Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Electronic Supplementary Information

Reductive Cleavage of N-O Bond: Elemental Sulfur-Mediated Conversion of *N*-Alkoxyamides to Amides

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1. General Information

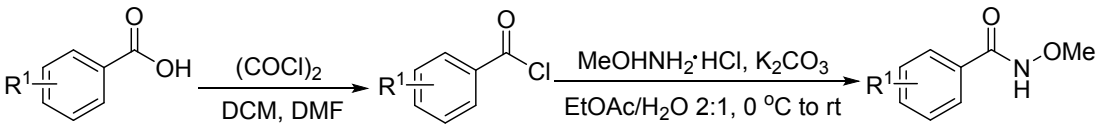
All reactions were carried out at room temperature under air unless otherwise stated. ^{1}H and ^{13}C NMR spectra were recorded on a 600 MHz spectrometer (150 MHz for ^{13}C NMR) at 25 °C. Chemical shift values were given in ppm and referred to the internal standard TMS set as 0.00 ppm. The peak patterns were indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; qui, quintet; m, multiplet; td, triplet of doublets; and dd, doublet of doublets. The coupling constants, J, are reported in Hertz (Hz). Melting points were determined with a micromelting point apparatus without corrections. Infrared spectra was recorded on dry compound using the TENSOR 27, BRUKER. TLC plates were visualized by UV fluorescence quenching and $KMnO_4$ staining.

Reagents and solvents were purchased as reagent grade and were used without further purification. All reactions were performed in standard glassware and heated at 70 °C for 3 h before use. Flash column chromatography was performed over silica gel 200−300 mesh, and the eluent was a mixture of ethyl acetate (EA) and petroleum ether (PE).

2. Experimental Section

2.1 Preparation of Substrates 1

General procedure [1]



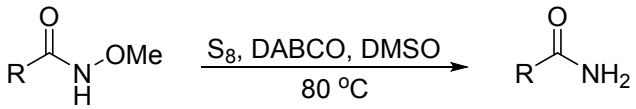
s1 **s2** **1**

A dried round-bottom flask was charged with the acid (4.5 mmol), DCM (15 mL), and 2 drops of DMF. Then, oxalyl chloride (0.60 mL, d = 1.48, 0.876 g, 6.9 mmol) was added dropwise within 5 min at 0 °C. The resulting mixture was stirred at rt for 3.5 h and concentrated under reduced pressure. The residue was dissolved in EA (40 mL) and K_2CO_3 (1.24 g, 9.0 mmol), $MeONH_2{\cdot}HCl$ (458 mg, 5.4 mmol) and water (20 mL)

were added sequentially. The resulting mixture was stirred for 20 h at rt and extracted with EA (50 mL × 2). The organic layer was washed with saturated aqueous $NaHCO_3$ (15 mL × 2), brine (15 mL × 2), and dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was recrystallized (EA) to afford the product. In the case of an oil, the crude product was purified by silica gel column chromatography, using EA/hexane as the eluent.

2.2 Preparation of Substrates 2

General Procedure

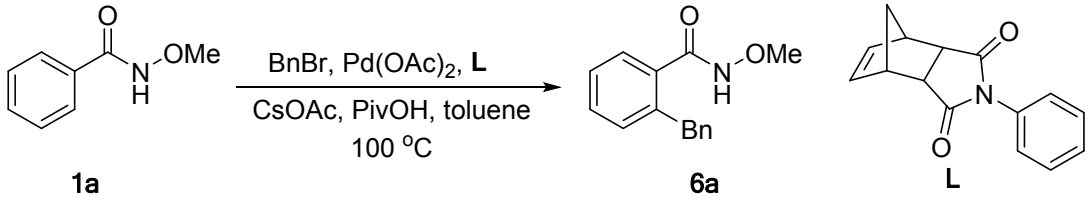


1 **2**

A mixture of *N*-methoxyamide **1** (1.0 equiv), S_8 (0.3 equiv) and DABCO (2.0 equiv) in DMSO (10 mL) was stirred at 80 °C. The reaction was monitored by TLC. After the reaction was completed, the reaction mixture was allowed to cool to room temperature. Then water (50 mL) was added, and the mixture was extracted with DCM (50 mL × 3). The combined organic mixture was washed with brine (30 mL × 2), and dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by silica gel column chromatography, using EA/Petroleum Ether (50/50) as the eluent.

2.3 Preparation of Mono-alkylation Alkoxyamides 6

General Procedure [2]



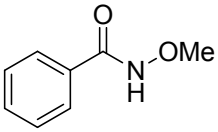
1a **6a** **L**

A solution of amide (0.2 mmol), alkyl halide (1.2 mmol), $Pd(OAc)_2$ (10 mol%), PivOH (0.4 mmol), CsOAc (0.6 mmol), **L** (0.2 mmol), toluene (2 mL) was heated in 100 °C under air. After the reaction completed, the reaction mixture was allowed to cool down to room temperature and saturated aqueous $NaHCO_3$ (20 mL) was added. The resulting mixture was extracted with EA (20 mL x 3). The organic layer was dried

over anhydrous Na_2SO_4, filtered and the solvent was removed under reduced pressure to provide the crude product. The purification was performed by flash column chromatography on silica gel.

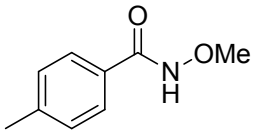
3. Spectral Data of Substrates and Products

N-Methoxybenzamide (1a)[5]



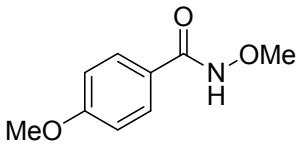
Following the general procedure, amide **1a** was isolated as a white solid. Yield: 80%; mp. 112-114 °C. ^{1}H (600 MHz, $CDCl_3$) δ 9.72 (s, 1H), 7.78 – 7.75 (m, 2H), 7.50 – 7.46 (m, 1H), 7.40 – 7.35 (m, 2H), 3.90 – 3.70 (m, 3H). ^{13}C (150 MHz, $CDCl_3$), δ 166.0, 132.0, 131.7, 128.6, 127.2, 64.3.

N-Methoxy-4-methylbenzamide (1b)[5]



Following the general procedure, amide **1b** was isolated as a white solid. Yield: 85%; mp. 70-72 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.2 Hz, 1H), 7.22 (d, *J* = 8.0 Hz, 1H), 3.86 (s, 1H), 2.38 (s, 1H). ^{13}C (150 MHz, $CDCl_3$), δ 166.3, 142.5, 129.2, 128.9, 127.2, 64.2, 21.5.

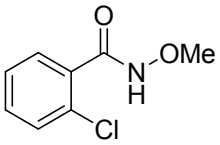
N,4-Dimethoxybenzamide (1c)[5]



Following the general procedure, amide **1c** was isolated as a white solid. Yield: 89%; mp. 99-102 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.76 – 7.70 (m, 2H), 6.90 (dd, *J* = 10.4 Hz, 7.7

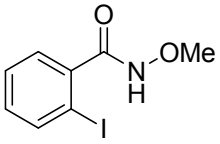
Hz, 2H), 3.86 (s, 3H), 3.85 (s, 3H). ^{13}C (150 MHz, $CDCl_3$), δ 166.0, 162.7, 129.0, 123.8, 113.9, 64.5, 55.4.

2-Chloro-*N*-methoxybenzamide (1d)[6]



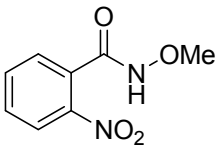
Following the general procedure, amide **1d** was isolated as a white solid. Yield: 70%; mp. 113-114 °C. ^{1}H (600 MHz, $CDCl_3$) δ 9.01 – 8.56 (m, 1H), 7.64 (d, *J* = 7.0 Hz, 1H), 7.41 (dd, *J* = 6.7 Hz, 1.5Hz, 2H), 7.37 – 7.32 (m, 1H), 3.93 (s, 3H). ^{13}C (150 MHz, $CDCl_3$) δ 164.4, 132.1, 131.9, 131.2, 130.3, 130.2, 127.1, 64.6.

2-Iodo-*N*-methoxybenzamide (1e)[6]



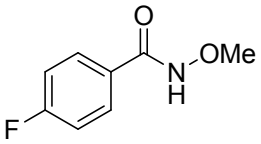
Following the general procedure, amide **1e** was isolated as a white solid. Yield: 91%; mp. 96-97 °C. ^{1}H (600 MHz, $CDCl_3$) δ 8.53 (s, 1H), 7.86 (d, *J* = 8.0, 2H), 7.41 – 7.34 (m, 2H), 7.16 – 7.10 (m, 1H), 3.93 (s, 3H). ^{13}C (150 MHz, $CDCl_3$) δ 165.8, 165.3, 164.2, 129.7, 129.6, 127.8, 127.8, 115.7, 115.6, 64.2.

***N*-Methoxy-2-nitrobenzamide (1f)**



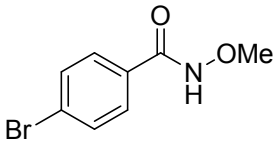
Following the general procedure, amide **1f** was isolated as a white solid. Yield: 91%; mp. 90-91 °C. ^{1}H (600 MHz, $CDCl_3$), δ 9.50 (s, 1H), 8.08 (d, 1H), 7.59 (m, 3H), 3.71 (m, 3H). ^{13}C (150 MHz, $CDCl_3$), δ 164.5, 146.4, 134.0, 133.9, 131.1, 129.5, 124.6, 64.1; HRMS (ESI) m/z calcd for $C_8H_8N_2NaO_4^+$ [M +Na^+] 219.0376, found 219.0357.

4-Fluoro-*N*-methoxybenzamide (1g)[5]



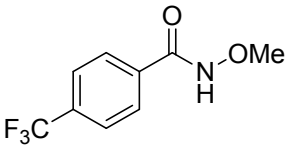
Following the general procedure, amide **1g** was isolated as a white solid. Yield: 91%; mp. 65-66 ^{o}C. ^{1}H (600 MHz, $CDCl_3$) δ 8.52 (s, 1H), 7.85 – 7.66 (m, 2H), 7.09 (t, *J* = 7.9, 2H,), 3.84 (s, 3H). ^{13}C (150 MHz, $CDCl_3$), δ 165.8, 165.3, 164.2, 129.7, 129.6, 127.8, 127.8, 115.8, 115.6, 64.2.

4-Bromo-*N*-methoxybenzamide (1h)[5]



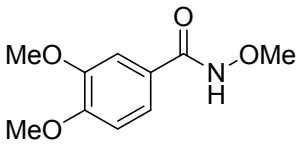
Following the general procedure, amide **1h** was isolated as a white solid. Yield: 89%; mp. 155-157 ^{o}C. ^{1}H (600 MHz, DMSO) δ 11.89 (s, 1H), 7.77 – 7.67 (m, 4H), 3.74 (s, 3H). ^{13}C (150 MHz, DMSO), δ 162.9, 131.5, 131.3, 129.1, 125.3, 63.2.

***N*-Methoxy-4-(trifluoromethyl)benzamide (1i)[5]**



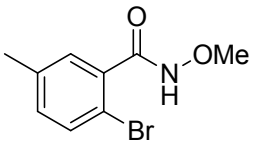
Following the general procedure, amide **1i** was isolated as a white solid. Yield: 78%; mp. 124-126 ^{o}C. ^{1}H (600 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.1 Hz, 2H), 7.70 (d, *J* = 8.2 Hz, 2H), 3.91 (s, 3H). ^{13}C (150 MHz, $CDCl_3$) , δ 164.1, 134.8, 133.8, 133.5, 127.6, 125.7, 125.7, 125.6, 125.6, 124.4, 122.6, 64.3.

***N*, 3, 4-Trimethoxybenzamide (1j)[5]**



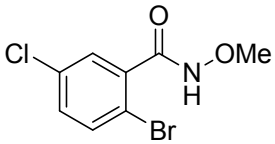
Following the general procedure, amide **1j** was isolated as a white solid. Yield: 86%; mp. 128-129 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.37 (d, *J* = 1.9 Hz, 1H), 7.29 – 7.22 (m, 1H), 6.86 (d, *J* = 8.3 Hz, 1H), 3.94 (s, 3H), 3.93 (s, 3H), 3.89 (s, 3H). ^{13} C (150 MHz, $CDCl_3$) , δ 166.1, 152.2, 149.1, 124.1, 120.0, 110.4, 64.4, 56.0, 56.0.

2-Bromo-*N*-methoxy-5-methylbenzamide (1k)



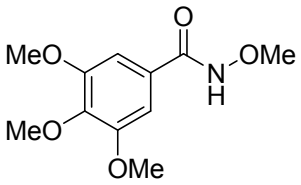
Following the general procedure, amide **1k** was isolated as a white solid. Yield: 93%; mp. 116-118 °C. ^{1}H (600 MHz, $CDCl_3$) δ 8.55 (s, 1H), 7.46 (d, *J* = 8.2 Hz, 1H), 7.34 (s, 1H), 7.12 (dd, *J* = 8.2 Hz, 2.0 Hz, 1H), 3.93 (s, 3H), 2.33 (s, 3H). C (150 Hz, $CDCl_3$), δ 165.4, 137.8, 134.2, 133.1, 132.8, 130.6, 116.3, 64.6, 20.8. HRMS (ESI) m/z calcd for $C_9H_{10}BrNNaO_2^+$ [M +Na^+]265.9787, found 265.9767.

2-Bromo-5-chloro-*N*-methoxybenzamide (1l)



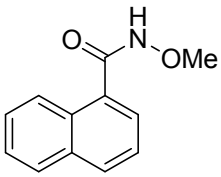
Following the general procedure, amide **1l** was isolated as a white solid. Yield: 89%; mp. 112-114 °C. ^{1}H (600 MHz, $CDCl_3$) δ 9.05 (s, 1H), 7.50 (d, *J* = 8.6 Hz, 1H), 7.43 (s, 1H), 7.27 (dd, *J* = 8.6 Hz, 2.5 Hz, 1H), 3.90 (s, 3H). ^{13}C (150 MHz, $CDCl_3$), δ 163.9, 135.9, 134.5, 133.9, 131.9, 129.8, 117.8, 64.6; HRMS (ESI) m/z calcd for $C_8H_7BrClNNaO_2^+$ [M+Na^+] 285.9241, found 285.9237.

***N*, 3, 4, 5-Tetramethoxybenzamide (1m)**



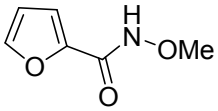
Following the general procedure, amide **1m** was isolated as a white solid. Yield: 91%; mp. 140-142 °C. ^{1}H (600 MHz, $CDCl_3$) δ 6.98 (s, 2H), 3.89 (s，6H), 3.88 (d, *J* = 1.7 Hz，3H). ^{13}C (150 MHz, $CDCl_3$), δ 165.9, 153.3, 141.4, 126.8, 104.4, 64.5, 60.9, 56.3. HRMS (ESI) m/z calcd for $C_{11}H_{15}NNaO_5^+$ [M+Na$^+$] 264.0848, found 264.0826.

_N_-Methoxy-1-naphthamide (1n)[5]



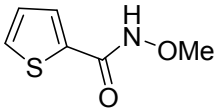
Following the general procedure, amide **1n** was isolated as a white solid. Yield: 91%; mp.156-157 °C.^{1}H (600 MHz, DMSO) δ 11.72 (s，1H), 8.19 (d, *J* = 8.2 Hz, 1H), 8.05 (d, *J* = 8.2 Hz, 1H), 7.99 (d, *J* = 7.7 Hz, 1H), 7.60 (dt, *J* = 8.4 Hz, 6.7 Hz, 3H), 7.56 – 7.51 (m, 1H), 3.82 (s, 3H). ^{13}C (150 MHz, DMSO), δ 165.4, 133.1, 131.5, 130.4, 129.9, 128.3, 127.0, 126.4, 125.7, 124.9, 63.4.

_N_-Methoxyfuran-2-carboxamide (1o)



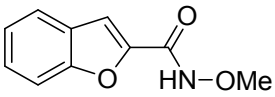
Following the general procedure, amide **1o** was isolated as a purple oil. ^{1}H (600 MHz, $CDCl_3$) δ 8.96 (s, 1H), 7.45 (d, *J* = 1.0 Hz, 1H), 7.21 (d, *J* = 3.5 Hz, 1H), 6.53 (dd, *J* = 3.5 Hz, 1.7 Hz, 1H), 3.89 (s, 1 H). ^{13}C (150 MHz, $CDCl_3$), δ 157.5, 145.7, 144.4, 115.8, 112.1, 65.0. HRMS (ESI) m/z calcd for $C_6H_7NNaO_3^+$ (M+Na$^+$) 164.0318; found 164.0305.

_N_-Methoxythiophene-2-carboxamide (1p)[7]



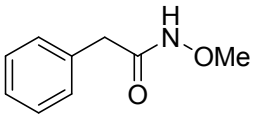
Following the general procedure, amide **1p** was isolated as a white solid. Yield: 70%; mp. 63-65 °C. ^{1}H (600 MHz, $CDCl_3$) δ 9.82 (s, 1H), 7.75 (s, 1H), 7.52 (d, *J* = 4.8 Hz, 1H), 7.07 (dd, *J* = 4.8 Hz, 3.9 Hz, 1H), 3.84 (s, 1H). ^{13}C (150 MHz, $CDCl_3$), δ 161.8, 134.7, 131.1, 129.9, 127.7, 64.6.

_N_-Methoxybenzofuran-2-carboxamide (1q)[8]



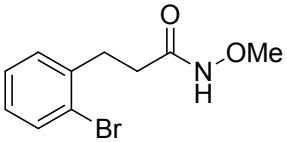
Following the general procedure, amide **1q** was isolated as a white solid. Yield: 70%; mp. 123-125 °C. ^{1}H (600 MHz, $CDCl_3$) δ 9.49 (s, 1H), 7.68 – 7.59 (m, 1H), 7.55 (dd, *J* = 7.3 Hz, 1.3 Hz, 1H), 7.50 – 7.34 (m, 2H), 7.32 – 7.21 (m, 1H), 3.93 (dd, *J* = 8.2 Hz, 2.6 Hz, 3H). ^{13}C (150 MHz, $CDCl_3$) δ157.5, 154.8, 146.6, 127.3, 127.1, 123.9, 122.7, 111.8, 111.7, 64.9.

_N_-Methoxy-2-phenylacetamide (1r)[9]



Following the general procedure, amide **1p** was isolated as a white solid. Yield: 93%; mp. 70-72 °C. ^{1}H (600 MHz, DMSO) δ 11.25 (1H, s), 7.30 (t, *J* = 7.5 Hz, 2H), 7.24 (dd, *J* = 13.3 Hz, 7.2 Hz, 3H), 3.58 (s, 3H), 3.28 (s, 2H). ^{13}C (150 MHz, DMSO) δ 166.9, 135.5, 128.9, 128.3, 126.5, 99.5, 63.2.

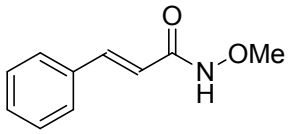
3-(2-Bromophenyl)-_N_-methoxypropanamide (1s)[10]



Following the general procedure, amide **1s** was isolated as a white solid. Yield: 87%; mp. 75-76°C. ^{1}H (600 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.9 Hz, 1H,), 7.30 – 7.25 (m, 1H), 7.23 (t, *J* = 7.4 Hz, 1H), 7.08 (td, *J* = 7.7 Hz, 1.7 Hz, 1H), 3.69 (s, 3H), 3.15 – 3.03 (m,

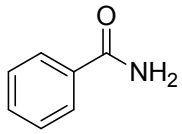
2H), 2.87 – 2.67 (m, 0.5H), 2.41 (s, 1.5H). ^{13}C (150 MHz, $CDCl_3$) δ 169.7, 139.5, 132.9, 130.9, 128.2, 127.7, 124.2, 64.4, 32.9, 31.8.

_N_-Methoxycinnamamide (1t)[11]



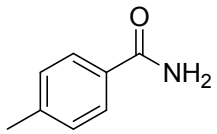
Following the general procedure, amide **1t** was isolated as a white solid. Yield: 90%; mp. 93-94 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.74 (d, *J* = 15.3 Hz, 1H), 7.49 (s, 2H), 7.31 (s, 3H), 6.60 (s, 1H), 3.84 (s, 3H). ^{13}C (150 MHz, $CDCl_3$) δ 164.4, 141.8, 134.7, 130.0, 128.9, 128.0, 117.0, 64.4.

Benzamide (2a)[12]



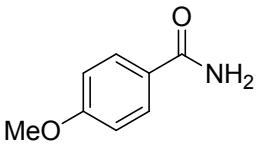
Following the general procedure, benzamide **2a** was isolated as a white solid. Yield: 85%; mp. 125-127 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.82 (dd, *J* = 8.3 Hz, 1.2 Hz, 2H), 7.54 (dd, *J* = 10.7 Hz, 4.2 Hz , 1H), 7.46 (t, *J* = 7.7 Hz, 2 H), 6.09 (d, 1H), 6.18 (s, 1H), 5.99 (s, 1H).

4-Methylbenzamide (2b)[12]



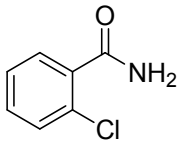
Following the general procedure, 2**b** was isolated as a white solid. Yield: 85%; mp. 160-162 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.2 Hz, 2H), 7.26 (d, *J* = 7.5 Hz, 2H), 6.08 (s, 1H), 5.69 (s, 1H), 2.41 (s, 3H).

4-*M*ethoxybenzamide (2c)[13]



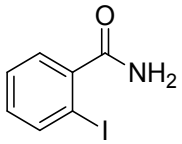
Following the general procedure, **2c** was isolated as a white solid. Yield: 82%; mp. 155-157 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.8 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 6.21 – 5.51 (m, 2H), 3.86 (s, 3H).

2-Chlorobenzamide (2d)[14]



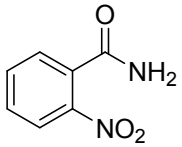
Following the general procedure, **2d** was isolated as a white solid. Yield: 77%; mp. 138-139 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 7.6 Hz, 1.6 Hz, 1H), 7.46 – 7.41 (m, 1H), 7.42 (d, *J* = 1.6 Hz, 1H), 7.36 (td, *J* = 7.4 Hz, 1.3 Hz, 1H), 6.37 (s, 1H), 5.94 (s, 1H).

2-Iodobenzamide (2e)



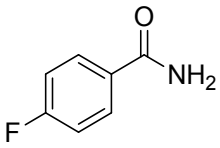
Following the general procedure, **2e** was isolated as a white solid. Yield: 76%; mp. 185-187 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.9 Hz, 1H), 7.48 (dd, *J* = 7.6 Hz, 1.4 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.13 (td, *J* = 7.8 Hz, 1.4 Hz, 1H), 5.89 (s, 1H), 5.85 (s, 1H).

2-Nitrobenzamide (2f)[14]



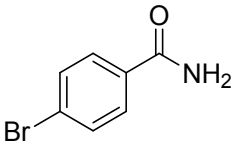
Following the general procedure, **2f** was isolated as a white solid. Yield: 94%; mp. 175-177 °C. ^{1}H (600 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.2 Hz, 1H), 7.71 (td, *J* = 7.5 Hz, 1.0 Hz, 1H), 7.65 – 7.59 (m, 2H), 5.83 (s, 2H).

4-Fluorobenzamide (2g)[13]



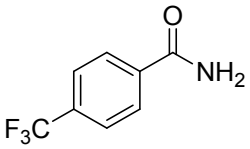
Following the general procedure, **2g** was isolated as a white solid. Yield: 90%; mp. 155-156 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.84 (dd, *J* = 8.8 Hz, 5.3 Hz, 2H), 7.13 (t, *J* = 8.6 Hz, 2H), 6.08 (s, 1H), 5.92 (s, 1H).

4-Bromobenzamide (2h)[12]



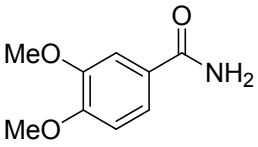
Following the general procedure, **2h** was isolated as a white solid. Yield: 70%; mp. 187-191 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.71 – 7.67 (2 H, m), 7.62 – 7.59 (2 H, m), 6.06 (1 H, s), 5.73 (1 H, s).

4-(Trifluoromethyl)benzamide (2i)[14]



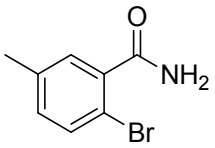
Following the general procedure, **2i** was isolated as a white solid. Yield: 80%; mp. 183-185 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.1 Hz, 1H), 7.73 (d, *J* = 8.1 Hz, 1H), 6.13 (s, 1H), 5.81 (s, 1H).

3,4-Dimethoxybenzamide (2j)[13]



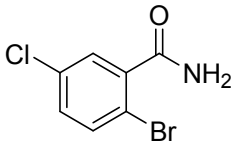
Following the general procedure, **2j** was isolated as a white solid. Yield: 82%; mp. 164-165 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.47 (d, *J* = 1.9 Hz, 1H), 7.34 (dd, *J* = 8.3 Hz, 2.0 Hz, 1H), 6.89 (d, *J* = 8.3 Hz, 1H), 6.11 (s, 1H), 5.70 (s, 1H), 3.95 (s, 3H), 3.94 (s, 3H).

2-Bromo-5-methylbenzamide (2k)[15]



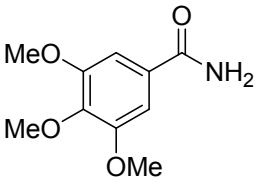
Following the general procedure, **2k** was isolated as a white solid. Yield: 80%; mp. 197-199 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.48 (d, *J* = 2.7 Hz, 1H), 7.48 (d, *J* = 3.8 Hz, 1H), 7.11 (dd, *J* = 8.2 Hz, 1.8 Hz, 1 H), 6.18 (s, 1H), 6.09 (s, 1H), 2.34 (s, 3H).

2-Bromo-5-chlorobenzamide (2l)



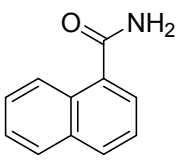
Following the general procedure, **2l** as isolated as a white solid. Yield: 76%; mp. 160-162 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.64 (d, *J* = 2.6 Hz, 1H), 7.55 (d, *J* = 8.5 Hz, 1H), 7.29 (dd, *J* = 8.5 Hz, 2.6 Hz, 1H), 6.13 (s, 1H), 5.99 (s, 1H). HRMS (ESI) m/z calcd for $C_7H_5BrClNNaO^+$ ($M + Na^+$) 255.9135; found 255.9130.

3,4,5-Trimethoxybenzamide (2m)[12]



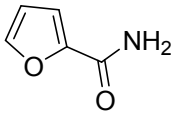
Following the general procedure, **2m** as isolated as a white solid. Yield: 85%; mp. 173-174 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.05 (s, 2H), 6.07 (s, 1H), 5.73 (s, 1H), 3.92 (s, 6H), 3.90 (s, 3H).

α-Naphthamide (2n)[12]



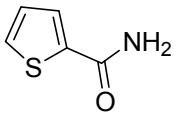
Following the general procedure, **2n** was isolated as a white solid. Yield: 83%; mp. 197-199 °C. ^{1}H (600 MHz, $CDCl_3$) δ 8.42 (d, *J* = 8.4 Hz, 1H), 7.96 (d, *J* = 8.3 Hz, 1H), 7.89 (d, *J* = 7.9 Hz, 1H), 7.72 (dd, *J* = 7.0 Hz, 0.9 Hz, 1H), 7.59 (ddd, *J* = 8.4 Hz, 6.9 Hz, 1.3 Hz, 1H), 7.56 – 7.51 (m, 1H), 7.48 (dd, *J* = 8.1 Hz, 7.2 Hz, 1H), 6.08 (s, 2H).

Furan-2-carboxamide (2o)[13]



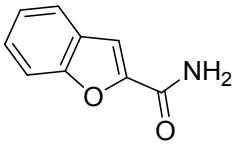
Following the general procedure, **2o** was isolated as a white solid. Yield: 76%; mp. 140-141 °C. ^{1}H (600 MHz, DMSO) δ 7.81 (dd, *J* = 1.6 Hz, 0.7 Hz, 1H), 7.78 (s, 1H), 7.38 (s, 1H), 7.10 (dd, *J* = 3.4 Hz, 0.6 Hz, 1H), 6.60 (dd, *J* = 3.4 Hz, 1.7 Hz, 1H).

Thiophene-2-carboxamide (2p)[12]



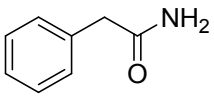
Following the general procedure, **2p** was isolated as a white solid. Yield: 81%; mp. 190-191 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.57 – 7.55 (m, 1H), 7.54 (d, *J* = 5.0 Hz, 1H), 7.10 (dt, *J* = 12.0 Hz, 6.0 Hz, 1H), 5.85 (s, 2H).

Benzofuran-2-carboxamide (2q)[16]



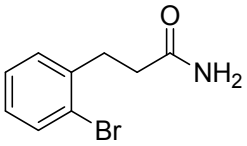
Following the general procedure, **2q** was isolated as a white solid. Yield: 85%; mp. 155-157 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.9 Hz, 1H), 7.52 (d, *J* = 7.1 Hz, 2H), 7.44 (t, *J* = 7.8 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 6.57 (s, 1H), 6.04 (s, 1H).

***N*-Methoxy-2-phenylacetamide (2r) [17]**



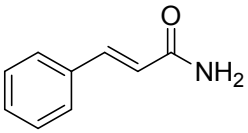
Following the general procedure, **2r** was isolated as a white solid. Yield: 70%; mp. 154-156 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.37 (t, *J* = 7.4 Hz, 2H), 7.31 (t, *J* = 4.7 Hz, 1H), 7.30 – 7.27 (m, 2H), 5.69 (s, 1H), 5.40 (s, 1H), 3.59 (s, 2H).

3-(2-Bromophenyl)-*N*-methoxypropanamide (2s)[18]



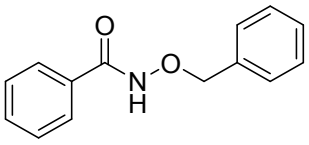
Following the general procedure, **2s** was isolated as a white solid. Yield: 77%; mp. 105-107 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.54 (dd, *J* = 8.0 Hz, 1.0 Hz, 1H), 7.28 (dd, *J* = 7.6 Hz, 1.7 Hz, 1H), 7.24 (td, *J* = 7.5 Hz, 1.1 Hz, 1H), 7.09 (td, *J* = 7.7 Hz, 1.8 Hz, 1H), 5.55 (s, 1H), 5.43 (s, 1H), 3.37 – 2.89 (m, 2H), 2.74 – 2.33 (m, 2H).

***N*-Cinnamamide (2t)[17]**



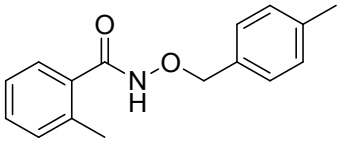
Following the general procedure, **2t** was isolated as a white solid. Yield: 88%; mp. 145-147 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.66 (d, *J* = 15.7 Hz, 1 H), 7.52 (dd, *J* = 6.5 Hz, 3.0 Hz, 2H), 7.39 (dd, *J* = 5.0 Hz, 1.9 Hz, 3H), 6.47 (d, *J* = 15.7 Hz, 1H), 5.69 (s, 1H).

***N*-(Benzyloxy)benzamide (3a)[5]**



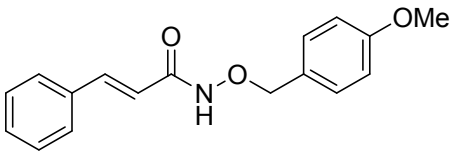
Following the modified procedure, **3a** was isolated as a white solid. Yield: 78%; m.p. 107-110 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.67 (d, *J* = 7.5 Hz, 1H), 7.50 (dd, *J* = 10.7 Hz, 4.2 Hz, 1H), 7.44 (d, *J* = 7.2 Hz, 1H), 7.42 – 7.34 (m, 2H), 5.03 (s, 1H).

Methyl-*N*-((4-methylbenzyl)oxy)benzamide (3b)



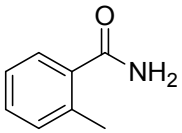
Following the modified procedure, **3b** was isolated as a white solid. Yield: 95%; m.p. 84-86 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.38 – 7.29 (m, 3H), 7.24 (d, *J* = 7.3 Hz, 1H), 7.20 (t, *J* = 7.8, 3H), 7.15 (t, *J* = 7.4, 1H), 5.02 (s, 2H), 2.41 (s, 3H), 2.36 (s, 3H). HRMS (ESI) m/z calcd for $C_{16}H_{17}NNaO_2^+$ [M + Na^+] 278.1151, found 278.1137.

***N*-((4-Methoxybenzyl)oxy)cinnamamide (3c)**



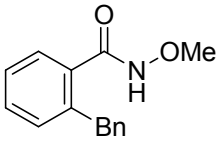
Following the modified procedure, **3c** was isolated as a white solid. Yield: 88%; m.p. 117-118 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.68 (d, *J* = 15.7 Hz, 1H), 7.46 (s, 2H), 7.33 (d, *J* = 8.2 Hz, 5H), 6.87 (d, *J* = 8.3 Hz, 2H), 6.34 (br s, 1H), 4.90 (s, 2H), 3.76 (s, 3H). HRMS (ESI) m/z calcd for $C_{17}H_{17}NNaO_3^+$ [M + Na^+] 306.1101, found 306.1088

2-Methylbenzamide (4b)[5]



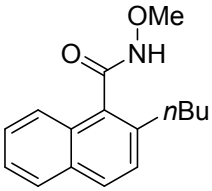
Following the general procedure, **4b** was isolated as a white solid. Yield: 78%; mp. 143-144 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.46 (1 H, dd, *J* = 7.6, 1.0), 7.34 (1 H, td, *J* = 7.6, 1.3), 7.24 (1 H, d, *J* = 7.7), 7.22 (1 H, t, *J* = 7.5), 5.97 (1H, s), 5.91 – 5.58 (1 H, m), 2.50 (1 H, s).

2-Benzyl-*N*-methoxybenzamide (6a)[2]



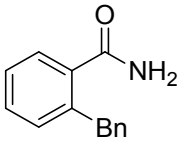
Following the general procedure, **6a** was isolated as a white solid. Yield: 70%; mp. 100-102 °C. ^{1}H (600 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.8 Hz, 1H), 7.41 – 7.37 (m, 1H), 7.35 (d, *J* = 7.2 Hz, 1H), 7.29 – 7.22 (m, 5H), 7.20 – 7.12 (m, 3H), 4.19 (s, 2H), 3.70 (s, 3H).

2-Butyl-*N*-methoxy-1-naphthamide (6b)[2]



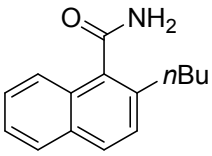
Following the general procedure, **6b** was isolated as a colorless oil. Yield: 65%. ^{1}H (600 MHz, $CDCl_3$) δ 8.42 (s, 1H), 7.79 – 7.68 (m, 4H), 7.42 (dd, *J* = 11.2 Hz, 4.0 Hz, 2H), 7.37 (t, *J* = 7.4 Hz, 2H), 7.25 (d, *J* = 8.5 Hz, 1H), 3.90 (s, 4H), 2.73 – 2.64 (m, 3H), 1.61 (dd, *J* = 15.0 Hz, 7.5 H, 3Hz), 1.36 – 1.27 (m, 3H), 0.80 (t, *J* = 7.0 Hz, 4H).

2-Benzylbenzamide (7a)



Following the general procedure, 7**a** was isolated as a white solid. Yield: 80%; mp. 165-167 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.48 (dd, *J* = 7.6 Hz, 0.9 Hz, 1H), 7.41 – 7.34 (m, 1H), 7.31 – 7.25 (m, 4H), 7.24 (t, *J* = 7.1 Hz, 1H), 7.18 (t, *J* = 8.0 Hz, 3H), 5.84 (s, 1 H), 5.70 (s, 1H), 4.24 (s, 2H). HRMS (ESI) m/z calcd for $C_{14}H_{13}NNaO^+$ [M + Na^+]234.0889, found 306.1073.

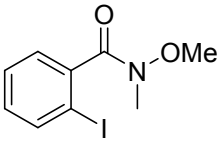
2-Butyl-1-naphthamide (7b)



Following the general procedure, **7b** was isolated as a white solid. Yield: 89%; mp. 68-70 °C.^{1}H (600 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.3 Hz, 1H), 7.81 (d, *J* = 3.3 Hz, 1H), 7.80 (d, *J* = 3.8 Hz, 1H), 7.58 – 7.48 (m, 1H), 7.48 – 7.41 (m, 1H), 7.36 (d, *J* = 8.5 Hz, 1 H), 6.31 (s, 1H), 5.91 (s, 1H), 2.99 – 2.58 (m, 2H), 1.70 (dt, *J* = 15.6 Hz, 7.7 Hz, 2H), 1.48 – 1.35 (m, 2H), 0.89 (dd, *J* = 9.3 Hz, 4.8 Hz, 3H). HRMS (ESI) m/z calcd for

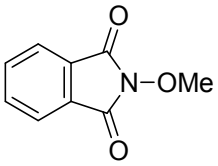
$C_{15}H_{17}NNaO^+$ [M + Na^+] 250.1202, found 250.1193.

2-Iodo-*N*-methoxy-*N*-methylbenzamide (6)



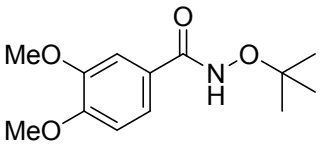
Following the literature procedure,[1] **6** was isolated as a white solid. Yield: 50%; mp. 55-58 °C.^{1}H (600 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 8.0 Hz, 0.9 Hz, 1H), 7.39 (t, *J* = 7.4 Hz, 1H), 7.27 (dd, *J* = 7.6 Hz, 1.4 Hz, 1H), 7.10 (td, *J* = 7.8 Hz, 1.4 Hz, 1H), 3.93 (s, 1H), 3.46 (s, 2H), 3.40 (s, 2H), 3.11 (s, 1H). ^{13}C (150 MHz, $CDCl_3$) δ 170.8, 141.7 138.9, 130.3, 127.7, 127.2, 92.5, 61.4, 32.6

2-Methoxyisoindoline-1,3-dione (7)



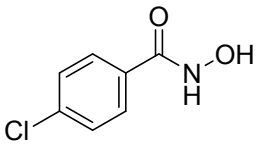
Following the literature procedure,[3] **7** was isolated as a white solid. Yield: 89%; mp. 135-138 °C.^{1}H (600 MHz, $CDCl_3$) δ 7.85 (ddd, *J* = 5.3 Hz, 3.1 Hz, 1.0 Hz, 2H), 7.78 – 7.75 (m, 2H), 4.08 (d, *J* = 0.9 Hz, 3H). ^{13}C (150 MHz, $CDCl_3$) δ 163.3, 134.6, 128.9, 123.6, 65.9.

***N*-(*tert*-Butoxy)-3,4-dimethoxybenzamide (8)**



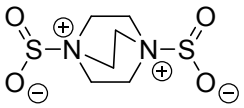
Following the literature procedure,[1] **8** was isolated as a white solid. Yield: 71%; mp. 114-115 °C. ^{1}H (600 MHz, $CDCl_3$) δ 7.39 (d, *J* = 1.5 Hz, 1H), 7.30 (d, *J* = 7.9 Hz, 1 H), 6.85 (d, *J* = 8.3 Hz, 1H), 3.93 (s, 3H), 3.92 (s, 3H), 1.34 (s, 8H). ^{13}C (150 MHz, $CDCl_3$) δ 167.2, 152.1, 149.2, 124.8, 119.6, 110.7, 110.3, 82.3, 56.1, 56.0, 26.4. HRMS (ESI) m/z calcd for $C_{13}H_{19}NNaO_4^+$ [M + Na^+] 276.1206, found 276.1177.

4-Chloro-*N*-hydroxybenzamide (9)



Following the literature procedure,[4] **9** was isolated as a white solid. Yield: 70 %; mp. 183-185 ^{o}C. ^{1}H (600 MHz, DMSO) δ 11.30 (s, 1H), 9.11 (s, 1H), 7.76 (d, *J* = 8.6 Hz, 2H), 7.53 (d, *J* = 8.5Hz, 2H). ^{13}C (150 MHz, DMSO) δ 163.1, 135.9, 131.5, 128.8, 128.5.

1,4-Diazabicyclo[2.2.2]octane-1,4-diium-1,4-disulfinate



In the reaction of substrate **1a**, DABSO was isolated as a white solid. ^{1}H (600 MHz, CD_3OD) δ 3.20 (s); ^{13}C (150 MHz, MeOD) δ 45.59. IR ν max (KBr)/cm^{-1} 3441, 3010, 2802, 2284, 1646, 1474, 1411, 1174, 1098, 1055.

.

4. References

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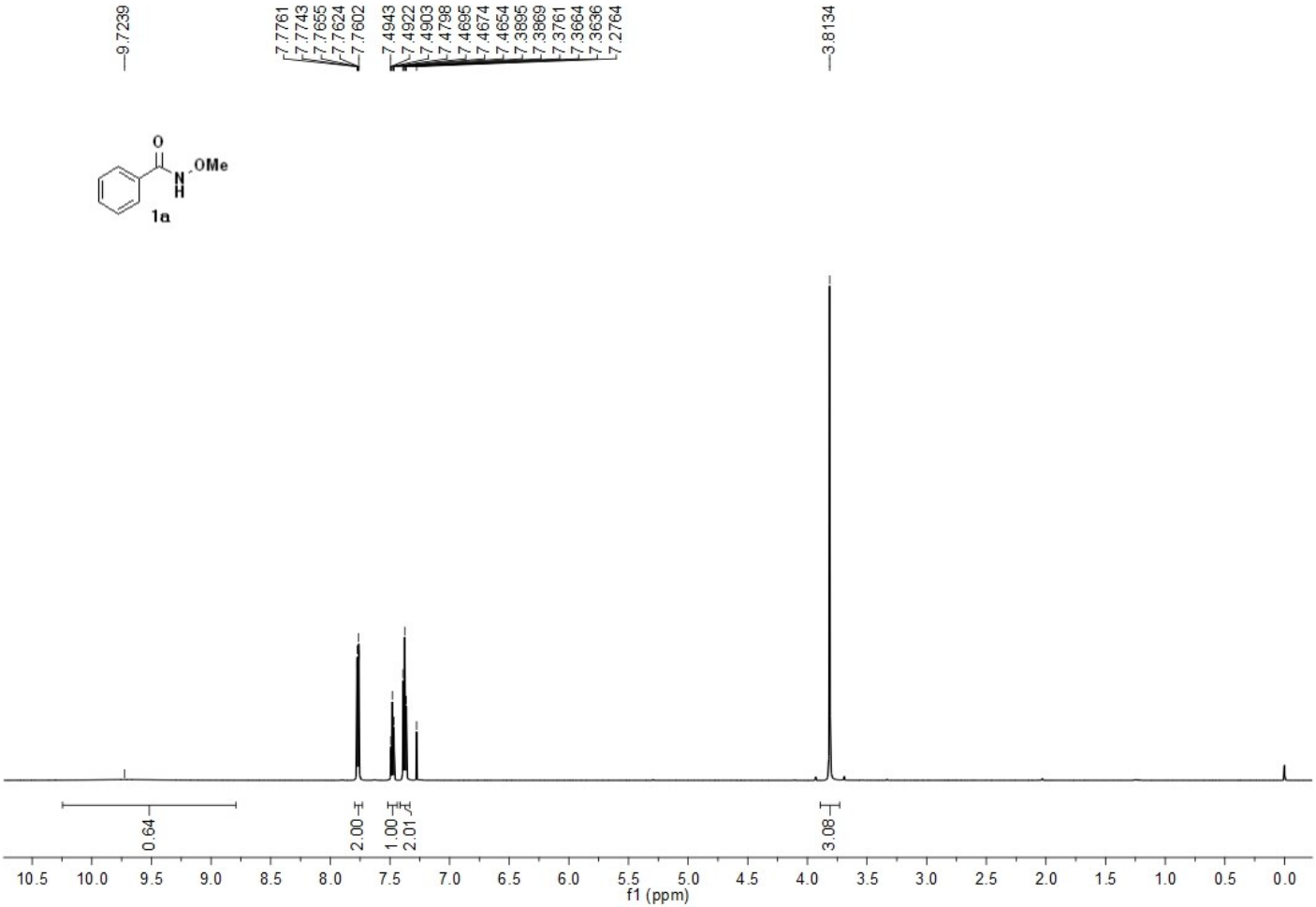
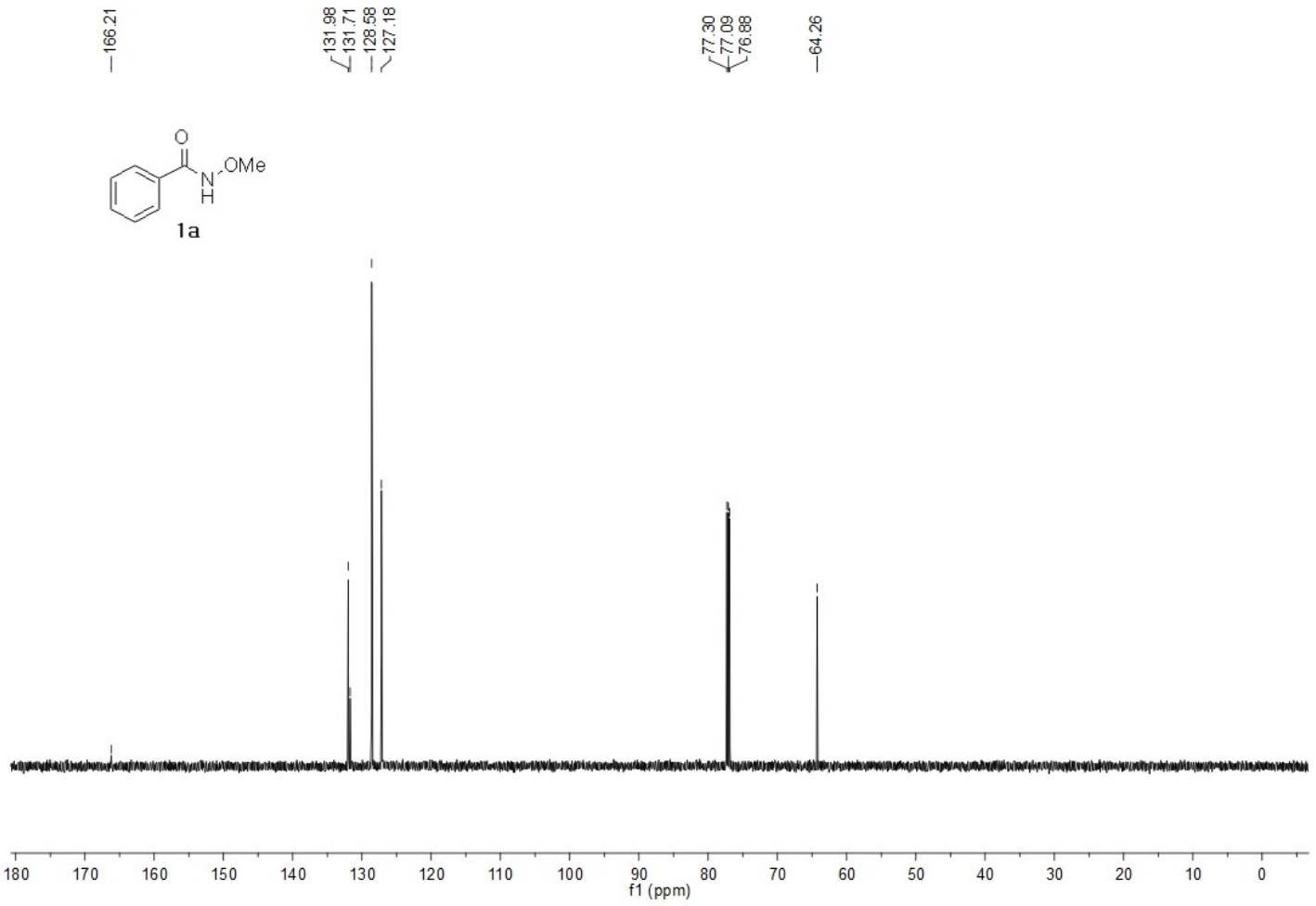
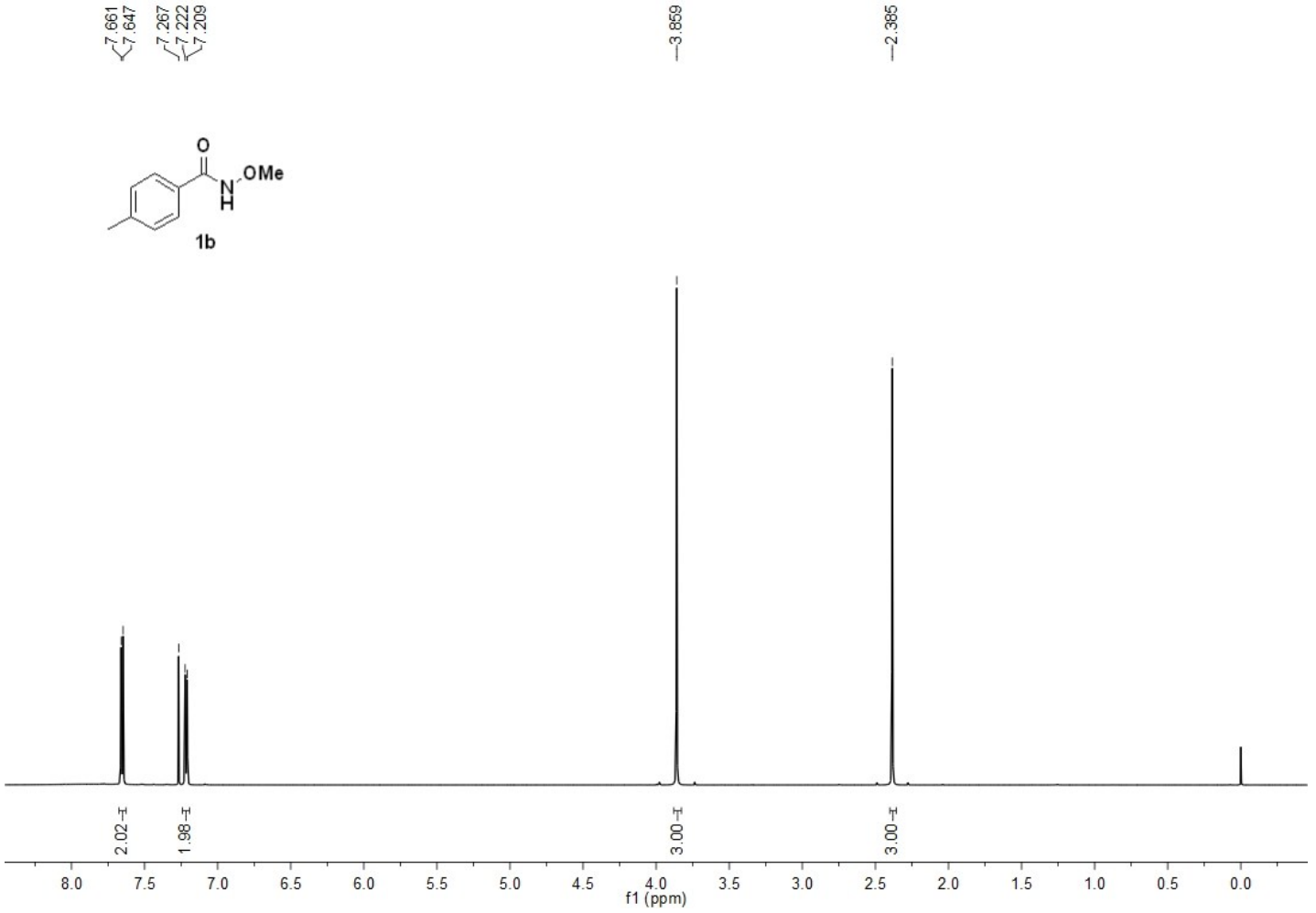
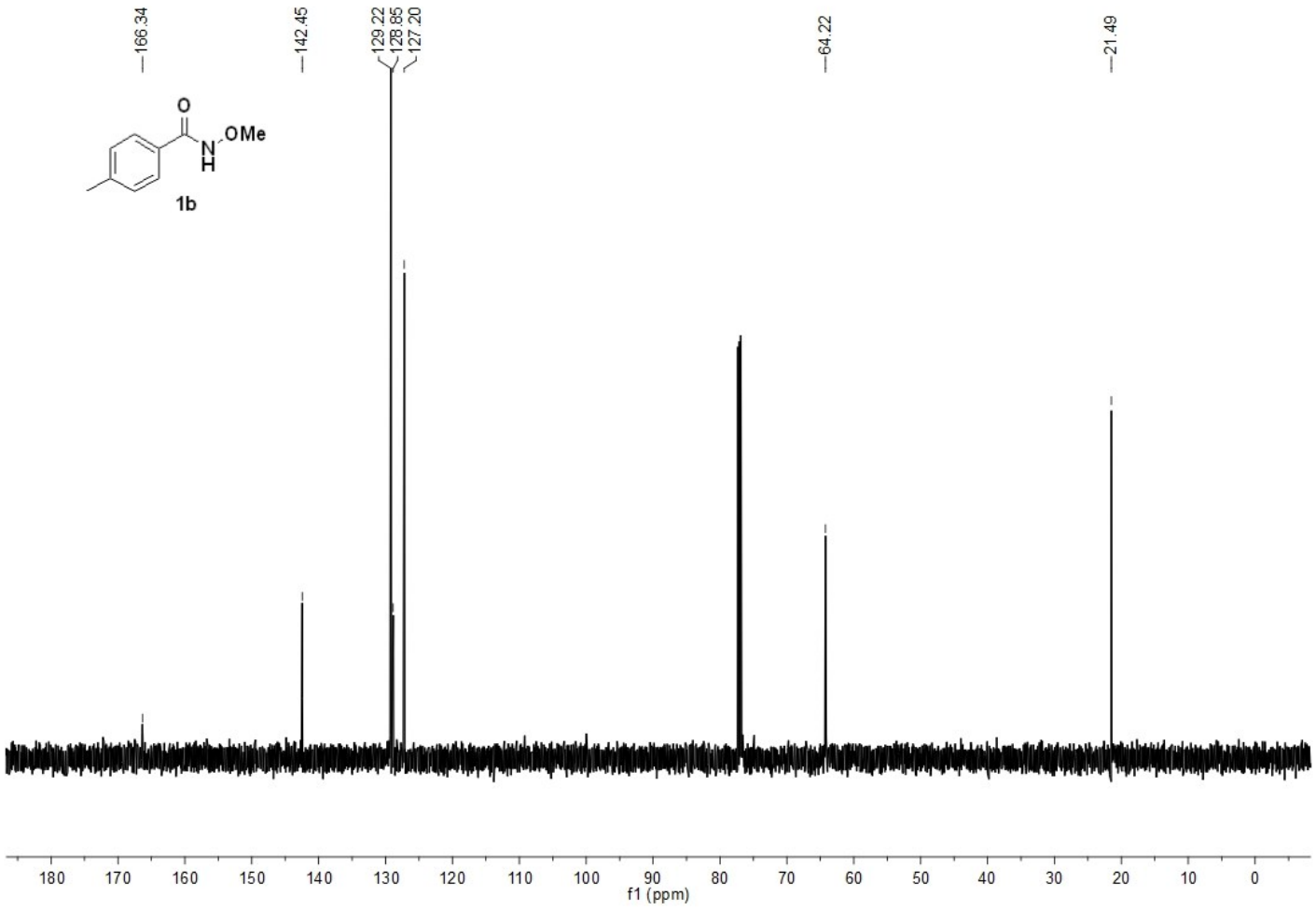
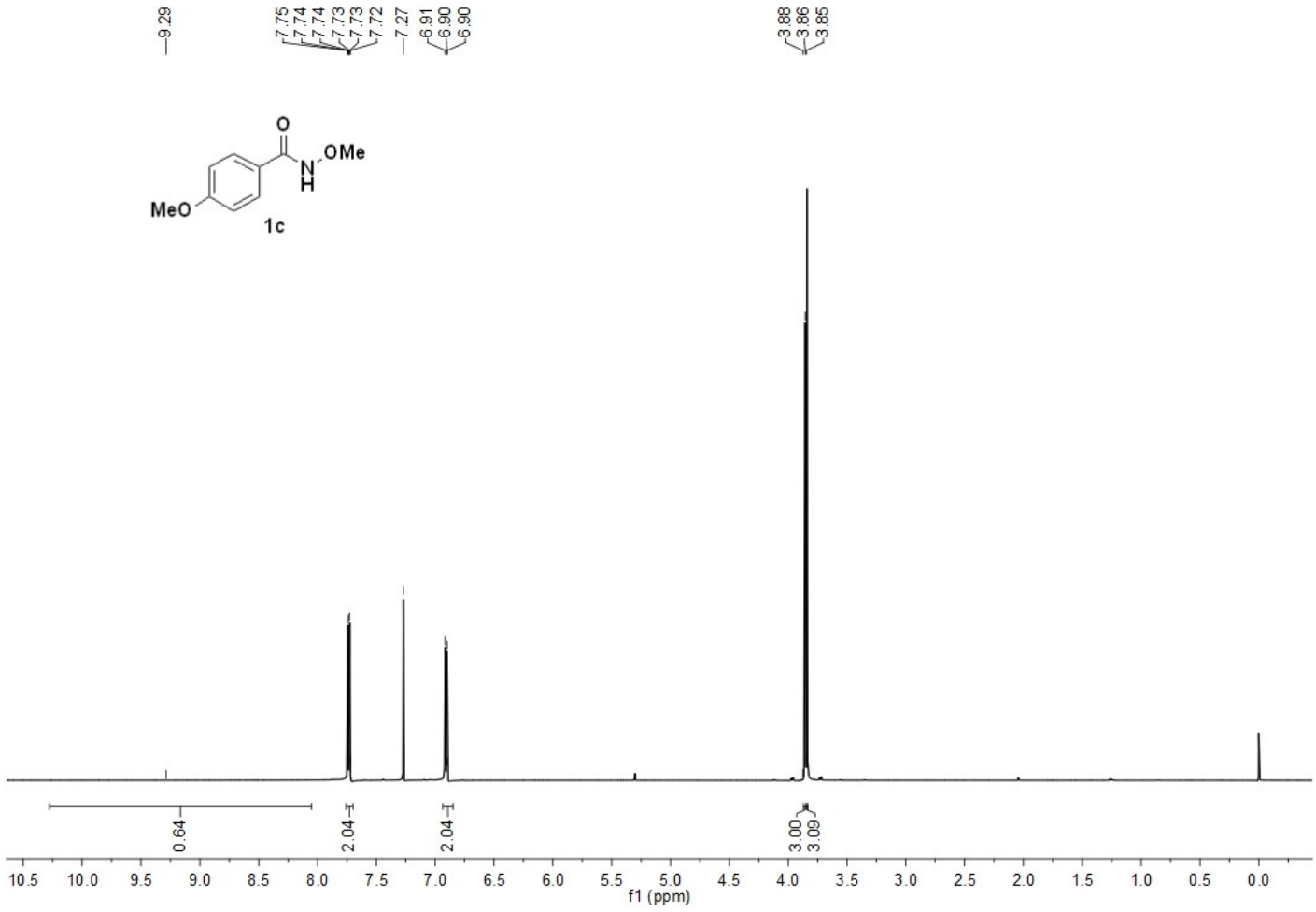
5. ^{1}H-NMR and ^{13}C-NMR Spectra




166.21
131.98
131.71
128.58
127.18
77.30
77.09
76.88
64.26
O
N
H
OMe
1a
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.661
7.647
7.267
7.222
7.209
3.859
2.385
O
N
H
OMe
1b
2.02
1.98
3.00
3.00
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)


166.34
142.45
129.22
128.85
127.20
64.22
21.49
O
N
OMe
H
1b
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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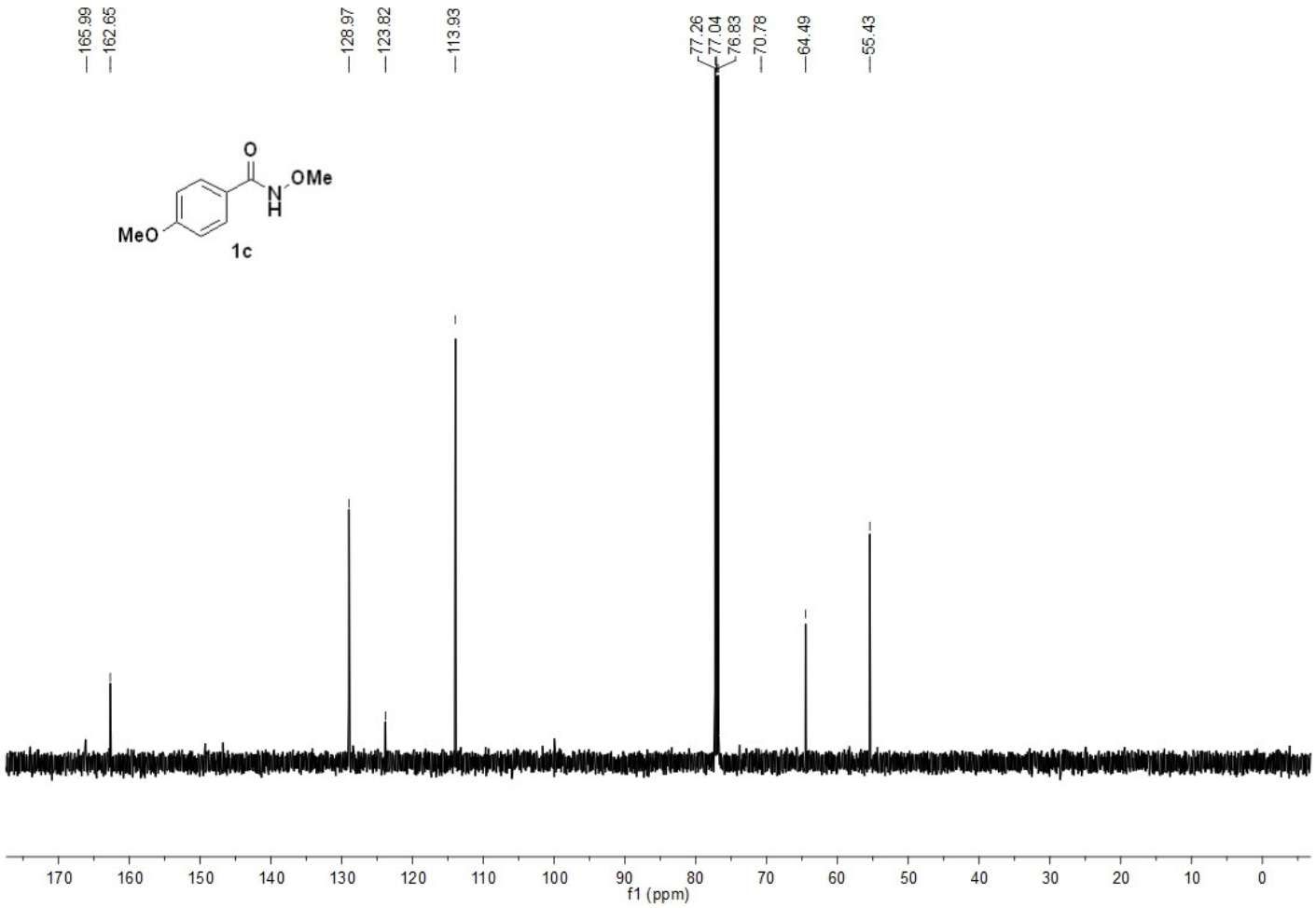
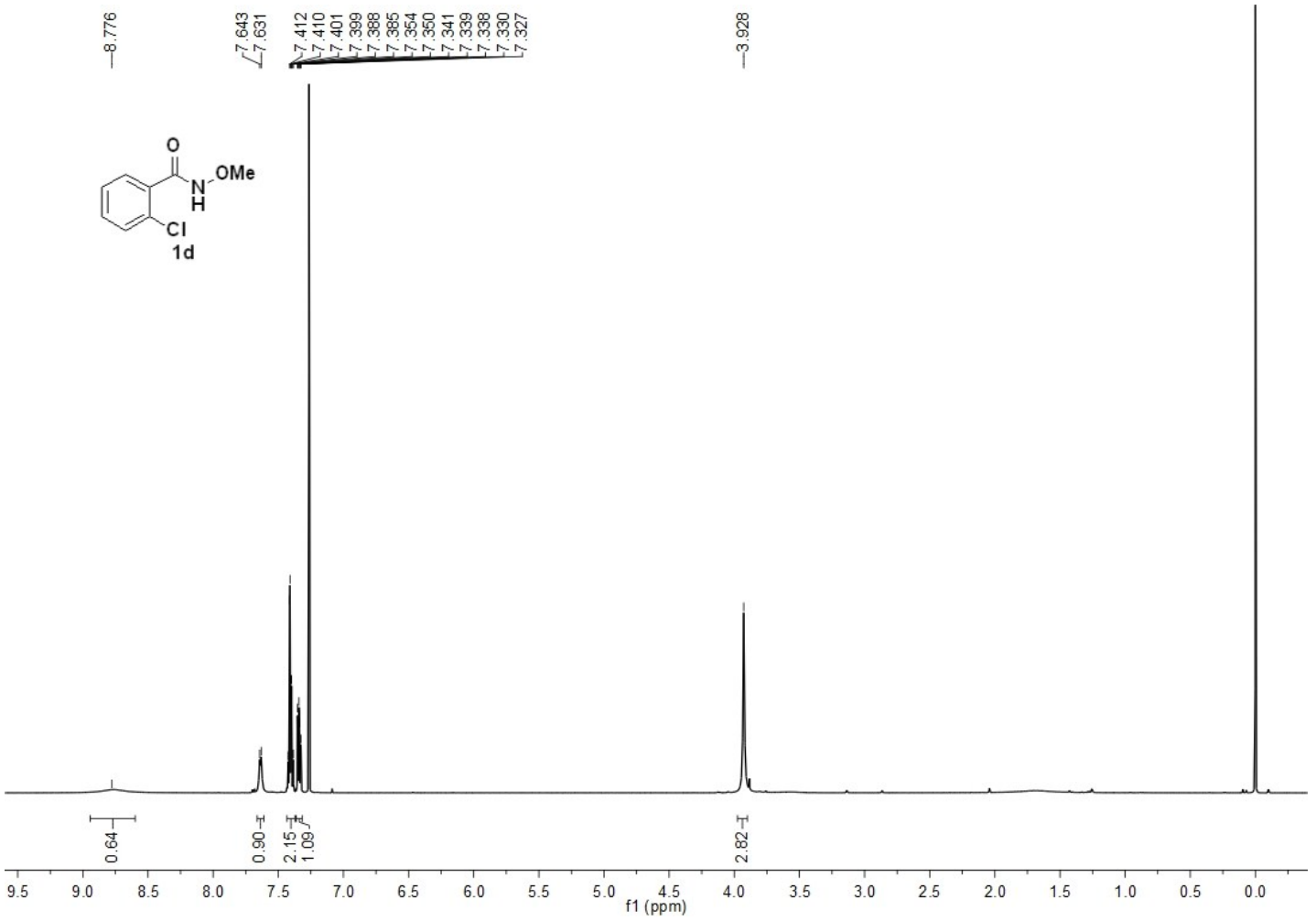
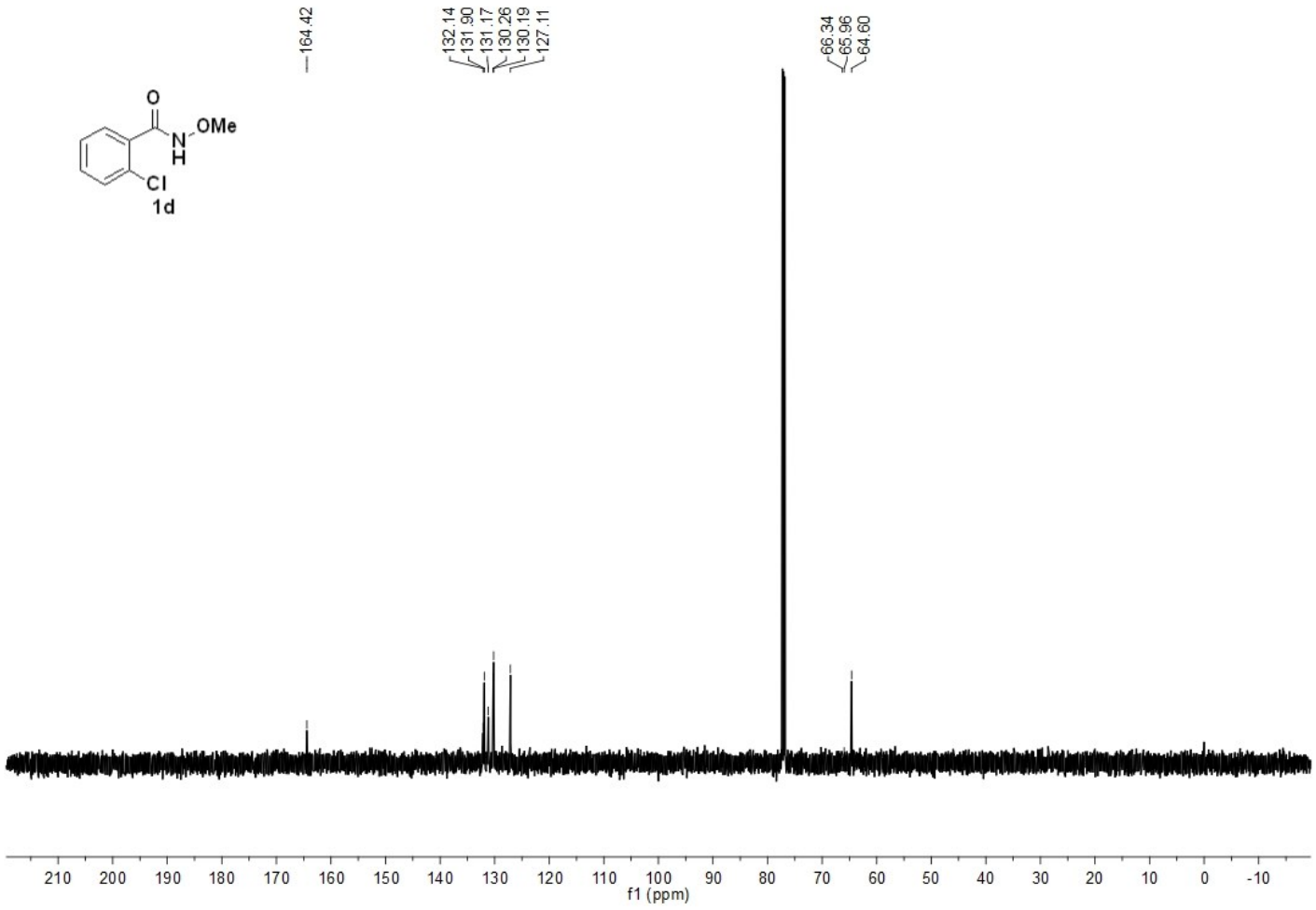
7.75
7.74
7.74
7.73
7.73
7.72
7.27
6.91
6.90
6.90
3.88
3.86
3.85
O
N
H
OMe
MeO
1c
0.64
2.04
2.04
3.00
3.09
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


165.99
162.65
128.97
123.82
113.93
77.26
77.04
76.83
70.78
64.49
55.43
O
N
H
OMe
MeO
1c
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.776
7.643
7.631
7.412
7.410
7.401
7.399
7.388
7.385
7.354
7.350
7.341
7.339
7.338
7.330
7.327
3.928
O
N
OMe
H
Cl
1d
0.64
0.90
2.15
1.09
2.82
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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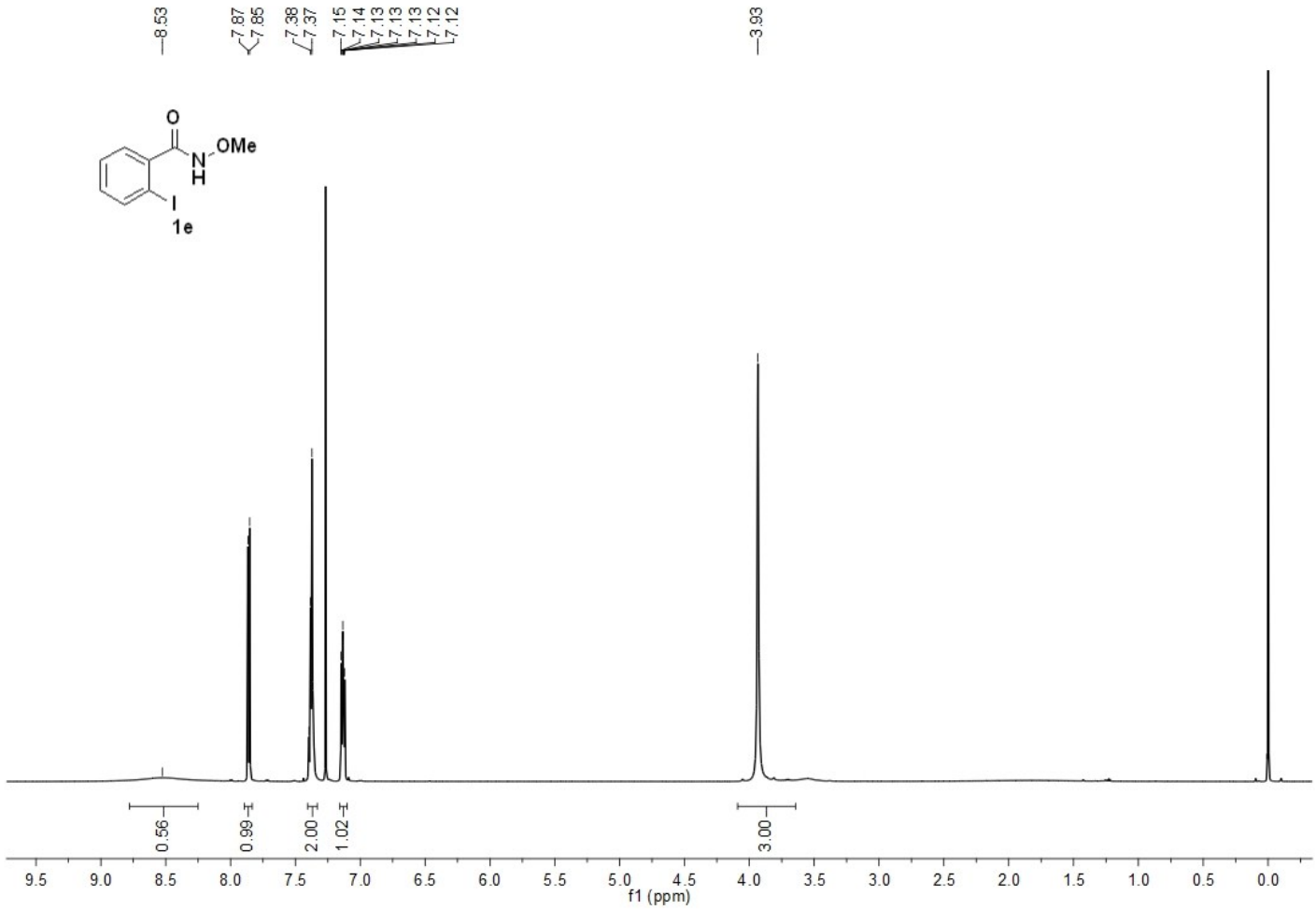
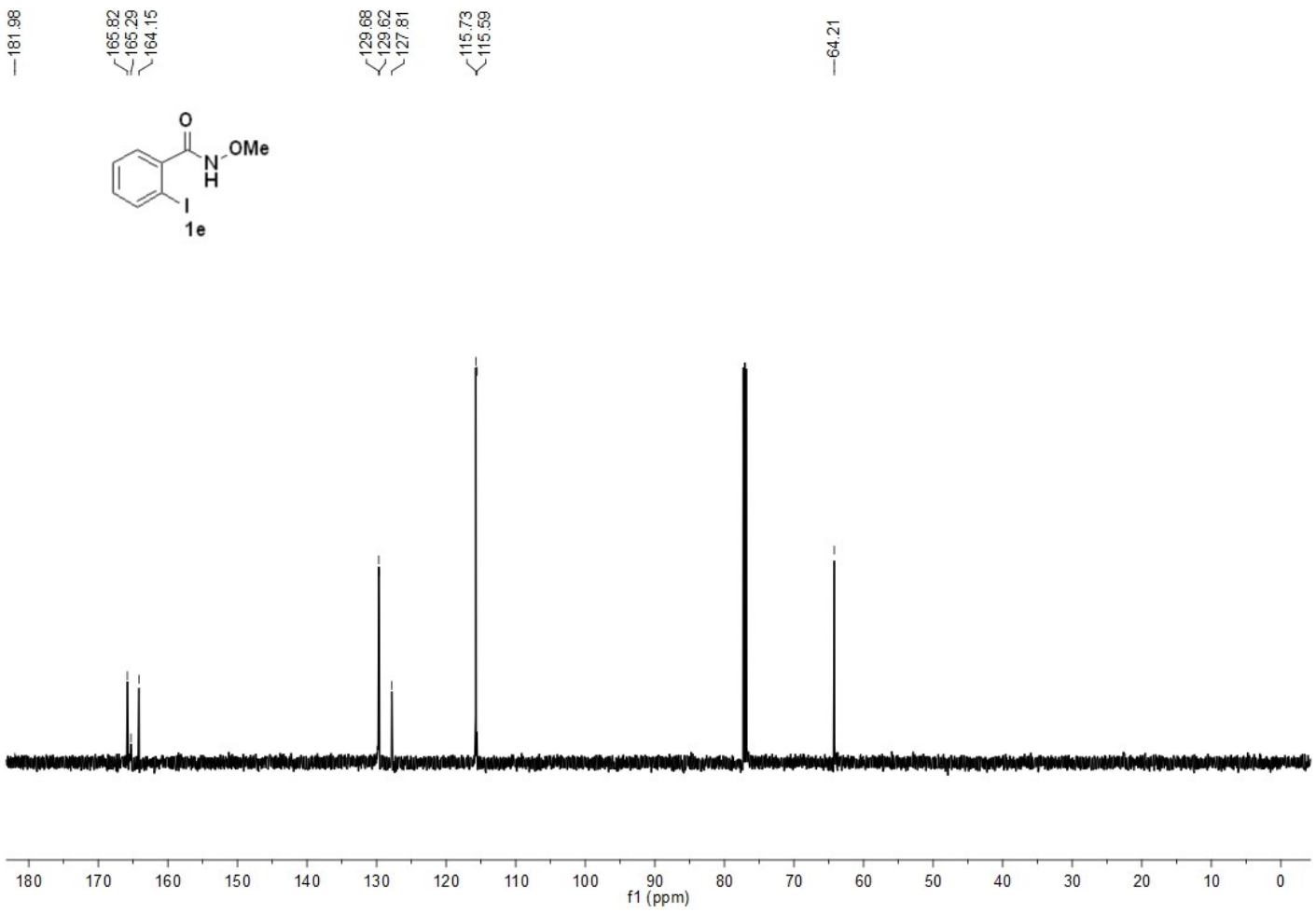
131.90
131.17
130.26
130.19
127.11
66.34
65.96
64.60
O
N
H
OMe
Cl
1d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


8.53
7.87
7.85
7.38
7.37
7.15
7.14
7.13
7.13
7.13
7.12
7.12
3.93
O
N
H
OMe
I
1e
0.56
0.99
2.00
1.02
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)


181.98
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165.29
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129.62
127.81
115.73
115.59
64.21
O
N
H
OMe
I
1e
180
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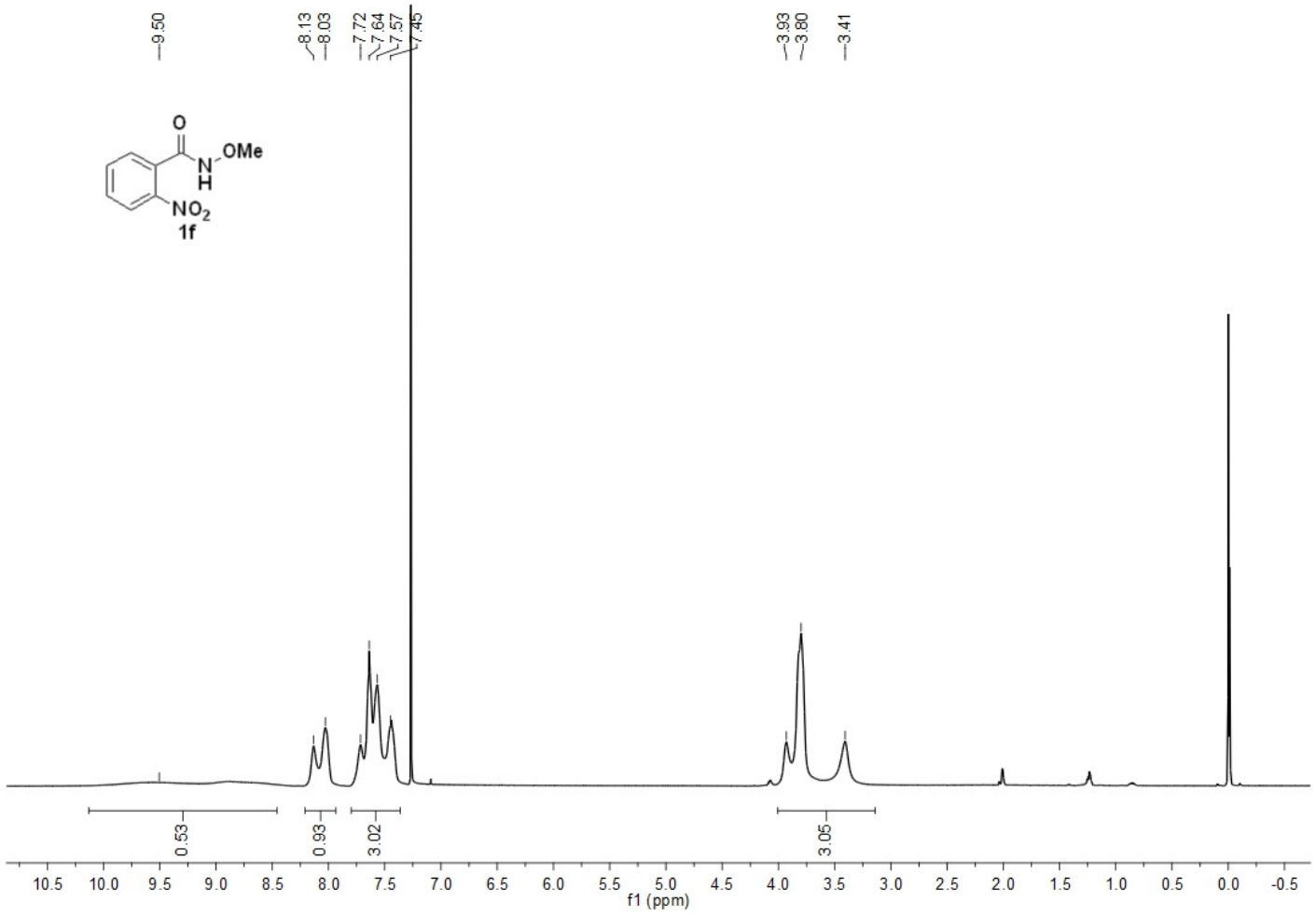
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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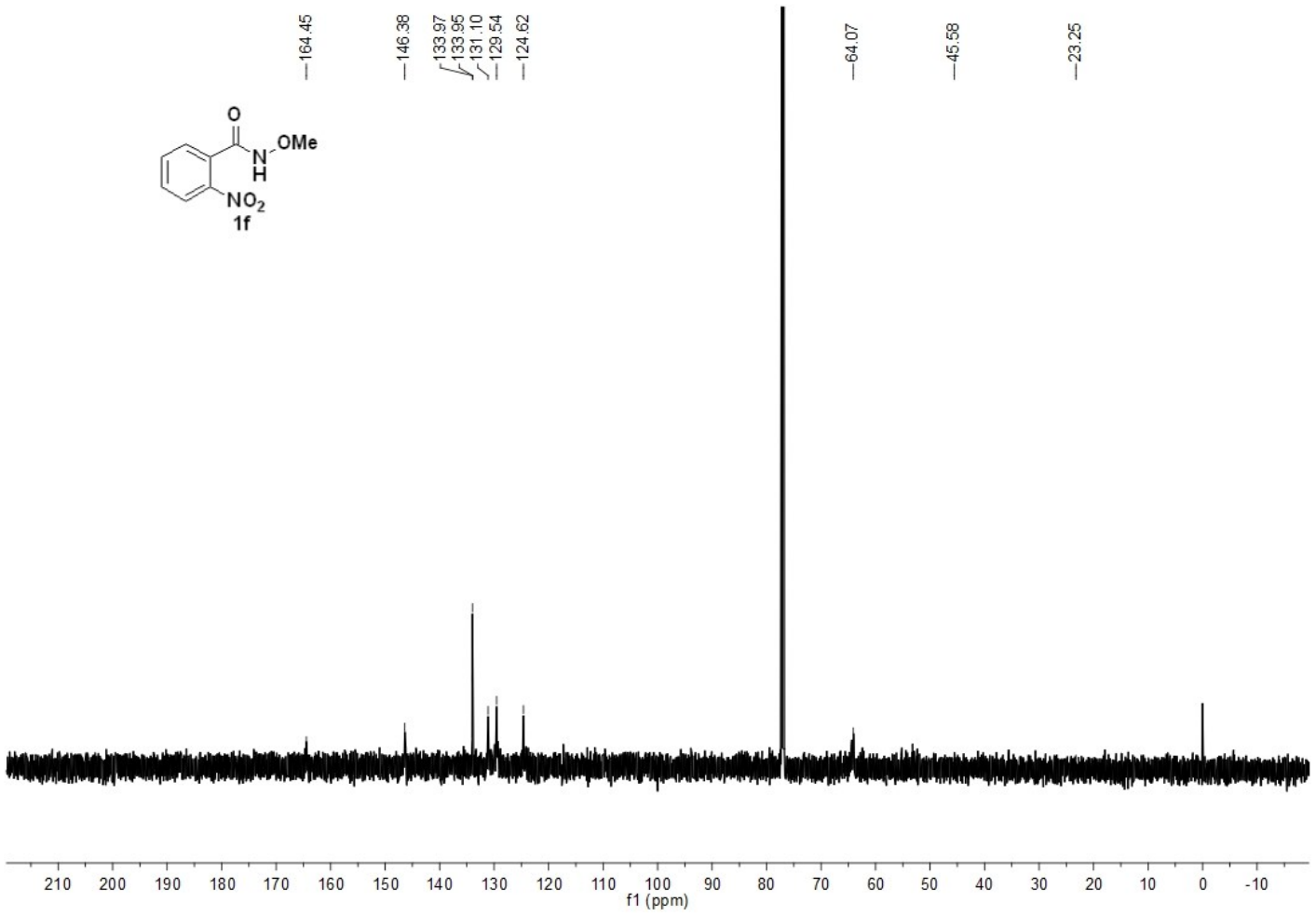
8.13
8.03
7.72
7.64
7.57
7.45
3.93
3.80
3.41
O
N
H
OMe
NO2
1f
0.53
0.93
3.02
3.05
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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133.95
131.10
129.54
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64.07
45.58
23.25
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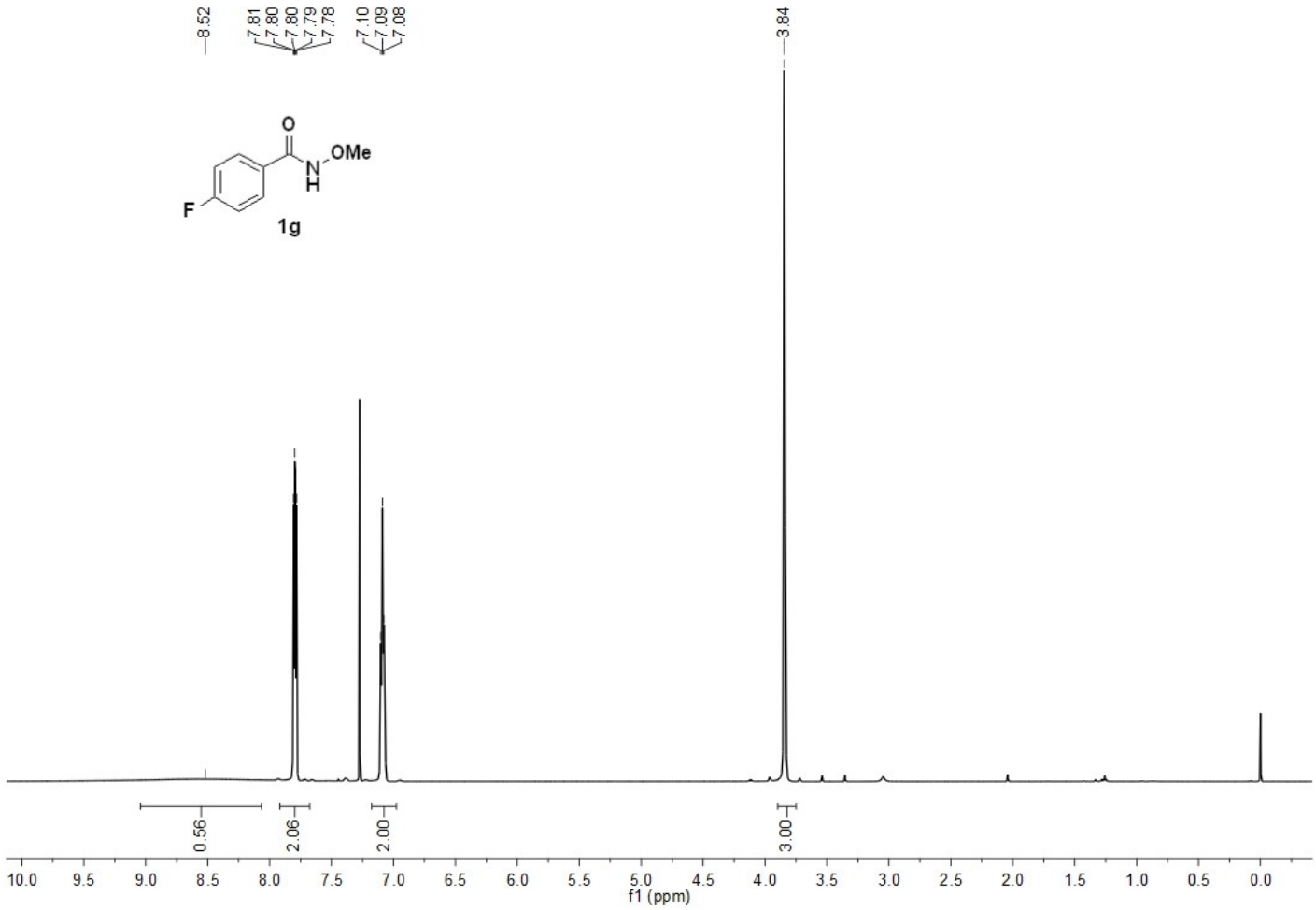
N
OMe
H
NO2
1f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.08
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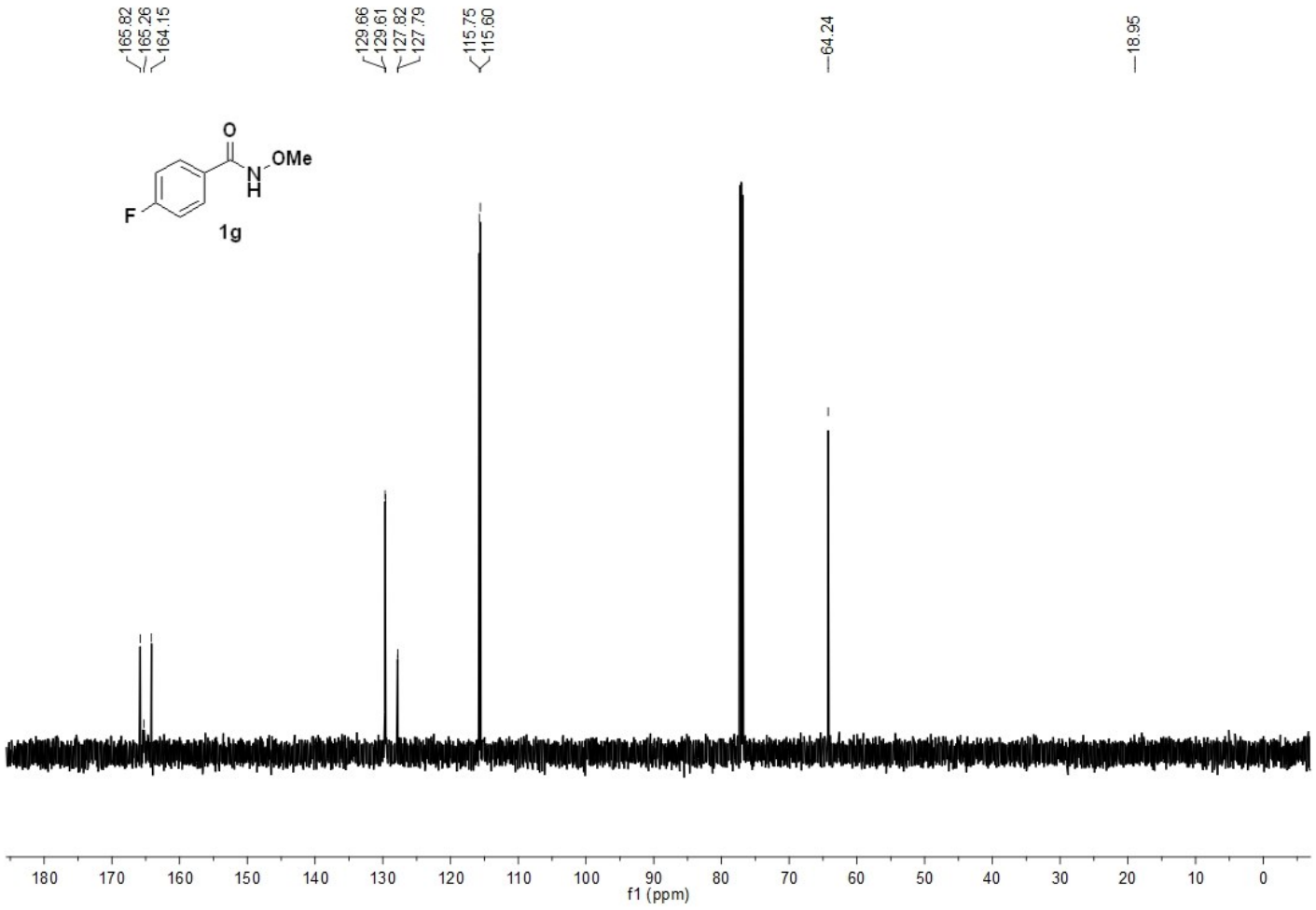
H
OMe
F
1g
0.56
2.06
2.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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127.79
115.75
115.60
64.24
18.95
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OMe
H
F
1g
180
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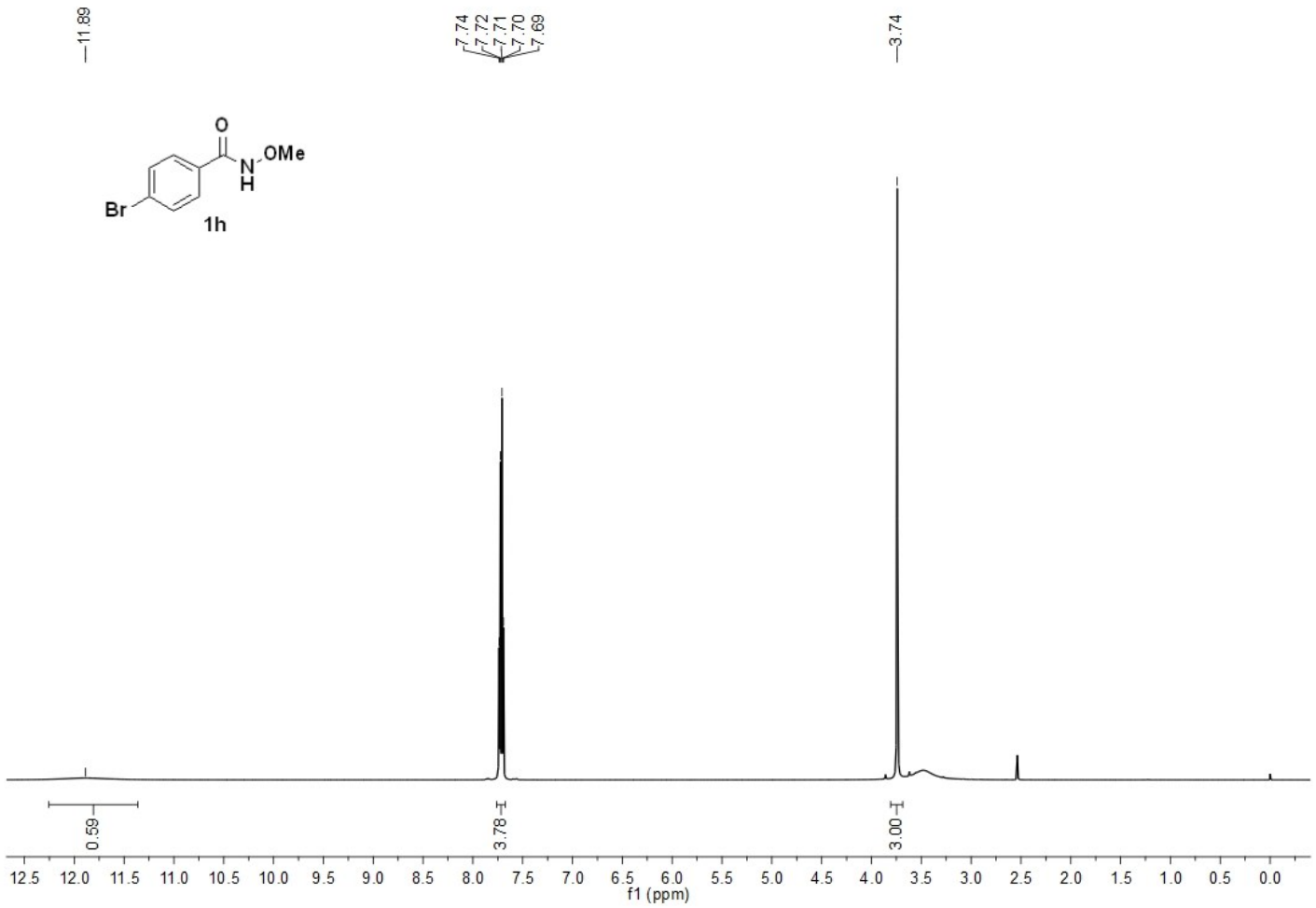
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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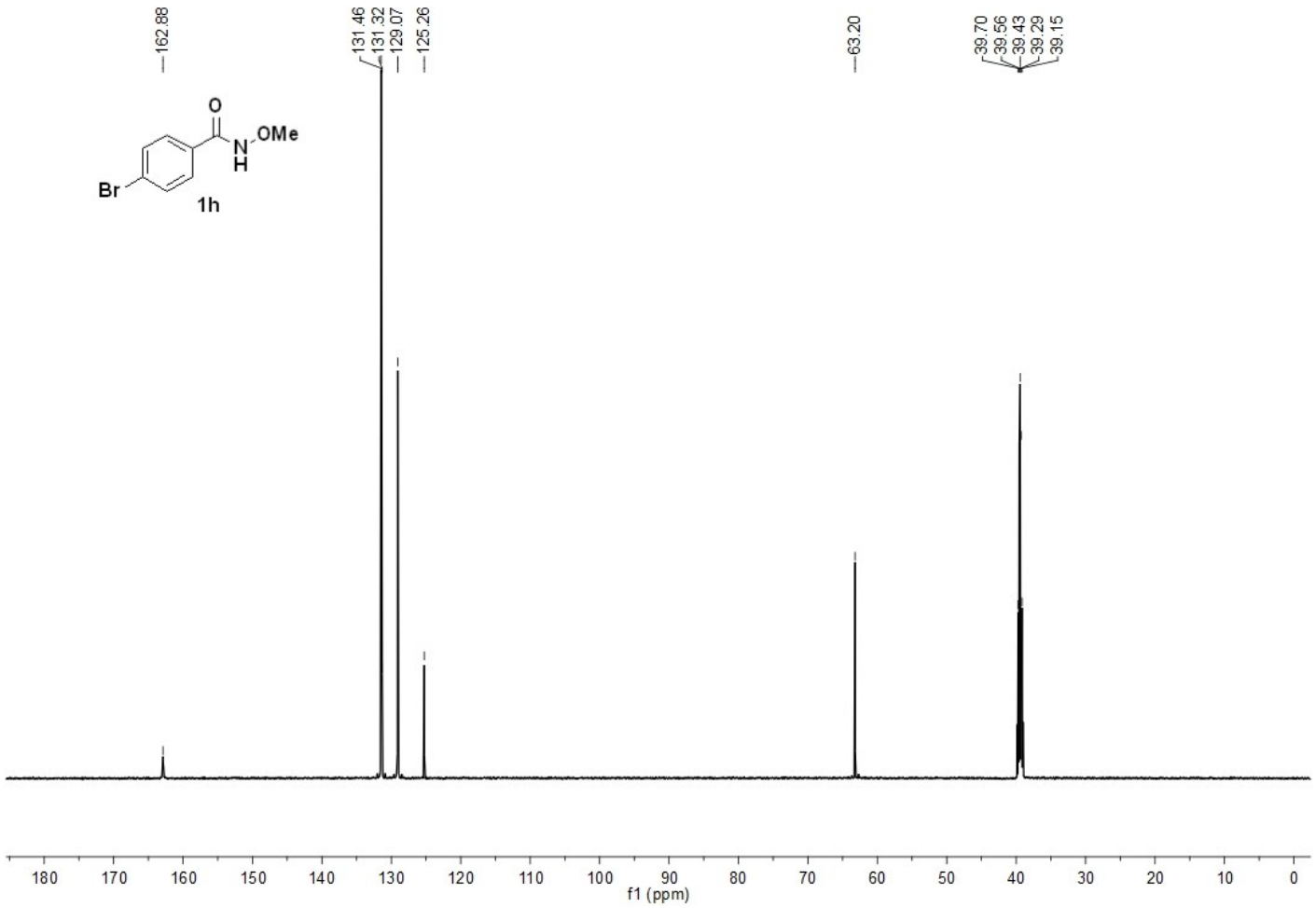
7.72
7.71
7.70
7.69
3.74
O
N
H
OMe
Br
1h
0.59
3.78
3.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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39.29
39.15
O
N
H
OMe
Br
1h
180
170
160
150
140
130
120
110
100
90
80
70
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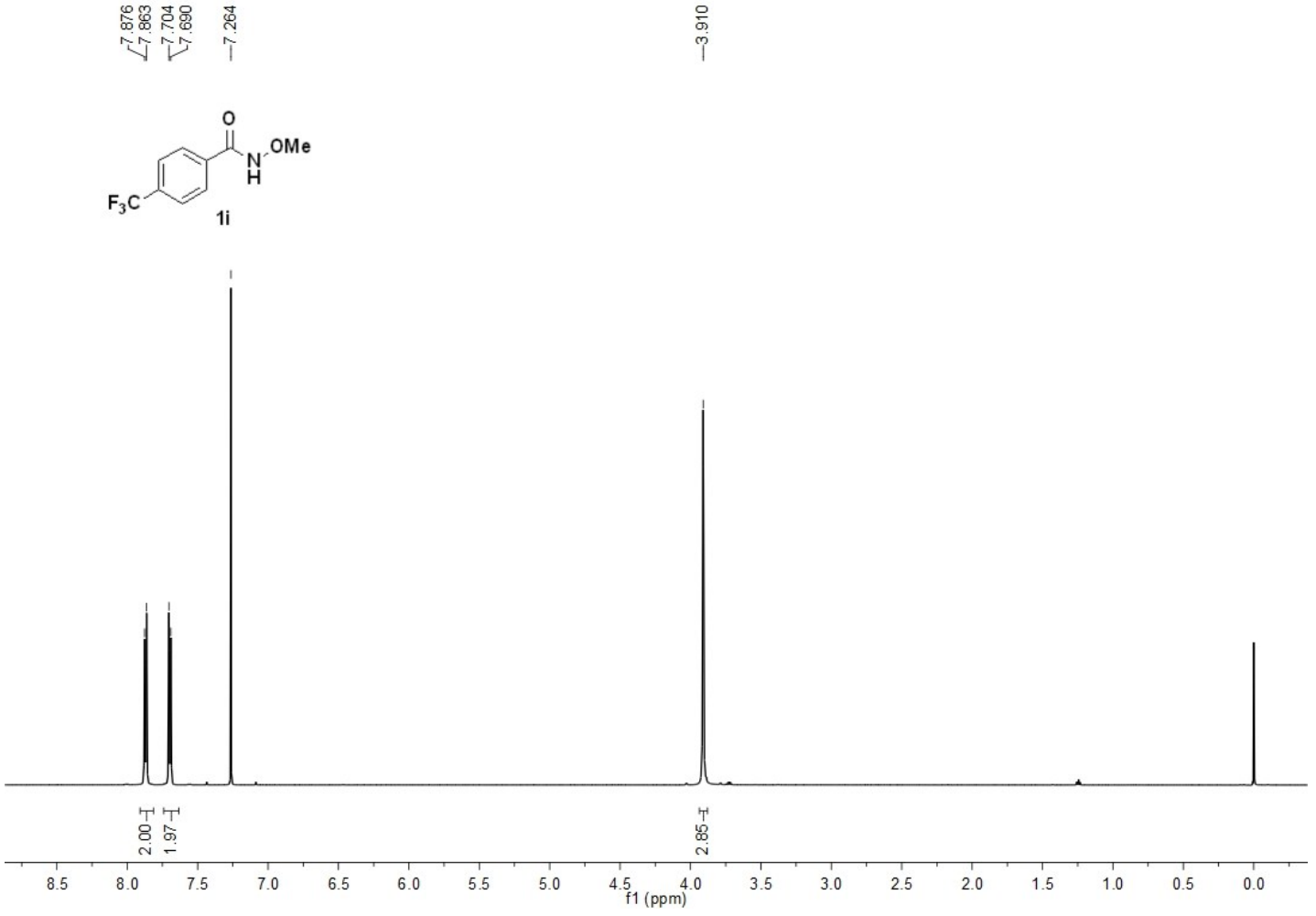
50
40
30
20
10
0
f1 (ppm)


7.876
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3.910
O
N
H
OMe
F3C
1i
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1.97
2.85
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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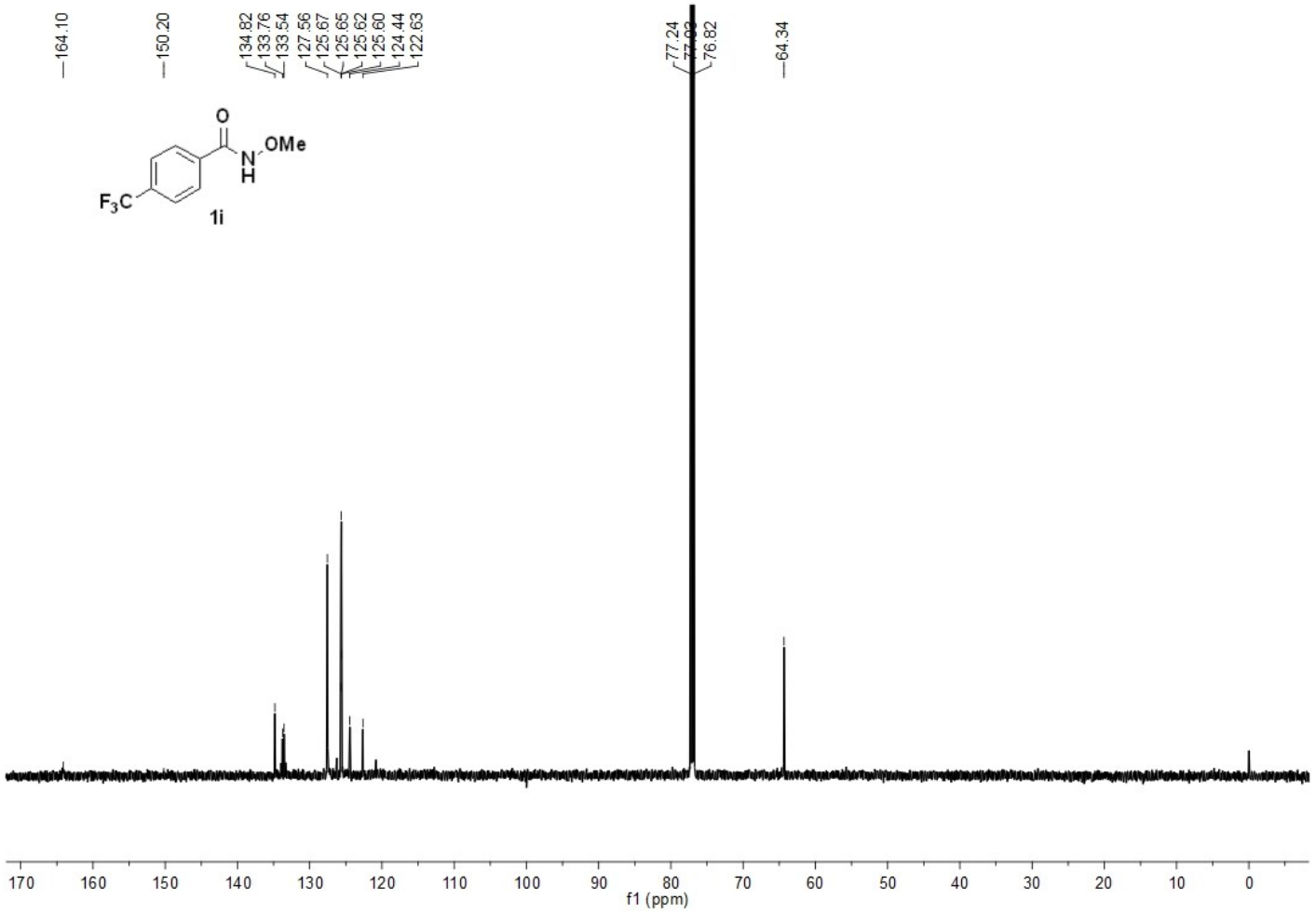
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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150.20
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133.76
133.54
127.56
125.67
125.65
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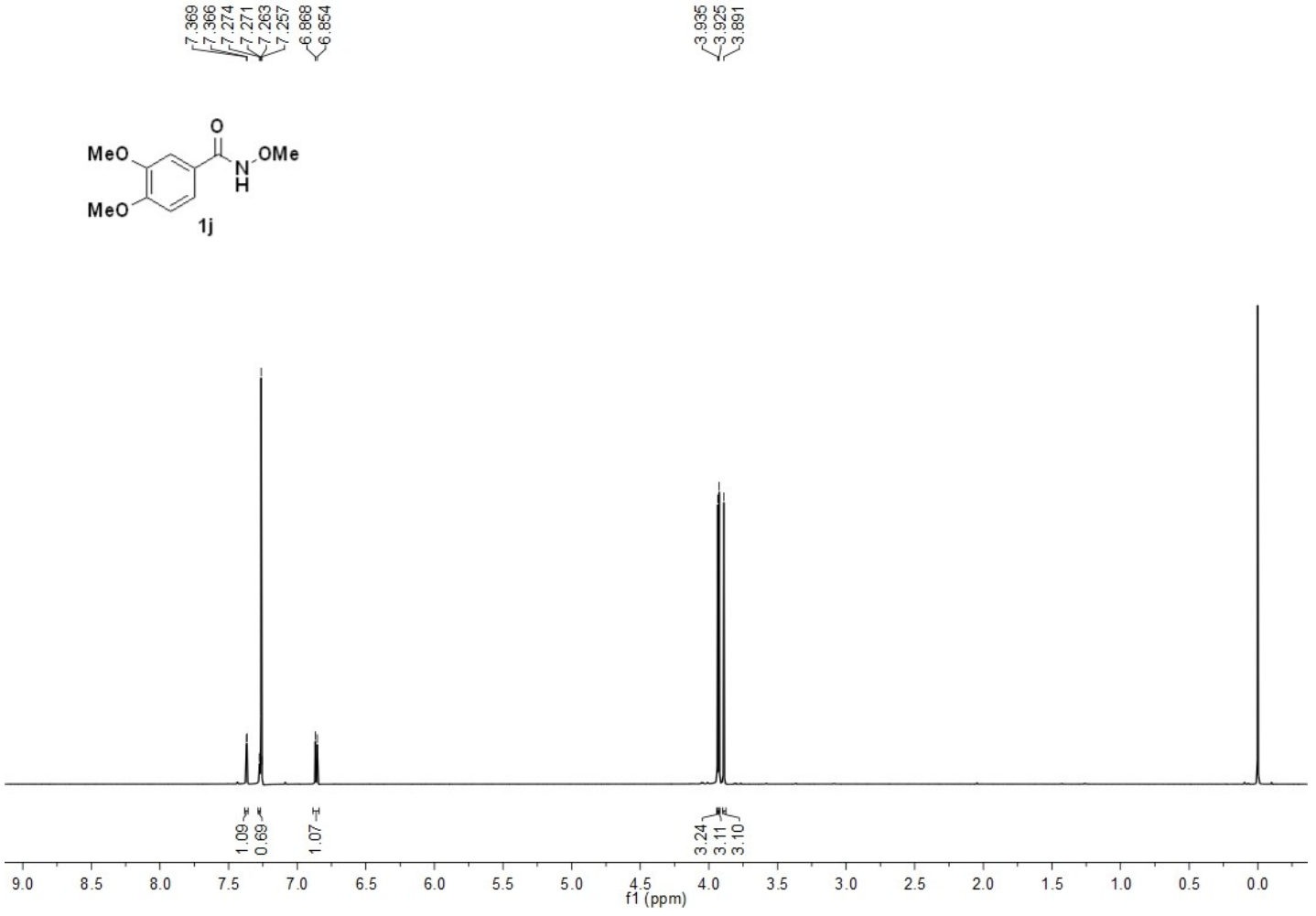
125.60
124.44
122.63
77.24
77.03
76.82
64.34
O
N
H
OMe
F3C
1i
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.854
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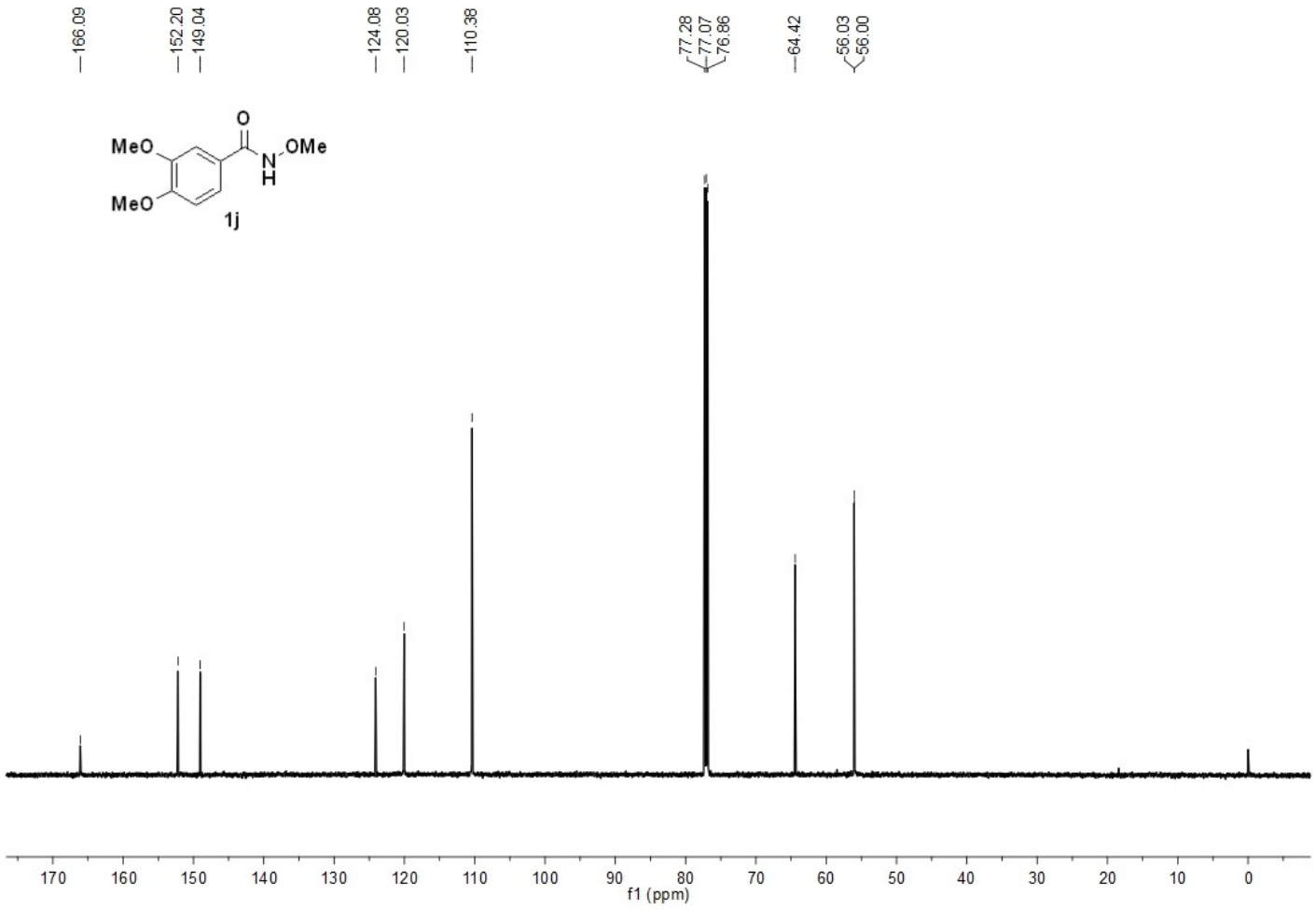
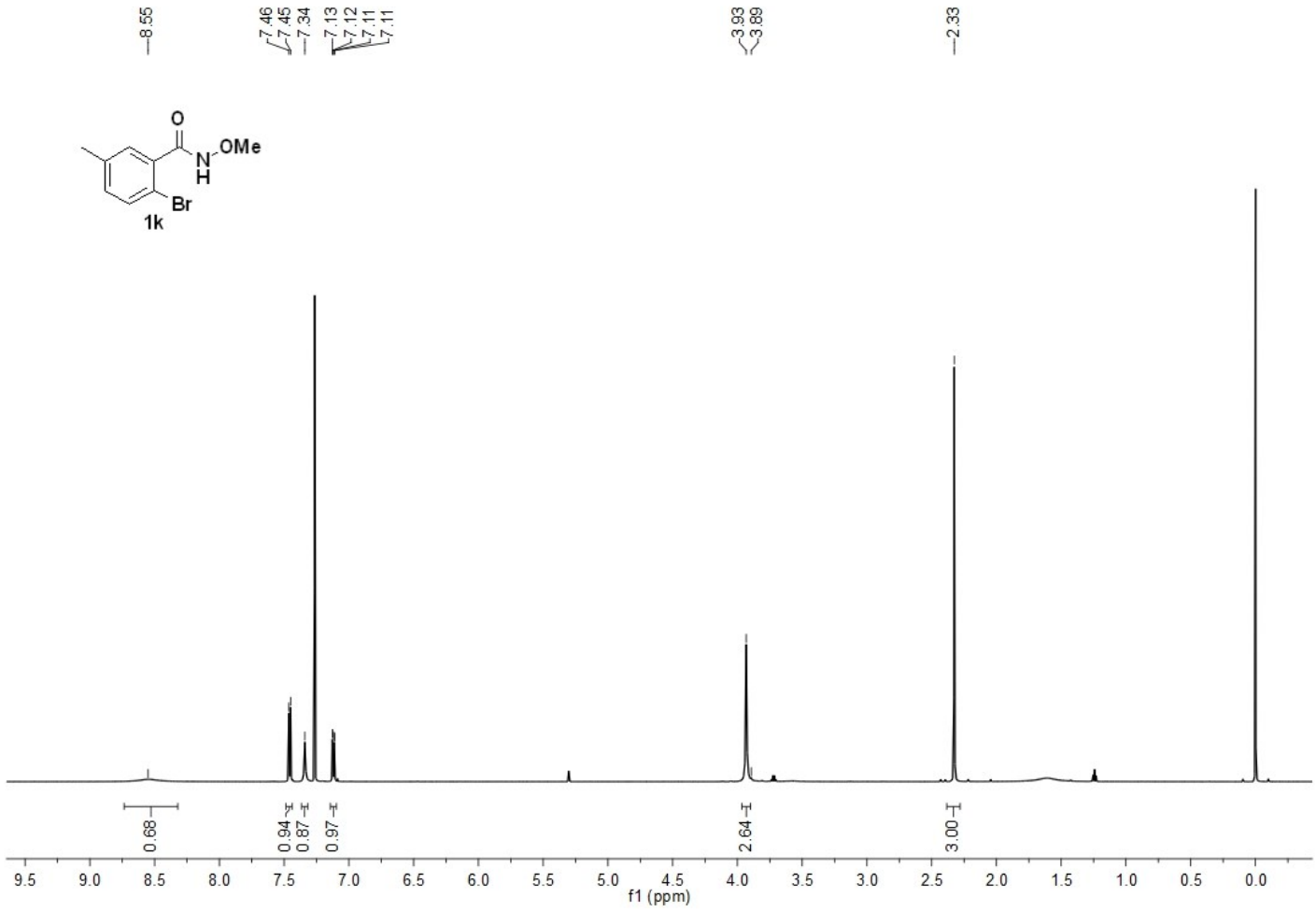
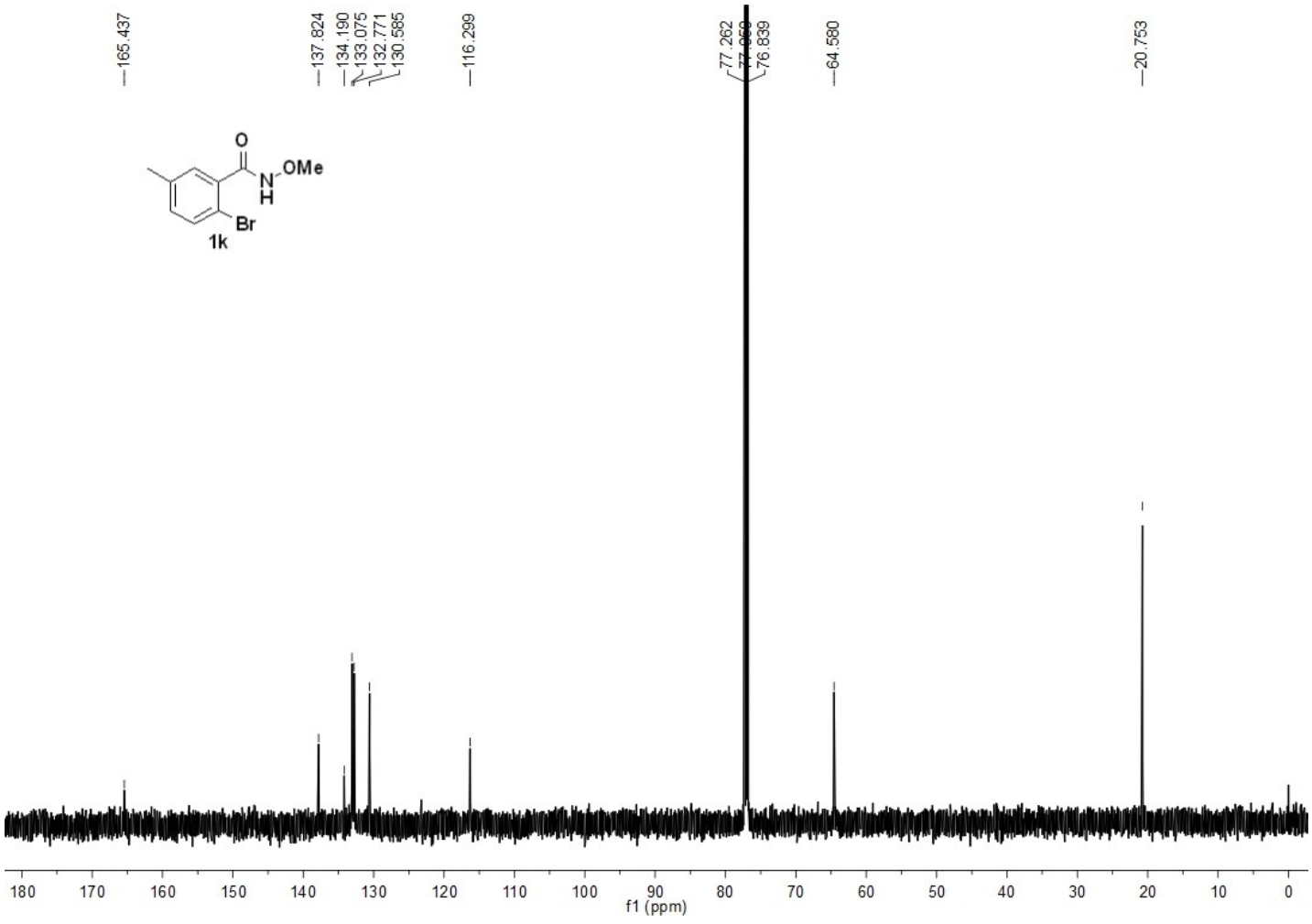
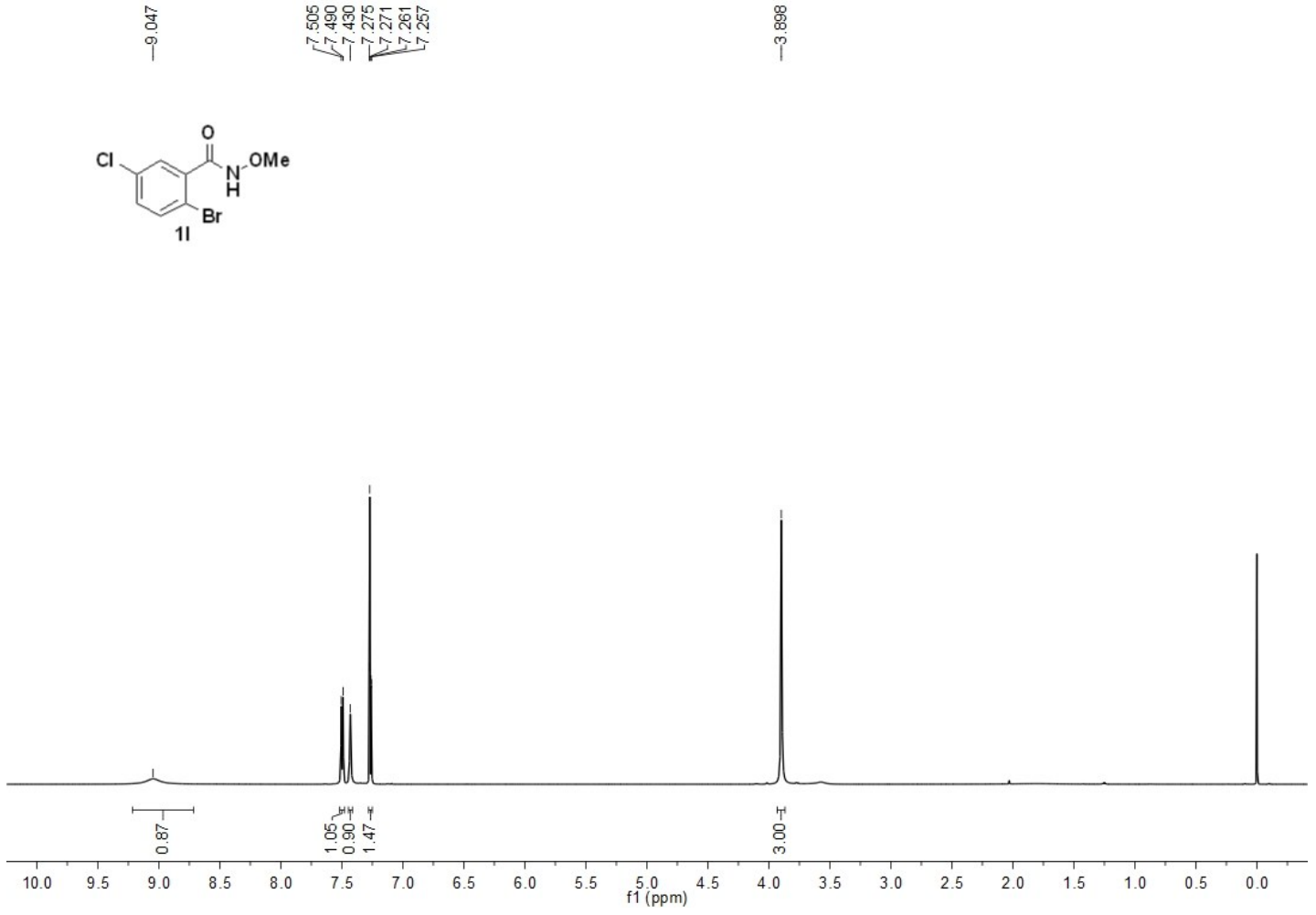
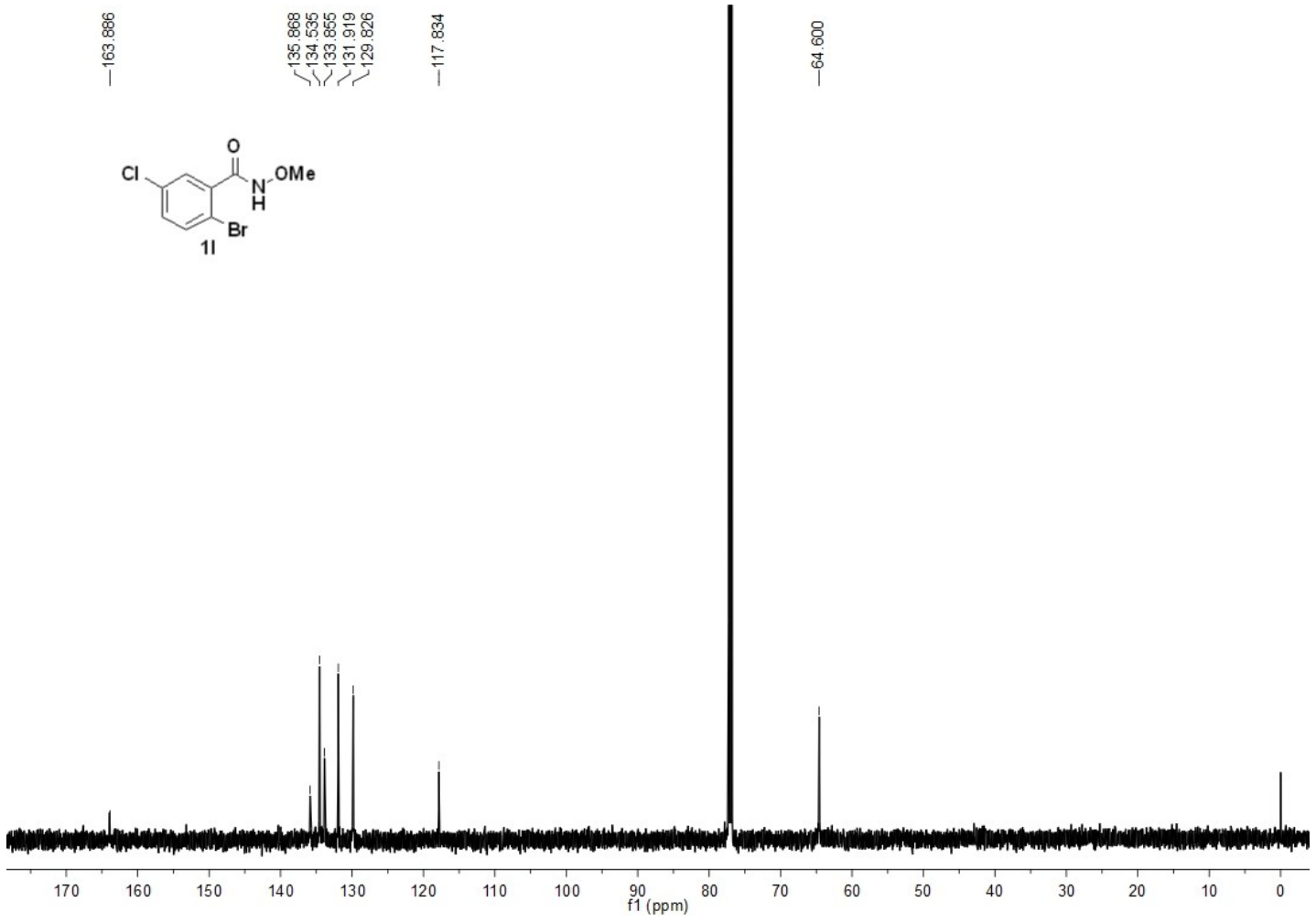
3.925
3.891
MeO
MeO
O
N
H
OMe
1j
1.09
0.69
1.07
3.24
3.11
3.10
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

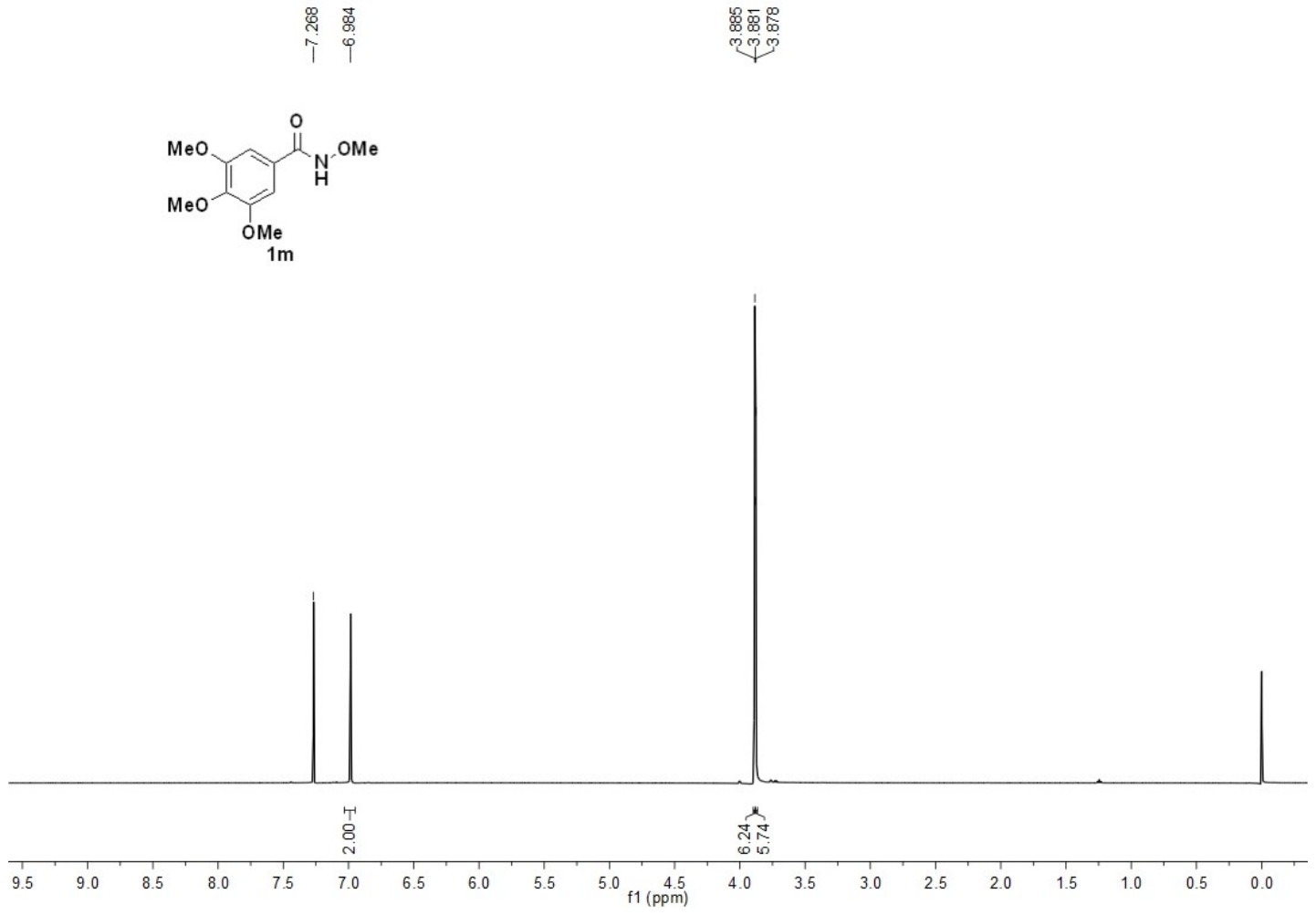

166.09
152.20
149.04
124.08
120.03
110.38
77.28
77.07
76.86
64.42
56.03
56.00
MeO
MeO
O
N
H
OMe
1j
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

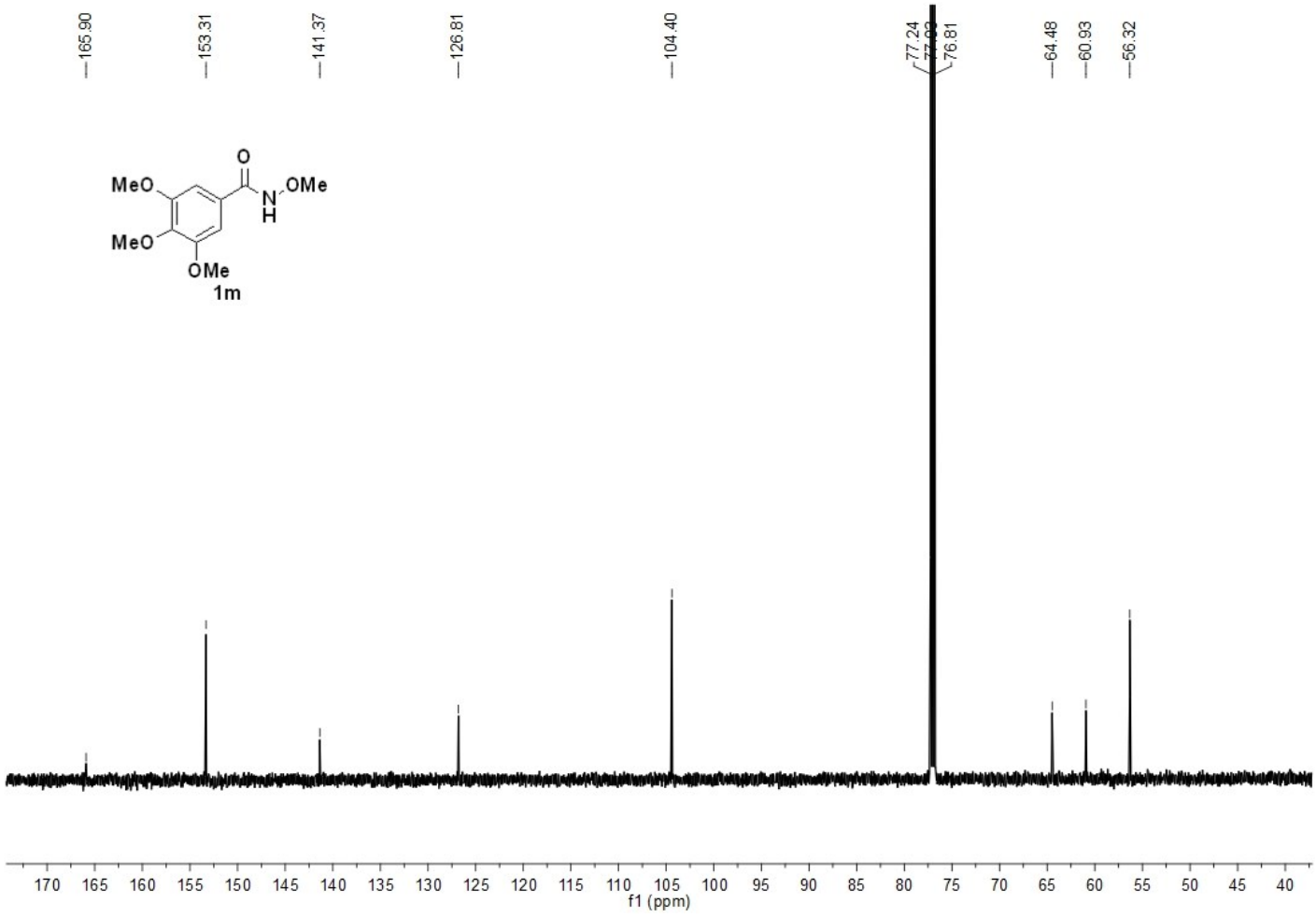

8.55
7.46
7.45
7.34
7.13
7.12
7.11
7.11
3.93
3.89
2.33
O
N
H
OMe
Br
1k
0.68
0.94
0.87
0.97
2.64
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

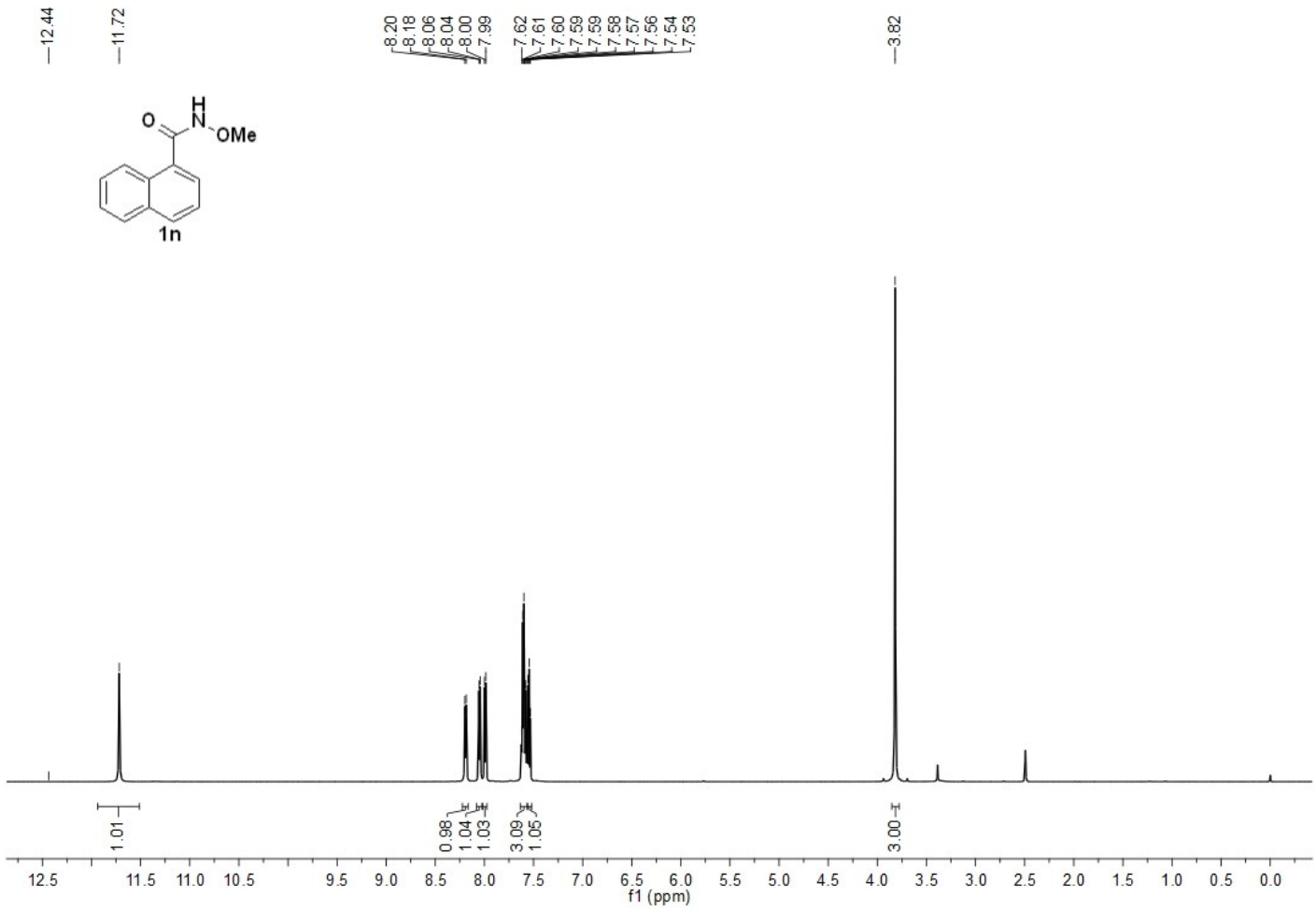

165.437
137.824
134.190
133.075
132.771
130.585
116.299
77.262
76.839
64.580
20.753
O
N
H
OMe
Br
1k
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

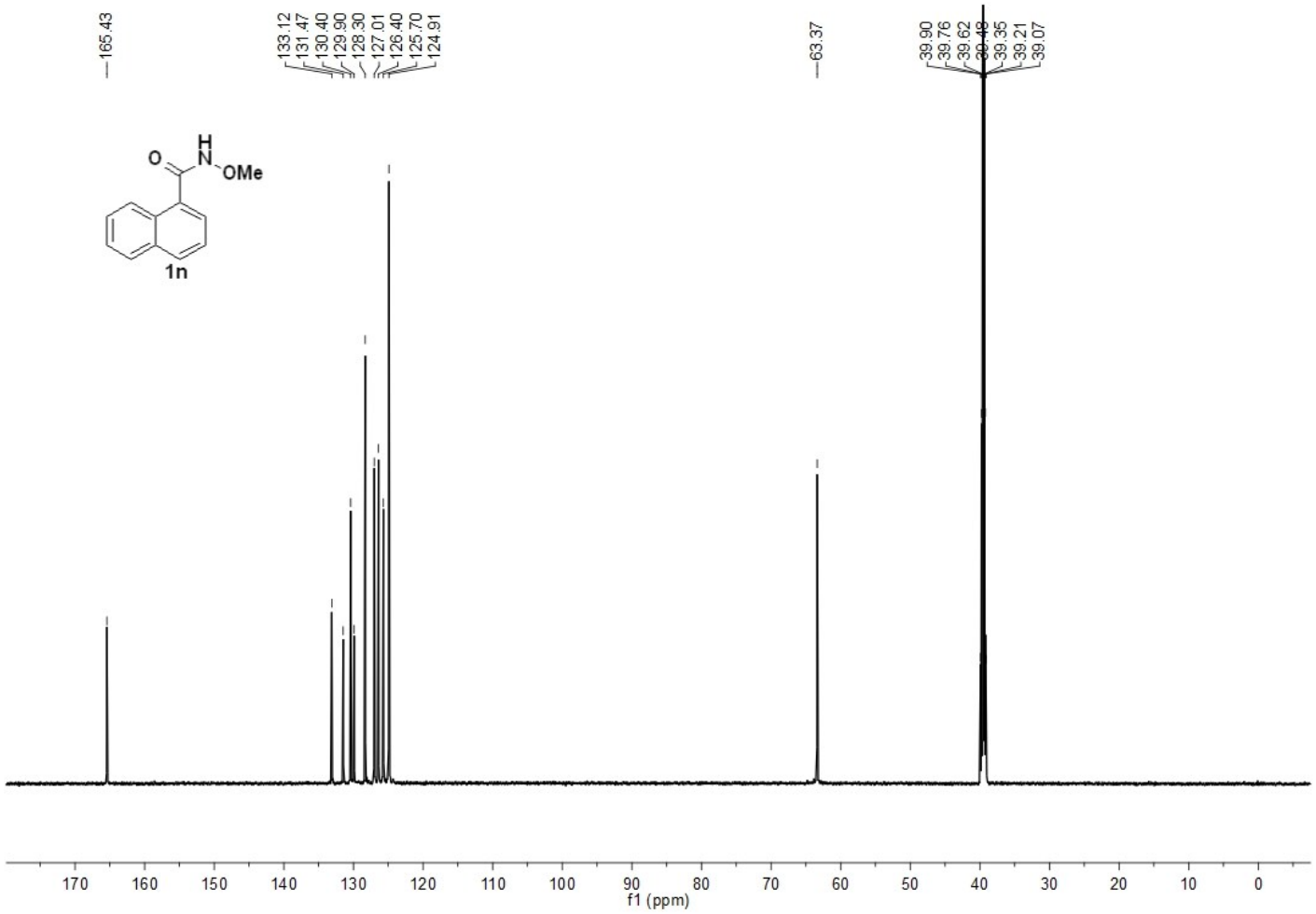

9.047
7.505
7.490
7.430
7.275
7.271
7.261
7.257
3.898
Cl
O
N
H
OMe
Br
1l
0.87
1.05
0.90
1.47
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

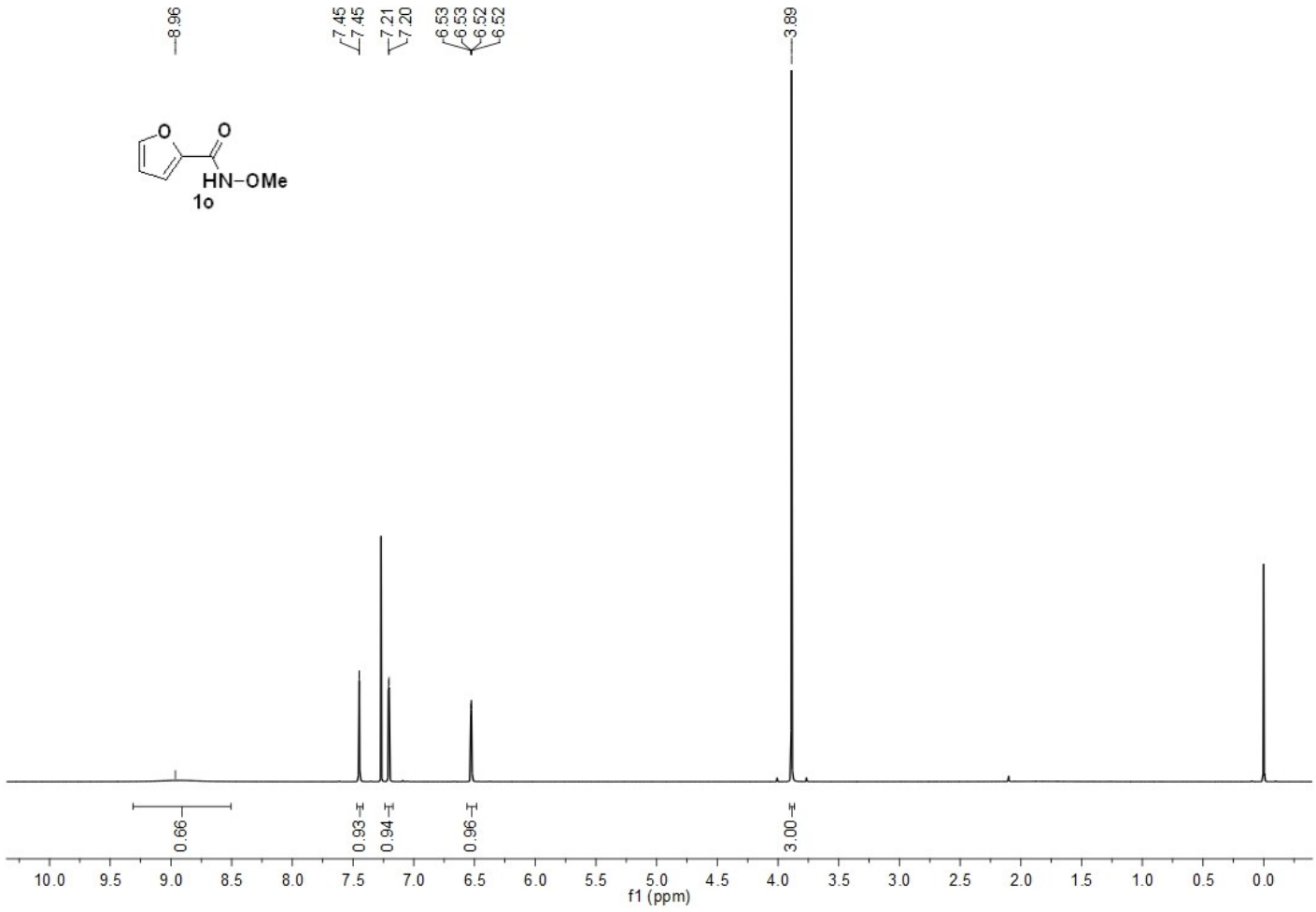

163.886
135.868
134.535
133.855
131.919
129.826
117.834
64.600
Cl
O
N
H
OMe
Br
1l
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

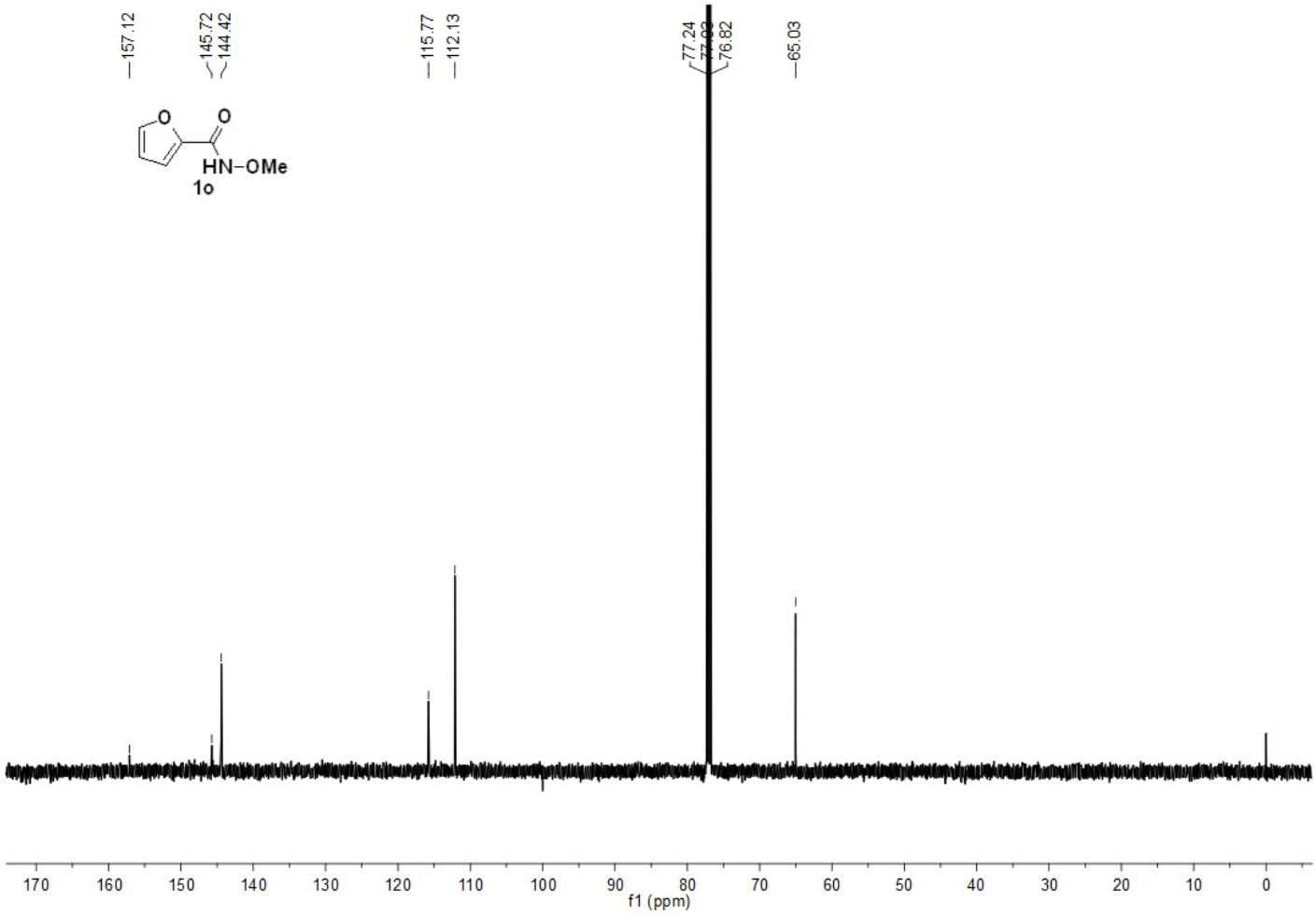

7.268
6.984
3.885
3.881
3.878
MeO
MeO
OMe
O
N
H
OMe
1m
2.00
6.24
5.74
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

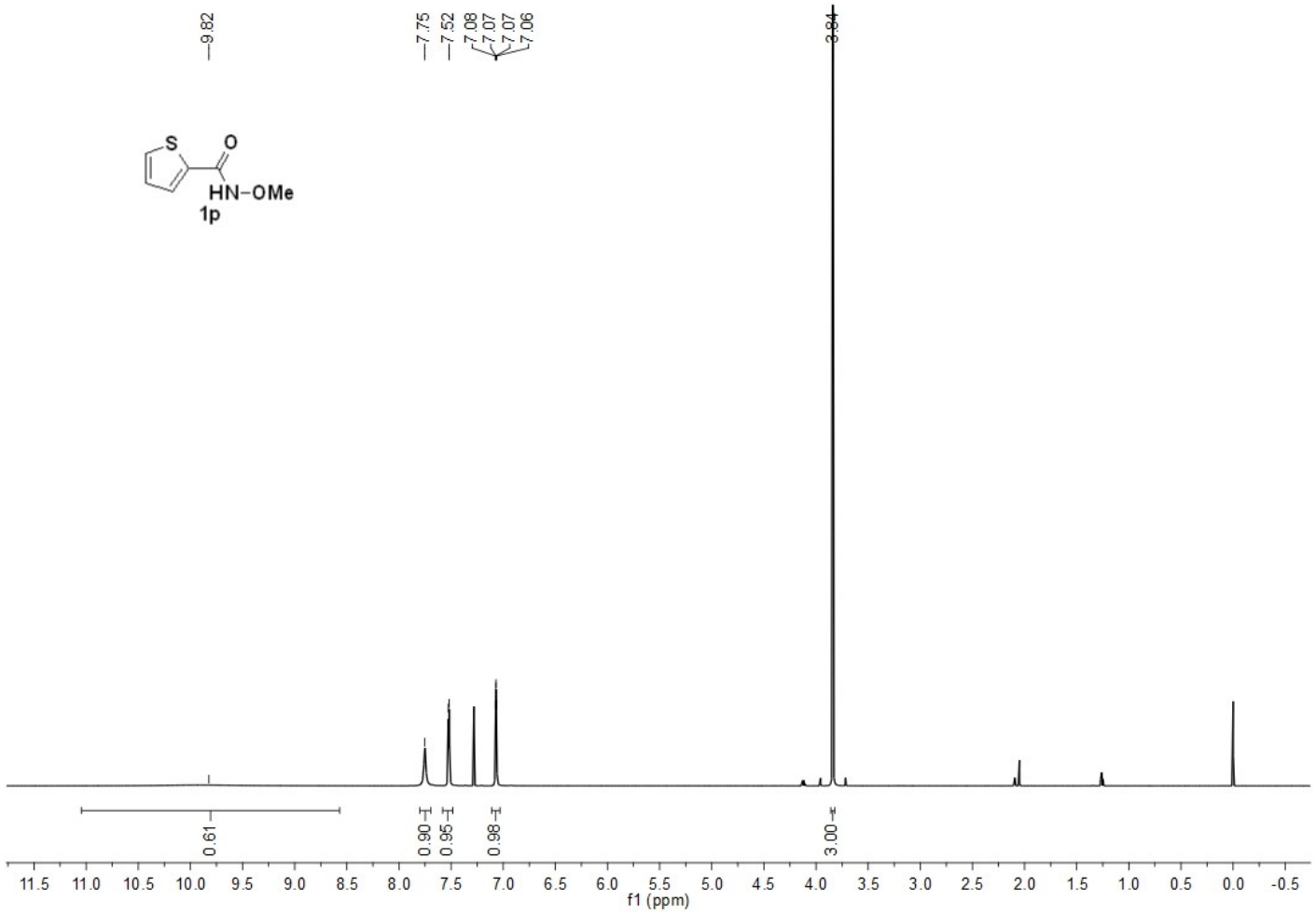

165.90
153.31
141.37
126.81
104.40
77.24
77.03
76.81
64.48
60.93
56.32
MeO
MeO
OMe
O
N
H
OMe
1m
170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40
f1 (ppm)

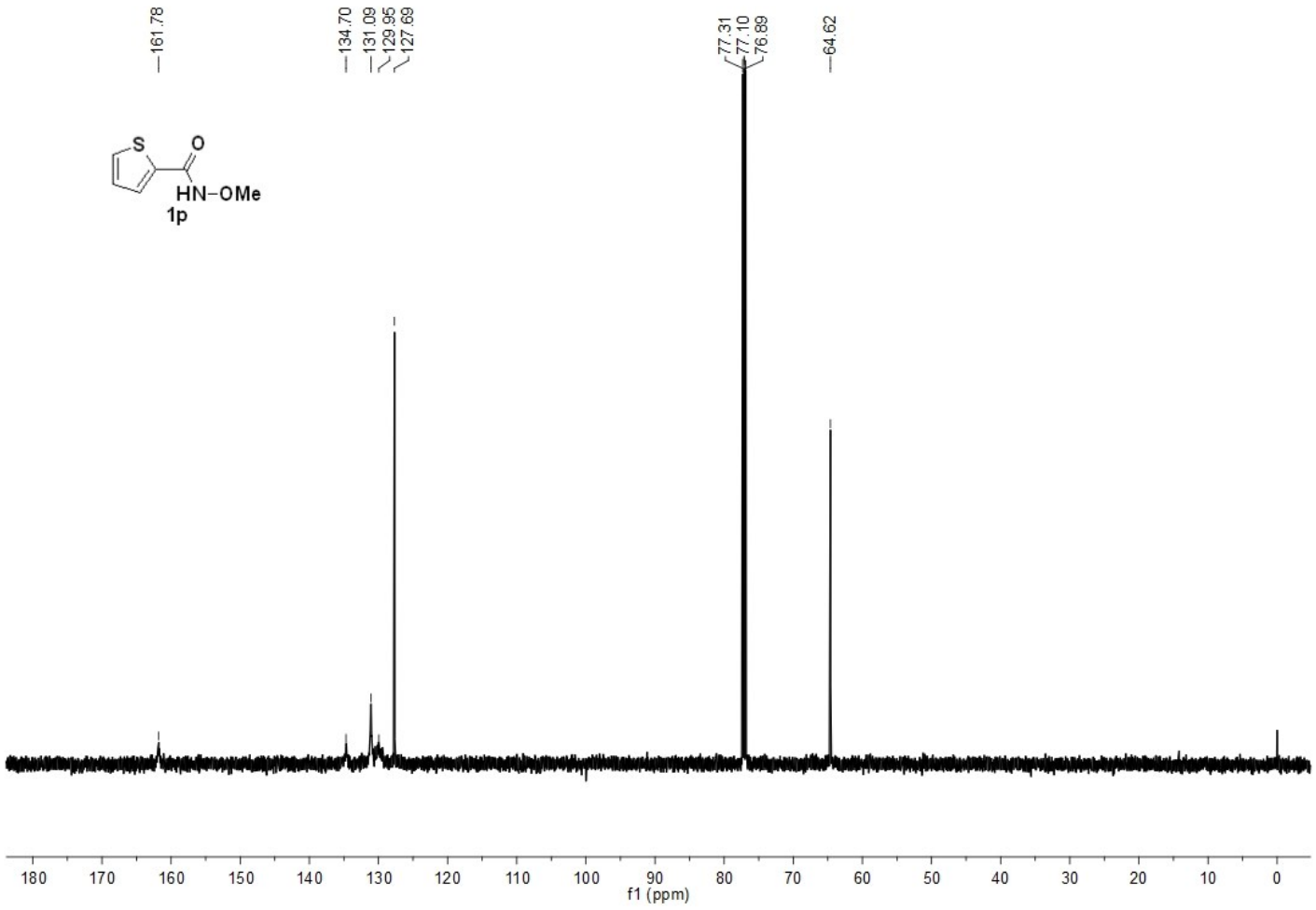

12.44
11.72
8.20
8.18
8.06
8.04
8.00
7.99
7.62
7.61
7.60
7.59
7.59
7.58
7.57
7.56
7.54
7.53
3.82
O
H
N
OMe
1n
1.01
0.98
1.04
1.03
3.09
1.05
3.00
12.5
11.5
11.0
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

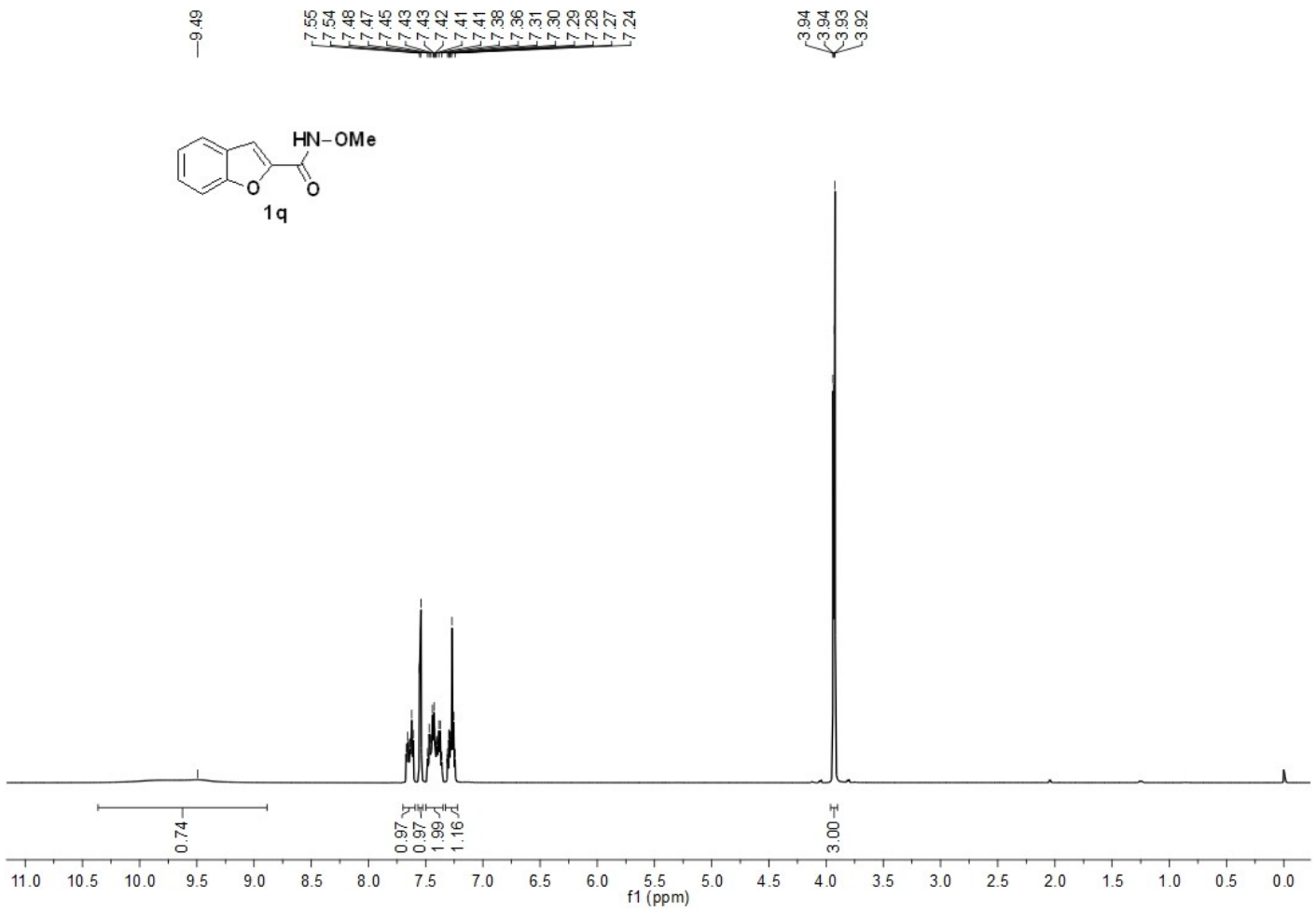

165.43
133.12
131.47
130.40
129.90
128.30
127.01
126.40
125.70
124.91
63.37
39.90
39.76
39.62
39.48
39.35
39.21
39.07
O
H
N
OMe
1n
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

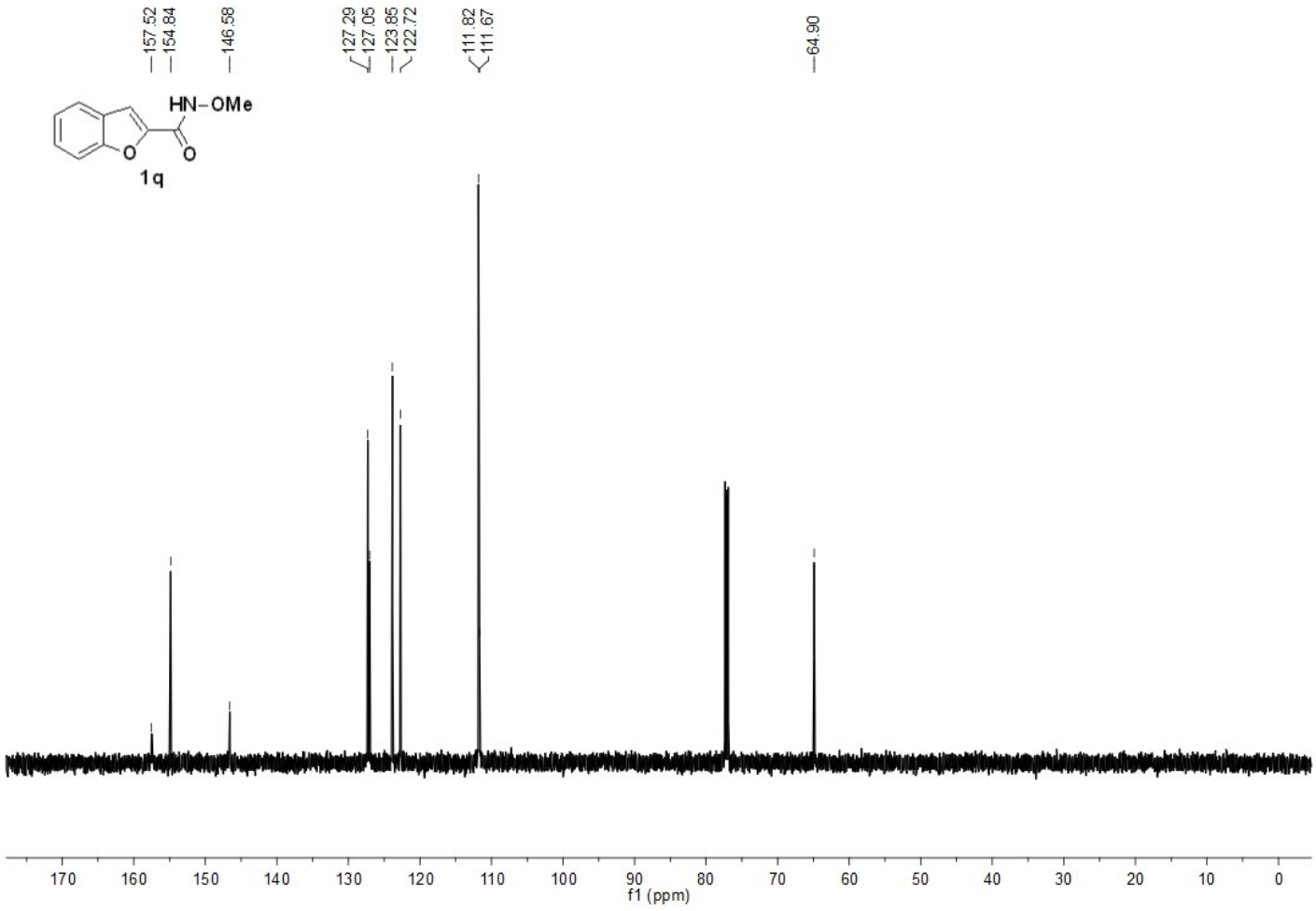

8.96
7.45
7.45
7.21
7.20
6.53
6.53
6.52
6.52
3.89
O
O
HN–OMe
1o
0.66
0.93
0.94
0.96
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

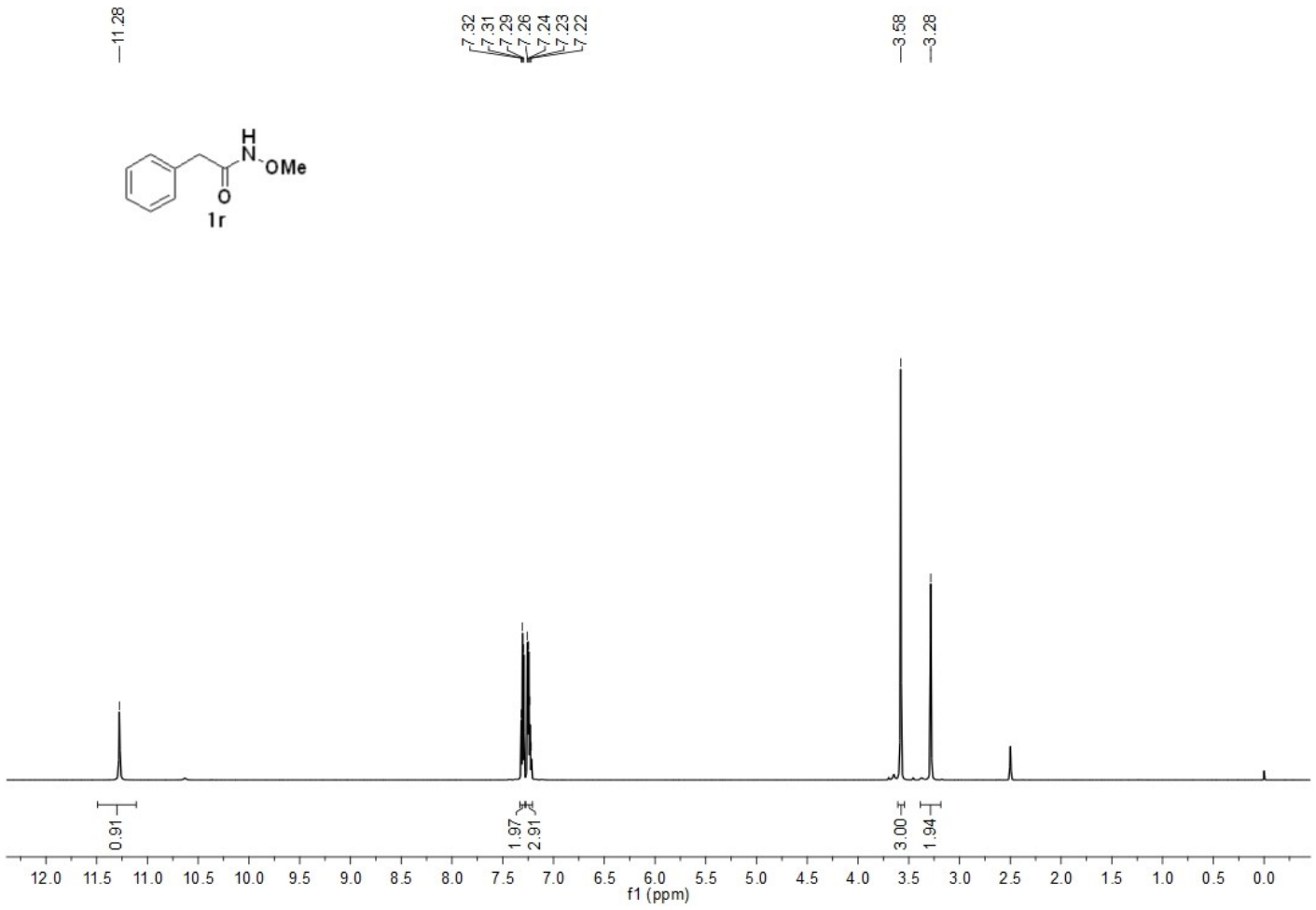

157.12
145.72
144.42
115.77
112.13
77.24
77.03
76.82
65.03
O
O
HN–OMe
1o
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

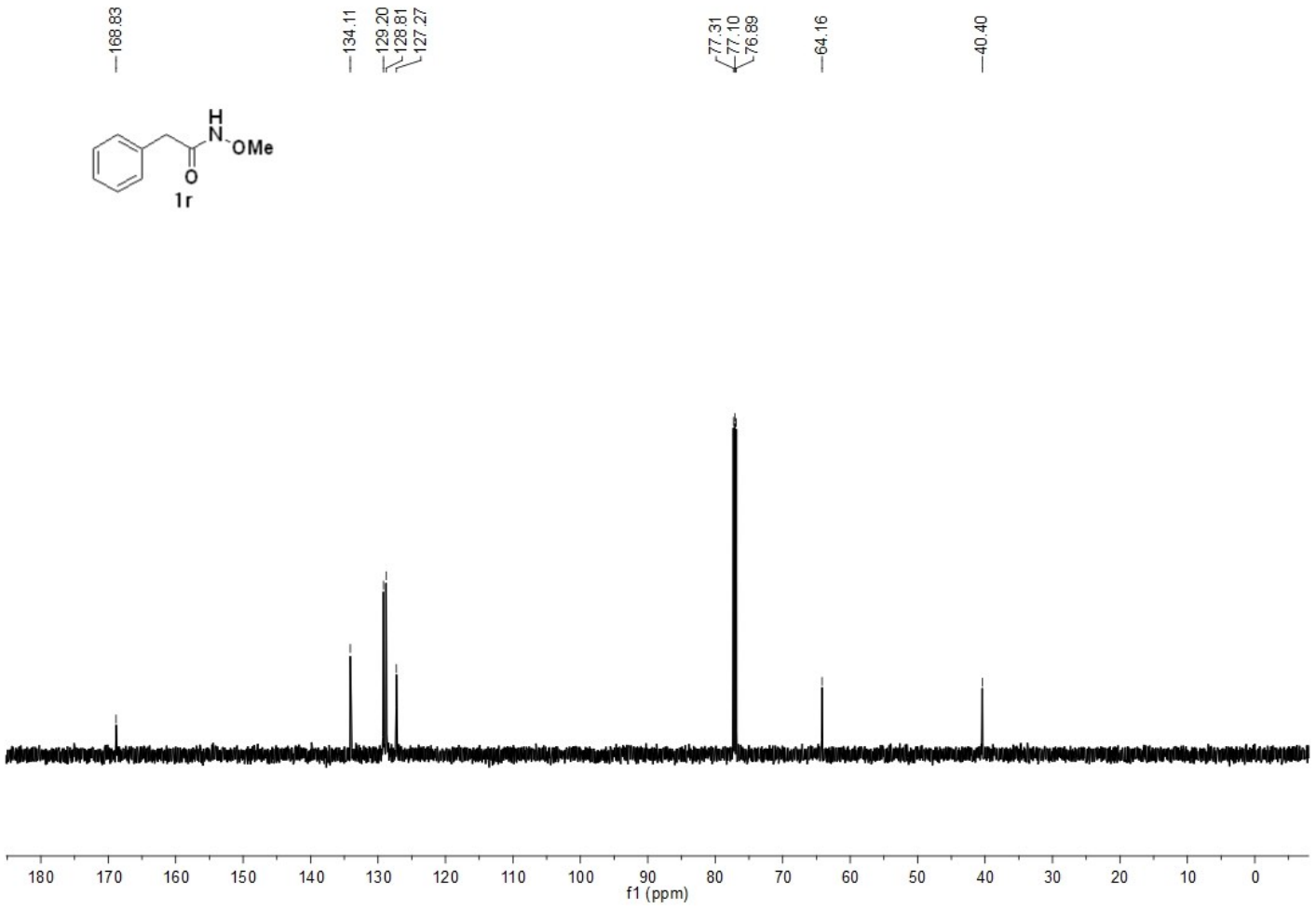

9.82
7.75
7.52
7.08
7.07
7.07
7.06
3.84
S
O
HN–OMe
1p
0.61
0.90
0.95
0.98
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

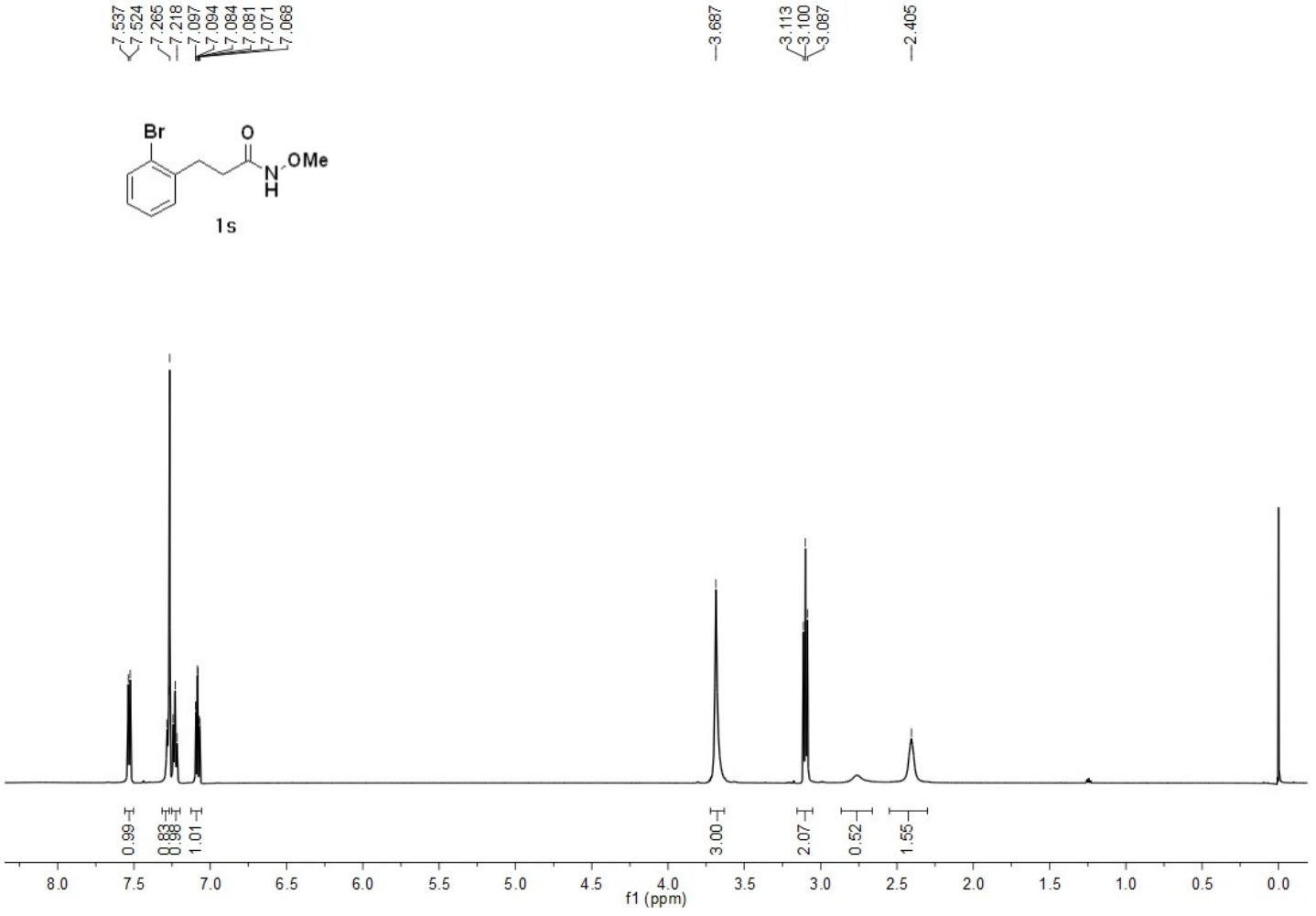

161.78
134.70
131.09
129.95
127.69
77.31
77.10
76.89
64.62
S
O
HN–OMe
1p
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

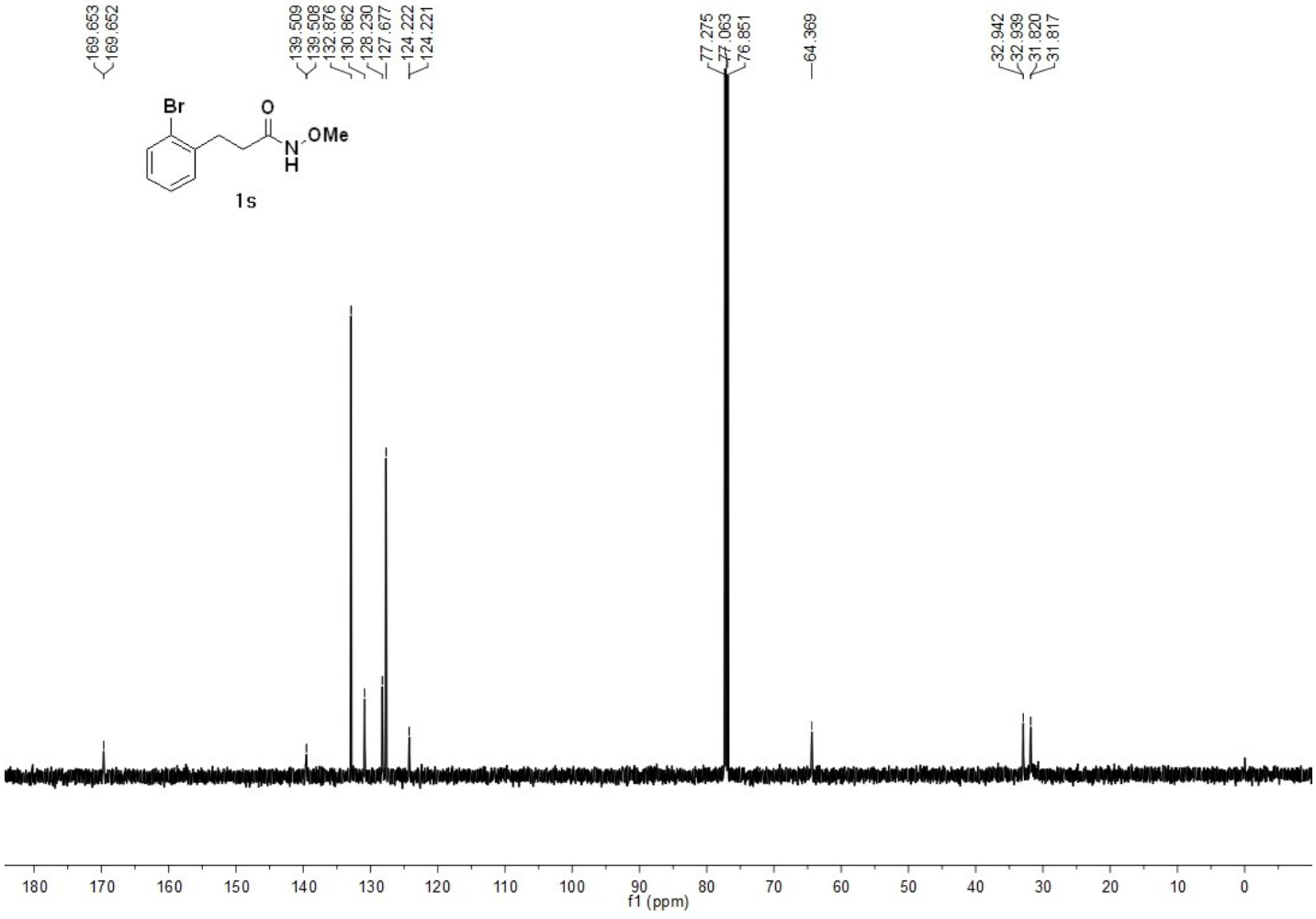

9.49
7.55
7.54
7.48
7.47
7.45
7.43
7.43
7.42
7.41
7.41
7.38
7.36
7.31
7.30
7.29
7.28
7.27
7.24
3.94
3.94
3.93
3.92
HN–OMe
O
O
1q
0.74
0.97
0.97
1.99
1.16
3.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

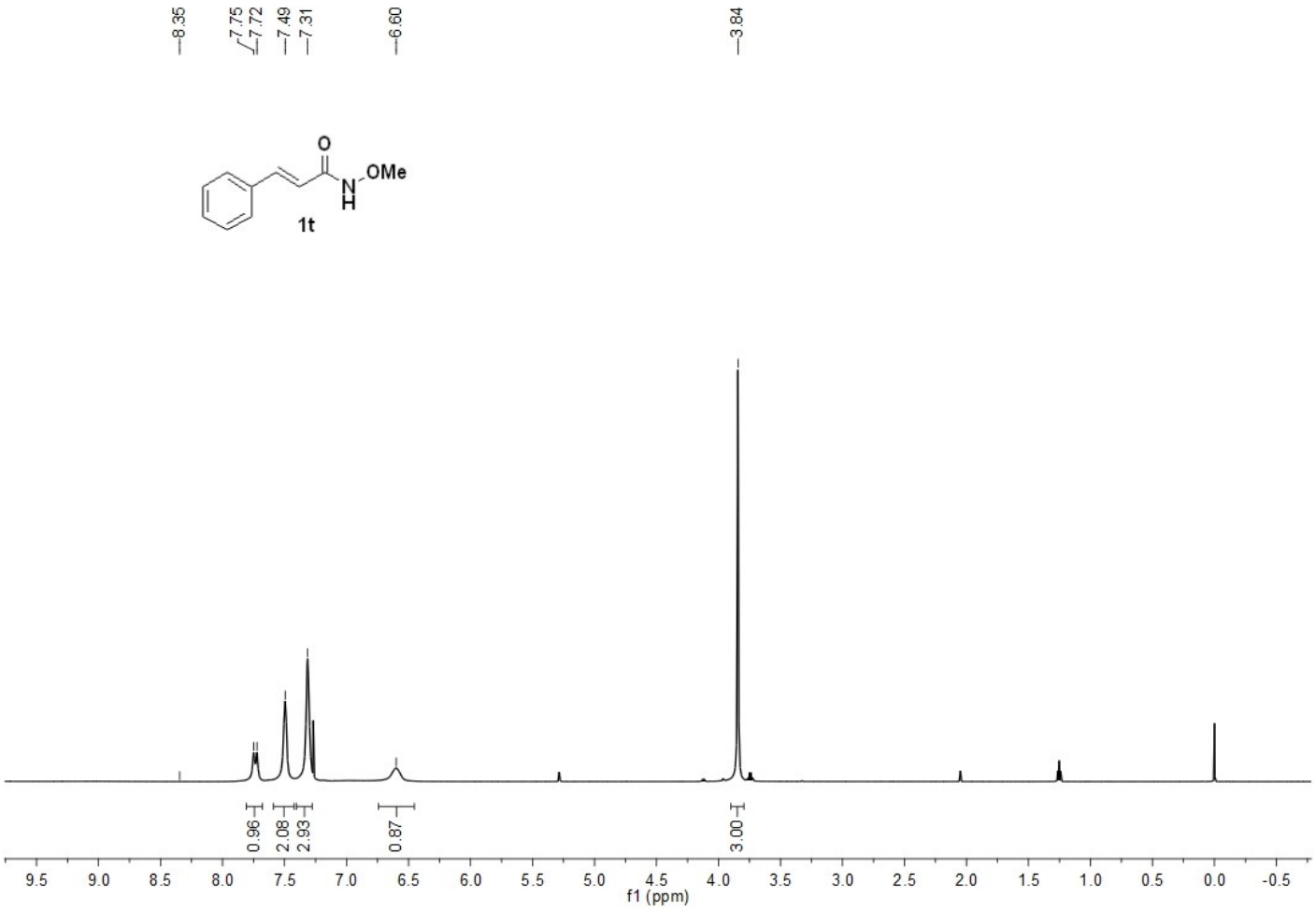

157.52
154.84
146.58
127.29
127.05
123.85
122.72
111.82
111.67
64.90
HN–OMe
O
O
1q
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

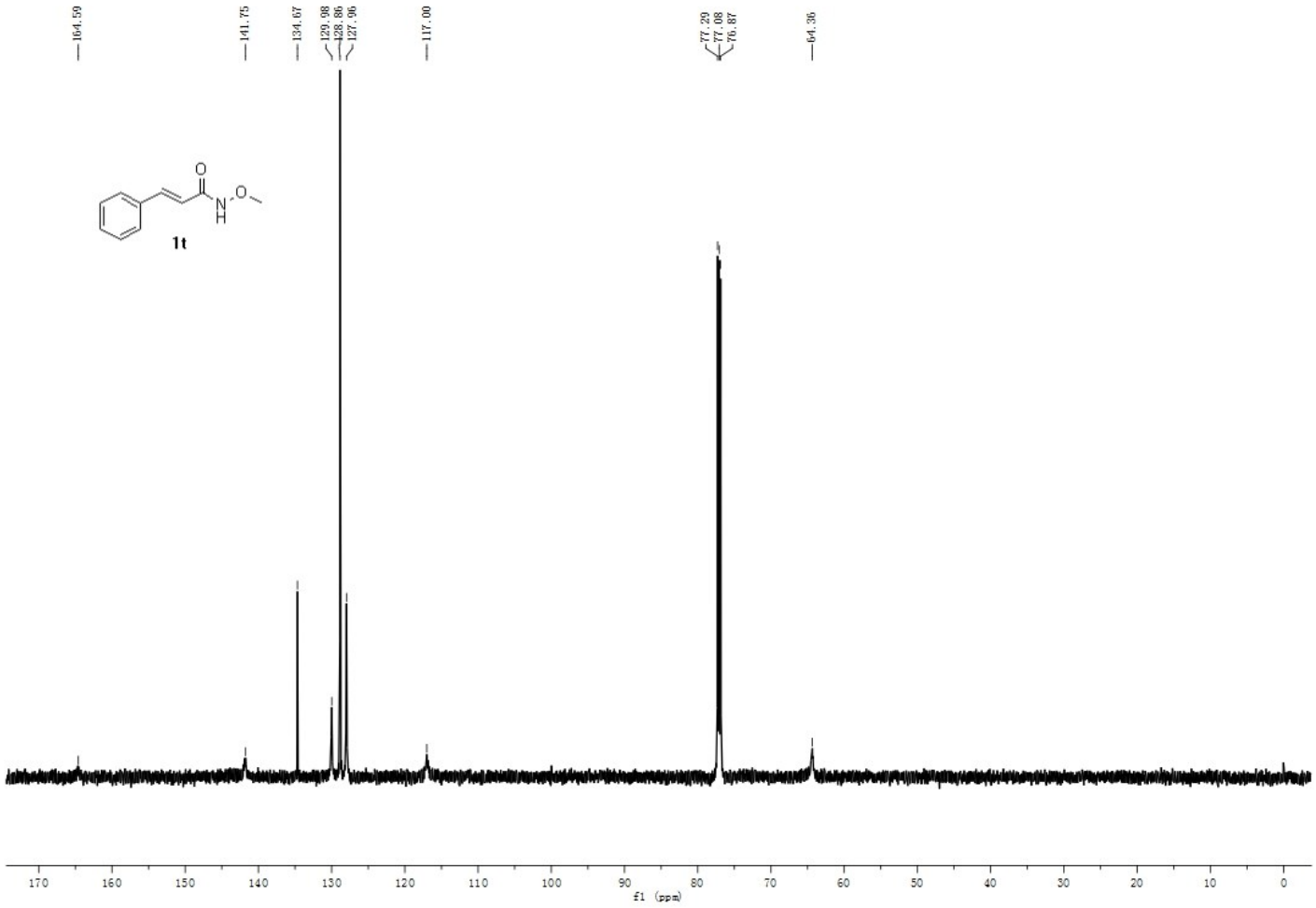

11.28
7.32
7.31
7.29
7.26
7.24
7.23
7.22
3.58
3.28
H
N
OMe
O
1r
0.91
1.97
2.91
3.00
1.94
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

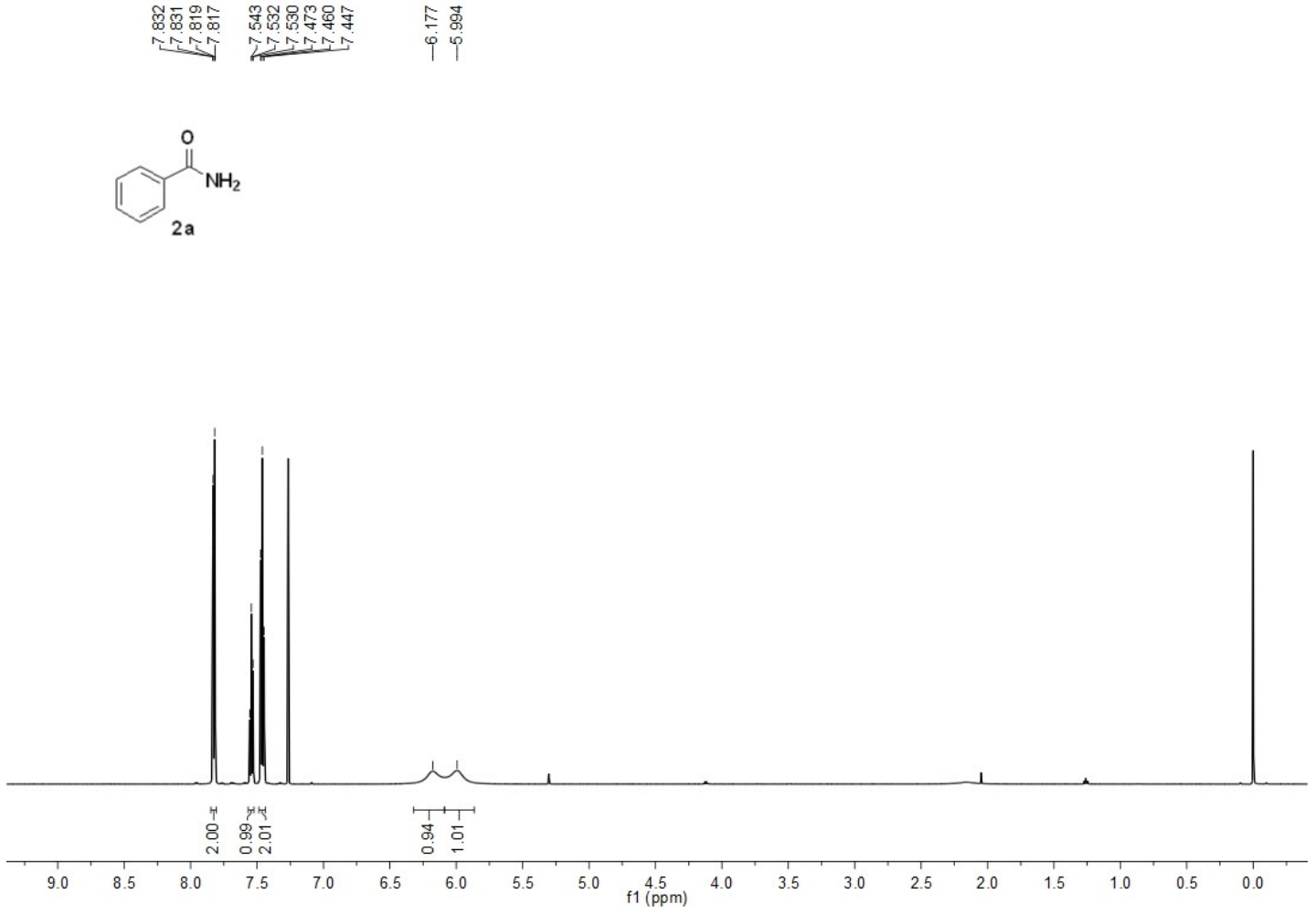

168.83
134.11
129.20
128.81
127.27
77.31
77.10
76.89
64.16
40.40
H
N
OMe
O
1r
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

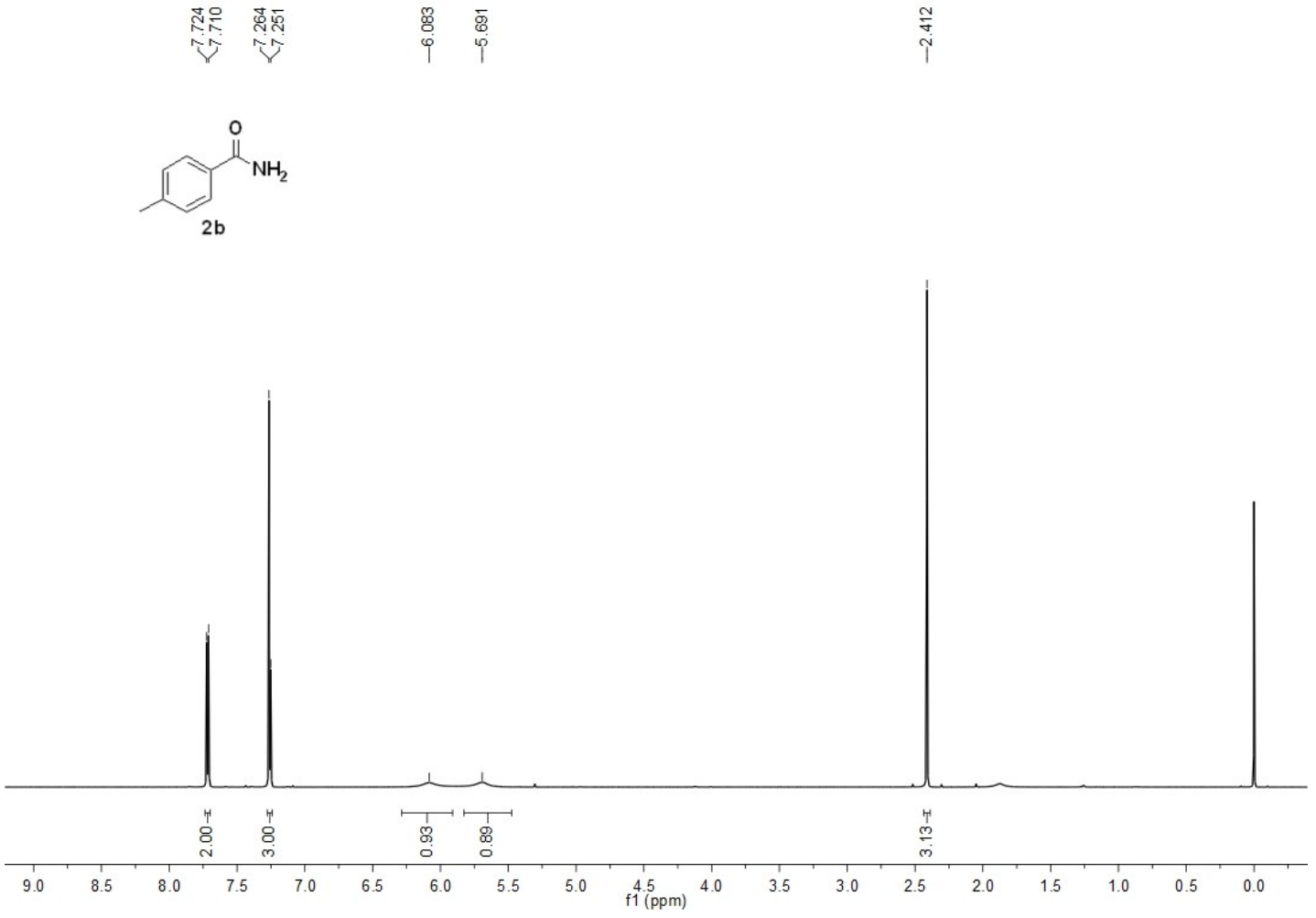

7.537
7.524
7.265
7.218
7.097
7.094
7.084
7.081
7.071
7.068
3.687
3.113
3.100
3.087
2.405
Br
O
N
H
OMe
1s
0.99
0.83
0.98
1.01
3.00
2.07
0.52
1.55
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

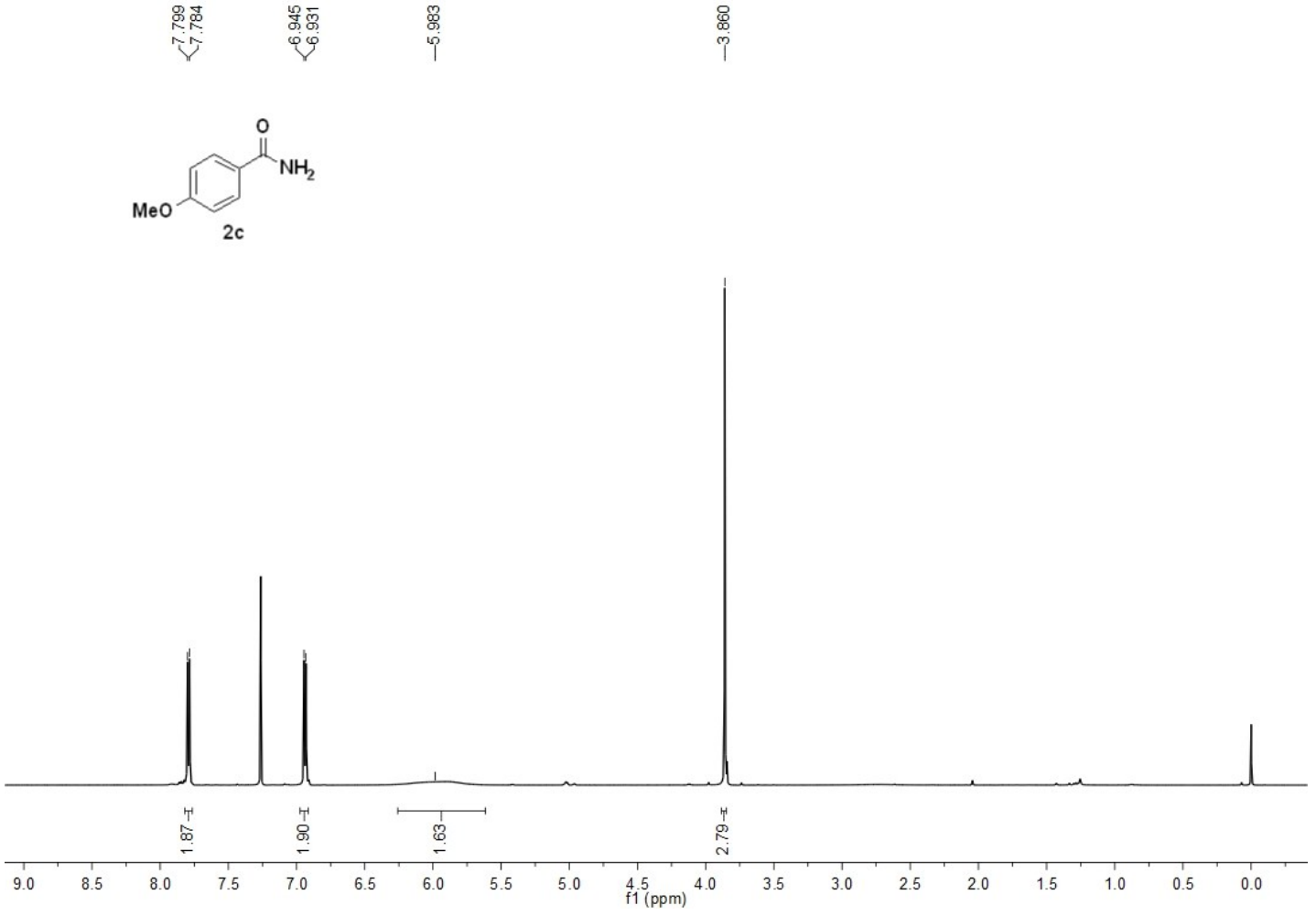

169.653
169.652
139.509
139.508
132.876
130.862
128.230
127.677
124.222
124.221
77.275
77.063
76.851
64.369
32.942
32.939
31.820
31.817
Br
O
N
H
OMe
1s
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

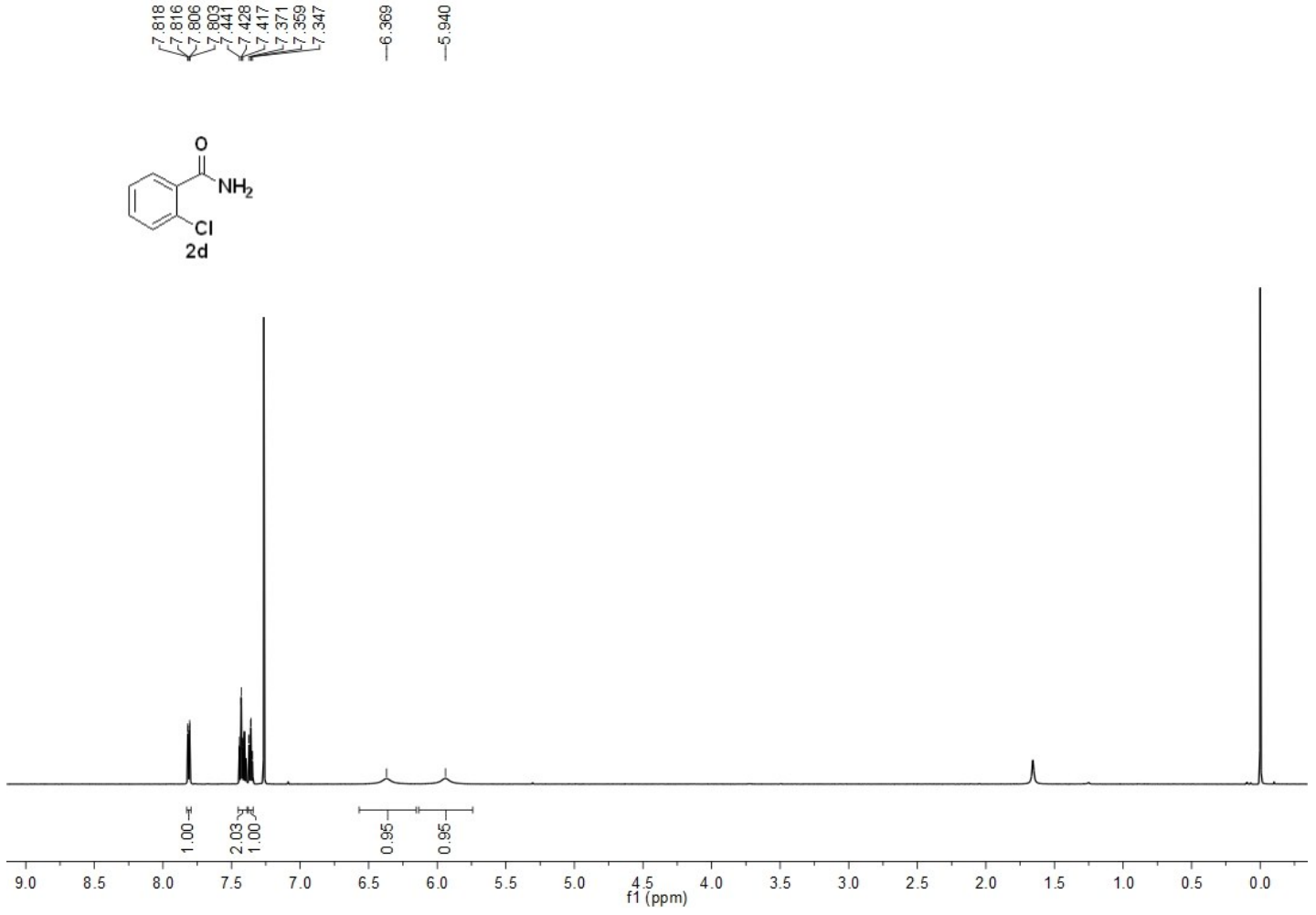

8.35
7.75
7.72
7.49
7.31
6.60
3.84
O
N
H
OMe
1t
0.96
2.08
2.93
0.87
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

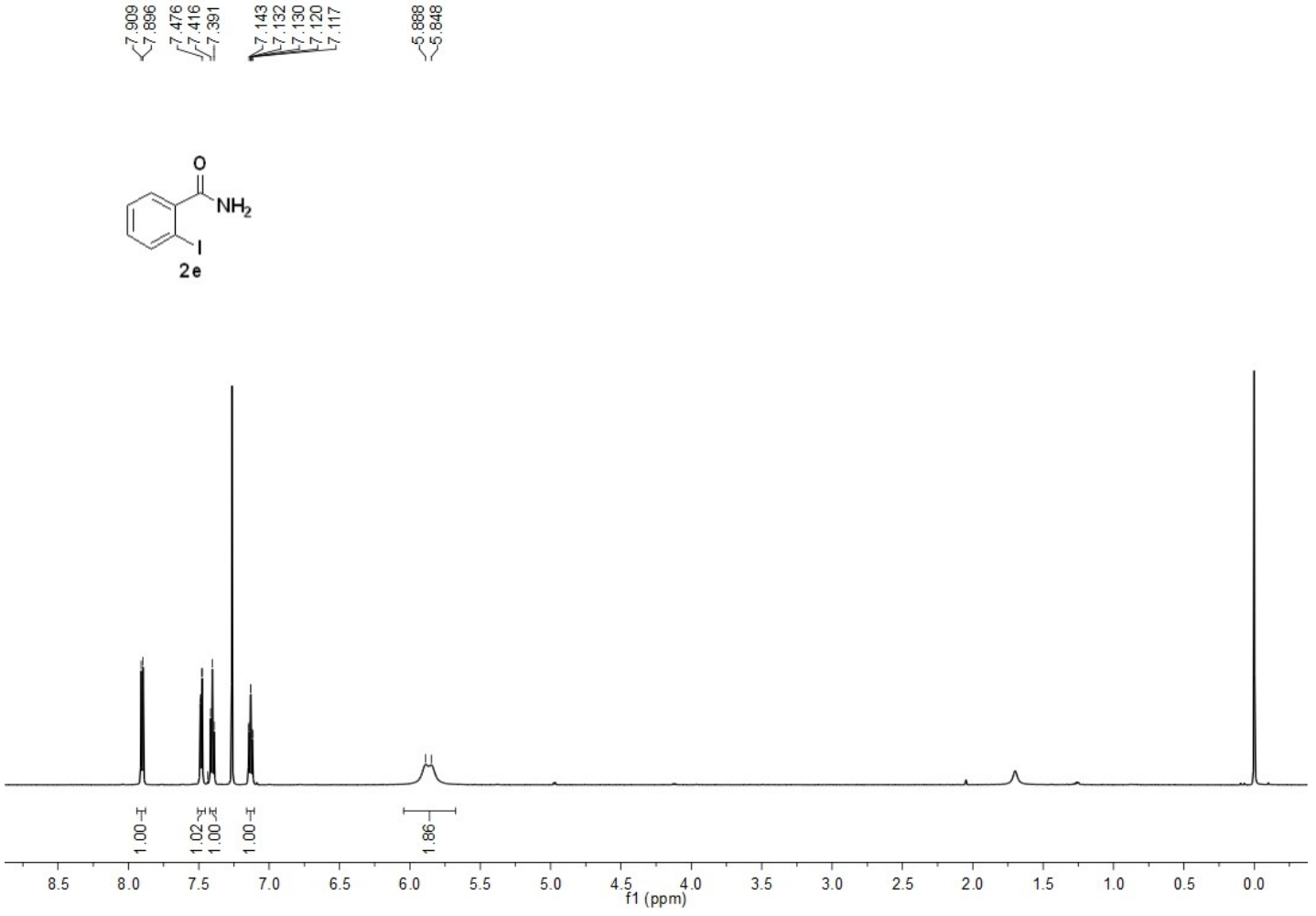

164.59
141.75
134.67
129.98
128.86
127.96
117.00
77.29
77.08
76.87
64.36
1t
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

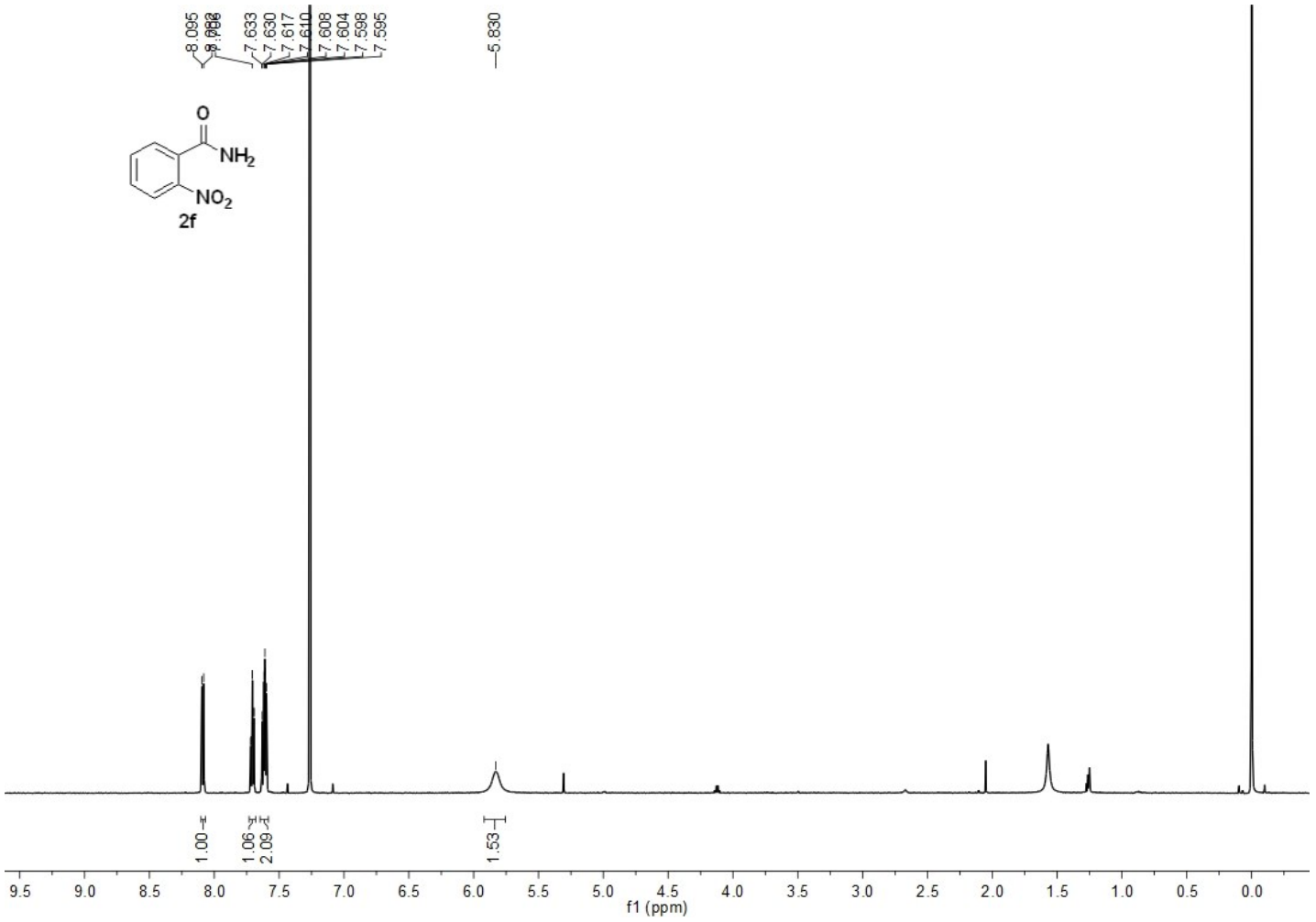

7.832
7.831
7.819
7.817
7.543
7.532
7.530
7.473
7.460
7.447
6.177
5.994
O
NH2
2a
2.00
0.99
2.01
0.94
1.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


7.724
7.710
7.264
7.251
6.083
5.691
2.412
O
NH2
2b
2.00
3.00
0.93
0.89
3.13
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)


7.799
7.784
6.945
6.931
5.983
3.860
O
NH2
MeO
2c
1.87
1.90
1.63
2.79
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)


7.818
7.816
7.806
7.803
7.441
7.428
7.417
7.371
7.359
7.347
6.369
5.940
O
NH2
Cl
2d
1.00
2.03
1.00
0.95
0.95
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)


7.909
7.896
7.476
7.416
7.391
7.143
7.132
7.130
7.120
7.117
5.888
5.848
O
NH2
I
2e
1.00
1.02
1.00
1.00
1.86
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)


8.095
7.633
7.630
7.617
7.610
7.608
7.604
7.598
7.595
5.830
O
NH2
NO2
2f
1.00
1.06
2.09
1.53
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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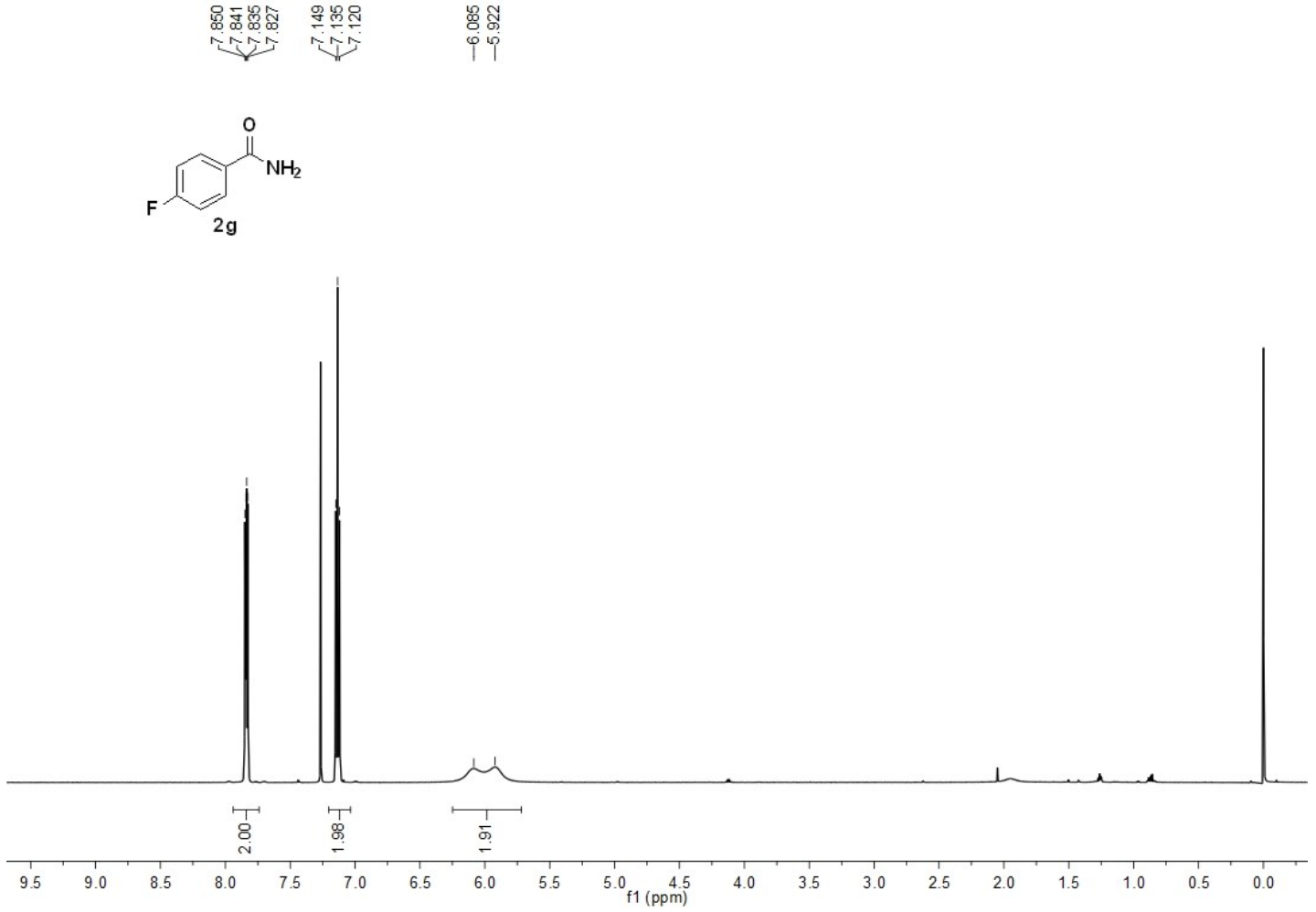
f1 (ppm)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6.085
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O
NH2
F
2g
2.00
1.98
1.91
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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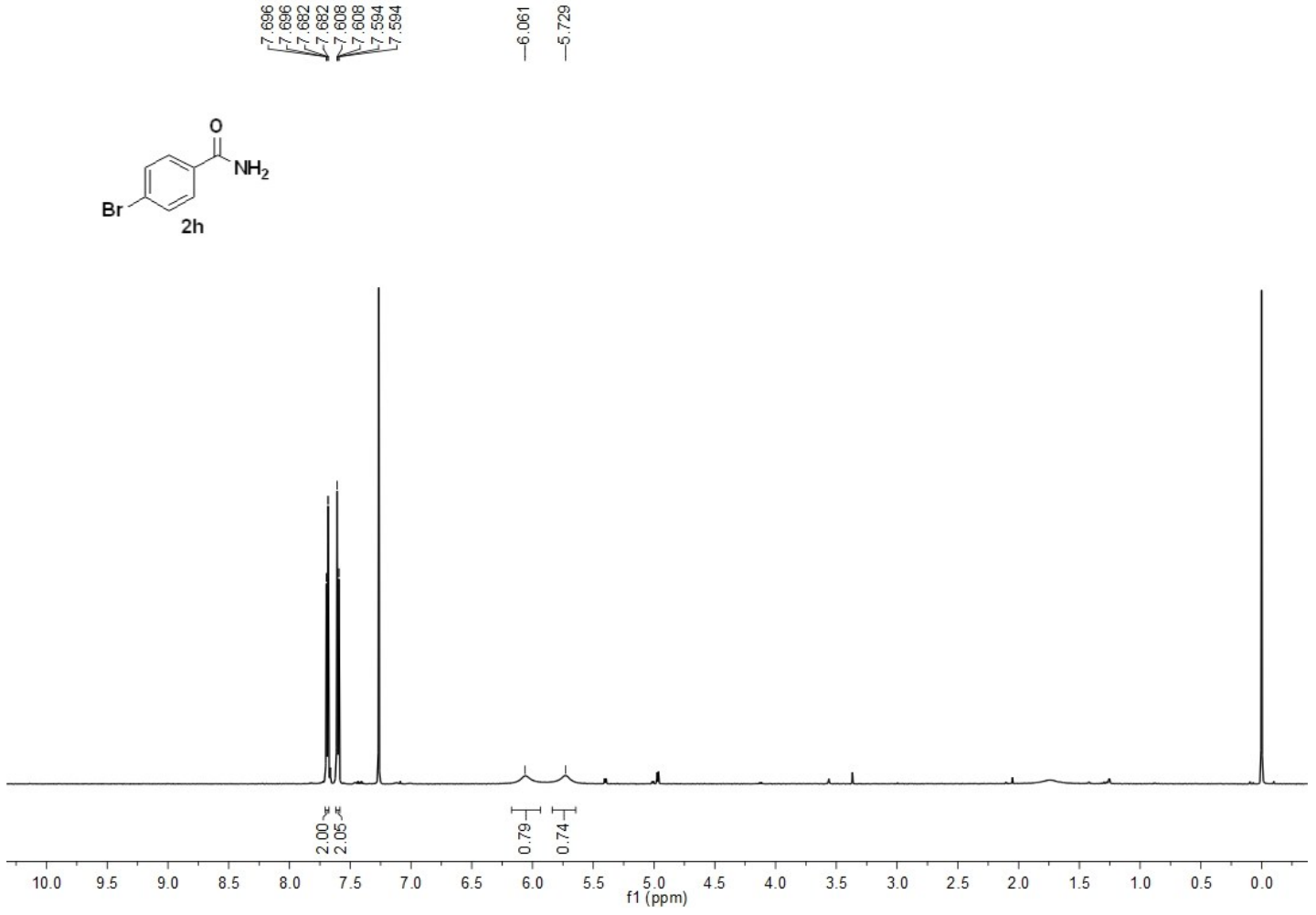
0.0
f1 (ppm)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7.696
7.682
7.682
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7.608
7.594
7.594
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5.729
O
NH2
Br
2h
2.00
2.05
0.79
0.74
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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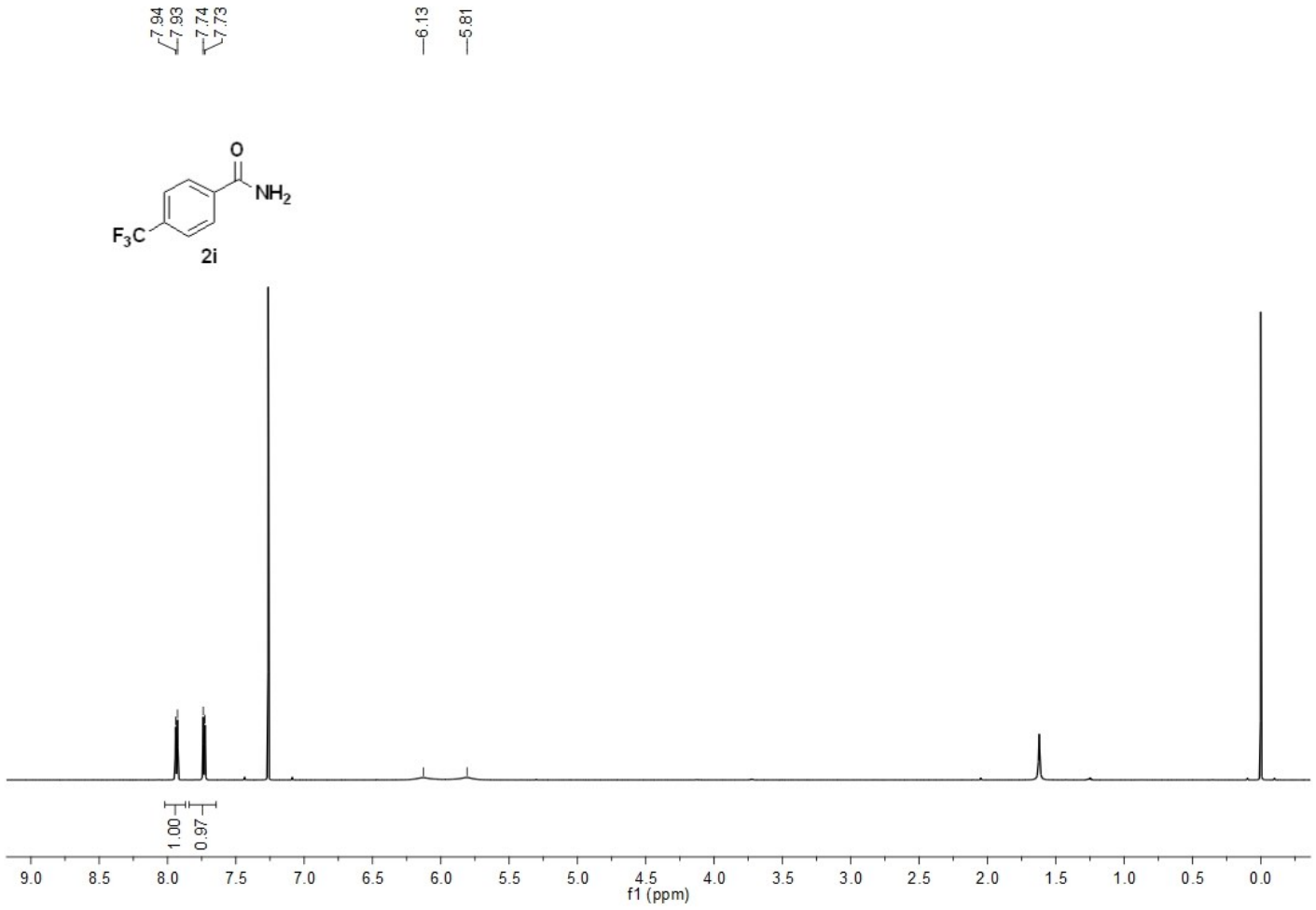
0.0
f1 (ppm)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F3C
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8.5
8.0
7.5
7.0
6.5
6.0
5.5
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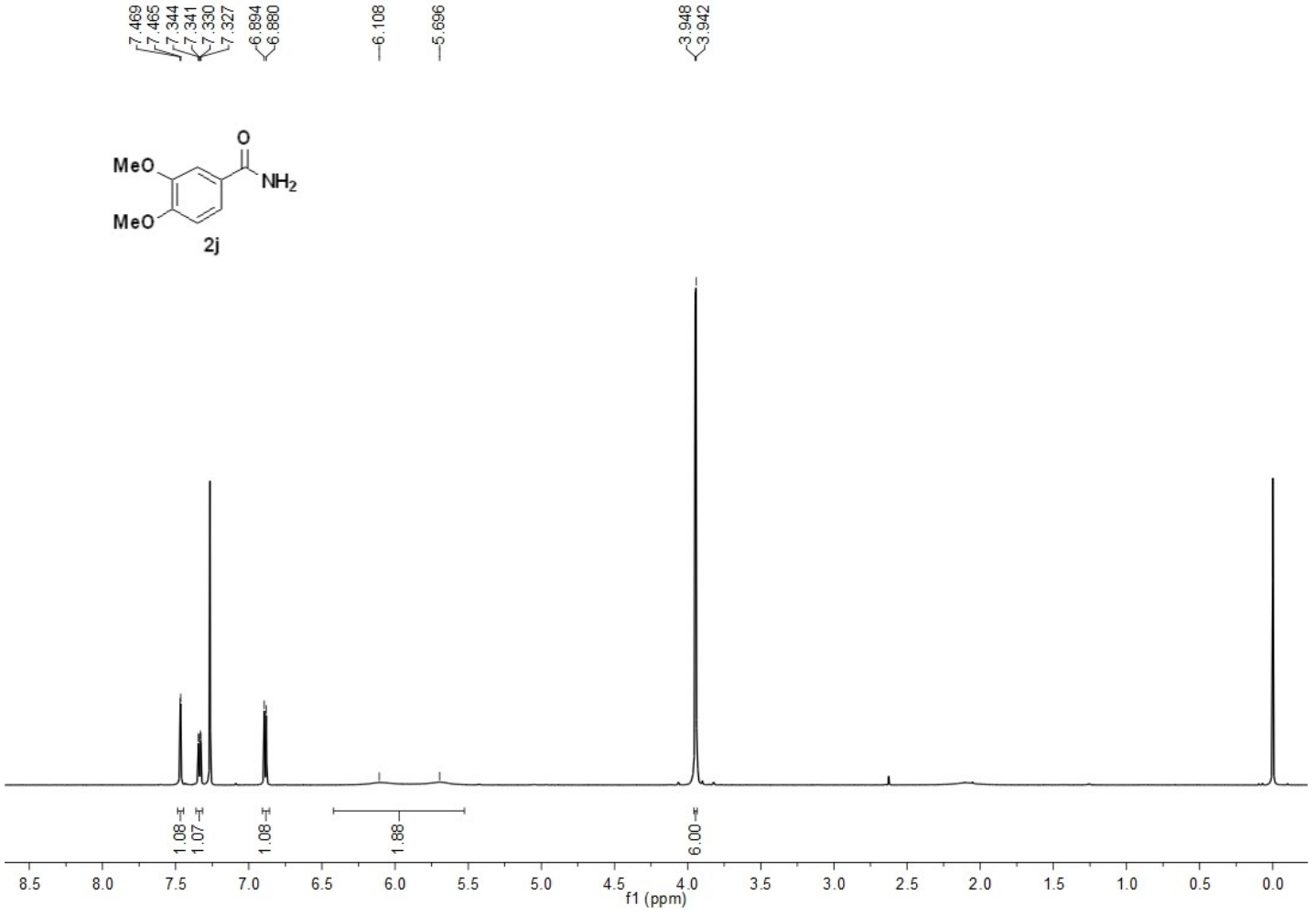
f1 (ppm)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7.465
7.344
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7.330
7.327
6.894
6.880
6.108
5.696
3.948
3.942
MeO
MeO
O
NH2
2j
1.08
1.07
1.08
1.88
6.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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2.5
2.0
1.5
1.0
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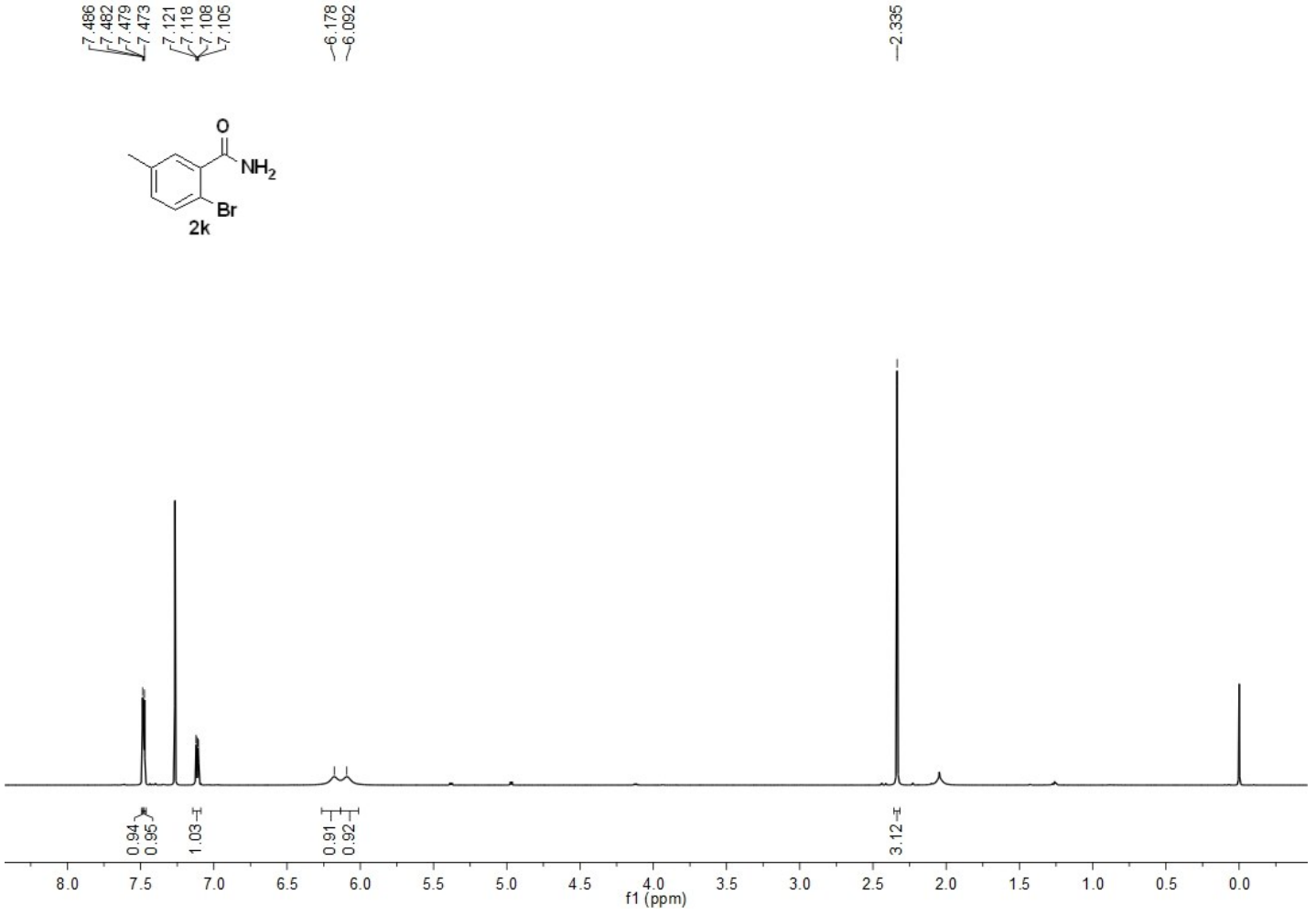
0.0
f1 (ppm)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7.473
7.121
7.118
7.108
7.105
6.178
6.092
2.335
O
NH2
Br
2k
0.94
0.95
1.03
0.91
0.92
3.12
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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2.0
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1.0
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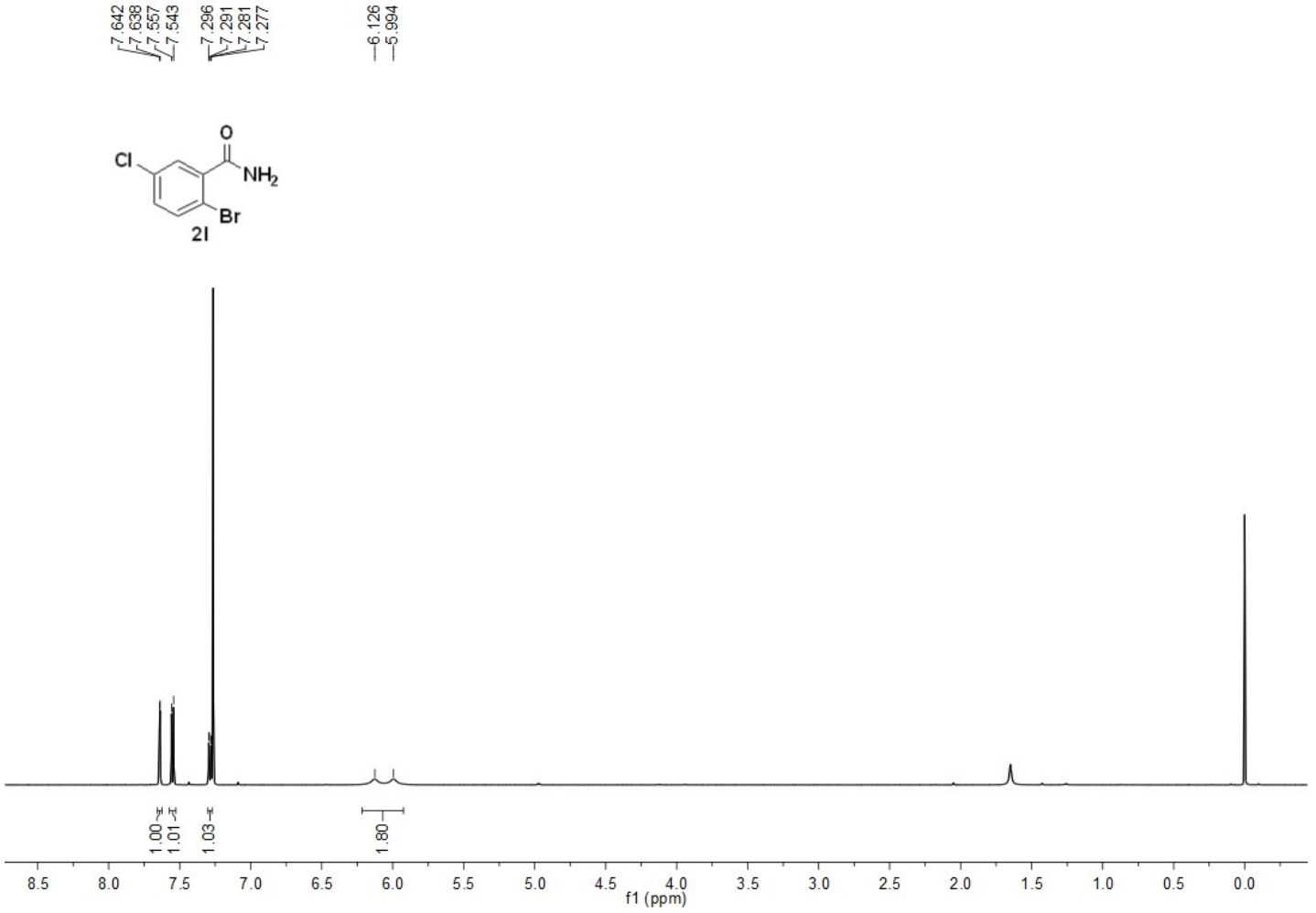
0.0
f1 (ppm)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7.277
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O
Cl
NH2
Br
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1.00
1.01
1.03
1.80
8.5
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7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
1.5
1.0
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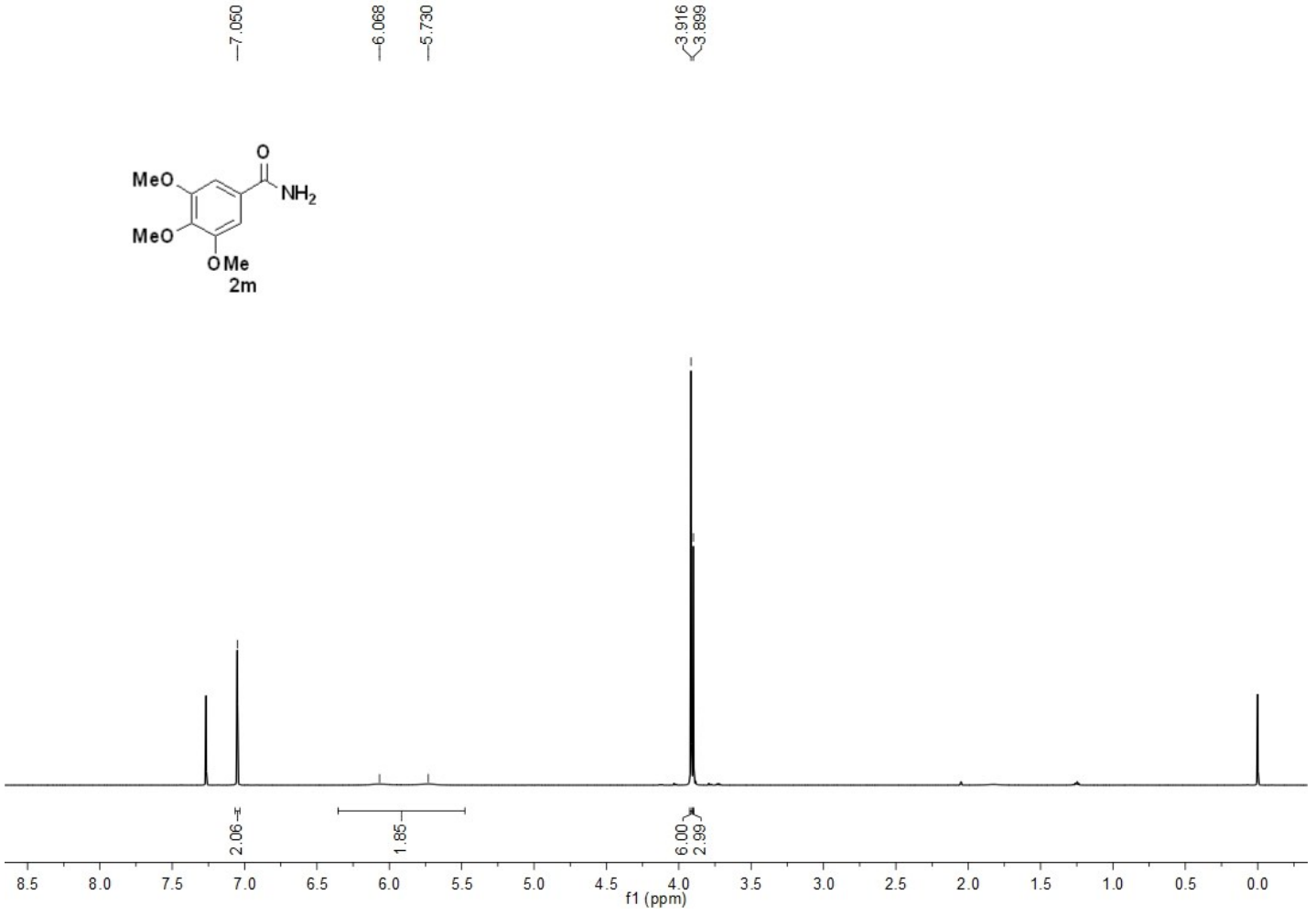
0.0
f1 (ppm)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MeO
OMe
O
NH2
2m
2.06
1.85
6.00
2.99
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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3.5
3.0
2.5
2.0
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1.0
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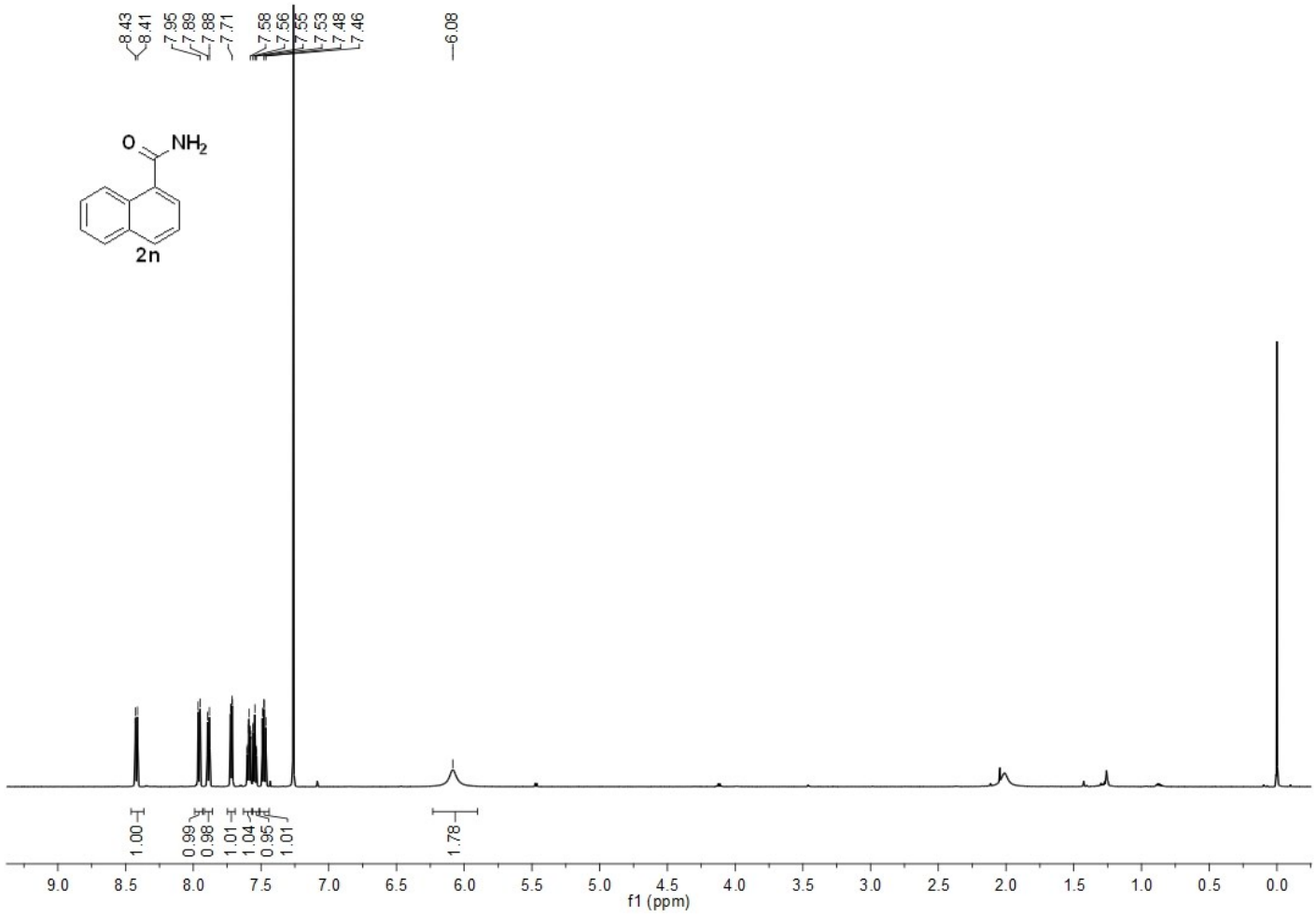
0.0
f1 (ppm)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8.41
7.95
7.89
7.88
7.71
7.58
7.56
7.55
7.53
7.48
7.46
6.08
O
NH2
2n
1.00
0.99
0.98
1.01
1.04
0.95
1.01
1.78
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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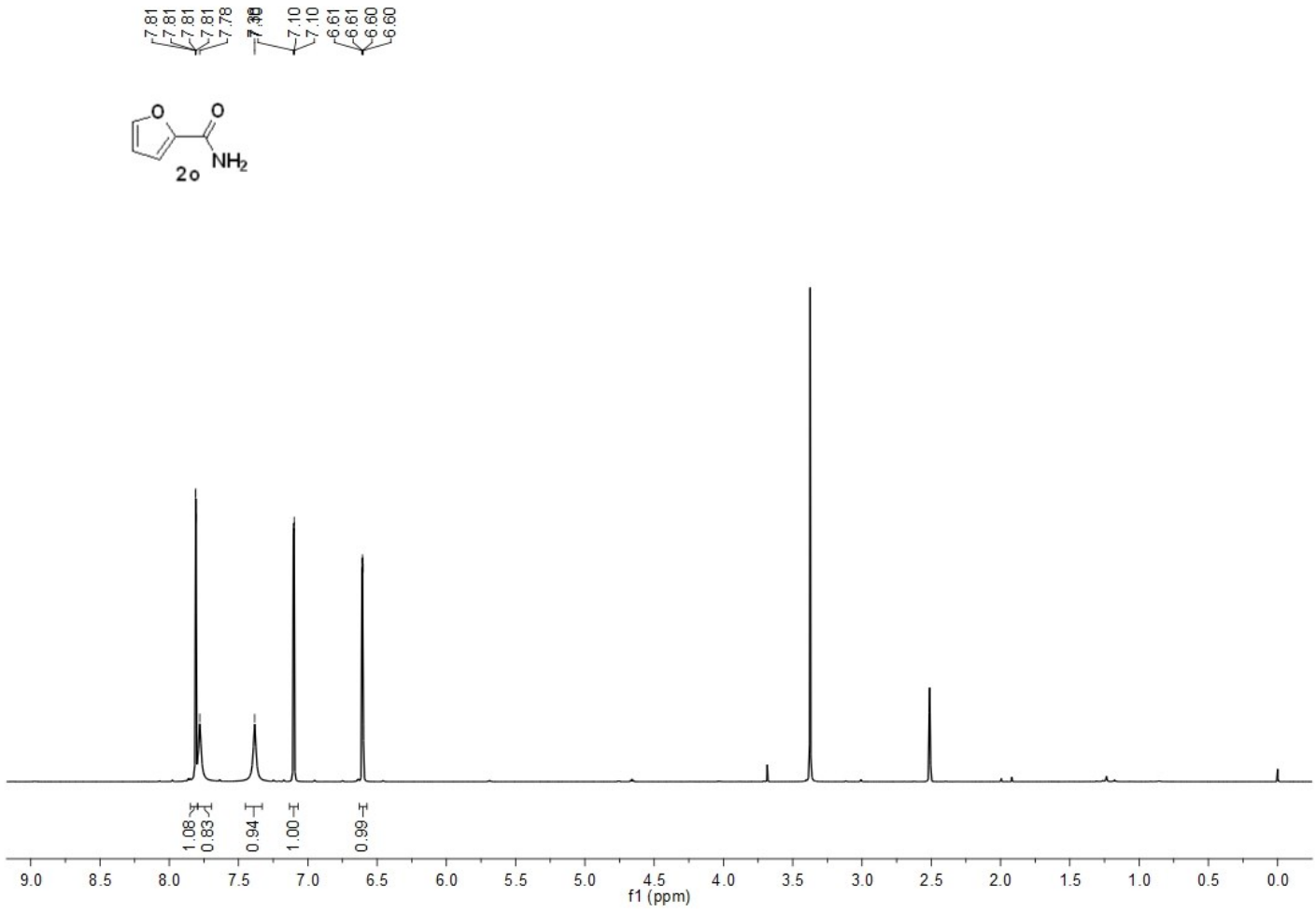
f1 (ppm)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7.81
7.81
7.81
7.78
7.10
7.10
6.61
6.61
6.60
6.60
O
O
NH2
2o
1.08
0.83
0.94
1.00
0.99
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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1.0
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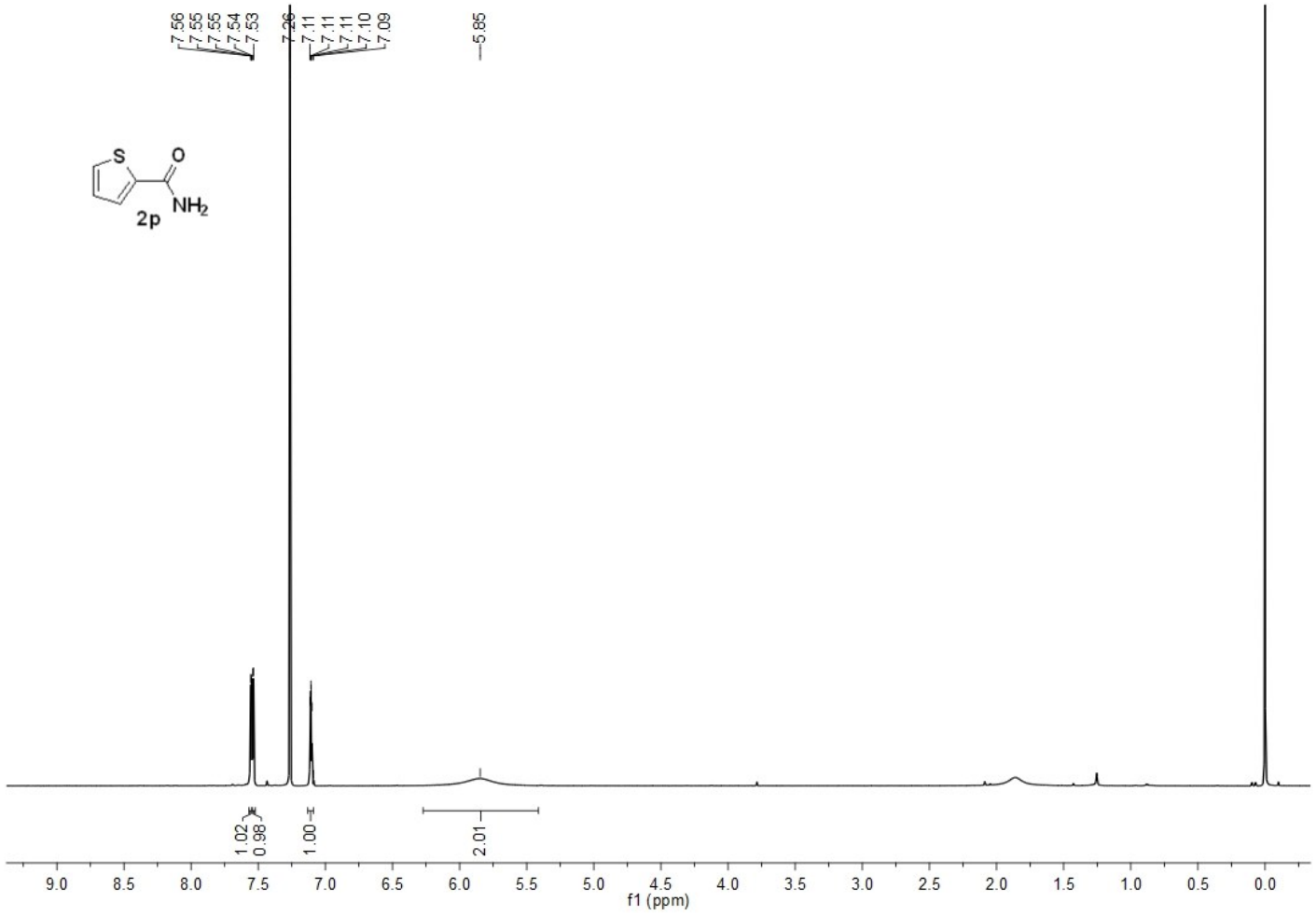
0.0
f1 (ppm)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7.55
7.54
7.53
7.26
7.11
7.11
7.11
7.10
7.09
5.85
S
O
NH2
2p
1.02
0.98
1.00
2.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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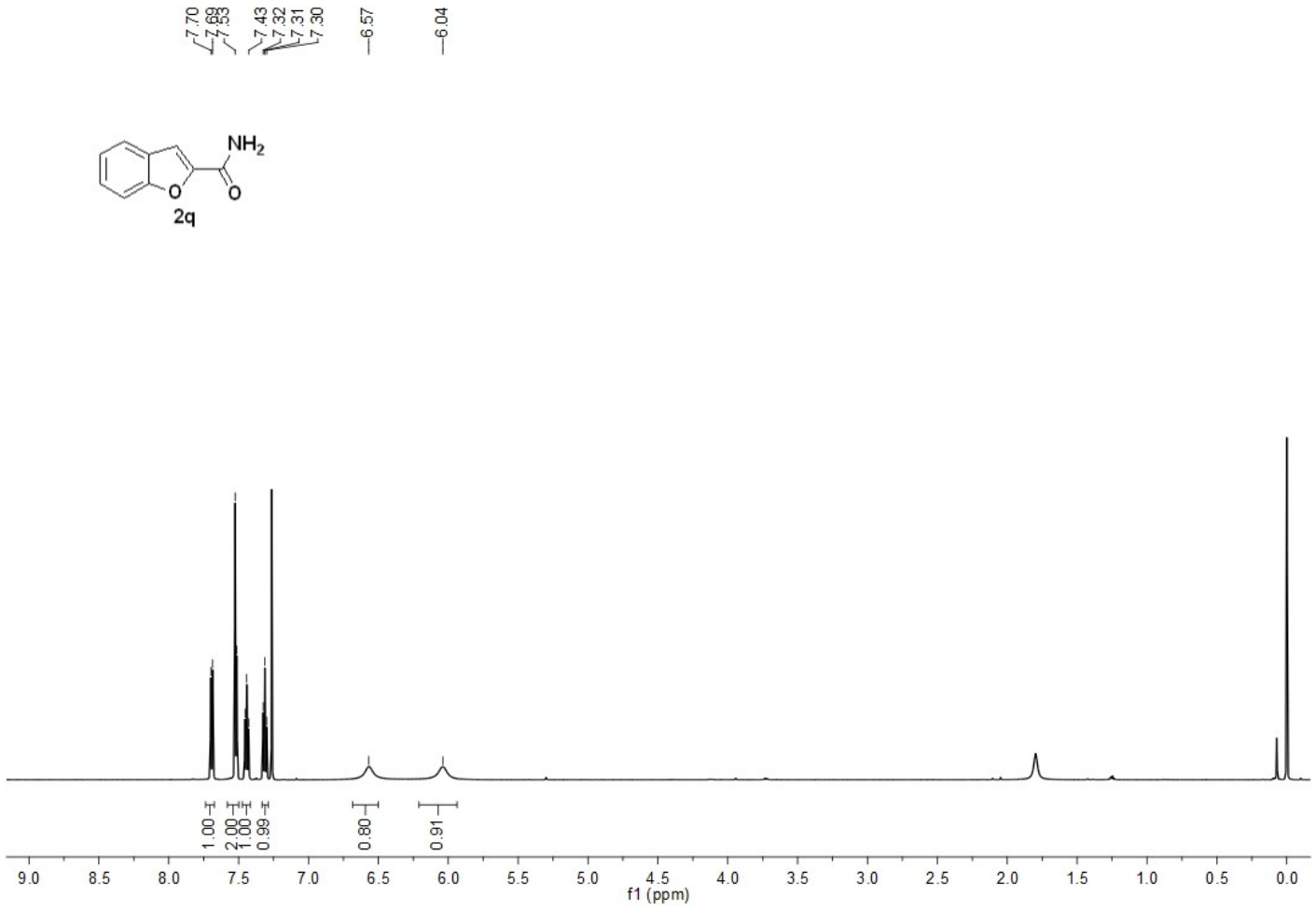
0.0
f1 (ppm)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7.53
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7.32
7.31
7.30
6.57
6.04
NH2
O
O
2q
1.00
2.00
1.00
0.99
0.80
0.91
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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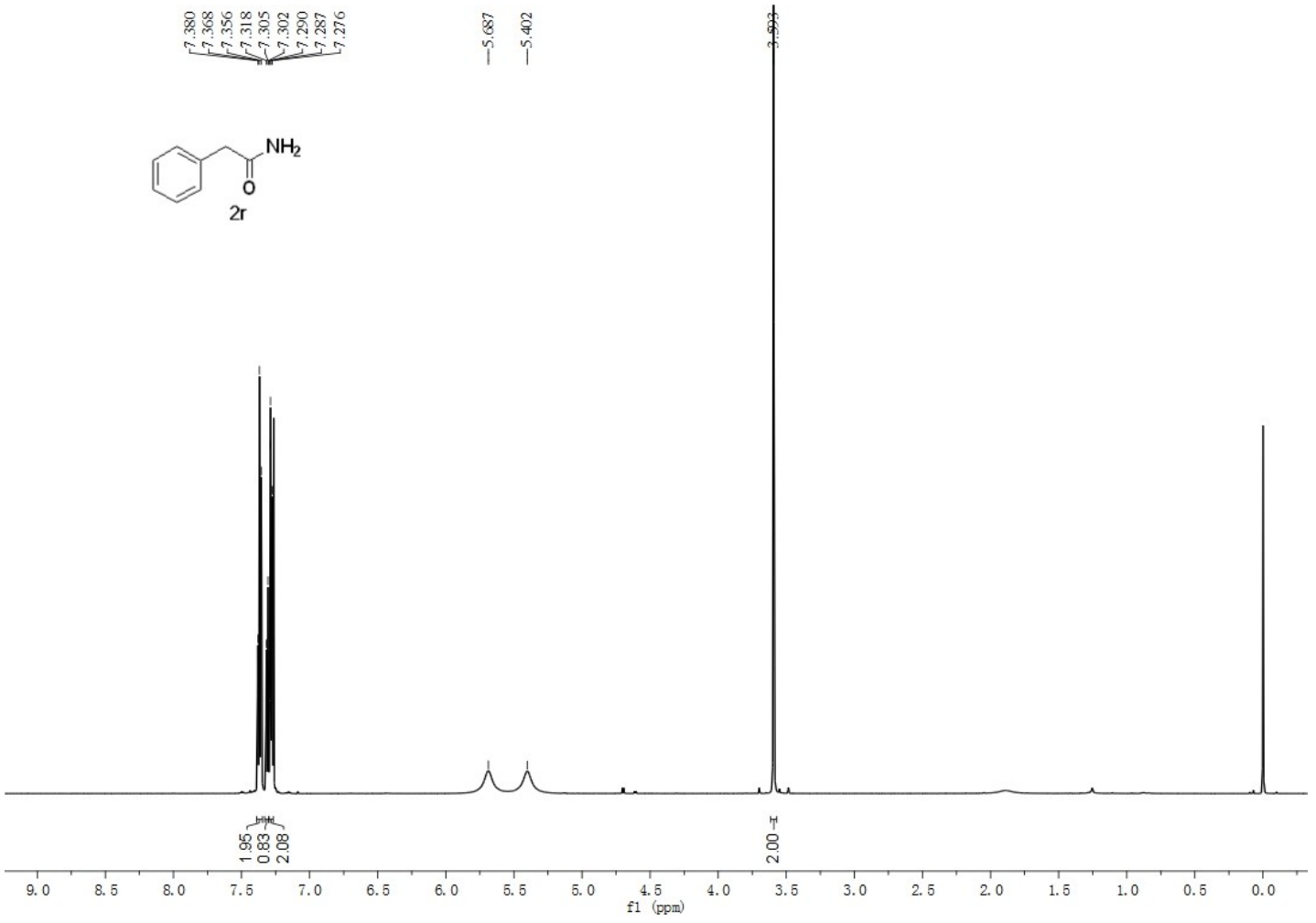
0.0
f1 (ppm)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7.302
7.290
7.287
7.276
5.687
5.402
3.593
NH2
O
2r
1.95
0.83
2.08
2.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.5
3.0
2.5
2.0
1.5
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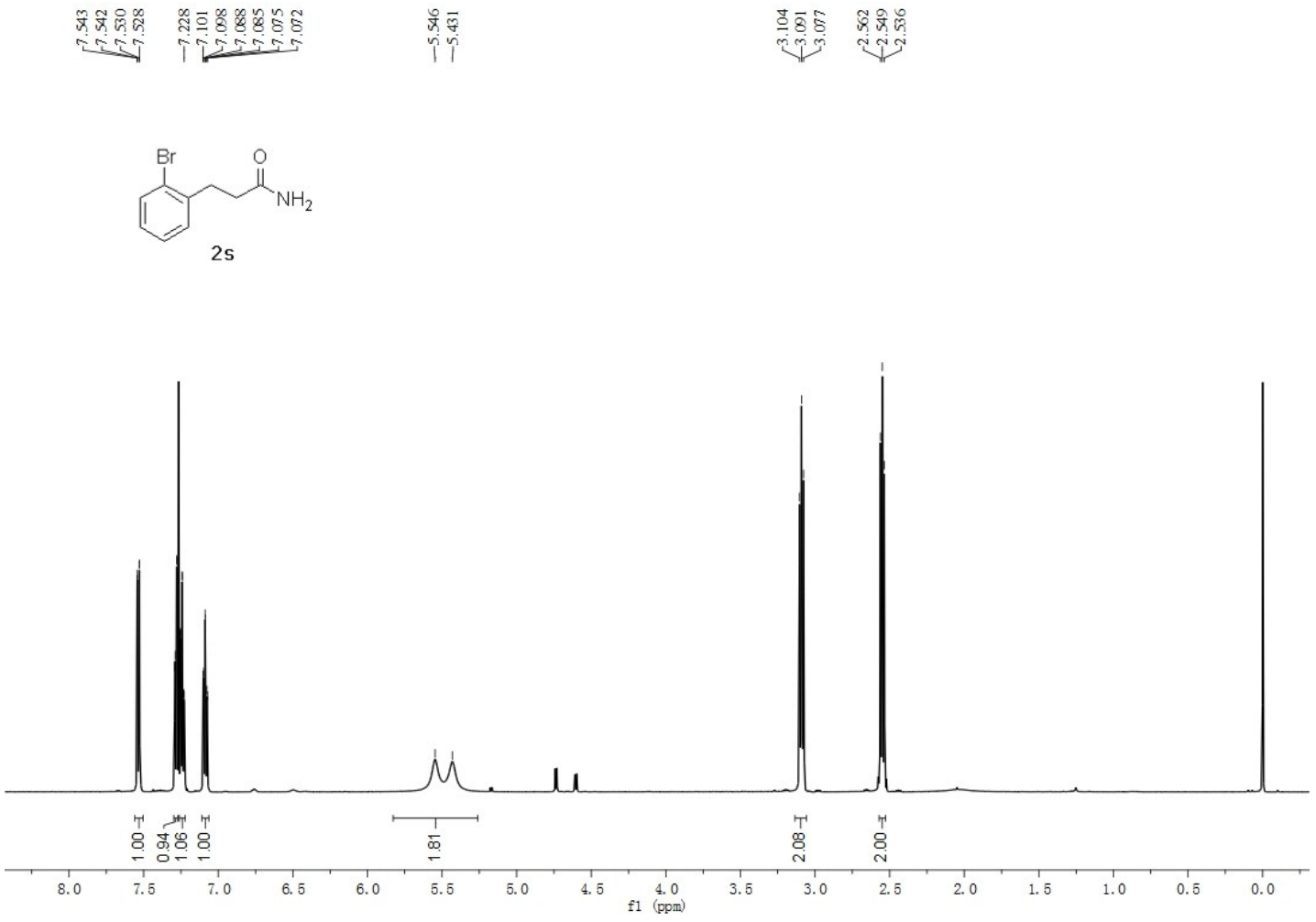
f1 (ppm)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7.228
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7.098
7.088
7.085
7.075
7.072
5.546
5.431
3.104
3.091
3.077
2.562
2.549
2.536
Br
O
NH2
2s
1.00
0.94
1.06
1.00
1.81
2.08
2.00
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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2.0
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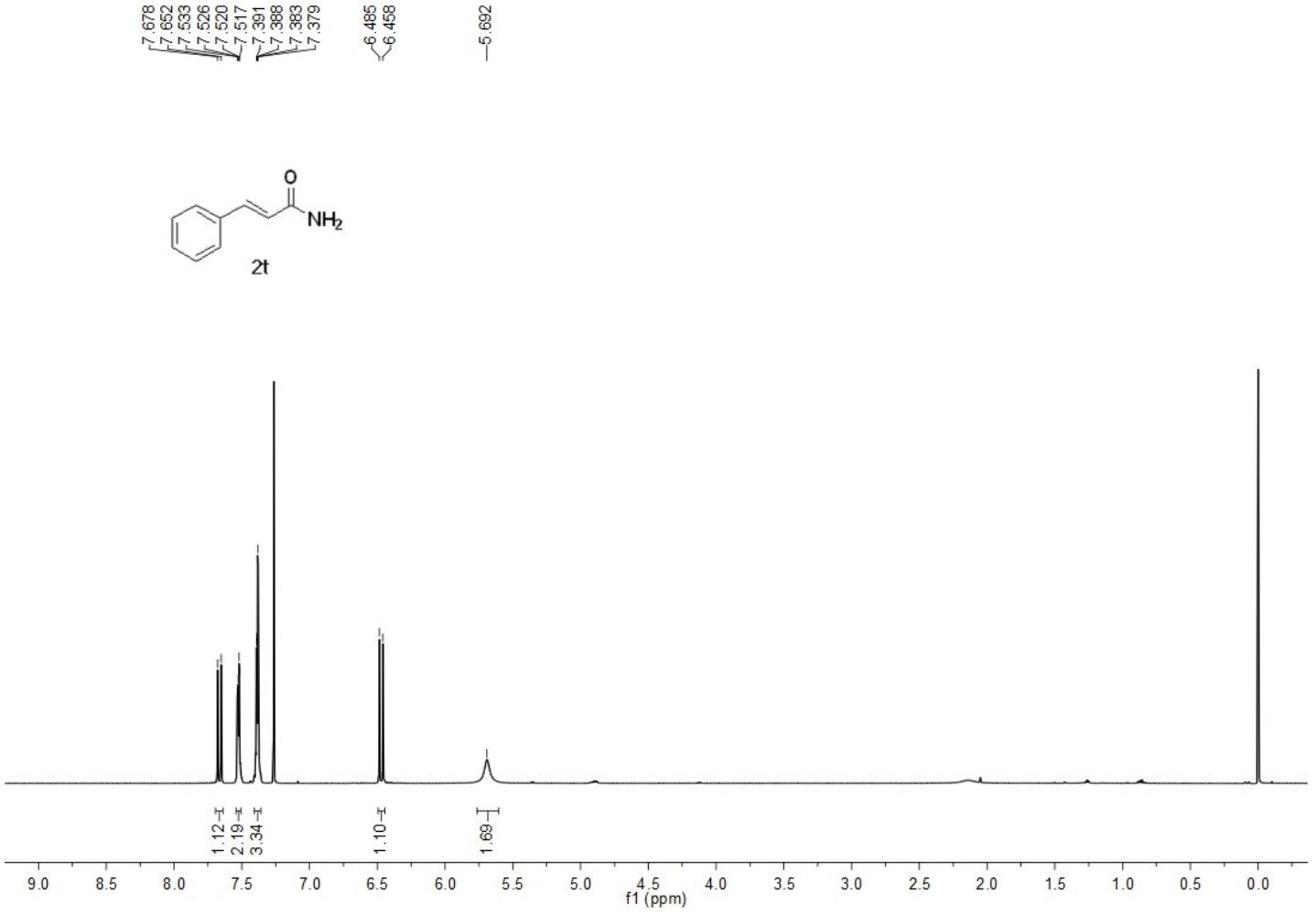
f1 (ppm)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7.526
7.520
7.517
7.391
7.388
7.383
7.379
6.485
6.458
5.692
O
NH2
2t
1.12
2.19
3.34
1.10
1.69
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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3.5
3.0
2.5
2.0
1.5
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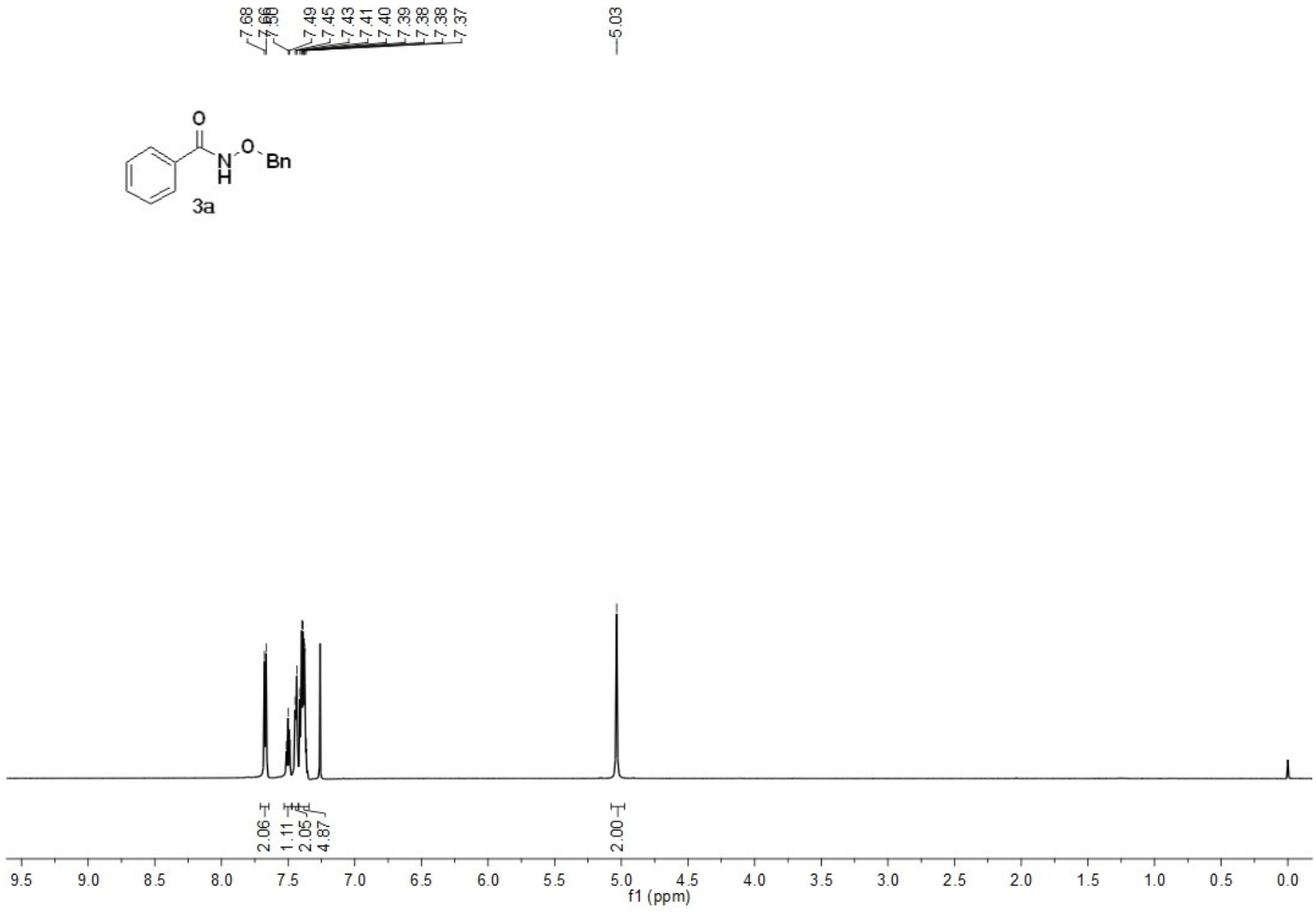
f1 (ppm)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7.39
7.38
7.38
7.37
5.03
O
N
H
O
Bn
3a
2.06
1.11
2.05
4.87
2.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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1.0
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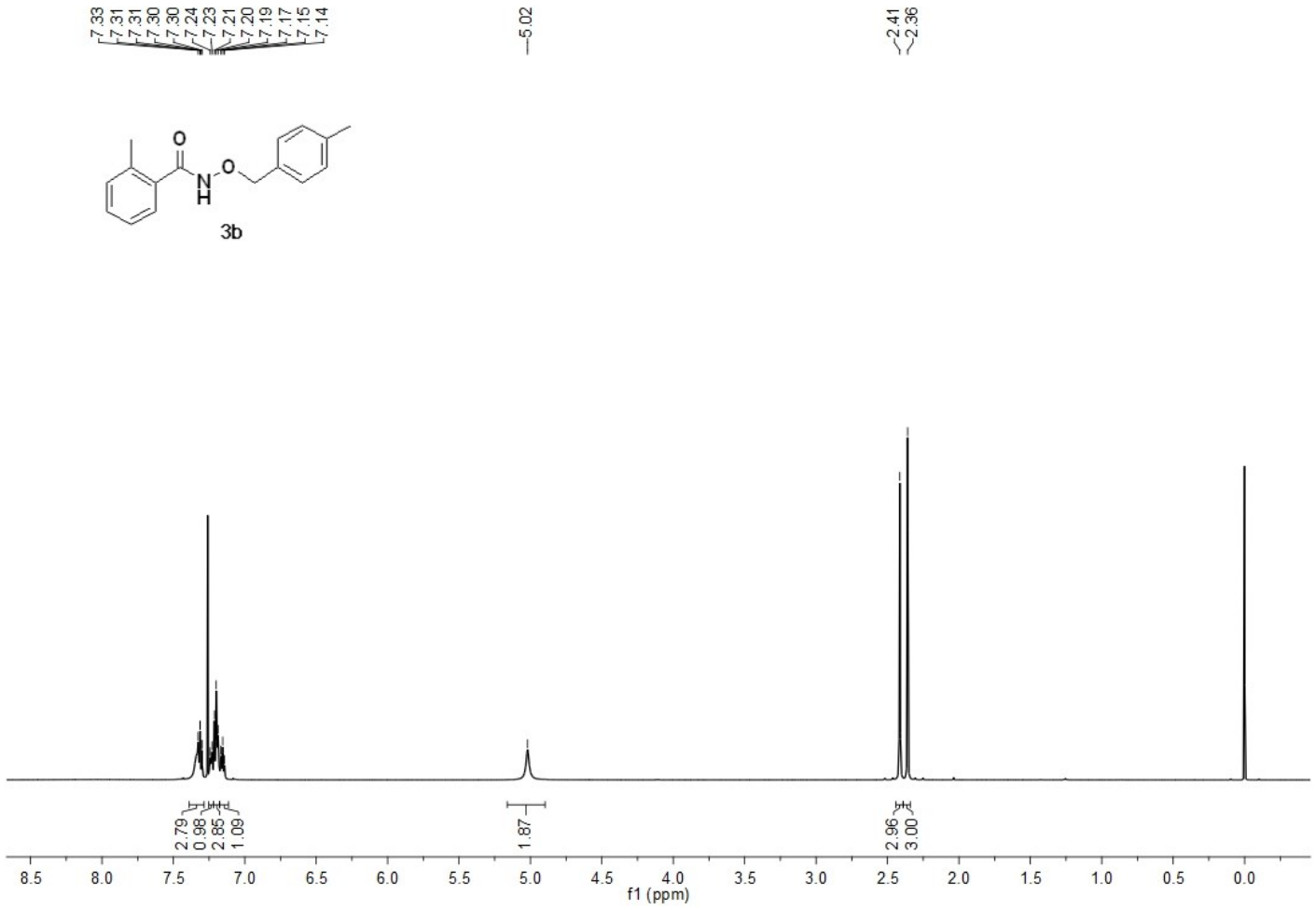
0.0
f1 (ppm)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7.23
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7.19
7.17
7.15
7.14
5.02
2.41
2.36
O
N
H
O
3b
2.79
0.98
2.85
1.09
1.87
2.96
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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1.0
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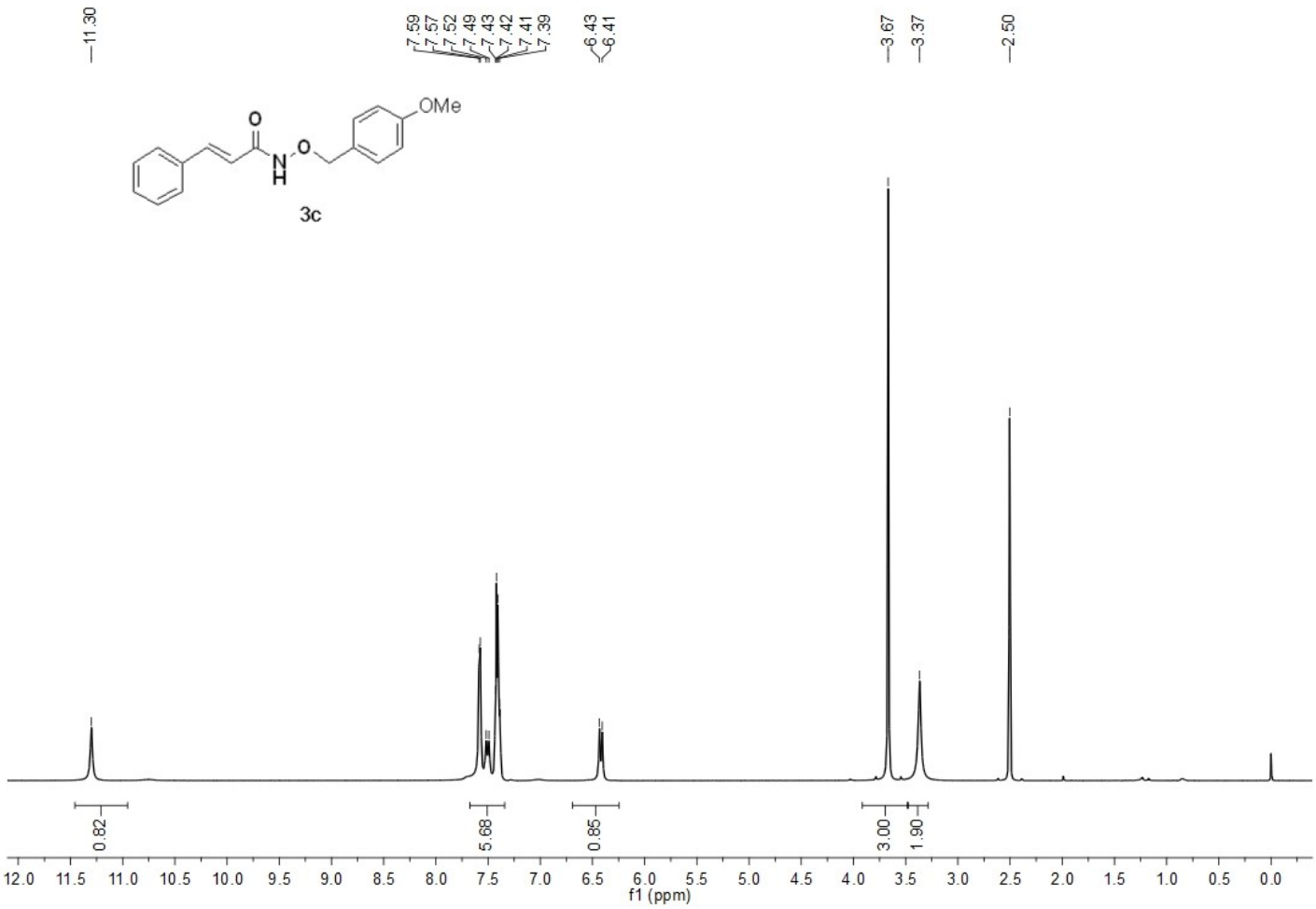
f1 (ppm)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7.41
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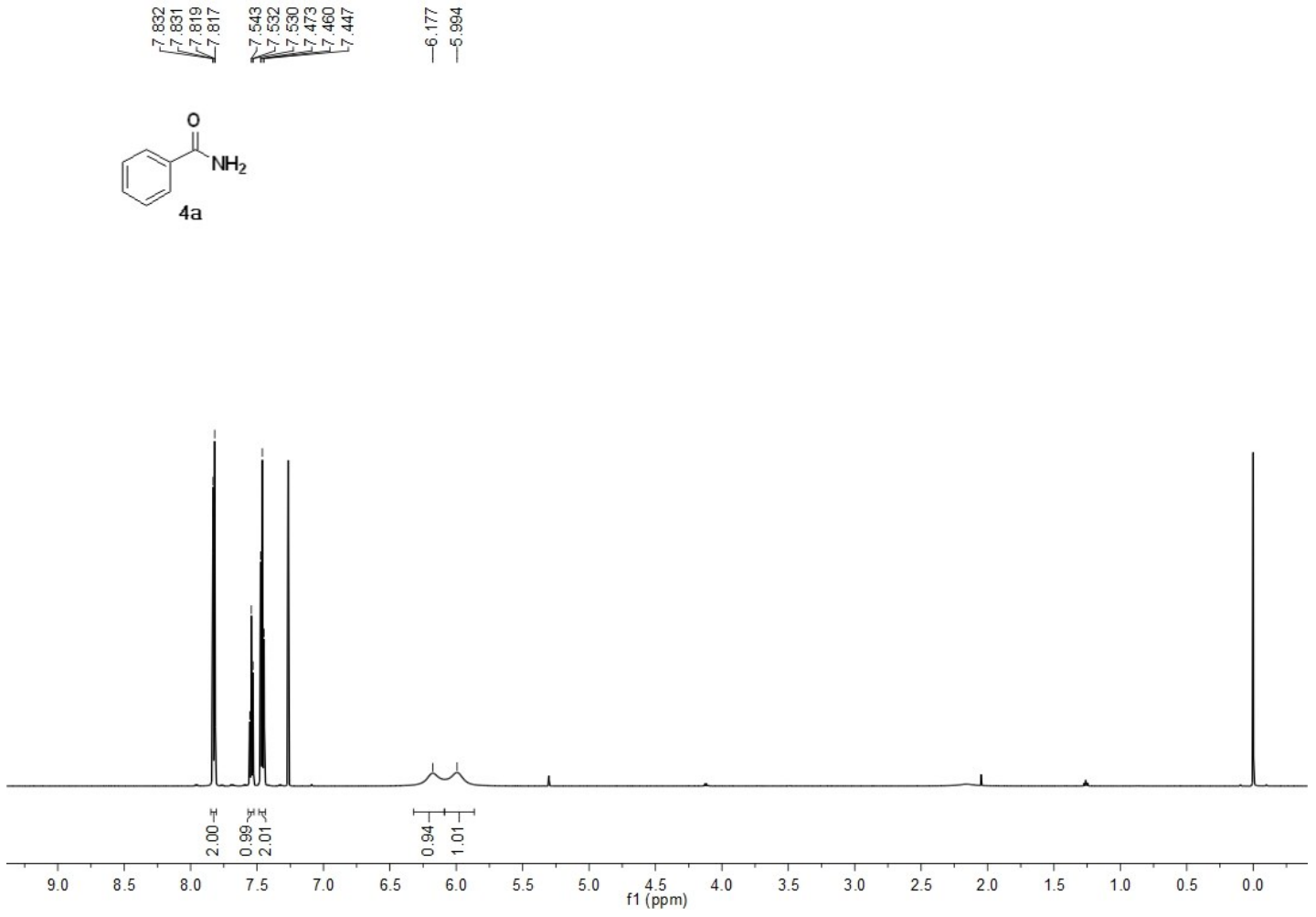
6.43
6.41
3.67
3.37
2.50
OMe
O
N
H
O
3c
0.82
5.68
0.85
3.00
1.90
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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6.177
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4a
2.00
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2.01
0.94
1.01
9.0
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8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
1.5
1.0
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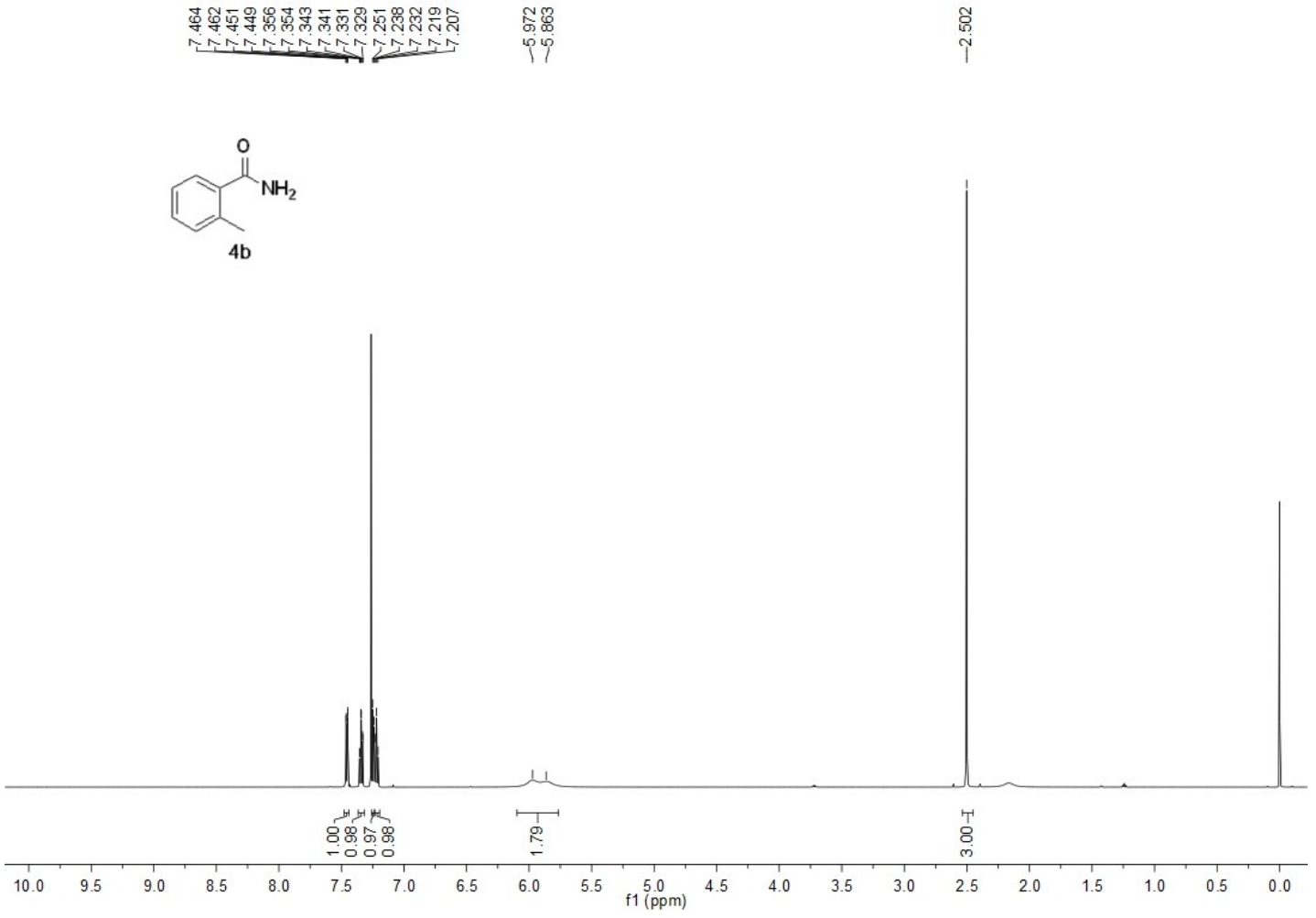
0.0
f1 (ppm)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7.219
7.207
5.972
5.863
2.502
O
NH2
4b
1.00
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0.97
0.98
1.79
3.00
10.0
9.5
9.0
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8.0
7.5
7.0
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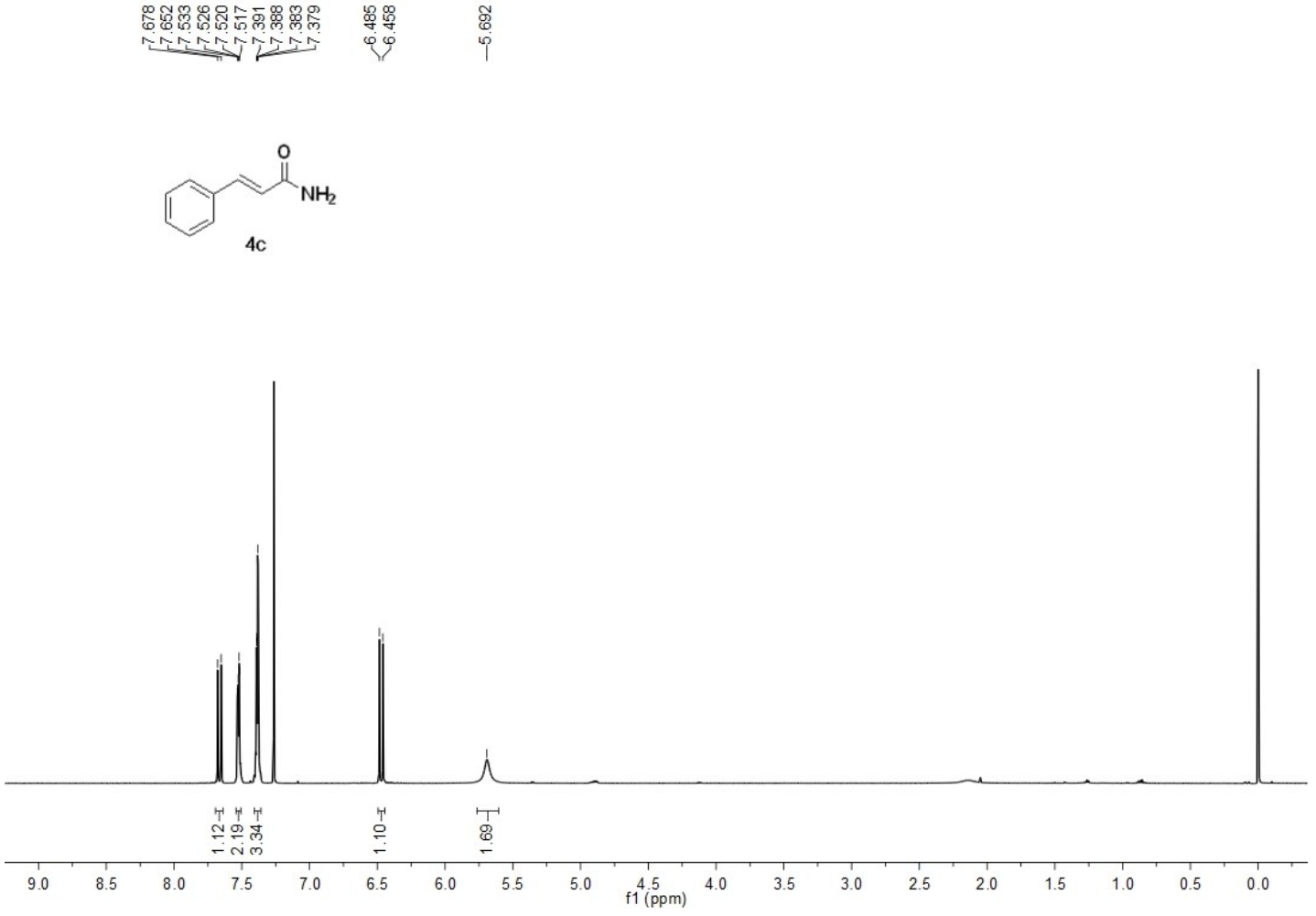
f1 (ppm)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7.652
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6.485
6.458
5.692
O
NH2
4c
1.12
2.19
3.34
1.10
1.69
9.0
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8.0
7.5
7.0
6.5
6.0
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1.0
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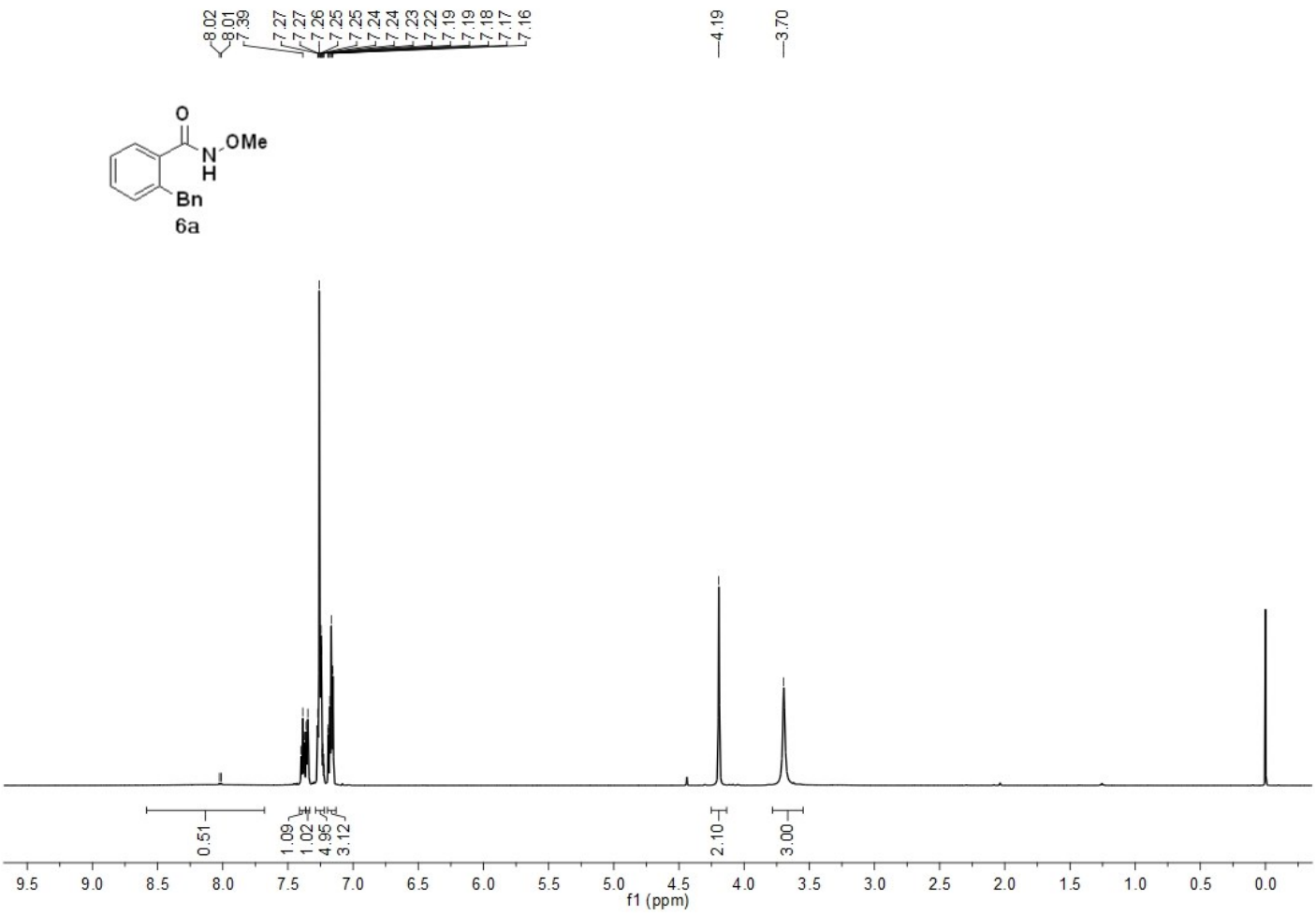
f1 (ppm)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7.19
7.18
7.17
7.16
4.19
3.70
O
N
H
OMe
Bn
6a
0.51
1.09
1.02
4.95
3.12
2.10
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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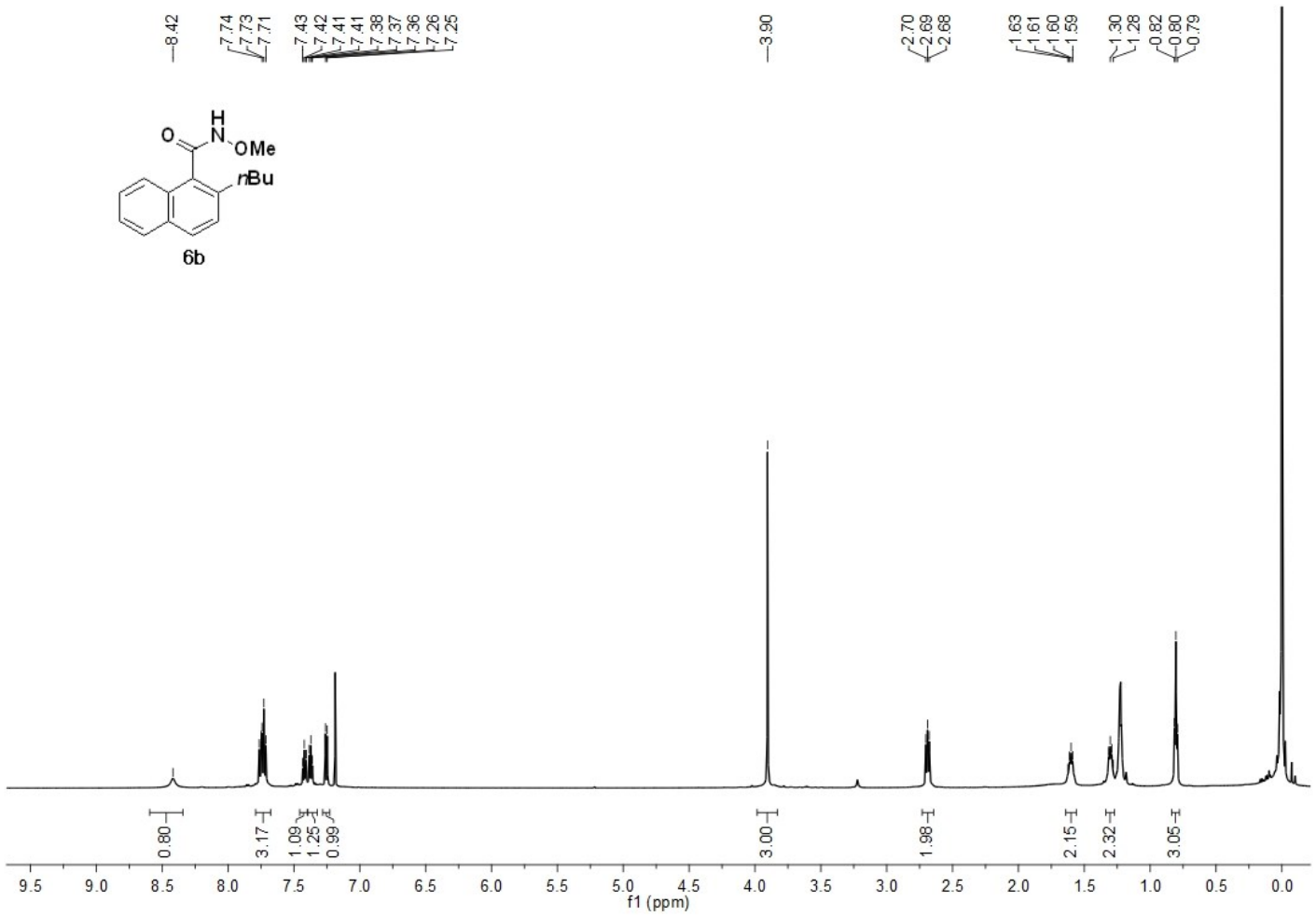
f1 (ppm)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2.70
2.69
2.68
1.63
1.61
1.60
1.59
1.30
1.28
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0.80
0.79
O
H
N
OMe
nBu
6b
0.80
3.17
1.09
1.25
0.99
3.00
1.98
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2.32
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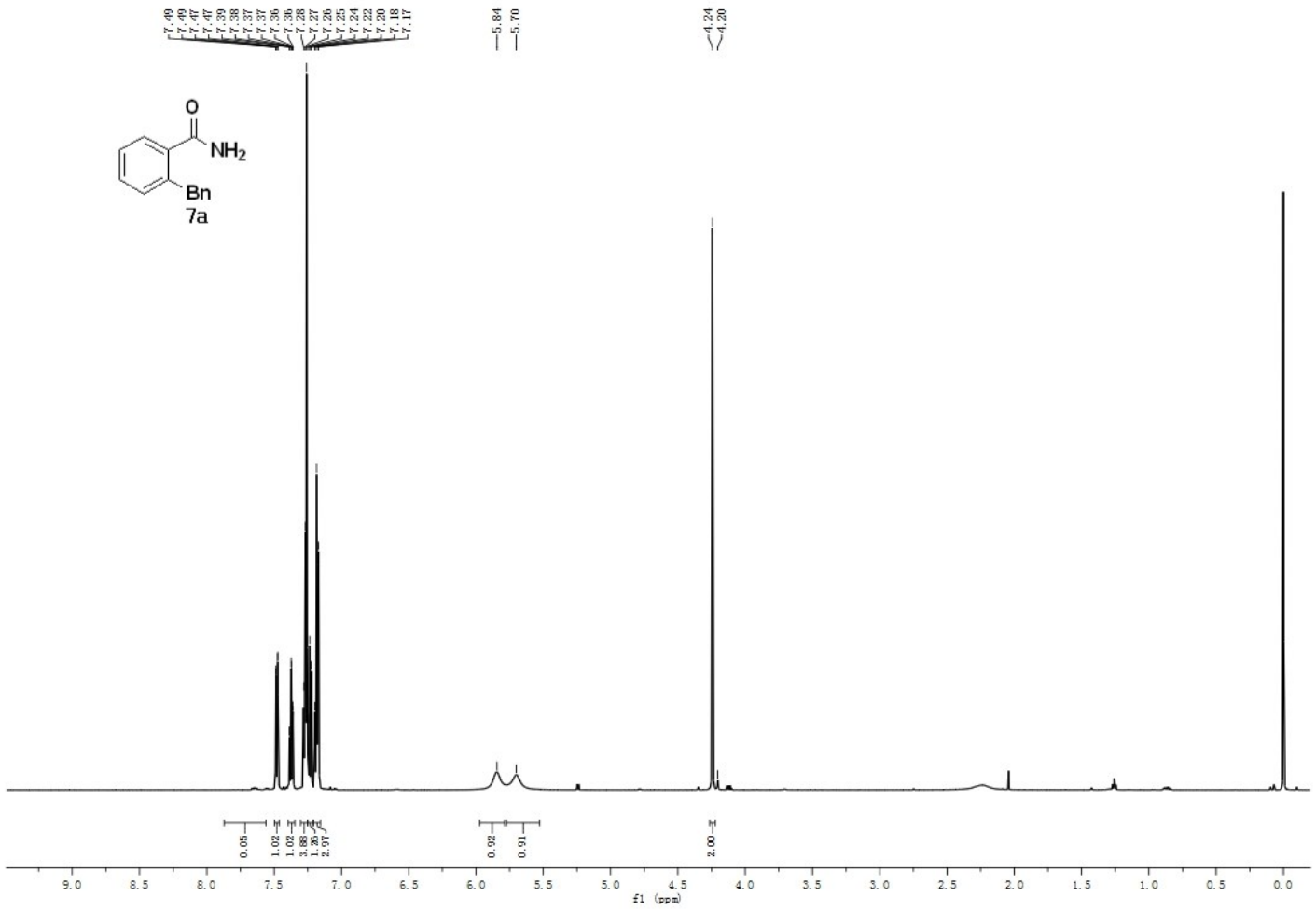
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7.25
7.24
7.22
7.20
7.18
7.17
5.84
5.70
4.24
4.20
O
NH2
Bn
7a
0.05
1.02
1.02
3.98
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2.97
0.92
0.91
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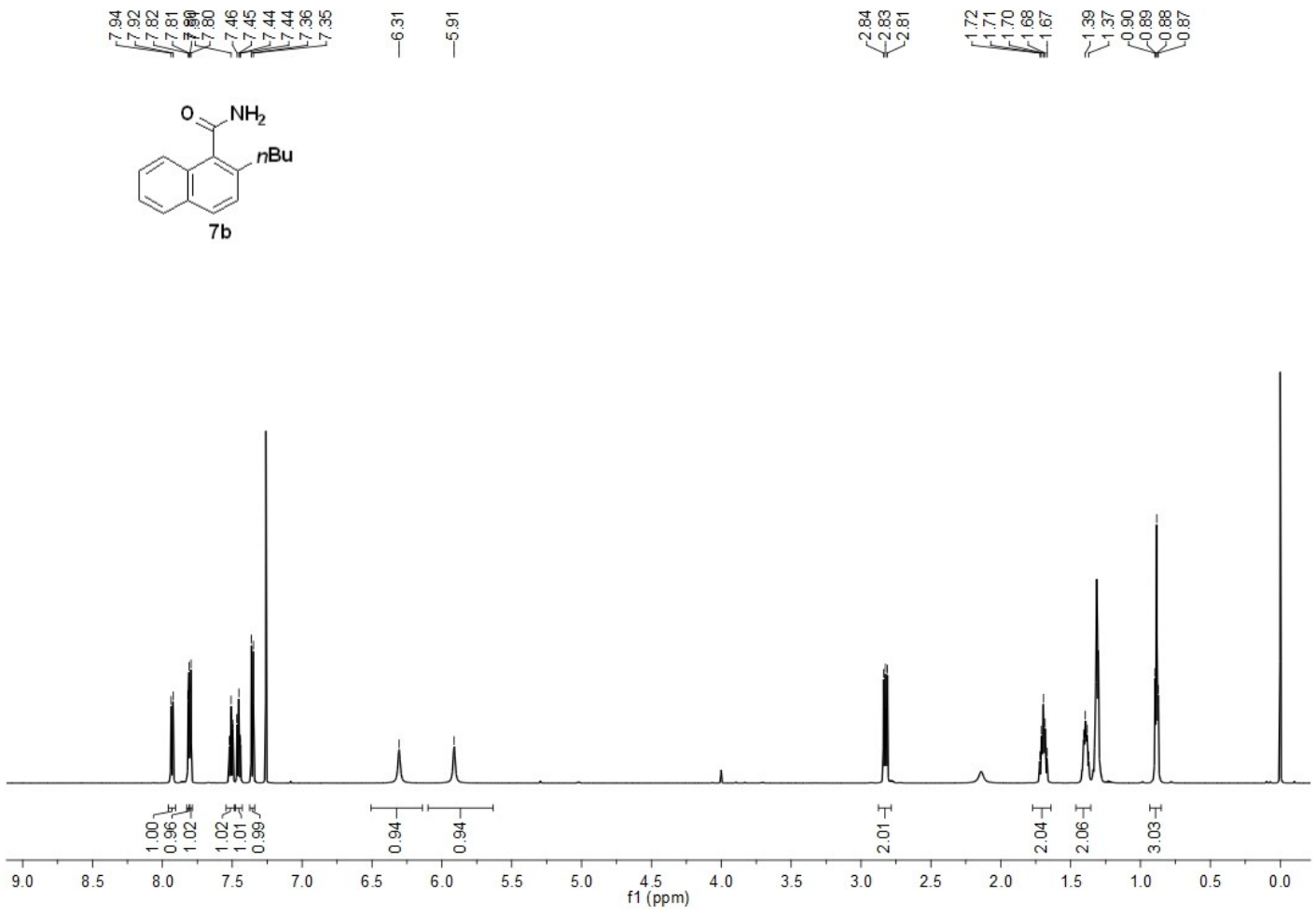
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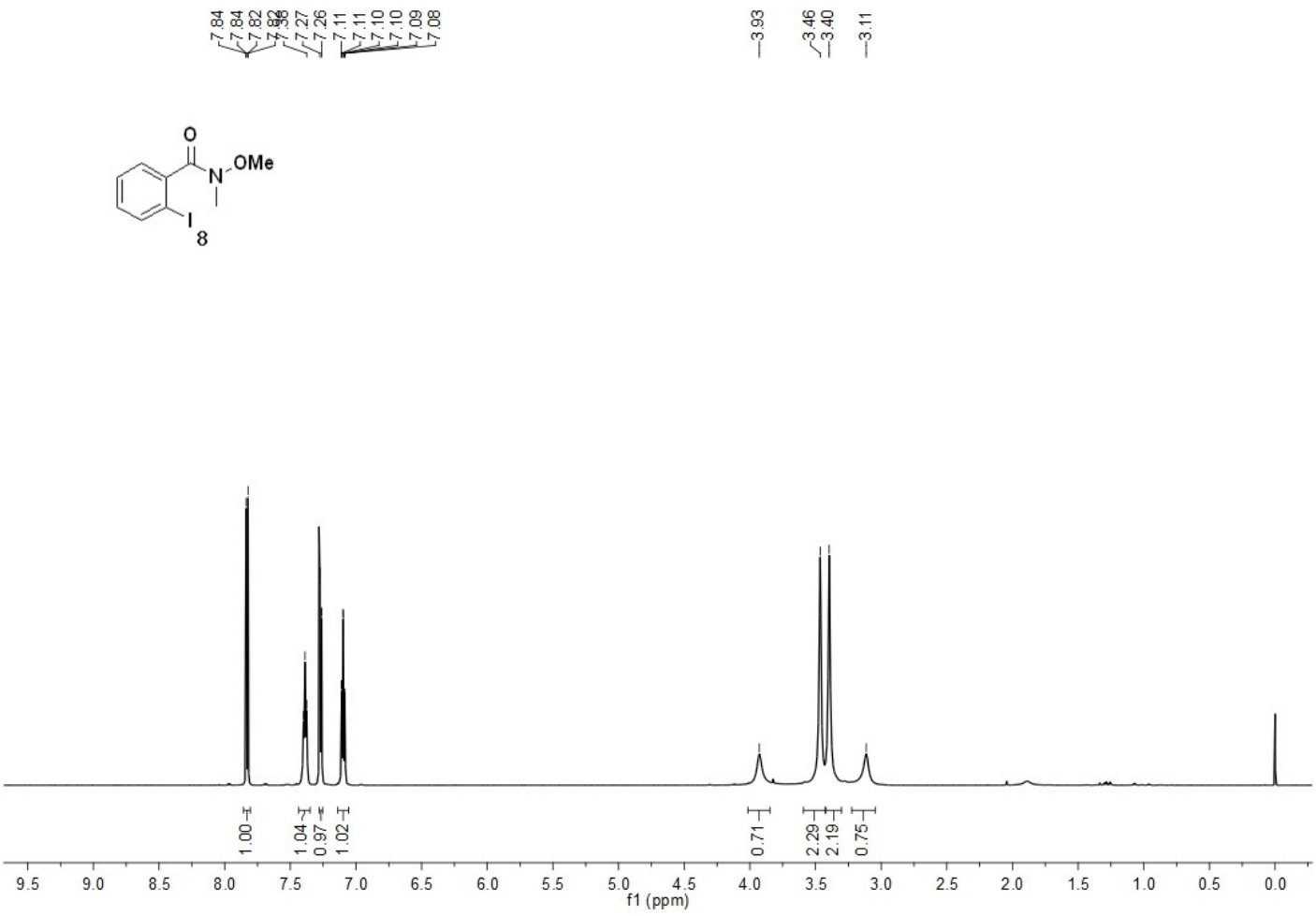
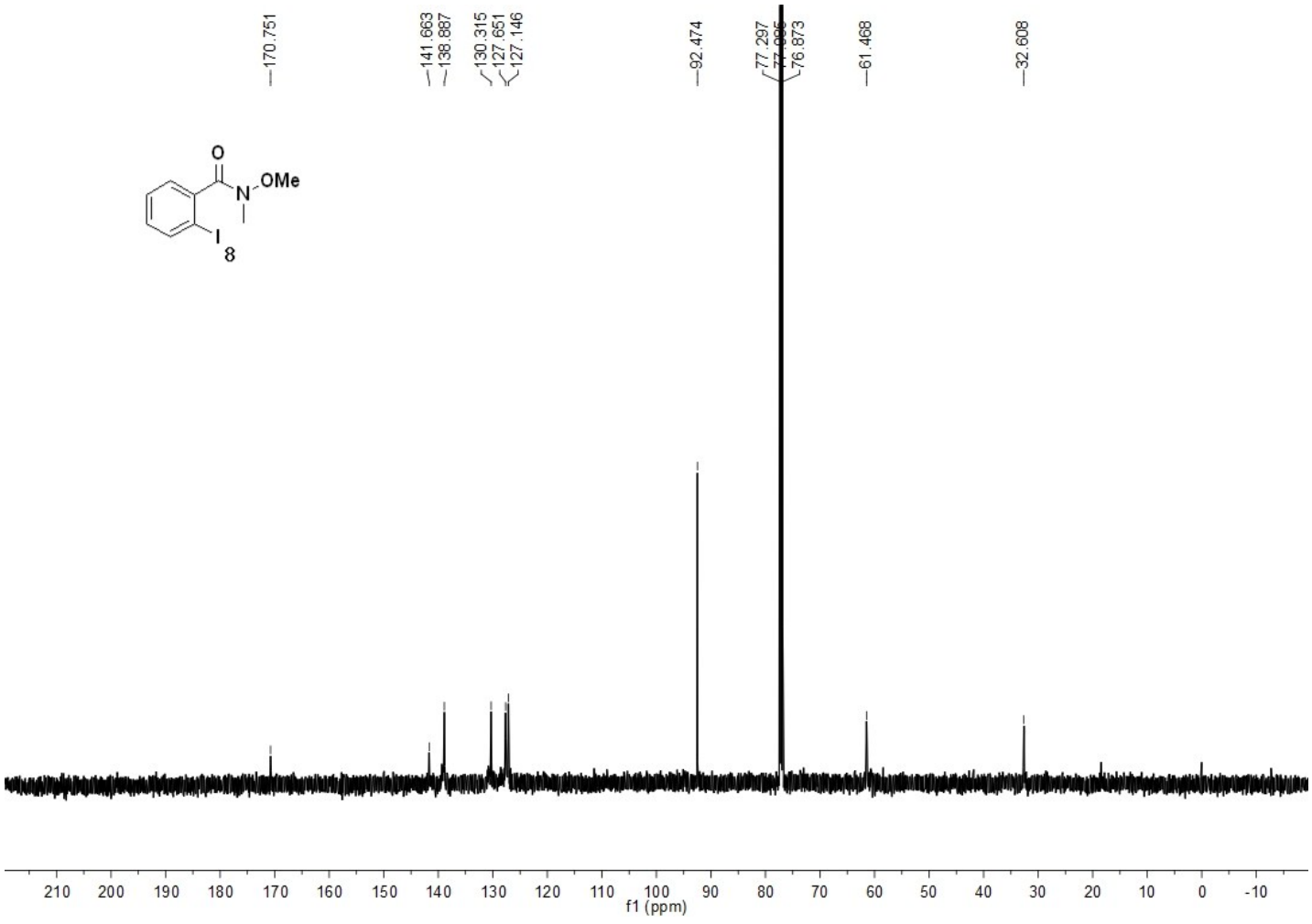
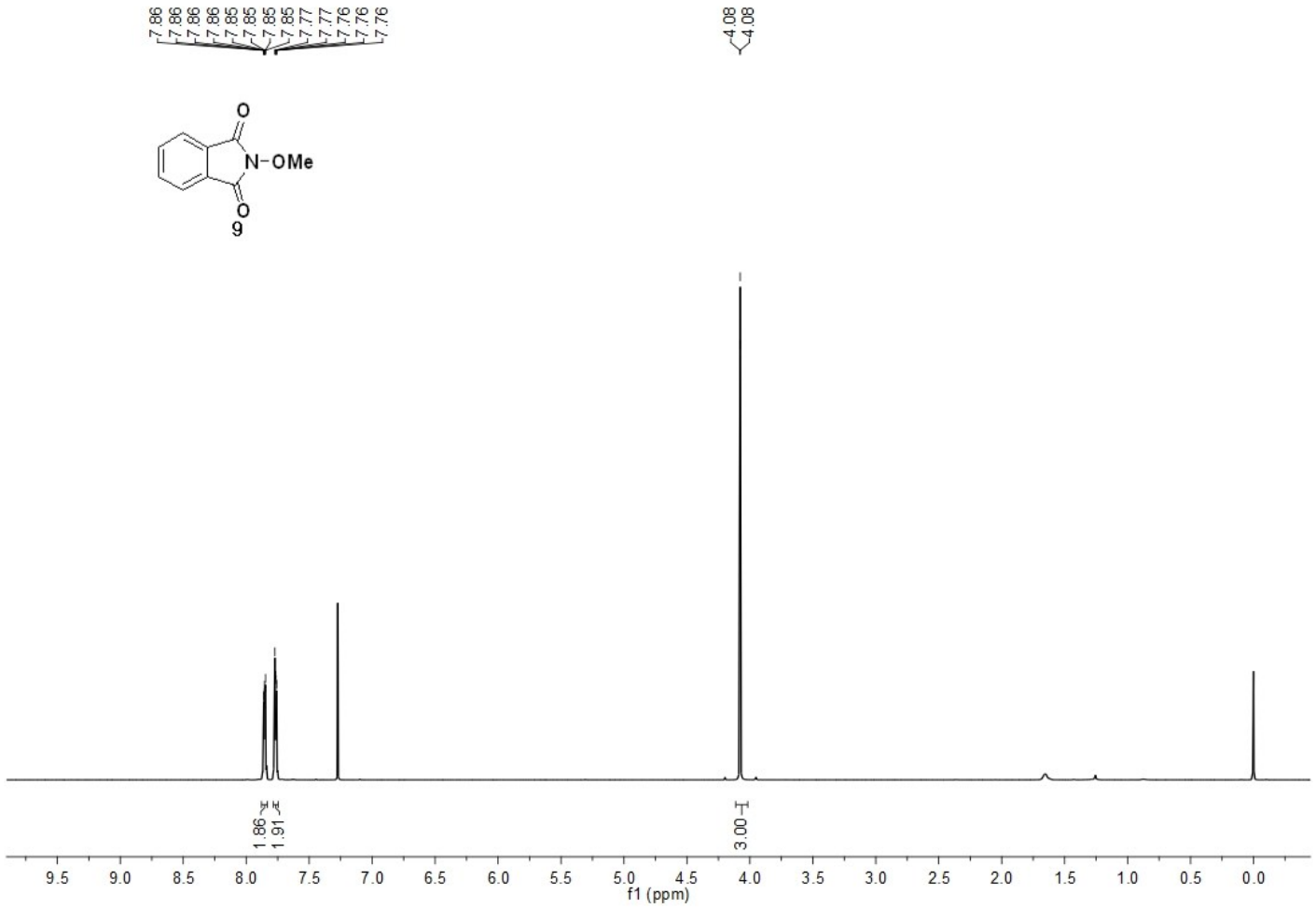
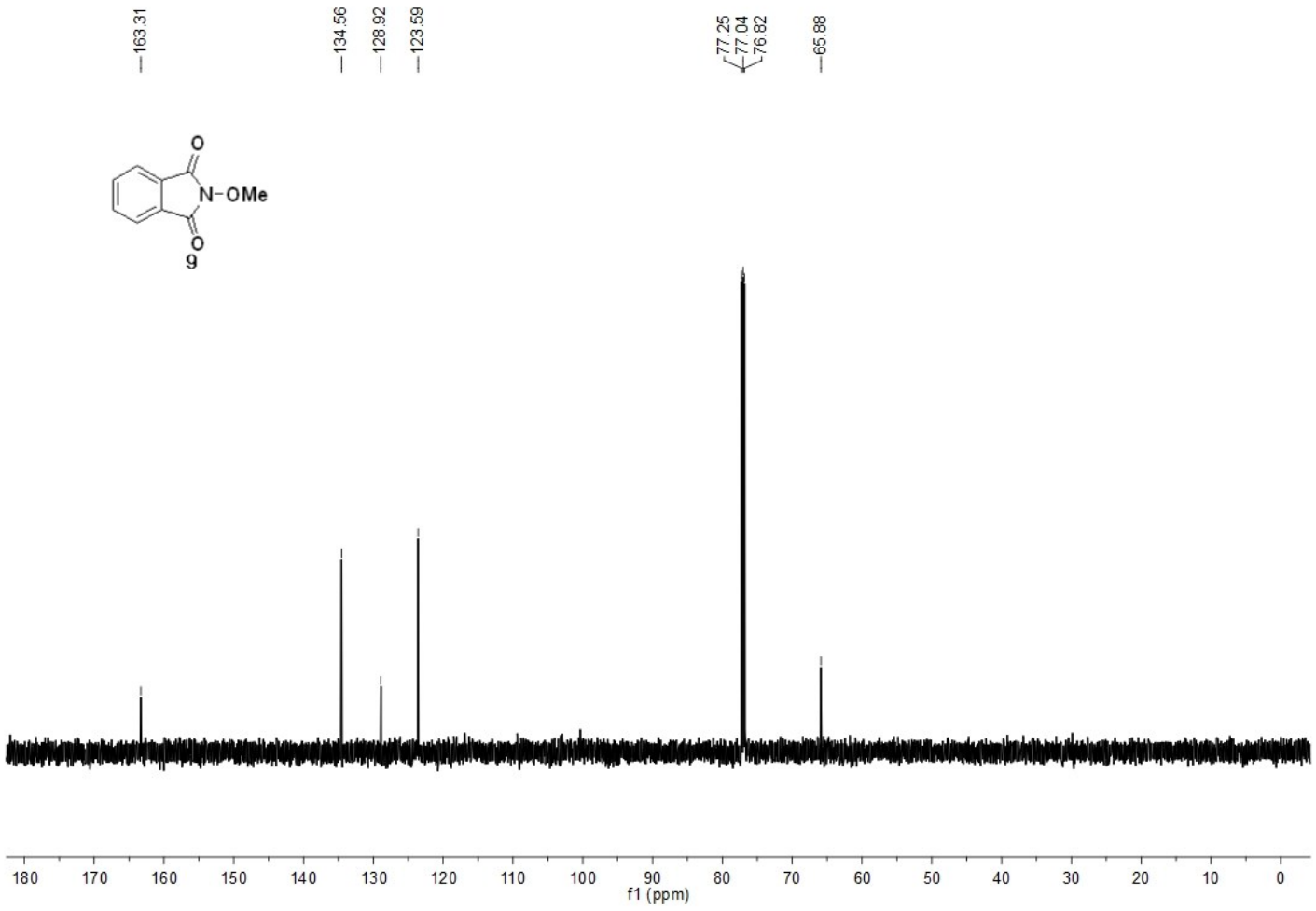
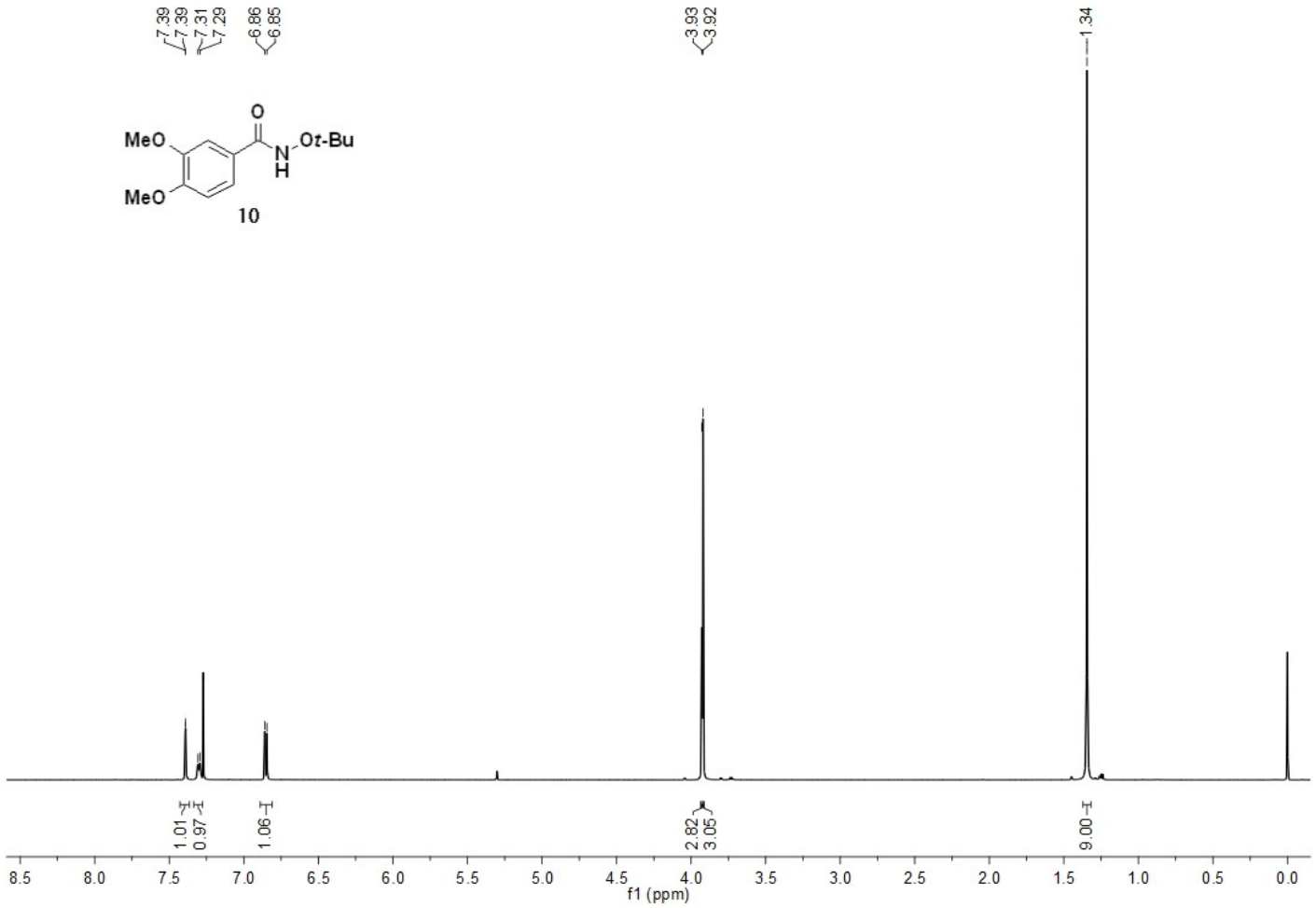
1.71
1.70
1.68
1.67
1.39
1.37
0.90
0.89
0.88
0.87
O
NH2
nBu
7b
1.00
0.96
1.02
1.02
1.01
0.99
0.94
0.94
2.01
2.04
2.06
3.03
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

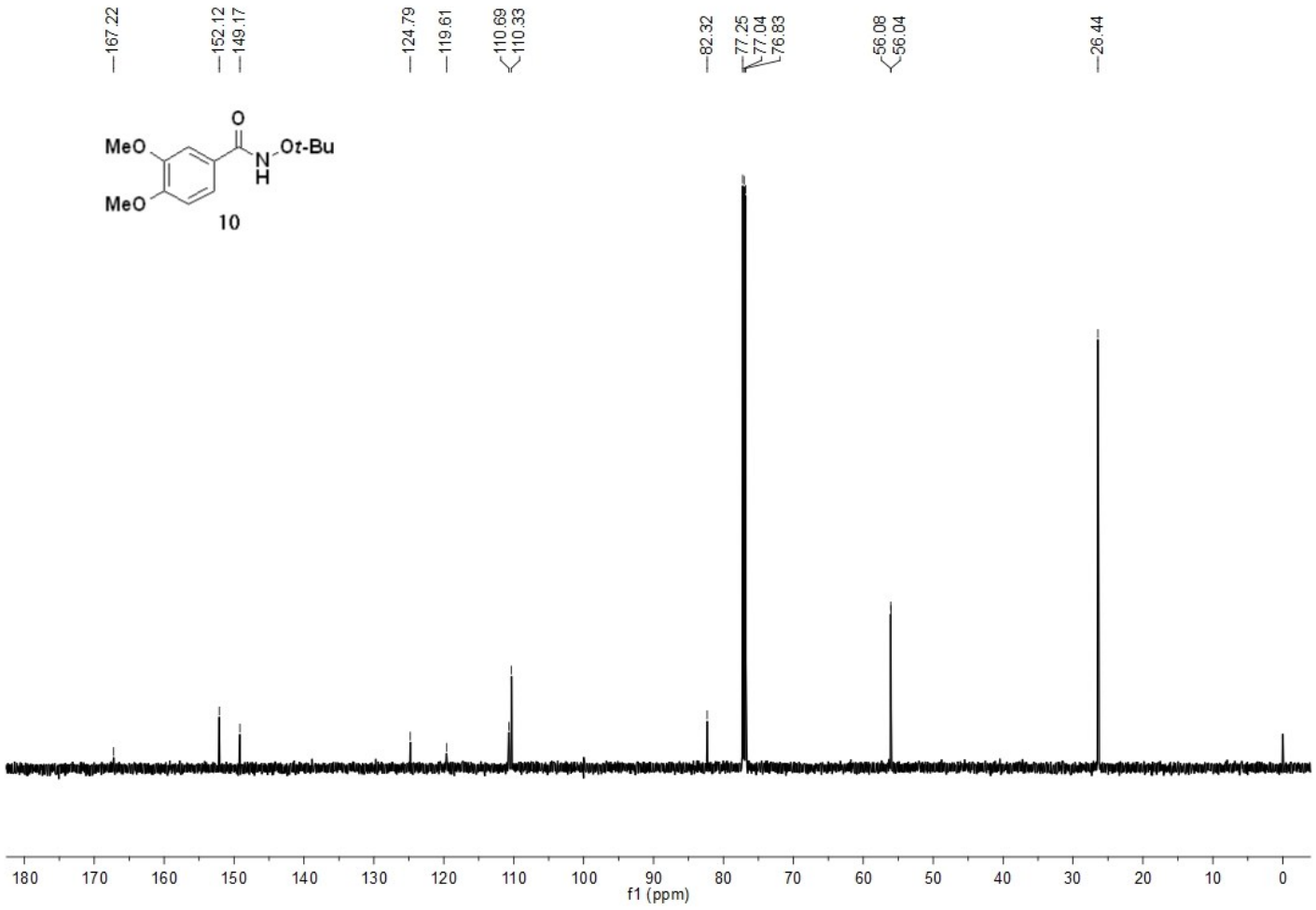

7.84
7.84
7.82
7.82
7.27
7.26
7.11
7.11
7.10
7.10
7.09
7.08
3.93
3.46
3.40
3.11
O
N
OMe
I
8
1.00
1.04
0.97
1.02
0.71
2.29
2.19
0.75
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

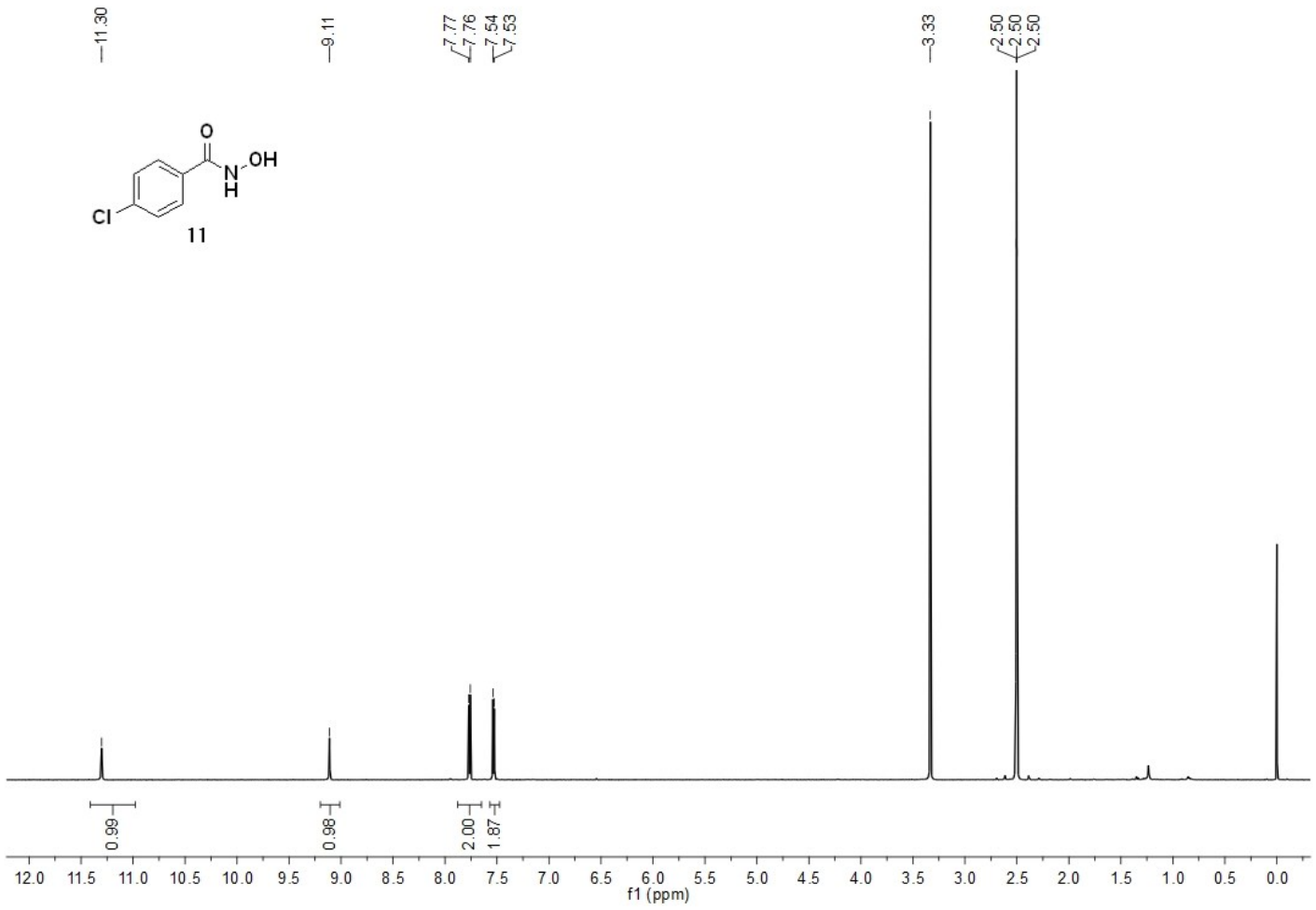

170.751
141.663
138.887
130.315
127.651
127.146
92.474
77.297
76.873
61.468
32.608
O
N
OMe
I
8
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

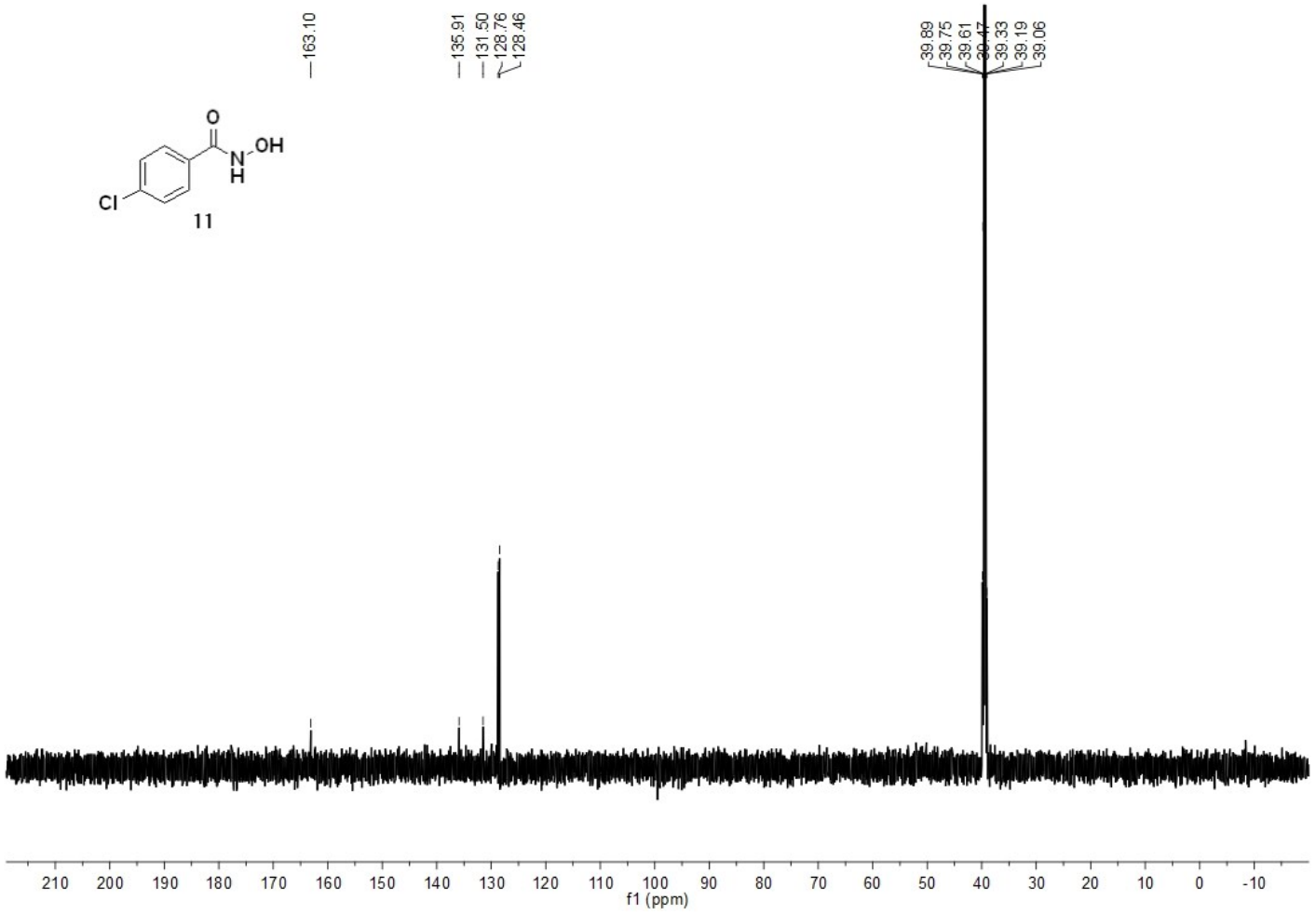

7.86
7.86
7.86
7.86
7.85
7.85
7.85
7.85
7.77
7.77
7.76
7.76
7.76
4.08
4.08
O
N-OMe
O
9
1.86
1.91
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

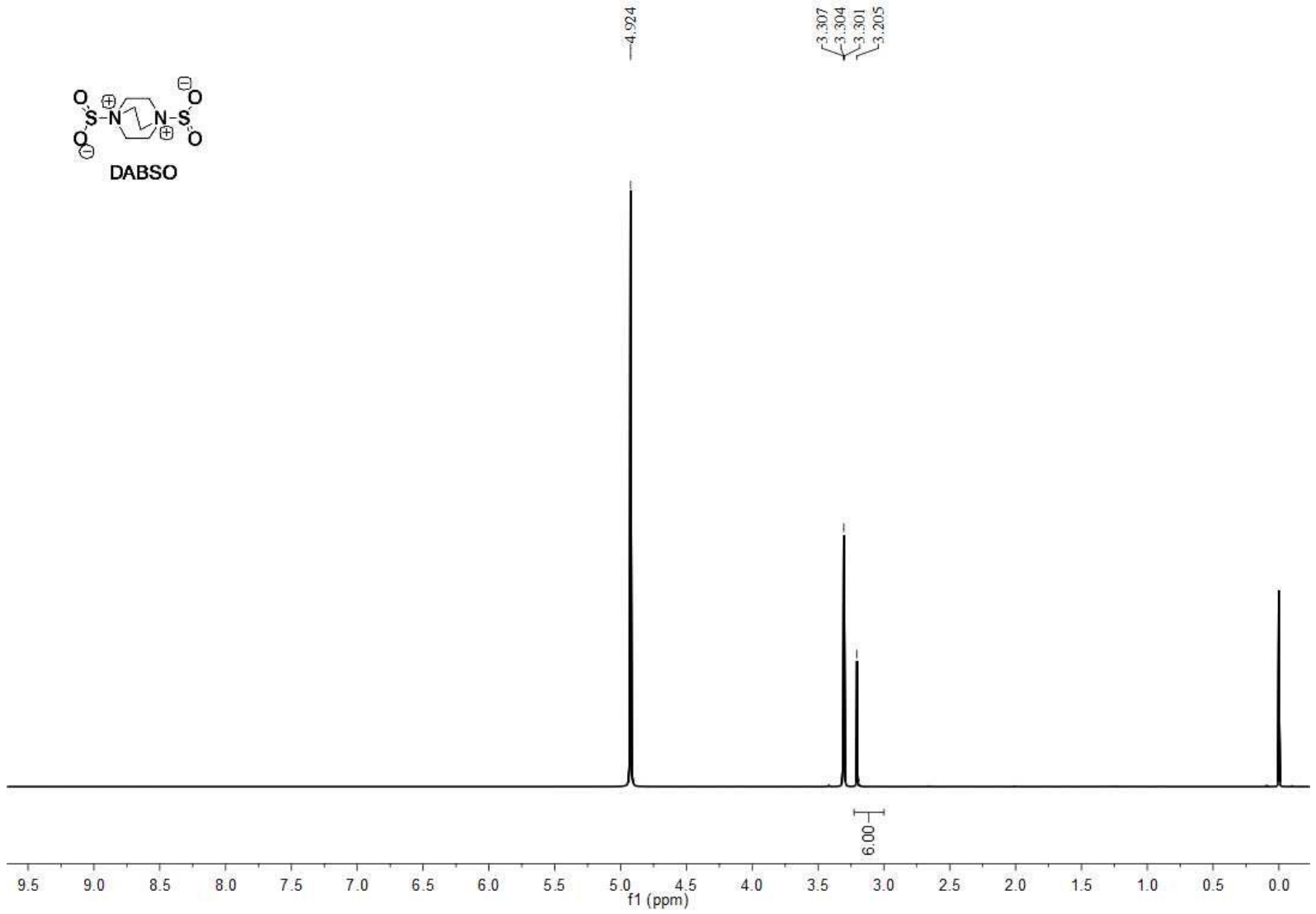

163.31
134.56
128.92
123.59
77.25
77.04
76.82
65.88
O
N-OMe
O
9
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

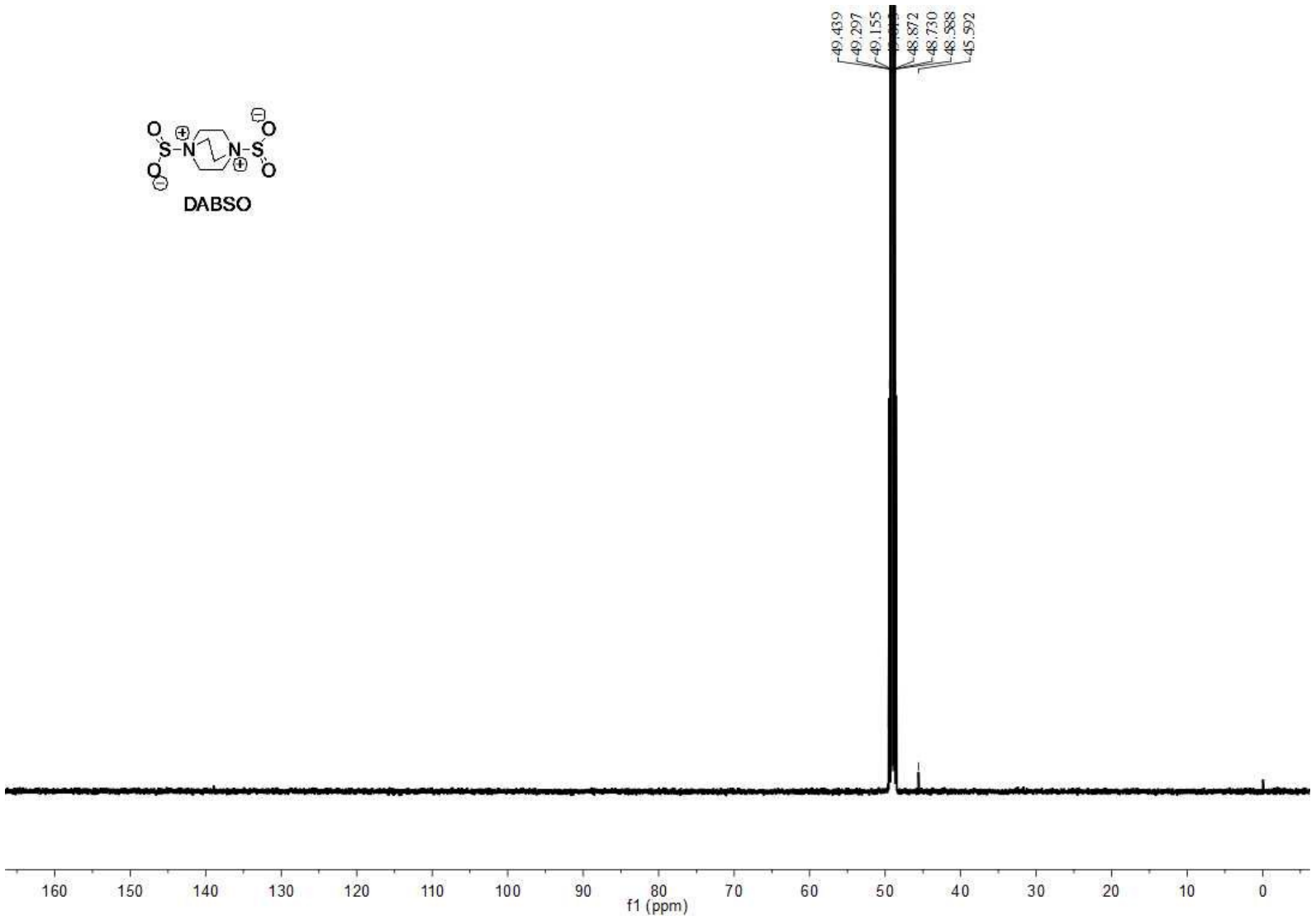

7.39
7.39
7.31
7.29
6.86
6.85
3.93
3.92
1.34
MeO
MeO
O
N
H
Ot-Bu
10
1.01
0.97
1.06
2.82
3.05
9.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


167.22
152.12
149.17
124.79
119.61
110.69
110.33
82.32
77.25
77.04
76.83
56.08
56.04
26.44
MeO
MeO
O
N
H
Ot-Bu
10
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


11.30
9.11
7.77
7.76
7.54
7.53
3.33
2.50
2.50
2.50
O
N
OH
H
Cl
11
0.99
0.98
2.00
1.87
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)


163.10
135.91
131.50
128.76
128.46
39.89
39.75
39.61
39.47
39.33
39.19
39.06
O
N
OH
H
Cl
11
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


DABSO
4.924
3.307
3.304
3.301
3.205
6.00
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)


49.439
49.297
49.155
48.872
48.730
48.588
45.992
DABSO
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

IR Spectrum of DABSO