Supporting Information

A Protocol for Selective Synthesis of Thiol-Functionalized Allylic Sulfonyl Fluorides

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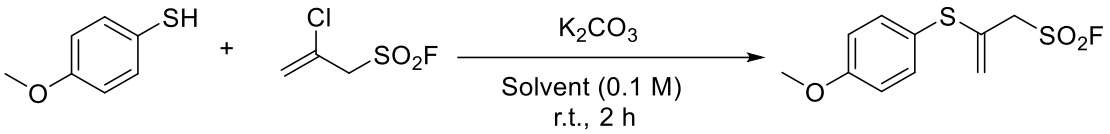
1. General Information

Instrumentation:

The reactions were conducted under air atmosphere unless otherwise noted. NMR spectra were recorded in $CDCl_3$ using a spectrometer operating at 500 MHz for ^{1}H, 471 MHz for ^{19}F, and 126 MHz for ^{13}C. (Note: $CDCl_3$: δ H = 7.264 ppm, δ C = 77.16 ppm). Chemical shifts were reported in ppm relative to TMS (0 ppm for ^{1}H NMR) as the internal standard. HPLC analyses were performed on a Waters e2695 system with a J&K RP-C18 column (5 μm, 4.6 × 150 mm). Product yields were determined using pure compounds as external standards. Coupling constants (J values) were reported in Hertz (Hz). The following abbreviations describe multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, hept = heptet, m = multiplet, brs = broad singlet, dd = doublet of doublets. Compound numbering for NMR spectral assignments is arbitrary and does not adhere to IUPAC nomenclature. Chemical structure names were generated using ChemDraw software. High-resolution mass spectrometry (HRMS) was performed using a TOF-Q EI instrument. Melting points were measured without correction. All reagents used in the reactions were obtained commercially and used as received, without additional purification. Thin-layer chromatography (TLC) spots were visualized under UV light (254 nm or 365 nm) and further stained with potassium permanganate or phosphomolybdic acid. Starting thiols were purchased and used directly without further modification.

2. Optimization of the Reaction Conditions

Table S1 Screening the Solvent.[a]

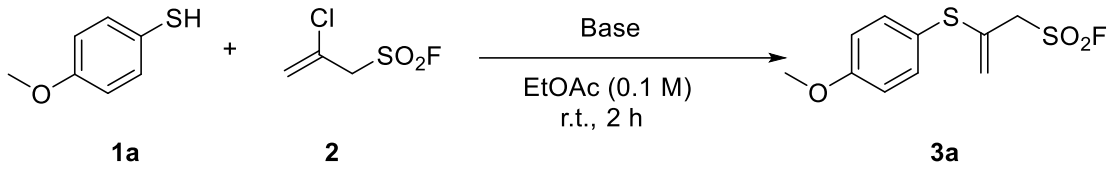


1a **2** **3a**

Entry	Solvent	Yield (3a, %)[b]
1	THF	53
2	MeCN	13
3	DCE	12
4	DCM	27
5	1,4-Dioxane	74
6	DMF	40
7	**EtOAc**	**85**
8	NMP	N.D.
9	C_2H_5OH	N.D.

[a]Reaction conditions: a mixture of 4-methoxybenzenethiol (**1a**, 28 mg, 0.2 mmol, 1.0 equiv.), CESF (**2**, 38 mg, 0.24 mmol, 1.2 equiv.) and K_2CO_3 (55 mg, 0.4 mmol, 2.0 equiv.) in solvent (2.0 mL) was stirred at room temperature for 2 h under air. [b]The yield was determined by HPLC using pure **3a** as the external standard ($t_{R,\mathbf{3a}}$ = 3.1 min, λ_{max} = 231.1 nm, Acetonitrile/ water = 80: 20 (v/v). N.D. = Not detectable.

Table S2 Screening the base.[a]

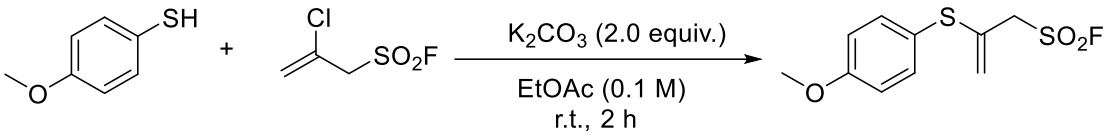


1a **2** **3a**

Entry	Base (2.0 equiv.)	Yield (3a, %)[b]
1	**K_2CO_3**	**85**
2	DIPEA	N.D.
3	Et_3N	N.D.
4	Tripopylamine	N.D.
5	TMEDA	N.D.
6	Na_2CO_3	50
7	Cs_2CO_3	69
8	$NaHCO_3$	48
9	Na_2SO_3	N.D.
10	KOH	43
11	NaH_2PO_4	N.D.

[a]Reaction conditions: a mixture of 4-methoxybenzenethiol (**1a**, 28 mg, 0.2 mmol, 1.0 equiv.), CESF (**2**, 38 mg, 0.24 mmol, 1.2 equiv.) and base (0.4 mmol, 2.0 equiv.) in EtOAc (2.0 mL) was stirred at room temperature for 2 h under air. [b]The yield was determined by HPLC using pure **3a** as the external standard ($t_{R,\mathbf{3a}}$ = 3.1 min, λ_{max} = 231.1 nm, Acetonitrile/ water = 80: 20 (v/v). N.D. = Not detectable.

Table S3 Screening the loading of CESF **(2)** [a]

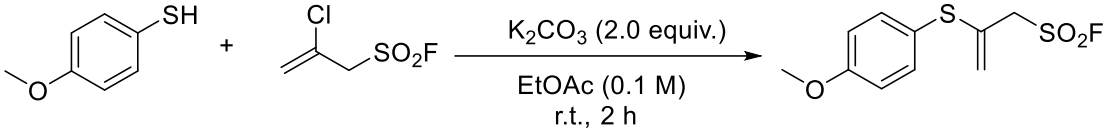


1a **2, X equuv.** **3a**

Entry	X equiv.	Yield (3a, %)[b]
1	1.0	61
2	1.2	85
3	**1.5**	**99**
4	2.0	94

[a]Reaction conditions: a mixture of 4-methoxybenzenethiol (**1a**, 28 mg, 0.2 mmol, 1.0 equiv.), CESF (**2**, X equiv.) and K_2CO_3 (55 mg, 0.4 mmol, 2.0 equiv.) in EtOAc (2.0 mL) was stirred at room temperature for 2 h under air. [b]The yield was determined by HPLC using pure **3a** as the external standard ($t_{R,\mathbf{3a}}$ = 3.1 min, λ_{max} = 231.1 nm, Acetonitrile/ water = 80: 20 (v/v).

Table S4 Screening of temperature[a]

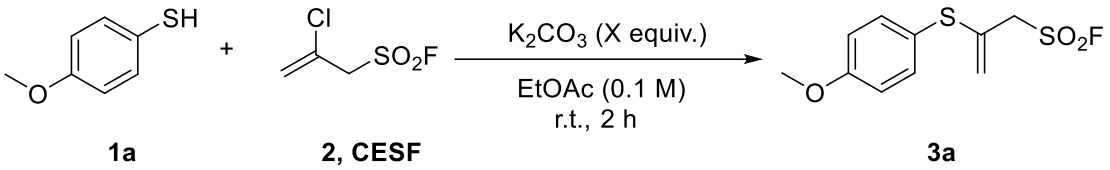


1a **2, CESF** **3a**

Entry	T (°C).	Yield (3a, %)[b]
1	0	99
2	**r.t**	**99**
3	40	87
4	60	85

[a]Reaction conditions: a mixture of 4-methoxybenzenethiol (**1a**, 28 mg, 0.2 mmol, 1.0 equiv.), CESF (**2**, 47 mg, 0.3 mmol, 1.5 equiv.) and K_2CO_3 (55 mg, 0.4 mmol, 2.0 equiv.) in EtOAc (2.0 mL) was stirred at room temperature for 2 h under air. [b]The yield was determined by HPLC using pure **3a** as the external standard ($t_{R,\mathbf{3a}}$ = 3.1 min, λ_{max} = 231.1 nm, Acetonitrile/ water = 80: 20 (v/v).

Table S5 Screening of the loading of base[a]



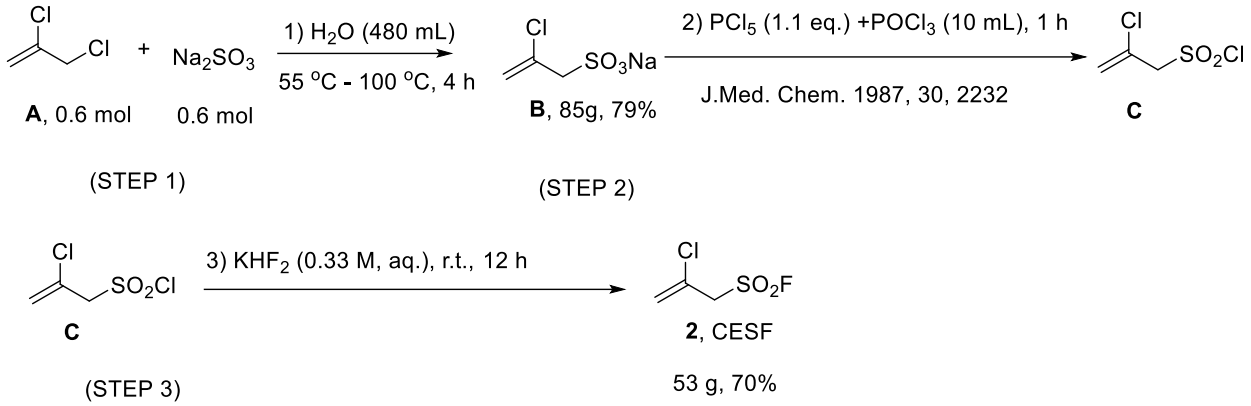
1a **2, CESF** **3a**

Entry	K_2CO_3 (X equiv.)	Yield (3a, %)[b]
1	1.0	80
2	**1.5**	**99**
3	2.0	99
4	2.5	90

[a]Reaction conditions: a mixture of 4-methoxybenzenethiol (**1a**, 28 mg, 0.2 mmol, 1.0 equiv.), CESF (**2**, 47 mg, 0.3 mmol, 1.5 equiv.) and K_2CO_3 (X equiv.) in EtOAc (2.0 mL) was stirred at room temperature for 2 h under air. [b]The yield was determined by HPLC using pure **3a** as the external standard ($t_{R,\mathbf{3a}}$ = 3.1 min, λ_{max} = 231.1 nm, Acetonitrile/ water = 80: 20 (v/v).

3. Experimental Procedures

3.1 Preparation of 2-chloroprop-2-ene-1-sulfonyl fluoride (CESF).[1]



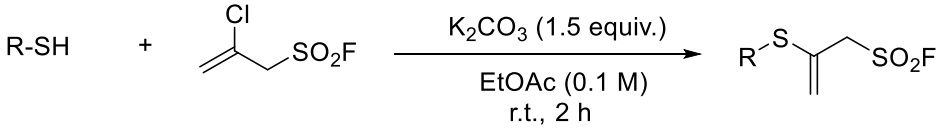
Step1: A solution of 1,2-dichloro-2-propene (**A**, 0.6 mol) was added gradually to an aqueous solution of Na_2SO_3 (0.6 mol) in 480 mL of water under continuous stirring. The reaction mixture was maintained at 55 °C using an oil bath and then refluxed for 4 hours. Afterward, the solvent was removed under reduced pressure. The resulting residue was treated with 500 mL of ethanol, and the mixture was refluxed with stirring for 10 minutes. Insoluble material was removed by hot filtration, and the filtrate was cooled to allow crystallization. The crystalline sodium 2-chloro-2-propene-1-sulfonate was obtained by filtration, yielding 85.0 g (**B**, 0.476 mol, 79%).

Step 2: A mixture of the sodium salt (**B**, 0.476 mol, 85.0 g) and PCl_5 (1.1 equivalents, 0.474 mol, 99 g) was stirred vigorously until it liquefied, via exothermic process over approximately 10 minutes. Phosphorus oxychloride (10 mL) was then used to rinse any residual solids from the bottle walls. The mixture was quickly heated in an oil bath at 120 °C for 1 hour, then allowed to cool. It was subsequently poured onto ice with vigorous stirring and left to stand for 30 minutes at room temperature.

Step 3: The evaporated 2-chloro-2-propenesulfonyl chloride (**C**) was directly reacted in the solution of KHF_2 (112 g, 0.33 M) for 12 hours. The reaction mixture was further extracted with CH_2Cl_2, then dried, and evaporated in vacuo. 2-chloroprop-2-ene-1- sulfonyl fluoride (CESF) was distilled under reduced pressure to give colorless oil 53 g (**2**, 70%). **^{1}H NMR** (500 MHz, $CDCl_3$) δ

5.75 (dd, J_1 = 19.6 Hz, J_2 = 2.3 Hz, 2H), 4.33 (d, J = 3.6 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 56.4 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 126.6, 123.6 (d, J = 1.6 Hz), 59.34(d, J = 19.4 Hz). HRMS-ESI (m/z) calcd. for $[C_3H_5ClFO_2S]^+$ ($[M+H]^+$): 158.9677, found: 158.9674.

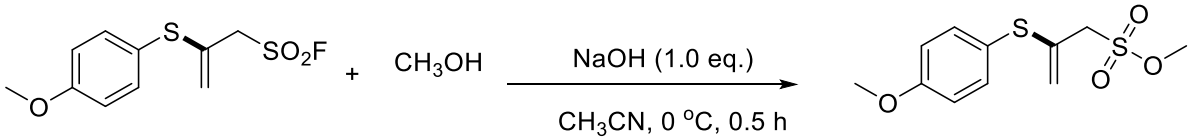
3.2 Procedure for the Synthesis of 3.



1 **2, CESF** **3**

An oven-dried reaction tube (20 mL) equipped with a magnetic stirring bar was charged with thiols (**1**, 1.0 mmol, 1.0 equiv.), CESF (**2**, 238 mg, 1.5 mmol, 1.5 equiv.) and 10.0 mL EtOAc. Then, potassium carbonate (207 mg, 2.0 mmol, 1.5 equiv.) was added to the solution. The reaction mixture was stirred at room temperature for 2 hours under an air atmosphere, with progress monitored by TLC. Once the reaction was complete, 20 mL of water was added, and the mixture was extracted three times with ethyl acetate (3 × 10 mL). The combined organic layers were dried over anhydrous sodium sulfate (Na_2SO_4) and concentrated under reduced pressure. The resulting residue was purified by column chromatography on silica gel using a mixture of petroleum ether and ethyl acetate as the eluent to obtain the desired product (**3**).

3.3 General procedure for synthesis of allyl sulfonamide from methanol.[2]

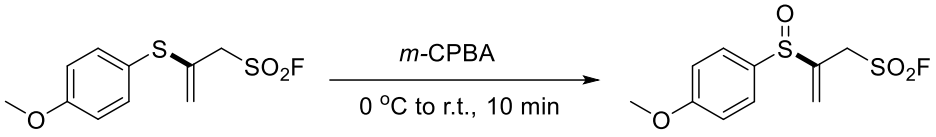


3a **Methanol** **4,** 95%

Methanol (1.0 mmol, 2.0 equiv., 32.0 mg) and sodium hydroxide (0.5 mmol, 1.0 equiv., 20.0 mg) were added to a stirred solution of 2-((4-methoxyphenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3a**, 0.5 mmol, 131 mg) dissolved in acetonitrile (2 mL) and the resulting mixture reacted at 0 ℃ for

0.5 h. The reaction was concentrated to dryness and the residue was further purified by column chromatography on silica gel by gradient elution with petroleum ether/ethyl acetate (10:1 to 5:1 v/v) as eluent to obtain pure allyl sulfonamide **4** as colorless oil (130 mg, 95% yield).

3.4 General procedure of oxidation for synthesis of sulfoxide 3a.[3]

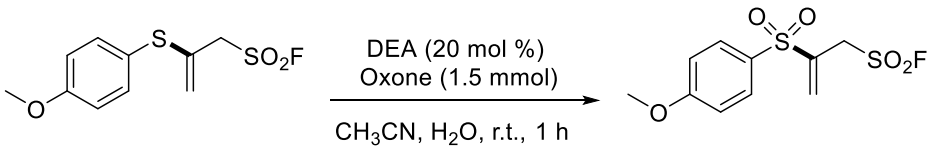


3a **5**, 99%

Sulfoxide

2-((4-methoxyphenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3a**, 0.3 mmol, 79 mg) was dissolved in acetonitrile (2 mL). subsequently, meta-Chloroperoxybenzoic acid (1.2 equiv.) was added to the solution and stirred at 0 °C to room temperature for 10 min and monitored by TLC to detect a new point of reaction completeness. Then, the reaction mixture was extracted with ethyl acetate (3×10 mL) and the combined organic layers was dried over anhydrous sodium sulfate, filtered and concentrated under reduced pressure. The crude product was further purified by column chromatography on silica gel by gradient elution with petroleum ether/ethyl acetate (5:1 to 3:1, v/v) as eluent to obtain pure sulfoxide **5** as white solid (83 mg, 99%).

3.5 General procedure of oxidation for synthesis of sulfone from 3a.[4]



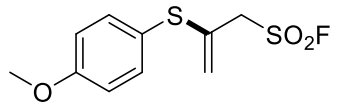
3a **6**, 41%

Sulfone

A mixture of 2-((4-methoxyphenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3a**, 0.3 mmol, 79 mg) and diethylamine (20 mol%) in acetonitrile (2 mL) was added to a solution of Oxone (1.5 mmol) in water (4 mL). The reaction was stirred for 1 hour, with progress monitored by TLC. After completion, the reaction mixture was extracted with ethyl acetate (3 × 10 mL), and the

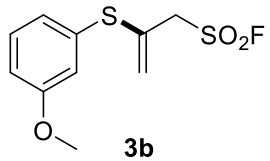
combined organic layers were dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was then purified via column chromatography on silica gel using a gradient elution of petroleum ether/ethyl acetate (5:1 to 3:1, v/v) as the mobile phase, yielding the pure sulfone **6** as a colorless oil (36 mg, 41%).

4. Characterization



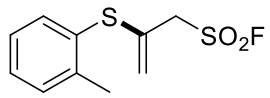
3a

2-((4-methoxyphenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3a**). light yellow oil, 257 mg, 98% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.7 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 5.56 (s, 1H), 5.29 (s, 1H), 4.13 (d, *J* = 3.1 Hz, 2H), 3.84 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.6 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 161.0, 136.5, 132.7, 121.0, 120.7, 115.5, 56.3 (d, *J* = 18.1 Hz), 55.6 (d, *J* = 3.0 Hz). **HRMS-ESI** (m/z) calcd. for $[C_{10}H_{12}FO_3S_2]^+$ ($[M+H]^+$): 263.0206, found: 263.0207.



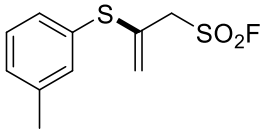
3b

2-((3-methoxyphenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3b**). light yellow oil, 234 mg, 89% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.30 (t, *J* = 8.0 Hz, 1H), 7.04 (d, *J* = 7.7 Hz, 1H), 7.00 (t, *J* = 2.1 Hz, 1H), 6.91 (dd, J_1 = 8.8 Hz, J_2 = 2.1 Hz, 1H), 5.73 (s, 1H), 5.60 (s, 1H), 4.16 (d, *J* = 3.1 Hz, 2H), 3.82 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.8 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 159.5, 131.2, 130.0, 129.7, 124.5, 123.4, 117.5, 114.2, 55.4 (d, *J* = 17.8 Hz), 54.6 (d, *J* = 3.0 Hz). **HRMS-ESI** (m/z) calcd. for $[C_{10}H_{12}FO_3S_2]^+$ ($[M+H]^+$): 263.0206, found: 263.0207.



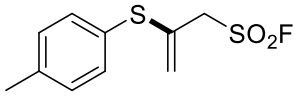
3c

2-(o-tolylthio)prop-2-ene-1-sulfonyl fluoride (**3c**). light yellow oil, 226 mg, 92% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.2 Hz, 1H), 7.31 – 7.23 (m, 2H), 7.23 – 7.14 (m, 1H), 5.52 (s, 1H), 5.07 (s, 1H), 4.10 (d, *J* = 3.2 Hz, 2H), 2.38 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.6 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 142.4, 135.5, 131.3, 130.9, 130.1, 129.5, 127.3, 120.5, 56.5 (d, *J* = 18.2 Hz), 20.4 (d, *J* = 2.4 Hz). **HRMS-ESI** (m/z) calcd. for $[C_{10}H_{12}FO_2S_2]^+$ ($[M+H]^+$): 247.0257, found: 247.0264.



3d

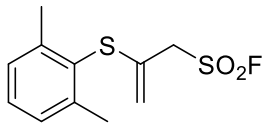
2-(m-tolylthio)prop-2-ene-1-sulfonyl fluoride (**3d).** light yellow oil, 245 mg, 99% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.33 – 7.26 (m, 3H), 7.19 (d, *J* = 6.6 Hz, 1H), 5.69 (s, 1H), 5.51 (s, 1H), 4.15 (d, *J* = 3.2 Hz, 2H), 2.37 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.7 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 140.0, 134.3, 131.5, 130.8, 130.8, 130.2, 129.8, 123.5, 56.5 (d, *J* = 17.8 Hz), 21.5 (d, *J* = 1.8 Hz). **HRMS-ESI** (m/z) calcd. for $[C_{10}H_{12}FO_2S_2]^+$ ($[M+H]^+$): 247.0257, found: 247.0264.



3e

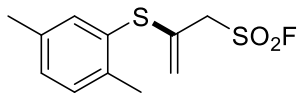
2-(p-tolylthio)prop-2-ene-1-sulfonyl fluoride (**3e).** Colorless oil, 199 mg, 81% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.2 Hz, 2H), 7.21 (d, *J* = 8.0 Hz, 2H), 5.63 (s, 1H), 5.42 (s,

1H), 4.13 (d, *J* = 3.2 Hz, 2H), 2.38 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.7 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 139.8, 134.1, 131.9, 130.7, 127.1, 122.3, 56.3 (d, *J* = 17.7 Hz), 21.4. **HRMS-ESI** (m/z) calcd. for $[C_{10}H_{12}FO_2S_2]^+$ ($[M+H]^+$): 247.0257, found: 247.0264.



3f

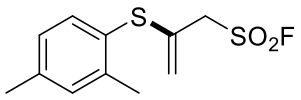
2-((2,6-dimethylphenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3f)**. light yellow solid, 252 mg, 97% yield. M p. 62−63 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.24 (d, *J* = 6.7 Hz, 1H), 7.18 (d, *J* = 7.5 Hz, 2H), 5.35 (s, 1H), 4.65 (s, 1H), 4.20 (d, *J* = 3.2 Hz, 2H), 2.46 (s, 6H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.3 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 144.0, 130.4, 130.2, 128.9, 128.4, 115.7, 56.7 (d, *J* = 18.4 Hz), 21.3 (d, *J* = 2.1 Hz). **HRMS-ESI** (m/z) calcd. for $[C_{11}H_{14}FO_2S_2]^+$ ($[M+H]^+$): 261.0414, found: 261.0411.



3g

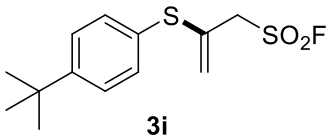
2-((2,5-dimethylphenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3g**). light yellow solid, 259 mg, 99% yield. M p. 60−61 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.29 (s, 1H), 7.20 (d, *J* = 7.7 Hz, 1H), 7.13 (d, *J* = 7.7 Hz, 1H), 5.56 (s, 1H), 5.12 (s, 1H), 4.16 (d, *J* = 3.1 Hz, 2H), 2.38 (s, 3H), 2.32 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.6 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 139.3, 137.2, 136.0, 131.2, 131.1, 129.1, 120.3, 56.6 (d, *J* = 18.2 Hz), 20.9, 20.0. **HRMS-ESI** (m/z) calcd. for $[C_{11}H_{14}FO_2S_2]^+$ ($[M+H]^+$): 261.0414, found: 261.0411.

Note: In the ^{13}C NMR spectrum of **3g**, there are theoretically eleven peaks expected. However, due to their close proximity, it is difficult to resolve the overlapping peaks clearly.



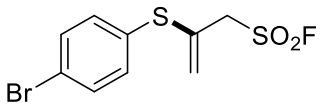
3h

2-((2,4-dimethylphenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3h**). light yellow oil, 258 mg, 99% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 7.7 Hz, 1H), 7.14 (s, 1H), 7.04 (d, *J* = 7.9 Hz, 1H), 5.52 (s, 1H), 5.06 (s, 1H), 4.15 (d, *J* = 3.1 Hz, 2H), 2.37 (d, *J* = 23.0 Hz, 6H). . **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.5 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 142.4, 140.5, 135.8, 132.1, 131.2, 128.1, 125.8, 119.4, 56.3 (d, *J* = 18.1 Hz), 21.28, 20.3. **HRMS-ESI** (m/z) calcd. for $[C_{11}H_{14}FO_2S_2]^+$ ($[M+H]^+$): 261.0414, found: 261.0411.



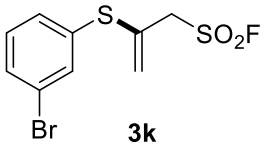
3i

2-((4-(tert-butyl)phenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3i).** light yellow oil, 253 mg, 89% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.41 (d, *J* = 1.4 Hz, 4H), 5.66 (s, 1H), 5.47 (d, *J* = 1.0 Hz, 1H), 4.15 (d, *J* = 3.1 Hz, 2H), 1.33 (s, 9H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.7 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 152.9, 133.7, 131.8, 127.3, 127.0, 122.8, 56.4 (d, *J* = 17.9 Hz), 34.9, 31.3. **HRMS-ESI** (m/z) calcd. for $[C_{13}H_{18}FO_2S_2]^+$ ($[M+H]^+$): 289.0727, found: 289.0782.



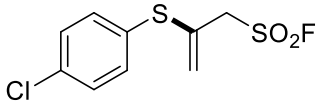
3j

2-((4-bromophenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3j**). light yellow solid, 282 mg, 91% yield. M p. 42−43 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.4 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 5.73 (s, 1H), 5.51 (s, 1H), 4.15 (d, *J* = 3.1 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 56.0 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 135.1, 133.1, 130.9, 130.1, 124.3, 123.8, 56.5 (d, *J* = 18.2 Hz). **HRMS-ESI** (m/z) calcd. for $[C_9H_9BrFO_2S_2]^+$ ($[M+H]^+$): 310.9206, found 310.9208.



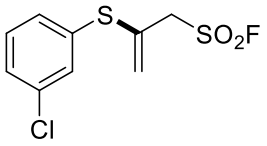
3k

2-((3-bromophenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3k**). light yellow solid, 285 mg, 92% yield. M p. 63−64 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.60 (t, *J* = 1.9 Hz, 1H), 7.54 – 7.48 (m, 1H), 7.40 (d, *J* = 7.8 Hz, 1H), 7.27 (t, *J* = 7.8 Hz, 1H), 5.79 (s, 1H), 5.60 (s, 1H), 4.16 (d, *J* = 3.1 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.9 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 135.6, 133.4, 132.2, 131.7, 131.2, 130.4, 125.6, 123.5, 56.5 (d, *J* = 17.8 Hz). **HRMS-ESI** (m/z) calcd. for $[C_9H_9BrFO_2S_2]^+$ ($[M+H]^+$): 310.9206, found 310.9208.



3l

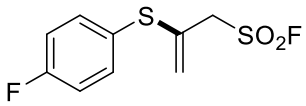
2-((4-chlorophenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3l**). light yellow oil, 153 mg, 57% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.40 (qd, J_1 = 8.6 Hz, J_2 = 2.1 Hz, 4H), 5.72 (s, 1H), 5.49 (s, 1H), 4.15 (d, *J* = 2.7 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.9 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 135.7, 135.0, 131.0 (d, *J* = 5.2 Hz), 130.1, 129.4, 124.0, 56.5 (d, *J* = 18.0 Hz). **HRMS-ESI** (m/z) calcd. for $[C_9H_9ClFO_2S_2]^+$ ($[M+H]^+$): 266.9711, found 266.9711



3m

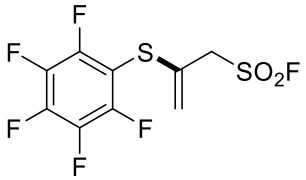
2-((3-chlorophenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3m**). light yellow oil, 260 mg, 97% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.45 (t, *J* = 1.8 Hz, 1H), 7.38 – 7.31 (m, 3H), 5.79 (s, 1H), 5.61 (s, 1H), 4.16 (d, *J* = 3.0 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.9 (s, 1F). **^{13}C NMR** (126

MHz, $CDCl_3$) δ 135.6, 133.1, 132.7, 131.2, 130.9, 130.4, 129.3, 125.6, 56.5 (d, *J* = 18.1 Hz). **HRMS-ESI** (m/z) calcd. for $[C_9H_9ClFO_2S_2]^+$ ($[M+H]^+$): 266.9711, found 266.9711.



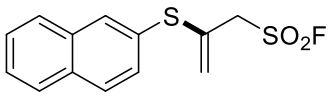
3n

2-((4-fluorophenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3n**). light yellow oil, 215 mg, 86% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.53 – 7.46 (m, 2H), 7.15 – 7.08 (m, 2H), 5.65 (s, 1H), 5.37 (s, 1H), 4.14 (d, *J* = 3.1 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.8 (s, 1F), -110.74 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 164.6, 162.6, 136.5 (d, *J* = 8.6 Hz), 131.8, 126.0 (d, *J* = 3.6 Hz), 122.5, 117.3, 117.1, 56.4 (d, *J* = 18.2 Hz). **HRMS-ESI** (m/z) calcd. for $[C_9H_9F_2O_2S_2]^+$ ($[M+H]^+$): 251.0007, found 251.0009.



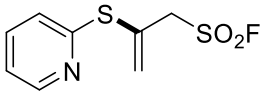
3o

2-((perfluorophenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3o**). White solid, 216 mg, 67% yield. M.p. 73−74 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.81 (s, 1H), 5.56 (s, 1H), 4.22 (d, *J* = 3.1 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.4 (s, 1F), -130.6 – -130.8 (m, 2F), -148.3 (t, 1F) (t, *J* = 20.7 Hz), -159.0 (td, J_1 = 22.8 Hz, J_2 = 22.1 Hz, J_3 = 7.5 Hz) (m, 2F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 149.4 – 148.2 (m), 147.2 – 146.4 (m), 139.2, 137.2, 127.7, 127.4, 127.1, 125.8, 56.8 (d, *J* = 19.4 Hz). **HRMS-ESI** (m/z) calcd. for $[C_9H_5F_6O_2S_2]^+$ ($[M+H]^+$): 322.9630, found 322.9632.



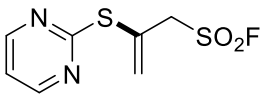
3p

2-(naphthalen-2-ylthio)prop-2-ene-1-sulfonyl fluoride (**3p**). light yellow solid, 209 mg, 74% yield. M p. 51−52 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.00 (s, 1H), 7.86 (d, *J* = 8.1 Hz, 2H), 7.85 – 7.80 (m, 1H), 7.59 – 7.52 (m, 2H), 7.50 (d, *J* = 8.4 Hz, 1H), 5.74 (s, 1H), 5.55 (s, 1H), 4.19 (d, *J* = 3.2 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.8 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 133.9, 133.4, 133.3, 131.3, 130.0, 129.7, 128.2, 128.0, 127.9, 127.4, 127.2, 123.80, 56.53 (d, *J* = 18.1 Hz). **HRMS-ESI** (m/z) calcd. for $[C_{13}H_{12}FO_2S_2]^+$ ($[M+H]^+$): 283.0257, found 283.0266.



3q

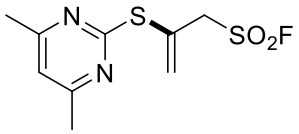
2-(pyridin-2-ylthio)prop-2-ene-1-sulfonyl fluoride (**3q**). Yellow solid, 176 mg, 76% yield. M.p. 76−77 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 5:1to 3:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.46 (d, *J* = 4.3 Hz, 1H), 7.60 (td, J_1 = 7.7 Hz, J_2 = 1.9 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 1H), 7.11 (dd, J_1 = 6.9 Hz, J_2 = 5.5 Hz, 1H), 6.09 (s, 1H), 6.06 (s, 1H), 4.57 (d, *J* = 3.6 Hz, 2H)).**^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.6 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 156.8, 150.3, 137.2, 132.0, 127.4, 123.3, 121.1, 57.1 (d, *J* = 16.7 Hz). **HRMS-ESI** (m/z) calcd. for $[C_8H_9FNO_2S_2]^+$ ($[M+H]^+$): 234.0053, found 234.0027.



3r

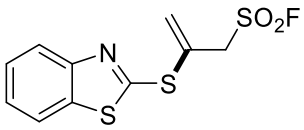
2-(pyrimidin-2-ylthio)prop-2-ene-1-sulfonyl fluoride (**3r**). Yellow solid, 176 mg, 76% yield. M.p. 64−65 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 5:1to 3:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.56 (d, *J* = 4.9 Hz, 2H), 7.08 (t, *J* = 4.9 Hz, 1H), 6.17 (s, 1H), 6.14 (s, 1H), 4.69 (d, *J* = 3.8 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.6 (s,

1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 171.1, 158.0, 133.8, 126.6, 117.7, 57.2 (d, *J* = 16.7 Hz). **HRMS-ESI** (m/z) calcd. for $[C_7H_8FN_2O_2S_2]^+$ ($[M+H]^+$): 235.0006, found 235.0009.



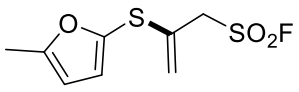
3s

2-((4,6-dimethylpyrimidin-2-yl)thio)prop-2-ene-1-sulfonyl fluoride (**3s**). Yellow solid, 176 mg, 76% yield. M.p. 88−89 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 3:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 6.78 (s, 1H), 6.13 (s, 1H), 6.08 (s, 1H), 4.73 (d, *J* = 4.0 Hz, 2H), 2.41 (s, 6H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.5 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 169.6, 167.9, 132.8, 127.0, 116.9, 57.0 (d, *J* = 16.2 Hz), 23.9. **HRMS-ESI** (m/z) calcd. for $[C_9H_{12}FN_2O_2S_2]^+$ ($[M+H]^+$): 263.0319, found 263.0319.



3t

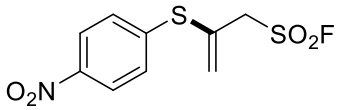
2-(benzo[d]thiazol-2-ylthio)prop-2-ene-1-sulfonyl fluoride (**3t**). Yellow solid, 229 mg, 79% yield. M.p. 63−64 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.1 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 1H), 7.38 (t, *J* = 7.7 Hz, 1H), 6.25 (s, 1H), 6.17 (s, 1H), 4.68 (d, *J* = 3.8 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.7 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 162.7, 153.3, 135.9, 133.6, 126.6, 126.5, 125.4, 122.4, 121.4, 56.9 (d, *J* = 17.6 Hz). **HRMS-ESI** (m/z) calcd. for $[C_{10}H_9FNO_2S_3]^+$ ($[M+H]^+$): 289.9774, found 289.9779.



3u

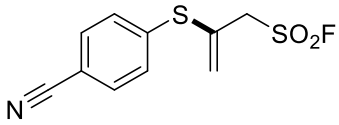
2-((5-methylfuran-2-yl)thio)prop-2-ene-1-sulfonyl fluoride (**3u**). Colorless oil, 150 mg, 63% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 2.0 Hz, 1H), 6.37 (d, *J* = 2.0 Hz, 1H), 5.53

(s, 1H), 5.26 (d, *J* = 1.2 Hz, 1H), 4.14 (d, *J* = 3.2 Hz, 2H), 2.34 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.4 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 157.8, 141.8, 131.1, 119.3, 115.1, 106.2, 56.0 (d, *J* = 18.7 Hz), 11.9 (d, *J* = 2.2 Hz). **HRMS-ESI** (m/z) calcd. for $[C_8H_{10}FO_3S_2]^+$ ($[M+H]^+$): 237.0050, found 237.0022.



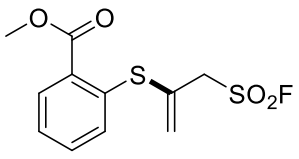
3v

2-((4-nitrophenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3v**). Yellow solid, 232 mg, 84% yield. M.p. 93−94 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 8.22 (d, *J* = 7.0 Hz, 2H), 7.53 (d, *J* = 7.0 Hz, 2H), 6.07 (s, 1H), 5.93 (s, 1H), 4.23 (d, *J* = 2.5 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 56.2 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 147.4, 141.4, 131.0, 130.9, 128.3, 124.7, 56.8 (d, *J* = 18.0 Hz).



3w

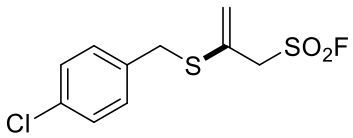
2-((4-cyanophenyl)thio)prop-2-ene-1-sulfonyl fluoride (**3w**). White solid, 209 mg, 81% yield. M.p. 88−89 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.68 – 7.62 (m, 2H), 7.49 (dd, J_1 = 8.4 Hz, J_2 = 1.9 Hz, 2H), 6.00 (s, 1H), 5.84 (s, 1H), 4.20 (d, *J* = 3.1 Hz, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 56.2 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 138.8, 133.2, 131.4, 129.9, 128.6, 118.2, 111.9, 56.7 (d, *J* = 17.8 Hz).



3x

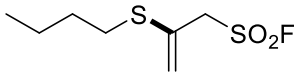
methyl 2-((3-(fluorosulfonyl)prop-1-en-2-yl)thio)benzoate (**3x**). White solid, 258 mg, 89% yield. M.p. 82−83 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl

acetate = 10:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.8 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 1H), 7.29 (d, *J* = 8.0 Hz, 2H), 6.02 (s, 1H), 5.93 (s, 1H), 4.14 (d, *J* = 3.1 Hz, 2H), 3.87 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 56.1 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 166.7, 135.6, 132.7, 131.6, 130.9, 130.8, 130.0, 129.3, 127.0, 56.5 (d, *J* = 17.2 Hz), 52.6 (d, *J* = 5.3 Hz).



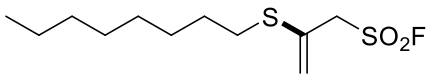
3y

2-((4-chlorobenzyl)thio)prop-2-ene-1-sulfonyl fluoride (**3y**). Colorless oil, 150 mg, 63% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.35 (s, 4H), 5.63 (d, *J* = 1.5 Hz, 1H), 5.34 (d, *J* = 1.6 Hz, 1H), 4.19 (d, *J* = 3.2 Hz, 2H), 4.05 (s, 2H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.3 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 133.8 (d, *J* = 12.3 Hz), 130.6, 130.3 (d, *J* = 7.3 Hz), 129.4, 129.04, 119.1, 57.9 (d, *J* = 18.2 Hz), 36.9. **HRMS-ESI** (m/z) calcd. for $[C_{10}H_{11}ClFO_2S_2]^+$ ($[M+H]^+$): 280.9868, found 280.9861.



3z

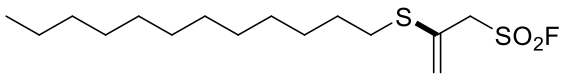
2-(butylthio)prop-2-ene-1-sulfonyl fluoride (**3z**). light yellow oil, 187 mg, 88% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.55 (s, 1H), 5.25 (s, 1H), 4.16 (d, *J* = 3.1 Hz, 2H), 2.80 (t, *J* = 7.4 Hz, 2H), 1.66 (q, *J* = 7.5 Hz, 2H), 1.45 (q, *J* = 7.5 Hz, 2H), 0.94 (t, *J* = 7.4 Hz, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.2(s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 131.3, 116.4, 58.13(d, *J* = 18.0 Hz), 32.3, 29.8, 22.2, 13.7. **HRMS-ESI** (m/z) calcd. for $[C_7H_{14}FO_2S_2]^+$ ($[M+H]^+$): 213.0414, found 213.0413.



3aa

2-(octylthio)prop-2-ene-1-sulfonyl fluoride (**3aa**). light yellow oil, 170 mg, 63% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.56 (s, 1H), 5.25 (d, *J* = 1.5 Hz, 1H), 4.16 (d, *J* = 3.0 Hz, 2H),

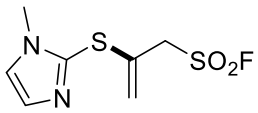
2.80 (t, *J* = 7.4 Hz, 2H), 1.71 – 1.65 (m, 2H), 1.42 (t, *J* = 7.4 Hz, 2H), 1.29 (dd, J_1 = 9.2 Hz, J_2 = 4.6 Hz, 8H), 0.88 (d, *J* = 7.1 Hz, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.2 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 131.3, 116.4, 58.2 (d, *J* = 17.8 Hz), 32.6, 31.9, 29.2, 29.2, 29.1, 27.8, 22.8, 14.2. **HRMS-ESI** (m/z) calcd. for $[C_{11}H_{22}FO_2S_2]^+$ ($[M+H]^+$): 269.1040, found 269.1077.



3ab

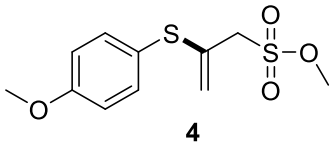
2-(dodecylthio)prop-2-ene-1-sulfonyl fluoride (**3ab**). Light yellow oil, 247 mg, 76% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 5.56 (s, 1H), 5.25 (d, *J* = 1.5 Hz, 1H), 4.16 (d, *J* = 3.0 Hz, 2H), 2.80 (t, *J* = 7.4 Hz, 2H), 1.68 (p, *J* = 7.5 Hz, 2H), 1.27 (s, 18H), 0.89 (t, *J* = 6.8 Hz, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.2 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 131.2, 116.3, 58.0 (d, *J* = 17.9 Hz), 32.5, 31.9, 29.6, 29.5, 29.4, 29.3, 29.1, 29.0, 27.7, 22.7, 14.1. **HRMS-ESI** (m/z) calcd. for $[C_{15}H_{30}FO_2S_2]^+$ ($[M+H]^+$): 325.1666, found 325.1670.

Note: In the ^{13}C NMR spectrum of **3y**, fifteen peaks are theoretically expected. However, the overlapping peaks are challenging to distinguish clearly due to their close proximity.



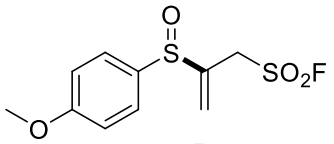
3ac

2-((1-methyl-1H-imidazol-2-yl)thio)prop-2-ene-1-sulfonyl fluoride (**3ac**). Yellow solid, 195 mg, 83% yield. M.p. 146−147 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 6.76 (d, J = 4.6 Hz, 2H), 5.75 (s, 1H), 5.65 (s, 1H), 5.13 (d, J = 4.3 Hz, 2H), 3.59 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 55.2 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 162.3, 133.5, 123.4, 119.1, 118.6, 53.4 (d, *J* = 17.5 Hz), 35.1 (d, *J* = 2.5 Hz). **HRMS-ESI** (m/z) calcd. for $[C_7H_{10}FN_2O_2S_2]^+$ ($[M+H]^+$): 237.0162, found 237.0162.



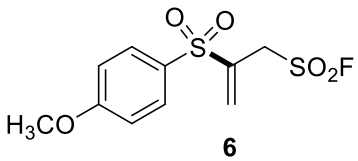
4

methyl 2-((4-methoxyphenyl)thio)prop-2-ene-1-sulfonate (**4**). Colorless oil, 130 mg, 95% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 to 5:1 (v/v) as eluent. **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.8 Hz, 2H), 6.76 (d, *J* = 8.7 Hz, 2H), 5.36 (s, 1H), 5.00 (s, 1H), 3.81 (s, 3H), 3.75 (s, 2H), 3.67 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 160.8, 137.3, 136.4, 134.3, 121.6, 119.2, 116.0, 115.4, 57.0 (d, *J* = 2.8 Hz), 55.5 (d, *J* = 2.7 Hz), 55.5. **HRMS-ESI** (m/z) calcd. for $[C_{11}H_{15}O_4S_2]^+$ ($[M+H]^+$): 275.0406, found 275.0403.



5

(R)-2-((4-methoxyphenyl)sulfinyl)prop-2-ene-1-sulfonyl fluoride (**5**). White solid, 83 mg, 99% yield. M p. 80−81 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 5:1to 3:1 (v/v) as eluent. **^{1}H NMR** δ 7.82 (d, *J* = 9.0 Hz, 2H), 7.04 (d, *J* = 9.0 Hz, 2H), 6.79 (s, 1H), 6.42 (s, 1H), 4.36 (d, *J* = 3.6 Hz, 2H), 3.90 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 56.1 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 163.2, 141.9, 131.6, 127.5, 125.7, 115.6, 55.9 (d, *J* = 3.0 Hz), 47.9 (d, *J* = 20.8 Hz). **HRMS-ESI** (m/z) calcd. for $[C_{10}H_{12}FO_4S_2]^+$ ($[M+H]^+$): 279.0156, found 279.0131.

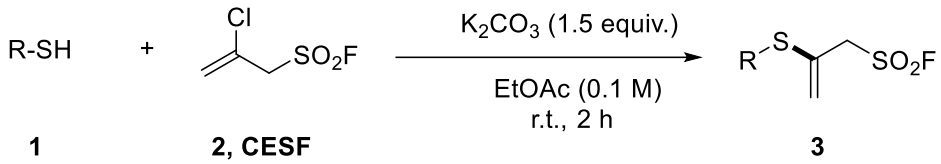


6

2-((4-methoxyphenyl)sulfonyl)prop-2-ene-1-sulfonyl fluoride (**6**). Colorless oil, 36 mg, 41% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 3:1 (v/v) as eluent **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.8 Hz, 2H), 7.03 (d, *J* = 8.8 Hz, 2H), 6.50 (s, 1H), 6.30 (s, 1H), 4.21 (dd, J_1 = 15.9 Hz, J_2 = 3.3 Hz, 1H), 3.89 (d, *J* = 4.0 Hz, 1H), 3.86 (s, 3H). **^{19}F NMR** (471 MHz, $CDCl_3$) δ 56.3 (s, 1F). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 164.7, 138.7, 131.5, 131.1,

128.5, 115.2, 55.9 (d, *J* = 3.0 Hz), 49.5 (d, *J* = 21.3 Hz). **HRMS-ESI** (m/z) calcd. for $[C_{10}H_{12}FO_5S_2]^+$ ($[M+H]^+$): 295.0105, found 295.0105.

5. Scale-up reaction procedure for (3)



1 **2, CESF** **3**

A 250 mL oven-dried reaction tube equipped with a magnetic stirring bar was loaded with thiols (**1**, 10.0 mmol, 1.0 equiv.), CESF (**2**, 2.37 g, 15.0 mmol, 1.5 equiv.), and 80.0 mL of DCE. Potassium carbonate (2.07 g, 15.0 mmol, 1.5 equiv.) was then added drop wise to the solution. The reaction mixture was stirred at room temperature for 2 hours under an air atmosphere, with progress monitored by TLC. Once the reaction was complete, 50 mL of water was added, and the mixture was extracted three times with ethyl acetate (3 × 50 mL). The combined organic layers were dried over anhydrous sodium sulfate (Na_2SO_4) and concentrated under reduced pressure. The resulting residue was purified by column chromatography on silica gel using a mixture of petroleum ether and ethyl acetate as the eluent to obtain the desired product (**3**).

6. References

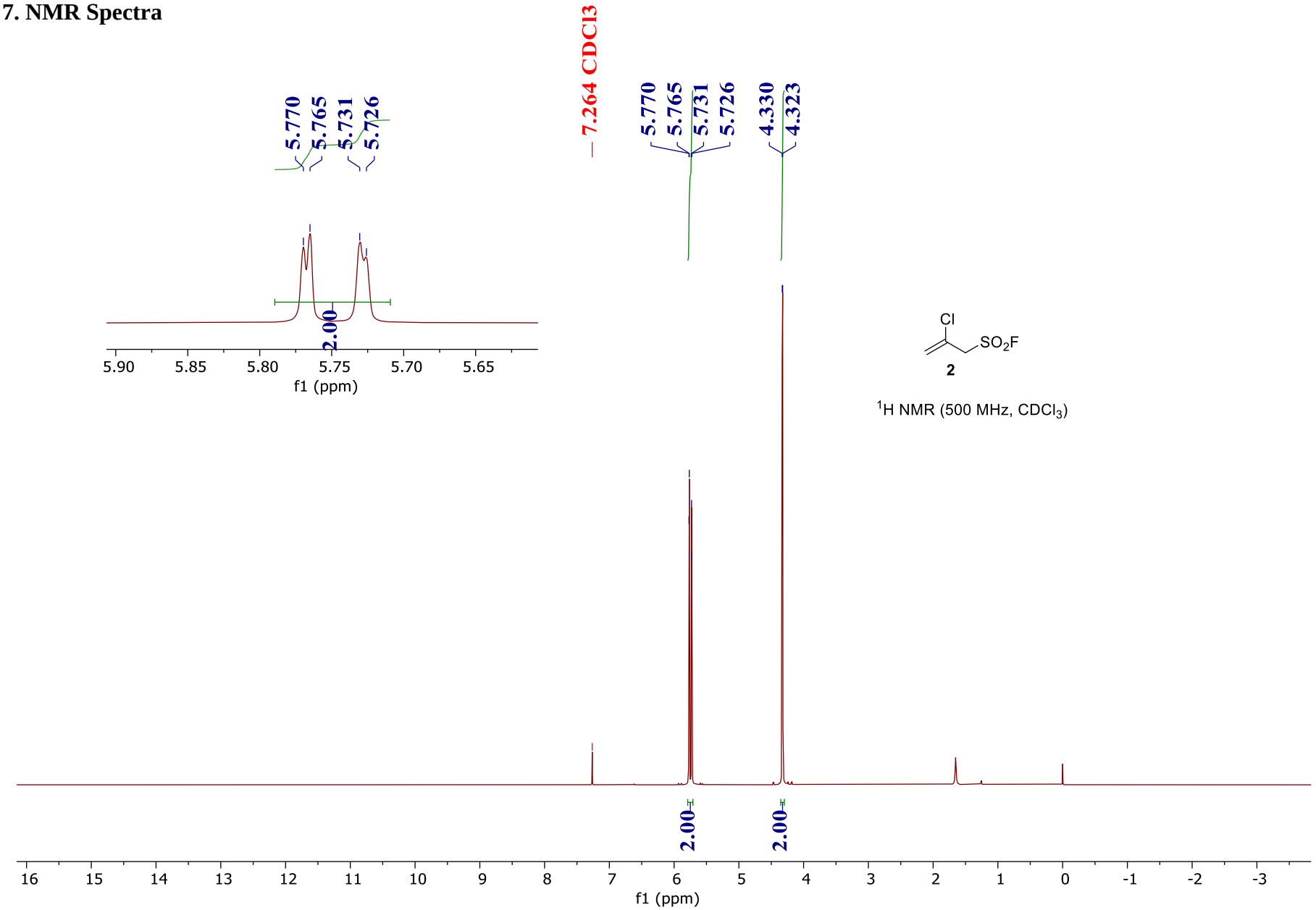
1 M. J. Brienne, D. Varech, M. Leclercq, J. Jacques, N. Radembino, C. Dessalles, G. Mahuzier, C. Gueyouche and C. Bories, *J. Med. Chem.*, 1987, **30**, 2232–2239.

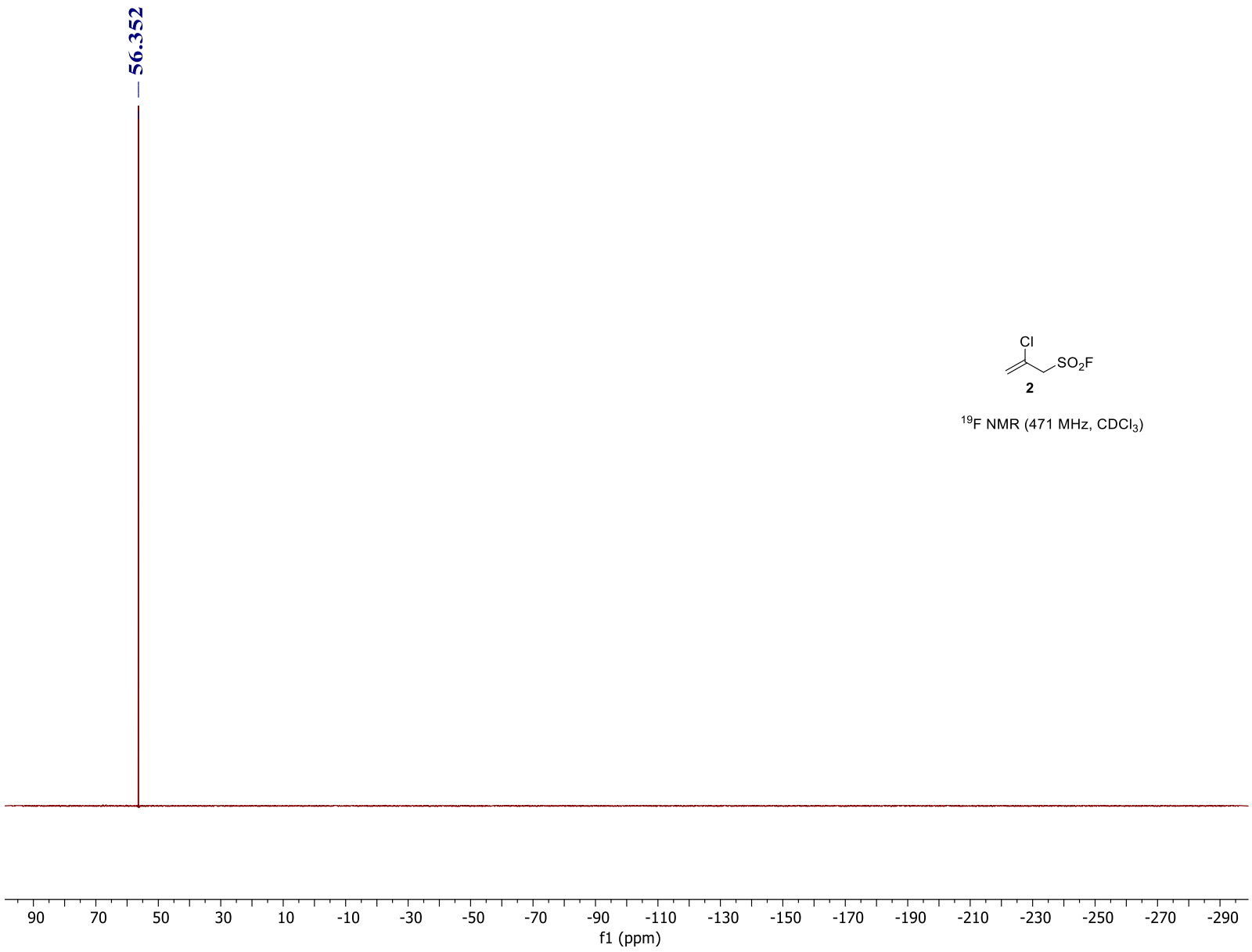
2 H. Xiong, J. Wu and H.-L. Qin, *Org. Chem. Front.*, 2023, **10**, 342–347.

3 K. Zhao, Q. Liu, S. Cheng, Z. Zhao and X. Li, *J. Org. Chem.*, 2023, **88**, 15626–15638.

4 R. Kupwade, S. Khot, U. Lad, U. Desai and P. Wadgaonkar, *Res. Chem. Intermed.*, 2017, **43**, 6875–6888.

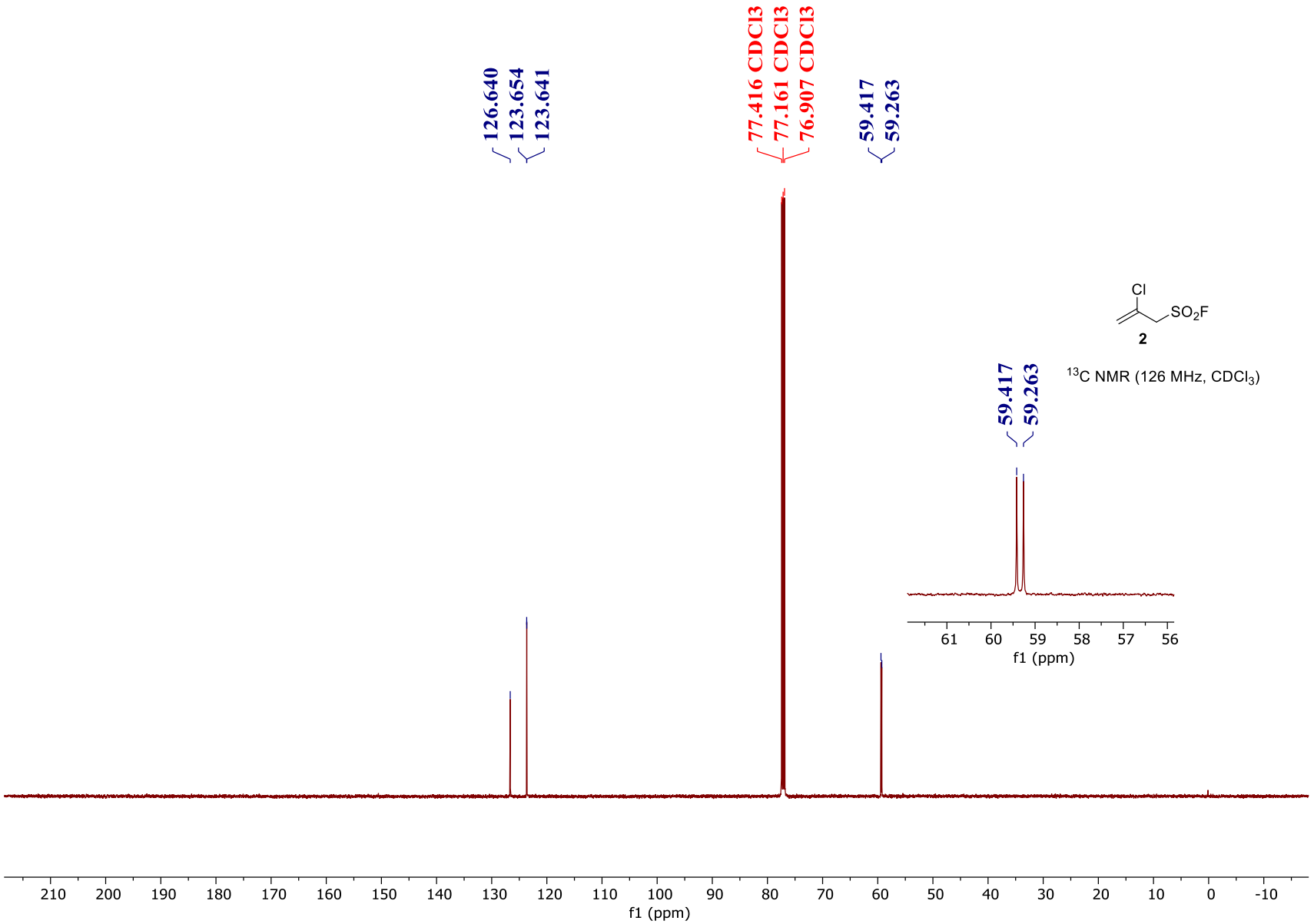
7. NMR Spectra

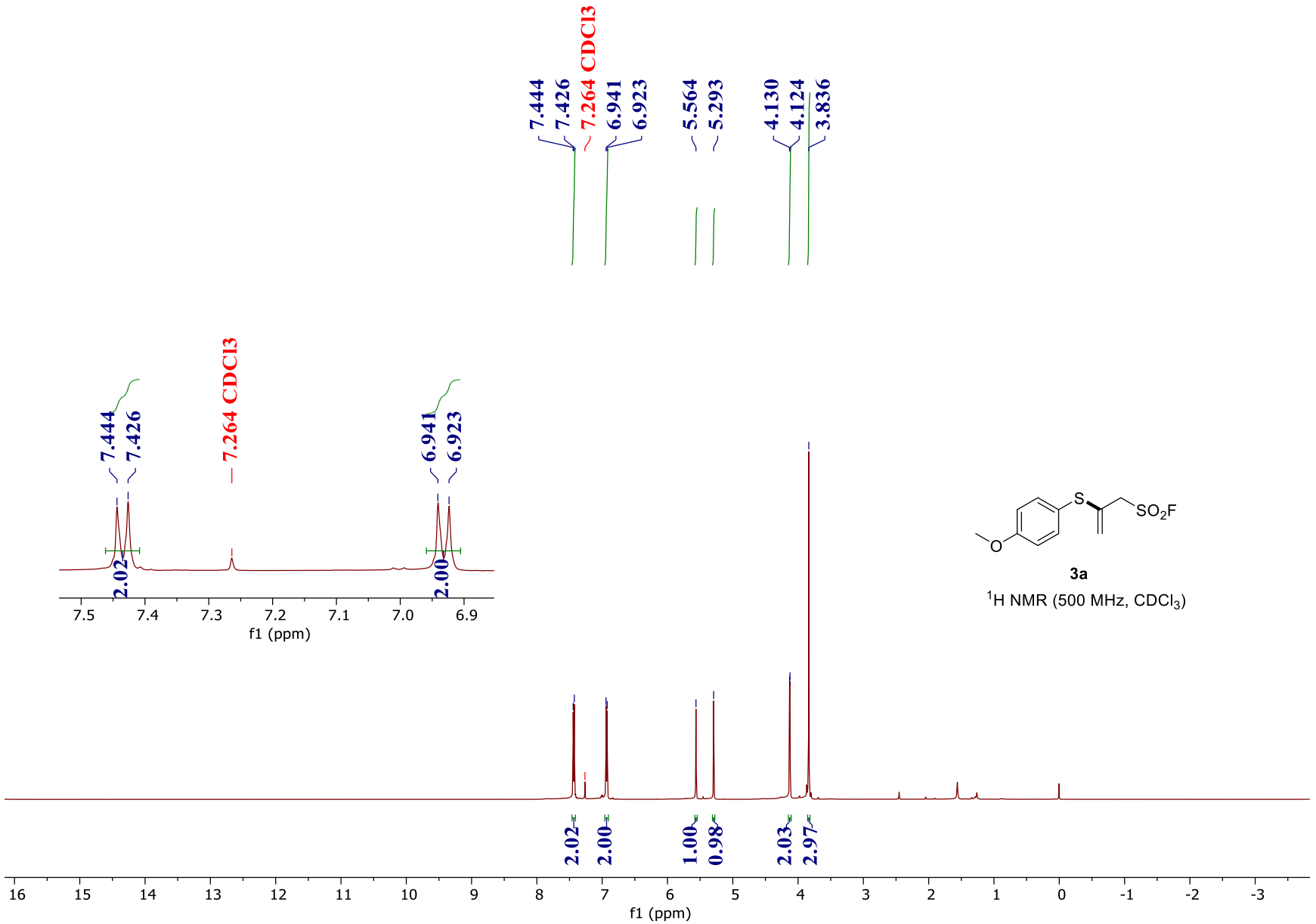


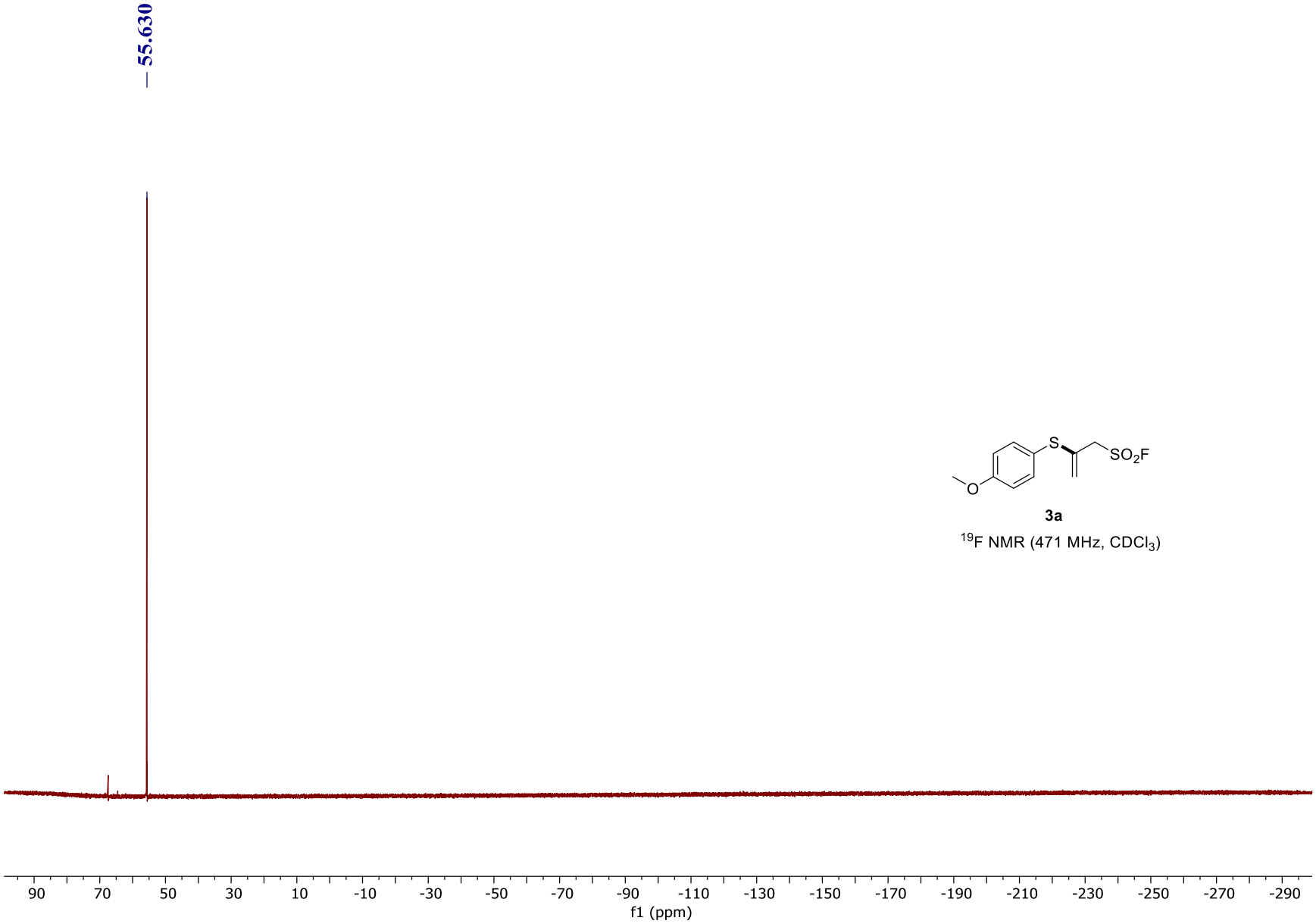


2

^{19}F NMR (471 MHz, $CDCl_3$)

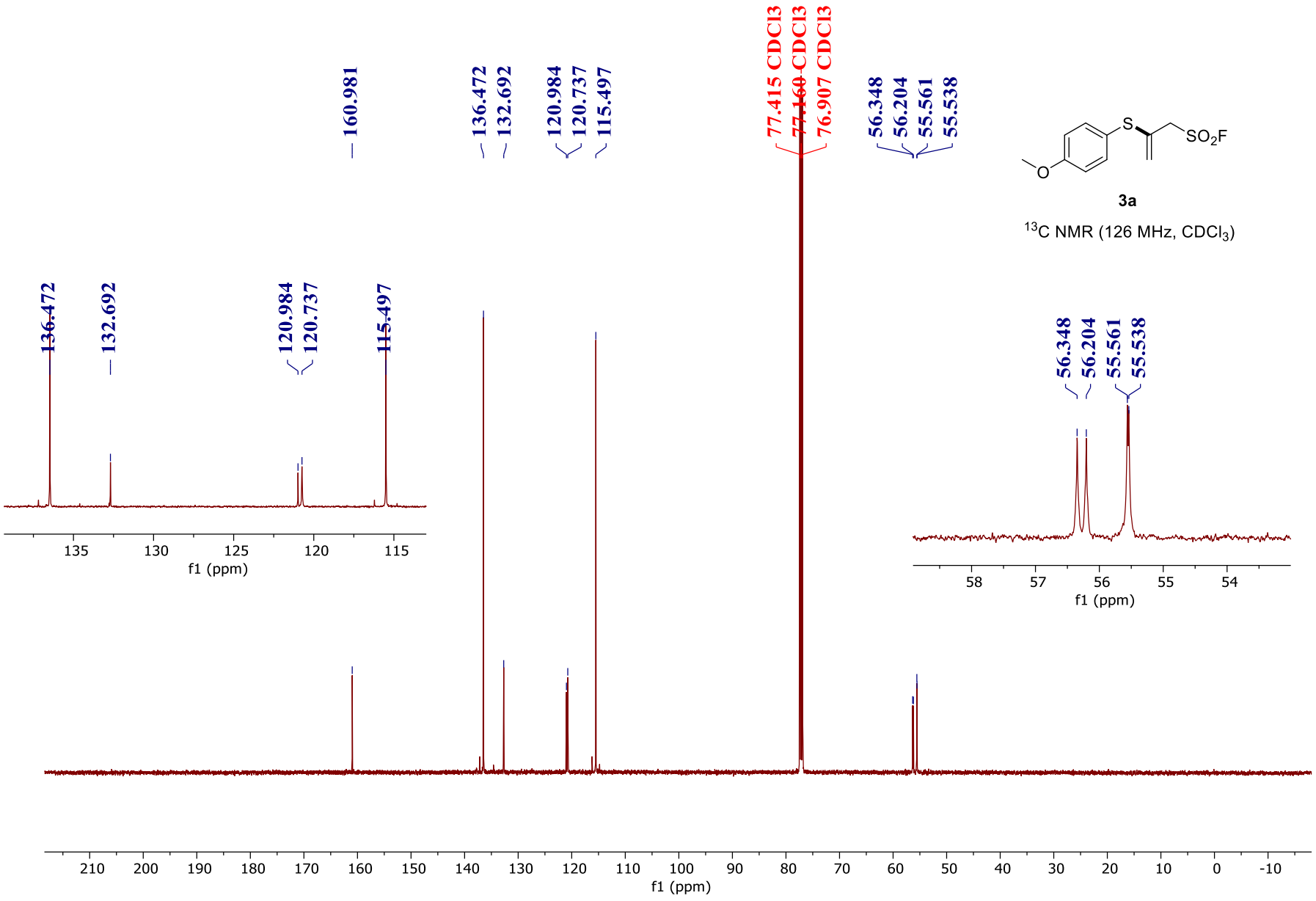

126.640
123.654
123.641
77.416 CDCl3
77.161 CDCl3
76.907 CDCl3
59.417
59.263
Cl
SO2F
2
13C NMR (126 MHz, CDCl3)
59.417
59.263
61 60 59 58 57 56
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

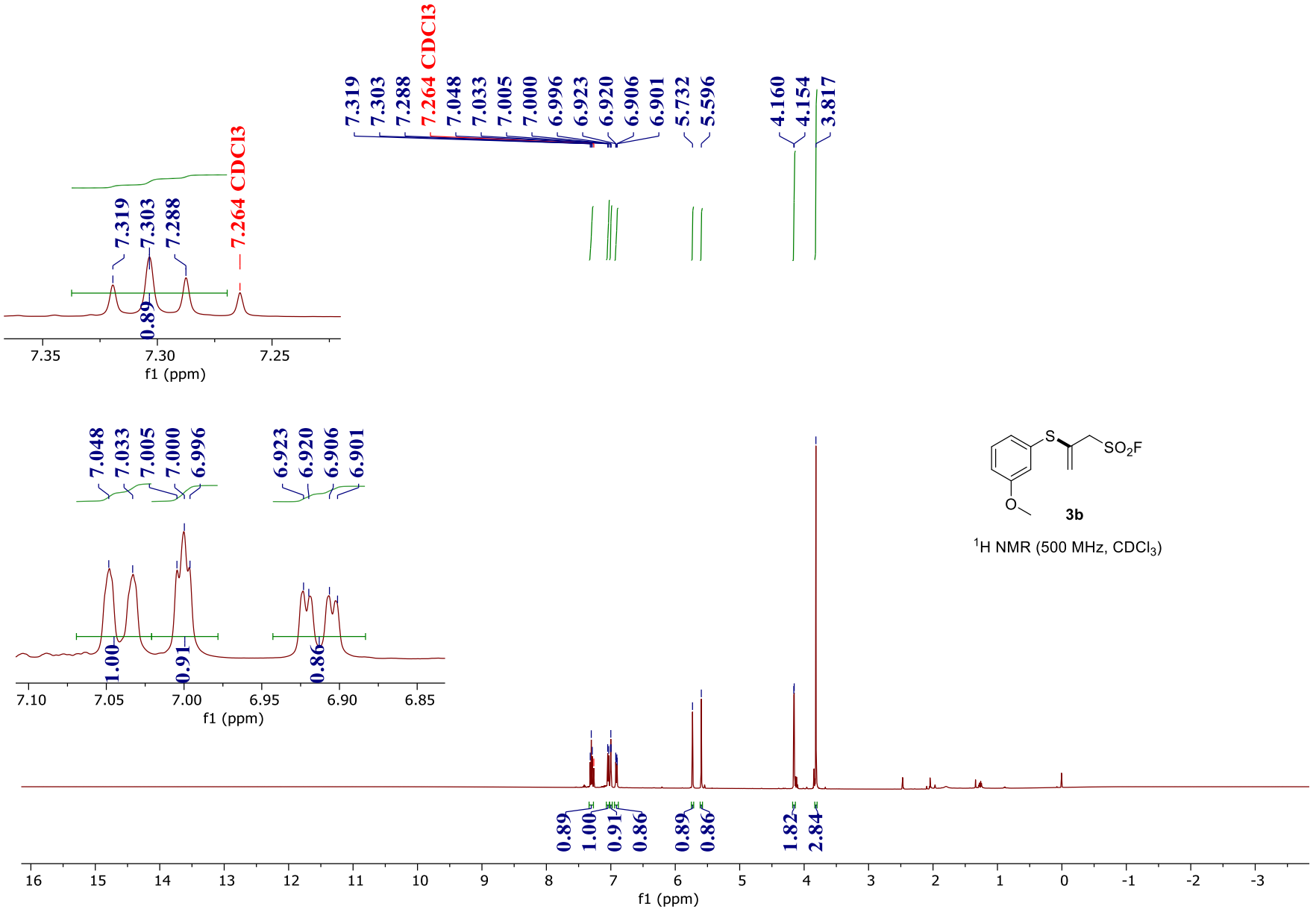

7.444
7.426
7.264 CDCl3
6.941
6.923
5.564
5.293
4.130
4.124
3.836
2.02
2.00
1.00
0.98
2.03
2.97
f1 (ppm)
S
SO2F
O
3a
1H NMR (500 MHz, CDCl3)

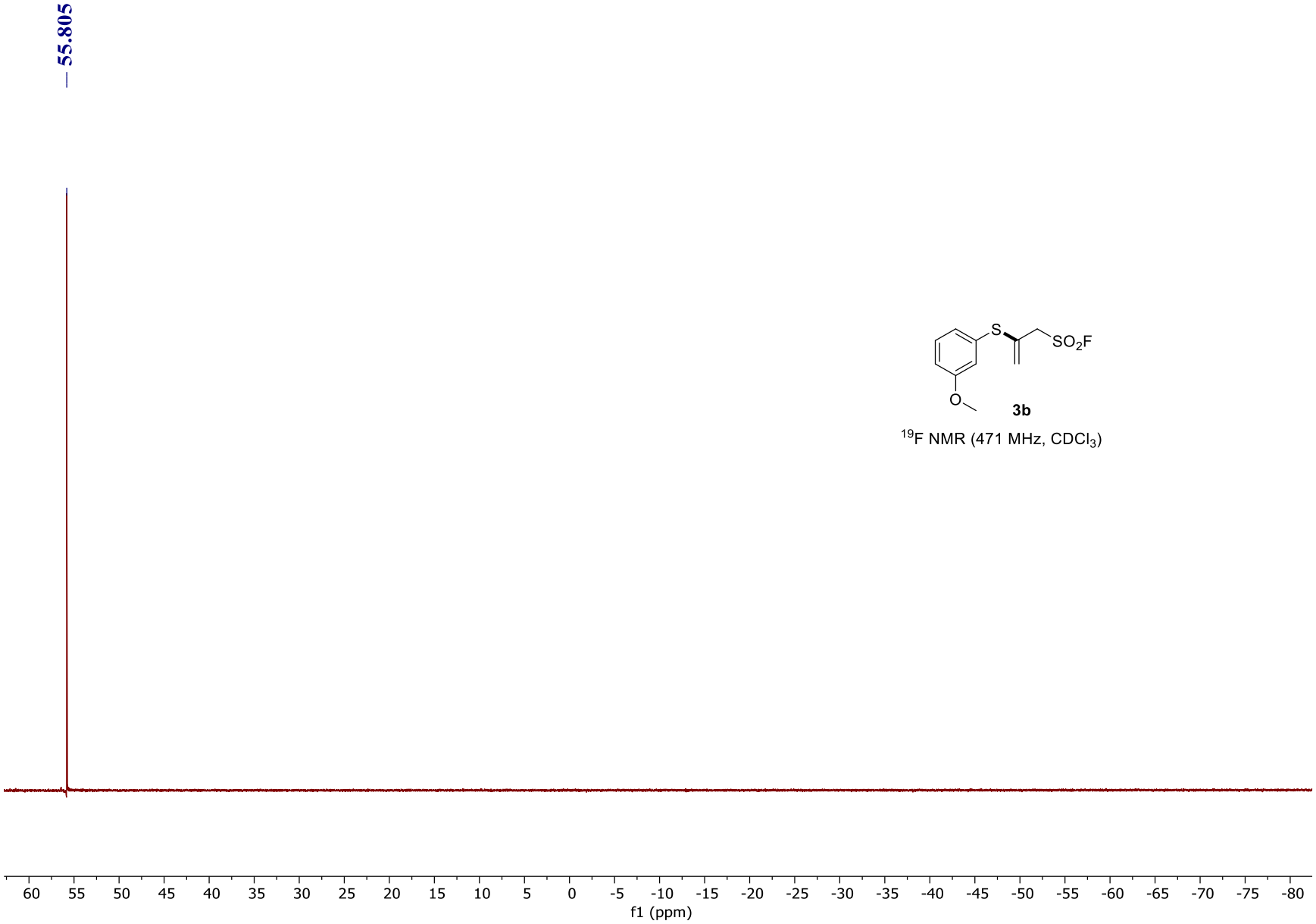


3a

^{19}F NMR (471 MHz, $CDCl_3$)

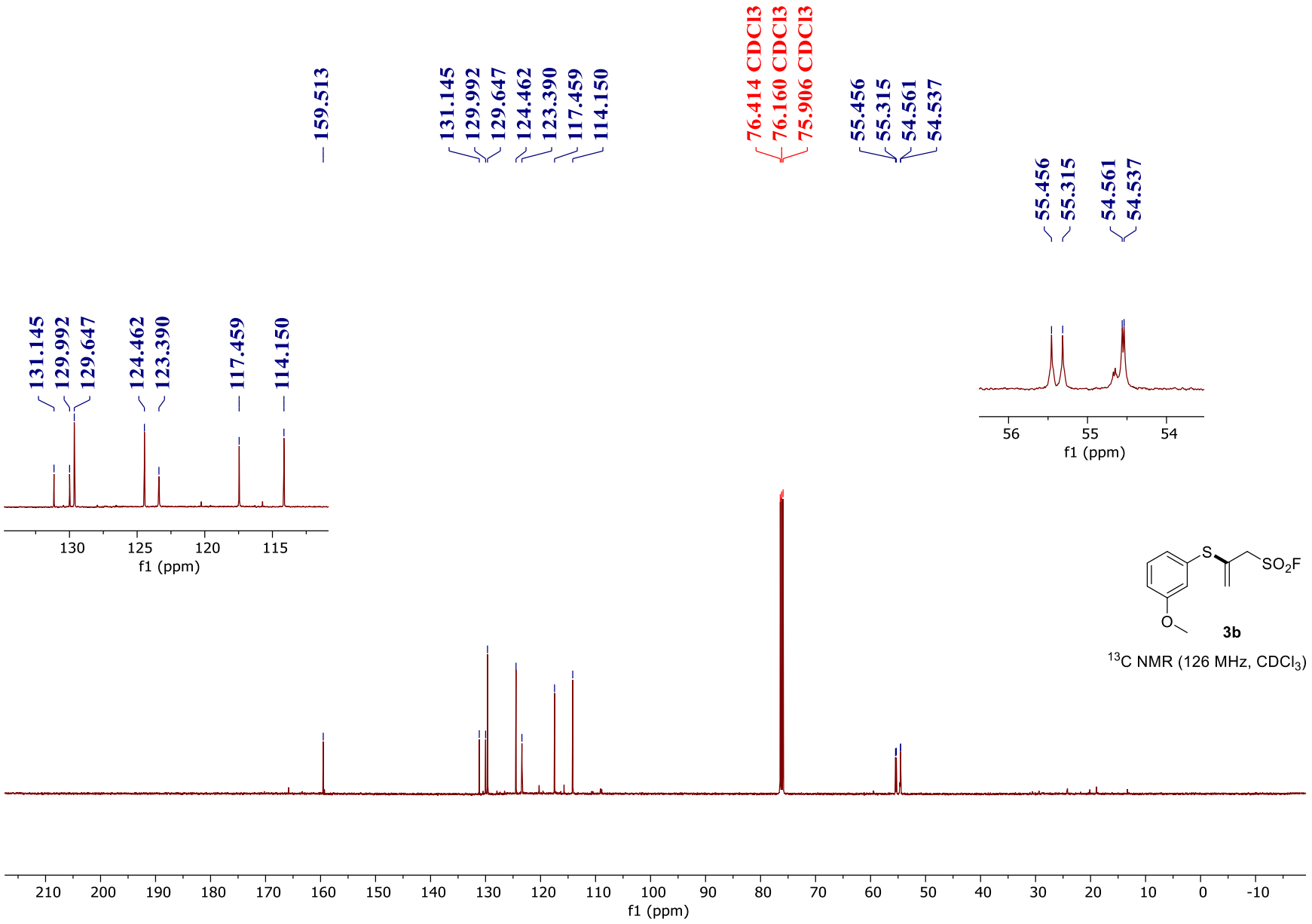

160.981
136.472
132.692
120.984
120.737
115.497
77.415 CDCl3
77.160 CDCl3
76.907 CDCl3
56.348
56.204
55.561
55.538
S
SO2F
O
3a
13C NMR (126 MHz, CDCl3)
f1 (ppm)

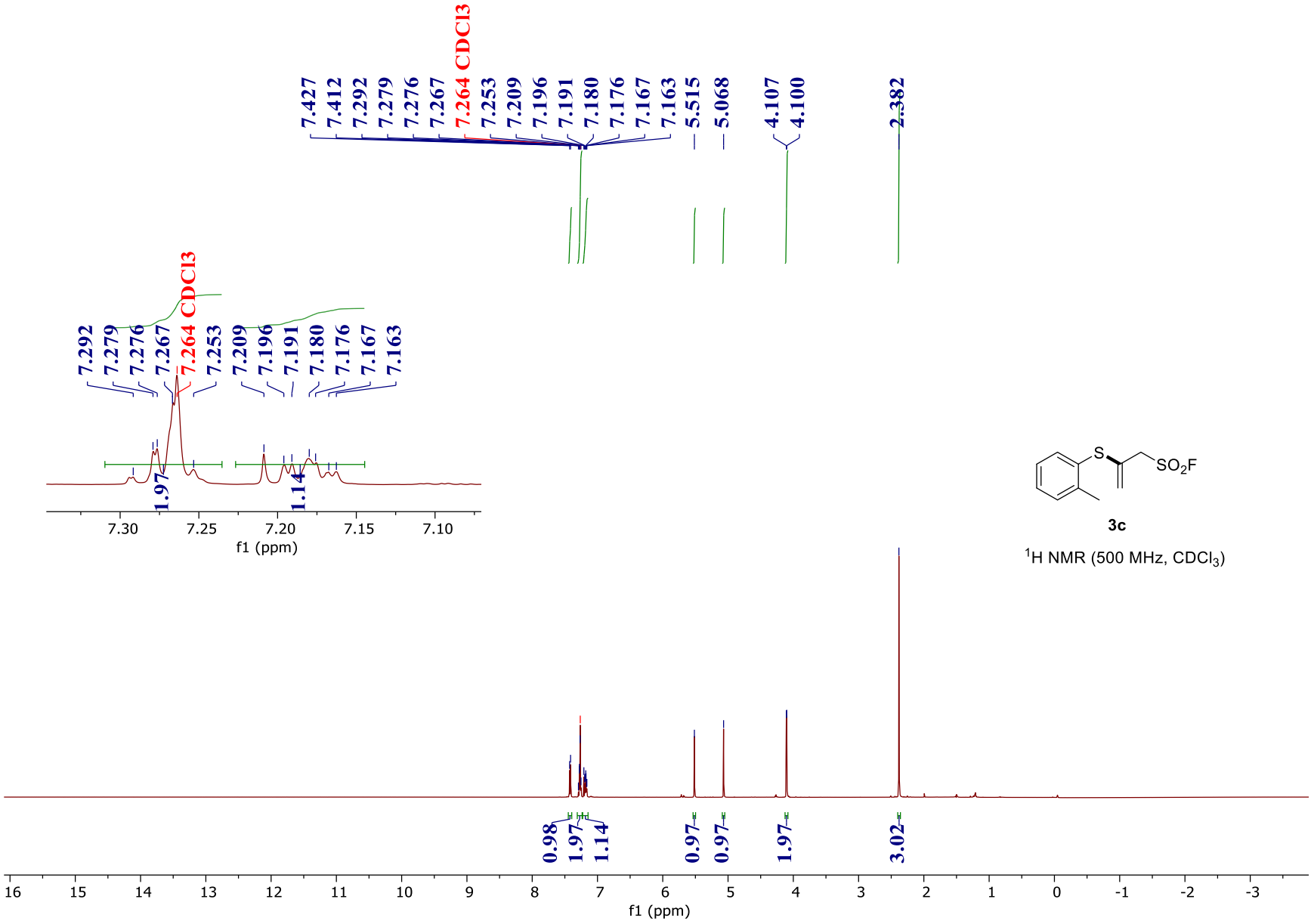

7.319
7.303
7.288
7.264 CDCl3
7.048
7.033
7.005
7.000
6.996
6.923
6.920
6.906
6.901
5.732
5.596
4.160
4.154
3.817
0.89
7.35
7.30
7.25
f1 (ppm)
1.00
0.91
0.86
7.10
7.05
7.00
6.95
6.90
6.85
f1 (ppm)
0.89
1.00
0.91
0.86
0.89
0.86
1.82
2.84
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)
S
SO2F
O
3b
1H NMR (500 MHz, CDCl3)

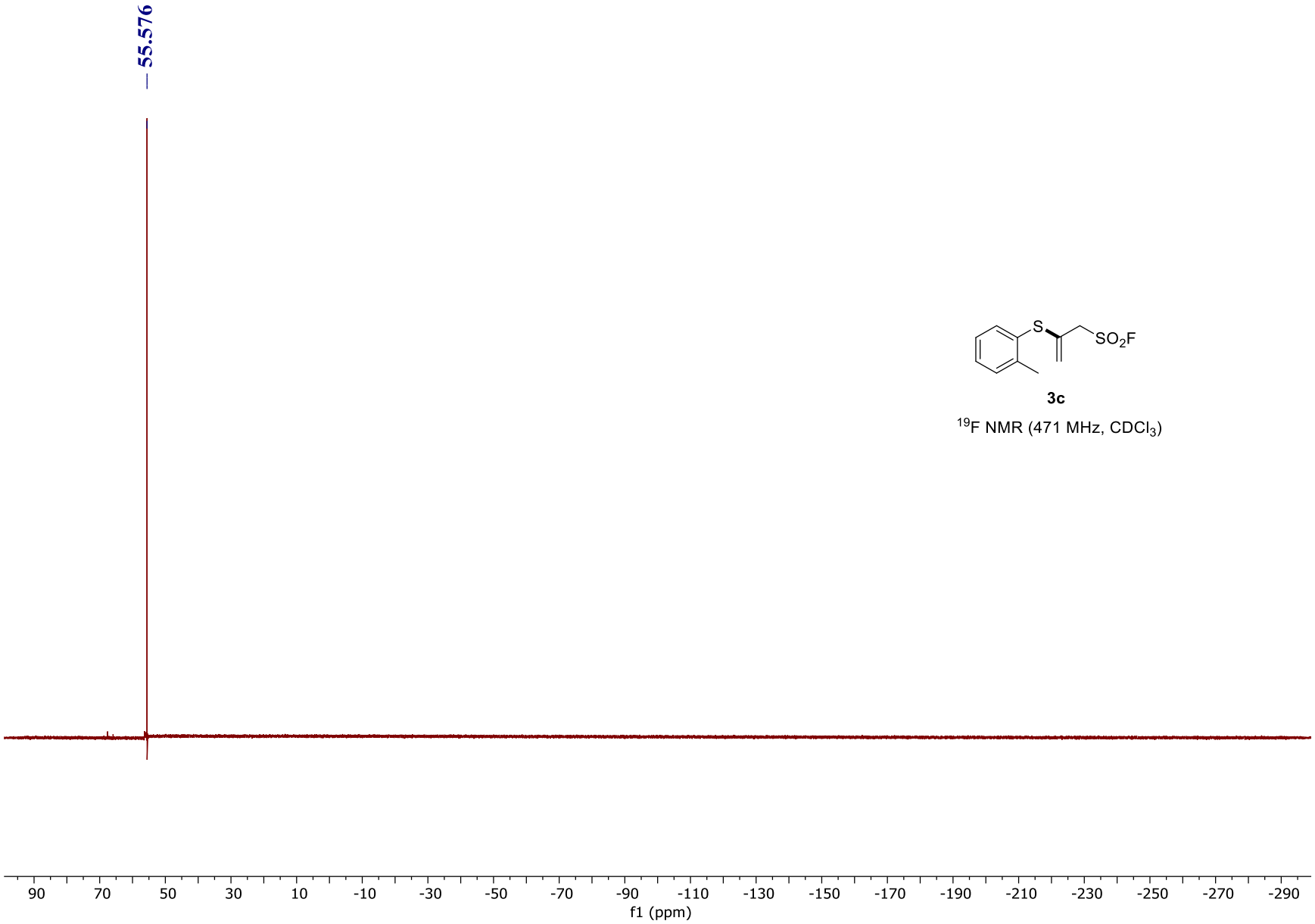


3b

^{19}F NMR (471 MHz, $CDCl_3$)

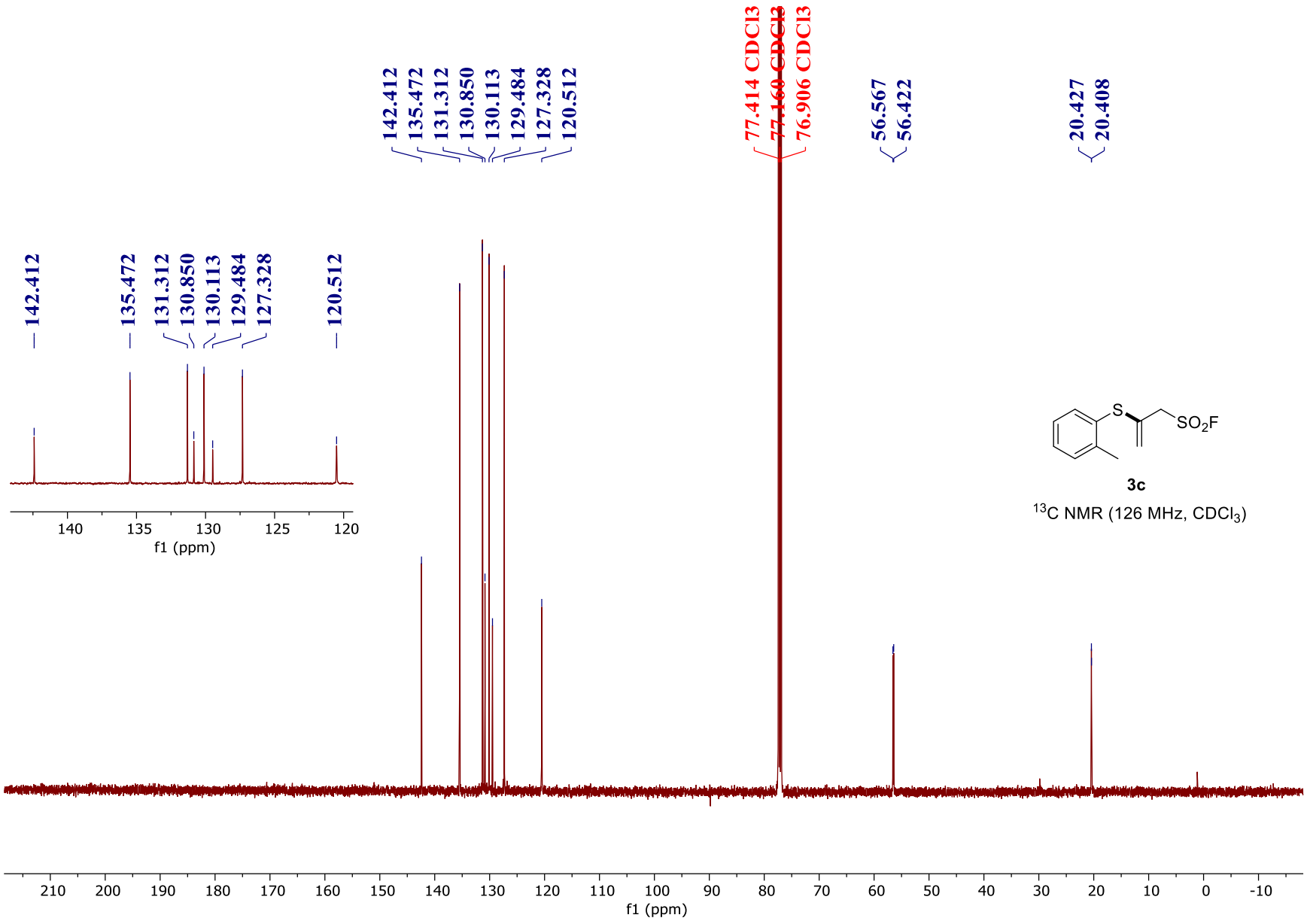

159.513
131.145
129.992
129.647
124.462
123.390
117.459
114.150
76.414 CDCl3
76.160 CDCl3
75.906 CDCl3
55.456
55.315
54.561
54.537
f1 (ppm)
SO2F
3b
13C NMR (126 MHz, CDCl3)

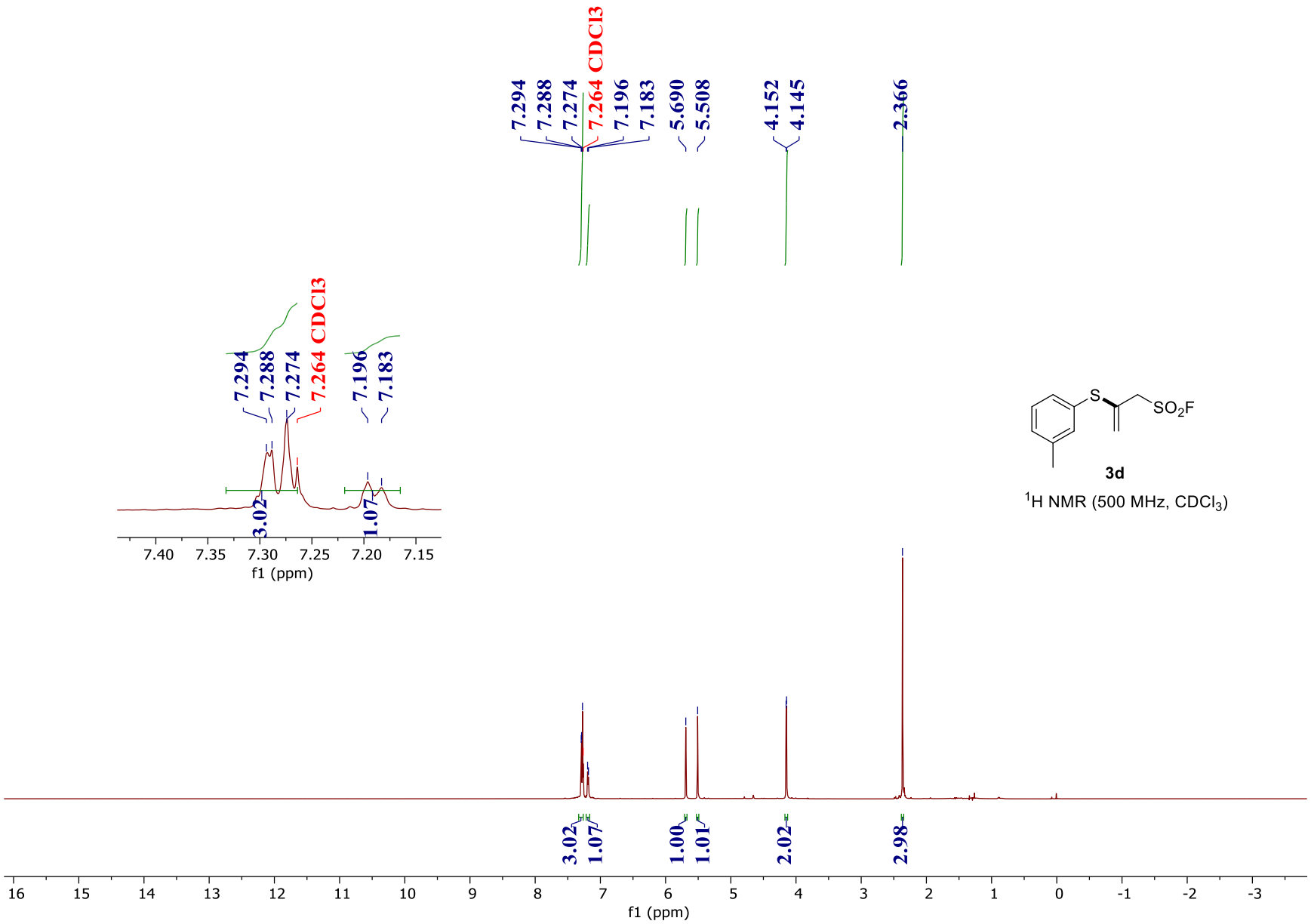

7.427
7.412
7.292
7.279
7.276
7.267
7.264 CDCl3
7.253
7.209
7.196
7.191
7.180
7.176
7.167
7.163
5.515
5.068
4.107
4.100
2.382
7.292
7.279
7.276
7.267
7.264 CDCl3
7.253
7.209
7.196
7.191
7.180
7.176
7.167
7.163
1.97
1.14
7.30
7.25
7.20
7.15
7.10
f1 (ppm)
S
SO2F
3c
1H NMR (500 MHz, CDCl3)
0.98
1.97
1.14
0.97
0.97
1.97
3.02
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

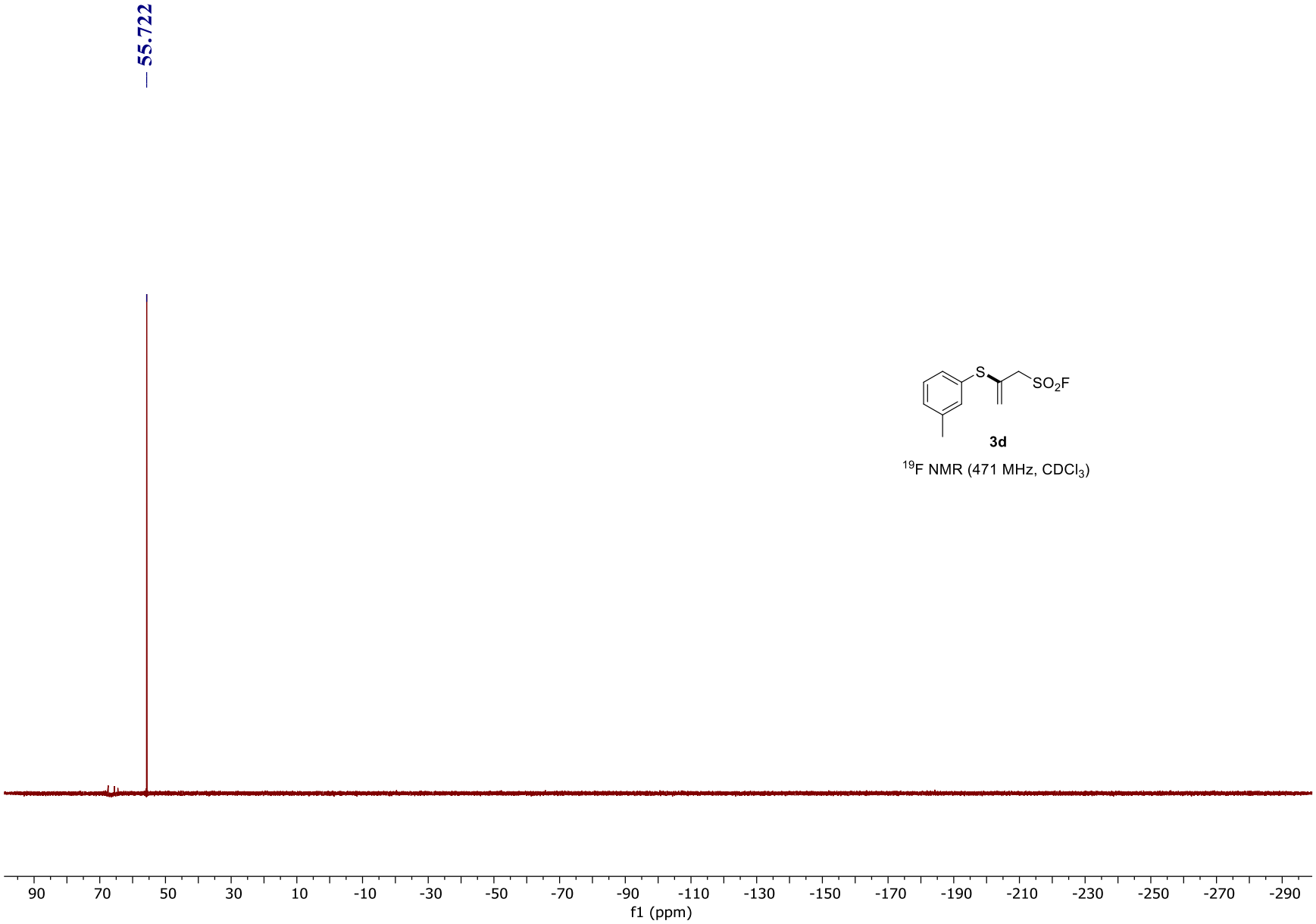


3c

^{19}F NMR (471 MHz, $CDCl_3$)

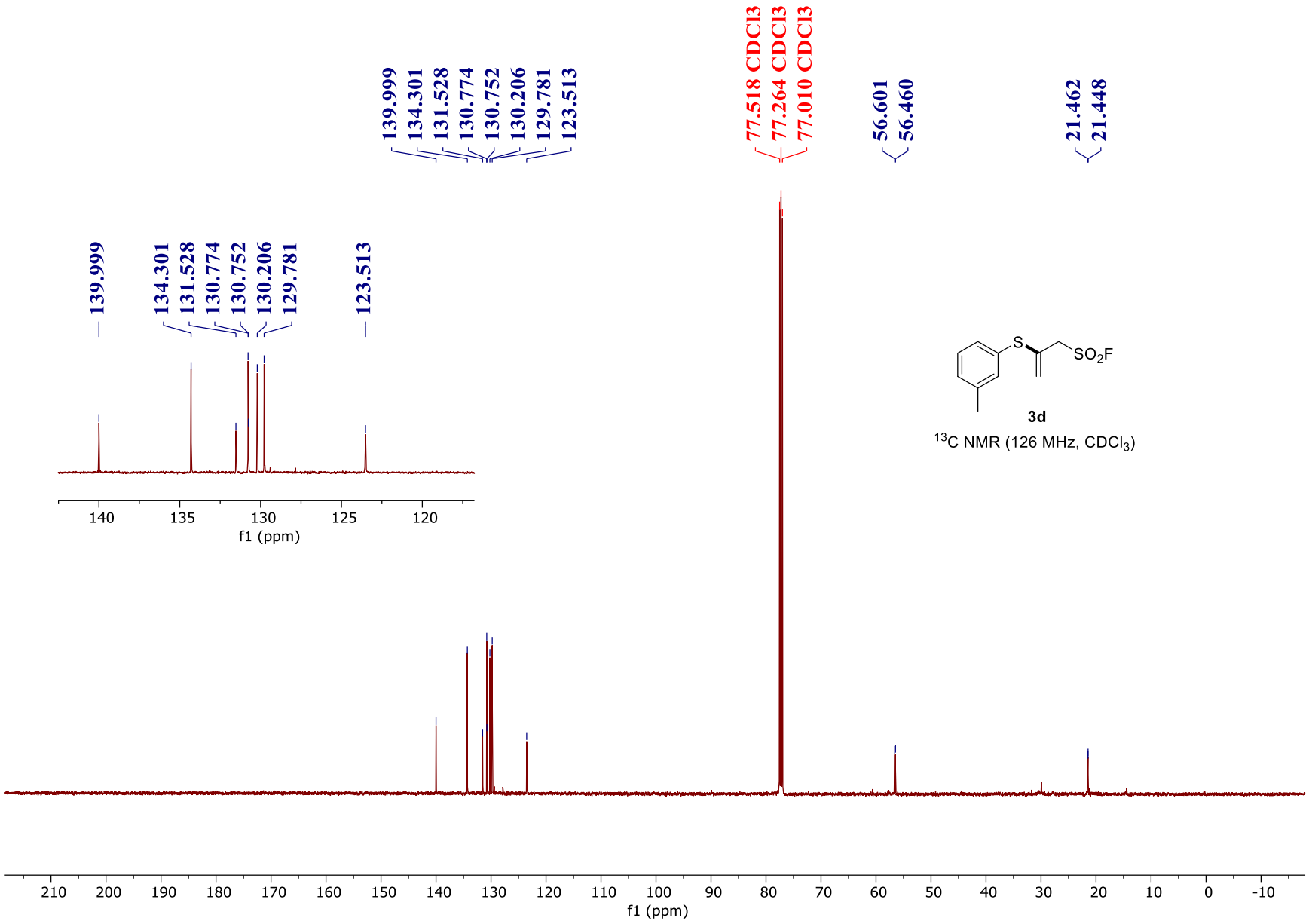

142.412
135.472
131.312
130.850
130.113
129.484
127.328
120.512
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
56.567
56.422
20.427
20.408
f1 (ppm)
S
SO2F
3c
13C NMR (126 MHz, CDCl3)

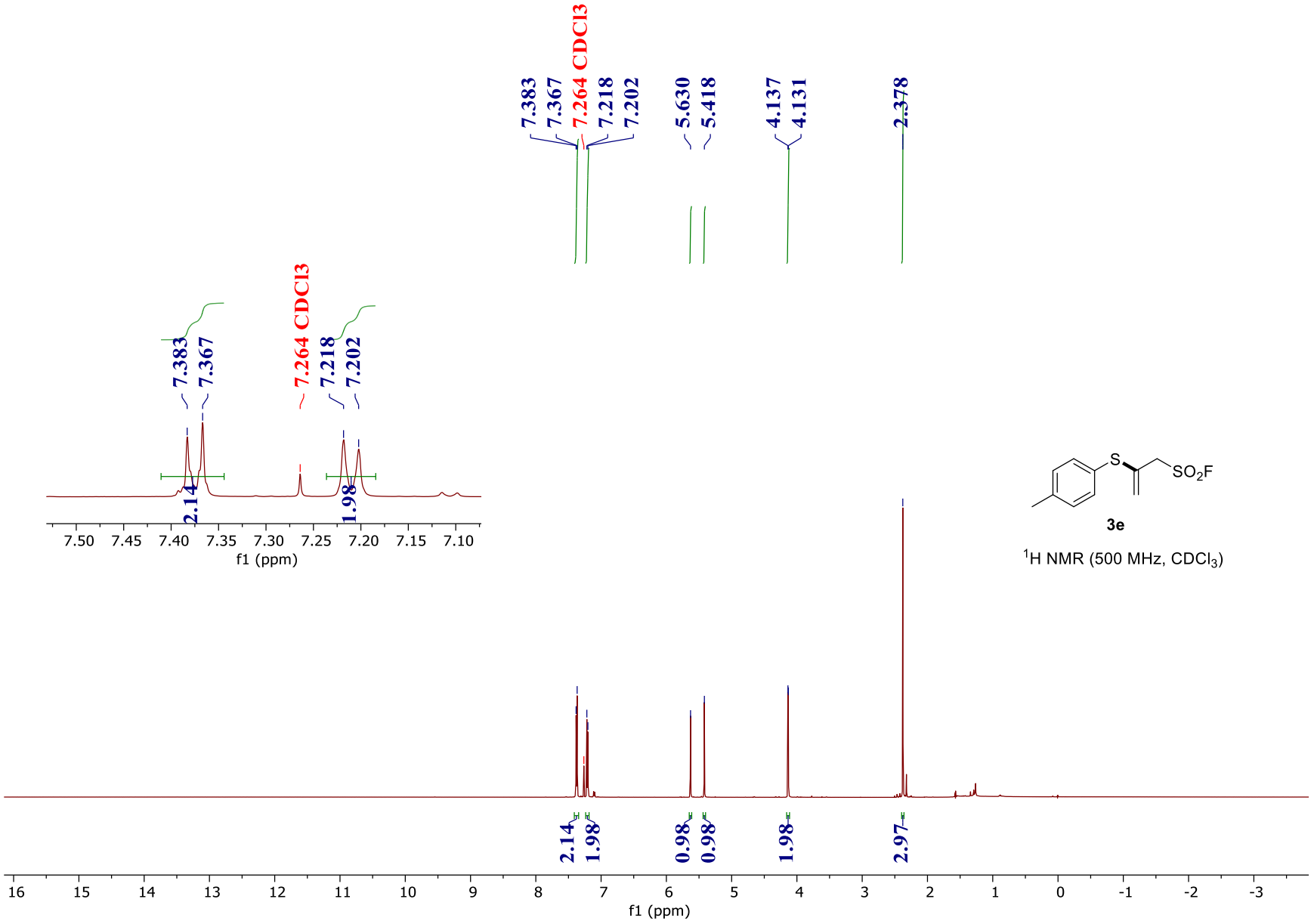

7.294
7.288
7.274
7.264 CDCl3
7.196
7.183
5.690
5.508
4.152
4.145
2.366
3.02
1.07
1.00
1.01
2.02
2.98
f1 (ppm)
SO2F
3d
1H NMR (500 MHz, CDCl3)

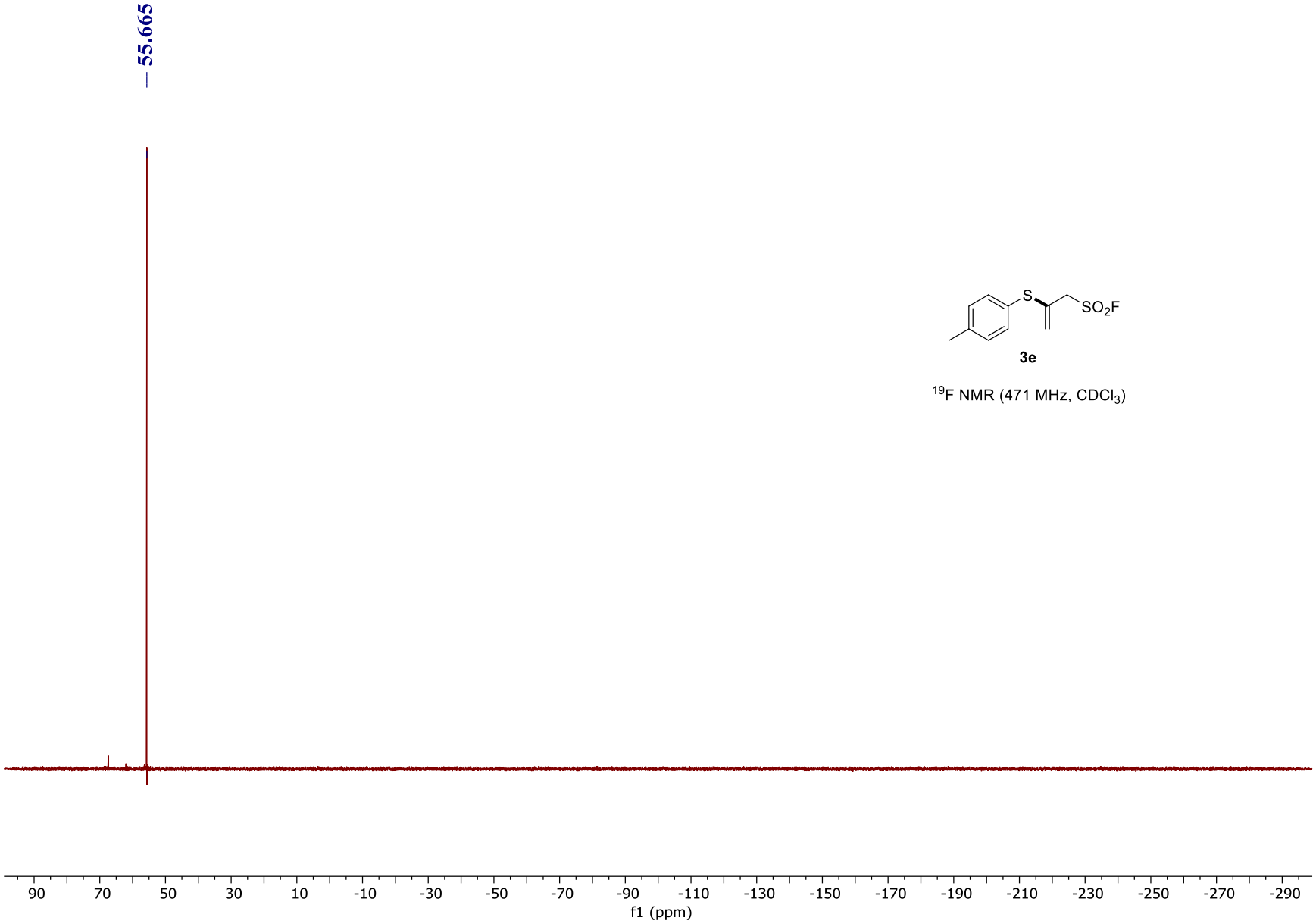


3d

^{19}F NMR (471 MHz, $CDCl_3$)

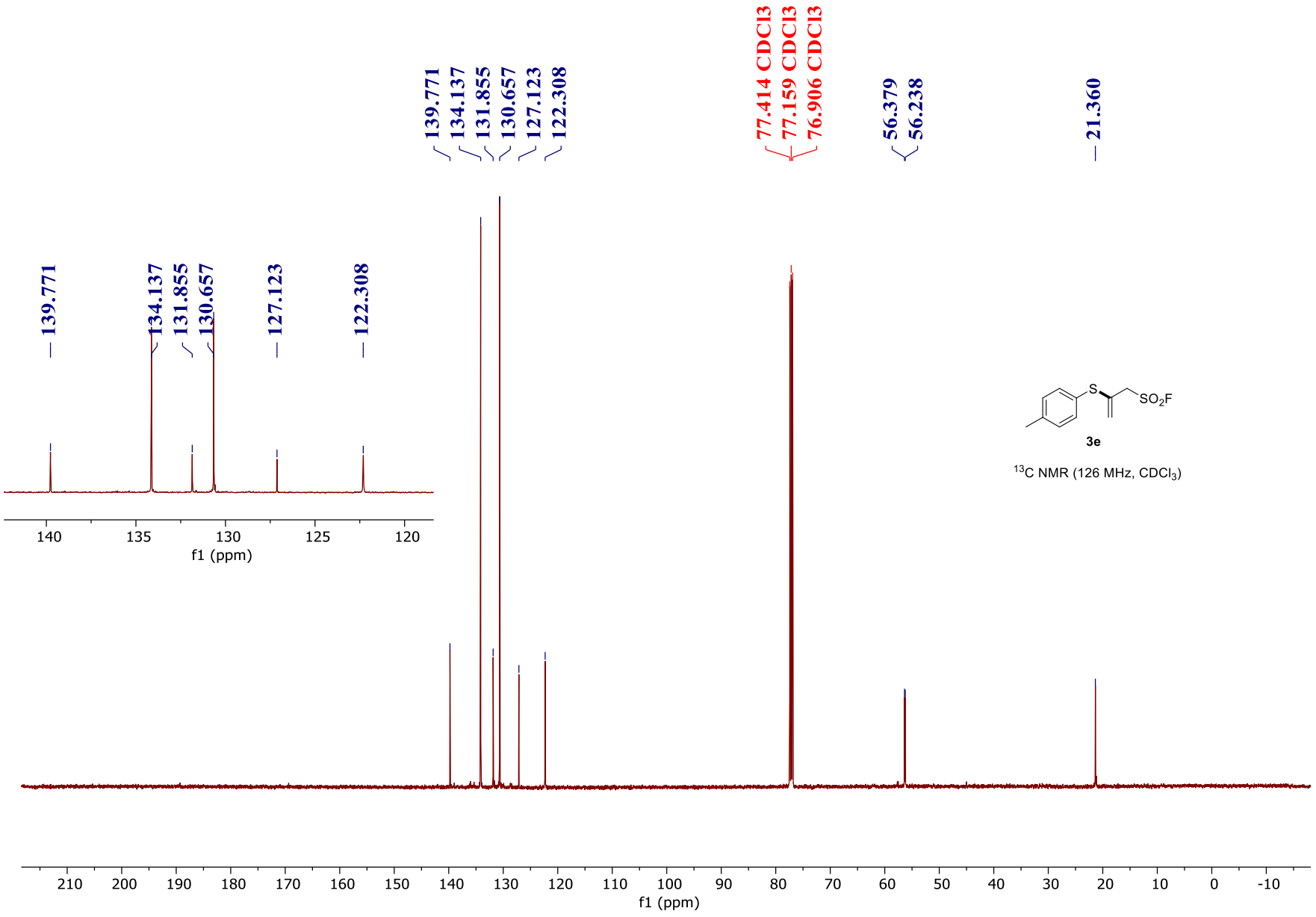

139.999
134.301
131.528
130.774
130.752
130.206
129.781
123.513
77.518 CDCl3
77.264 CDCl3
77.010 CDCl3
56.601
56.460
21.462
21.448
S
SO2F
3d
13C NMR (126 MHz, CDCl3)
f1 (ppm)

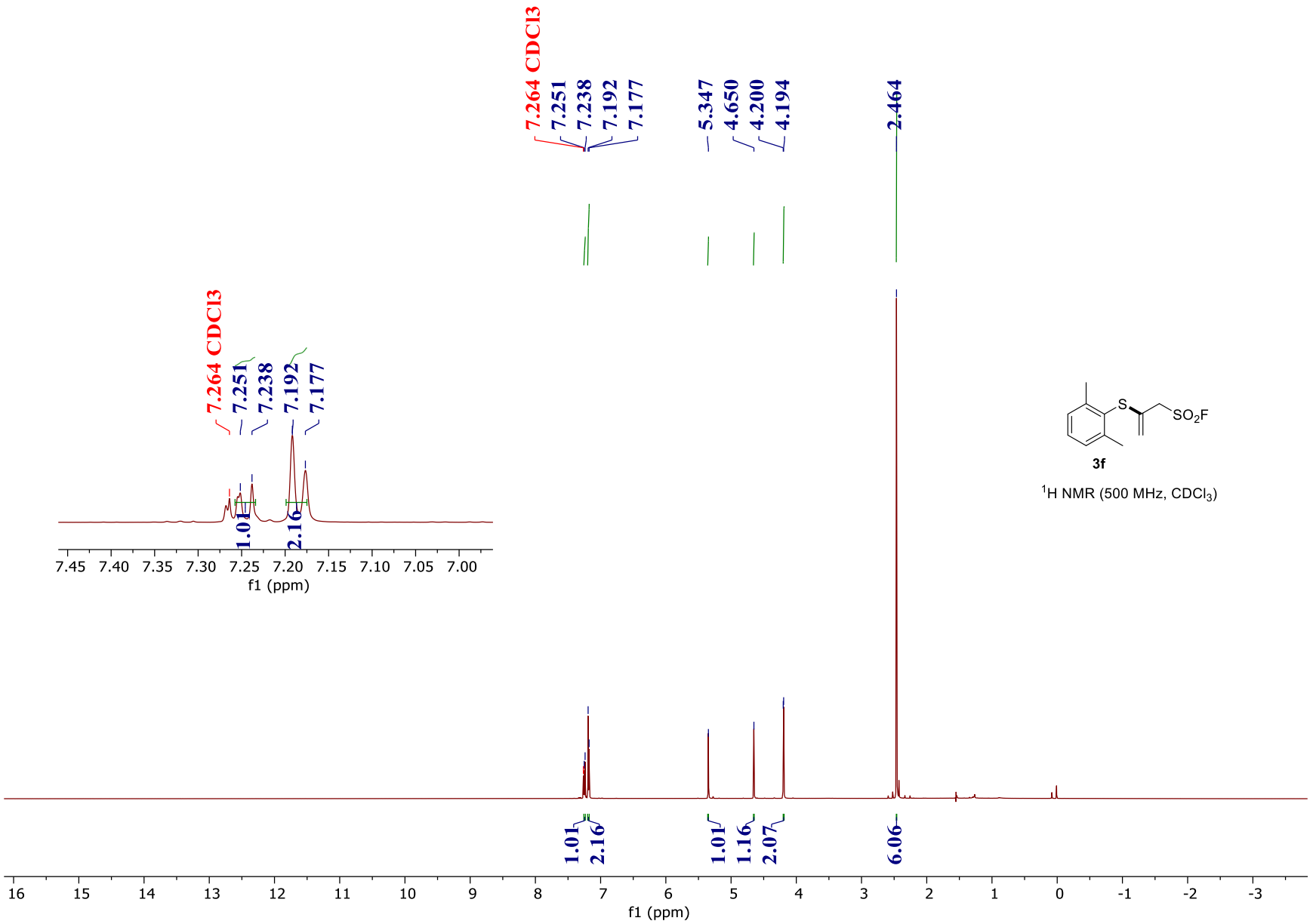

7.383
7.367
7.264 CDCl3
7.218
7.202
5.630
5.418
4.137
4.131
2.378
2.14
1.98
0.98
0.98
1.98
2.97
f1 (ppm)
S
SO2F
3e
1H NMR (500 MHz, CDCl3)

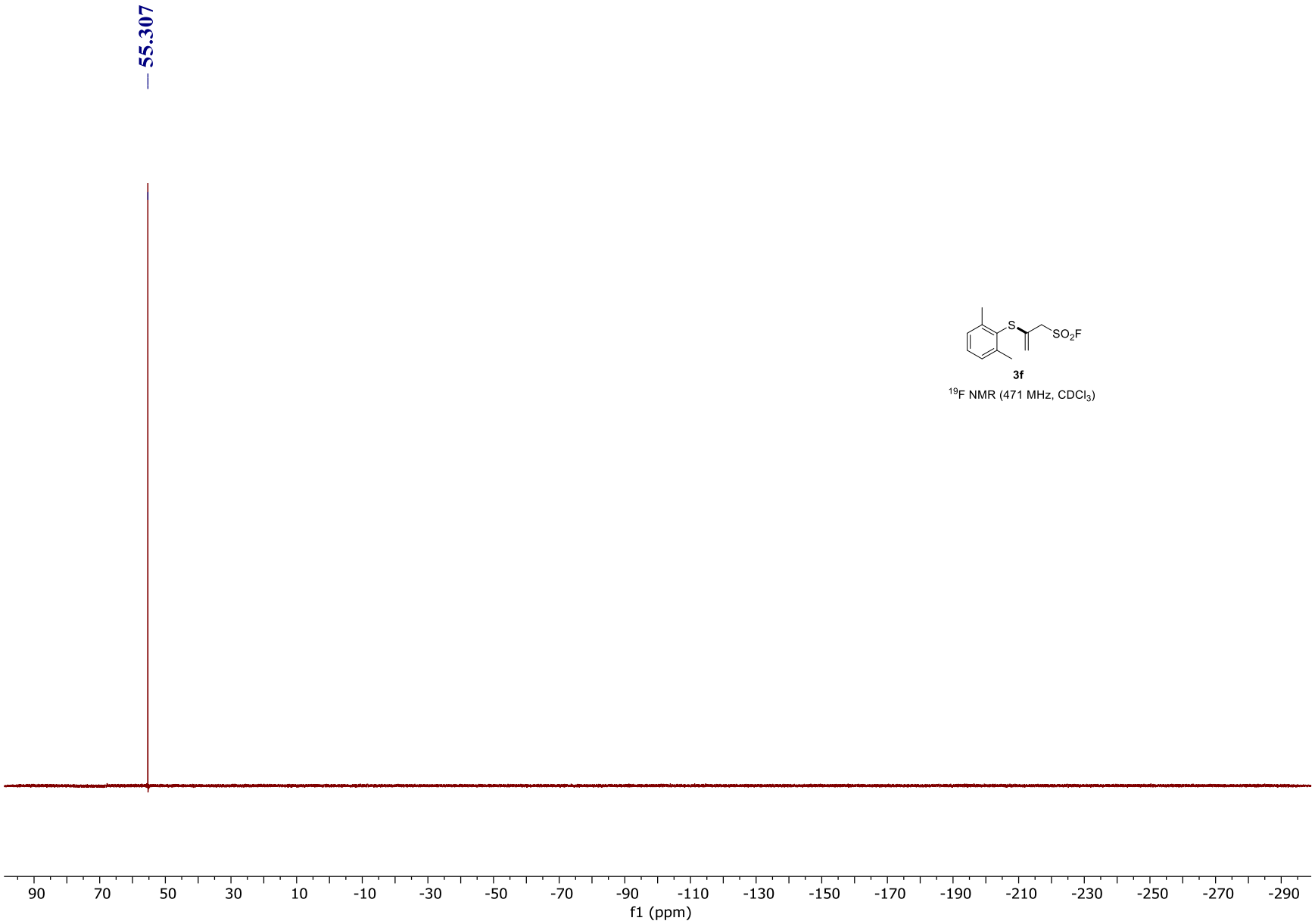


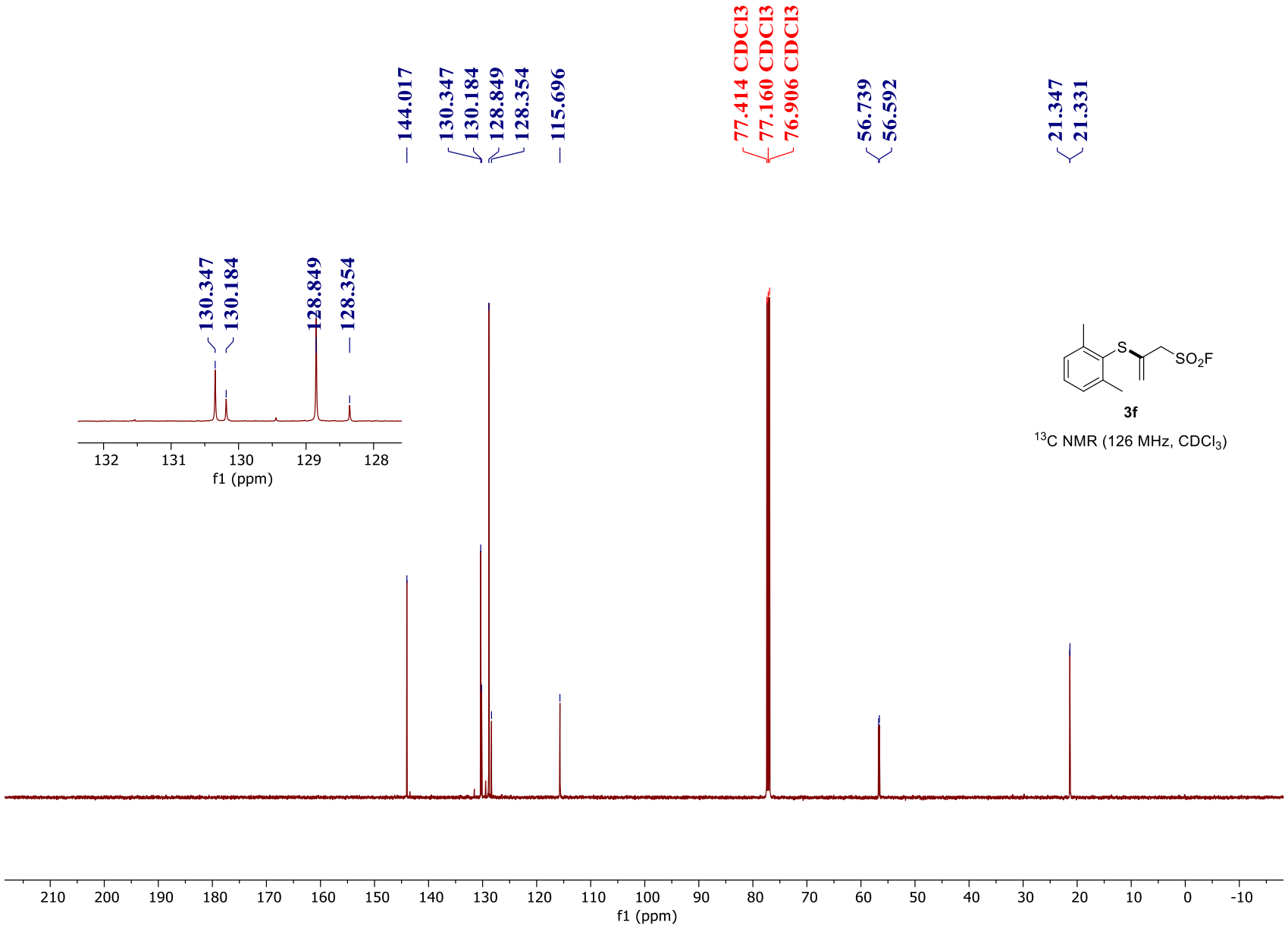
3e

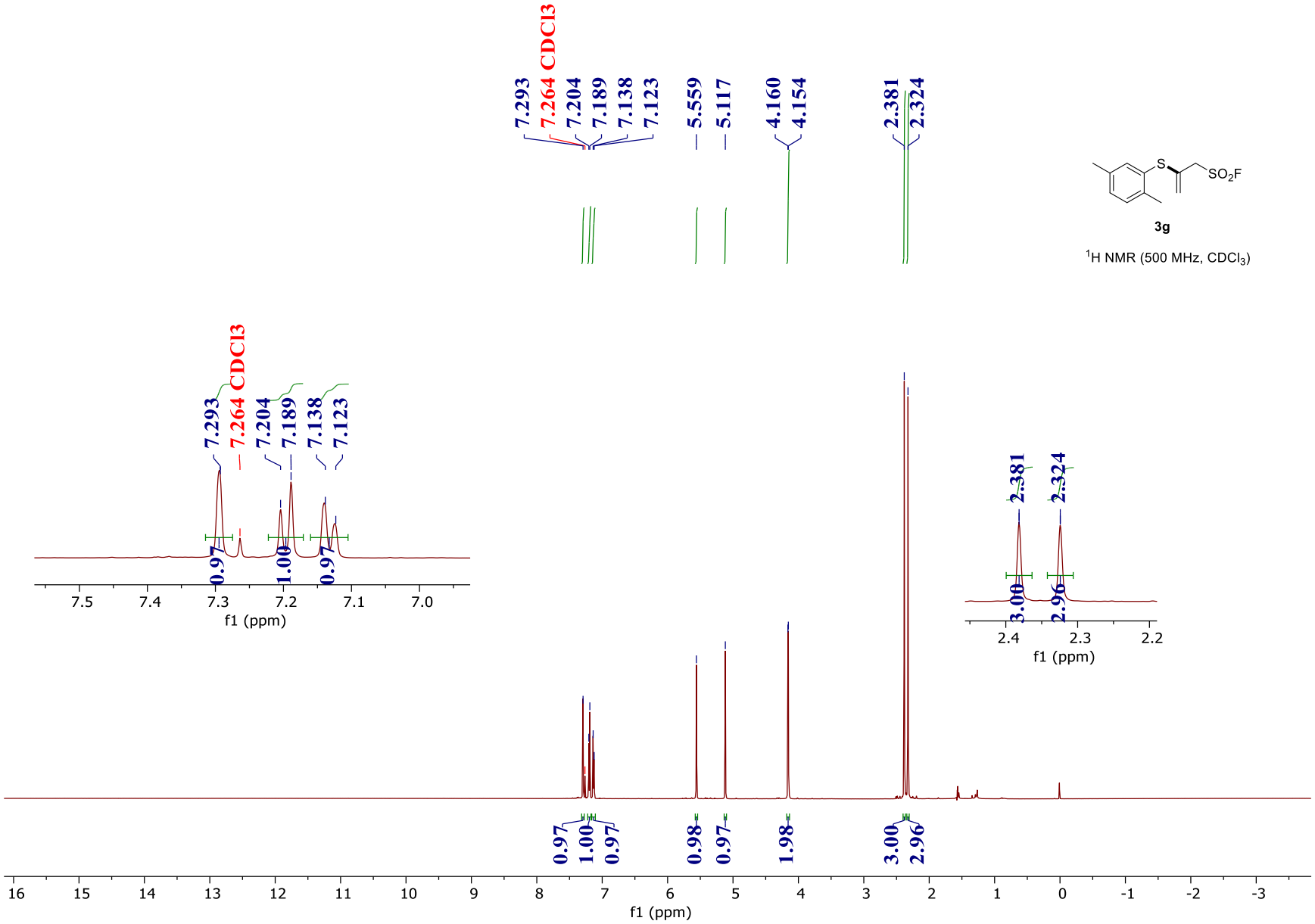
^{19}F NMR (471 MHz, $CDCl_3$)

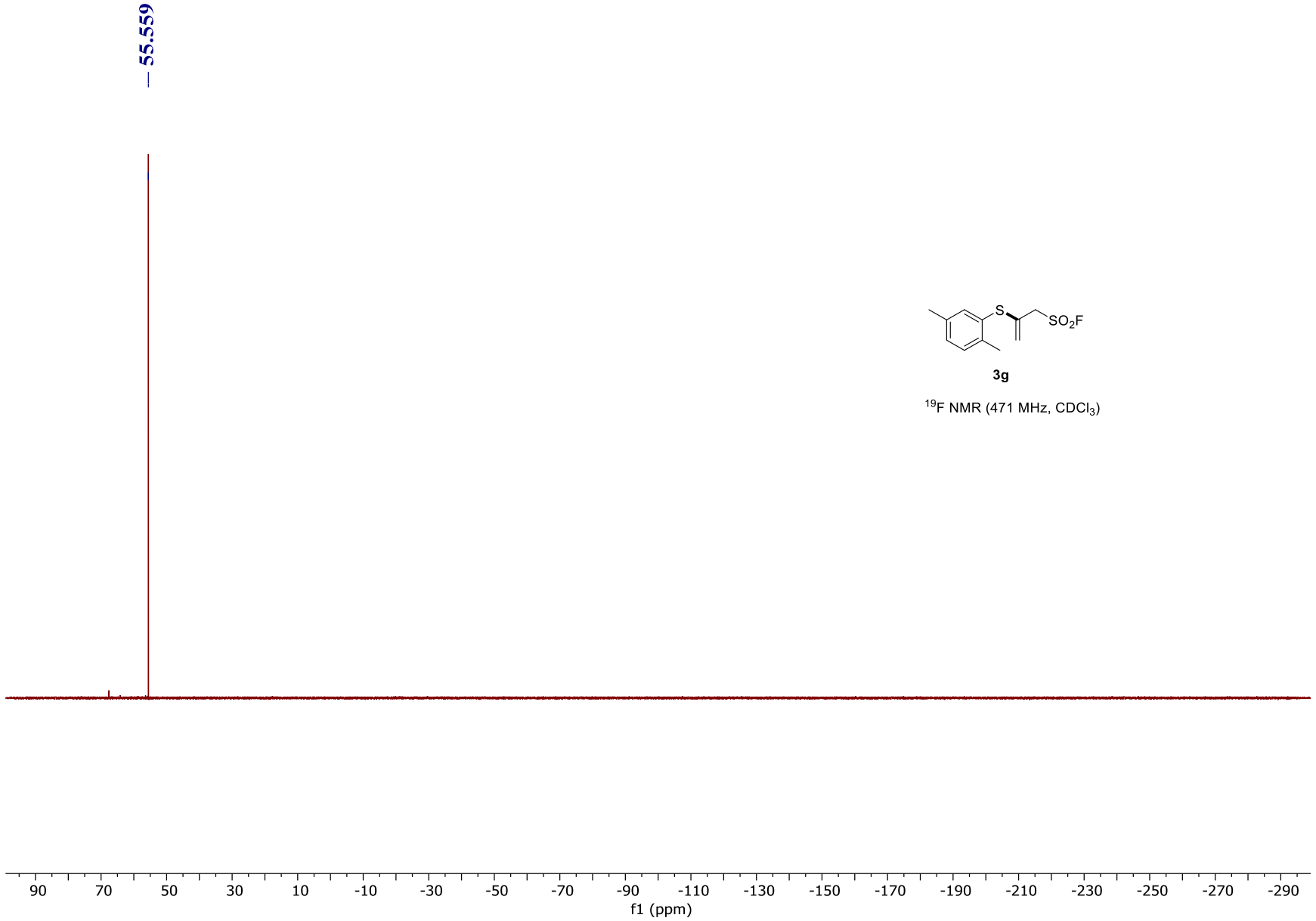

139.771
134.137
131.855
130.657
127.123
122.308
77.414 CDCl3
77.159 CDCl3
76.906 CDCl3
56.379
56.238
21.360
f1 (ppm)
SO2F
3e
13C NMR (126 MHz, CDCl3)


7.264 CDCl3
7.251
7.238
7.192
7.177
5.347
4.650
4.200
4.194
2.464
1.01
2.16
1.01
1.16
2.07
6.06
f1 (ppm)
7.45 7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00
f1 (ppm)
S
SO2F
3f
1H NMR (500 MHz, CDCl3)


55.307
S
SO2F
3f
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

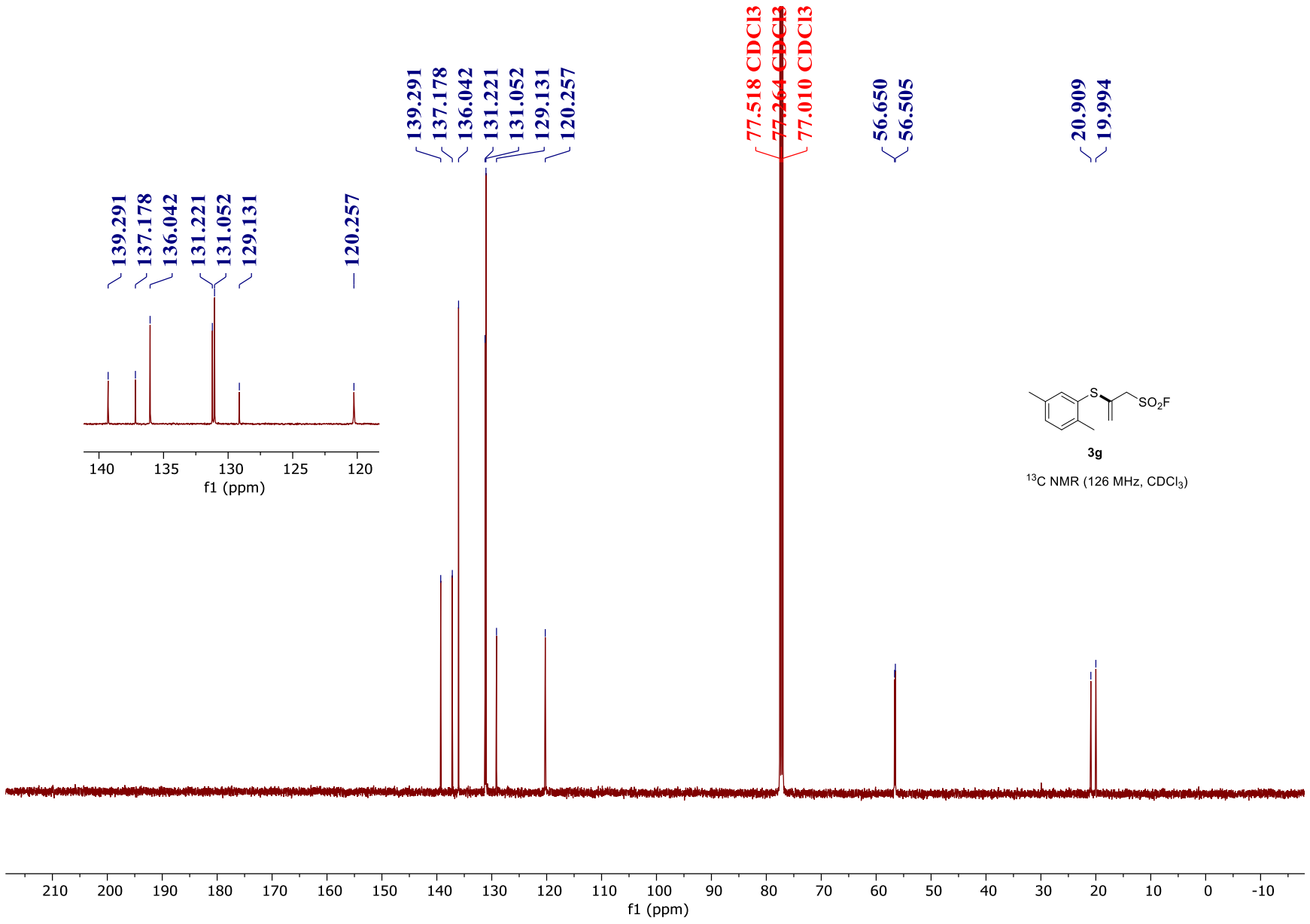

144.017
130.347
130.184
128.849
128.354
115.696
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
56.739
56.592
21.347
21.331
130.347
130.184
128.849
128.354
132
131
130
129
128
f1 (ppm)
S
SO2F
3f
13C NMR (126 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

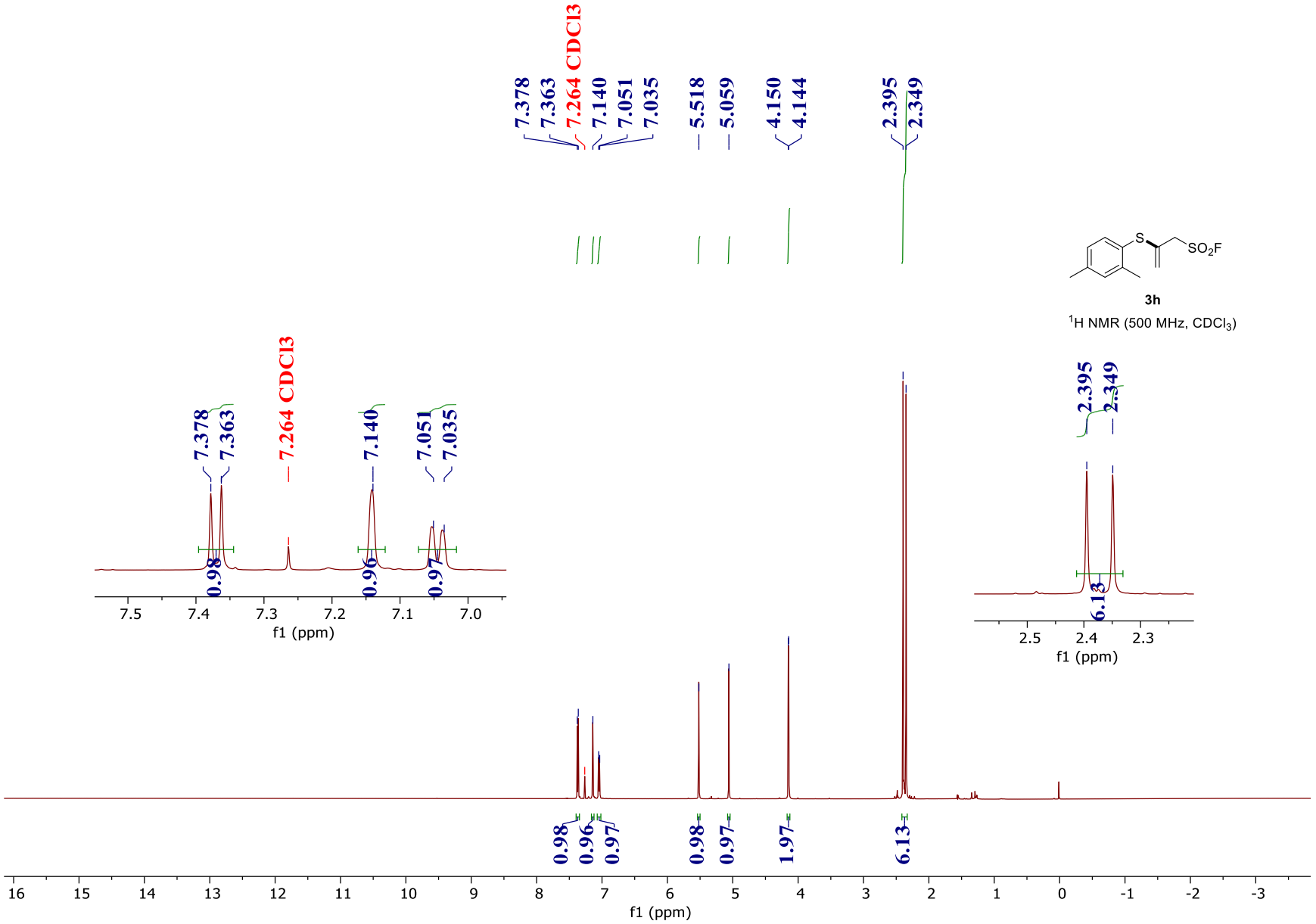

7.293
7.264 CDCl3
7.204
7.189
7.138
7.123
5.559
5.117
4.160
4.154
2.381
2.324
S
SO2F
3g
1H NMR (500 MHz, CDCl3)
0.97
1.00
0.97
0.98
0.97
1.98
3.00
2.96
f1 (ppm)

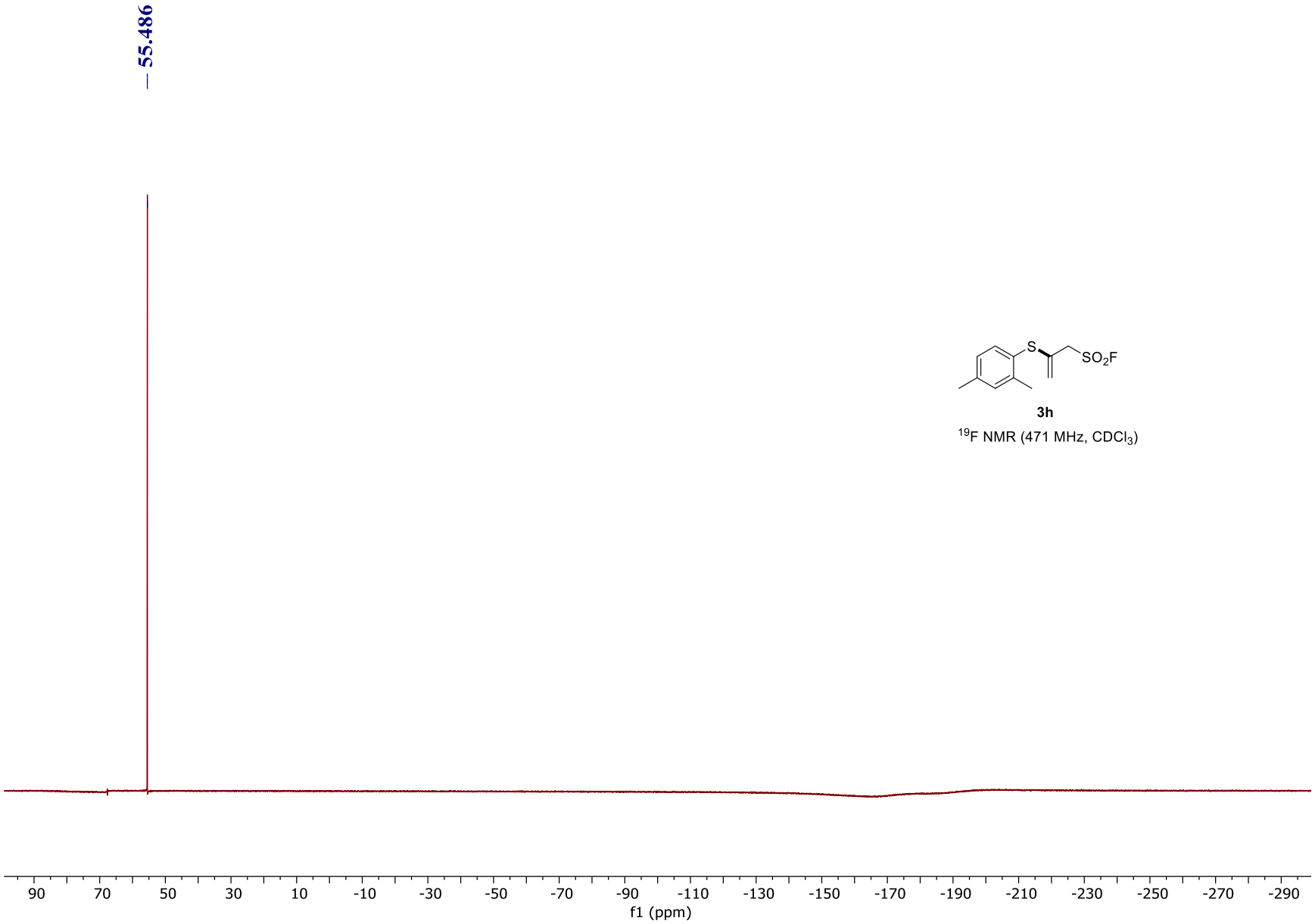


3g

^{19}F NMR (471 MHz, $CDCl_3$)

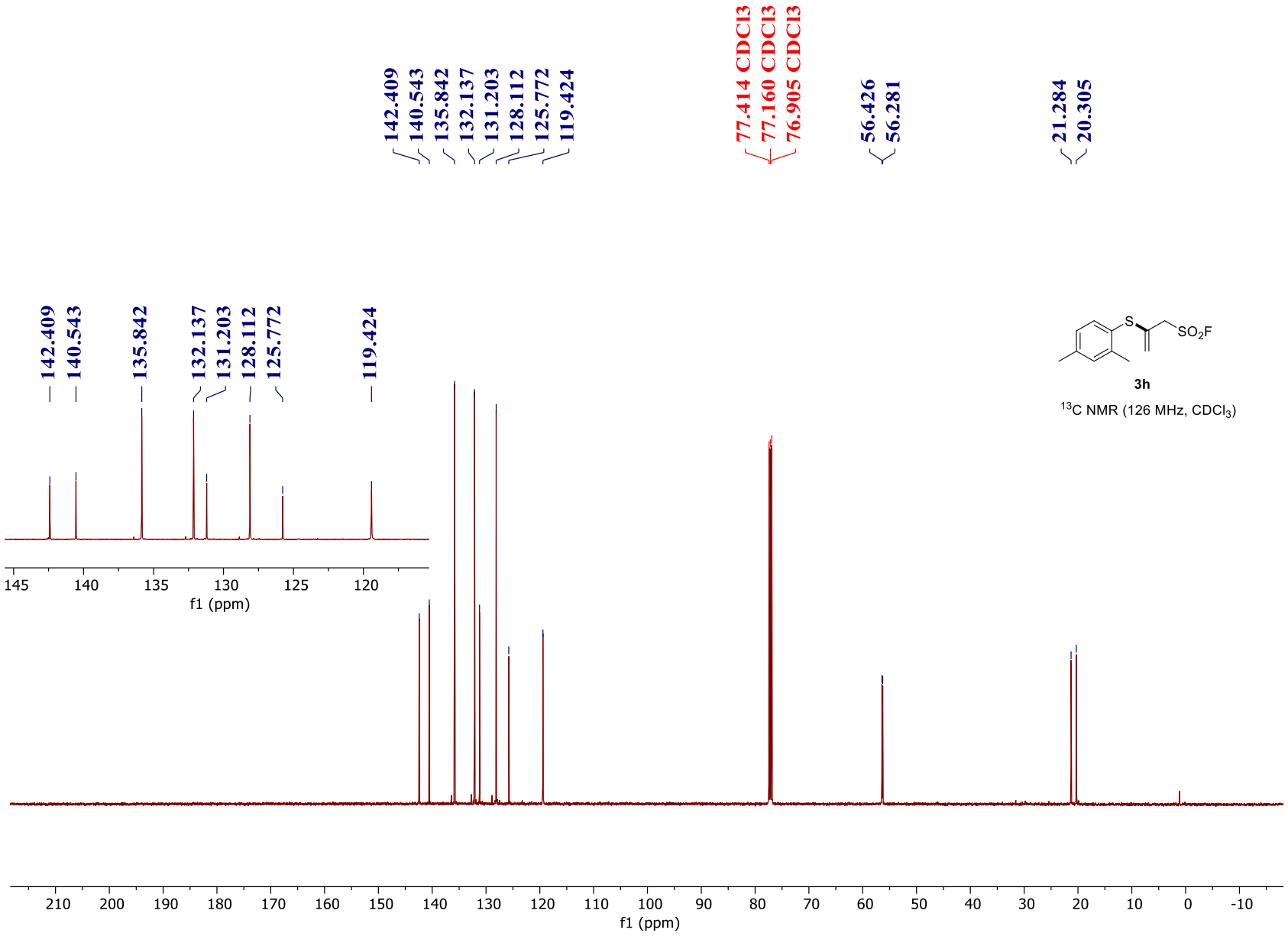

139.291
137.178
136.042
131.221
131.052
129.131
120.257
77.518 CDCl3
77.264 CDCl3
77.010 CDCl3
56.650
56.505
20.909
19.994
f1 (ppm)
SO2F
3g
13C NMR (126 MHz, CDCl3)

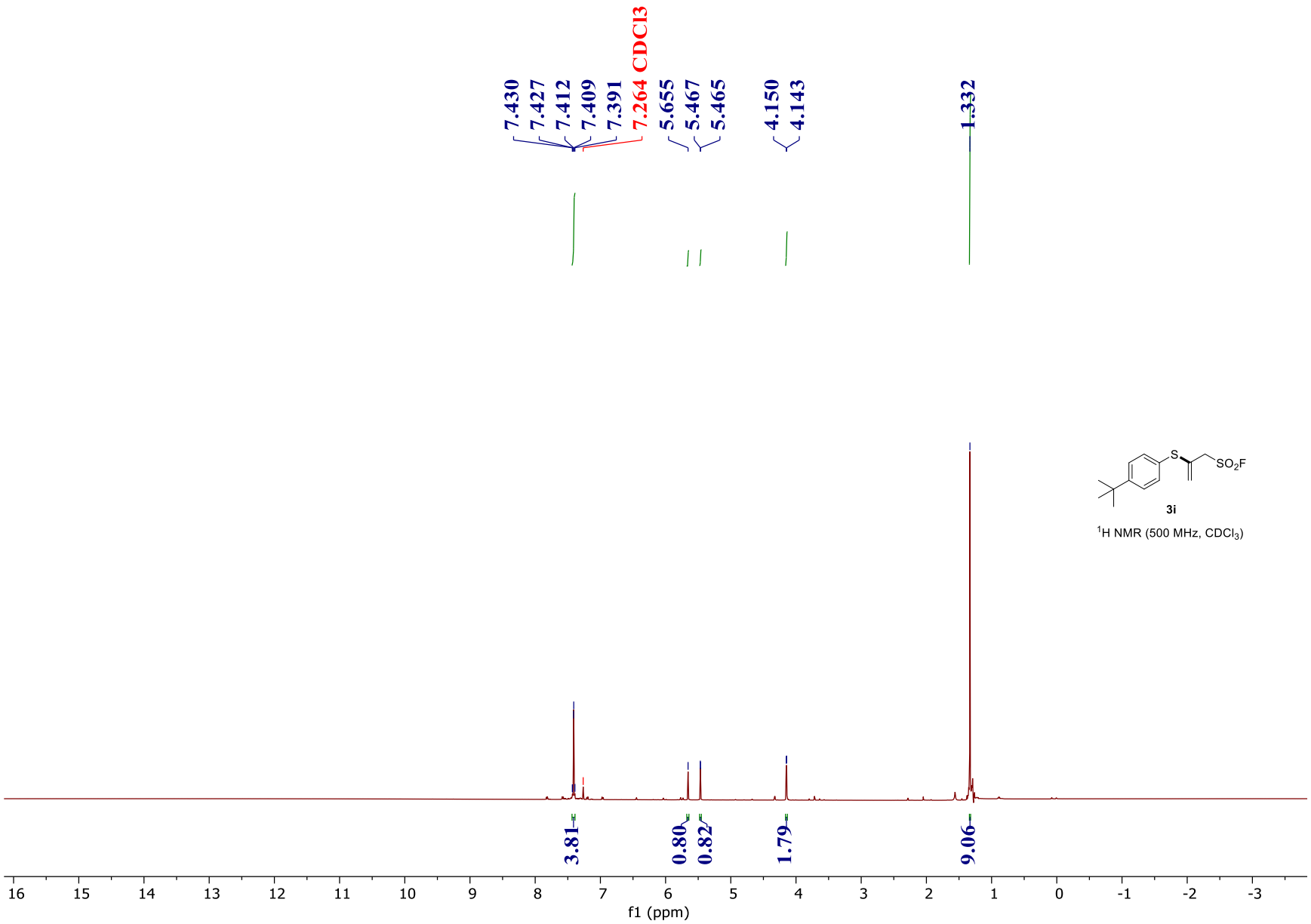

7.378
7.363
7.264 CDCl3
7.140
7.051
7.035
5.518
5.059
4.150
4.144
2.395
2.349
SO2F
3h
1H NMR (500 MHz, CDCl3)
0.98
0.96
0.97
0.98
0.97
1.97
6.13
f1 (ppm)

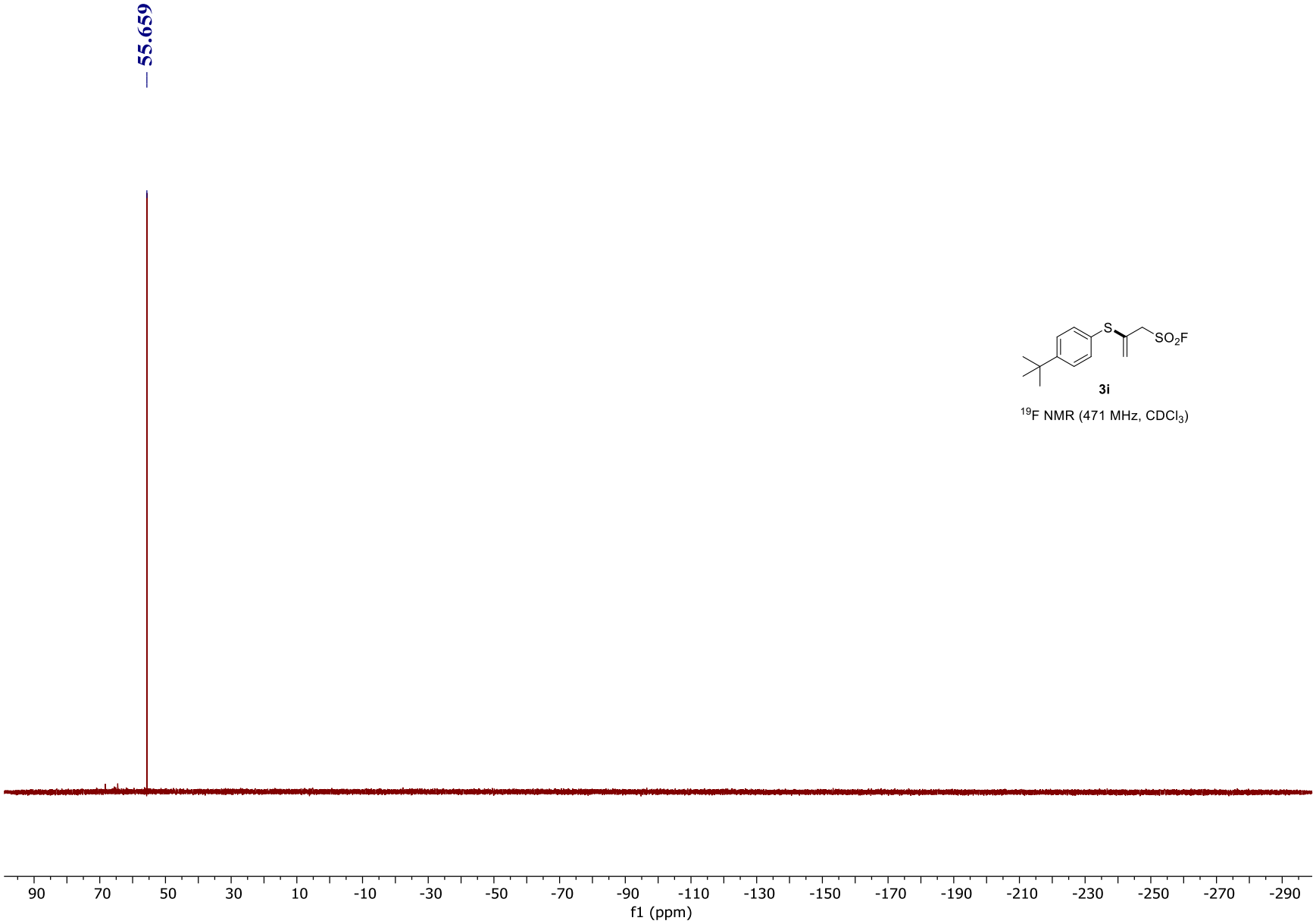


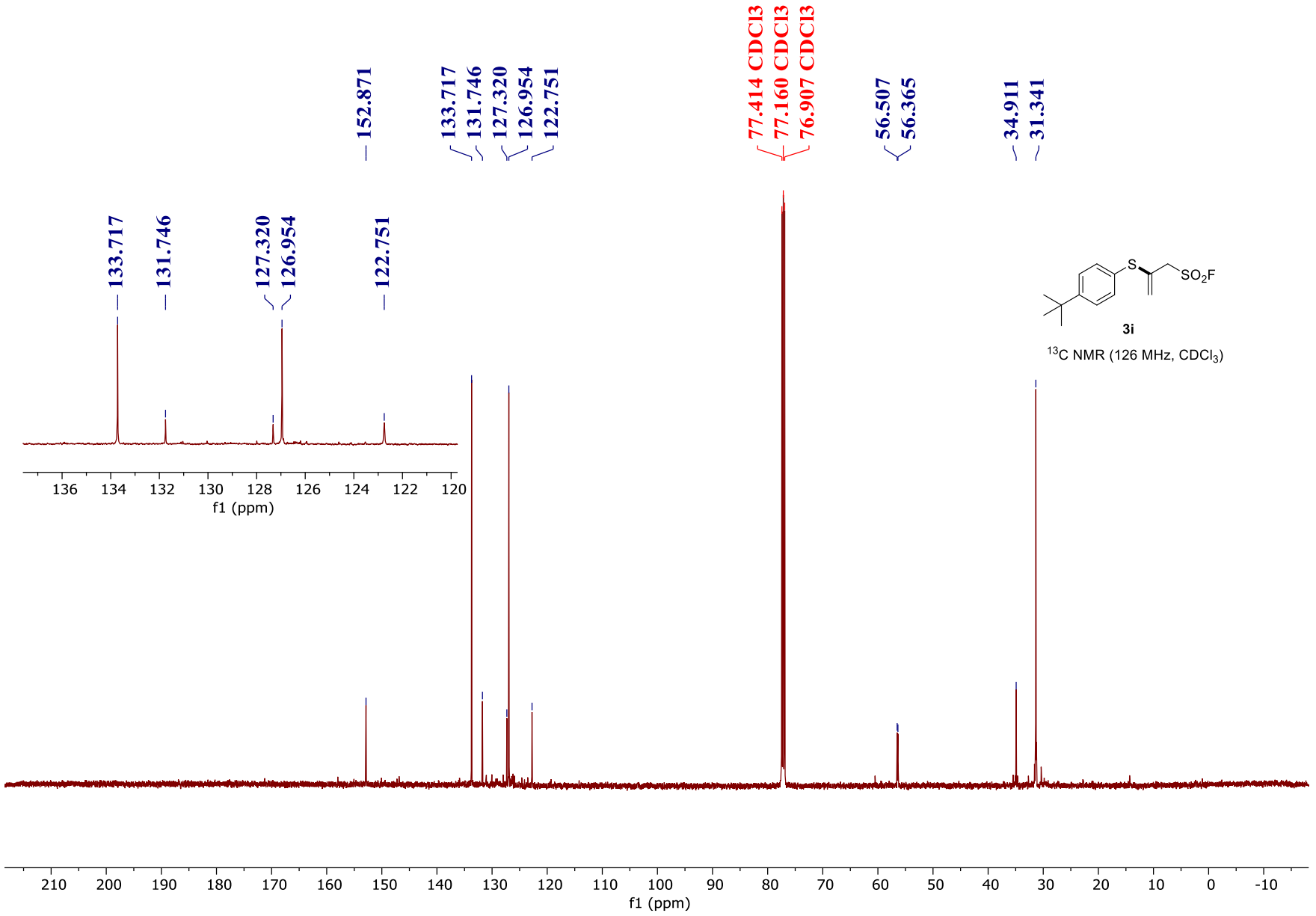
3h

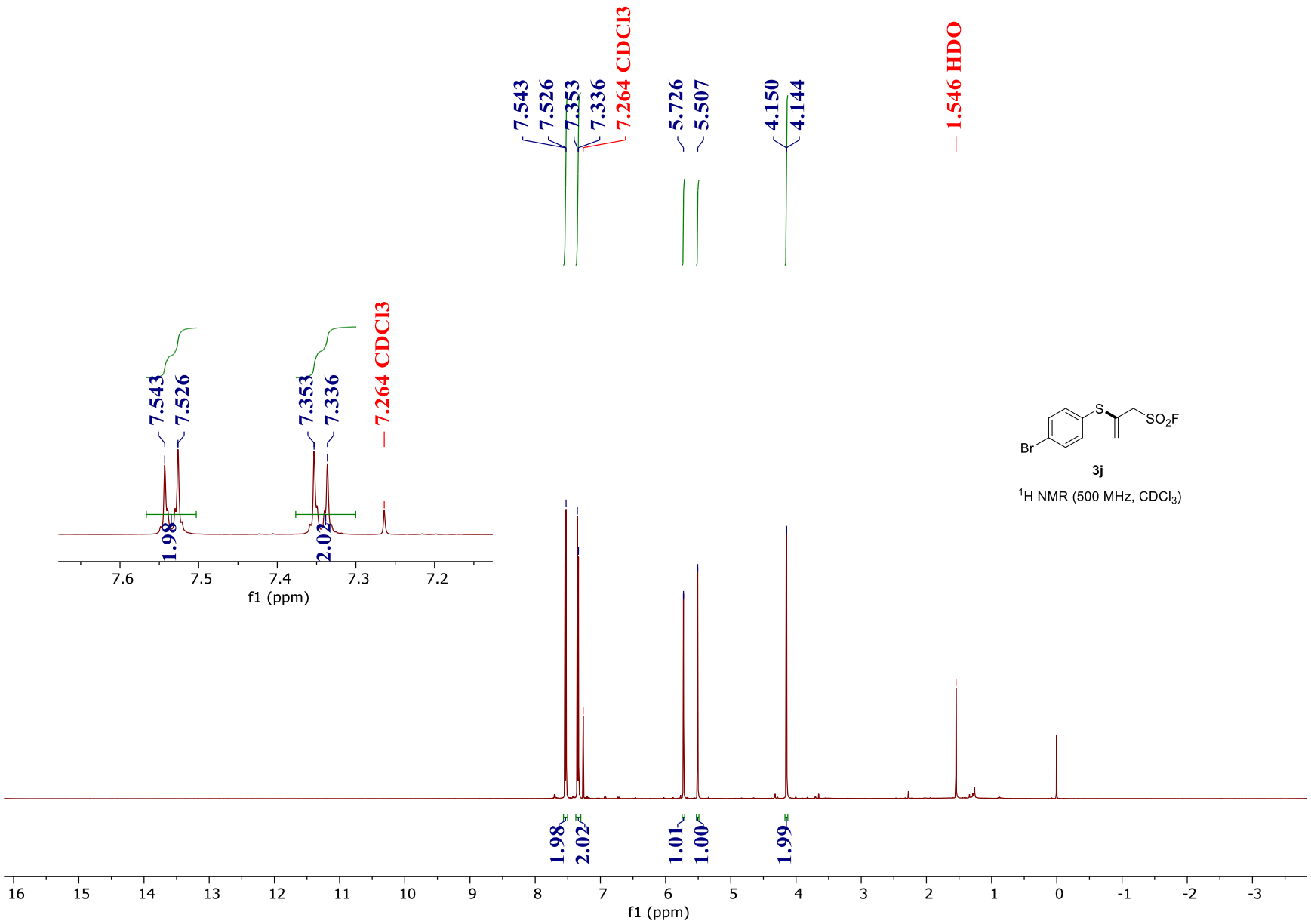
^{19}F NMR (471 MHz, $CDCl_3$)

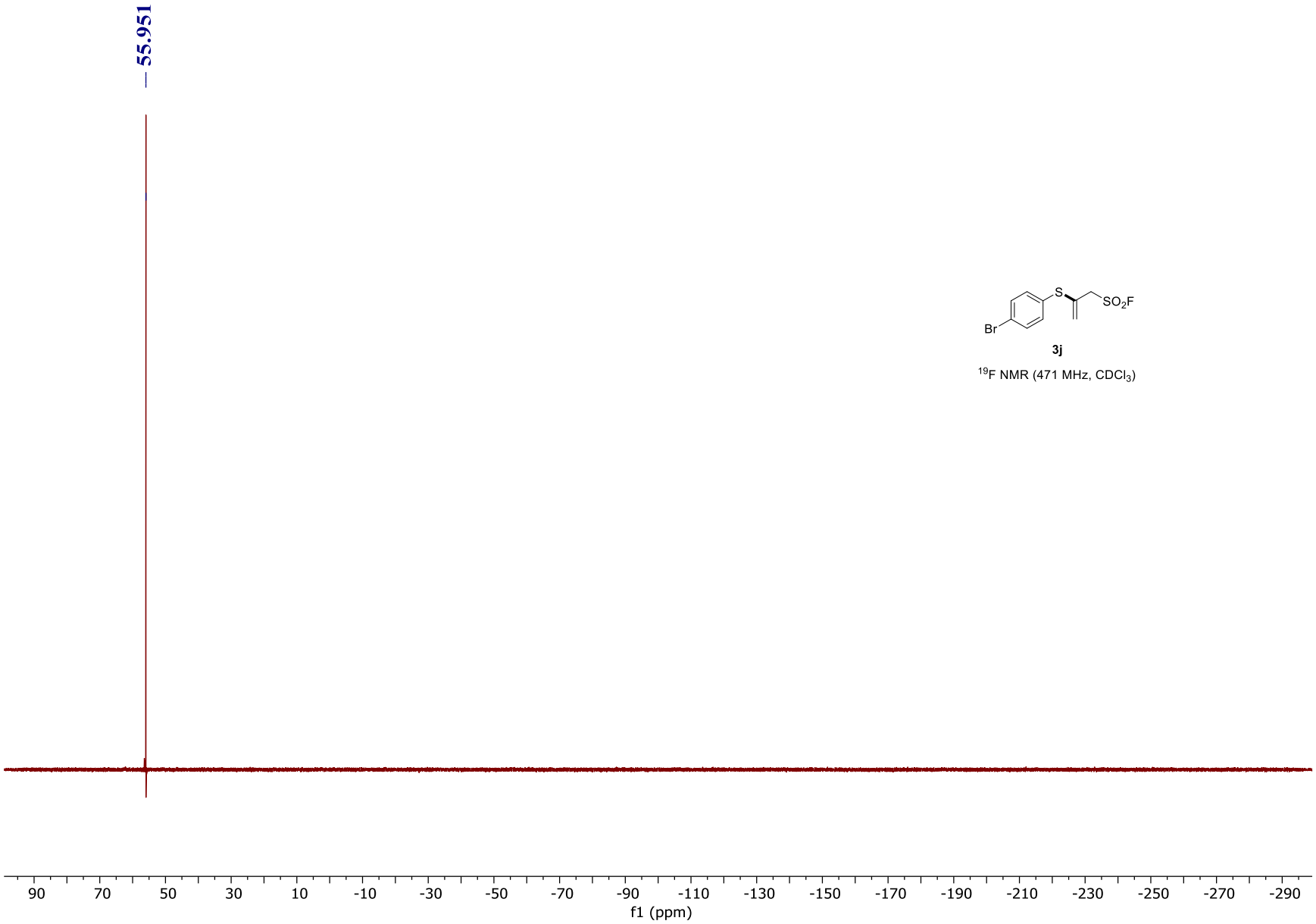

142.409
140.543
135.842
132.137
131.203
128.112
125.772
119.424
77.414 CDCl3
77.160 CDCl3
76.905 CDCl3
56.426
56.281
21.284
20.305
f1 (ppm)
S
SO2F
3h
13C NMR (126 MHz, CDCl3)


7.430
7.427
7.412
7.409
7.391
7.264 CDCl3
5.655
5.467
5.465
4.150
4.143
1.332
S
SO2F
3i
1H NMR (500 MHz, CDCl3)
3.81
0.80
0.82
1.79
9.06
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


55.659
S
SO2F
3i
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

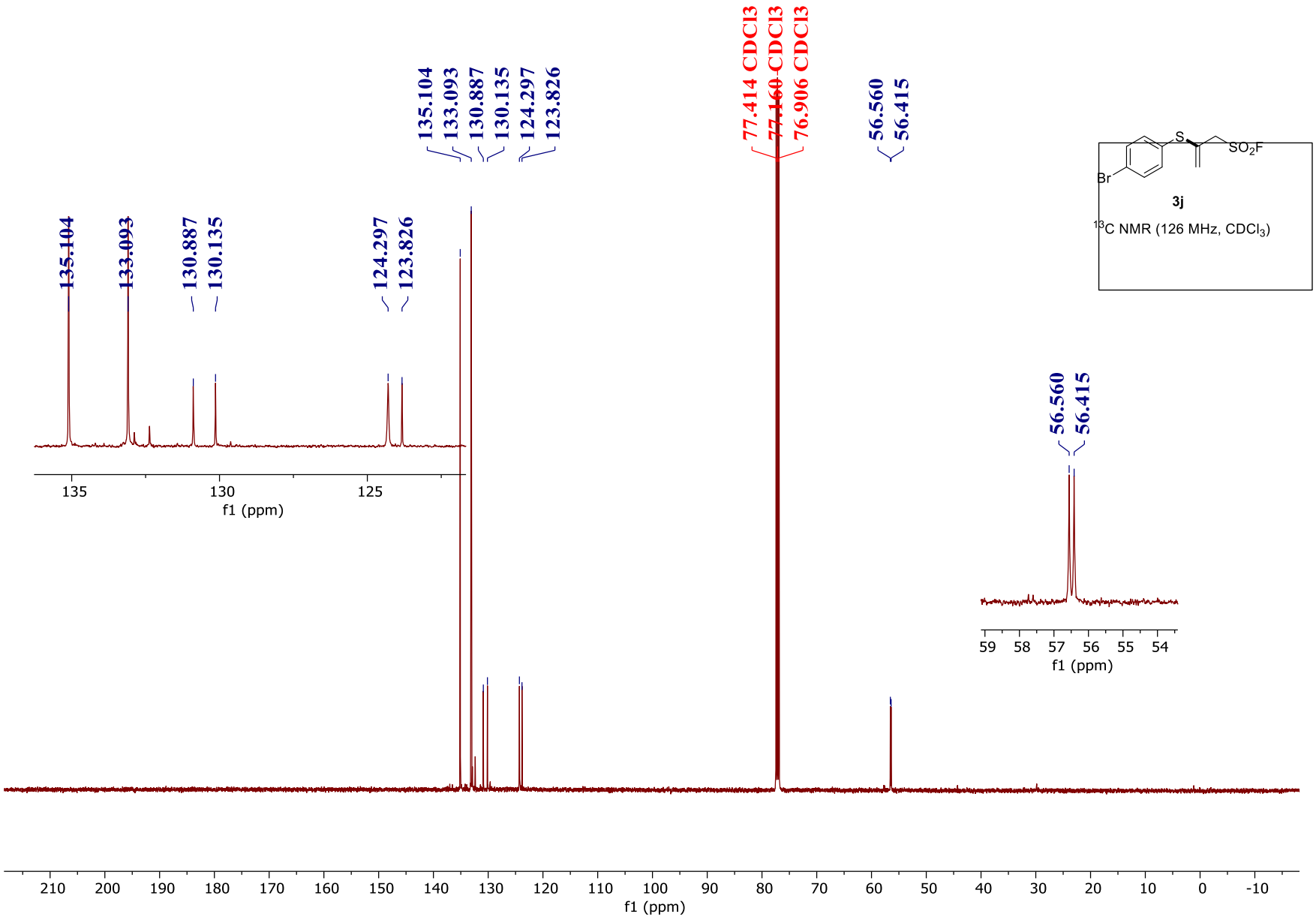

152.871
133.717
131.746
127.320
126.954
122.751
77.414 CDCl3
77.160 CDCl3
76.907 CDCl3
56.507
56.365
34.911
31.341
f1 (ppm)
3i
13C NMR (126 MHz, CDCl3)

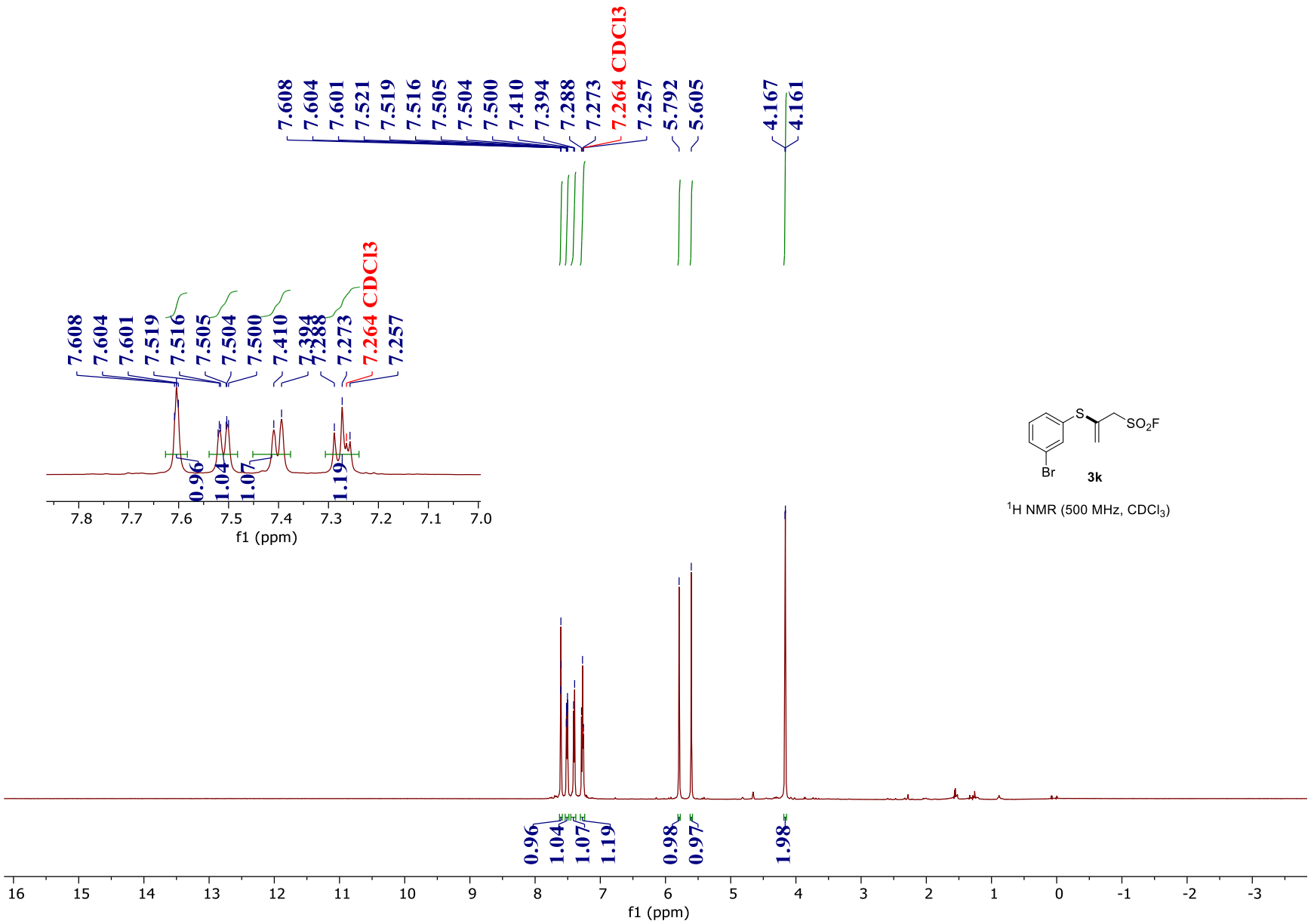

7.543
7.526
7.353
7.336
7.264 CDCl3
5.726
5.507
4.150
4.144
1.546 HDO
1.98
2.02
1.01
1.00
1.99
f1 (ppm)
Br
S
SO2F
3j
1H NMR (500 MHz, CDCl3)

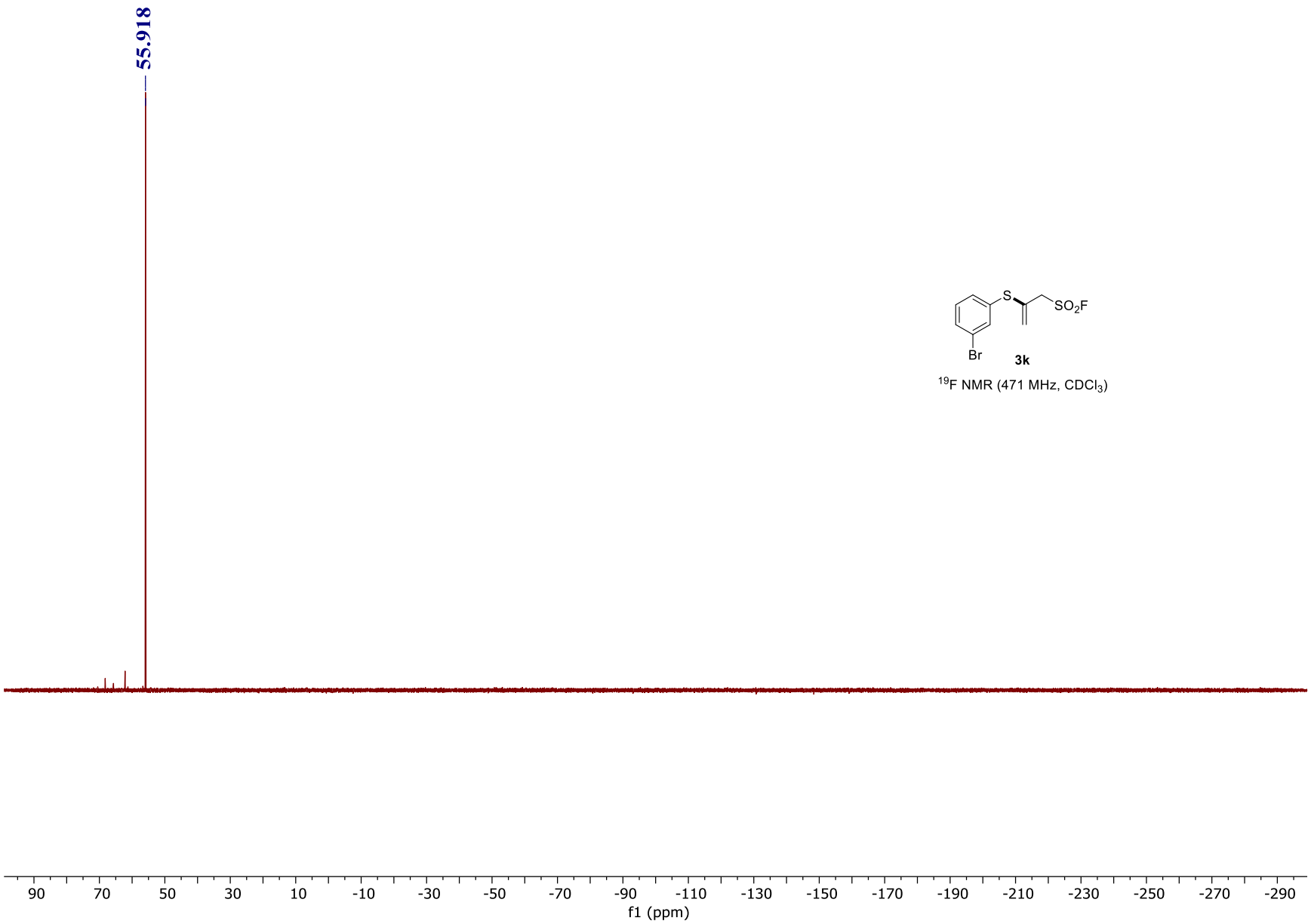


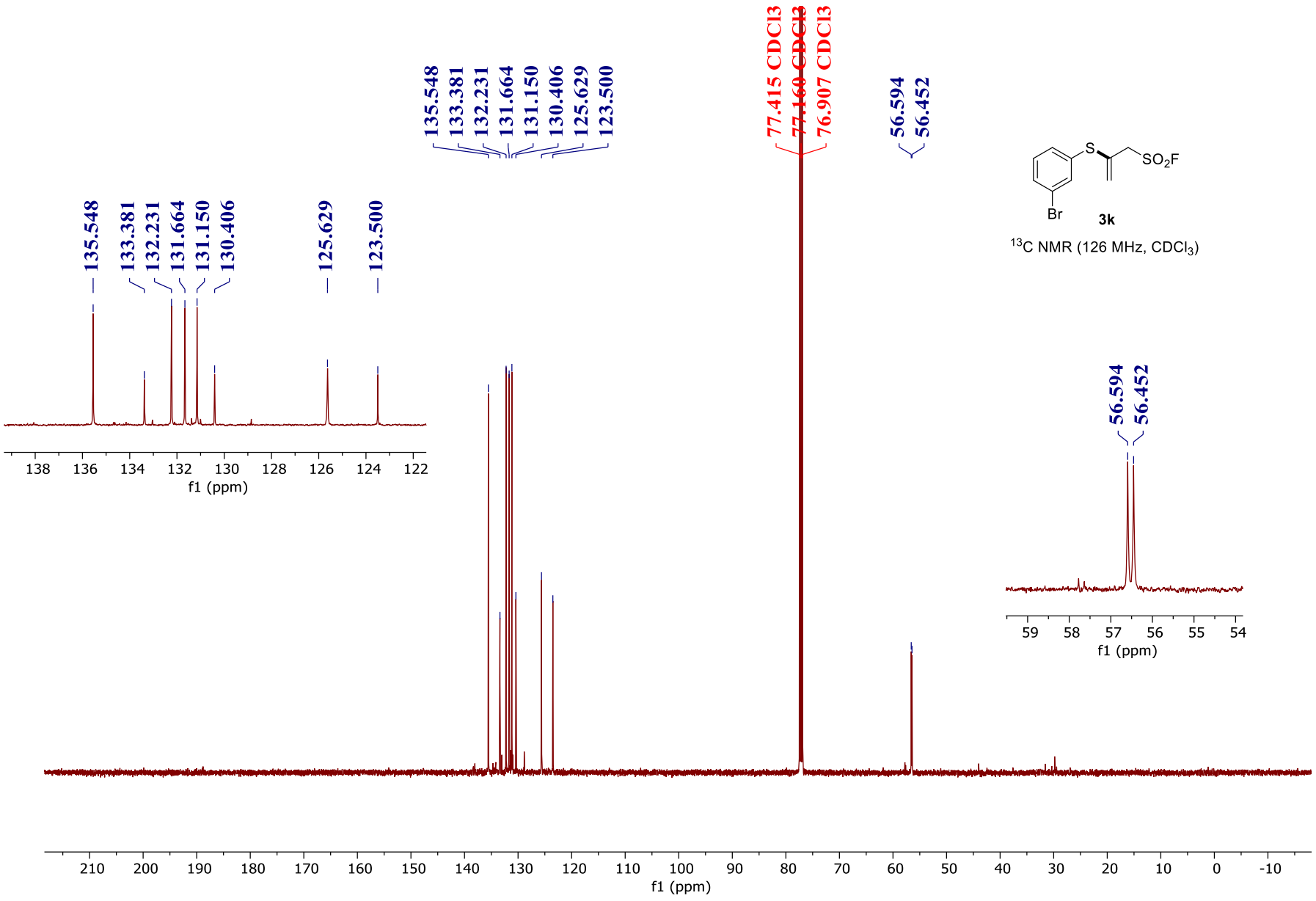
3j

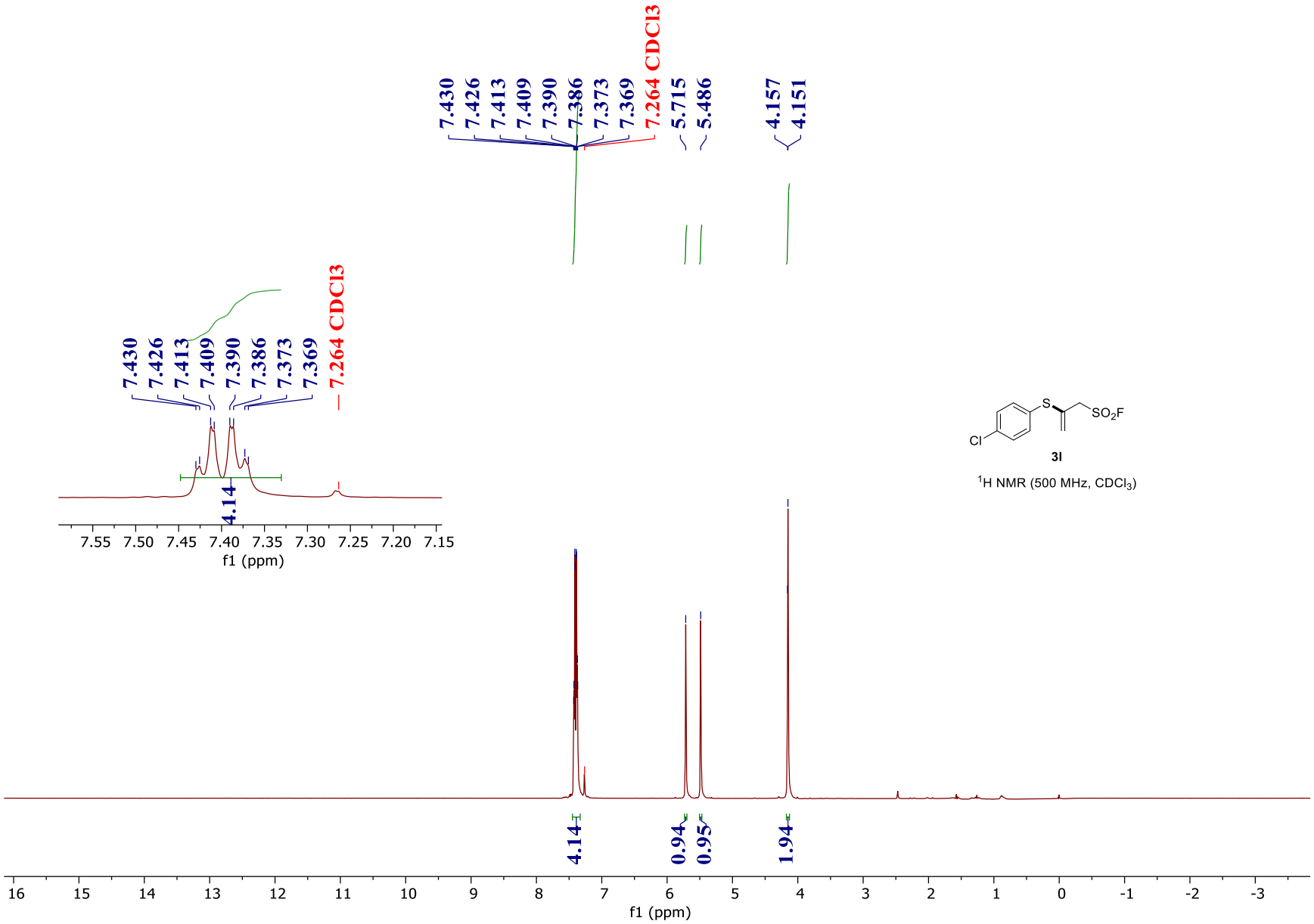
^{19}F NMR (471 MHz, $CDCl_3$)

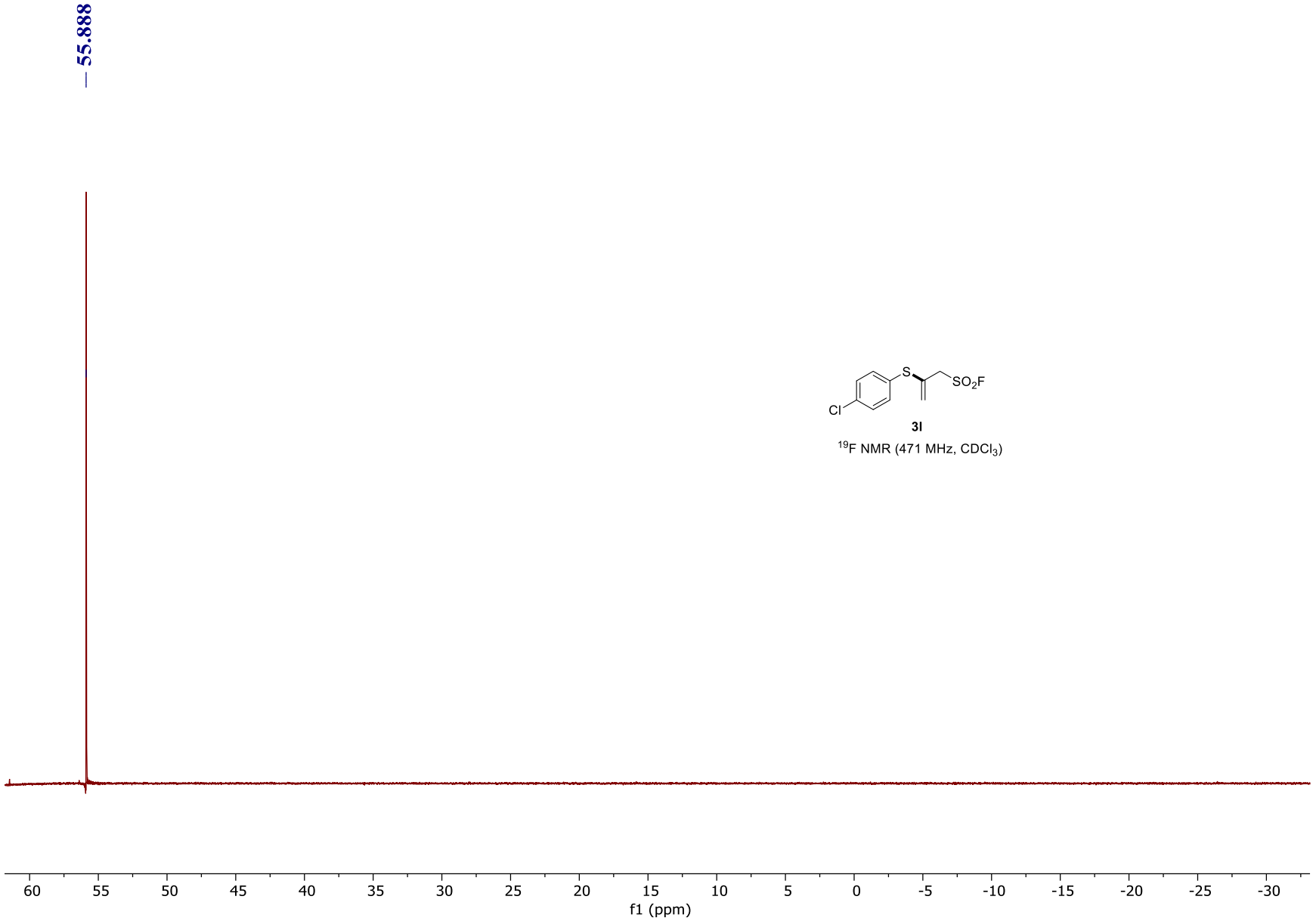

135.104
133.093
130.887
130.135
124.297
123.826
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
56.560
56.415
Br
S
SO2F
3j
13C NMR (126 MHz, CDCl3)
f1 (ppm)
f1 (ppm)
f1 (ppm)

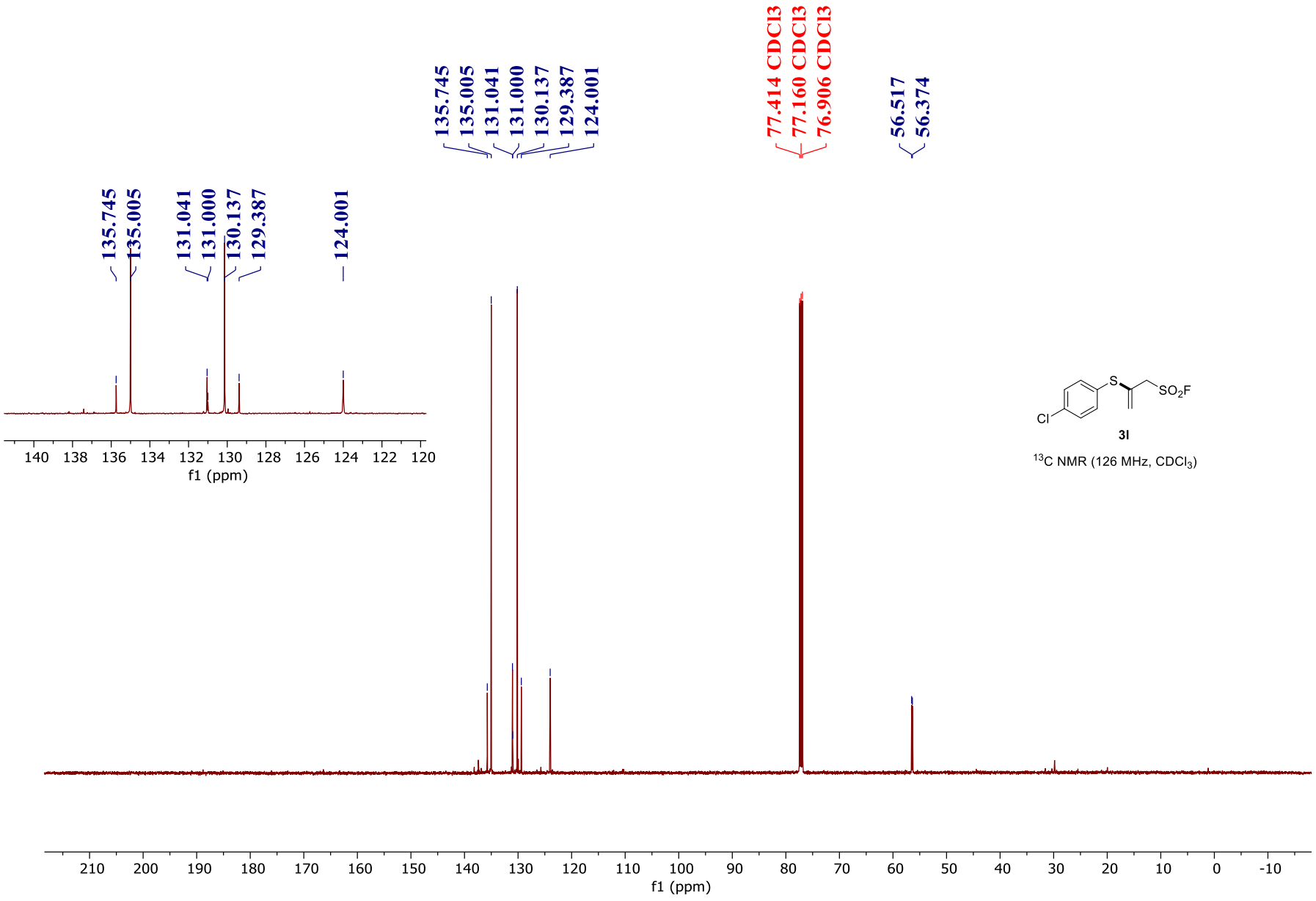

7.608
7.604
7.601
7.521
7.519
7.516
7.505
7.504
7.500
7.410
7.394
7.288
7.273
7.264 CDCl3
7.257
5.792
5.605
4.167
4.161
0.96
1.04
1.07
1.19
0.98
0.97
1.98
f1 (ppm)
S
SO2F
Br
3k
1H NMR (500 MHz, CDCl3)

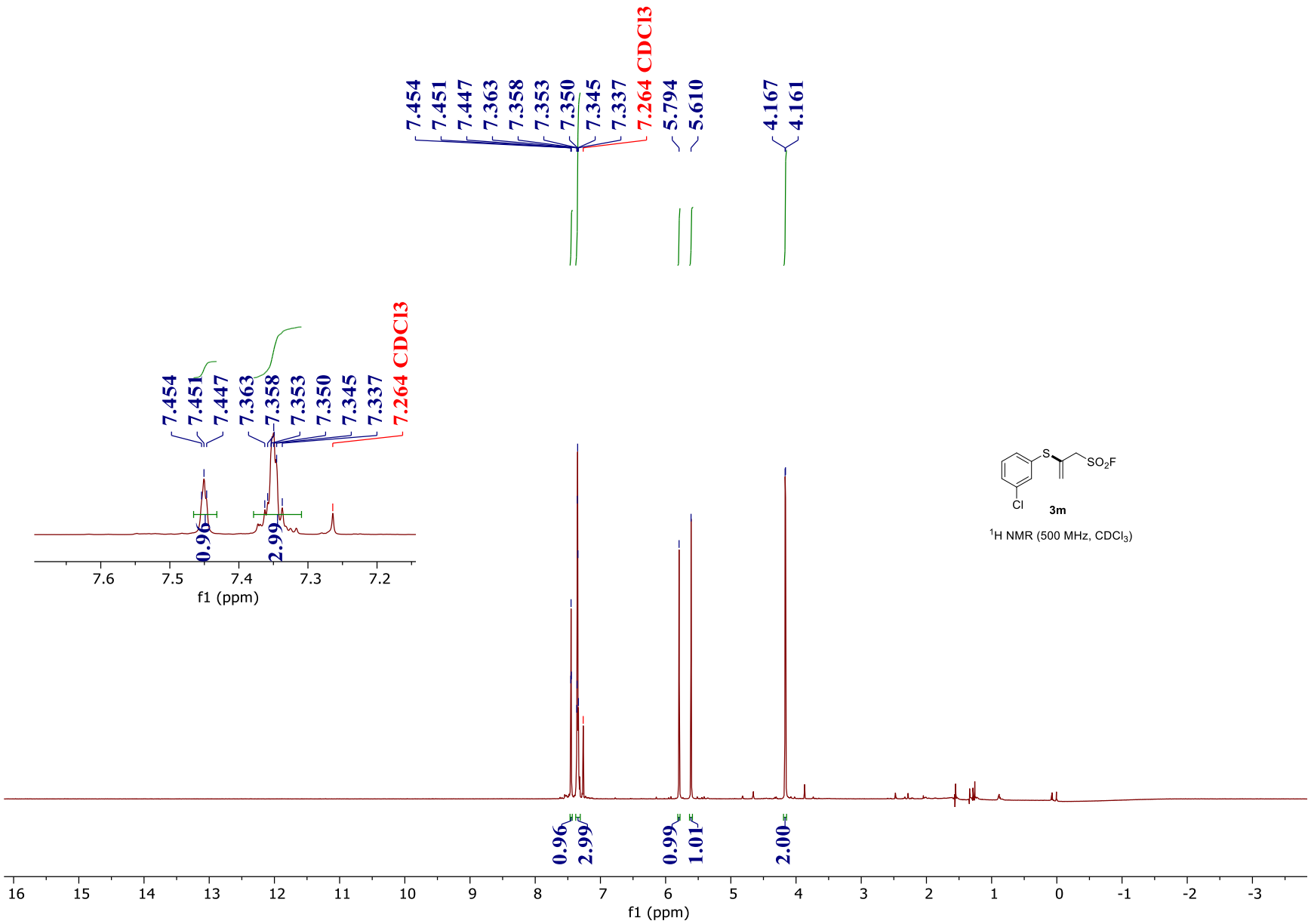

55.918
S
SO2F
Br
3k
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

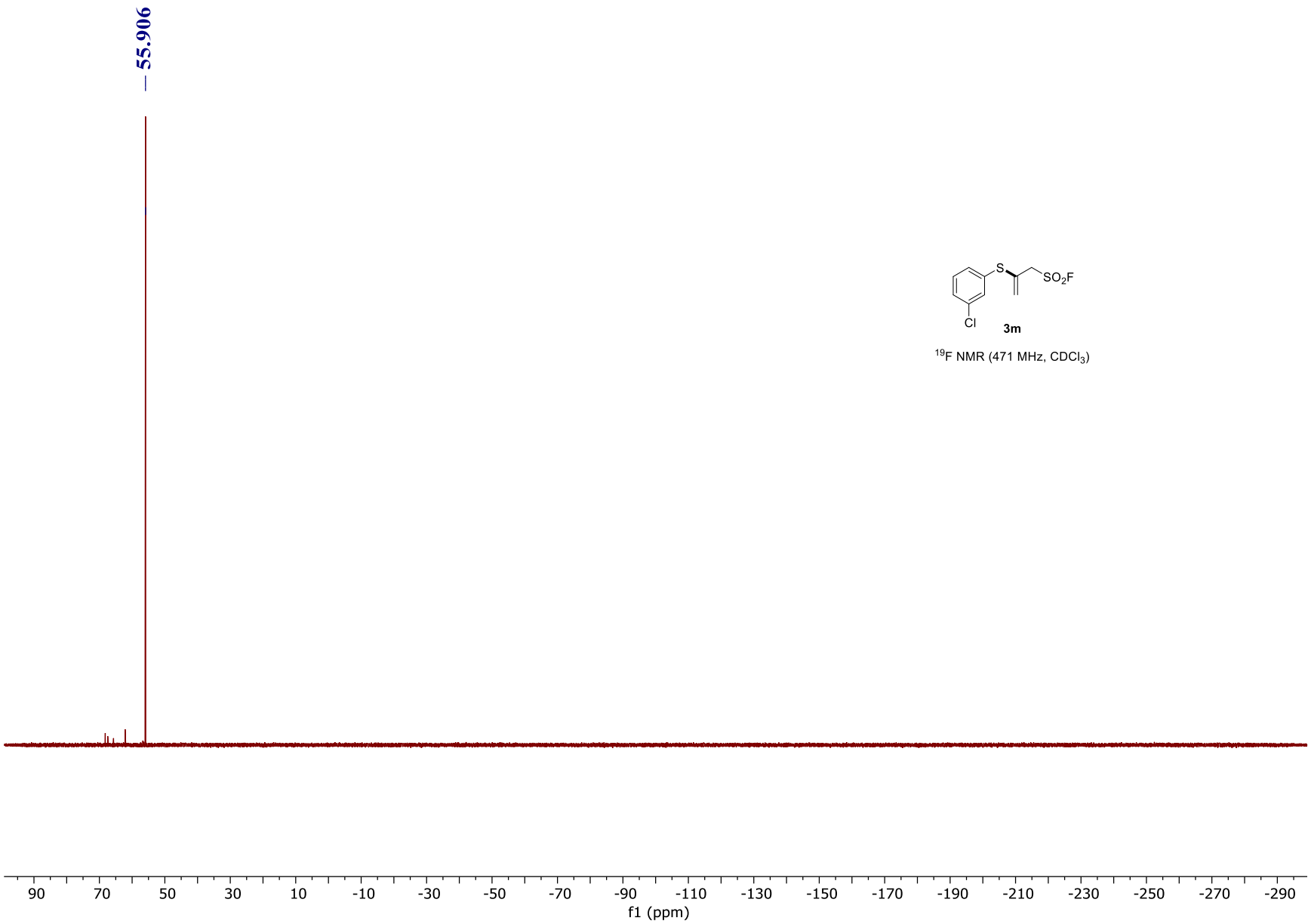

135.548
133.381
132.231
131.664
131.150
130.406
125.629
123.500
77.415 CDCl3
77.160 CDCl3
76.907 CDCl3
56.594
56.452
S
SO2F
Br
3k
13C NMR (126 MHz, CDCl3)
f1 (ppm)

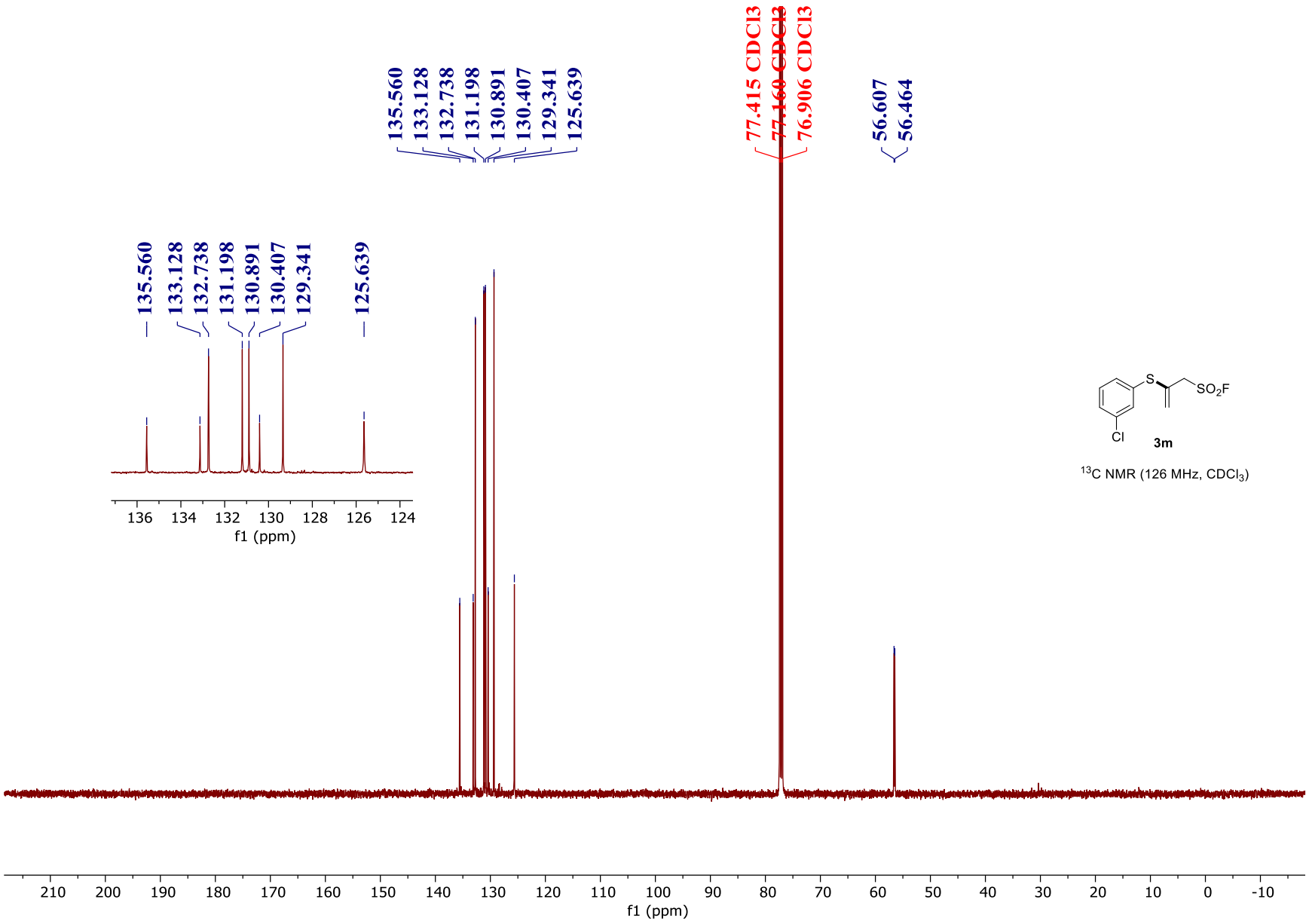

7.430
7.426
7.413
7.409
7.390
7.386
7.373
7.369
7.264 CDCl3
5.715
5.486
4.157
4.151
4.14
0.94
0.95
1.94
f1 (ppm)
Cl
S
SO2F
3l
1H NMR (500 MHz, CDCl3)

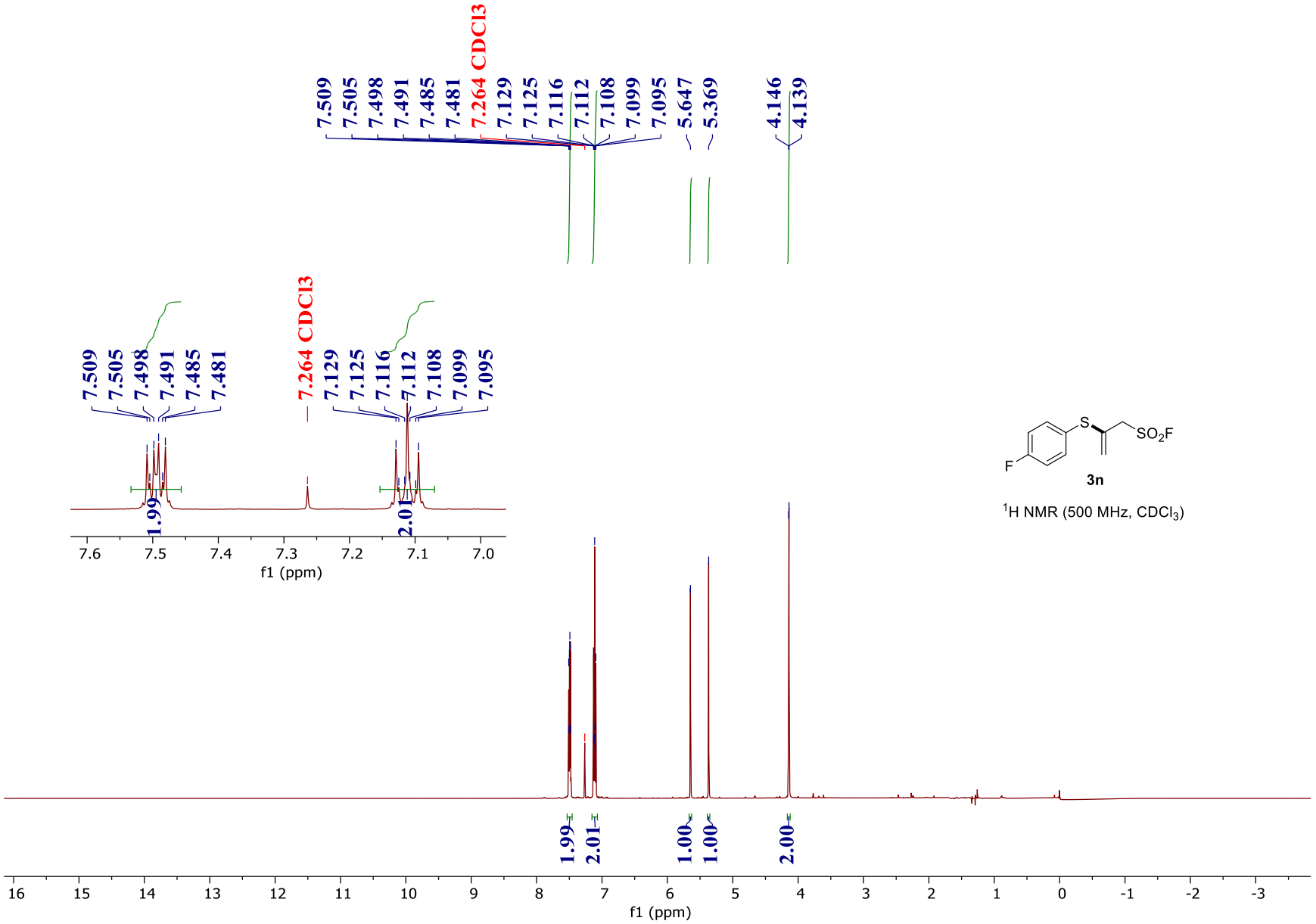

55.888
Cl
S
SO2F
3l
19F NMR (471 MHz, CDCl3)
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
-20
-25
-30
f1 (ppm)

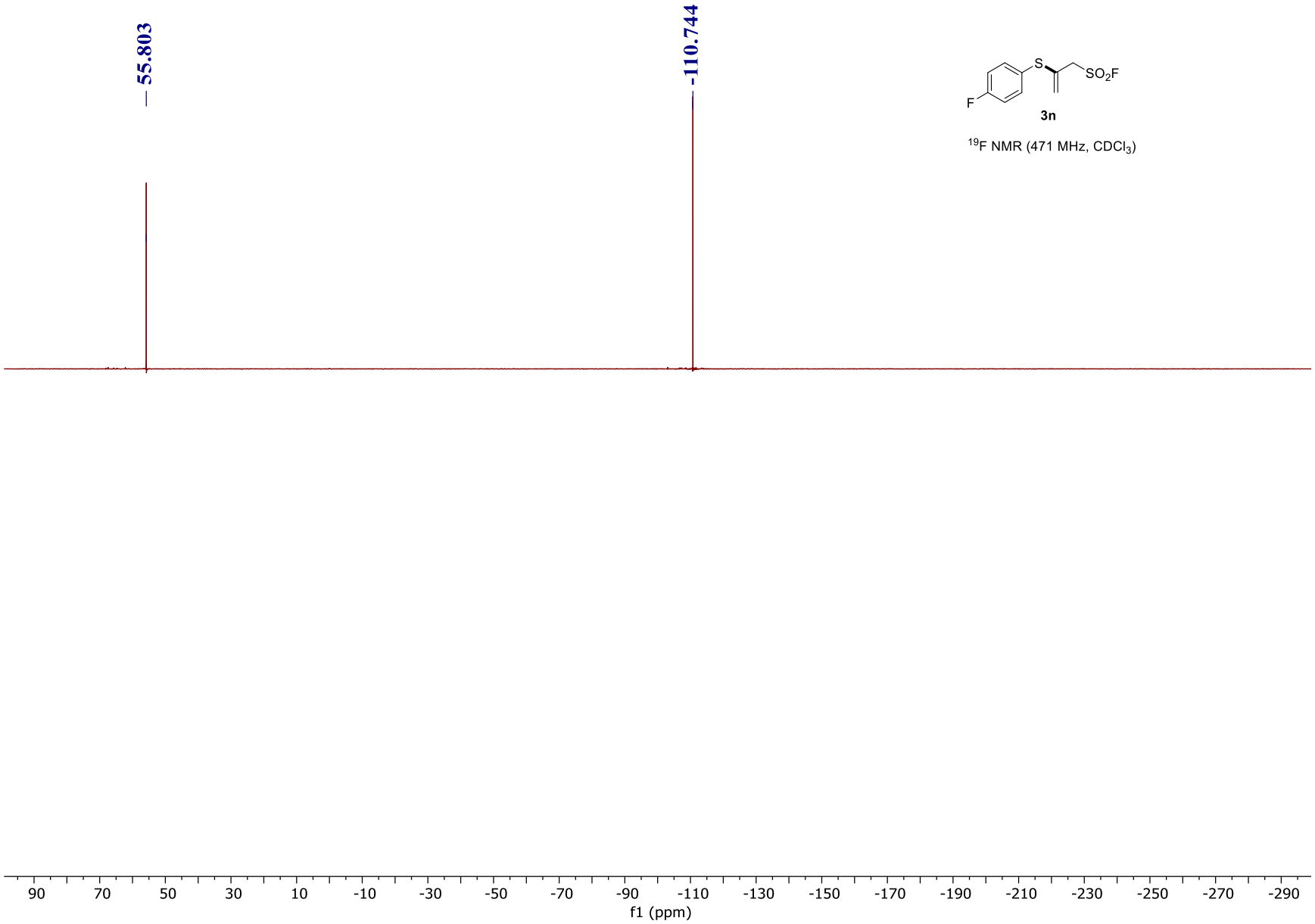

135.745
135.005
131.041
131.000
130.137
129.387
124.001
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
56.517
56.374
f1 (ppm)
Cl
S
SO2F
3l
13C NMR (126 MHz, CDCl3)

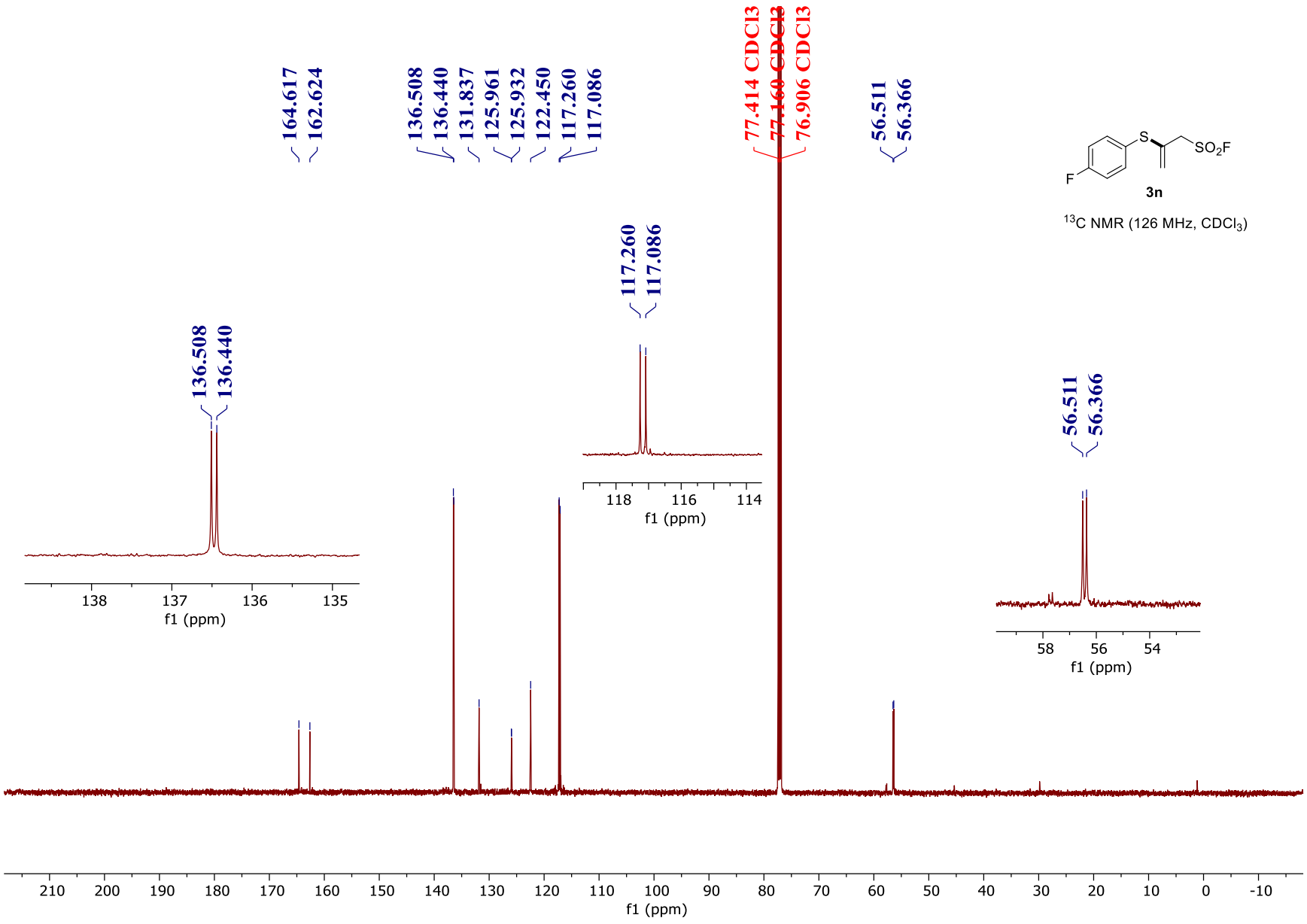

7.454
7.451
7.447
7.363
7.358
7.353
7.350
7.345
7.337
7.264 CDCl3
5.794
5.610
4.167
4.161
0.96
2.99
0.99
1.01
2.00
f1 (ppm)
SO2F
Cl
3m
1H NMR (500 MHz, CDCl3)

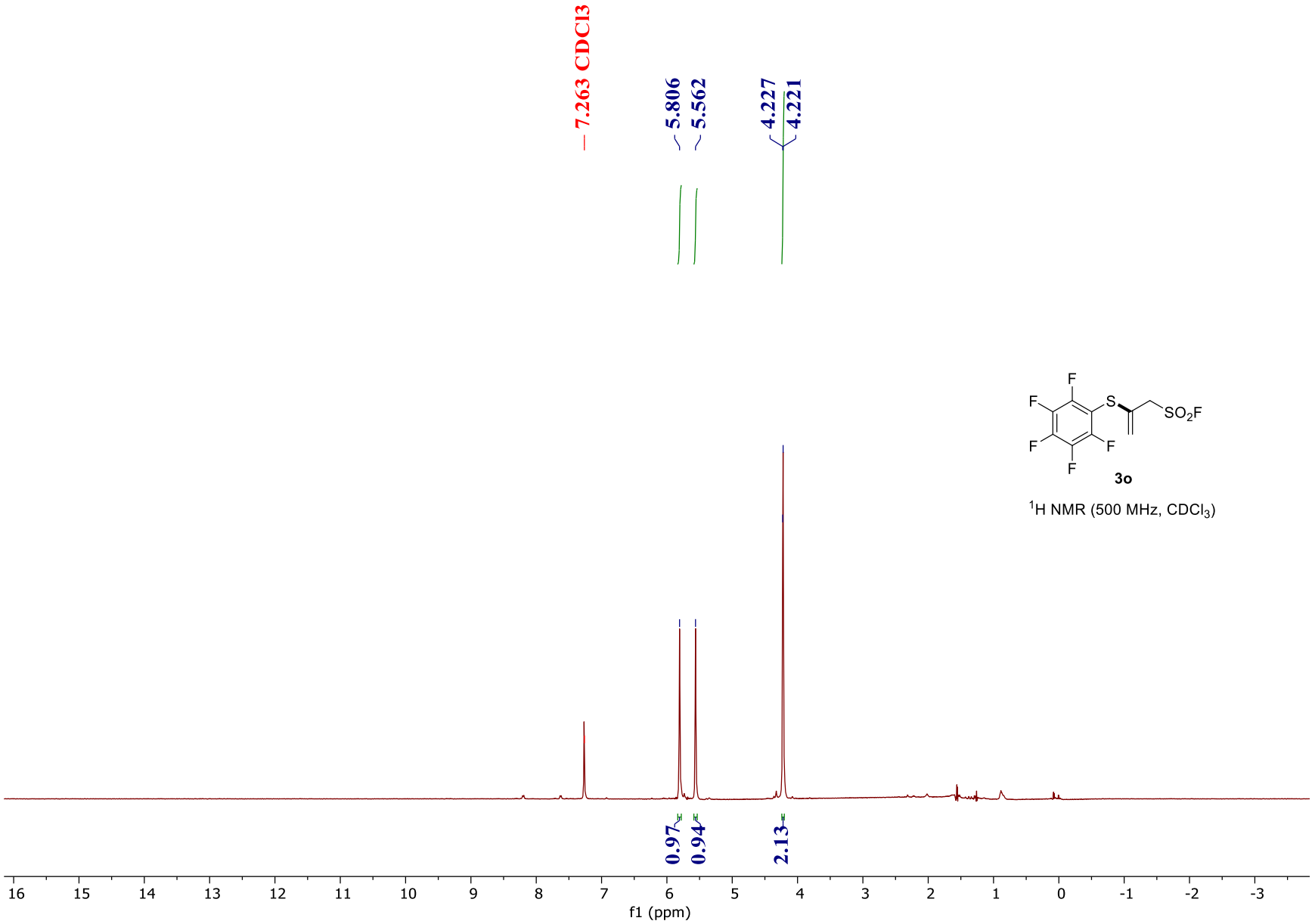

55.906
S
SO2F
Cl
3m
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

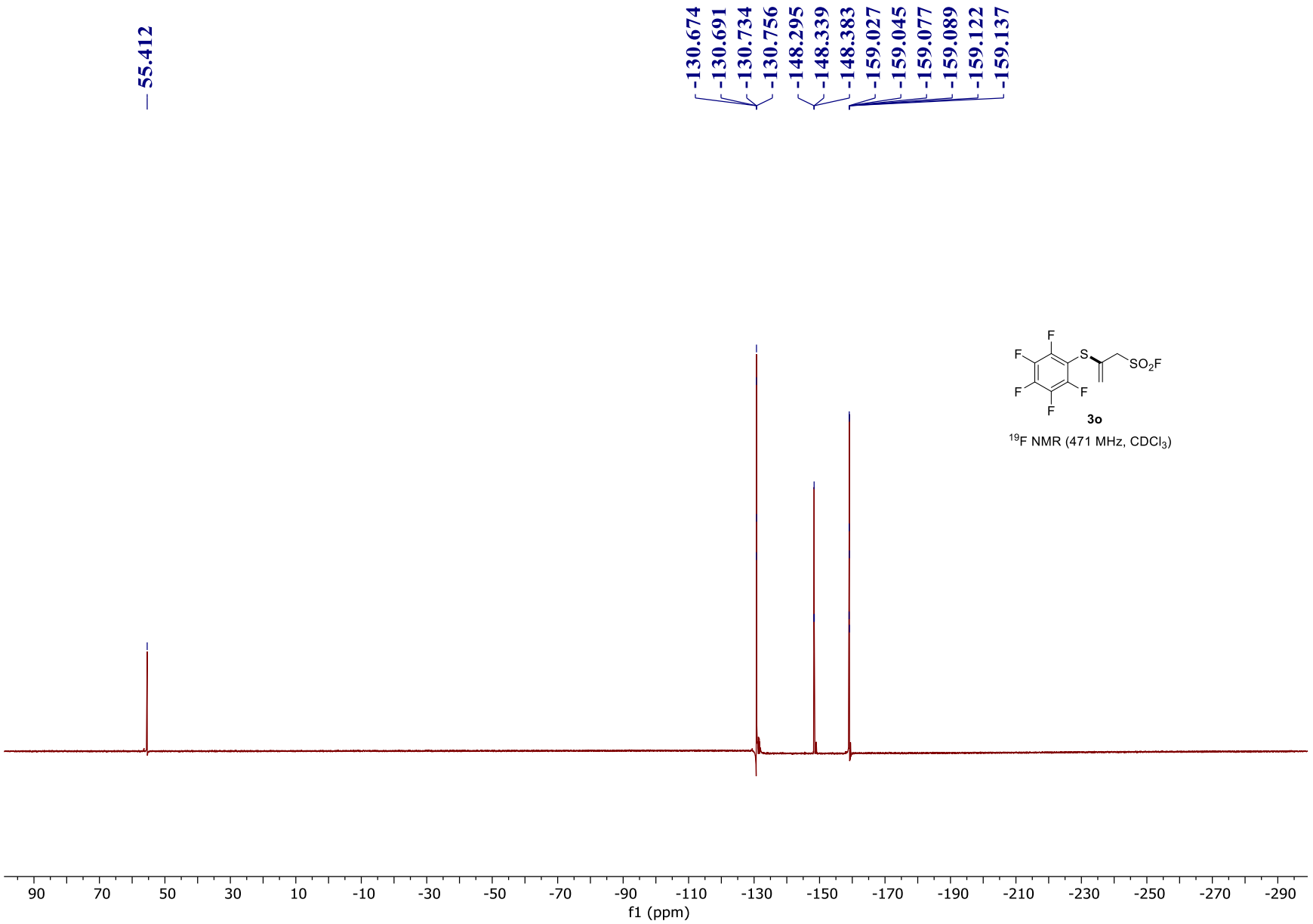

135.560
133.128
132.738
131.198
130.891
130.407
129.341
125.639
77.415 CDCl3
77.160 CDCl3
76.906 CDCl3
56.607
56.464
f1 (ppm)
S
SO2F
Cl
3m
13C NMR (126 MHz, CDCl3)

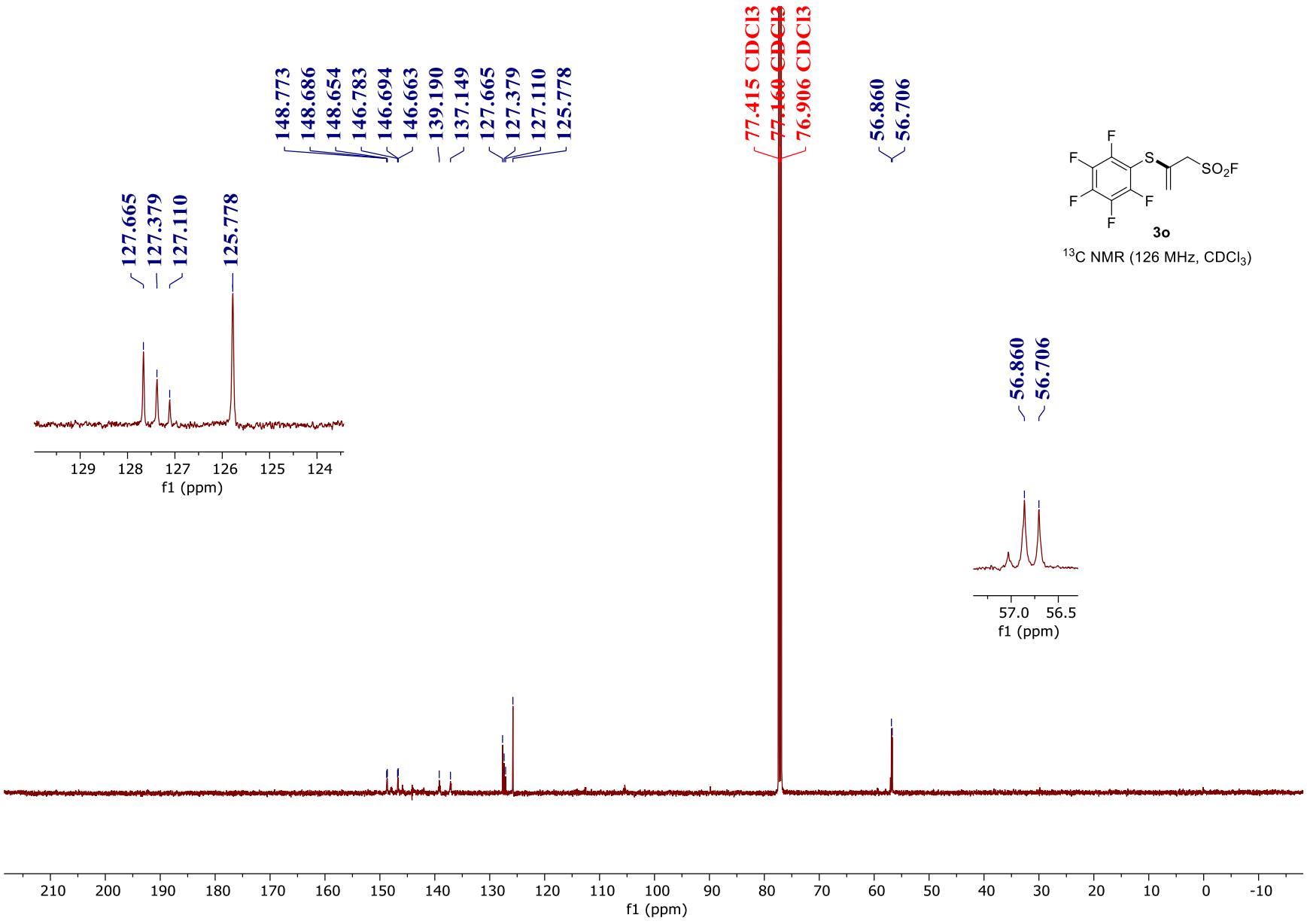

7.509
7.505
7.498
7.491
7.485
7.481
7.264 CDCl3
7.129
7.125
7.116
7.112
7.108
7.099
7.095
5.647
5.369
4.146
4.139
1.99
2.01
1.00
1.00
2.00
f1 (ppm)
F
S
SO2F
3n
1H NMR (500 MHz, CDCl3)

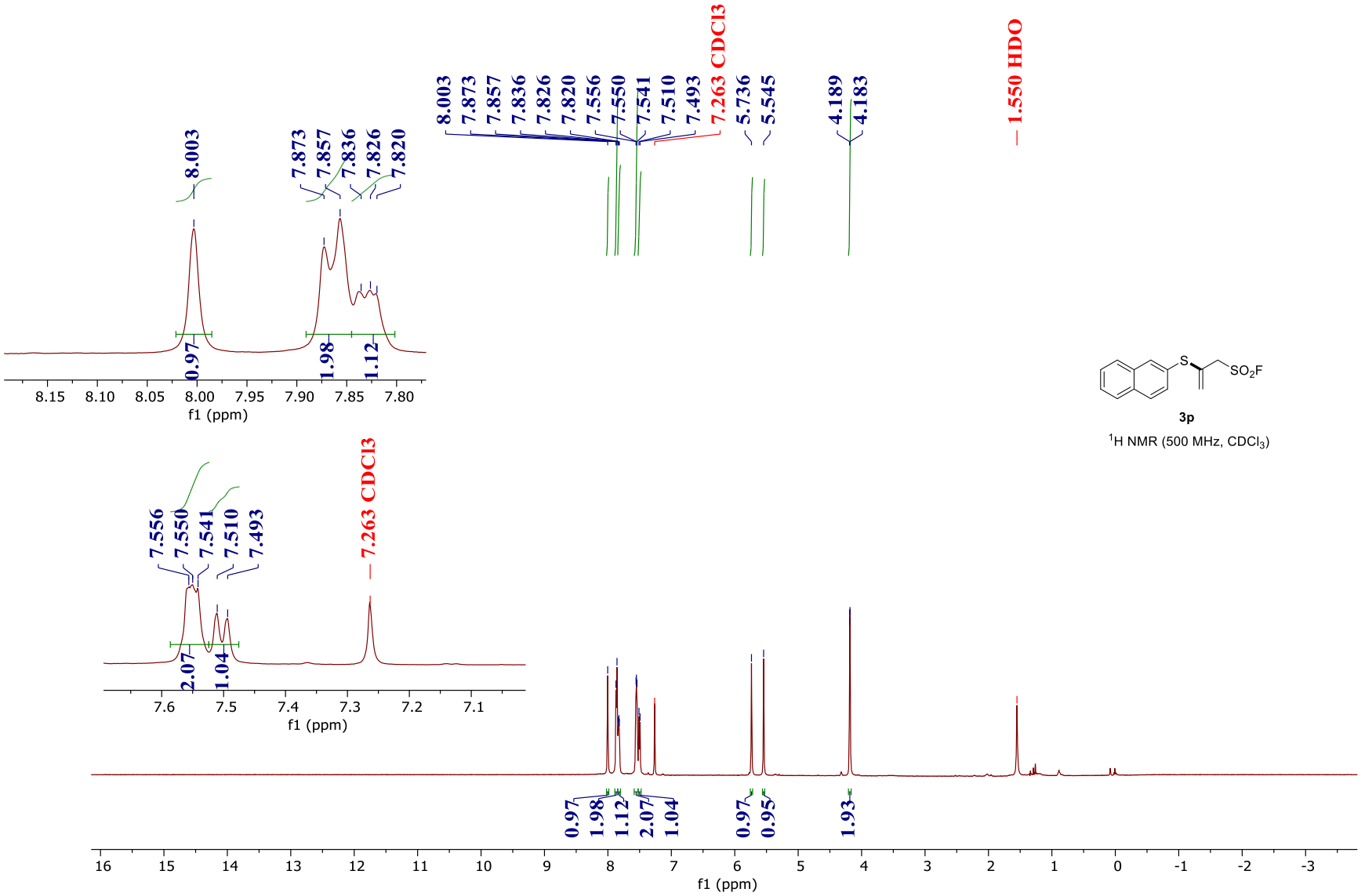

55.803
-110.744
S
SO2F
F
3n
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

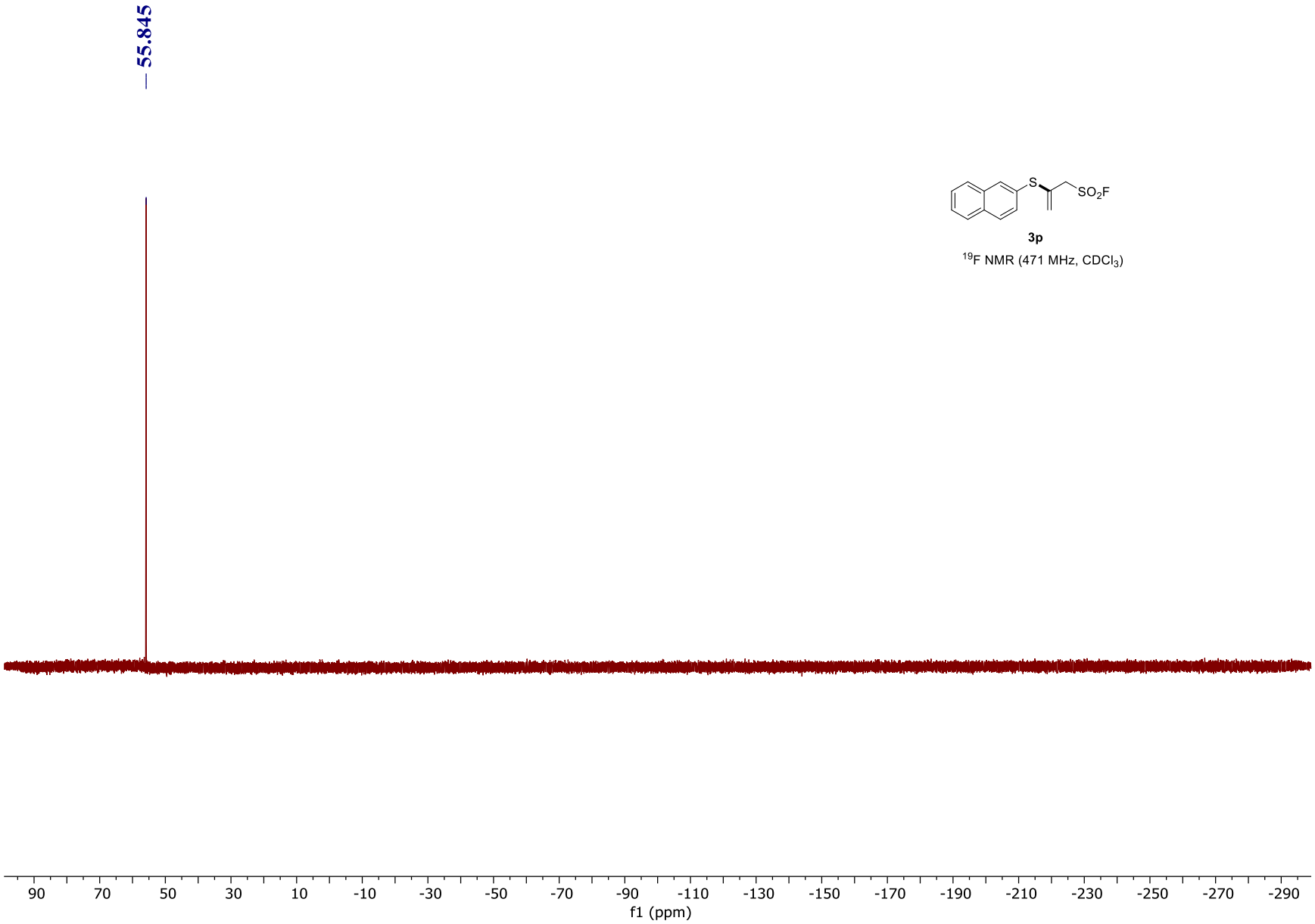

164.617
162.624
136.508
136.440
131.837
125.961
125.932
122.450
117.260
117.086
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
56.511
56.366
F
S
SO2F
3n
13C NMR (126 MHz, CDCl3)
f1 (ppm)

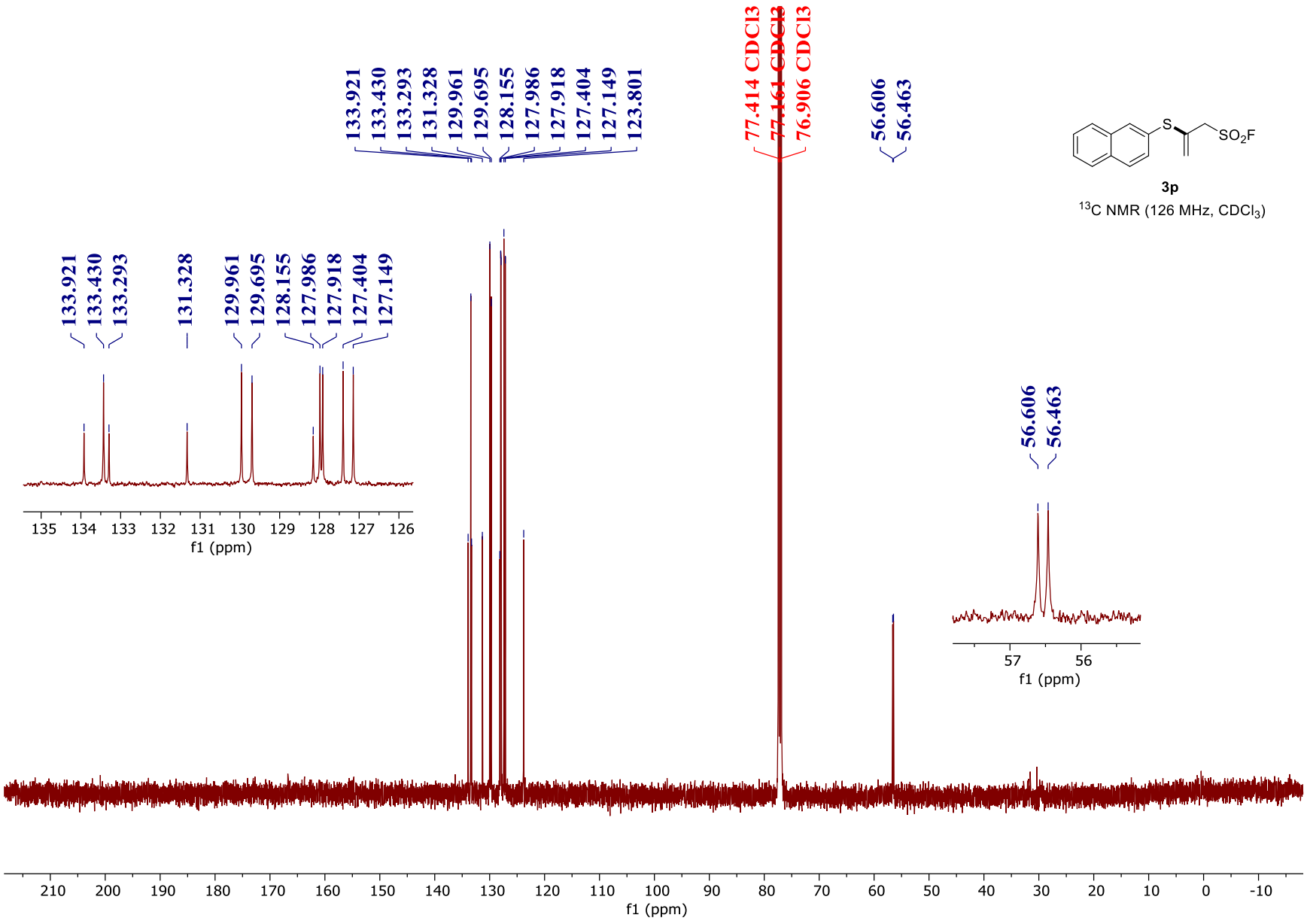

7.263 CDCl3
5.806
5.562
4.227
4.221
F
F
F
F
F
S
SO2F
3o
1H NMR (500 MHz, CDCl3)
0.97
0.94
2.13
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

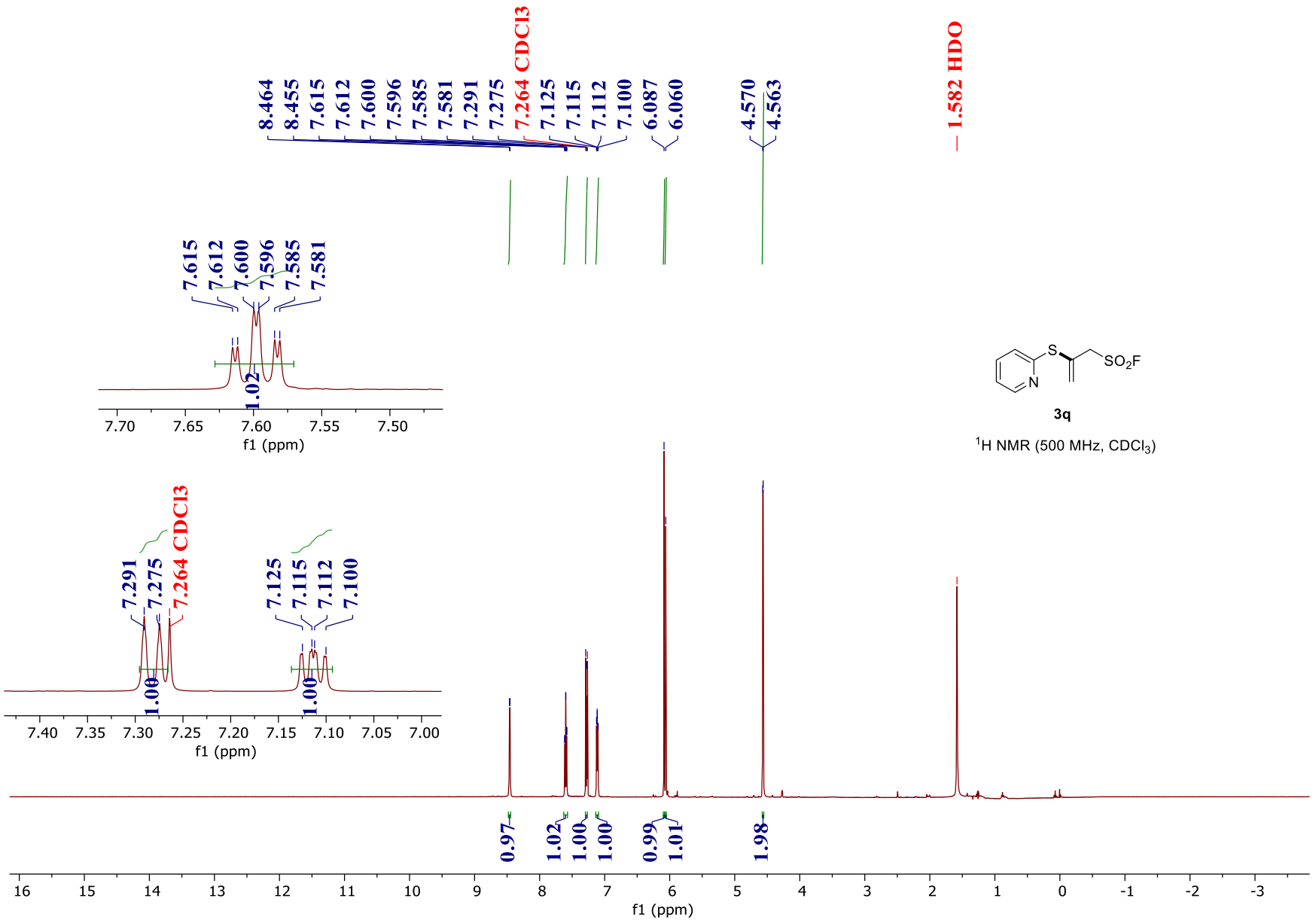

-55.412
-130.674
-130.691
-130.734
-130.756
-148.295
-148.339
-148.383
-159.027
-159.045
-159.077
-159.089
-159.122
-159.137
F
F
S
SO2F
F
F
F
3o
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

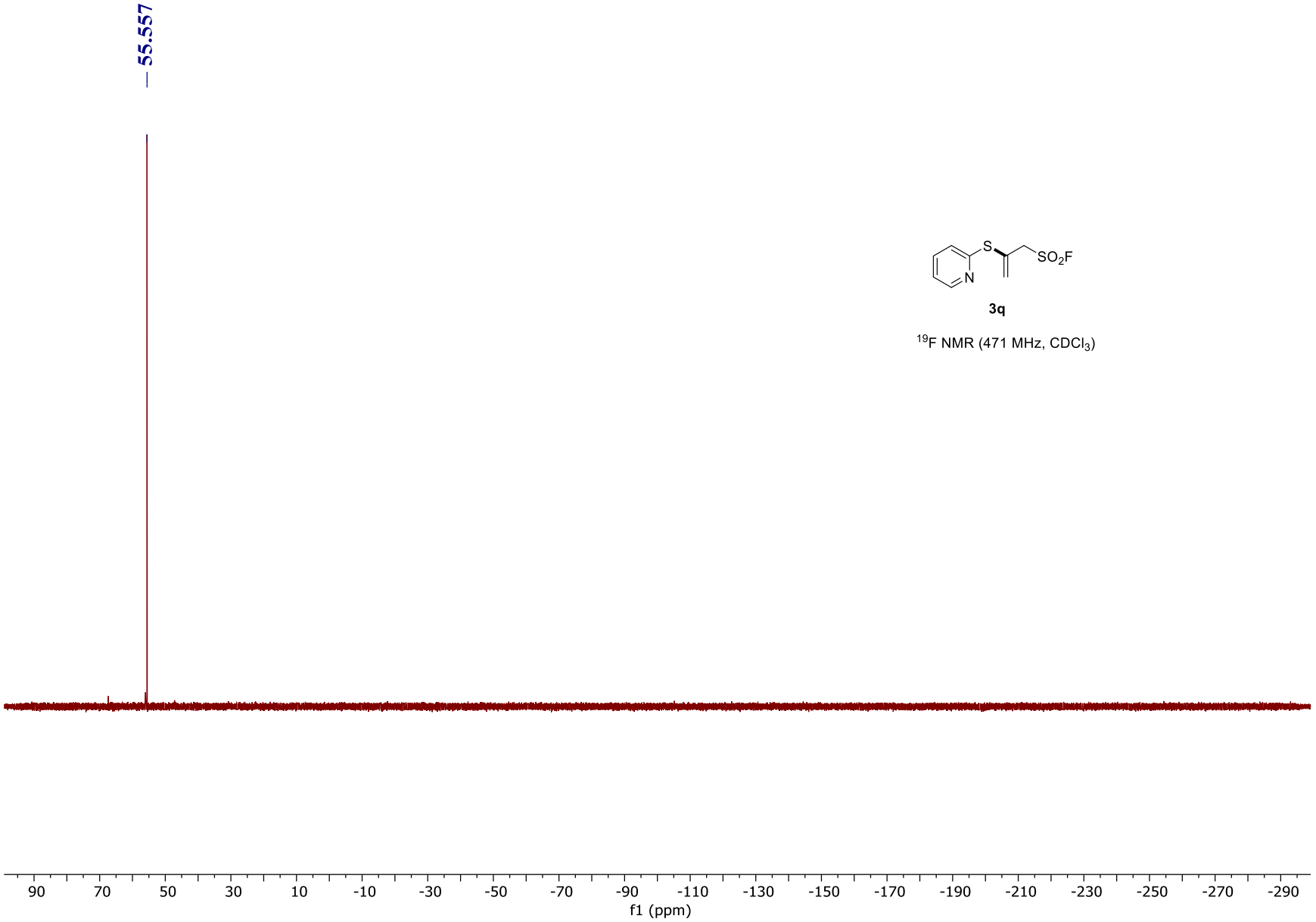

148.773
148.686
148.654
146.783
146.694
146.663
139.190
137.149
127.665
127.379
127.110
125.778
77.415 CDCl3
77.160 CDCl3
76.906 CDCl3
56.860
56.706
F
F
F
F
F
S
SO2F
3o
13C NMR (126 MHz, CDCl3)
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10


8.003
7.873
7.857
7.836
7.826
7.820
7.556
7.550
7.541
7.510
7.493
7.263 CDCl3
5.736
5.545
4.189
4.183
1.550 HDO
0.97
1.98
1.12
2.07
1.04
0.97
0.95
1.93
f1 (ppm)
SO2F
3p
1H NMR (500 MHz, CDCl3)


55.845
S
SO2F
3p
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

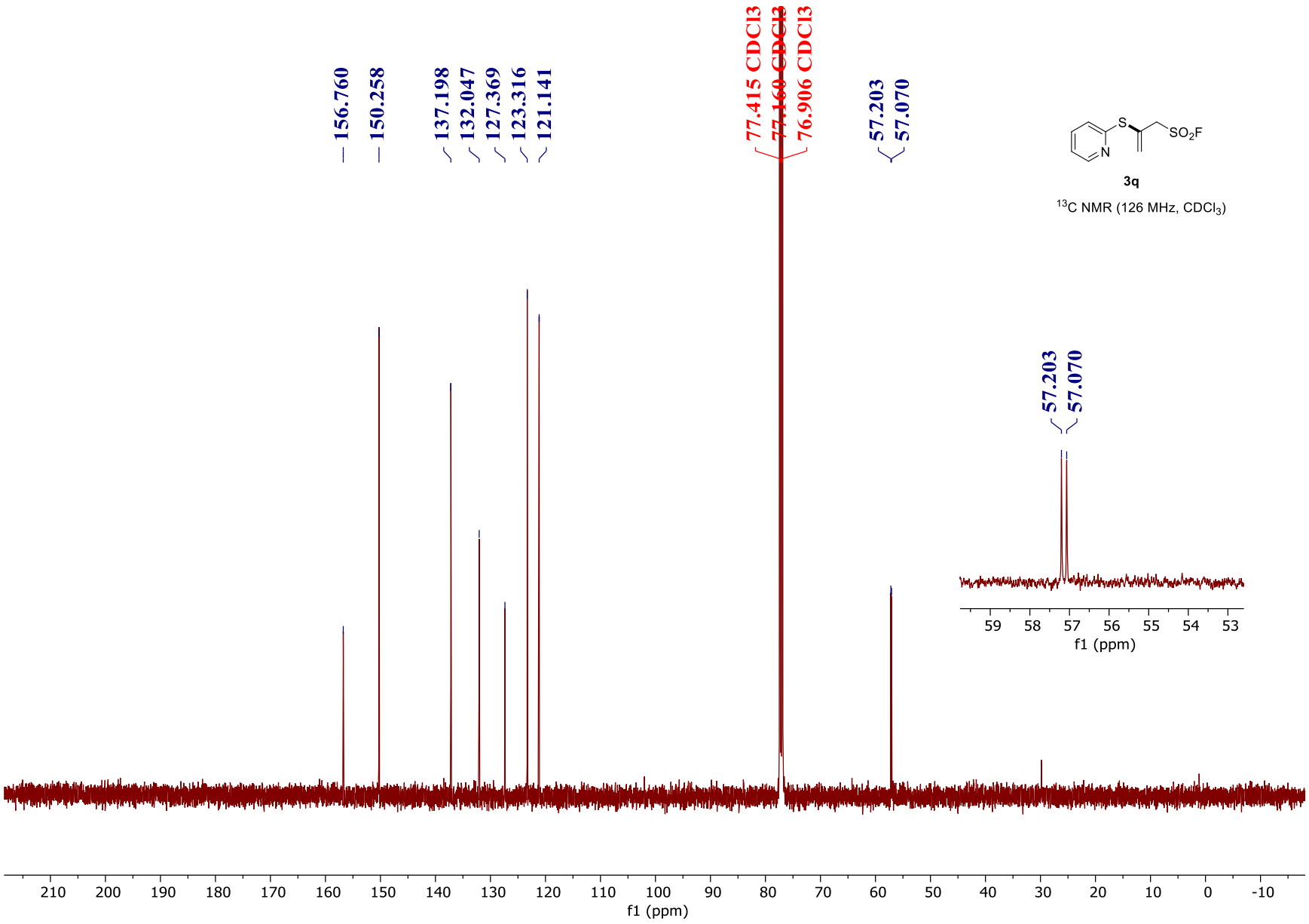

133.921
133.430
133.293
131.328
129.961
129.695
128.155
127.986
127.918
127.404
127.149
123.801
77.414 CDCl3
77.161 CDCl3
76.906 CDCl3
56.606
56.463
S
SO2F
3p
13C NMR (126 MHz, CDCl3)
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

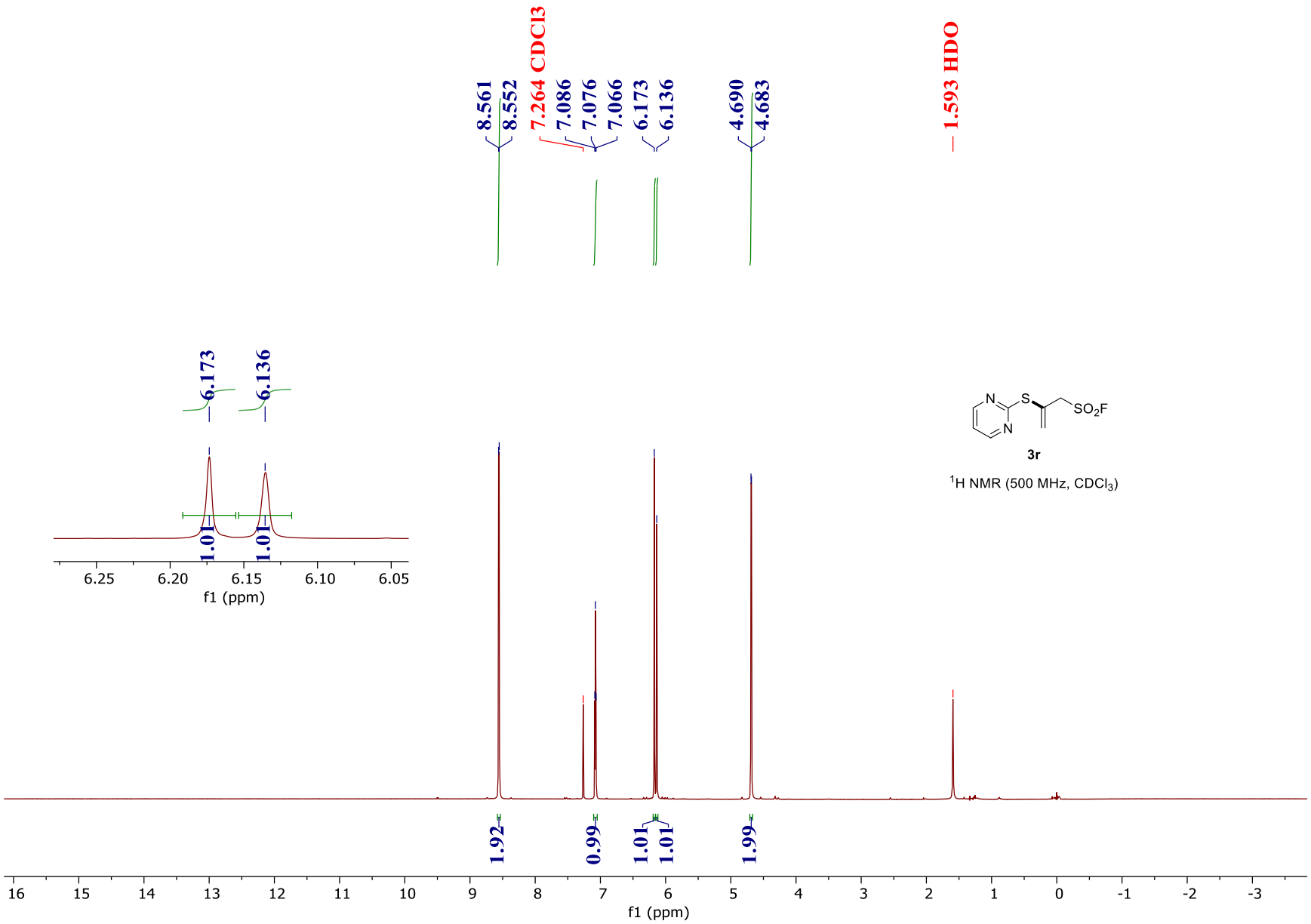

8.464
8.455
7.615
7.612
7.600
7.596
7.585
7.581
7.291
7.275
7.264 CDCl3
7.125
7.115
7.112
7.100
6.087
6.060
4.570
4.563
1.582 HDO
7.615
7.612
7.600
7.596
7.585
7.581
1.02
7.70 7.65 7.60 7.55 7.50
f1 (ppm)
7.291
7.275
7.264 CDCl3
7.125
7.115
7.112
7.100
1.00
1.00
7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00
f1 (ppm)
0.97
1.02
1.00
1.00
0.99
1.01
1.98
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)
S
N
SO2F
3q
1H NMR (500 MHz, CDCl3)

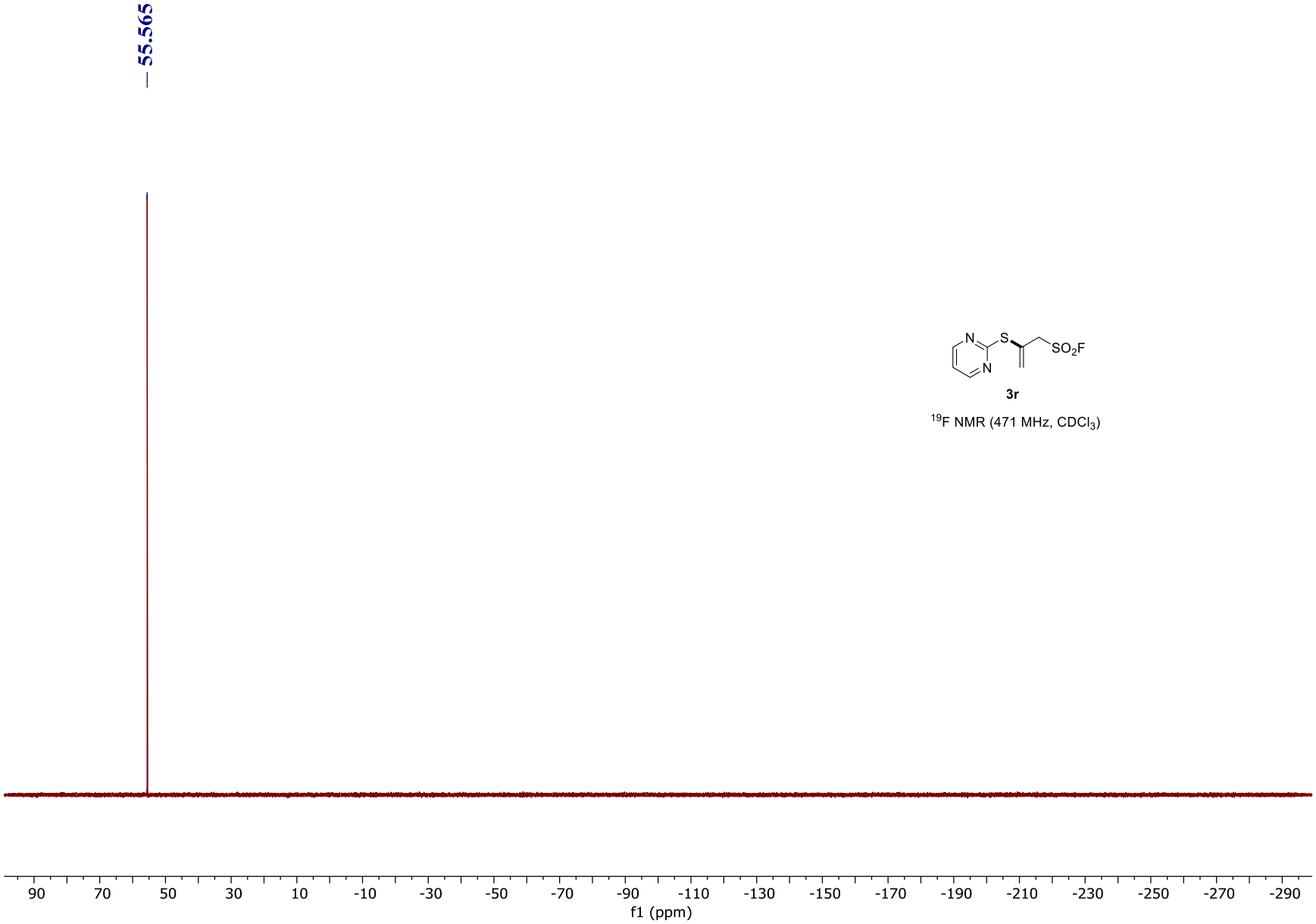


3q

^{19}F NMR (471 MHz, $CDCl_3$)

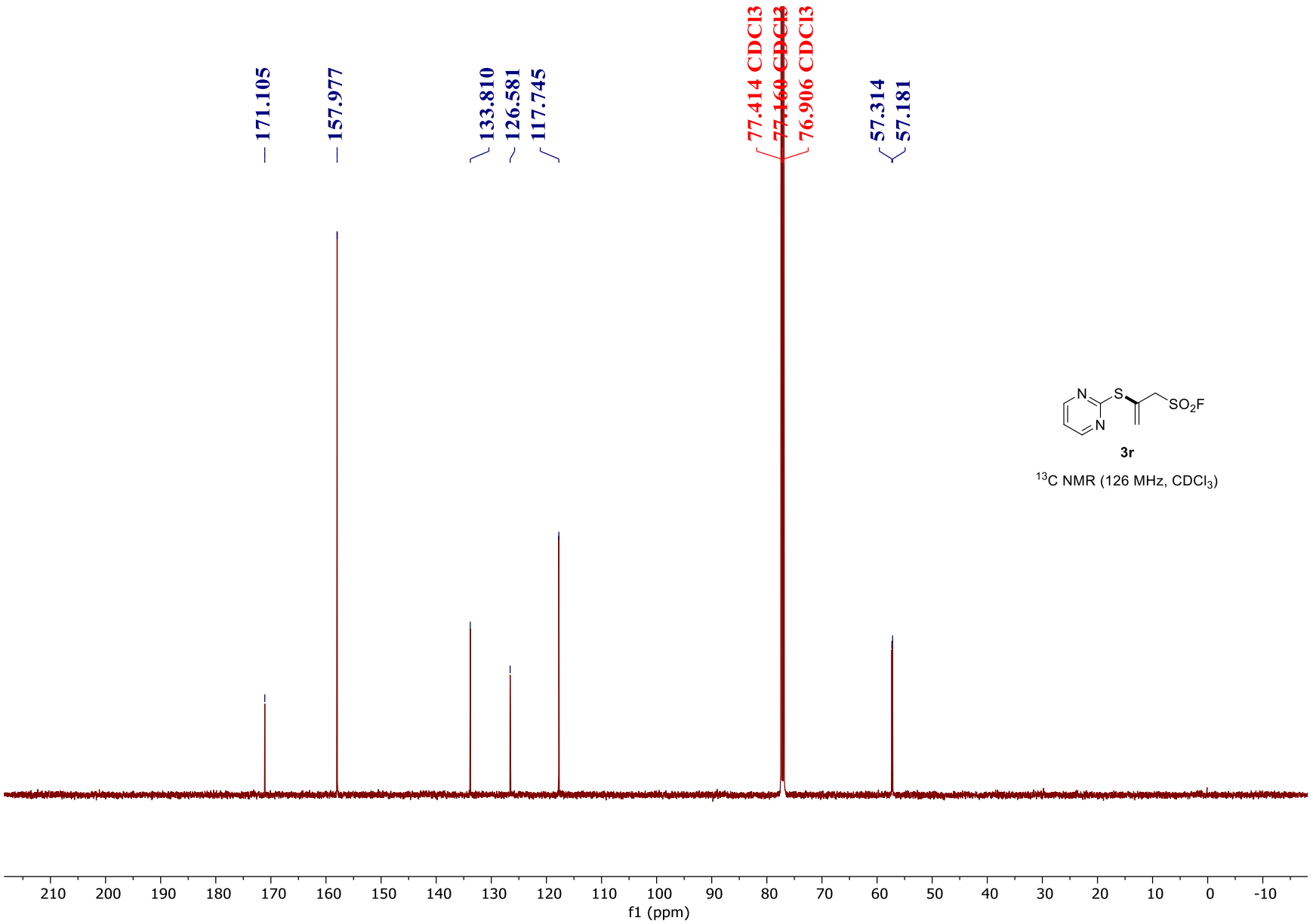

156.760
150.258
137.198
132.047
127.369
123.316
121.141
77.415 CDCl3
77.160 CDCl3
76.906 CDCl3
57.203
57.070
3q
13C NMR (126 MHz, CDCl3)
f1 (ppm)

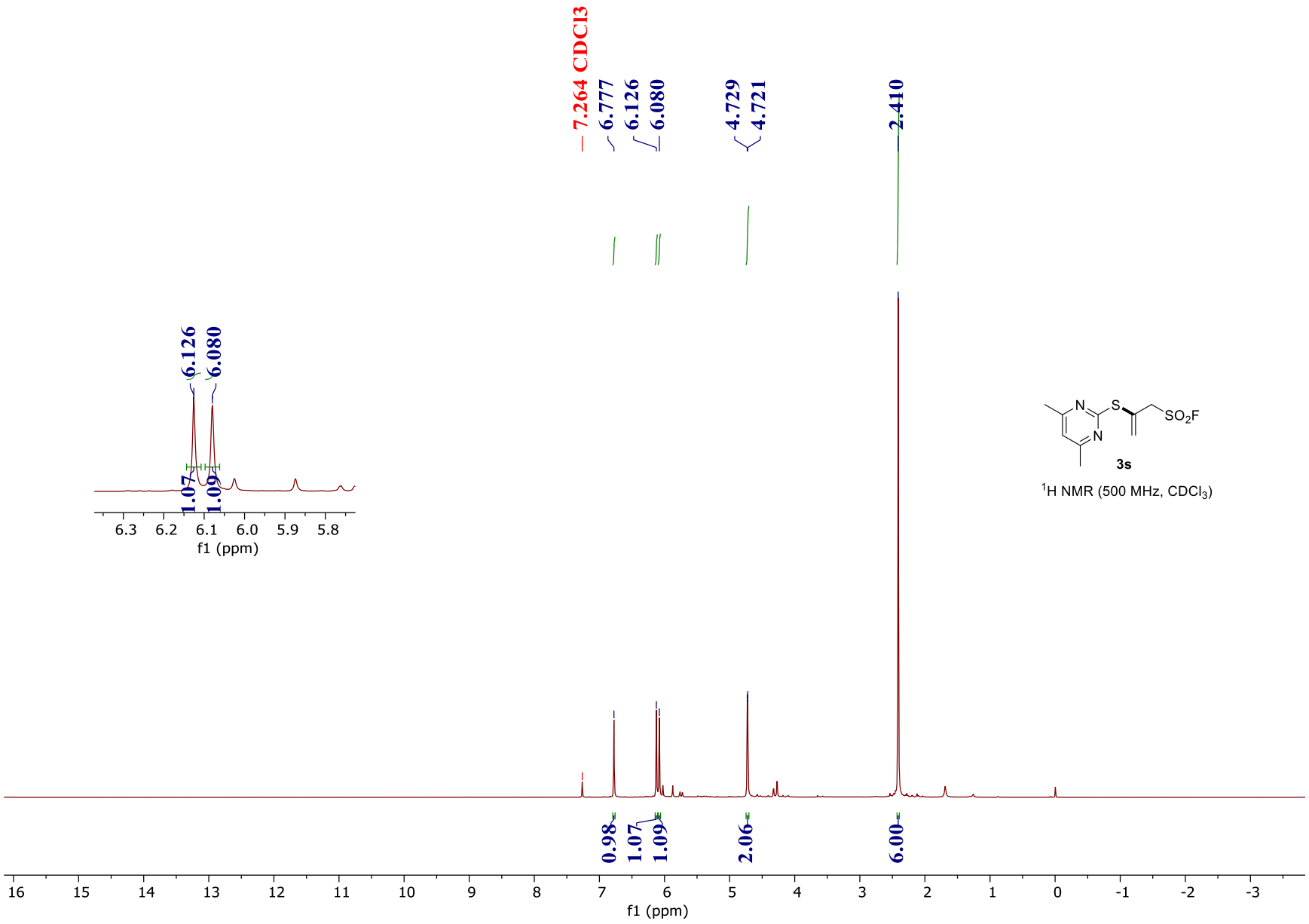

8.561
8.552
7.264 CDCl3
7.086
7.076
7.066
6.173
6.136
4.690
4.683
1.593 HDO
6.173
6.136
1.01
1.01
6.25
6.20
6.15
6.10
6.05
f1 (ppm)
N
S
N
SO2F
3r
1H NMR (500 MHz, CDCl3)
1.92
0.99
1.01
1.01
1.99
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

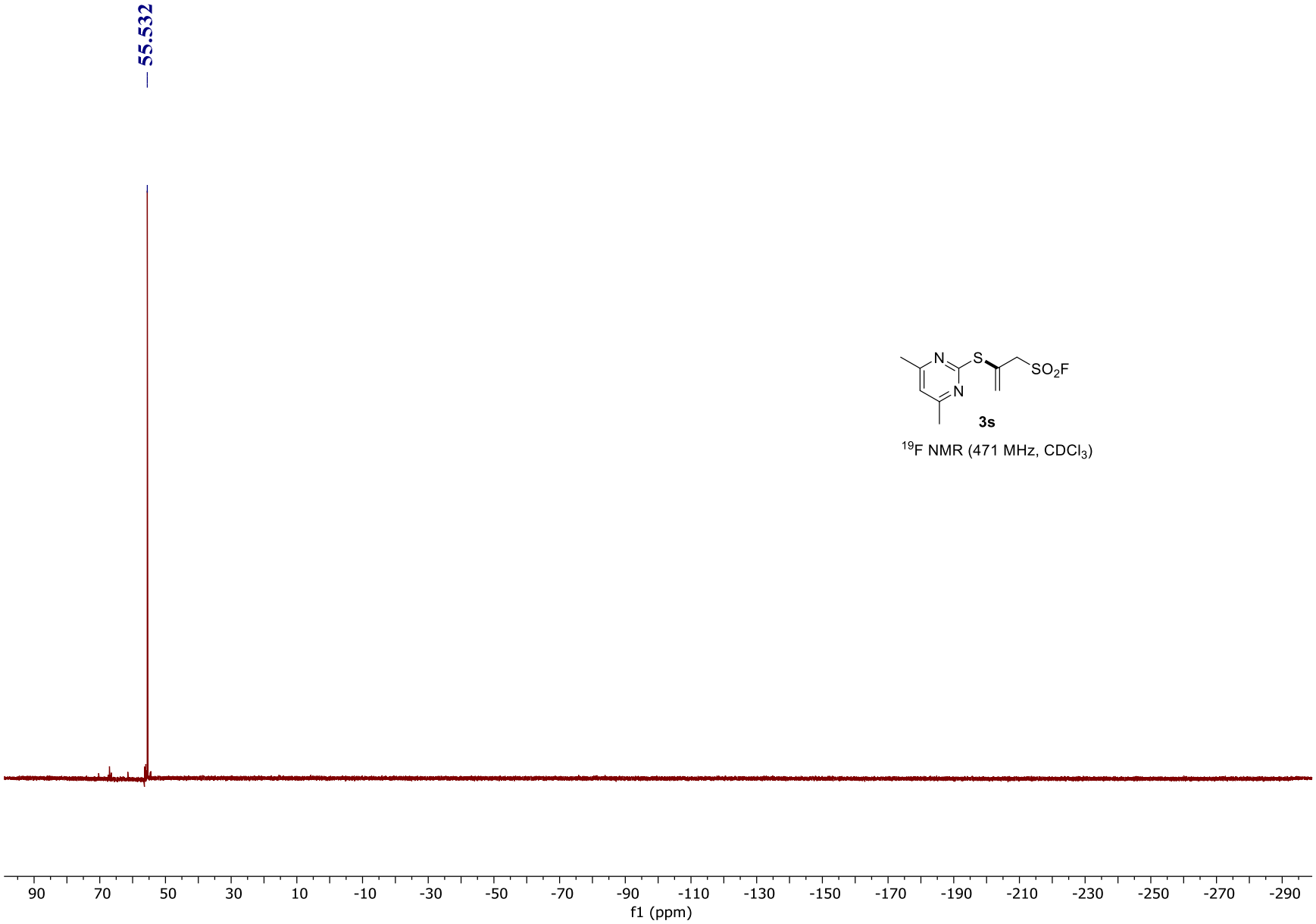


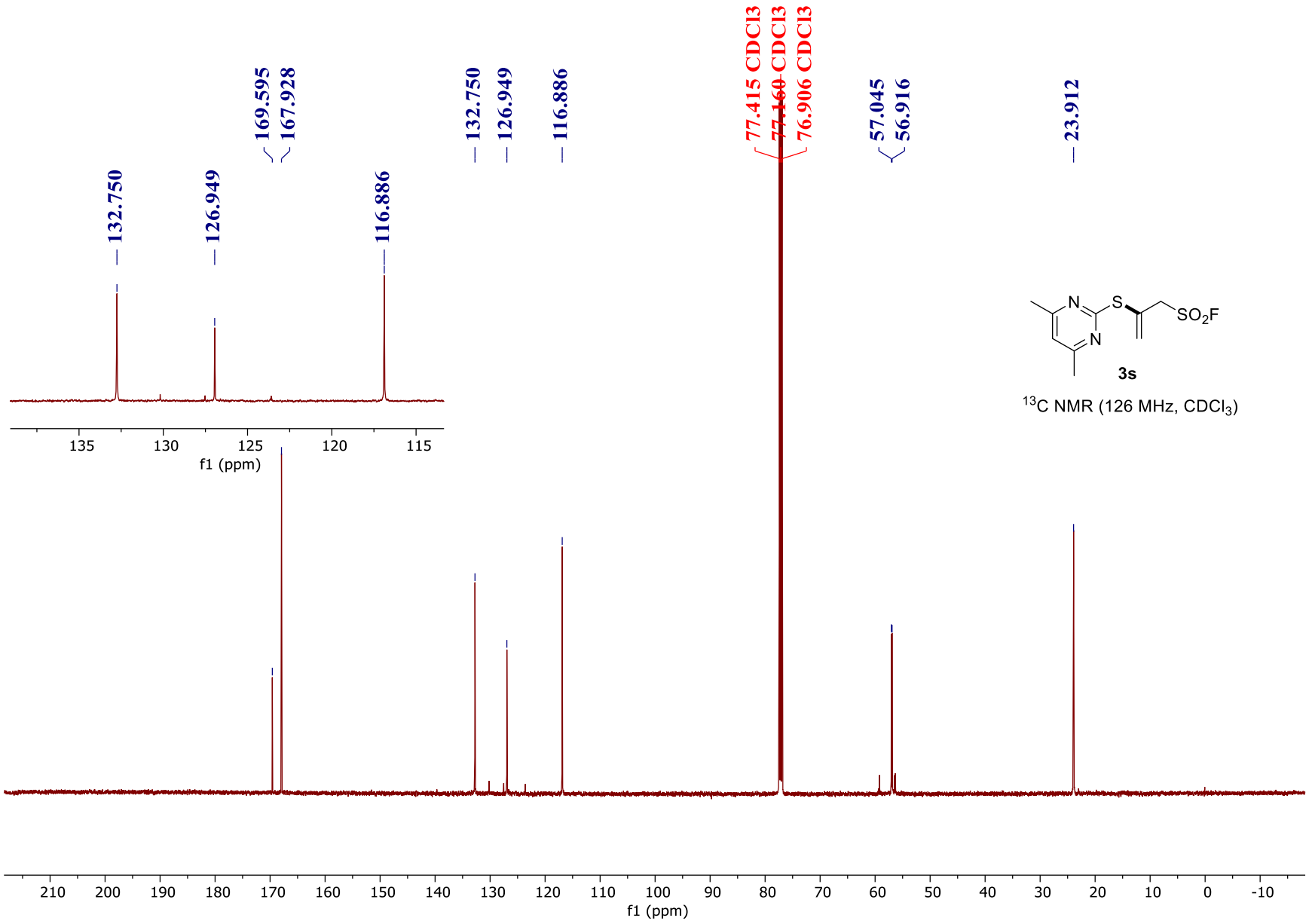
3r

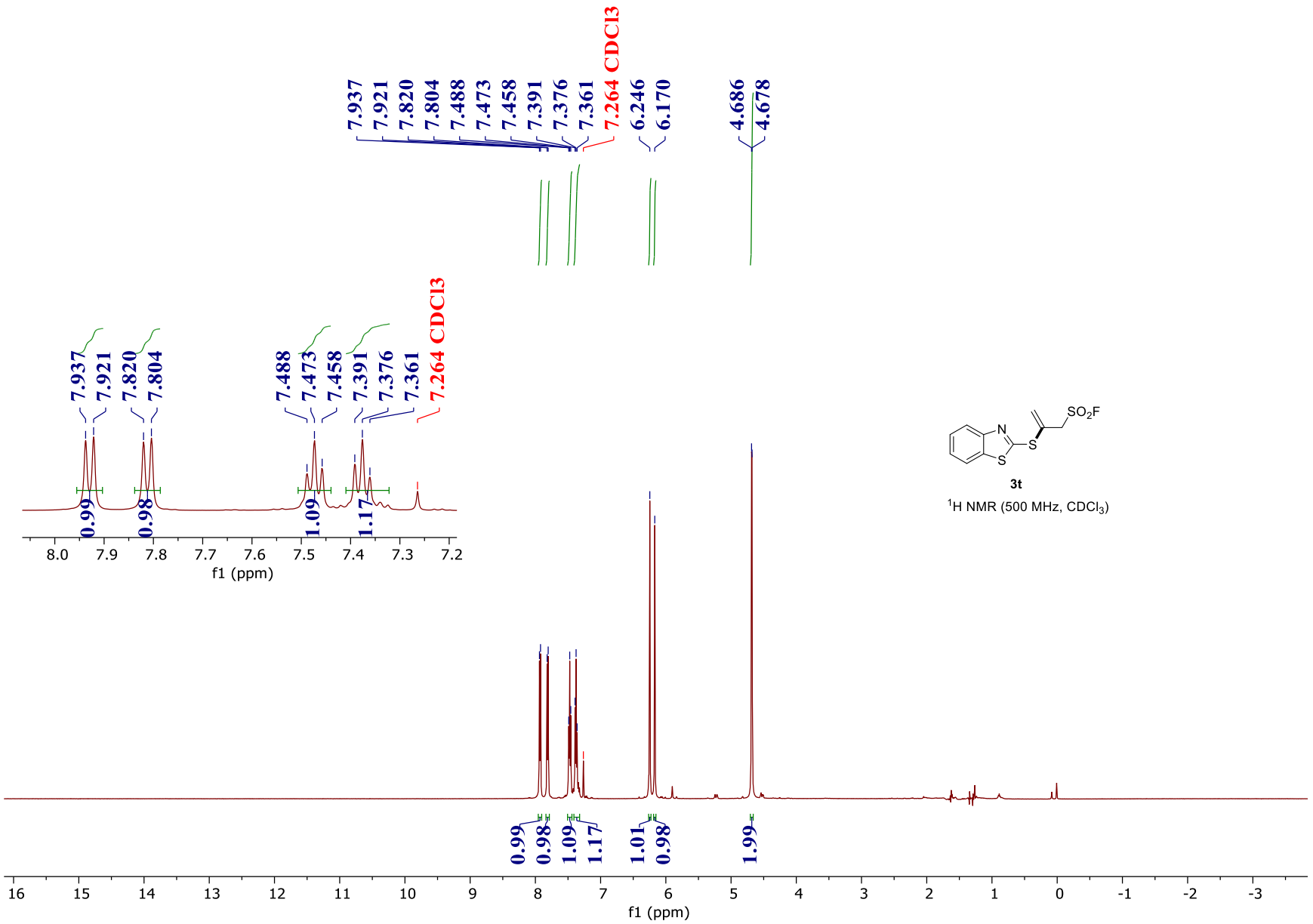
^{19}F NMR (471 MHz, $CDCl_3$)

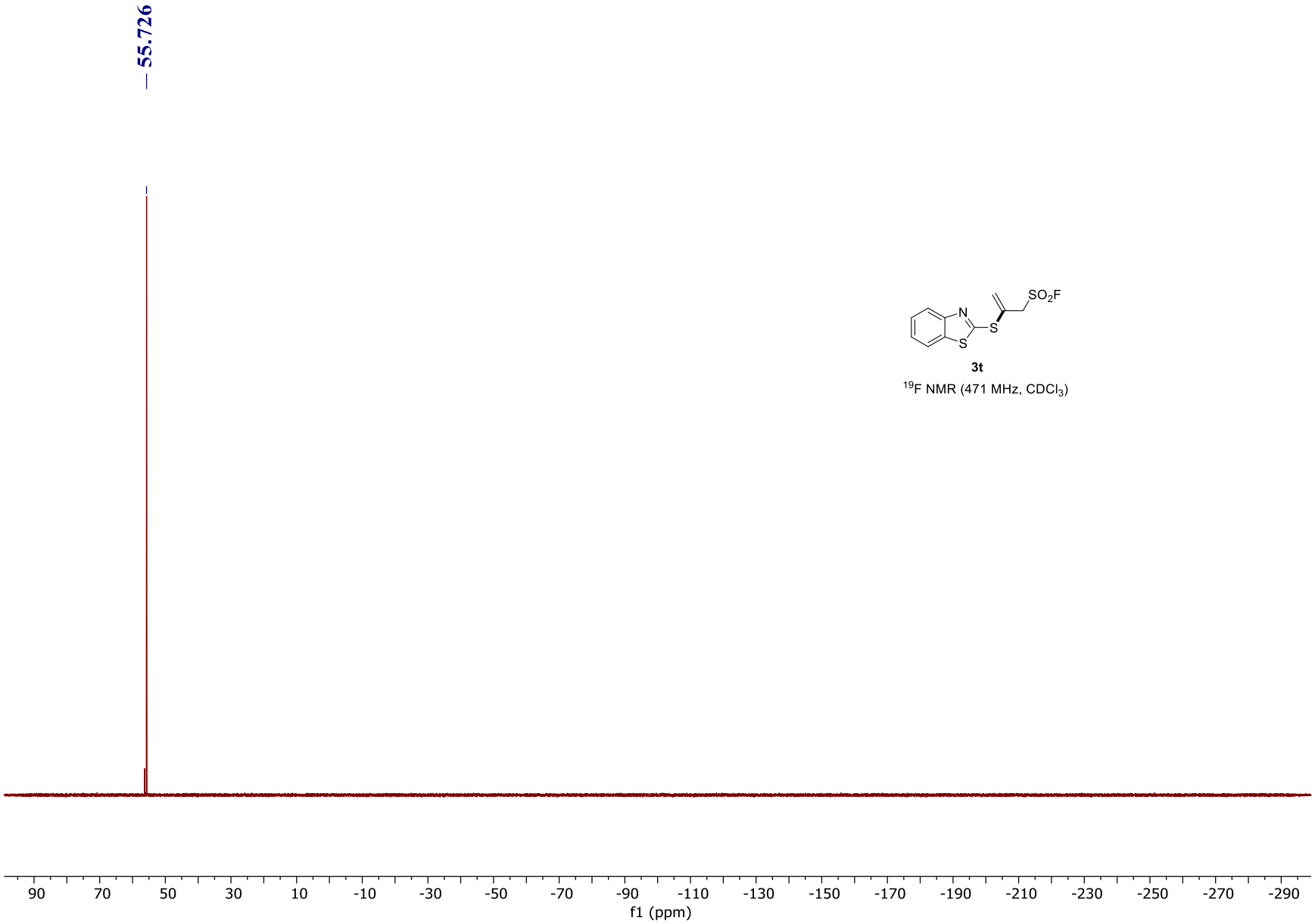

171.105
157.977
133.810
126.581
117.745
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
57.314
57.181
N
S
N
SO2F
3r
13C NMR (126 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.264 CDCl3
6.777
6.126
6.080
4.729
4.721
2.410
6.126
6.080
1.07
1.09
6.3 6.2 6.1 6.0 5.9 5.8
f1 (ppm)
N
S
N
SO2F
3s
1H NMR (500 MHz, CDCl3)
0.98
1.07
1.09
2.06
6.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)


55.532
N
S
N
SO2F
3s
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

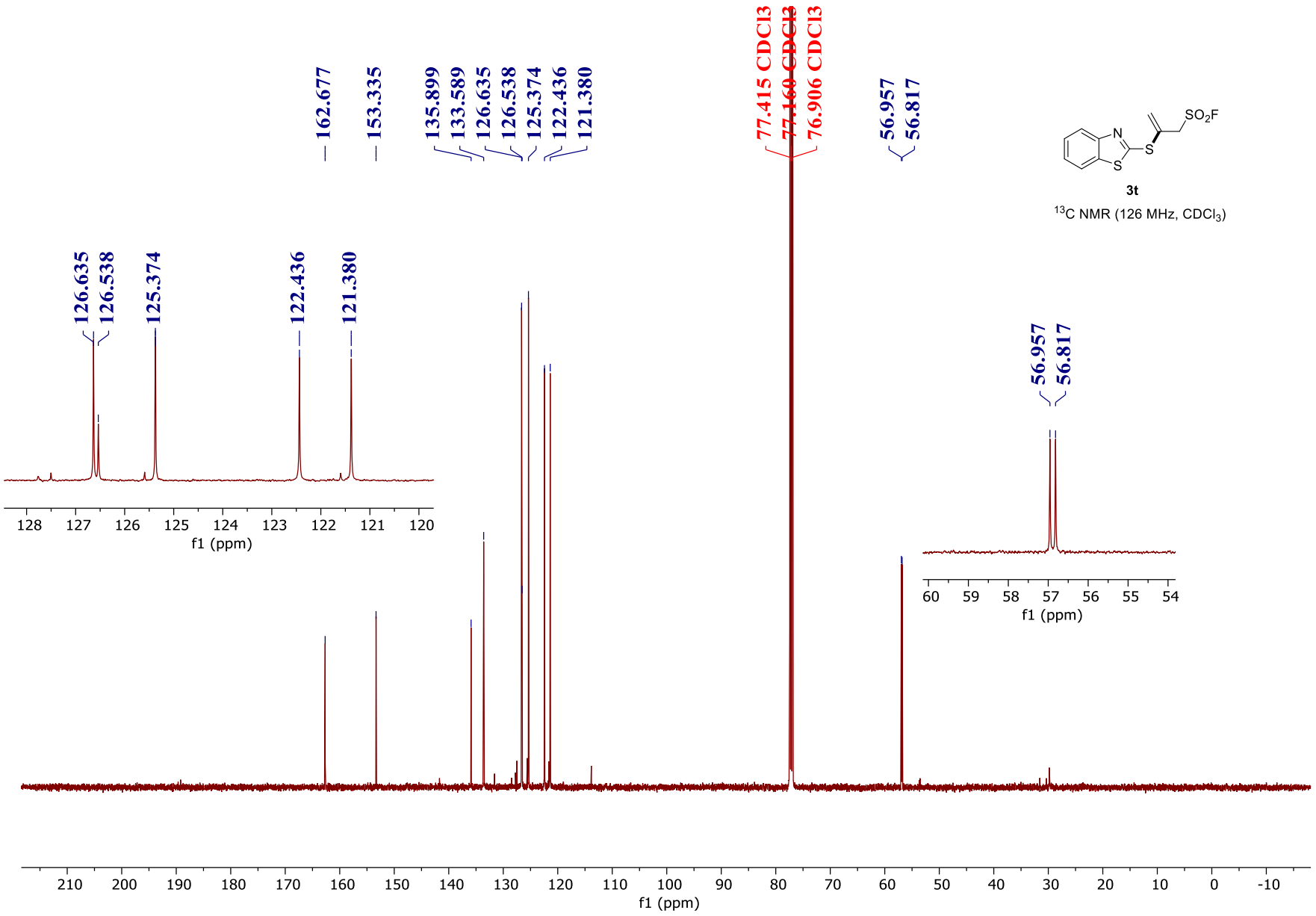

132.750
126.949
169.595
167.928
116.886
132.750
126.949
116.886
77.415 CDCl3
77.160 CDCl3
76.906 CDCl3
57.045
56.916
23.912
f1 (ppm)
f1 (ppm)
N
S
N
SO2F
3s
13C NMR (126 MHz, CDCl3)

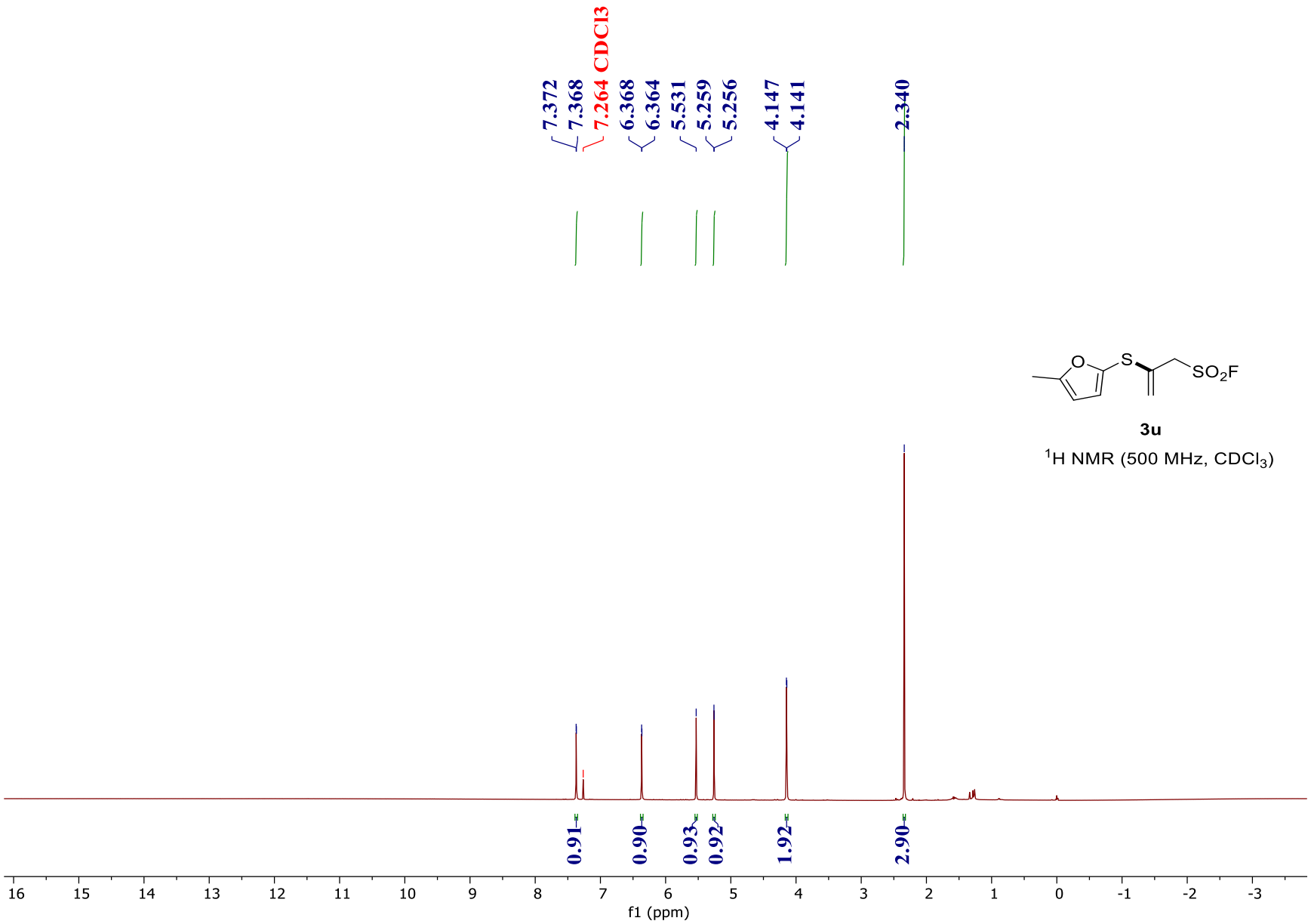

7.937
7.921
7.820
7.804
7.488
7.473
7.458
7.391
7.376
7.361
7.264 CDCl3
6.246
6.170
4.686
4.678
0.99
0.98
1.09
1.17
1.01
0.98
1.99
f1 (ppm)
SO2F
N
S
S
3t
1H NMR (500 MHz, CDCl3)

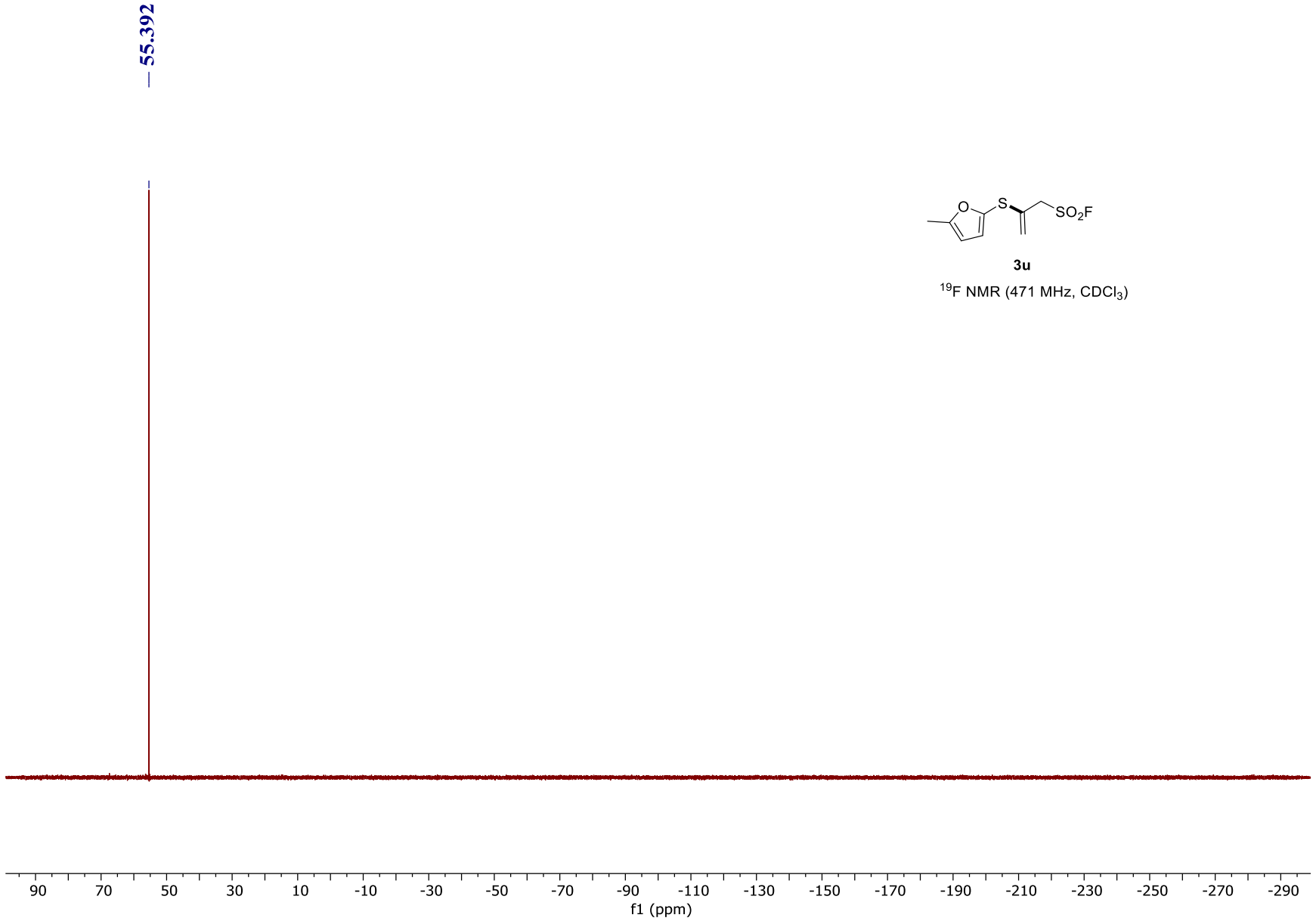


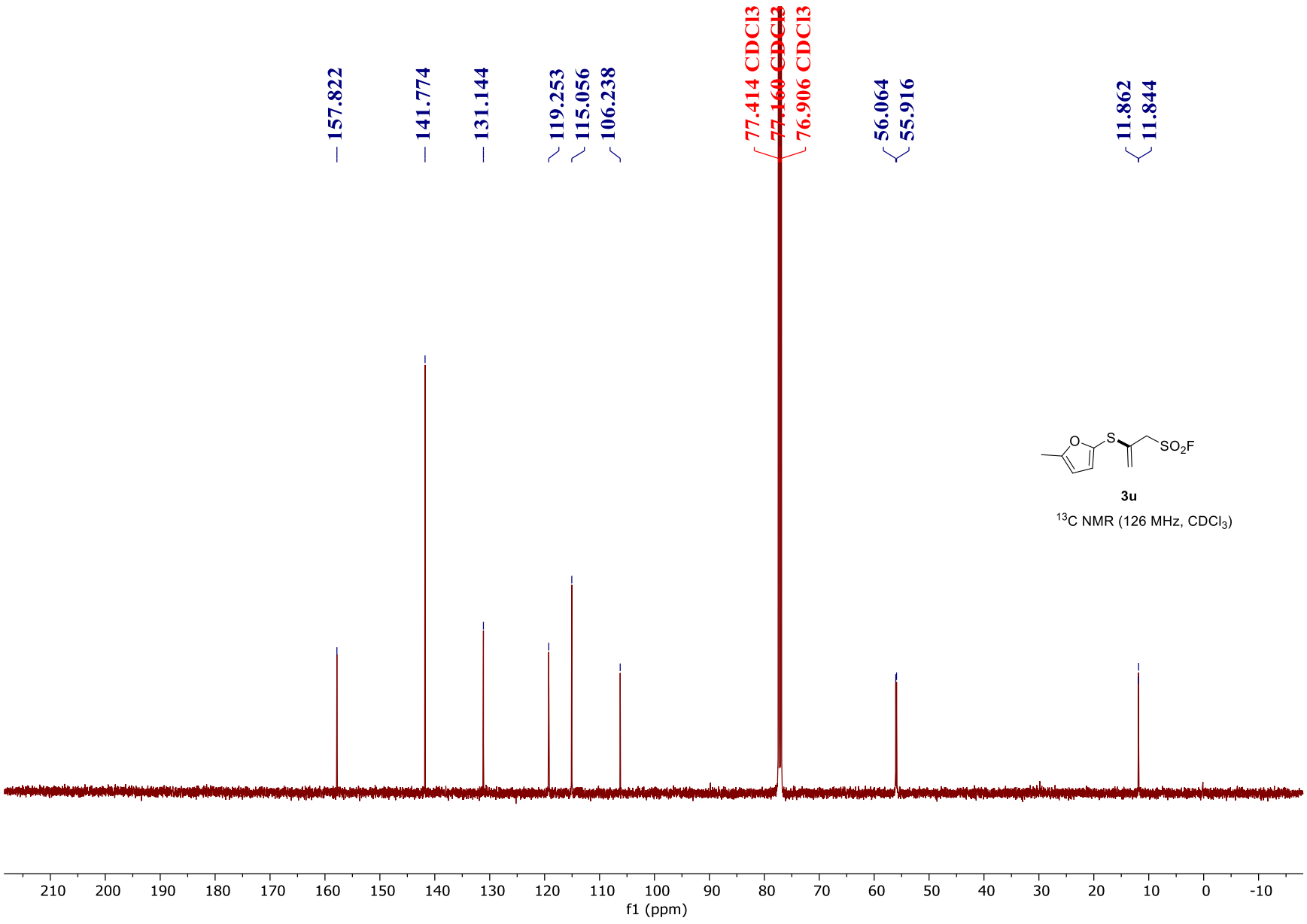
3t

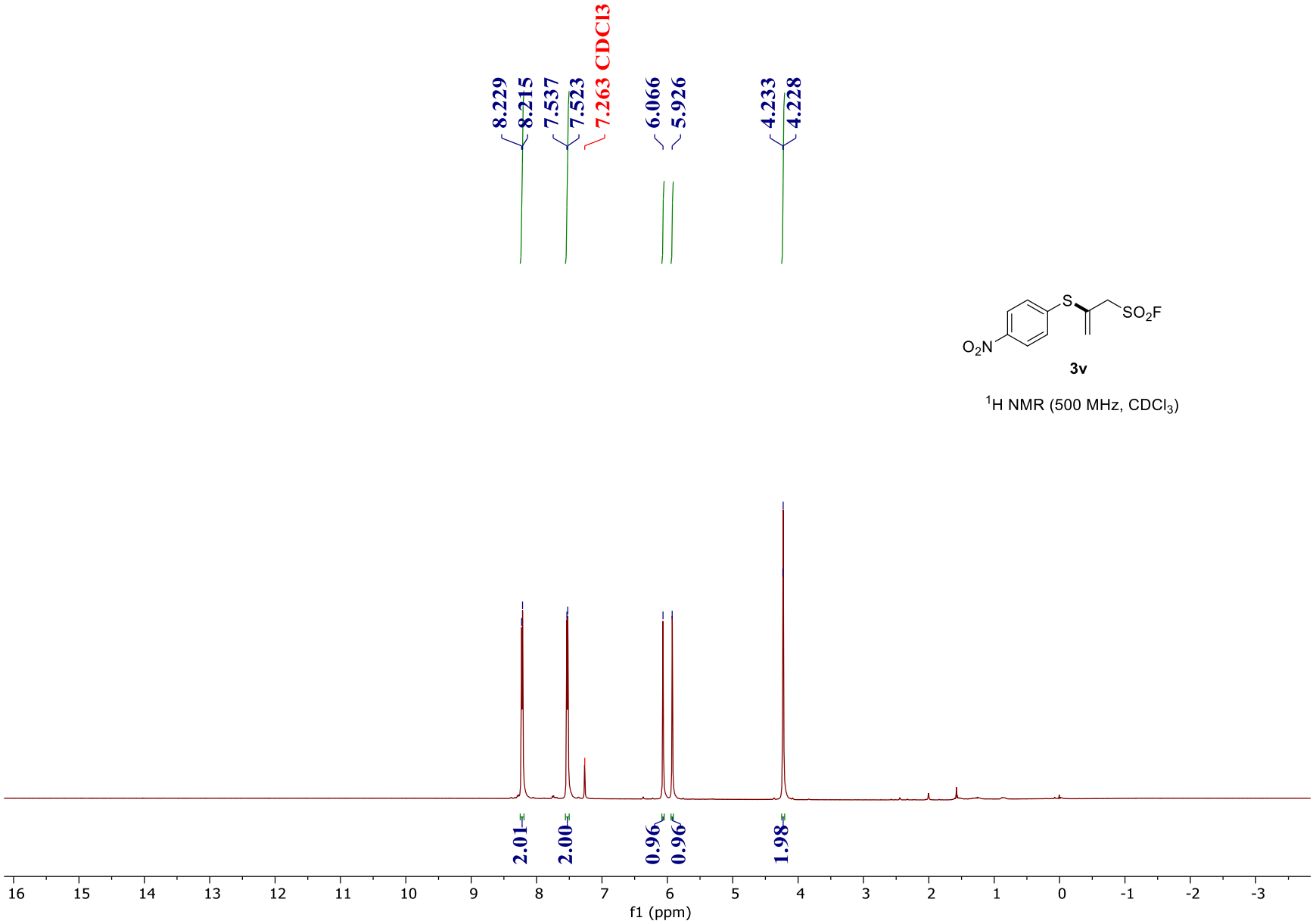
^{19}F NMR (471 MHz, $CDCl_3$)

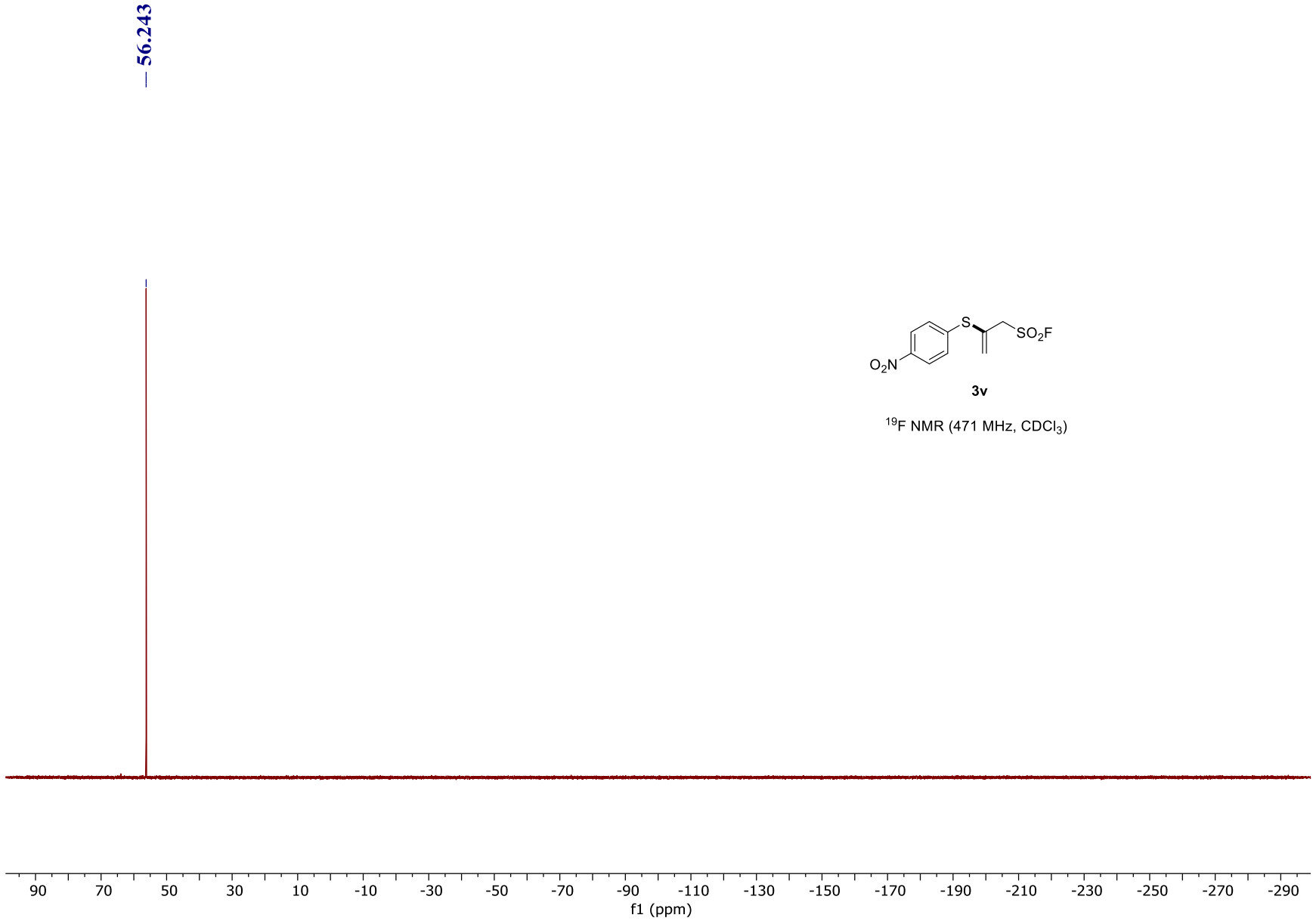

162.677
153.335
135.899
133.589
126.635
126.538
125.374
122.436
121.380
77.415 CDCl3
77.160 CDCl3
76.906 CDCl3
56.957
56.817
SO2F
N
S
S
3t
13C NMR (126 MHz, CDCl3)
f1 (ppm)
f1 (ppm)
f1 (ppm)

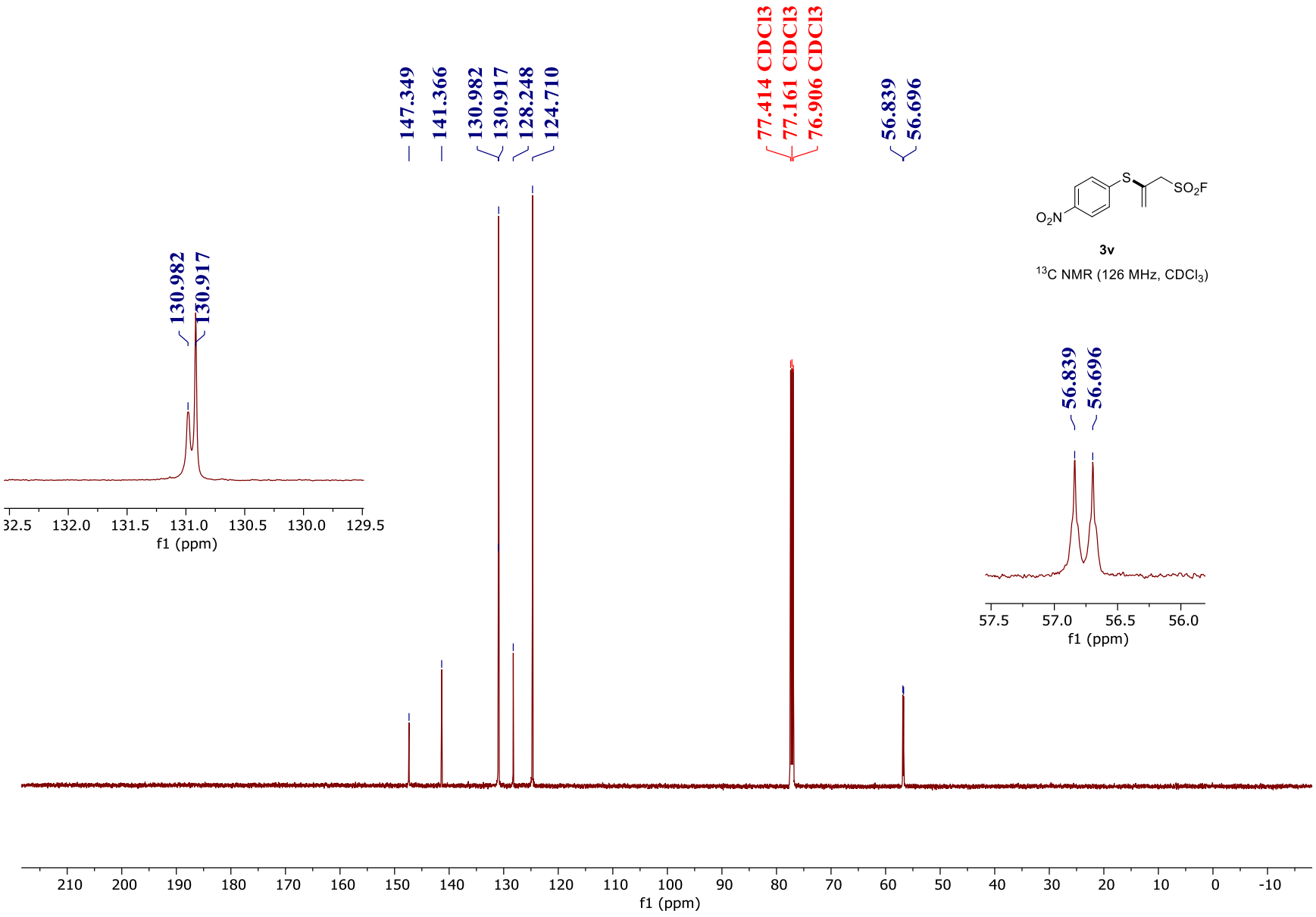

7.372
7.368
7.264 CDCl3
6.368
6.364
5.531
5.259
5.256
4.147
4.141
2.340
O
S
SO2F
3u
1H NMR (500 MHz, CDCl3)
0.91
0.90
0.93
0.92
1.92
2.90
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

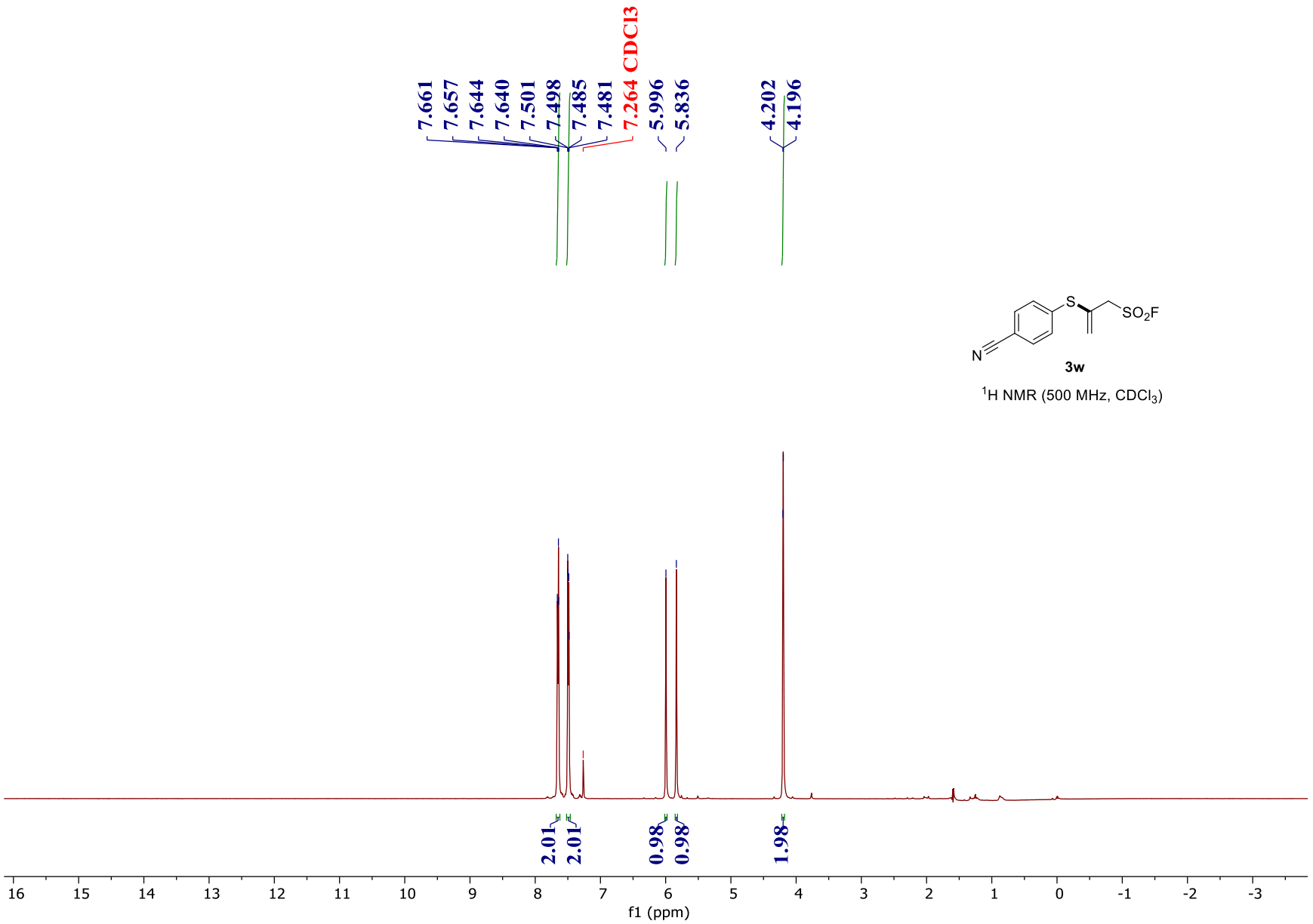

55.392
O
S
SO2F
3u
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

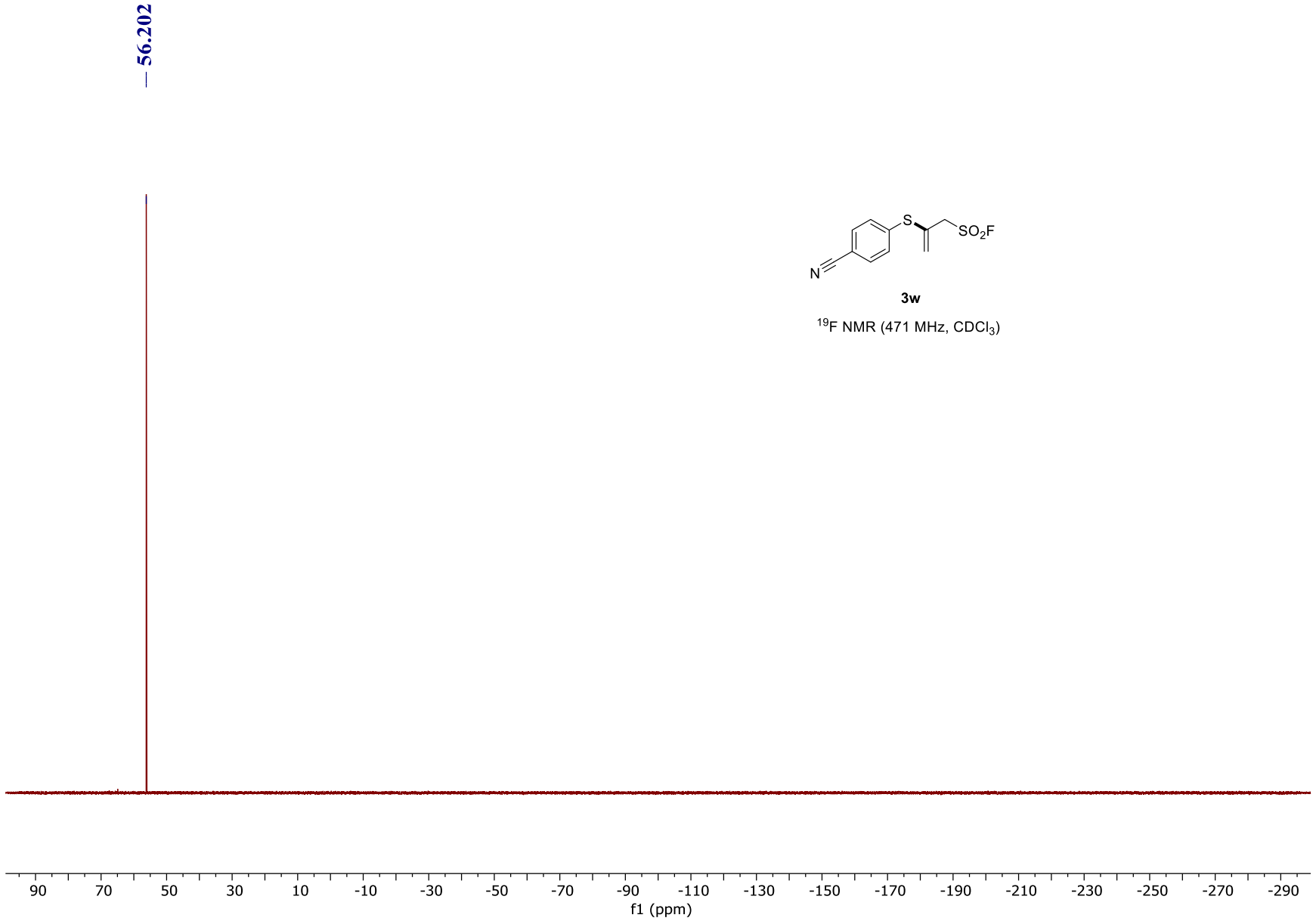

157.822
141.774
131.144
119.253
115.056
106.238
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
56.064
55.916
11.862
11.844
O
S
SO2F
3u
13C NMR (126 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

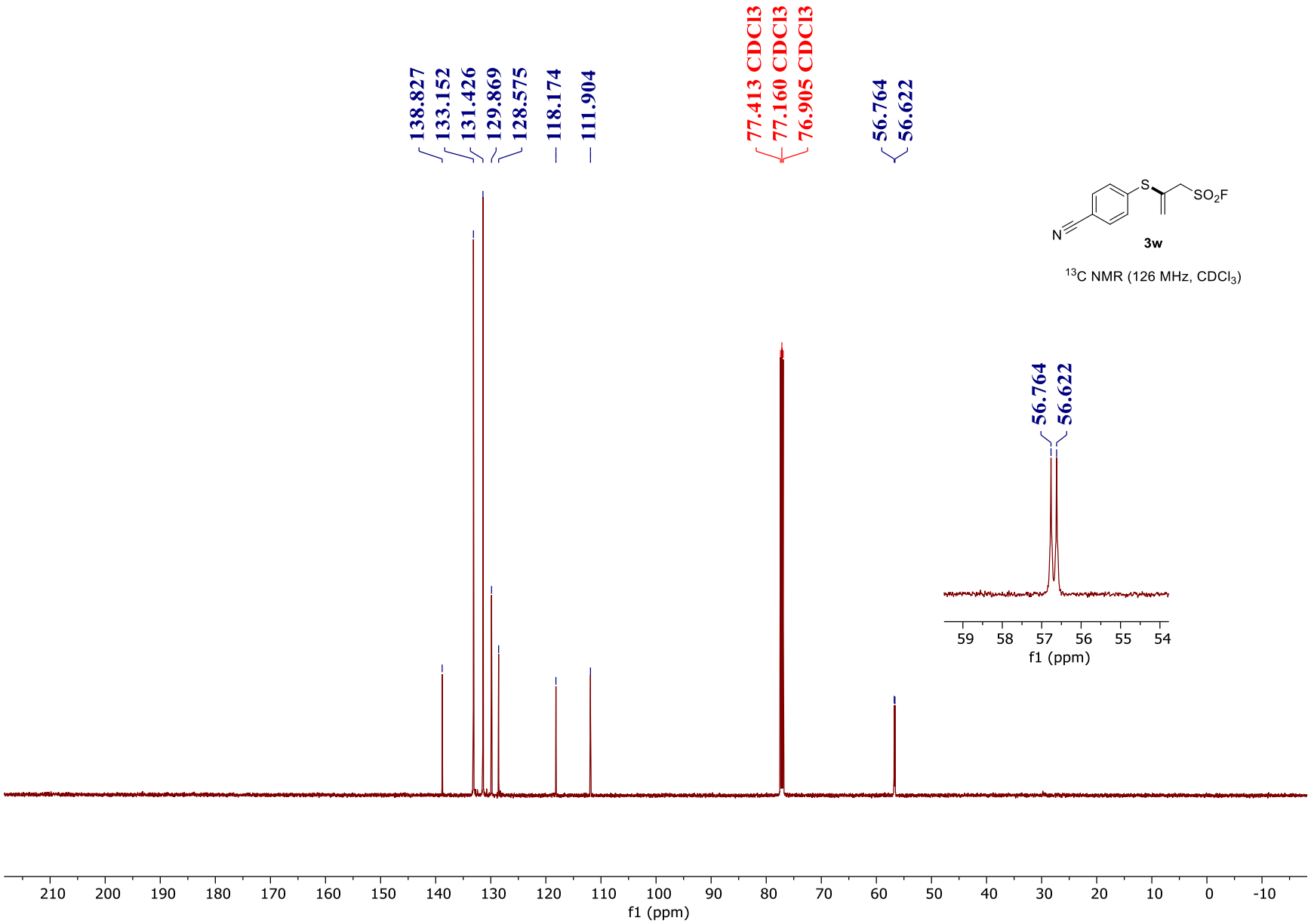

8.229
8.215
7.537
7.523
7.263 CDCl3
6.066
5.926
4.233
4.228
S
SO2F
O2N
3v
1H NMR (500 MHz, CDCl3)
2.01
2.00
0.96
0.96
1.98
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

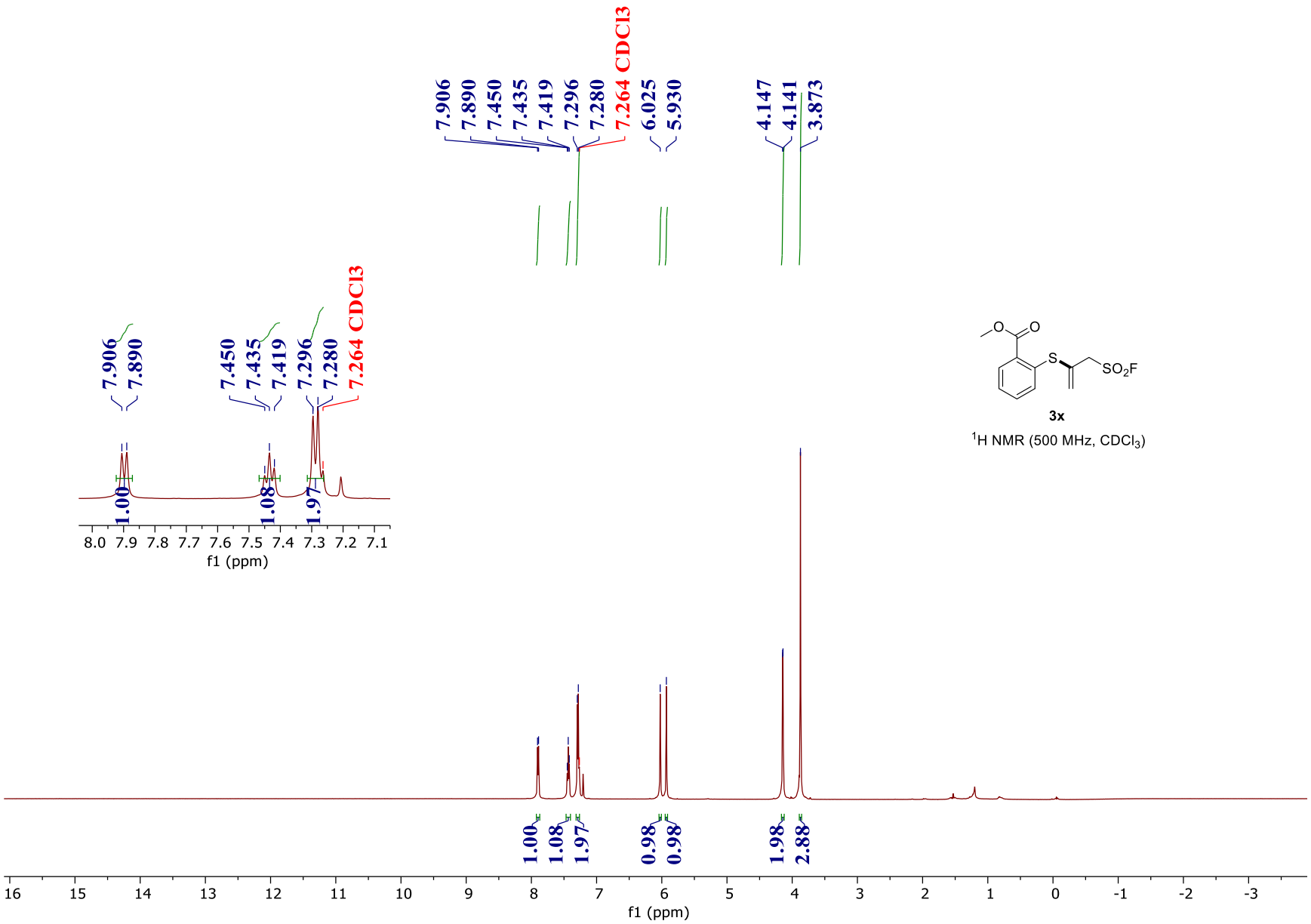

— 56.243
S
SO2F
O2N
3v
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

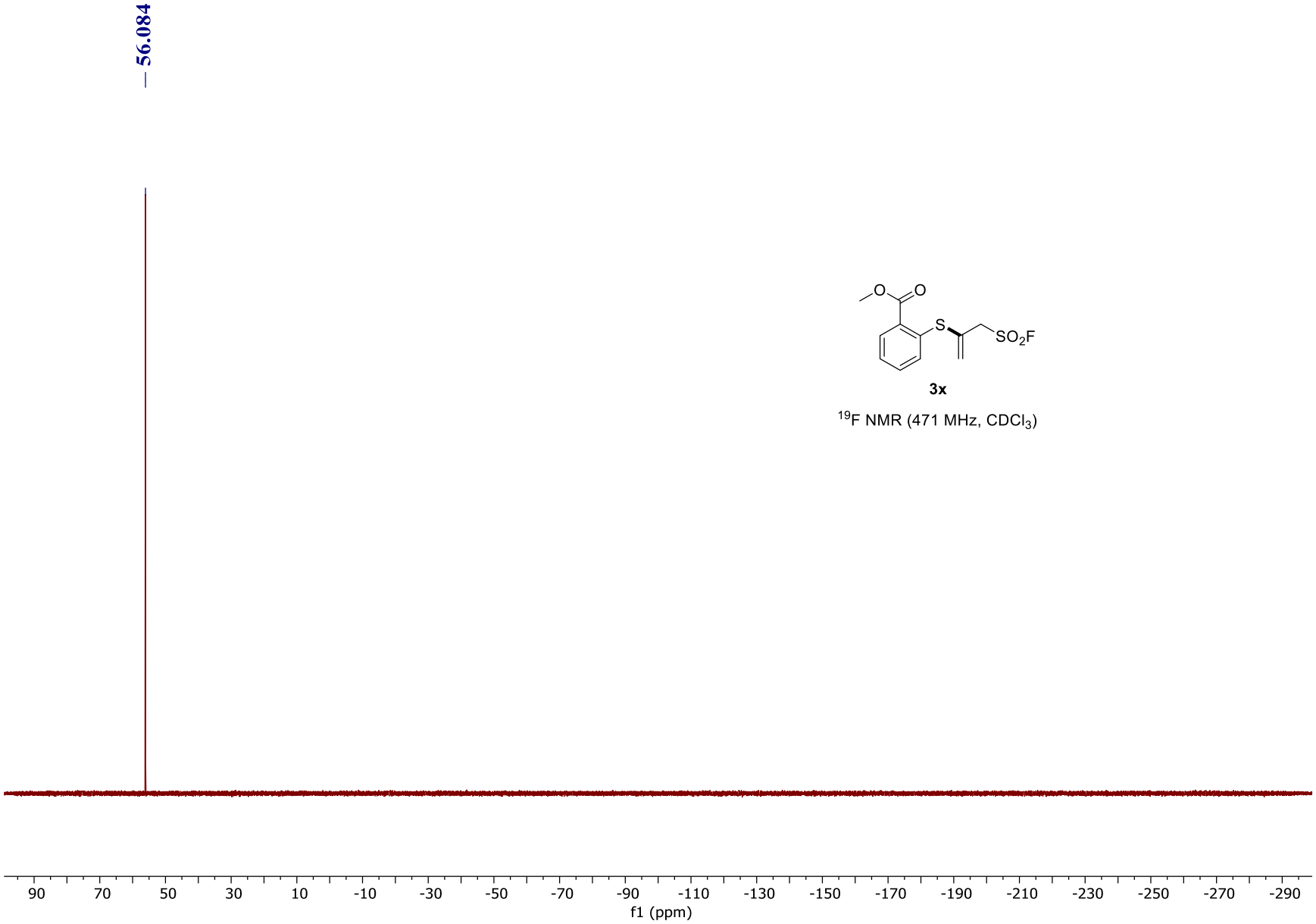

147.349
141.366
130.982
130.917
128.248
124.710
77.414 CDCl3
77.161 CDCl3
76.906 CDCl3
56.839
56.696
130.982
130.917
32.5 132.0 131.5 131.0 130.5 130.0 129.5
f1 (ppm)
56.839
56.696
57.5 57.0 56.5 56.0
f1 (ppm)
S
SO2F
O2N
3v
13C NMR (126 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.661
7.657
7.644
7.640
7.501
7.498
7.485
7.481
7.264 CDCl3
5.996
5.836
4.202
4.196
S
SO2F
N
3w
1H NMR (500 MHz, CDCl3)
2.01
2.01
0.98
0.98
1.98
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)


— 56.202
S
SO2F
N
3w
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

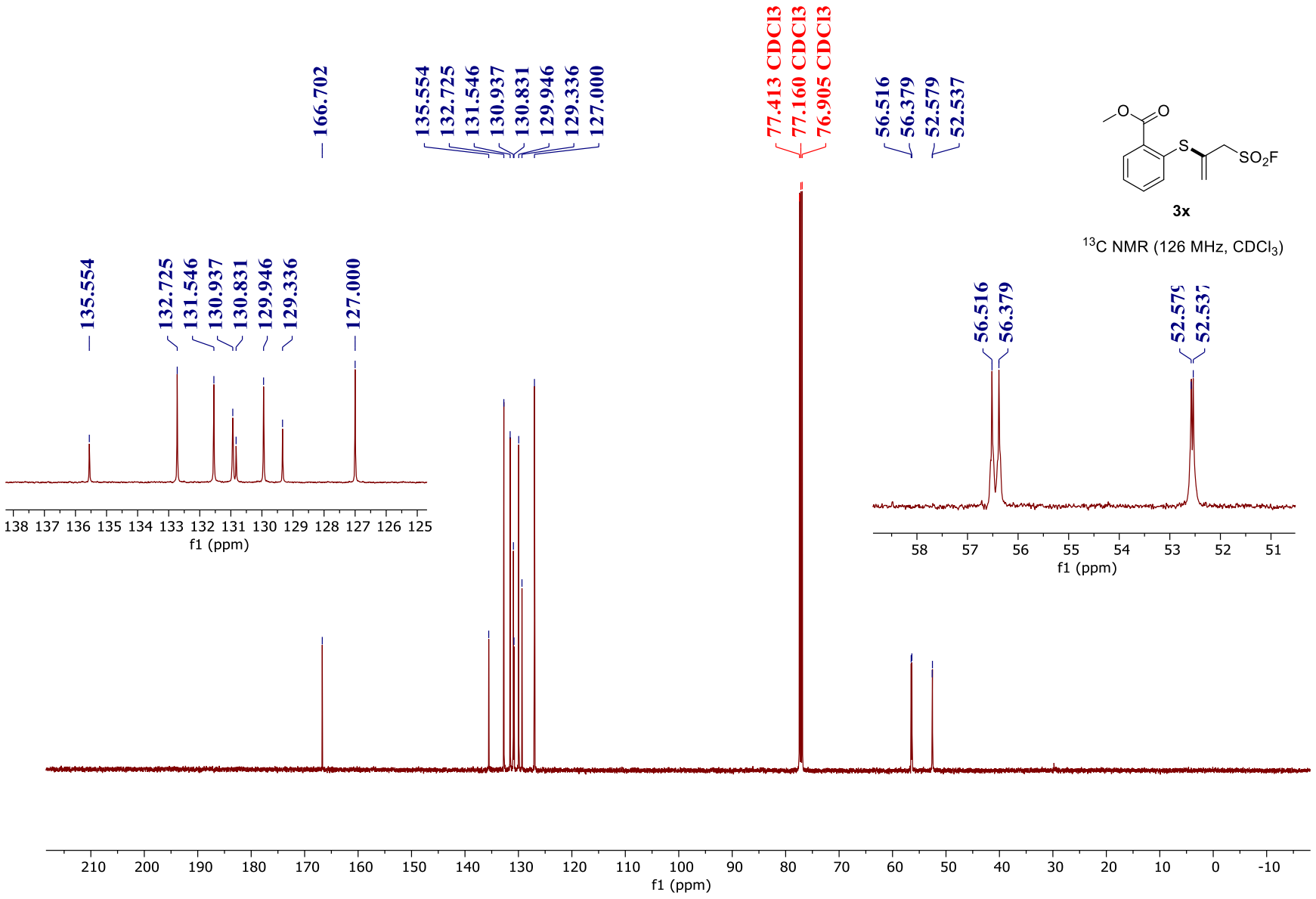

138.827
133.152
131.426
129.869
128.575
118.174
111.904
77.413 CDCl3
77.160 CDCl3
76.905 CDCl3
56.764
56.622
S
SO2F
N
3w
13C NMR (126 MHz, CDCl3)
56.764
56.622
59 58 57 56 55 54
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

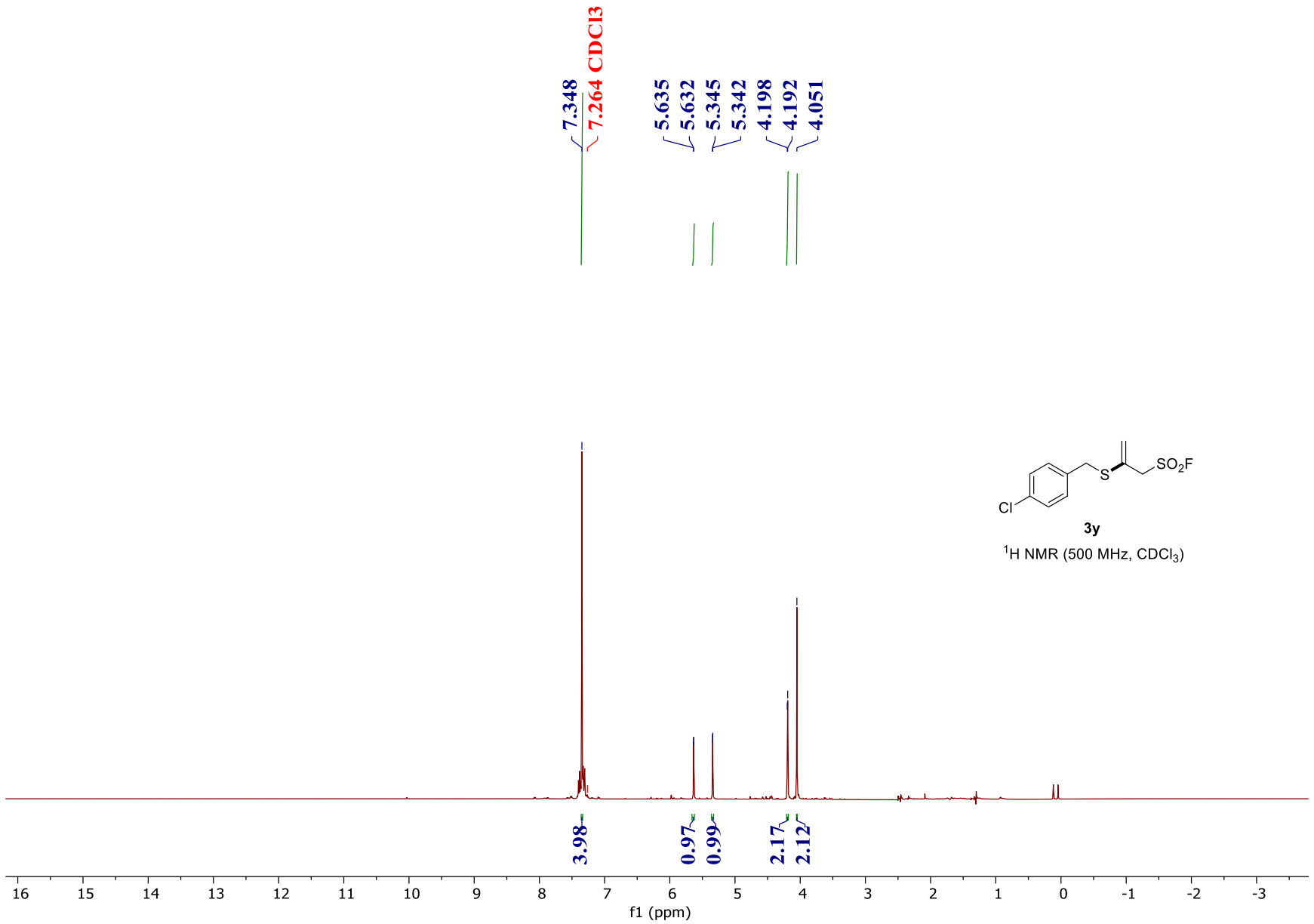

7.906
7.890
7.450
7.435
7.419
7.296
7.280
7.264 CDCl3
6.025
5.930
4.147
4.141
3.873
1.00
1.08
1.97
0.98
0.98
1.98
2.88
f1 (ppm)
3x
1H NMR (500 MHz, CDCl3)

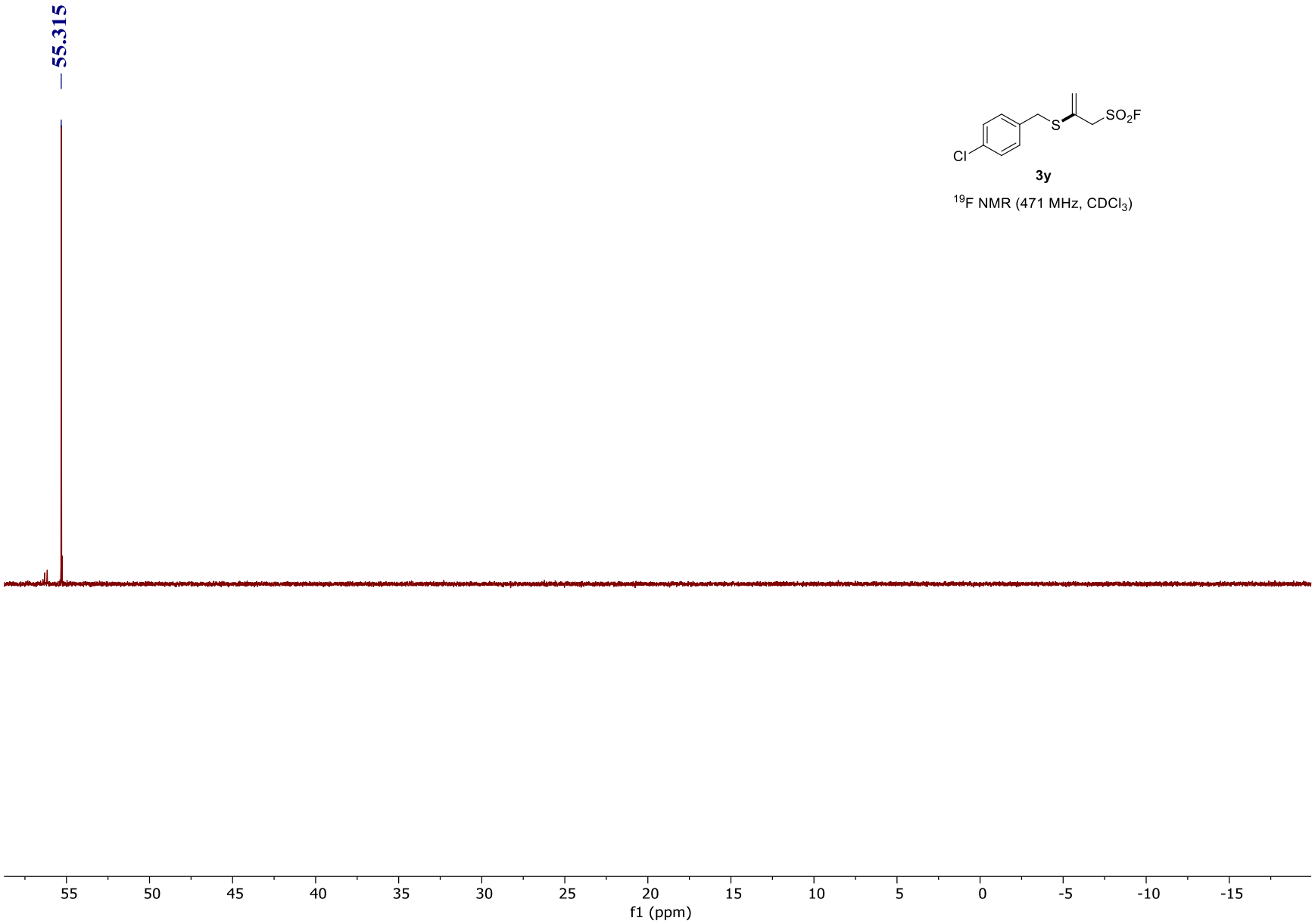


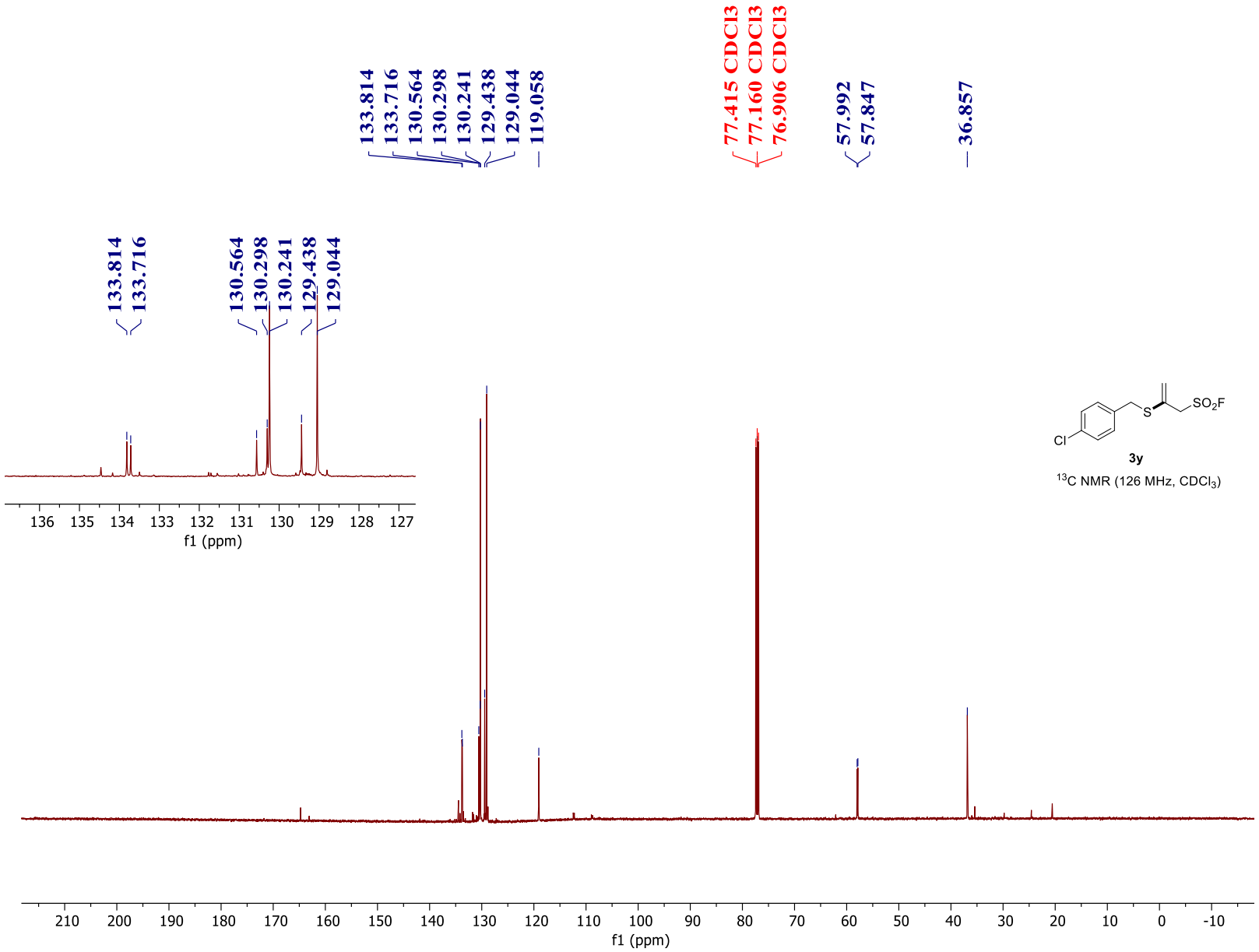
3x

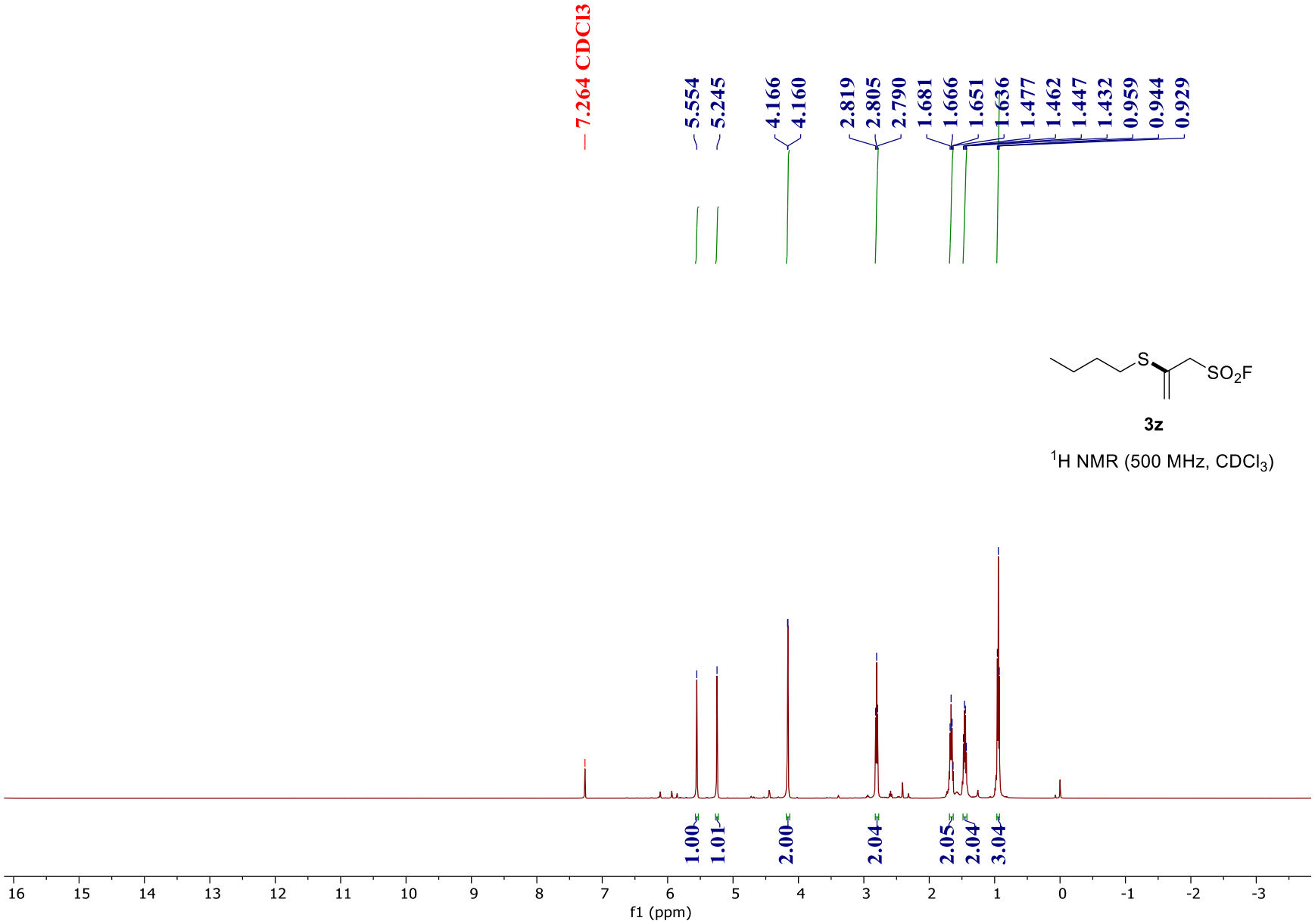
^{19}F NMR (471 MHz, $CDCl_3$)

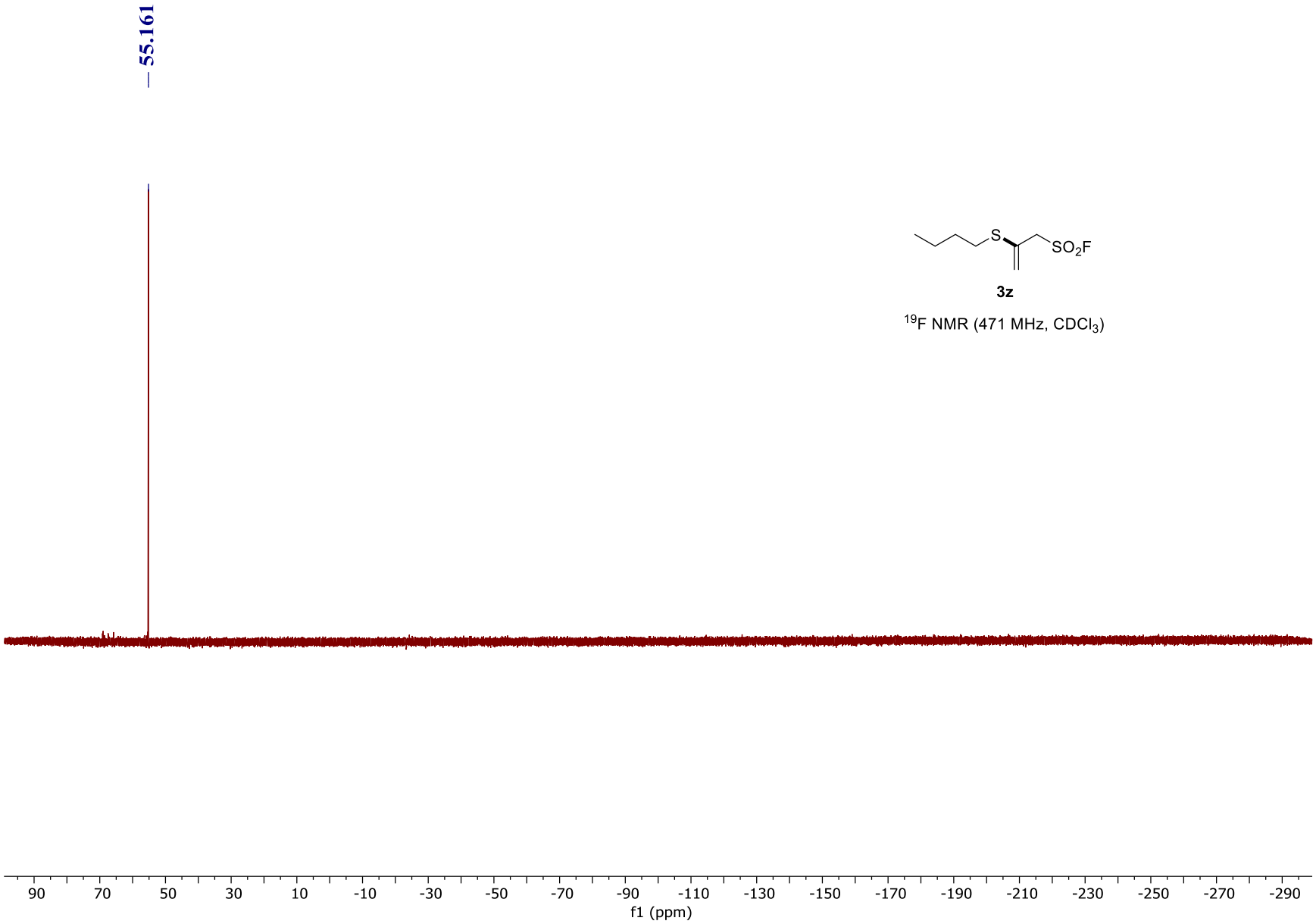

166.702
135.554
132.725
131.546
130.937
130.831
129.946
129.336
127.000
77.413 CDCl3
77.160 CDCl3
76.905 CDCl3
56.516
56.379
52.579
52.537
3x
13C NMR (126 MHz, CDCl3)
f1 (ppm)

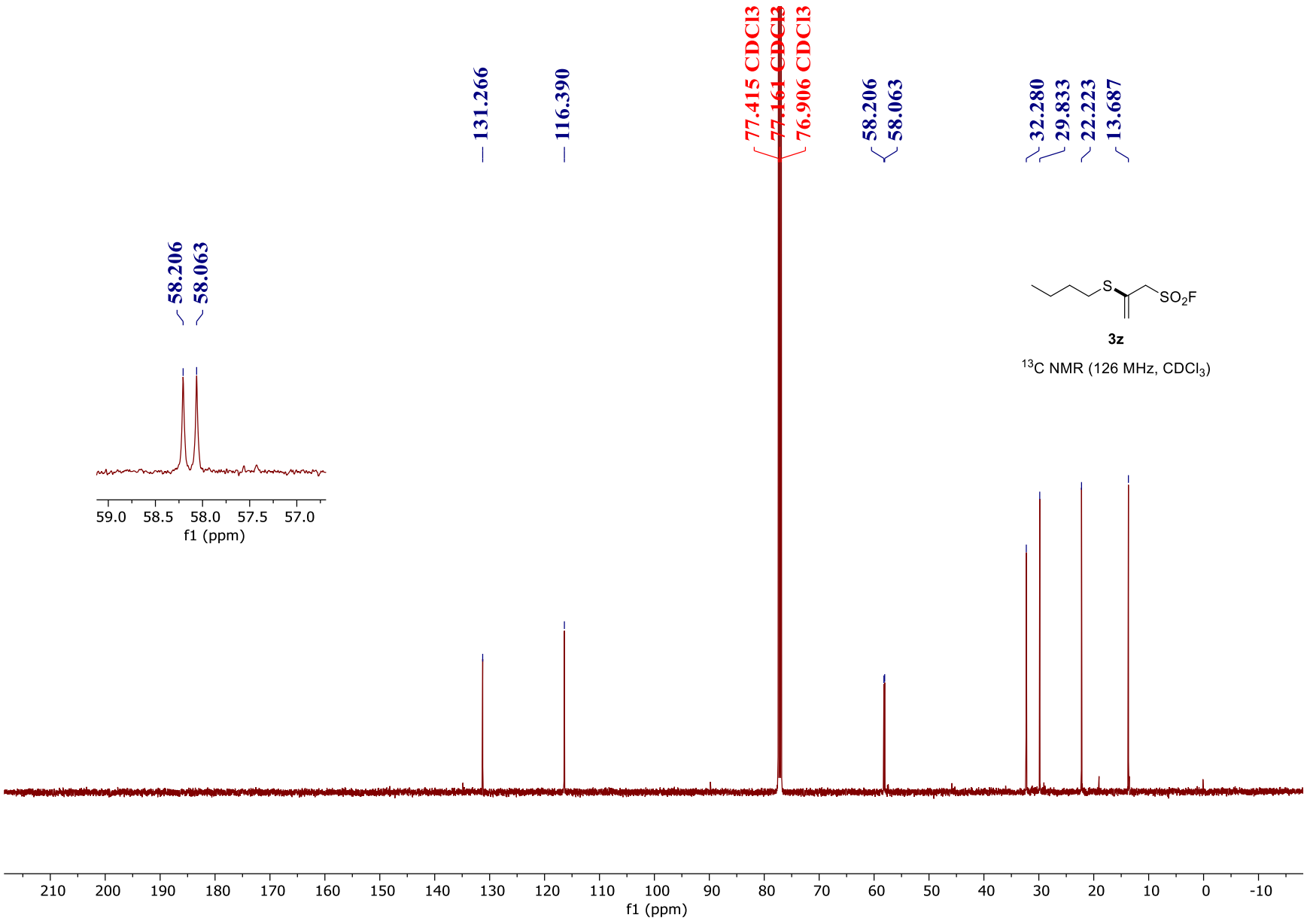

7.348
7.264 CDCl3
5.635
5.632
5.345
5.342
4.198
4.192
4.051
Cl
S
SO2F
3y
1H NMR (500 MHz, CDCl3)
3.98
0.97
0.99
2.17
2.12
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

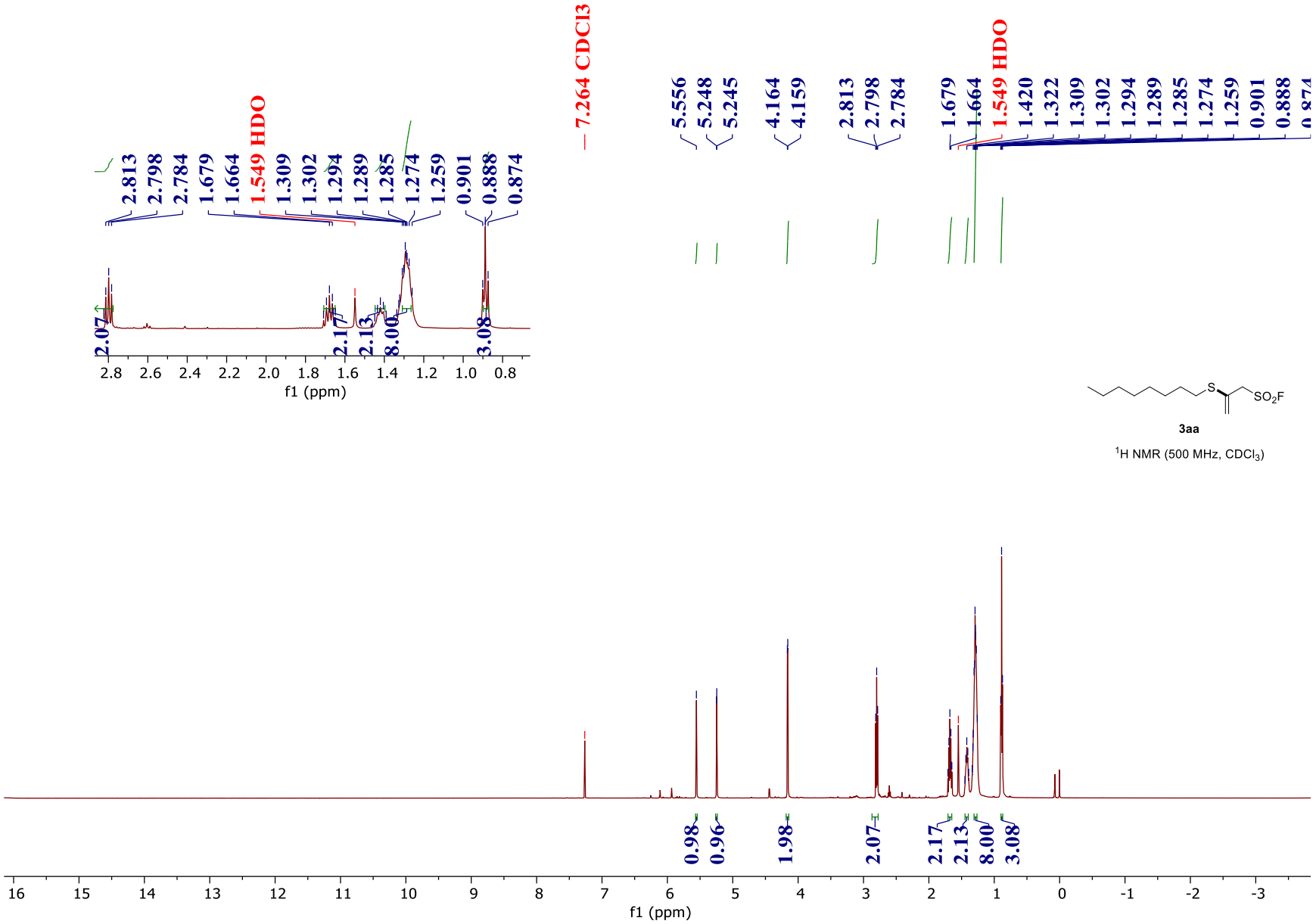

55.315
Cl
S
SO2F
3y
19F NMR (471 MHz, CDCl3)
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-15
f1 (ppm)

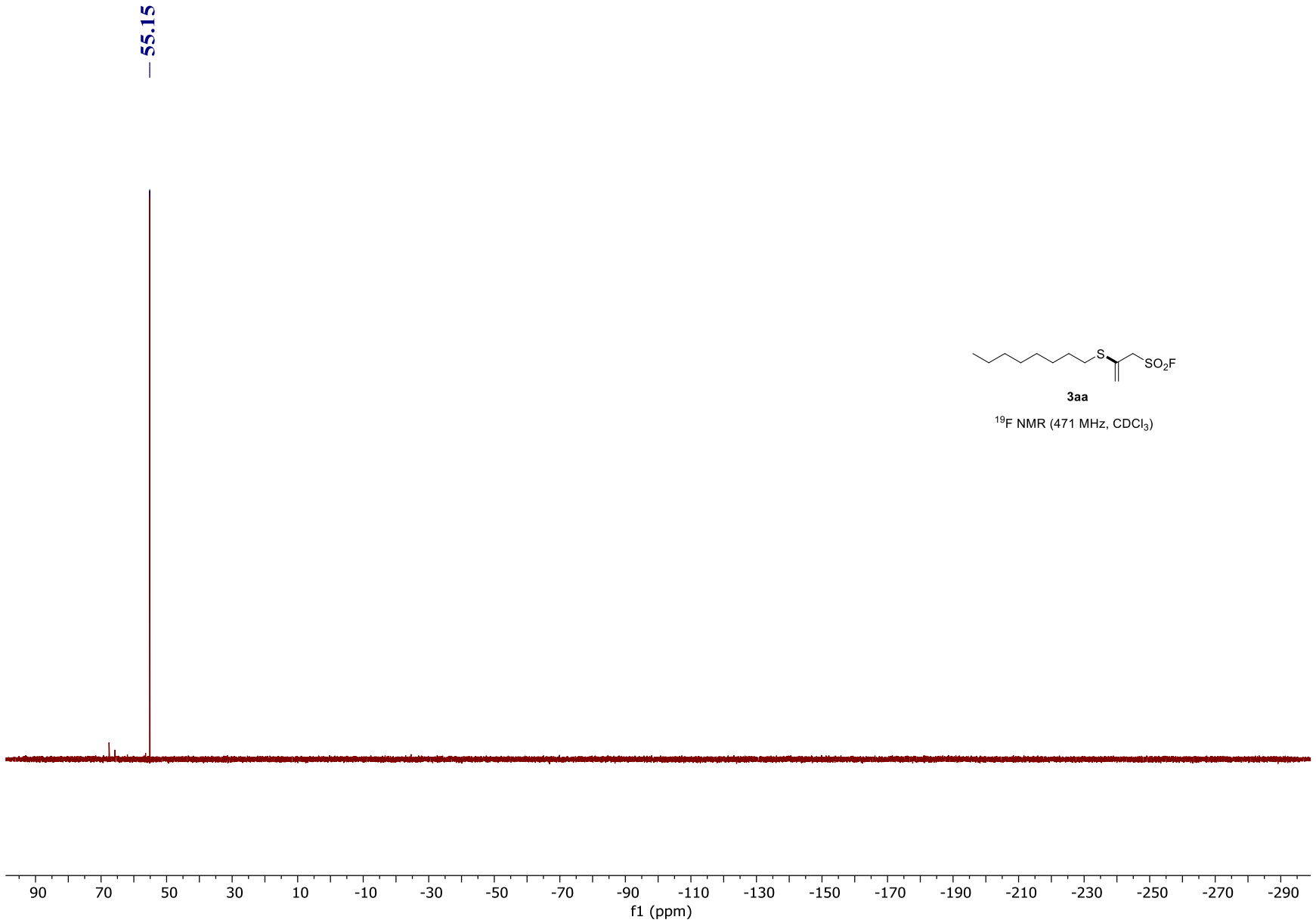

133.814
133.716
130.564
130.298
130.241
129.438
129.044
119.058
77.415 CDCl3
77.160 CDCl3
76.906 CDCl3
57.992
57.847
36.857
f1 (ppm)
Cl
S
SO2F
3y
13C NMR (126 MHz, CDCl3)


7.264 CDCl3
5.554
5.245
4.166
4.160
2.819
2.805
2.790
1.681
1.666
1.651
1.636
1.477
1.462
1.447
1.432
0.959
0.944
0.929
S
SO2F
3z
1H NMR (500 MHz, CDCl3)
1.00
1.01
2.00
2.04
2.05
2.04
3.04
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)


55.161
S
SO2F
3z
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

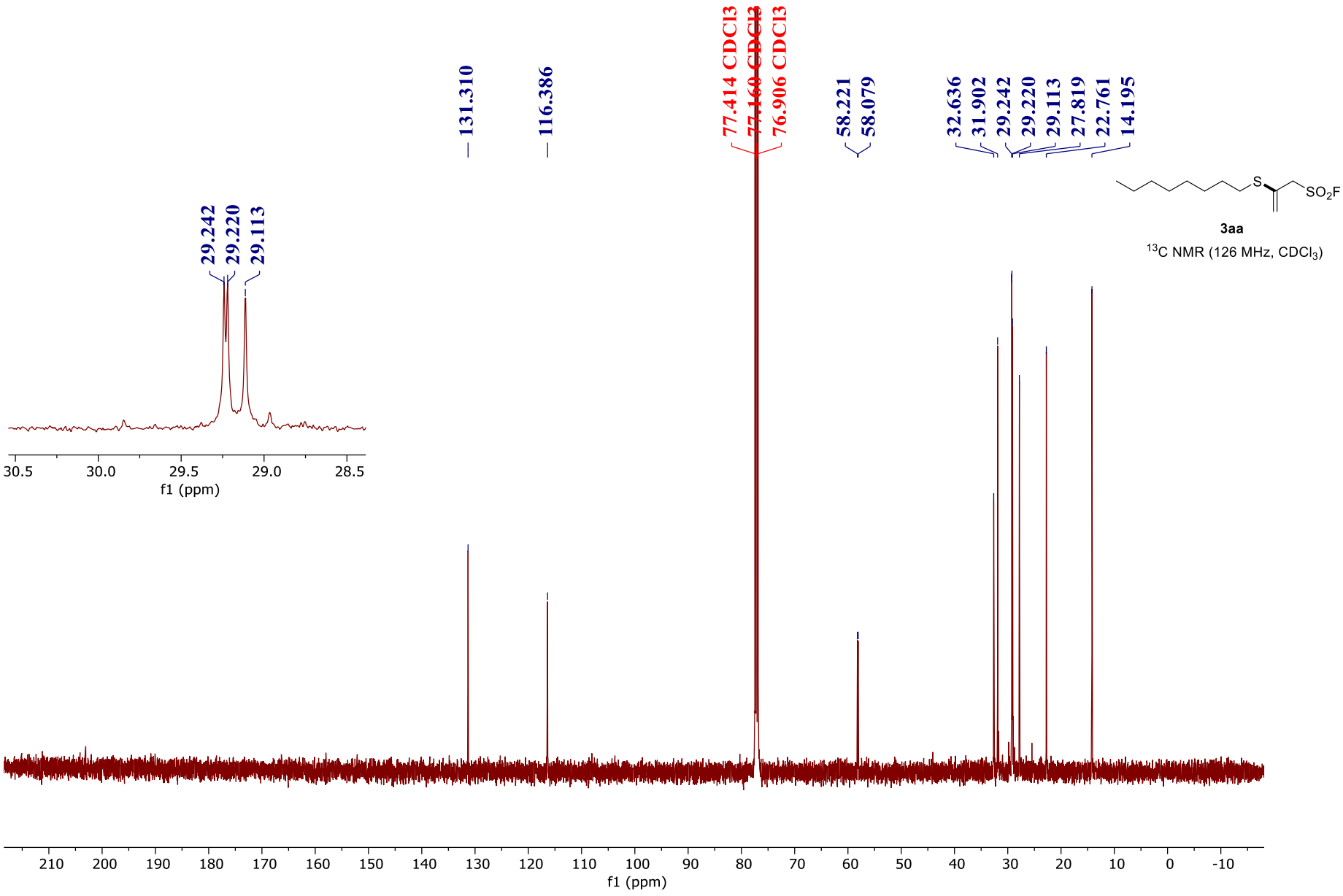

131.266
116.390
77.415 CDCl3
77.161 CDCl3
76.906 CDCl3
58.206
58.063
32.280
29.833
22.223
13.687
58.206
58.063
59.0 58.5 58.0 57.5 57.0
f1 (ppm)
S
SO2F
3z
13C NMR (126 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

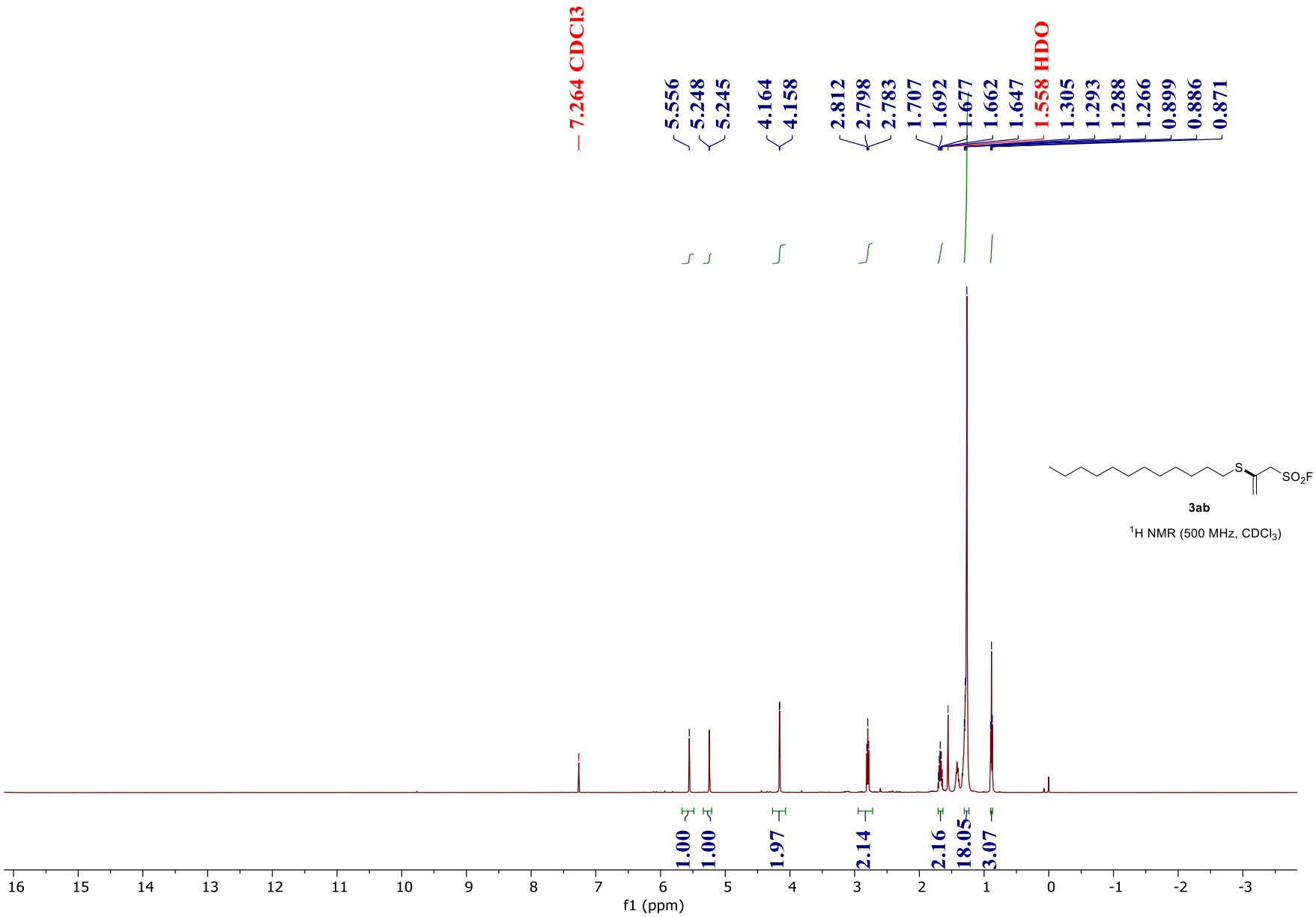

7.264 CDCl3
5.556
5.248
5.245
4.164
4.159
2.813
2.798
2.784
1.679
1.664
1.549 HDO
1.420
1.322
1.309
1.302
1.294
1.289
1.285
1.274
1.259
0.901
0.888
0.874
0.98
0.96
1.98
2.07
2.17
2.13
8.00
3.08
f1 (ppm)
S
SO2F
3aa
1H NMR (500 MHz, CDCl3)

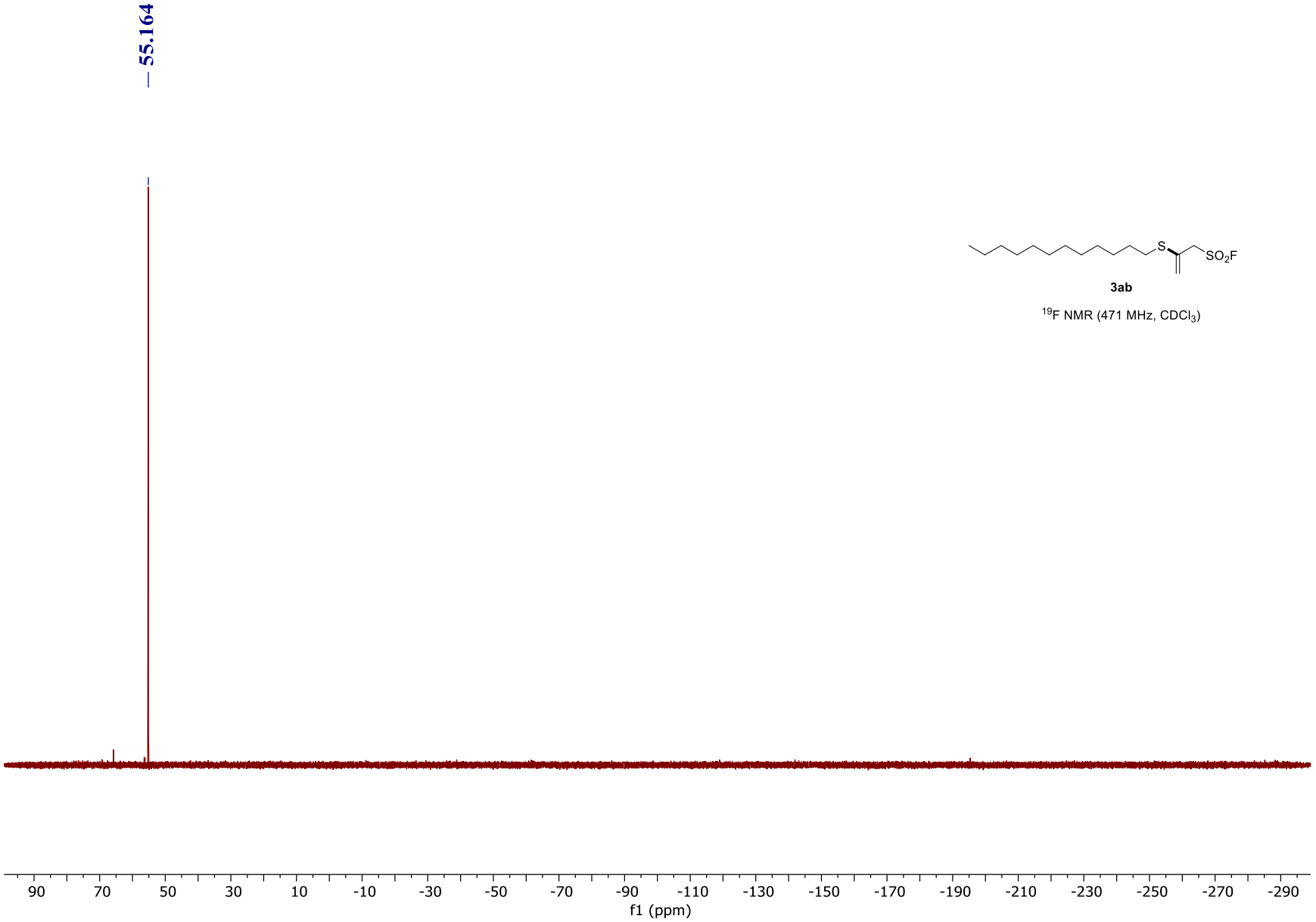


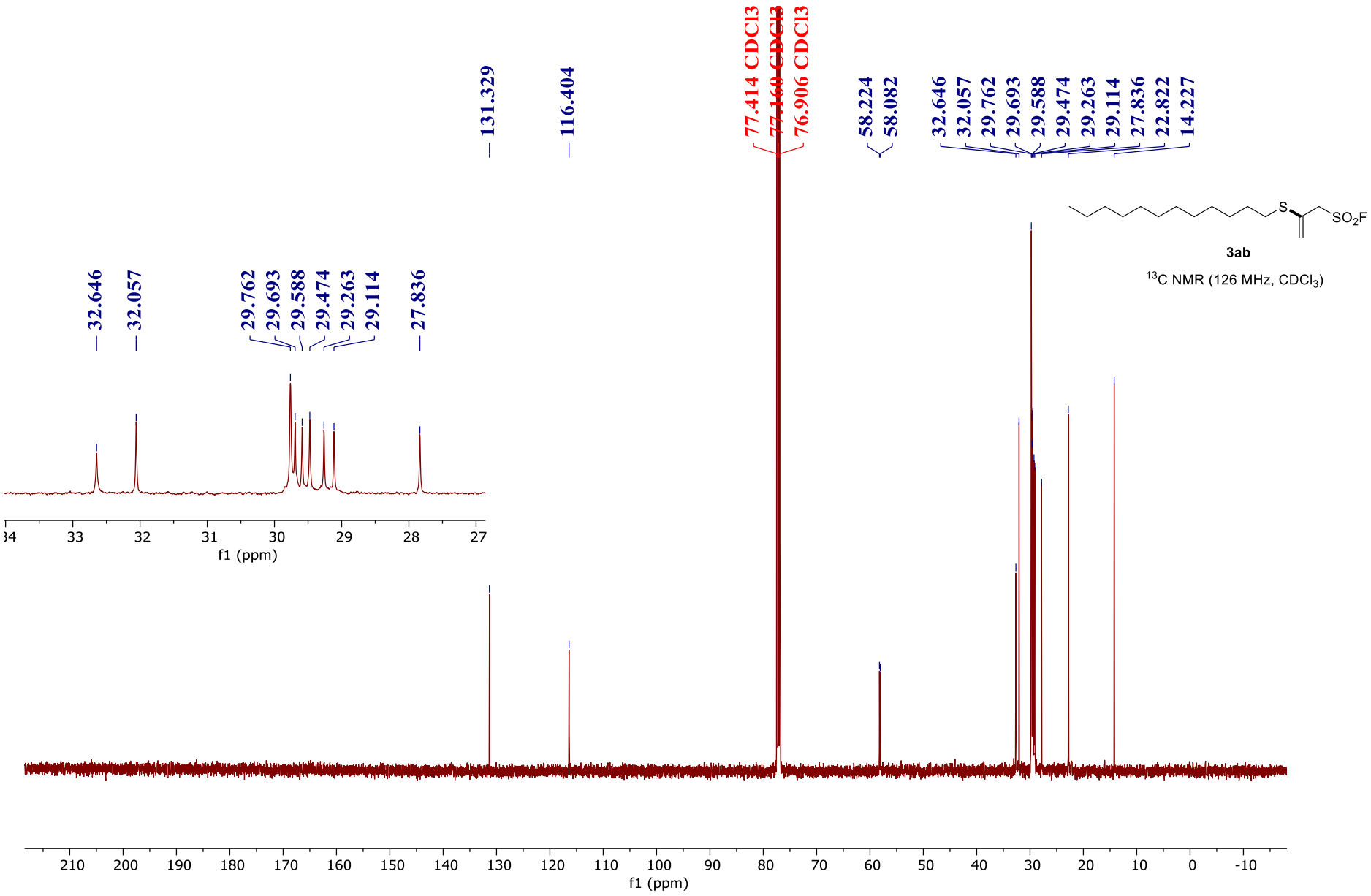
3aa

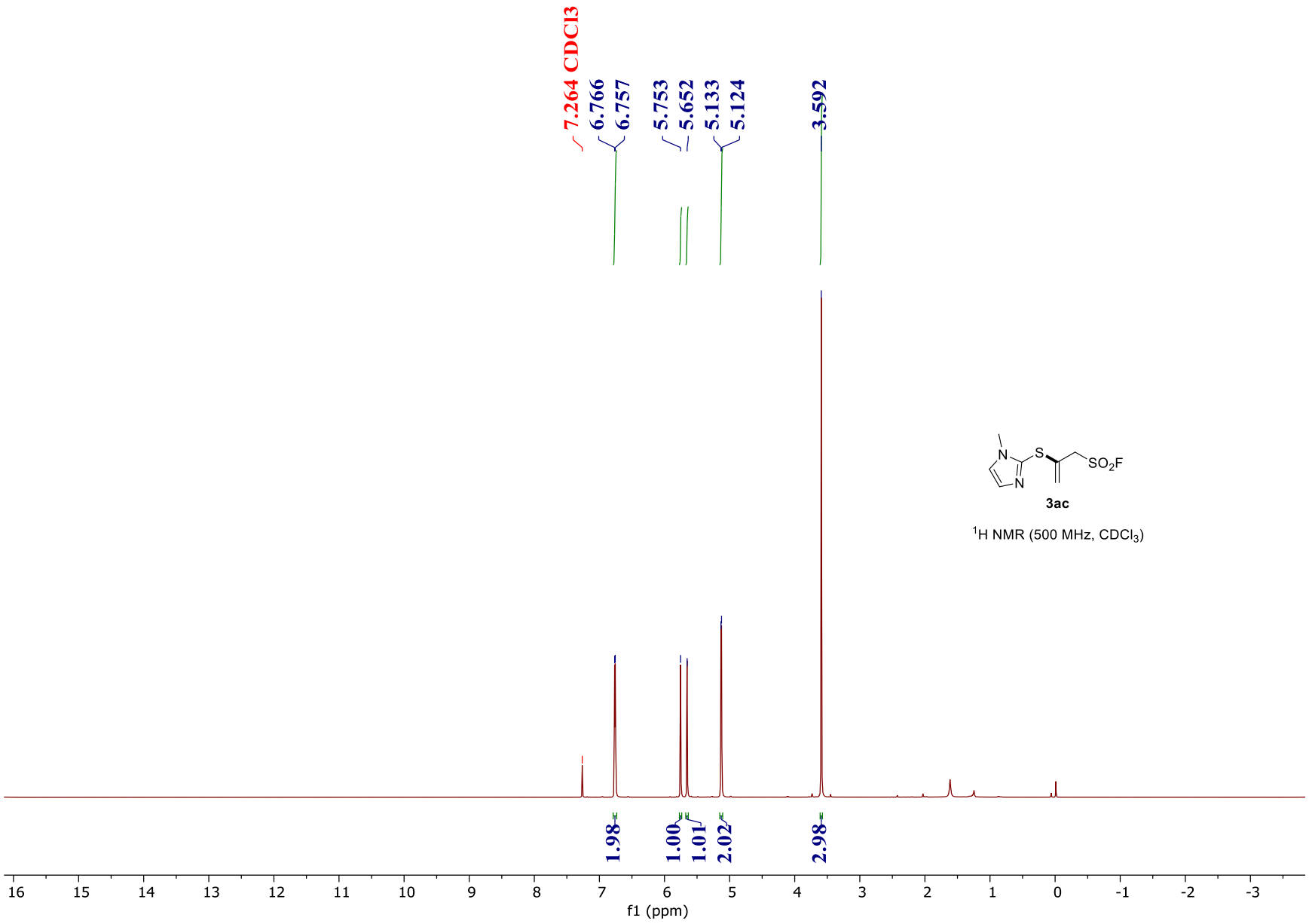
^{19}F NMR (471 MHz, $CDCl_3$)

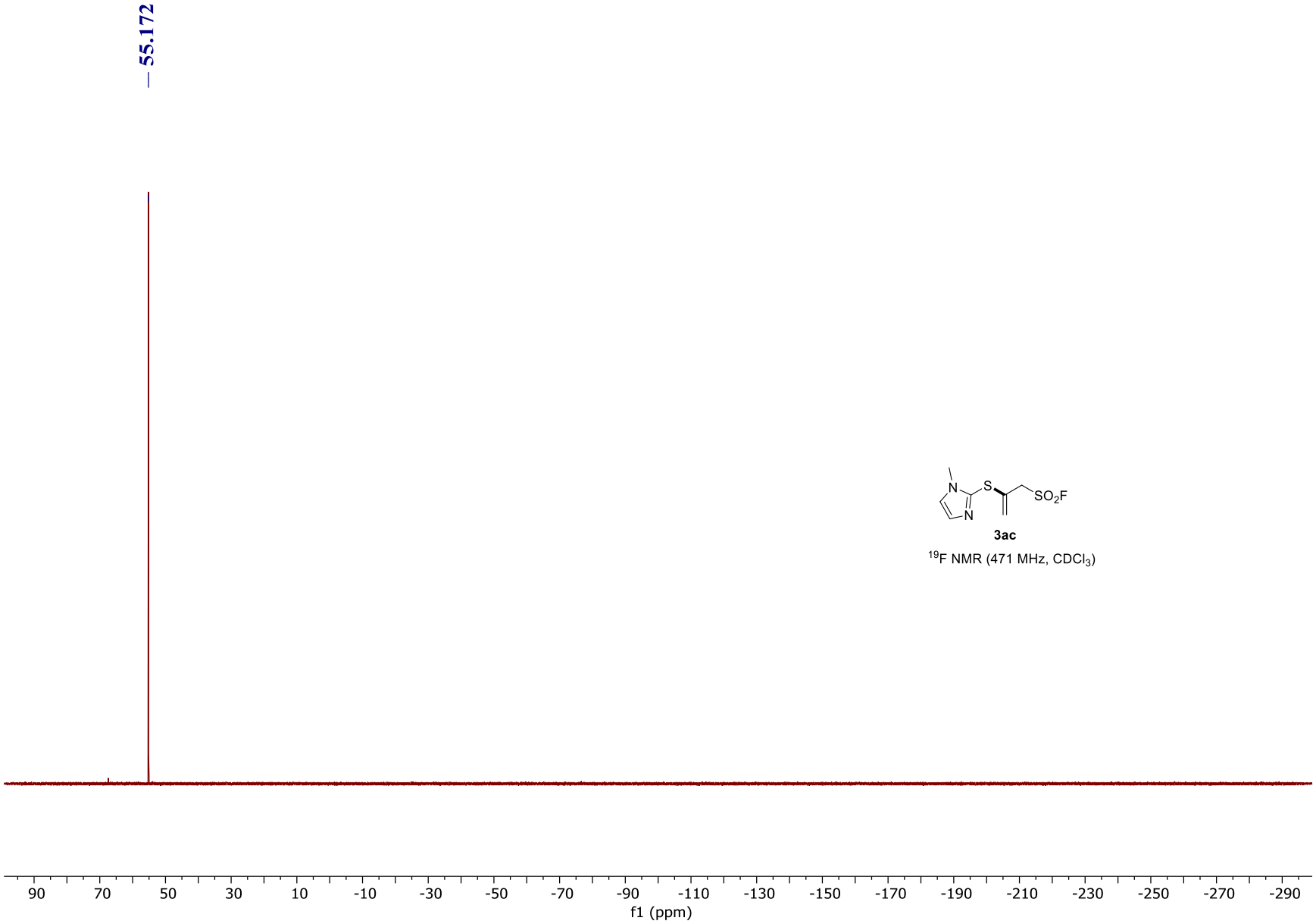

131.310
116.386
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
58.221
58.079
32.636
31.902
29.242
29.220
29.113
27.819
22.761
14.195
29.242
29.220
29.113
30.5
30.0
29.5
29.0
28.5
f1 (ppm)
S
SO2F
3aa
13C NMR (126 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.264 CDCl3
5.556
5.248
5.245
4.164
4.158
2.812
2.798
2.783
1.707
1.692
1.677
1.662
1.647
1.558 HDO
1.305
1.293
1.288
1.266
0.899
0.886
0.871
1.00
1.00
1.97
2.14
2.16
18.05
3.07
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)
S
SO2F
3ab
1H NMR (500 MHz, CDCl3)


55.164
S
SO2F
3ab
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

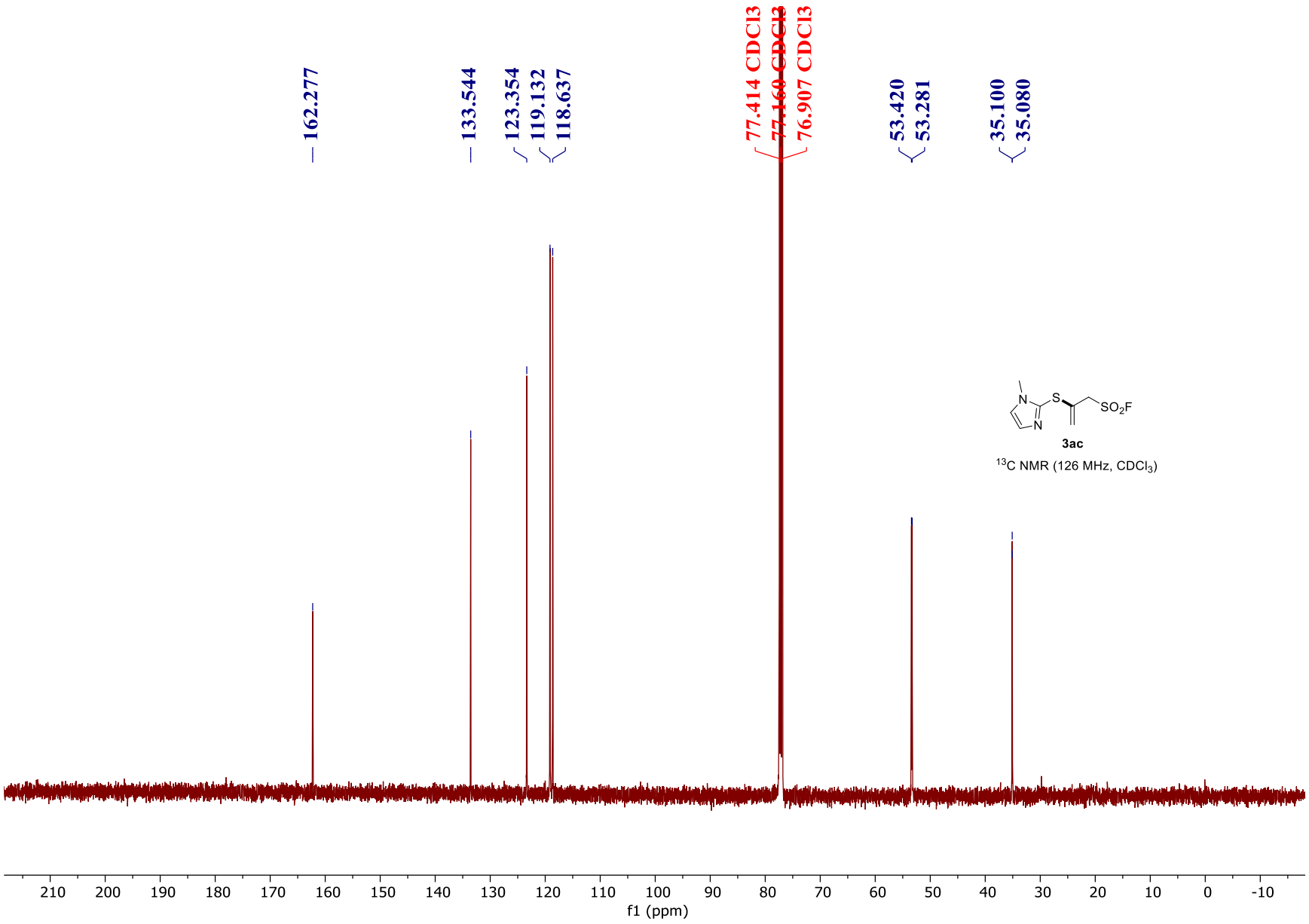

131.329
116.404
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
58.224
58.082
32.646
32.057
29.762
29.693
29.588
29.474
29.263
29.114
27.836
22.822
14.227
S
SO2F
3ab
13C NMR (126 MHz, CDCl3)
f1 (ppm)

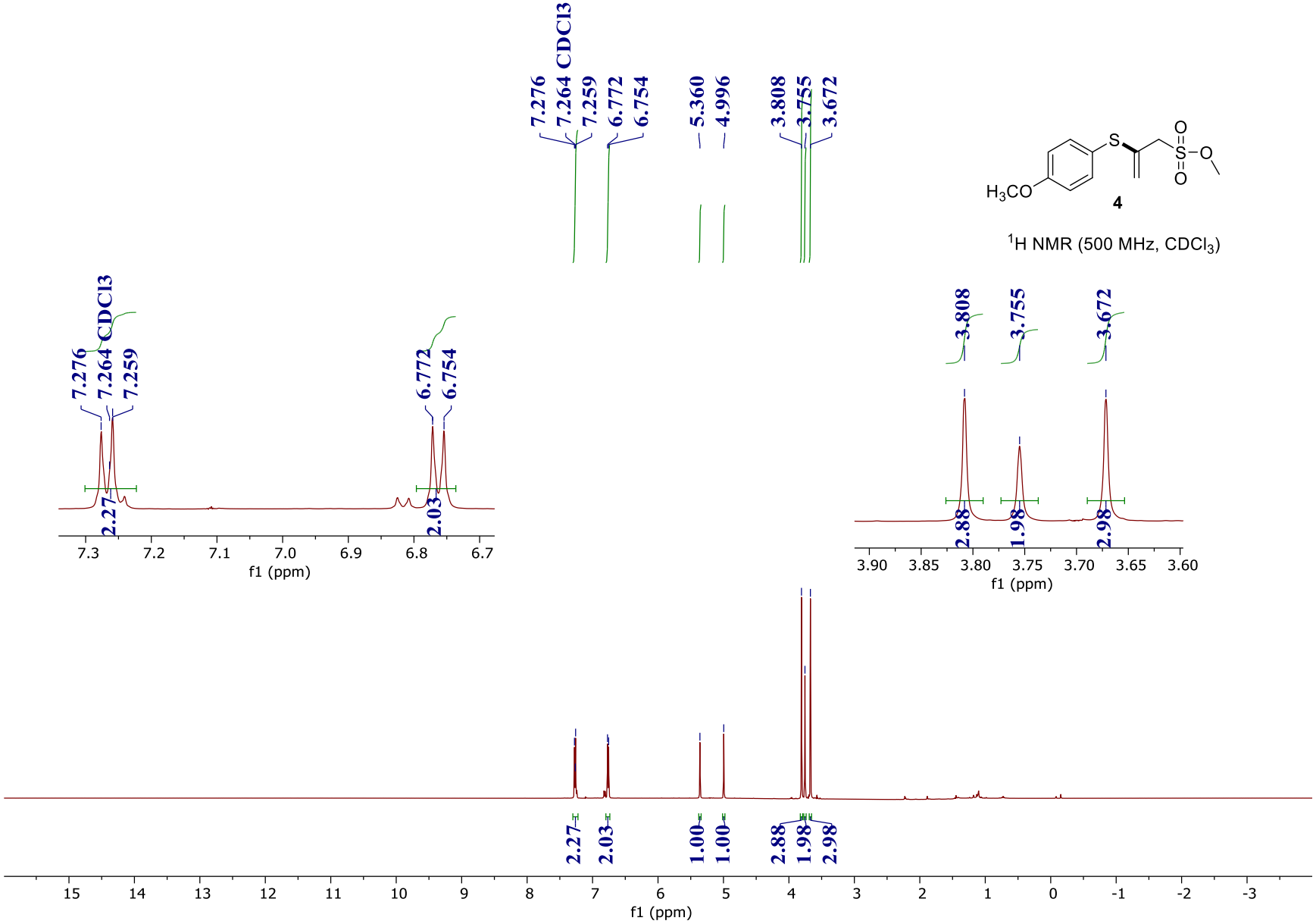

7.264 CDCl3
6.766
6.757
5.753
5.652
5.133
5.124
3.592
1.98
1.00
1.01
2.02
2.98
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)
N
S
N
SO2F
3ac
1H NMR (500 MHz, CDCl3)

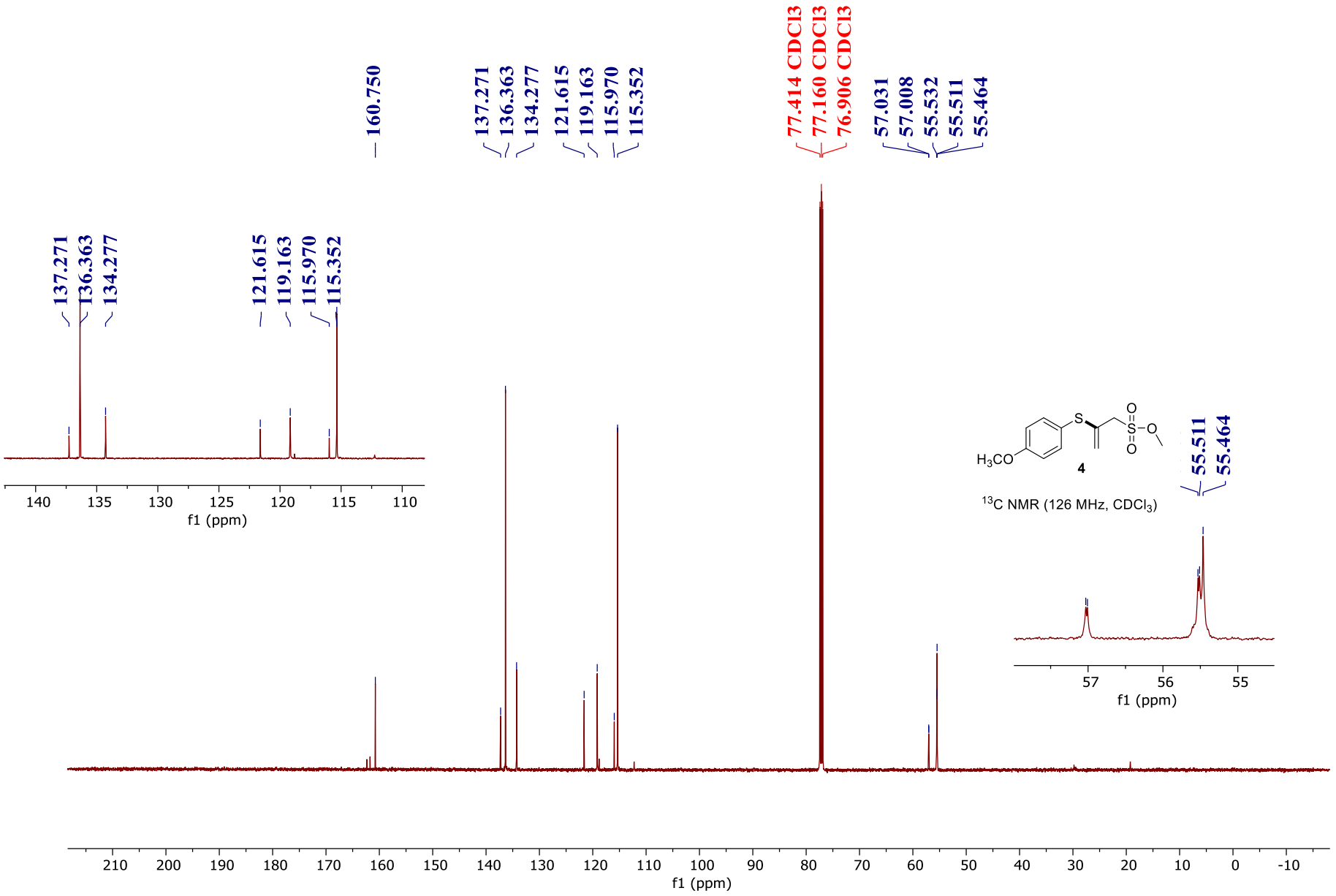


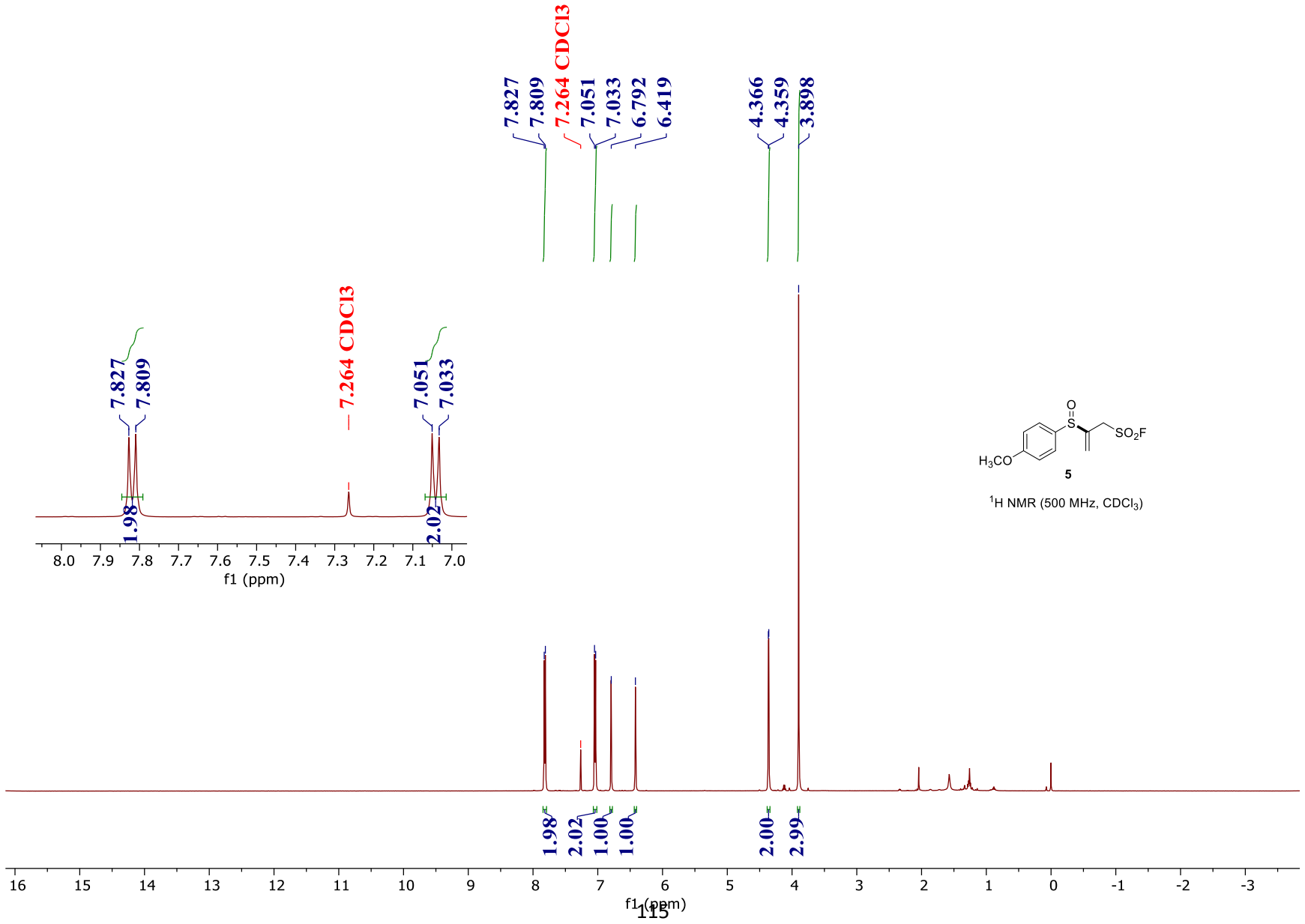
3ac

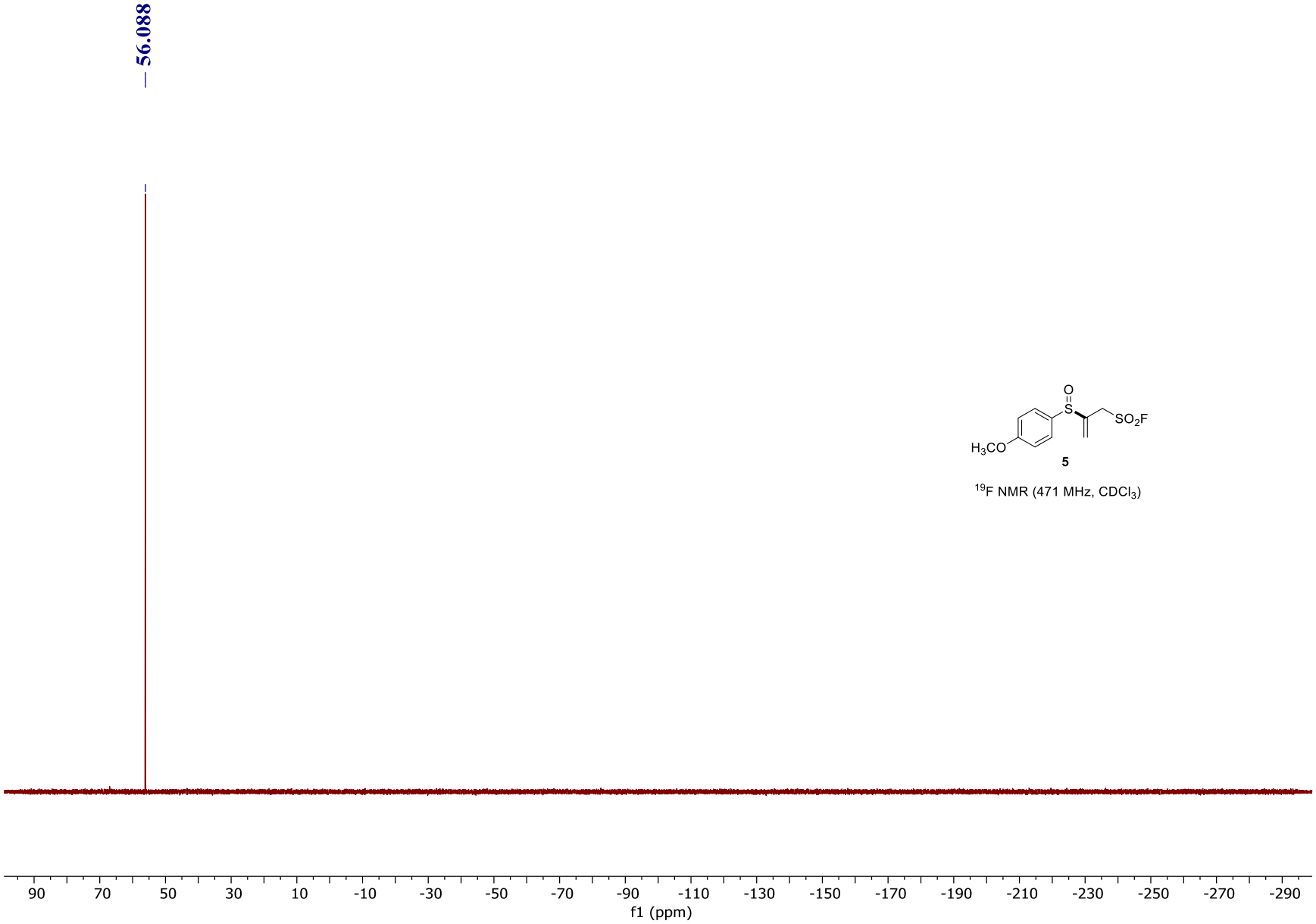
^{19}F NMR (471 MHz, $CDCl_3$)

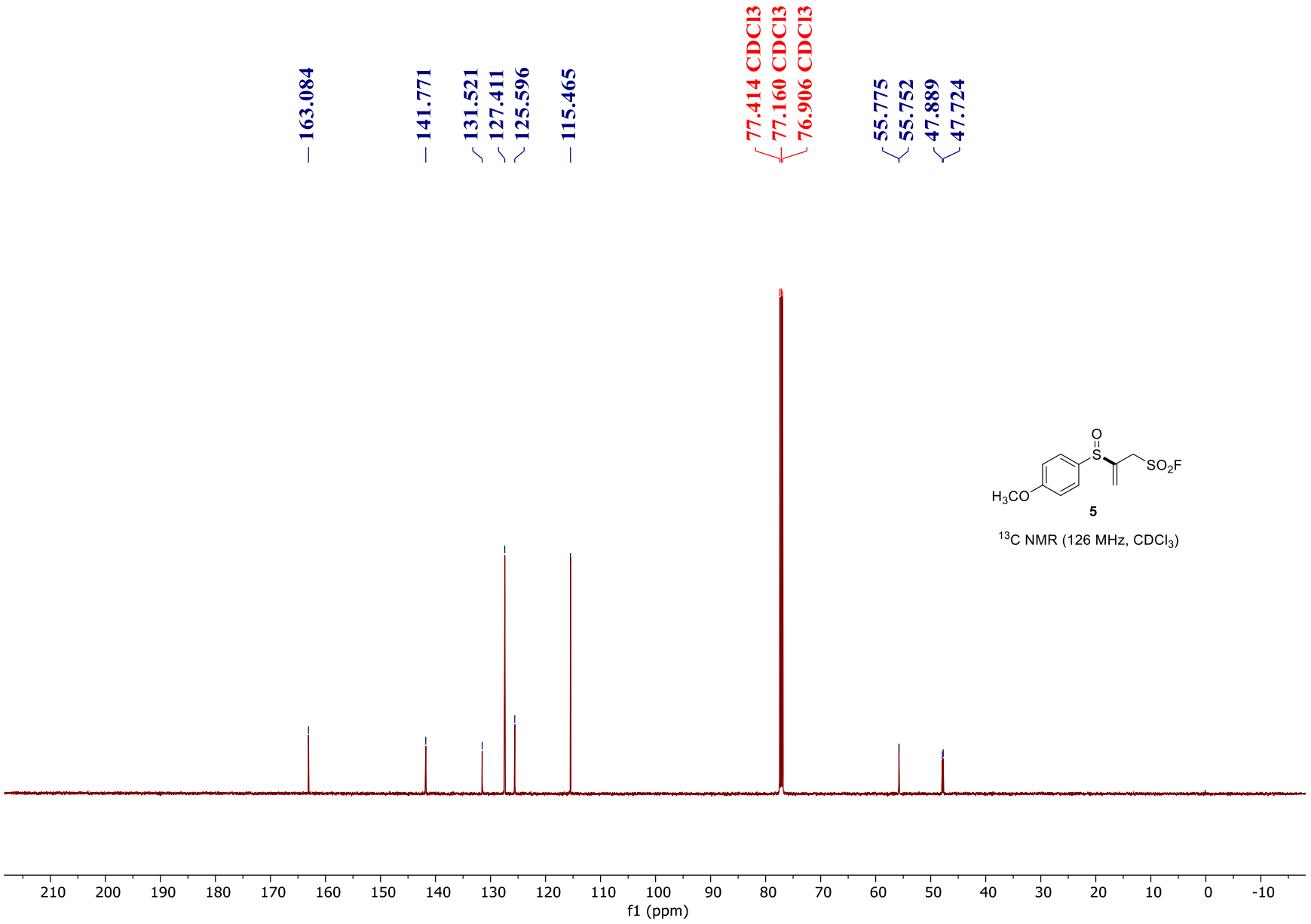

162.277
133.544
123.354
119.132
118.637
77.414 CDCl3
77.160 CDCl3
76.907 CDCl3
53.420
53.281
35.100
35.080
SO2F
3ac
13C NMR (126 MHz, CDCl3)
f1 (ppm)

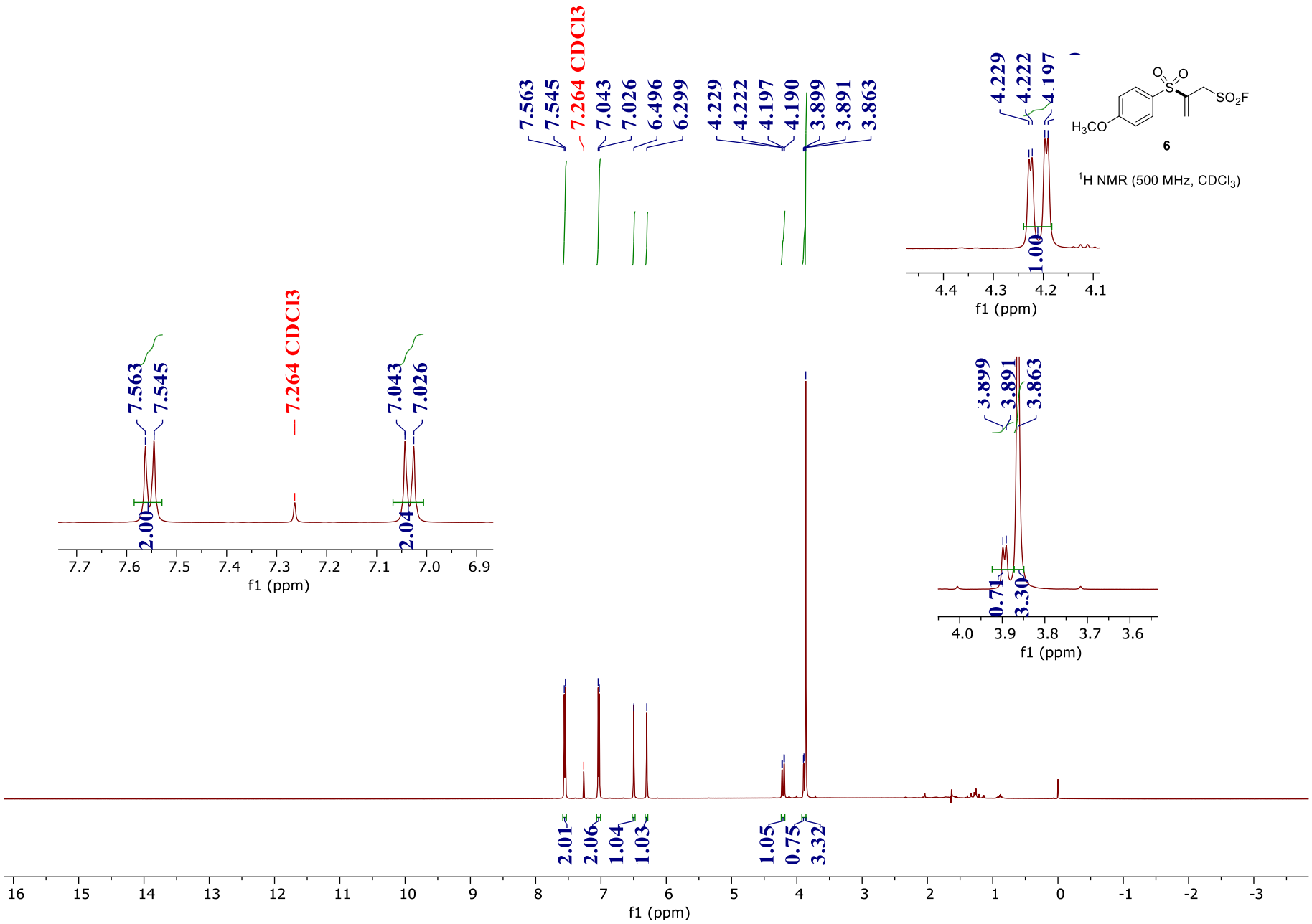

7.276
7.264 CDCl3
7.259
6.772
6.754
5.360
4.996
3.808
3.755
3.672
H3CO
S
O
S
O
O
4
1H NMR (500 MHz, CDCl3)
2.27
2.03
1.00
1.00
2.88
1.98
2.98
f1 (ppm)

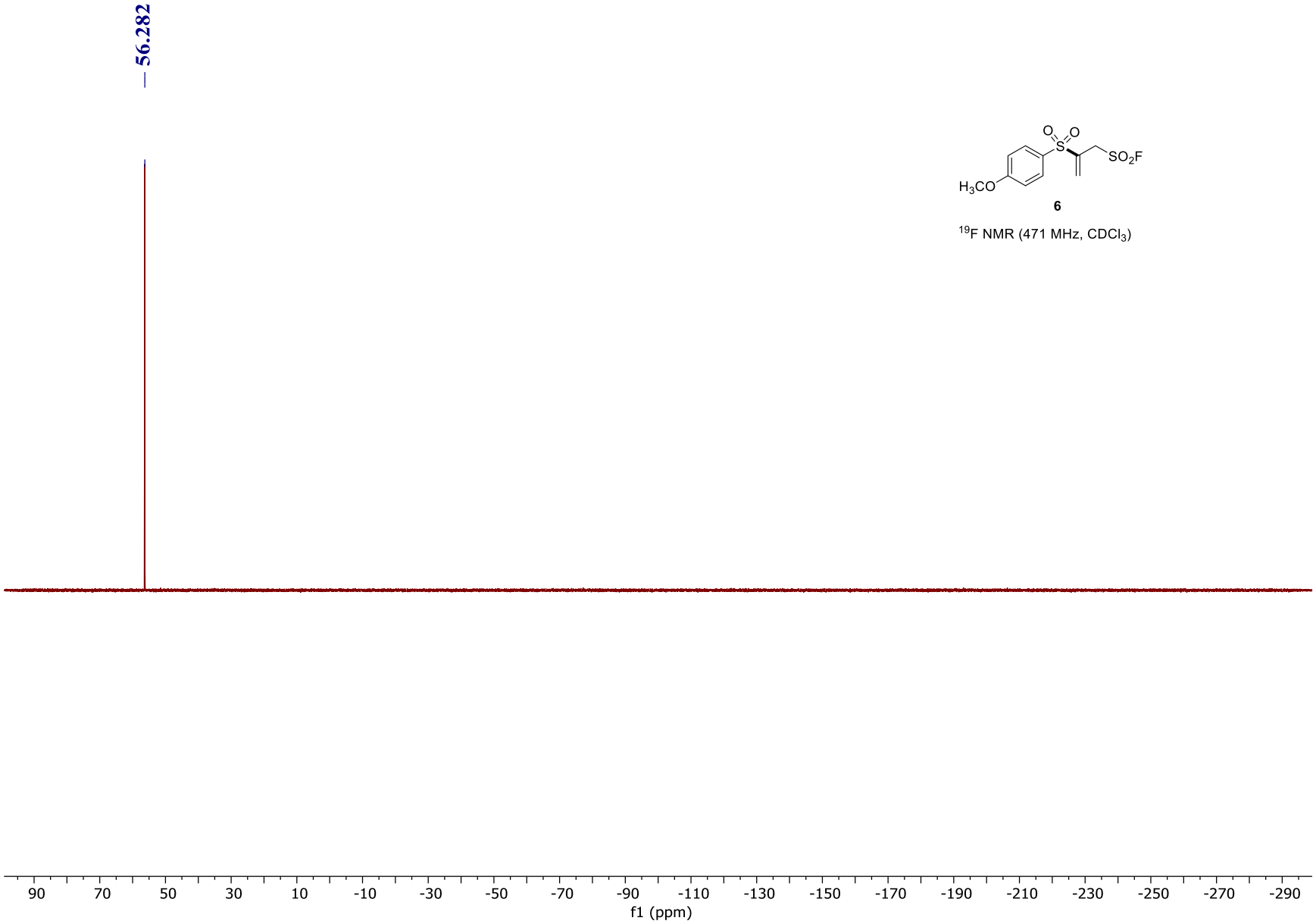

160.750
137.271
136.363
134.277
121.615
119.163
115.970
115.352
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
57.031
57.008
55.532
55.511
55.464
f1 (ppm)
4
13C NMR (126 MHz, CDCl3)


7.827
7.809
7.264 CDCl3
7.051
7.033
6.792
6.419
4.366
4.359
3.898
1.98
2.02
1.00
1.00
2.00
2.99
f1 (ppm)
7.827
7.809
7.264 CDCl3
7.051
7.033
1.98
2.02
f1 (ppm)
H3CO
SO2F
5
1H NMR (500 MHz, CDCl3)


56.088
O
S
SO2F
H3CO
5
19F NMR (471 MHz, CDCl3)
90
70
50
30
10
-10
-30
-50
-70
-90
-110
-130
-150
-170
-190
-210
-230
-250
-270
-290
f1 (ppm)

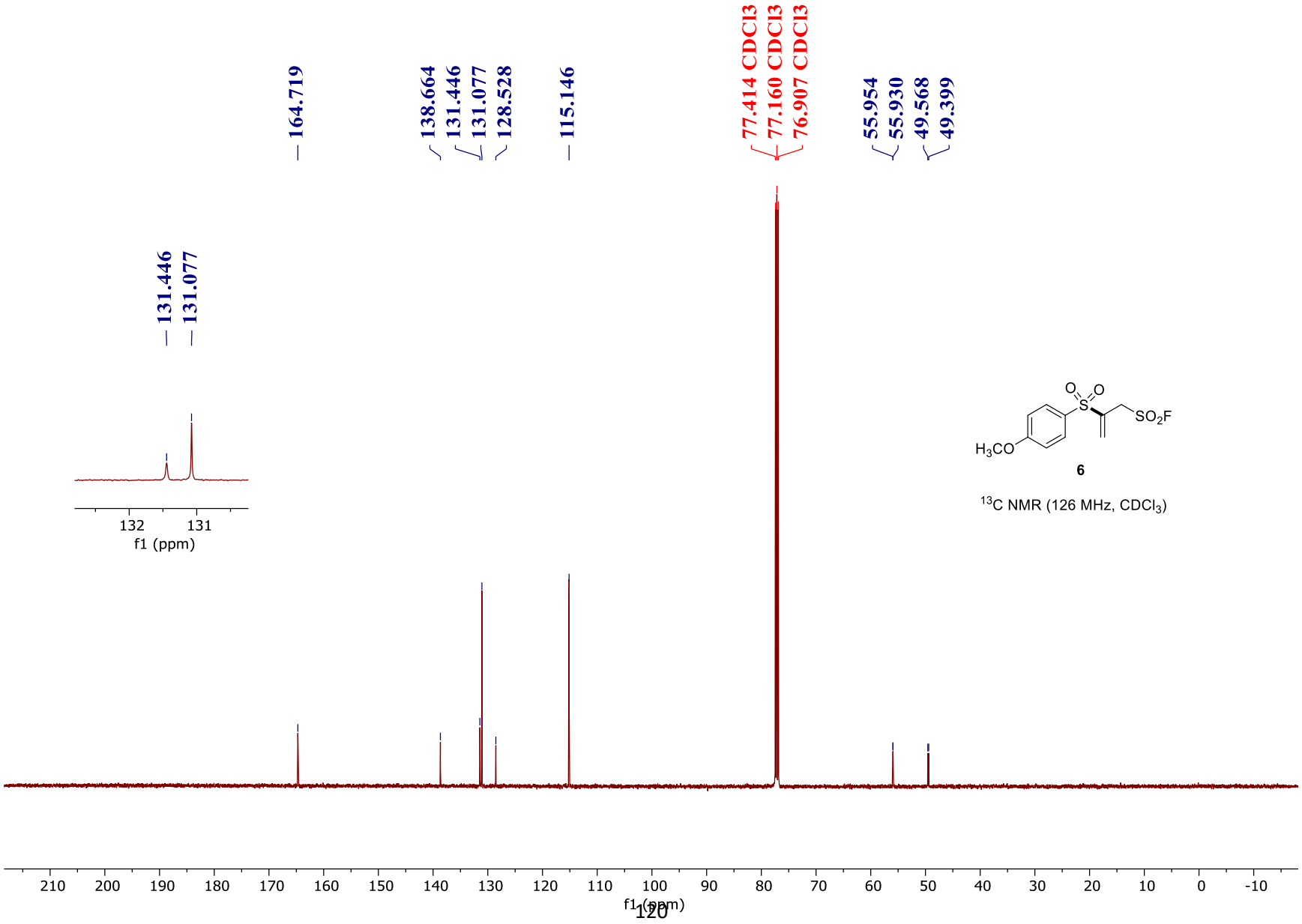

163.084
141.771
131.521
127.411
125.596
115.465
77.414 CDCl3
77.160 CDCl3
76.906 CDCl3
55.775
55.752
47.889
47.724
O
S
SO2F
H3CO
5
13C NMR (126 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.563
7.545
7.264 CDCl3
7.043
7.026
6.496
6.299
4.229
4.222
4.197
4.190
3.899
3.891
3.863
4.229
4.222
4.197
1.00
4.4 4.3 4.2 4.1
f1 (ppm)
O
O
S
SO2F
H3CO
6
1H NMR (500 MHz, CDCl3)
7.563
7.545
7.264 CDCl3
7.043
7.026
2.00
2.04
7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9
f1 (ppm)
3.899
3.891
3.863
0.71
3.30
4.0 3.9 3.8 3.7 3.6
f1 (ppm)
2.01
2.06
1.04
1.03
1.05
0.75
3.32
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)



6

^{19}F NMR (471 MHz, $CDCl_3$)


164.719
138.664
131.446
131.077
128.528
115.146
77.414 CDCl3
77.160 CDCl3
76.907 CDCl3
55.954
55.930
49.568
49.399
131.446
131.077
132
131
f1 (ppm)
O
O
S
SO2F
H3CO
6
13C NMR (126 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)