

Supporting Information

Metal-Free Direct Amidation of Alkynes Using *N*-Boc-*O*-tosyl-hydroxylamine via Cope-type Hydroamination and Beckmann Rearrangement

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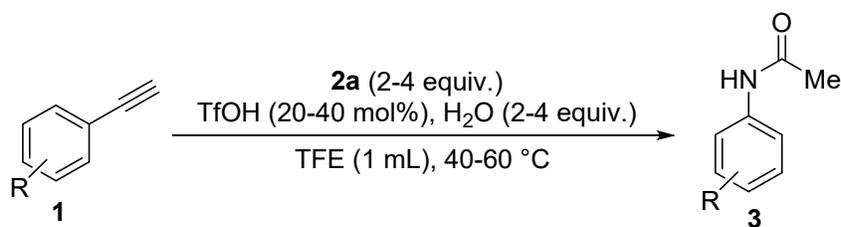
Table of contents

1.	<i>General information</i>	S2
2.	<i>General procedure for the synthesis of amides from terminal alkynes</i>	S2
3.	<i>General procedure for the synthesis of amides from internal alkynes</i>	S3
4.	<i>Synthesis of phenacetin</i>	S3
5.	<i>Characterization data of amides</i>	S4-S8
6.	<i>References</i>	S8-S9
7.	<i>Copies of ¹H and ¹³C NMR spectra</i>	S11-S56

1. General Information:

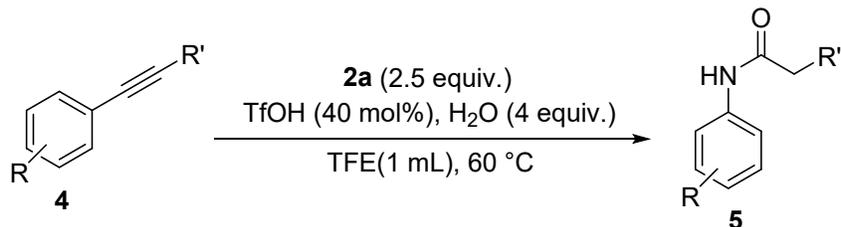
Unless otherwise stated, the starting materials alkynes (**1a-1i**, **1k-1s**, **1u'-1x**, **4a-4c** and **4h**), TfOH, TFA, MeSO₃H, all solvents and reagent HOSA (Hydroxylamine-*O*-sulfonic acid) were purchased from commercial suppliers (Aldrich, TCI, and Alfa Aesar) and were used without further purification, and reactions were carried out under open atmosphere. Starting materials alkynes (**1j**^{1a}, **1t**^{1b}-**1u**^{1c} and (**4d-4g**)^{1d}) were synthesized from reported literature. The aminating agent TsONHBoc^{2a}, (*O*-(2-pyrimidinyl)-*N*-Boc hydroxylamine) PymONHBoc^{2b} was synthesized from reported literature and stored at RT. TLC was carried out on precoated plates (Merck silica gel 60, F₂₅₄), and the spots were visualized with UV light and by charring the plates dipped in DNP, KMnO₄ solution. NMR spectra were recorded on 400 MHz Bruker/Jeol instrument in CDCl₃ or DMSO-*d*₆ as the solvent. In the ¹H and ¹³C spectra, chemical shifts are reported relative to tetramethylsilane (δ 0.00) or (CDCl₃: 7.26/77.2 ppm; DMSO-*d*₆: 2.50/39.5 ppm). ¹H NMR splitting patterns are designated as singlet (s), doublet (d), triplet (t), quartet (q), dd (doublet of doublets); m (multiplets), and etc. All first-order splitting patterns were assigned on the basis of the appearance of the multiplet.

2. General procedure for the synthesis of *N*-aryl acetamides from terminal alkynes:



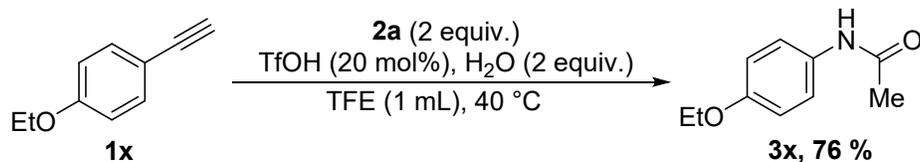
N-Boc-*O*-tosylhydroxylamine **2a** (2-4 equiv.) was added to a solution of alkynes **1** (0.5 mmol, 1.0 equiv.) in TFE (1 mL) and H₂O (1-2 mmol, 2.0 - 4.0 equiv., 18 μ L) at room temperature, followed by the addition of TfOH (20 - 40 mol%, 0.1 - 0.2 mmol, 9-18 μ L) and the reaction mixture was stirred at 40 °C or 60 °C for a specified time. The progress of the reaction was monitored by TLC. After completion, the solvent of reaction mixture evaporated under reduced pressure. The crude obtained was purified through silica gel column chromatography using ethyl acetate/hexane as eluent.

3. General procedure for the synthesis of amides from internal alkynes:



N-Boc-*O*-tosylhydroxylamine **2a** (1 equiv.) was added to the solution of alkynes **4** (0.5 mmol, 1.0 equiv.) in TFE (1 mL) and H₂O (2 mmol, 4.0 equiv., 36 μL) at room temperature followed by the addition of TfOH (40 mol%, 0.2 mmol, 18 μL) and the reaction mixture was stirred at 60 °C for 1 hour. Further, 1.5 equiv. of **2a** was added and the reaction mixture was stirred at 60 °C till completion of the reaction. Progress of the reaction was monitored by TLC. After completion, solvent of the reaction mixture was evaporated under reduced pressure. The crude obtained was purified through silica gel column chromatography using ethyl acetate/hexane as eluent.

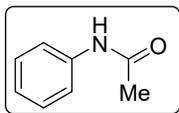
4. Procedure for the synthesis of Phenacetin:



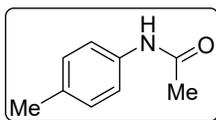
N-Boc-*O*-tosylhydroxylamine **2a** (2 equiv.) was added to a solution of *para*-ethoxy phenyl acetylene **1x** (0.5 mmol, 1.0 equiv.) in TFE (1 mL) and H₂O (1 mmol, 2.0 equiv., 18 μL) at room temperature, followed by the addition of TfOH (20 mol%, 0.1 mmol, 9 μL) and the reaction mixture was stirred at 40 °C for a 16 h. The progress of the reaction was monitored by TLC. After completion, the solvent of reaction mixture evaporated under reduced pressure. The crude obtained was purified through silica gel column chromatography using 30% ethyl acetate/hexane as eluent to obtain phenacetin **3x** in 76% yield.

Phenacetin (**3x**)³: 73% yield (40 mg), White solid, 76% yield (63 mg), hexane:ethyl acetate (70:30) as the eluent, mp 133–135 °C (lit. mp 133–136 °C).

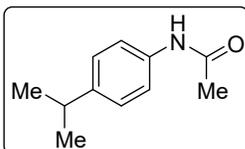
5. Characterization data of products:



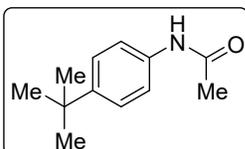
N-acetyl amide (**3a**):⁴ 84% yield (57 mg), White solid, mp 115–119 °C (lit. mp 115–120 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



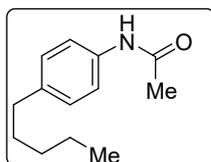
N-(4-methylphenyl)acetamide (**3b**):⁴ 81% yield (60 mg), White solid, mp 148–152 °C (lit. mp 145–147 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



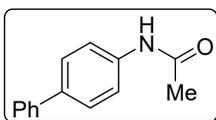
N-(4-isopropylphenyl)acetamide (**3c**):⁵ 83% yield (74 mg), mp 95–98 °C (lit. mp 96–100 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



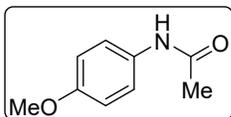
N-(4-*t*-Butylphenyl)acetamide (**3d**):⁴ 68% yield (65 mg), White solid, mp 150–152 °C (lit. mp 150–152 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



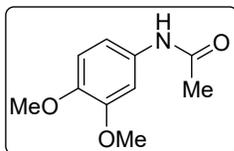
N-(4-pentylphenyl)acetamide (**3e**): 92% yield (95 mg), White solid, mp 102–105 °C, hexane:ethyl acetate (75:25) as the eluent; ¹H NMR (400 MHz, CDCl₃) δ 7.72 (br s, 1H), 7.39 (d, *J* = 8.1 Hz, 2H), 7.10 (d, *J* = 8.0 Hz, 2H), 2.55 (t, *J* = 7.7 Hz, 2H), 2.13 (s, 3H), 1.62 – 1.53 (m, 2H), 1.38 – 1.29 (m, 4H), 0.88 (t, *J* = 6.7 Hz, 3H); ¹³C {¹H} NMR (101 MHz, CDCl₃) δ 168.64, 139.07, 135.56, 128.82, 120.16, 35.34, 31.45, 31.21, 24.44, 22.55, 14.07; HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₁₃H₁₉NO [M+H]⁺ 206.147, found 206.150



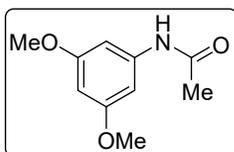
N-([1,1'-biphenyl]-4-yl)acetamide (**3f**):⁴ 75% yield (79 mg), Light brown solid, mp 168–170 °C (lit. mp 165–170 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



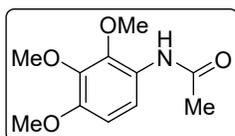
N-(4-methoxyphenyl)acetamide (**3g**):⁴ 76% yield (63 mg), White solid, mp 125–130 °C (lit. mp 128–130 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



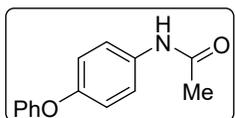
N-(3,4-dimethoxyphenyl)acetamide (**3h**):⁹ 74% yield (80 mg), White solid, mp 126–130 °C (lit. mp 128–130 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



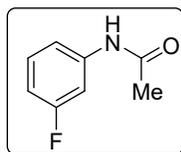
N-(3,5-dimethoxyphenyl)acetamide (**3i**):⁴ 61% yield (60 mg), White solid, mp 142–145 °C (lit. mp 143–145 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



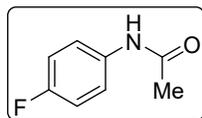
N-(2,3,4-trimethoxyphenyl)acetamide (**3j**): 71% yield (80 mg), White solid, mp 80–84 °C, hexane:ethyl acetate (60:40) as the eluent. ¹H NMR (400 MHz, CDCl₃): δ 7.98 (d, *J* = 9.1 Hz, 1H), 7.60 (br s, 1H), 6.62 (d, *J* = 9.1 Hz, 1H), 3.93 (s, 3H), 3.86 (s, 3H), 3.83 (s, 3H), 2.18 (s, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 168.13, 149.73, 142.75, 141.61, 125.33, 114.92, 106.99, 61.16, 60.90, 56.13, 24.74; HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₁₁H₁₅NO₄ [M+H]⁺ 226.100, found 226.104.



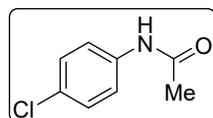
N-(4-phenoxyphenyl)acetamide (**3k**):⁵ 79% yield (90 mg), White solid, mp 107–110 °C (lit. mp 108–110 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



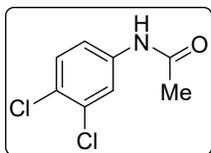
N-(3-Fluorophenyl)acetamide (**3l**):⁴ 53% yield (41 mg), Colourless solid, mp 85–88 °C (lit. mp 84–86 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



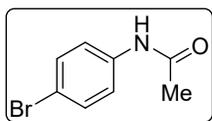
N-(4-Fluorophenyl)acetamide (**3m**):⁴ 52% yield (40 mg), Colourless solid, mp 155–157 °C (lit. mp 153–155 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



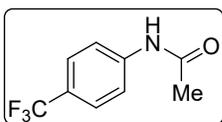
N-(4-Chlorophenyl)acetamide (**3n**):⁴ 81% yield (69 mg), White solid, mp 176–178 °C (lit. mp 175–178 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



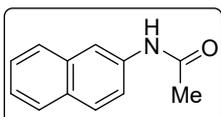
N-(3,4-dichlorophenyl)acetamide (**3o**):⁴ 39% yield (40 mg), Light yellowish solid, mp 128-130 °C (lit. mp 125-128 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



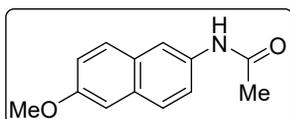
N-(4-bromophenyl)acetamide (**3p**):⁴ 74% yield (80 mg), White solid, mp 168-172 °C (lit. mp 168-170 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



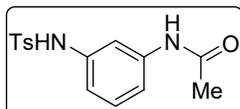
N-(4-trifluoromethylphenyl)acetamide (**3q**):⁴ 45% yield (46 mg), White solid, mp 108-110 °C (lit. mp 106-108 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



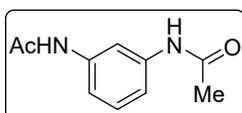
N-(naphthalen-2-yl)acetamide (**3r**):⁴ 65% yield (60 mg), White solid, mp 137-138 °C (lit. mp 138-140 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



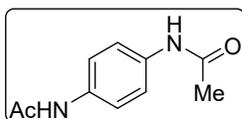
N-(6-methoxynaphthalen-2-yl)acetamide (**3s**):⁸ 80% yield (86 mg), White solid, mp 148-150 °C (lit. mp 148-150 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



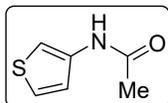
N-(3-((4-methylphenyl)sulfonamido)phenyl)acetamide (**3t**): 62% yield (95 mg), Light yellow solid, hexane:ethyl acetate (40:60) as the eluent, mp 158–160 °C. ¹H NMR (400 MHz, DMSO-*d*₆): δ 10.20 (s, 1H), 9.91 (s, 1H), 7.67 (d, *J* = 7.9 Hz, 2H), 7.45 (s, 1H), 7.33 (d, *J* = 7.9 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 1H), 7.10 (t, *J* = 8.2 Hz, 1H), 6.74 (d, *J* = 8.0 Hz, 1H), 2.33 (s, 3H), 2.00 (s, 3H). ¹³C{¹H} NMR (101 MHz, DMSO-*d*₆) δ 168.77, 143.66, 140.43, 138.65, 137.19, 130.09, 129.67, 127.20, 114.91, 114.74, 110.73, 24.45, 21.41; HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₁₅H₁₆N₂O₃S [M+H]⁺ 305.088, found 305.091.



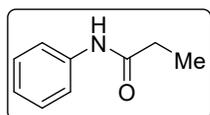
N,N'-(1,3-phenylene)diacetamide (**3u**):⁶ 60% (58 mg) Light yellow solid, from *N*-(3-ethynylphenyl)acetamide & 62% (60mg) from 1,3-diethynylbenzene, ethyl acetate (100%) as the eluent, mp 182–185 °C.



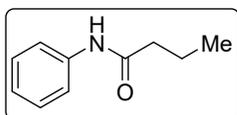
N,N'-(1,4-phenylene)diacetamide (**3v**):⁷ 57% (55 mg), Light yellow solid, ethyl acetate (100%) as the eluent, mp > 300 °C.



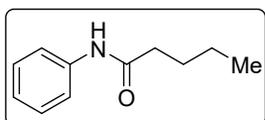
N-(thiophen-3-yl)acetamide (**3w**):⁵ 73% yield (40 mg), White solid, mp 146-148 °C (lit. mp 145-148 °C), purified by column chromatography (hexane:ethyl acetate = 70:30).



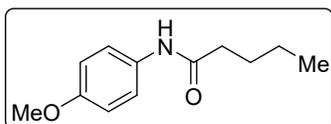
N-phenylpropionamide (**5a**):⁵ 67% yield (50 mg), White solid, mp 105-108 °C (lit. mp 106-108 °C), purified by column chromatography (hexane:ethyl acetate = 75:25).



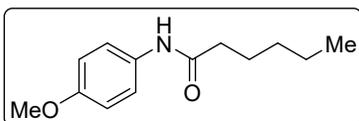
N-phenylbutyramide (**5b**):⁵ 81% yield (66 mg), White solid, mp 95-96 °C (lit. mp 96-97 °C), purified by column chromatography (hexane:ethyl acetate = 80:20).



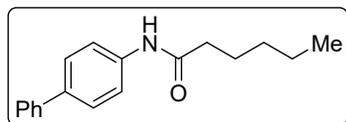
N-phenylpentanamide (**5c**):⁴ 68% yield (60 mg), White solid, mp 60-62 °C (lit. mp 60-63 °C), purified by column chromatography (hexane:ethyl acetate = 80:20).



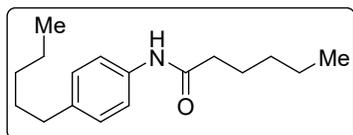
N-(4-methoxyphenyl)pentanamide (**5d**):¹⁰ 74% yield (77 mg), White solid, mp 81-82 °C (lit. mp 79-80 °C), purified by column chromatography (hexane:ethyl acetate = 80:20).



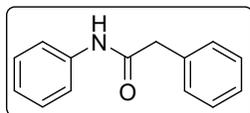
N-(4-methoxyphenyl)hexanamide (**5e**):¹¹ 65% yield (72 mg), White solid, mp 85-88 °C (lit. mp 84-86- °C), purified by column chromatography (hexane:ethyl acetate = 80:20).



N-([1,1'-biphenyl]-4-yl)hexanamide (**5f**): 59% yield (80 mg), White solid, hexane:ethyl acetate (90:10) as the eluent, mp 132-135 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.60 (d, *J* = 8.3 Hz, 2H), 7.55 (t, *J* = 6.9 Hz, 3H), 7.42 (t, *J* = 7.5 Hz, 3H), 7.33 (d, *J* = 7.5 Hz, 1H), 2.37 (t, *J* = 7.6 Hz, 2H), 1.75 (t, *J* = 7.4 Hz, 2H), 1.43 – 1.32 (m, 4H), 0.92 (d, *J* = 6.6 Hz, 3H). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 171.64, 140.52, 137.29, 137.05, 128.80, 127.62, 127.11, 126.85, 120.15, 37.84, 31.47, 25.38, 22.48, 13.99; HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₁₈H₂₁NO [M+H]⁺ 268.162, found 268.168.



N-(4-pentylphenyl)hexanamide (**5g**): 57% yield (75 mg), Creamish Solid, hexane:ethyl acetate (90:10) as the eluent, mp 55-58 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.34 (d, *J* = 8.1 Hz, 2H), 7.19 (s, 1H), 7.03 (d, *J* = 8.0 Hz, 2H), 2.48 (t, *J* = 7.7 Hz, 2H), 2.26 (t, *J* = 7.6 Hz, 2H), 1.63 (q, *J* = 7.5 Hz, 2H), 1.56 – 1.44 (m, 2H), 1.25 (m, 8H), 0.81 (t, *J* = 7.0 Hz, 6H). ¹³C{¹H} NMR (101 MHz, CDCl₃): δ 171.52, 138.96, 135.57, 128.83, 119.94, 37.73, 35.34, 31.45, 31.43, 31.22, 25.43, 22.55, 22.46, 14.05, 13.97; HRMS (ESI) *m/z*: [M+H]⁺ Calcd for C₁₇H₂₇NO [M+H]⁺ 262.209, found 262.213.



N,2-diphenylacetamide (**5h**):⁵ **2c** reagent was used instead of **2a** in general procedure **3**. White solid, 61% yield (65 mg), hexane:ethyl acetate (90:10) as the eluent, mp 105-107 °C (lit. mp 103-105 °C).

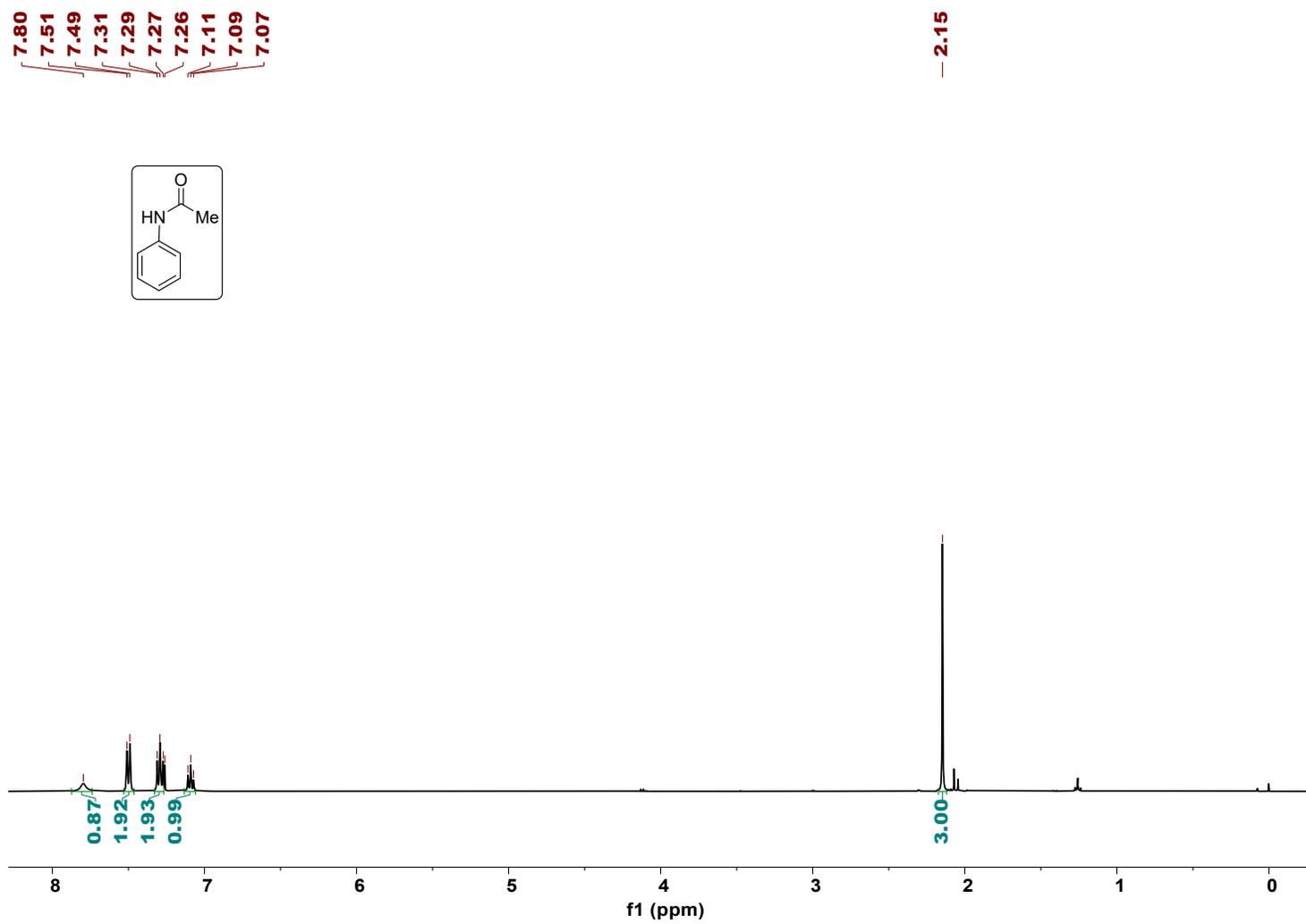
8. References:

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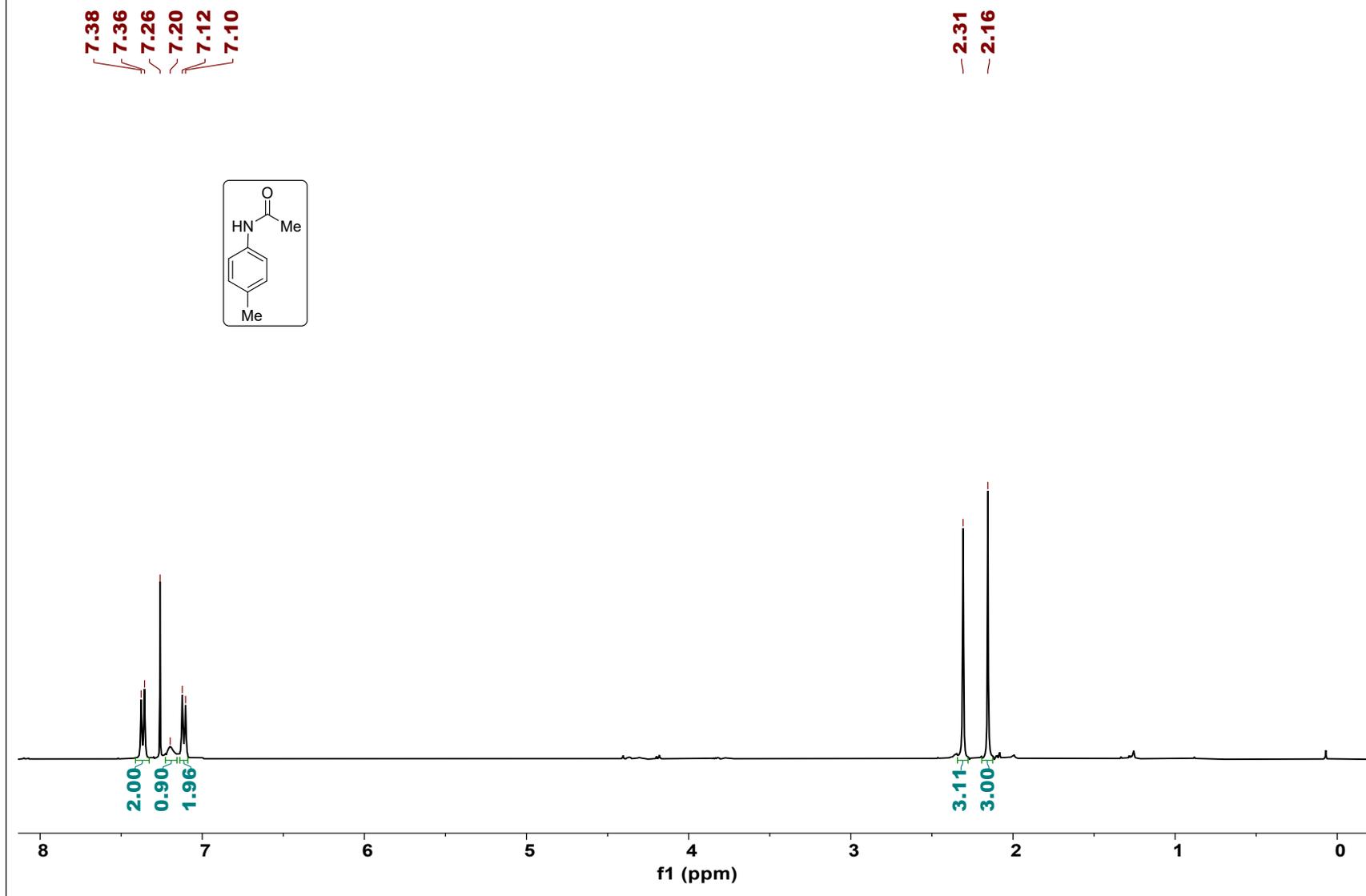
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9. Copies of ^1H and ^{13}C NMR spectra:

¹H NMR spectrum of compound **3a** (400 MHz/CDCl₃)



^1H NMR spectrum of compound **3b** (400 MHz/ CDCl_3)

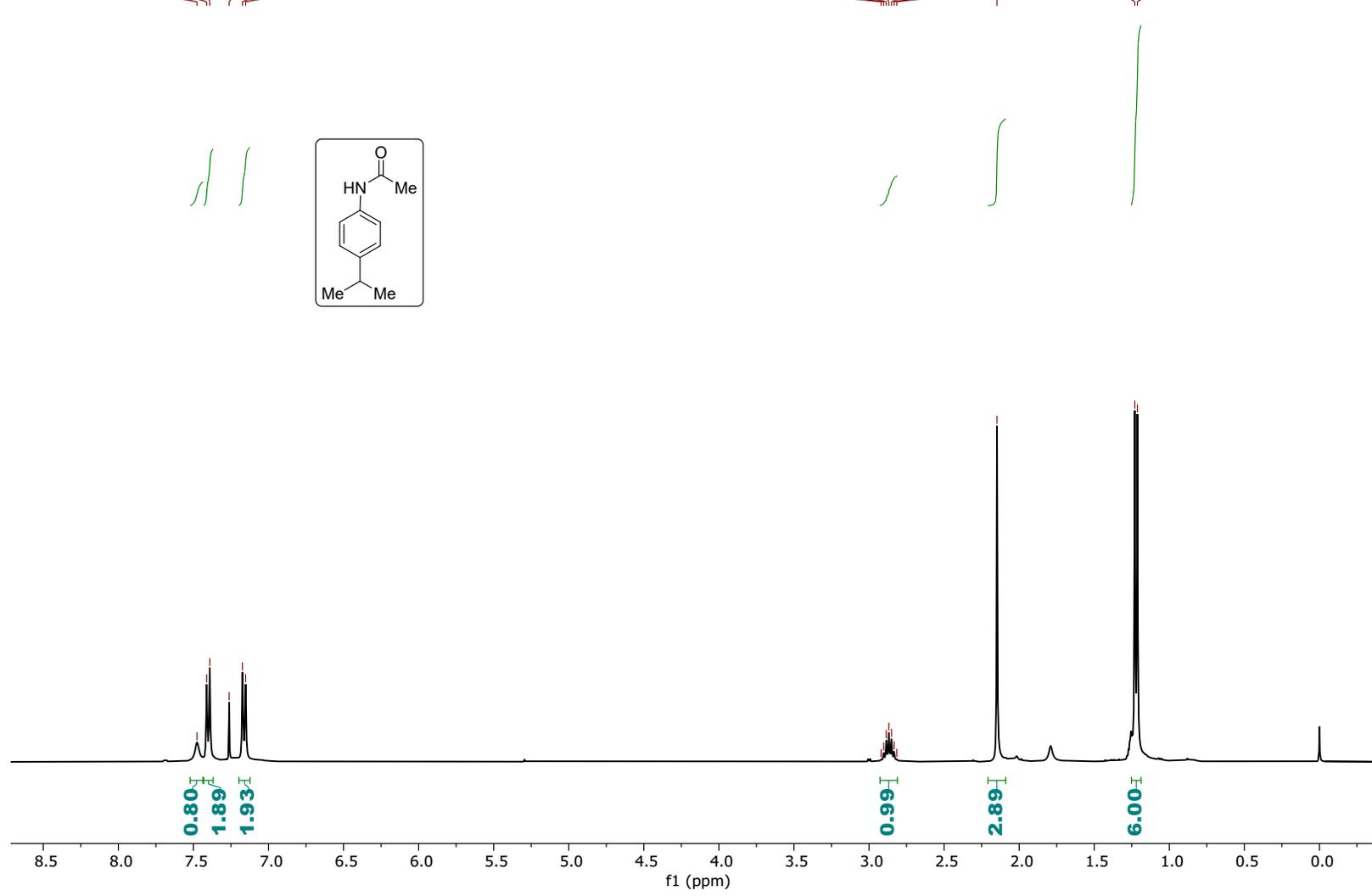
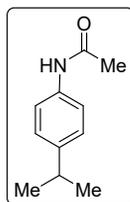


¹H NMR spectrum of compound **3c** (400 MHz/CDCl₃)

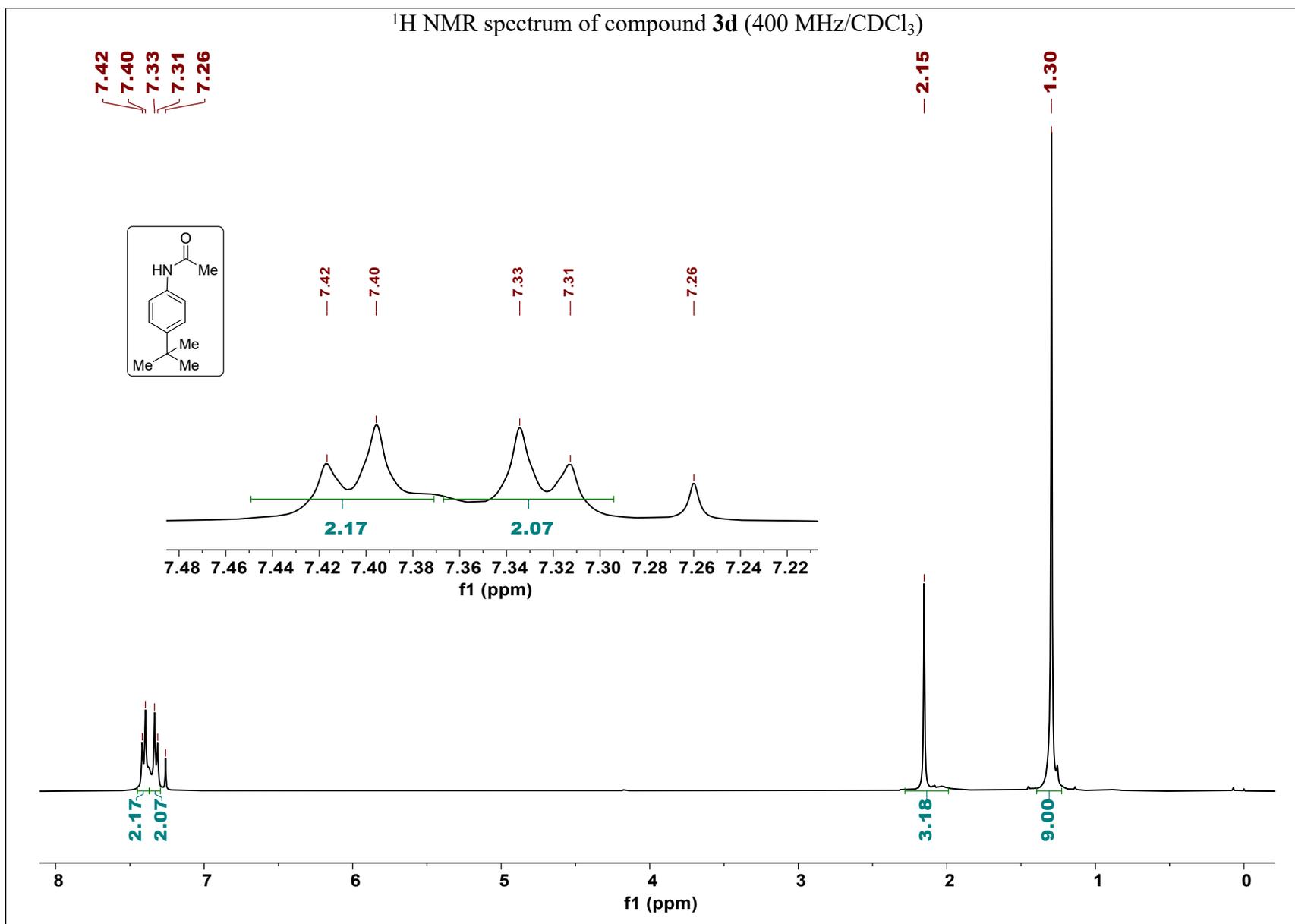
7.48
7.41
7.39
7.26
7.17
7.15

2.92
2.90
2.89
2.87
2.85
2.83
2.82
2.15

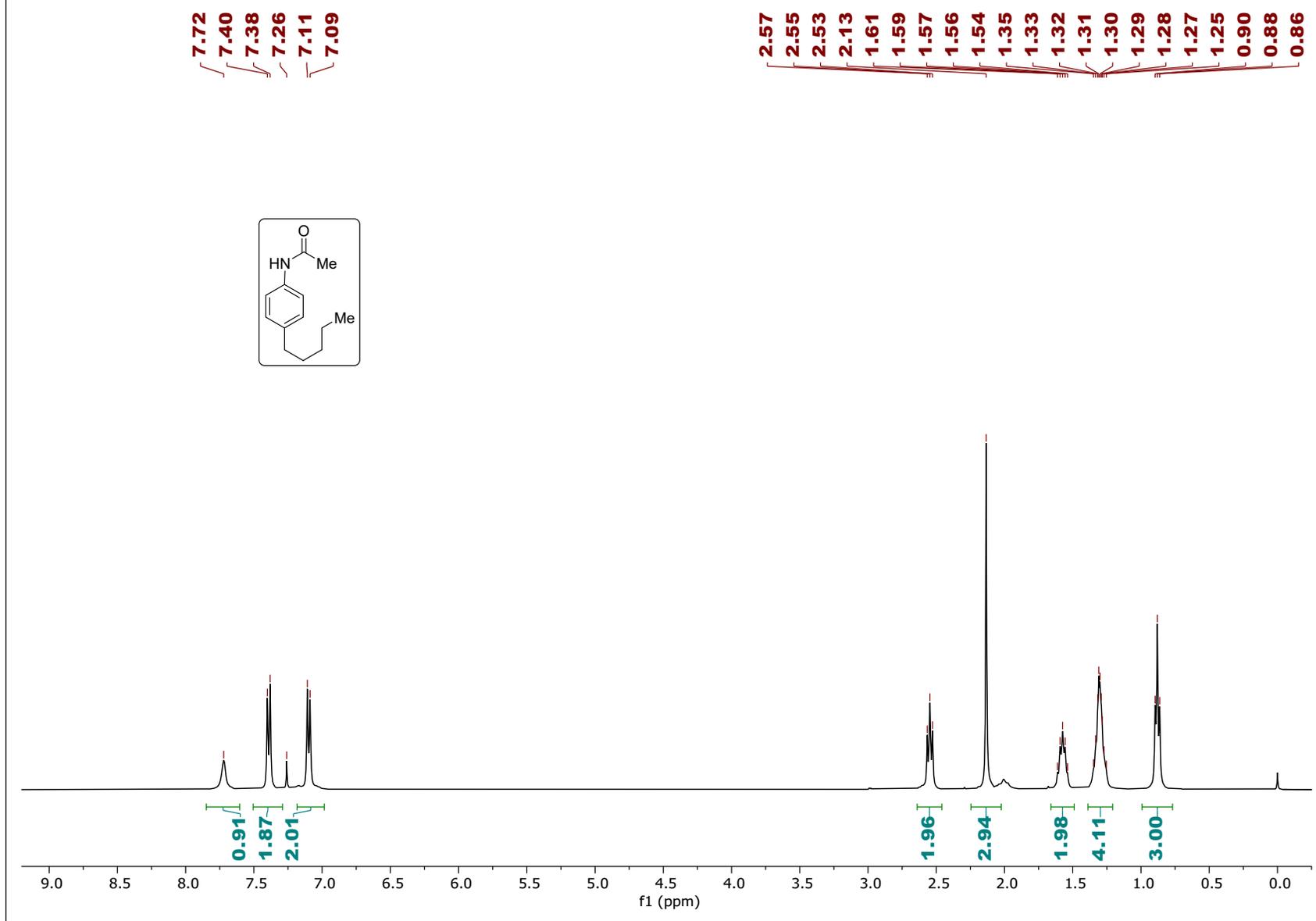
1.23
1.21



¹H NMR spectrum of compound **3d** (400 MHz/CDCl₃)



¹H NMR spectrum of compound **3e** (400 MHz/CDCl₃)



$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **3e** (101 MHz/ CDCl_3)

168.64

139.07

135.56

128.82

120.16

77.40

77.08

76.77

35.34

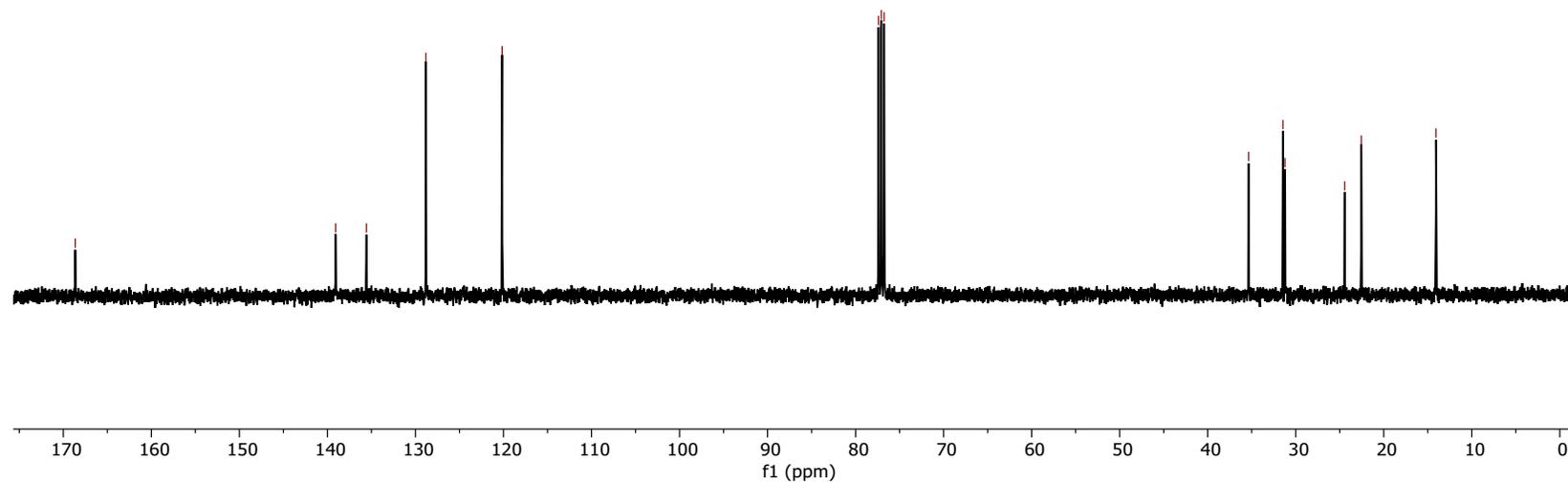
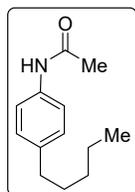
31.45

31.21

24.44

22.55

14.07



¹H NMR spectrum of compound **3f** (400 MHz/¹³CDCl₃)

7.84
7.60
7.58
7.56
7.55
7.53
7.44
7.42
7.40
7.35
7.33
7.31
7.26

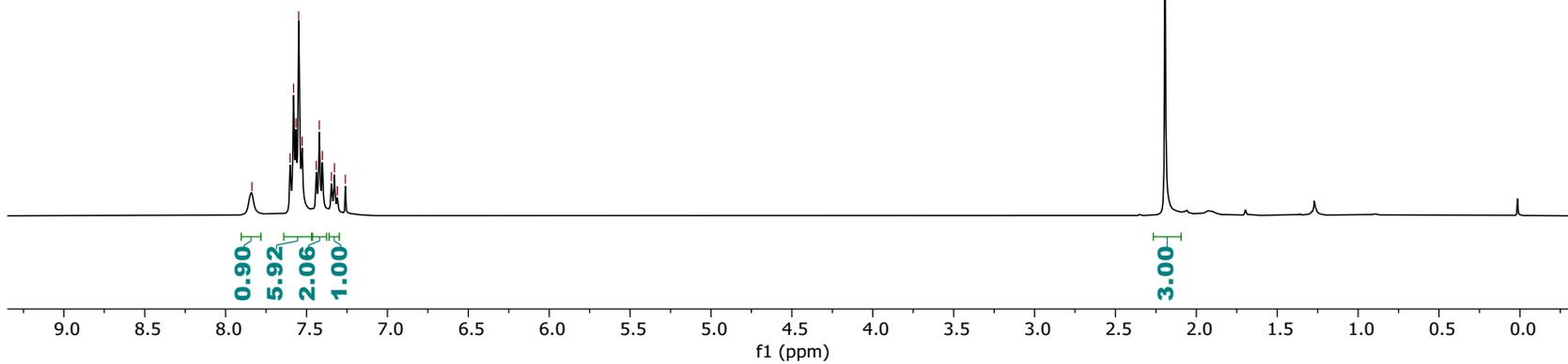
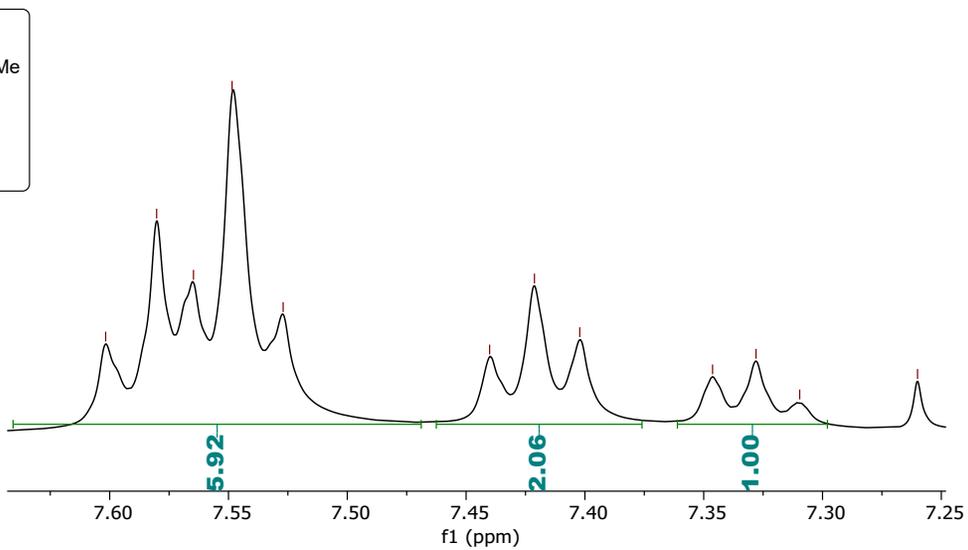
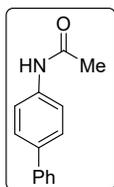
7.60
7.58
7.56
7.55
7.53

7.44
7.42
7.40

7.35
7.33
7.31

7.26

2.19

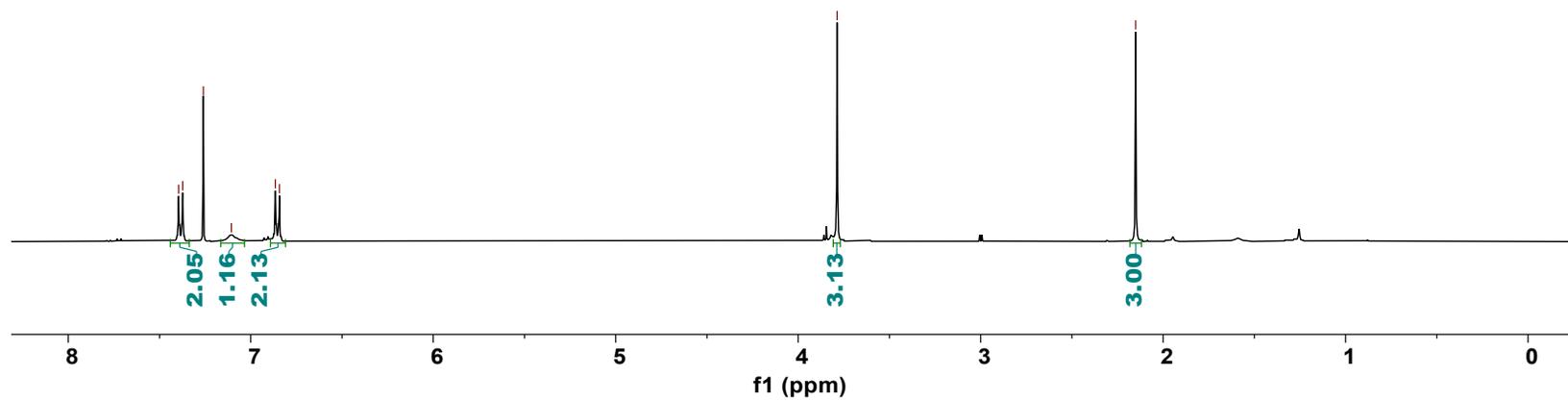
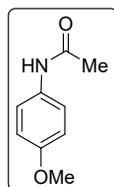


¹H NMR spectrum of compound **3g** (400 MHz/CDCl₃)

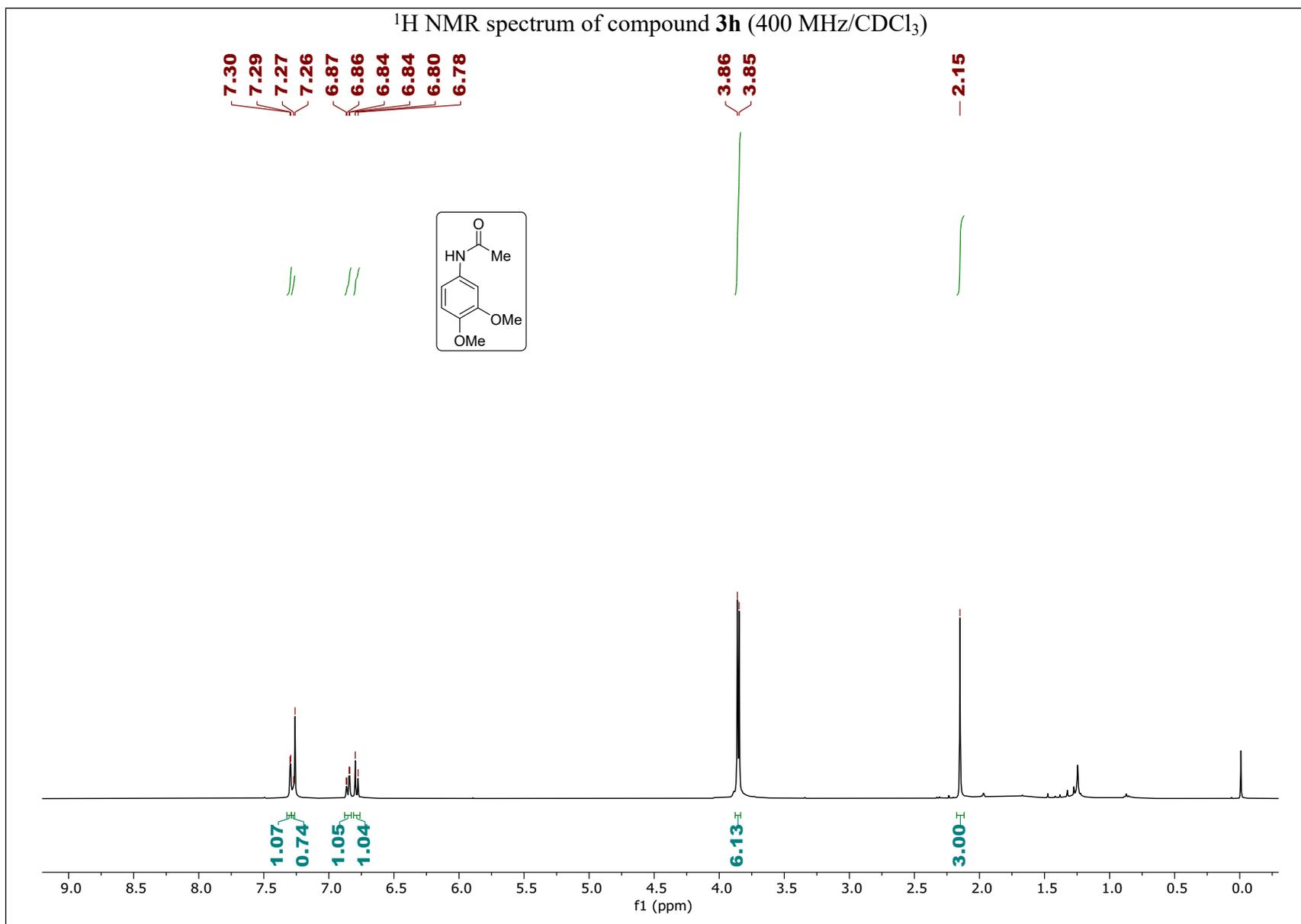
7.40
7.37
7.26
7.11
6.87
6.84

3.79

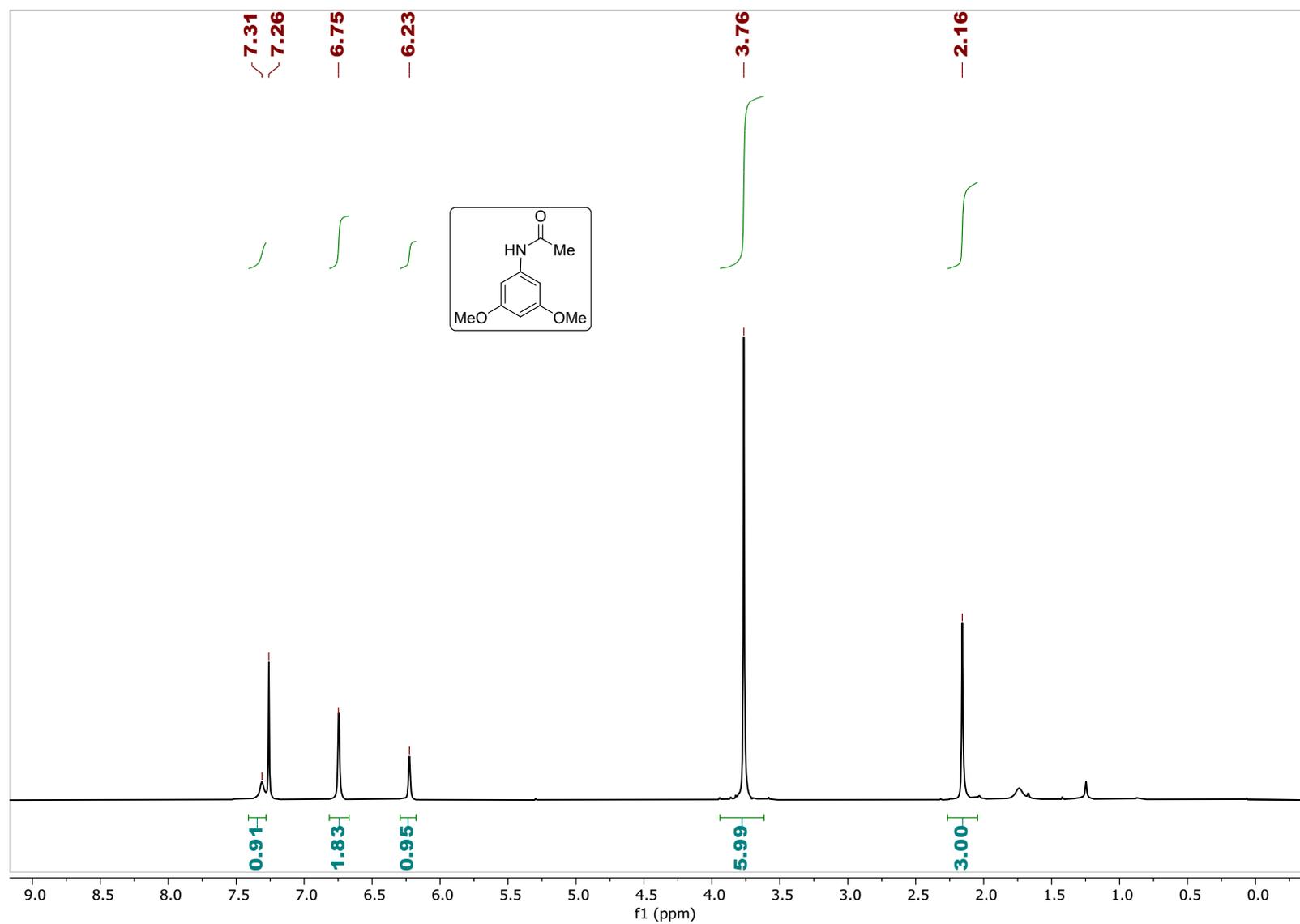
2.15



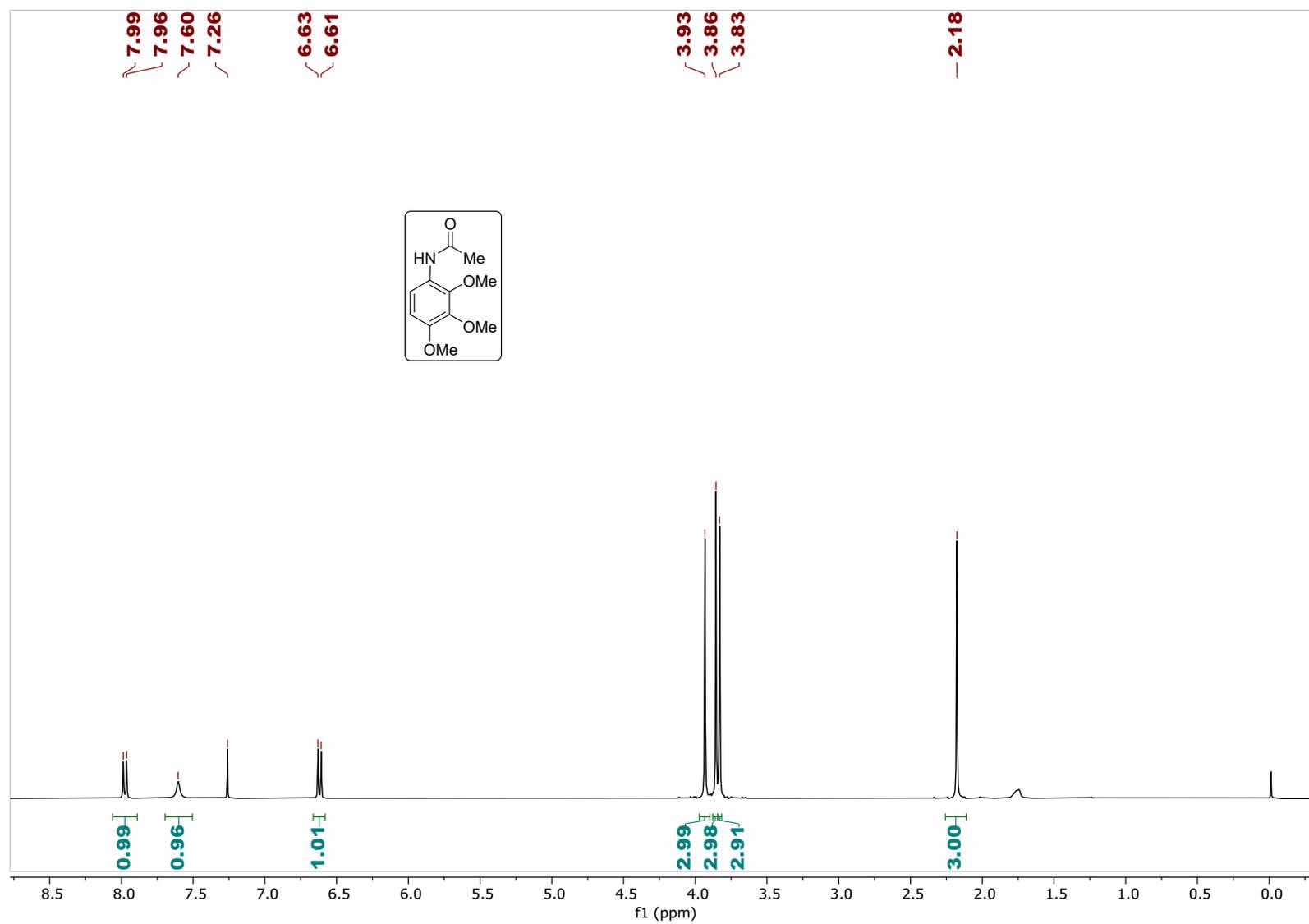
¹H NMR spectrum of compound **3h** (400 MHz/CDCl₃)



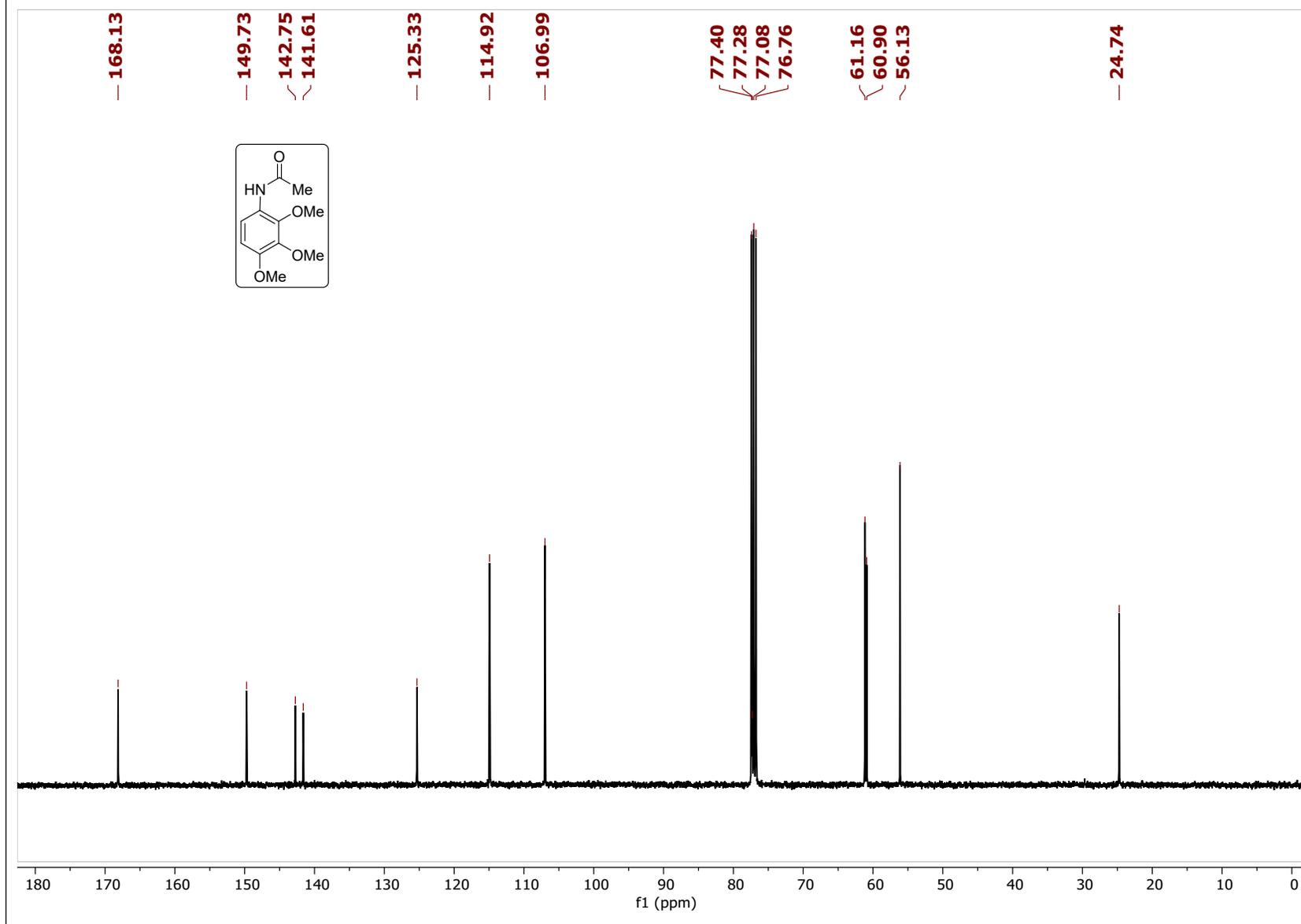
¹H NMR spectrum of compound **3i** (400 MHz/CDCl₃)



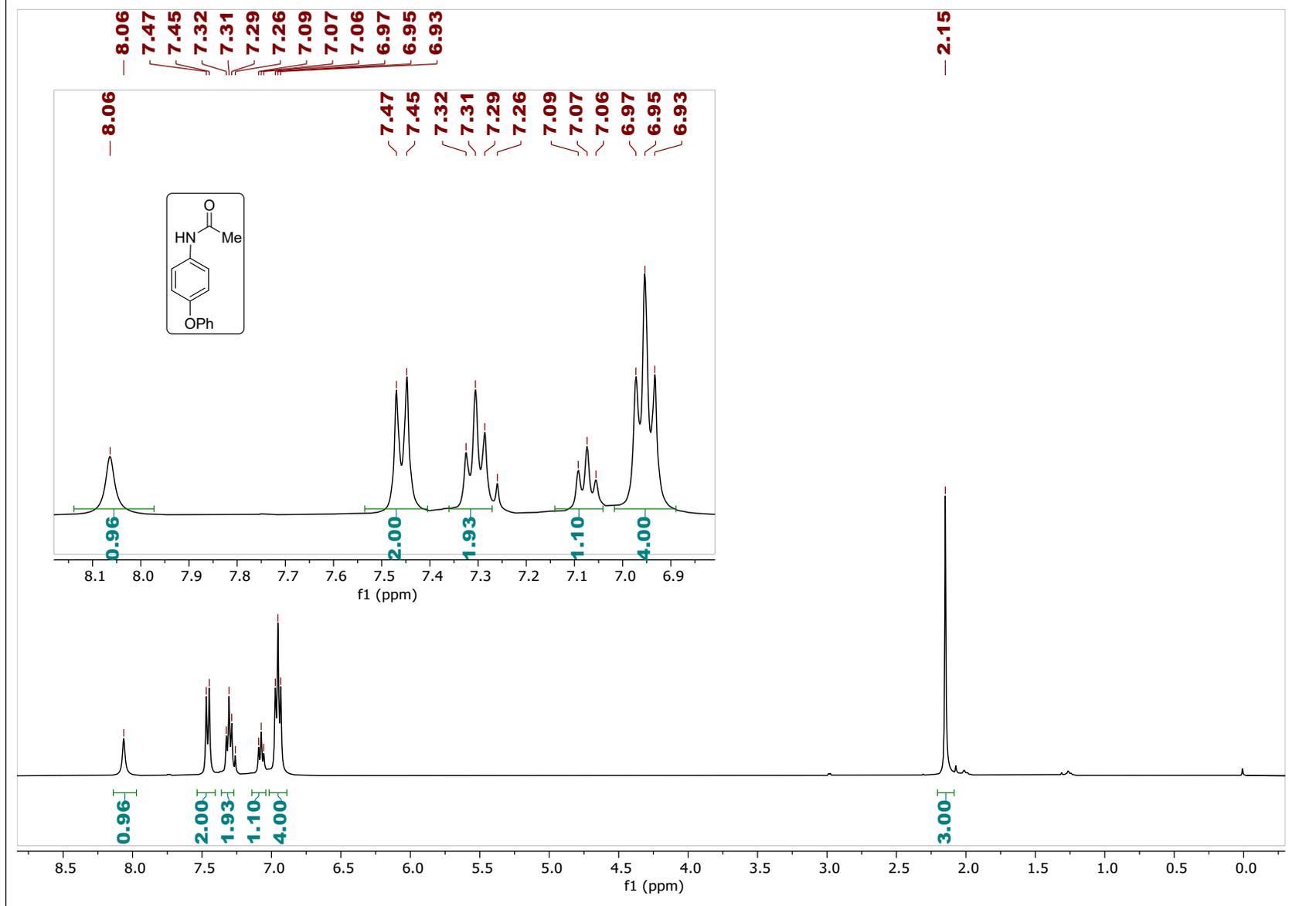
¹H NMR spectrum of compound **3j** (400 MHz/CDCl₃)



$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **3j** (101 MHz/ CDCl_3)



¹H NMR spectrum of compound **3k** (400 MHz/CDCl₃)



¹H NMR spectrum of compound **31** (400 MHz/CDCl₃)

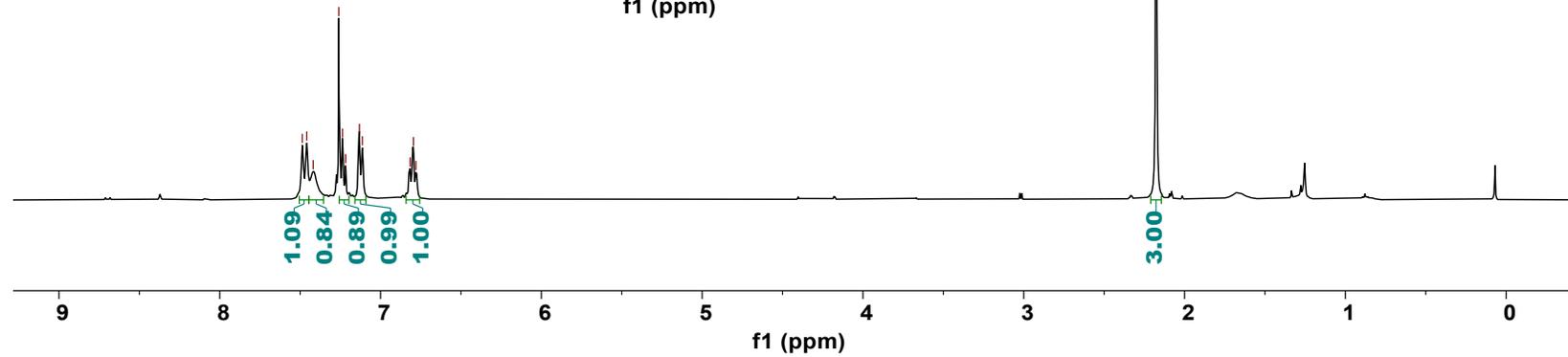
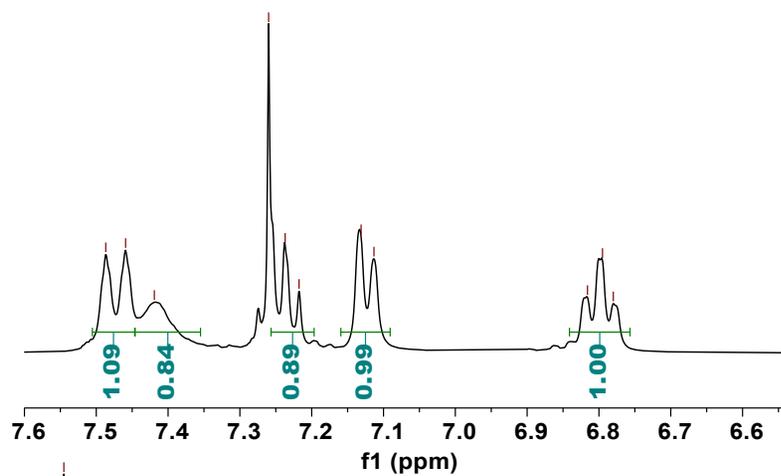
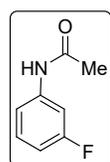
7.49
7.46
7.42
7.26
7.24
7.22
7.13
7.11
6.82
6.80
6.78

7.49
7.46
7.42

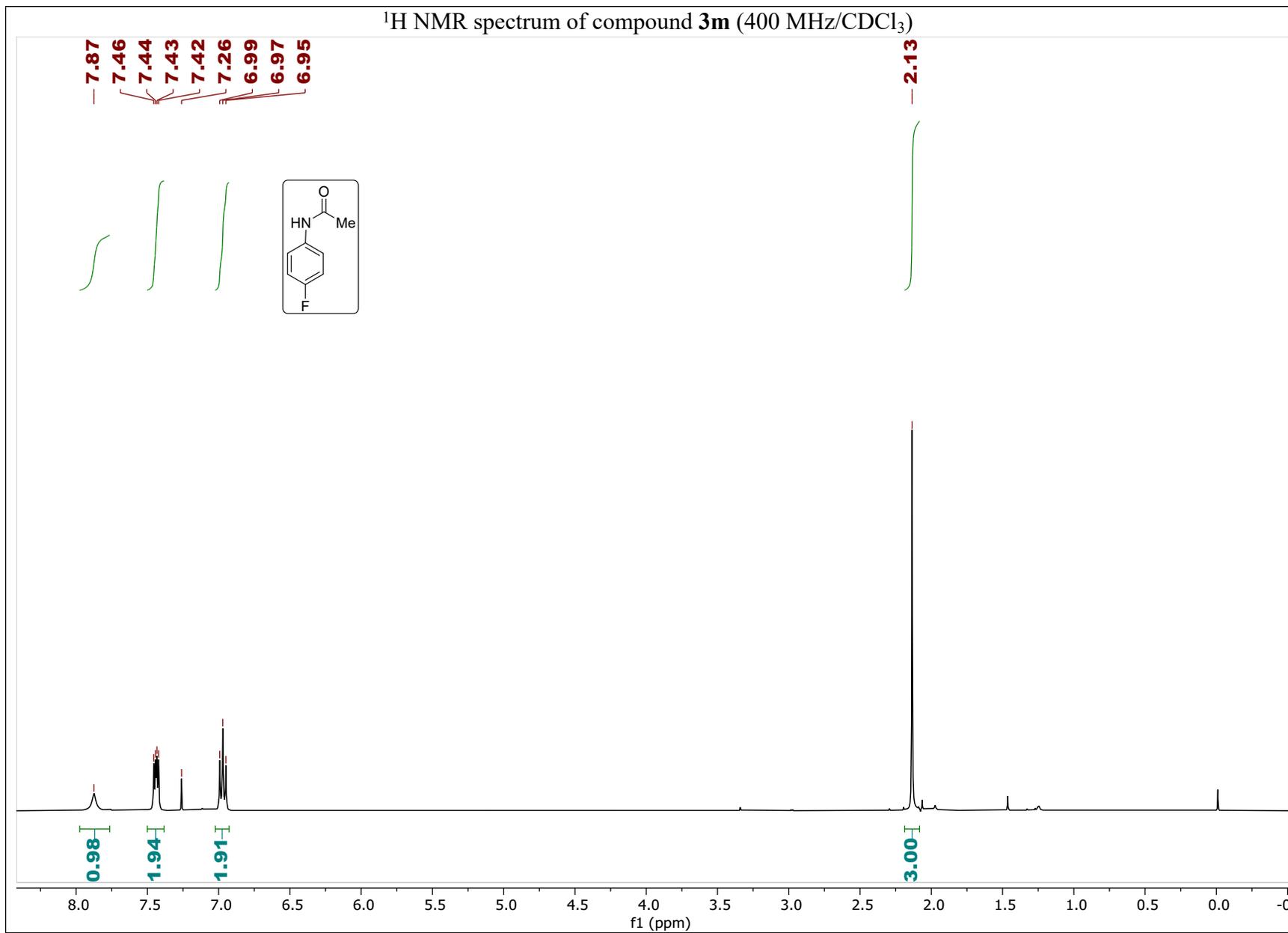
7.26
7.24
7.22
7.13
7.11

6.82
6.80
6.78

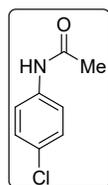
2.18

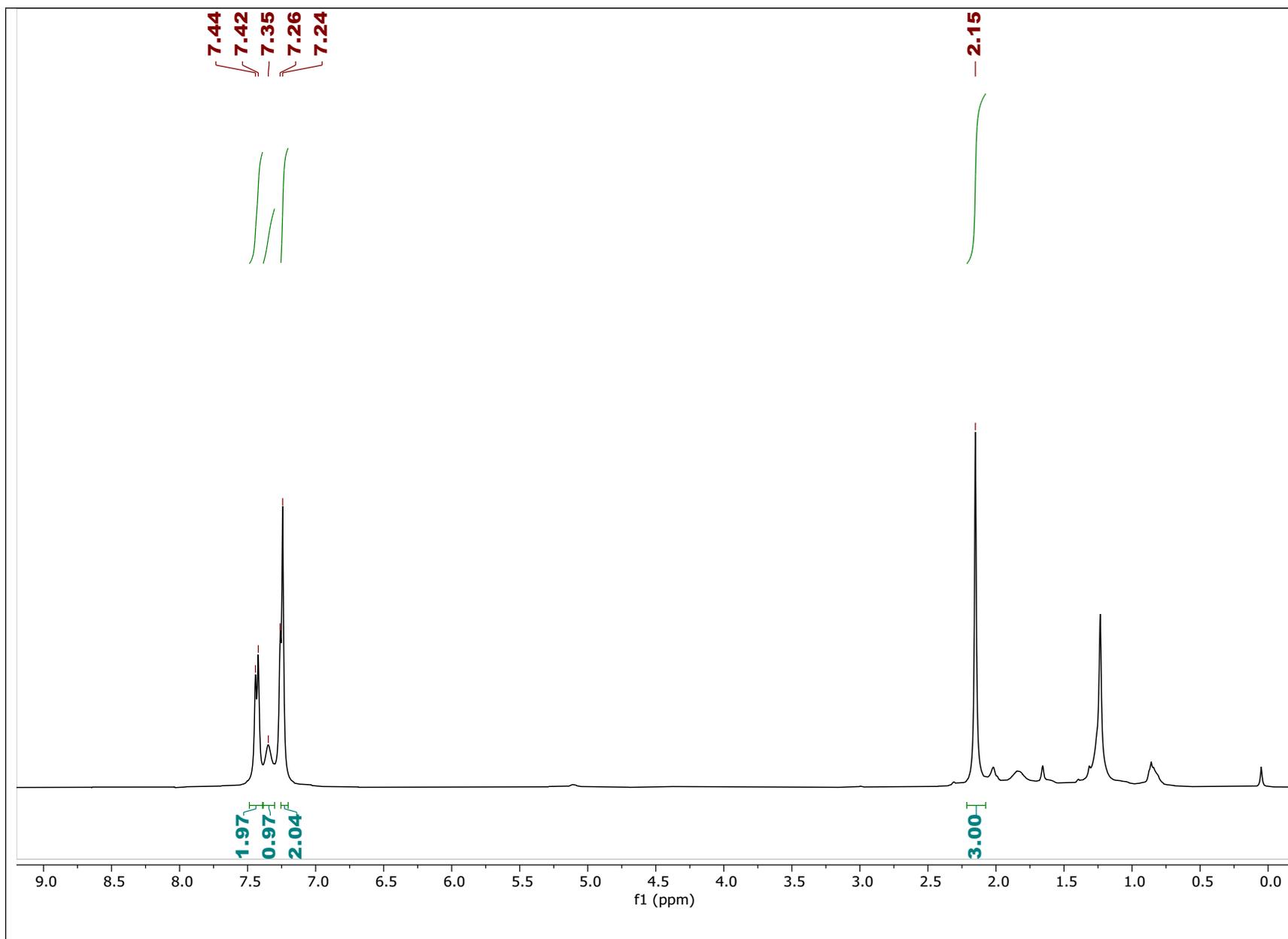


¹H NMR spectrum of compound **3m** (400 MHz/CDCl₃)

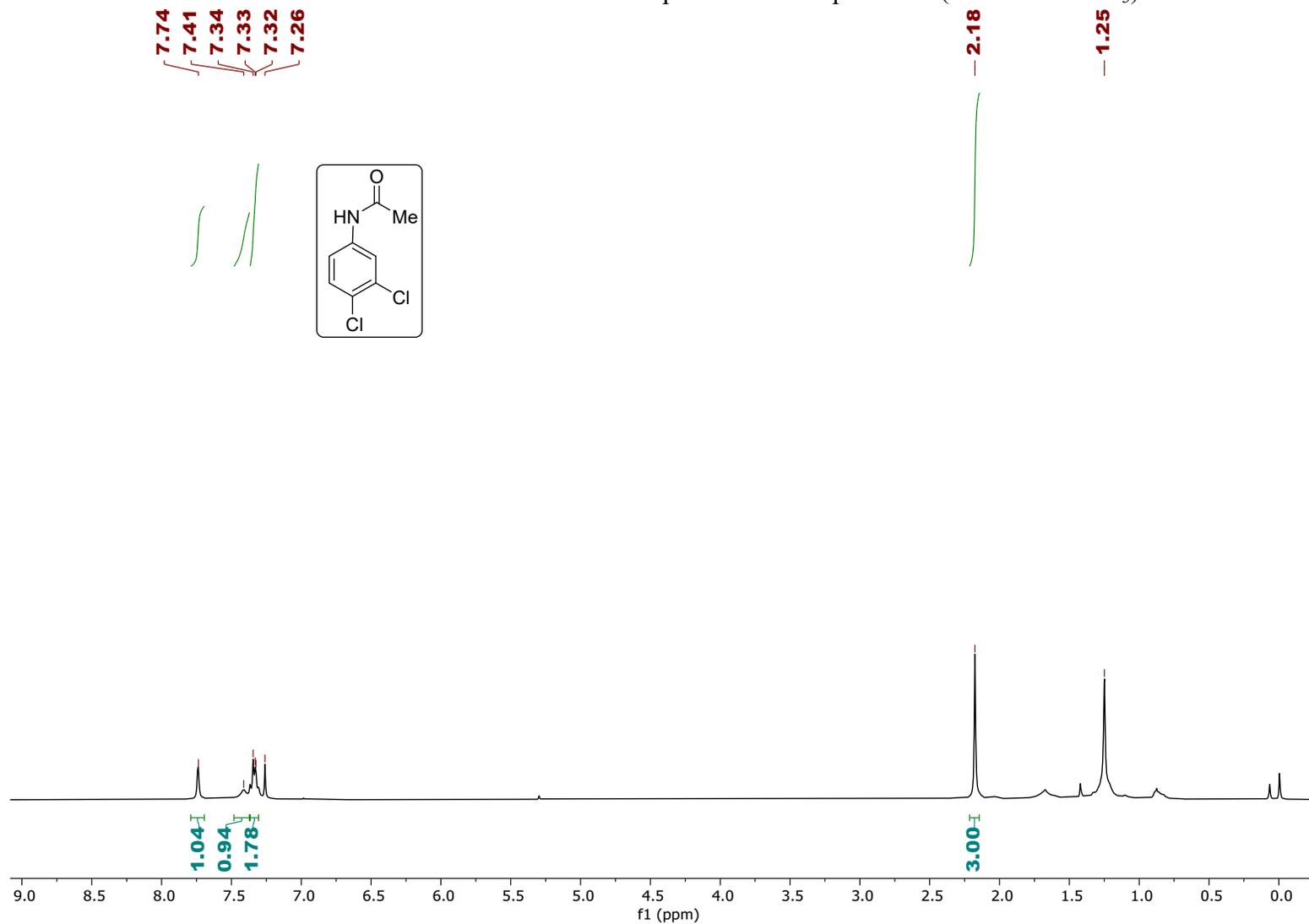


^1H NMR spectrum of compound **3n** (400 MHz/ CDCl_3)

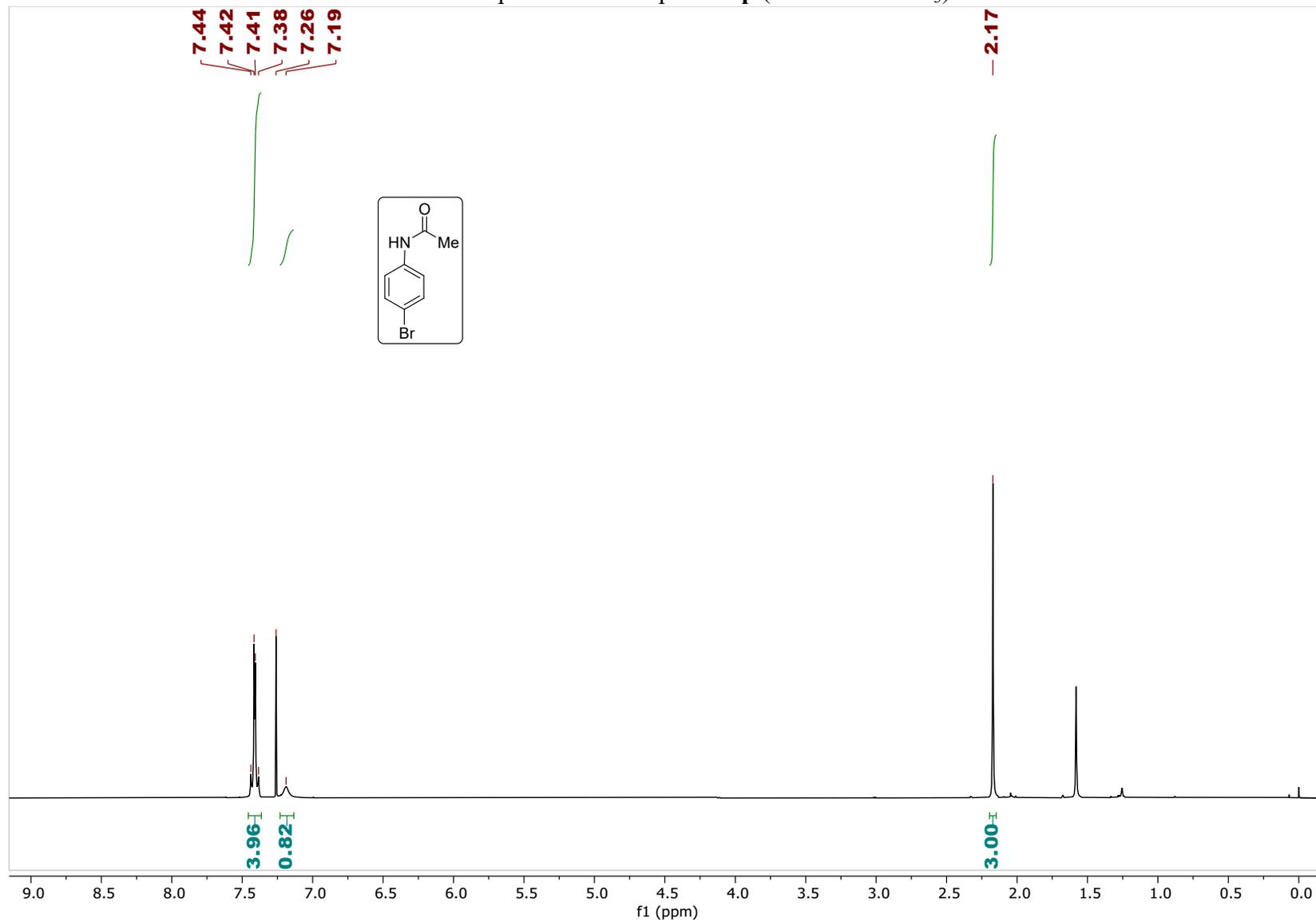




¹H NMR spectrum of compound **3o** (400 MHz/CDCl₃)



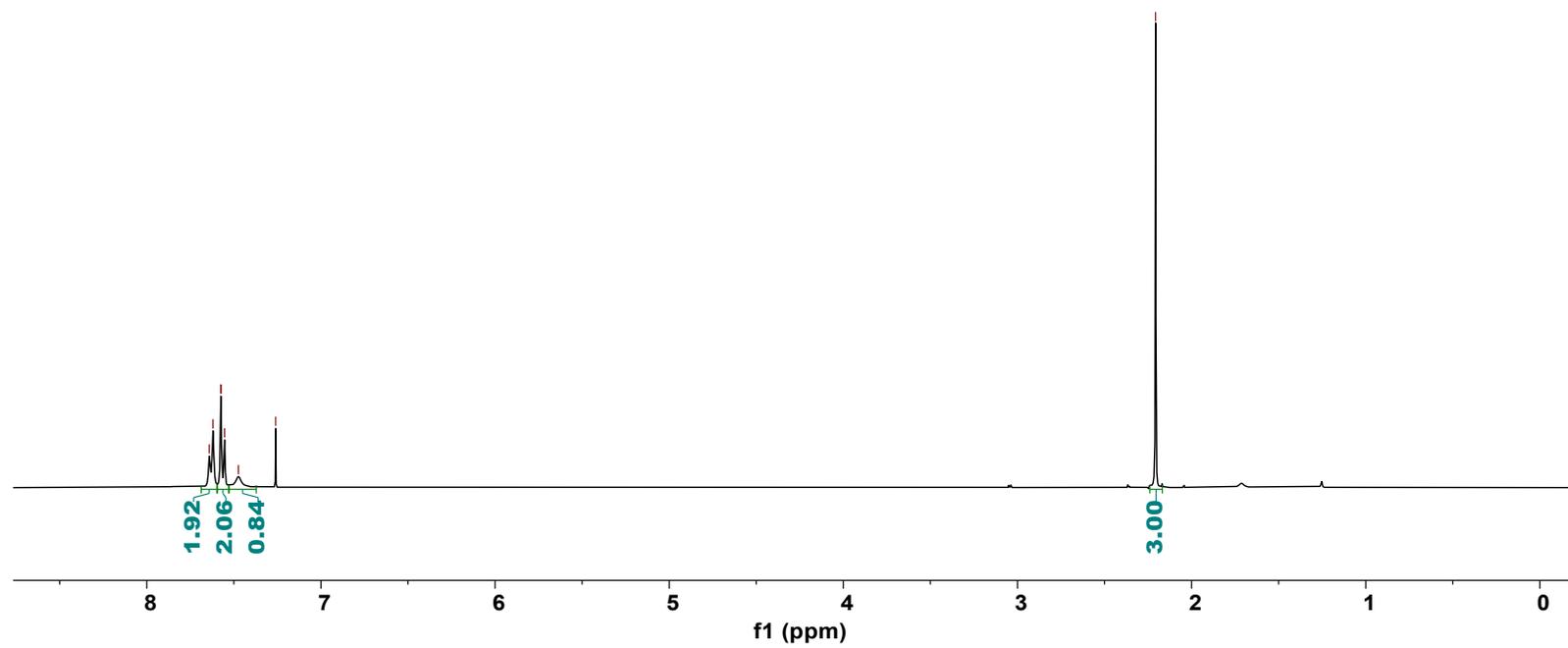
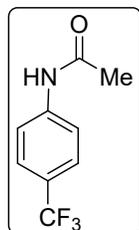
¹H NMR spectrum of compound **3p** (400 MHz/CDCl₃)



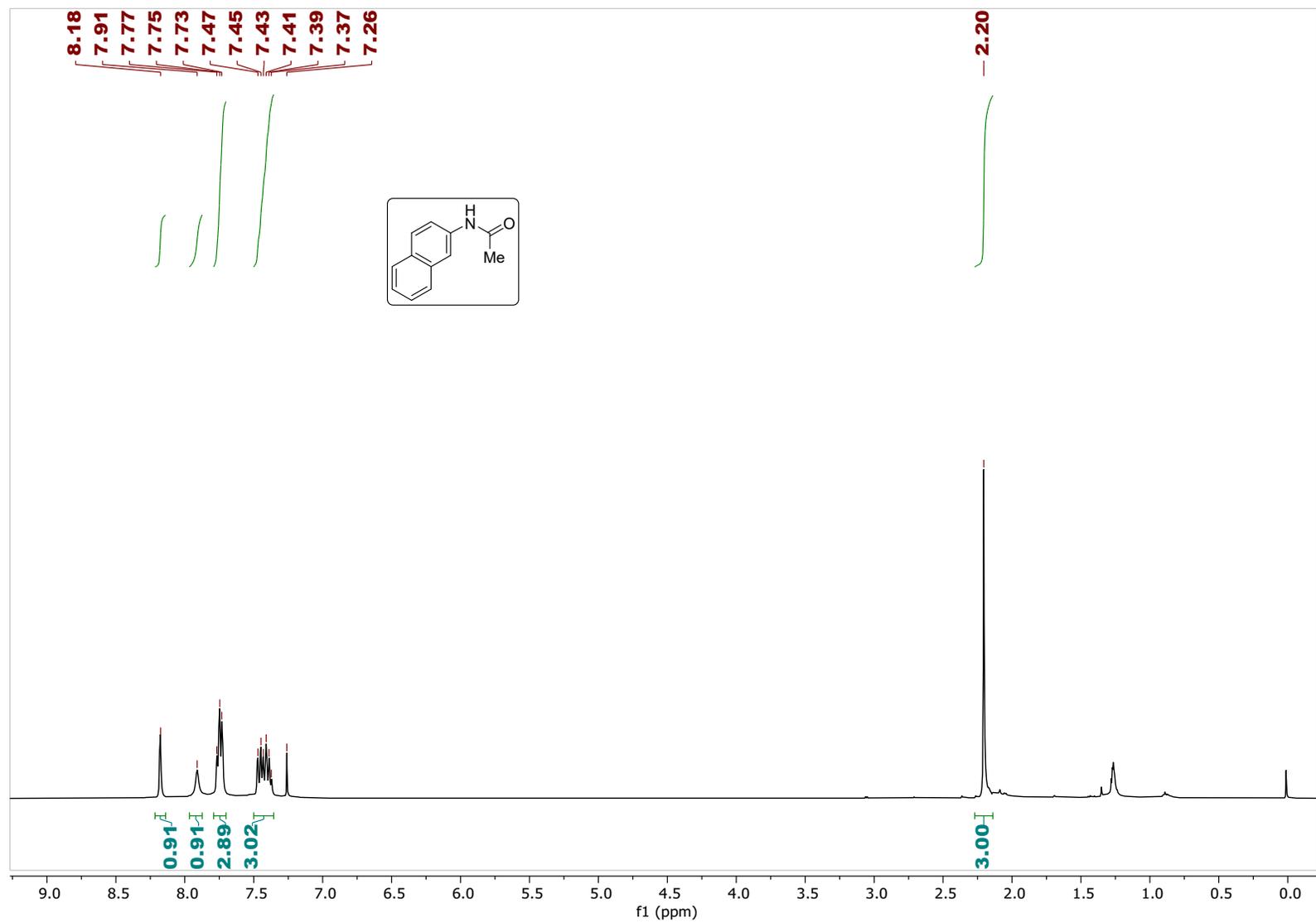
¹H NMR spectrum of compound **3q** (400 MHz/CDCl₃)

7.64
7.62
7.57
7.57
7.55
7.47
7.26

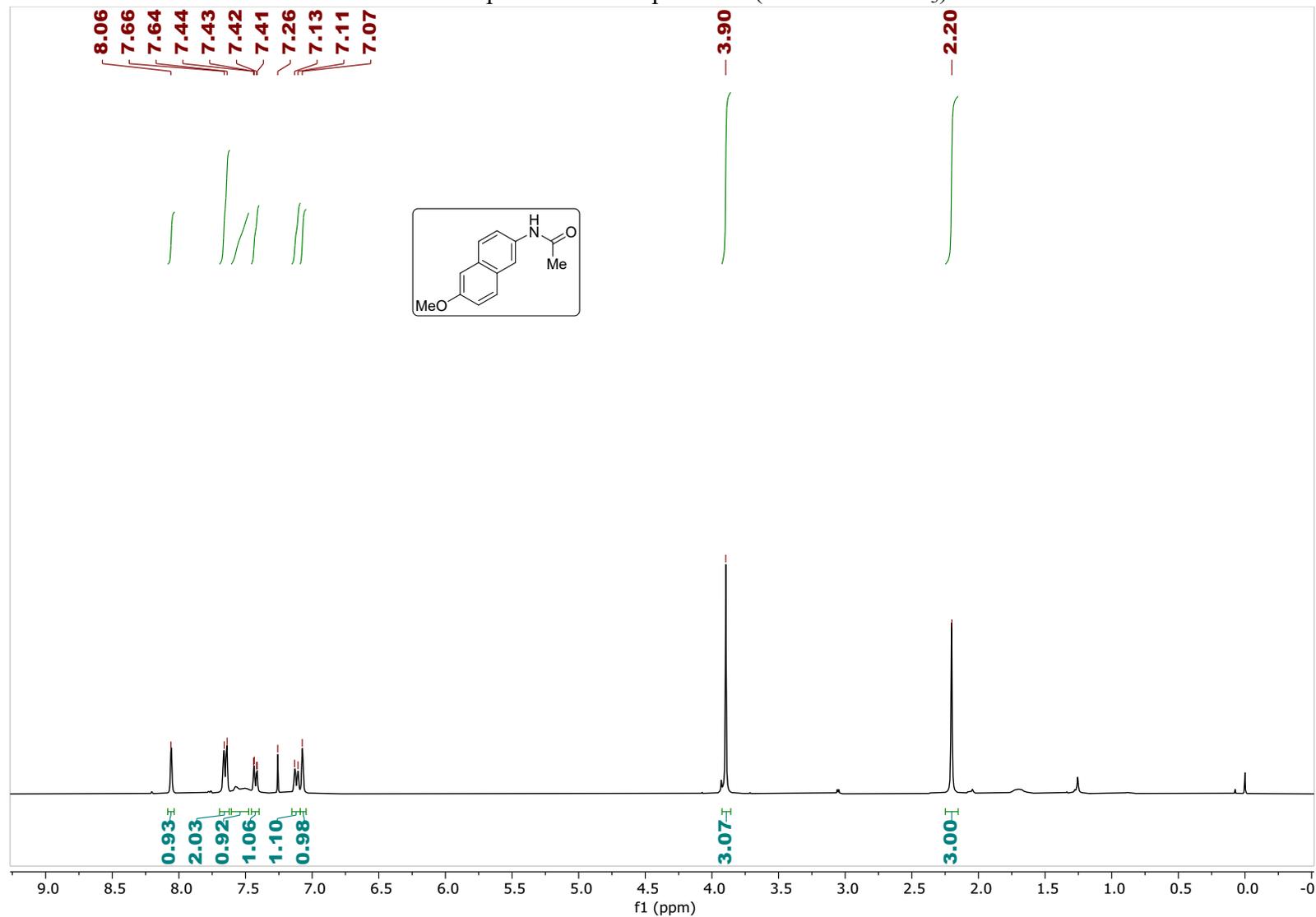
2.21



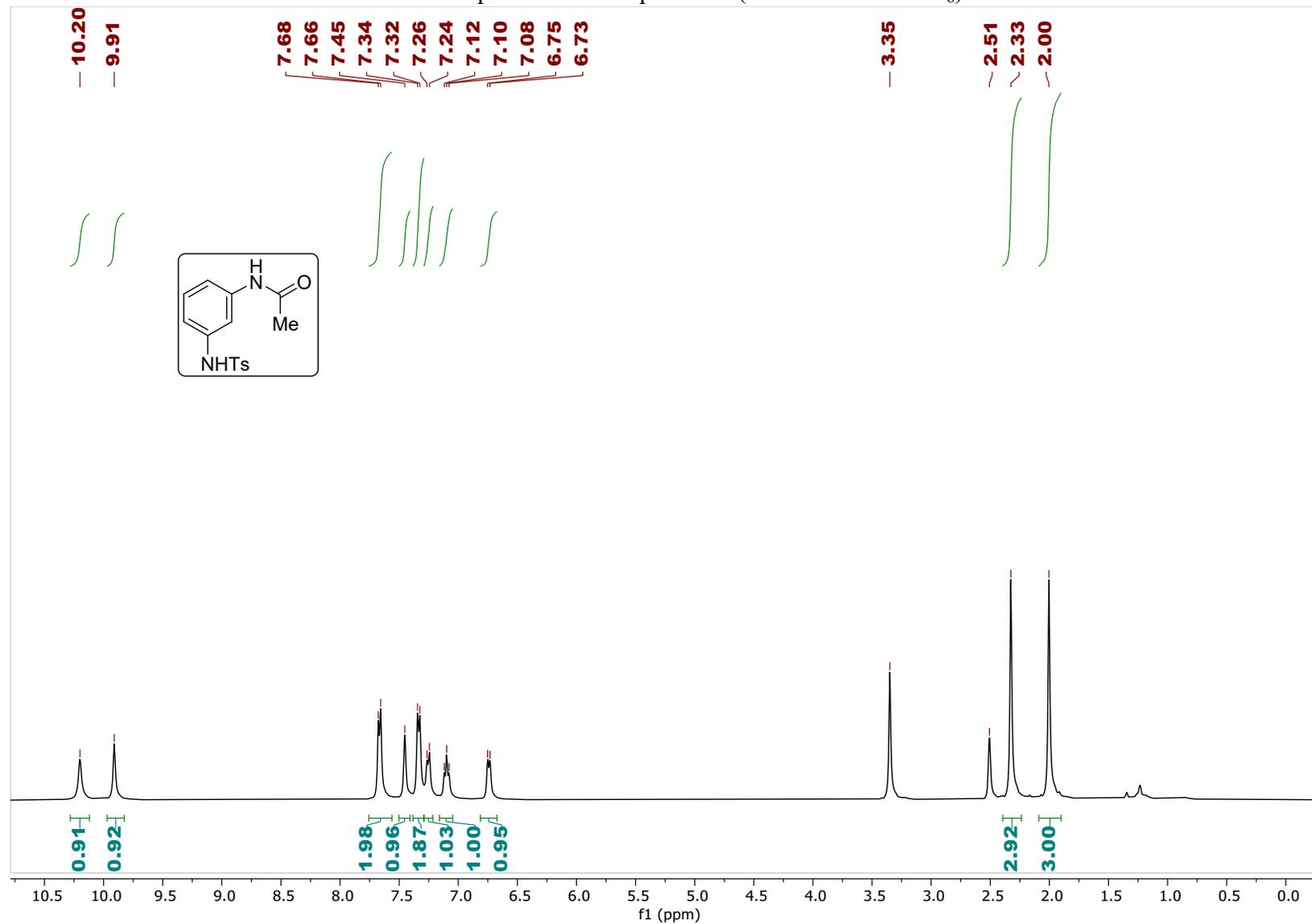
¹H NMR spectrum of compound **3r** (400 MHz/CDCl₃)



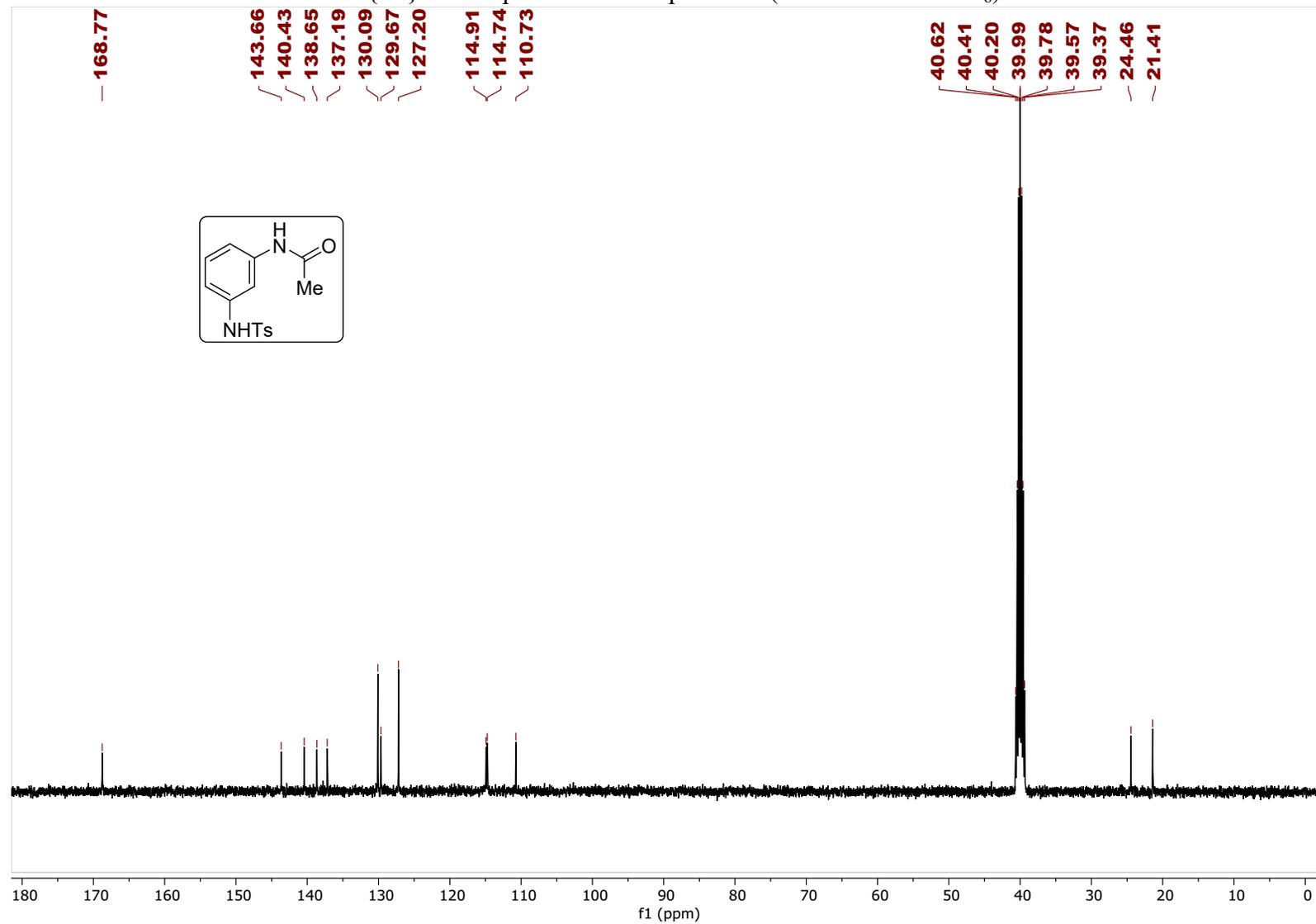
¹H NMR spectrum of compound **3s** (400 MHz/CDCl₃)



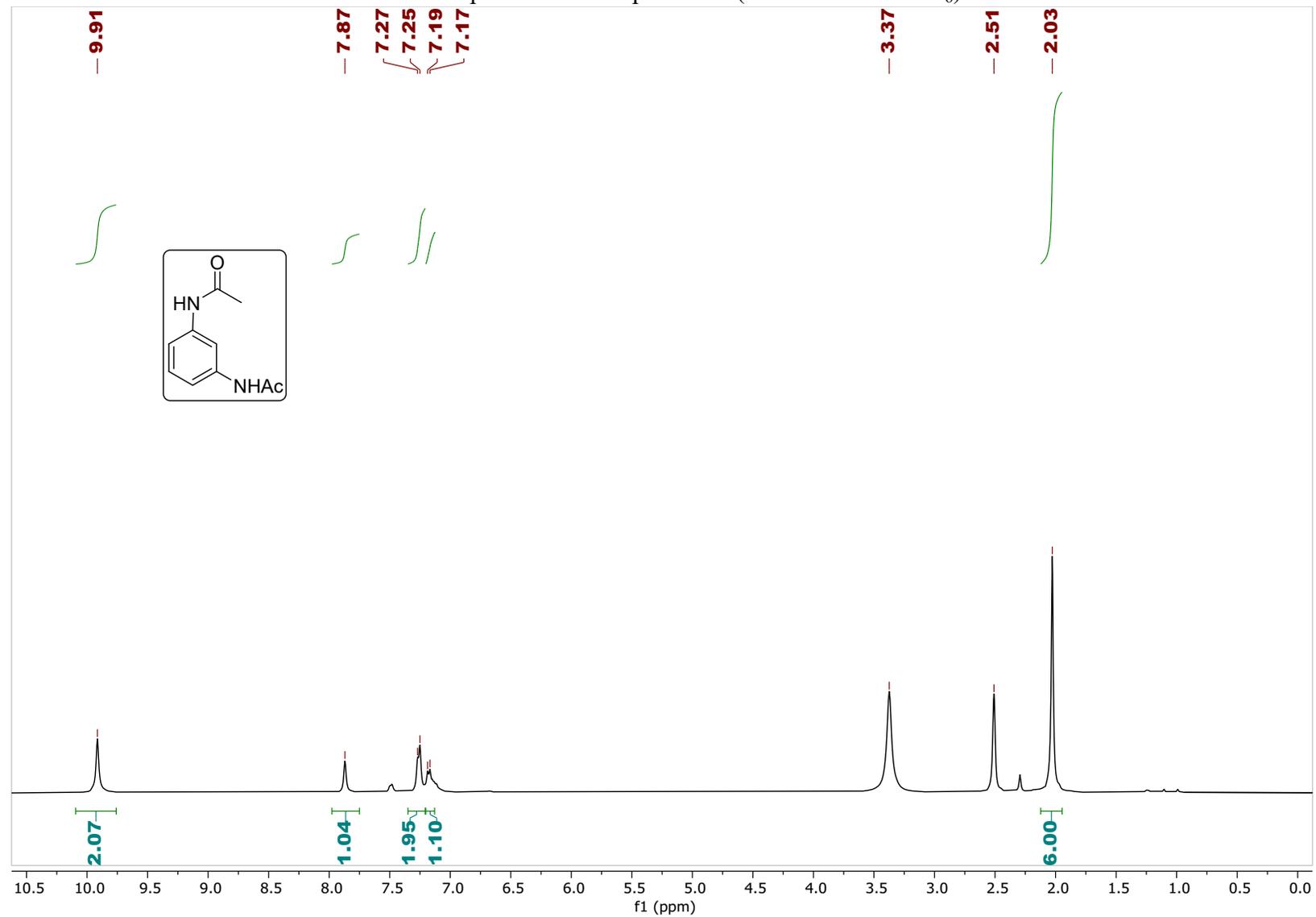
¹H NMR spectrum of compound **3t** (400 MHz/DMSO-*d*₆)



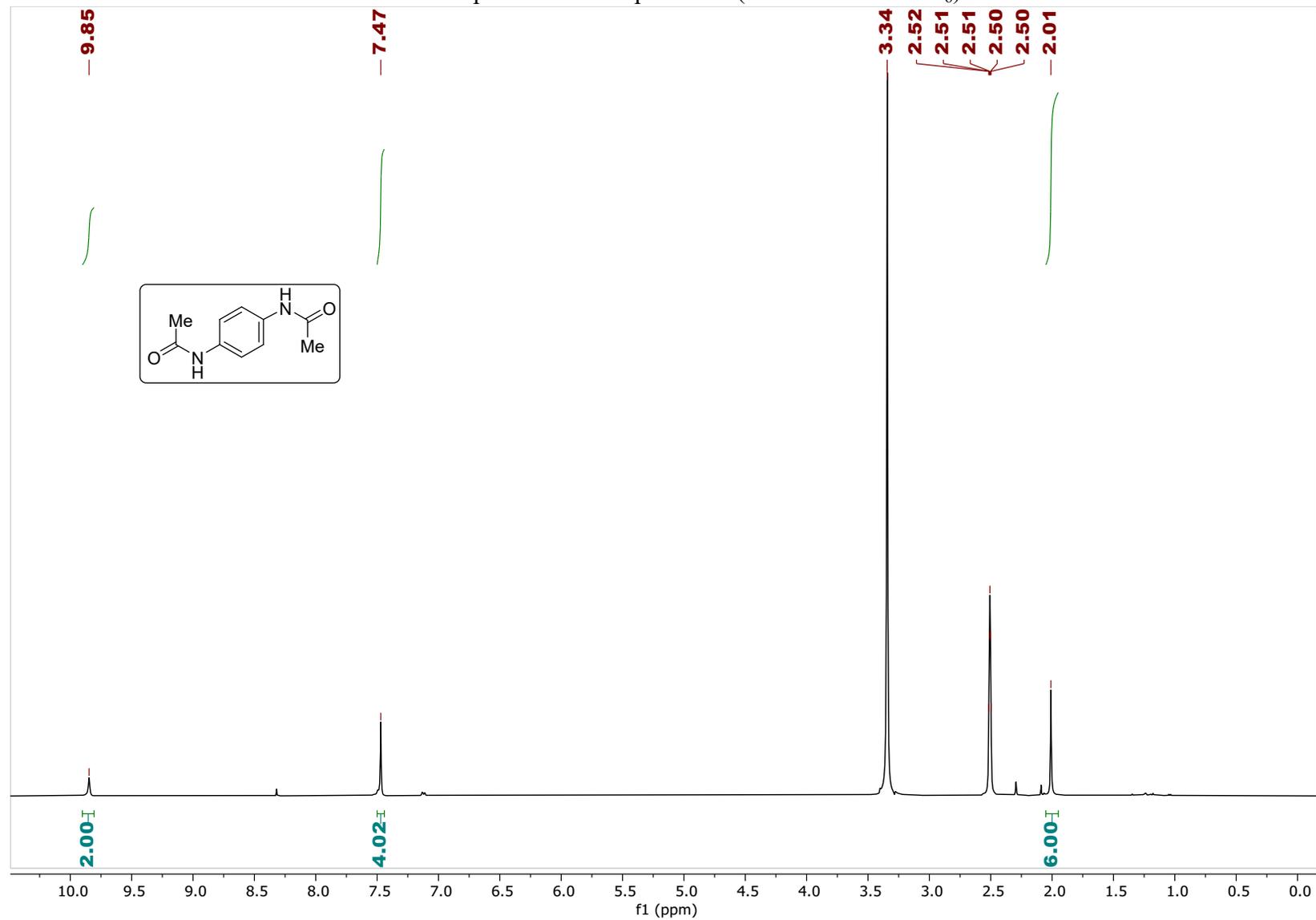
$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **3t** (101 MHz/DMSO- d_6)



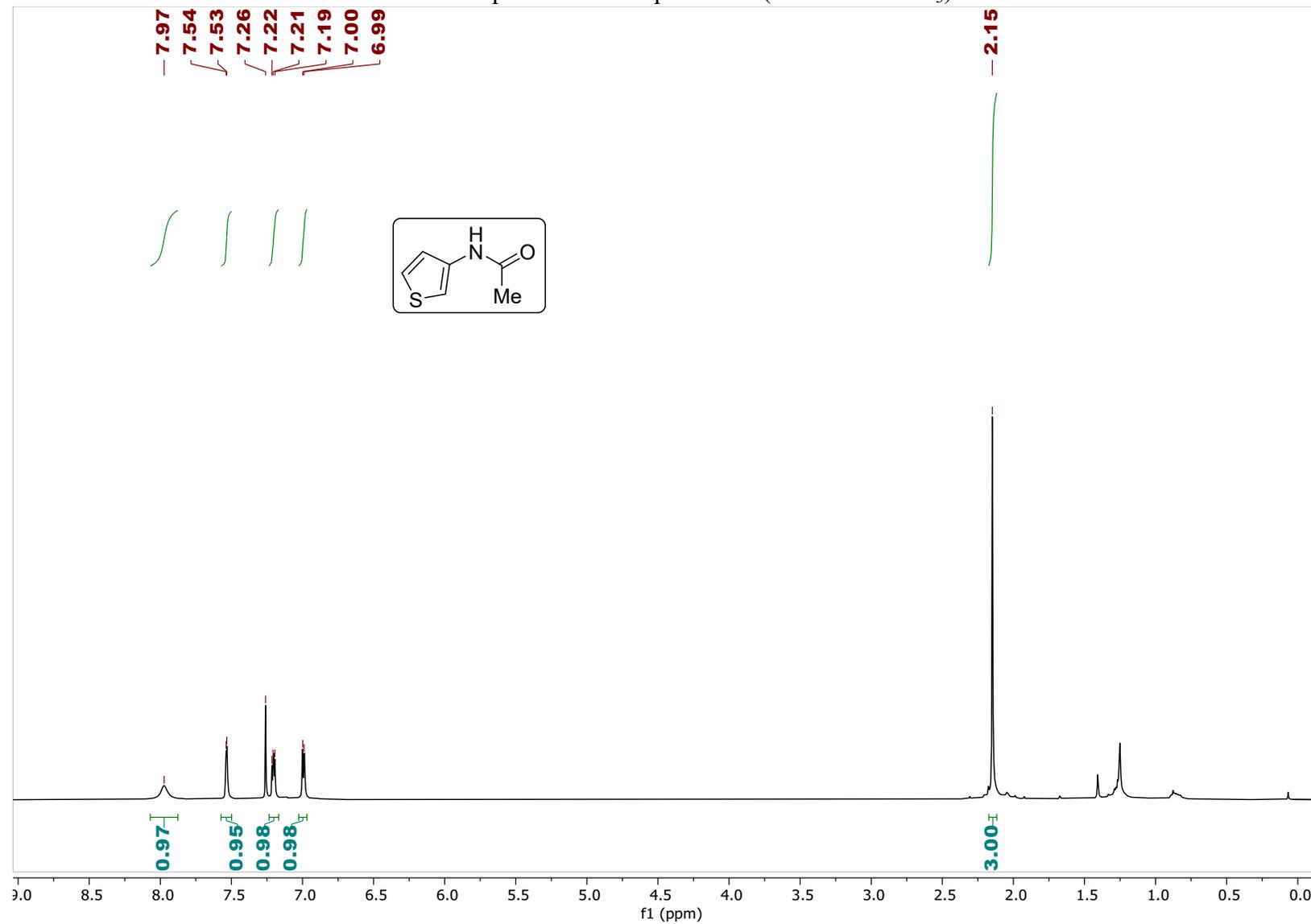
¹H NMR spectrum of compound **3u** (400 MHz/DMSO-*d*₆)



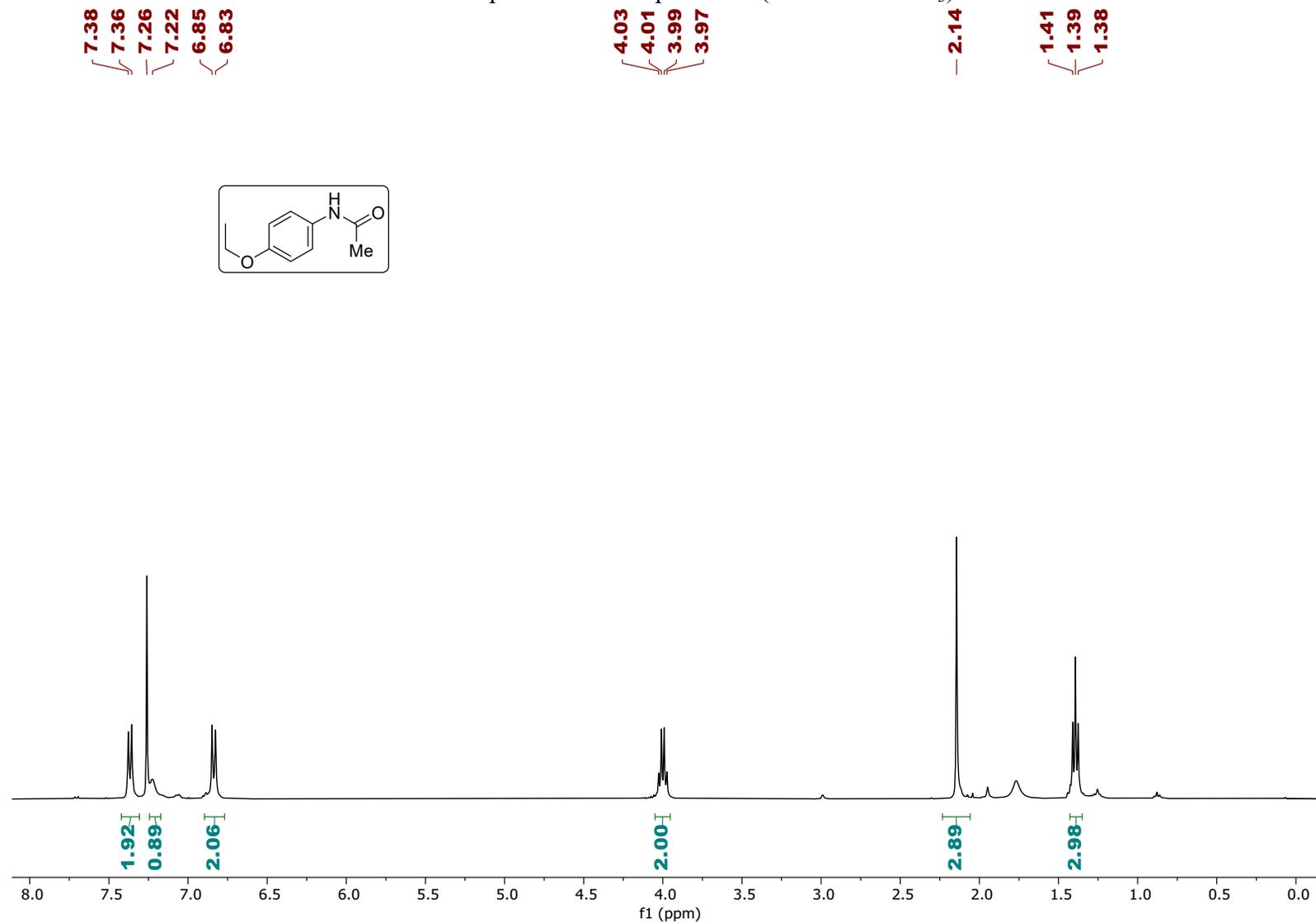
¹H NMR spectrum of compound **3v** (400 MHz/DMSO-*d*₆)



¹H NMR spectrum of compound **3w** (400 MHz/CDCl₃)



¹H NMR spectrum of compound **3x** (400 MHz/CDCl₃)

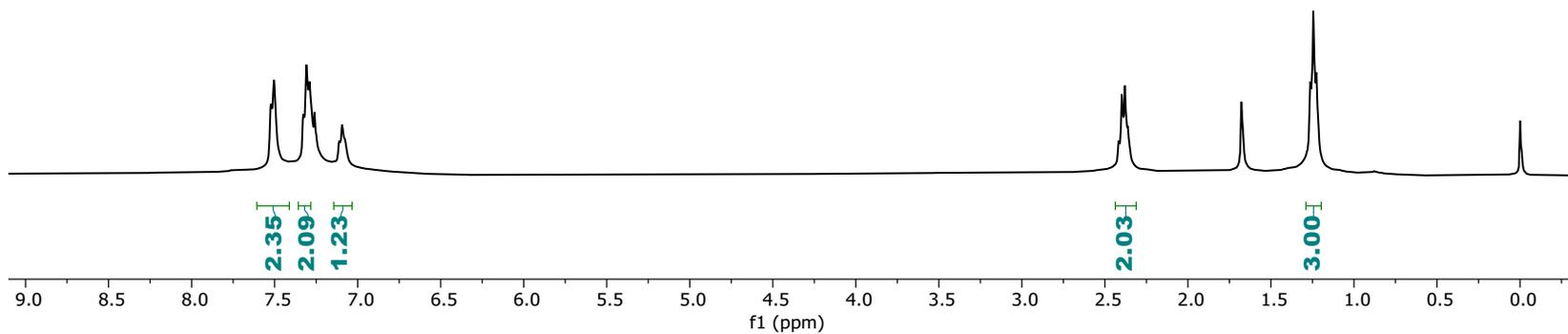
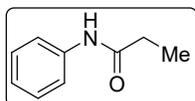


¹H NMR spectrum of compound **5a** (400 MHz/CDCl₃)

7.521
7.501
7.321
7.301
7.291
7.251
7.111
7.091
7.071

2.411
2.391
2.381
2.361

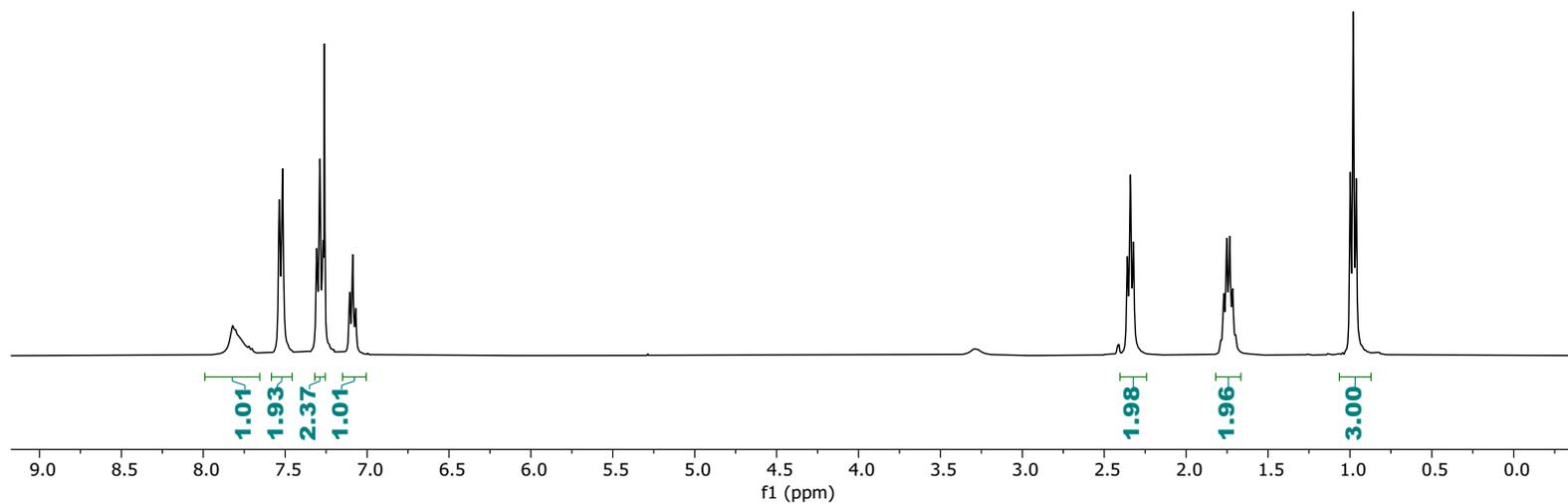
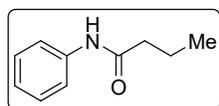
1.261
1.251
1.241



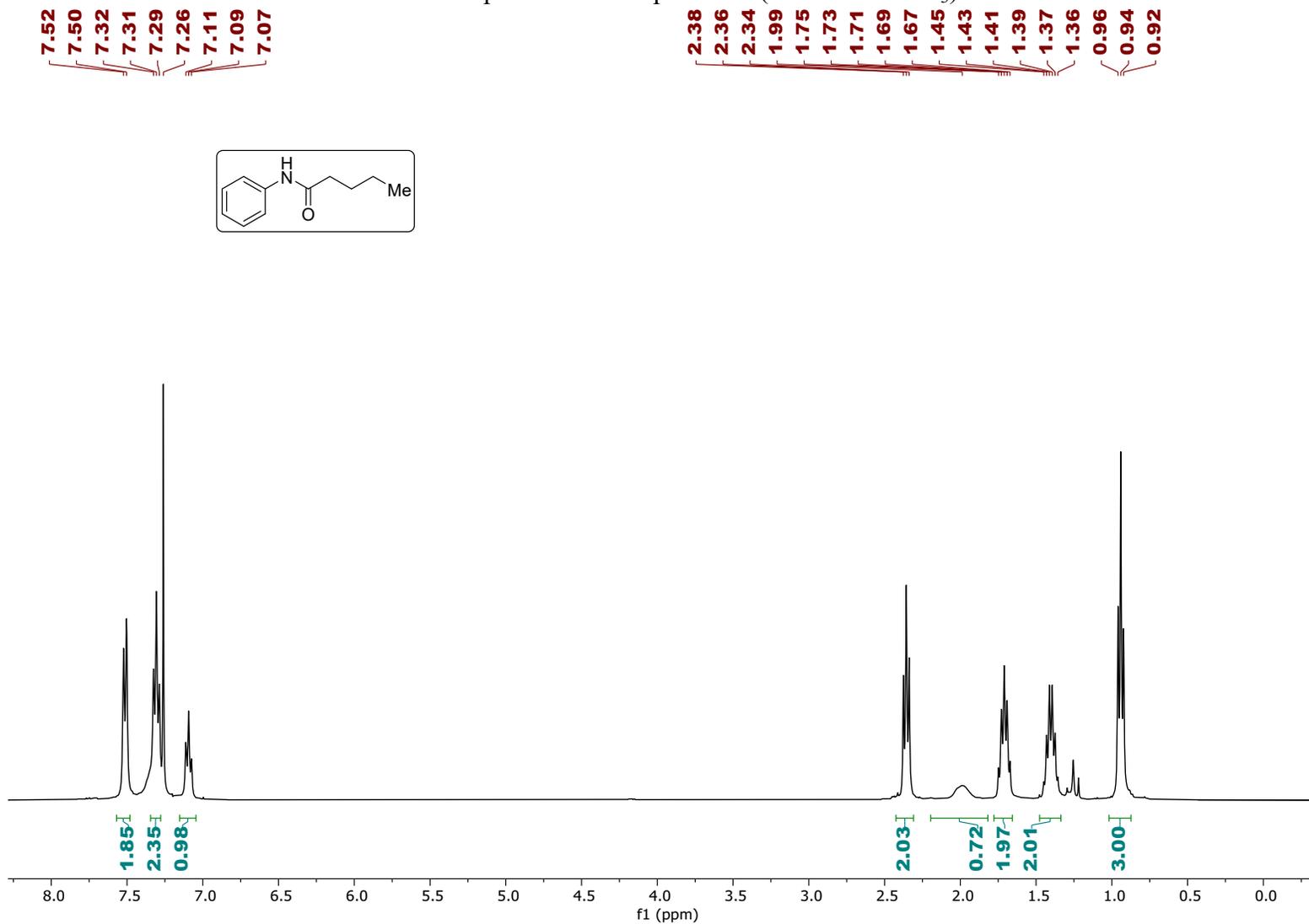
¹H NMR spectrum of compound **5b** (400 MHz/CDCl₃)

7.82
7.54
7.52
7.31
7.29
7.27
7.26
7.11
7.09
7.07

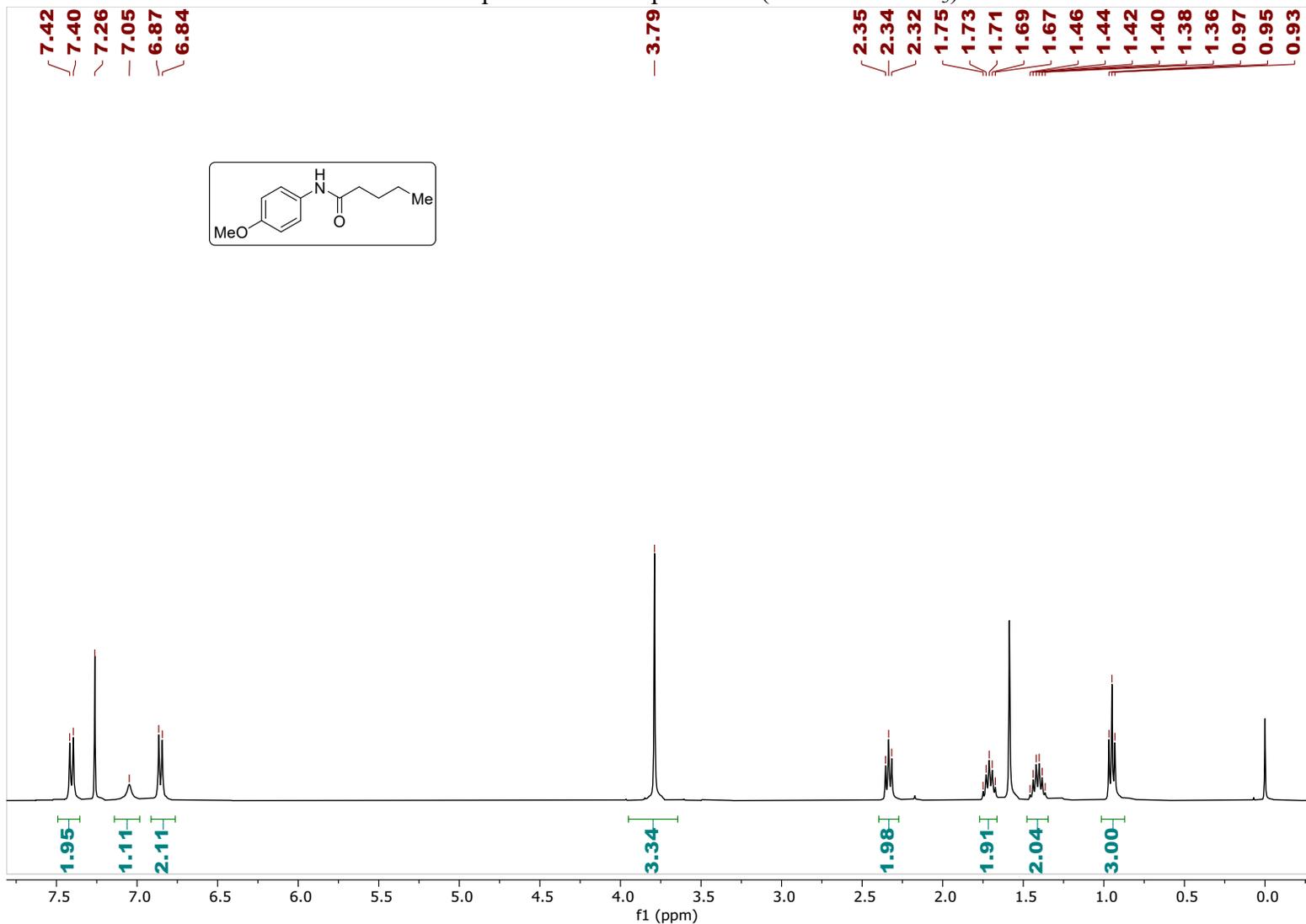
2.36
2.34
2.32
1.79
1.77
1.75
1.73
1.72
1.70
1.00
0.98
0.96



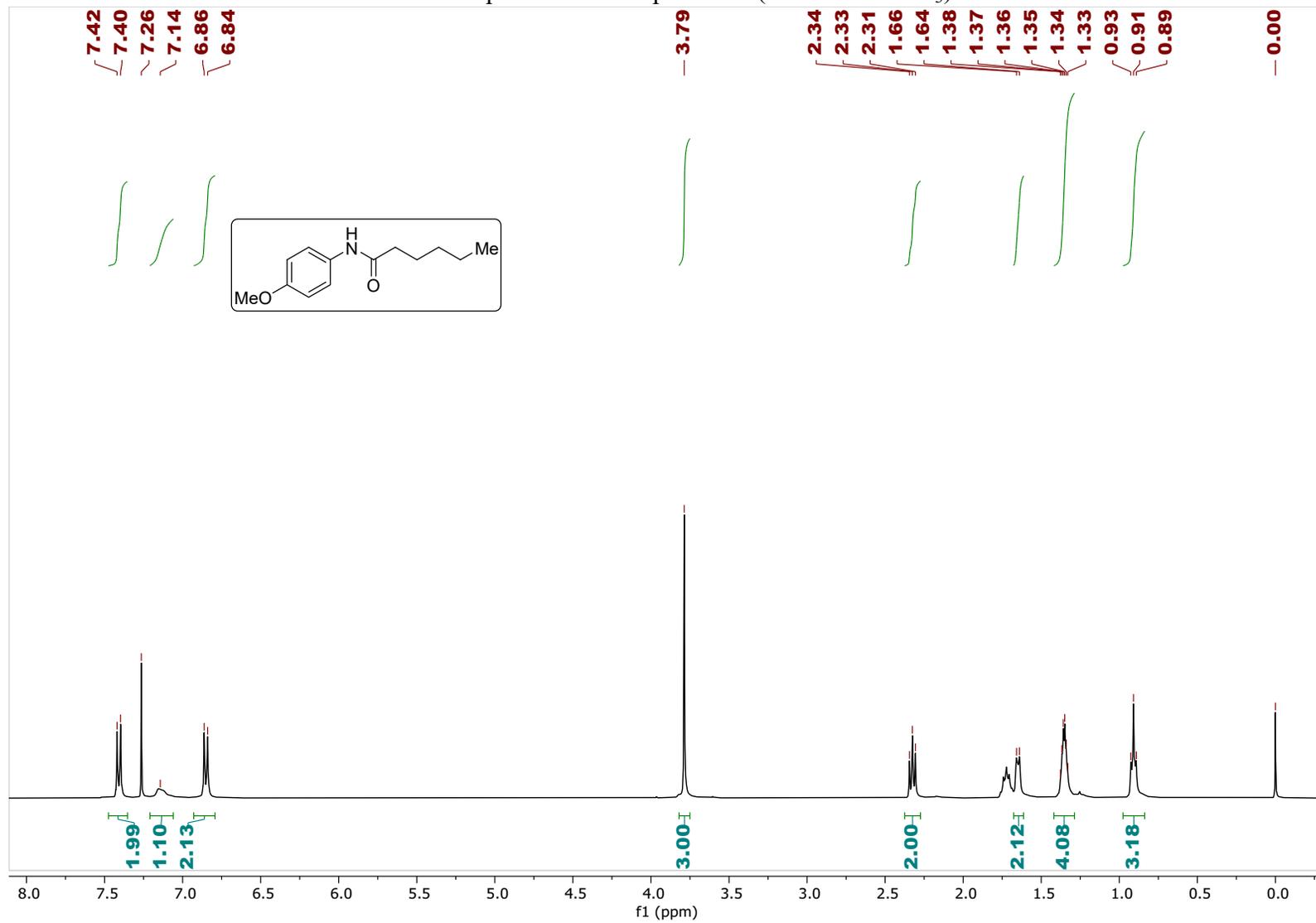
¹H NMR spectrum of compound **5c** (400 MHz/CDCl₃)



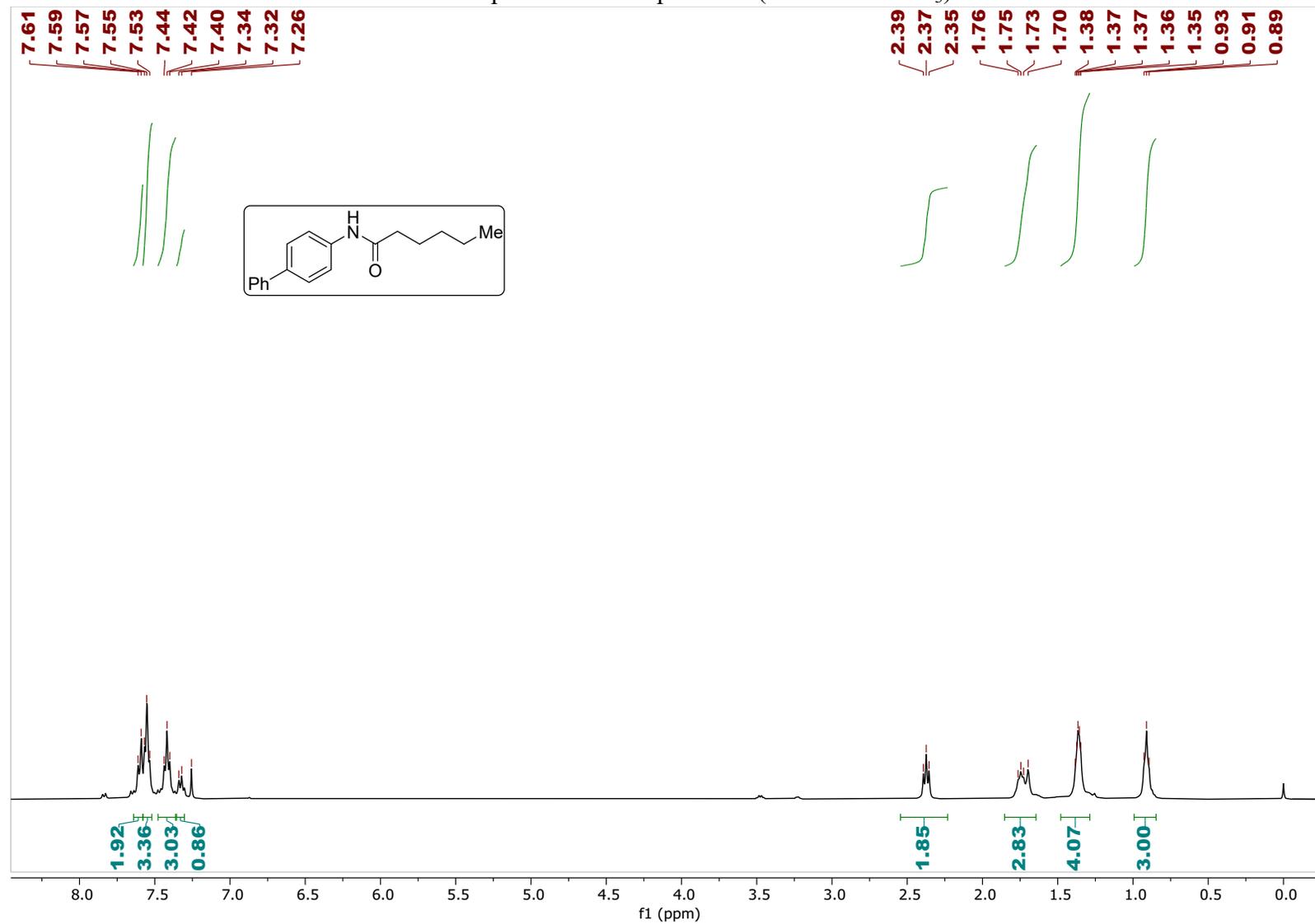
¹H NMR spectrum of compound **5d** (400 MHz/CDCl₃)



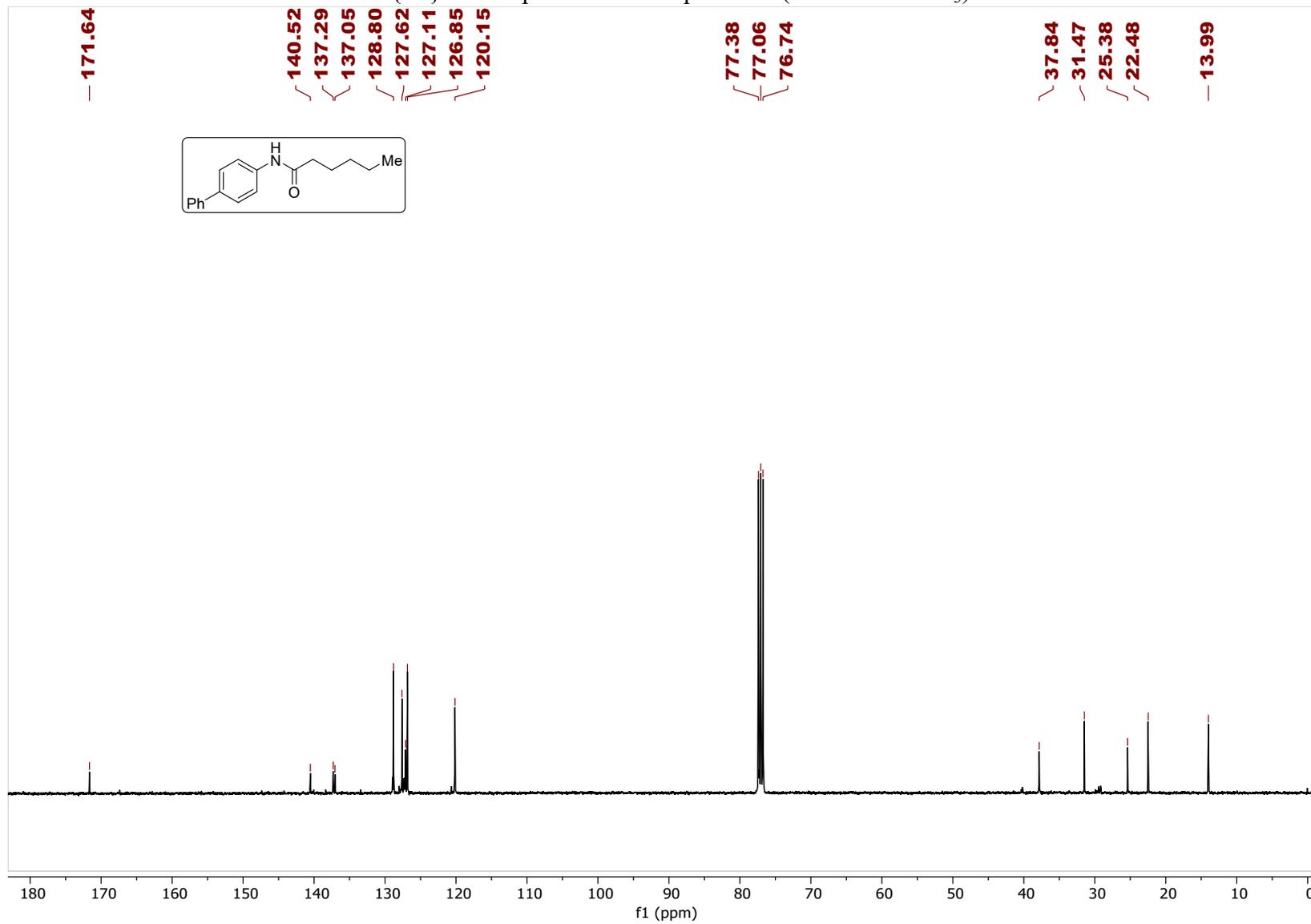
¹H NMR spectrum of compound **5e** (400 MHz/¹³CDCl₃)



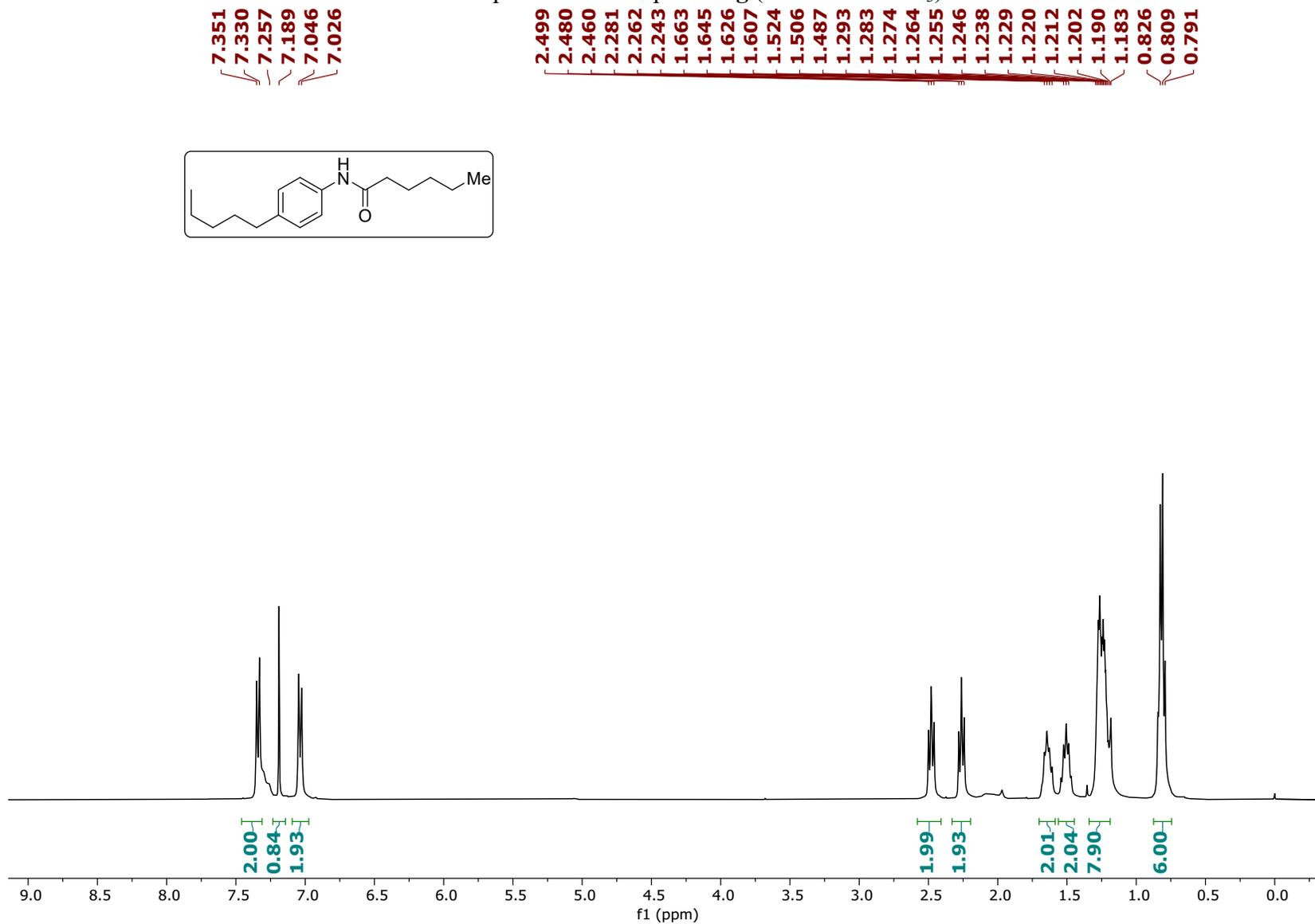
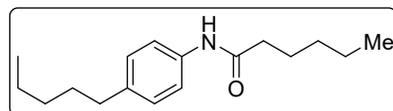
¹H NMR spectrum of compound **5f** (400 MHz/CDCl₃)



$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **5f** (101 MHz/ CDCl_3)



¹H NMR spectrum of compound **5g** (400 MHz/CDCl₃)

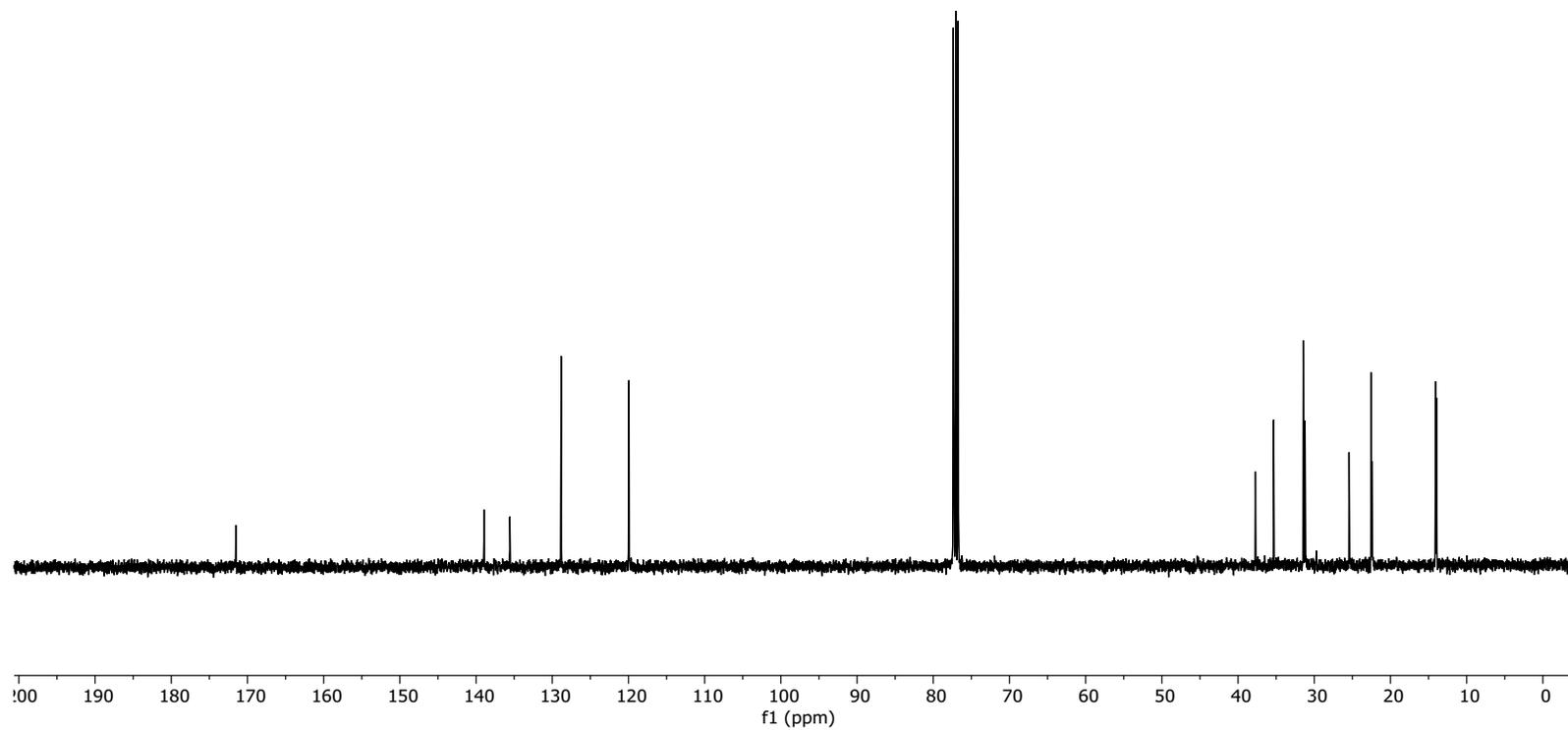
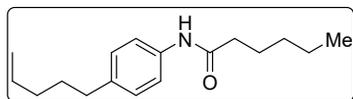


$^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of compound **5g** (101 MHz/ CDCl_3)

— 171.525
~ 138.961
~ 135.570
~ 128.835
— 119.943

{ 77.375
{ 77.054
{ 76.738

{ 37.729
{ 35.337
{ 31.452
{ 31.434
{ 31.221
{ 25.435
{ 22.553
{ 22.461
{ 14.054
{ 13.966



¹H NMR spectrum of compound **5h** (400 MHz/CDCl₃)

