

Supporting information

Transition-metal-free chemoselective reduction of α , β -unsaturated ketones using H₂O as hydrogen sources

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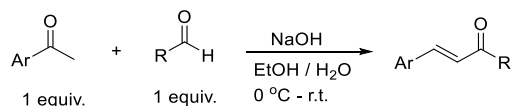
1. General information

All reactions were carried out under air atmosphere unless otherwise noted. Column chromatography was performed using silica gel (200-300 mesh). ^1H NMR and ^{13}C NMR spectra were recorded on Bruker-AV (400 and 100 MHz, respectively) instrument using CDCl_3 as solvent and TMS as an internal standard. Mass spectra were measured on Agilent 5977 GC-MS instrument (EI). The structures of known compounds were further corroborated by comparing their ^1H NMR, ^{13}C NMR data and MS data with those of literature.

2. General procedure for preparation of substrates

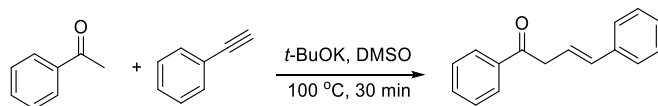
Substrates **1a**, **1ac** - **1aj** were purchased from commercial sources; **1b** – **1ab** were prepared from the corresponding aldehydes and ketones according to the literature; while **1ak** were prepared as reported in respective literature.

Preparation of the α , β -unsaturated ketones¹:



To a solution of ketone (10 mmol, 1.0 equiv.) in 6 mL ethanol was added a solution of NaOH (520 mg, 13 mmol, 1.3 equiv.) in water (10 mL), then the corresponding aldehyde (10 mmol, 1.0 equiv.) was added gradually at 0 °C. The mixture was then allowed to warm to room temperature and stirred overnight. The solid product was collected by suction filtration on a Buchner funnel and washed repeatedly with cold water. Recrystallization from ethanol or purification by silica gel chromatography for liquid products.

Preparation of **1ak**²:

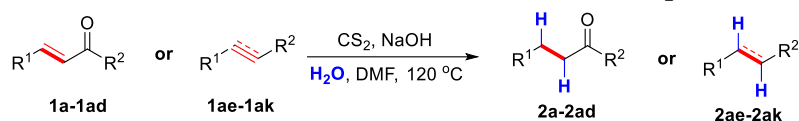


To a solution of acetophenone (2 g, 16.64 mmol, 1 equiv.) and phenyl acetylene (1.83 mL, 16.64 mmol) in DMSO (42 mL) was added *t*-BuOK (1.87 g, 16.64 mmol). The resulting solution was stirred at 100 °C for 0.5 h. The reaction mixture was cooled to room temperature, and the reaction was quenched with the addition of a saturated aqueous NH_4Cl solution (50 mL). The reaction mixture was extracted with EtOAc (50 mL \times 2). The combined organic solution was washed with brine (25 mL), dried over MgSO_4 and concentrated in vacuo to give crude residue. The resulting

residue was purified by flash chromatography to yield **1am** as a white solid (3 g, 82% yield).

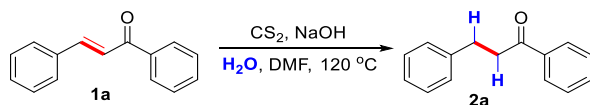
3. General experimental procedure

General Procedure for Chemoselective Reduction Unsaturated Compounds.



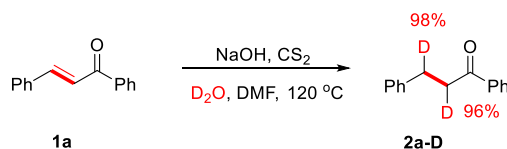
unsaturated compound **1** (0.2 mmol), NaOH (8.0 mg, 0.2 mmol) and CS₂ (42 μL, 0.7 mmol) were added to a reaction vessel (10 mL). *N,N*-dimethylacetamide (1.0 mL) and H₂O (20 μL) was added by syringe. The reaction was stirred in the oil bath at 120 °C under air for 12 h. After cooling to room temperature, the reaction mixture was diluted with ethyl acetate (15.0 mL) and washed by saturated sodium chloride solution. The organic layer was separated and the aqueous layer was extracted with ethyl acetate for three times. The combined organic layer was dried over MgSO₄ and the volatiles were removed under reduced pressure. The resulting mixture was purified by column chromatography on silica gel (eluted with EtOAc/PE) to afford the desired product.

Gram scale synthesis of **2a**



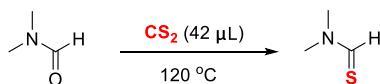
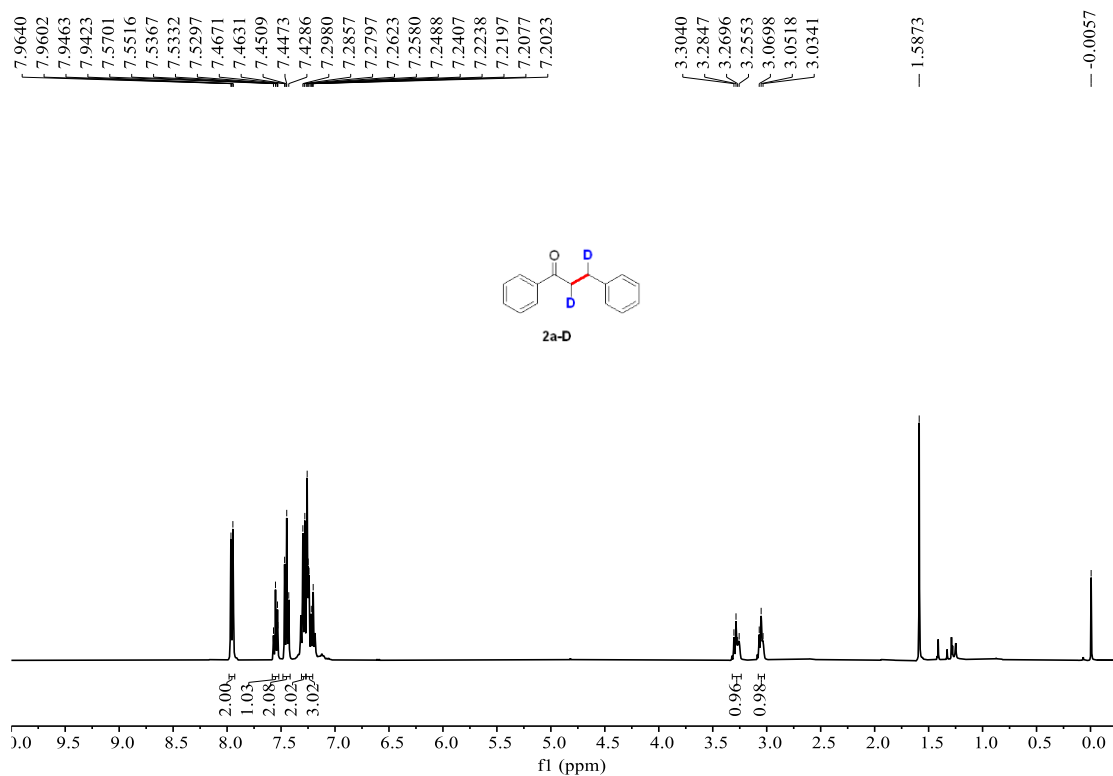
(*E*)-chalcone **1a** (1.04 g, 5 mmol), NaOH (200 mg, 5 mmol) and CS₂ (1.05 mL, 17.5 mmol) were added to a round-bottom flask (100 mL). *N,N*-dimethylacetamide (25 mL) and H₂O (500 μL) was added by syringe. The reaction was stirred in the oil bath at 120 °C under air for 12 h. After cooling to room temperature, the reaction mixture was diluted with ethyl acetate (15.0 mL) and washed by saturated sodium chloride solution. The organic layer was separated and the aqueous layer was extracted with ethyl acetate for three times. The combined organic layer was dried over MgSO₄ and the volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate=30:1) to yield the desired product 1,3-diphenylpropan-1-one **2a** as white solid (892.5 mg, 85% yield).

4. The reaction procedure of Control Experiments

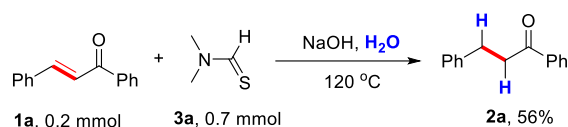
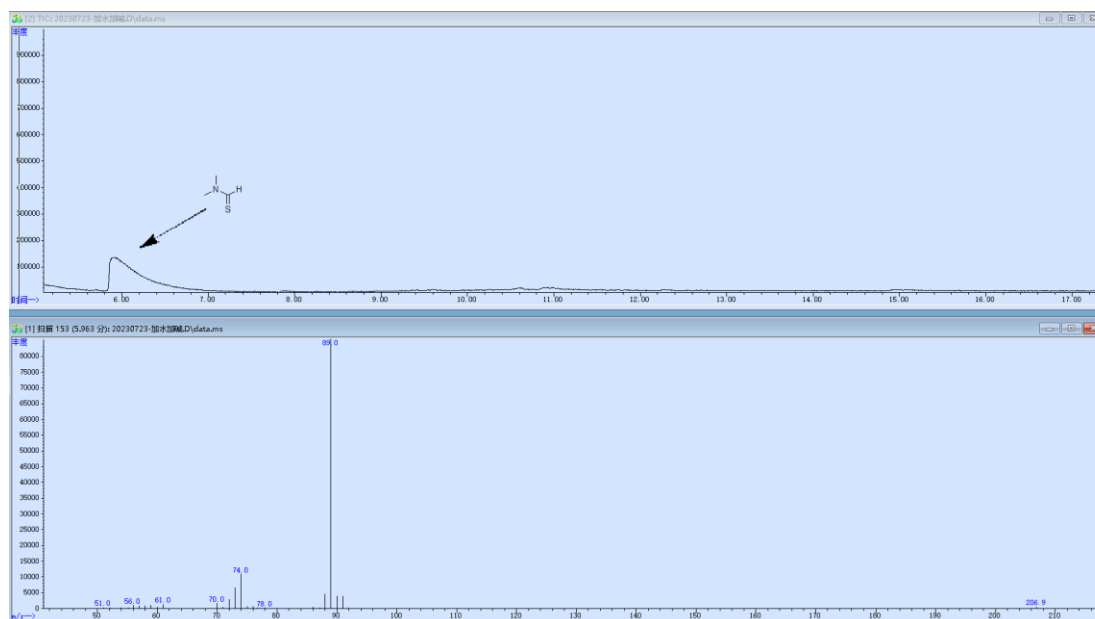


(*E*)-chalcone **1a** (0.2 mmol), NaOH (8.0 mg, 0.2 mmol) and CS₂ (42 μL, 0.7 mmol) were added to

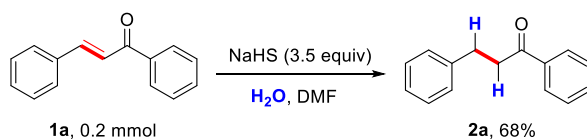
a reaction vessel (10 mL). *N,N*-dimethylacetamide (1.0 mL) and D₂O (20 μL) was added by syringe. The reaction was stirred in the oil bath at 120 °C under air for 12 h. After cooling to room temperature, the reaction mixture was diluted with ethyl acetate (15.0 mL) and washed by saturated sodium chloride solution. The organic layer was separated and the aqueous layer was extracted with ethyl acetate for three times. The combined organic layer was dried over MgSO₄ and the volatiles were removed under reduced pressure. The resulting mixture was purified by column chromatography on silica gel (eluted with EtOAc/PE) to afford the desired product **2a-D** in 89% isolated yield, and the ¹H NMR spectrum showed 98 % d-incorporation at the β position and 96 % d-incorporation at α position:



N,N-Dimethylformamide (1 mL), and CS₂ (42 μL, 0.7 mmol) were added to a reaction vessel (10 mL). The reaction was stirred in the oil bath at 120 °C under air for 12 h. After cooling to room temperature, the reaction mixture was diluted with ethyl acetate (15.0 mL) and washed by saturated sodium chloride solution. The organic layer was separated and the aqueous layer was extracted with ethyl acetate for three times. The combined organic layer was dried over MgSO₄ and the volatiles were removed under reduced pressure. The consequence was detected by GC-MS.

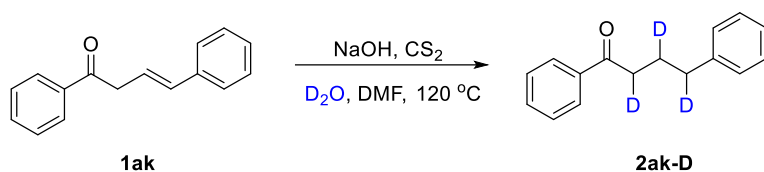


(*E*)-chalcone **1a** (0.2 mmol), NaOH (8.0 mg, 0.2 mmol) and **3a** (59.5 μL , 0.7 mmol) were added to a reaction vessel (10 mL). H₂O (20 μL) was added by syringe. The reaction was stirred in the oil bath at 120 $^\circ\text{C}$ under air for 12 h. After cooling to room temperature, the reaction mixture was diluted with ethyl acetate (15.0 mL) and washed by saturated sodium chloride solution. The organic layer was separated and the aqueous layer was extracted with ethyl acetate for three times. The combined organic layer was dried over MgSO₄ and the volatiles were removed under reduced pressure. The resulting mixture was purified by column chromatography on silica gel (eluted with EtOAc/PE) to afford the desired product **2a** in 56% yield.



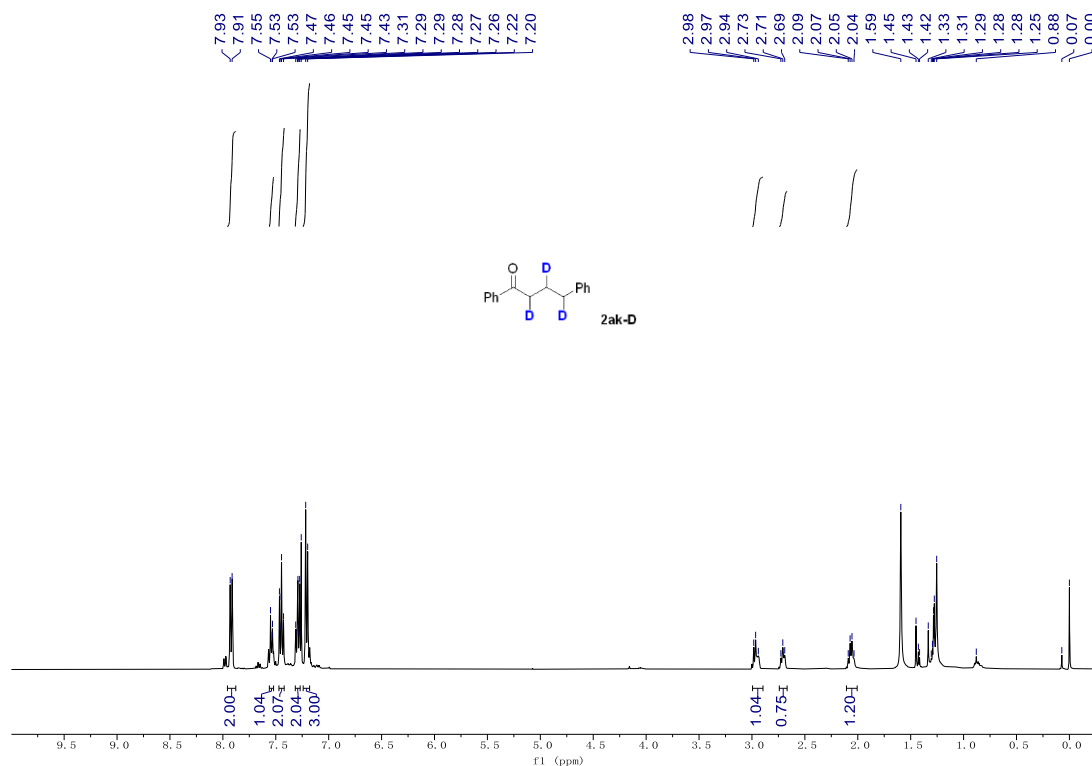
(*E*)-chalcone **1a** (0.2 mmol), NaHS (39.2 mg, 3.5 equiv.) were added to a reaction vessel (10 mL). *N,N*-dimethylacetamide (1.0 mL) and H₂O (20 μL) was added by syringe. The reaction was stirred in the oil bath at 120 $^\circ\text{C}$ under air for 12 h. After cooling to room temperature, the reaction mixture was diluted with ethyl acetate (15.0 mL) and washed by saturated sodium chloride solution. The organic layer was separated and the aqueous layer was extracted with ethyl acetate for three times. The combined organic layer was dried over MgSO₄ and the volatiles were removed under reduced

pressure. The resulting mixture was purified by column chromatography on silica gel (eluted with EtOAc/PE) to afford the desired product **2a** in 68% yield.



(*E*)-1,4-diphenylbut-3-en-1-one **1ak** (0.2 mmol), NaOH (8.0 mg, 0.2 mmol) and CS₂ (42 μL, 0.7 mmol) were added to a reaction vessel (10 mL). *N,N*-dimethylacetamide (1.0 mL) and D₂O (20 μL) was added by syringe. The reaction was stirred in the oil bath at 120 °C under air for 12 h. After cooling to room temperature, the reaction mixture was diluted with ethyl acetate (15.0 mL) and washed by saturated sodium chloride solution. The organic layer was separated and the aqueous layer was extracted with ethyl acetate for three times. The combined organic layer was dried over MgSO₄ and the volatiles were removed under reduced pressure. The resulting mixture was purified by column chromatography on silica gel (eluted with EtOAc/PE) to afford the desired product **2ak-D** in 54% isolated yield,

¹H NMR (400 MHz, CDCl₃) δ 7.92 (d, *J* = 7.3 Hz, 2H), 7.54 (d, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.32 – 7.27 (m, 2H), 7.24 – 7.18 (m, 3H), 2.96 (t, *J* = 8.7 Hz, 1H), 2.71 (t, *J* = 7.3 Hz, 1H), 2.06 (q, *J* = 7.3 Hz, 1H).

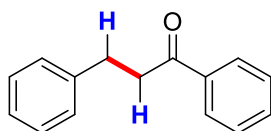


Detection of H₂S: The color change of lead acetate test paper after reaction.



5. Characterization data of products

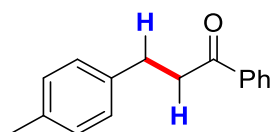
1,3-diphenylpropan-1-one (2a, CAS: 1083-30-3)¹



38.6 mg, 92% yield; white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.96 (d, *J* = 7.1 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.32 – 7.24 (m, 4H), 7.22 (t, *J* = 7.0 Hz, 1H), 3.31 (t, *J* = 7.7 Hz, 2H), 3.07 (t, *J* = 7.7 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 199.2, 141.2, 136.8, 133.1, 128.6, 128.5, 128.4, 128.0, 126.1, 40.4, 30.1.

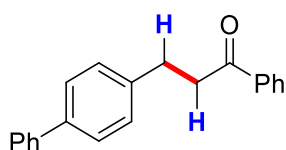
1-phenyl-3-(p-tolyl)propan-1-one (2b)³



40.3 mg, 90% yield; white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 6.8 Hz, 2H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.13 (q, *J* = 8.0 Hz, 4H), 3.27 (t, *J* = 7.7 Hz, 2H), 3.02 (t, *J* = 7.7 Hz, 2H), 2.32 (s, 3H).

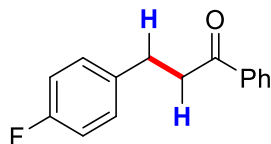
¹³C NMR (101 MHz, CDCl₃) δ 199.3, 138.1, 136.8, 135.6, 133.0, 129.1, 128.5, 128.2, 128.0, 40.6, 29.6, 21.0.

3-([1,1'-biphenyl]-4-yl)-1-phenylpropan-1-one (2c)³



45.8 mg, 80% yield; yellow solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.98 (d, $J = 7.1$ Hz, 2H), 7.60 – 7.51 (m, 5H), 7.49 – 7.40 (m, 4H), 7.36 – 7.30 (m, 3H), 3.34 (t, $J = 7.6$ Hz, 2H), 3.11 (t, $J = 7.7$ Hz, 2H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 199.1, 140.9, 140.4, 139.1, 136.8, 133.1, 128.8, 128.7, 128.6, 128.0, 127.2, 127.1, 127.0, 40.3, 29.7.

3-(4-fluorophenyl)-1-phenylpropan-1-one (2d)³

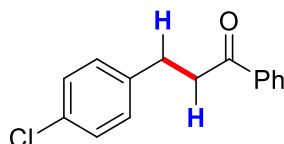


39.7 mg, 87% yield; white solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.95 (d, $J = 7.0$ Hz, 2H), 7.55 (t, $J = 7.3$ Hz, 1H), 7.45 (t, $J = 7.7$ Hz, 2H), 7.23 – 7.17 (m, 2H), 6.97 (t, $J = 8.7$ Hz, 2H), 3.28 (t, $J = 7.6$ Hz, 2H), 3.04 (t, $J = 7.5$ Hz, 2H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 199.0, 161.3 (d, $J = 242$ Hz), 136.8 (d, $J = 3$ Hz), 136.7, 133.1, 129.8 (d, $J = 7$ Hz), 128.6, 128.0, 115.2 (d, $J = 21$ Hz), 40.3, 29.2.

$^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -117.21.

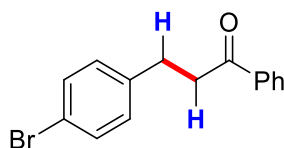
3-(4-chlorophenyl)-1-phenylpropan-1-one (2e)⁴



40.5 mg, 83% yield; white solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.95 (d, $J = 8.1$ Hz, 2H), 7.56 (d, $J = 7.7$ Hz, 1H), 7.46 (t, $J = 7.7$ Hz, 2H), 7.27 (d, $J = 4.7$ Hz, 2H), 7.19 (d, $J = 8.5$ Hz, 2H), 3.29 (t, $J = 7.6$ Hz, 2H), 3.05 (t, $J = 7.5$ Hz, 2H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 198.8, 139.7, 136.7, 133.2, 131.8, 129.8, 128.6, 128.6, 128.0, 40.1, 29.3.

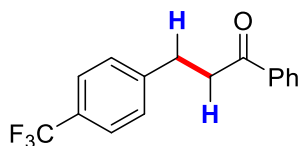
3-(4-bromophenyl)-1-phenylpropan-1-one (2f)⁴



49.0 mg, 85% yield; white solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.95 (d, $J = 7.0$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.49 – 7.38 (m, 4H), 7.13 (d, $J = 8.3$ Hz, 2H), 3.28 (t, $J = 7.5$ Hz, 2H), 3.02 (t, $J = 7.5$ Hz, 2H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 198.8, 140.2, 136.6, 133.2, 131.5, 130.2, 128.6, 128.0, 119.8, 40.0, 29.4.

1-phenyl-3-(4-(trifluoromethyl)phenyl)propan-1-one (2g)¹

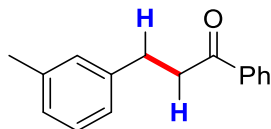


40.0 mg, 72% yield; white solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.96 (d, $J = 7.0$ Hz, 2H), 7.60 – 7.52 (m, 3H), 7.46 (t, $J = 7.7$ Hz, 2H), 7.37 (d, $J = 8.0$ Hz, 2H), 3.33 (t, $J = 7.5$ Hz, 2H), 3.14 (t, $J = 7.5$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 198.6, 145.4, 136.7, 133.2, 129.3, 128.8, 128.7, 128.0, 125.6(t, $J = 272$ Hz), 125.4 (q, $J = 3.8$ Hz), 39.8, 29.8.

^{19}F NMR (376 MHz, CDCl_3) δ -62.35.

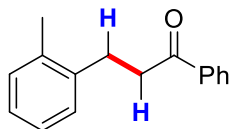
1-phenyl-3-(*m*-tolyl)propan-1-one (2h)¹



40.8 mg, 91% yield; white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.96 (d, $J = 7.0$ Hz, 2H), 7.59 – 7.52 (m, 1H), 7.45 (t, $J = 7.7$ Hz, 2H), 7.20 (t, $J = 7.5$ Hz, 1H), 7.09 – 7.00 (m, 3H), 3.29 (t, $J = 7.8$ Hz, 2H), 3.03 (t, $J = 7.8$ Hz, 2H), 2.34 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 199.3, 141.2, 138.1, 136.8, 133.0, 129.2, 128.6, 128.4, 128.0, 126.8, 125.4, 40.5, 30.0, 21.4.

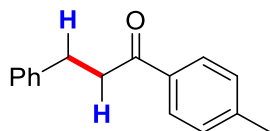
1-phenyl-3-(*o*-tolyl)propan-1-one (2i)⁴



27.3 mg, 61% yield; white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.97 (d, $J = 7.0$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.46 (t, $J = 7.7$ Hz, 2H), 7.22 – 7.13 (m, 4H), 3.26 (t, $J = 7.7$ Hz, 2H), 3.06 (t, $J = 7.9$ Hz, 2H), 2.35 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 199.4, 139.4, 136.8, 136.0, 133.1, 130.3, 128.7, 128.6, 128.0, 126.3, 126.2, 39.1, 27.5, 19.4.

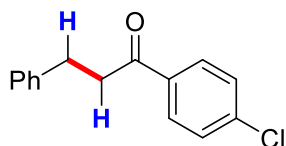
3-phenyl-1-(*p*-tolyl)propan-1-one (2j)¹



37.6 mg, 84% yield; white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.85 (d, $J = 8.2$ Hz, 2H), 7.30 – 7.19 (m, 7H), 3.27(t, $J = 7.7$ Hz, 2H), 3.05 (t, $J = 7.7$ Hz, 2H), 2.39 (s, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 203.2, 141.1, 138.0, 137.8, 131.9, 131.2, 128.4, 128.3, 128.3, 126.0, 125.6, 43.1, 30.2, 21.2.

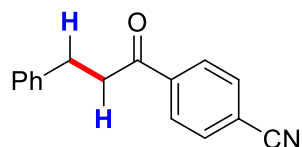
1-(4-chlorophenyl)-3-phenylpropan-1-one (2k)¹



15.1 mg, 31% yield; white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.89 (d, $J = 8.6$ Hz, 2H), 7.42 (d, $J = 8.6$ Hz, 2H), 7.30 (t, $J = 7.4$ Hz, 2H), 7.26 – 7.18 (m, 3H), 3.27 (t, $J = 7.7$ Hz, 2H), 3.06 (t, $J = 7.6$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 198.0, 141.1, 139.5, 135.2, 129.5, 128.9, 128.6, 128.4, 126.2, 40.4, 30.1.

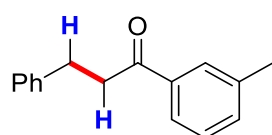
4-(3-phenylpropanoyl)benzonitrile (**2l**)⁵



21.6 mg, 46% yield; yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.99 – 7.87 (m, 4H), 7.73 (s, 1H), 7.31 (t, *J* = 7.4 Hz, 2H), 7.23 (d, *J* = 8.9 Hz, 2H), 3.31 (t, *J* = 7.6 Hz, 2H), 3.07 (t, *J* = 7.6 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 201.7, 198.5, 142.8, 140.9, 139.0, 128.6, 128.4, 128.2, 127.2, 126.3, 40.8, 30.0.

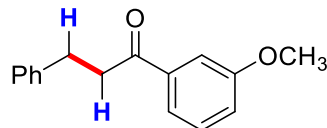
3-phenyl-1-(*m*-tolyl)propan-1-one (**2m**)⁶



38.5 mg, 86% yield; white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, *J* = 8.9 Hz, 2H), 7.38 – 7.26 (m, 5H), 7.25 – 7.19 (m, 2H), 3.28 (t, *J* = 7.7 Hz, 2H), 3.06 (t, *J* = 7.7 Hz, 2H), 2.39 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 198.9, 143.8, 141.3, 134.3, 129.2, 128.5, 128.4, 128.1, 126.0, 40.3, 30.1, 21.6.

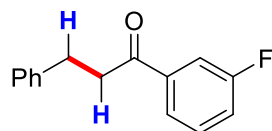
1-(3-methoxyphenyl)-3-phenylpropan-1-one (**2n**)⁵



43.2 mg, 90% yield; white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.53 (d, *J* = 7.7 Hz, 1H), 7.49 (t, *J* = 2.1 Hz, 1H), 7.38 – 7.27 (m, 4H), 7.25 – 7.20 (m, 2H), 7.12 – 7.08 (m, 1H), 3.84 (s, 3H), 3.29 (t, *J* = 7.7 Hz, 2H), 3.06 (t, *J* = 7.7 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 199.0, 159.8, 141.2, 138.2, 129.6, 128.5, 128.4, 126.1, 120.6, 119.6, 112.2, 55.4, 40.5, 30.1.

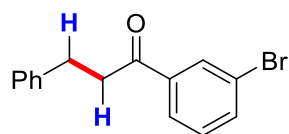
1-(3-fluorophenyl)-3-phenylpropan-1-one (**2o**)⁷



33.3 mg, 73% yield; white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.73 (d, *J* = 7.7 Hz, 1H), 7.63 (d, *J* = 9.6 Hz, 1H), 7.45 – 7.40 (m, 1H), 7.33 – 7.26 (m, 3H), 7.25 – 7.21 (m, 3H), 3.28 (t, *J* = 7.7 Hz, 2H), 3.07 (t, *J* = 7.6 Hz, 2H).

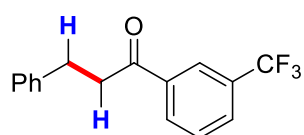
¹³C NMR (101 MHz, CDCl₃) δ 197.9, 162.8(d, *J* = 246 Hz), 141.0, 138.9(d, *J* = 5 Hz), 130.2(d, *J* = 8 Hz), 128.4(q, *J* = 17.7 Hz), 126.2, 123.7(d, *J* = 3 Hz), 120.2, 120.0, 114.8(d, *J* = 22 Hz), 40.6, 29.9.

¹⁹F NMR (376 MHz, CDCl₃) δ -111.78.

1-(3-bromophenyl)-3-phenylpropan-1-one (2p)⁶

46.1 mg, 90% yield; white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.92 (s, 1H), 7.83 (d, *J* = 9.2 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.40 (t, *J* = 7.9 Hz, 1H), 7.33 – 7.28 (m, 2H), 7.26 – 7.18 (m, 3H), 3.28 (t, *J* = 7.6 Hz, 2H), 3.07 (t, *J* = 7.6 Hz, 2H).

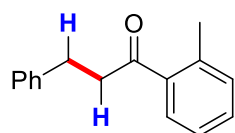
¹³C NMR (101 MHz, CDCl₃) δ 197.9, 141.0, 138.3, 134.9, 133.0, 129.9, 128.6, 128.4, 128.2, 126.2, 126.1, 40.6, 29.9.

3-phenyl-1-(3-(trifluoromethyl)phenyl)propan-1-one (2q)⁸

37.8 mg, 68% yield; white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.20 (s, 1H), 8.13 (d, *J* = 7.9 Hz, 1H), 7.81 (d, *J* = 7.8 Hz, 1H), 7.60 (t, *J* = 7.8 Hz, 1H), 7.31 (t, *J* = 7.4 Hz, 2H), 7.26 – 7.20 (m, 3H), 3.33 (t, *J* = 7.6 Hz, 2H), 3.09 (t, *J* = 7.6 Hz, 2H).

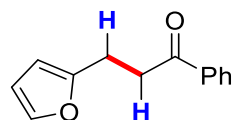
¹³C NMR (101 MHz, CDCl₃) δ 197.8, 140.9, 137.3, 131.4, 131.1, 129.5 (q, *J* = 3.7 Hz), 129.3, 128.5 (t, *J* = 19 Hz), 126.3, 125.0, 124.9 (q, *J* = 3.8 Hz), 122.3, 40.6, 29.9.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.78.

3-phenyl-1-(o-tolyl)propan-1-one (2r)⁸

38.5 mg, 86% yield; white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.57 (d, *J* = 6.6 Hz, 1H), 7.35 – 7.25 (m, 3H), 7.23 – 7.16 (m, 5H), 3.20 (t, *J* = 7.6 Hz, 2H), 3.03 (t, *J* = 7.6 Hz, 2H), 2.46 (s, 3H).

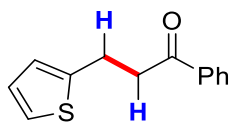
¹³C NMR (101 MHz, CDCl₃) δ 199.4, 141.3, 138.3, 136.8, 133.8, 128.5, 128.5, 128.4, 128.4, 126.1, 125.2, 40.5, 30.1, 21.3.

3-(furan-2-yl)-1-phenylpropan-1-one (2s)⁸

32.0 mg, 80% yield; yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.98 (d, *J* = 7.2 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.31 (s, 1H), 6.28 (s, 1H), 6.06 (s, 1H), 3.34 (t, *J* = 7.5 Hz, 2H), 3.09 (t, *J* = 7.5 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 198.7, 154.7, 141.1, 136.7, 133.1, 128.6, 128.0, 110.2, 105.3, 36.9, 22.5.

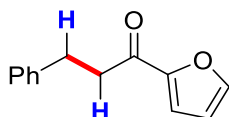
1-phenyl-3-(thiophen-2-yl)propan-1-one (2t)⁸



35.0 mg, 81% yield; white oil. ^1H NMR (400 MHz, CDCl_3) δ 7.97 (d, $J = 7.0$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.47 (t, $J = 7.7$ Hz, 2H), 7.13 (d, $J = 3.9$ Hz, 1H), 6.92 (t, $J = 4.3$ Hz, 1H), 6.87 (d, $J = 2.3$ Hz, 1H), 3.37 (t, $J = 7.0$ Hz, 2H), 3.30 (t, $J = 6.8$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 198.6, 143.9, 136.7, 133.2, 128.6, 128.0, 126.8, 124.7, 123.4, 40.5, 24.2.

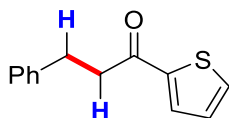
1-(furan-2-yl)-3-phenylpropan-1-one (2u)⁸



28.8 mg, 72% yield; white oil. ^1H NMR (400 MHz, CDCl_3) δ 7.55 (s, 1H), 7.29 (t, $J = 7.4$ Hz, 2H), 7.25 – 7.18 (m, 3H), 7.16 (d, $J = 3.6$ Hz, 1H), 6.51 (dd, $J = 3.6, 1.7$ Hz, 1H), 3.15 (t, $J = 7.3$ Hz, 2H), 3.04 (t, $J = 7.7$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 188.4, 152.5, 146.3, 140.9, 128.4, 128.3, 126.1, 117.0, 112.1, 40.1, 29.8.

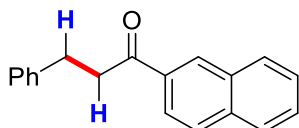
3-phenyl-1-(thiophen-2-yl)propan-1-one (2v)⁸



30.2 mg, 70% yield; yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.69 (d, $J = 3.8$ Hz, 1H), 7.63 (d, $J = 5.0$ Hz, 1H), 7.29 (t, $J = 7.4$ Hz, 2H), 7.26 – 7.18 (m, 3H), 7.11 (t, $J = 7.3$ Hz, 1H), 3.24 (t, $J = 7.9$ Hz, 2H), 3.07 (t, $J = 7.7$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 192.2, 144.1, 141.0, 133.6, 131.8, 128.5, 128.4, 128.1, 126.2, 41.1, 30.3.

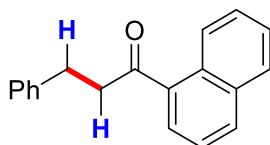
1-(naphthalen-2-yl)-3-phenylpropan-1-one (2w)⁵



40.6 mg, 78% yield; white solid. ^1H NMR (400 MHz, CDCl_3) δ 8.45 (s, 1H), 8.03 (d, $J = 8.6$ Hz, 1H), 7.95 – 7.83 (m, 3H), 7.62 – 7.50 (m, 2H), 7.34 – 7.27 (m, 4H), 7.24 – 7.21 (m, 1H), 3.43 (t, $J = 7.7$ Hz, 2H), 3.13 (t, $J = 7.7$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 199.1, 141.3, 135.5, 134.1, 132.5, 129.7, 129.5, 128.5, 128.4, 128.4, 127.7, 126.7, 126.1, 123.8, 40.5, 30.2.

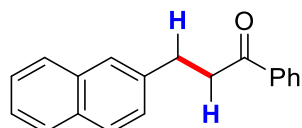
1-(naphthalen-1-yl)-3-phenylpropan-1-one (2x)⁸



32.8 mg, 63% yield; white solid. ^1H NMR (400 MHz, CDCl_3) δ 8.55 (d, $J = 7.8$ Hz, 1H), 7.96 (d, $J = 8.2$ Hz, 1H), 7.86 (d, $J = 8.0$ Hz, 1H), 7.80 (d, $J = 7.2$ Hz, 1H), 7.54 (m, 2H), 7.45 (t, $J = 7.7$ Hz, 1H), 7.31 – 7.26 (m, 3H), 7.25 – 7.17 (m, 2H), 3.37 (t, $J = 7.6$ Hz, 2H), 3.13 (t, $J = 7.6$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 203.5, 141.1, 135.9, 133.9, 132.6, 130.1, 128.5, 128.4, 128.4, 127.8, 127.4, 126.4, 126.1, 125.7, 124.3, 43.8, 30.5.

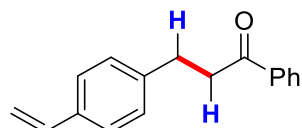
3-(naphthalen-2-yl)-1-phenylpropan-1-one (2y)⁸



44.2 mg, 85% yield; white solid. ^1H NMR (400 MHz, CDCl_3) δ 8.04 (d, $J = 8.2$ Hz, 1H), 7.94 (d, $J = 7.0$ Hz, 2H), 7.86 (d, $J = 7.7$ Hz, 1H), 7.73 (t, $J = 4.8$ Hz, 1H), 7.54 – 7.45 (m, 3H), 7.45 – 7.36 (m, 4H), 3.53 (t, $J = 7.4$ Hz, 2H), 3.41 (t, $J = 7.3$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 199.2, 137.3, 136.7, 133.9, 133.1, 131.6, 128.9, 128.6, 128.0, 127.0, 126.1, 126.0, 125.6, 125.6, 123.5, 39.7, 27.1.

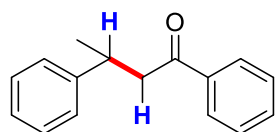
1-phenyl-3-(4-vinylphenyl)propan-1-one (2z)⁹



17.9 mg, 38% yield; yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.95 (d, $J = 7.7$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.45 (t, $J = 7.6$ Hz, 2H), 7.35 (d, $J = 7.8$ Hz, 2H), 7.21 (d, $J = 7.8$ Hz, 2H), 6.69 (dd, $J = 17.6$, 10.8 Hz, 1H), 5.71 (d, $J = 17.6$ Hz, 1H), 5.20 (d, $J = 10.9$ Hz, 1H), 3.29 (t, $J = 7.7$ Hz, 2H), 3.06 (t, $J = 7.7$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 199.1, 141.0, 136.8, 136.5, 135.6, 133.1, 128.6, 128.0, 126.3, 113.2, 40.3, 29.8.

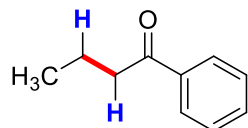
(S)-1,3-diphenylbutan-1-one (2ab)¹



26.0 mg, 58% yield; white solid. ^1H NMR (400 MHz, CDCl_3) δ 7.93 (d, $J = 7.0$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.44 (t, $J = 7.7$ Hz, 2H), 7.33 – 7.26 (m, 4H), 7.20 (m, $J = 1$ Hz), 3.51 (dq, $J = 13.9$, 6.9 Hz, 1H), 3.30 (dd, $J = 16.5$, 5.7 Hz, 1H), 3.19 (dd, $J = 16.5$, 8.3 Hz, 1H), 1.34 (d, $J = 6.9$ Hz, 3H).

^{13}C NMR (101 MHz, CDCl_3) δ 199.1, 146.6, 137.2, 133.0, 128.6, 128.5, 128.1, 126.8, 126.3, 47.0, 35.6, 21.9.

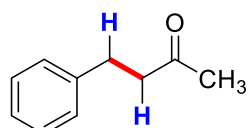
1-phenylbutan-1-one (2ac, CAS:495-40-9)¹⁰



17.8 mg, 60% yield; colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.97 (d, $J = 7.0$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.46 (t, $J = 7.5$ Hz, 2H), 2.95 (t, $J = 7.3$ Hz, 2H), 1.83 – 1.72 (m, 2H), 1.01 (t, $J = 7.4$ Hz, 3H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 200.5, 137.1, 132.9, 128.5, 128.0, 40.5, 17.7, 13.9.

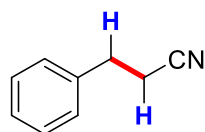
4-phenylbutan-2-one (2ad, CAS: 2550-26-7)¹



18.4 mg, 62% yield; colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.26 (t, $J = 7.5$ Hz, 2H), 7.18 (t, $J = 7.5$ Hz, 3H), 2.89 (t, $J = 7.6$ Hz, 2H), 2.76 (t, $J = 7.4$ Hz, 2H), 2.14 (s, 3H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 208.0, 140.9, 128.4, 128.2, 126.1, 45.1, 30.0, 29.7.

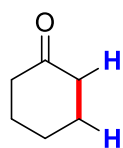
3-phenylpropanenitrile (2ae, CAS: 645-59-0)¹¹



8.4 mg, 32% yield; colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.38 – 7.30 (m, 2H), 7.28 (d, $J = 7.3$ Hz, 1H), 7.23 (d, $J = 7.0$ Hz, 3H), 2.96 (t, $J = 7.4$ Hz, 2H), 2.62 (t, $J = 7.4$ Hz, 2H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 138.0, 128.8, 128.2, 127.2, 119.1, 31.5, 19.3.

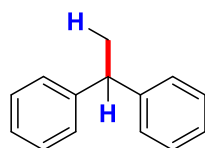
Cyclohexanone (2af, CAS: 108-94-1)¹²



2.9 mg, 15% yield; colorless oil. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 2.34 (t, $J = 6.6$ Hz, 4H), 1.87 (t, $J = 6.2$ Hz, 4H), 1.75 – 1.70 (m, 2H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 212.2, 41.9, 27.0, 25.0.

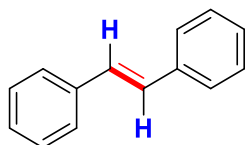
ethane-1,1-diylidibenzene (2ag, CAS: 612-00-0)¹³



26.2 mg, 72% yield; colorless solid. $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.32 – 7.25 (m, 4H), 7.25 – 7.14 (m, 6H), 4.15 (q, $J = 7.2$ Hz, 1H), 1.64 (d, $J = 5.4$ Hz, 3H).

$^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 146.3, 128.3, 127.6, 126.0, 44.7, 21.8.

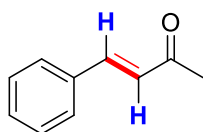
(E)-1,2-diphenylethene (2ah, CAS: 103-30-0)³



21.2 mg, 59% yield; white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.52 (d, *J* = 7.0 Hz, 4H), 7.36 (t, *J* = 7.6 Hz, 4H), 7.25 (d, *J* = 7.5 Hz, 2H), 7.11 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 137.3, 128.7, 127.6, 126.5.

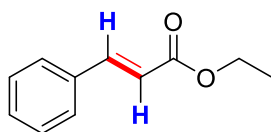
(E)-4-phenylbut-3-en-2-one (1ad, CAS: 1896-62-4)¹⁴



9.6 mg, 33% yield; colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.59 – 7.49 (m, 3H), 7.43 – 7.38 (m, 3H), 6.72 (d, *J* = 16.3 Hz, 1H), 2.39 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 198.5, 143.5, 134.4, 130.5, 129.0, 128.2, 127.1, 27.5.

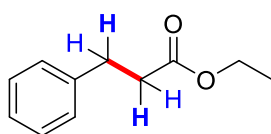
ethyl cinnamate (2aj, CAS: 4192-77-2)¹⁵



20.1 mg, 57% yield; colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, *J* = 16.0 Hz, 1H), 7.51 (dd, *J* = 6.6, 3.0 Hz, 2H), 7.40 – 7.34 (m, 3H), 6.43 (d, *J* = 16.0 Hz, 1H), 4.26 (q, *J* = 7.1 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 167.0, 144.6, 134.4, 130.2, 128.8, 128.0, 118.2, 60.5, 14.3.

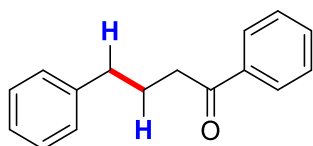
ethyl 3-phenylpropanoate (2aj)', CAS: 2021-28-5)¹⁵



4.6 mg, 13% yield; faint yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 7.32 – 7.26 (m, 2H), 7.23 – 7.17 (m, 3H), 4.13 (q, *J* = 7.1 Hz, 2H), 2.95 (t, *J* = 7.9 Hz, 2H), 2.62 (t, *J* = 7.8 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 172.9, 140.6, 128.5, 128.3, 126.2, 60.4, 35.9, 31.0, 14.2.

1,4-diphenylbutan-1-one (2ak, CAS: 5407-91-0)¹⁶



30.0 mg, 67% yield; faint yellow solid. ^1H NMR (400 MHz, CDCl_3) δ 7.92 (d, $J = 7.1$ Hz, 2H), 7.55 (t, $J = 7.4$ Hz, 1H), 7.45 (t, $J = 7.7$ Hz, 2H), 7.33 – 7.27 (m, 2H), 7.24 – 7.16 (m, 3H), 2.98 (t, $J = 7.3$ Hz, 2H), 2.73 (t, $J = 7.6$ Hz, 2H), 2.09 (p, $J = 7.4$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 200.1, 141.7, 137.0, 132.9, 128.5, 128.5, 128.4, 128.0, 125.9, 37.7, 35.2, 25.7.

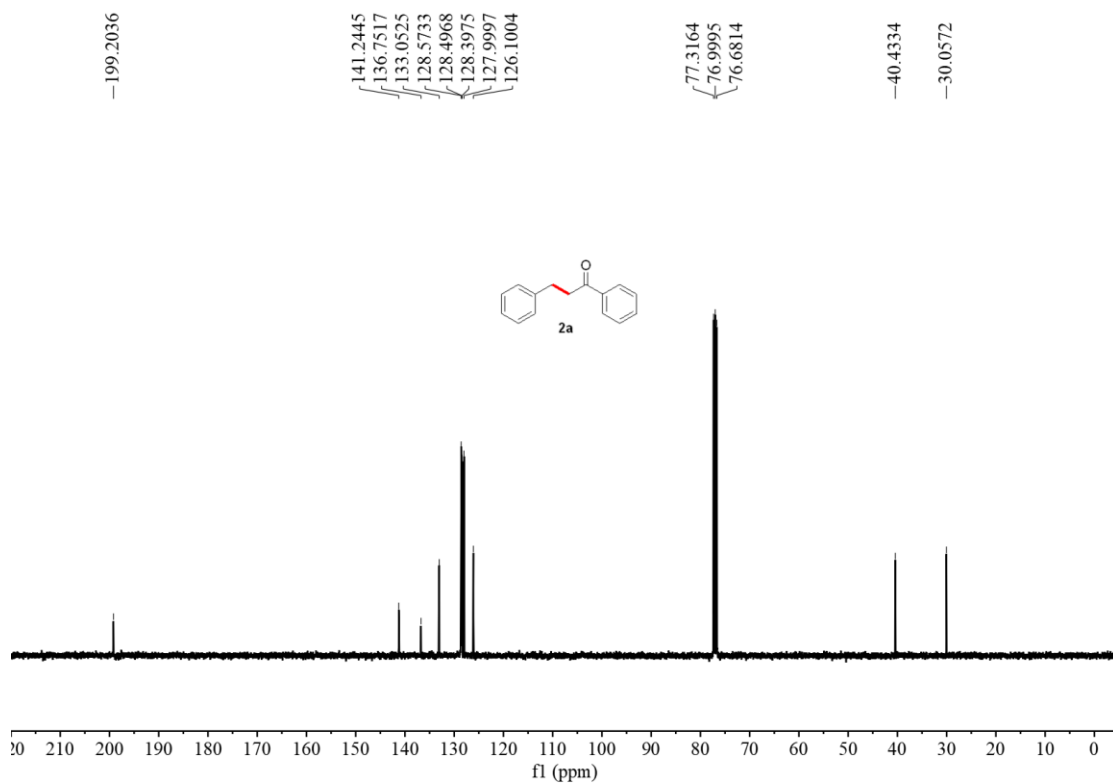
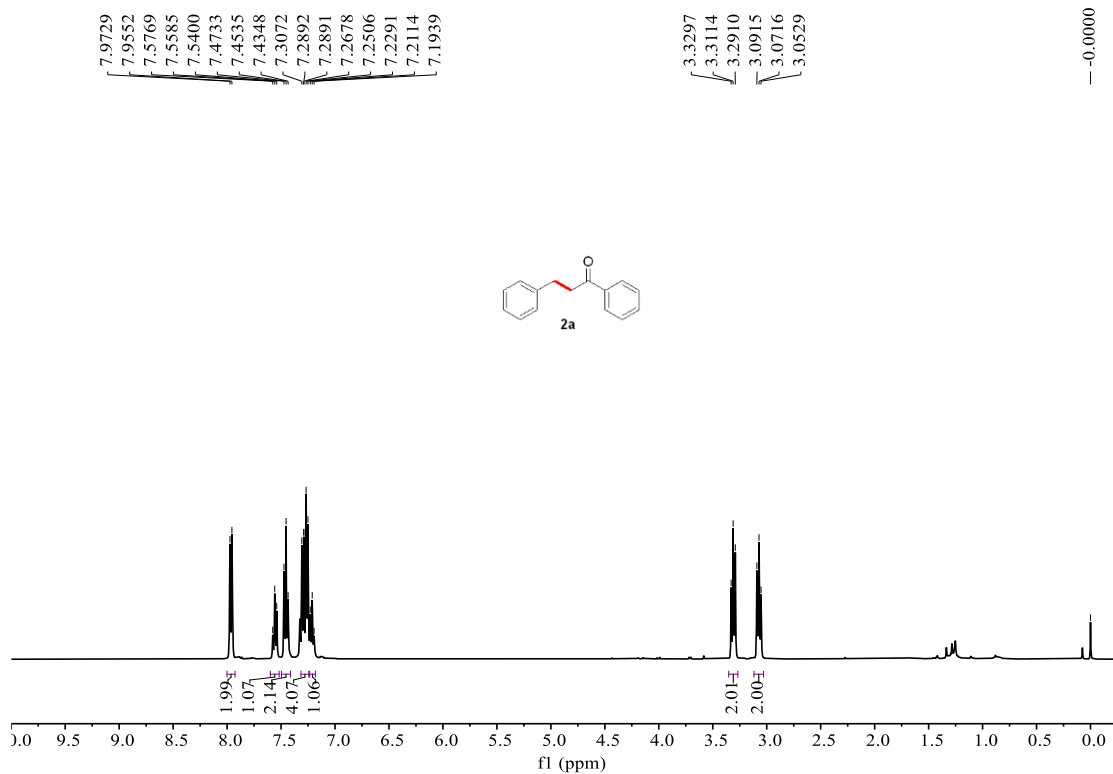
6. Reference

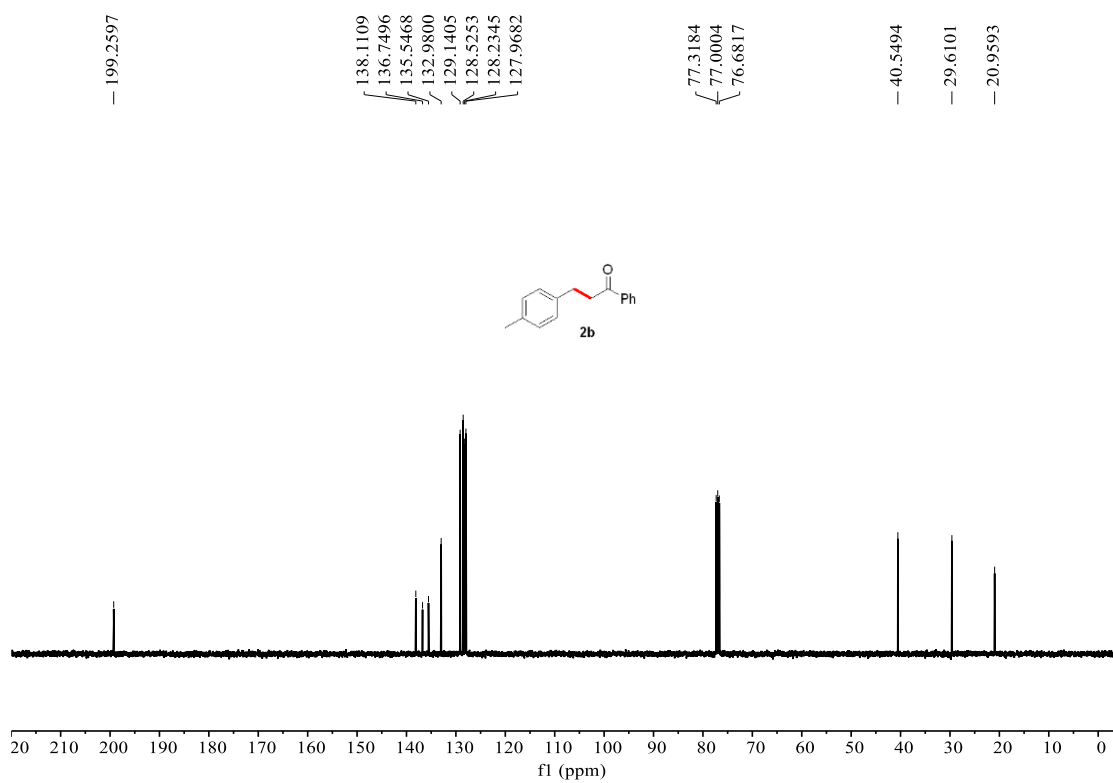
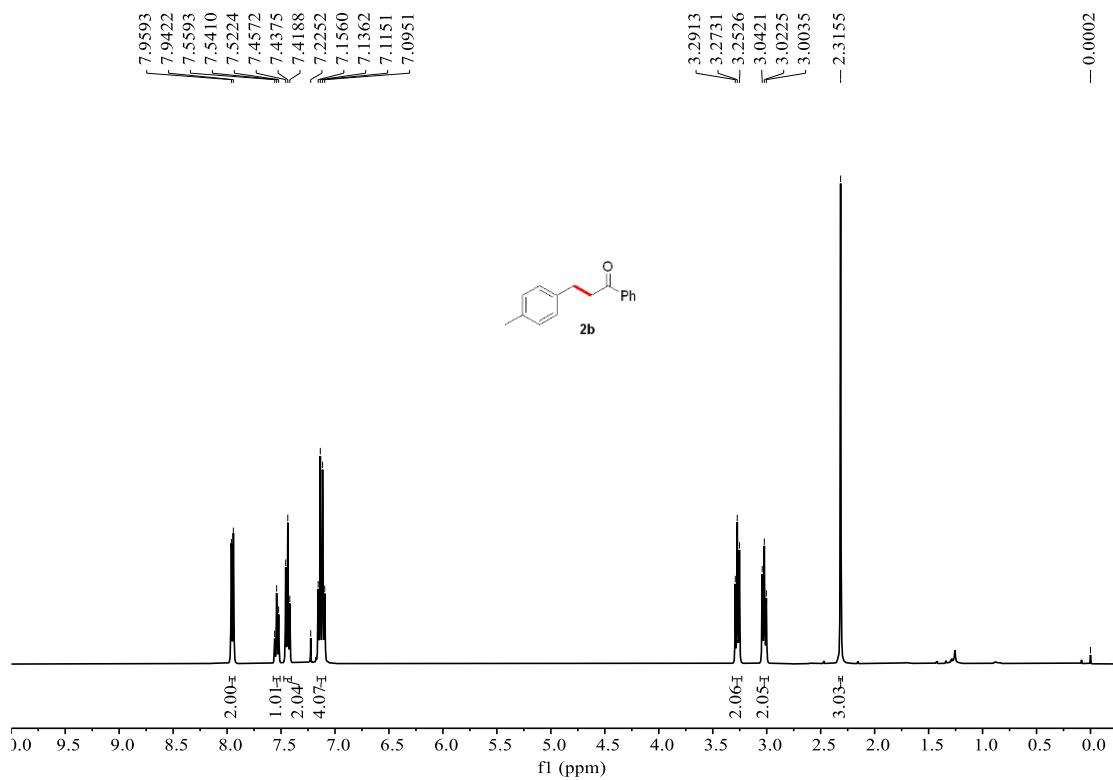
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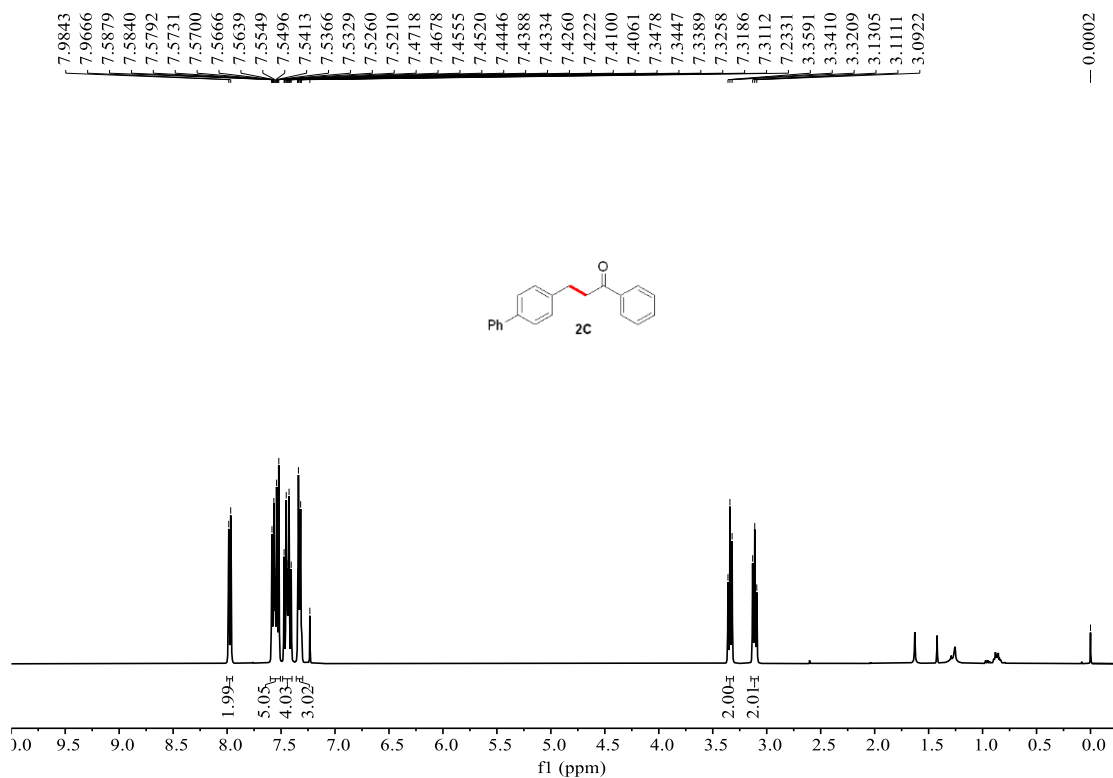
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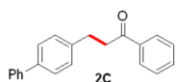
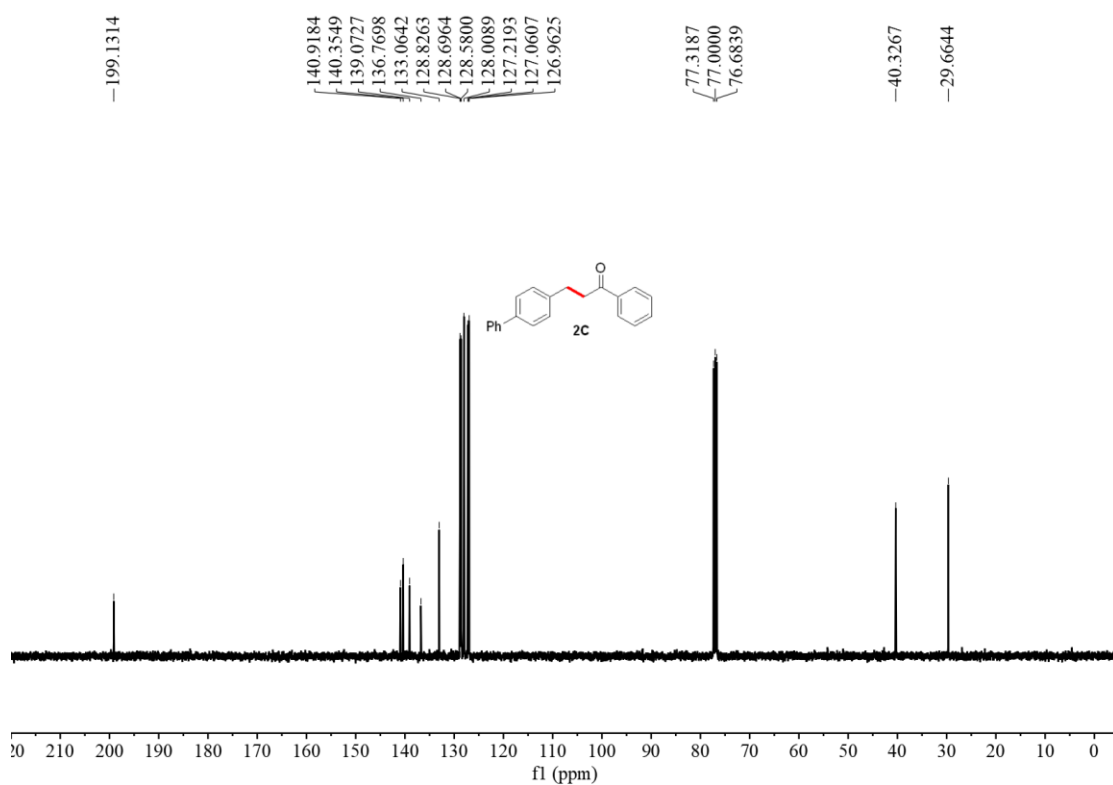
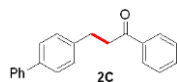
7. NMR Spectra for the compounds prepared

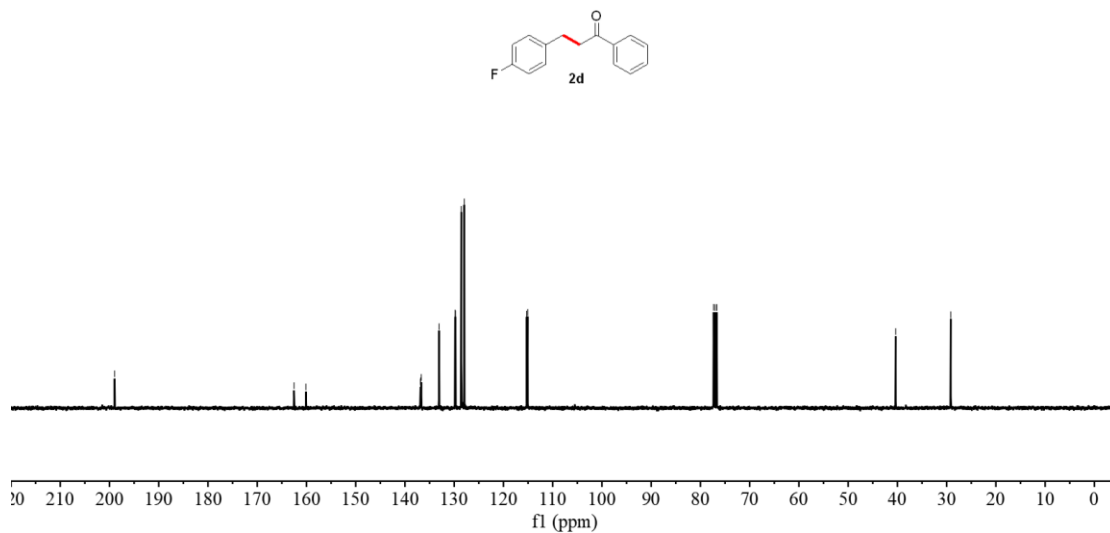
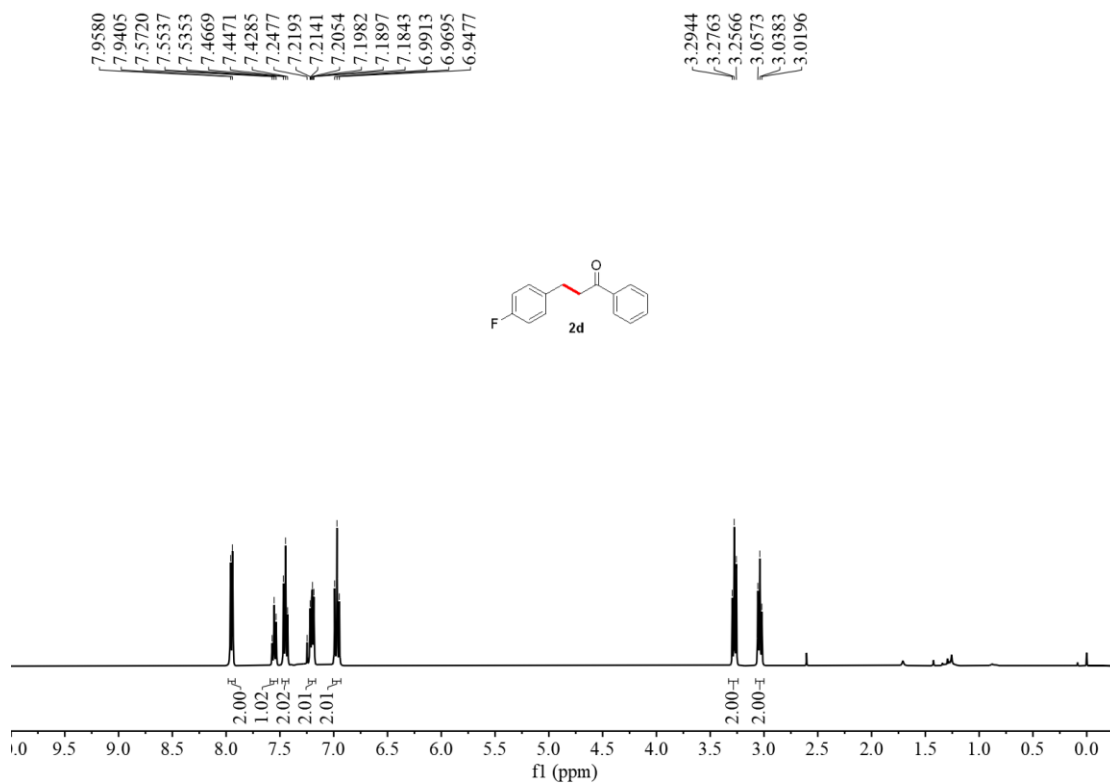


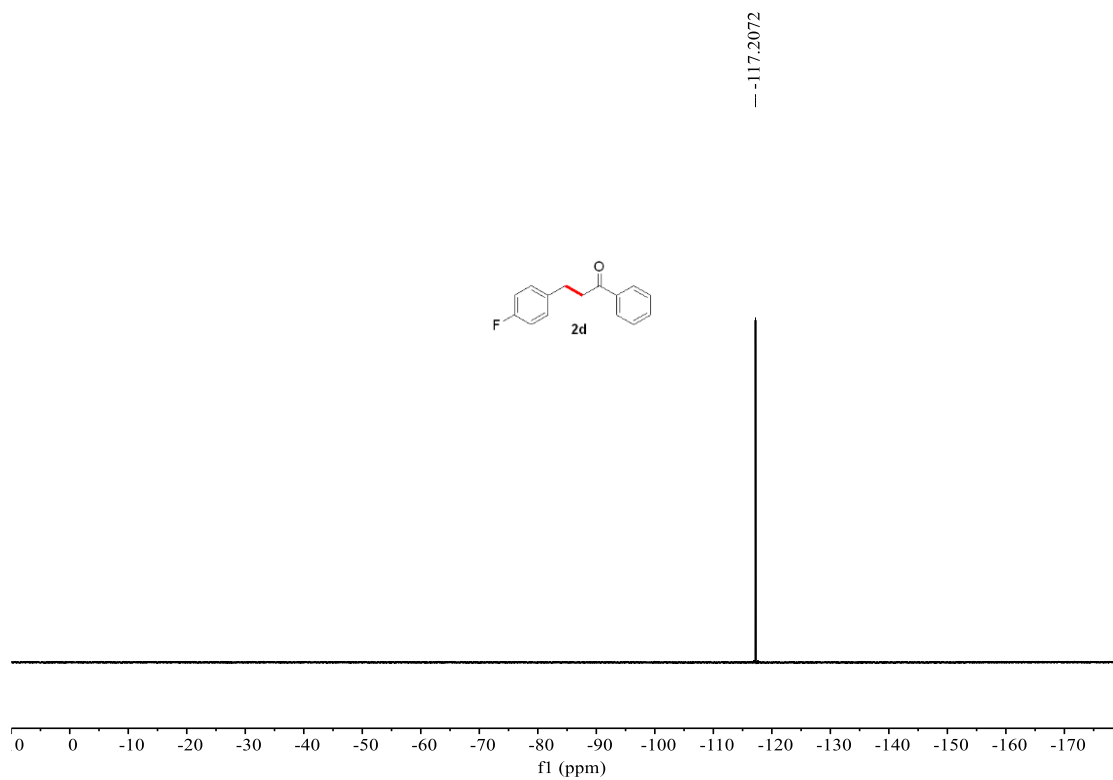




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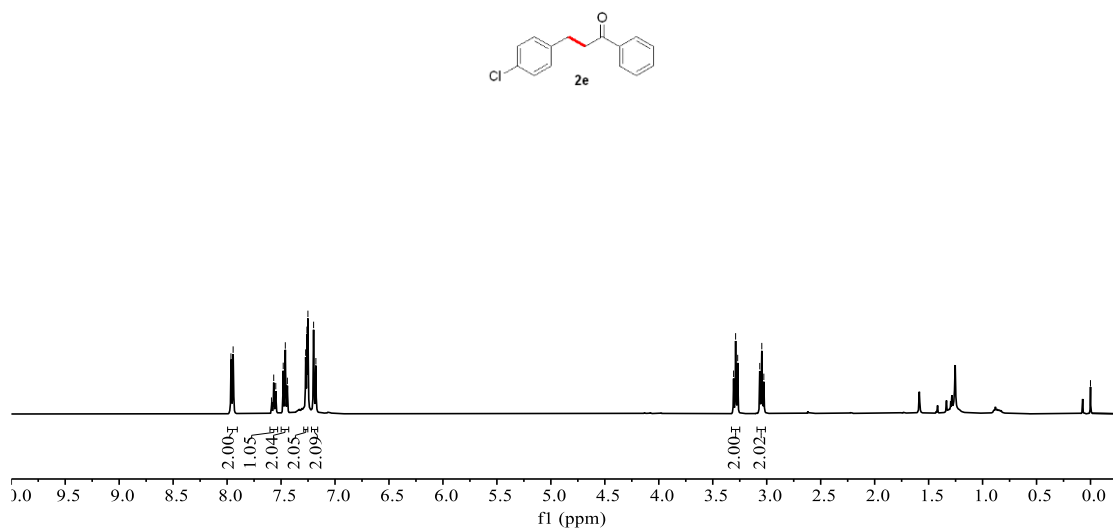




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7.5890
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7.2720
7.2603
7.2511
7.1985
7.1773

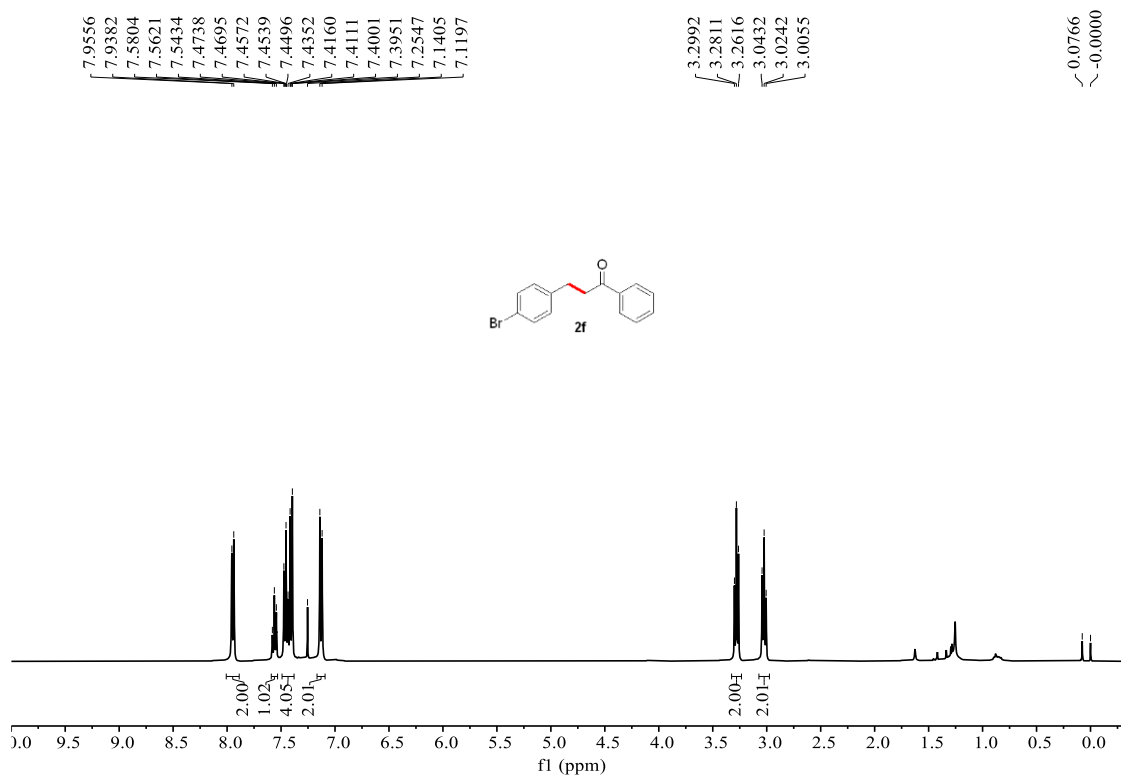
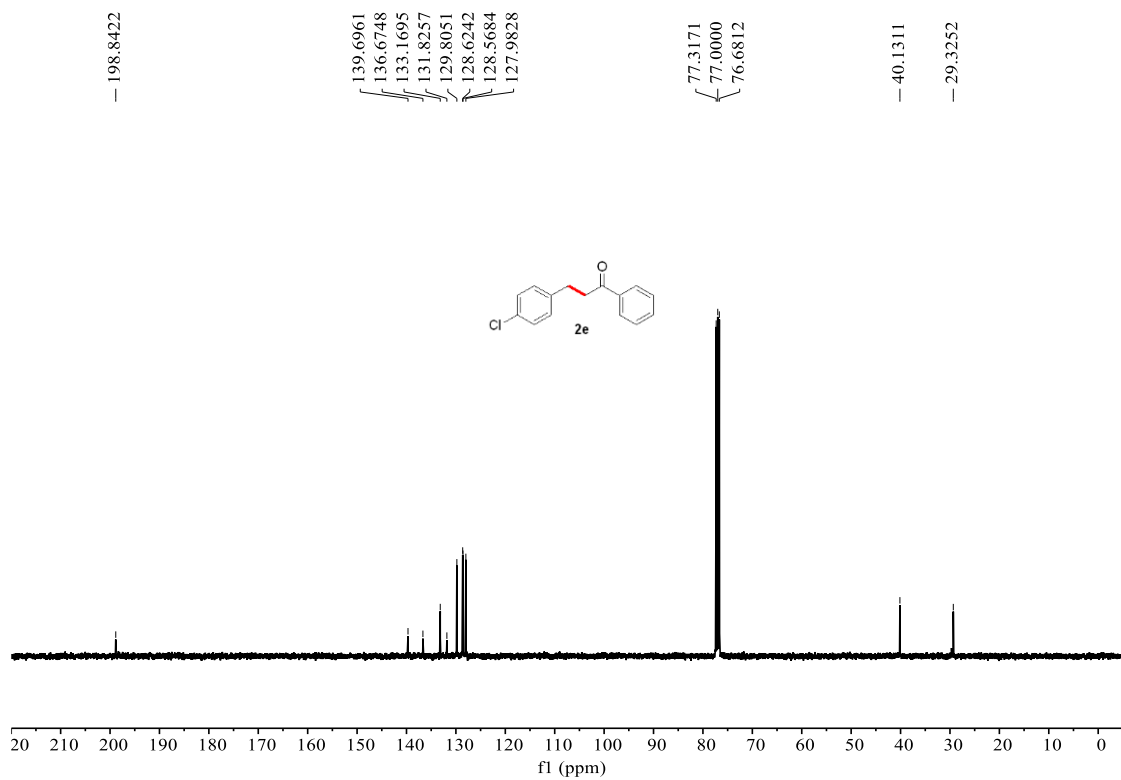
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3.0269

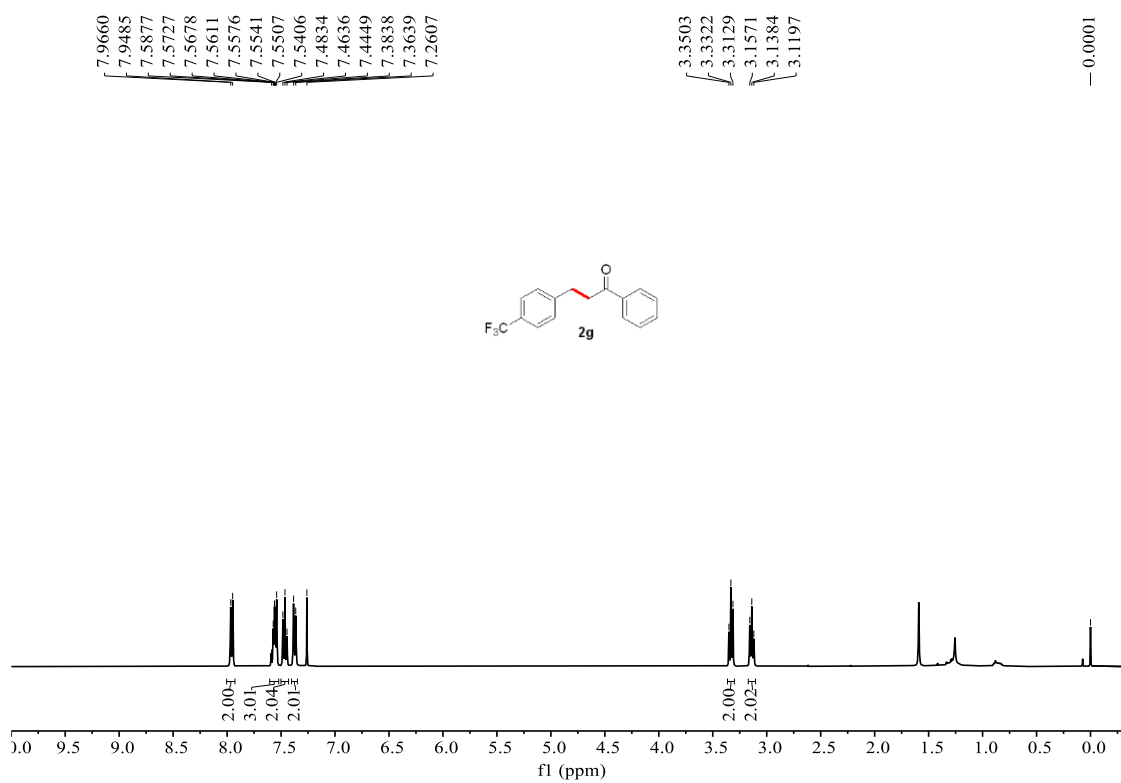
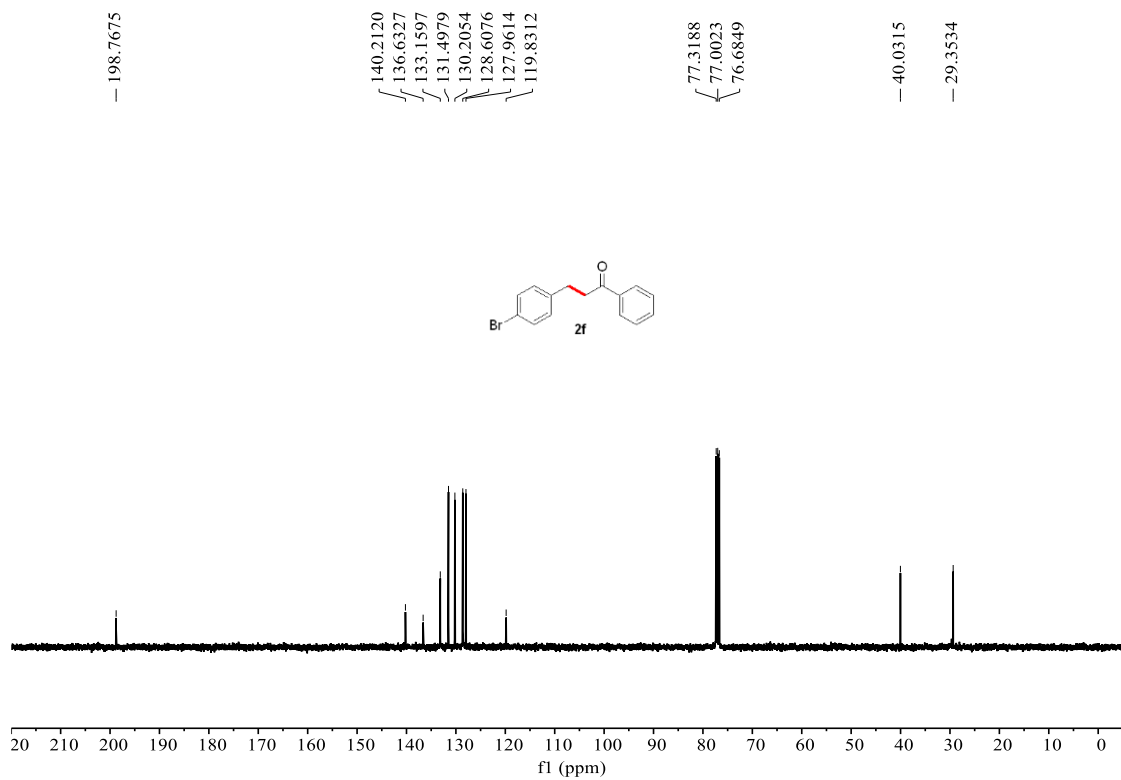
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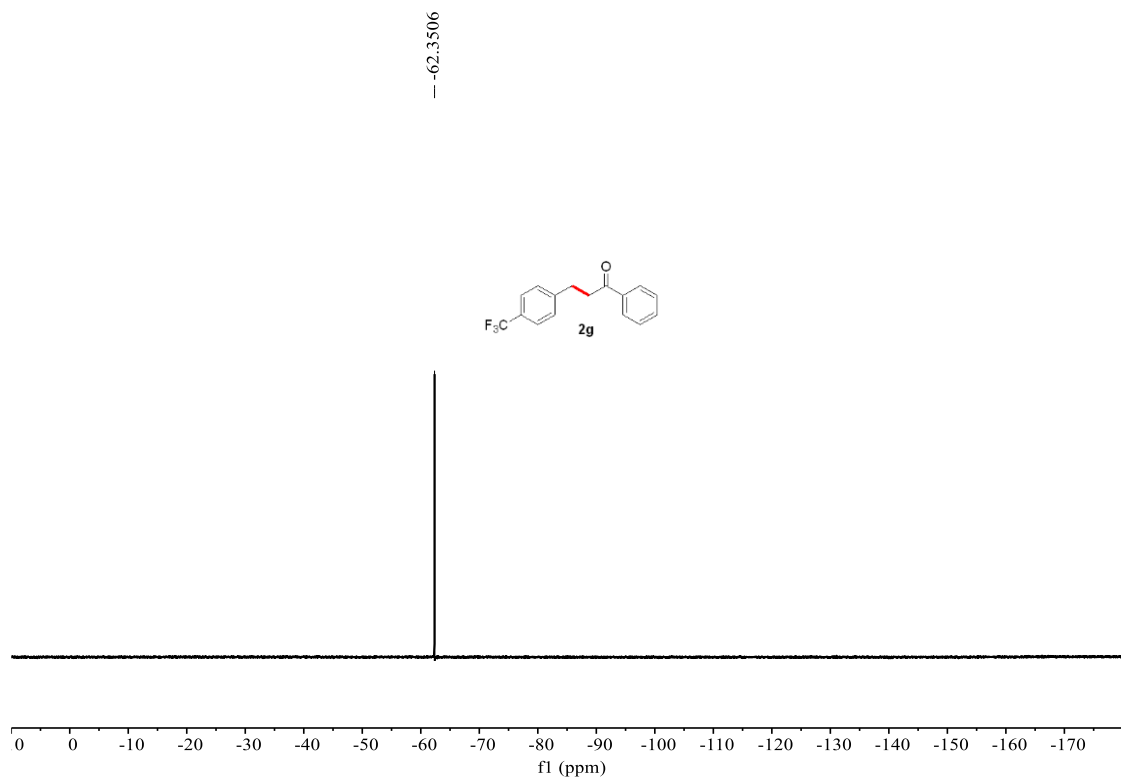
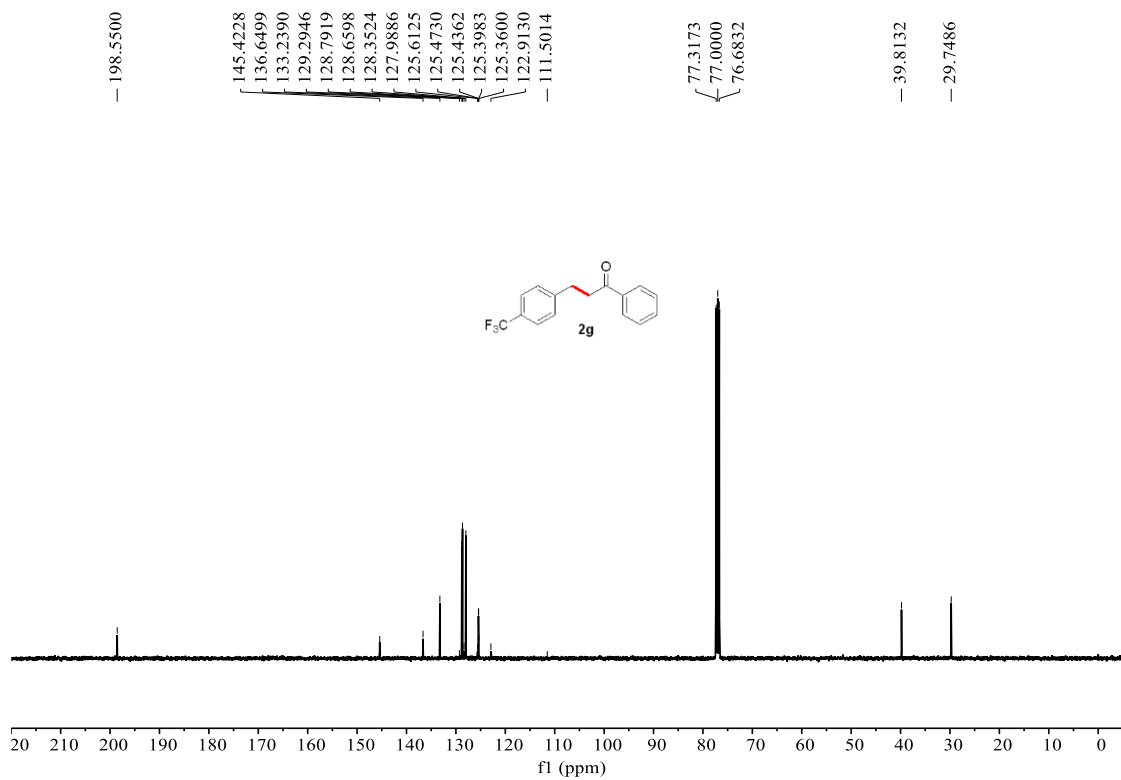


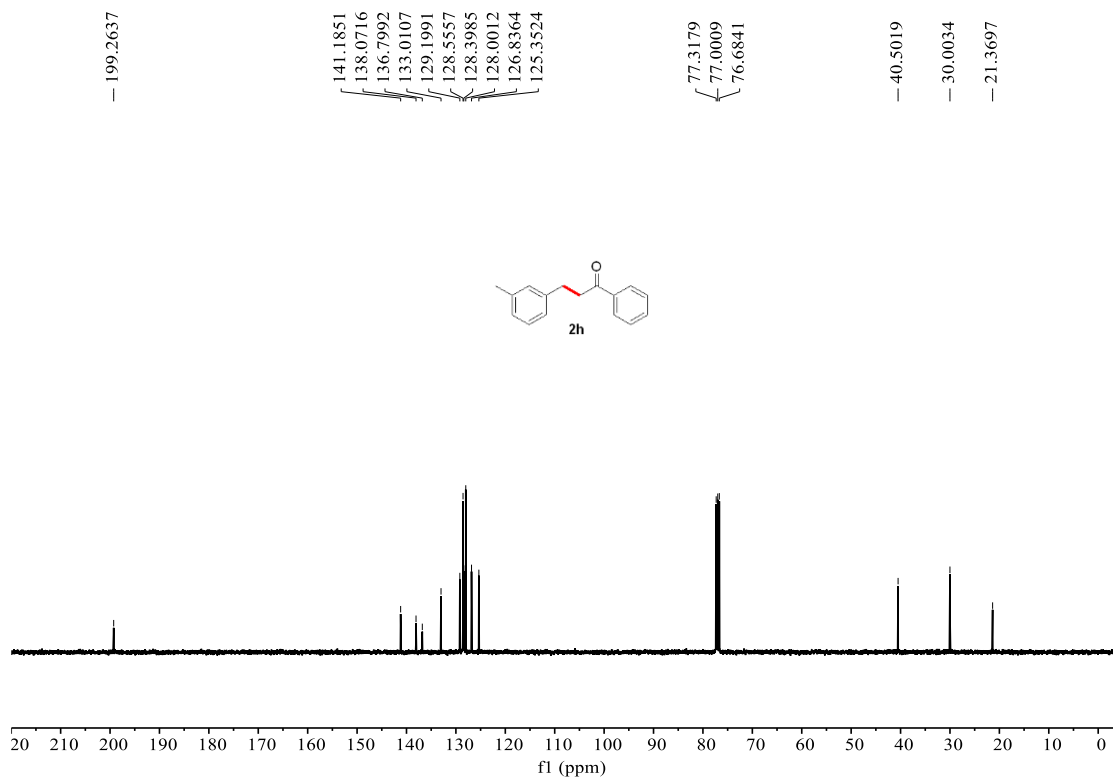
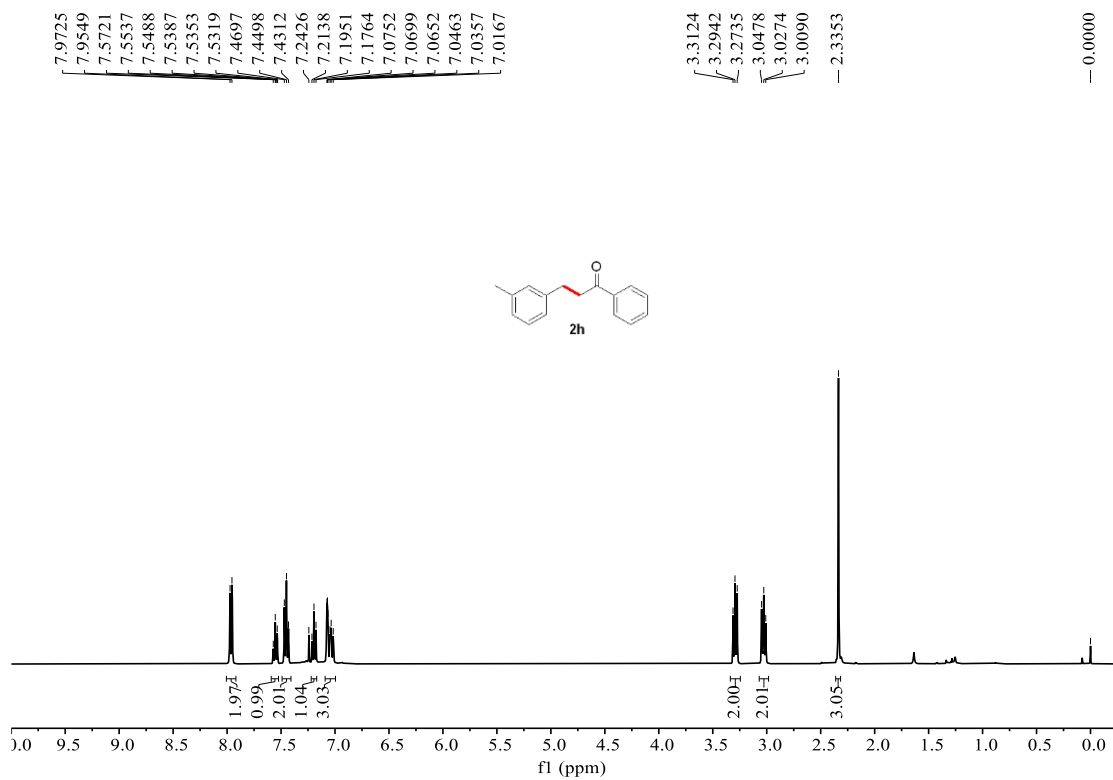
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2.04H
2.05H
2.09H

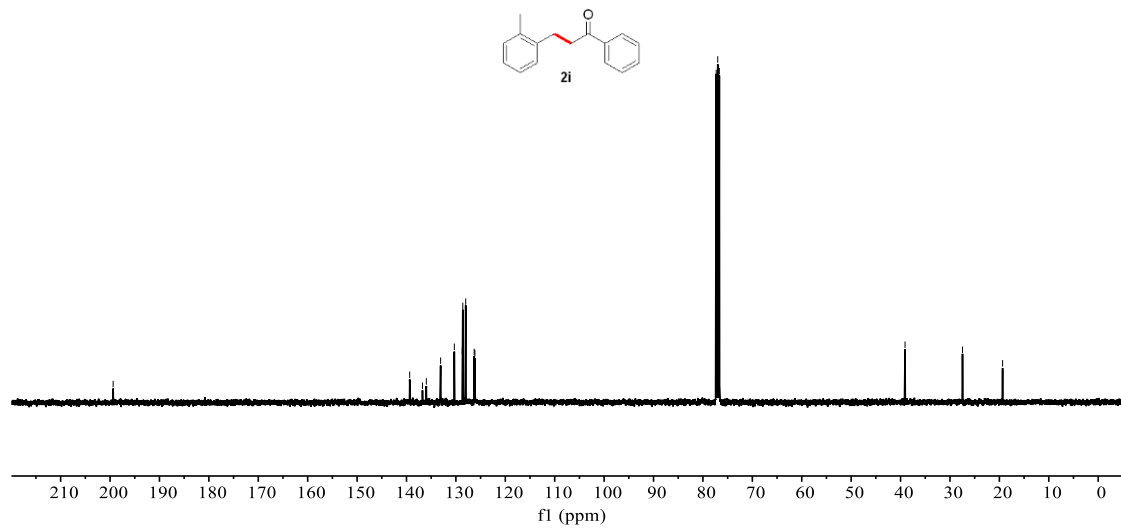
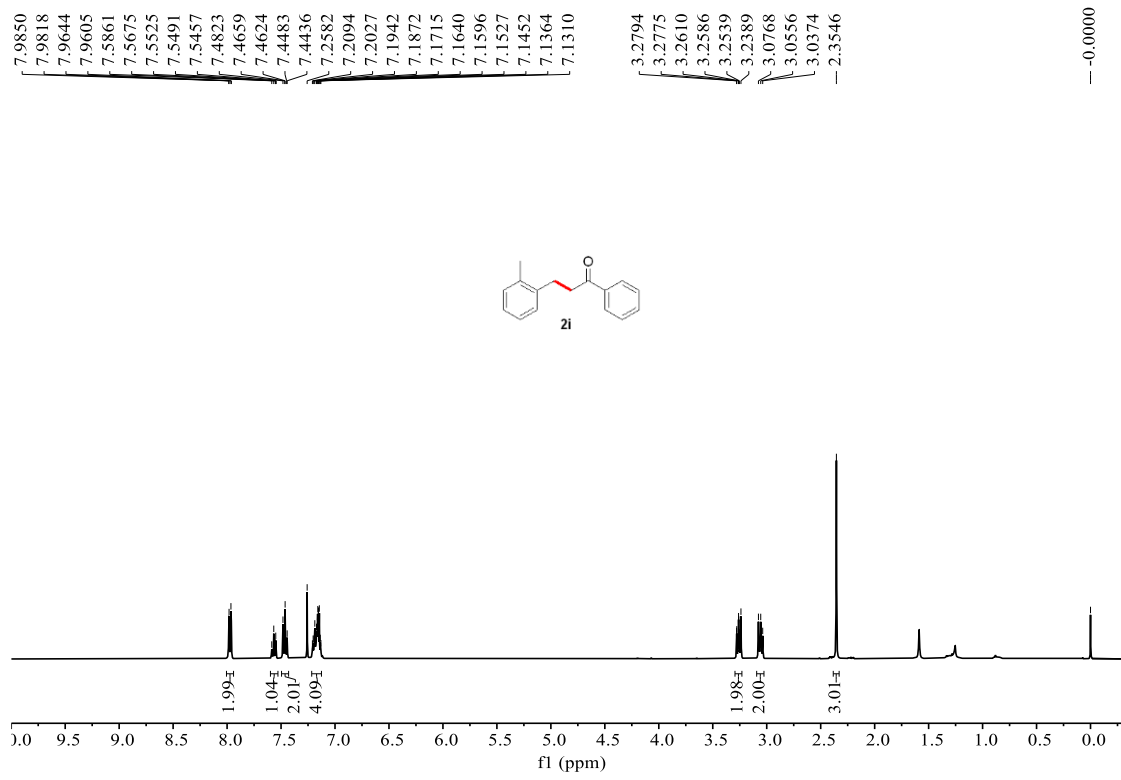
2.00H
2.02H

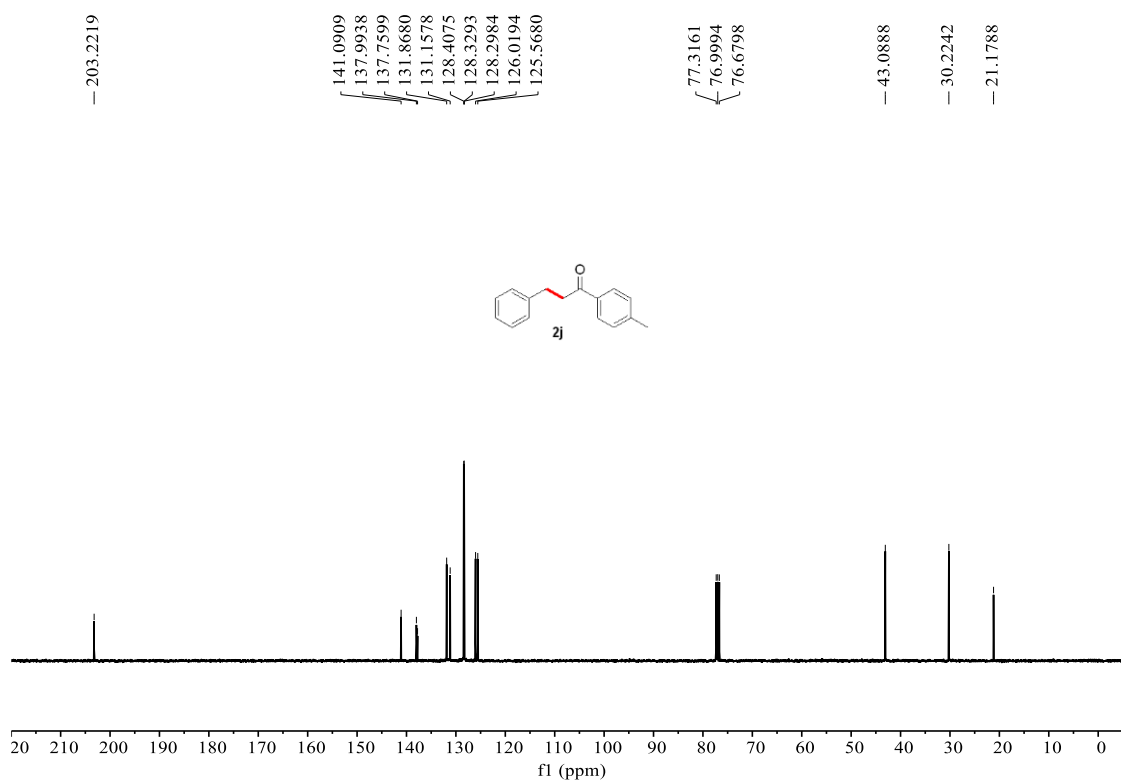
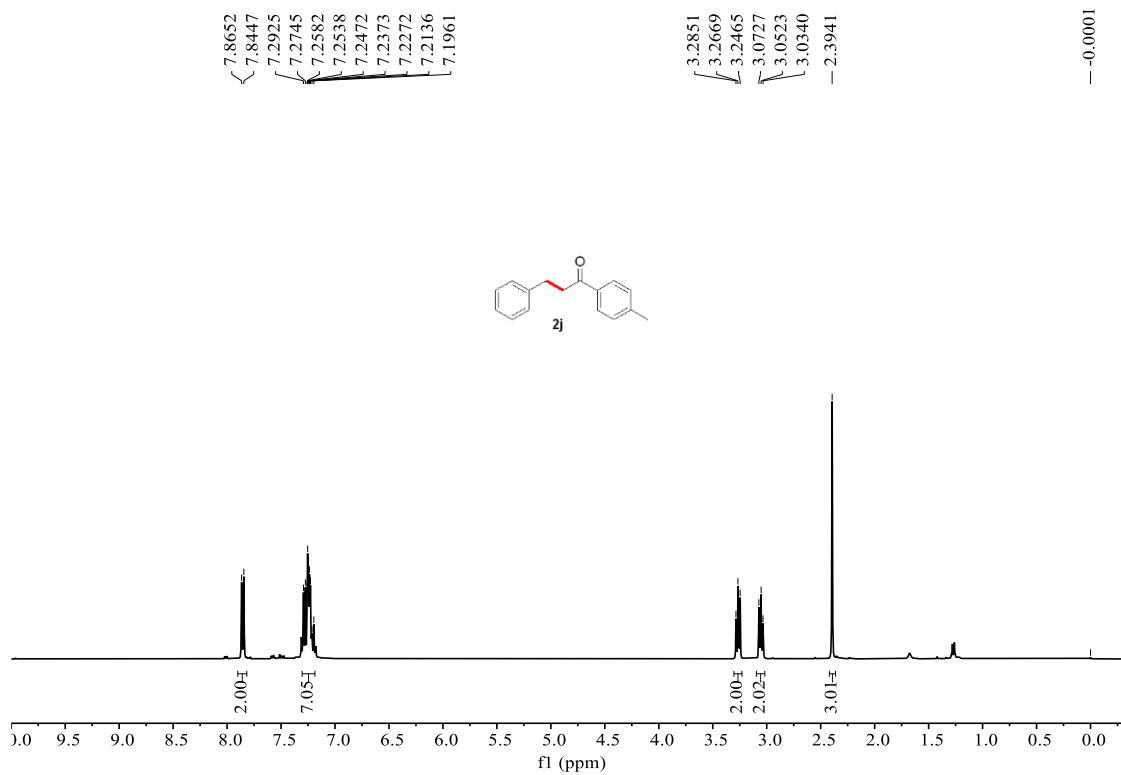


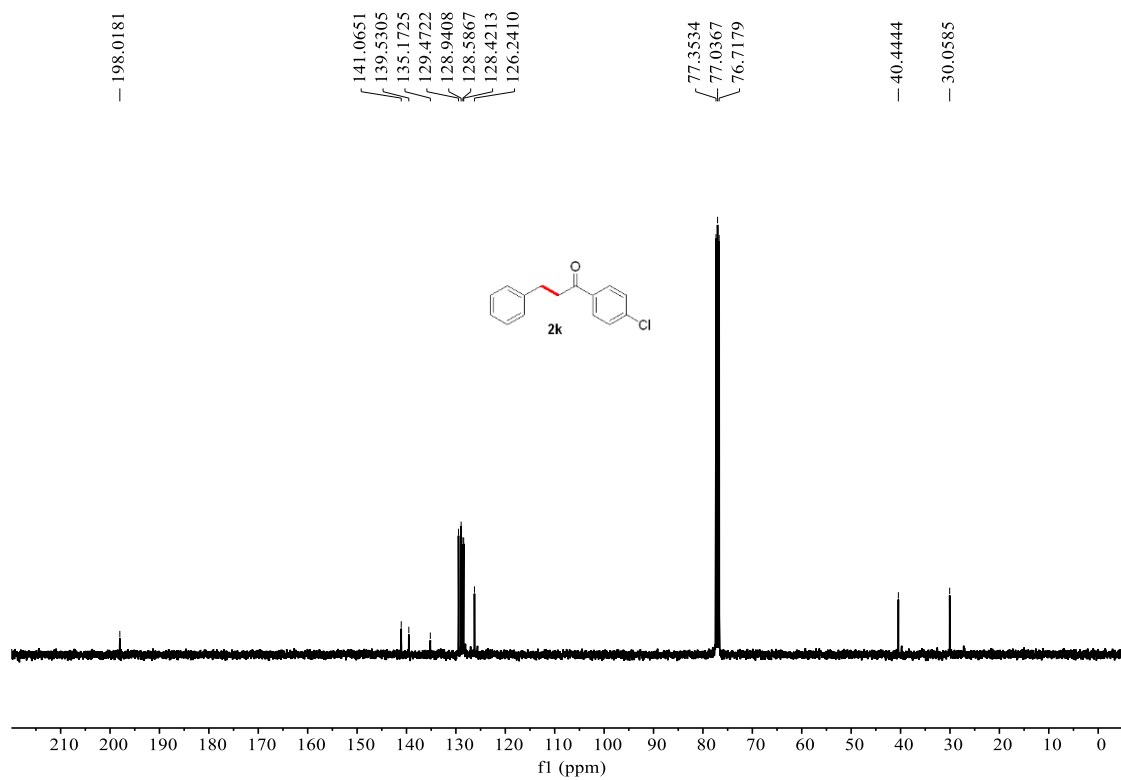
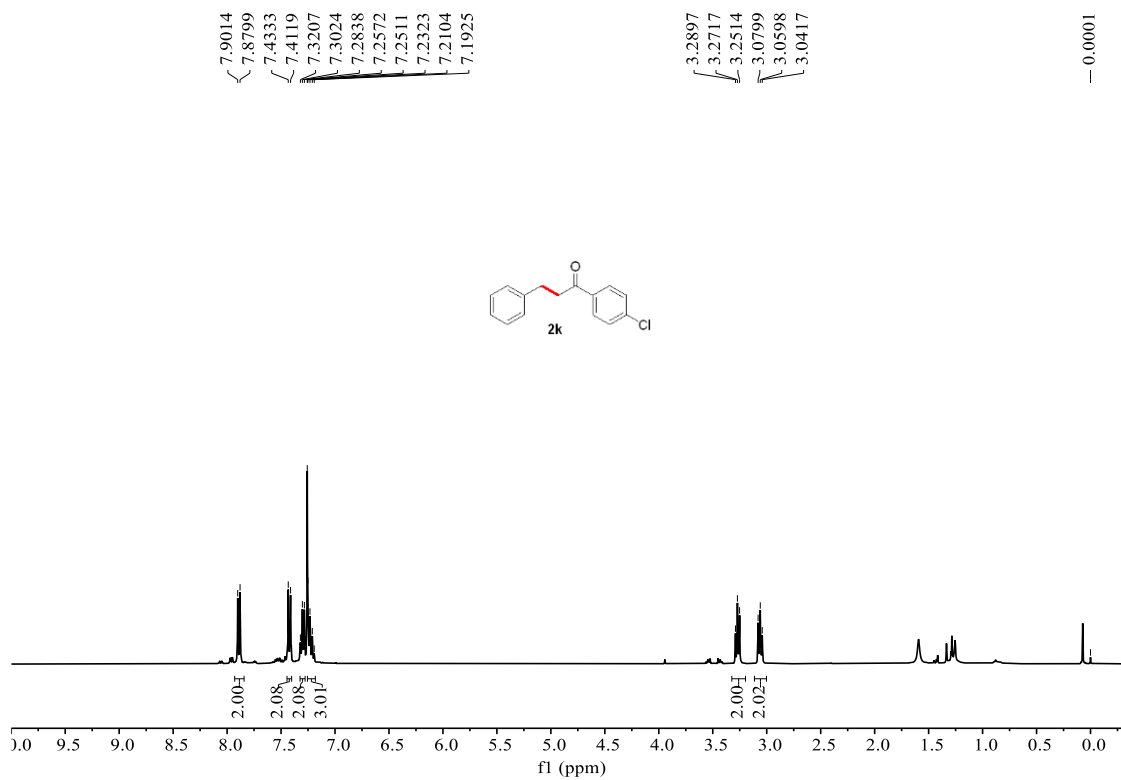


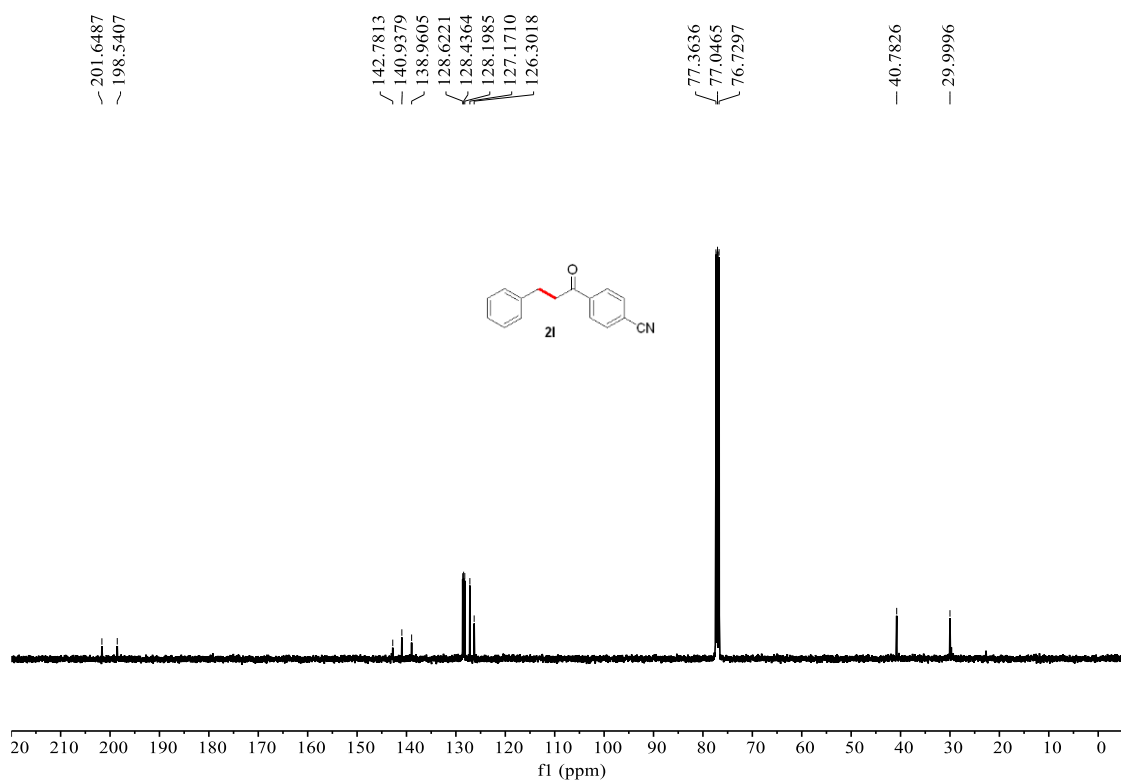
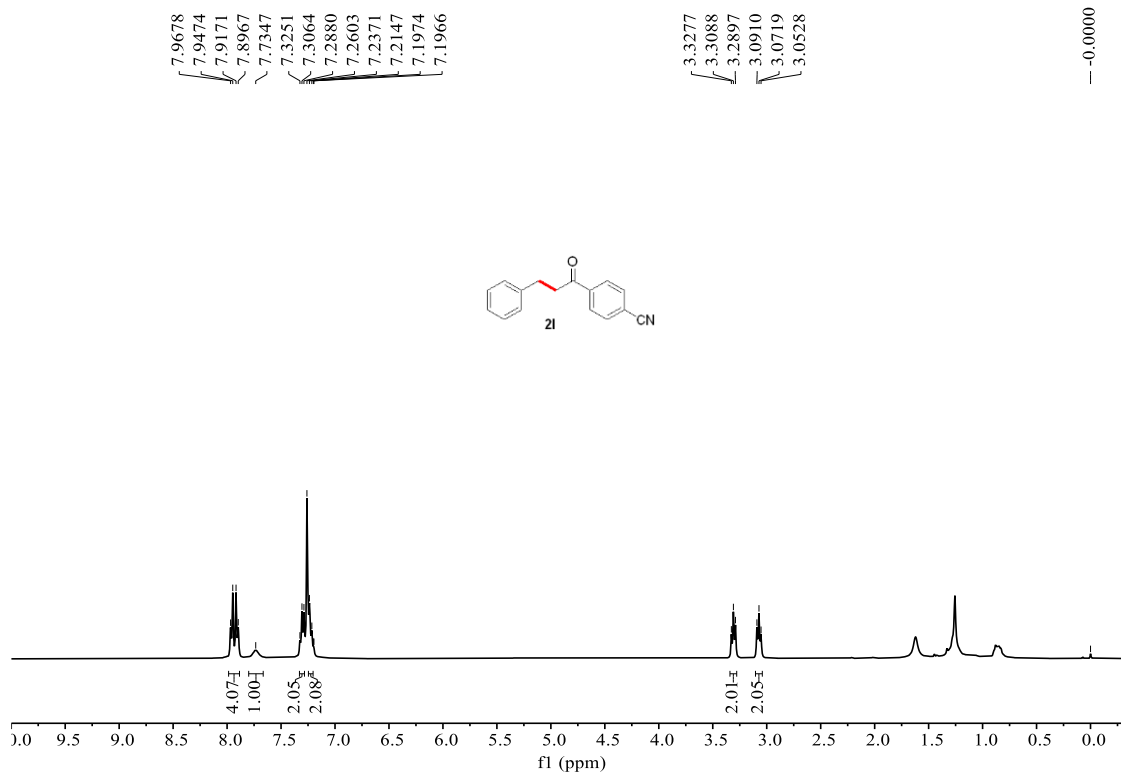


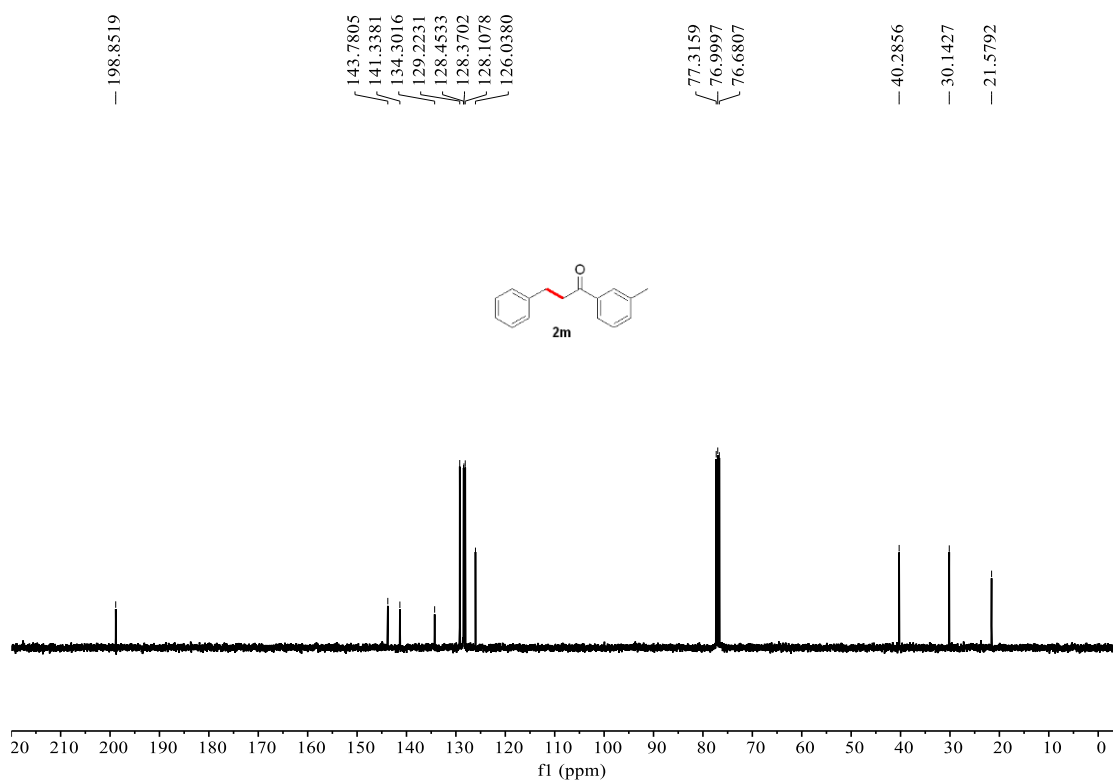
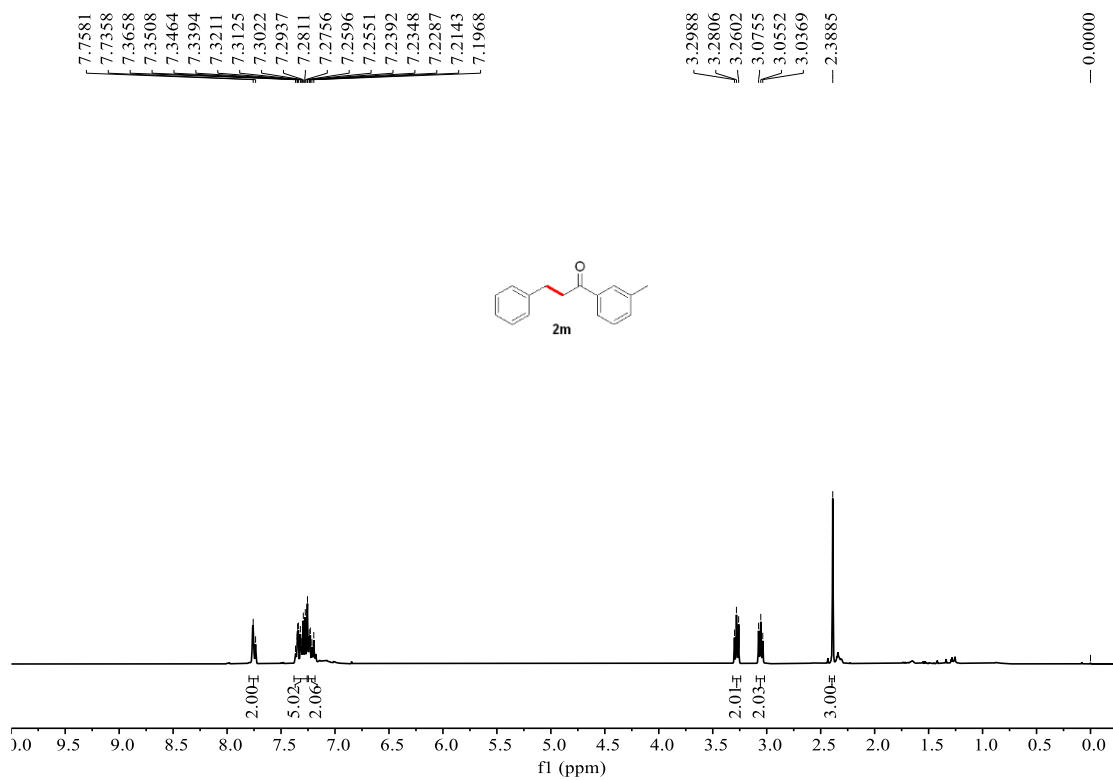


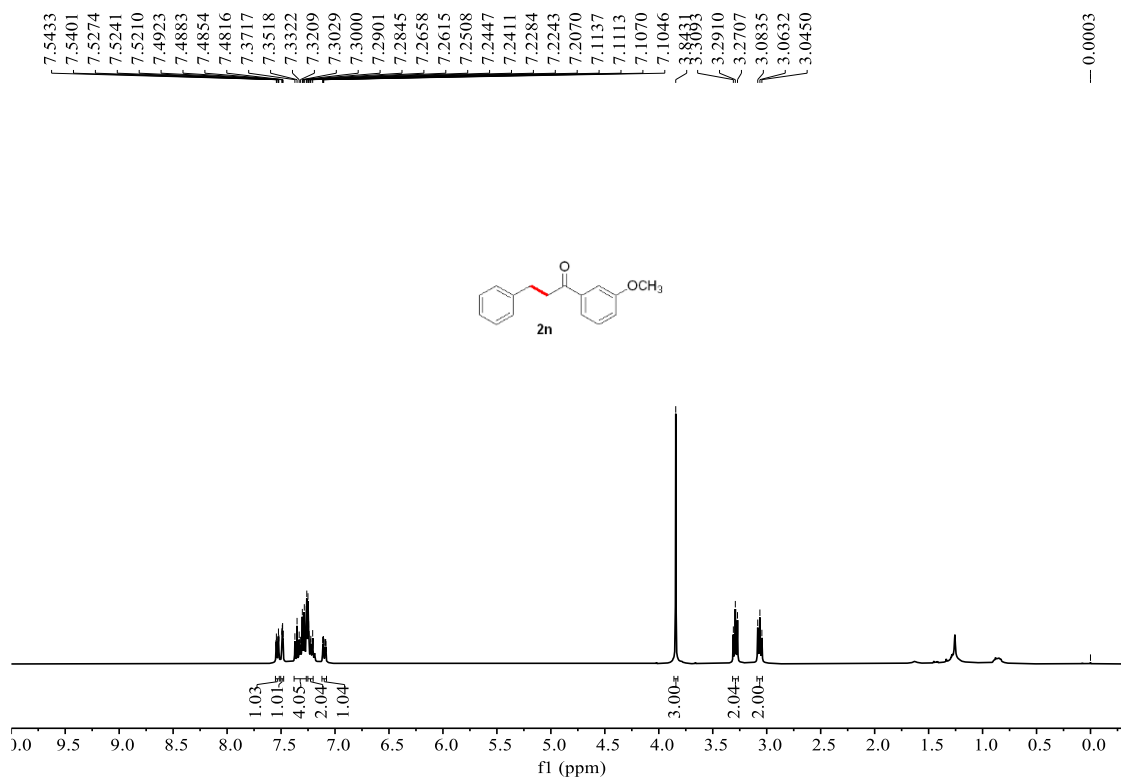












— 0.0003

