

## Supporting Information

# Diethylzinc-Promoted Carboxylation of Aryl/Alkenyl Boronic Acids with CO<sub>2</sub>

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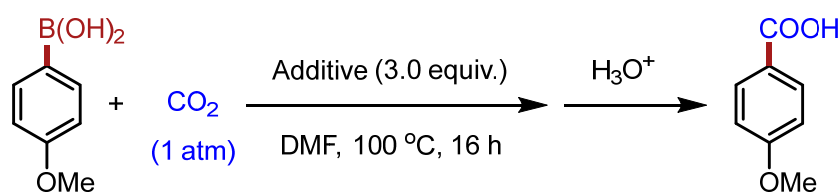
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## 1. Optimization Studies

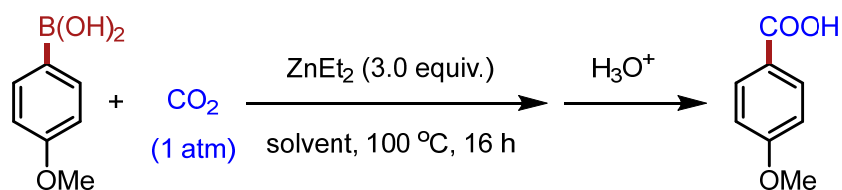
General procedure: An oven-dried 50 mL schlenk tube containing a stirring bar was charged with 4-methoxyphenylboronic acid (60.8 mg, 0.4 mmol, 1.0 equiv.). The schlenk tube was evacuated and back-filled under CO<sub>2</sub> flow for three times. Then, anhydrous solvent (2.0 mL) and ZnEt<sub>2</sub> solution (2 M in toluene) was added under CO<sub>2</sub> flow, and the resulting mixture was stirred at 100 °C for 16 h. The mixture was then allowed to cool to room temperature, carefully quenched with 1 M HCl and stirred for 5 minutes. The reaction mixture was extracted five times with EtOAc and the combined organic phases were washed with brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and filtered. The solvent was then removed under reduced pressure. The crude products were purified by flash chromatography (AcOH/EA/PE = 0/1/10 to 0.01/1/1).

**Table S1** Optimization of additive<sup>a</sup>



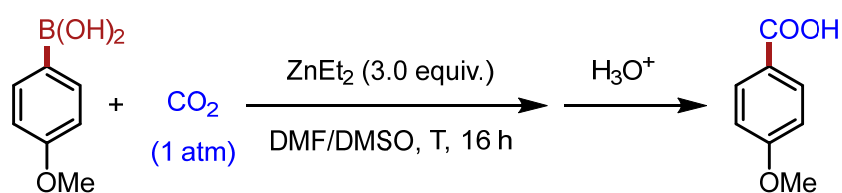
Entry	Additive	Solvent	T [°C]	Yield [%]
1	<b>ZnEt<sub>2</sub> (3.0 eq.)</b>	<b>DMF</b>	<b>100</b>	<b>94</b>
2	ZnEt <sub>2</sub> (0.0 eq.)	DMF	100	0
3	ZnMe <sub>2</sub> (3.0 eq.)	DMF	100	72
4	KHMDS	DMF	100	0
5	KO <sup>t</sup> Bu	DMF	100	0
6	Cs <sub>2</sub> CO <sub>3</sub>	DMF	100	0
7	CsF	DMF	100	0

<sup>a</sup> Reaction performed on 0.40 mmol scale. Isolated yields are given.

**Table S2** Optimization of solvent <sup>a</sup>

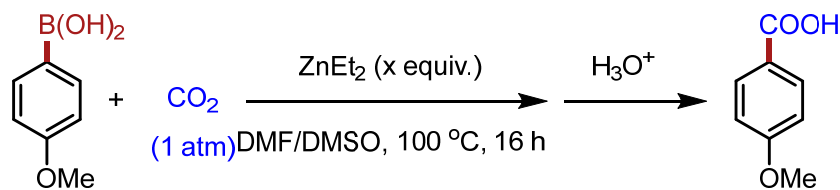
Entry	Additive	Solvent	T [°C]	Yield [%]
1	<b>ZnEt<sub>2</sub> (3.0 eq.)</b>	<b>DMF</b>	<b>100</b>	<b>94</b>
2	ZnEt <sub>2</sub> (3.0 eq.)	THF	100	46
3	ZnEt <sub>2</sub> (3.0 eq.)	1,4-dioxane	100	66
4	ZnEt <sub>2</sub> (3.0 eq.)	DMA	100	77
5	<b>ZnEt<sub>2</sub> (3.0 eq.)</b>	<b>DMSO</b>	<b>100</b>	<b>94</b>

<sup>a</sup> Reaction performed on 0.40 mmol scale. Isolated yields are given.

**Table S3** Optimization of temperature <sup>a</sup>

Entry	Additive	Solvent	T [°C]	Yield [%]
1	<b>ZnEt<sub>2</sub> (3.0 eq.)</b>	<b>DMF</b>	<b>100</b>	<b>94</b>
2	ZnEt <sub>2</sub> (3.0 eq.)	DMF	70	84
3	ZnEt <sub>2</sub> (3.0 eq.)	DMF	50	60
4	ZnEt <sub>2</sub> (3.0 eq.)	DMF	25	16
5	<b>ZnEt<sub>2</sub> (3.0 eq.)</b>	<b>DMSO</b>	<b>100</b>	<b>94</b>
6	ZnEt <sub>2</sub> (3.0 eq.)	DMSO	70	76

<sup>a</sup> Reaction performed on 0.40 mmol scale. Isolated yields are given.

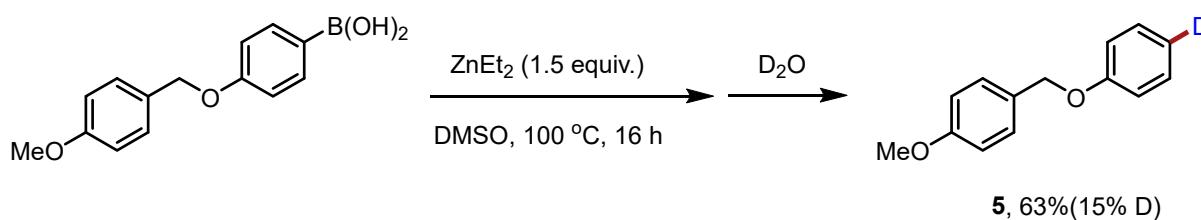
**Table S4** Optimization of the amount of diethylzinc <sup>a</sup>

Entry	Additive	Solvent	T [°C]	Yield [%]
1	ZnEt <sub>2</sub> (3.0 eq.)	DMF	100	94
2	ZnEt <sub>2</sub> (2.0 eq.)	DMF	100	84
3	ZnEt <sub>2</sub> (1.5 eq.)	DMF	100	80
4	ZnEt <sub>2</sub> (1.0 eq.)	DMF	100	73
5	ZnEt <sub>2</sub> (3.0 eq.)	DMSO	100	94
6	ZnEt <sub>2</sub> (2.0 eq.)	DMSO	100	94
7	<b>ZnEt<sub>2</sub> (1.5 eq.)</b>	<b>DMSO</b>	<b>100</b>	<b>94</b>
8	ZnEt <sub>2</sub> (1.2 eq.)	DMSO	100	74
9	ZnEt <sub>2</sub> (1.0 eq.)	DMSO	100	62

<sup>a</sup> Reaction performed on 0.40 mmol scale. Isolated yields are given.

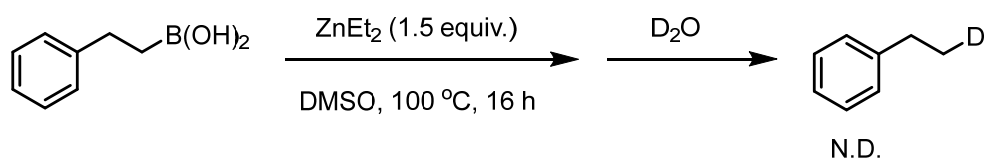
## 2. Mechanism Research

### Quenching experiment in the absence of CO<sub>2</sub>

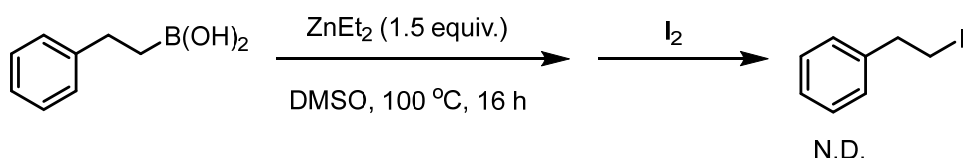


A 50 mL Schlenk tube was charged with (4-((4-methoxybenzyl)oxy)phenyl)boronic acid (**1e**, 103.2 mg, 0.4 mmol), and the tube was evacuated and refilled with Ar (3 times). DMSO (2.0 mL) and ZnEt<sub>2</sub> (2 M in toluene, 0.6 mmol, 1.5 equiv.) were then added, and the resulting mixture was stirred vigorously for 16 h at 100 °C. The reaction was quenched with D<sub>2</sub>O (4.0 mL), and the resulting mixture was stirred for 1 h. The organic layer was extracted with EtOAc (3 × 10 mL). The combined organic layer was passed through a pad of Na<sub>2</sub>SO<sub>4</sub>, then all volatiles

were removed under reduced pressure. The residue was purified by column chromatography to afford 1-((4-methoxybenzyl)oxy)benzene-4-*d* (**5**) as a white solid (54.2 mg, 63%).



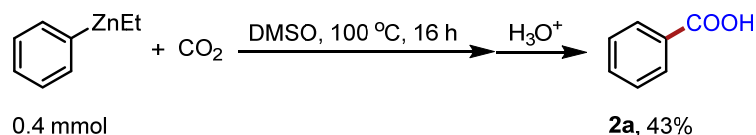
A 50 mL Schlenk tube was charged with phenethylboronic acid (60.0 mg, 0.4 mmol), and evacuated and refilled with Ar (3 times). DMSO (2.0 mL) and  $\text{ZnEt}_2$  (2 M in toluene, 0.6 mmol, 1.5 equiv.) were then added, and the resulting mixture was stirred vigorously for 16 h at  $100\text{ }^\circ\text{C}$ . The reaction was quenched with  $\text{D}_2\text{O}$  (4.0 mL), and the resulting mixture was stirred for 1 h. The organic layer was extracted with EtOAc ( $3 \times 10\text{ mL}$ ) and the combined organic layer was passed through a pad of  $\text{Na}_2\text{SO}_4$ . Then, organic layer was detected by GC-MS.



A 50 mL Schlenk tube was charged with phenethylboronic acid (60.0 mg, 0.4 mmol), and evacuated and refilled with Ar (3 times). DMSO (2.0 mL) and  $\text{ZnEt}_2$  (2 M in toluene, 0.6 mmol, 1.5 equiv.) were then added, and the resulting mixture was stirred vigorously for 16 h at  $100\text{ }^\circ\text{C}$ . The reaction was quenched with  $\text{I}_2$  (1.2 mmol, 3.0 equiv. in DMSO), and the resulting mixture was stirred for 1 h. Then, 30 mL saturated  $\text{NaS}_2\text{O}_3$  aqueous solution was added and stirred for 30 min. The organic layer was extracted with EtOAc ( $3 \times 10\text{ mL}$ ) and the combined organic layer was passed through a pad of  $\text{Na}_2\text{SO}_4$ . Then, organic layer was detected by GC-MS.

### Preparation of $\text{PhZnEt}$ solution<sup>1</sup>

A dry and nitrogen flushed 100 mL three-necked flask, equipped with a magnetic stirrer, a thermometer, a nitrogen inlet, and a septum, was charged with zinc chloride (817.8 mg, 6 mmol, 1 equiv.) and THF (6 mL). After exothermic dissolution of zinc chloride in THF, phenylmagnesium bromide (6.3 mL, 1 M, 6.3 mmol, 1.05 equiv.) was added dropwise at room temperature. Stirring was continued for 15 min, then, ethylmagnesium chloride (3.1 mL, 2 M, 6.3 mmol, 1.05 equiv.) was added dropwise at room temperature. After completion of the addition, the reaction mixture was stirred for 15 min.



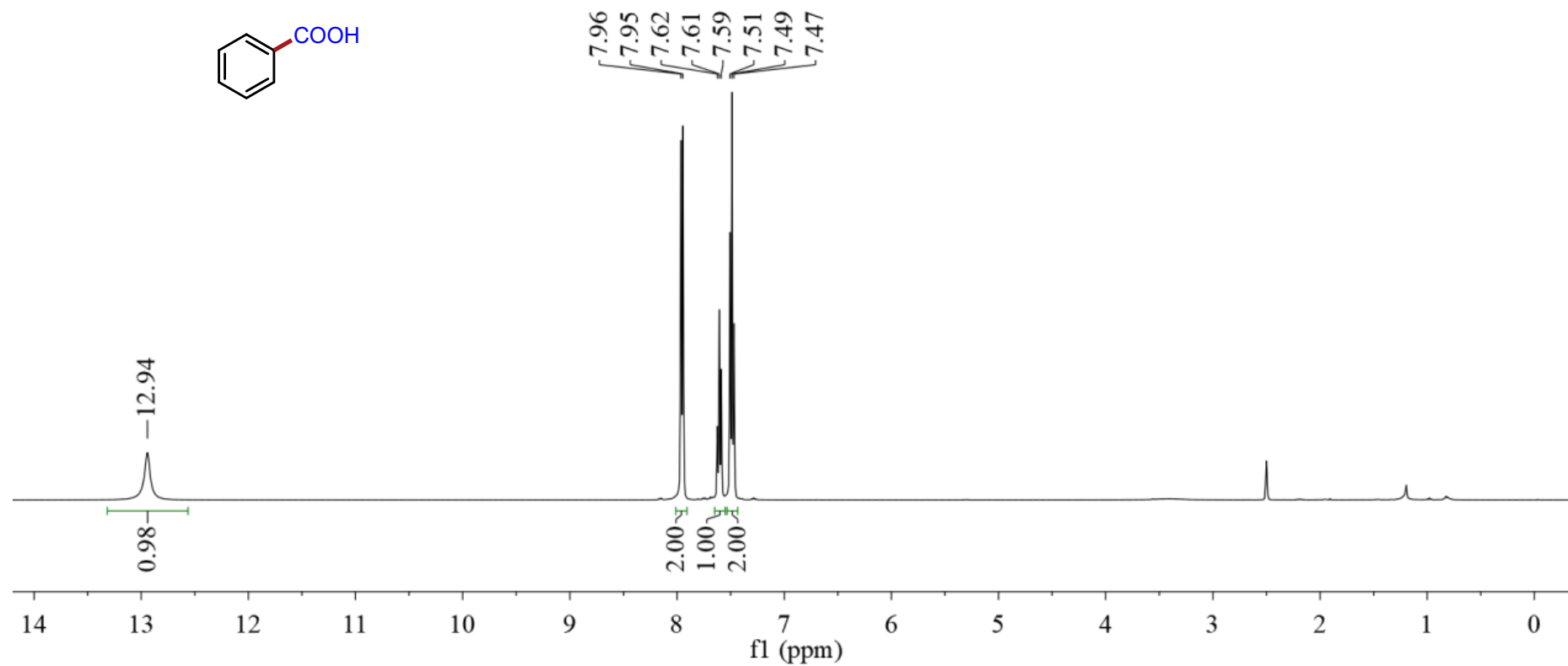
An oven-dried 50 mL Schlenk tube containing a stirring bar was evacuated and back-filled under CO<sub>2</sub> flow (this procedure was repeated three times). Then, anhydrous DMSO (1.0 mL) and PhZnEt (0.4 mmol, 1.0 equiv, 0.4 M in THF) was added under CO<sub>2</sub> flow, and the resulting mixture was stirred at 100 °C for 16 h. Then, the mixture was carefully quenched with 1 M HCl and stirred for 5 minutes. The reaction mixture was extracted five times with EtOAc and the combined organic phases were washed with brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and filtered. The solvent was then removed under reduced pressure. The crude products were purified by flash chromatography affording benzoic acid (**2a**) in 43% yield.

## References

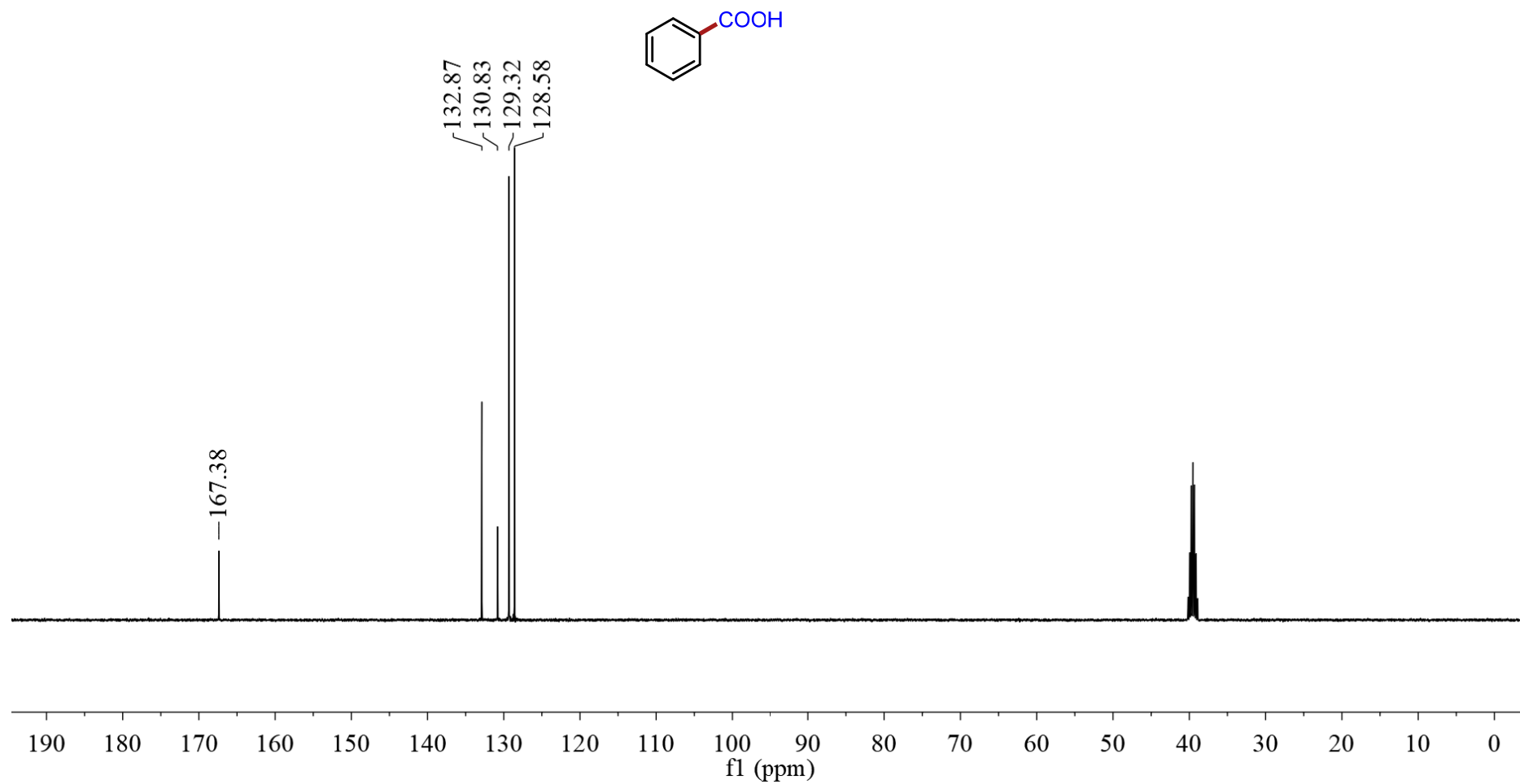
- (1) Cahiez, G.; Foulgoc, L.; Moyeux, A. Iron-Catalyzed Oxidative Heterocoupling between Aliphatic and Aromatic Organozinc Reagents: A Novel Pathway for Functionalized Aryl-Alkyl Cross-Coupling Reactions. *Angew. Chem. Int. Ed.* **2009**, *48*, 2969-2972.

# Copies of $^1\text{H}$ , $^{13}\text{C}$ and $^{19}\text{F}$ NMR Spectra for Compounds

$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2a**

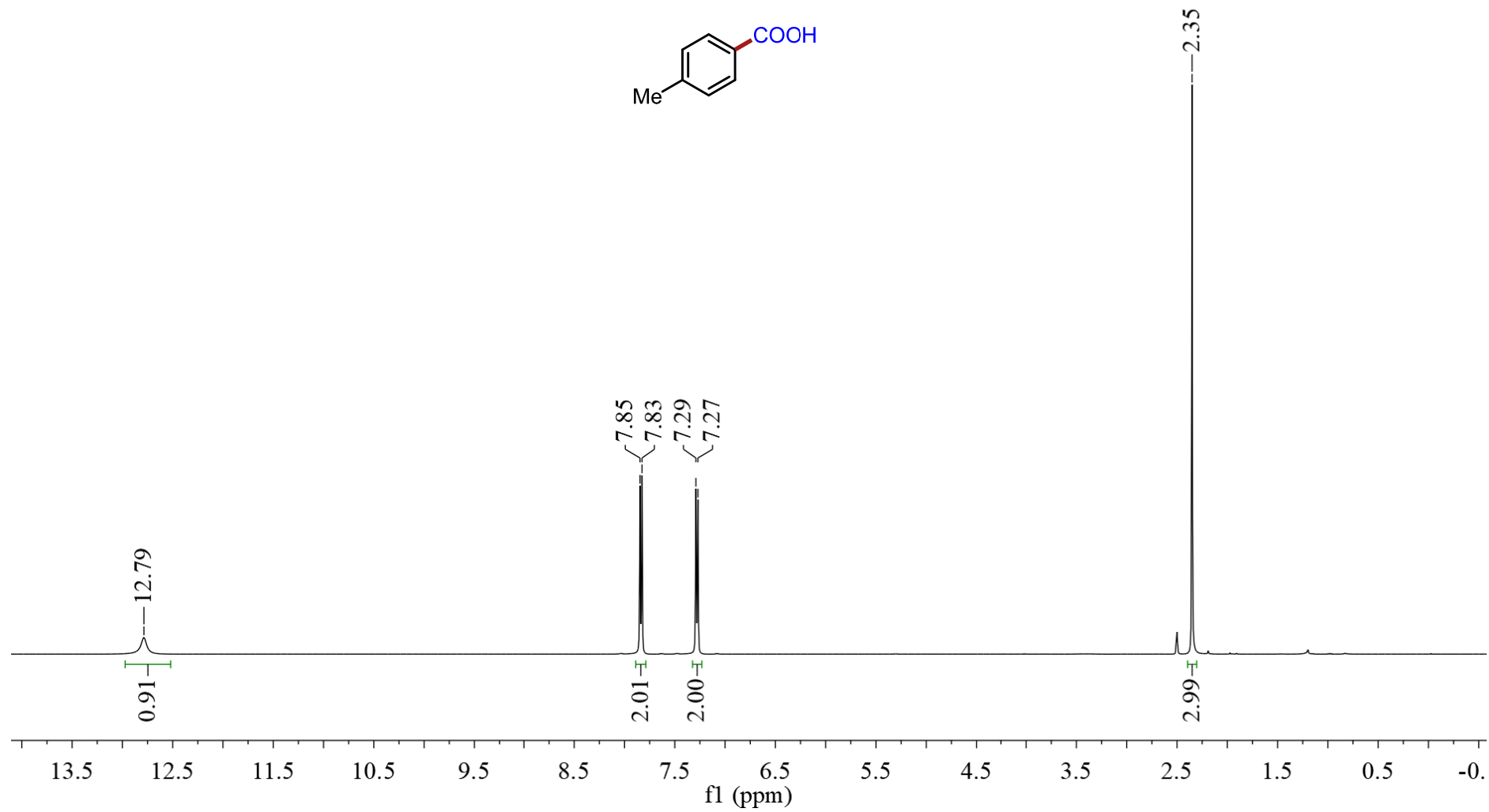
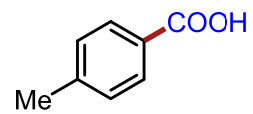


$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2a**

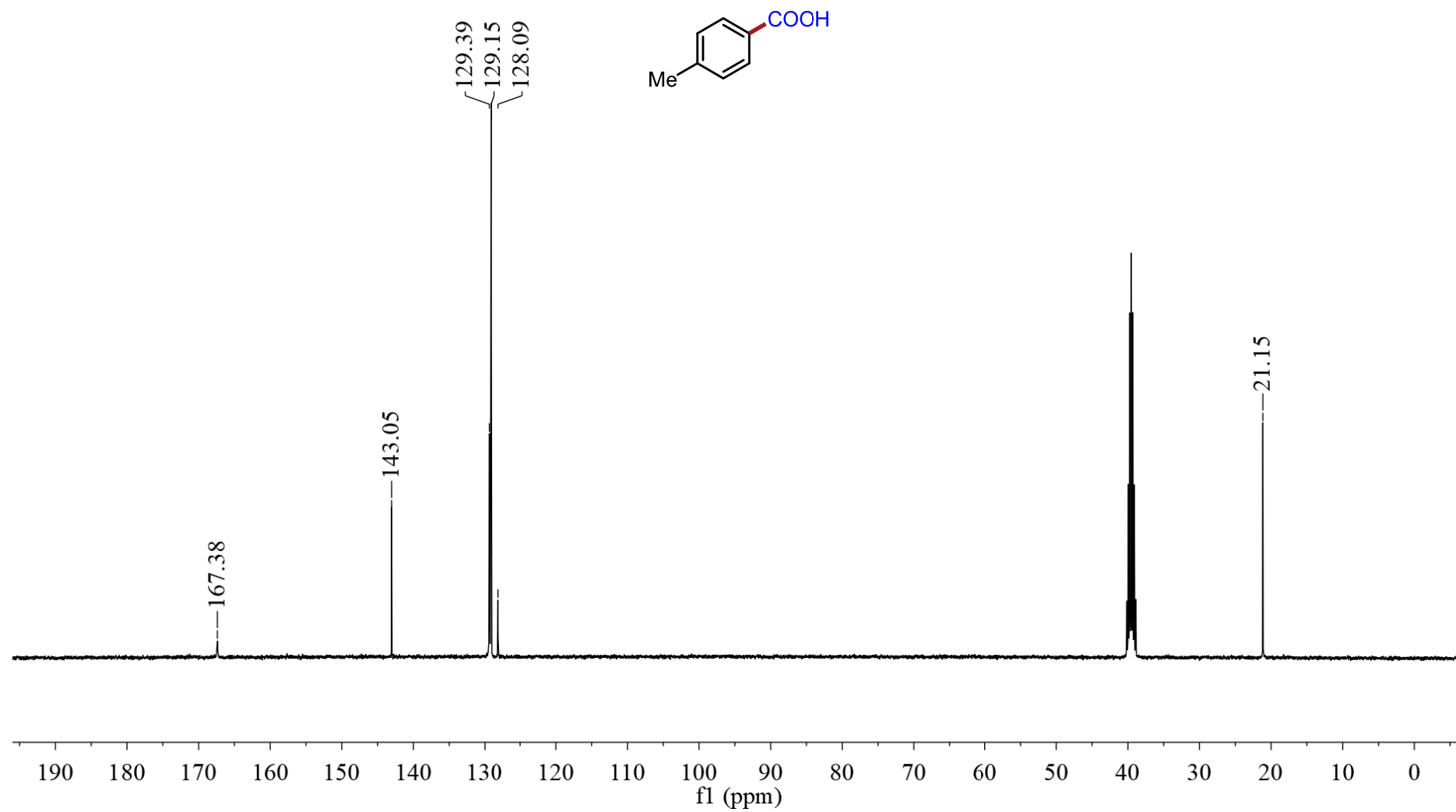




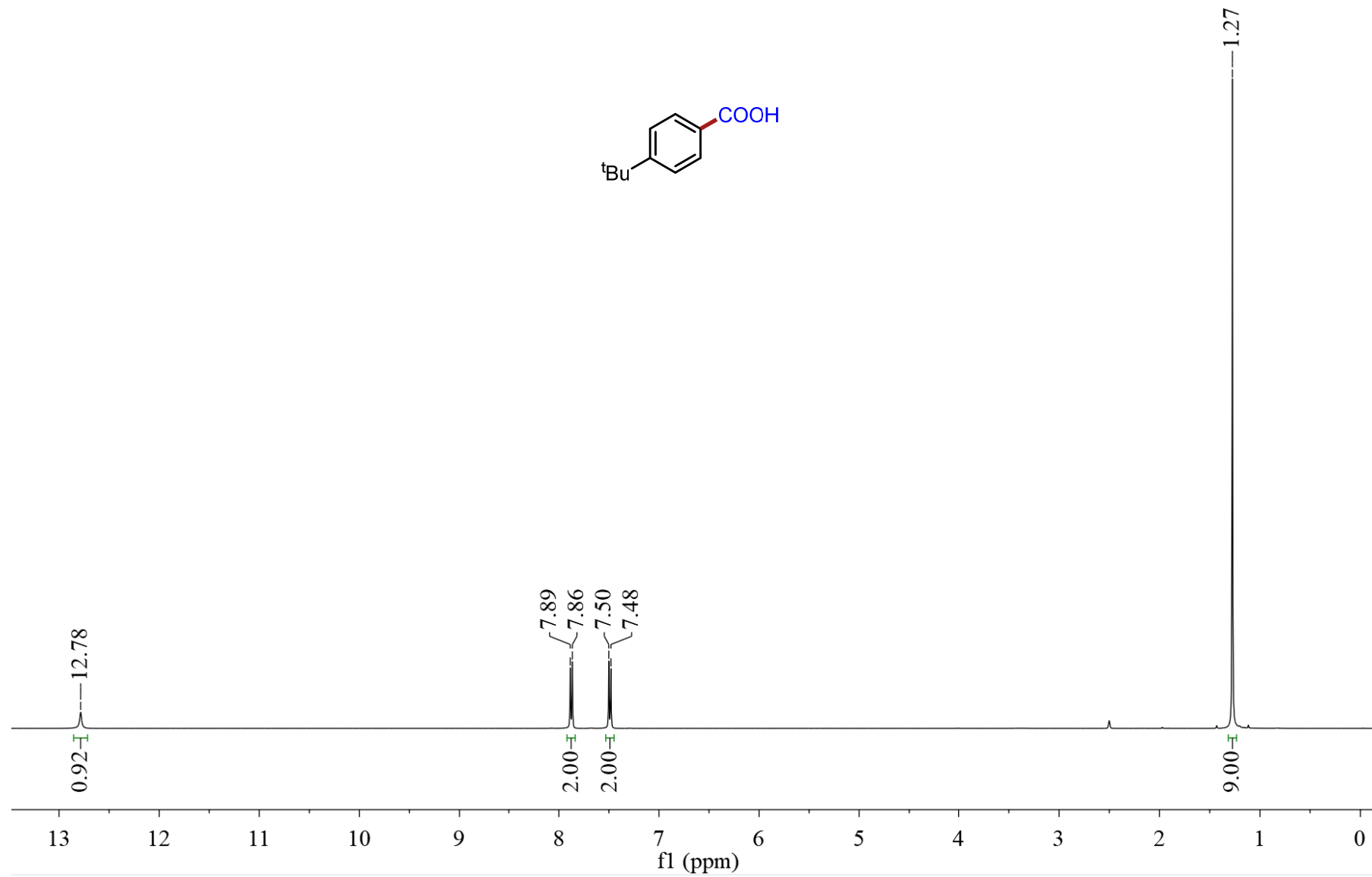
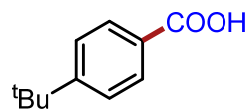
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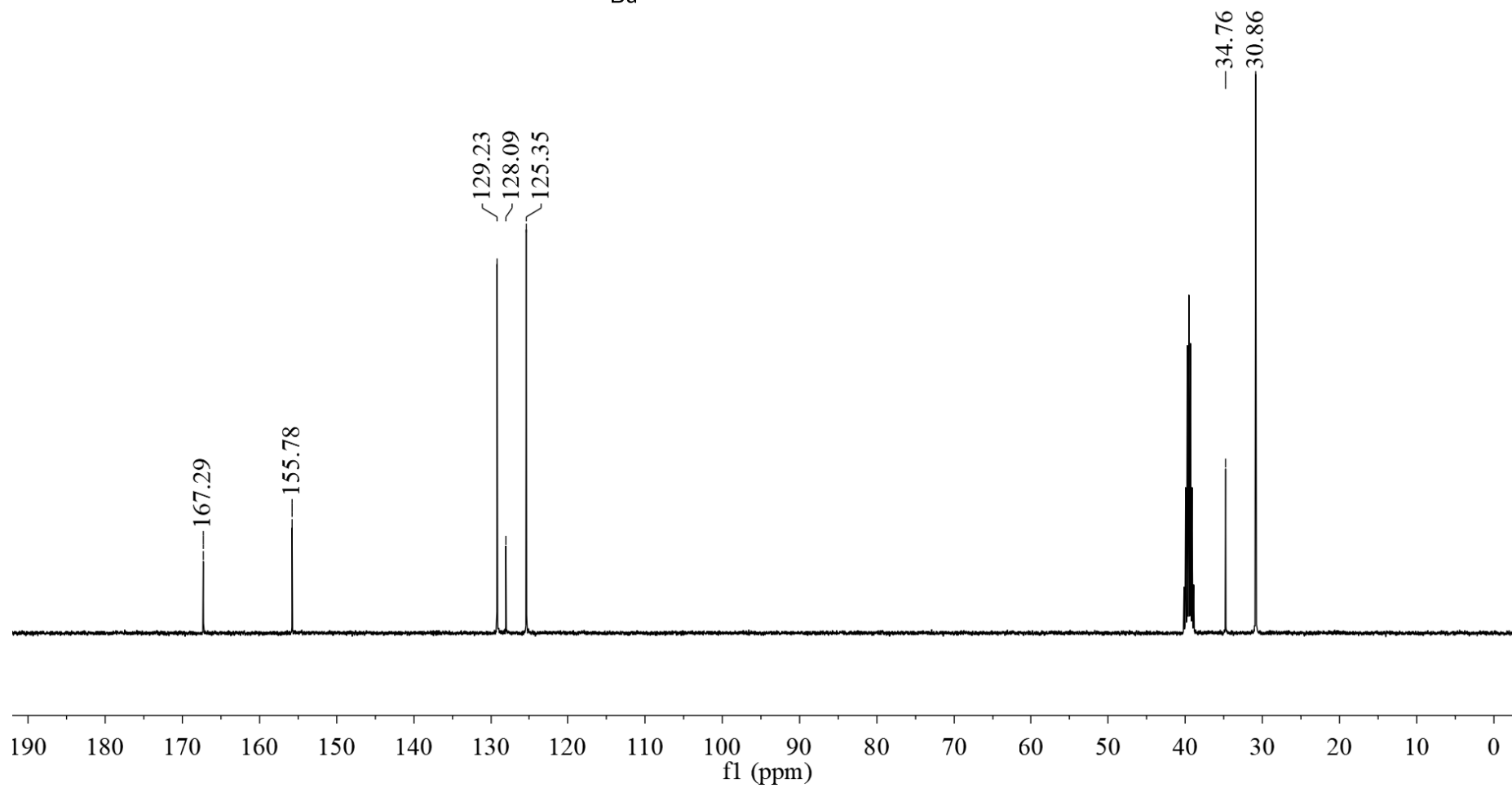
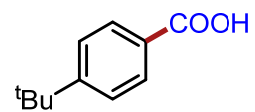
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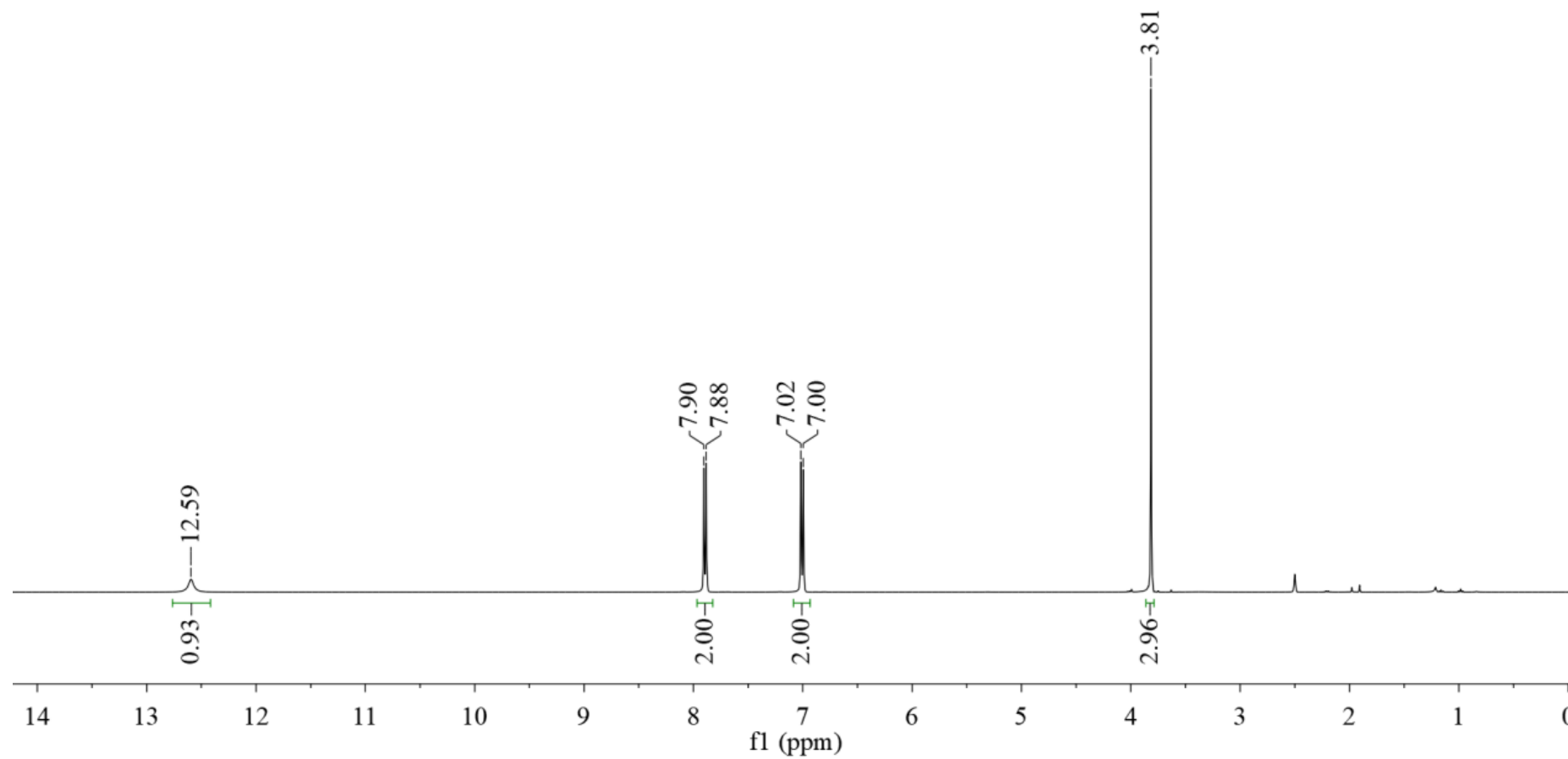
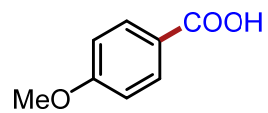
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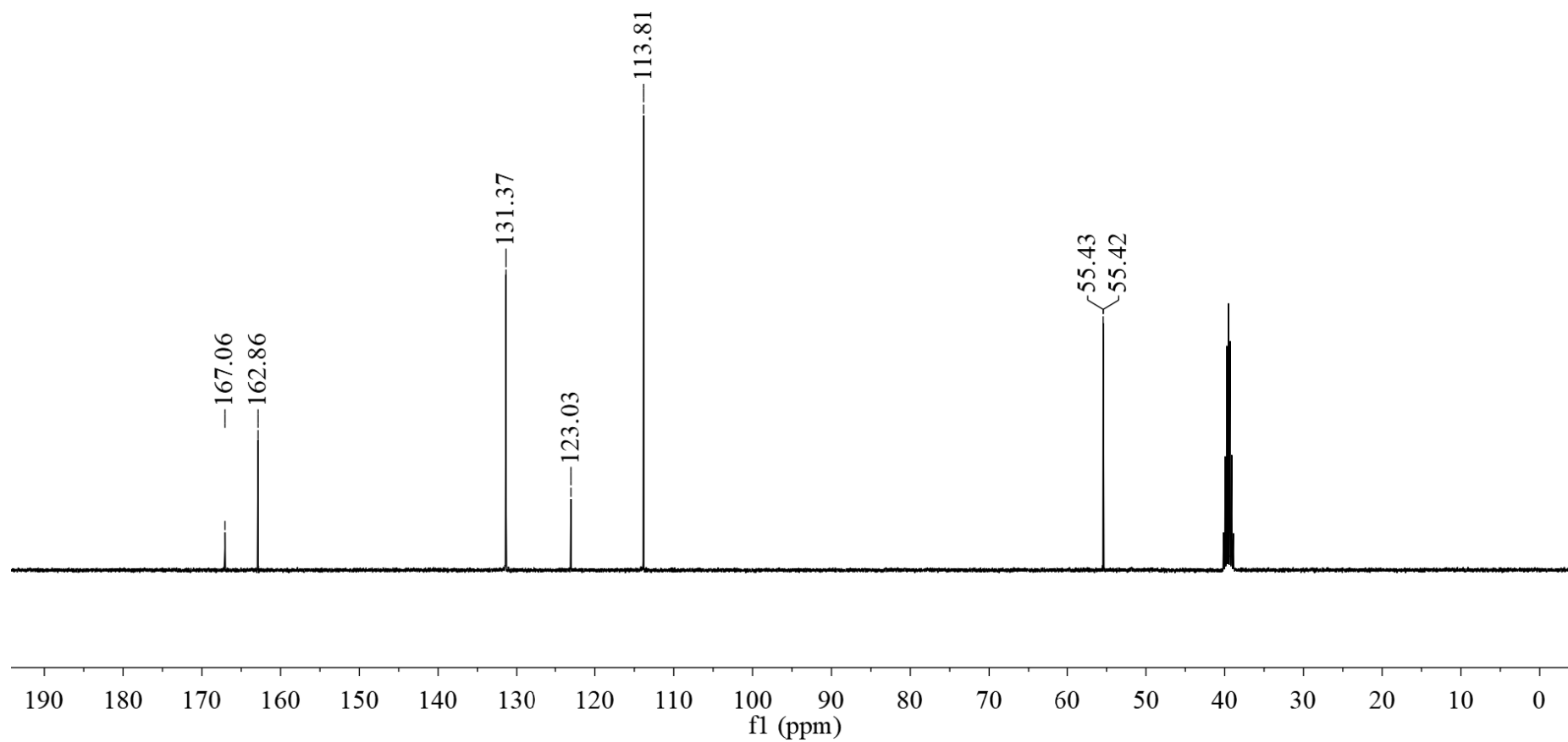
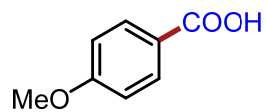
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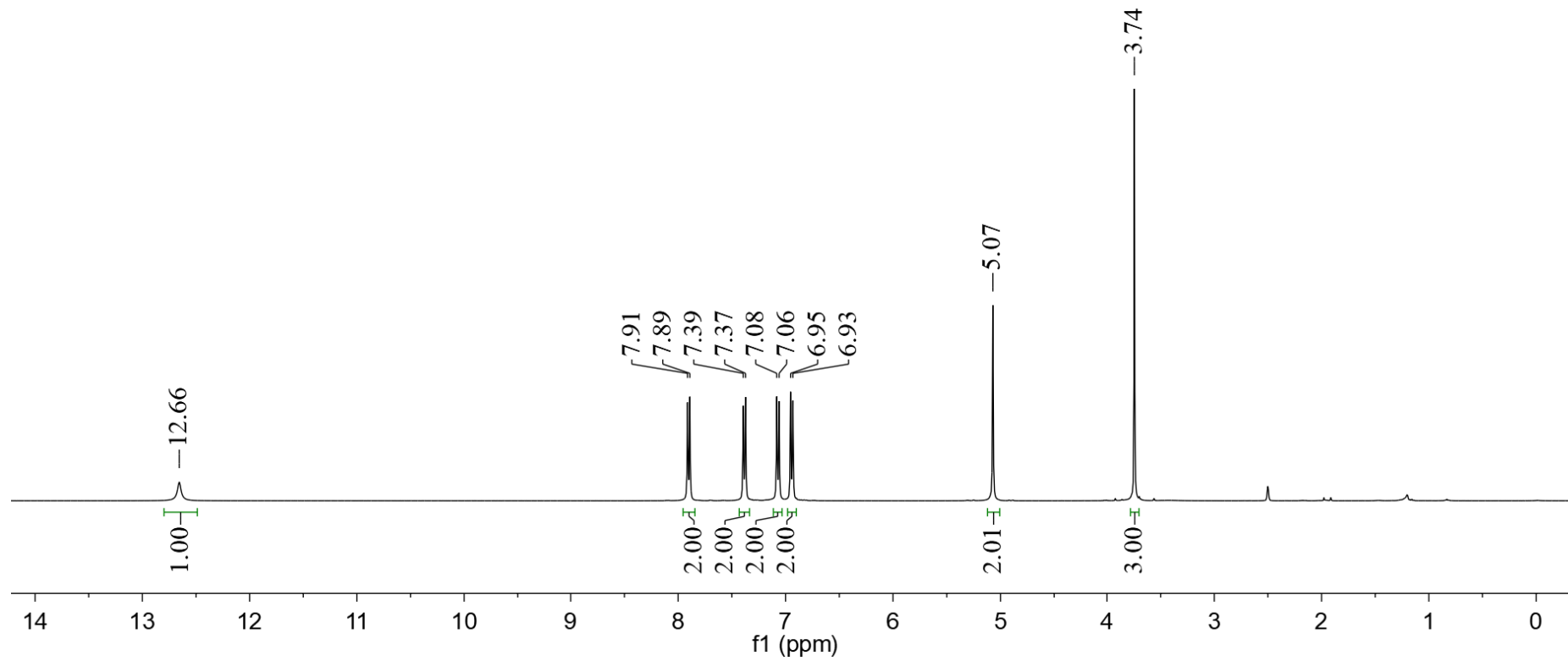
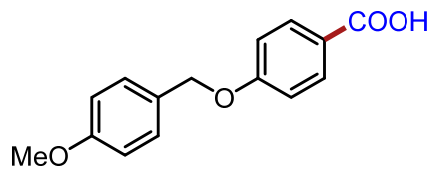
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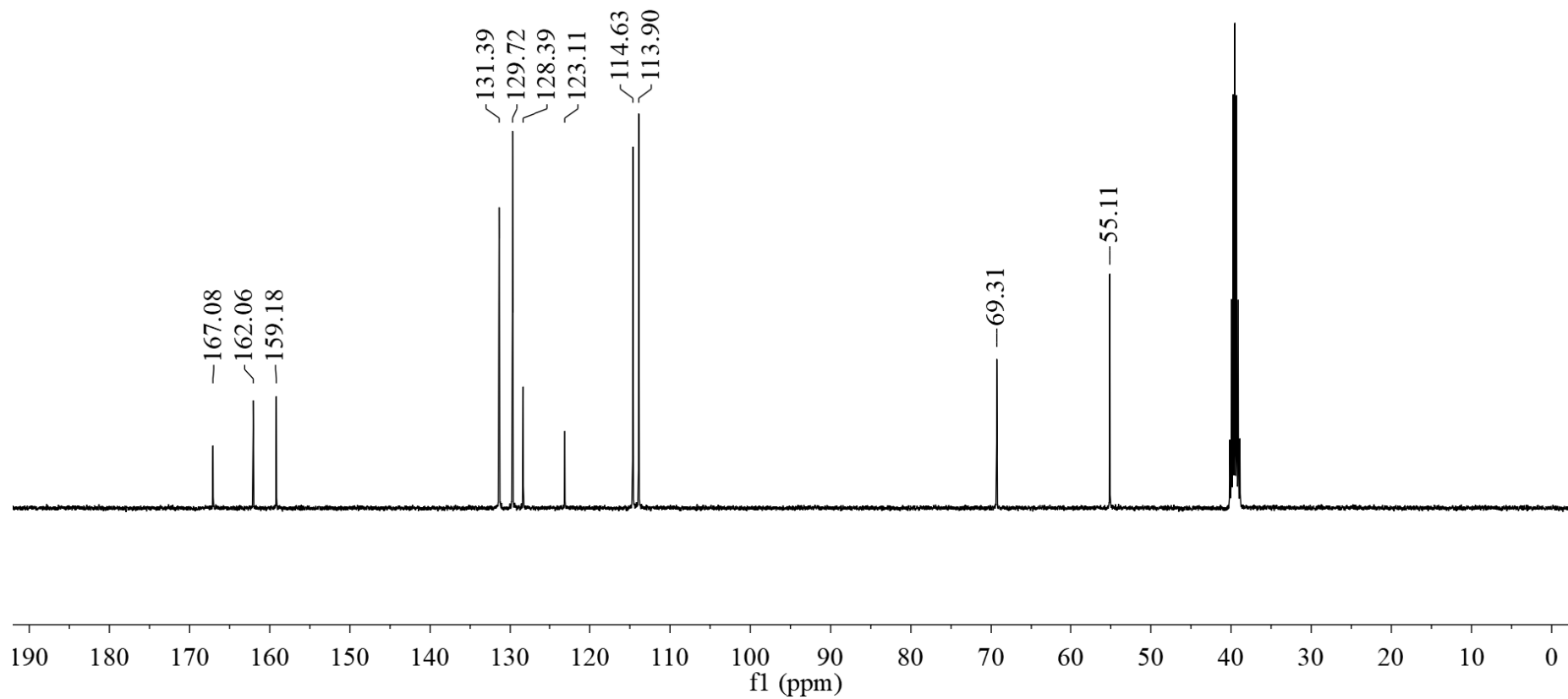
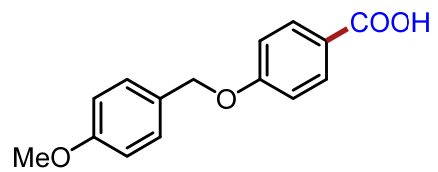
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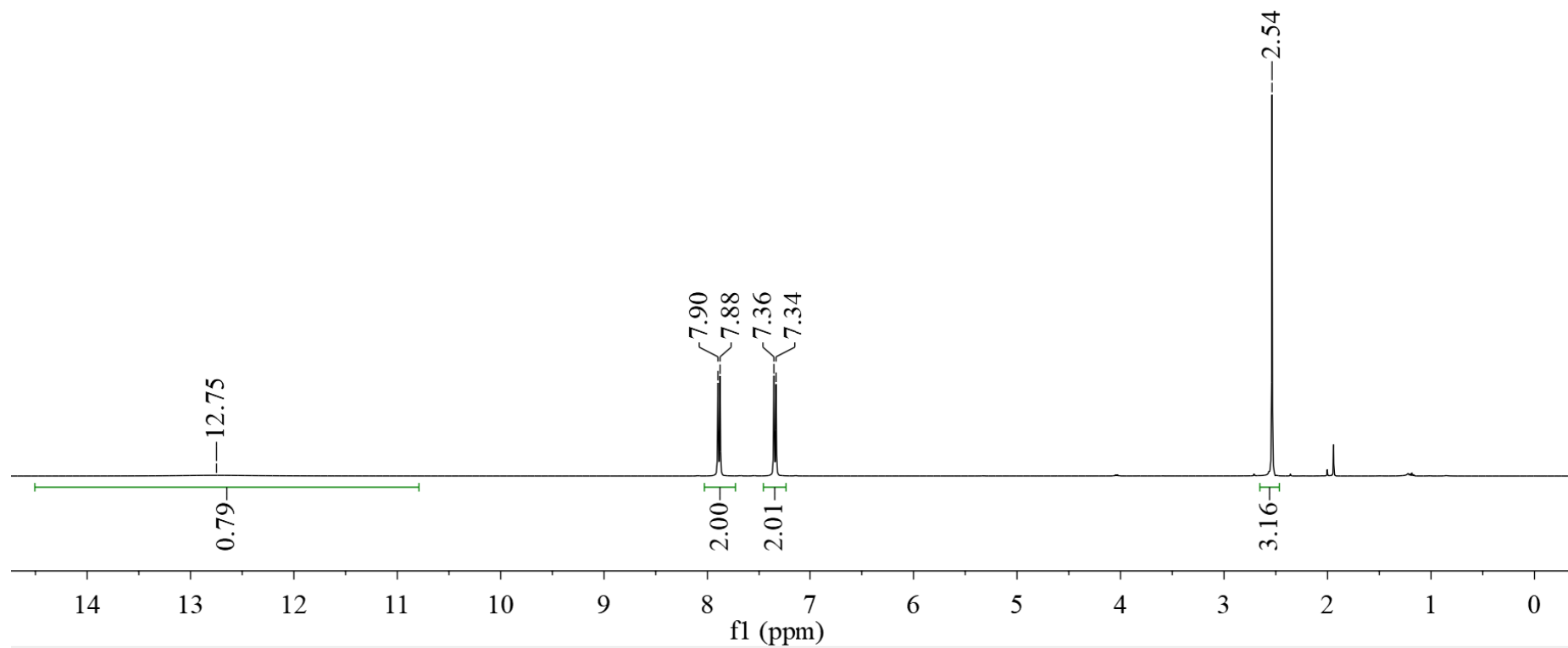
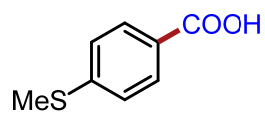


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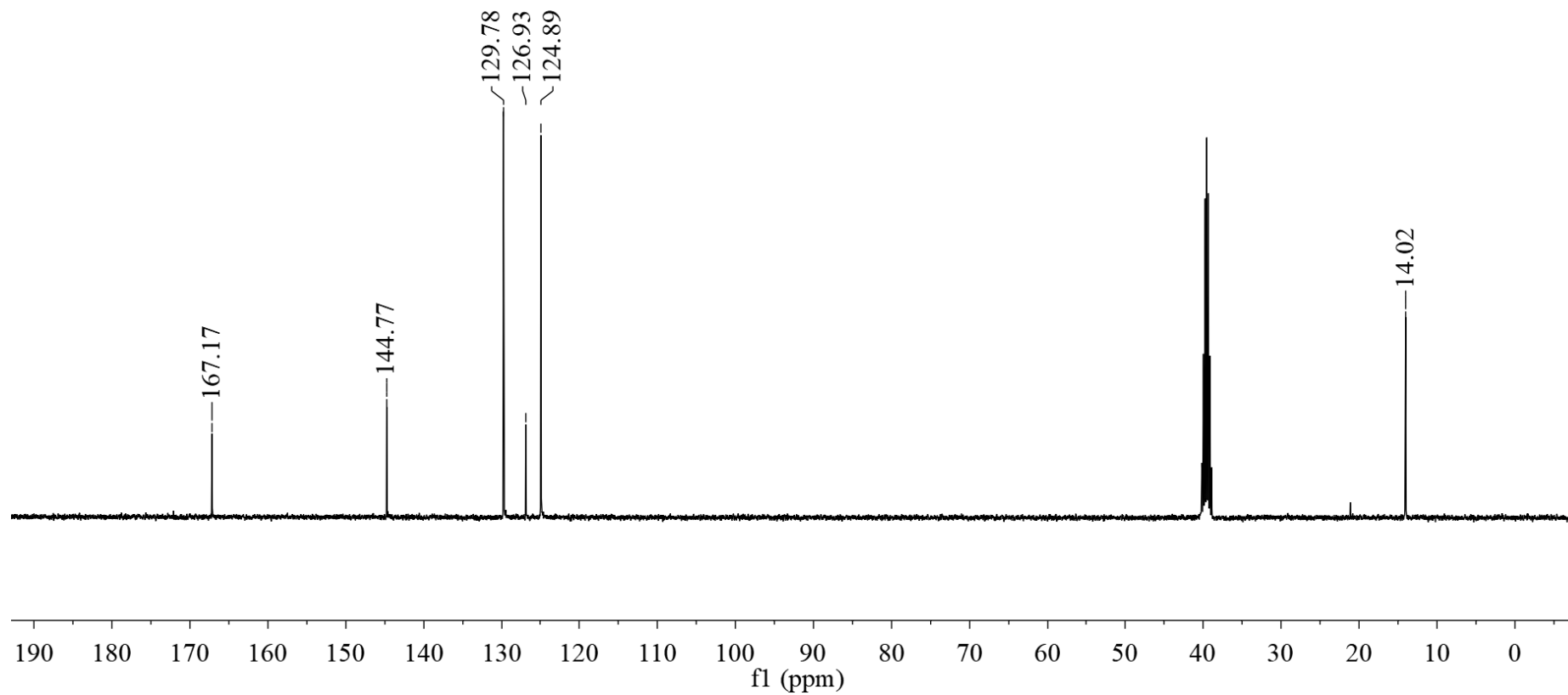
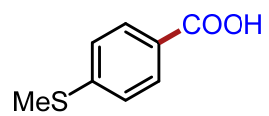




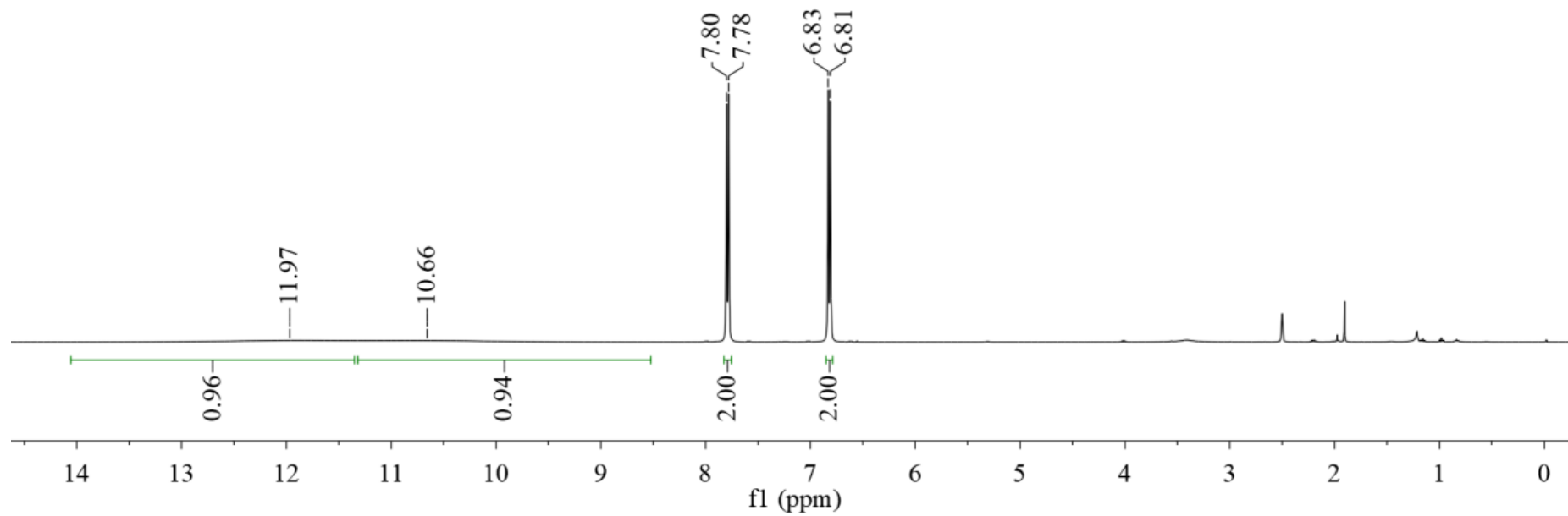
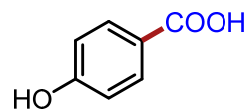
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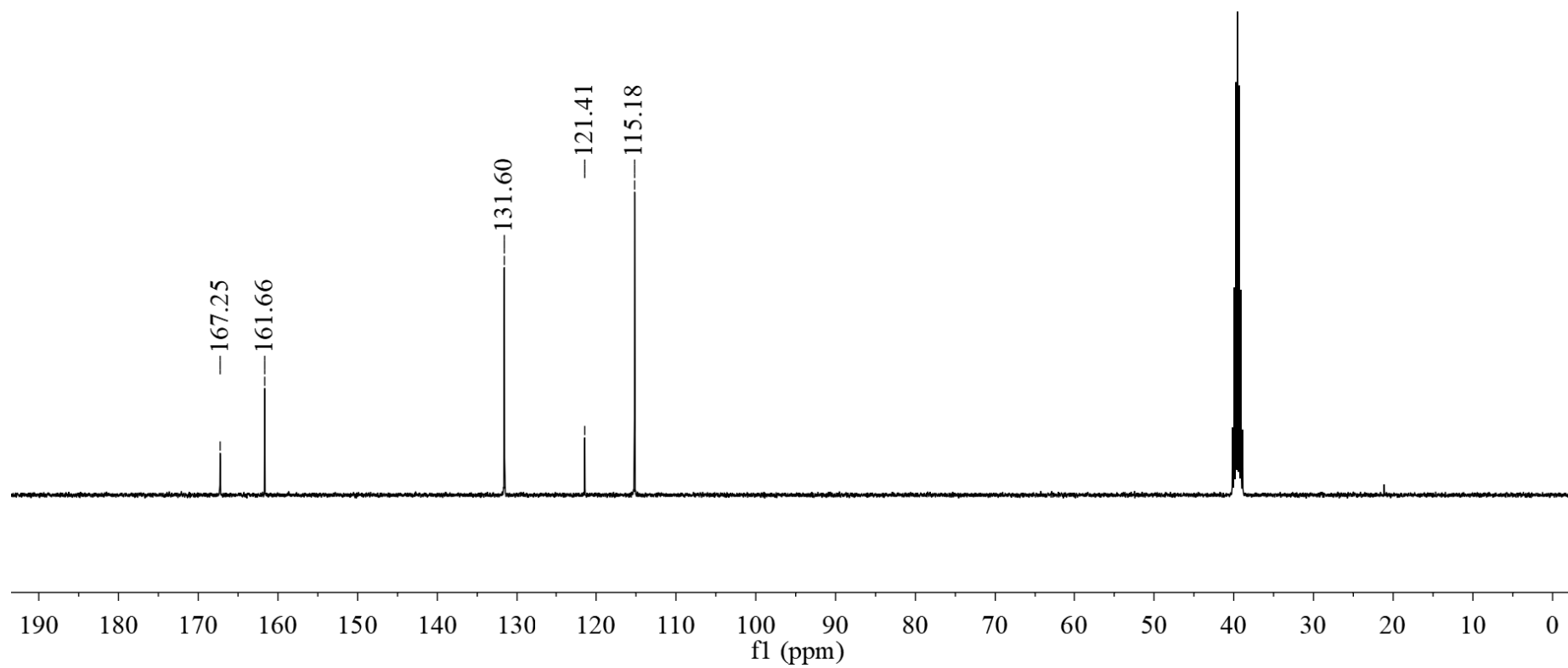
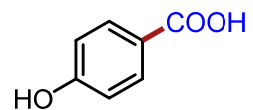
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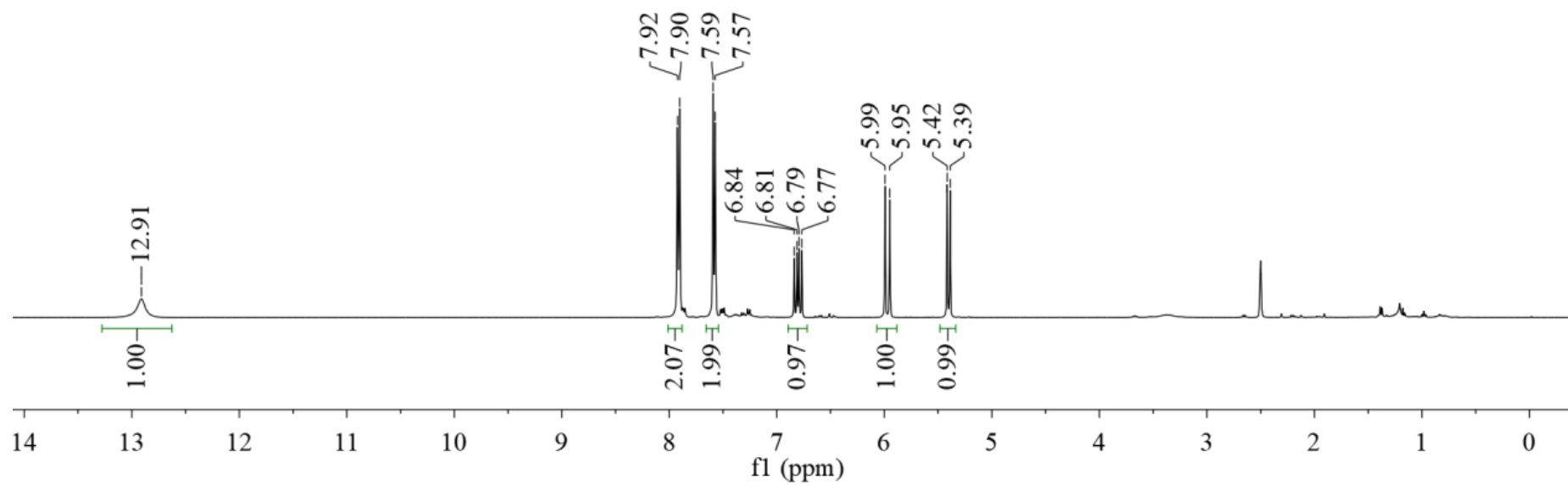
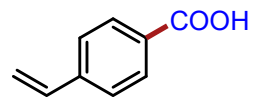
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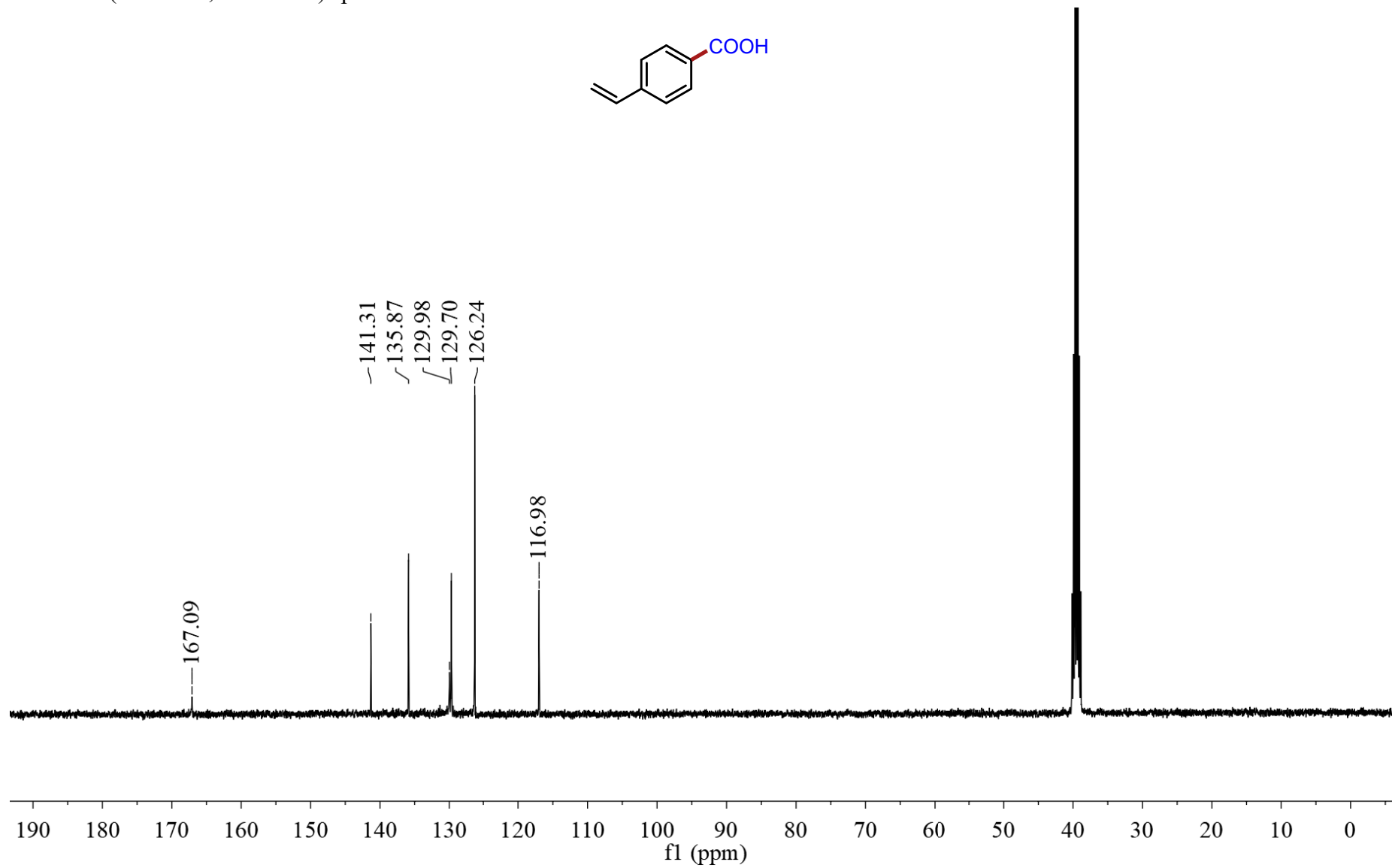
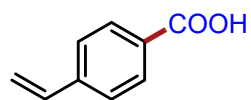
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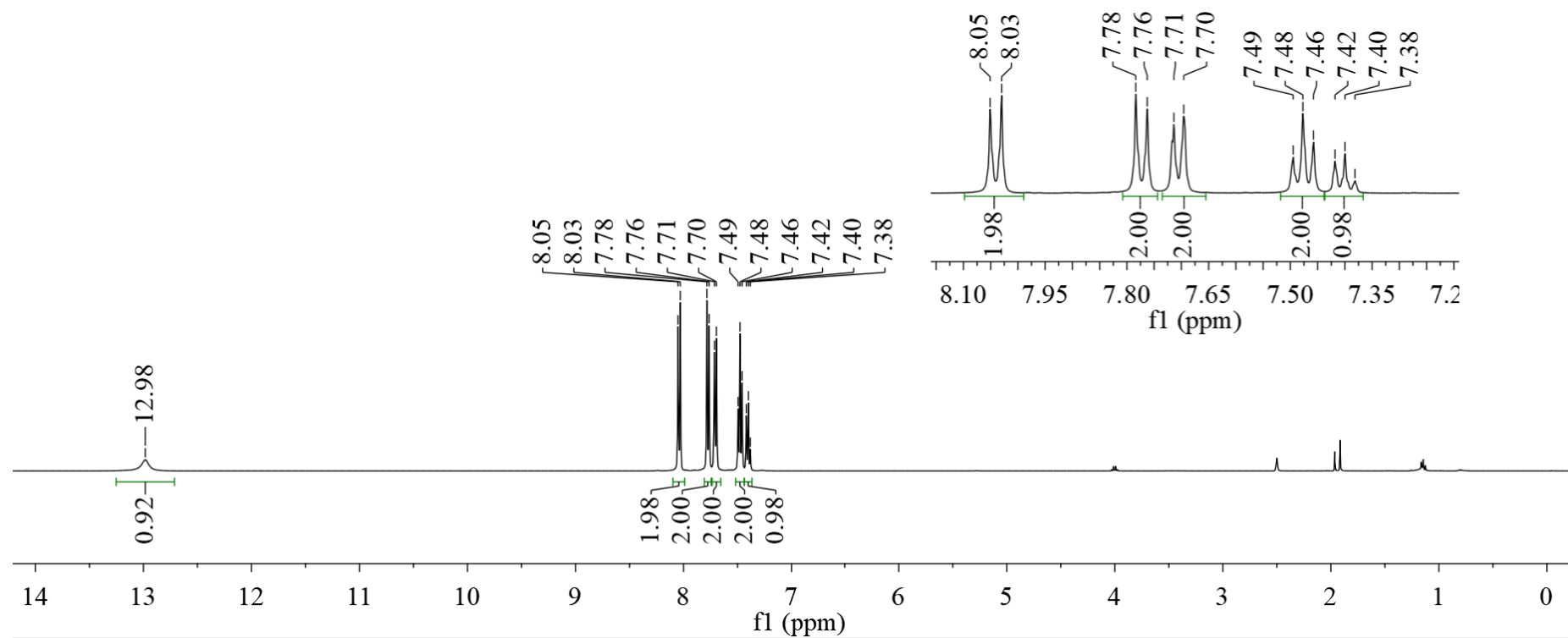
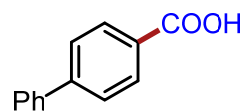
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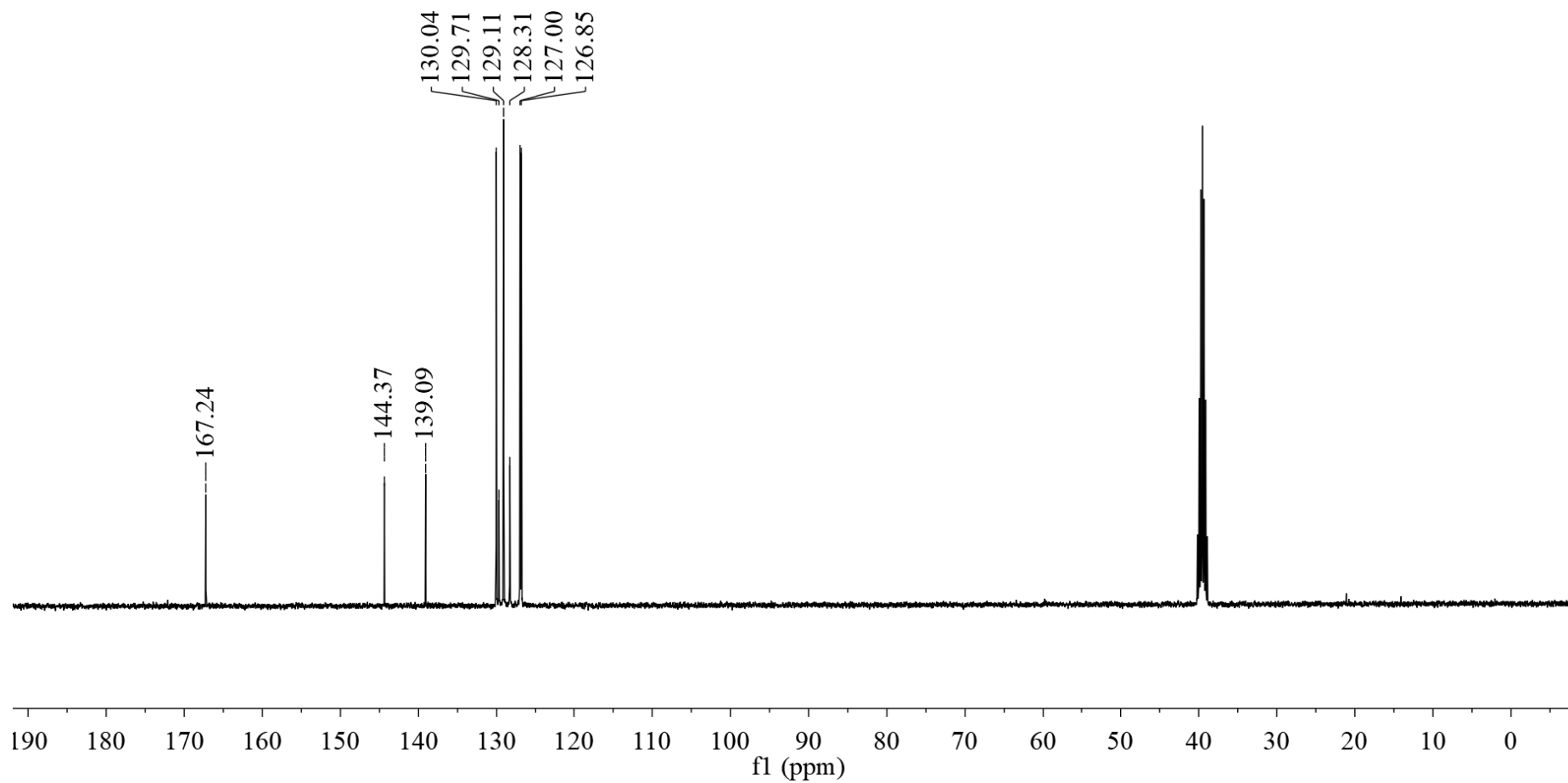
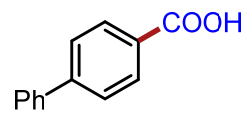
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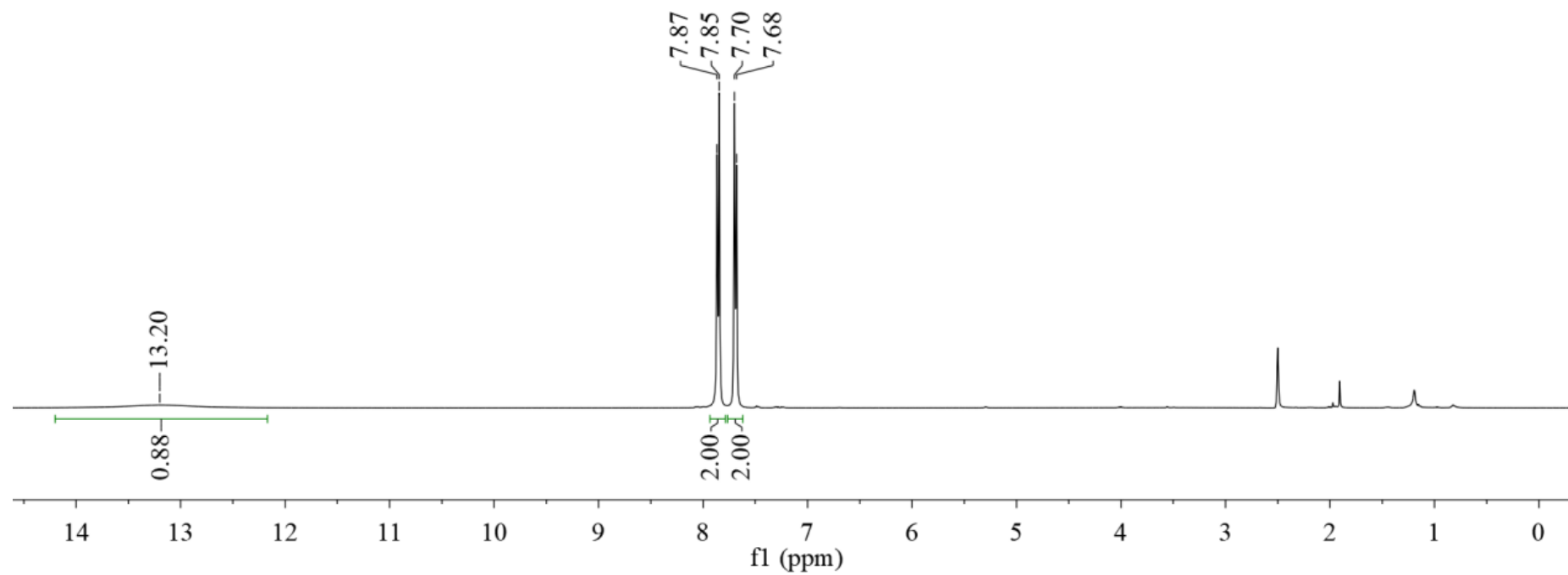
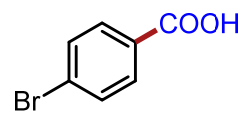


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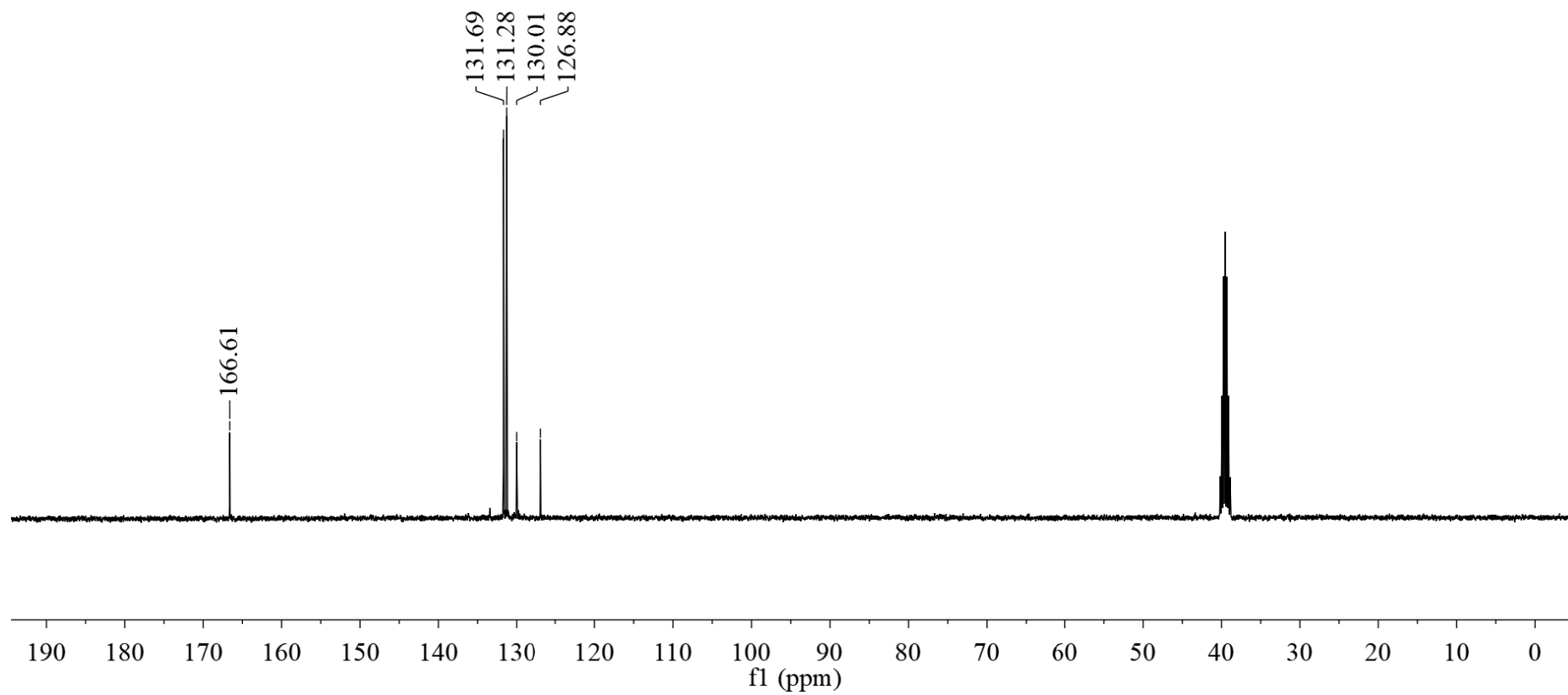
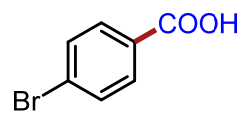




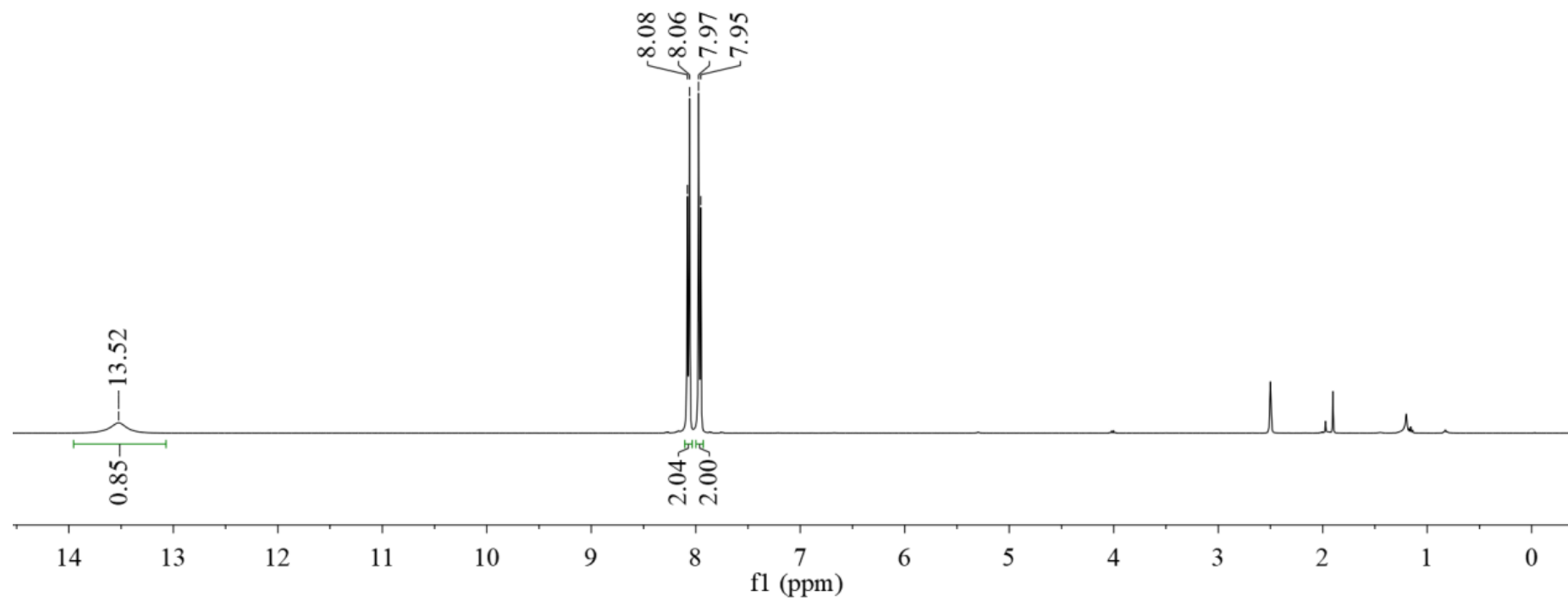
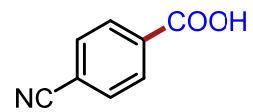
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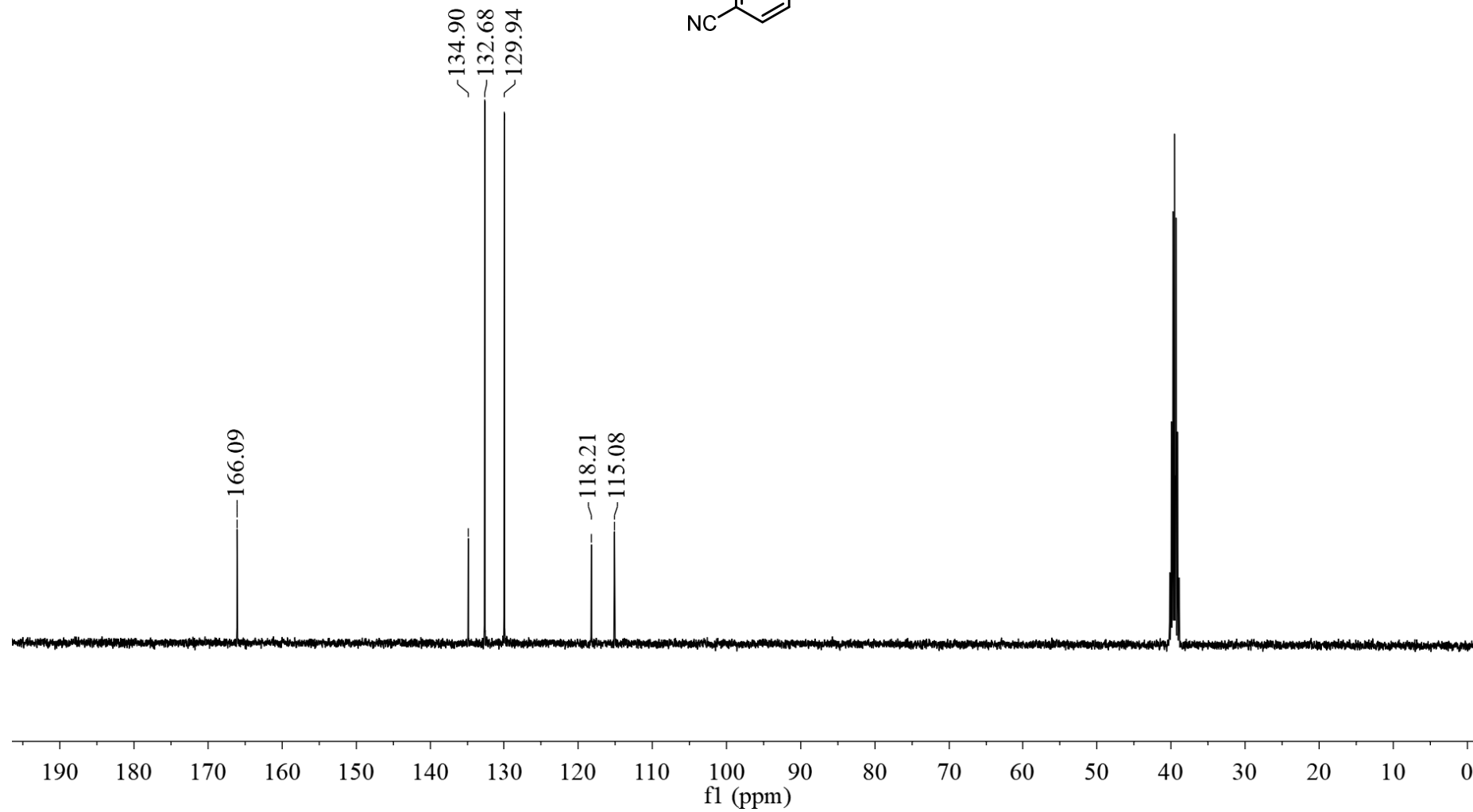
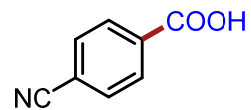
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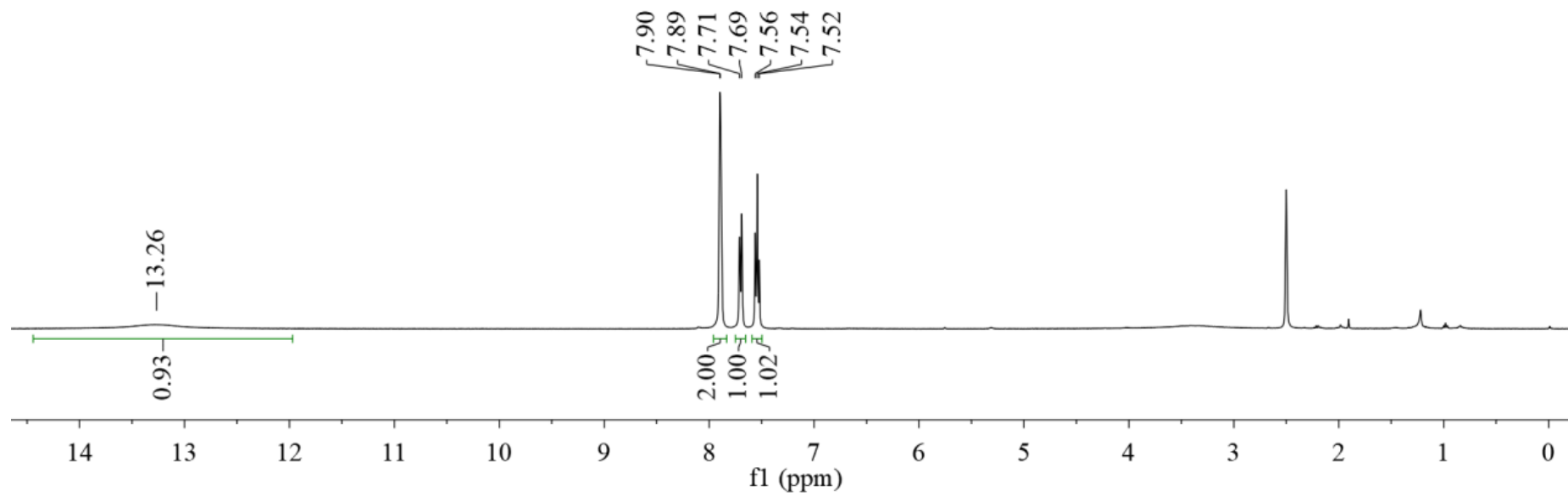
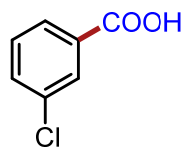
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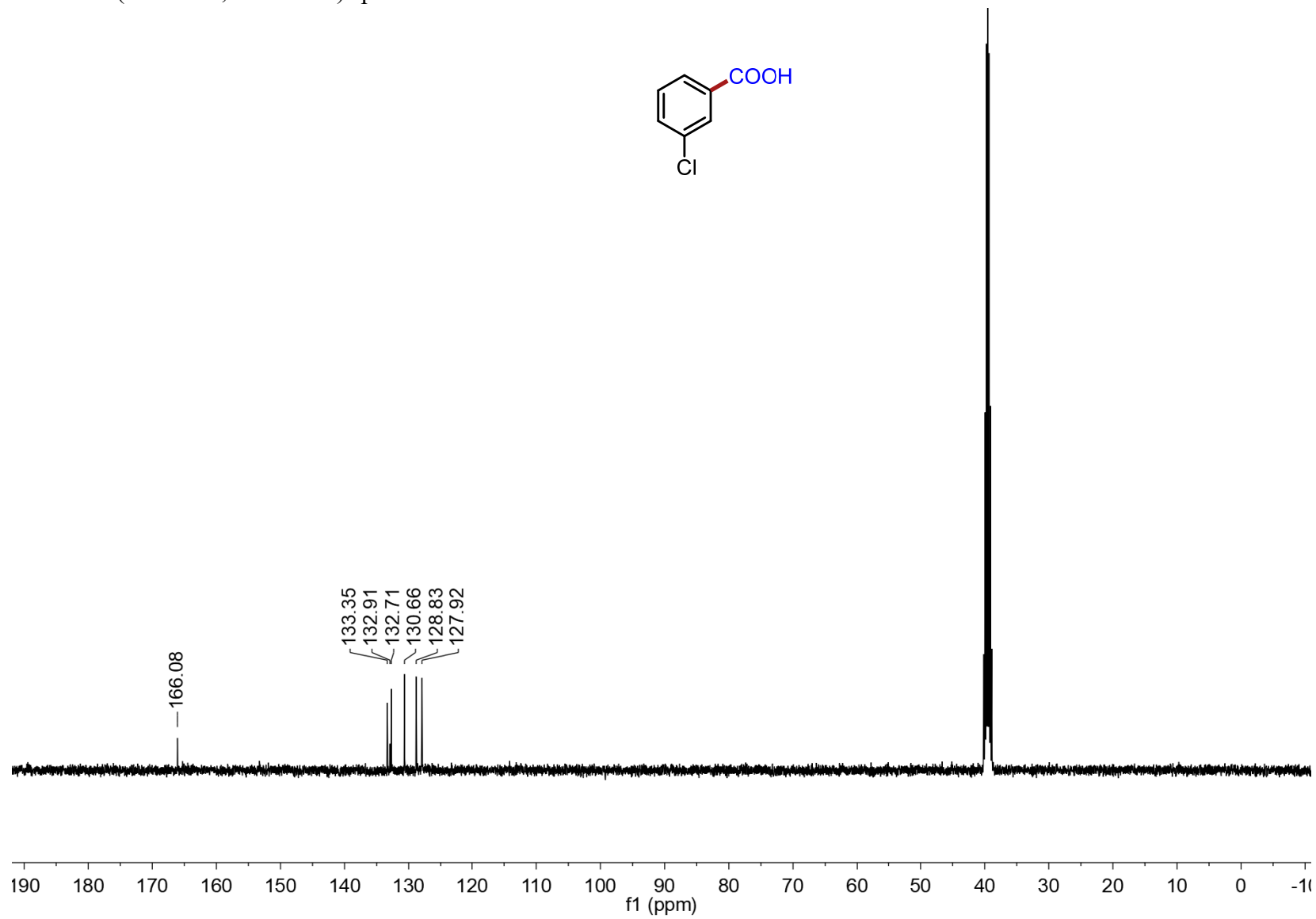
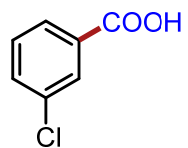
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2k**



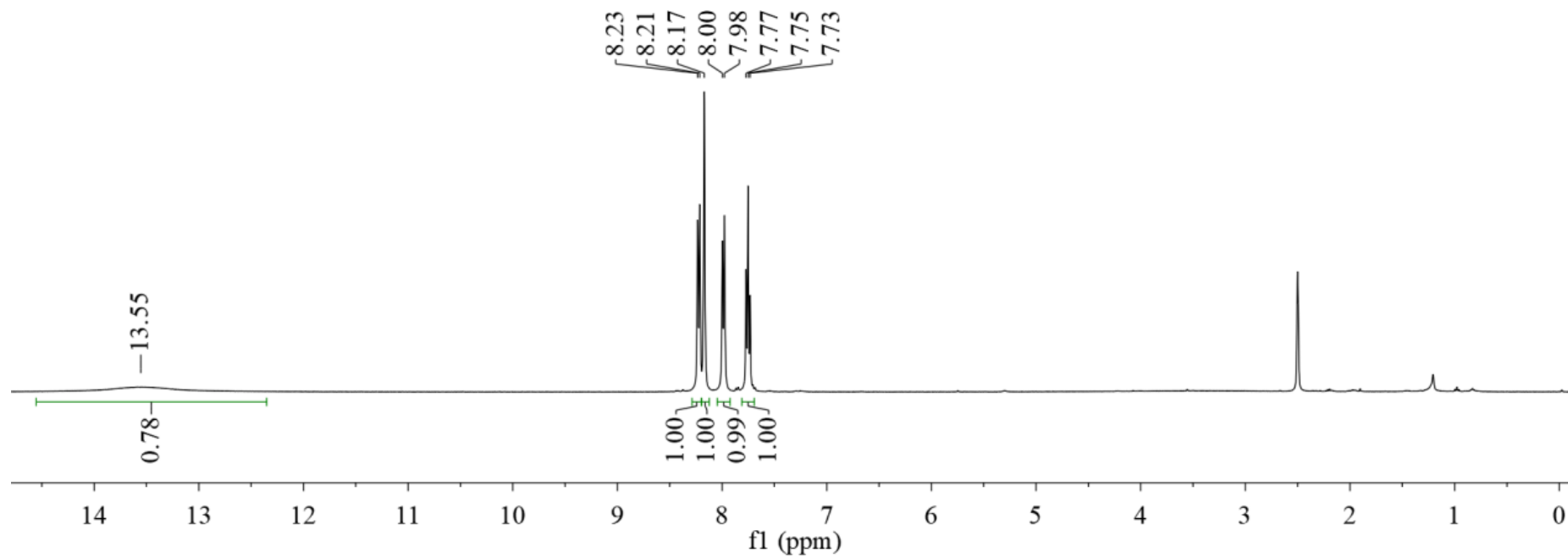
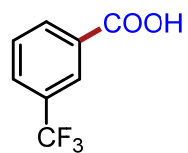
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **21**



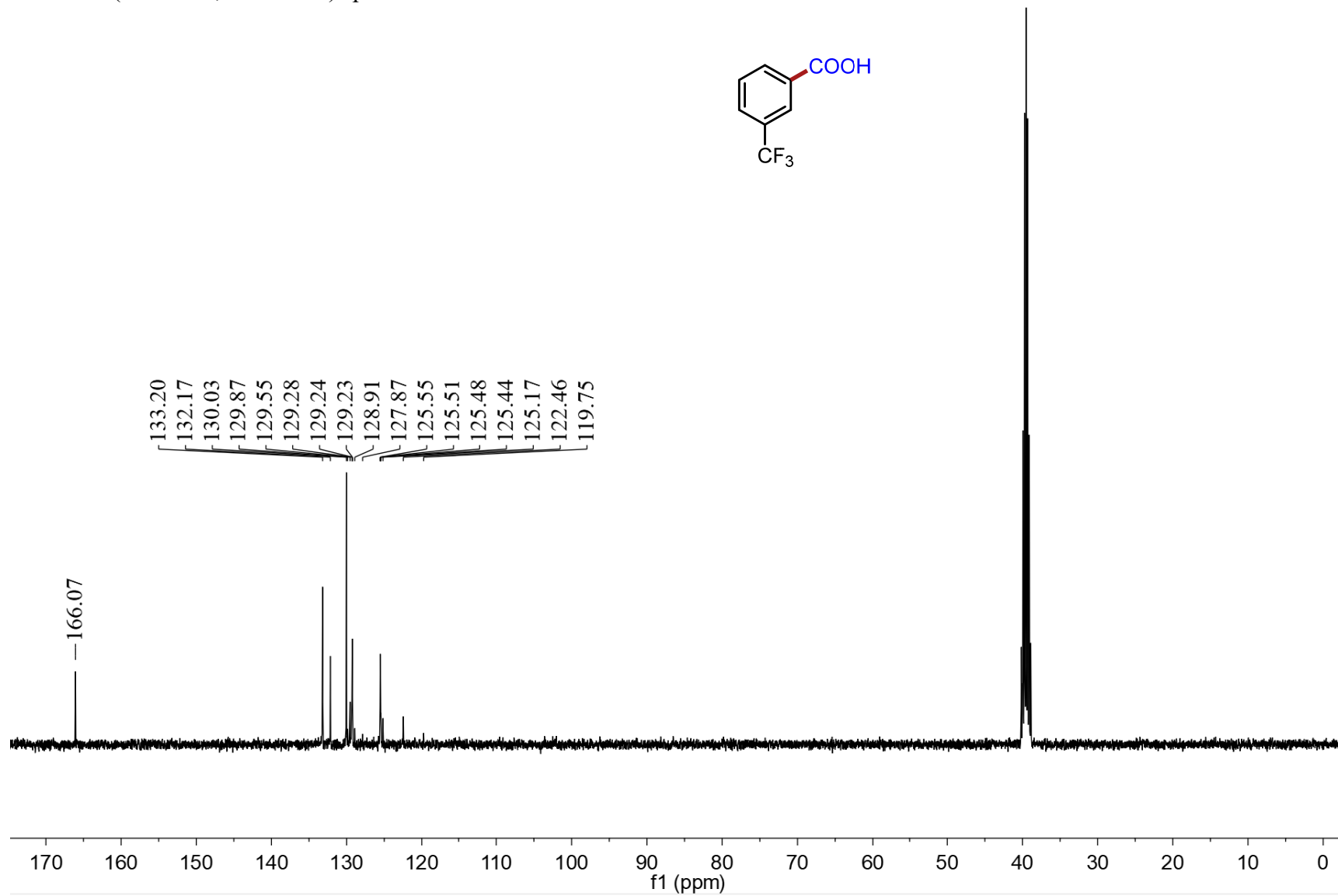
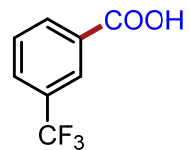
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **21**



$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2m**

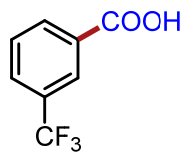


$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2m**

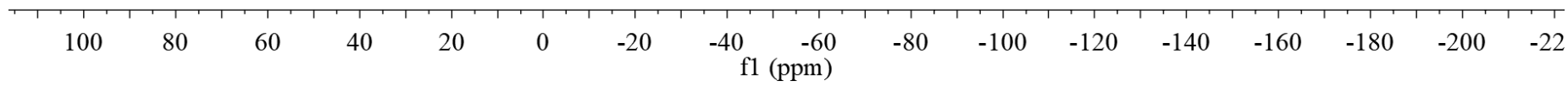




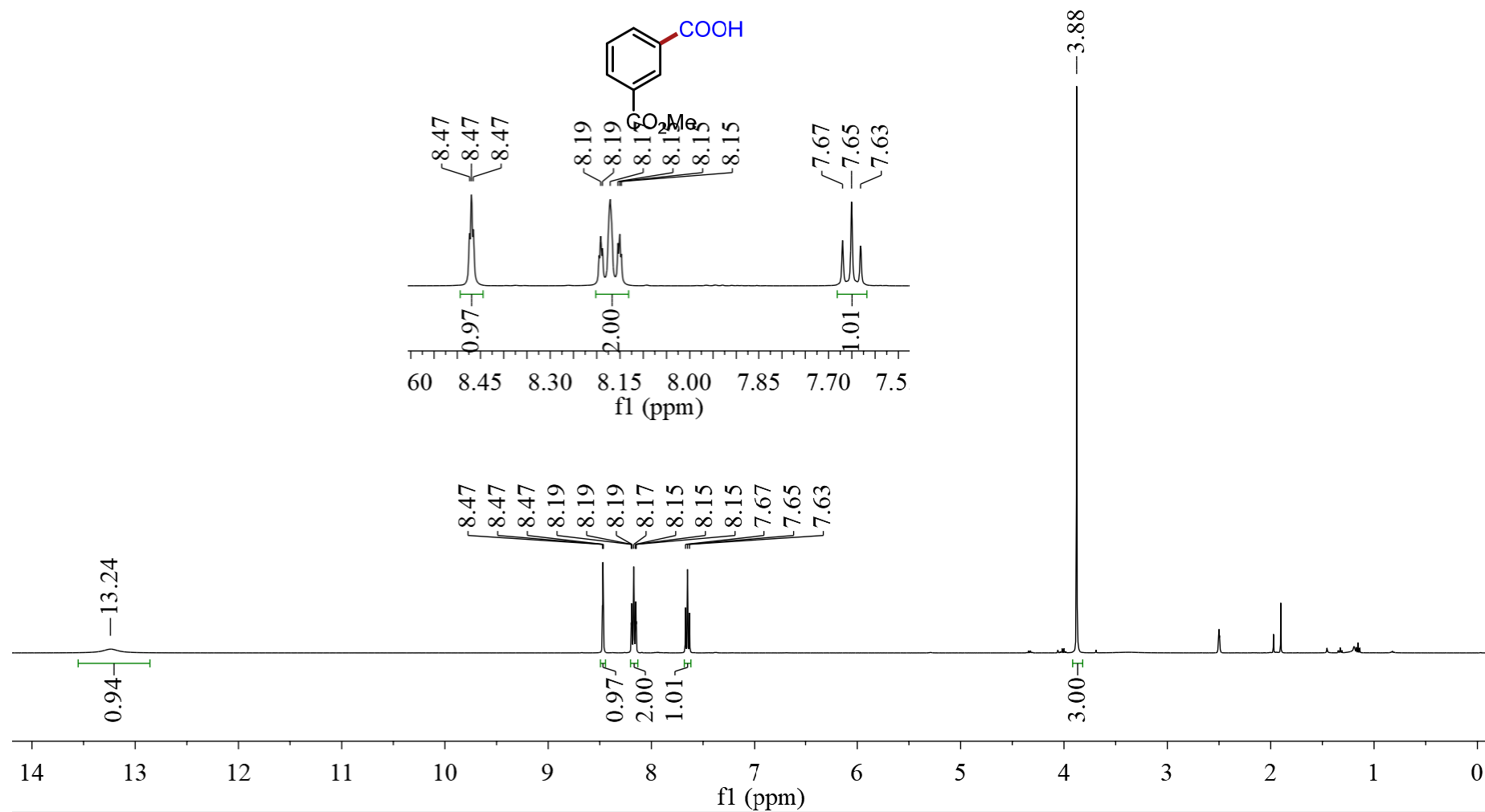
$^{19}\text{F}$  NMR (376 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2m**



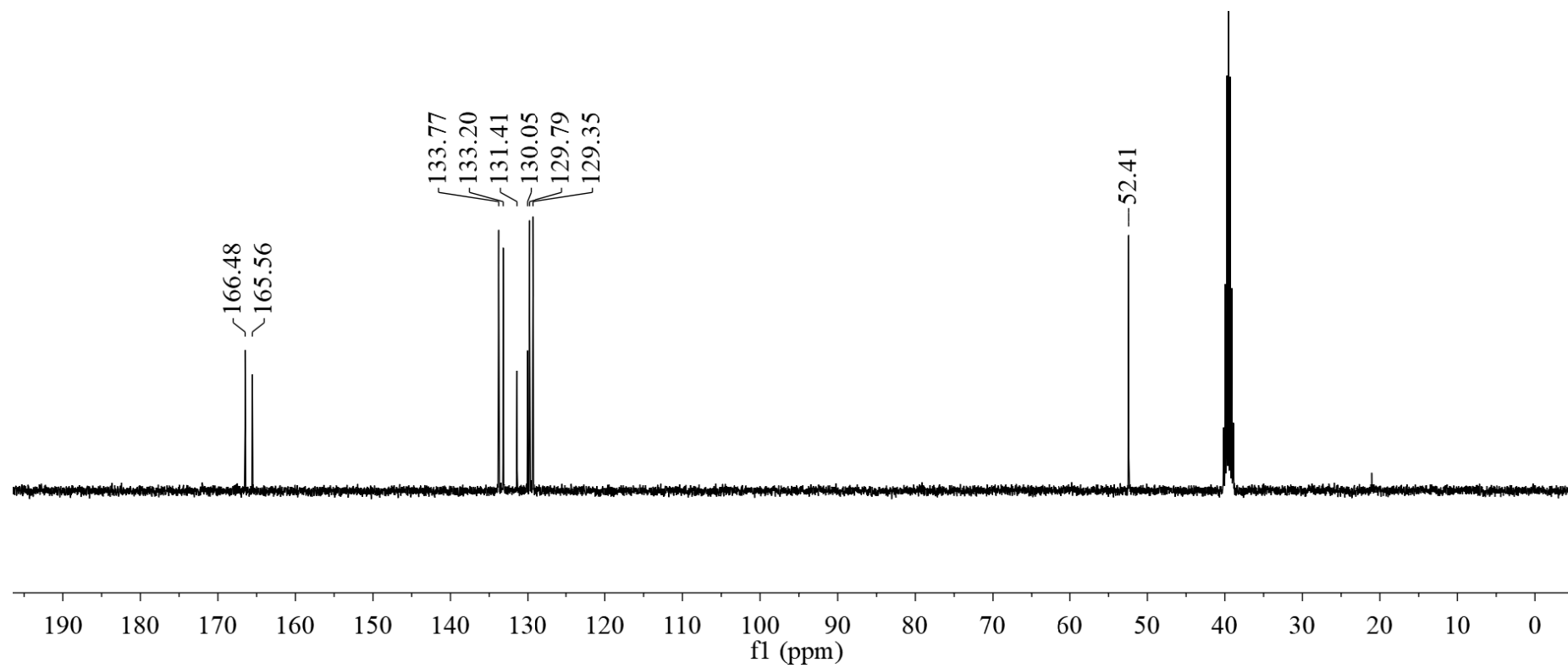
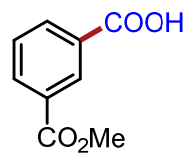
-61.43



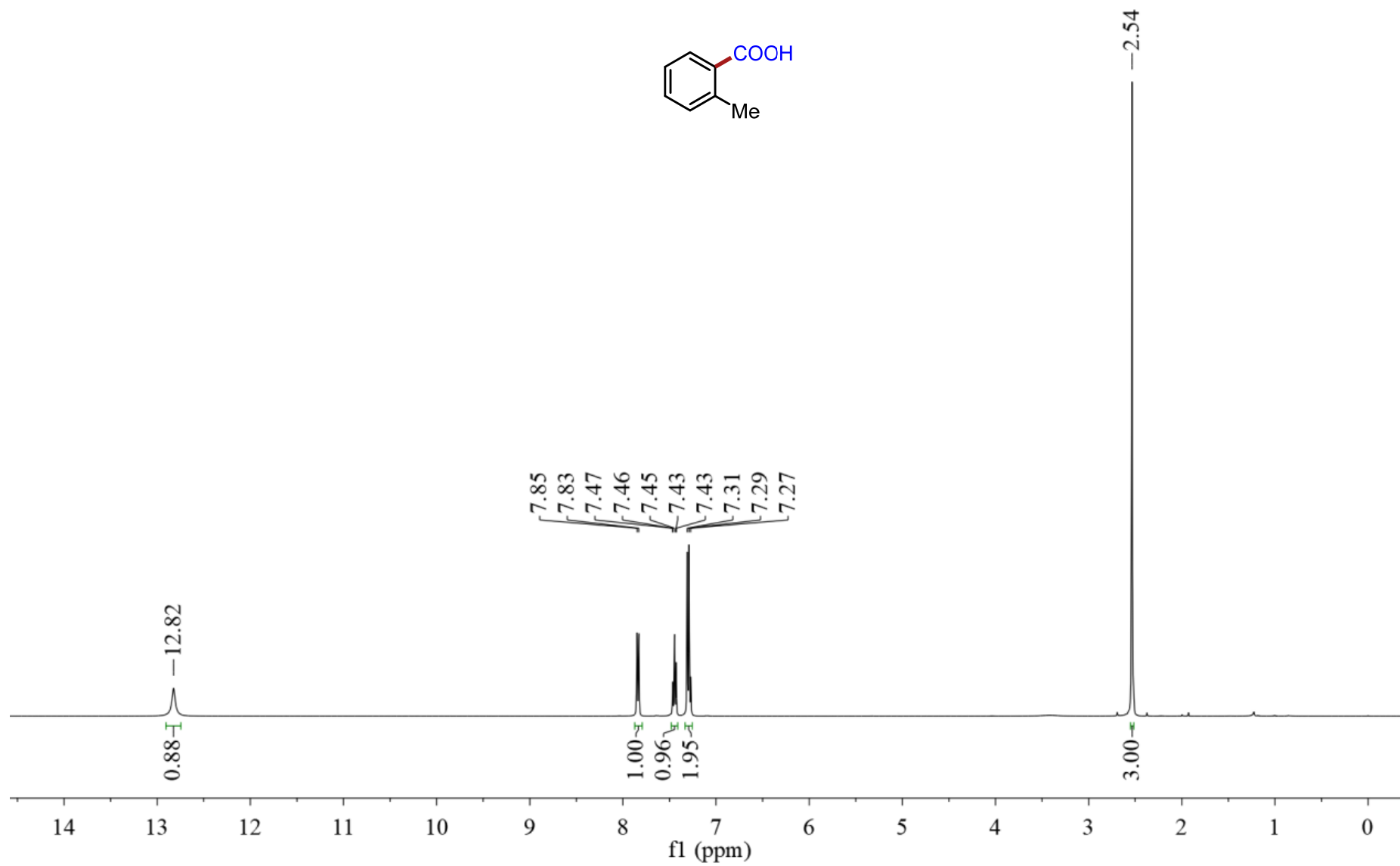
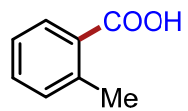
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2n**



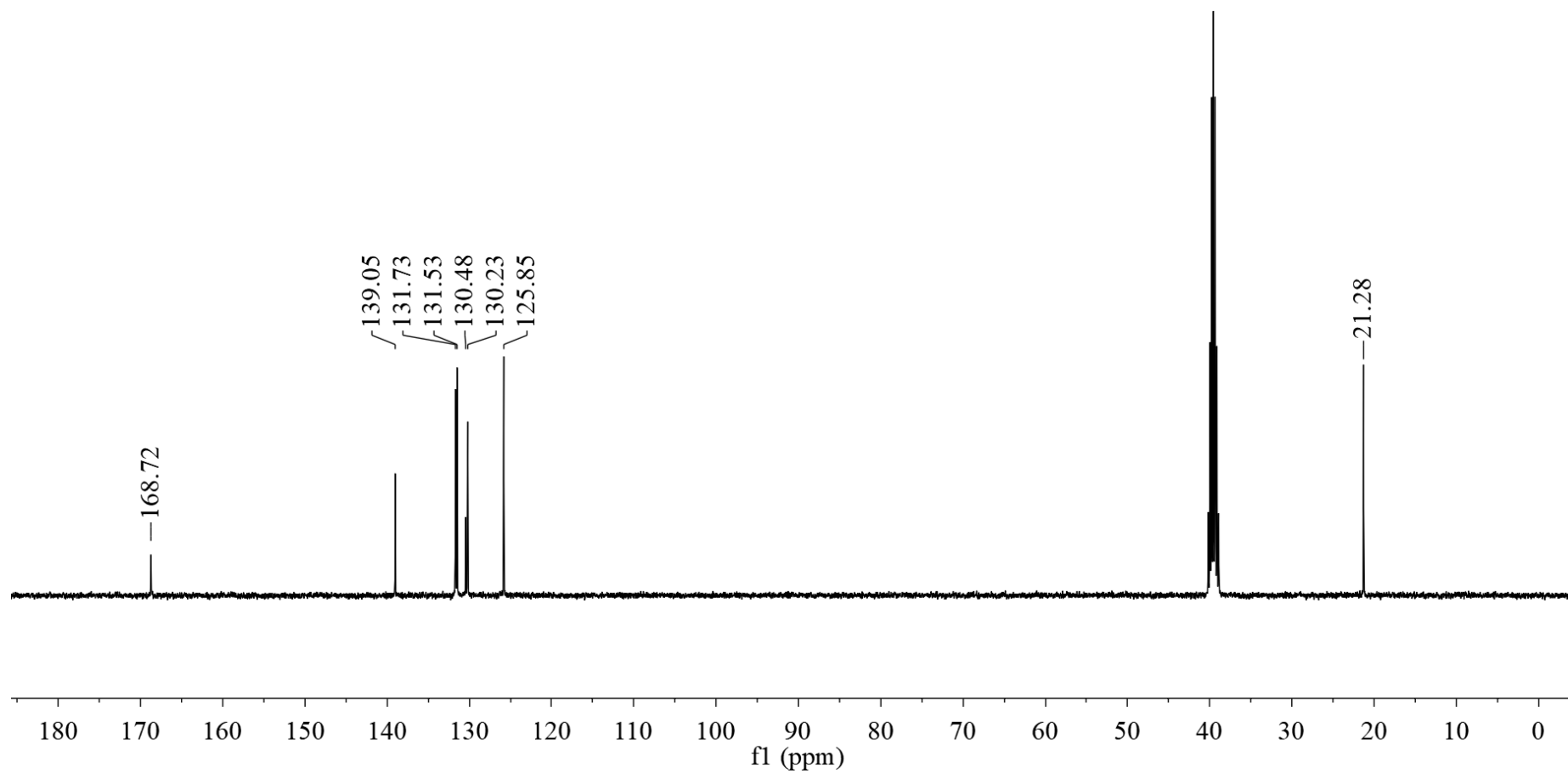
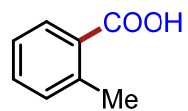
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2n**



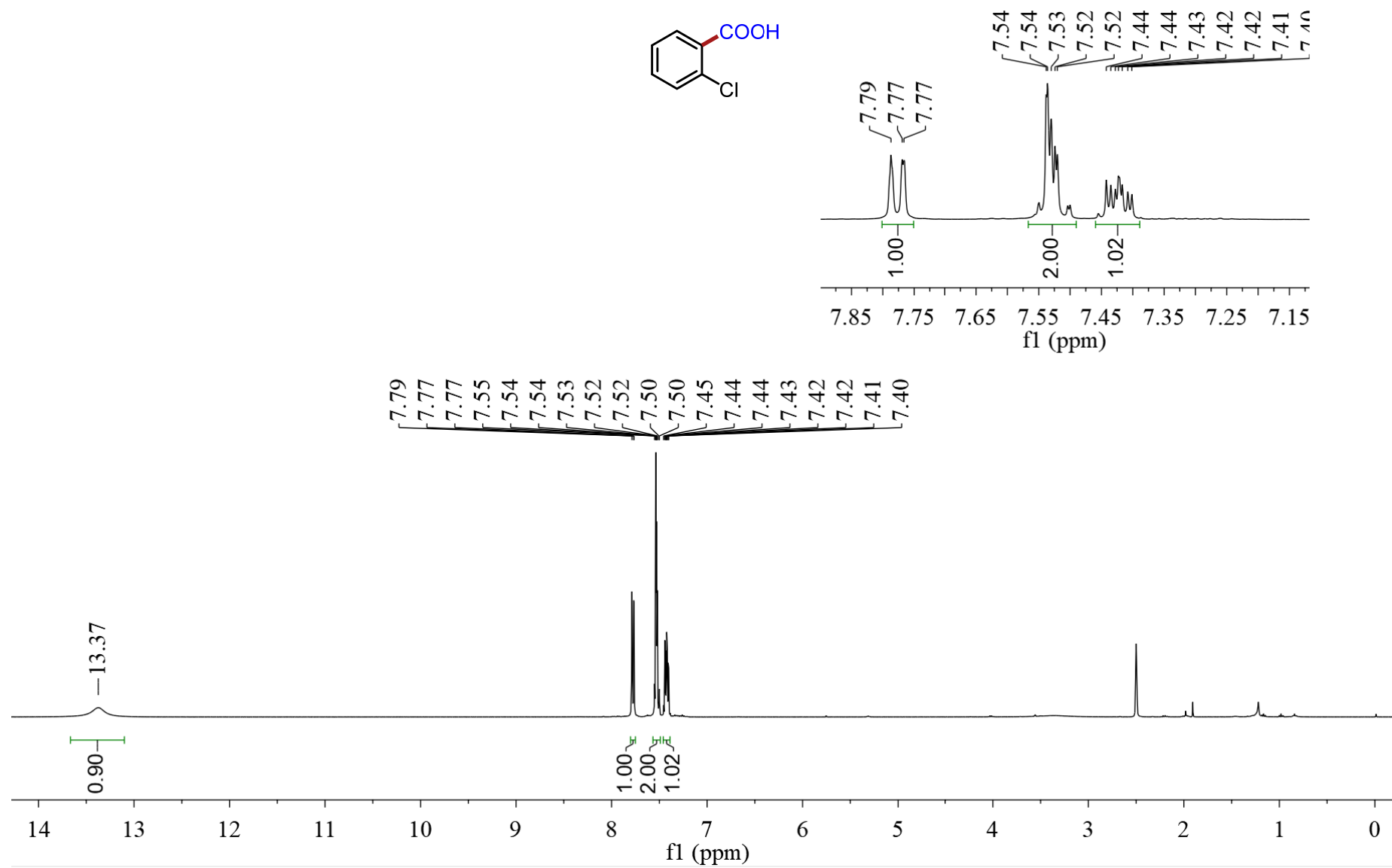
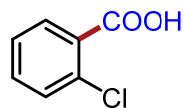
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2o**



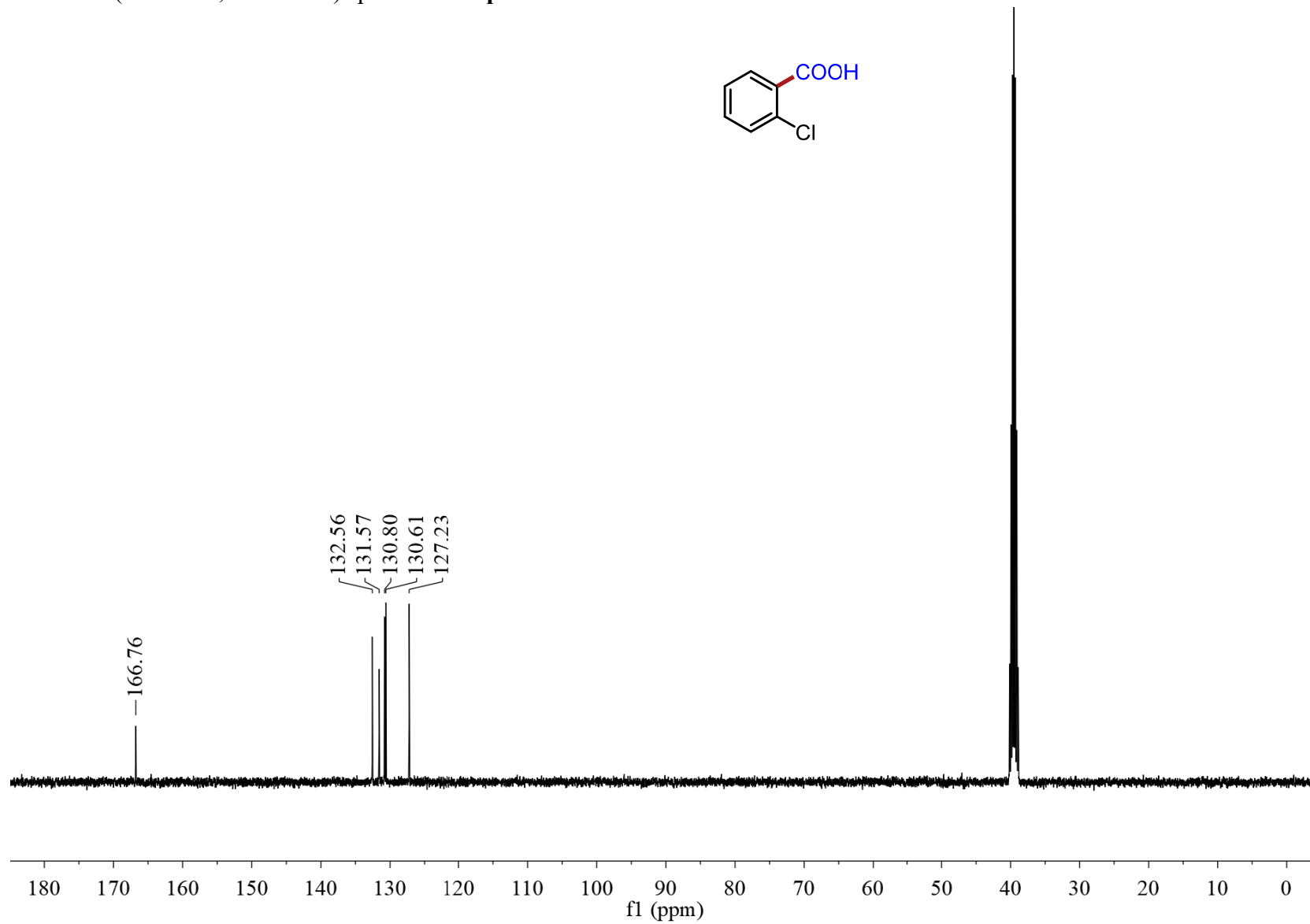
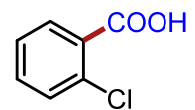
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2o**



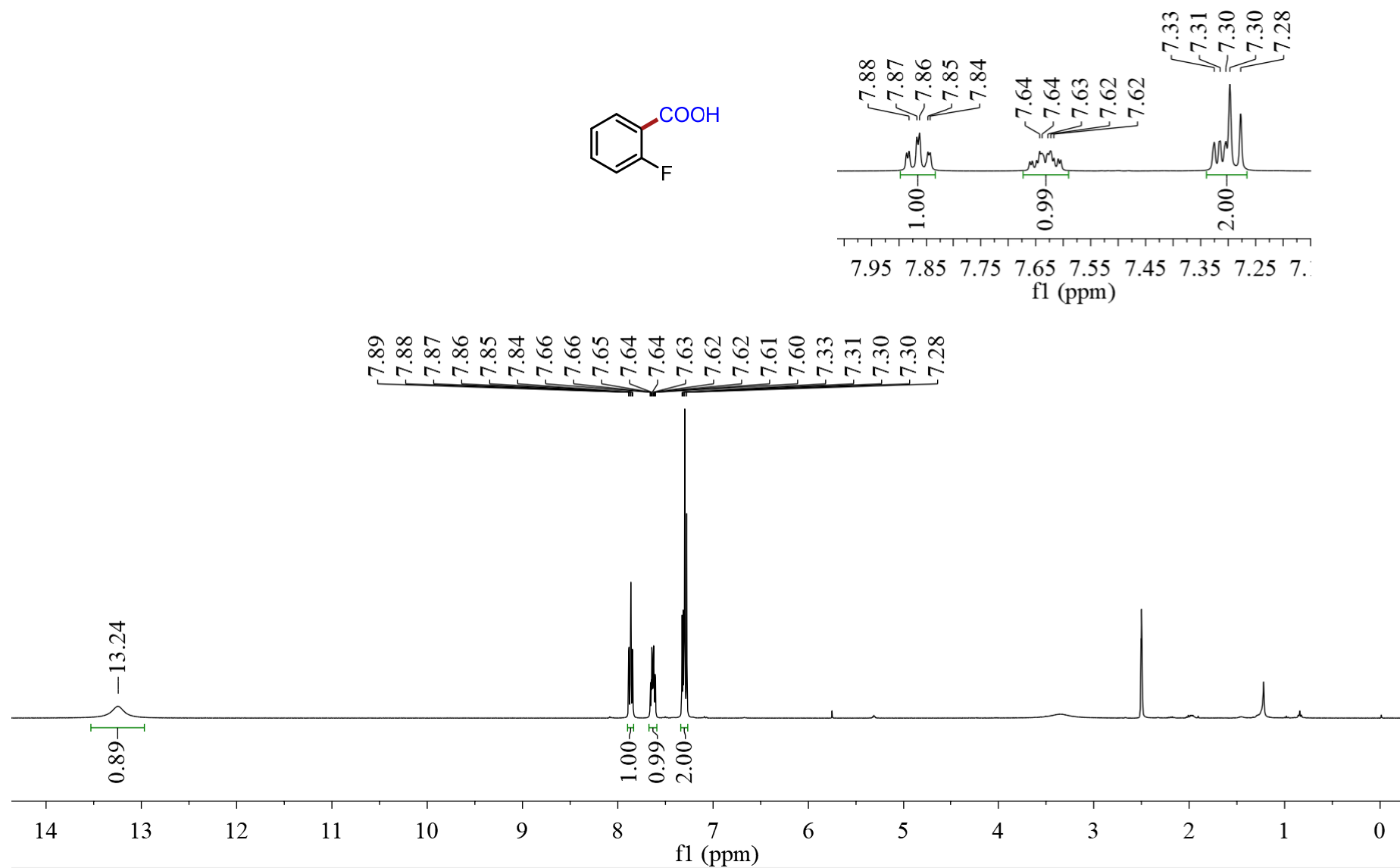
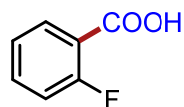
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2p**



$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2p**

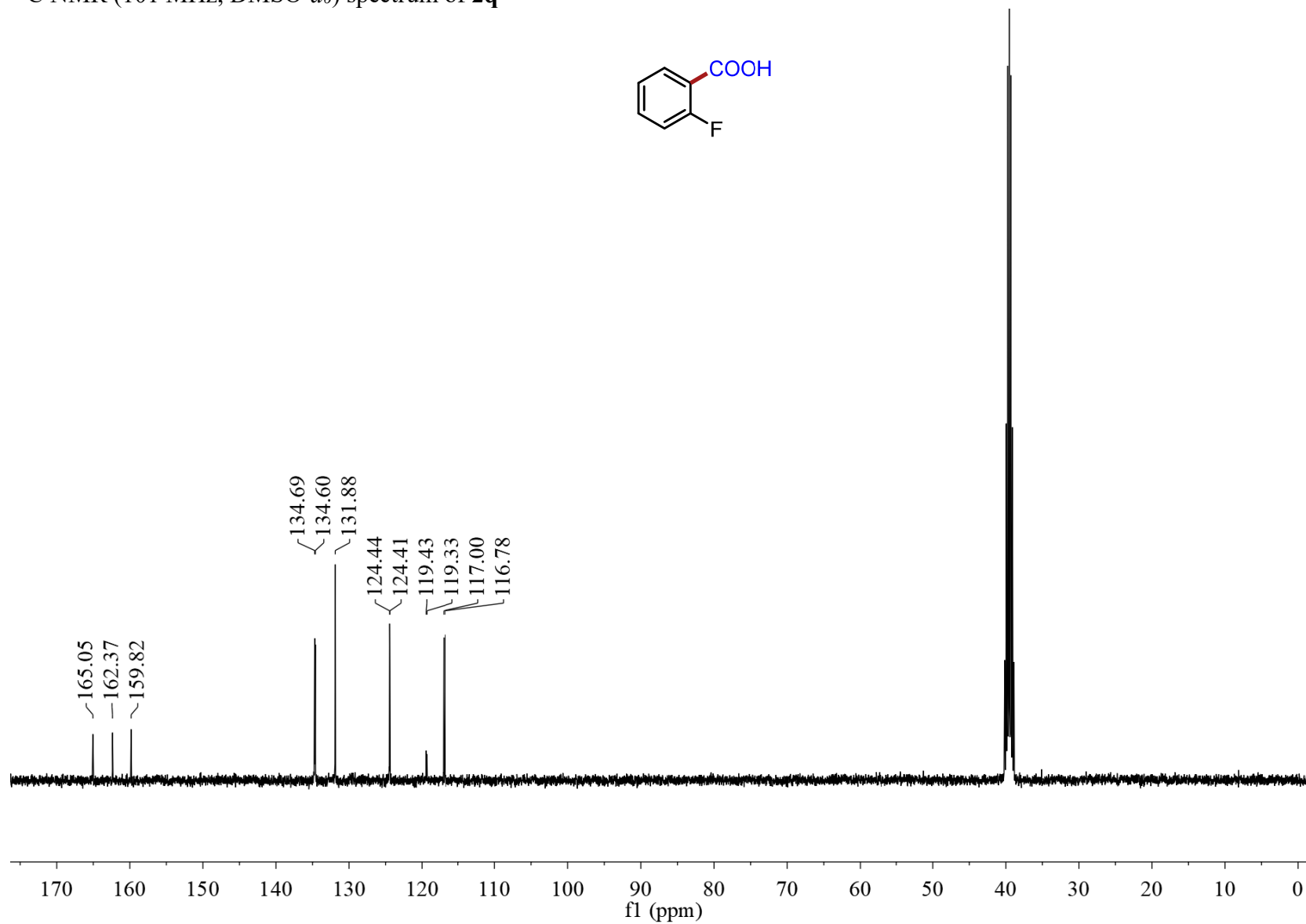
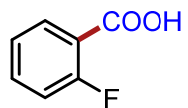


$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2q**

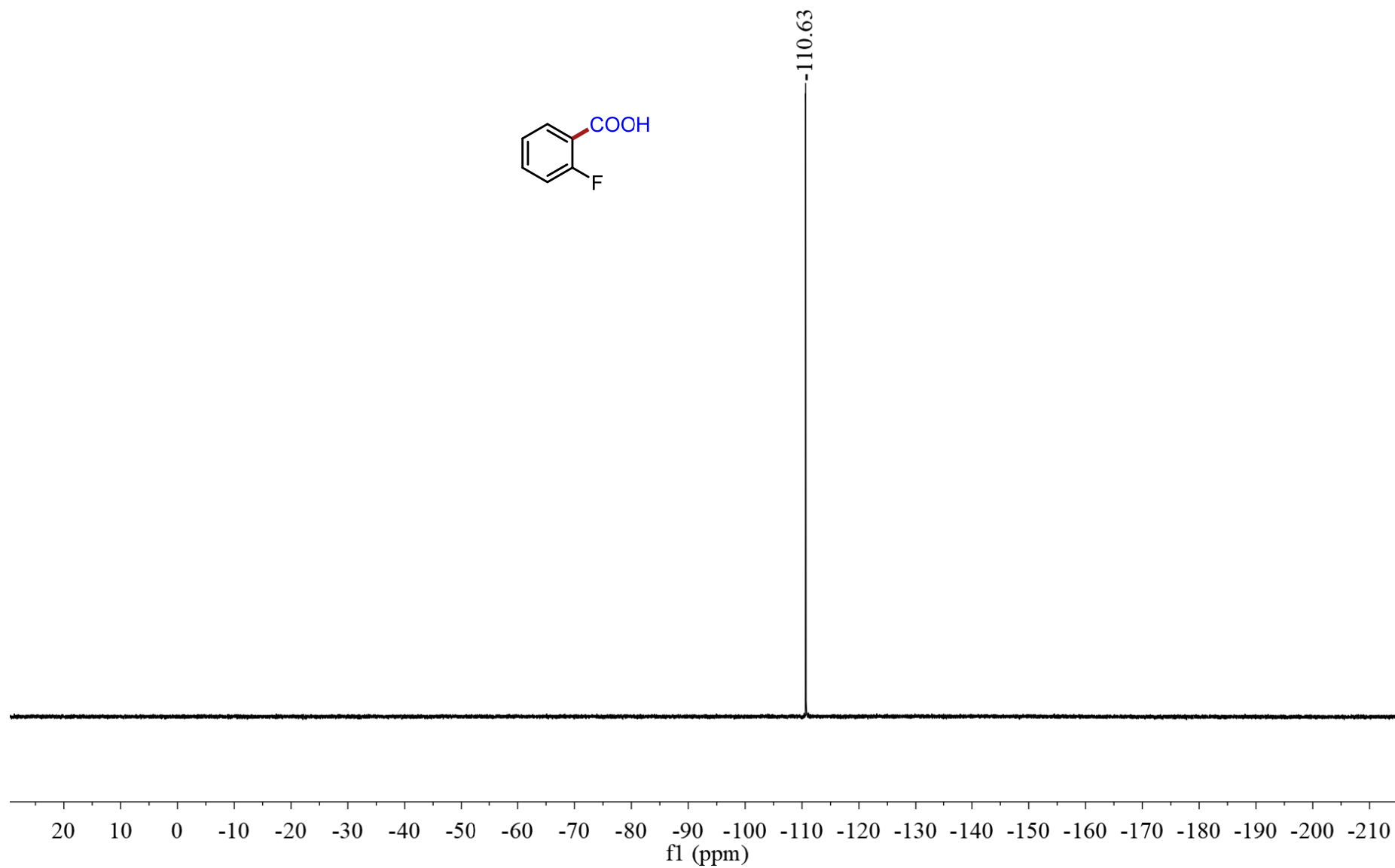
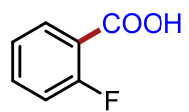




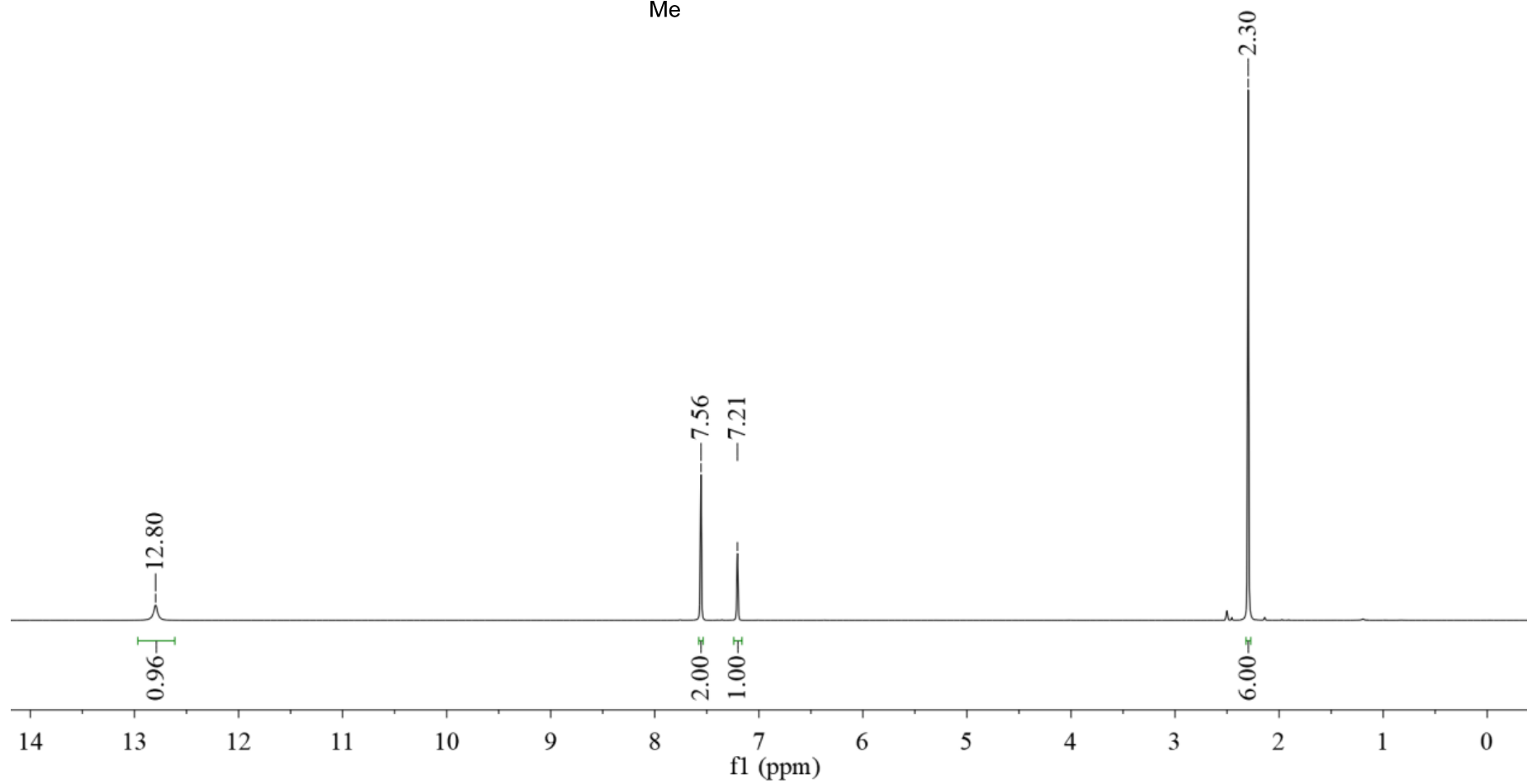
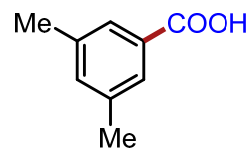
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2q**



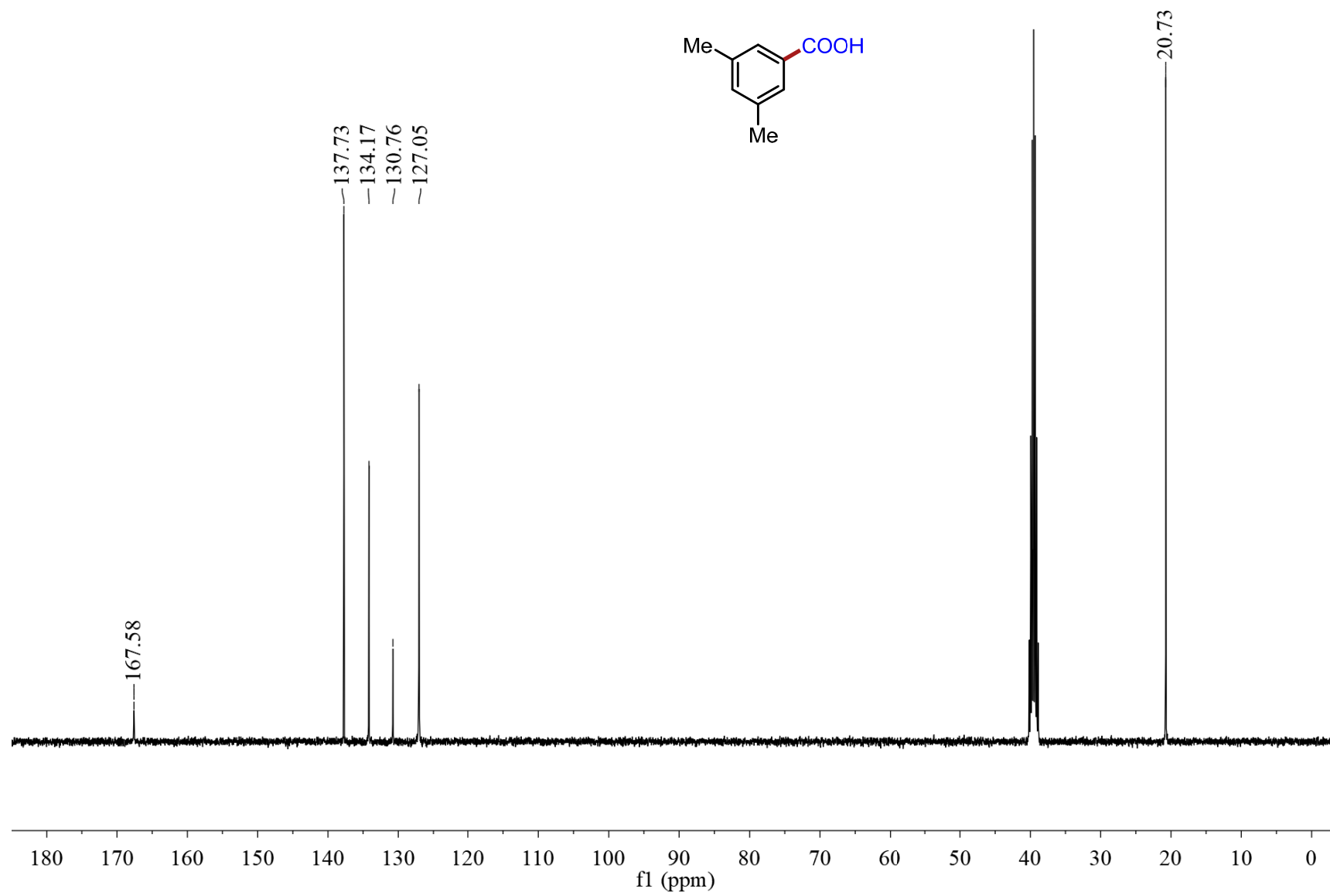
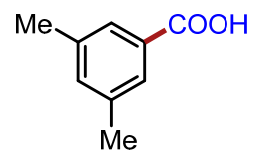
$^{19}\text{F}$  NMR (376 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2q**



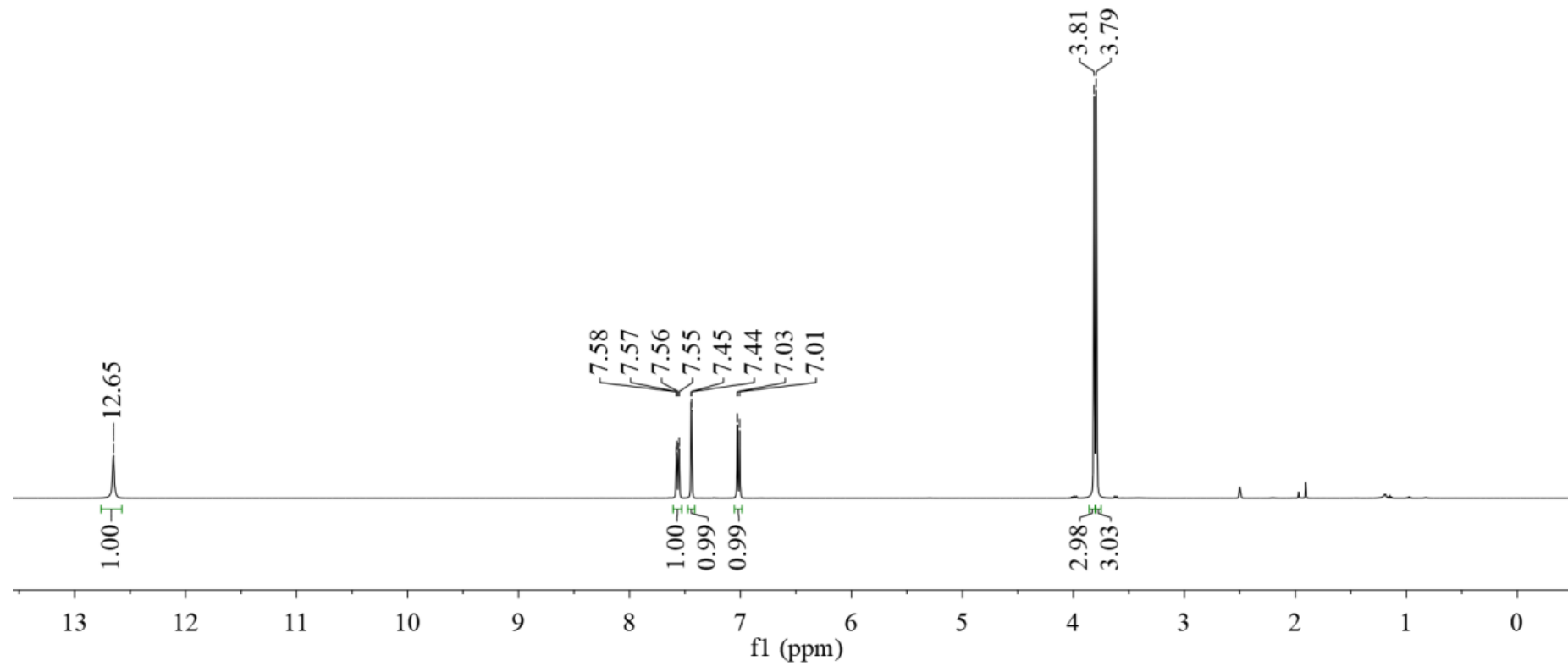
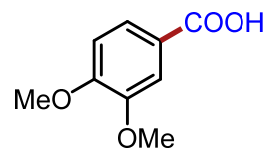
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2r**



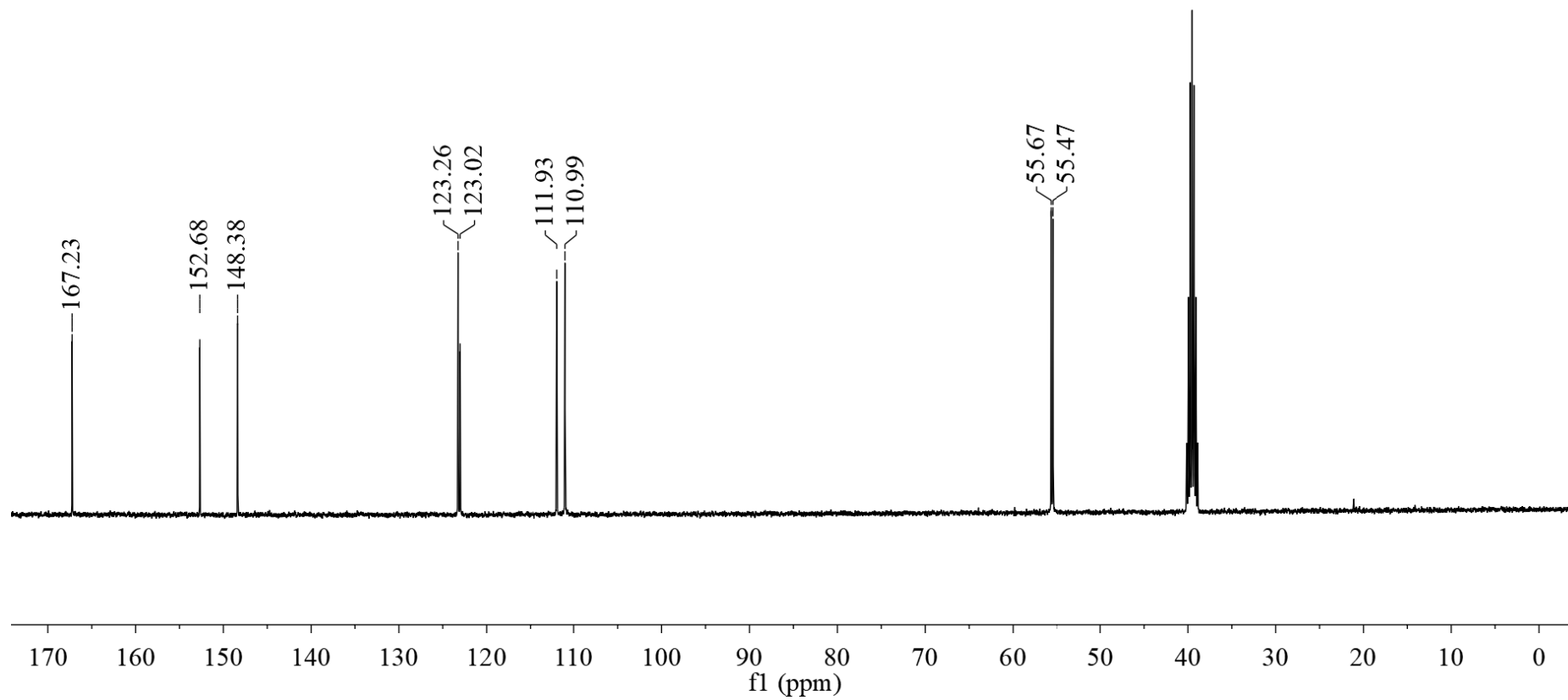
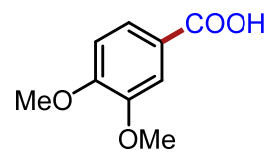
<sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>) spectrum of **2r**



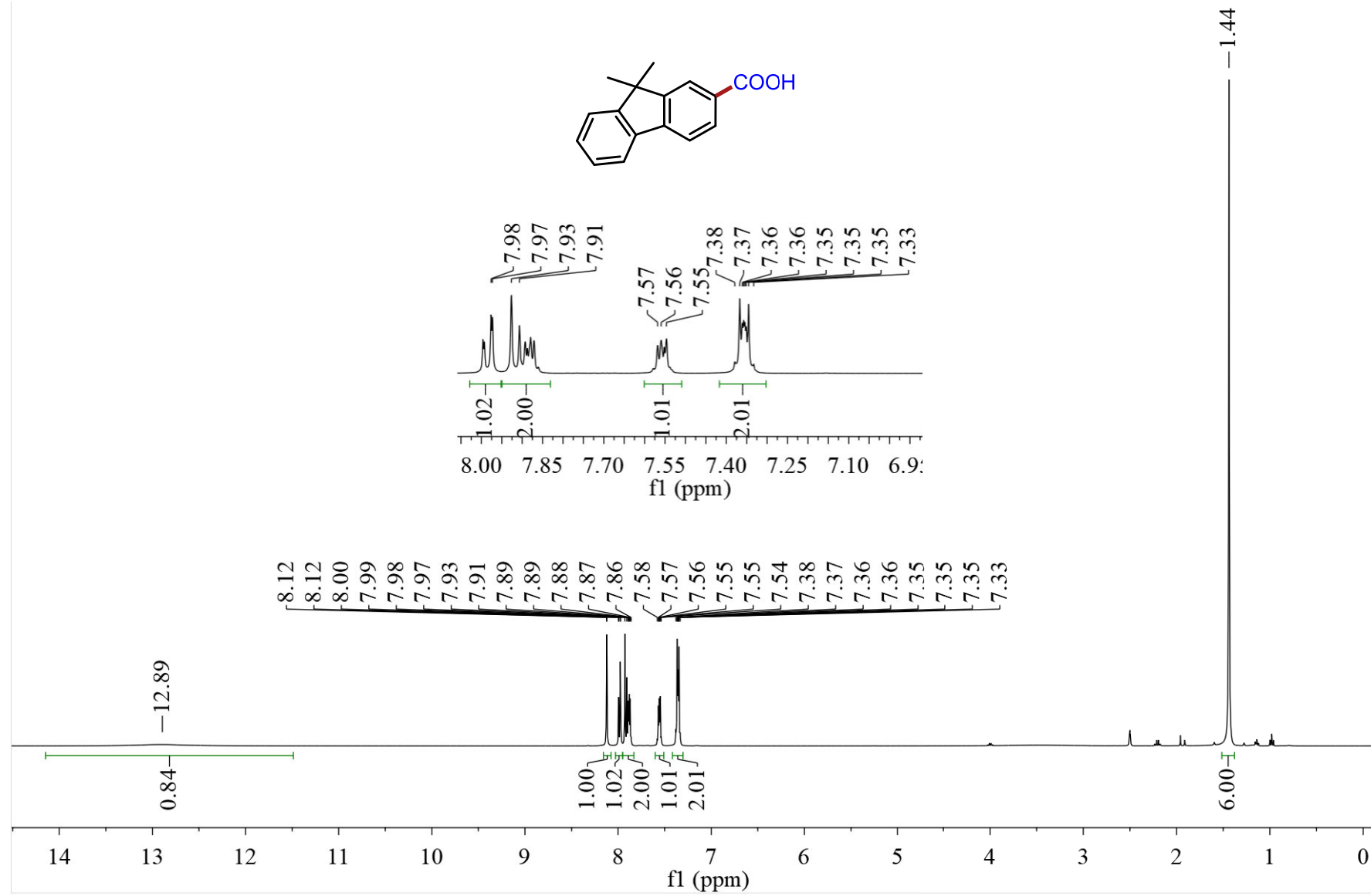
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2s**



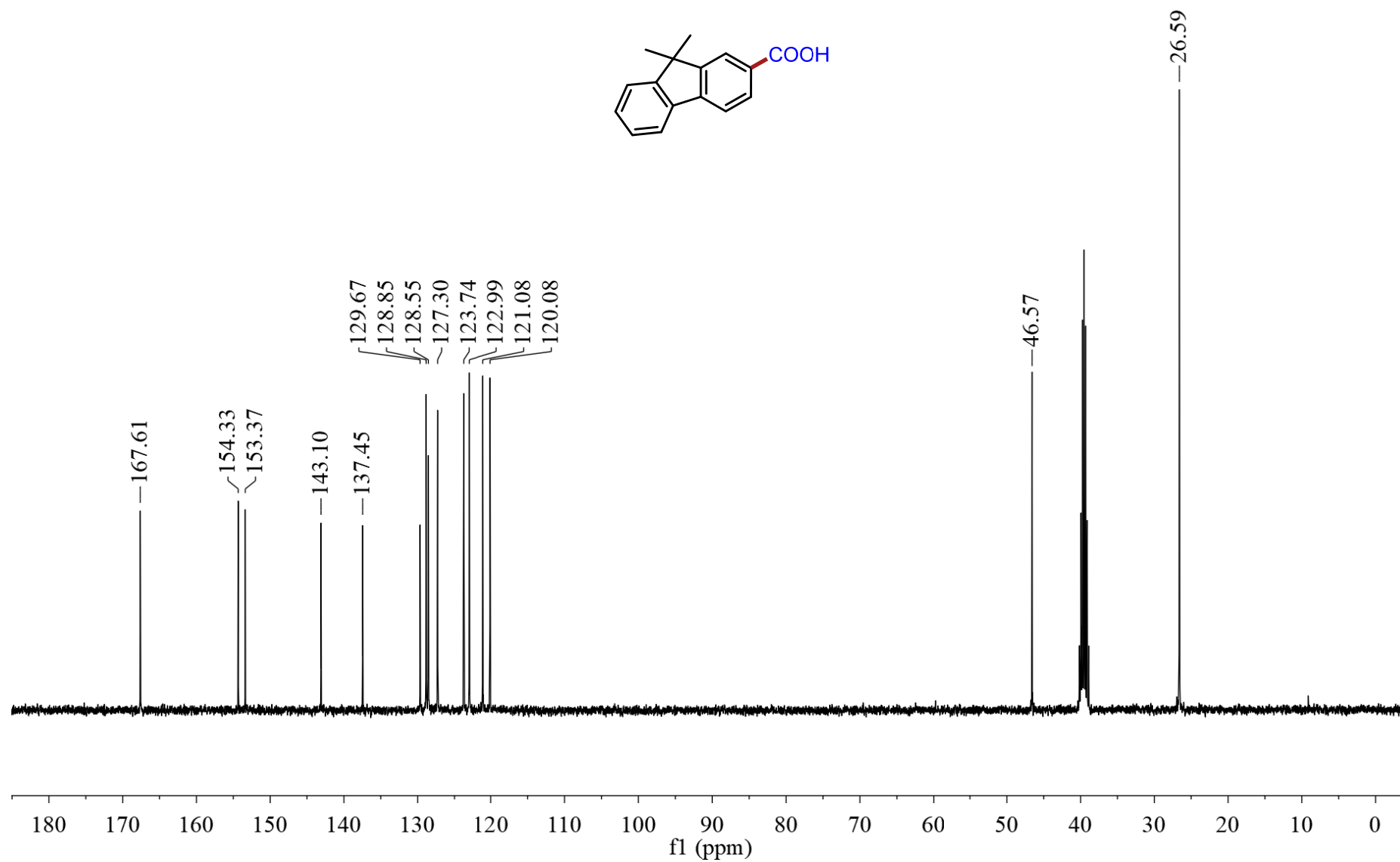
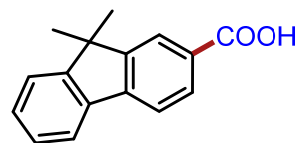
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2s**



<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) spectrum of **2t**

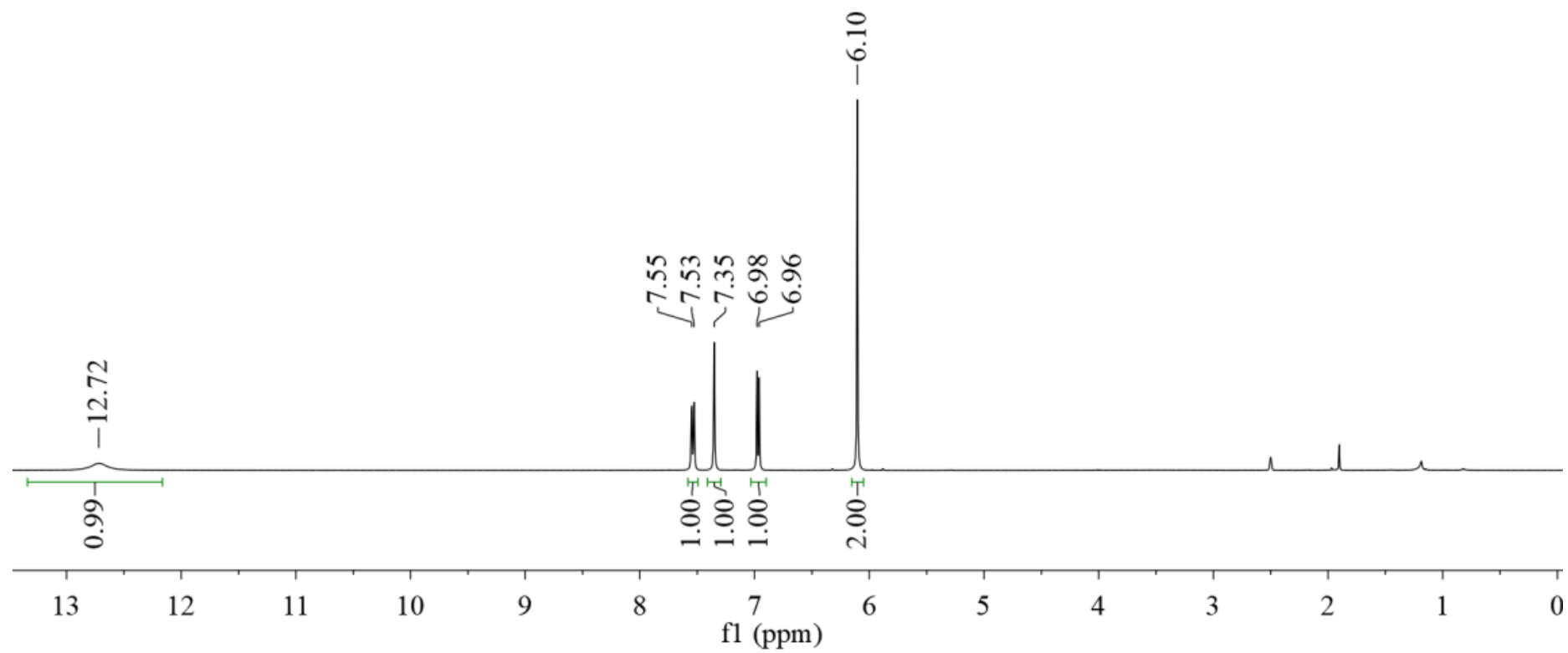
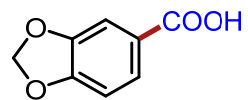


$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2t**

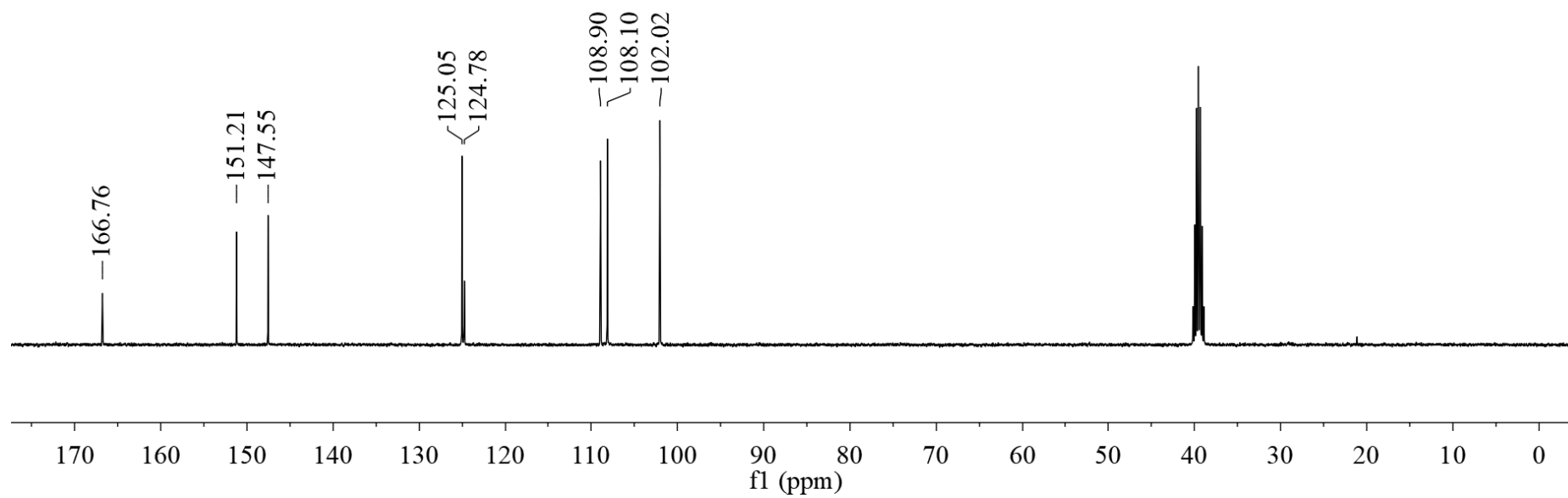
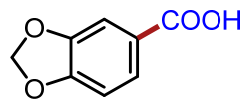




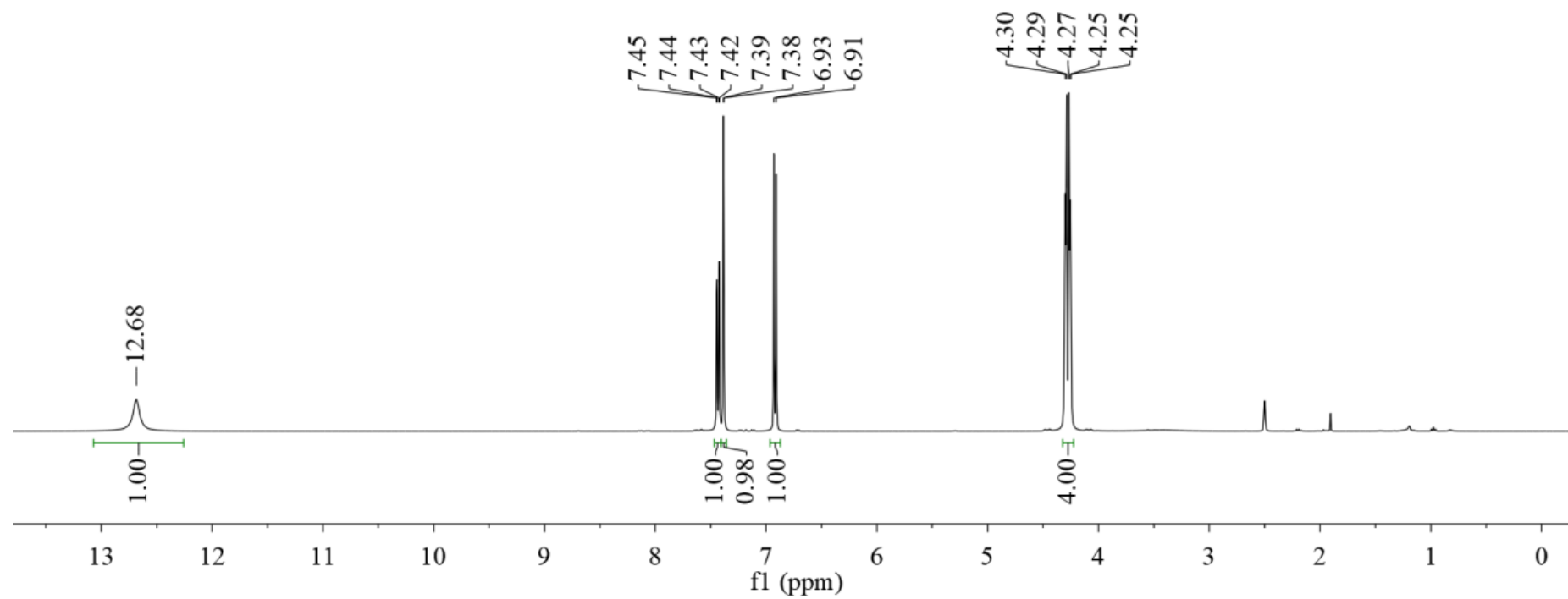
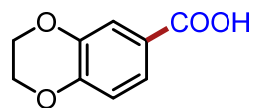
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2u**



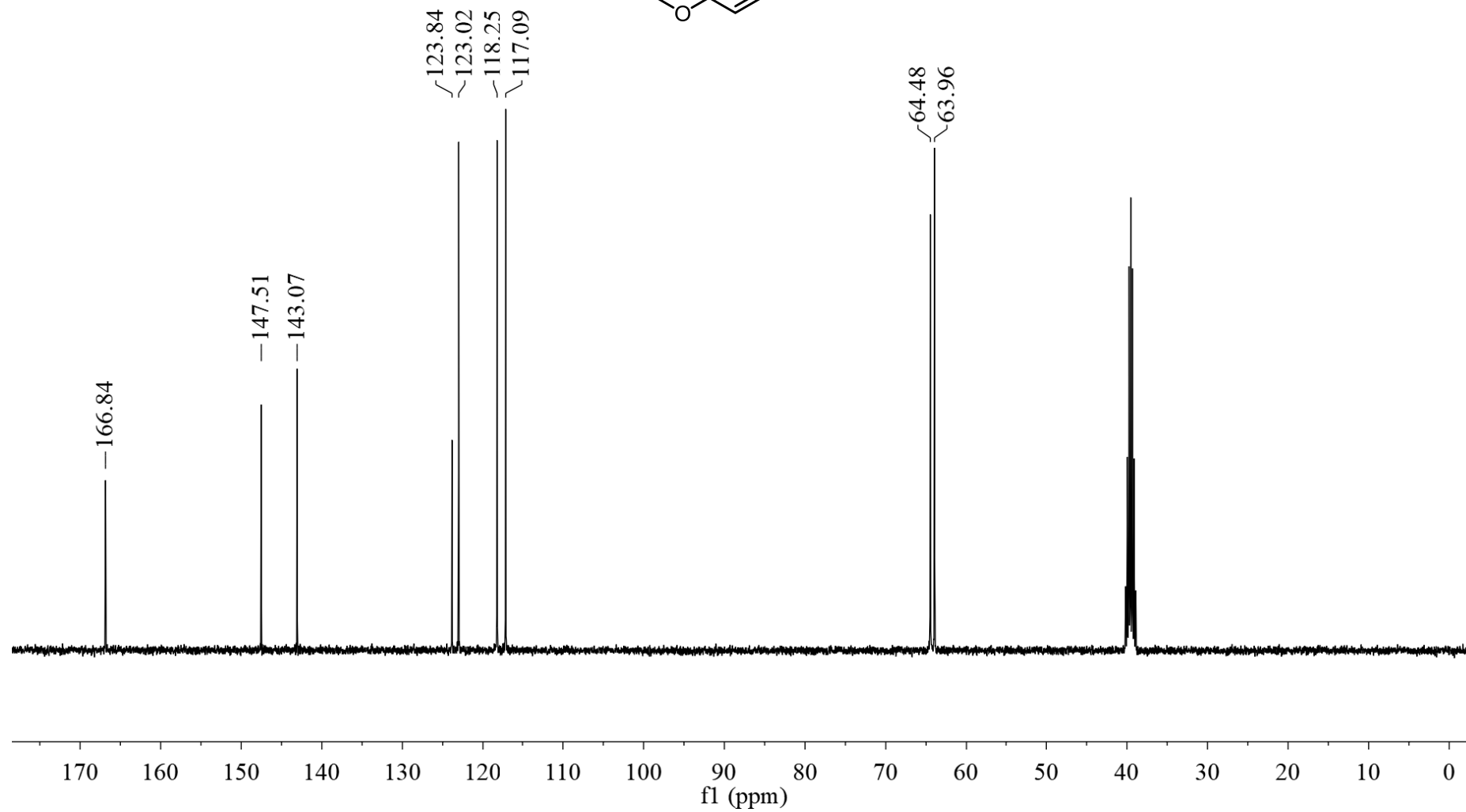
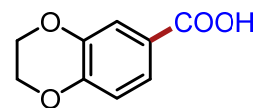
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2u**



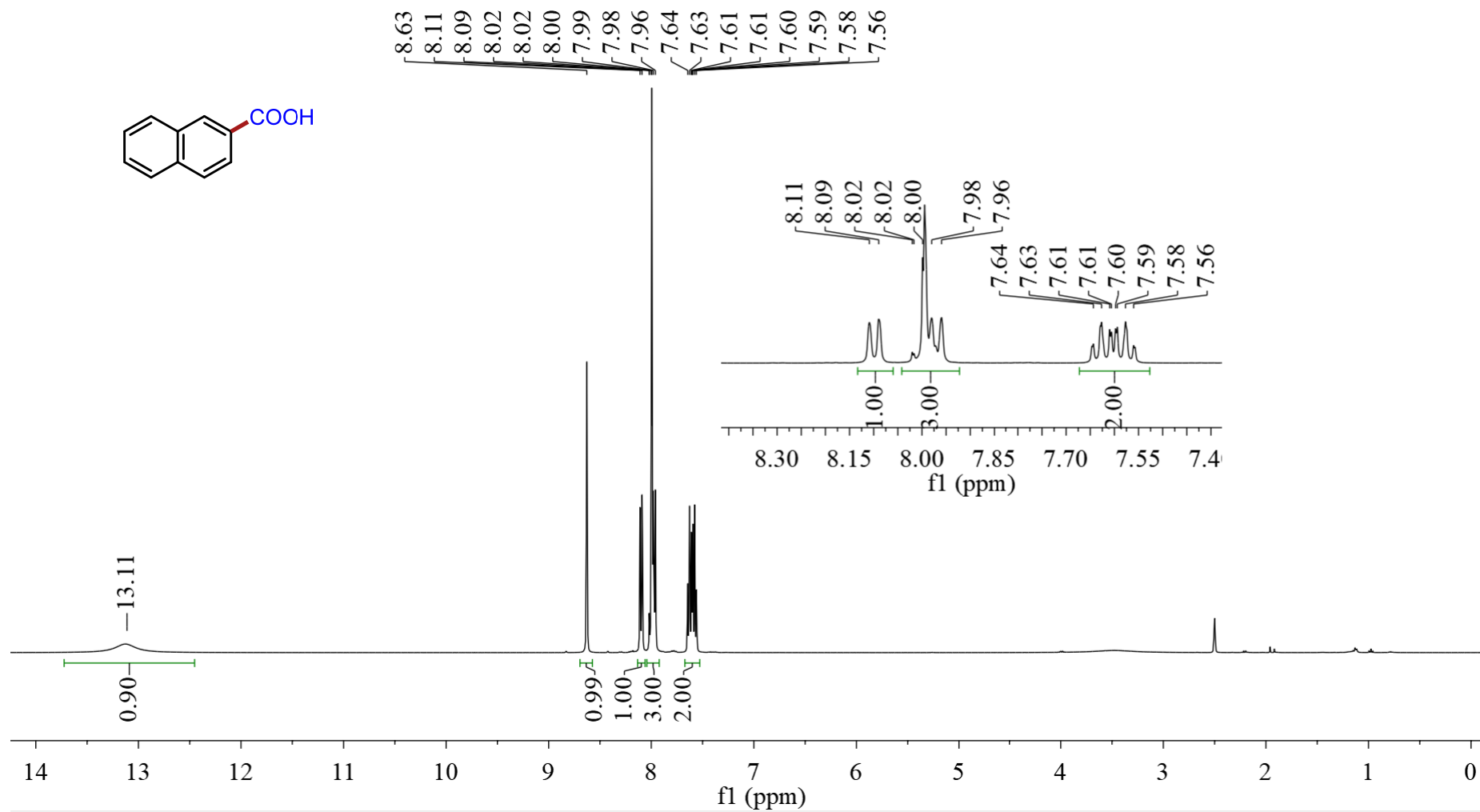
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2v**



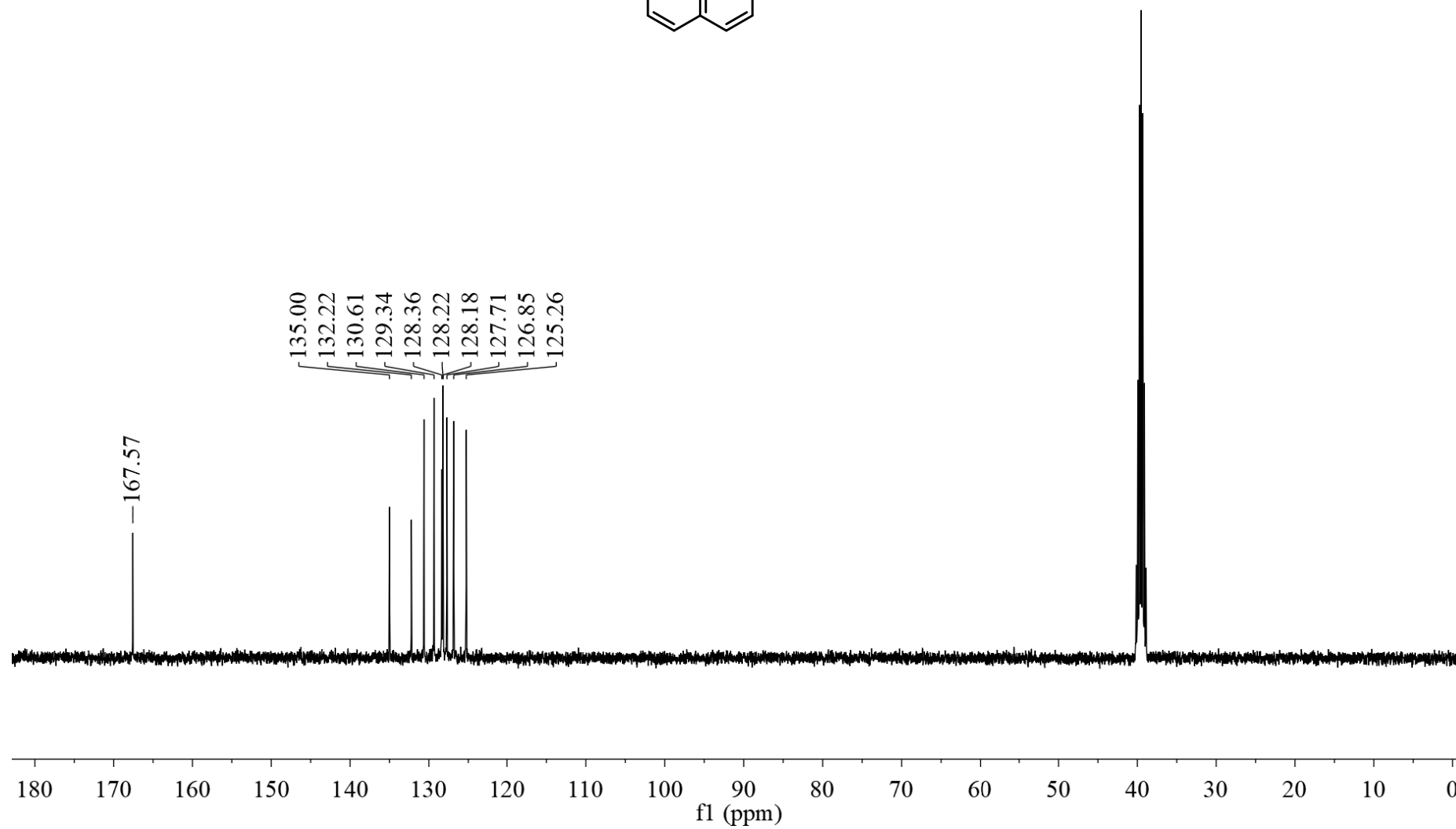
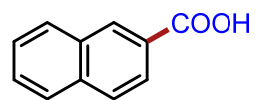
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2v**



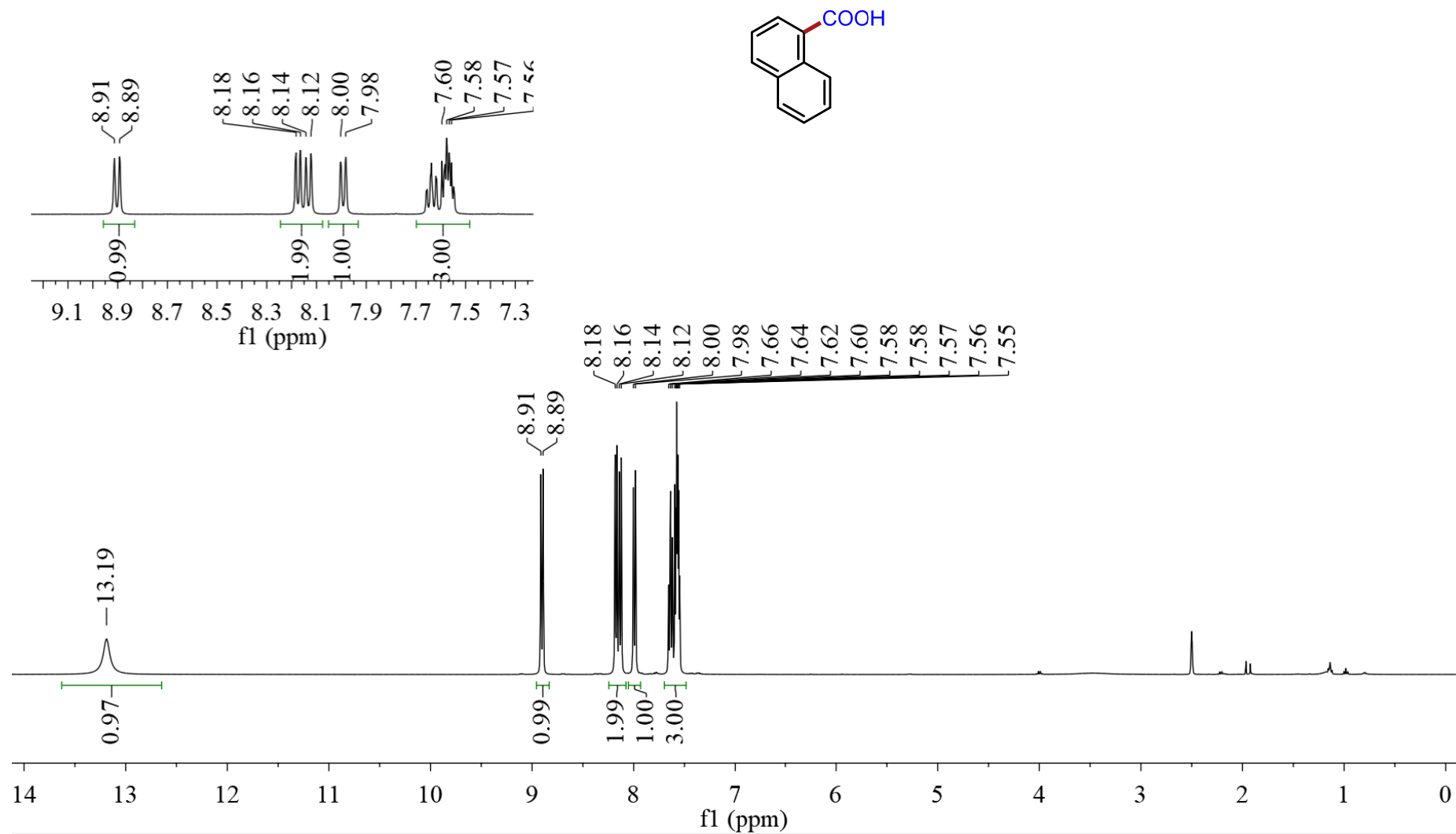
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2w**



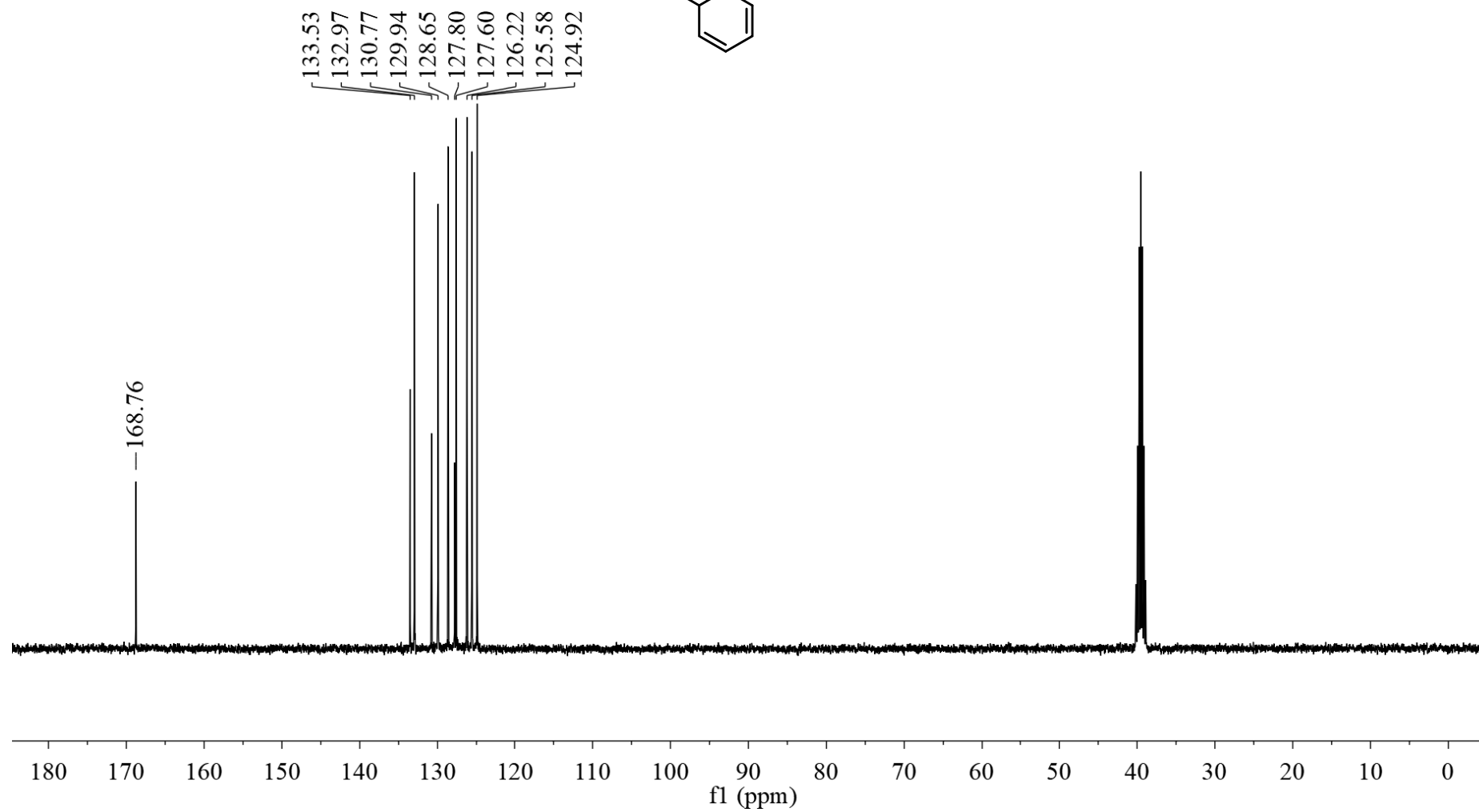
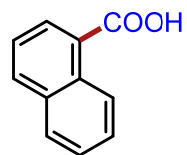
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2w**



$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2x**

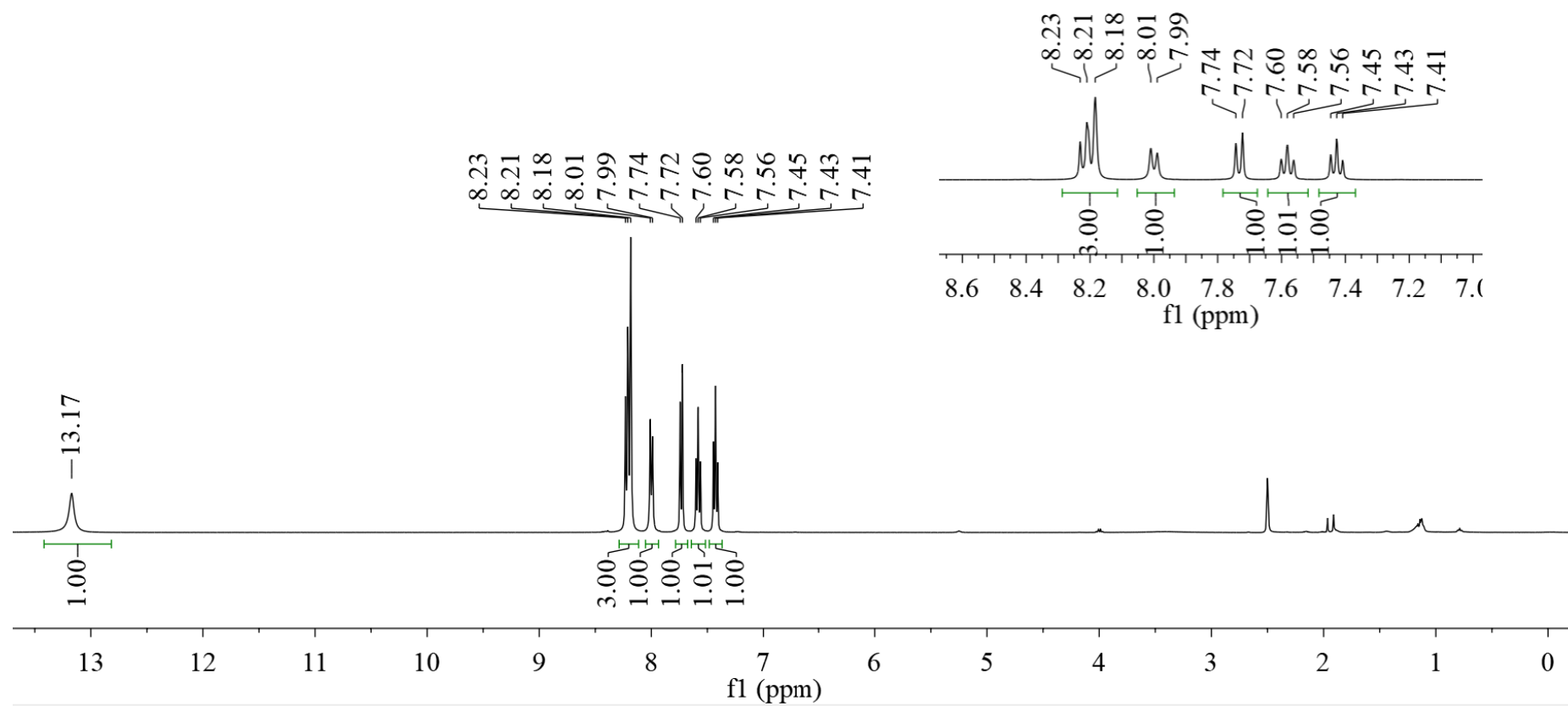
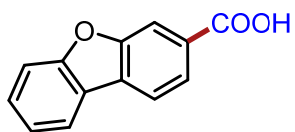


$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2x**

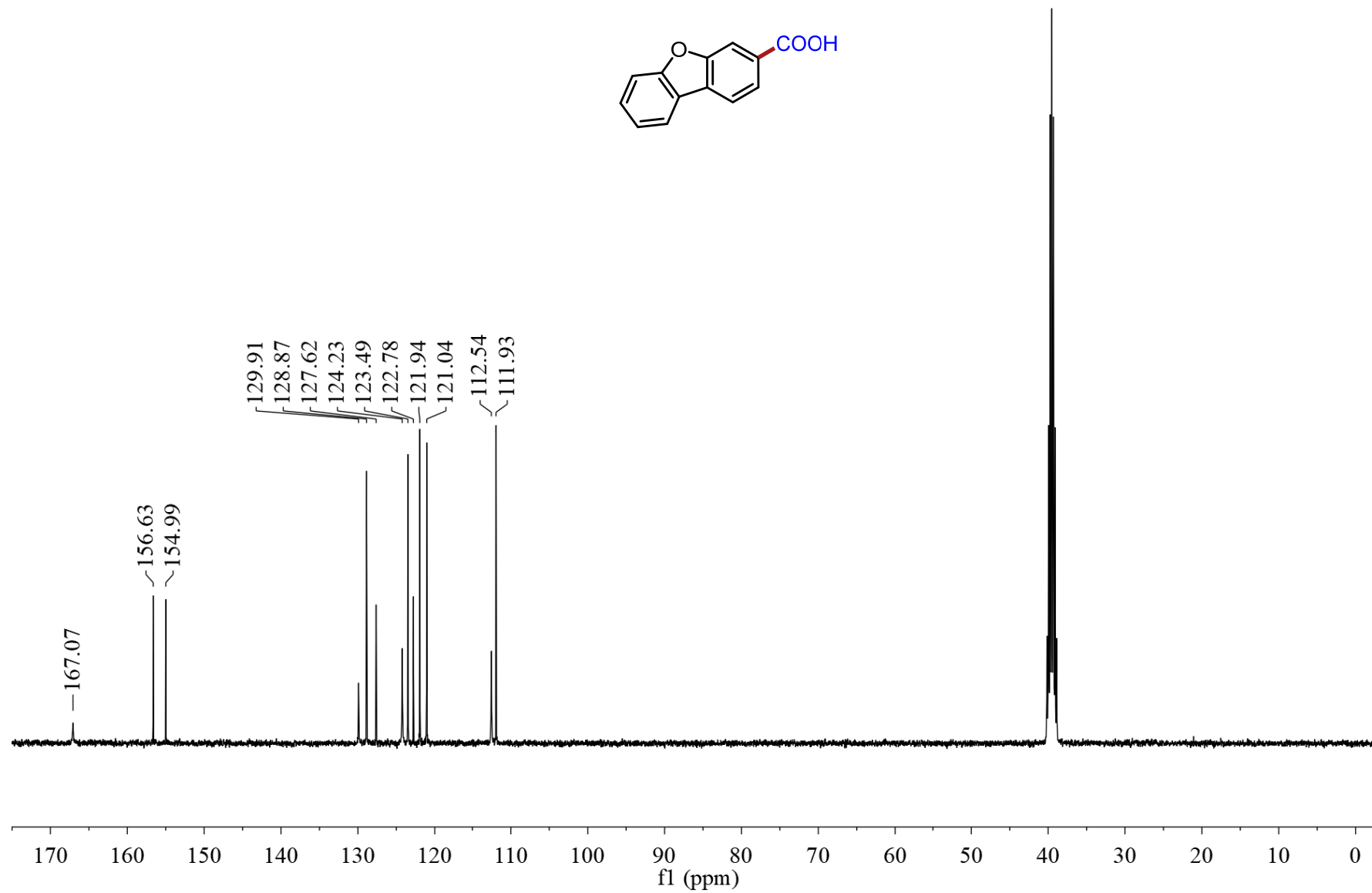
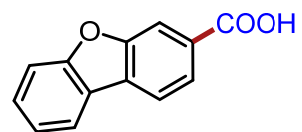




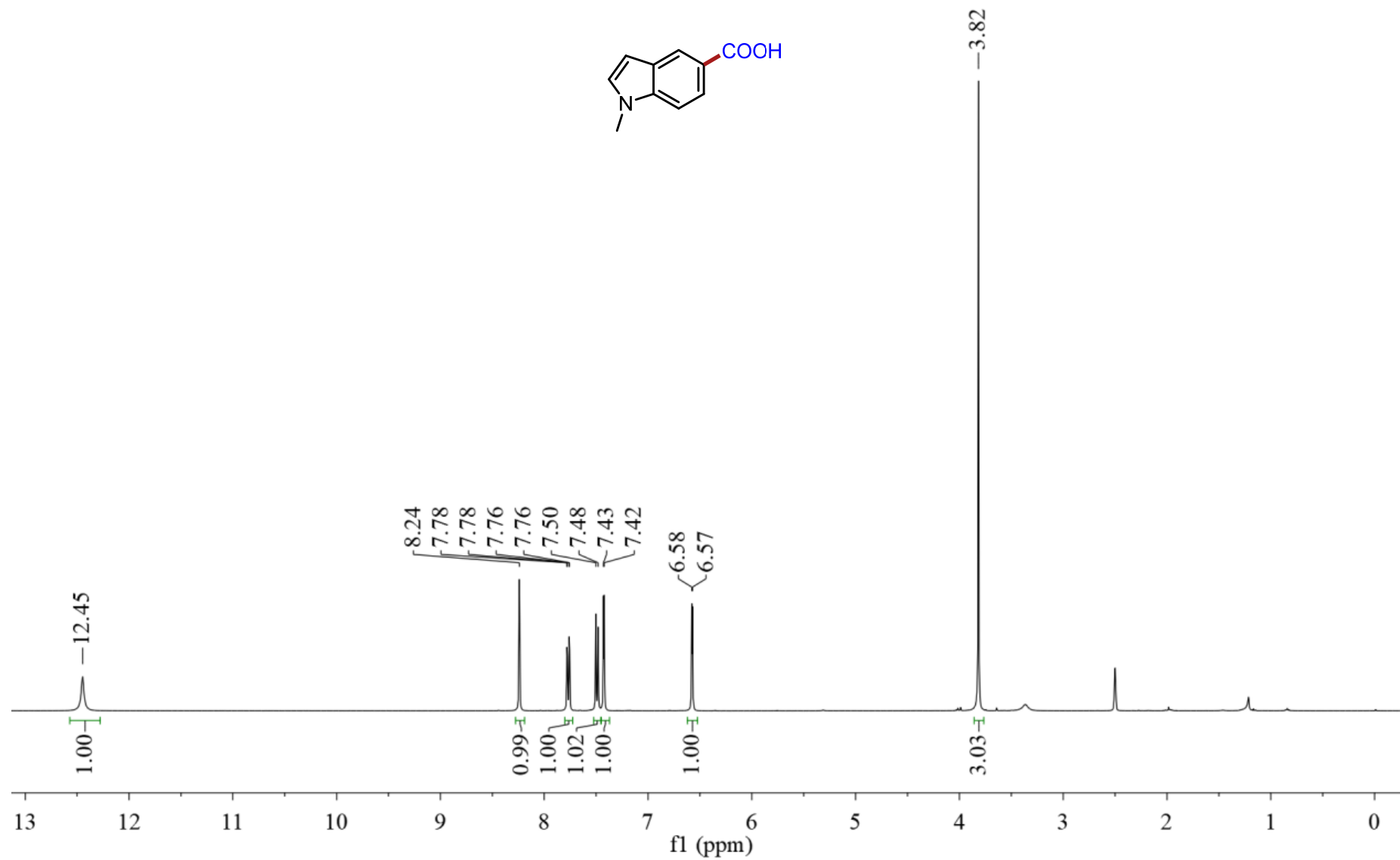
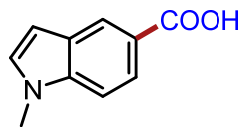
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2y**



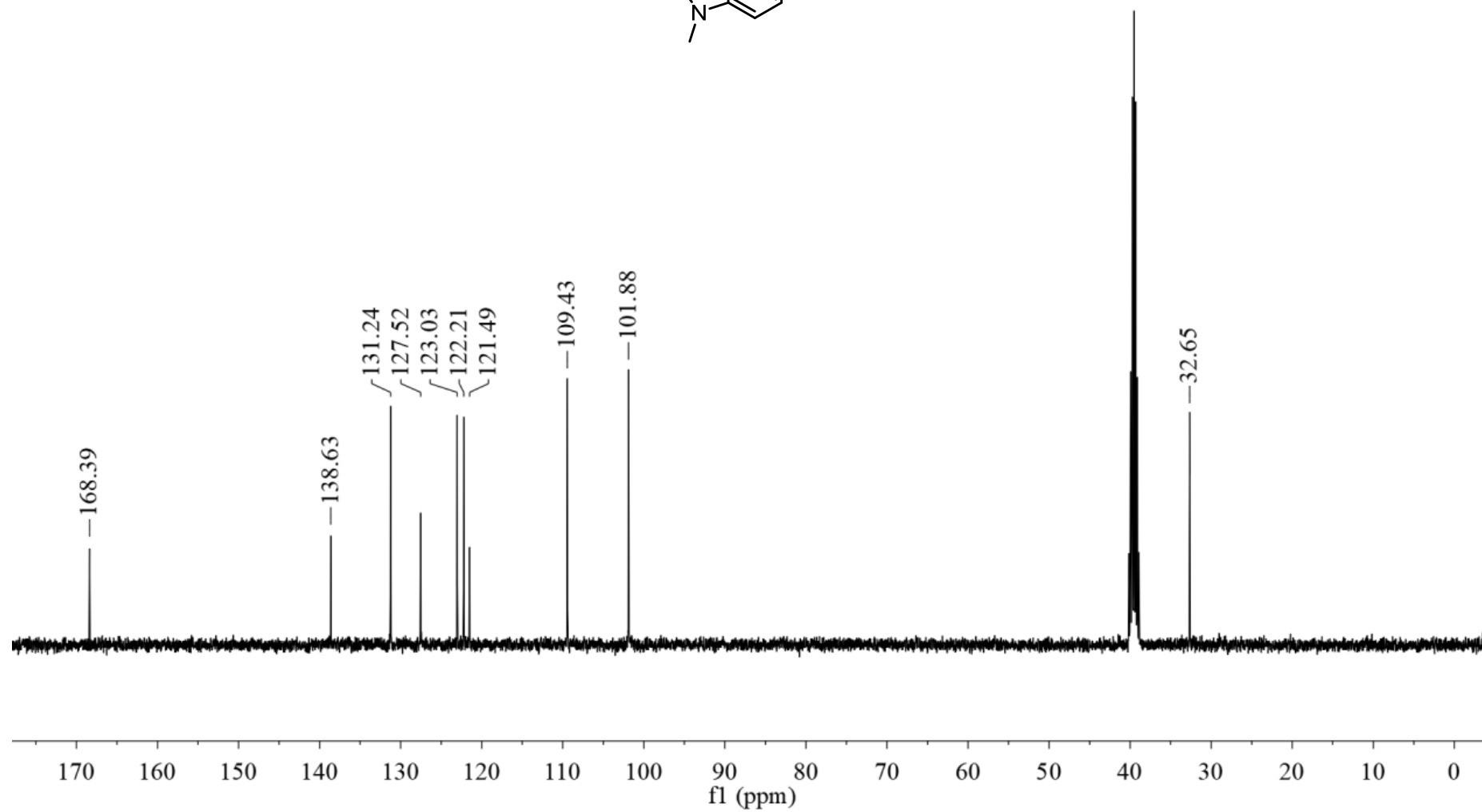
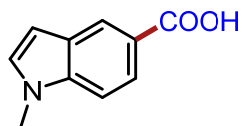
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2y**



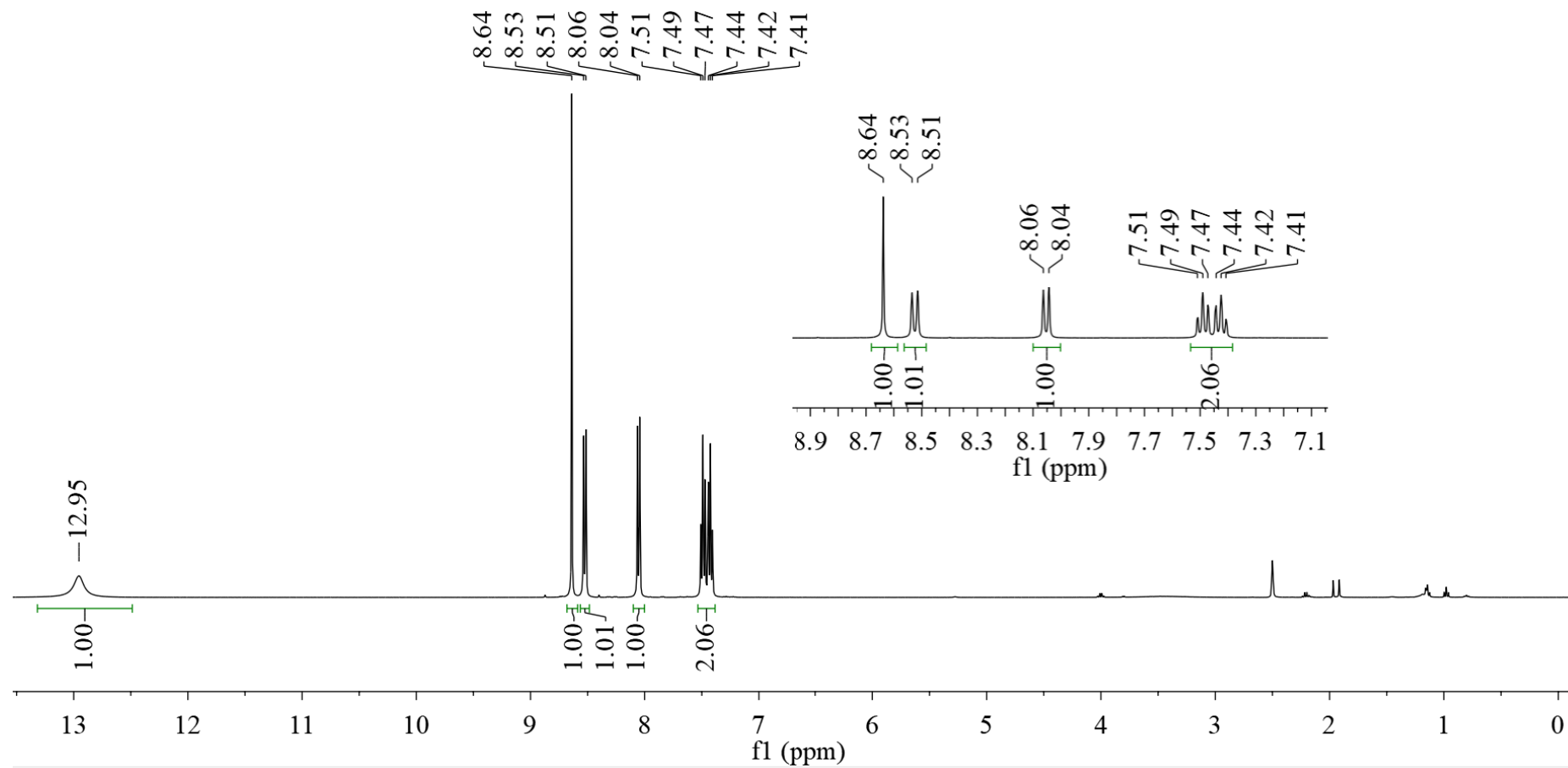
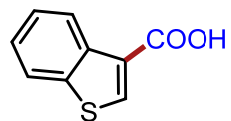
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2z**



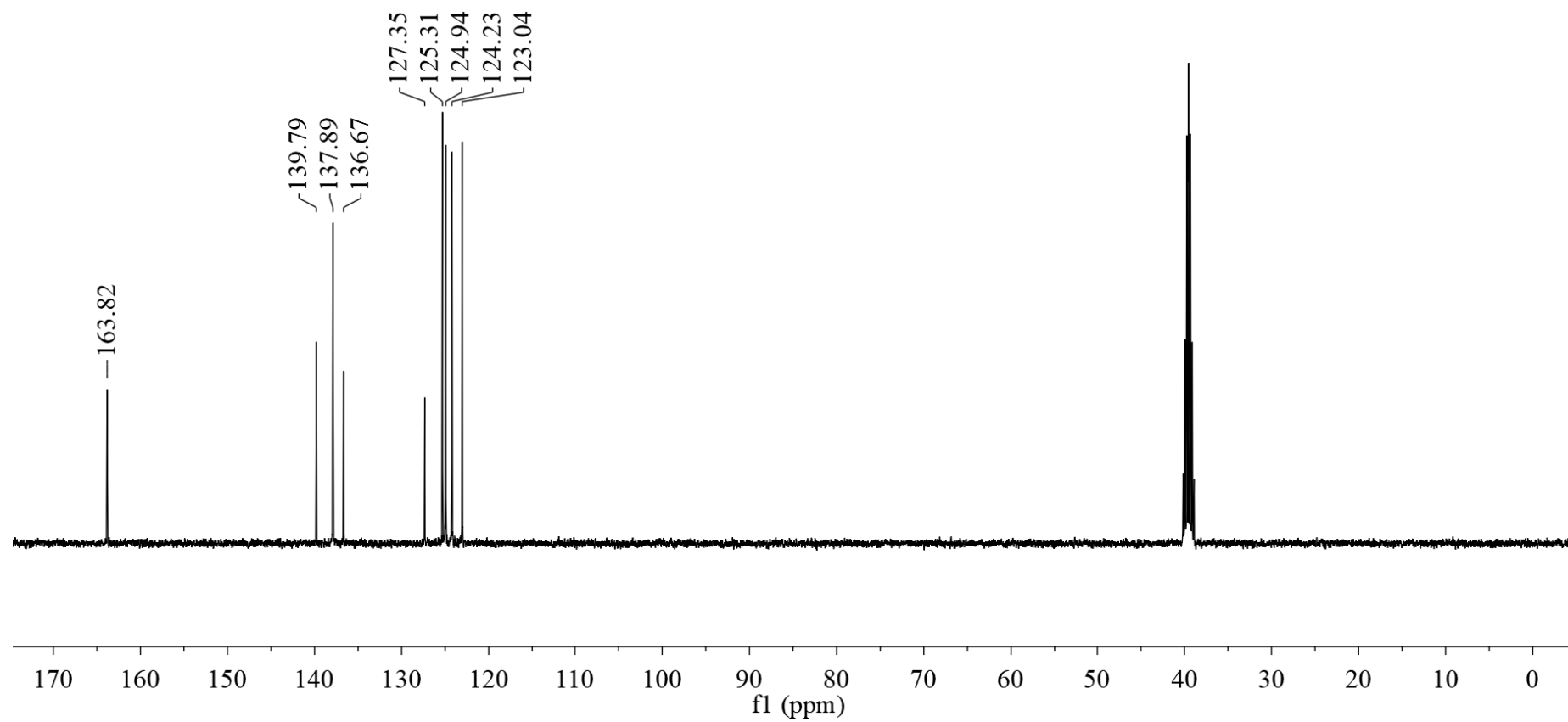
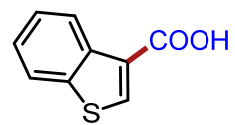
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2z**



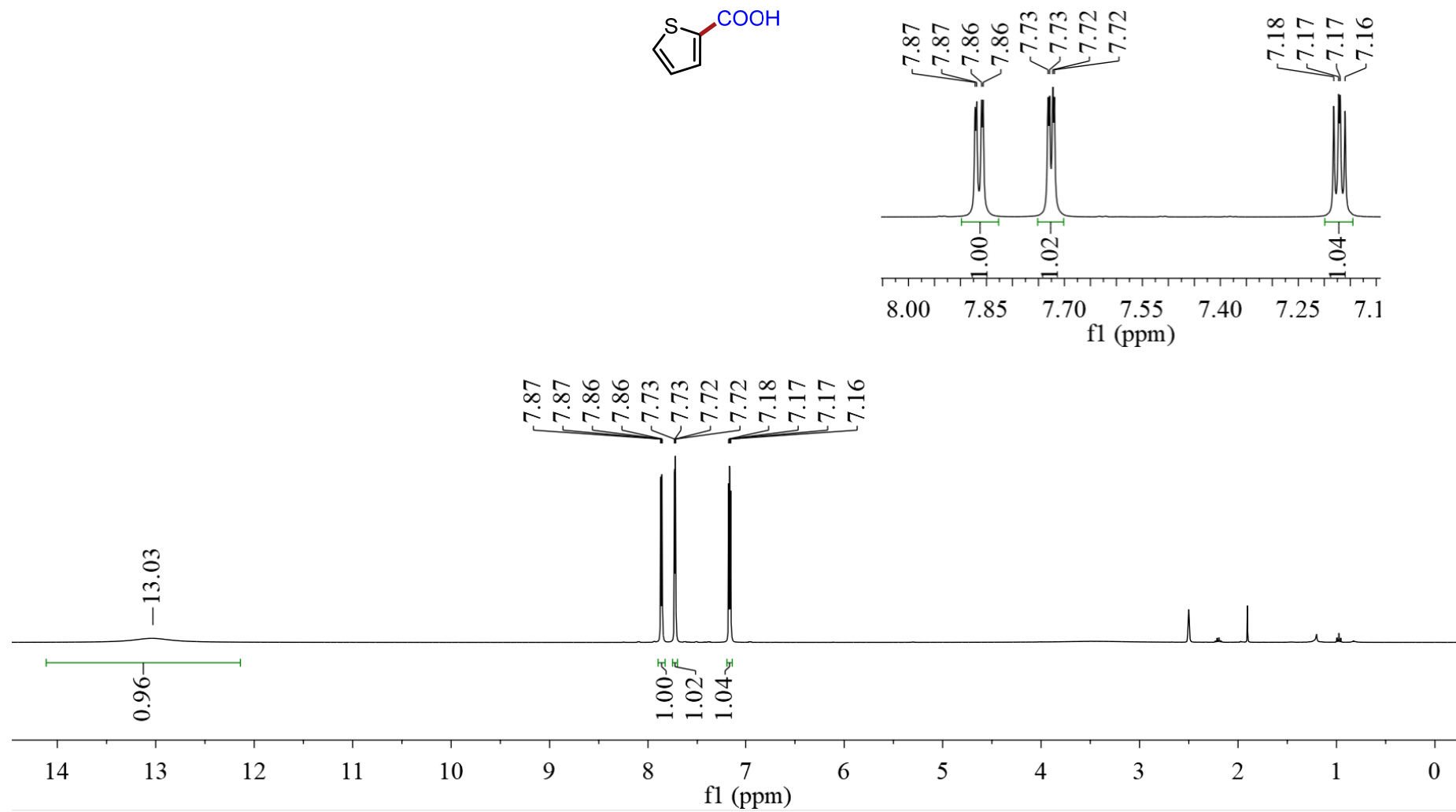
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2aa**



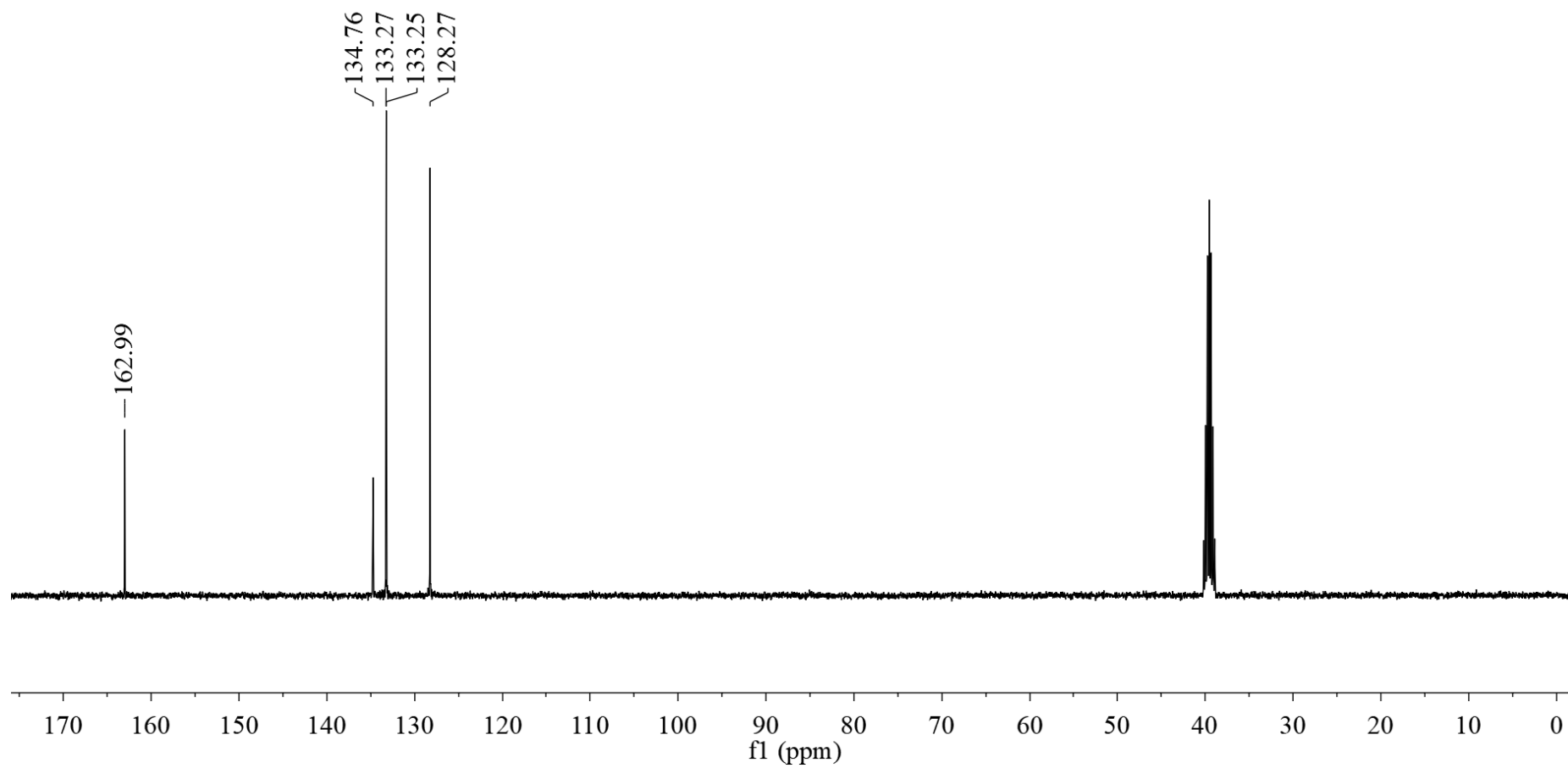
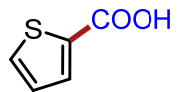
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2aa**



<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) spectrum of **2ab**

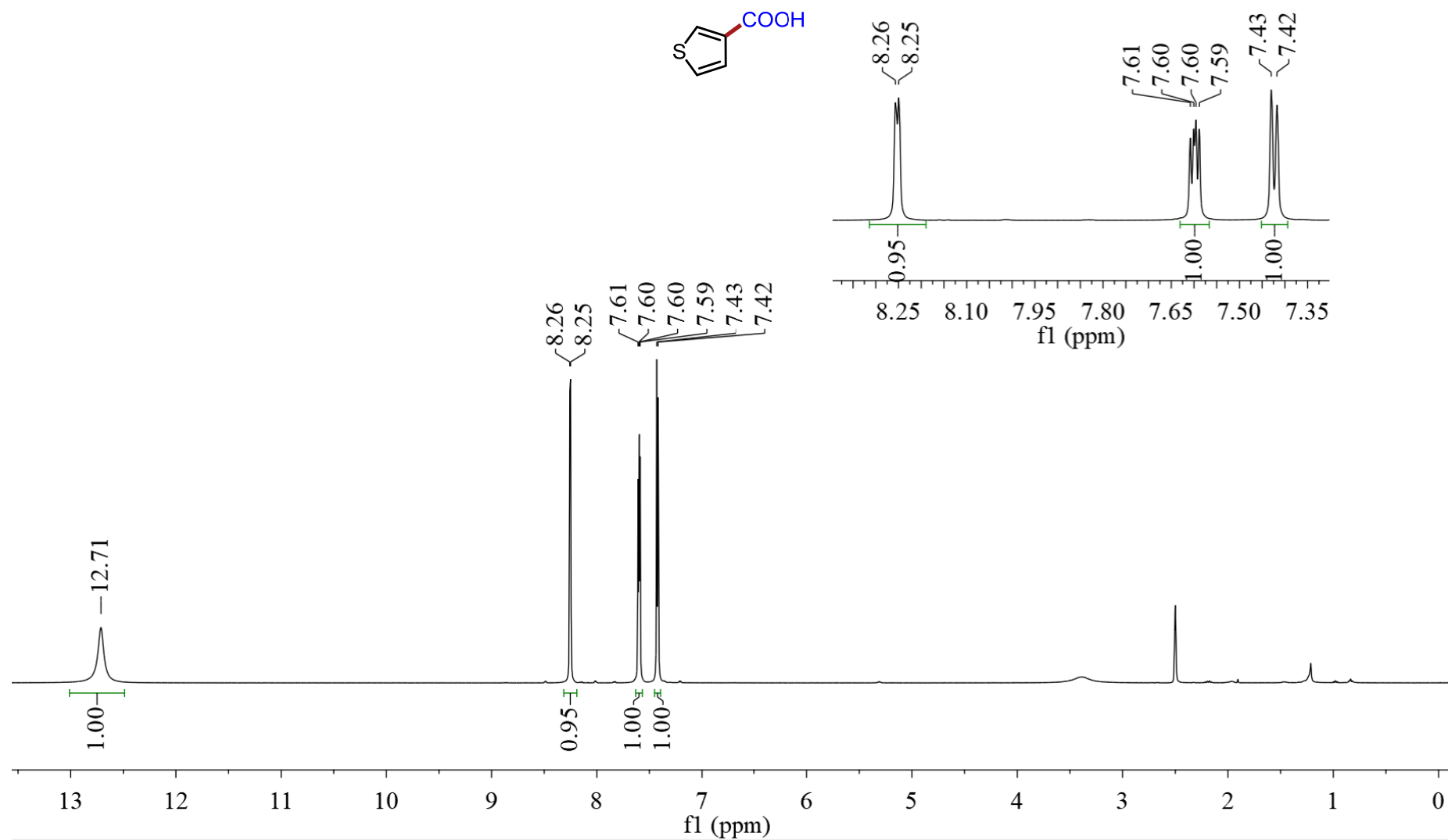


$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ab**

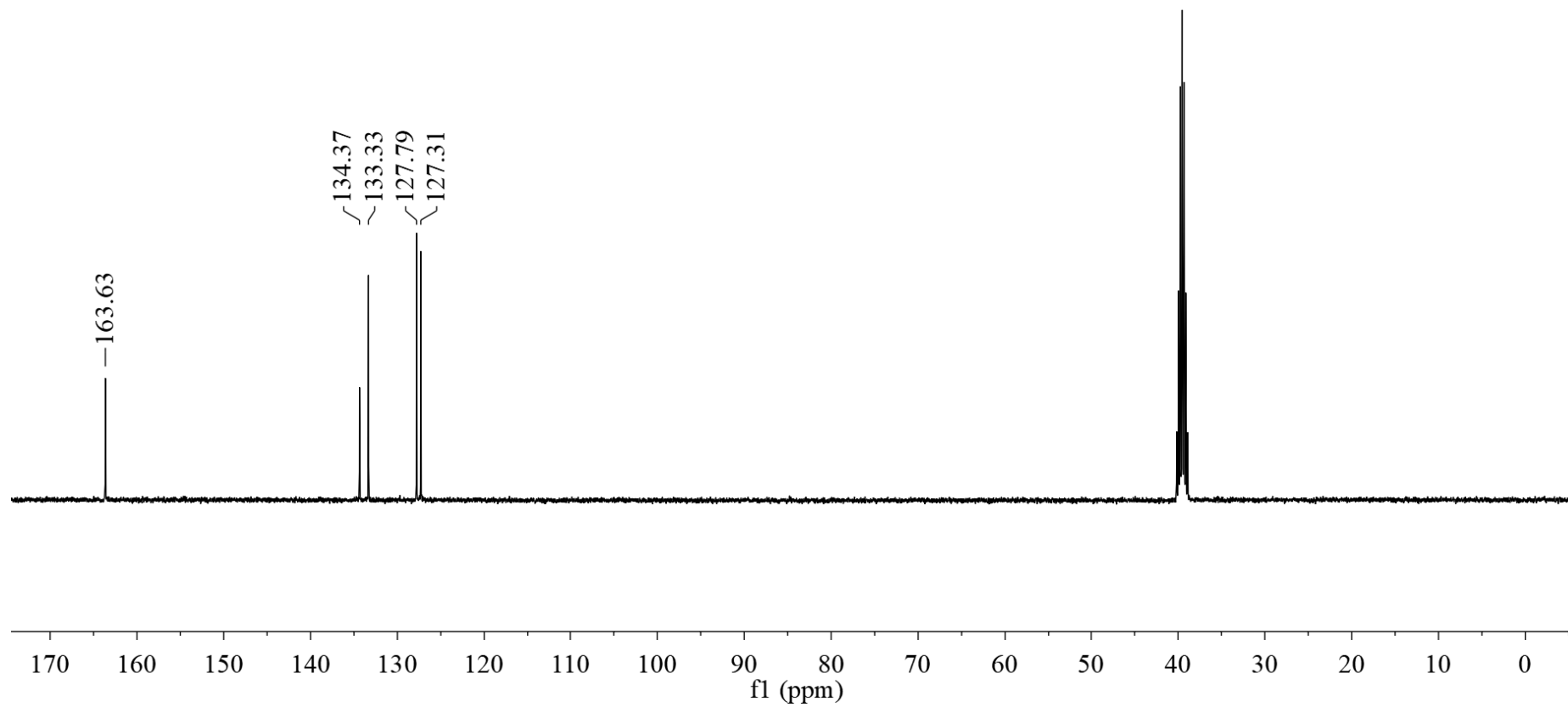
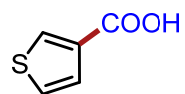




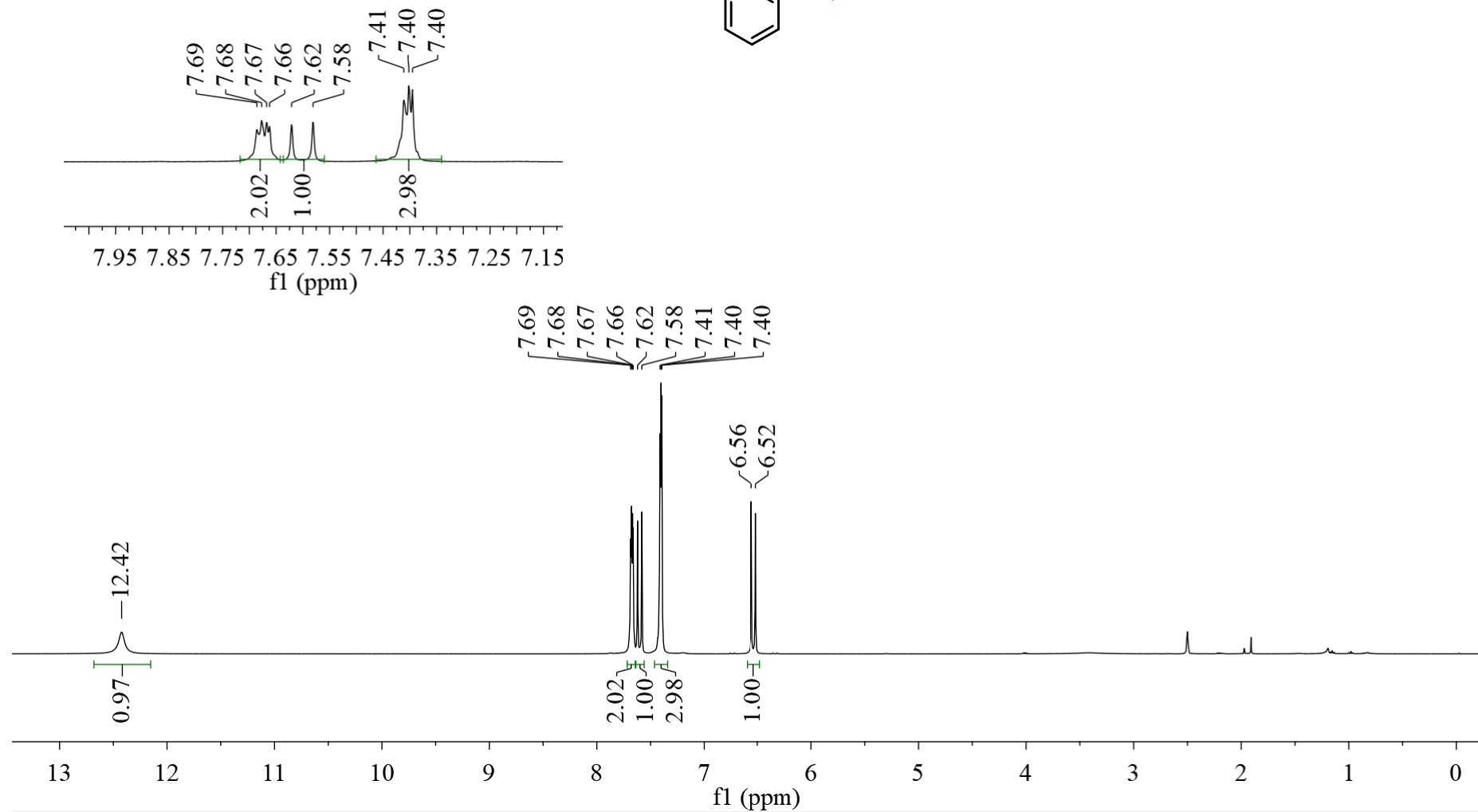
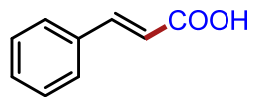
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ac**



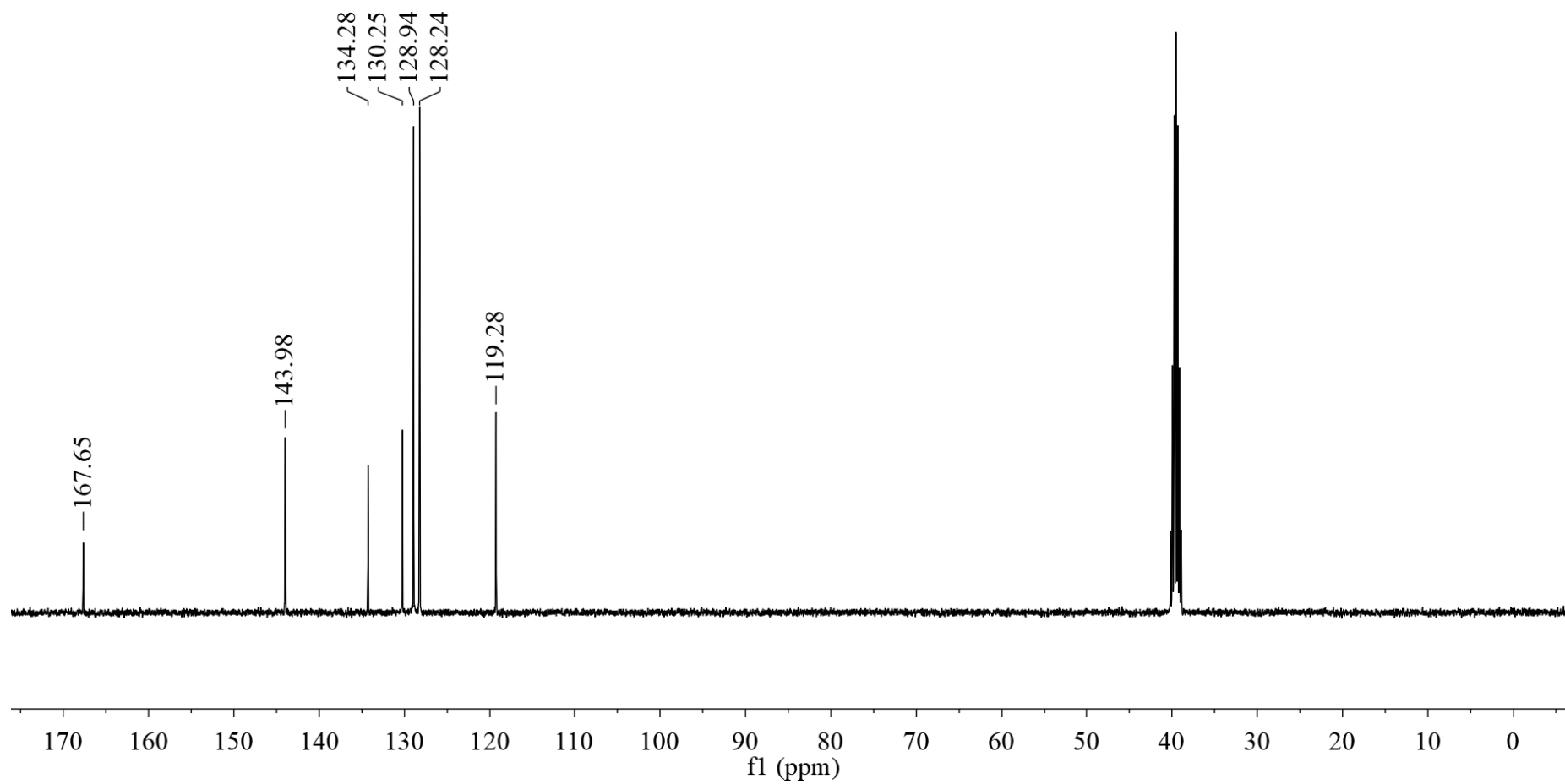
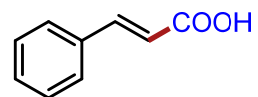
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ac**



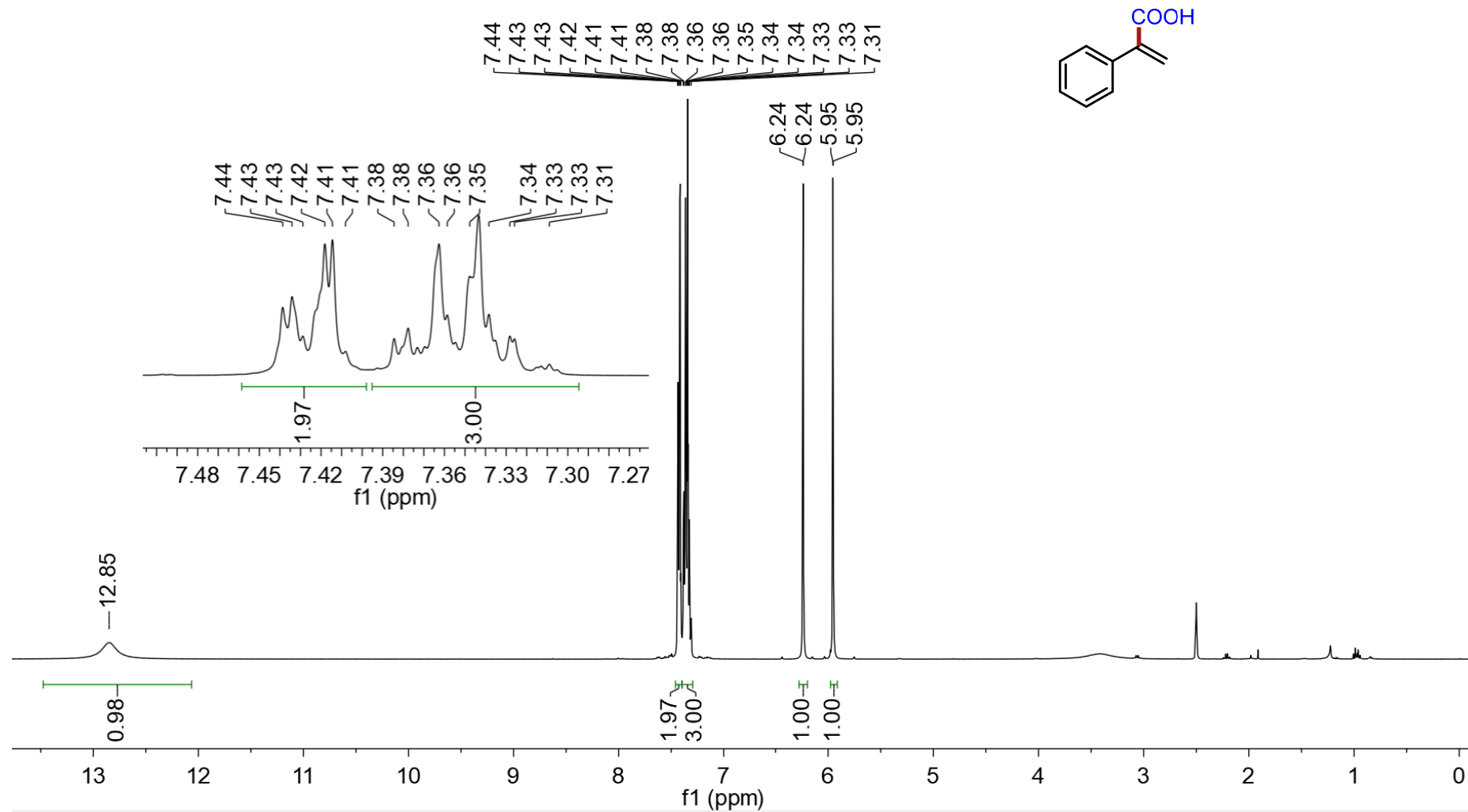
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ad**



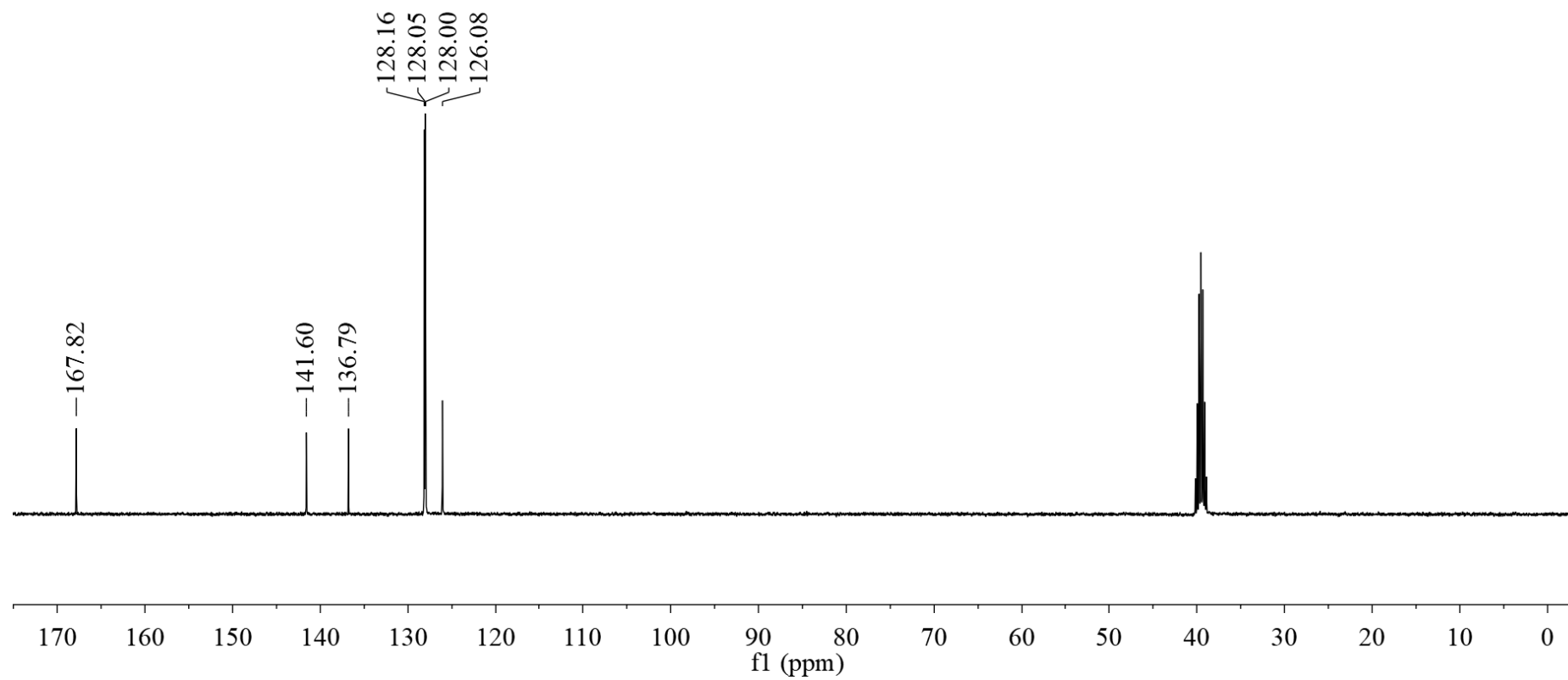
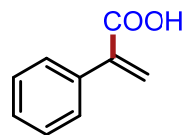
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ad**



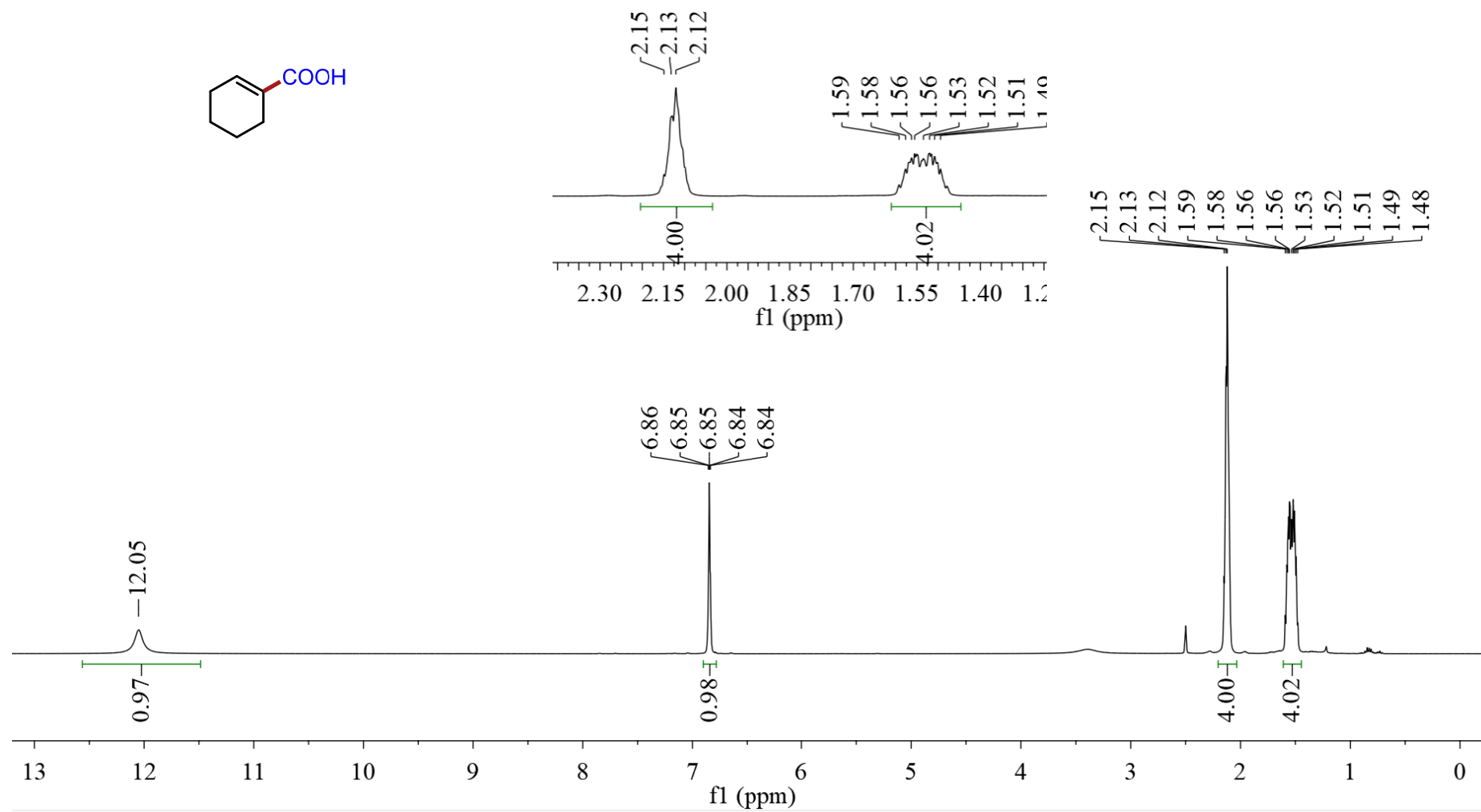
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ae**



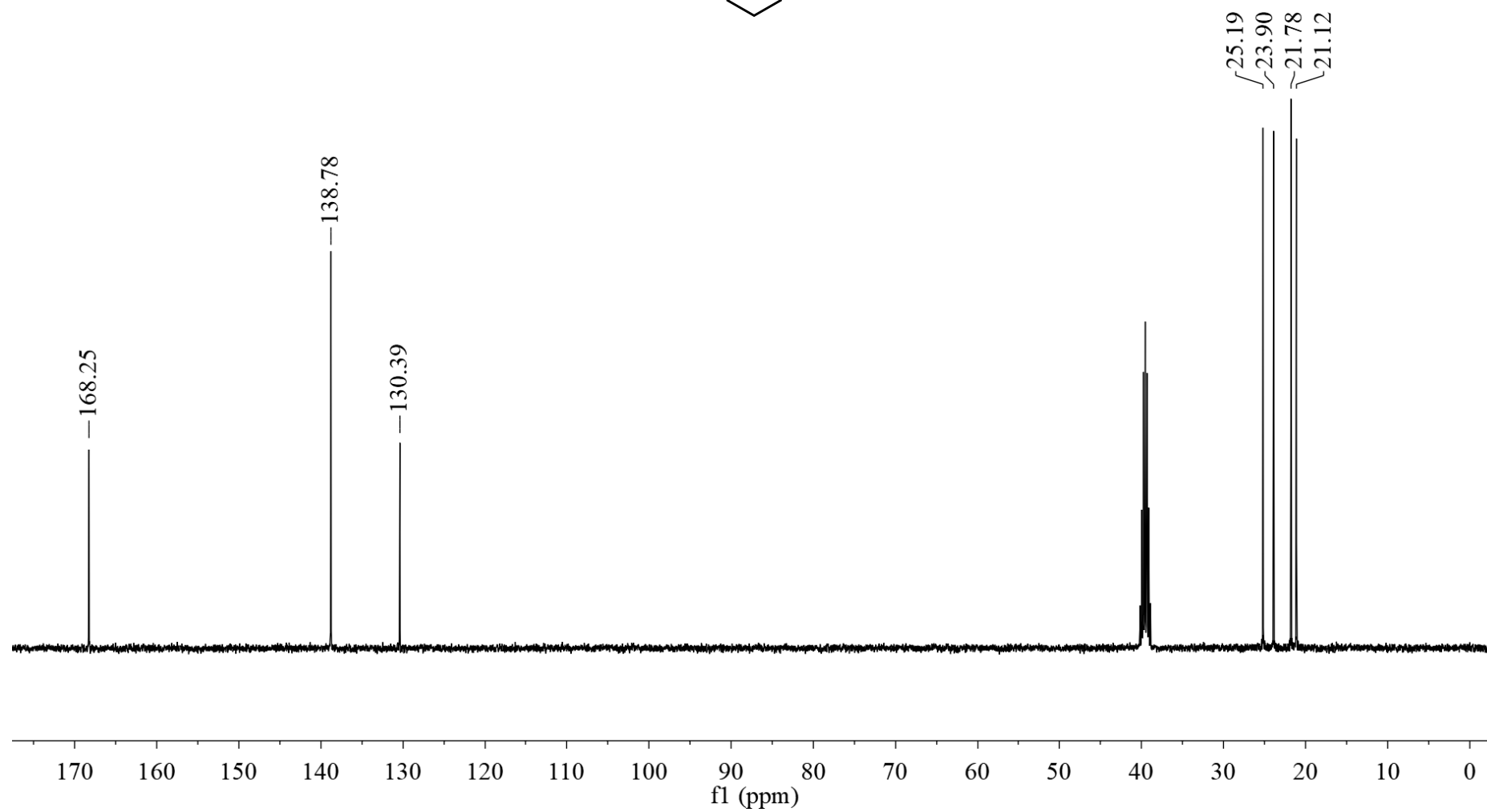
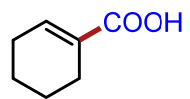
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ae**



$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2af**

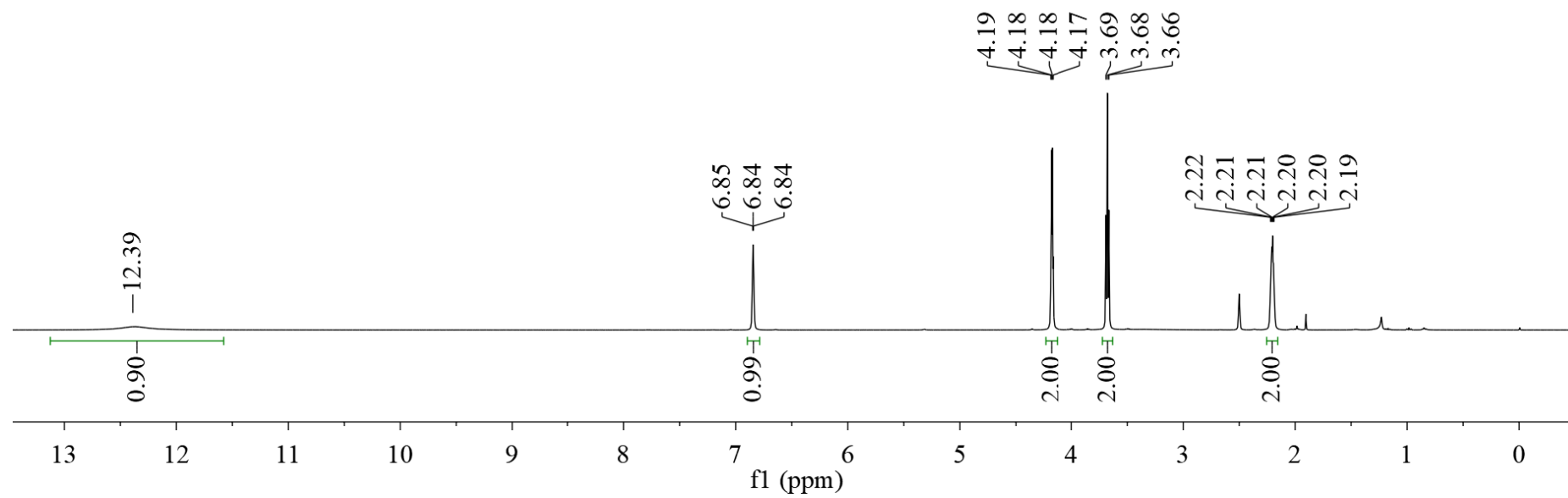
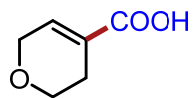


$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2af**

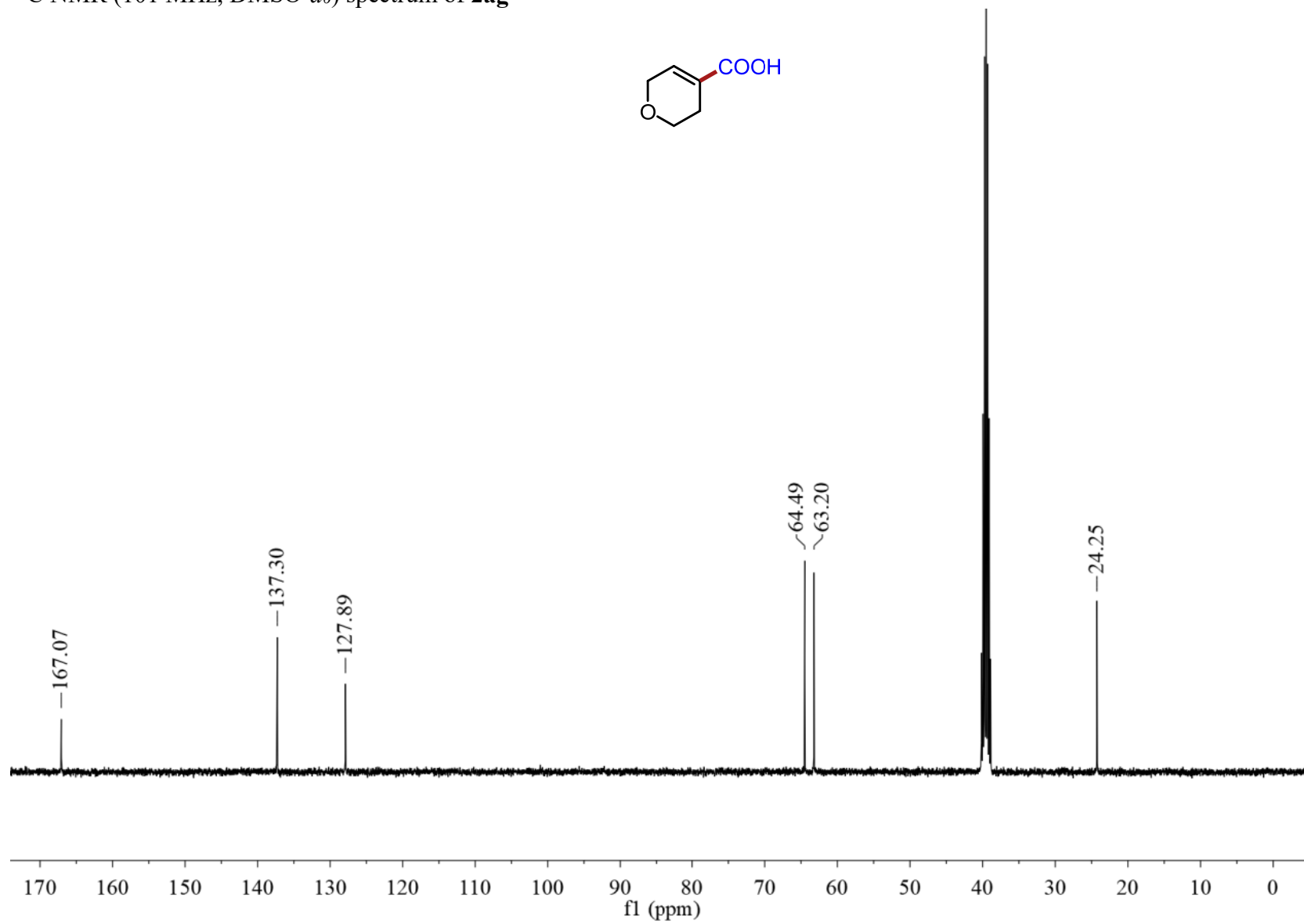
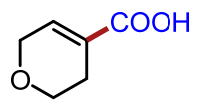




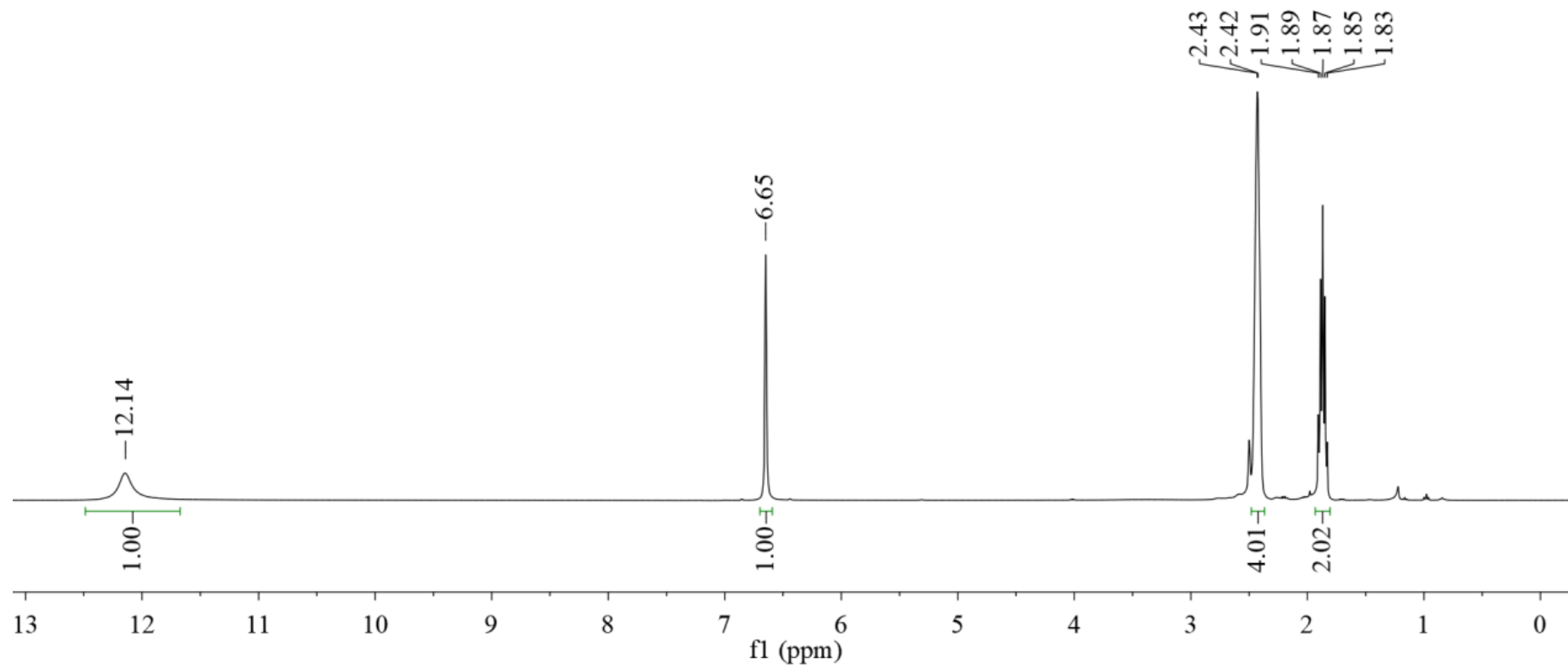
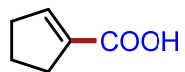
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ag**



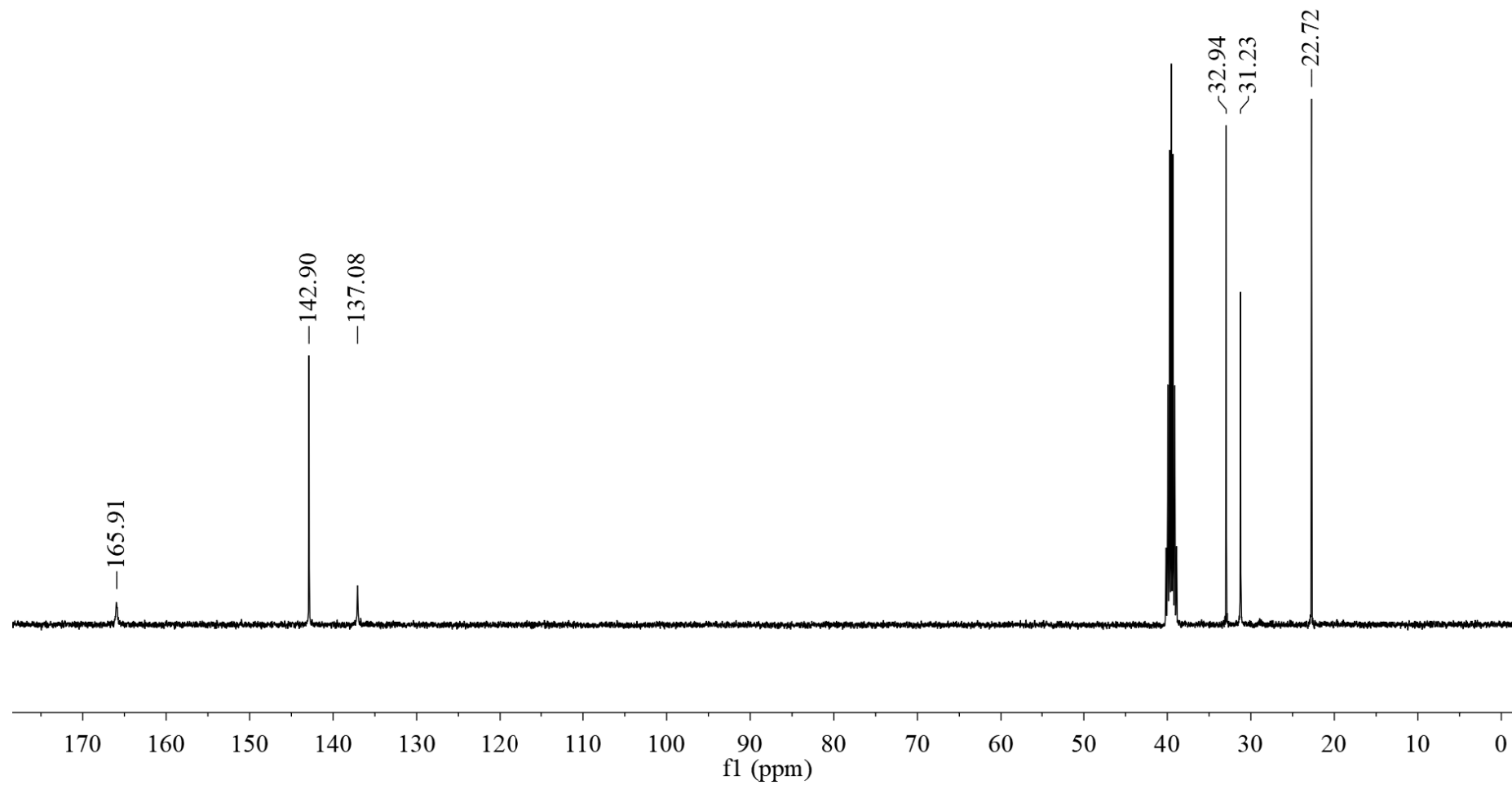
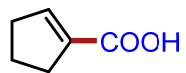
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ag**



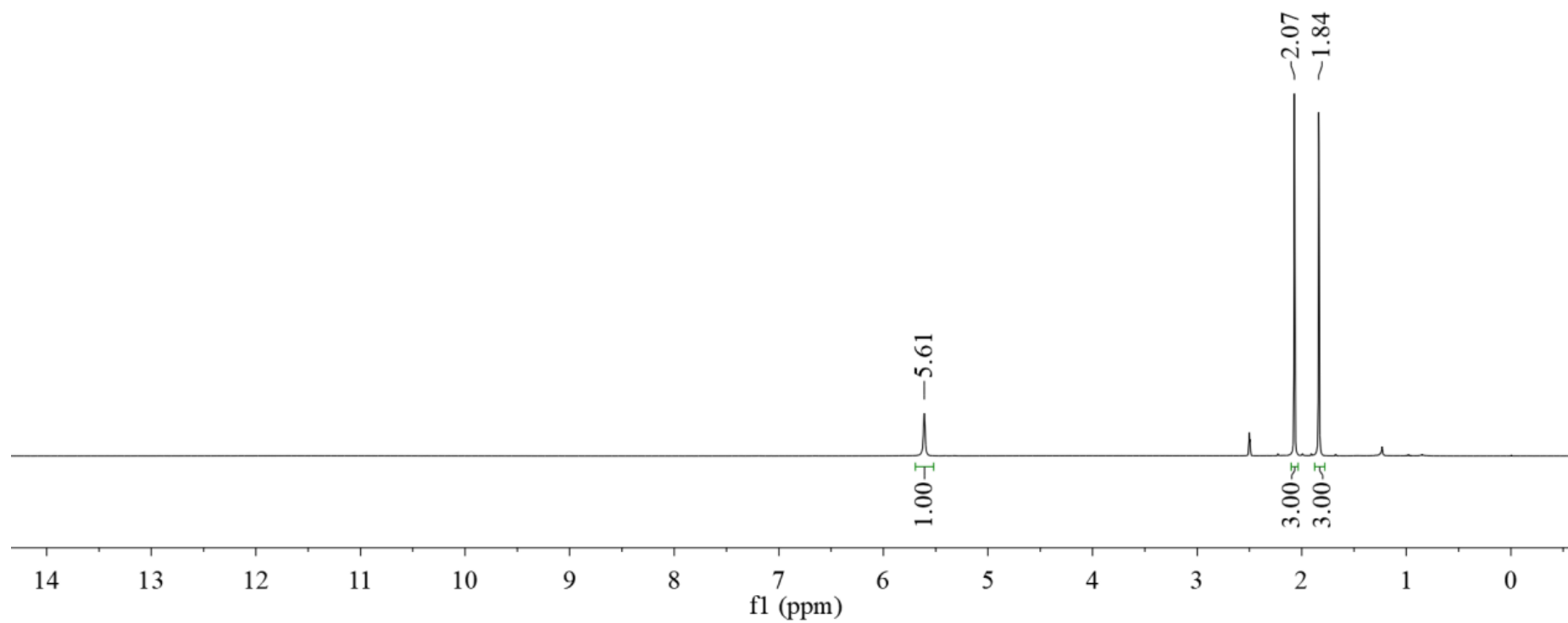
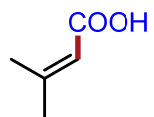
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ah**



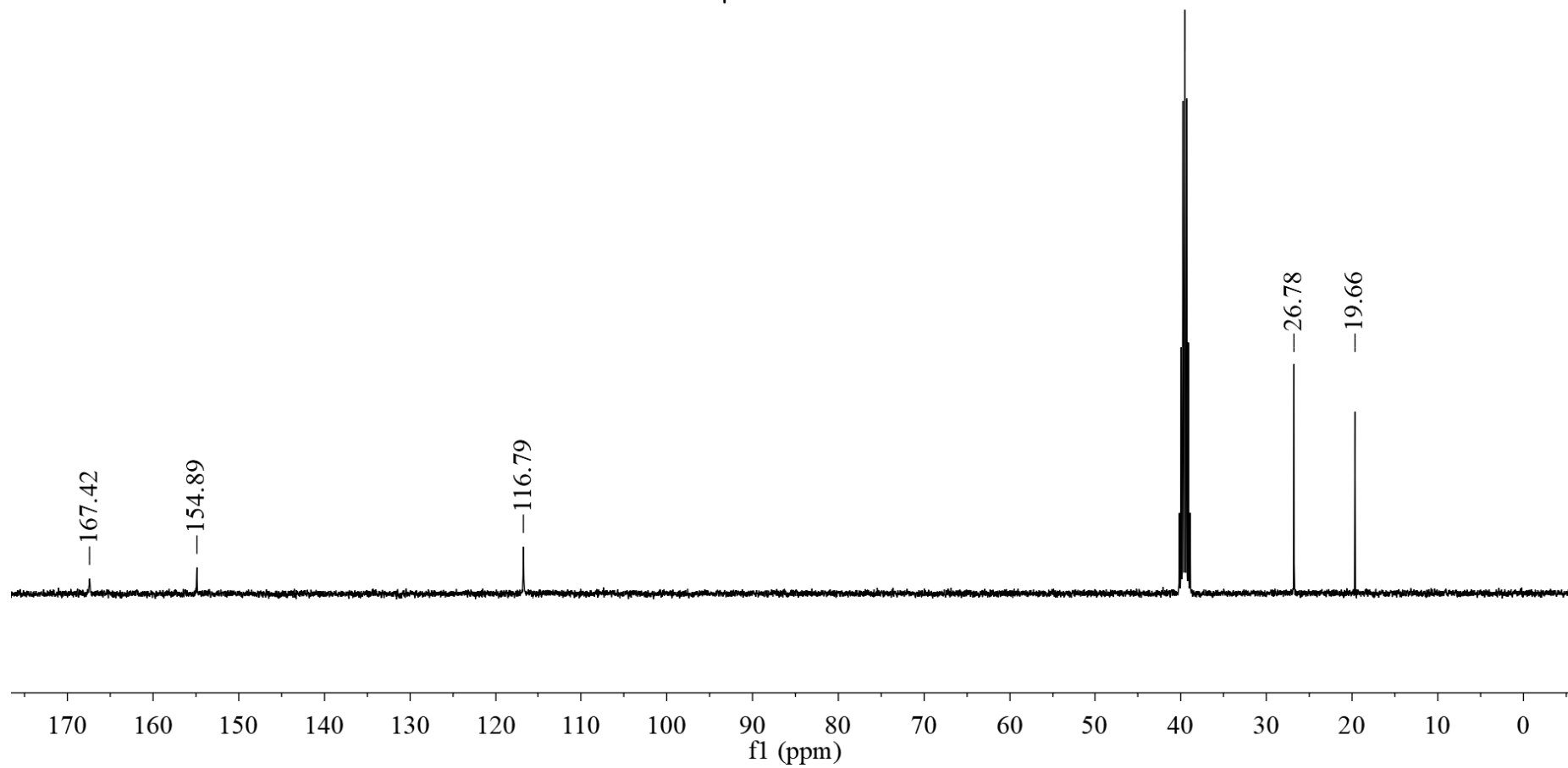
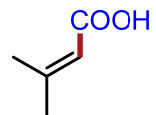
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ah**



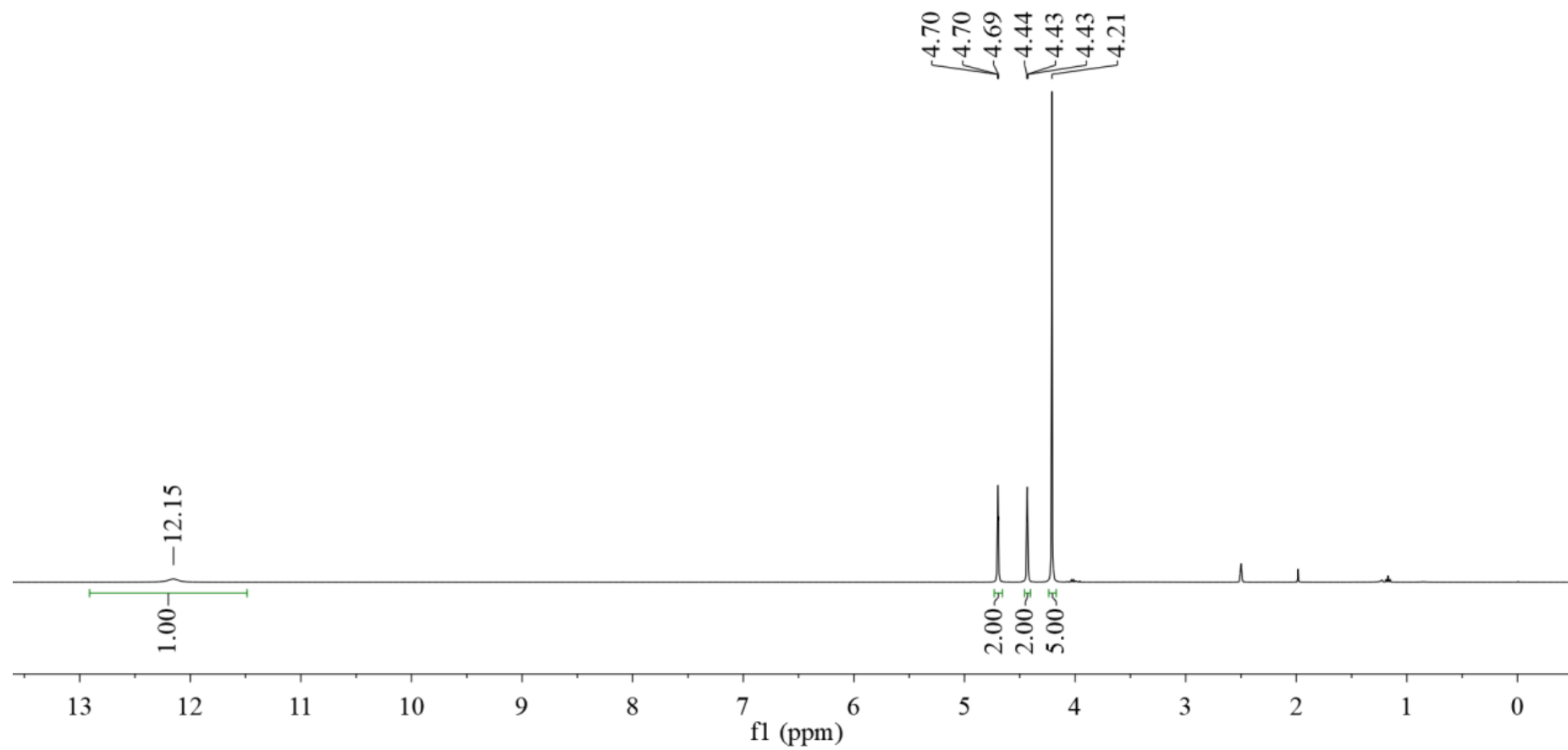
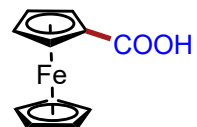
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ai**



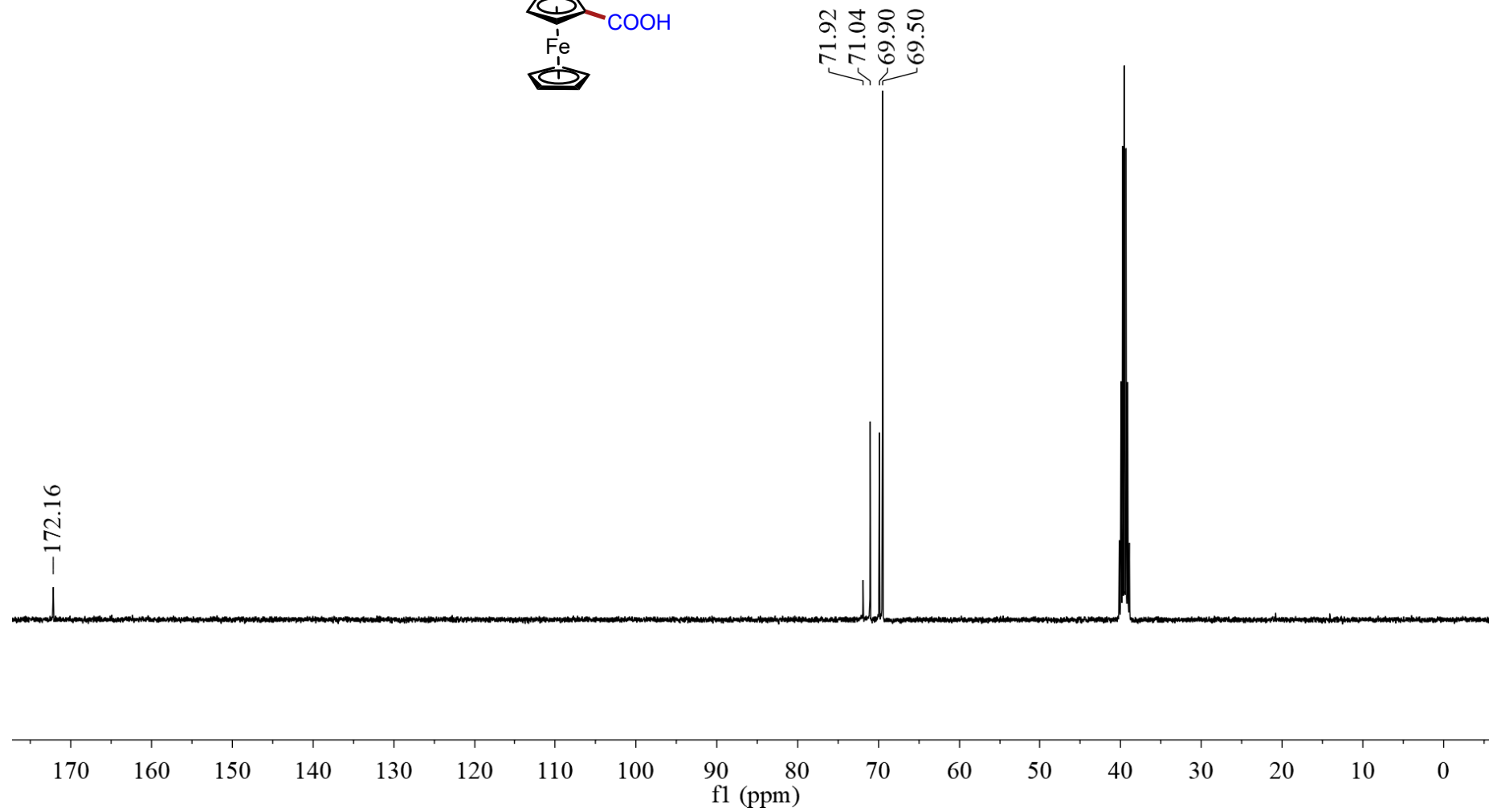
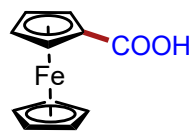
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2ai**



$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2aj**

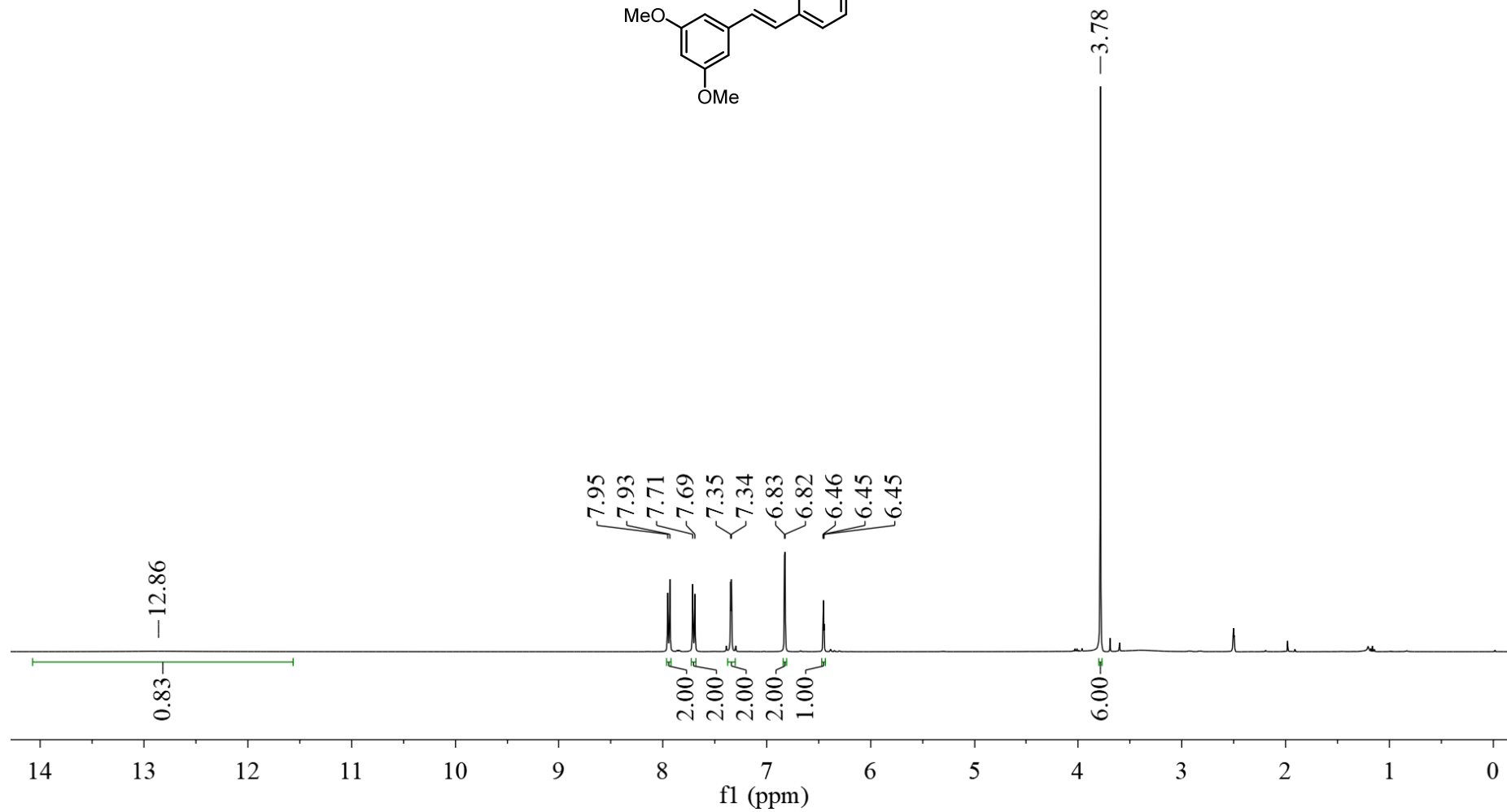
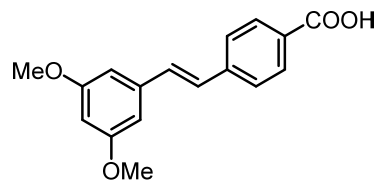


$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **2aj**

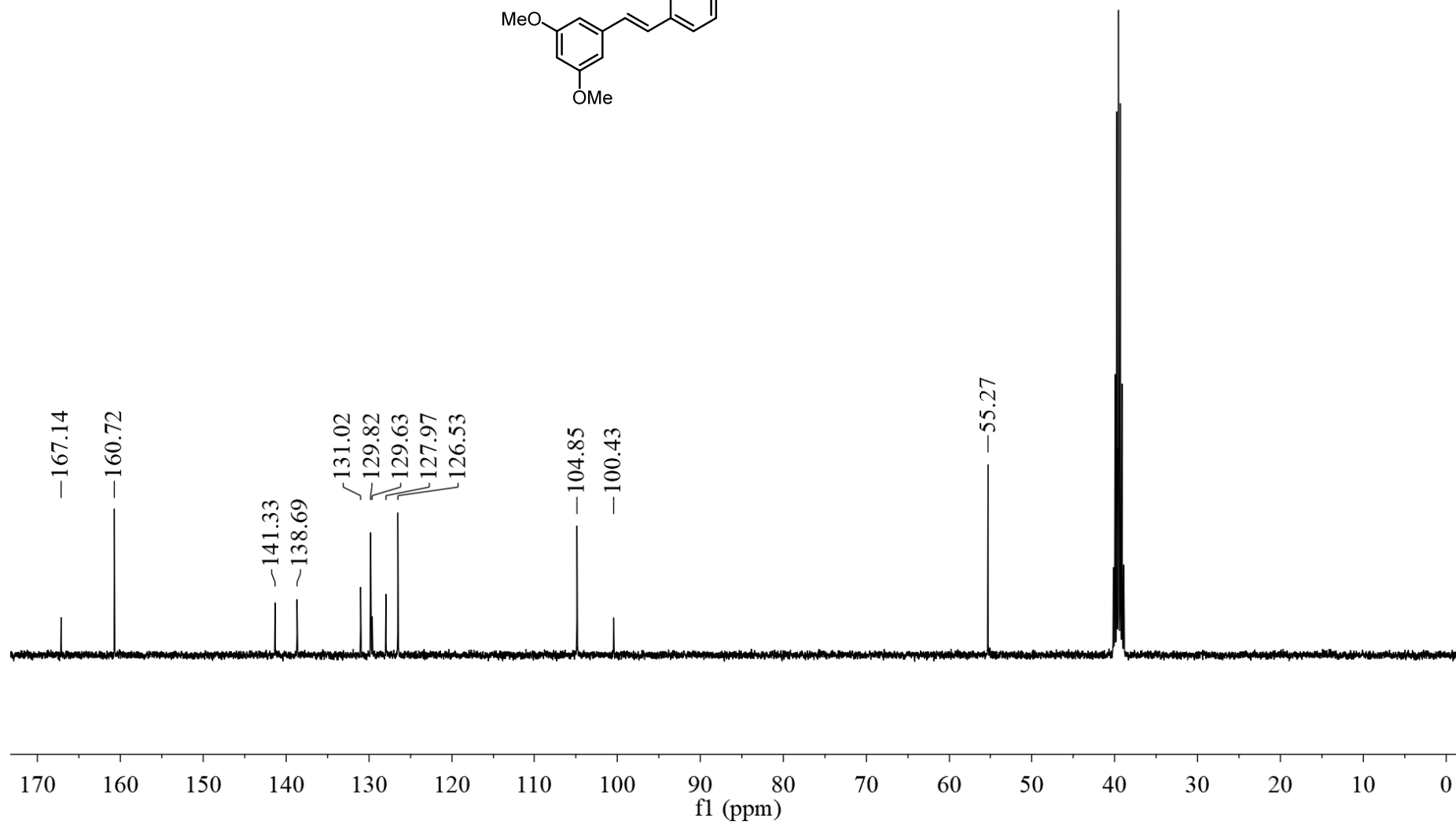
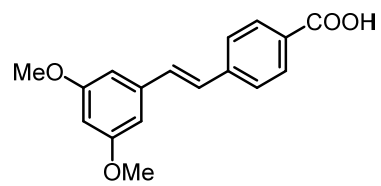




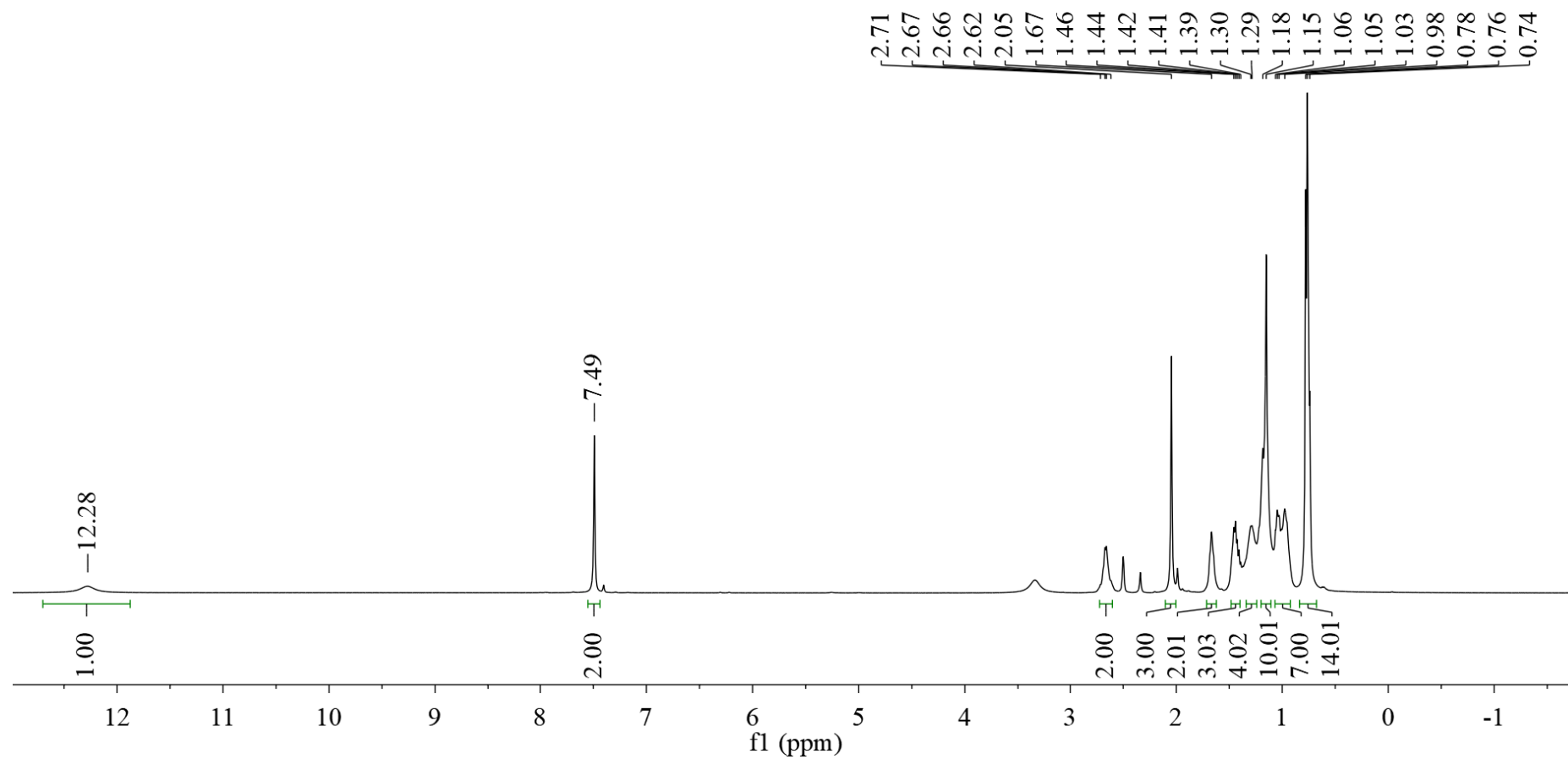
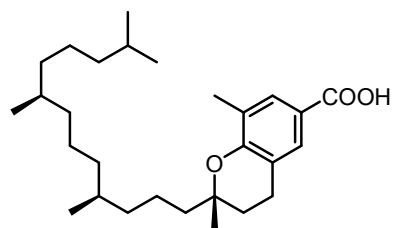
<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) spectrum of **3**



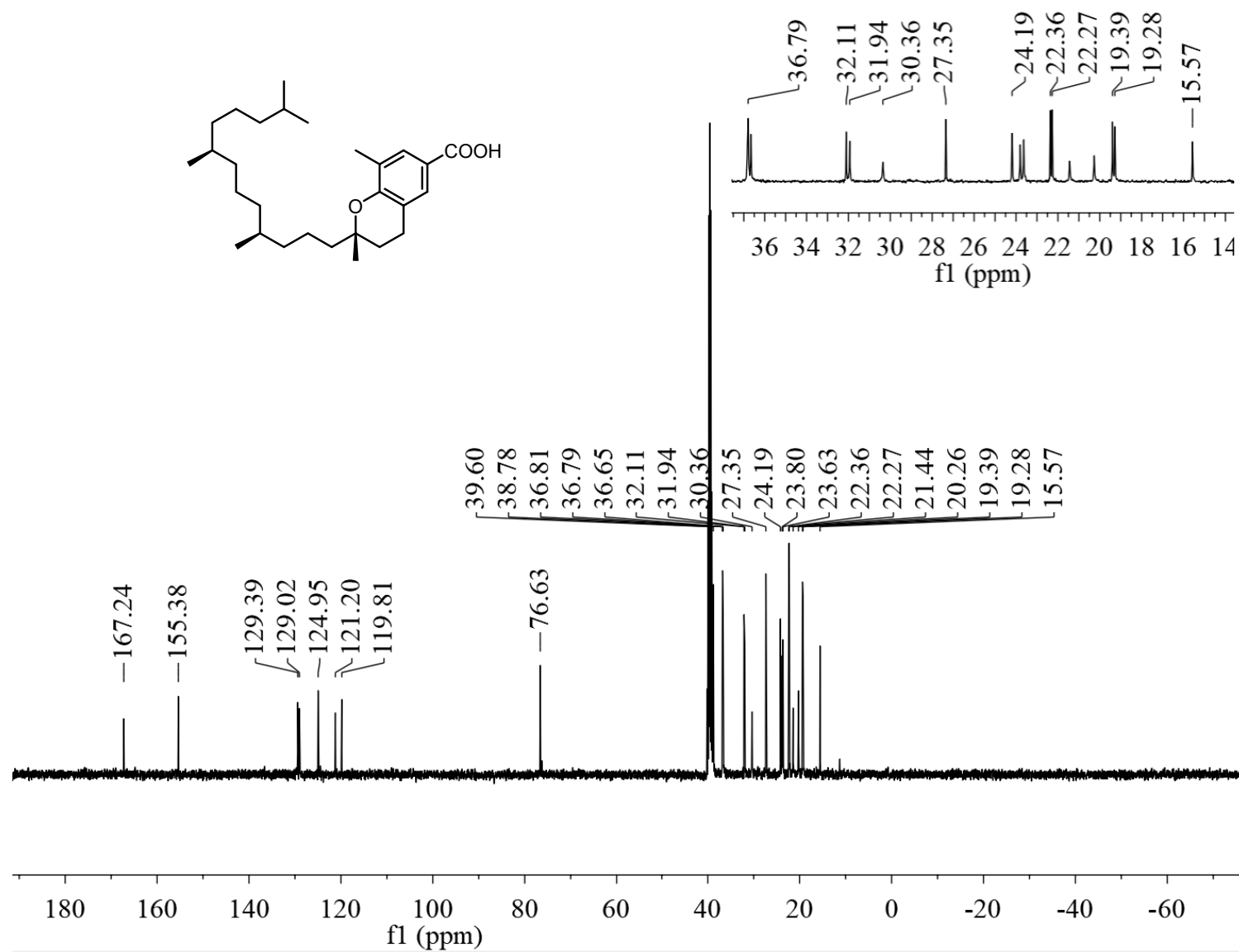
$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of **3**



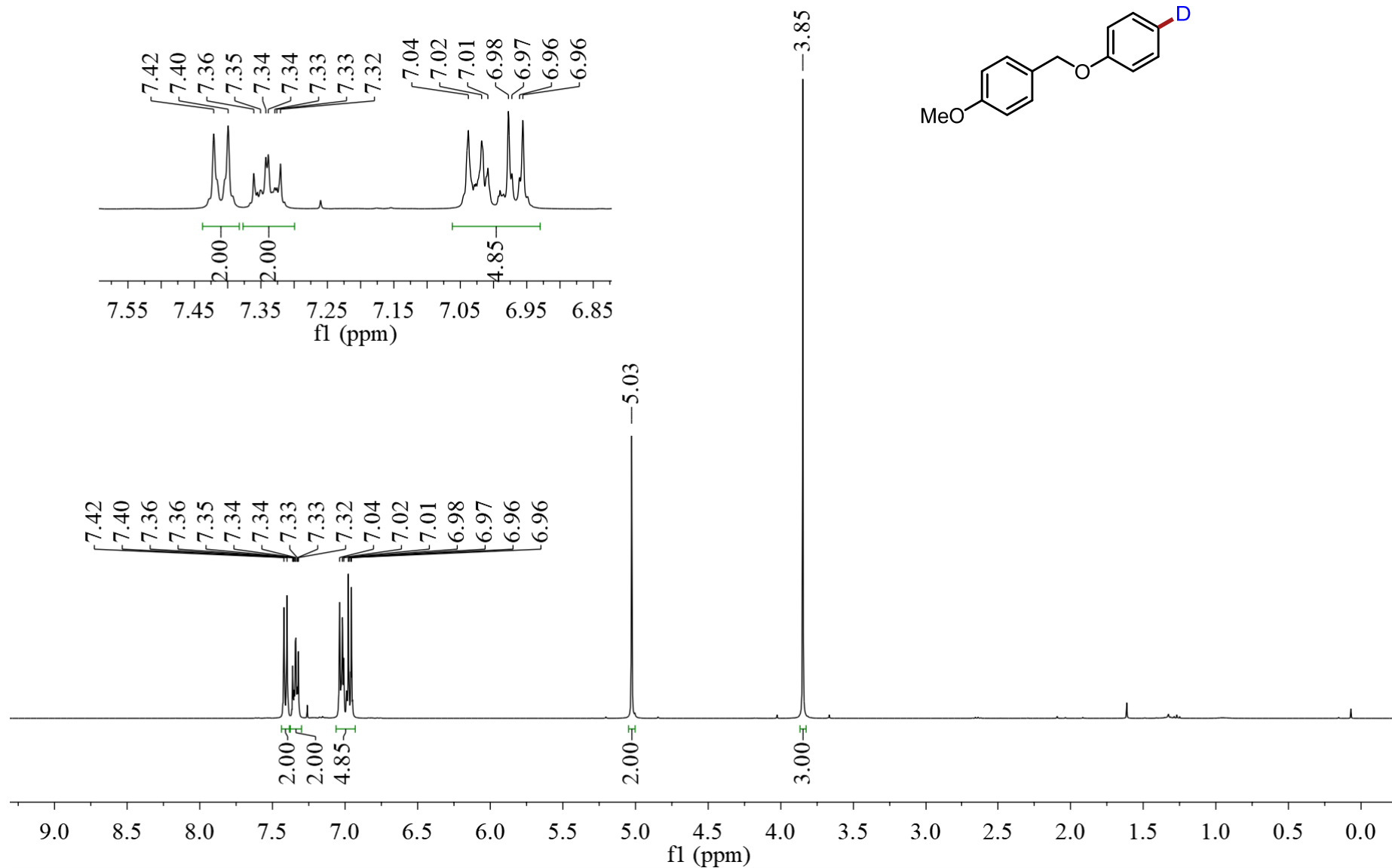
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) spectrum of **4**



$^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectrum of 4



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectrum of **5**



$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectrum of **5**

