

# A Selective, Efficient and Environmentally Friendly Method for the Oxidative Cleavage of Glycols

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## Electronic Supplementary Information

### Index

General methods .....	2
Preparation of catalysts.....	2
General procedure for the oxidative cleavage of glycols and oxidation of acyloins .....	3
Recycling study for the oxidative cleavage of 2,3-diphenylbutane-2,3-diol .....	3
E-factors calculations for the oxidative cleavage of 2,3-diphenylbutane-2,3-diol .....	4
NMR spectra of reactions of dichlorodioxomolybdenum(VI) complexes with DMSO.....	5
NMR spectra of crude reaction mixtures of the recycling study before extraction .....	7
NMR spectra of crude products in Tables 1-2 and Schemes 1 and 4.....	13
Selection of GC-MS chromatograms and spectra .....	96

**General methods:** All reactions were assembled under air atmosphere unless otherwise noted. All common reagents and solvents were obtained from commercial suppliers and used without any further purification. Non-commercially available glycols were prepared by known procedures for the pinacol coupling.<sup>1</sup> TLC was performed on aluminum-backed plates coated with silica gel 60 with F<sub>254</sub> indicator; the chromatograms were visualized under ultraviolet light and/or by staining with a Ce/Mo reagent and subsequent heating. NMR spectra were measured on Varian Mercury-Plus 300 MHz and Varian Inova-400 MHz spectrometers. GC-MS were recorded on an Agilent 6890N/5973 Network GC System, equipped with a HP-5MS column. Products were characterized by NMR spectroscopy<sup>2</sup> and capillary gas chromatography (GC). The microwave heating was performed in a microwave reactor (CEM Discover S-Class) with a single-mode microwave cavity producing continuous irradiation (temperature measurements were conducted using an IR sensor located below the microwave cavity floor, and reaction times refer to the total hold time at the indicated temperature. The maximum wattage supplied was 300 W).

**Preparation of catalysts: MoO<sub>2</sub>Cl<sub>2</sub>(L)<sub>2</sub>; Typical Procedure:<sup>3</sup>**

The catalysts can be prepared as previously reported<sup>4</sup> or more readily as follows:

To a solution of powdered Na<sub>2</sub>MoO<sub>4</sub>·2H<sub>2</sub>O (2.42 g, 10 mmol) in H<sub>2</sub>O (5 mL), concd HCl (8.3 g, ca. 100 mmol) was added. The mixture was stirred at room temperature for 10–15 min resulting in a colorless solution along with a significant amount of crystallized NaCl. Then Et<sub>2</sub>O (15 mL) was added with stirring and the mixture vigorously shaken for 1–2 min. The upper Et<sub>2</sub>O layer was collected and the extraction process repeated twice more. The combined ethereal extract was stirred for 15 min with anhydrous MgSO<sub>4</sub> (2 g). The solution was collected by filtration and the MgSO<sub>4</sub> was washed with Et<sub>2</sub>O (3 × 3 mL).

(a) Synthesis of MoO<sub>2</sub>Cl<sub>2</sub>(dmf)<sub>2</sub>: The resulting solution, containing approximately 98% of the original molybdenum, was treated with a solution of DMF (1.54 g, 21 mmol) in

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<sup>1</sup> (a) C. Wang, Y. Pan, A. Wu, *Tetrahedron* 2007, **63**, 429; (b) J. M. Khurana, A. Sehgal, A. Gogia, A. Manian, G. C. Maikap, *J. Chem. Soc., Perkin Trans. 1* 1996, 2213.

<sup>2</sup> Spectroscopic data of the synthesized carbonyl compounds were identical to those of commercially available samples.

<sup>3</sup> (a) F. J. Arnáiz, R. Aguado, M. R. Pedrosa and A. De Cian, *Inorg. Chim. Acta* 2003, **347**, 33; (b) R. Sanz, J. Escribano, R. Aguado, M. R. Pedrosa, F. J. Arnáiz, *Synthesis* 2004, 1629.

<sup>4</sup> F. J. Arnáiz, *Inorg. Synth.* 1997, **31**, 246.

Et<sub>2</sub>O (10 mL). The resulting mixture was stirred for 5 min and the white microcrystalline precipitate filtered, washed with Et<sub>2</sub>O (3 × 3 mL) and dried under vacuum. Yield: 3.37 g (97.5%).

(b) Synthesis of MoO<sub>2</sub>Cl<sub>2</sub>(dmsO)<sub>2</sub>: The resulting solution was treated with a solution of DMSO (1.64 g, 21 mmol) in Et<sub>2</sub>O (10 mL). The resulting mixture was stirred for 5 min and the white microcrystalline precipitate filtered, washed with Et<sub>2</sub>O (3 × 3 mL) and dried under vacuum. Yield: 3.45 g (97%).

### **General procedure for the oxidative cleavage of glycols and oxidation of**

**acyloins:** A mixture of DMSO (6 equiv), the corresponding glycol or acyloin (1 equiv), and MoO<sub>2</sub>Cl<sub>2</sub>(dmsO)<sub>2</sub> (2 mol%) was irradiated in a sealed tube in the microwave cavity at the reported temperature for the required time (see Tables 1-2 and Scheme 4). For reactions carried out in DMSO-d<sub>6</sub> the crude mixture was analyzed by NMR after cooling to room temperature. For the experiments performed in DMSO and for calculating the yield, the reaction mixture was cooled to room temperature and an ethereal solvent (Et<sub>2</sub>O or cyclopentyl methyl ether) (5 mL) and H<sub>2</sub>O (5 mL) were added. The layers were separated and the aqueous layer extracted with the ether (2 × 5 mL). The combined organic layers were washed with water to completely remove the excess of DMSO, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and then the solvents were removed under reduced pressure. The corresponding crude mixtures were analyzed by NMR.

### **Recycling study for the oxidative cleavage of 2,3-diphenylbutane-2,3-**

**diol:** A mixture of DMSO-d<sub>6</sub> (1 mL, ~15 mmol), 2,3-diphenylbutane-2,3-diol (242 mg, 1 mmol) and MoO<sub>2</sub>Cl<sub>2</sub>(dmsO)<sub>2</sub> (7 mg, 2.0 mol%) was irradiated in a sealed tube in the microwave cavity at 130 °C for 10 min. After cooling to room temperature, the crude mixture was analyzed by NMR and then extracted with a 10:1 mixture of cyclopentyl methyl ether and *n*-heptane (2 × 1 mL). The combined organic layers were washed with water (0.3 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered, and then the solvents were removed under reduced pressure. Acetophenone was obtained in almost pure form without further purification in the yields reported in Scheme 1. The DMSO-phase containing the catalyst was reused in the next cycle by adding a new batch of diol dissolved in 0.2 mL of DMSO-d<sub>6</sub>.

## E-factors calculations for the oxidative cleavage of 2,3-diphenylbutane-

**2,3-diol:** The E-factor is the ratio between the total mass of the waste and the mass of the product. In our calculations the solvents employed in the purification were considered. We calculate the E-factor for the cleavage of 2,3-diphenylbutane-diol for both the conventional process (Table 2, entry 4) and the recycling experiment (Scheme 1).

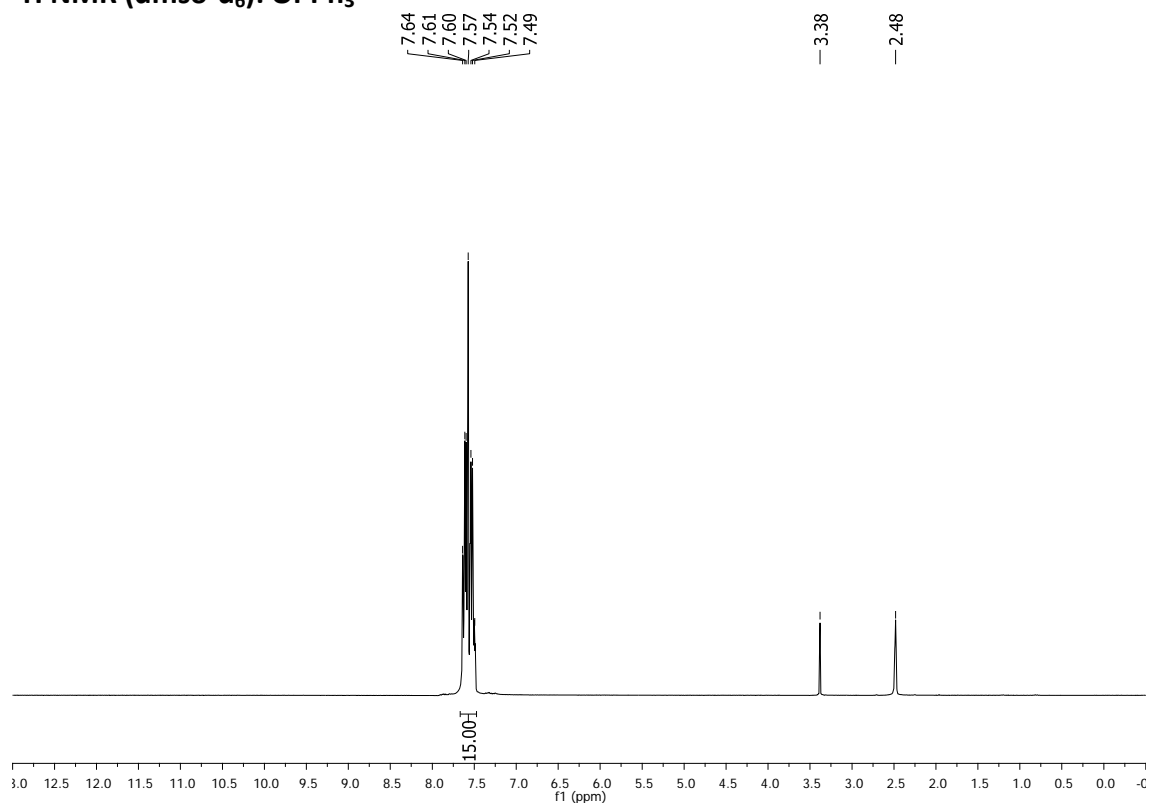
### Conventional process:

### Recycling process:

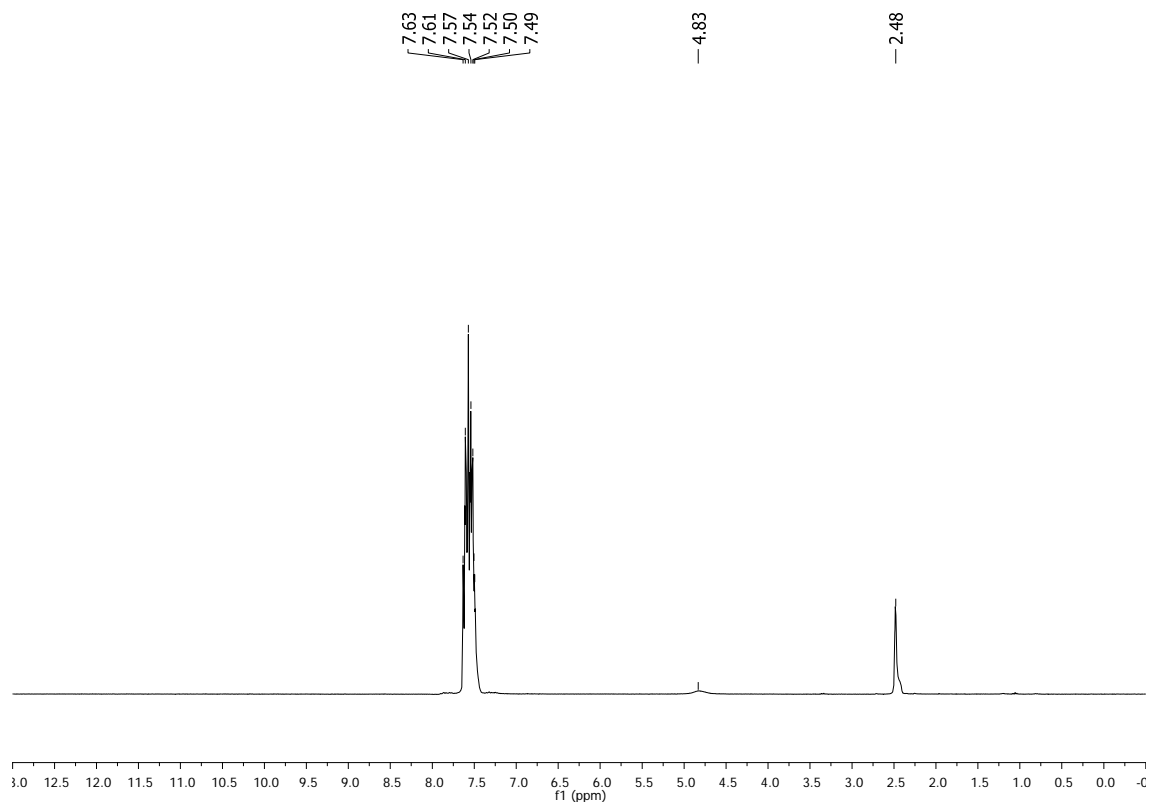
Reactant/Reagent	Mass (g)	Reactant/Reagent (5 cycles)	Mass (g)
2,3-diphenylbutane-2,3-diol	0.242	2,3-diphenylbutane-2,3-diol	1.21
DMSO (0.4 mL)	0.44	DMSO (1 mL + 0.2 mL x 4)	1.98
MoO <sub>2</sub> Cl <sub>2</sub> (dmsO) <sub>2</sub>	0.007	MoO <sub>2</sub> Cl <sub>2</sub> (dmsO) <sub>2</sub>	0.007
Et <sub>2</sub> O (15 mL)	10.7	cyclopentyl methyl ether (9.1 mL)	7.826
		<i>n</i> -heptane (0.9 mL)	0.616
Total	11.389	Total	11.639
Acetophenone (93% yield)	0.223	Acetophenone (79% av. yield)	0.953
Total waste	11.166	Total waste	10.686
<b>E factor</b>	<b>50.1</b>	<b>E factor</b>	<b>11.2</b>

## NMR spectra of reactions of dichlorodioxomolybdenum(VI) complexes with DMSO:

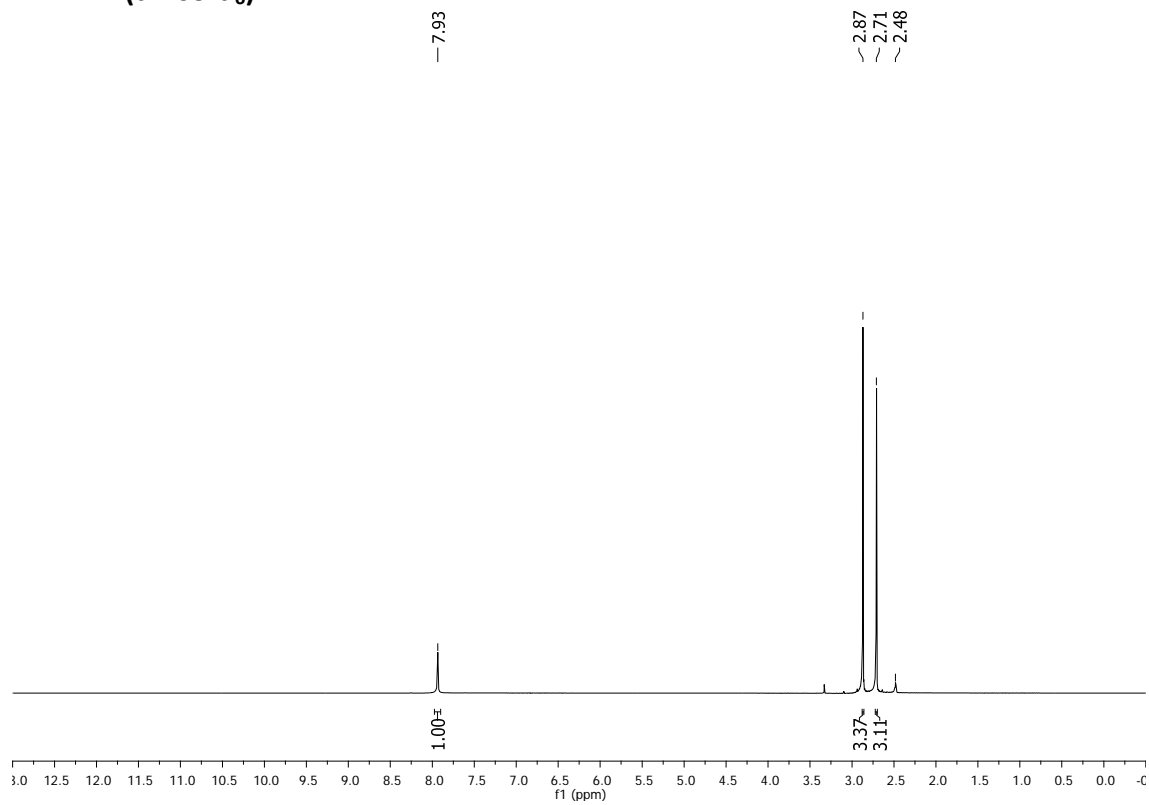
$^1\text{H}$  NMR (dms $o$ -d $_6$ ):  $\text{OPPh}_3$



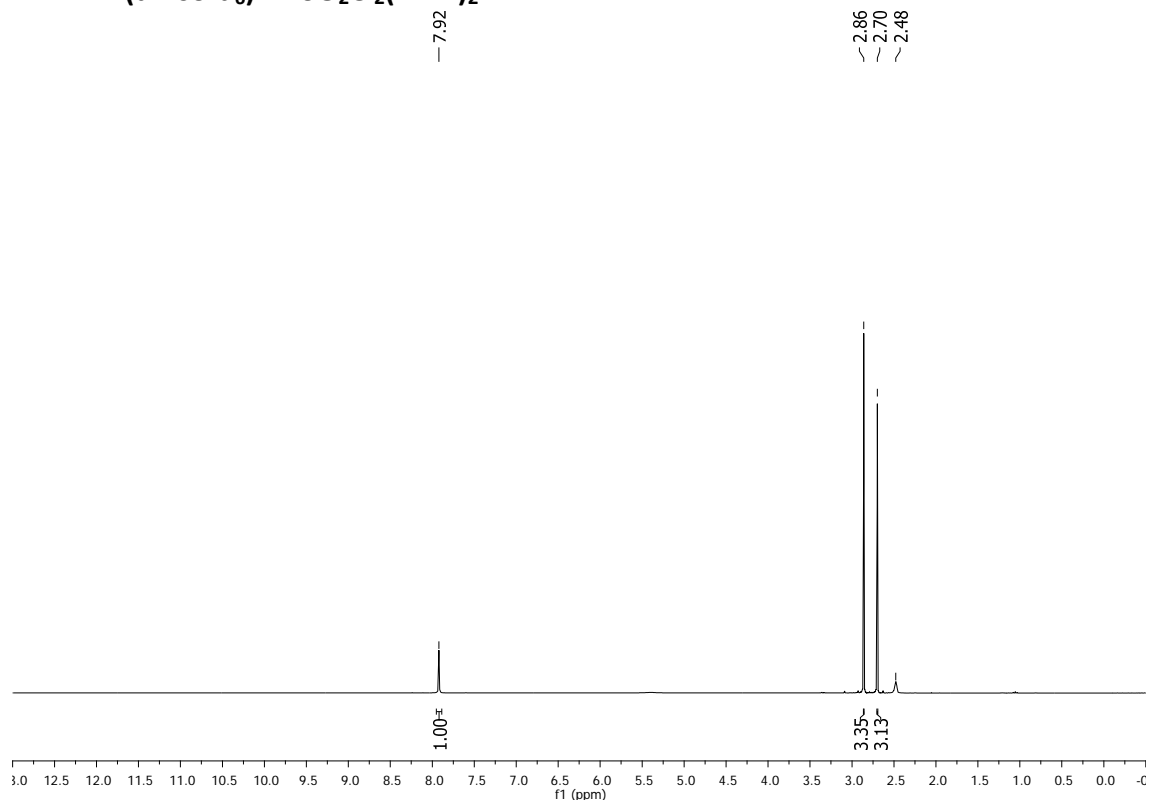
$^1\text{H}$  NMR (dms $o$ -d $_6$ ):  $\text{MoO}_2\text{Cl}_2(\text{OPPh}_3)_2$



**$^1\text{H}$  NMR (dms $o$ -d $_6$ ): DMF**



**$^1\text{H}$  NMR (dms $o$ -d $_6$ ): MoO $_2$ Cl $_2$ (DMF) $_2$**



**NMR spectra of crude reaction mixtures of the recycling study before extraction**

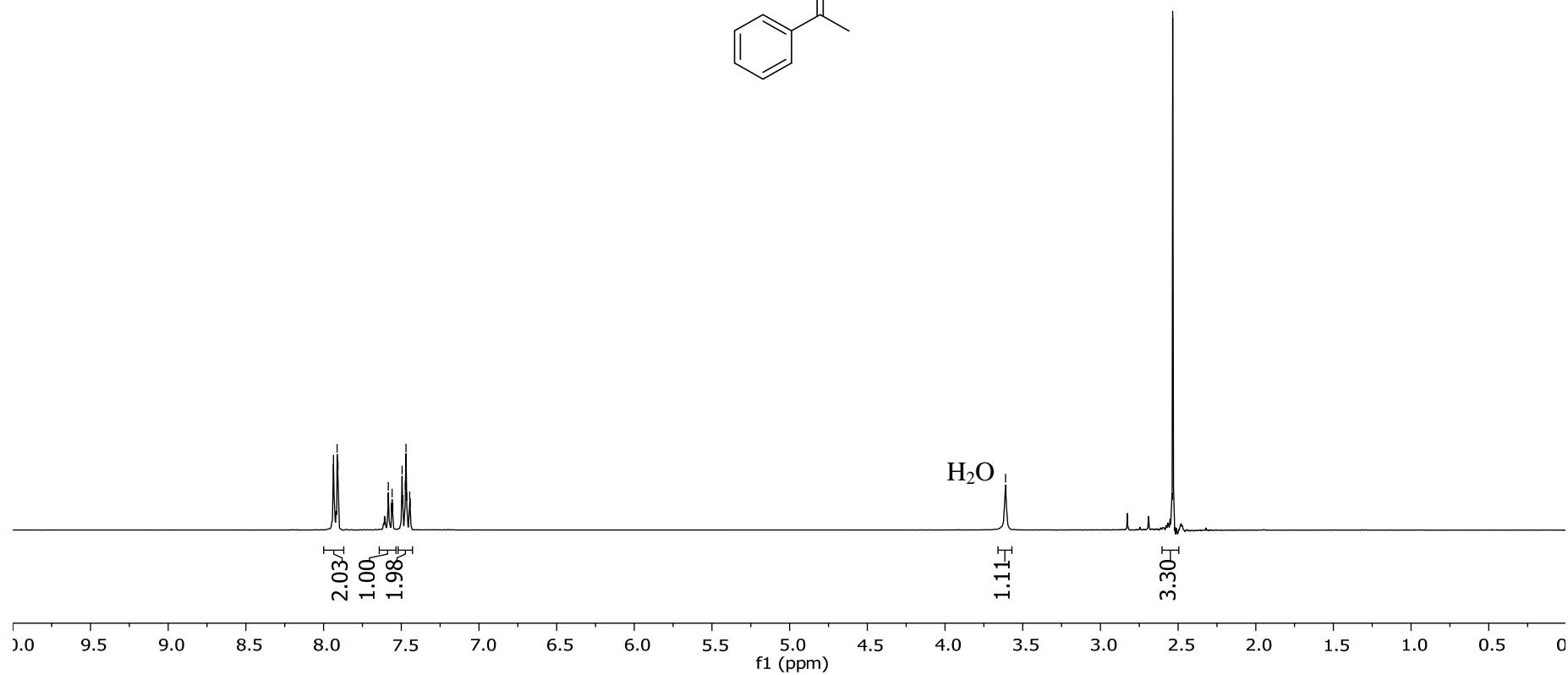
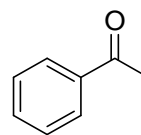
7.94  
7.91  
7.91  
7.58  
7.56  
7.56  
7.55  
7.49  
7.49  
7.47  
7.47  
7.46  
7.45  
7.44

— 3.61

— 2.53

Recycling experiment: Cycle 1

$^1\text{H-NMR}$  (dms $o$ -d $_6$ , 300 MHz)





— 7.91  
~ 7.54  
~ 7.43

— 3.67

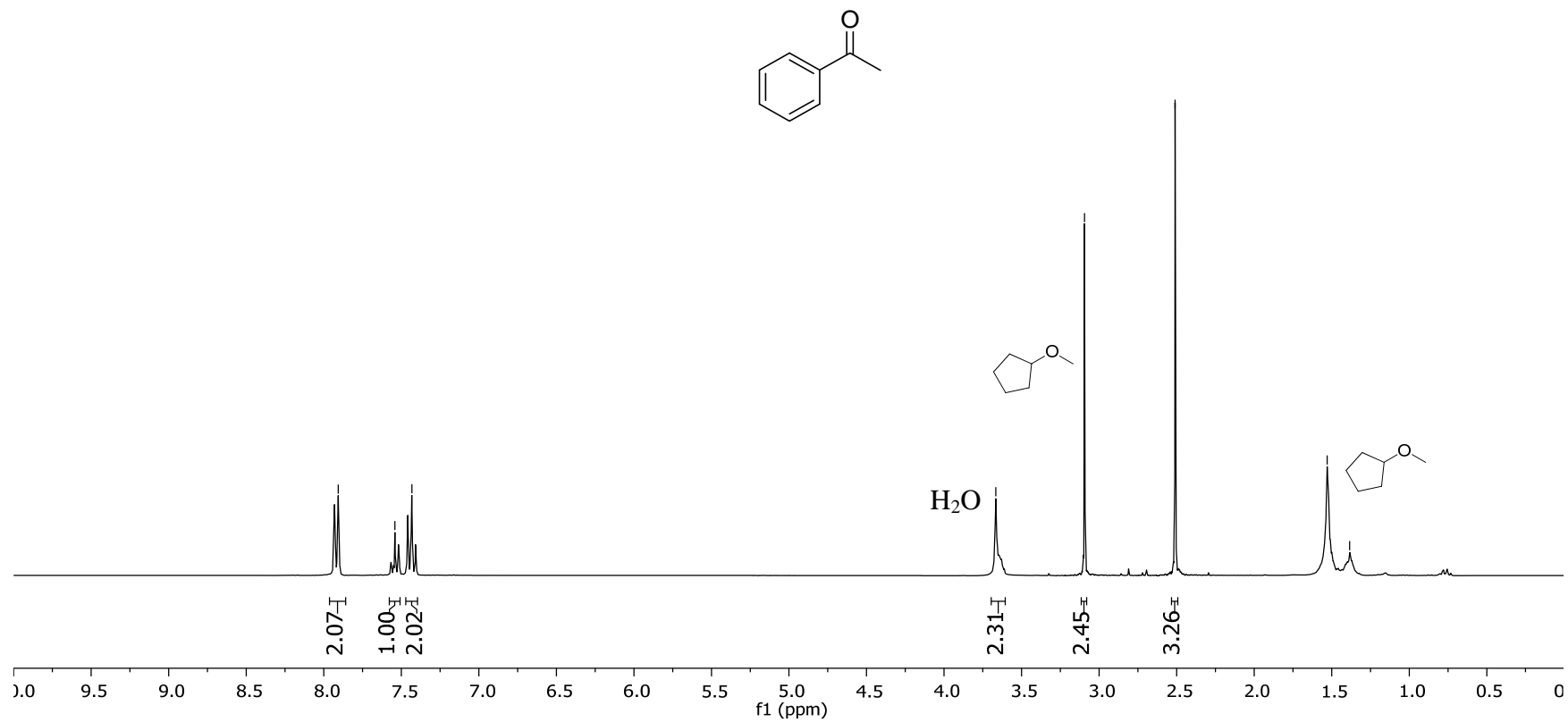
— 3.09

— 2.51

~ 1.53  
~ 1.38

Recycling experiment: Cycle 2

<sup>1</sup>H-NMR (dmsO-d<sub>6</sub>, 300 MHz)



~ 7.89  
~ 7.55  
~ 7.53  
~ 7.44

— 3.70

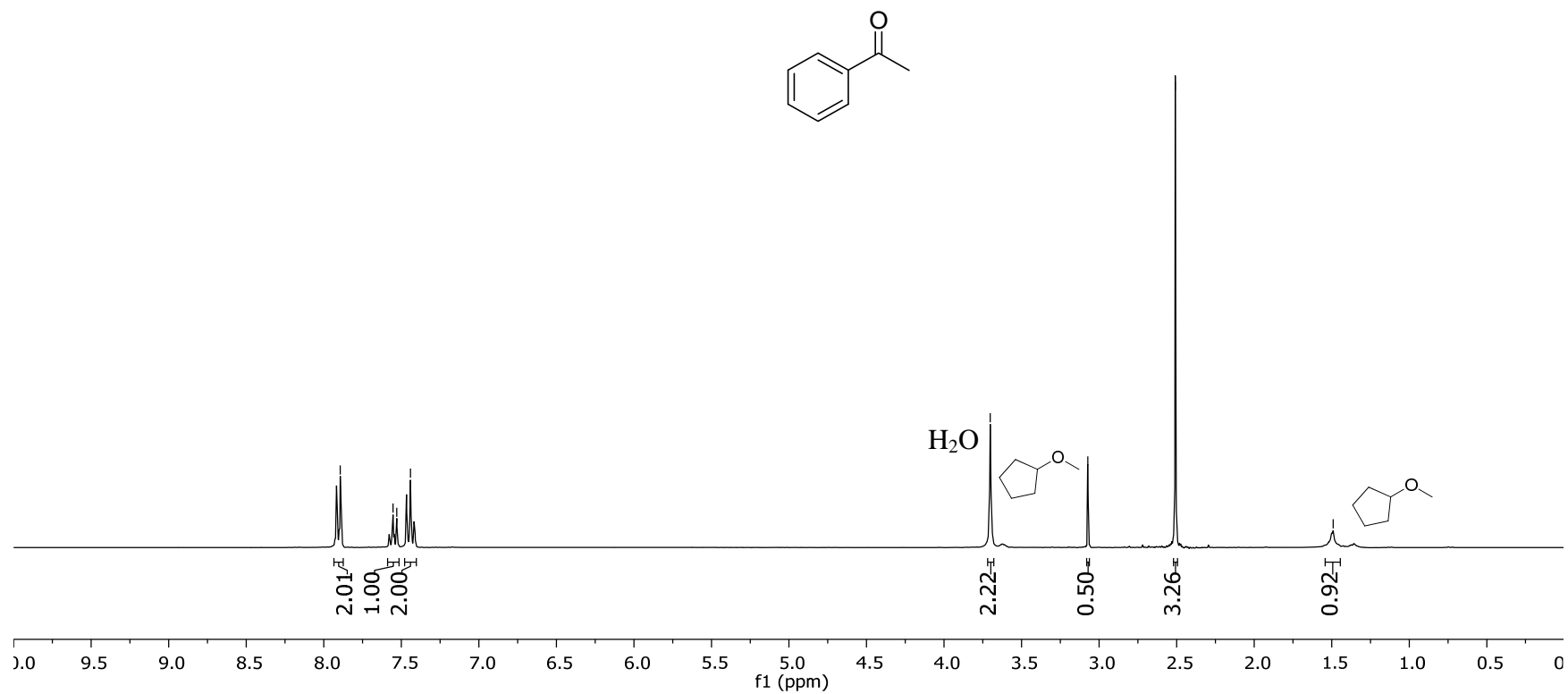
— 3.07

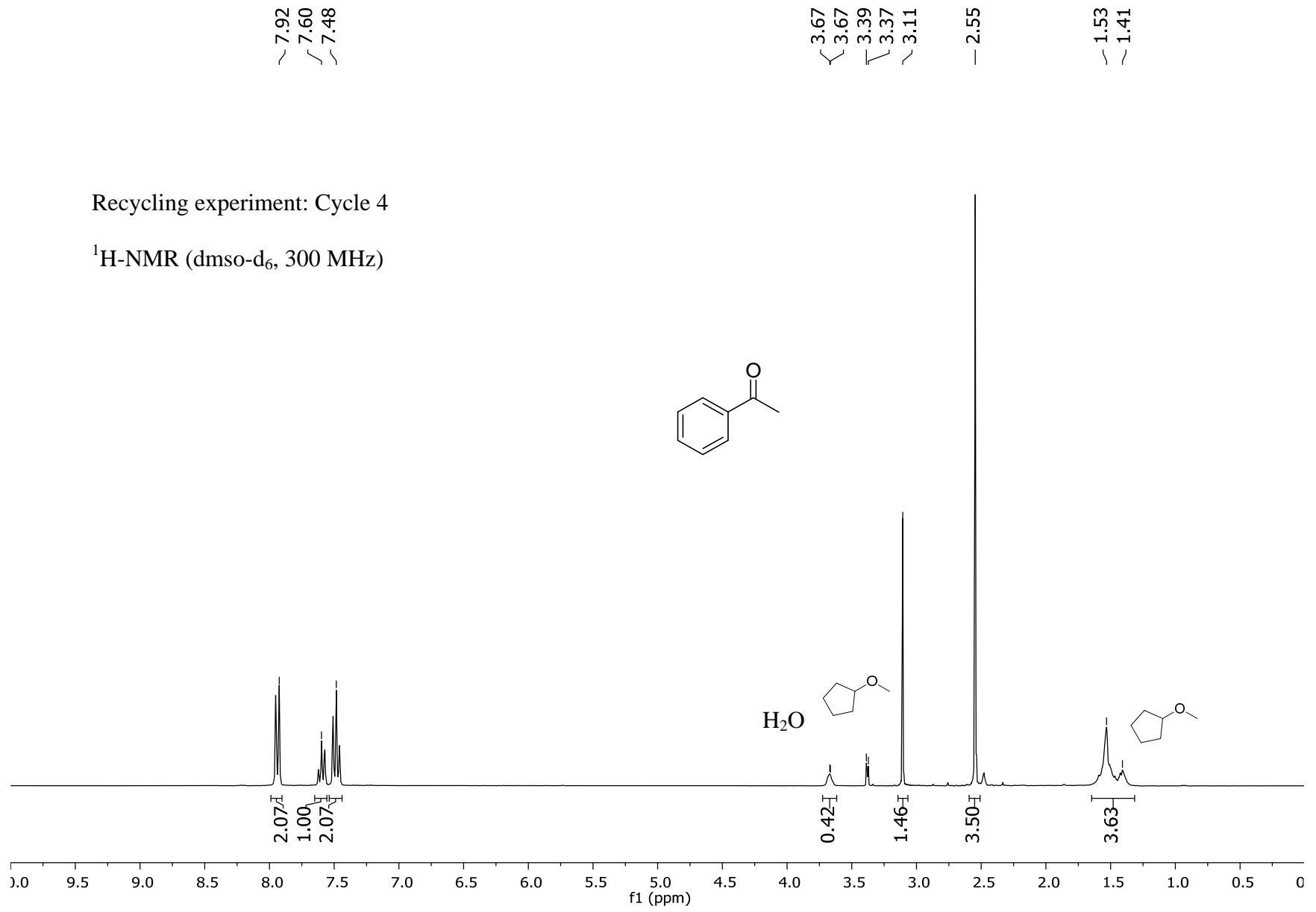
— 2.51

— 1.49

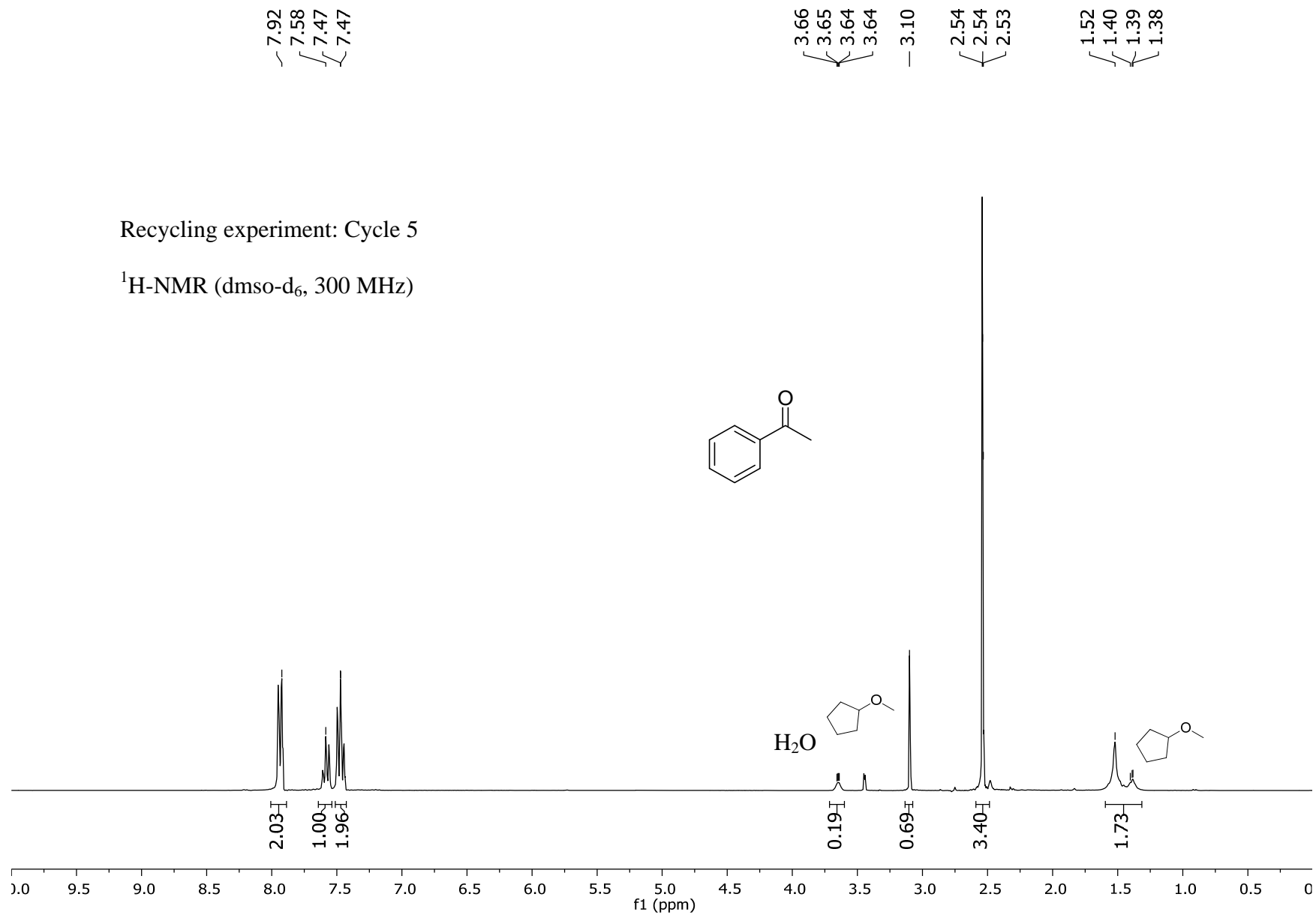
Recycling experiment: Cycle 3

$^1\text{H-NMR}$  (dms $o$ -d $_6$ , 300 MHz)





Recycling experiment: Cycle 5  
<sup>1</sup>H-NMR (dmsO-d<sub>6</sub>, 300 MHz)



**NMR spectra of crude products in Tables 1-2 and Schemes 1 and 4**

Table 1, Entry 1

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)

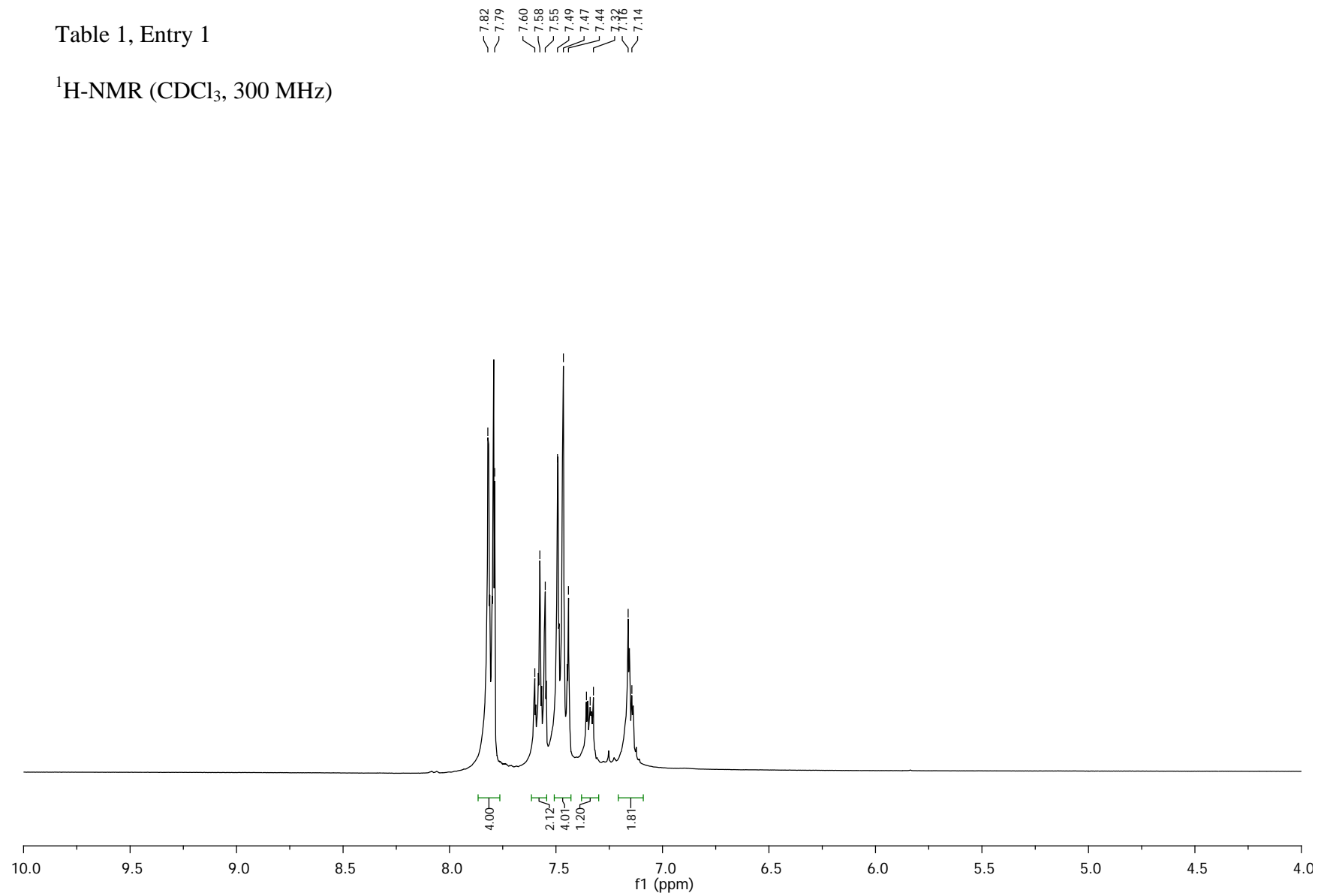
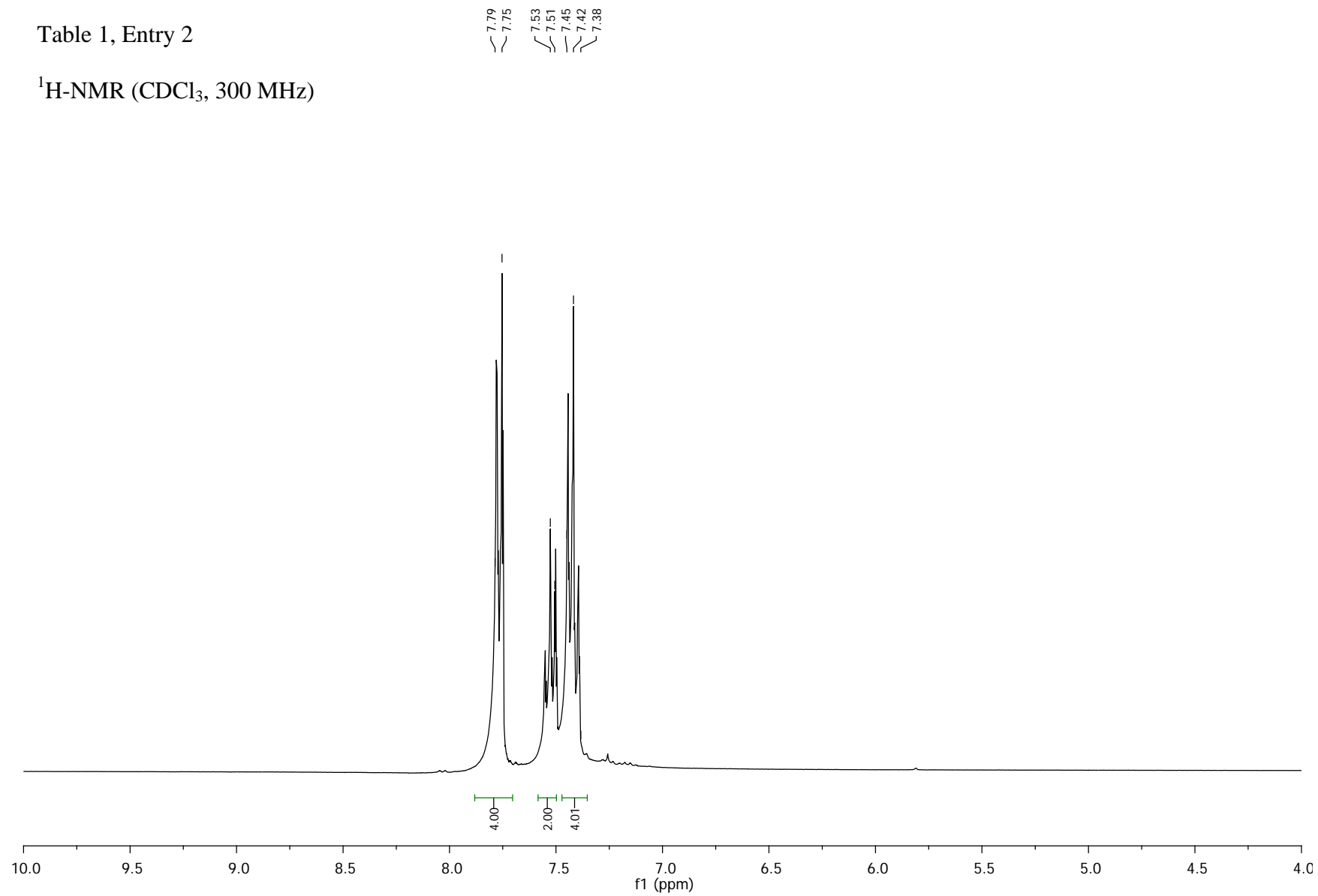


Table 1, Entry 2

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)



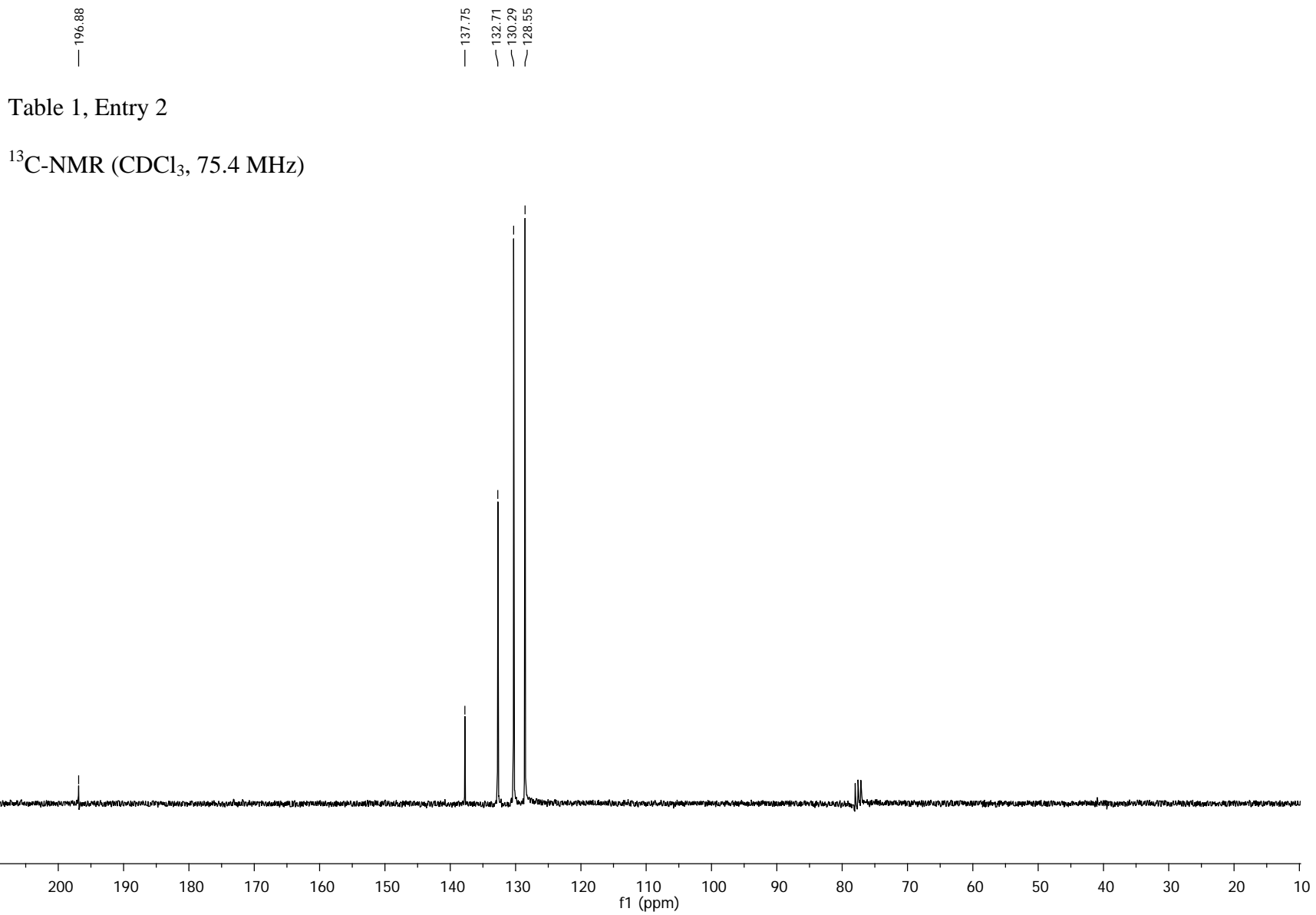




Table 1, Entry 3

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)

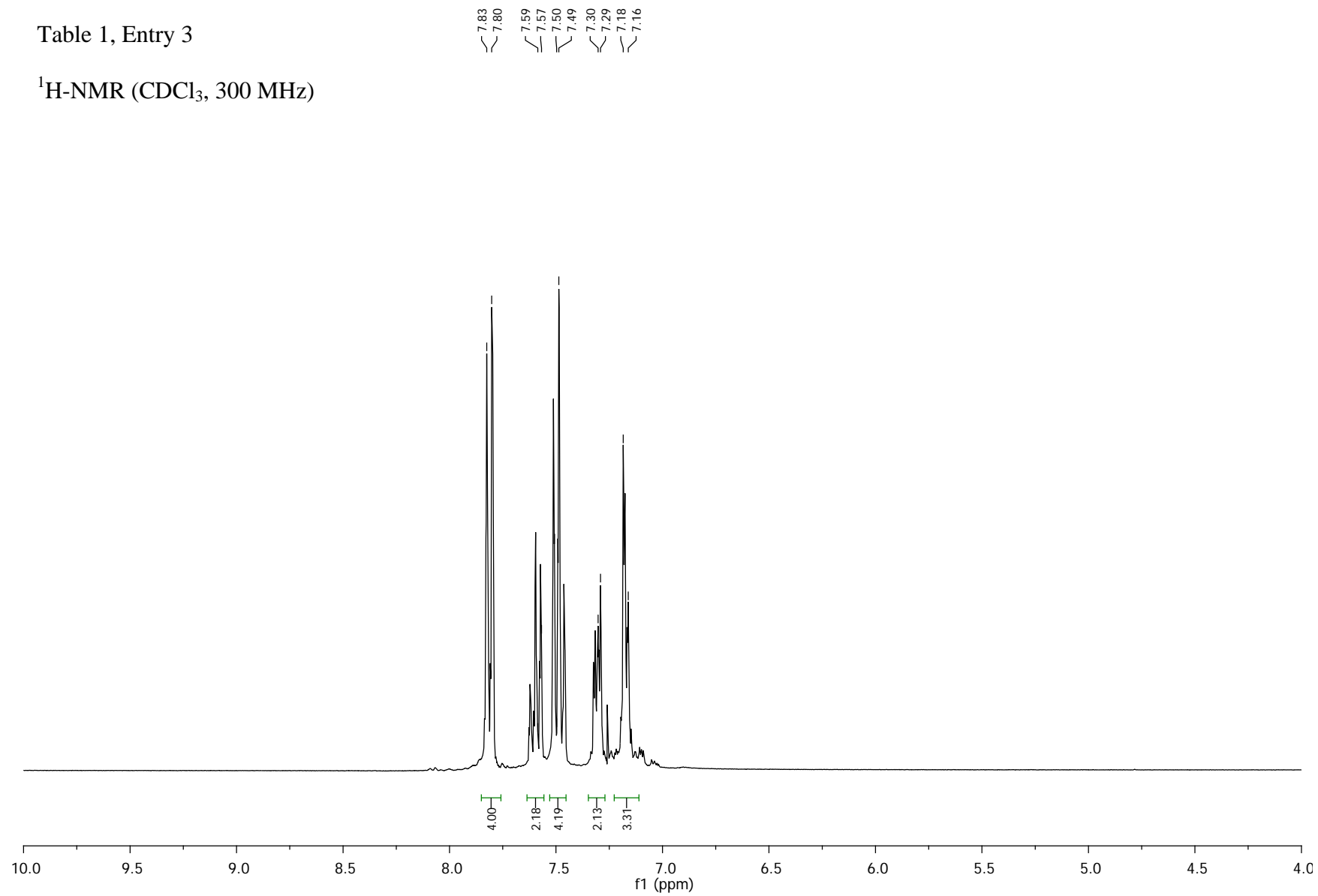


Table 1, Entry 4

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)

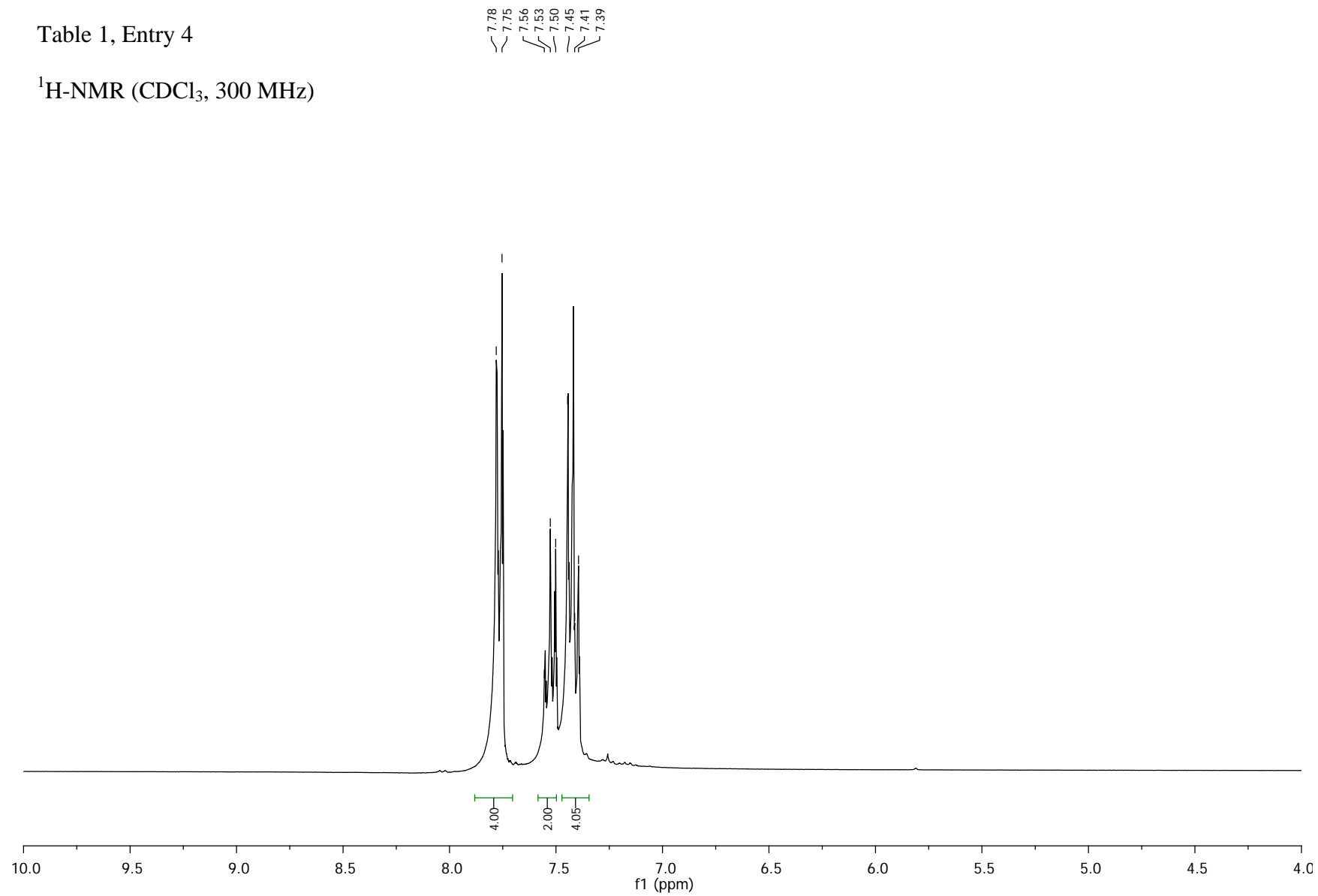
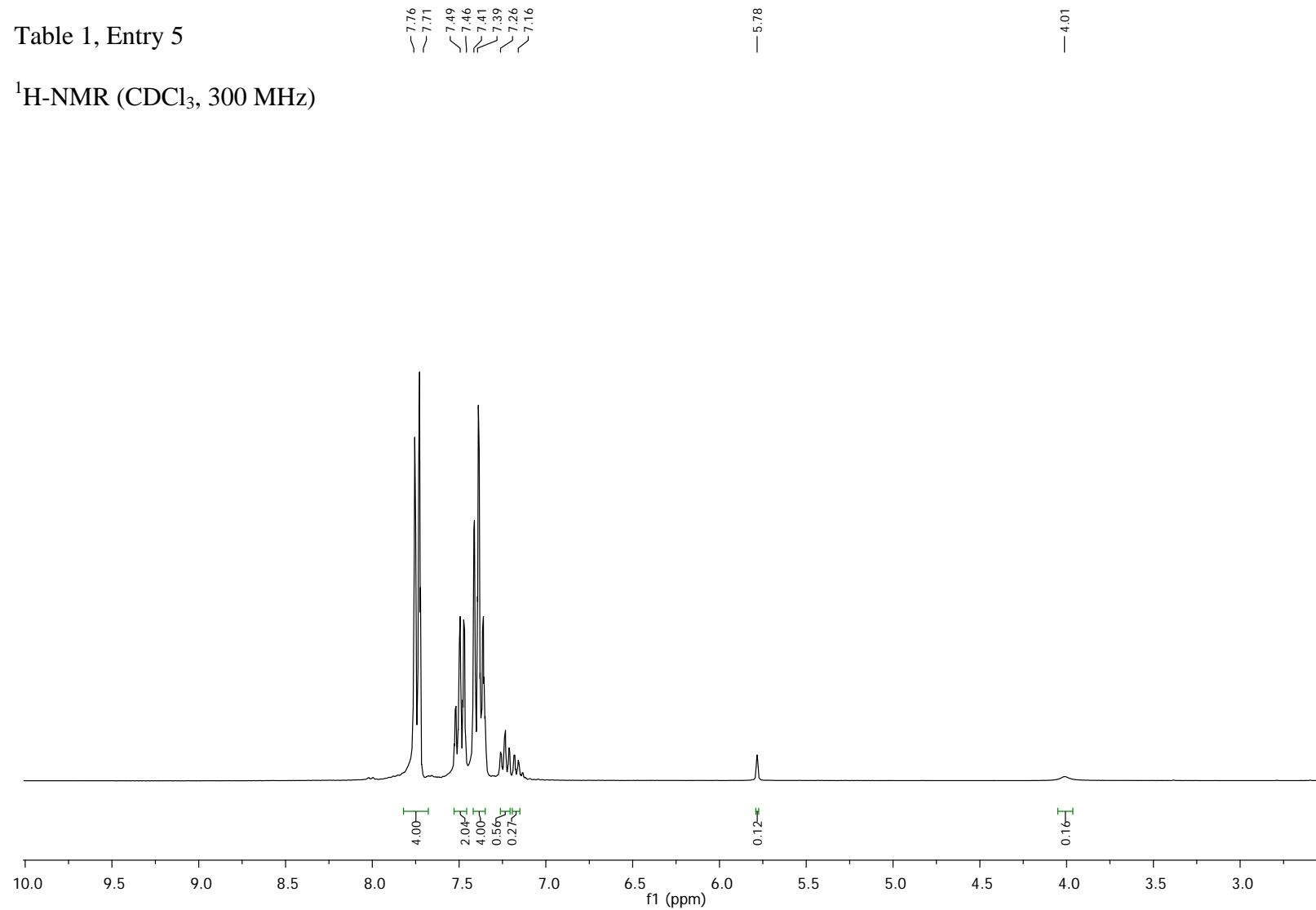


Table 1, Entry 5

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)



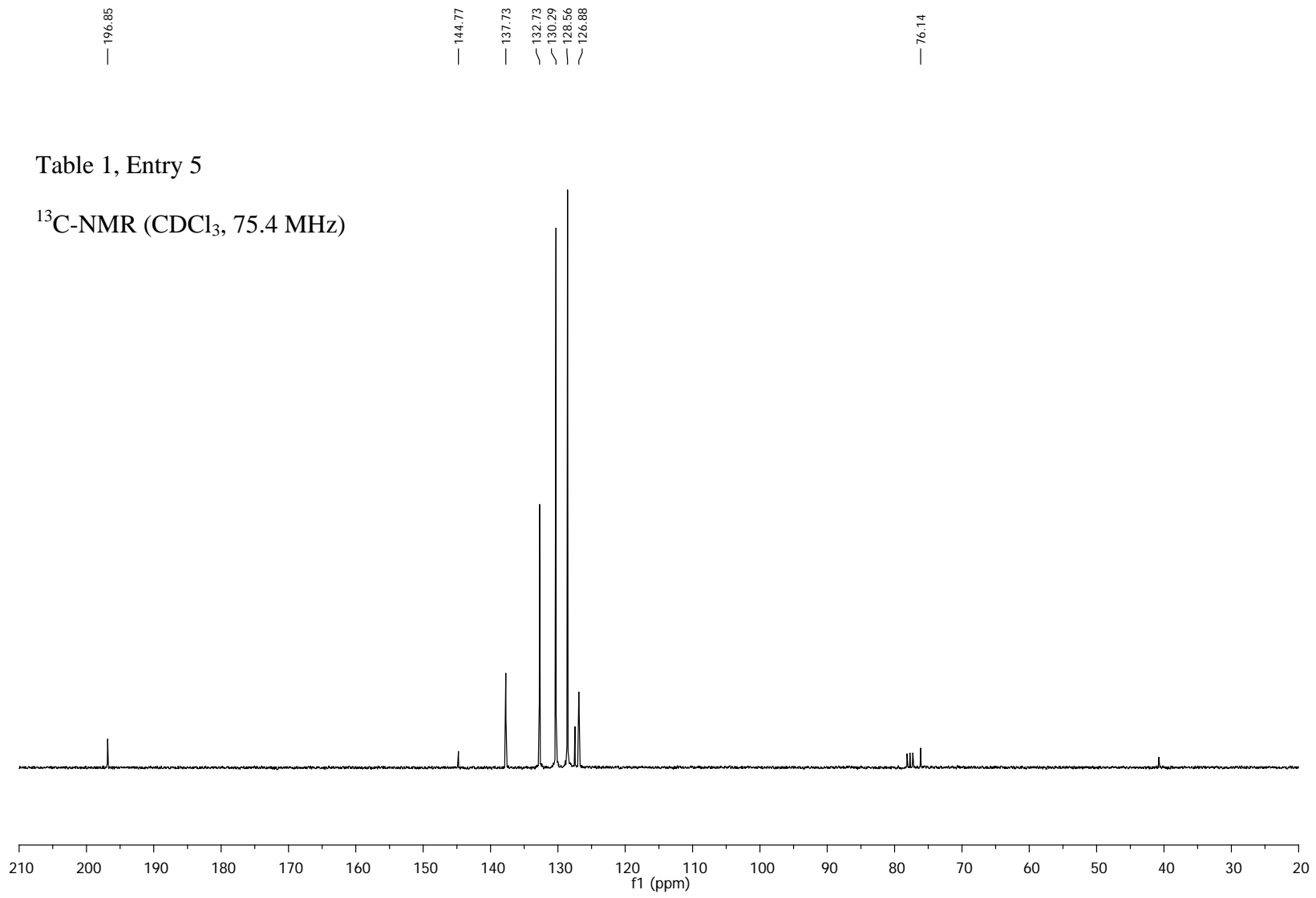


Table 1, Entry 6

$^1\text{H-NMR}$  (dms $o$ -d $_6$ , 300 MHz)

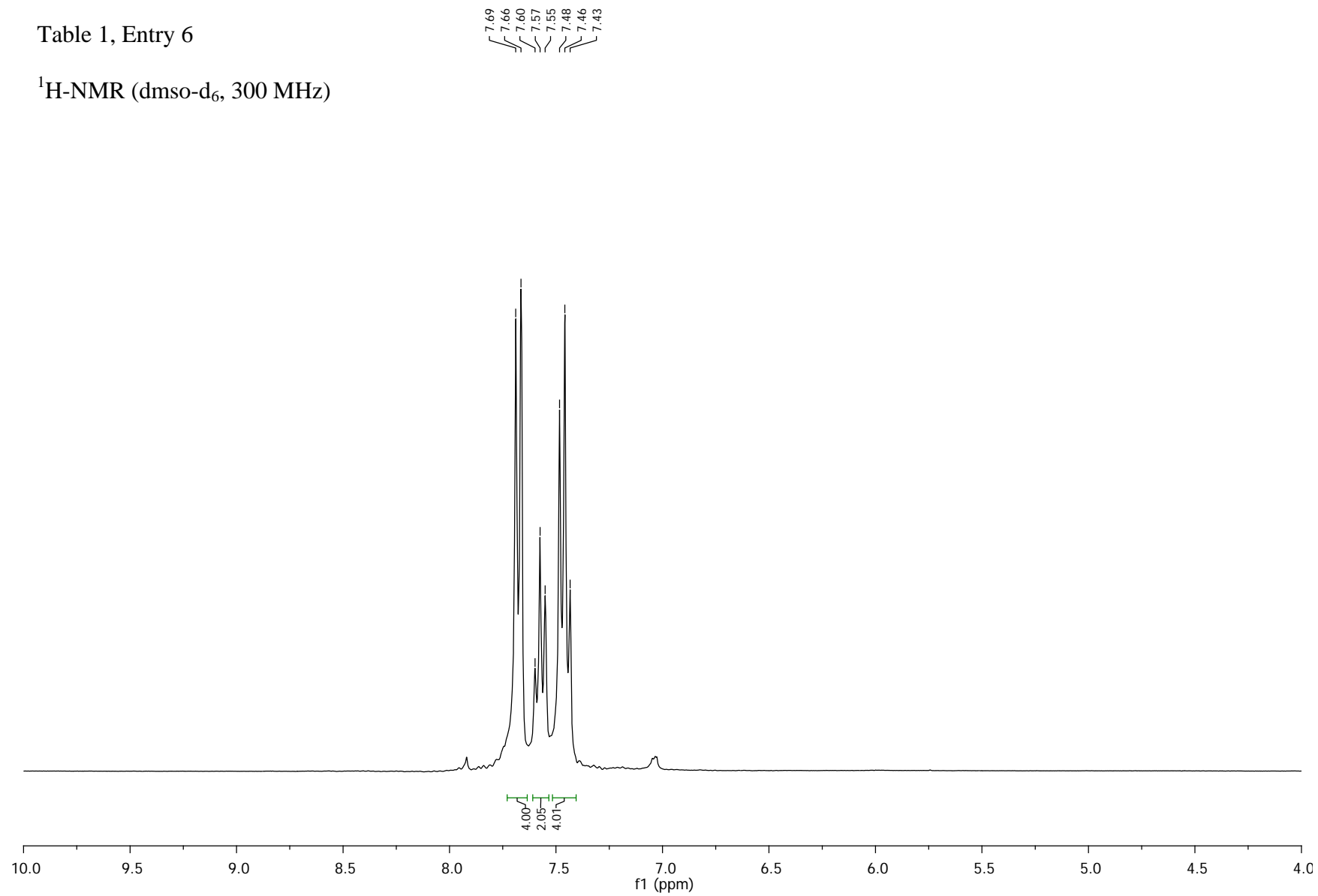


Table 1, Entry 6

$^{13}\text{C}$ -NMR (dms $o$ - $d_6$ , 75.4 MHz)

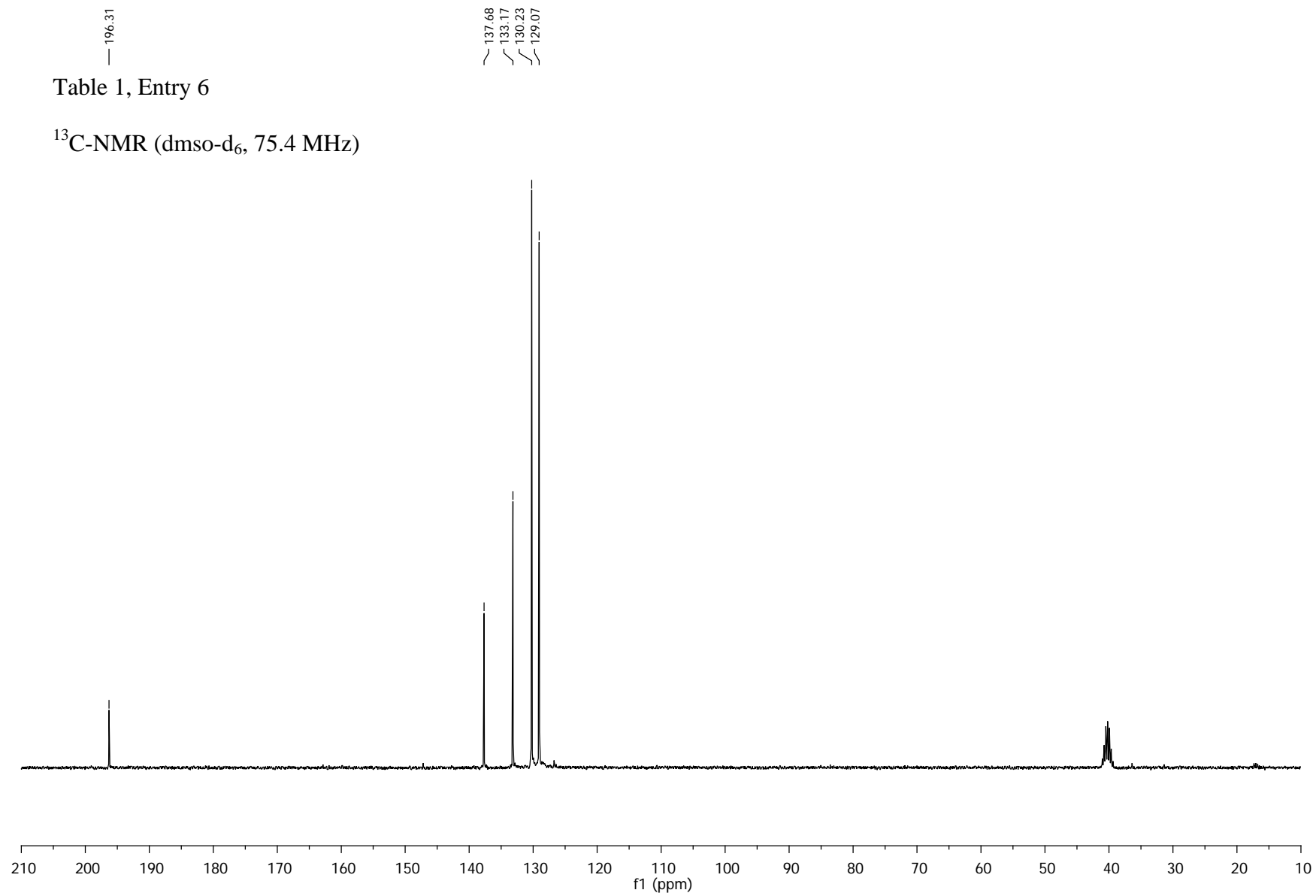
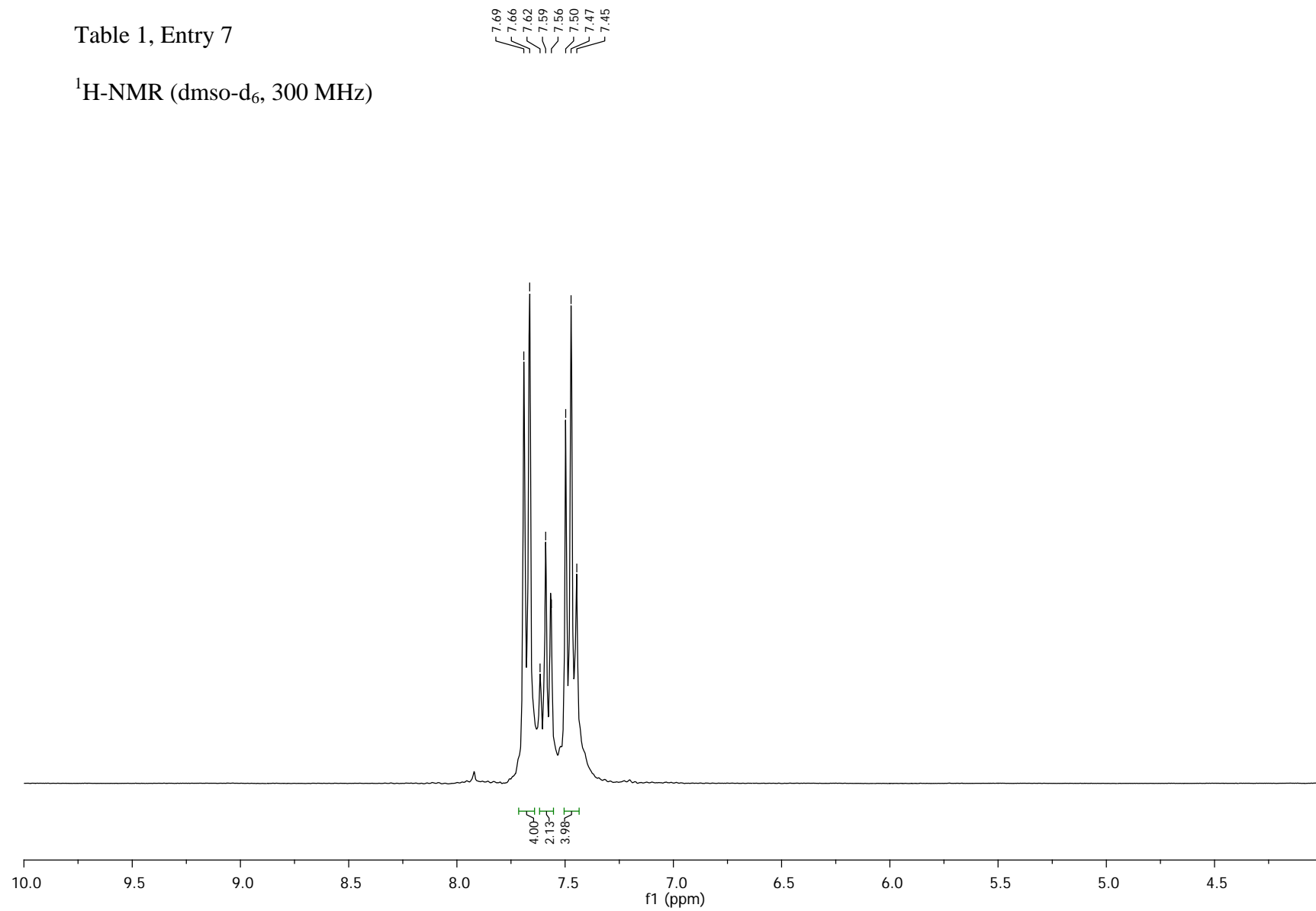


Table 1, Entry 7

$^1\text{H-NMR}$  (dms $o$ -d $_6$ , 300 MHz)



— 196.34

~ 137.67  
~ 133.20  
~ 130.23  
~ 129.09

Table 1, Entry 7

$^{13}\text{C}$ -NMR (dms $o$ -d $_6$ , 75.4 MHz)

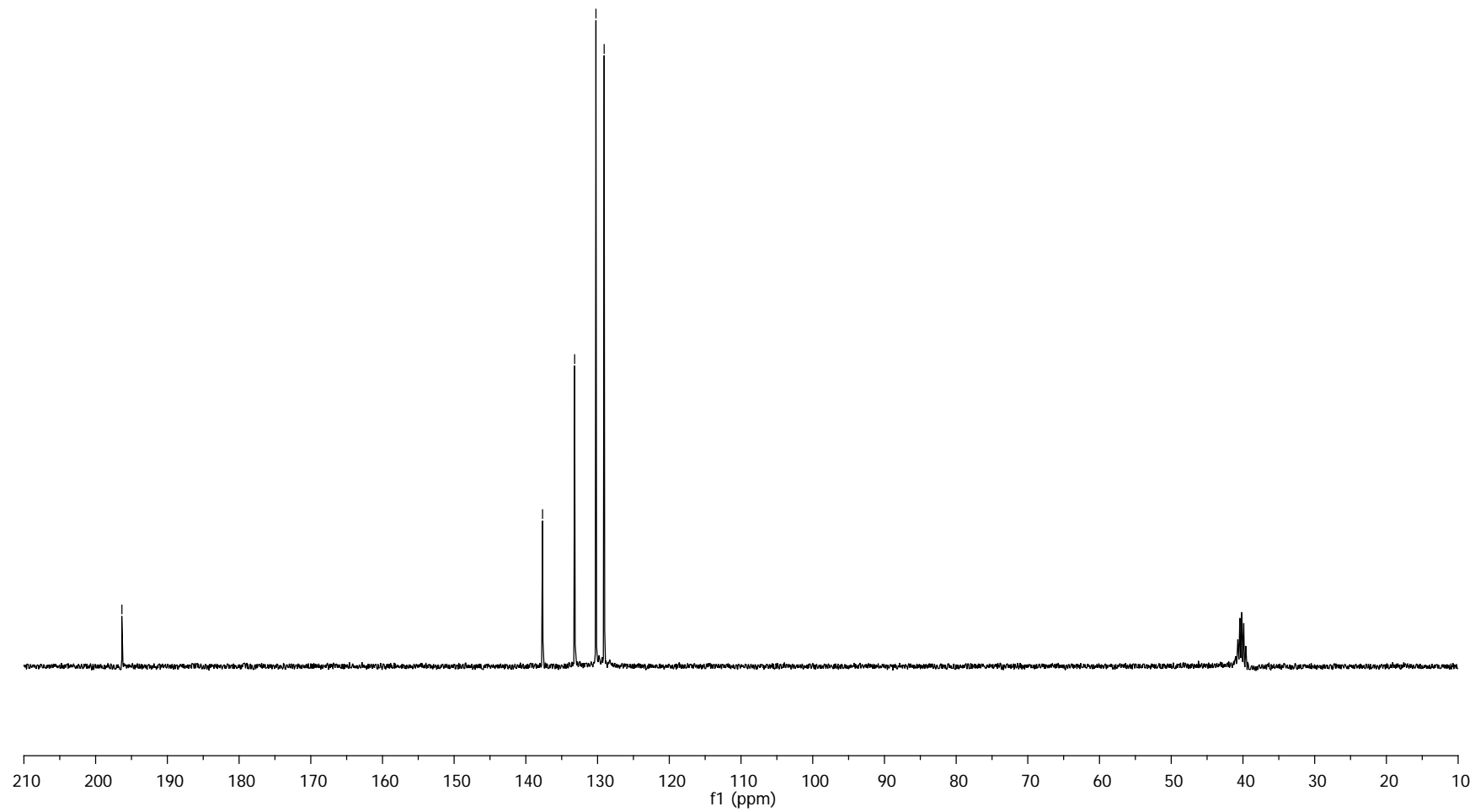




Table 1, Entry 8

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)

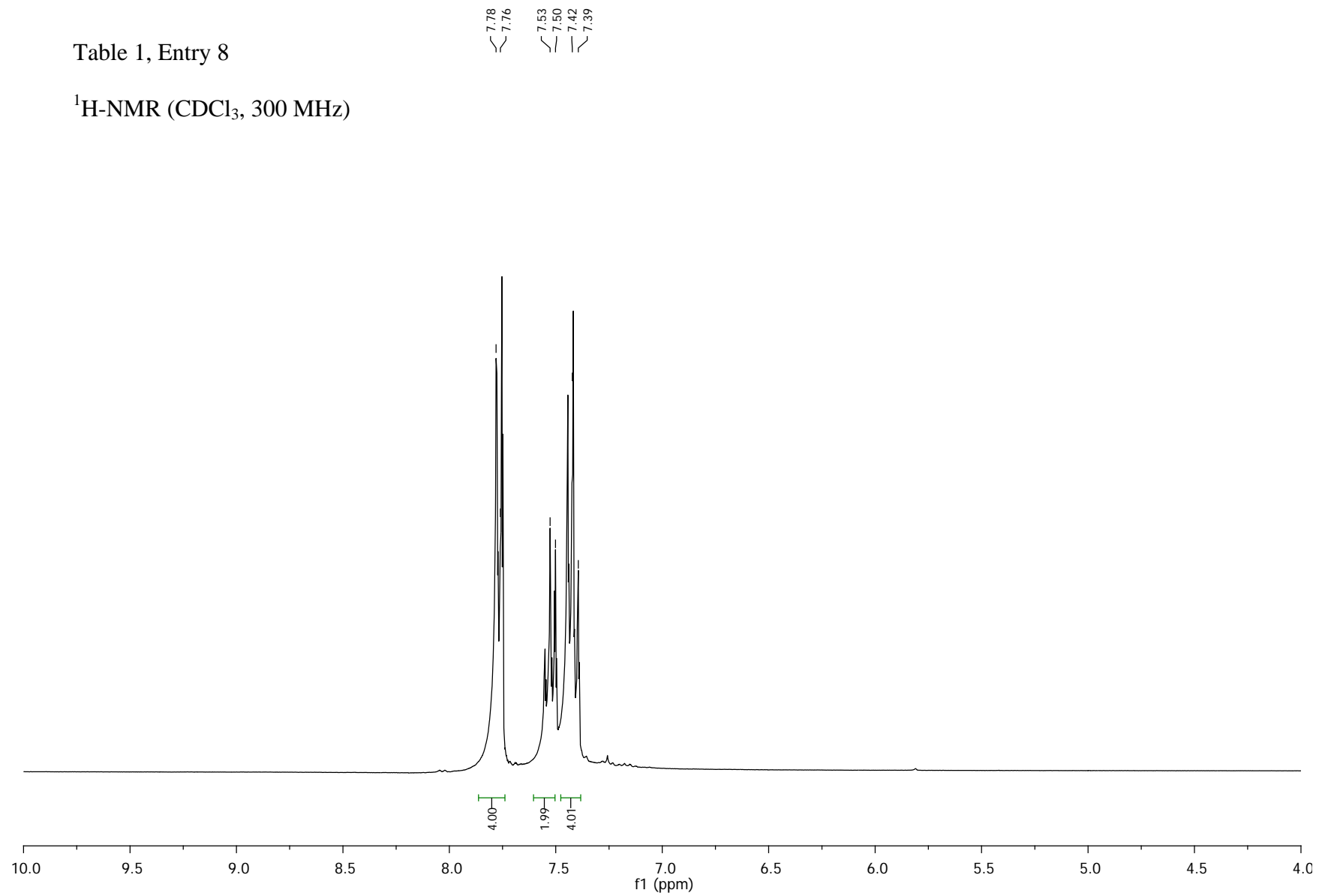
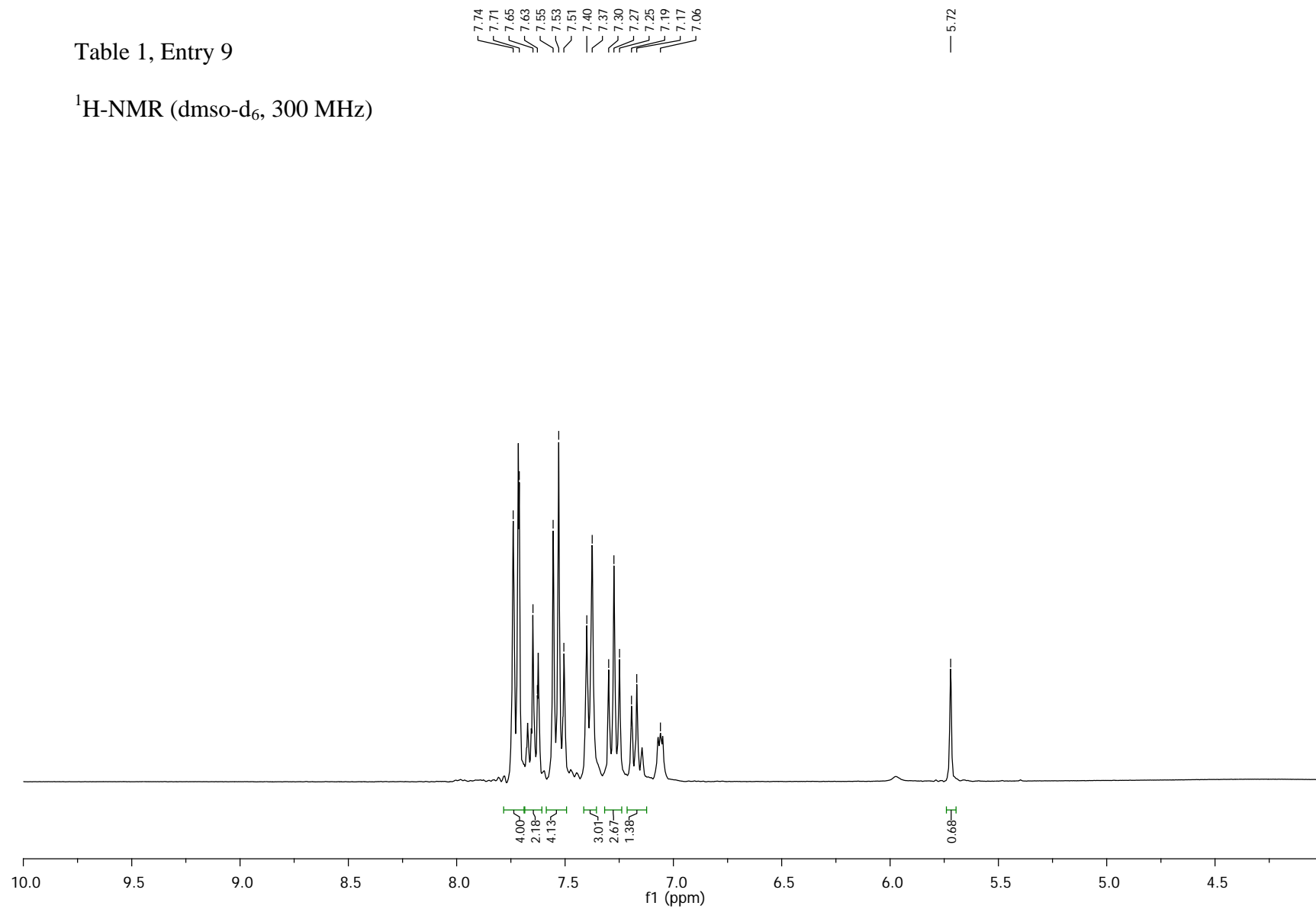


Table 1, Entry 9

$^1\text{H-NMR}$  (dms $o$ -d $_6$ , 300 MHz)



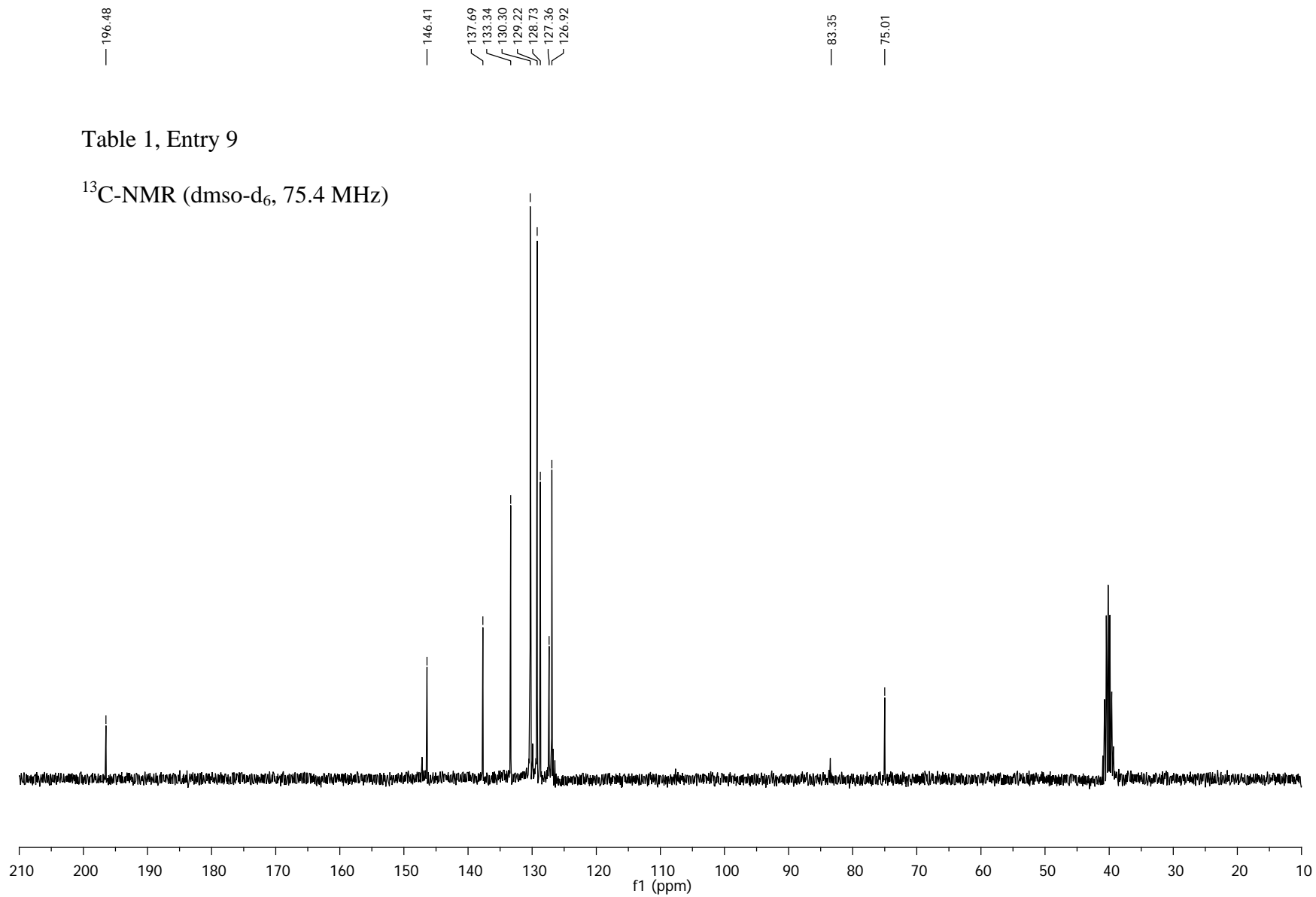
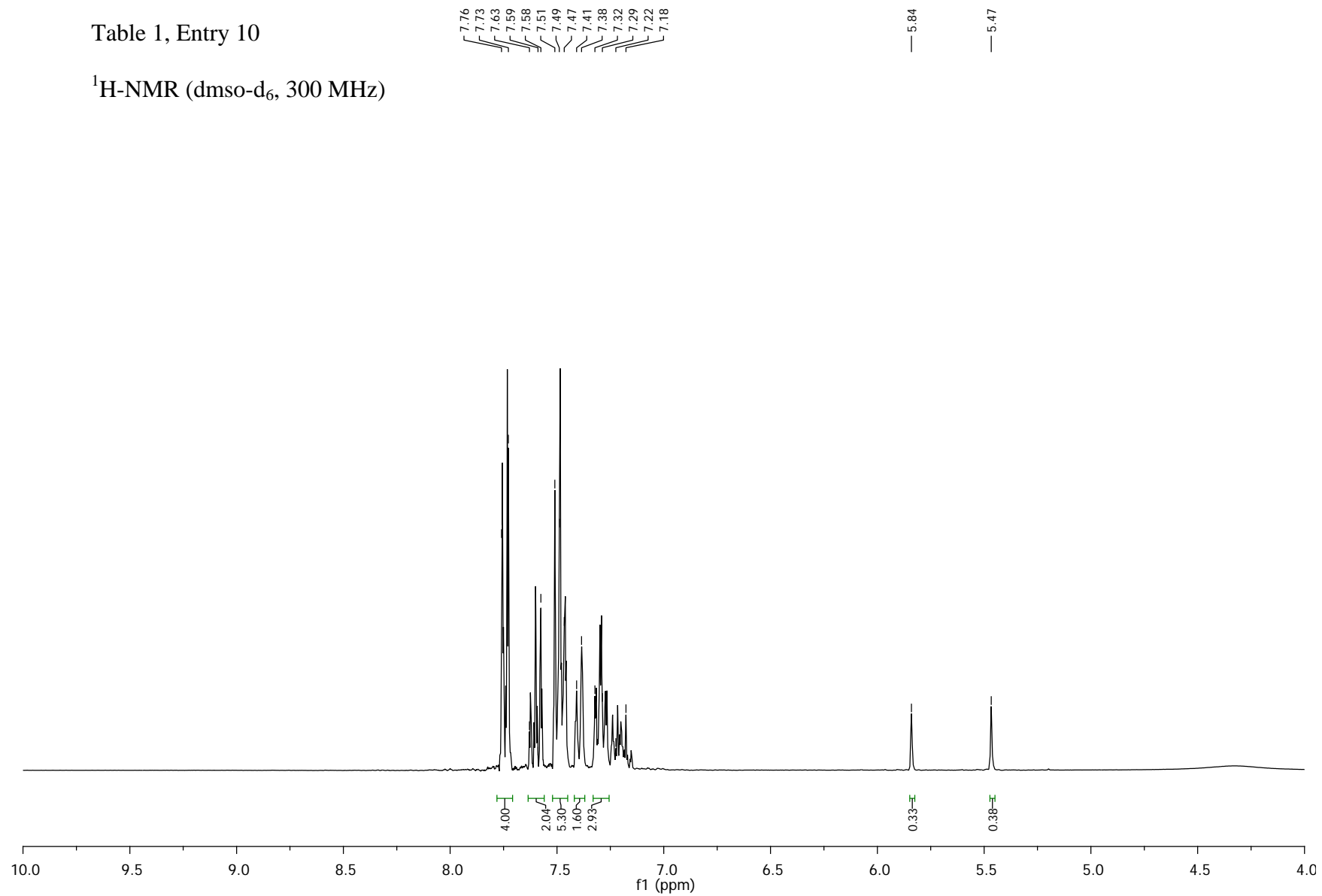


Table 1, Entry 10

$^1\text{H-NMR}$  (dms $o$ -d $_6$ , 300 MHz)



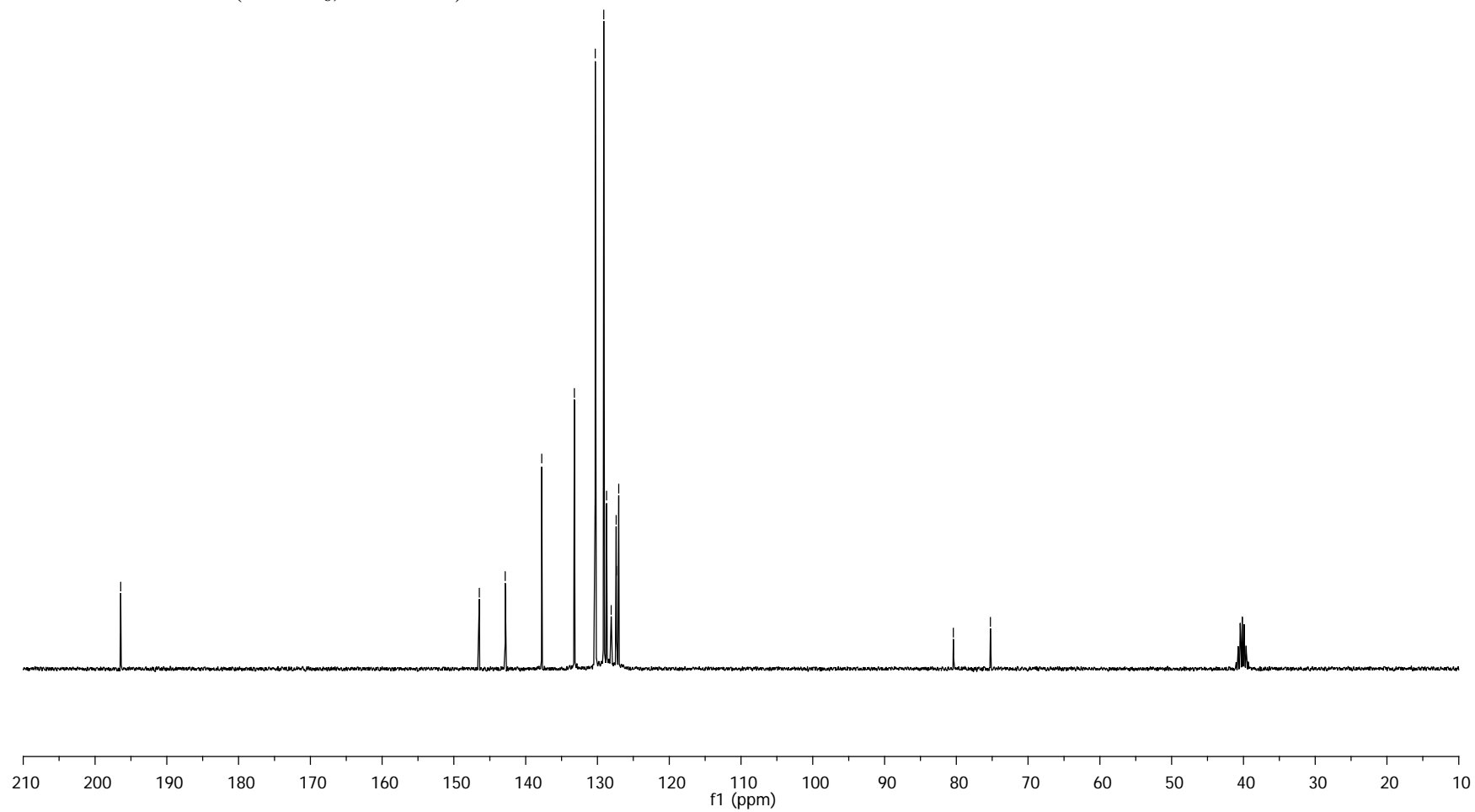
— 196.43

— 146.46  
— 142.84  
— 137.76  
— 133.22  
— 130.31  
— 129.12  
— 128.72  
— 128.08  
— 127.39  
— 127.36  
— 127.04

— 80.41  
— 75.24

Table 1, Entry 10

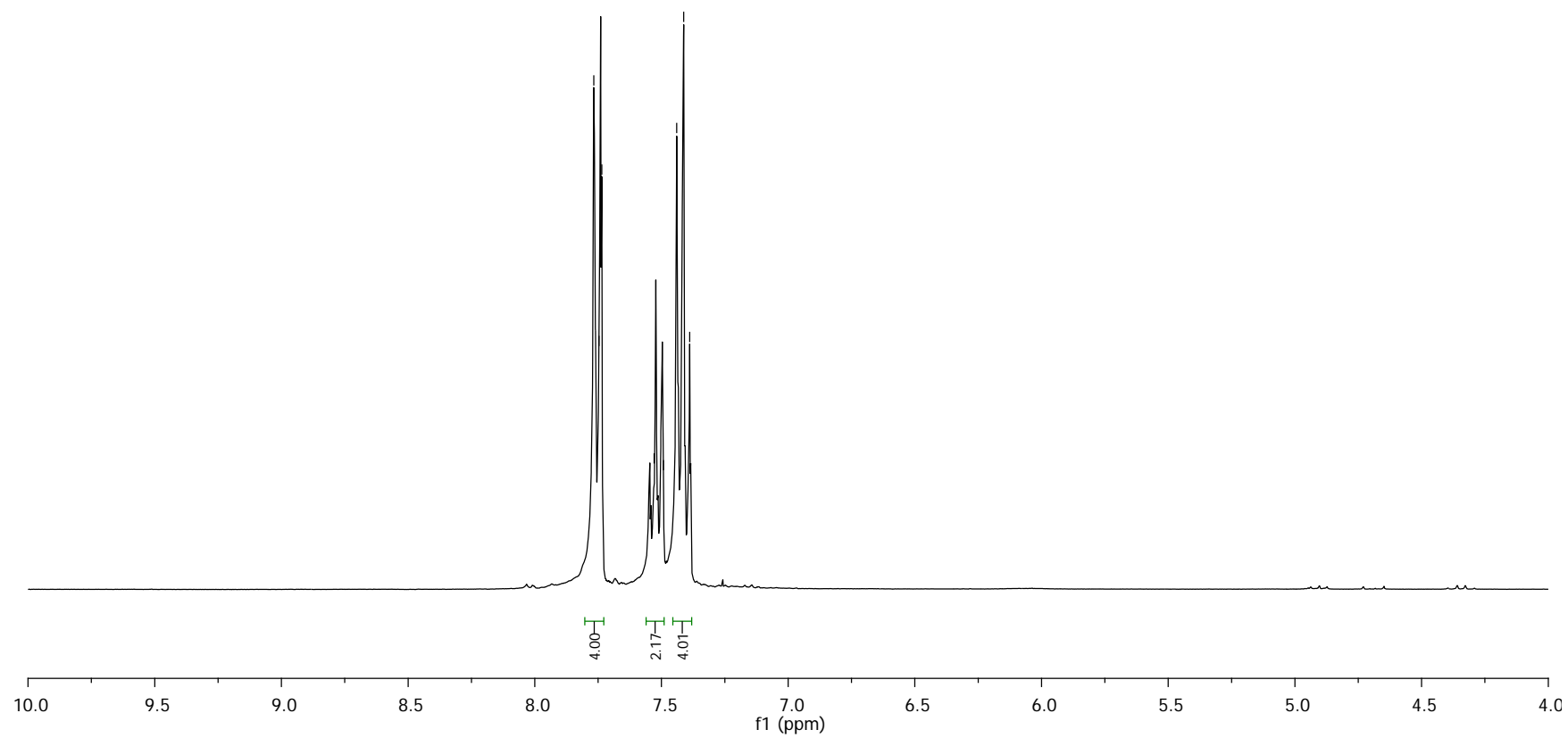
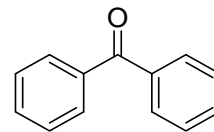
$^{13}\text{C}$ -NMR (dms $o$ -d $_6$ , 75.4 MHz)

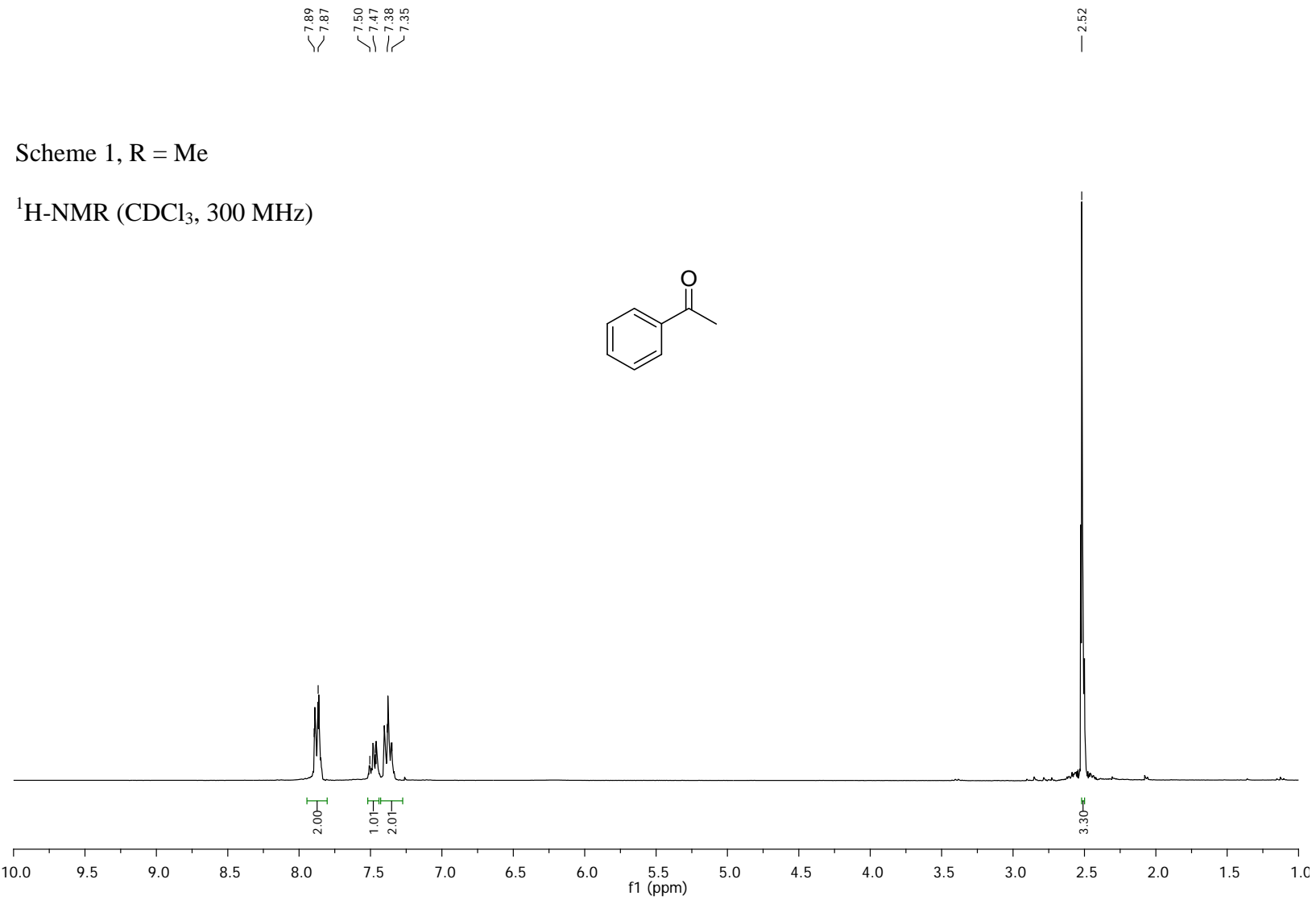


Scheme 1, R = Ph

7.77  
7.75  
7.74  
7.53  
7.49  
7.44  
7.41  
7.39

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)





— 9.93

7.82  
7.79  
7.56  
7.48  
7.45  
7.43

Scheme 1, R = H

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)

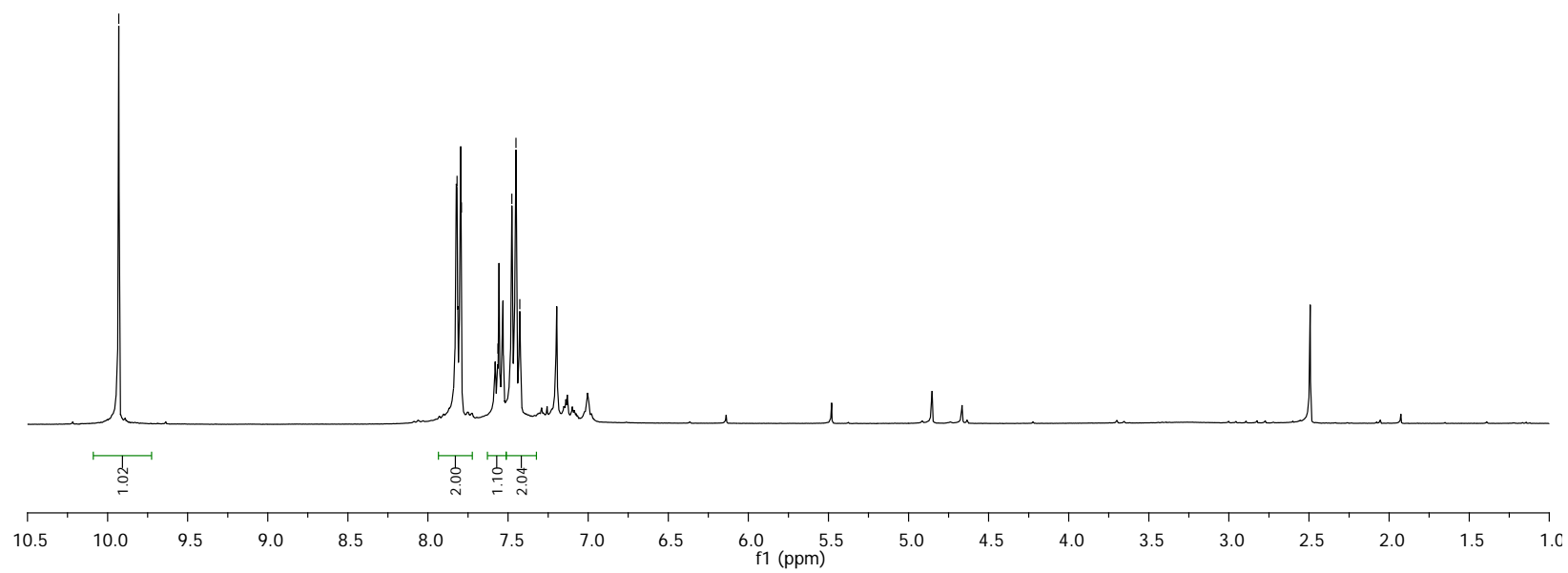
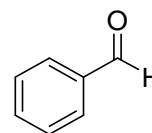
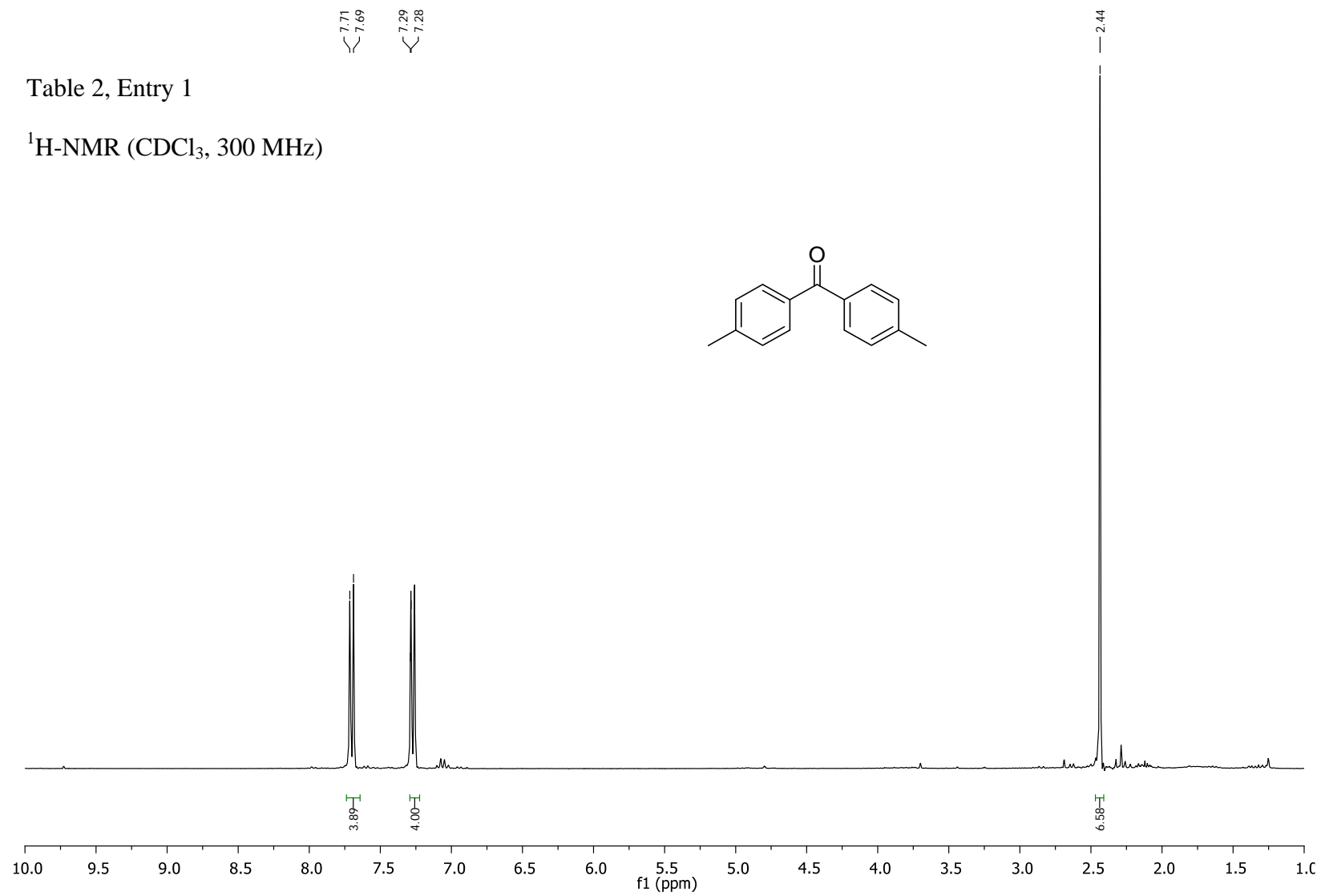




Table 2, Entry 1

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)



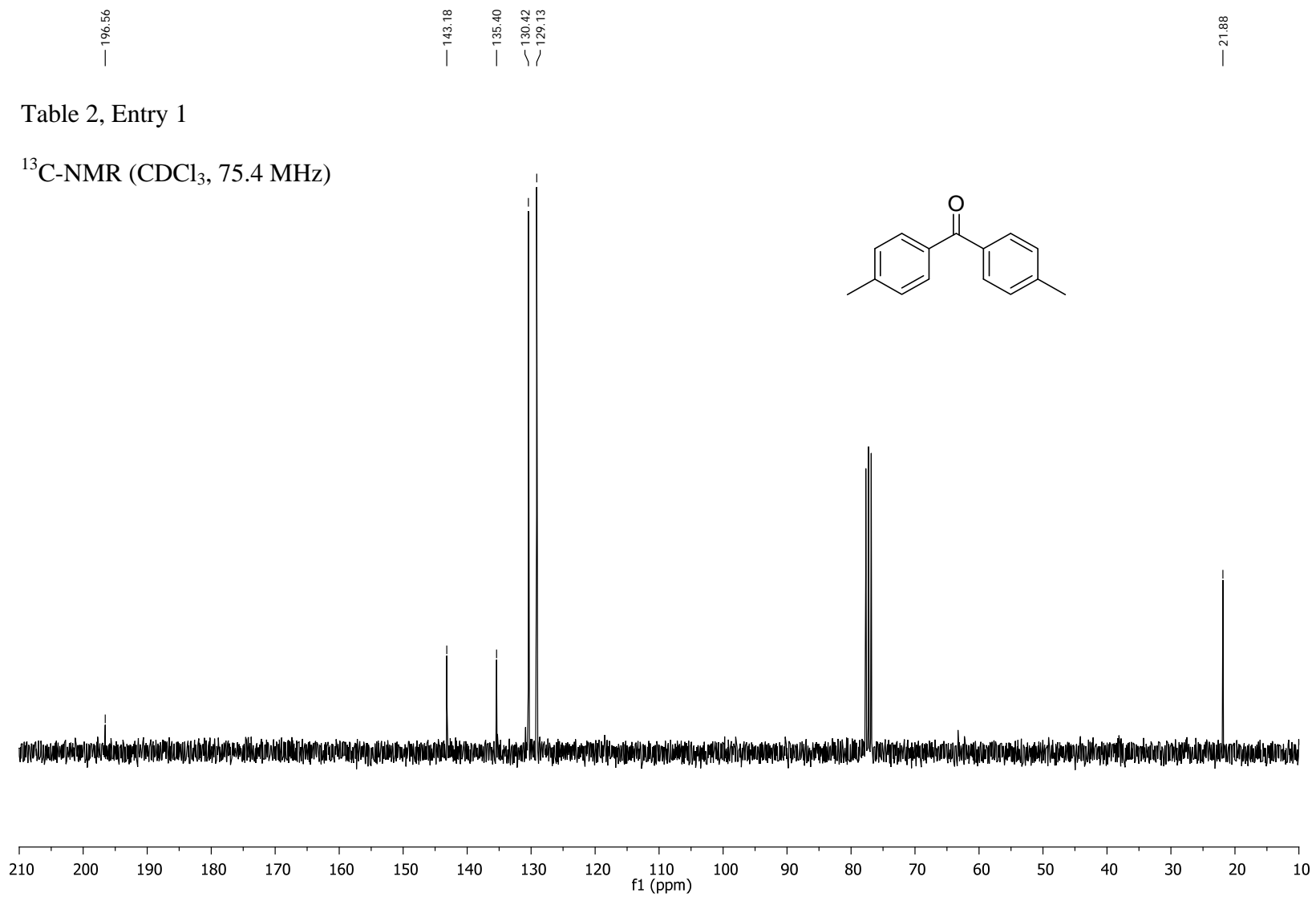
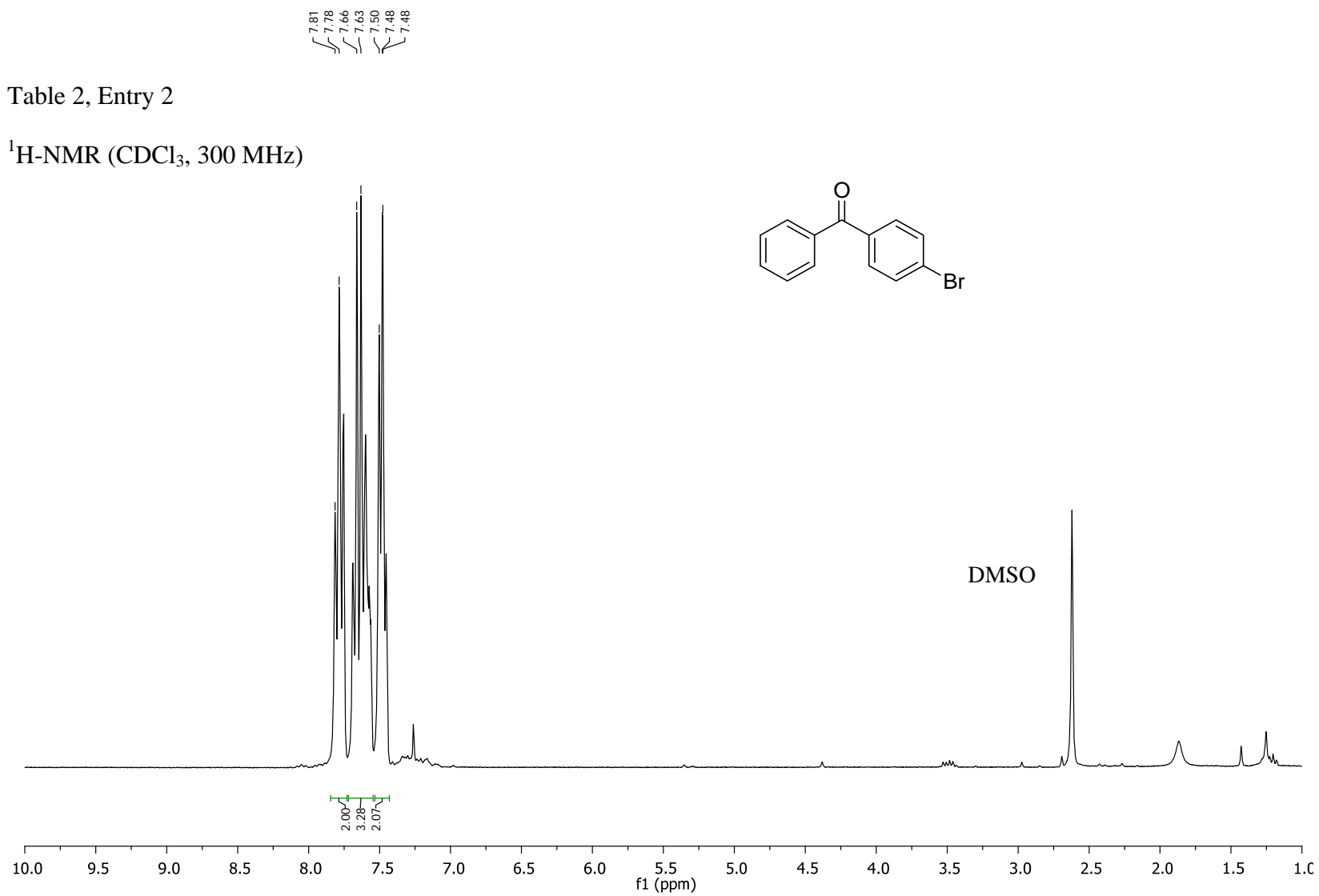


Table 2, Entry 2

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)



— 195.87

137.79  
137.36  
136.51  
132.93  
132.66  
131.84  
131.80  
130.29  
130.17  
128.65  
128.51  
127.75

Table 2, Entry 2

$^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 75.4 MHz)

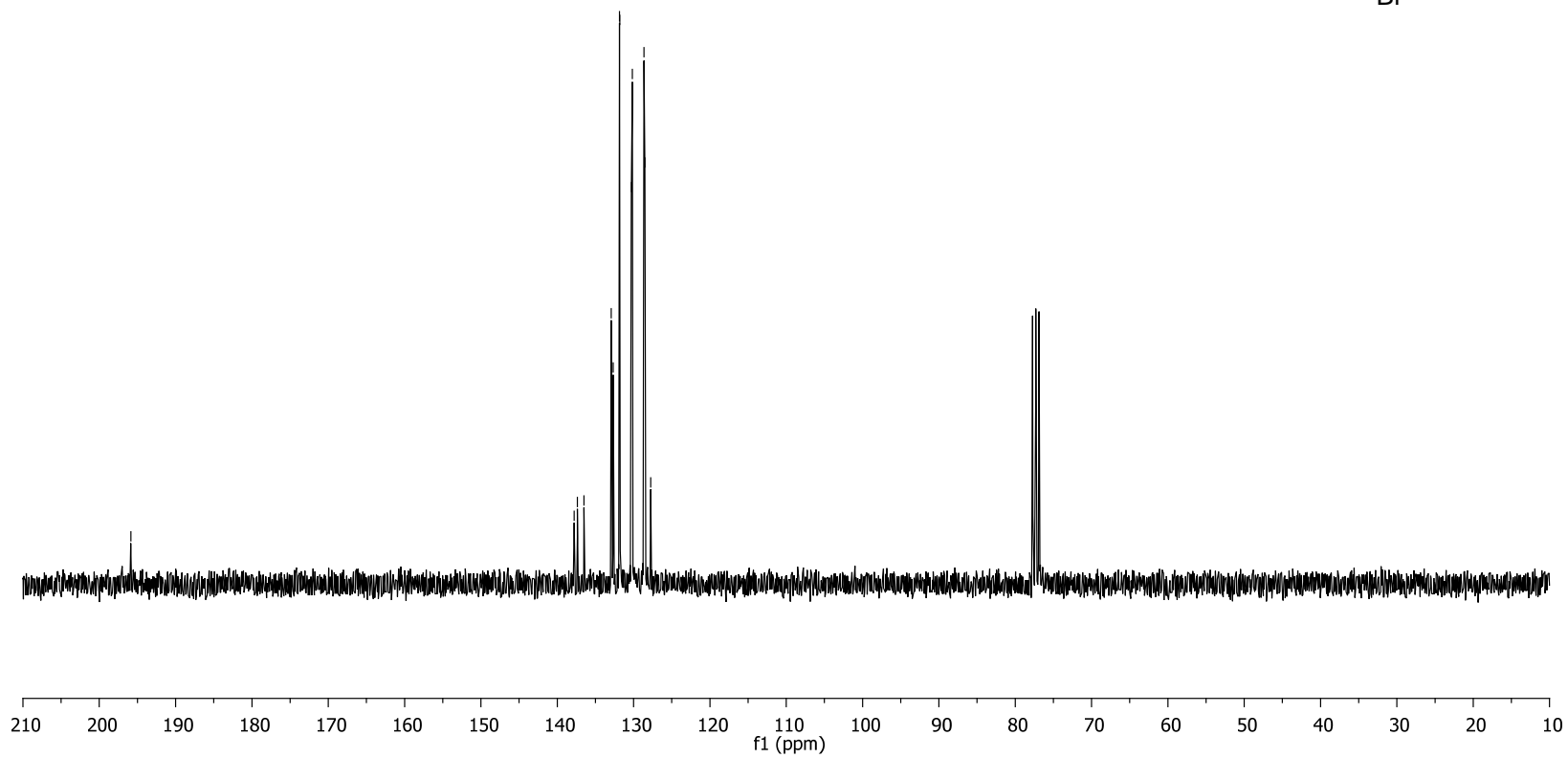
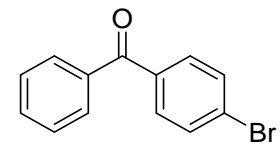
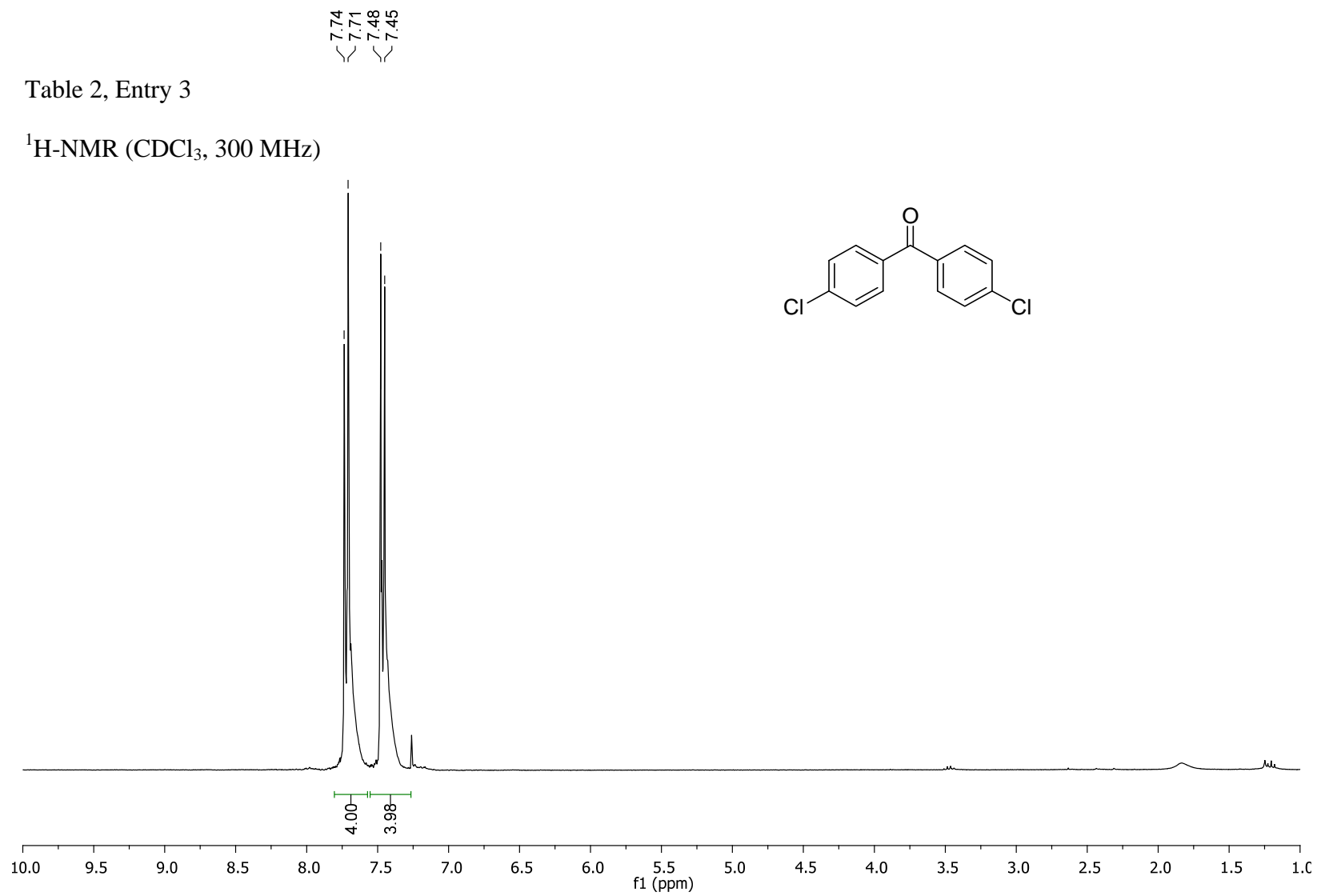


Table 2, Entry 3

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)



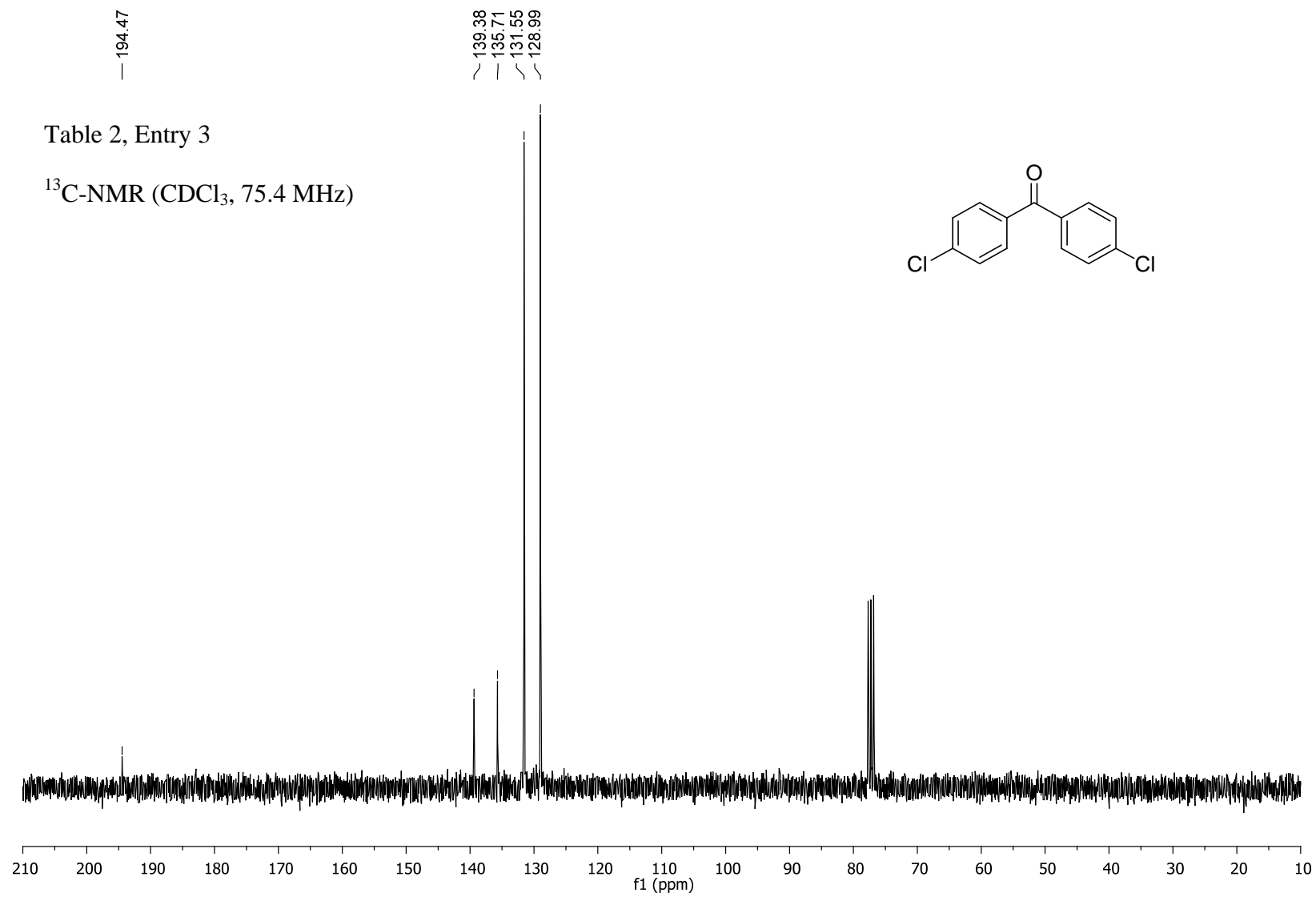
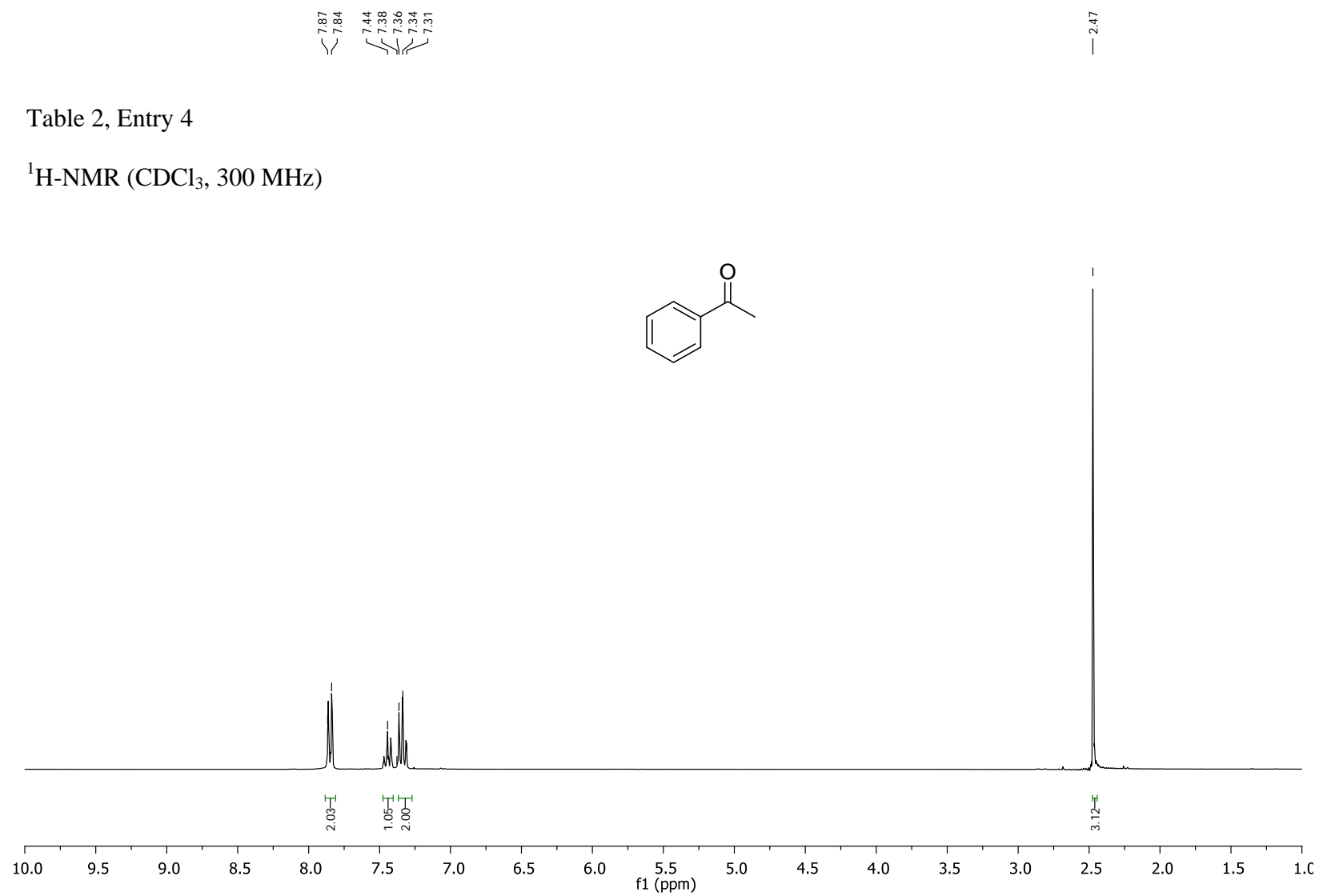
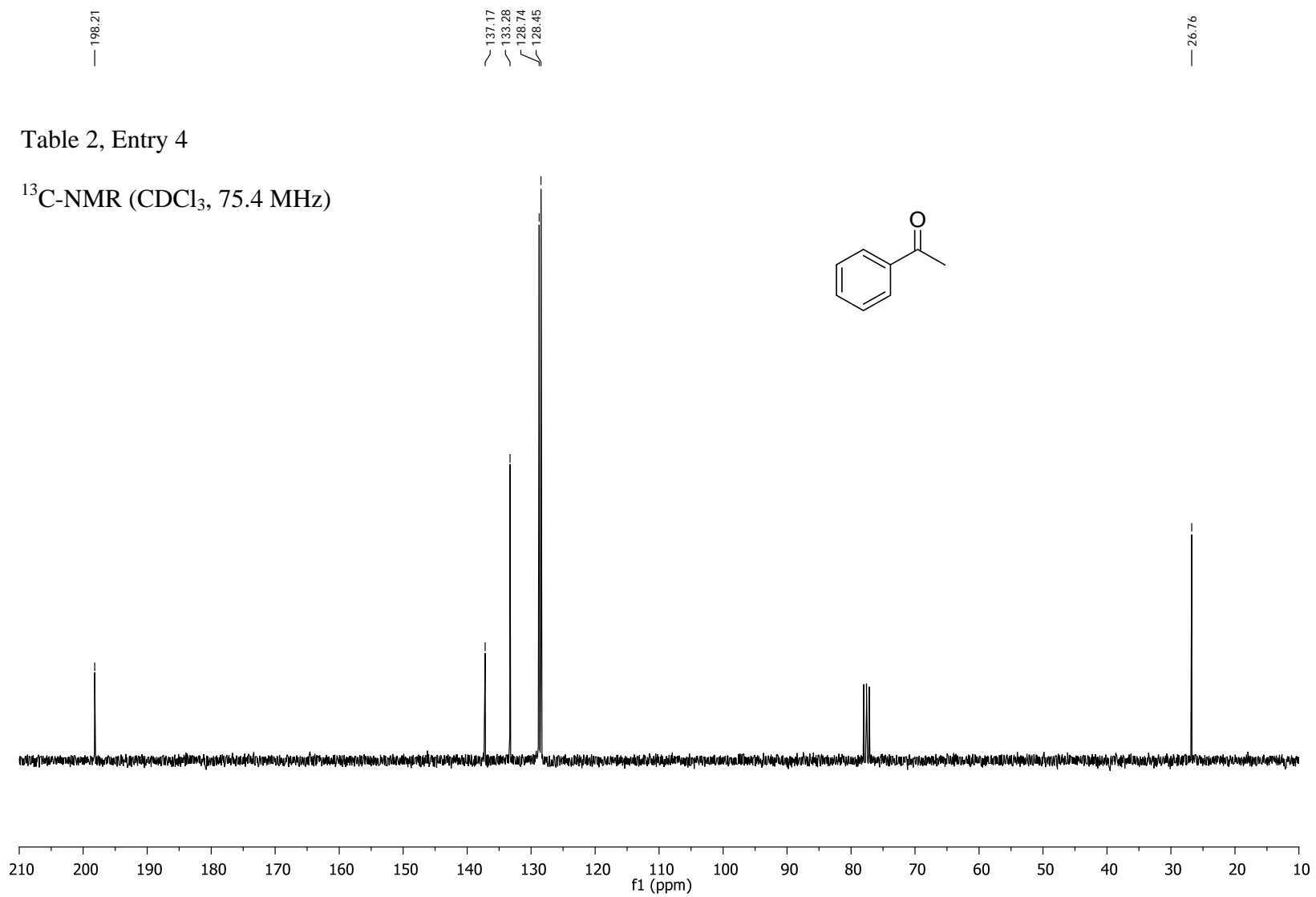


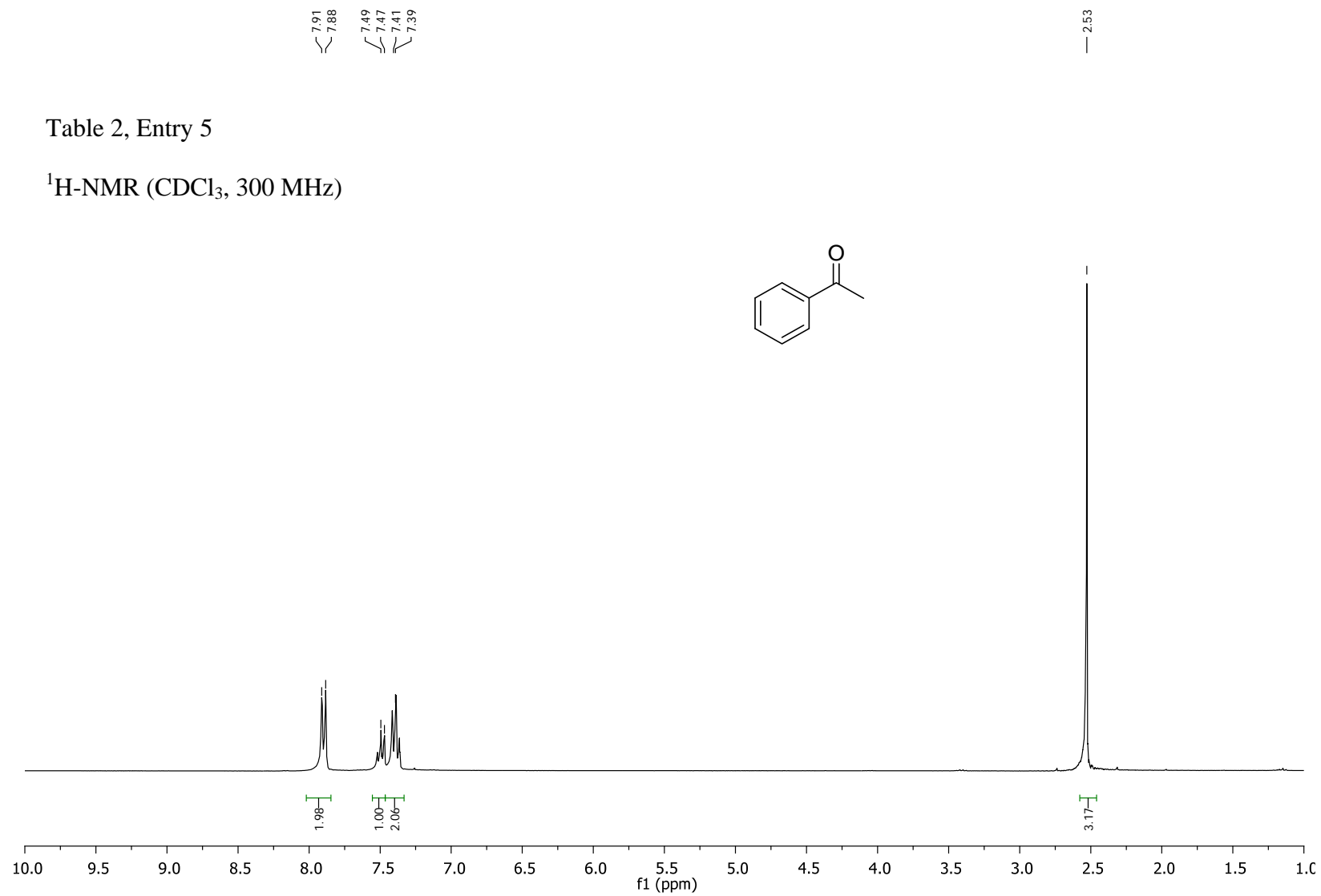
Table 2, Entry 4

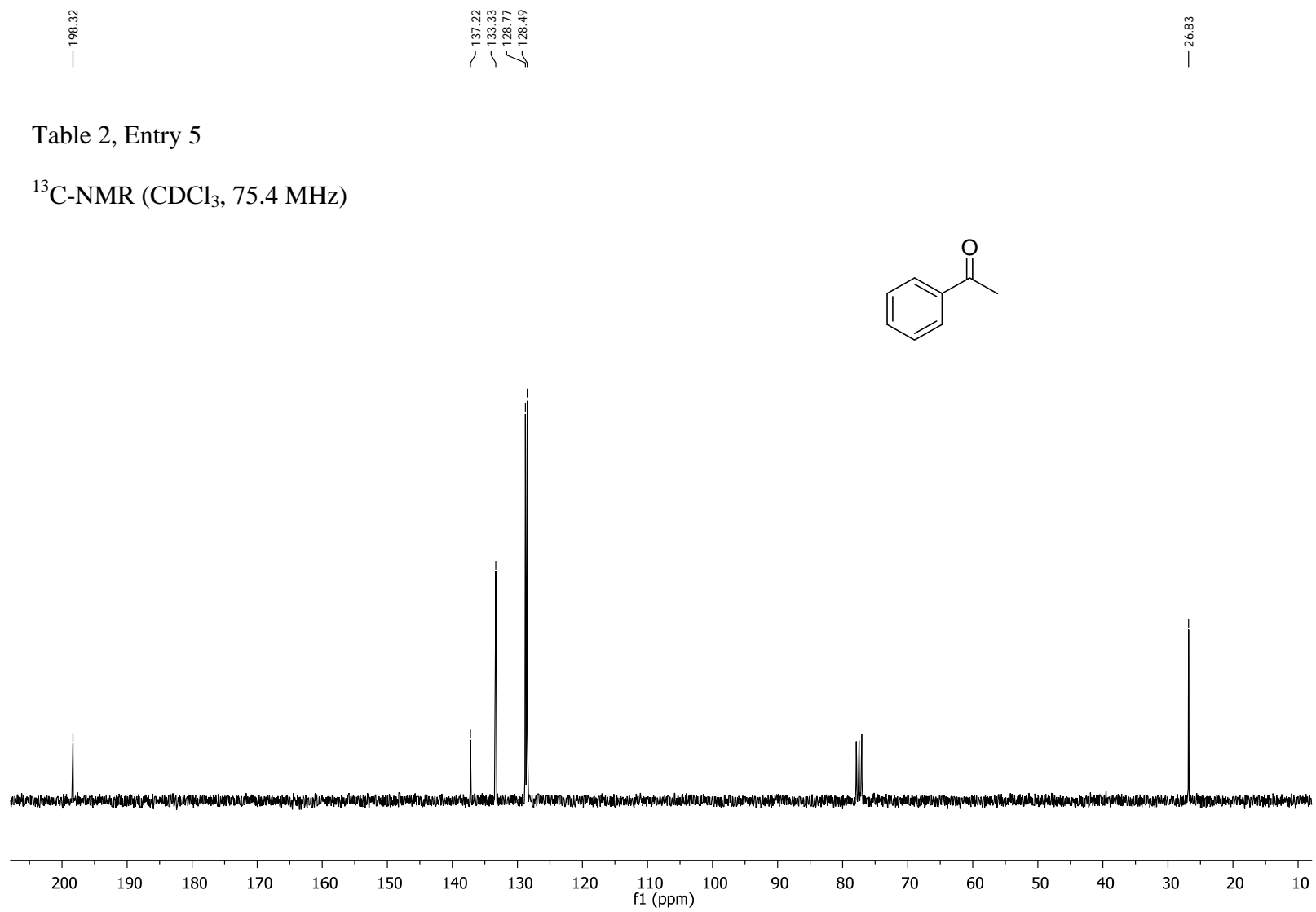
$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)

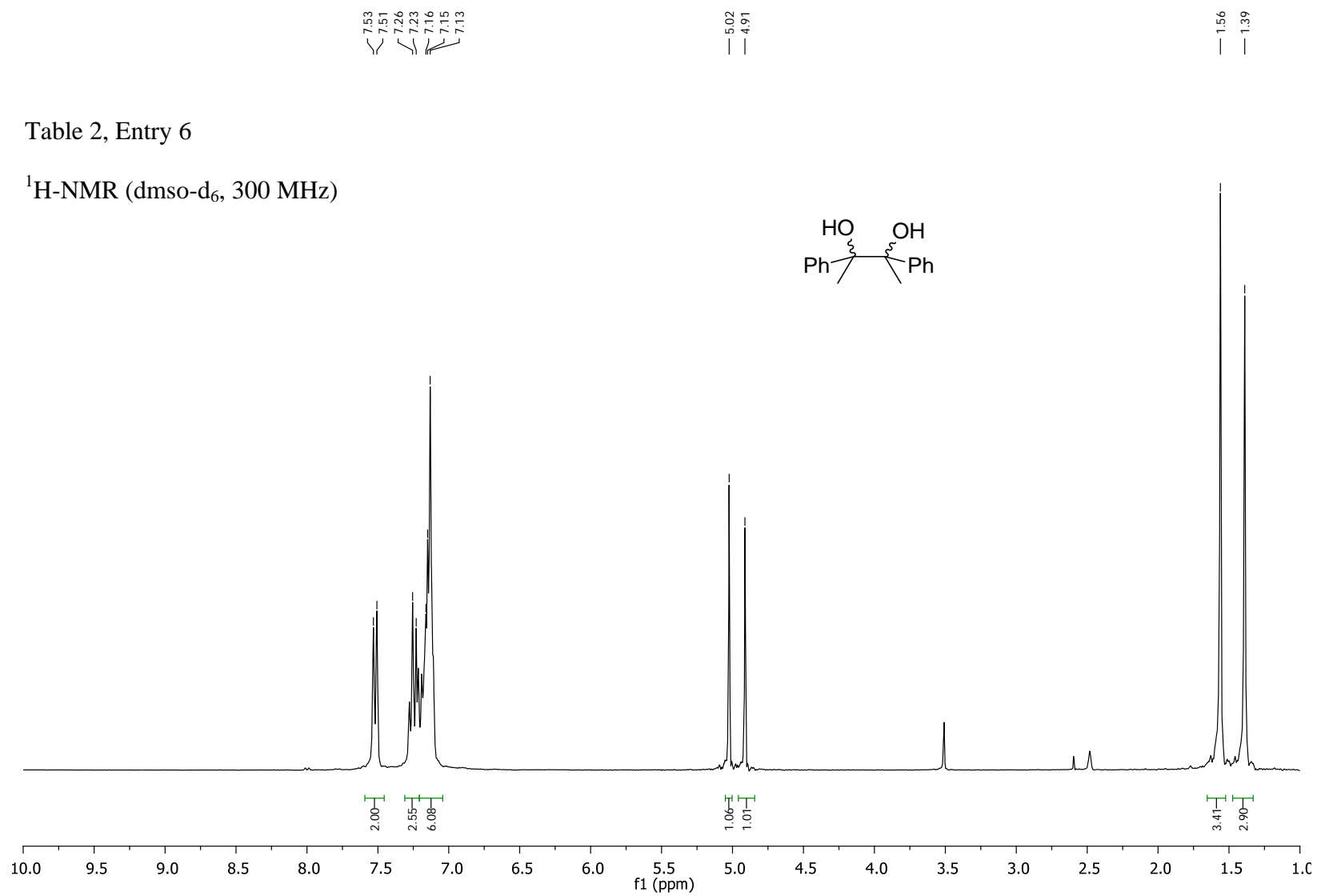


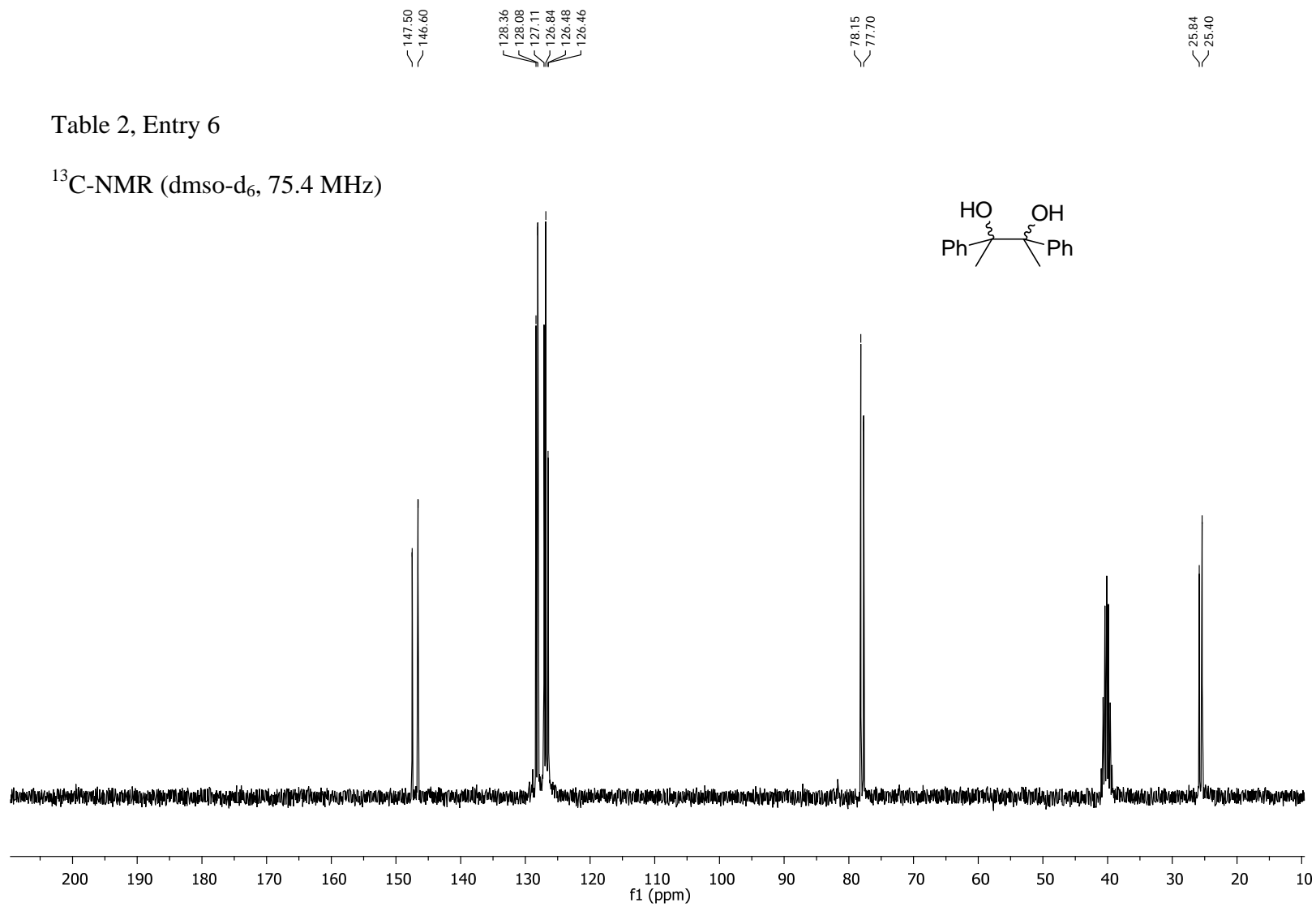


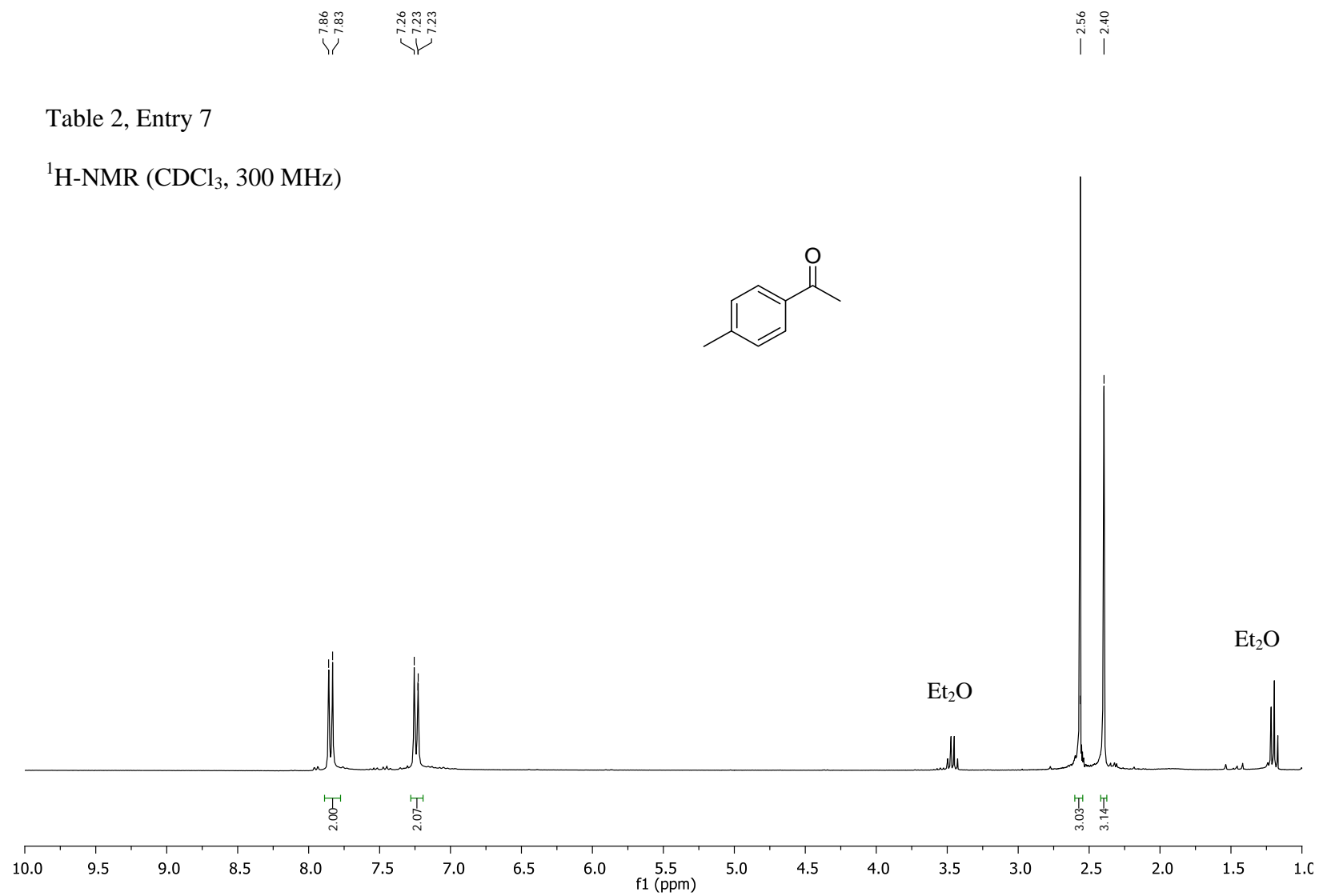












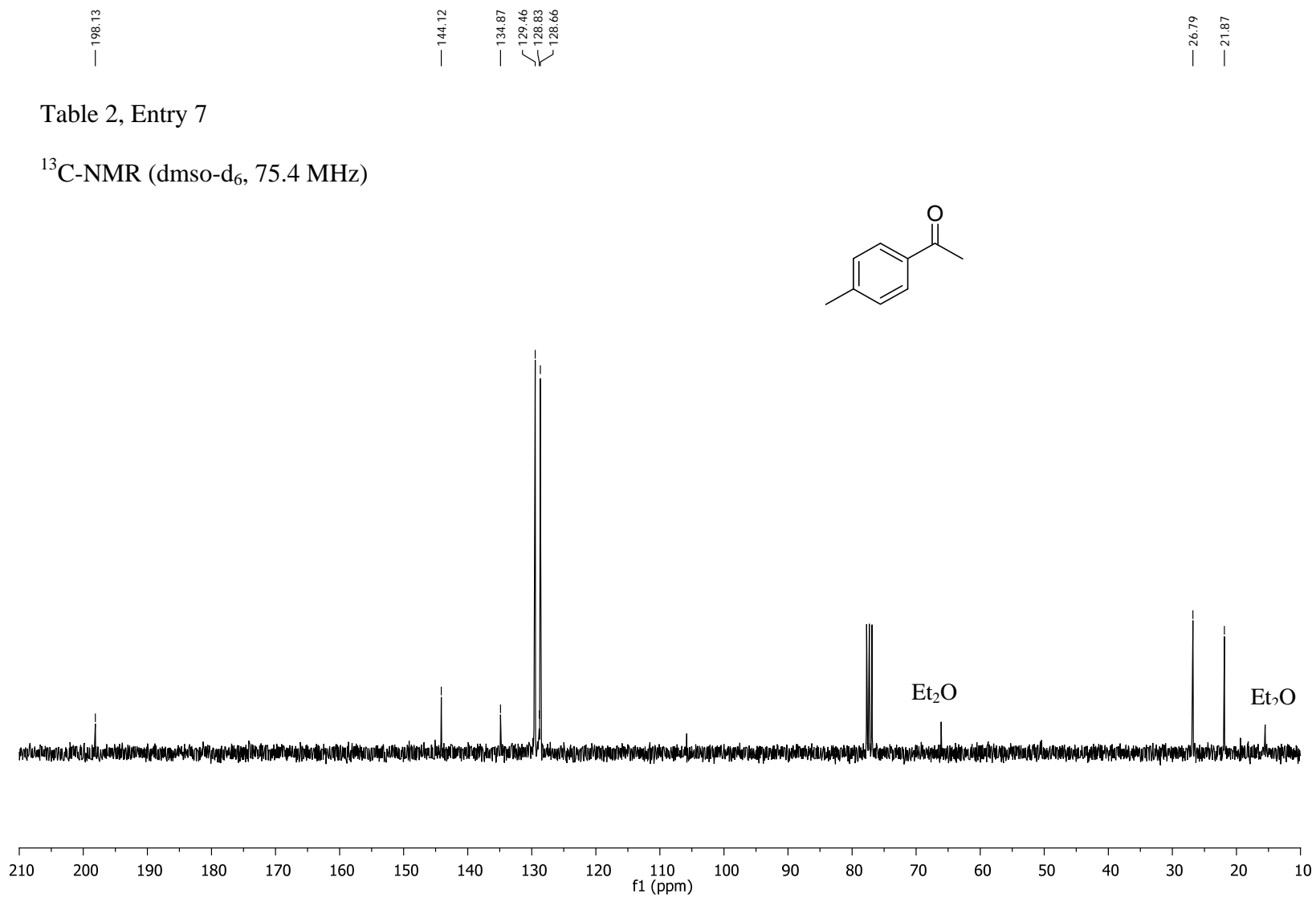
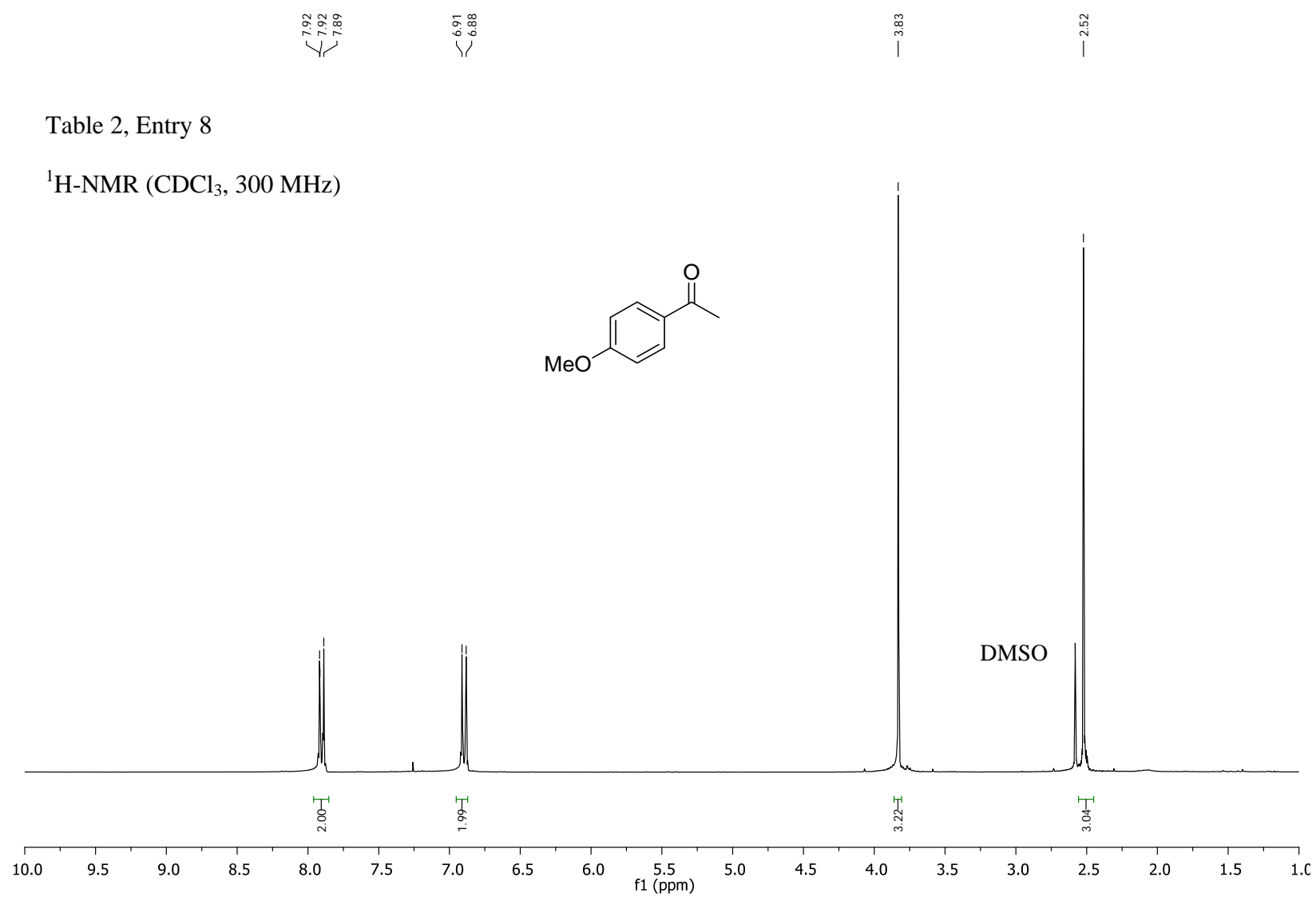
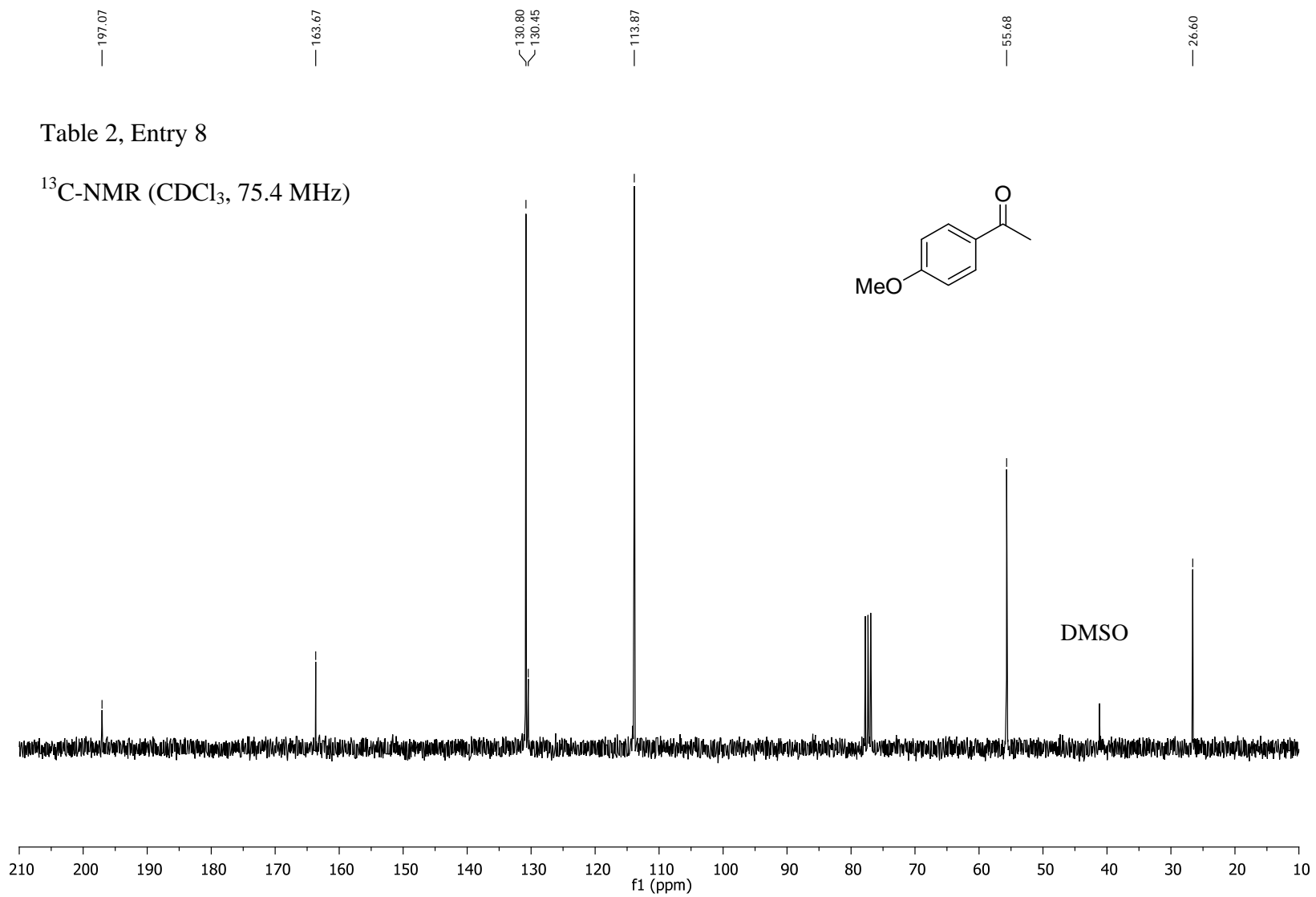


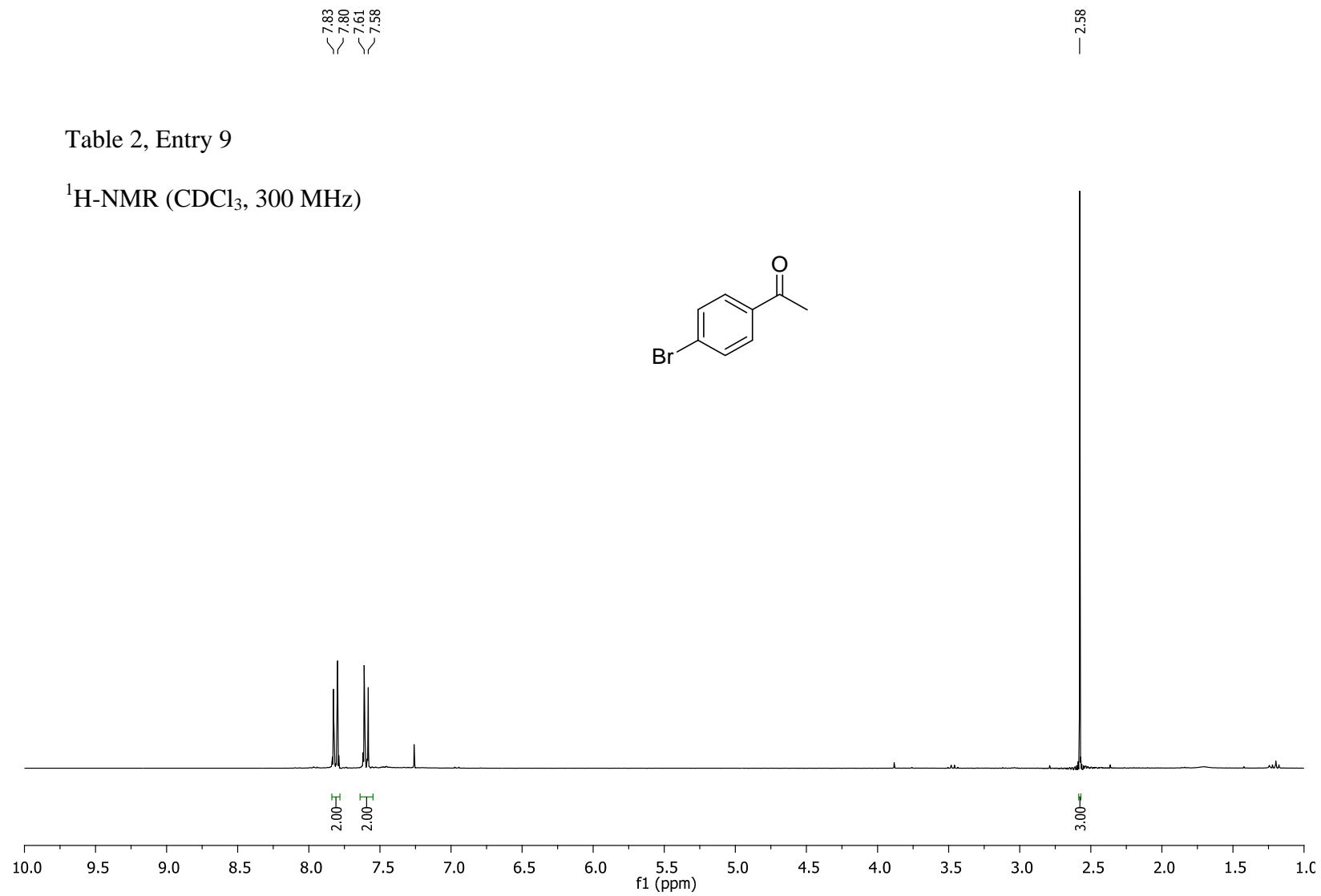
Table 2, Entry 8

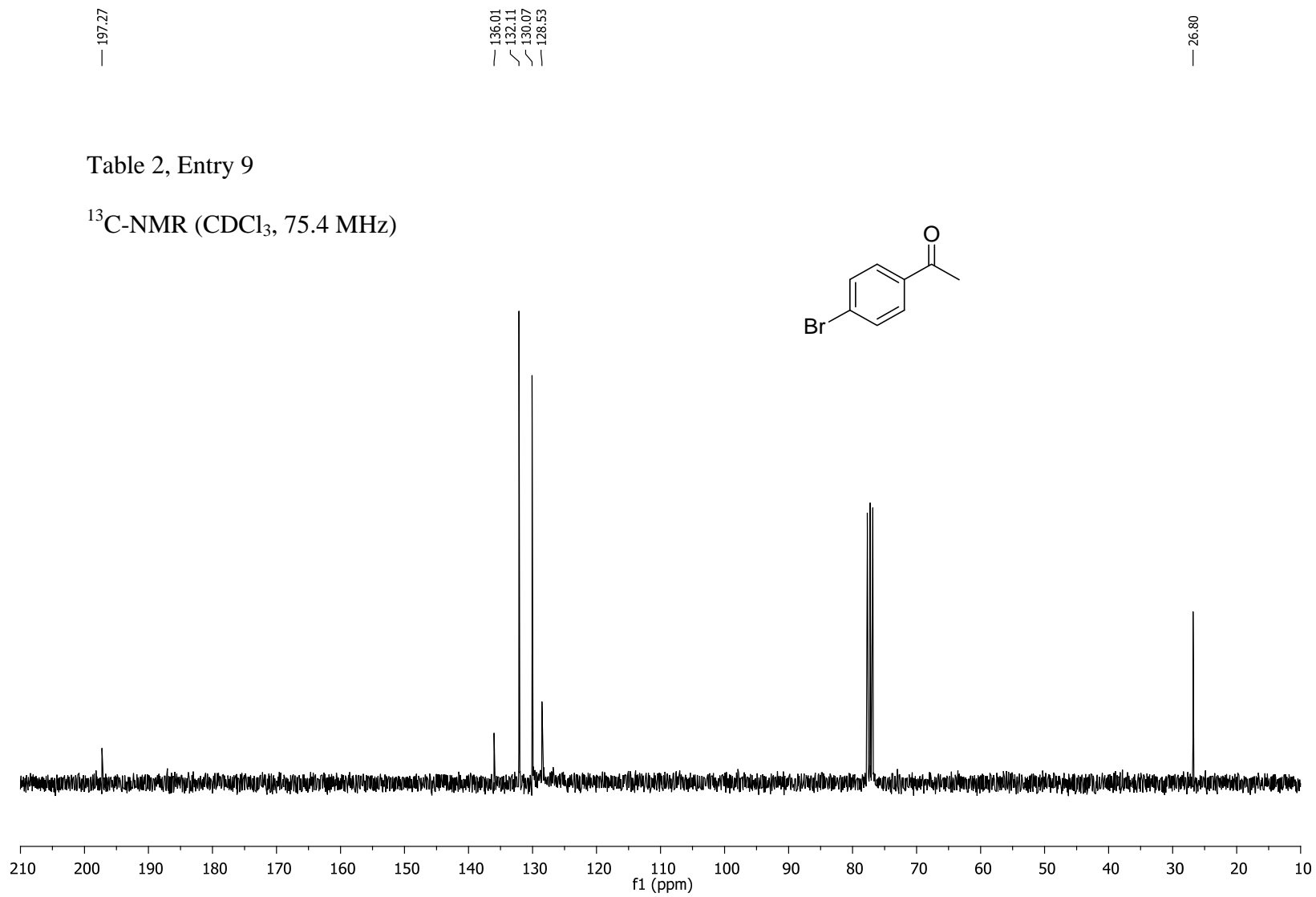
$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)

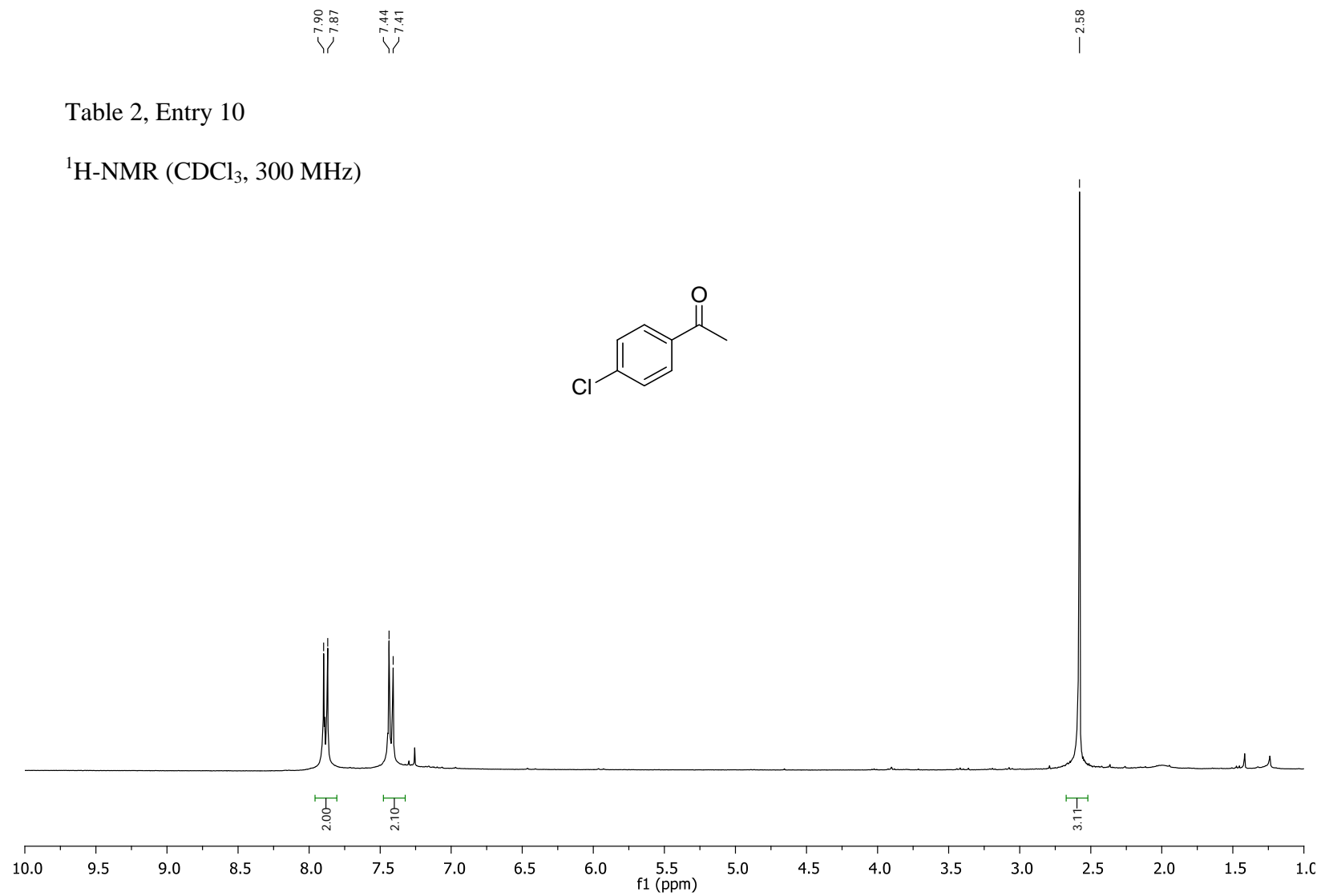












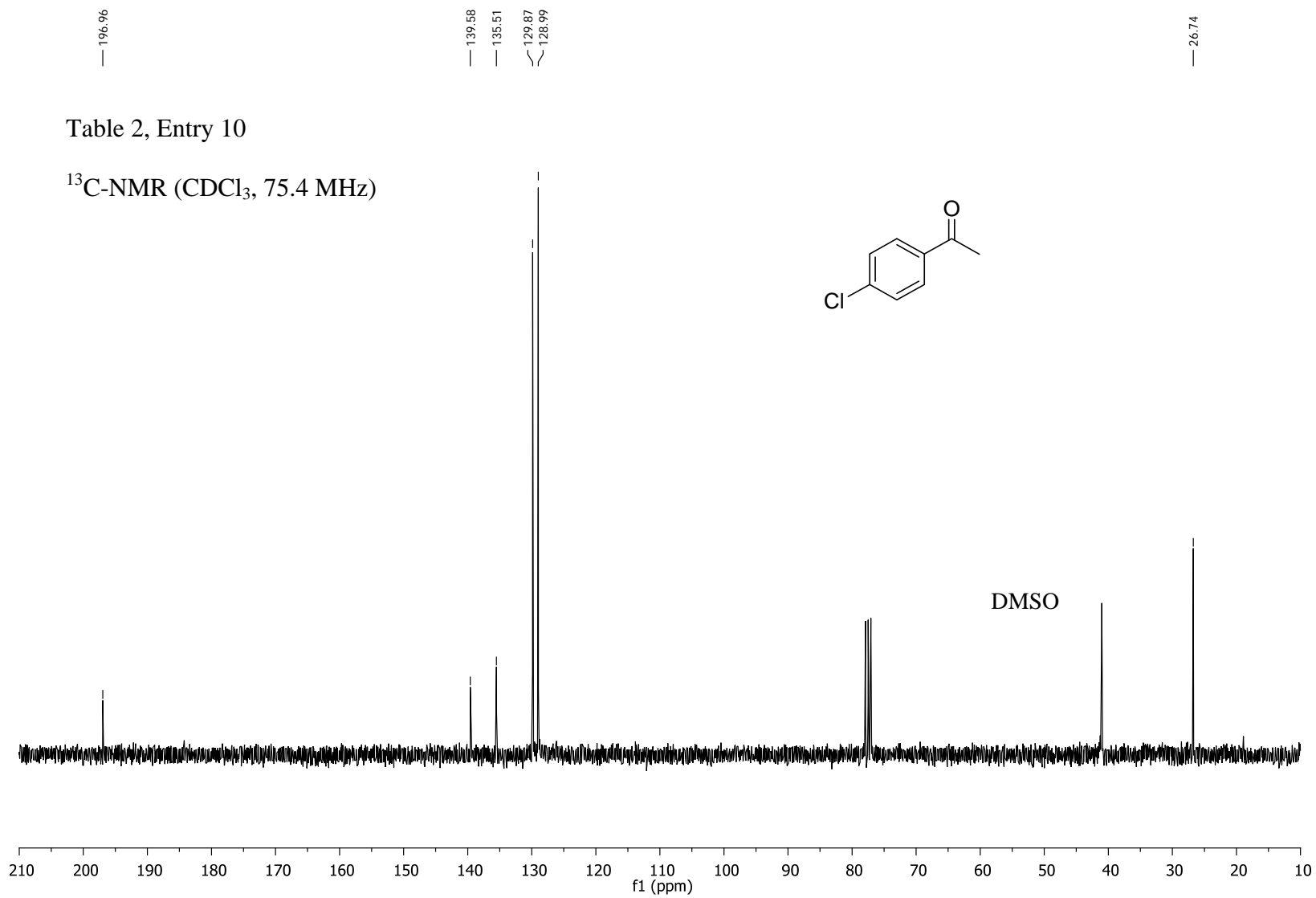
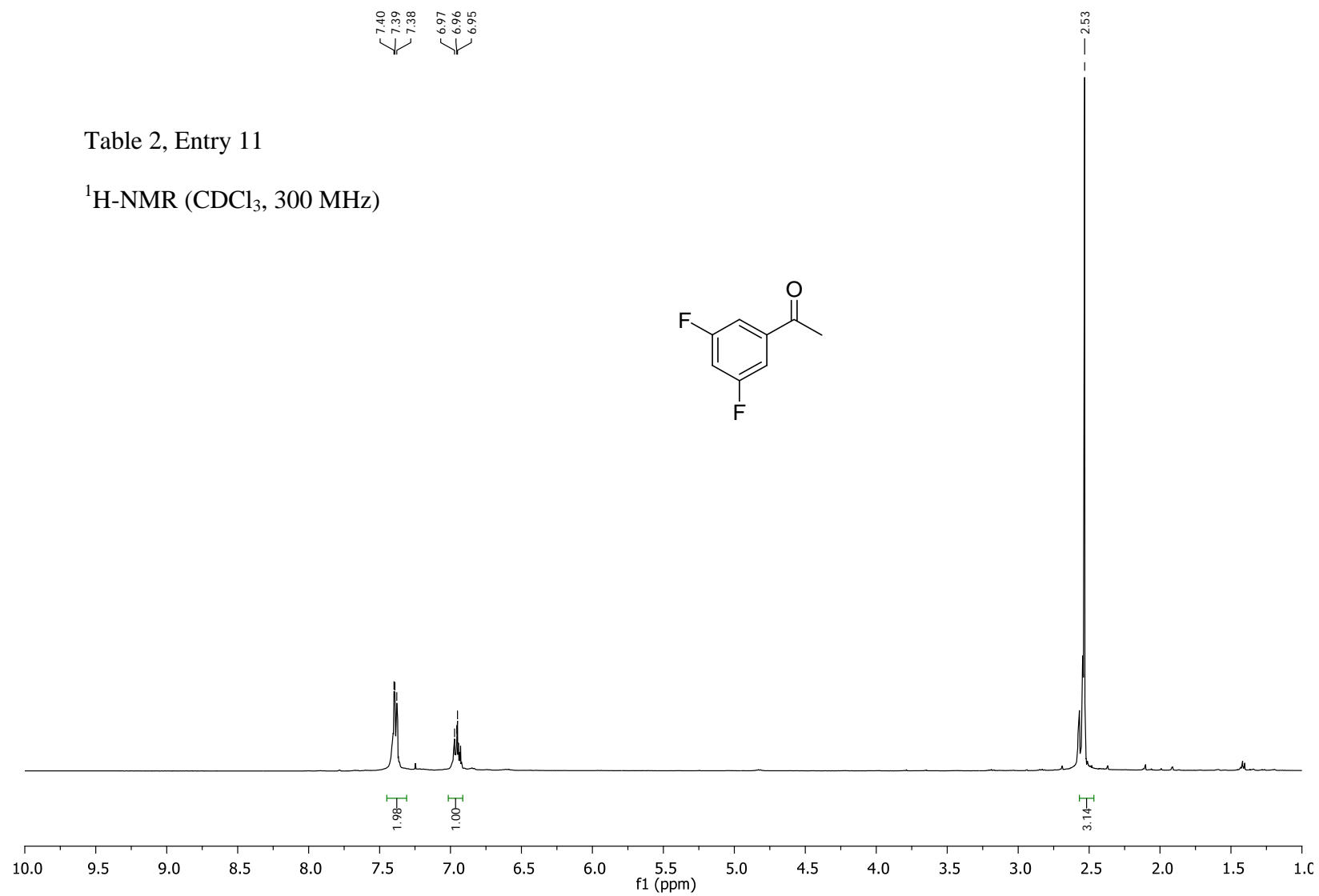


Table 2, Entry 11

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)



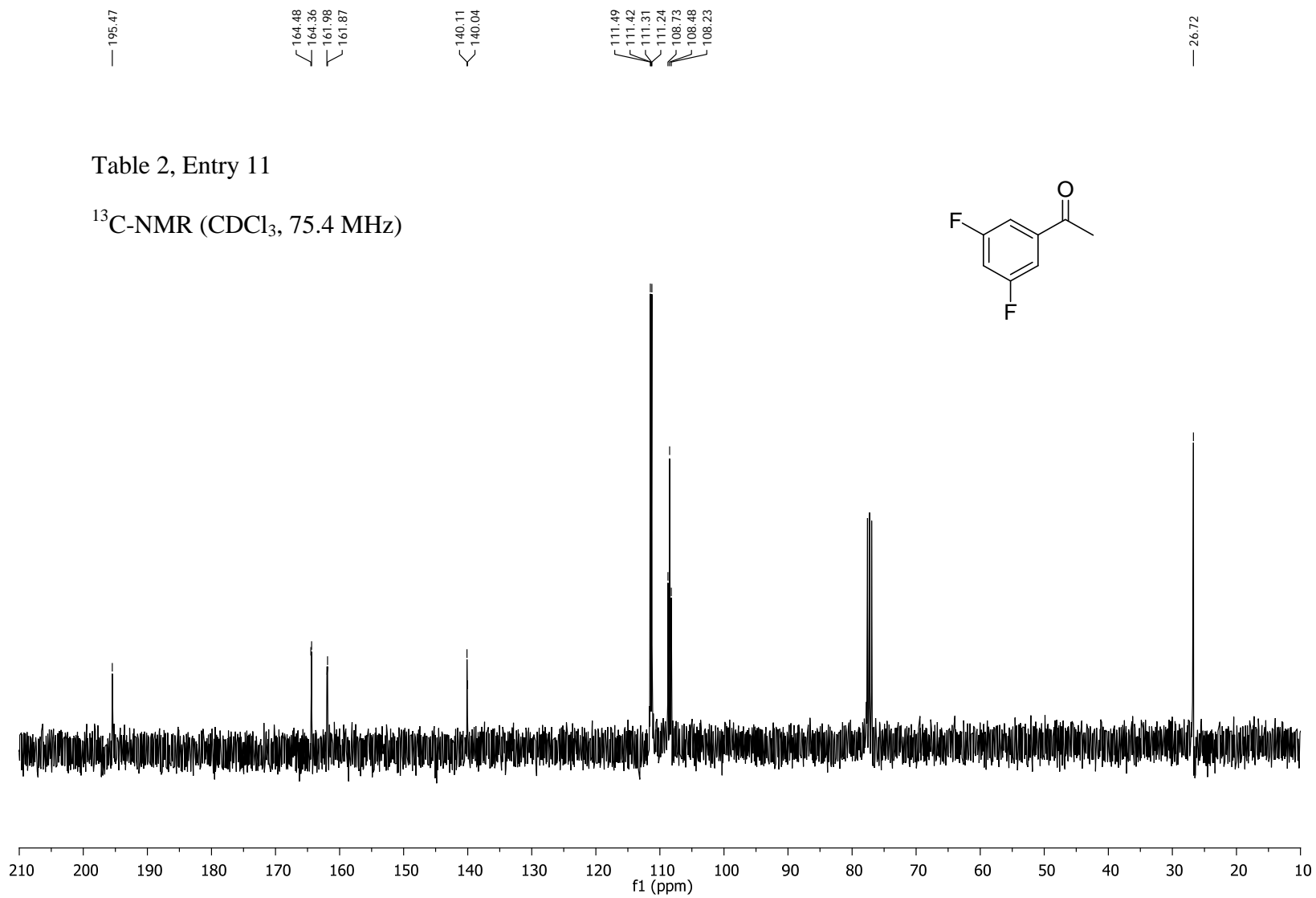
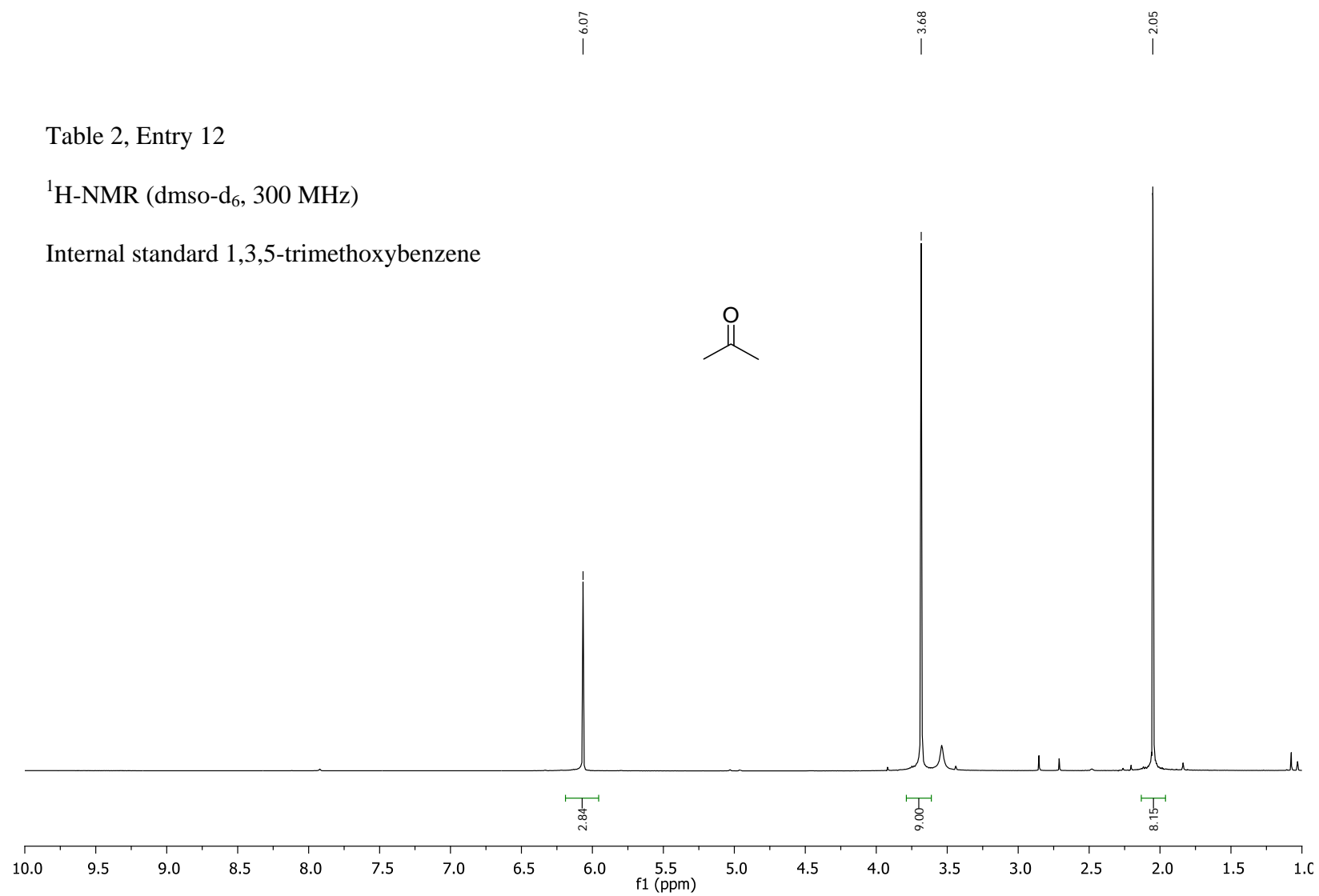


Table 2, Entry 12

$^1\text{H-NMR}$  (dms $o$ -d $_6$ , 300 MHz)

Internal standard 1,3,5-trimethoxybenzene



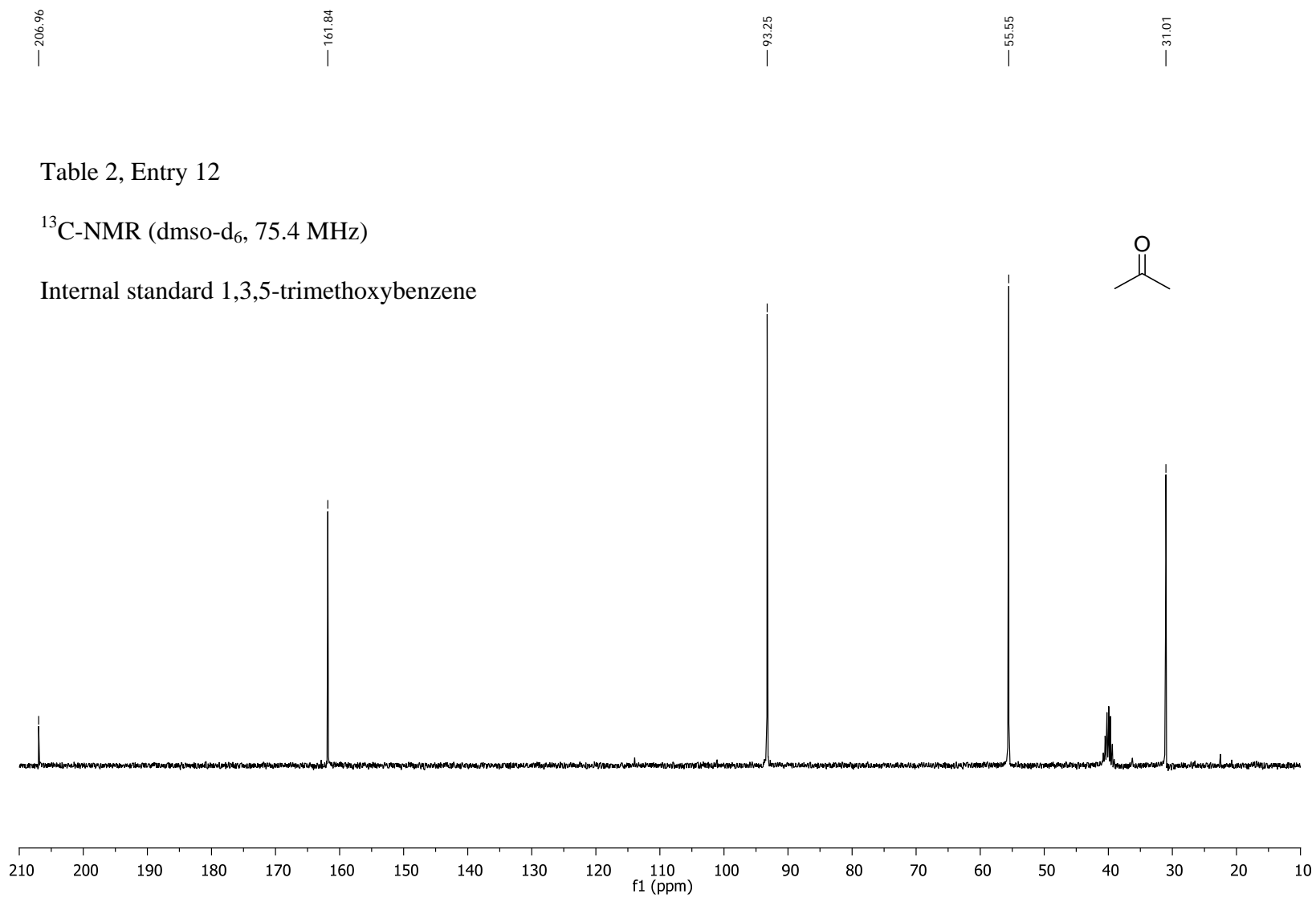
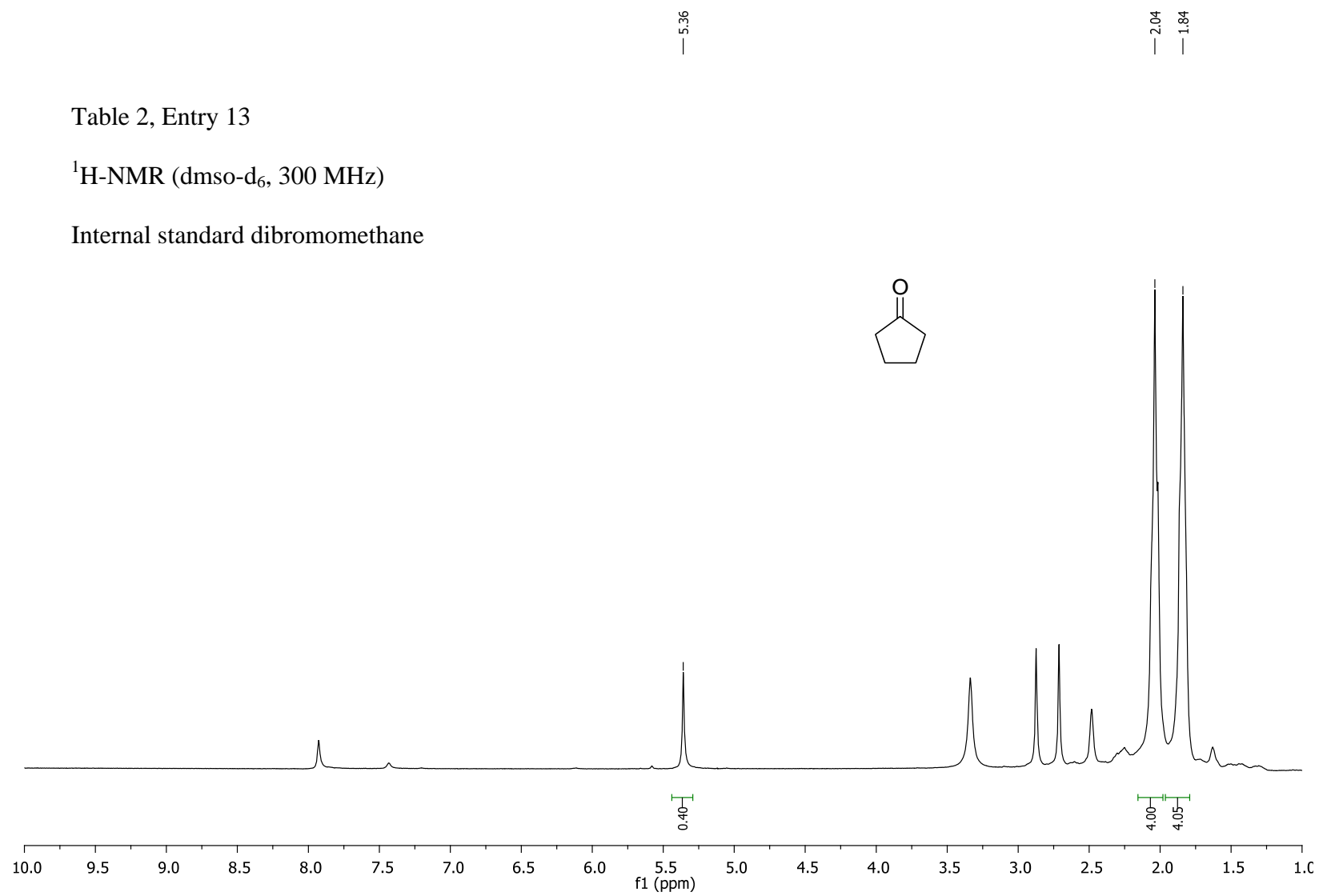




Table 2, Entry 13

$^1\text{H-NMR}$  (dms $o$ -d $_6$ , 300 MHz)

Internal standard dibromomethane

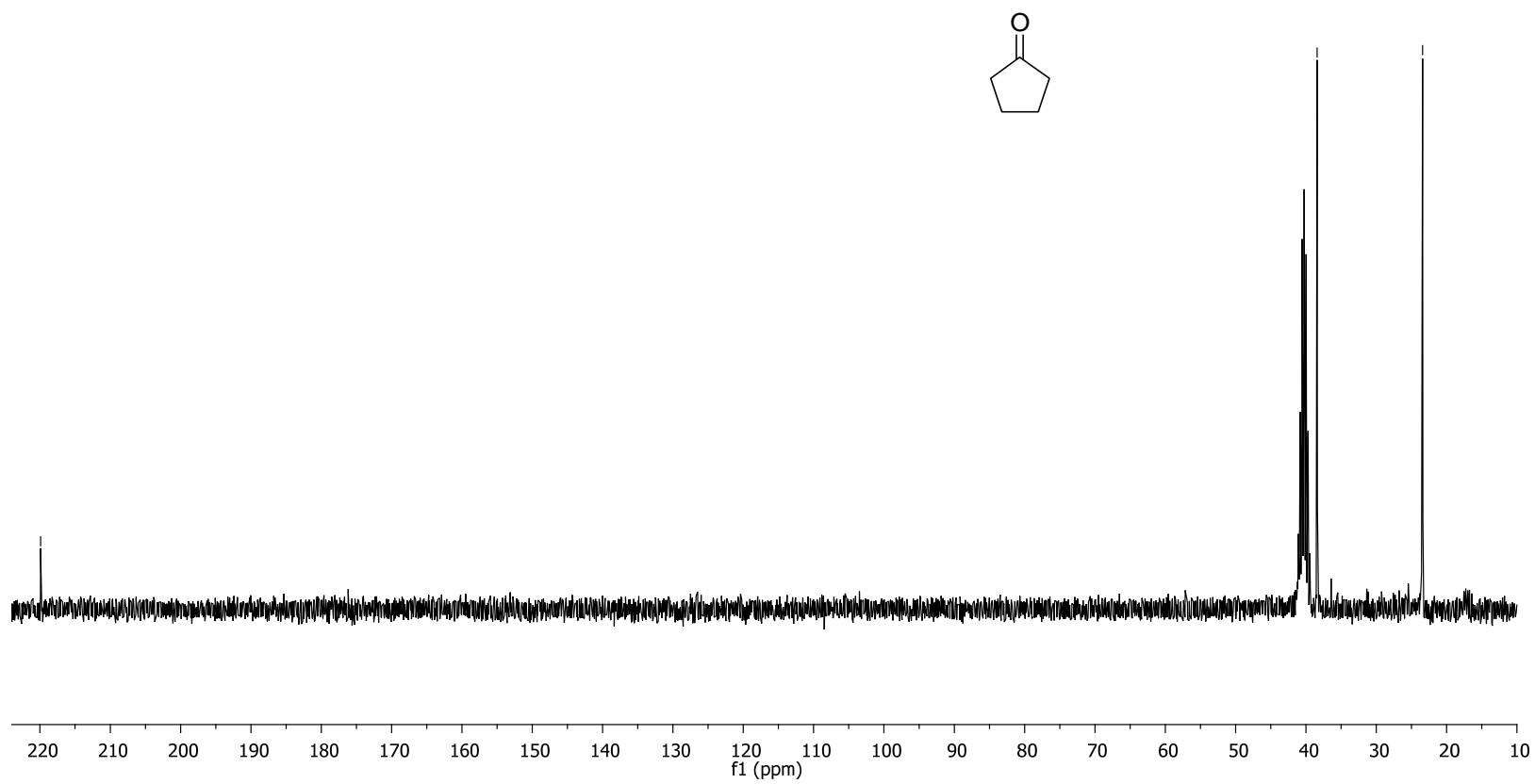


— 219.91

Table 2, Entry 13

$^{13}\text{C}$ -NMR (dms $o$ - $d_6$ , 75.4 MHz)

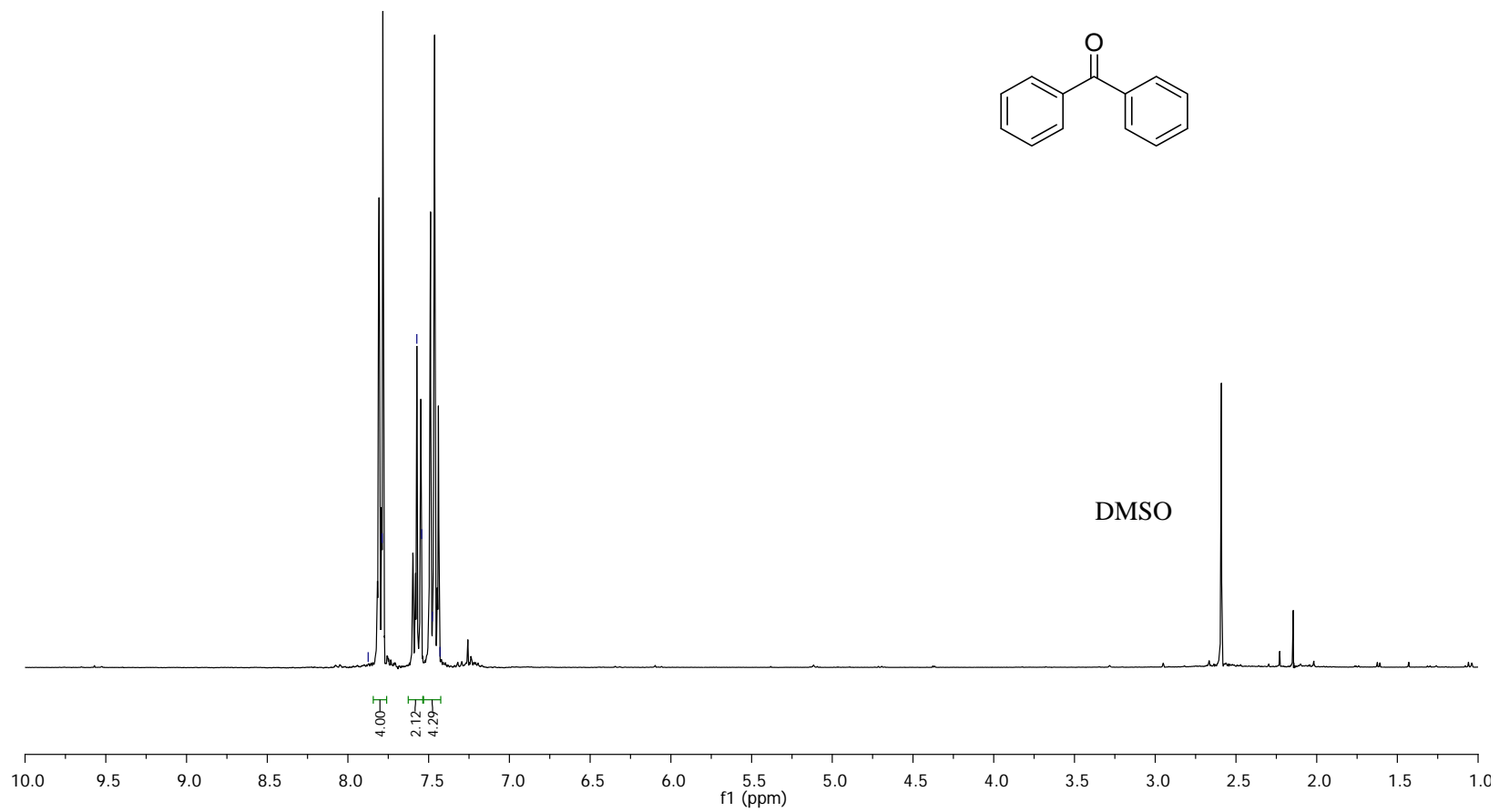
Internal standard dibromomethane

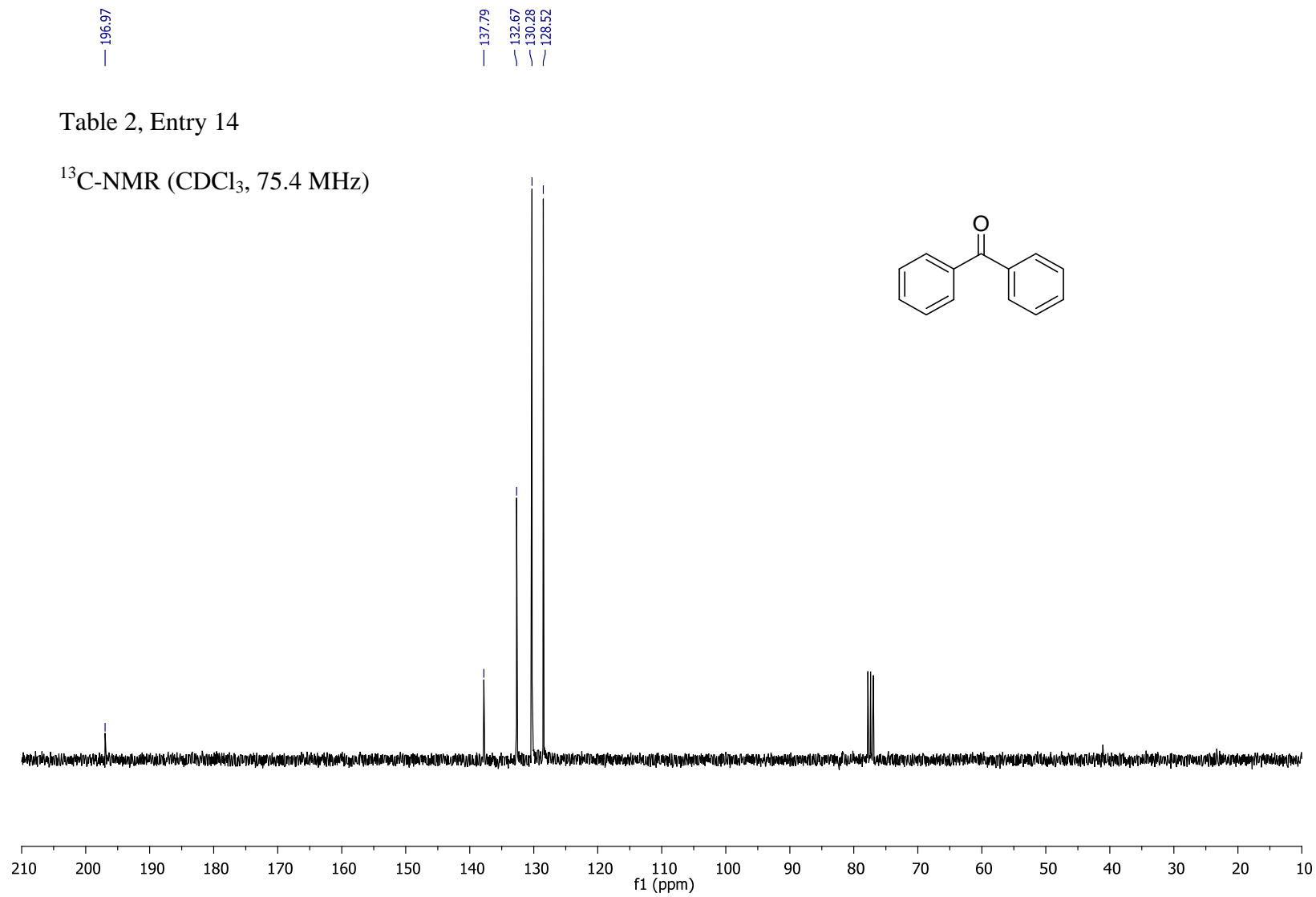


7.87  
7.79  
7.57  
7.54  
7.48  
7.43

Table 2, Entry 14

$^1\text{H-NMR}$ ( $\text{CDCl}_3$ , 300 MHz)





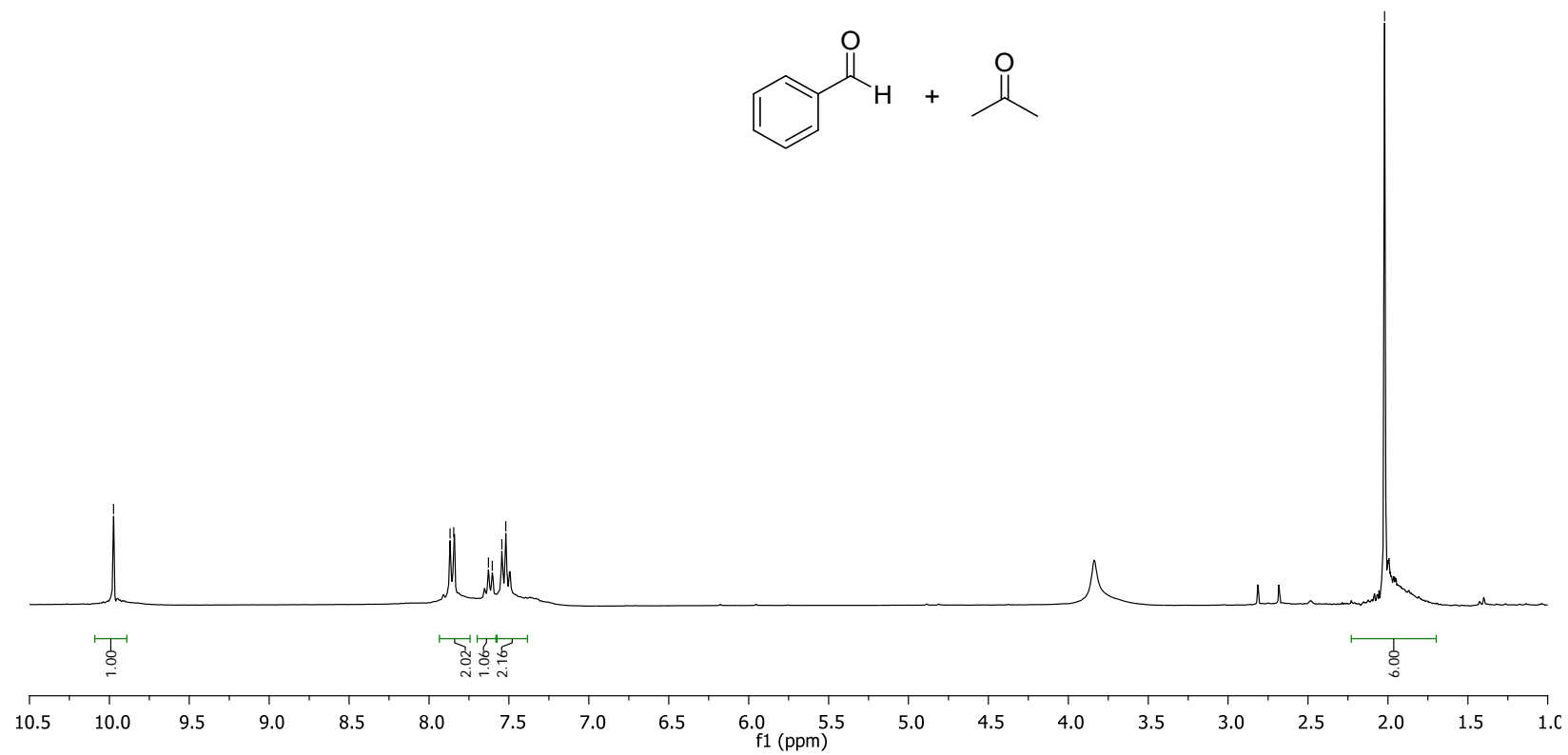
— 9.97

7.87  
7.85  
7.63  
7.60  
7.54  
7.52

— 2.02

Table 2, Entry 15

<sup>1</sup>H-NMR (dms<sub>o</sub>-d<sub>6</sub>, 300 MHz)



— 206.87

— 193.41

— 136.82

— 135.00

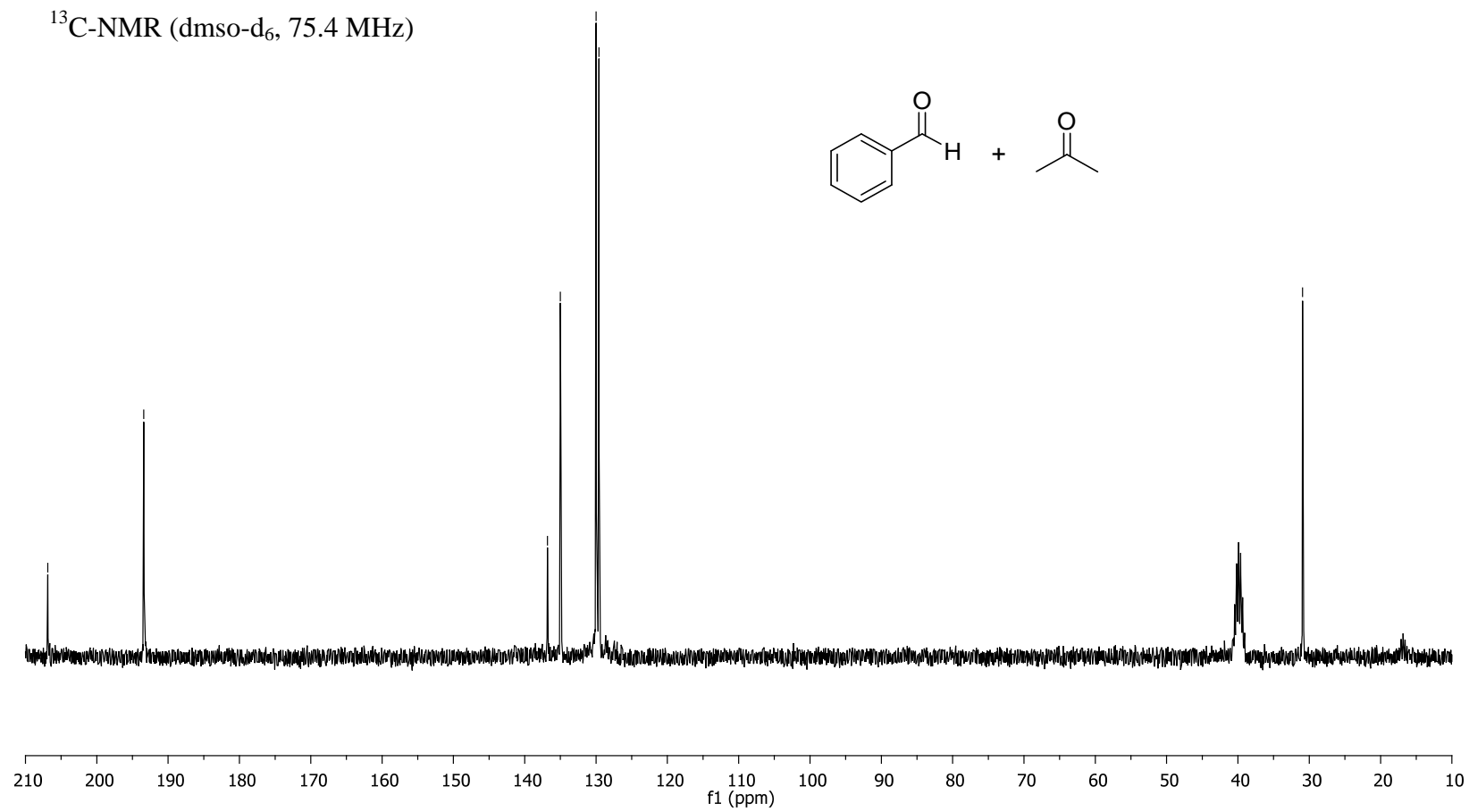
— 129.99

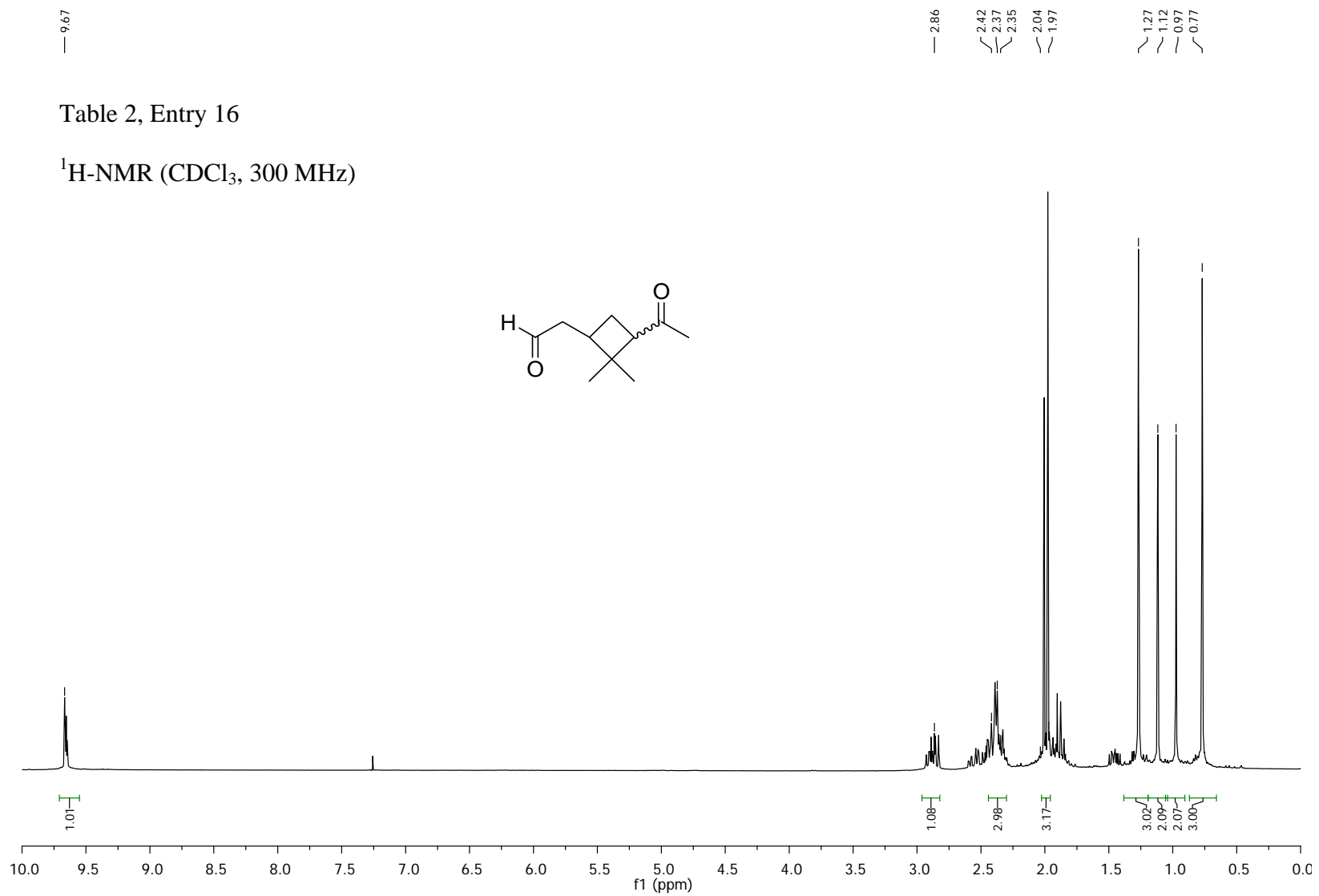
— 129.60

— 30.95

Table 2, Entry 15

$^{13}\text{C}$ -NMR (dms $o$ -d $_6$ , 75.4 MHz)



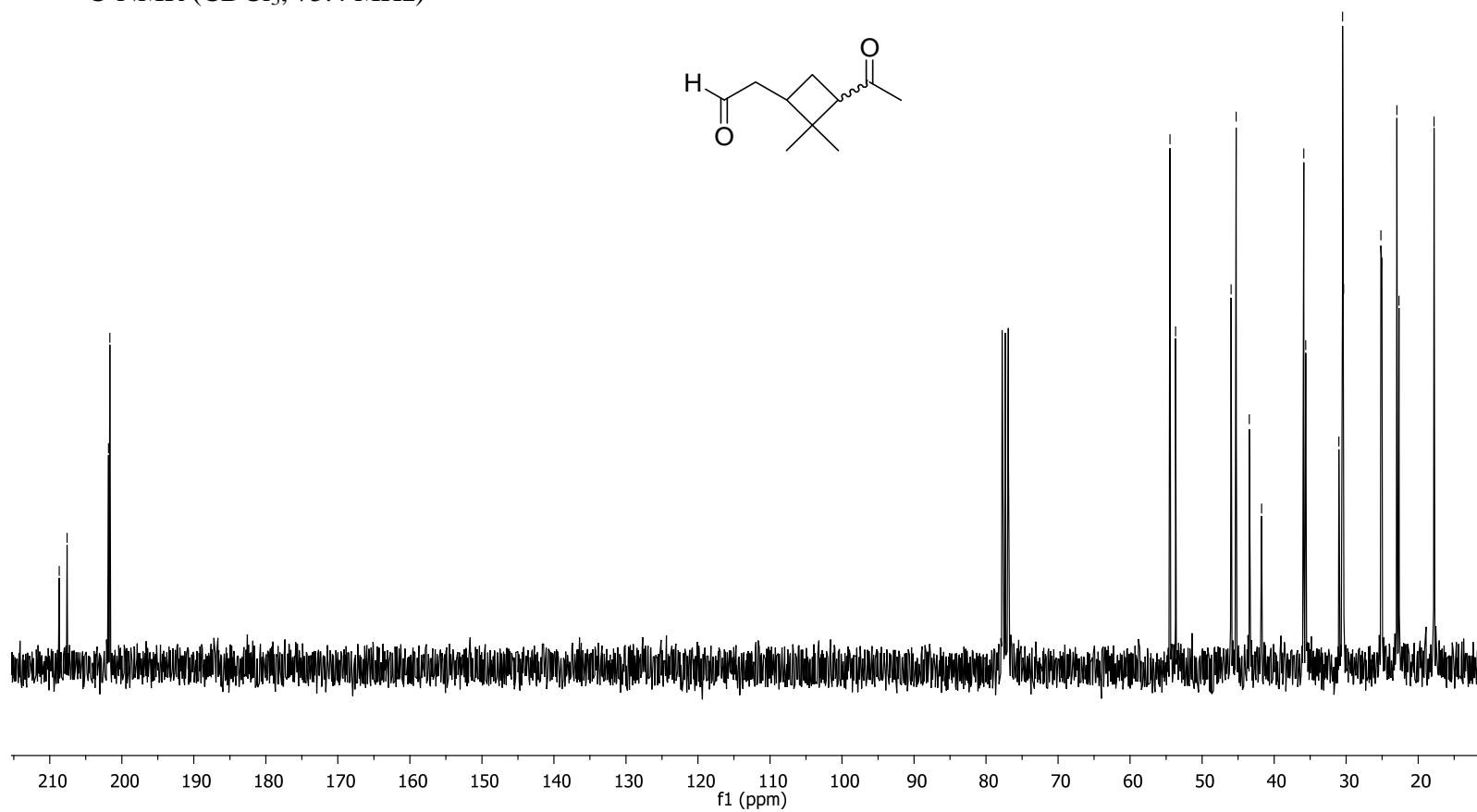
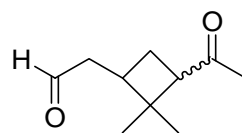


208.70  
207.60  
201.85  
201.66

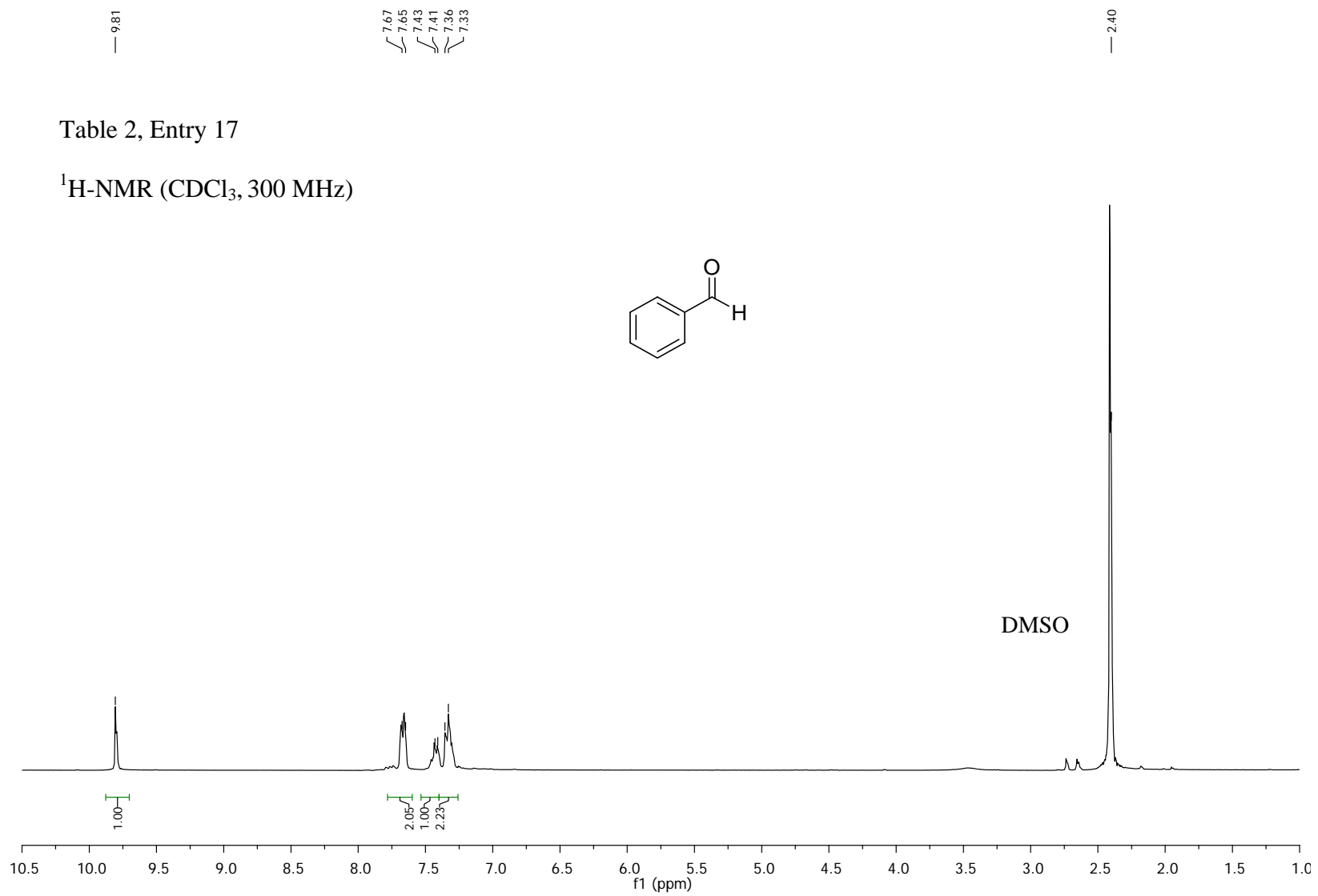
54.46  
53.68  
45.97  
45.27  
43.43  
41.73  
35.88  
35.61  
30.49  
30.35  
23.18  
25.03  
22.95  
22.66  
17.80

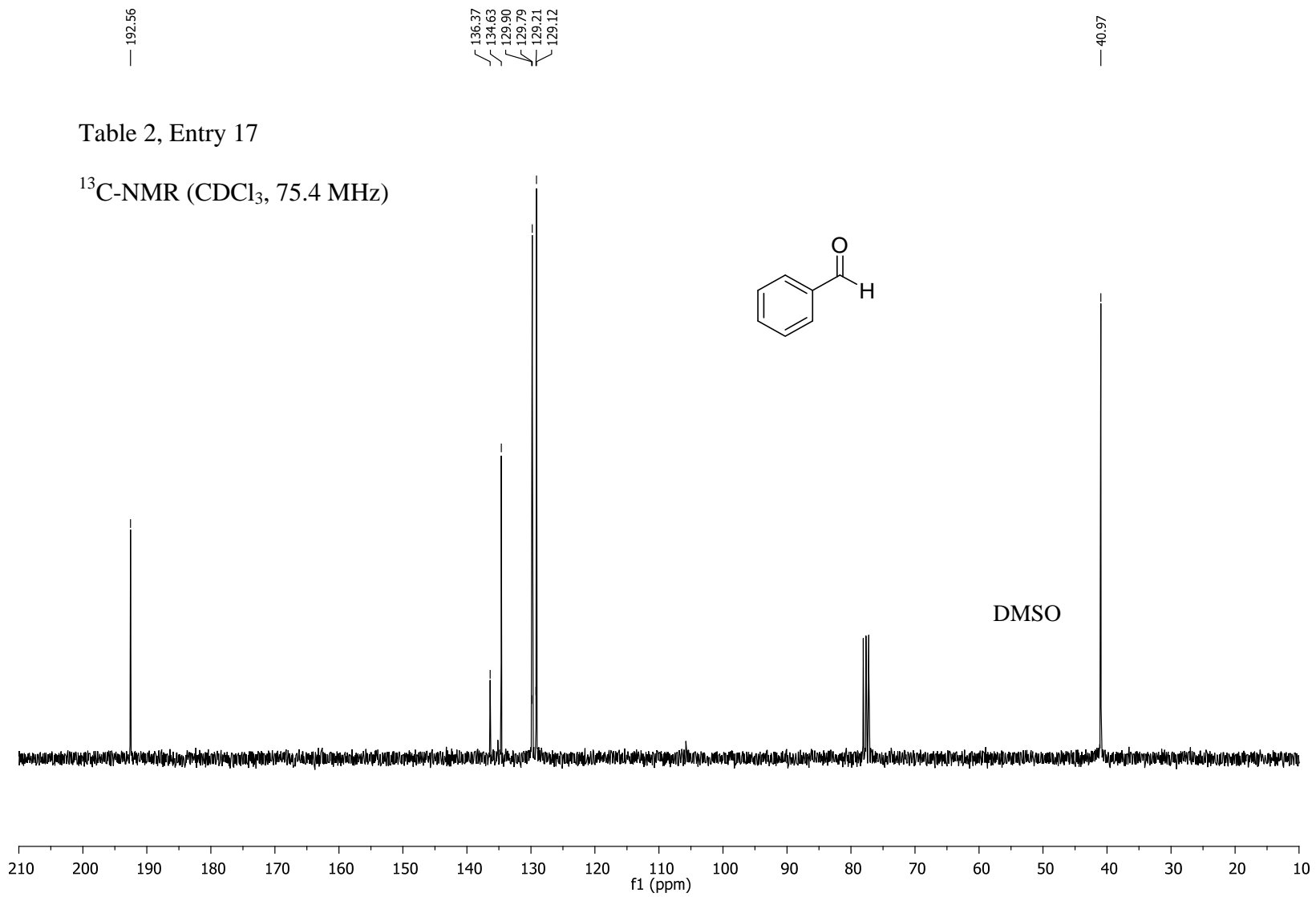
Table 2, Entry 16

$^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 75.4 MHz)









10.00

7.87  
7.85  
7.61  
7.58  
7.54  
7.48

Table 2, Entry 18

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)

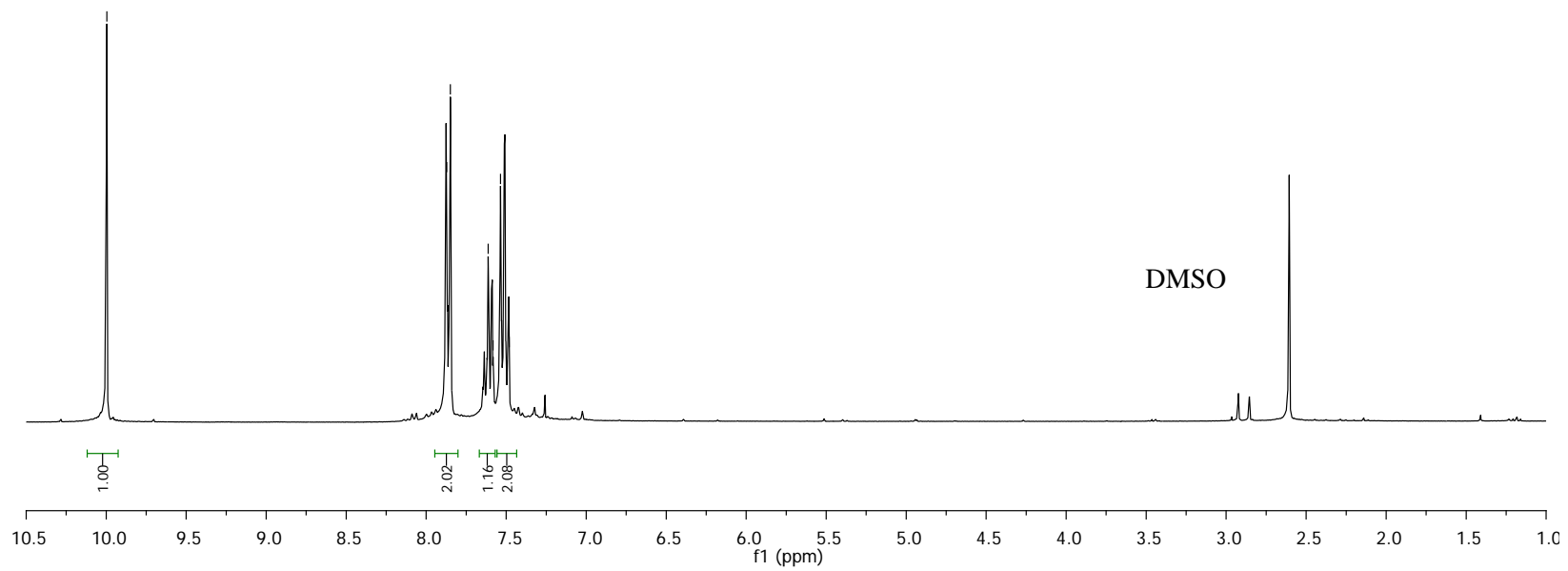
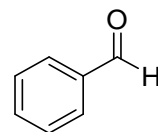


Table 2, Entry 19

Without catalyst

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)

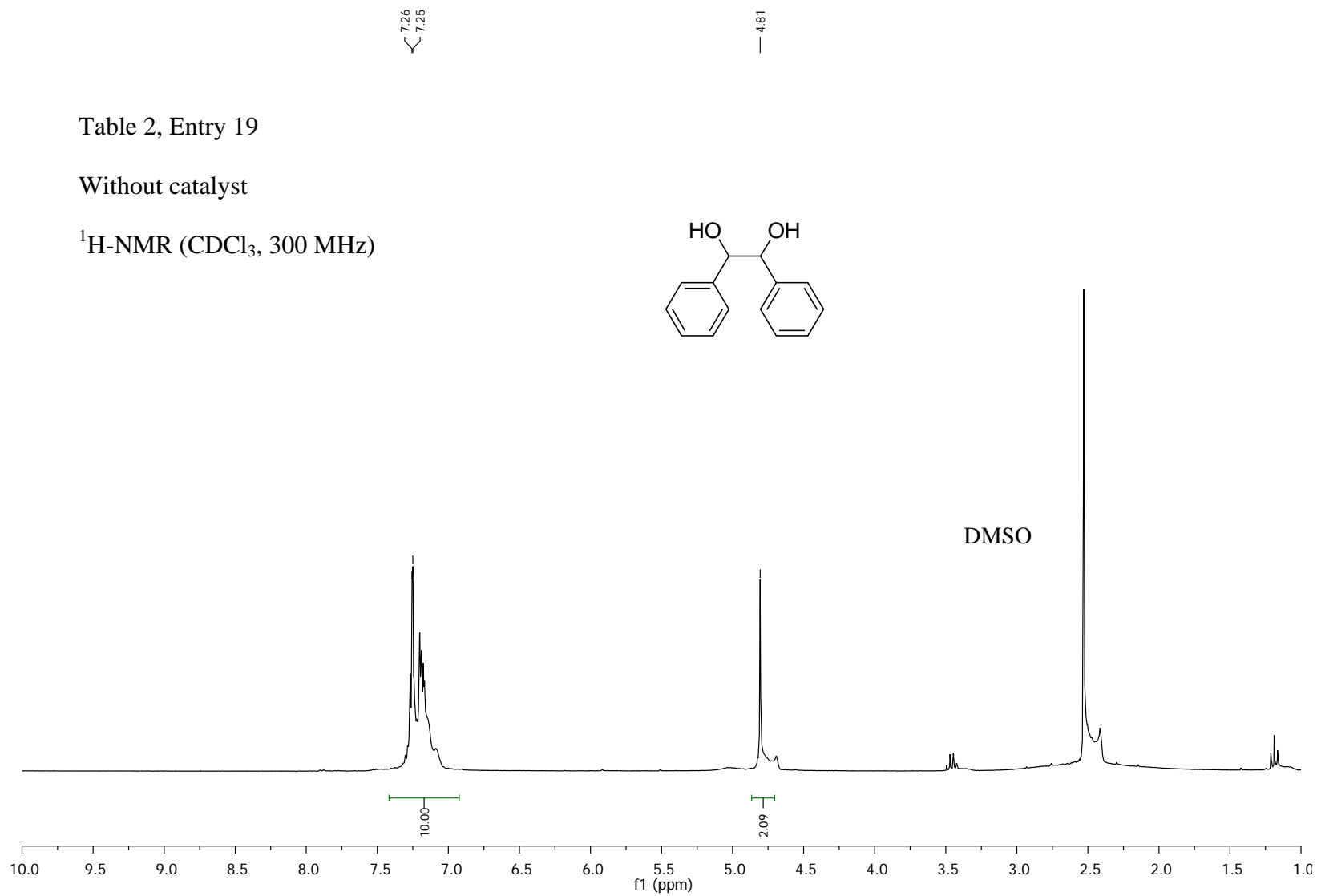
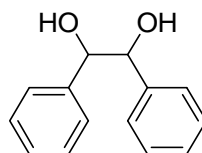


Table 2, Entry 19

Without catalyst

$^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 75.4 MHz)

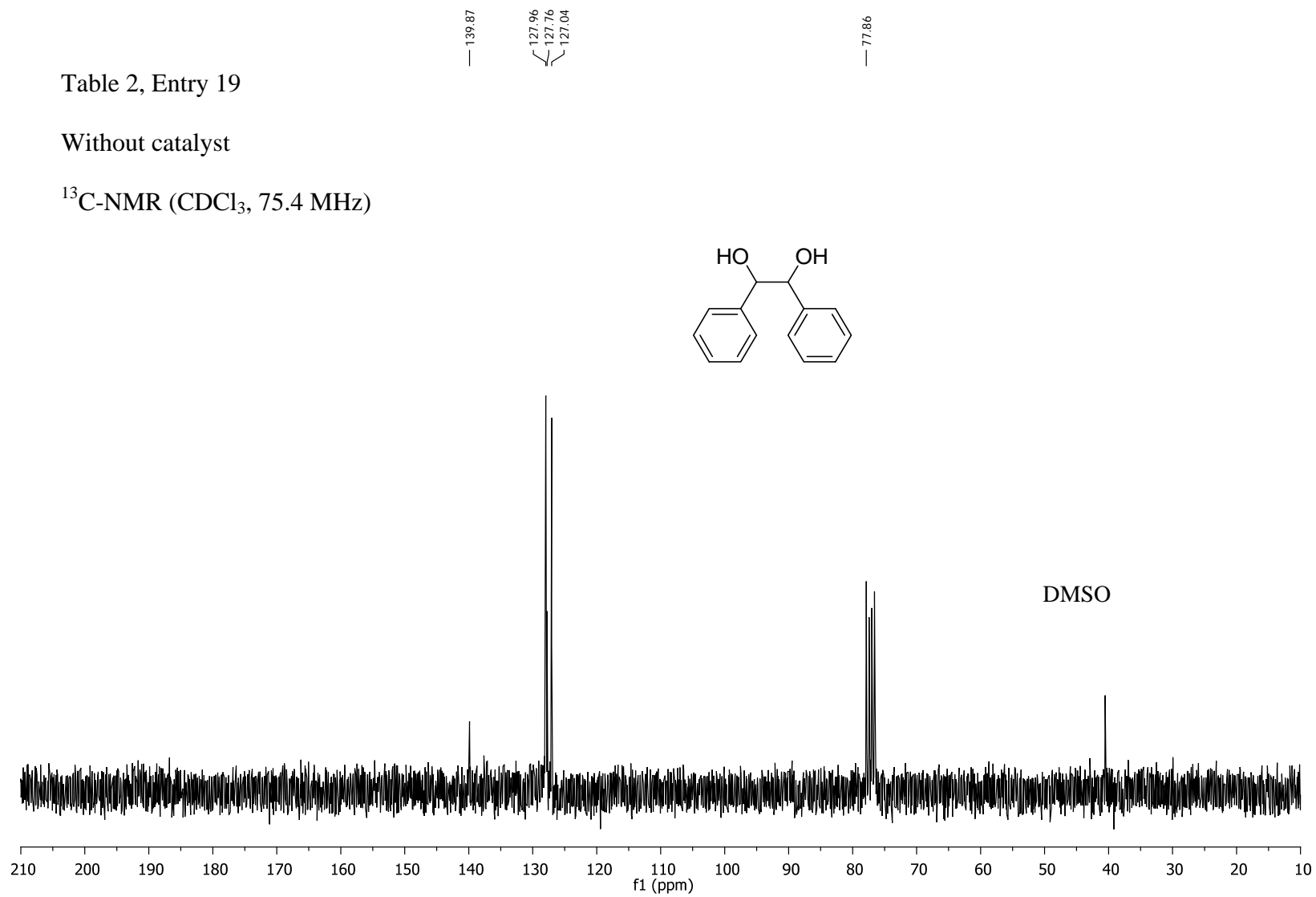
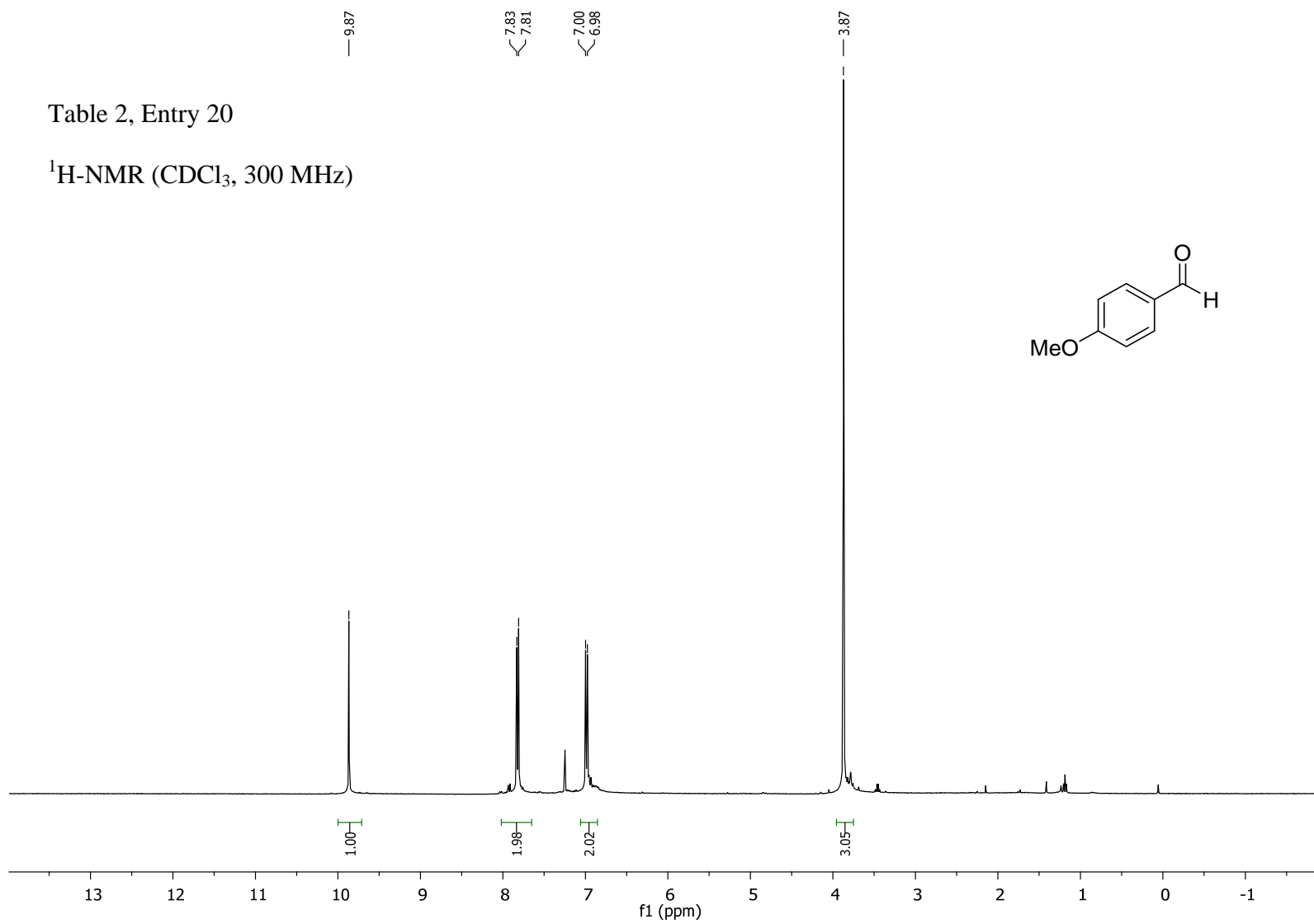
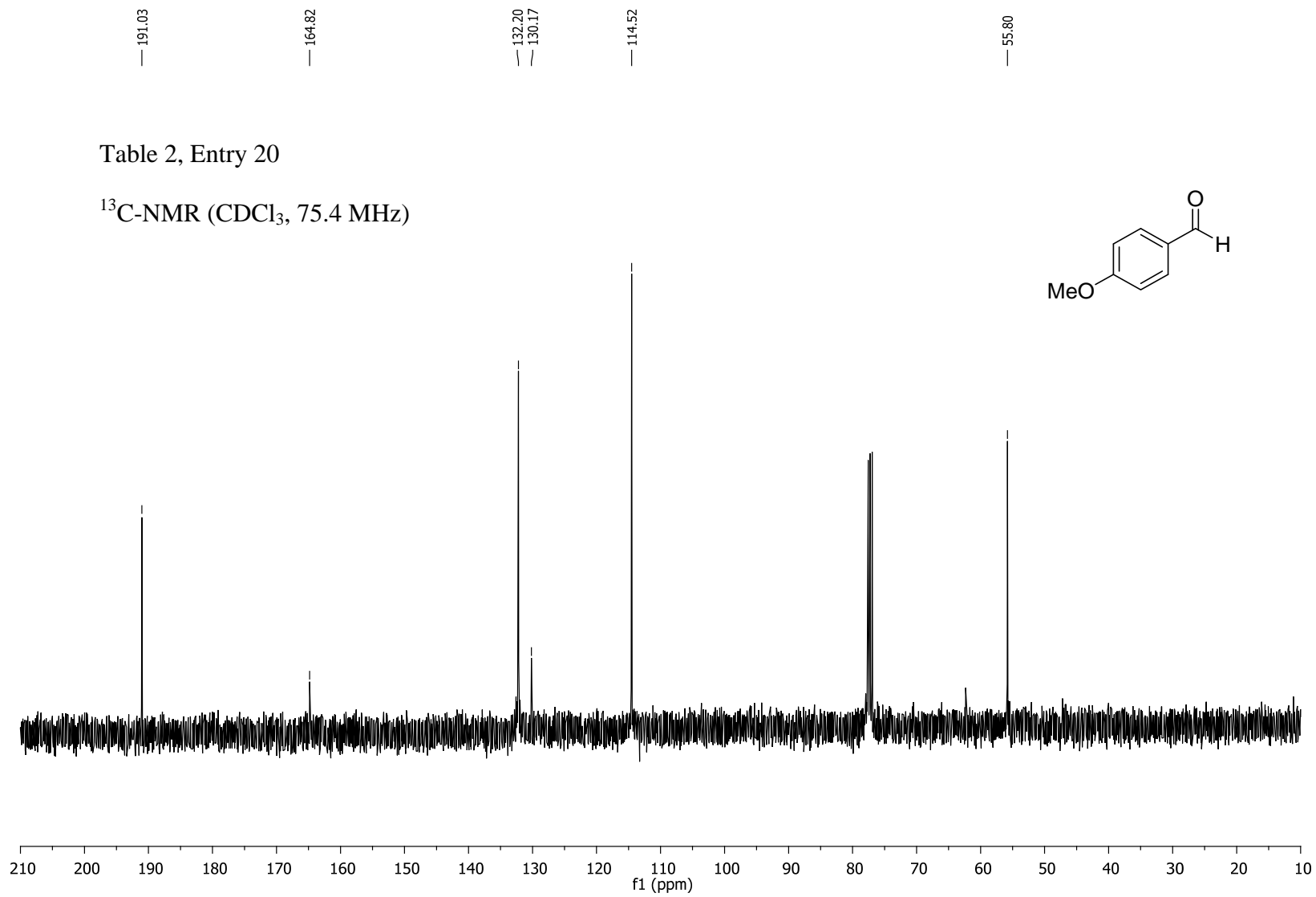


Table 2, Entry 20

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)



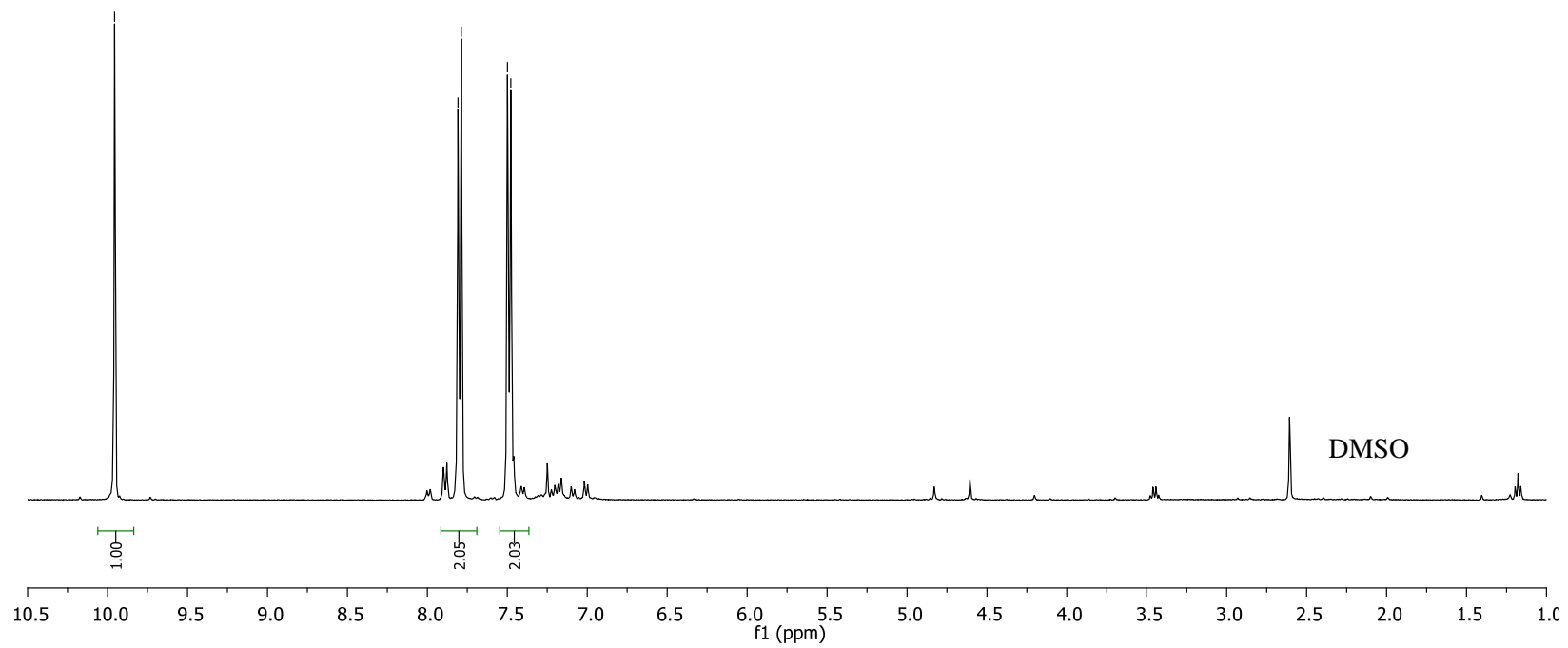
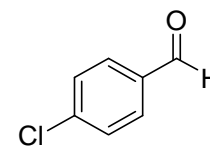


— 9.96

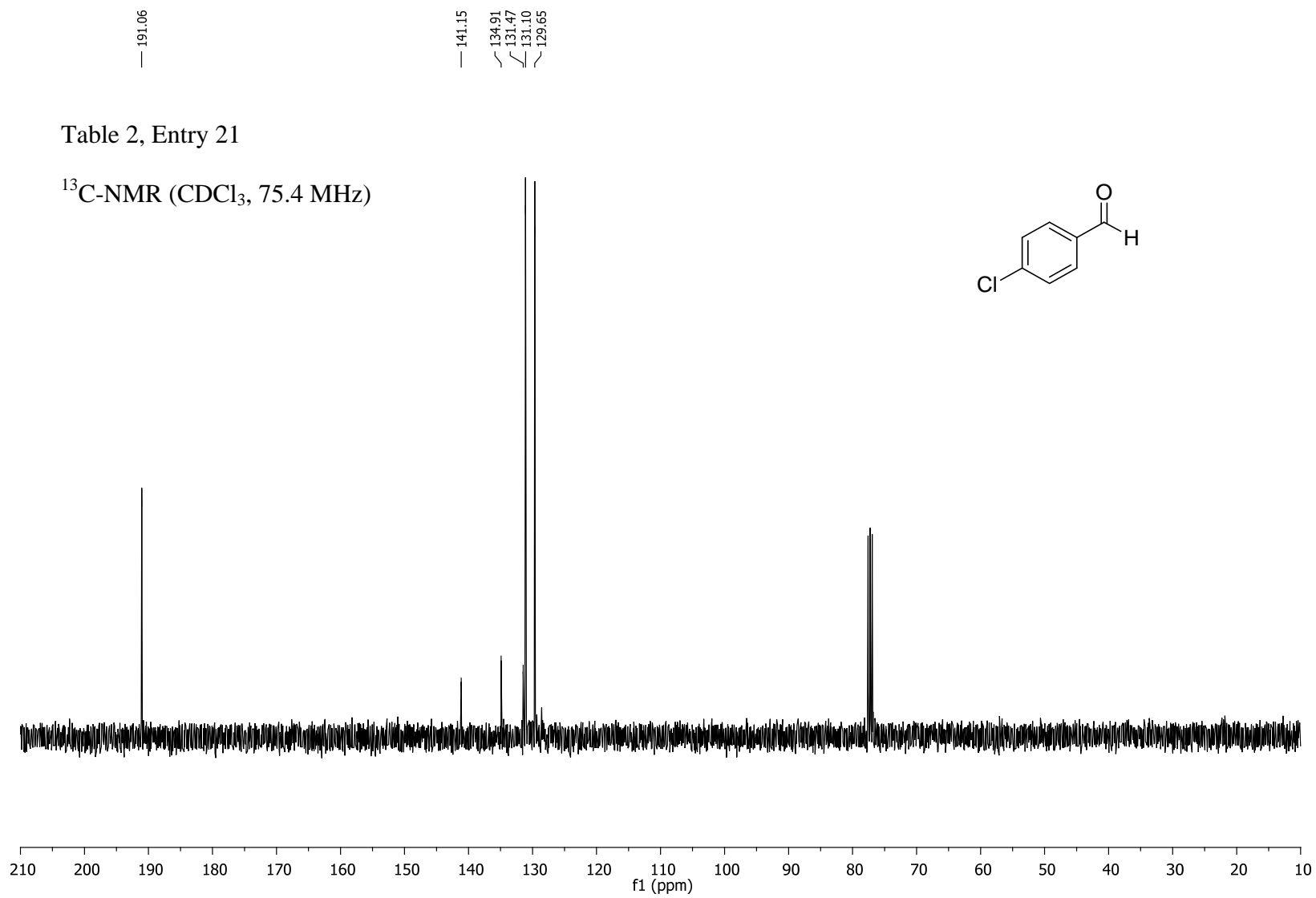
< 7.81  
< 7.79  
< 7.50  
< 7.48

Table 2, Entry 21

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)





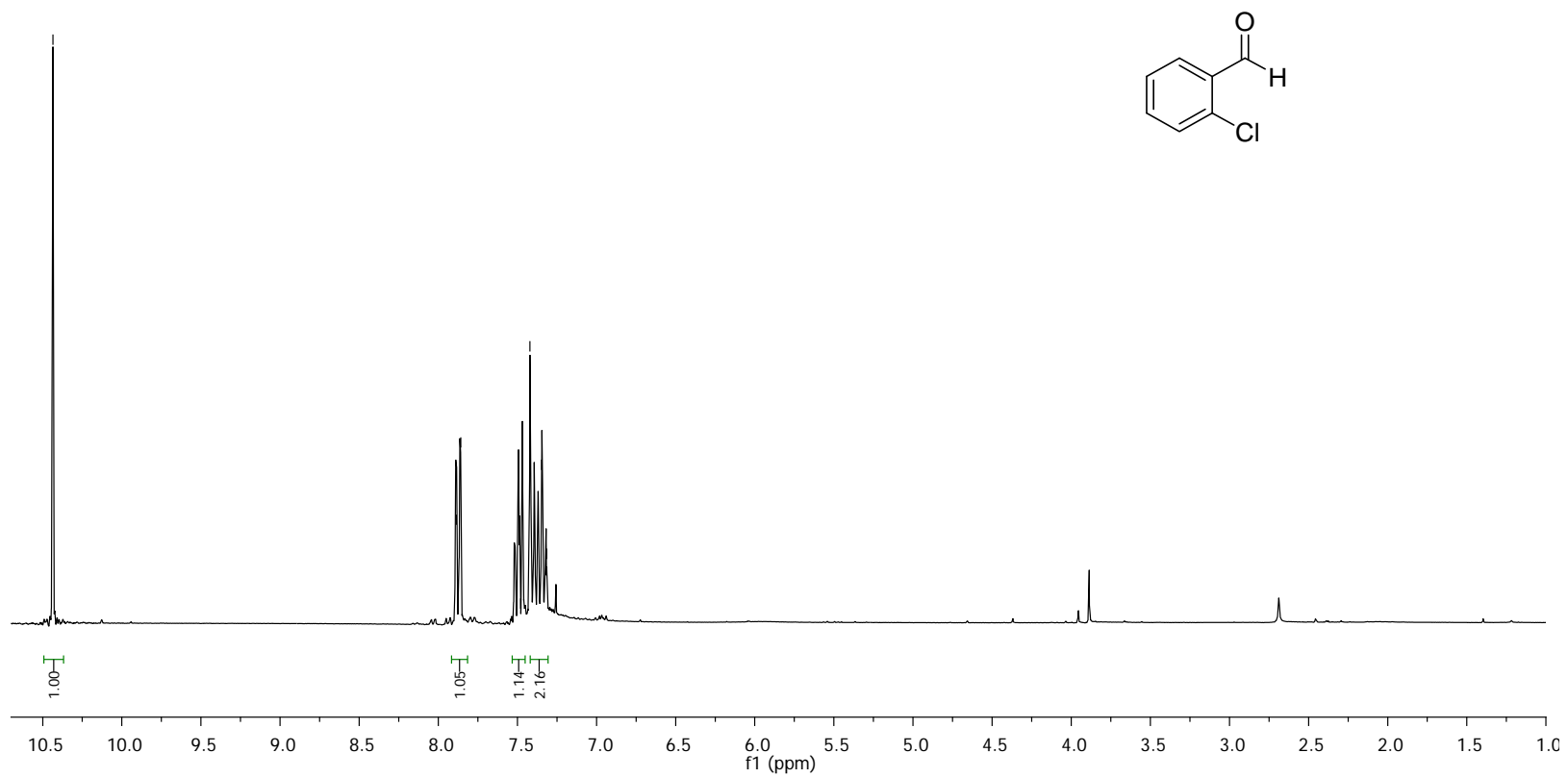


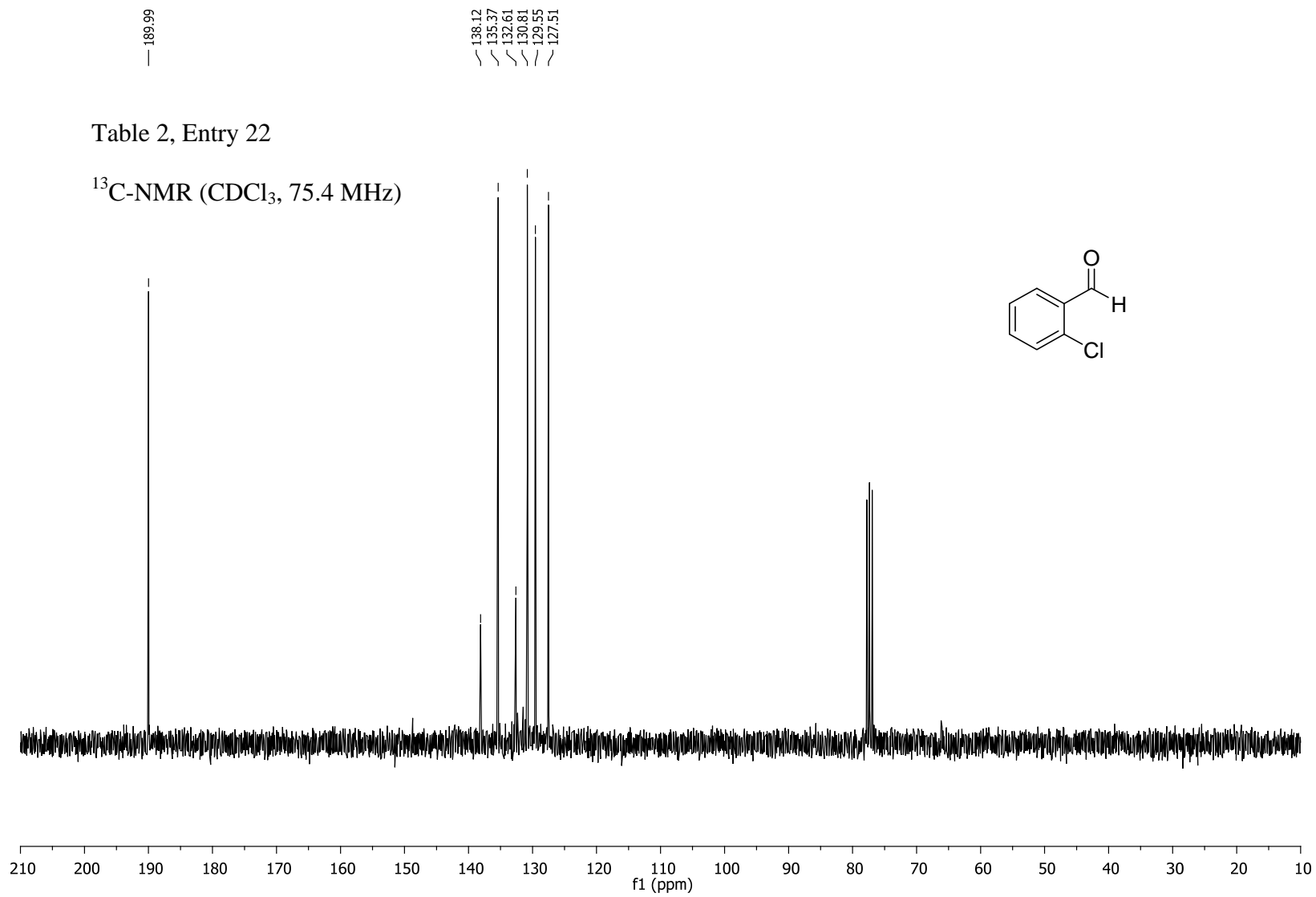
— 10.44

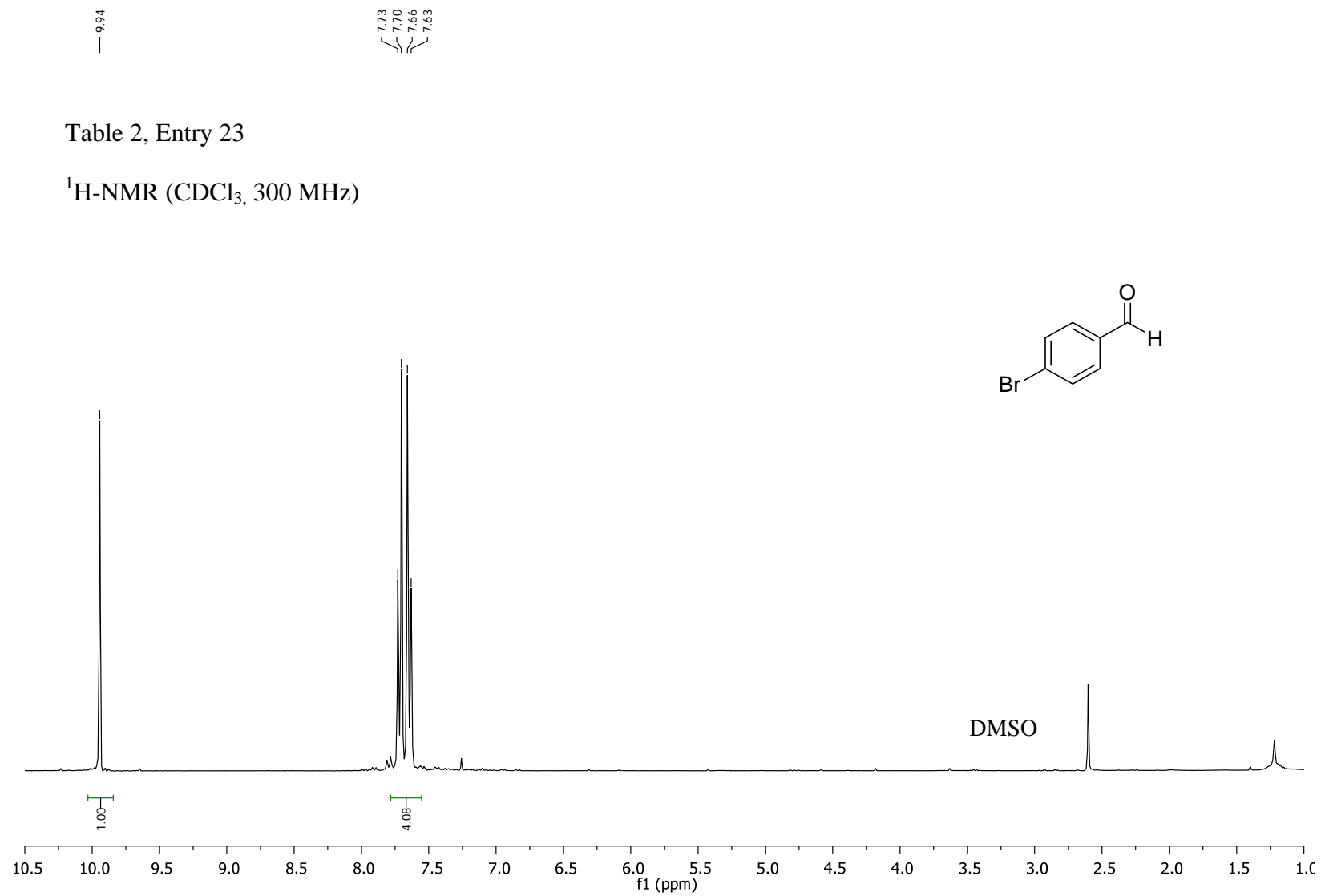
7.89  
7.86  
7.48  
7.42  
7.35  
7.32

Table 2, Entry 22

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)







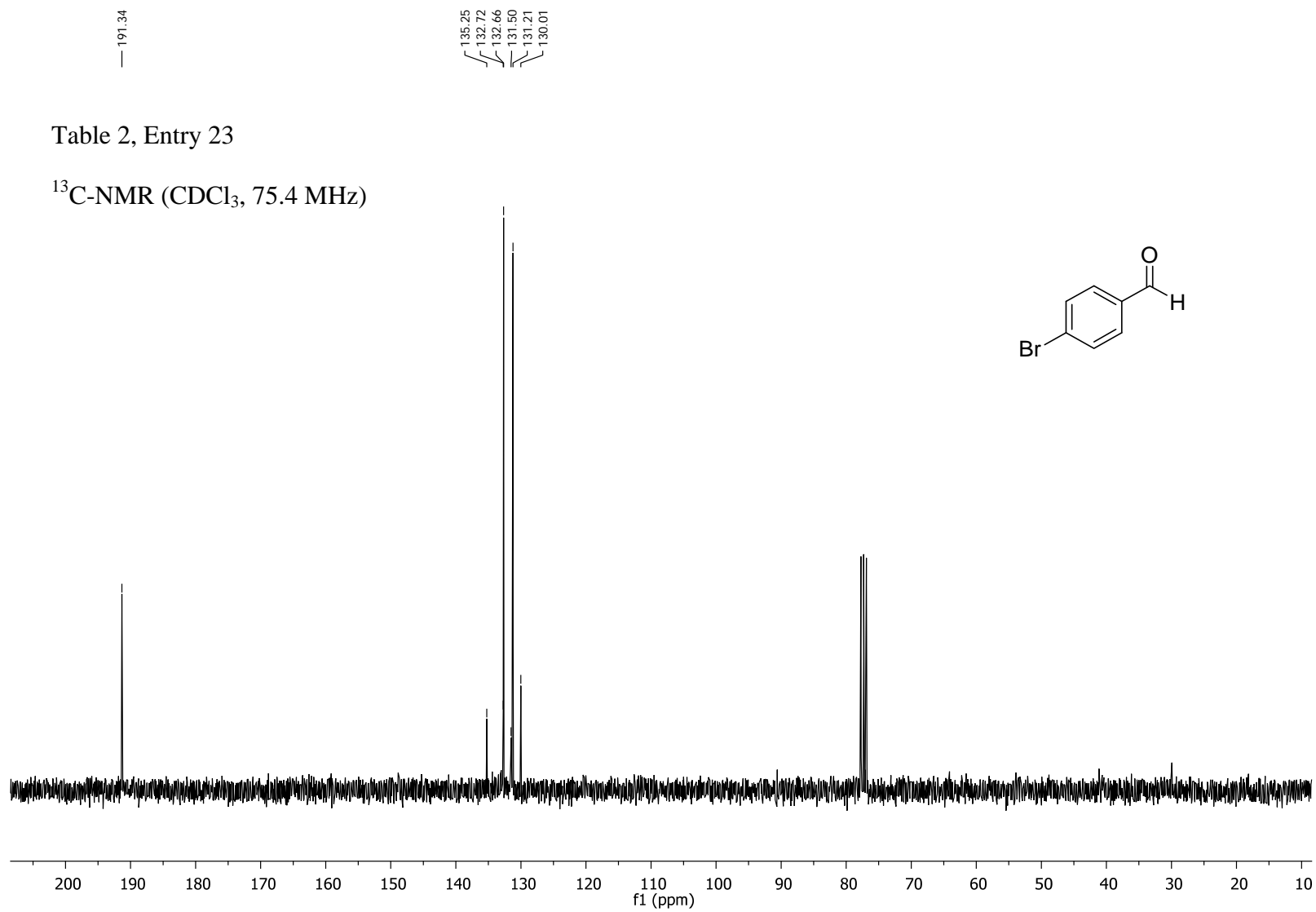
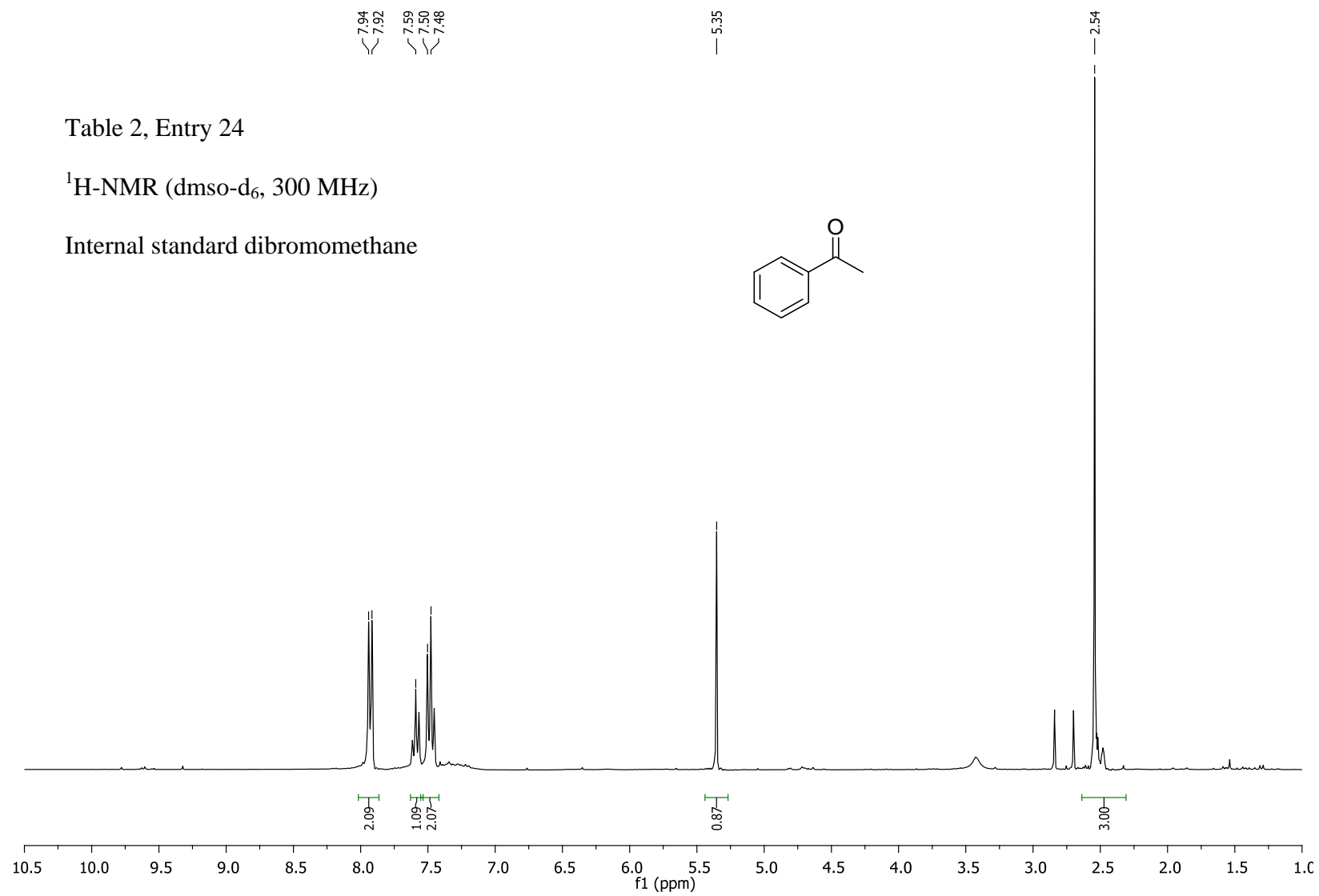
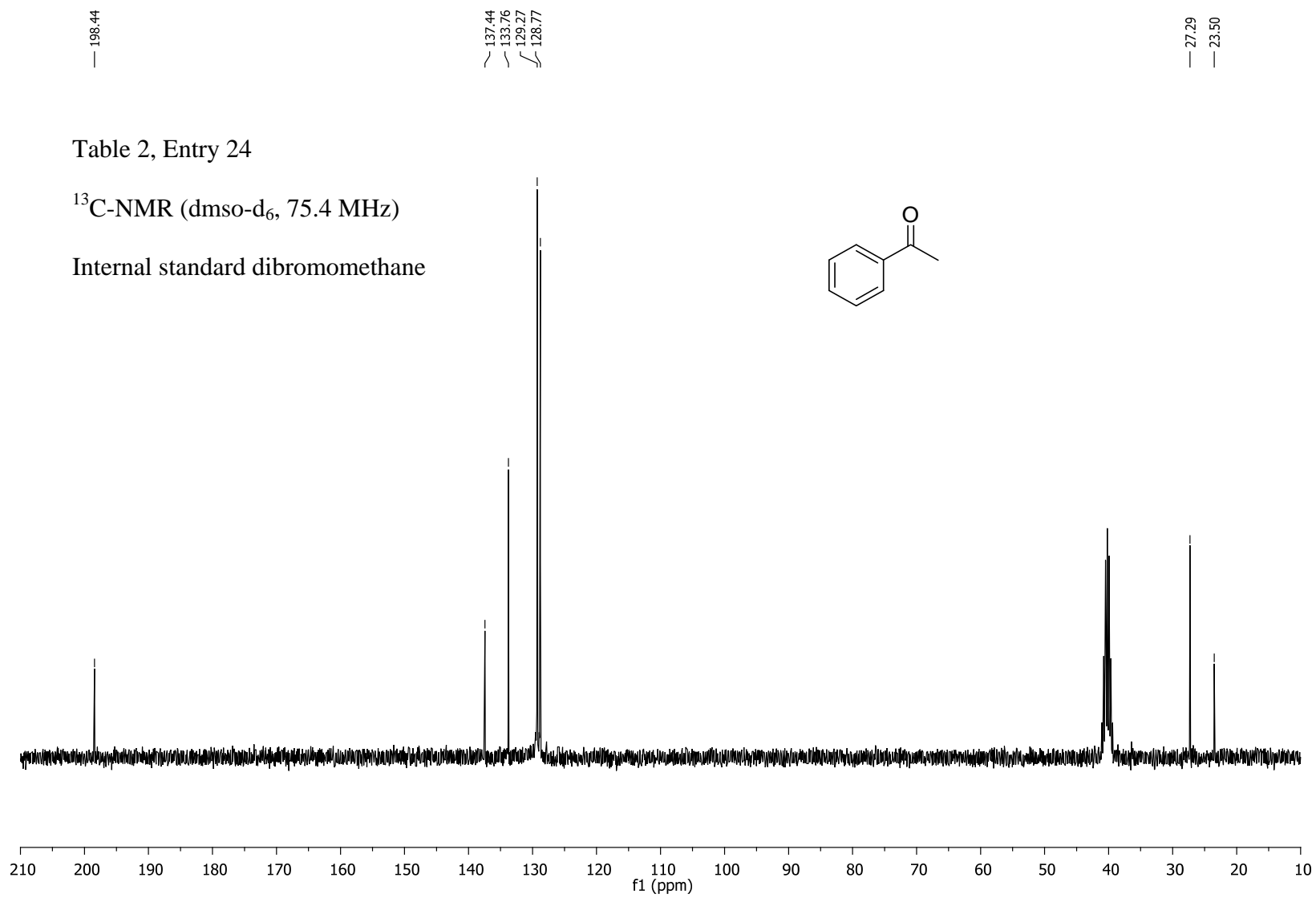


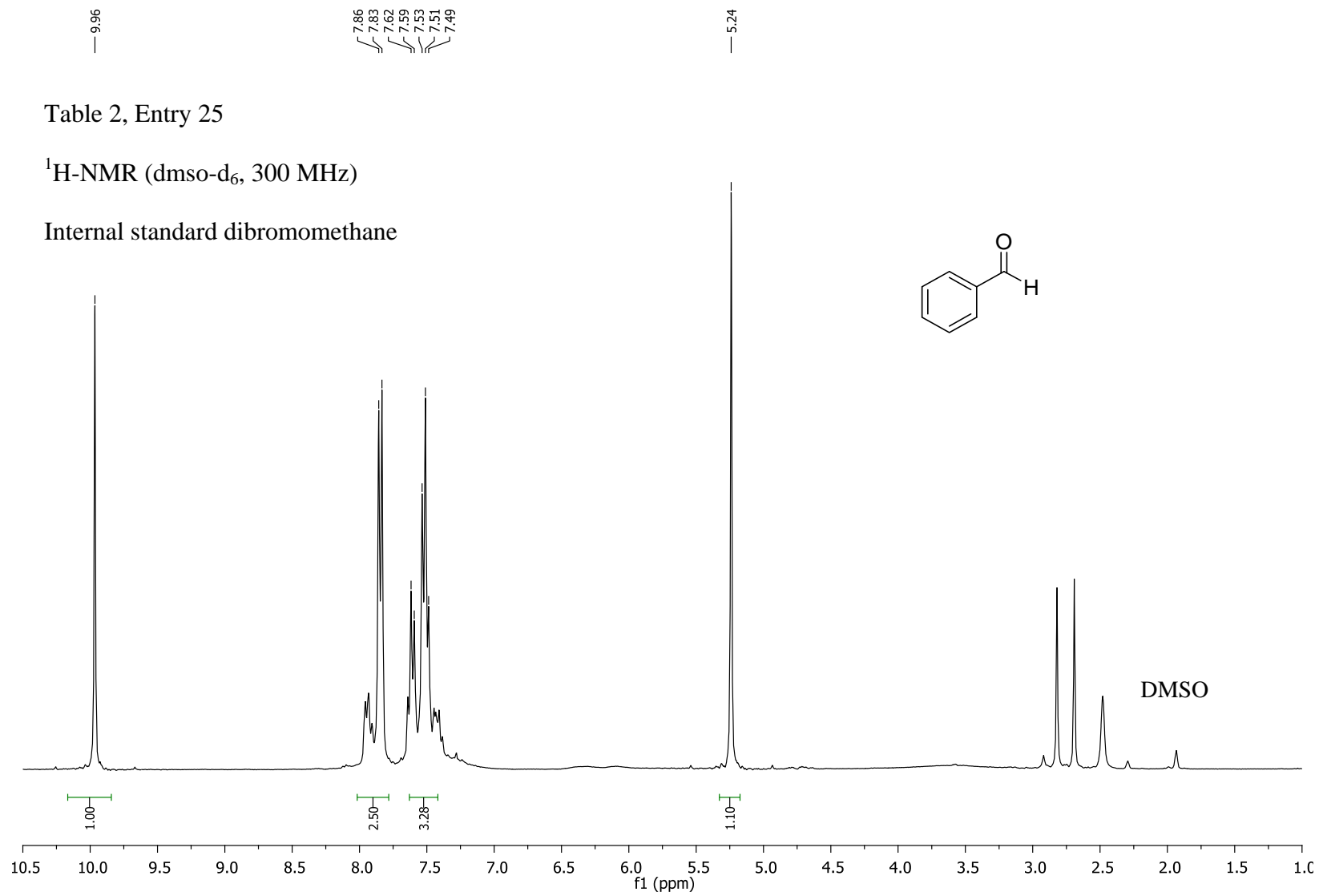
Table 2, Entry 24

$^1\text{H-NMR}$  (dms $o$ -d $_6$ , 300 MHz)

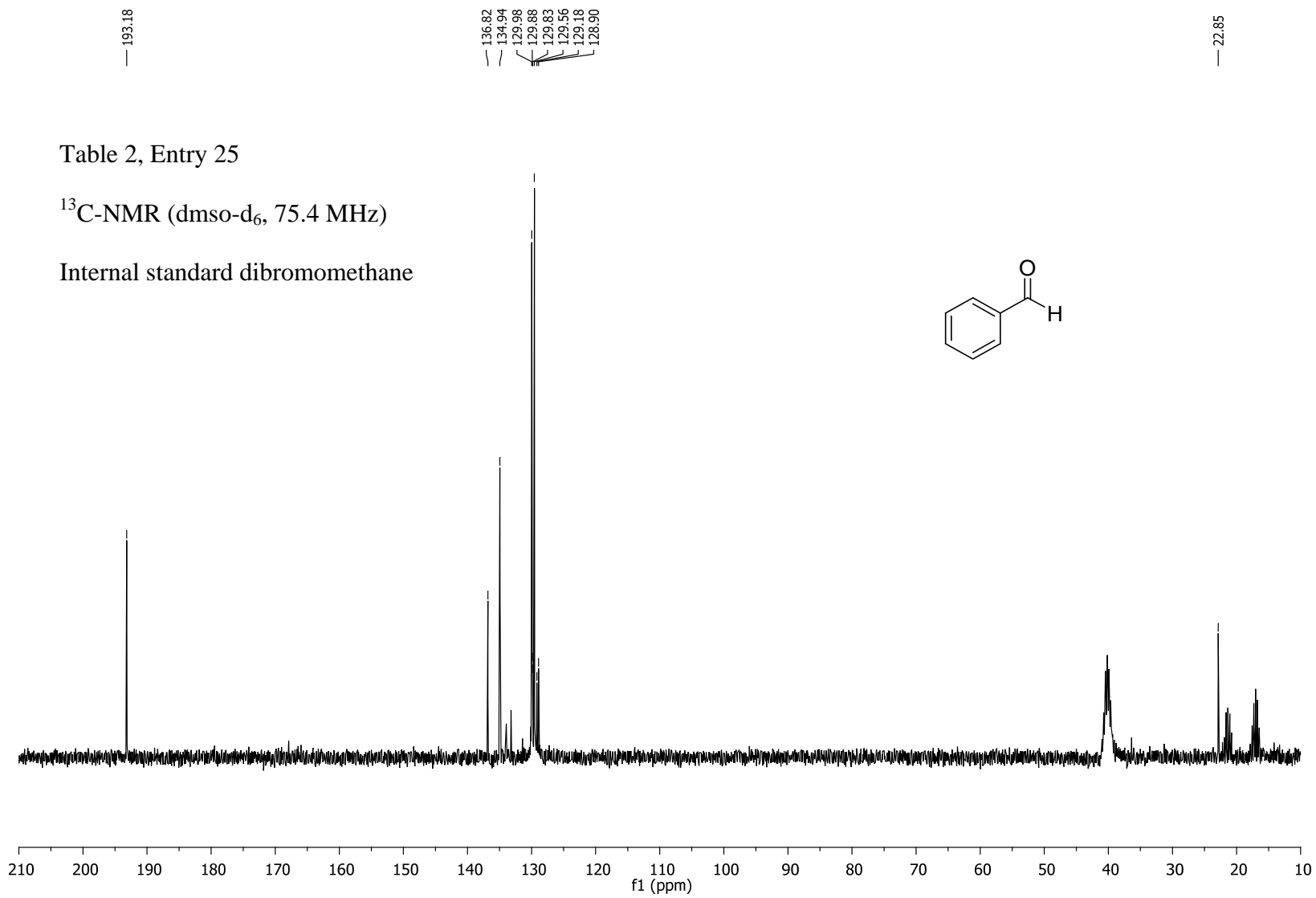
Internal standard dibromomethane







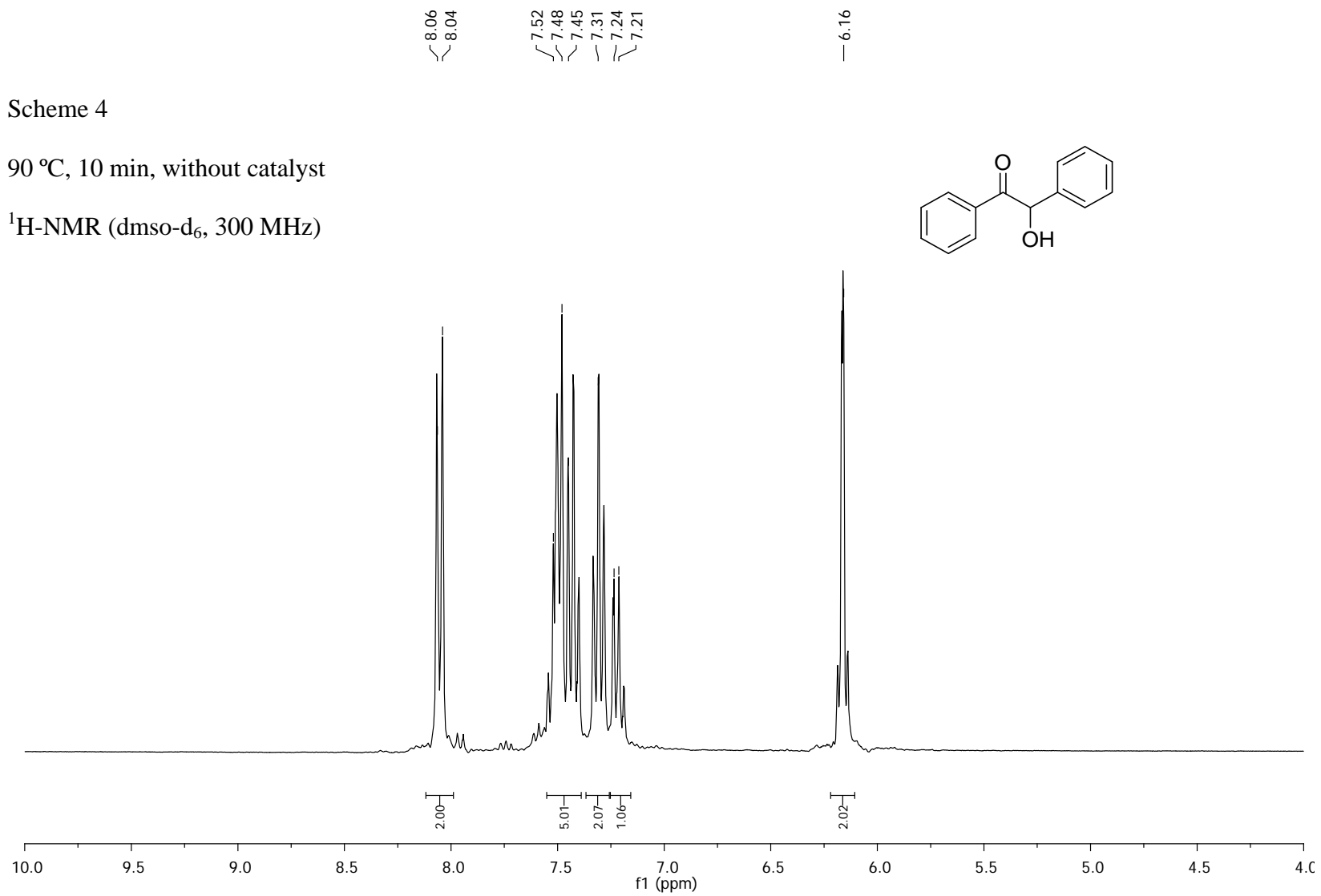


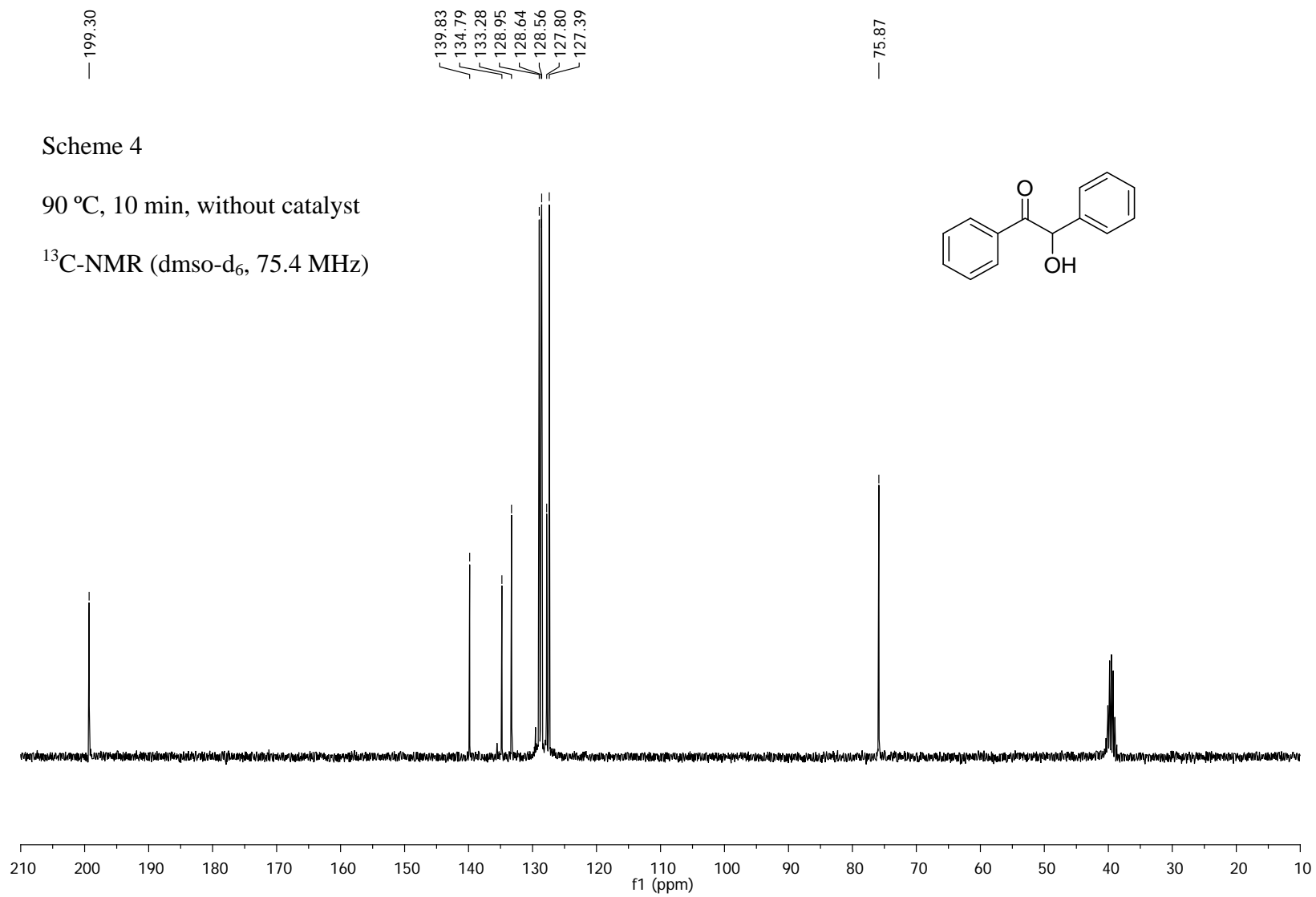


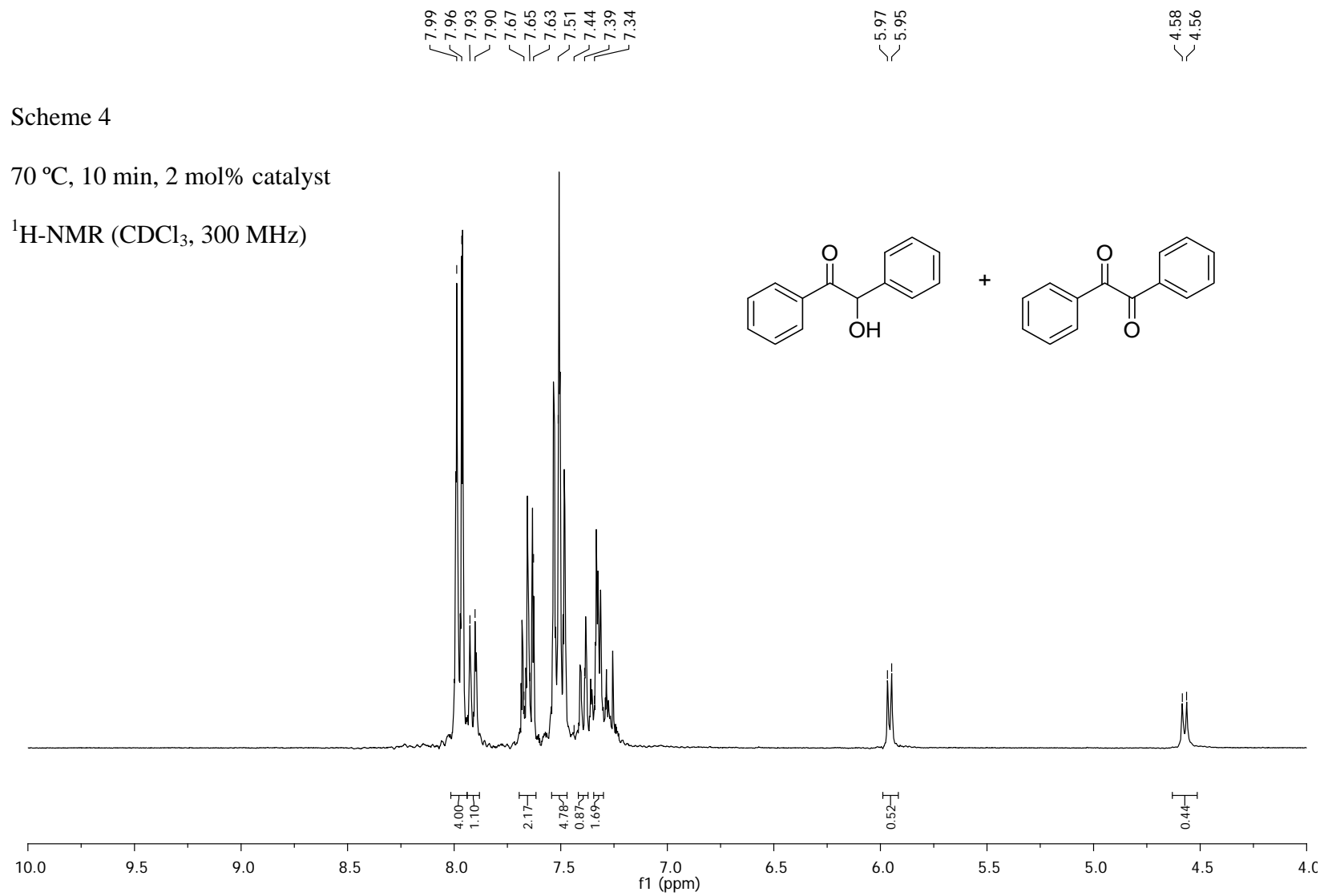
Scheme 4

90 °C, 10 min, without catalyst

$^1\text{H-NMR}$  (dms $o$ - $d_6$ , 300 MHz)







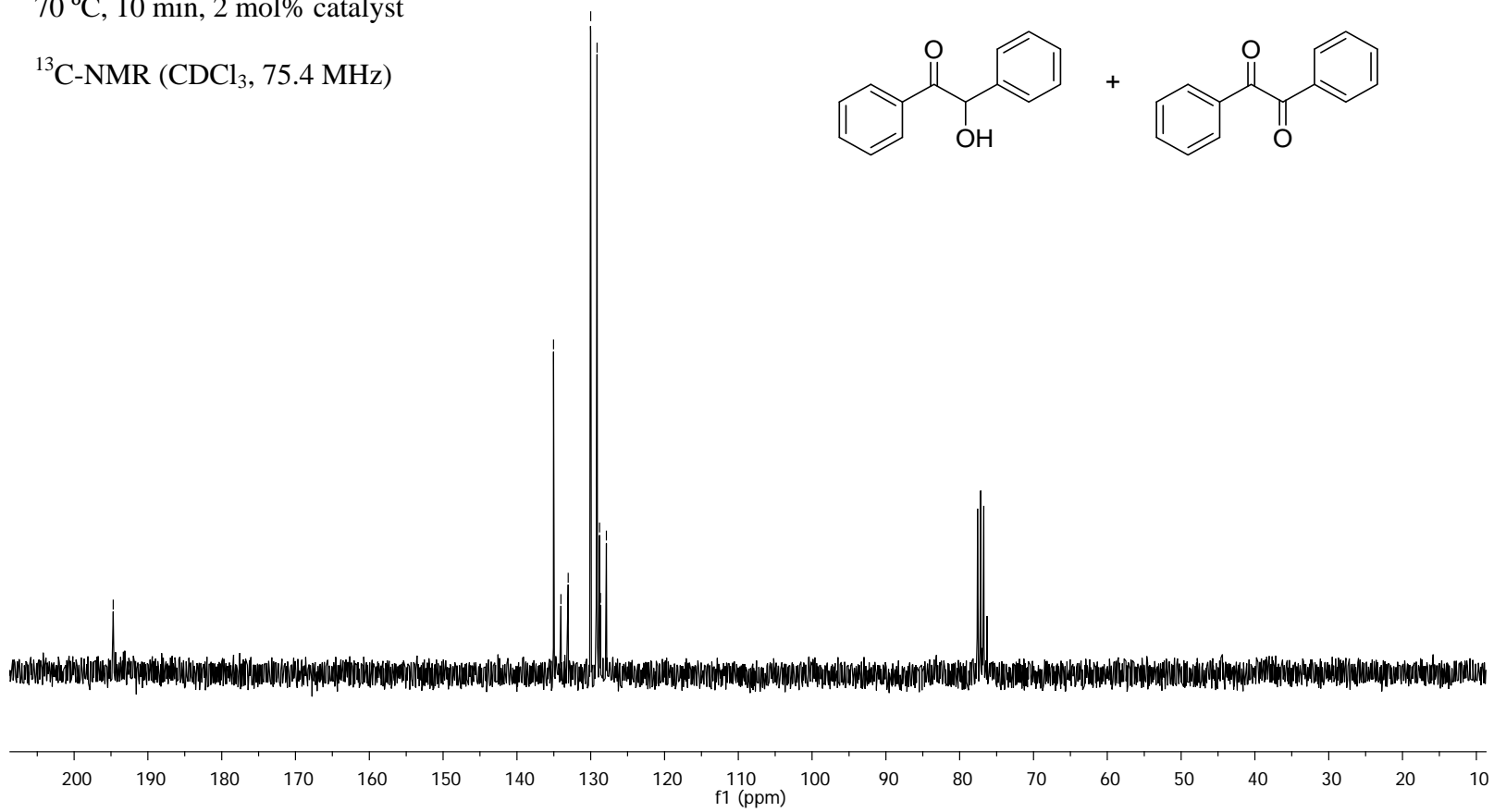
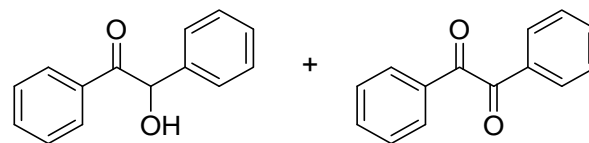
— 194.71

135.03  
134.04  
133.05  
130.01  
129.24  
129.14  
128.79  
128.68  
127.87

Scheme 4

70 °C, 10 min, 2 mol% catalyst

$^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 75.4 MHz)

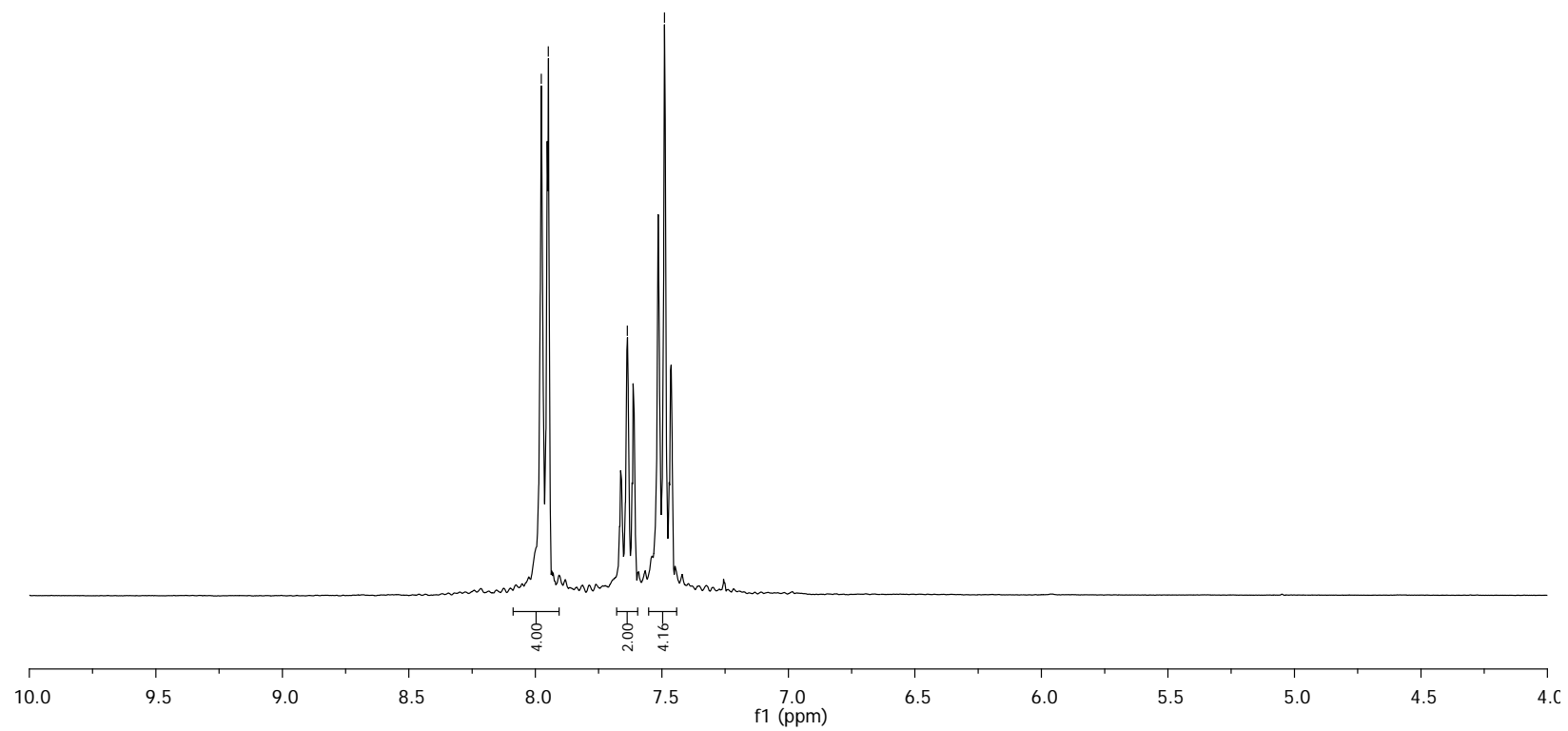
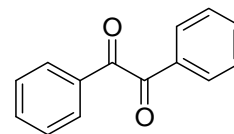


7.98  
7.95  
7.64  
7.49

Scheme 4

$R^1 = R^2 = \text{Ph}$

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)



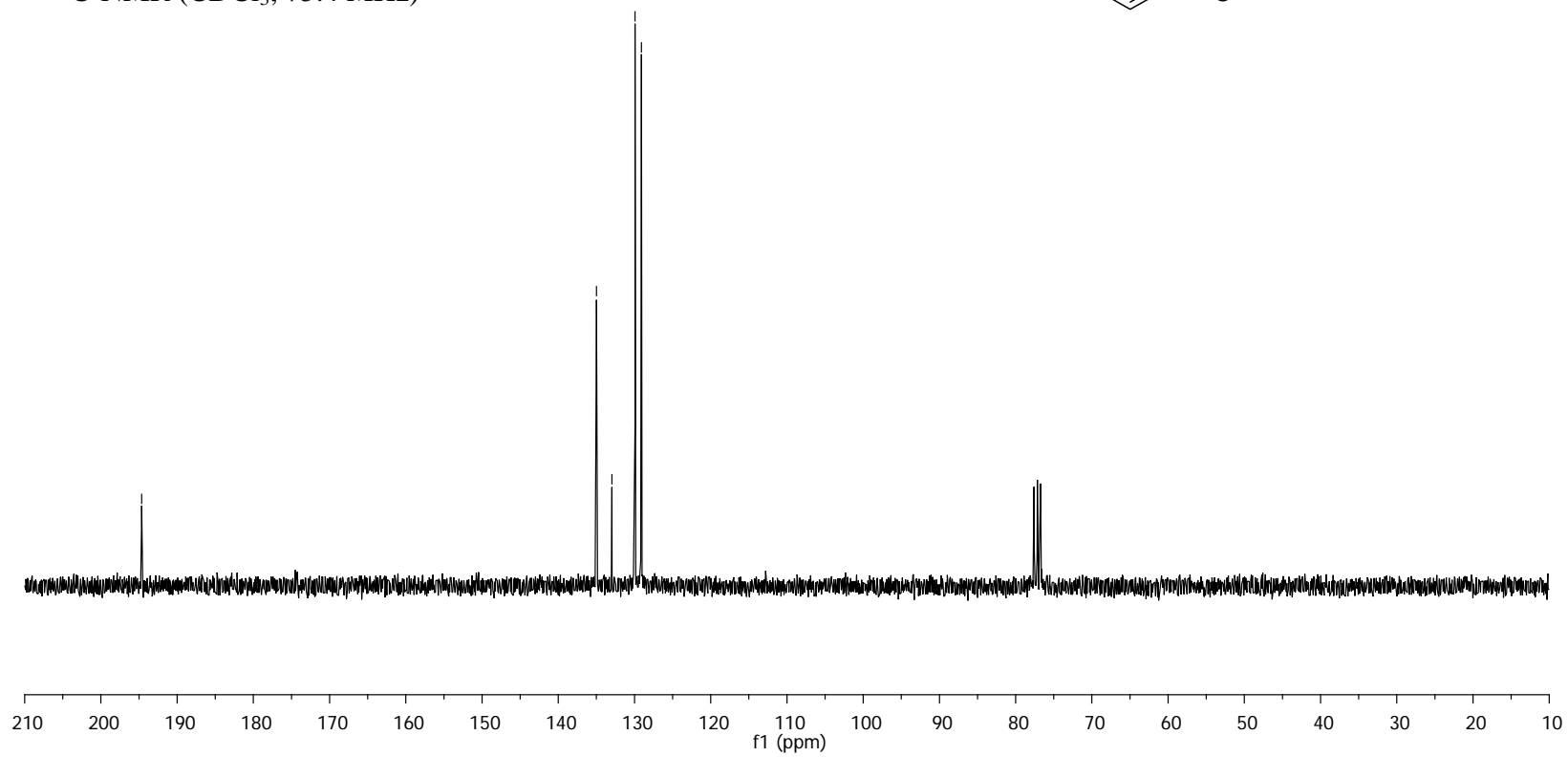
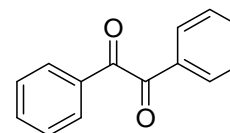
— 194.66

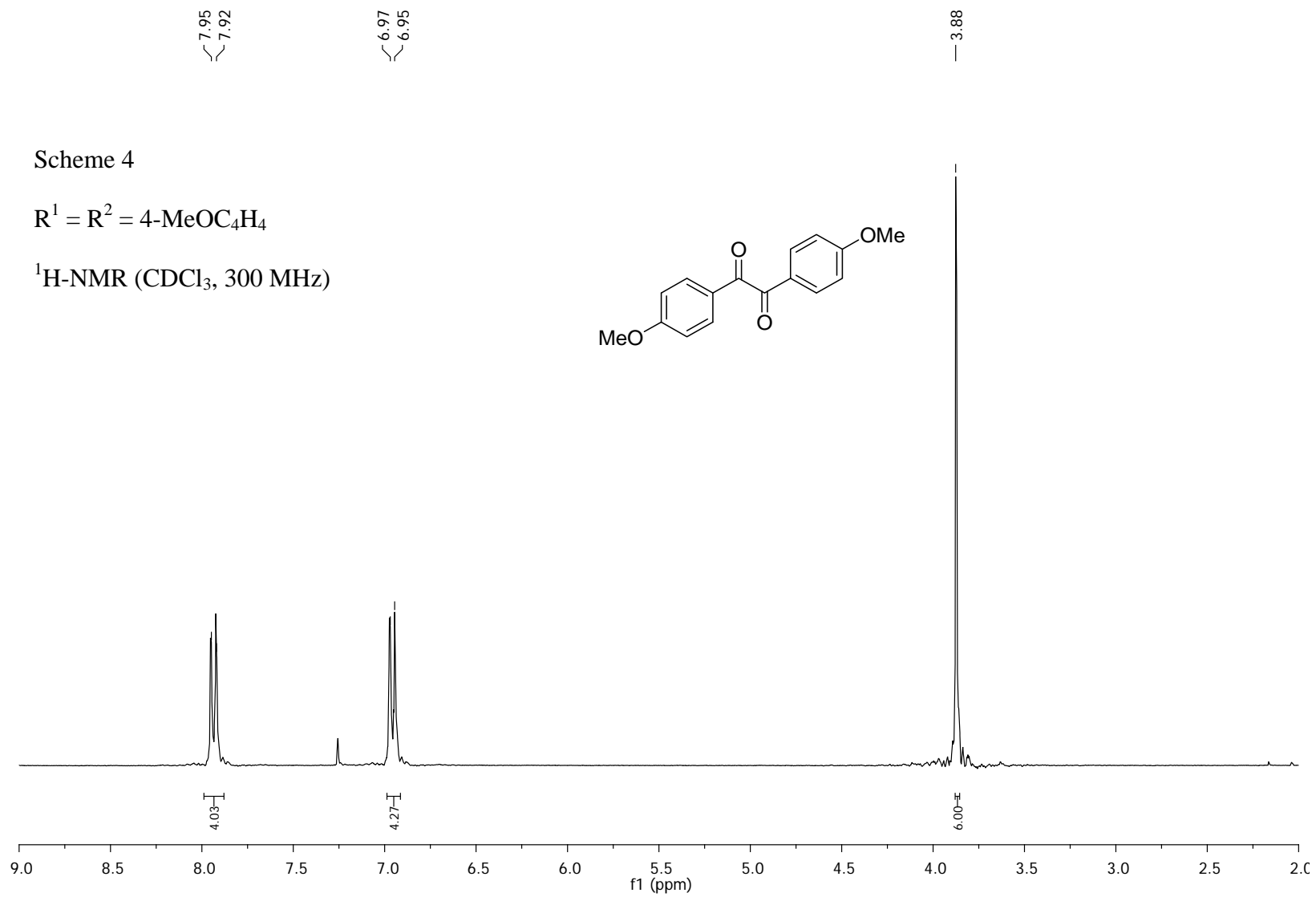
134.99  
132.96  
129.92  
129.09

Scheme 4

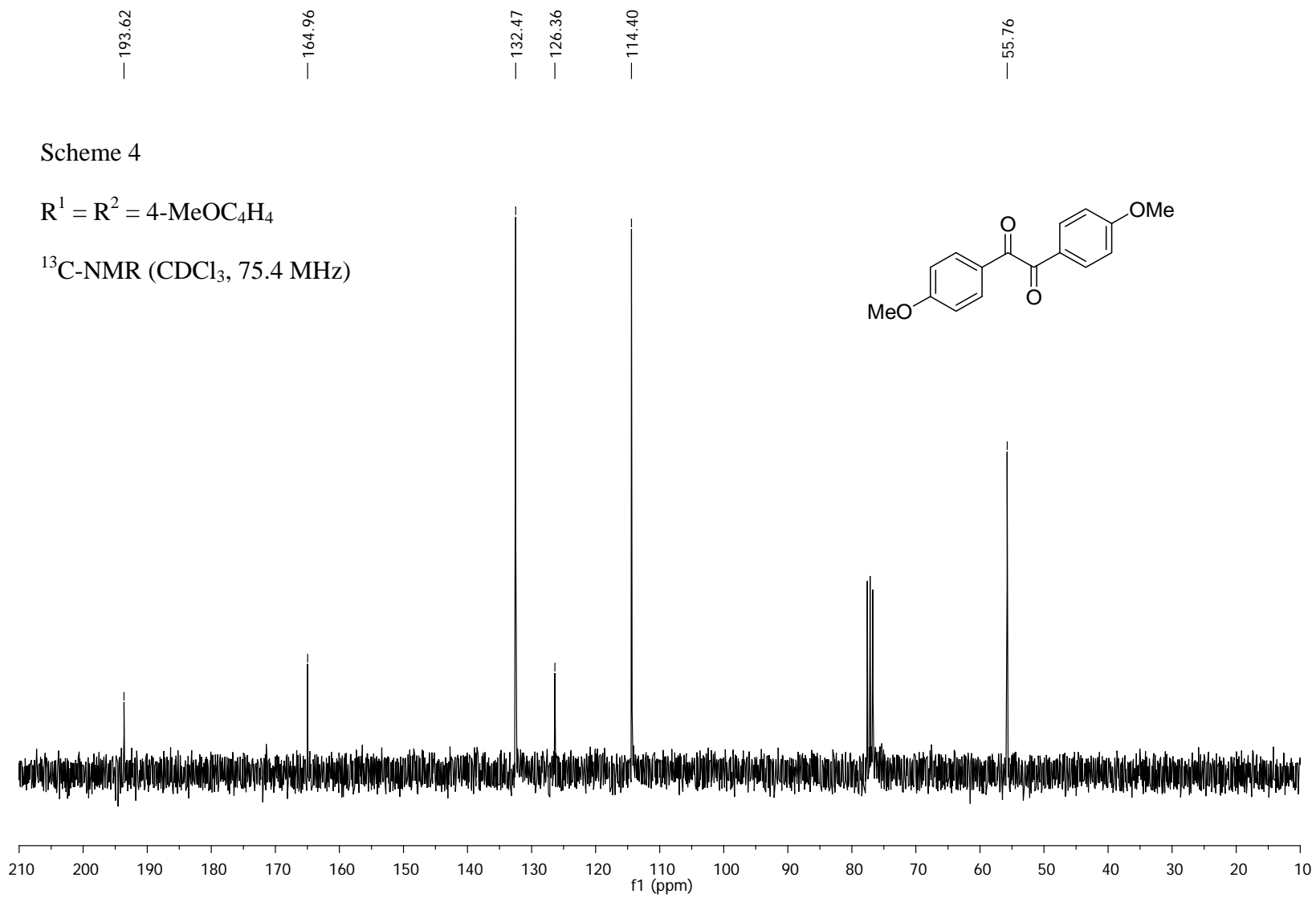
$R^1 = R^2 = \text{Ph}$

$^{13}\text{C}$ -NMR ( $\text{CDCl}_3$ , 75.4 MHz)







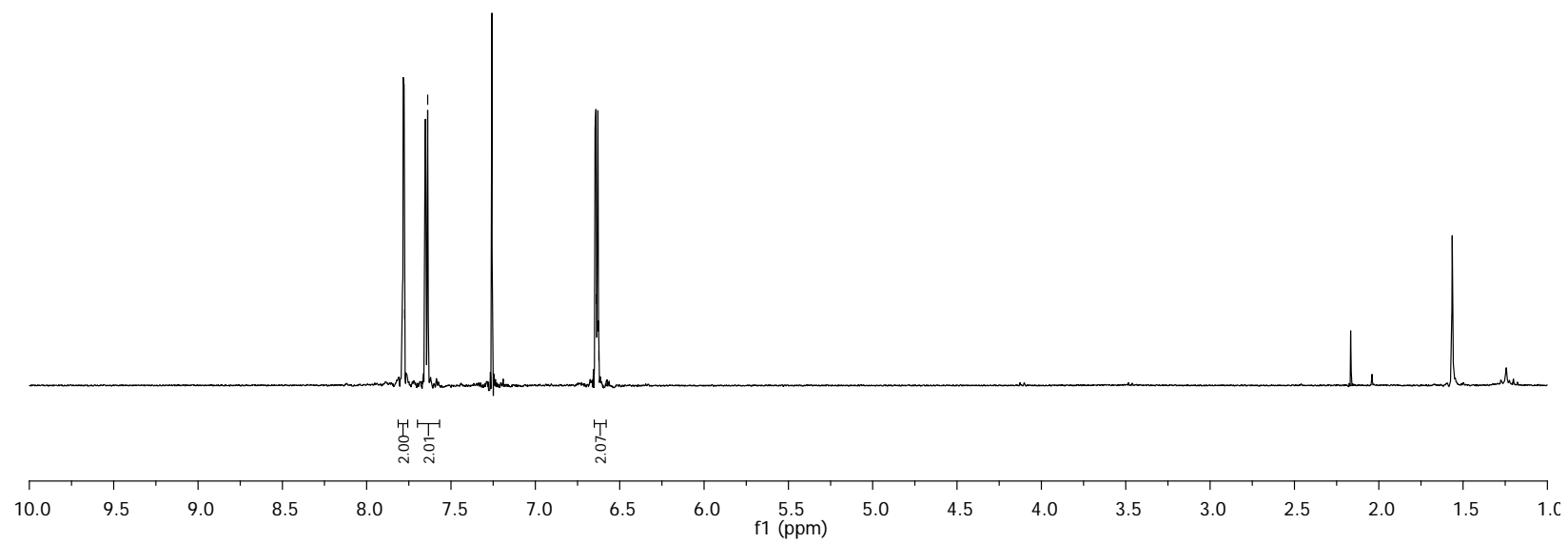
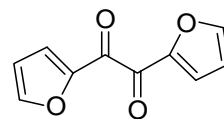


7.77  
7.65  
7.64  
6.64  
6.62

Scheme 4

$R^1 = R^2 = 2\text{-Fu}$

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)

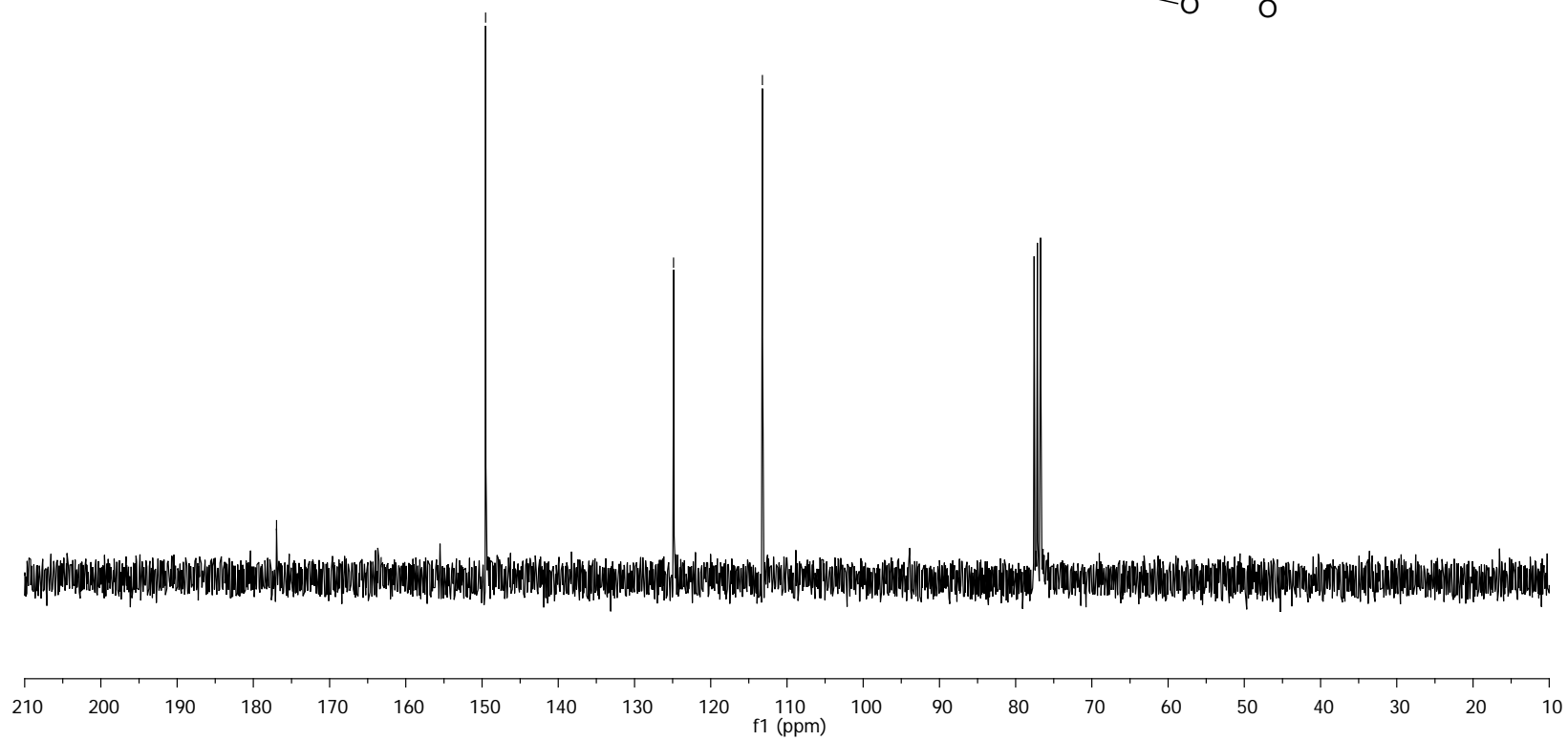
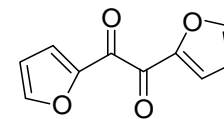


— 176.95  
— 163.25  
— 149.53  
— 124.87  
— 113.22

Scheme 4

$R^1 = R^2 = 2\text{-Fu}$

$^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , 75.4 MHz)

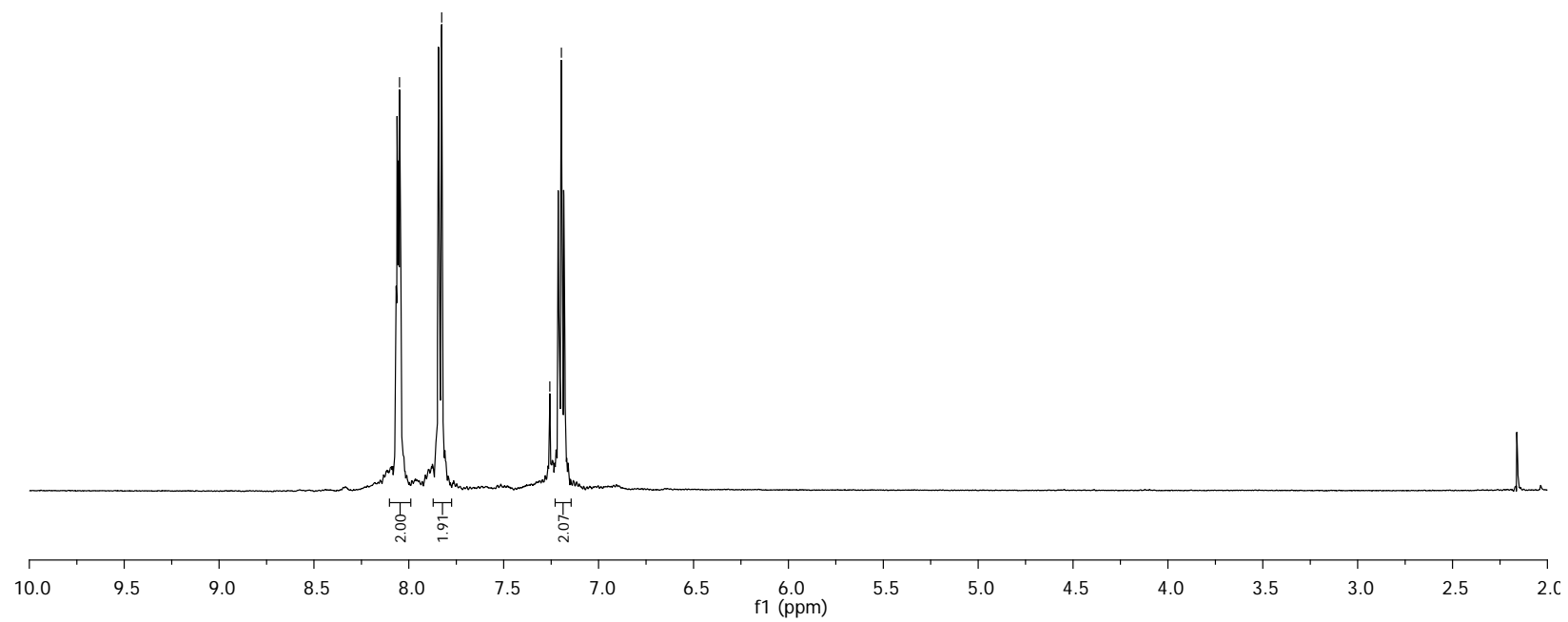
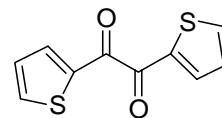


— 8.05  
— 7.83  
~ 7.26  
~ 7.20

Scheme 4

$R^1 = R^2 = 2\text{-Th}$

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)



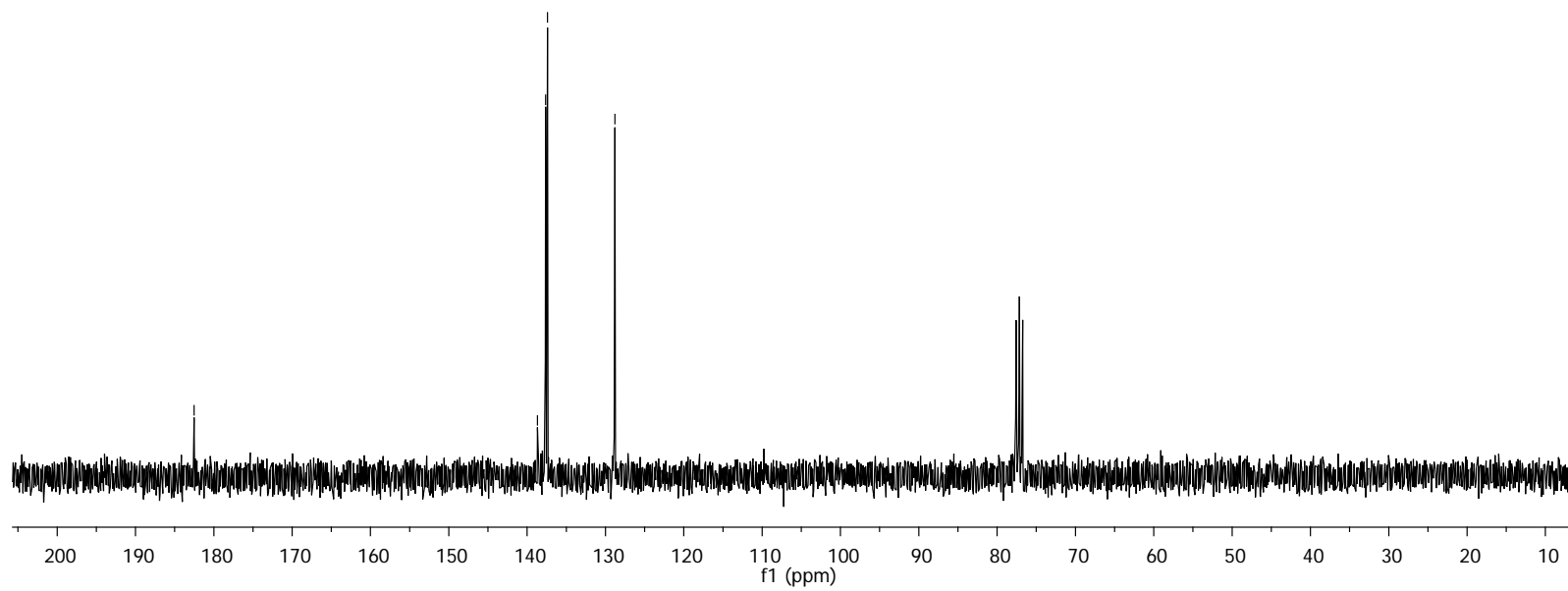
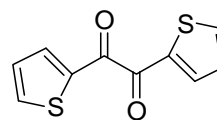
— 182.52

138.68  
137.64  
137.40  
— 128.80

Scheme 4

$R^1 = R^2 = 2\text{-Th}$

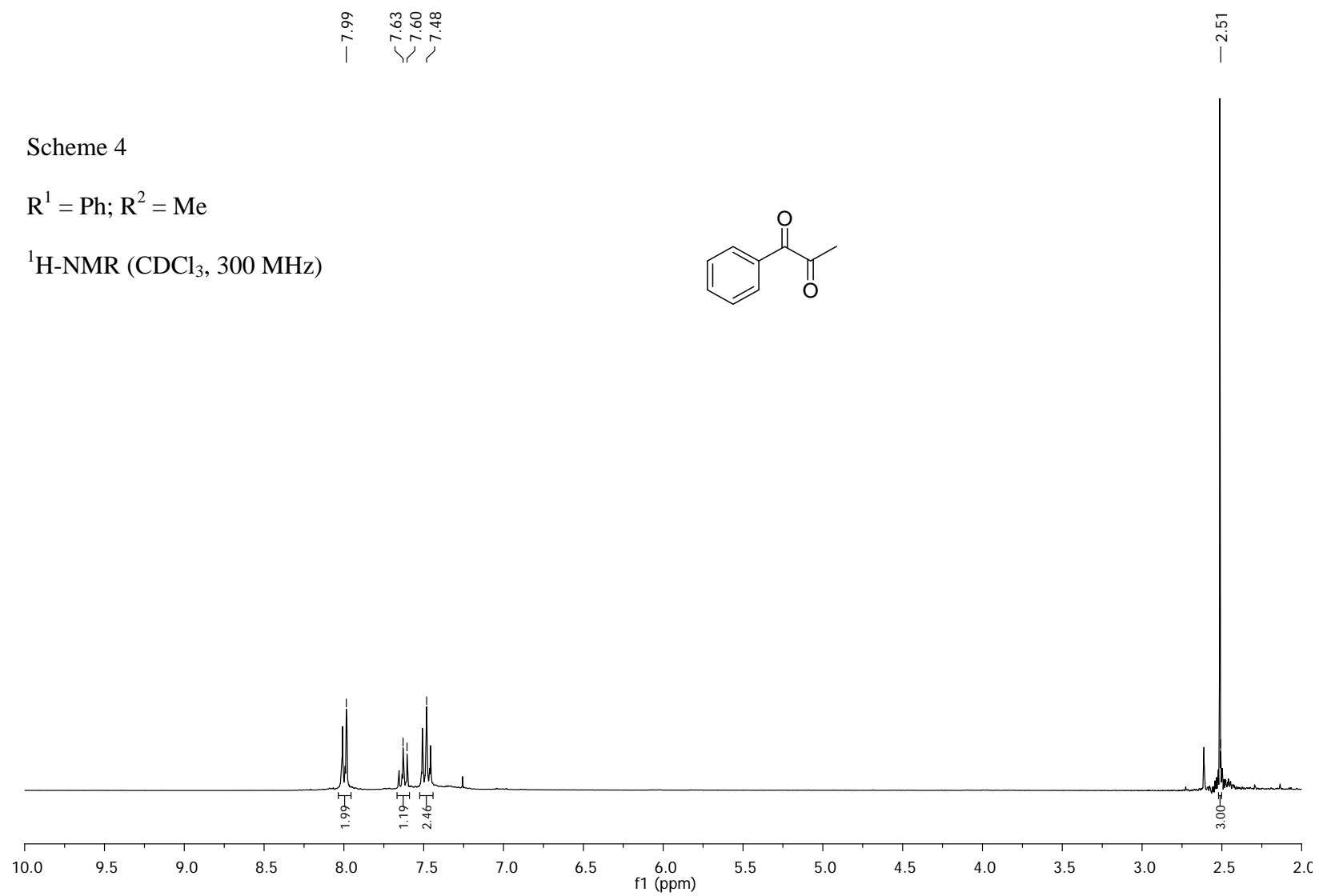
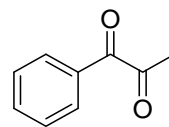
$^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , 75.4 MHz)



Scheme 4

$R^1 = \text{Ph}$ ;  $R^2 = \text{Me}$

$^1\text{H-NMR}$  ( $\text{CDCl}_3$ , 300 MHz)



— 200.67  
— 191.49

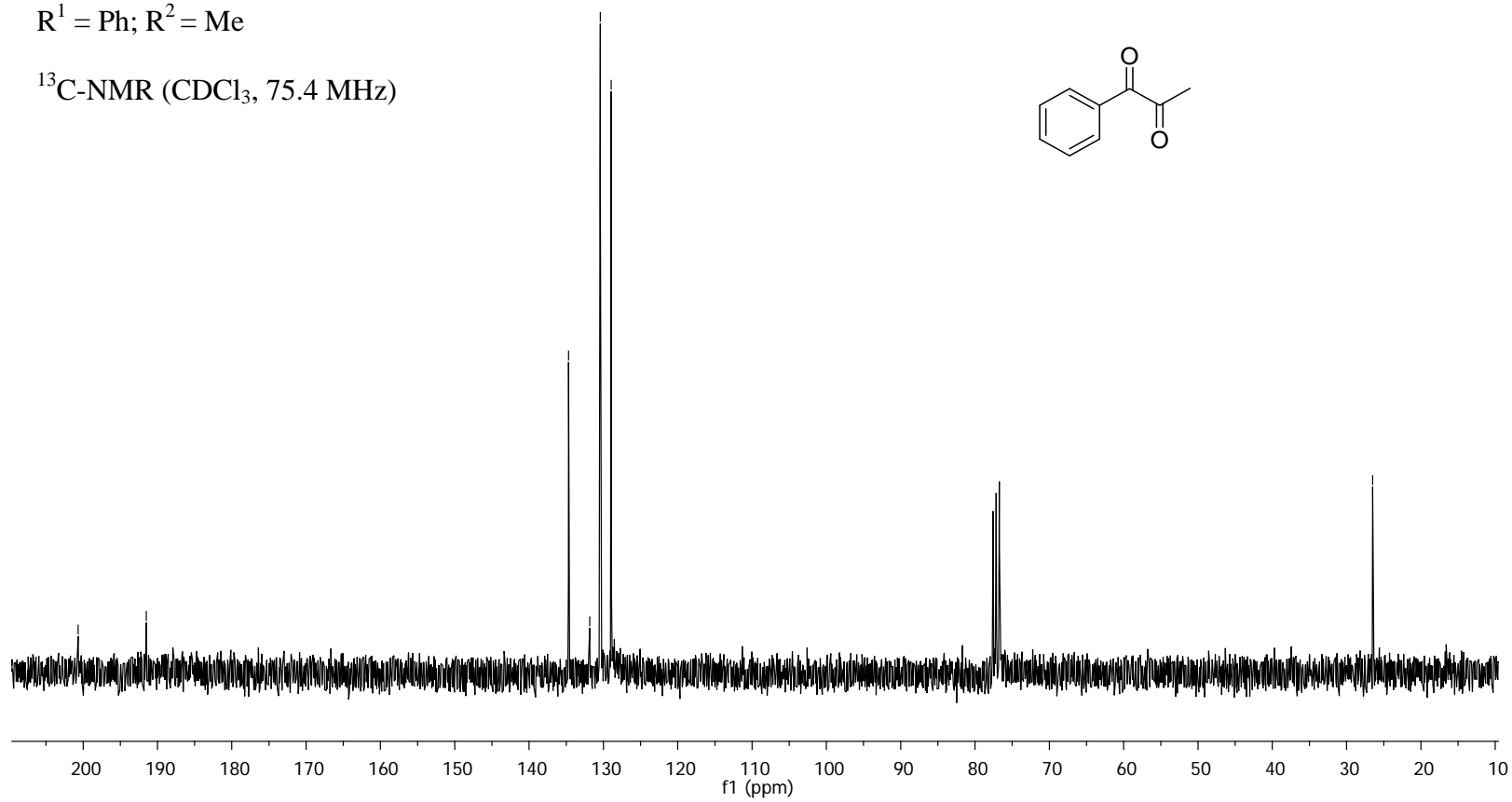
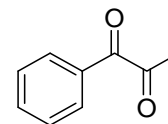
— 134.69  
— 131.85  
— 130.41  
— 128.94

— 26.49

Scheme 4

$R^1 = \text{Ph}; R^2 = \text{Me}$

$^{13}\text{C-NMR}$  ( $\text{CDCl}_3$ , 75.4 MHz)



## Selection of GC-MS chromatograms and spectra

Table 2,  
Entry 1

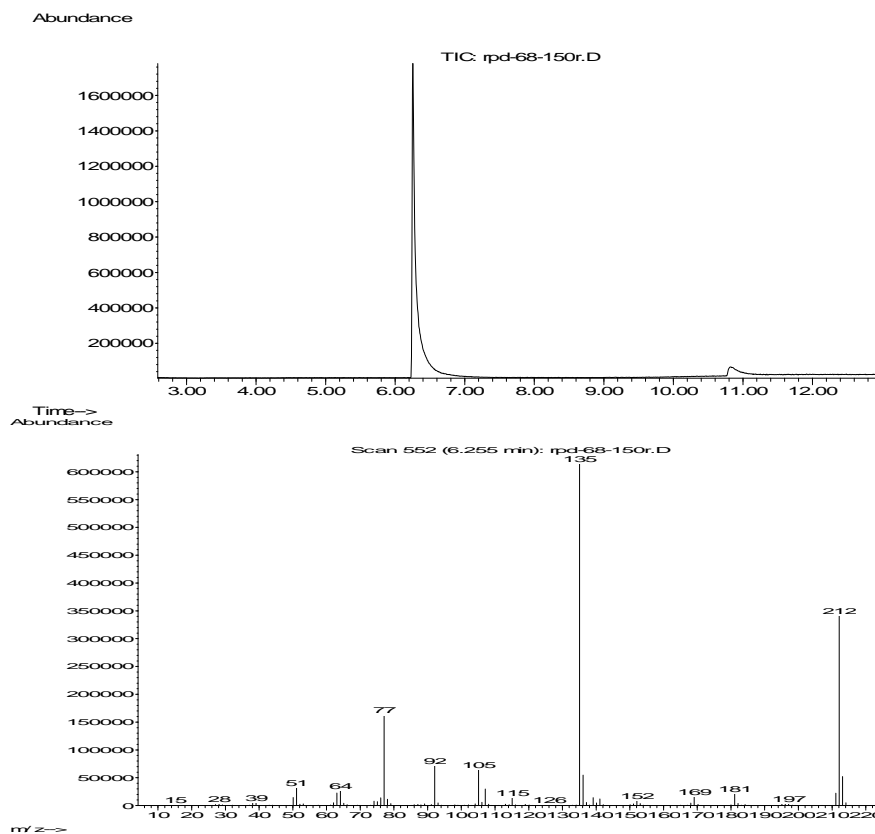
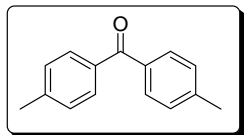


Table 2,  
Entry 2

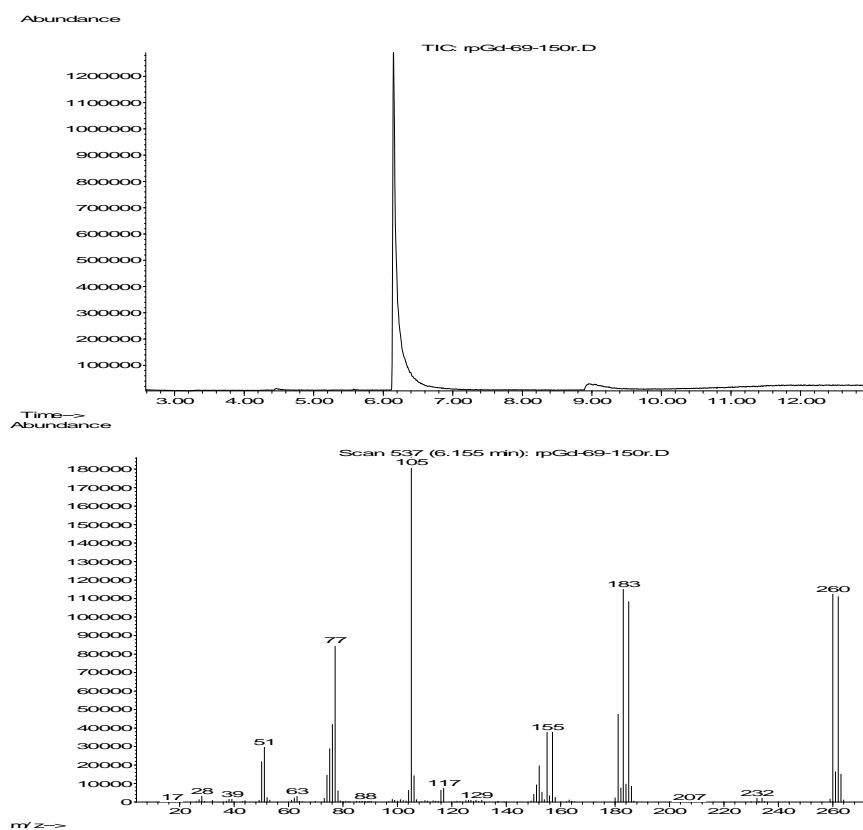
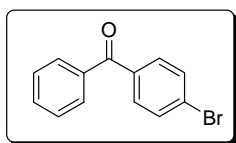




Table 2,  
Entry 3

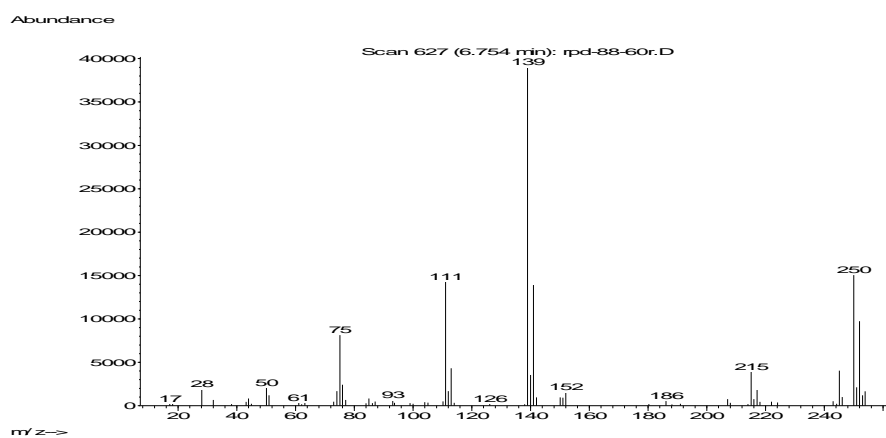
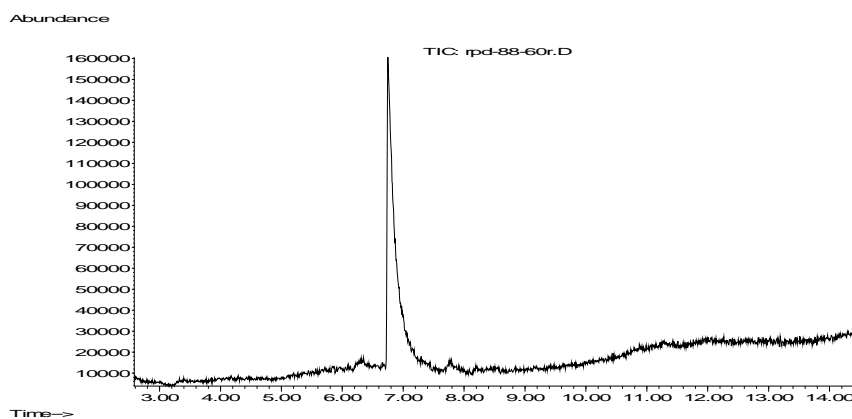
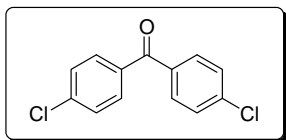


Table 2,  
Entry 7

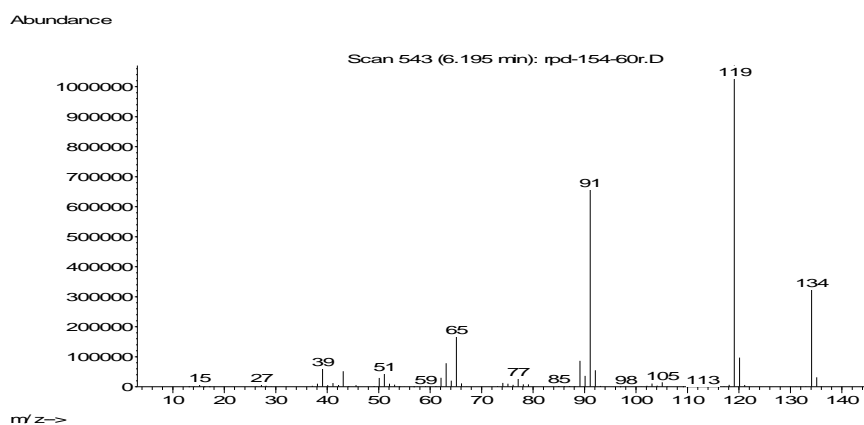
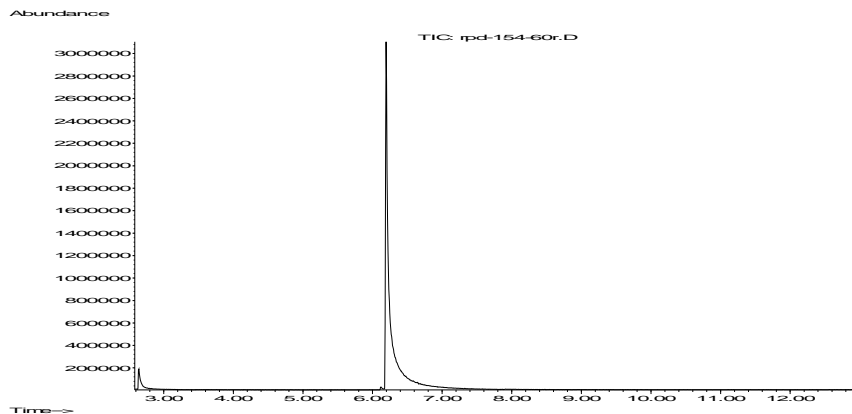
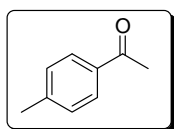


Table 2,  
Entry 8

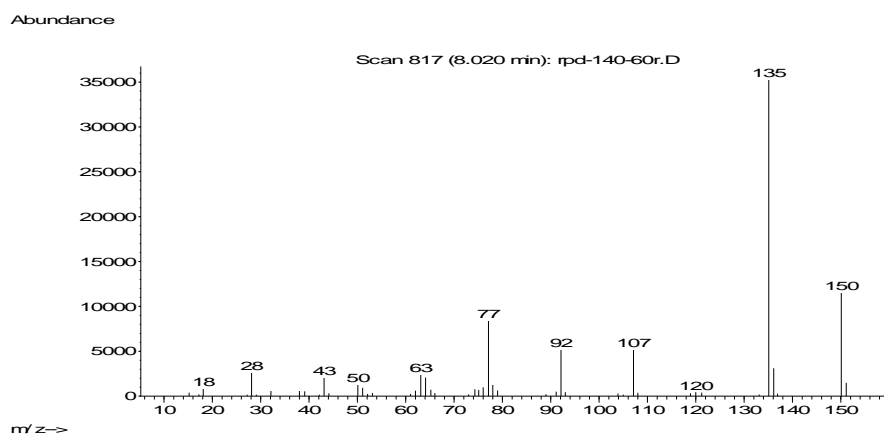
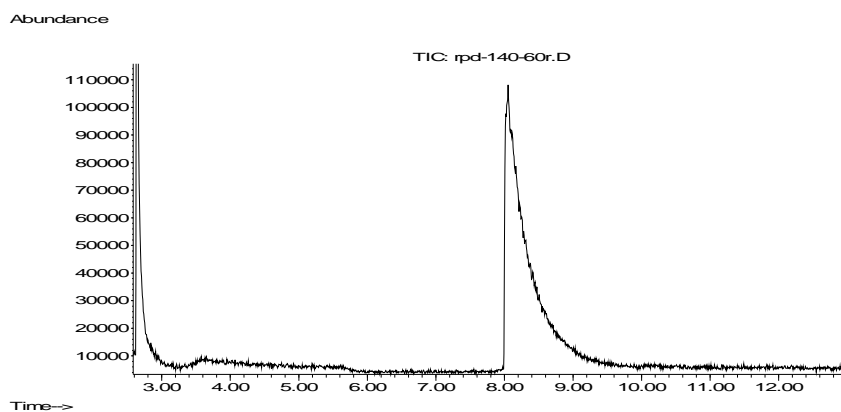
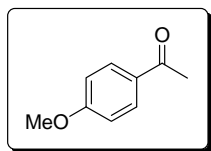


Table 2,  
Entry 9

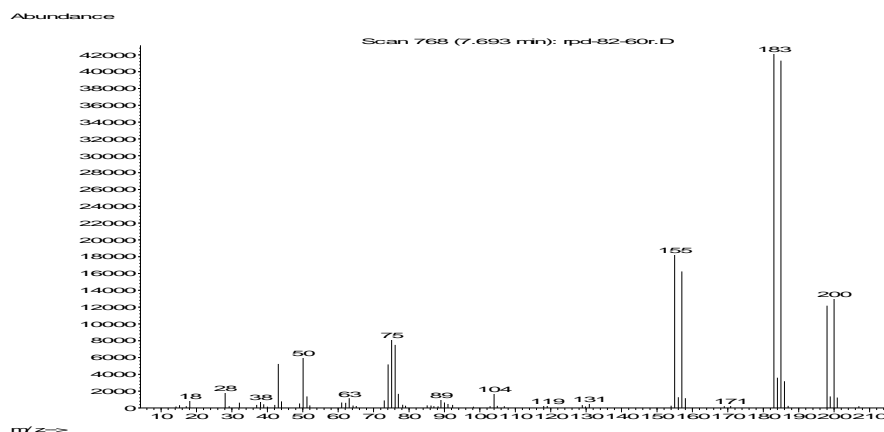
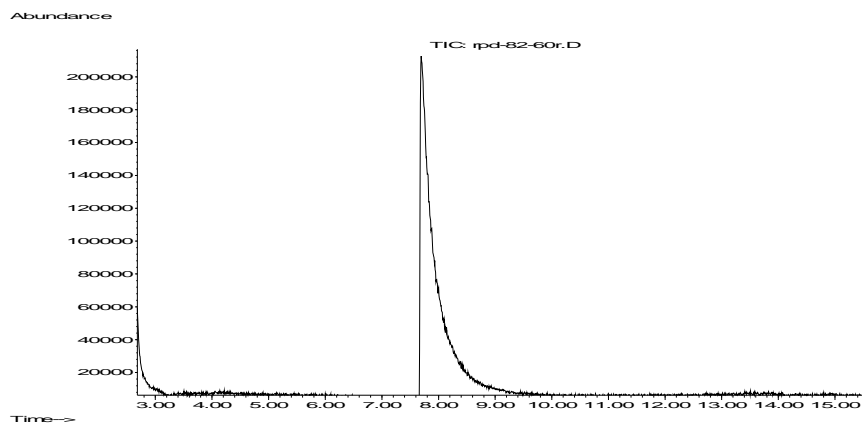
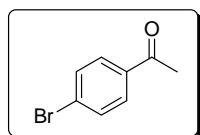


Table 2,  
Entry 10

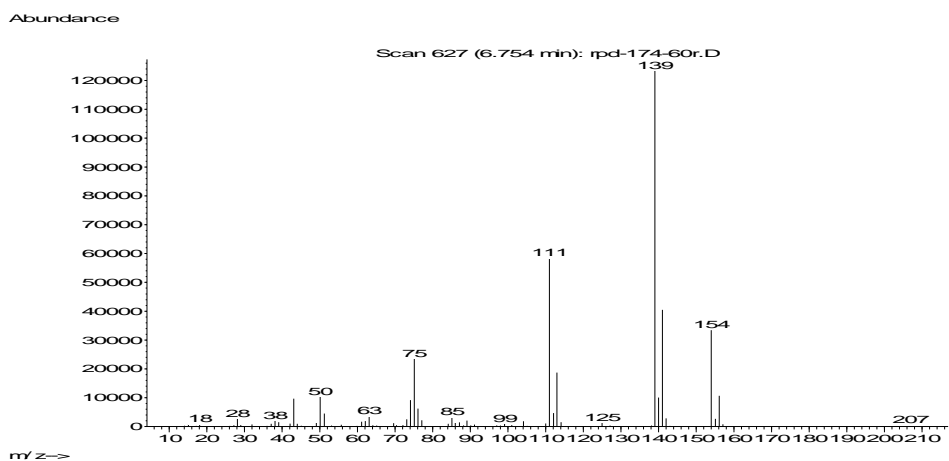
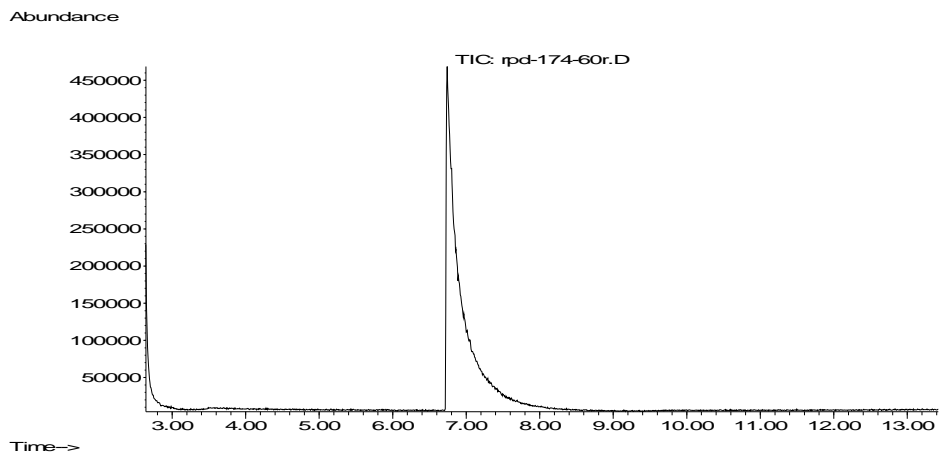
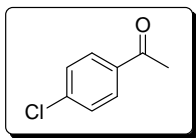


Table 2,  
Entry 11

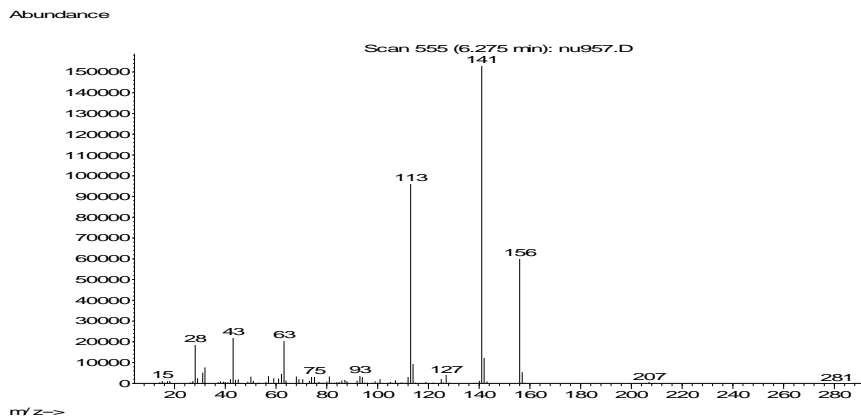
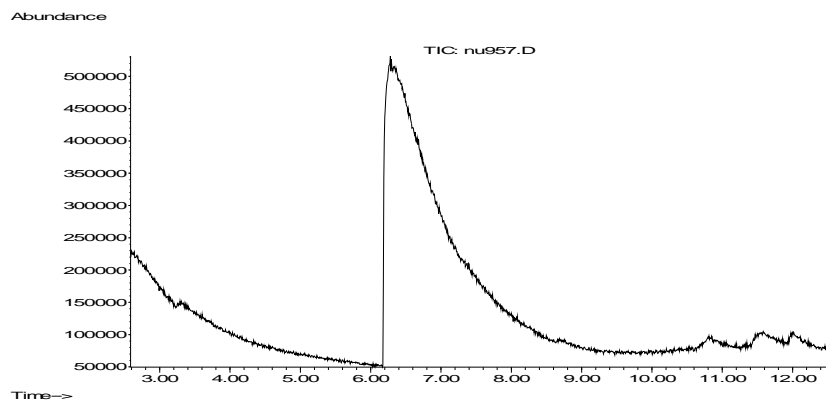
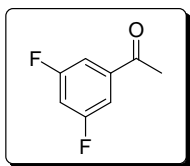


Table 2,  
Entry 13

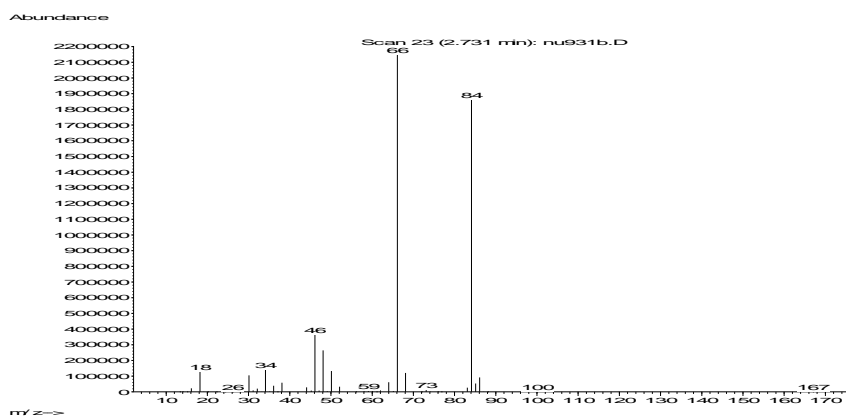
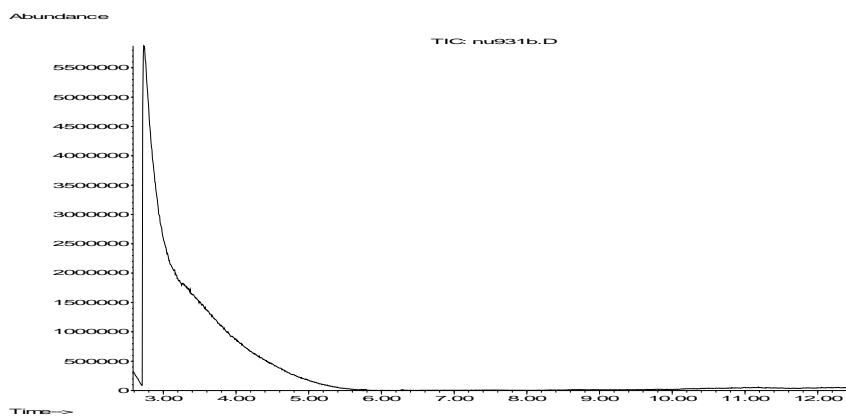
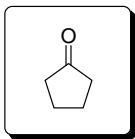


Table 2,  
Entry 14

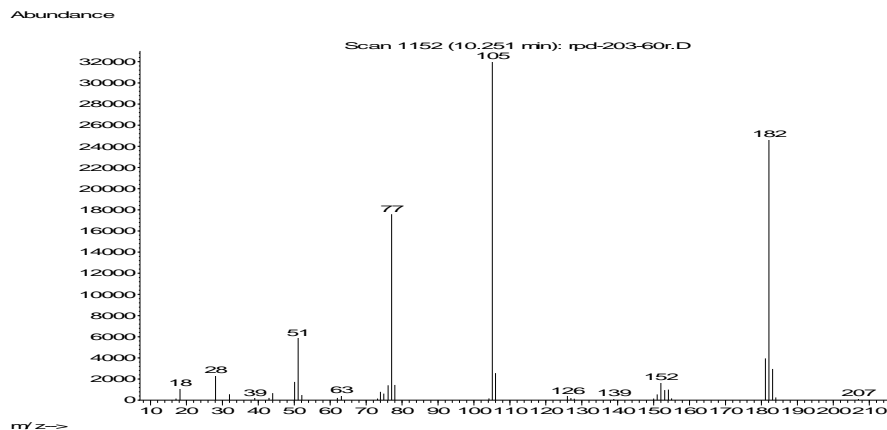
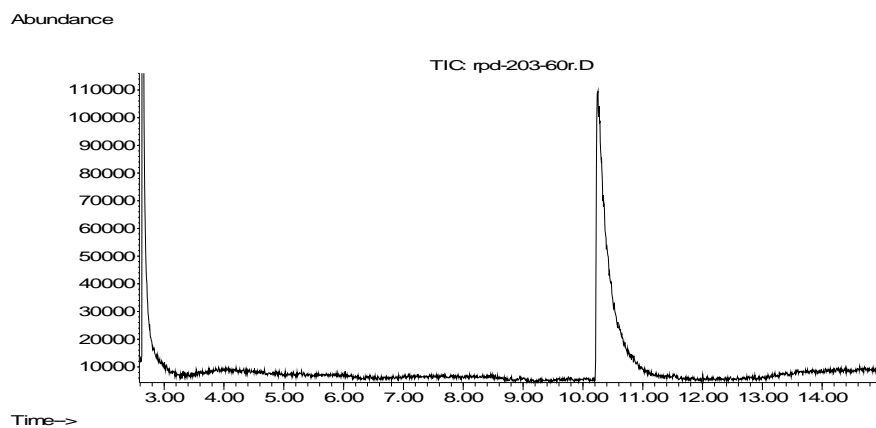
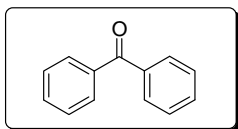


Table 2,  
Entry 16

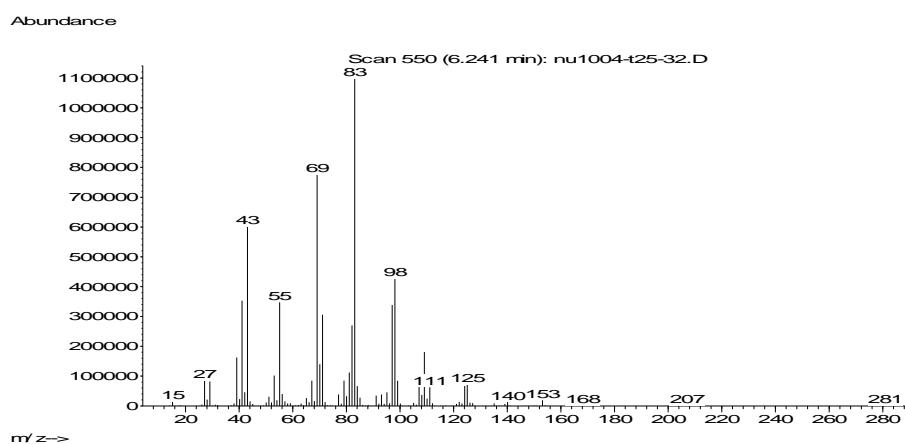
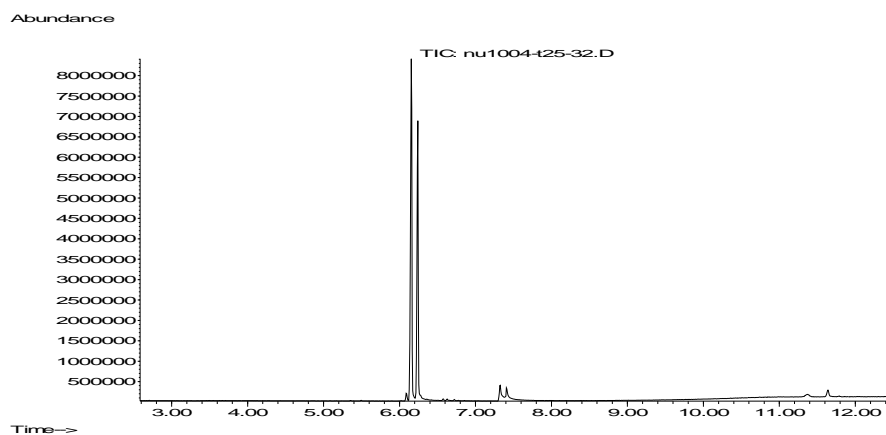
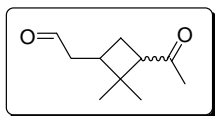


Table 2,  
Entry 17

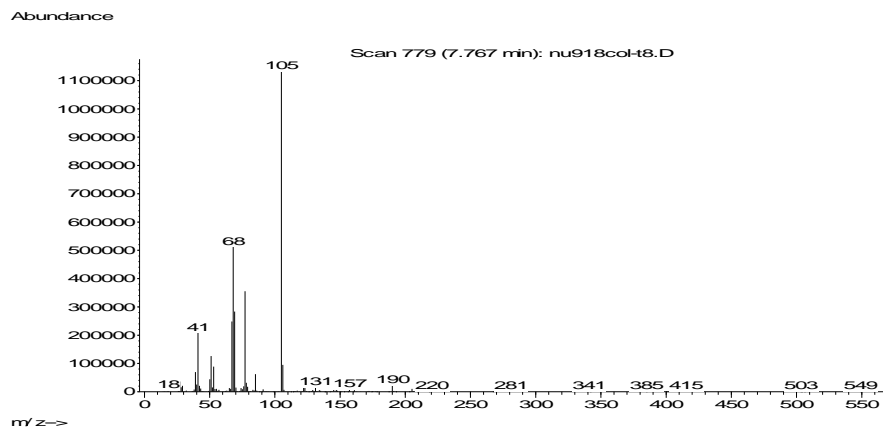
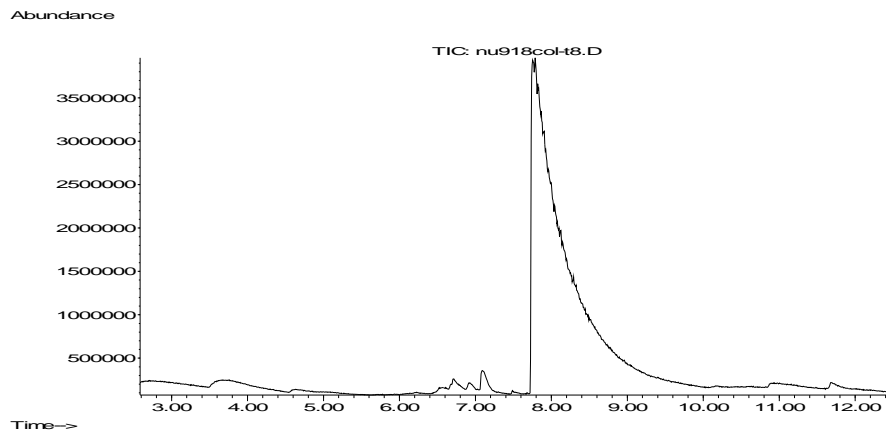
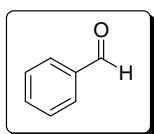


Table 2,  
Entry 18

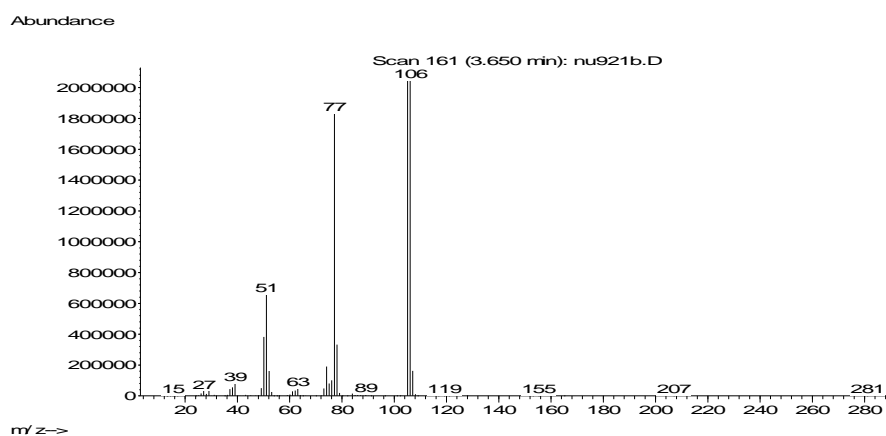
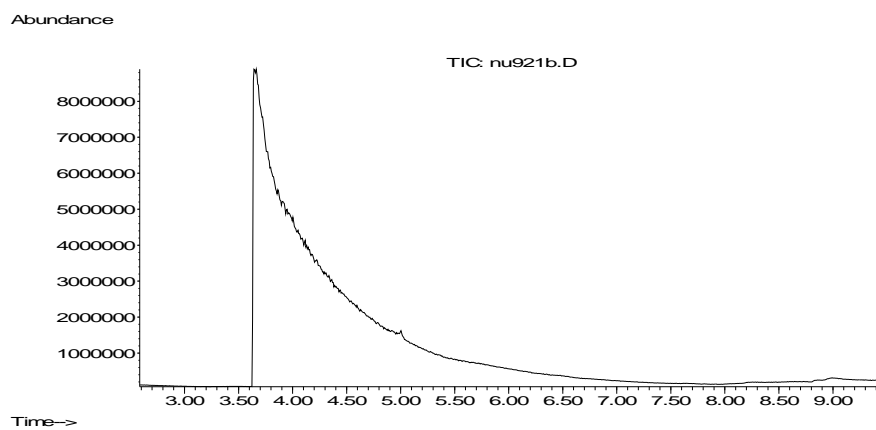
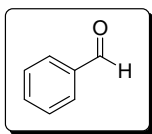


Table 2,  
Entry 21

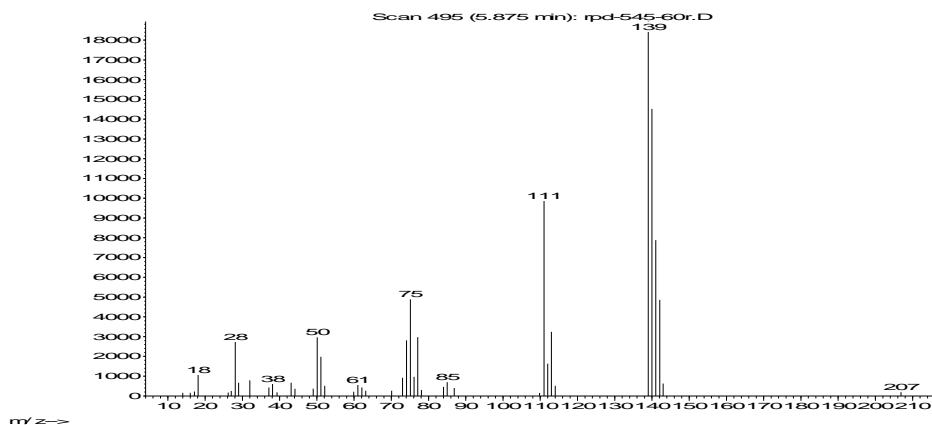
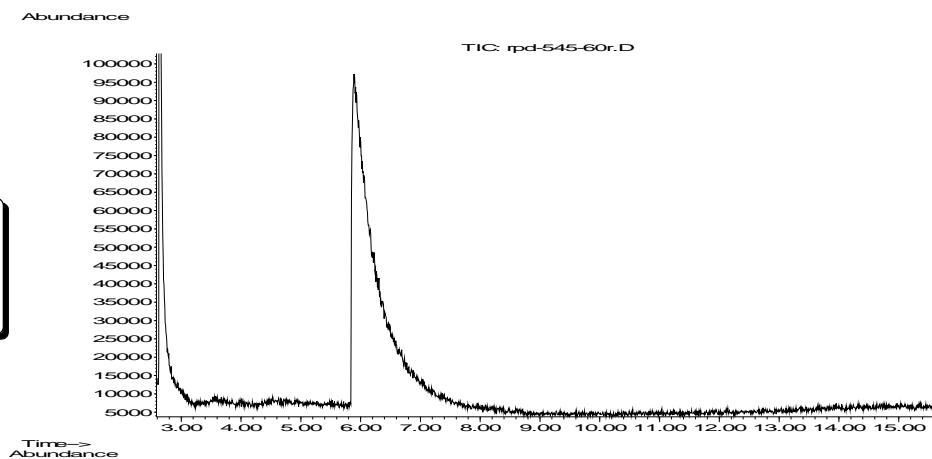
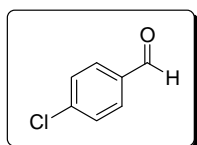


Table 2,  
Entry 22

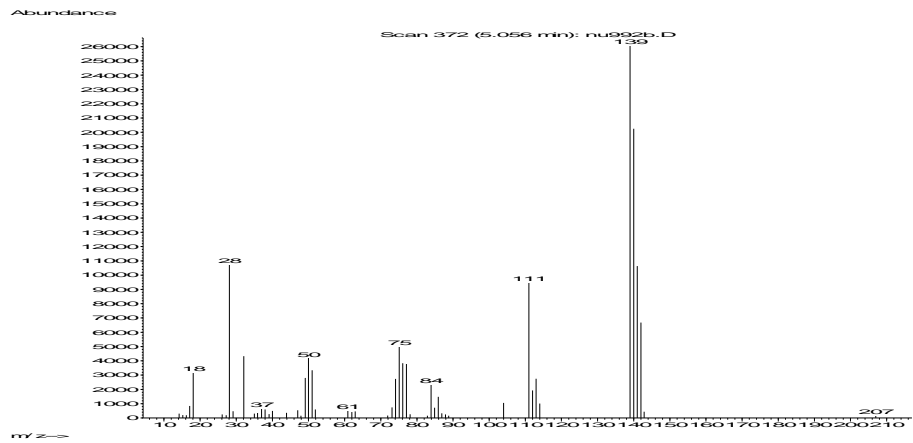
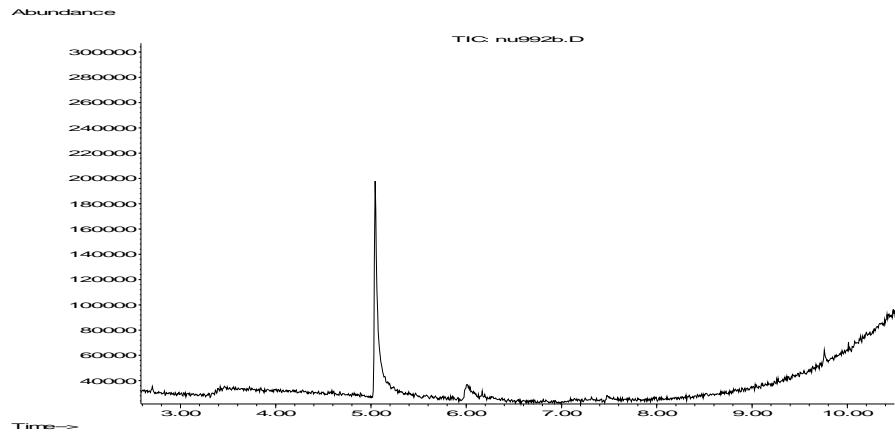
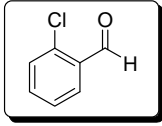


Table 2,  
Entry 23

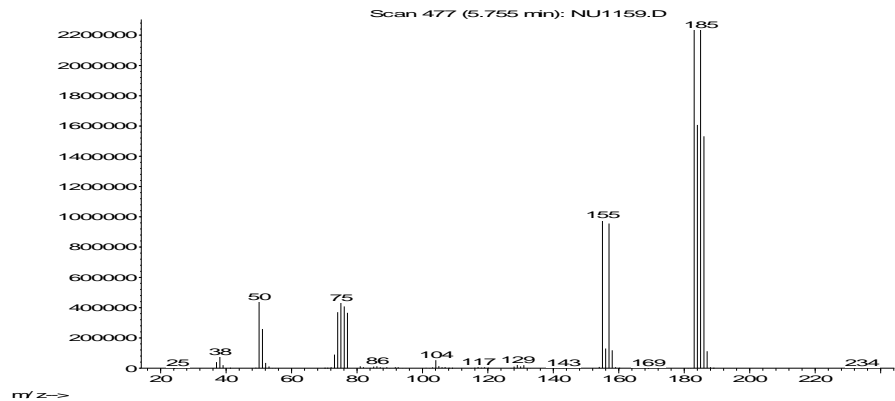
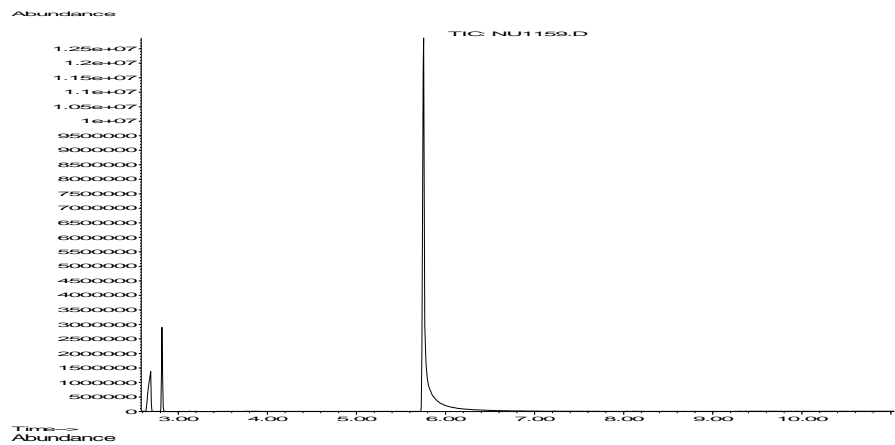
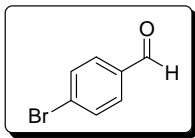


Table 2,  
Entry 25

