

## Supporting Information

### A Simple Protocol for Stereoselective Construction of Novel $\beta$ -Sulfanyl Vinyl Sulfonyl Fluorides

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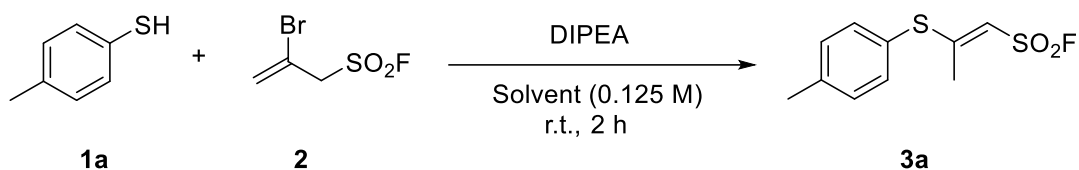
## 1. General Information

### Instrumentation:

The reactions were conducted under ambient air unless otherwise noted. NMR spectra were recorded in CDCl<sub>3</sub> or DMSO-*d*<sub>6</sub> using a spectrometer with frequencies of 500 MHz (for <sup>1</sup>H), 471 MHz (for <sup>19</sup>F), and 126 MHz (for <sup>13</sup>C). (note: CDCl<sub>3</sub>: δ H = 7.264 ppm, δ C = 77.16 ppm; DMSO-*d*<sub>6</sub>: δ H = 2.500 ppm, δ C = 39.52 ppm). Chemical shifts were reported in ppm relative to TMS (0 ppm for <sup>1</sup>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, br s = broad singlet, dd = doublet of doublets. The numbering of compounds for assigning NMR spectra is arbitrary and does not follow IUPAC conventions. Chemical structure names were generated using Chemdraw software. High-resolution mass spectrometry (HRMS) was conducted on a TOF-Q EI instrument. Melting points were measured and are uncorrected. All reagents used in the reactions were obtained from commercial sources and used without further purification. Thin layer chromatography (TLC) spots were visualized under UV light (254 nm or 365 nm) and subsequently stained with potassium permanganate or phosphomolybdic acid. The starting thiols were purchased and used without further modification.

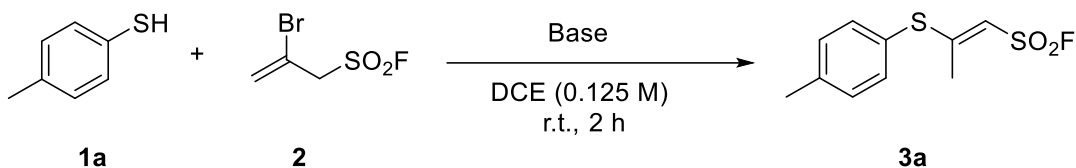
## 2. Optimization of the Reaction Conditions

**Table S1** Screening the Solvent.<sup>a</sup>



Entry	Solvent	Yield ( <b>3a</b> , %) <sup>b</sup>
1	DMSO	66
<b>2</b>	<b>DCE</b>	<b>98</b>
3	DCM	95
4	Toluene	N.D.
5	MeCN	91
6	THF	91
7	1,4-Dioxane	68
8	DMF	16
9	C <sub>2</sub> H <sub>5</sub> OH	58
10	NMP	N.D.
11	EtOAc	91
12	Acetone	69

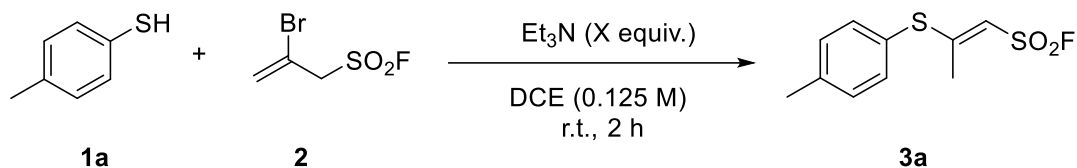
<sup>a</sup>Reaction conditions: a mixture of 4-methylbenzenethiol (**1a**, 25 mg, 0.2 mmol, 1.0 equiv.), BPESF (**2**, 61 mg, 0.3 mmol, 1.5 equiv.) and DIPEA (65 mg, 0.5 mmol, 2.5 equiv.) in solvent (1.6 mL) was stirred at r.t. for 2 h under air. <sup>b</sup>The yield was determined by HPLC using pure **3a** as the external standard ( $t_R = 5.0$  min,  $\lambda_{max} = 219.3$  nm, water/methanol = 30: 70 (v/v). N.D. = Not detectable.

**Table S2** Screening the base.<sup>a</sup>

Entry	Base (2.5 equiv.)	Yield ( <b>3a</b> , %) <sup>b</sup>
1	DIPEA	98
2	DABCO	75
3	DBU	77
4	CS <sub>2</sub> CO <sub>3</sub>	6
5	TMEDA	84
<b>6</b>	<b>Et<sub>3</sub>N</b>	<b>99</b>
7	NaHCO <sub>3</sub>	N.D.
8	K <sub>2</sub> CO <sub>3</sub>	N.D.
9	Tripopylamine	89
10	Na <sub>2</sub> CO <sub>3</sub>	2

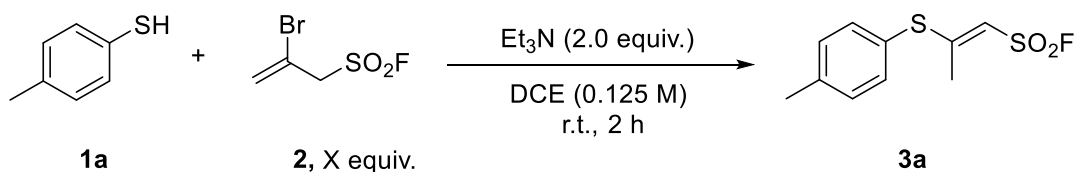
<sup>a</sup>Reaction conditions: a mixture of 4-methylbenzenethiol (**1a**, 25 mg, 0.2 mmol, 1.0 equiv.), BPESF (**2**, 61 mg, 0.3 mmol, 1.5 equiv.) and base (0.5 mmol, 2.5 equiv.) in DCE (1.6 mL) was stirred at r.t. for 2 h under air. <sup>b</sup>The yield was determined by HPLC using pure **3a** as the external standard ( $t_R = 5.0$  min,  $\lambda_{max} = 219.3$  nm, water/methanol = 30: 70 (v/v)). N.D. = Not detectable.



**Table S3** Screening the loading of base.<sup>a</sup>

Entry	$\text{Et}_3\text{N}$ (x equiv.)	Yield ( <b>3a</b> , %) <sup>b</sup>
1	1.0	42
2	1.5	56
<b>3</b>	<b>2.0</b>	<b>99</b>
4	2.5	99

<sup>a</sup>Reaction conditions: a mixture of 4-methylbenzenethiol (**1a**, 25 mg, 0.2 mmol, 1.0 equiv.), BPESF (**2**, 61 mg, 0.3 mmol, 1.5 equiv.) and  $\text{Et}_3\text{N}$  (X equiv.) in DCE (1.6 mL) was stirred at r.t. for 2 h under air. <sup>b</sup>The yield was determined by HPLC using pure **3a** as the external standard ( $t_{\text{R}} = 5.0$  min,  $\lambda_{\text{max}} = 219.3$  nm, water/methanol = 30: 70 (v/v)).

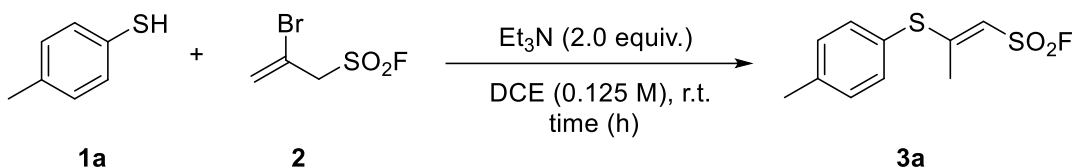
**Table S4** Screening the loading of BPESF (**2**)<sup>a</sup>

Entry	X equiv.	Yield ( <b>3a</b> , %) <sup>b</sup>
1	1.0	93
<b>2</b>	<b>1.2</b>	<b>98</b>
3	1.5	99
4	2.0	89

<sup>a</sup>Reaction conditions: a mixture of 4-methylbenzenethiol (**1a**, 25 mg, 0.2 mmol, 1.0 equiv.), BPESF (**2**, X equiv.) and  $\text{Et}_3\text{N}$  (40 mg, 0.4 mmol, 2.0 equiv.) in DCE (1.6 mL) was stirred at r.t. for 2 h under

air. <sup>b</sup>The yield was determined by HPLC using pure **3a** as the external standard ( $t_R = 5.0$  min,  $\lambda_{max} = 219.3$  nm, water/methanol = 30: 70 (v/v)).

**Table S5** Screening the time of reaction<sup>a</sup>

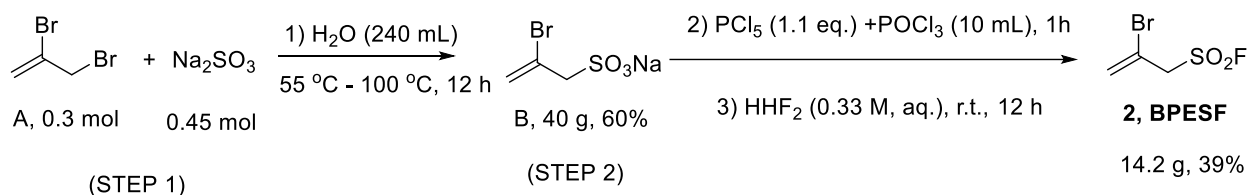


Entry	Time (h)	Yield ( <b>3a</b> , %) <sup>b</sup>
1	0.5	90
2	1.0	87
<b>3</b>	<b>2.0</b>	<b>98</b>

<sup>a</sup>Reaction conditions: a mixture of 4-methylbenzenethiol (**1a**, 25 mg, 0.2 mmol, 1.0 equiv.), BPESF (**2**, 49 mg, 0.24 mmol, 1.2 equiv.) and Et<sub>3</sub>N (40 mg, 0.4 mmol, 2.0 equiv.) in DCE (1.6 mL) was stirred at r.t. for X h under air. <sup>b</sup>The yield was determined by HPLC using pure **3a** as the external standard ( $t_R = 5.0$  min,  $\lambda_{max} = 219.3$  nm, water/methanol = 30: 70 (v/v)).

### 3. Experimental Procedures

#### 3.1 Preparation of 2-chloroprop-2-ene-1-sulfonyl fluoride (BPESF).<sup>1</sup>



**Step 1:** A mixture of 2,3-dibromopropene (60 g, 0.3 mol) and Na<sub>2</sub>SO<sub>3</sub> (37.8 g, 0.45 mol, 1.5 equiv.) was added to 240 mL of water and stirred while being heated. The mixture was then refluxed at 100 °C for 12 hours. After the reaction, the solvent was removed under reduced pressure. The resulting residue was dissolved in 300 mL of ethanol, and the mixture was refluxed with stirring at 80 °C for 1 hour. After removing insoluble materials by filtering the hot mixture, the filtrate

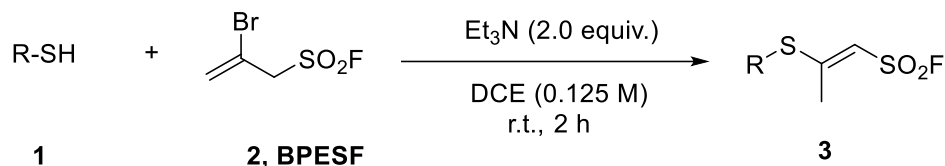
was cooled to -20 °C. The resulting crystalline form of sodium 2-bromoprop-2-ene-1-sulfonate were collected by filtration, yielding 40.0 g (0.179 mol, 60%).

**Step 2:** A mixture of the sodium salt (40.0 g, 0.179 mol) and  $\text{PCl}_5$  (41.0 g, 0.197 mol, 1.1 equiv.) was vigorously stirred until the mixture liquefied, which took about 10 minutes due to the exothermic reaction. Any solids remaining on the bottle walls were rinsed down with 5 mL of  $\text{POCl}_3$ . The mixture was then rapidly heated to 120 °C for 1 hour, cooled to room temperature, and poured onto ice with vigorous stirring. The reaction mixture was maintained at a low temperature (below 20 °C) while being stirred and was extracted with  $\text{CH}_2\text{Cl}_2$  (3 × 50 mL). The organic phase was washed with ice-cold water (1 × 50 mL) and a 5%  $\text{NaHCO}_3$  solution (2 × 100 mL), then dried and evaporated under reduced pressure. The resulting 2-bromoprop-2-ene-1-sulfonyl chloride was added dropwise to a solution of  $\text{KHF}_2$  (50 g, 0.33 M) while stirring for 12 hours. The reaction mixture was further extracted with  $\text{CH}_2\text{Cl}_2$  (2 × 50 mL), then dried and evaporated under reduced pressure. The product, 2-bromoprop-2-ene-1-sulfonyl fluoride (BPESF), was distilled at 70 °C under reduced pressure, yielding a colorless oil (14.2 g, 39% yield over 2 steps). For long-term stability, BPESF should be stored at -20 °C.

Note: The vigorous stirring of the sodium salt with  $\text{PCl}_5$  generates heat. Prepare ice water to cool the reaction bottle.

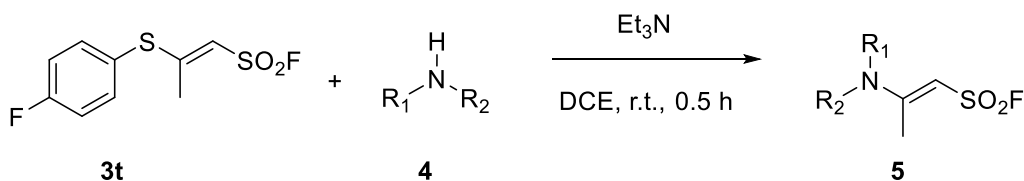
*2-bromoprop-2-ene-1-sulfonyl fluoride (2)*. Colorless liquid.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  6.18 (d,  $J$  = 2.6 Hz, 1H), 6.03 (d,  $J$  = 2.6 Hz, 1H), 4.43 (d,  $J$  = 3.5 Hz, 2H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  56.3.  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  128.5, 114.3, 61.0 (d,  $J$  = 19.0 Hz). ESI-MS HRMS  $[\text{M} + \text{Na}]^+$  calculated for  $\text{C}_3\text{H}_4\text{BrFNaO}_2\text{S}$ , 224.8992; found, 224.8995.

### 3.2 Procedure for the Synthesis of 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.), BPESF (**2**, 244 mg, 1.2 mmol, 1.2equiv.) and 8.0 mL DCE. Then, trimethylamine (202 mg, 2.0 mmol, 2.0 equiv.) was added dropwise to the above solution. The mixture was stirred at room temperature for 2 h under an air atmosphere monitored by TLC. After the reaction was completed, 20 mL of water was added to the mixture. The resulting mixture was then extracted with ethyl acetate in three portions of 10 mL each. The combined extracts were dried over anhydrous sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) and concentrated under reduced pressure. The residue was then purified by column chromatography on silica gel, using a mixture of petroleum ether and ethyl acetate as the eluent, yielding the desired product **3**.

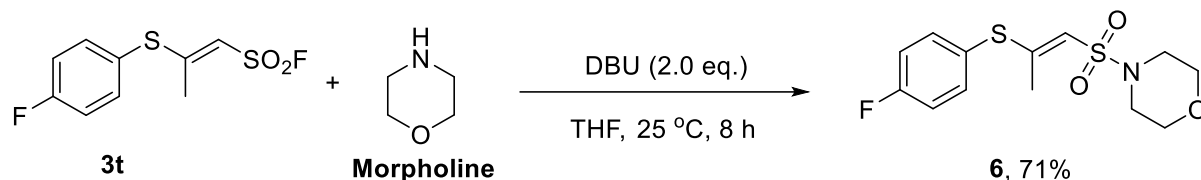
### 3.3 Diversification procedure for construction of enaminyll sulfonyl fluorides from 3t.<sup>2</sup>



According to a modified procedure from our previous work, an oven-dried reaction tube (20 mL) equipped with a magnetic stirring bar was charged with (E)-2-((4-fluorophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3t**, 0.5 mmol, 125 mg), amines (**4**, 0.5 mmol, 1.0 equiv.), and 4.0 mL DCE. Then, trimethylamine (101 mg, 1.0 mmol, 2.0 equiv.) was added dropwise to the above solution. The mixture was stirred at room temperature for 0.5 h under an air atmosphere monitored by TLC. After the reaction was completed, water (20 mL) was added to the mixture and the reaction mixture was extracted with ethyl acetate (3×10 mL). The extracts were dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated under reduced pressure. The residue was purified by column

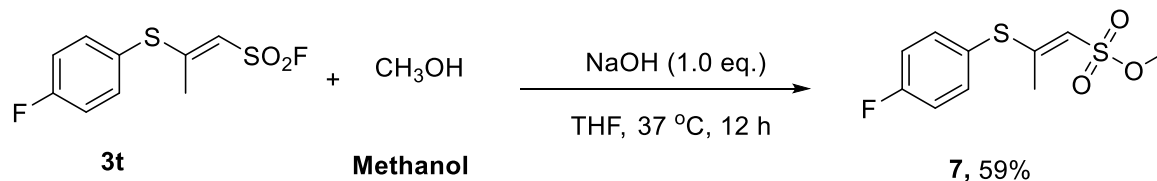
chromatography on silica gel using a mixture of petroleum ether and ethyl acetate as eluents to give the desired product **5**.

### 3.4 General procedure for synthesis of vinyl sulfonamide from morpholine.<sup>3</sup>



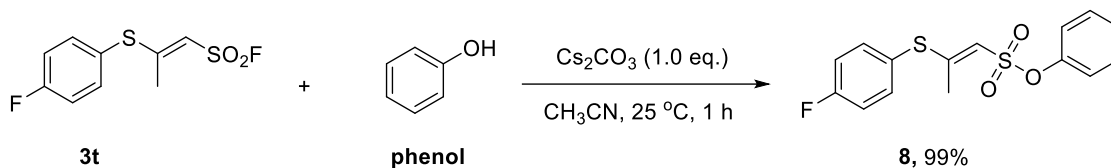
Morpholine (1.0 mmol, 2.0 eq., 87.1 mg) and DBU (1.0 mmol, 2.0 eq., 152.2 mg) were added to a stirred solution of (*E*)-2-((4-fluorophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3t**, 0.5 mmol, 125 mg) dissolved in tetrahydrofuran (2 mL) and the resulting mixture reacted at 25 °C for 8 h. The reaction was concentrated to dryness and the residue was further purified by column chromatography on silica gel by gradient elution with petroleum ether/ethyl acetate (5:1 to 3:1, v/v) as eluent to obtain pure vinyl sulfonamide **6** as white solid (114 mg, 71% yield).

### 3.5 General procedure for synthesis of vinyl sulfonate from methanol.



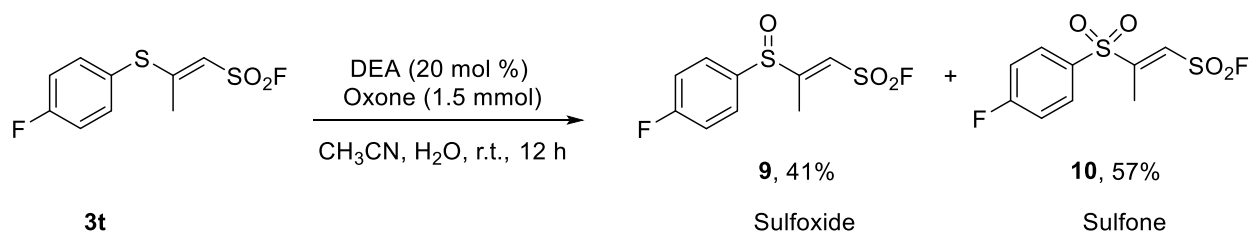
Methanol (1.0 mmol, 2.0 eq., 32.0 mg) and sodium hydroxide (0.5 mmol, 1.0 equiv, 20.0 mg) were added to a stirred solution of (*E*)-2-((4-fluorophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3t**, 0.5 mmol, 125 mg) dissolved in tetrahydrofuran (2 mL) and the resulting mixture reacted at 37 °C for 12 h. 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 (5:1 v/v) as eluent to obtain pure vinyl sulfonamide **7** as white solid (77 mg, 59% yield).

### 3.6 General procedure for synthesis of vinyl sulfonate from phenol.



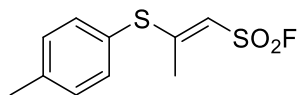
(*E*)-2-((4-fluorophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3t**, 0.5 mmol, 125 mg), phenol (0.5 mmol, 1.0 eq., 47mg), cesium carbonate (0.5 mmol, 1.0 eq., 163.4 mg) were added in a solution of acetonitrile (2 mL) and reacted at room temperature in an air atmosphere for 1 h. The reaction mixture was extracted with ethyl acetate (3×20 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, using gradient elution with a mixture of petroleum ether and ethyl acetate (10:1, v/v) as the eluent to obtain pure vinyl sulfonate **8** as light yellow solid (160 mg, 99% yield).

### 3.7 General procedure of oxidation for synthesis of sulfoxide and sulfone from **3t**.<sup>4</sup>



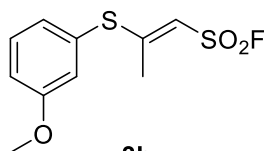
(*E*)-2-((4-fluorophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3t**, 0.5 mmol, 125 mg) and diethylamine (20 mol%) in acetonitrile (2 mL) was added to the solution of Oxone (1.5 mmol) in water (4 mL). The reaction was stirred for 12 h while monitoring by TLC, which indicated the formation of two new oxidation products. Then, the reaction mixture was extracted with ethyl acetate (3×20 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, v/v) as eluent to obtain pure sulfoxide as white solid (55 mg, 41%) and sulfone as colorless oil (81 mg, 57% yield).

## 4. Characterization



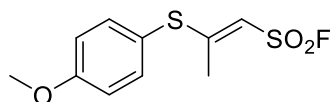
**3a**

(*E*)-2-(*p*-tolylthio)prop-1-ene-1-sulfonyl fluoride (**3a**). Light yellow solid, 222 mg, 90% yield. M.p. 69–70 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.39 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 7.9 Hz, 2H), 5.46 (s, 1H), 2.47 (s, 3H), 2.42 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ 67.5 (s, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 167.7 (d, *J* = 1.7 Hz), 142.0, 135.5, 131.4, 123.9, 109.6 (d, *J* = 25.4 Hz), 21.6, 20.0. HRMS-ESI (m/z) calcd. for [C<sub>10</sub>H<sub>12</sub>FO<sub>2</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 247.0257, found: 247.0257.



**3b**

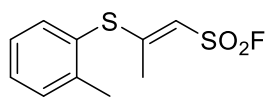
(*E*)-2-((3-methoxyphenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3b**). Light yellow solid, 262 mg, 100% yield. M.p. 83–84 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.41 (t, *J* = 8.0 Hz, 1H), 7.11 – 7.02 (m, 3H), 5.54 (s, 1H), 3.85 (s, 3H), 2.47 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ 67.5 (s, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 166.8, 161.0, 131.5, 128.2, 127.6, 120.6, 117.3, 110.9 (d, *J* = 25.5 Hz), 55.7, 20.0. HRMS-ESI (m/z) calcd. for [C<sub>10</sub>H<sub>12</sub>FO<sub>3</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 263.0206, found: 263.0207.



**3c**

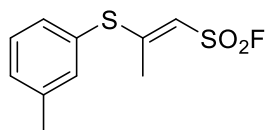
(*E*)-2-((4-methoxyphenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3c**). Light yellow oil, 257 mg, 98% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.41 (d, *J* = 8.8 Hz, 2H), 7.00 (d, *J* = 8.8 Hz, 2H), 5.44 (s, 1H), 3.87 (s, 3H), 2.45 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ 67.5 (s, 1F). <sup>13</sup>C NMR (126 MHz,

CDCl<sub>3</sub>)  $\delta$  168.3 (d,  $J$  = 1.7 Hz), 162.1, 137.2, 117.9, 116.2, 109.5 (d,  $J$  = 25.4 Hz), 55.7, 19.8. **HRMS-ESI** (m/z) calcd. for [C<sub>10</sub>H<sub>12</sub>FO<sub>3</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 263.0206, found: 263.0207.



**3d**

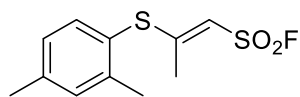
*(E)*-2-(*o*-tolylthio)prop-1-ene-1-sulfonyl fluoride (**3d**). Light yellow oil, 246 mg, 100% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent. **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.46 (dd,  $J_1$  = 22.6 Hz,  $J_2$  = 7.6 Hz, 2H), 7.40 (d,  $J$  = 6.1 Hz, 1H), 7.31 (t,  $J$  = 7.4 Hz, 1H), 5.33 (s, 1H), 2.50 (s, 3H), 2.43 (s, 3H). **<sup>19</sup>F NMR** (471 MHz, CDCl<sub>3</sub>)  $\delta$  67.6 (s, 1F). **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  165.8, 142.8, 136.5, 131.8, 131.8, 128.0, 126.6, 109.0 (d,  $J$  = 25.6 Hz), 20.2, 19.9. **HRMS-ESI** (m/z) calcd. for [C<sub>10</sub>H<sub>12</sub>FO<sub>2</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 247.0257, found: 247.0257.



**3e**

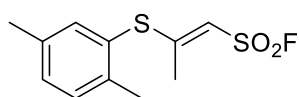
*(E)*-2-(*m*-tolylthio)prop-1-ene-1-sulfonyl fluoride (**3e**). Light yellow oil, 236 mg, 96% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>)  $\delta$  7.38 (d,  $J$  = 7.5 Hz, 1H), 7.36 – 7.29 (m, 3H), 5.49 (s, 1H), 2.47 (s, 3H), 2.41 (s, 3H). **<sup>19</sup>F NMR** (471 MHz, CDCl<sub>3</sub>)  $\delta$  67.4 (s, 1F). **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>)  $\delta$  167.3, 140.8, 136.0, 132.5, 132.2, 130.4, 127.1, 109.8 (d,  $J$  = 25.5 Hz), 21.3, 20.0. **HRMS-ESI** (m/z) calcd. for [C<sub>10</sub>H<sub>12</sub>FO<sub>2</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 247.0257, found: 247.0258.





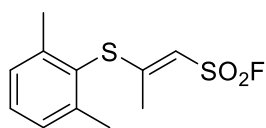
**3f**

*(E)*-2-((2,4-dimethylphenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3f**). Light yellow oil, 239 mg, 97% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35 (d,  $J = 7.8$  Hz, 1H), 7.20 (s, 1H), 7.11 (d,  $J = 8.0$  Hz, 1H), 5.33 (s, 1H), 2.48 (s, 3H), 2.38 (d,  $J = 3.9$  Hz, 6H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.6 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  166.5, 142.5, 142.5, 136.4, 132.7, 128.9, 123.3, 108.9 (d,  $J = 25.2$  Hz), 21.4, 20.2, 19.9. **HRMS-ESI** (m/z) calcd. for  $[\text{C}_{11}\text{H}_{14}\text{FO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 261.0414, found: 261.0416.



**3g**

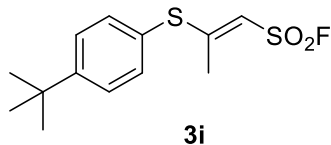
*(E)*-2-((2,5-dimethylphenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3g**). Light yellow solid, 237 mg, 91% yield. M.p. 40–41 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.28 (s, 1H), 7.26 – 7.20 (m, 2H), 5.33 (s, 1H), 2.47 (s, 3H), 2.34 (d,  $J = 8.5$  Hz, 6H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.7 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  166.1, 139.5, 137.9, 136.8, 132.8, 131.7, 126.3, 109.0 (d,  $J = 25.4$  Hz), 20.8, 19.9, 19.7. **HRMS-ESI** (m/z) calcd. for  $[\text{C}_{11}\text{H}_{14}\text{FO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 261.0414, found: 261.0415.



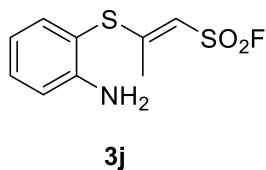
**3h**

*(E)*-2-((2,6-dimethylphenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3h**). White solid, 259 mg, 99% yield. M.p. 71–72 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 40:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.24 (d,  $J = 7.6$  Hz, 1H), 7.15 (d,

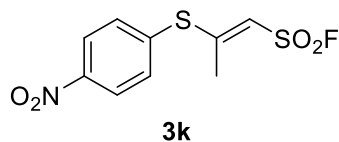
$J = 7.6$  Hz, 2H), 5.21 (s, 1H), 2.45 (s, 3H), 2.36 (s, 6H).  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.8 (s, 1F).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  165.0, 143.4, 131.5, 129.4, 126.4, 108.0(d,  $J = 25.3$  Hz), 21.2, 20.0.



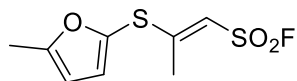
*(E)*-2-((4-(*tert*-butyl)phenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3i**). Light yellow solid, 267 mg, 92% yield. M.p. 60–61 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.51 (d,  $J = 8.4$  Hz, 2H), 7.43 (d,  $J = 8.4$  Hz, 2H), 5.52 (s, 1H), 2.47 (s, 3H), 1.36 (s, 9H).  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.5 (s, 1F).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  167.6, 155.02, 135.2, 127.8, 123.9, 109.6 (d,  $J = 25.1$  Hz), 35.2, 31.3, 20.0. HRMS-ESI (m/z) calcd. for  $[\text{C}_{13}\text{H}_{18}\text{FO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 289.0727, found: 289.0727.



*(E)*-2-((2-aminophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3j**). Colorless oil, 245 mg, 99% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.31 (t,  $J = 7.4$  Hz, 2H), 6.84 – 6.77 (m, 2H), 5.56 (s, 1H), 4.13 (d,  $J = 7.1$  Hz, 2H), 2.48 (s, 3H).  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.5 (s, 1F).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  163.9 (d,  $J = 1.8$  Hz), 148.7, 137.0, 133.4, 119.6, 116.2, 109.8 (d,  $J = 25.6$  Hz), 109.2, 19.7. HRMS-ESI (m/z) calcd. for  $[\text{C}_9\text{H}_{11}\text{FNO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 248.0210, found: 248.0211.

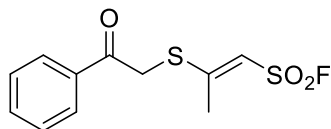


(*E*)-2-((4-nitrophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3k**). White solid, 136 mg, 67% yield. M.p. 104–105 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 8.36 (d, *J* = 8.7 Hz, 2H), 7.75 (d, *J* = 8.7 Hz, 2H), 5.56 (s, 1H), 2.51 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ 67.4 (s, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 164.2 (d, *J* = 1.9 Hz), 149.5, 136.5, 135.2, 125.4, 111.8 (d, *J* = 26.7 Hz), 20.2. HRMS-ESI (m/z) calcd. for [C<sub>9</sub>H<sub>9</sub>FNO<sub>4</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 277.9952, found: 277.9953.



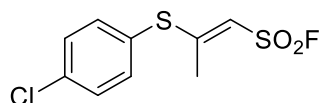
**3l**

(*E*)-2-((5-methylfuran-2-yl)thio)prop-1-ene-1-sulfonyl fluoride (**3l**). Light yellow oil, 235 mg, 99% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.43 (d, *J* = 2.0 Hz, 1H), 6.36 (d, *J* = 2.0 Hz, 1H), 5.66 (s, 1H), 2.44 (s, 3H), 2.35 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ 67.5 (s, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 165.7 (d, *J* = 1.8 Hz), 158.5, 142.6, 114.3, 109.9 (d, *J* = 25.9 Hz), 104.4, 19.5, 12.0. HRMS-ESI (m/z) calcd. for [C<sub>8</sub>H<sub>10</sub>FO<sub>3</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 237.0050, found: 237.0050.



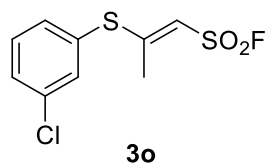
**3m**

(*E*)-2-((2-oxo-2-phenylethyl)thio)prop-1-ene-1-sulfonyl fluoride (**3m**). Light yellow solid, 160 mg, 58% yield. M.p. 121–122 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.98 (d, *J* = 7.0 Hz, 2H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.54 (t, *J* = 7.8 Hz, 2H), 6.10 (s, 1H), 4.33 (s, 2H), 2.46 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ 67.5 (s, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 191.3, 163.5, 134.7, 134.6, 129.2, 128.7, 109.6 (d, *J* = 25.8 Hz), 40.0, 20.8. HRMS-ESI (m/z) calcd. for [C<sub>11</sub>H<sub>12</sub>FO<sub>3</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 275.0206, found: 275.0209.

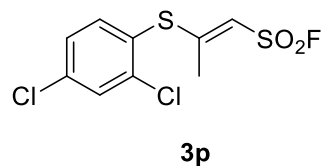


**3n**

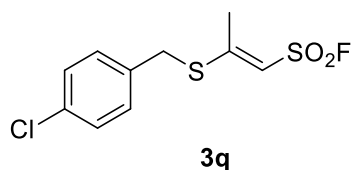
(*E*)-2-((4-chlorophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3n**). Light yellow solid, 201 mg, 75% yield. M.p. 80–81 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.47 (q,  $J = 8.6$  Hz, 4H), 5.47 (s, 1H), 2.47 (s, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.4 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  166.3, 138.2, 136.89, 131.0, 125.8, 110.4 (d,  $J = 26.1$  Hz), 19.9. **HRMS-ESI** ( $m/z$ ) calcd. for  $[\text{C}_9\text{H}_9\text{ClFO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 266.9711, found: 266.9712.



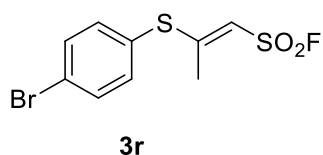
(*E*)-2-((3-chlorophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3o**). Yellow solid, 169 mg, 63% yield. M.p. 71–72 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53 (dd,  $J_1 = 9.2$ ,  $J_2 = 1.6$  Hz, 2H), 7.50 – 7.39 (m, 2H), 5.52 (s, 1H), 2.48 (s, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.5 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  165.7 (d,  $J = 1.9$  Hz), 136.3, 135.3, 133.8, 131.7, 131.7, 129.1, 110.8 (d,  $J = 26.2$  Hz), 20.0. **HRMS-ESI** ( $m/z$ ) calcd. for  $[\text{C}_9\text{H}_9\text{ClFO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 266.9711, found: 266.9713.



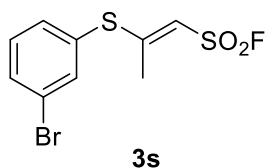
(*E*)-2-((2,4-dichlorophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3p**). Yellow solid, 171 mg, 57% yield. M.p. 99–100 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.63 (d,  $J = 2.2$  Hz, 1H), 7.56 (d,  $J = 8.3$  Hz, 1H), 7.39 (dd,  $J_1 = 8.3$  Hz,  $J_2 = 2.2$  Hz, 1H), 5.43 (s, 1H), 2.48 (s, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.6 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  163.3, 140.6, 139.1, 138.4, 131.4, 129.1, 125.1, 110.5 (d,  $J = 26.4$  Hz), 19.7. **HRMS-ESI** ( $m/z$ ) calcd. for  $[\text{C}_9\text{H}_8\text{Cl}_2\text{FO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 300.9321, found: 300.9322.



*(E)*-2-((4-chlorobenzyl)thio)prop-1-ene-1-sulfonyl fluoride (**3q**). Light yellow solid, 250 mg, 89% yield. M.p. 50–51 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35 (d,  $J$  = 8.4 Hz, 2H), 7.28 (d,  $J$  = 8.5 Hz, 2H), 5.94 (s, 1H), 4.01 (s, 2H), 2.42 (s, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.6 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  164.8, 134.5, 131.5, 130.3, 129.5, 108.8 (d,  $J$  = 25.7 Hz), 36.8, 20.6. **HRMS-ESI** (m/z) calcd. for  $[\text{C}_{10}\text{H}_{11}\text{ClFO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 280.9868, found: 280.9868.

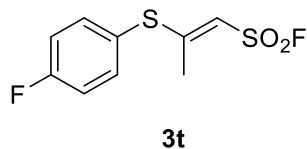


*(E)*-2-((4-bromophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3r**). Yellow solid, 92 mg, 59% yield. M.p. 76–77°C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.65 (d,  $J$  = 8.4 Hz, 2H), 7.39 (d,  $J$  = 8.3 Hz, 2H), 5.49 (s, 1H), 2.47 (s, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.4 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  166.0 (d,  $J$  = 1.9 Hz), 137.1, 134.0, 126.5, 126.4, 110.5 (d,  $J$  = 26.0 Hz), 20.0. **HRMS-ESI** (m/z) calcd. for  $[\text{C}_9\text{H}_9\text{BrFO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 310.9206, found: 310.9207.

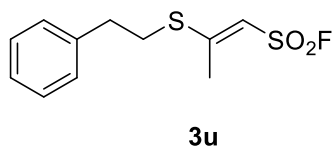


*(E)*-2-((3-bromophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3s**). Light yellow solid, 262 mg, 84% yield. M.p. 95–96 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.68 (d,  $J$  = 9.5 Hz, 2H), 7.47 (d,  $J$  = 8.0 Hz, 1H), 7.40 (t,  $J$  = 7.8 Hz, 1H), 5.51 (s, 1H), 2.47 (s, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.5

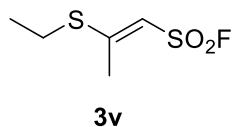
(s, 1F).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  165.8, 138.1, 134.6, 134.2, 131.9, 129.3, 124.1, 110.7 (d,  $J = 26.0$  Hz), 19.9. **HRMS-ESI** (m/z) calcd. for  $[\text{C}_9\text{H}_9\text{BrFO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 310.9206, found: 310.9207.



*(E)*-2-((4-fluorophenyl)thio)prop-1-ene-1-sulfonyl fluoride (**3t**). Light yellow oil, 247 mg, 99% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52 (t,  $J = 6.1$  Hz, 2H), 7.21 (t,  $J = 8.5$  Hz, 2H), 5.44 (s, 1H), 2.47 (s, 3H).  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.4 (s, 1F), -107.3 (s, 1F).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  166.9, 165.5, 163.5, 137.9 (d,  $J = 8.8$  Hz), 122.7 (d,  $J = 3.6$  Hz), 118.1, 117.9, 110.1 (d,  $J = 26.1$  Hz), 19.8. **HRMS-ESI** (m/z) calcd. for  $[\text{C}_9\text{H}_9\text{F}_2\text{O}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 251.0007, found: 251.0008.

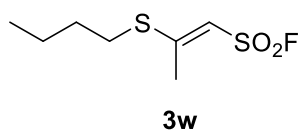


*(E)*-2-(phenethylthio)prop-1-ene-1-sulfonyl fluoride (**3u**). Light yellow oil, 226 mg, 87% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35 (t,  $J = 7.3$  Hz, 2H), 7.29 (t,  $J = 7.4$  Hz, 1H), 7.22 (d,  $J = 6.8$  Hz, 2H), 5.88 (s, 1H), 3.07 (t,  $J = 6.8$  Hz, 2H), 2.99 (t,  $J = 7.0$  Hz, 2H), 2.41 (s, 3H).  $^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.6 (s, 1F).  $^{13}\text{C}$  NMR (126 MHz,  $\text{CDCl}_3$ )  $\delta$  165.5, 138.6, 129.0, 128.5, 127.3, 108.0 (d,  $J = 25.4$  Hz), 33.9, 33.3, 20.9. **HRMS-ESI** (m/z) calcd. for  $[\text{C}_{11}\text{H}_{14}\text{FO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 261.0414, found: 261.0416.

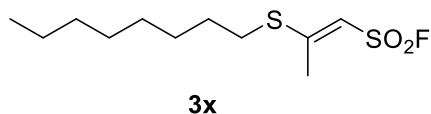


*(E)*-2-(ethylthio)prop-1-ene-1-sulfonyl fluoride (**3v**). Light yellow oil, 128 mg, 69% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1

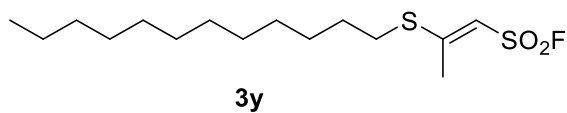
(v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.86 (s, 1H), 2.83 (q,  $J = 7.4$  Hz, 2H), 2.41 (s, 3H), 1.37 (t,  $J = 7.5$  Hz, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.5 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  165.9, 107.7 (d,  $J = 25.4$  Hz), 26.6, 20.8, 12.1. **HRMS-ESI** (m/z) calcd. for  $[\text{C}_5\text{H}_{10}\text{FO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 85.0101, found: 85.0101.



*(E)*-2-(butylthio)prop-1-ene-1-sulfonyl fluoride (**3v**). Light yellow oil, 152 mg, 72% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.86 (s, 1H), 2.79 (t,  $J = 7.4$  Hz, 2H), 2.41 (d,  $J = 1.0$  Hz, 3H), 1.68 (p,  $J = 7.4$  Hz, 2H), 1.47 (h,  $J = 7.4$  Hz, 2H), 0.96 (t,  $J = 7.4$  Hz, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.6 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  166.2, 107.7 (d,  $J = 25.3$  Hz), 32.3, 29.0, 22.2, 21.0, 13.6. **HRMS-ESI** (m/z) calcd. for  $[\text{C}_7\text{H}_{14}\text{FO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 213.0414, found: 213.0415.

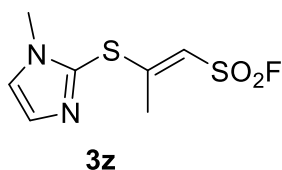


*(E)*-2-(octylthio)prop-1-ene-1-sulfonyl fluoride (**3x**). Light yellow oil, 210 mg, 78% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.85 (s, 1H), 2.78 (t,  $J = 7.4$  Hz, 2H), 2.41 (d,  $J = 1.2$  Hz, 3H), 1.69 (p,  $J = 7.4$  Hz, 2H), 1.42 (q,  $J = 7.4$  Hz, 2H), 1.36 – 1.26 (m, 8H), 0.92 – 0.86 (m, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.6 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  166.2, 107.7 (d,  $J = 25.3$  Hz), 32.6, 31.85, 29.2, 29.1, 29.0, 27.0, 22.7, 21.0, 14.2. **HRMS-ESI** (m/z) calcd. for  $[\text{C}_{11}\text{H}_{22}\text{FO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 269.1040, found: 269.1040.

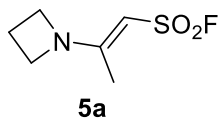


(*E*)-2-(dodecylthio)prop-1-ene-1-sulfonyl fluoride (**3y**). Yellow oil, 210 mg, 99% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  5.85 (s, 1H), 2.78 (t,  $J = 7.4$  Hz, 2H), 2.41 (s, 3H), 1.71 – 1.65 (m, 2H), 1.28 (d,  $J = 10.0$  Hz, 18H), 0.89 (d,  $J = 6.6$  Hz, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  67.6 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  166.01, 107.8 (d,  $J = 25.3$  Hz), 32.6, 32.0, 29.73, 29.6, 29.5, 29.5, 29.1, 29.0, 27.0, 22.8, 20.9, 14.2. **HRMS-ESI** ( $m/z$ ) calcd. for  $[\text{C}_{15}\text{H}_{30}\text{FO}_2\text{S}_2]^+$  ( $[\text{M}+\text{H}]^+$ ): 325.1666, found: 325.1667.

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

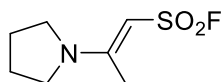


(*E*)-2-((1-methyl-1H-imidazol-2-yl)thio)prop-1-ene-1-sulfonyl fluoride (**3z**). yellow solid, 209 mg, 88% yield. M.p. 76–77 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 3:1 to 2:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{DMSO}-d_6$ )  $\delta$  7.73 (s, 1H), 7.37 (s, 1H), 6.11 (s, 1H), 3.85 (s, 3H), 2.64 (s, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{DMSO}-d_6$ )  $\delta$  68.9 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{DMSO}-d_6$ )  $\delta$  165.6 (d,  $J = 2.4$  Hz), 131.6, 130.7 (d,  $J = 8.6$  Hz), 126.4 (d,  $J = 9.6$  Hz), 110.6 (dd,  $J_1 = 25.2$  Hz,  $J_2 = 6.2$  Hz), 33.6, 18.9.



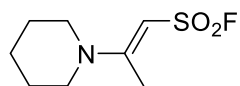
(*E*)-2-(azetidin-1-yl)prop-1-ene-1-sulfonyl fluoride (**5a**). Colorless oil, 85 mg, 95% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 5:1 (v/v) as eluent.  $^1\text{H NMR}$  (500 MHz,  $\text{CDCl}_3$ )  $\delta$  4.49 (d,  $J = 2.1$  Hz, 1H), 4.17 (t,  $J = 6.7$  Hz, 2H), 3.92 (t,  $J = 7.7$  Hz, 2H), 2.40 (p,  $J = 7.6$  Hz, 2H), 2.12 (s, 3H).  $^{19}\text{F NMR}$  (471 MHz,  $\text{CDCl}_3$ )  $\delta$  73.0 (s, 1F).  $^{13}\text{C NMR}$  (126 MHz,  $\text{CDCl}_3$ )  $\delta$  161.8, 78.8 (d,  $J = 24.2$  Hz), 51.4, 51.0, 15.0, 13.8. **HRMS-ESI** ( $m/z$ ) calcd. for  $[\text{C}_6\text{H}_{11}\text{FNO}_2\text{S}]^+$  ( $[\text{M}+\text{H}]^+$ ): 180.0489, found: 180.0489.





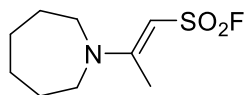
**5b**

(*E*)-2-(pyrrolidin-1-yl)prop-1-ene-1-sulfonyl fluoride (**5b**). Colorless oil, 222 mg, 97% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 3:1 (v/v) as eluent. **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 4.73 (s, 1H), 3.49 (t, *J* = 5.5 Hz, 2H), 3.19 (t, *J* = 6.7 Hz, 2H), 2.36 (s, 3H), 2.01 (td, *J*<sub>1</sub> = 12.4 Hz, *J*<sub>2</sub> = 12.0, 6.6 Hz, 4H). **<sup>19</sup>F NMR** (471 MHz, CDCl<sub>3</sub>) δ 73.4 (s, 1F). **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 160.6, 81.6 (d, *J* = 23.5 Hz), 49.0, 25.4, 25.0, 17.4. **HRMS-ESI** (m/z) calcd. for [C<sub>7</sub>H<sub>13</sub>FNO<sub>2</sub>S]<sup>+</sup> ([M+H]<sup>+</sup>): 194.0646, found: 194.0647.



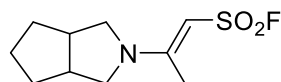
**5c**

(*E*)-2-(piperidin-1-yl)prop-1-ene-1-sulfonyl fluoride (**5c**). Yellow oil, 102 mg, 98% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 3:1 (v/v) as eluent. **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 5.02 (s, 1H), 3.36 (t, *J* = 5.5 Hz, 4H), 2.33 (s, 3H), 1.67 (dq, *J*<sub>1</sub> = 27.3 Hz, *J*<sub>2</sub> = 5.4 Hz, 6H). **<sup>19</sup>F NMR** (471 MHz, CDCl<sub>3</sub>) δ 73.4 (s, 1F). **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 161.5, 83.6 (d, *J* = 23.7 Hz), 48.3, 25.5, 23.9, 16.6. **HRMS-ESI** (m/z) calcd. for [C<sub>8</sub>H<sub>15</sub>FNO<sub>2</sub>S]<sup>+</sup> ([M+H]<sup>+</sup>): 208.0802, found: 208.0804.



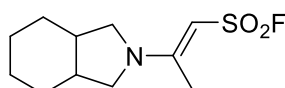
**5d**

(*E*)-2-(azepan-1-yl)prop-1-ene-1-sulfonyl fluoride (**5d**). Light yellow oil, 63 mg, 57% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **<sup>1</sup>H NMR** (500 MHz, DMSO-*d*<sub>6</sub>) δ 5.00 (s, 1H), 3.54 (t, *J* = 6.0 Hz, 2H), 3.41 (t, *J* = 6.2 Hz, 2H), 2.32 (s, 3H), 1.67 (d, *J* = 21.8 Hz, 4H), 1.49 (s, 4H). **<sup>19</sup>F NMR** (471 MHz, CDCl<sub>3</sub>) δ 73.3 (s, 1F). **<sup>13</sup>C NMR** (126 MHz, DMSO-*d*<sub>6</sub>) δ 162.6, 79.3 (d, *J* = 21.7 Hz), 50.5, 49.9, 28.3, 26.1, 25.7, 24.54, 15.6. **HRMS-ESI** (m/z) calcd. for [C<sub>9</sub>H<sub>17</sub>FNO<sub>2</sub>S]<sup>+</sup> ([M+H]<sup>+</sup>): 222.0959, found: 222.0959.



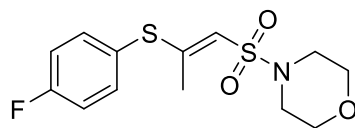
**5e**

(*E*)-2-(hexahydrocyclopenta[*c*]pyrrol-2(1*H*)-yl)prop-1-ene-1-sulfonyl fluoride (**5e**). White solid, 115 mg, 99% yield. M.p. 42–43 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 3:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 4.71 (s, 1H), 3.70 (s, 1H), 3.41 (s, 1H), 3.28 (d, *J* = 11.3 Hz, 1H), 2.96 (d, *J* = 9.8 Hz, 1H), 2.76 (s, 2H), 2.33 (s, 3H), 1.90 (dq, *J*<sub>1</sub> = 14.3 Hz, *J*<sub>2</sub> = 7.5 Hz, 2H), 1.79 (tt, *J*<sub>1</sub> = 14.3 Hz, *J*<sub>2</sub> = 7.0 Hz, 1H), 1.73 – 1.64 (m, 1H), 1.56 (s, 2H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ 73.3 (s, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 160.5, 82.4 (d, *J* = 23.6 Hz), 55.0, 51.9, 42.8, 42.1, 31.6, 25.3, 17.4. HRMS-ESI (*m/z*) calcd. for [C<sub>10</sub>H<sub>17</sub>FNO<sub>2</sub>S]<sup>+</sup> ([M+H]<sup>+</sup>): 234.0959, found: 234.0960.



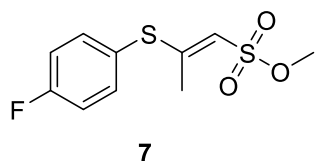
**5f**

(*E*)-2-(octahydro-2*H*-isoindol-2-yl)prop-1-ene-1-sulfonyl fluoride (**5f**). White solid, 117 mg, 95% yield. M.p. 106–107 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 3:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 4.72 (s, 1H), 3.53 – 3.47 (m, 1H), 3.35 (dd, *J*<sub>1</sub> = 11.6 Hz, *J*<sub>2</sub> = 6.8 Hz, 1H), 3.21 (dd, *J*<sub>1</sub> = 10.7 Hz, *J*<sub>2</sub> = 7.8 Hz, 1H), 3.05 (dd, *J*<sub>1</sub> = 11.2 Hz, *J*<sub>2</sub> = 6.1 Hz, 1H), 2.34 (s, 3H), 2.29 (p, *J* = 6.2 Hz, 2H), 1.64 (ddt, *J*<sub>1</sub> = 12.5 Hz, *J*<sub>2</sub> = 8.1 Hz, *J*<sub>3</sub> = 3.6 Hz, 2H), 1.54 – 1.38 (m, 6H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ 73.4 (s, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 161.3, 81.3 (d, *J* = 23.5 Hz), 53.0, 52.9, 37.0, 36.6, 25.8, 25.7, 22.7, 22.5, 17.3. HRMS-ESI (*m/z*) calcd. for [C<sub>11</sub>H<sub>19</sub>FNO<sub>2</sub>S]<sup>+</sup> ([M+H]<sup>+</sup>): 248.1115, found: 248.1116.

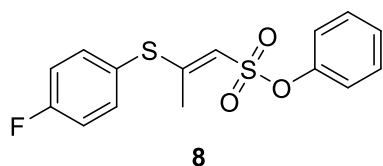


**6**

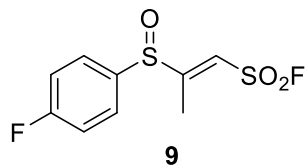
(*E*)-4-((2-((4-fluorophenyl)thio)prop-1-en-1-yl)sulfonyl)morpholine (**6**). White solid, 114 mg, 71% yield. M.p. 100–101 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 5:1 to 3:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.54 – 7.47 (m, 2H), 7.17 (t, *J* = 8.3 Hz, 2H), 5.26 (s, 1H), 3.75 – 3.69 (m, 4H), 3.03 – 2.97 (m, 4H), 2.38 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ -108.5 (s, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 165.2, 163.2, 158.4, 138.0 (d, *J* = 8.7 Hz), 124.3 (d, *J* = 3.5 Hz), 117.8, 117.6, 112.8, 66.3, 45.8, 19.3. HRMS-ESI (*m/z*) calcd. for [C<sub>13</sub>H<sub>17</sub>FNO<sub>3</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 318.0628, found: 318.0629.



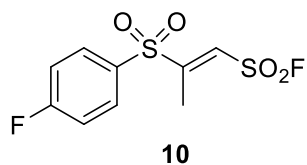
methyl (*E*)-2-((4-fluorophenyl)thio)prop-1-ene-1-sulfonate (**7**). White solid, 77 mg, 59% yield. M.p. 115–116 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 5:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.54 – 7.46 (m, 2H), 7.17 (t, *J* = 8.5 Hz, 2H), 5.38 (s, 1H), 3.74 (s, 3H), 2.39 (d, *J* = 1.0 Hz, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ -108.2 (s, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 165.3, 163.3, 161.0, 137.9 (d, *J* = 8.8 Hz), 123.7 (d, *J* = 3.6 Hz), 117.9, 117.7, 113.1, 55.5, 19.3. HRMS-ESI (*m/z*) calcd. for [C<sub>10</sub>H<sub>12</sub>FO<sub>3</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 263.0206, found: 263.0206.



phenyl (*E*)-2-((4-fluorophenyl)thio)prop-1-ene-1-sulfonate (**8**). Light yellow solid, 160 mg, 99% yield. M.p. 89–90 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 10:1 (v/v) as eluent. <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.40 (dt, *J*<sub>1</sub> = 21.0 Hz, *J*<sub>2</sub> = 8.1 Hz, 4H), 7.31 (d, *J* = 7.4 Hz, 1H), 7.14 (t, *J* = 7.5 Hz, 4H), 5.38 (s, 1H), 2.27 (s, 3H). <sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>) δ -108.0 (s, 1F). <sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>) δ 165.3, 163.3, 162.4, 149.6, 137.9 (d, *J* = 8.7 Hz), 129.8, 127.3, 123.4, 122.5, 117.8, 117.6, 113.0, 19.4.

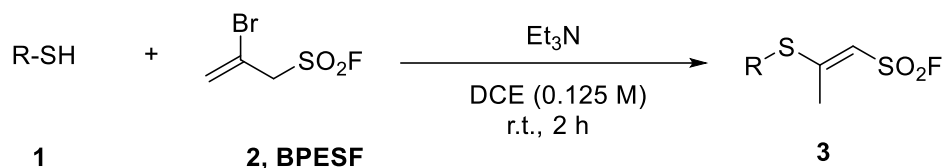


*(E)*-2-((4-fluorophenyl)sulfinyl)prop-1-ene-1-sulfonyl fluoride (**9**). White solid, 55 mg, 41% yield. M.p. 74–75 °C. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.67 – 7.60 (m, 2H), 7.27 (d, *J* = 1.5 Hz, 1H), 7.23 – 7.19 (m, 2H), 2.03 (s, 3H). **<sup>19</sup>F NMR** (471 MHz, CDCl<sub>3</sub>) δ 67.1 (s, 1F), -103.9 (s, 1F). **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 166.7, 166.3 (d, *J* = 3.2 Hz), 164.6, 135.5 (d, *J* = 3.4 Hz), 128.8 (d, *J* = 9.4 Hz), 120.8 (d, *J* = 30.3 Hz), 117.8, 117.7, 13.4. **HRMS-ESI** (*m/z*) calcd. for [C<sub>9</sub>H<sub>9</sub>F<sub>2</sub>O<sub>3</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 266.9956, found: 266.9957.



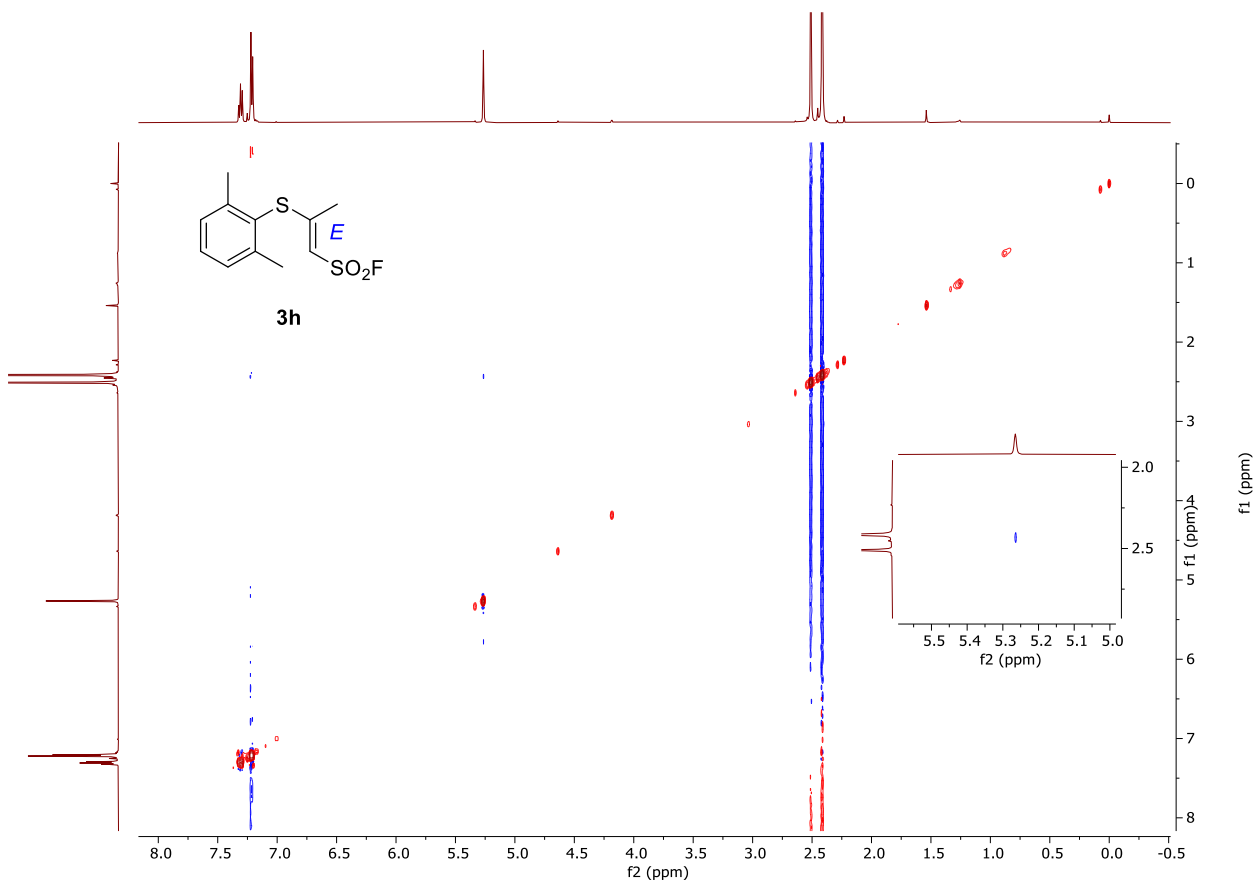
*(E)*-2-((4-fluorophenyl)sulfonyl)prop-1-ene-1-sulfonyl fluoride (**10**). Colorless oil, 81 mg, 57% yield. Purification by column chromatography on silica gel using petroleum ether / ethyl acetate = 20:1 (v/v) as eluent. **<sup>1</sup>H NMR** (500 MHz, CDCl<sub>3</sub>) δ 7.95 (dd, *J*<sub>1</sub> = 8.6 Hz, *J*<sub>2</sub> = 4.9 Hz, 2H), 7.54 – 7.47 (m, 1H), 7.33 (t, *J* = 8.4 Hz, 2H), 2.35 (s, 3H). **<sup>19</sup>F NMR** (471 MHz, CDCl<sub>3</sub>) δ 67.2 (s, 1F), -99.8 (s, 1F). **<sup>13</sup>C NMR** (126 MHz, CDCl<sub>3</sub>) δ 168.0, 165.9, 157.7 (d, *J* = 3.6 Hz), 132.3 (d, *J* = 10.0 Hz), 131.8 (d, *J* = 3.2 Hz), 127.8 (d, *J* = 30.9 Hz), 117.8, 117.6, 13.8. **HRMS-ESI** (*m/z*) calcd. for [C<sub>9</sub>H<sub>9</sub>F<sub>2</sub>O<sub>4</sub>S<sub>2</sub>]<sup>+</sup> ([M+H]<sup>+</sup>): 282.9905, found: 282.9905.

### 5. Scale-up reaction procedure for (3)



An oven-dried reaction tube (250 mL) equipped with a magnetic stirring bar was charged with thiols (**1**, 10.0 mmol, 1.0 equiv.), BPESF (**2**, 2.44 g, 12.0 mmol, 1.2equiv.) and 80.0 mL DCE. Then, trimethylamine (2.02 g, 20.0 mmol, 2.0 equiv.) was added dropwise to the above solution. The mixture was stirred at room temperature for 2 h under an air atmosphere monitored by TLC. After the reaction was completed, 50 mL of water was added to the mixture. The resulting mixture was then extracted with ethyl acetate (3 x 50 mL). The combined extracts were dried over anhydrous sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) and concentrated under reduced pressure. The residue was then purified by column chromatography on silica gel, using a mixture of petroleum ether and ethyl acetate as the eluent, yielding the desired product **3**.

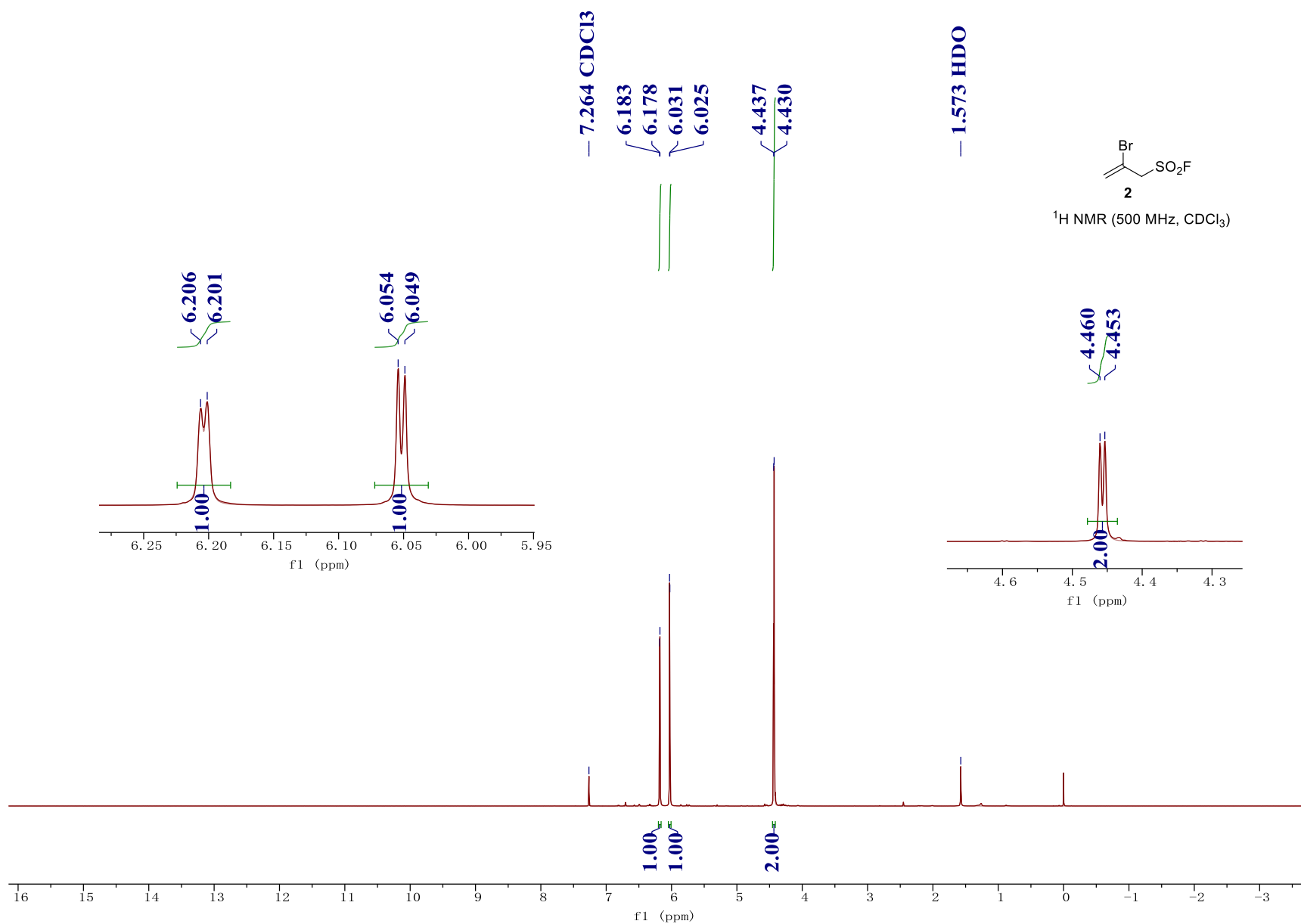
## 6. NOESY of (*E*)-2-((2,6-dimethylphenyl)thio)prop-1-ene-1-sulfonyl fluoride (3h)

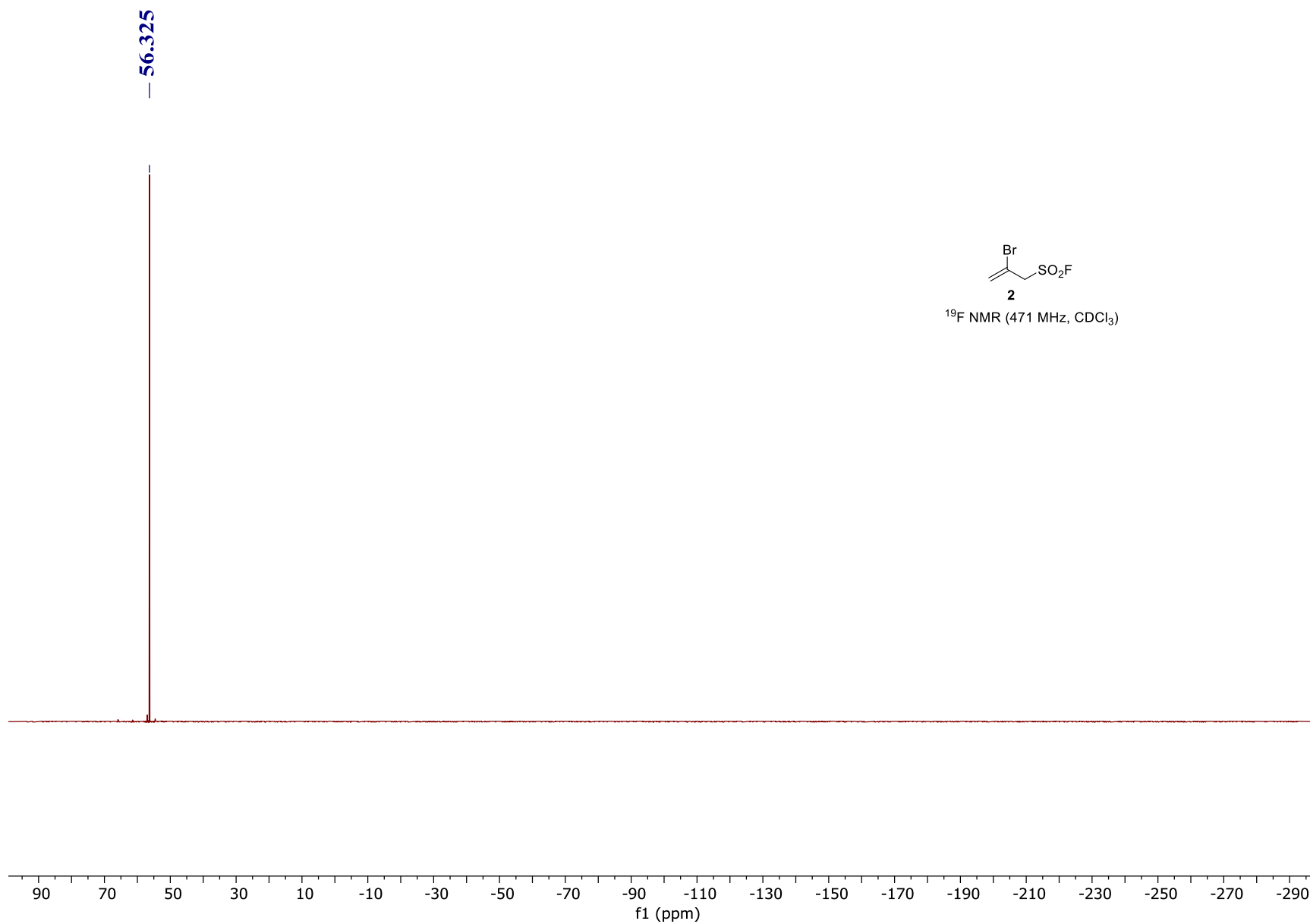


## 7. References

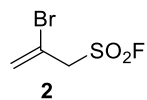
- 1X. Zhang and H.-L. Qin, *Org. Lett.*, 2022, **24**, 9311–9315.
- 2M. Liu, W. Tang and H.-L. Qin, *J. Org. Chem.*, 2023, **88**, 1909–1917.
- 3X. Zhang, W. Fang, R. Leikkala, W. Tang and H. Qin, *Adv. Synth. Catal.*, 2020, **362**, 3358–3363.
- 4R. Kupwade, S. Khot, U. Lad, U. Desai and P. Wadgaonkar, *Res. Chem. Intermed.*, 2017, **43**, 6875–6888.

## 8. NMR Spectra

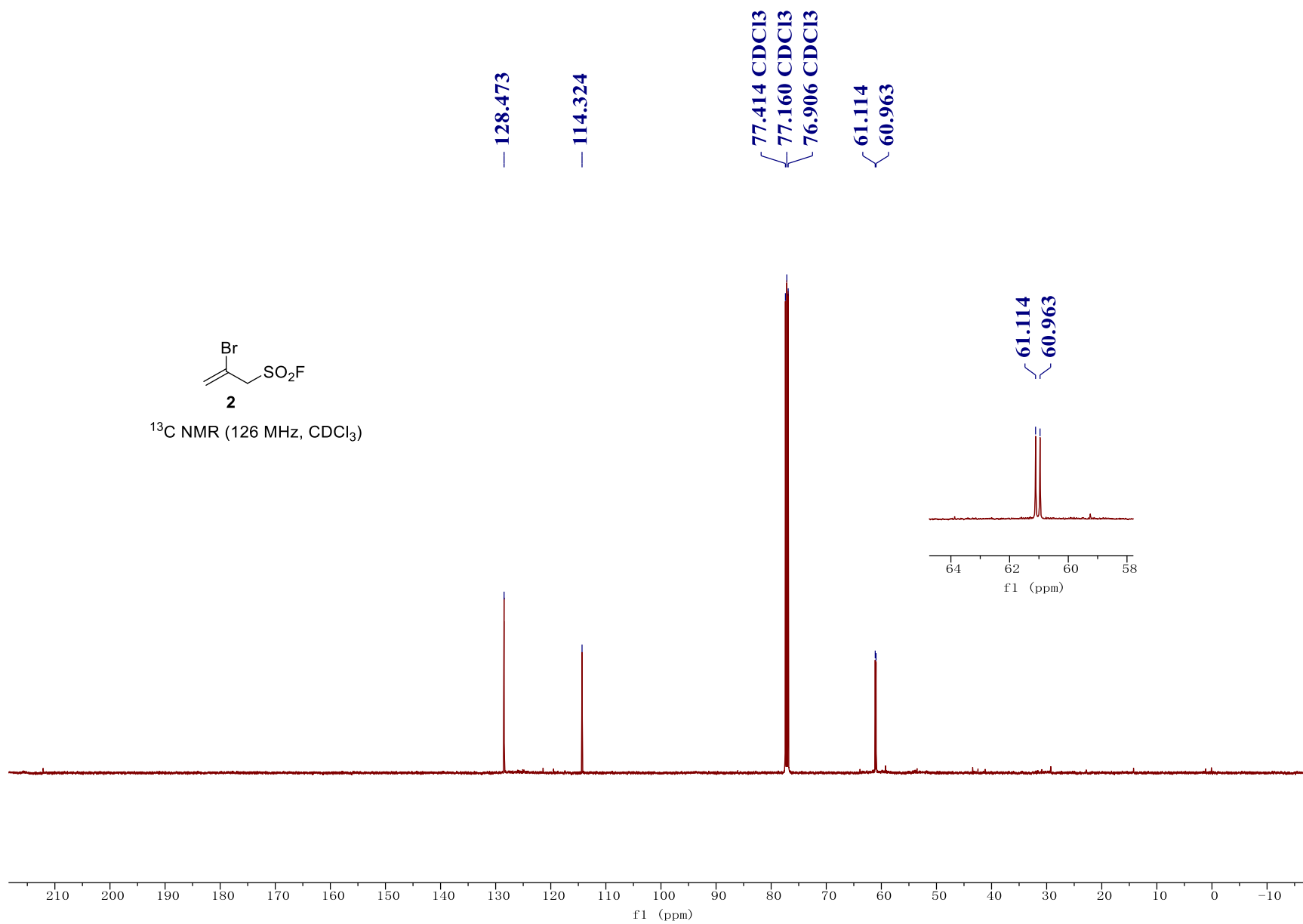


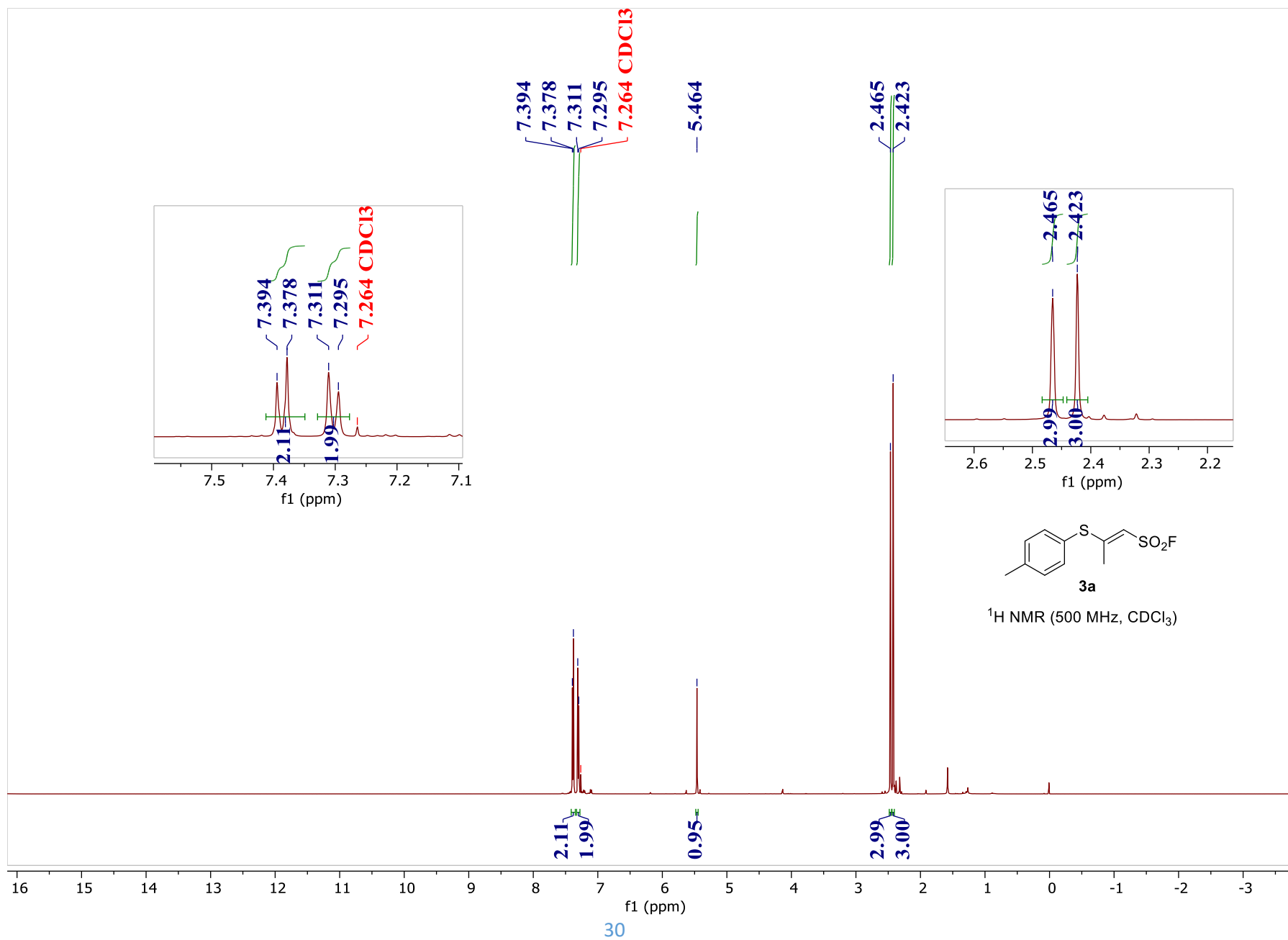




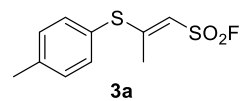


<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)

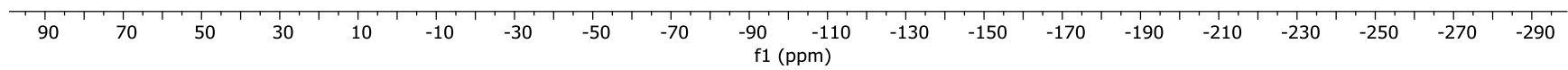


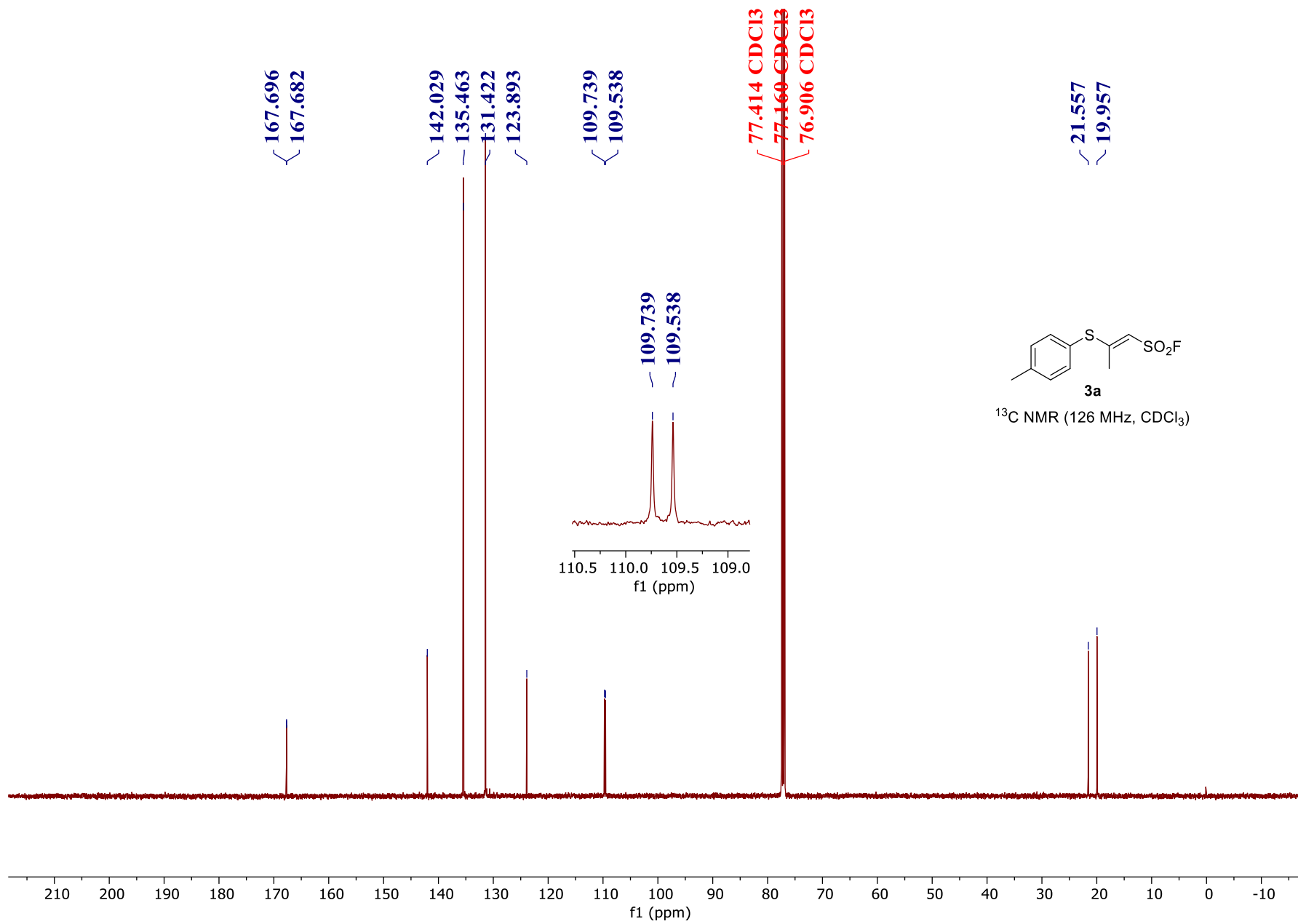


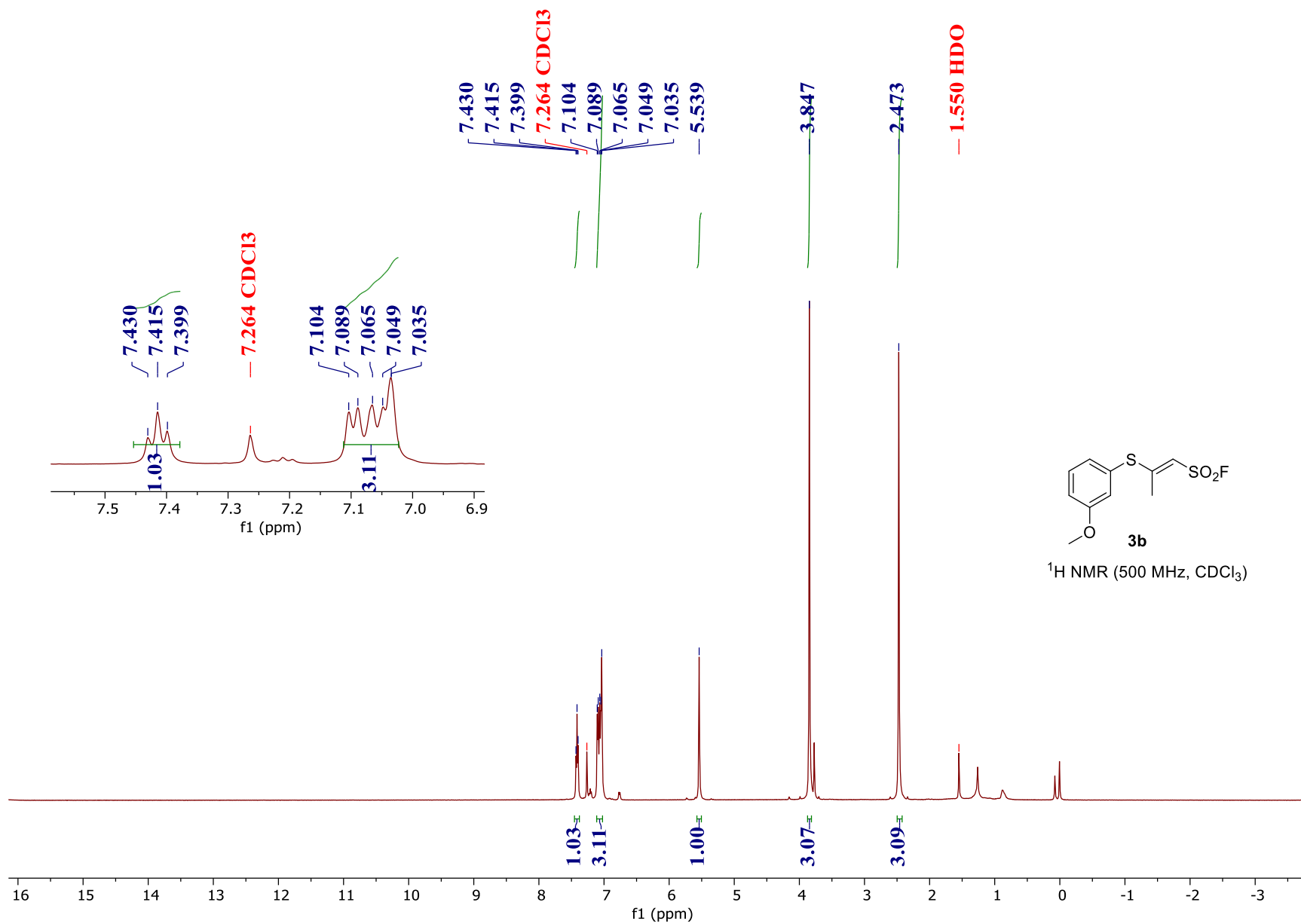
— 67.445

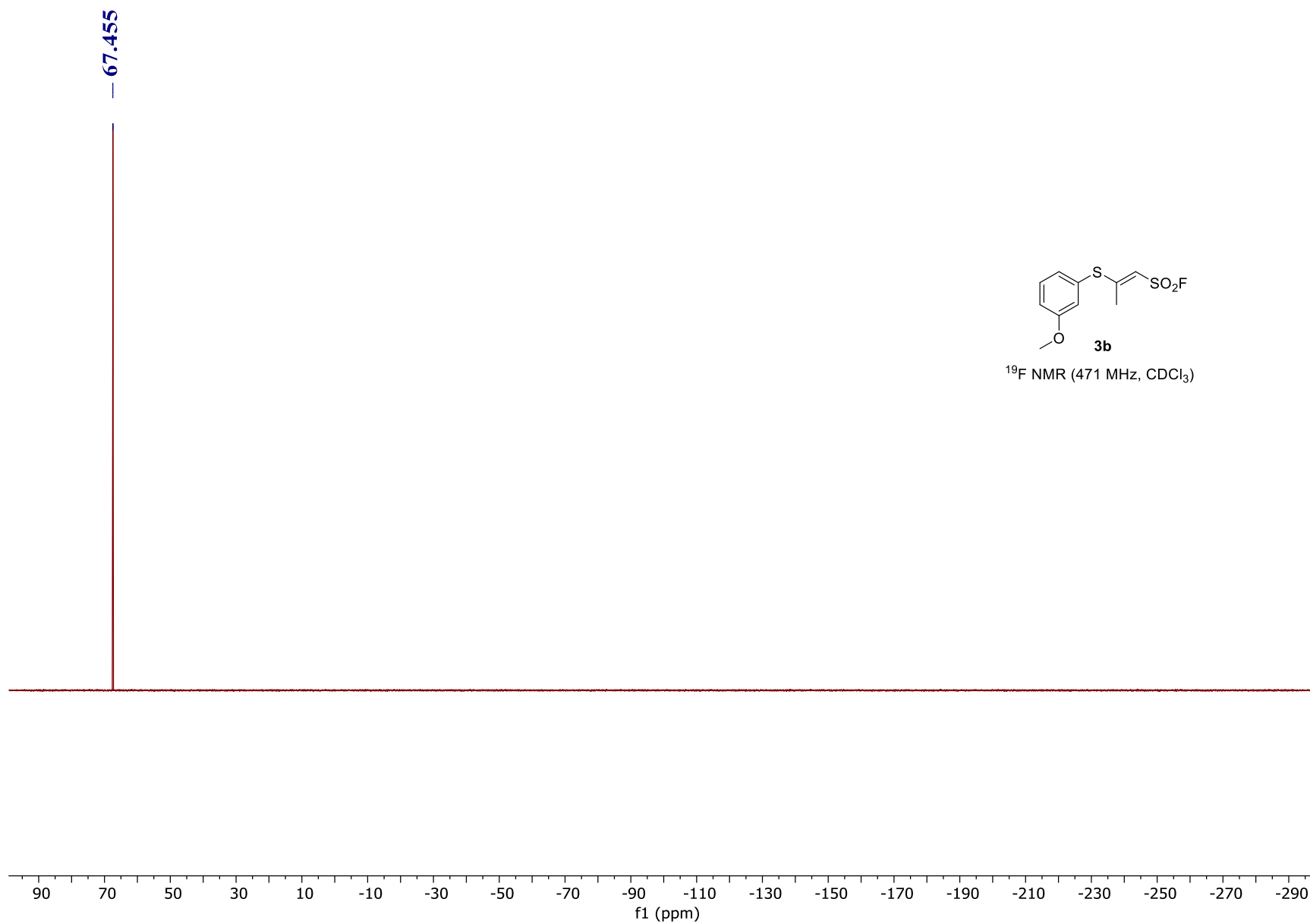


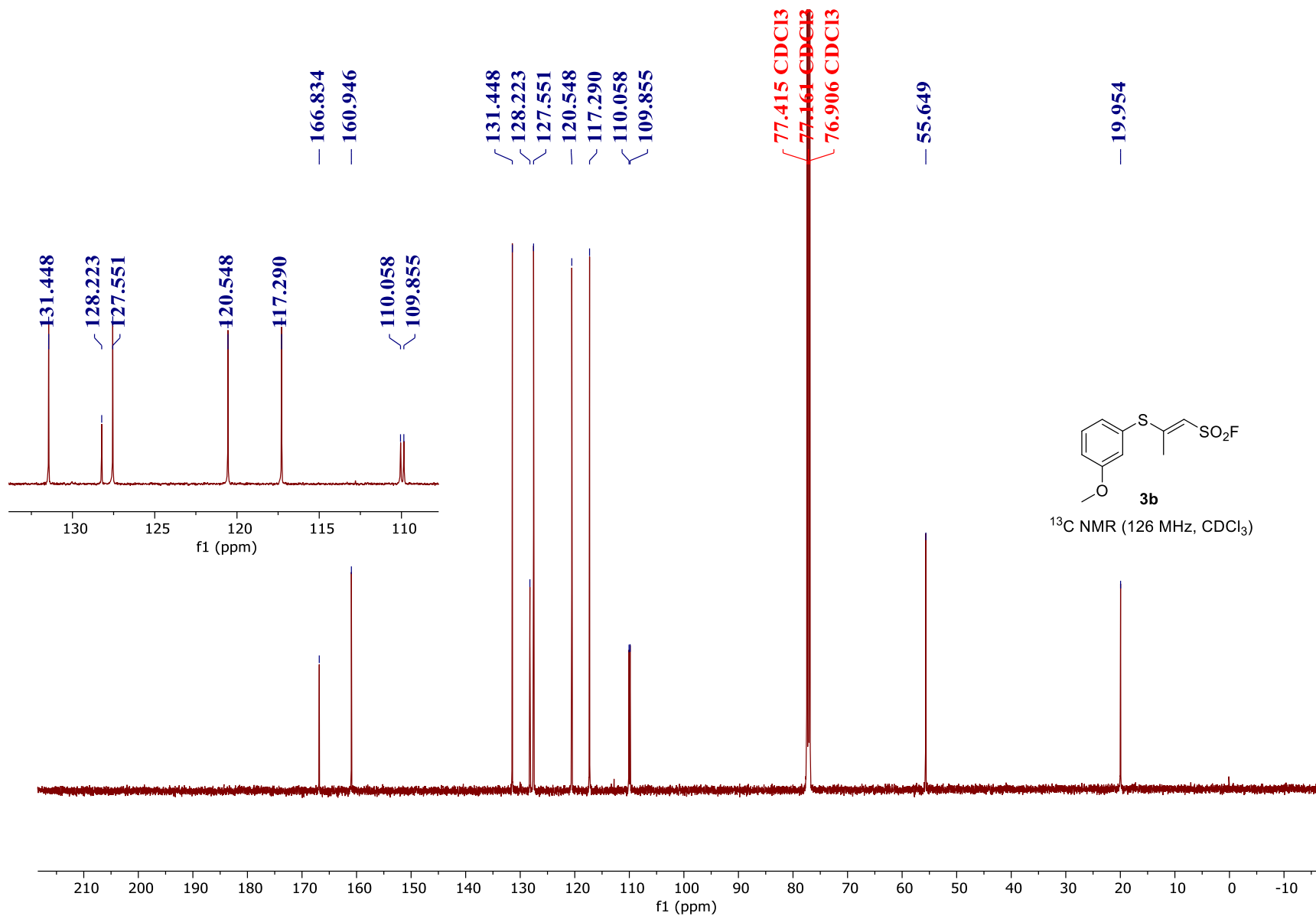
<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)

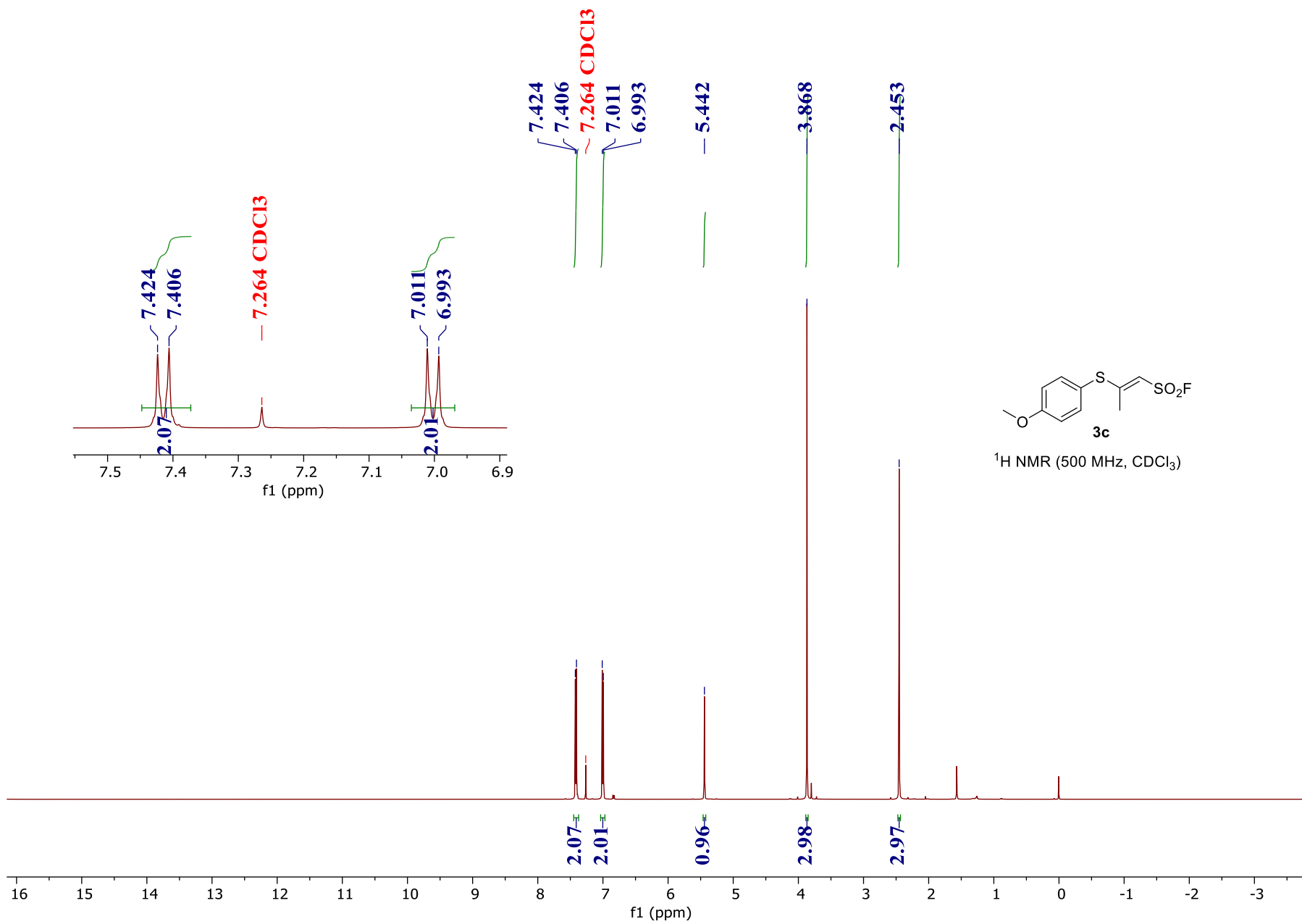






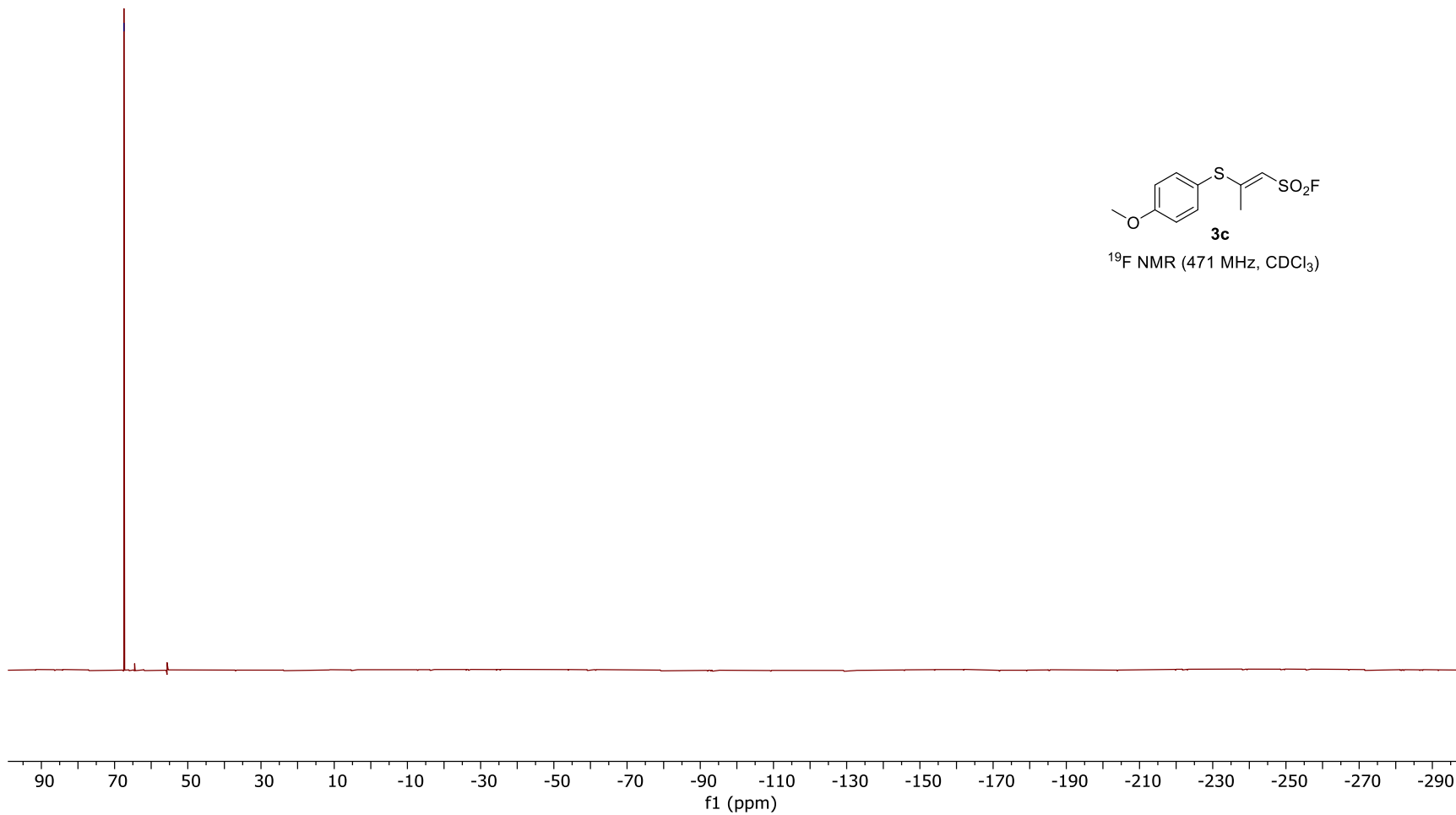


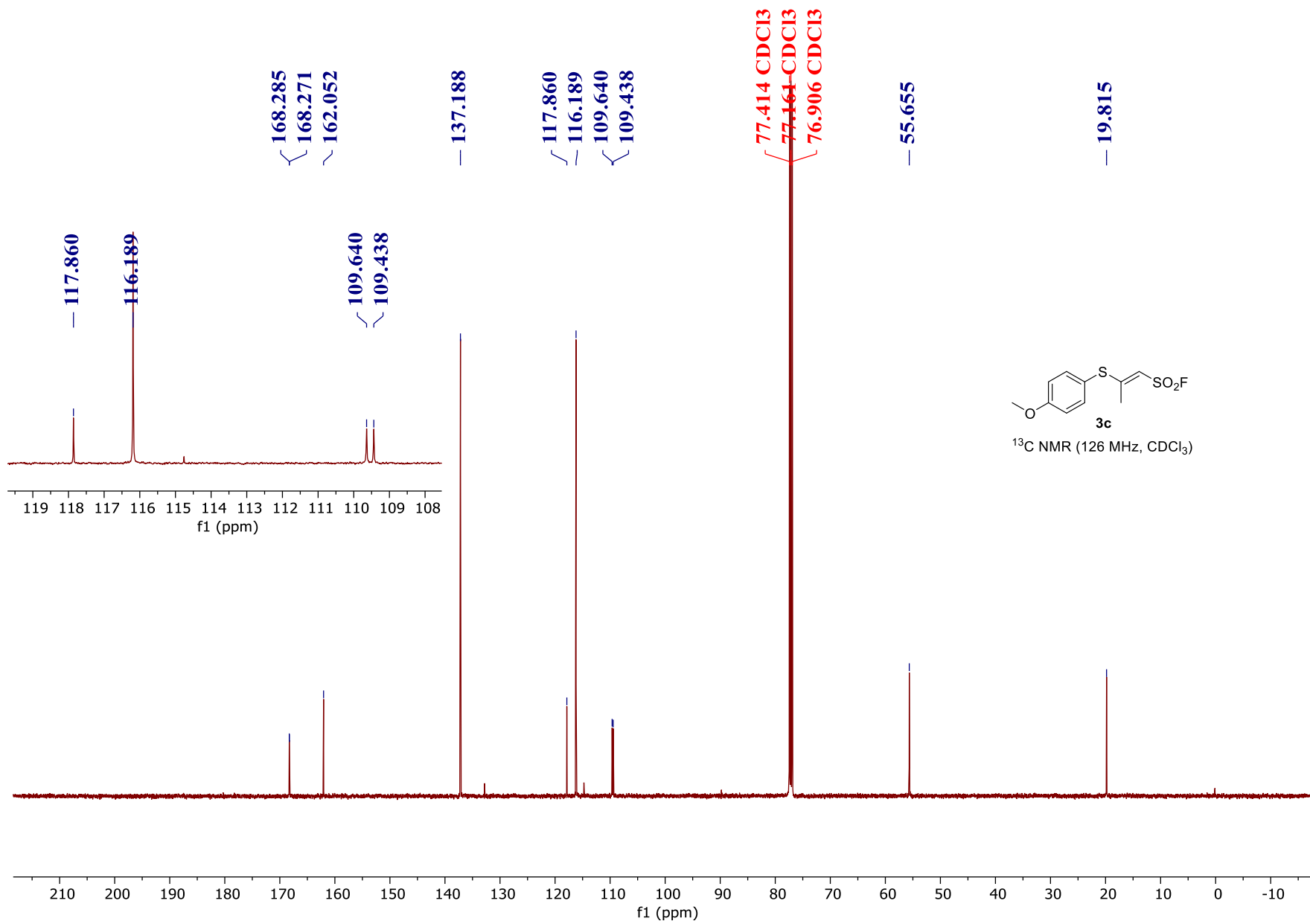


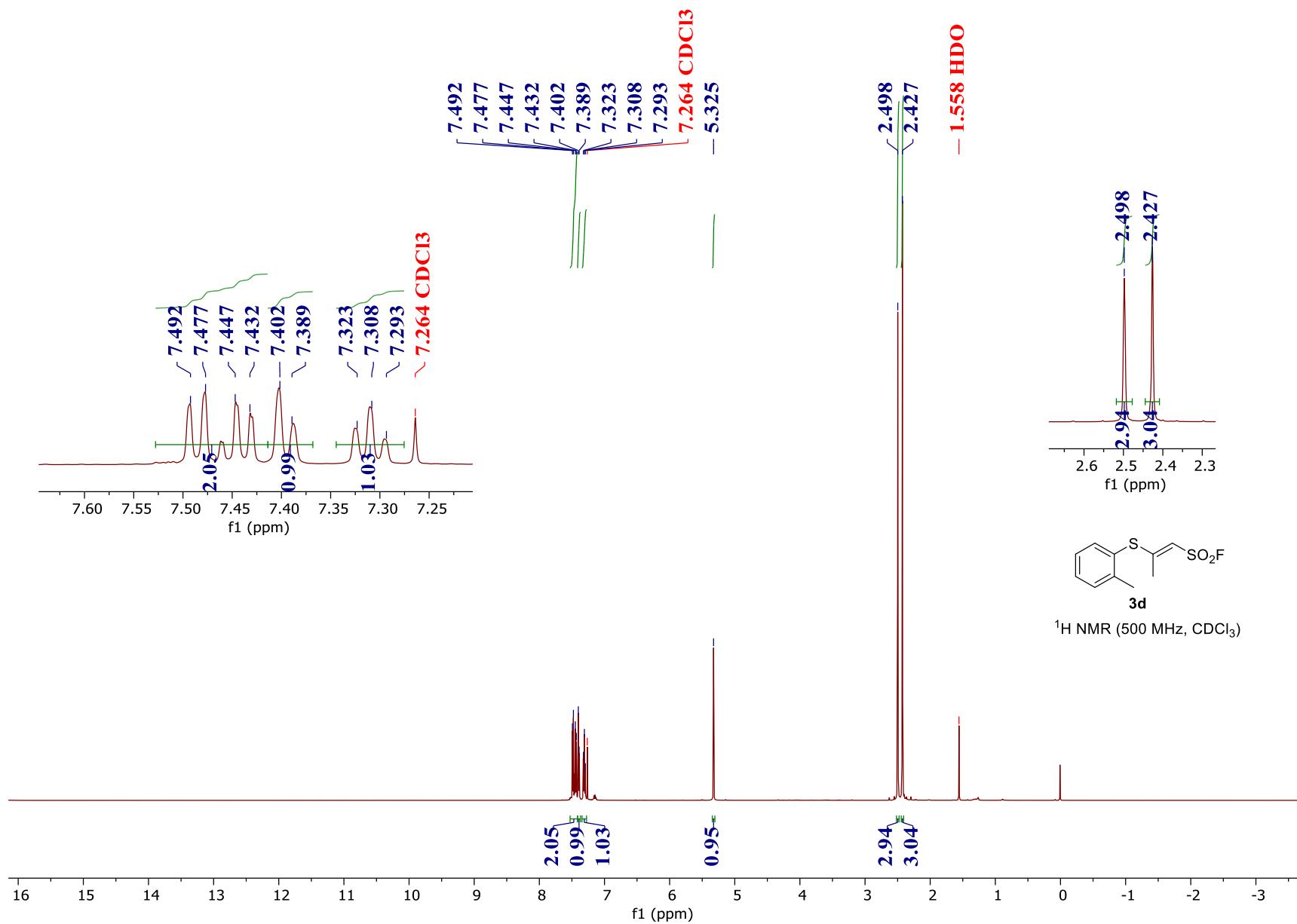


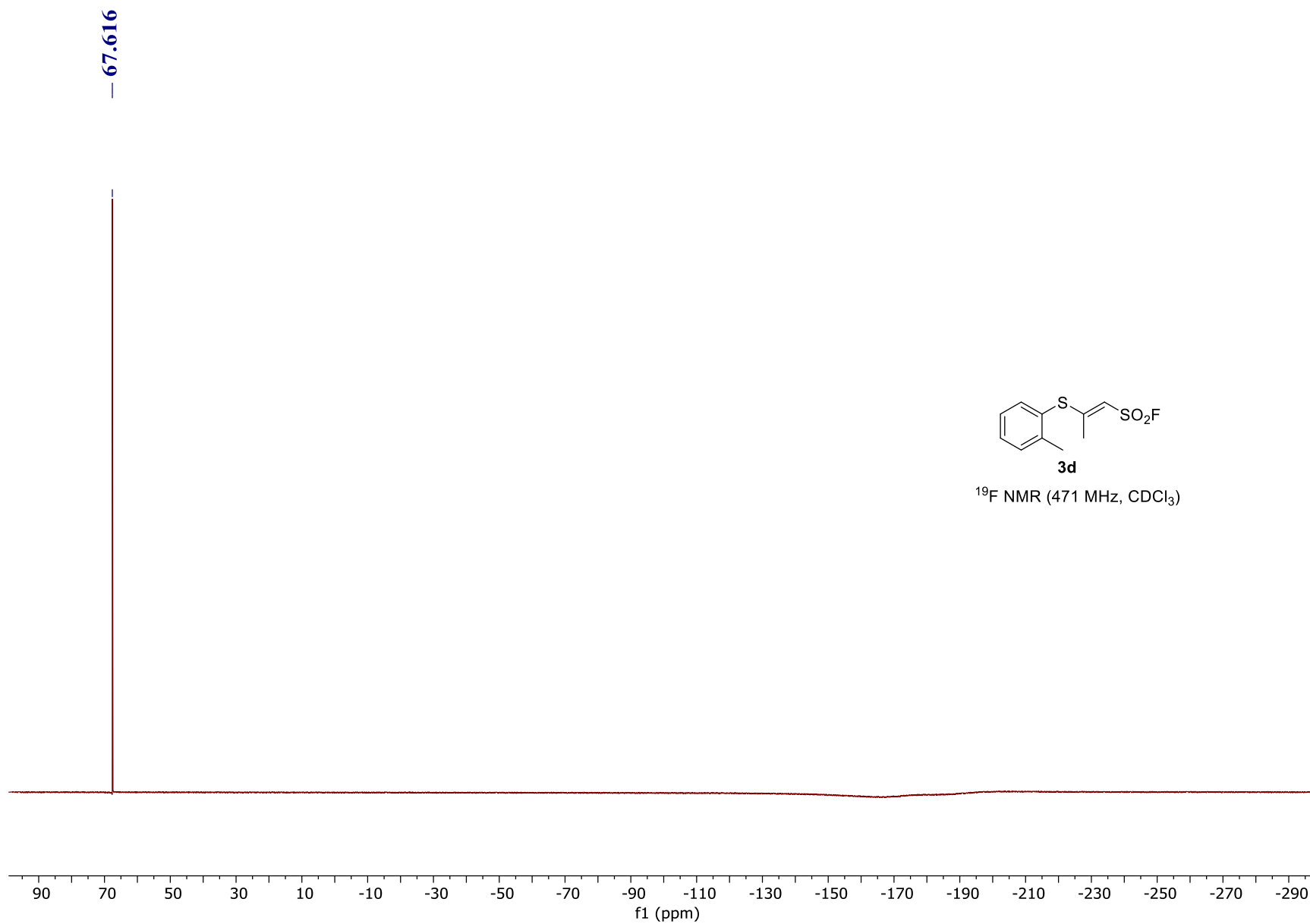


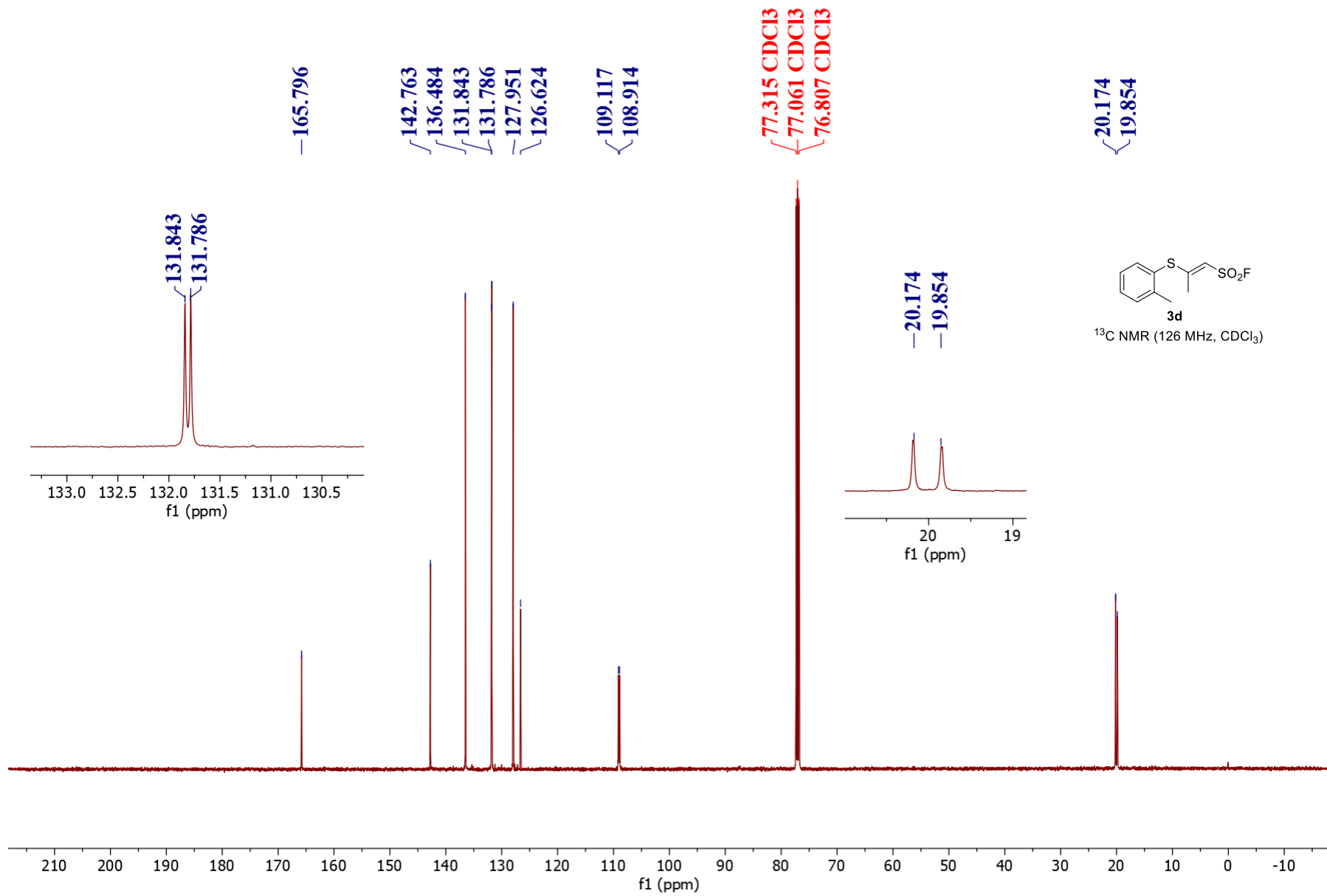
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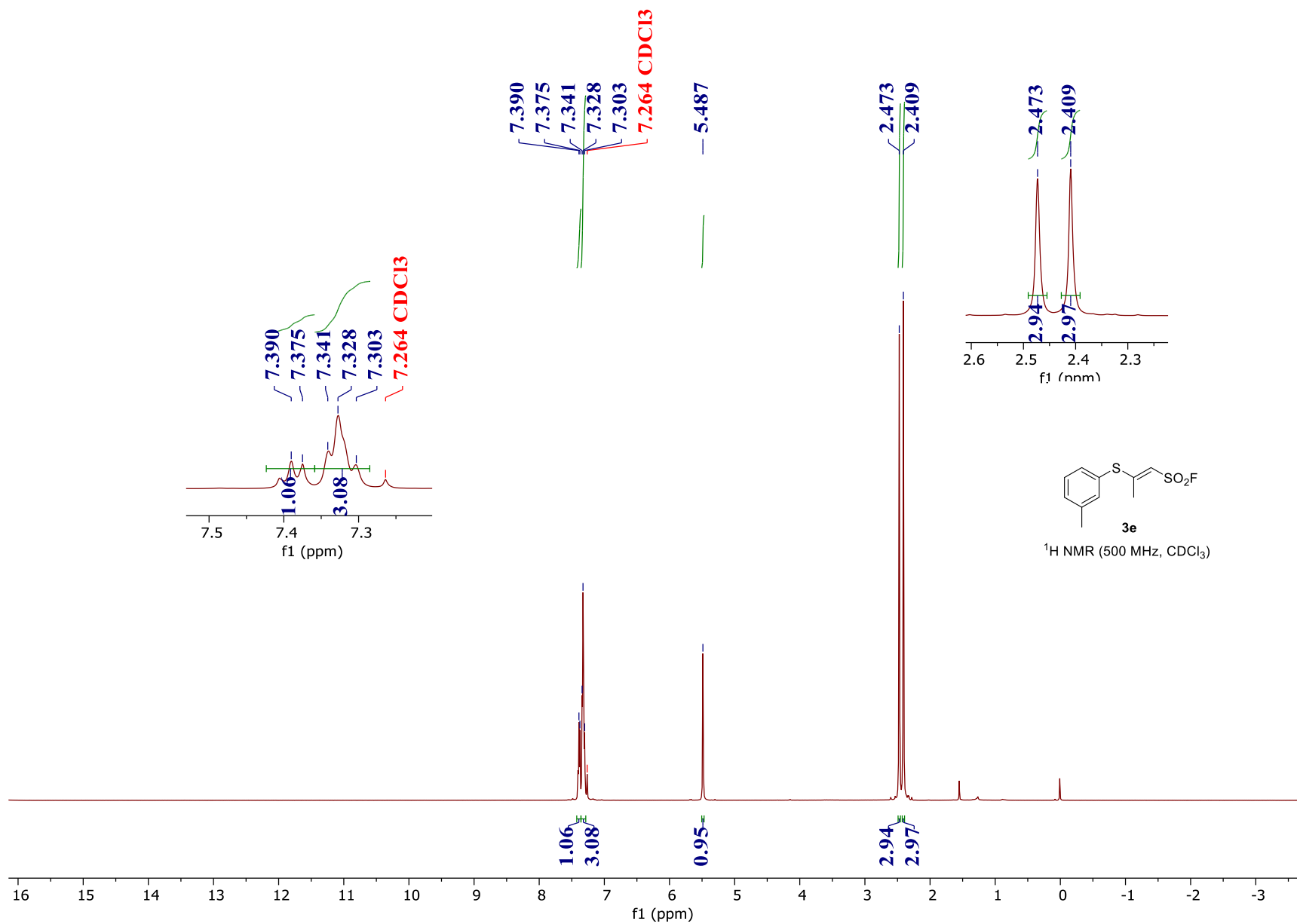




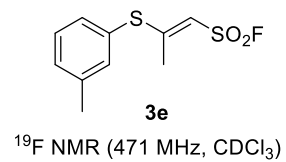
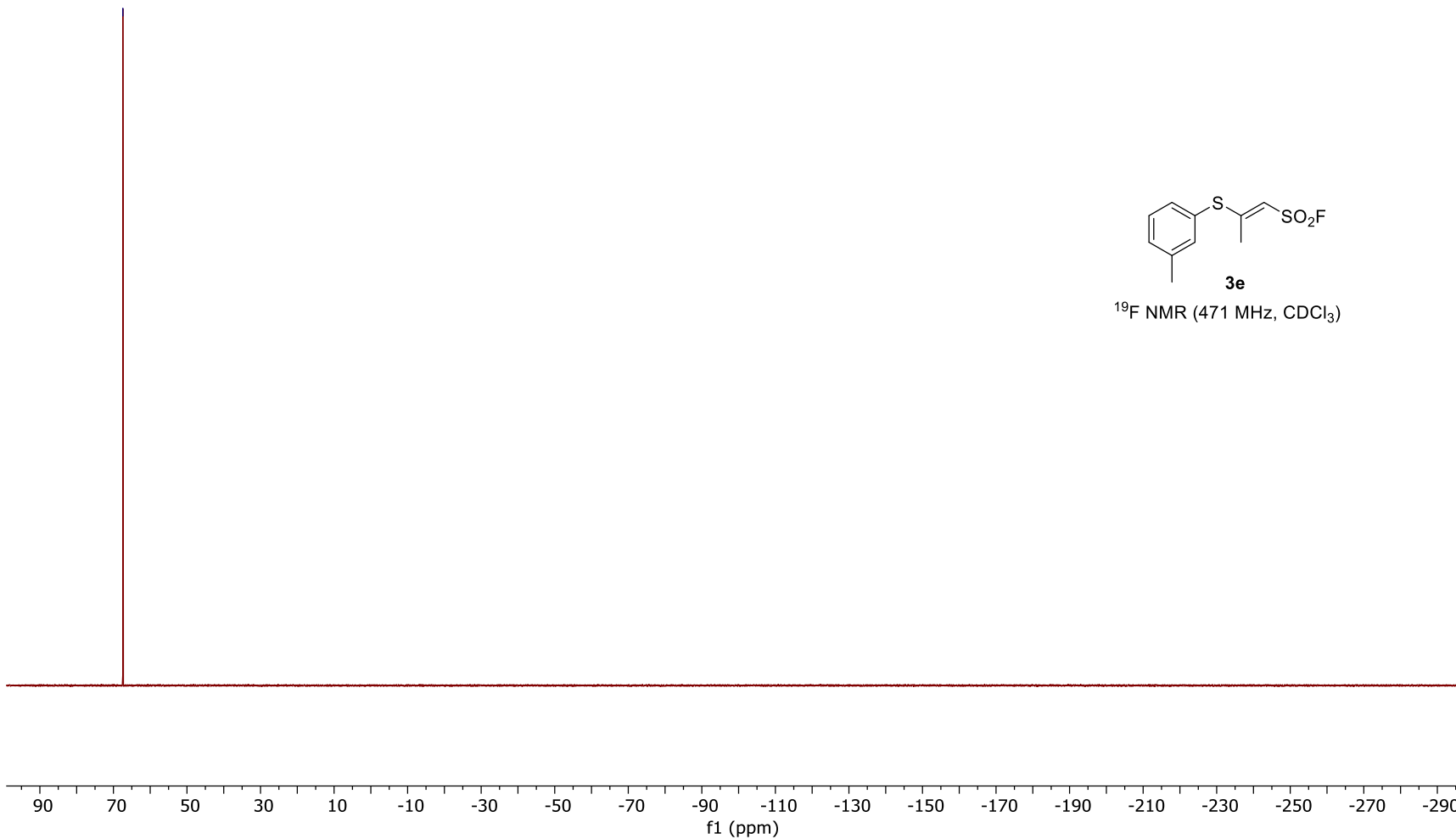


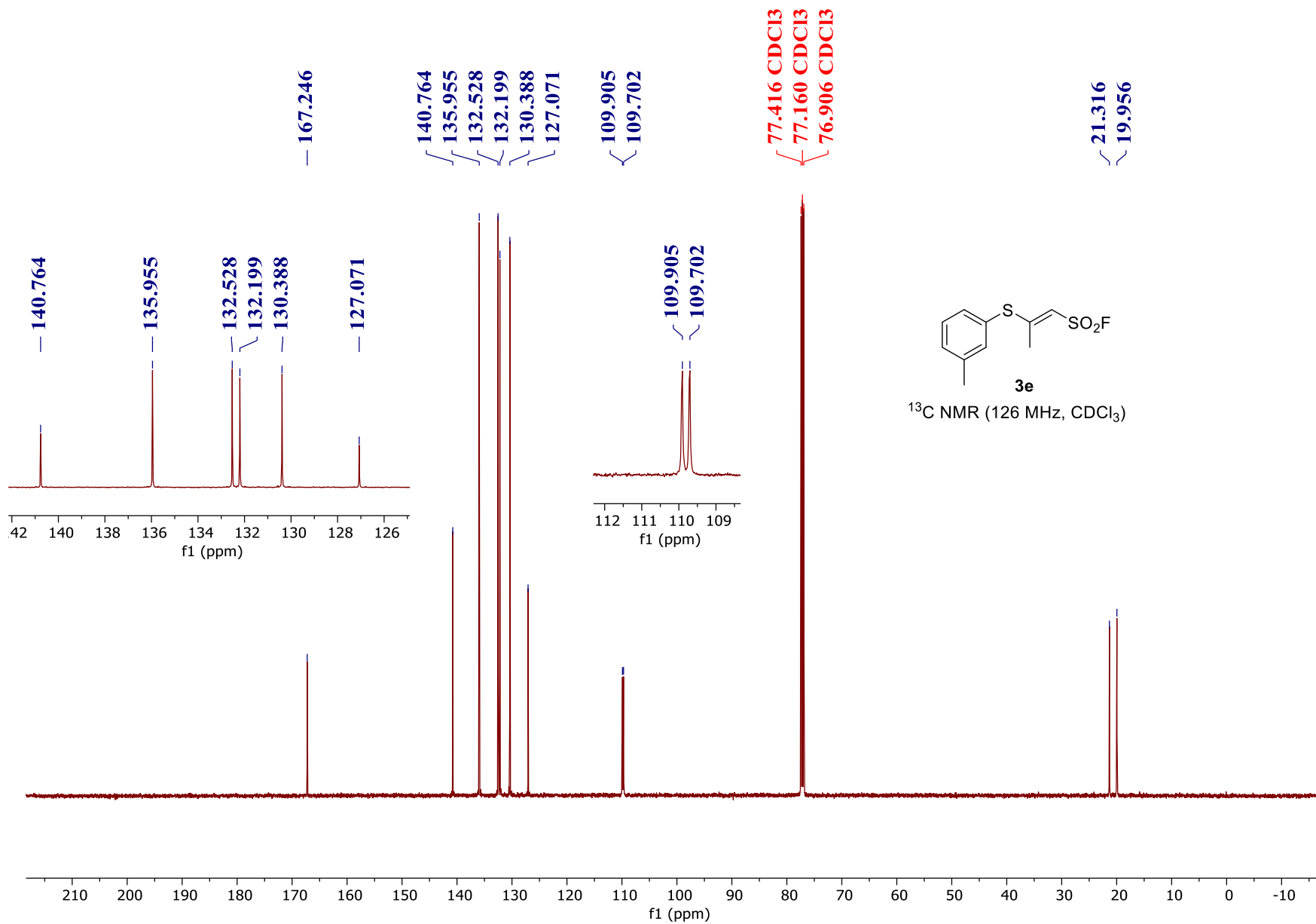




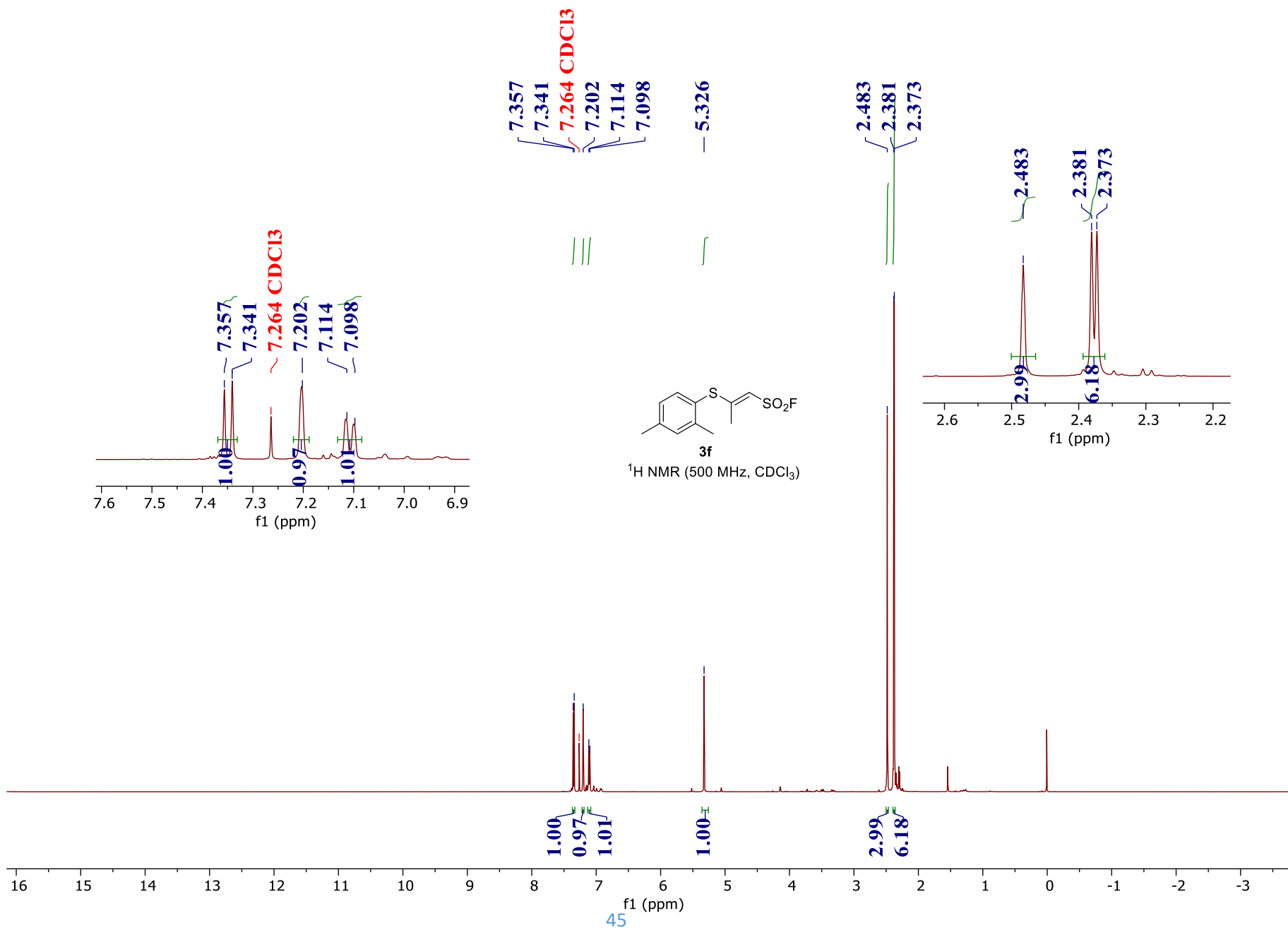


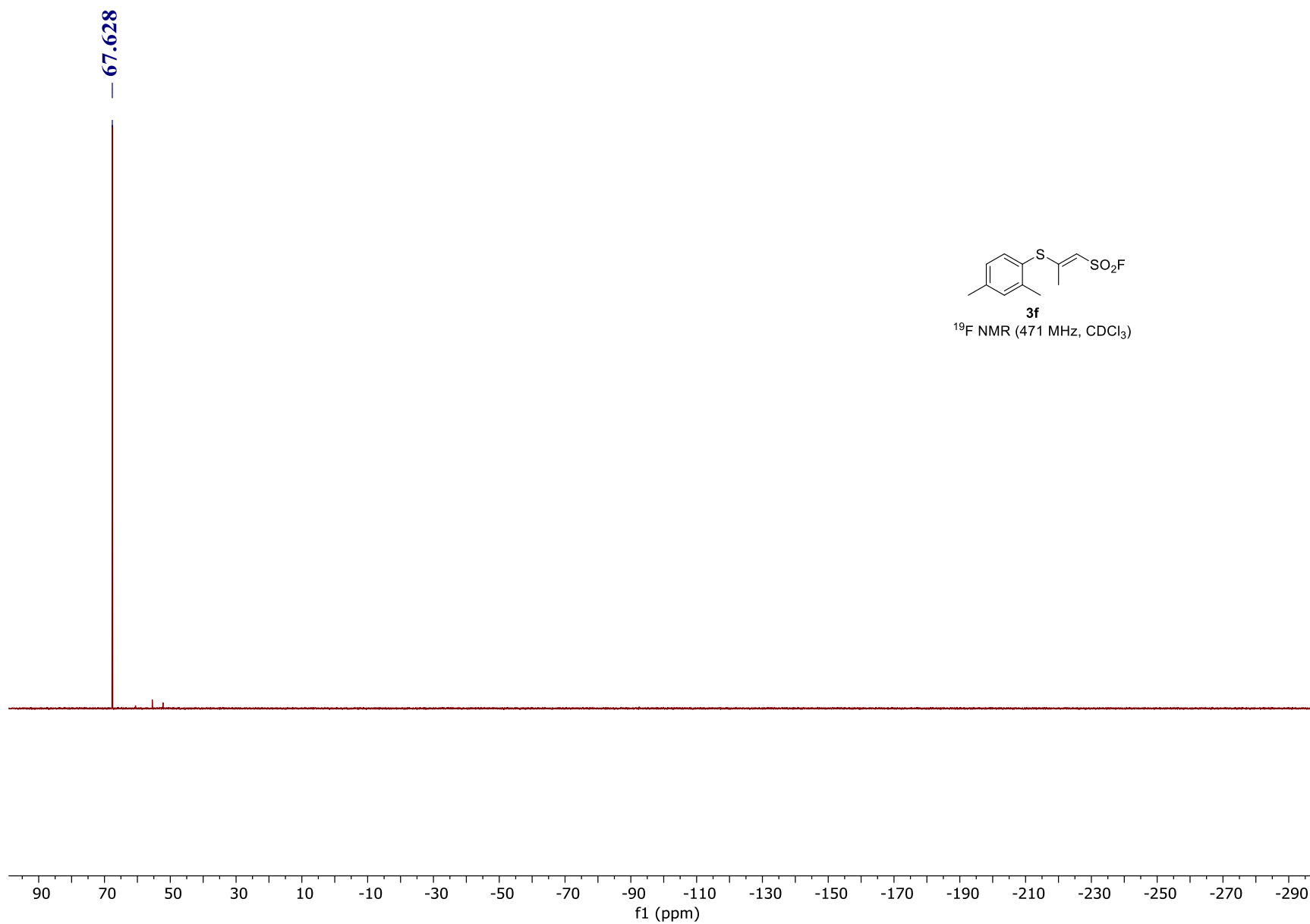
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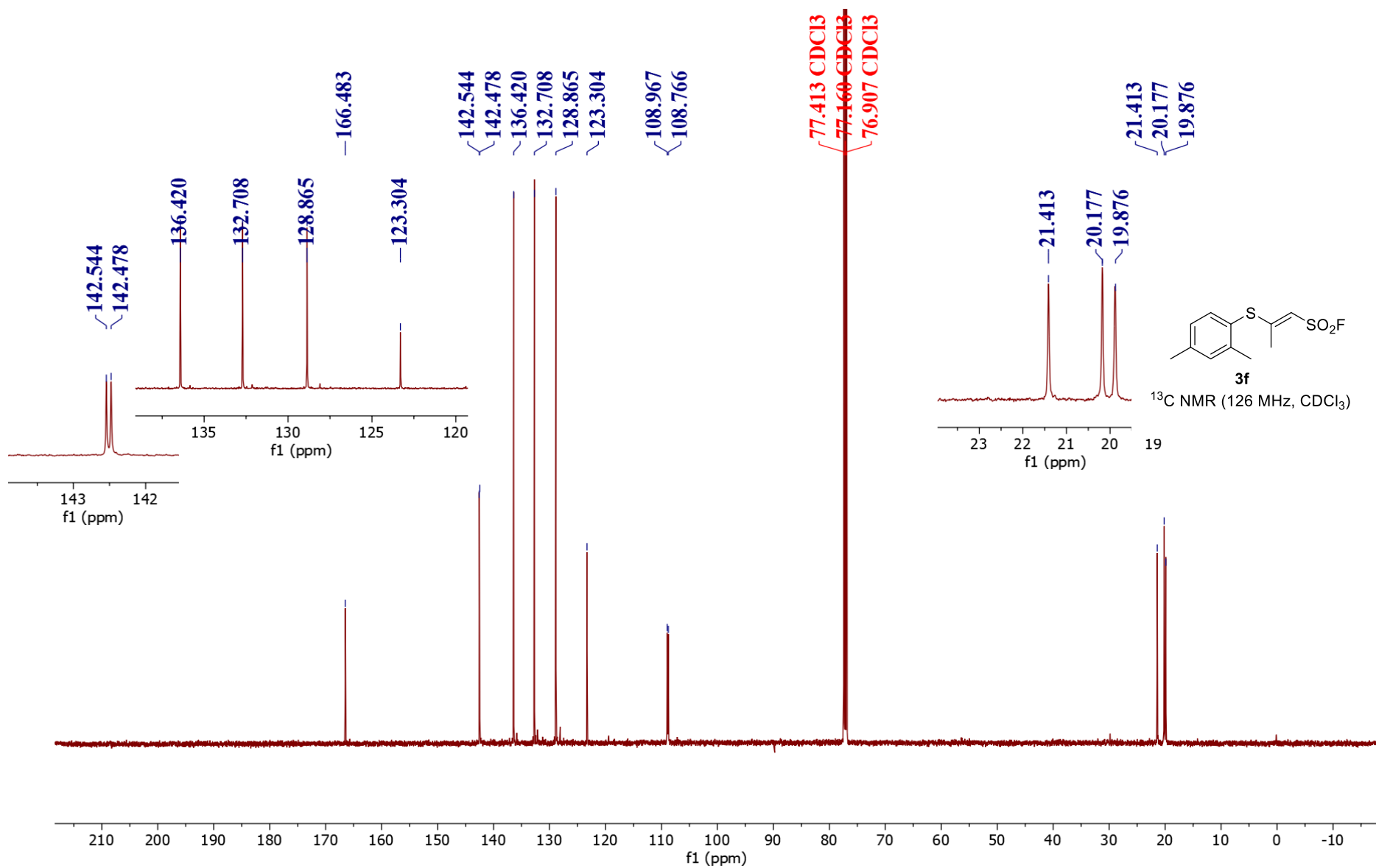


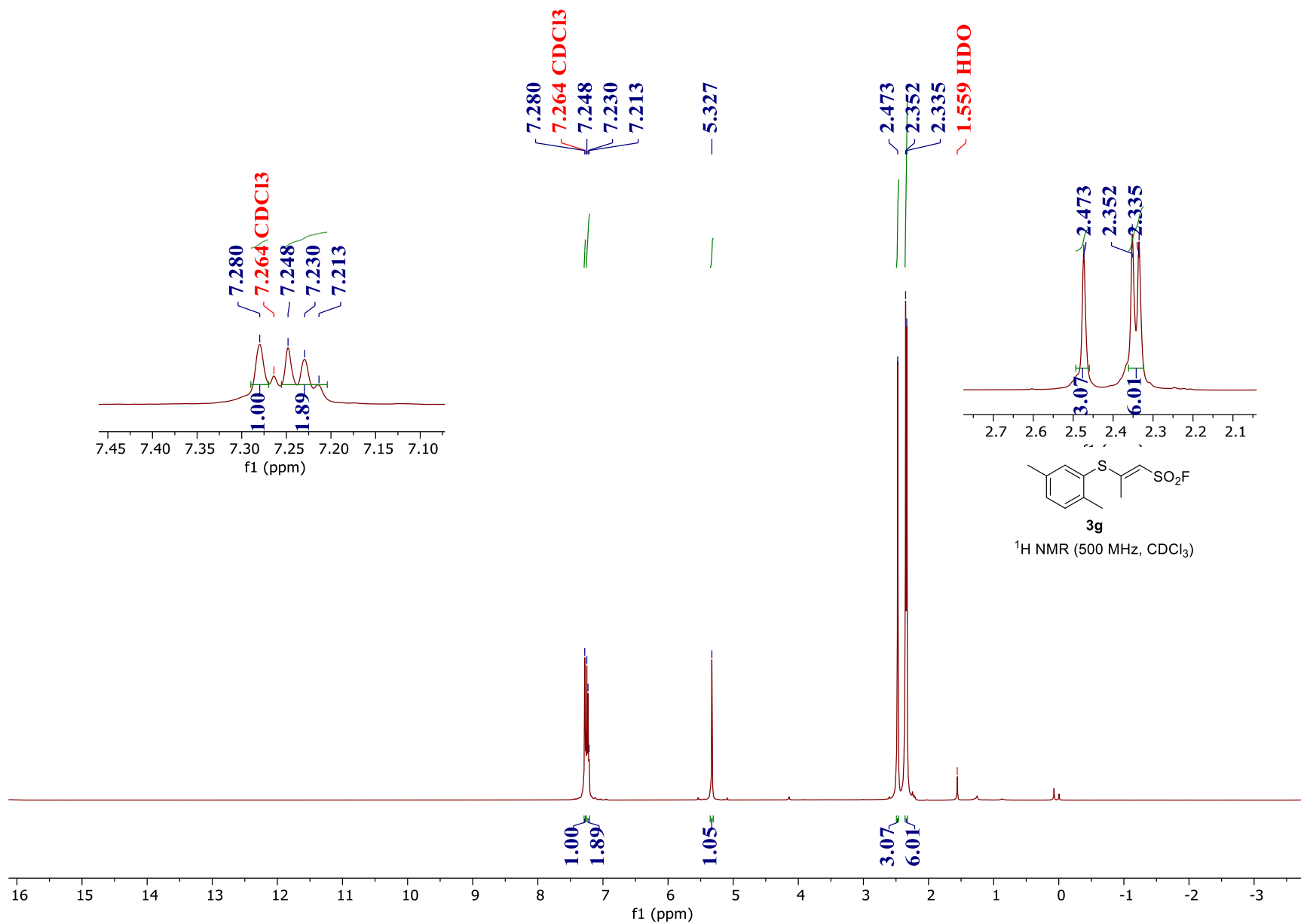




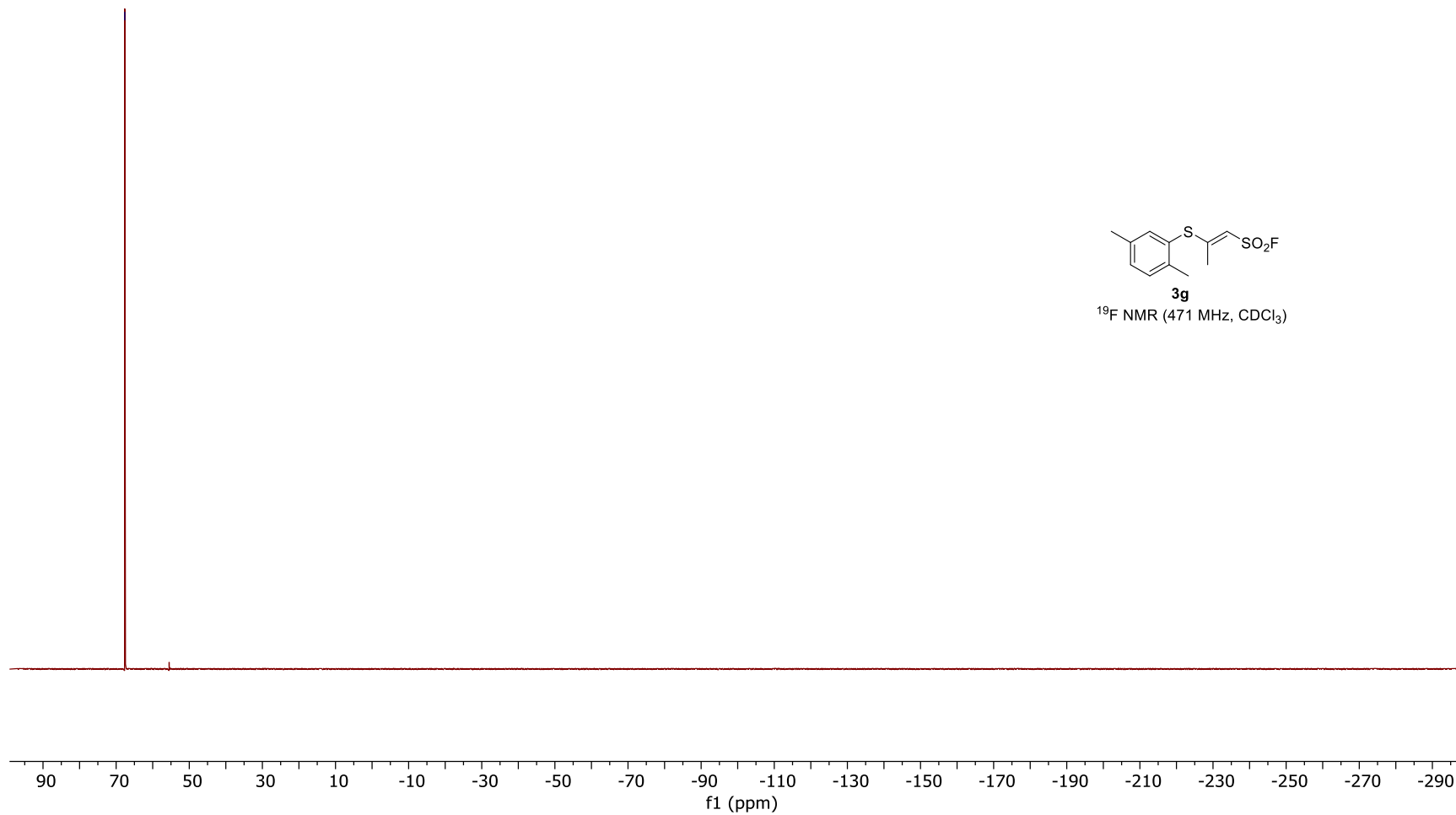


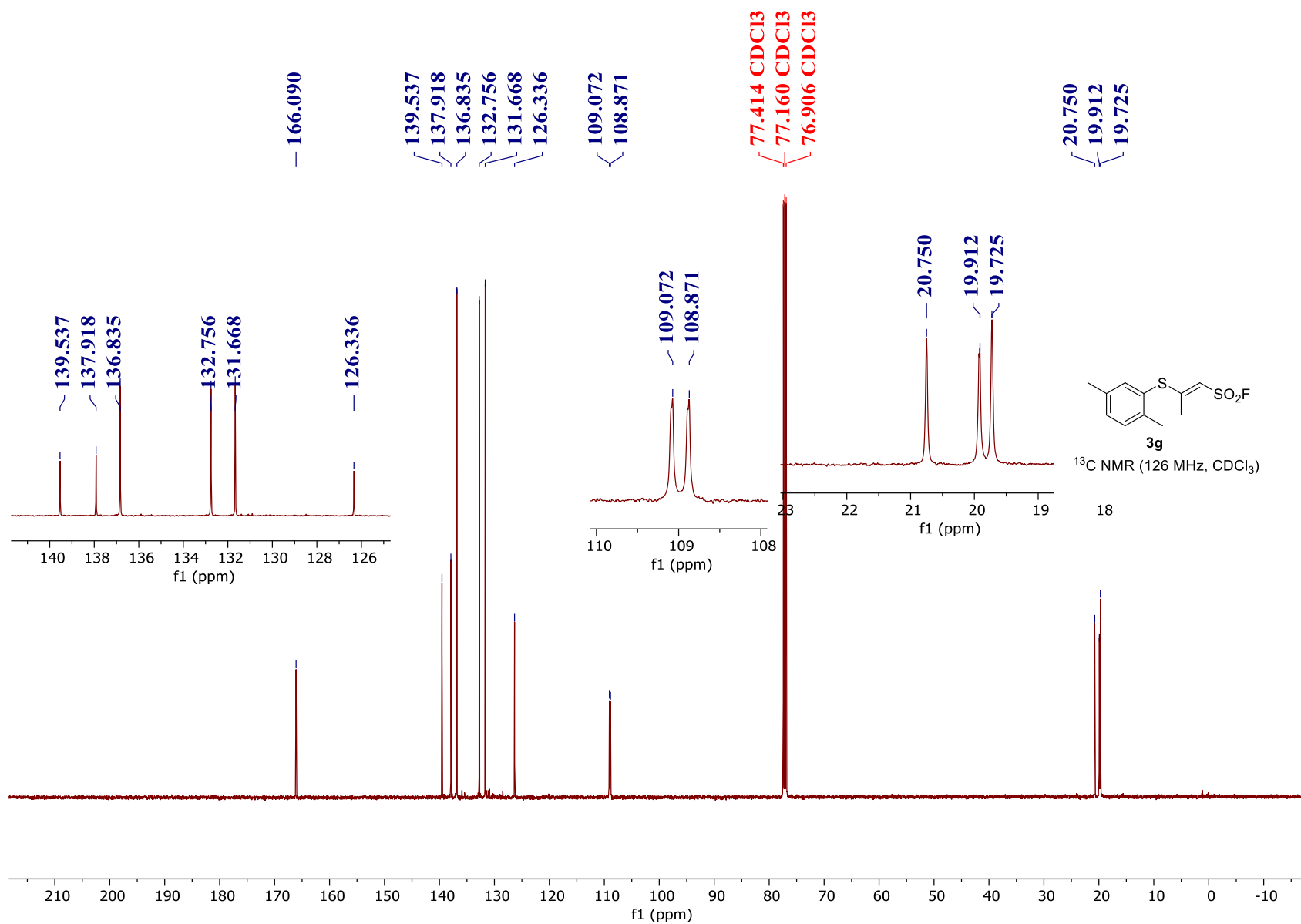


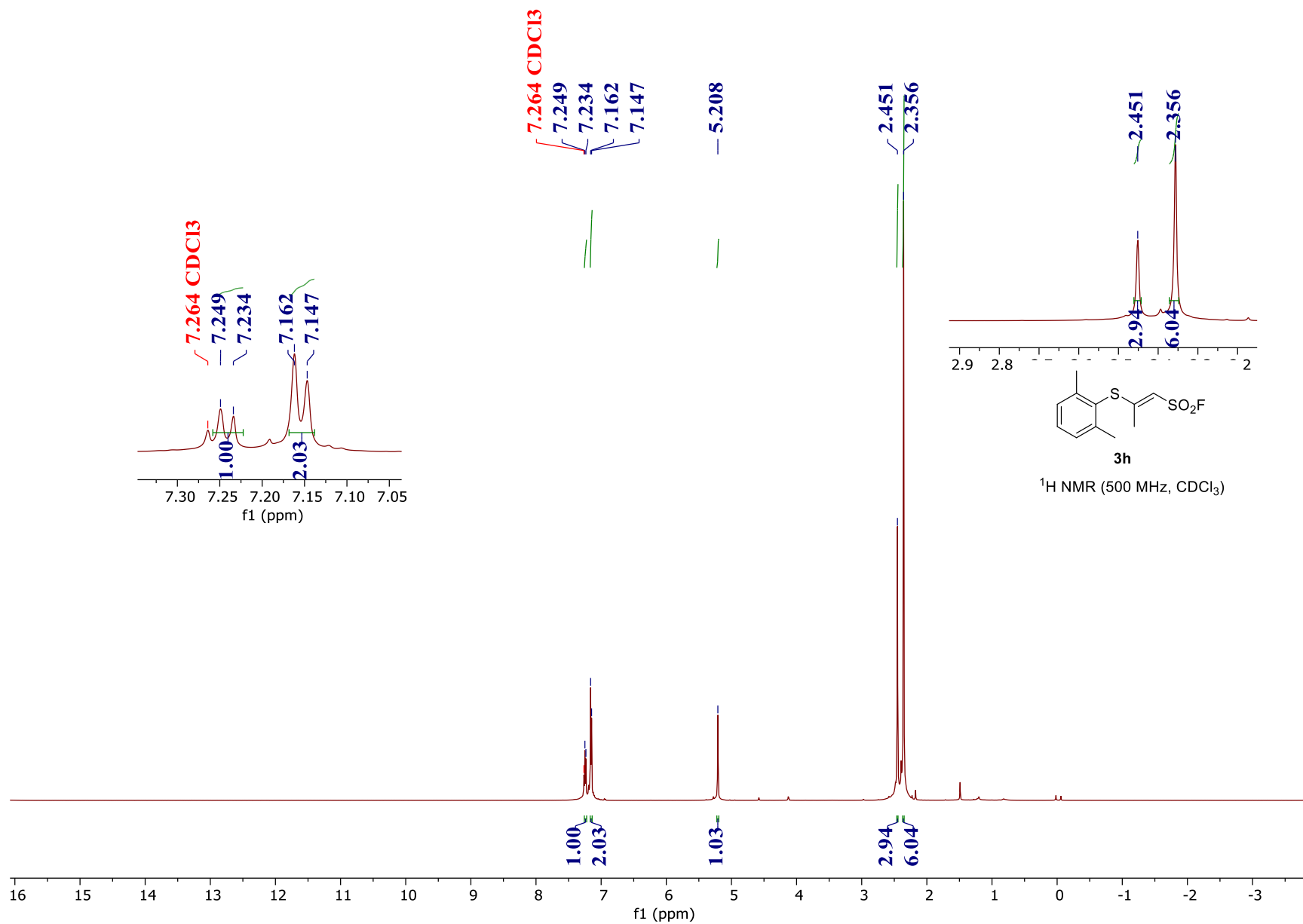




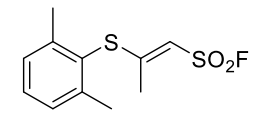
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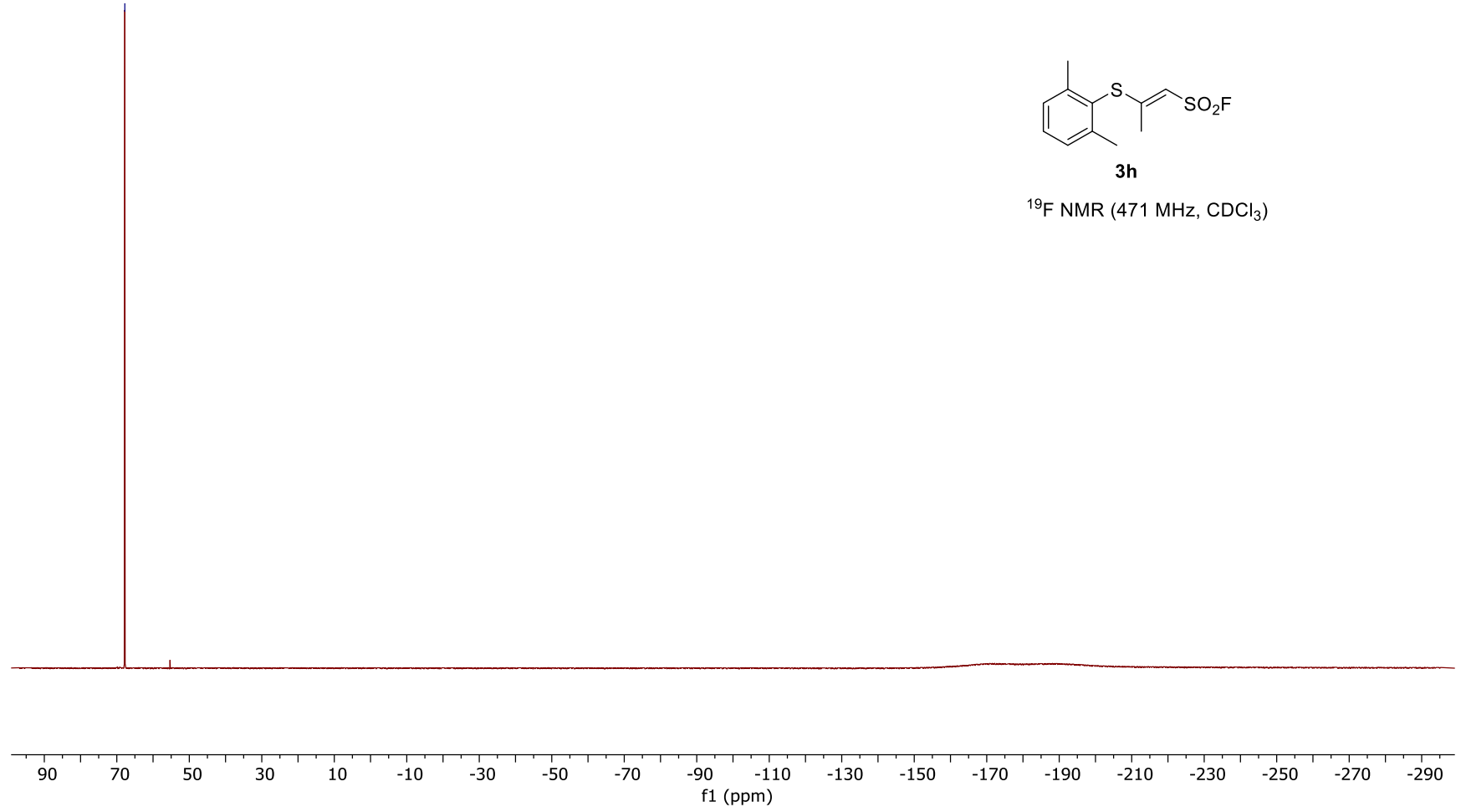


— 67.786

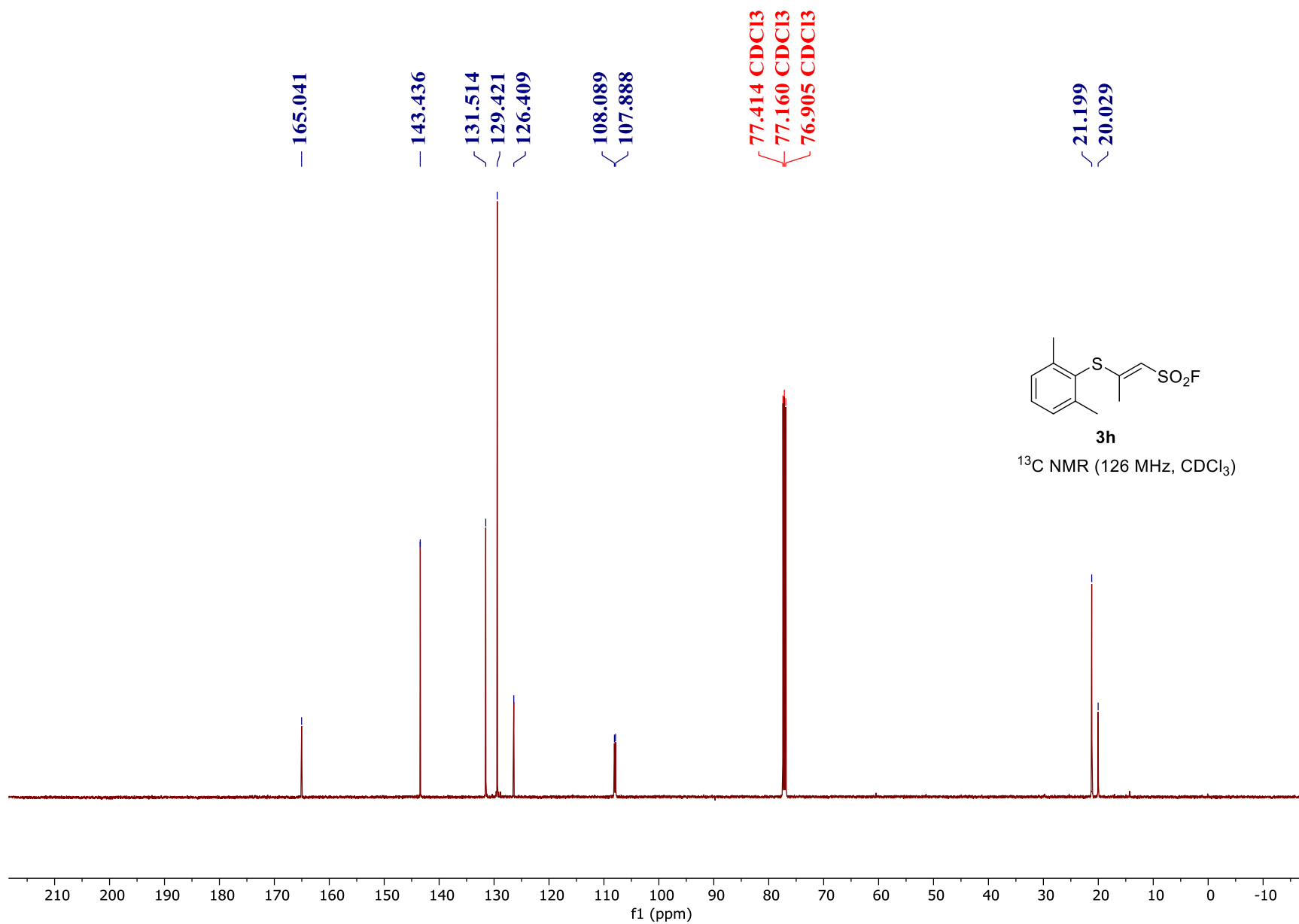


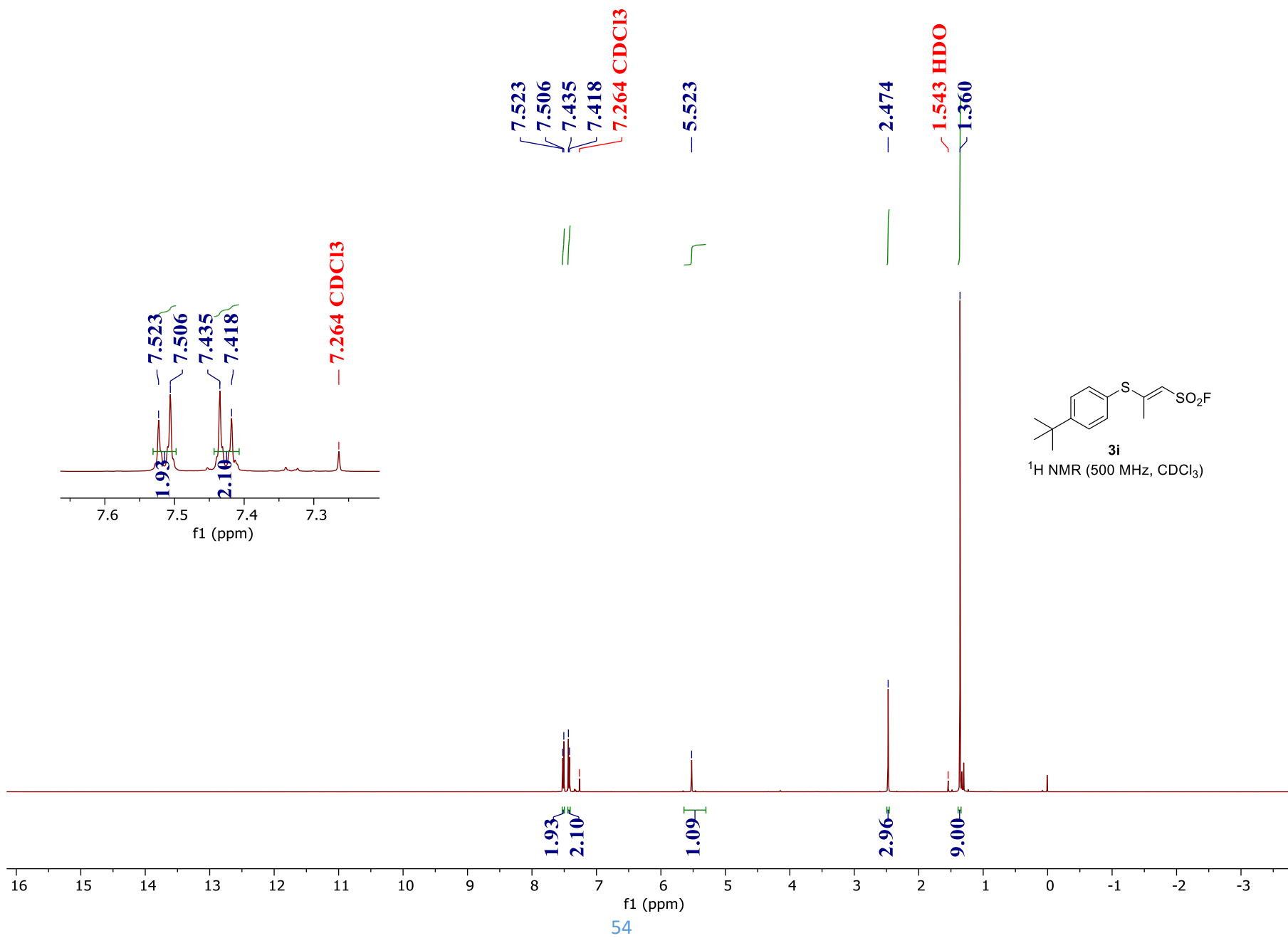
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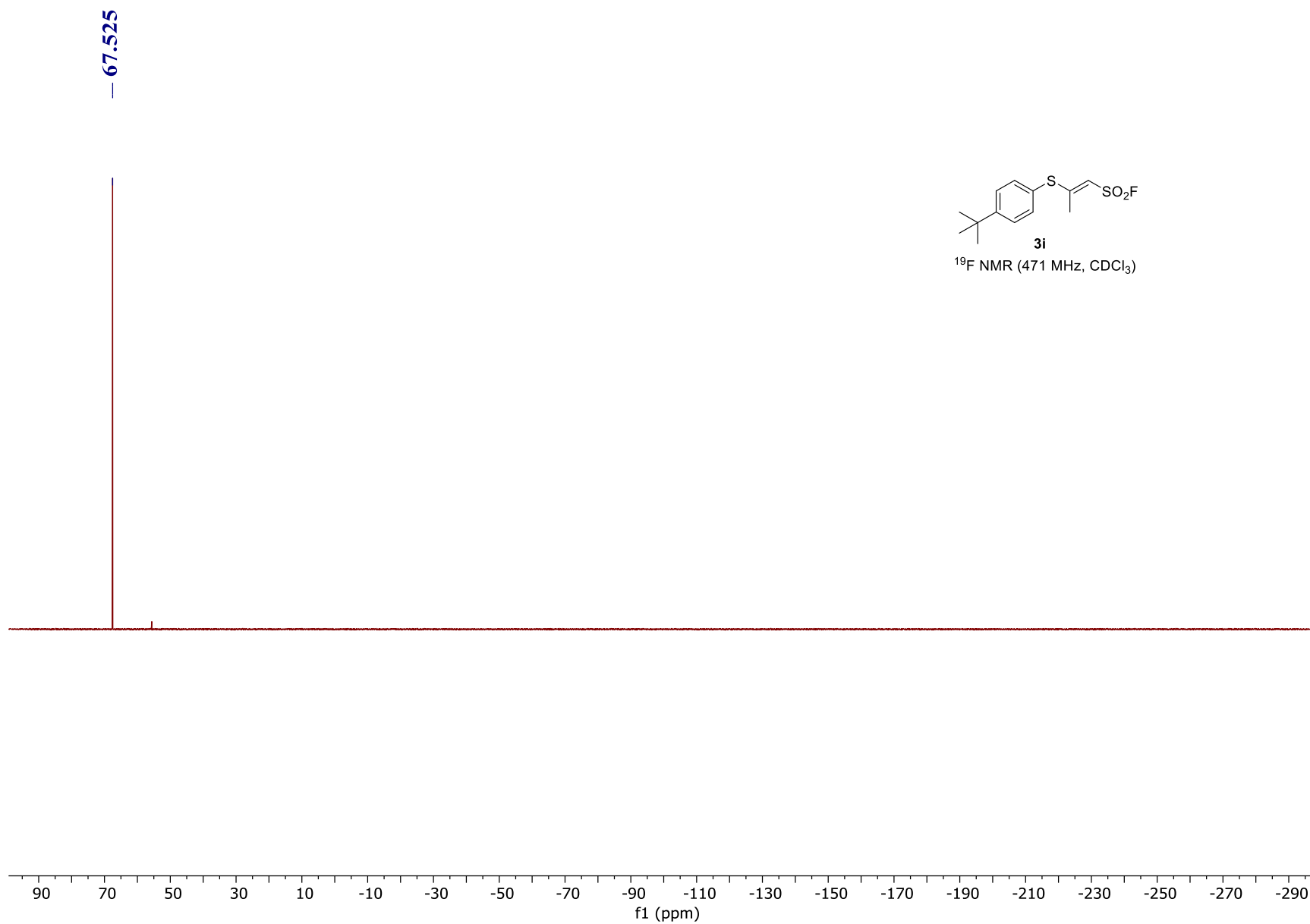
<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)

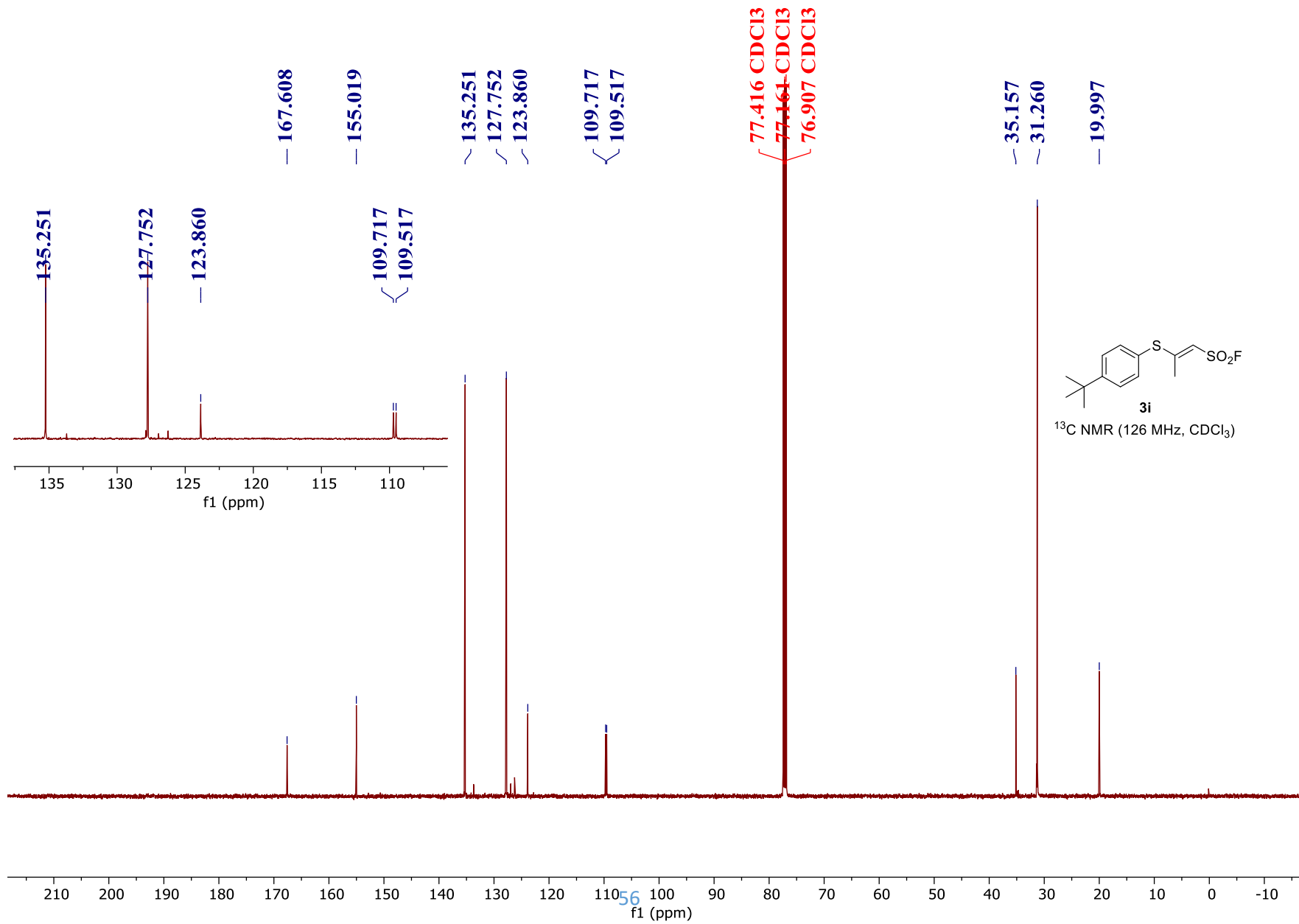


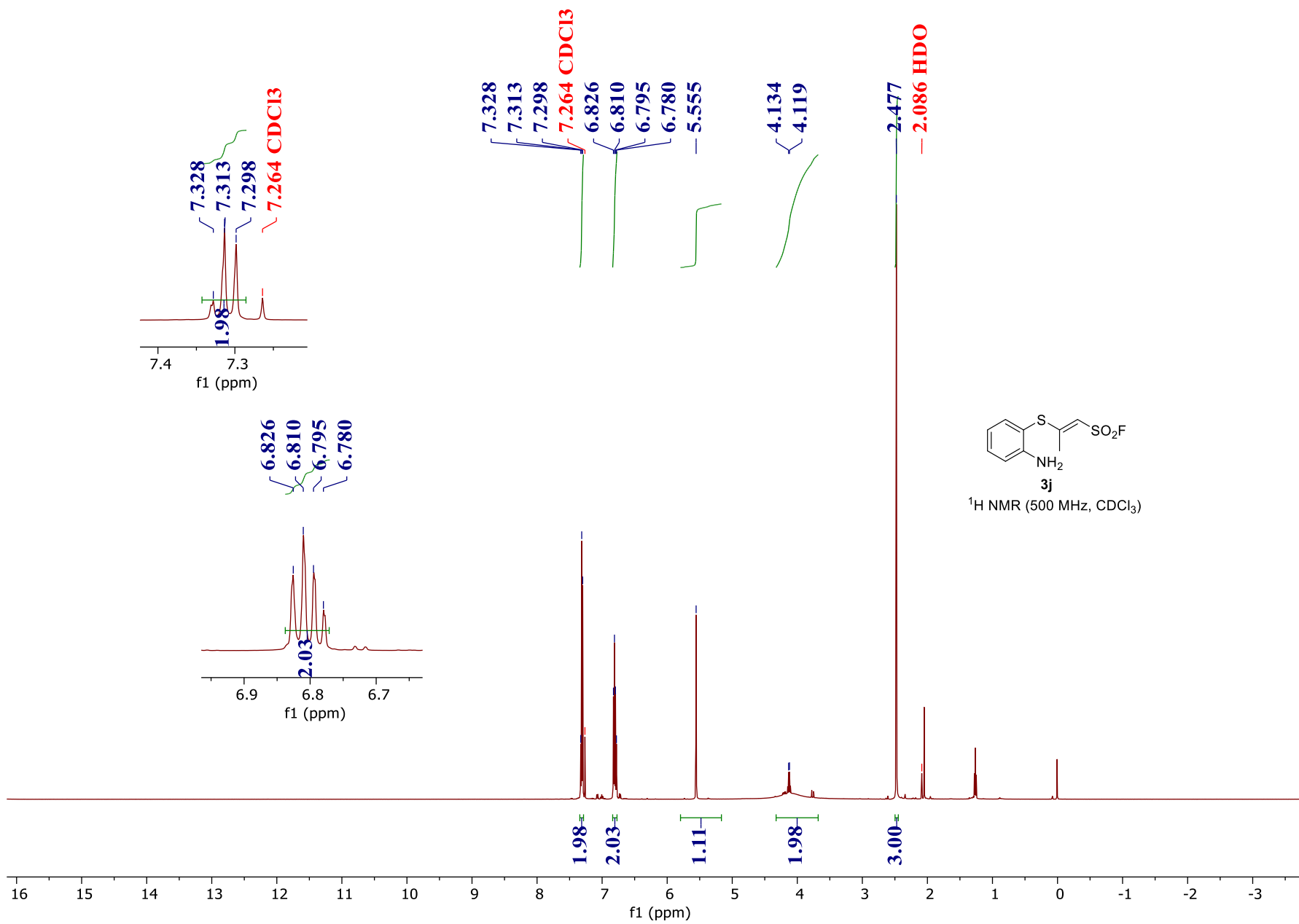


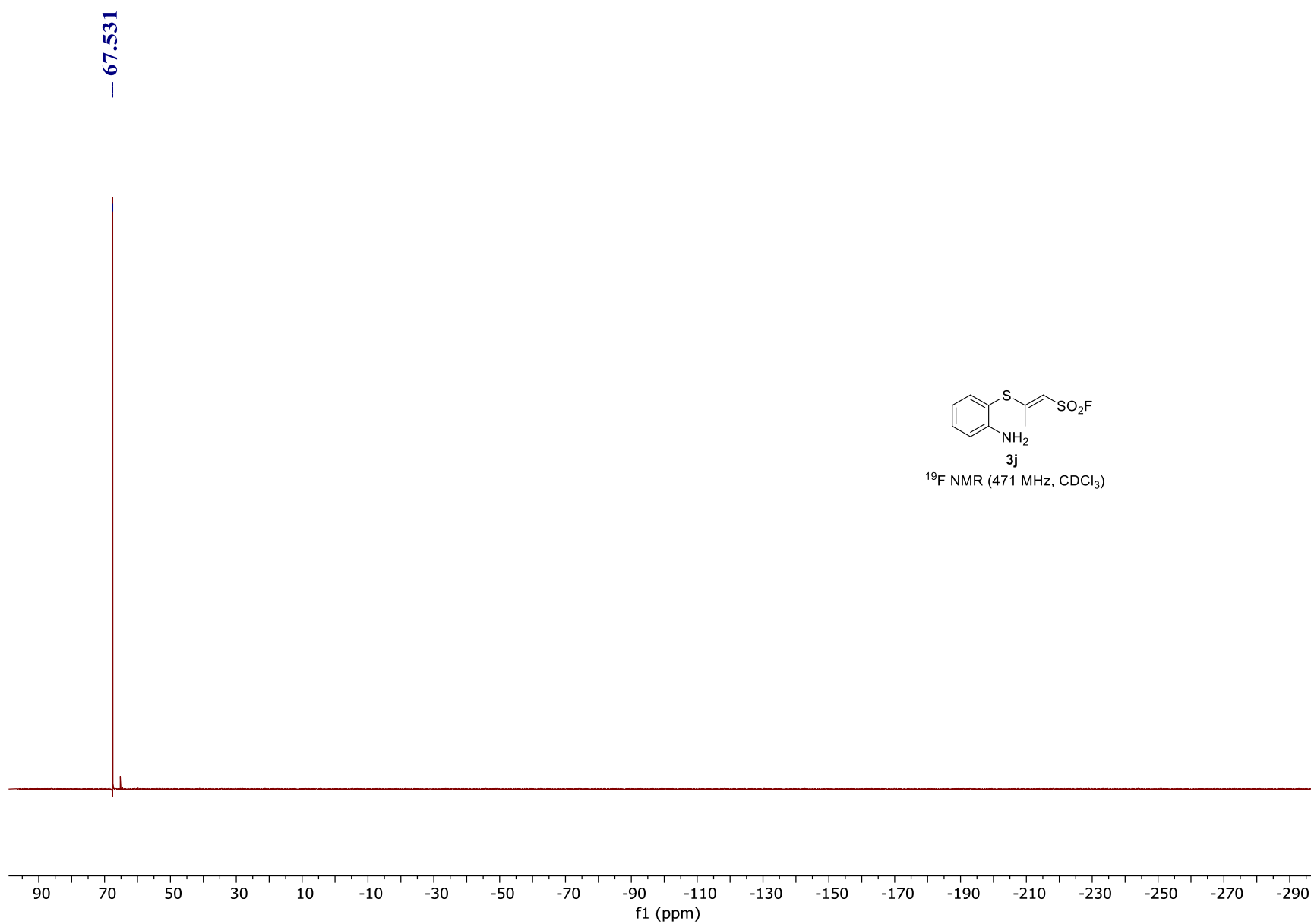


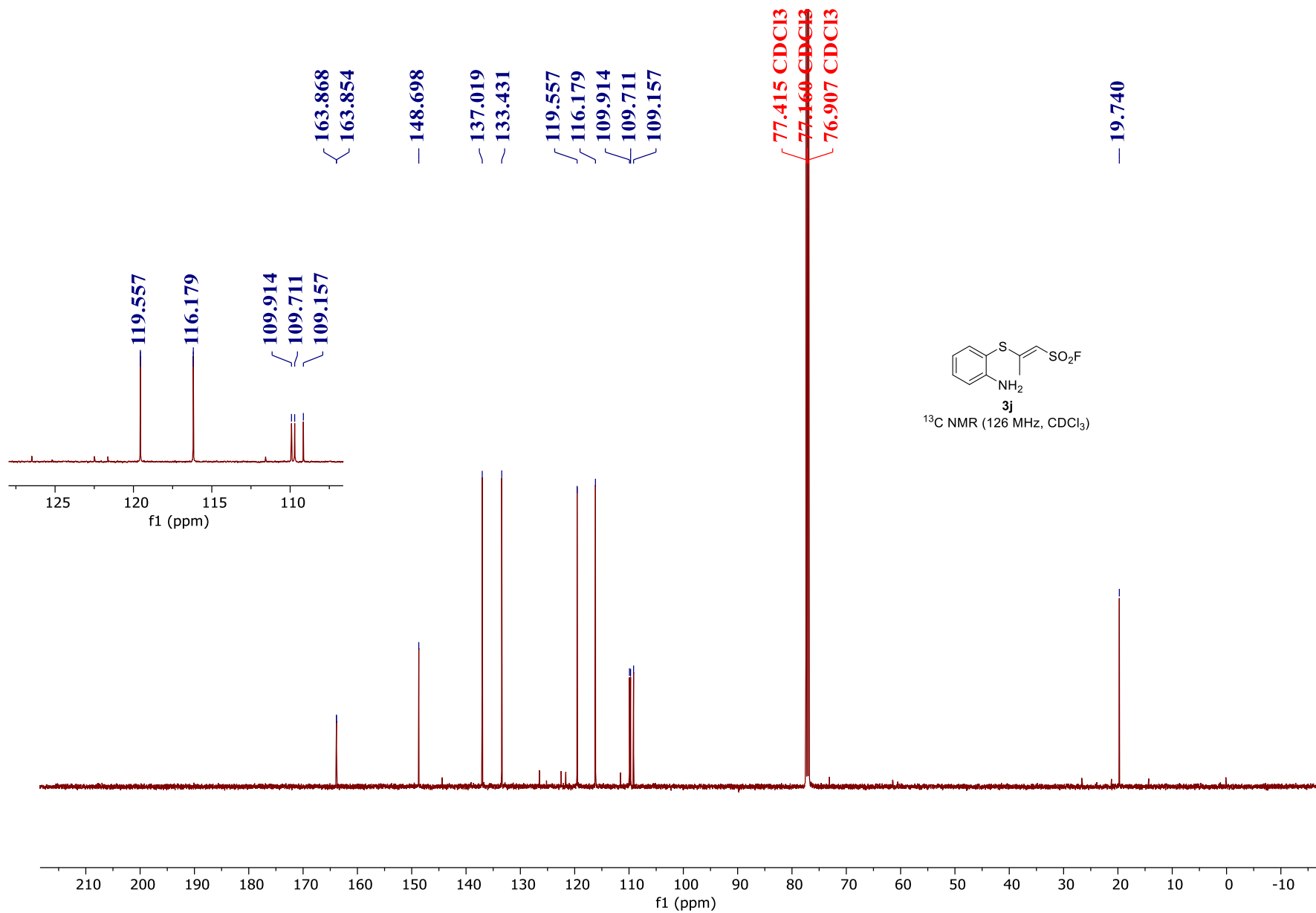


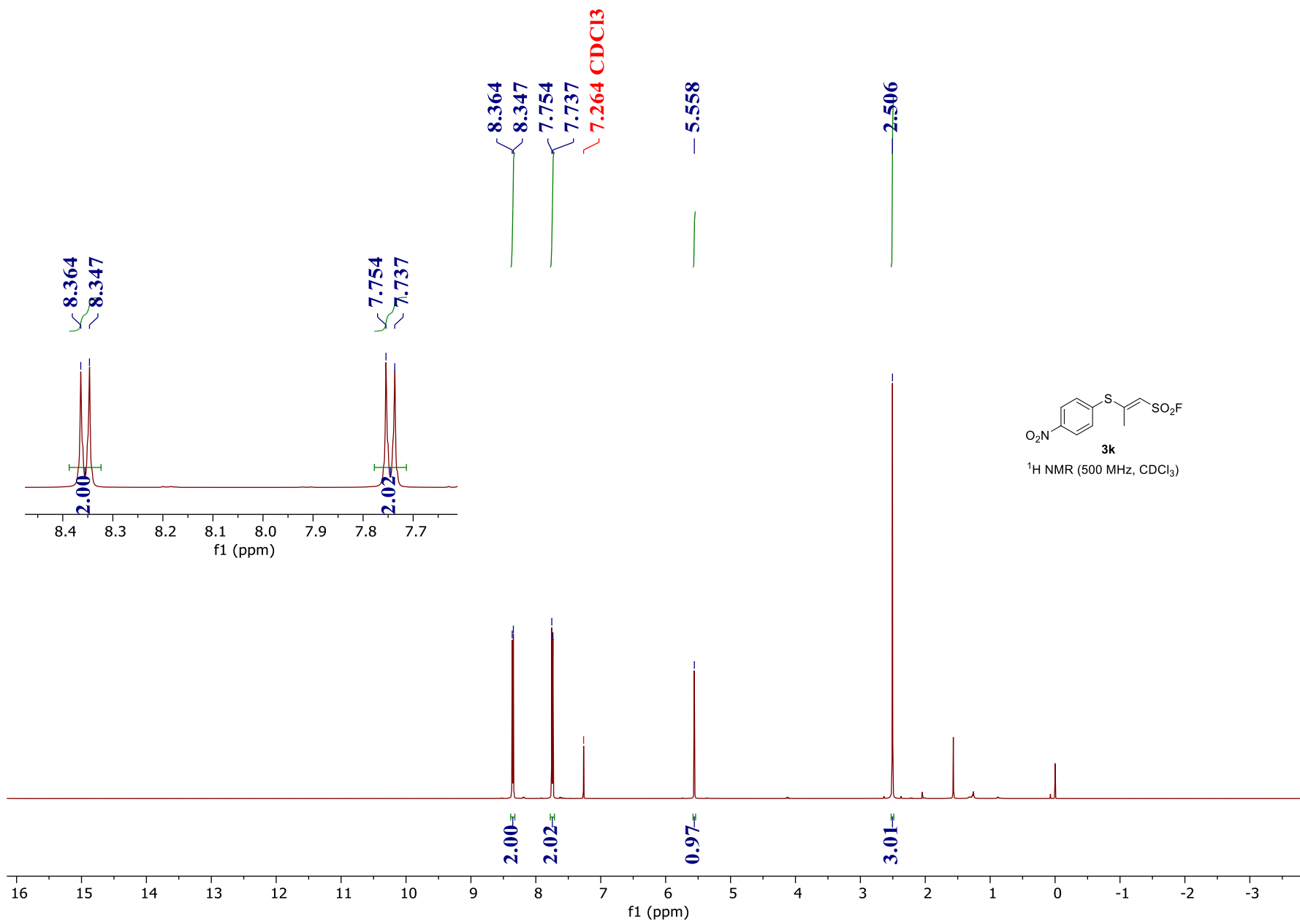






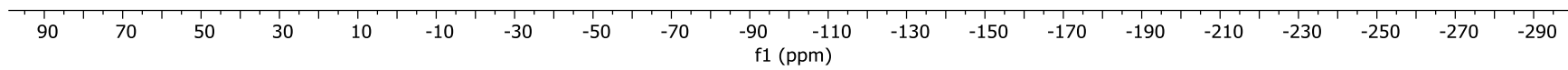
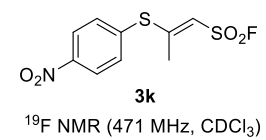


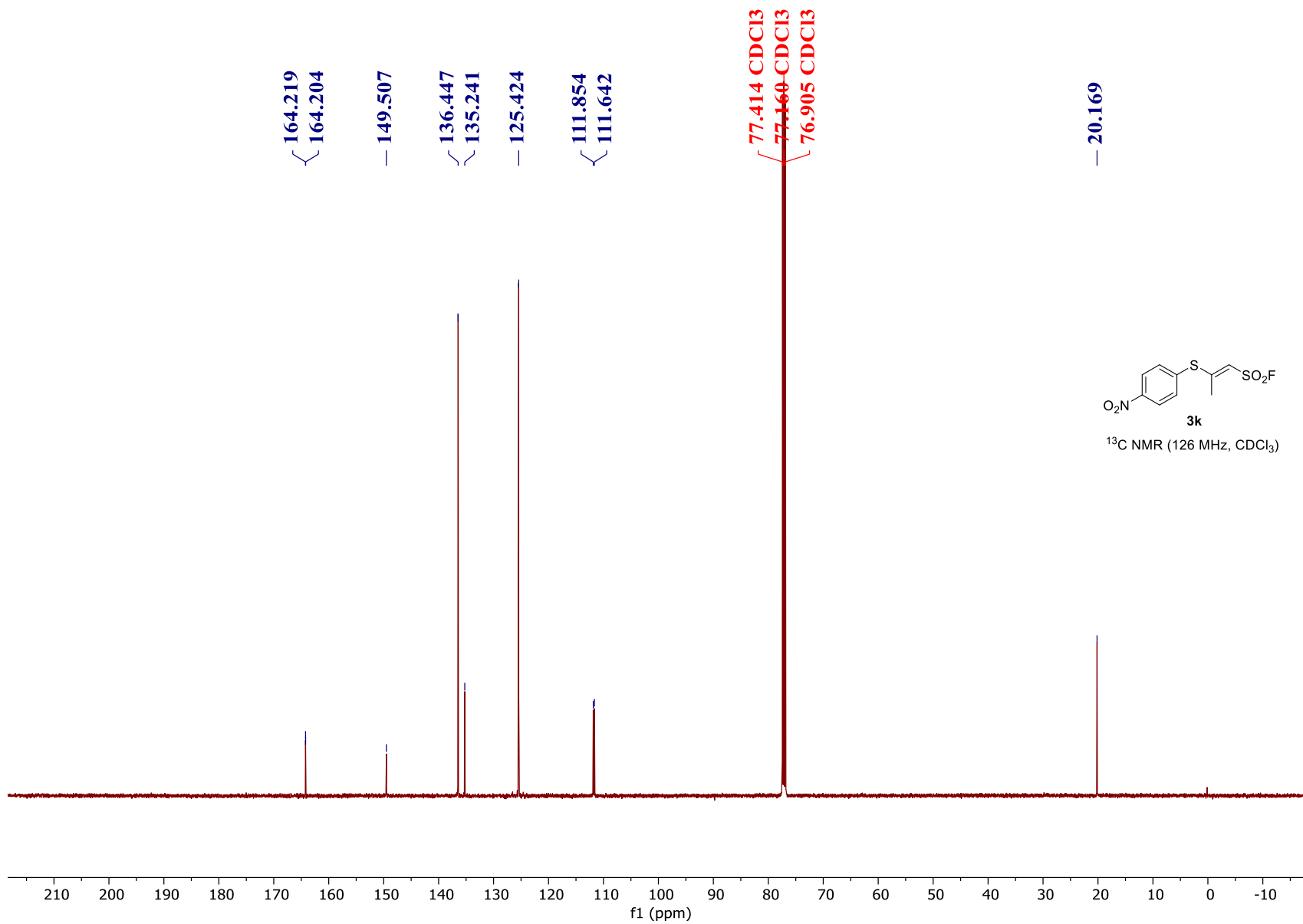


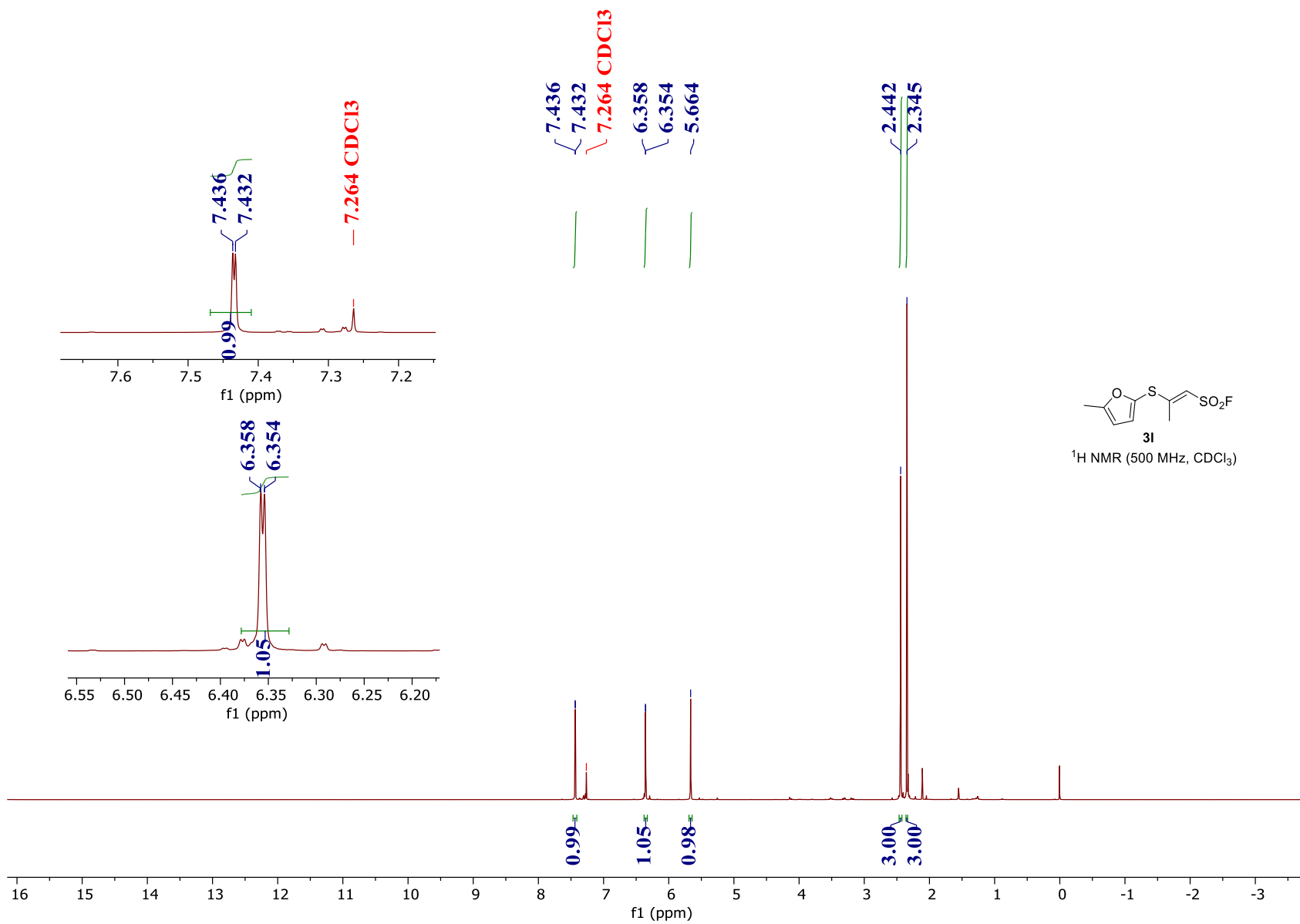




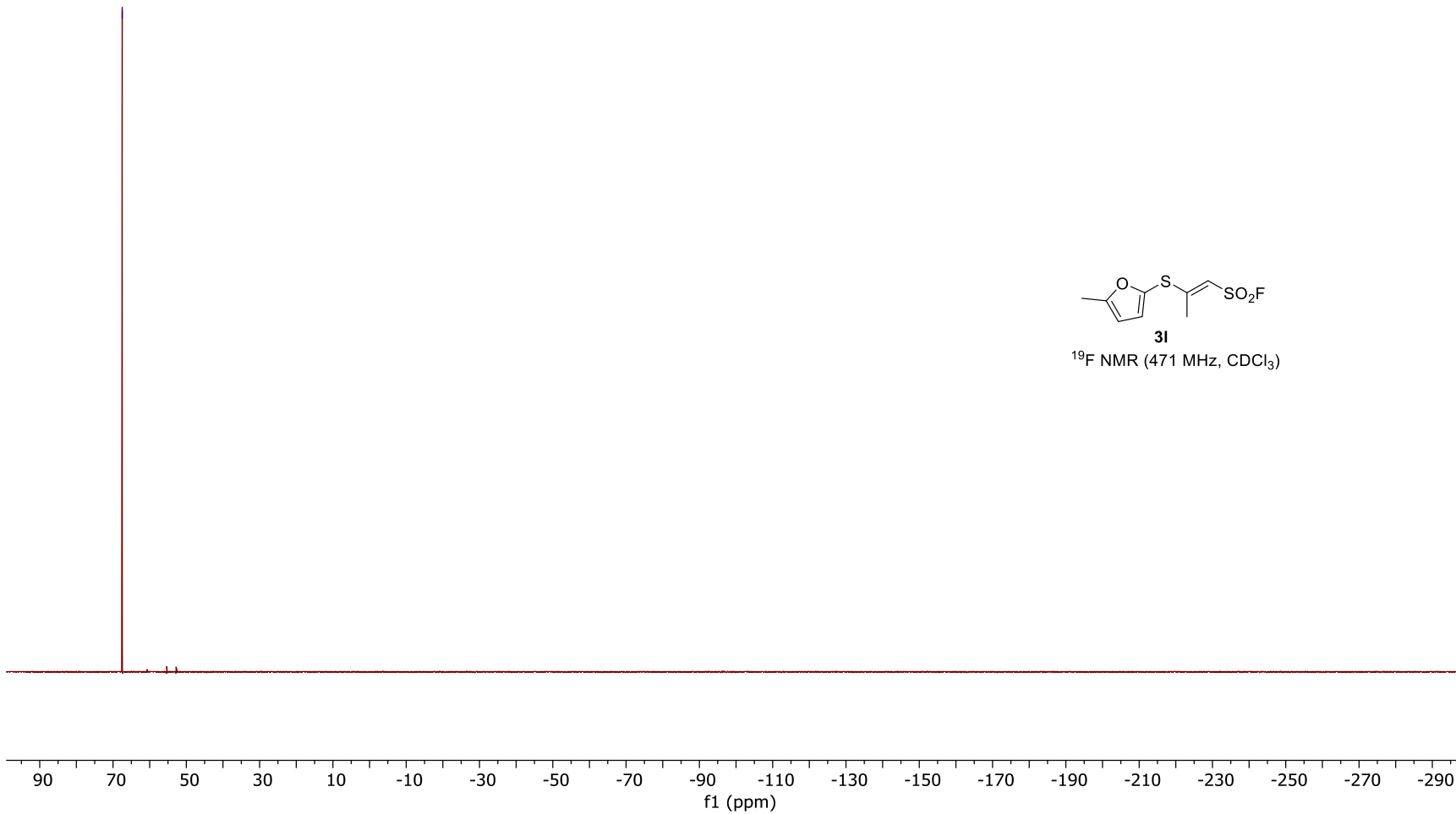
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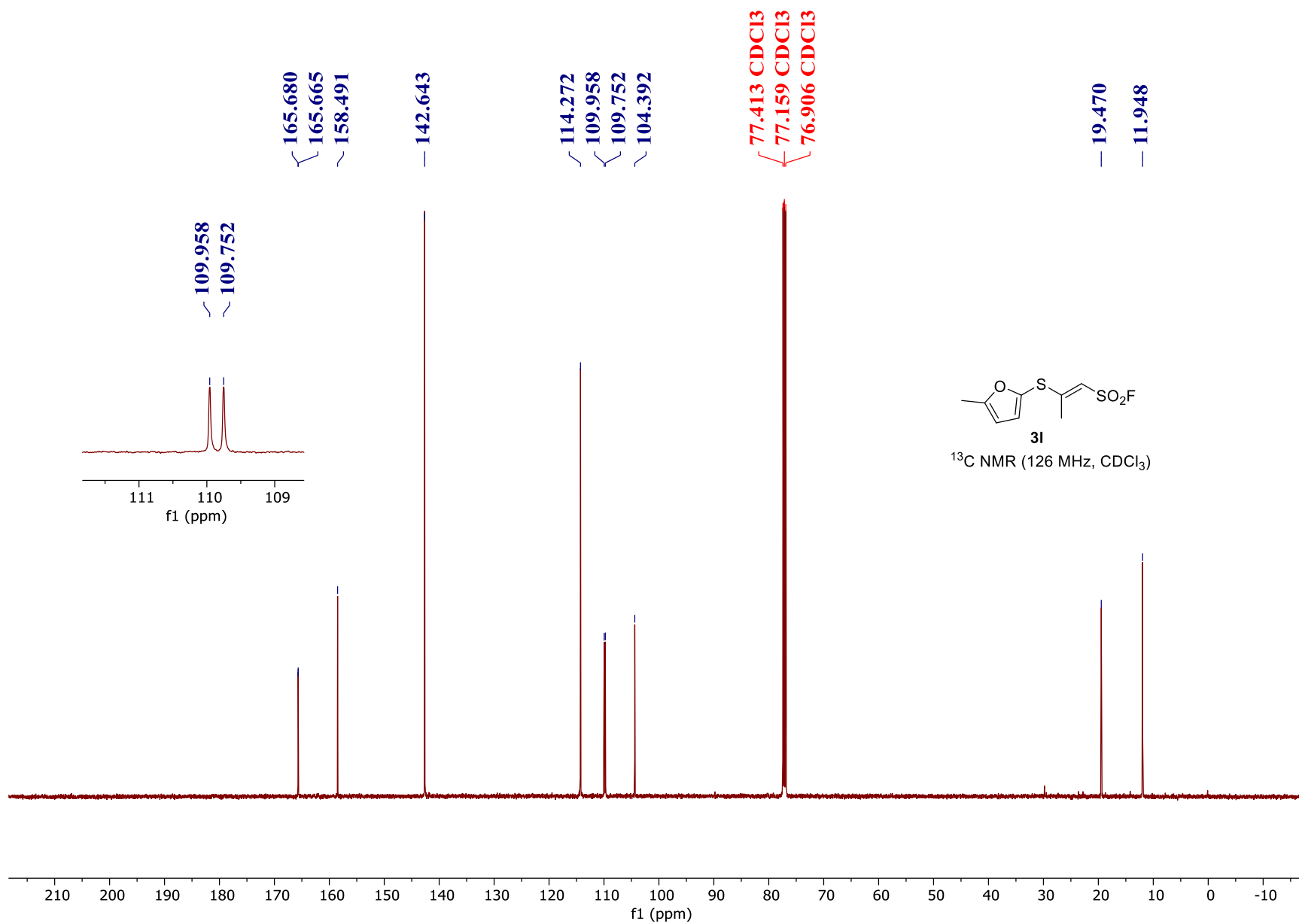


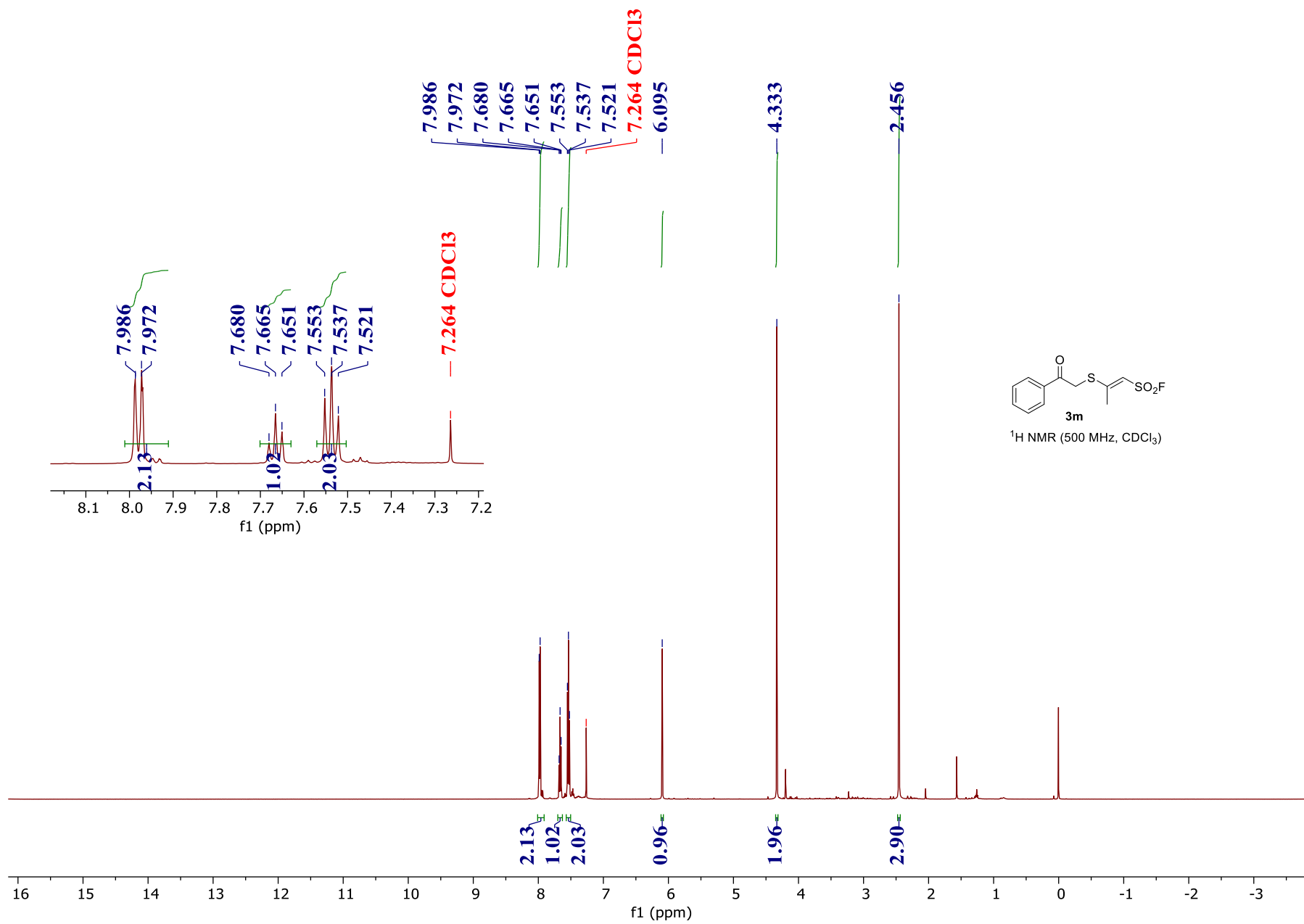




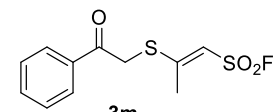
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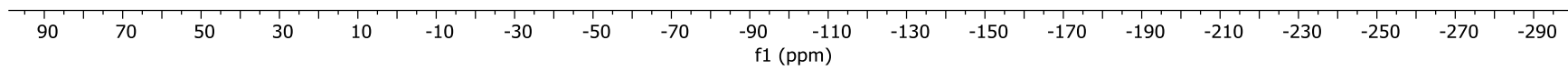


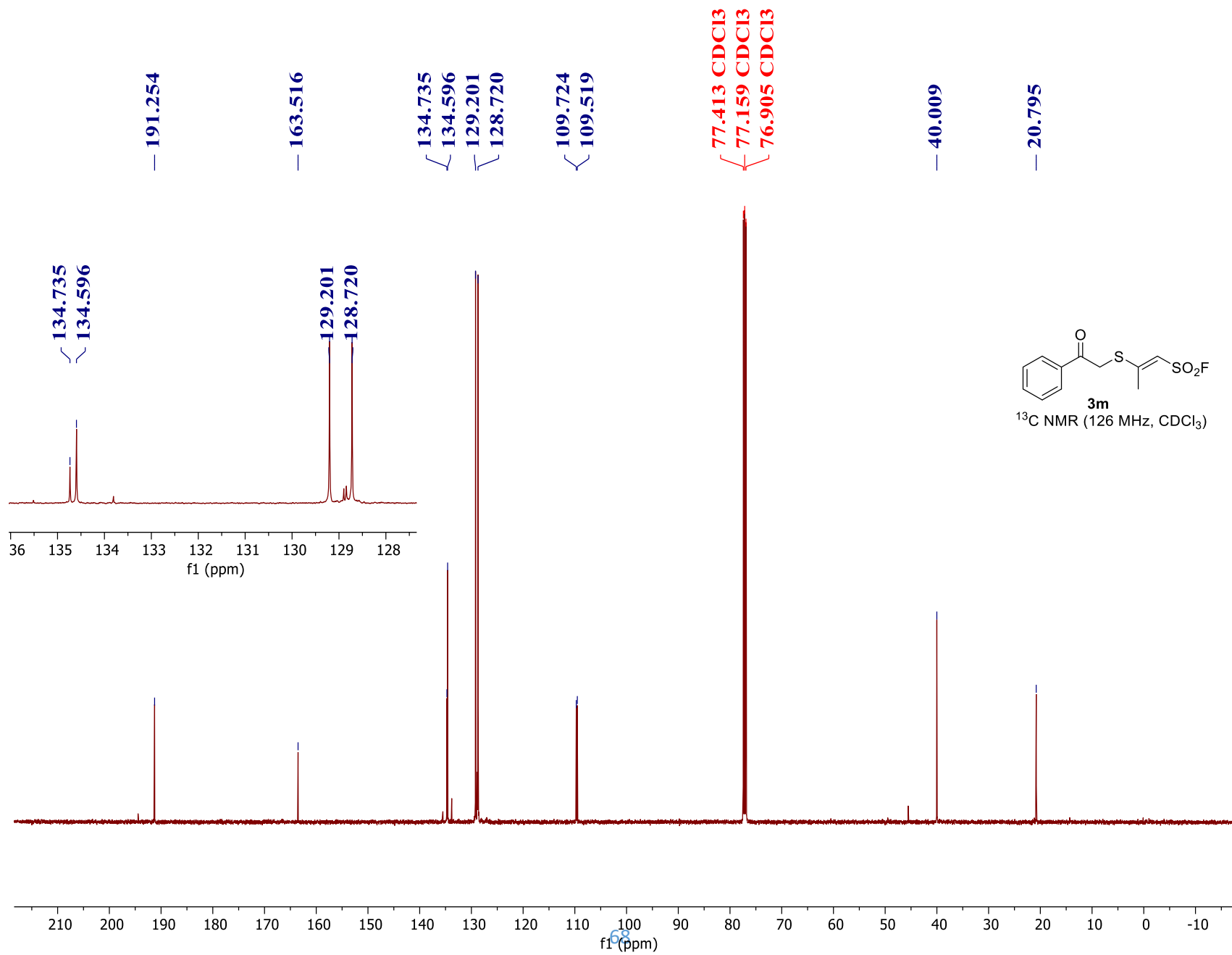


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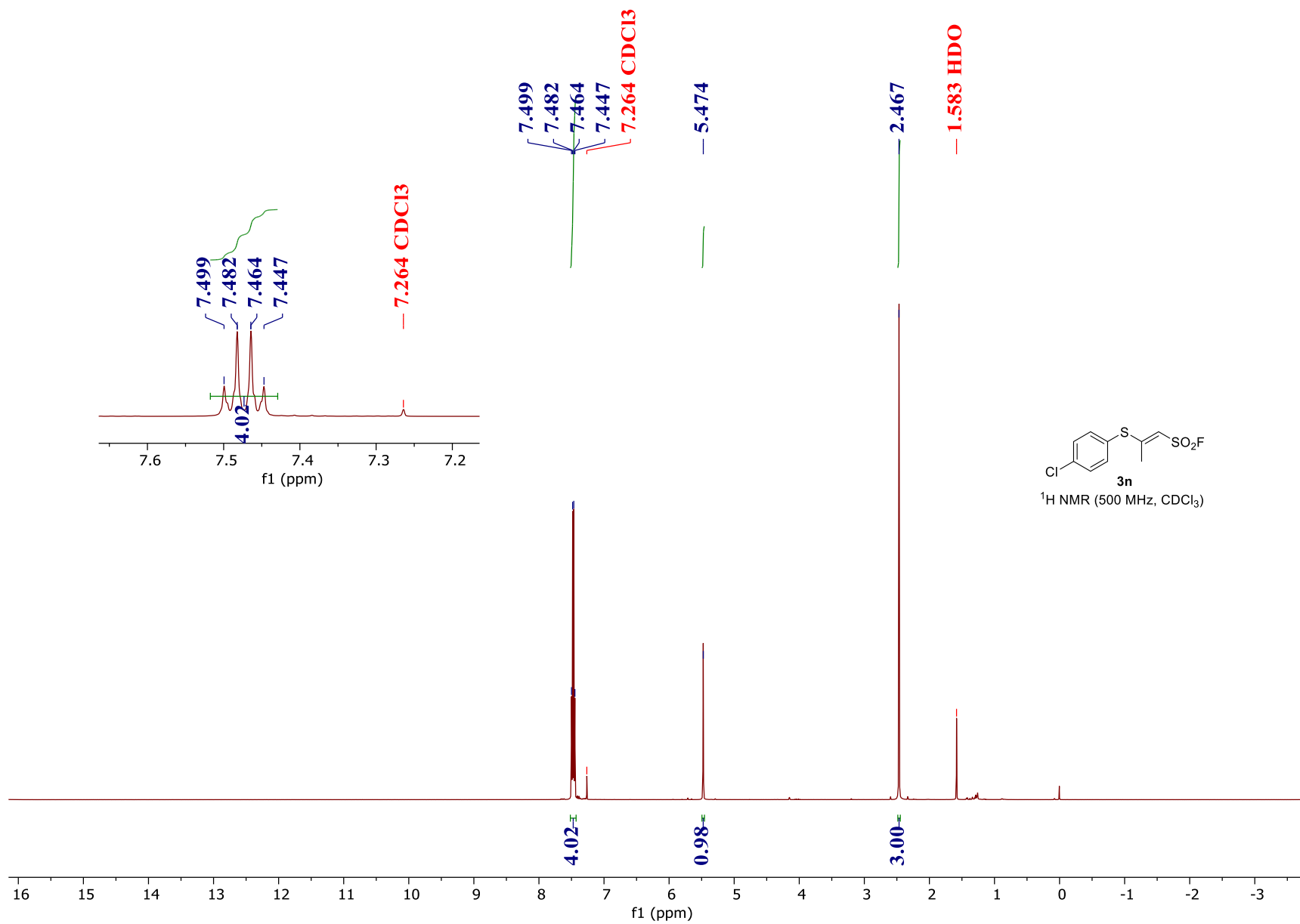


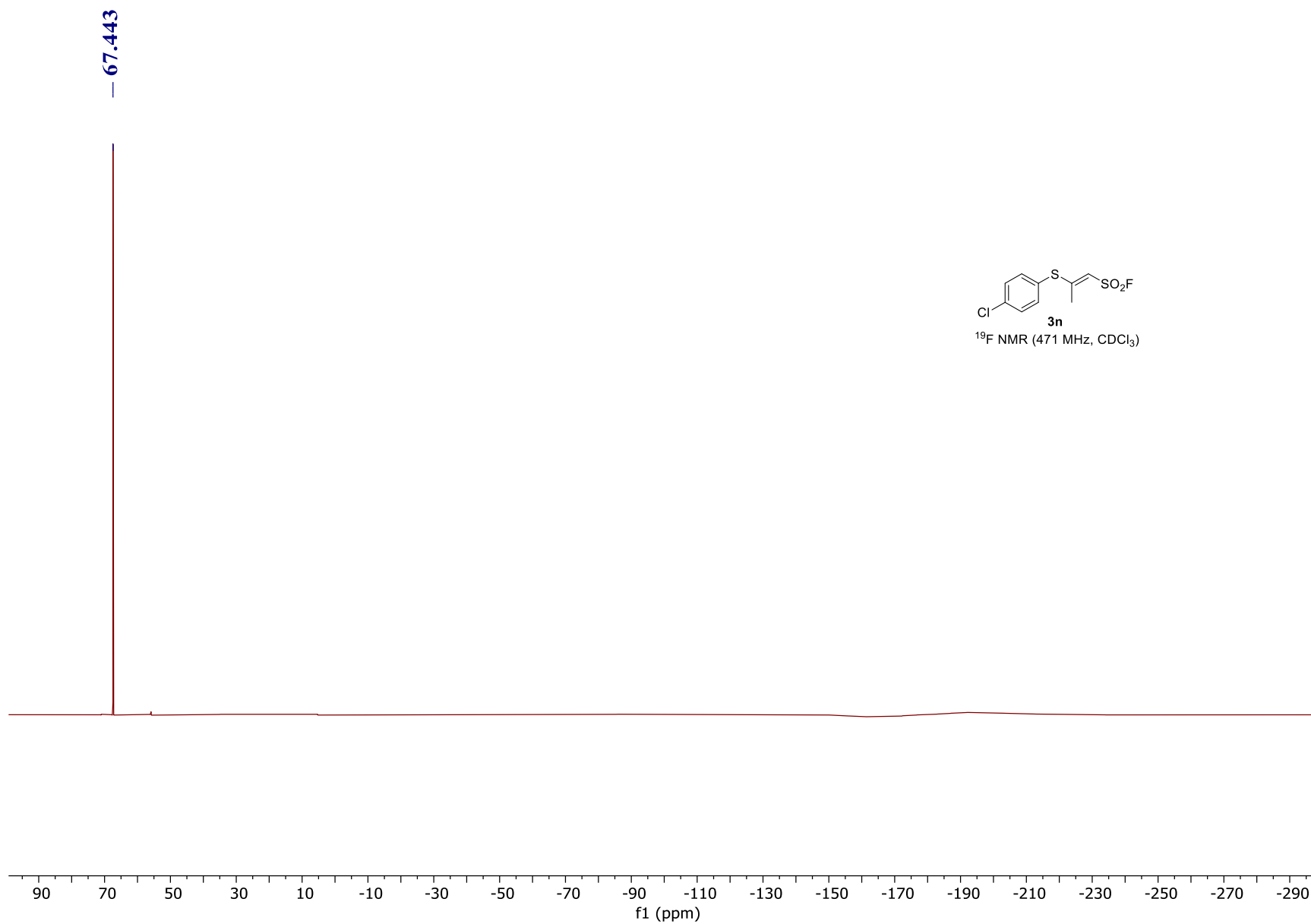
<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)

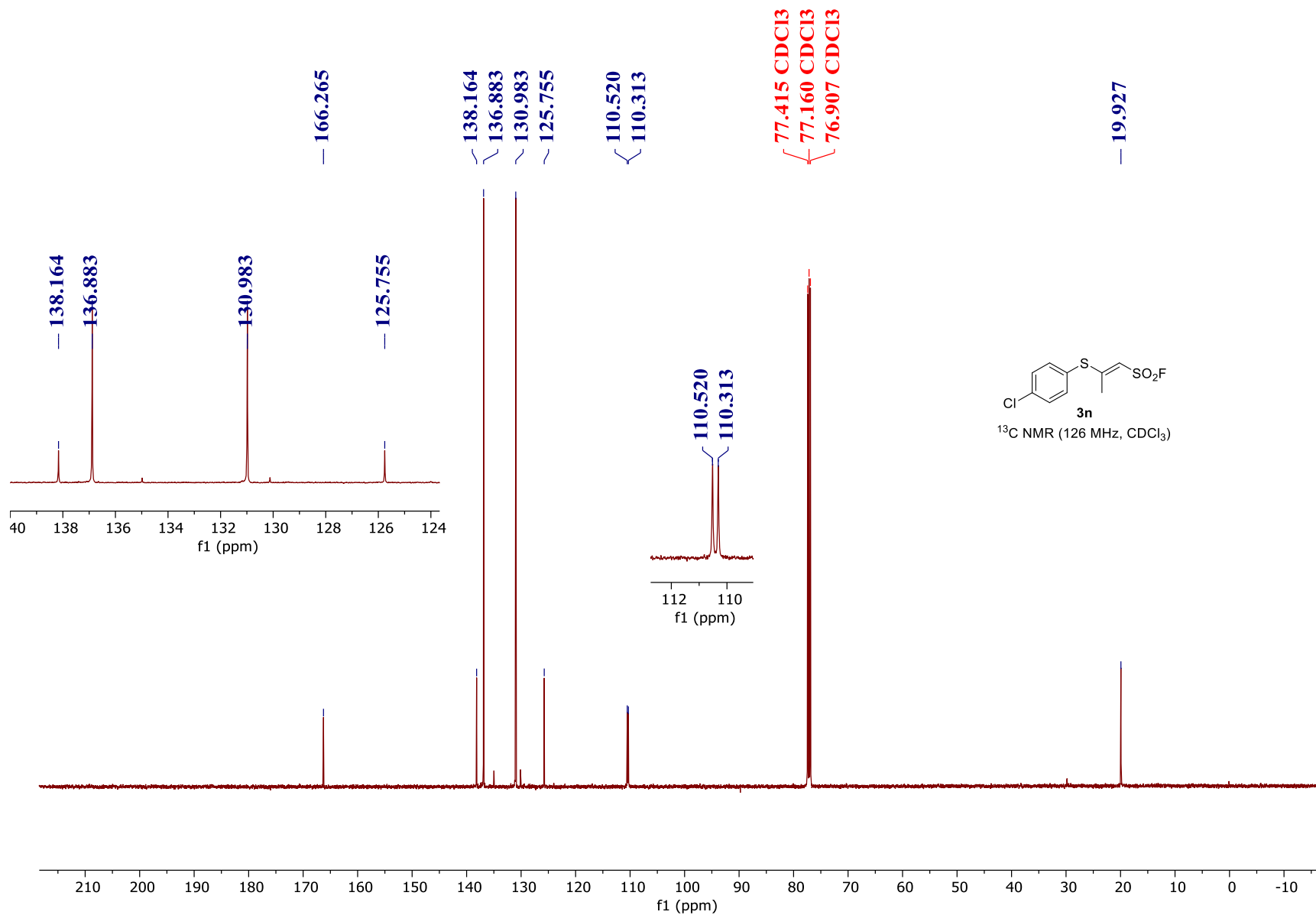


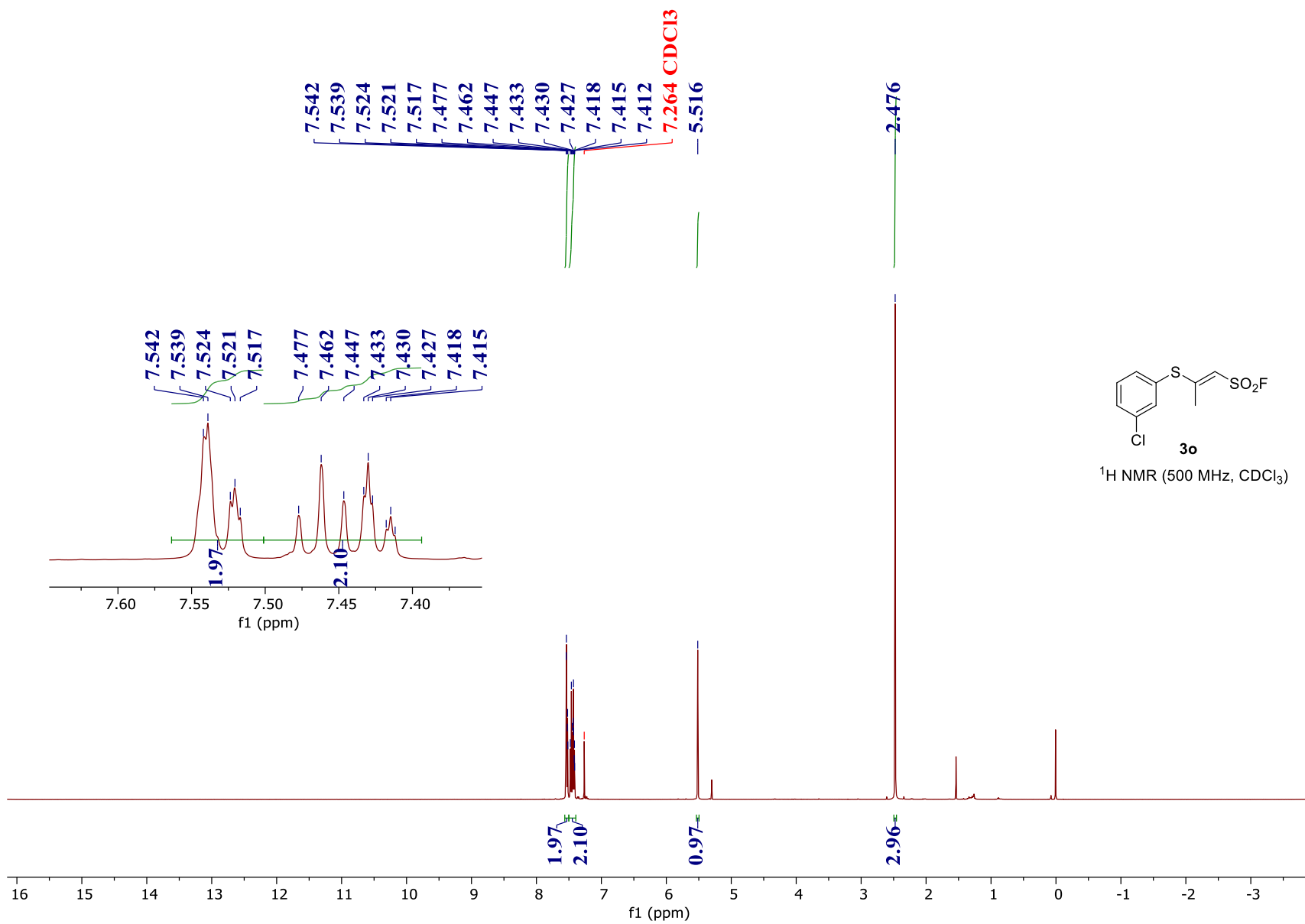




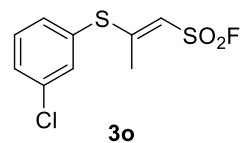




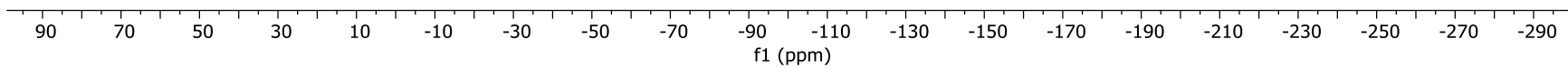


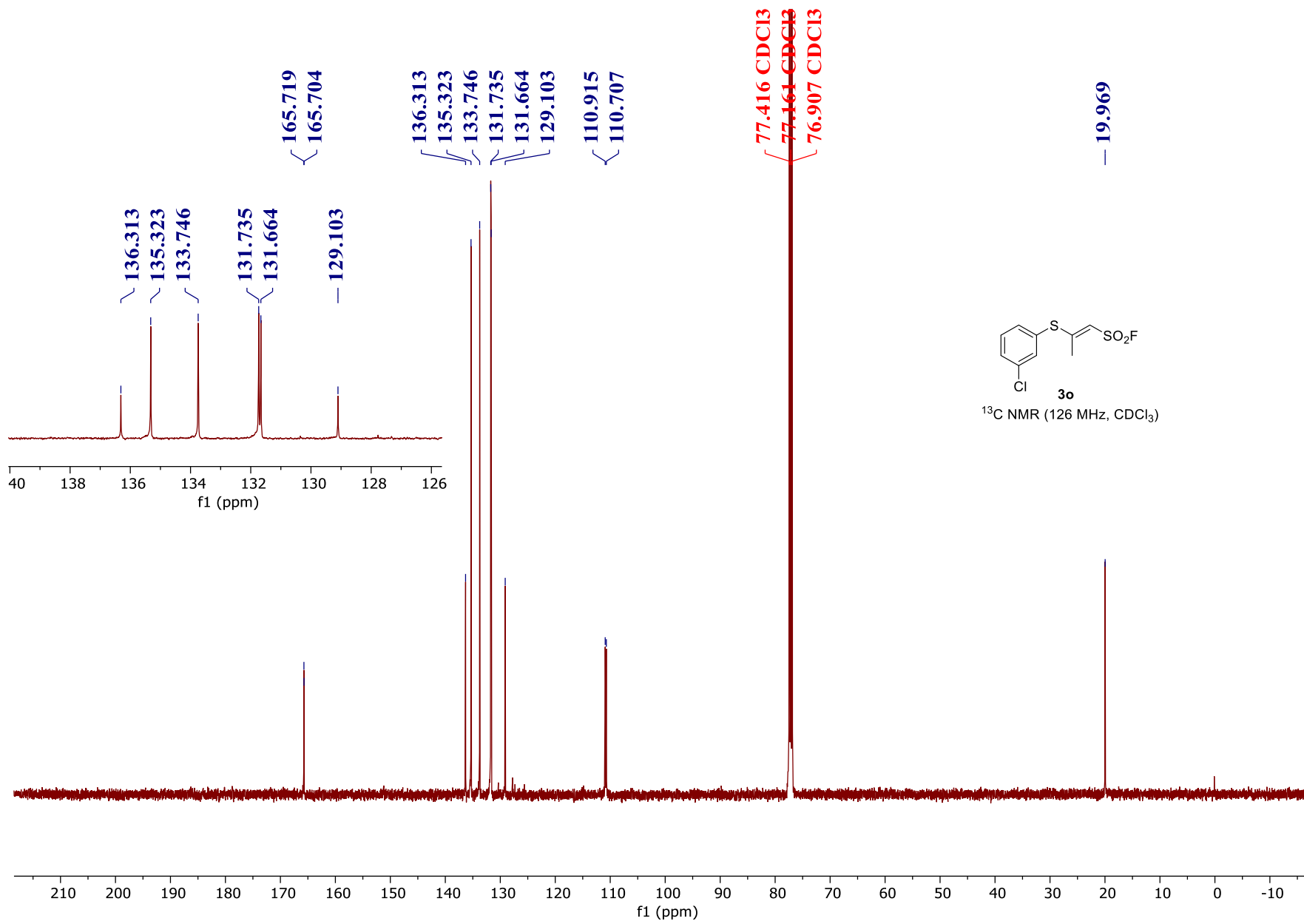


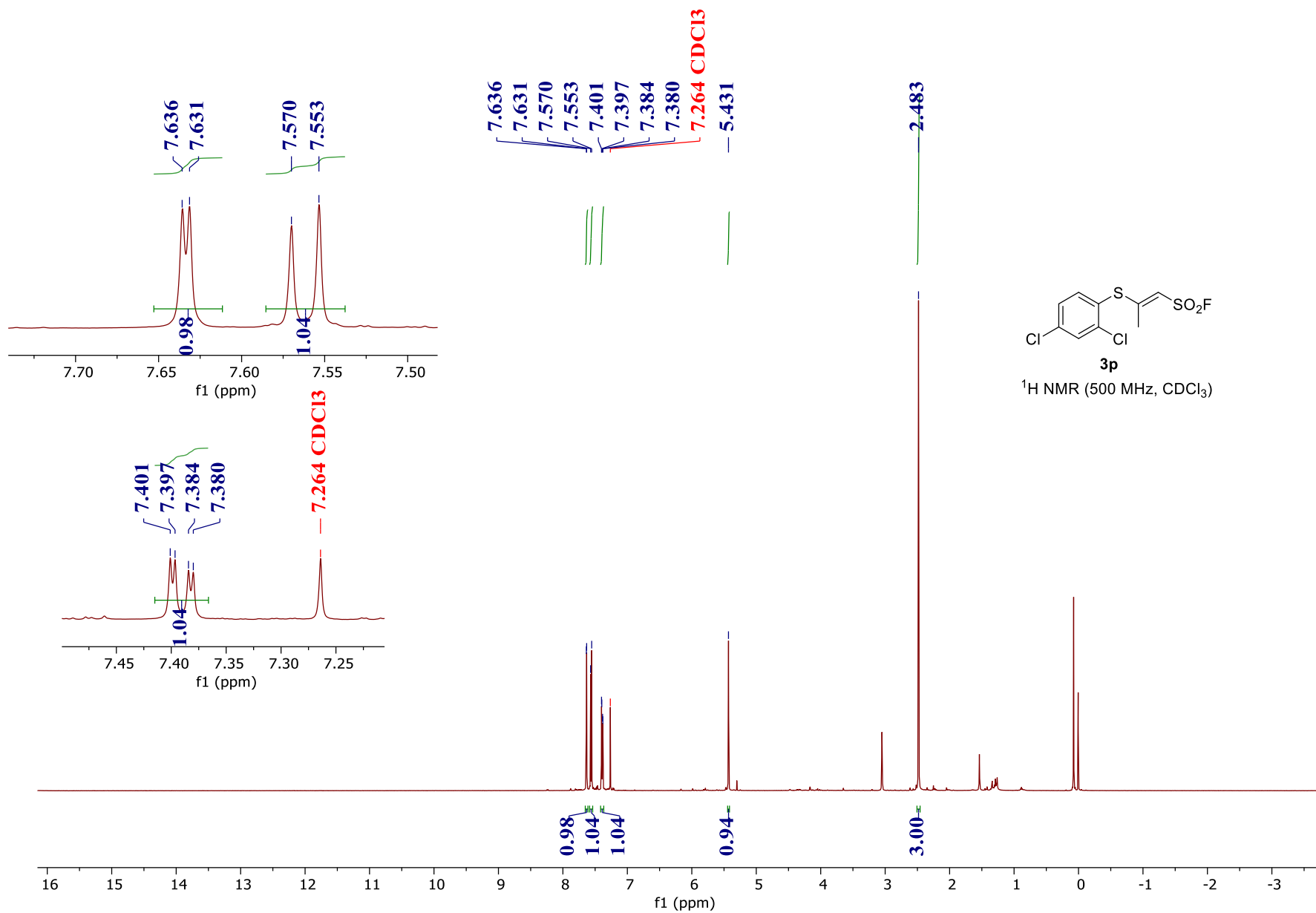
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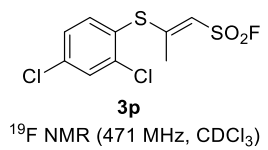
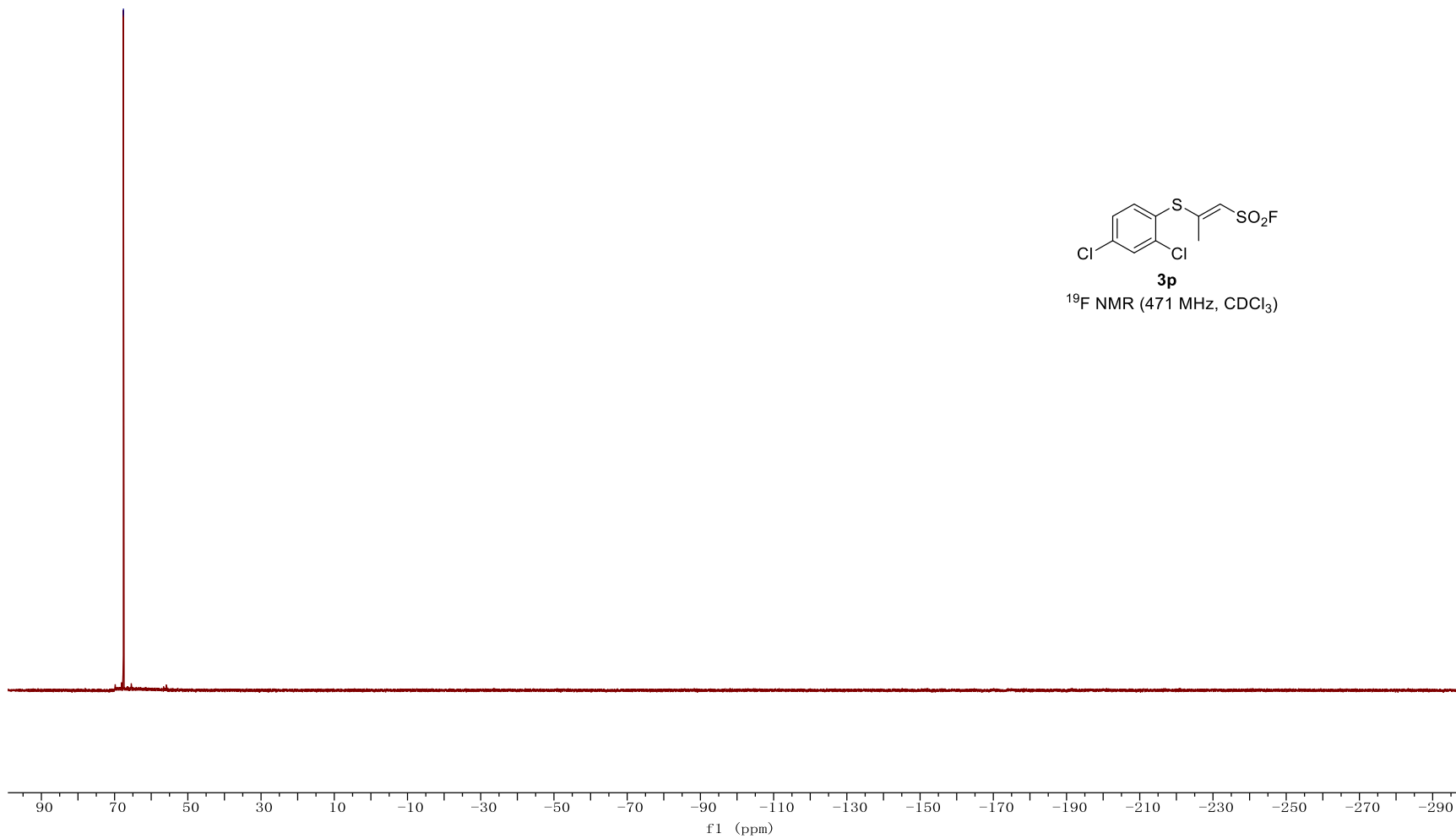
<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)



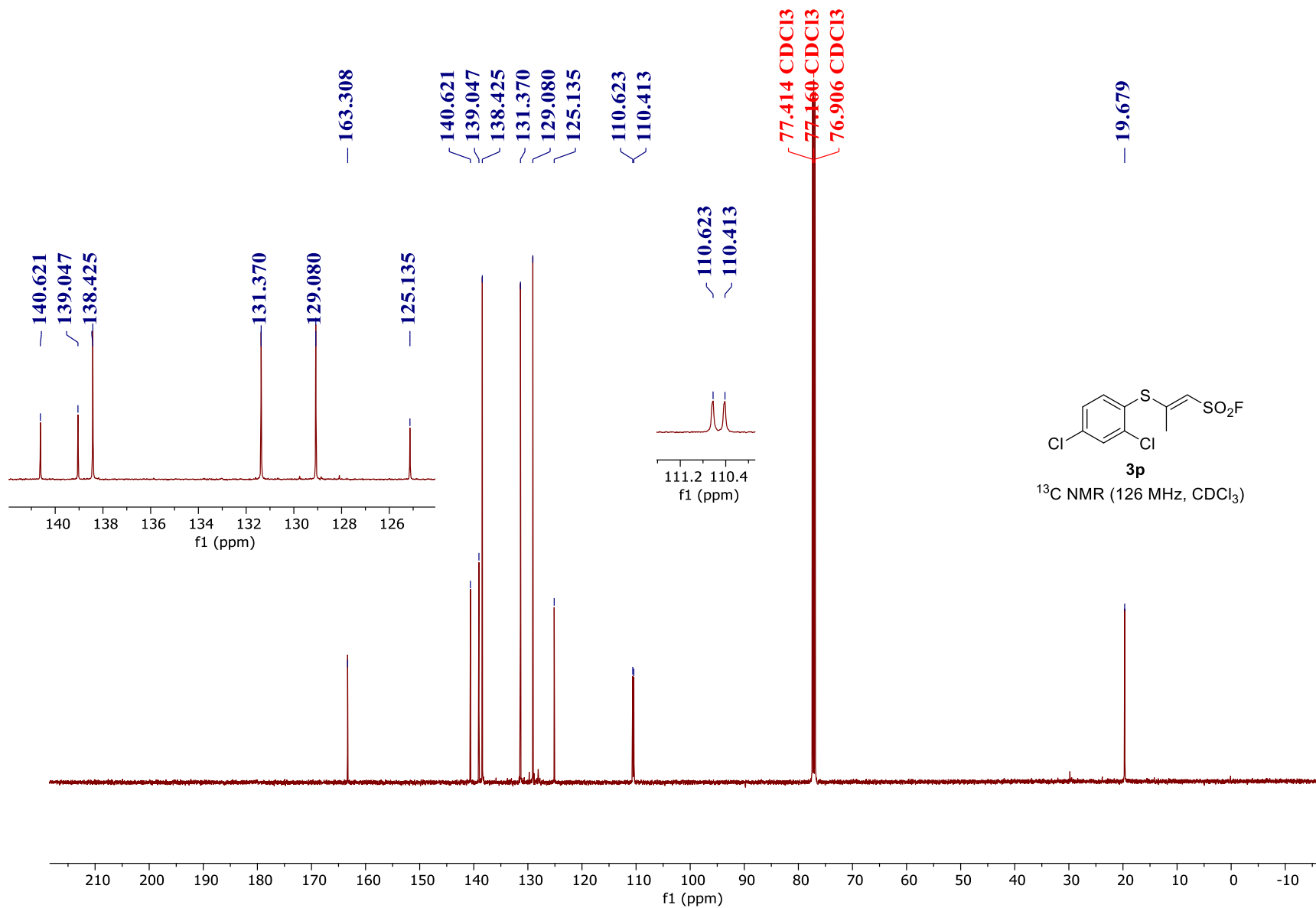


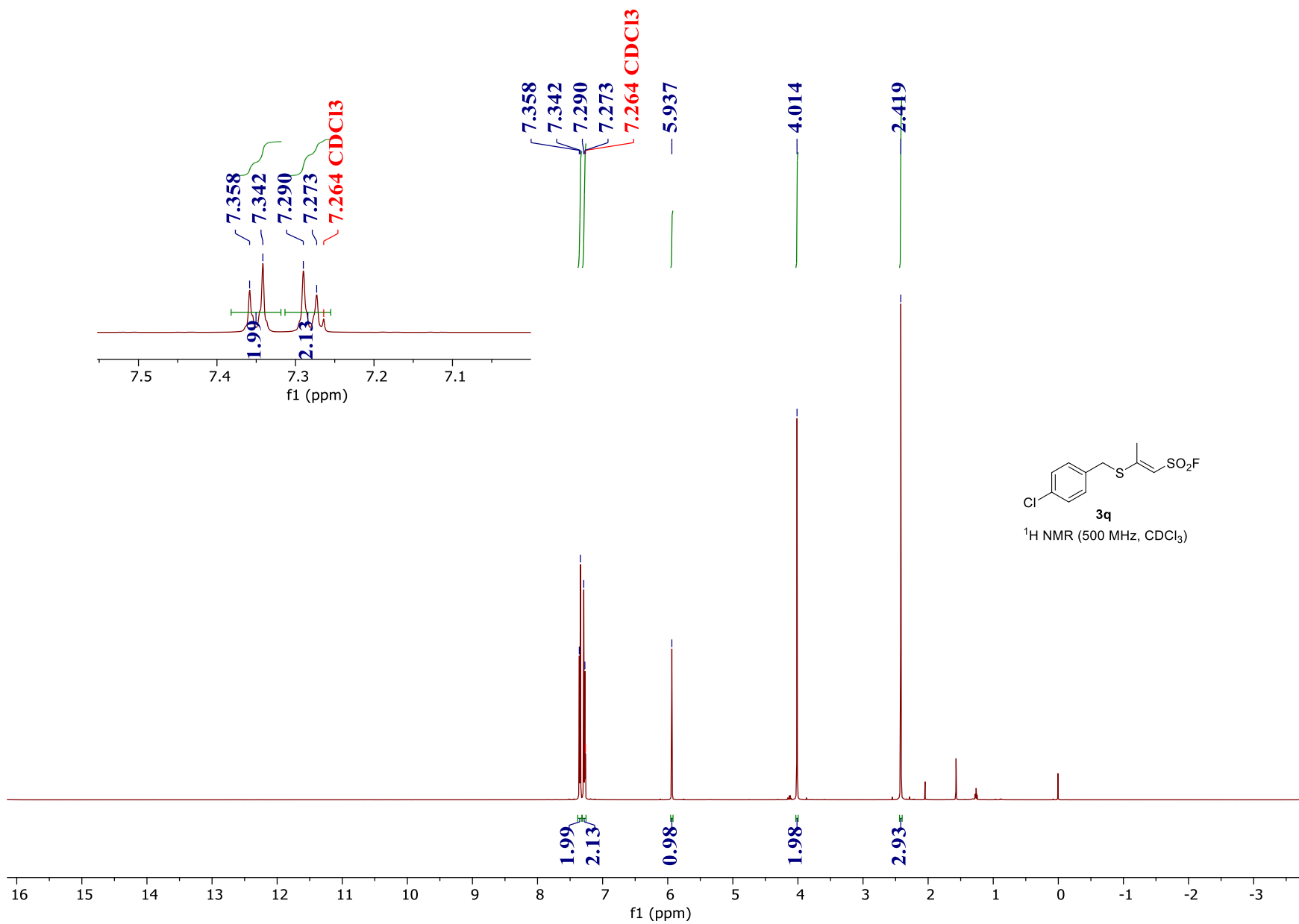


— 67.592

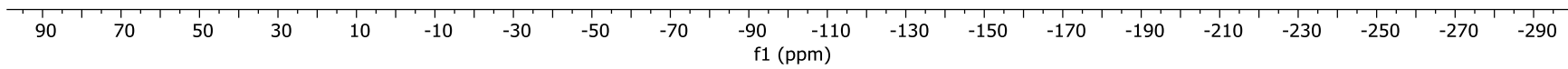
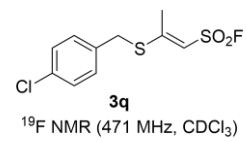
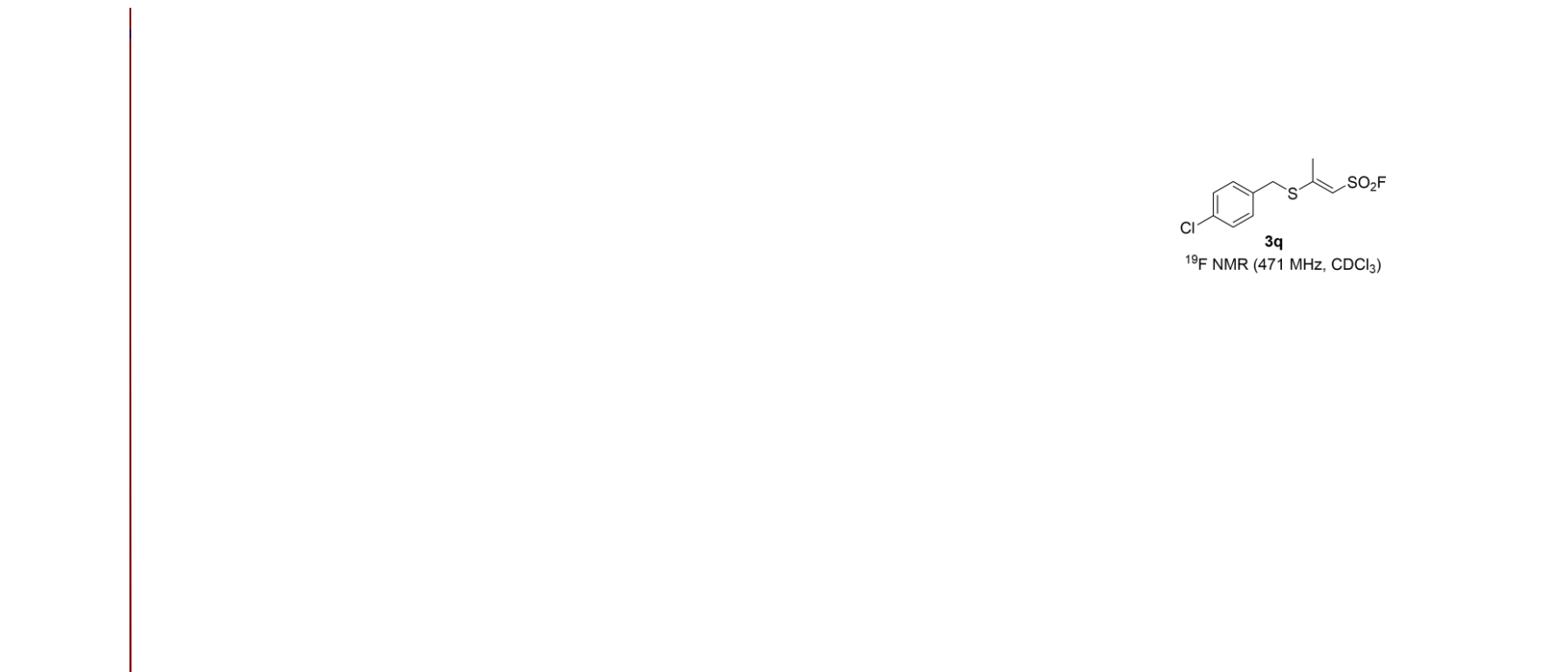


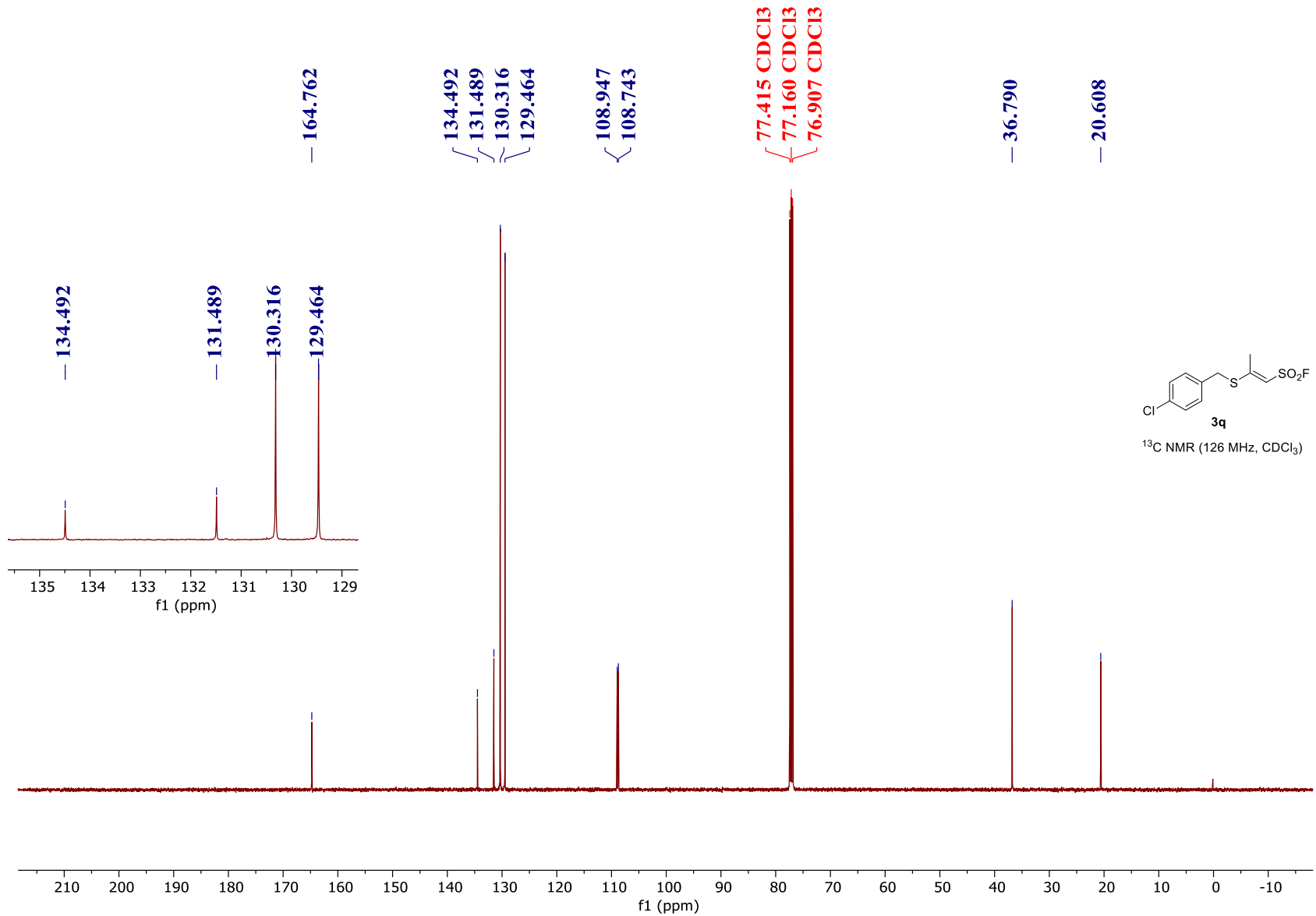


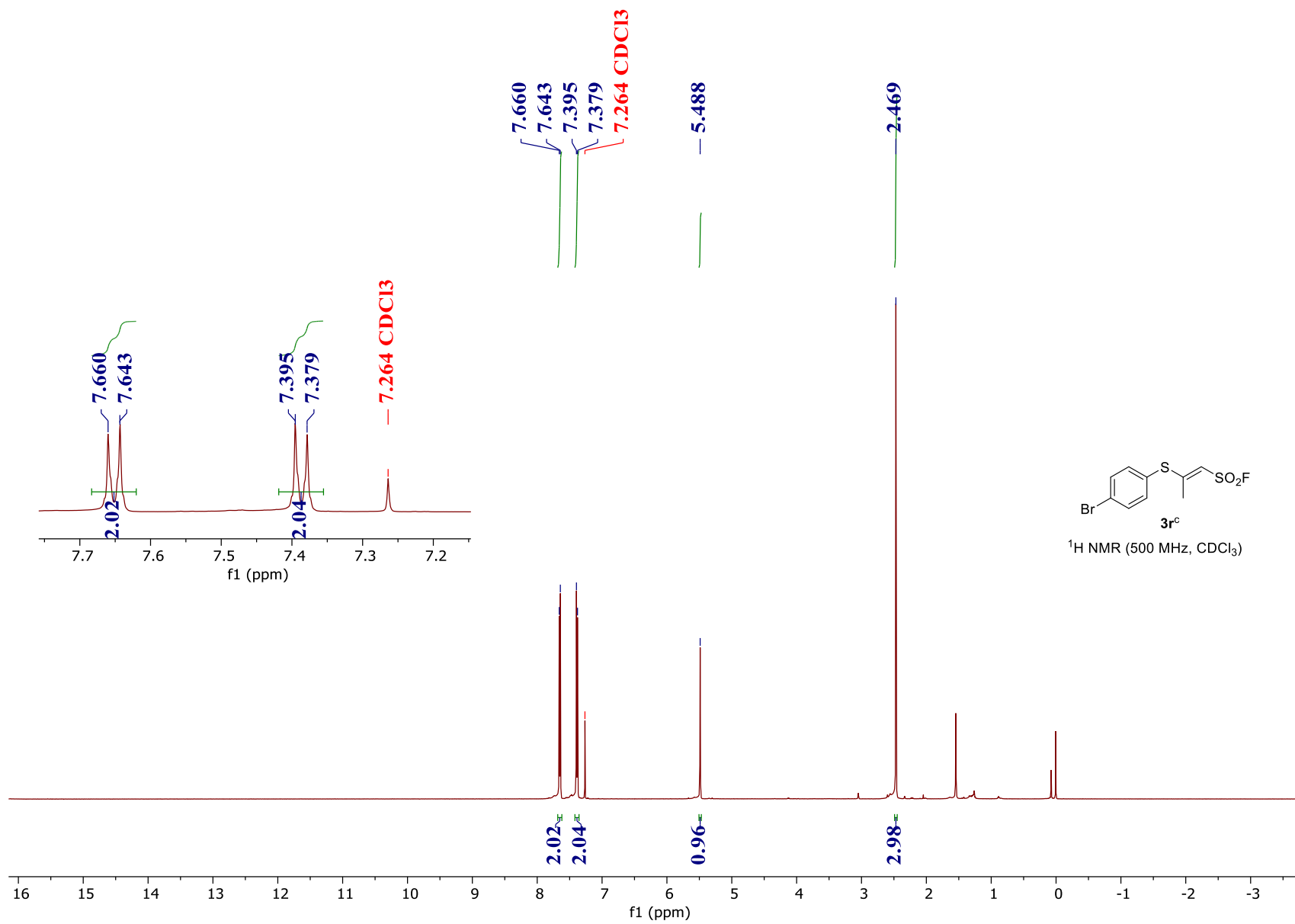


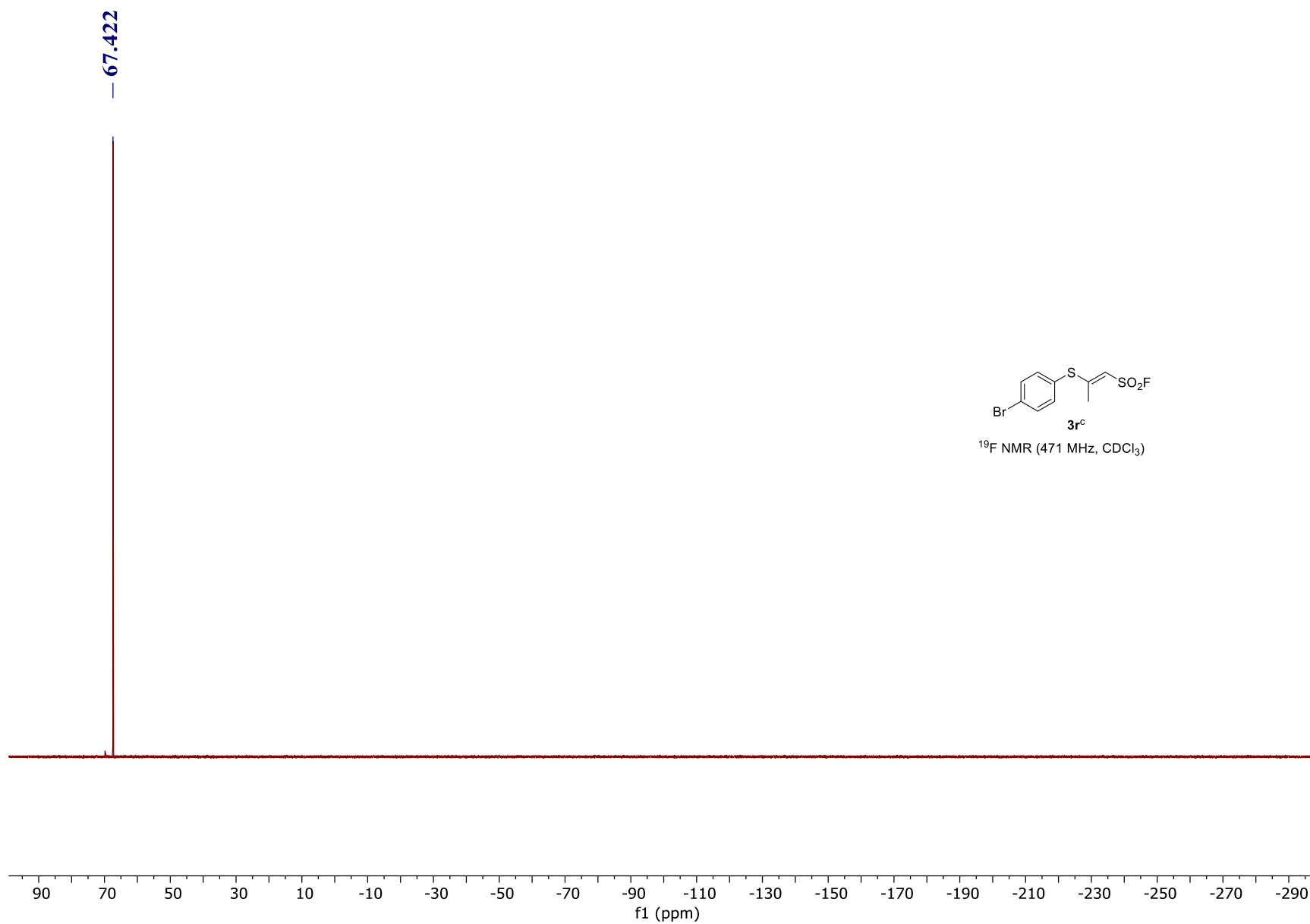


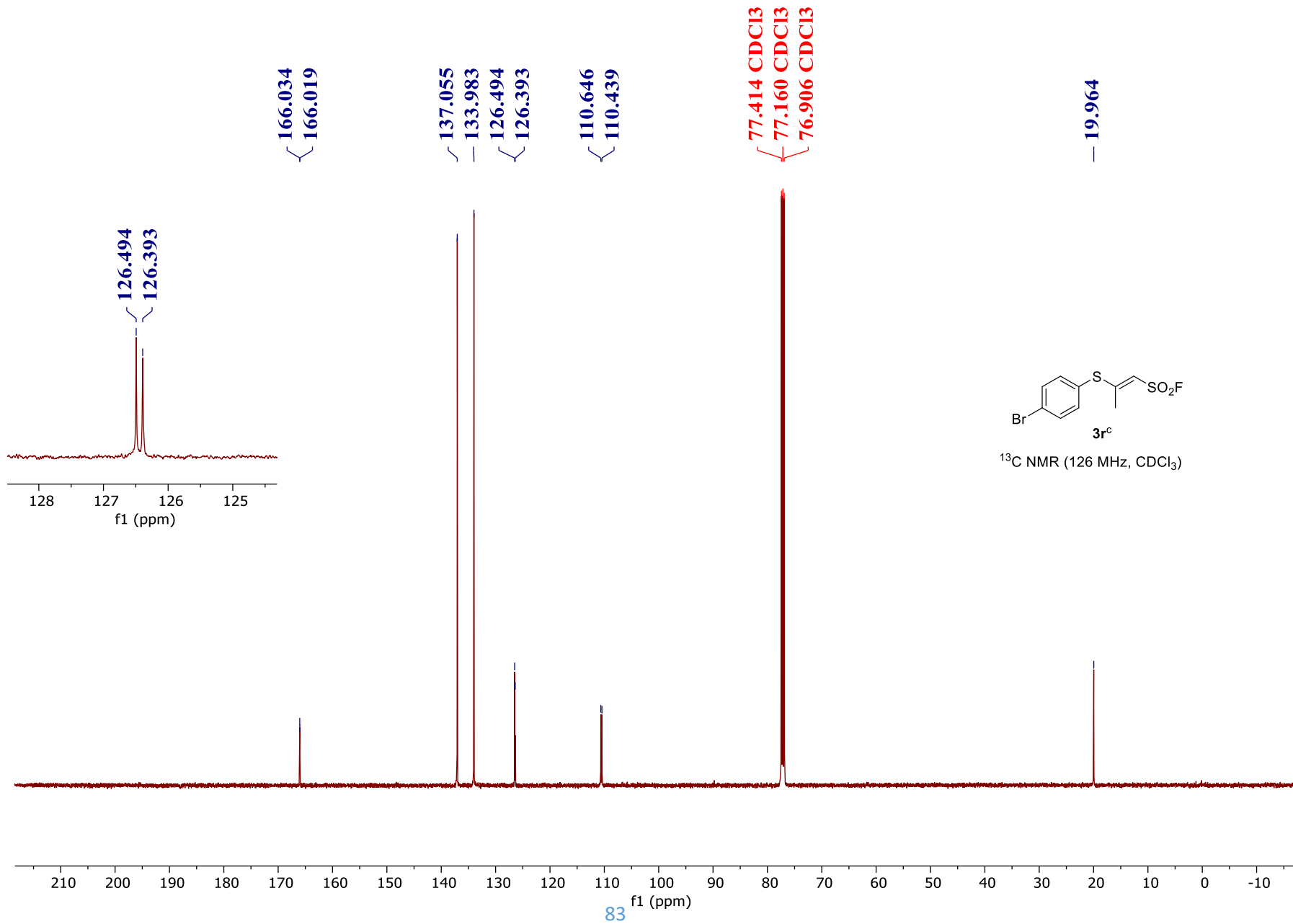
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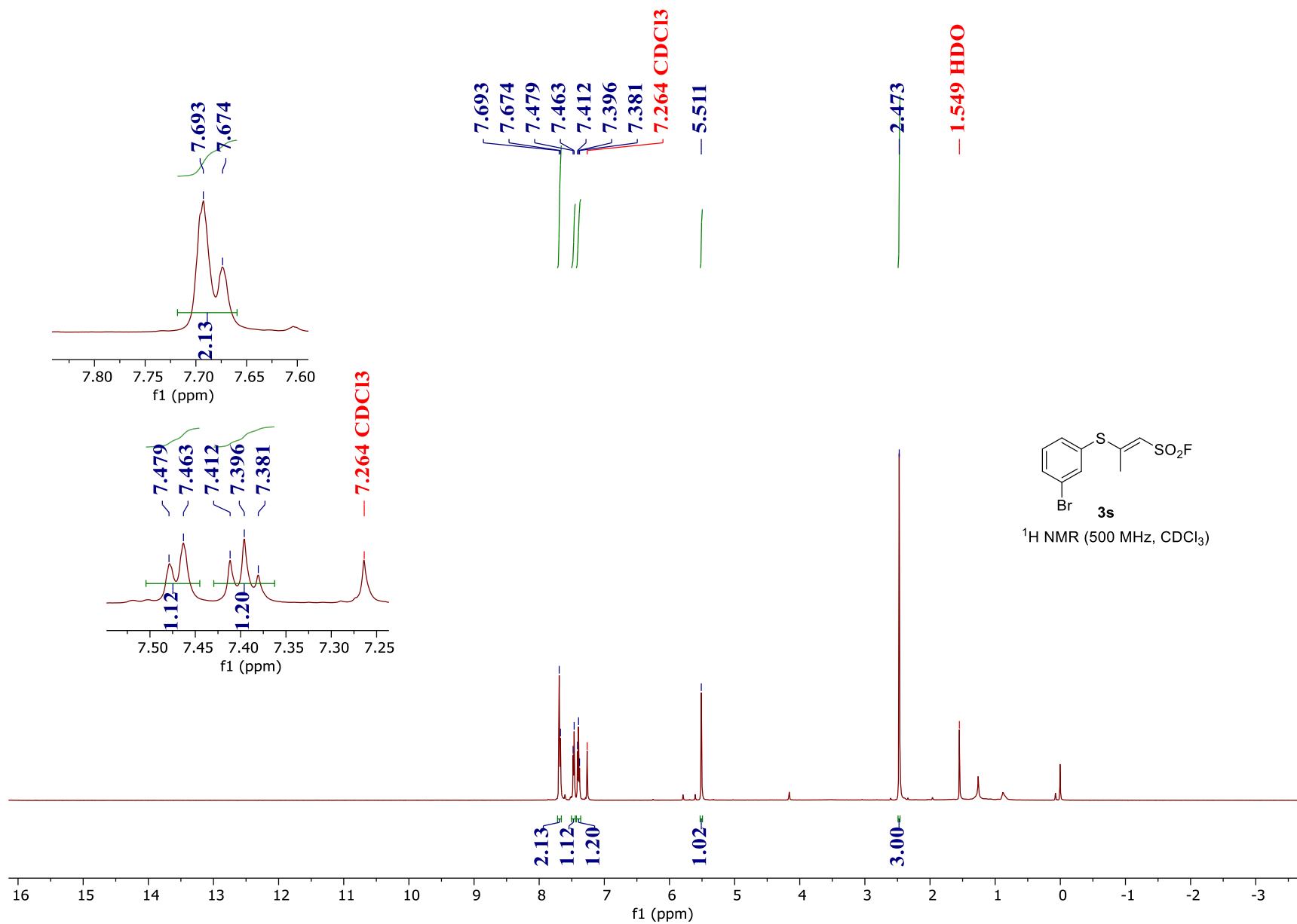




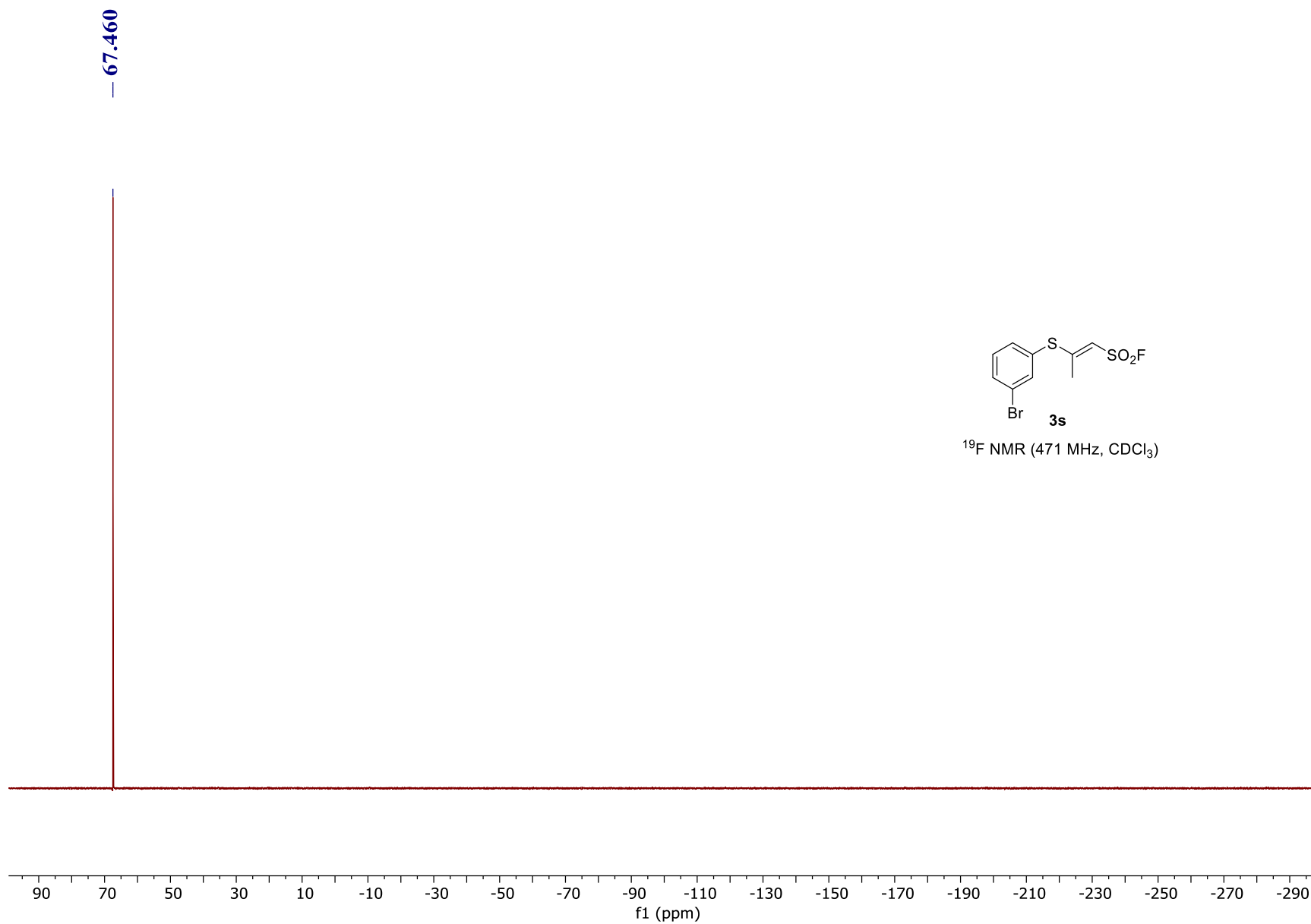


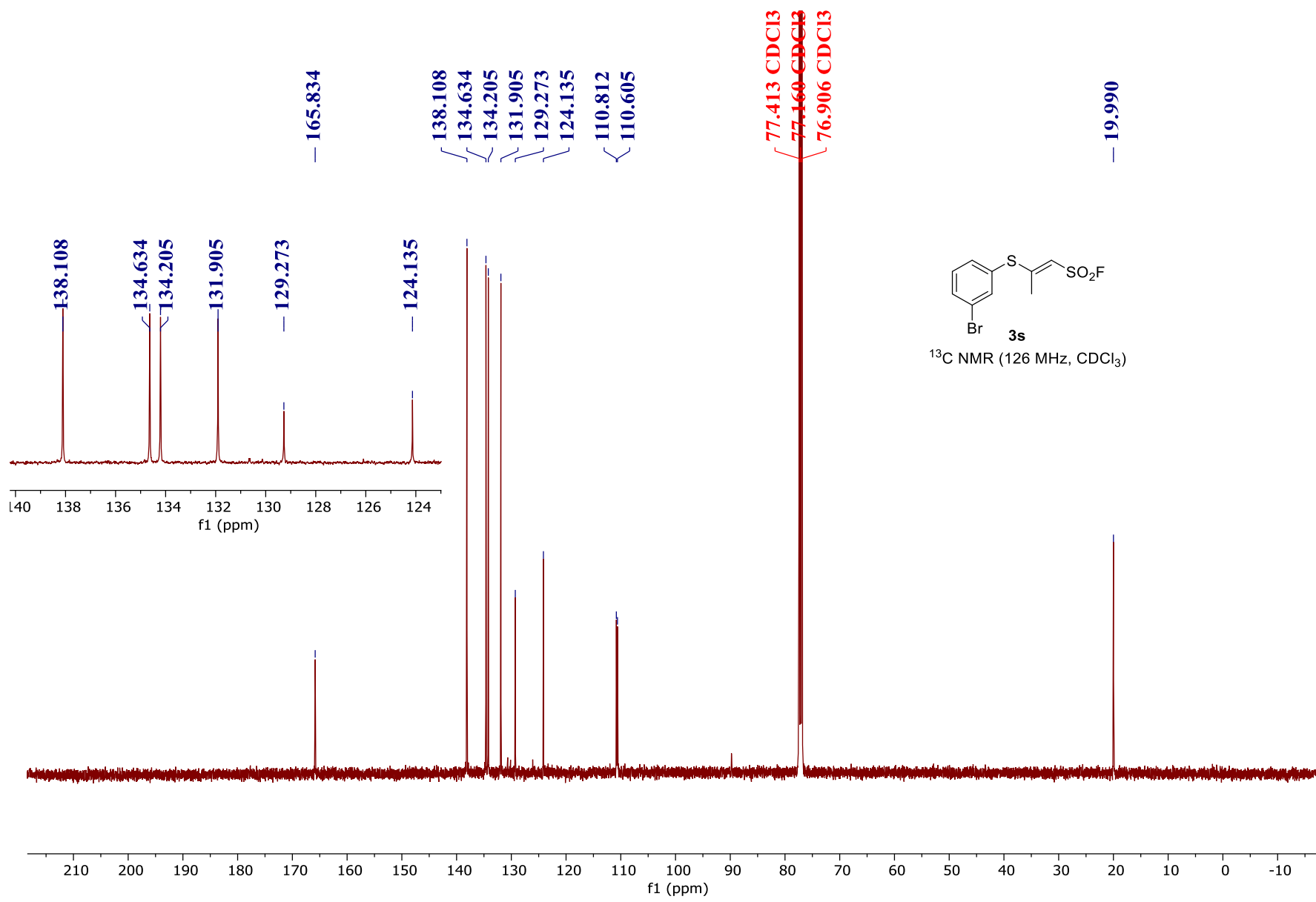


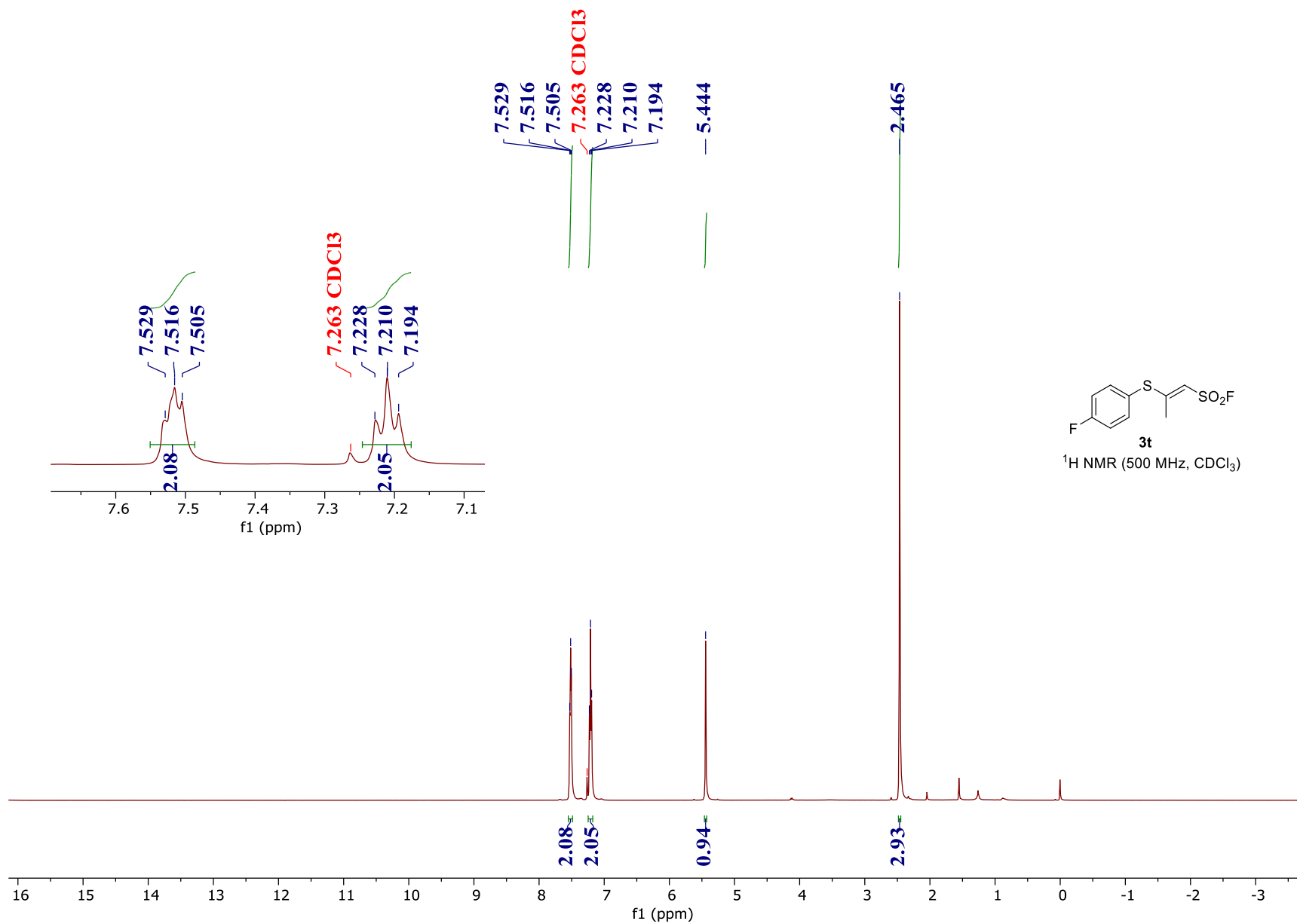


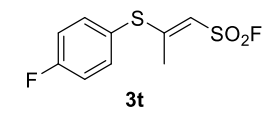




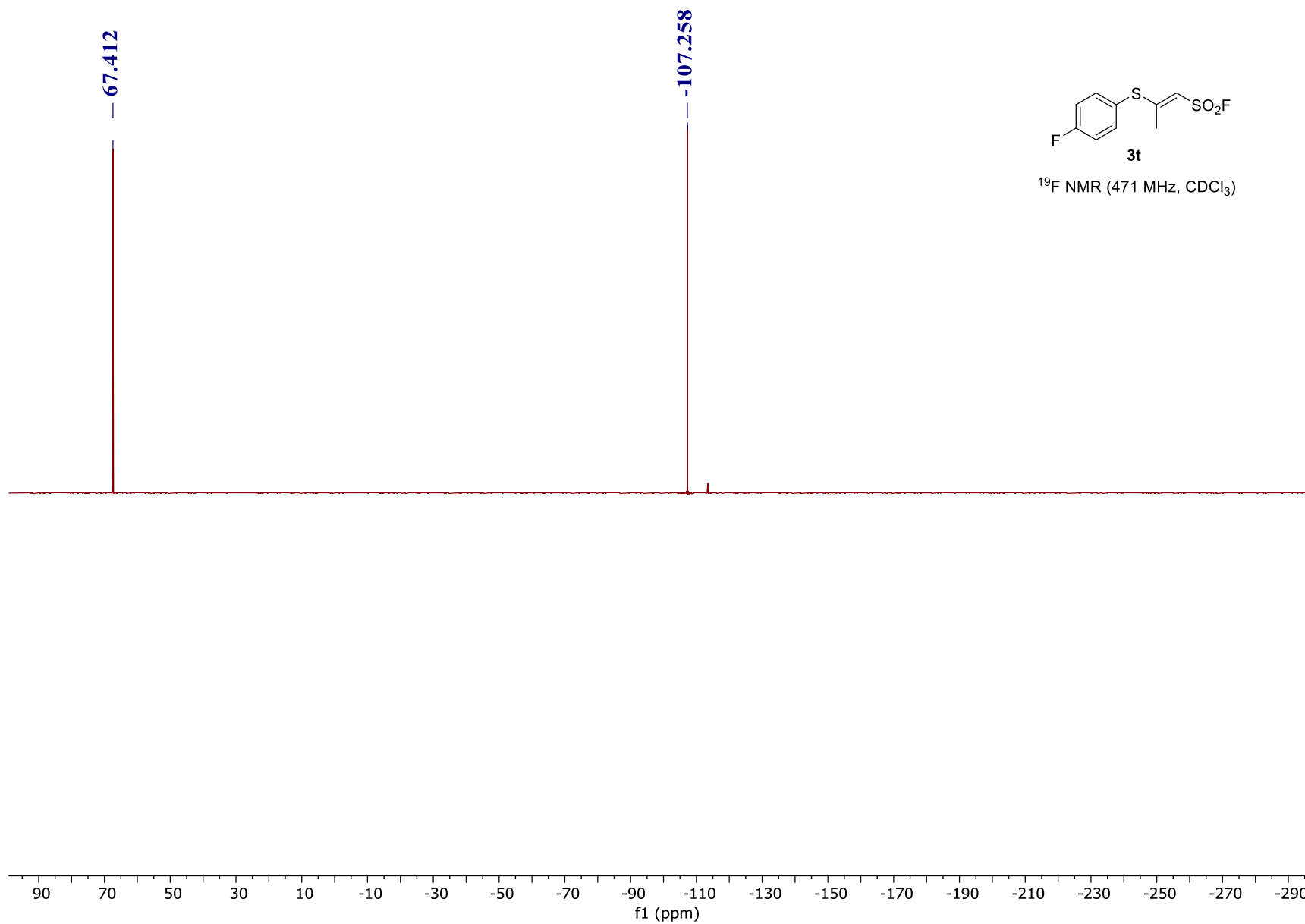


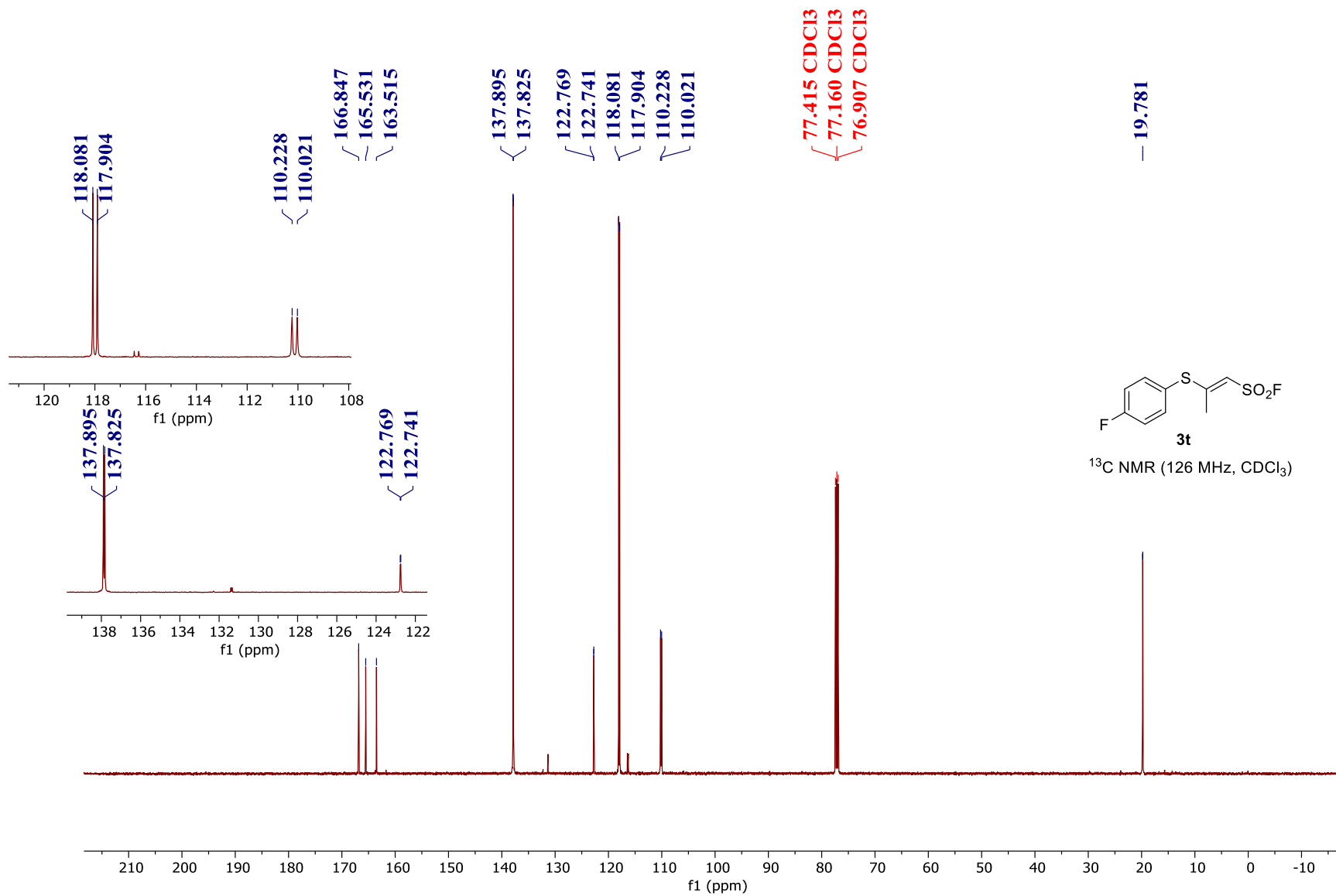


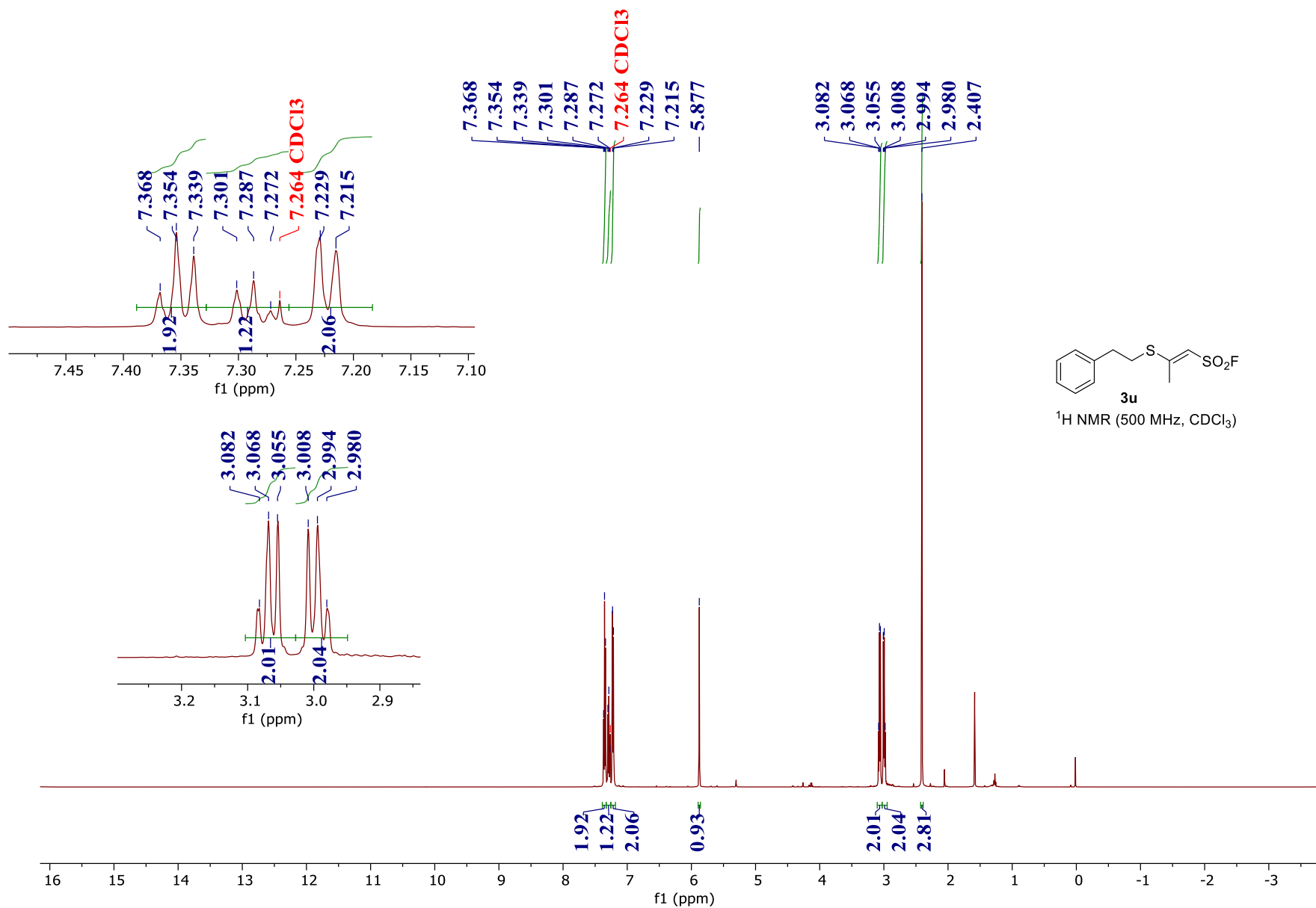




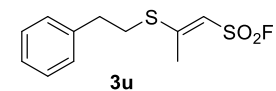
<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)



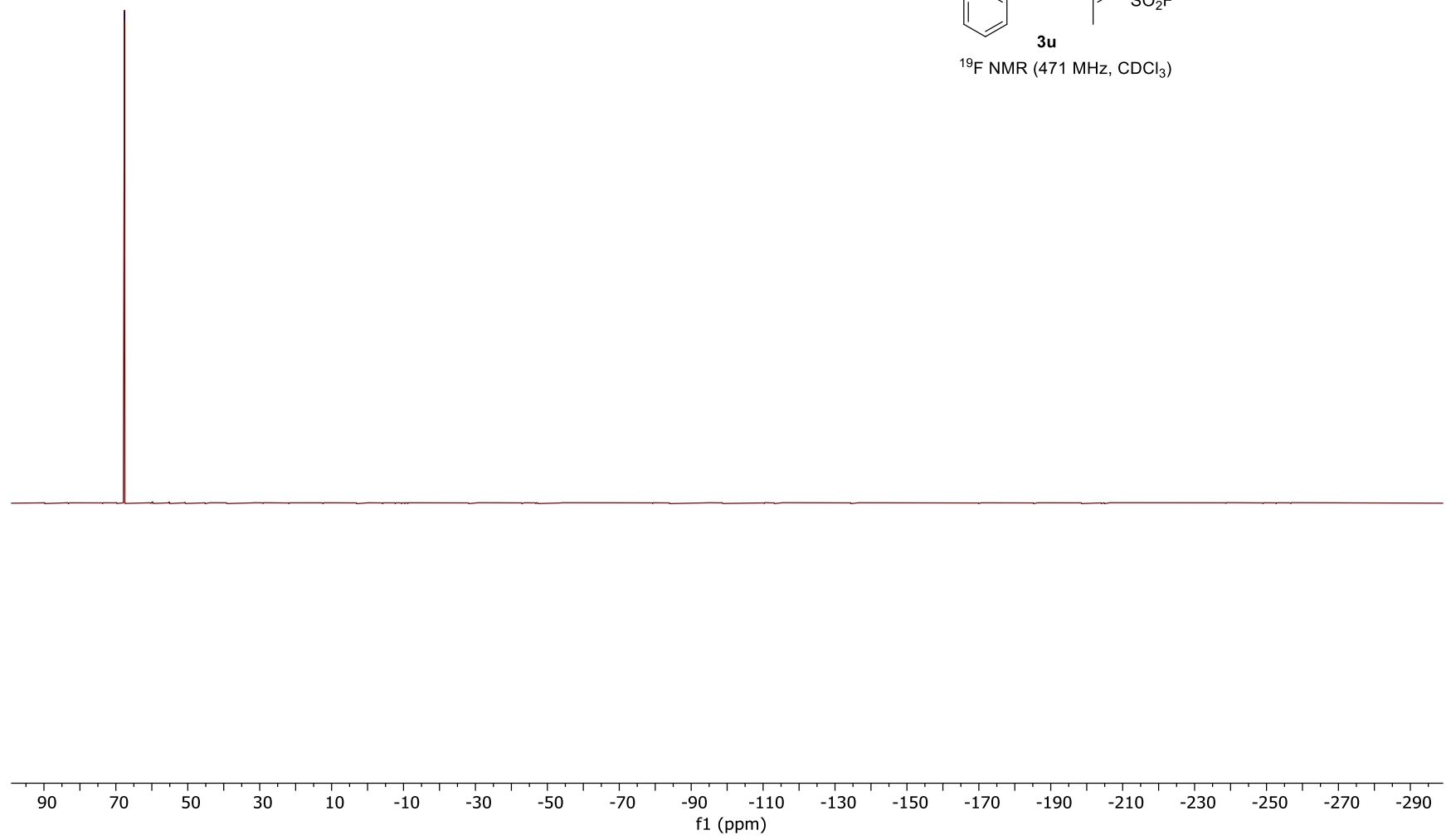


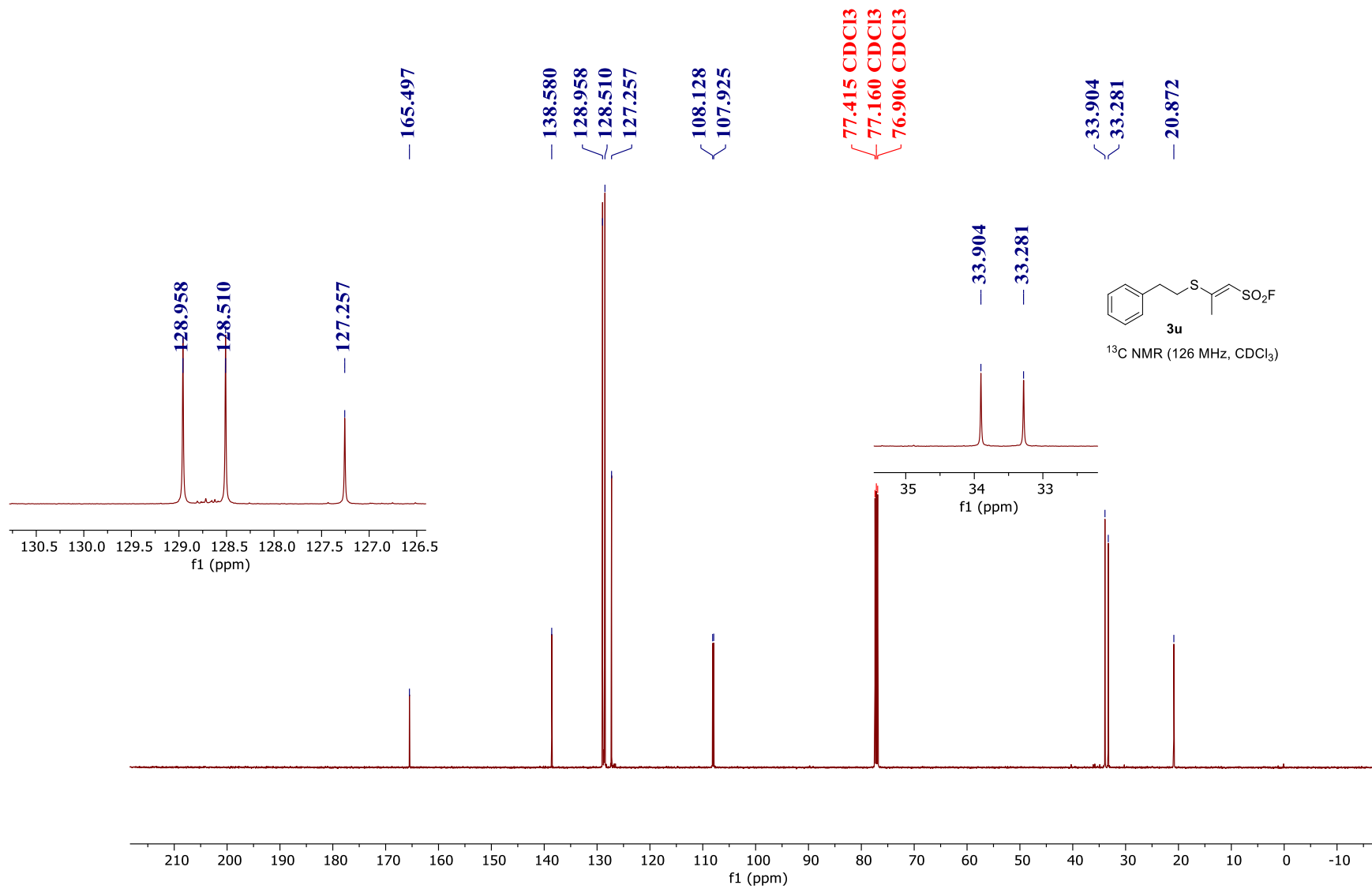


- 67.613

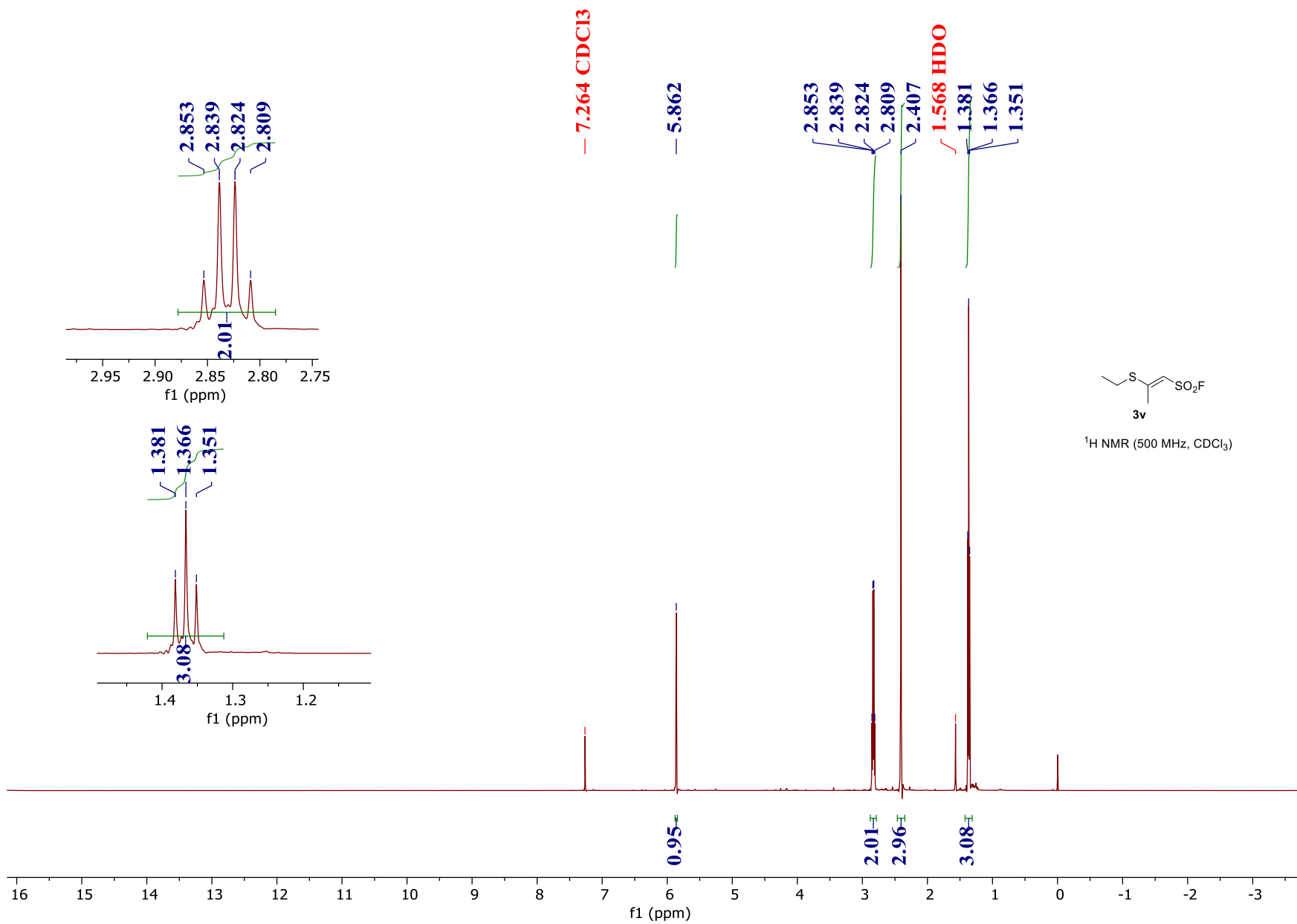


<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)

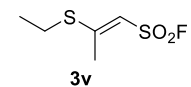




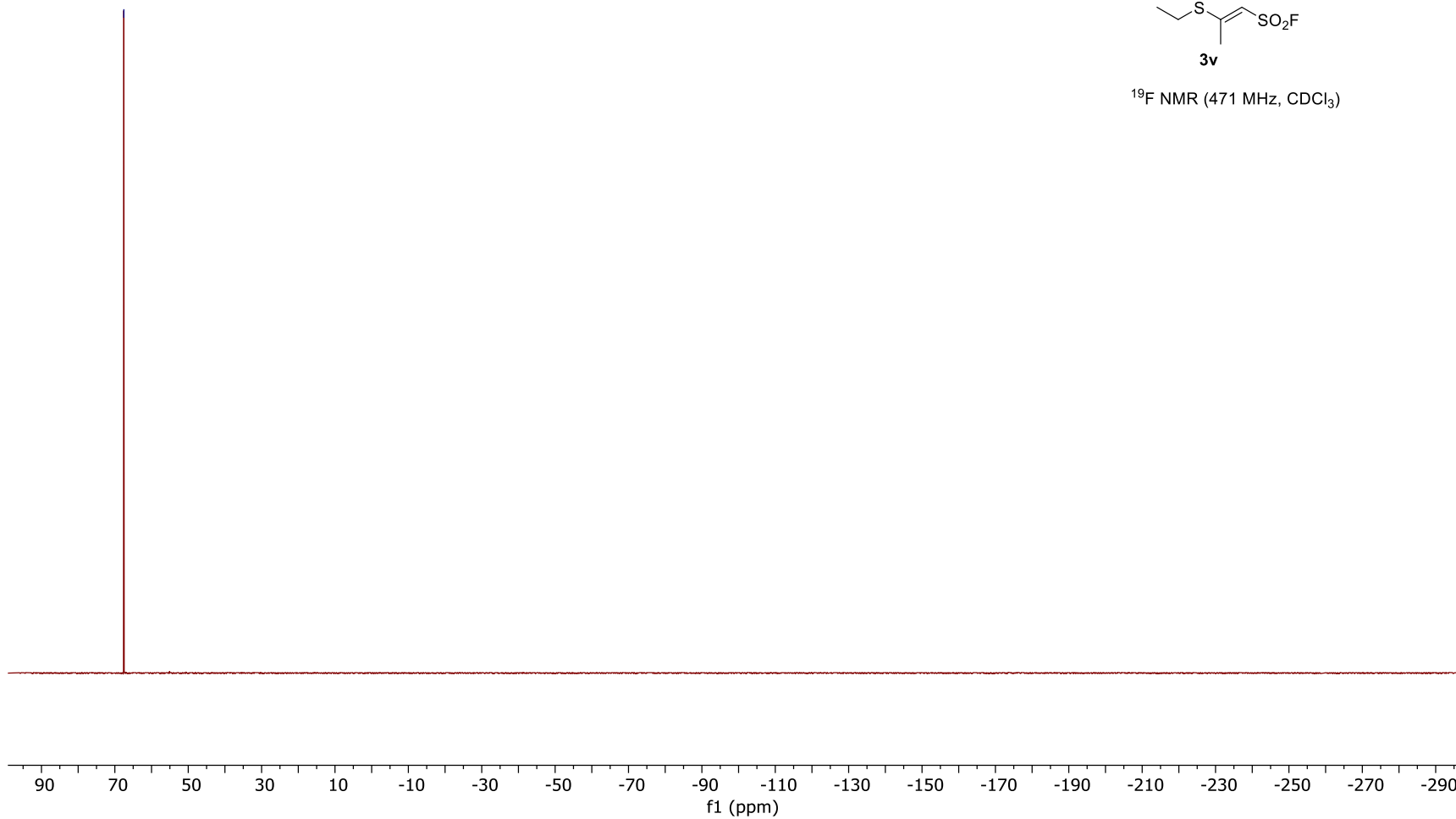


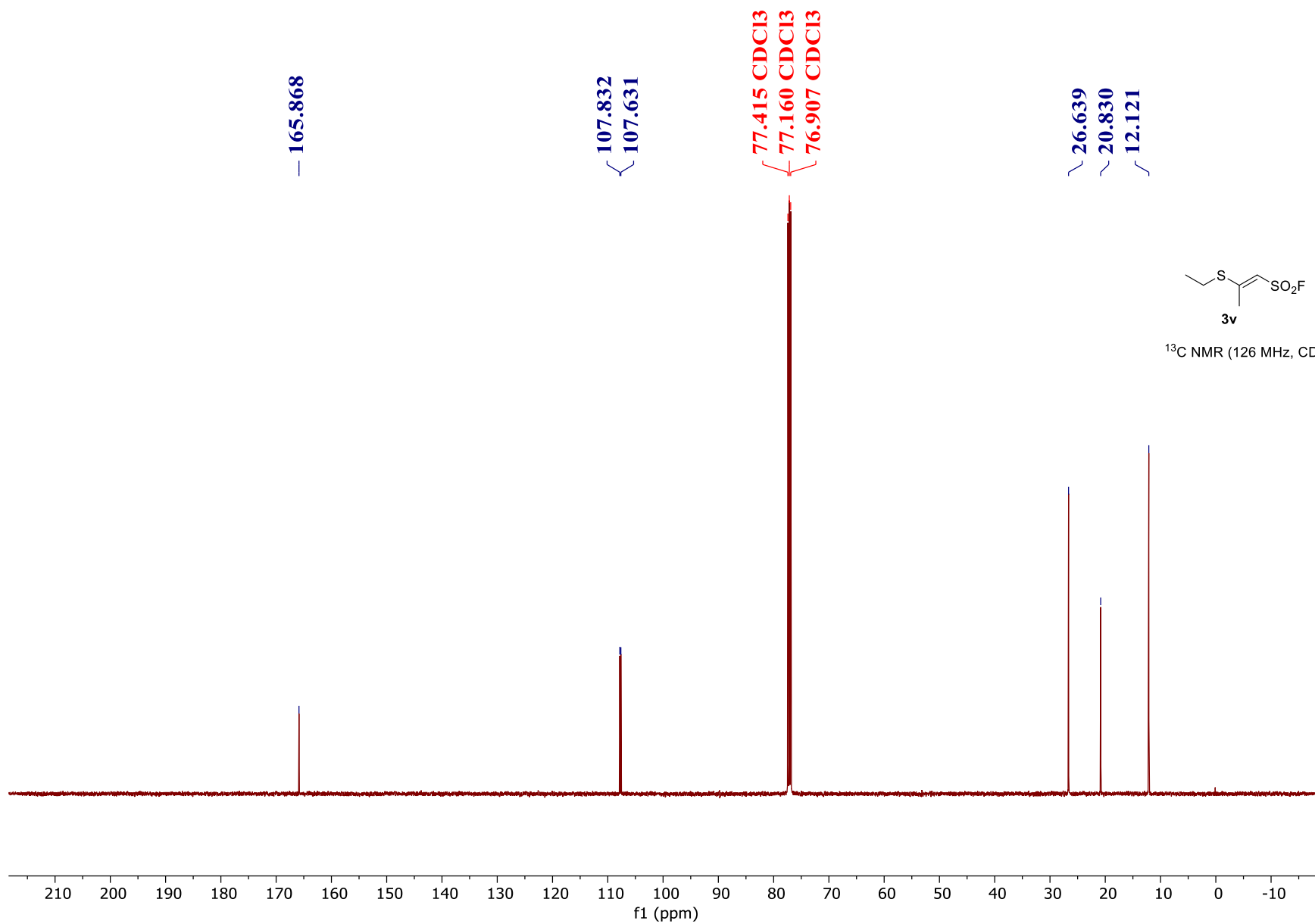


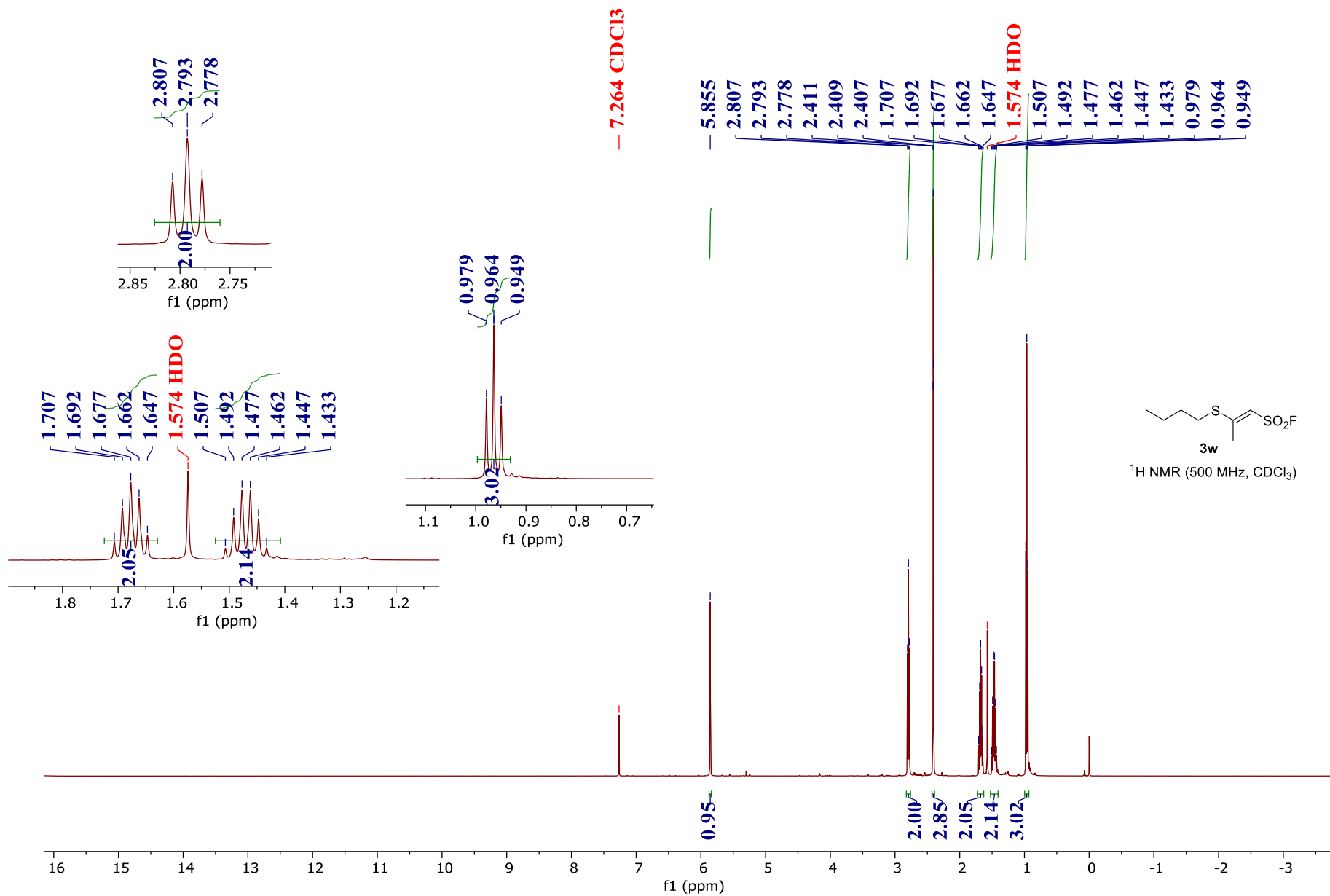
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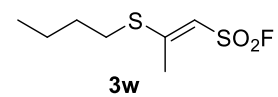
<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)



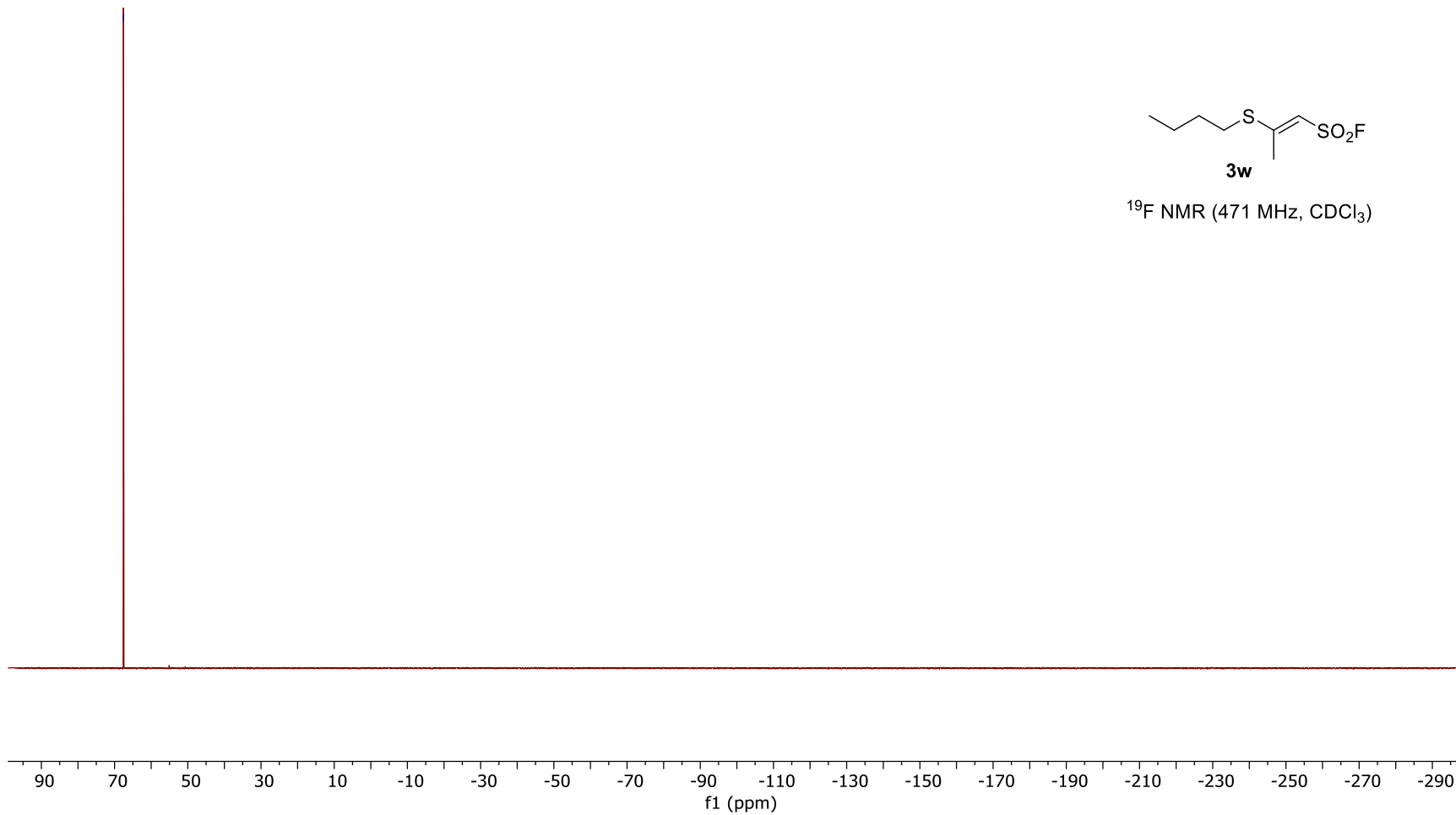


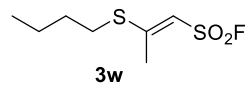


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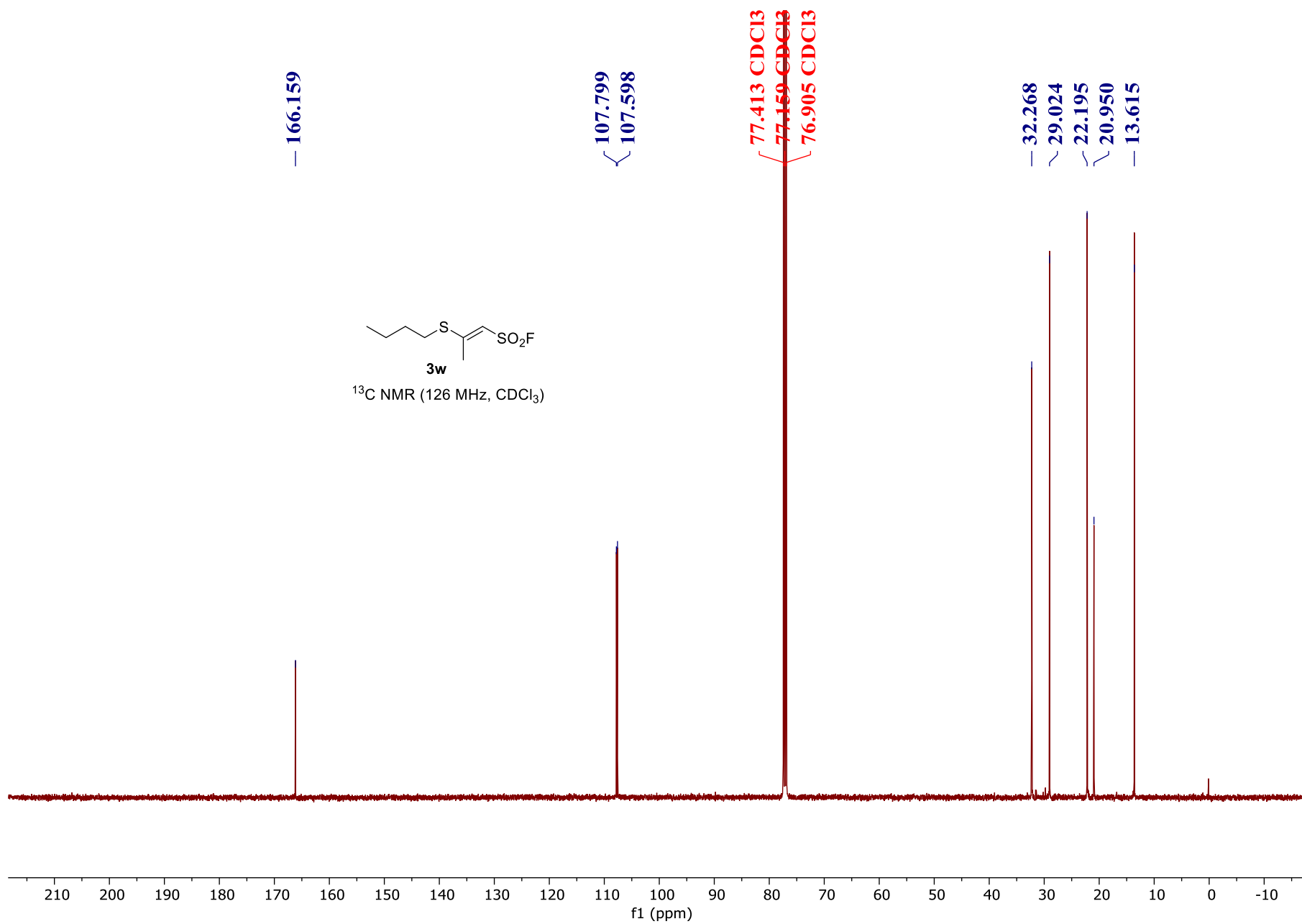


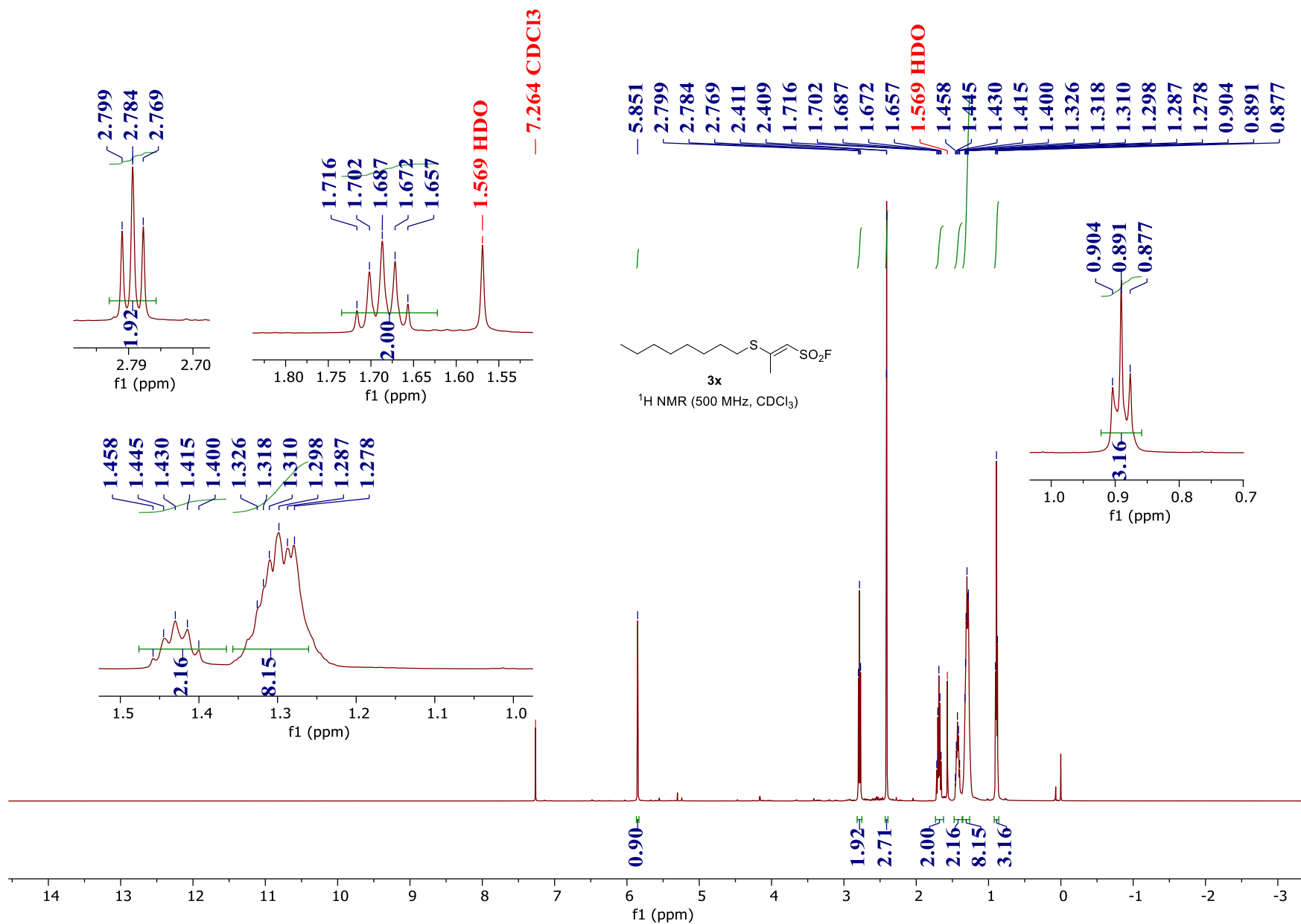
<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)



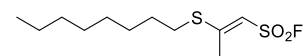


<sup>13</sup>C NMR (126 MHz, CDCl<sub>3</sub>)



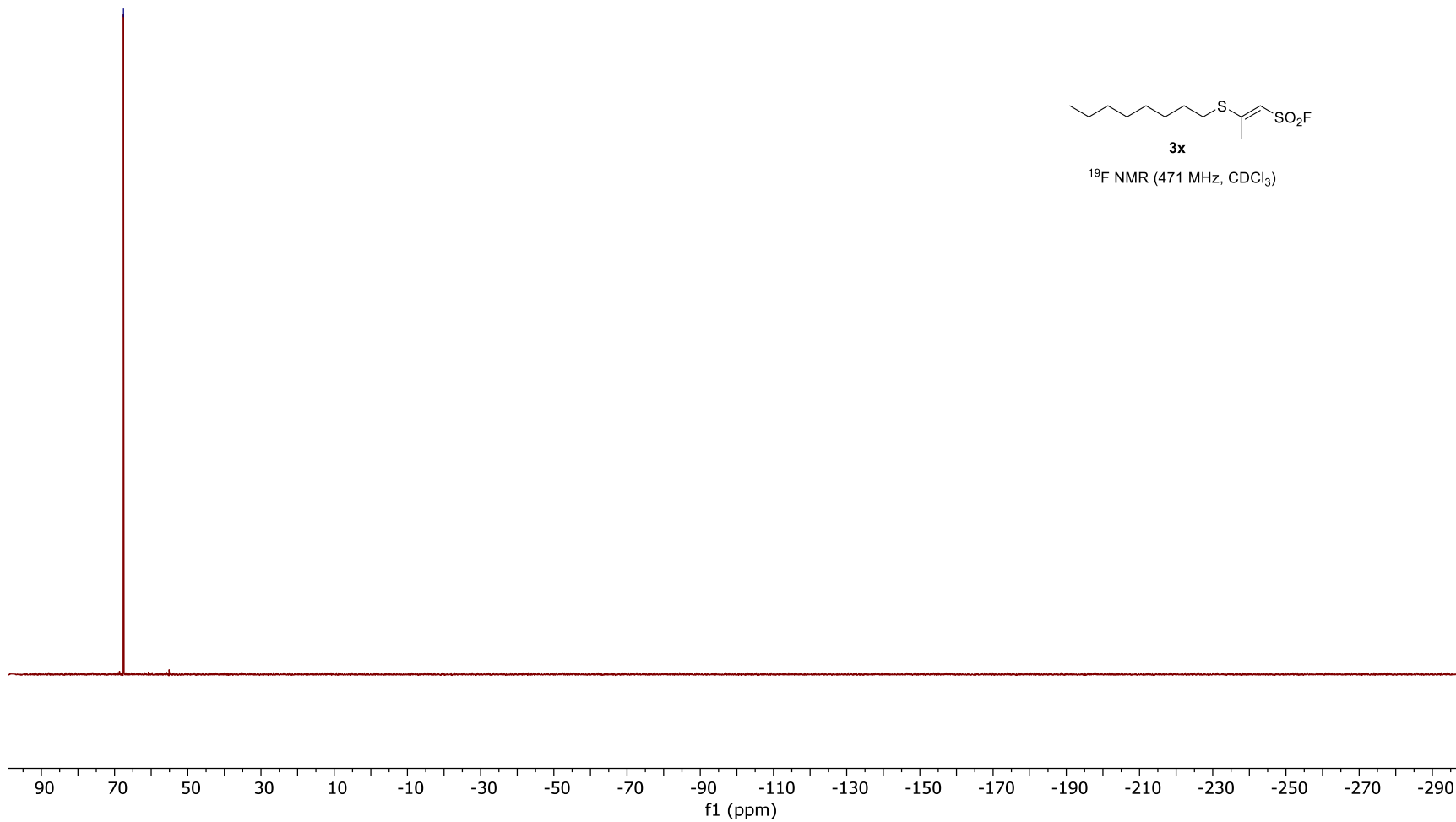


— 67.570

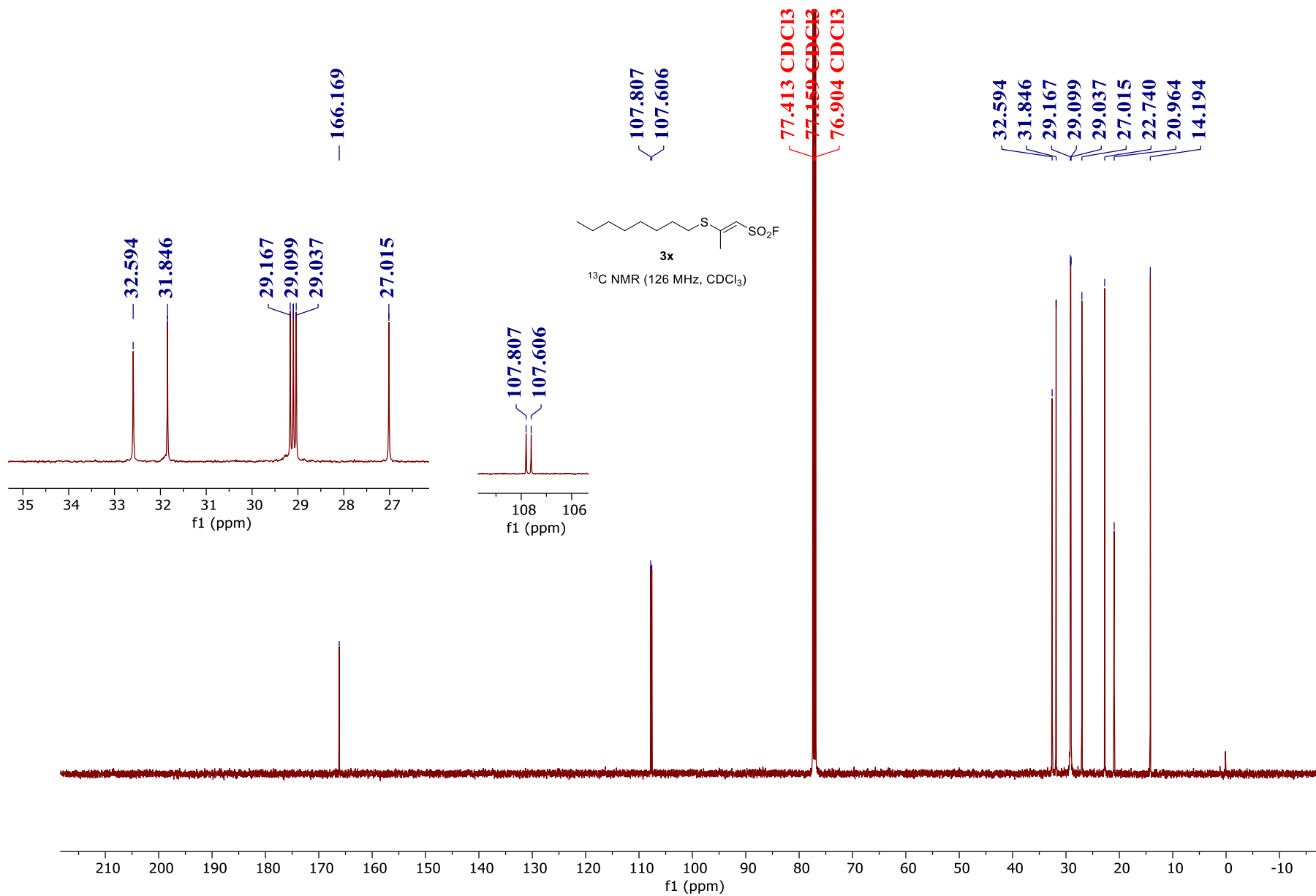


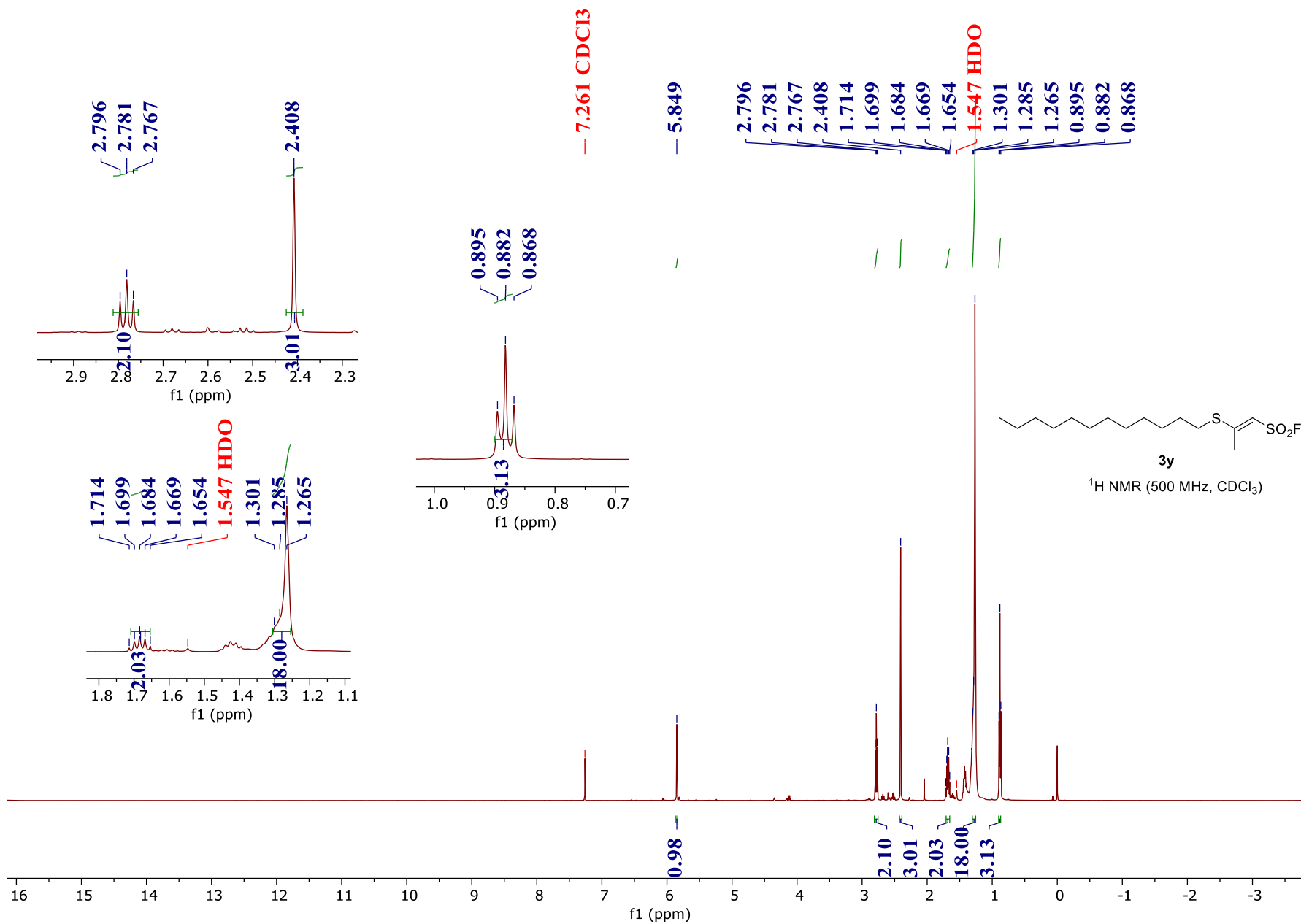
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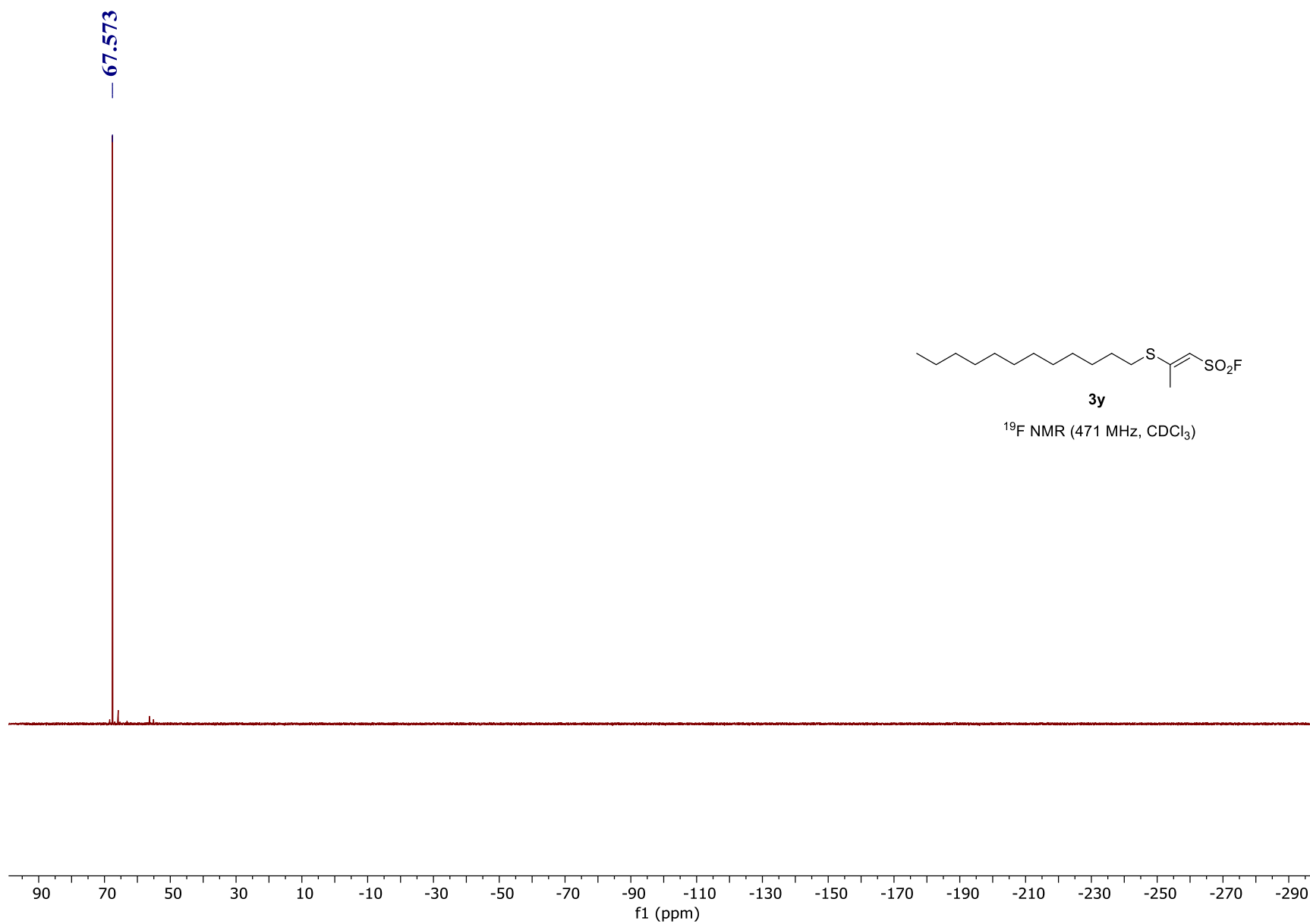
<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)

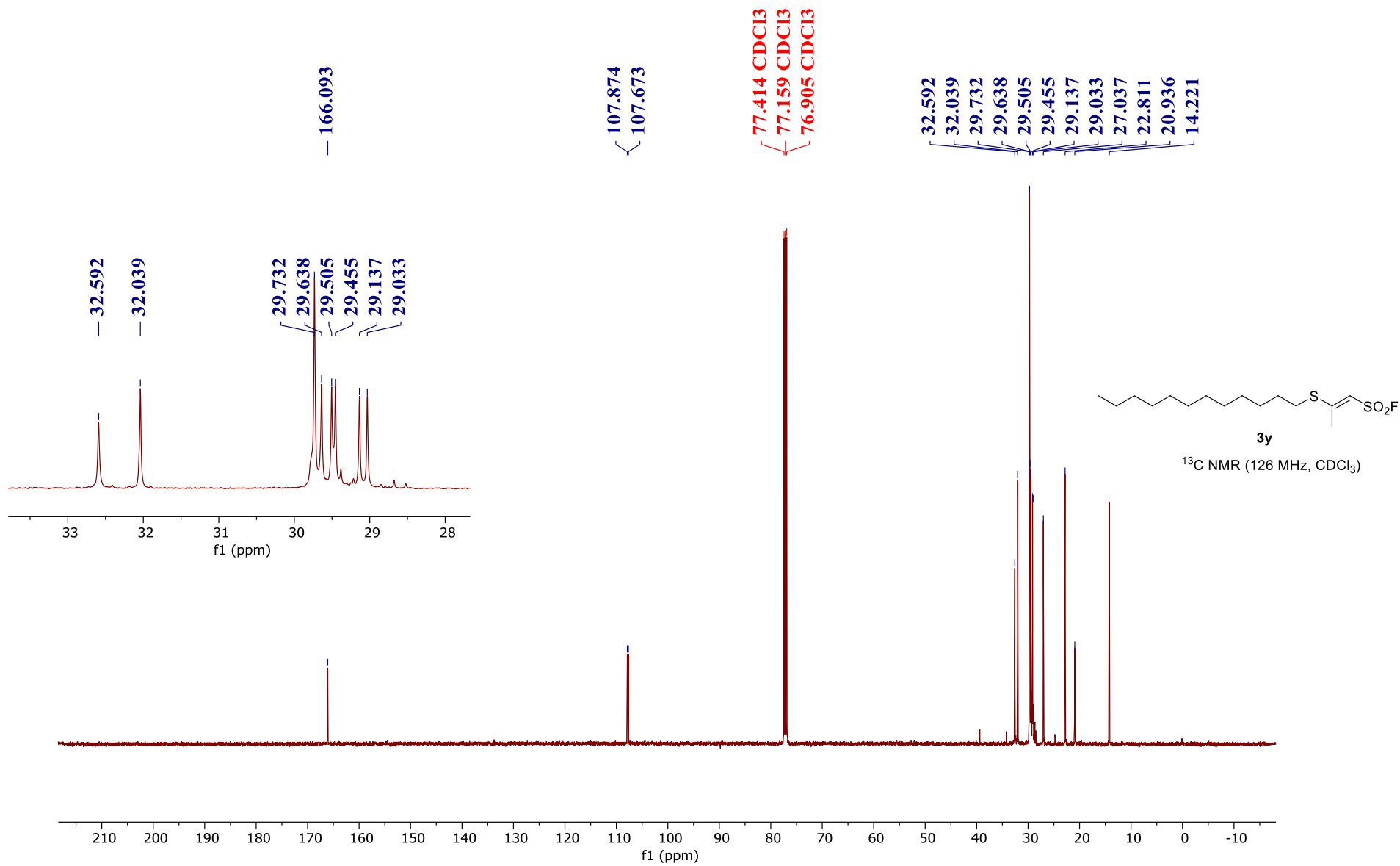


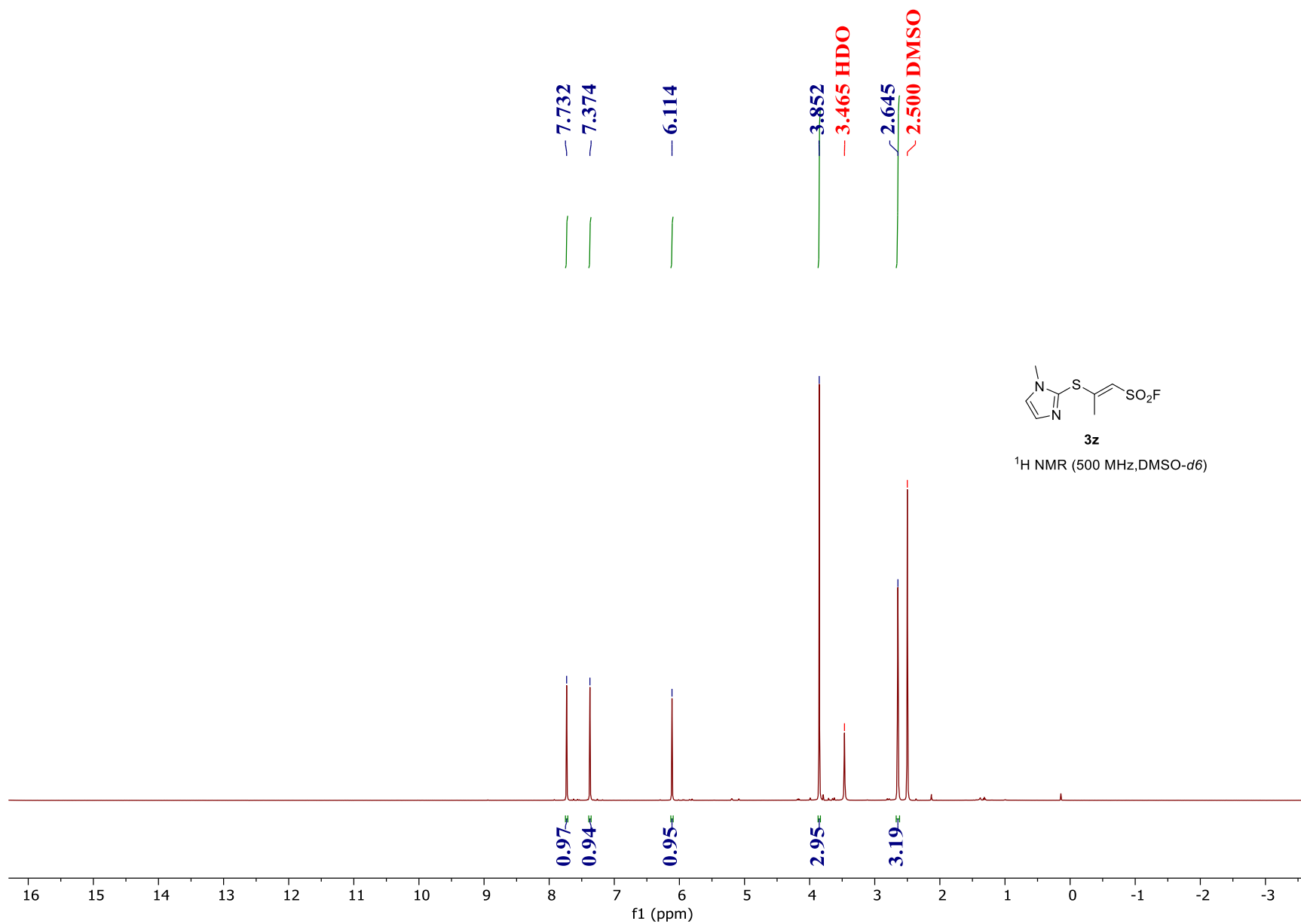


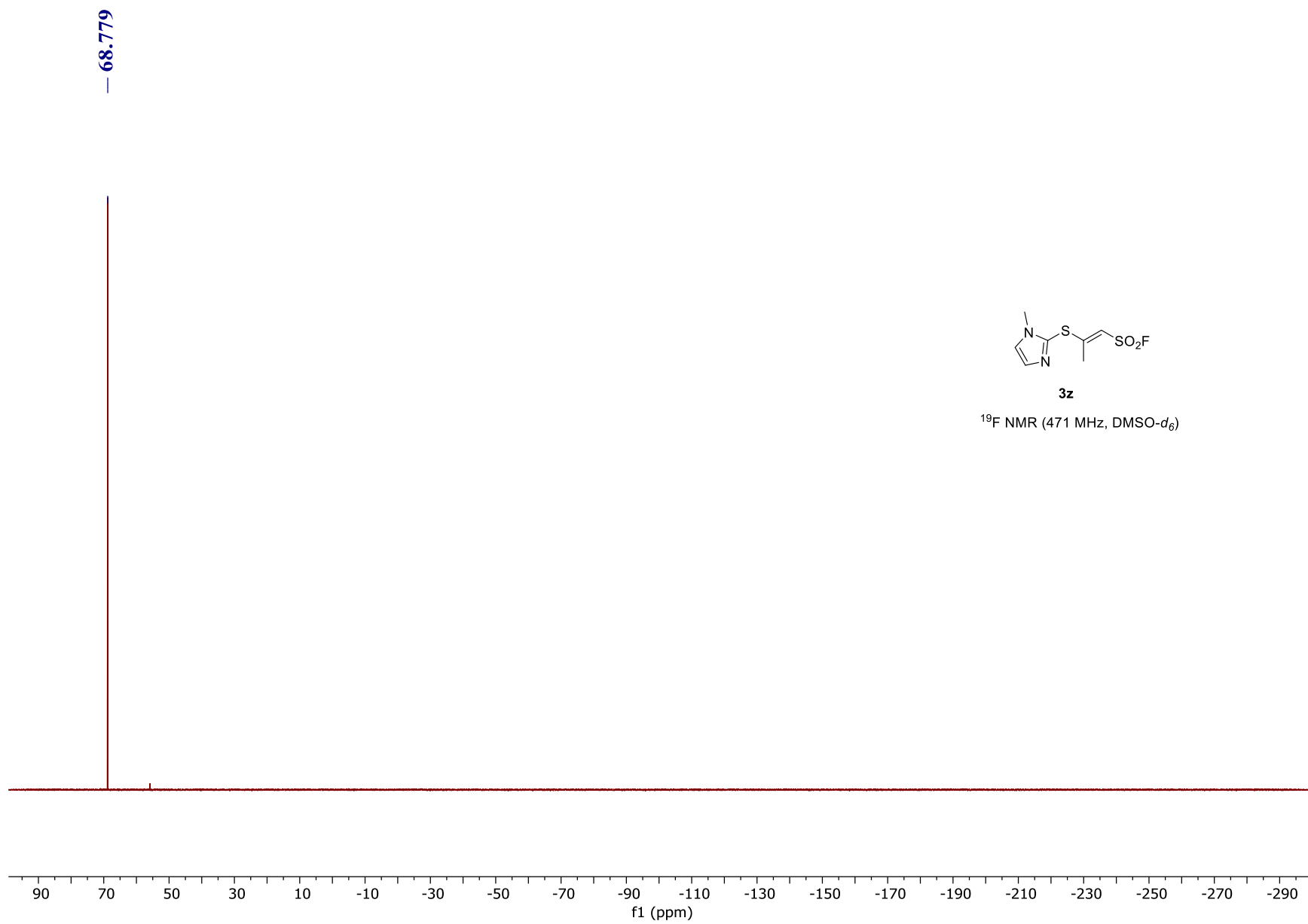


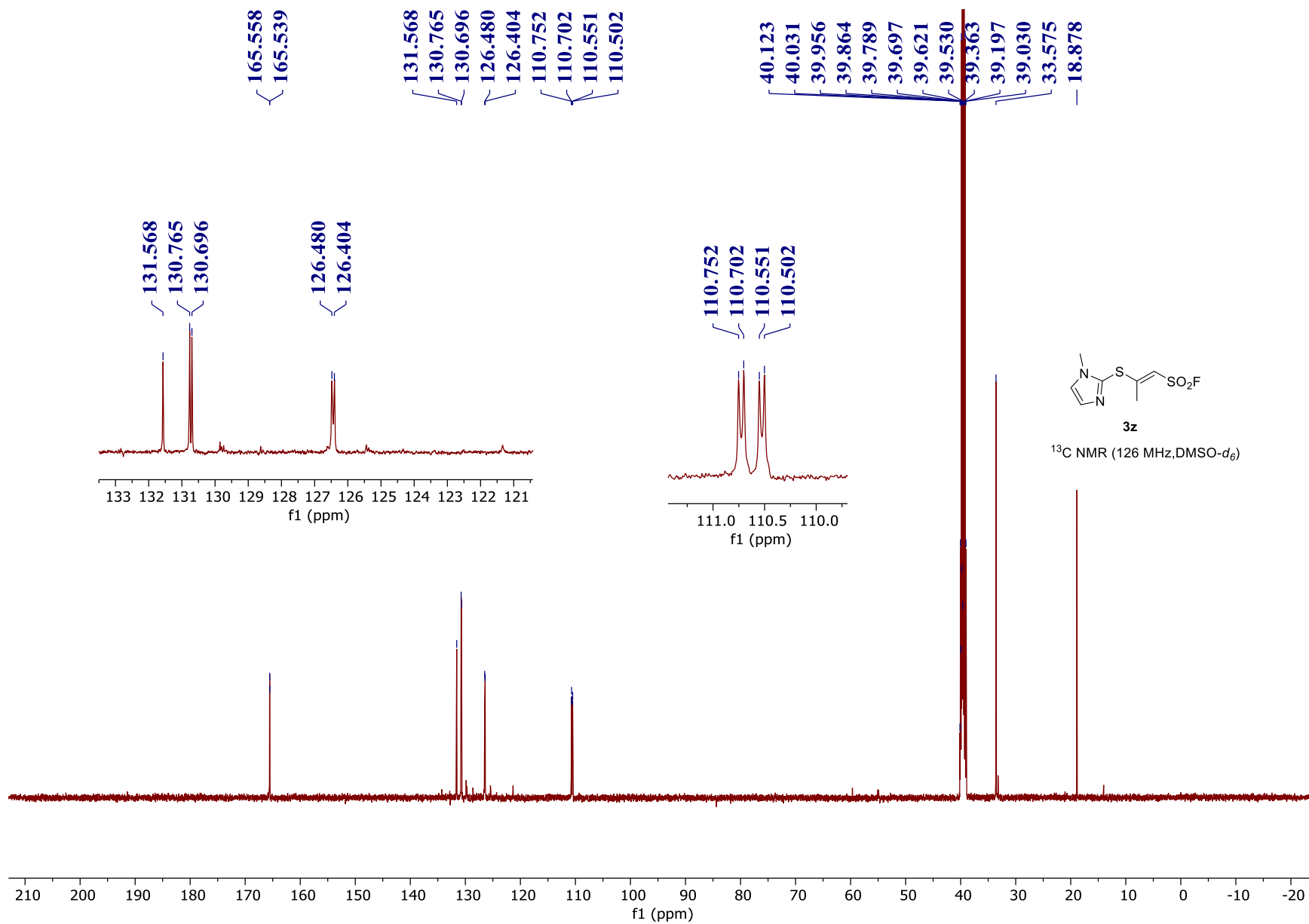


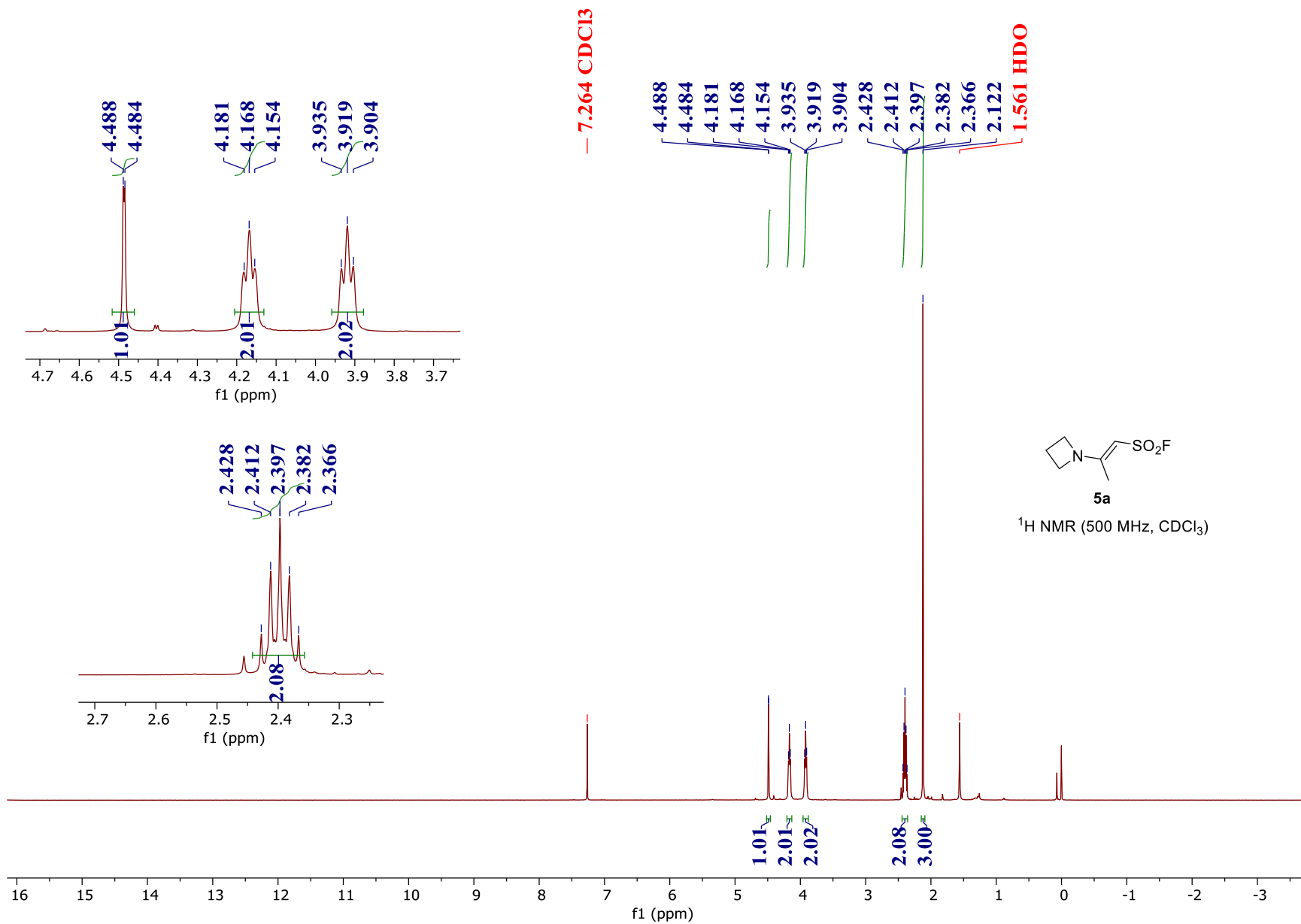




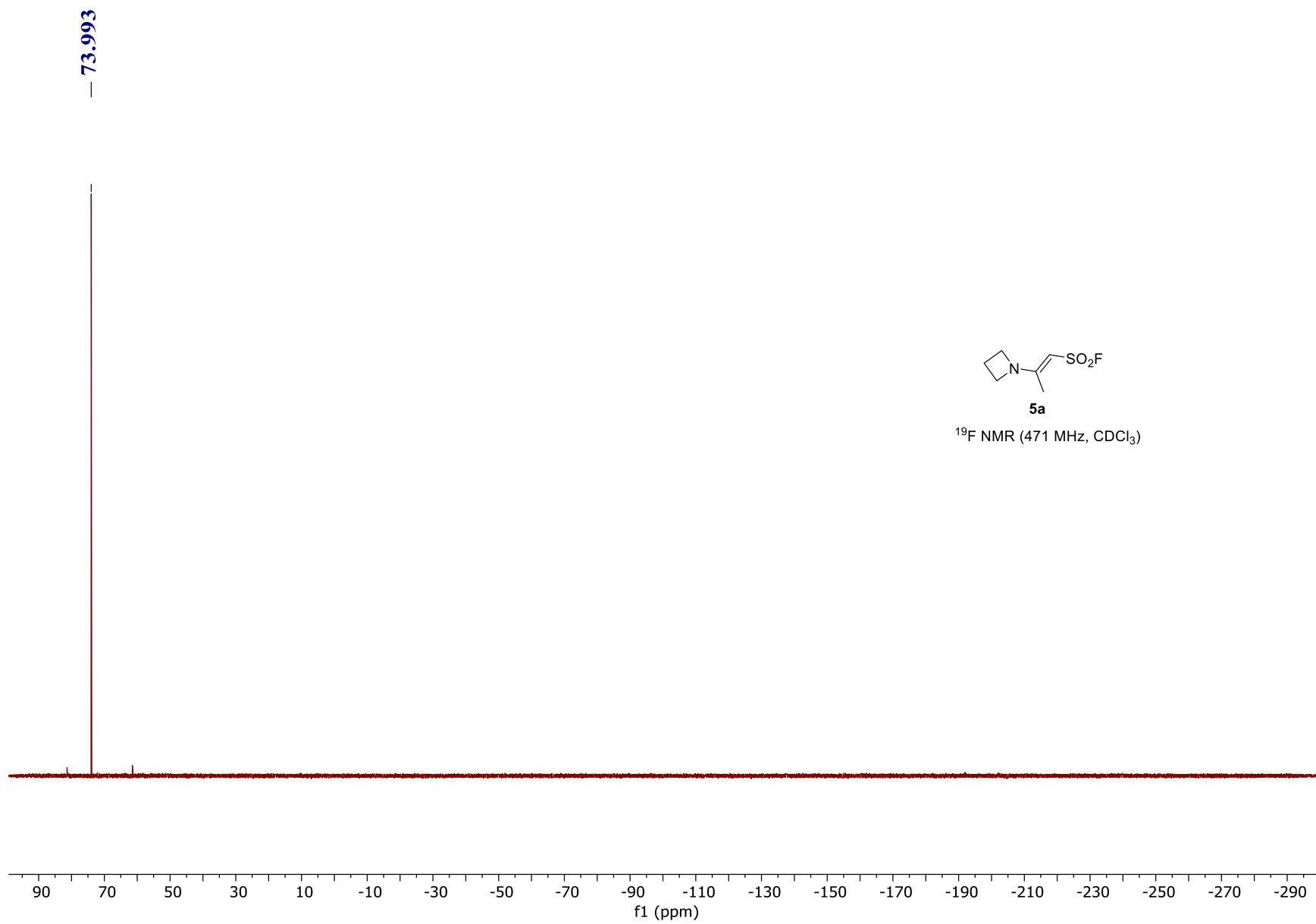


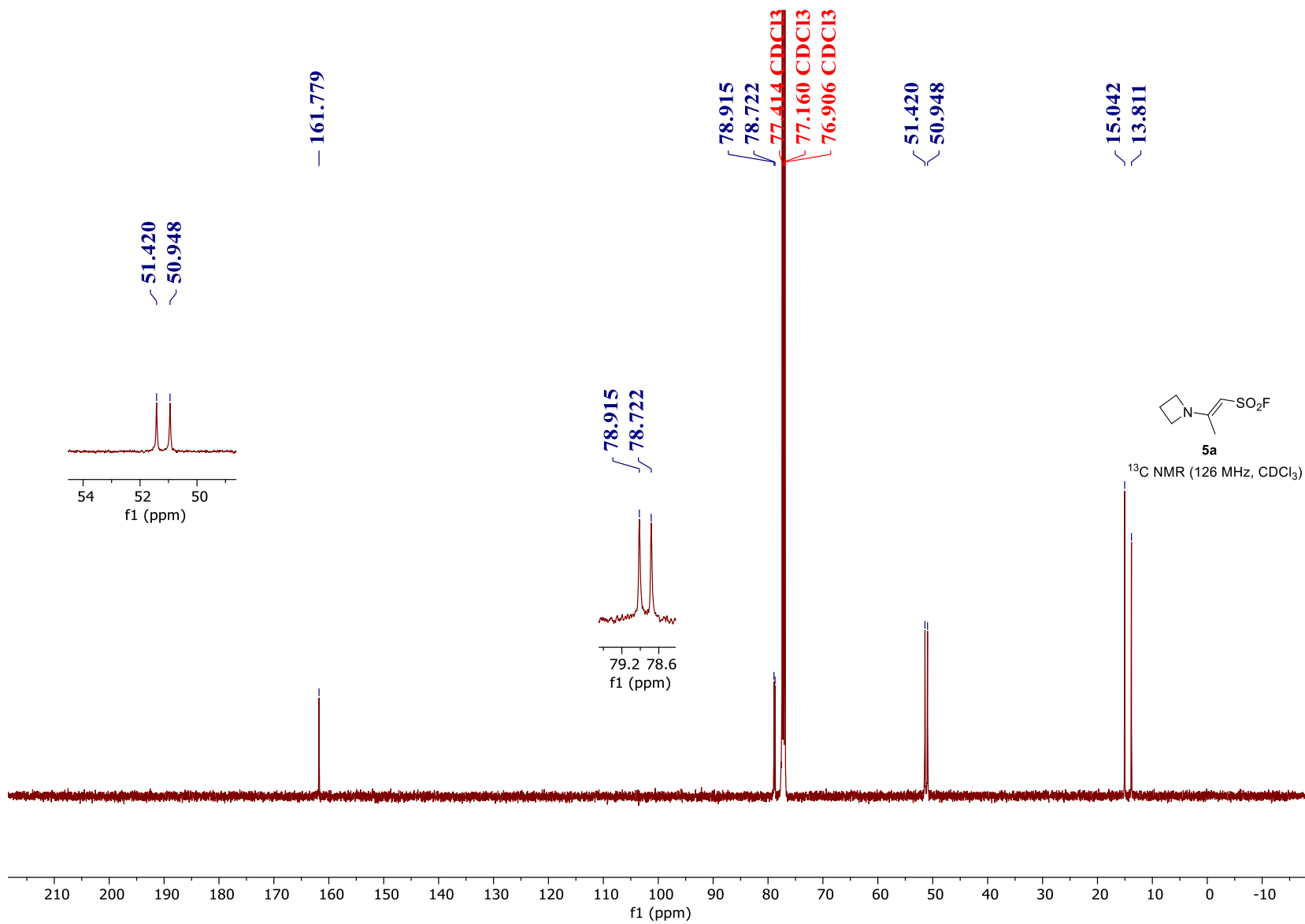


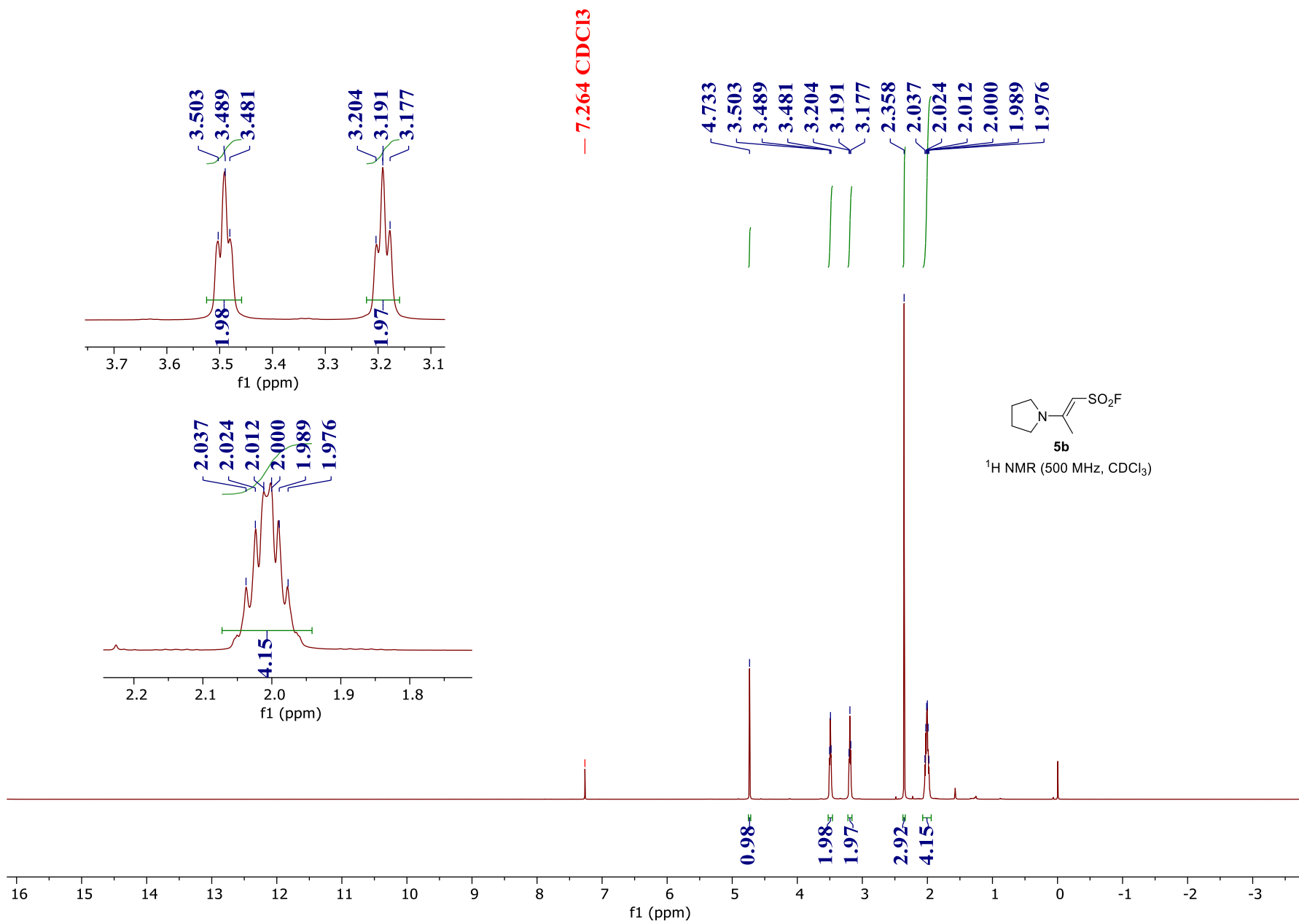




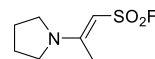






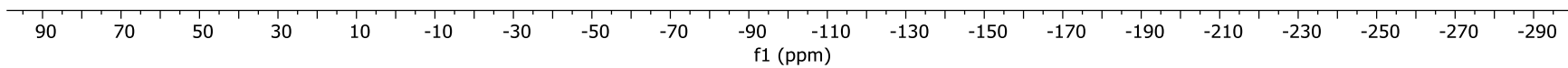


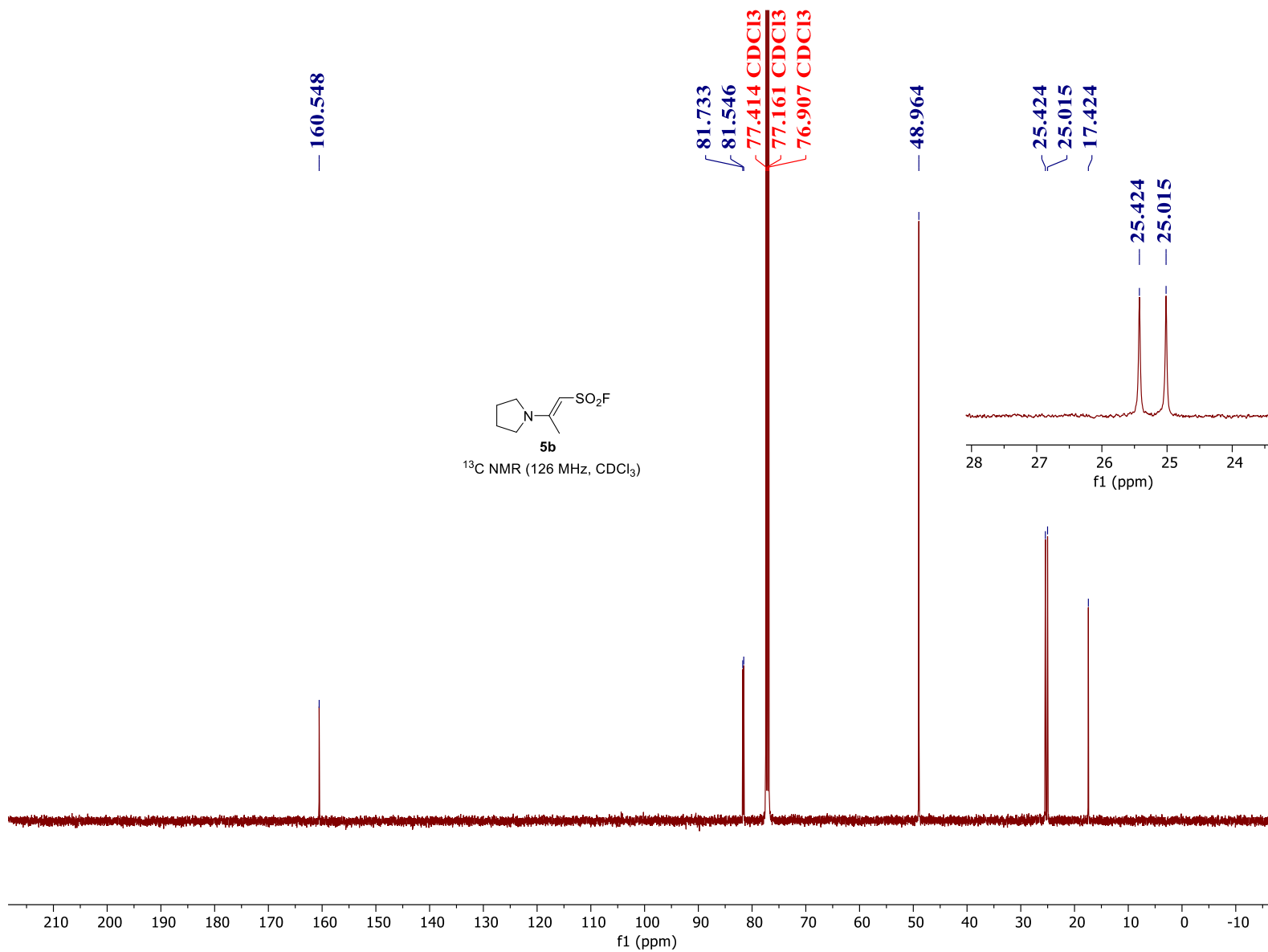
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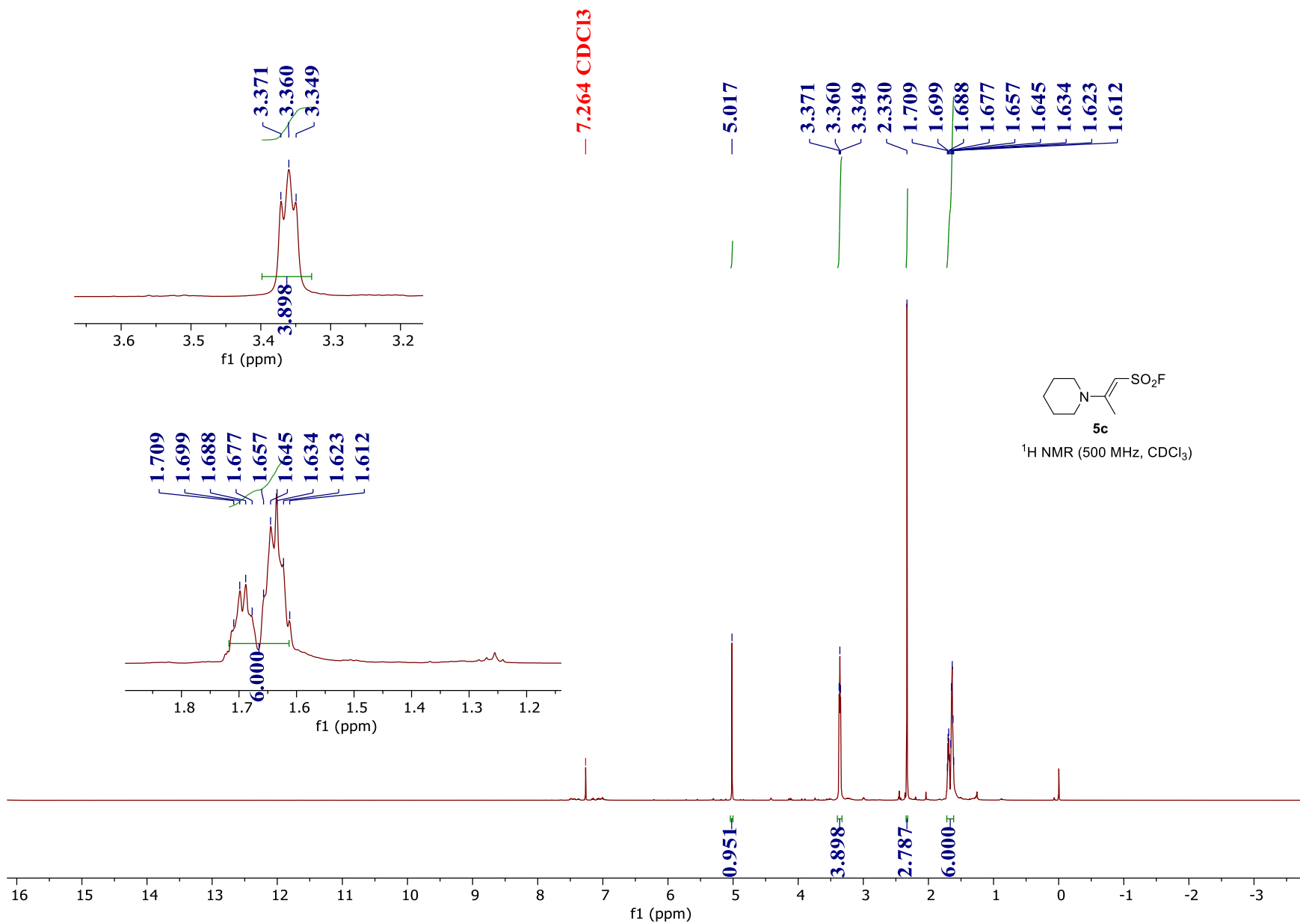


**5b**

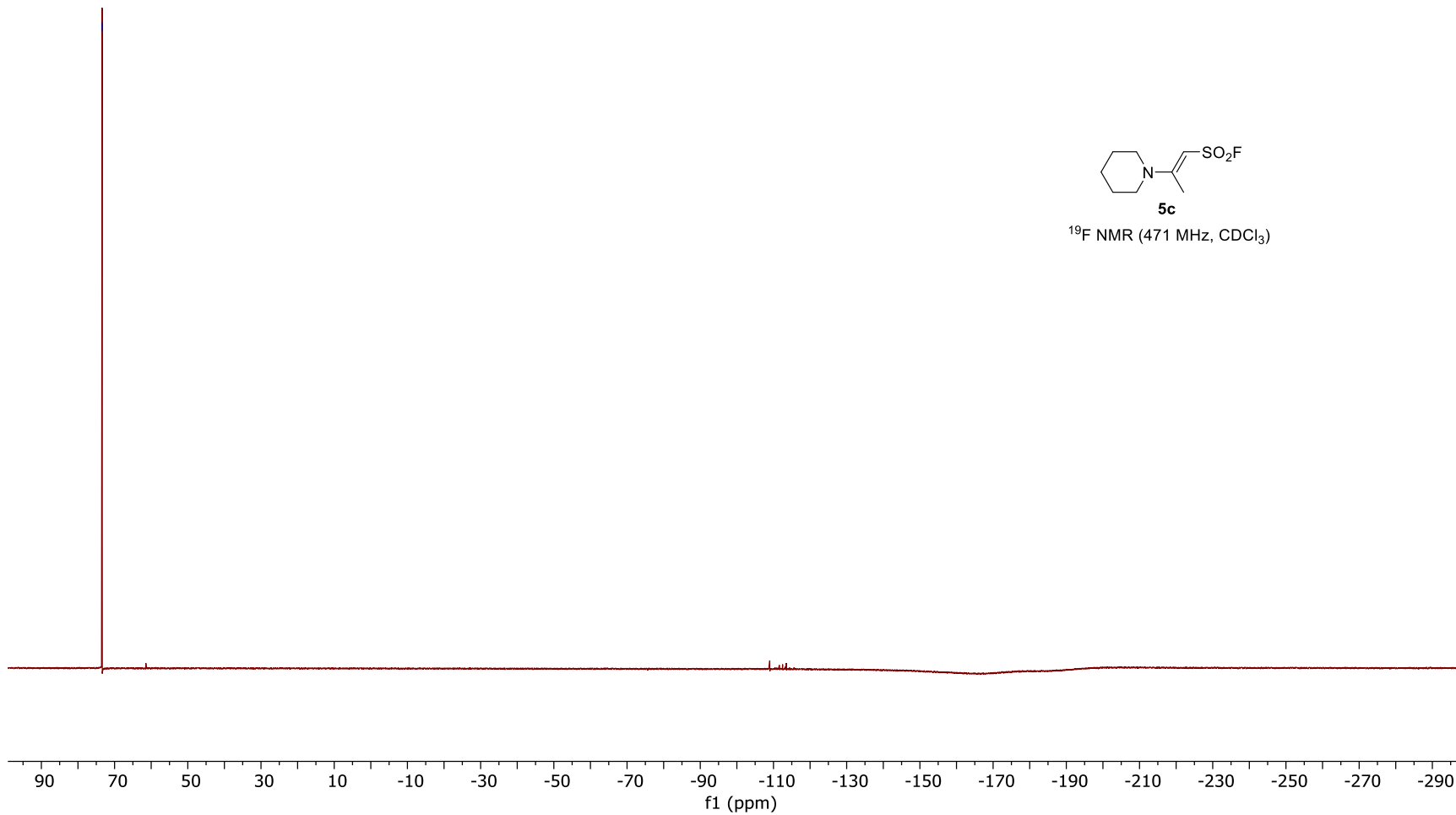
<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)

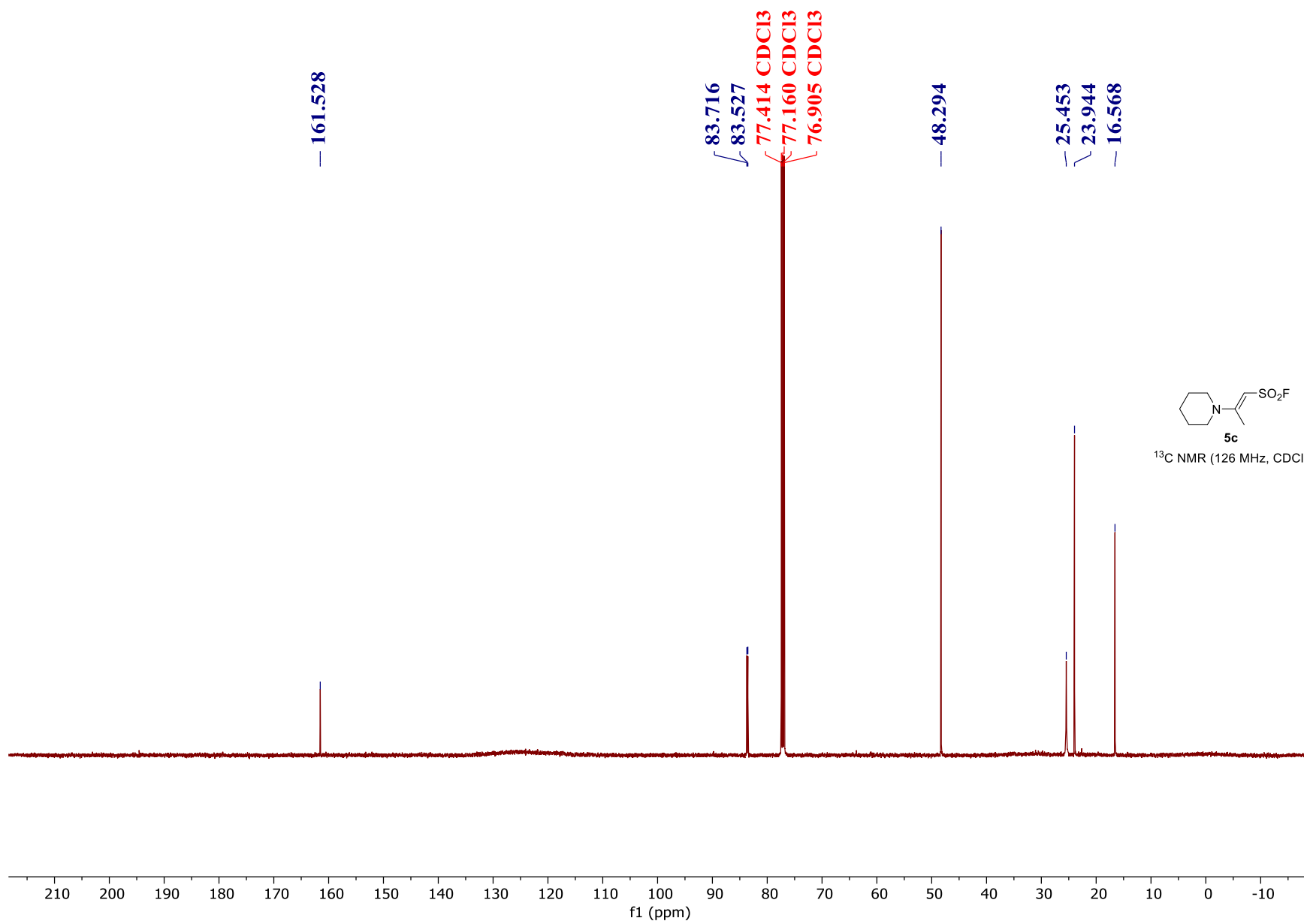




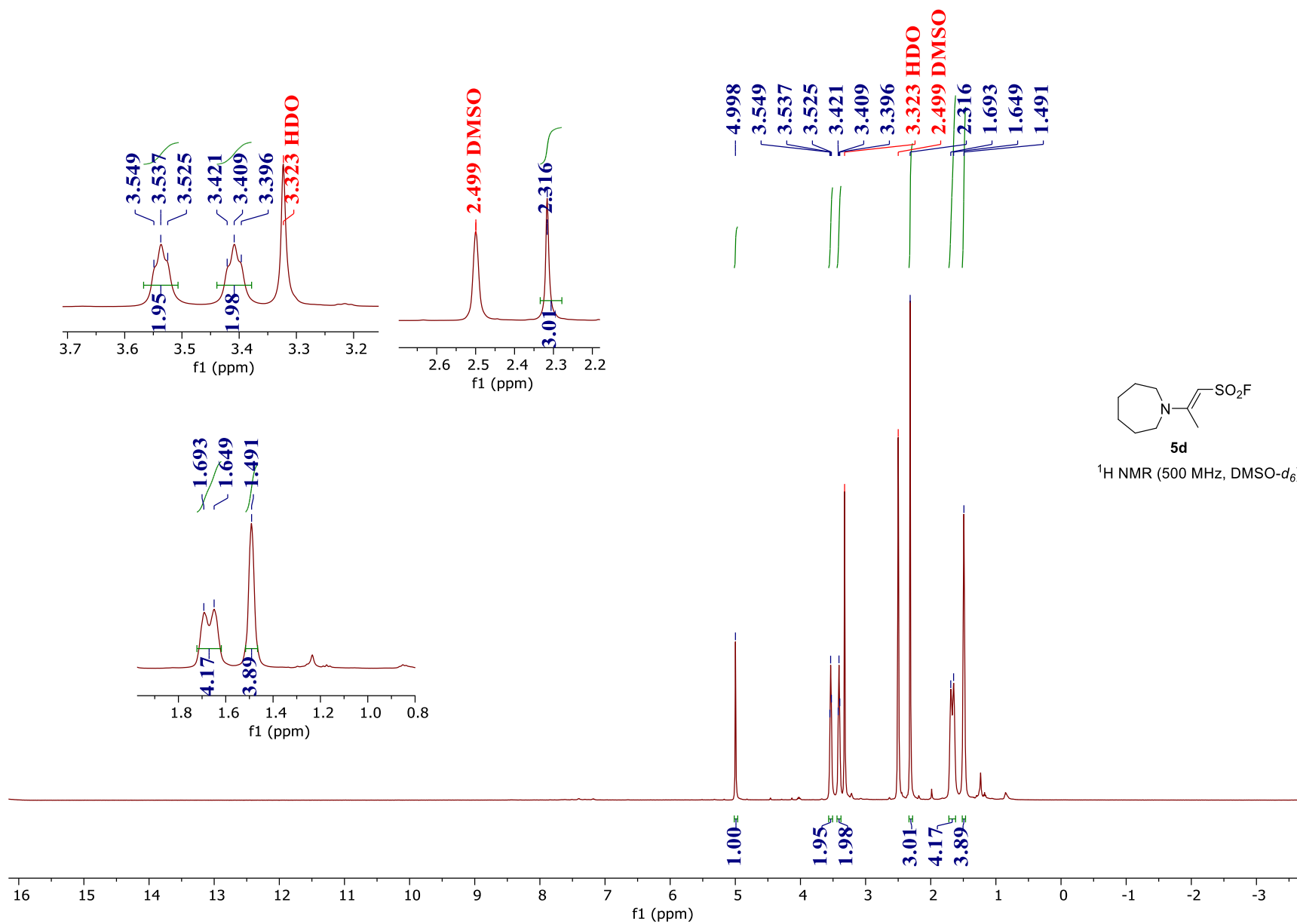


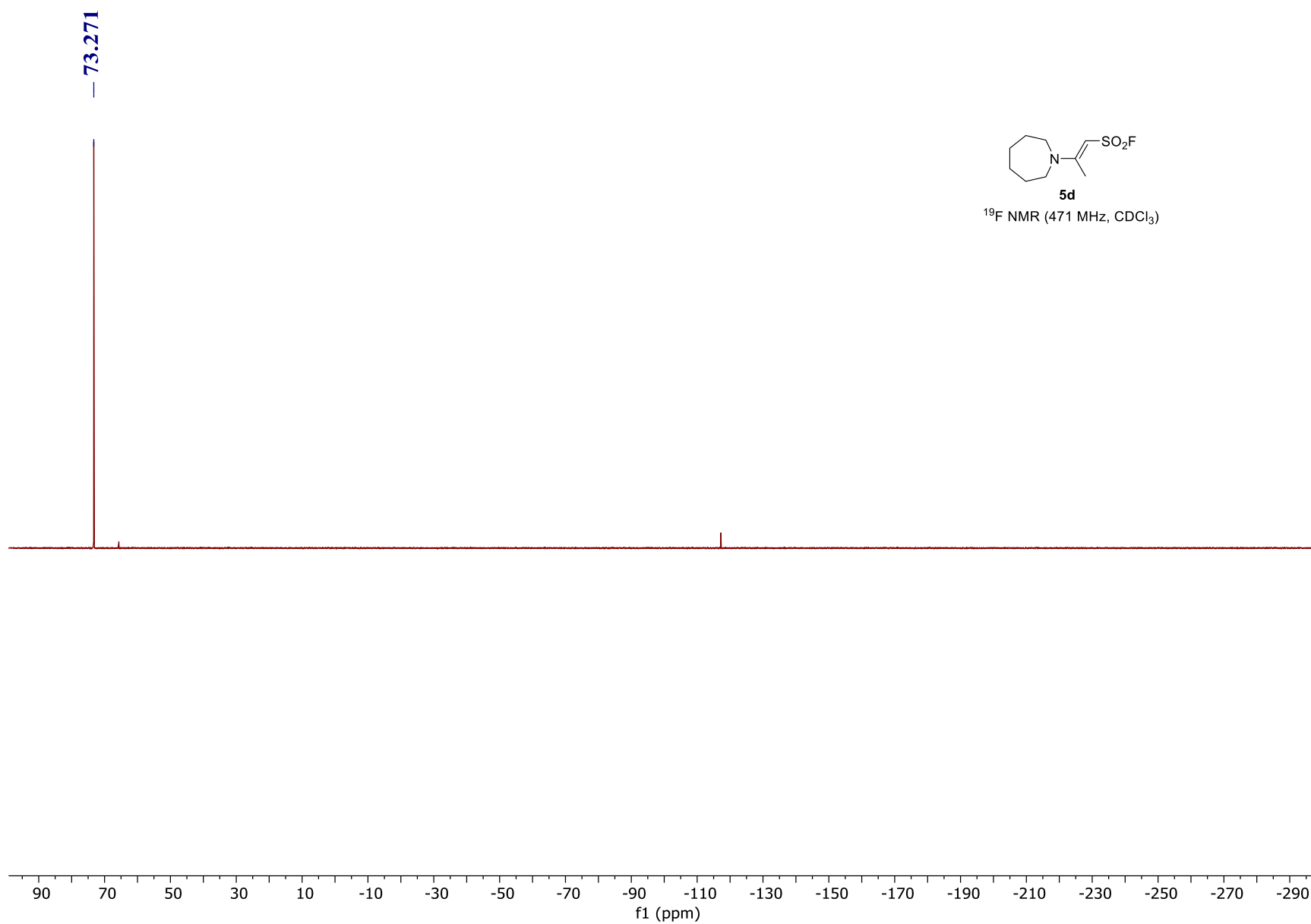
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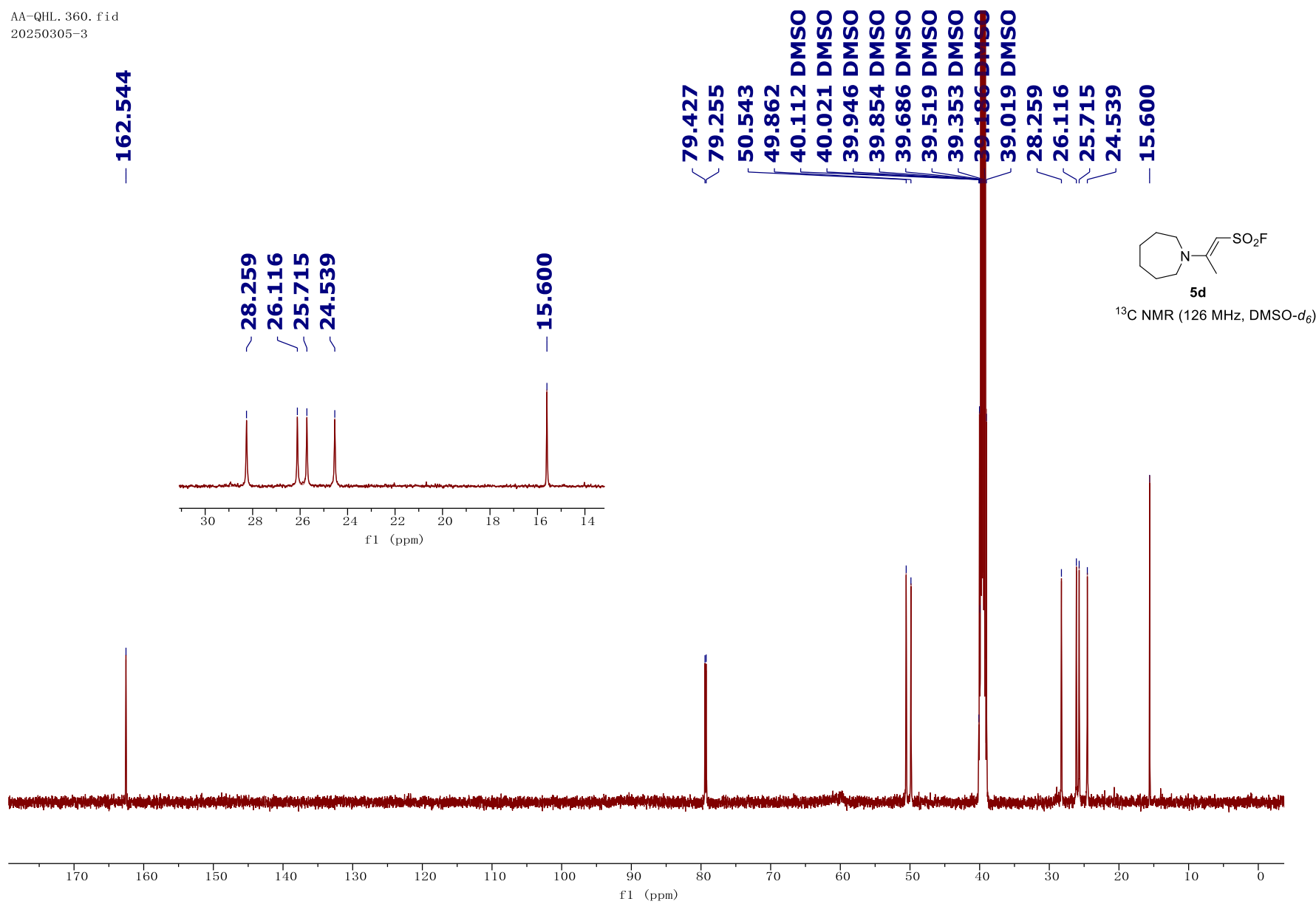


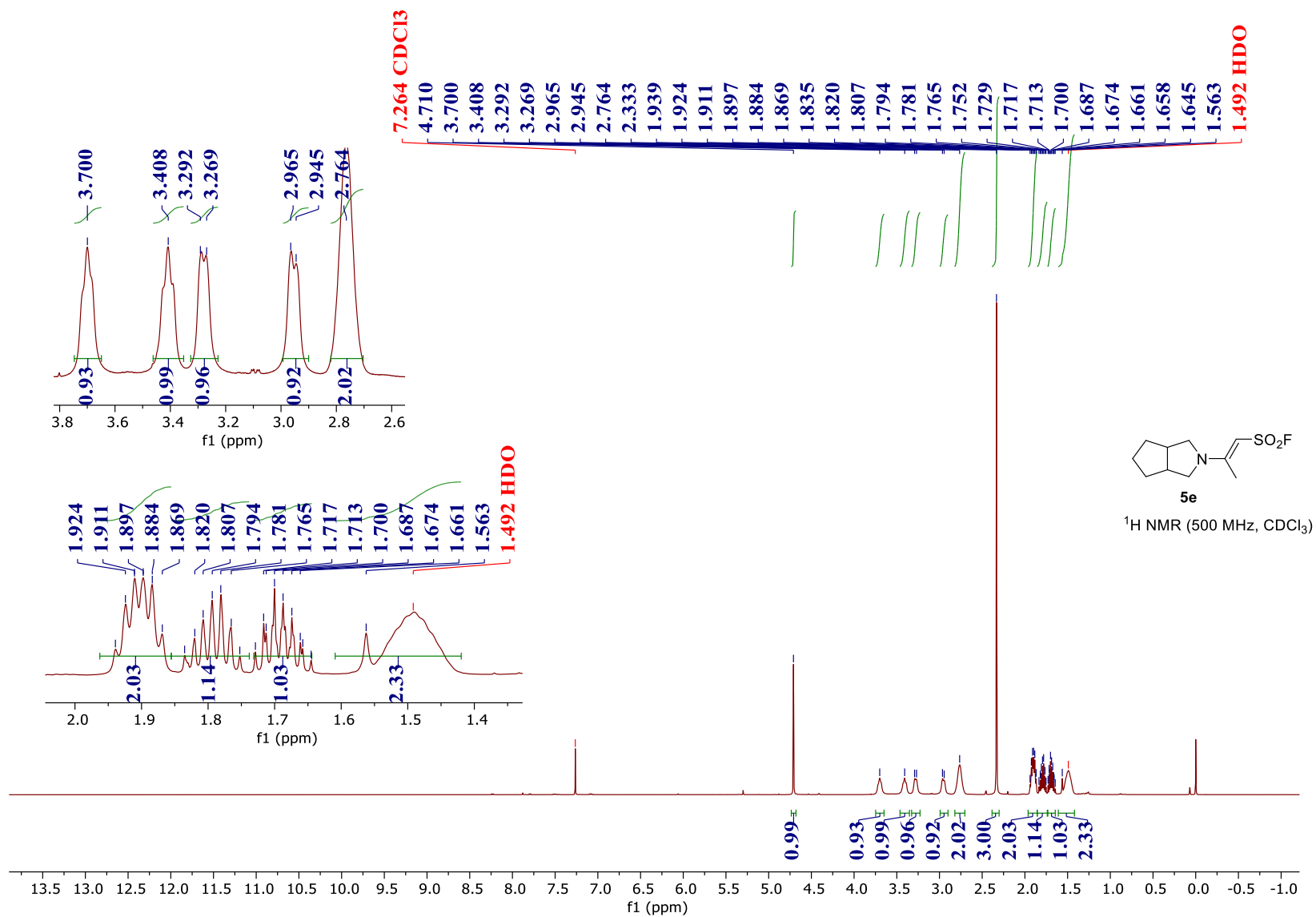


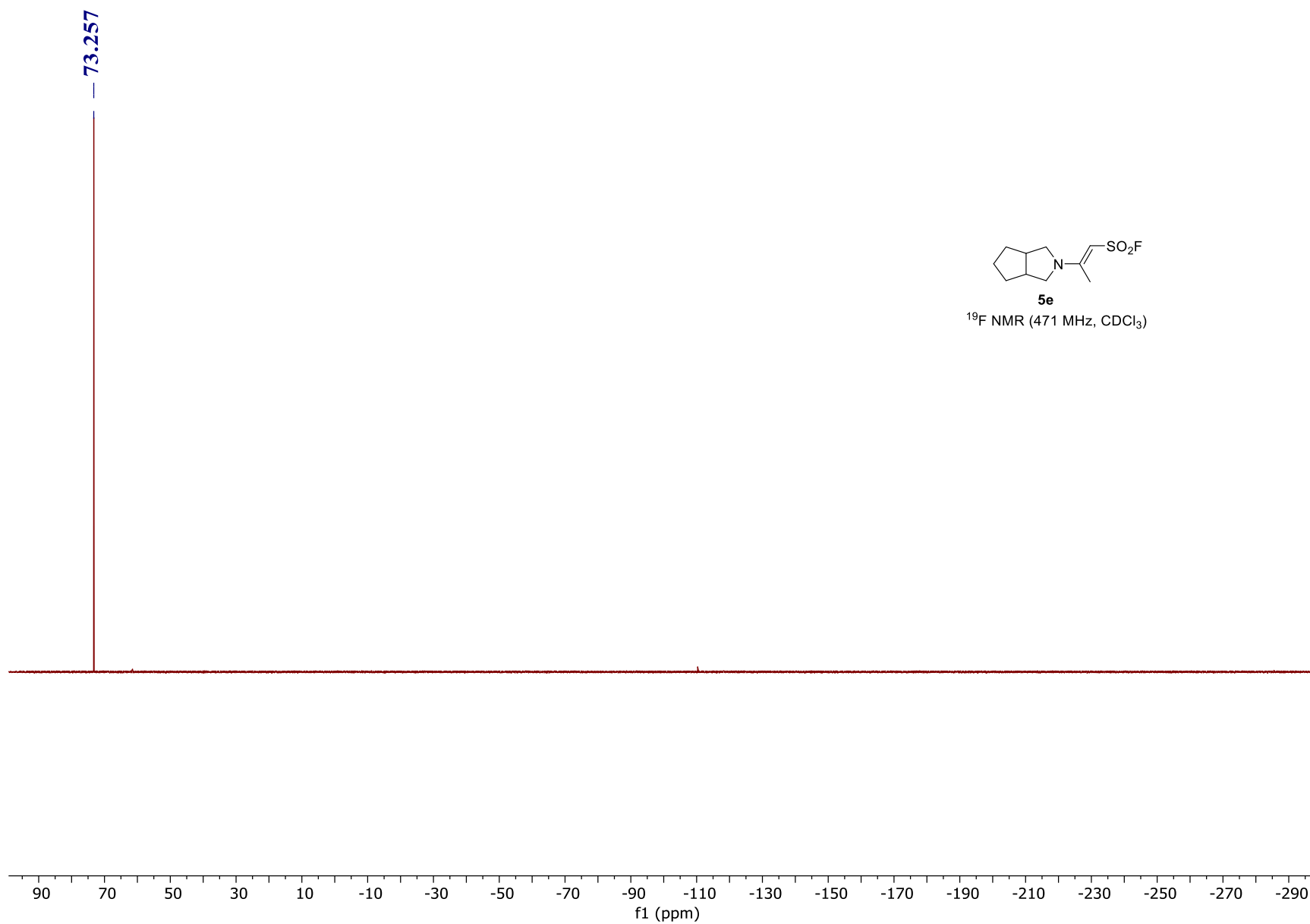




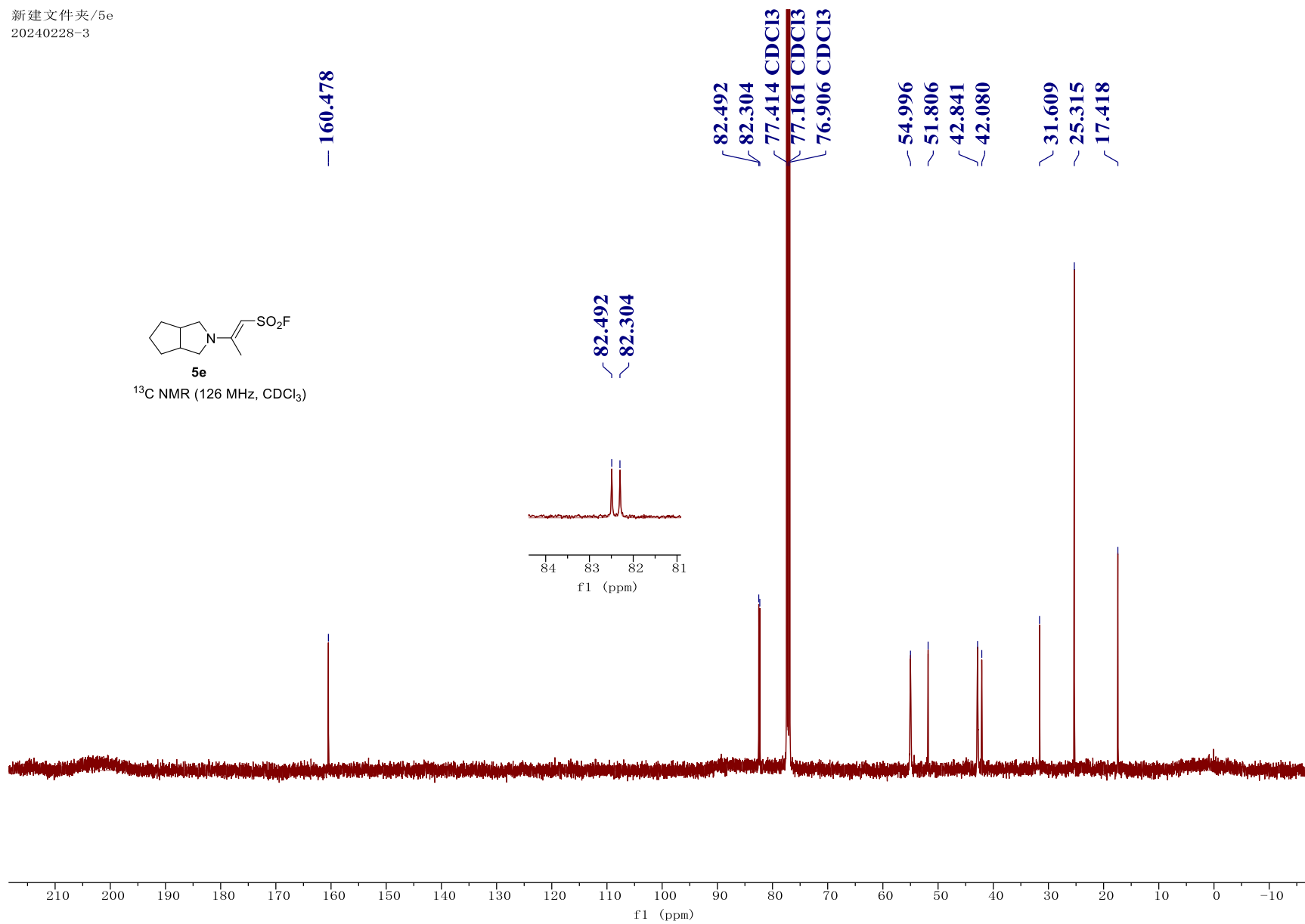
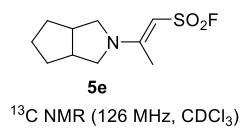
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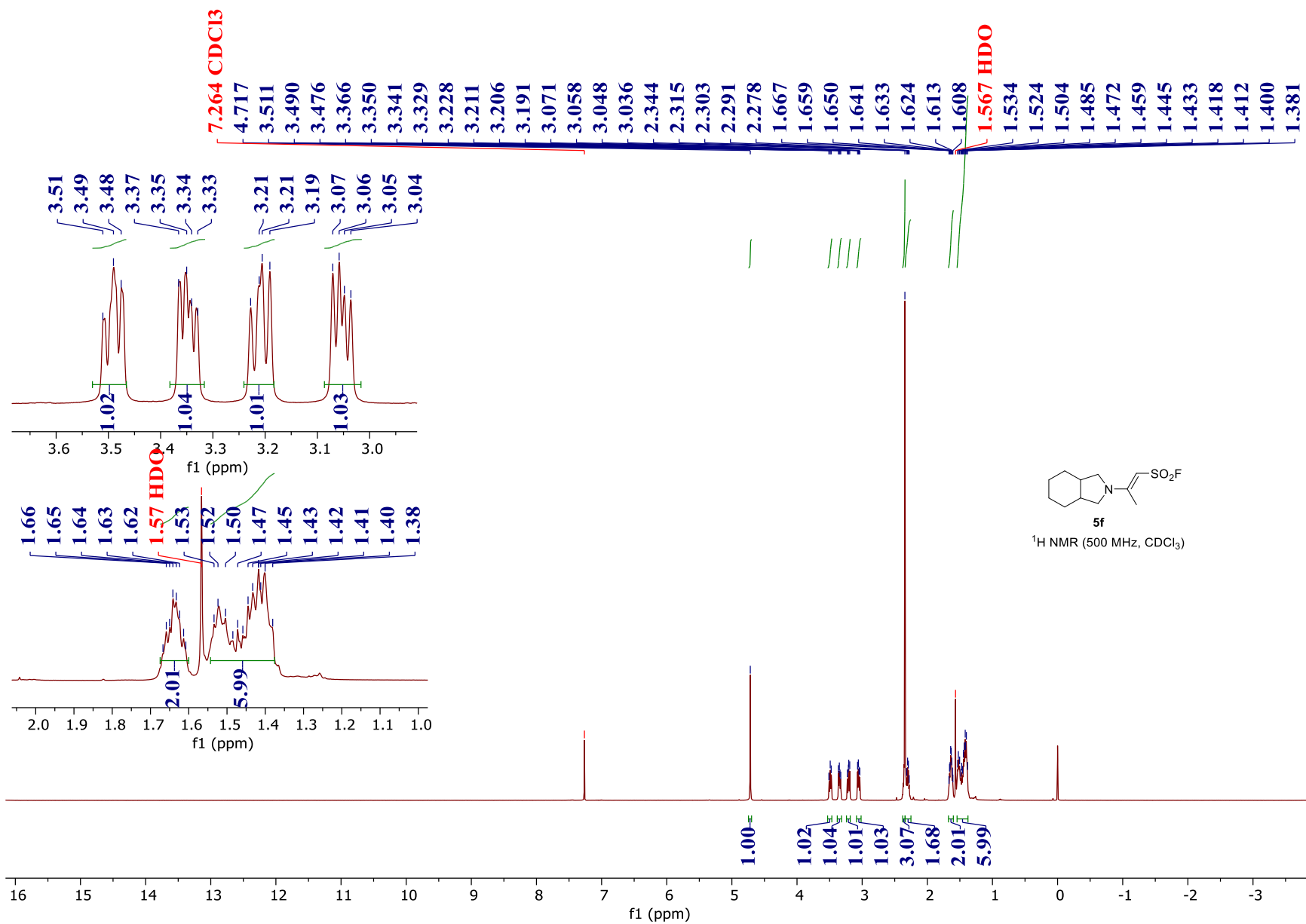




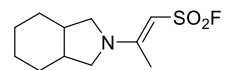


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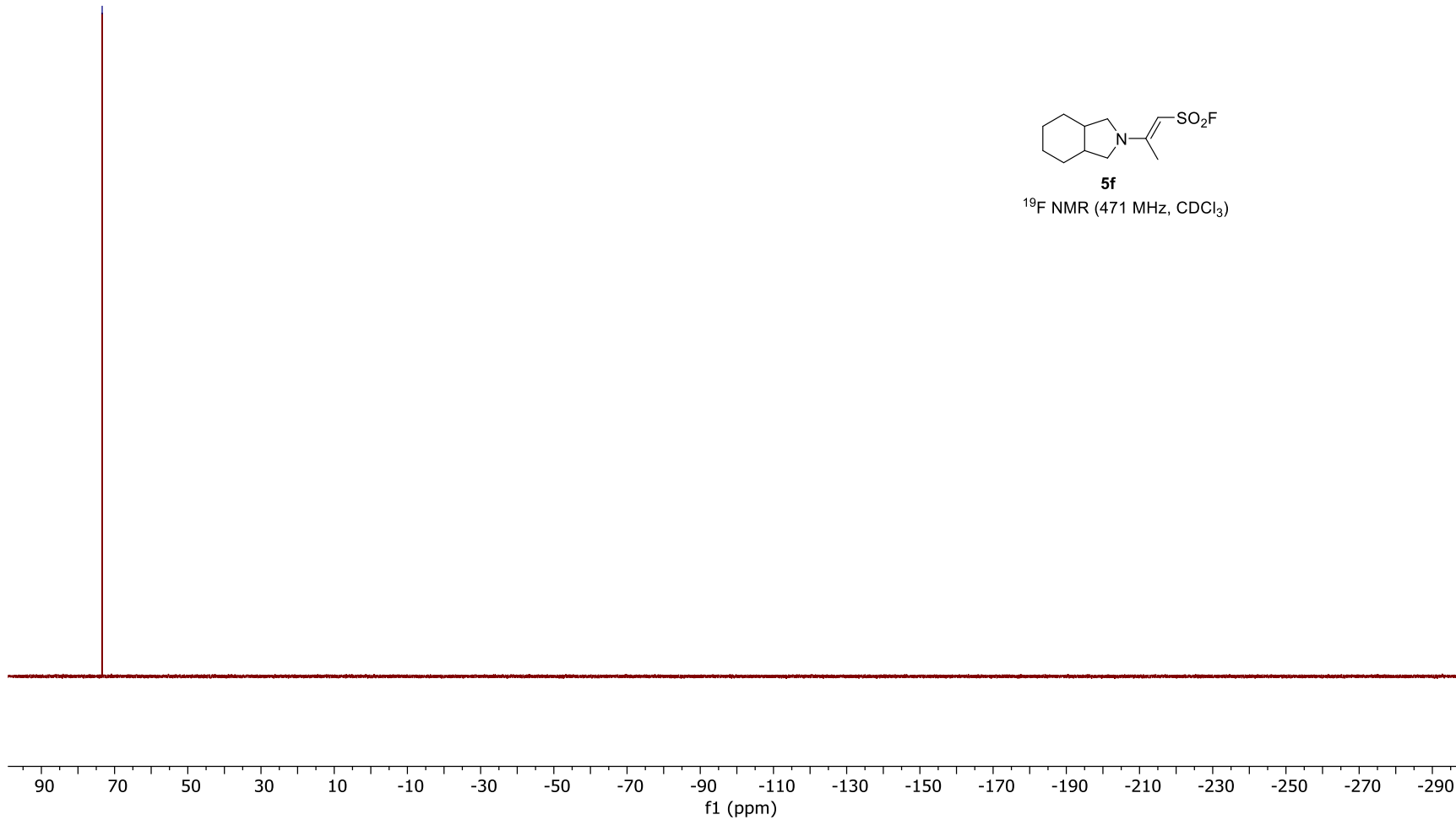


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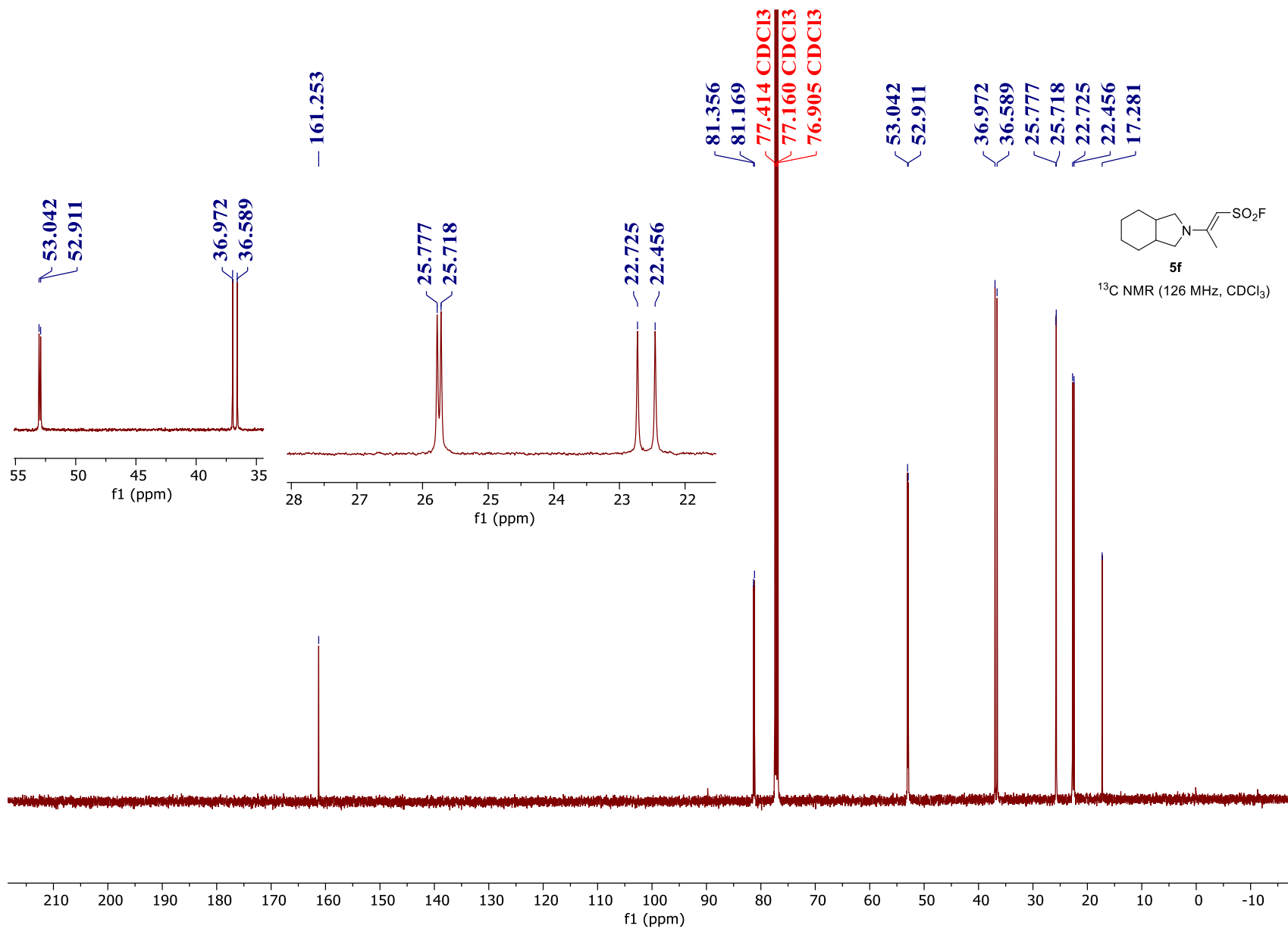


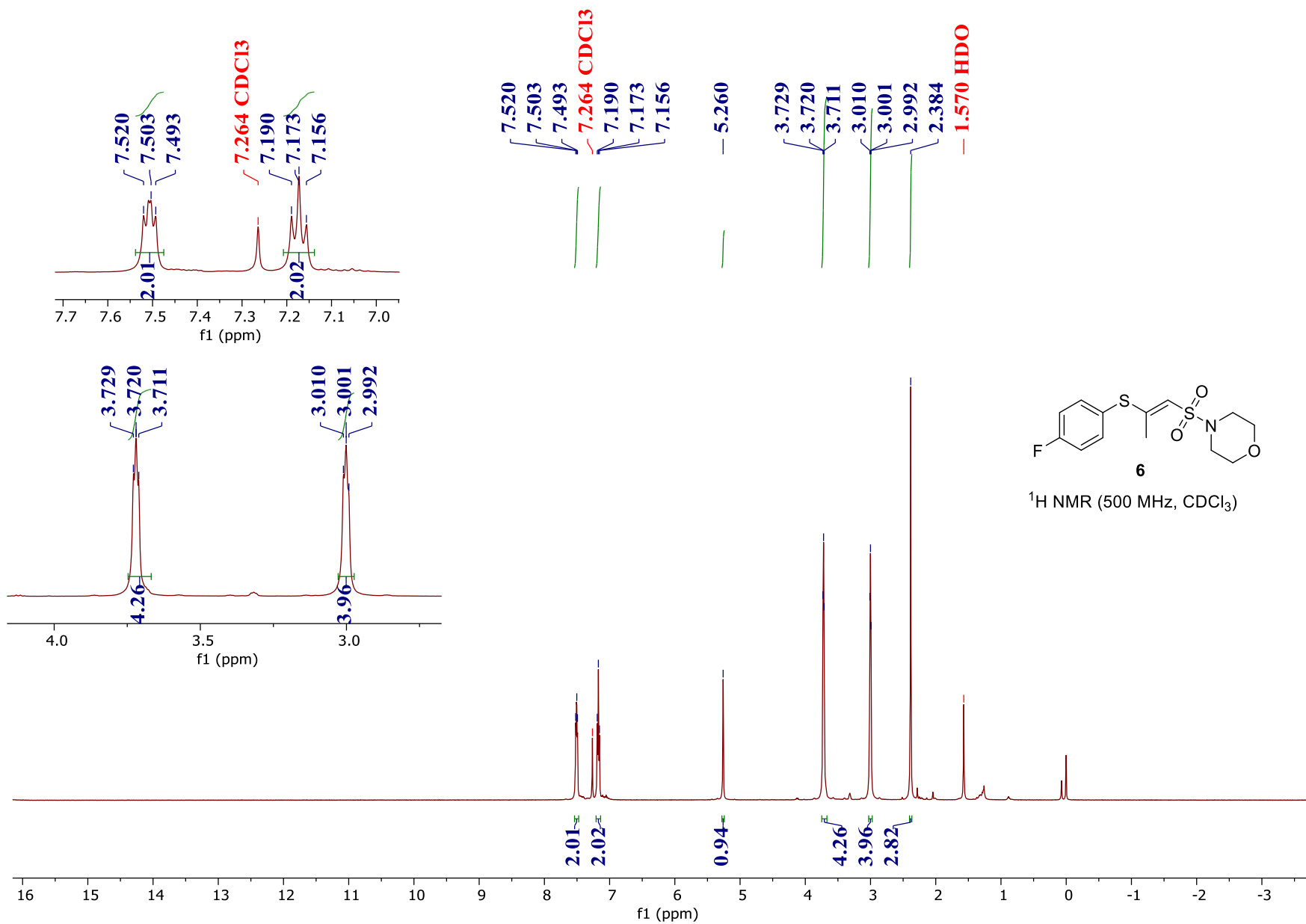
**5f**

<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)

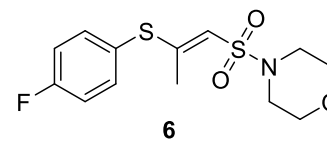




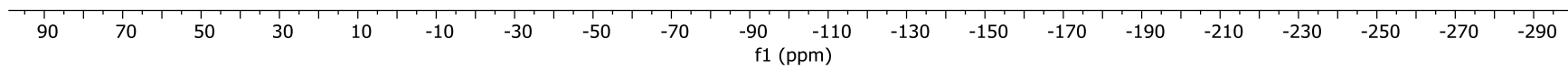


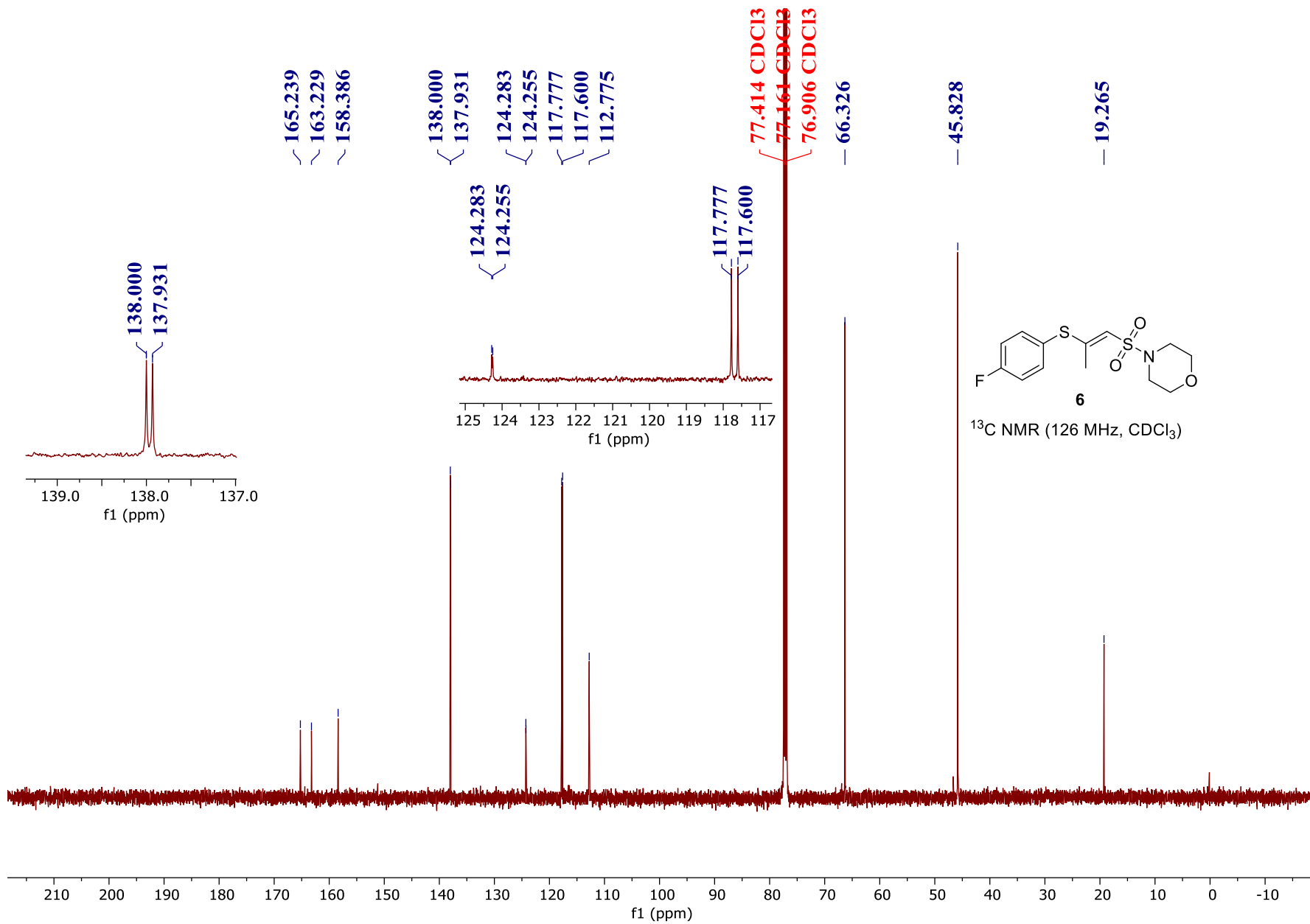


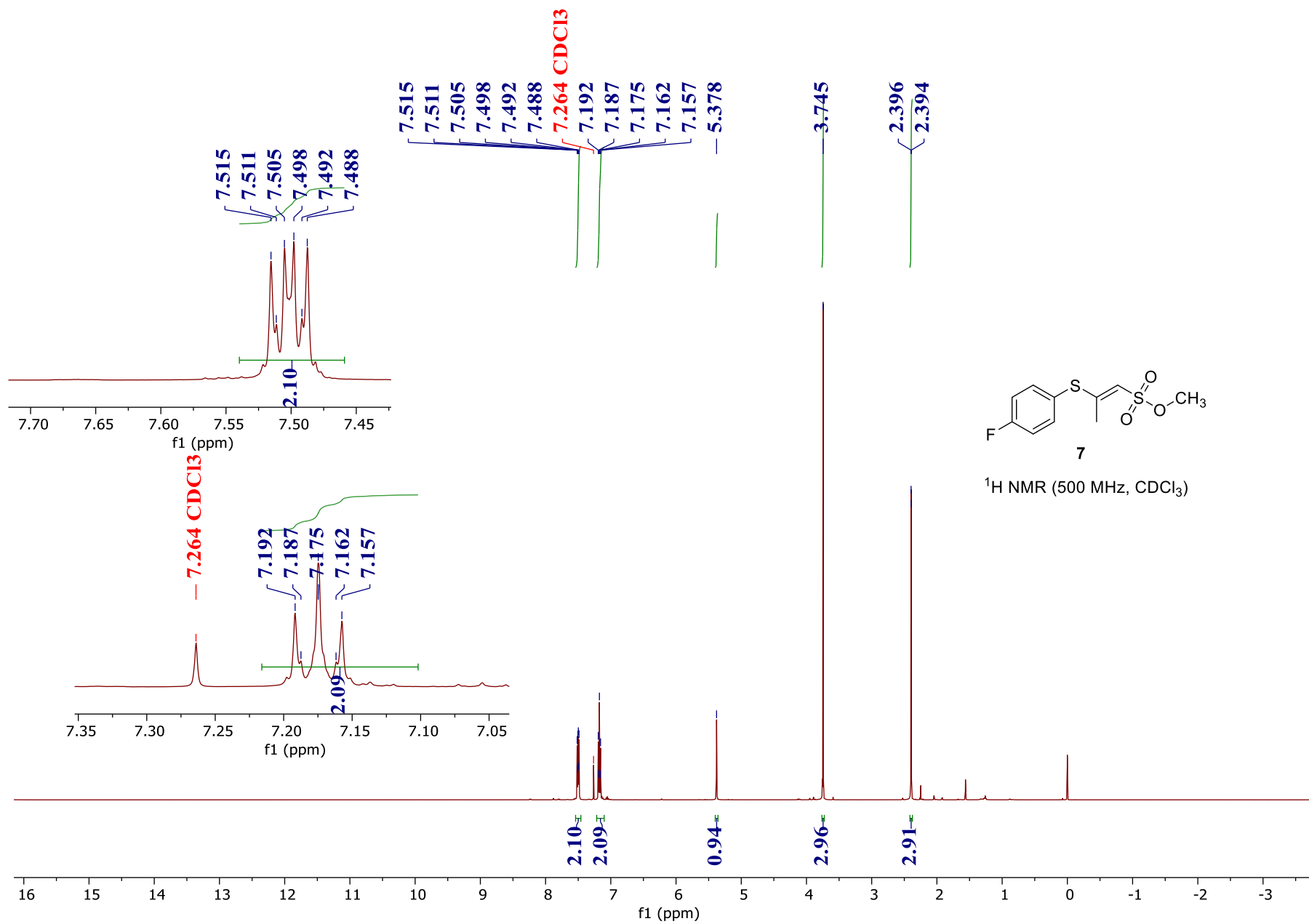
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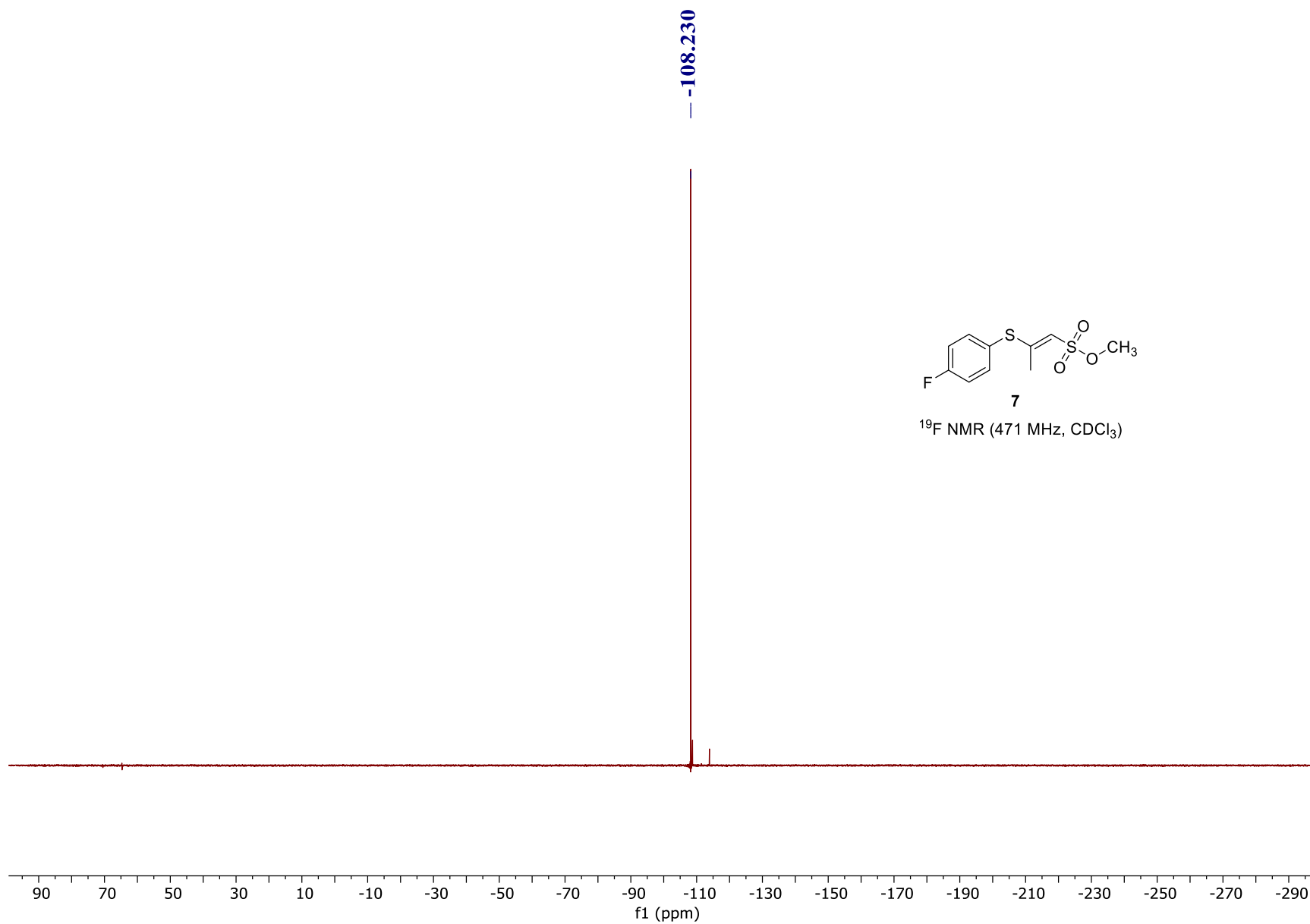


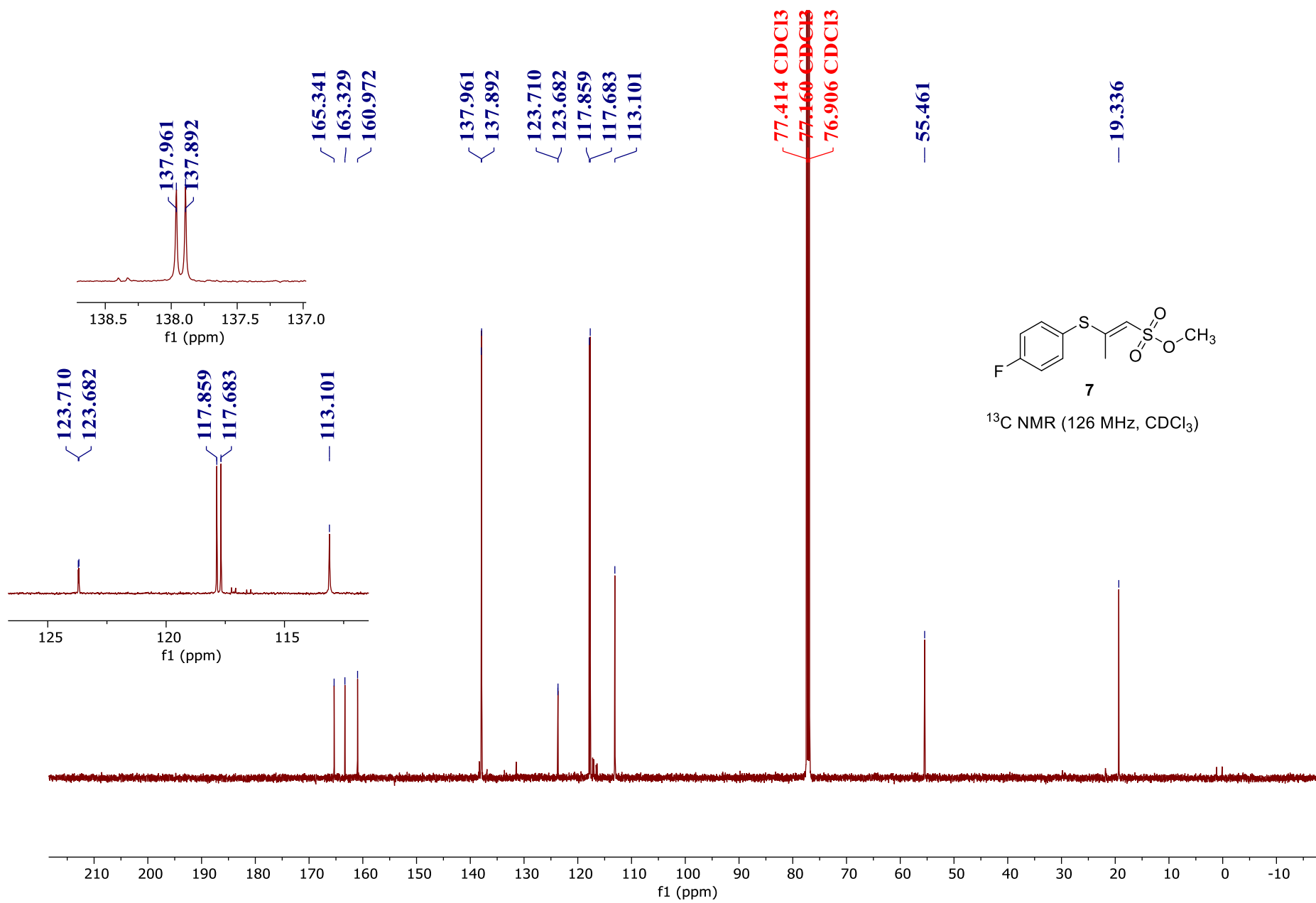
$^{19}\text{F}$  NMR (471 MHz,  $\text{CDCl}_3$ )

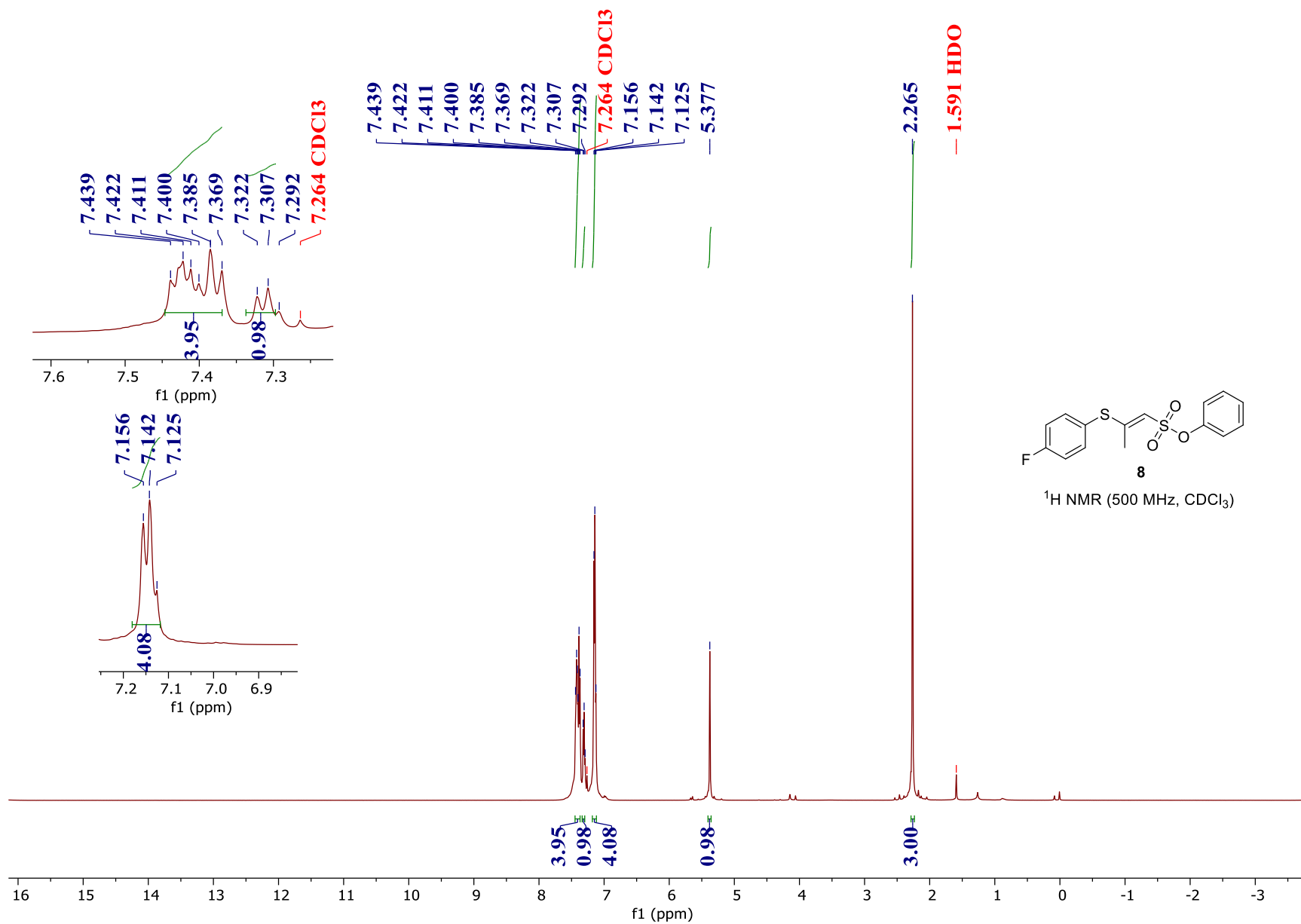




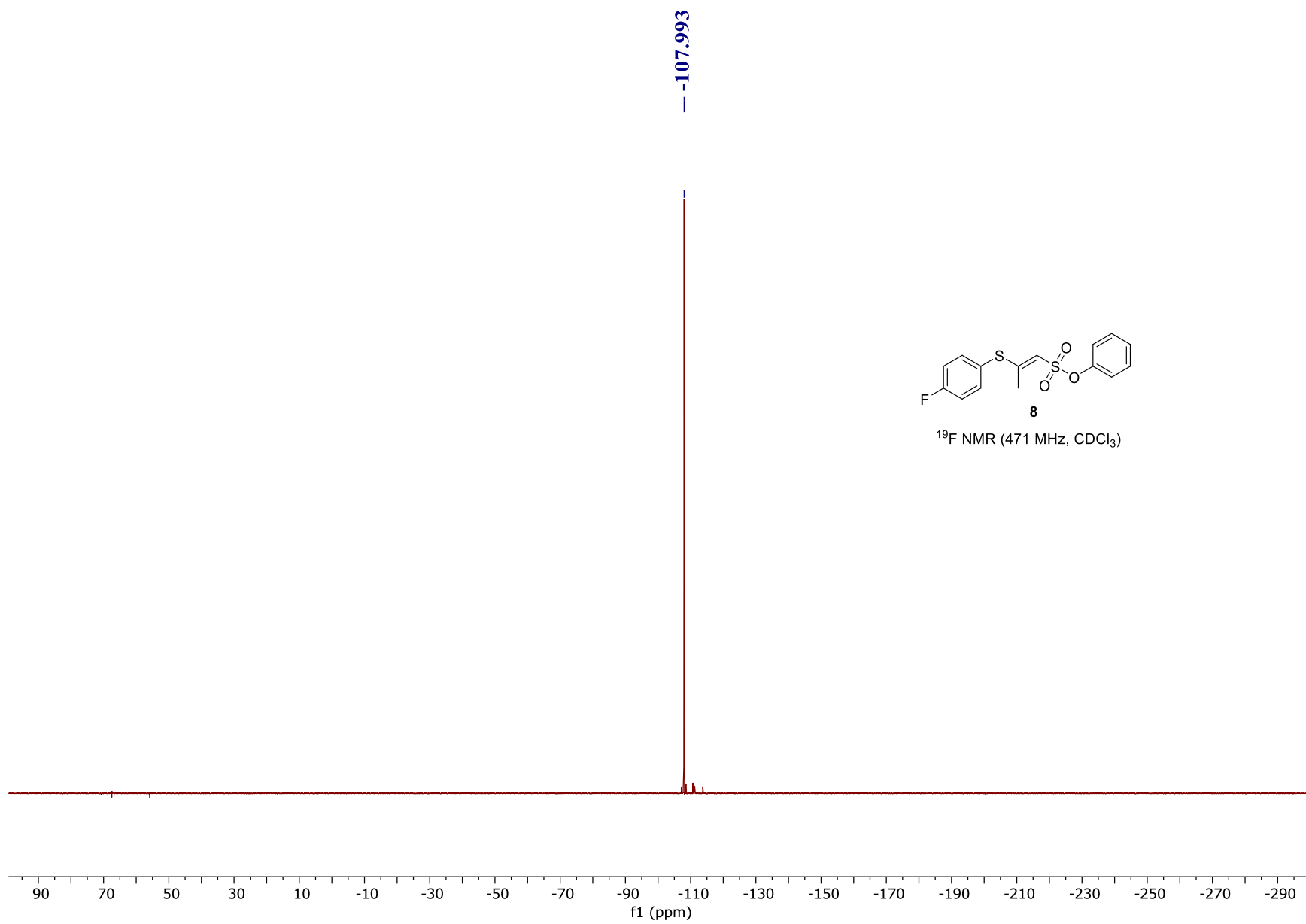


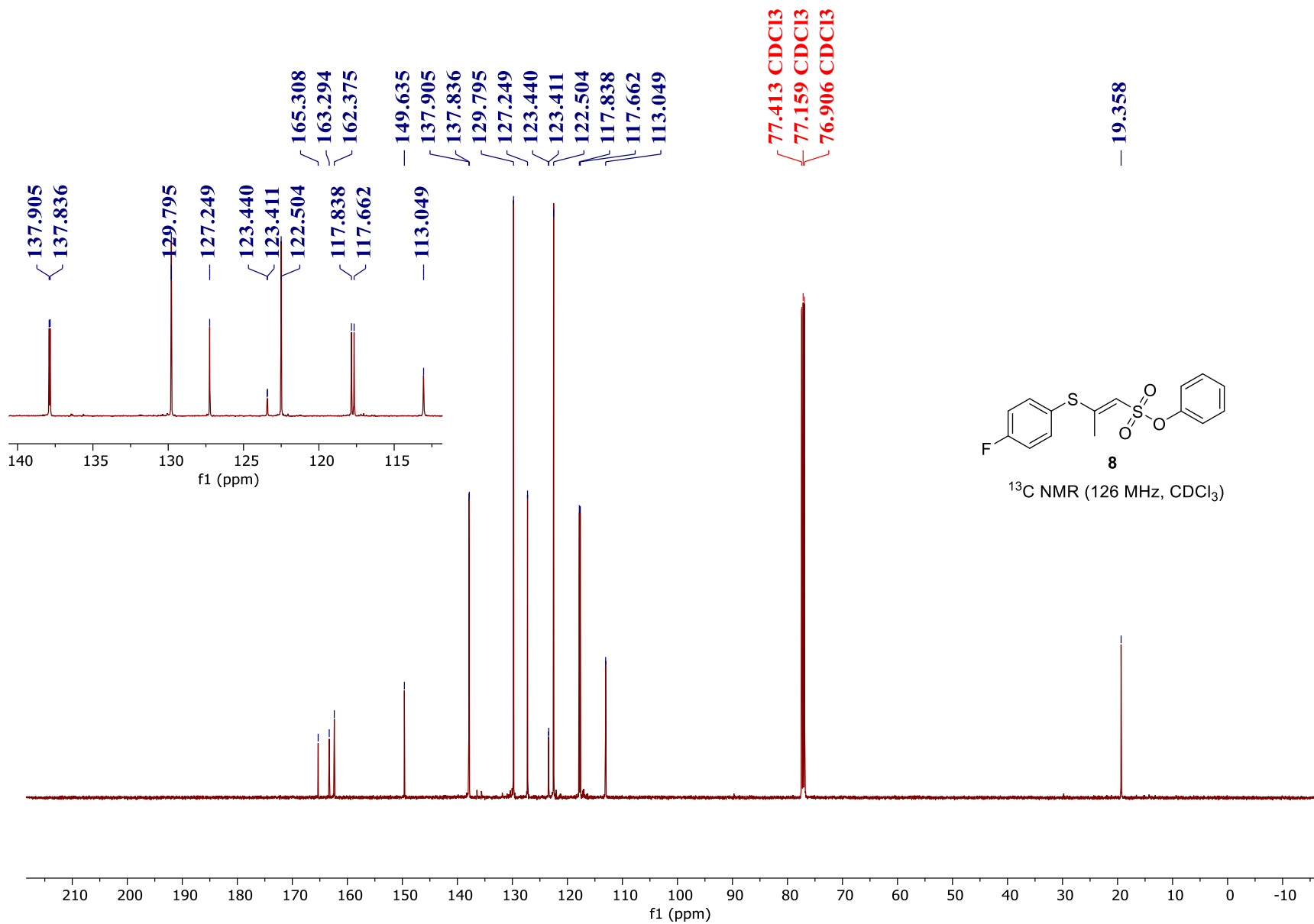


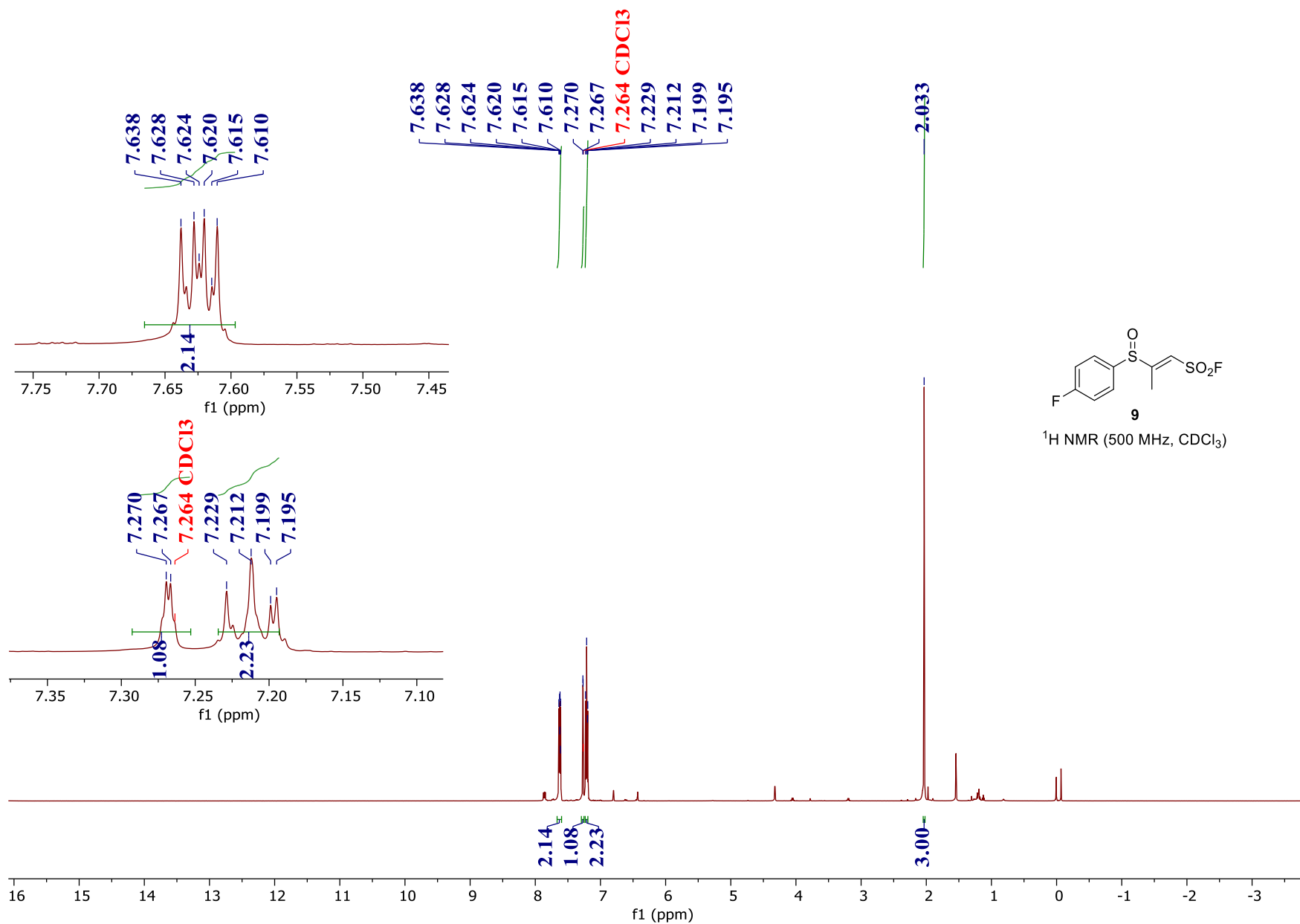


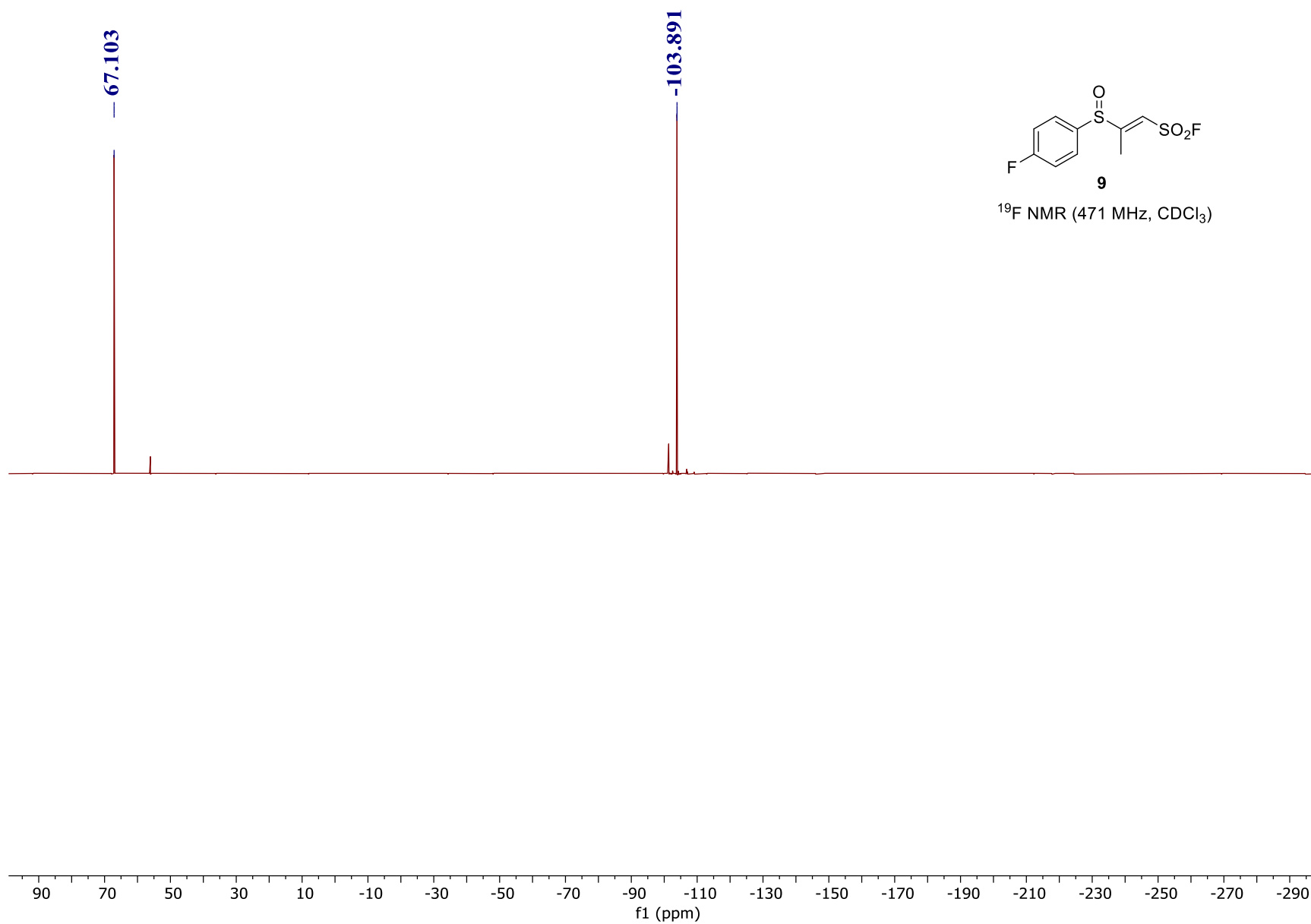


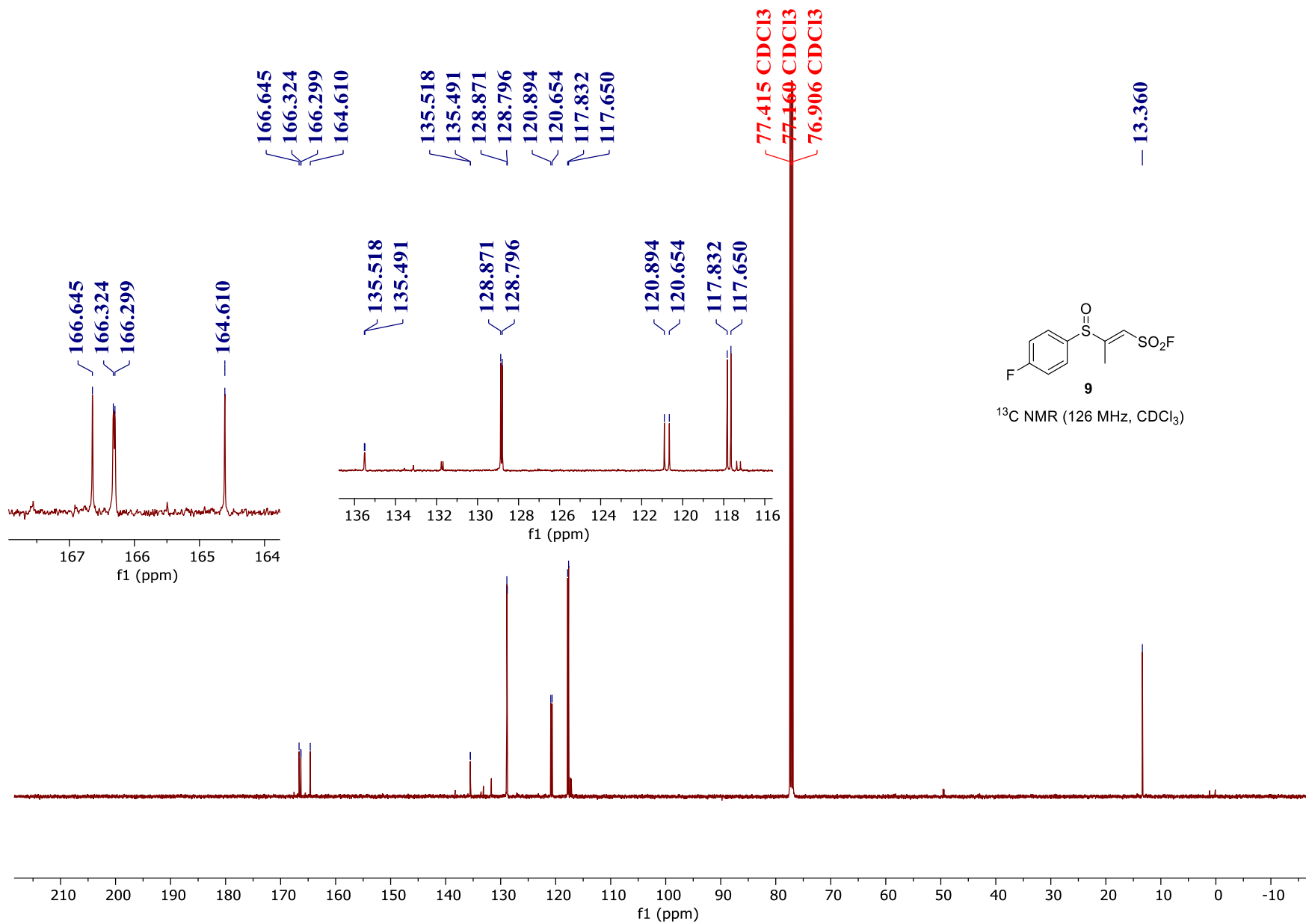


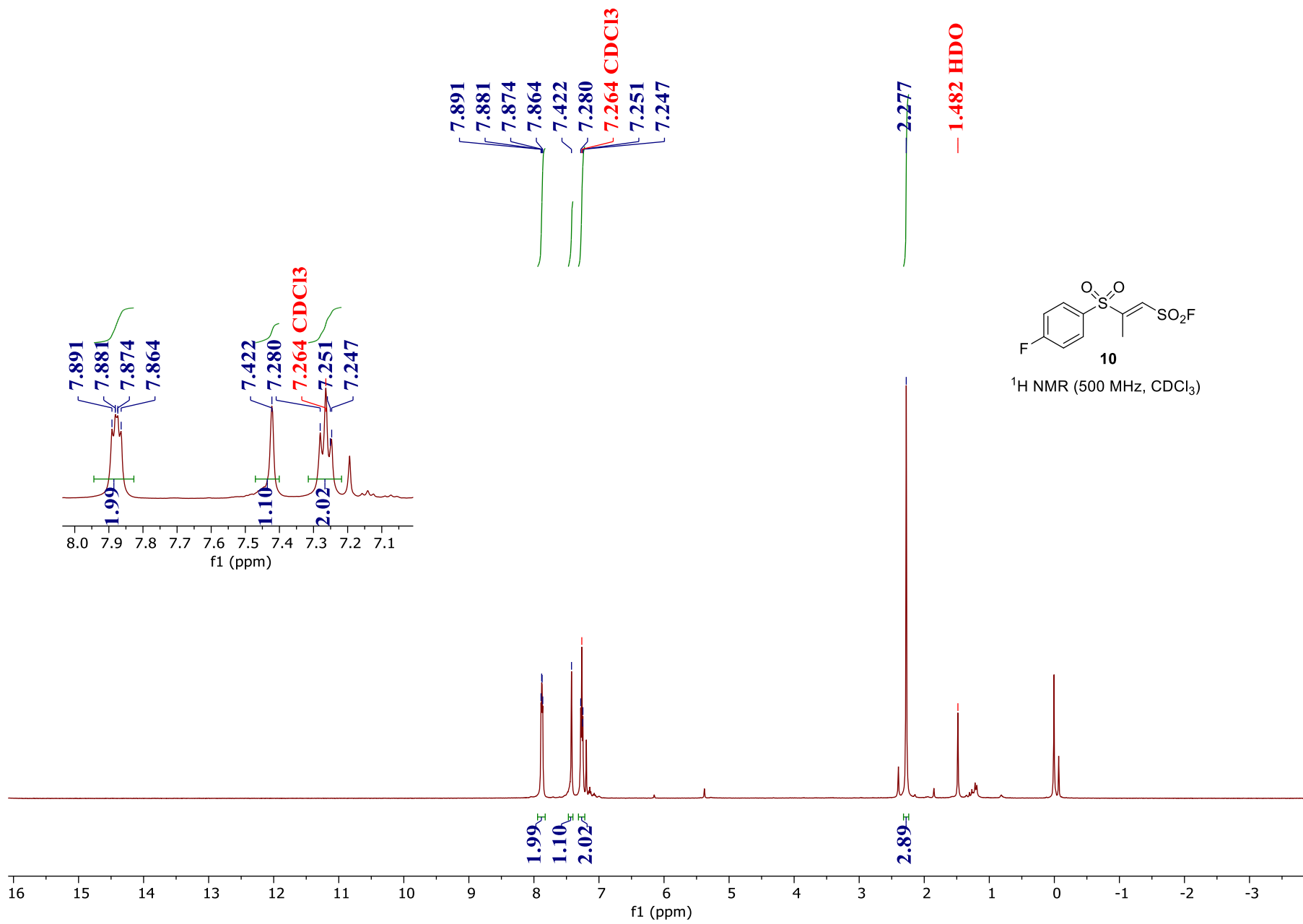


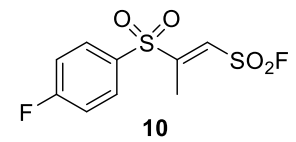












<sup>19</sup>F NMR (471 MHz, CDCl<sub>3</sub>)

