Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

One-pot synthesis of α-iodoketones from alcohols using ammonium iodide and Oxone® in water

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General information

All chemicals used were reagent grade and used as received without further purification. ^{1}H NMR spectra were recorded at 300, 400 and 500 MHz and ^{13}C NMR spectra 75 MHz in $CDCl_3$. The chemical shifts (δ) are reported in ppm units relative to TMS as an internal standard for ^{1}H NMR and $CDCl_3$ for ^{13}C NMR spectra. Coupling constants (J) are reported in hertz (Hz) and multiplicities are indicated as follows: s (singlet), d (doublet), dd (doublet of doublet), t (triplet), q (quartet), and m (multiplet). Mass spectra were recorded under impact (EI) conditions at 70 eV. Column chromatography was carried out using silica gel (230-400 mesh).

General procedure

Oxone® (2.2 mmol) was slowly added to a well stirred solution of NH_4I (2.2 mmol) and alcohol (2 mmol) in water (10 ml) and the reaction mixture was allowed to stirr at 70 °C. After disappearance of alcohol (reaction was monitored by TLC) or after the appropriate time, the organic product mixture was extracted with DCM (3 x 25 mL). The organic layer was washed with 5% aqueous sodium thiosulfate solution (10 mL) and dried over anhydrous Na_2SO_4. The solvent was removed in vacuo and the residue was purified by column chromatography over silica gel using n-hexane-ethyl acetate as eluent to give desired products. All the products were identified by their ^{1}H NMR, ^{13}C NMR and mass spectra.

^{1}H, ^{13}C NMR and Mass Spectral data

2-Iodo-1-phenylethanone[1] (Table 2, entry 1)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.31 (s, 2H), 7.43-7.59 (m, 3H), 7.91-8.0 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ =1.6, 128.8, 130.0, 133.4, 133.7, 192.8.

MS (EI): *m/z* (%) = 246 (M^+) (10), 105 (100), 91 (32), 77 (41), 51 (33).

2-Iodo-1-(4-methylphenyl)ethanone[1] (Table 2, entry 2)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.43 (s, 3H), 4.29 (s, 2H), 7.19-7.31 (m, 2H) 7.81-7.91 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 1.9, 21.8, 129.1, 129.5, 130.9, 144.8, 192.5.

1-(4-Bromophenyl)-2-iodoethanone[1] (Table 2, entry 4)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.26 (s, 2H), 7.62 (d, J = 8.40 Hz, 2H), 7.84 (d, J = 8.40 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 1.1, 129.0, 130.4, 132.1, 191.8.

MS (EI): *m/z* (%) = 324 (M^+) (15), 183 (100), 169 (21), 90 (62), 50 (92).

1-(4-Fluorophenyl)-2-iodoethanone[1] (Table 2, entry 5)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.27 (s, 2H), 7.11-7.19 (m, 2H), 7.96-8.05 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 1.3, 115.9 (d, J_{CF} = 22.0 Hz), 129.8, 131.7 (d, J_{CF} = 9.9 Hz), 165.9 (d, J_{CF} = 256.4 Hz), 191.3.

2-Iodo-1-(4-nitrophenyl)ethanone[1] (Table 2, entry 6)

^{1}H NMR (300 MHz, $CDCl_3$) δ = 4.34 (s, 2H), 8.14 (d, J = 8.8 Hz, 2H), 8.33 (d, J = 8.8 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ = 0.9, 123.9, 130.0, 137.9, 150.5, 191.2.

2-Iodo-1-phenypropanone[1] (Table 2, entry 7)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.07 (d, J = 6.1 Hz, 3H), 5.44 (q, J = 6.1 Hz, 1H), 7.41-7.59 (m, 3H), 7.98 (d, J = 7.2 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 18.1, 21.9, 128.6, 128.7, 130.1, 133.4, 194.7.

MS (EI): *m/z* (%) = 260 (M^+) (10), 105 (100), 91 (32), 77 (92), 51 (54).

2-Iodocycloheptanone[2] (Table 2, entry 8)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.16-2.49 (m, 9H), 2.88-2.98 (m, 1H), 4.51 (dd, J = 5.28, 10.57 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 25.4, 28.8, 30.0, 31.9, 35.6, 38.1, 207.7.

2,7-Diiodocyclooctanone (Table 2, entry 9)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.32-1.69 (m, 6H), 2.34-2.43 (m, 4H), 4.76-4.83 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 22.4, 23.6, 23.8, 28.1, 37.5, 205.4.

1-Iodo-2-nonanone (Table 2, entry 10)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.89 (t, J = 6.8 Hz, 3H), 1.23-1.35 (m, 8H), 1.55-1.67 (m, 2H), 2.69 (t, J = 7.5 Hz, 2H), 3.73 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 6.2, 14.0, 22.5, 24.1, 28.9, 31.6, 39.3, 203.2.

3-Iodo-2-nonanone (Table 2, entry 10)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.82-0.97 (m, 3H), 1.19-1.39 (m, 8H), 1.83-1.97 (m, 2H), 2.39 (s, 3H), 4.39 (t, J = 7.5 Hz, 1H).

1,3-Diiodo-2-nonanone (Table 2, entry 10)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.89 (t, J = 6.7 Hz, 3H), 1.24-1.37 (m, 8H), 1.92-2.07 (m, 2H), 3.85 (d, J = 9.8 Hz, 1H), 4.36 (d, J = 9.8 Hz, 1H), 4.95 (t, J = 7.5 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 2.6, 13.9, 22.4, 28.4, 29.0, 31.4, 34.3, 196.3.

1,3-Diiodo-2-octanone (Table 2, entry 11)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.84-0.93 (m, 3H), 1.27-1.38 (m, 6H), 1.92-2.07 (m, 2H), 3.85 (d, J = 9.8 Hz, 1H), 4.36 (d, J = 9.8 Hz, 1H), 4.95 (t, J = 7.5 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 2.5, 13.9, 22.3, 28.4, 28.8, 30.9, 34.3, 196.4.

2-Iodo-3-octanone (Table 2, entry 12)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.89 (t, J = 6.8 Hz, 3H), 1.23-1.35 (m, 4H), 1.58-1.71 (m, 2H), 1.89 (d, J = 7.5 Hz, 3H), 2.54-2.67 (m, 1H), 2.80-2.94 (m, 1H), 4.62 (q, J = 6.8 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.9, 21.6, 22.4, 24.1, 24.7, 31.2, 38.4, 205.3.

4-Iodo-3-octanone (Table 2, entry 12)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.91 (t, J = 6.9 Hz, 3H), 1.14 (t, J = 7.3 Hz, 3H), 1.23-1.45 (m, 4H), 1.86-1.99 (m, 2H), 2.55-2.70 (m, 1H), 2.79-2.95 (m, 1H), 4.46 (t, J = 7.5 Hz, 1H).

2,4-Diiodo-3-octanone (Table 2, entry 12)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 0.93 (t, J = 7.2 Hz, 3H), 1.35-1.45 (m, 4H), 1.98 (d, J = 6.7 Hz, 3H), 2.01-2.13 (m, 2H), 5.02 (t, J = 7.4 Hz, 1H), 5.17 (q, J = 6.7 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 13.8, 21.8, 21.9, 22.2, 28.9, 31.0, 33.8, 197.1.

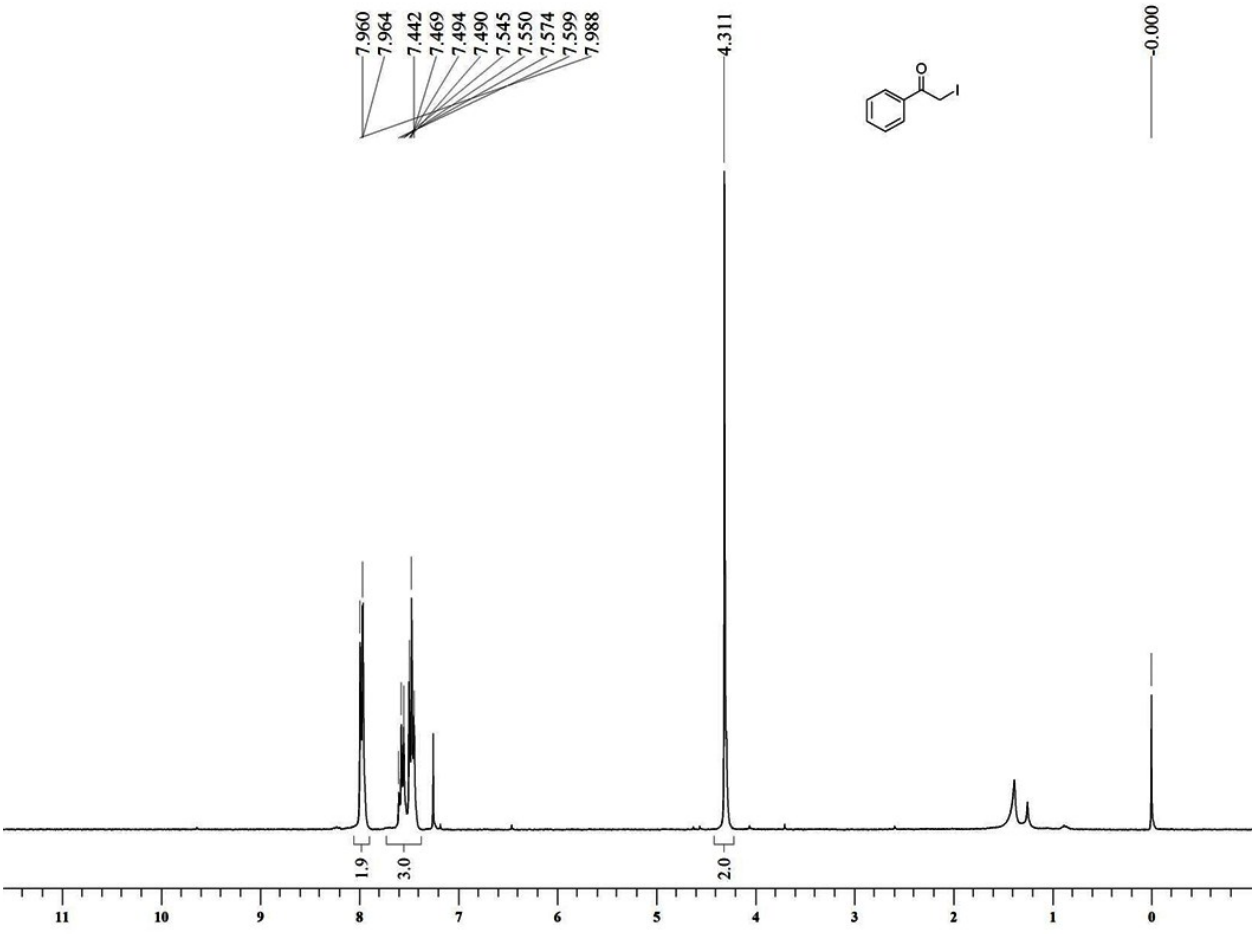
1-Iodo-4-phenyl-2-butanone[3] (Table 2, entry 13)

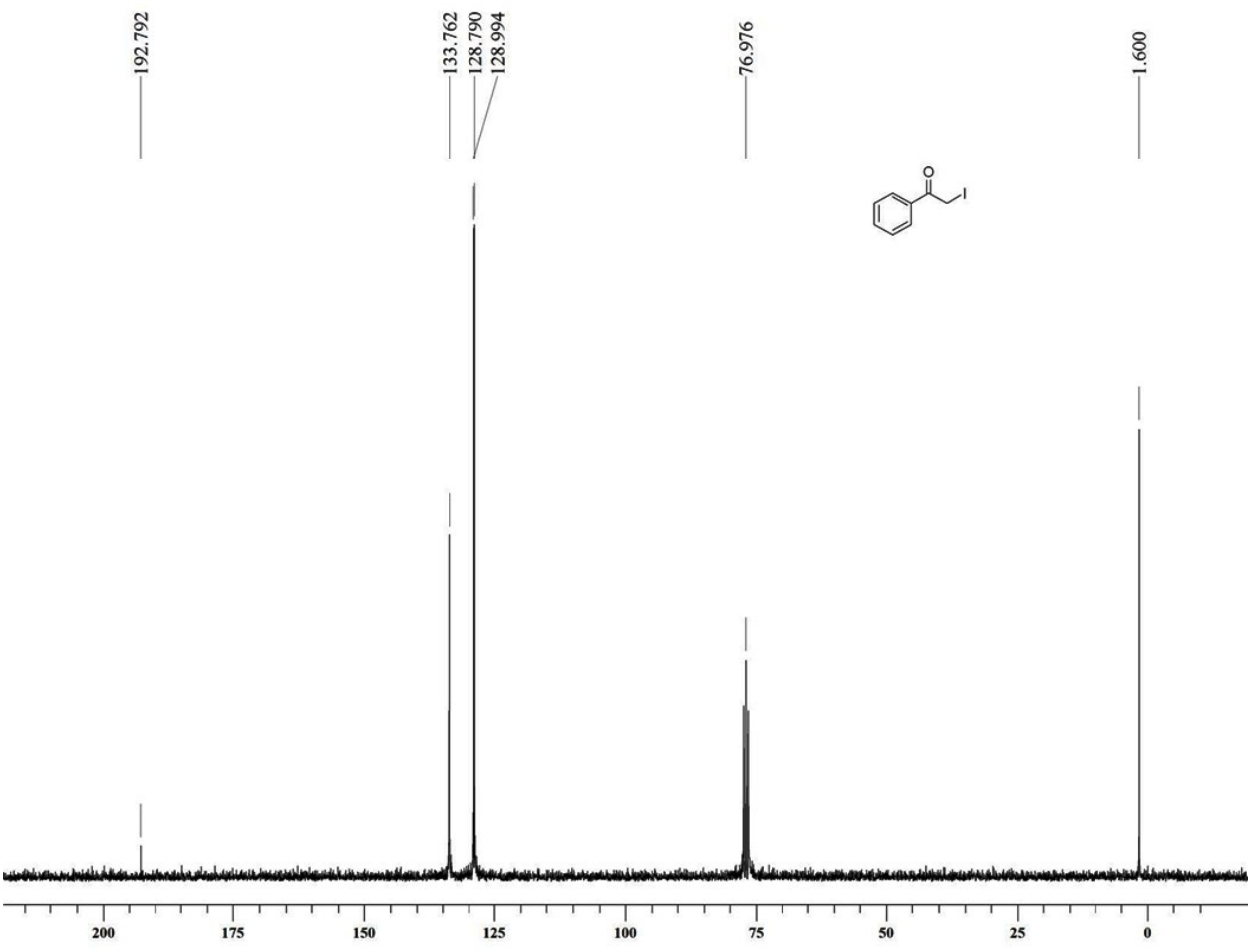
^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.87-3.05 (m, 4H), 3.69 (s, 2H), 7.11-7.29 (m, 5H).

3-Iodo-4-phenyl-2-butanone (Table 2, entry 13)

^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.33 (s, 3H), 3.09-3.21 (m, 1H), 3.41-3.52 (m, 1H), 4.64 (m, 1H), 7.11-7.33 (m, 5H).

Copies of ^{1}H and ^{13}C NMR Spectra

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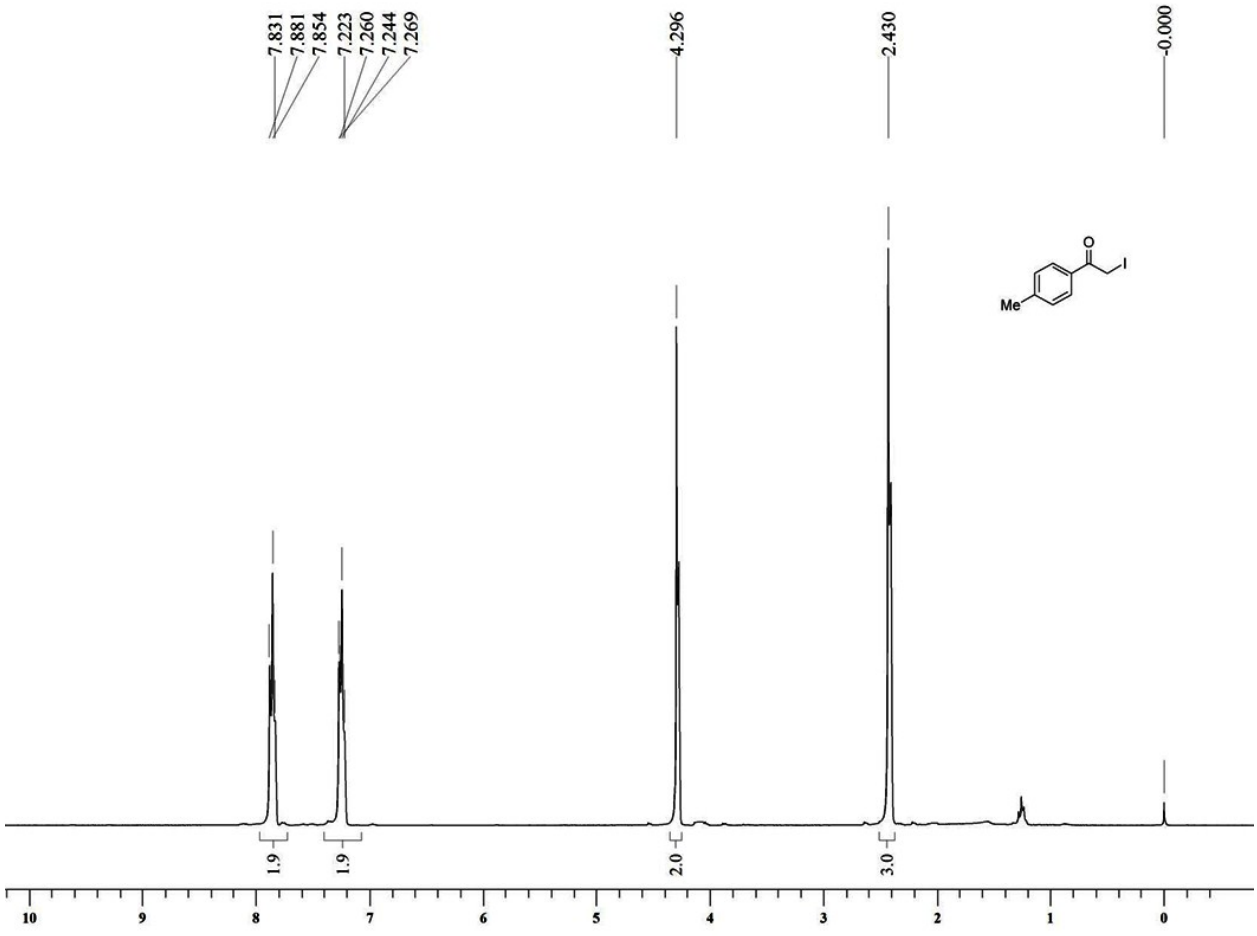
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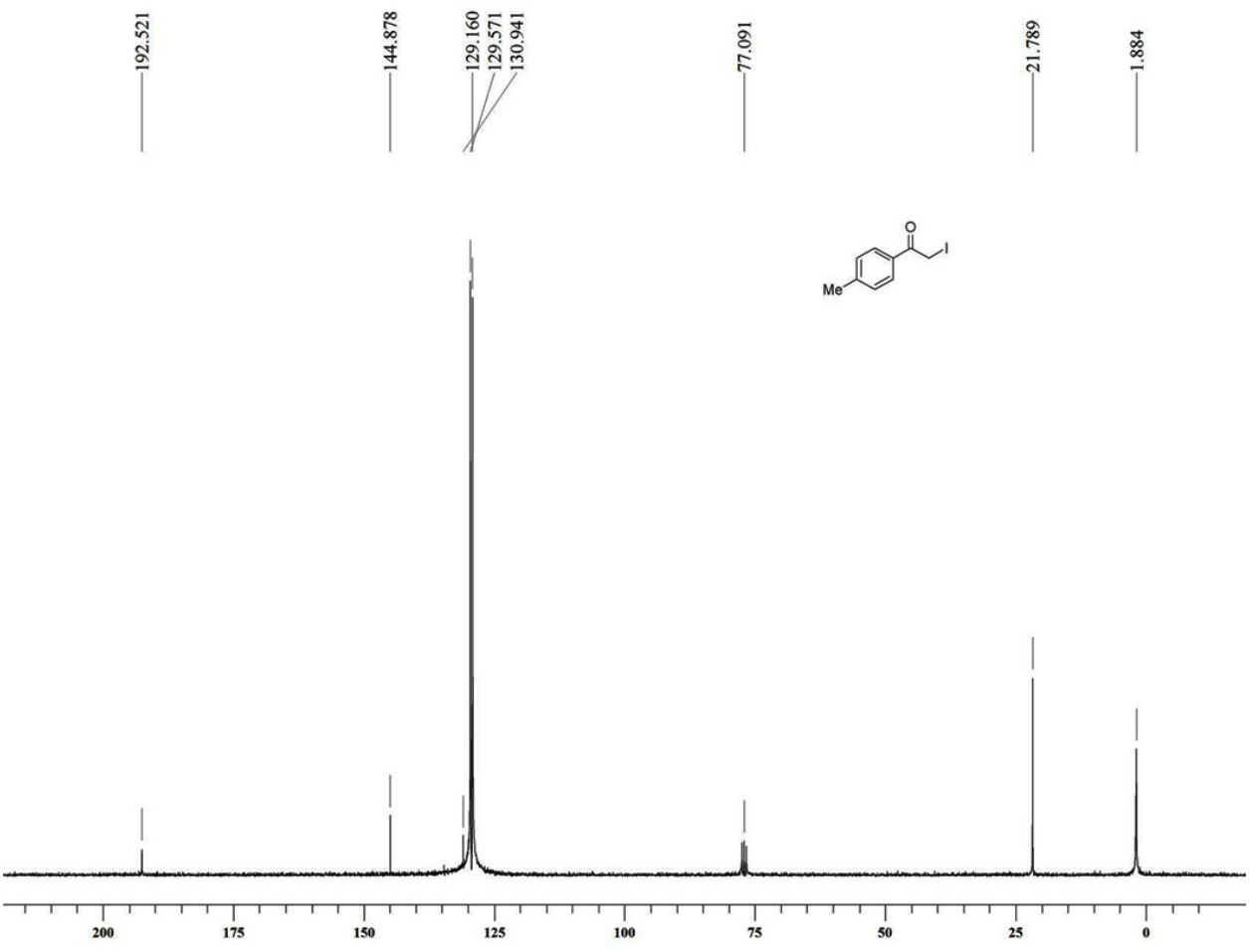
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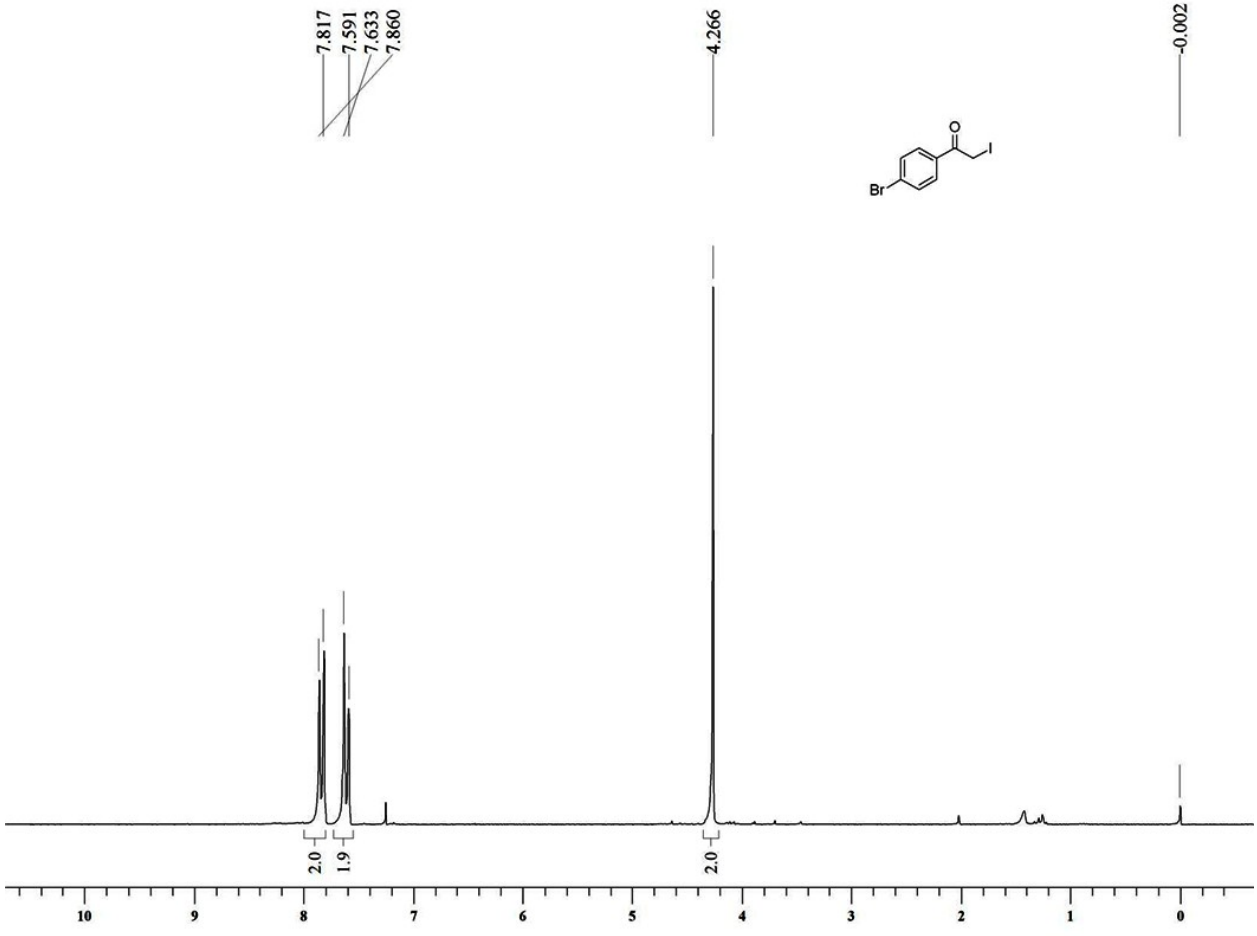
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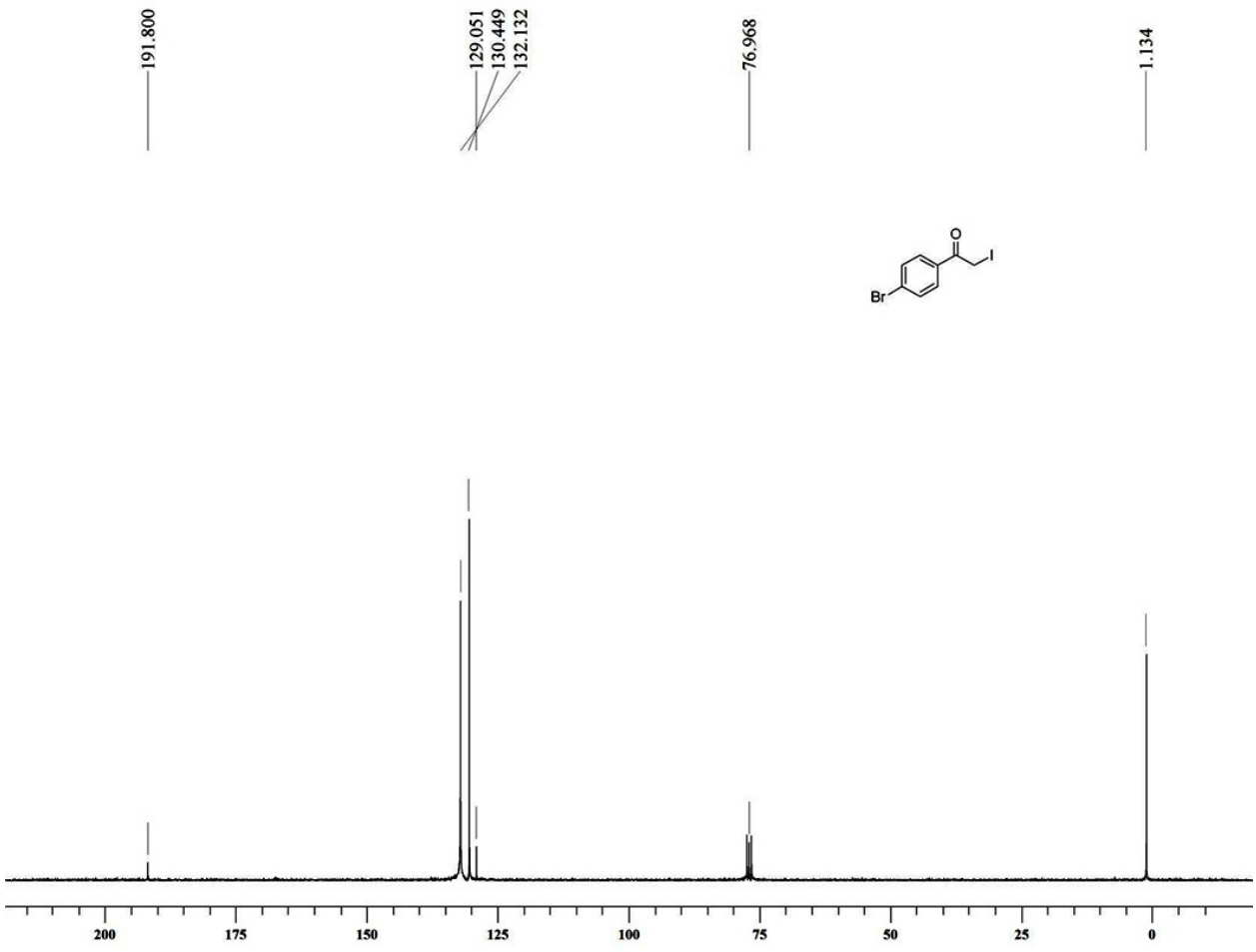
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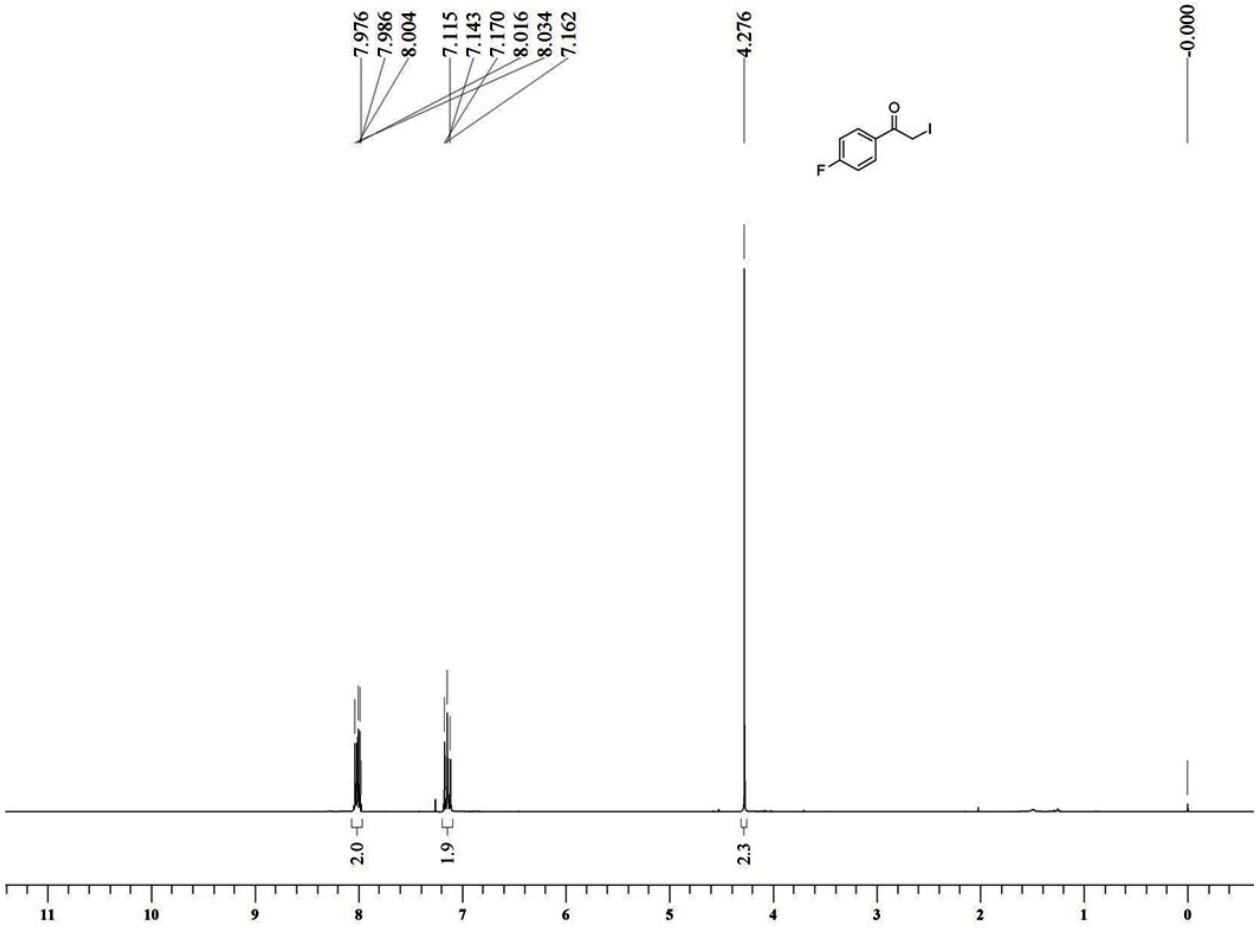
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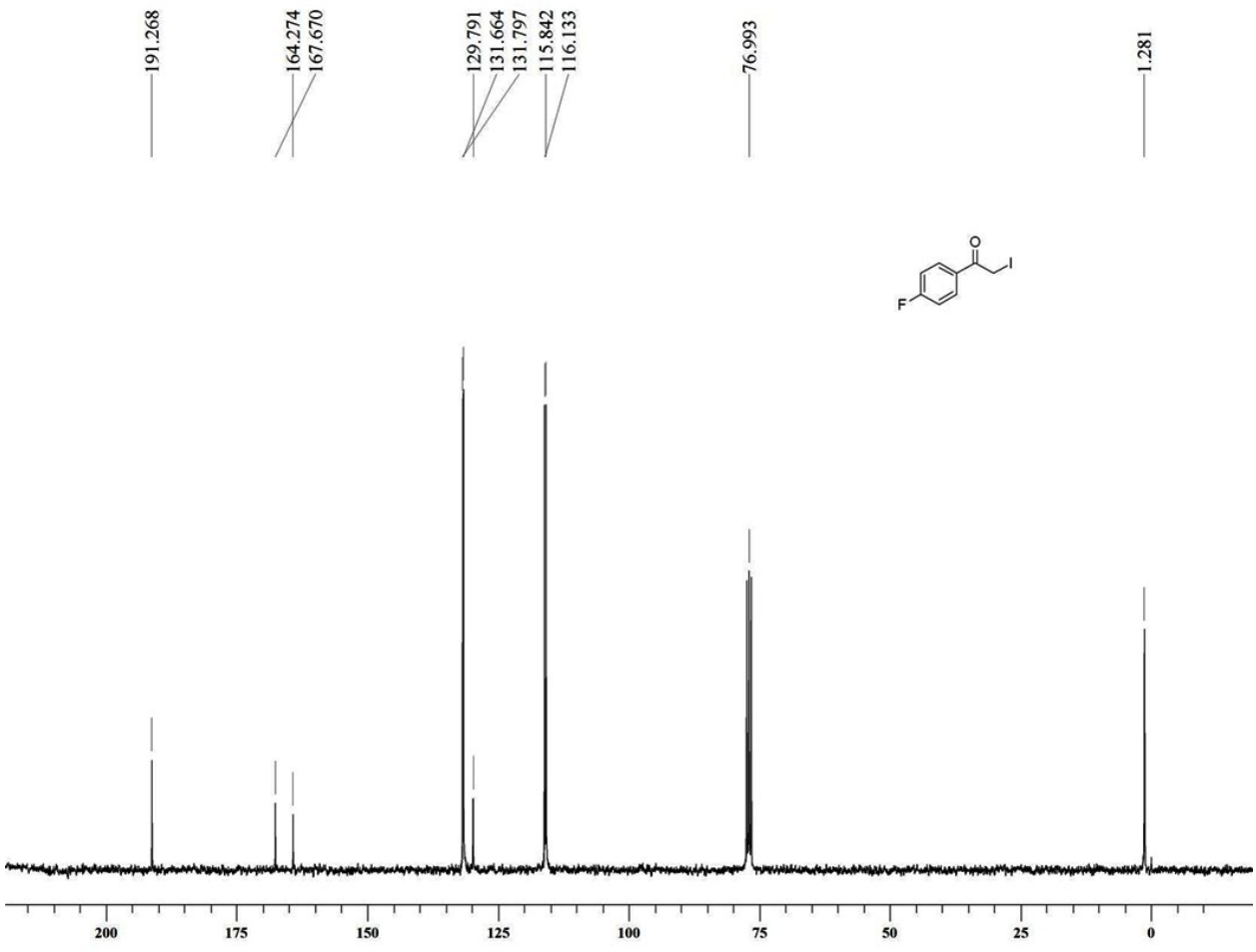
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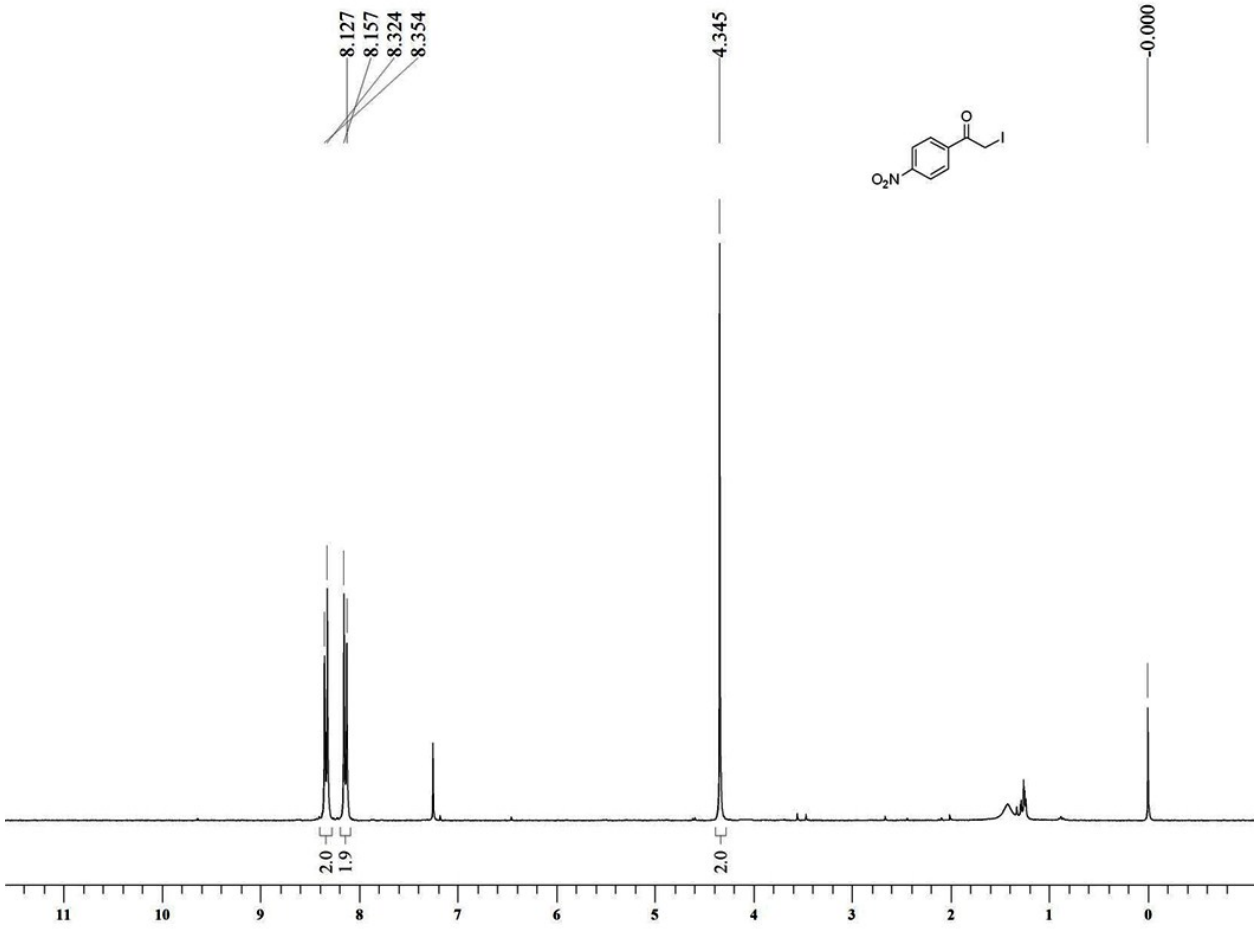
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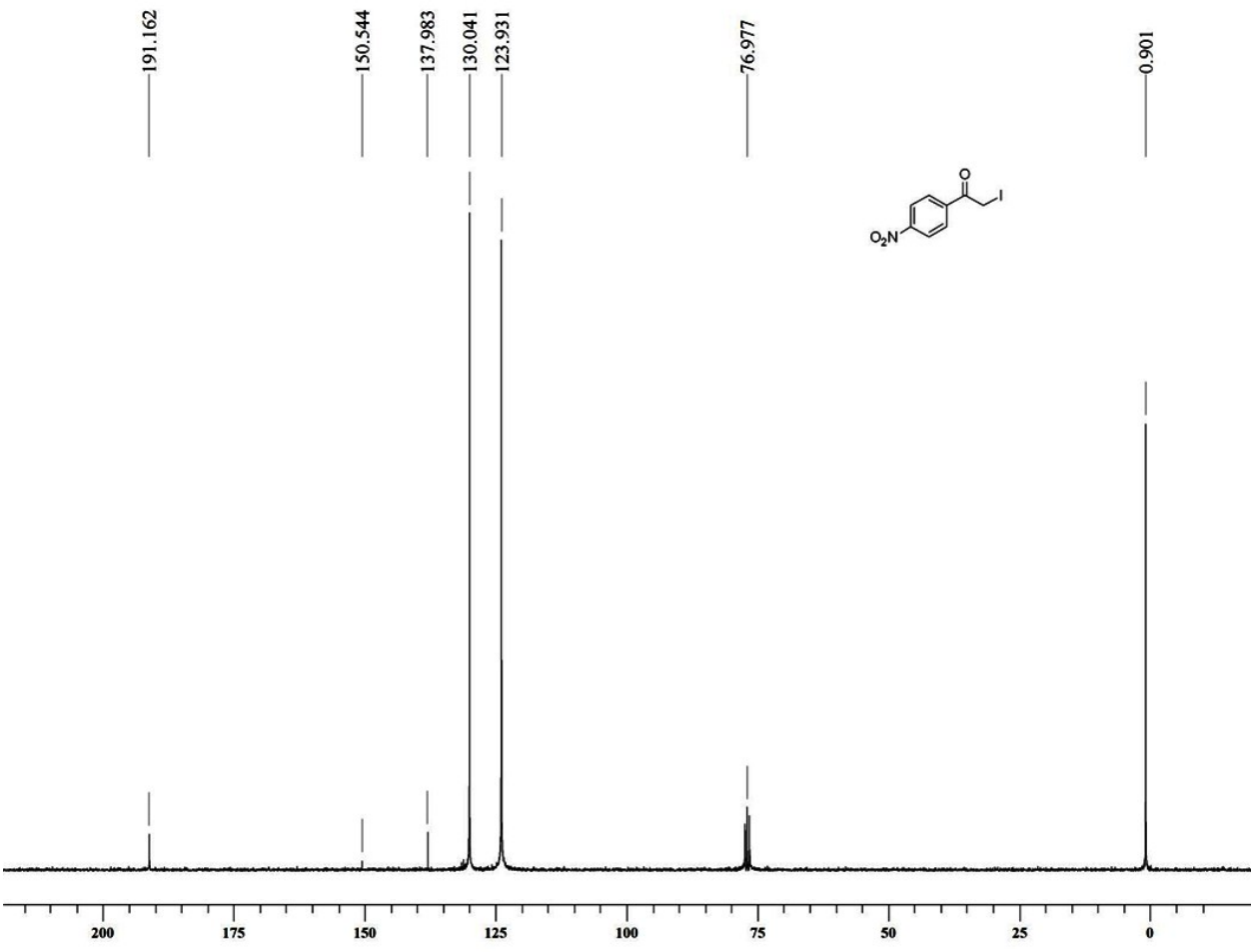
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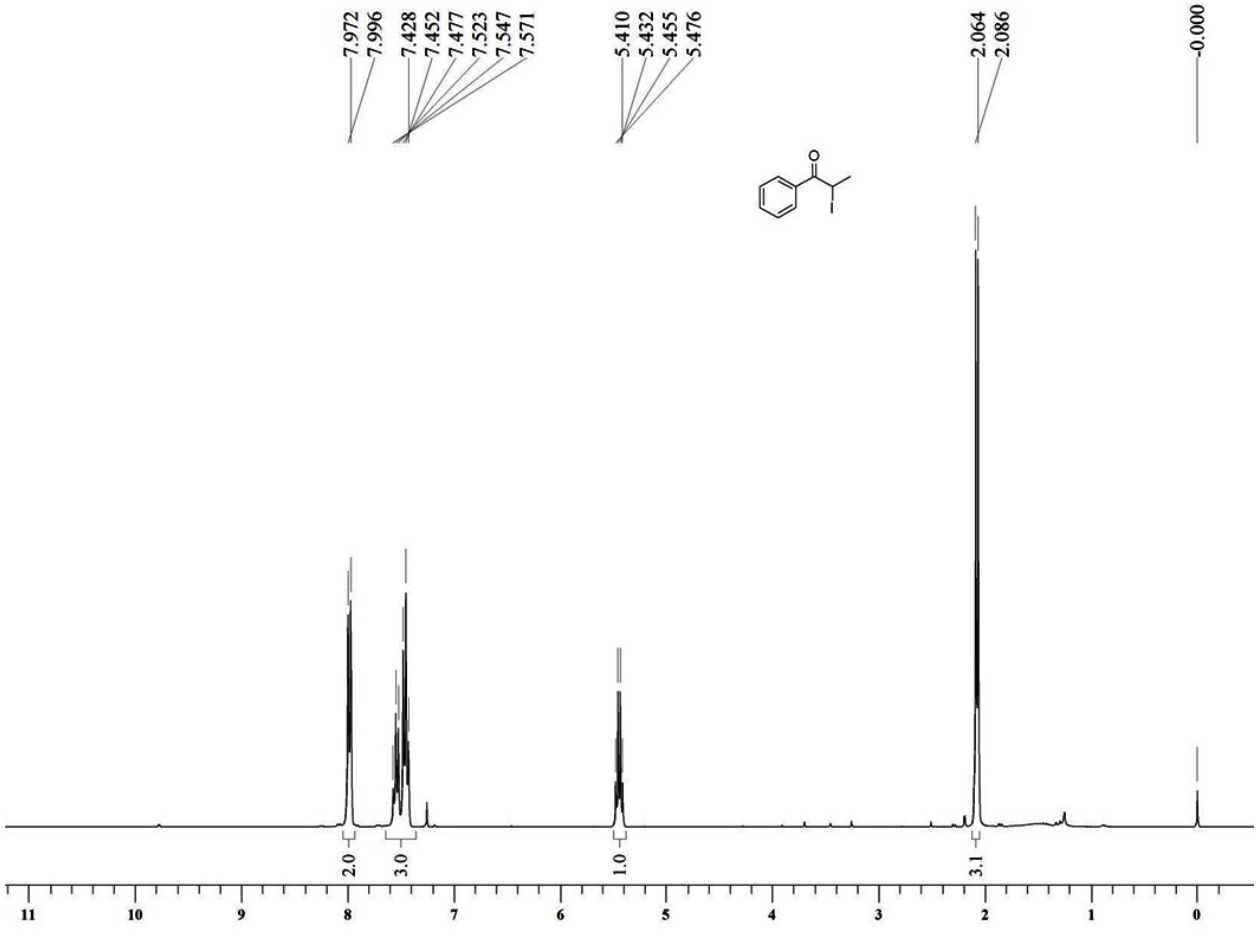
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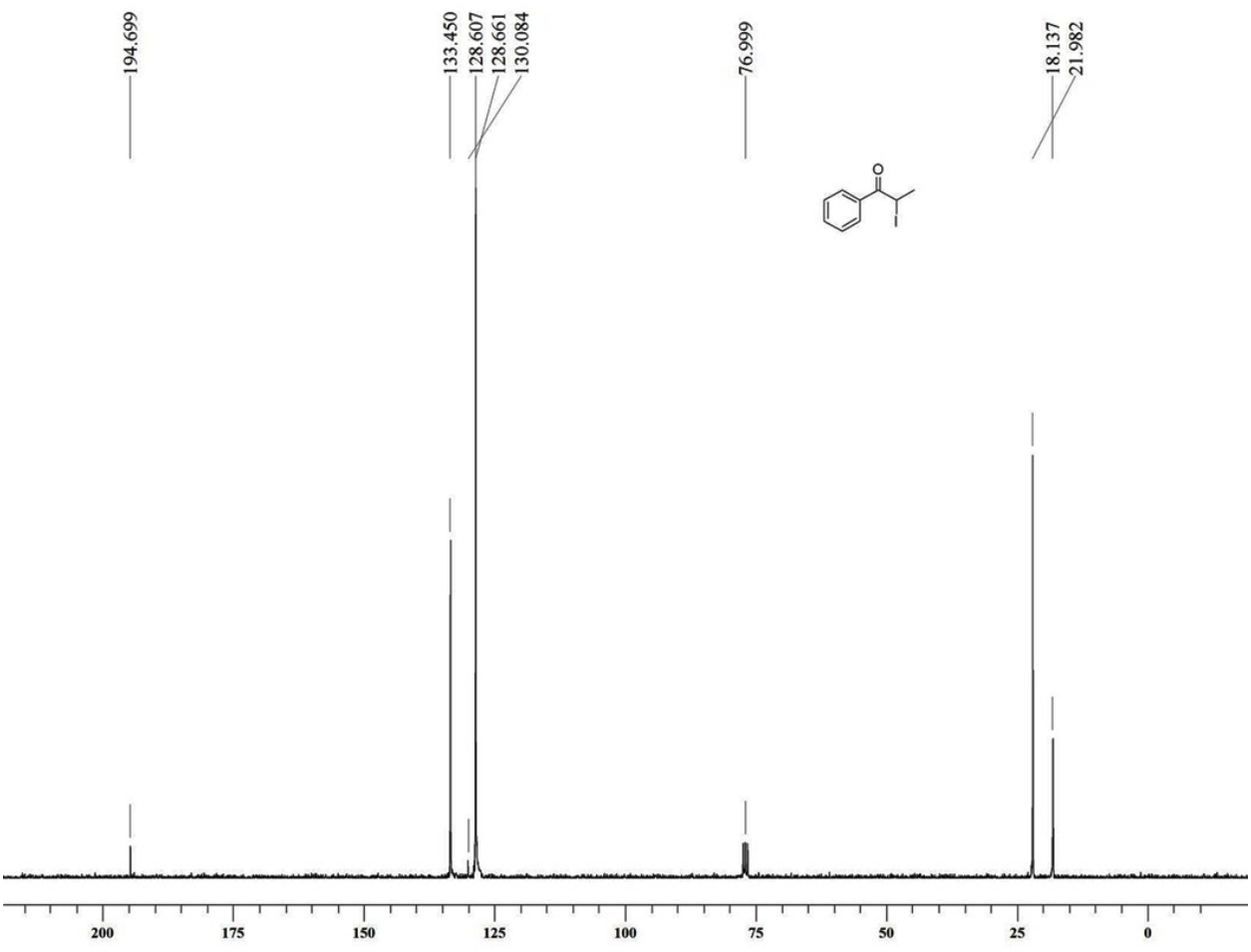
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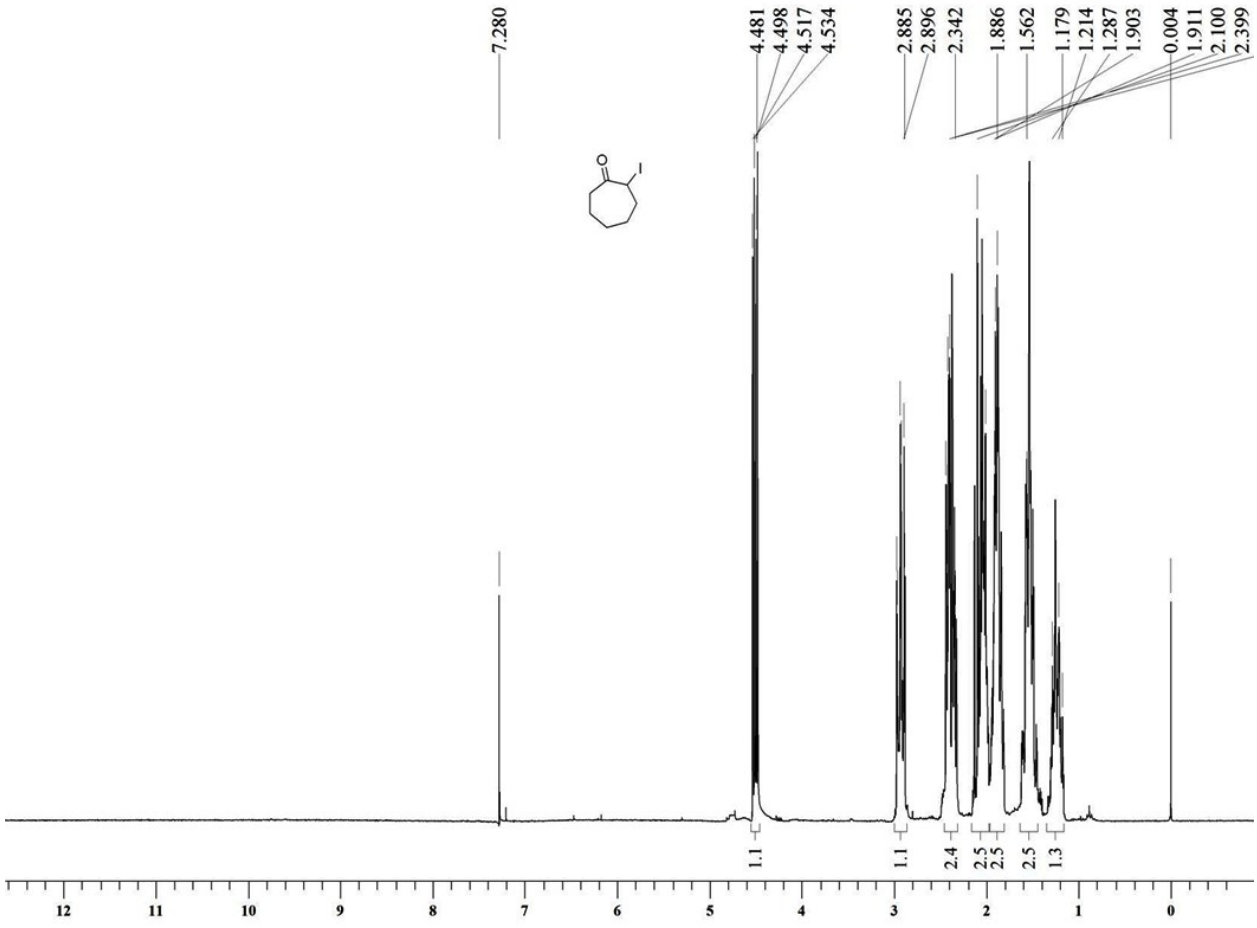
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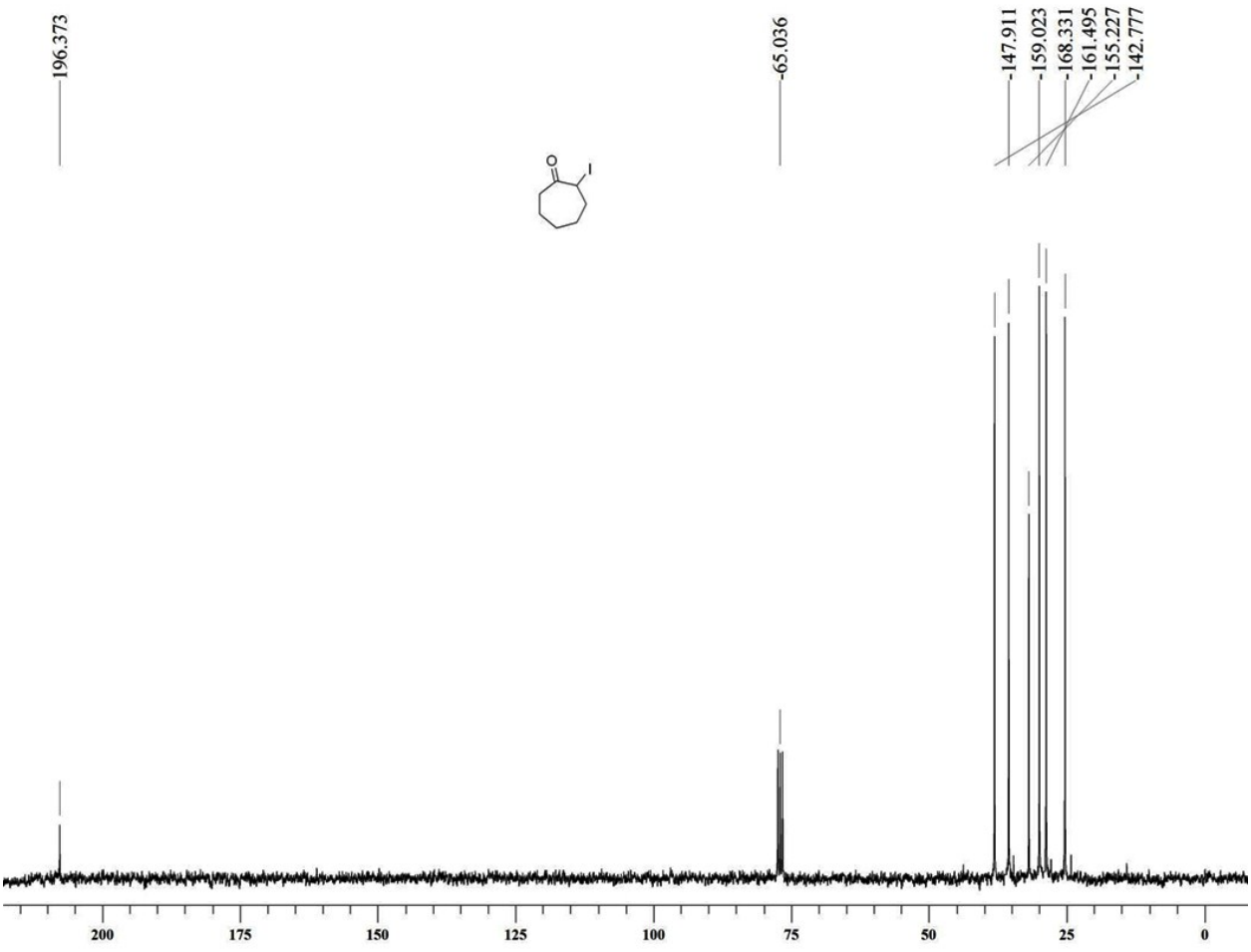
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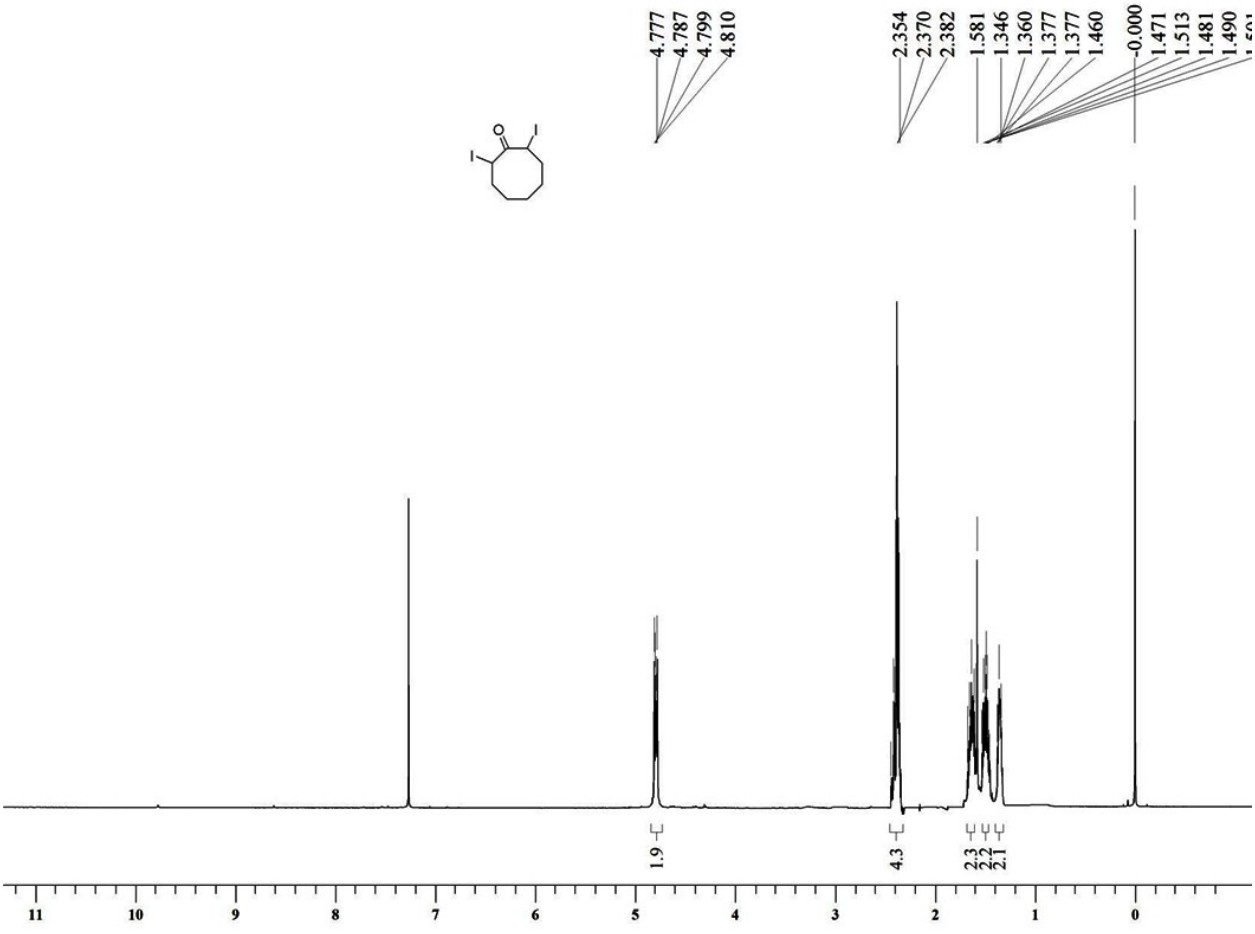
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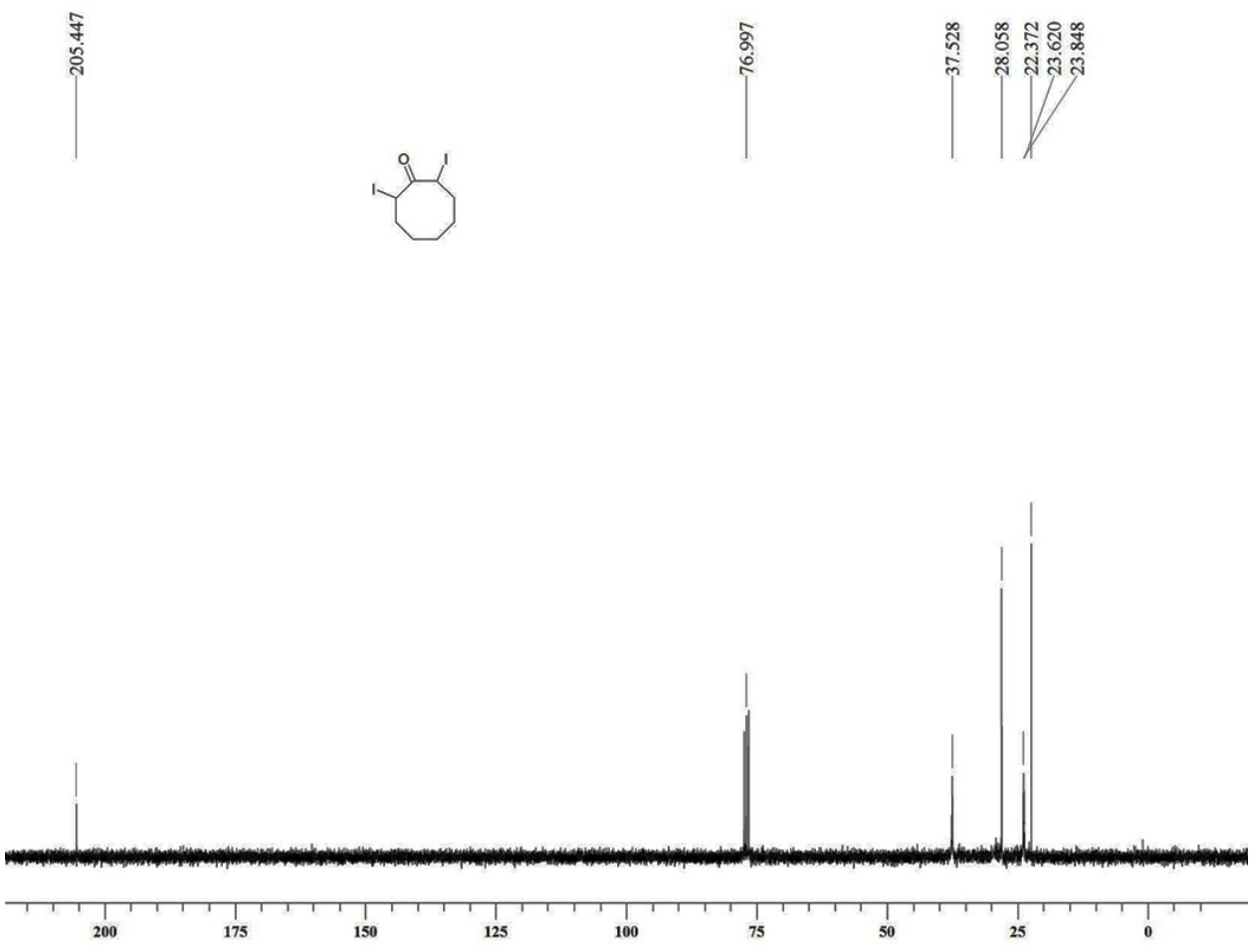
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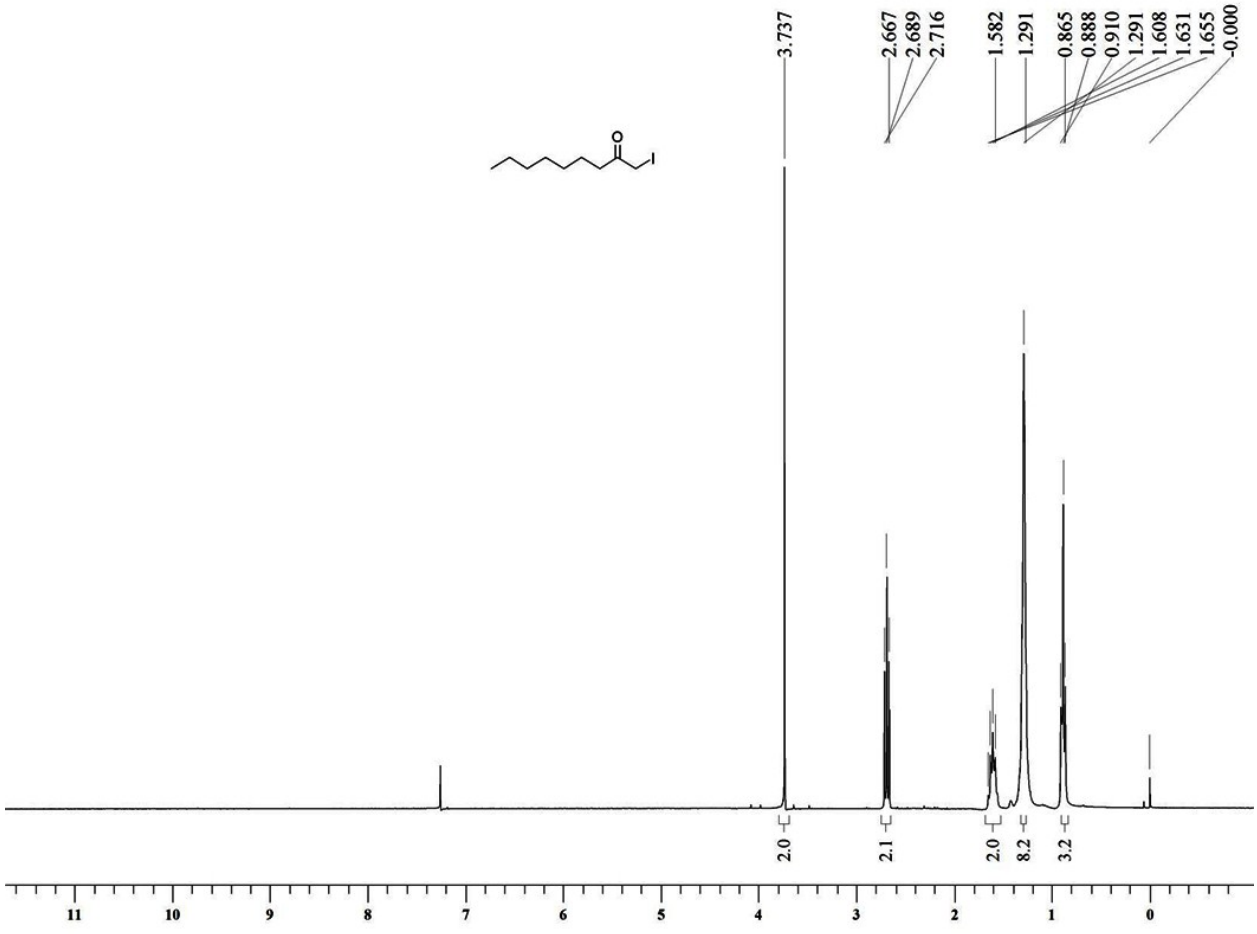
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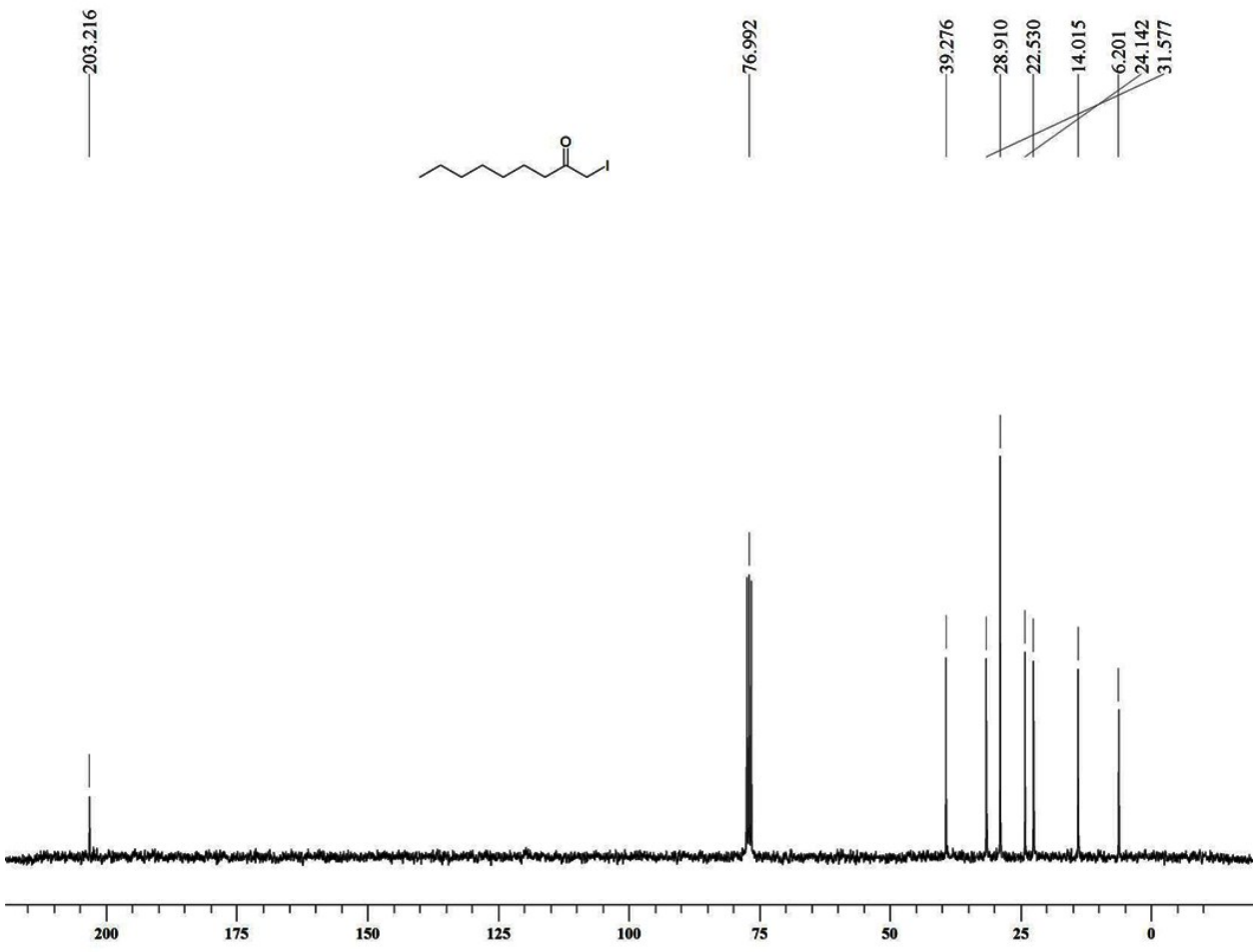
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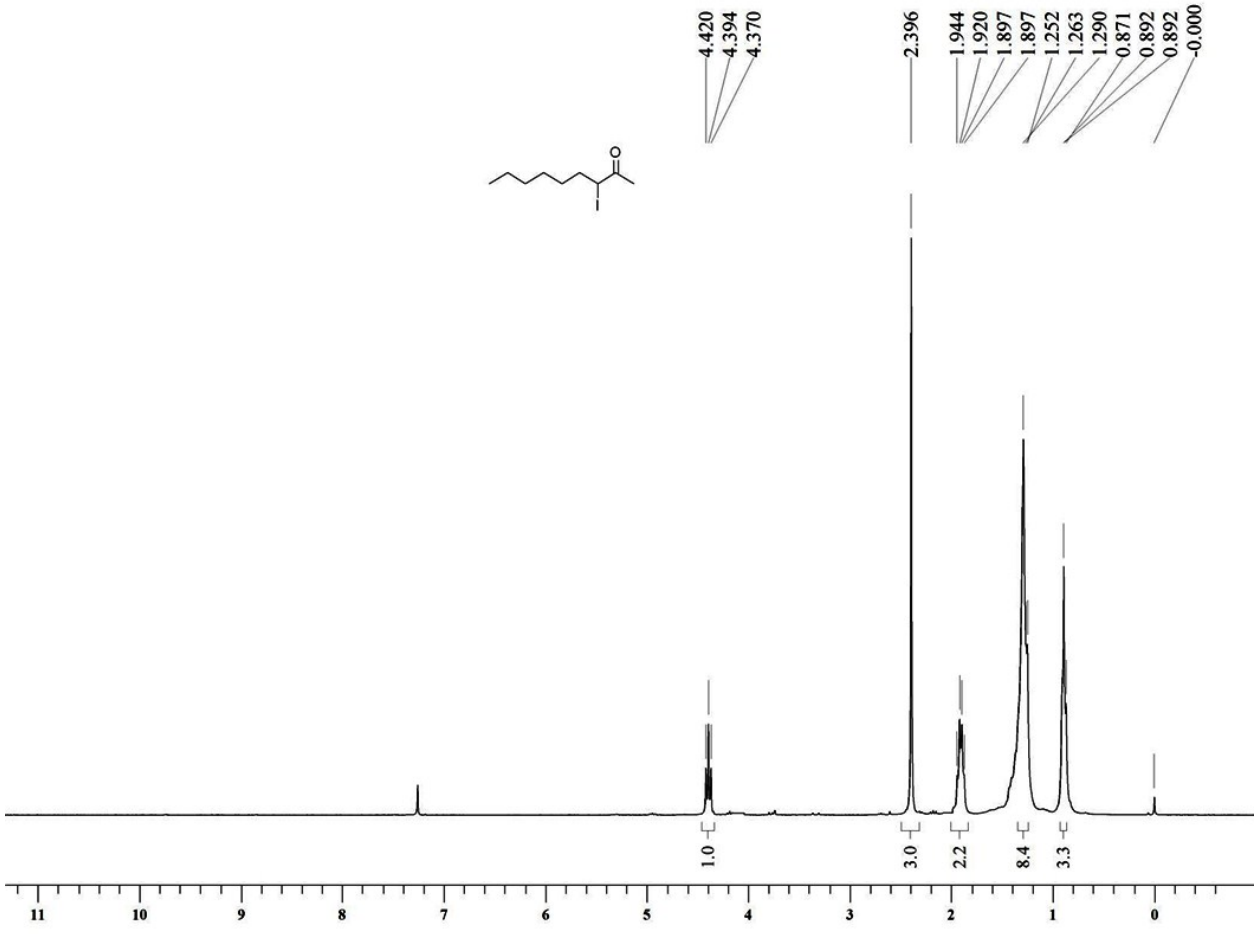
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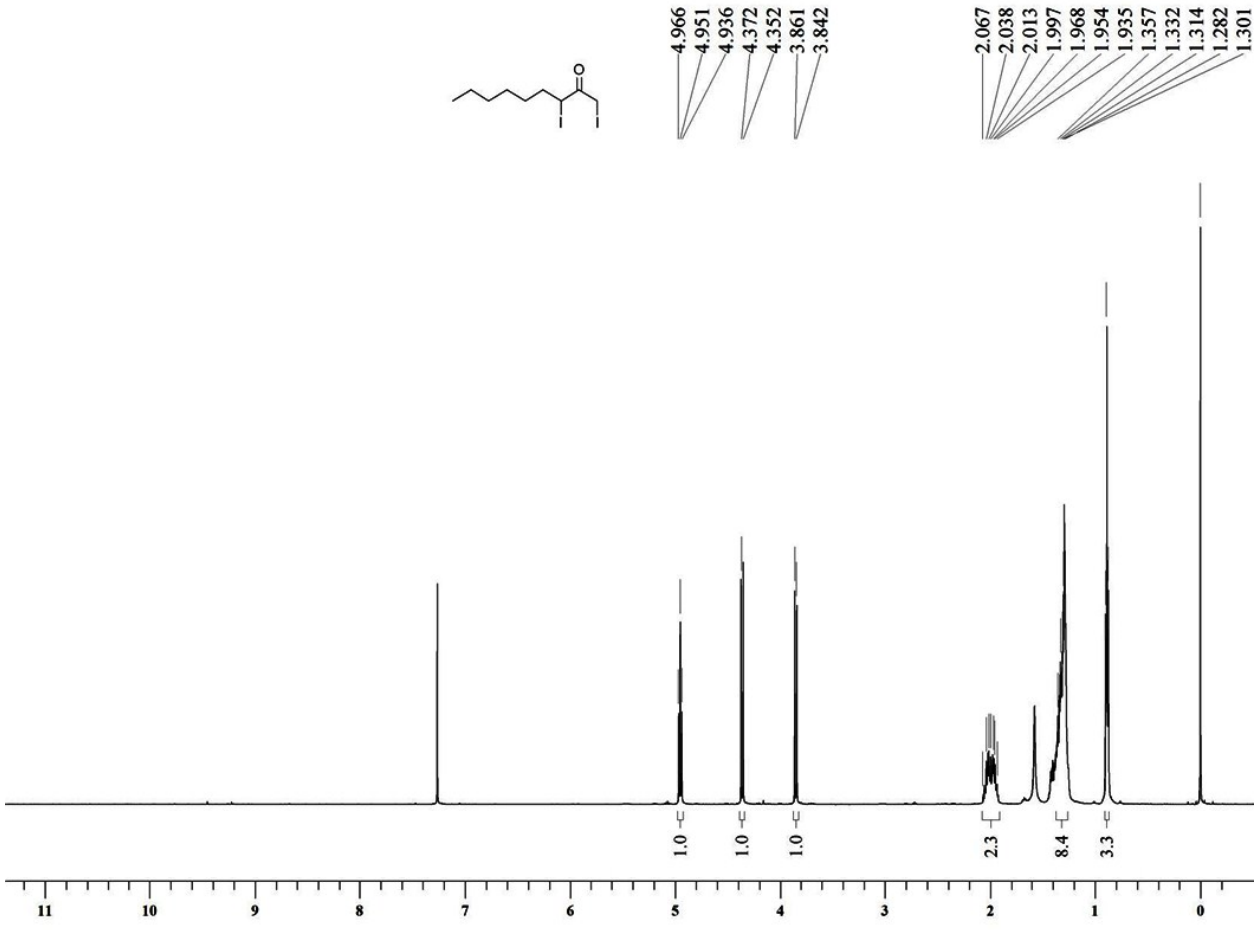
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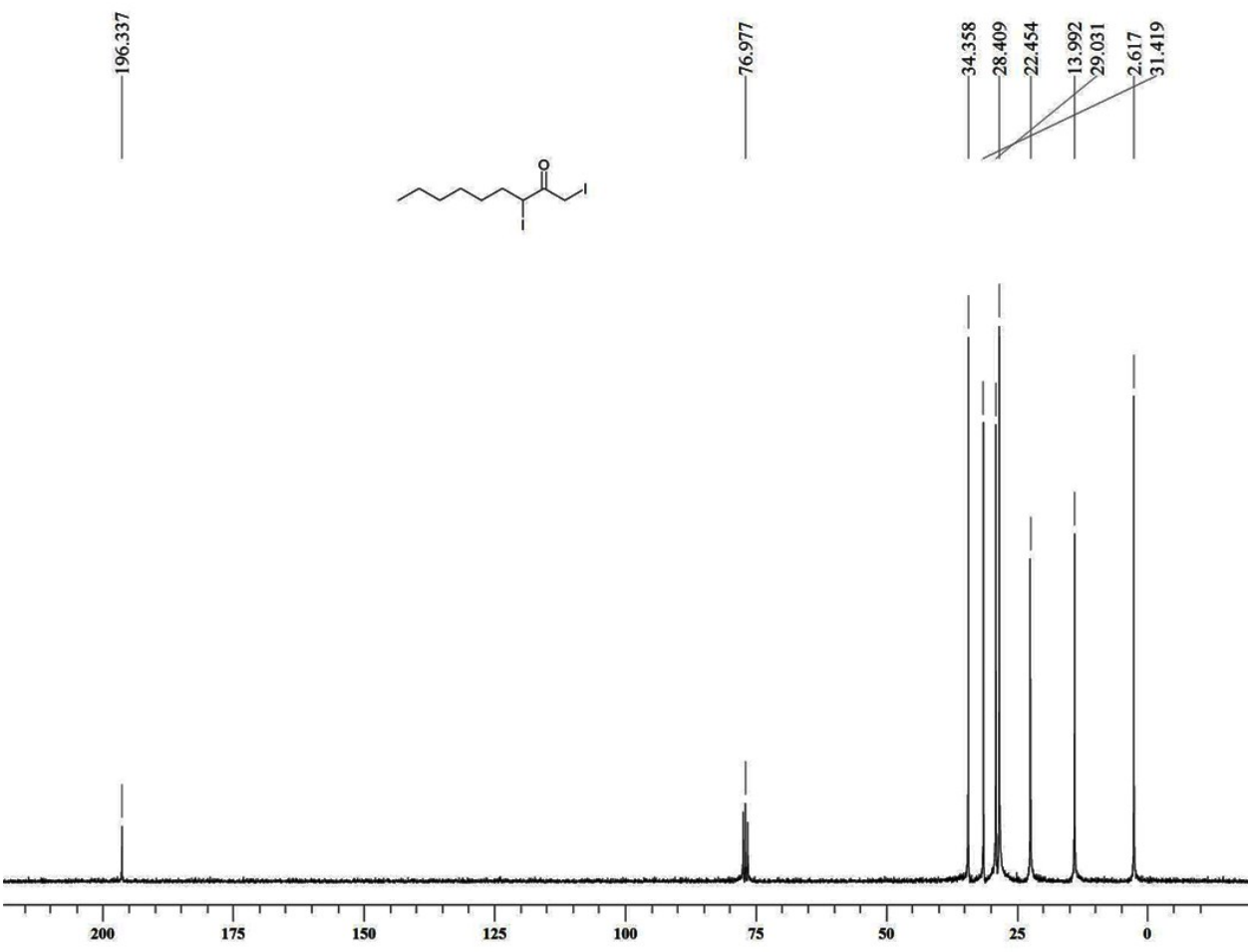
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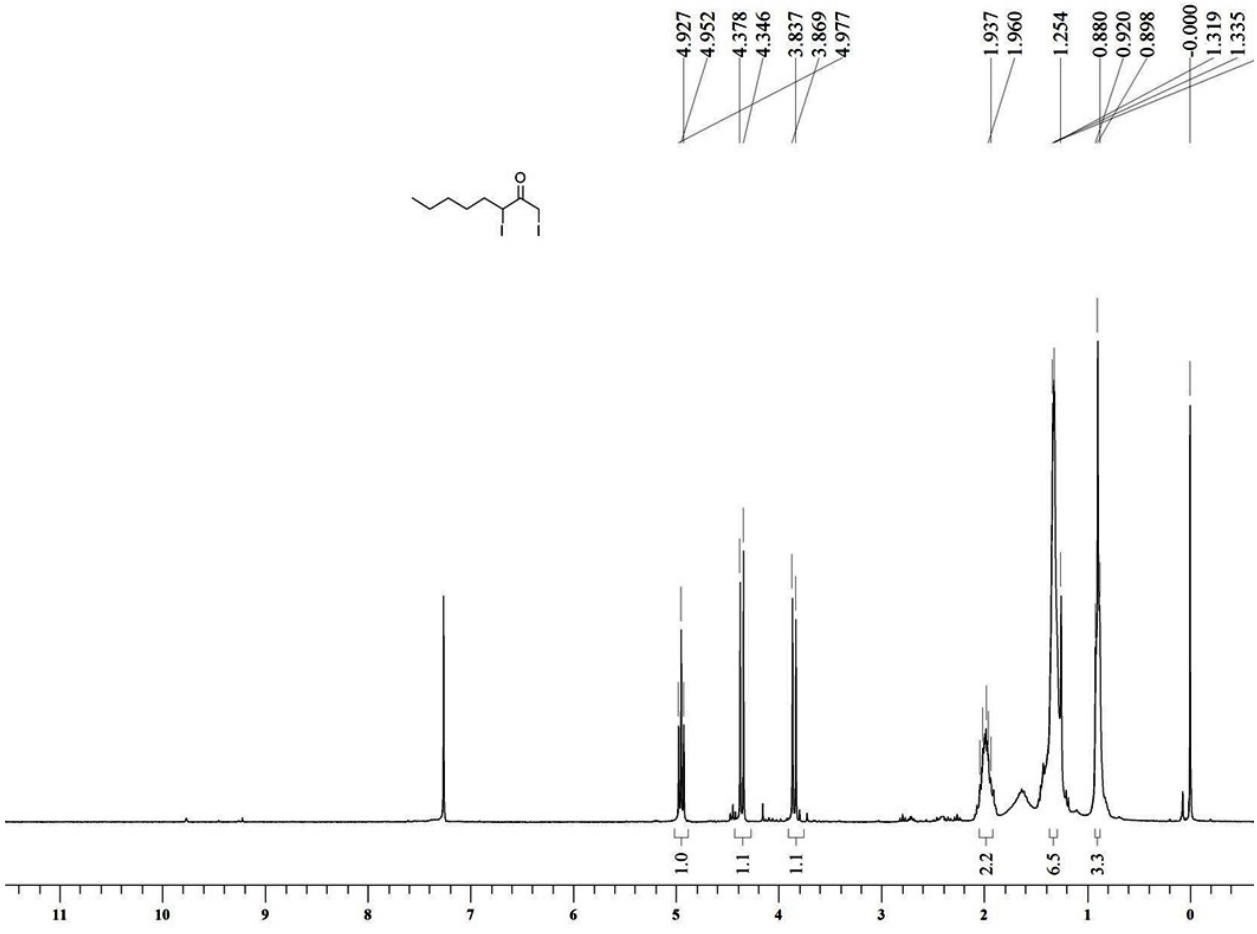
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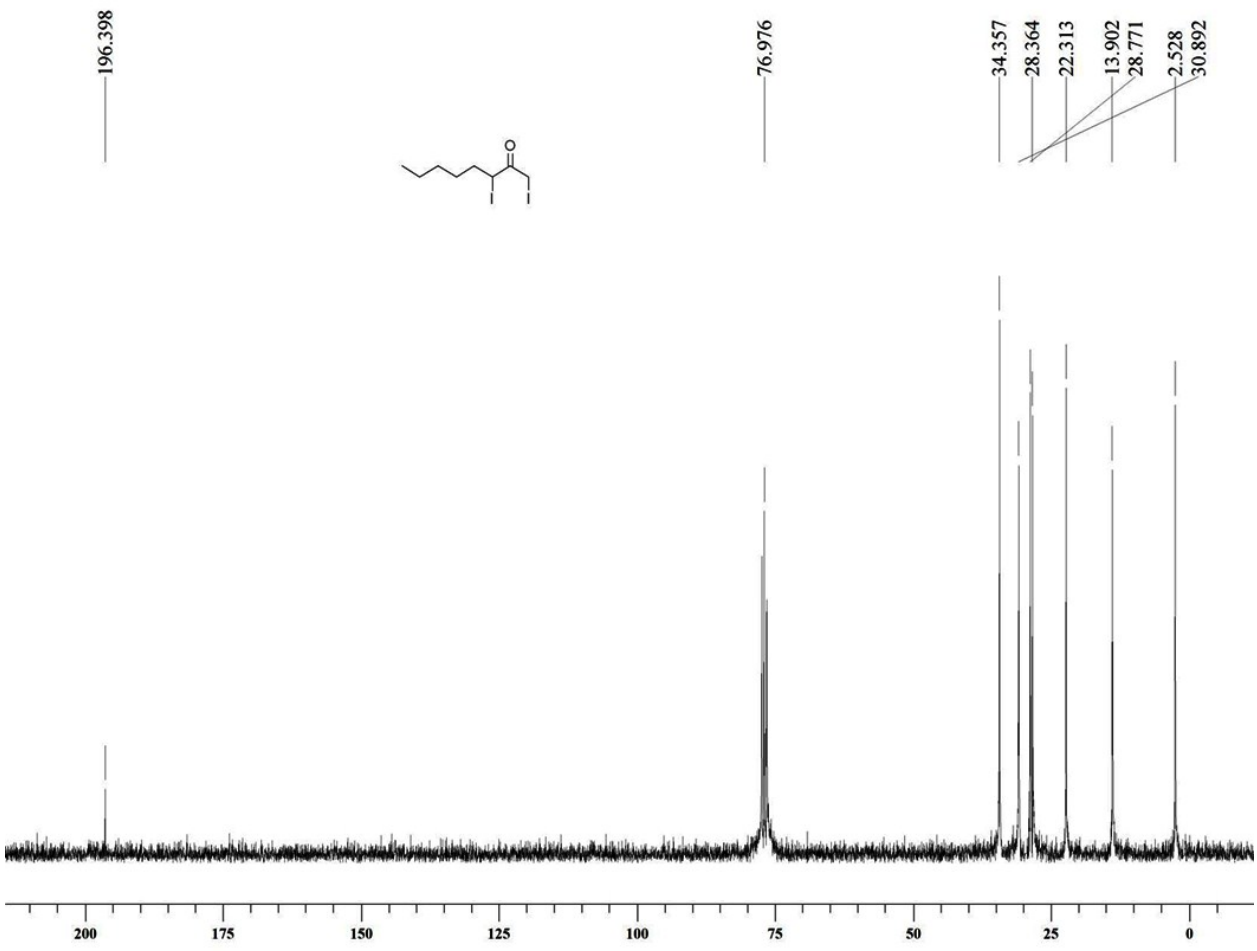
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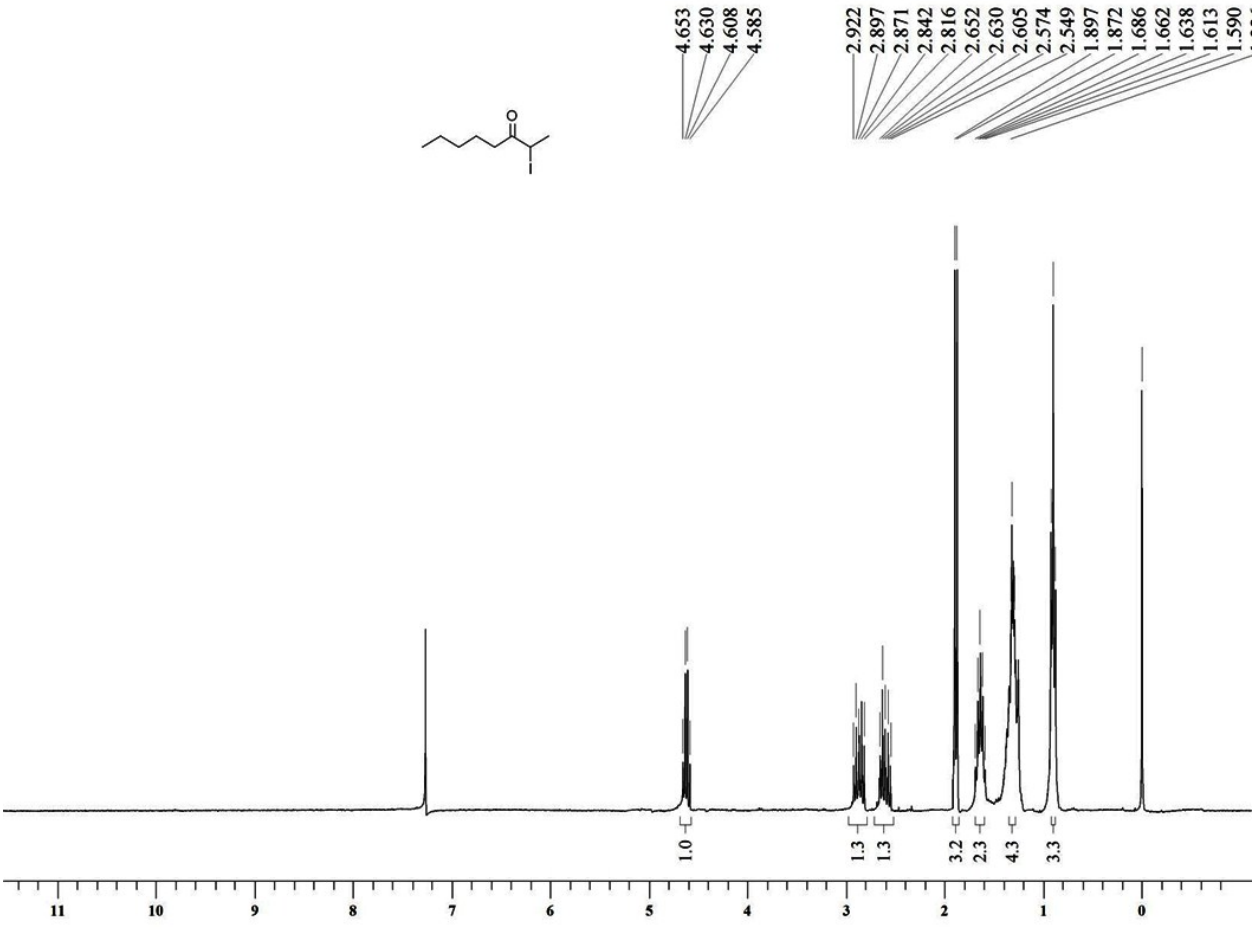
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1.686
1.662
1.638
1.613
1.590
O
I
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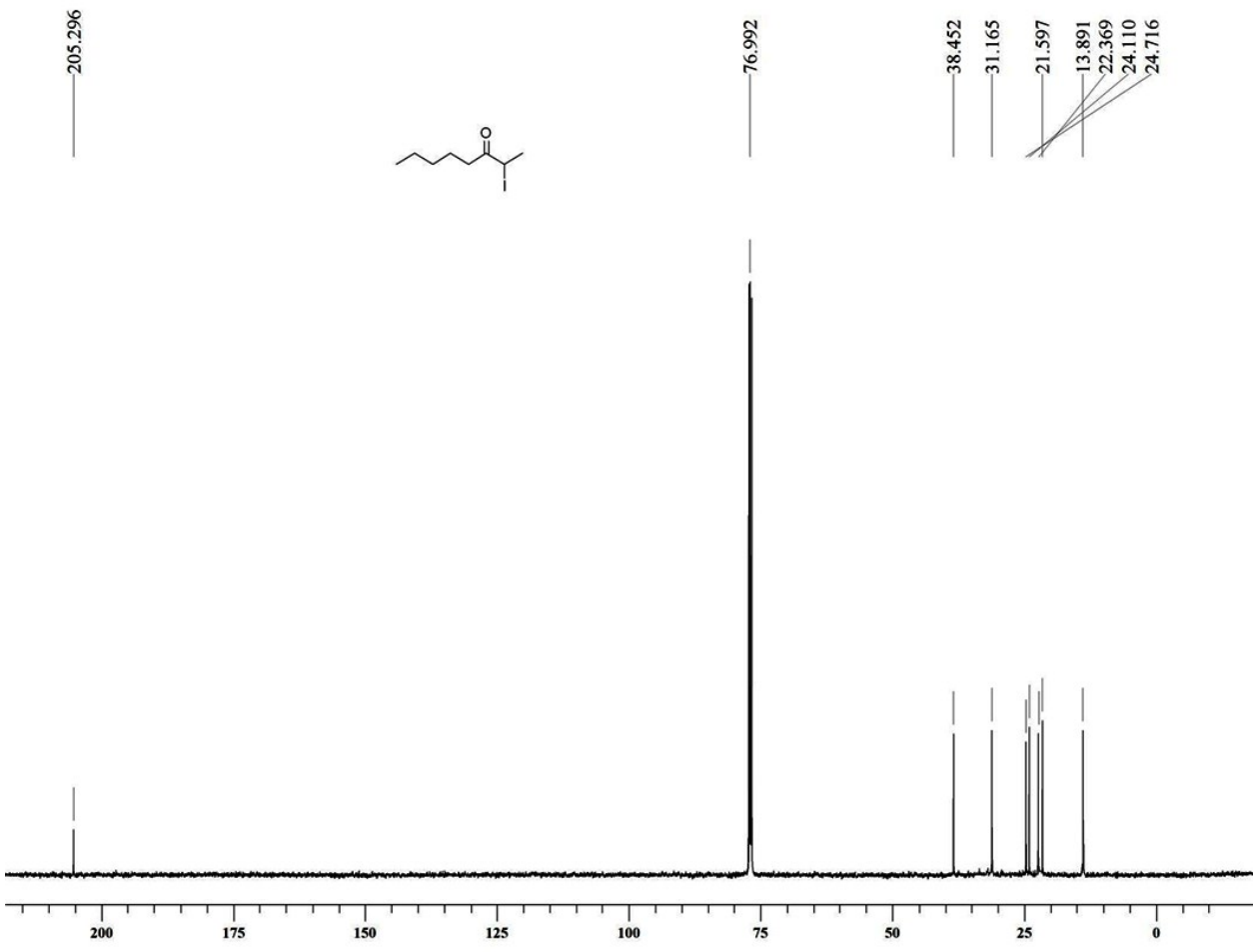
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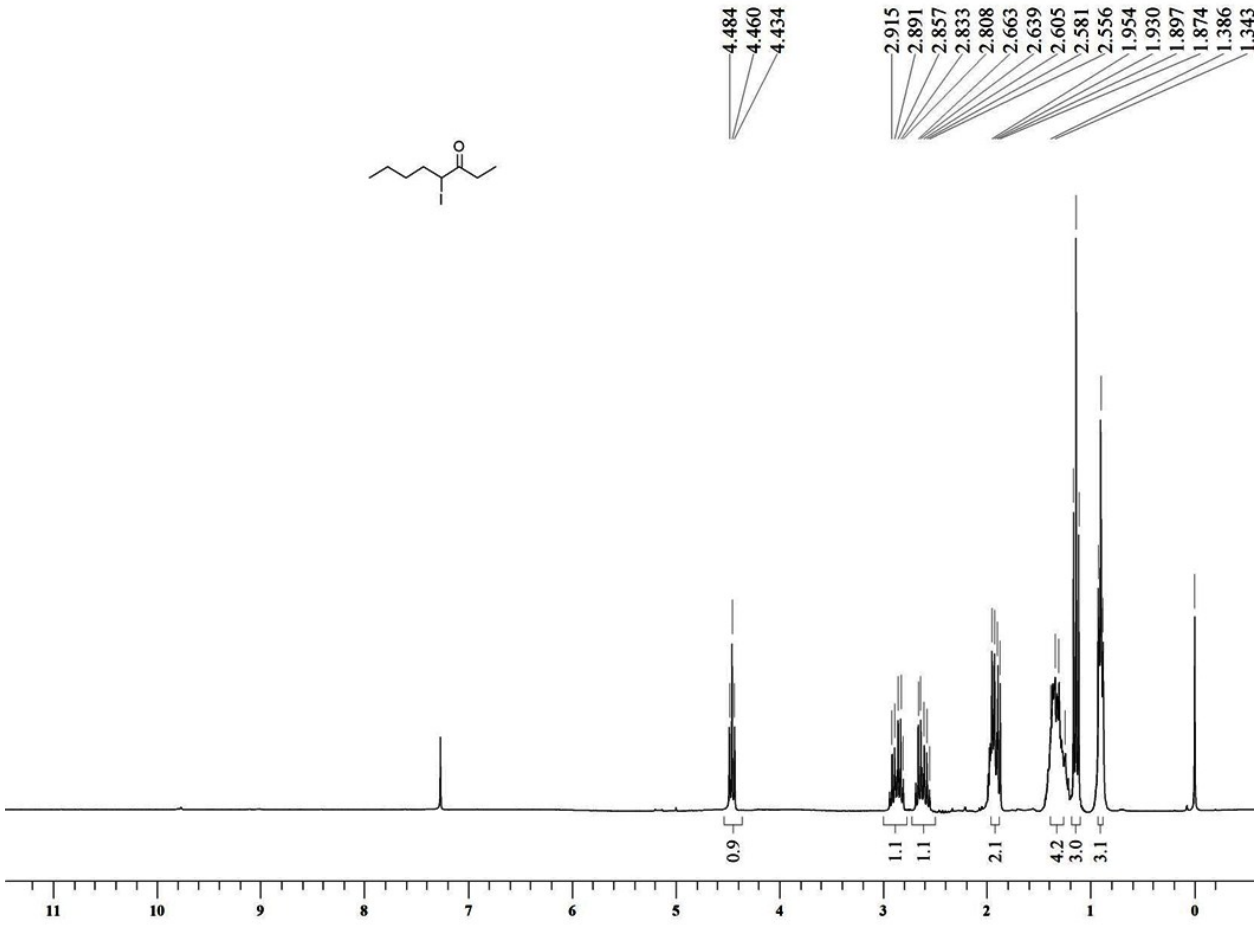
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2.605
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2.556
1.954
1.930
1.897
1.874
1.386
1.343
O
I
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1.1
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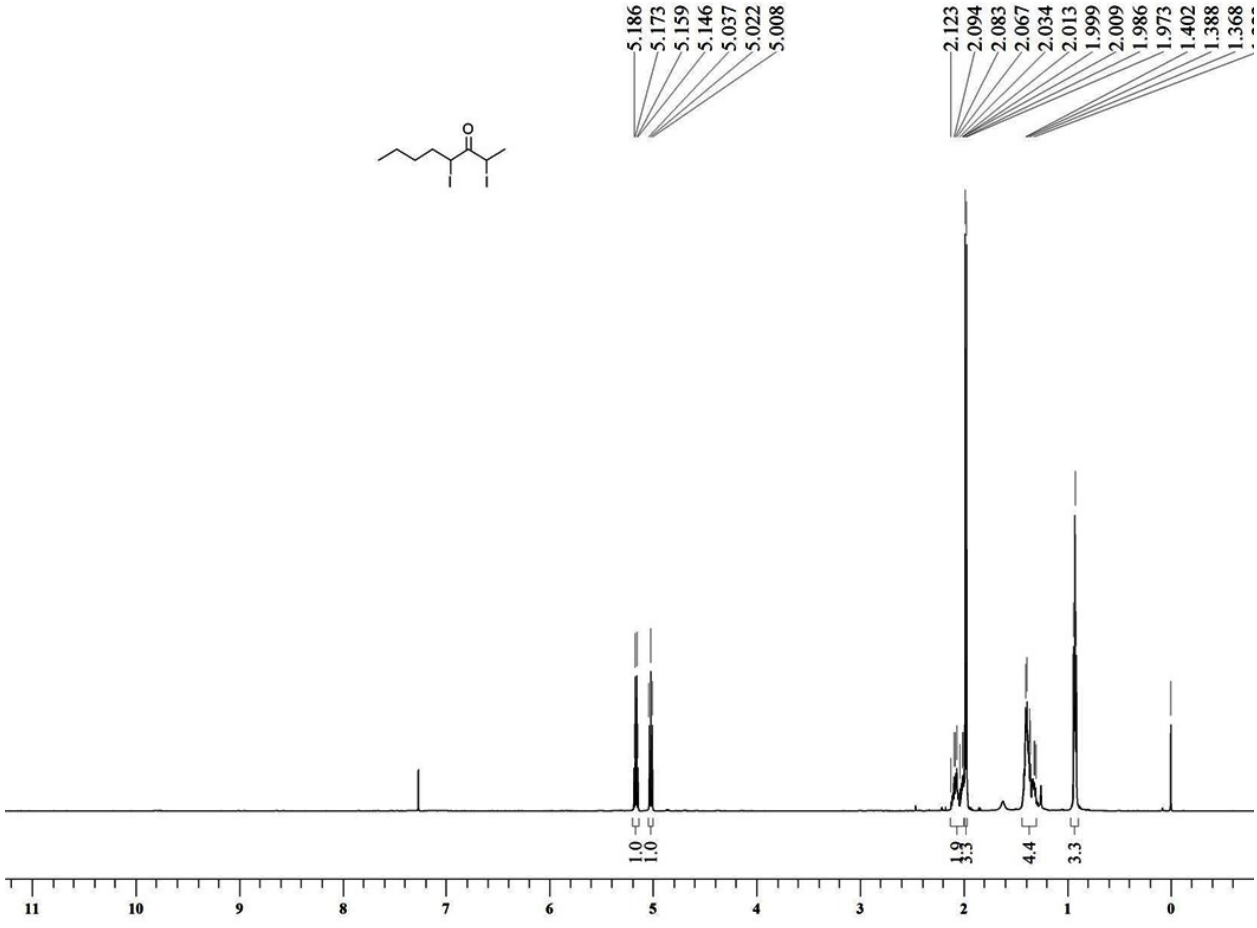
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2.009
1.986
1.973
1.402
1.388
1.368
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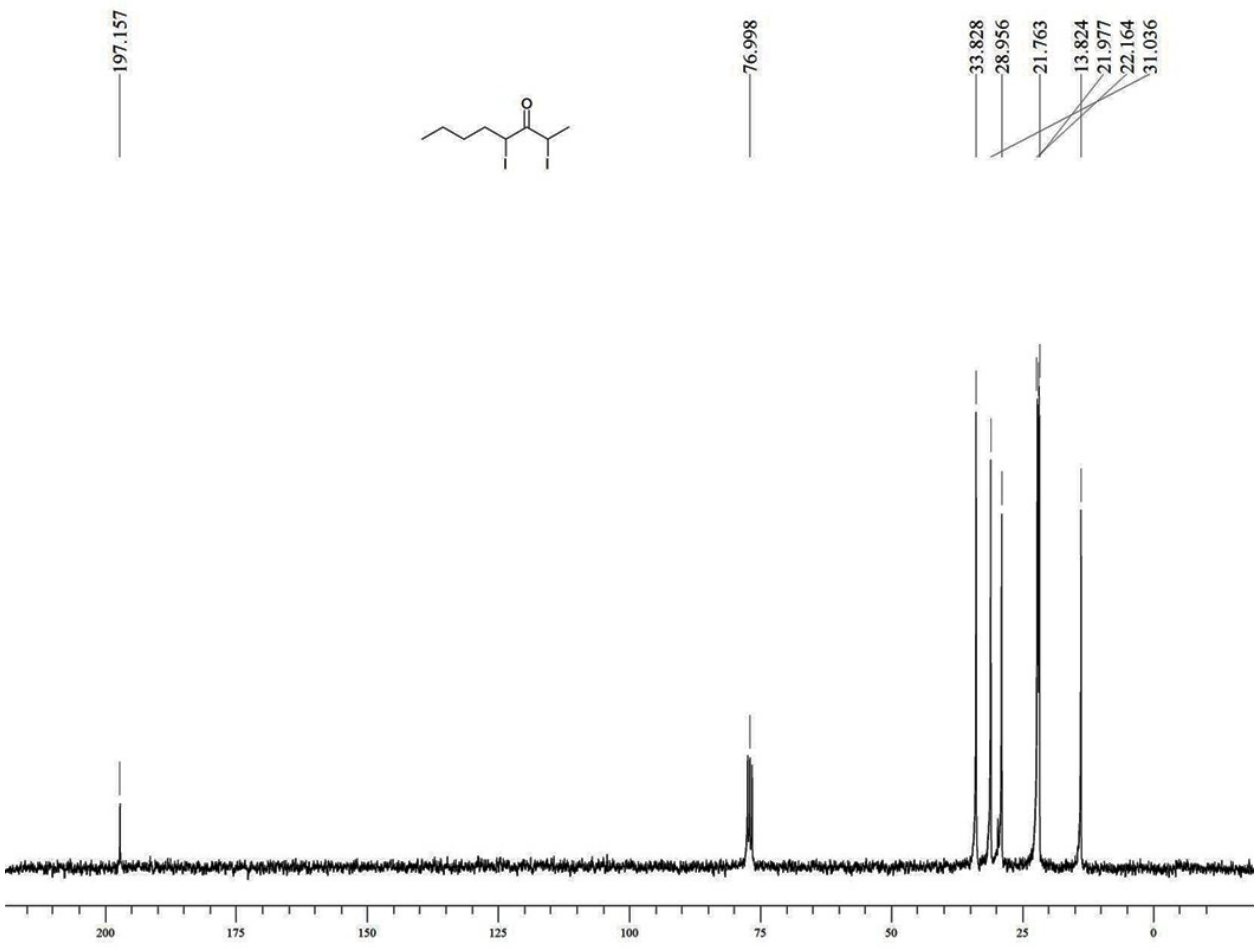
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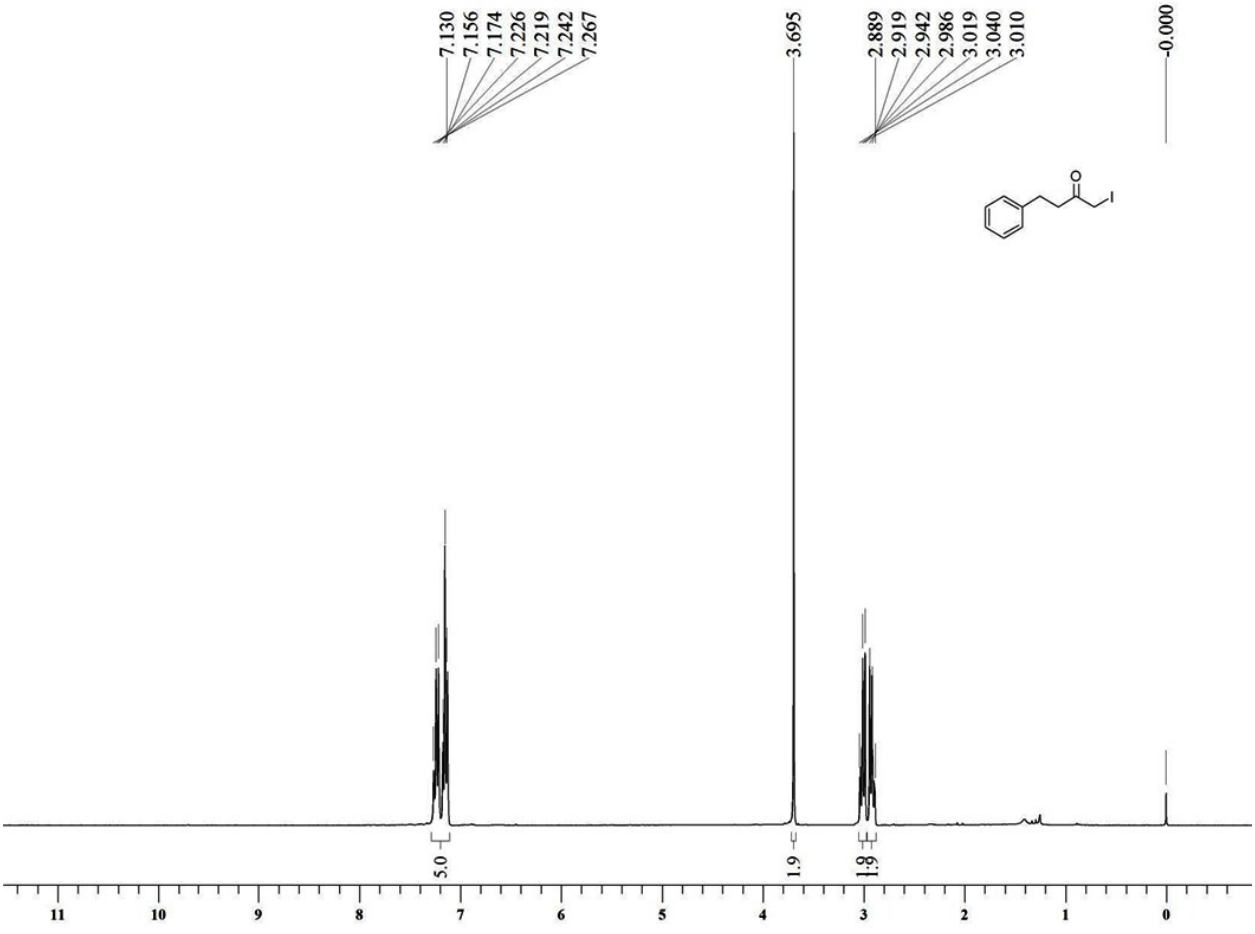
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3.019
3.040
3.010
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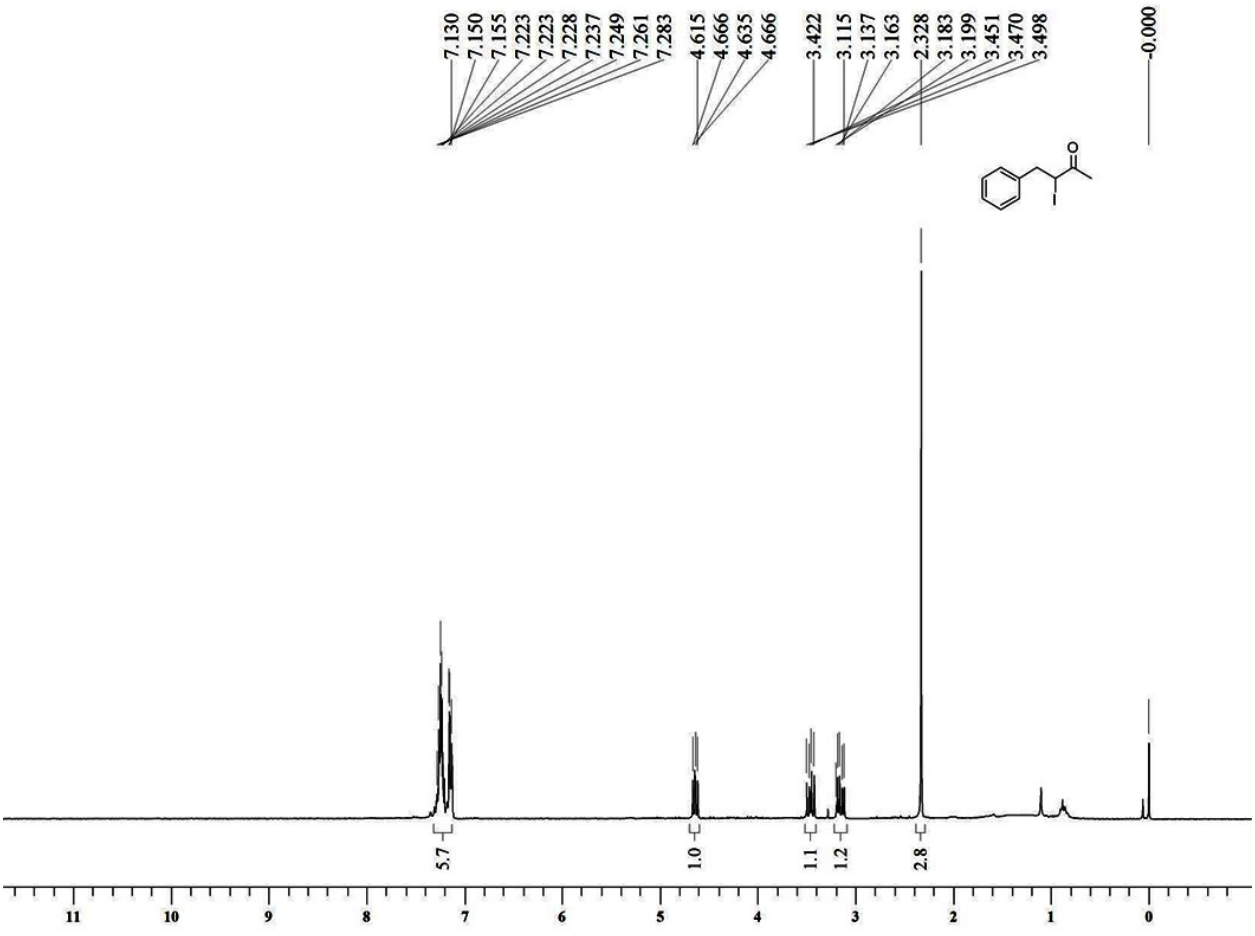
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4.666
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1.2
2.8
11
10
9
8
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6
5
4
3
2
1
0

References:

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