Electronic Supplementary Material (ESI) for Green Chemistry.

Supporting Information for:

Atom Economic Amidation with Acid Anhydrides using an Electrochemical Approach

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1. **Picture of the electrochemical cell**

Figure S1. Electrochemical H-Cell used to perform reactions described in the manuscript. Reticulated Vitreous Carbon electrodes were employed as cathode and anode.

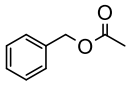
2. **Picture of the electrochemical reaction driven by 9-V battery.**

Figure S2. Electrochemical H-Cell used to perform reactions described in the manuscript. Reticulated Vitreous Carbon electrodes were employed as cathode and anode. Electrolysis is carried out by connecting the electrodes to a commercially available battery.

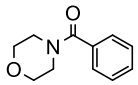
3. List of ^{1}H NMR and ^{13}C NMR peaks of products

Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet).

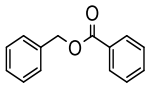
colorless oil, 72 mg, ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.03 (s, 3H), 3.39 (t, 2H, *J* = 4.88 Hz), 3.54 (t, 2H, *J* = 4.86 Hz), 3.61 (q, 4H, *J* = 5.17 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.1, 41.7, 46.7, 66.6, 66.8, 169.3.



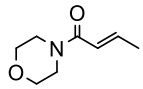
colorless oil, 77 mg, ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.102 (s, 3H,), 5.103 (s, 2H), 7.355 (m, 5H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 21, 66, 128.2, 128.5



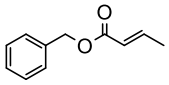
colorless oil, 100 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 3.63 (broad singlet, 8H), 7.43 (m, 5H). ^{13}C NMR (D-acetone, 100 MHz) δ 66.5, 127.2, 128.3, 129.4, 136.2, 169.4.



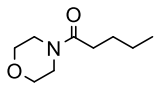
colorless oil, 114 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 5.38 (s, 2H), 7.39 (3H, *J* = 6.09 Hz), 7.52 (t, 4H, *J* = 7.60 Hz), 7.63 (m, 1H, *J* = 7.40 Hz), 8.06 (m, 2H, *J* = 7.12 Hz). ^{13}C NMR (D-acetone, 100 MHz) δ 66.2, 128.1, 128.5, 128.6, 129.4, 130.3, 133.1, 136.6, 165.7.



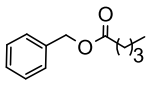
colorless oil, 79 mg, ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.87 (d, 3H, *J* = 2.85 Hz), 3.66 (broad multiplet, 8H, *J* = 7.60 Hz), 6.21 (d, 1H, *J* = 5.55 Hz), 6.88 (m, 1H, *J* = 7.11 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 18.3, 42.2, 46.1, 66.8, 121, 142.2, 165.7.



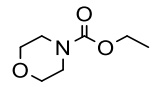
colorless oil, 83 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 1.97 (dd, 3H, *J* = 2.88 Hz), 5.25 (s, 2H), 6.02 (dd, 1H, *J* = 5.76 Hz), 7.12 (m, 1H, *J* = 5.62 Hz), 7.48 (m, 5H, *J* = 4.48 Hz). ^{13}C NMR (D-acetone, 100 MHz) δ 17.7, 40.1, 66.1, 117.9, 122.6, 128.6, 129.1, 137.3, 146.0, 166.4



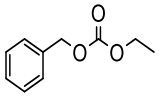
yellow oil, 86 mg, ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.88 (t, 3H, *J* = 7.34 Hz), 1.28 (m, 2H, *J* = 5.62 Hz), 1.55 (m, 2H, *J* = 5.10 Hz), 2.24 (t, 2H, *J* = 7.70 Hz), 3.42 (m, 2H, *J* = 5.00 Hz), 3.56 (m, 2H, *J* = 5.16 Hz), 3.61 (m, 4H, *J* = 4.12 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 13.8, 22.5, 27.3, 32.8, 41.8, 46.0, 66.6, 66.9, 171.9



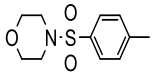
white solid, 106 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 1.00 (t, 3H, *J* = 7.36 Hz), 1.44 (m, 2H, *J* = 5.62 Hz), 1.66 (m, 2H, *J* = 4.30 Hz), 2.45 (t, 2H, *J* = 7.48 Hz), 5.19 (s, 2H), 7.47 (bs, 5H). ^{13}C NMR (D-acetone, 100 MHz) δ 13.6, 22.5, 27.4, 34.1, 66.1, 117.9, 128.5, 129.1, 137.3, 173.8



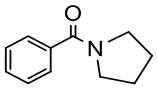
yellow oil, 87 mg, ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.26 (t, 3H, *J* = 7.12 Hz), 3.46 (m, 4H, *J* = 4.88 Hz), 3.64 (m, 4H, *J* = 4.46 Hz), 4.13 (q, 2H, , *J* = 7.12 Hz). ^{13}C NMR (D-acetone, 100 MHz) δ 14.6, 20.3, 61.5, 66.6, 126.9, 127, 128, 155.6



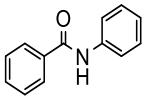
white solid, 92 mg, ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.30 (t, 3H, J = 7.14 Hz), 4.20 (q, 2H, J = 7.15 Hz), 5.15 (s, 2H), 7.36 (m, 5H, J = 2.28 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 14.3, 64.1, 69.4, 128.3, 128.6, 135.3



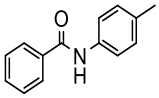
yellow solid, 137 mg, ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.45 (s, 3H), 2.91 (m, 4H, J = 4.72 Hz), 3.67 (m, 4H, J = 4.74 Hz), 7.47 (d, 2H, J = 8.04 Hz), 7.66 (d, 2H, J = 8.24 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 12.9, 20.6, 46.2, 58.4, 65.7, 126.9, 129.5, 132.4, 143.9



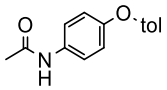
brown oil, 109 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 1.89 (m, 4H, J = 7.93 Hz), 3.44 (t, 2H), 3.54 (t, 2H), 7.44 (m, 3H), 7.54 (m, 2H, J = 7.72 Hz). ^{13}C NMR (D-acetone, 100 MHz) δ 24.1, 26.2, 45.9, 49.1, 127.1, 128.1, 129.5, 137.8, 168.6



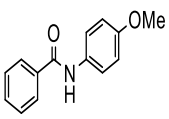
white solid, 110 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 7.10 (t, 1H, J = 7.4 Hz), 7.35 (t, 2H, J = 7.98 Hz), 7.51 (m, 3H, J = 6.76 Hz), 7.56 (d, 2H, J = 7.6 Hz), 7.98 (m, 2H, J = 4.3 Hz), 9.52 (s,1H). ^{13}C NMR (D-acetone, 100 MHz) δ 120.1, 123.6, 127.4, 128.4, 131.4, 139.5



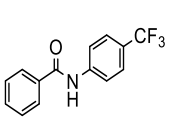
orange solid, 122 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 2.29 (s, 3H), 7.15 (d, 2H, J = 8.24 Hz), 7.52 (m, 3H, J = 8.8 Hz), 7.72 (d, 2H, J = 8.4 Hz), 7.98 (d, 2H, J = 7.28 Hz), 9.46 (s,1H). ^{13}C NMR (D-acetone, 100 MHz) δ 19.9, 120.2, 127.4, 128.4, 129.1, 131.3, 132.9, 135.5, 136.9, 165.2



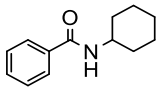
yellowish white solid, 125 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 2.08 (s, 3H), 2.23 (s, 3H), 6.86 (m, 3H, J = 4.12 Hz), 7.06 (t, 1H, J = 7.96 Hz), 7.17 (t, 1H, J = 7.72 Hz), 7.28 (d,1H, J = 4.06 Hz), 7.64 (d,2H, J = 9.0 Hz), 9.18 (s,1H). ^{13}C NMR (D-acetone, 100 MHz) δ 15.4, 23.3, 117.7, 118.8, 120.6, 121, 123.7, 127.2, 129.2, 131.4, 134.8, 153.3, 155.1, 167.7



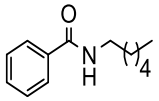
white solid, 126 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 3.66 (s, 3H), 6.78 (d, 2H, J = 3.02 Hz), 7.41 (m, 3H, J = 7.21 Hz), 7.62 (d, 2H, J = 9.04 Hz), 7.86 (d, 2H, J = 7.11 Hz), 9.28 (s,1H). ^{13}C NMR (D-acetone, 100 MHz) δ 54.75, 113.7, 121.7, 127.3, 128.3, 131.3, 132.6, 135.6, 156.2, 165.0



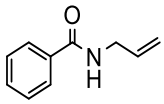
white solid, 123 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 7.53 (t, 2H, J = 7.44 Hz), 7.60 (d, 1H, J = 7.34 Hz), 7.72 (d, 2H, J = 8.64 Hz), 8.02 (d, 2H, J = 7.32 Hz), 8.10 (d, 2H, J = 8.40 Hz), 9.86 (s,1H). ^{13}C NMR (D-acetone, 100 MHz) δ 119.8, 119.9, 125.8, 127.6, 128.5, 131.8, 134.9, 142.9, 165.9



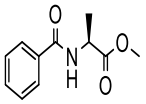
white solid, 120 mg, ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.14 (m, 3H, J = 8.42 Hz), 1.31 (quartet of triplets, 2H, J = 12.43 Hz), 1.56 (doublet of triplets, 1H, J = 12.96 Hz), 1.66 (doublet of triplets, 2H, J = 13.63 Hz), 1.93 (doublet of doublets, 2H, J = 12.30 Hz), 3.88 (m, 1H, J = 4.05 Hz), 6.15(broad doublet, 1H, J = 6.48 Hz), 7.31 (tt, 2H, J = 6.70 Hz), 7.38 (tt, 1H, J = 6.69 Hz), 7.68 (m, 2H, J = 7.05 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 24.9, 25.6, 30.9, 33.2, 77.1, 126.9, 128.4, 131.2, 135.1, 166.7



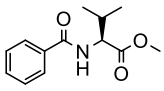
colorless oil, 118 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 0.89 (tr, 3H, J = 6.85 Hz), 1.33 (m, 6H, J = 6.63 Hz), 1.62 (quint, 2H, J = 7.26 Hz), 3.40 (quart, 2H J = 7.34 Hz), 7.44 (m, 2H, J = 7.34 Hz), 7.52 (m, 1H, J = 7.39 Hz), 7.84 (s, 1H), 7.92 (m, 2H, J = 7.08 Hz). ^{13}C NMR (D-acetone, 100 MHz) δ 13.5, 22.4, 26.6, 31.5, 39.6, 127.1, 128.2, 130.9, 135.3, 166.4



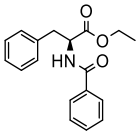
brown oil, 92 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 4.02 (tt, 2H, *J* = 5.62 Hz), 5.21 (dq, 2H, *J* = 17.19 Hz), 5.95 (dq, 1H, *J* = 27.46 Hz), 7.46 (t, 2H, *J* = 7.36 Hz), 7.52 (t, 1H, *J* = 7.27 Hz), 7.94 (d, 2H, *J* = 7.07 Hz). ^{13}C NMR (D-acetone, 100 MHz) δ 41.9, 114.9, 127.2, 128.5, 129.6, 131.2, 132.8, 134.9, 135.3, 166.6



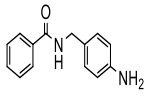
yellow oil, 114 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 1.49 (d, 3H, *J* = 7.32 Hz), 3.7 (s, 3H), 4.68 (quint, 1H, *J* = 7.33 Hz), 7.46 (t, 2H, *J* = 7.45 Hz), 7.53 (m, 1H, *J* = 7.33 Hz), 7.95 (d, 2H, *J* = 7.31 Hz), 8.06 (m, 1H, *J* = 4.22 Hz). ^{13}C NMR (D-acetone, 100 MHz) δ 16.7, 48.5, 51.5, 127.3, 128.3, 131.4, 134.3, 166.5, 173.1



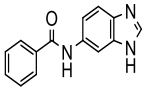
white solid, 116 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 1.03 (dd, 6H, *J* = 5.83 Hz), 2.26 (m, 1H, *J* = 6.77 Hz), 3.73 (s, 3H), 4.60 (dd, 1H, *J* = 4.96 Hz), 7.46 (t, 2H, *J* = 7.44 Hz), 7.55 (t, 1H, *J* = 7.34 Hz), 7.73 (s, 1H), 7.93 (dd, 2H, *J* = 7.16 Hz). ^{13}C NMR (D-acetone, 100 MHz) δ 17.9, 18.7, 30.6, 51.2, 58.2, 127.4, 128.3, 131.3, 134.53, 166.9, 172.2.



white solid, ^{1}H NMR (D-acetone, 400 MHz) δ 1.18 (t, 3H, *J* = 7.12 Hz), 3.21 (m, 1H, *J* = 9.31 Hz), 4.13 (quart, 2H, *J* = 4.22 Hz), 4.88 (m, 1H, *J* = 4.22 Hz), 7.2 (m, 1H, *J* = 7.08 Hz), 7.3 (m, 4H, *J* = 7.15 Hz), 7.42 (m, 2H, *J* = 7.46 Hz), 7.50 (m, 1H, *J* = 7.41 Hz), 7.83 (m, 2H, *J* = 7.04 Hz), 7.88 (m, 1H). ^{13}C NMR (D-acetone, 100 MHz) δ 13.6, 37.2, 54.4, 60.7, 126.6, 127.3, 128.1, 128.3, 131.4, 134.4, 137.5, 166.6, 171.6



white solid, 120 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 4.08 (s, 2H), 4.37 (d, 2H, *J* = 5.96 Hz), 6.57 (d, 2H, *J* = 8.40 Hz), 7.06 (d, 2H, *J* = 8.44 Hz), 7.35 (s, 1H), 7.43 (m, 2H, *J* = 7.36 Hz), 7.49 (m, 1H, *J* = 7.32 Hz), 7.77 (m, 2H, *J* = 7.08 Hz). ^{13}C NMR (D-acetone, 100 MHz) δ 43.3, 114.9, 117.9, 127.5, 128.9, 129.3, 131.7, 135.4, 147.6, 167.2



white solid, 137 mg, ^{1}H NMR (D-acetone, 400 MHz) δ 7.52 (m, 2H, *J* = 6.38 Hz), δ 7.61 (m, 2H, *J* = 7.2 Hz), 7.73 (dt, 1H, *J* = 8.93 Hz), 8.03 (d, 2H, *J* = 7.24 Hz), 8.40 (m, 1H, *J* = 2.44 Hz), 9.57 (s, 1H). ^{13}C NMR (D-acetone, 400 MHz) δ 109.9, 110.9, 121.2, 123.9, 127.4, 128.4, 131.3, 132.6, 133.7, 135.6, 165.3

4. Spectra of synthesized compounds

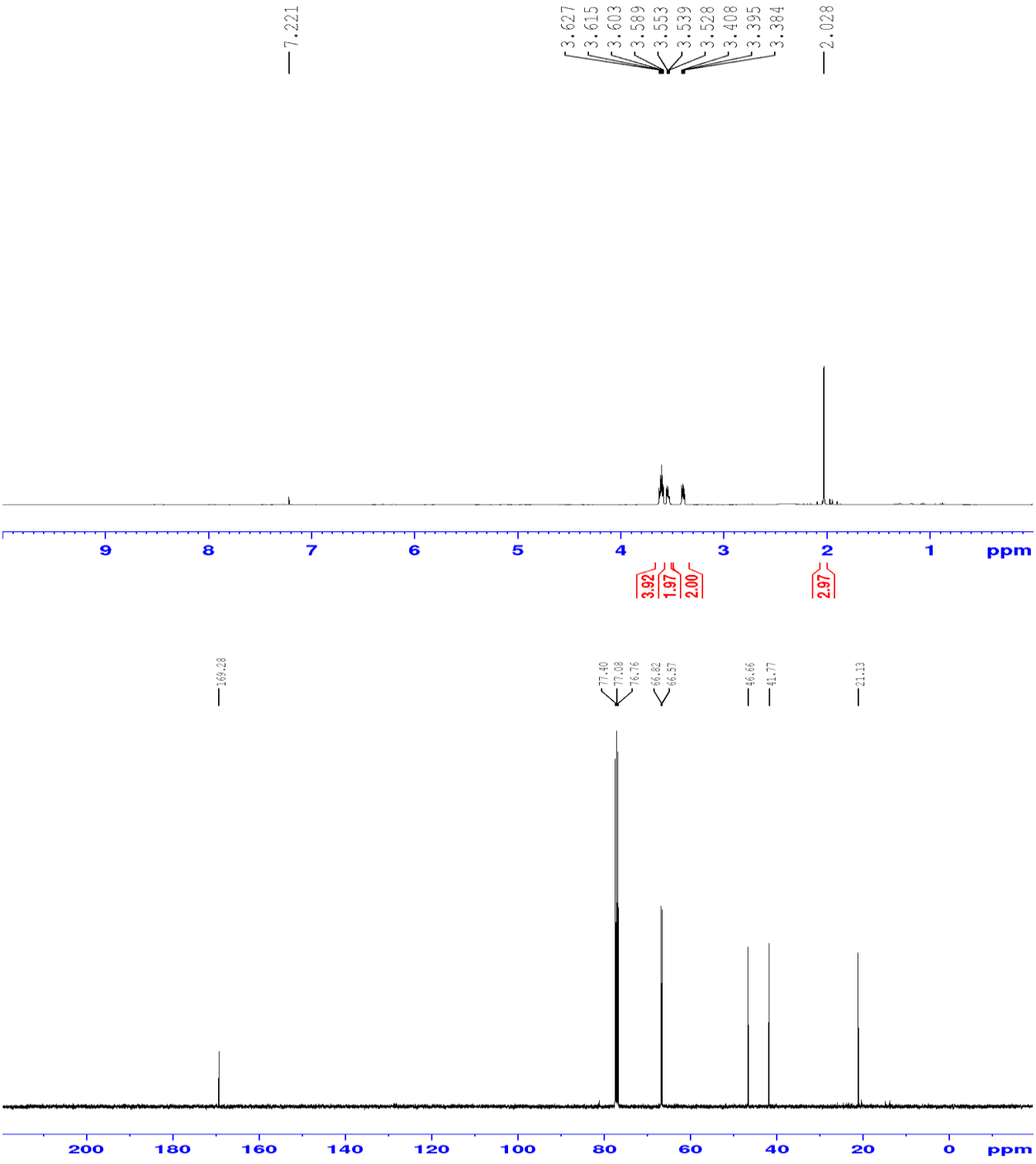


Figure S3 ^{1}H & ^{13}C spectra of product 1a in $CDCl_3$. The spectra matched reference[18]

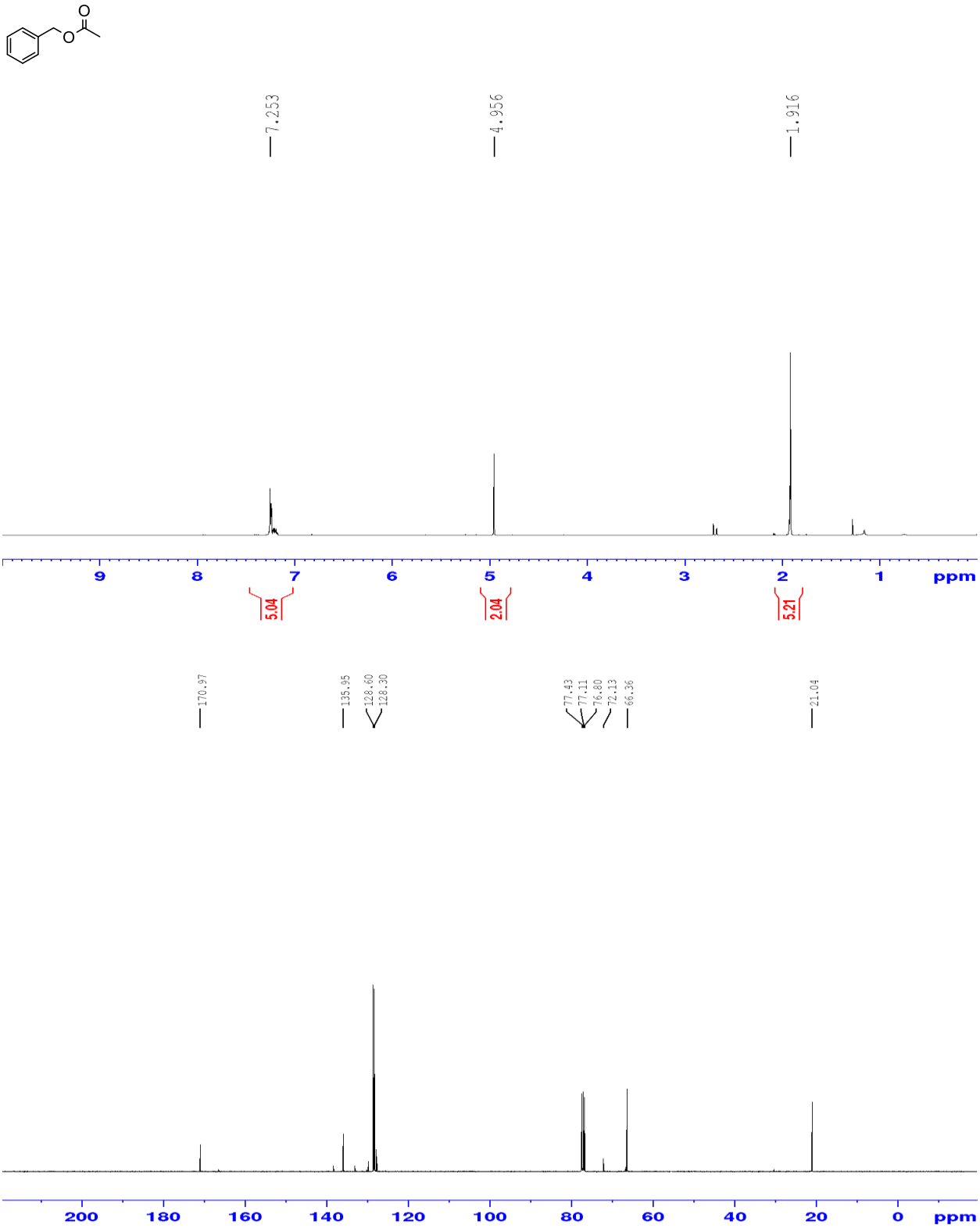


Figure S4 ^{1}H (D-acetone) and & ^{13}C ($CDCl_3$) spectra of product 1b. The peak at 2.84 ppm is water from the acetone solvent. The spectra matched reference[5]

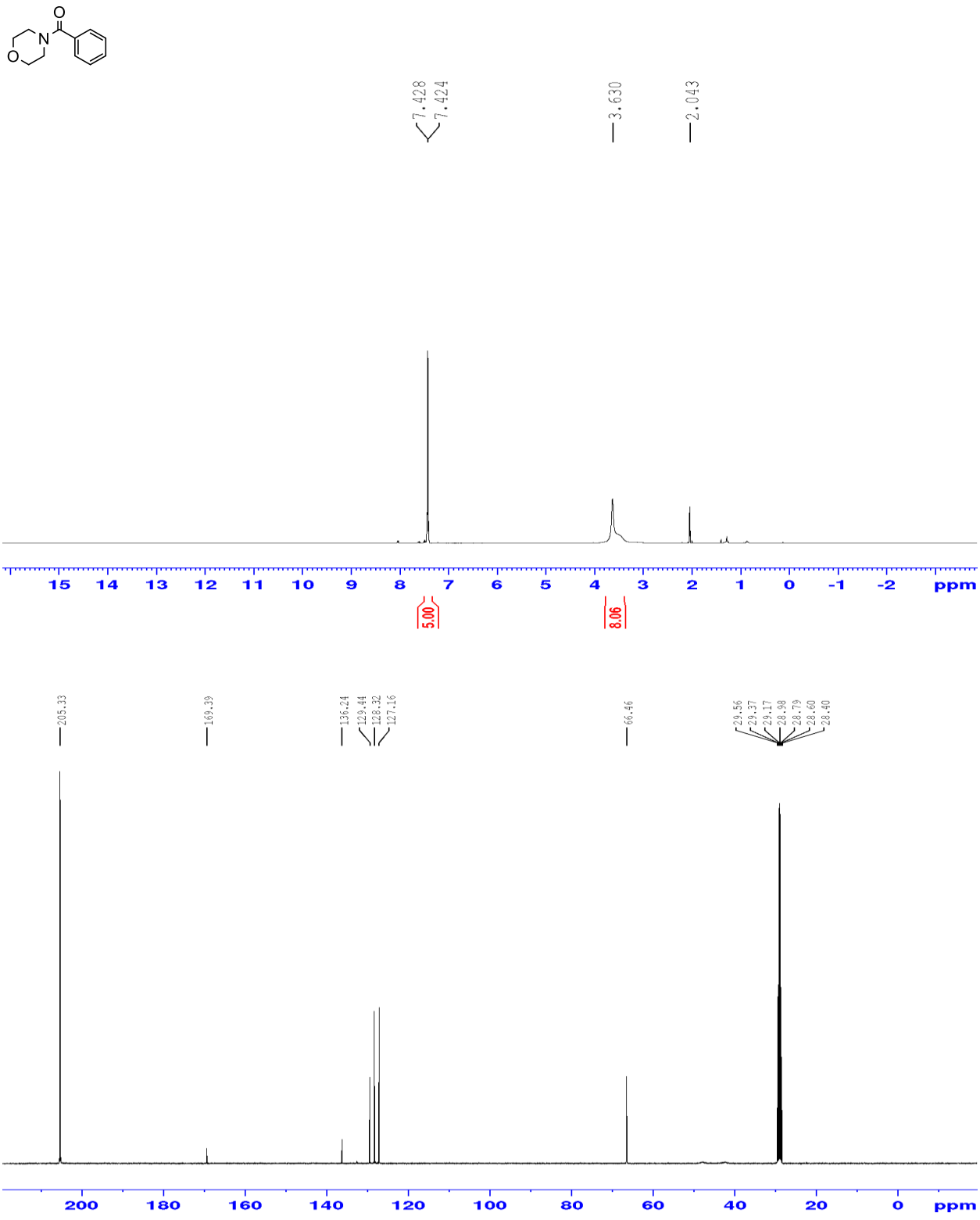


Figure S5 ^{1}H & ^{13}C spectra of product 2a in D-acetone. The spectra matched reference[12]

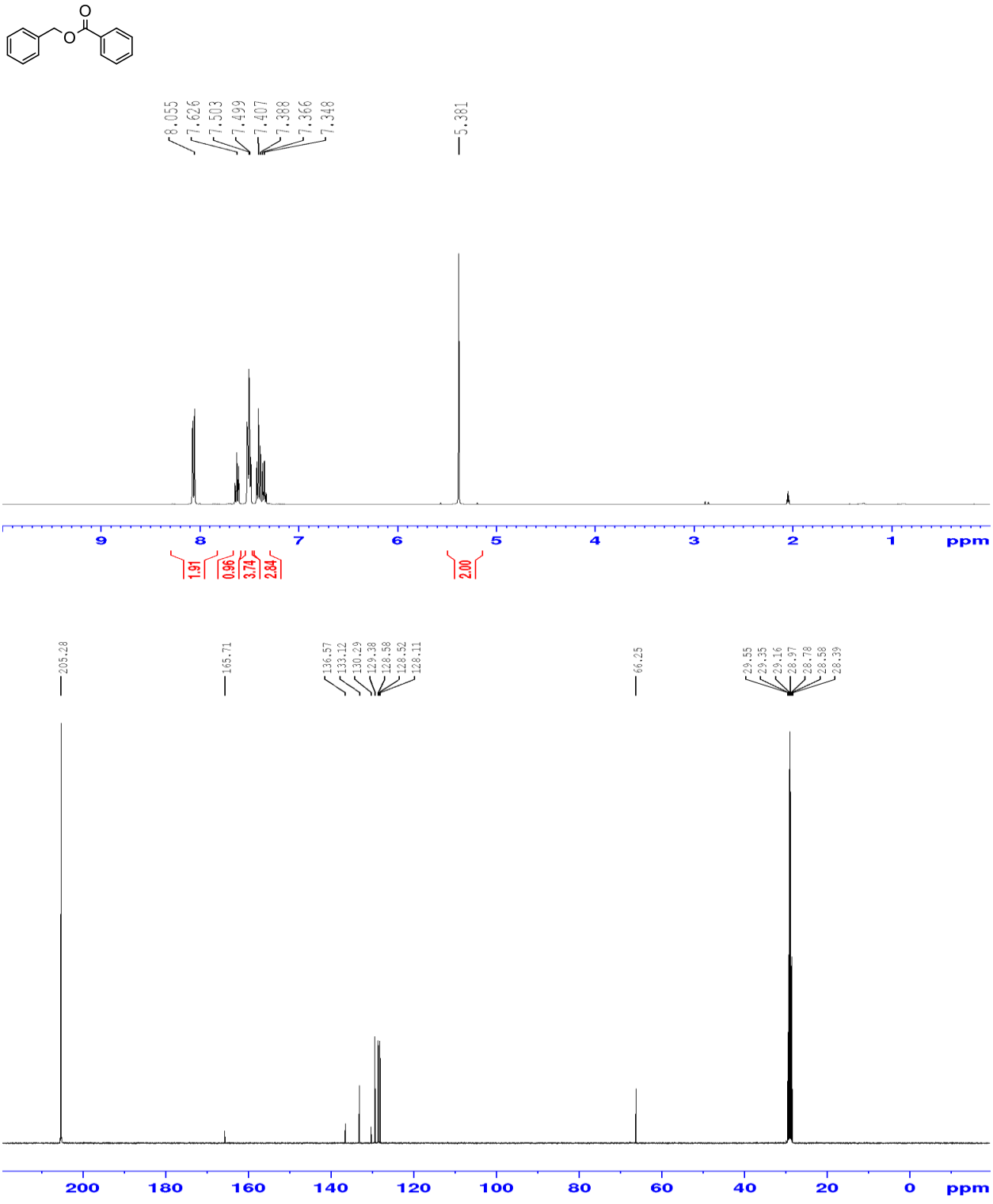


Figure S6 ^{1}H & ^{13}C spectra of product 2b in D-acetone. The spectra matched reference[16]

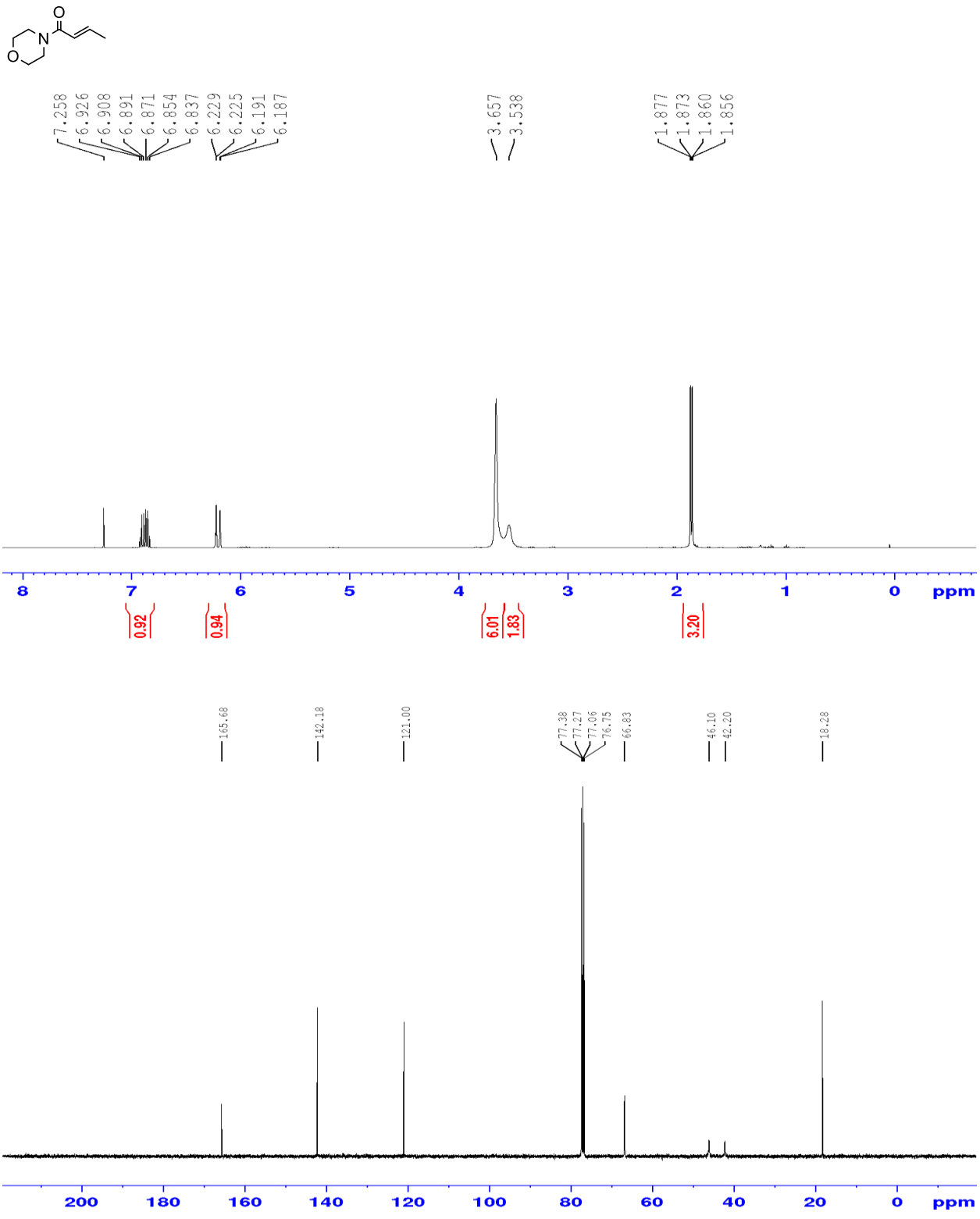


Figure S7 ^{1}H & ^{13}C spectra of product 3a in $CDCl_3$. The spectra matched reference[21]

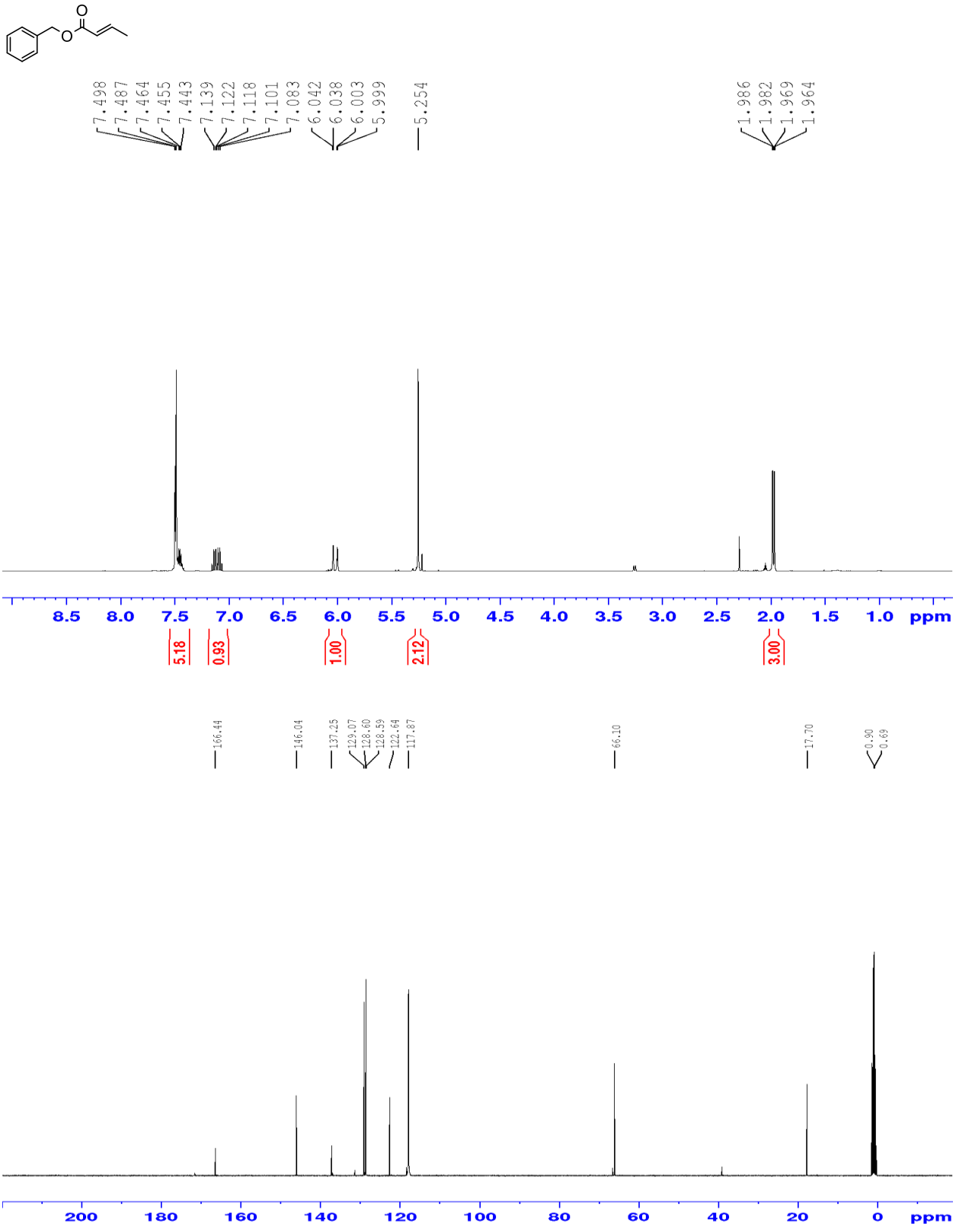


Figure S8 [1] H & [13] C spectra of product 3b in in D-acetone. The spectra matched reference[17]

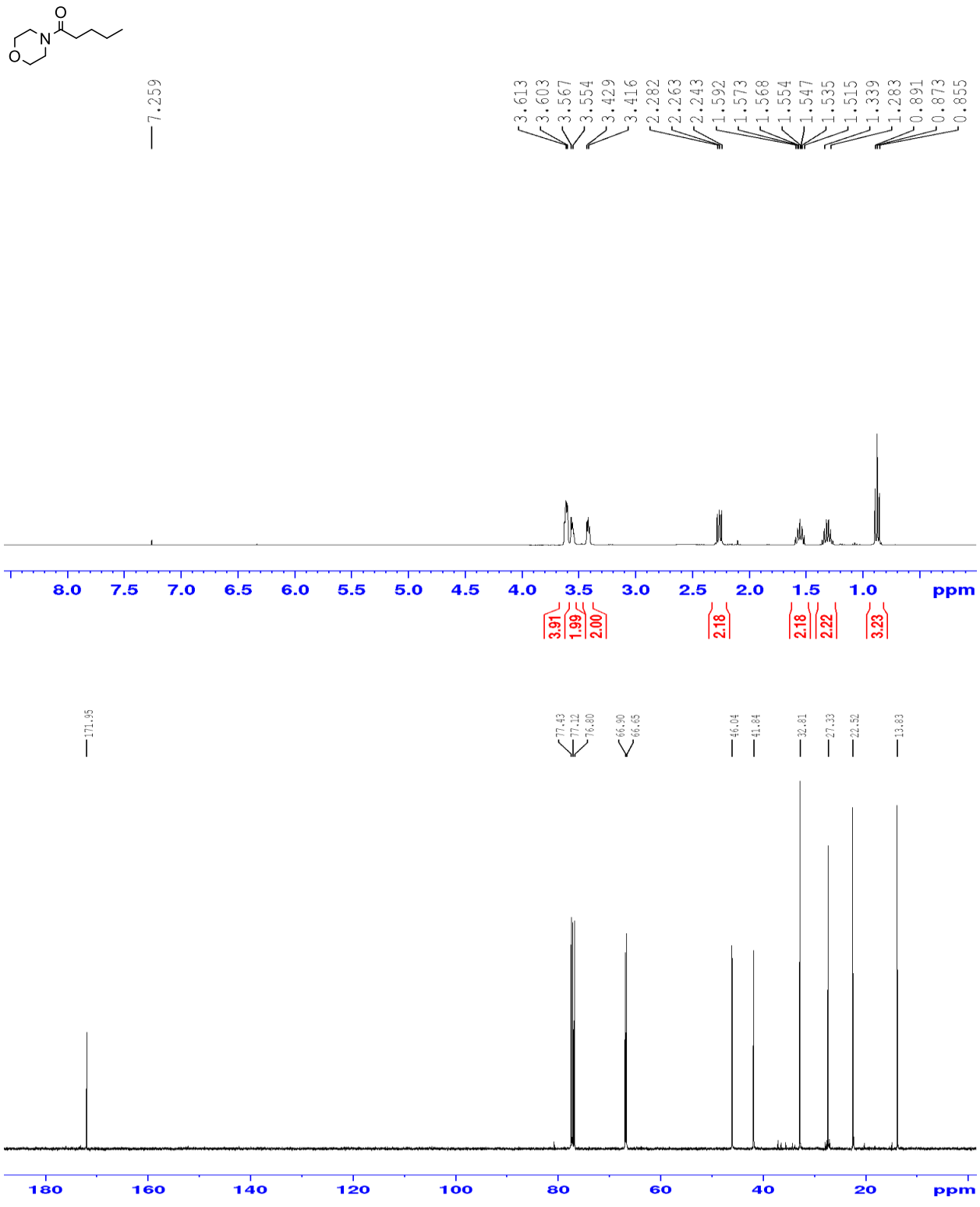


Figure S9 ^{1}H & ^{13}C spectra of product 4a in $CDCl_3$. The spectra matched reference[11]

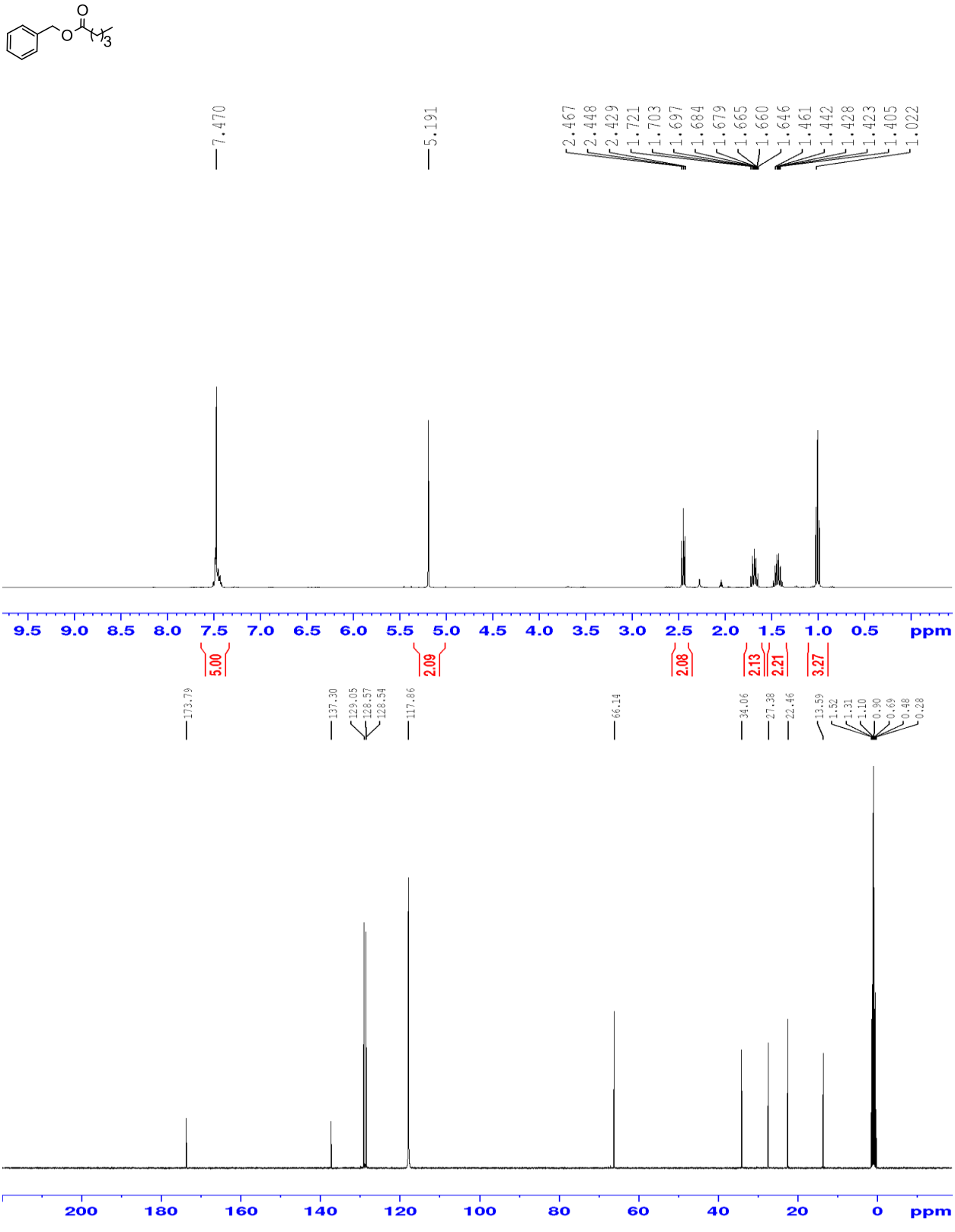


Figure S10 ^{1}H & ^{13}C spectra of product 4b in in D-acetone. The spectra matched reference[8]

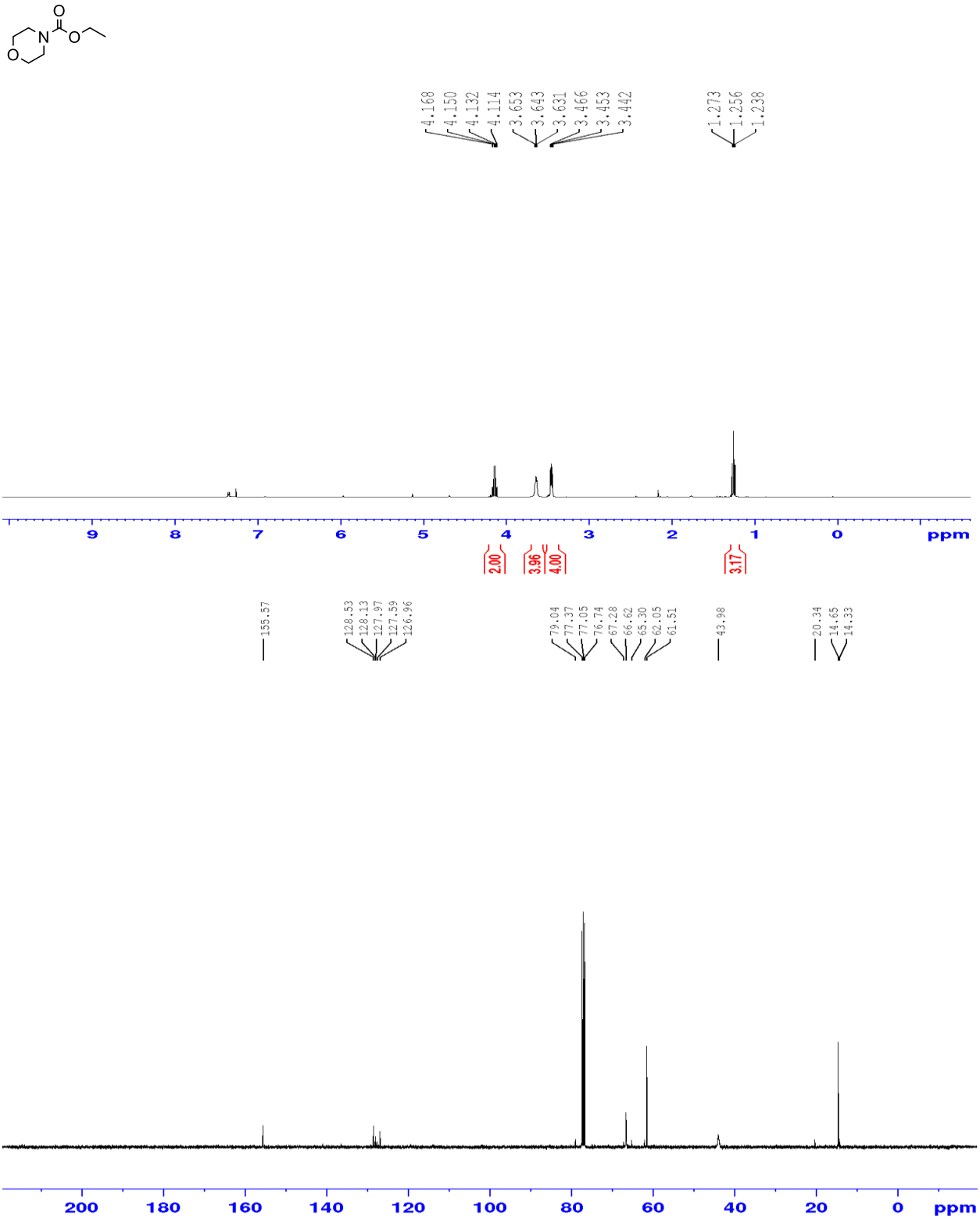


Figure S11 ^{1}H & ^{13}C spectra of product 5a in $CDCl_3$. The spectra matched reference[4]

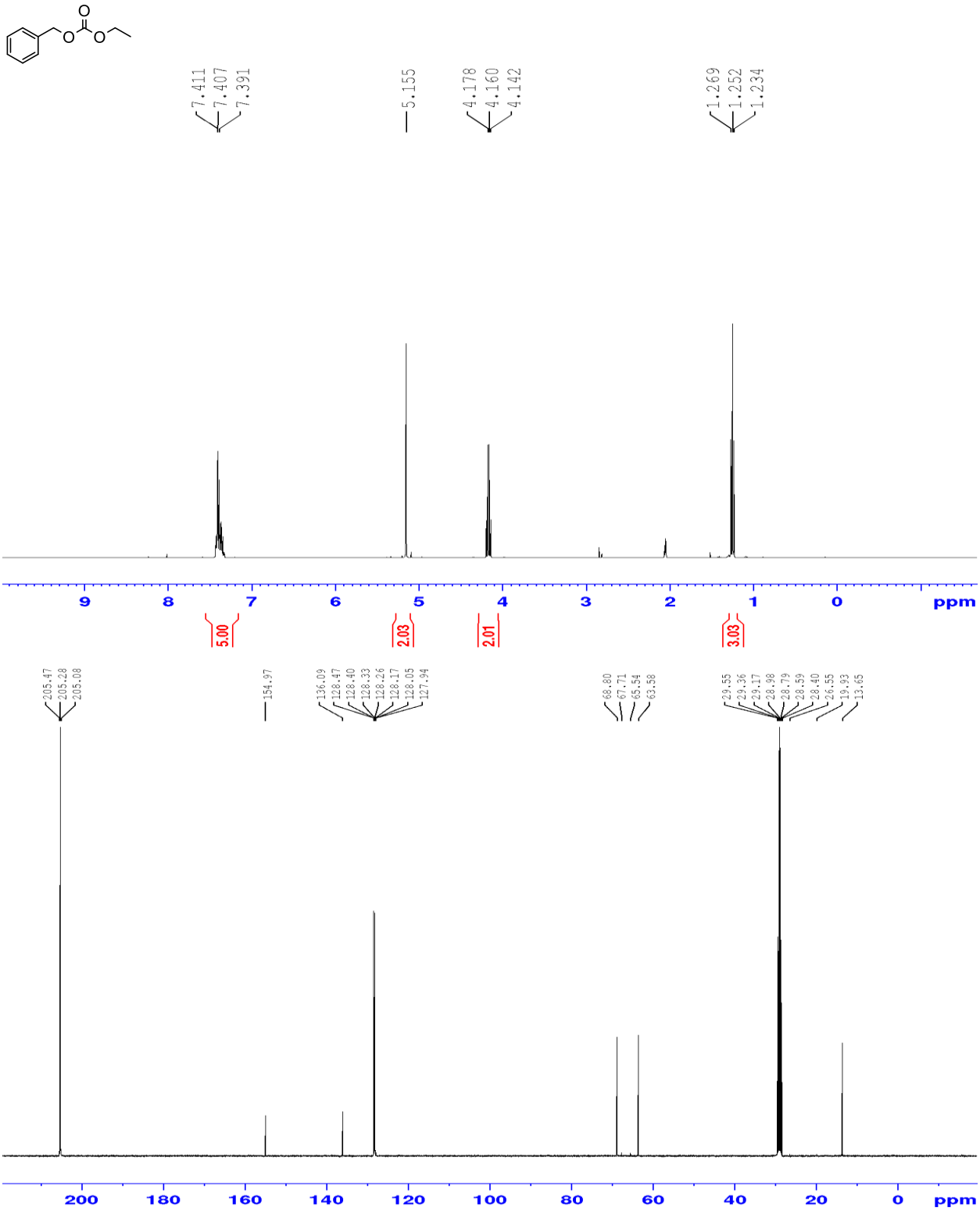


Figure S12 [1] H & [13] C spectra of product 5b in D-acetone. The spectra matched reference[7]

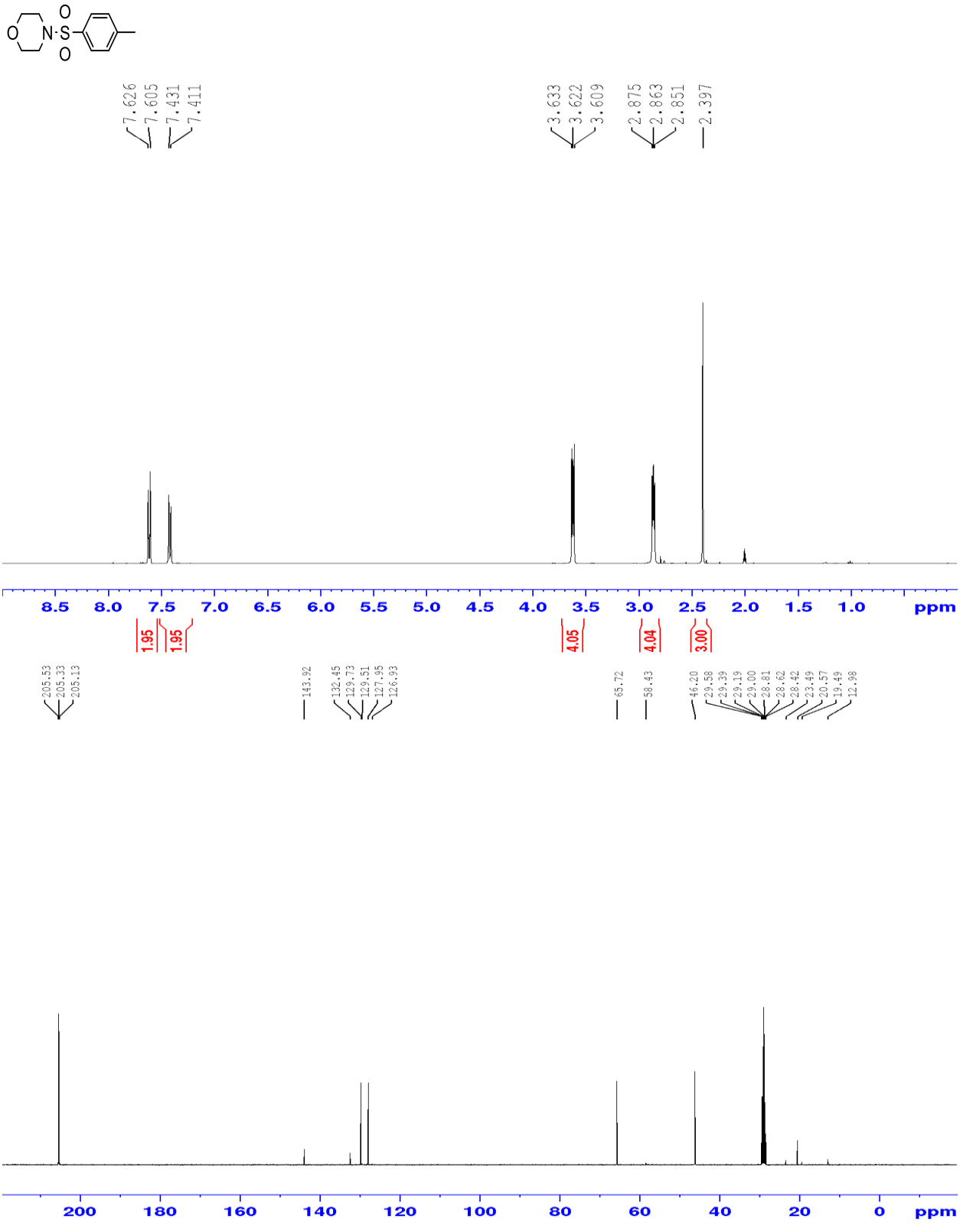


Figure S13 [1] H & [13] C spectra of product 6a in D-acetone. The spectra matched reference[13]

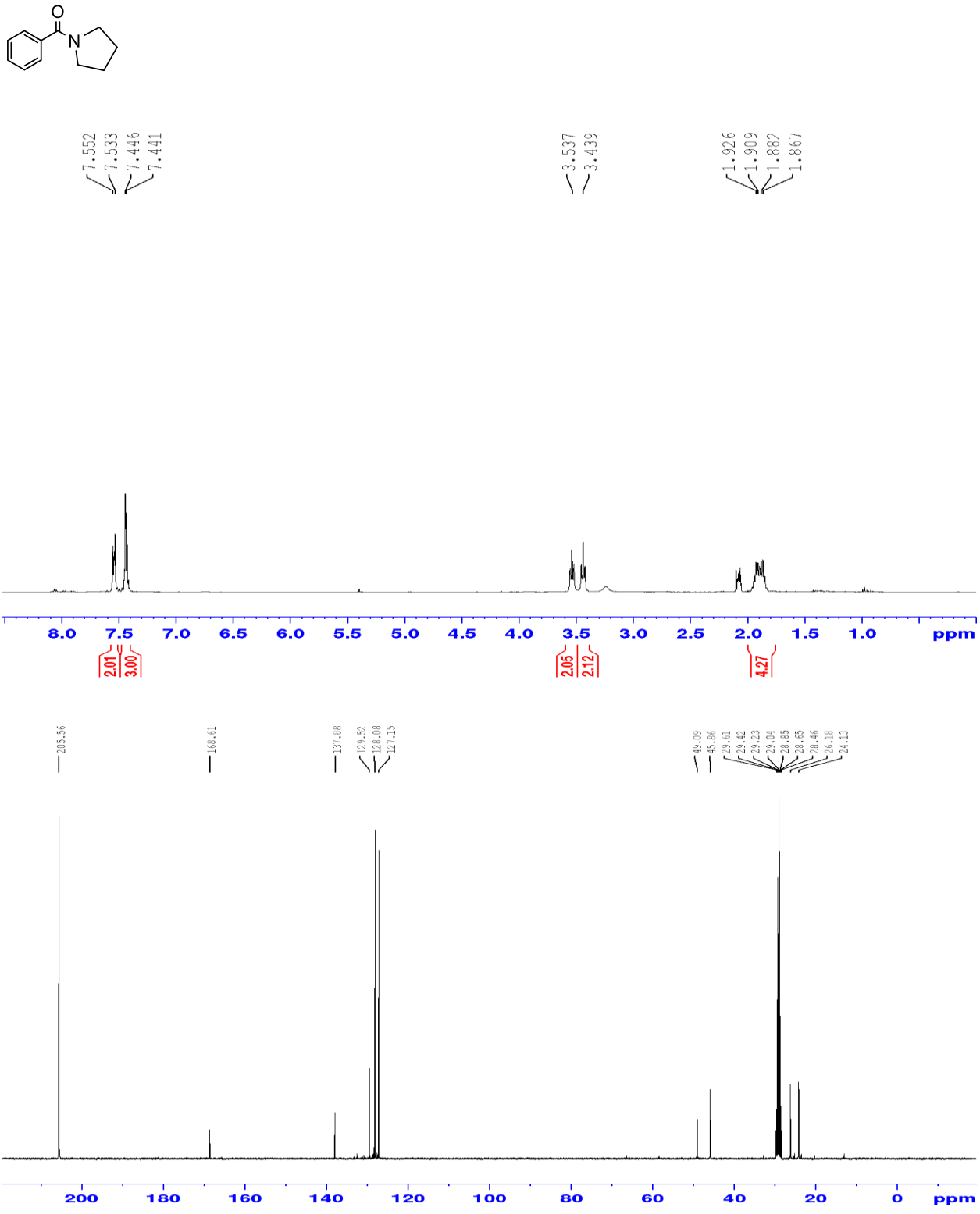


Figure S14 [1] H & [13] C spectra of product 7 in D-acetone. The spectra matched reference[12]

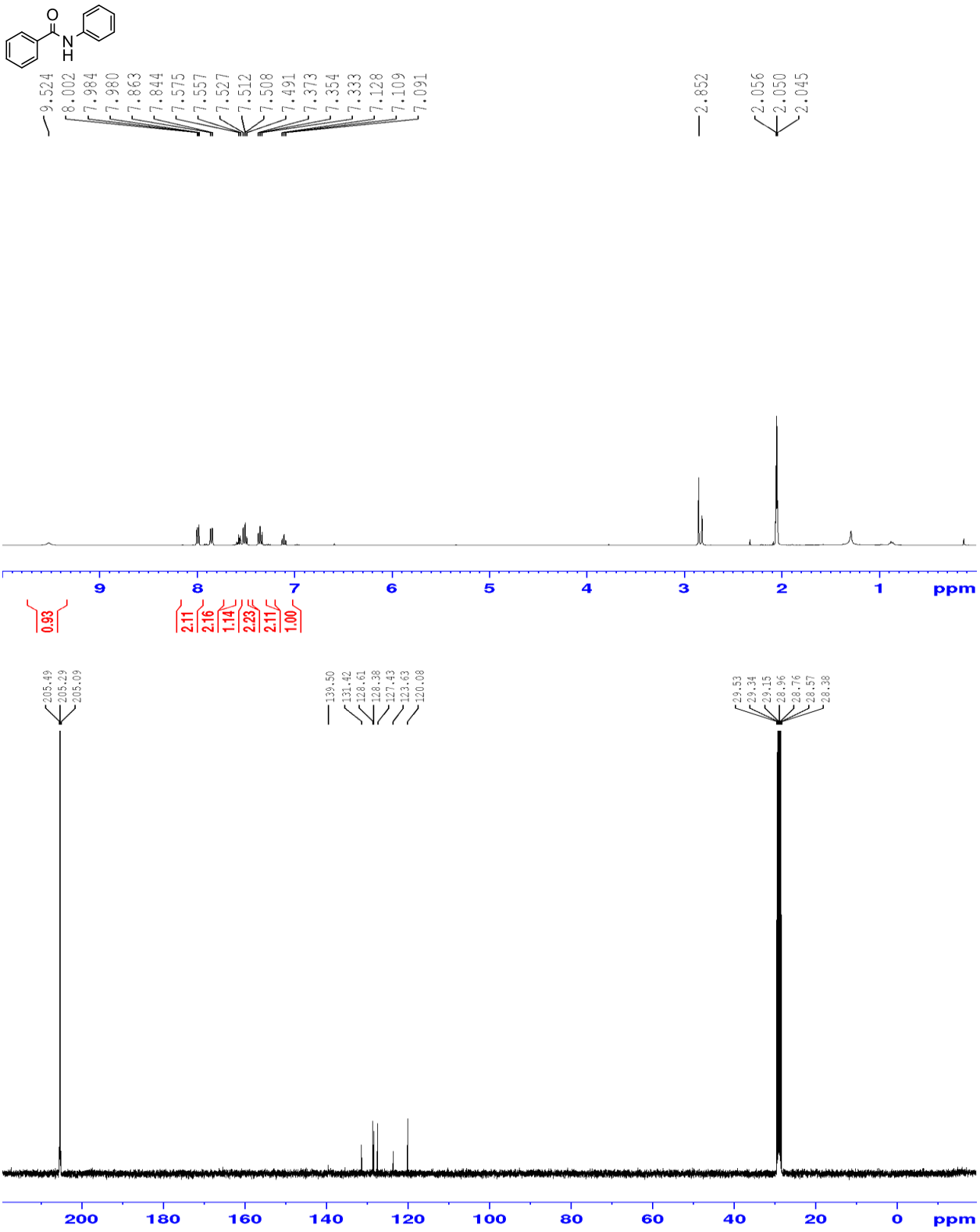


Figure S15 [1] H & [13] C spectra of product 8 in D-acetone. The spectra matched reference[15]

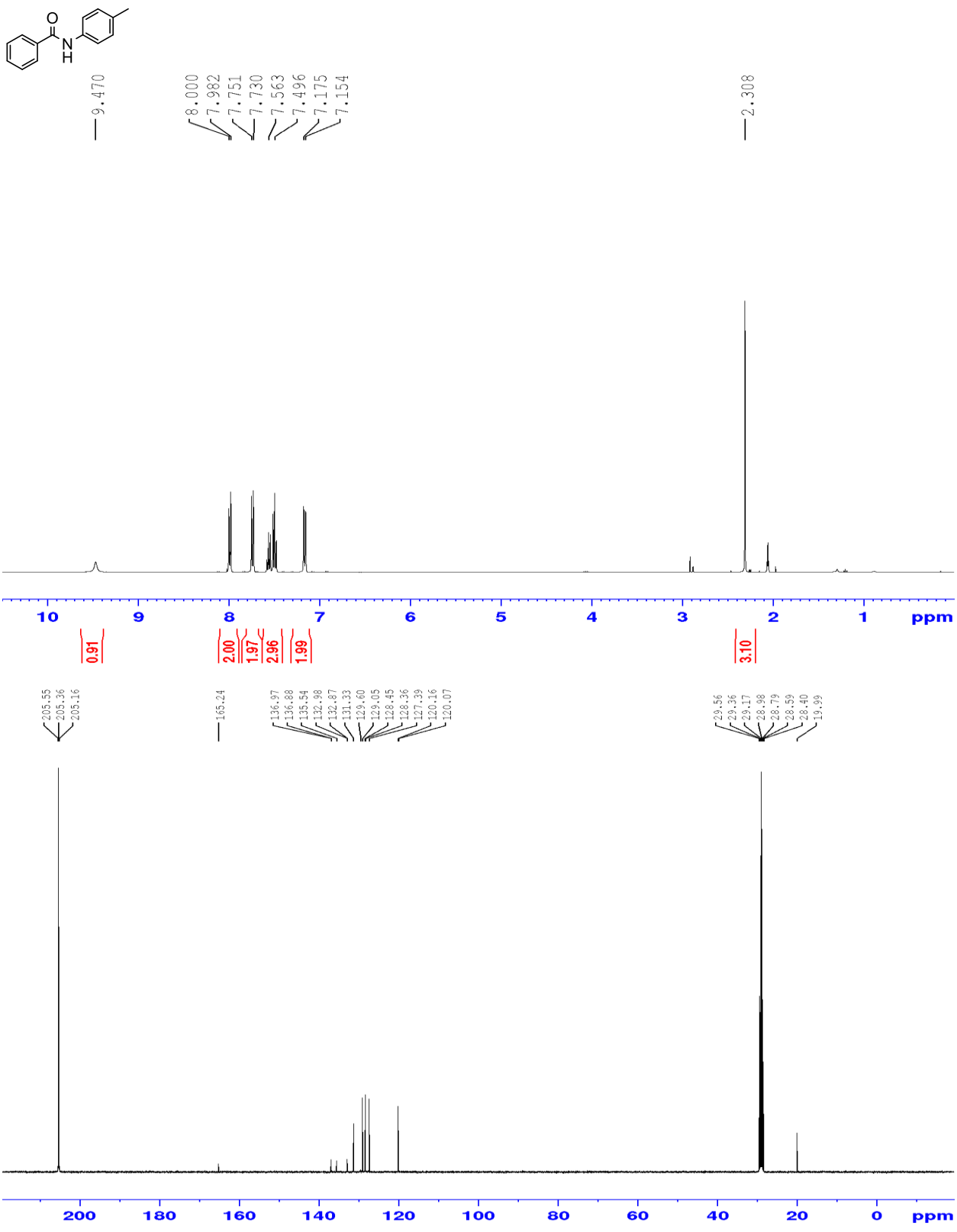


Figure S16 ^{1}H & ^{13}C spectra of product 9 in D-acetone. The spectra matched reference[6]

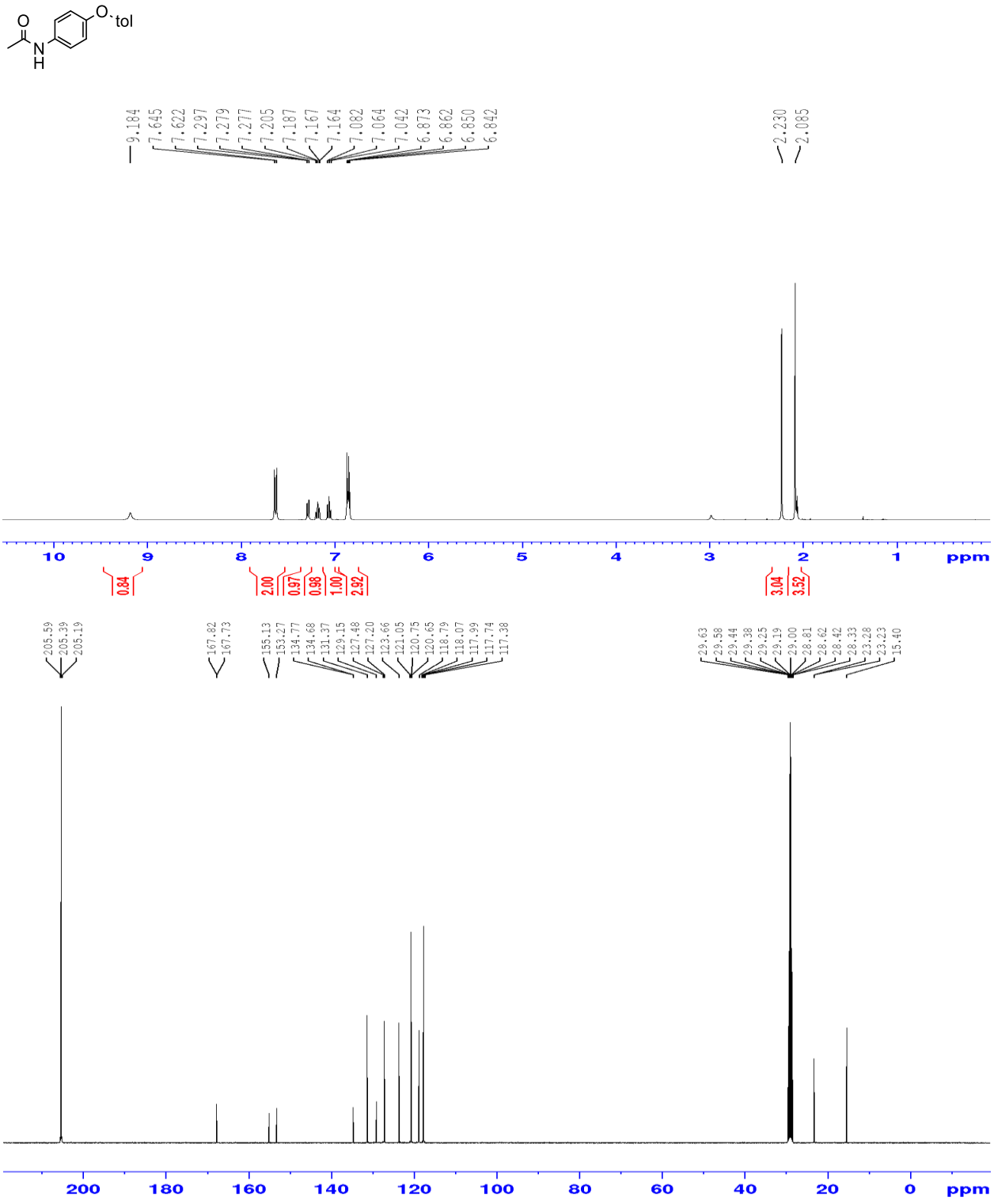


Figure S17 [1] H & [13] C spectra of product 10 in D-acetone. The spectra matched reference[2]

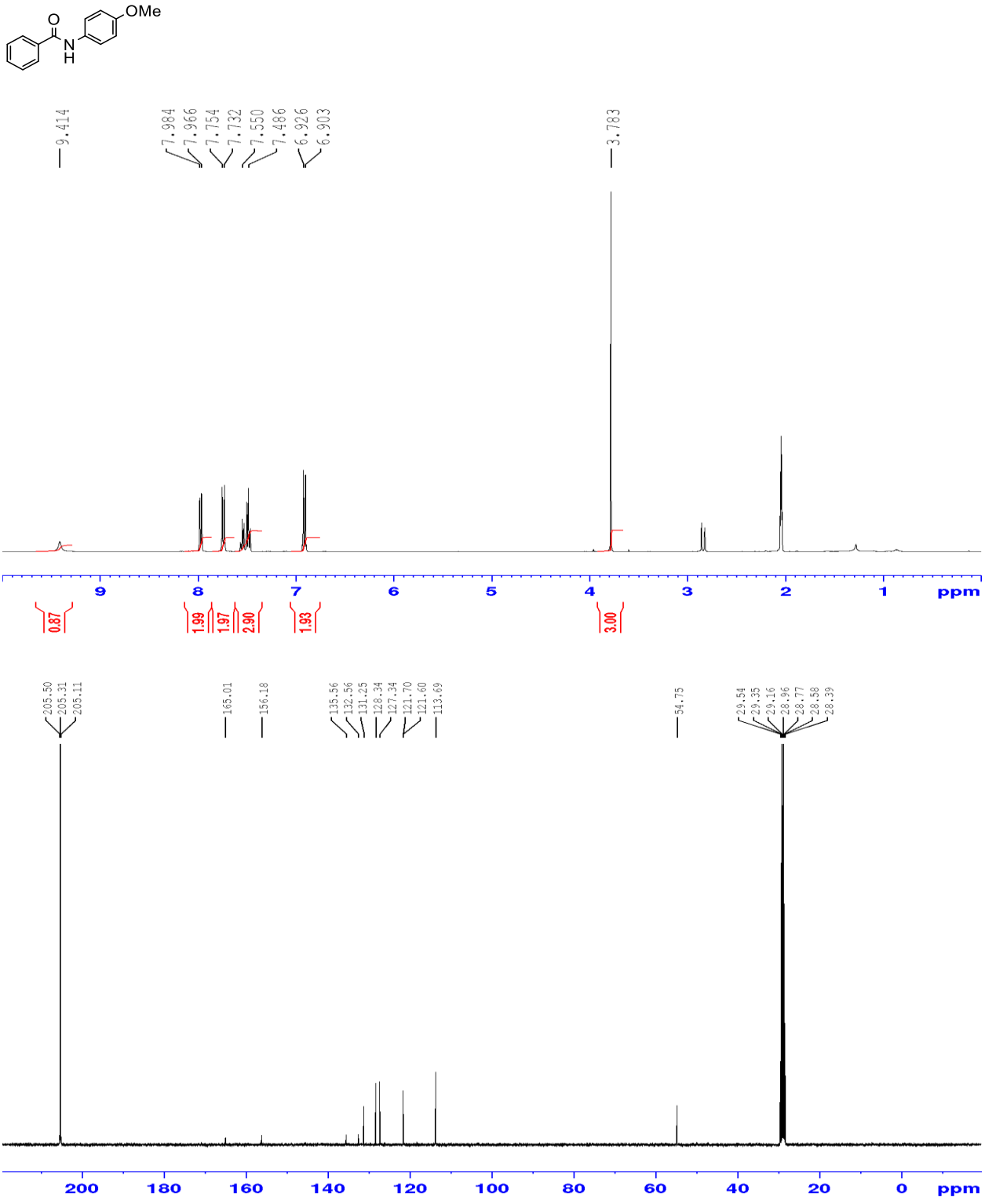


Figure S18 [1] H & [13] C spectra of product 11 in D-acetone. The spectra matched reference[2]

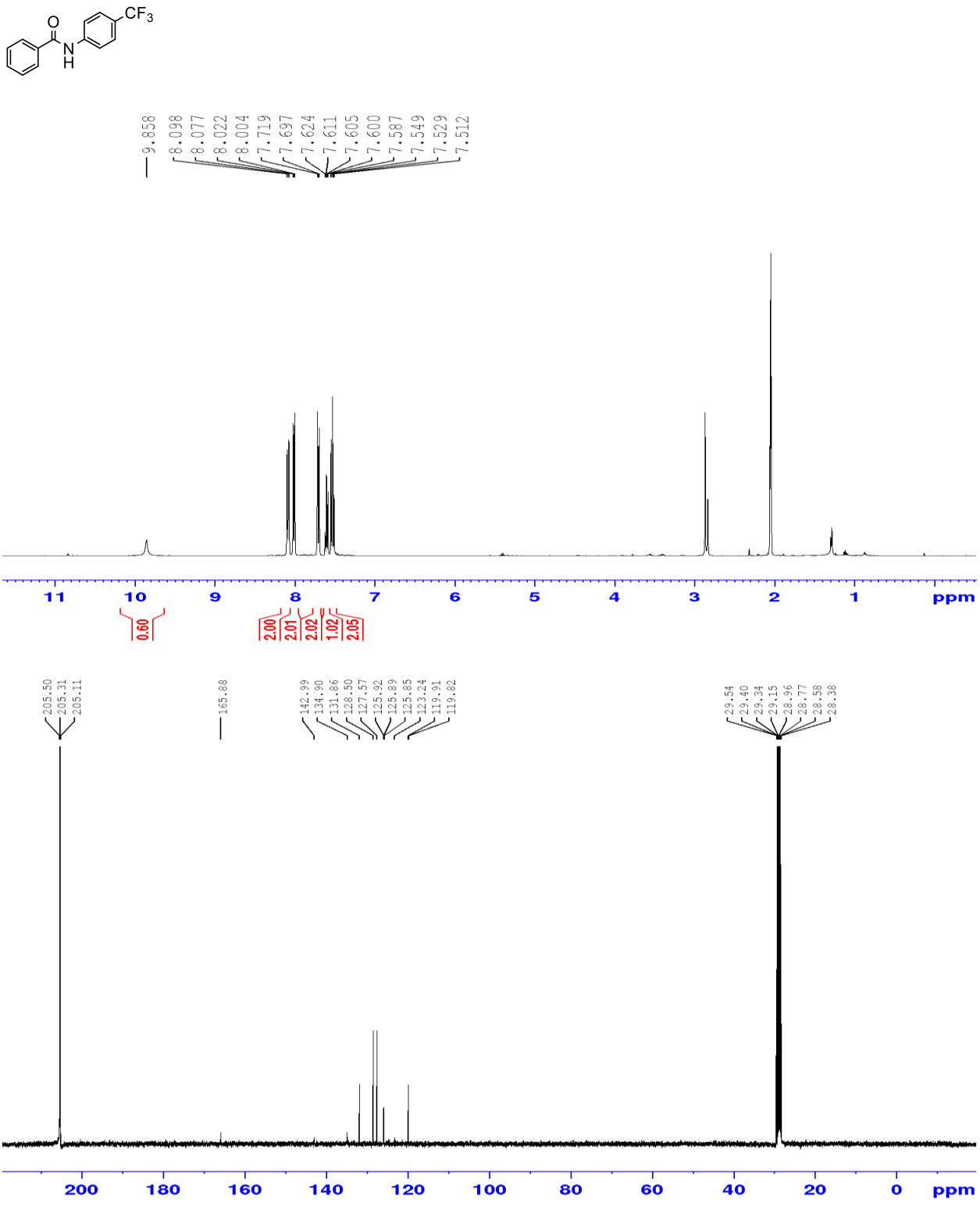


Figure S19 ^{1}H & ^{13}C spectra of product 12 in D-acetone. The spectra matched reference[16]

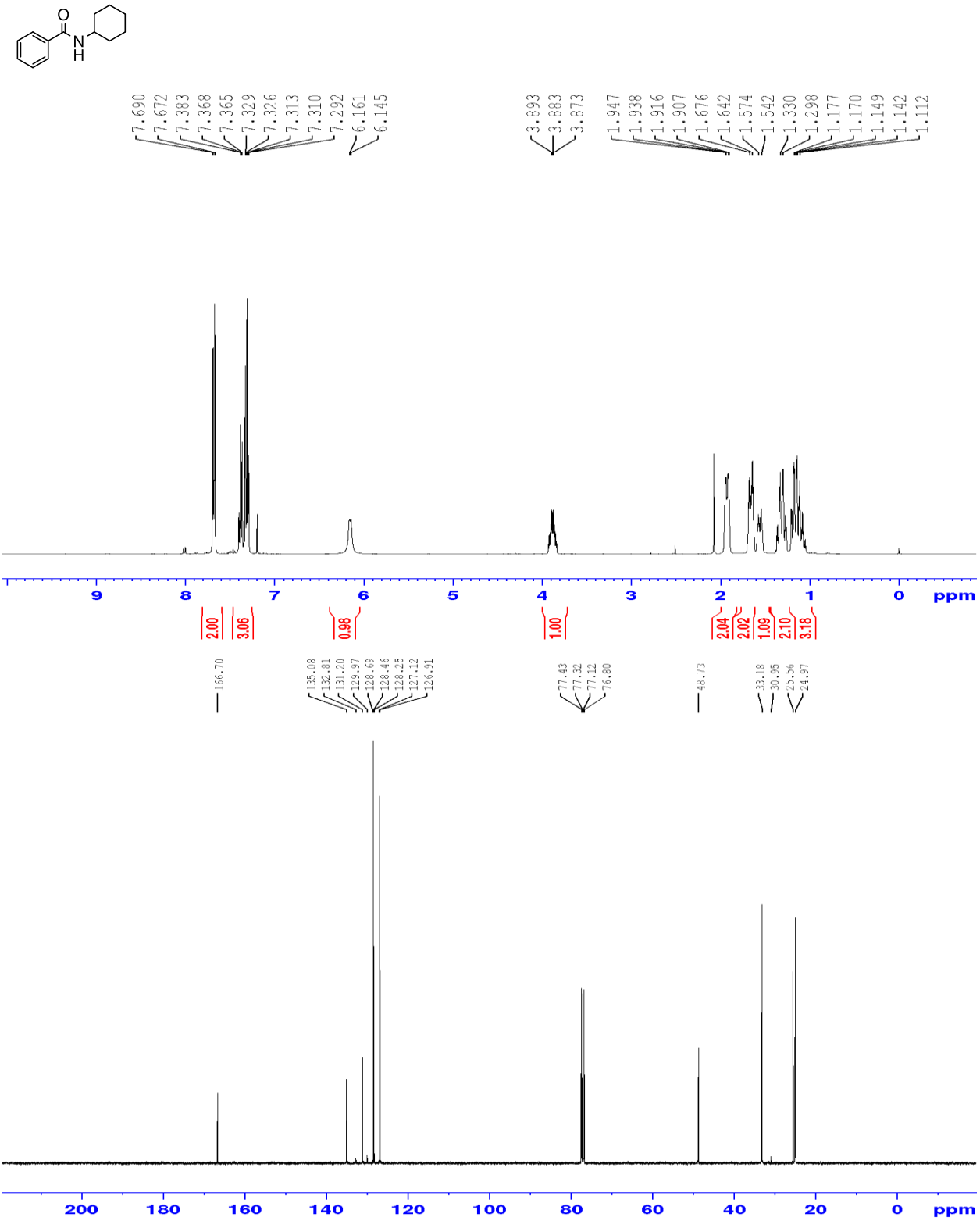


Figure S20 ^{1}H & ^{13}C spectra of product 13 in $CDCl_3$. The spectra matched reference[3]

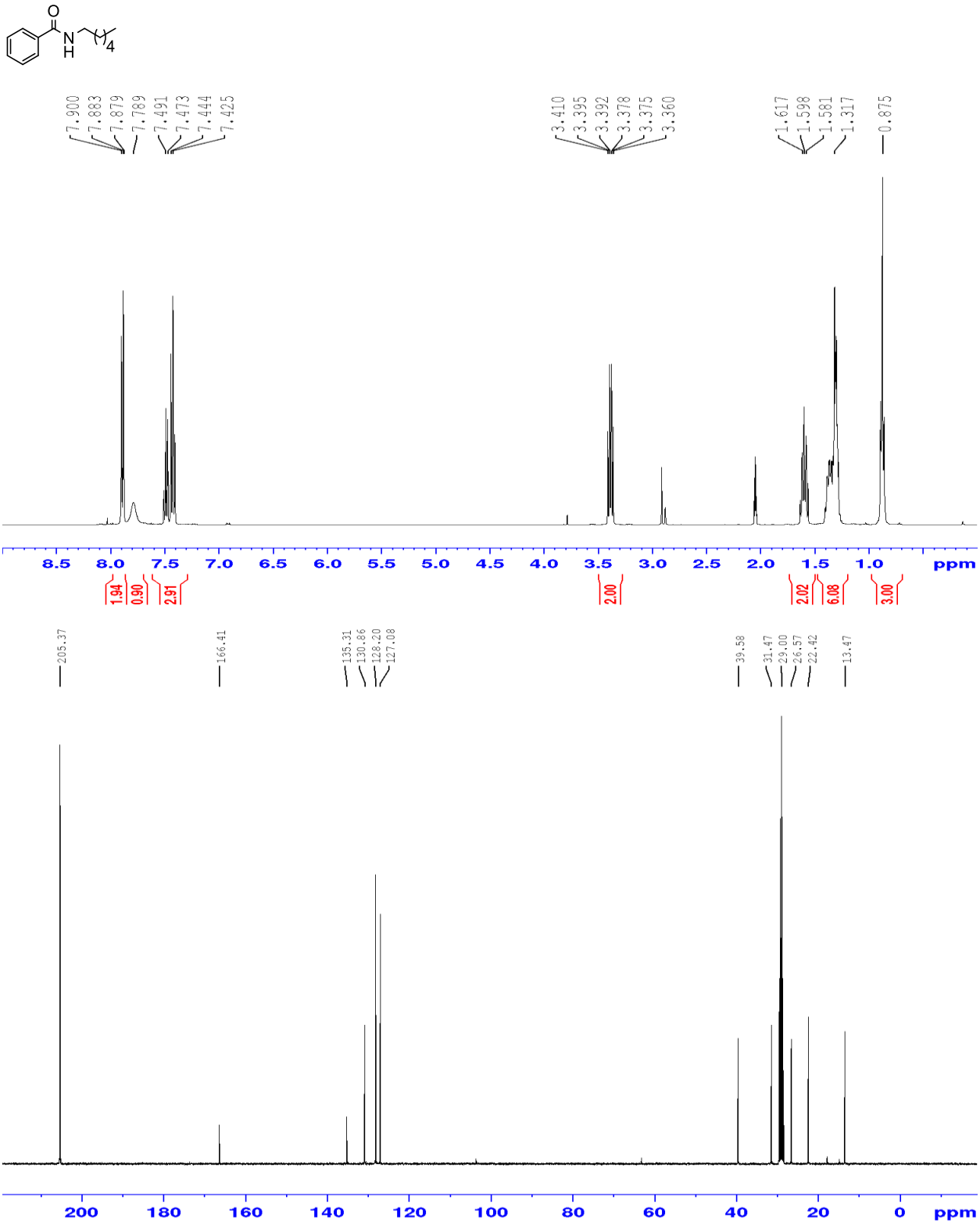


Figure S21 ^{1}H & ^{13}C spectra of product 14 in D-acetone. The spectra matched reference[10]

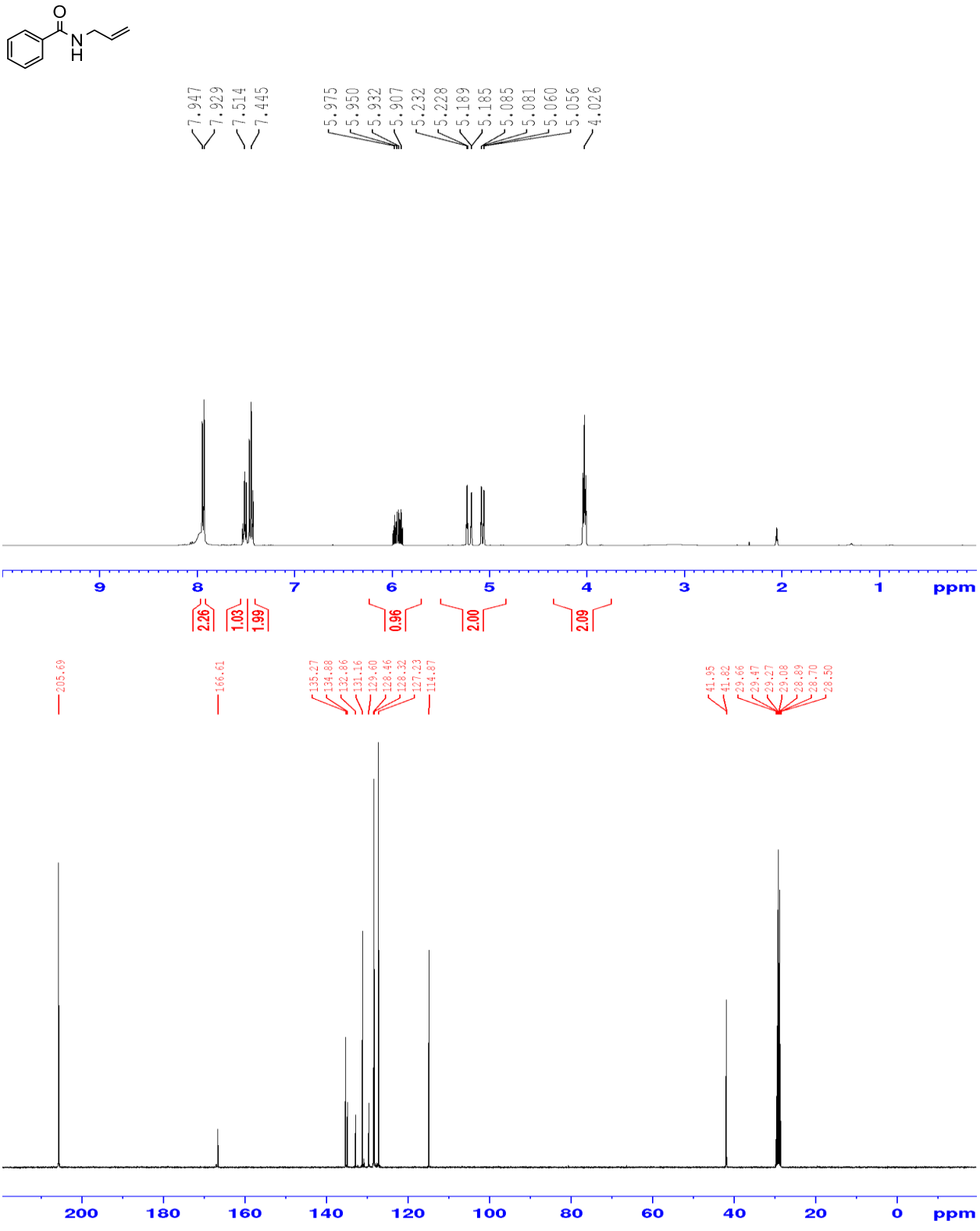


Figure S22 [1] H & [13] C spectra of product 16 in D-acetone. The spectra matched reference[19]

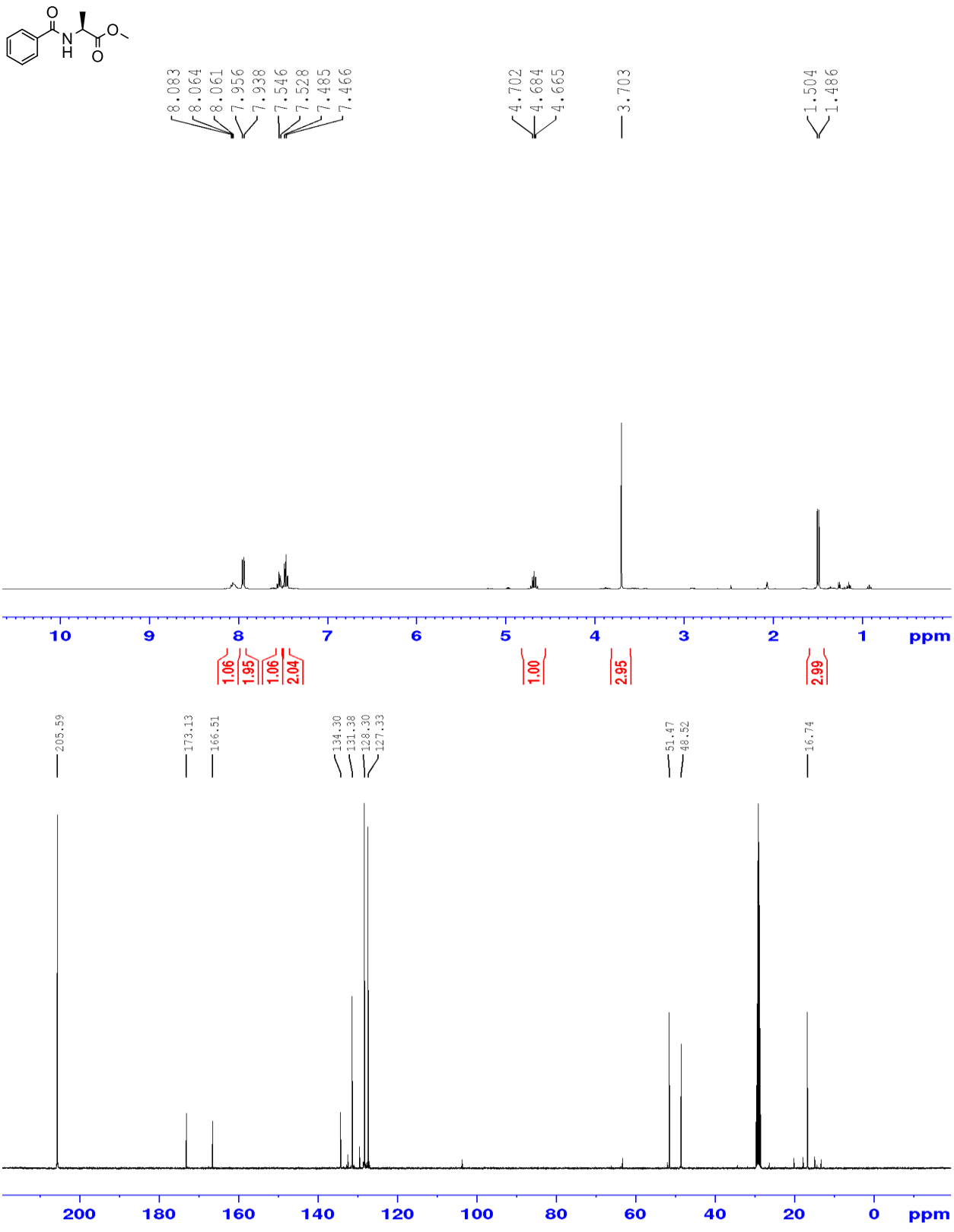


Figure S23 ^{1}H & ^{13}C spectra of product 17 in D-acetone. The spectra matched reference[9]

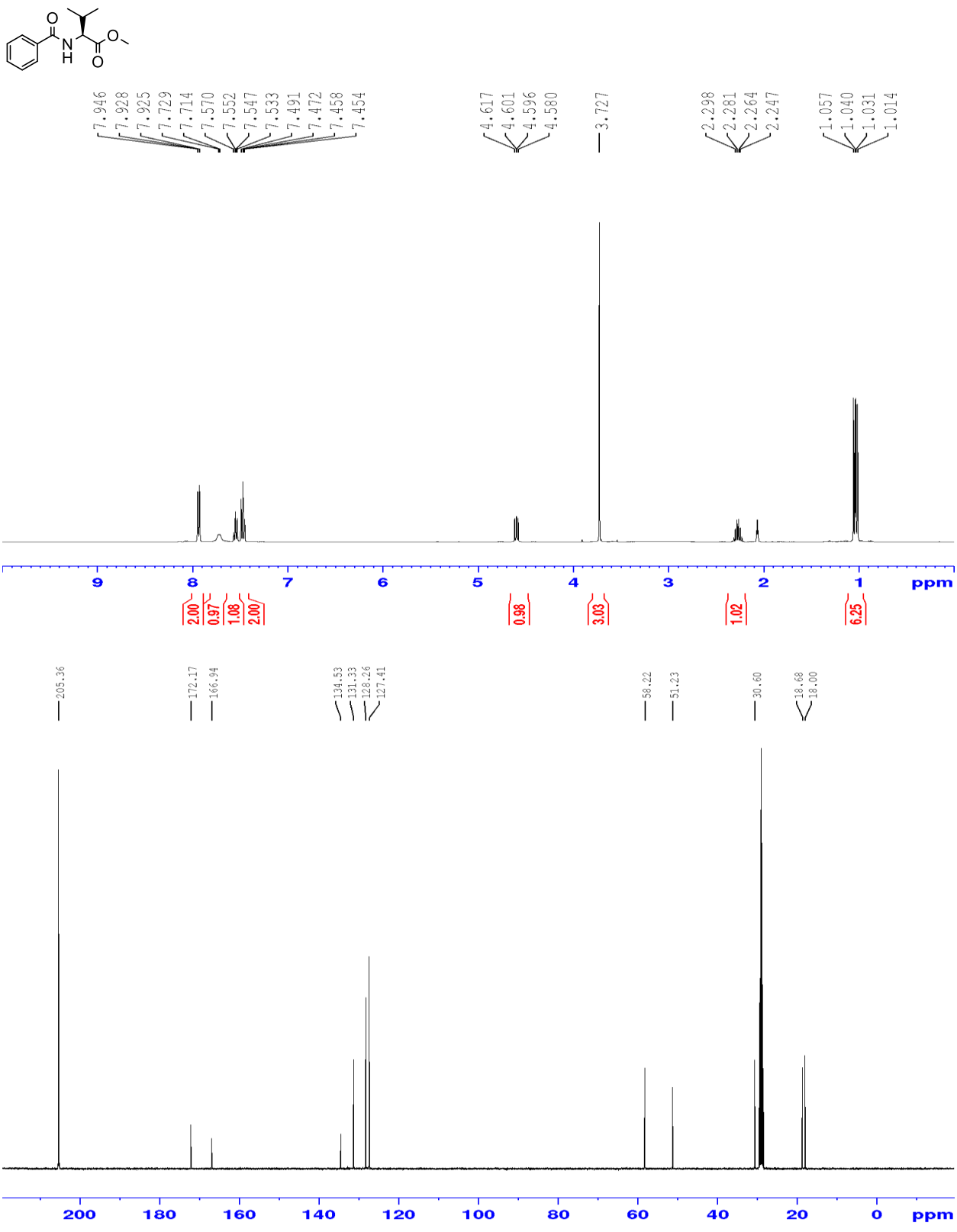


Figure S24 ^{1}H & ^{13}C spectra of product 18 in D-acetone. The spectra matched reference[14]

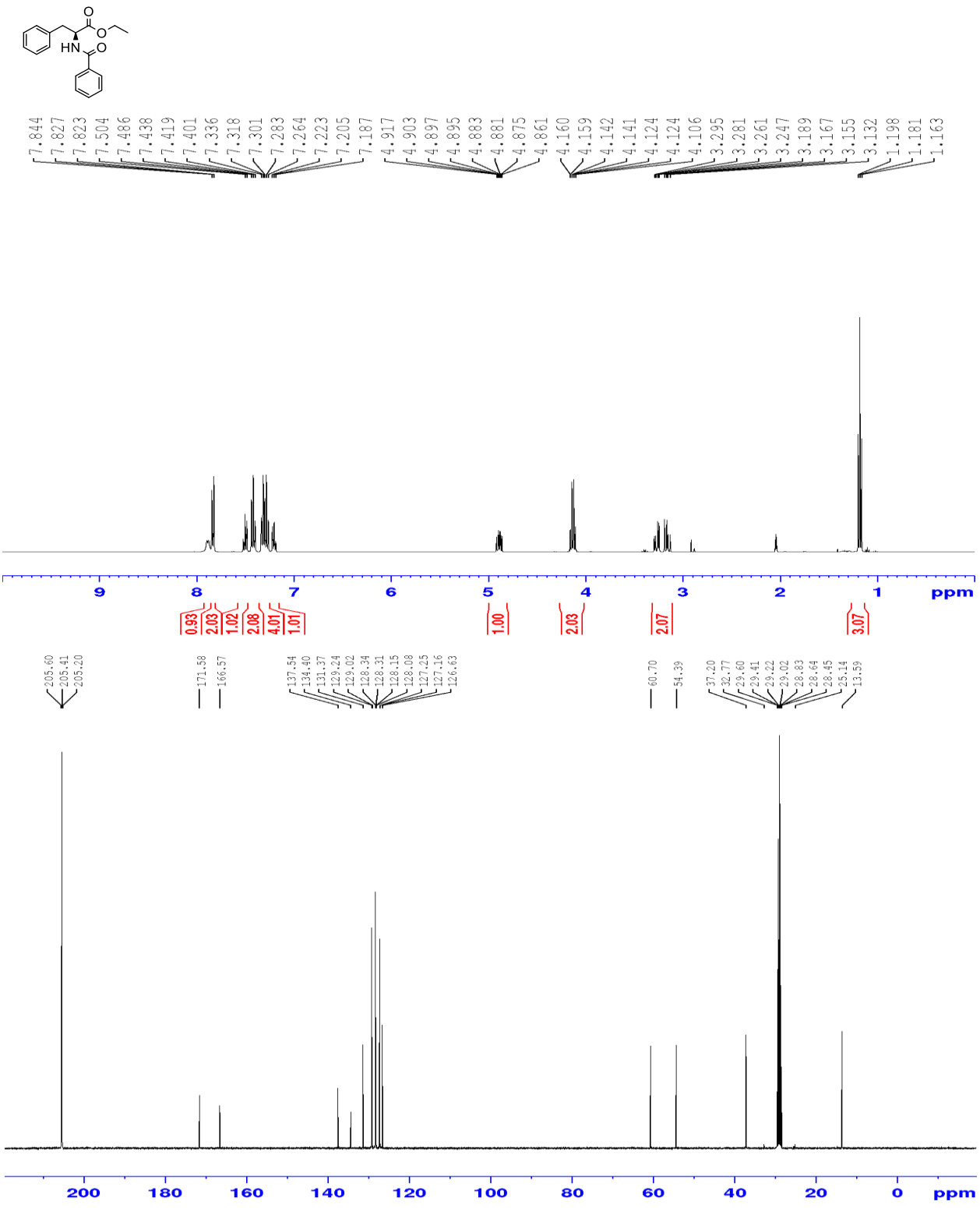


Figure S25 [1] H & [13] C spectra of product 19 in D-acetone. The spectra matched reference[14]

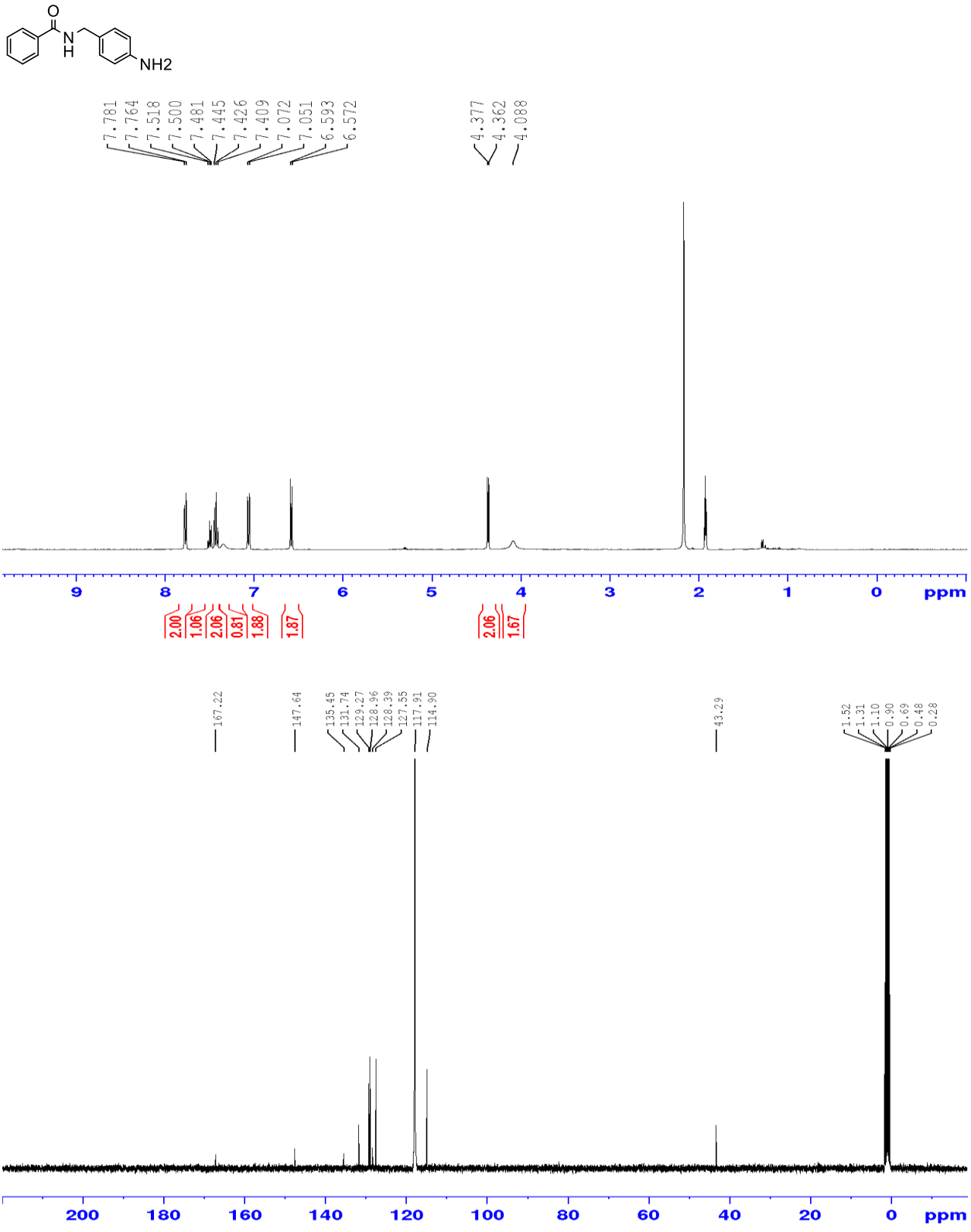


Figure S26 [1] H & [13] C spectra of product 20 in D-acetone. The spectra matched reference[14]

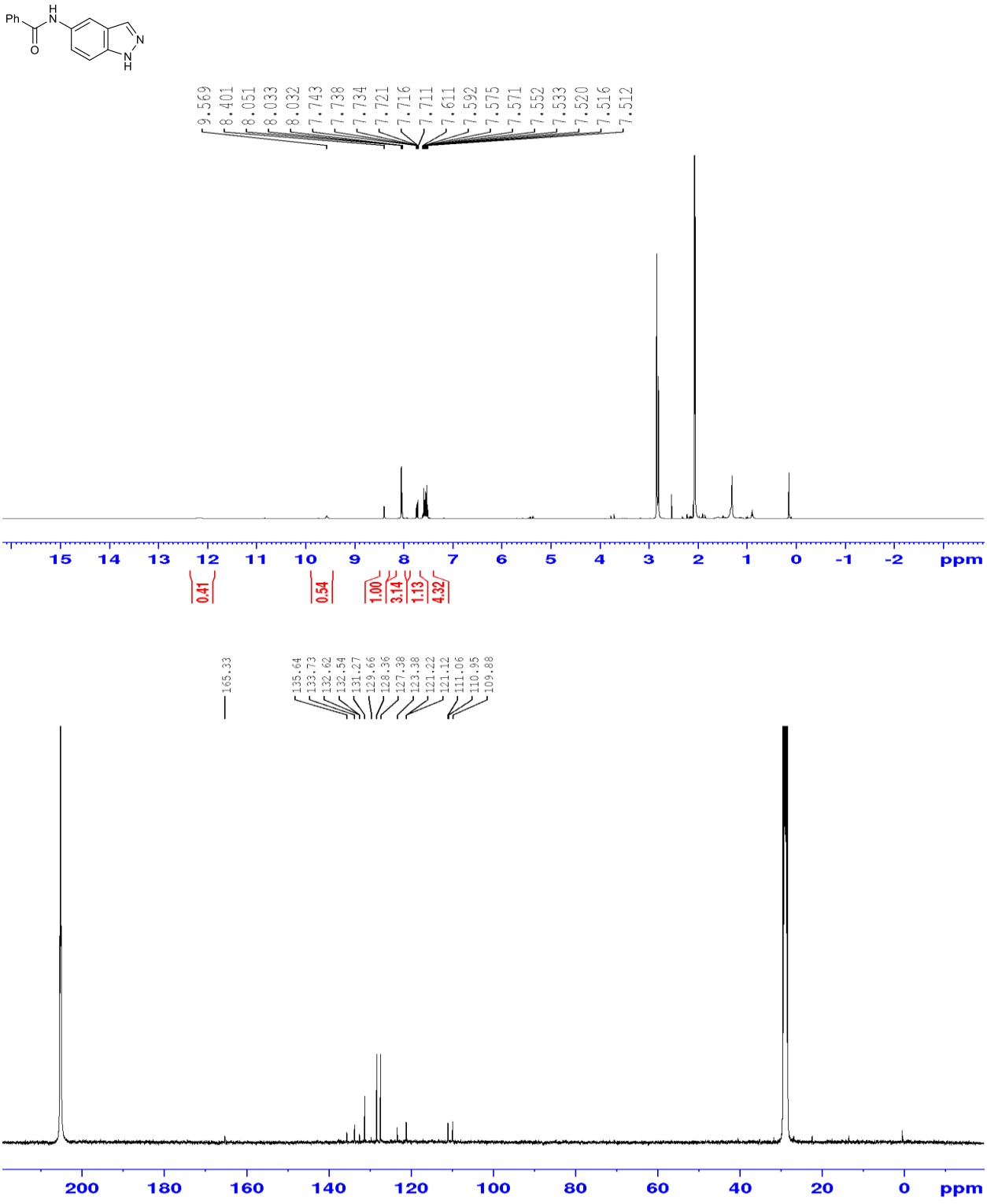


Figure S27 ^{1}H & ^{13}C spectra of product 20. The spectra matched reference[1]

PMI calculation for pyrrolidine amidation

A separate experiment was setup in an H cell using smaller volumes of solvent and electrolyte quantity. The volume of acetonitrile used for both cathode and anode was 3.5 mL. Each compartment contained 50 mM electrolyte. The cathode compartment contained 0.25 mmol of pyrrolidine and anode compartment contained 0.25 mmol ferrocene. After electrolysis is complete, an equivalent amount of benzoic anhydride was added to permit nucleophilic addition. It was followed by addition of an equivalent of benzyl bromide for nucleophilic substitution.

Mass of pyrrolidine = 0.018 g

Mass of benzoic anhydride = 0.056 g

Mass of benzyl bromide = 0.059 g

Mass of solvent = 5.5 g

Mass of electrolyte = 0.134 g

Mass of Ferrocene = 0.046 g

Mass of the amide = 0.096 g

Mass of the ester = 0.097 g

PMI = total mass/mass of products = 5.813/0.193 = 30.12

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