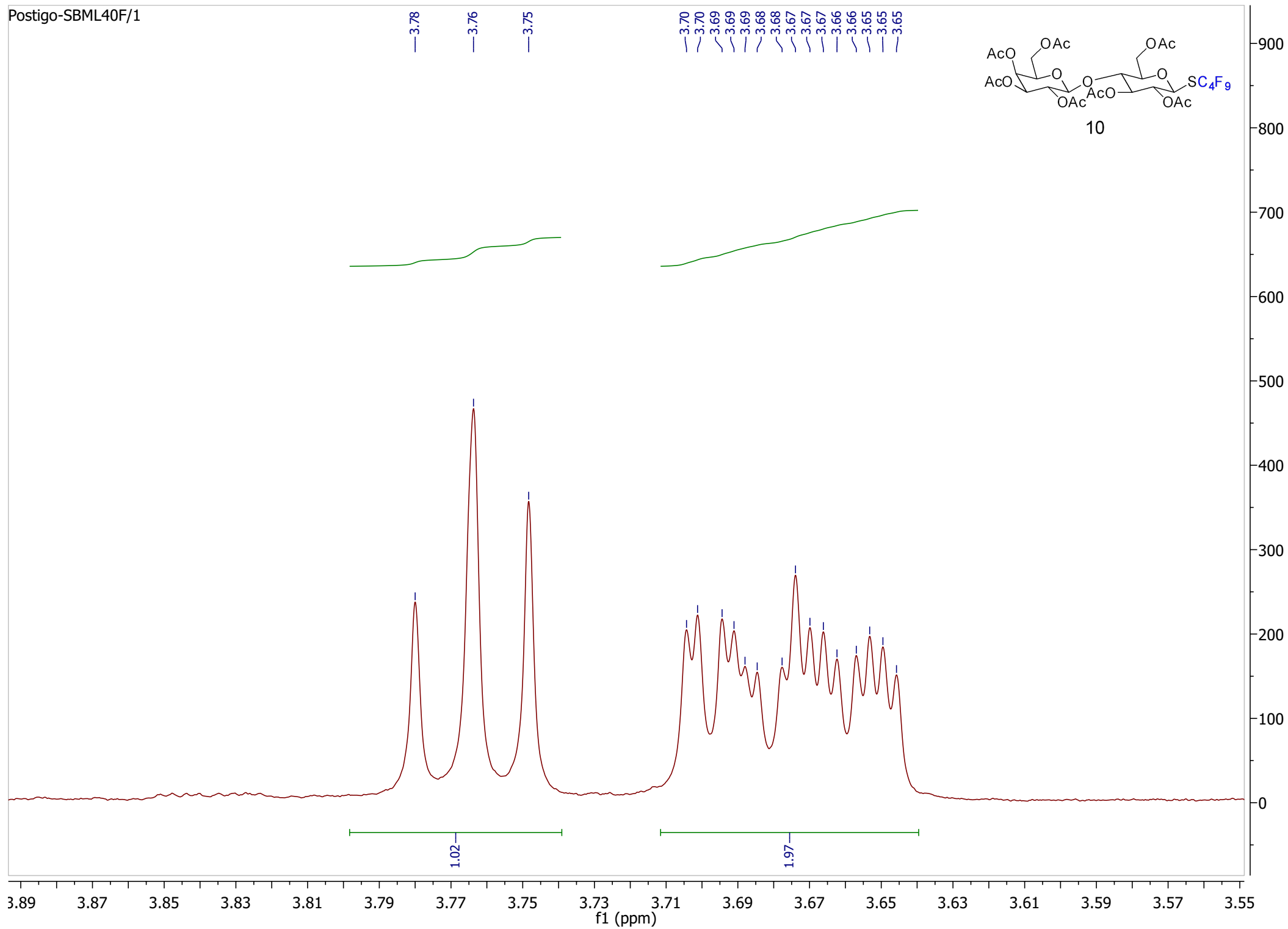


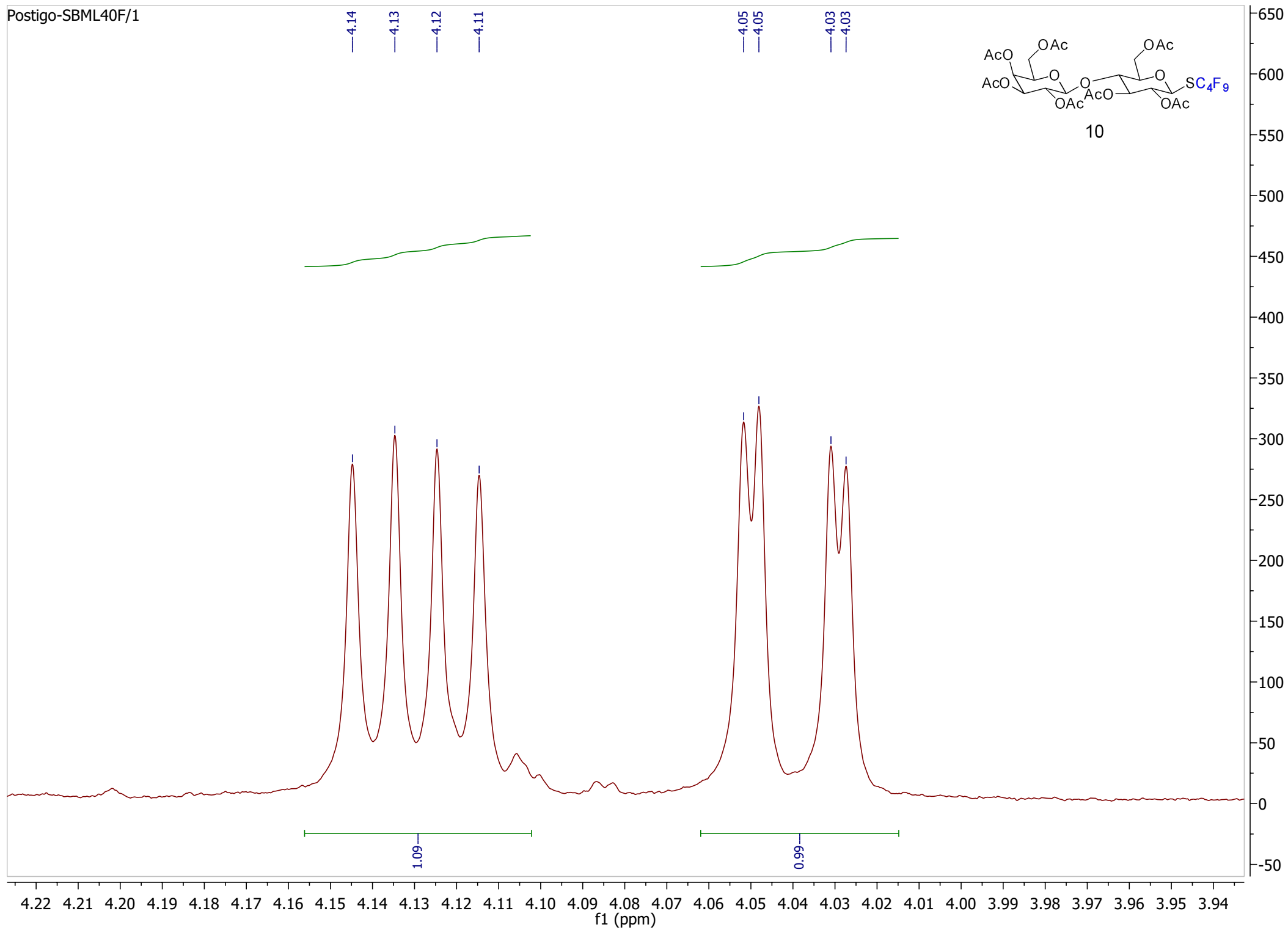
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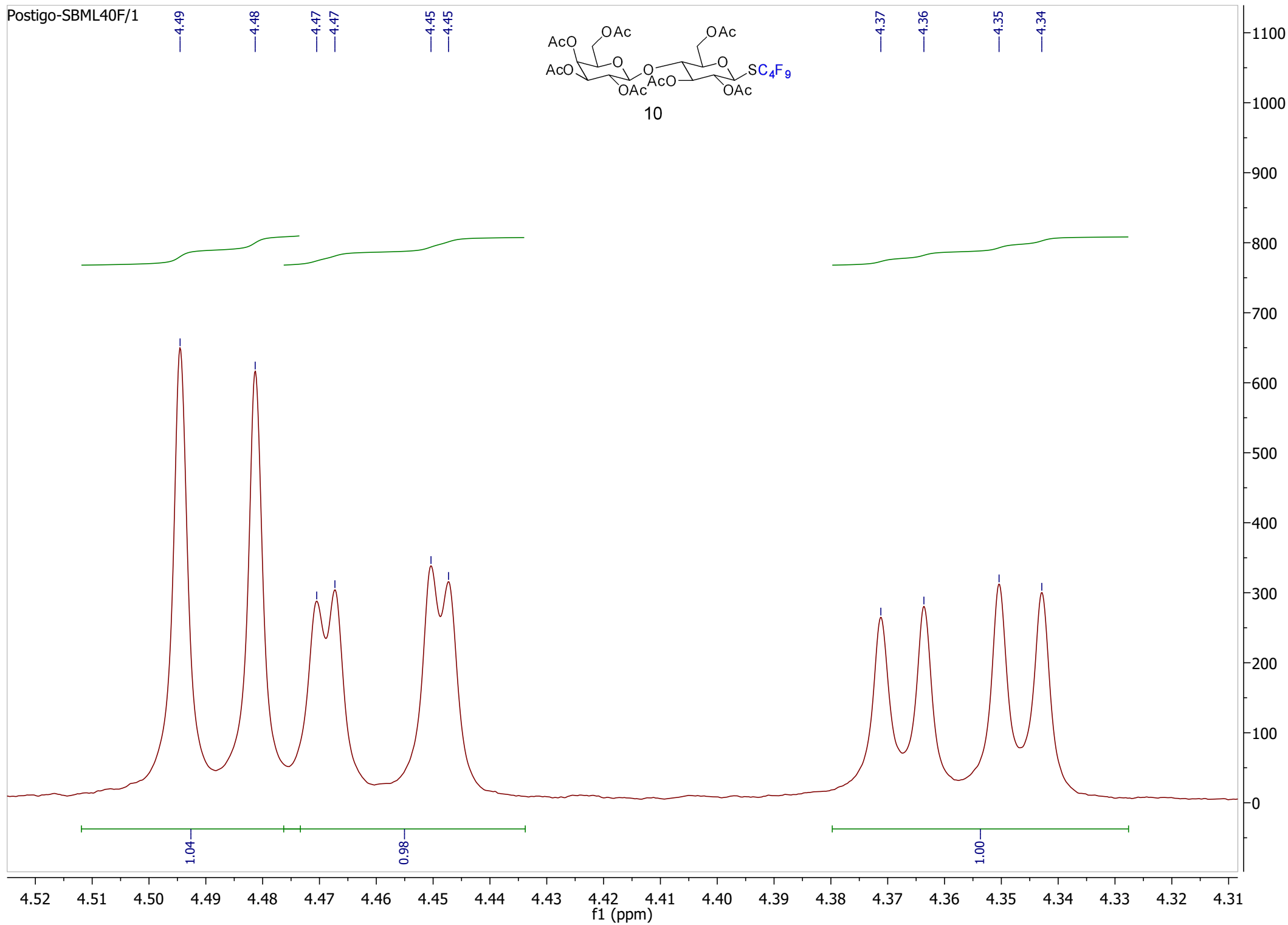


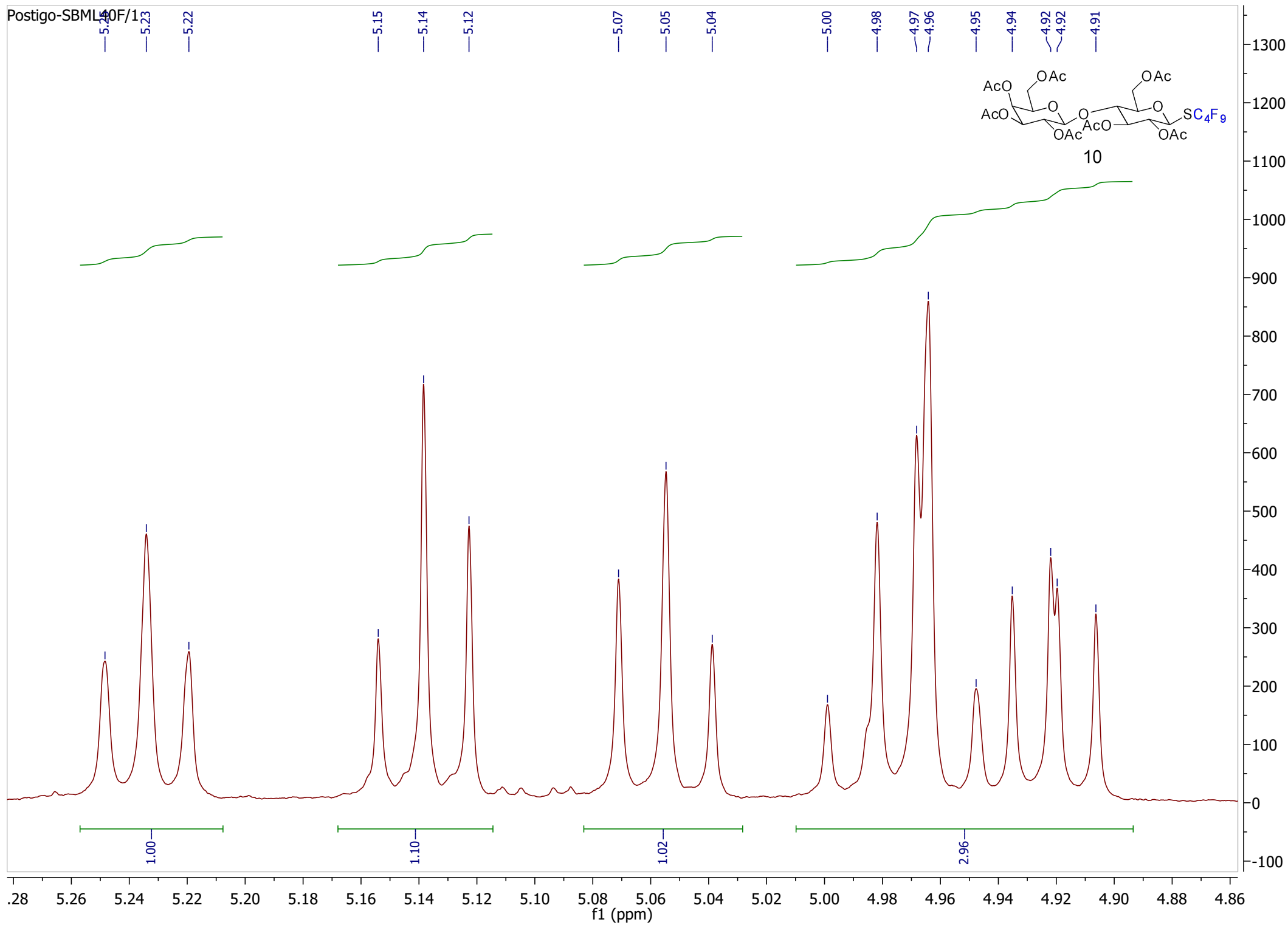


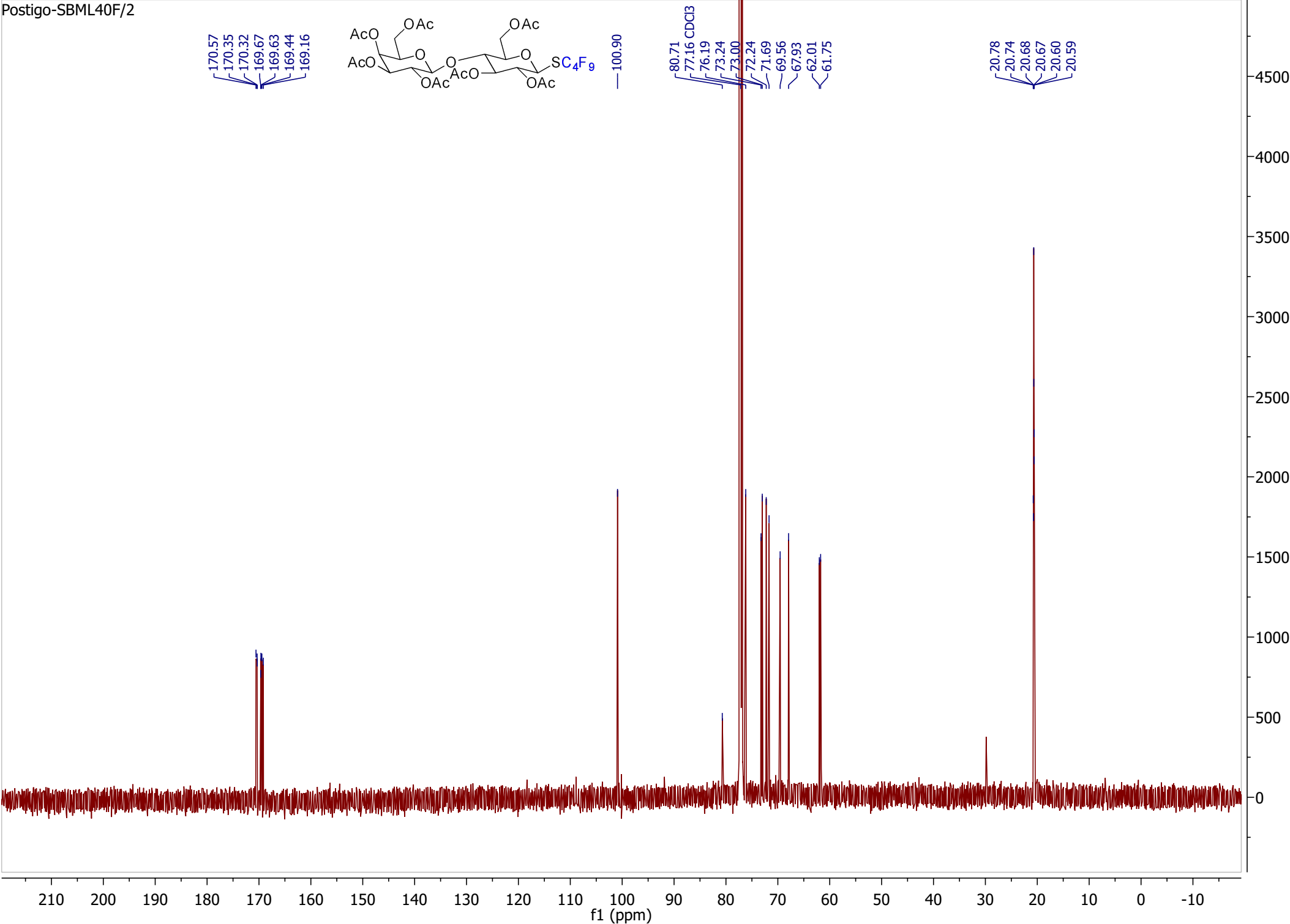


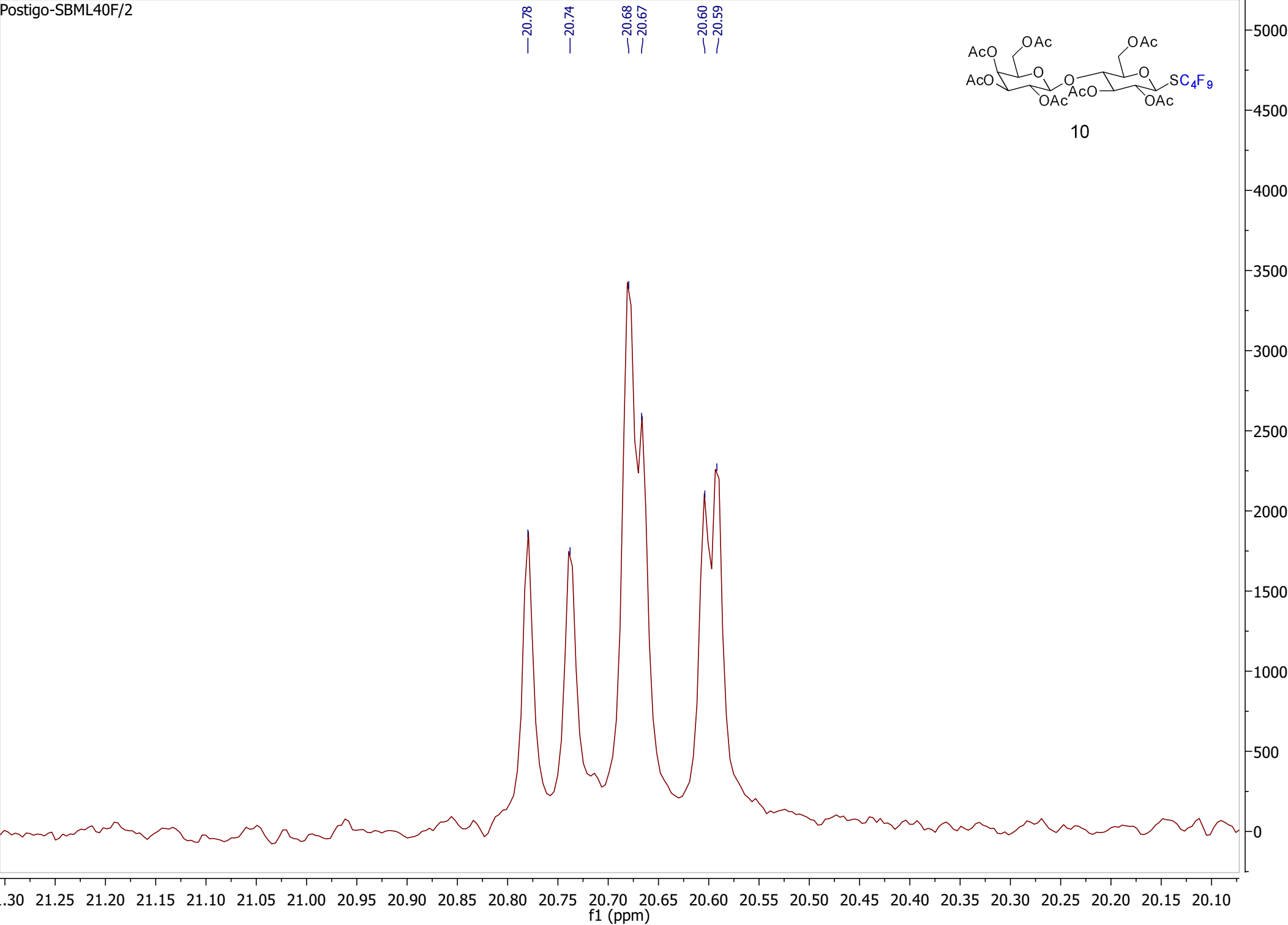


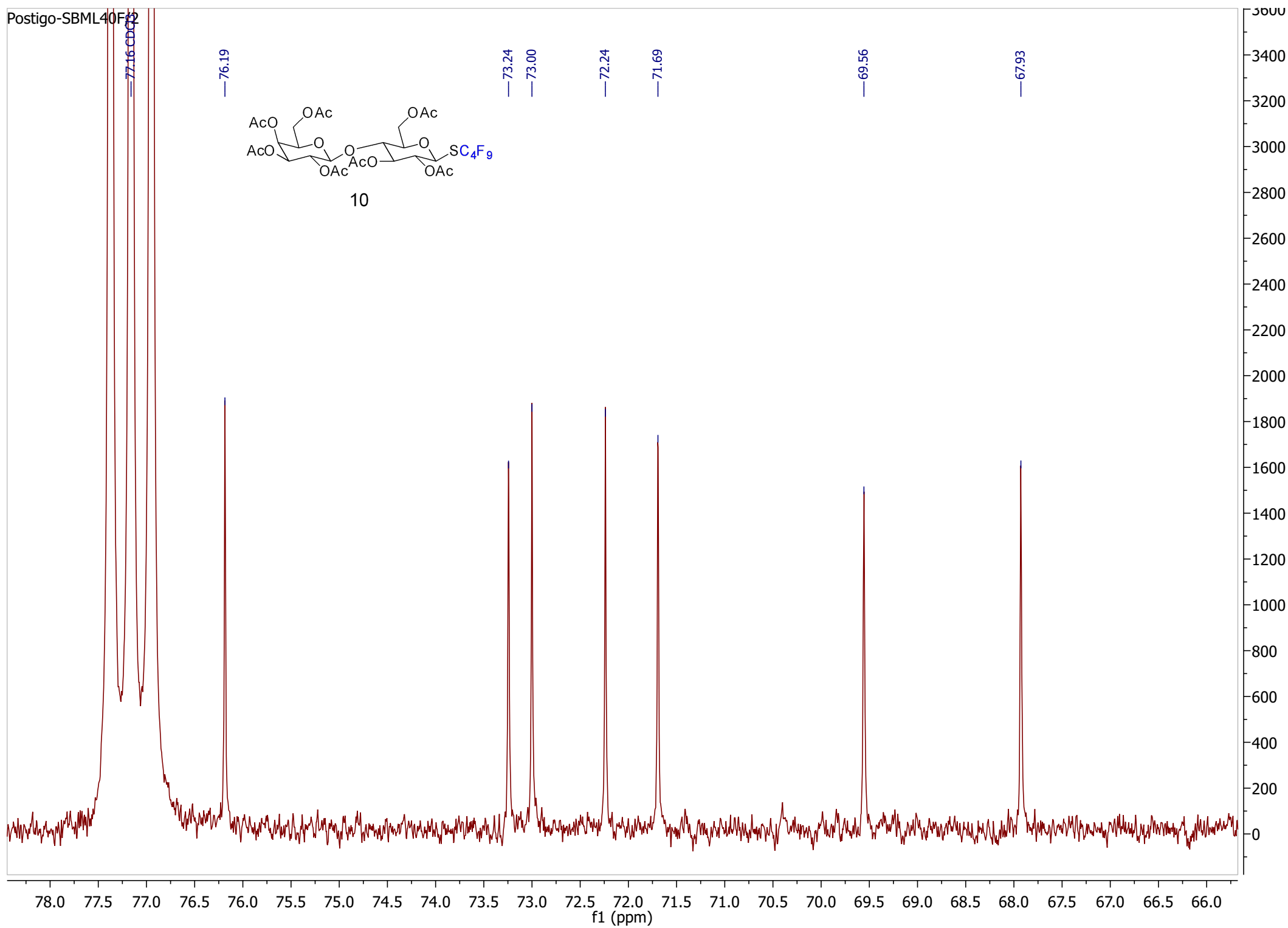


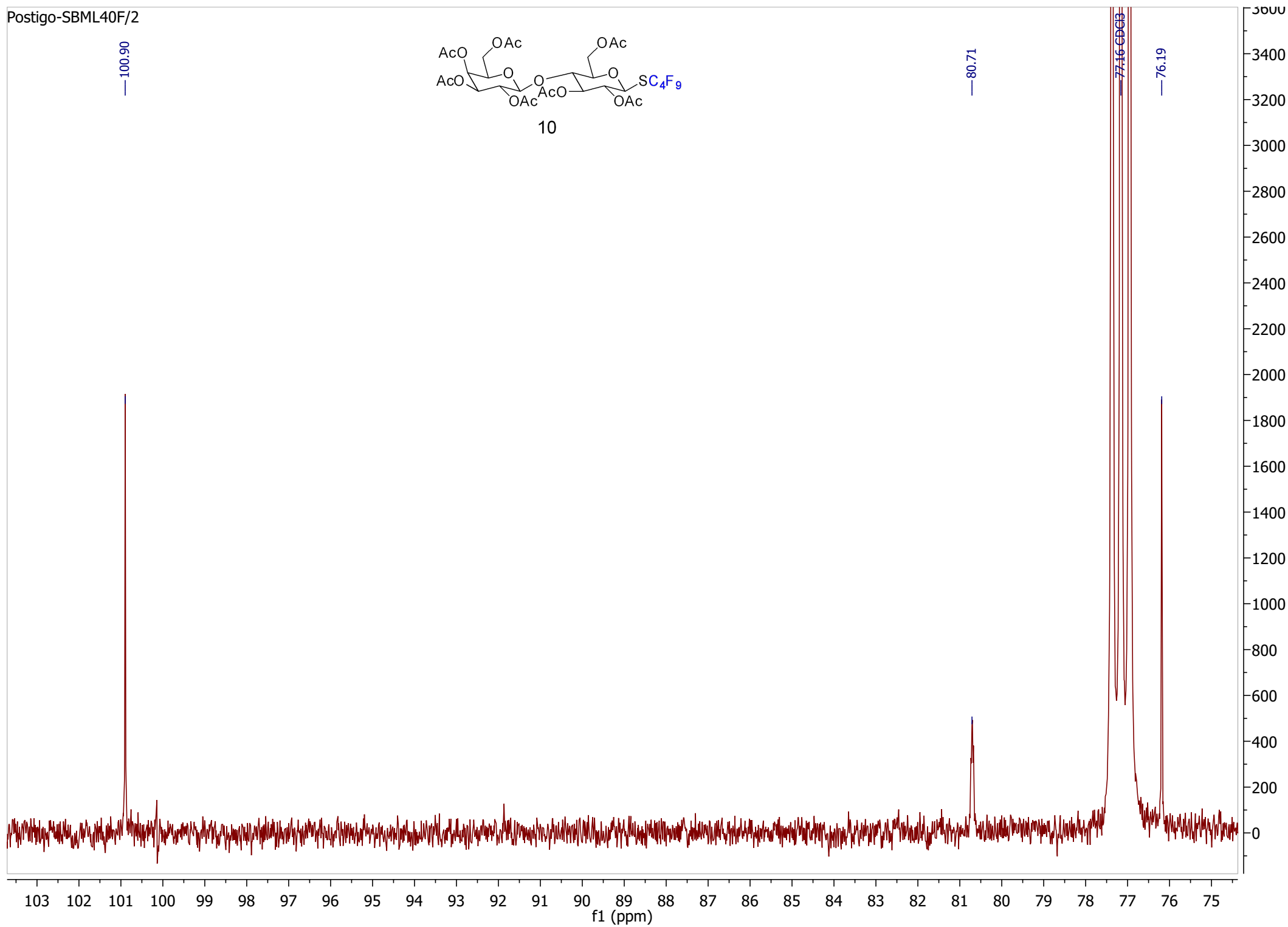


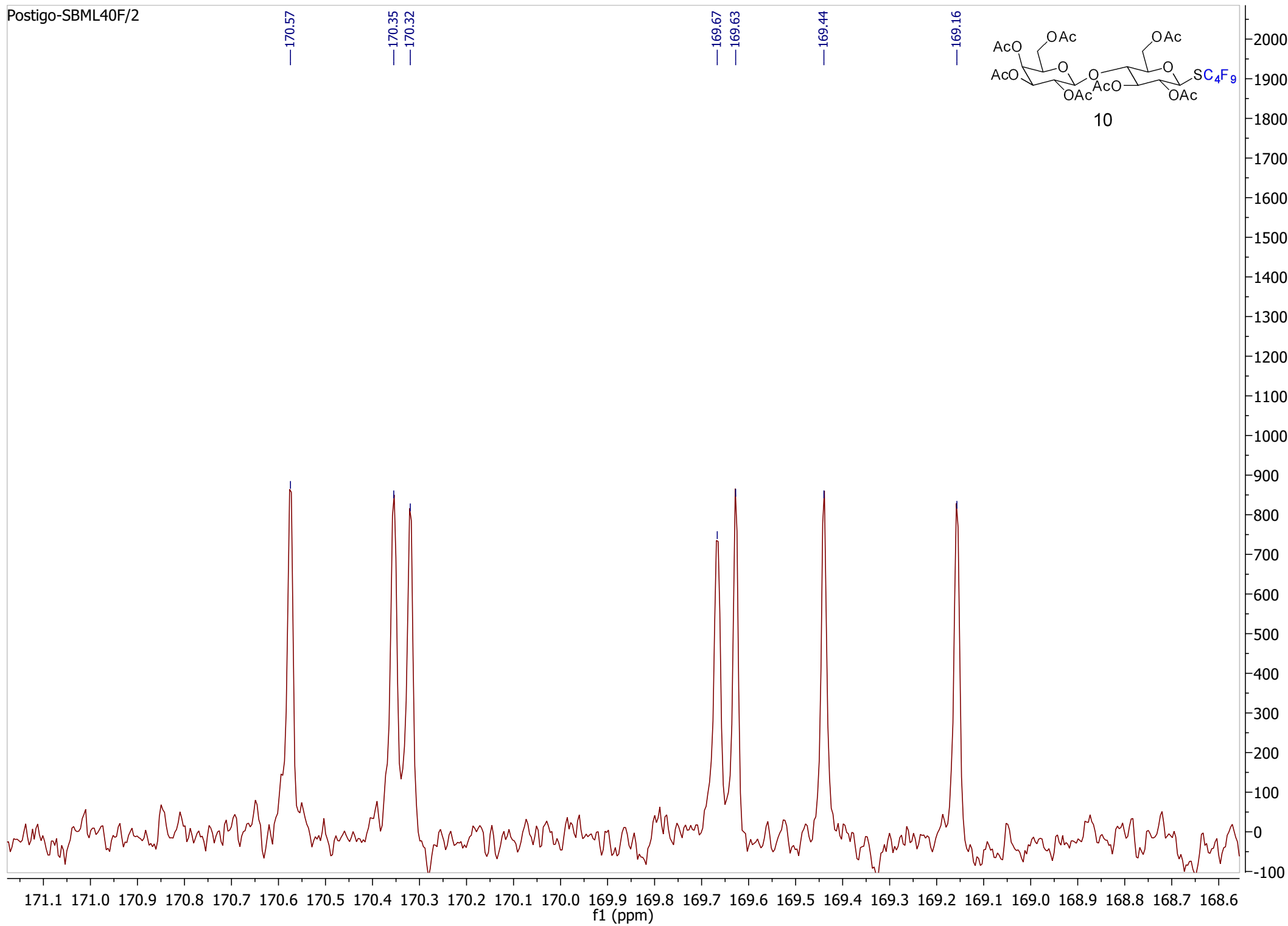


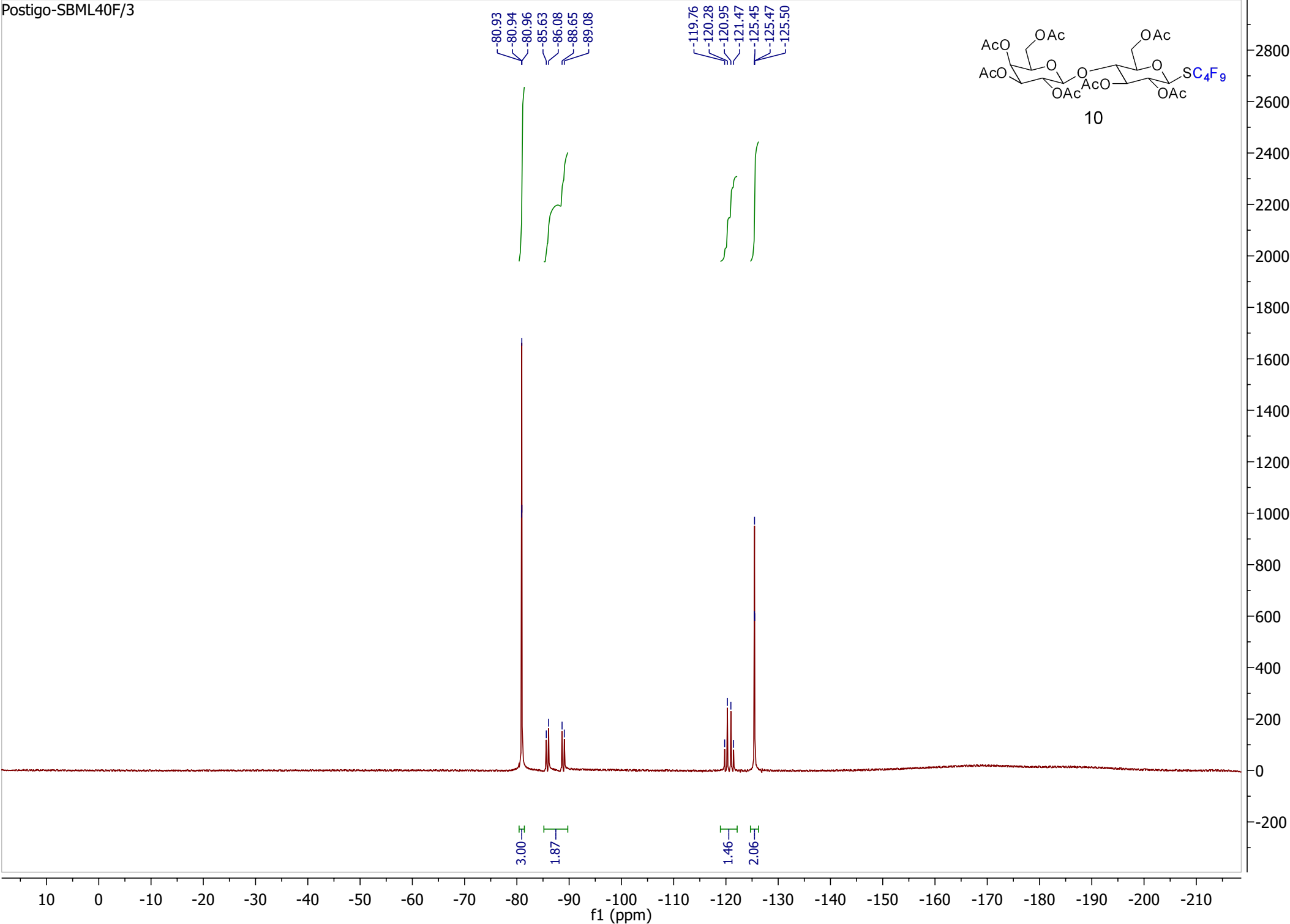


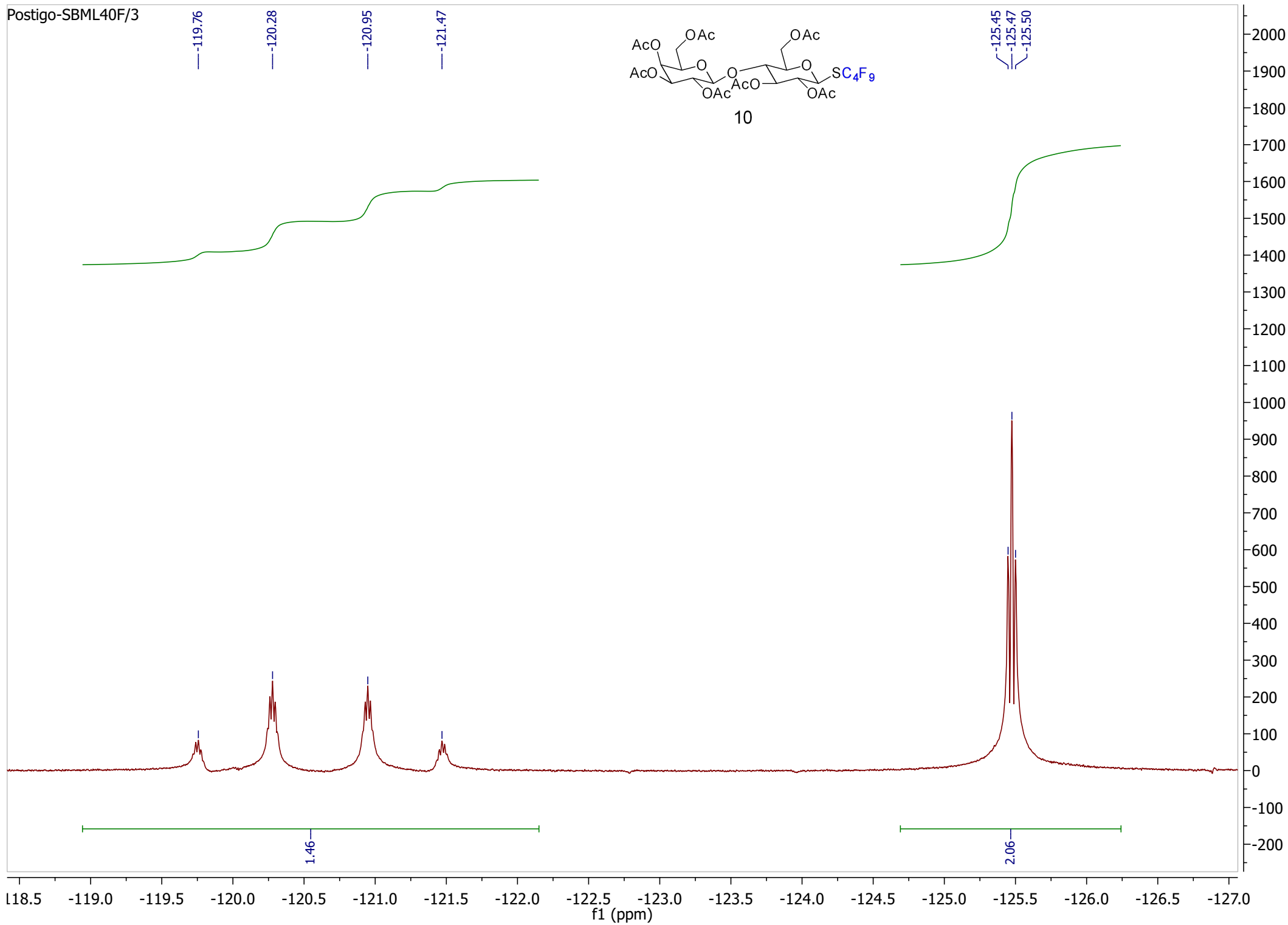


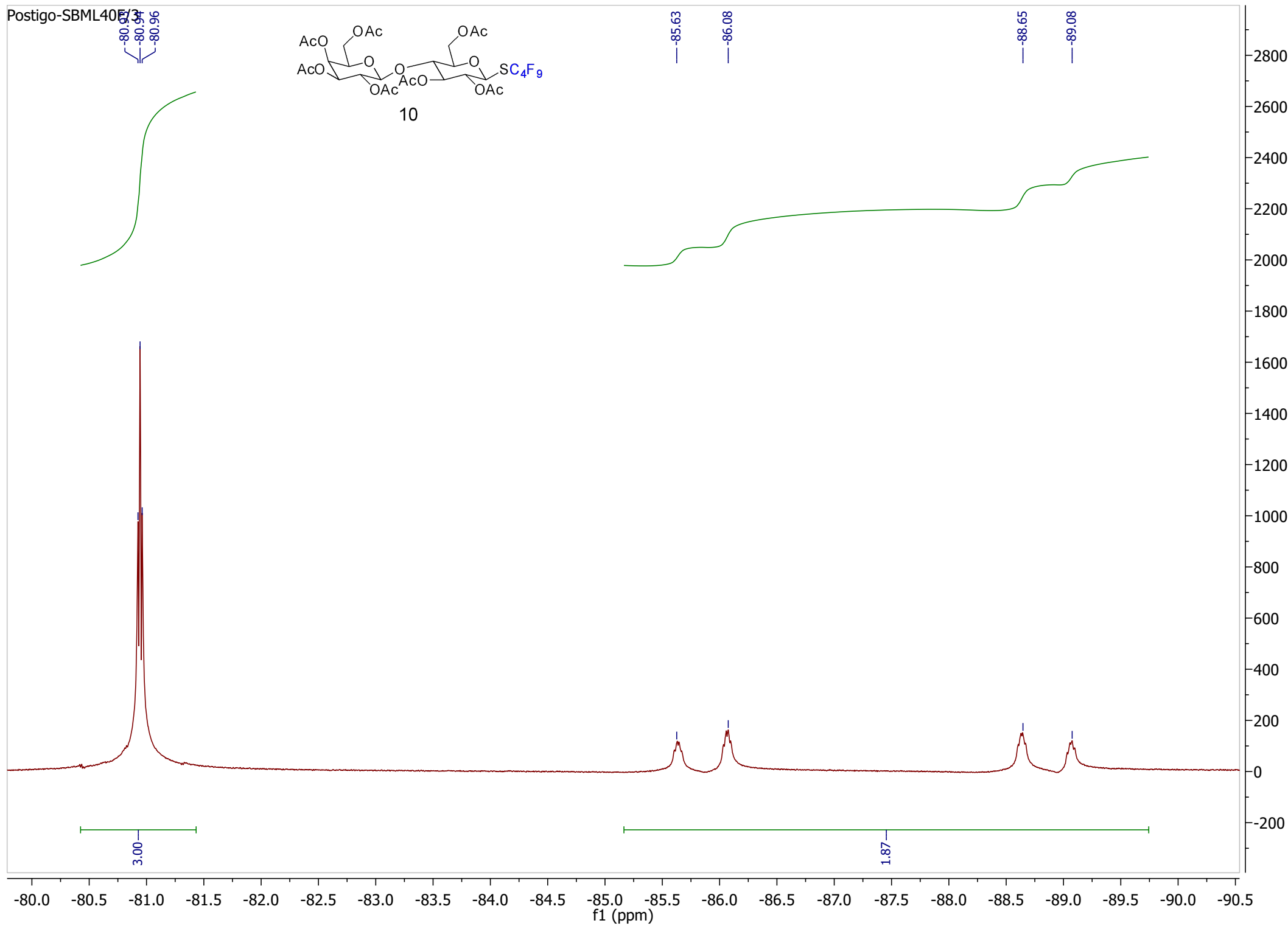


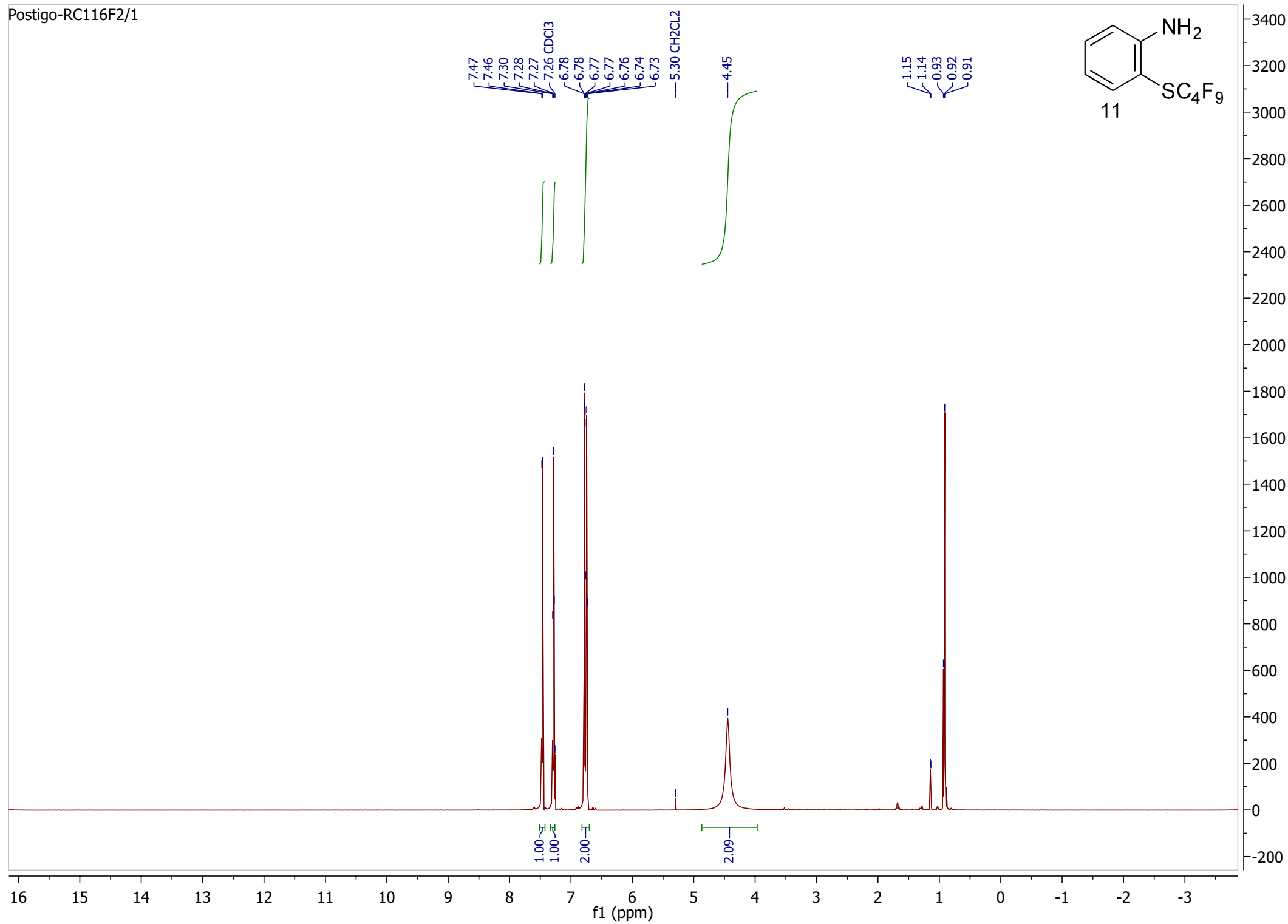








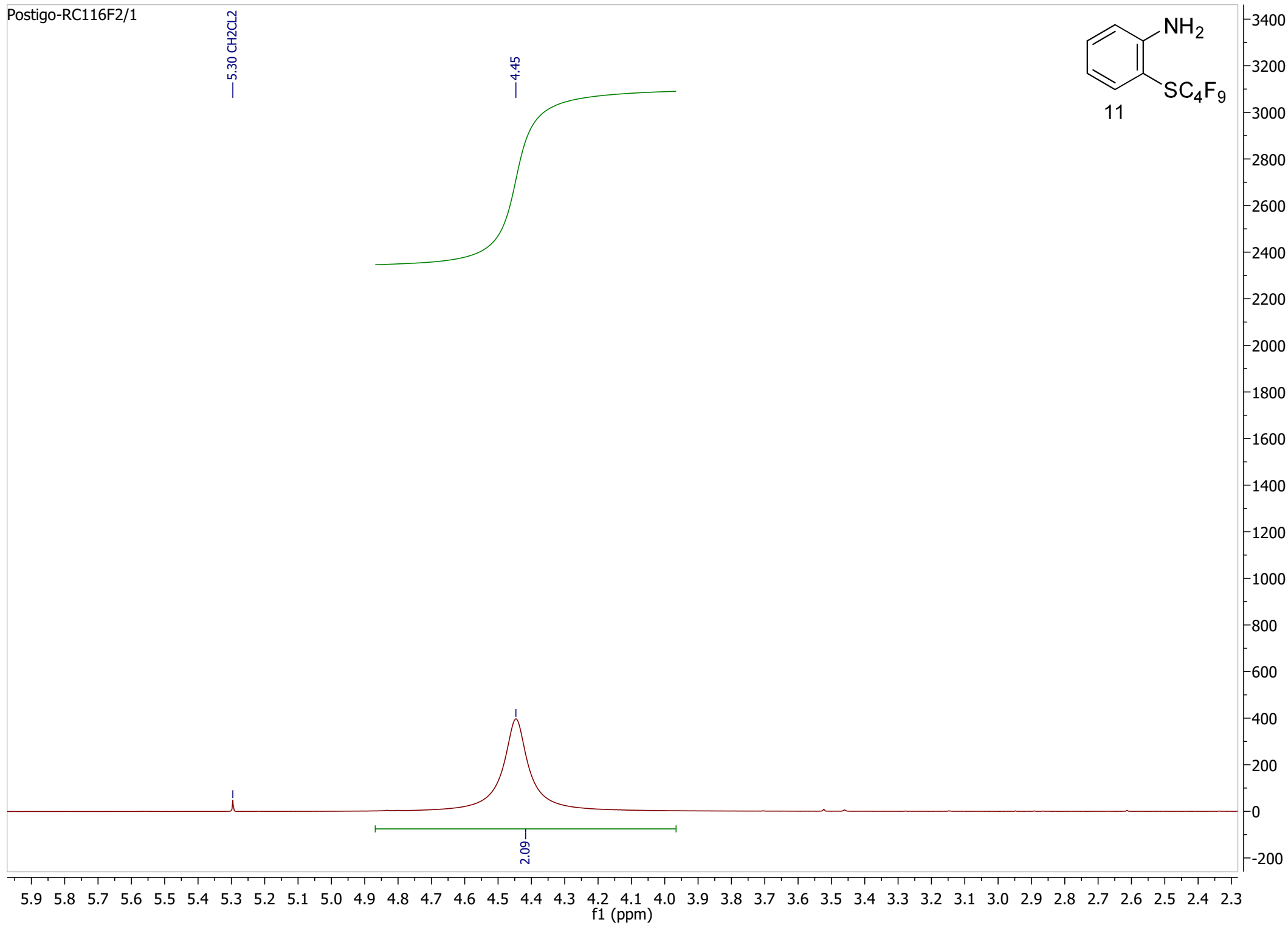
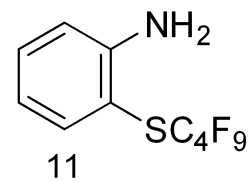


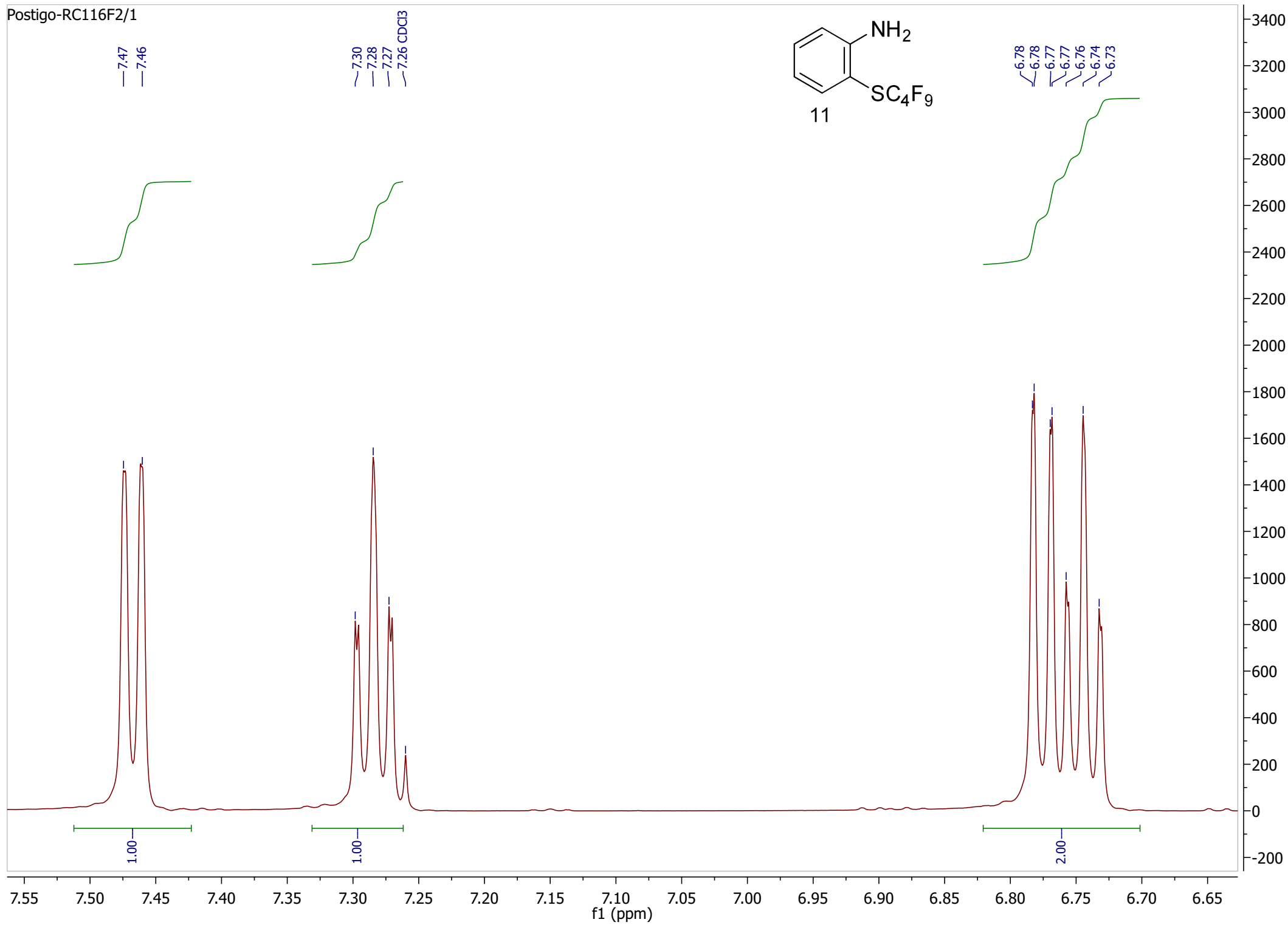


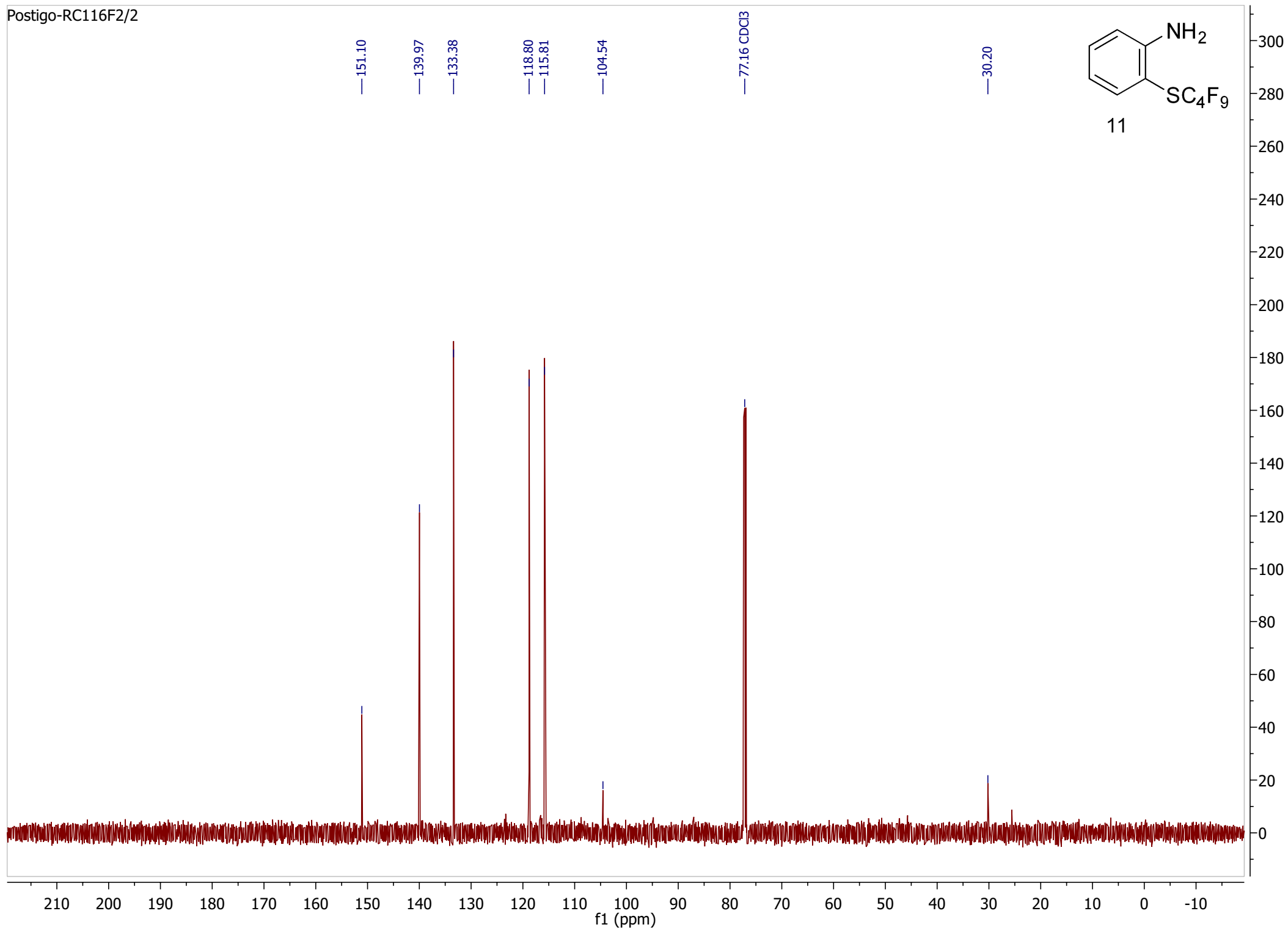
— 5.30 CH₂Cl₂

— 4.45

2.09



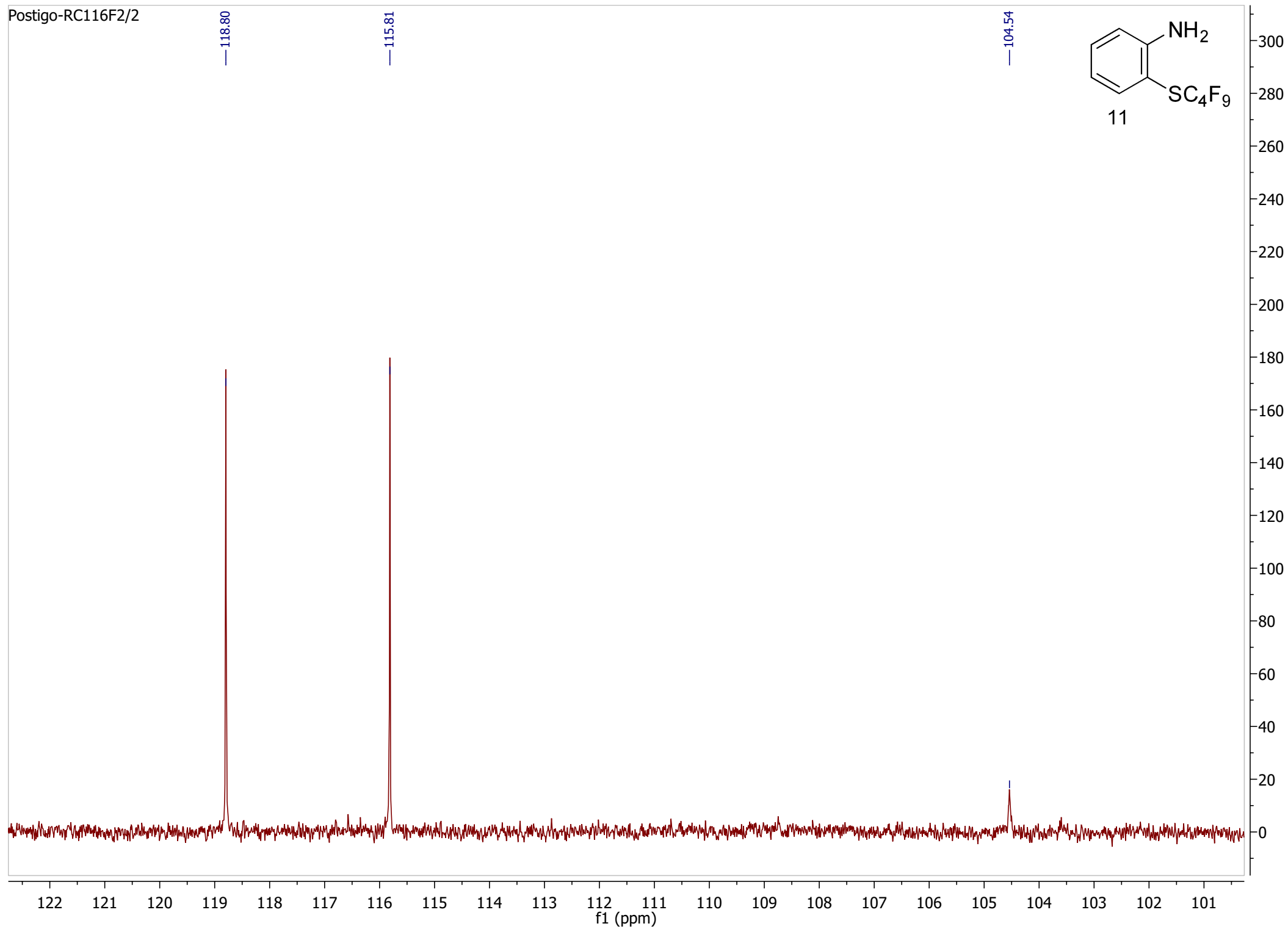
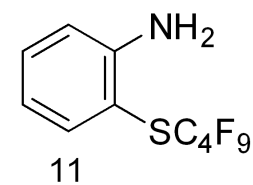


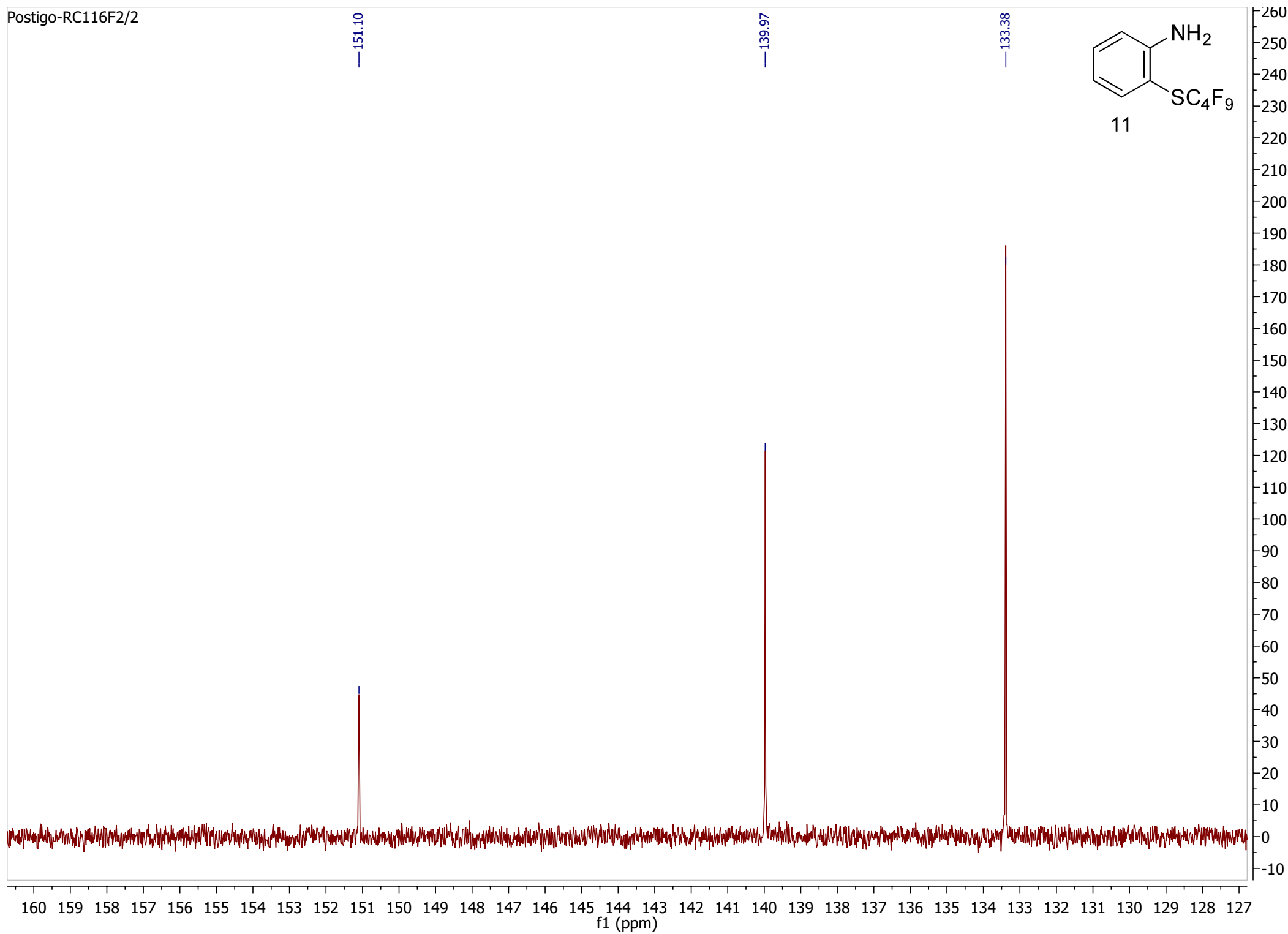


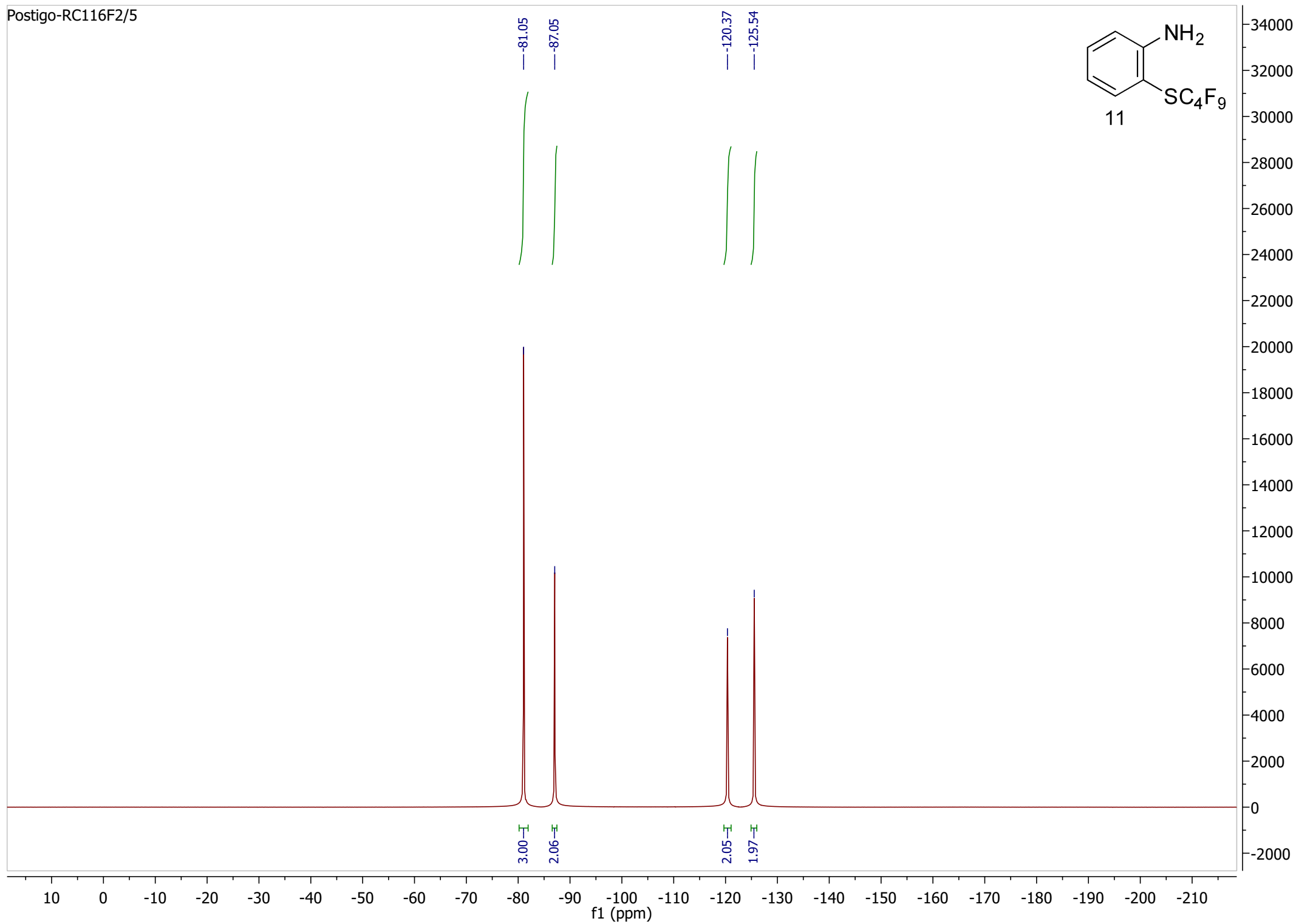
—118.80

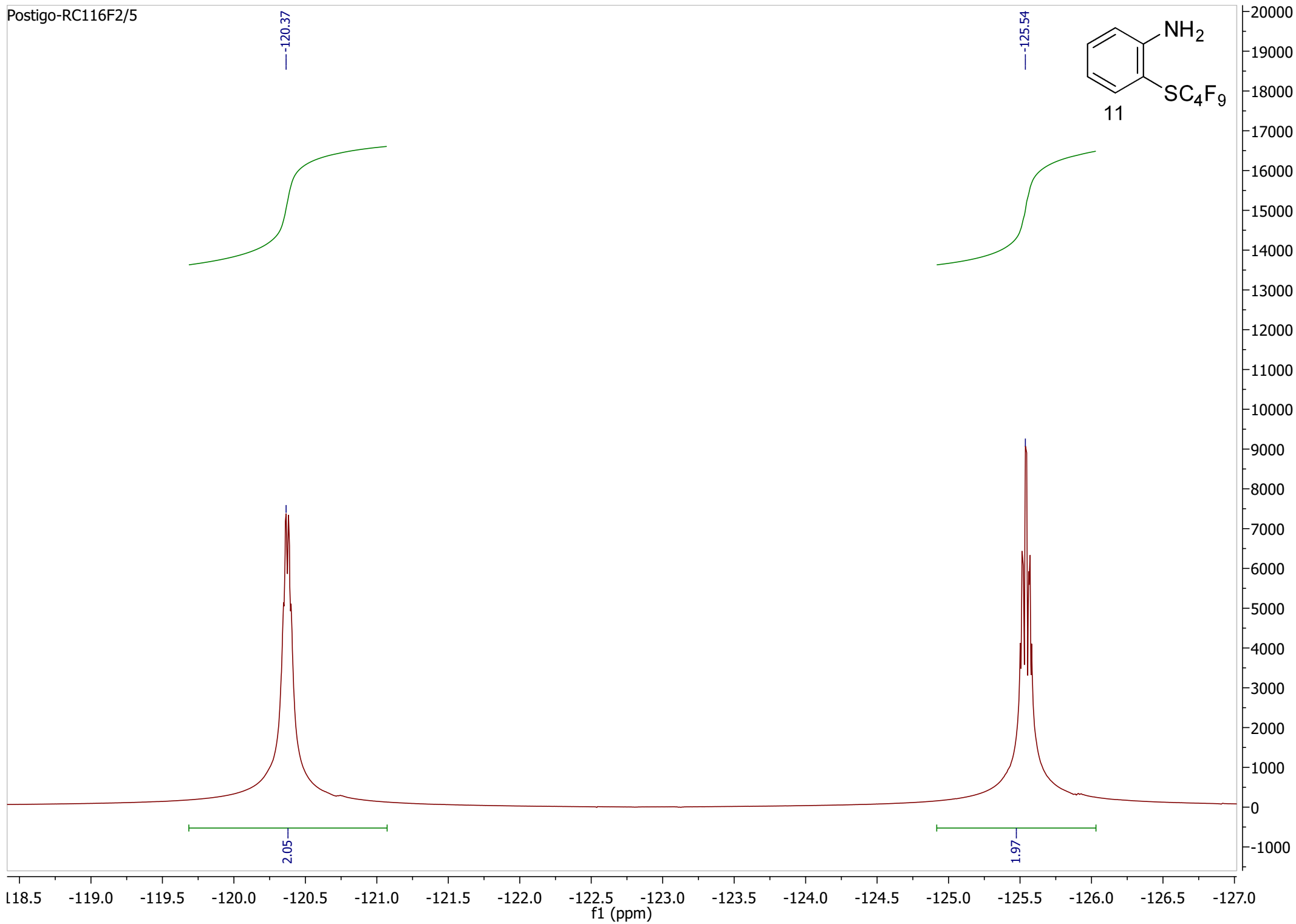
—115.81

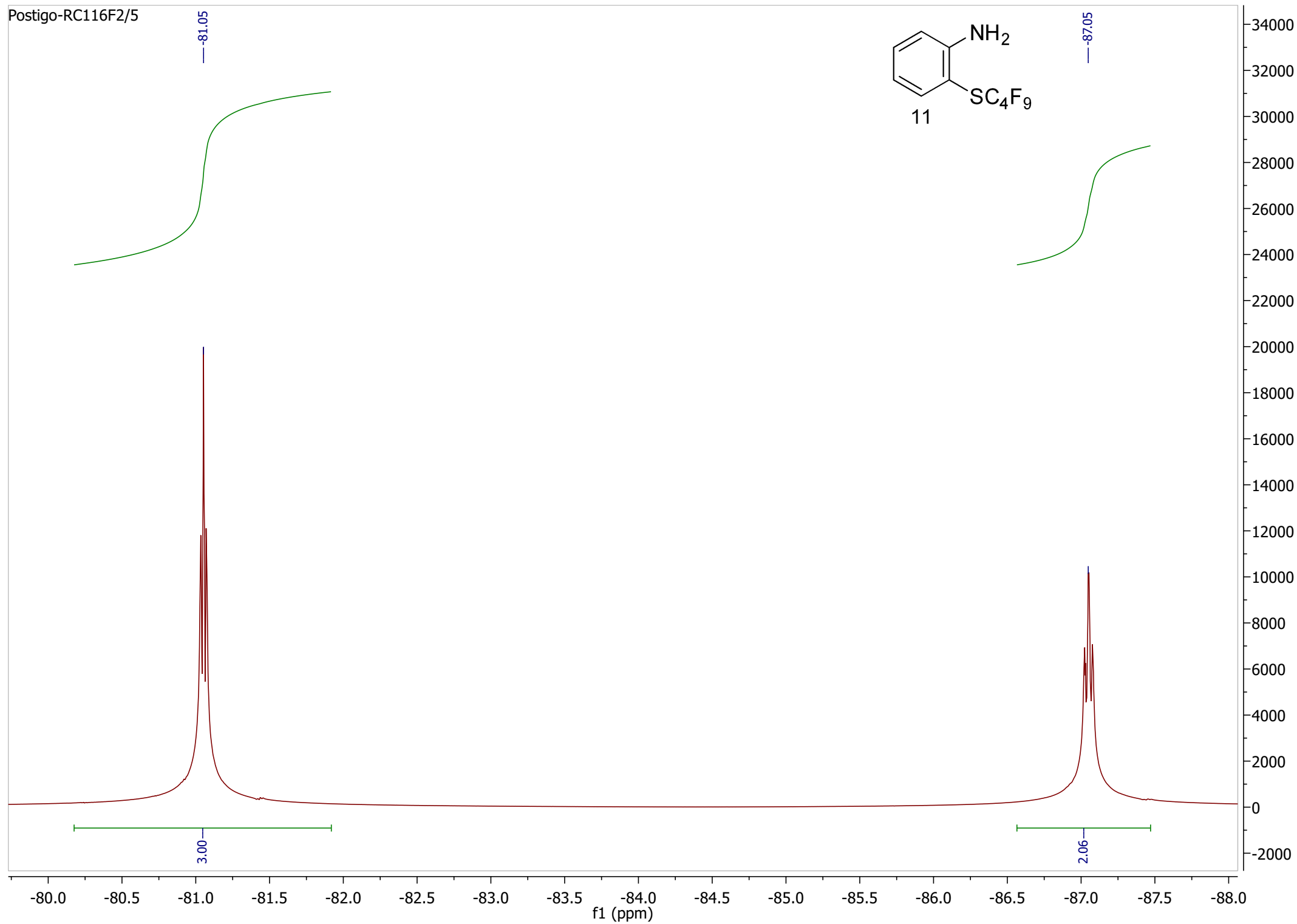
—104.54



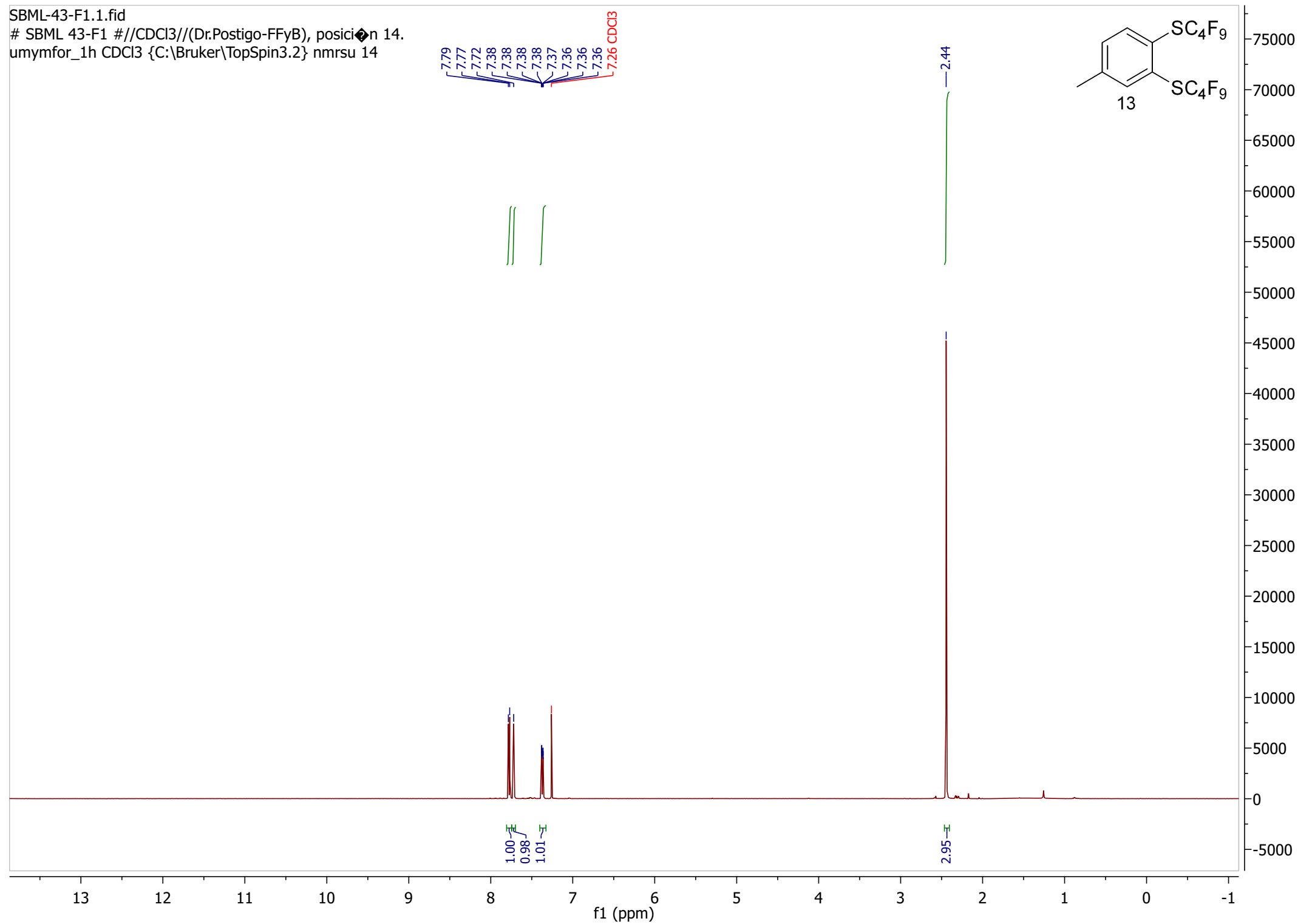








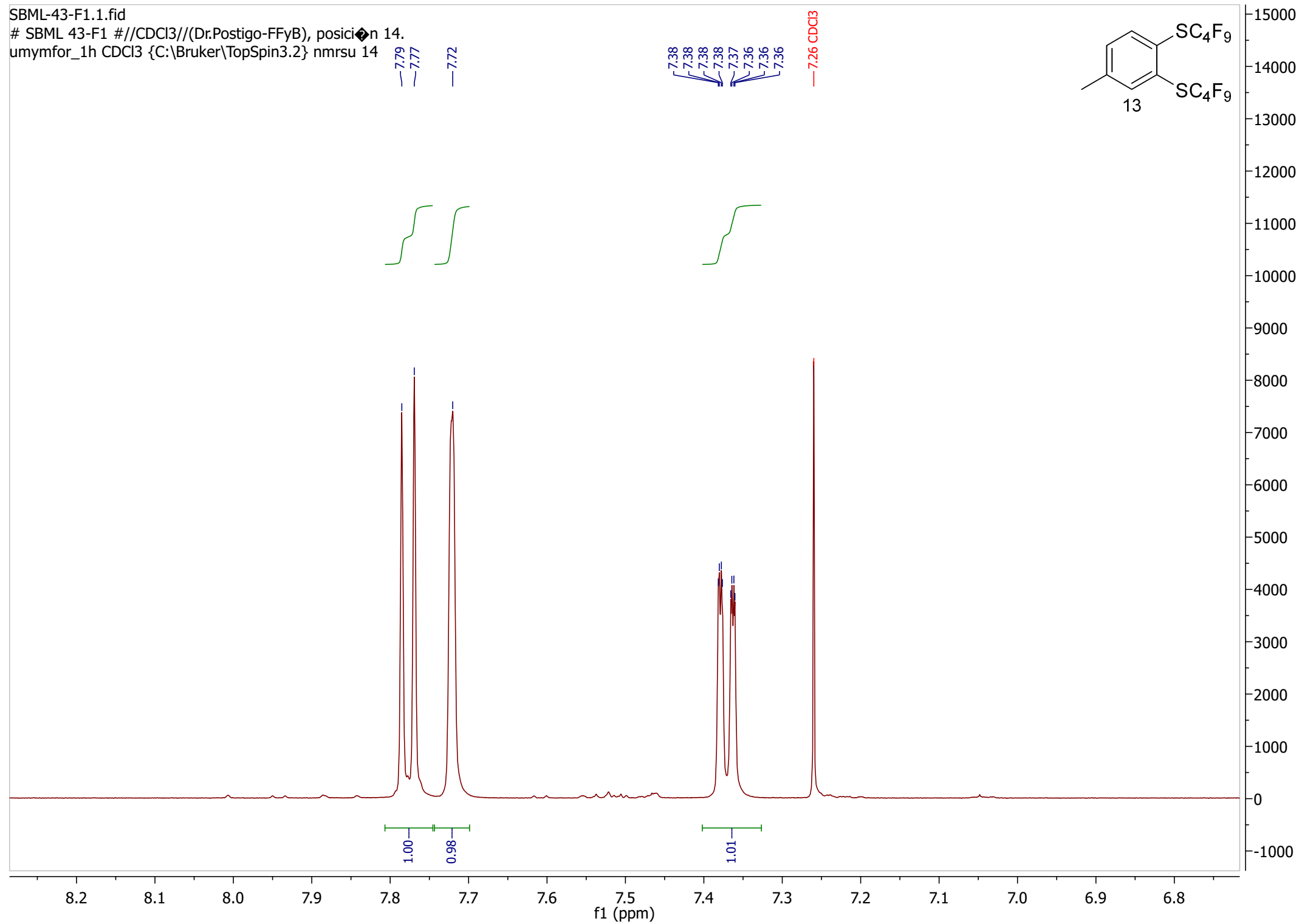
SBML-43-F1.1.fid
SBML 43-F1 #//CDCl3//(Dr.Postigo-FFyB), posición 14.
umymfor_1h CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 14



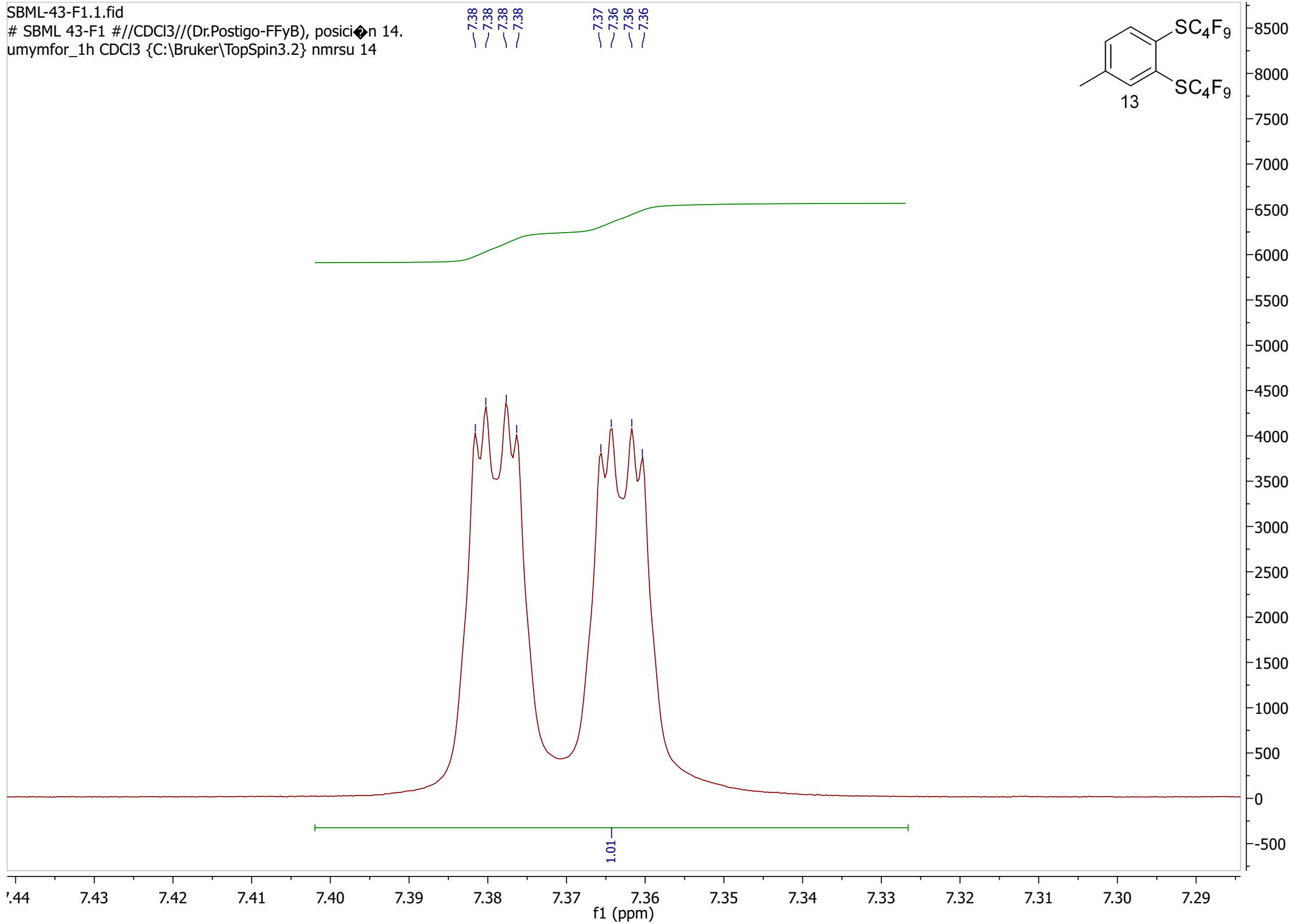
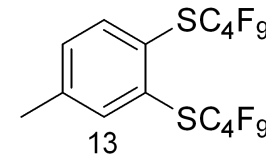
SBML-43-F1.1.fid

SBML 43-F1 #//CDCl3// (Dr.Postigo-FFyB), posición 14.

umymfor_1h CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 14



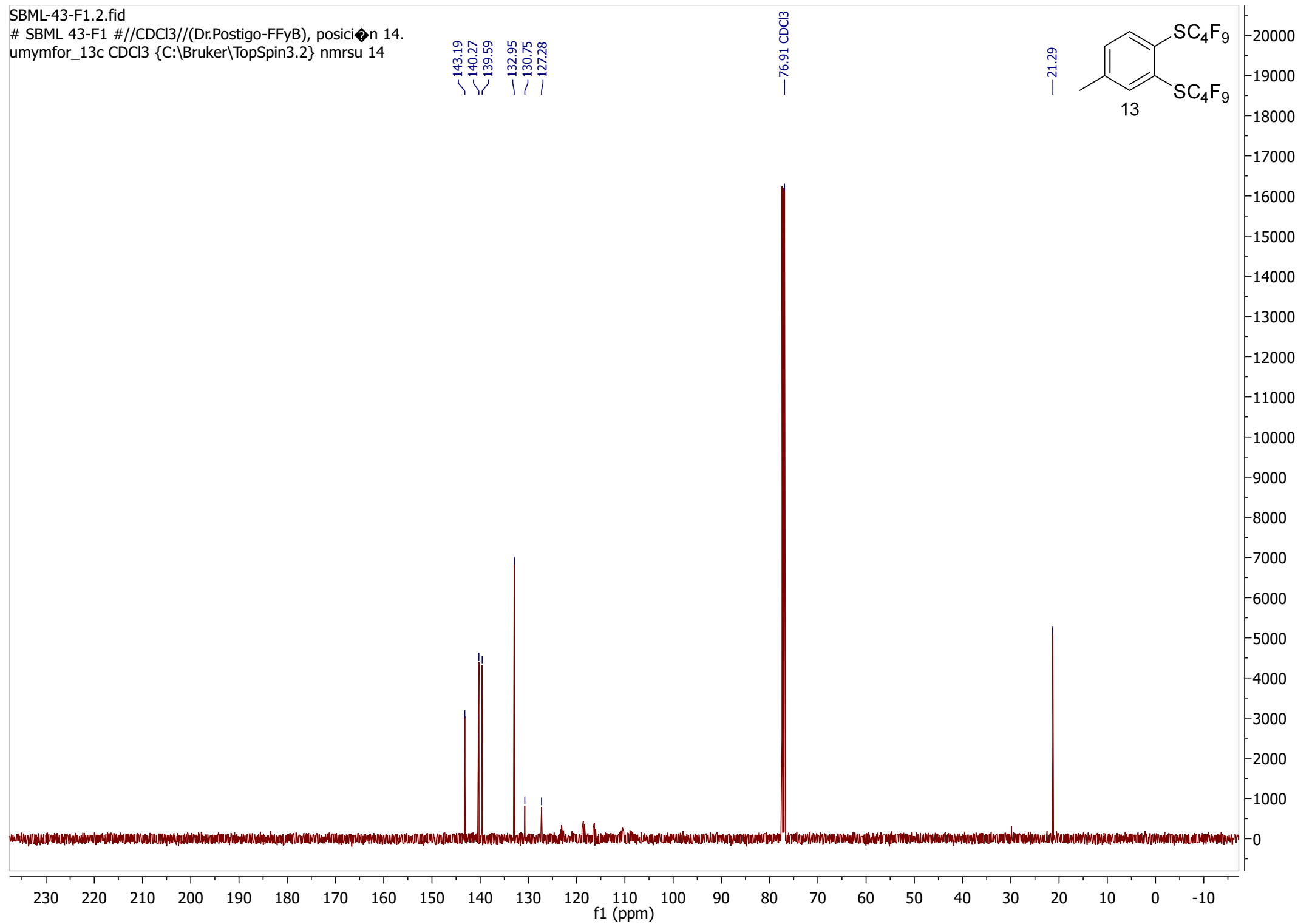
SBML-43-F1.1.fid
SBML 43-F1 #//CDCl3//(Dr.Postigo-FFyB), position 14.
umymfor_1h CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 14



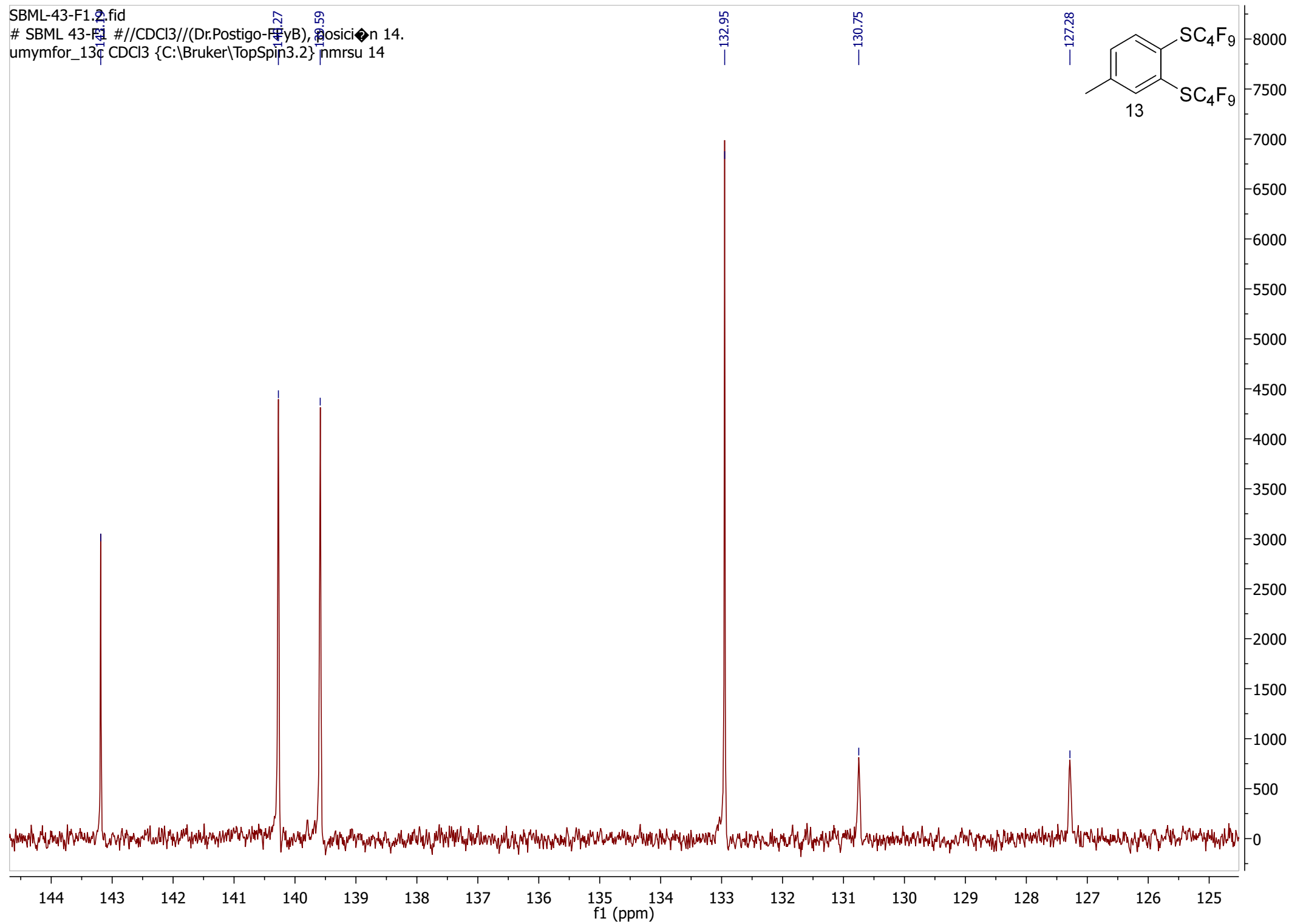
SBML-43-F1.2.fid

SBML 43-F1 #//CDCl3/(Dr.Postigo-FFyB), posición 14.

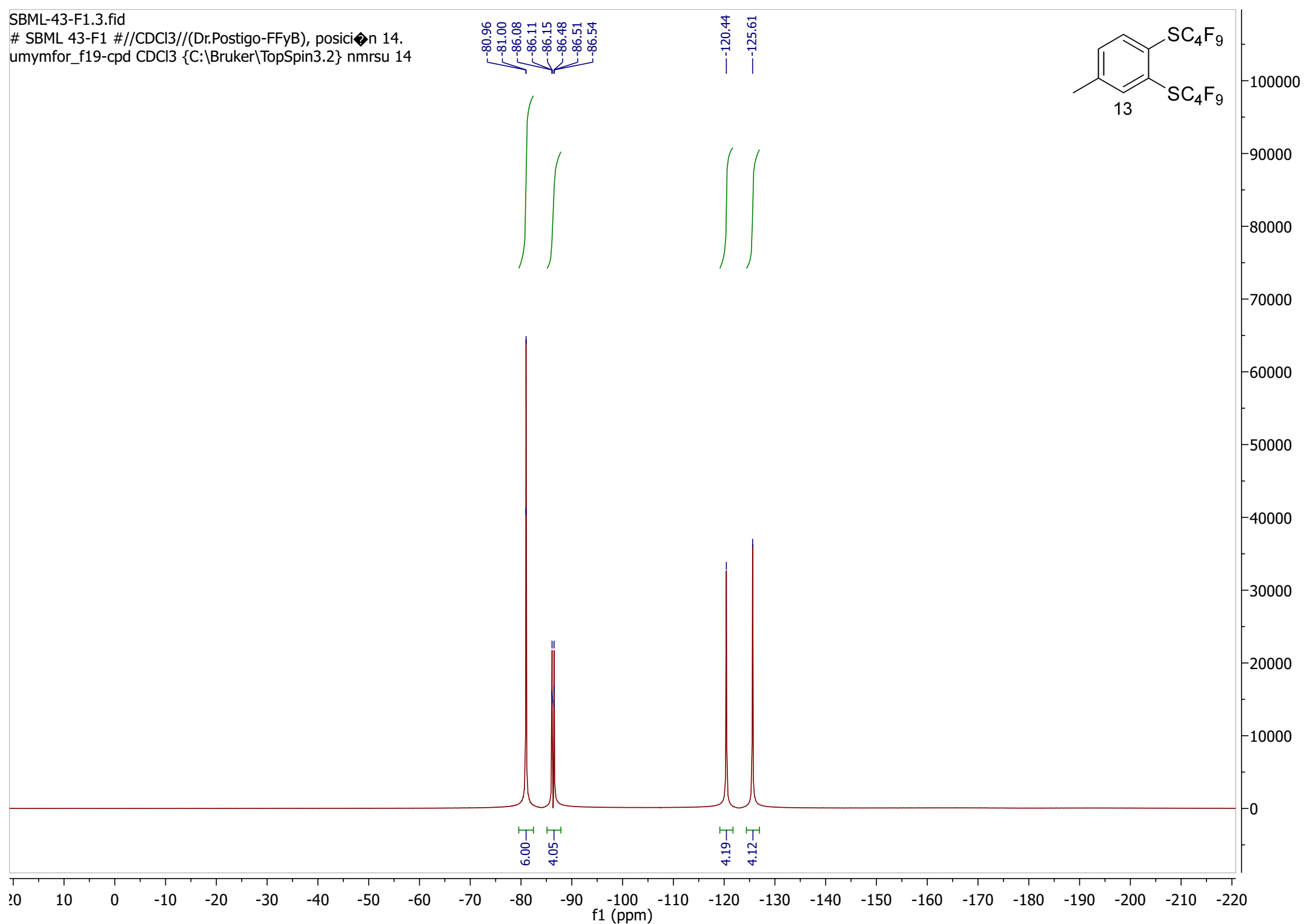
umymfor_13c CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 14



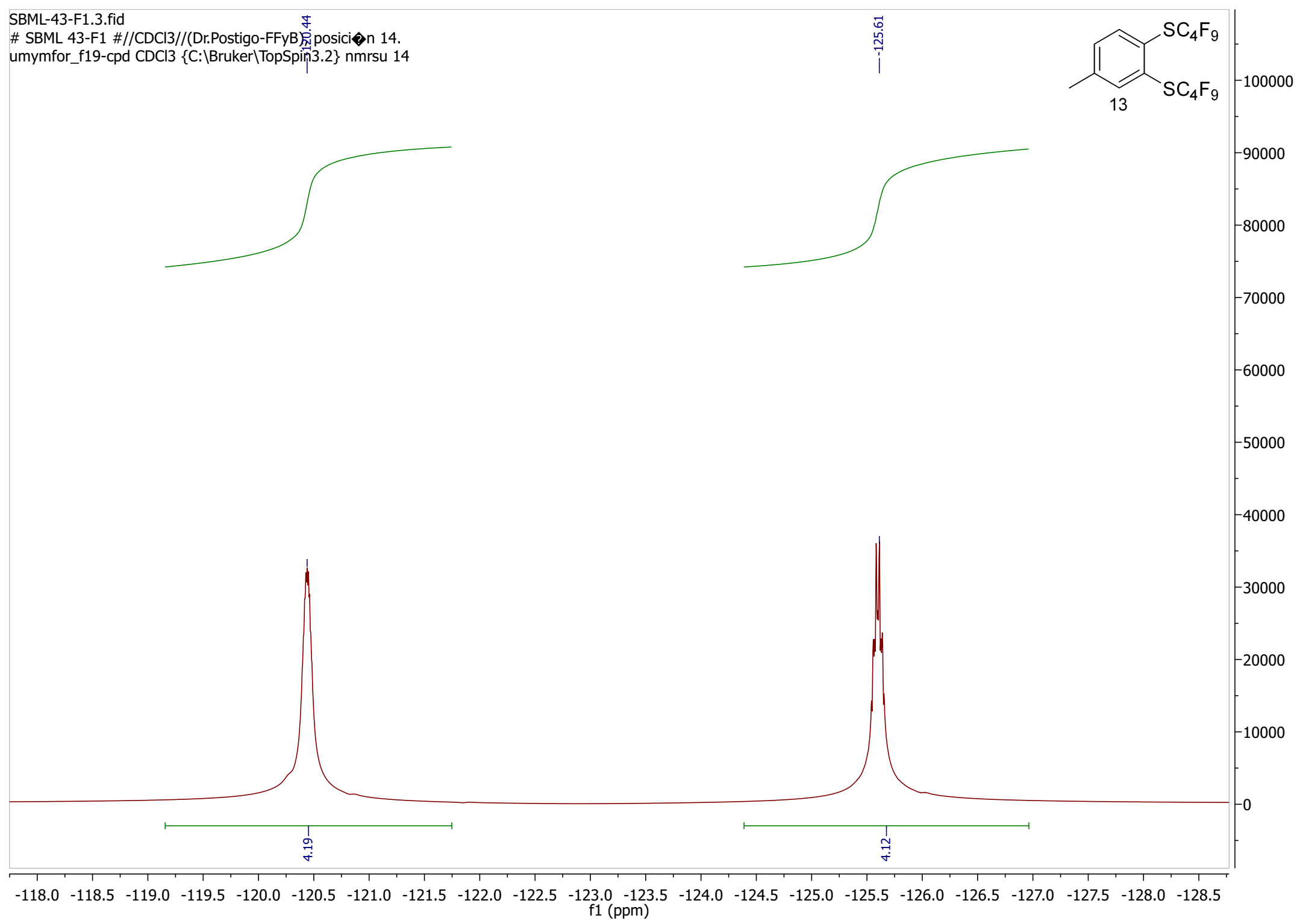
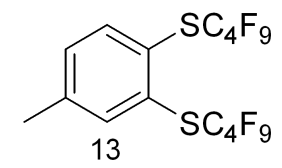
SBML-43-F1.2.fid
SBML 43-F1 #//CDCl3/(Dr.Postigo-FlyB), position 14.
umymfor_13c CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 14



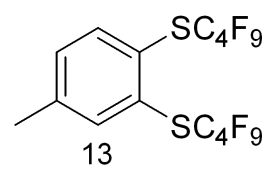
SBML-43-F1.3.fid
SBML 43-F1 #//CDCl3// (Dr.Postigo-FFyB), posición 14.
umymfor_f19-cpd CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 14



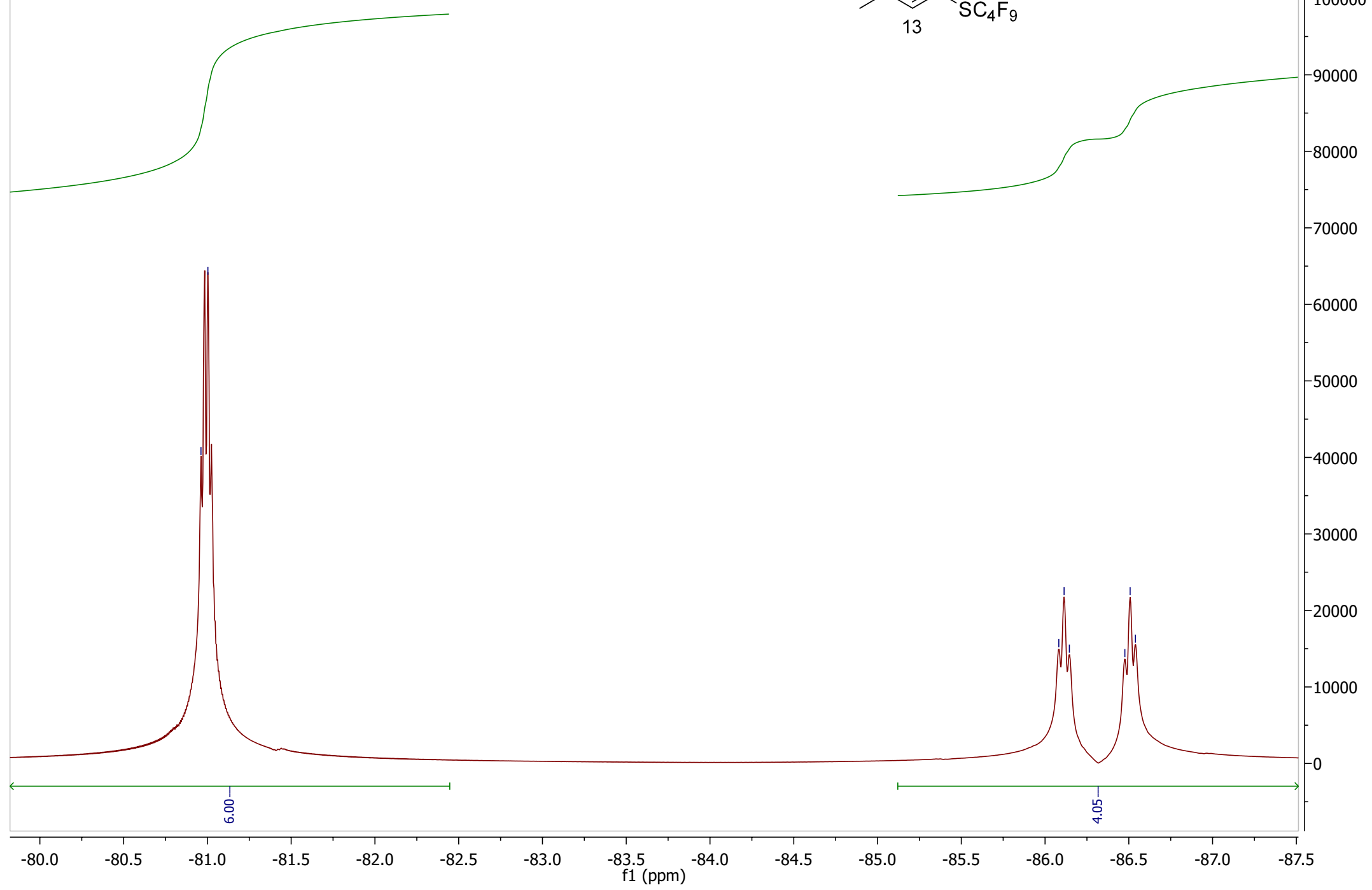
SBML-43-F1.3.fid
SBML 43-F1 #//CDCl3//(Dr.Postigo-FFyB) posición 14.
umymfor_f19-cpd CDCl3 {C:\Bruker\TopSpin3.2} nmrsu 14

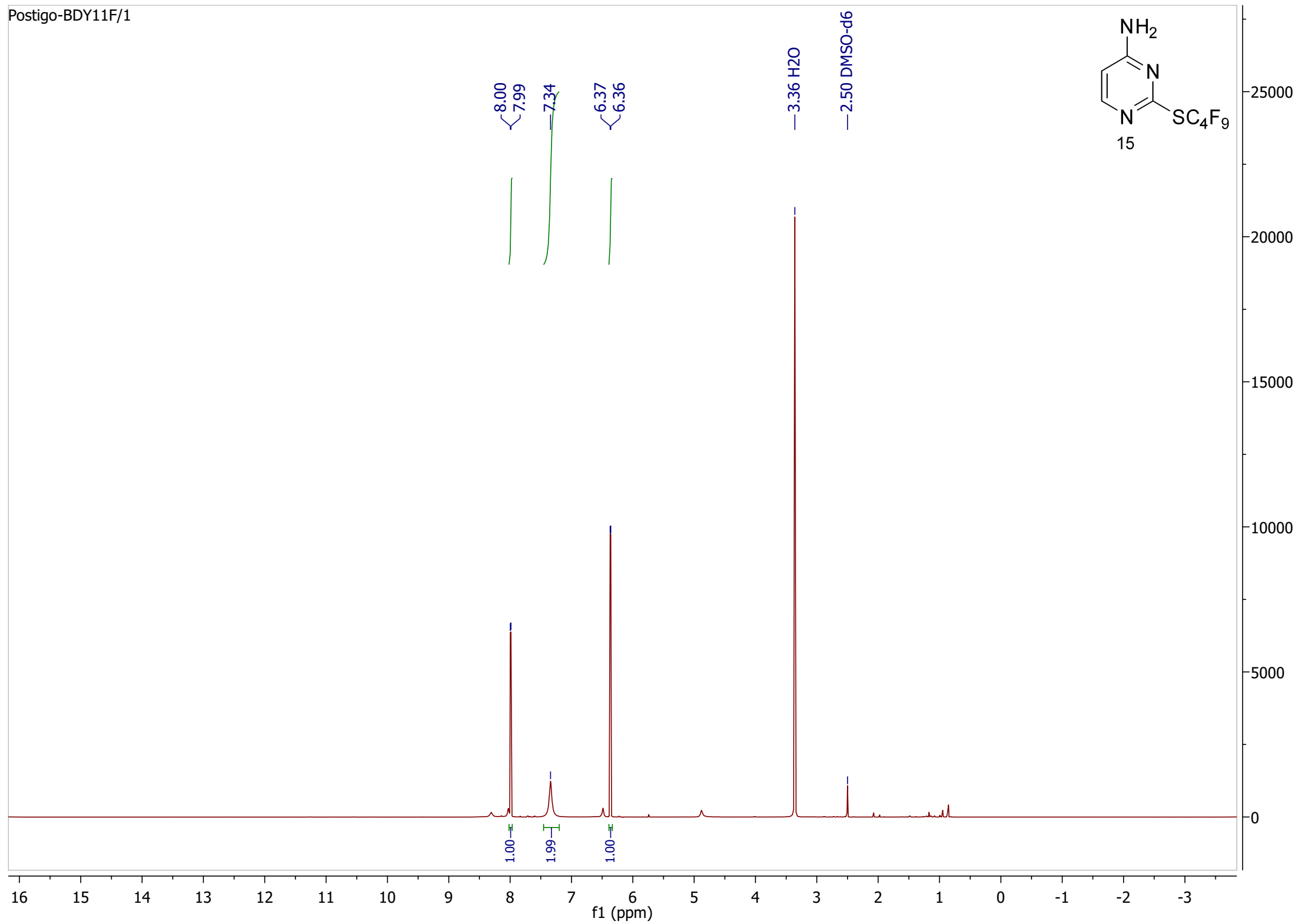


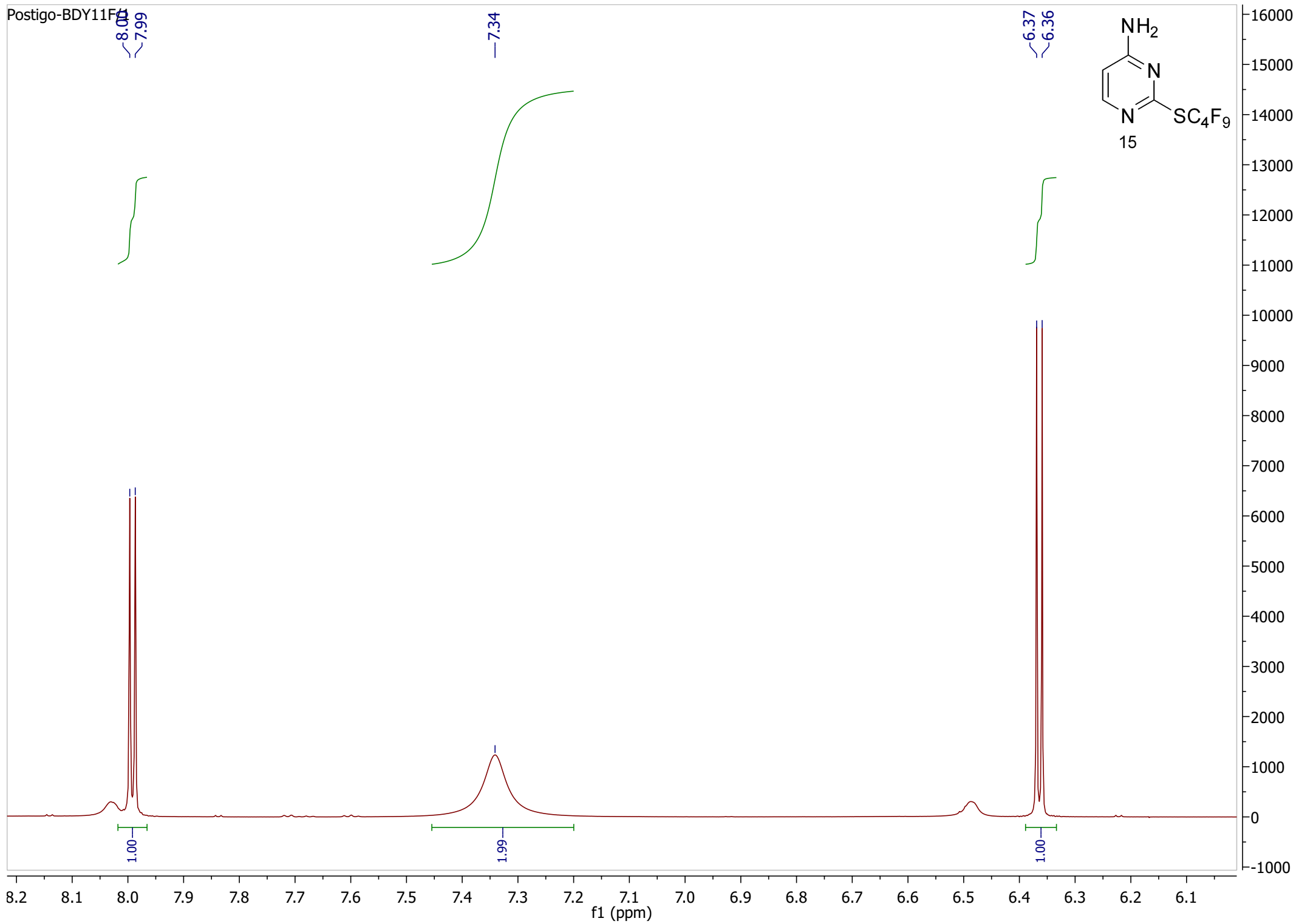
SBML-43-F1.3.fid
SBML 43-F1 #//CDCl3/400 MHz (Dr. Postigo-FFyB), posición 14.
umymfor_f19-cpd CDCl3 {C\Bruker\TopSpin3.2} nmrsu 14

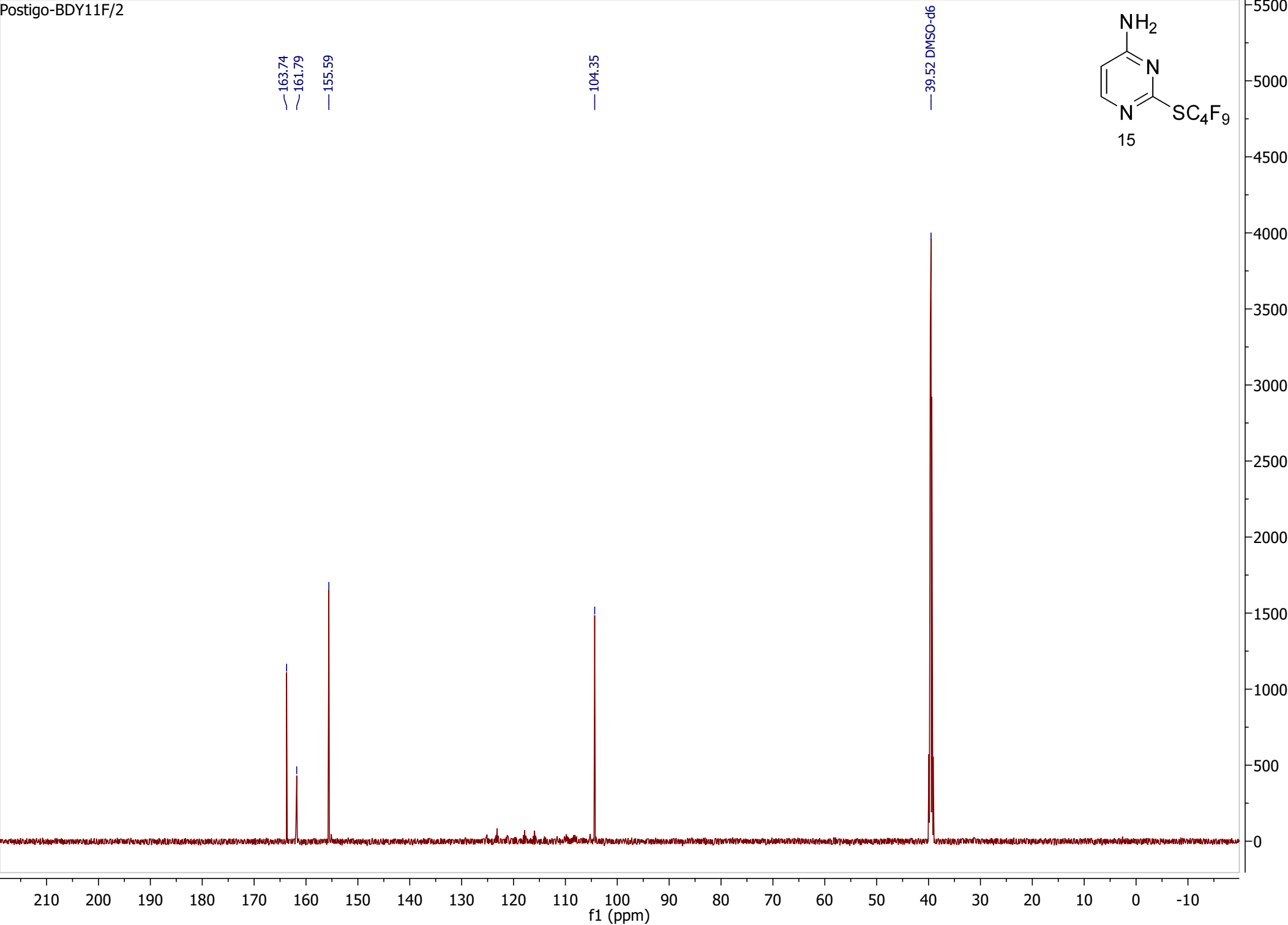


86.08
86.11
86.15
86.48
86.51
86.54









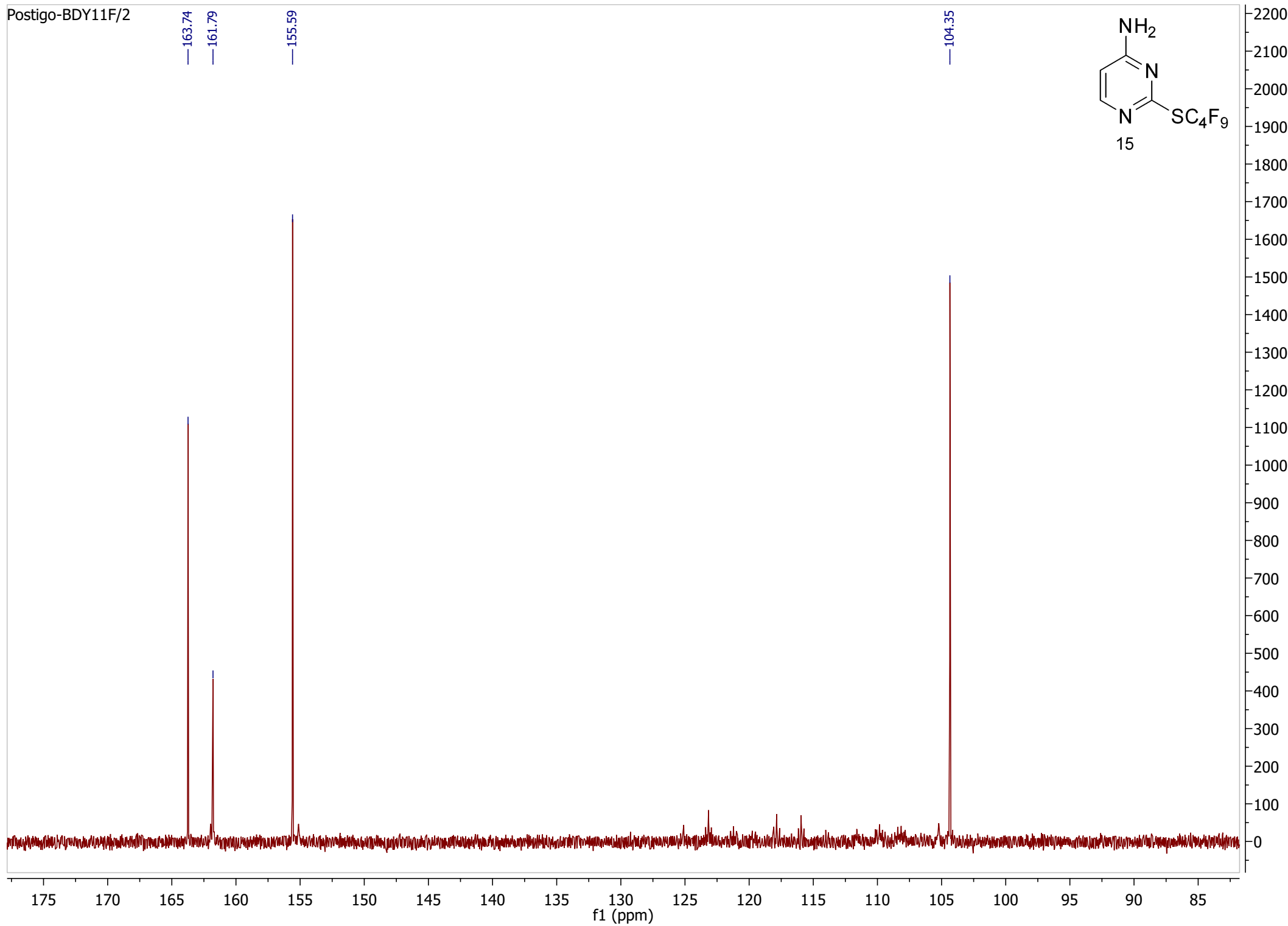
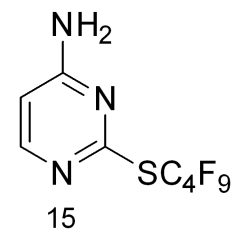
Postigo-BDY11F/2

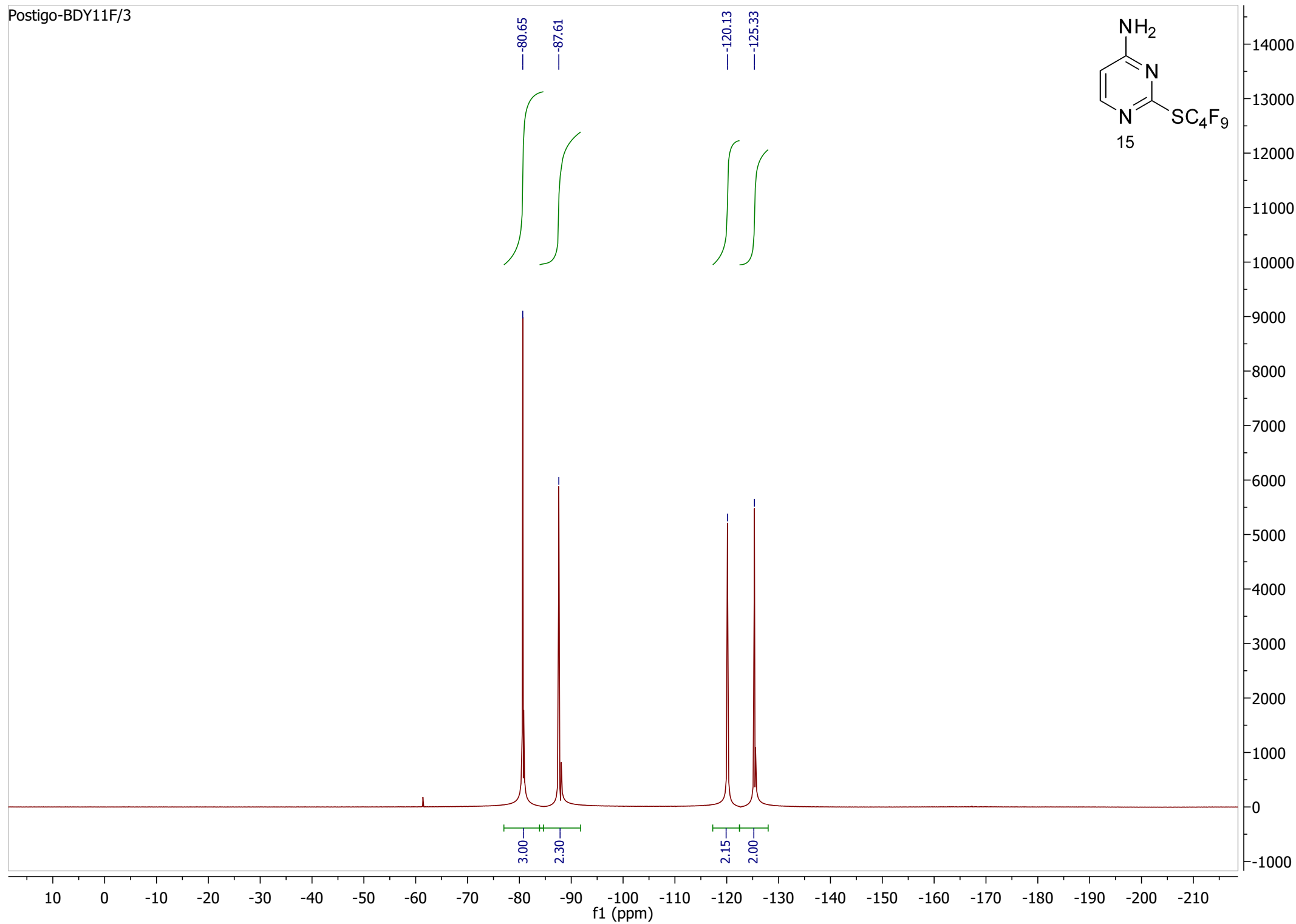
— 163.74

— 161.79

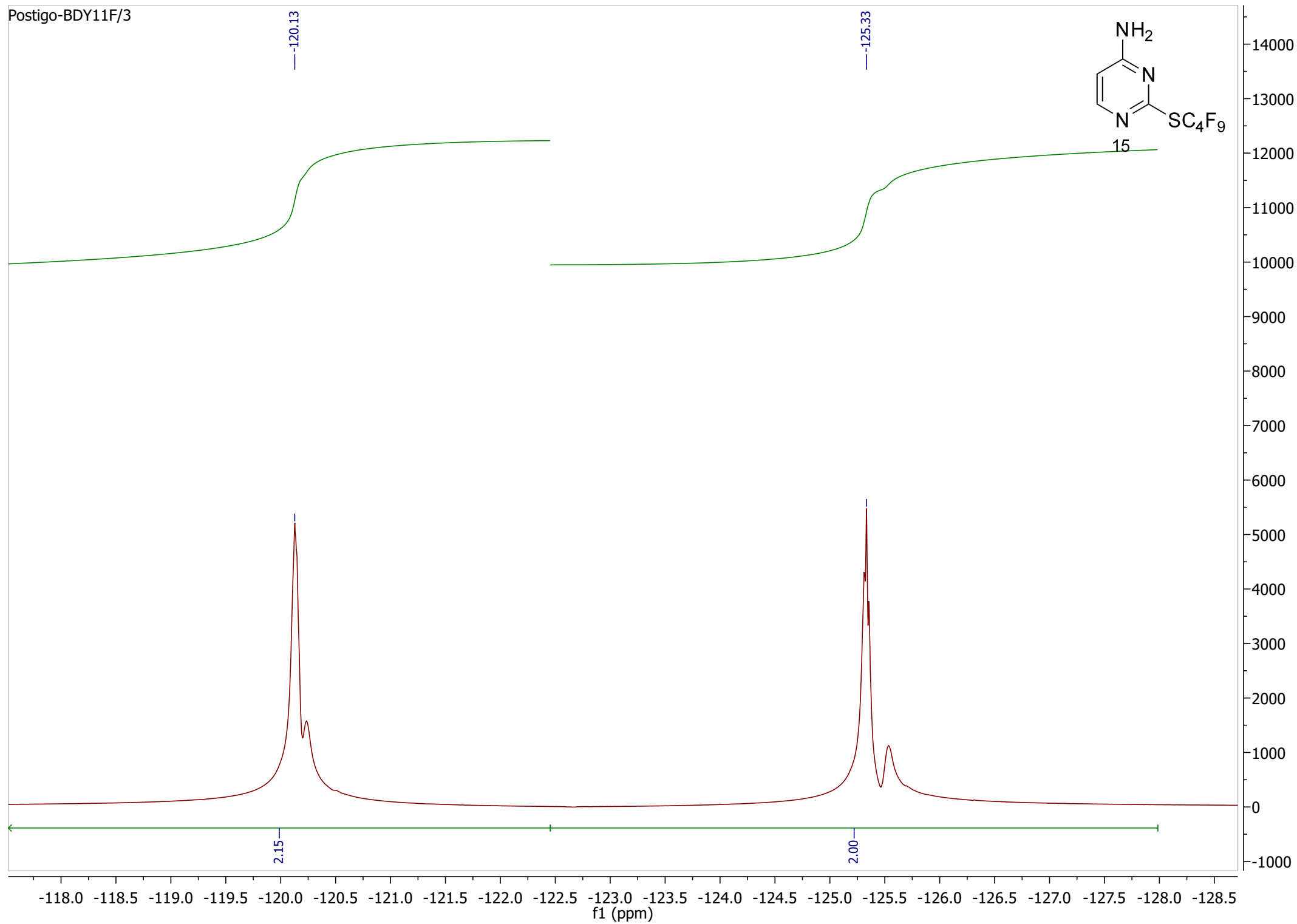
— 155.59

— 104.35





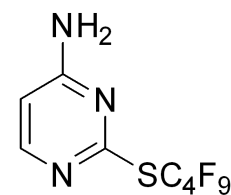
Postigo-BDY11F/3



Postigo-BDY11F/3

—80.65

—87.61



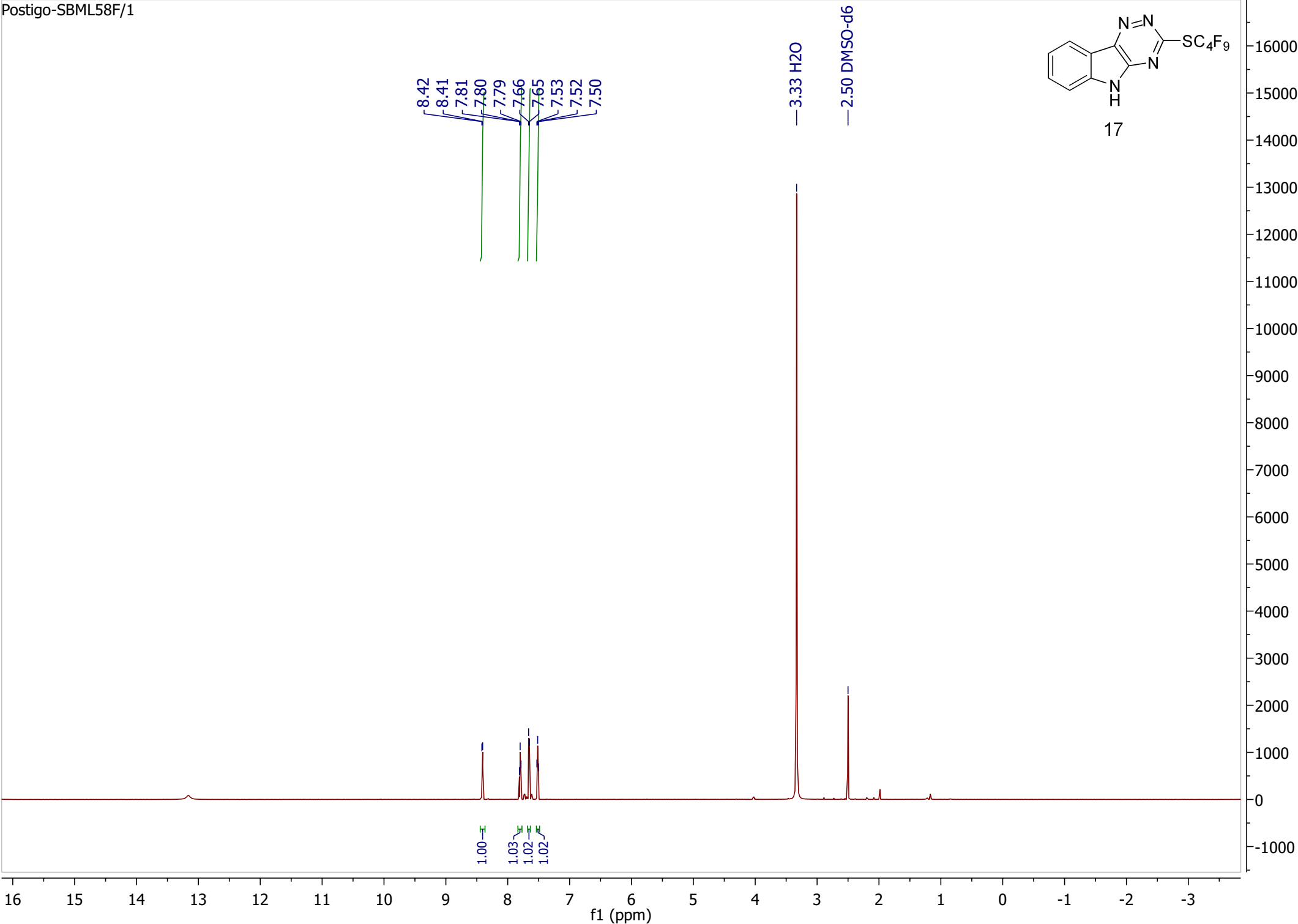
15

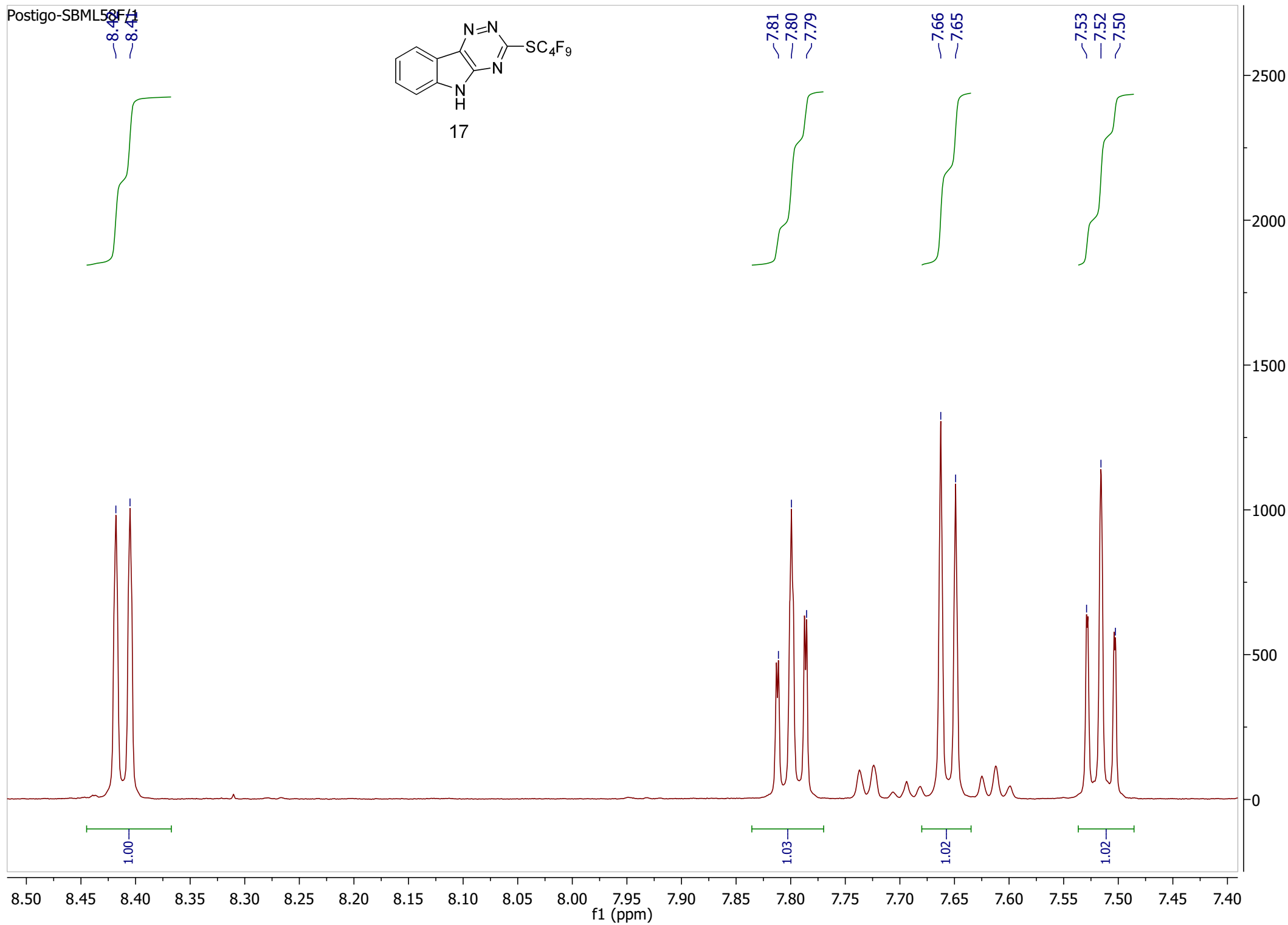
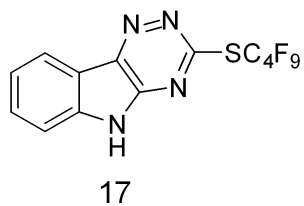
9.5 -80.0 -80.5 -81.0 -81.5 -82.0 -82.5 -83.0 -83.5 -84.0 -84.5 -85.0 -85.5 -86.0 -86.5 -87.0 -87.5 -88.0 -88.5 -89.0 -89.5

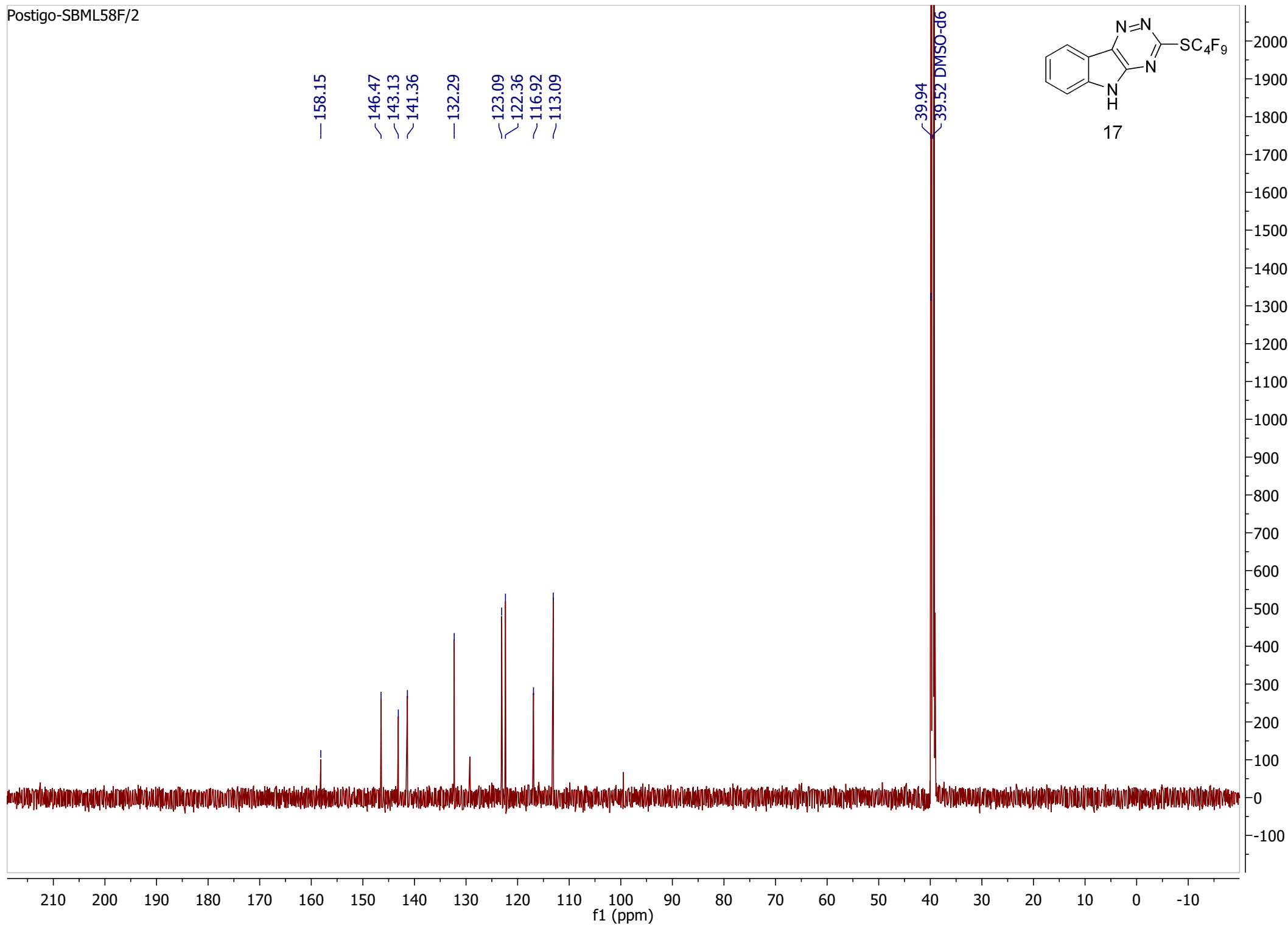
f1 (ppm)

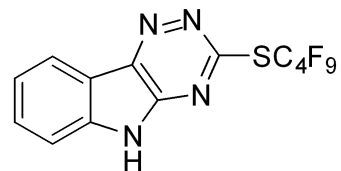
3.00

2.30

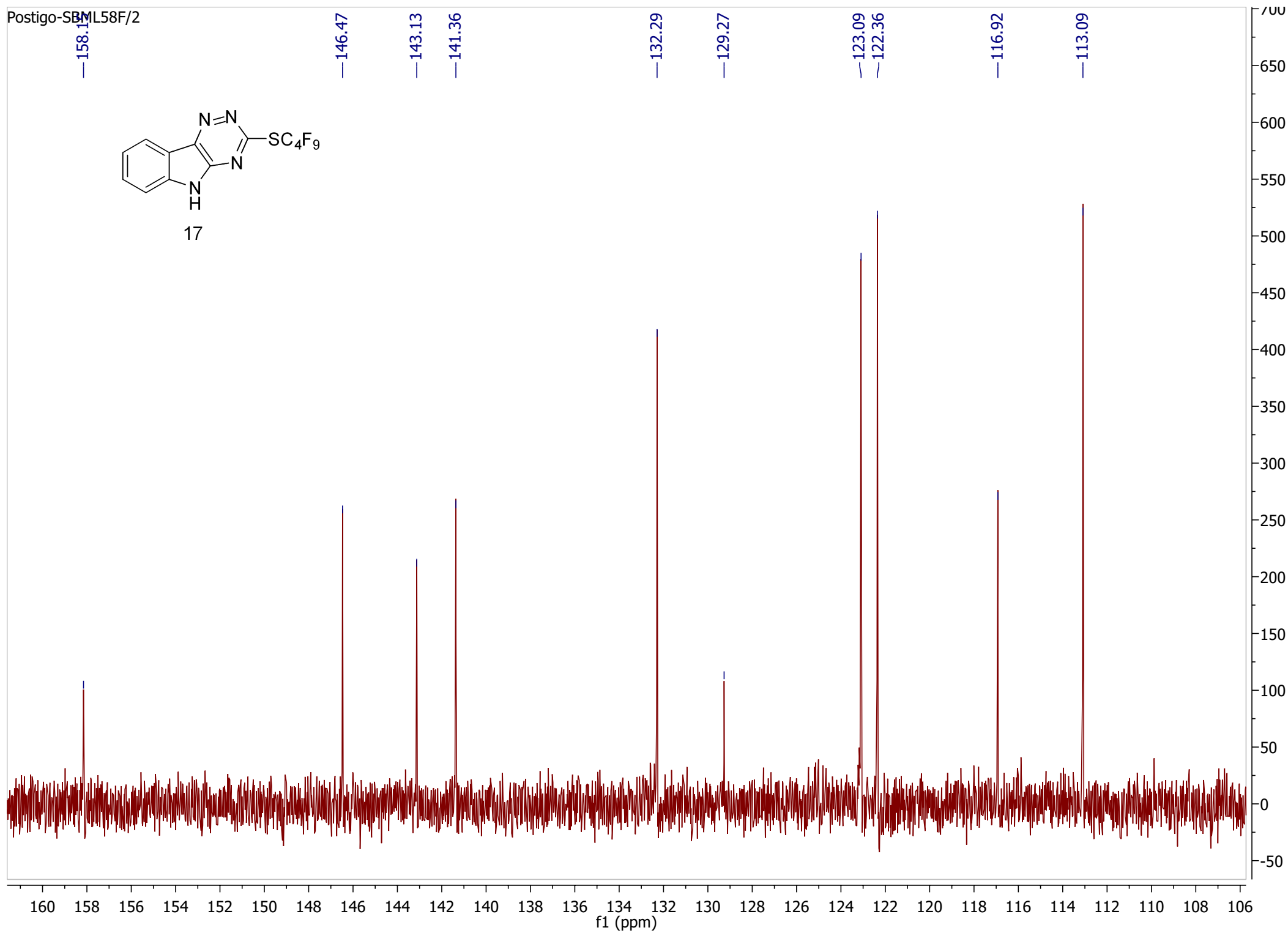


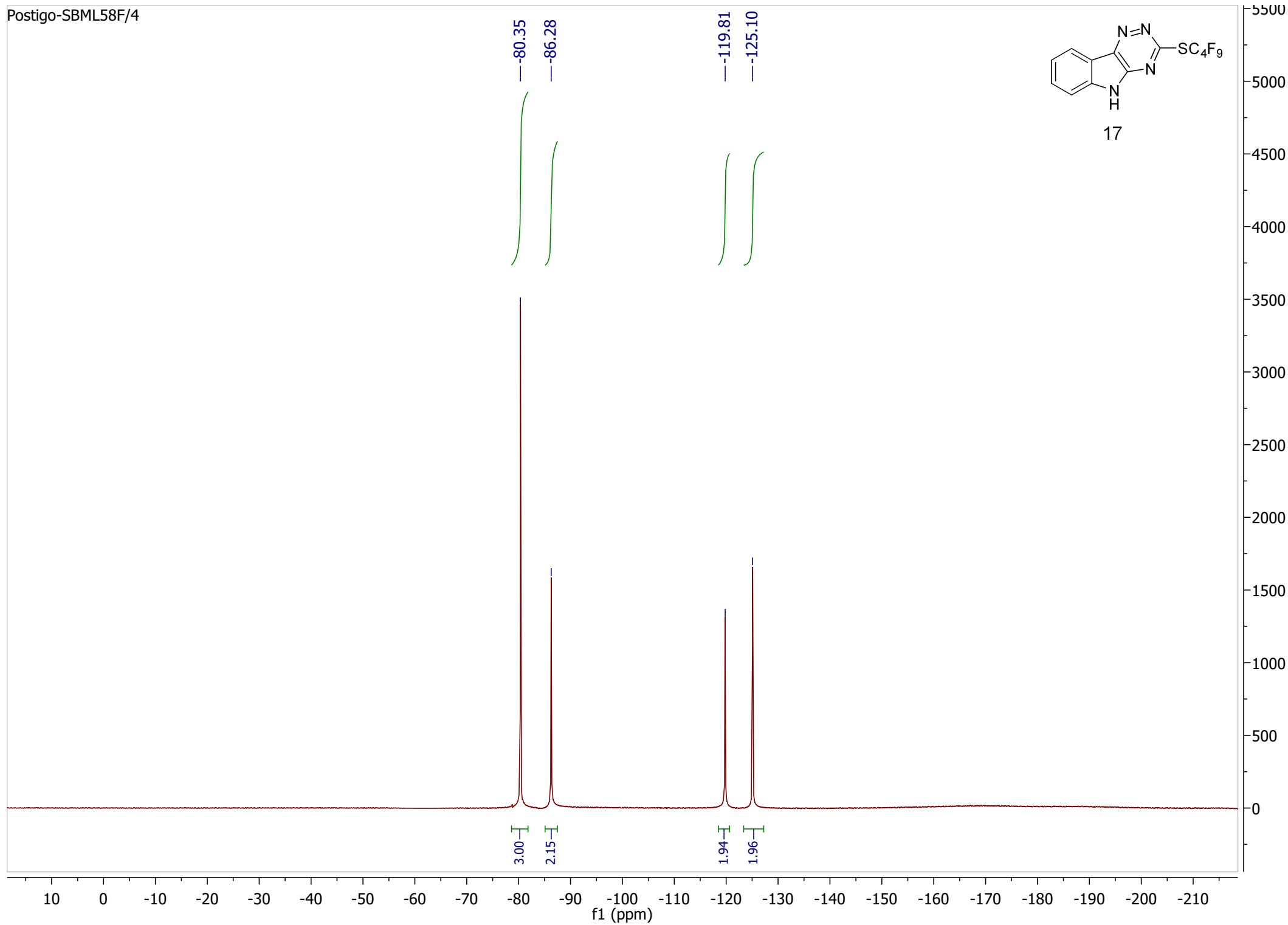


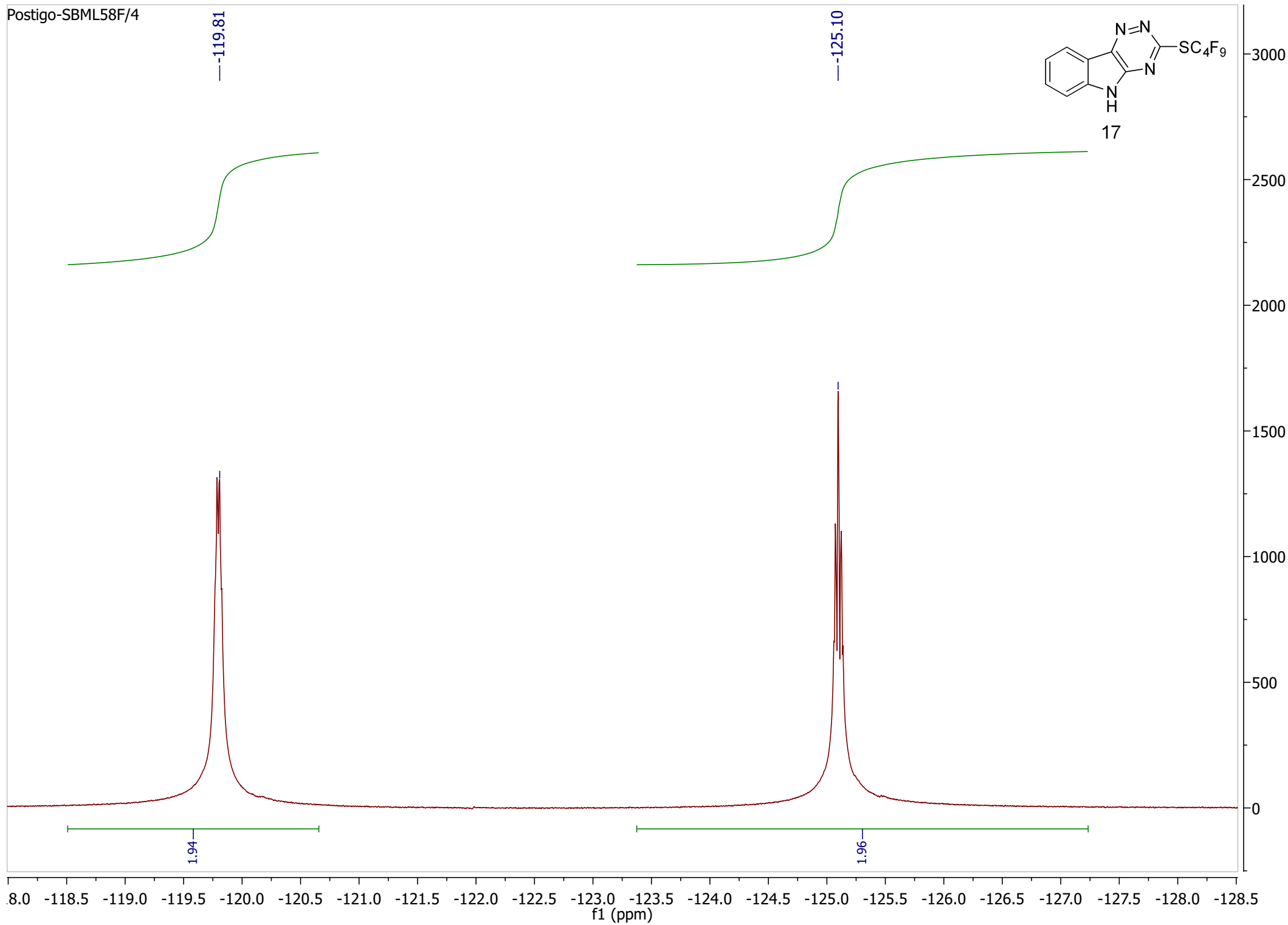


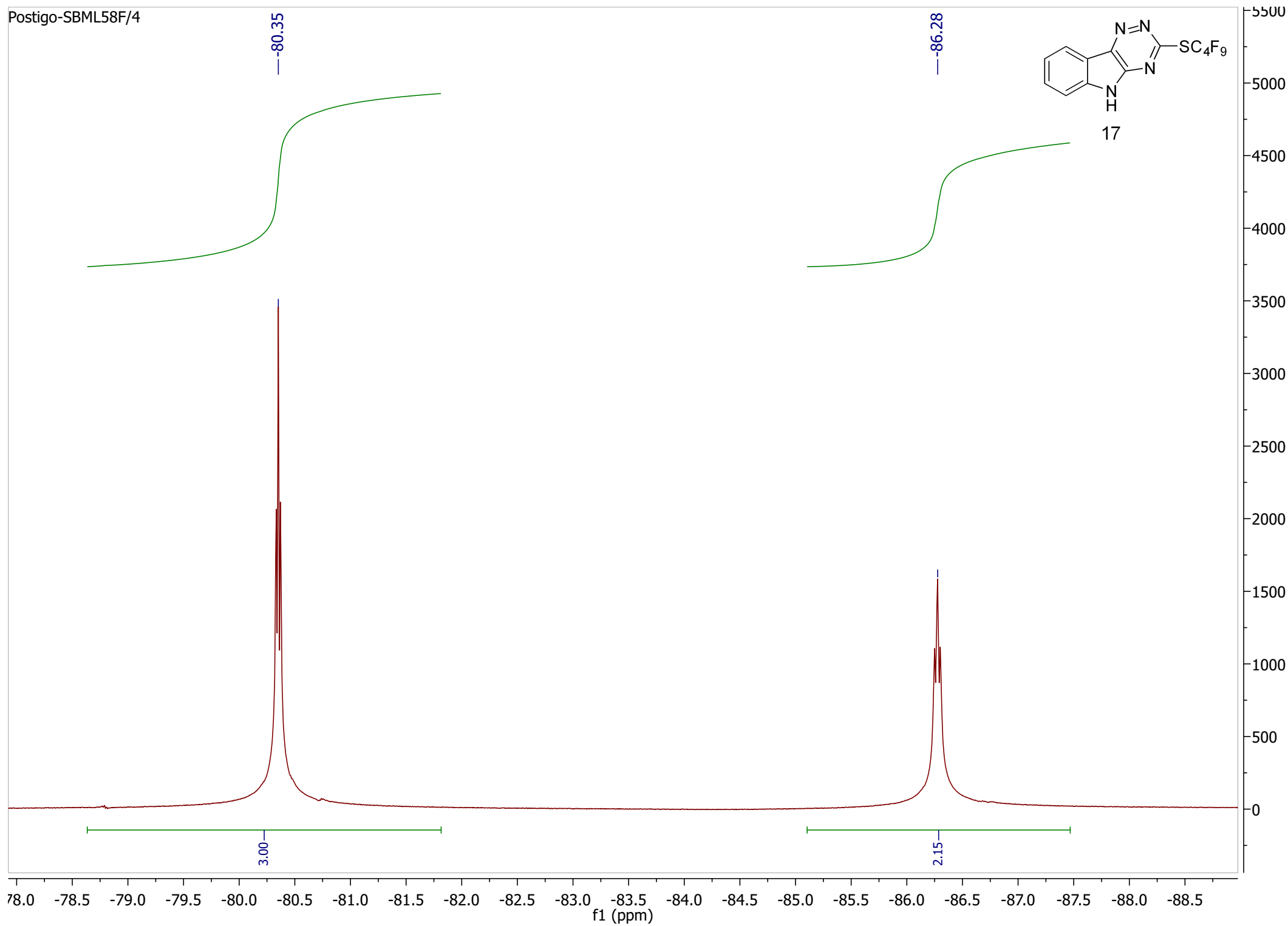


17



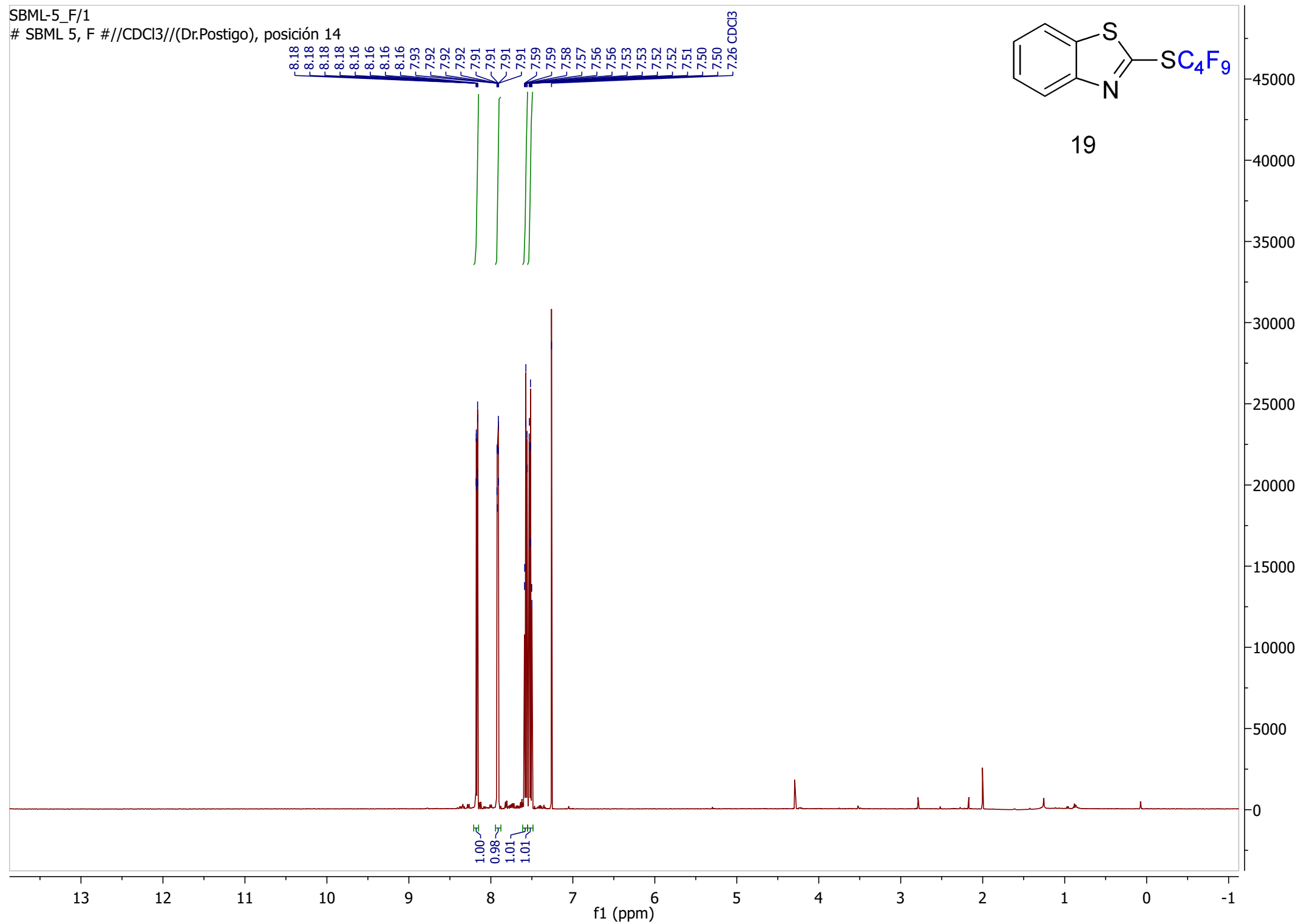




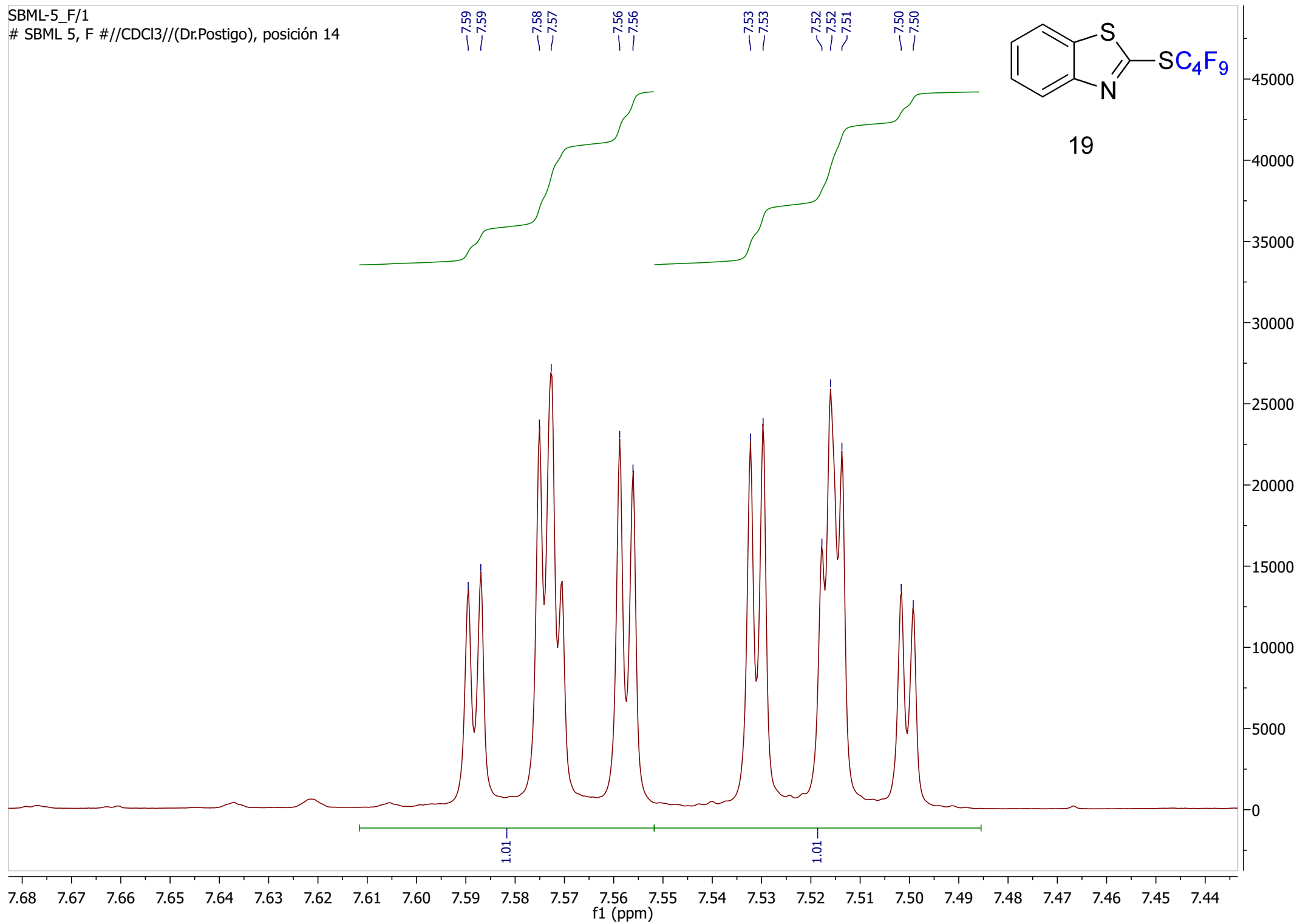


SBML-5_F/1

SBML 5, F #//CDCl3//(Dr.Postigo), posición 14

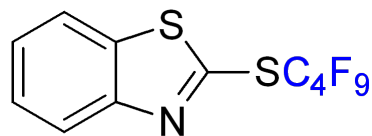


SBML-5_F/1
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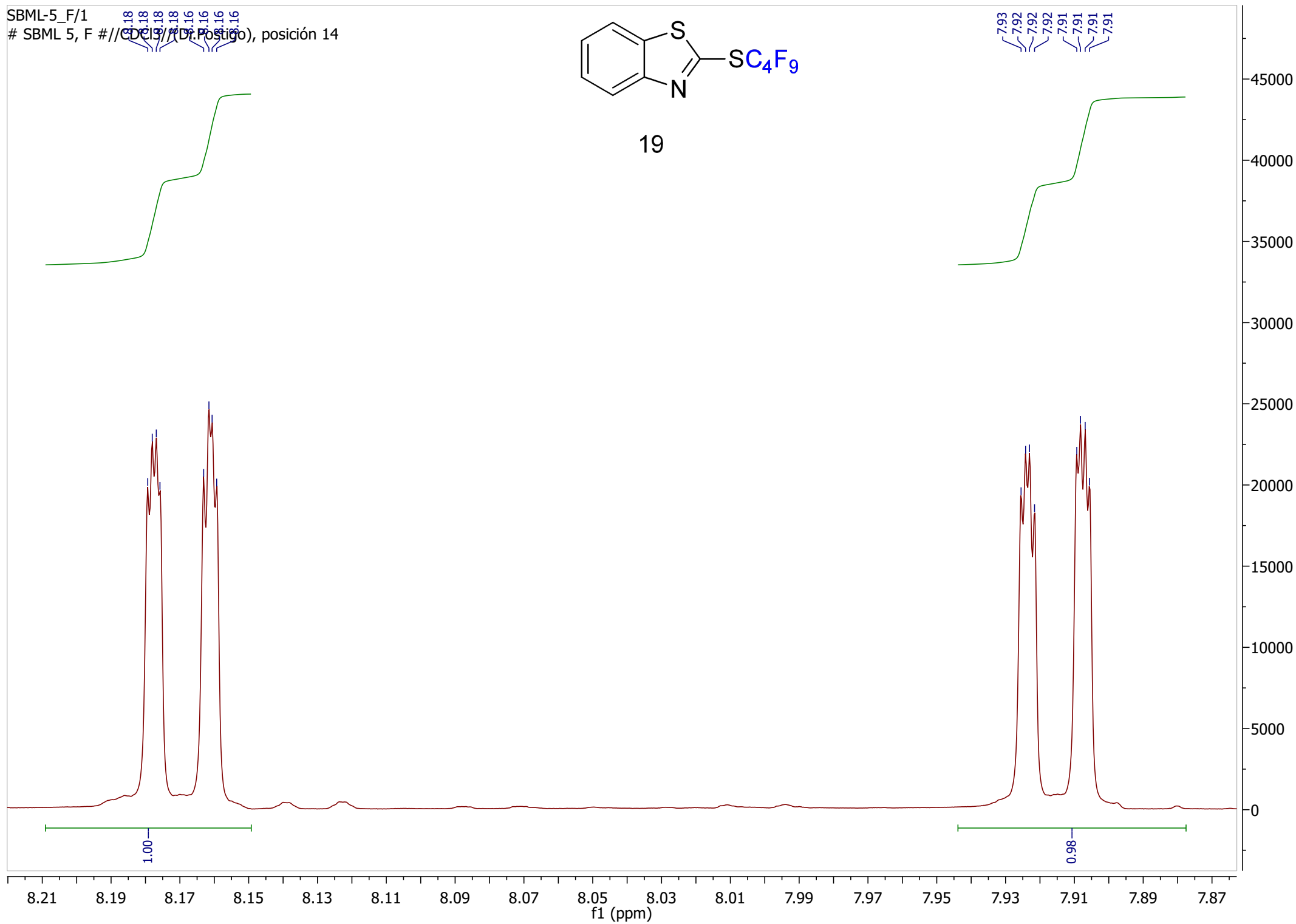


SBML-5_F/1

SBML 5, F #//CDC13// (Dr. Postigo), posición 14

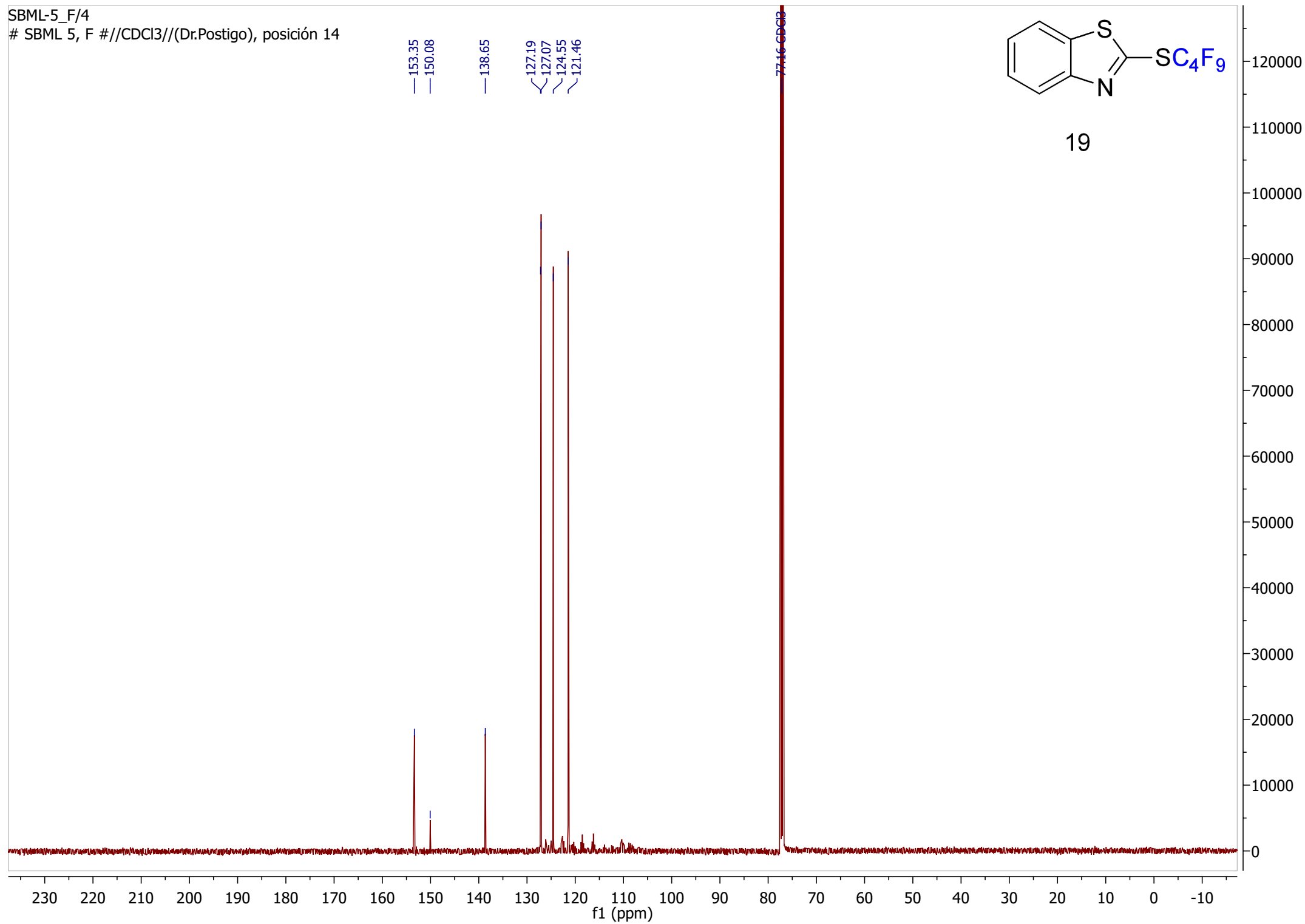


19

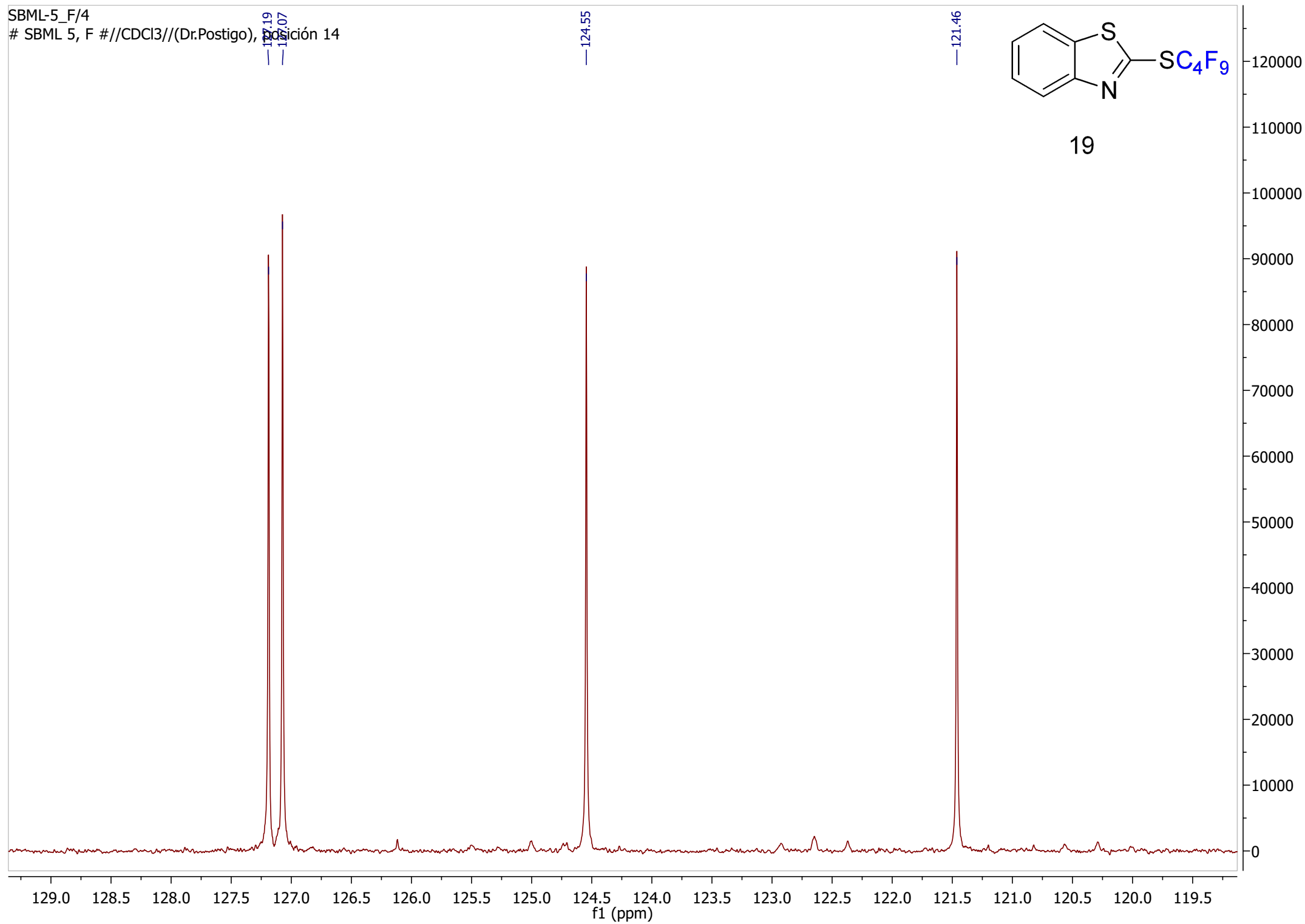


SBML-5_F/4

SBML 5, F #//CDCl3//(Dr.Postigo), posición 14

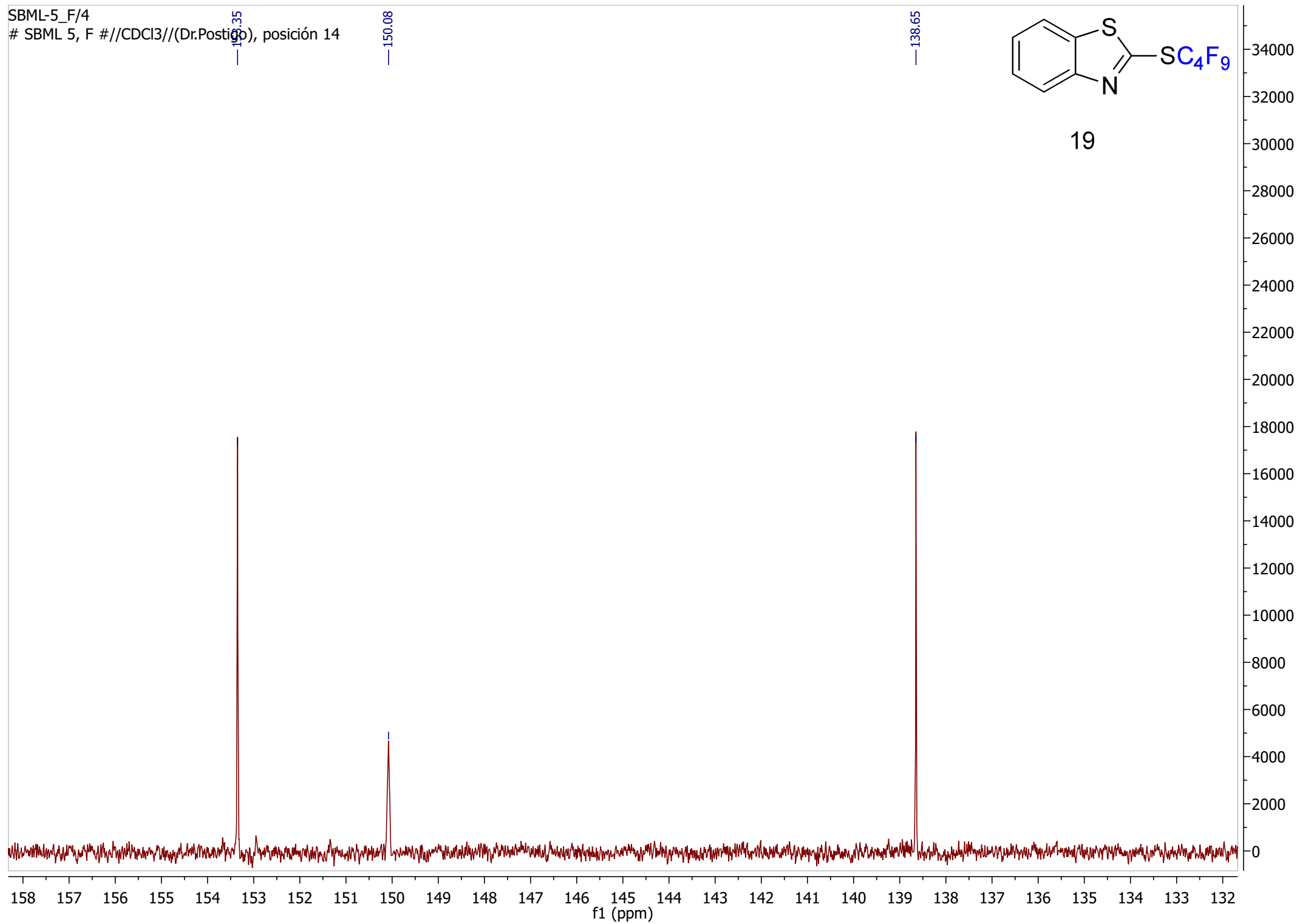


SBML-5_F/4
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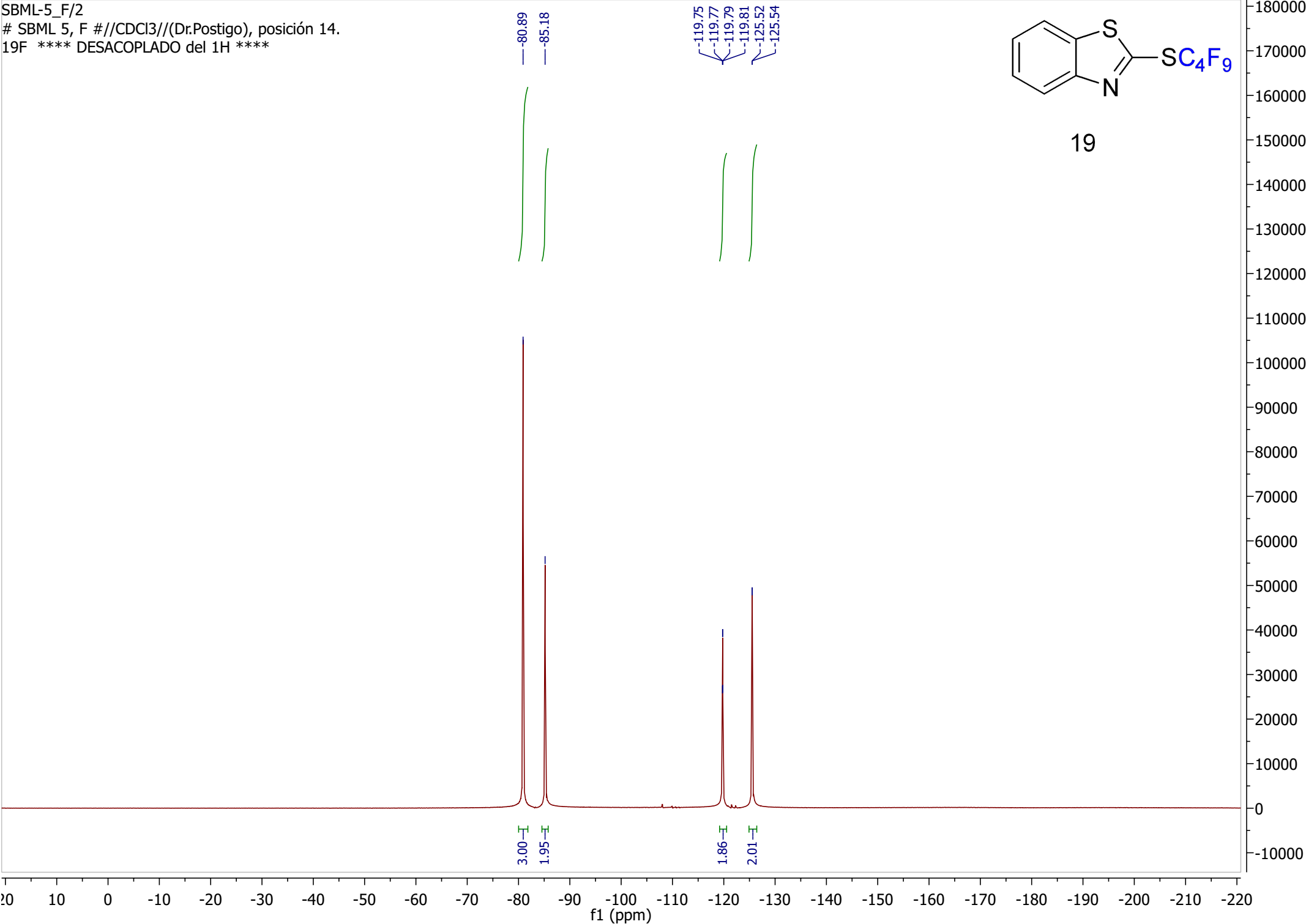


SBML-5_F/4

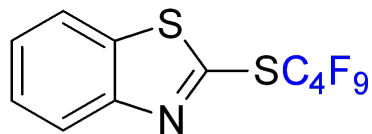
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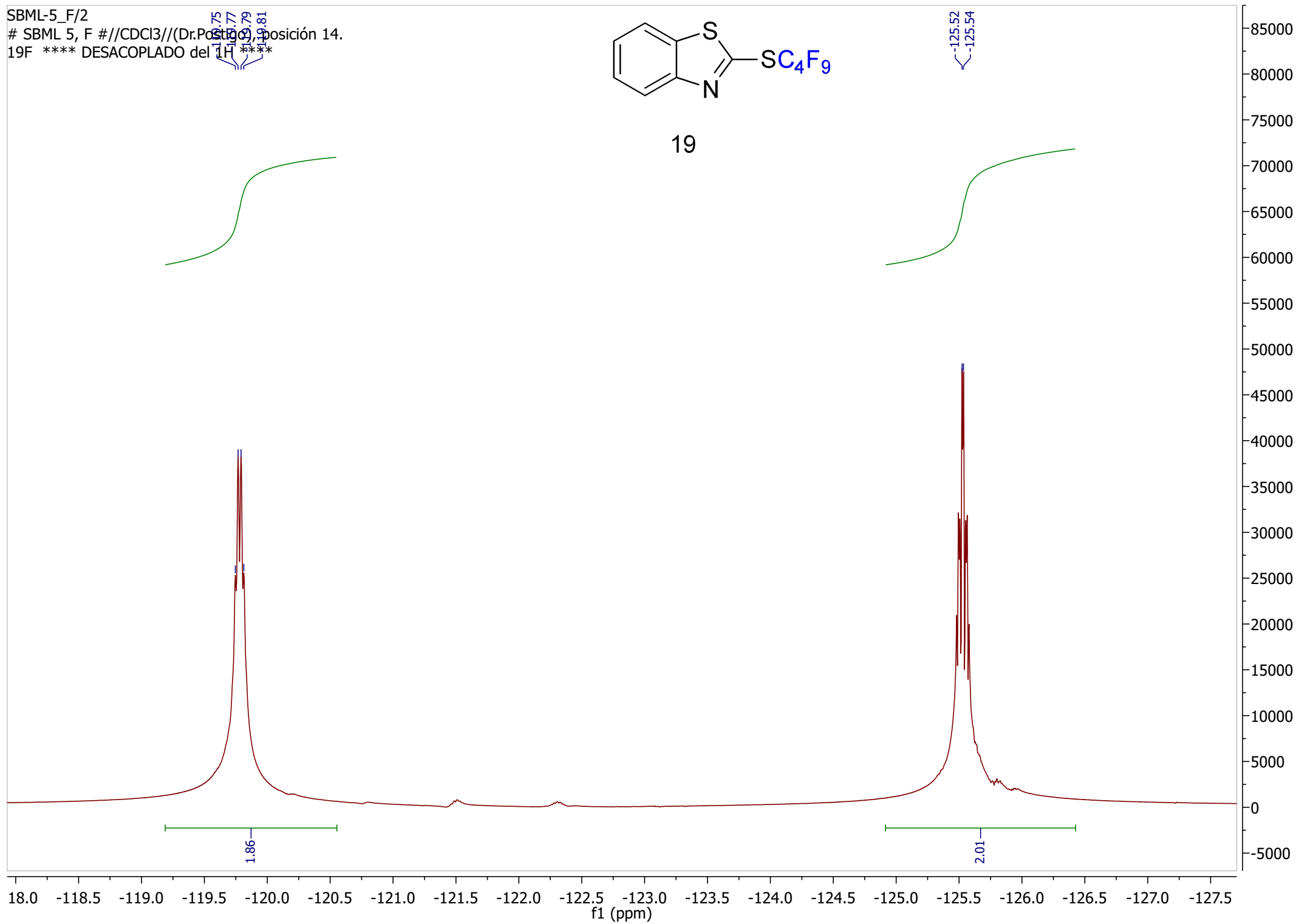
SBML-5_F/2
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19F **** DESACOPLADO del 1H ****



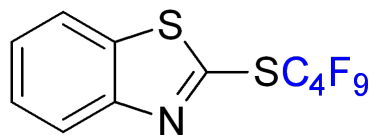
SBML-5_F/2
SBML 5, F #//CDCl3// (Dr. Postigo, posición 14.
19F **** DESACOPLADO del 1H ****



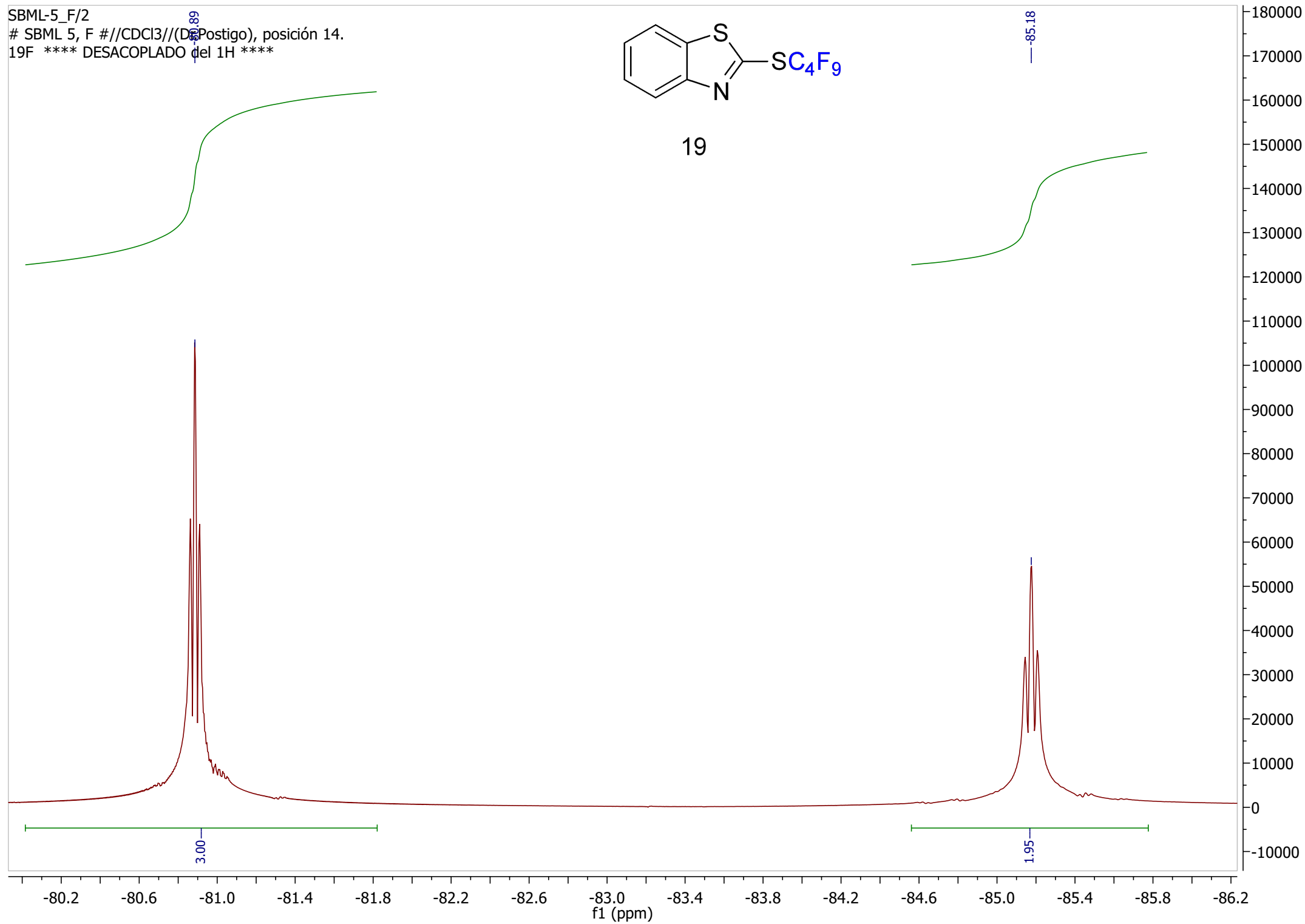
19

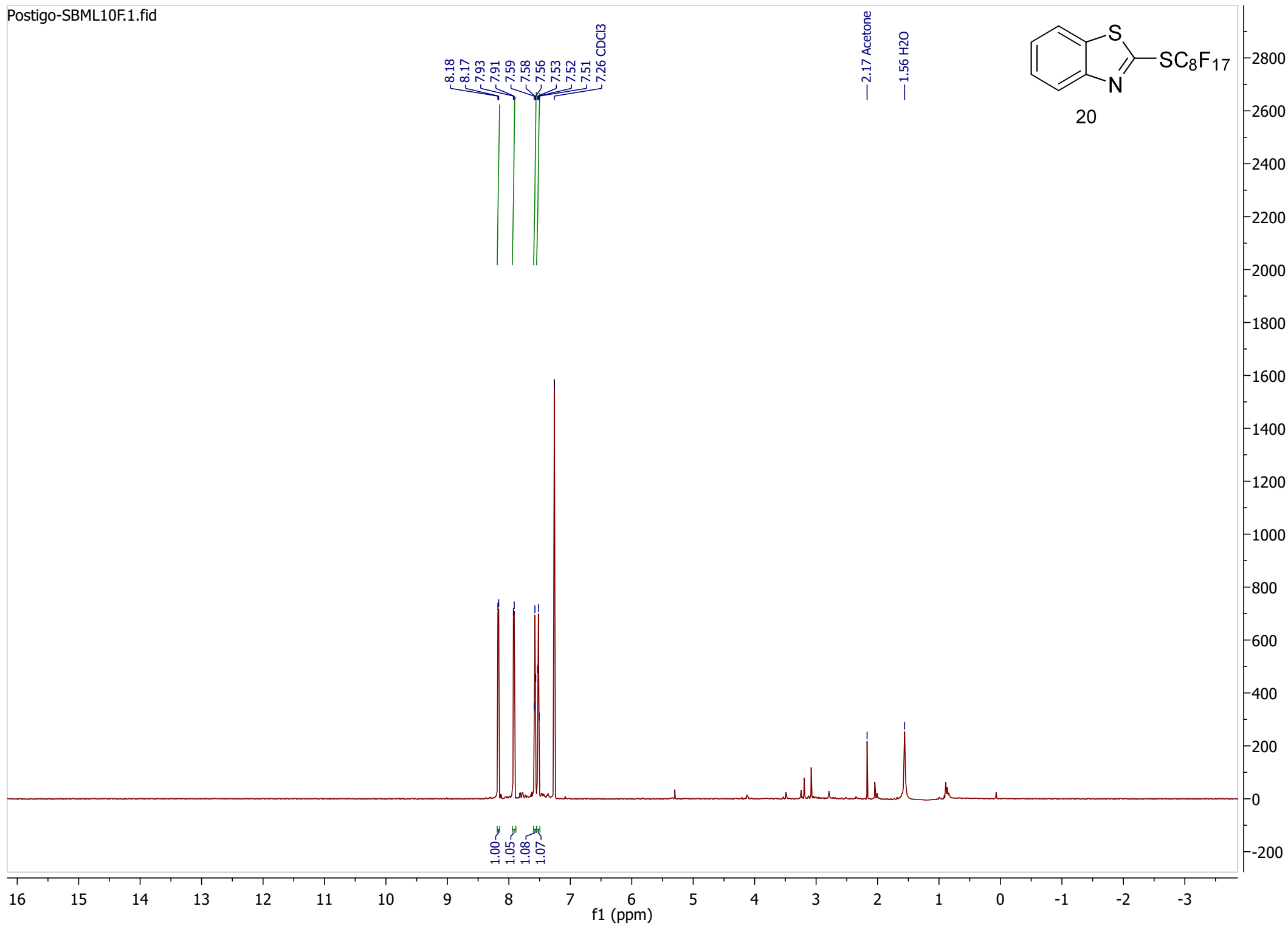


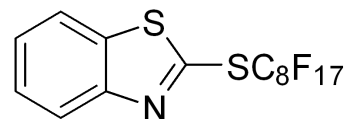
SBML-5_F/2
SBML 5, F #//CDCl3// (De Postigo), posición 14.
19F **** DESACOPLADO del 1H ****



19





8.18
8.177.93
7.91

20

7.59
7.58
7.567.53
7.52
7.518.30 8.25 8.20 8.15 8.10 8.05 8.00 7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45
f1 (ppm)

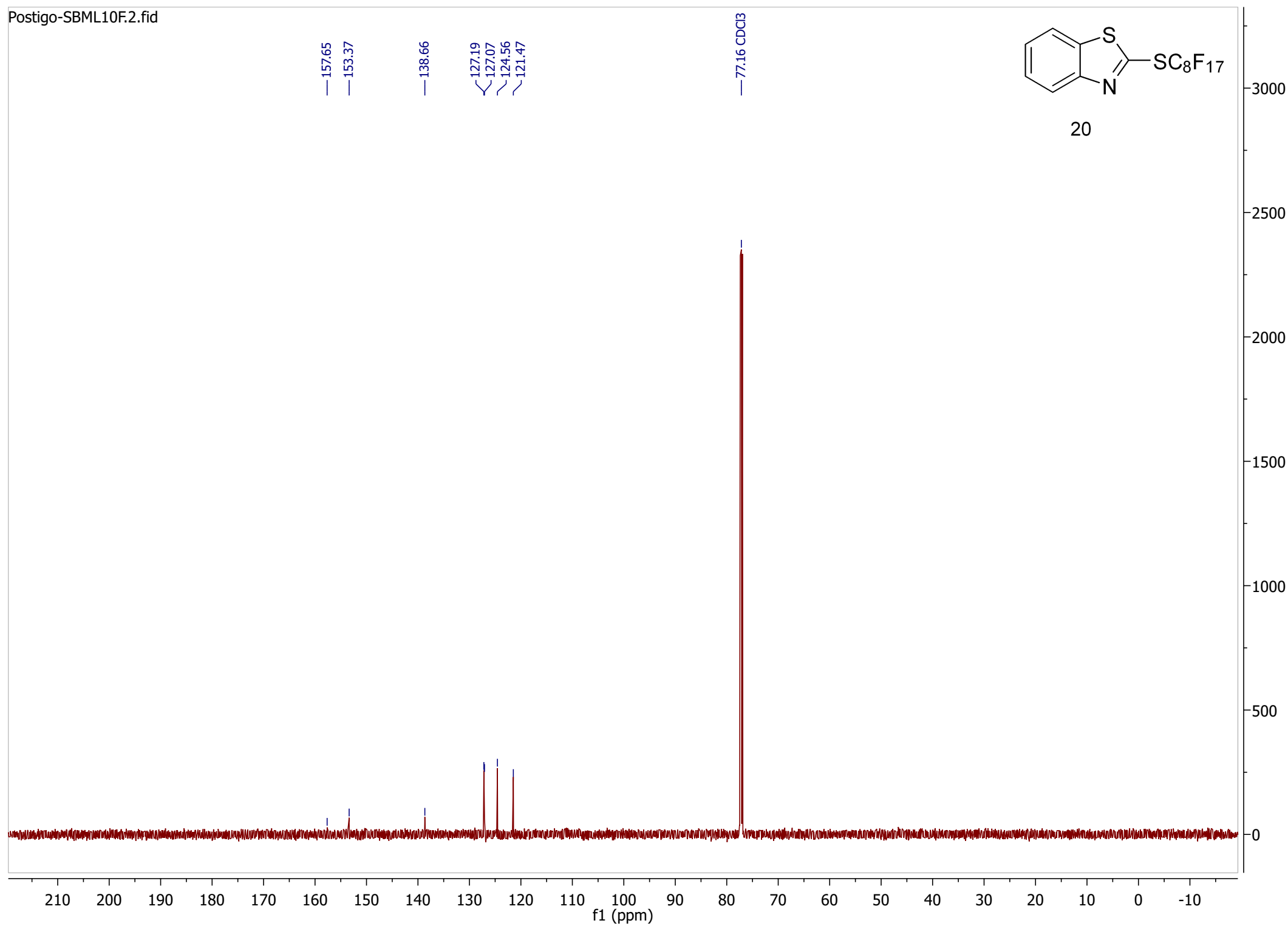
1.00

1.05

1.08

1.07

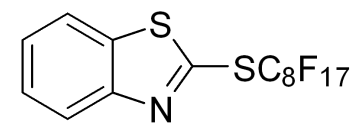
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100



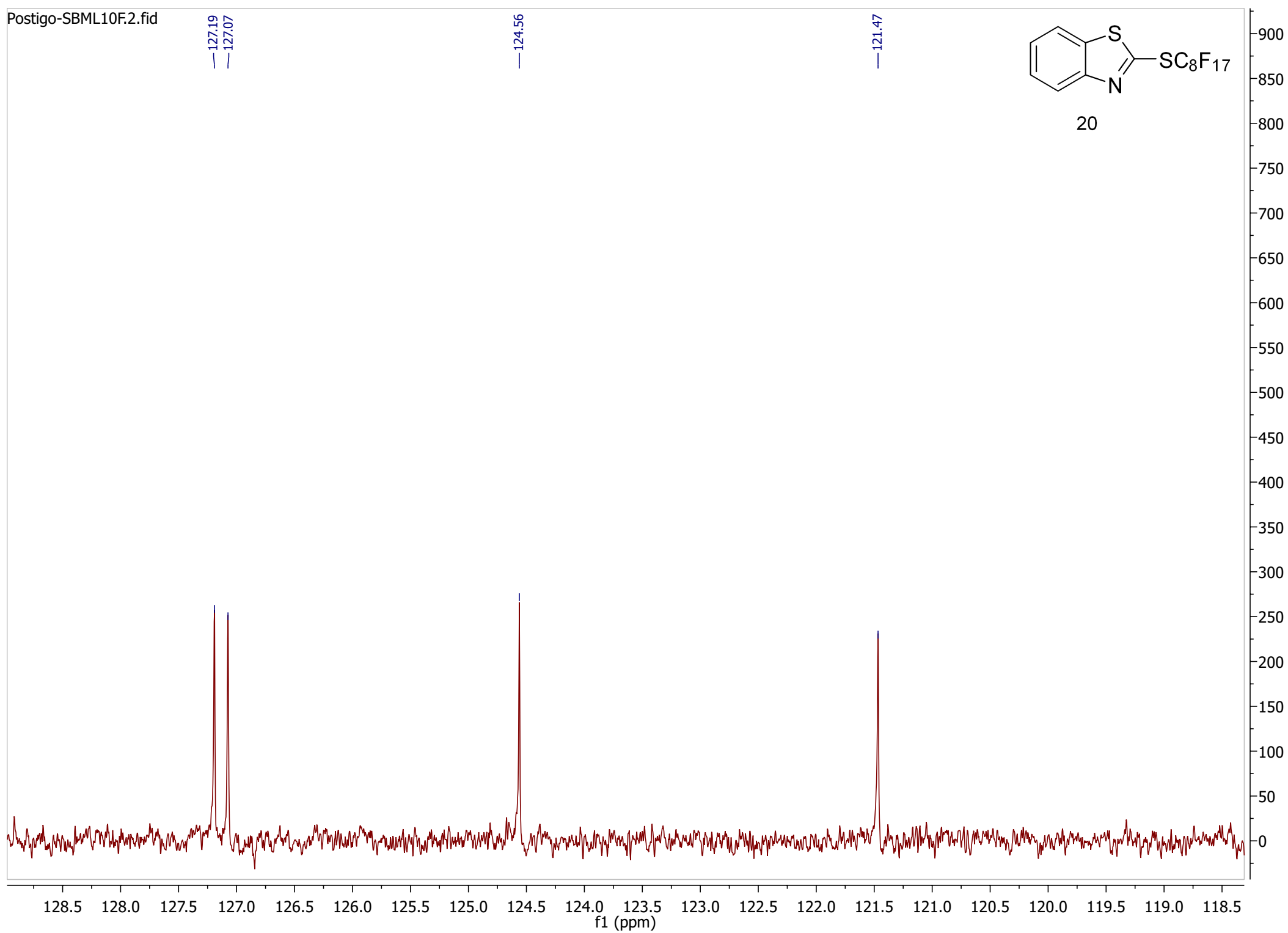
—127.19
—127.07

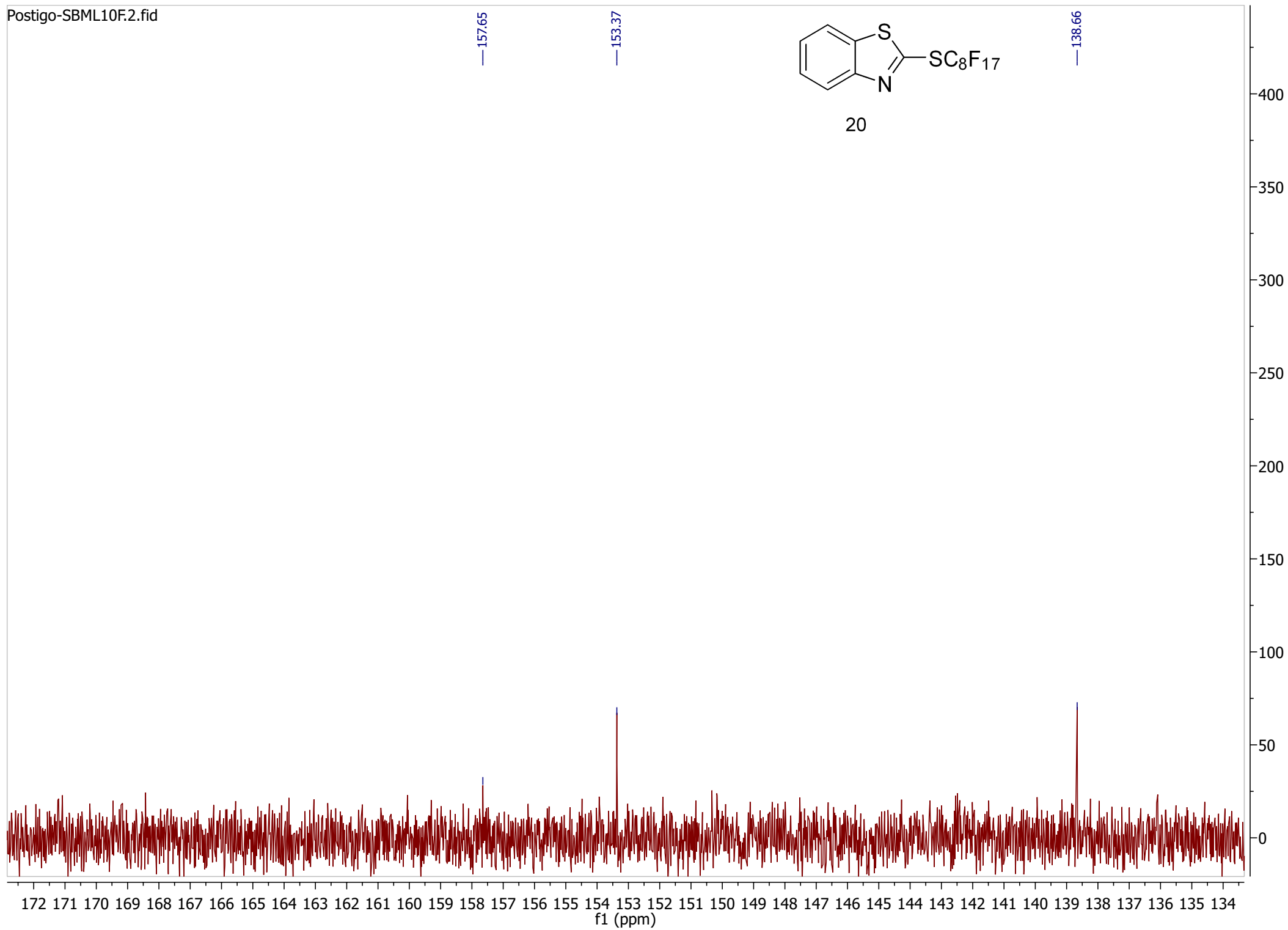
—124.56

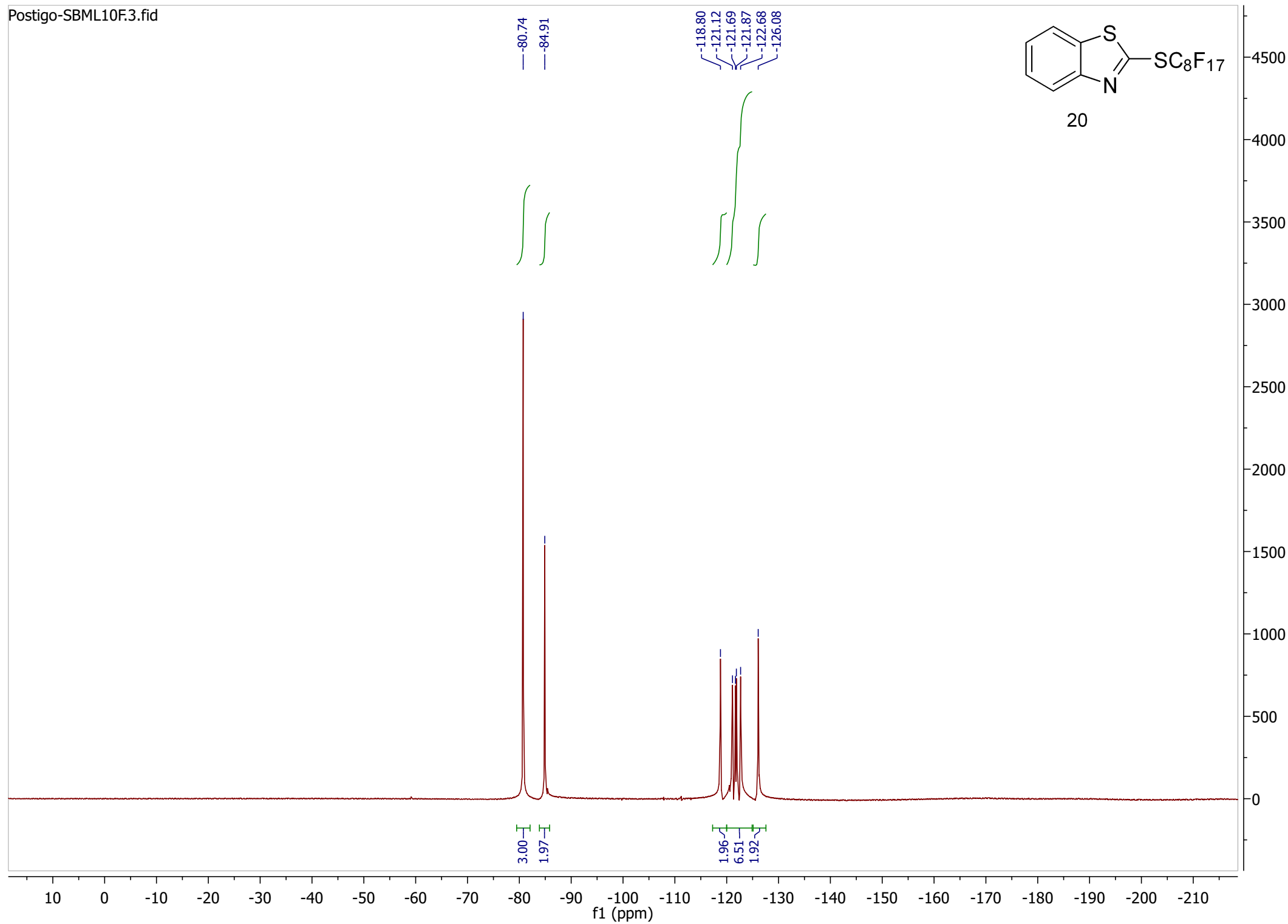
—121.47

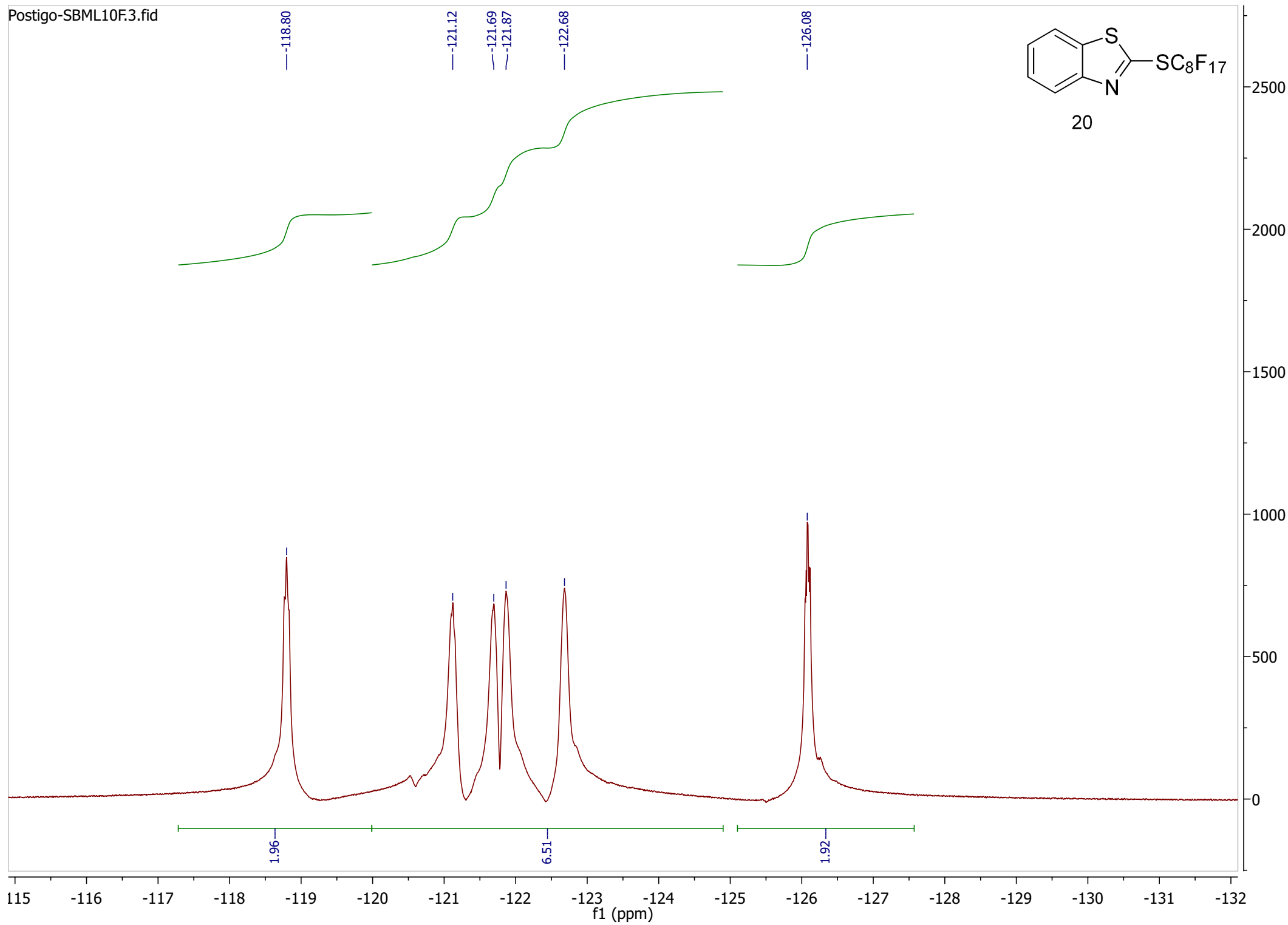


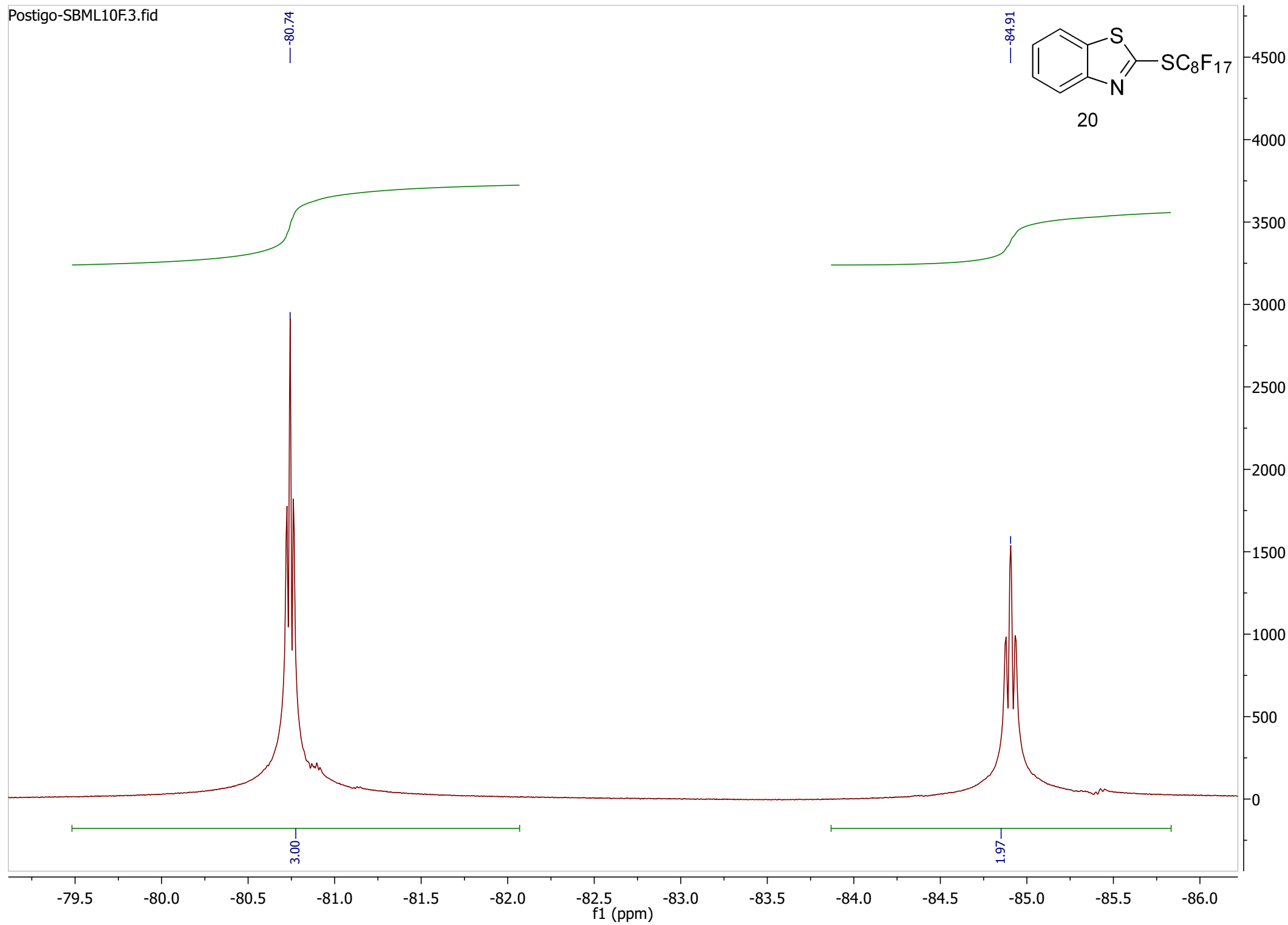
20

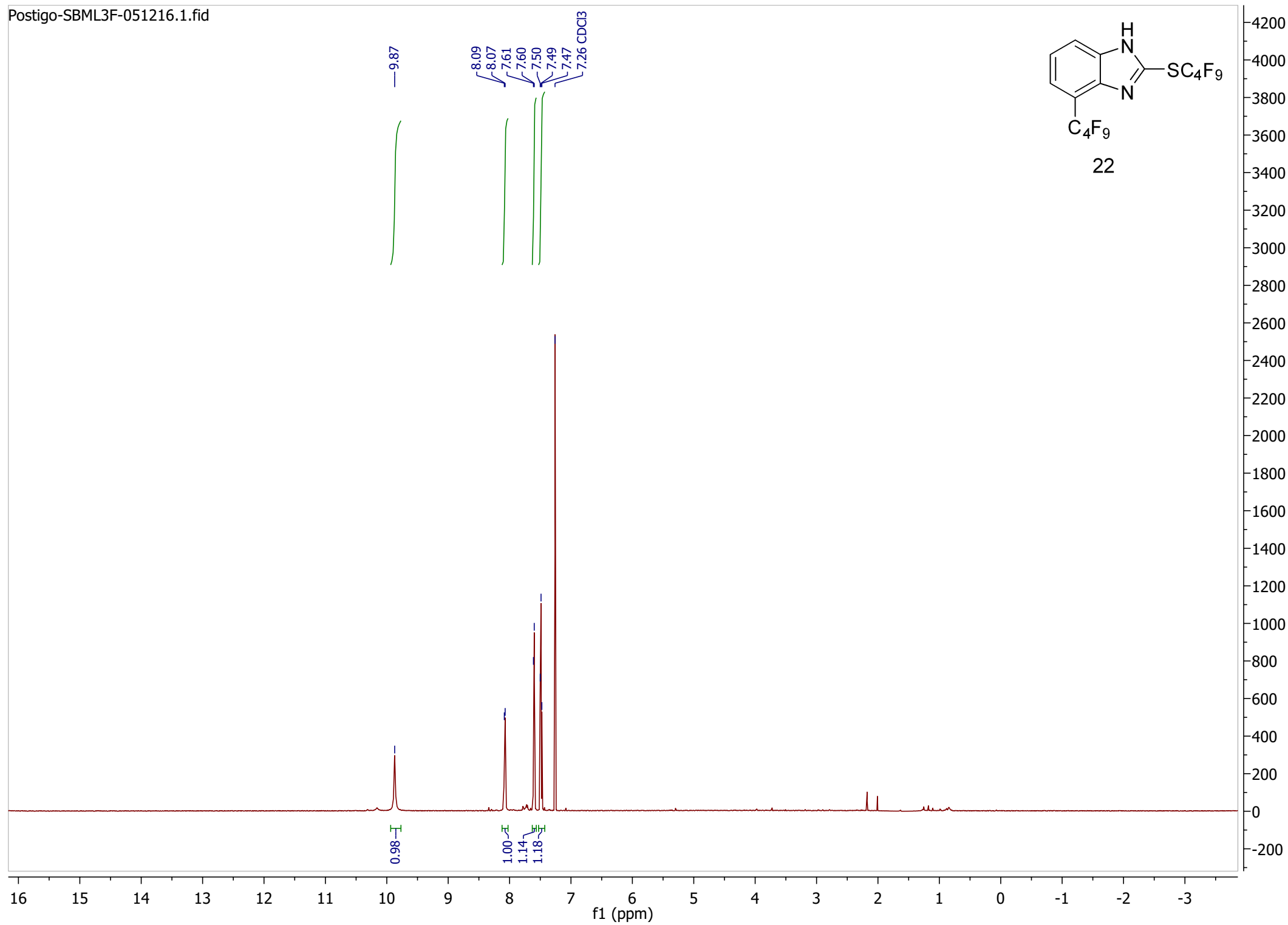


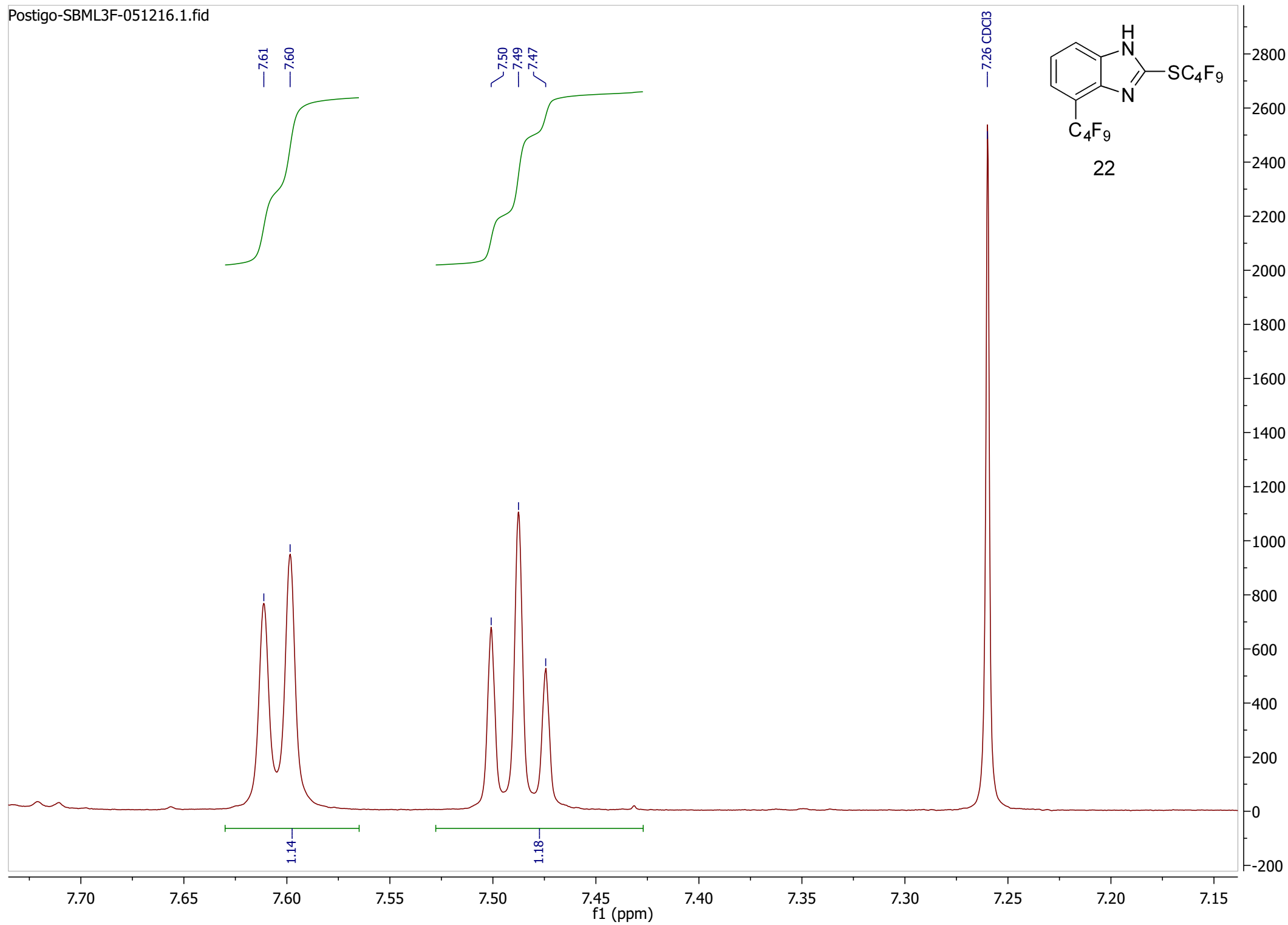


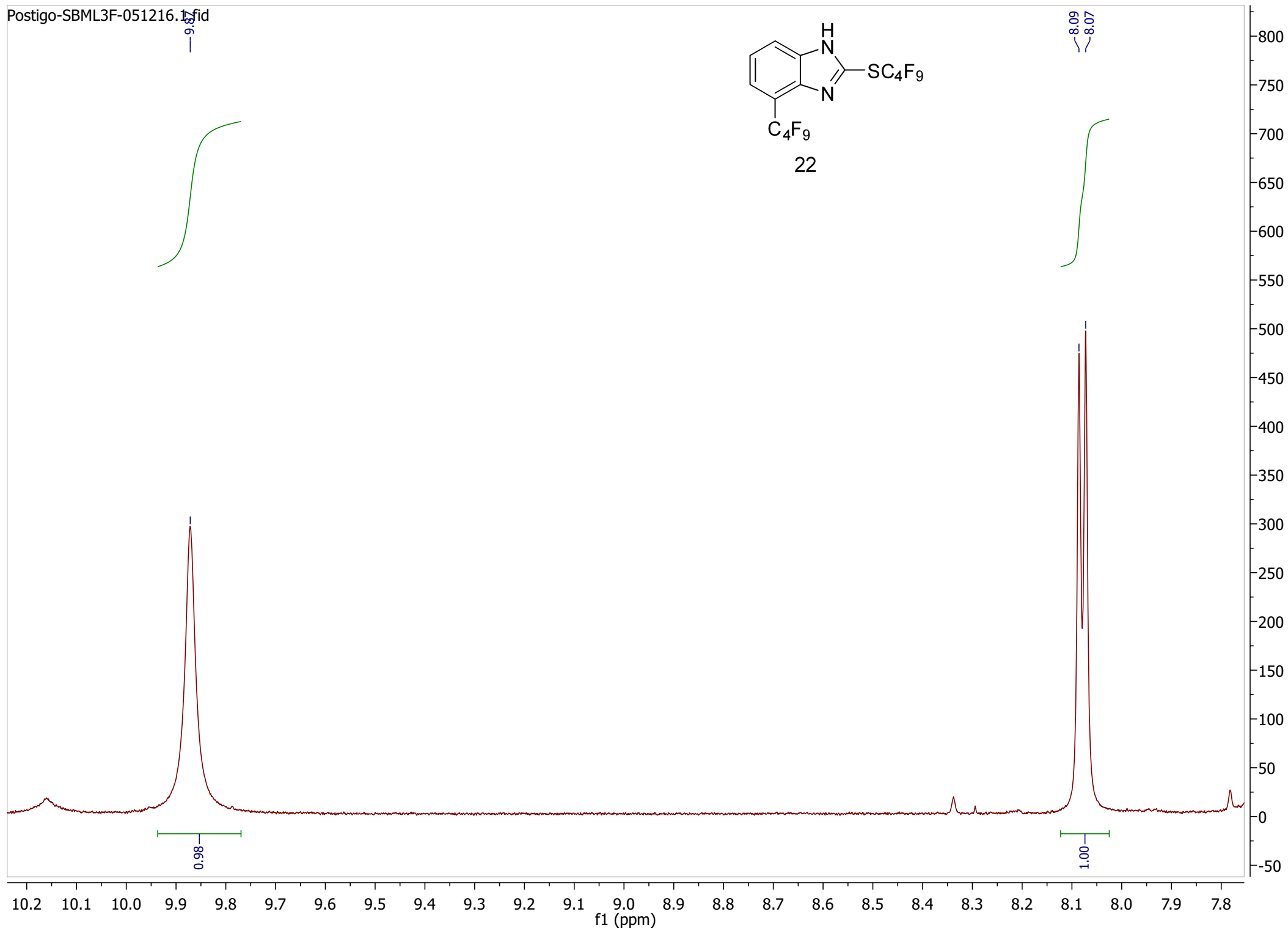


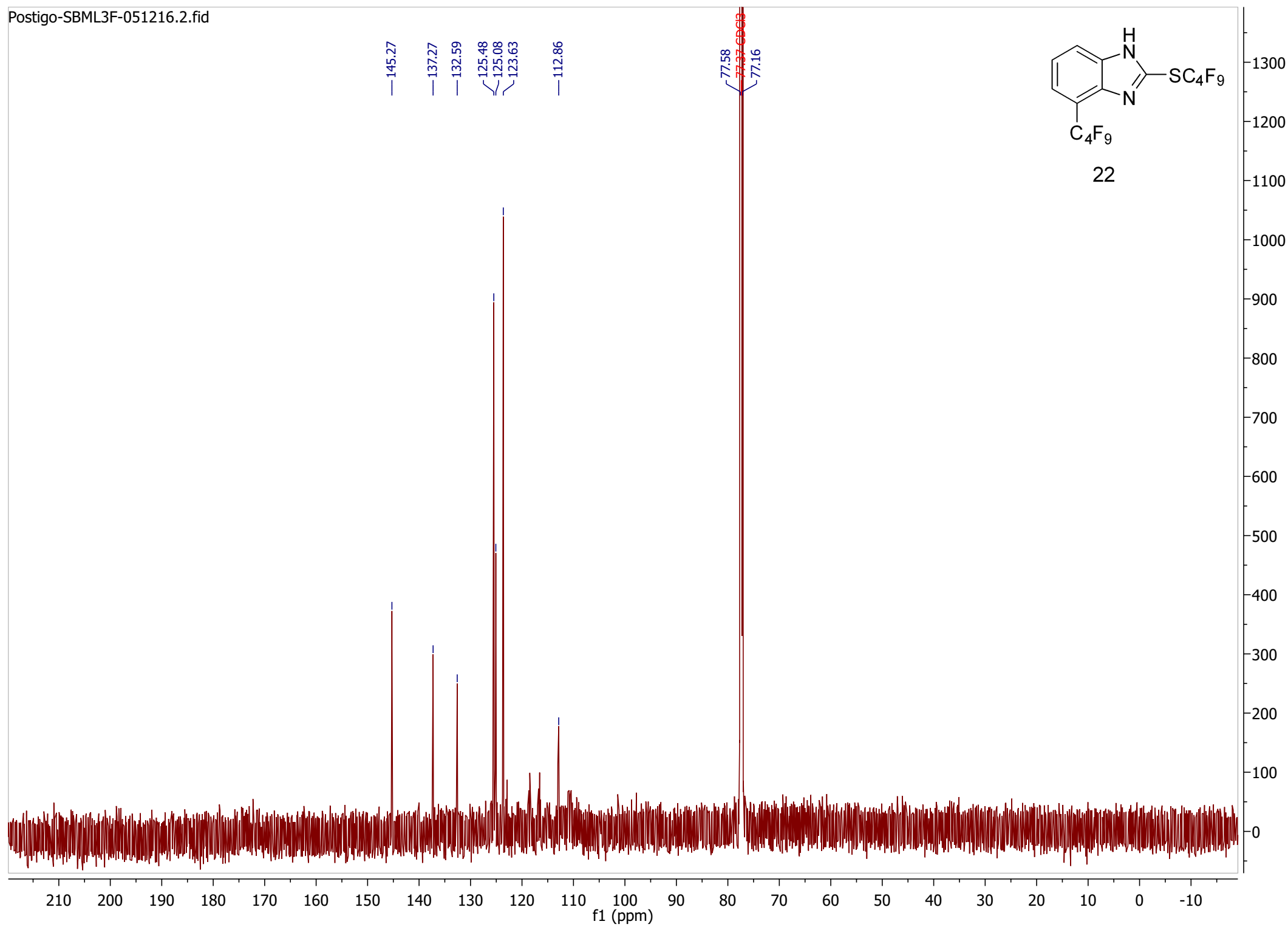


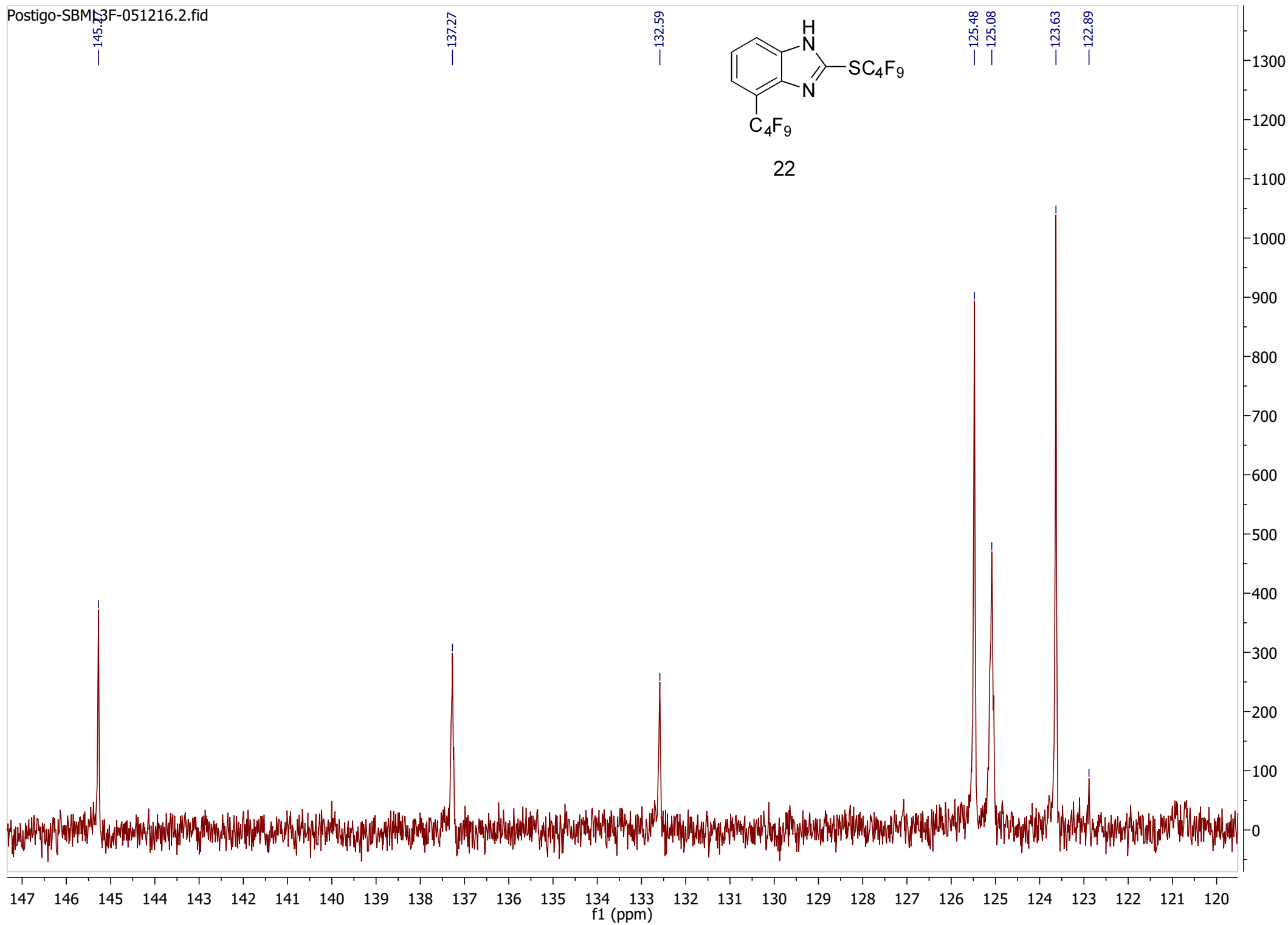


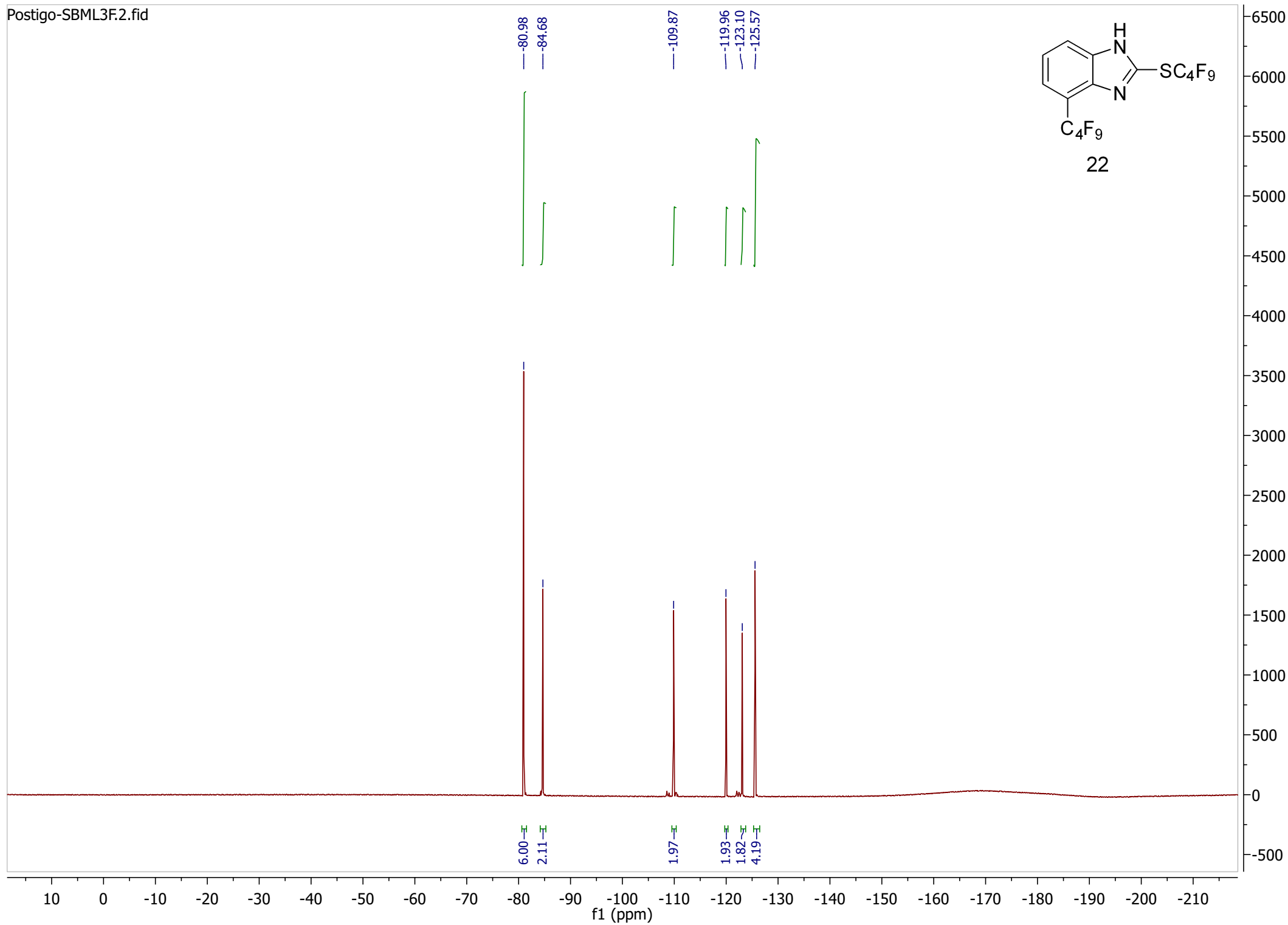


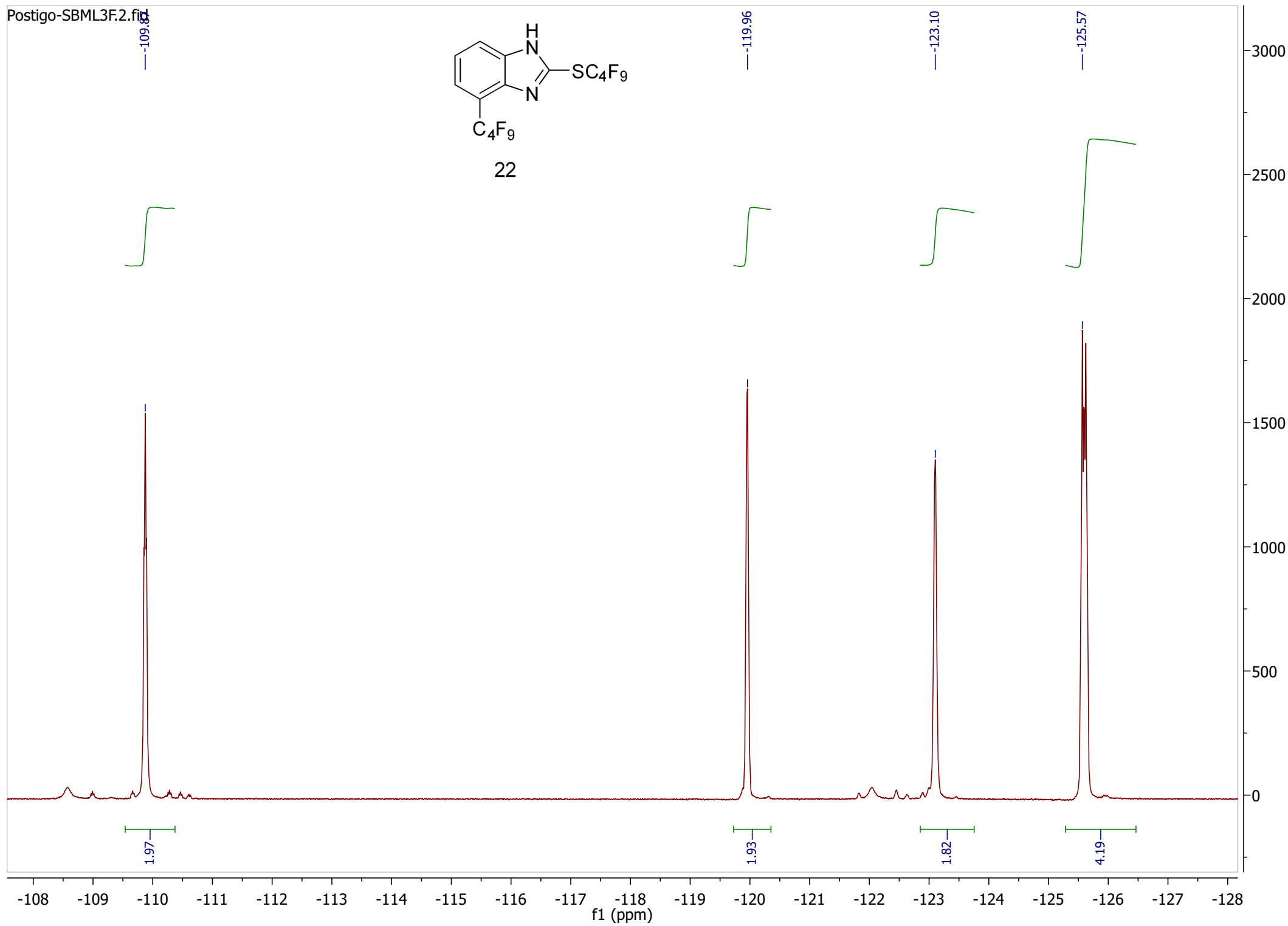


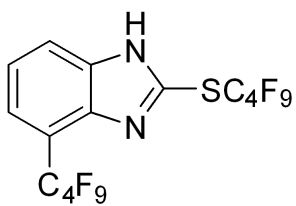




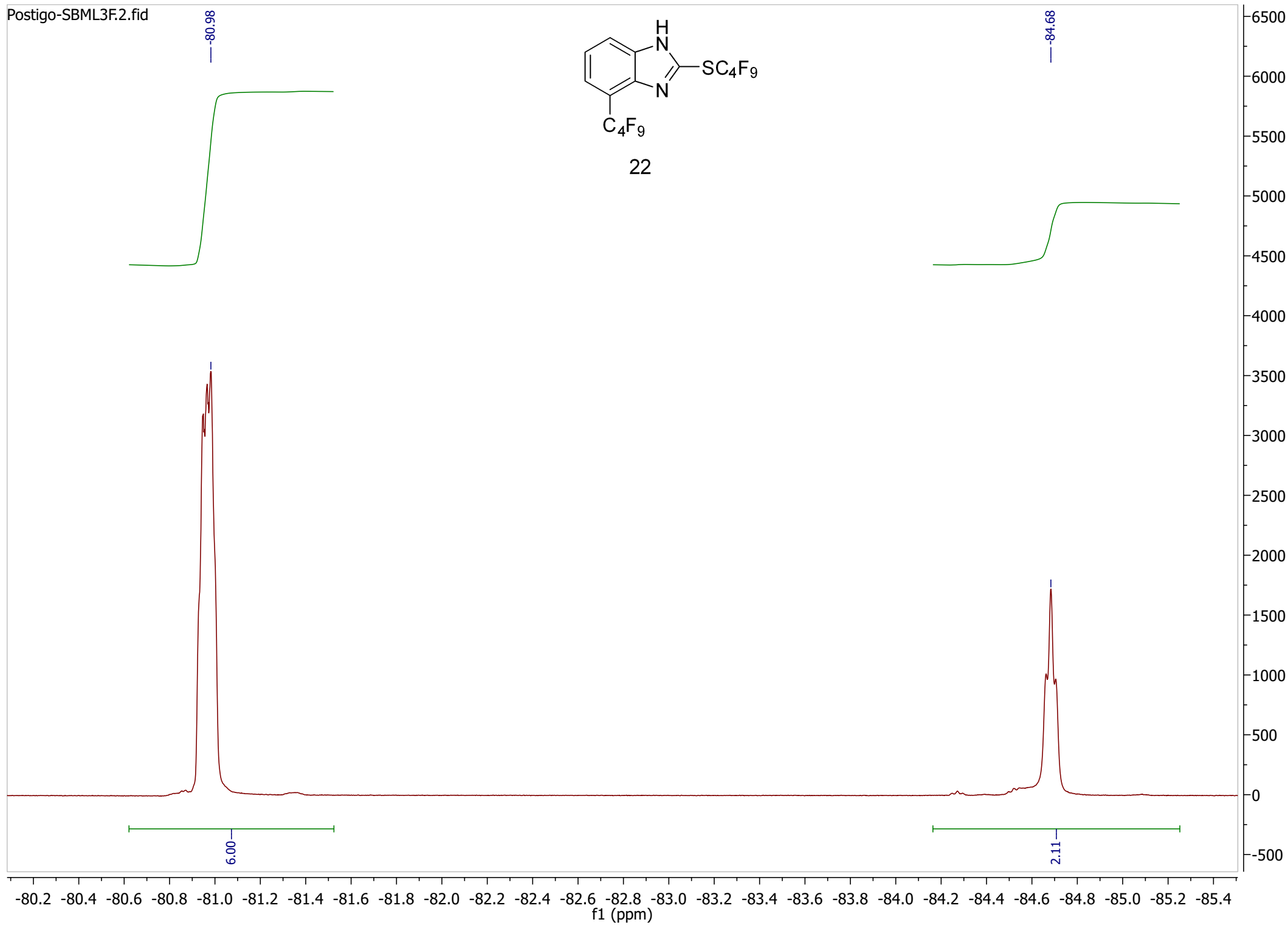








22



IR spectrum of the hydrazone derived from the carbonyl compound

