*Supporting Information*

# Metal-free synthesis of secondary amides using *N*-Boc-*O*-tosylhydroxylamine as nitrogen source *via* Beckmann rearrangement

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**List of Contents:**

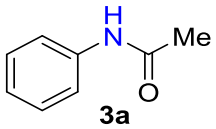
## 1. General Information:

Unless otherwise stated, all the reactions were carried out using oven dried glassware under an open atmosphere in a round bottom flask with a magnetic stirring bar at room temperature. Ketones were used as received without further purification. The aminating reagents were also prepared by following reported literature. TLC was carried out on pre-coated plates (Merck silica gel 60, $F_{254}$) and the spots were visualized with UV light or by charring the plates dipped in PMA or Ninhydrin or DNP solution. The compounds were purified by flash column chromatography using silica gel (100-200 mesh) with distilled solvents (EtOAc: Hexane) as mobile phase otherwise mentioned. $^{1}H$ and $^{13}C$ NMR spectra were recorded at 400 MHz and 100 MHz instruments respectively in $CDCl_3$ or DMSO-$d_6$ solvents. Chemical shifts (δ) are given in ppm. The residual solvent signals were used as references ($CDCl_3$: δ H = 7.26 ppm, DMSO-$d_6$: δ H = 2.5 ppm). The following abbreviations were used to explain NMR peak multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and br s = broad signal.

**2. Preparation of aminating reagents:** Aminatings **2a,**[1] **2b,**[2] **2c**[3] and **2d**[4] were prepared according to reported procedures.

**3. General procedure for preparation of *sec*-amides from ketones:** To a stirring solution of ketones (0.5 mmol, 1.0 equiv.) in TFE (0.5 mL) at room temperature in an open round bottom flask, aminating agent (0.75 mmol, 1.5 equiv.) was added. The reaction mixture was stirred at a specified temperature and duration as indicated in Scheme **2**. After completion, the reaction mixture was diluted with ethyl acetate (10 mL) and washed with a saturated aqueous solution of $NaHCO_3$ (3×5 mL). The organic layer was washed with brine solution (5 mL) and dried over anhydrous $Na_2SO_4$. The crude product was obtained after the removal of all volatiles in vacuo and was washed with *n*-hexane to remove some minor non-polar impurities (for 3a-d, 3g, 3k-n and 3r) or passed through a plug of silica gel using ethyl acetate and hexane as eluent to get pure amides.

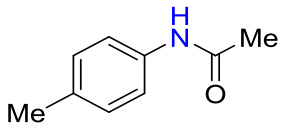
## 4. Characterization data of the products:




**3a**

***N*-Phenylacetamide (3a):** Prepared according to general procedure and titled amide was isolated as white solid (65 mg, 95% yield; mp 113–115 °C) whose spectral data was consistent with the literature values.[5]

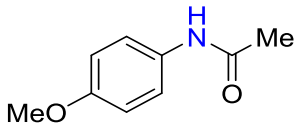
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.0 Hz, 2H), 7.31 (t, *J* = 7.7 Hz, 2H), 7.10 (t, *J* = 7.3 Hz, 1H), 2.16 (s, 3H).




**3b**

***N*-(p-Tolyl)acetamide (3b):** Prepared according to general procedure and titled amide was isolated as white solid (67 mg, 90% yield; mp 151–152 °C) whose spectral data was consistent with the literature values.[5]

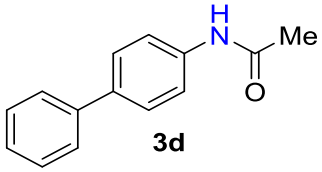
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.1 Hz, 2H), 7.20 (br s, 1H), 7.11 (d, *J* = 8.1 Hz, 2H), 2.31 (s, 3H), 2.16 (s, 3H).




**3c**

***N*-(4-Methoxyphenyl)acetamide (3c):** Prepared according to general procedure and titled amide was isolated as white solid (79 mg, 95% yield; mp = 130–132°C) whose spectral data was consistent with the literature values.[5]

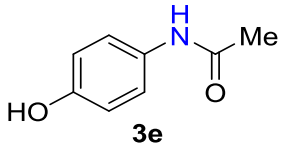
$^1$H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.5 Hz, 2H), 7.59 (d, *J* = 8.3 Hz, 2H), 7.48 (br s, 1H), 3.90 (s, 3H), 2.21 (s, 3H).




**3d**

***N*-([1,1'-biphenyl]-4-yl)acetamide (3d):** Prepared according to general procedure and titled amide was isolated as white solid (95 mg, 90% yield; mp = 170–172 °C) whose spectral data was consistent with the literature values.[6]

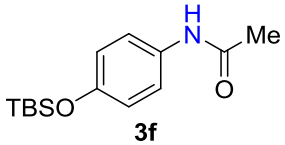
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.81 (s, 1H), 7.65 – 7.49 (m, 6H), 7.42 (t, *J* = 7.3 Hz, 2H), 7.37 – 7.29 (m, 1H), 2.19 (s, 3H).




**3e**

***N*-(4-Hydroxyphenyl)acetamide (3e):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 1:1, v/v) afforded the titled amide as brown solid (65 mg, 86% yield; mp 169–170 °C), whose spectral data was consistent with the literature values.[5]

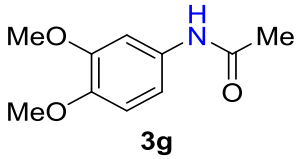
$^1$H NMR (400 MHz, DMSO-$d_6$) δ 9.64 (s, 1H), 9.13 (s, 1H), 7.33 (d, $J$ = 8.7 Hz, 2H), 6.66 (d, $J$ = 8.7 Hz, 2H), 1.97 (s, 3H).




**3f**

***N*-(4-((*tert*-butyldimethylsilyl)oxy)phenyl)acetamide (3f):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 3:1, v/v) afforded the titled amide as white solid (120 mg, 90% yield; mp = 120–122 °C) whose spectral data was consistent with the literature values.[7]

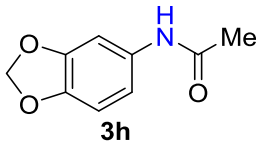
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.65 (br s, 1H), 7.32 (d, $J$ = 7.1 Hz, 2H), 6.75 (d, $J$ = 7.1 Hz, 2H), 2.10 (s, 3H), 0.95 (s, 9H), 0.15 (s, 6H).




**3g**

***N*-(3,4-Dimethoxyphenyl)acetamide (3g):** Prepared according to general procedure and titled amide was isolated as white solid (87 mg, 89% yield; mp 125–128 °C) whose spectral data was consistent with the literature values.[8]

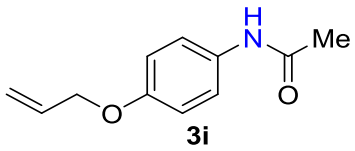
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, $J$ = 1.9 Hz, 1H), 7.17 (br s, 1H), 6.88-6.76 (m, 2H), 3.87 (s, 3H), 3.85 (s, 3H), 2.16 (s, 3H).




**3h**

***N*-(benzo[*d*][1,3]dioxol-5-yl)acetamide (3h):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 4:1, v/v) afforded the titled amide as white solid (86 mg, 96% yield; mp = 92–95 °C) whose spectral data was consistent with the literature values.[9]

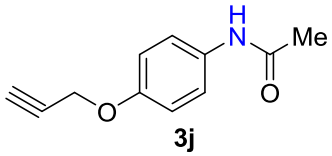
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.64 (br s, 1H), 7.17 (d, *J* = 2.0 Hz, 1H), 6.80 – 6.67 (m, 2H), 5.92 (s, 2H), 2.11 (s, 3H).




**3i**

***N*-(4-(Allyloxy)phenyl)acetamide (3i):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 3:1, v/v) afforded the titled amide as white solid (92 mg, 96% yield; mp = 92–95 °C) whose spectral data was consistent with the literature values.[10]

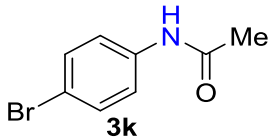
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.8 Hz, 2H), 7.14 (br s, 1H), 6.87 (d, *J* = 8.8 Hz, 2H), 6.11 – 5.97 (m, 1H), 5.40 (d, *J* = 16.9 Hz, 1H), 5.28 (d, *J* = 10.6 Hz, 1H), 4.51 (d, *J* = 5.2 Hz, 2H), 2.15 (s, 3H).




**3j**

***N*-(4-(prop-2-yn-1-yloxy)phenyl)acetamide (3j):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 3:1, v/v) afforded the titled amide as white solid (90 mg, 95% yield; mp = 92–95 °C) whose spectral data was consistent with the literature values.[11]

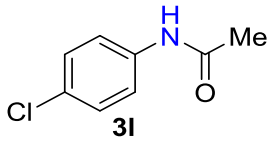
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.59 (br s, 1H), 7.40 (d, *J* = 8.5 Hz, 2H), 6.91 (d, *J* = 8.4 Hz, 2H), 4.65 (s, 2H), 2.51 (s, 1H), 2.12 (s, 3H).




**3k**

***N*-(4-Bromophenyl)acetamide (3k):** Prepared according to general procedure and titled amide was isolated as white solid (97.4 mg, 91% yield; mp = 166–170 °C) whose spectral data was consistent with the literature values.[5]

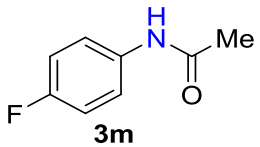
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.36 (m, 4H), 7.18 (br s, 1H), 2.17 (s, 3H).




**3l**

***N*-(4-Chlorophenyl)acetamide (3l):** Prepared according to general procedure and titled amide was isolated as white solid (76 mg, 90% yield; mp = 176–179 °C) whose spectral data was consistent with the literature values.[12]

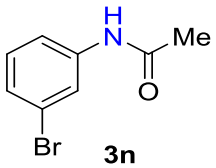
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.7 Hz, 2H), 2.17 (s, 3H).




**3m**

***N*-(4-Fluorophenyl)acetamide (3m):** Prepared according to general procedure and titled amide was isolated as white solid (66 mg, 86% yield; mp 155–158°C) whose spectral data was consistent with the literature values.[5]

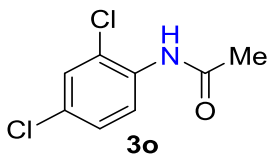
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.45 (dd, *J* = 8.7, 4.8 Hz, 2H), 7.38 (brs, 1H), 7.00 (t, *J* = 8.6 Hz, 2H), 2.16 (s, 3H)




**3n**

***N*-(3-Bromophenyl)acetamide (3n):** Prepared according to general procedure and titled amide was isolated as white solid (97 mg, 90% yield; mp 83–85 °C) whose spectral data was consistent with the literature values.[9]

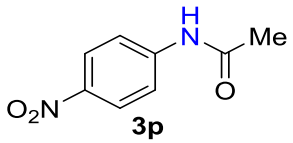
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.76 (s, 1H), 7.40 (d, *J* = 7.8 Hz, 1H), 7.25 – 7.13 (m, 2H), 2.18 (s, 3H).




**3o**

***N*-(2,4-Dichlorophenyl)acetamide (3o):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 4:1, v/v) afforded the titled amide as brown solid (92 mg, 90% yield; mp = 142–144 °C) whose spectral data was consistent with the literature values.[13]

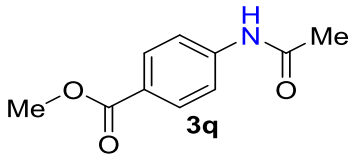
$^1$H NMR (400 MHz, $CDCl_3$) δ 8.33 (d, *J* = 8.8 Hz, 1H), 7.56 (br s, 1H), 7.37 (d, *J* = 2.4 Hz, 1H), 7.25 – 7.20 (m, 1H), 2.24 (s, 3H).



**3p**

***N*-(4-Nitrophenyl)acetamide (3p):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 4:1, v/v) afforded the titled amide as yellow solid (81 mg, 90% yield; mp 210–212 °C) whose spectral data was consistent with the literature values.[14]

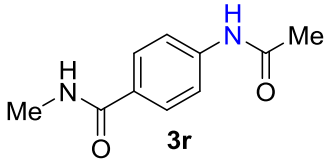
$^1$H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 9.0 Hz, 2H), 7.69 (d, *J* = 9.0 Hz, 2H), 7.46 (br s, 1H), 2.25 (s, 3H).



**3q**

**Methyl 4-acetamidobenzoate (3q):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 4:1, v/v) afforded the titled amide as white solid (81 mg, 84% yield; mp 112-115 °C) whose spectral data was consistent with the literature values.[5]

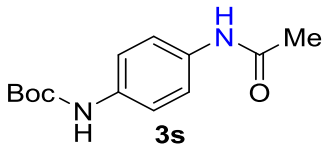
$^1$H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.5 Hz, 2H), 7.59 (d, *J* = 8.3 Hz, 2H), 7.48 (br s, 1H), 3.90 (s, 3H), 2.21 (s, 3H).



**3r**

**4-acetamido-*N*-methylbenzamide (3r):** Prepared according to general procedure and titled amide was isolated as white solid (94 mg, 98% yield; mp 183-185 °C) whose spectral data was consistent with the literature values.[15]

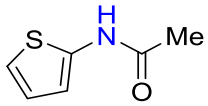
$^1$H NMR (400 MHz, DMSO-$d_6$) δ 10.04 (br s, 2H), 7.48 (s, 4H), 2.00 (s, 6H).



**3s**

***tert*-Butyl (4-acetamidophenyl)carbamate (3s):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 3:1, v/v) afforded the titled amide as white solid (106.4 mg, 85% yield; mp 162–165 °C) whose spectral data was consistent with the literature values.[5]

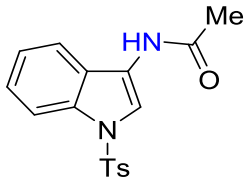
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.7 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.15 (br s, 1H), 6.45 (br s, 1H), 2.15 (s, 3H), 1.51 (s, 9H).



**3t**

***N*-(Thiophen-2-yl)acetamide (3t):** Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 5:1, v/v) afforded the titled amide as brown solid (35 mg, 50% yield; mp 158–160 °C) whose spectral data was consistent with the literature values.[5]

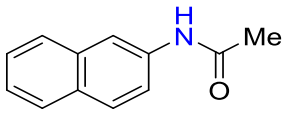
$^1$H NMR (400 MHz, $CDCl_3$) δ 8.20 (br s, 1H), 6.91 – 6.80 (m, 2H), 6.68 – 6.62 (m, 1H), 2.20 (s, 3H).



**3u**

***N*-(1-Tosyl-1*H*-indol-3-yl)acetamide (3u):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 4:1, v/v) afforded the titled amide as white solid (125 mg, 76% yield; mp 192–194 °C) whose spectral data was consistent with the literature values.[5]

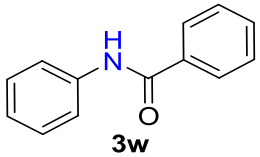
$^1$H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 1H), 8.07 (d, *J* = 8.4 Hz, 1H), 7.76 (d, *J* = 8.1 Hz, 2H), 7.41 – 7.32 (m, 2H), 7.29 (br s, 1H), 7.25 – 7.21 (m, 1H), 7.18 (d, *J* = 8.1 Hz, 2H), 2.31 (s, 3H), 2.24 (s, 3H).




**3v**

***N*-(Naphthalen-2-yl)acetamide (3v):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 3:1, v/v) afforded the titled amide as brown solid (85.2 mg, 92% yield; mp = 134–136 °C) whose spectral data was consistent with the literature values.[16]

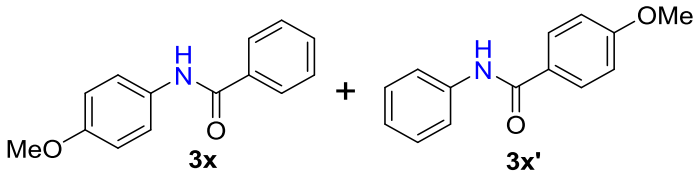
$^1$H NMR (400 MHz, $CDCl_3$) δ 8.18 (s, 1H), 7.78 (d, *J* = 7.8 Hz, 3H), 7.53 – 7.33 (m, 4H), 2.24 (s, 3H).




**3w**

***N*-Phenylbenzamide (3w):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 4:1, v/v) afforded the titled amide as white solid (69 mg, 70% yield; mp 168-170°C) whose spectral data was consistent with the literature values.[5]

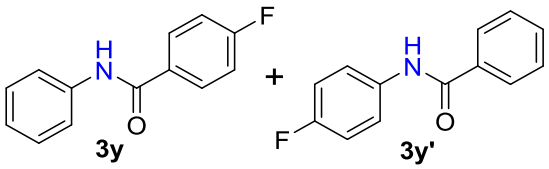
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.95 (br s, 1H), 7.86 (d, *J* = 7.4 Hz, 2H), 7.65 (d, *J* = 7.8 Hz, 2H), 7.58 – 7.51 (m, 1H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.36 (t, *J* = 7.7 Hz, 2H), 7.15 (t, *J* = 7.3 Hz, 1H).




**3x** **3x'**

***N*-(4-Methoxyphenyl)benzamide (3x) and 4-methoxy-N-phenylbenzamide (3x'):**[16] Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 4:1, v/v) afforded the titled amide as white solid (102 mg, 90% yield; 64:36 isomeric mixture; mp 110-115 °C) whose spectral data was consistent with the literature values.[17]

$^1$H NMR (400 MHz, DMSO-$d_6$) δ 10.07 (s, 1H), 10.03 (s, 0.6H), 7.89 (t, *J* = 7.9 Hz, 3.2H), 7.71 (d, *J* = 8.1 Hz, 1.2H), 7.62 (d, *J* = 8.7 Hz, 2H), 7.54 – 7.38 (m, 3H), 7.27 (t, *J* = 7.7 Hz, 1.2H), 7.07 – 6.93 (m, 1.8H), 6.86 (d, *J* = 8.8 Hz, 2H), 3.76 (s, 1.8H), 3.67 (s, 3H).

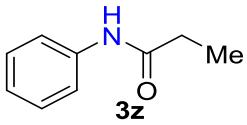



**4-fluoro-*N*-phenylbenzamide (3y) and *N*-(4-fluorophenyl)benzamide (3y'):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 5:1, v/v) afforded the titled amide as white solid (97 mg, 90% yield; 1:1 isomeric mixture; mp = 175-180 °C) whose spectral data was consistent with the literature values.[18]

$^{1}$H NMR (400 MHz, DMSO-$d_6$) δ 10.31 (s, 1H), 10.27 (s, 1H), 8.08 – 8.02 (m, 2H), 7.99 – 7.94 (m, 2H), 7.84 – 7.75 (m, 4H), 7.62 – 7.50 (m, 3H), 7.39 – 7.32 (m, 4H), 7.23 – 7.16 (m, 2H), 7.11 (t, *J* = 7.4 Hz, 1H).

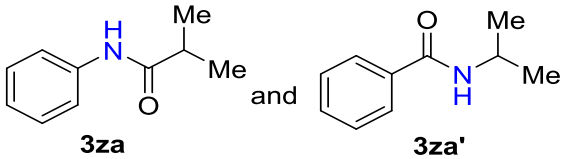
$^{13}$C NMR (100 MHz, DMSO-$d_6$) δ 165.52, 165.33, 164.48, 162.86, 159.54, 157.15, 139.09, 135.56, 135.53, 134.85, 131.59, 131.44, 131.41, 130.44, 130.35, 128.62, 128.40, 127.64, 123.75, 122.28, 122.20, 120.47, 115.42, 115.28, 115.20, 115.06.

$^{19}$F NMR (376 MHz, DMSO-$d_6$) δ -108.77 (s, 1F), -118.83 (s, 1F).




***N*-Phenylpropionamide (3z):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 5:1, v/v) afforded the titled amide as white solid (67 mg, 90% yield; mp 108-110°C) whose spectral data was consistent with the literature values.[19]

$^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.16 (br s, 1H), 7.53 (d, *J* = 7.9 Hz, 2H), 7.26 (t, *J* = 7.7 Hz, 2H), 7.06 (t, *J* = 7.2 Hz, 1H), 2.35 (q, *J* = 7.5 Hz, 2H), 1.19 (t, *J* = 7.5 Hz, 3H).




***N*-Phenylisobutyramide (3za) and *N*-isopropylbenzamide (3za'):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl

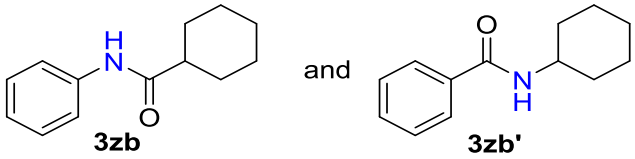
acetate = 5:1, v/v) afforded **3za** as white solid (53 mg, 65% yield; mp 110–111°C) along with **3za'** (19 mg, 24% yield) whose spectral data was consistent with the literature values.[5,20]

***N*-Phenylisobutyramide (3za):**[5]

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.0 Hz, 2H), 7.32 (t, *J* = 7.7 Hz, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 2.59 – 2.43 (m, 1H), 1.26 (d, *J* = 6.8 Hz, 6H).

***N*-Isopropylbenzamide (3za'):**[20]

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.69 (m, 2H), 7.51 – 7.33 (m, 3H), 6.09 (s, 1H), 4.36 – 4.19 (m, 1H), 1.24 (d, *J* = 6.5 Hz, 6H).



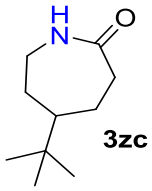

***N*-Phenylcyclohexanecarboxamide (3zb) and *N-cyclohexylbenzamide* (3zb'):** Prepared according to general procedure and crude was purified by silica gel column chromatography (hexane/ethyl acetate = 5:1, v/v) afforded the titled amide **3z** as white solid (71 mg, 70% yield; mp 141–144 °C) along with **3zb'** white solid (18 mg, 18% yield, mp 141–144 °C**)** whose spectral data was consistent with the literature values.[5]

***N*-Phenylcyclohexanecarboxamide (3zb):**

$^1$H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.9 Hz, 2H), 7.35 – 7.24 (m, 3H), 7.08 (t, *J* = 7.3 Hz, 1H), 2.27 – 2.18 (m, 1H), 1.95 (d, *J* = 12.6 Hz, 2H), 1.83 (d, *J* = 11.1 Hz, 2H), 1.73 – 1.66 (m, 1H), 1.60 – 1.49 (m, 2H), 1.38 – 1.25 (m, 3H).

***N*-cyclohexylbenzamide (3zb'):**

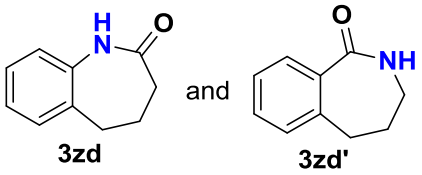
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, *J* = 7.4 Hz, 2H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.42 (t, *J* = 7.4 Hz, 2H), 5.96 (br s, 1H), 4.07 – 3.89 (m, 1H), 2.09 – 1.96 (m, 2H), 1.80 – 1.71 (m, 2H), 1.70 – 1.65 (m, 1H), 1.48 – 1.37 (m, 2H), 1.30 – 1.17 (m, 3H).




**5-(*tert*-Butyl)azepan-2-one (3zc):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 3:1, v/v) afforded the

titled amide as white solid (64 mg, 75% yield; mp 150-154 °C) whose spectral data was consistent with the literature values.[21]

$^1$H NMR (400 MHz, $CDCl_3$) δ 6.50 (br s, 1H), 3.28 – 3.14 (m, 2H), 2.57 – 2.28 (m, 2H), 2.02 – 1.89 (m, 2H), 1.32 – 1.14 (m, 3H), 0.86 (s, 9H).




3zd 3zd'

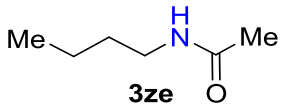
**1,3,4,5-tetrahydro-2*H*-benzo[*b*]azepin-2-one (3zd) and 2,3,4,5-tetrahydro-1*H*-benzo[*c*]-azepin-1-one (3zd'):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 5:1, v/v) afforded the titled amide as white solid (53 mg, 66% yield; mp 136-138 °C) along with **3zd'** as white solid (20 mg, 25% yield; mp 100-103 °C) whose spectral data was consistent with the literature values.[17, 22]

**1,3,4,5-tetrahydro-2*H*-benzo[*b*]azepin-2-one (3zd):**[17]

$^1$H NMR (400 MHz, $CDCl_3$) δ 9.14 (br s, 1H), 7.25 – 7.14 (m, 2H), 7.14 – 7.06 (m, 1H), 7.03 (d, *J* = 7.7 Hz, 1H), 2.80 (t, *J* = 7.2 Hz, 2H), 2.35 (t, *J* = 7.2 Hz, 2H), 2.29 – 2.15 (m, 2H).

**2,3,4,5-tetrahydro-1*H*-benzo[*c*]azepin-1-one (3zd'):** [22]

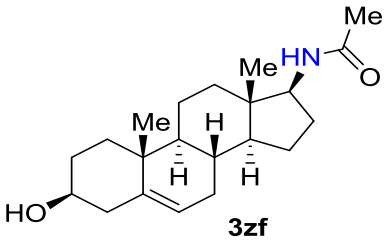
$^1$H NMR (400 MHz, $CDCl_3$) δ 7.75 (br s, 1H), 7.68 (dd, *J* = 7.5, 1.6 Hz, 1H), 7.37 (td, *J* = 7.4, 1.6 Hz, 1H), 7.31 (td, *J* = 7.5, 1.4 Hz, 1H), 7.16 (d, *J* = 7.8 Hz, 1H), 3.10 (q, *J* = 6.4 Hz, 2H), 2.83 (t, *J* = 7.1 Hz, 2H), 2.05 – 1.93 (m, 2H).




3ze

***N*-Butylacetamide (3ze):** Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 4:1, v/v) afforded the titled amide as clear oil (52 mg, 90% yield) whose spectral data was consistent with the literature values.[8]

$^1$H NMR (400 MHz, $CDCl_3$) δ 6.84 (br s, 3H), 3.22 (dd, *J* = 13.1, 7.0 Hz, 2H), 1.98 (s, 3H), 1.49 (dt, *J* = 14.9, 7.3 Hz, 2H), 1.43 – 1.28 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H).




**3zf**

***N*-((*3S,8R,9S,10R,13S,14S,17S*)-3-Hydroxy-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*] phen-anthren-17-yl)acetamide (3zf):**[5] Prepared according to general procedure and crude was purified by silica gel column choromatography (hexane/ethyl acetate = 3:1, v/v) afforded the titled amide as white solid (149 mg, 90% yield; mp 234–236 °C) whose spectral data was consistent with the literature values.[5]

$^1$H NMR (400 MHz, $CDCl_3$) δ 5.37 – 5.32 (m, 1H), 5.31 – 5.23 (m, 1H), 3.89 (q, *J* = 9.0 Hz, 1H), 3.52 (td, *J* = 11.1, 5.5 Hz, 1H), 2.35 – 2.06 (m, 3H), 2.05 – 1.93 (m, 4H), 1.84 (d, *J* = 10.2 Hz, 2H), 1.69 (dd, *J* = 15.7, 11.8 Hz, 2H), 1.64 – 1.53 (m, 3H), 1.53 – 1.35 (m, 3H), 1.34 – 1.18 (m, 3H), 1.16 – 1.03 (m, 2H), 1.01 (s, 3H), 0.96 (dd, *J* = 11.5, 4.6 Hz, 1H), 0.70 (s, 3H).

## 5. References: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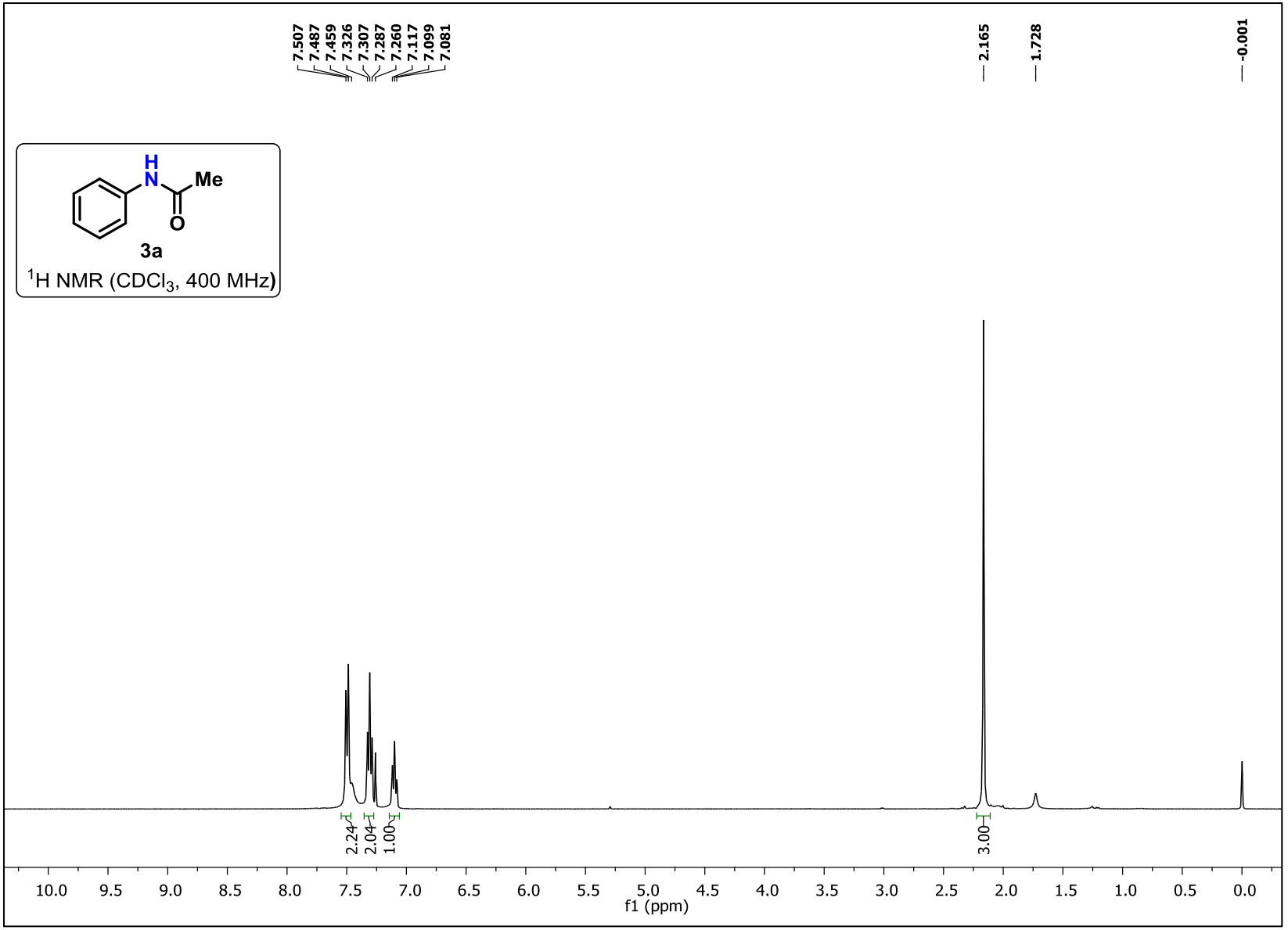
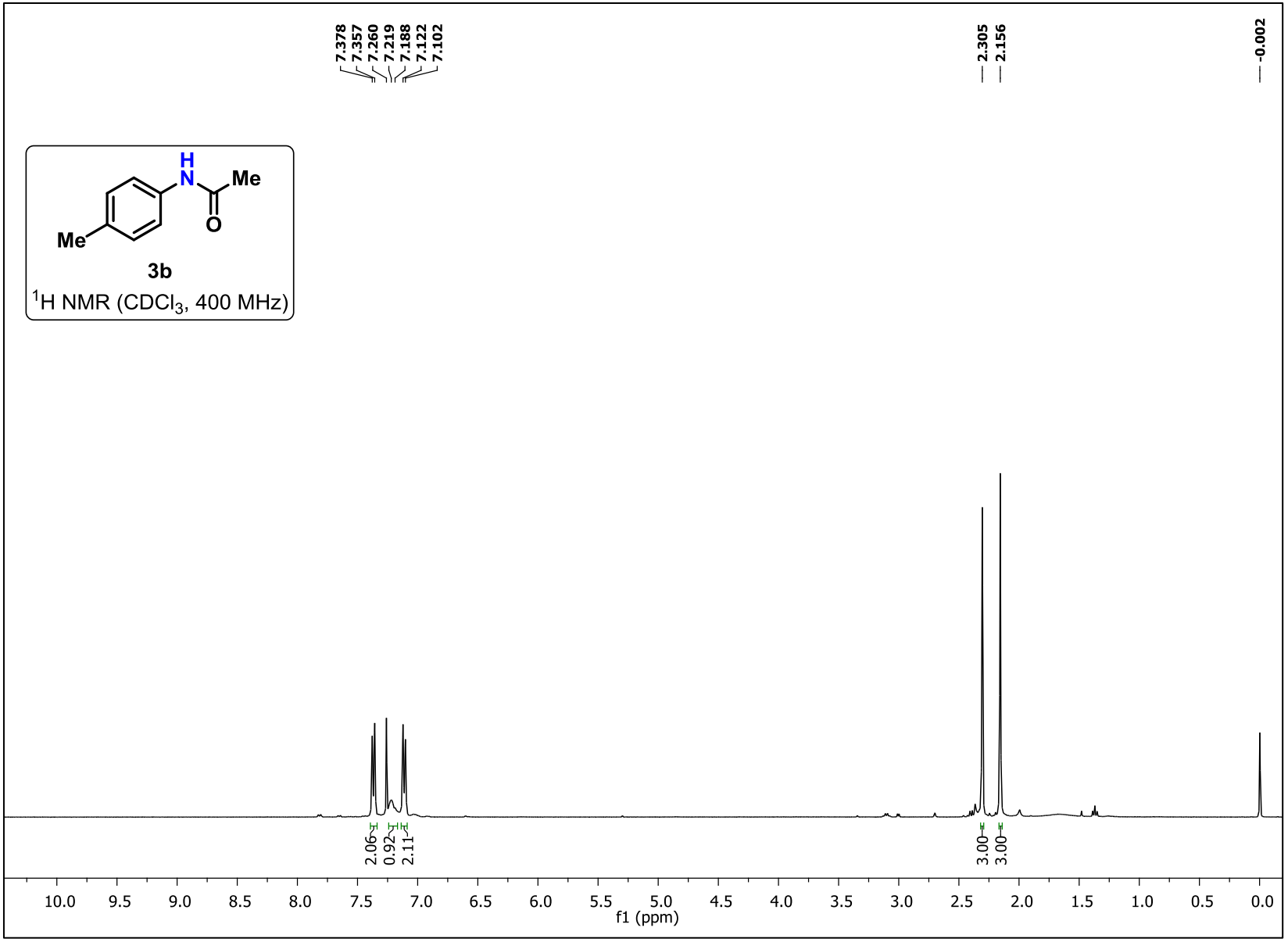
## 6. NMR spectra of products

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7.357
7.260
7.219
7.188
7.122
7.102
2.305
2.156
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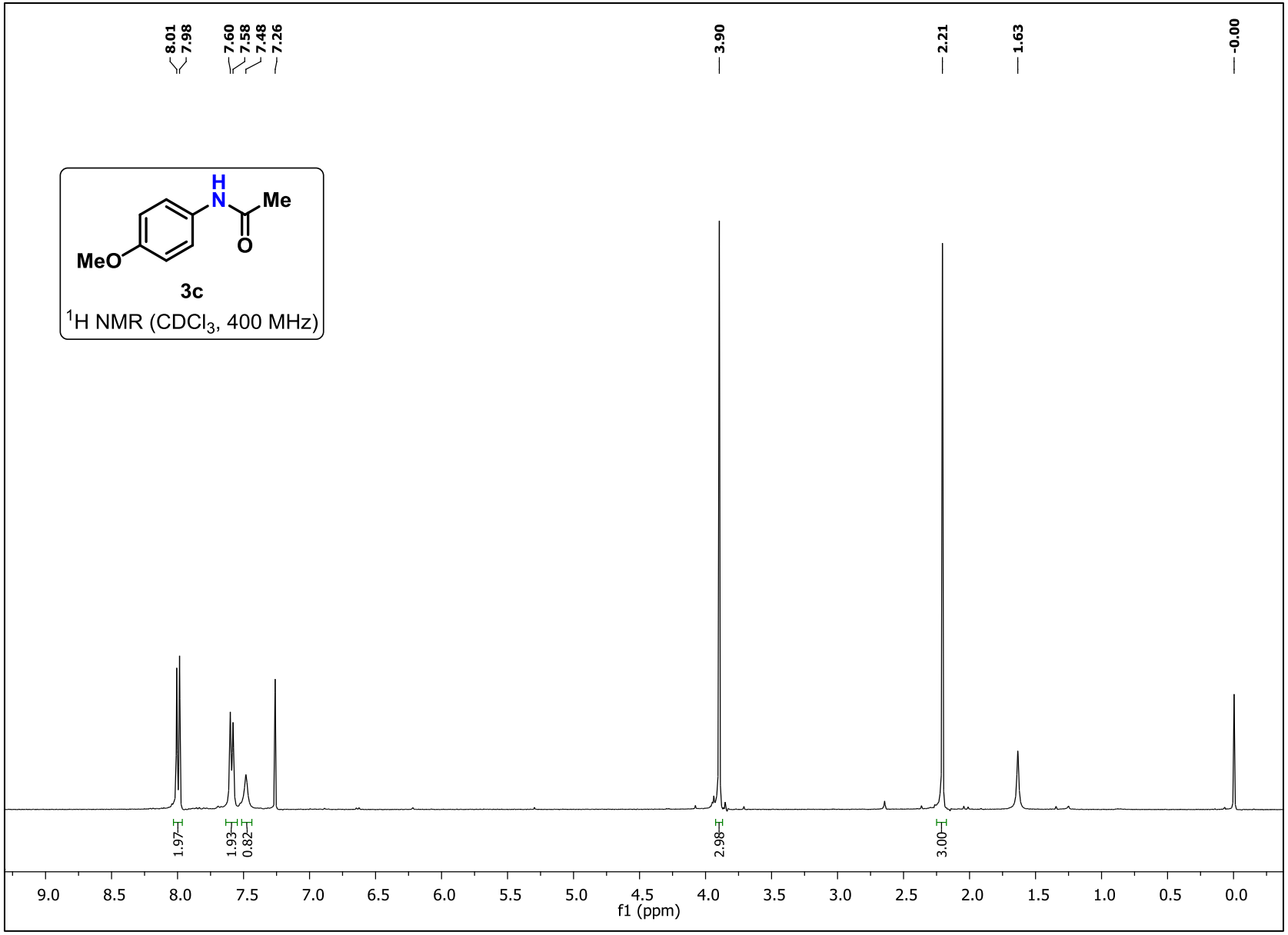
H
N
Me
Me
O
3b
1H NMR (CDCl3, 400 MHz)
2.06
0.92
2.11
3.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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7.98
7.60
7.58
7.48
7.26
3.90
2.21
1.63
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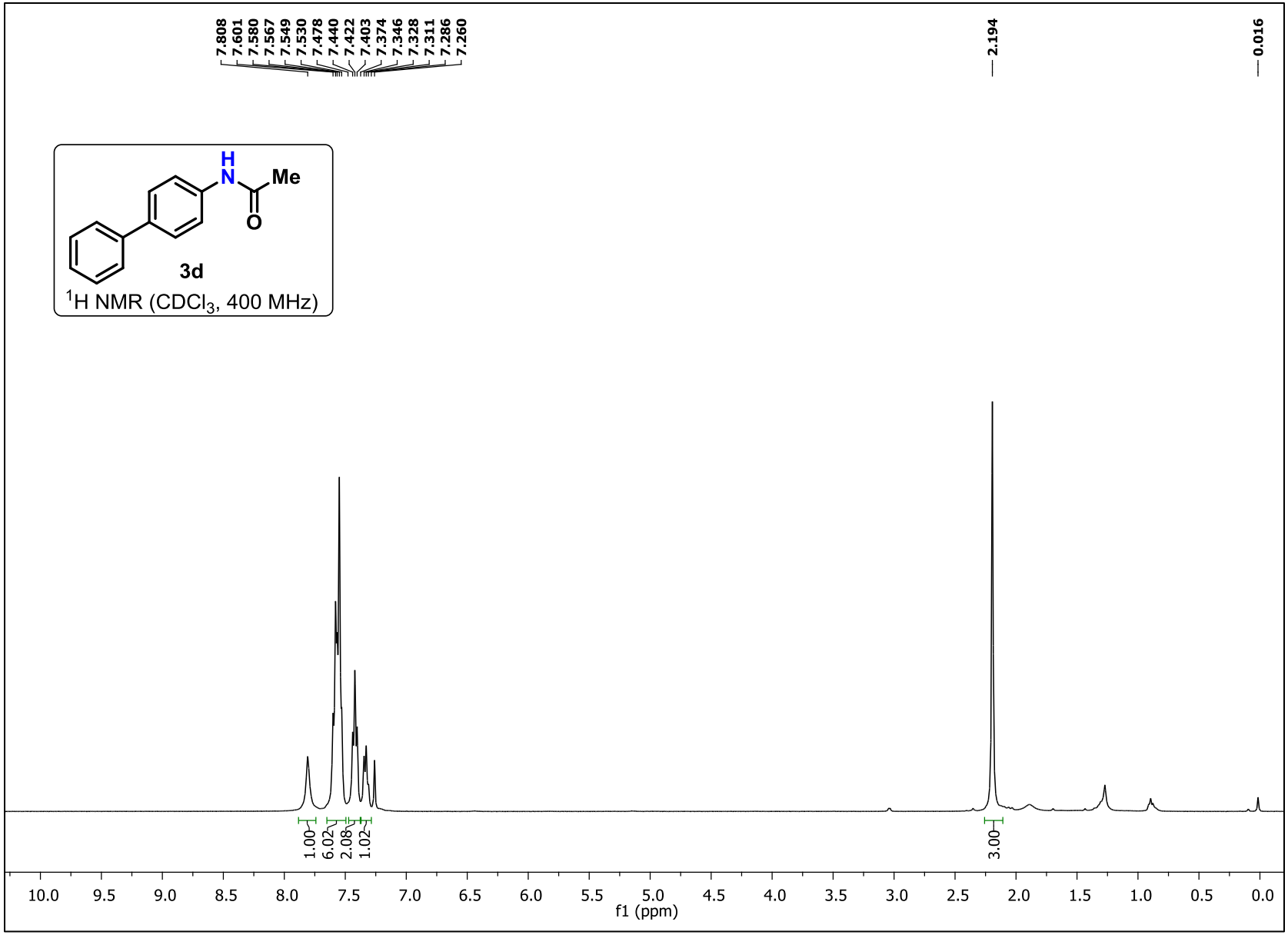
H
N
Me
O
MeO
3c
1H NMR (CDCl3, 400 MHz)
1.97
1.93
0.82
2.98
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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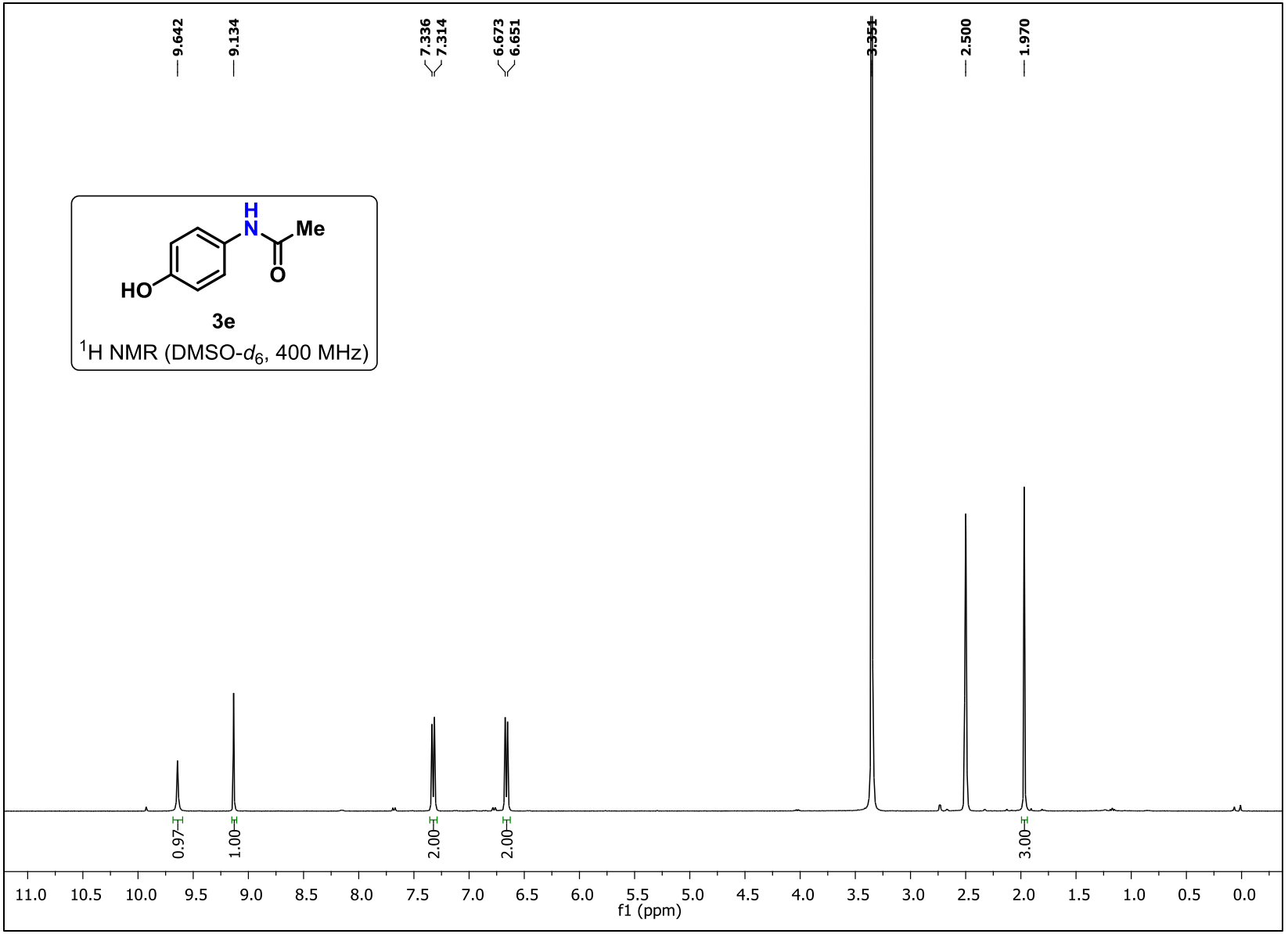
7.580
7.567
7.549
7.530
7.478
7.440
7.422
7.403
7.374
7.346
7.328
7.311
7.286
7.260
2.194
0.016
H
N
Me
O
3d
1H NMR (CDCl3, 400 MHz)
1.00
6.02
2.08
1.02
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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6.673
6.651
3.351
2.500
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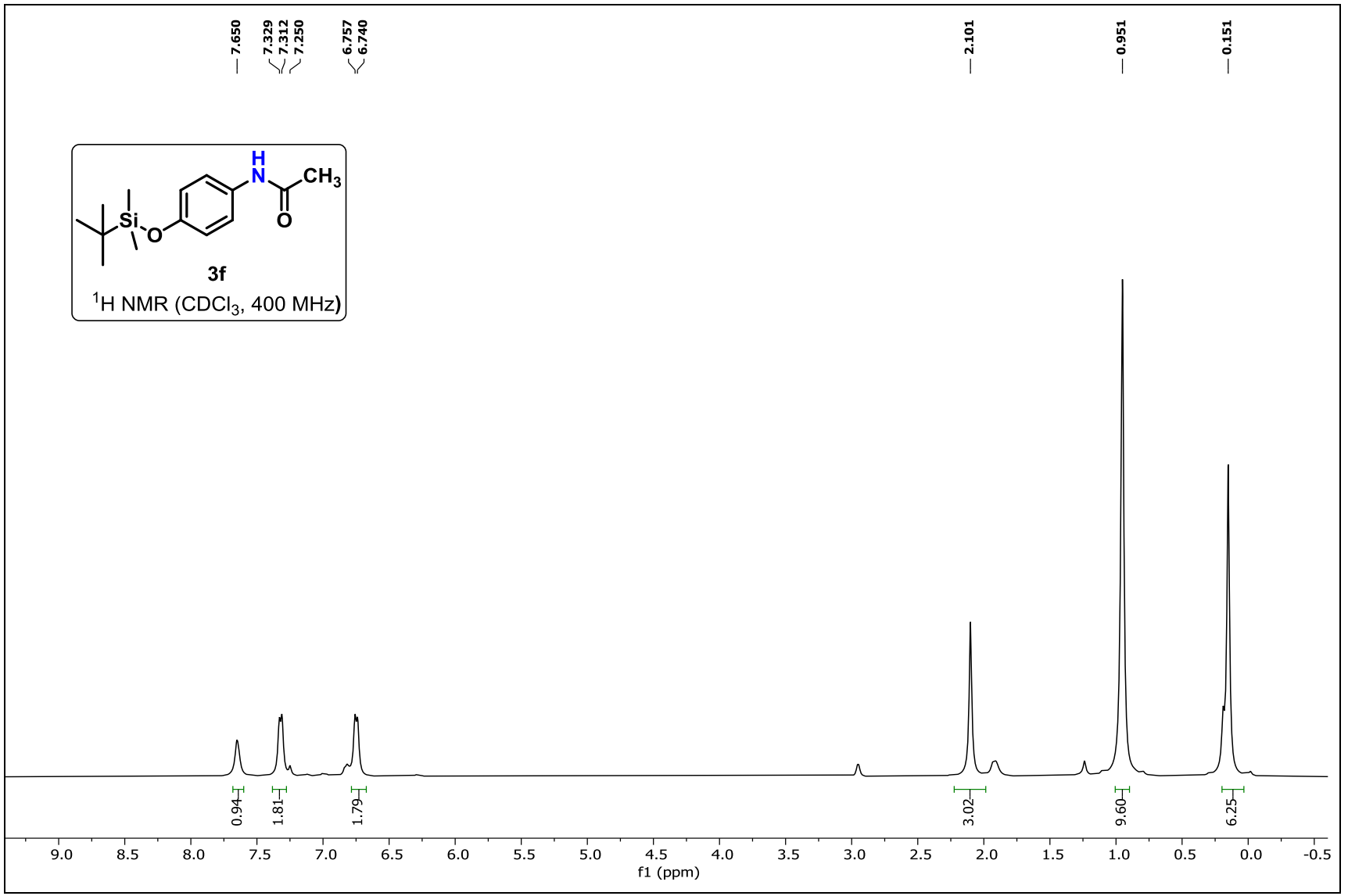
HO
N
H
Me
O
3e
1H NMR (DMSO-d6, 400 MHz)
0.97
1.00
2.00
2.00
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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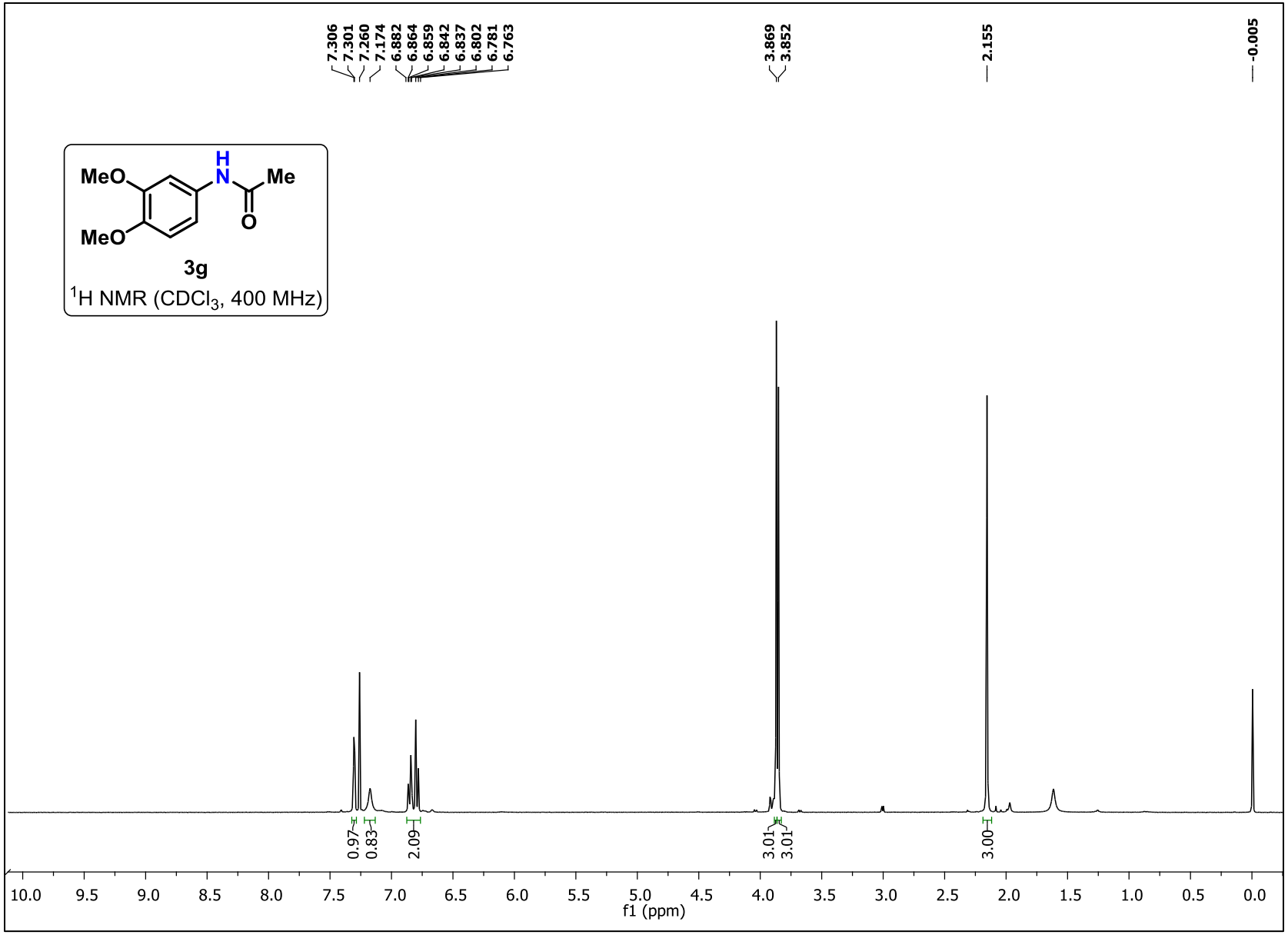
6.757
6.740
2.101
0.951
0.151
H
N
CH3
O
Si
O
3f
1H NMR (CDCl3, 400 MHz)
0.94
1.81
1.79
3.02
9.60
6.25
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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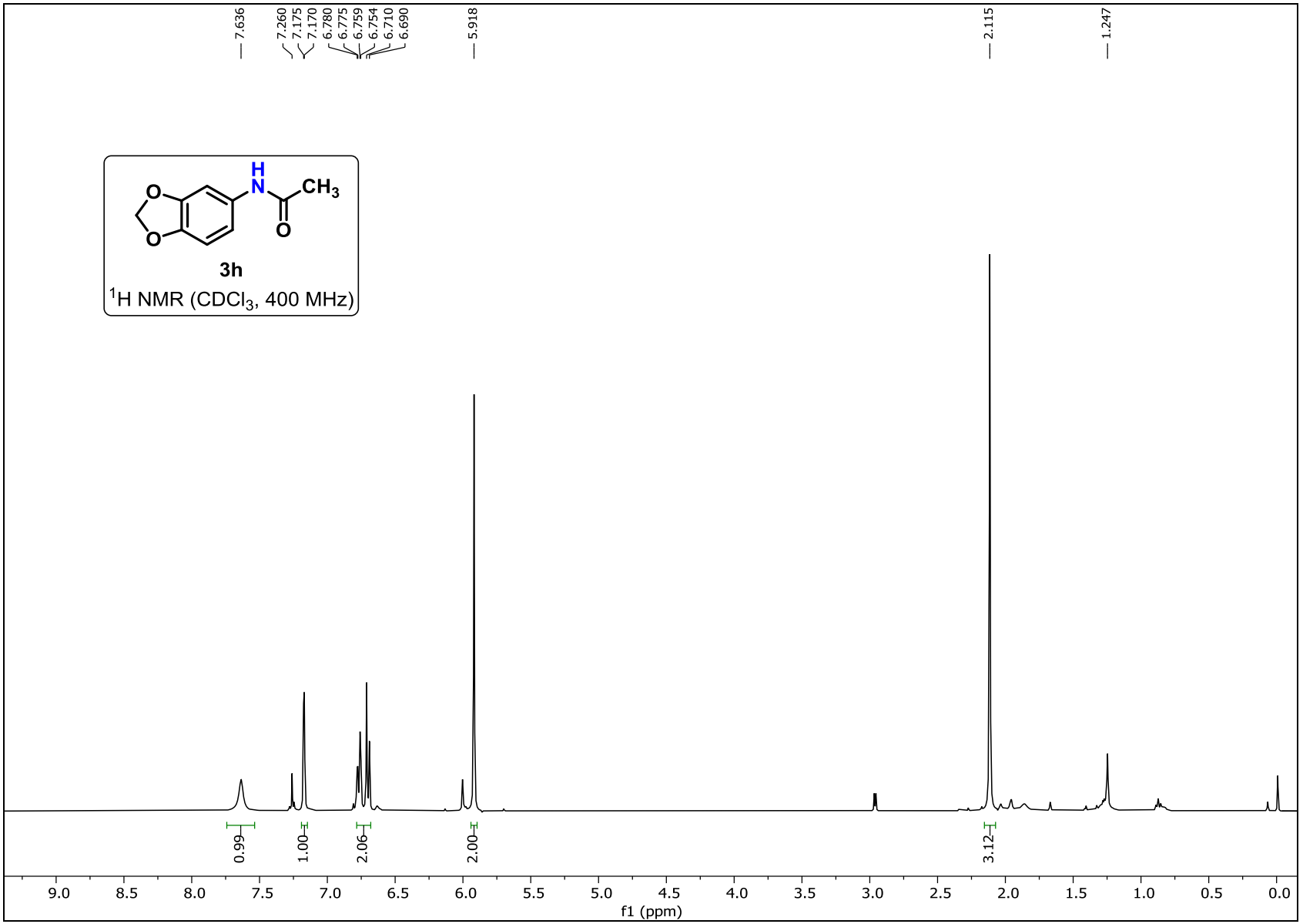
7.301
7.260
7.174
6.882
6.864
6.859
6.842
6.837
6.802
6.781
6.763
3.869
3.852
2.155
-0.005
MeO
MeO
H
N
Me
O
3g
1H NMR (CDCl3, 400 MHz)
0.97
0.83
2.09
3.01
3.01
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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7.175
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6.754
6.710
6.690
5.918
2.115
1.247
H
N
O
O
CH3
O
3h
1H NMR (CDCl3, 400 MHz)
0.99
1.00
2.06
2.00
3.12
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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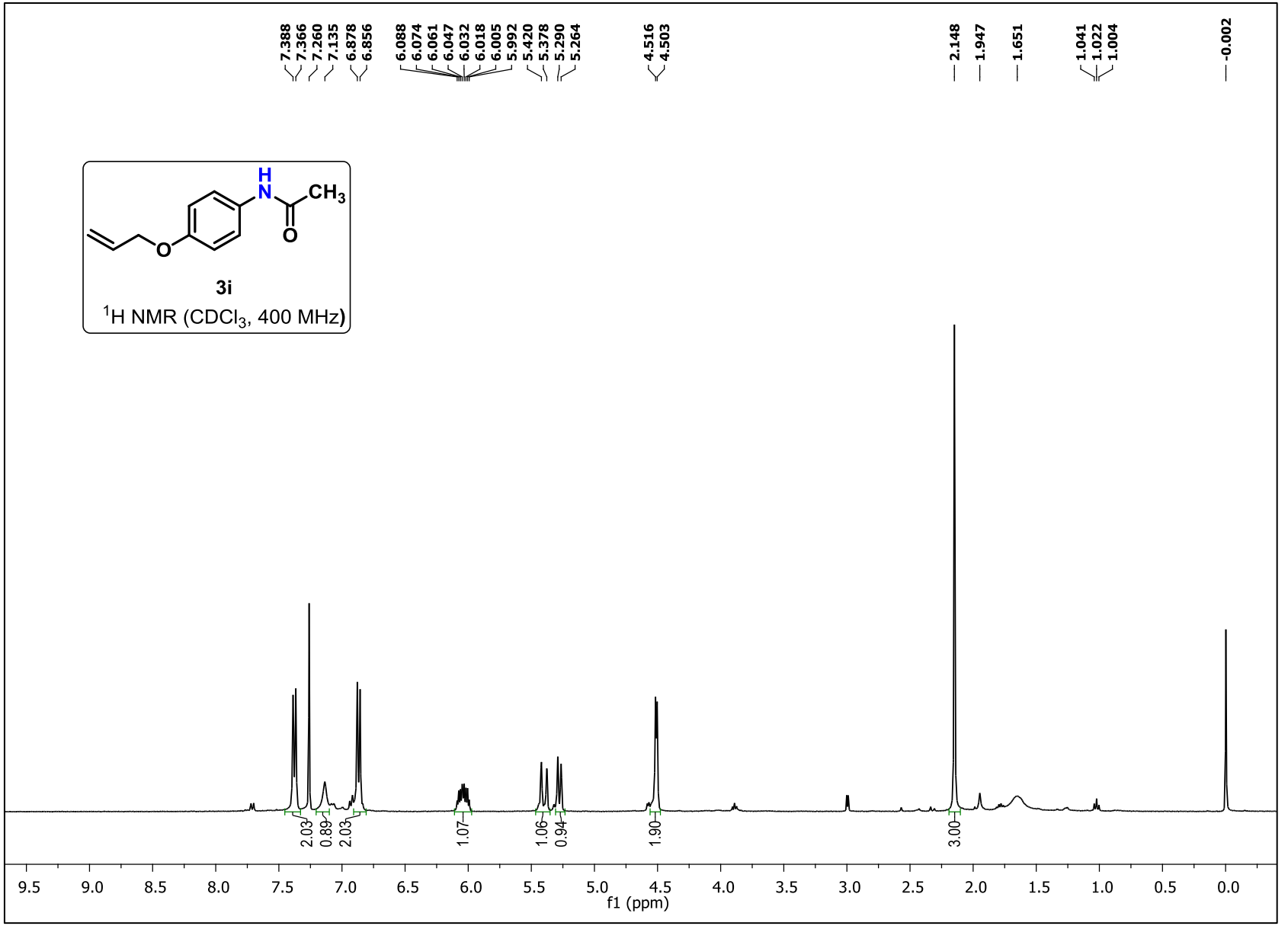
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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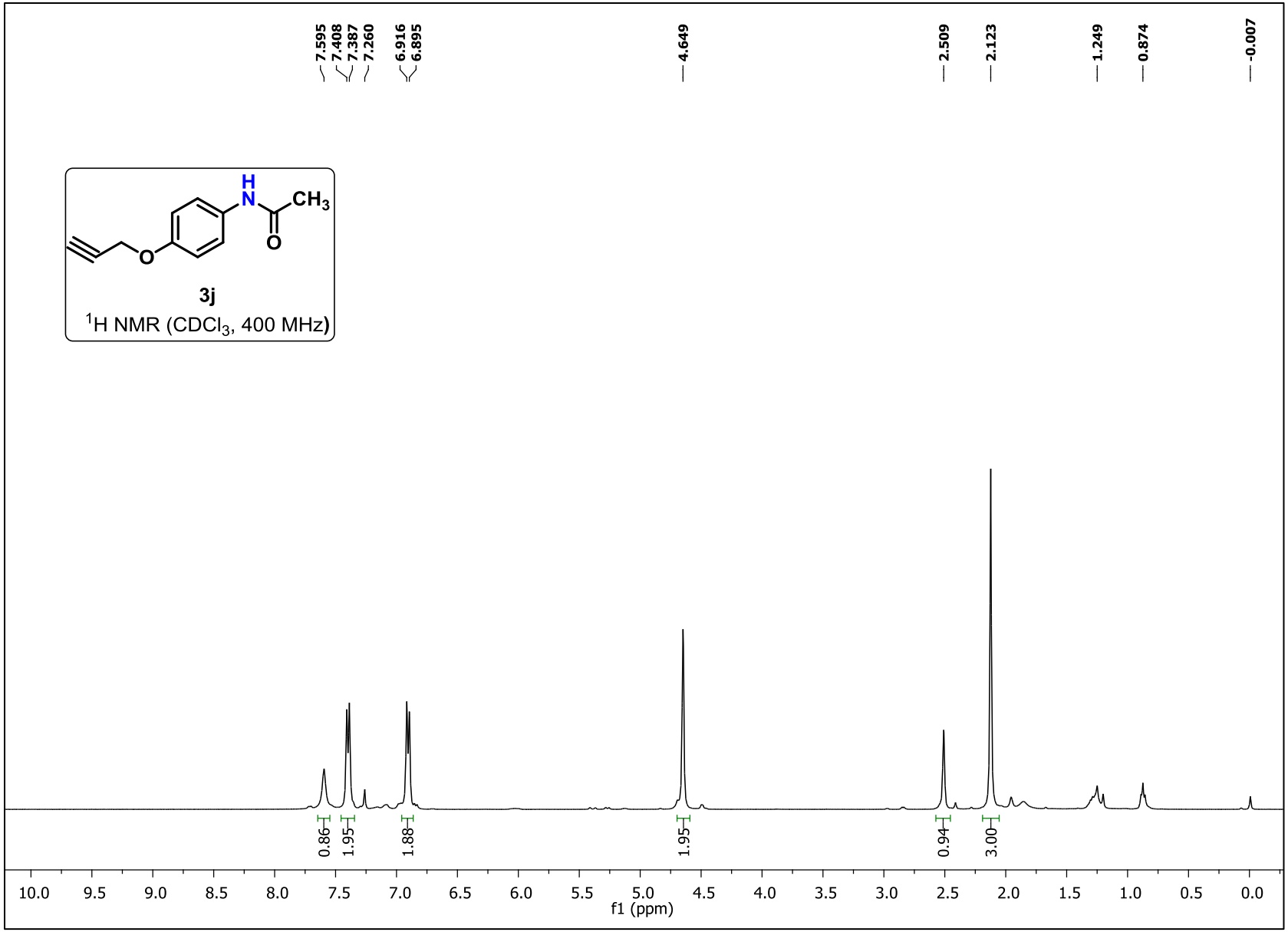
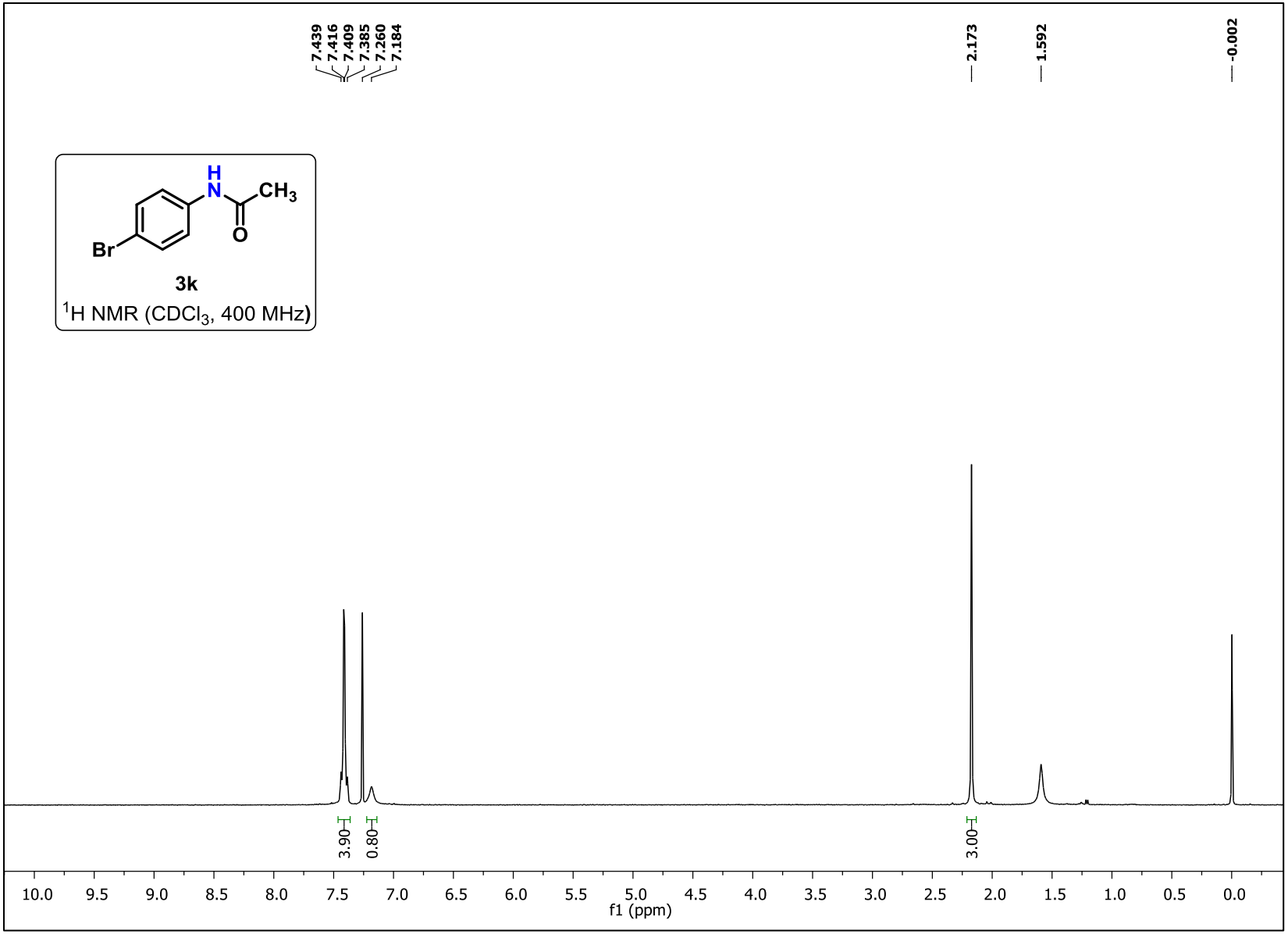
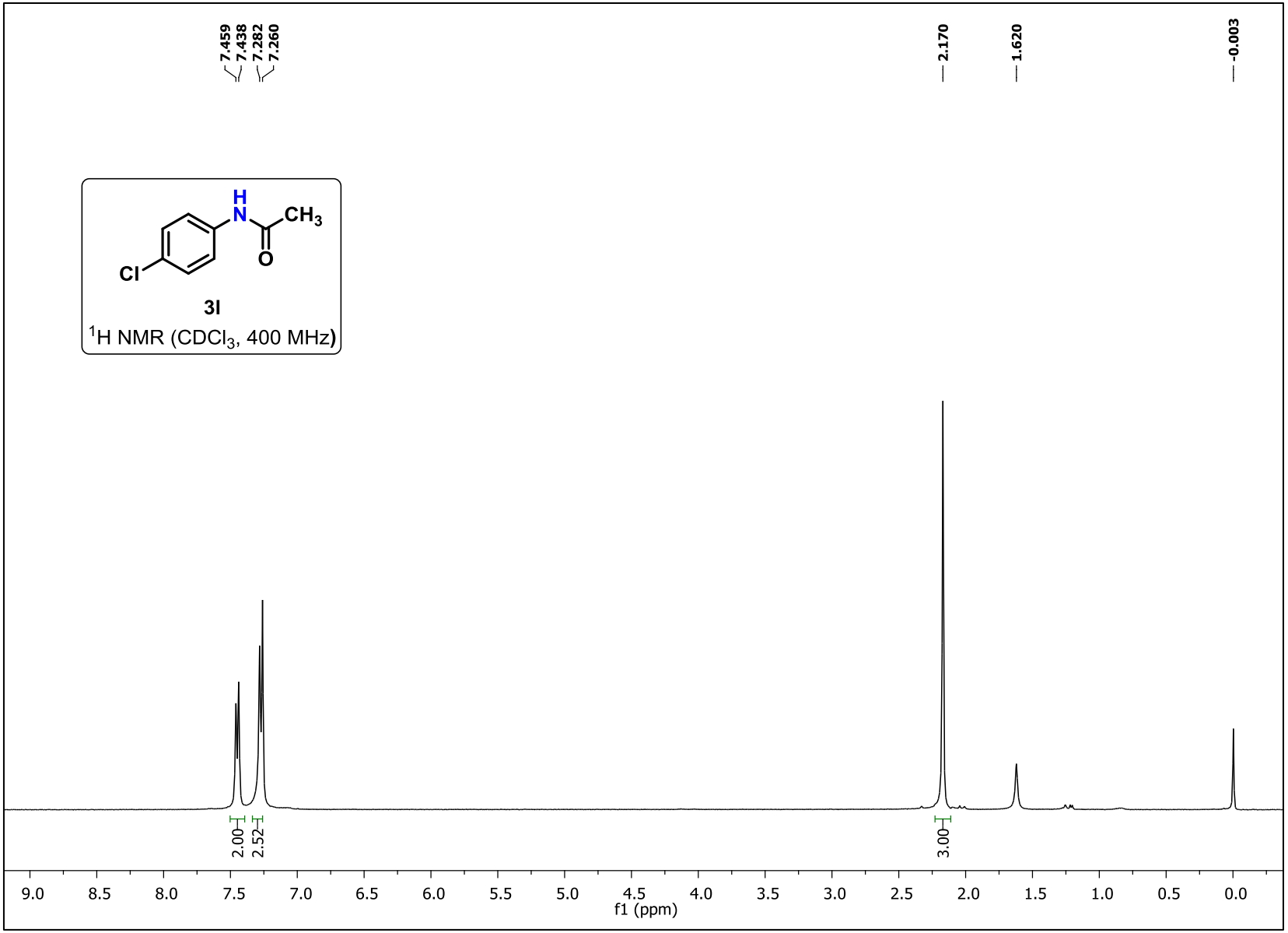
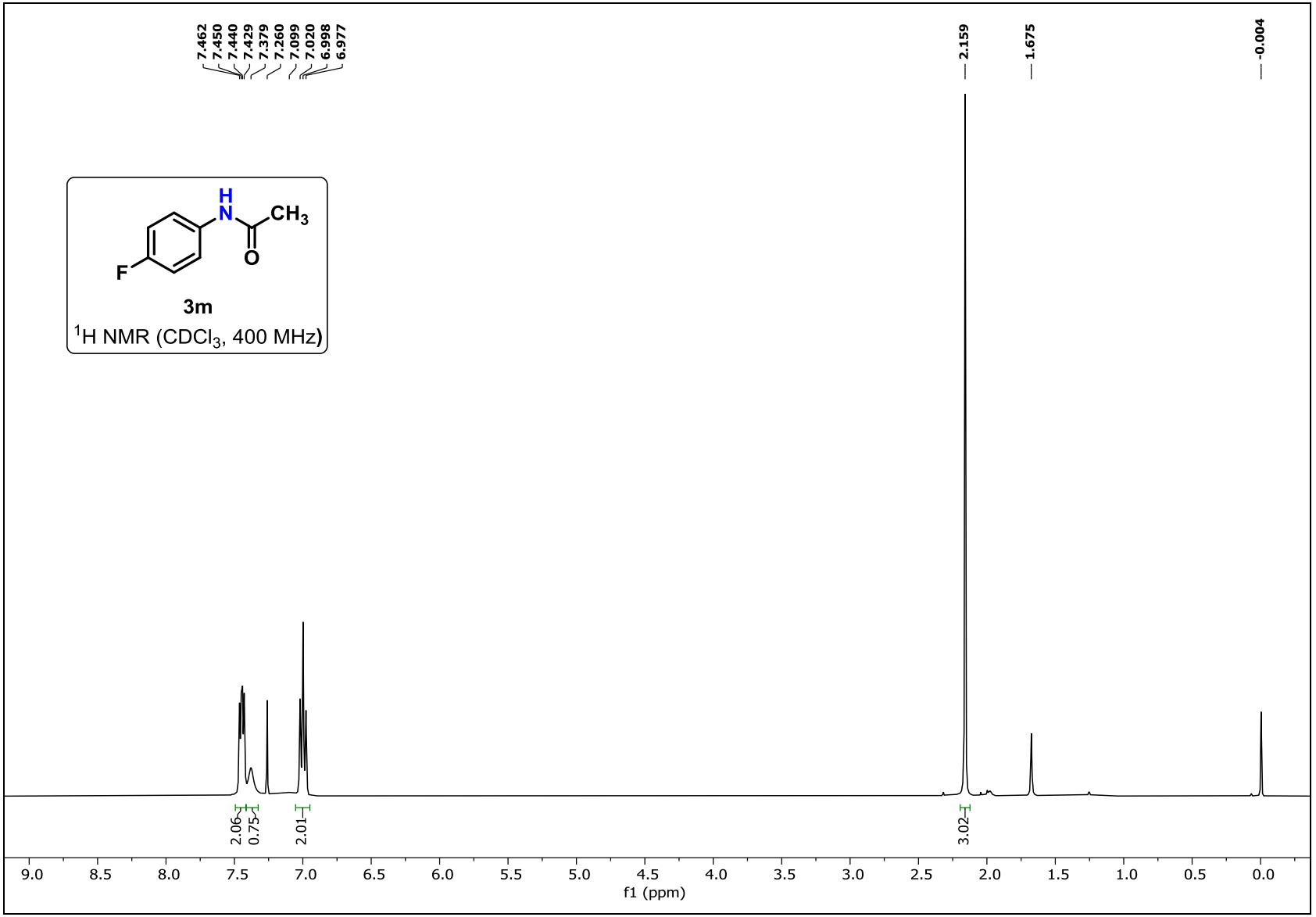
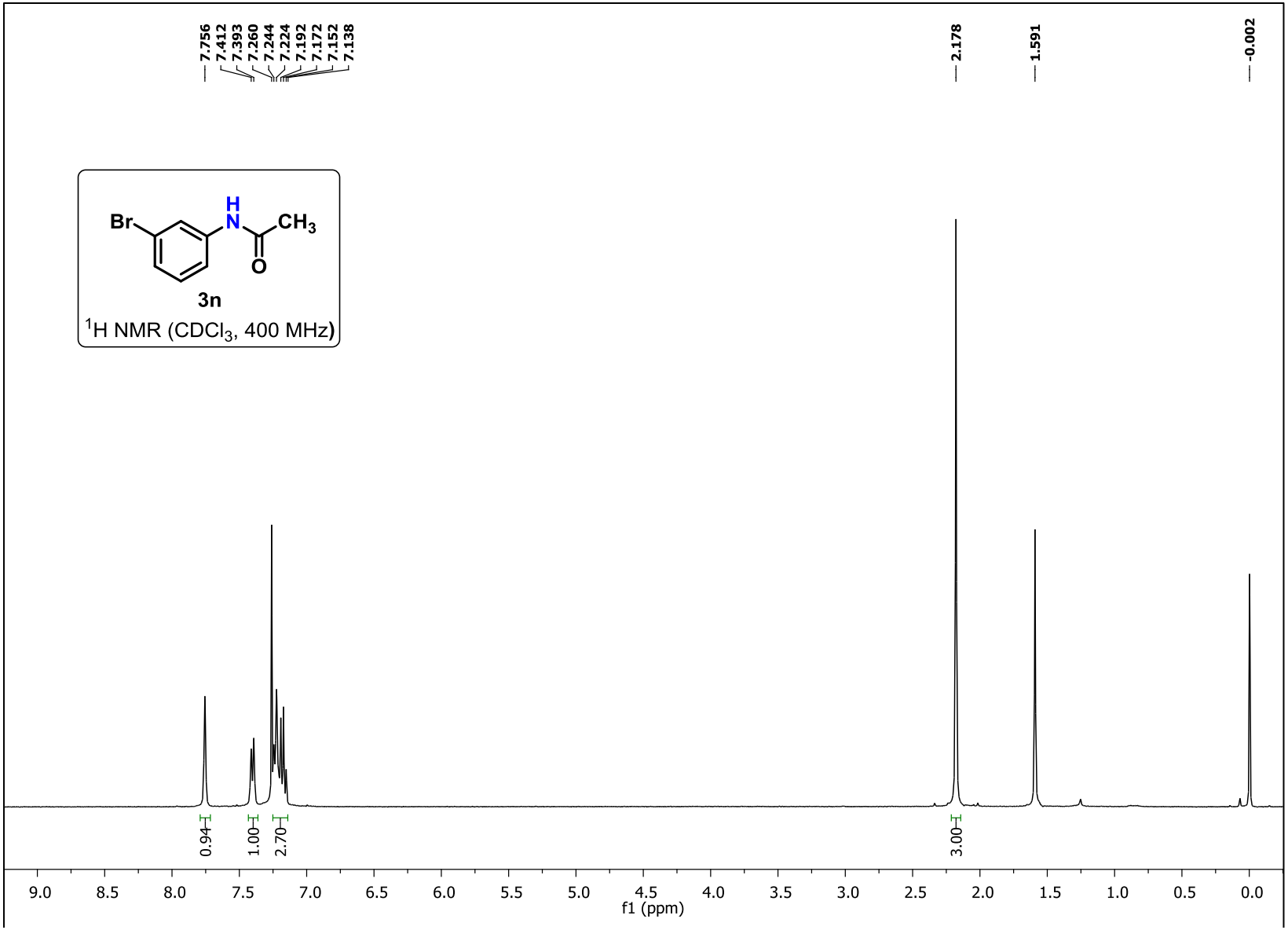
5.420
5.378
5.290
5.264
4.516
4.503
2.148
1.947
1.651
1.041
1.022
1.004
-0.002
H
N
CH3
O
O
3i
1H NMR (CDCl3, 400 MHz)
2.03
0.89
2.03
1.07
1.06
0.94
1.90
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

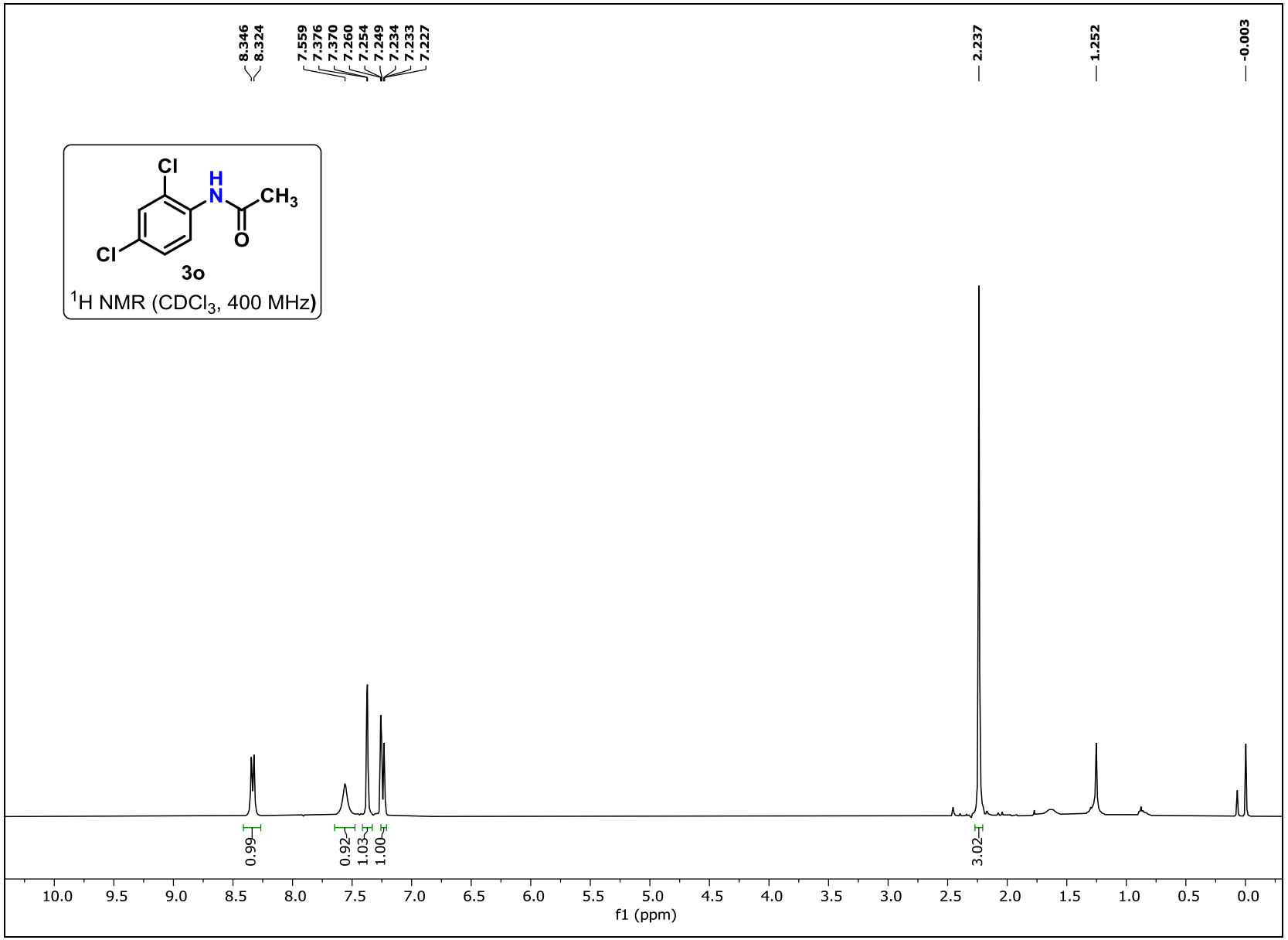

7.595
7.408
7.387
7.260
6.916
6.895
4.649
2.509
2.123
1.249
0.874
-0.007
H
N
CH3
O
O
3j
1H NMR (CDCl3, 400 MHz)
0.86
1.95
1.88
1.95
0.94
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

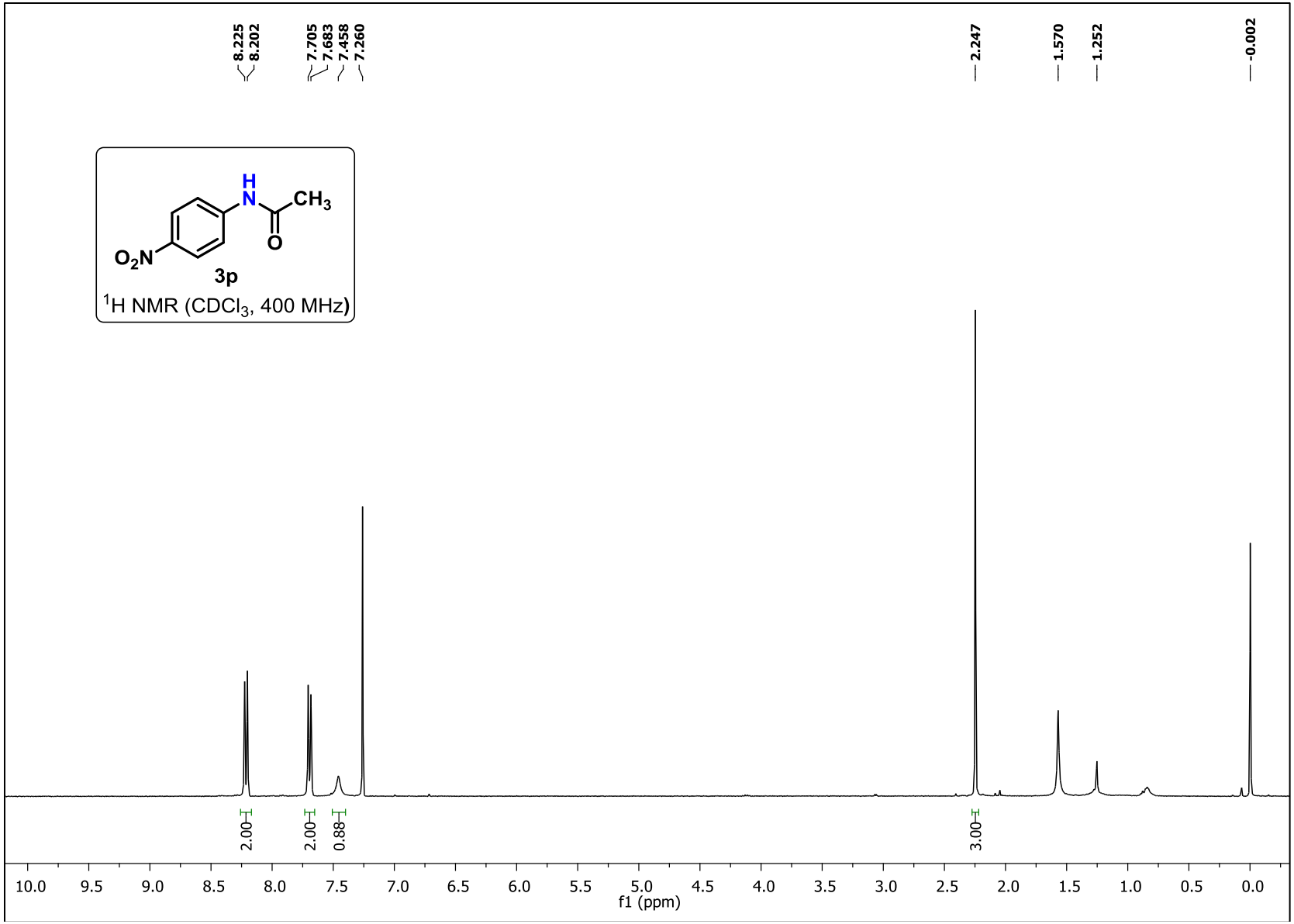

7.439
7.416
7.409
7.385
7.260
7.184
2.173
1.592
-0.002
H
N
CH3
O
Br
3k
1H NMR (CDCl3, 400 MHz)
3.90
0.80
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

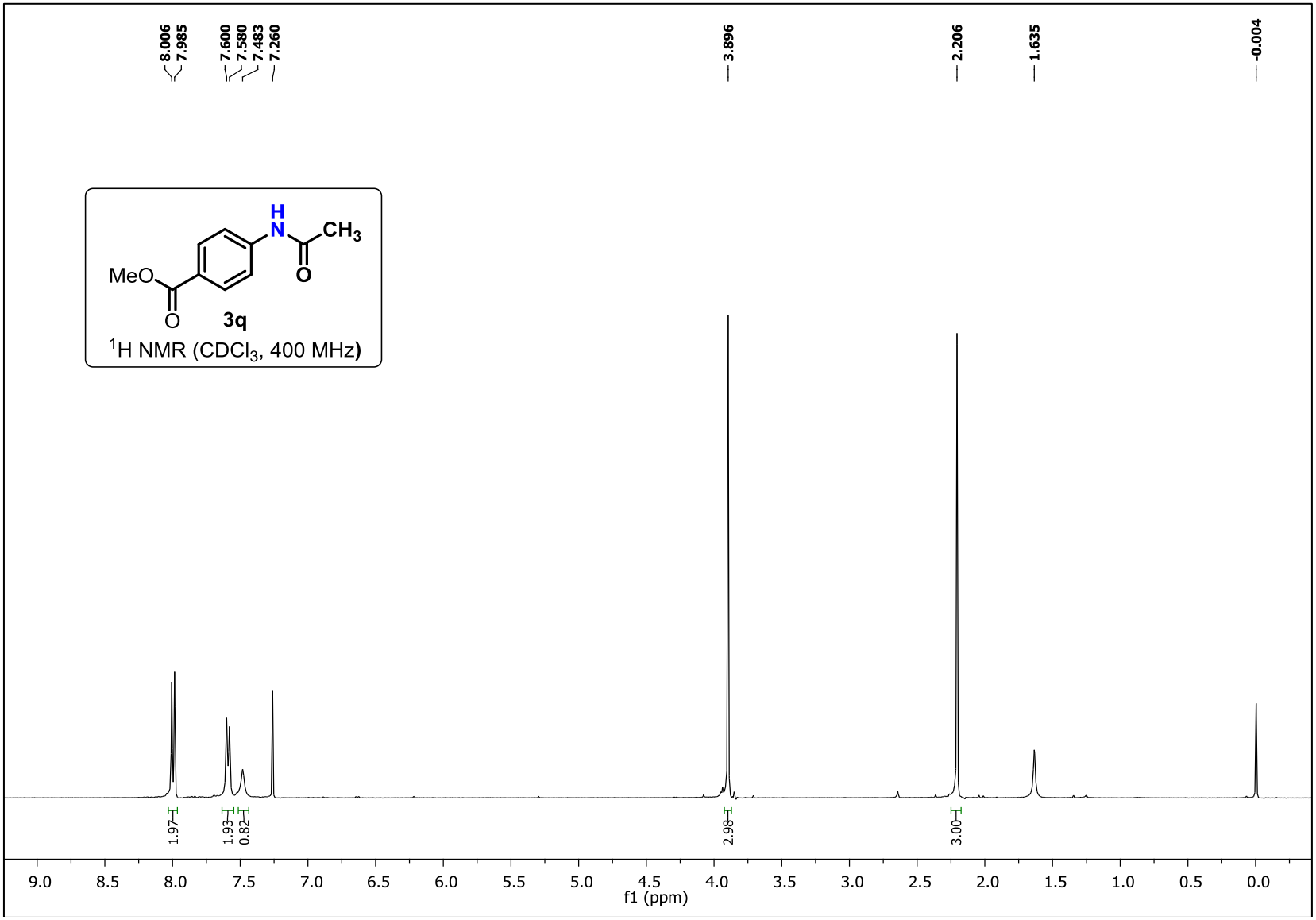

7.459
7.438
7.282
7.260
2.170
1.620
-0.003
H
N
CH3
O
Cl
3l
1H NMR (CDCl3, 400 MHz)
2.00
2.52
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

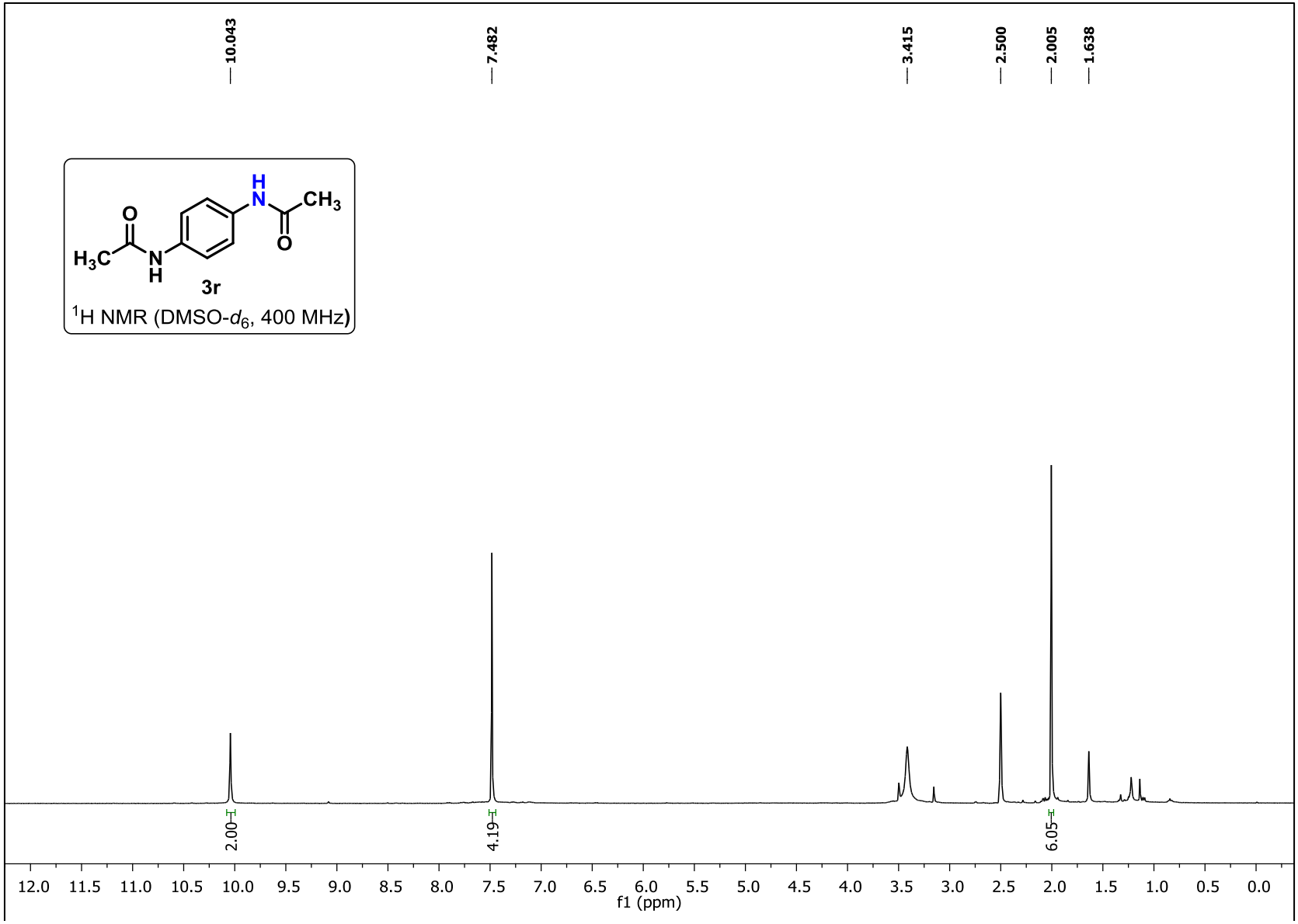

7.462
7.450
7.440
7.429
7.379
7.260
7.099
7.020
6.998
6.977
2.159
1.675
-0.004
H
N
CH3
O
F
3m
1H NMR (CDCl3, 400 MHz)
2.06
0.75
2.01
3.02
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

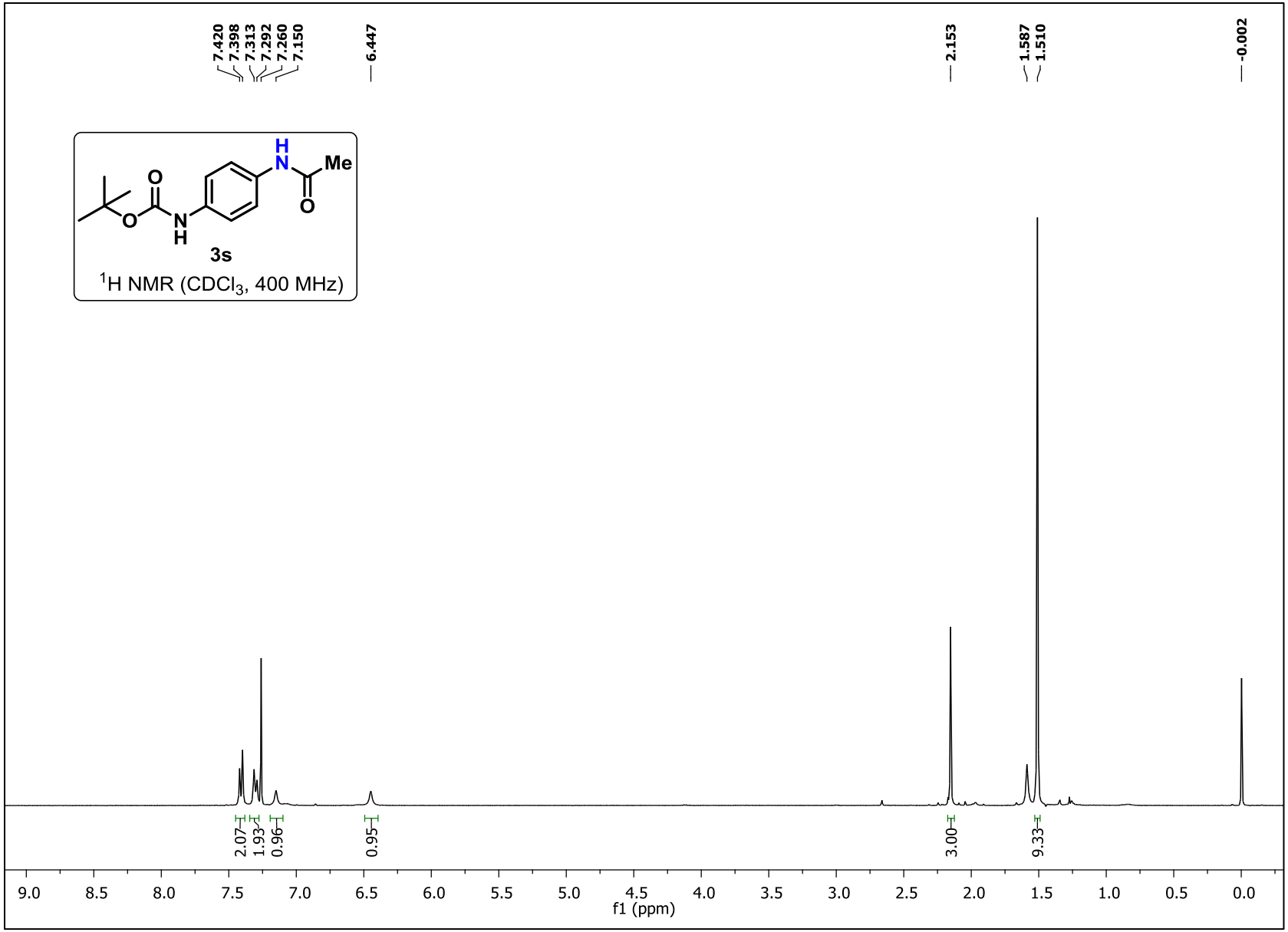

7.756
7.412
7.393
7.260
7.244
7.224
7.192
7.172
7.152
7.138
2.178
1.591
-0.002
Br
H
N
CH3
O
3n
1H NMR (CDCl3, 400 MHz)
0.94
1.00
2.70
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

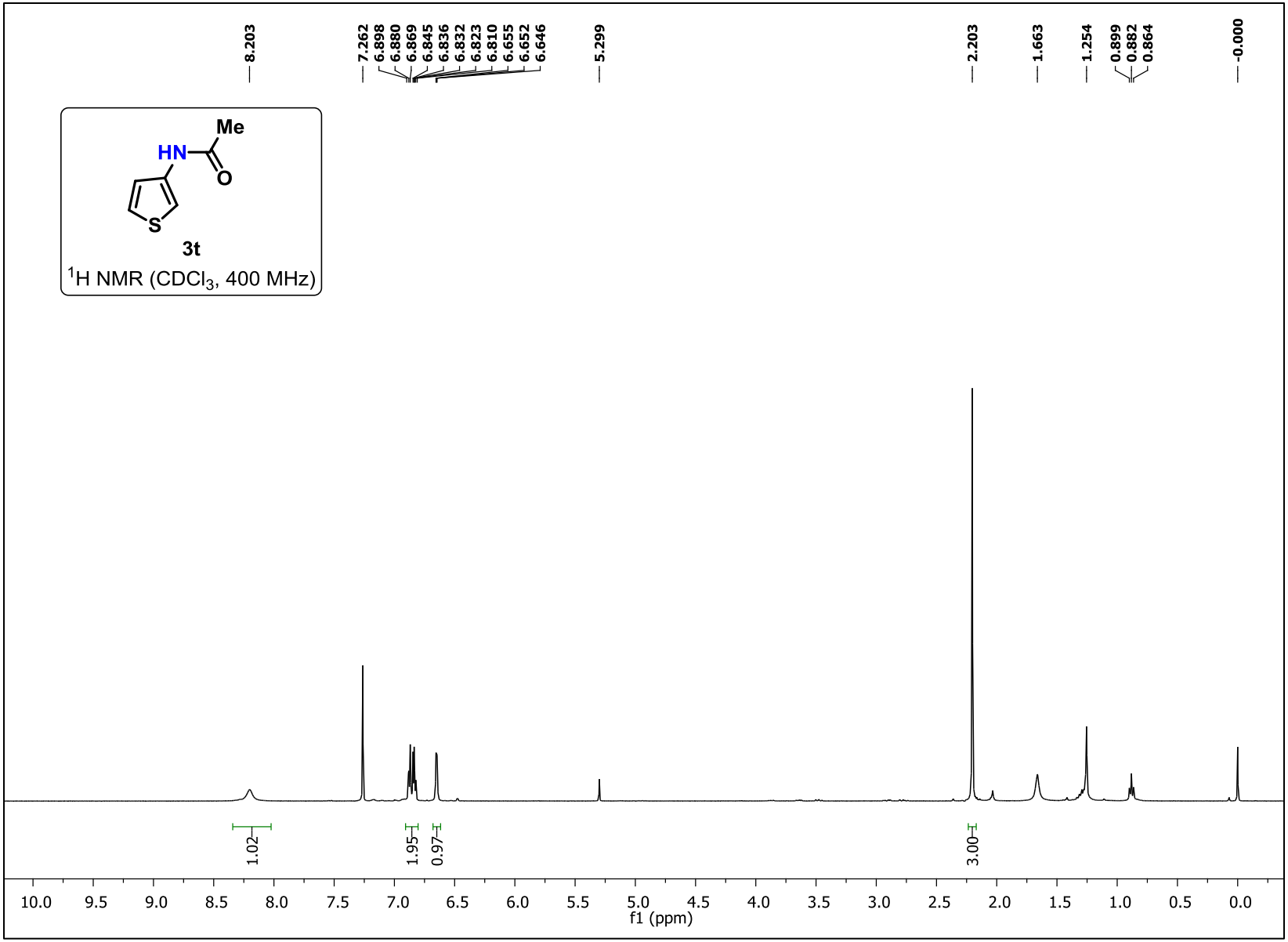

8.346
8.324
7.559
7.376
7.370
7.260
7.254
7.249
7.234
7.233
7.227
2.237
1.252
-0.003
Cl
H
N
CH3
O
Cl
3o
1H NMR (CDCl3, 400 MHz)
0.99
0.92
1.03
1.00
3.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

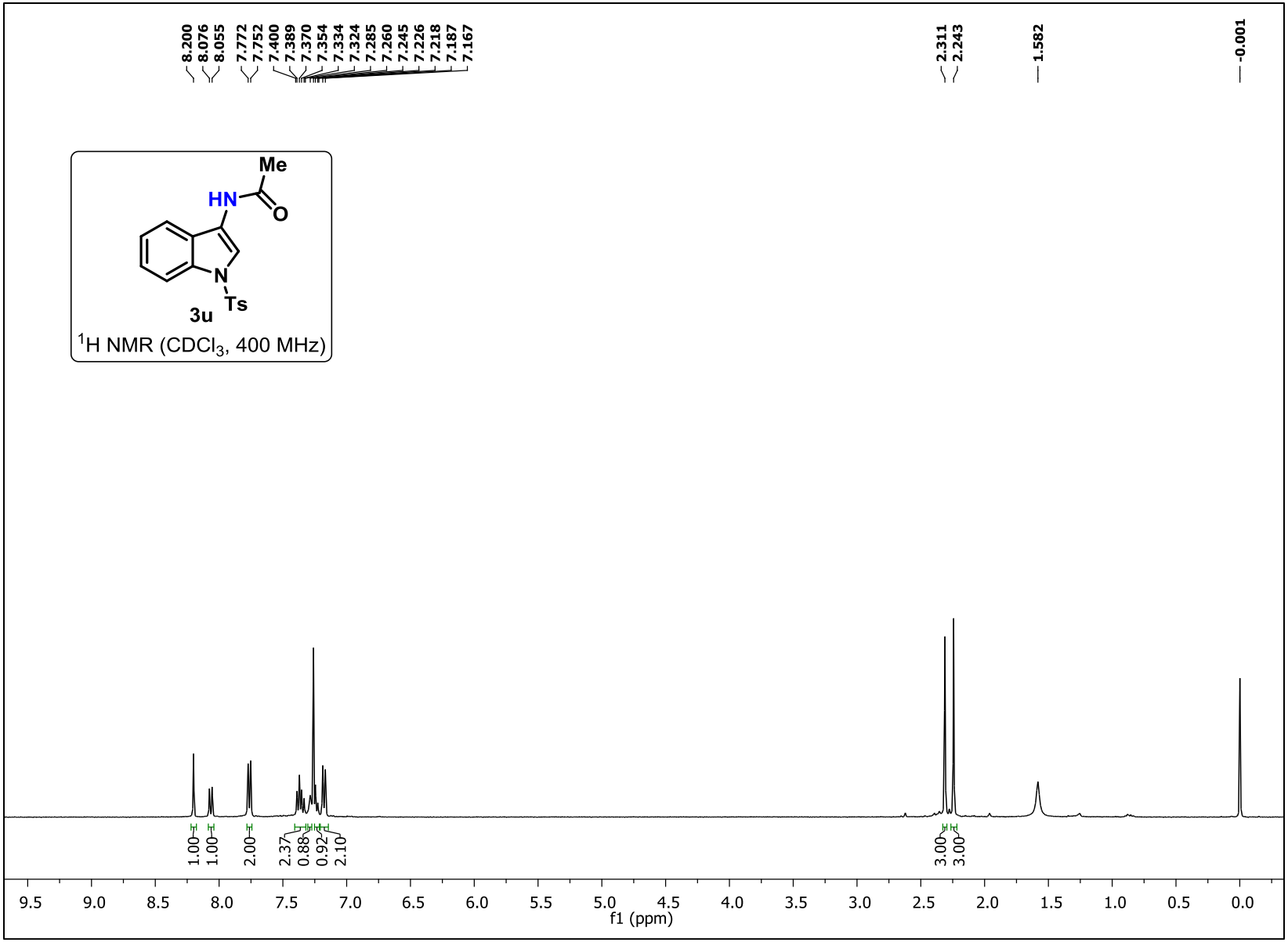

8.225
8.202
7.705
7.683
7.458
7.260
2.247
1.570
1.252
-0.002
H
N
CH3
O
O2N
3p
1H NMR (CDCl3, 400 MHz)
2.00
2.00
0.88
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

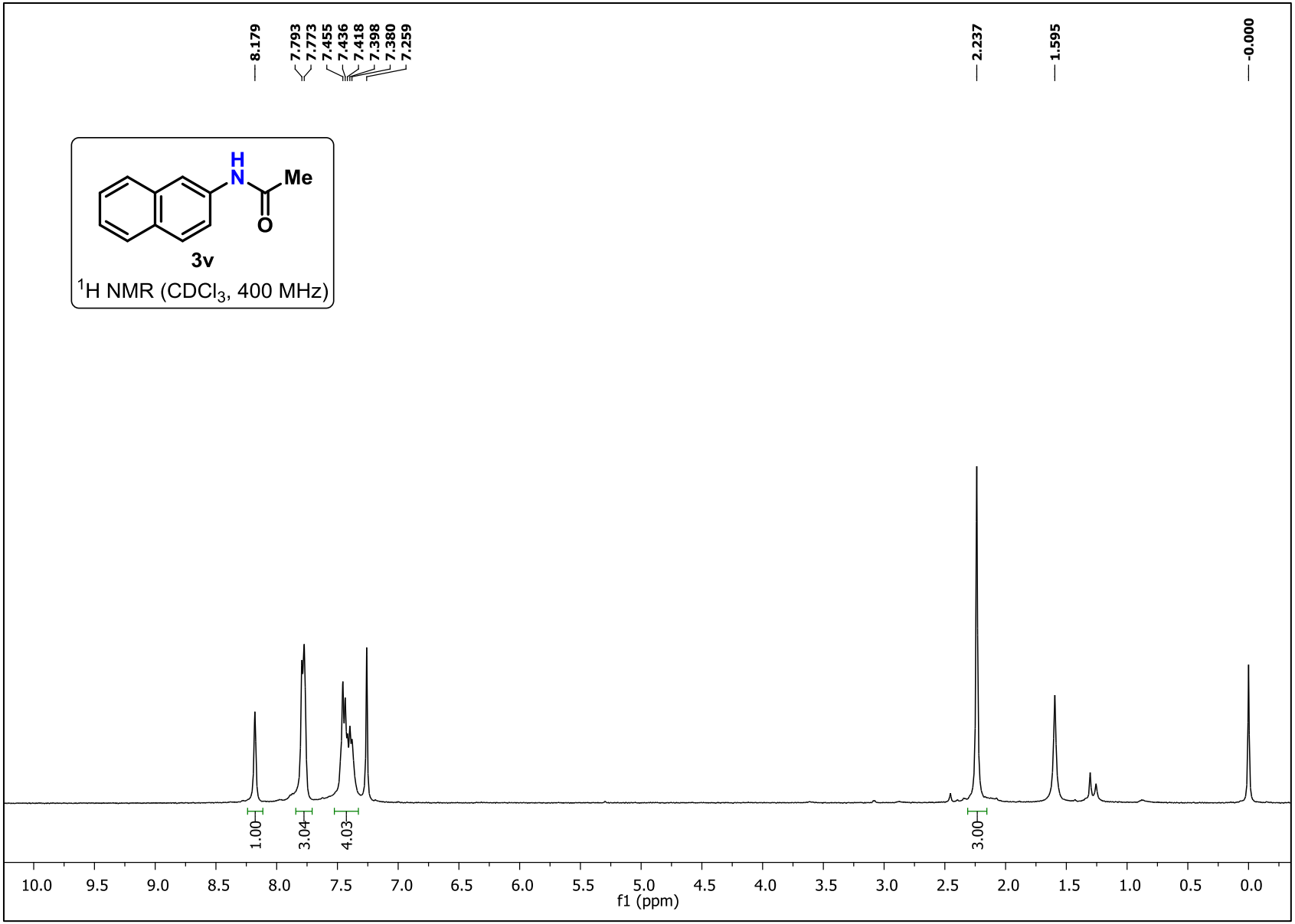

8.006
7.985
7.600
7.580
7.483
7.260
3.896
2.206
1.635
-0.004
MeO
O
O
H
N
CH3
3q
1H NMR (CDCl3, 400 MHz)
1.97
1.93
0.82
2.98
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

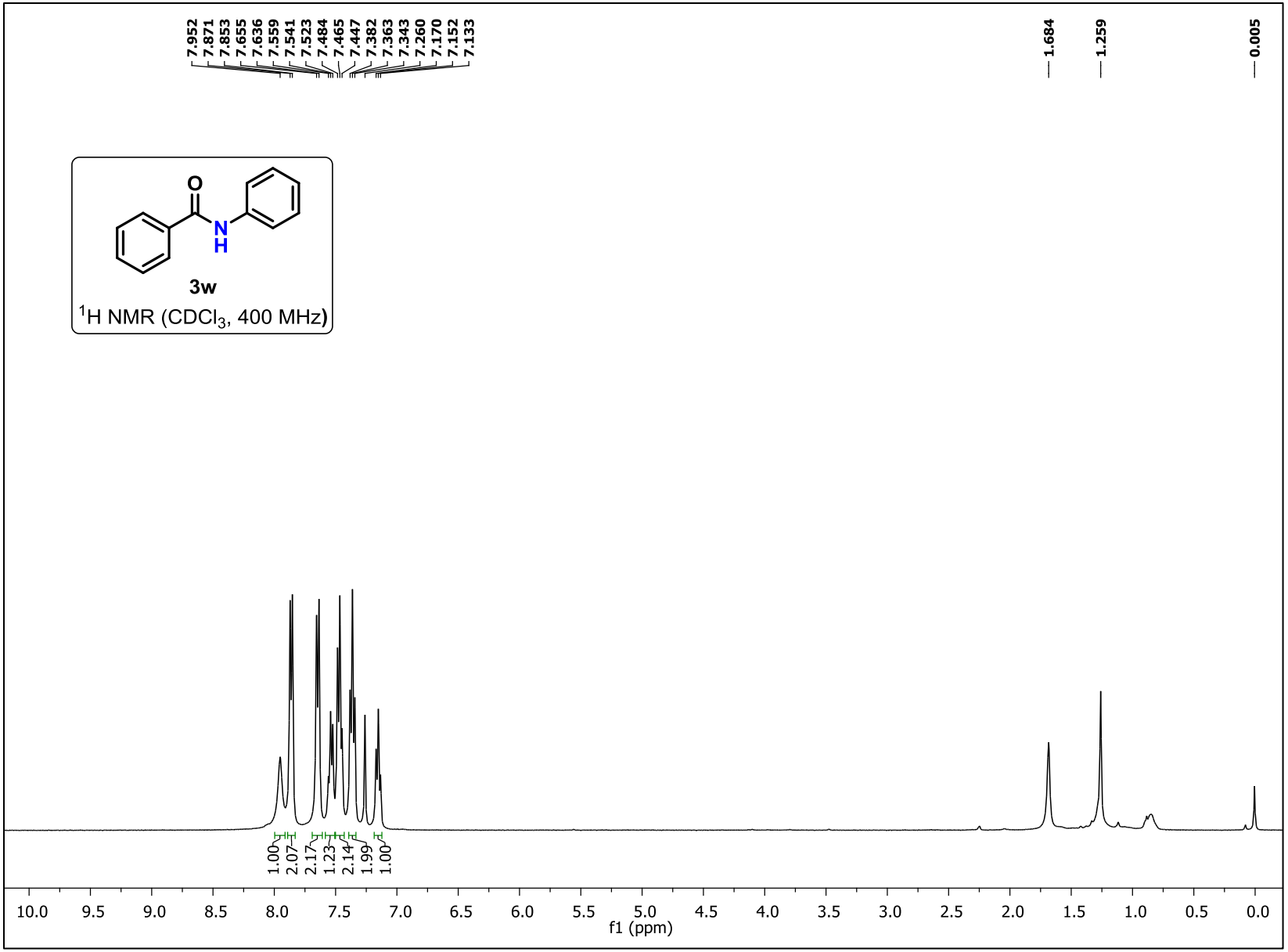

10.043
7.482
3.415
2.500
2.005
1.638
3r
1H NMR (DMSO-d6, 400 MHz)
2.00
4.19
6.05
f1 (ppm)

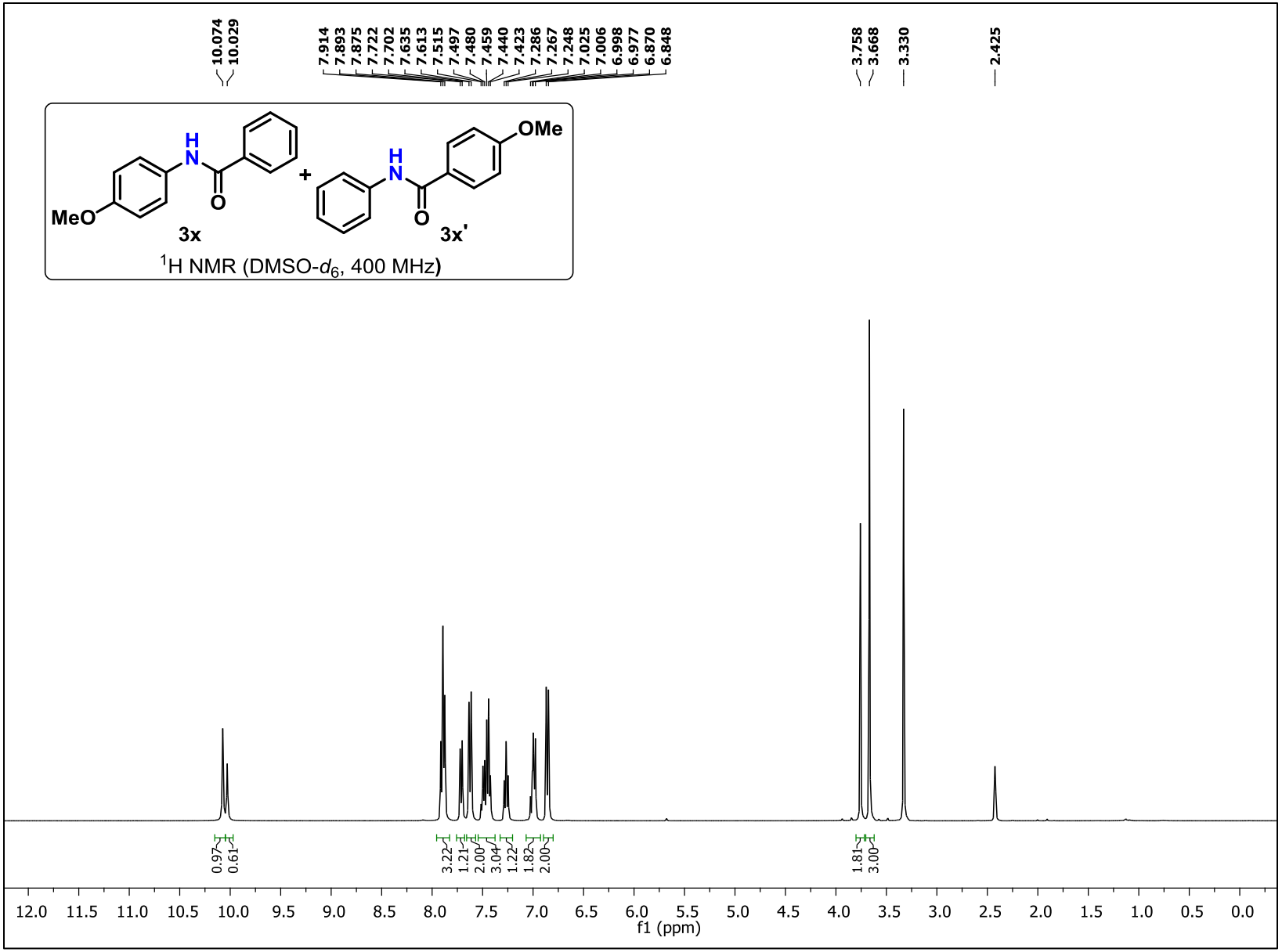

7.420
7.398
7.313
7.292
7.260
7.150
6.447
2.153
1.587
1.510
-0.002
3s
1H NMR (CDCl3, 400 MHz)
2.07
1.93
0.96
0.95
3.00
9.33
f1 (ppm)

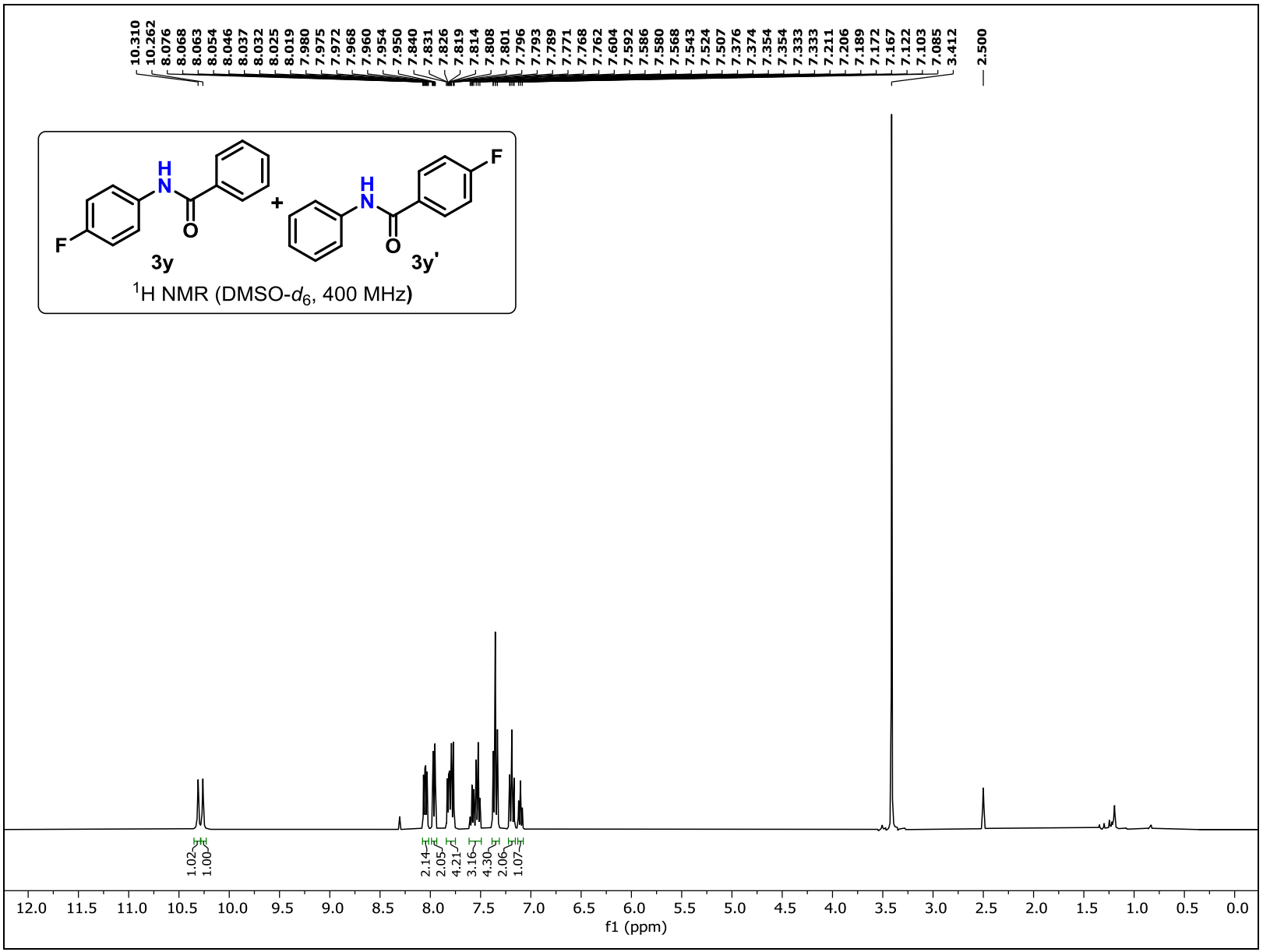

8.203
7.262
6.898
6.880
6.869
6.845
6.836
6.832
6.823
6.810
6.655
6.652
6.646
5.299
2.203
1.663
1.254
0.899
0.882
0.864
-0.000
Me
HN
O
S
3t
1H NMR (CDCl3, 400 MHz)
1.02
1.95
0.97
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

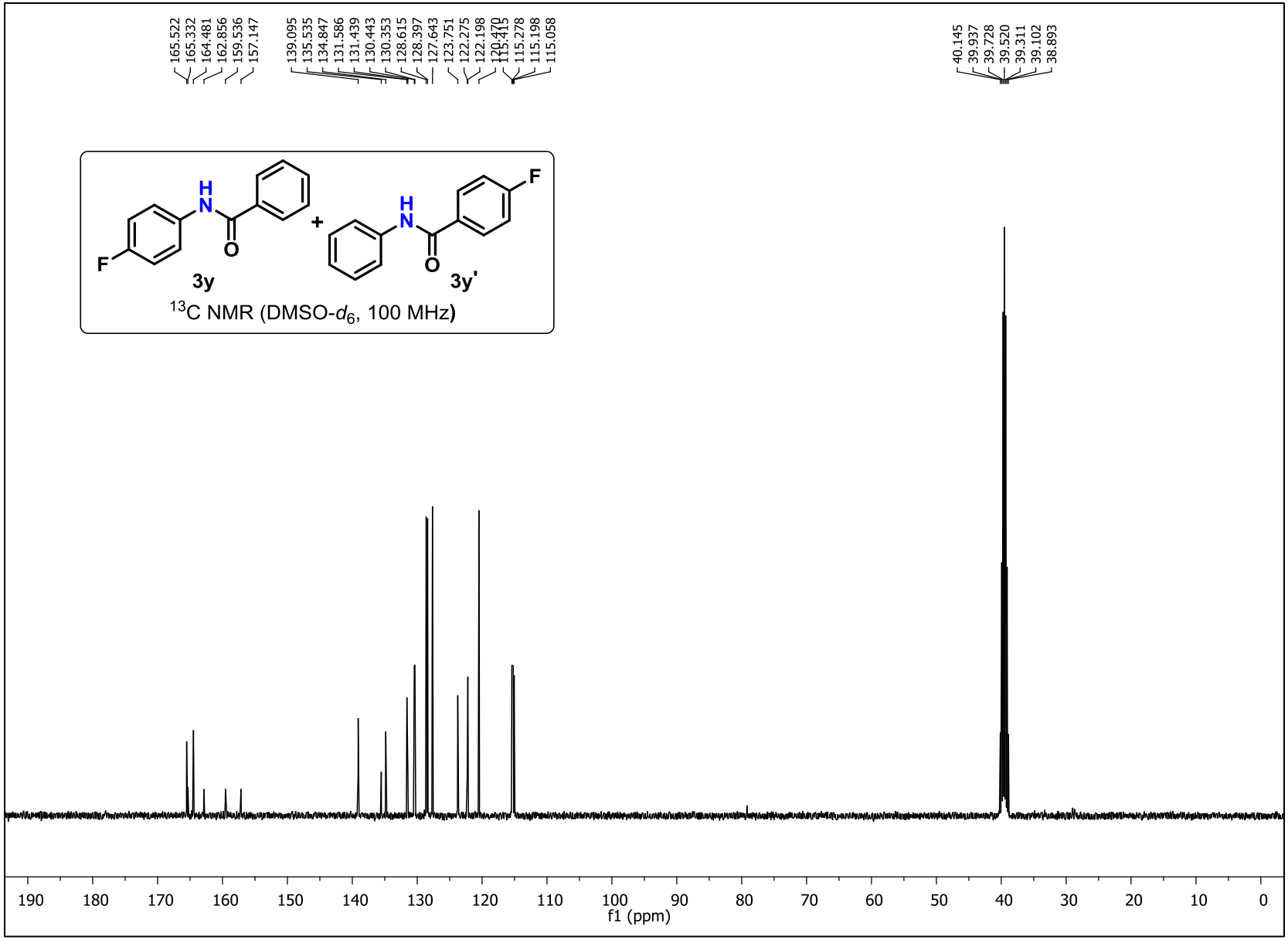

8.200
8.076
8.055
7.772
7.752
7.400
7.389
7.370
7.354
7.334
7.324
7.285
7.260
7.245
7.226
7.218
7.187
7.167
2.311
2.243
1.582
-0.001
Me
HN
O
N
Ts
3u
1H NMR (CDCl3, 400 MHz)
1.00
1.00
2.00
2.37
0.88
0.92
2.10
3.00
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

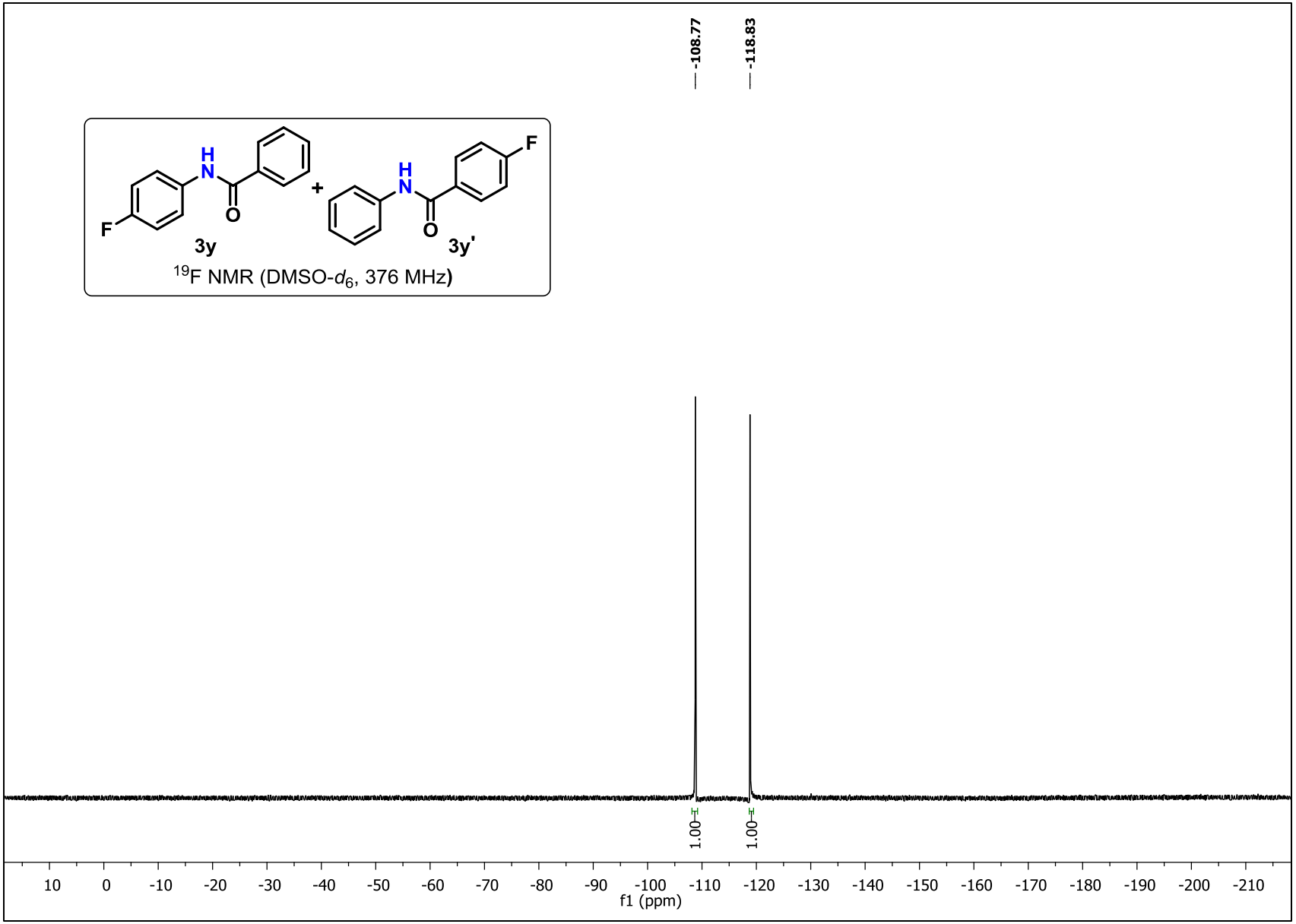

8.179
7.793
7.773
7.455
7.436
7.418
7.398
7.380
7.259
2.237
1.595
-0.000
H
N
Me
O
3v
1H NMR (CDCl3, 400 MHz)
1.00
3.04
4.03
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

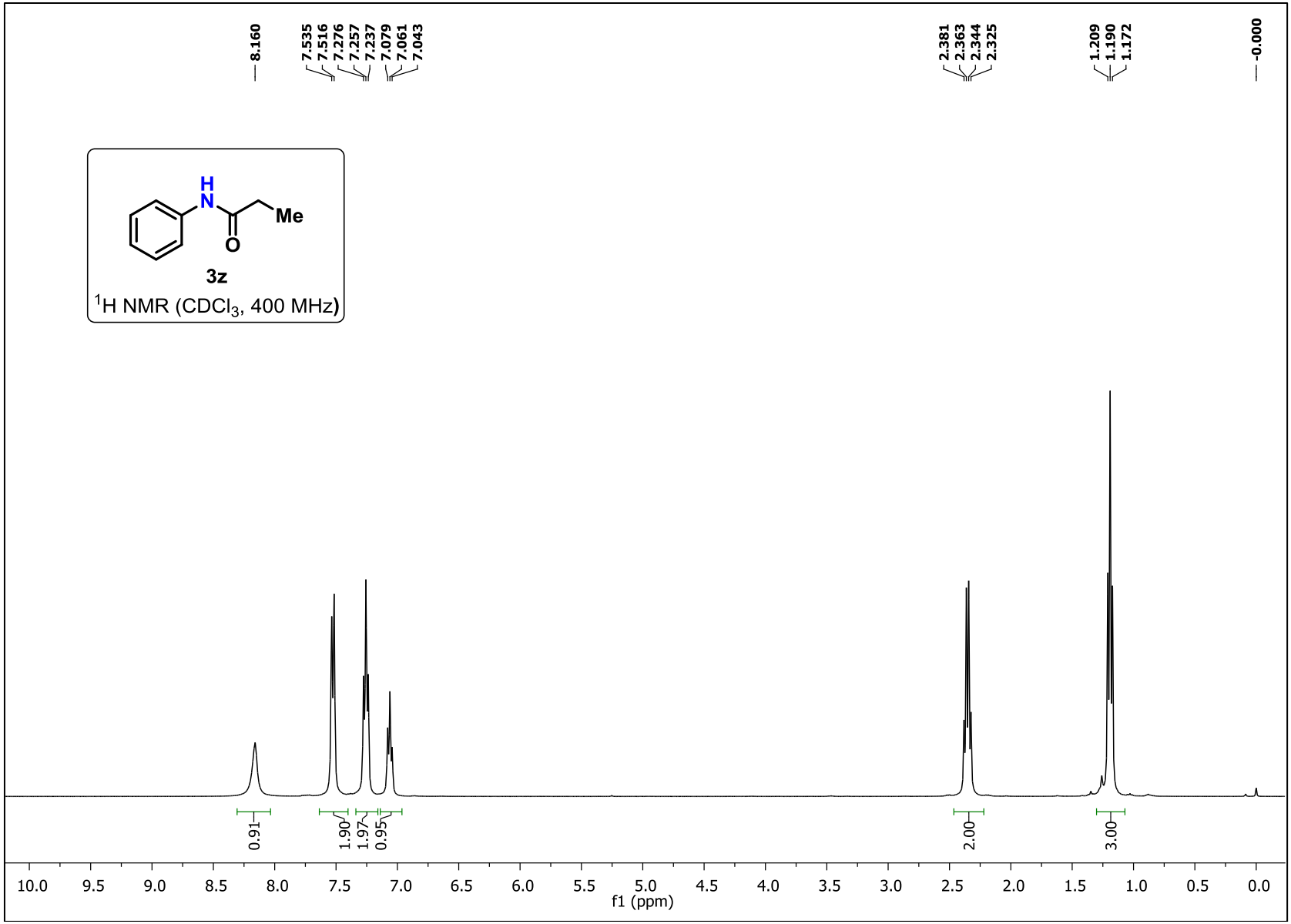

7.952
7.871
7.853
7.655
7.636
7.559
7.541
7.523
7.484
7.465
7.447
7.382
7.363
7.343
7.260
7.170
7.152
7.133
1.684
1.259
0.005
O
N
H
3w
1H NMR (CDCl3, 400 MHz)
1.00
2.07
2.17
1.23
2.14
1.99
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

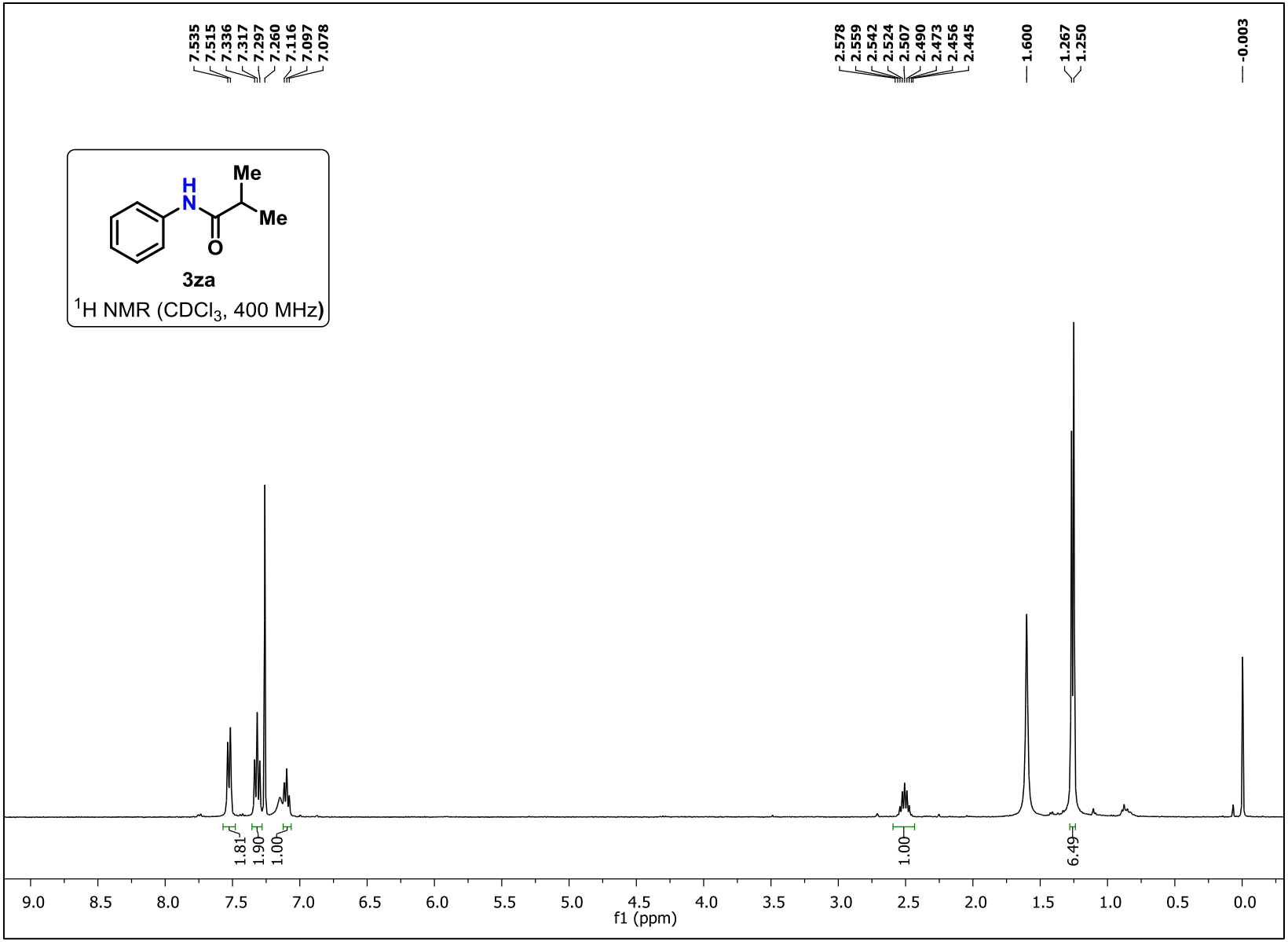

10.074
10.029
7.914
7.893
7.875
7.722
7.702
7.635
7.613
7.515
7.497
7.480
7.459
7.440
7.423
7.286
7.267
7.248
7.025
7.006
6.998
6.977
6.870
6.848
3.758
3.668
3.330
2.425
MeO
3x
OMe
3x'
$^1$H NMR (DMSO-$d_6$, 400 MHz)
0.97
0.61
3.22
1.21
2.00
3.04
1.22
1.82
2.00
1.81
3.00
f1 (ppm)

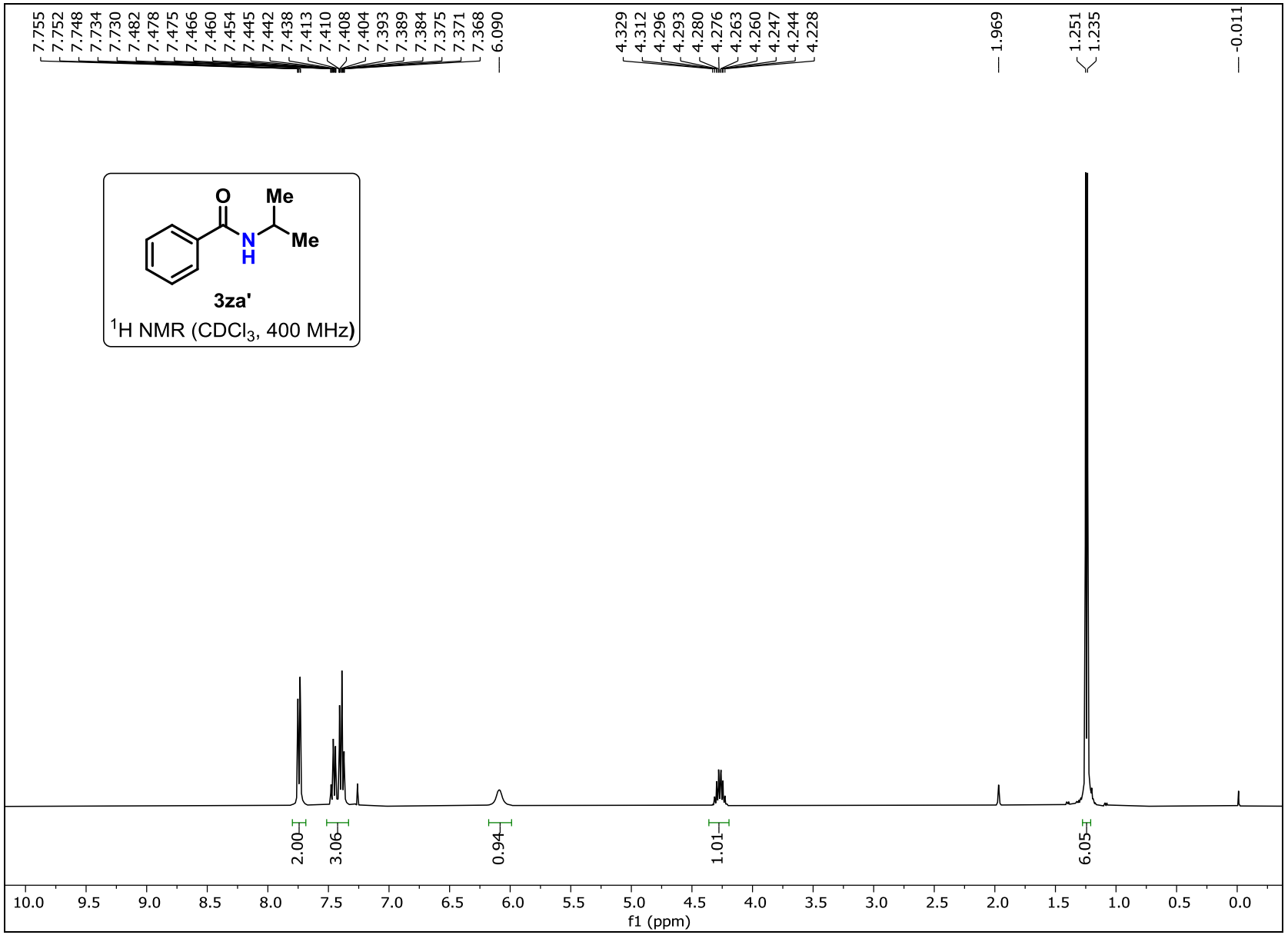

10.310
10.262
8.076
8.068
8.063
8.054
8.046
8.037
8.032
8.025
8.019
7.980
7.975
7.972
7.968
7.960
7.954
7.950
7.840
7.831
7.826
7.819
7.814
7.808
7.801
7.796
7.793
7.789
7.771
7.768
7.762
7.604
7.592
7.586
7.580
7.568
7.543
7.524
7.507
7.376
7.374
7.354
7.354
7.333
7.333
7.211
7.206
7.189
7.172
7.167
7.122
7.103
7.085
3.412
2.500
H
N
O
F
3y
+
H
N
O
F
3y'
1H NMR (DMSO-d6, 400 MHz)
1.02
1.00
2.14
2.05
4.21
3.16
4.30
2.06
1.07
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

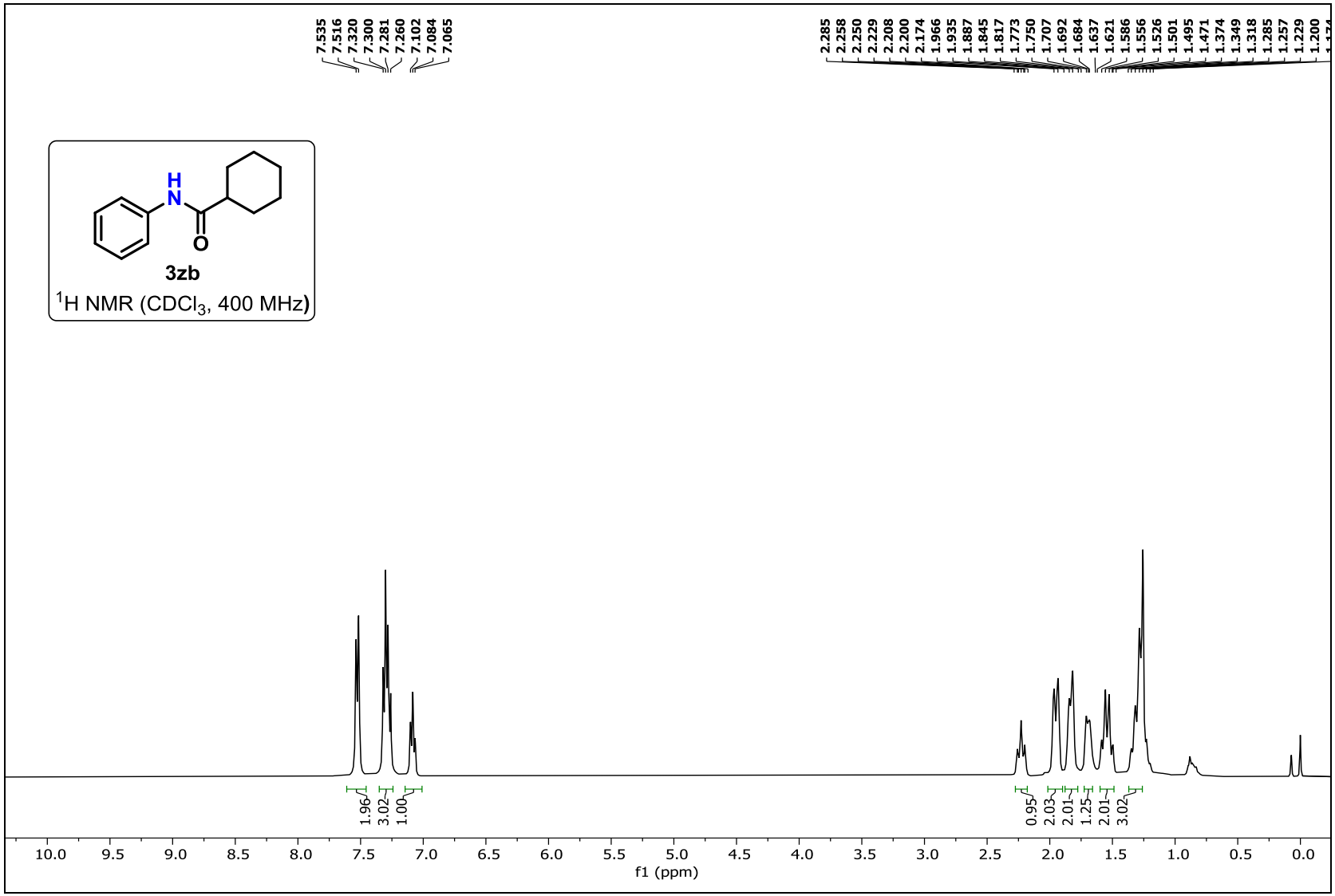

165.522
165.332
164.481
162.856
159.536
157.147
139.095
135.535
134.847
131.586
131.439
130.443
130.353
128.615
128.397
127.643
123.751
122.275
122.198
120.470
115.411
115.278
115.198
115.058
40.145
39.937
39.728
39.520
39.311
39.102
38.893
H
N
O
F
3y
+
H
N
O
F
3y'
13C NMR (DMSO-d6, 100 MHz)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

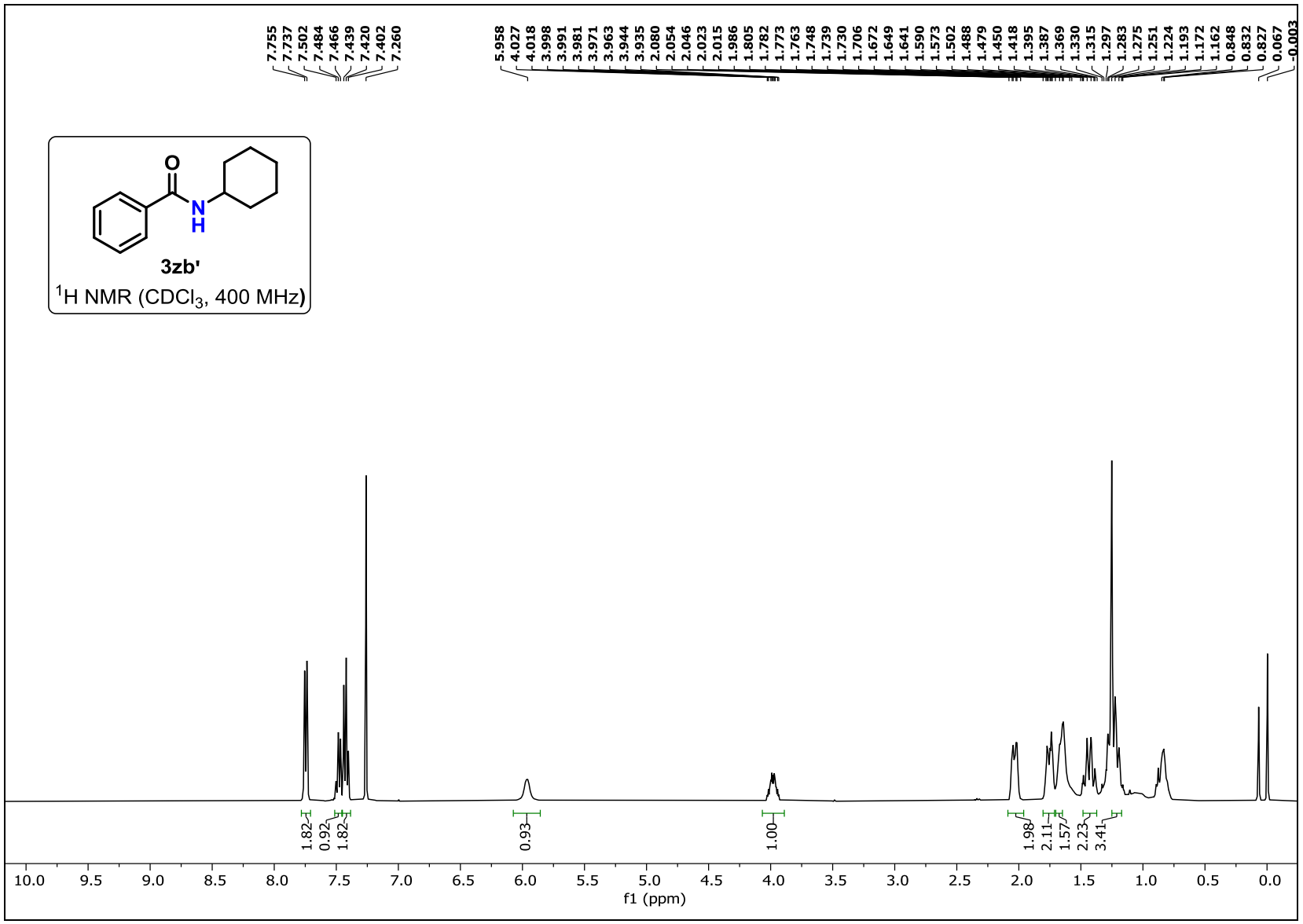

-108.77
-118.83
3y
3y'
$^{19}$F NMR (DMSO-$d_6$, 376 MHz)
1.00
1.00
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

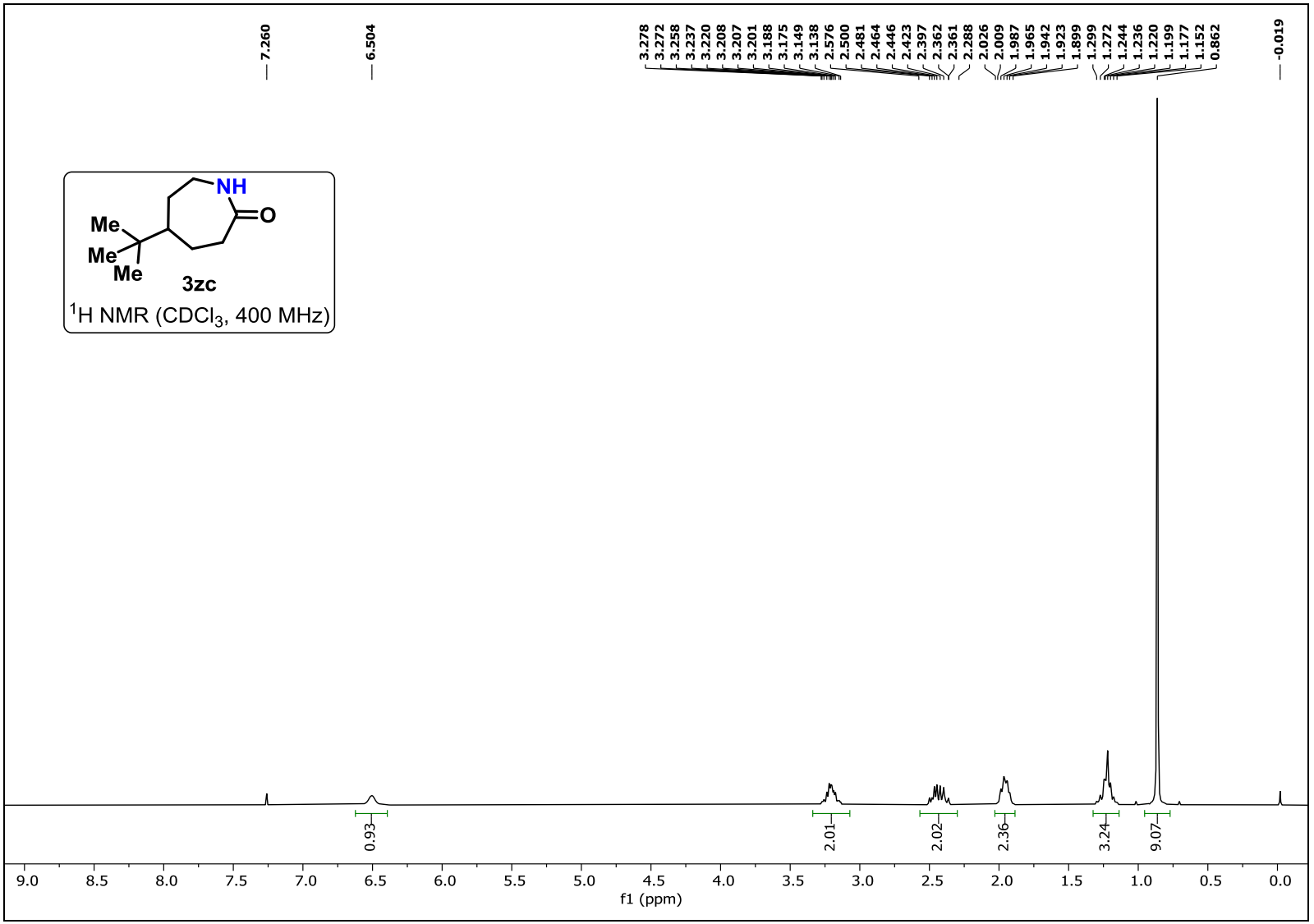

8.160
7.535
7.516
7.276
7.257
7.237
7.079
7.061
7.043
2.381
2.363
2.344
2.325
1.209
1.190
1.172
-0.000
Me
3z
1H NMR (CDCl3, 400 MHz)
0.91
1.90
1.97
0.95
2.00
3.00
f1 (ppm)

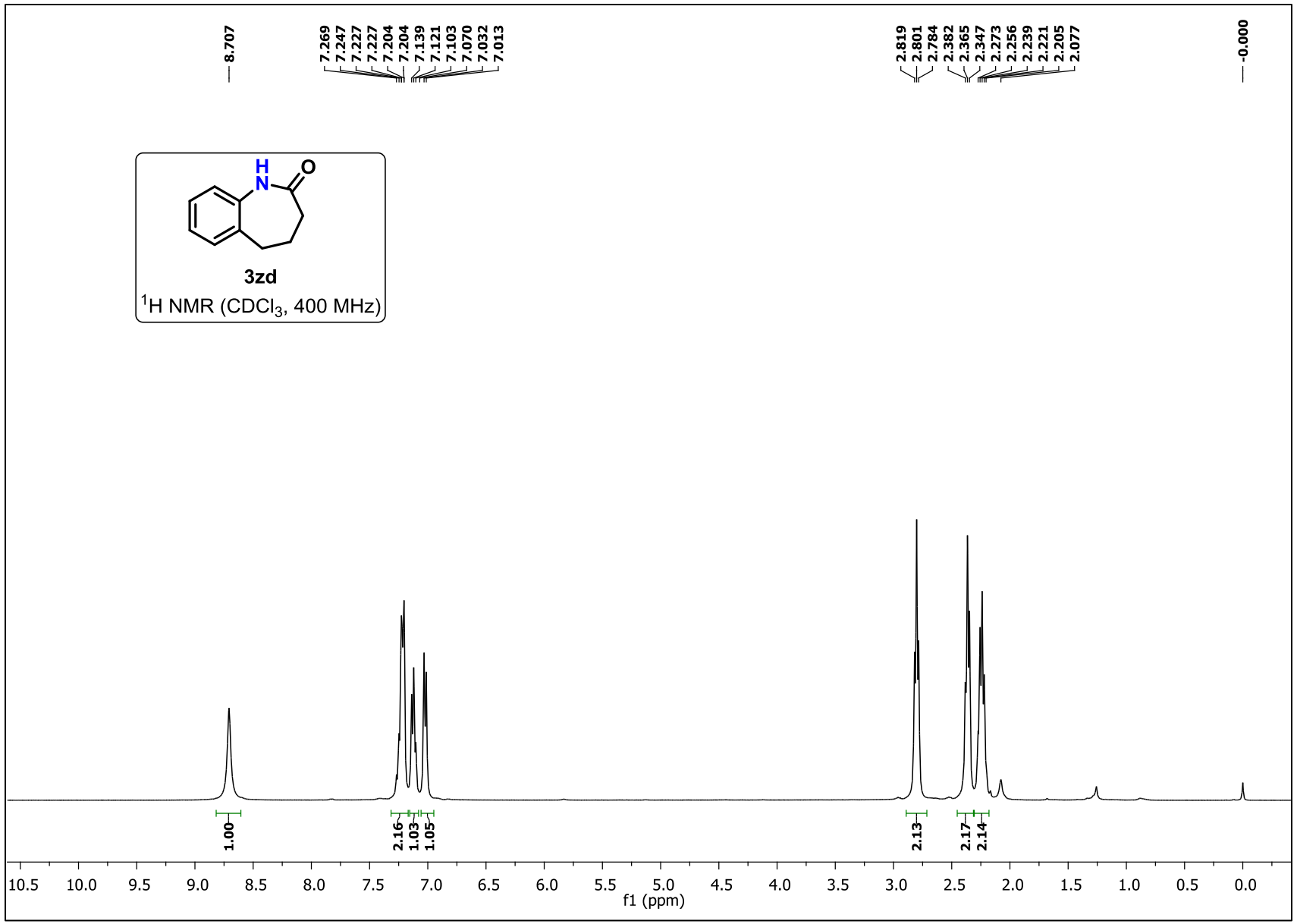

7.535
7.515
7.336
7.317
7.297
7.260
7.116
7.097
7.078
2.578
2.559
2.542
2.524
2.507
2.490
2.473
2.456
2.445
1.600
1.267
1.250
-0.003
Me
H
N
Me
O
3za
1H NMR (CDCl3, 400 MHz)
1.81
1.90
1.00
1.00
6.49
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

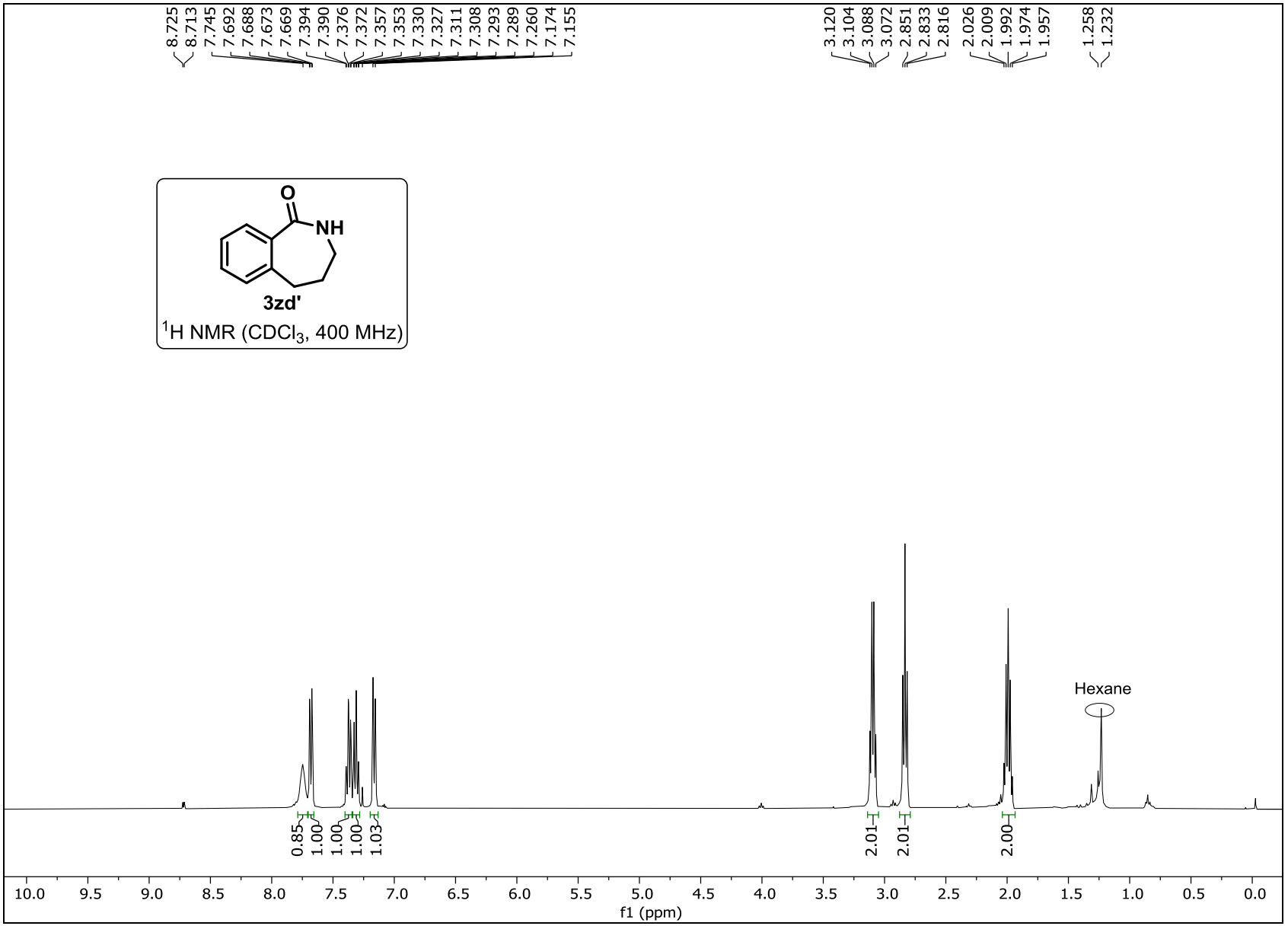

7.755
7.752
7.748
7.734
7.730
7.482
7.478
7.475
7.466
7.460
7.454
7.445
7.442
7.438
7.413
7.410
7.408
7.404
7.393
7.389
7.384
7.375
7.371
7.368
6.090
4.329
4.312
4.296
4.293
4.280
4.276
4.263
4.260
4.247
4.244
4.228
1.969
1.251
1.235
-0.011
O
Me
N
H
Me
3za'
1H NMR (CDCl3, 400 MHz)
2.00
3.06
0.94
1.01
6.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

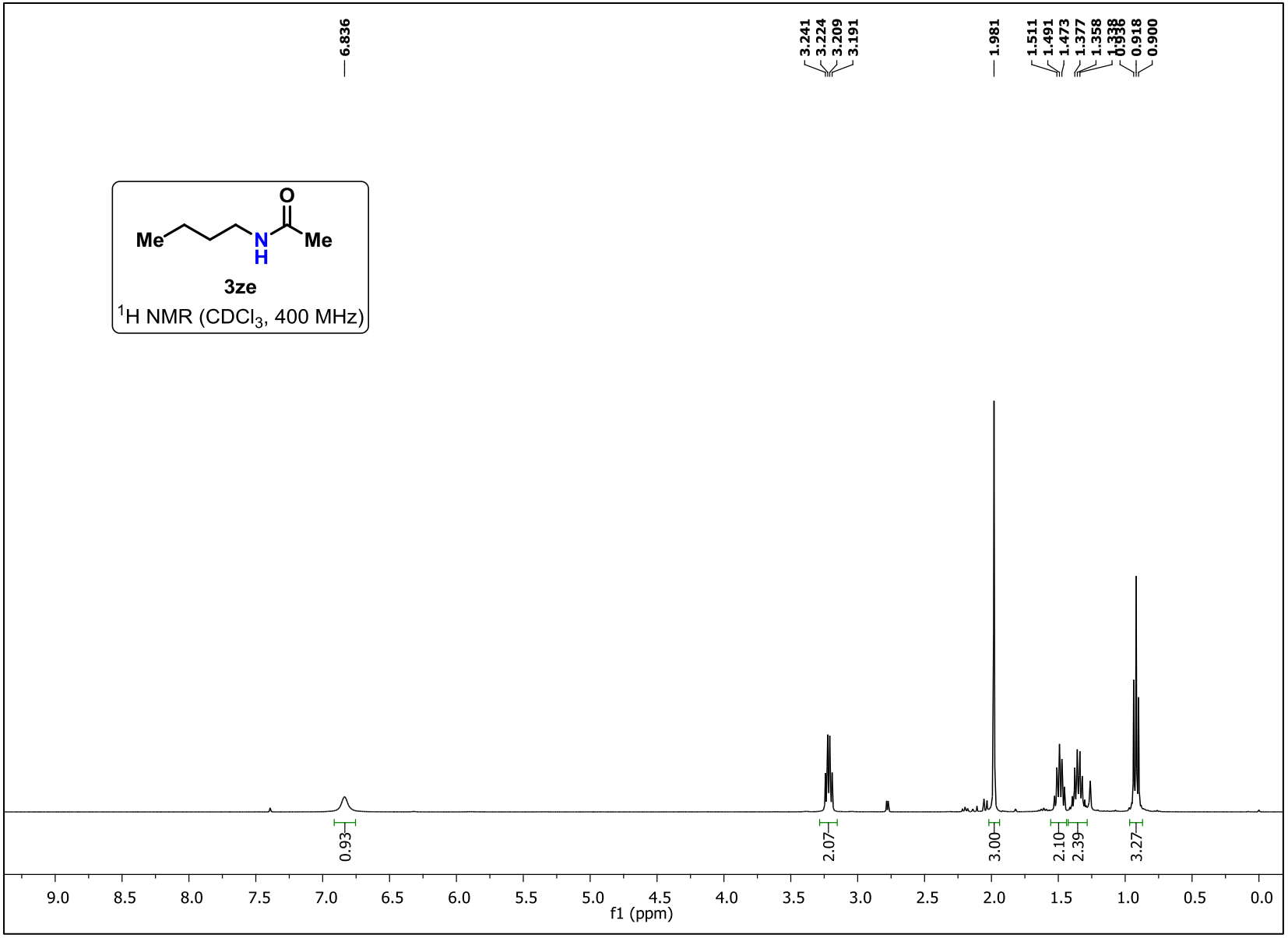

7.535
7.516
7.320
7.300
7.281
7.260
7.102
7.084
7.065
2.285
2.258
2.250
2.229
2.208
2.200
2.174
1.966
1.935
1.887
1.845
1.817
1.773
1.750
1.707
1.692
1.684
1.637
1.621
1.586
1.556
1.526
1.501
1.495
1.471
1.374
1.349
1.318
1.285
1.257
1.229
1.200
H
N
O
3zb
$^1$H NMR ($CDCl_3$, 400 MHz)
1.96
3.02
1.00
0.95
2.03
2.01
1.25
2.01
3.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

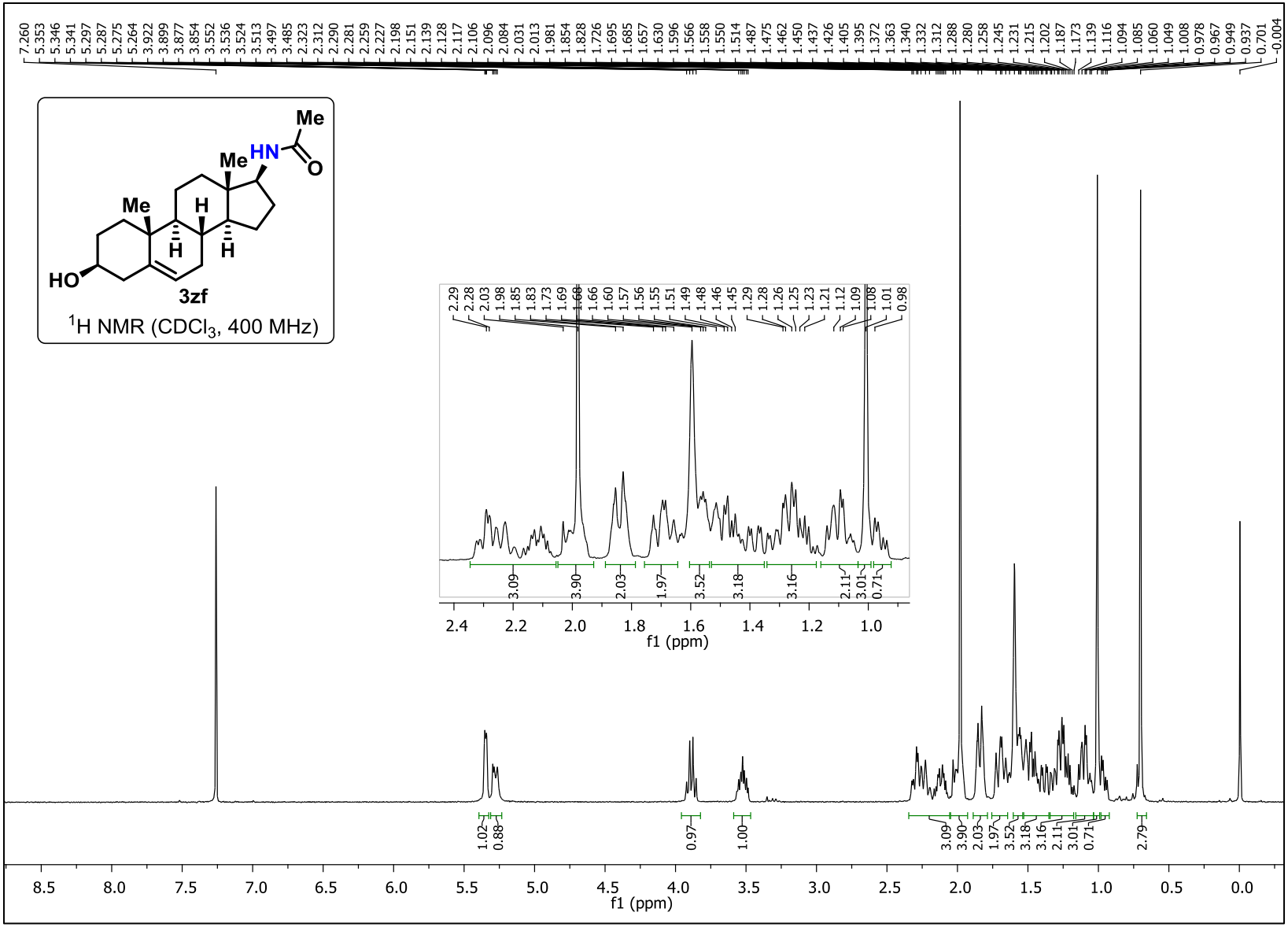

7.755
7.737
7.502
7.484
7.466
7.439
7.420
7.402
7.260
5.958
4.027
4.018
3.998
3.991
3.981
3.971
3.963
3.944
3.935
2.080
2.054
2.046
2.023
2.015
1.986
1.805
1.782
1.773
1.763
1.748
1.739
1.730
1.706
1.672
1.649
1.641
1.590
1.573
1.502
1.488
1.479
1.450
1.418
1.395
1.387
1.369
1.330
1.315
1.297
1.283
1.275
1.251
1.224
1.193
1.172
1.162
0.848
0.832
0.827
0.067
-0.003
O
N
H
3zb'
$^1$H NMR ($CDCl_3$, 400 MHz)
1.82
0.92
1.82
0.93
1.00
1.98
2.11
1.57
2.23
3.41
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


7.260
6.504
3.278
3.272
3.258
3.237
3.220
3.208
3.207
3.201
3.188
3.175
3.149
3.138
2.576
2.500
2.481
2.464
2.446
2.423
2.397
2.362
2.361
2.288
2.026
2.009
1.987
1.965
1.942
1.923
1.899
1.299
1.272
1.244
1.236
1.220
1.199
1.177
1.152
0.862
-0.019
NH
O
Me
Me
Me
3zc
1H NMR (CDCl3, 400 MHz)
0.93
2.01
2.02
2.36
3.24
9.07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


8.707
7.269
7.247
7.227
7.227
7.204
7.204
7.139
7.121
7.103
7.070
7.032
7.013
2.819
2.801
2.784
2.382
2.365
2.347
2.273
2.256
2.239
2.221
2.205
2.077
-0.000
H
N
O
3zd
1H NMR (CDCl3, 400 MHz)
1.00
2.16
1.03
1.05
2.13
2.17
2.14
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


8.725
8.713
7.745
7.692
7.688
7.673
7.669
7.394
7.390
7.376
7.372
7.357
7.353
7.330
7.327
7.311
7.308
7.293
7.289
7.260
7.174
7.155
3.120
3.104
3.088
3.072
2.851
2.833
2.816
2.026
2.009
1.992
1.974
1.957
1.258
1.232
O
NH
3zd'
1H NMR (CDCl3, 400 MHz)
Hexane
0.85
1.00
1.00
1.00
1.03
2.01
2.01
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


6.836
3.241
3.224
3.209
3.191
1.981
1.511
1.491
1.473
1.377
1.358
0.918
0.900
Me
N
H
O
Me
3ze
1H NMR (CDCl3, 400 MHz)
0.93
2.07
3.00
2.10
2.39
3.27
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


Me
HN
O
Me
Me
H
H
H
HO
3zf
$^1$H NMR ($CDCl_3$, 400 MHz)
f1 (ppm)