
Supplementary Material (ESI) for Organic & Biomolecular Chemistry

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Supporting Information

Synthesis of Imides via Palladium–Catalyzed Decarboxylative Amidation of α-Oxocarboxylic Acids with Secondary Amides

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Table of Contents for Supporting Information

1. General considerations

All ^{1}H NMR and ^{13}C NMR spectra were recorded on a 400 MHz Bruker FT-NMR spectrometers (400 MHz and 100 MHz respectively). All chemical shifts are given as δ value (ppm) with reference to tetramethylsilane (TMS) as an internal standard. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. The coupling constants, J, are reported in Hertz (Hz). High resolution mass spectroscopy data of the product were collected on an Agilent Technologies 6540 UHD Accurate-Mass Q-TOF LC/MS (ESI).

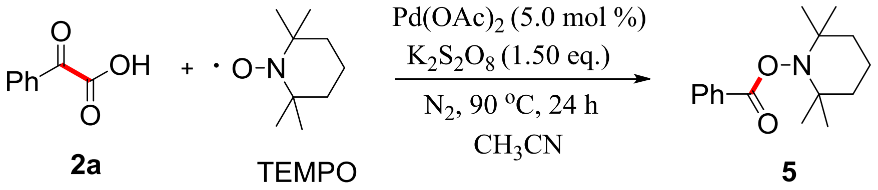
N-Substituted *N*-heteroarene-2-carboxamides were prepared from benzylamines and picolinic acid, according to reported method (L. D. Tran, J. Roane, O. Daugulis, *Angew. Chem.* **2013**, *125*, 6159–6123; *Angew. Chem. Int. Ed.,* **2013**, *52*, 6043–6026). α-Keto acids were prepared according to the literature (P. Fang, M. Li, H. Ge, *J. Am. Chem. Soc.,* **2010**, *132*,11898–11899 and L. Yu, P. Li, L. Wang, *Chem. Commun.* **2013**, *49*, 2368–2370). The chemicals and solvents were purchased from commercial suppliers either from Aldrich (USA) or Shanghai Chemical Company (China) without further purification. All the solvents were dried and freshly distilled prior to use. Products were purified by flash chromatography on 200–300 mesh silica gels, SiO_2.

2. Typical procedure for the decarboxylative amidation of α-oxocarboxylic acids with secondary amides

A 10 mL of reaction tube equipped with a stir bar was charged with a mixture of *N*-benzylpicolinamide (**1a**, 42.4 mg, 0.20 mmol), 2-oxo-2-phenylacetic acid (**2a**, 36 mg, 0.24 mmol), $Pd(OAc)_2$ (4.48 mg, 0.02 mmol), potassium persulfate (81 mg, 0.30 mmol) and acetonitrile (CH_3CN, 2.0 mL). After the reaction mixture was stirred at 90 ^{o}C for 24 h, it was cooled to room temperature, extracted with EtOAc (2×5.0 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated to yield the crude product, which was further purified by flash chromatography (silica gel, ethyl acetate/petroleum ether 5:1, v/v), affording the desired product **3a** as a white solid (50.6 mg, 80%).

3. Preliminary mechanistic study

(1) Free radical-trapping experiment



A mixture 2-oxo-2-phenylacetic acid (**2a**, 30 mg, 0.20 mmol), TEMPO (93.6 mg, 0.60 mmol) Pd(OAc)$_2$ (4.48 mg, 0.02 mmol), potassium persulfate (81 mg, 0.30 mmol) were dissolved in acetonitrile (CH_3CN, 2.0 mL) in a 10 mL oven-dried reaction vessel equipped with magnetic stirring bar. After the reaction mixture was stirred at 90 °C for 24 h, it was cooled to room temperature, extracted with EtOAc (2×5.0 mL). The organic layers were combined, dried over Na_2SO_4, and concentrated to yield the crude product, which was further purified by flash chromatography (silica gel, ethyl acetate/petroleum ether 1:30, v/v), affording the desired product **5** in 32% yield.

2,2,6,6-Tetramethylpiperidin-1-yl benzoate (**5**): White solid (H. Tan, H. Li, W. Ji and L. Wang, *Angew. Chem., Int. Ed.,* 2015, **54**, 8374). ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.14 (d, J = 7.4 Hz, 2H), 7.65–7.61 (m, 1H), 7.54–7.50 (m, 2H), 1.88–1.74 (m, 3H), 1.66–1.64 (m, 2H), 1.54–1.51 (m, 1H), 1.34 (s, 6H), 1.19 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.88, 131.41, 128.31, 128.09, 127.02, 58.92, 37.64, 30.52, 19.41, 15.58. HRMS (ESI) ([M+H]$^+$) Calcd. For $C_{16}H_{24}NO_2$: 262.1802, Found: 262.1797. Analysis of reaction mixture by HRMS was shown in Figure S1.

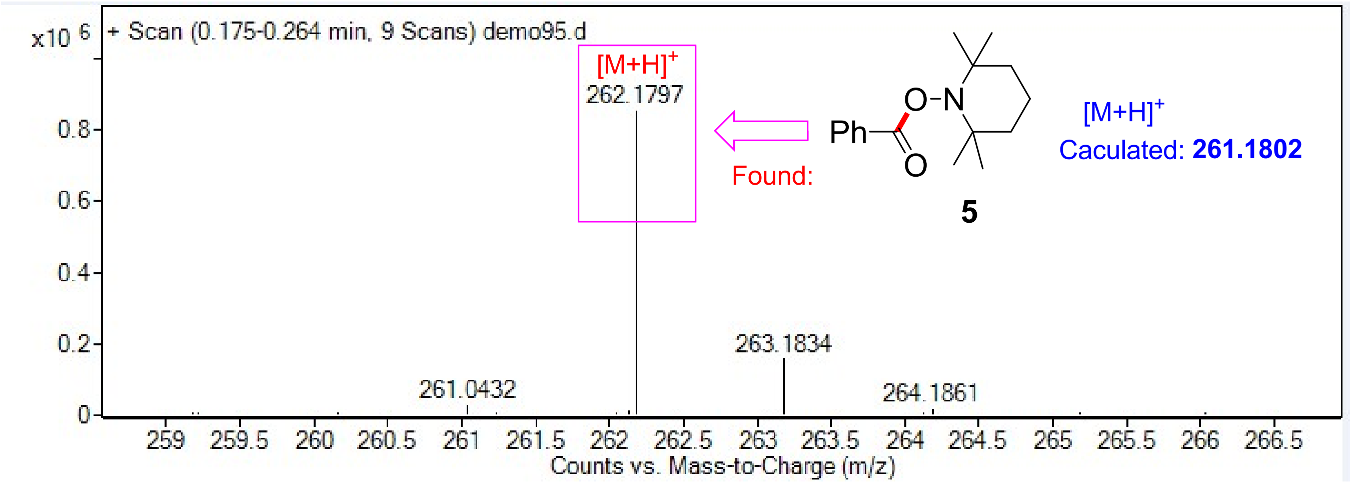
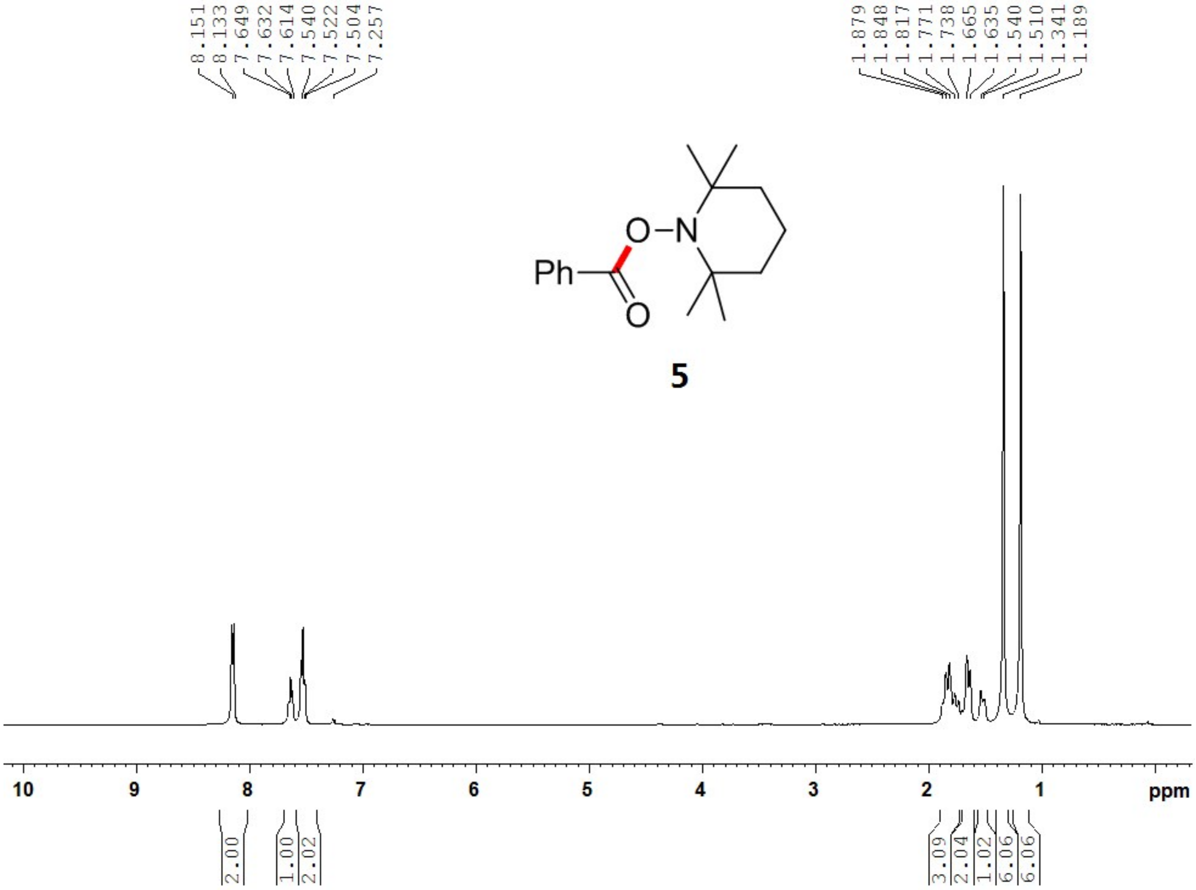


Figure S1. Analysis of reaction mixture by HRMS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7
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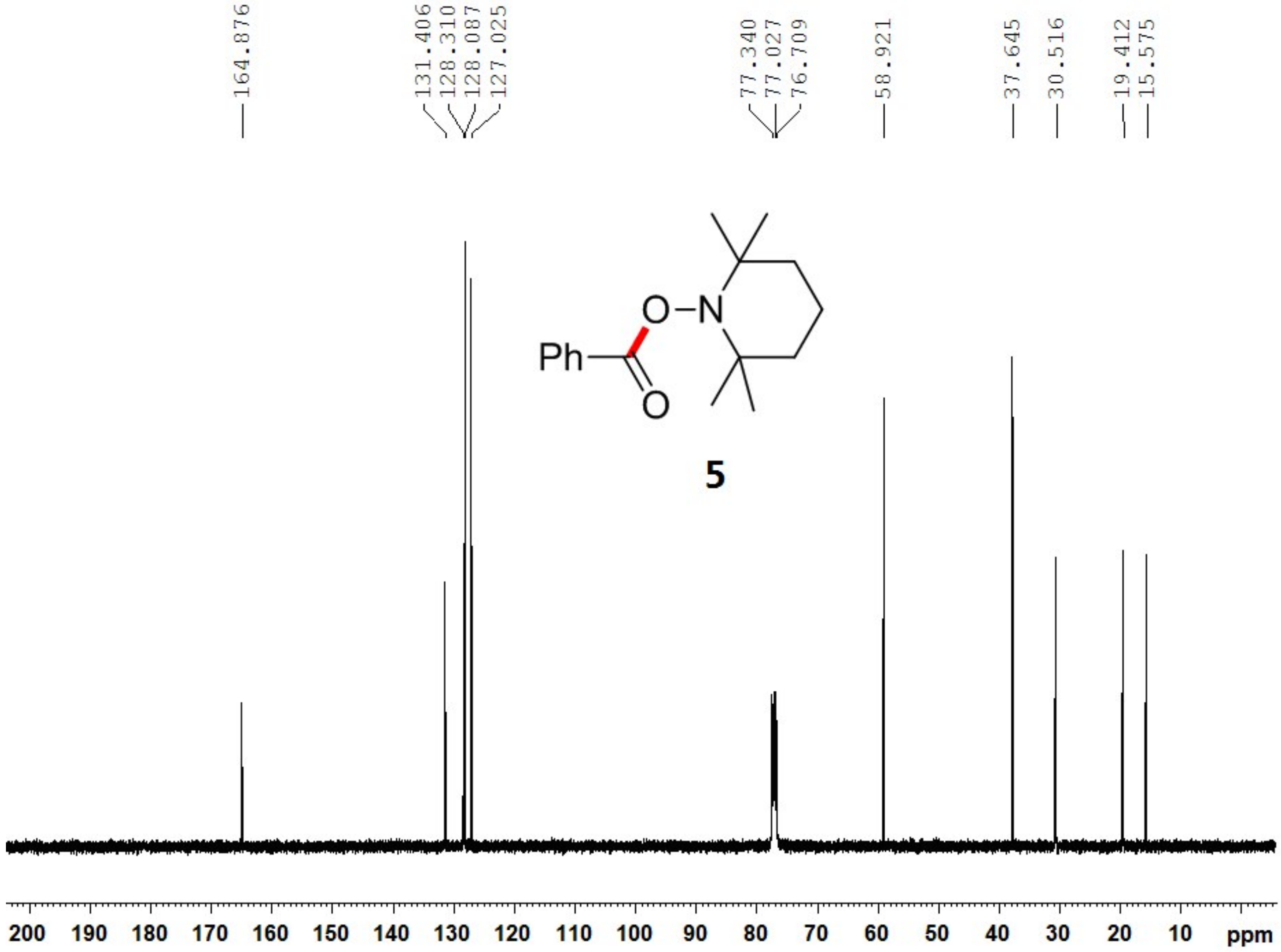
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150
140
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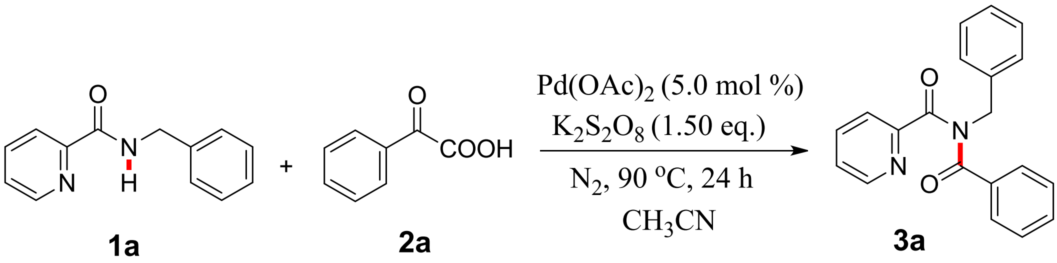
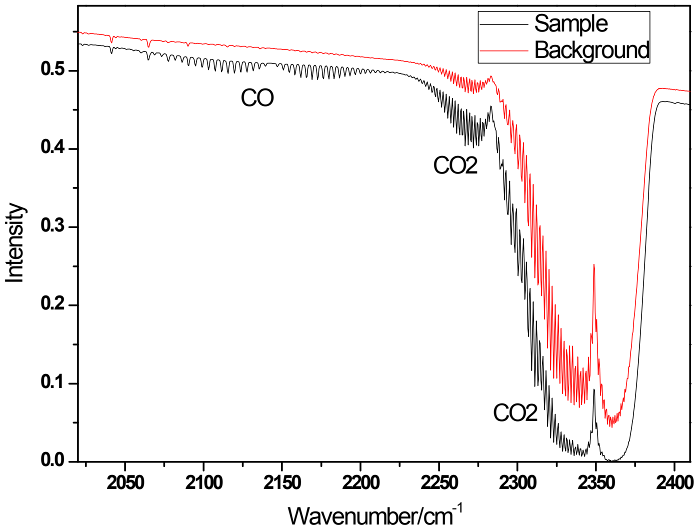
10
ppm

(2) Determination of CO_2 and CO during the reaction by FT-IR



An Schlenk tube equipped with a magnetic stirrer bar was charged with *N*-benzylpicolinamide (**1a**, 42.4 mg, 0.20 mmol), 2-oxo-2-phenylacetic acid (**2a**, 36 mg, 0.24 mmol), $Pd(OAc)_2$ (4.48 mg, 0.02 mmol), potassium persulfate (81 mg, 0.30 mmol) and acetonitrile (CH_3CN, 2.0 mL). After the reaction mixture was stirred at 90 °C for 24 h, the resulting gas from the reaction system was directly determined by a Bruker Tensor 27 FT-IR, and the concentration of CO_2 and CO was found to be 823.49 and 10.06 ppm, respectively (Figure S2 and Figure S3).



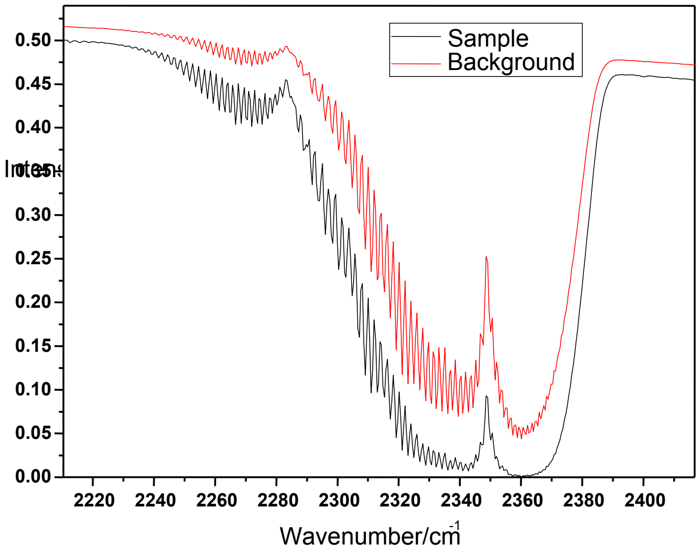


Figure S2. FT-IR analysis of the formation of CO_2 in the reaction

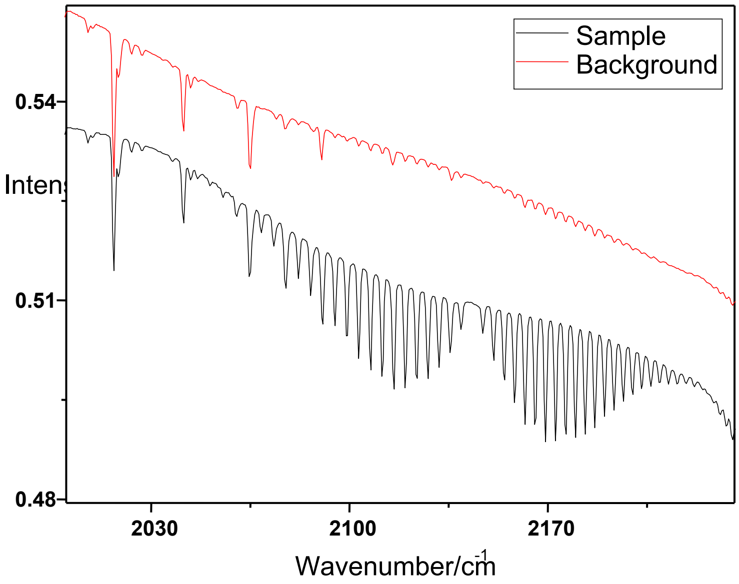
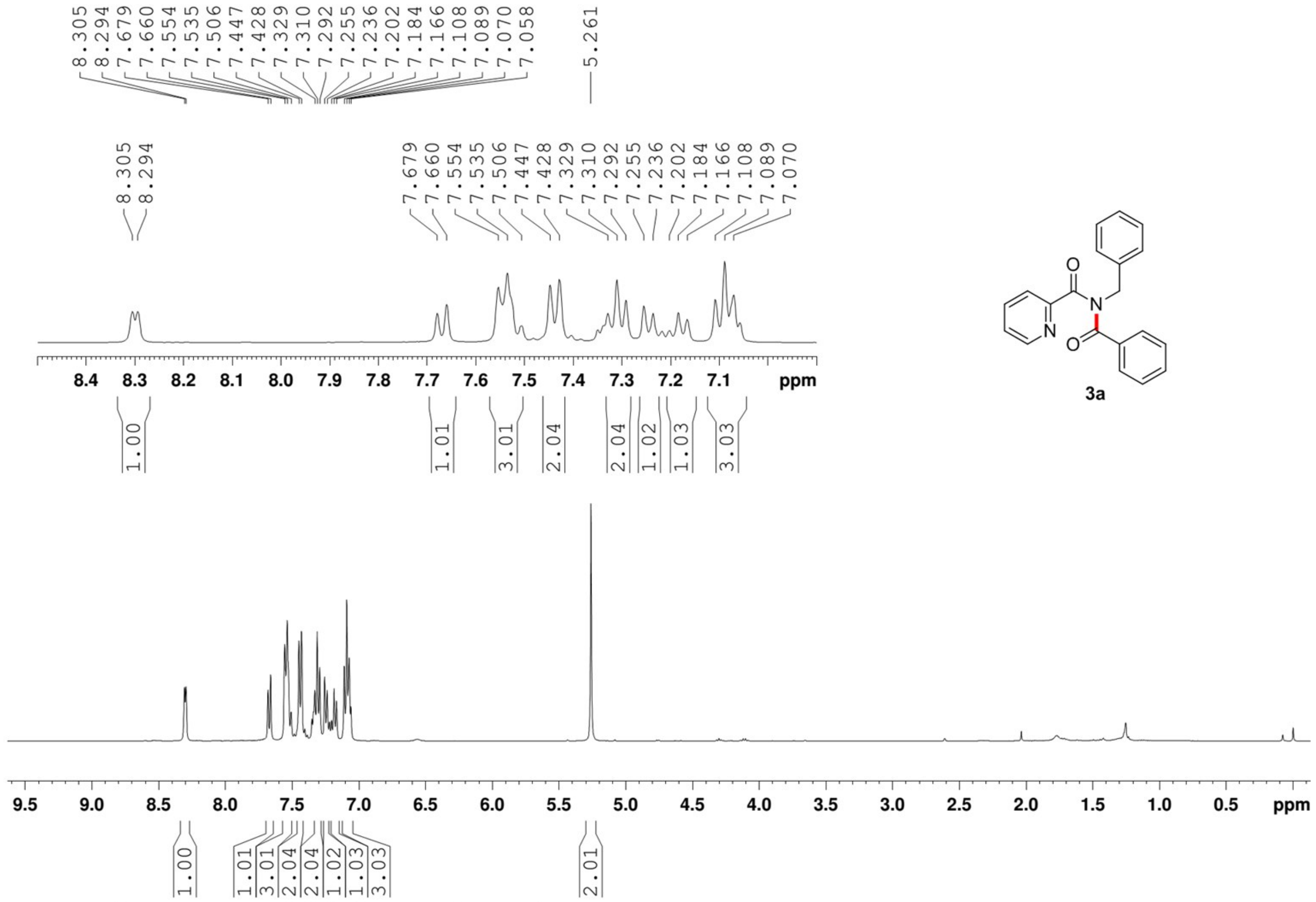
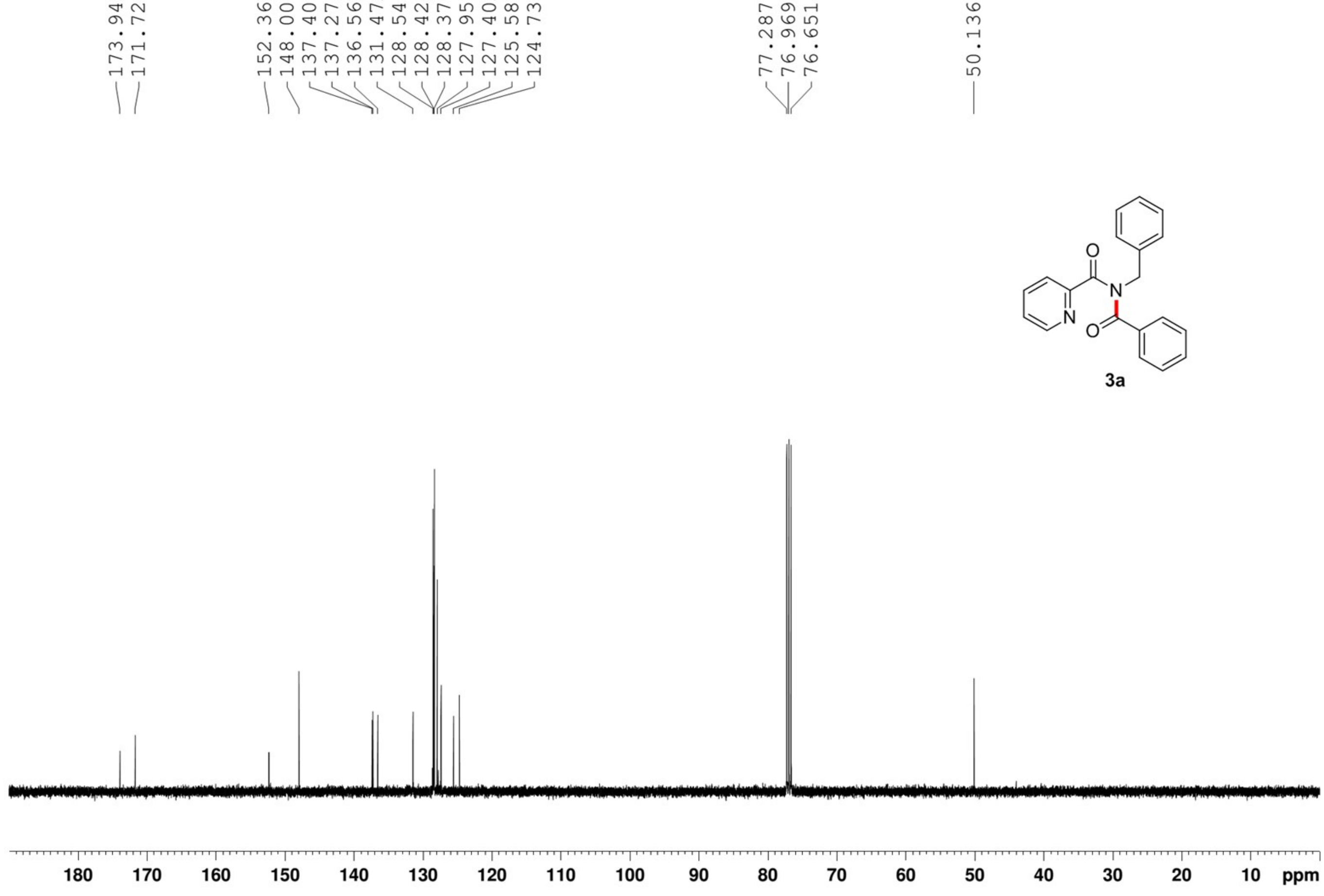
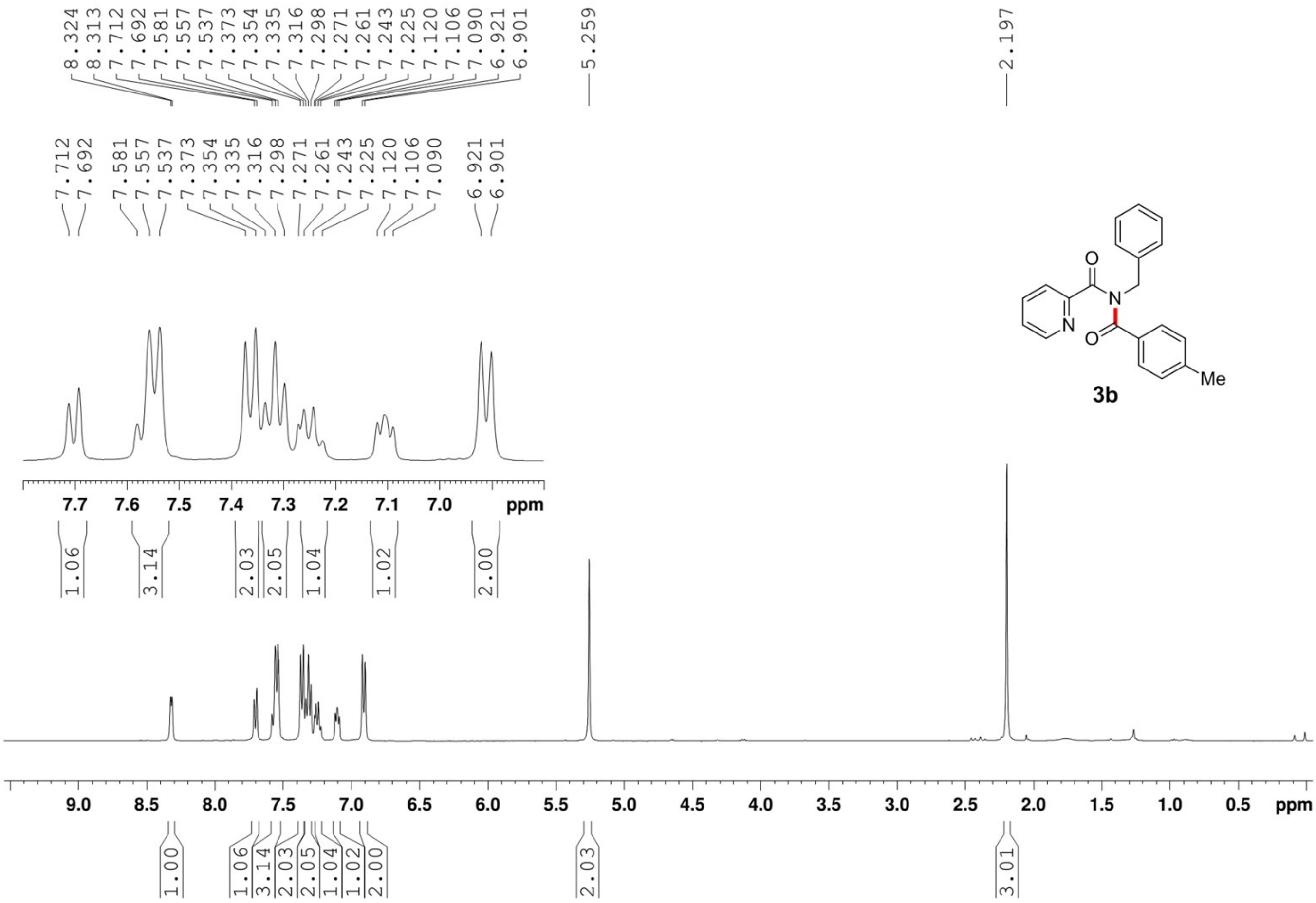


Figure S3. FT-IR analysis of the formation of CO in the reaction

4. ^{1}H and ^{13}C spectra of the products



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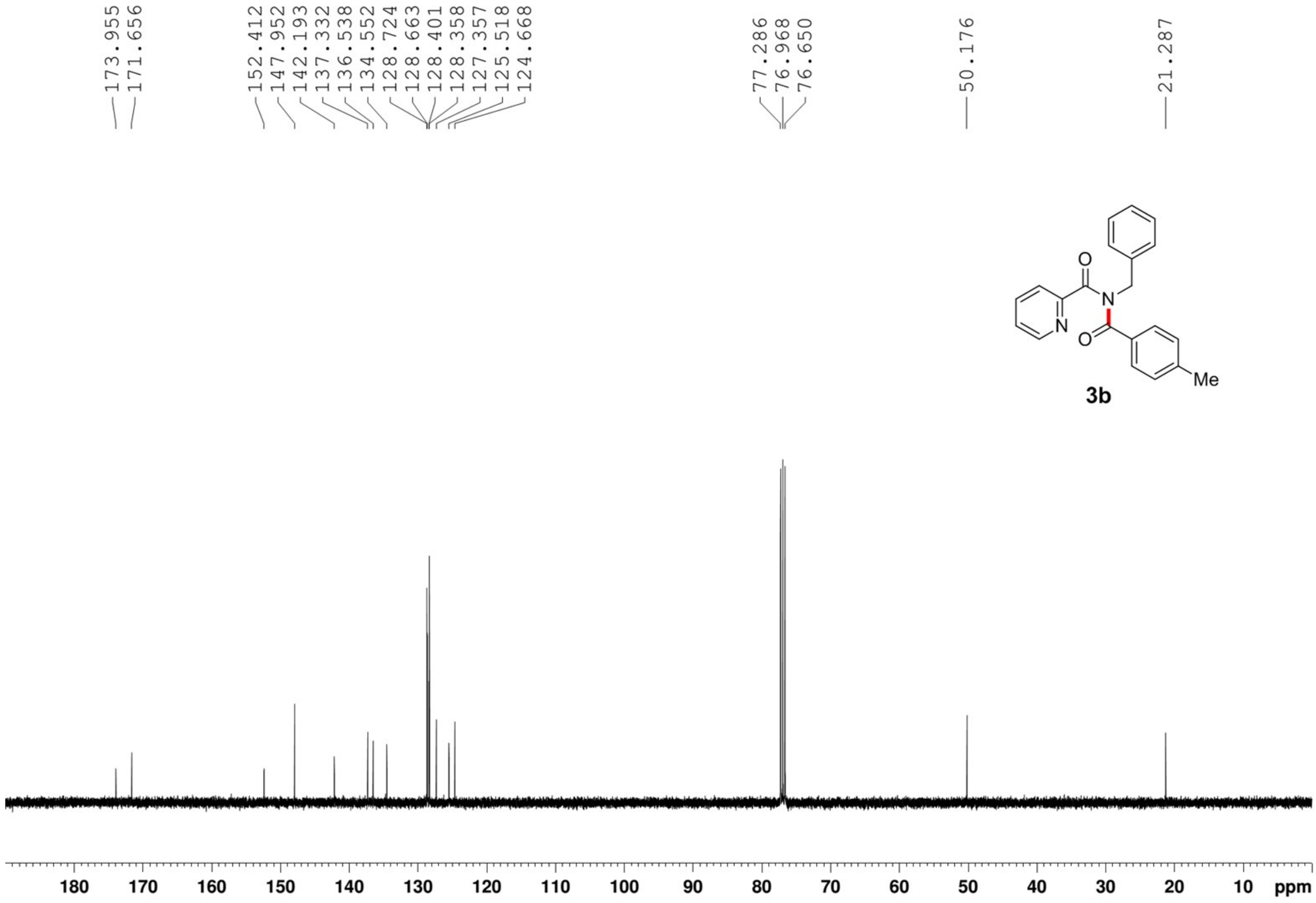
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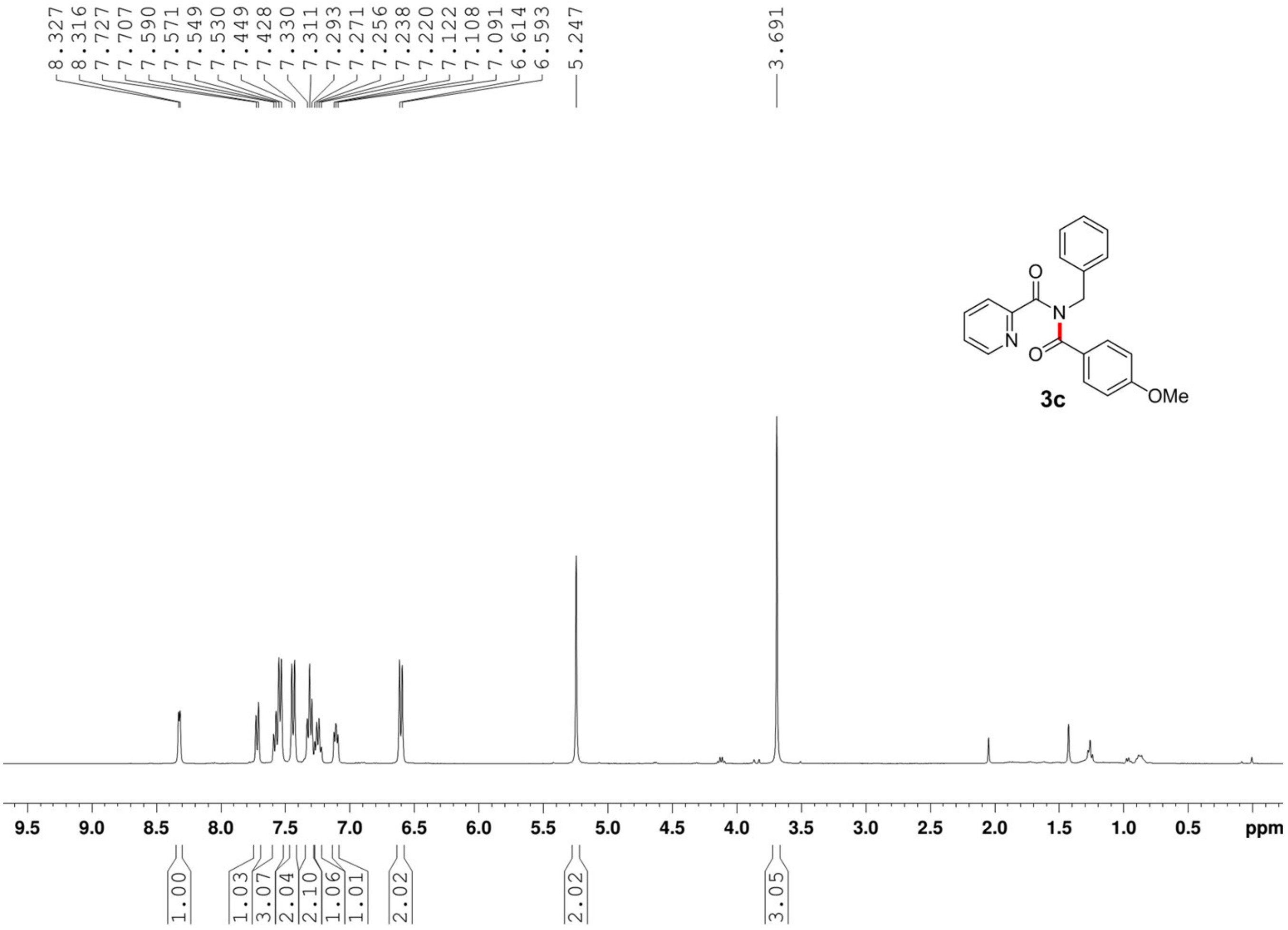
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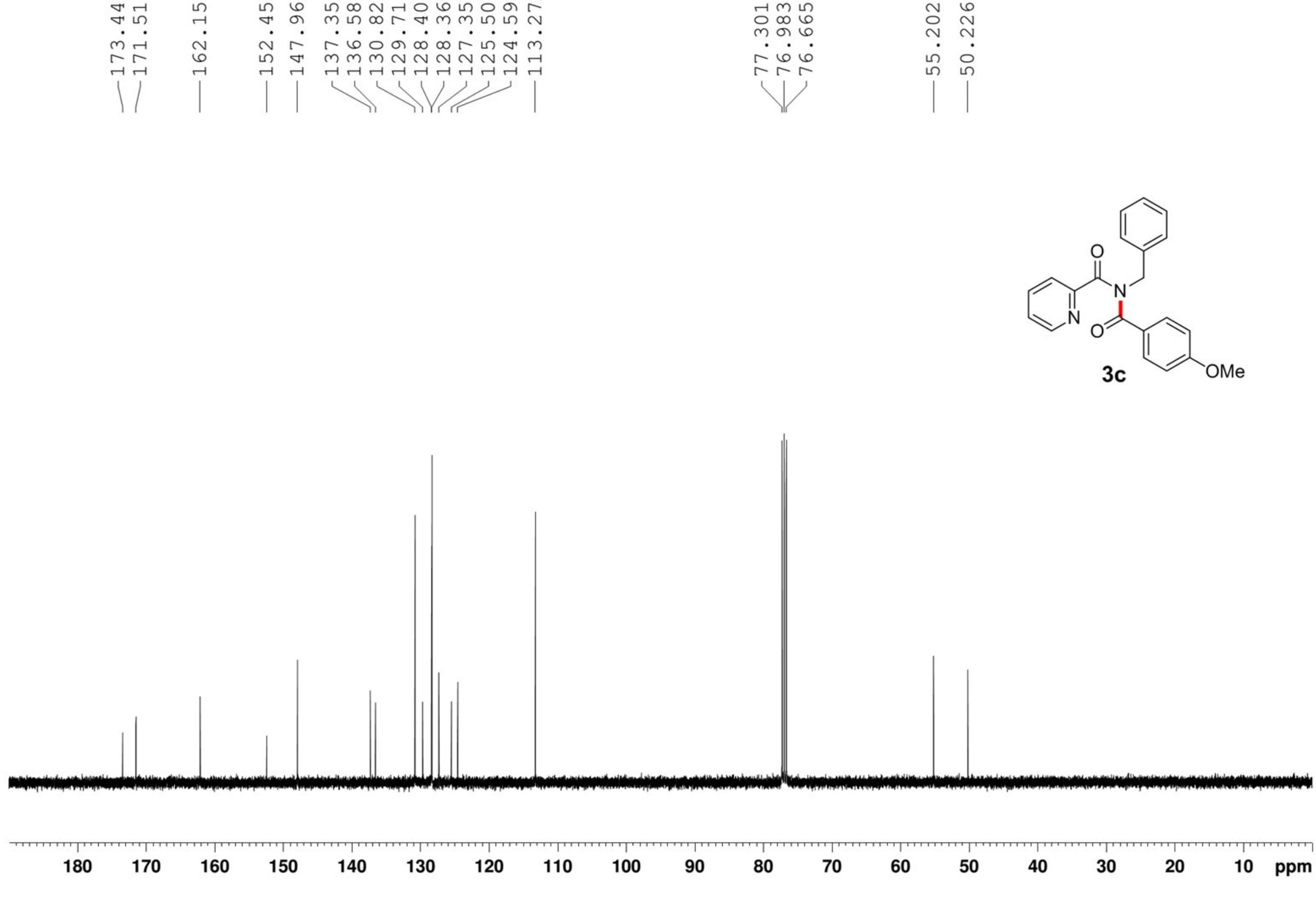
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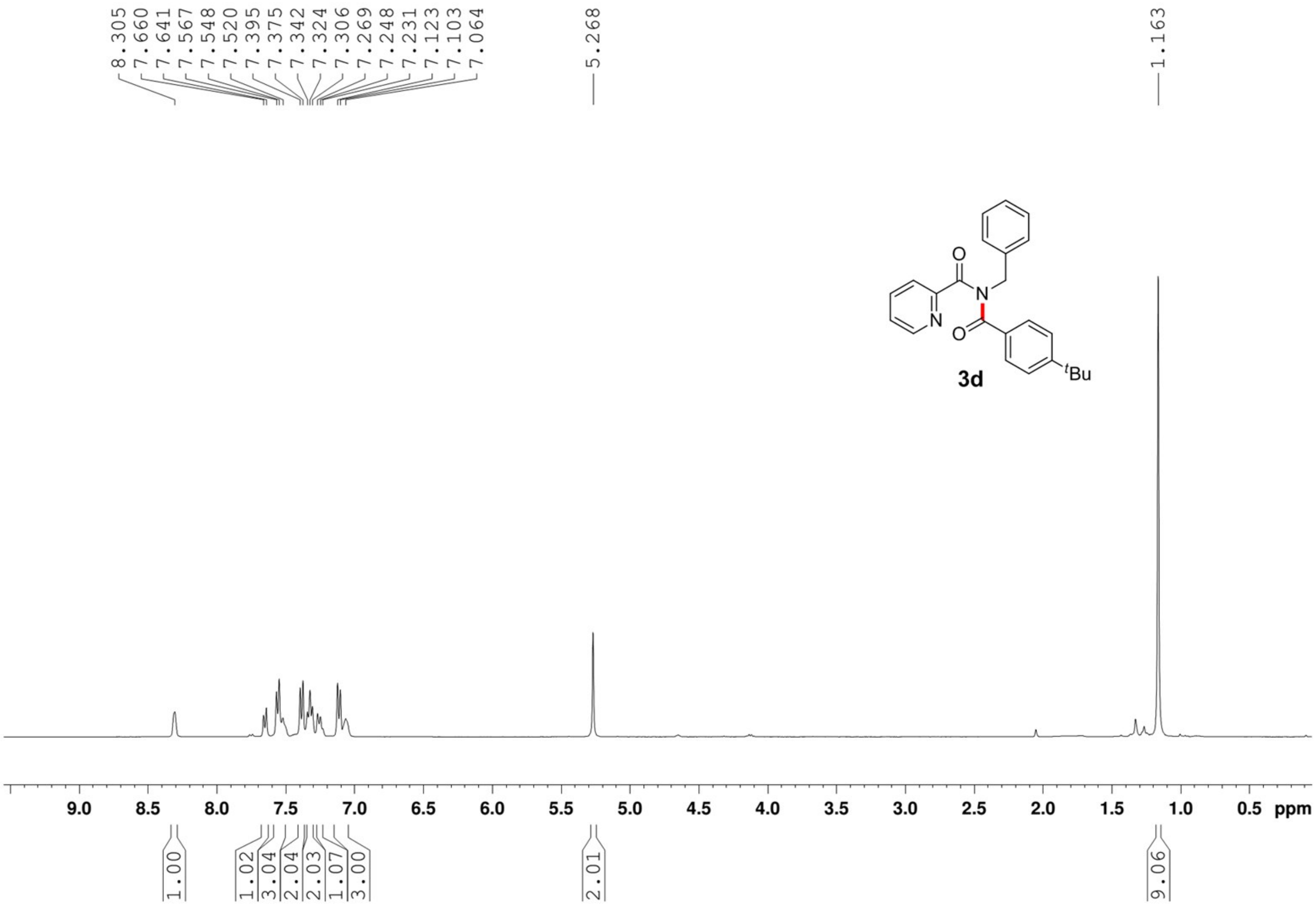
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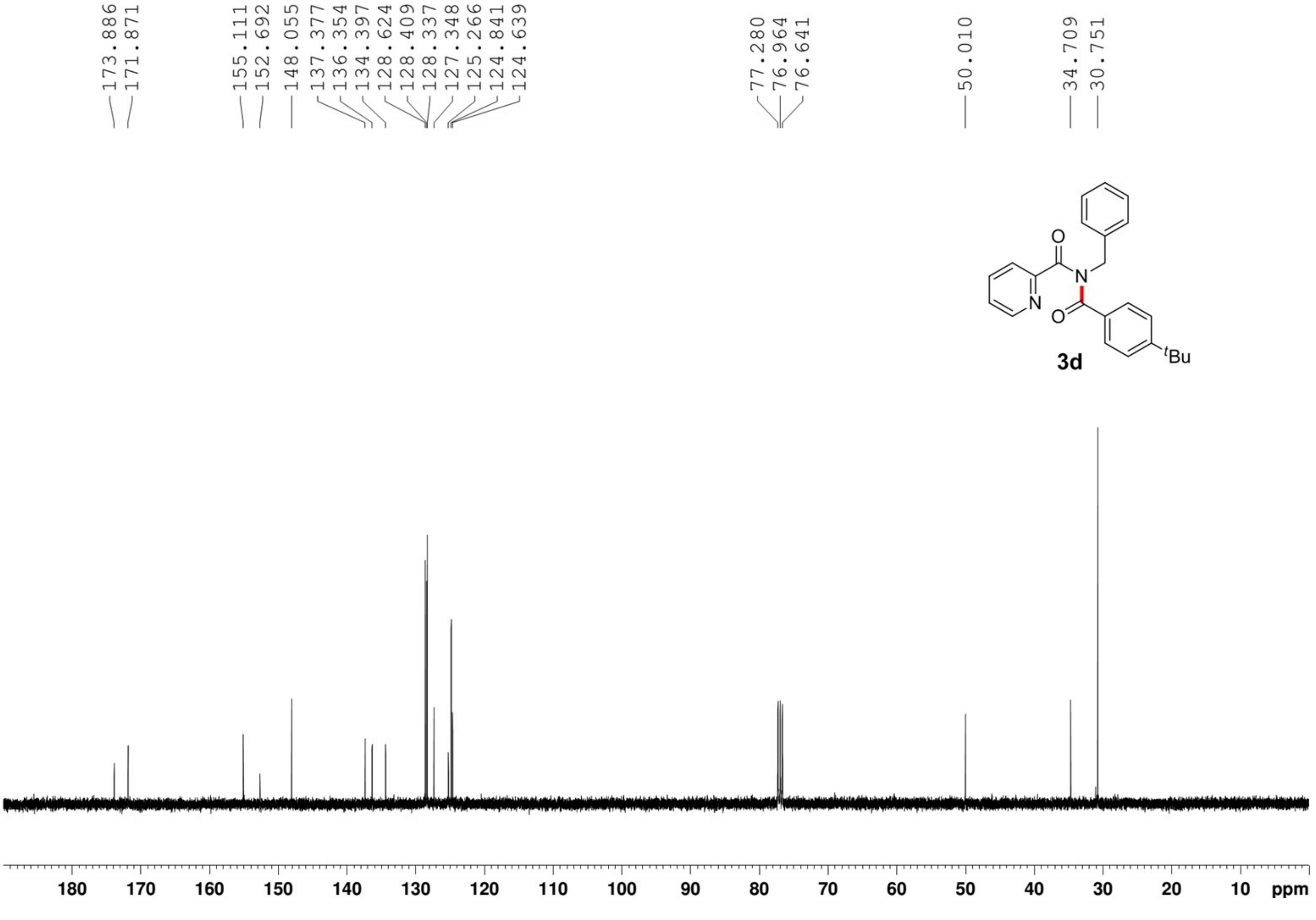
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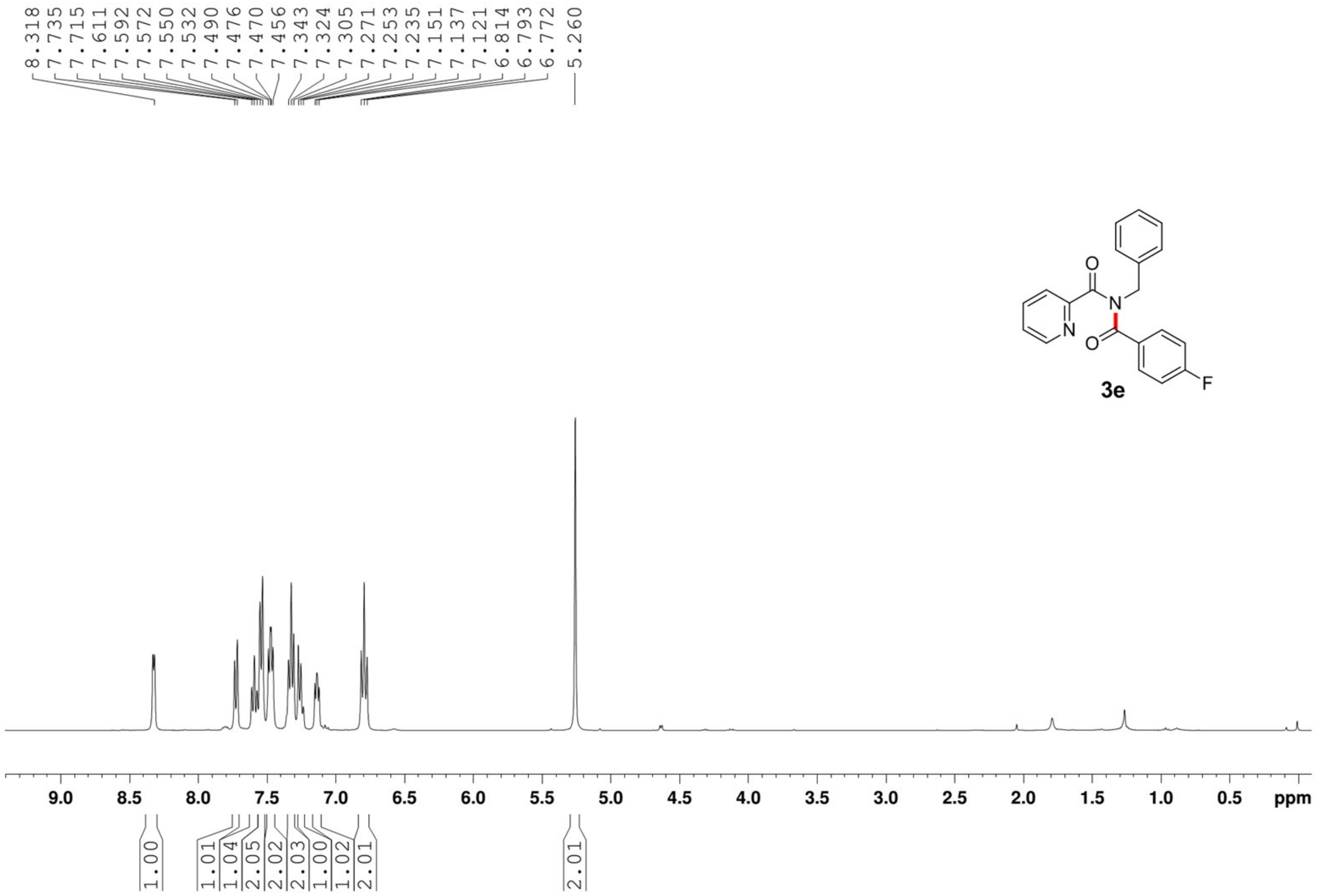
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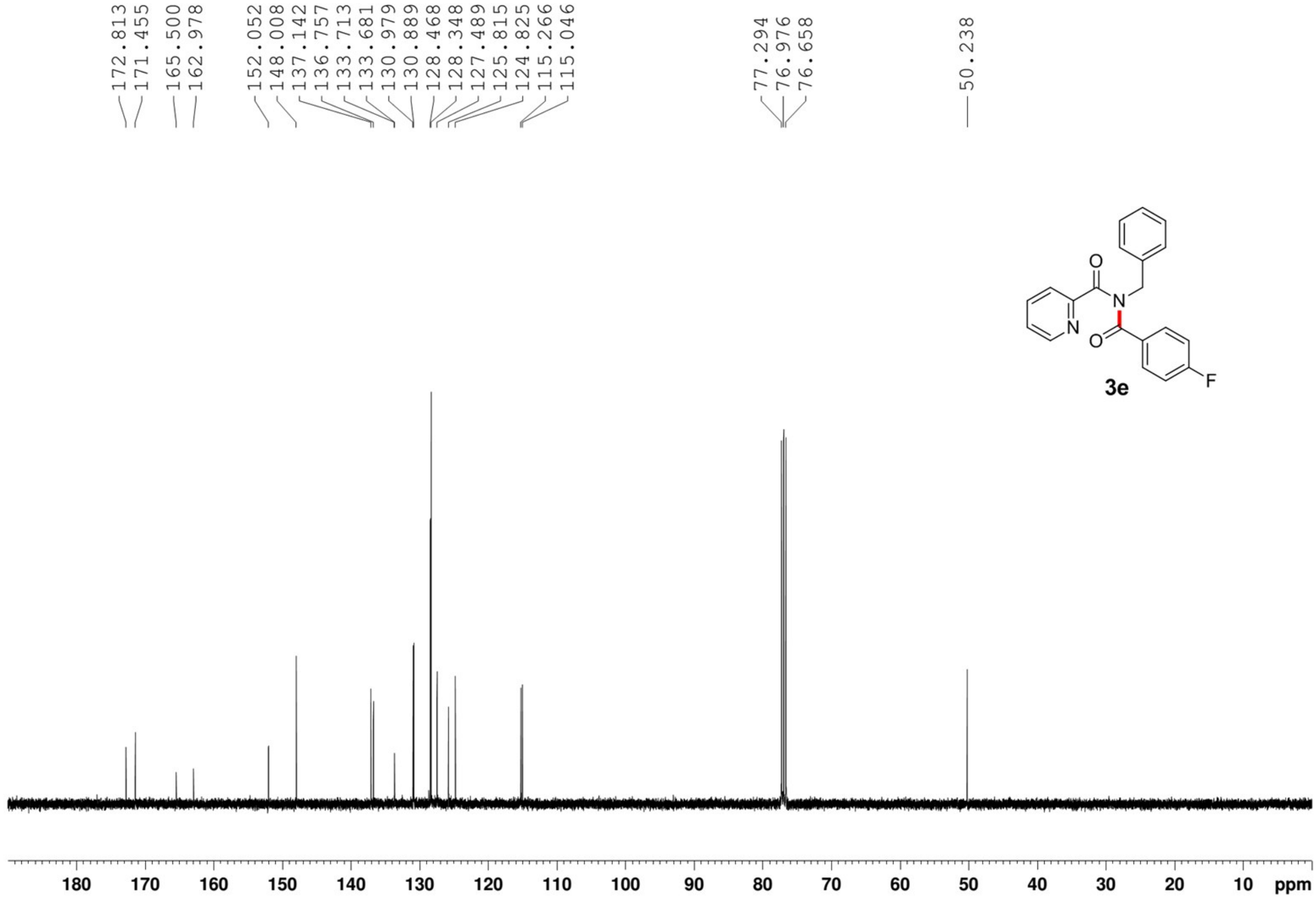
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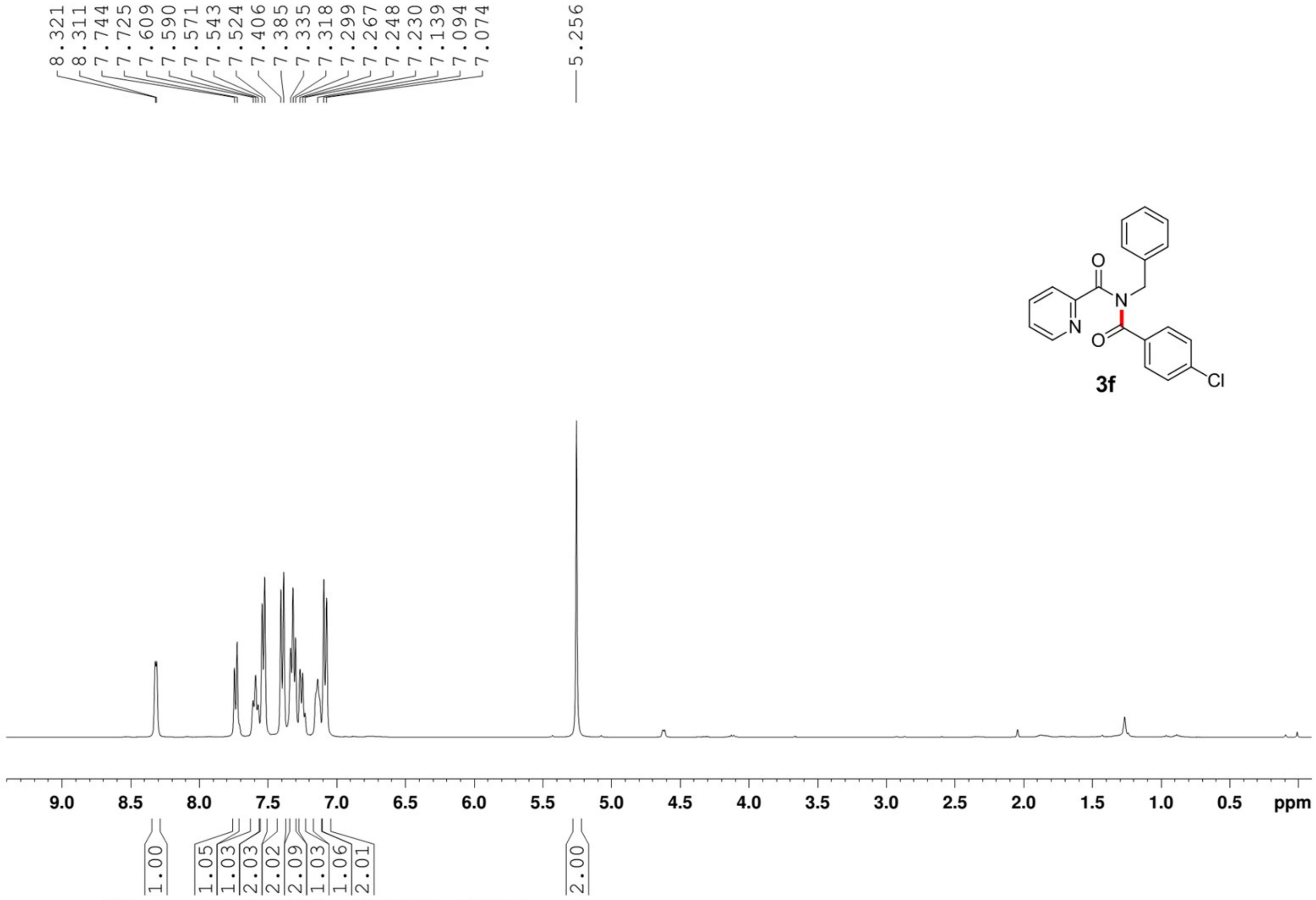
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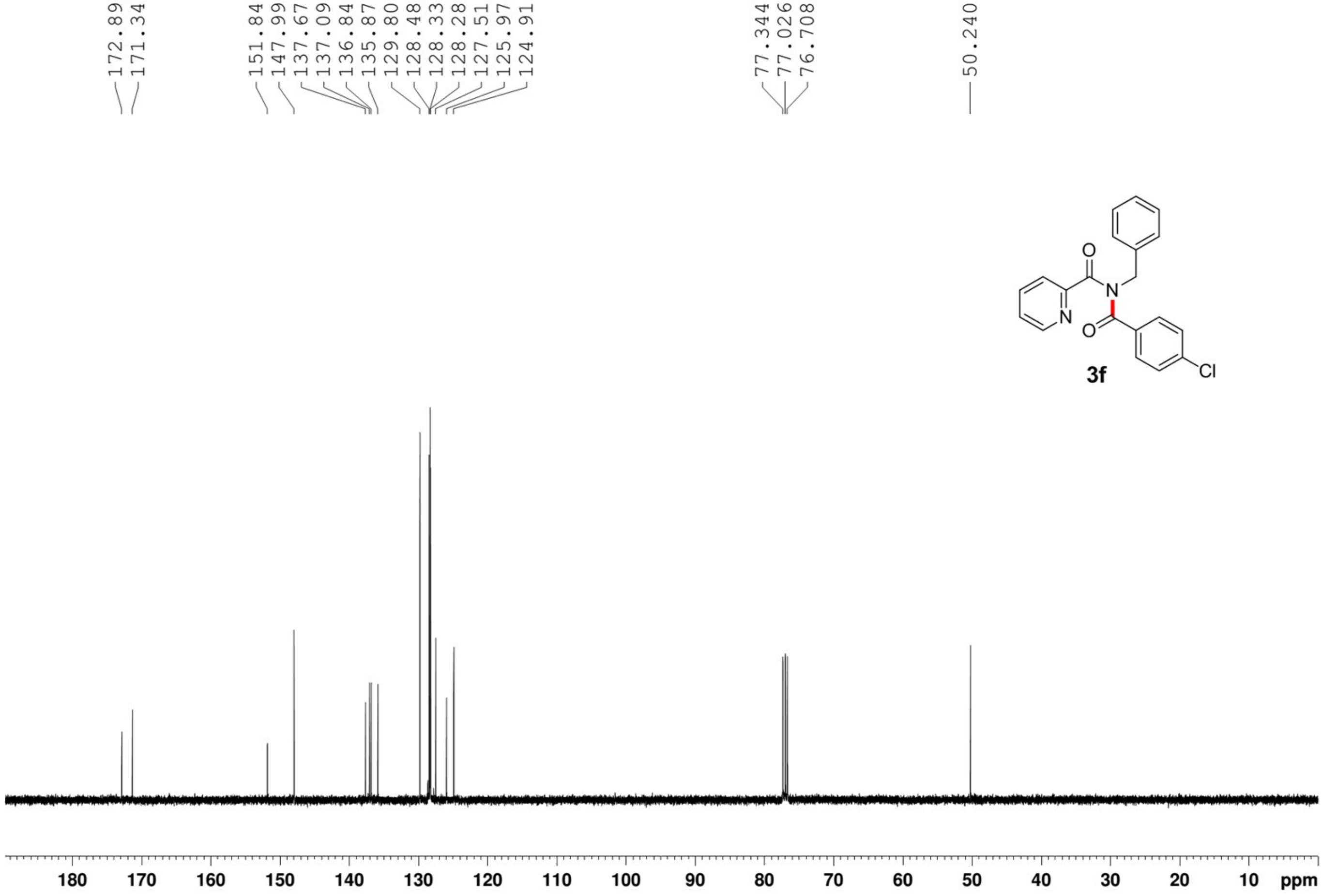
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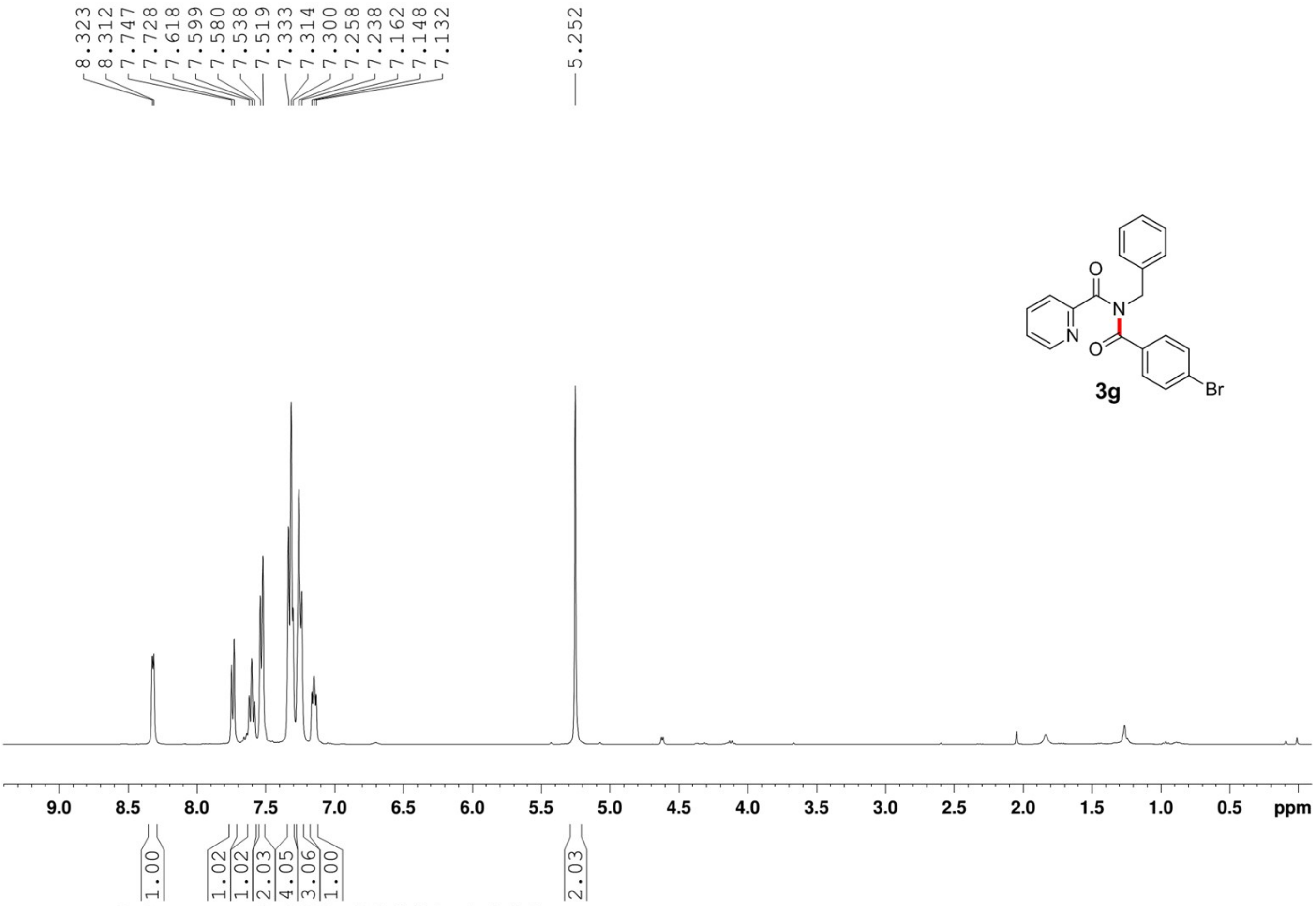
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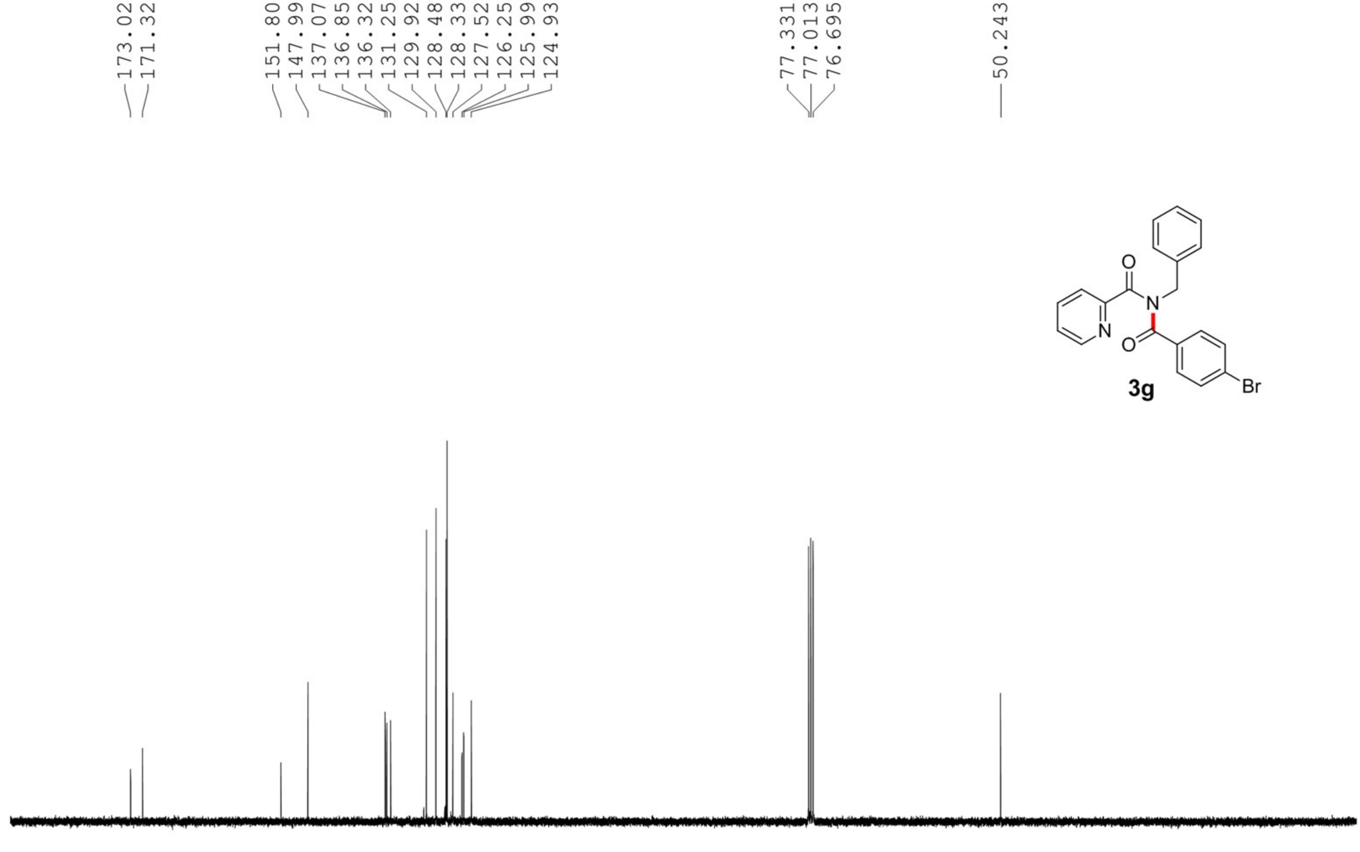
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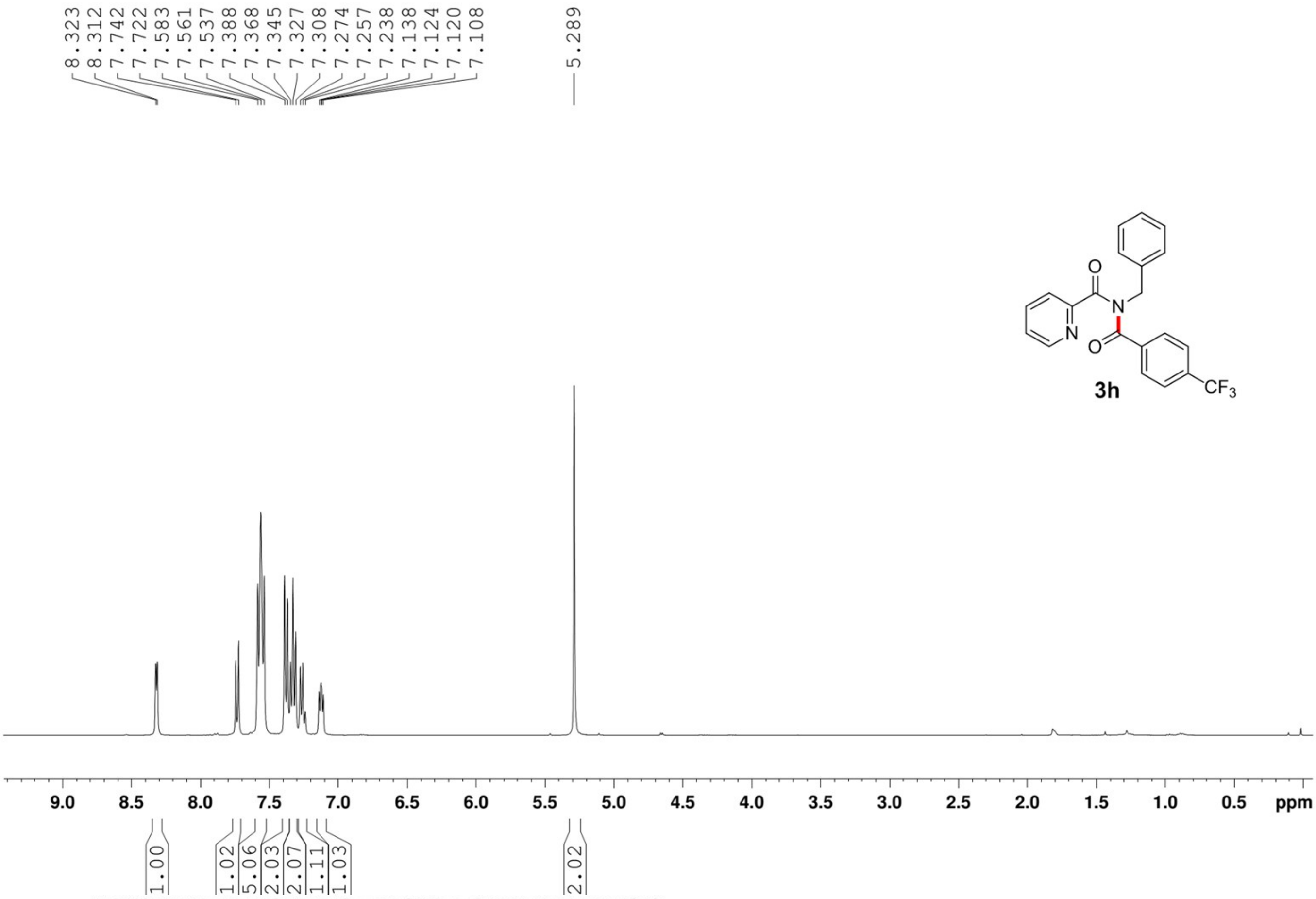
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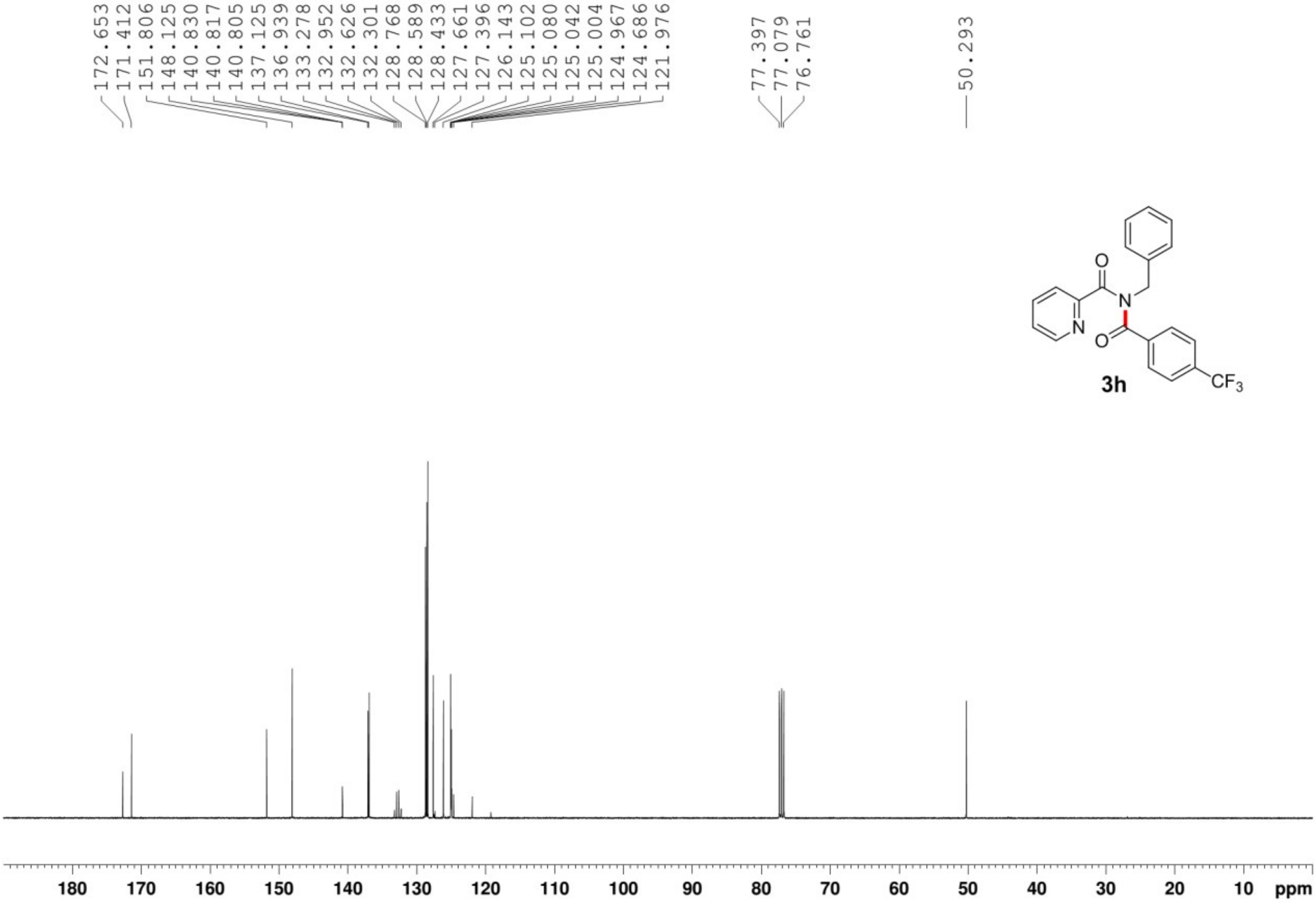
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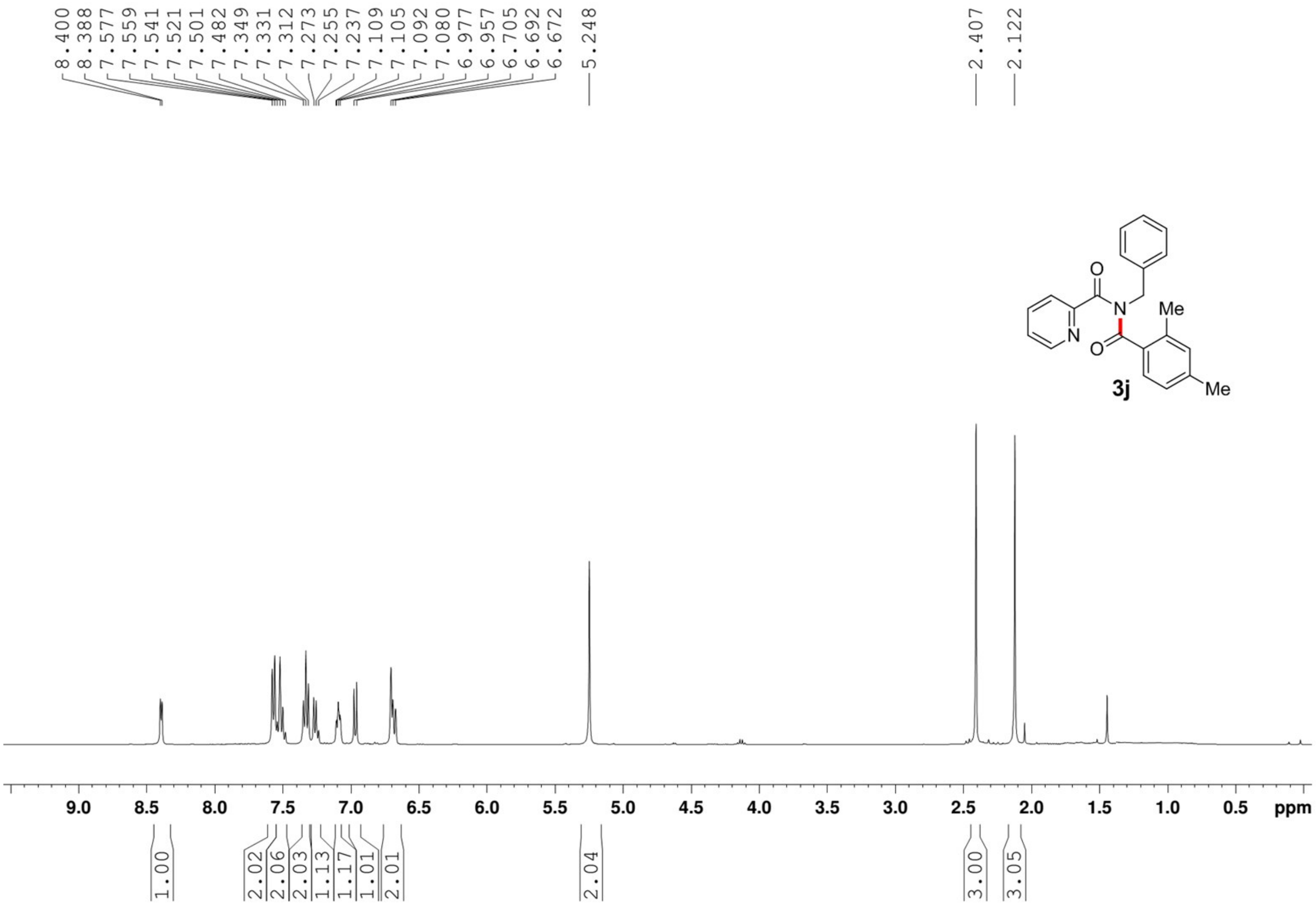
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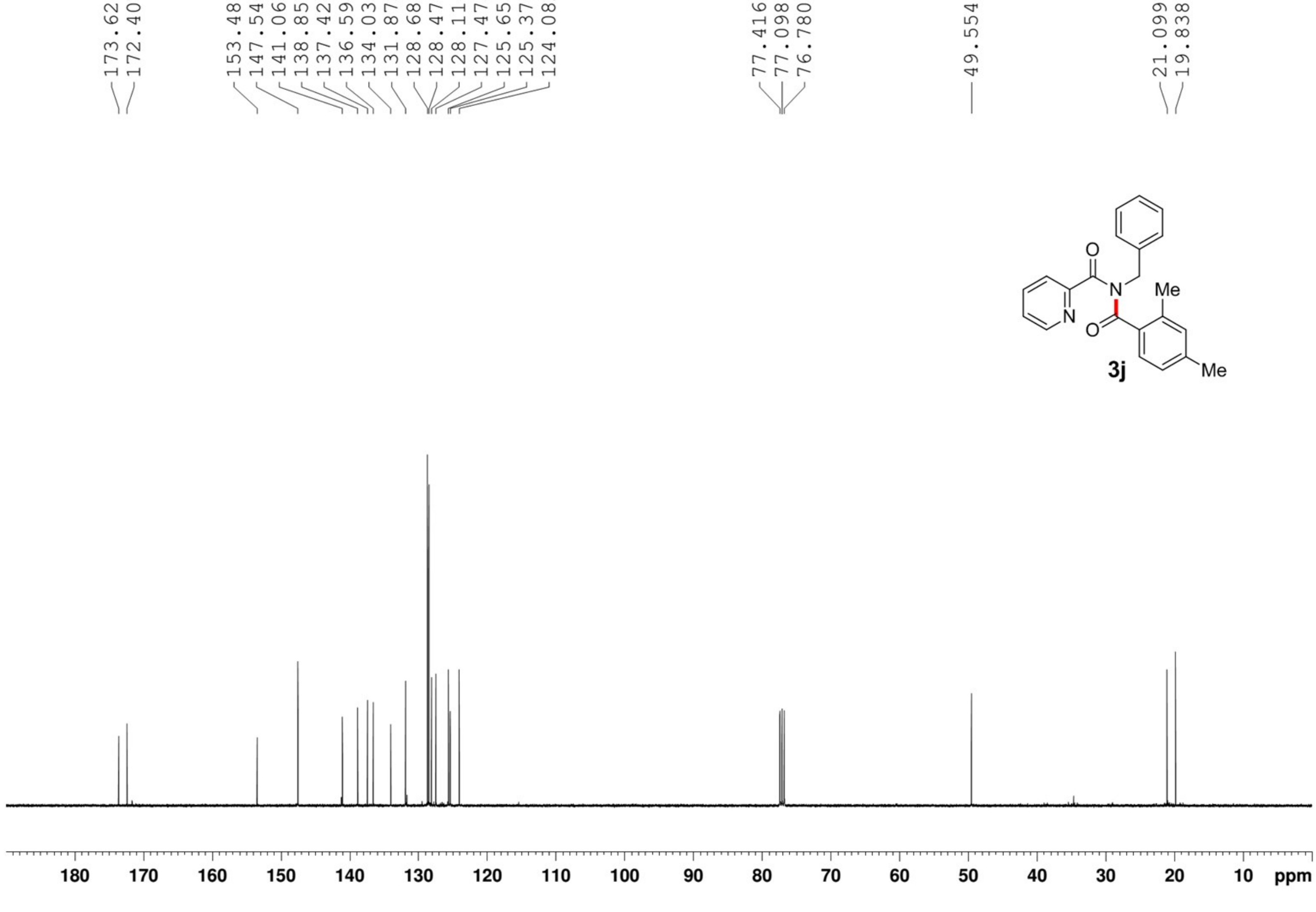
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76.780
49.554
21.099
19.838
O
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N
Me
O
3j
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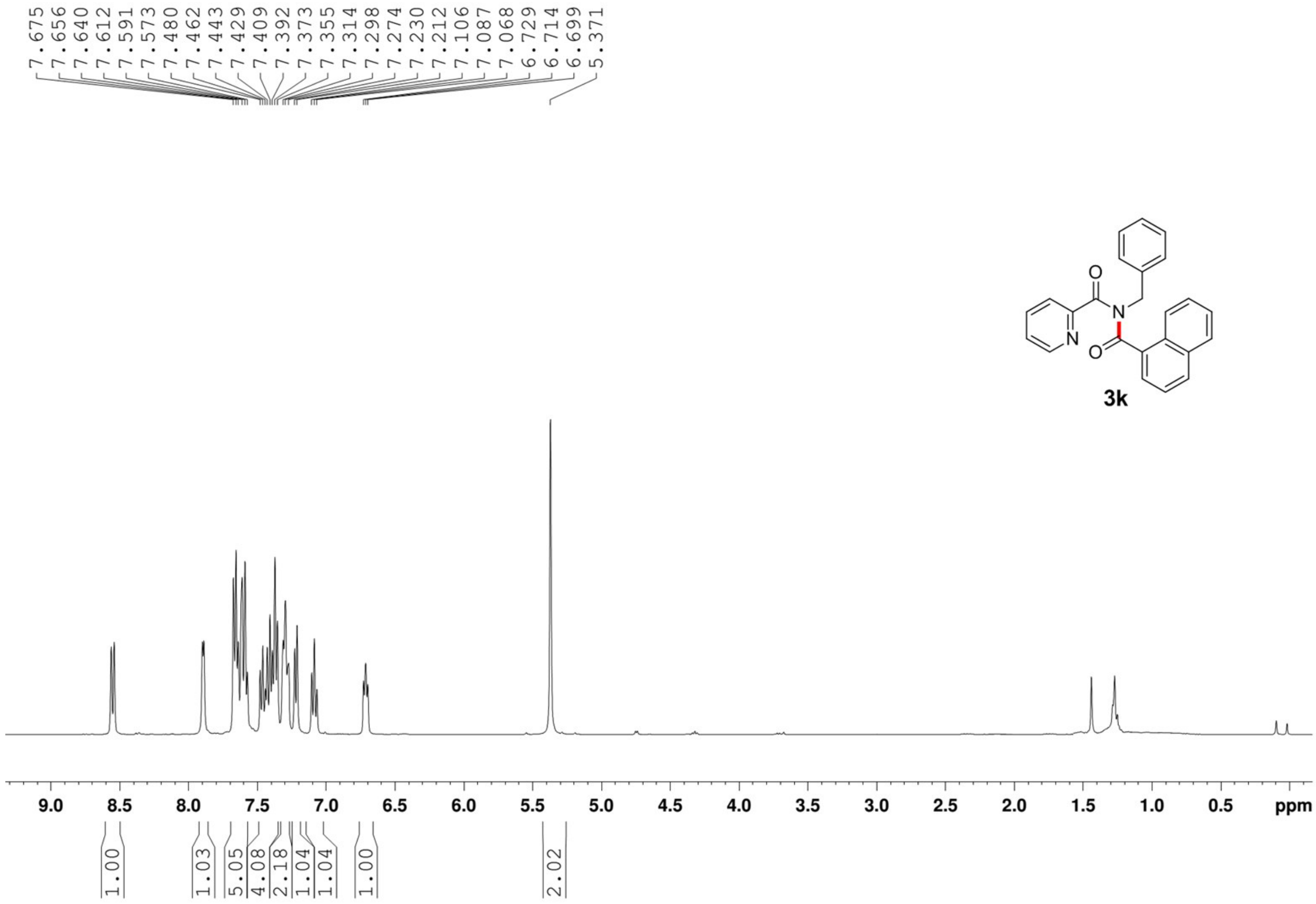
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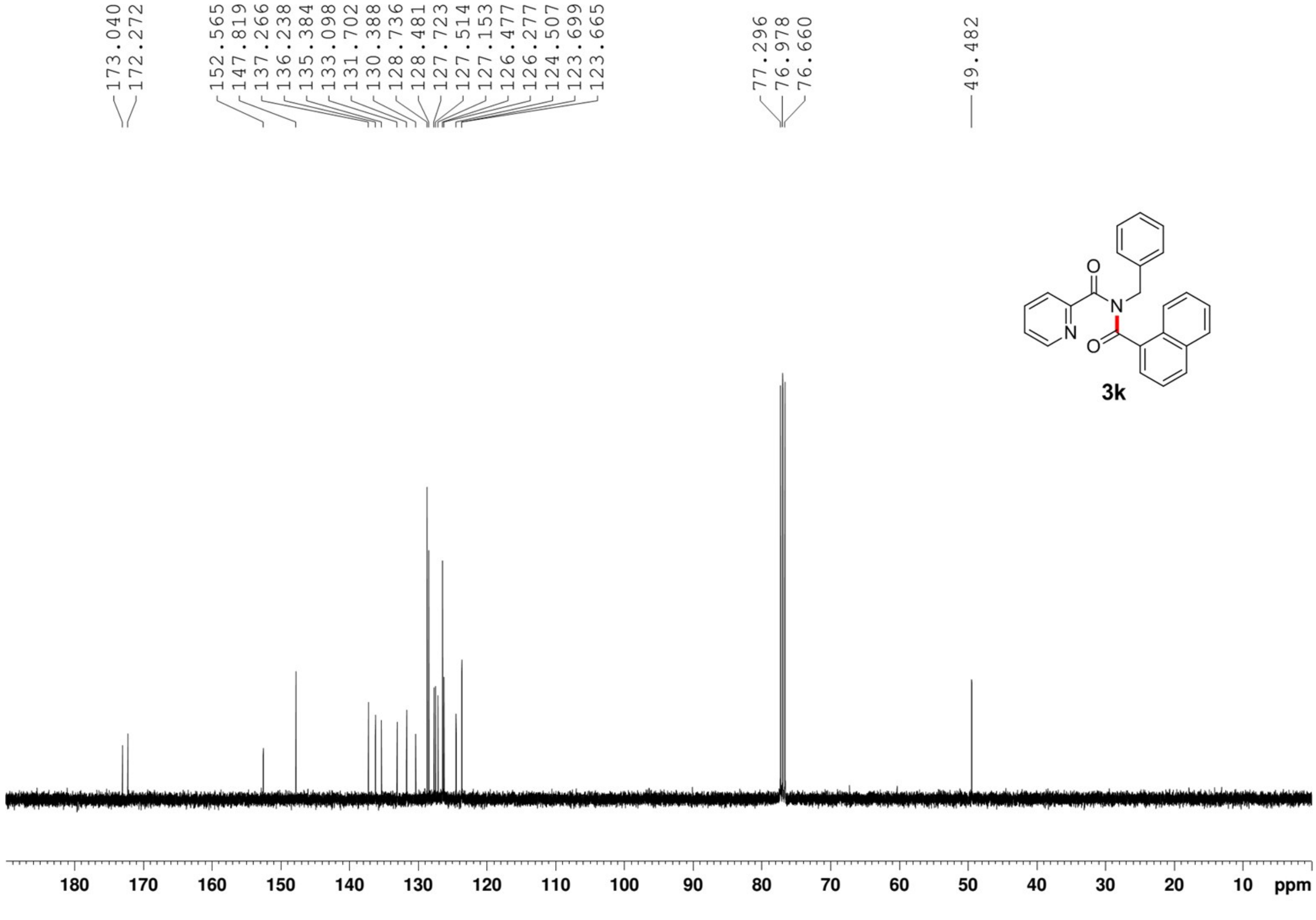
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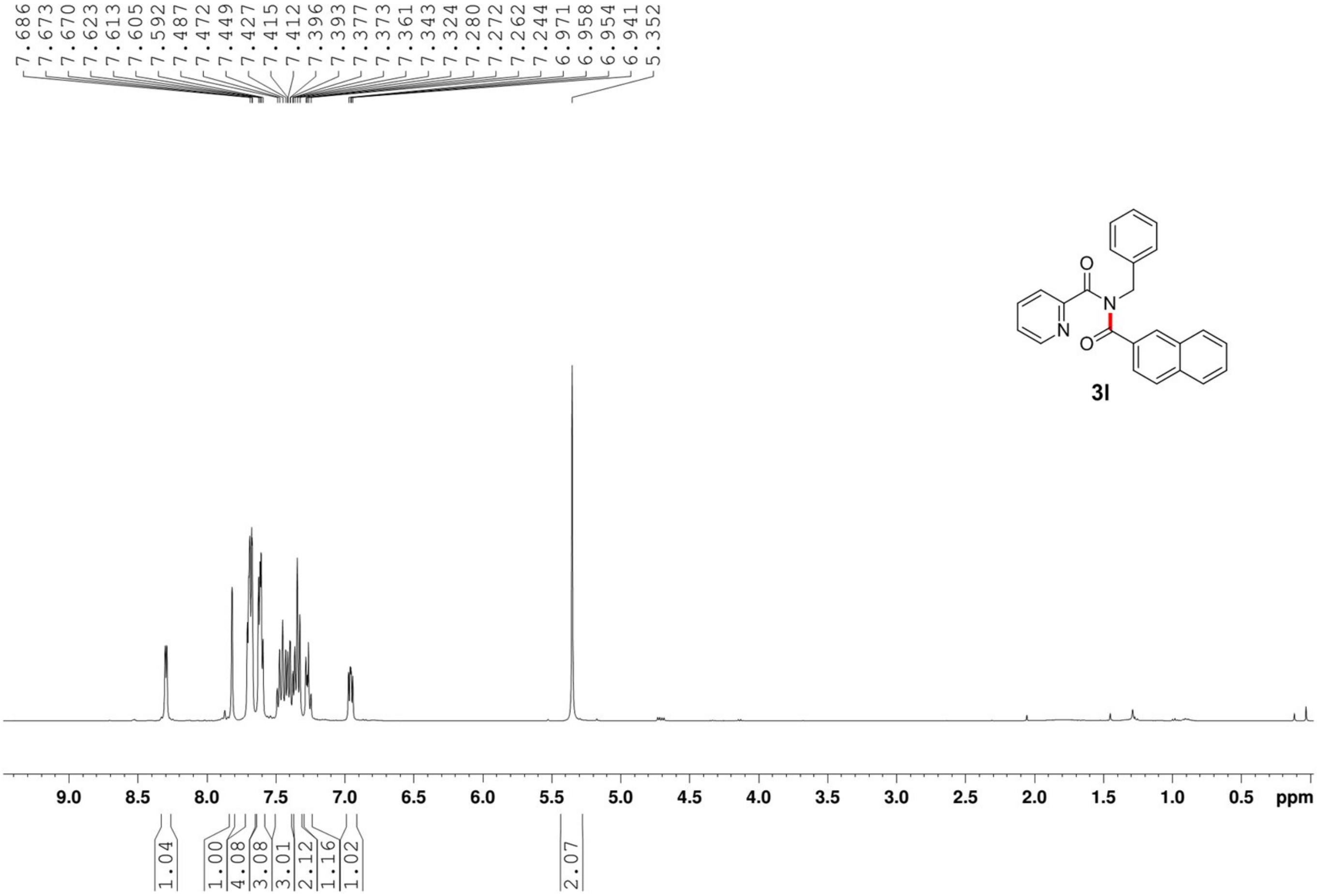
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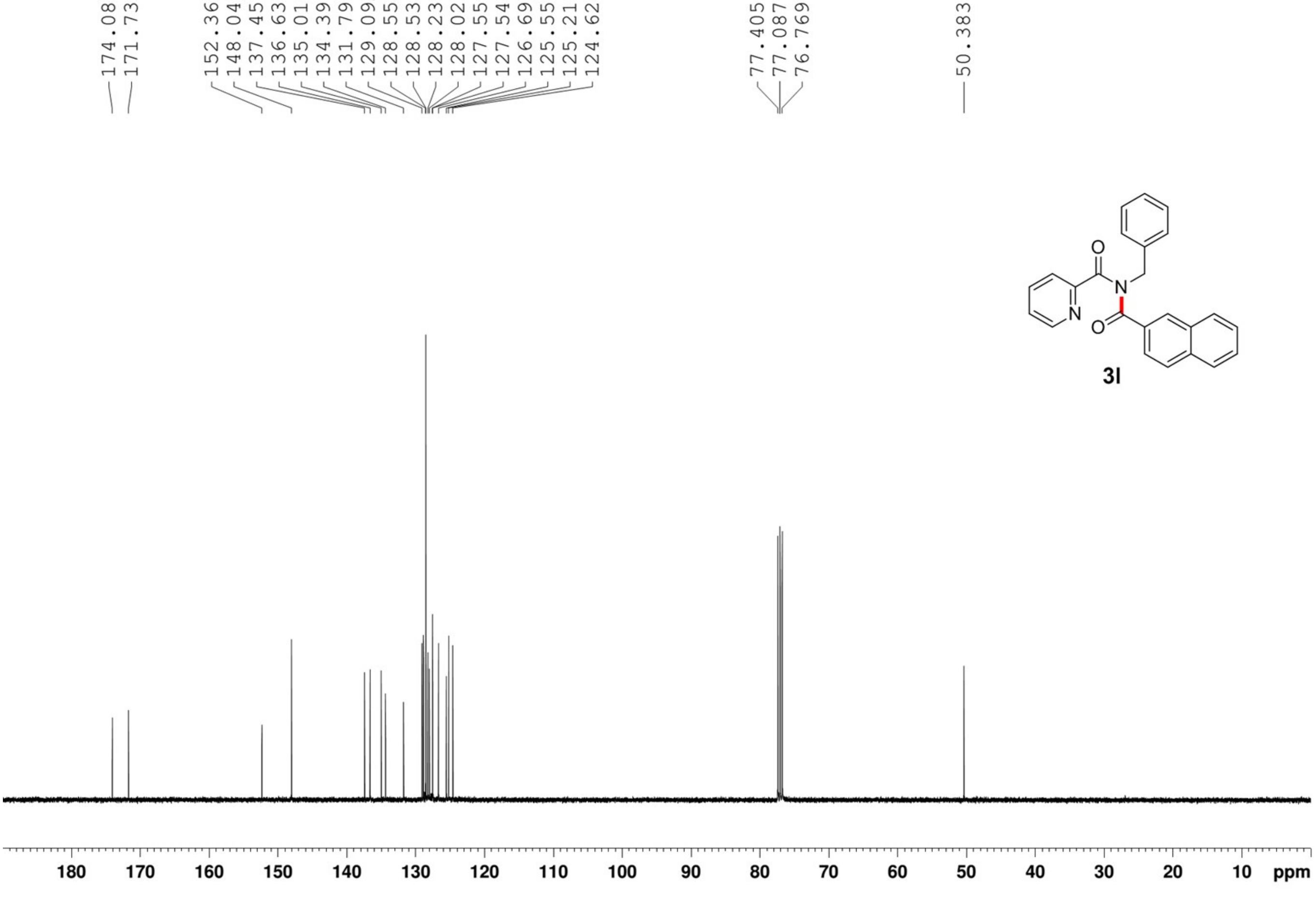
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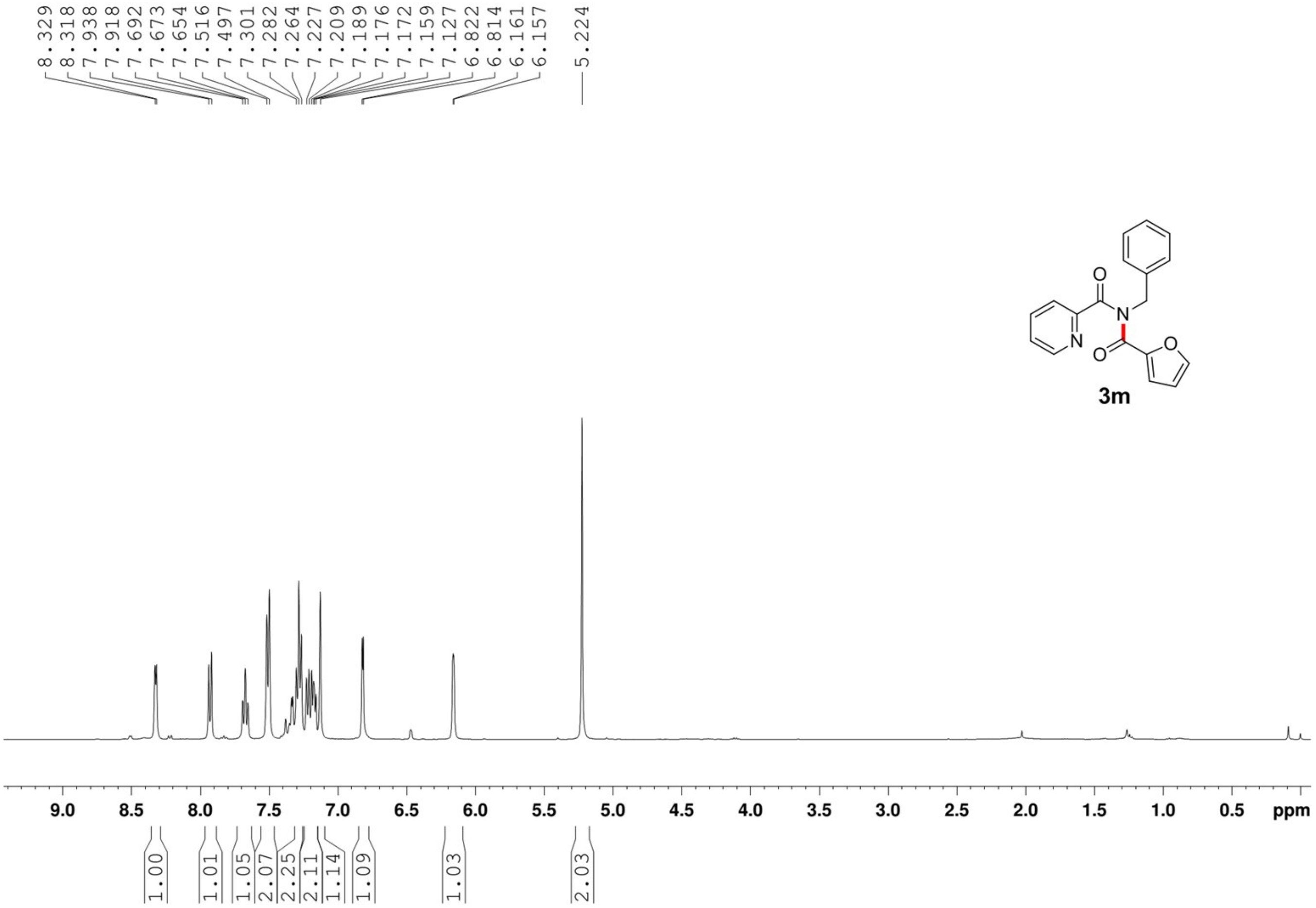
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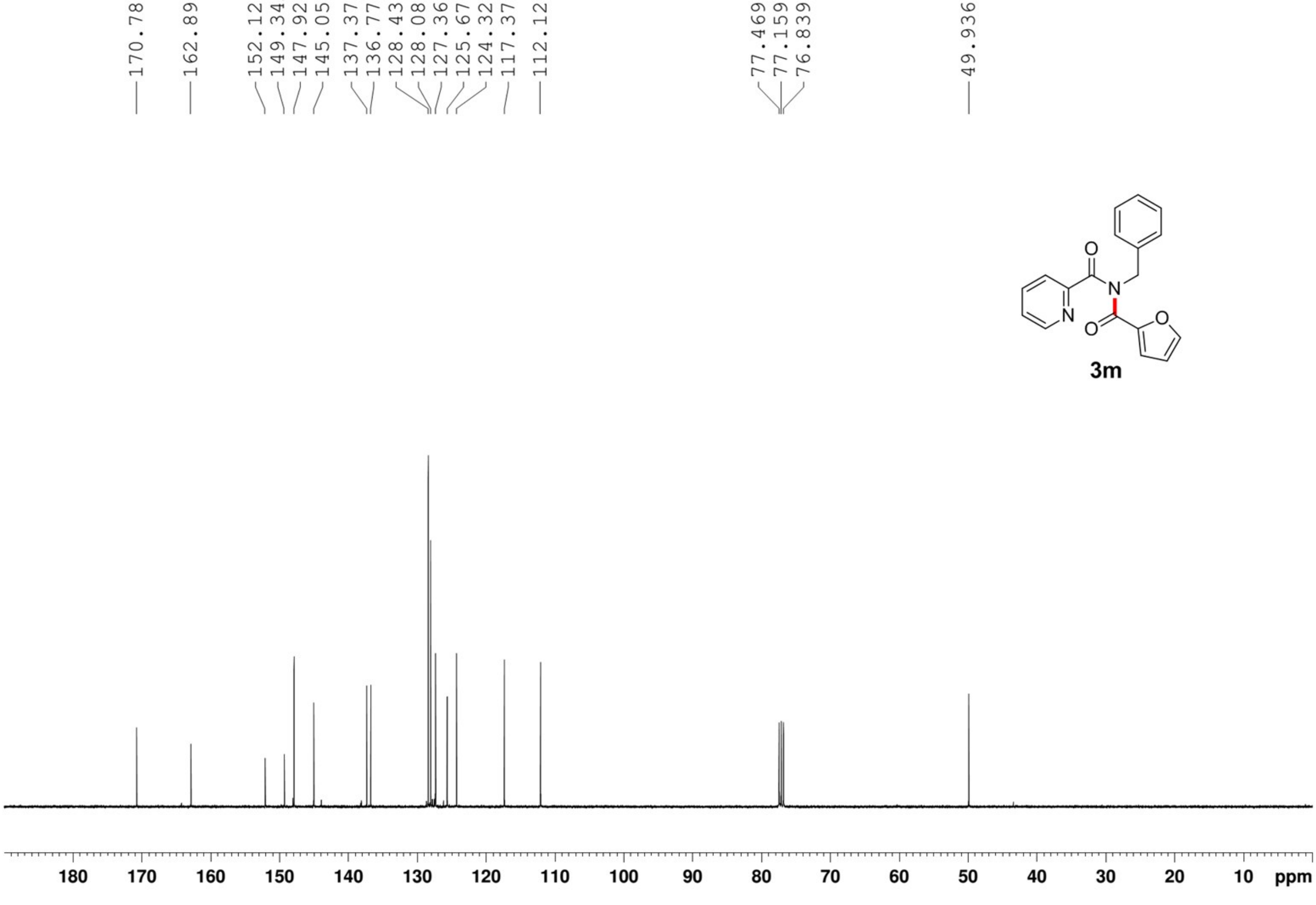
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77.159
76.839
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N
O
O
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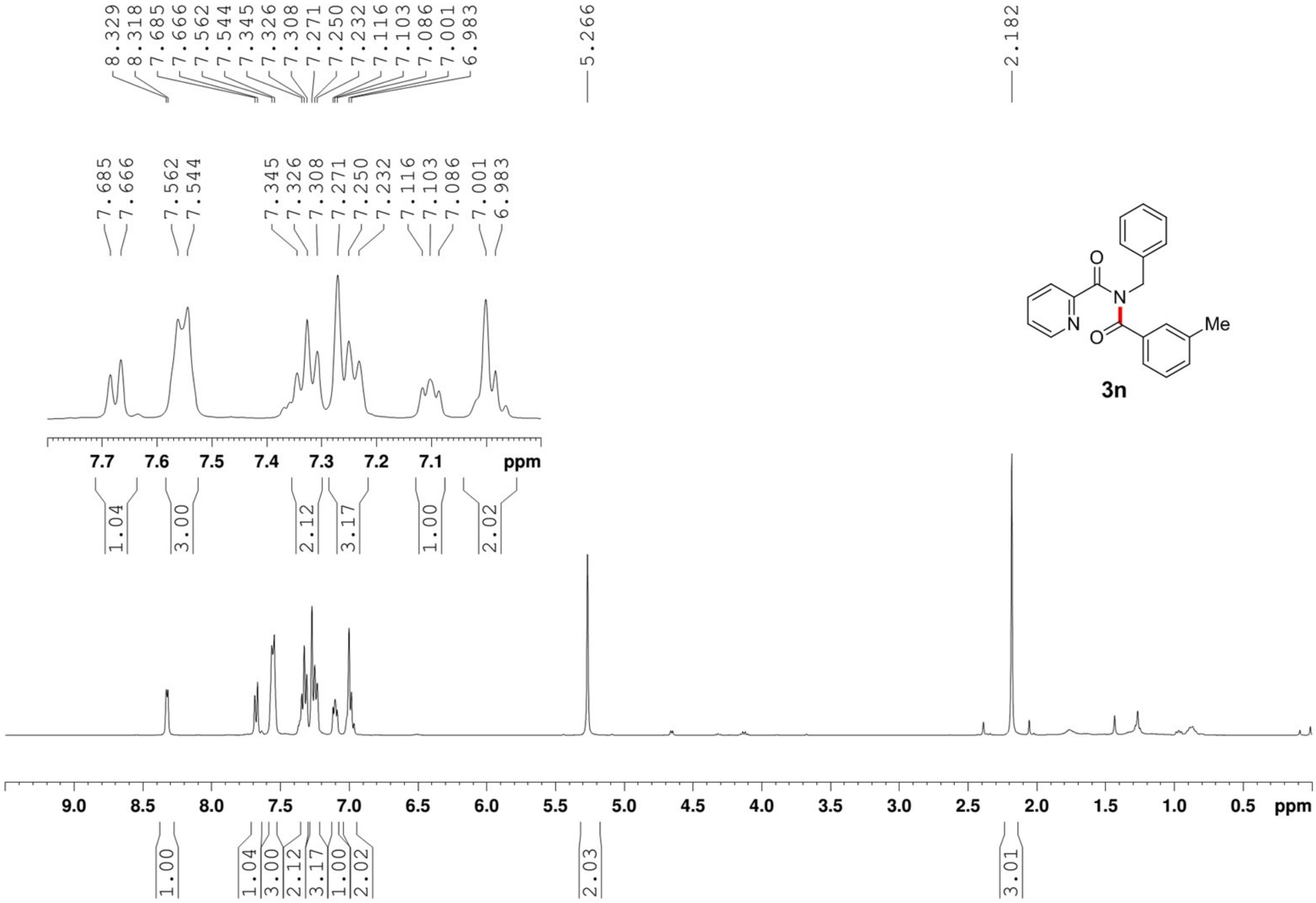
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7.116
7.103
7.086
7.001
6.983
5.266
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7.7
7.6
7.5
7.4
7.3
7.2
7.1
ppm
1.04
3.00
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3.17
1.00
2.02
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N
O
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3n
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7.0
6.5
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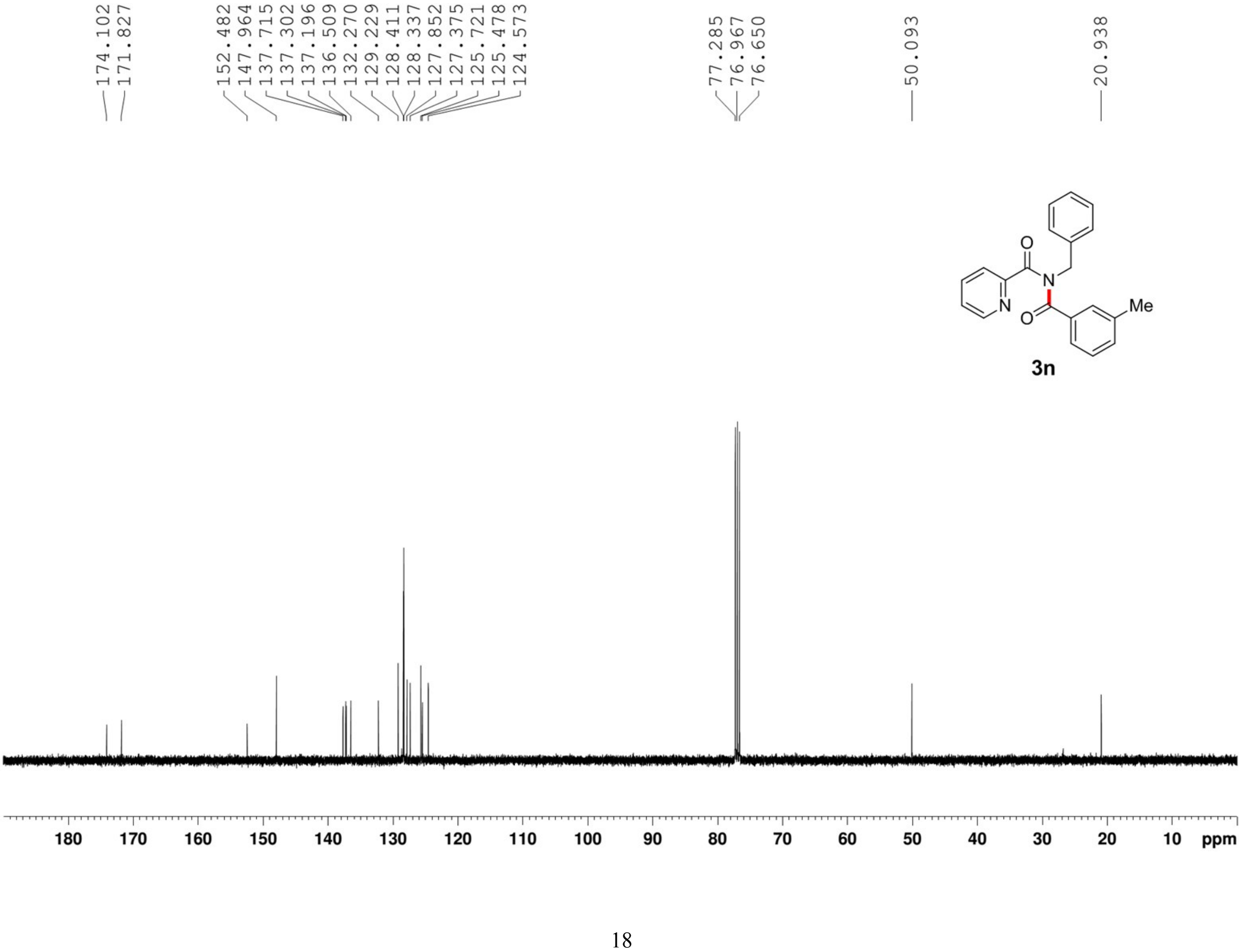
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N
O
Me
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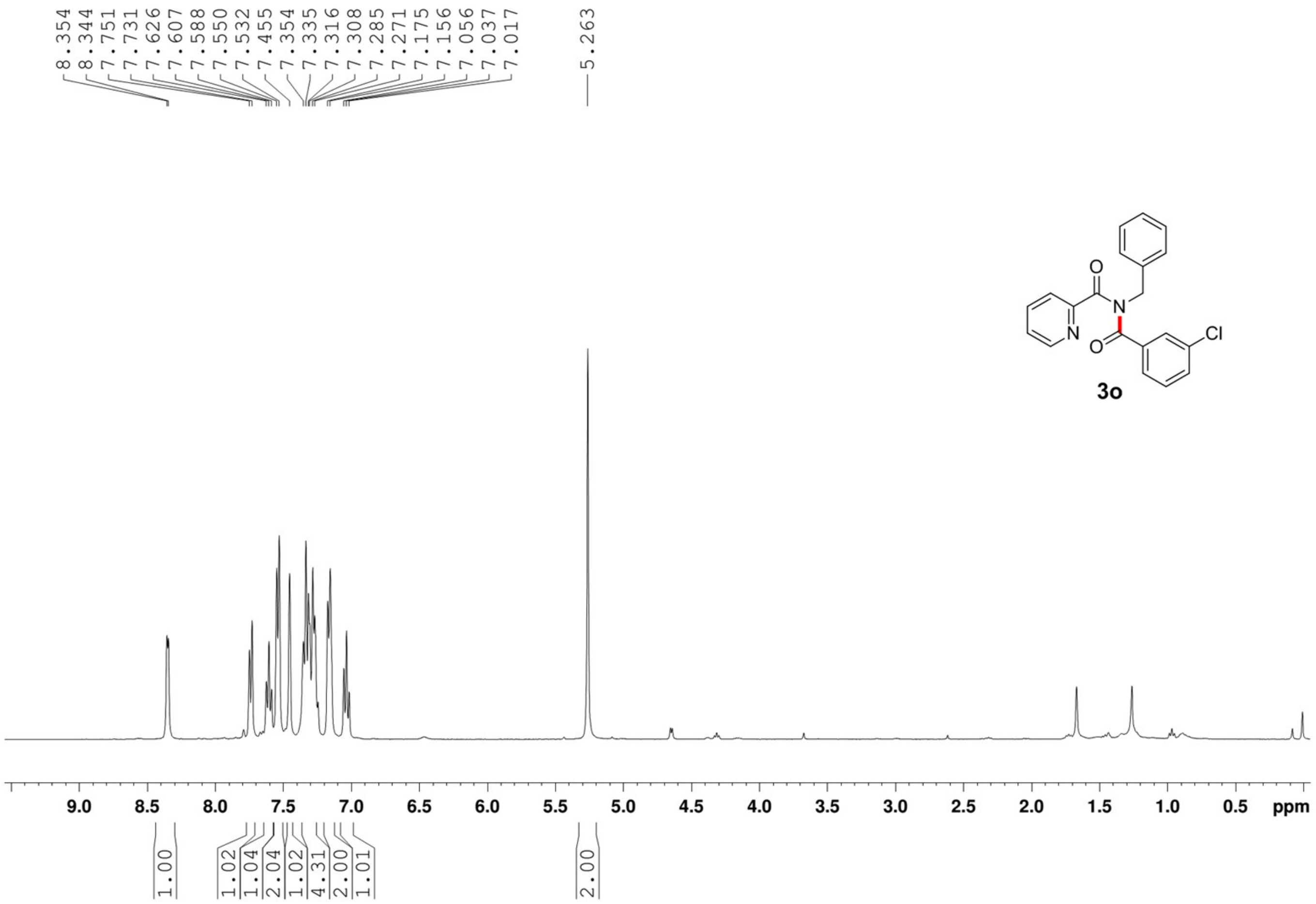
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7.156
7.056
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7.0
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6.0
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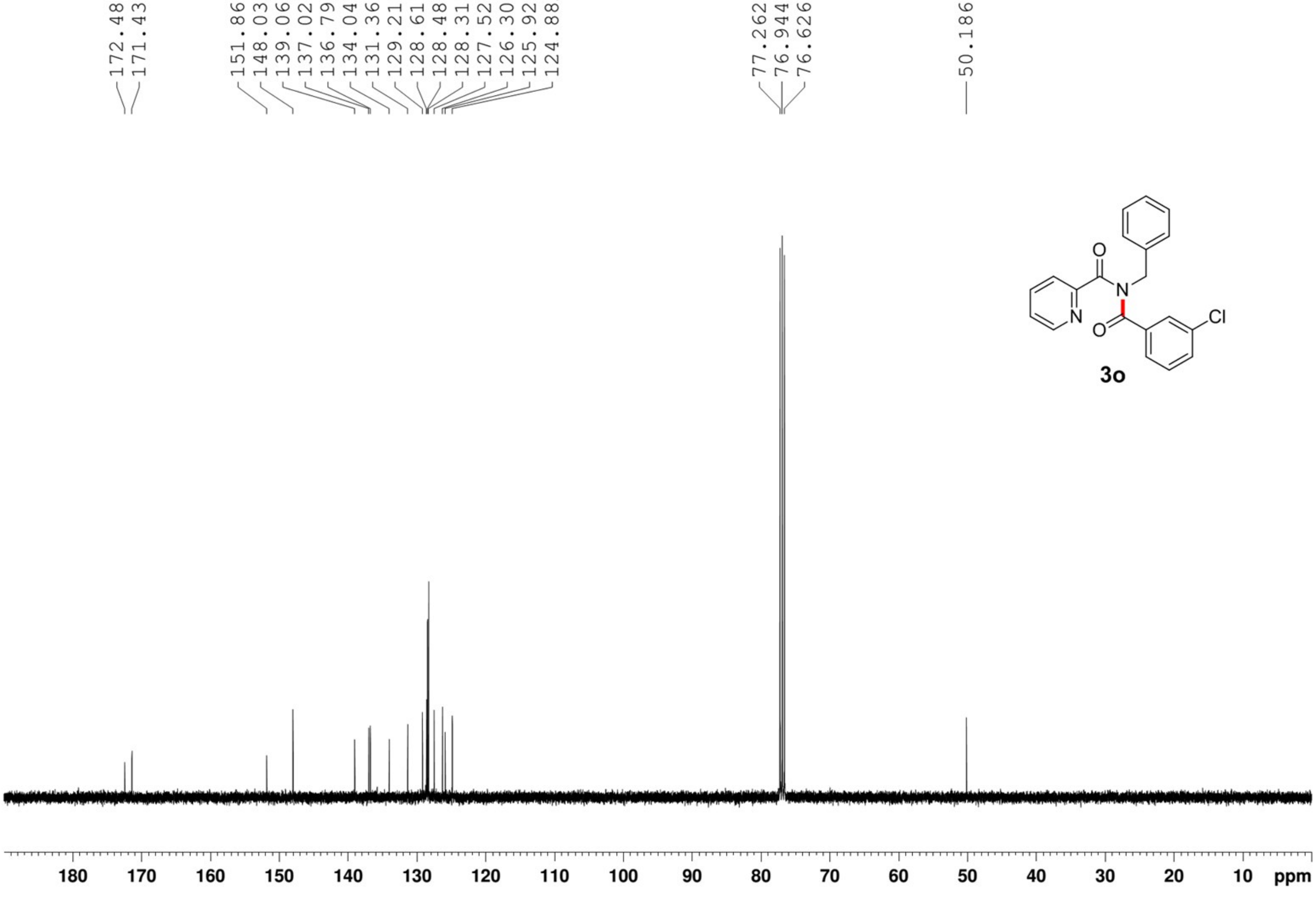
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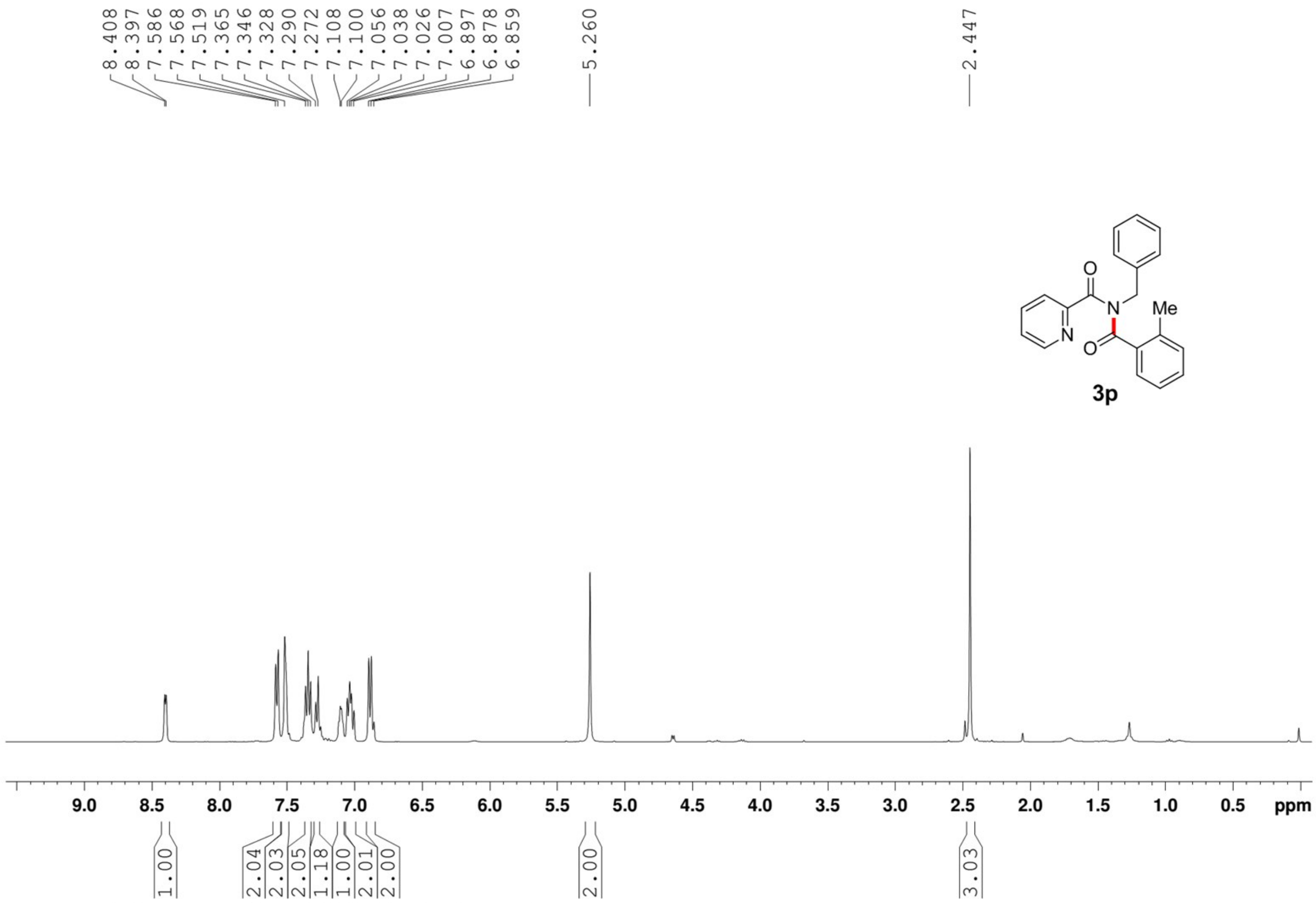
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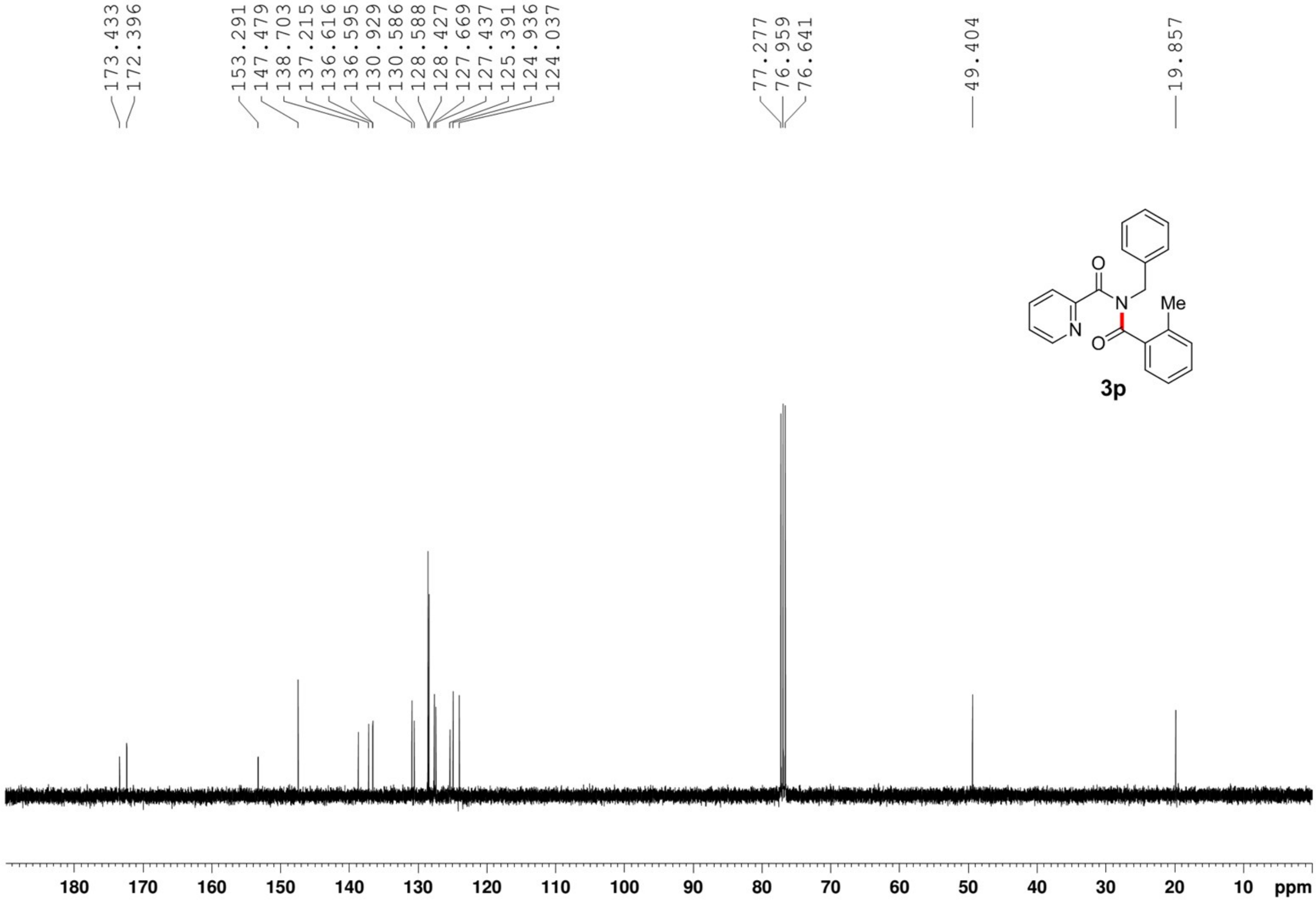
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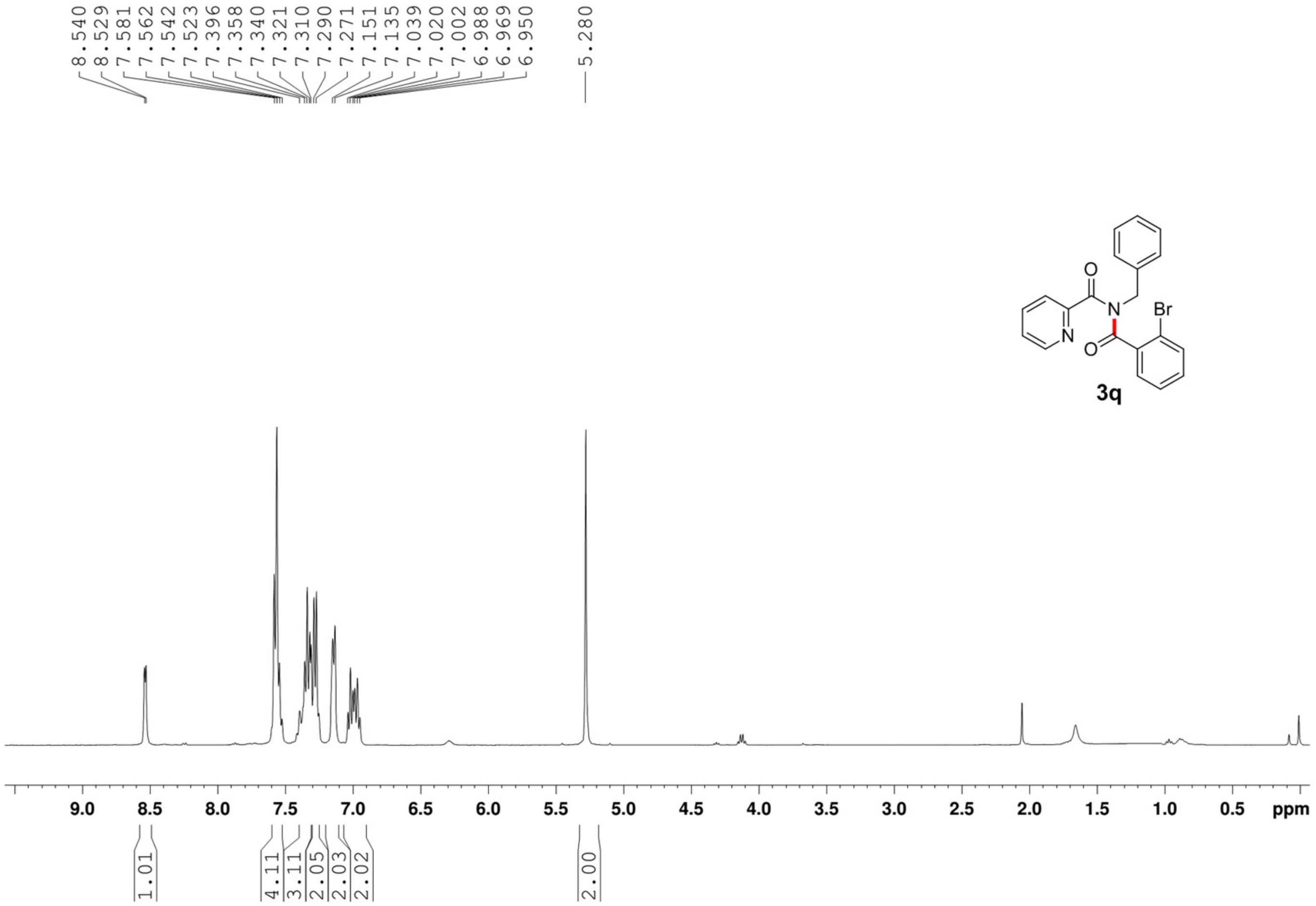
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7.135
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3.0
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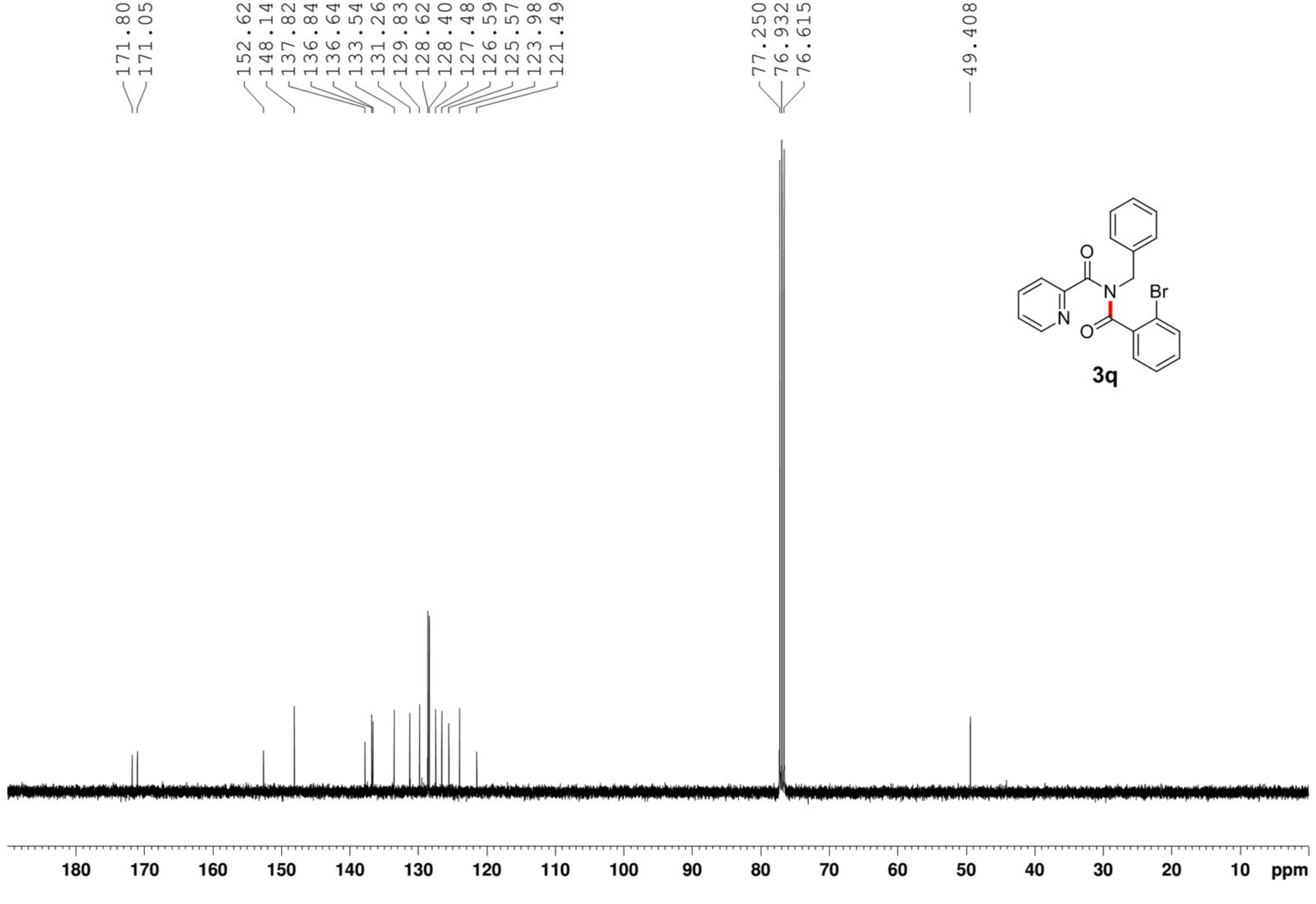
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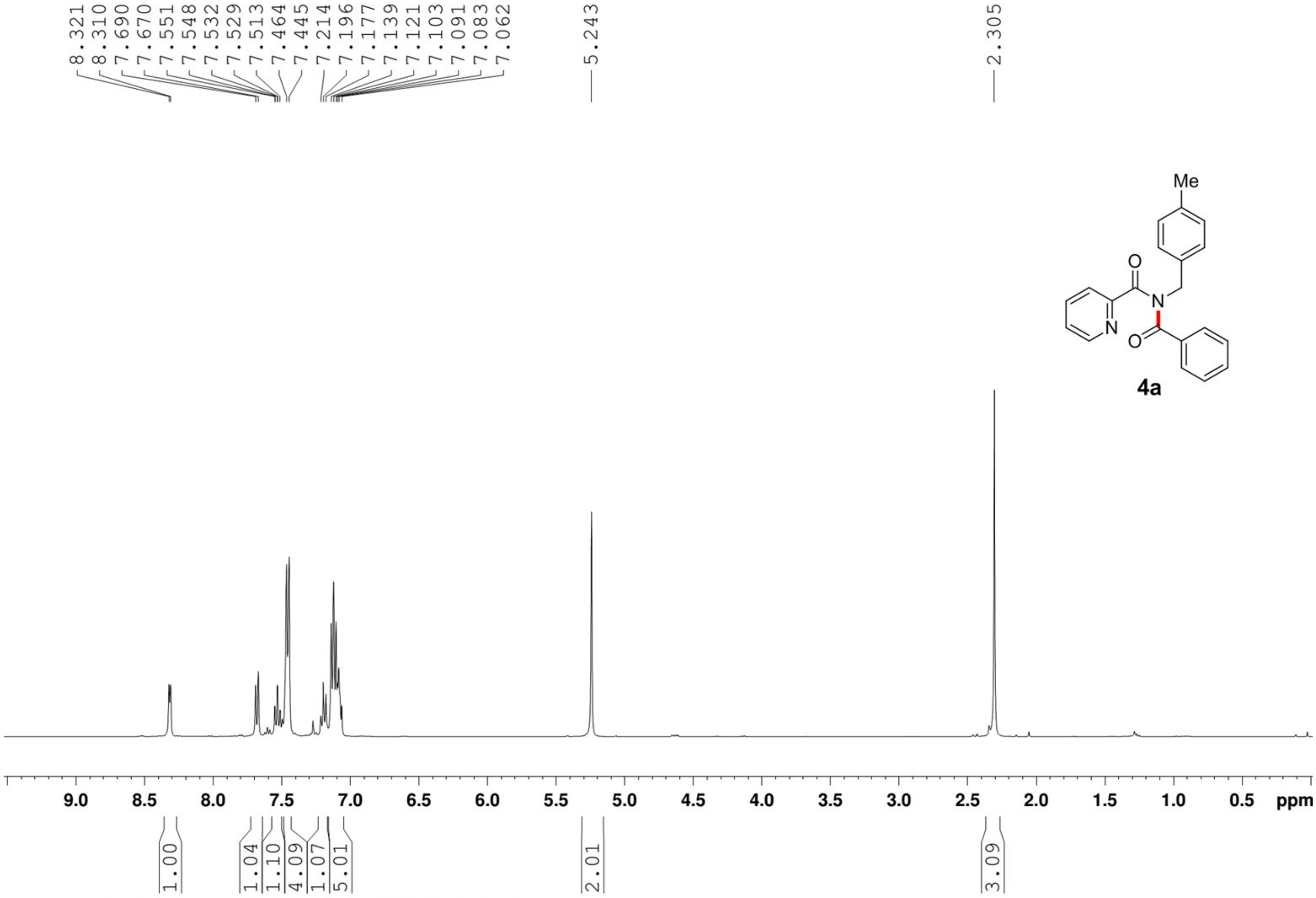
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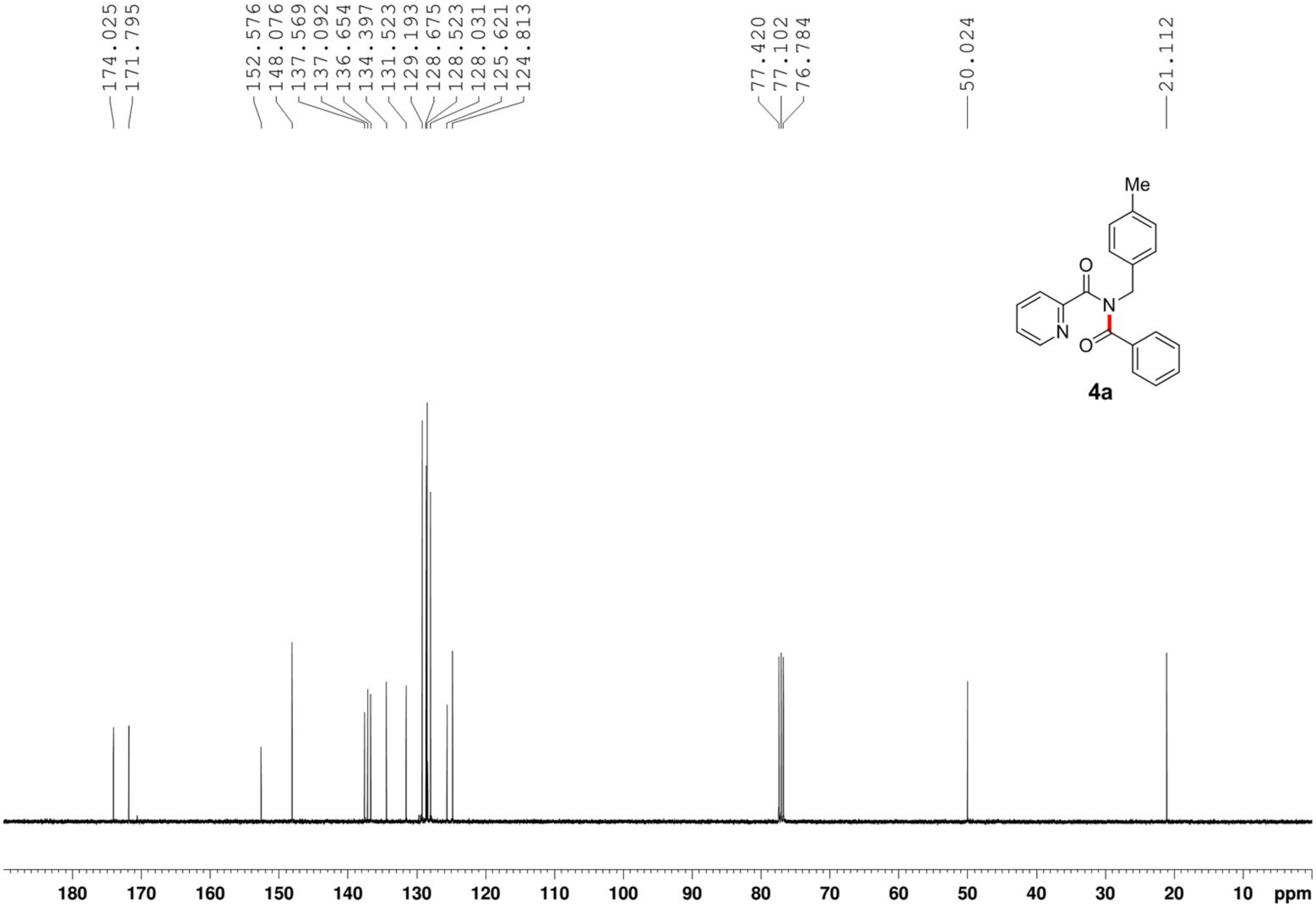
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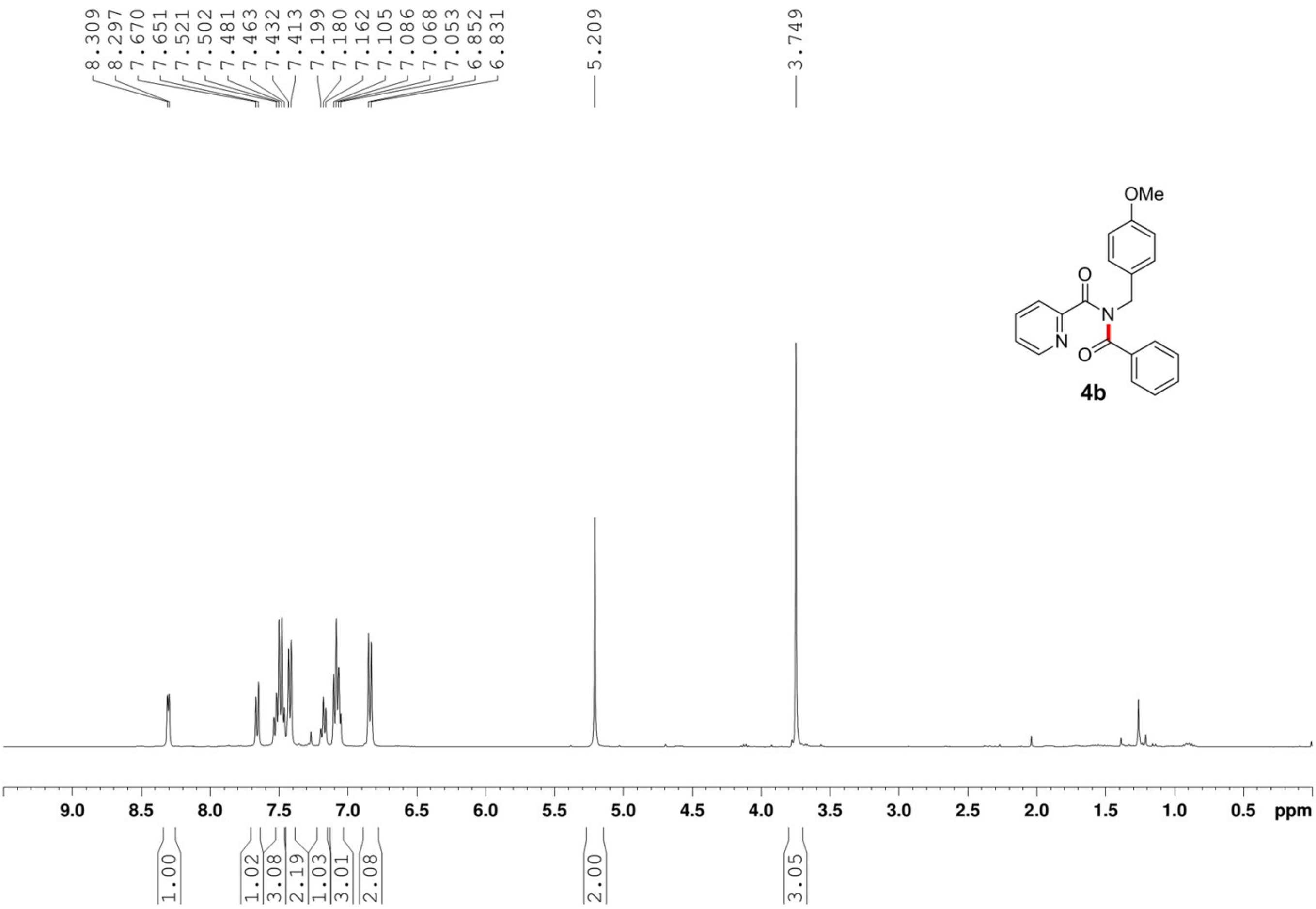
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7.068
7.053
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6.831
5.209
3.749
OMe
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N
O
4b
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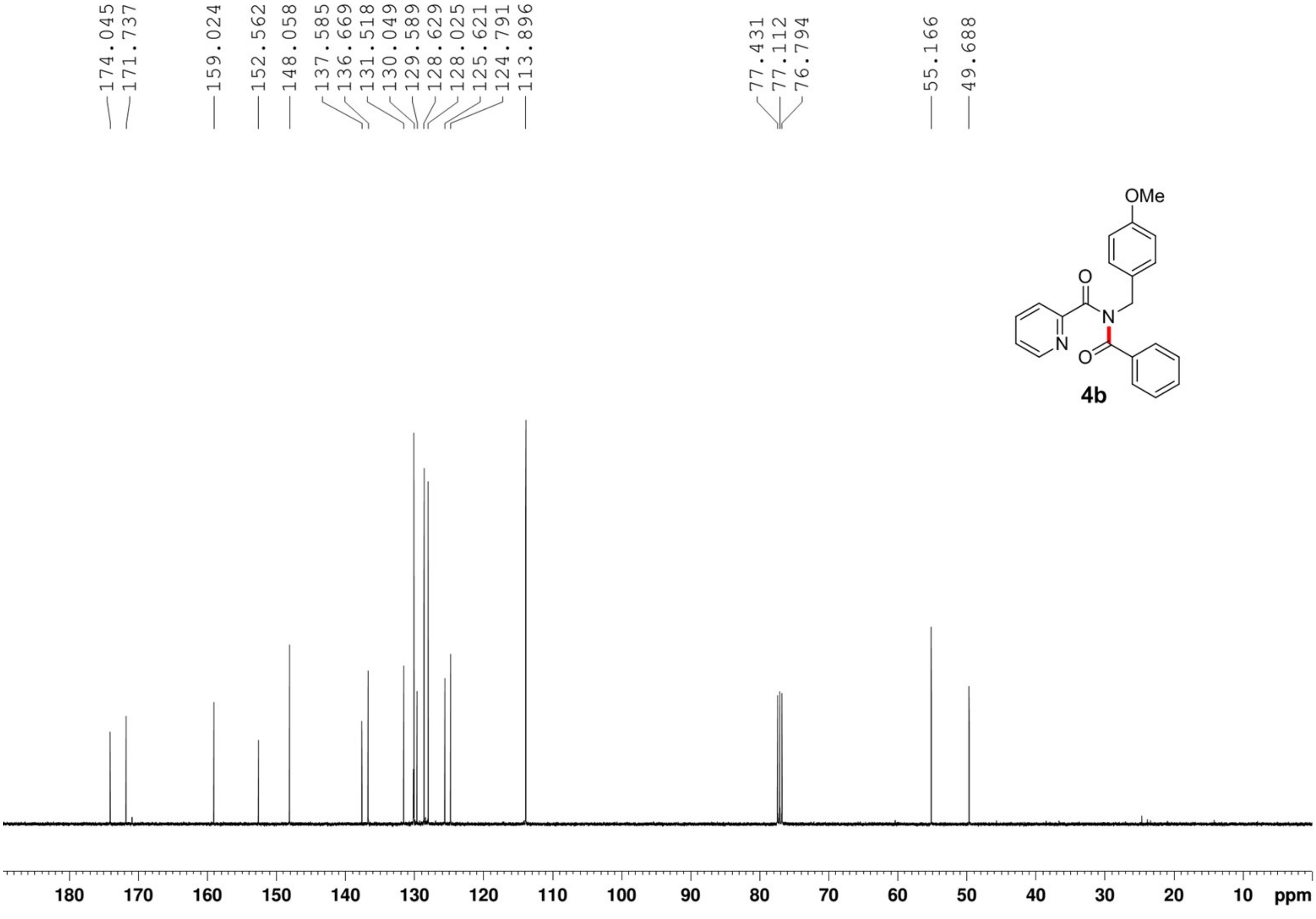
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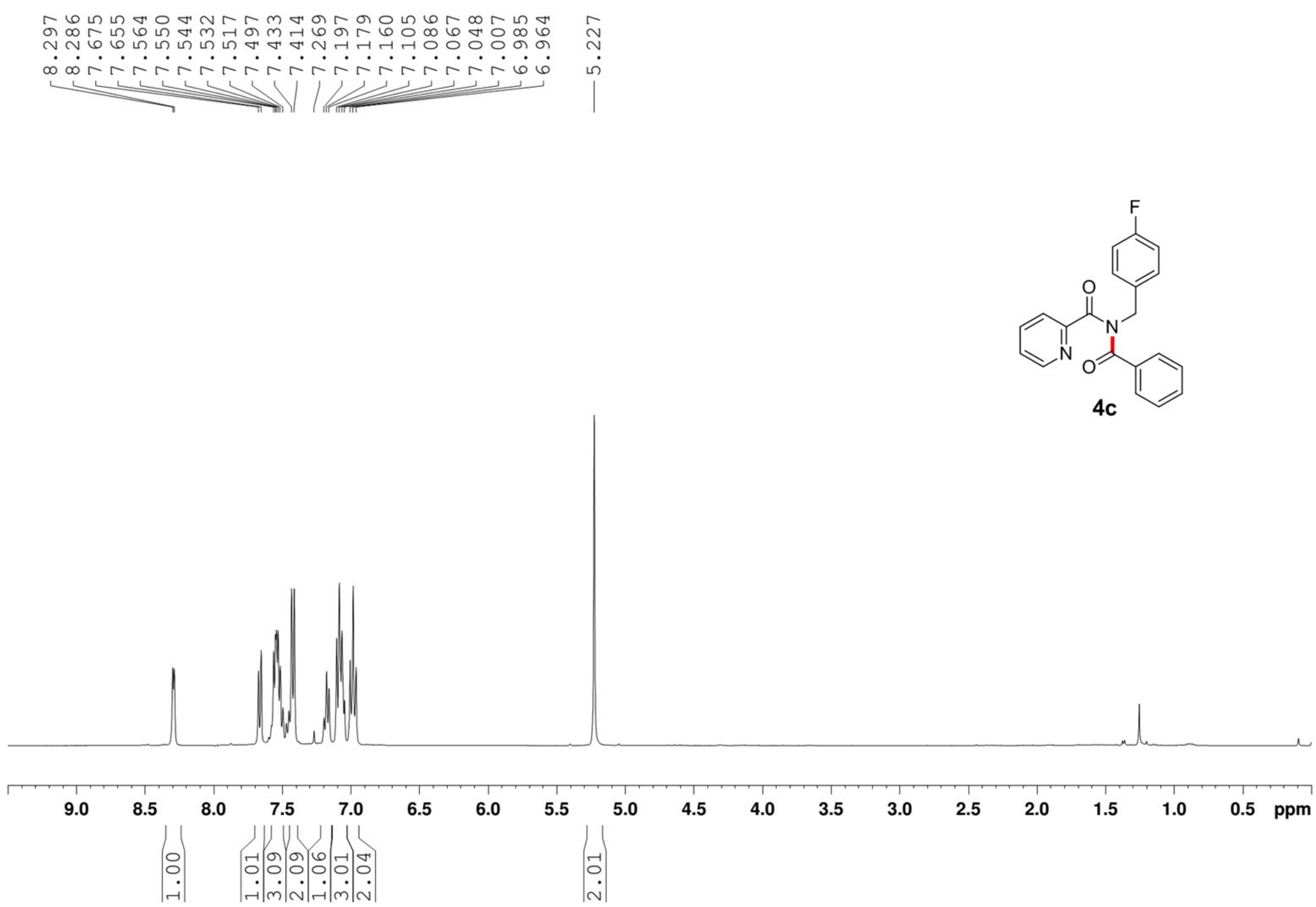
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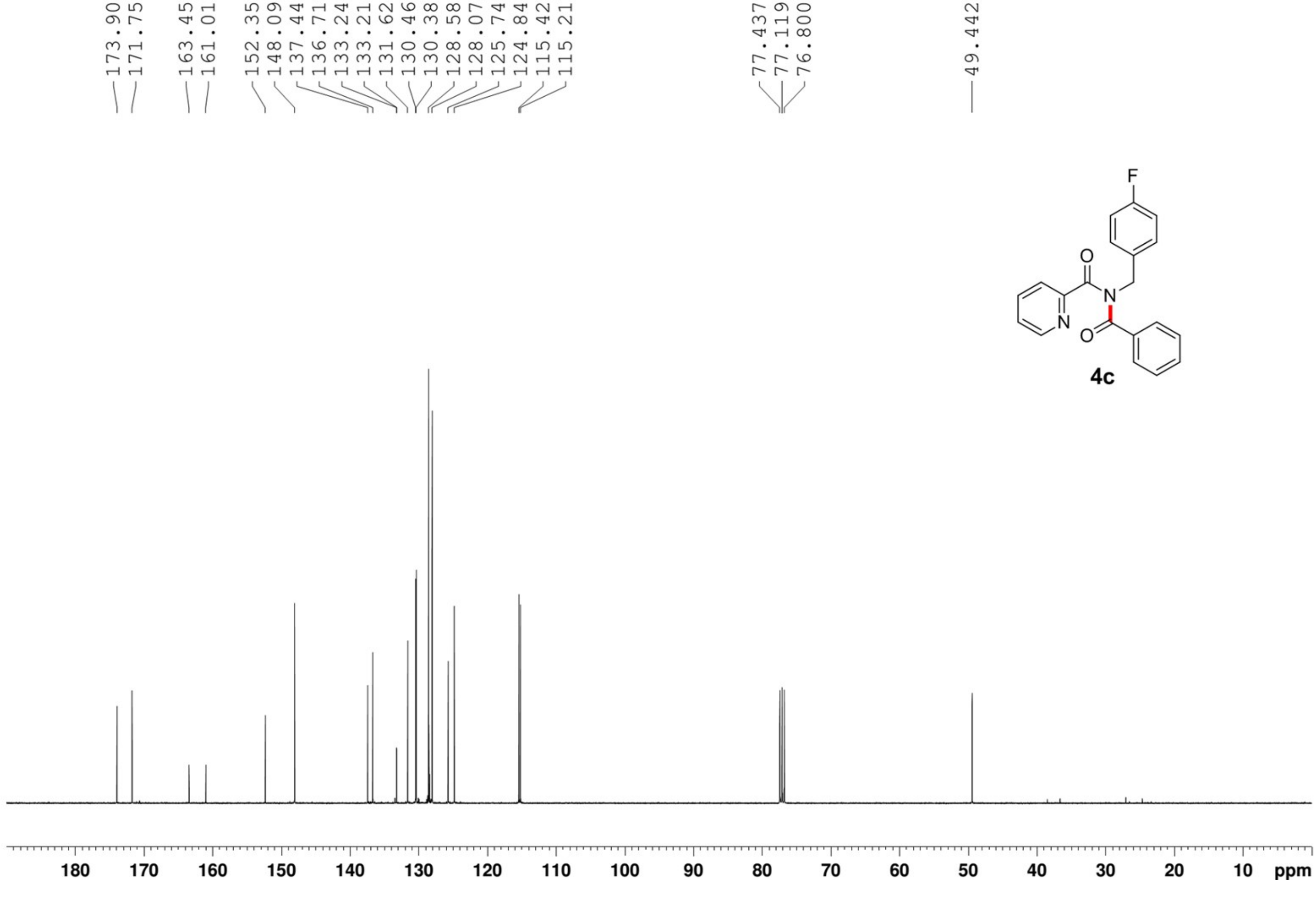
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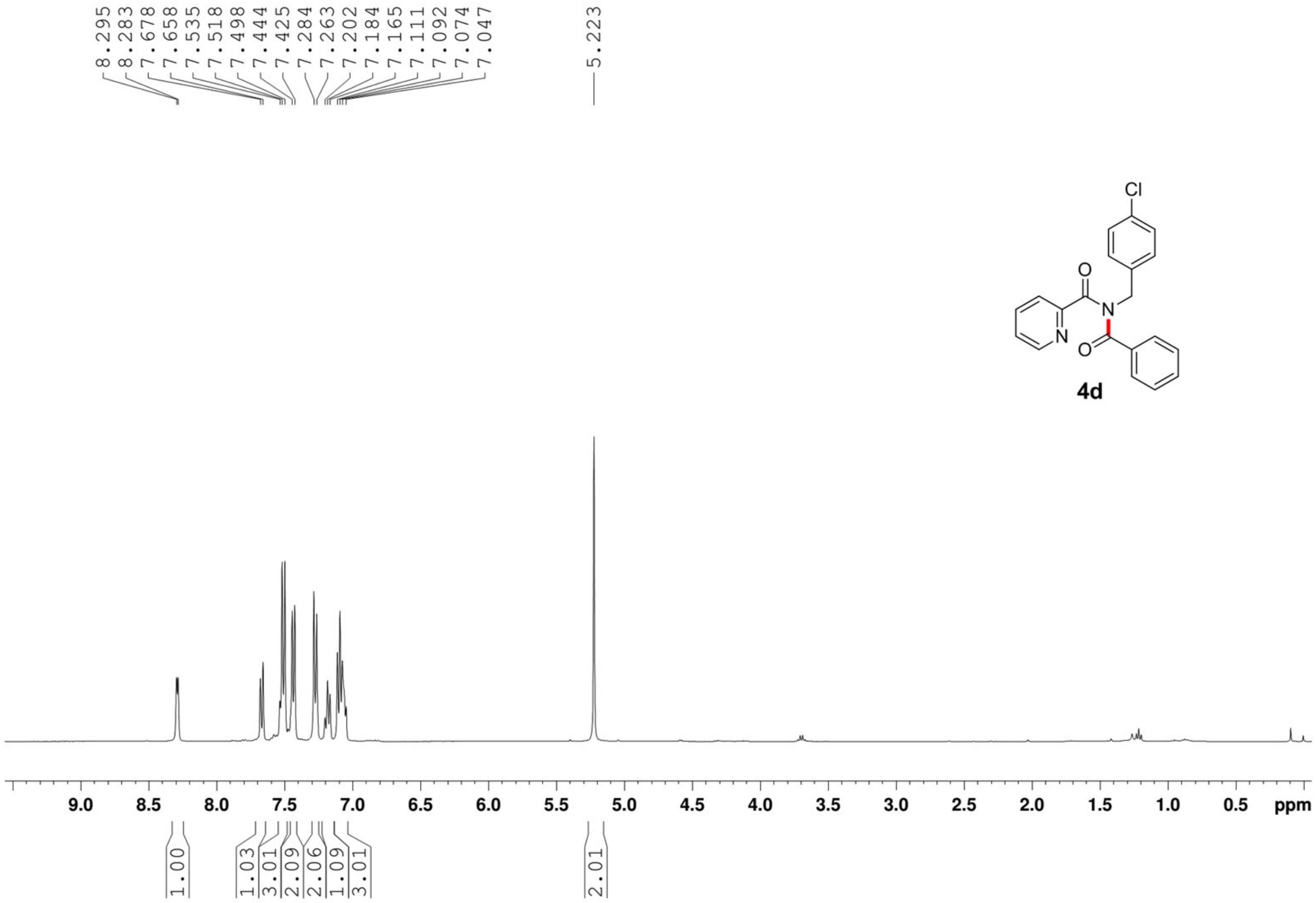
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7.074
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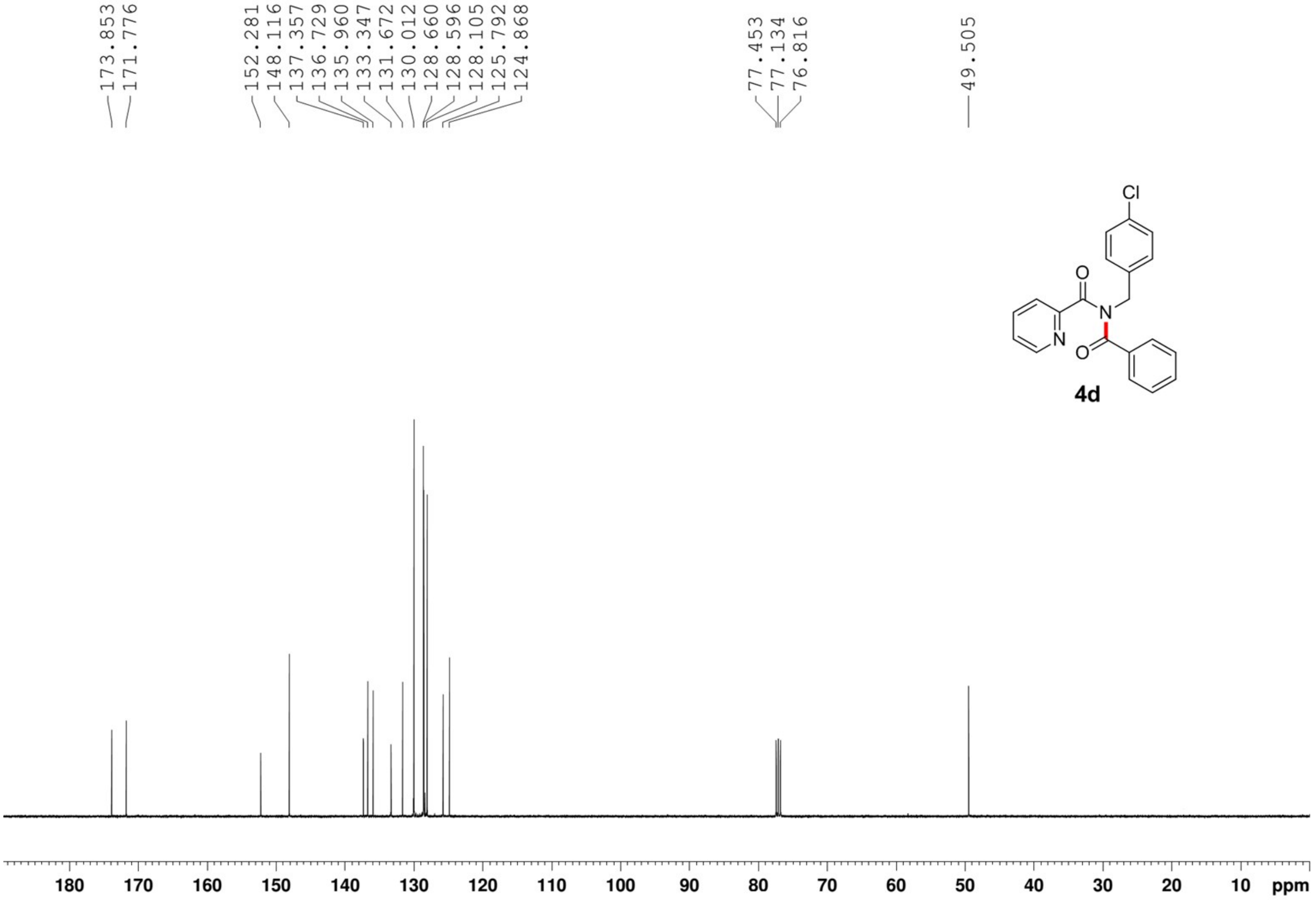
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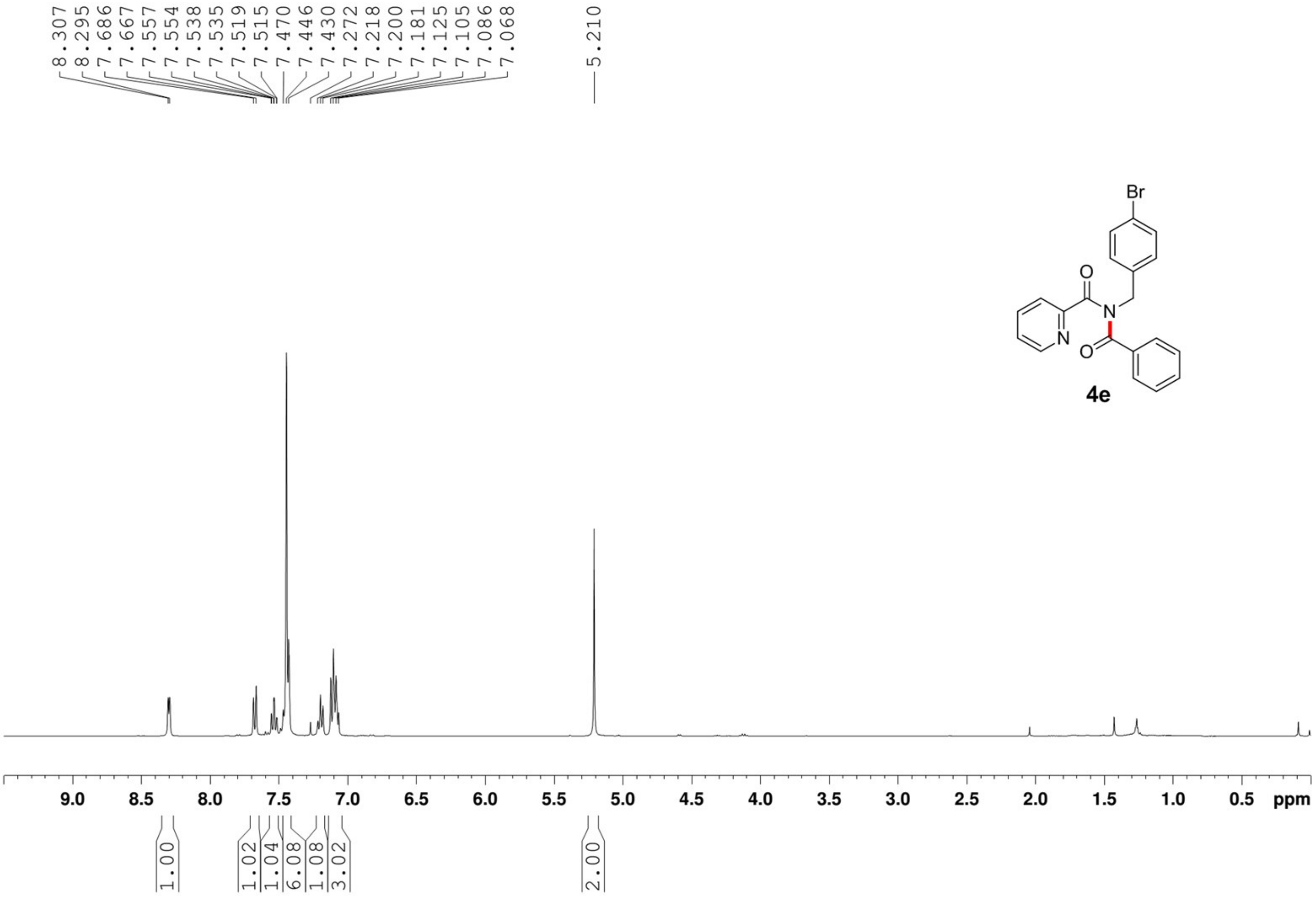
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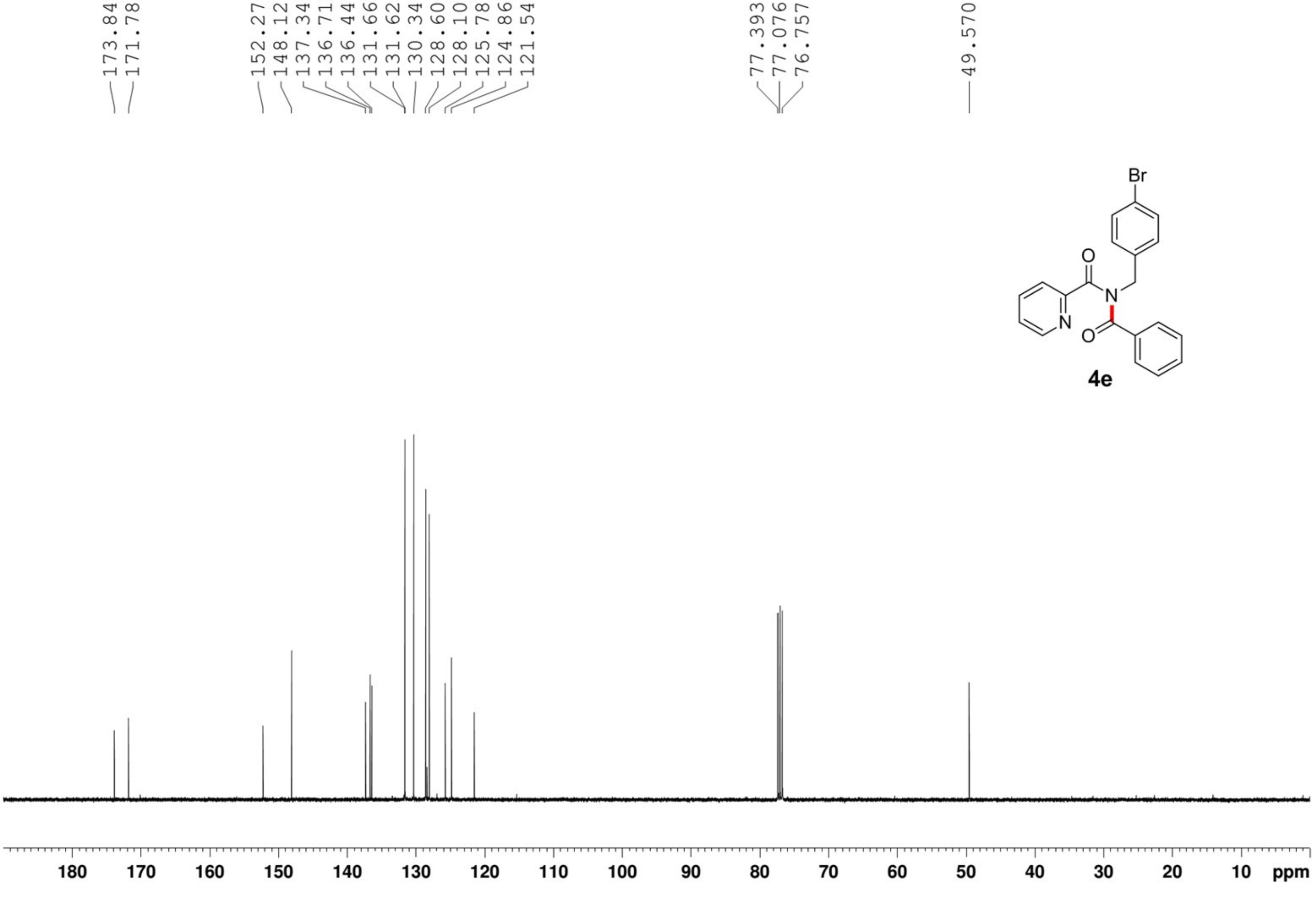
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121.545
77.393
77.076
76.757
49.570
Br
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N
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O
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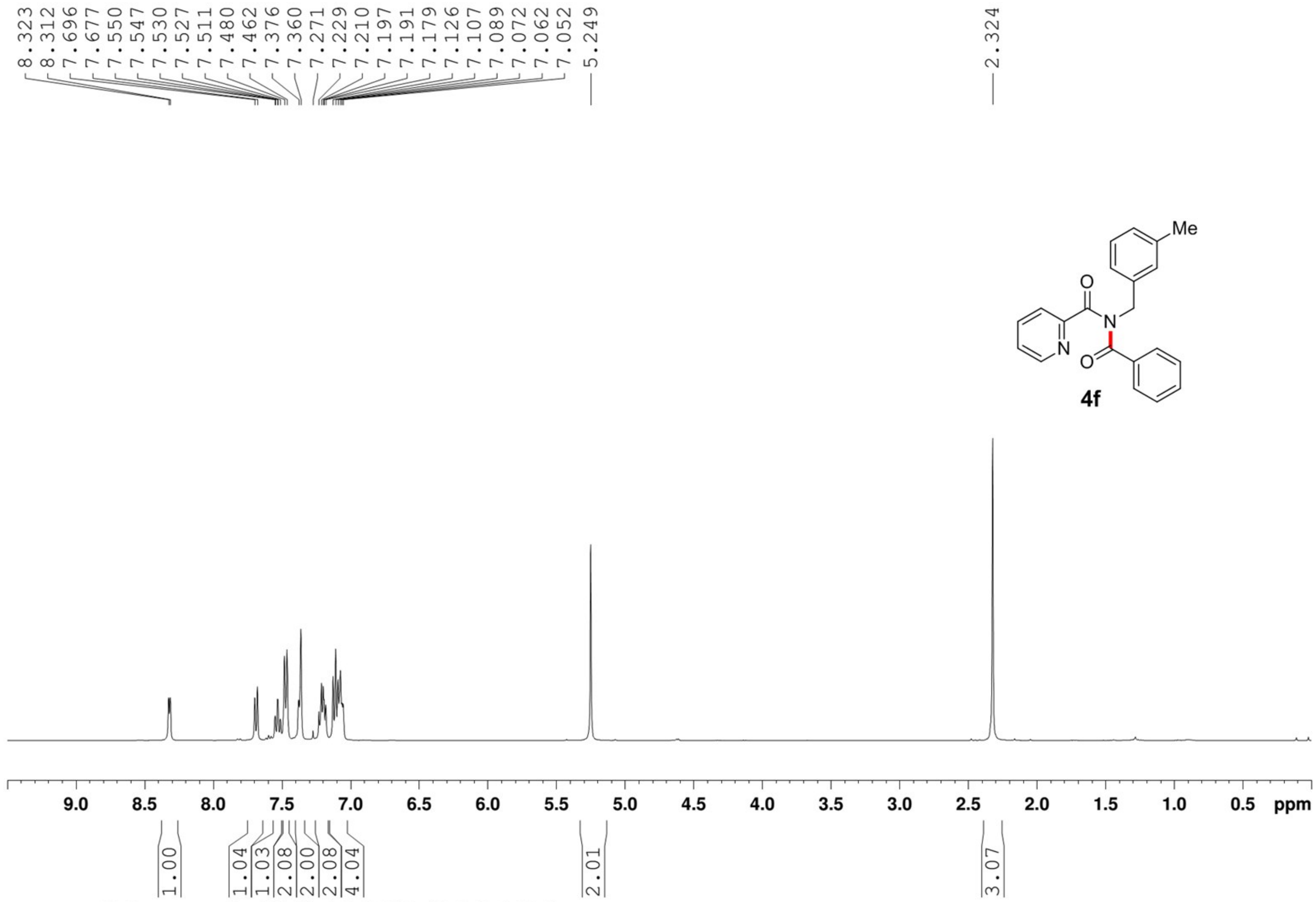
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7.271
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7.210
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7.191
7.179
7.126
7.107
7.089
7.072
7.062
7.052
5.249
2.324
Me
O
N
N
O
4f
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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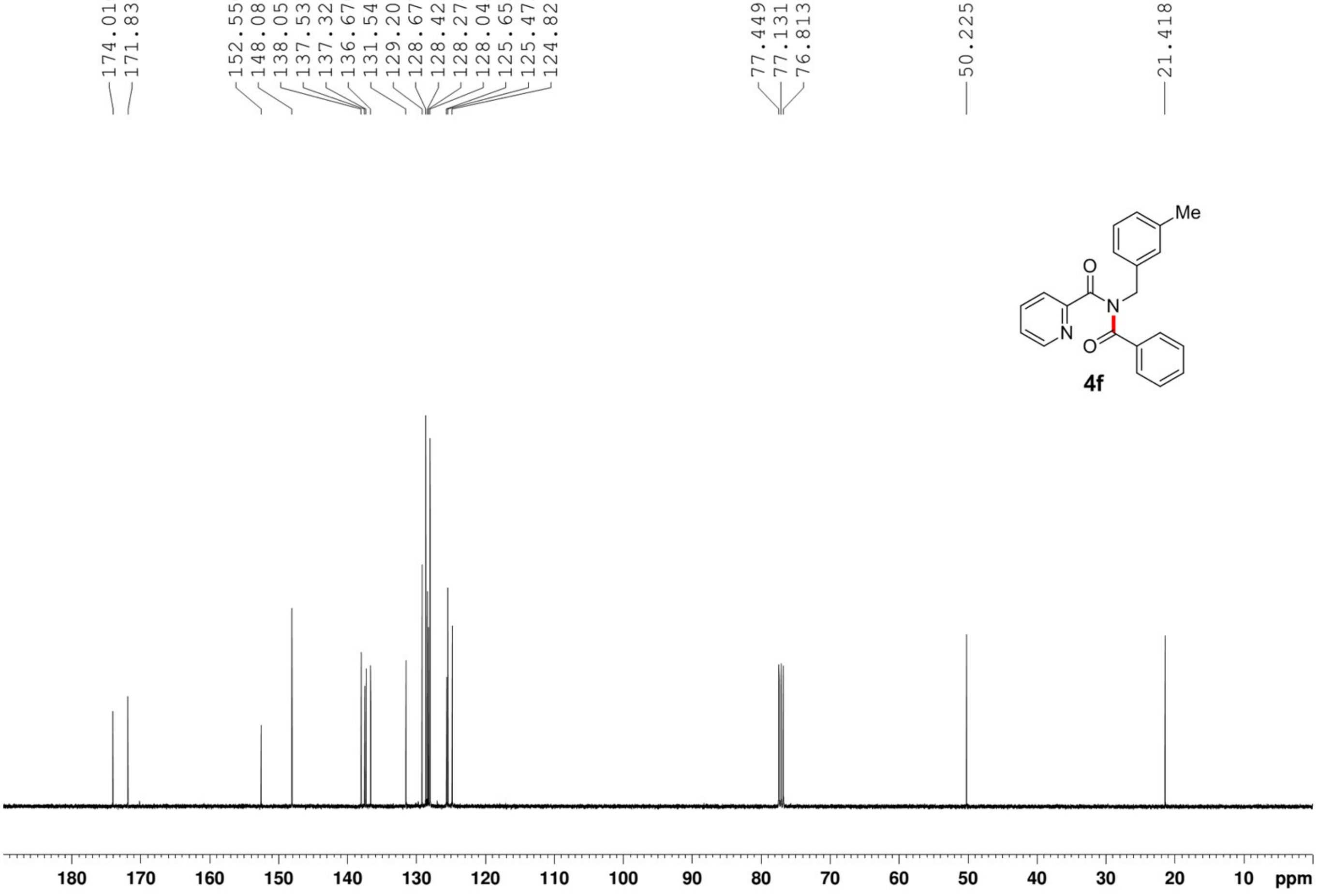
2.01
3.07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128.274
128.045
125.655
125.473
124.829
77.449
77.131
76.813
50.225
21.418
Me
O
N
N
O
4f
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140
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120
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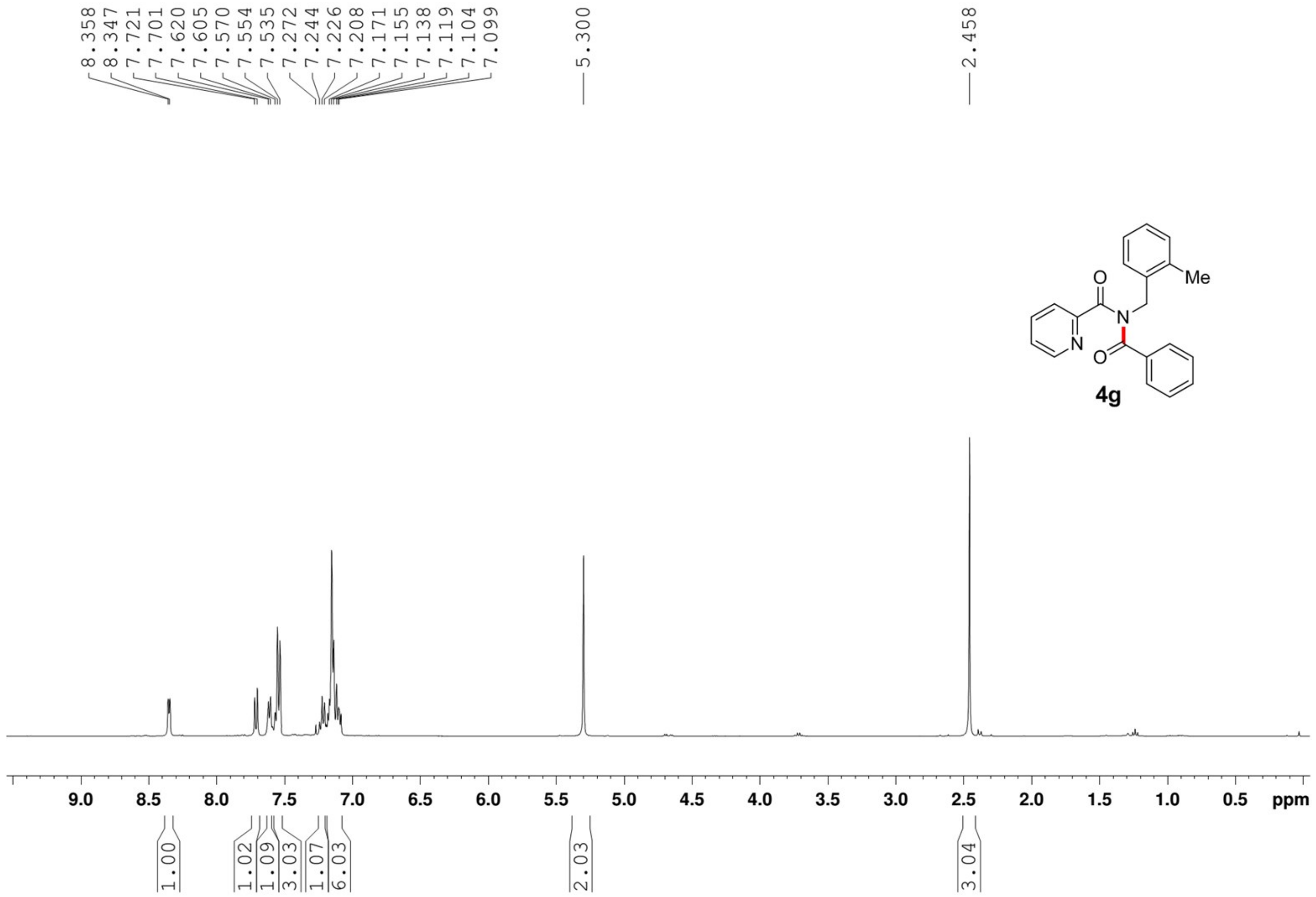
ppm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7.171
7.155
7.138
7.119
7.104
7.099
5.300
2.458
O
N
N
O
Me
4g
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
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3.03
1.07
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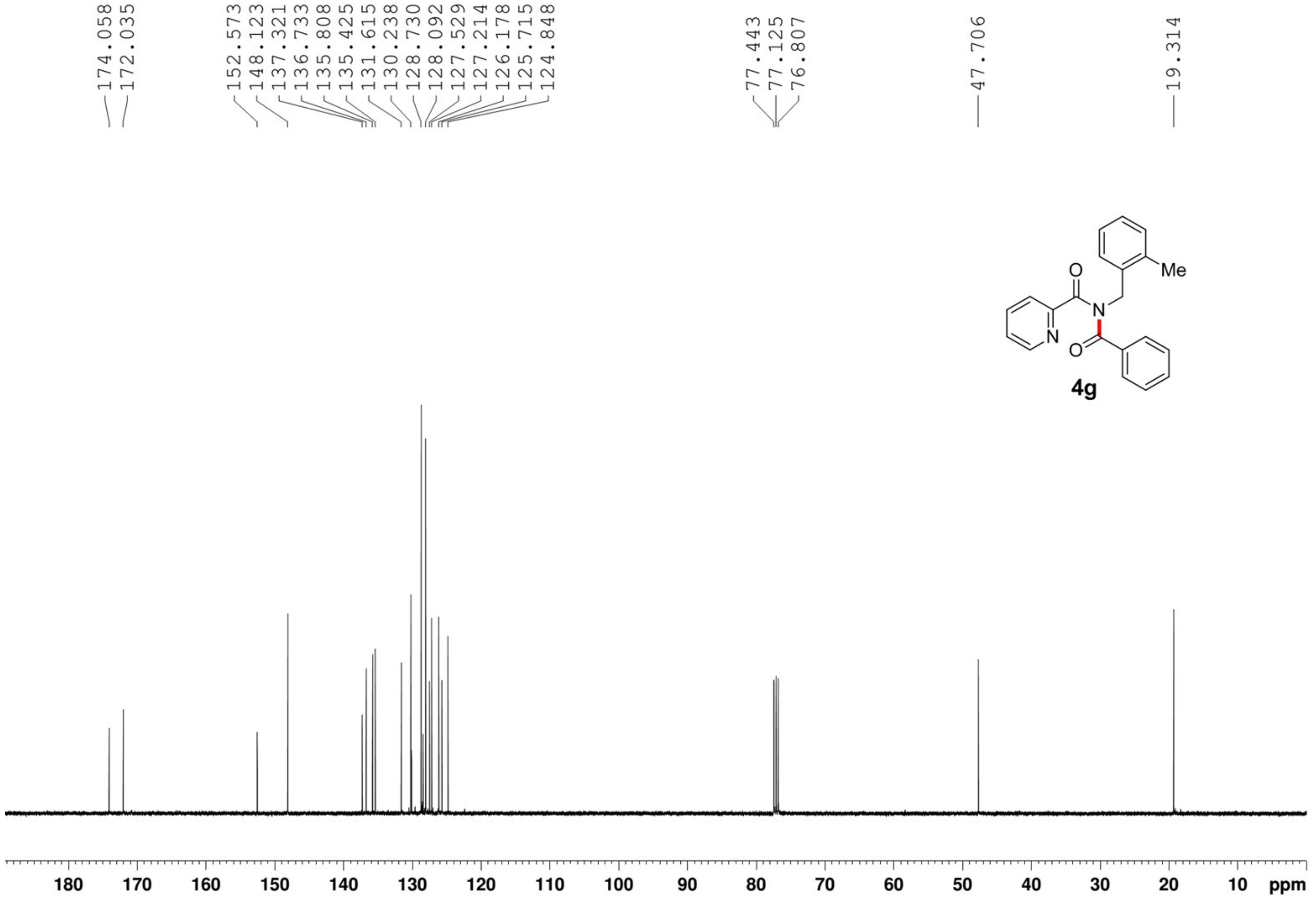
3.04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127.529
127.214
126.178
125.715
124.848
77.443
77.125
76.807
47.706
19.314
O
N
N
O
Me
4g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm