



Supporting Information

for

Synthesis of α -(perfluoroalkylsulfonyl)propiophenones: a new set of reagents for the light-mediated perfluoroalkylation of aromatics

Durbis J. Castillo-Pazos, Juan D. Lasso and Chao-Jun Li

Beilstein J. Org. Chem. **2022**, *18*, 788–795. doi:10.3762/bjoc.18.79

Detailed experimental procedures and compound characterization data

Table of contents

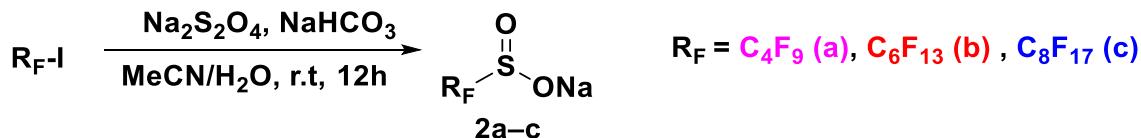
1. General experimental procedures:	S1
2. General procedure for the synthesis of the perfluoroalkylating reagents 1a–c (alkyl chains: -C ₈ F ₁₇ , -C ₆ F ₁₃ , -C ₄ F ₉)	S2
2.1 Step 1: Synthesis of sodium perfluoroalkyl sulfinate 2a–c	S2
2.2 Step 2: Synthesis of 2-iodopropiophenone (6)	S2
2.3 Step 3: Synthesis of the 1-phenyl-2-perfluoroalkylsulfonyl-propan-1-ones 1a–c	S3
2.4 Step 4: Conversion of byproduct B	S4
2.5 Single-crystal X-ray diffraction of byproduct B (perfluoroctyl analogue)	S5
3. General procedure for the perfluoroalkylation of aromatics	S6
3.1 Mechanistic evidence (Scheme 4)	S7
4. Characterization data of reported compounds:	S8
5. References	S12
Spectra collection	S13

1. General experimental procedures:

All reactions were carried out under argon atmosphere using standard Schlenk technique. ¹H NMR (500 MHz), ¹³C NMR (126 MHz) and ¹⁹F NMR (471 MHz) were recorded on an NMR spectrometer with CDCl₃ or acetone-*d*₆ as the solvent. Chemical shifts of ¹H, ¹³C and ¹⁹F NMR spectra are reported in parts per million (ppm). The residual solvent signals were used as references and the chemical shifts were converted to the TMS scale (CDCl₃: δ H = 7.26 ppm, δ C = 77.16 ppm). All coupling constants (*J* values) are reported in Hertz (Hz). High-resolution mass spectrometry was conducted through using atmospheric pressure chemical ionization (APCI) or electro-spraying ionization (ESI), and was performed at McGill University on a Thermo-Scientific Exactive Orbitrap. Protonated molecular ions [M + H]⁺ or sodium adducts [M+Na]⁺ were used for empirical formula confirmation. Column chromatography was performed either on silica gel 200–300 mesh, or on C8-reversed-phase perfluorinated silica gel.

2. General procedure for the synthesis of the perfluoroalkylating reagents 1a–c (alkyl chains: -C₈F₁₇, -C₆F₁₃, -C₄F₉)

2.1 Step 1: Synthesis of sodium perfluoroalkyl sulfinate 2a–c



The reaction was performed in an open 100 mL flask with a magnetic stirring bar. Sodium dithionite (5.22 g, 30 mmol) and sodium bicarbonate (2.0 g, 24 mmol) were suspended in a water/acetonitrile 2:1 solution (45 mL) cooled with a bath of ice and salt. Then, 25 mmol of the corresponding perfluoroalkyl iodide (perfluorooctyl-, perfluorohexyl-, perfluorobutyl-) were added. The reaction was left overnight to reach ambient temperature.[1] During this time, the generation of gas (carbon and sulfur dioxides) is observed, and the solution turns from colorless to pale transparent yellow with no presence of suspended salts.

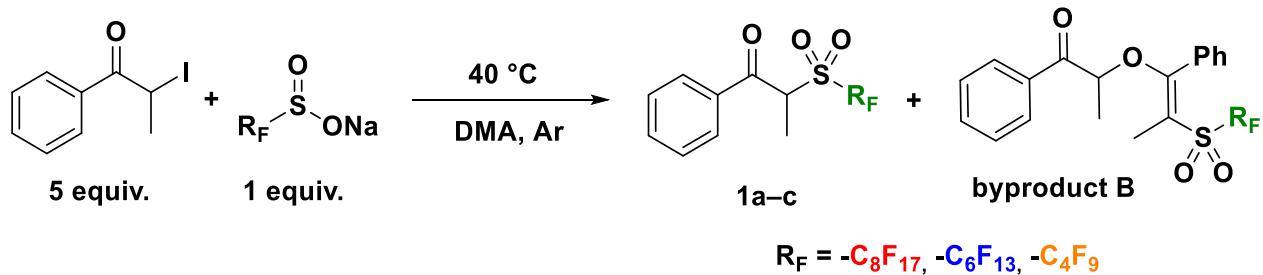
Once the reaction finished, the crude was evaporated to dryness while stirring at 82–100 °C. The solids at the bottom of the flask were washed with acetone until the fractions collected were colorless and passed through a fritted glass filter. For perfluorohexyl (**2b**) and perfluorooctyl sulfinate (**2c**) salts, the collected acetone fractions were evaporated with a rotary evaporator and under high vacuum to yield a pale-yellow solid that was later recrystallized from acetonitrile to give the final sodium sulfinate salt as a white crystalline salt. The crude sodium perfluorobutyl sulfinate (**2a**), was too soluble in acetonitrile and was instead washed with DCM until colorless on a fritted glass filter and allowed to dry. Isolated yields might range from quantitative to 50%, depending on the correct performance of the recrystallization step.

2.2 Step 2: Synthesis of 2-iodopropiophenone (**6**)



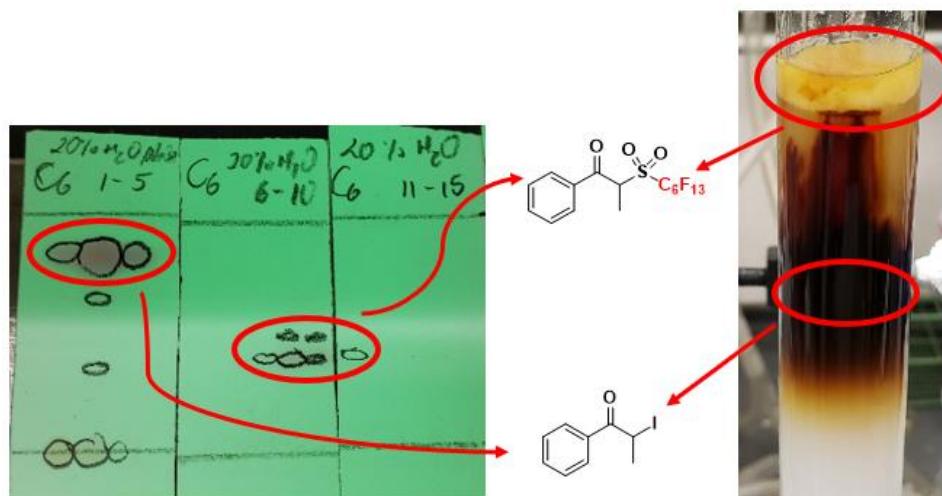
A solution of 20 mmol of sodium iodide (3.0 g) in 50 mL of acetone was stirred in a 100 mL open flask at room temperature for one or two minutes until saturation of the salt was evidenced by the appearance of a pale-yellow color. 2-Bromopropiophenone (2.13 g, 10 mmol) was added dropwise while stirring vigorously, leading to the formation of a cloudy yellow suspension due to the precipitation of sodium bromide (Finkelstein reaction) [2]. After 15 minutes, acetone was evaporated on a rotary evaporator and the product was extracted 3 times with ethyl acetate, washed with brine, and dried over anhydrous sodium sulfate. If the solution turned to a darker tone of brown, washing with saturated sodium thiosulfate would suffice to eliminate the forming iodine. The dried organic phases were evaporated to give a yellow oil in quantitative yield. The resulting product is sensitive to light and was typically synthesized for use on same day.

2.3 Step 3: Synthesis of the 1-phenyl-2-perfluoroalkylsulfonyl-propan-1-ones **1a–c**

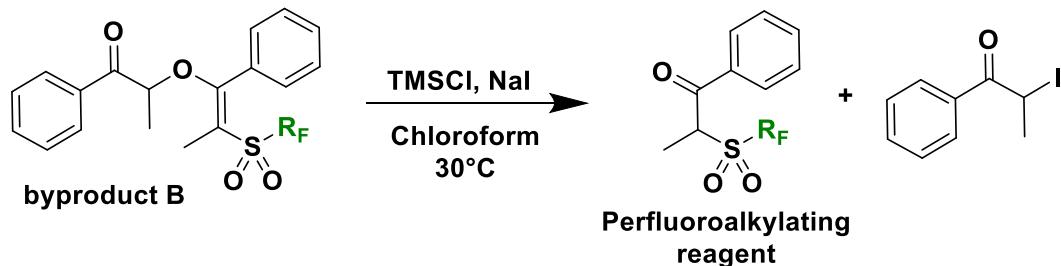


In a 25 mL flask equipped with a stirring bar, 3 mmol of the corresponding sodium sulfinate and 5 equivalents of 2-iodopropiophenone (13 g) were dissolved in 5 mL of dried DMA. The reaction was stirred vigorously for 16 h at 40 °C under inert atmosphere. Over time, the reaction mixture turned black due to the generation of iodine in solution. Once it was finished, the reaction was quenched with 30 mL of water, and the product was extracted with ethyl acetate ($3 \times 10 \text{ mL}$). The organic fractions were collected, washed with saturated sodium thiosulfate and brine, and evaporated. The excess of 2-iodopropiophenone was separated from the desired product through a C8 perfluorinated silica plug (40 g) using the following gradient: methanol/water 2:1 → methanol → acetone. The methanol and acetone fractions were collected and evaporated to give a clean mixture products **1a–c** and byproduct B (2:1, respectively) as a white powder. Isolated yield of the product at this stage can be up to 50%. The reaction demonstrated a scalability of up to 6 grams of product. Recycling of excess 2-iodopropiophenone utilized can be achieved by silica column with a gradient of 0% to 10% ethyl acetate in hexanes.

Note: When running the crude through the perfluorinated silica plug, constant pressure must be maintained for optimal separation. A successful separation is shown below:

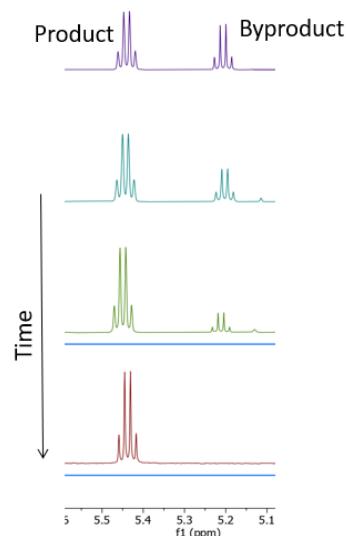


2.4 Step 4: Conversion of byproduct B



In a 50 mL flask, dissolve the mixture obtained in step 3 in the minimum amount possible of chloroform (to form a solution around 0.4 M). While stirring vigorously, add 5 equivalents of chlorotrimethylsilane and 5 equivalents of sodium iodide (calculated against the amount of byproduct B detected by NMR). Monitor the reaction at 30 °C until completion (approximately 1–3 hours) and wash with one portion of each: saturated bicarbonate solution, saturated sodium thiosulfate solution, and brine. After evaporating the chloroform on a rotary evaporator, the crude is purified through a C8 perfluorinated silica plug (40 g) using the following gradient: methanol/water 2:1 → methanol → acetone. The methanol and acetone fractions are collected and evaporated to give the final perfluoroalkylating reagent as a white powder. Isolated yield of the product at this final stage can be up to 70%.

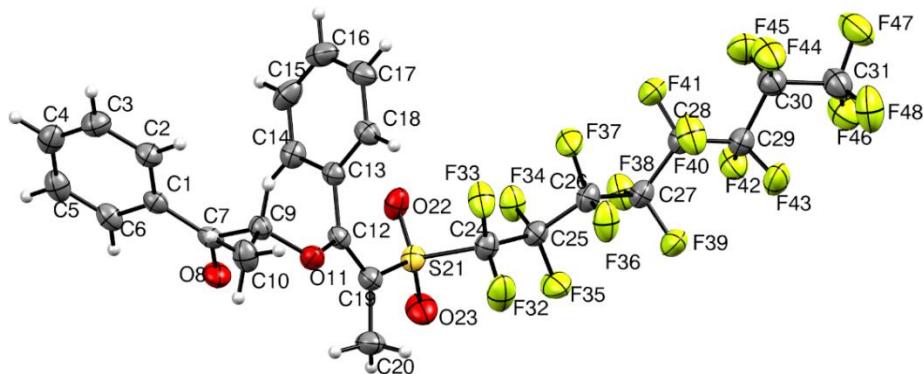
The most efficient way of monitoring the amount of byproduct present in the crude (if any) is to trace the proton in the alpha position through NMR as shown in the right:



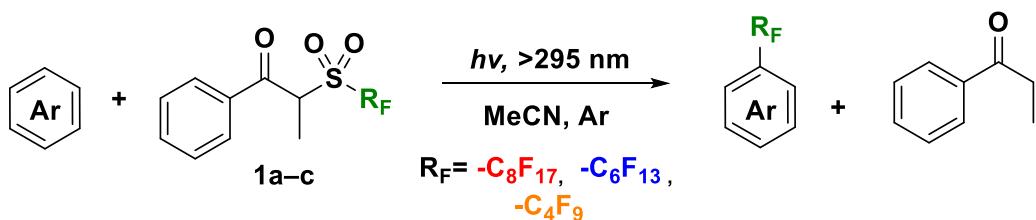
2.5 Single-crystal X-ray diffraction of byproduct B (perfluoroctyl analogue)

The byproduct was separated via column chromatography (100% toluene) and recrystallized from methanol over the course of 3 days at 4 °C to yield colorless needles. A Bruker D8 Advance powder X-ray diffractometer was used for the structure refinement and collection of the following crystal data:

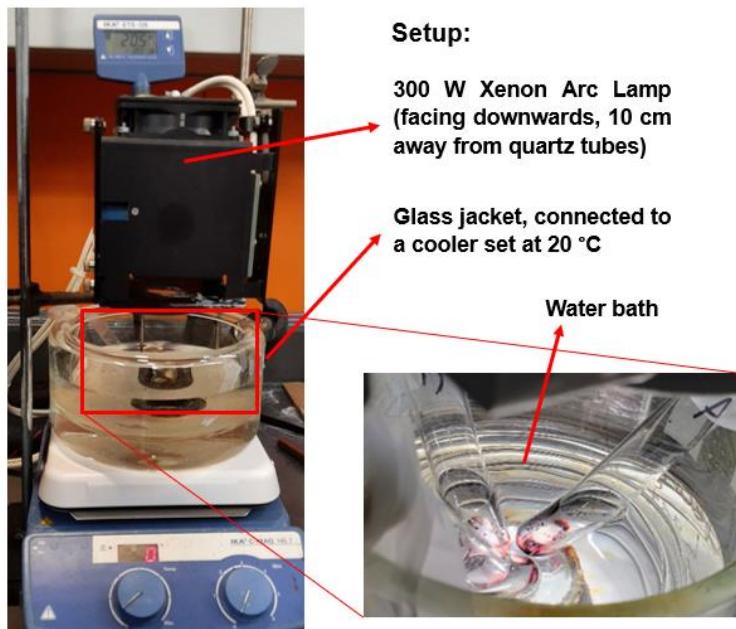
Empirical formula: C ₂₆ H ₁₇ F ₁₇ O ₄ S	Crystal size/mm ³ 0.540 × 0.320 × 0.310
Formula weight: 748.46	Radiation CuK α (λ = 1.54178)
Temperature/K: 180(2)	2 Θ range for data collection/° 7.416 to 144.608
Crystal system: monoclinic	Index ranges -58 ≤ h ≤ 58, -10 ≤ k ≤ 10, -12 ≤ l ≤ 17
Space group: C2/c	Reflections collected: 53403
a/Å: 47.7488(17)	Independent reflections: 5678 [R _{int} = 0.0485, R _{sigma} = 0.0260]
b/Å: 8.7035(3)	Data/restraints/parameters: 5678/0/435
c/Å: 14.0715(5)	Goodness-of-fit on F ₂ : 1.042
α /°: 90, β /°: 93.0800(10), γ /°: 90	Final R indexes [$I \geq 2\sigma(I)$] R ₁ = 0.0406, wR ₂ = 0.1067
Volume/Å ³ : 5839.4(4)	Final R indexes [all data] R ₁ = 0.0427, wR ₂ = 0.1090
Z: 8	Largest diff. peak/hole / e Å ⁻³ 0.28/-0.37
ρ calcg/cm ³ : 1.703	
μ /mm ⁻¹ : 2.329	
F(000): 2992.0	



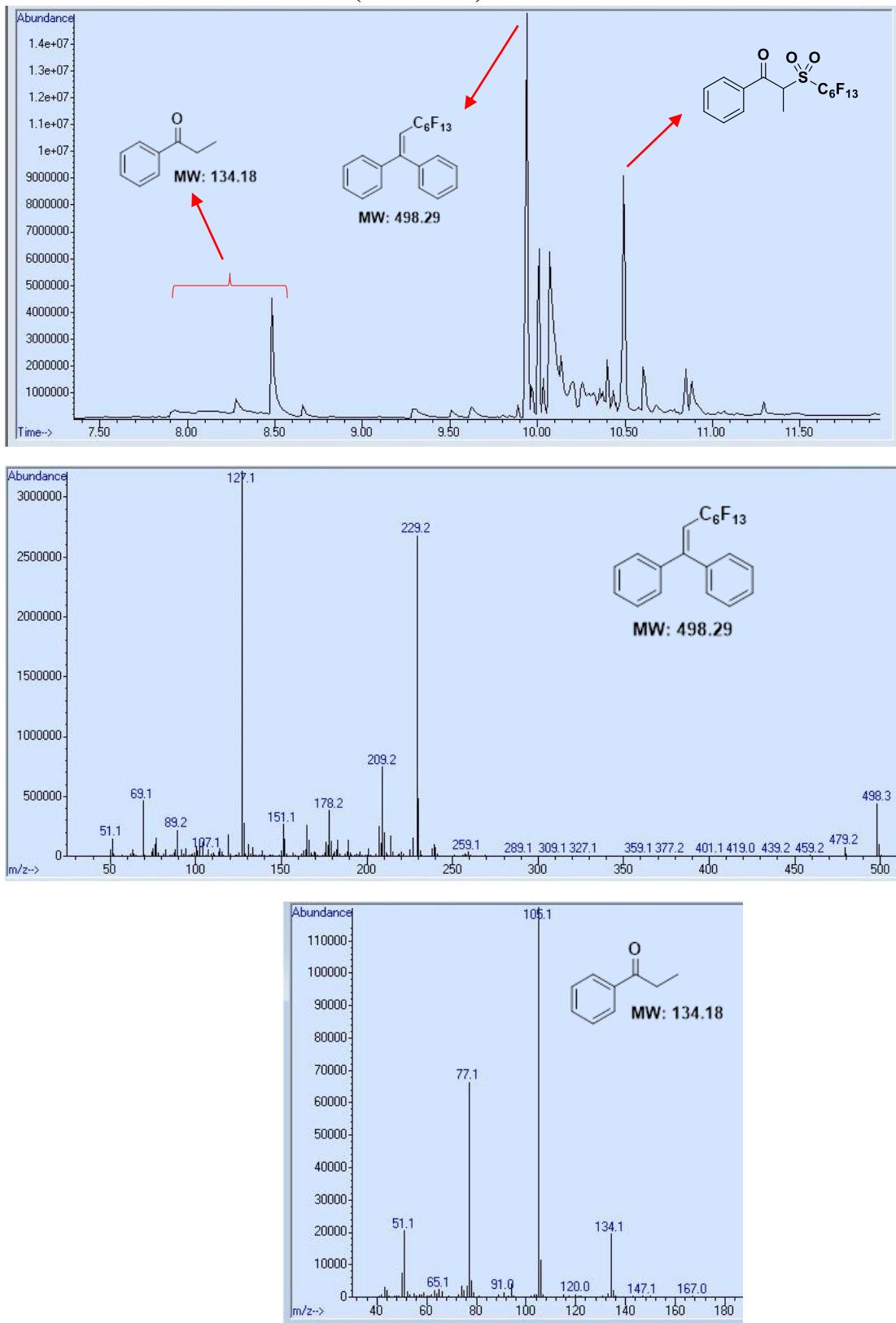
3. General procedure for the perfluoroalkylation of aromatics



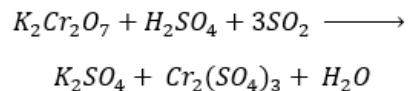
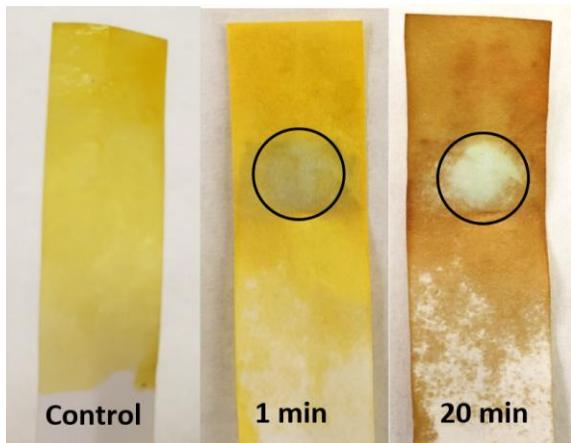
The aromatic substrate (0.1 mmol) and corresponding perfluoroalkylating reagent **1** (0.15–0.2 mmol) were added into 0.5 mL acetonitrile inside an air-tight quartz. Three freeze-pump-thaw cycles were carefully performed before setting the reaction under light irradiation at 20 °C using a 300 W xenon lamp. Temperature was controlled by a cold-water bath in a jacketed glass container connected to a cooler. After the reaction was finished (4–12 h), the product diluted with dichloromethane or acetone, evaporated under reduced pressure, and purified through a C8 perfluorinated silica plug (10–20 g) using the following gradient: methanol/water 2:1 → methanol → acetone. Methanol and acetone fractions were collected and evaporated to give the final product. An alternative purification method consists of preparative thin-layer chromatography, using ethyl acetate/hexanes or dichloromethane/hexanes as the eluent.



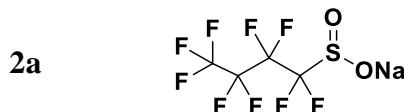
3.1 Mechanistic evidence (Scheme 4)



Detection of sulfur dioxide: A paper strip was coated in an acidic solution of potassium dichromate and was used to cap a needle that punctured the sealed reaction vessel. Over the course of 20 min, a blue-green coloration developed in the exposed area, indicating the presence of SO_2



4. Characterization data of reported compounds:



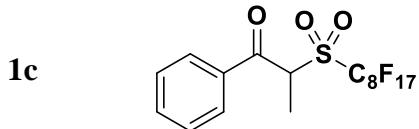
Sodium perfluorobutylsulfinate (2a) [3]: White crystals. Yield: 54%. ${}^{19}\text{F}$ NMR (471 MHz, Acetone) δ -81.90 – -82.00 (m), -124.32 (dd, J = 14.6, 7.3 Hz), -127.01 – -127.12 (m), -132.58 (td, J = 10.5, 9.1, 6.5 Hz). HRMS: m/z = 282.94816, theoretical = 282.94808



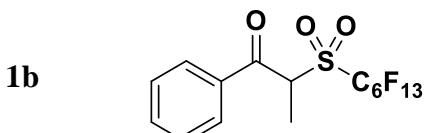
Sodium perfluorohexylsulfinate (2b) [3]: White crystals. Quantitative yield (highly dependent on workup conditions). ${}^{19}\text{F}$ NMR (471 MHz, Acetone) δ -81.58 – -81.85 (m), -115.16 – -115.45 (m), -121.11, -122.36, -123.36, -126.78 (ddd, J = 18.8, 11.1, 5.5 Hz). HRMS: m/z = 382.94052, theoretical = 382.94169



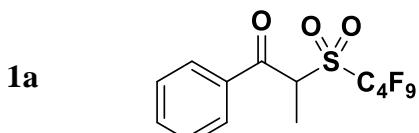
Sodium perfluorooctylsulfinate (2c) [4]: White crystals. Quantitative yield (highly dependent on workup conditions). ${}^{19}\text{F}$ NMR (471 MHz, Acetone) δ -81.67 (t, J = 10.1 Hz), -122.55 (d, J = 12.7 Hz), -123.26 (d, J = 19.5 Hz), -126.74, -132.39. HRMS: m/z = 482.93492, theoretical = 382.93530



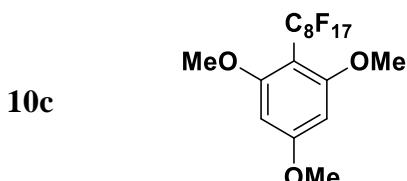
2-((Perfluorooctyl)sulfonyl)-1-phenylpropan-1-one (1c): White powder. 75% yield. ^1H NMR (500 MHz, CDCl_3) δ 8.00 – 7.94 (m, 2H), 7.72 – 7.65 (m, 1H), 7.58 – 7.51 (m, 2H), 5.42 (q, J = 7.0 Hz, 1H), 1.84 (dd, J = 7.0, 1.0 Hz, 3H). ^{13}C NMR (126 MHz, Acetone) δ 155.20, 150.96, 146.94, 136.54, 134.05, 128.72, 110.37, 33.40, 27.25. ^{19}F NMR (471 MHz, CDCl_3) δ -80.76, -109.18, -119.94, -121.36, -121.69, -121.90, -122.72, -126.11.



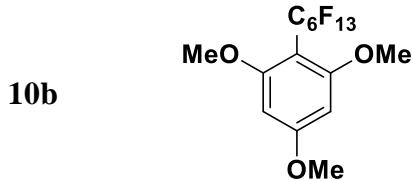
2-((Perfluorohexyl)sulfonyl)-1-phenylpropan-1-one (1b): White powder. 66% yield. ^1H NMR (500 MHz, CDCl_3) δ 8.02 – 7.96 (m, 2H), 7.75 – 7.68 (m, 1H), 7.61 – 7.54 (m, 2H), 5.44 (q, J = 7.0 Hz, 1H), 1.87 (d, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 188.83, 135.07, 134.90, 129.21, 128.96, 61.86, 13.17. ^{19}F NMR (471 MHz, CDCl_3) δ -80.72 (t, J = 10.0 Hz), -109.19 (t, J = 14.9 Hz), -119.97 (ddt, J = 30.5, 18.6, 7.6 Hz), -121.59 (d, J = 22.6 Hz), -122.50 – -122.72 (m), -126.05 (ddq, J = 14.3, 10.9, 4.0 Hz).



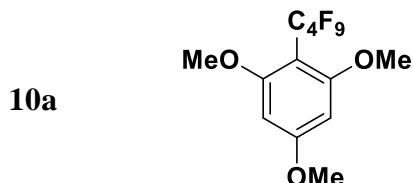
2-((Perfluorobutyl)sulfonyl)-1-phenylpropan-1-one (1a): Yellow solid (melts at room temperature). 30% yield. ^1H NMR (500 MHz, Acetone) δ 8.20 – 8.14 (m, 4H), 7.82 – 7.74 (m, 2H), 7.68 – 7.61 (m, 4H), 6.19 – 6.11 (m, 1H), 1.90 – 1.84 (m, 3H), 1.86 (s, 3H), 1.31 (s, 2H), 0.90 (t, J = 6.3 Hz, 1H). ^{19}F NMR (471 MHz, Acetone) δ -81.56 – -81.68 (m), -110.44 – -110.62 (m), -121.70 – -121.91 (m), -126.52 (td, J = 14.6, 14.1, 7.0 Hz).



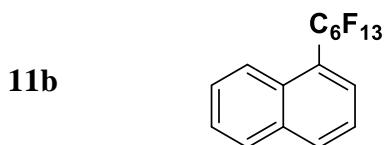
2-((Perfluorooctyl)-1,3,5-trimethoxybenzene (10c) [5]: White solid. 60% yield. ^1H NMR (500 MHz, CDCl_3) δ 6.17 (s, 2H), 3.86 (s, 3H), 3.83 (s, 6H). ^{13}C NMR (126 MHz, CDCl_3) δ 163.86, 161.76, 161.74, 91.70, 56.26, 55.28. ^{19}F NMR (471 MHz, CDCl_3) δ -80.88 (t, J = 10.0 Hz), -102.51 – -102.80 (m), -121.61 – -121.91 (m), -121.91 – -122.21 (m), -122.75 (dq, J = 26.7, 13.2, 11.2 Hz), -126.18 (dq, J = 14.5, 6.6 Hz).



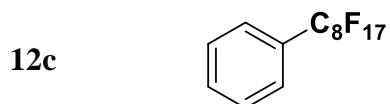
2-(Perfluorohexyl)-1,3,5-trimethoxybenzene (10b) [5]: White solid. 62% yield. ^1H NMR (500 MHz, CDCl_3) δ 6.17 (s, 2H), 3.86 (s, 3H), 3.83 (s, 6H). ^{13}C NMR (126 MHz, CDCl_3) δ 163.85, 161.76, 91.71, 56.30, 55.32. ^{19}F NMR (471 MHz, CDCl_3) δ -80.78 – -80.98 (m), -102.56 – -102.72 (m), -122.03 – -122.33 (m), -122.69 (qd, J = 16.8, 13.2, 5.7 Hz), -126.17 (td, J = 14.3, 6.4 Hz).



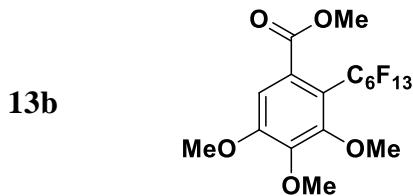
2-(Perfluorobutyl)-1,3,5-trimethoxybenzene (10a) [5]: White solid. 42% yield. ^1H NMR (500 MHz, CDCl_3) δ 6.17 (d, J = 1.0 Hz, 2H), 3.86 (s, 3H), 3.83 (s, 6H). ^{13}C NMR (126 MHz, CDCl_3) δ 163.84, 161.75, 91.71, 56.32, 55.33. ^{19}F NMR (471 MHz, CDCl_3) δ -80.89 – -81.05 (m), -102.83 (td, J = 13.6, 3.0 Hz), -122.97 (pq, J = 10.2, 4.6 Hz), -126.33 – -126.48 (m).



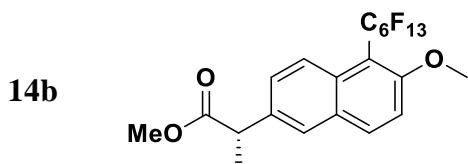
1-Perfluorohexylnaphthalene (11b): White solid. 72% yield. ^1H NMR (500 MHz, CDCl_3) δ 8.26 (d, J = 8.7 Hz, 1H), 8.09 (d, J = 8.2 Hz, 1H), 7.95 (dt, J = 9.1, 2.7 Hz, 1H), 7.86 (dd, J = 7.4, 1.2 Hz, 1H), 7.67 – 7.56 (m, 3H). ^{13}C NMR (201 MHz, CDCl_3) δ 134.10, 133.43, 130.29, 129.00, 128.03, 127.65, 127.16, 126.38, 124.83, 124.25. ^{19}F NMR (471 MHz, CDCl_3) δ -80.77, -104.42, -120.26, -121.46, -122.73, -126.07.



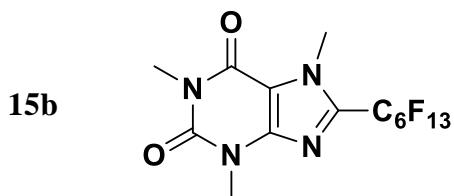
Perfluoroctylbenzene (12c): Yellow oil. 68% yield. ^1H NMR (500 MHz, CDCl_3) δ 7.64 – 7.57 (m, 3H), 7.53 (t, J = 7.6 Hz, 2H). ^{13}C NMR (201 MHz, CDCl_3) δ 131.92, 128.60, 126.78. ^{19}F NMR (471 MHz, CDCl_3) δ -80.81, -110.73, -121.27, -121.90, -122.73, -126.19.



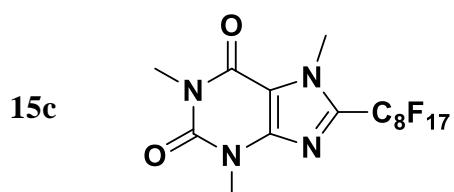
Methyl 3,4,5-trimethoxy-2-(perfluorohexyl)benzoate (13b) [6]: White solid. 64% yield. ^1H NMR (500 MHz, CDCl_3) δ 6.78 (s, 1H), 3.94 (d, $J = 1.5$ Hz, 6H), 3.91 (s, 3H), 3.89 (s, 3H). ^{13}C NMR (201 MHz, CDCl_3) δ 168.61, 156.17, 153.90, 144.01, 136.89, 136.16, 133.34, 132.93, 130.17, 128.79, 128.61, 128.50, 128.48, 107.33, 61.91, 60.85, 56.26, 52.86. ^{19}F NMR (471 MHz, CDCl_3) δ -80.74, -100.99.



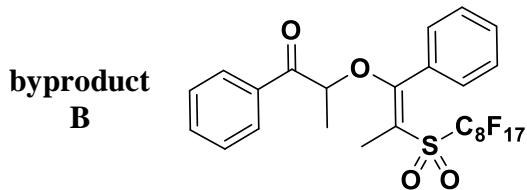
Naproxen methyl ester-C₆F₁₃ 14b: White solid. 20% yield. ^1H NMR (800 MHz, CDCl_3) δ 8.15 (d, $J = 9.2$ Hz, 1H), 7.98 (d, $J = 9.1$ Hz, 1H), 7.70 (d, $J = 2.1$ Hz, 1H), 7.49 (dd, $J = 9.2, 2.1$ Hz, 2H), 7.34 (d, $J = 9.1$ Hz, 1H), 3.97 (s, 3H), 3.87 (q, $J = 7.3$ Hz, 1H), 3.68 (s, 3H), 1.58 (d, $J = 7.1$ Hz, 5H). ^{13}C NMR (201 MHz, CDCl_3) δ 174.76, 159.04, 136.18, 134.93, 131.38, 129.46, 126.87, 125.18, 114.68, 57.42, 52.16, 44.93, 18.38. ^{19}F NMR (471 MHz, CDCl_3) δ -80.72, -100.17, -120.67, -121.94, -122.61, -126.06.



Caffeine-C₆F₁₃ 15b [7]: White solid. 25% yield. ^1H NMR (500 MHz, Acetone) δ 4.26 (t, $J = 2.0$ Hz, 6H), 3.51 (s, 6H), 3.33 (s, 6H), 2.10 (d, $J = 1.2$ Hz, 1H), 1.31 (s, 1H). ^{13}C NMR (126 MHz, Acetone) δ 155.20, 150.95, 146.94, 129.52, 128.43, 110.37, 33.40, 27.25. ^{19}F NMR (471 MHz, Acetone) δ -81.51 – -81.84 (m), -81.71, -109.18 (t, $J = 14.4$ Hz), -121.34 – -121.70 (m), -121.84, -122.32, -123.28, -126.69 (ddq, $J = 19.5, 11.7, 4.0$ Hz).



Caffeine-C₈F₁₇ 15c [7]: White solid. 23% yield. ^1H NMR (500 MHz, Acetone) δ 4.26 (t, $J = 2.0$ Hz, 3H), 3.52 (s, 3H), 3.33 (s, 3H). ^{19}F NMR (471 MHz, Acetone) δ -81.64 (t, $J = 10.3$ Hz), -109.16 (t, $J = 14.3$ Hz), -121.27 – -121.80 (m), -122.22, -122.33, -123.21, -126.51 – -126.89 (m).



(E)-2-((2-((Perfluorooctyl)sulfonyl)-1-phenylprop-1-en-1-yl)oxy)-1-phenylpropan-1-one

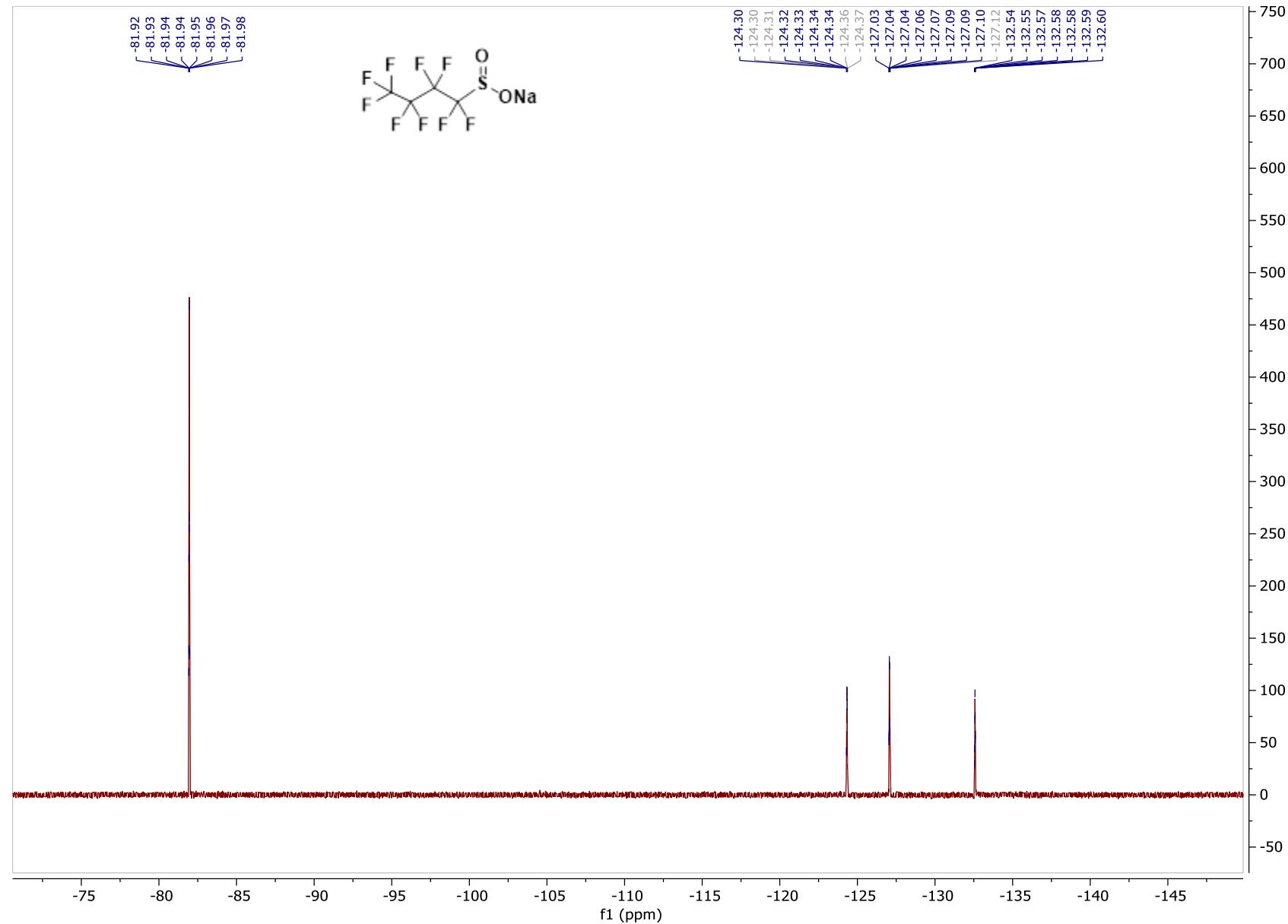
(byproduct B): White solid. ^1H NMR (500 MHz, acetone) δ 7.58 – 7.54 (m, 2H), 7.45 – 7.41 (m, 2H), 7.37 (tt, J = 7.5, 1.3 Hz, 1H), 5.50 (q, J = 6.9 Hz, 1H), 2.33 (d, J = 0.8 Hz, 3H), 1.55 (d, J = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, acetone) δ 205.15, 197.07, 189.54, 173.10, 142.56 – 120.65 (m), 133.71, 130.22, 129.14, 129.08, 128.74, 127.86, 109.73, 77.13, 62.37, 17.97, 12.78, 12.77. ^{19}F NMR (471 MHz, Acetone) δ -81.58 – -81.69 (m), -111.91 (dd, J = 17.3, 13.0 Hz), -120.88 (tt, J = 14.0, 5.5 Hz), -121.47 – -122.86 (m), -123.22, -126.02 – -128.00 (m).

5. References

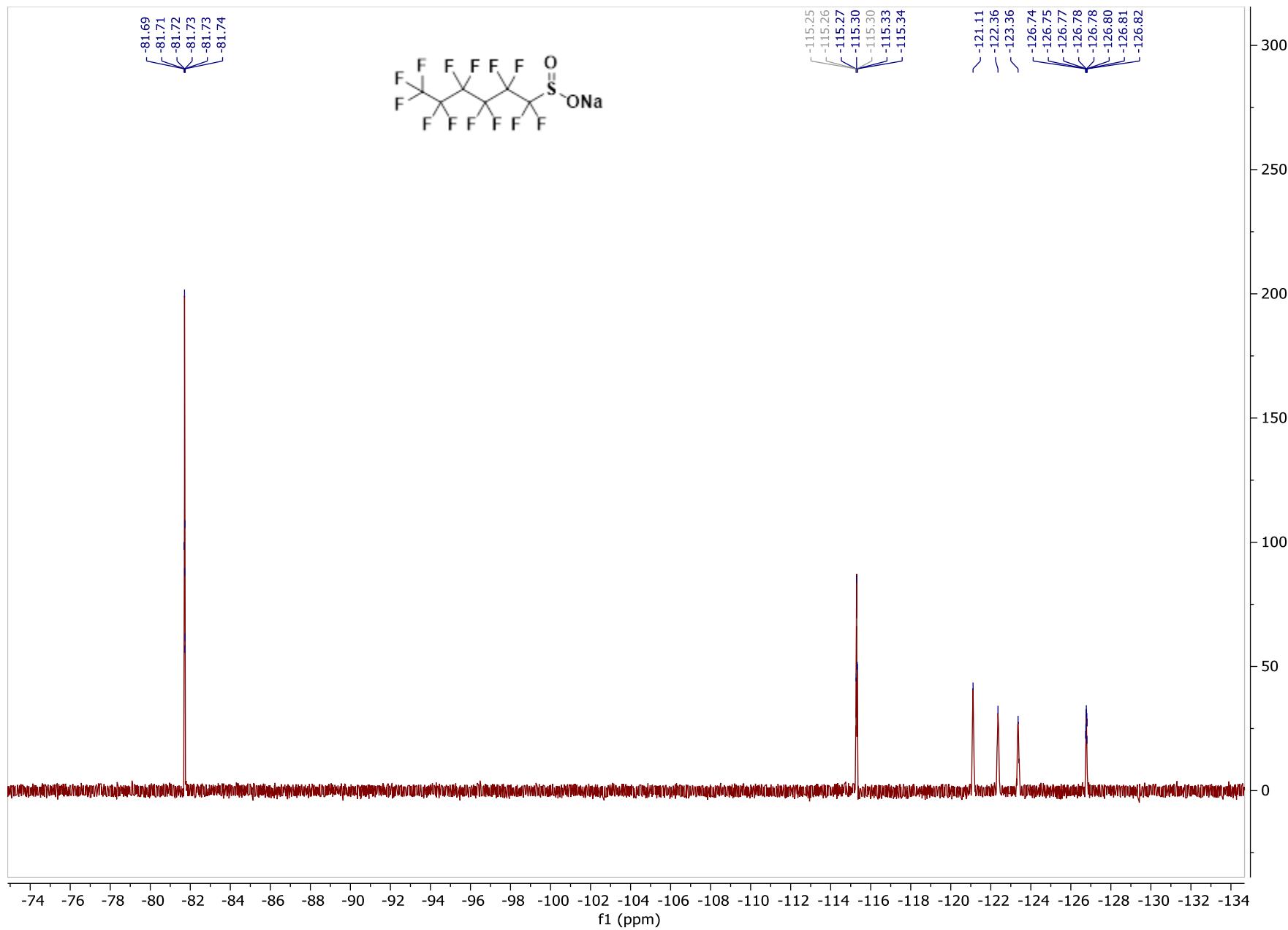
1. Dmowski, W.; Piasecka-Maciejewska, K. *J. Fluorine Chem.* **2005**, *126*, 877-882.
2. Finkelstein, H. *Berichte der deutschen chemischen Gesellschaft* **1910**, *43*, 1528-1532.
3. Hu, C.; Qing, F.; Huang, W. *J. Org. Chem.* **1991**, *56*, 2801-2804.
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5. Tasnim, T.; Ryan, C.; Christensen, M. L.; Fennell, C. J.; Pitre, S. P. *Org. Lett.* **2022**, *24*, 446-450.
6. JP Patent: JPS5775932A
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Spectra collection

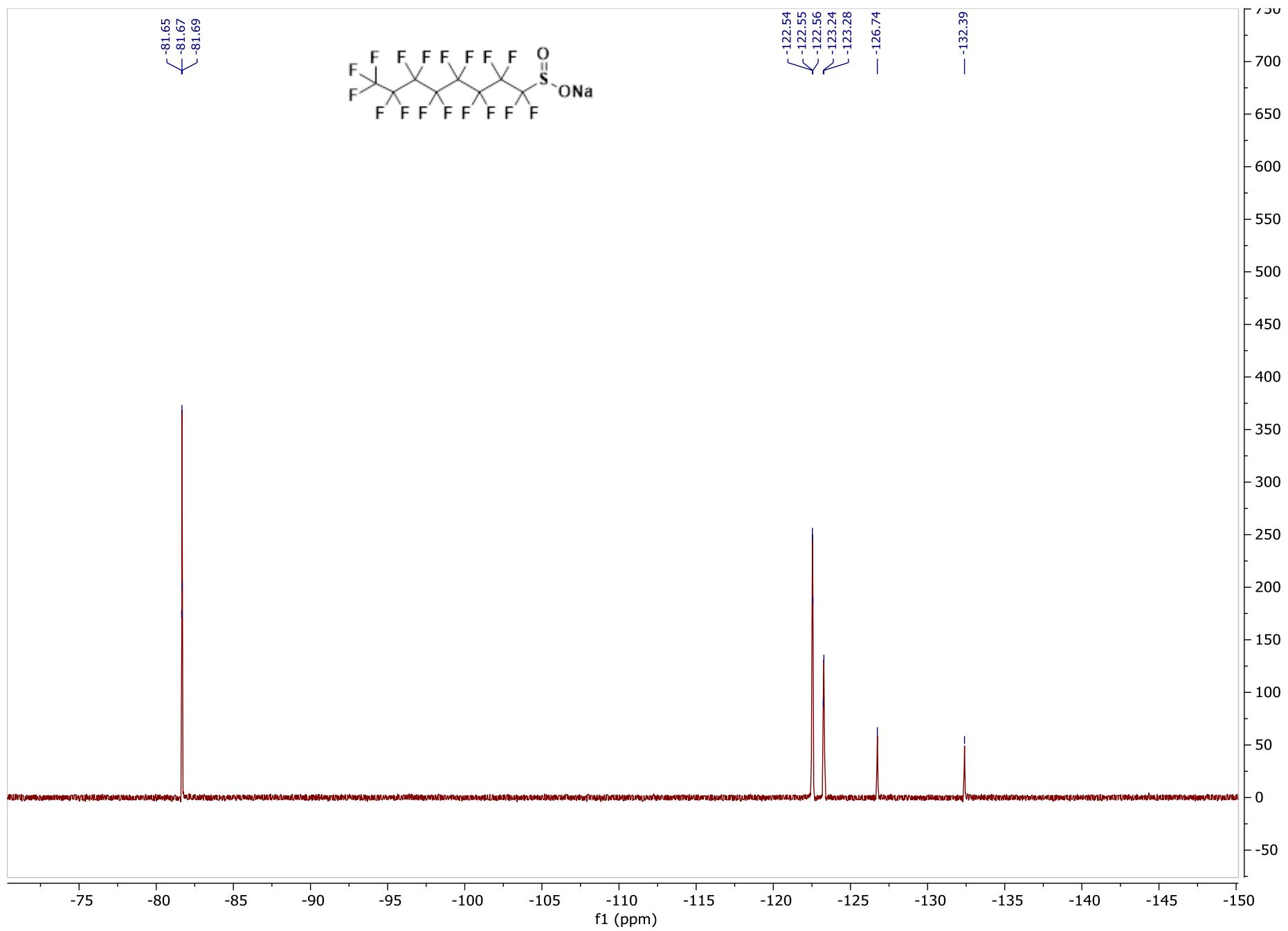
^{19}F NMR of **2a**, acetone- d_6 , 471 MHz



¹⁹F NMR of **2b**, acetone-*d*₆, 471 MHz

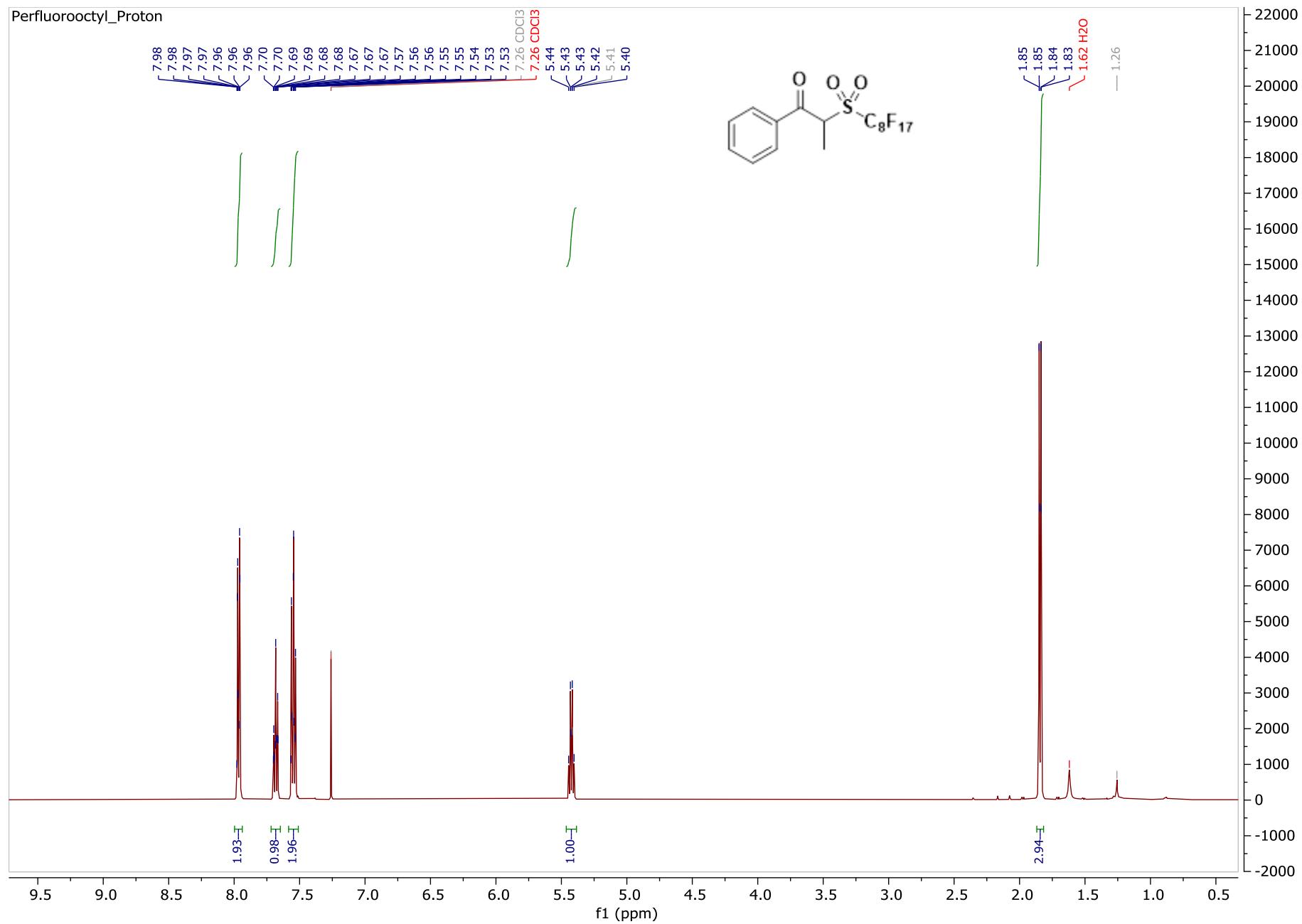


¹⁹F NMR of **2c**, acetone-*d*⁶, 471 MHz

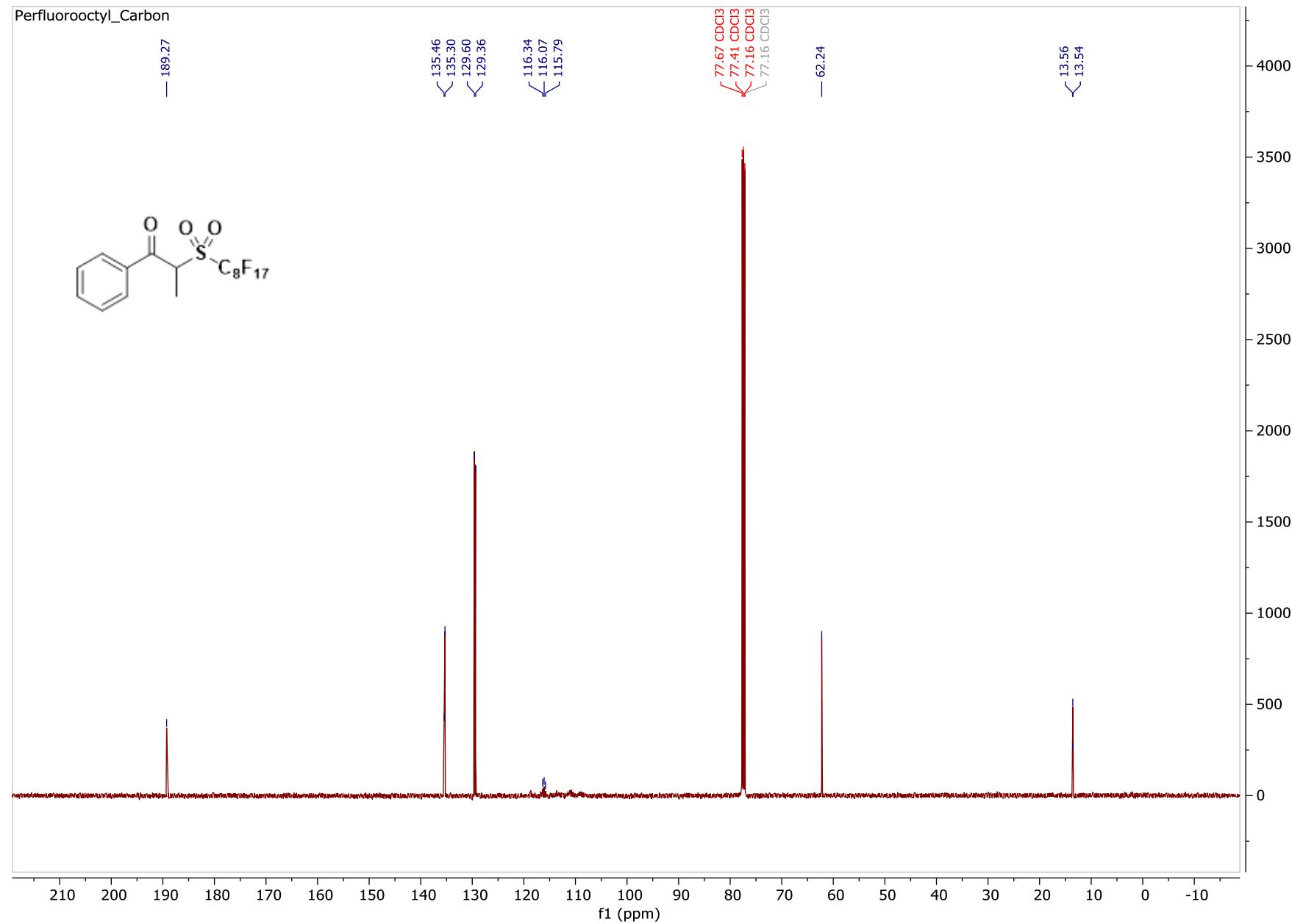


¹H NMR of **1c**, CDCl₃, 500 MHz

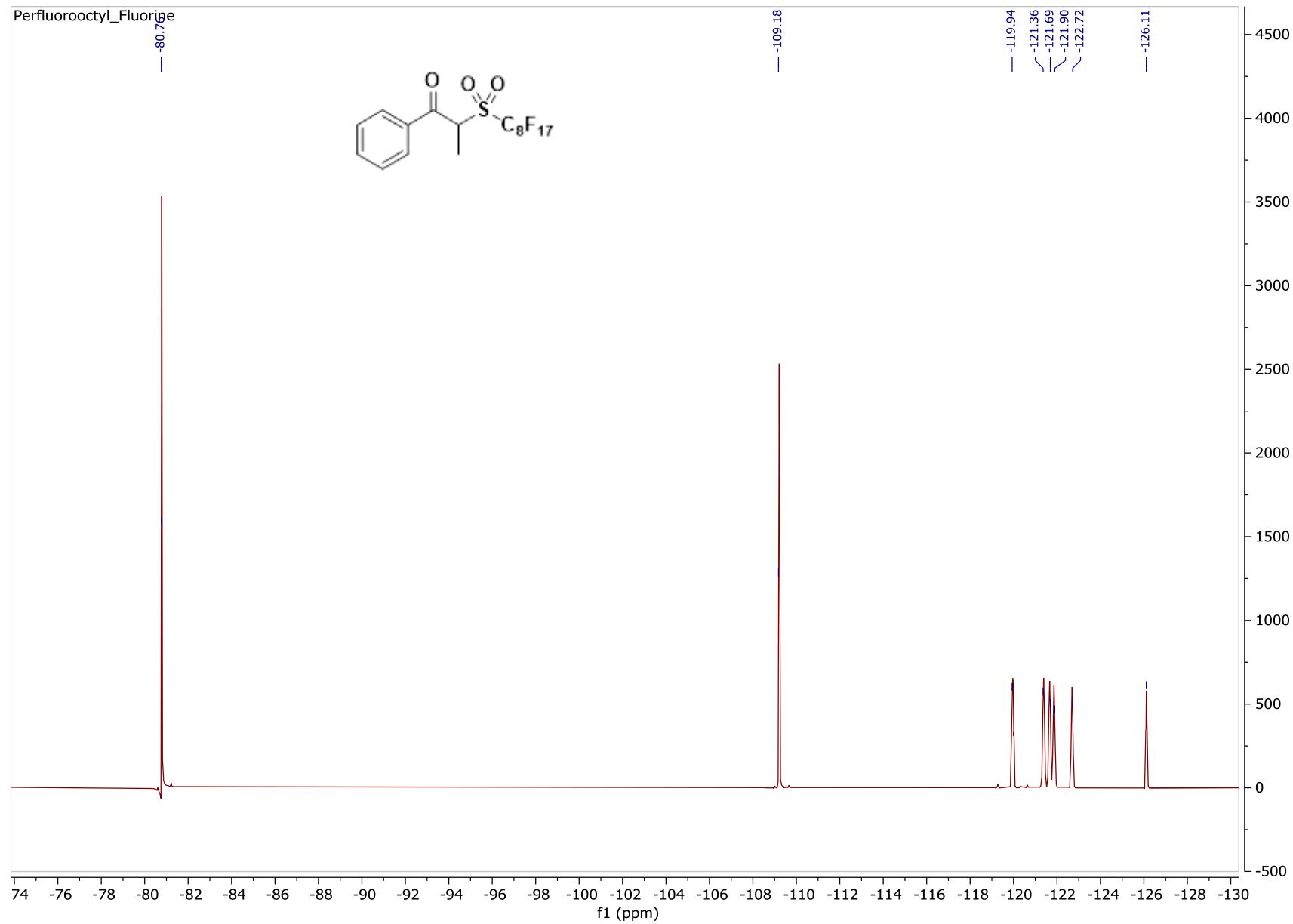
Perfluorooctyl_Proton



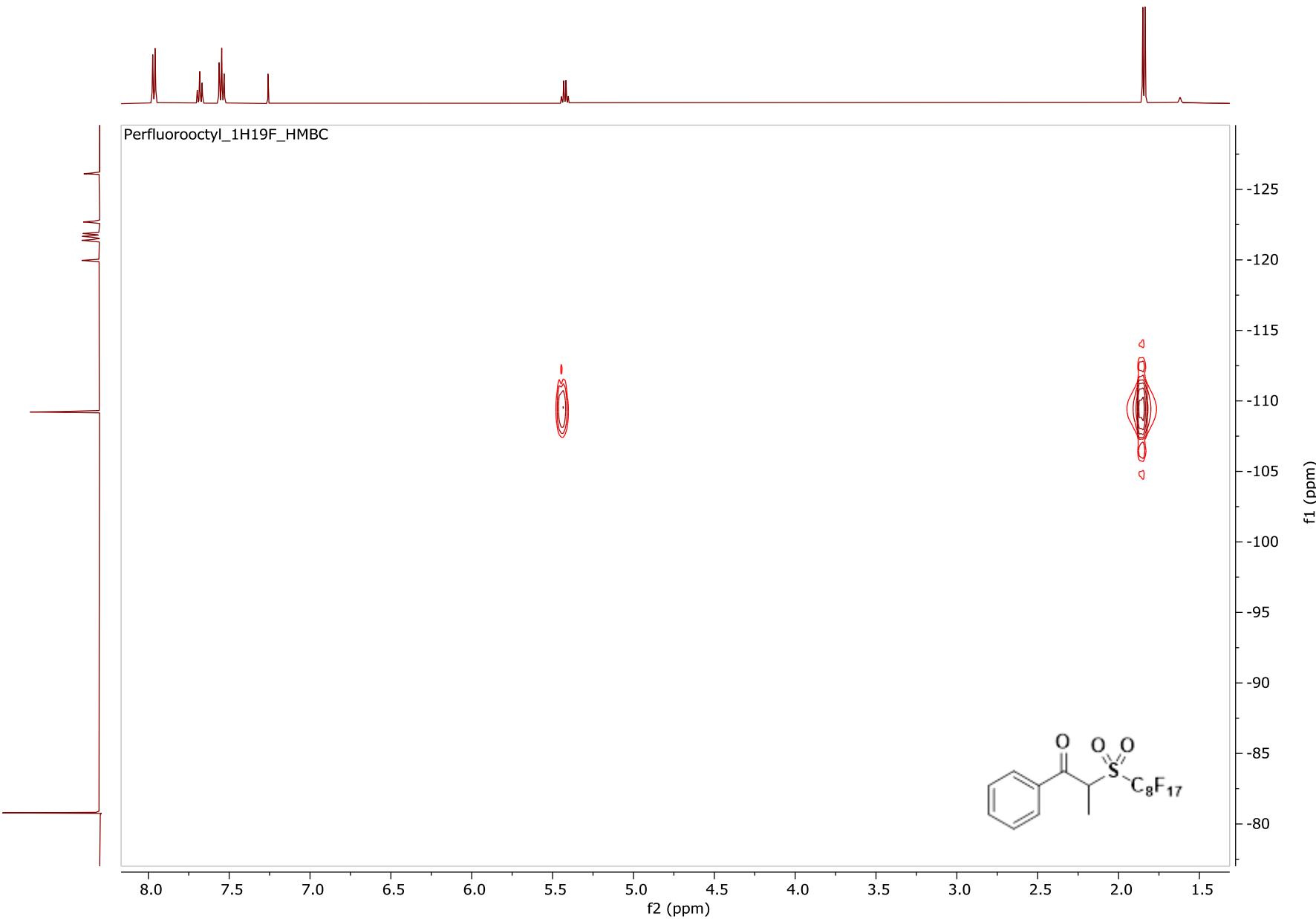
¹³C NMR of **1c**, CDCl₃, 126 MHz



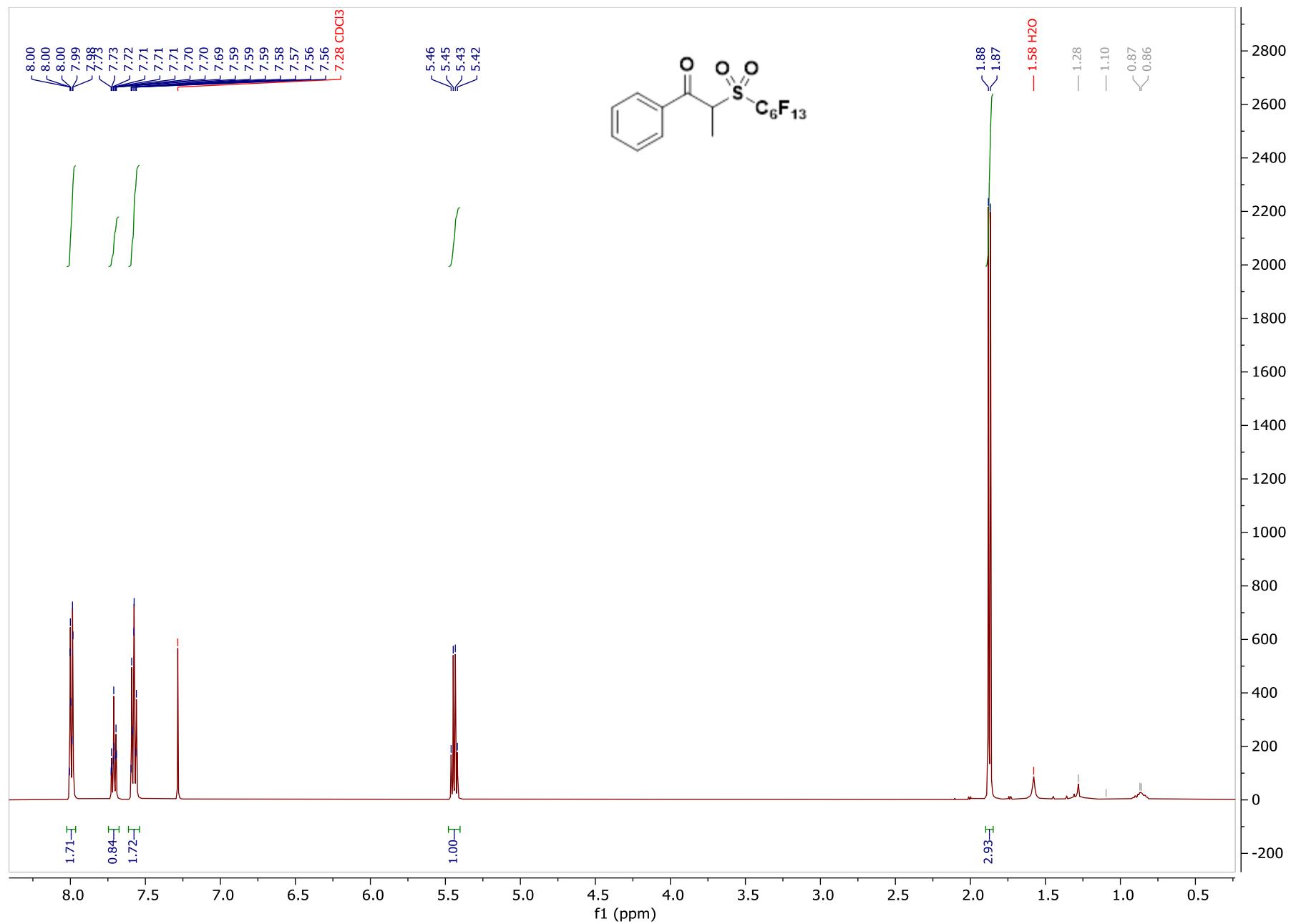
¹⁹F NMR of **1c**, CDCl₃, 471 MHz



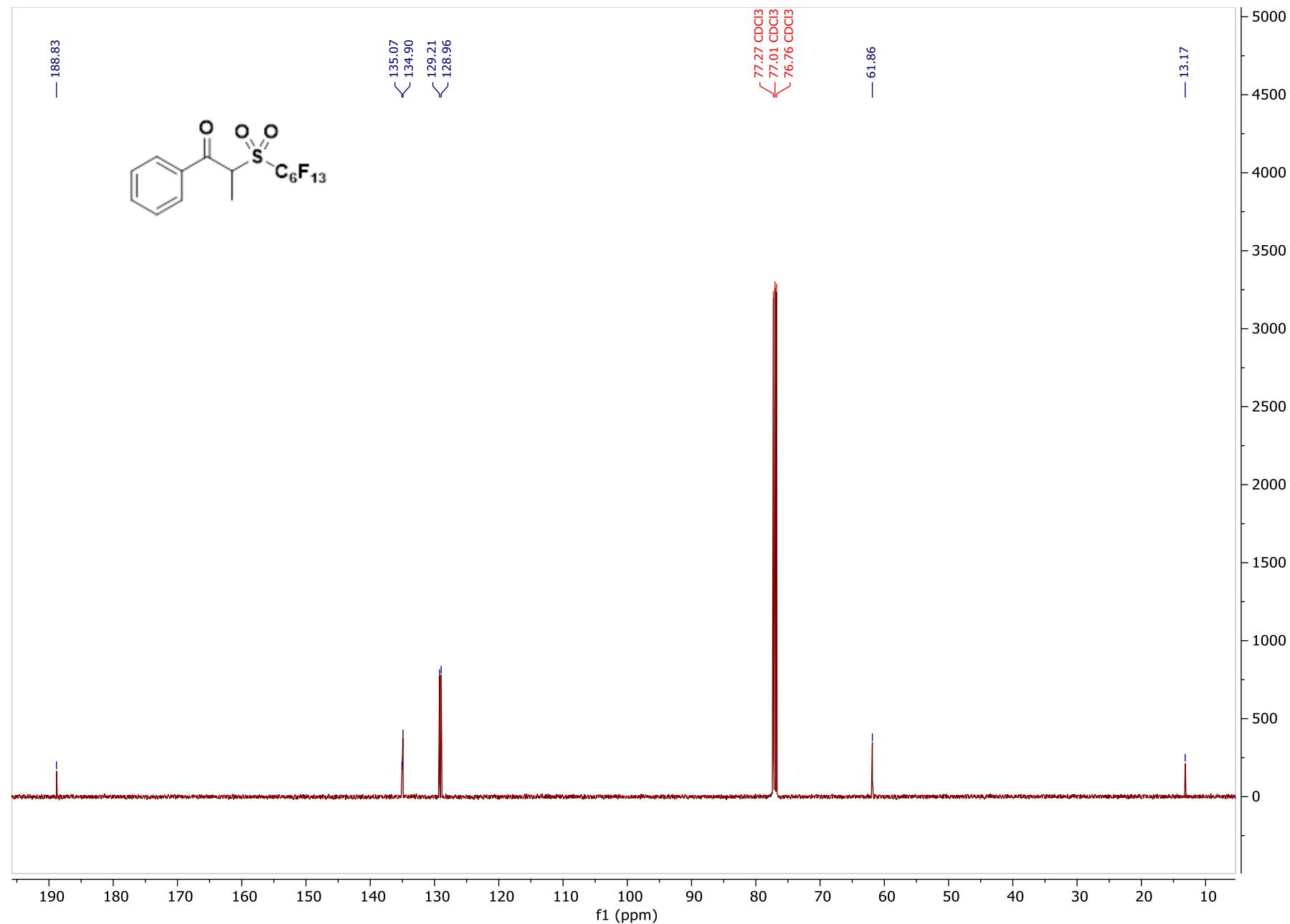
¹H-¹⁹F HMBC of **1c**



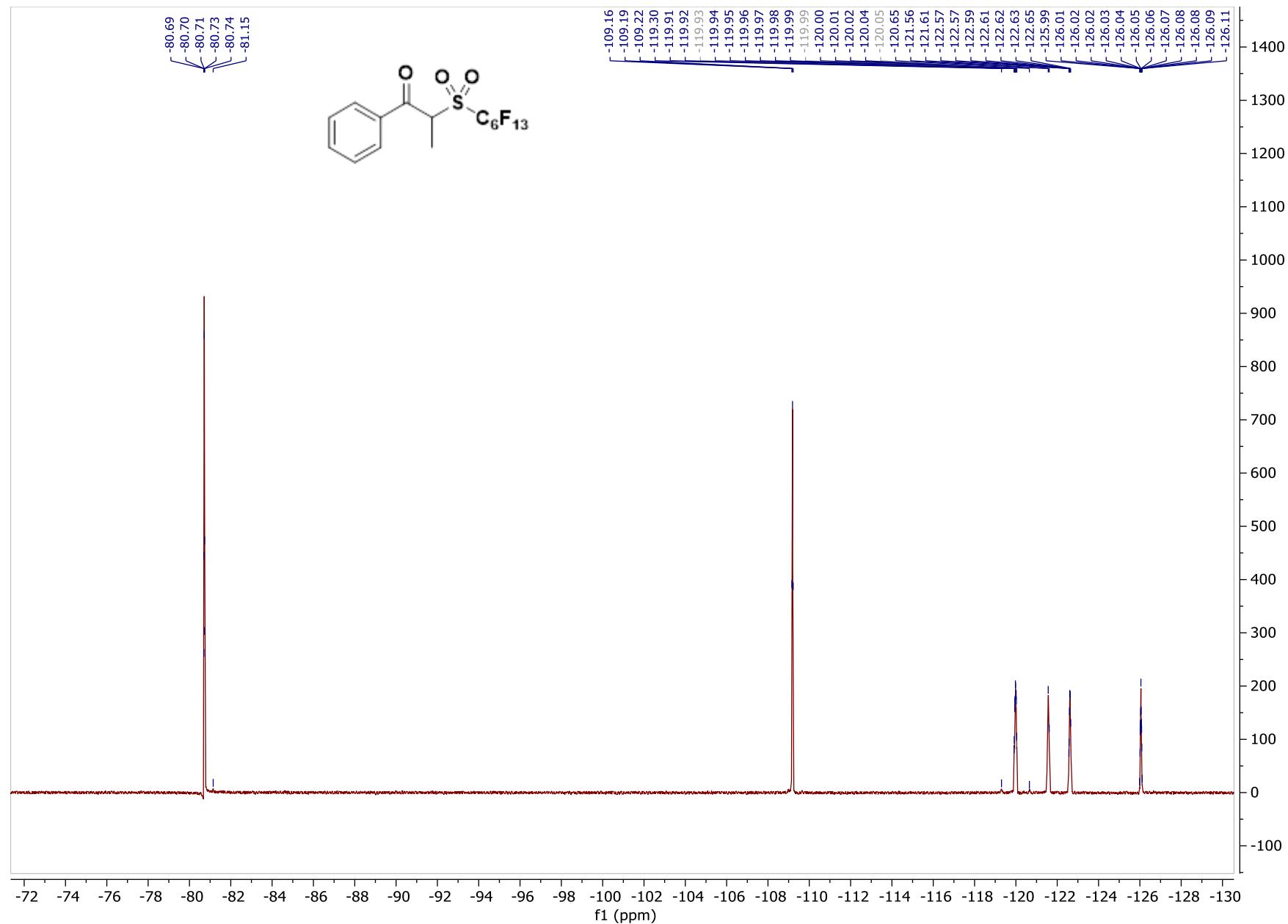
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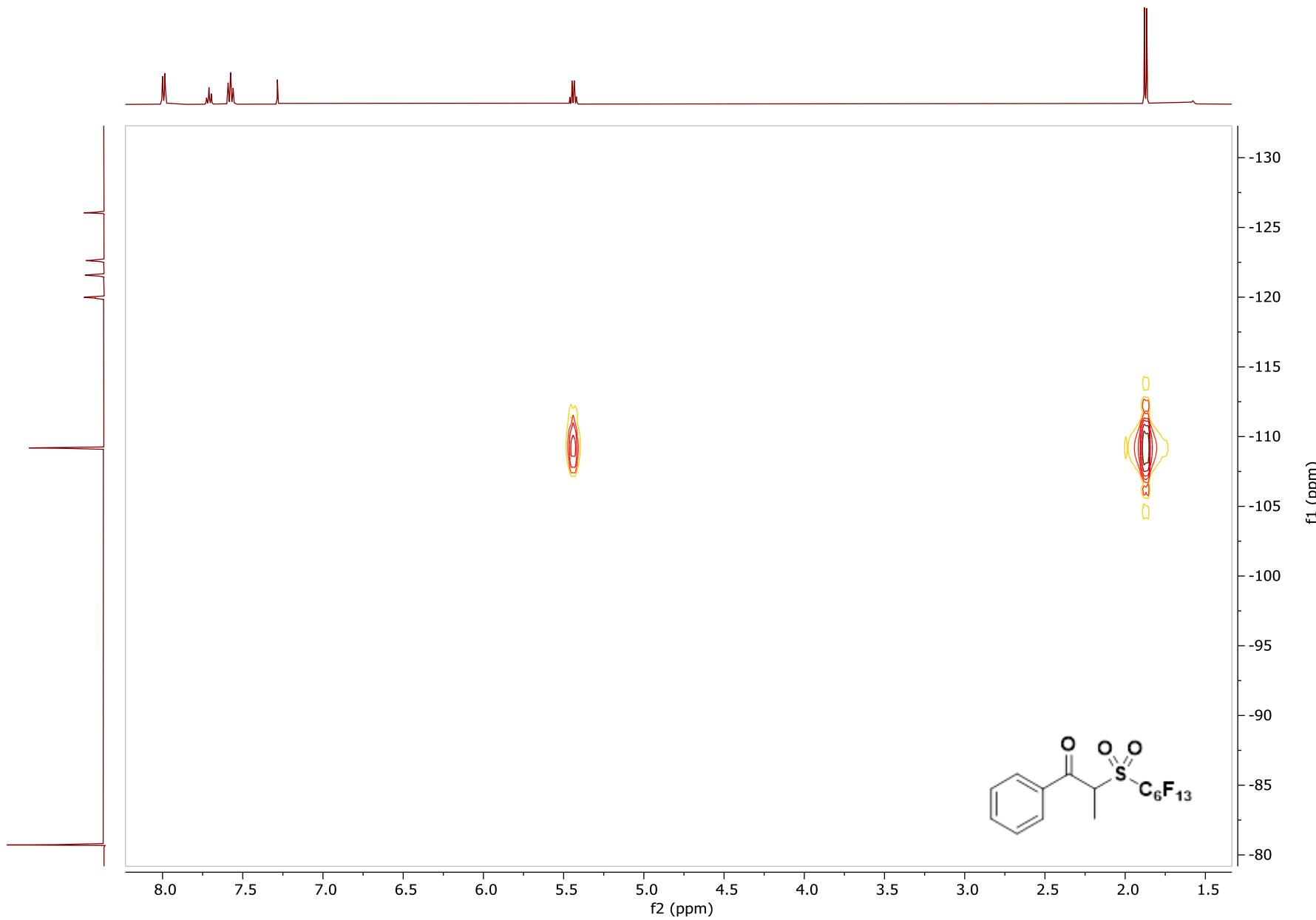
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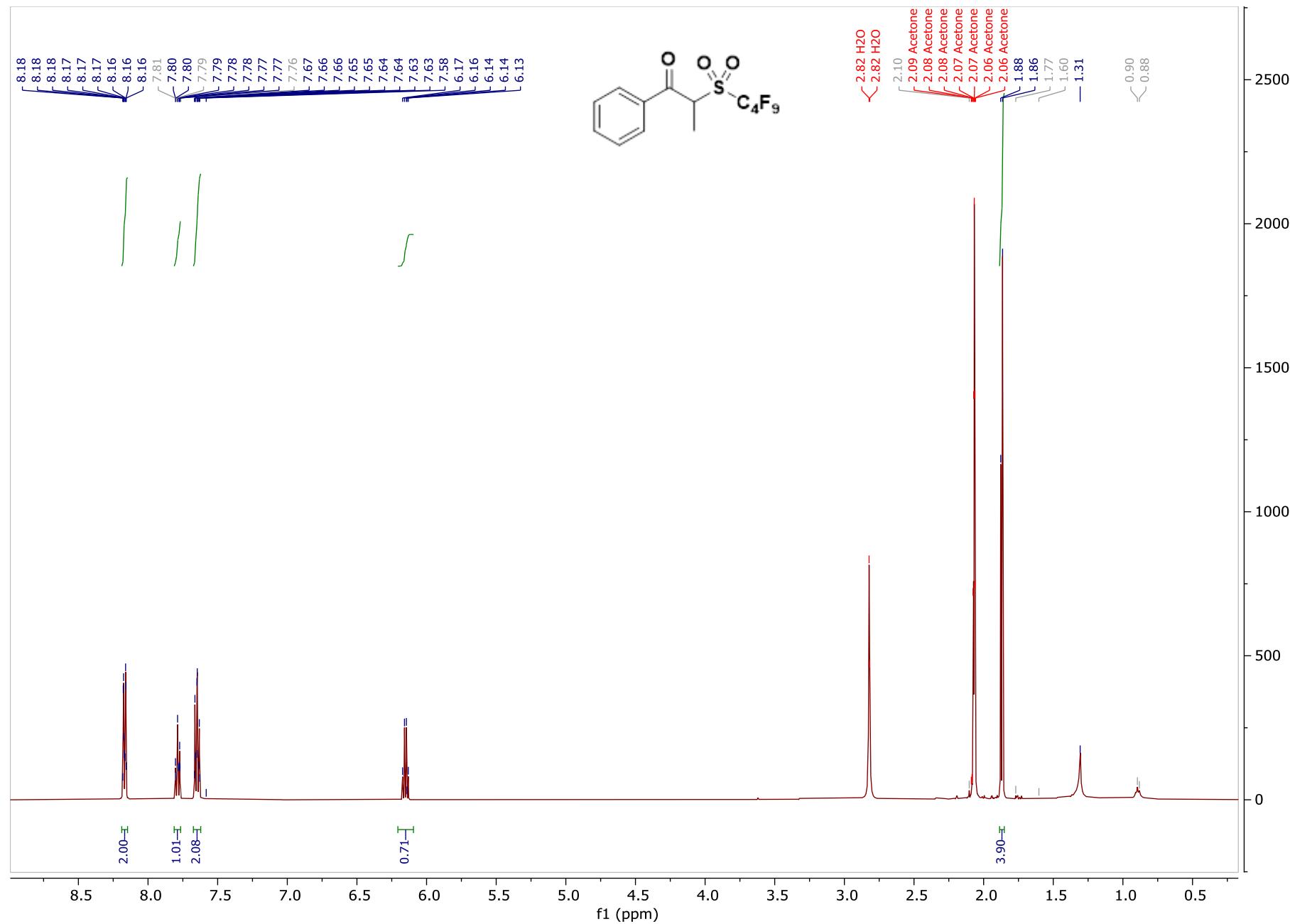
¹⁹F NMR of **1b**, CDCl₃, 471 MHz



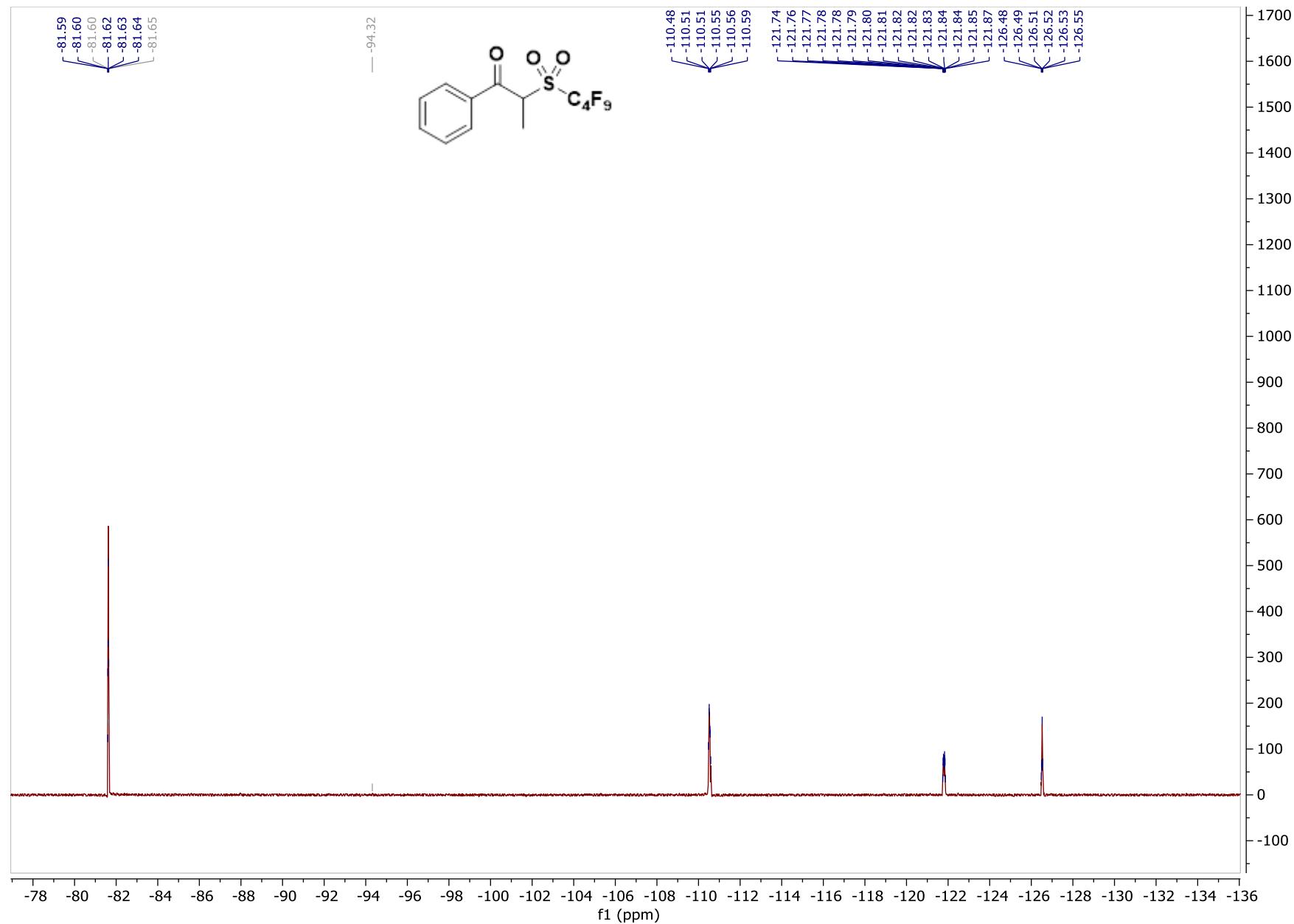
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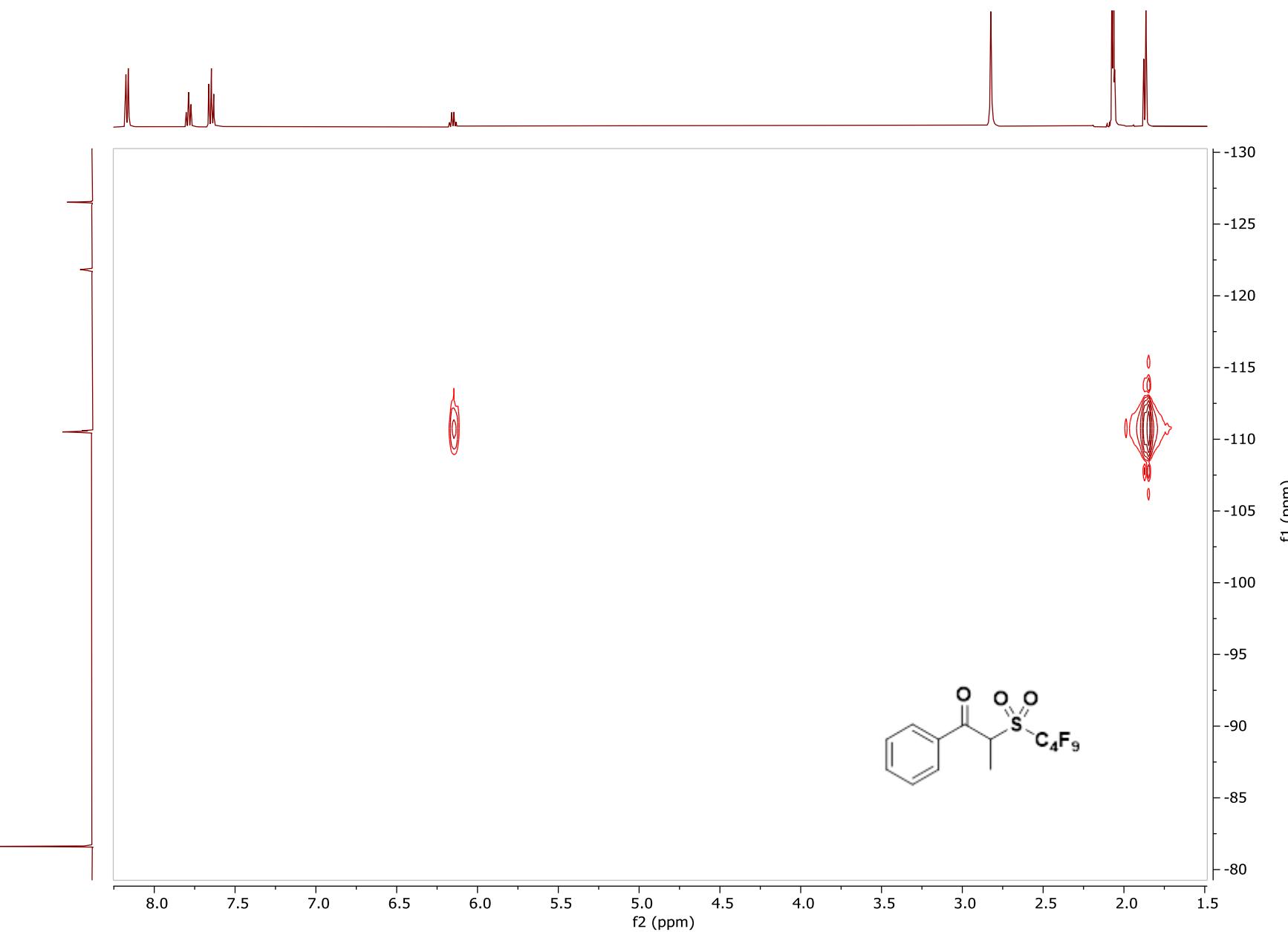
¹H NMR of **1a**, acetone-*d*₆, 500 MHz



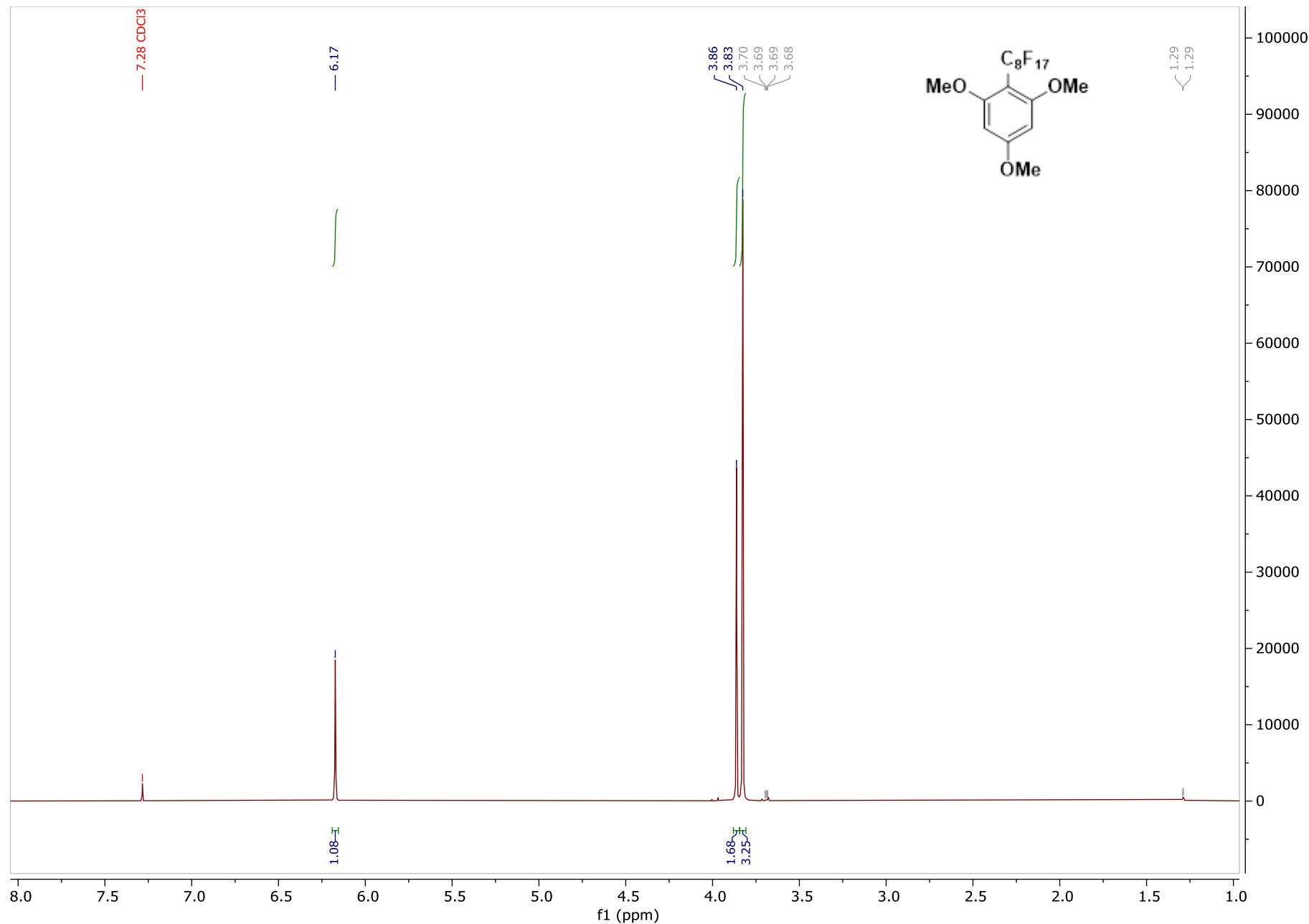
¹⁹F NMR of **1a**, acetone-*d*₆, 471 MHz



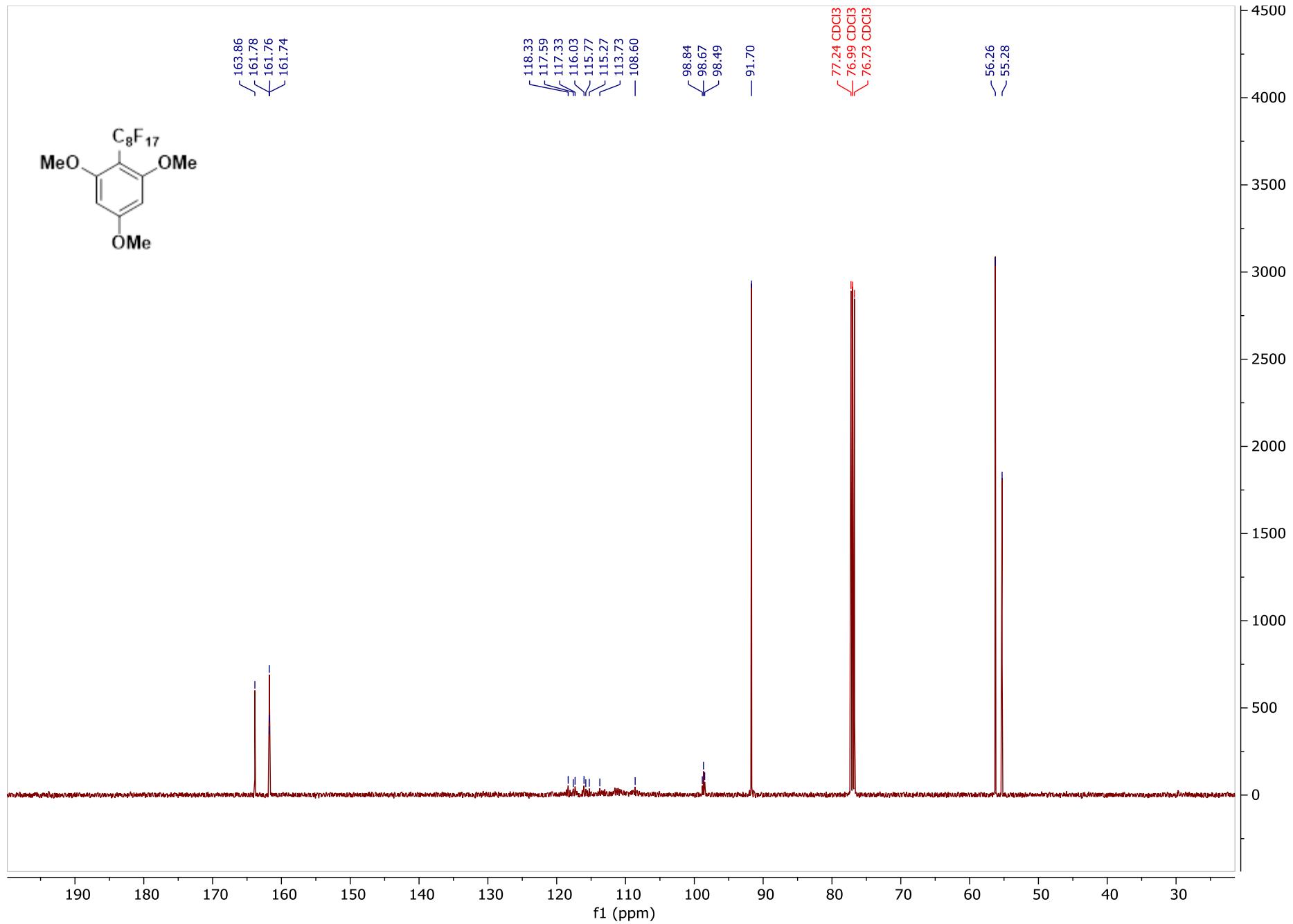
^1H - ^{19}F HMBC of **1a**



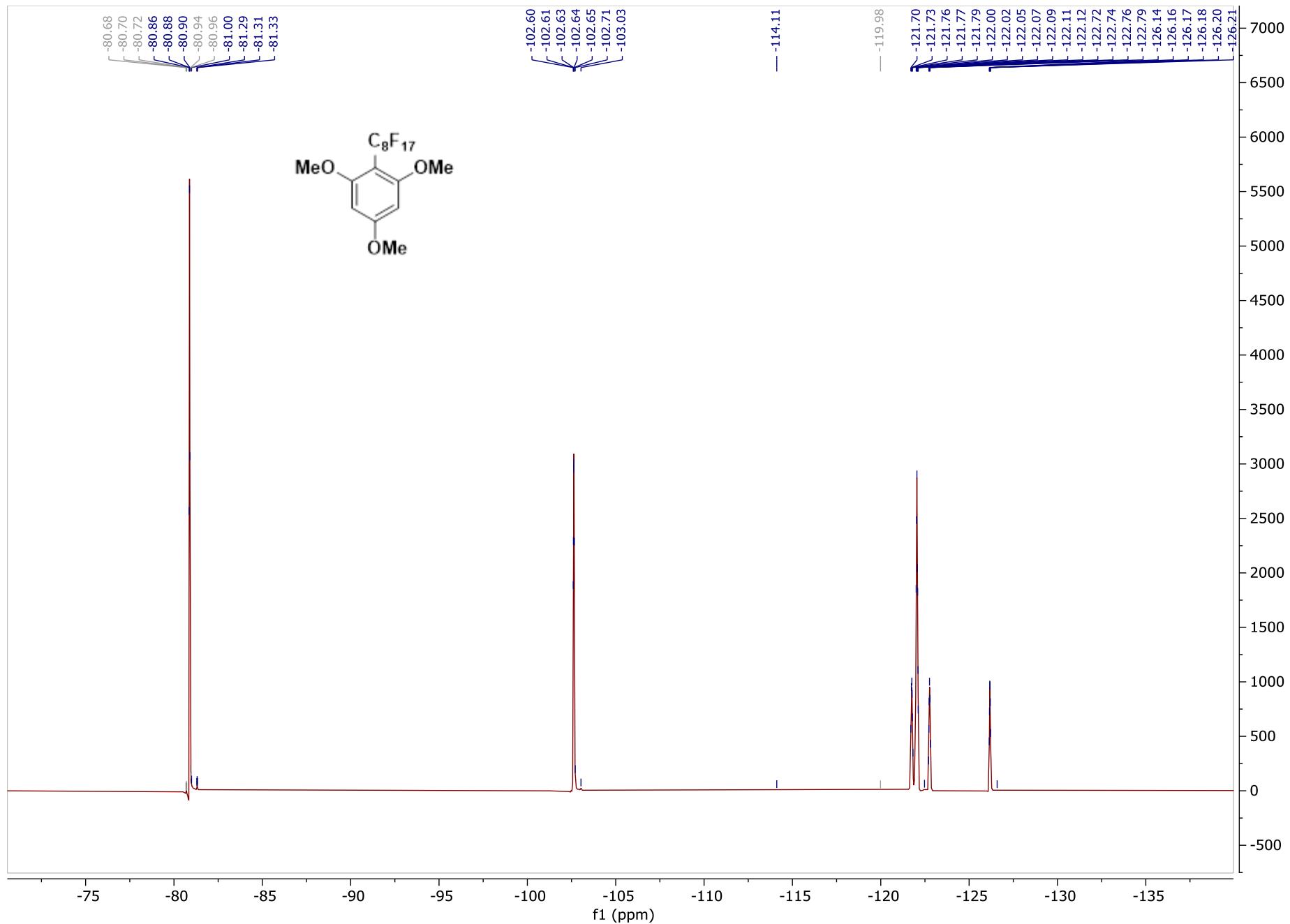
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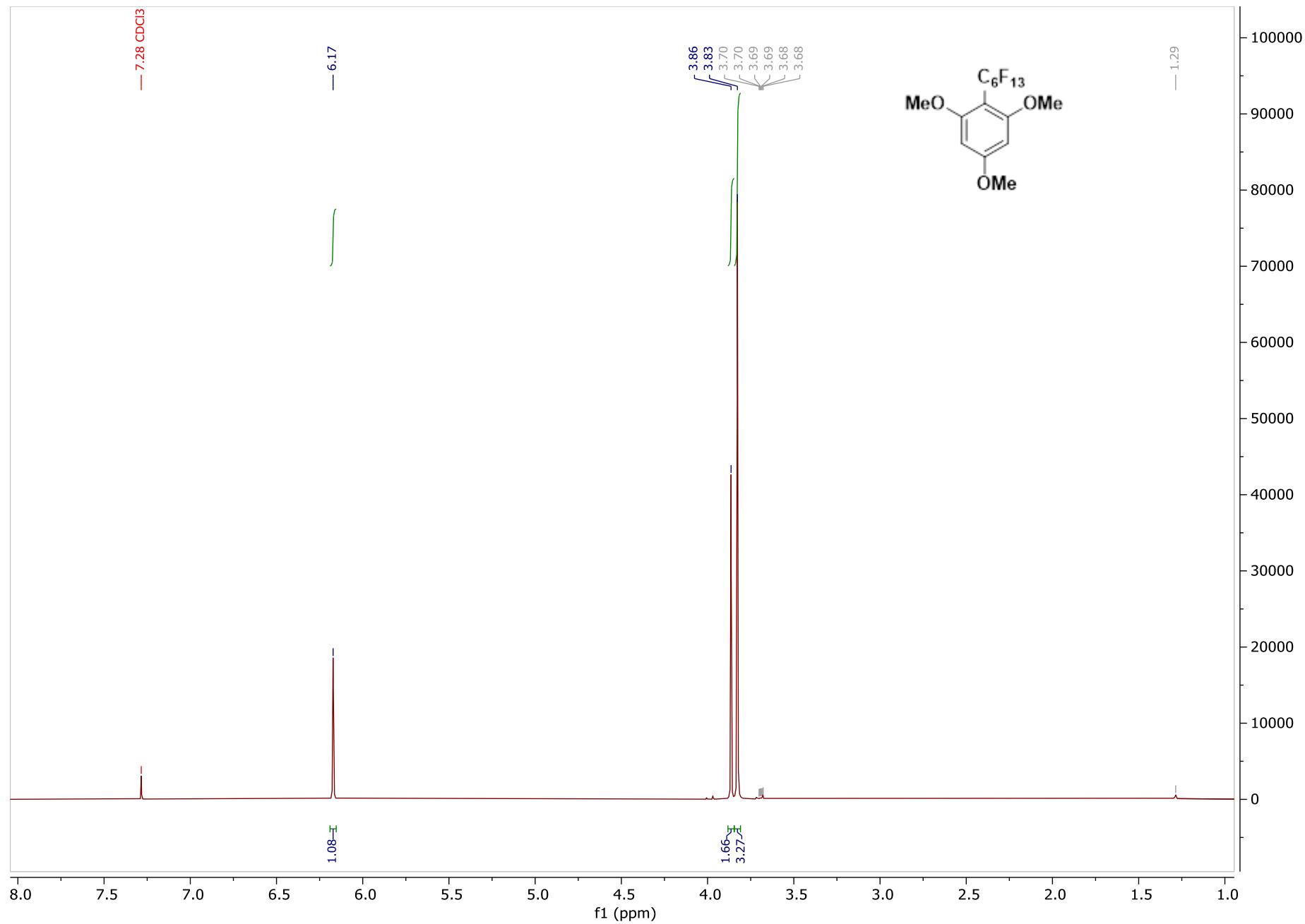
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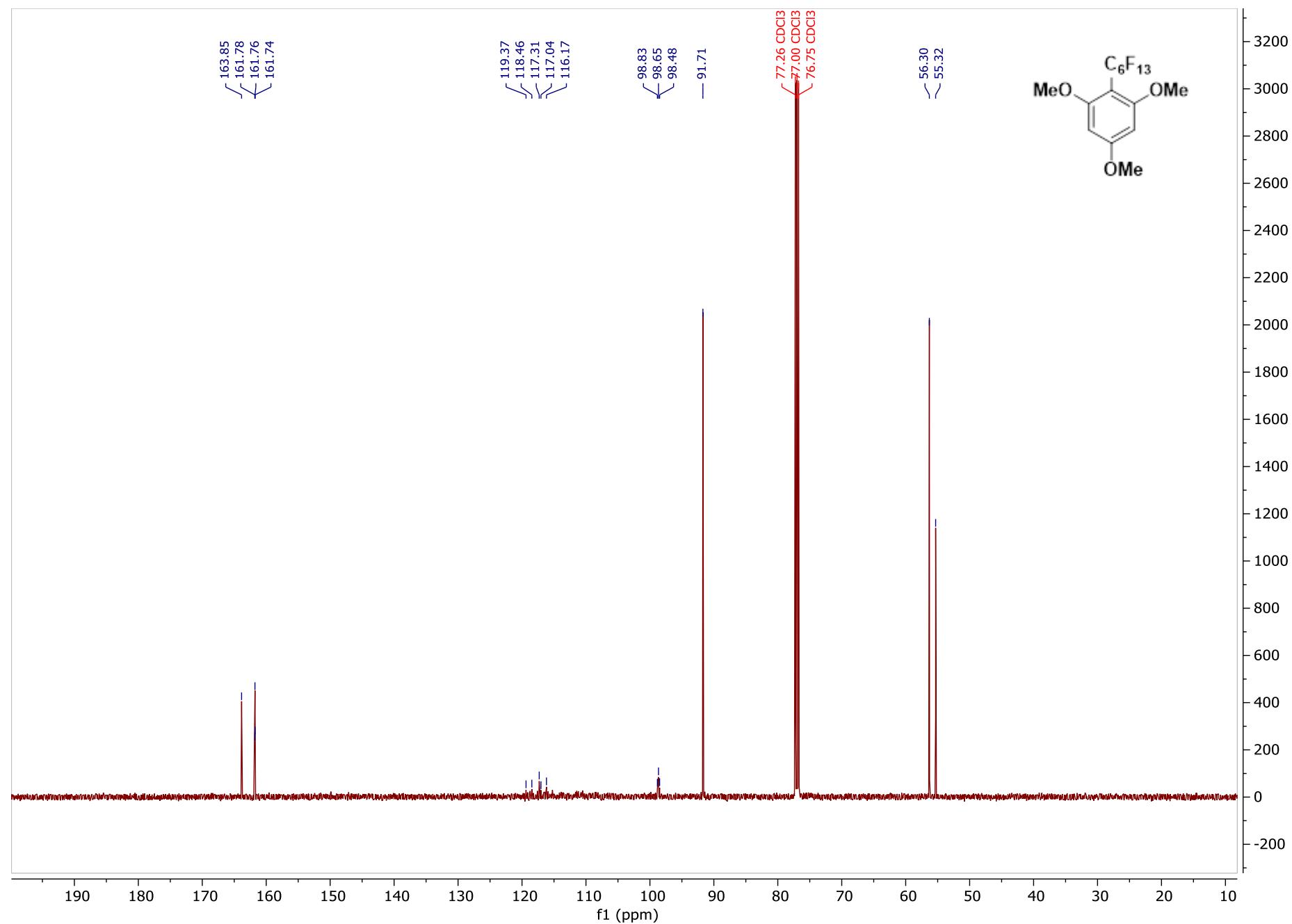
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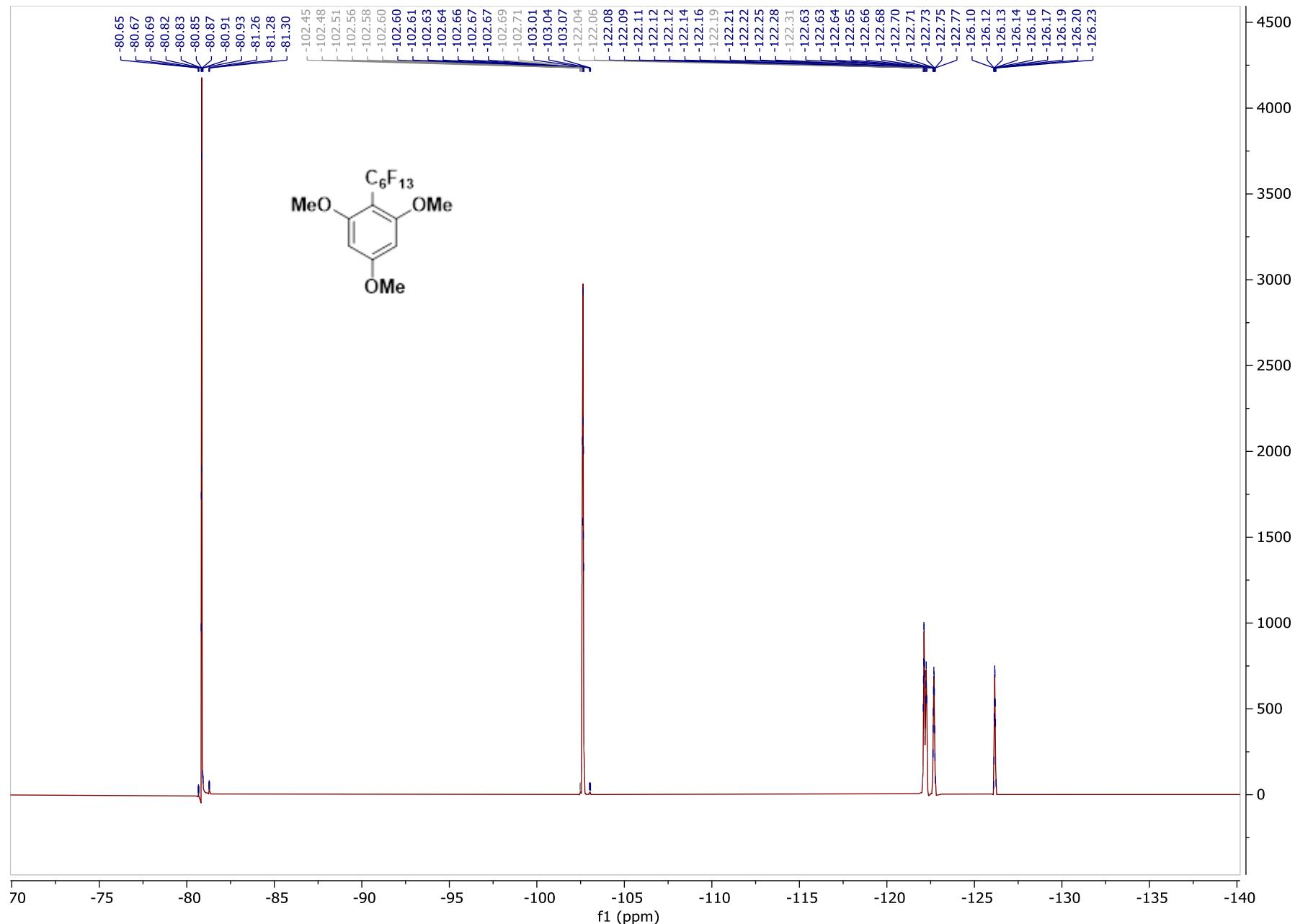
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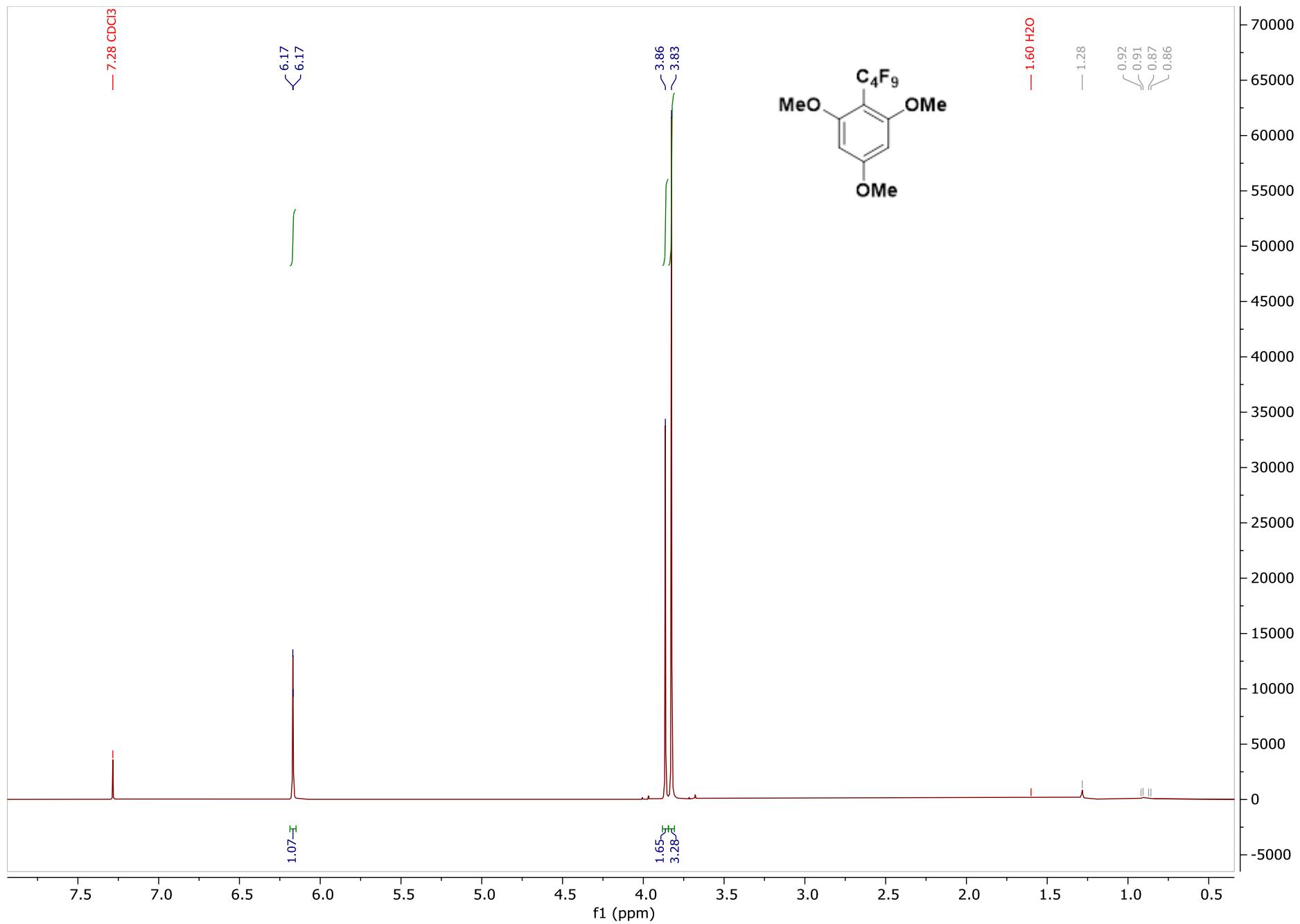
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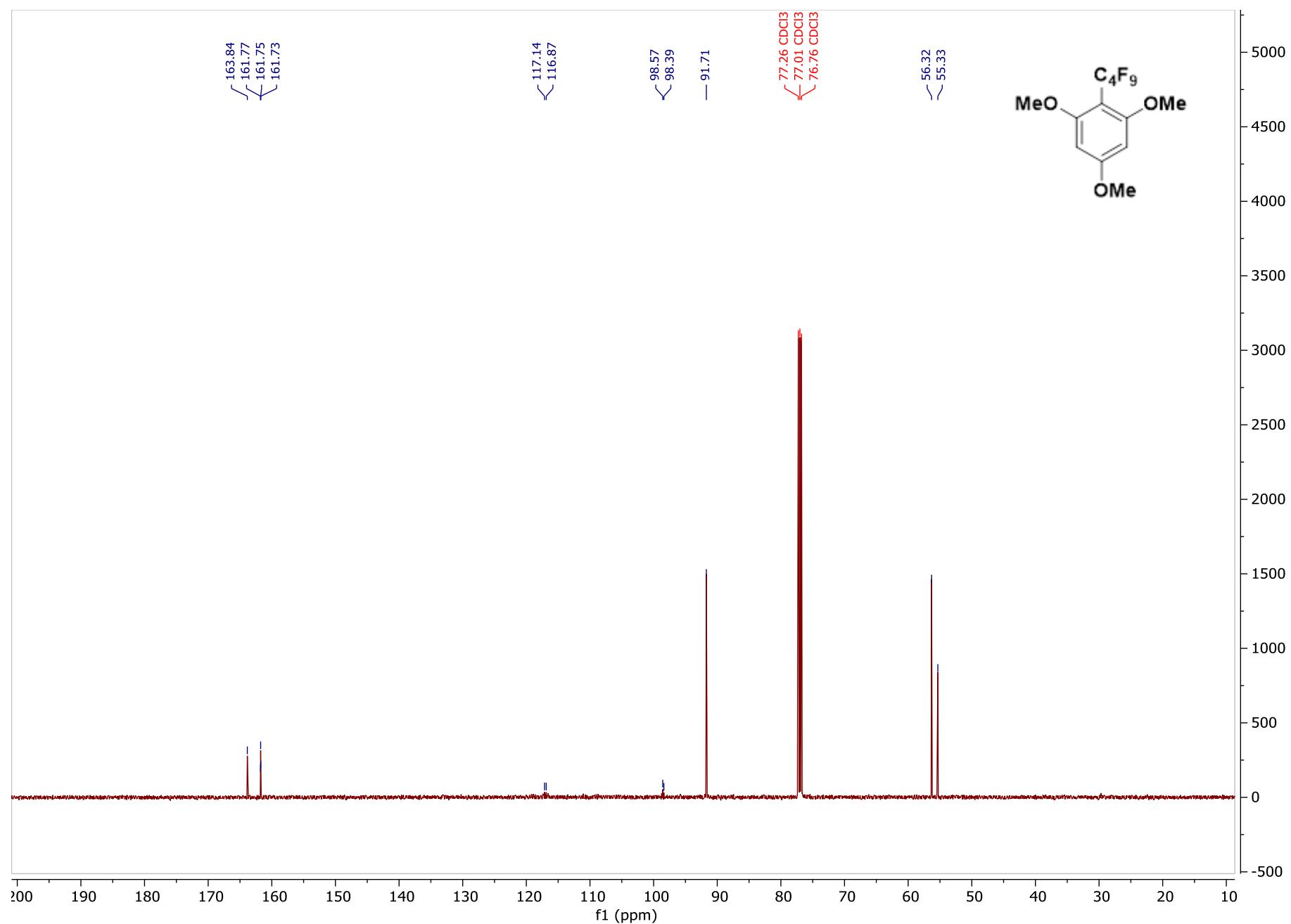
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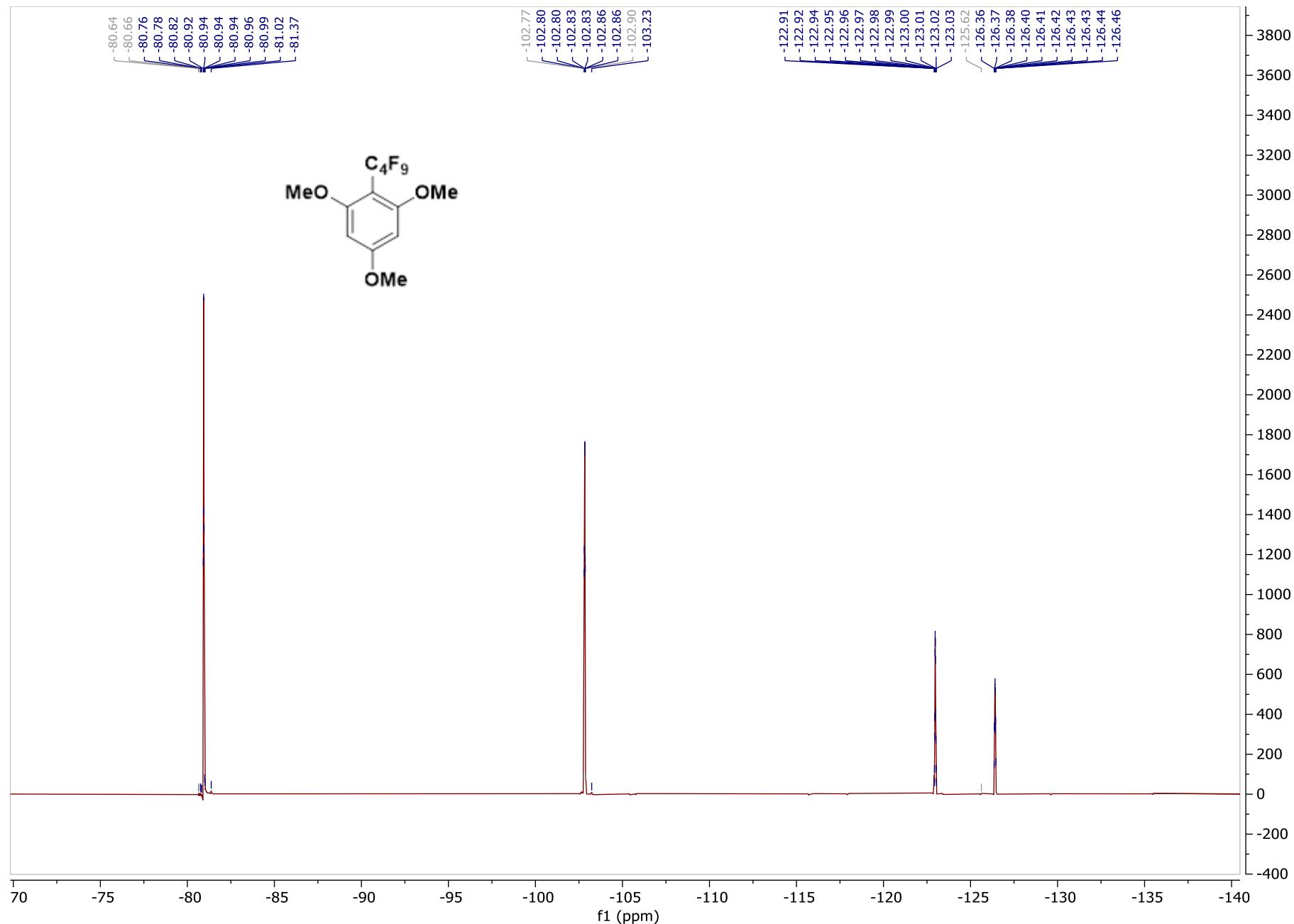
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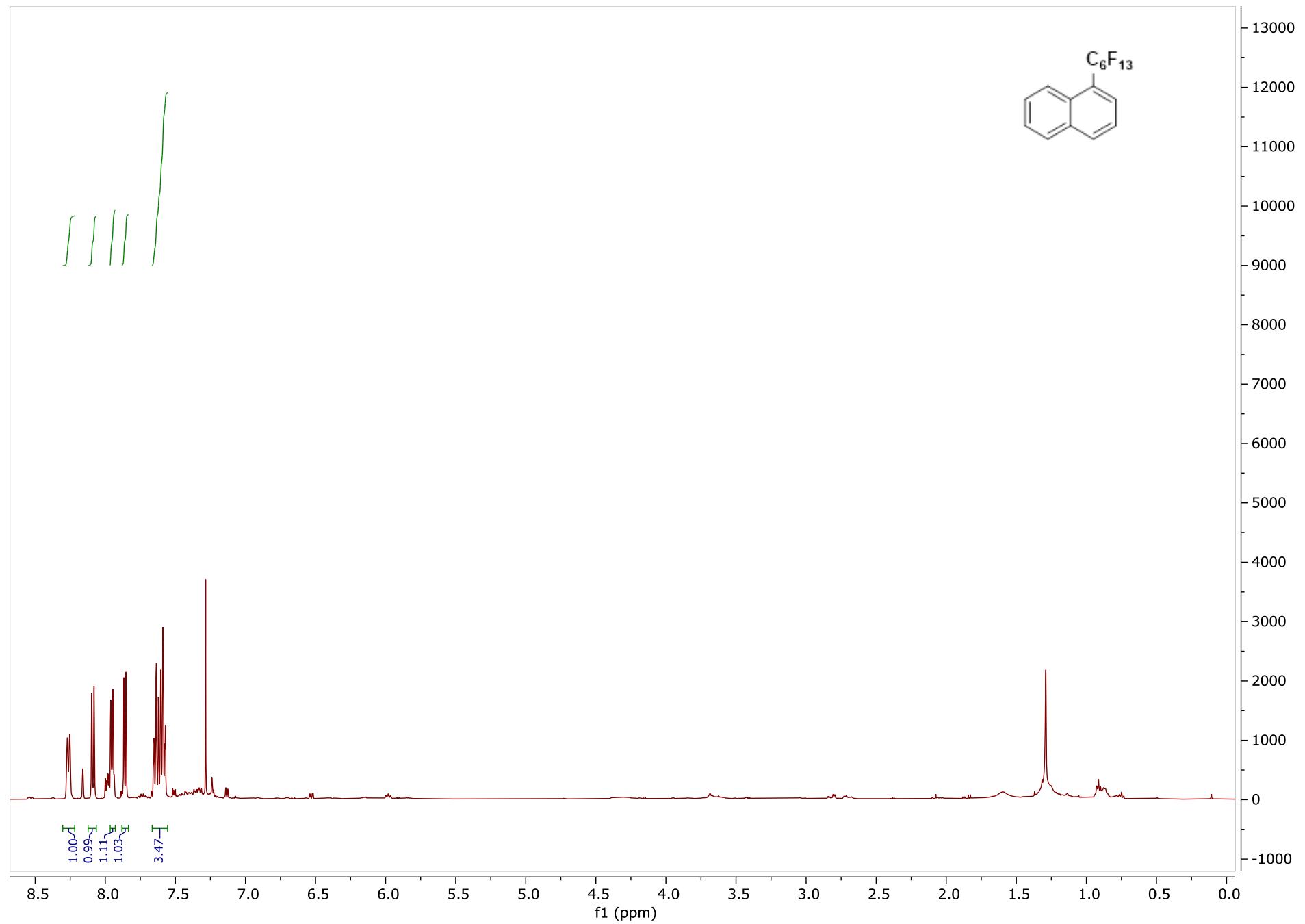
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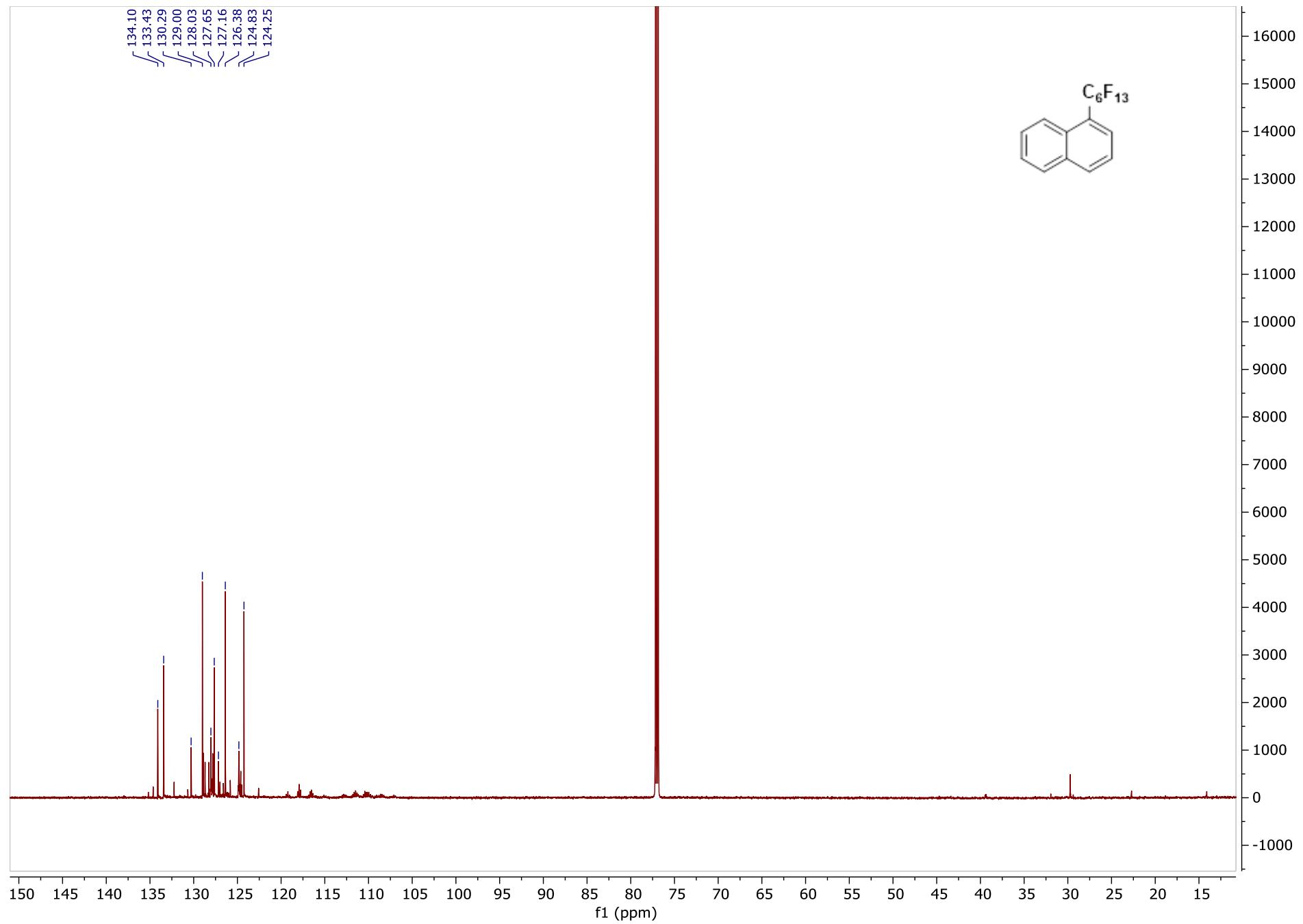
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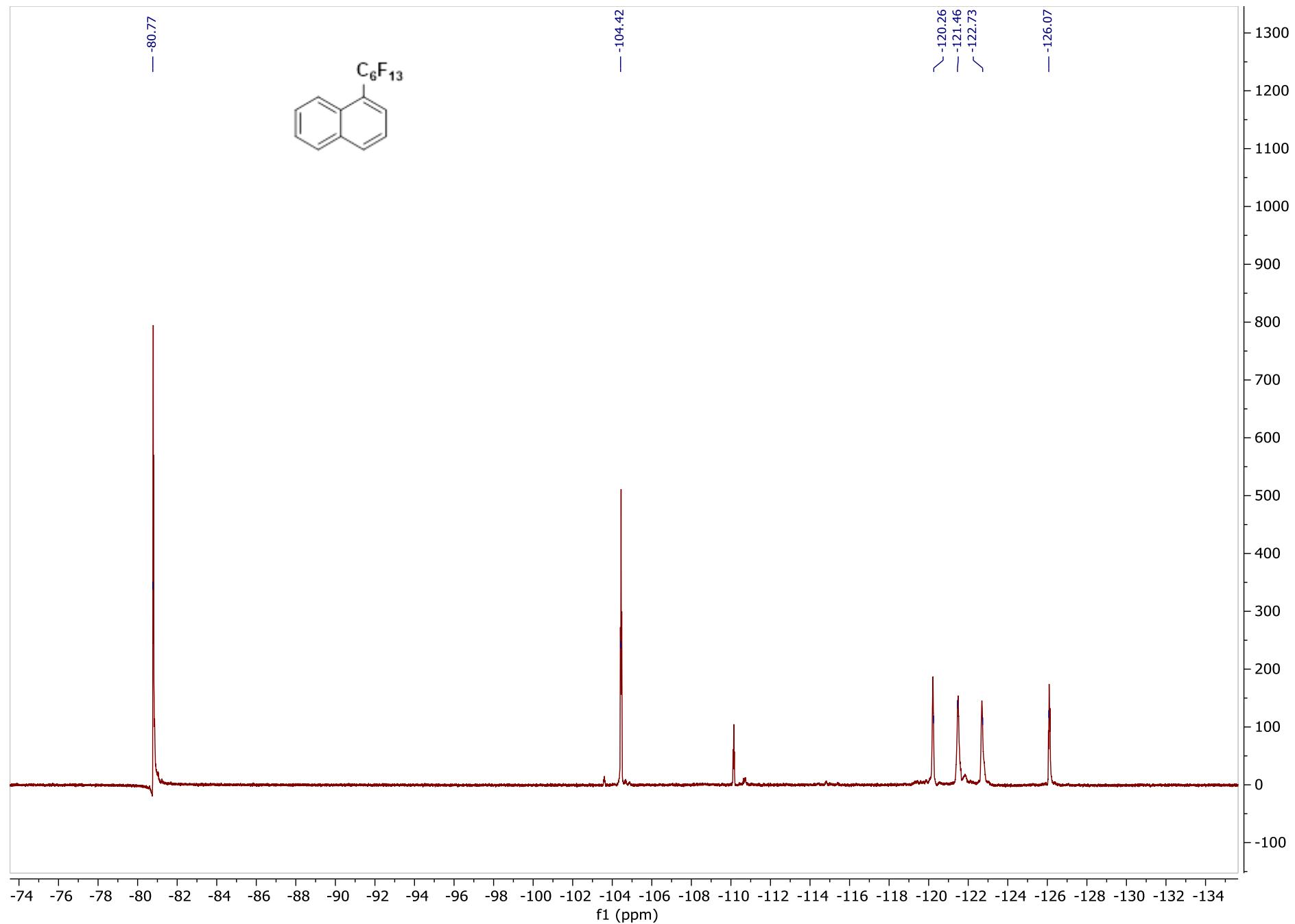
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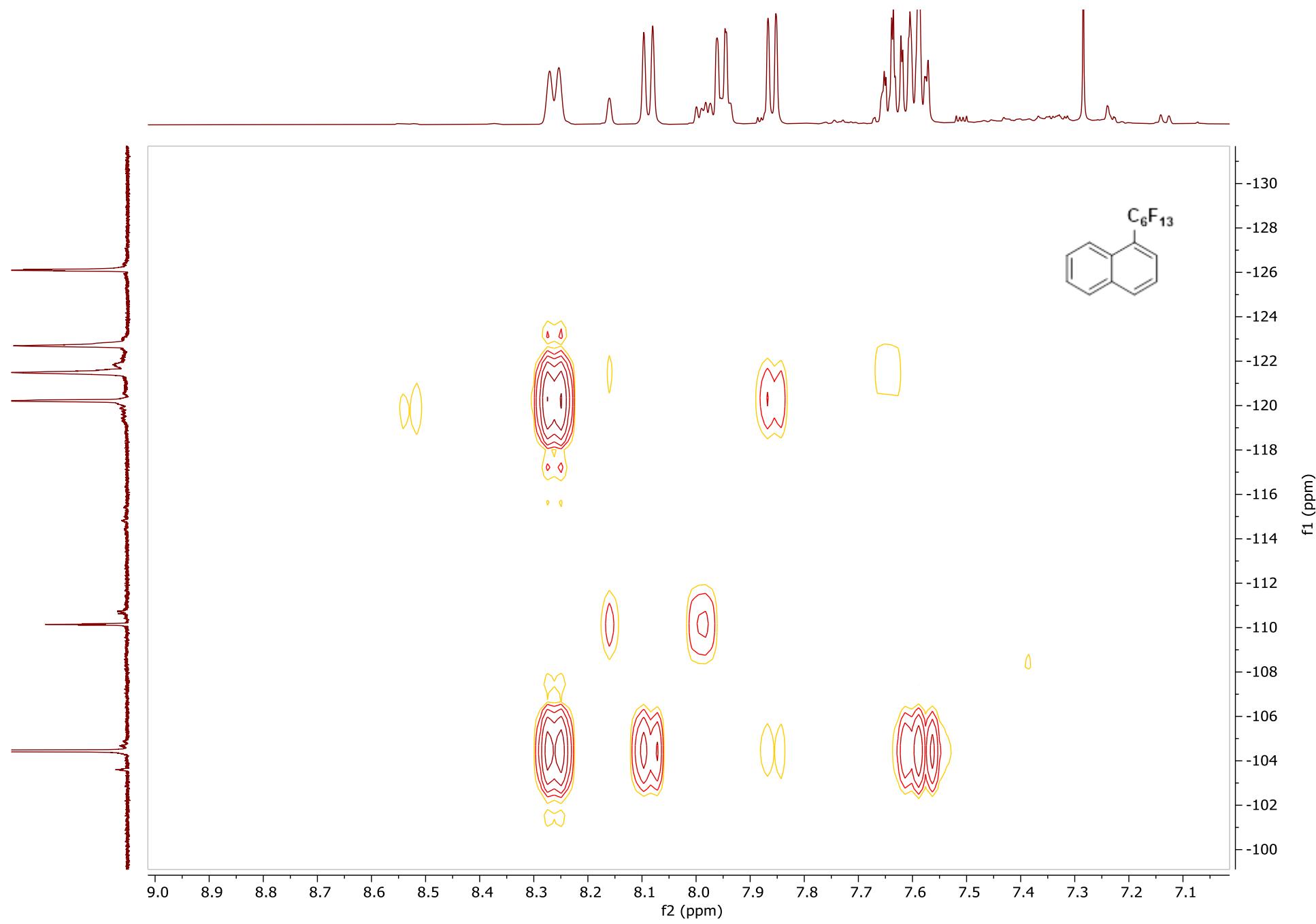
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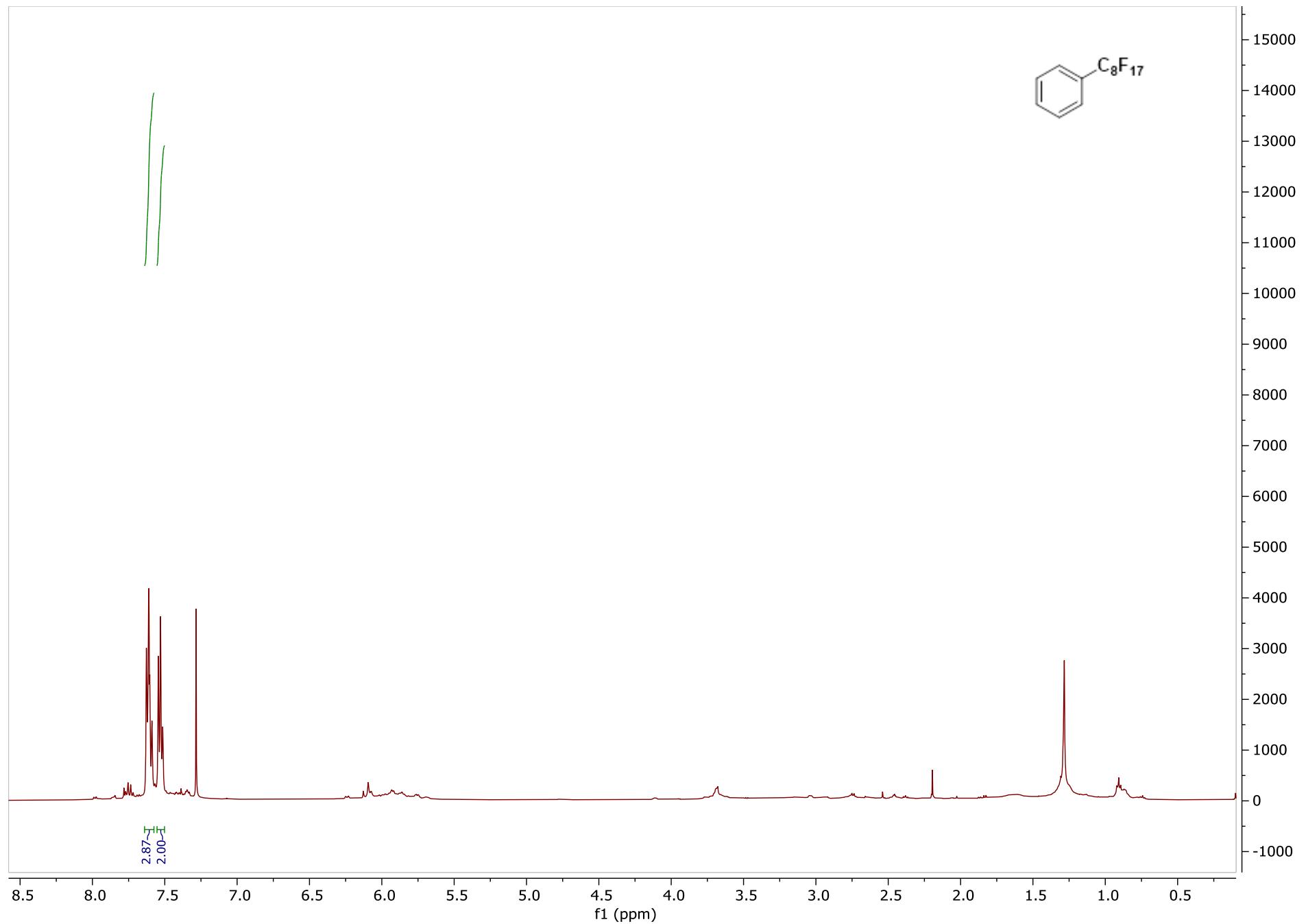
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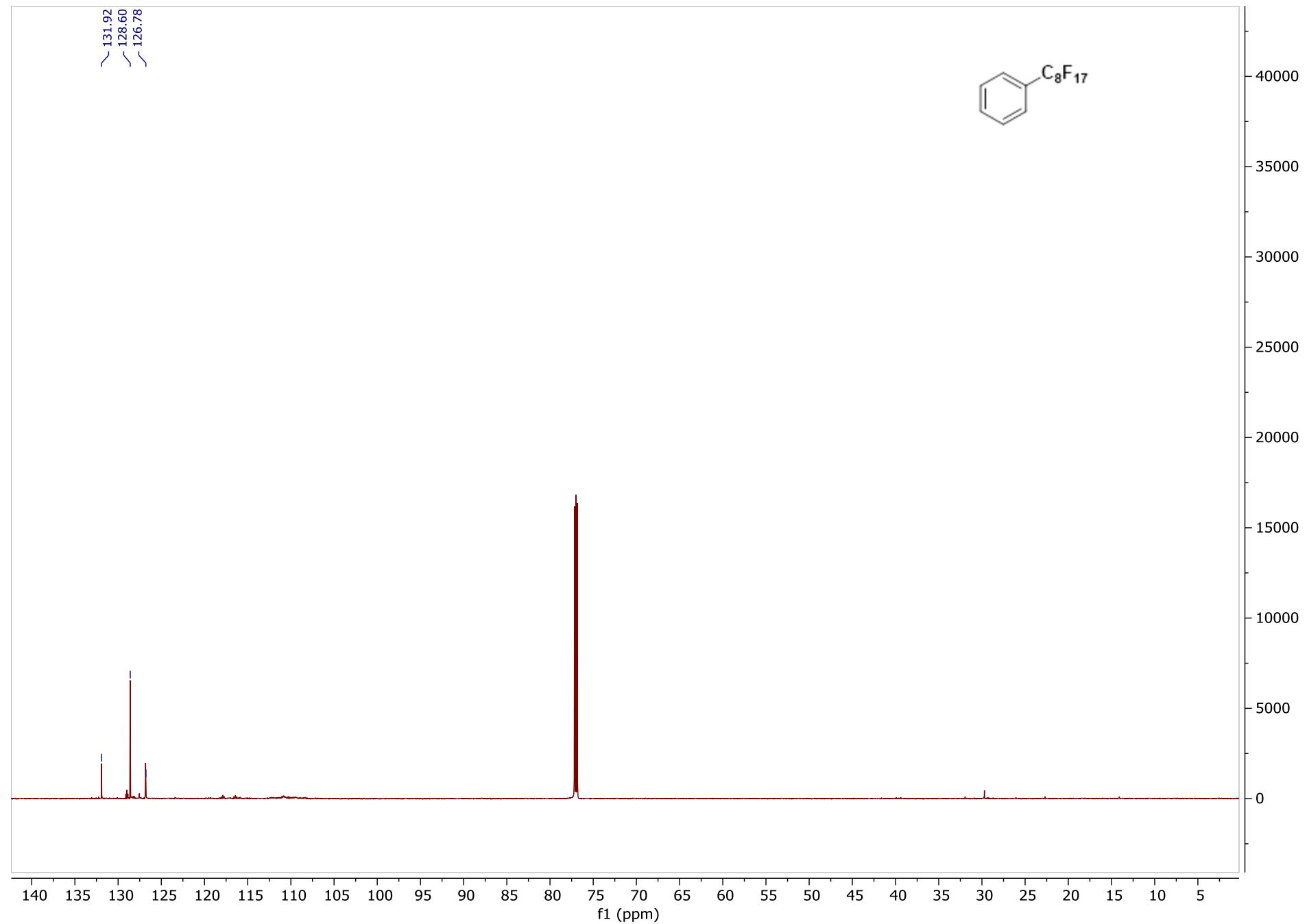
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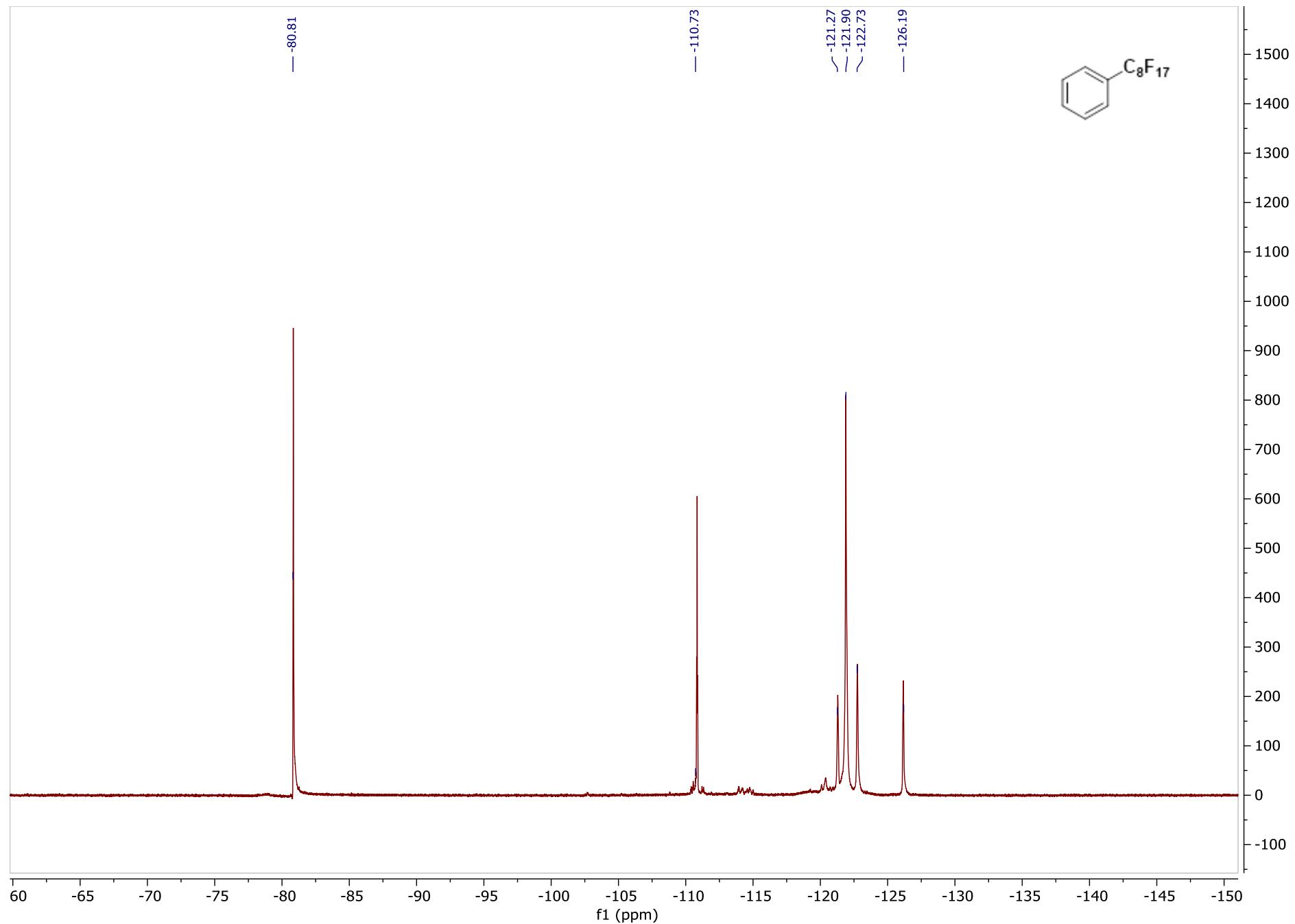
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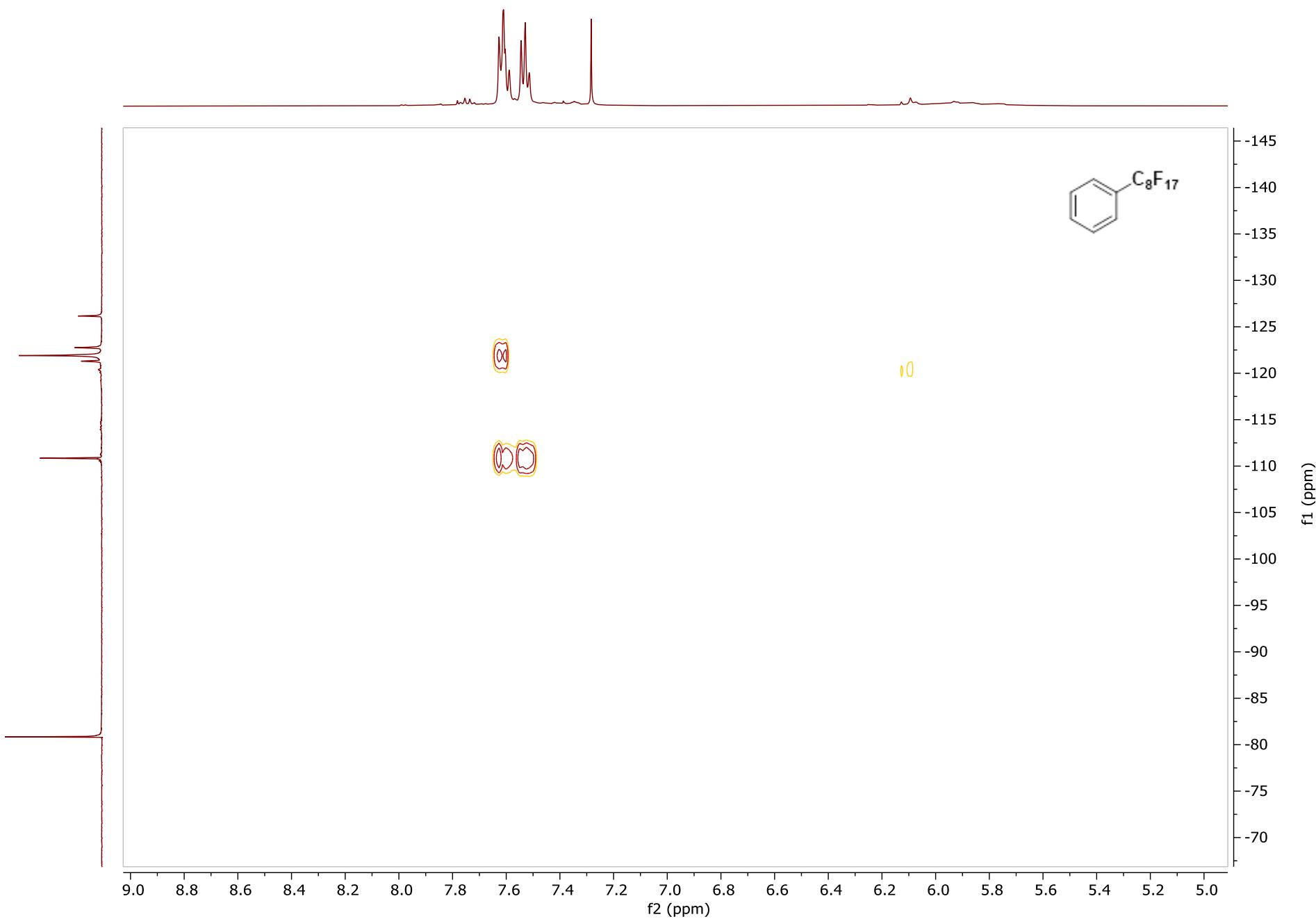
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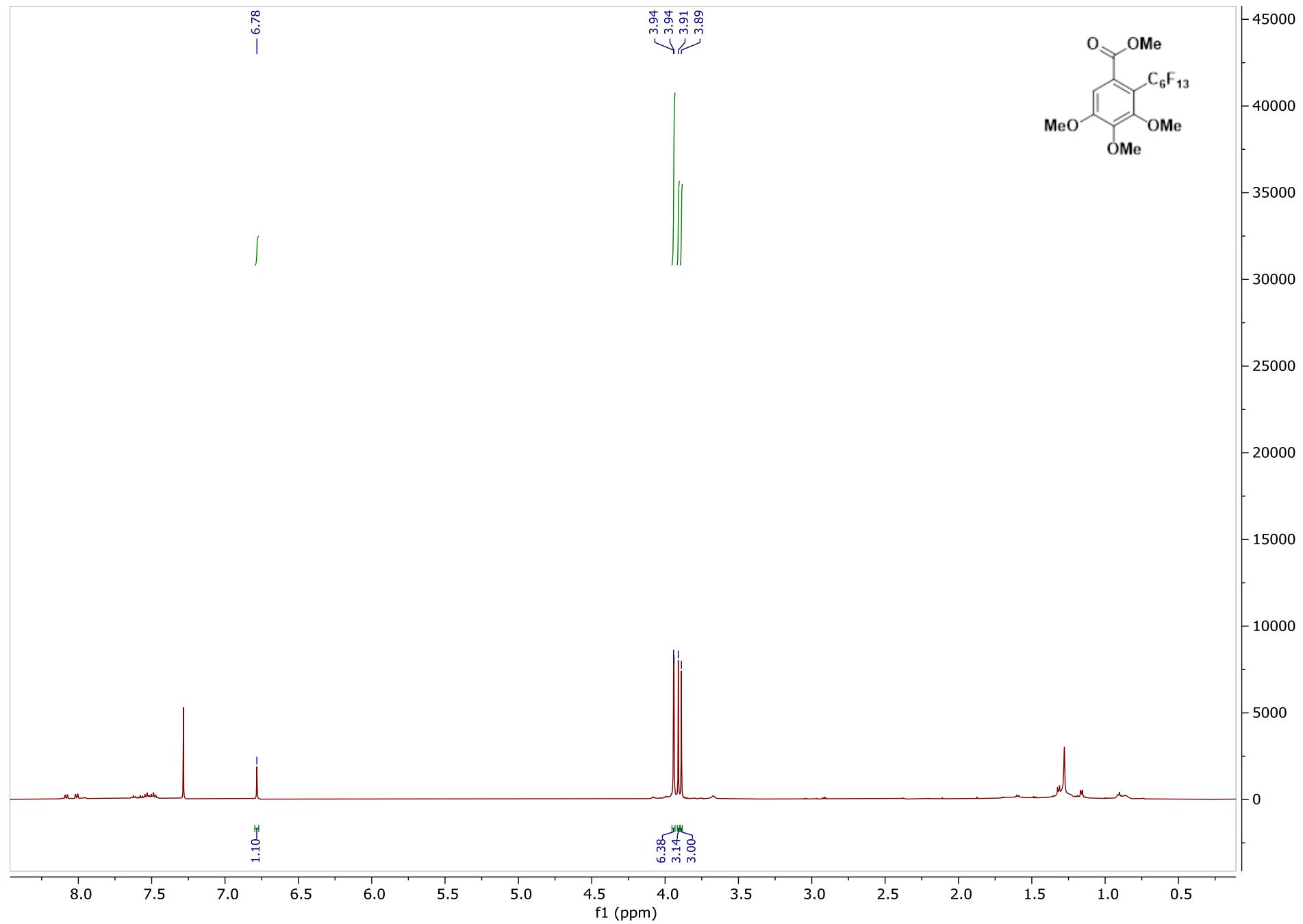
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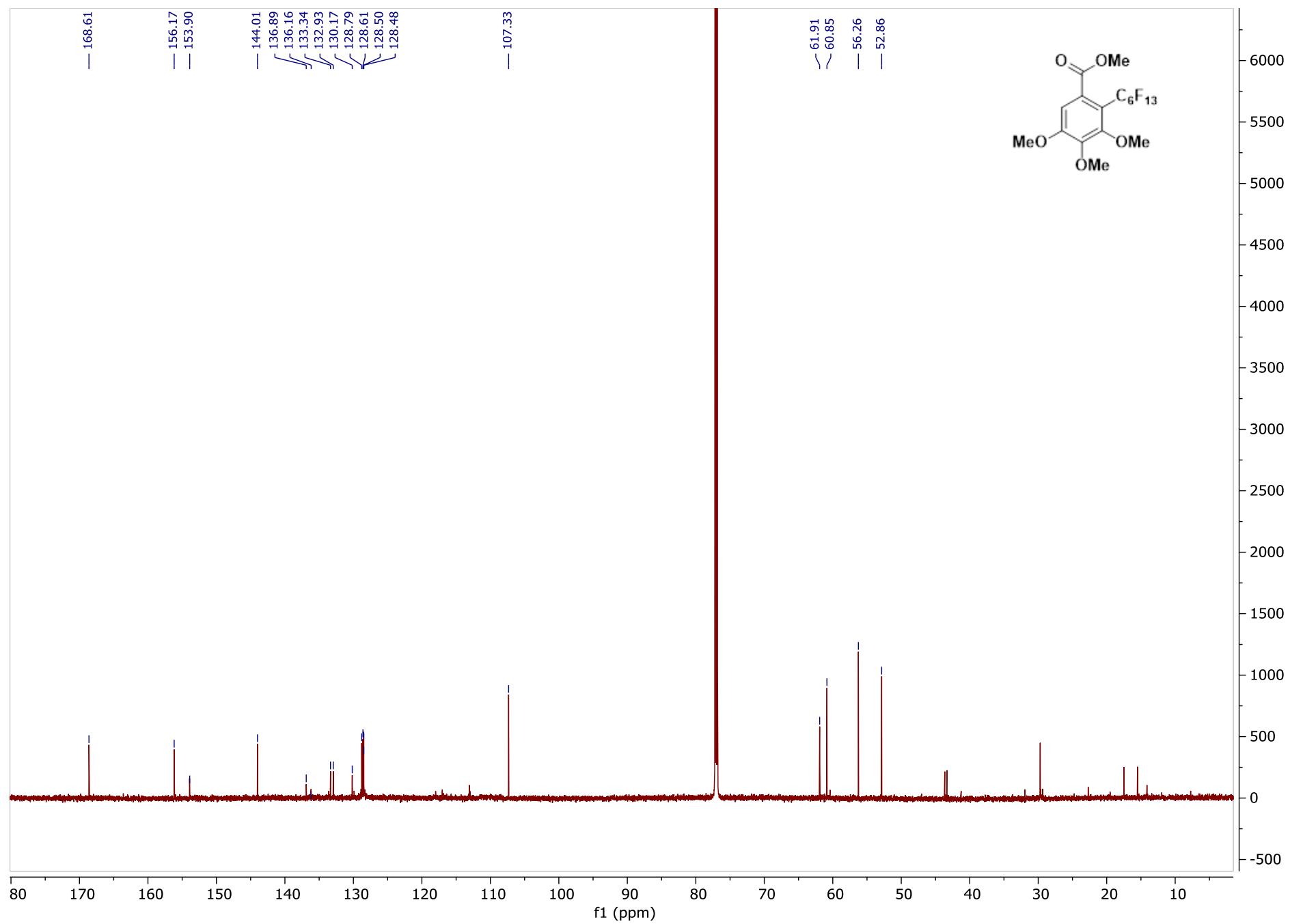
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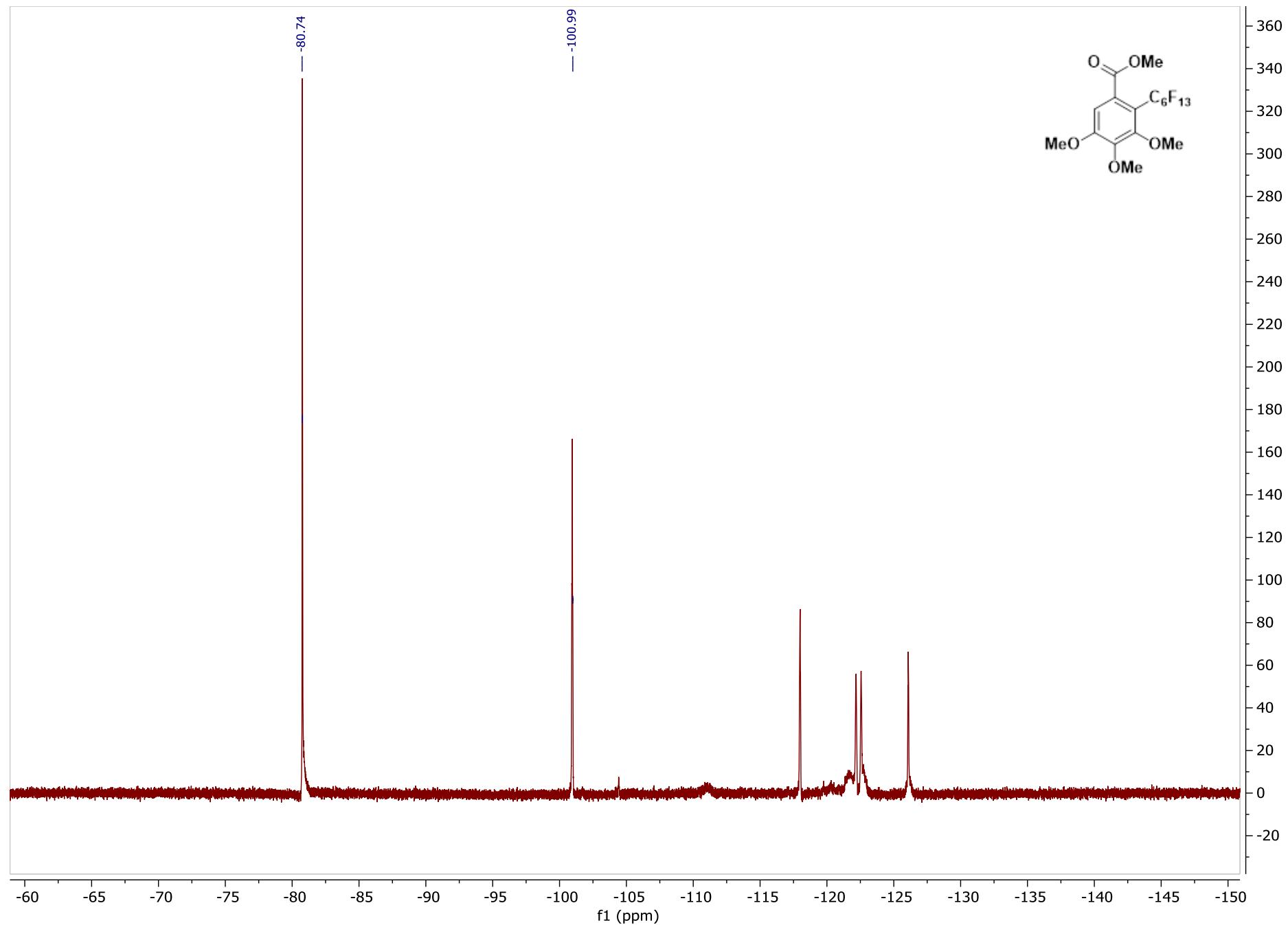
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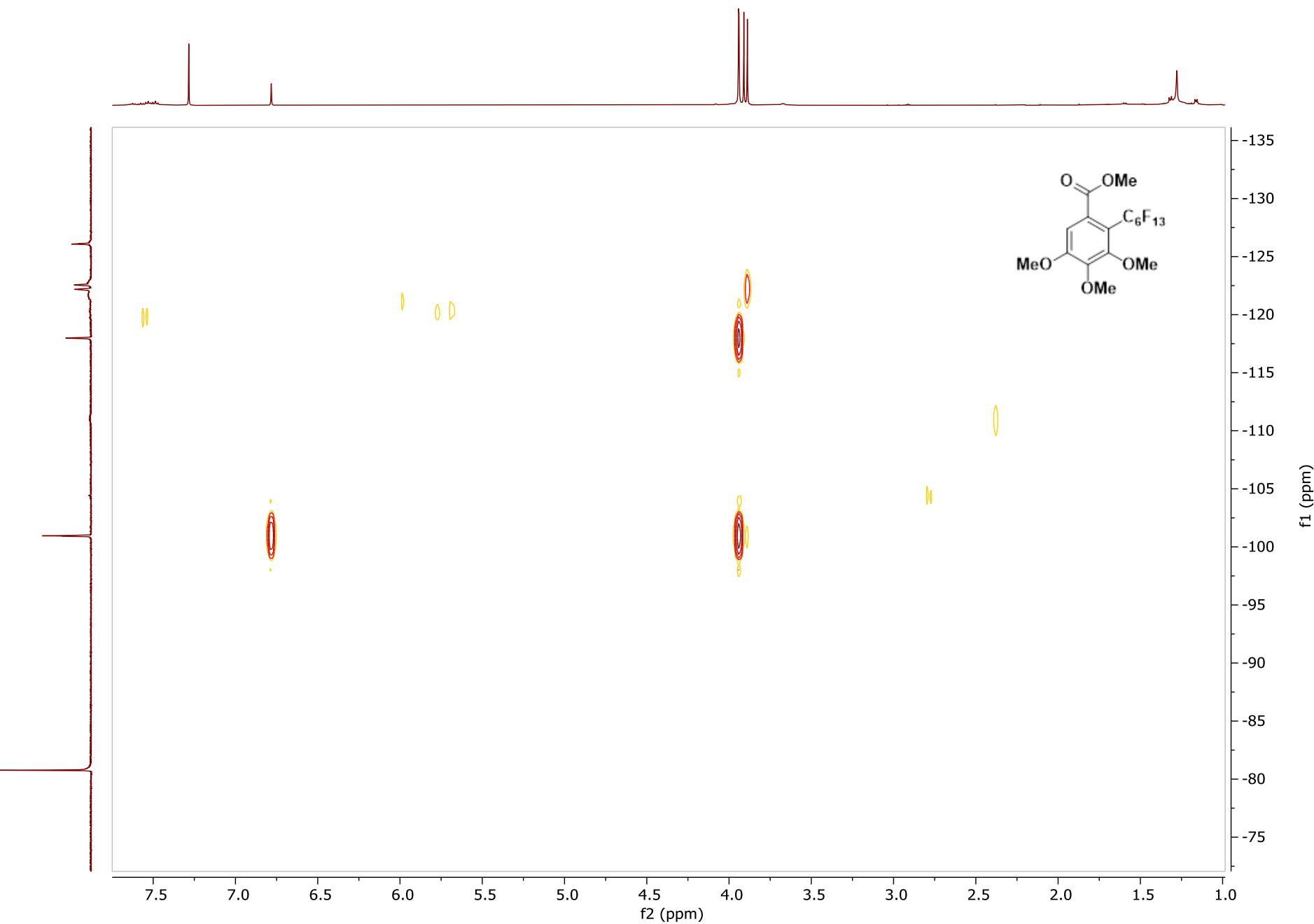
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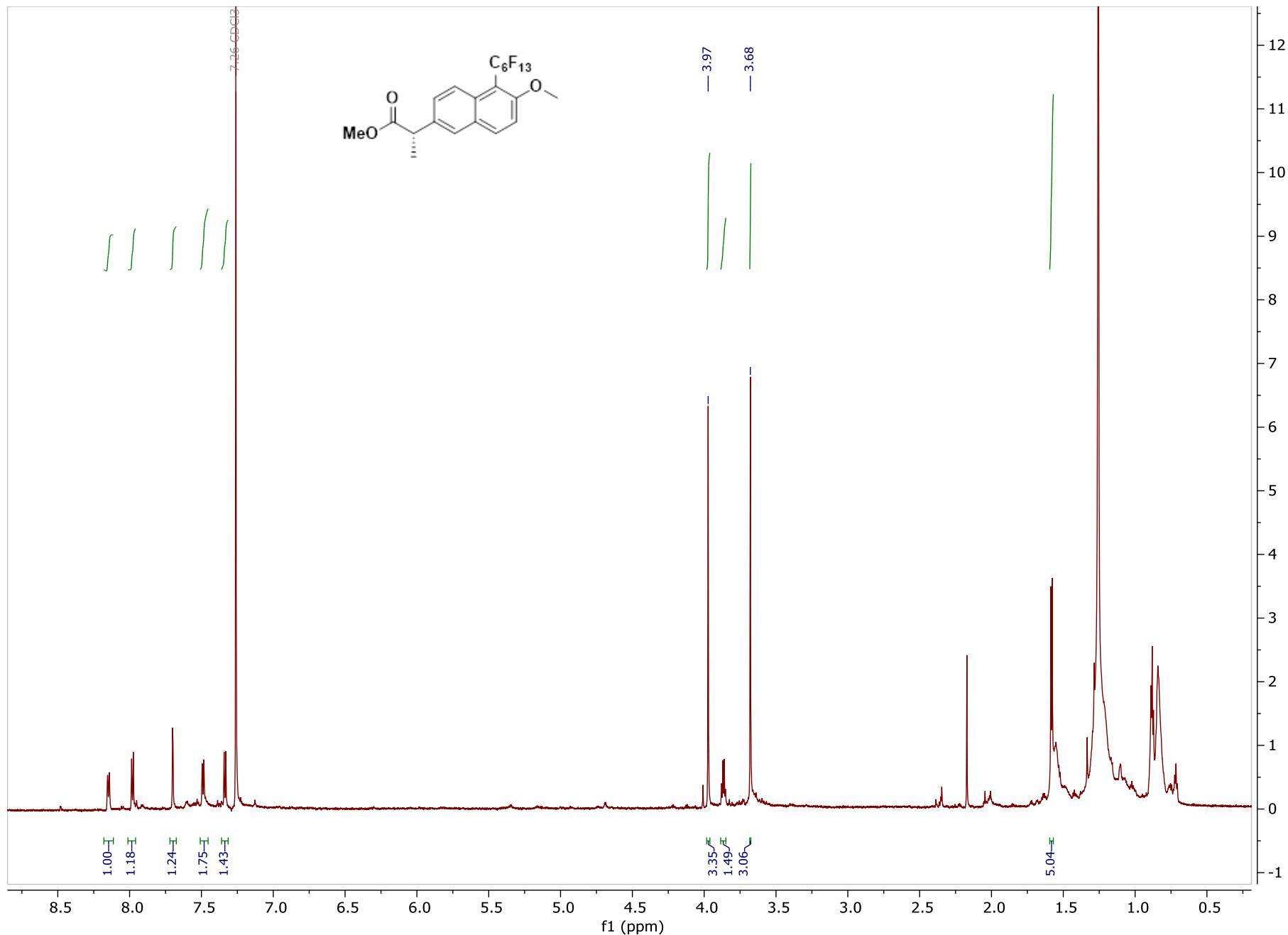
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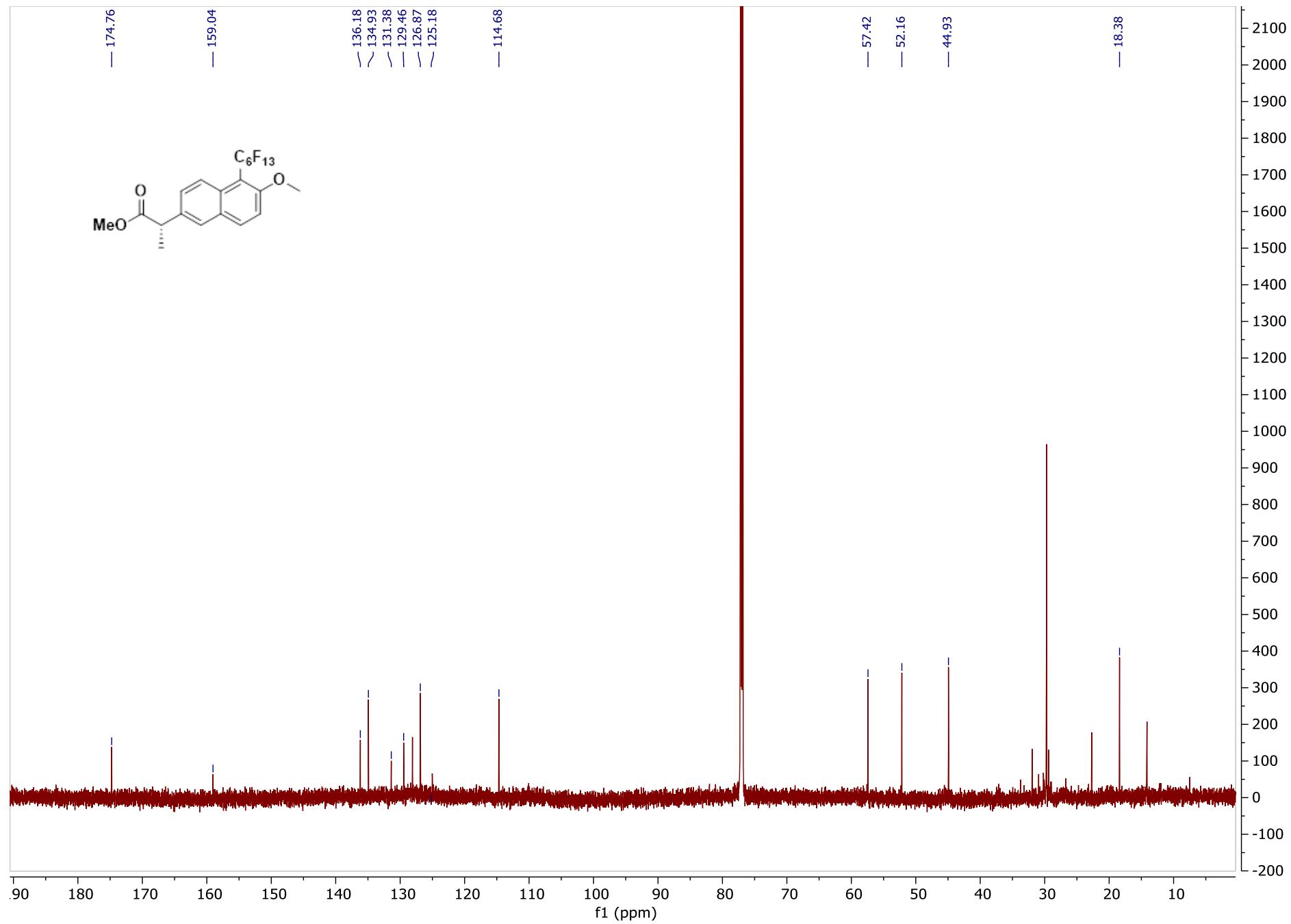
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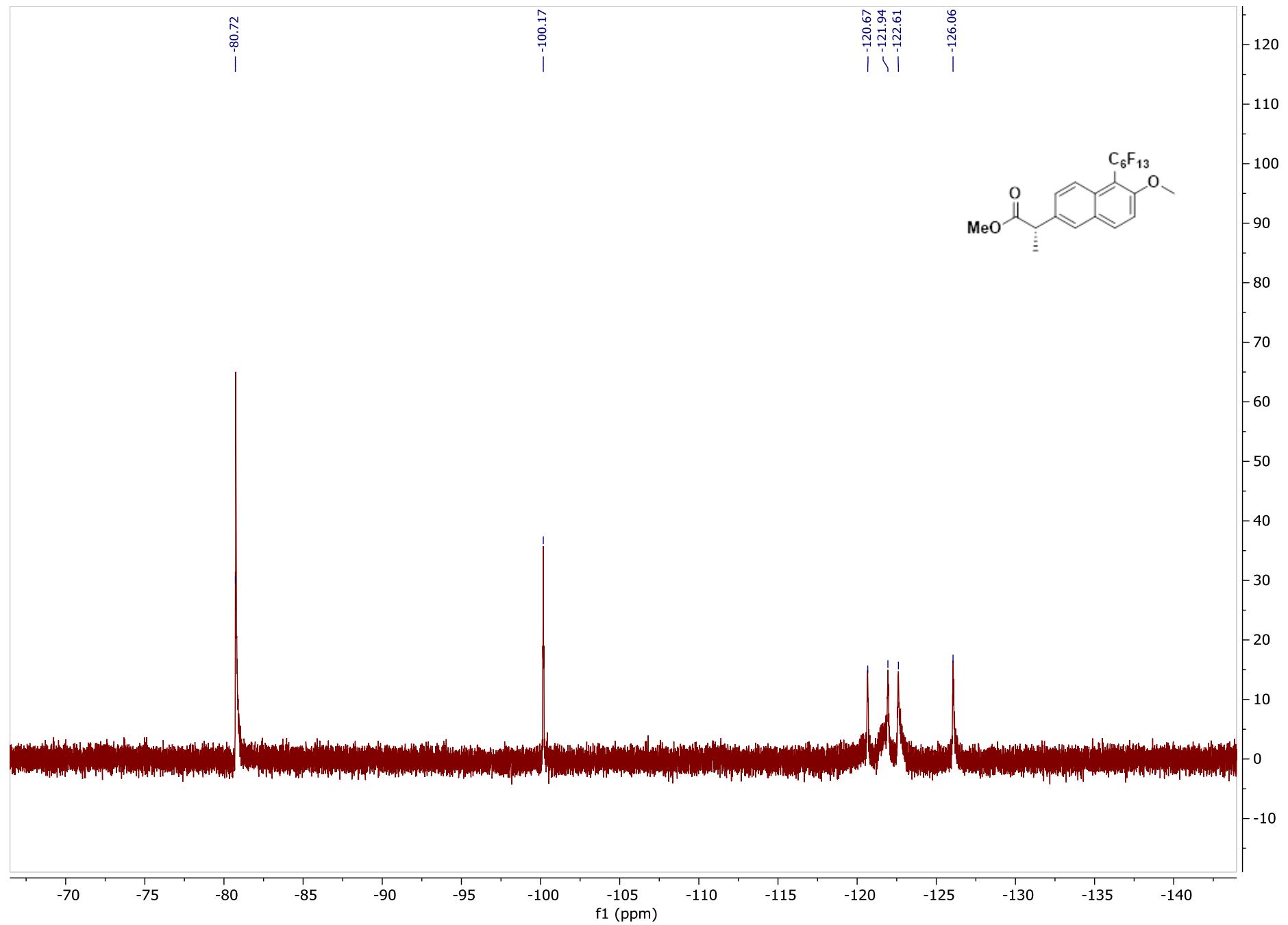
¹H NMR of **14b**, CDCl₃, 500 MHz



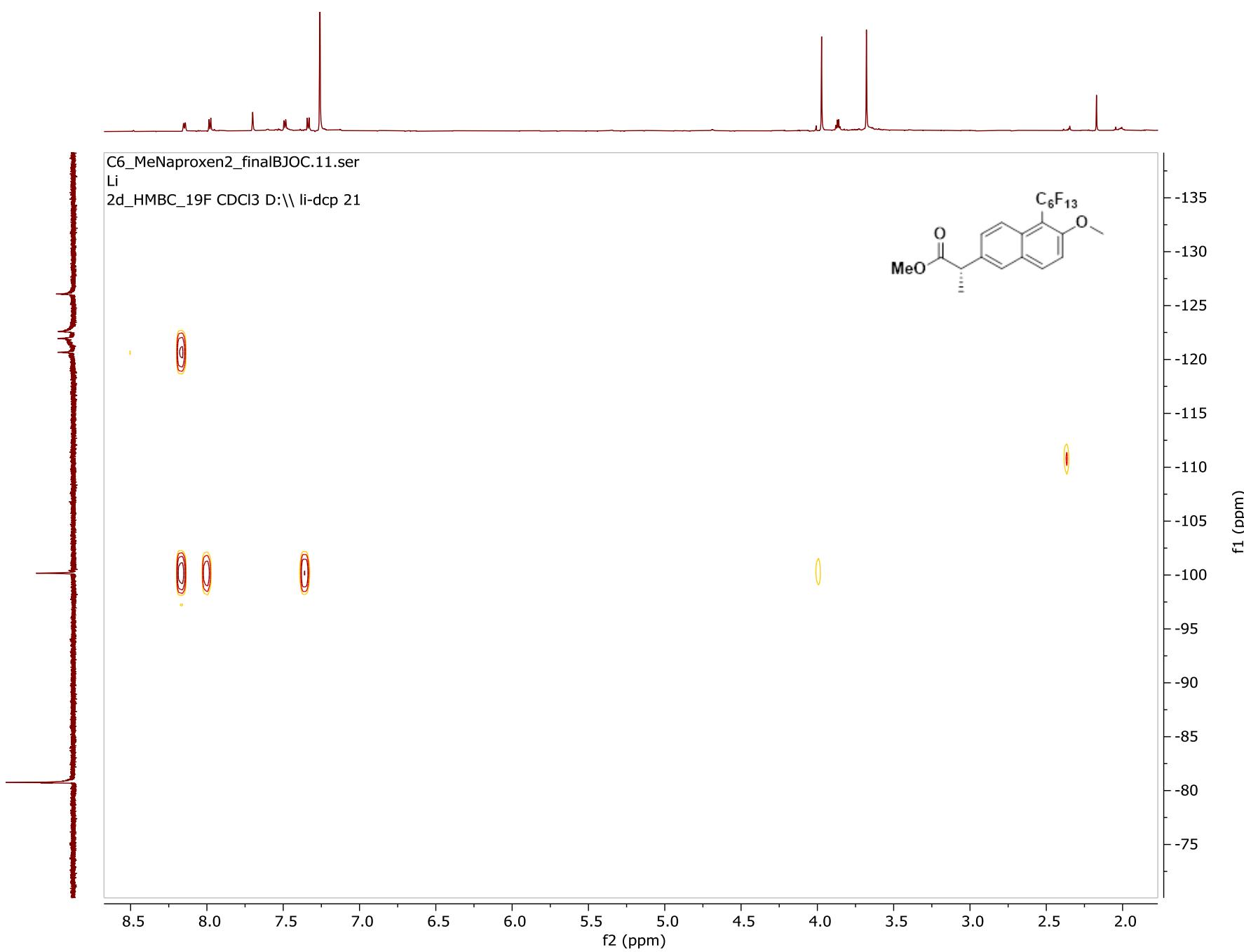
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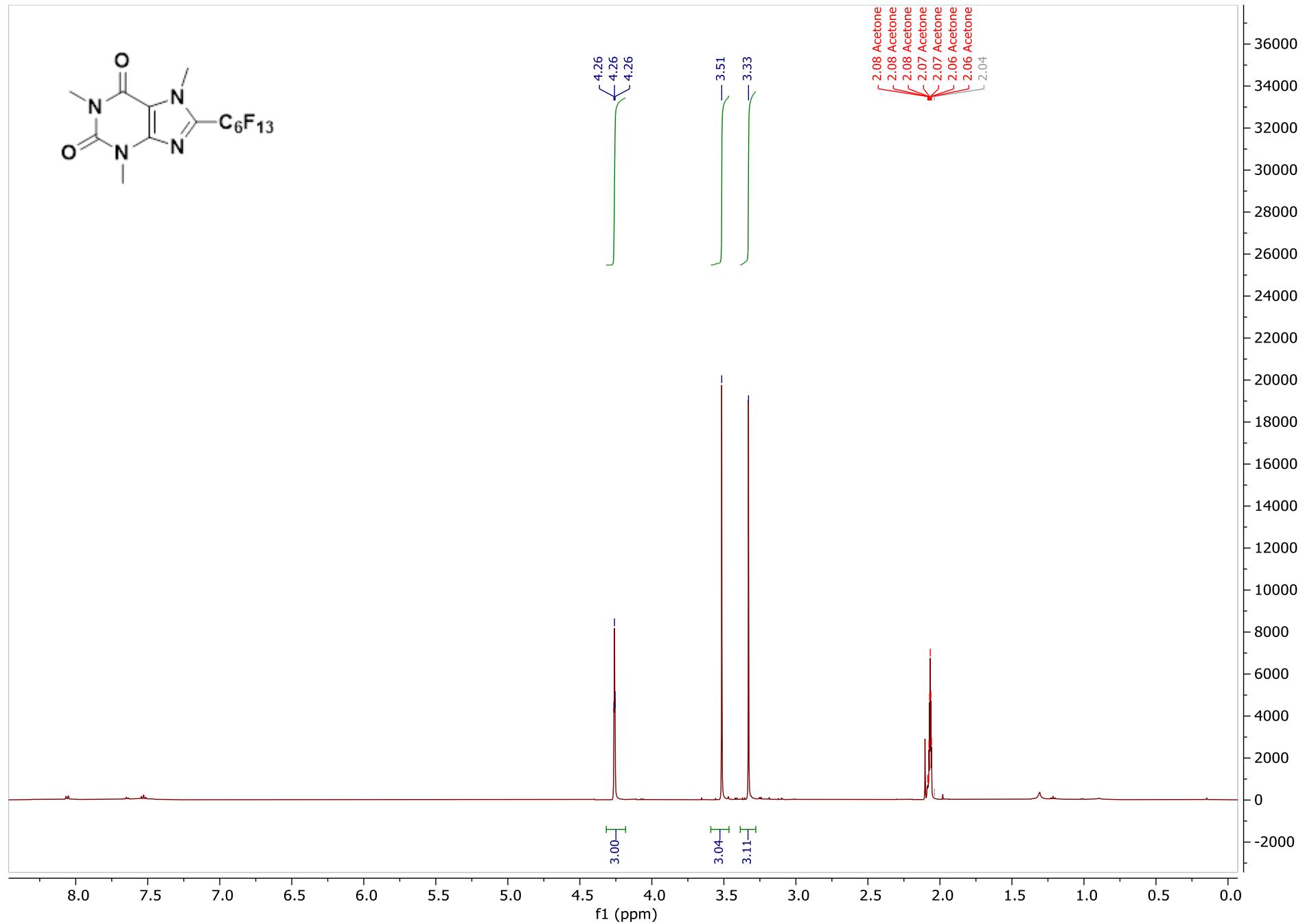
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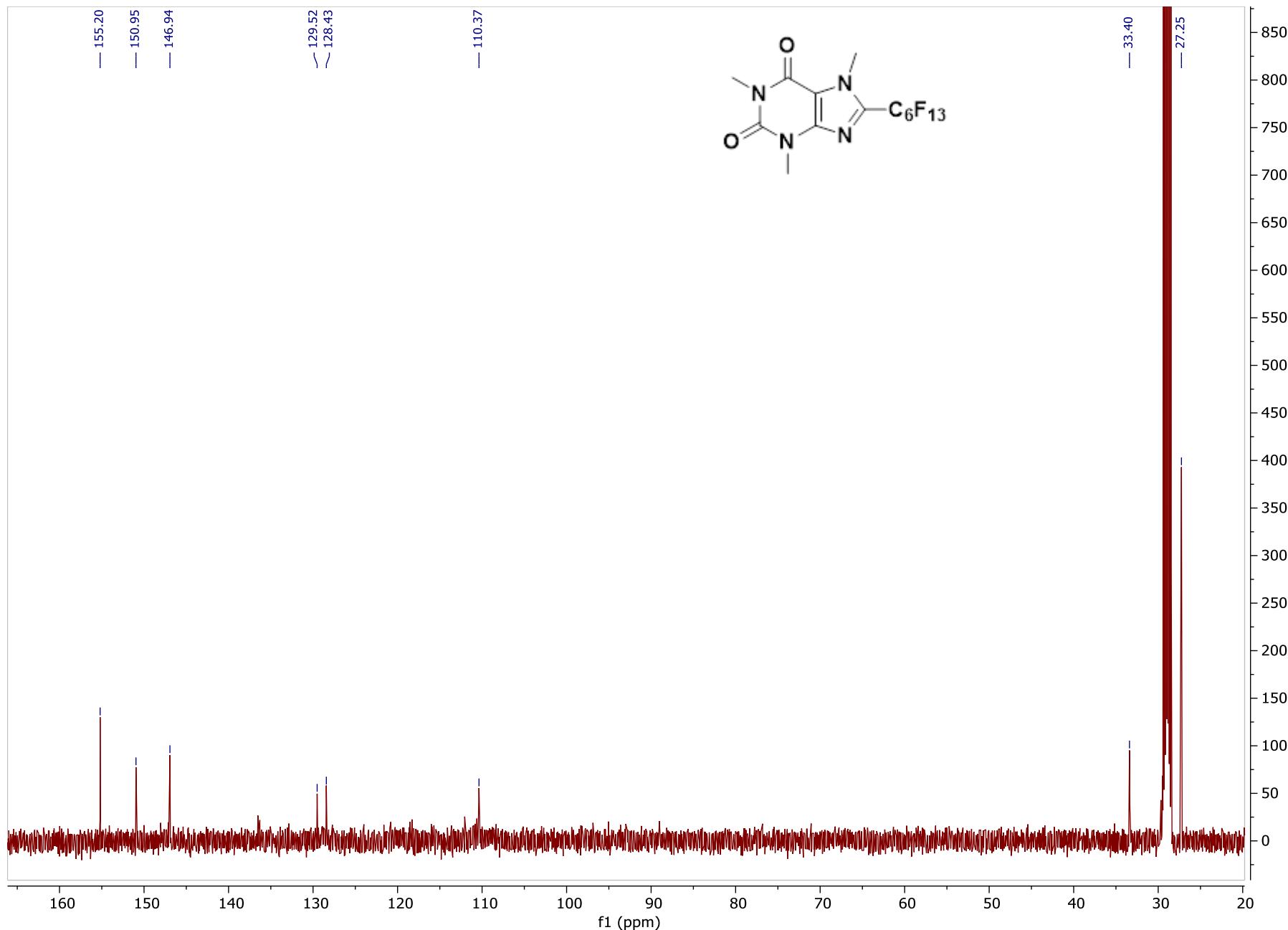
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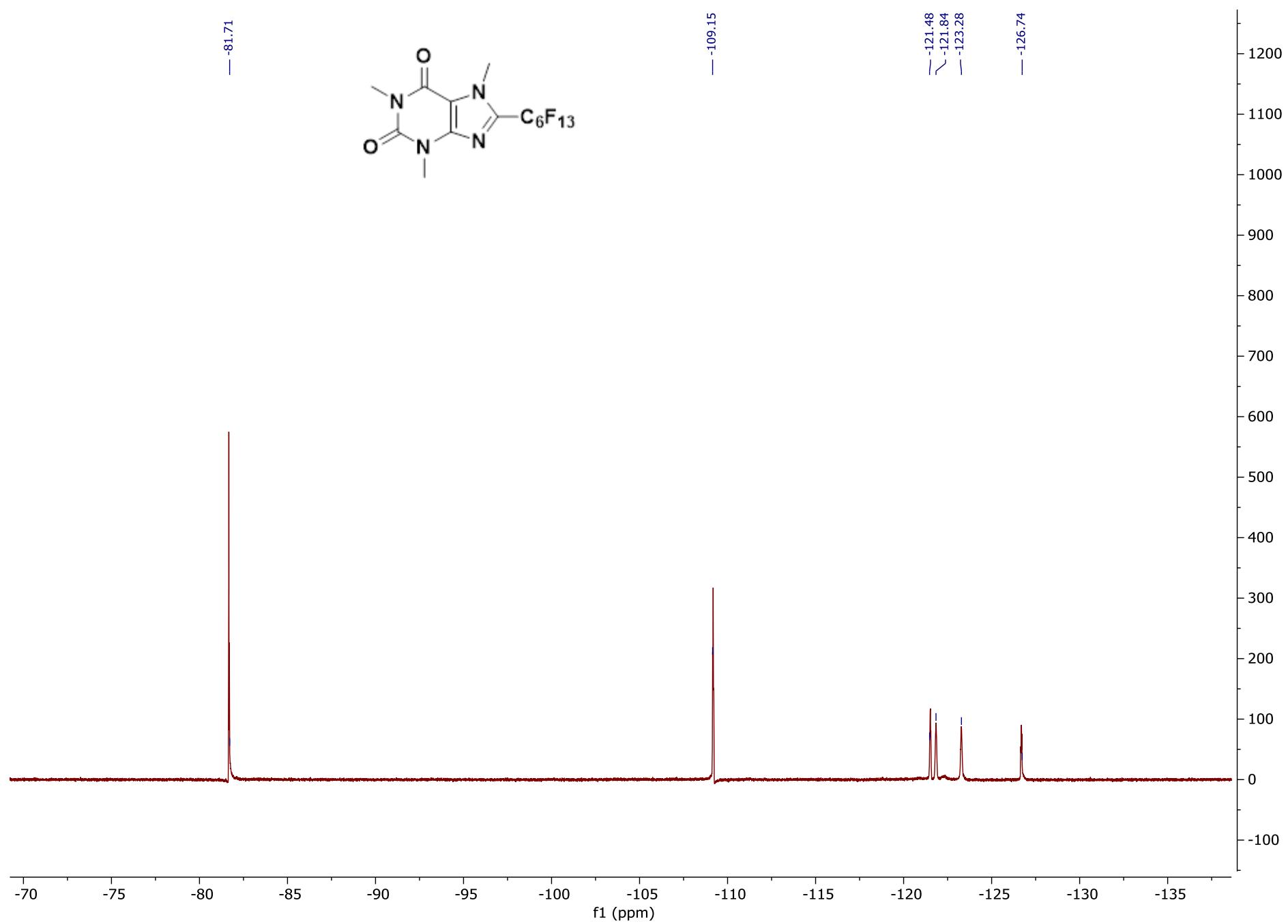
¹H NMR of **15b**, acetone-*d*₆, 500 MHz



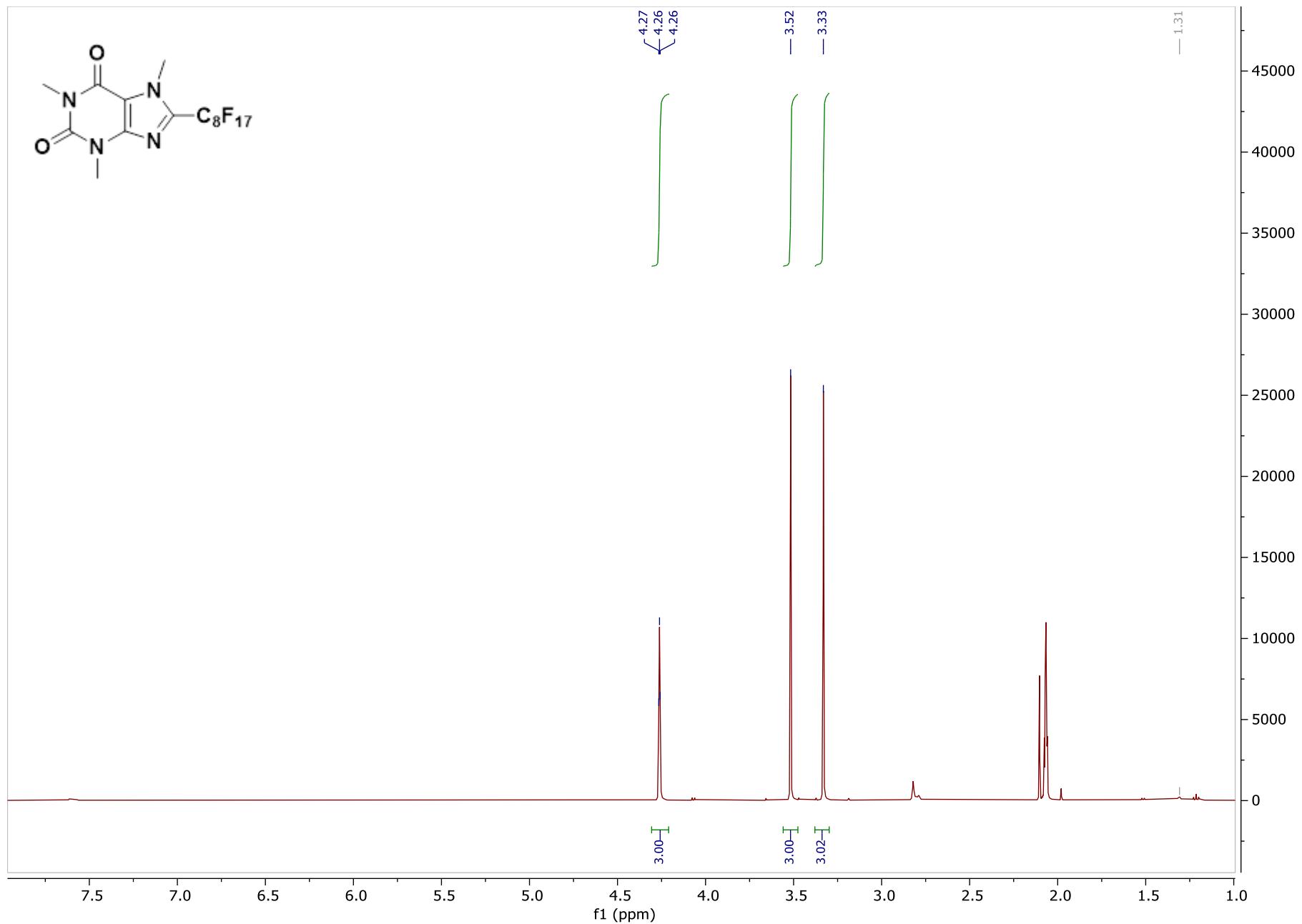
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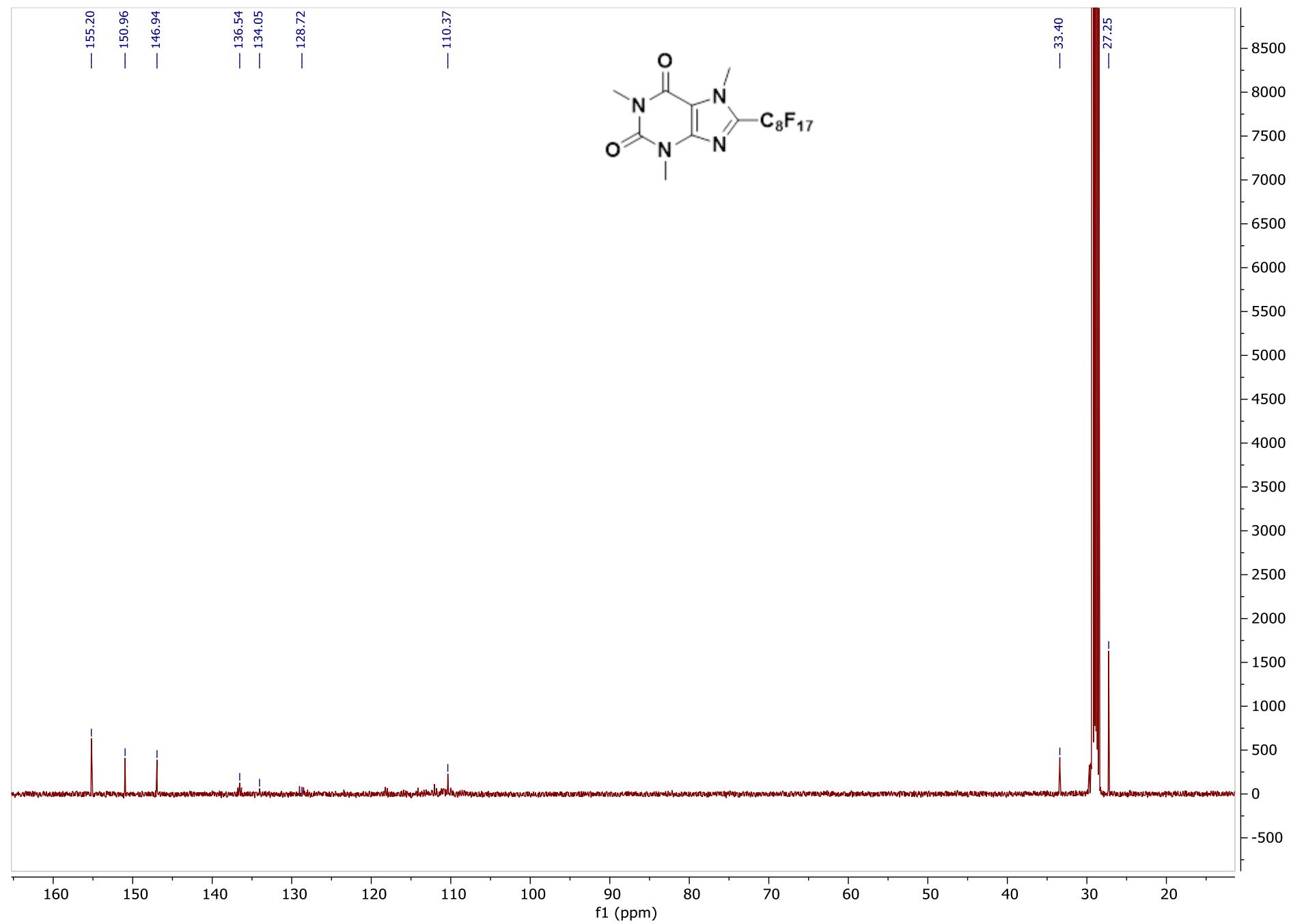
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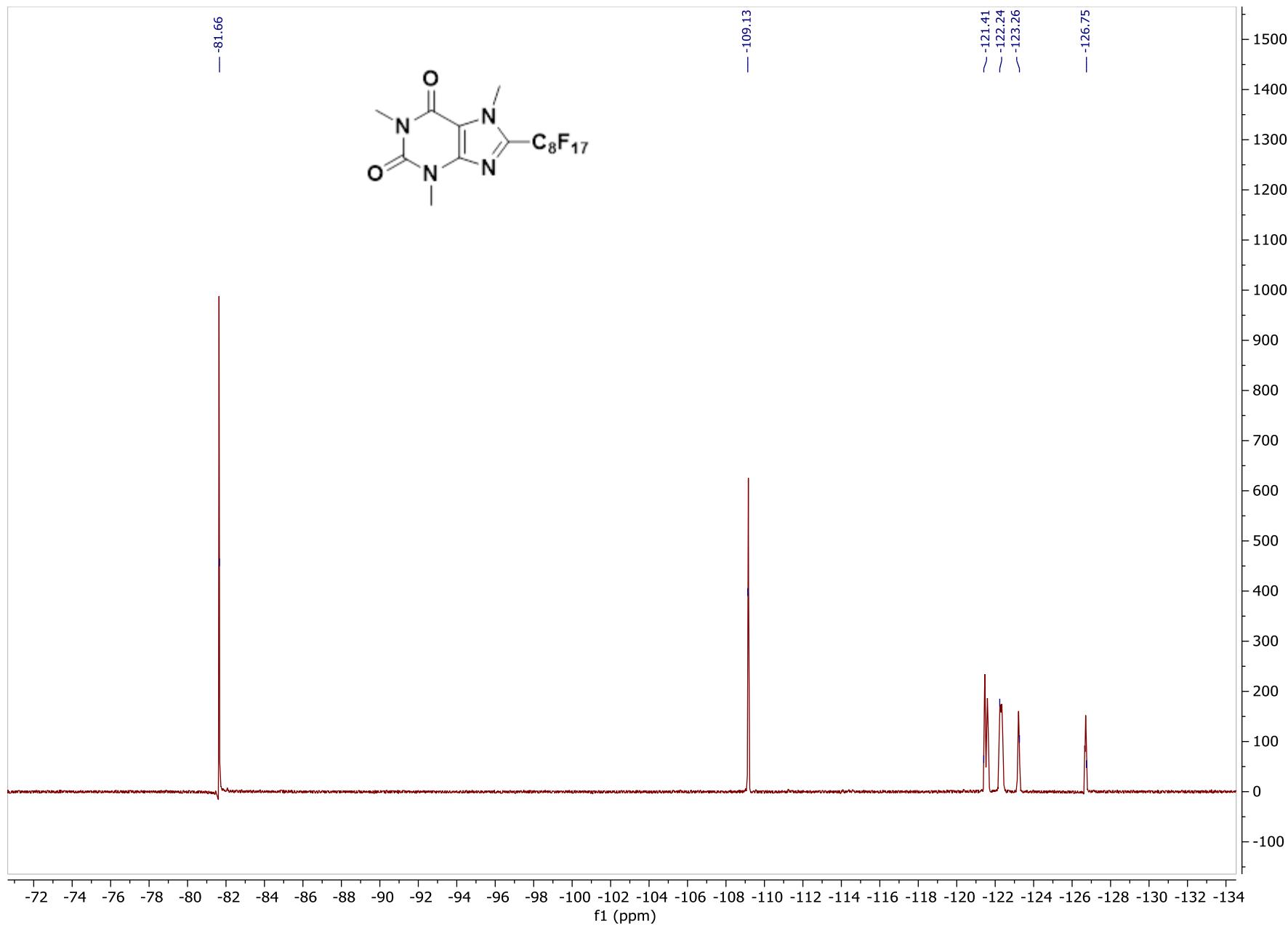
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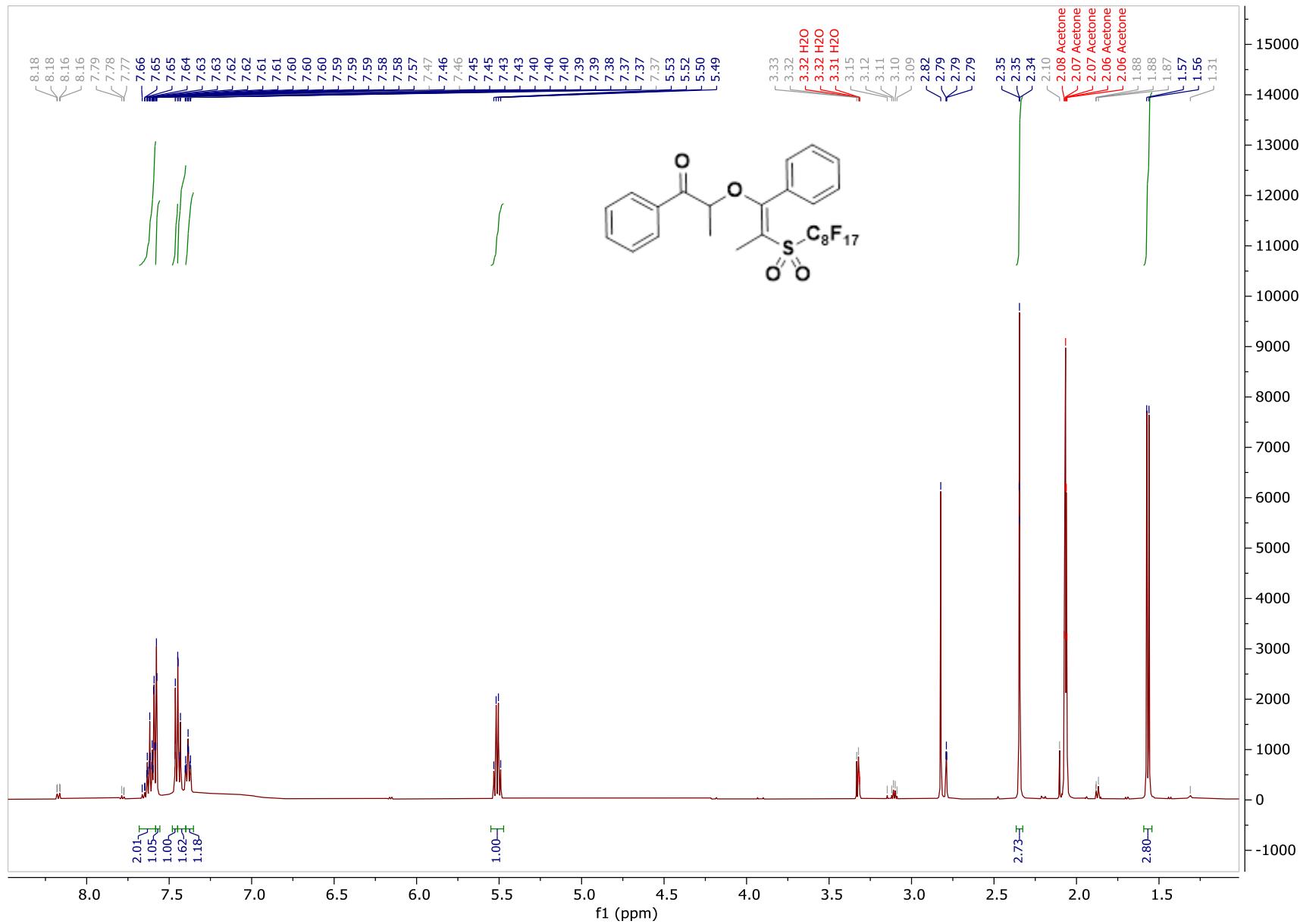
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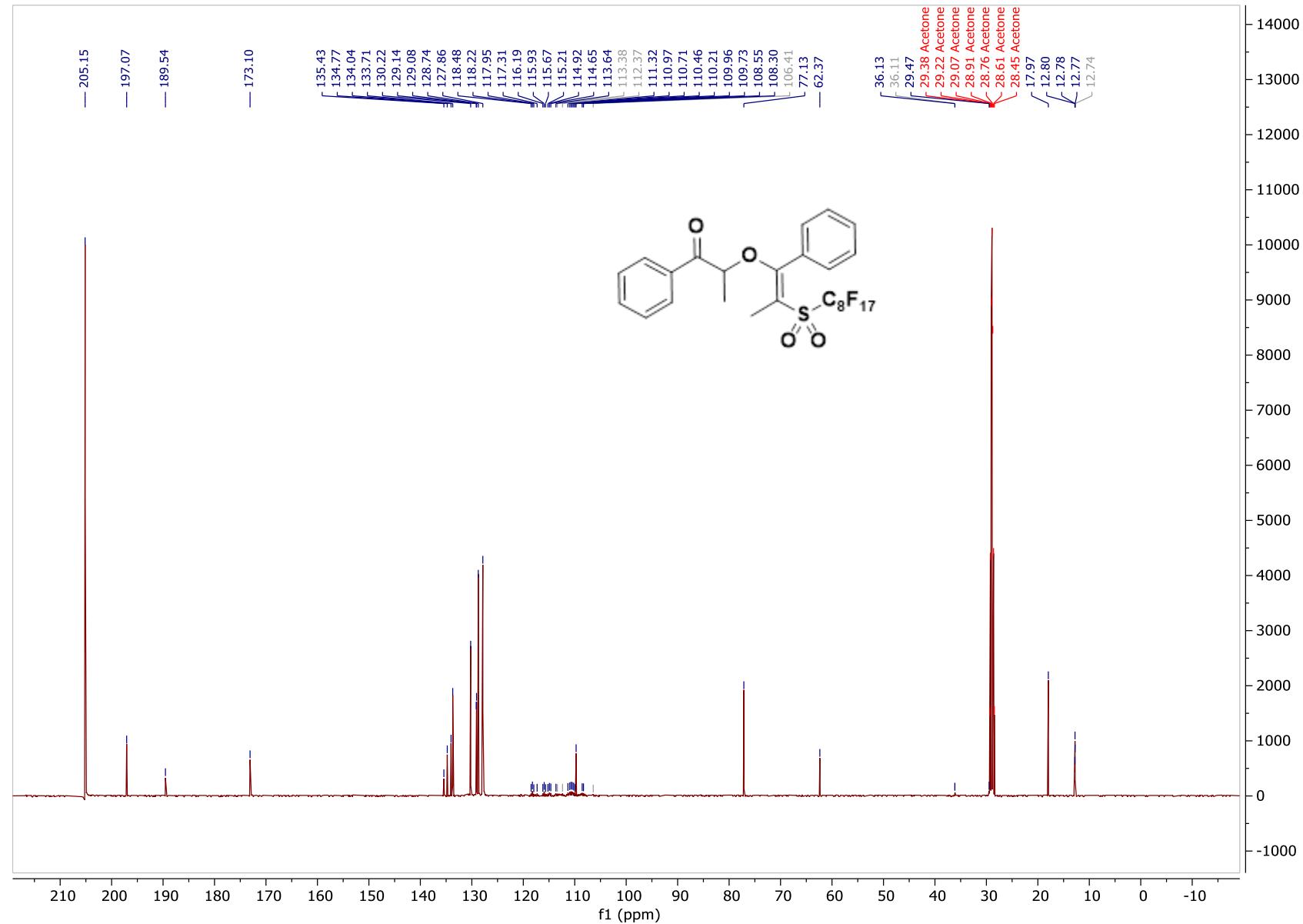
¹⁹F NMR of **15c**, acetone-*d*₆, 471 MHz



¹H NMR of byproduct B (perfluorooctyl analogue), acetone-*d*₆, 500 MHz



¹³C NMR of byproduct B (perfluorooctyl analogue), acetone-*d*₆, 126 MHz



¹⁹F NMR of byproduct B (perfluorooctyl analogue), acetone-*d*₆, 471 MHz

