

Ag(I)/persulfate-catalyzed Decarboxylative Coupling of α -Oxocarboxylates with Organotrifluoroborates under Room Temperature

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Supporting Materials

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General

Starting materials and solvents were purchased from common commercial sources and were used without additional purification. ^1H NMR spectra were recorded at 400 MHz and ^{13}C NMR spectra were recorded at 100 MHz, using TMS as internal standard. Multiplicities are reported as: singlet (s), doublet (d), triplet (t) and multiplet (m). HRMS (EI) data were collected on High Resolution mass spectrometer.

Preparation of Potassium Aryltrifluoroborate Salts:

The arylboronic acid (10 mmol) was dissolved in methanol (5 mL), A solution of saturated KHF_2 (10 mL) was added slowly to the mixture with intense stirring in 10 min. The precipitation was washed with cold methanol and using acetonitrile for recrystallization to afford the corresponding aryltrifluoroborate salts.

Preparation of Preparation of Potassium α -Oxocarboxylates:

A mixture of aryl methyl ketone (10.0 mmol) and selenium dioxide (20.0 mmol) in dry pyridine (5 mL) was stirred at 120°C under nitrogen for 12 h. After the disappearance of acetophenone detected by TLC, the mixture was filtrated and removing the organic solvent by evaporation. Then 2 M potassium hydroxide solution was added to the residue and some ethyl acetate followed. The aqueous was subsequently evaporated and potassium α -oxocarboxylates were obtained by recrystallization.

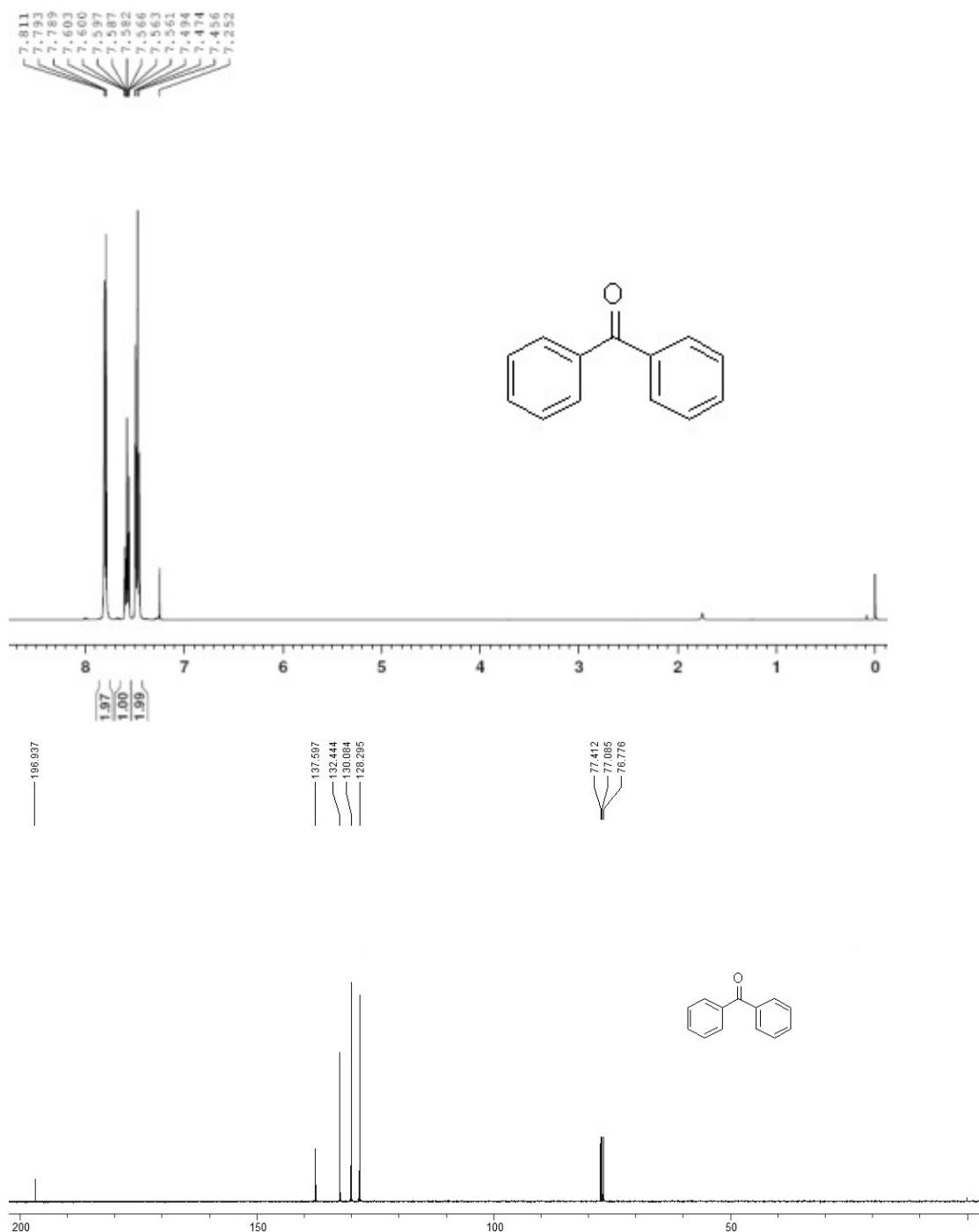
General procedure for Ag(I)/persulfate-catalyzed Decarboxylative Coupling

A mixture of potassium α -oxocarboxylates (1 mmol), aryltrifluoroborates (1.05 mmol), AgNO_3 (0.05 mol) and $\text{K}_2\text{S}_2\text{O}_8$ (1.5 mmol)was stirred in H_2O (2 mL) under room temperature for the indicated time. Afterward, the reaction solution was extracted four times with CH_2Cl_2 ($3 \times 2\text{mL}$), and the combined organic phase was subjected to evaporation The further purification of the product was achieved by flash chromatography on a silica gel column.

Characterization data of the product

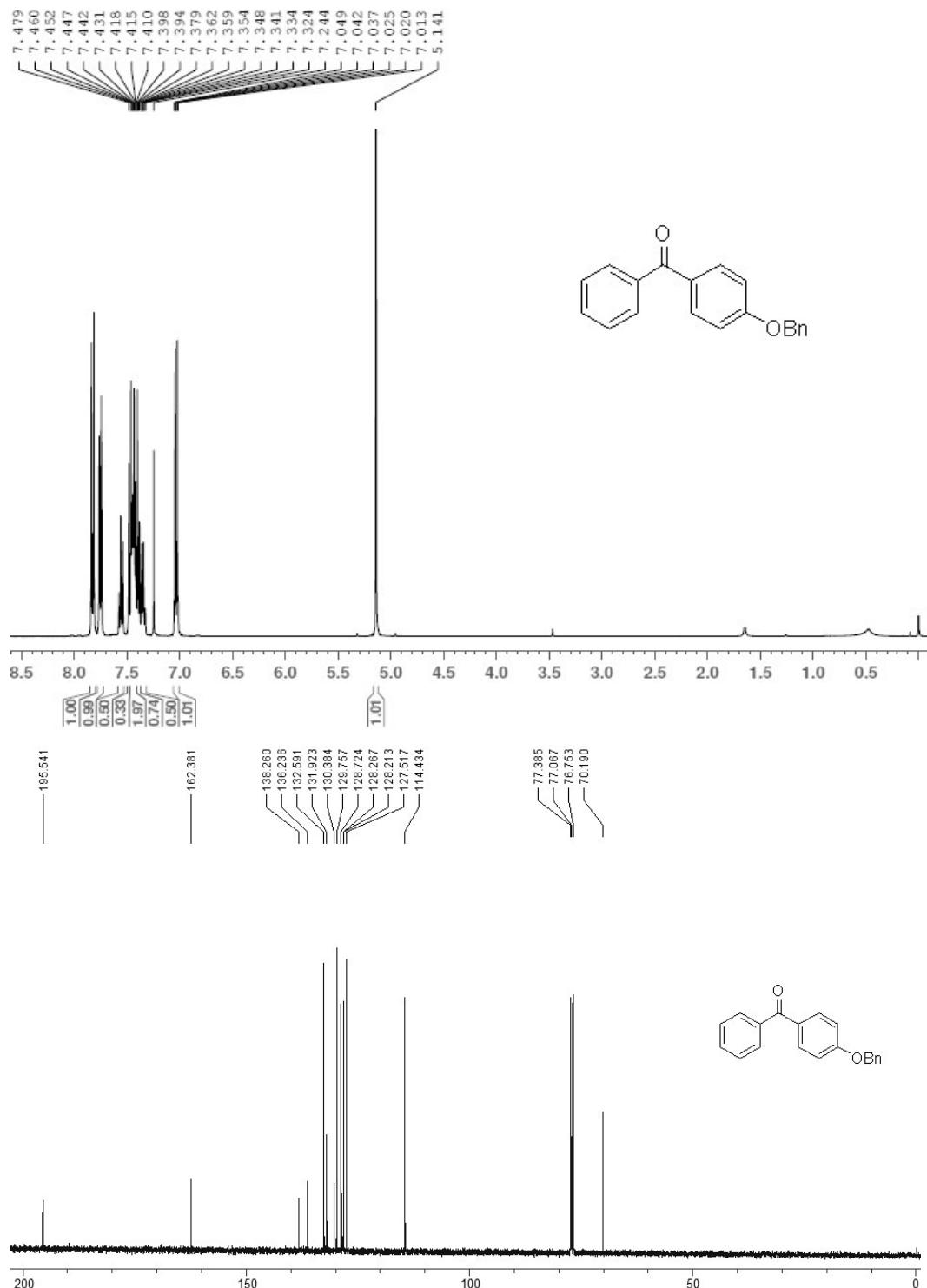
(1) benzophenone (3a)

m.p. 48–49 °C (lit.¹ mp 47–48 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.82 (d, *J* = 8.0 Hz, 4 H), 7.58 (t, *J* = 7.6 Hz, 2 H), 7.47 (t, *J* = 7.6 Hz, 4 H). ¹³C NMR (100 MHz, CDCl₃) δ 197.0, 137.6, 132.5, 130.1, 128.3. HRMS (EI) Calcd for C₁₃H₁₀O (M⁺) 182.0732, Found 182.0738.



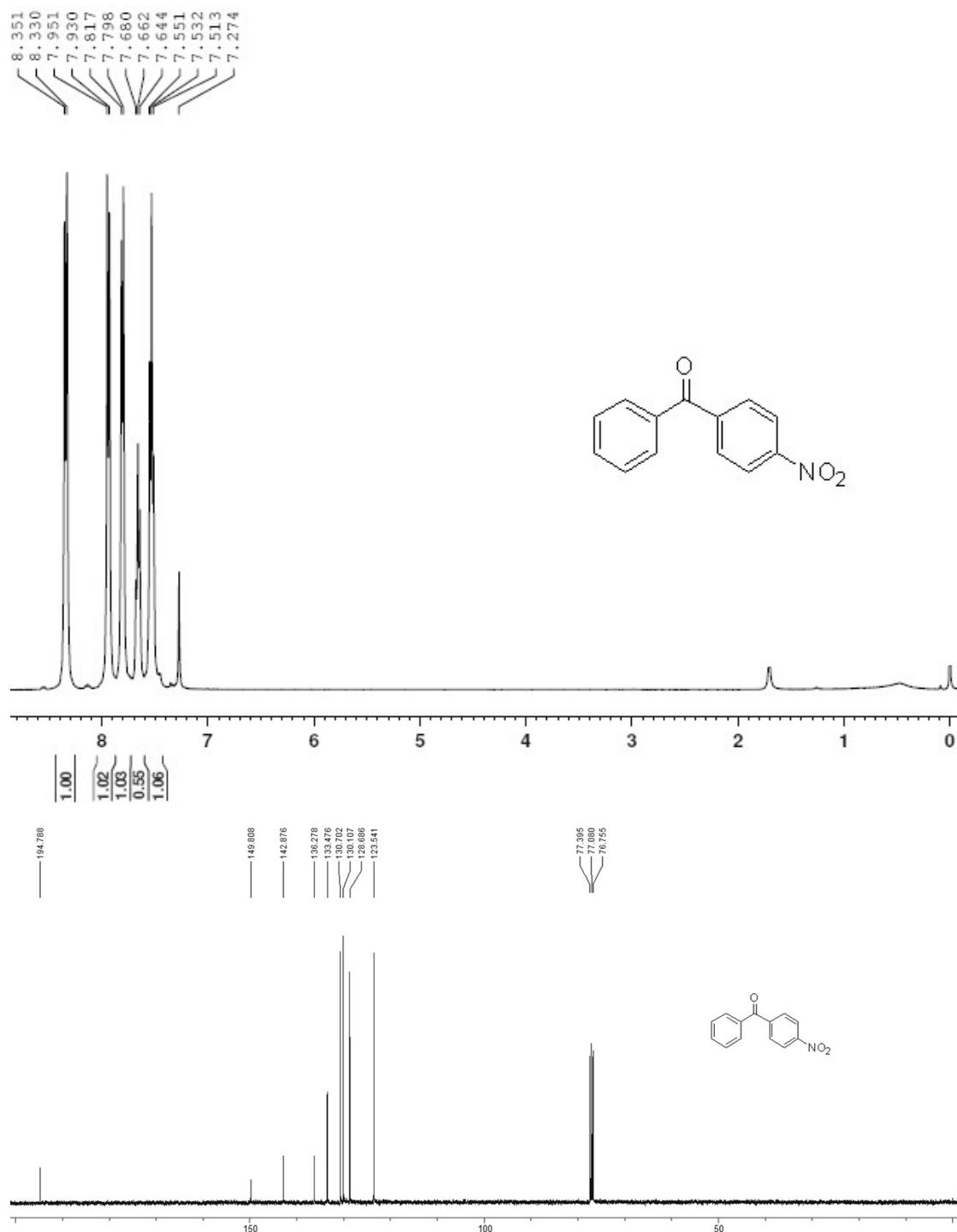
(2) 4-benzyloxy-benzophenone (3b)

m.p. 83–85 °C (lit.¹ mp 82–85 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.82 (d, *J* = 8.4 Hz, 2 H), 7.75 (t, *J* = 7.2 Hz, 2 H), 7.55 (t, *J* = 7.2 Hz, 1 H), 7.32–7.48 (m, 7 H), 7.03 (d, *J* = 8.4 Hz, 2 H), 5.14 (s, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 195.5, 162.4, 138.3, 136.2, 132.6, 131.9, 130.4, 129.8, 128.7, 128.3, 128.2, 127.5, 114.4, 70.2. HRMS (EI) Calcd for C₂₀H₁₆O₂ (M⁺) 288.1150, Found 288.1143.



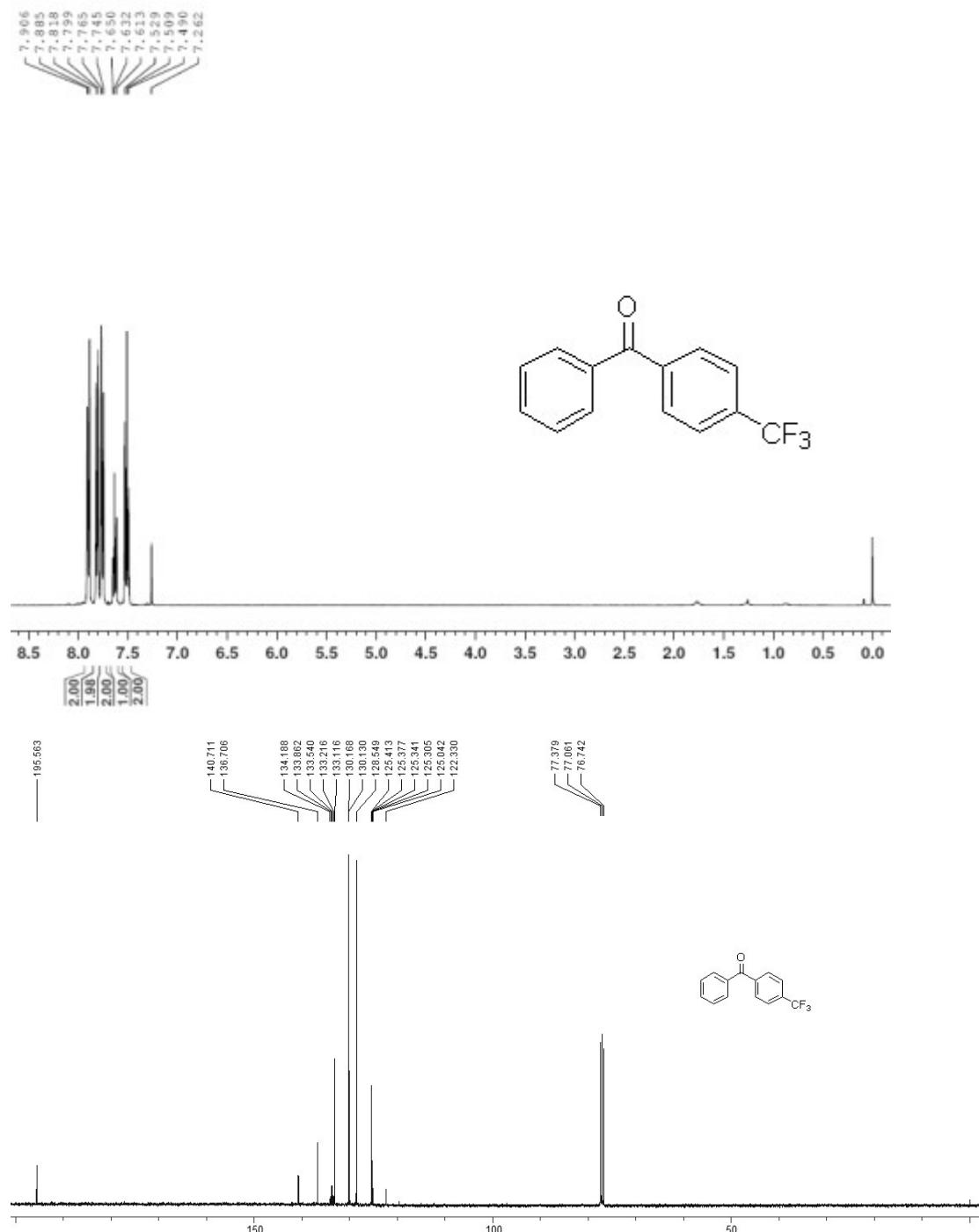
(3) 4-nitrobenzophenone (3c)

m.p. 142–143 °C (lit.² mp 140–142 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 8.36 (d, *J* = 7.6 Hz, 2 H), 7.94 (d, *J* = 7.6 Hz, 2 H), 7.83 (d, *J* = 7.6 Hz, 2 H), 7.67 (t, *J* = 7.6 Hz, 1 H), 7.51 (t, *J* = 7.6 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 194.8, 149.8, 142.9, 136.3, 133.5, 130.7, 130.1, 128.7, 123.5. HRMS (EI) Calcd for C₁₃H₉NO₃ (M⁺) 227.0582, Found 227.0586.



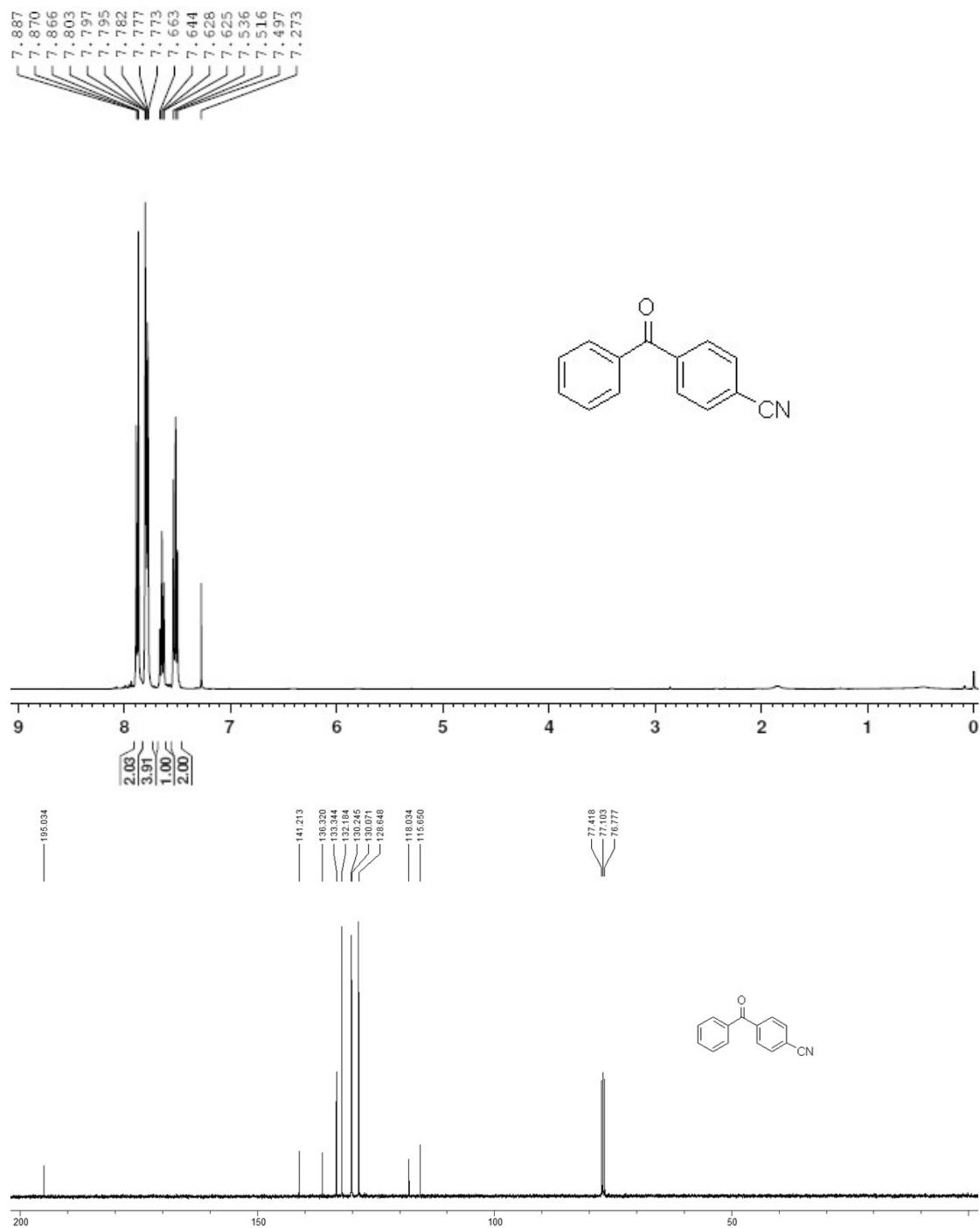
(4) 4-trifluoromethyl-benzophenone (3d)

m.p. 115–117 °C (lit.² mp 116–117 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.90 (d, *J* = 8.0 Hz, 2 H), 7.81 (d, *J* = 7.2 Hz, 2 H), 7.75 (d, *J* = 8.0 Hz, 2 H), 7.63 (t, *J* = 7.2 Hz, 1 H), 7.52 (t, *J* = 7.2 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 195.6, 140.7, 136.7, 133.7 (dd, *J*₁ = 64.6 Hz, *J*₂ = 32.6 Hz), 133.1, 130.2 (d, *J* = 3.8 Hz), 128.6, 125.4 (dd, *J*₁ = 7.2 Hz, *J*₂ = 3.6 Hz), 123.7 (d, *J* = 271.2 Hz). HRMS (EI) Calcd for C₁₄H₉F₃O (M⁺) 250.0605, Found 250.0602



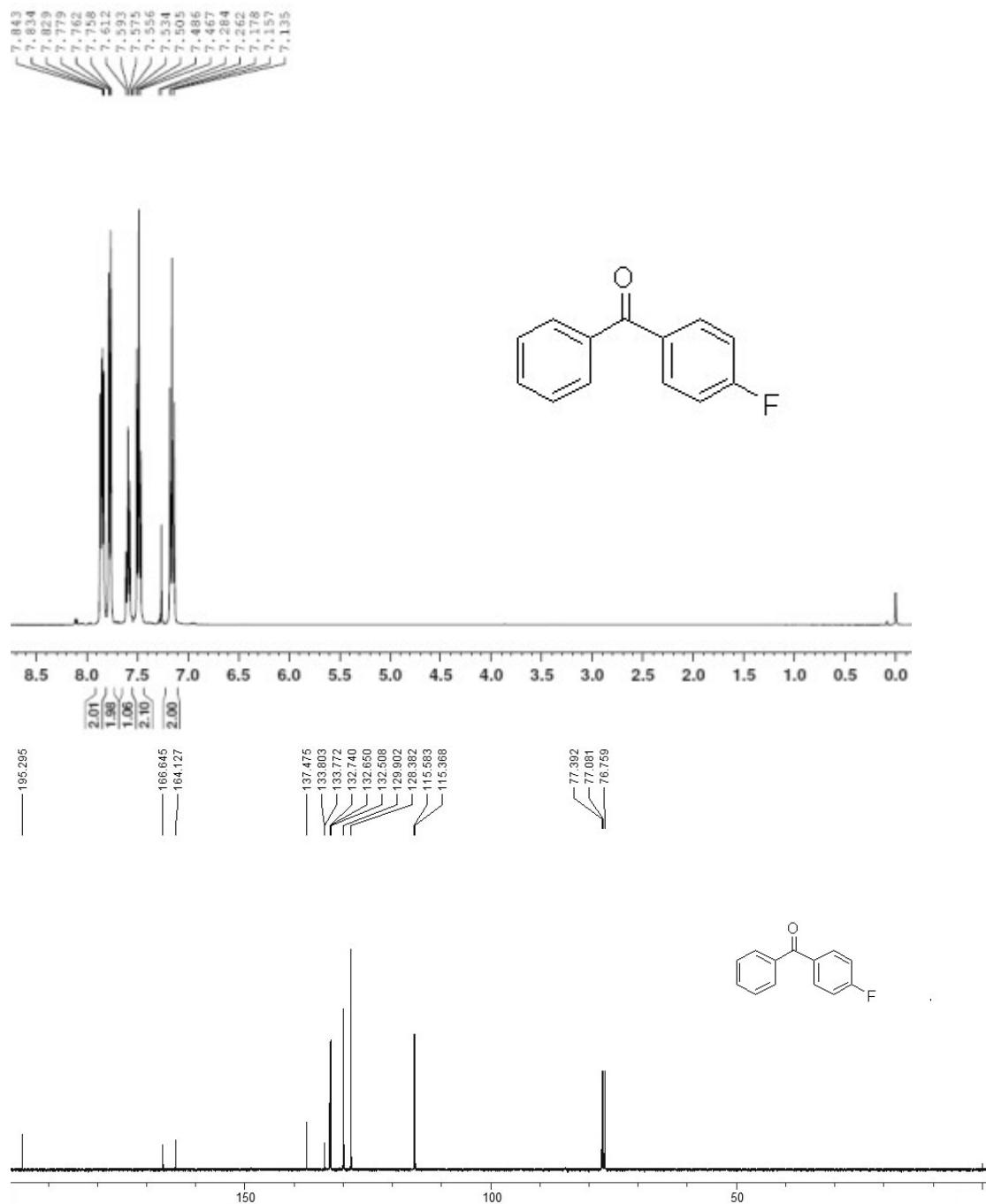
(5) 4-benzoylbenzonitrile (3e)

m.p. 114–115 °C (lit.² mp 114–116 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.88 (t, *J* = 8.0 Hz, 2 H), 7.77-7.80 (m, 4 H), 7.64 (t, *J* = 7.6 Hz, 1 H), 7.52 (t, *J* = 7.6 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 195.0, 141.2, 136.3, 133.3, 132.2, 130.3, 130.1, 128.7, 118.0, 115.7. HRMS (EI) Calcd for C₁₄H₉NO (M⁺) 207.0684, Found 207.0680.



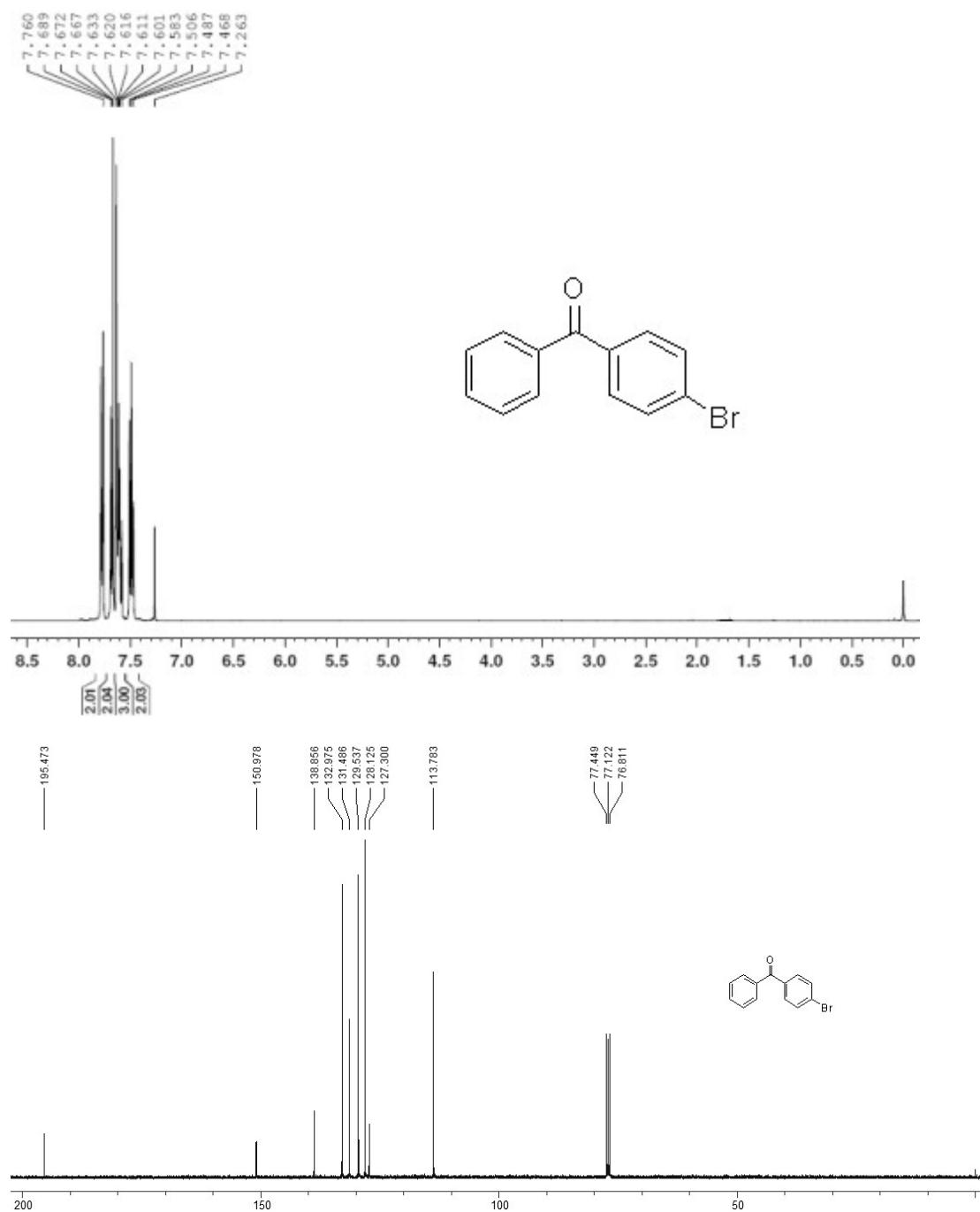
(6) 4-fluorobenzophenone (3f)

m.p. 46–48 °C (lit.³ mp 45–47 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.86 (dd, *J*₁ = 8.4 Hz, *J*₂ = 5.2 Hz, 2 H), 7.77 (d, *J* = 7.2 Hz, 2 H), 7.58 (t, *J* = 7.2 Hz, 1 H), 7.48 (t, *J* = 7.6 Hz, 2 H), 7.16 (t, *J* = 8.0 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 195.3, 166.7 (d, *J* = 252 Hz), 137.5, 133.8, 132.7 (d, *J* = 9.0 Hz), 132.5, 129.9, 128.4, 115.6 (d, *J* = 21 Hz). HRMS (EI) Calcd for C₁₃H₉FO (M⁺) 200.0637, Found 200.0638.



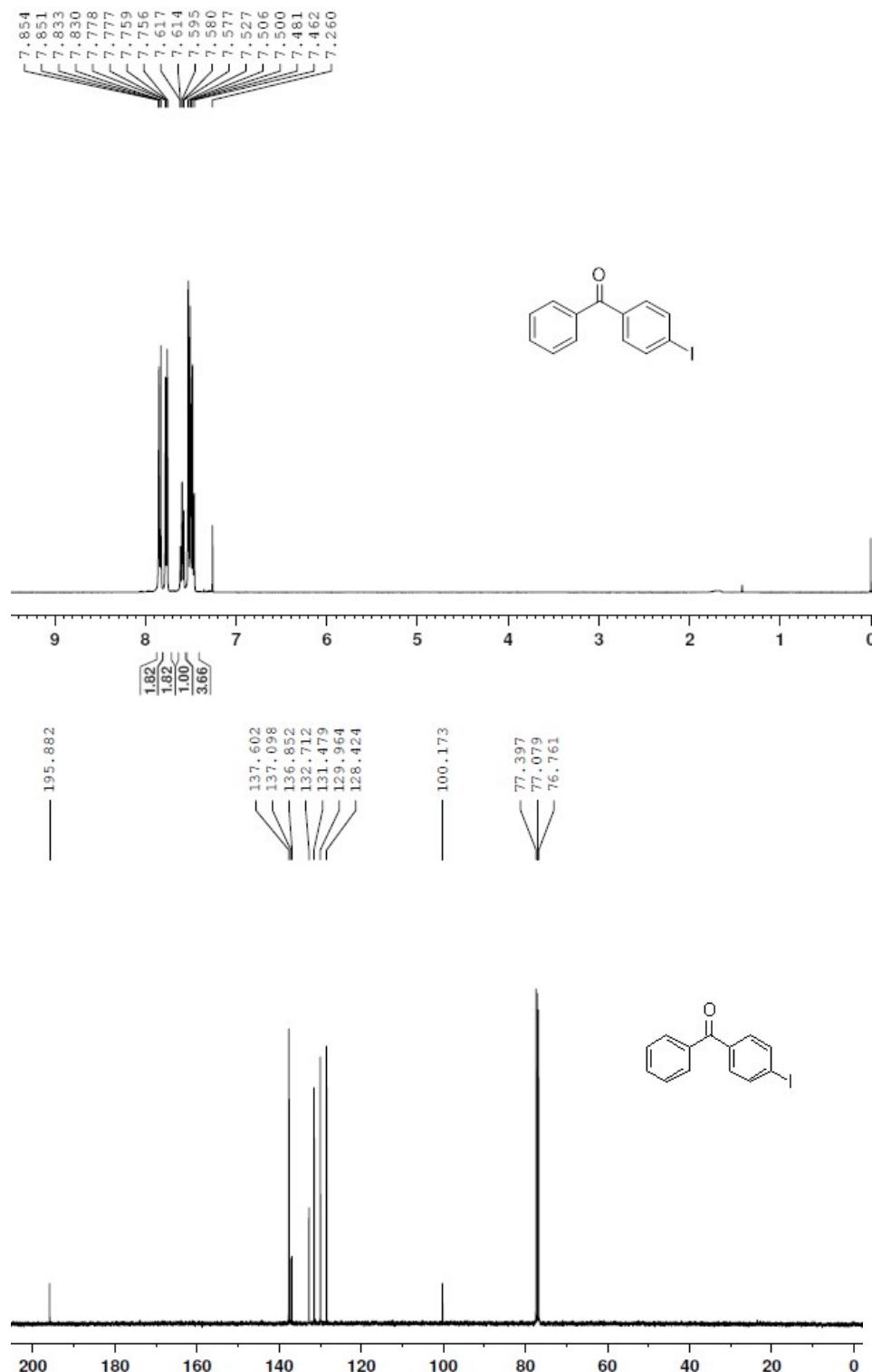
(7) 4-bromobenzophenone (3g)

m.p. 81–82 °C (lit.⁴ mp 81–83 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.77 (d, *J* = 8.4 Hz, 2 H), 7.58–7.69 (m, 5 H), 7.49 (t, *J* = 7.6 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 195.7, 137.1, 136.3, 132.7, 131.63, 131.60, 130.0, 128.4, 127.5. HRMS (EI) Calcd for C₁₃H₉BrO (M⁺) 259.9837, Found 259.9832.



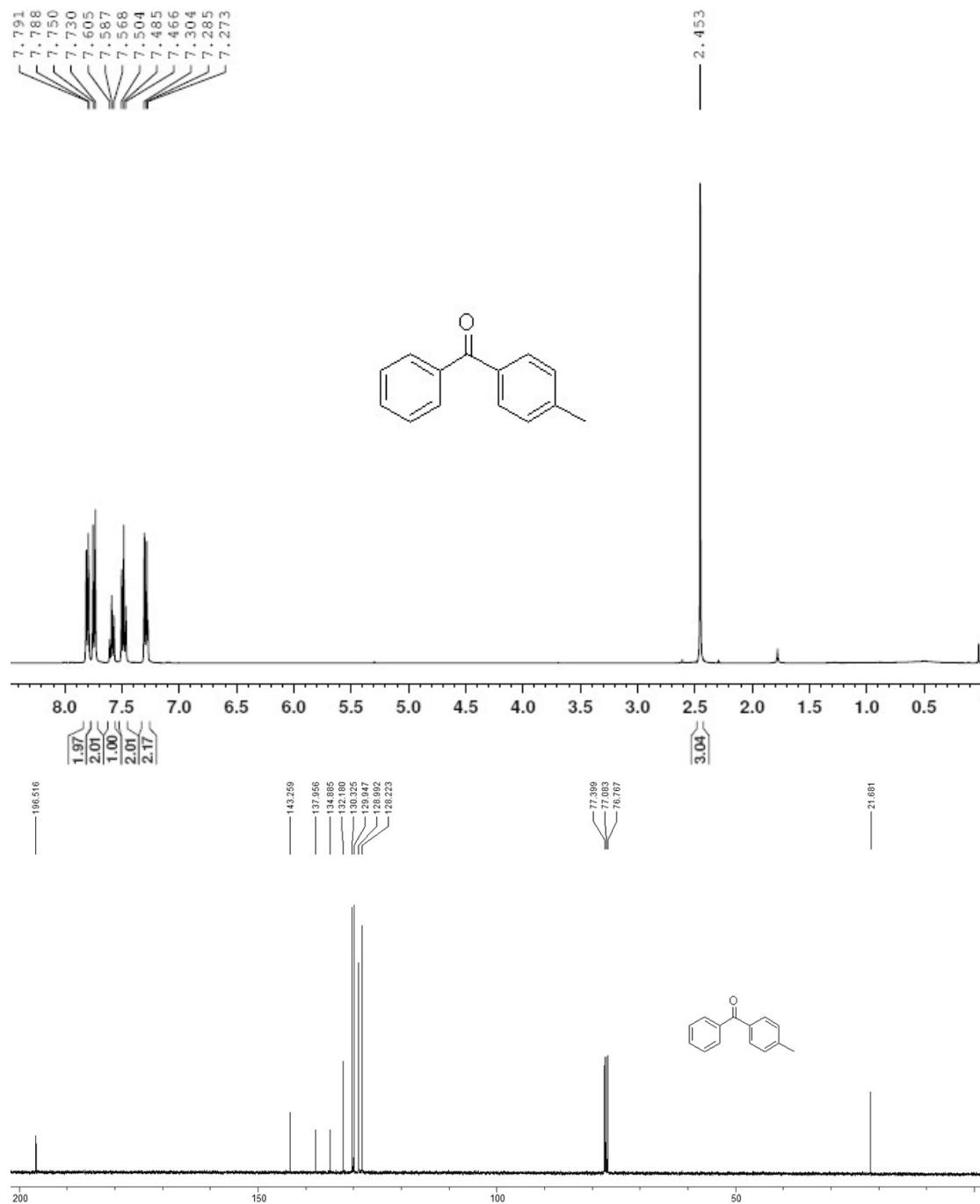
(8) 4-iodobenzophenone (3h)

m.p. 101–102 °C (lit.⁵ mp 100–101 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.84 (dd, *J*₁ = 8.4 Hz, *J*₂ = 1.2 Hz, 2 H), 7.77 (dd, *J*₁ = 8.4 Hz, *J*₂ = 1.2 Hz, 2 H), 7.60 (t, *J* = 7.2 Hz, 1 H), 7.46–7.53 (m, 3 H). ¹³C NMR (100 MHz, CDCl₃) δ 195.9, 137.6, 137.1, 136.9, 132.7, 131.5, 130.0, 128.4, 100.2. HRMS (EI) Calcd for C₁₃H₉IO (M⁺) 367.9698, Found 367.9692.



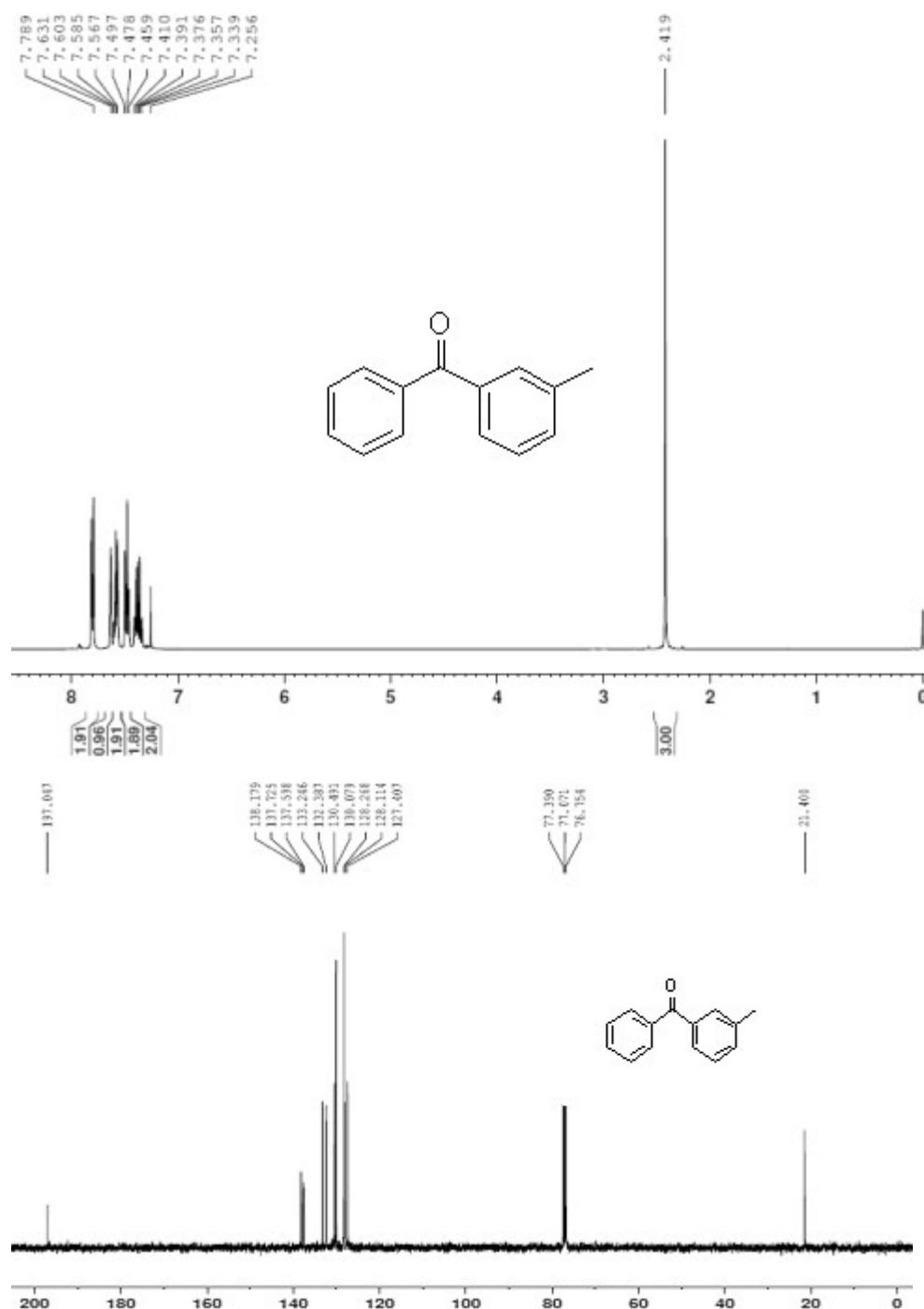
(9) 4-methylbenzophenone (3i)

m.p. 55–57 °C (lit.² mp 56–58 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.81 (d, *J* = 7.2 Hz, 2 H), 7.73 (d, *J* = 7.6 Hz, 2 H), 7.58 (t, *J* = 7.2 Hz, 1 H), 7.47 (t, *J* = 7.6 Hz, 2 H), 7.28 (d, *J* = 8.0 Hz, 2 H), 2.44 (s, 3 H). ¹³C NMR (100 MHz, CDCl₃) δ 196.5, 143.3, 138.0, 134.9, 132.2, 130.3, 129.9, 129.0, 128.2, 21.7. HRMS (EI) Calcd for C₁₄H₁₂O (M⁺) 196.0888, Found 196.0893.



(10) 3-methyl-benzophenone (3j)

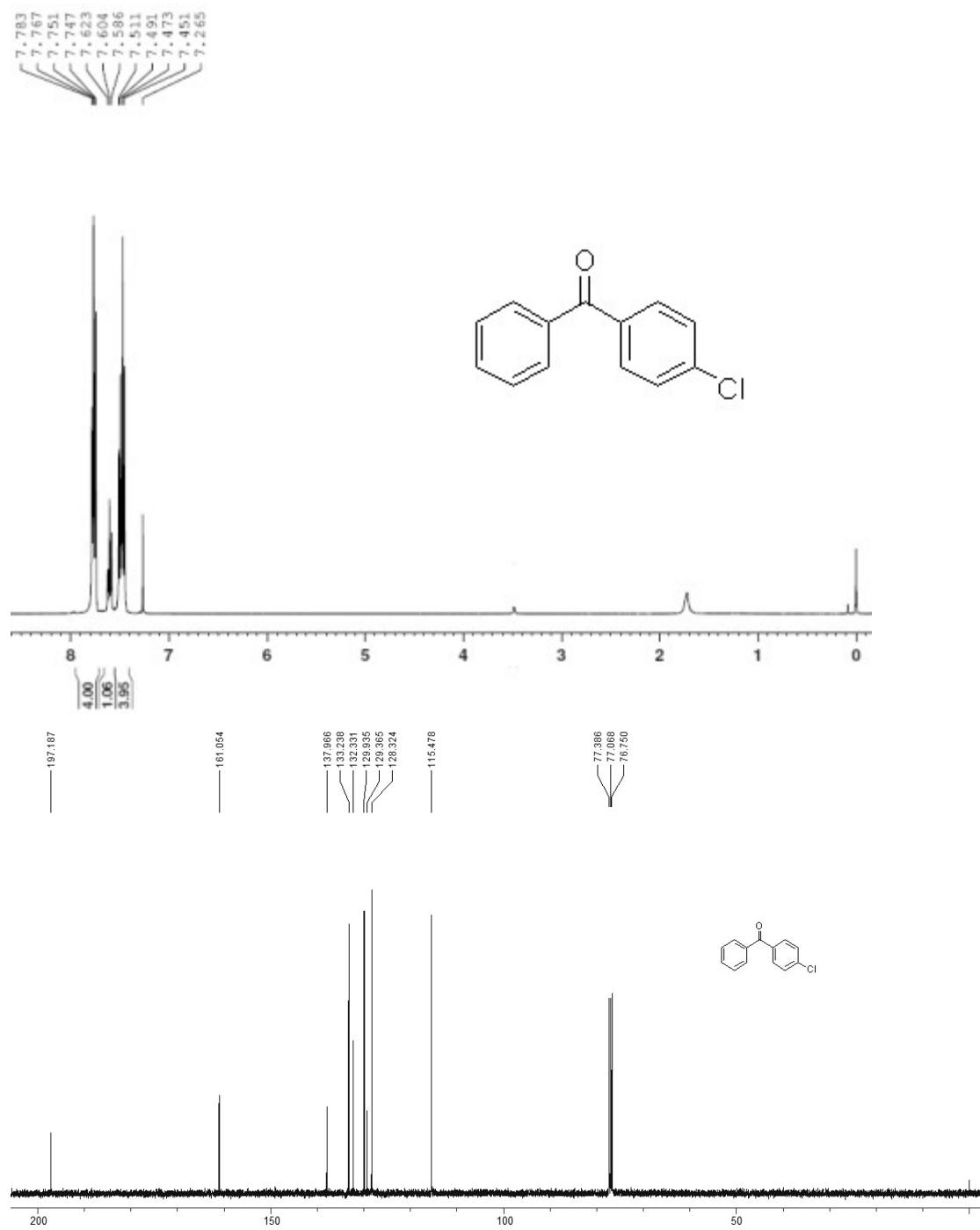
¹H NMR (400 MHz, CDCl₃, TMS) δ 7.82 (d, *J* = 6.8 Hz, 2 H), 7.64 (s, 1 H) 7.58 (d, *J* = 7.6 Hz, 2 H), 7.48 (t, *J* = 7.6 Hz, 2 H), 7.34-7.43 (m, 2 H), 2.43 (s, 3 H). ¹³C NMR (100 MHz, CDCl₃) δ 197.1, 138.2, 137.7, 137.6, 133.3, 132.4, 130.5, 130.1, 128.3, 128.1, 127.4, 21.4. HRMS (EI) Calcd for C₁₄H₁₂O (M⁺) 196.0888, Found 196.0883.



(11) 4-chloro-benzophenone (3k)

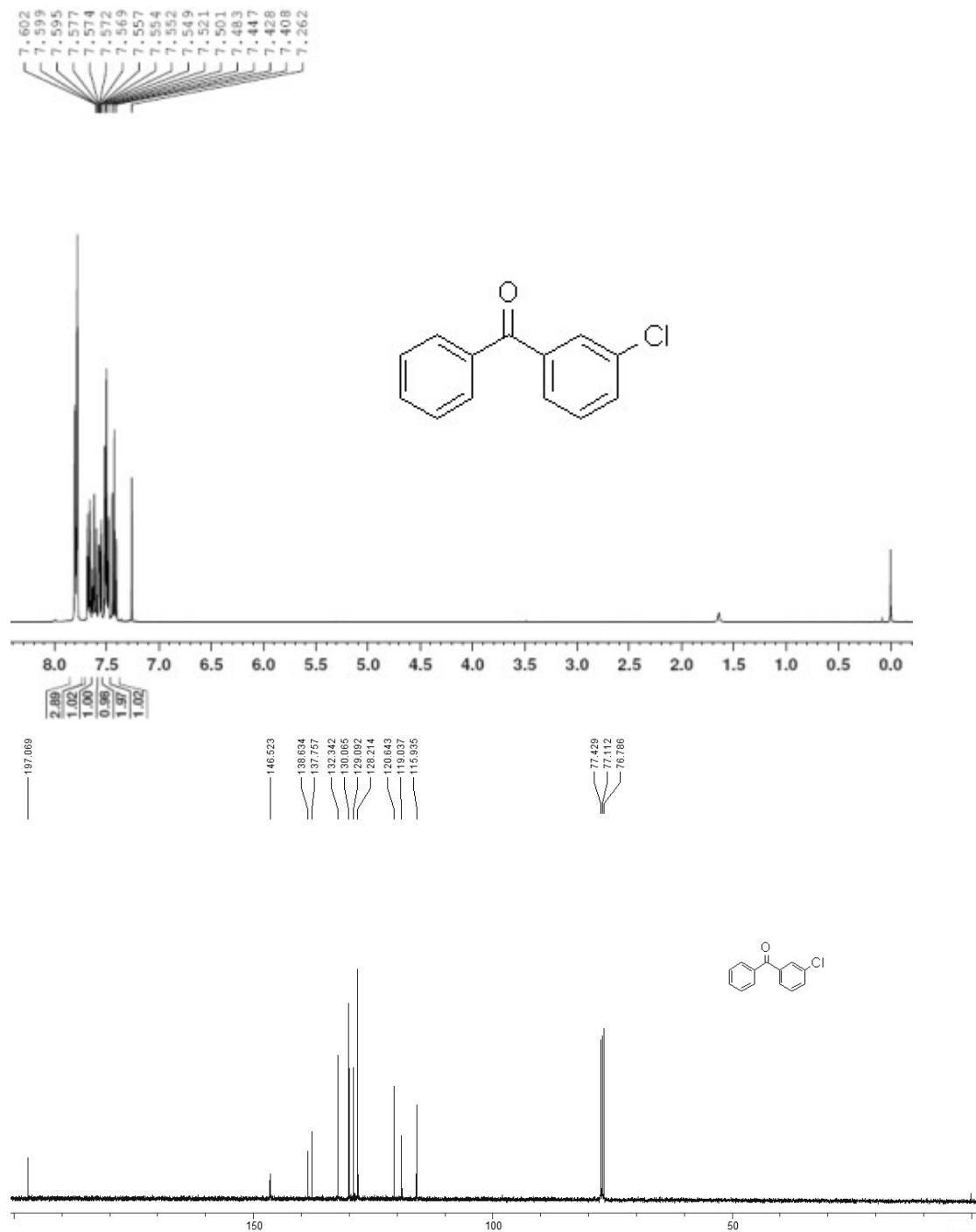
m.p. 73–75 °C (lit.¹ mp 74–75 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.81 (br, 1 H), 7.78 (d, J = 8.8 Hz, 4 H), 7.58 (t, J = 7.6 Hz, 1 H), 7.47 (t, J = 7.6 Hz, 2 H), 7.47 (d, J = 8.8 Hz, 2 H).
¹³C NMR (100 MHz, CDCl₃) δ 197.2, 161.1, 138.0, 133.2, 132.3, 129.9, 129.4, 128.3, 115.5.

HRMS (EI) Calcd for C₁₃H₉ClO (M⁺) 216.0342, Found 216.0347.



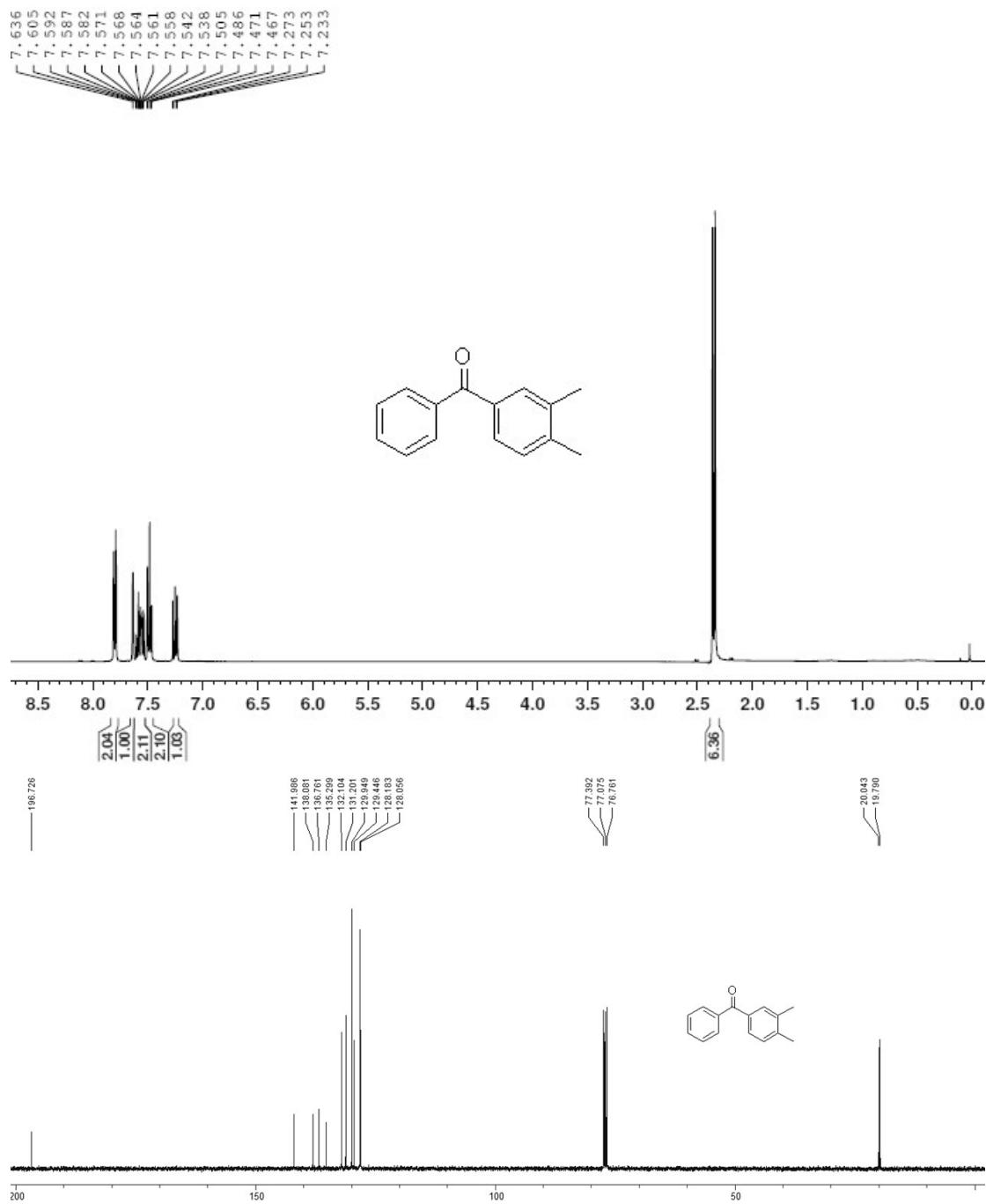
(12) 3-chloro-benzophenone (3l)

m.p. 84–85 °C (lit.⁶ mp 84 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.78 (d, *J* = 7.6 Hz, 2 H), 7.78 (s, 1 H), 7.66 (d, *J* = 6.8 Hz, 1 H), 7.62 (t, *J* = 7.6 Hz, 1 H), 7.56 (d, *J* = 8.0 Hz, 1 H), 7.50 (t, *J* = 7.6 Hz, 2 H), 7.42 (t, *J* = 8.0 Hz, 1 H). ¹³C NMR (100 MHz, CDCl₃) δ 195.3, 139.2, 136.9, 134.6, 132.9, 132.4, 130.1, 129.9, 129.7, 128.5, 128.1. HRMS (EI) Calcd for C₁₃H₉ClO (M⁺) 216.0342, Found 216.0335.



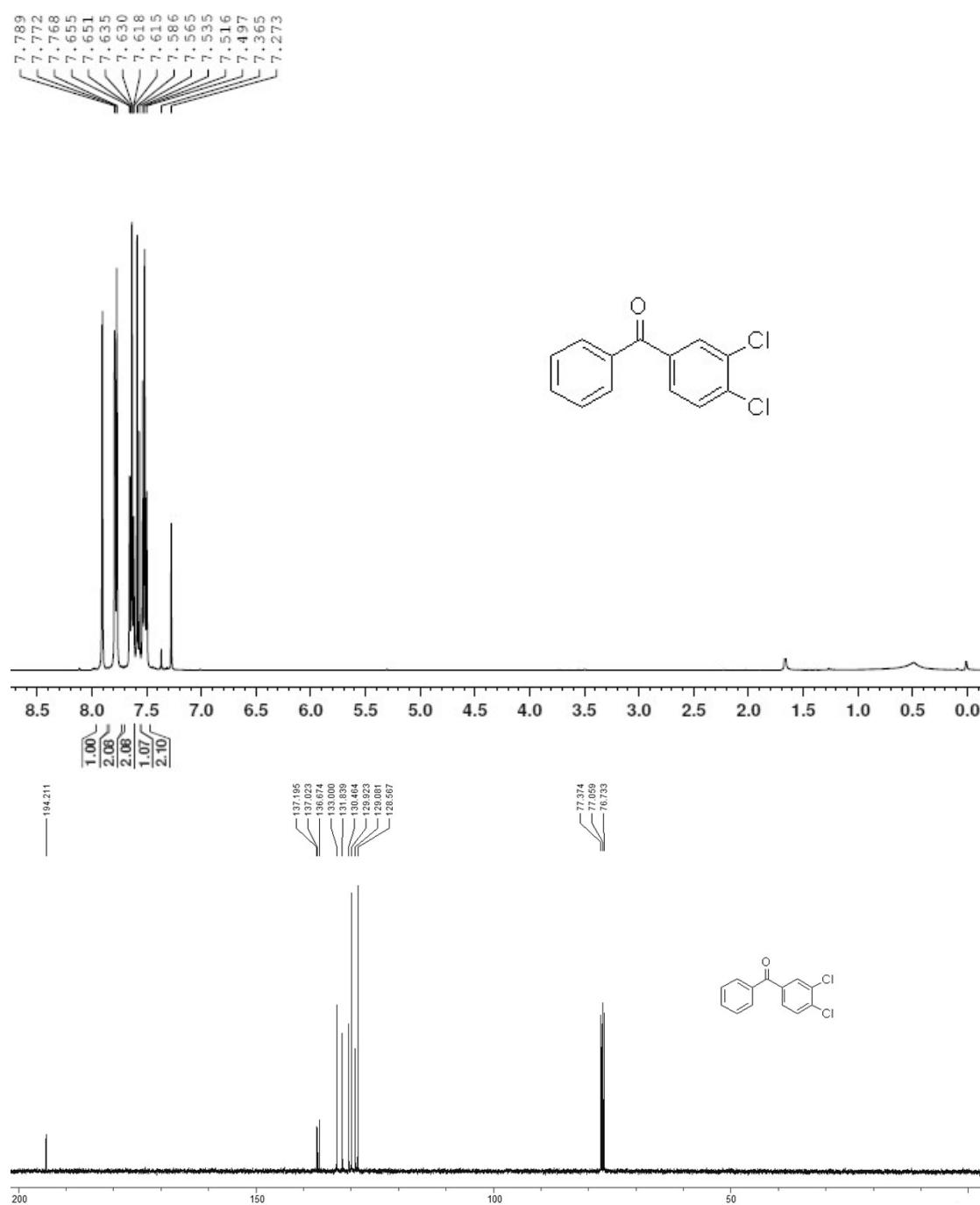
(13) 3,4-dimethylbenzophenone (3m)

m.p. 48–49 °C (lit.⁴ mp 47–49 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.83 (d, *J* = 8.0 Hz, 2 H), 7.65 (s, 1 H), 7.53–7.60 (m, 2 H), 7.49 (t, *J* = 7.6 Hz, 2 H), 7.24 (t, *J* = 8.0 Hz, 1 H), 2.37 (s, 3 H), 2.33 (s, 3 H). ¹³C NMR (100 MHz, CDCl₃) δ 196.7, 142.0, 138.1, 136.8, 135.3, 132.1, 131.2, 129.9, 129.5, 128.2, 128.1, 20.1, 19.8. HRMS (EI) Calcd for C₁₅H₁₄O (M⁺) 210.1045, Found 210.1040.



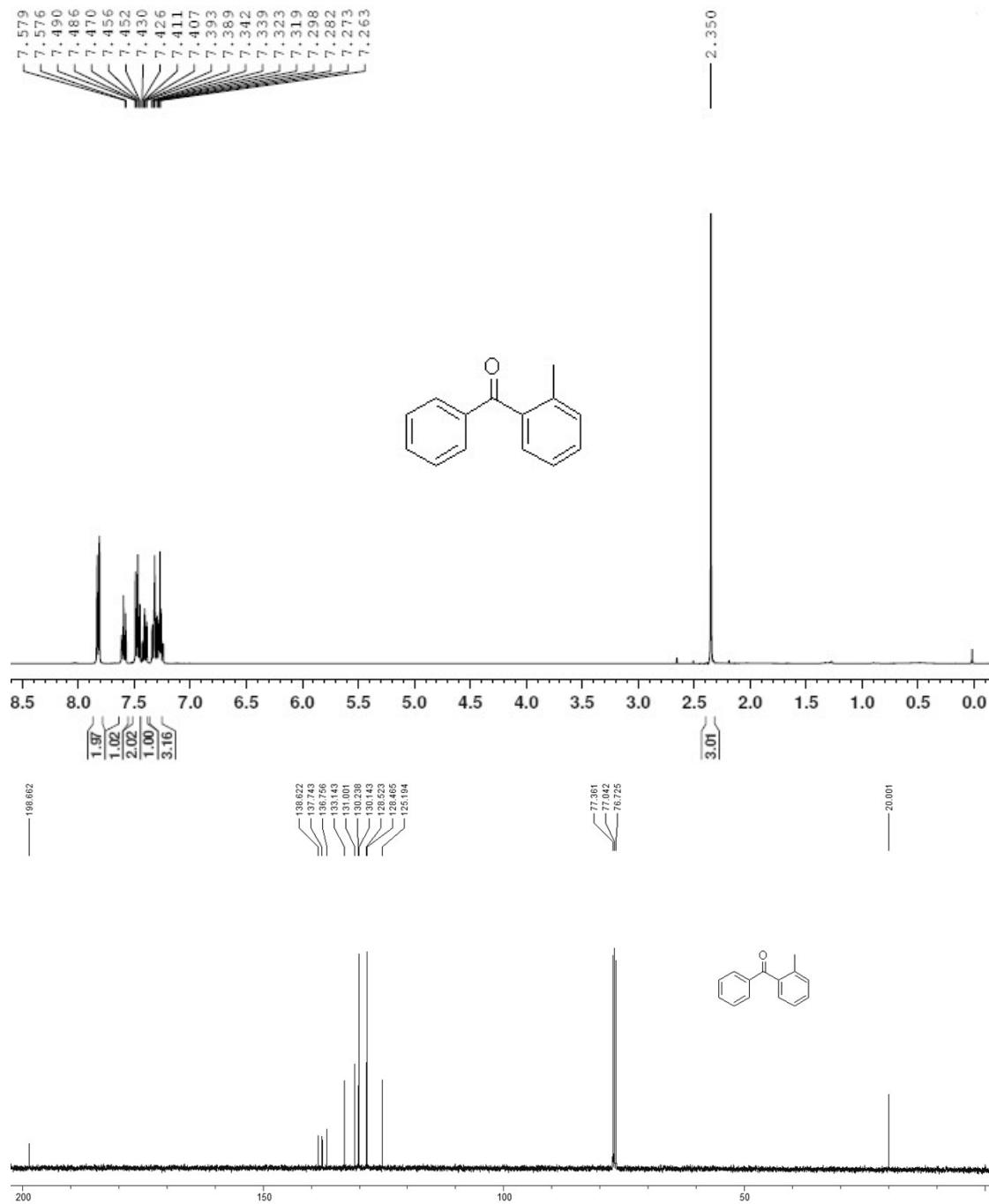
(14) 3,4-dichlorobenzophenone (3n)

m.p. 103–105 °C (lit.⁷ mp 102–103 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.92 (s, 1 H), 7.77 (d, J = 7.2 Hz, 2 H), 7.64 (t, J = 8.0 Hz, 2 H), 7.58 (d, J = 8.0 Hz, 1 H), 7.50 (t, J = 7.6 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 194.2, 137.2, 137.0, 136.7, 133.0, 131.9, 130.5, 129.9, 129.1, 128.6. HRMS (EI) Calcd for C₁₃H₈Cl₂O (M⁺) 249.9952, Found 249.9956.



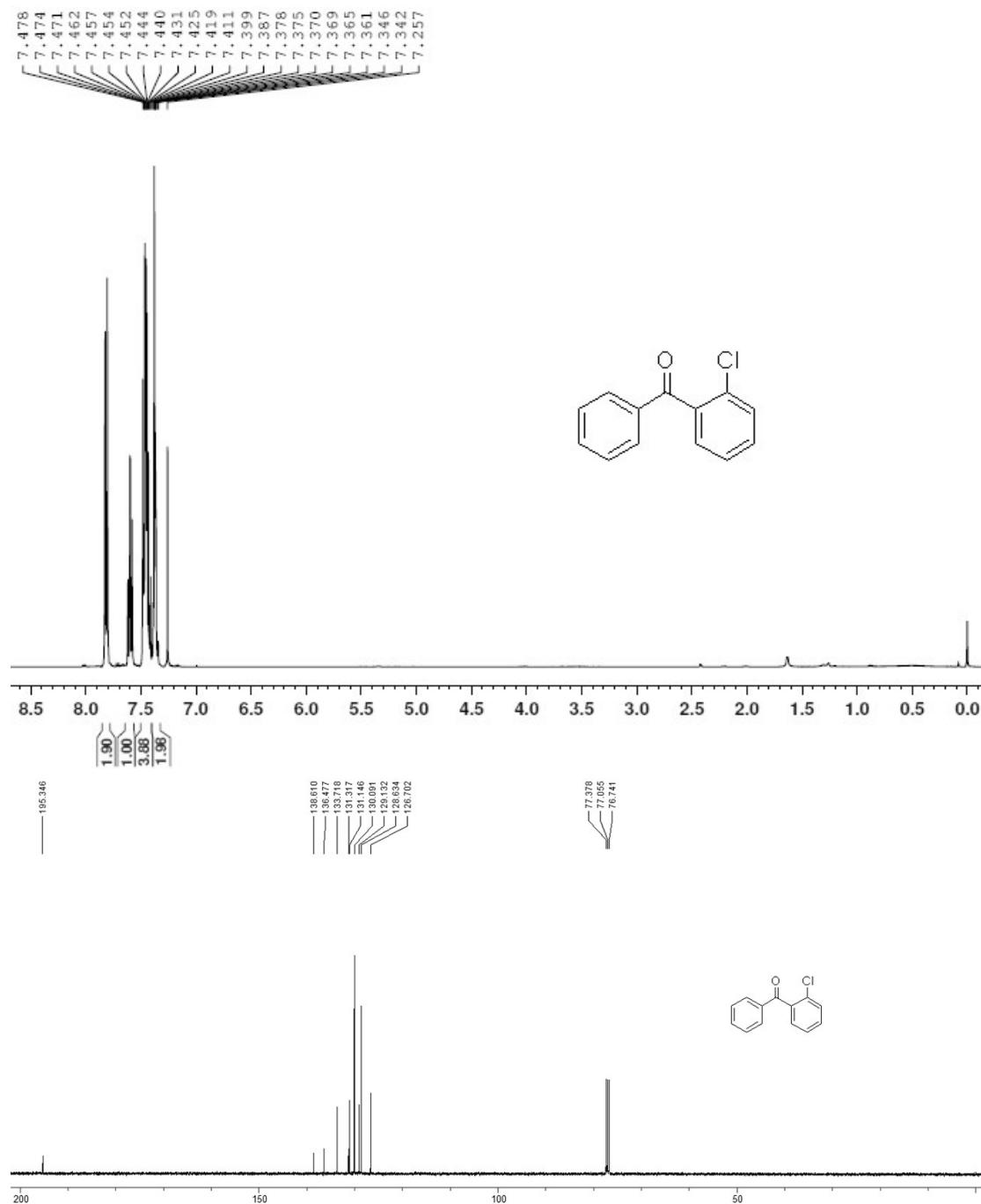
(15) 2-methylbenzophenone (3o)

¹H NMR (400 MHz, CDCl₃, TMS) δ 7.84 (d, J = 8.4 Hz, 2 H), 7.60 (t, J = 7.6 Hz, 1 H), 7.48 (t, J = 7.6 Hz, 2 H), 7.41 (t, J = 7.6 Hz, 1 H), 7.23-7.34 (m, 3 H), 2.36 (s, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 198.7, 138.6, 137.7, 136.8, 133.1, 131.0, 130.3, 130.1, 128.5, 128.5, 125.2, 20.0. HRMS (EI) Calcd for C₁₄H₁₂O (M⁺) 196.0888, Found 196.0882.



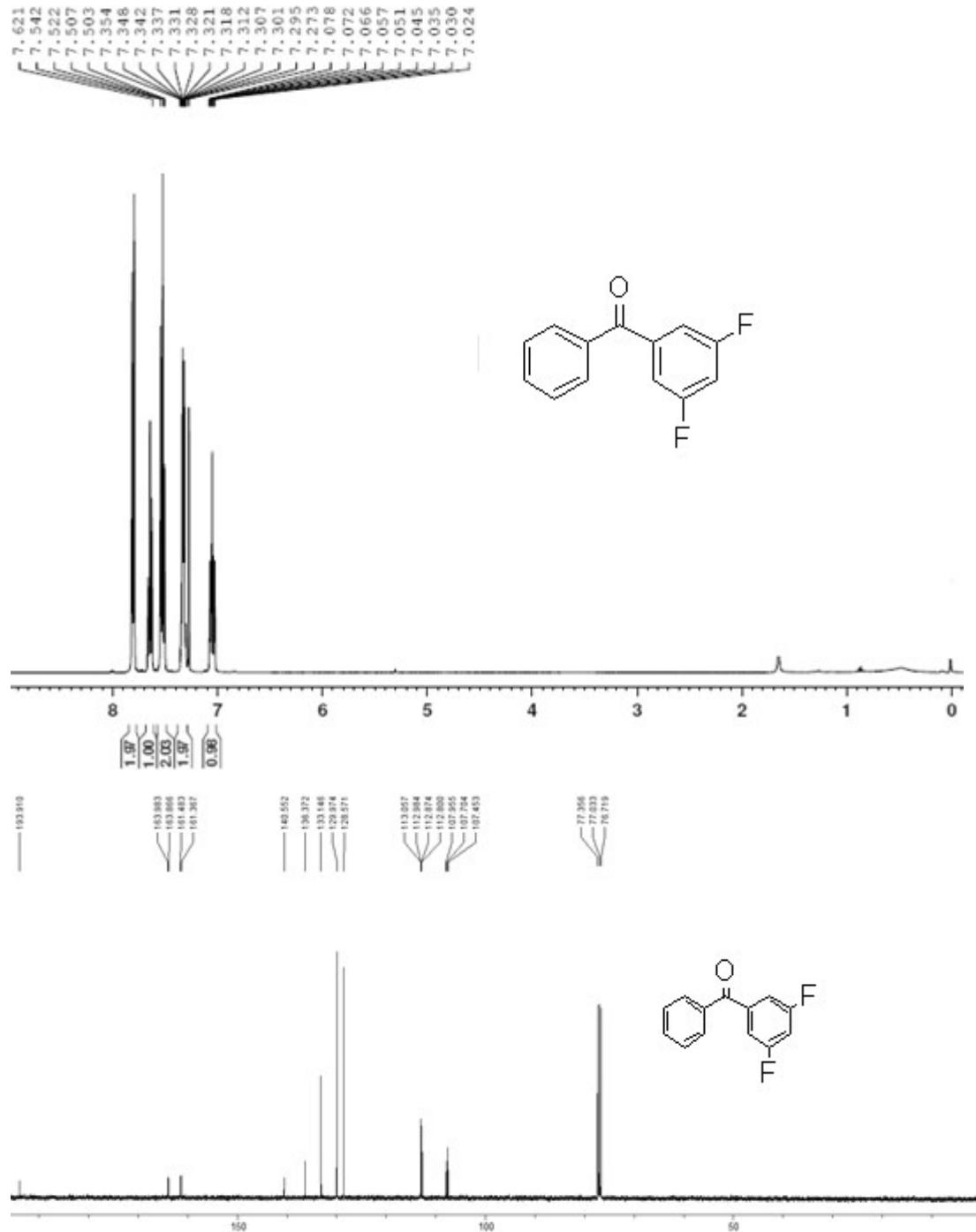
(16) 2-chlorobenzophenone (3p)

m.p. 44–45 °C (lit.¹ mp 43–45 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.81(d, *J* = 8.4 Hz, 2 H), 7.61 (t, *J* = 7.6 Hz, 1 H), 7.41-7.49 (m, 4 H), 7.35-7.39 (m, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 195.6, 138.6, 136.5, 133.7, 131.3, 131.2, 130.1, 129.1, 128.6, 126.7. HRMS (EI) Calcd for C₁₃H₉ClO (M⁺) 216.0342, Found 216.0340.



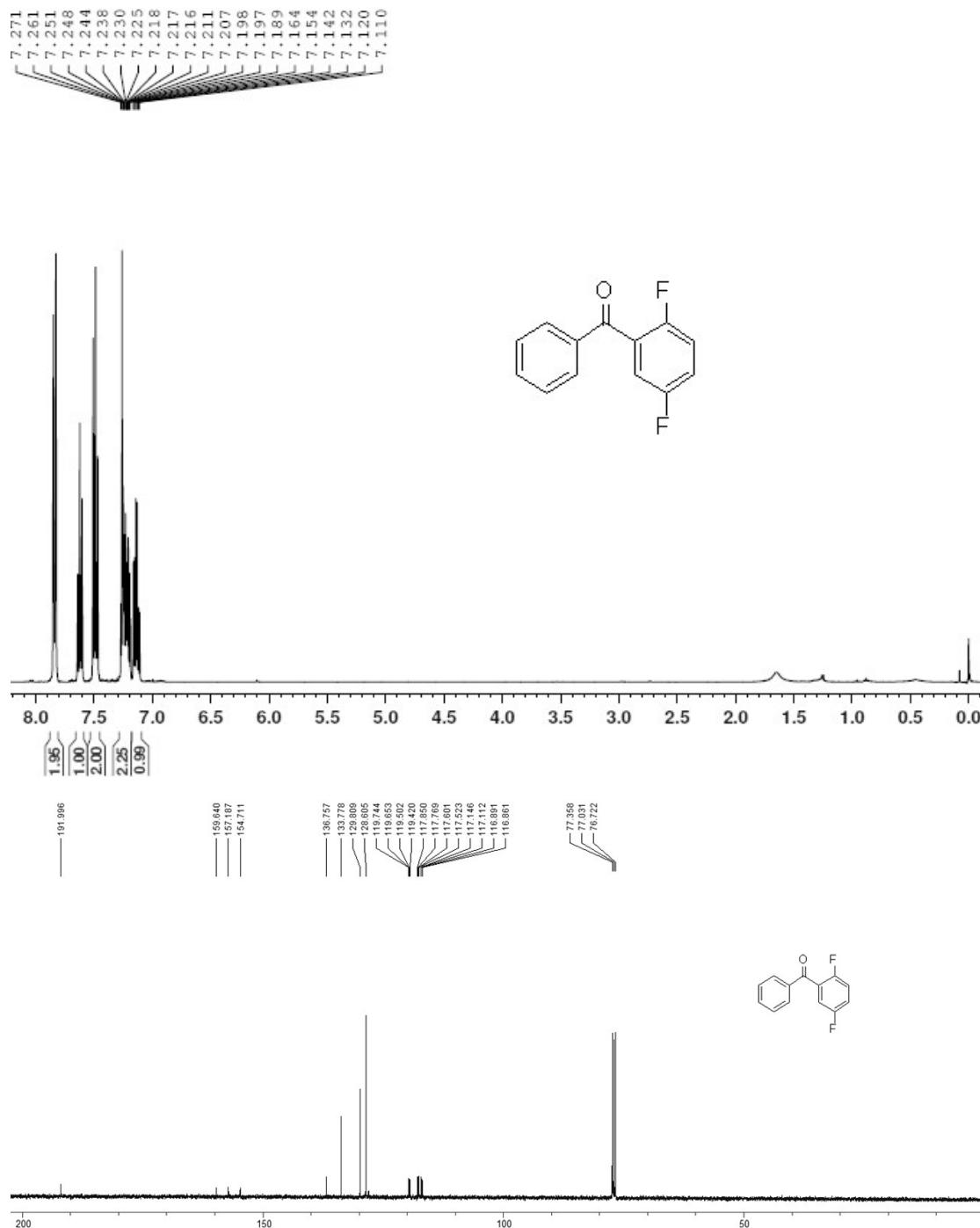
(17) 3,5-difluorobenzophenone (3q)

m.p. 58–60 °C (lit.⁸ mp 57–59 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.81 (d, *J* = 7.2 Hz, 2 H), 7.64 (t, *J* = 7.2 Hz, 1 H), 7.53 (t, *J* = 7.2 Hz, 2 H), 7.31 (dd, *J*₁ = 7.2 Hz, *J*₂ = 2.4 Hz, 2 H), 7.05 (tt, *J*₁ = 8.4 Hz, *J*₂ = 2.4 Hz, 1 H). ¹³C NMR (100 MHz, CDCl₃) δ 194.02, 163.92 (dd, *J*₁ = 250 Hz, *J*₂ = 11.7 Hz), 140.55 (t, *J* = 7.2 Hz), 136.4, 133.2, 130.0, 128.6, 112.9 (dd, *J*₁ = 18.4 Hz, *J*₂ = 7.2 Hz), 107.7 (t, *J* = 25.2 Hz). HRMS (EI) Calcd for C₁₃H₈F₂O (M⁺) 218.0543, Found 218.0549.



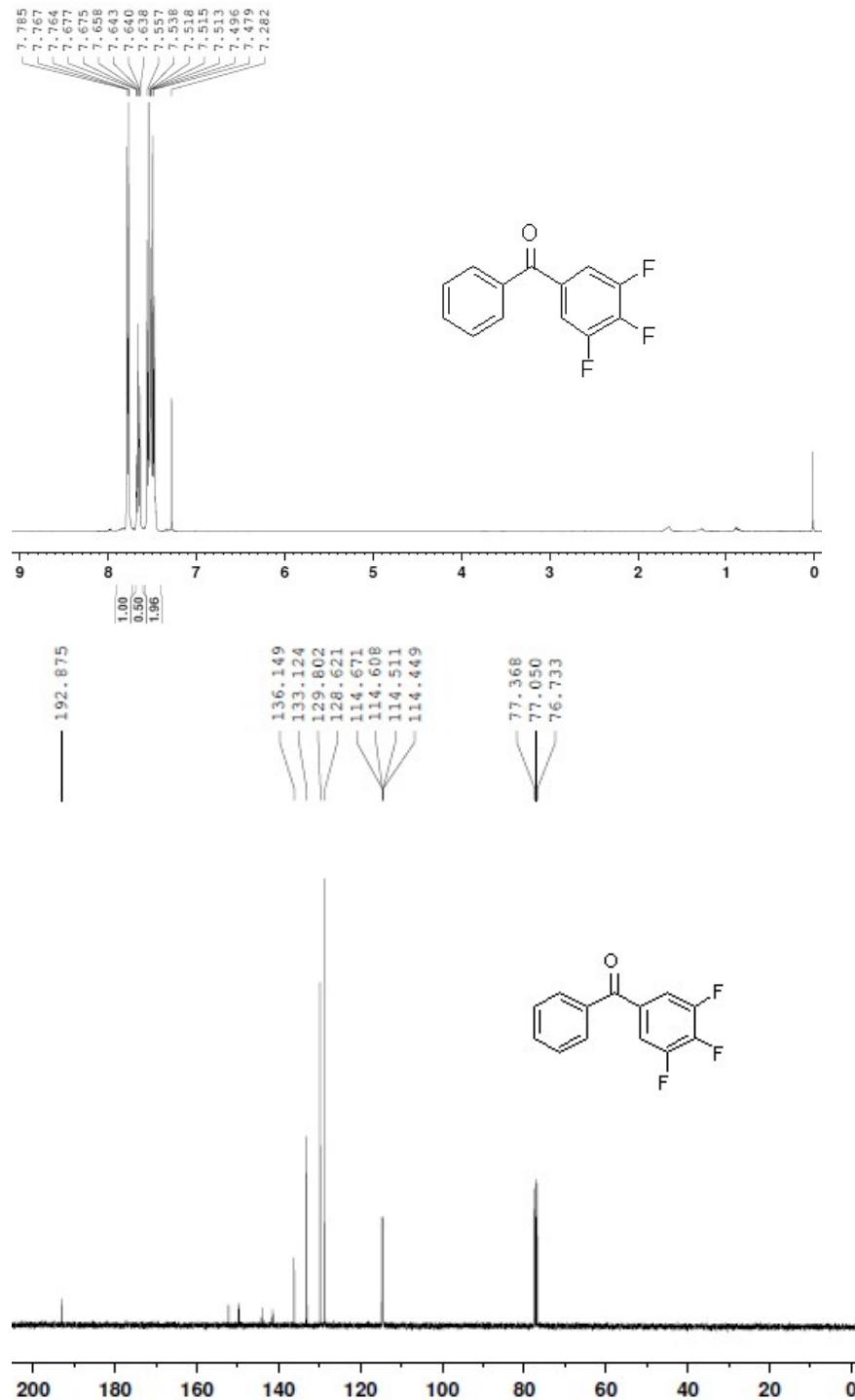
(18) 2,5-difluorobenzophenone (3r)

¹H NMR (400 MHz, CDCl₃, TMS) δ 7.83 (d, *J* = 8.4 Hz, 2 H), 7.62 (tt, *J*₁ = 7.2 Hz, *J*₂ = 1.6 Hz, 1 H), 7.48 (d, *J* = 7.6 Hz, 2 H), 7.19-7.28 (m, 2 H), 7.11-7.17 (m, 1 H). ¹³C NMR (100 MHz, CDCl₃) δ 192.0, 159.7, 157.2 (d, *J* = 252 Hz), 136.8, 133.8, 129.8, 128.6, 128.1 (dd, *J*₁ = 17.2 Hz, *J*₂ = 6.4 Hz), 119.6 (dd, *J*₁ = 23.6 Hz, *J*₂ = 8.6 Hz), 117.7 (dd, *J*₁ = 24.8 Hz, *J*₂ = 8.0 Hz), 117.0 (dd, *J*₁ = 18.4 Hz, *J*₂ = 7.2 Hz), 112.9 (dd, *J*₁ = 25.2 Hz, *J*₂ = 3.2 Hz). HRMS (EI) Calcd for C₁₃H₈F₂O (M⁺) 218.0543, Found 218.0548.



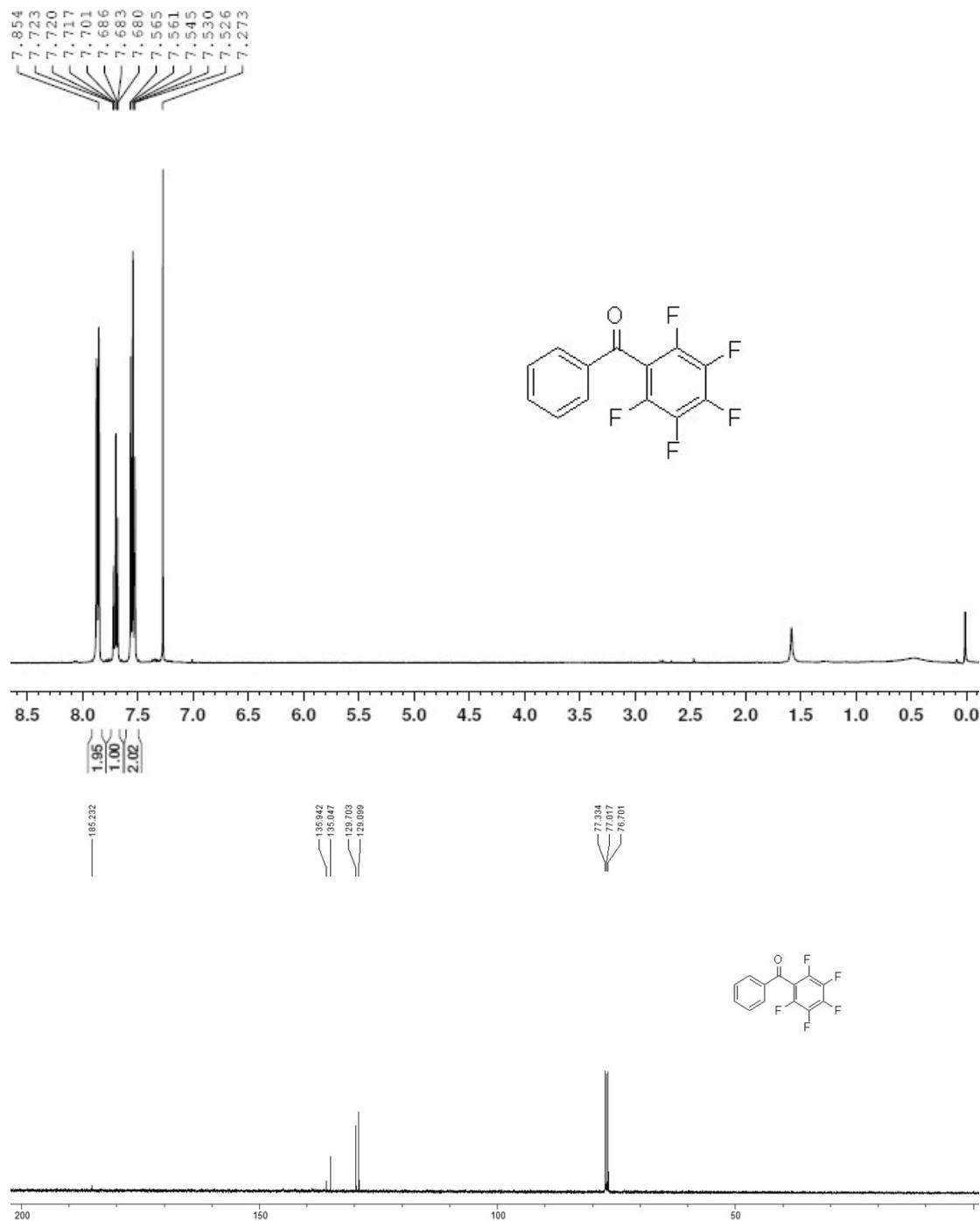
(19) 3,4,5-trifluorobenzophenone (3s)

m.p. 120–122 °C (lit.⁸ mp 119–122 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.81 (d, *J* = 8.4 Hz, 2 H), 7.66 (t, *J* = 7.6 Hz, 1 H), 7.54 (t, *J* = 8.0 Hz, 2 H), 7.50 (t, *J* = 7.2 Hz, 1 H). ¹³C NMR (100 MHz, CDCl₃) δ 192.9, 152.2 (dd, *J*₁ = 41.2 Hz, *J*₂ = 12 Hz), 149.7 (dd, *J*₁ = 40.8 Hz, *J*₂ = 14 Hz), 144.0 (t, *J* = 62.4 Hz), 141.4 (t, *J* = 61.6 Hz), 136.2, 133.1, 129.8, 128.6, 114.6 (dd, *J*₁ = 64 Hz, *J*₂ = 24.8 Hz). HRMS (EI) Calcd for C₁₃H₇F₃O (M⁺) 236.0449, Found 236.0438.



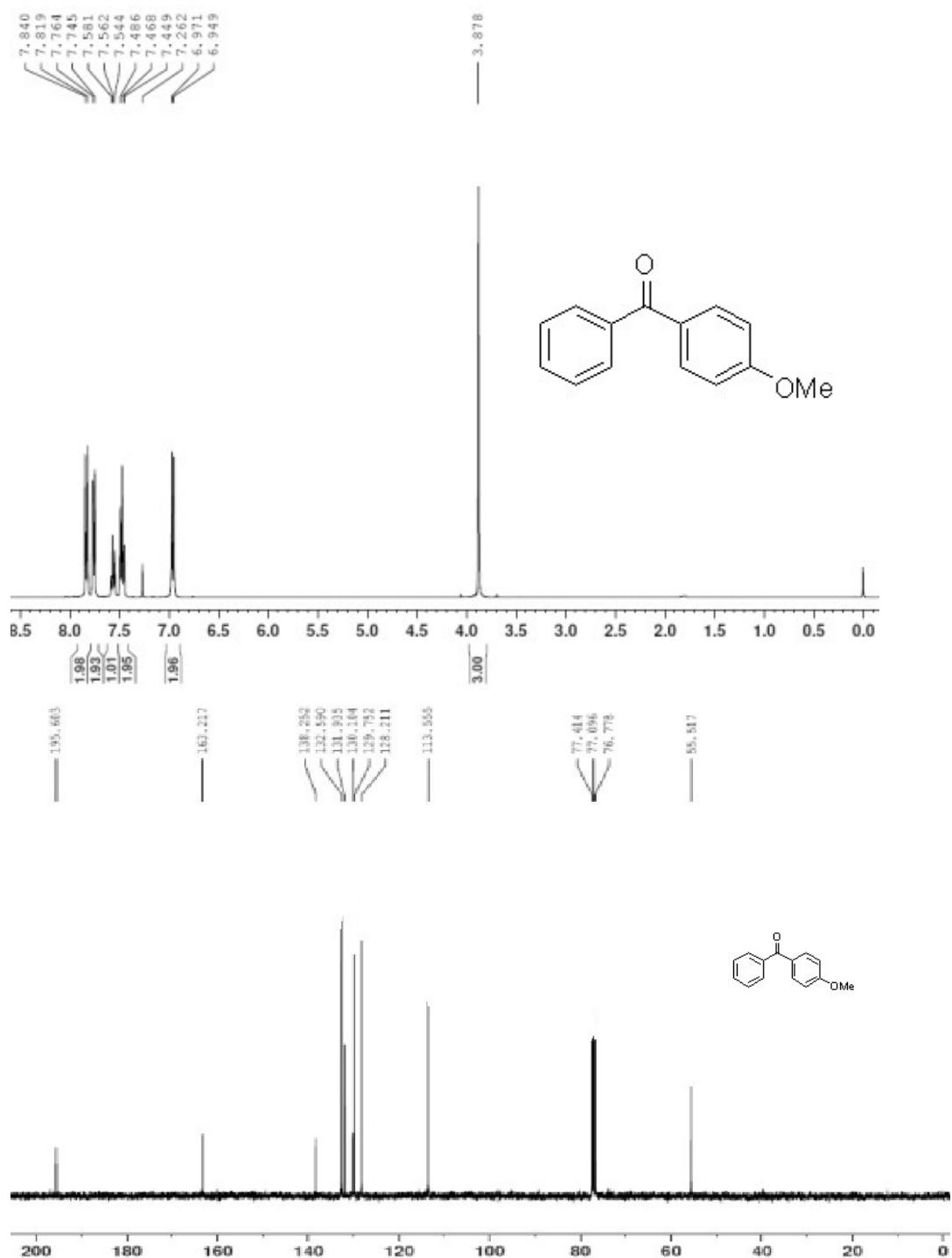
(20) 2,3,4,5,6-perfluoro-benzophenone (3t)

m.p. 35–36 °C (lit.⁹ mp 33–34 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.85 (d, *J* = 7.6 Hz, 2 H), 7.70 (t, *J* = 7.6 Hz, 1 H), 7.54 (t, *J* = 7.6 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 185.3, 135.9, 135.1, 129.7, 129.1. HRMS (EI) Calcd for C₁₃H₅F₅O (M⁺) 272.0261, Found 272.0265.



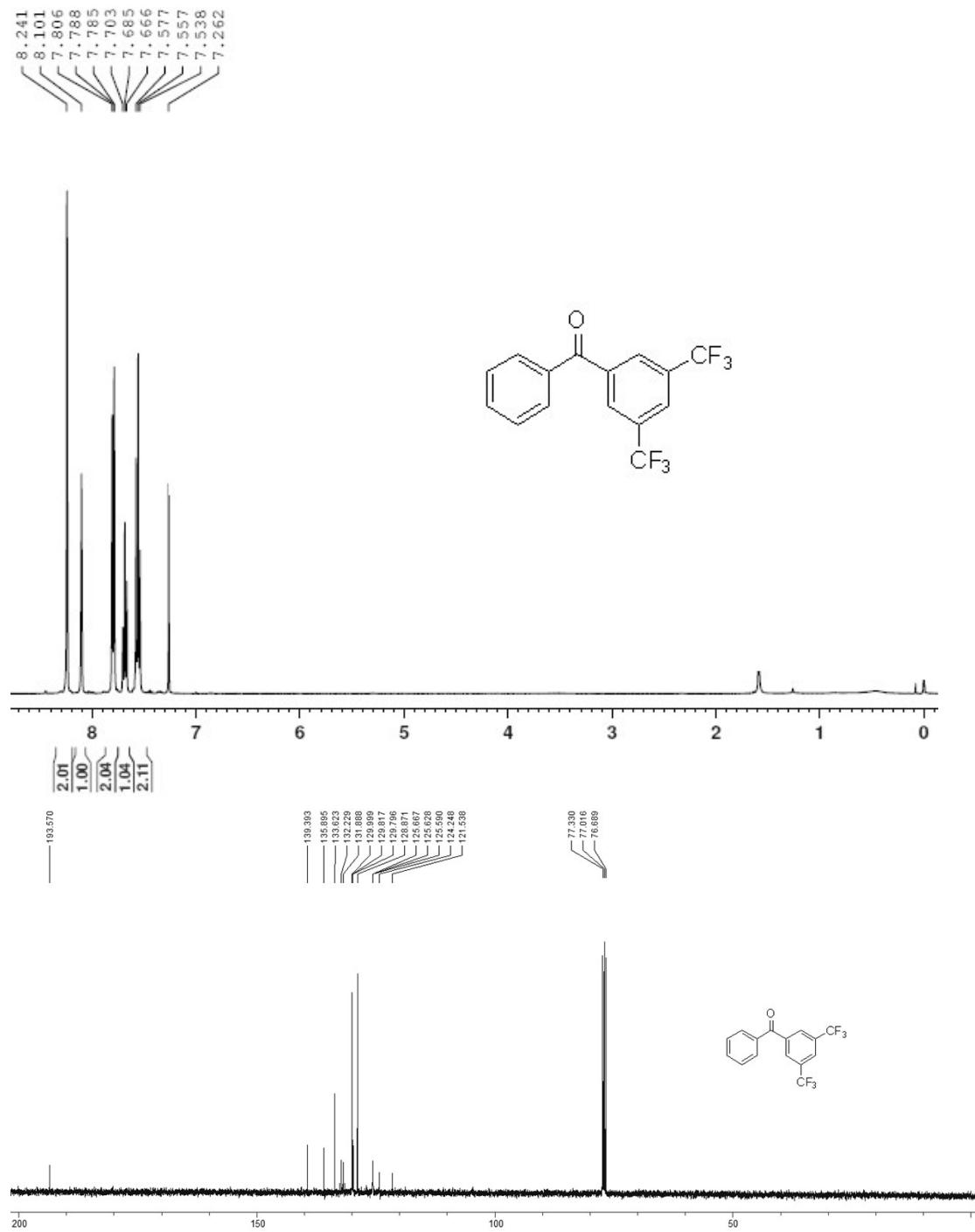
(21) 4-methoxy-benzophenone (4a)

m.p. 57–58 °C (lit.¹ mp 58–59 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.82 (d, *J* = 8.4 Hz, 2 H), 7.74 (t, *J* = 7.6 Hz, 2 H), 7.56 (t, *J* = 7.6 Hz, 1 H), 7.46 (t, *J* = 7.6 Hz, 2 H), 6.95 (d, *J* = 8.8 Hz, 2 H), 3.87 (s, 3 H). ¹³C NMR (100 MHz, CDCl₃) δ 195.6, 163.2, 138.3, 132.6, 131.9, 130.1, 129.8, 128.2, 113.6, 55.5. HRMS (EI) Calcd for C₁₄H₁₂O₂ (M⁺) 212.0837, Found 212.0843.



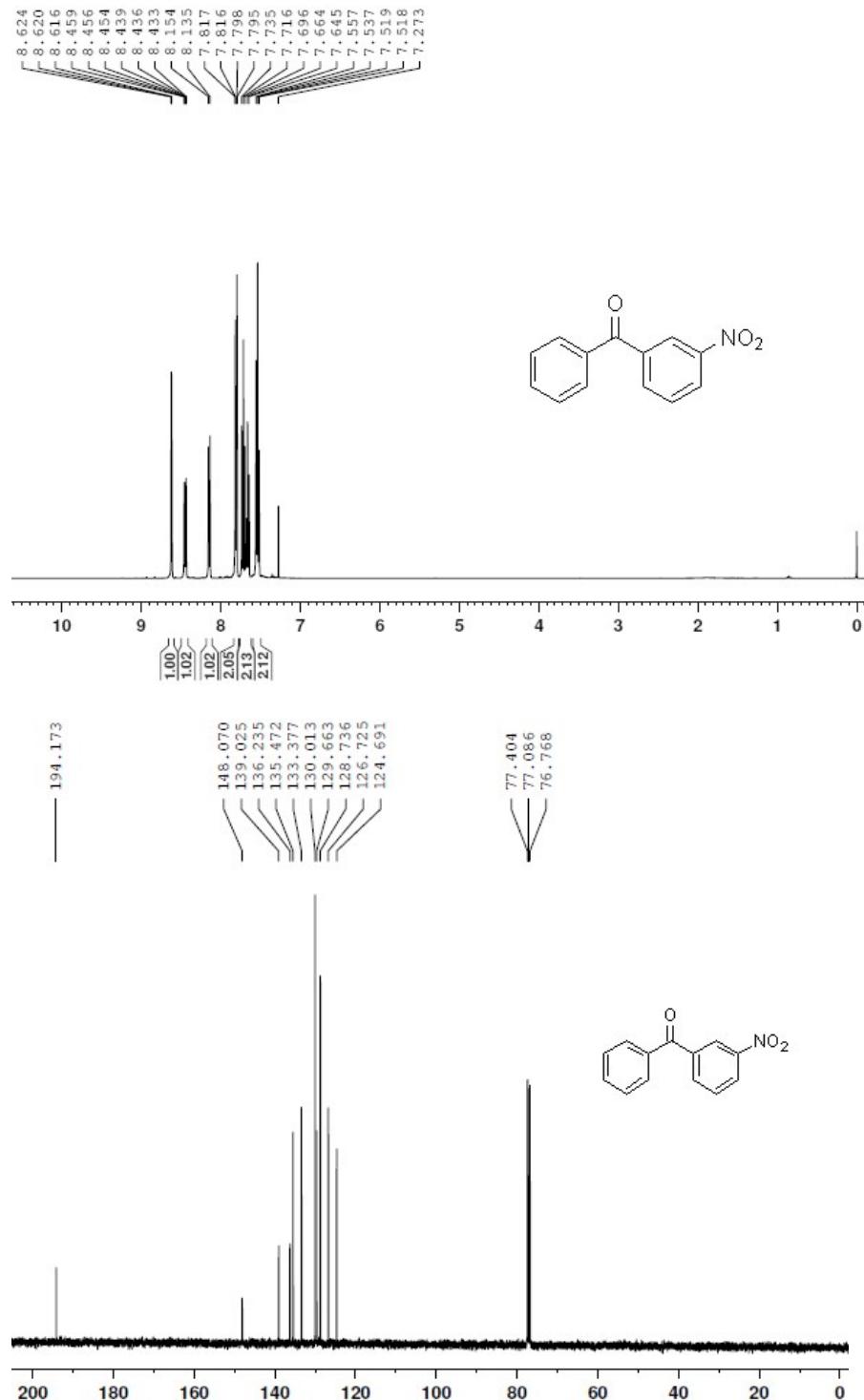
(22) 3,5-bis(trifluoromethyl)benzophenone (4e)

m.p. 109–112 °C (lit.¹⁰ mp 109–111 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.80 (d, *J* = 7.2 Hz, 2 H), 7.65 (t, *J* = 7.2 Hz, 1 H), 7.52 (t, *J* = 7.6 Hz, 2 H), 7.31 (dd, *J*₁ = 7.2 Hz, *J*₂ = 2.4 Hz, 2 H), 7.04 (tt, *J*₁ = 8.4 Hz, *J*₂ = 2.4 Hz, 1 H). ¹³C NMR (100 MHz, CDCl₃) δ 194.0, 163.9 (dd, *J*₁ = 250 Hz, *J*₂ = 11.6 Hz), 140.6 (t, *J* = 7.2 Hz), 136.4, 133.2, 129.9, 128.6, 112.9 (dd, *J*₁ = 18.4 Hz, *J*₂ = 7.2 Hz), 107.7 (t, *J* = 25.2 Hz). HRMS (EI) Calcd for C₁₃H₈F₂O (M⁺) 218.0543, Found 218.0541.



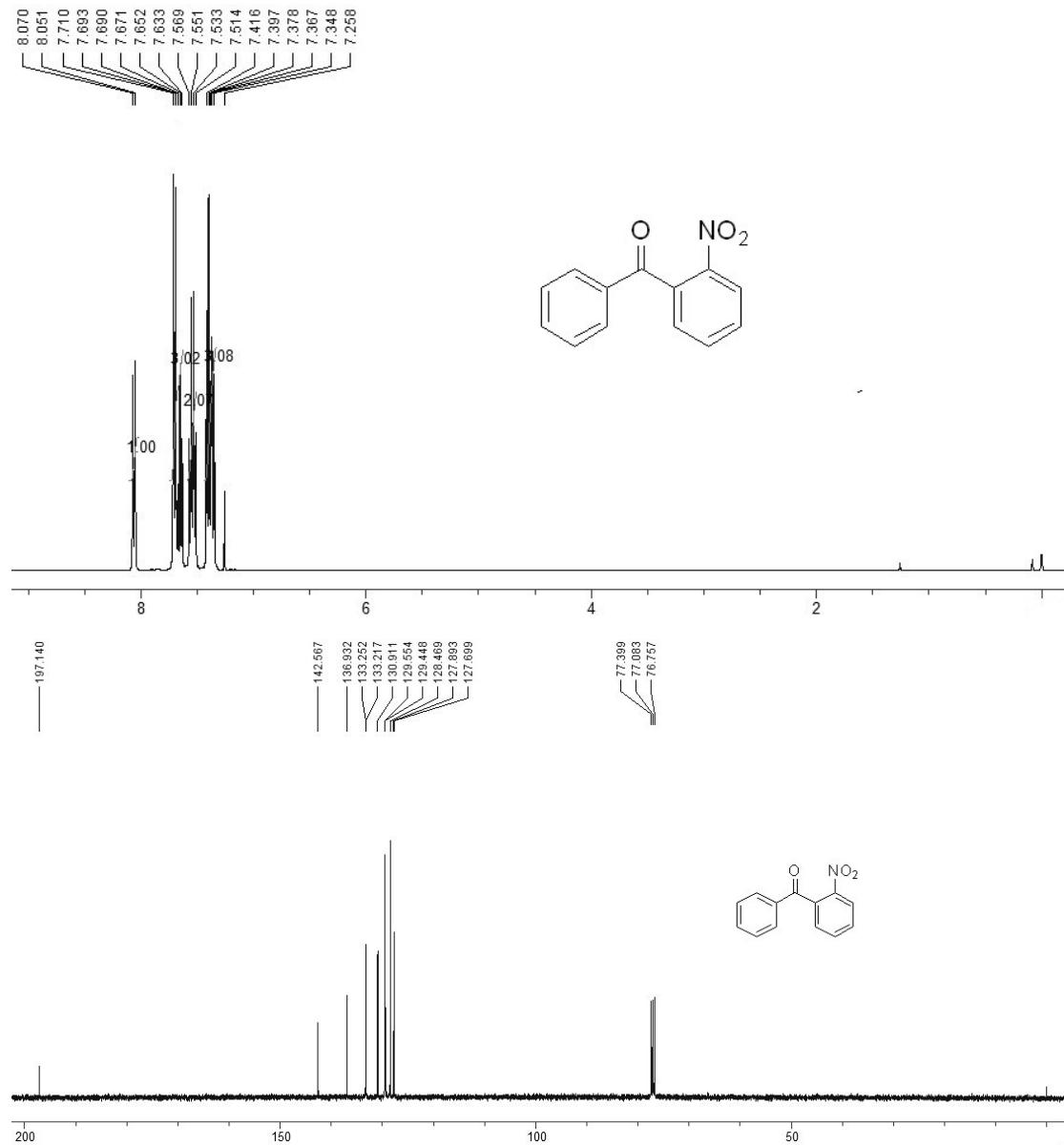
(23) 3-nitrobenzophenone (4l)

m.p. 95–97 °C (lit.¹¹ mp 95-96 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 8.62 (s, 1 H), 8.45 (d, J = 8.0 Hz, 1 H), 8.14 (d, J = 8.0 Hz, 1 H), 7.81 (d, J = 8.0 Hz, 2 H), 7.72 (t, J = 8.0 Hz, 1 H), 7.66 (t, J = 7.6 Hz, 1 H), 7.54 (t, J = 8.0 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 194.2, 148.1, 139.0, 136.2, 135.5, 135.4, 130.0, 129.7, 128.7, 126.7, 124.7. HRMS (EI) Calcd for C₁₃H₉NO₃ (M⁺) 227.0582, Found 227.0586.



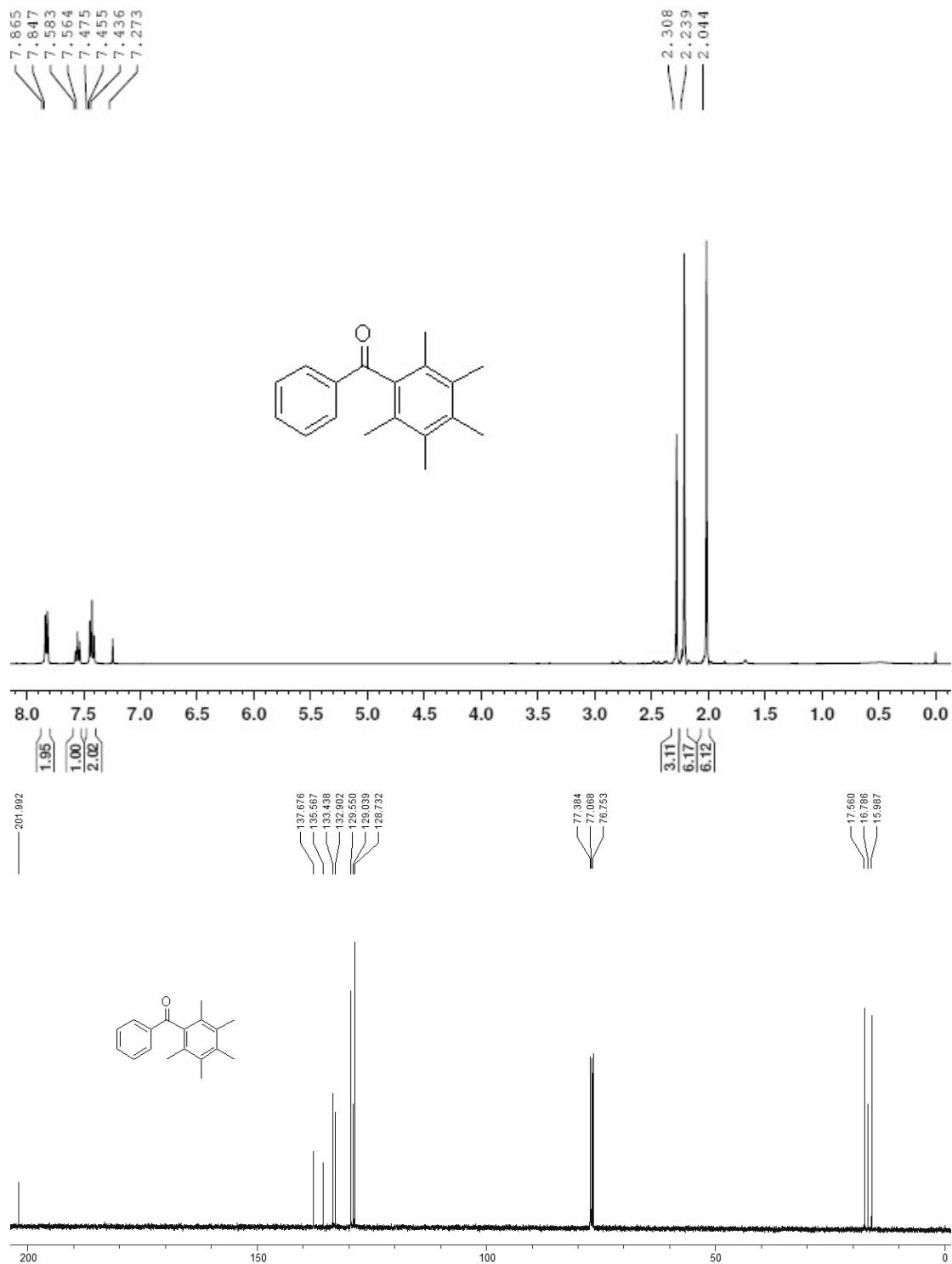
(24) 2-nitrobenzophenone (4n)

m.p. 104–105 °C (lit.¹² mp 105–106 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 8.06 (d, *J* = 7.6 Hz, 1 H), 7.63–7.71 (m, 3 H), 7.54 (q, *J* = 7.2 Hz, 2 H), 7.34–7.42 (m, 3 H). ¹³C NMR (100 MHz, CDCl₃) δ 197.1, 142.6, 136.9, 133.3, 133.2 130.9, 129.6, 129.4, 128.5, 127.9, 127.7. HRMS (EI) Calcd for C₁₃H₉NO₃ (M⁺) 227.0582, Found 227.0589.



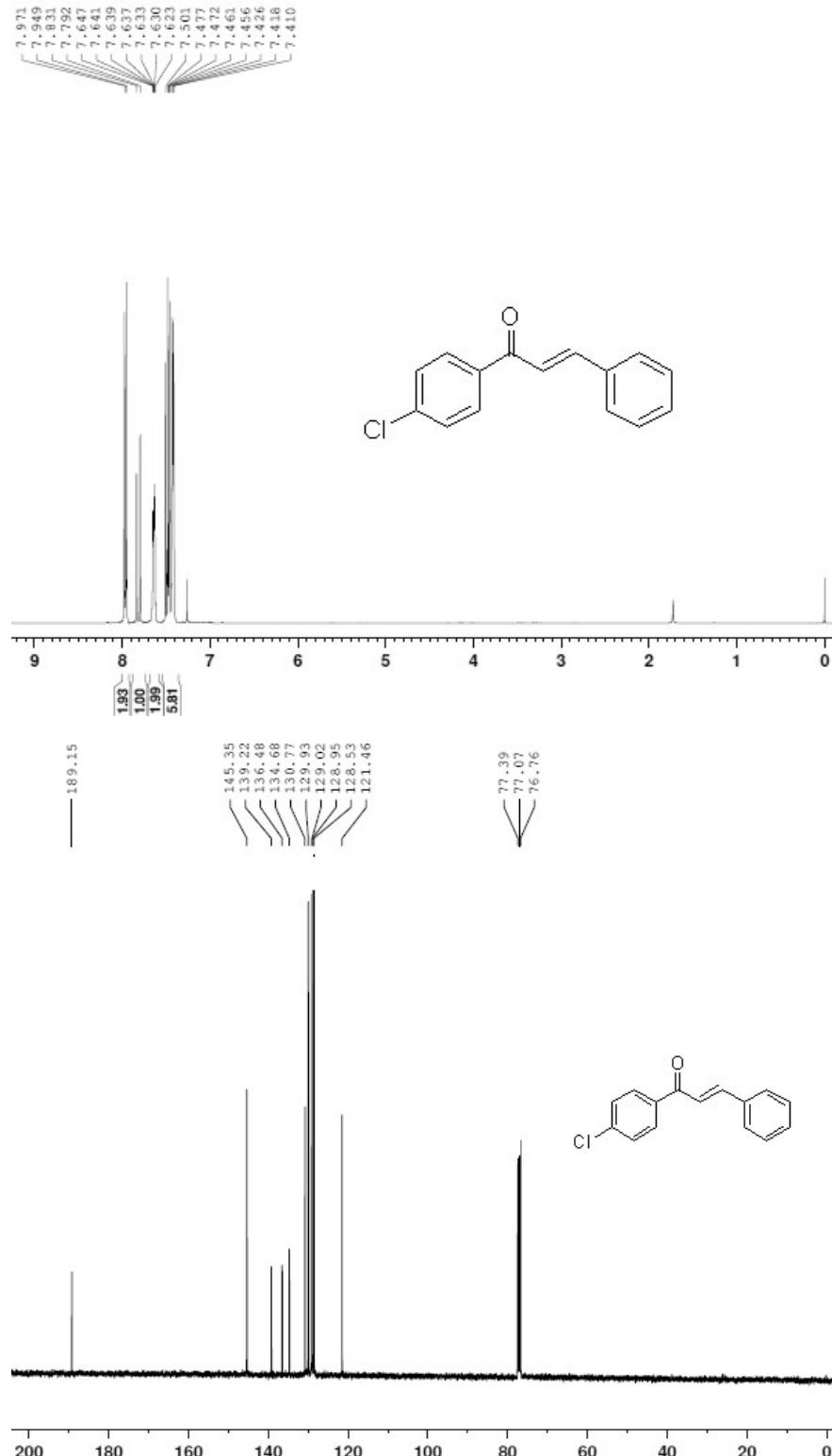
(25) 2,3,4,5,6-pentamethyl-benzophenone (4o)

m.p. 135–137 °C (lit.¹³ mp 135–136 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.82 (d, *J* = 7.2 Hz, 2 H), 7.55 (t, *J* = 7.2 Hz, 1 H), 7.43 (t, *J* = 7.6 Hz, 2 H), 2.28 (s, 3 H), 2.21 (s, 6 H), 2.02 (s, 6 H). ¹³C NMR (100 MHz, CDCl₃) δ 202.0, 137.7, 135.6, 133.4, 132.9, 129.6, 129.0, 128.7, 17.6, 16.8, 15.9. HRMS (EI) Calcd for C₁₈H₂₀O (M⁺) 252.1514, Found 252.1516.



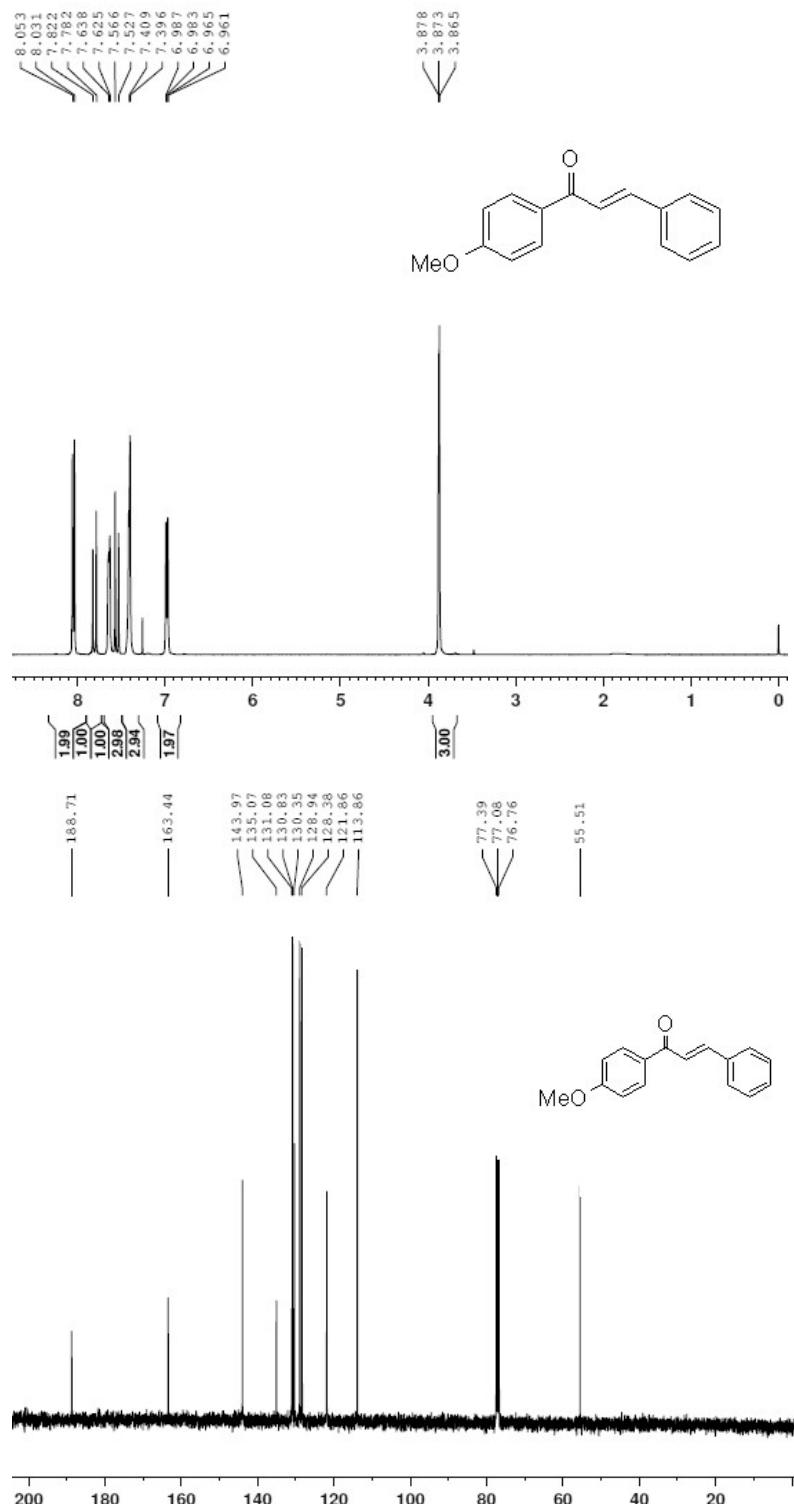
(26) (E)-1-(4-chlorophenyl)-3-phenylprop-2-en-1-one

m.p. 94-95 °C (lit.¹⁴ mp 95 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 7.96 (d, *J* = 8.8 Hz, 2 H), 7.81 (d, *J* = 16.0 Hz, 1 H), 7.62-7.65 (m, 2 H), 7.48 (d, *J* = 16.0 Hz, 1 H), 7.46 (d, *J* = 8.8 Hz, 2 H), 7.42 (t, *J* = 3.2 Hz, 2 H). ¹³C NMR (100 MHz, CDCl₃) δ 189.2, 145.4, 139.2, 136.5, 134.7, 130.8, 129.9, 129.0, 128.5, 121.5. HRMS (EI) Calcd for C₁₅H₁₁ClO (M⁺) 242.0498, Found 242.0491.



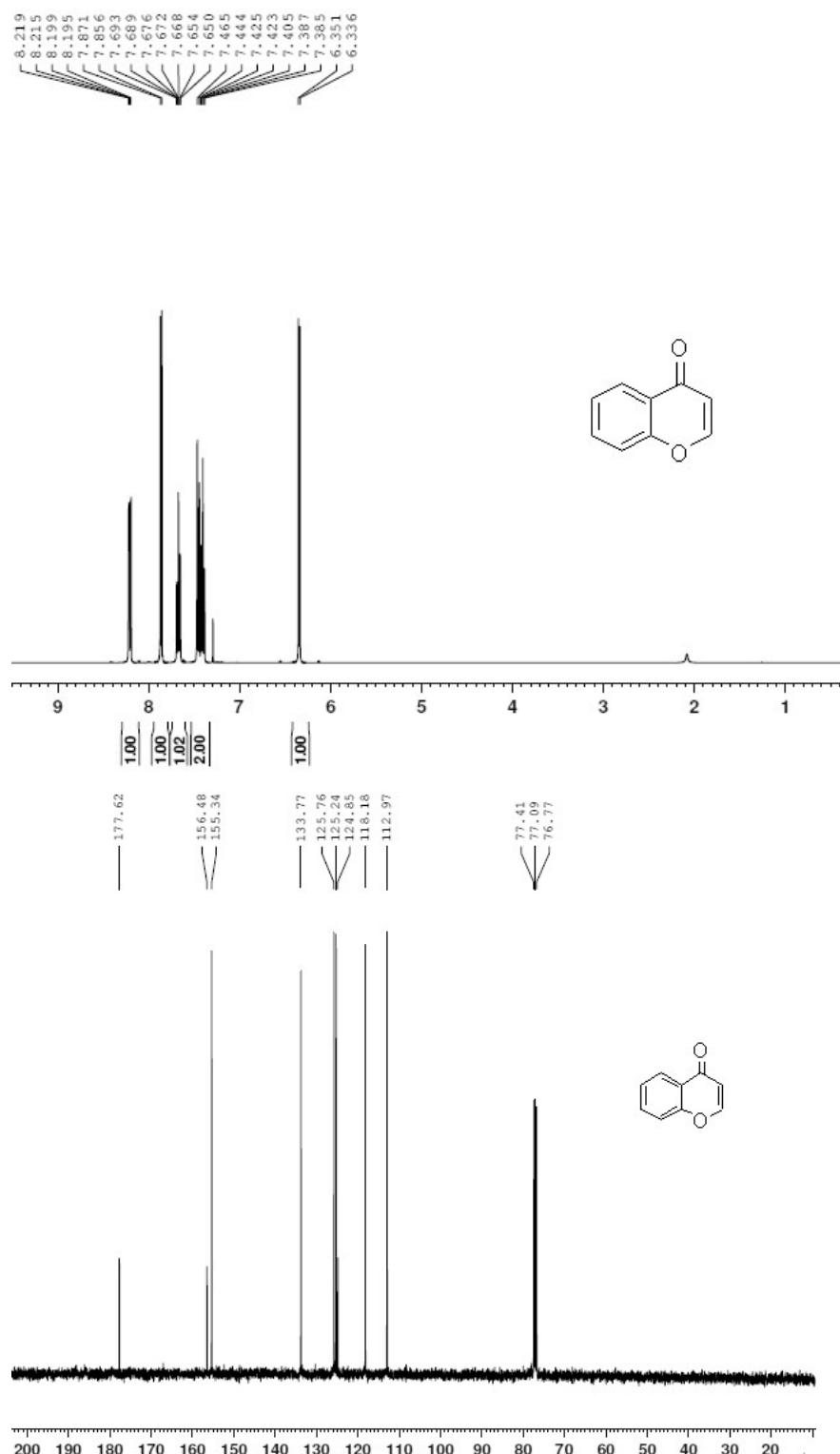
(27)(E)-1-(4-methoxyphenyl)-3-phenylprop-2-en-1-one

m.p. 104-106 °C (lit.¹⁵ mp 104-105 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 8.04 (d, *J* = 8.8 Hz, 2 H), 7.80 (d, *J* = 15.6 Hz, 1 H), 7.62-7.64 (m, 2 H), 7.54 (d, *J* = 15.6 Hz, 1 H), 7.39-7.41 (m, 3 H), 6.97 (dd, *J*₁ = 8.8 Hz, *J*₂ = 1.6 Hz, 2 H), 3.87 (s, 3 H). ¹³C NMR (100 MHz, CDCl₃) δ 188.7, 163.5, 144.0, 135.1, 131.1, 130.8, 130.4, 128.9, 128.4, 121.9, 113.9, 55.5. HRMS (EI) Calcd for C₁₆H₁₄O₂(M⁺) 238.0994, Found 238.0999.



(28) 4H-chromen-4-one

m.p. 59-60 °C (lit.¹⁶ mp 58-60 °C); ¹H NMR (400 MHz, CDCl₃, TMS) δ 8.21 (dd, *J*₁= 8.0 Hz, *J*₂= 1.6 Hz, 1 H), 7.86 (d, *J*= 6.0 Hz, 1 H), 8.21 (dt, *J*₁= 7.6 Hz, *J*₂= 1.6 Hz, 1 H), 7.45 (d, *J*= 8.8 Hz, 1 H), 7.41 (t, *J*= 8.0 Hz, 1 H), 6.34 (d, *J*= 6.0 Hz, 1 H). ¹³C NMR (100 MHz, CDCl₃) δ 177.6, 156.5, 155.3, 133.8, 125.8, 125.2, 124.9, 116.2, 113.0. HRMS (EI) Calcd for C₉H₆O₂(M⁺) 146.0348, Found 146.0355.



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