

## Supporting Information

### A Practical and Mild Method for One-Pot Selective Dehydroxylation of $\alpha$ -Hydroxyl Carbonyl Compounds

Xiaobo Xu,<sup>\*a</sup> Leyu Yan,<sup>a</sup> Zhi-Kai Zhang,<sup>b</sup> Bingqing Lu,<sup>a</sup> Zhuangwen Guo,<sup>a</sup> Mengyue Chen<sup>a</sup> and  
Zhong-Yan Cao<sup>\*b</sup>

<sup>a</sup>College of Chemistry and Pharmaceutical Engineering, Huanghuai University,  
Zhumadian 463000, China

<sup>b</sup>College of Chemistry and Chemical Engineering, Henan University, Kaifeng  
475004, China

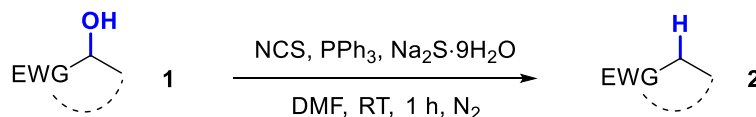
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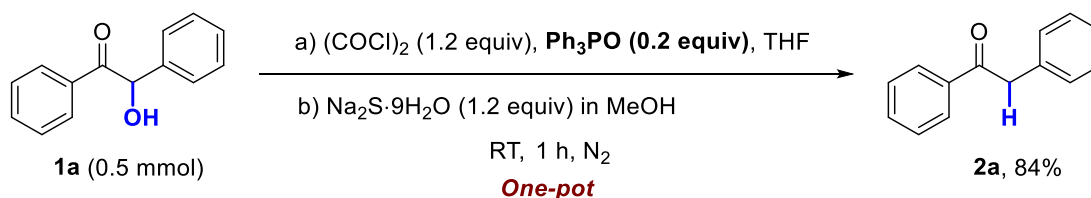
## 1. General Information

Unless otherwise noted, reactions were carried out in oven-dried glassware or sealed tube under ambient atmosphere. *N, N*-Dimethylformamide (DMF) was distilled from calcium hydride. Tetrahydrofuran (THF) was dried and distilled from sodium. Reactions were monitored by analytical thin-layer chromatography (TLC) on Merck silica gel 60 F<sub>254</sub> plates (0.25 mm), visualized by ultraviolet light (254 nm) or by staining with ceric ammonium molybdate. <sup>1</sup>H NMR spectra were obtained on a Bruker AVANCE 400 MHz spectrometer at ambient temperature. Data were reported as follows: chemical shift on the  $\delta$  scale using residual proton solvent as internal standard [ $\delta$  TMS: 0.00 ppm], multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublets), integration, and coupling constant (J) in hertz (Hz). <sup>13</sup>C NMR spectra were obtained with proton decoupling on a Bruker AVANCE (100 MHz) spectrometer and were reported in ppm with residual solvent for internal standard [ $\delta$  77.0 (CHCl<sub>3</sub>)].

## 2. Experimental procedures

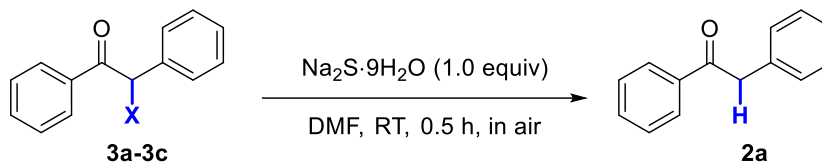


**General dihydroxylation procedure:** To a solution of  $\alpha$ -hydroxyl carbonyl compounds **1** (0.5 mmol) and PPh<sub>3</sub> (0.5 mmol) in dry DMF (2.0 mL) under nitrogen atmosphere at room temperature, followed by the addition of NCS (0.5 mmol). The resulting reaction mixture was stirred for 0.5 h and Na<sub>2</sub>S·9H<sub>2</sub>O (0.5 mmol) was then added to the mixture. After 0.5 h the reaction was completed, the reaction was quenched with H<sub>2</sub>O (5 mL), and the resulting mixture was extracted with ethyl acetate (3  $\times$  20 mL). The organic phases were combined, washed with saturated brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated in vacuo. The resultant residue was purified by flash chromatography (petroleum ether/ethyl acetate = 50/1–10/1) on silica gel to afford the desired product **2**.



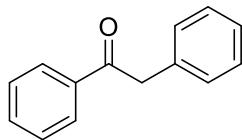
**General procedure for catalytic triphenylphosphine oxide mediated reaction:** At room temperature, benzoin **1a** (0.5 mmol, 106 mg) and Ph<sub>3</sub>PO (0.2 equiv, 28 mg) were added to a dry round-bottom flask, and the gas was purged three times under a nitrogen atmosphere. Then, the solvent dry THF (2 mL) and (COCl)<sub>2</sub> (1.2 equiv, 51  $\mu$ L) were added. The reaction was initiated at room temperature, and the mixture was stirred for 0.5 h, followed by the addition of Na<sub>2</sub>S·9H<sub>2</sub>O (1.2 equiv, 144 mg) in MeOH (1 mL). After 0.5 h the reaction was completed, the reaction was quenched with H<sub>2</sub>O (5 mL), and the resulting mixture was extracted with ethyl acetate (3  $\times$  20 mL). The organic phases were combined, washed with saturated brine, dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and

and concentrated in vacuo. The resultant residue was purified by flash chromatography (petroleum ether/ethyl acetate = 50/1–10/1) on silica gel to afford the desired product **2a** as a white solid (82 mg, 84% yield).

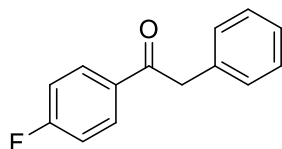


**General procedure for  $\text{Na}_2\text{S}$ -mediated dehalogenation:** To a solution of  $\alpha$ -halogenated carbonyl compounds **3a-3c** (0.5 mmol) and  $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$  (1.0 equiv, 0.5 mmol) in DMF (2 mL) at room temperature. The resulting reaction mixture was stirred for 0.5 h and  $\text{H}_2\text{O}$  (5 mL) was then added to the mixture. The resulting mixture was extracted with ethyl acetate ( $3 \times 20$  mL). The organic phases were combined, washed with saturated brine, dried over anhydrous  $\text{Na}_2\text{SO}_4$ , and concentrated in vacuo. The resultant residue was purified by flash chromatography (petroleum ether/ethyl acetate = 50/1–10/1) on silica gel to afford the desired product **2a** as a white solid.

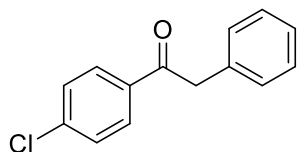
### 3. Characterization of Products



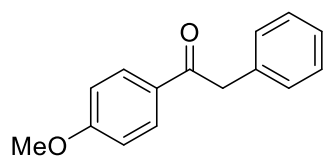
**1,2-Diphenylethan-1-one (2a)**[1, 2]: Known compound. Isolated yield 93% (91 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 8.08–8.06 (m, 2H), 7.59 (ddd,  $J$  = 6.7, 3.9, 1.2 Hz, 1H), 7.49 (dd,  $J$  = 10.5, 4.7 Hz, 2H), 7.40–7.34 (m, 2H), 7.33–7.28 (m, 3H), 4.33 (s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 197.5, 136.5, 134.4, 133.1, 129.4, 128.6, 128.5, 128.5, 126.8, 45.4.



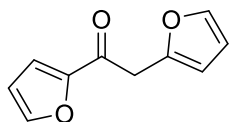
**1-(4-Fluorophenyl)-2-phenylethan-1-one (2b)**[2]: Known compound. Isolated yield 88% (94 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 8.09–8.06 (m, 2H), 7.37 (dd,  $J$  = 8.0, 6.8 Hz, 2H), 7.30 (d,  $J$  = 6.9 Hz, 3H), 7.15 (t,  $J$  = 8.6 Hz, 2H), 4.29 (s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 195.9, 166.9, 164.4, 134.3, 132.9, 132.9, 131.2, 131.1, 129.3, 128.6, 126.9, 115.7, 115.5, 45.4.



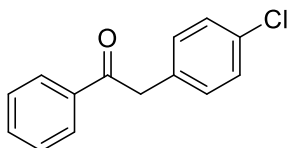
**1-(4-Chlorophenyl)-2-phenylethan-1-one (2c)**[1, 2]: Known compound. Isolated yield 90% (104 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 7.99–7.96 (m, 2H), 7.46–7.44 (m, 2H), 7.38–7.35 (m, 2H), 7.31–7.28 (m, 3H), 4.28 (s, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 196.4, 139.5, 134.8, 134.1, 133.0, 129.3, 128.9, 128.7, 127.0, 45.6.



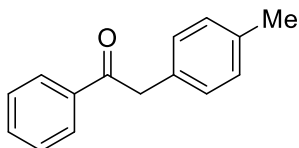
**1-(4-Methoxyphenyl)-2-phenylethan-1-one (2d)**[2]: Known compound. Isolated yield 95% (107 mg).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 8.05–8.02 (m, 2H), 7.37–7.27 (m, 5H), 6.97–6.95 (m, 2H), 4.26 (s, 2H), 3.87 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 196.1, 163.4, 134.9, 130.8, 129.5, 129.3, 128.5, 126.7, 113.7, 55.3, 45.1.



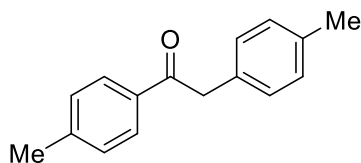
**1,2-Di(furan-2-yl)ethan-1-one (2e)**[3]: Known compound. Isolated yield 84% (74 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 8.60 (d,  $J = 7.9$  Hz, 1H), 8.28 (d,  $J = 8.1$  Hz, 1H), 8.03 (t,  $J = 7.6$  Hz, 1H), 7.71 (dt,  $J = 8.6, 4.0$  Hz, 3H), 7.42 (dd,  $J = 5.3, 1.9$  Hz, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 145.08, 144.56, 143.84, 135.36, 131.54, 131.41, 129.25, 126.77, 126.28, 125.30.



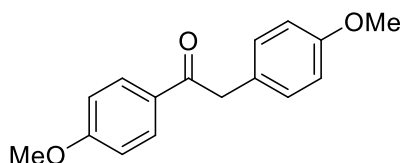
**2-(4-Chlorophenyl)-1-phenylethan-1-one (2f)**[2]: Known compound. Isolated yield 91% (105 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 8.04–8.02 (m, 2H), 7.62–7.58 (m, 1H), 7.50 (dd,  $J = 8.4, 7.0$  Hz, 2H), 7.34–7.32 (m, 2H), 7.23–7.21 (m, 2H), 4.29 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 197.0, 136.3, 133.3, 132.9, 132.8, 130.9, 128.7, 128.7, 128.4, 44.6.



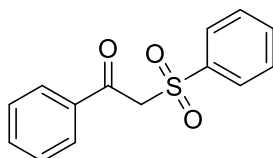
**1-Phenyl-2-(p-tolyl)ethan-1-one (2g)**[2]: Known compound. Isolated yield 92% (97 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 8.07–8.05 (m, 2H), 7.59–7.57 (m, 1H), 7.51–7.47 (m, 2H), 7.23–7.17 (m, 4H), 4.29 (s, 2H), 2.37 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 197.7, 136.5, 136.4, 133.0, 131.7, 129.3, 129.2, 128.5, 45.0, 21.0.



**1,2-Di-p-tolyethan-1-one (2h)**[1]: Known compound. Isolated yield 94% (105 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.83–7.81 (m, 2H), 7.15 (d,  $J = 8.2$  Hz, 2H), 7.08–7.02 (m, 4H), 4.12 (s, 2H), 2.30 (s, 3H), 2.23 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 197.4, 143.8, 136.3, 134.1, 131.6, 129.3, 129.2, 129.2, 128.7, 45.0, 21.6, 21.0.

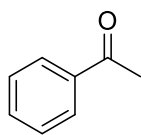


**1,2-Bis(4-methoxyphenyl)ethan-1-one (2i)**[1]: Known compound. Isolated yield 90% (115 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 8.03–8.01 (m, 2H), 7.22–7.20 (m, 2H), 6.95–6.94 (m, 2H), 6.90–6.87 (m, 2H), 4.19 (s, 2H), 3.86 (s, 3H), 3.79 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 196.4, 163.3, 158.3, 130.7, 130.2, 129.5, 126.8, 113.9, 113.6, 55.3, 55.0, 44.2.

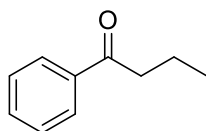


**1-Phenyl-2-(phenylsulfonyl)ethan-1-one (2j)**[4]: Known compound. Isolated yield 82% (107 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.94–7.89 (m, 4H), 7.67–7.61 (m, 2H),

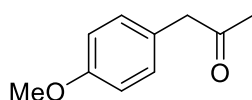
7.59–7.48 (m, 2H), 7.46 (t,  $J = 7.9$  Hz, 2H), 4.77 (s, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 187.9, 138.7, 135.6, 134.2, 134.1, 129.1, 129.0, 128.7, 128.4, 63.2.



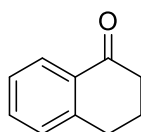
**Acetophenone (2k)**[3, 5]: Known compound. Isolated yield 65% (39 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.93–7.91 (m, 2H), 7.54–7.50 (m, 1H), 7.44–7.40 (m, 2H), 2.55 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 197.6, 136.7, 132.7, 128.2, 127.9, 26.2.



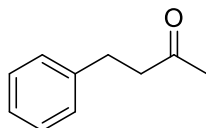
**1-Phenylbutan-1-one (2l)**[5]: Known compound. Isolated yield 94% (70 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.98–7.96 (m, 2H), 7.57–7.53 (m, 1H), 7.48–7.44 (m, 2H), 2.95 (t,  $J = 7.3$  Hz, 2H), 1.78 (p,  $J = 7.4$  Hz, 2H), 1.02 (t,  $J = 7.4$  Hz, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 200.2, 137.0, 132.7, 128.4, 127.9, 40.4, 17.7, 13.8.



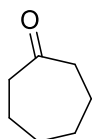
**1-(4-Methoxyphenyl)propan-2-one (2m)**[1]: Known compound. Isolated yield 95% (78 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.13–7.10 (m, 2H), 6.88–6.86 (m, 2H), 3.78 (s, 3H), 3.62 (s, 2H), 2.13 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 206.4, 158.4, 130.1, 126.1, 113.9, 54.9, 49.7, 28.8.



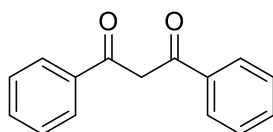
**3,4-Dihydronaphthalen-1(2H)-one (2n)**[6]: Known compound. Isolated yield 88% (64 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.99 (dd,  $J = 7.8, 1.5$  Hz, 1H), 7.41 (td,  $J = 7.5, 1.5$  Hz, 1H), 7.25 (t,  $J = 7.6$  Hz, 1H), 7.20 (d,  $J = 7.7$  Hz, 1H), 2.91 (t,  $J = 6.1$  Hz, 2H), 2.60 (dd,  $J = 7.3, 5.8$  Hz, 2H), 2.08 (p,  $J = 6.4$  Hz, 2H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 197.9, 144.2, 133.1, 132.3, 128.5, 126.8, 126.3, 38.8, 29.4, 23.0.



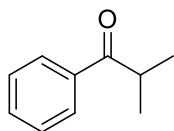
**4-Phenylbutan-2-one (2o)**[7]: Known compound. Isolated yield 86% (64 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.33–7.30 (m, 2H), 7.23 (td,  $J = 5.9, 1.9$  Hz, 3H), 2.93 (t,  $J = 7.6$  Hz, 2H), 2.80–2.76 (m, 2H), 2.16 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 207.6, 140.8, 128.3, 128.1, 125.9, 44.9, 29.8, 29.5.



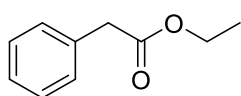
**Cycloheptanone (2p)**[8]: Known compound. Isolated yield 75% (42 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 2.41–2.38 (m, 4H), 1.62–1.56 (m, 4H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 214.9, 43.5, 30.1, 24.0.



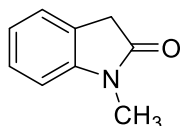
**1,3-Diphenylpropane-1,3-dione (2q)**[9]: Known compound. Isolated yield 89% (100 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 8.04–8.02 (m, 4H), 7.61–7.54 (m, 2H), 7.52 (dd,  $J = 8.3, 6.5$  Hz, 4H), 6.90 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 194.3, 185.7, 135.5, 132.4, 128.6, 127.1, 93.1.



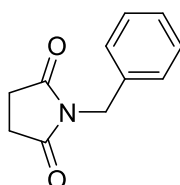
**2-Methyl-1-phenylpropan-1-one (2r)**[3]: Known compound. Isolated yield 73% (54 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.96–7.94 (m, 2H), 7.54–7.50 (m, 1H), 7.48–7.42 (m, 2H), 3.53 (dq,  $J = 13.7, 6.8$  Hz, 1H), 1.21 (d,  $J = 7.0$  Hz, 6H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 204.1, 136.0, 136.2, 132.6, 128.4, 128.1, 35.1, 18.9.



**Ethyl 2-phenylacetate (2s)**[10]: Known compound. Isolated yield 81% (66 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.39 (dt,  $J = 6.8, 1.4$  Hz, 1H), 7.35–7.31 (m, 4H), 4.20 (q,  $J = 7.1$  Hz, 2H), 3.66 (s, 2H), 1.30 (t,  $J = 7.1$  Hz, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 171.3, 134.0, 129.0, 128.3, 126.8, 60.6, 41.2, 13.9.

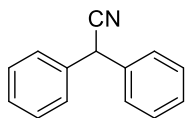


**1-Methylindolin-2-one (2t)**[11]: Known compound. Isolated yield 87% (64 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.22–7.14 (m, 2H), 6.95 (td,  $J = 7.6, 1.1$  Hz, 1H), 6.73 (d,  $J = 7.8$  Hz, 1H), 3.42 (s, 2H), 3.12 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$  (ppm) 175.0, 145.1, 127.8, 124.4, 124.2, 122.2, 108.0, 35.6, 26.0.

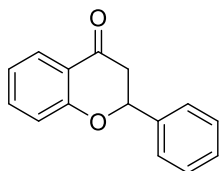


**1-Benzylpyrrolidine-2,5-dione (2u)**[12]: Known compound. Isolated yield 76% (72 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$  (ppm) 7.39–7.38 (m, 2H), 7.33–7.27 (m, 3H), 4.64

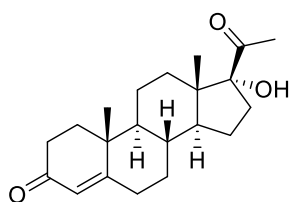
(s, 2H), 2.67 (s, 4H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$ (ppm) 176.7, 135.7, 128.7, 128.5, 127.8, 42.2, 28.0.



**2,2-Diphenylacetonitrile (2v)**[13]: Known compound. Isolated yield 78% (75 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$ (ppm) 7.42–7.38 (m, 10H), 5.19 (s, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$ (ppm) 135.8, 129.0, 128.1, 127.6, 119.6, 42.4.



**Flavanone (2w)**[14]: Known compound. Isolated yield 87% (97 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$ (ppm) 7.97 (dd,  $J$  = 8.0, 1.8 Hz, 1H), 7.56–7.42 (m, 6H), 7.10–7.07 (m, 2H), 5.51 (dd,  $J$  = 13.4, 2.9 Hz, 1H), 3.12 (dd,  $J$  = 16.9, 13.3 Hz, 1H), 2.92 (dd,  $J$  = 16.9, 2.9 Hz, 1H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):  $\delta$ (ppm) 191.9, 161.5, 138.7, 136.1, 128.8, 128.7, 127.0, 126.1, 121.6, 120.9, 118.1, 79.5, 44.6.



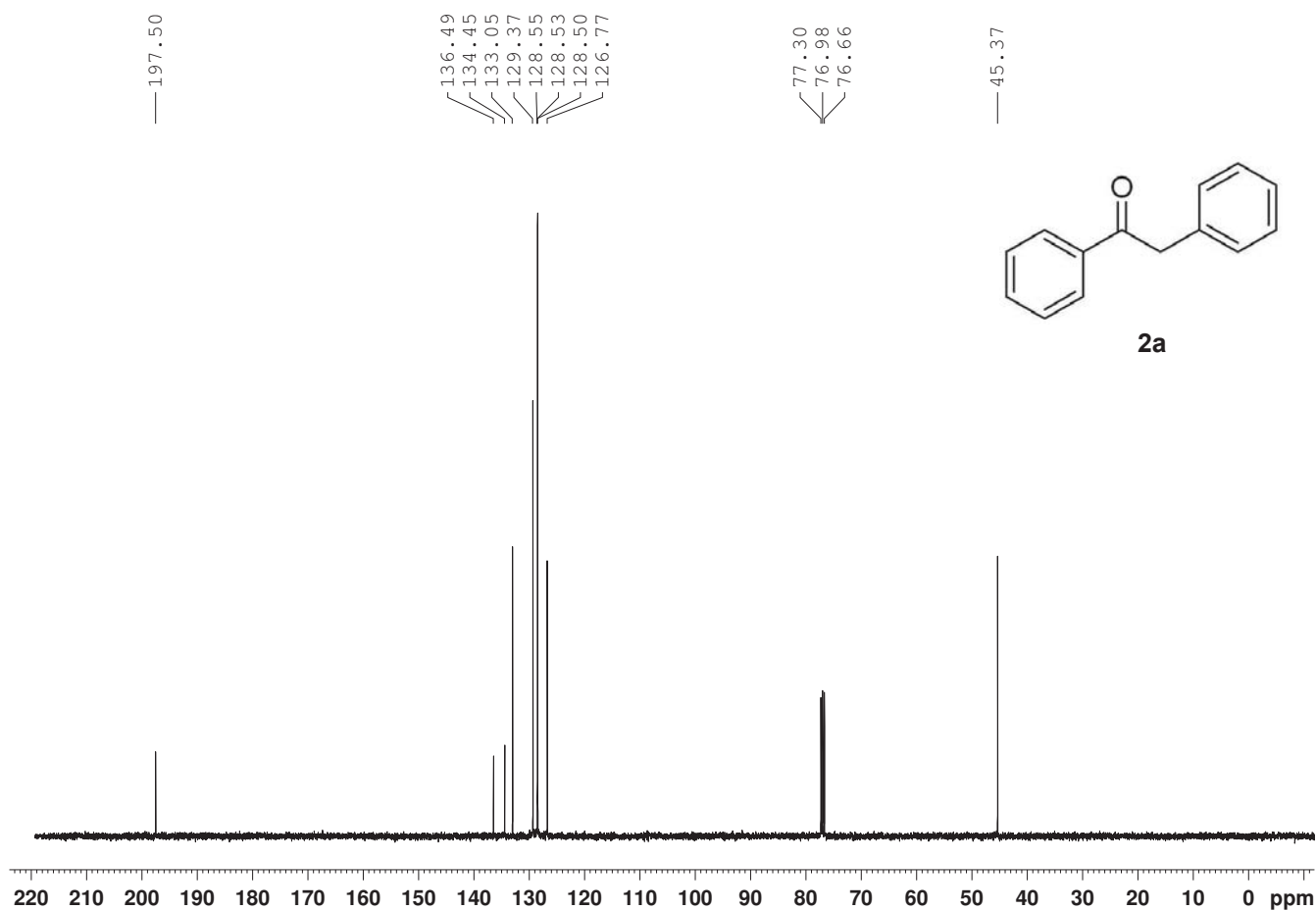
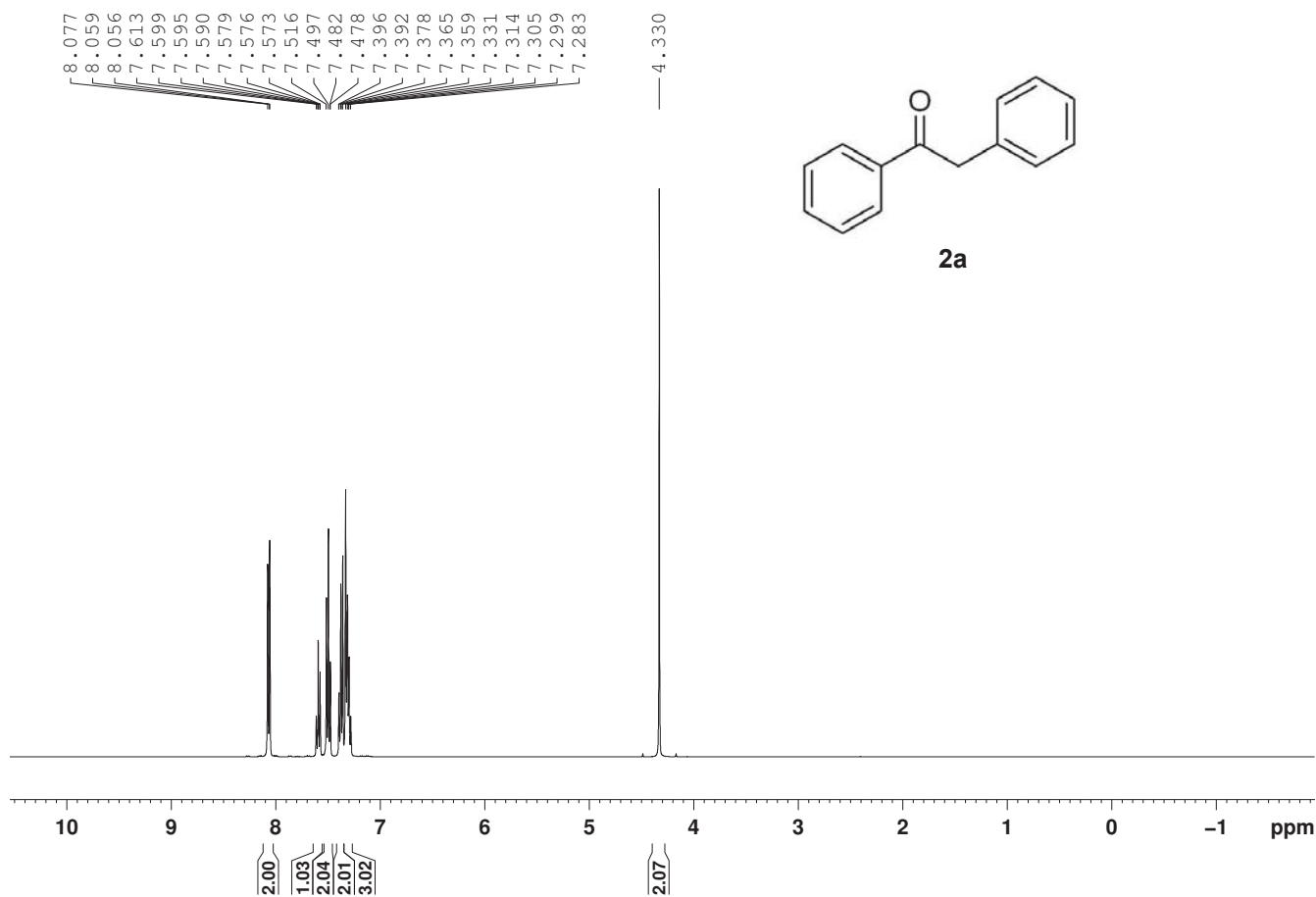
**Hydroxyprogesterone (2x)**[15]: Known compound. Isolated yield 53% (87 mg).  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):  $\delta$ (ppm) 5.70 (d,  $J$  = 1.8 Hz, 1H), 3.00 (s, 1H), 2.66 (ddd,  $J$  = 14.7, 11.4, 2.9 Hz, 1H), 2.39–2.26 (m, 4H), 2.24 (s, 3H), 2.02 (ddd,  $J$  = 13.4, 5.0, 3.3 Hz, 1H), 1.99–1.53 (m, 8H), 1.43–1.30 (m, 3H), 1.16 (s, 3H), 0.95–0.94 (m, 2H), 0.71 (s, 3H);  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz): 211.5, 199.6, 171.2, 171.2, 123.8, 89.8, 53.2, 49.9, 47.9, 38.5, 35.6, 35.4, 33.8, 33.3, 32.7, 31.9, 30.0, 27.7, 23.8, 20.4, 17.3, 15.2.

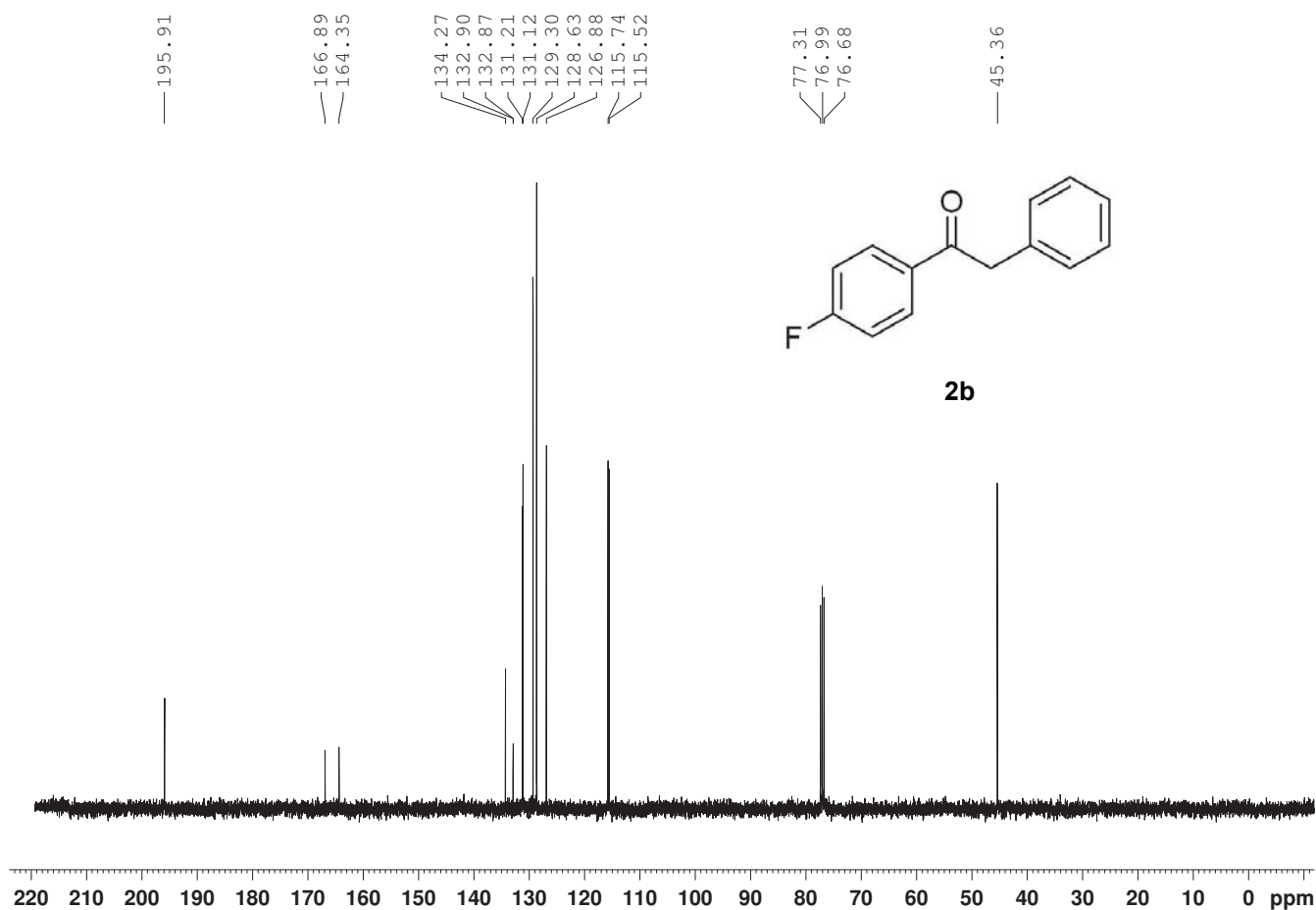
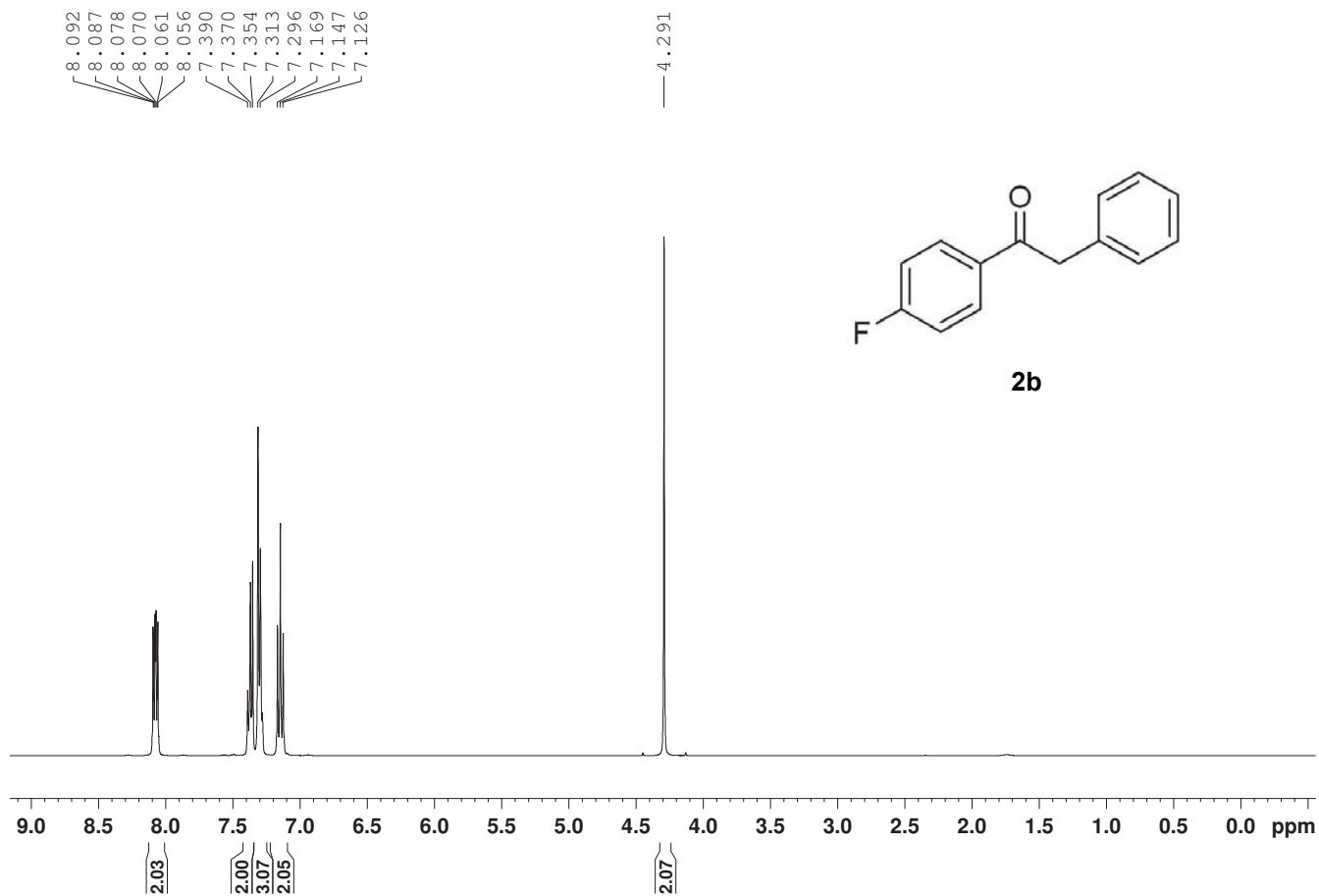
## 4. References

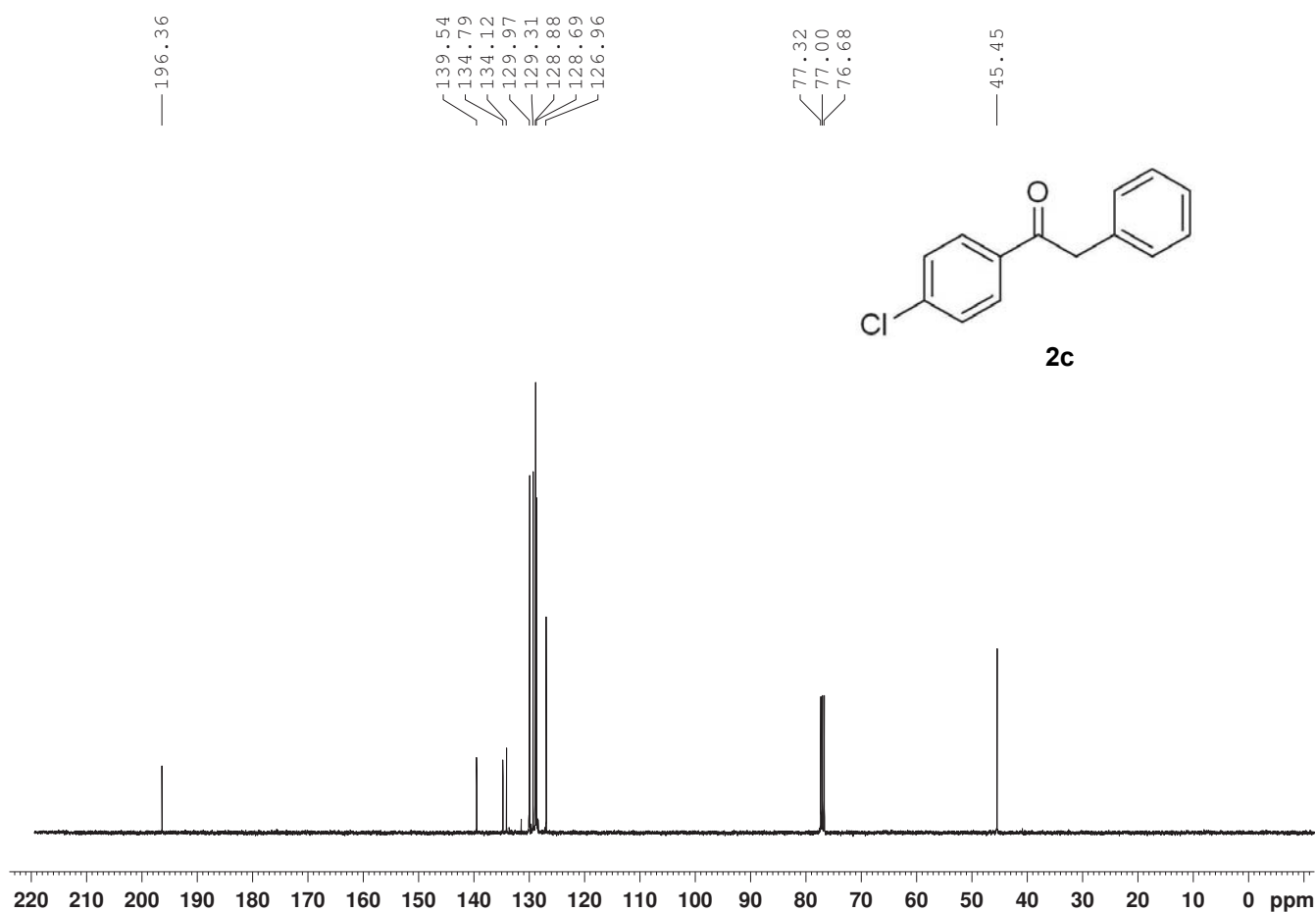
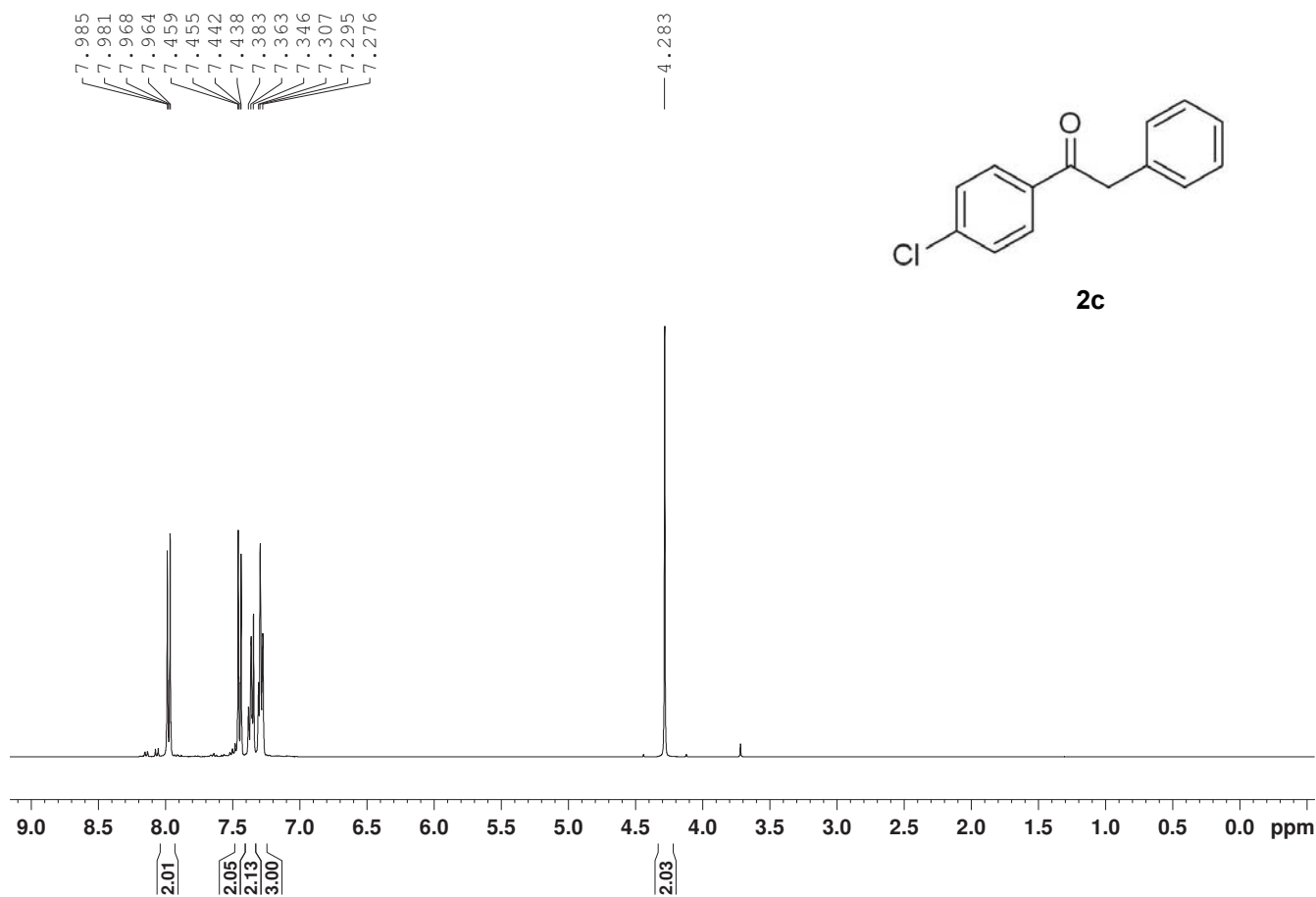
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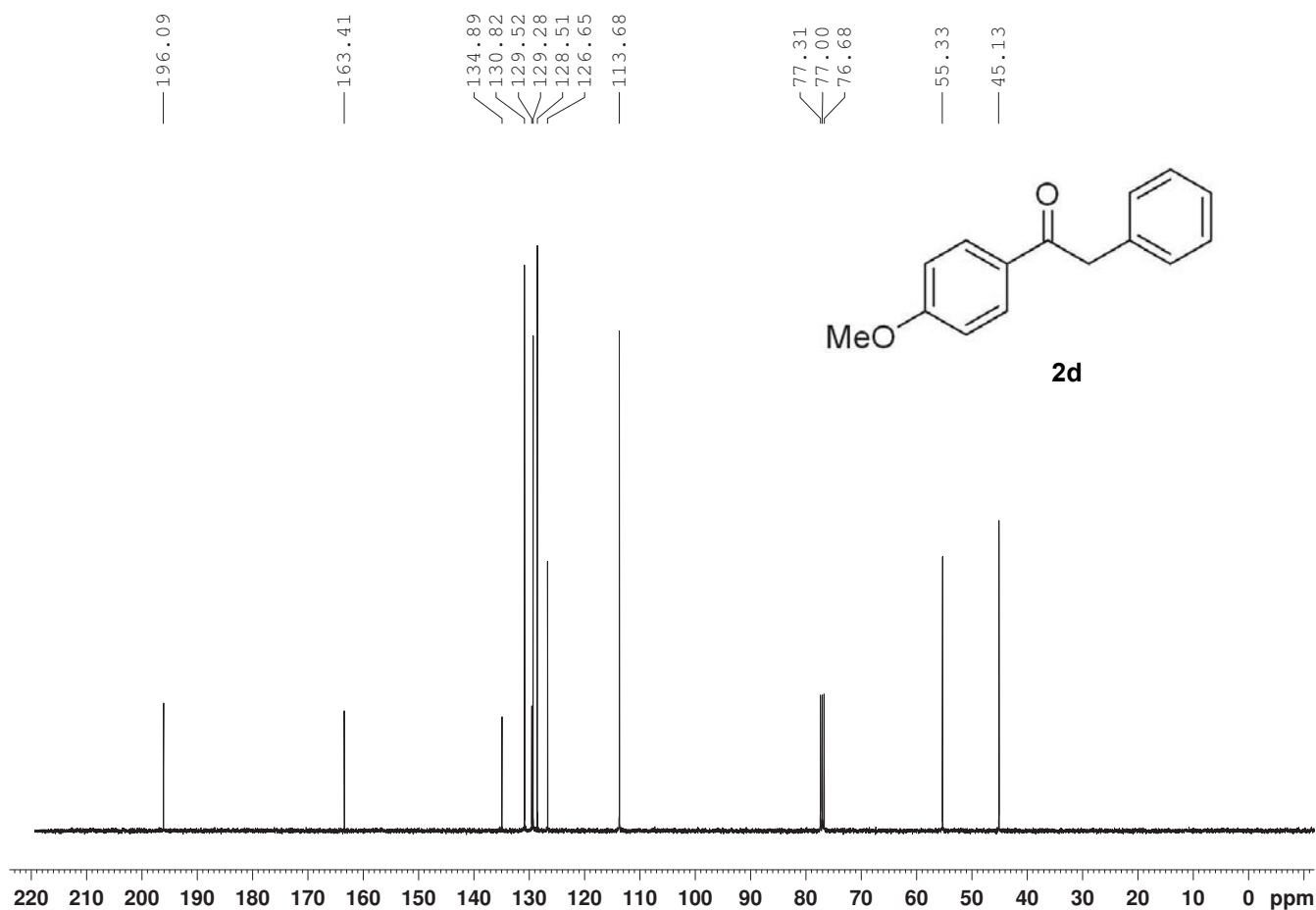
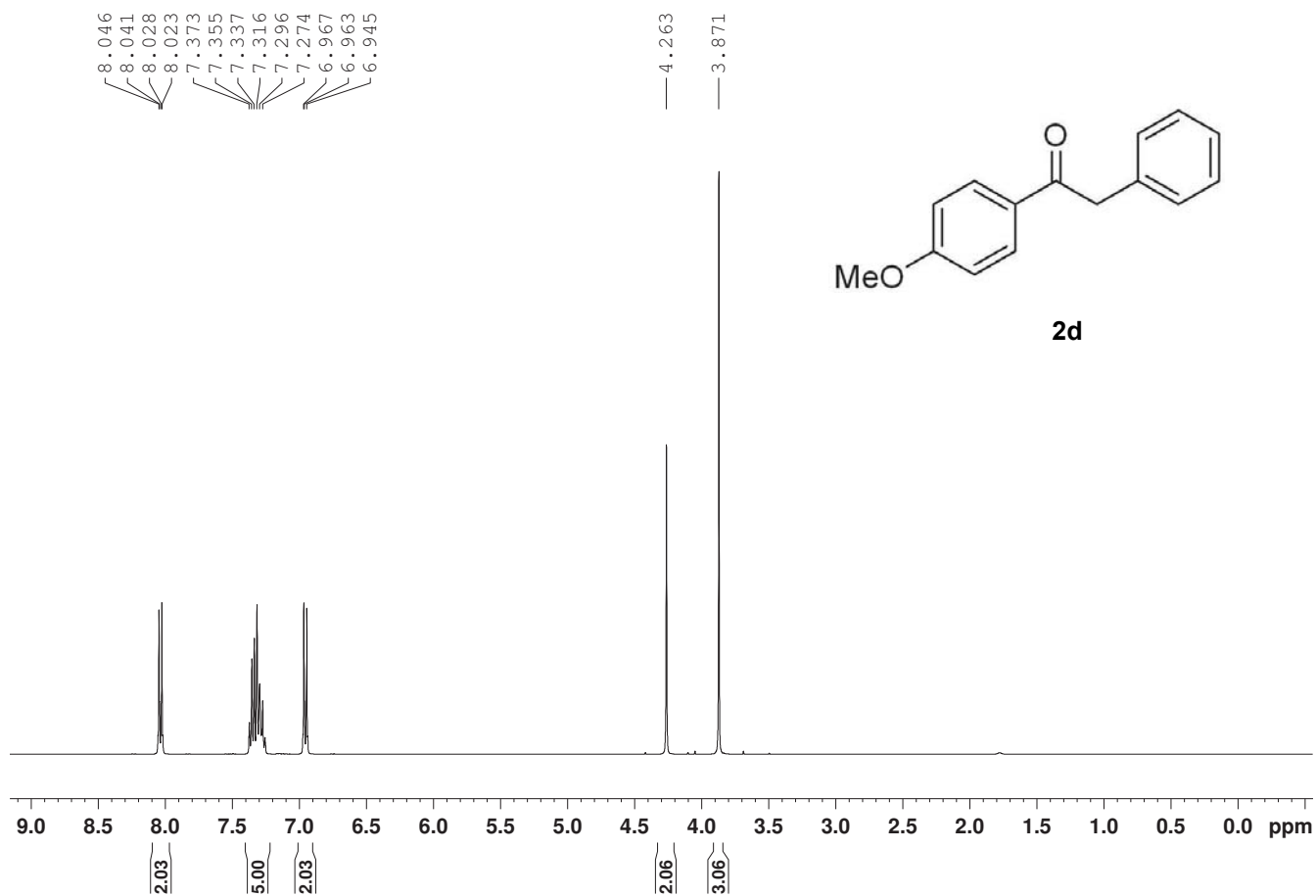
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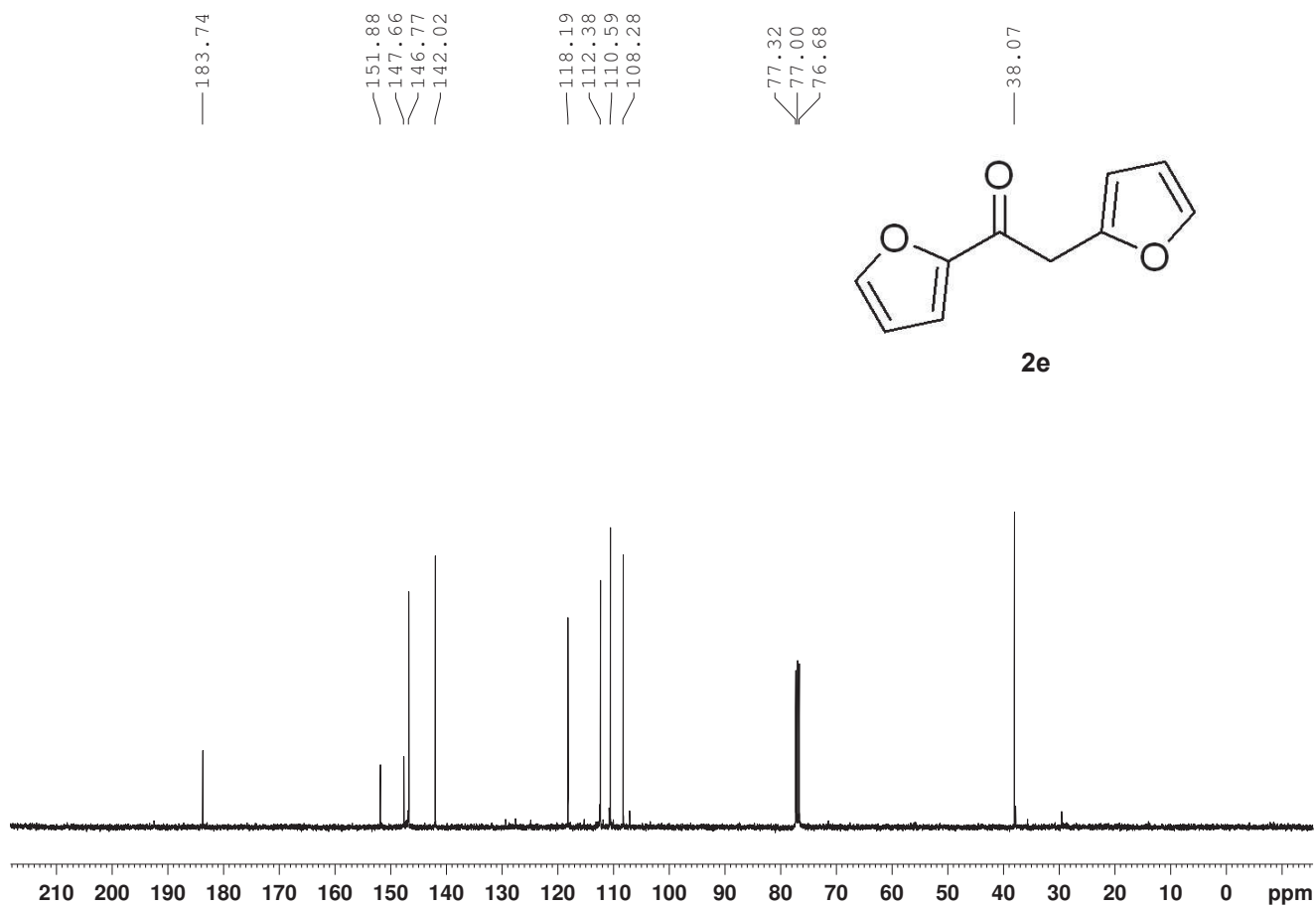
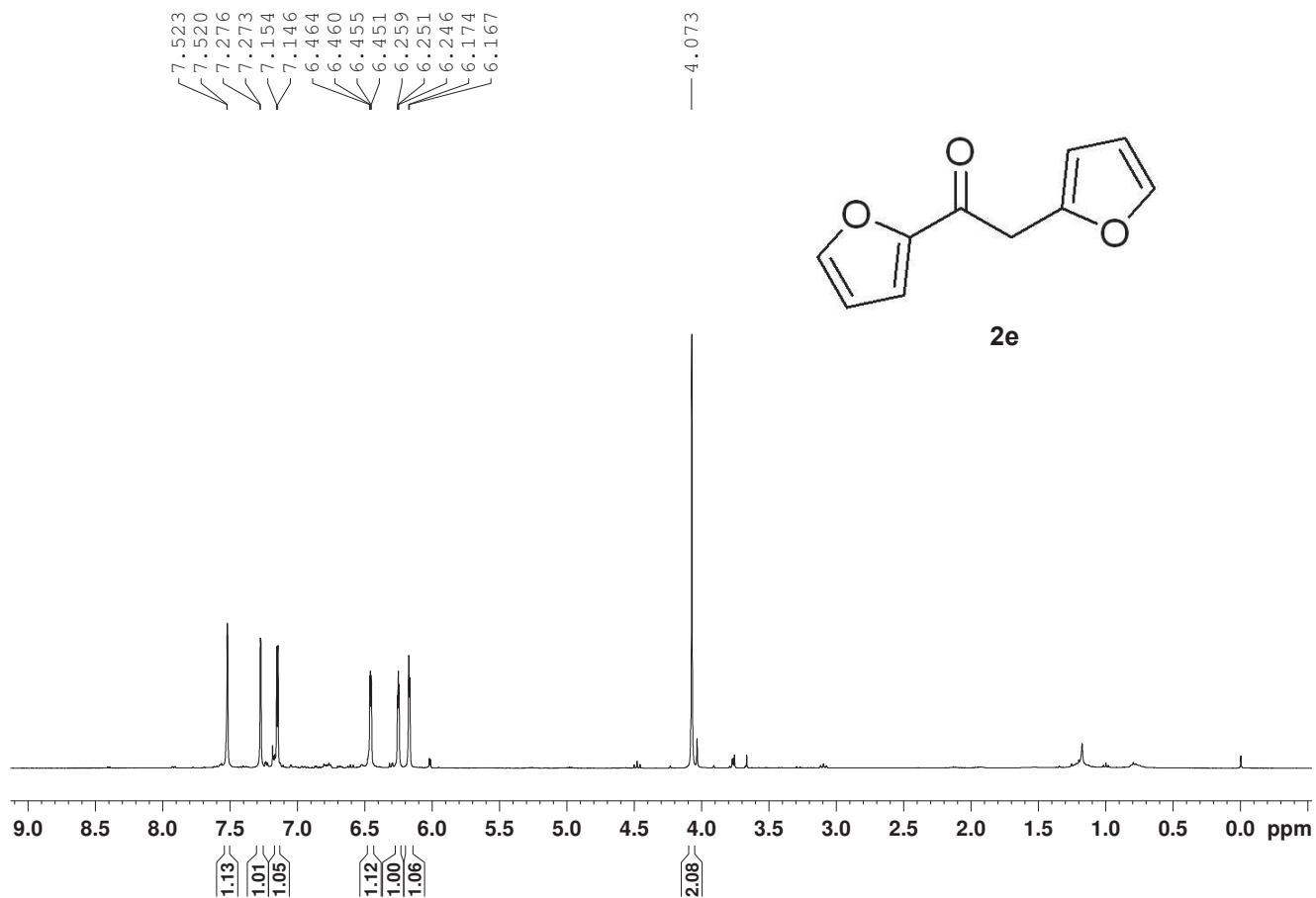
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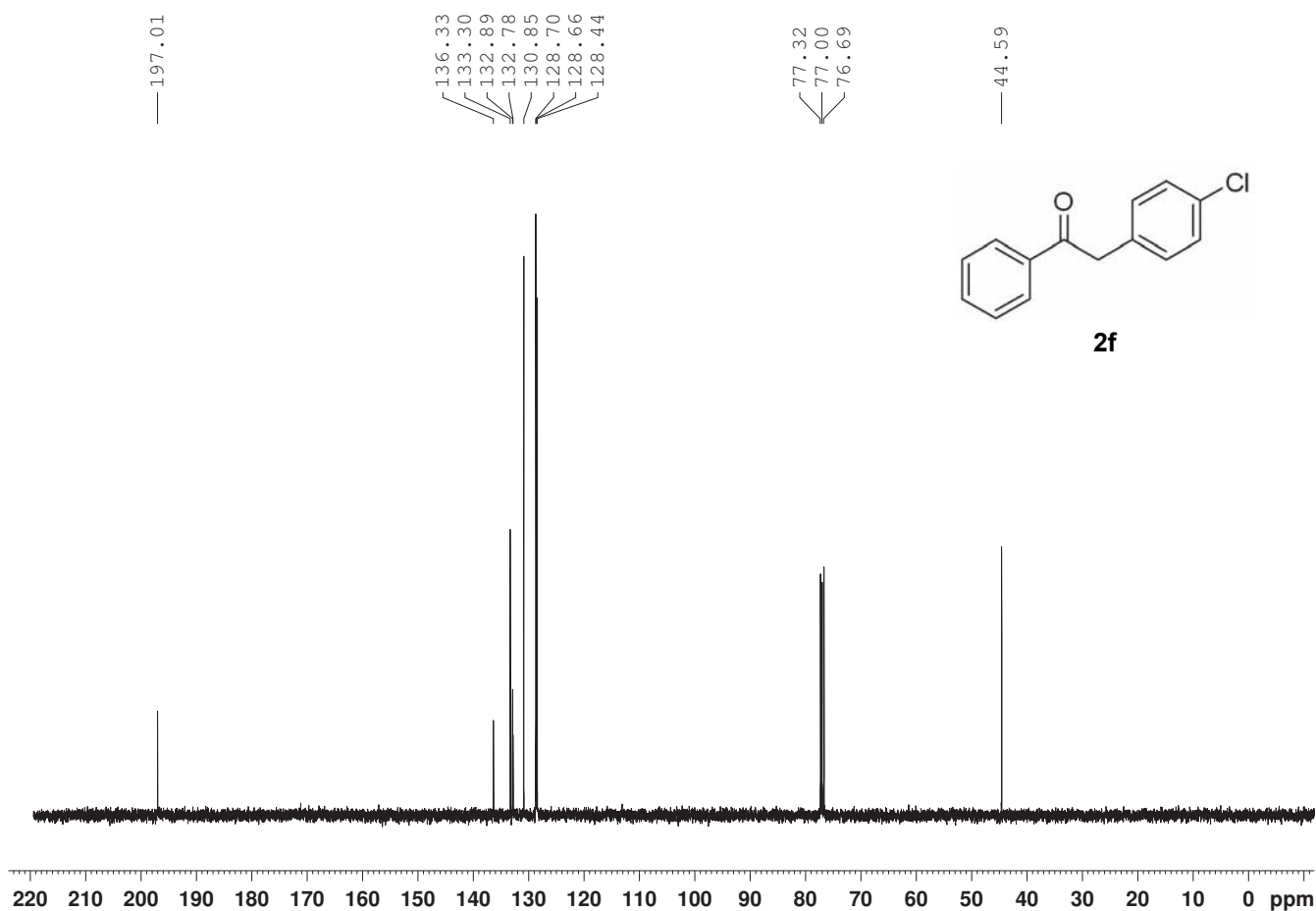
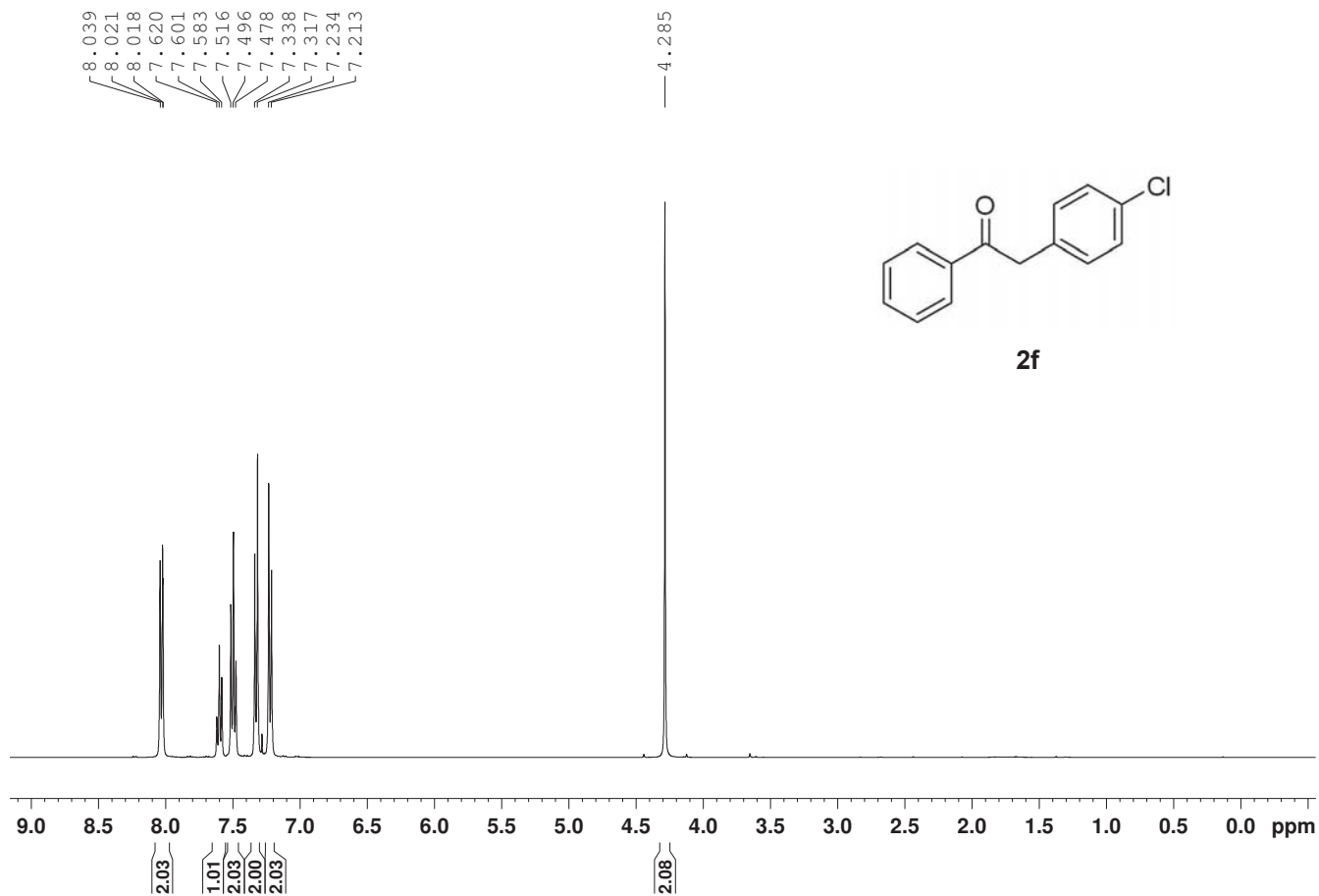


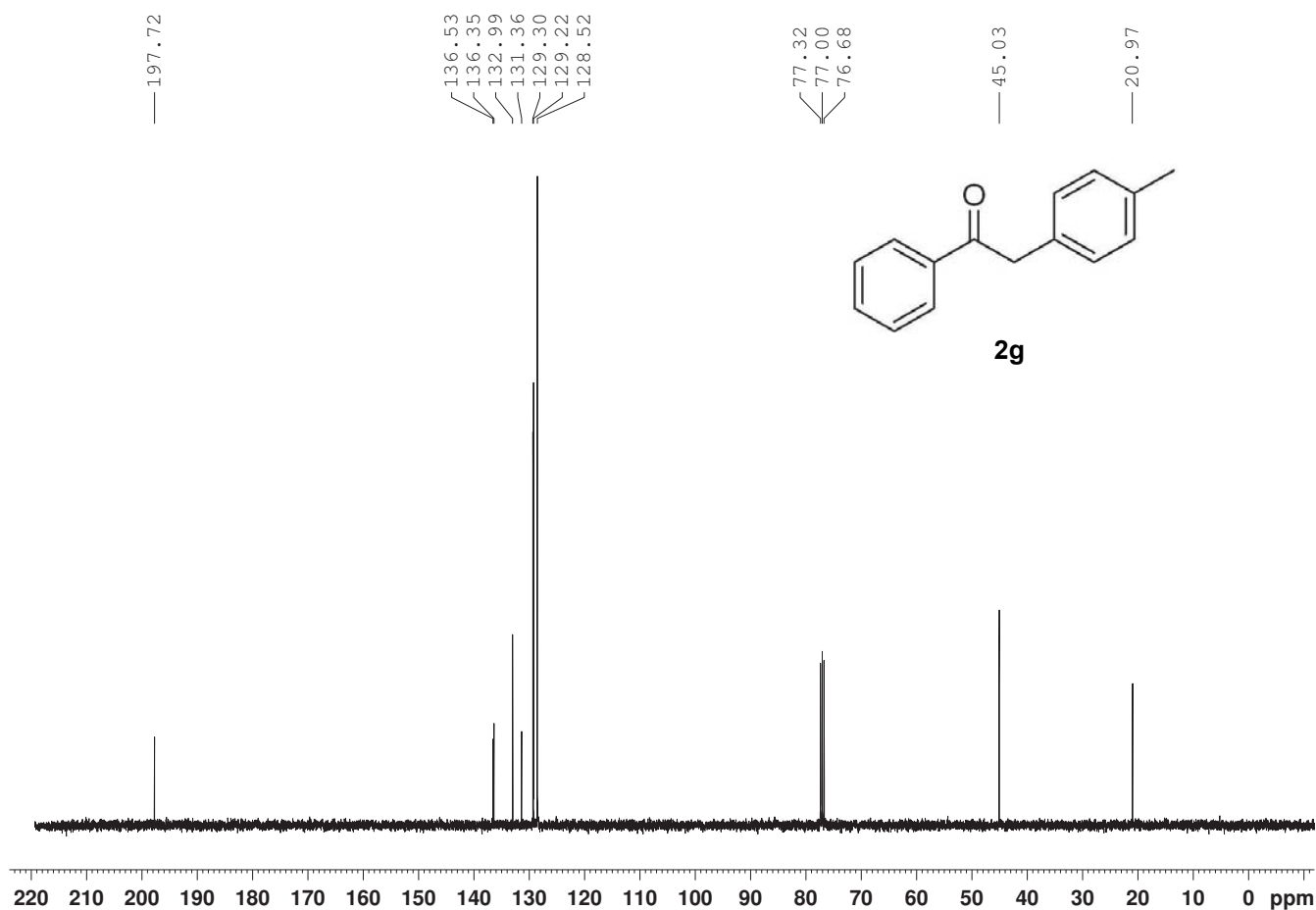
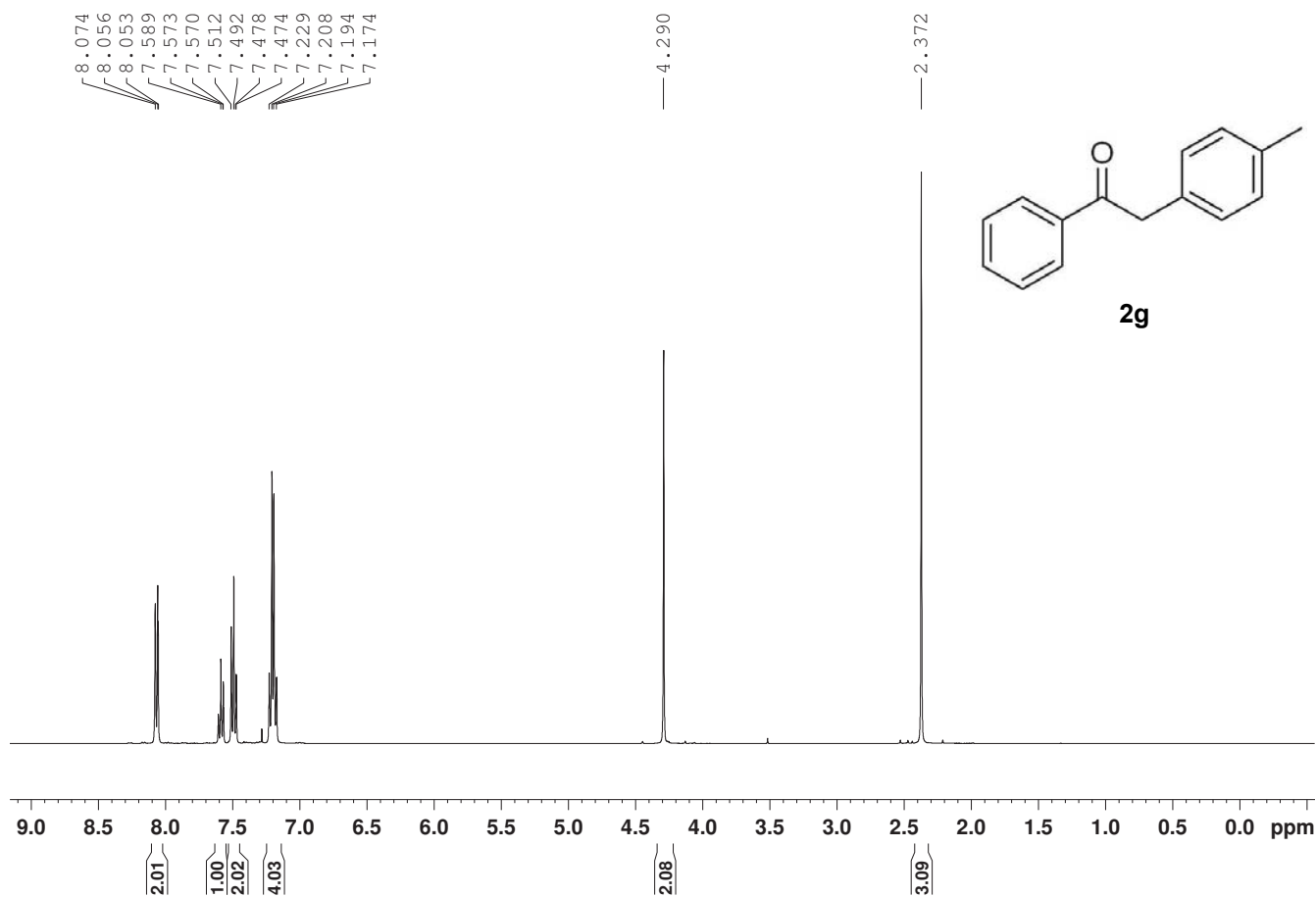








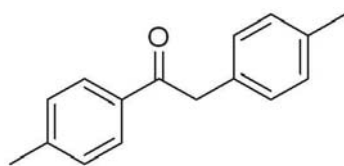




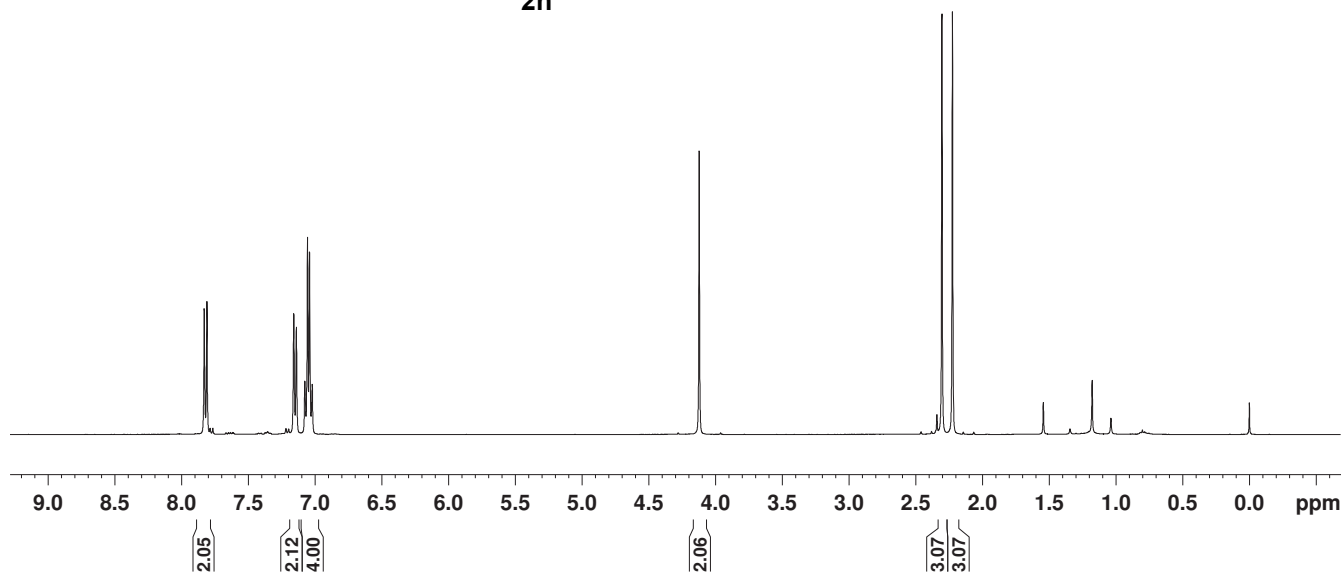
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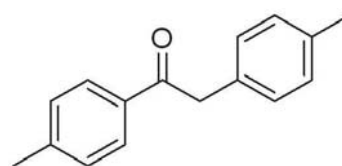
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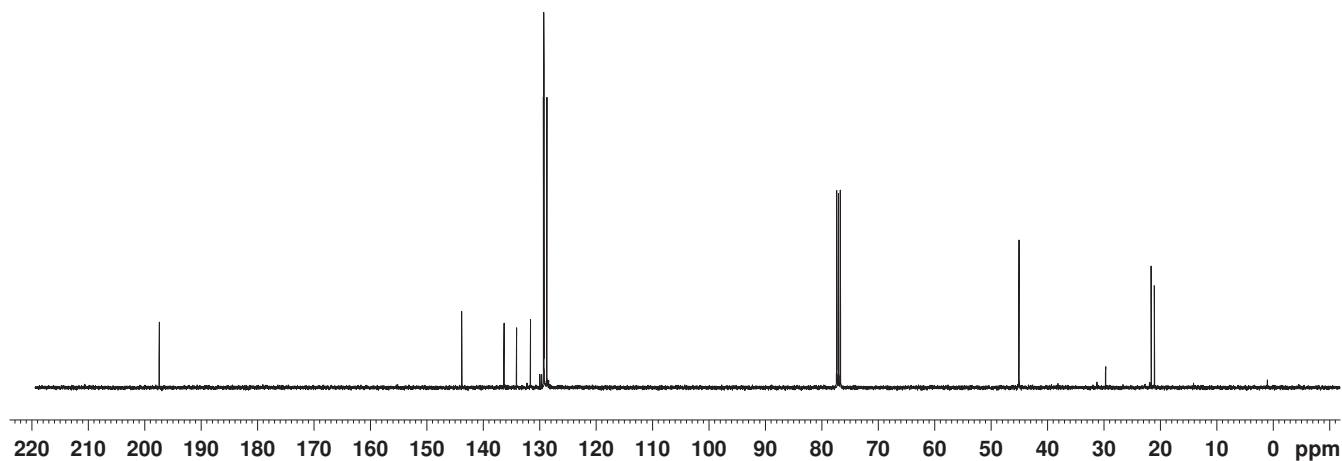
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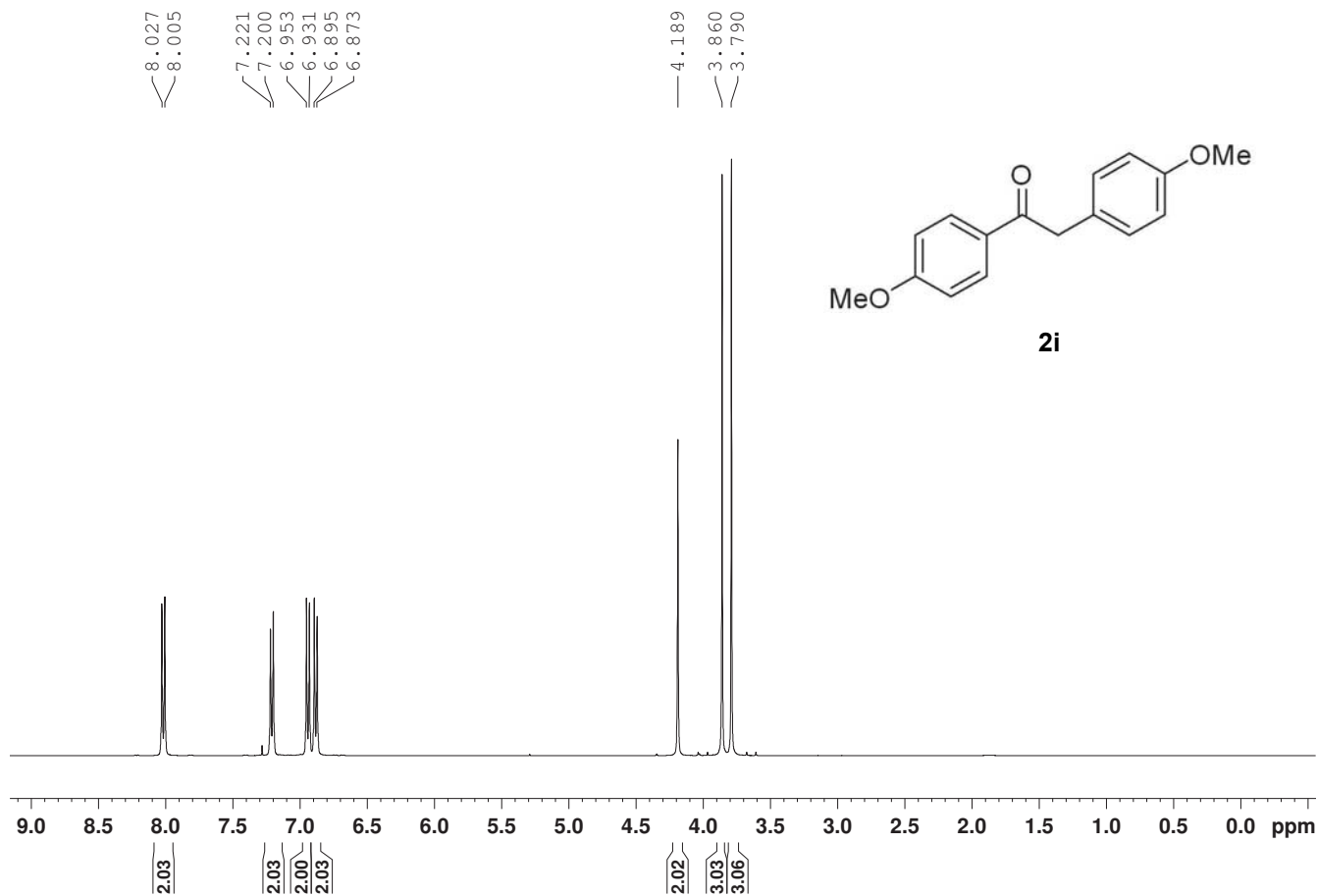
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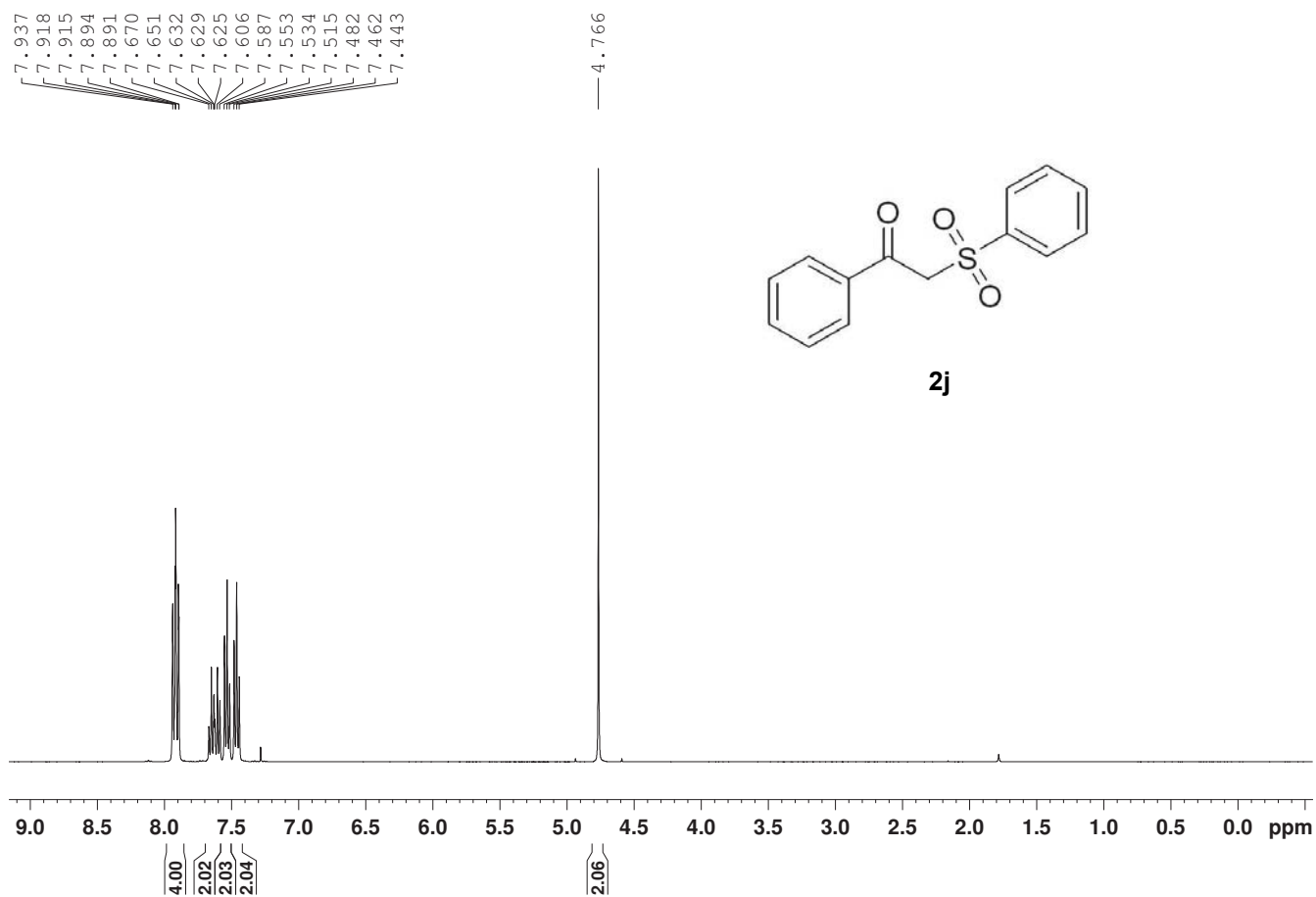
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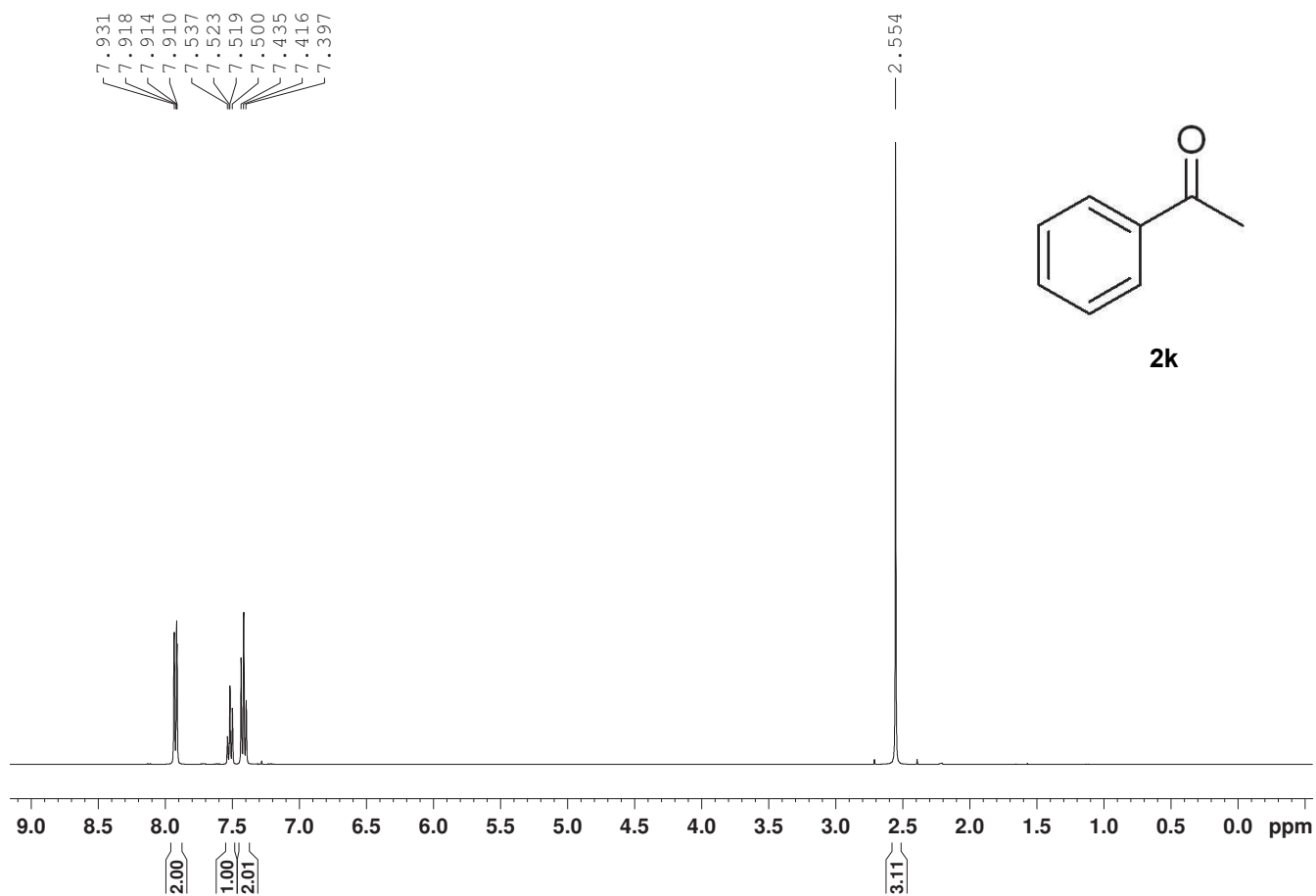


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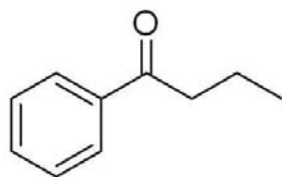


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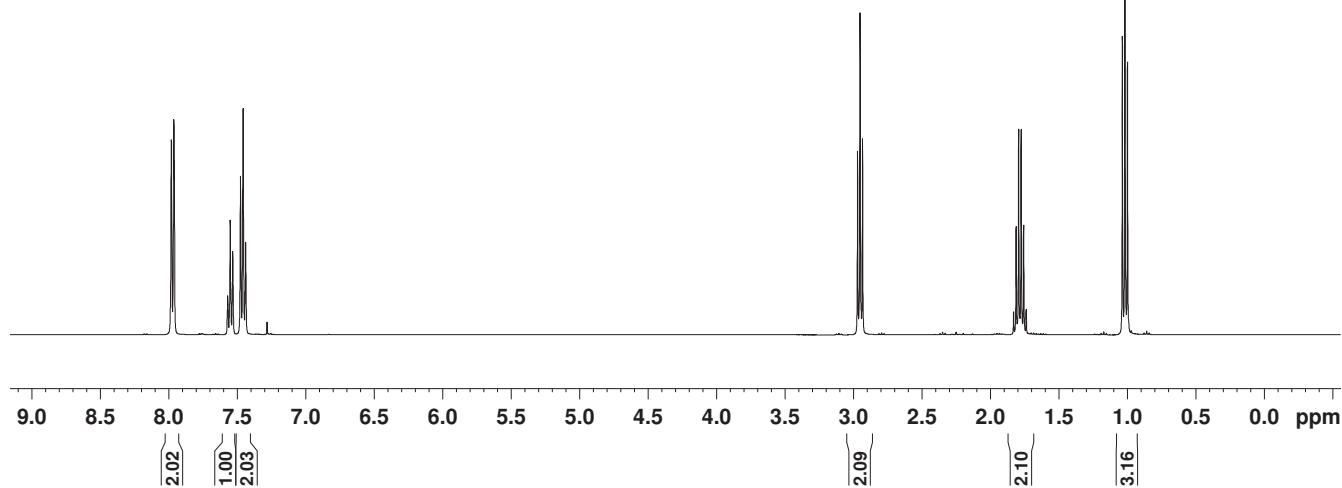
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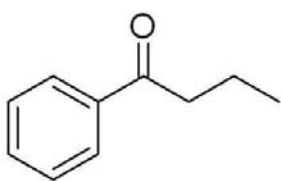
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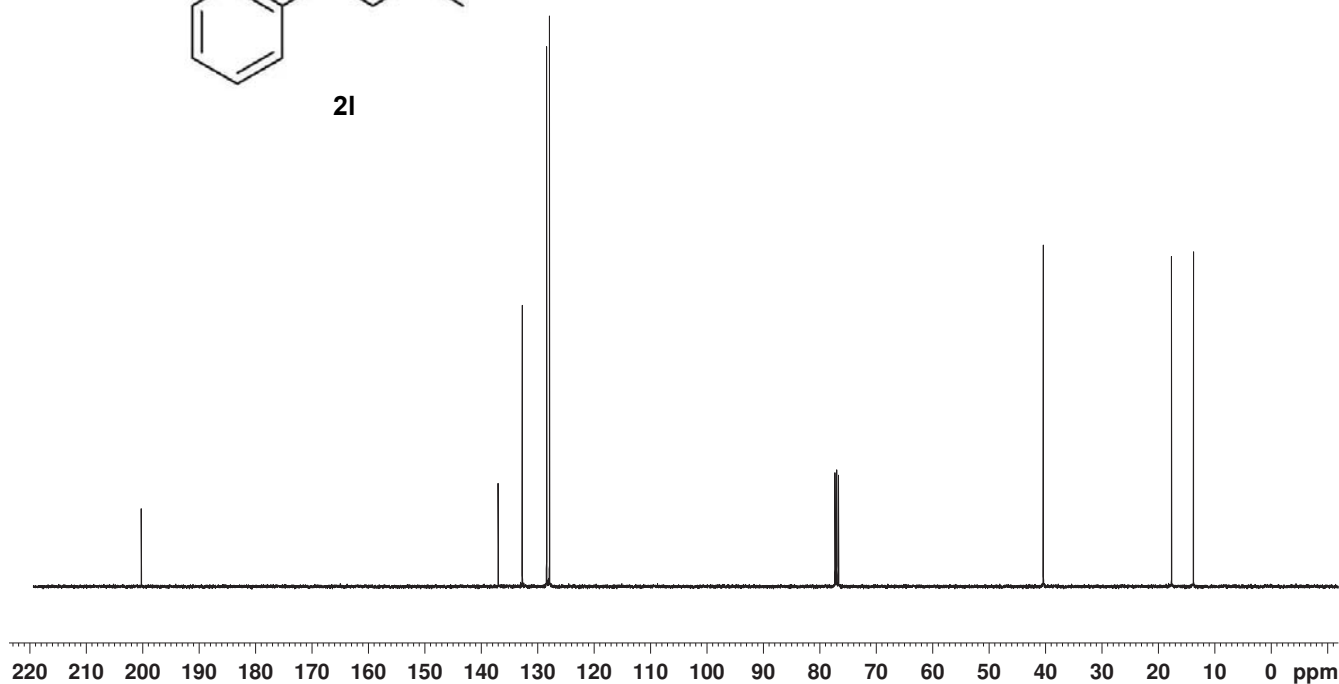
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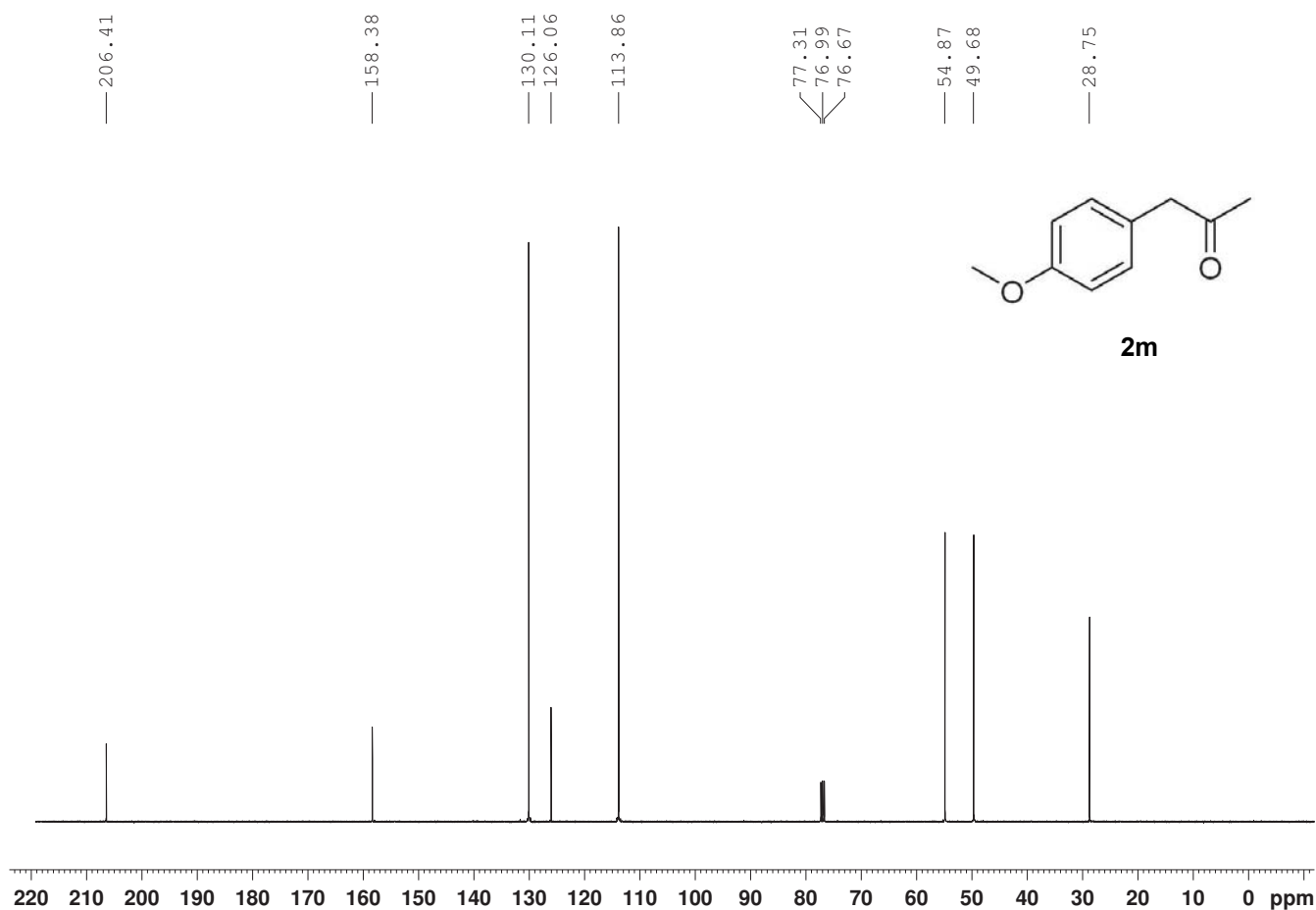
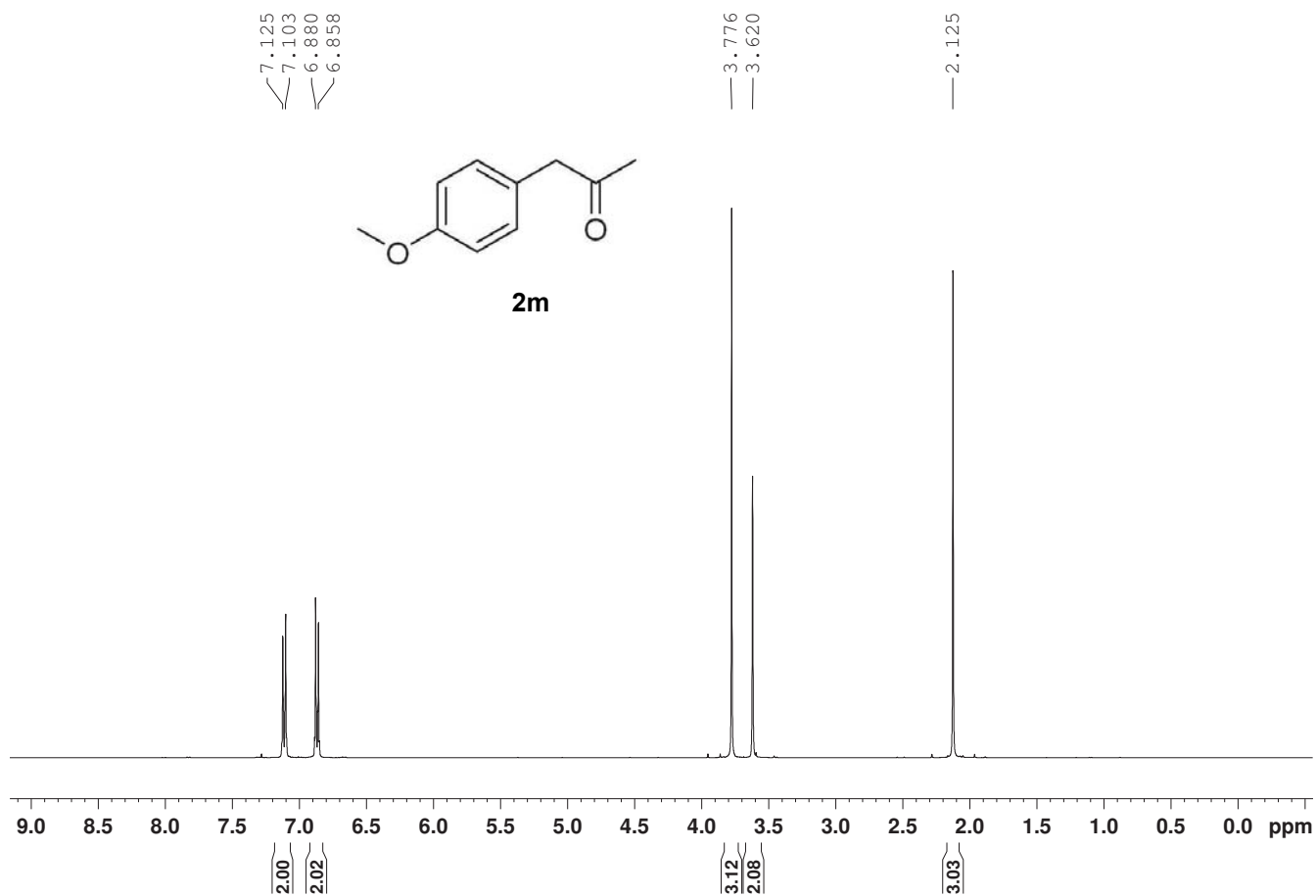
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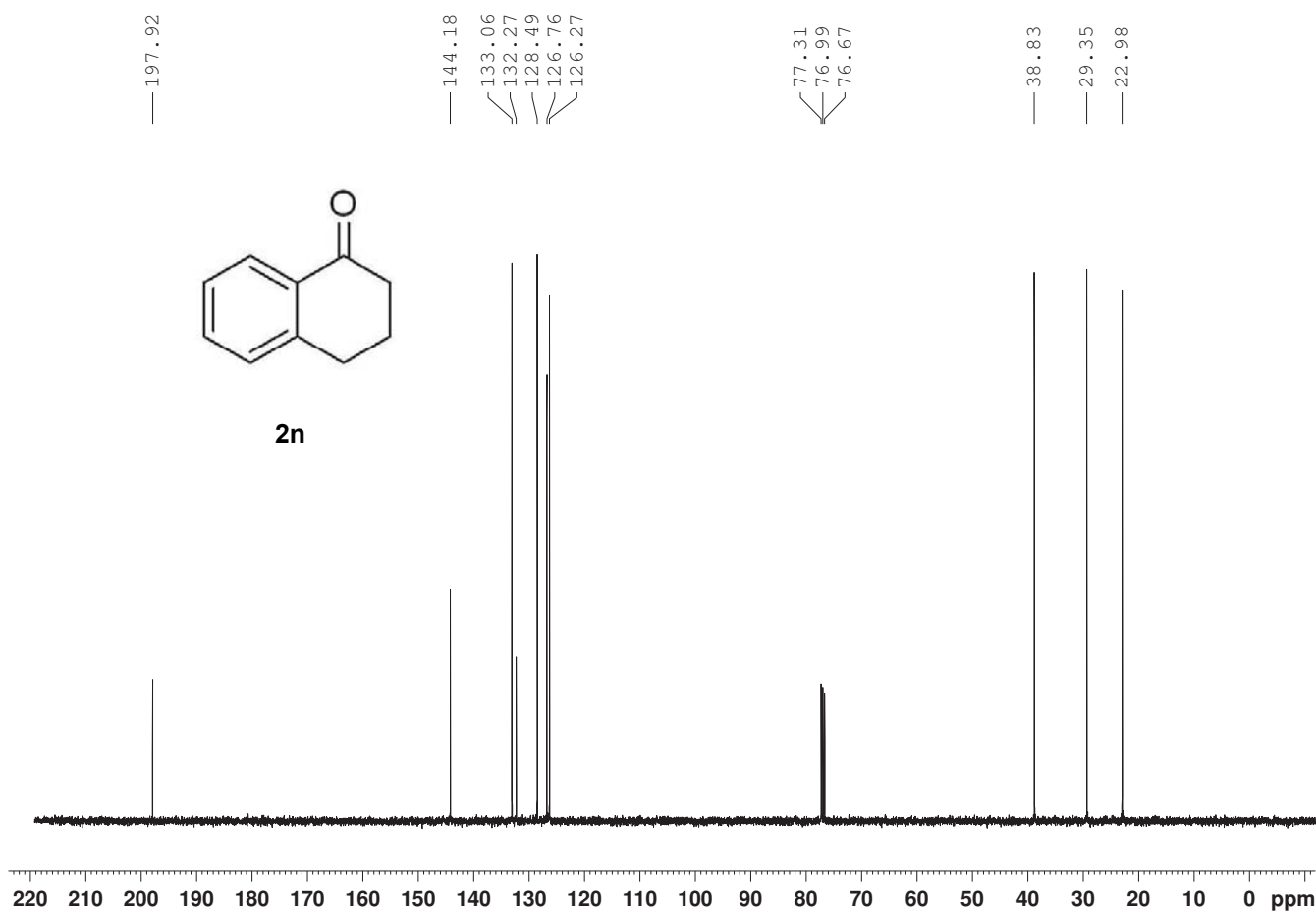
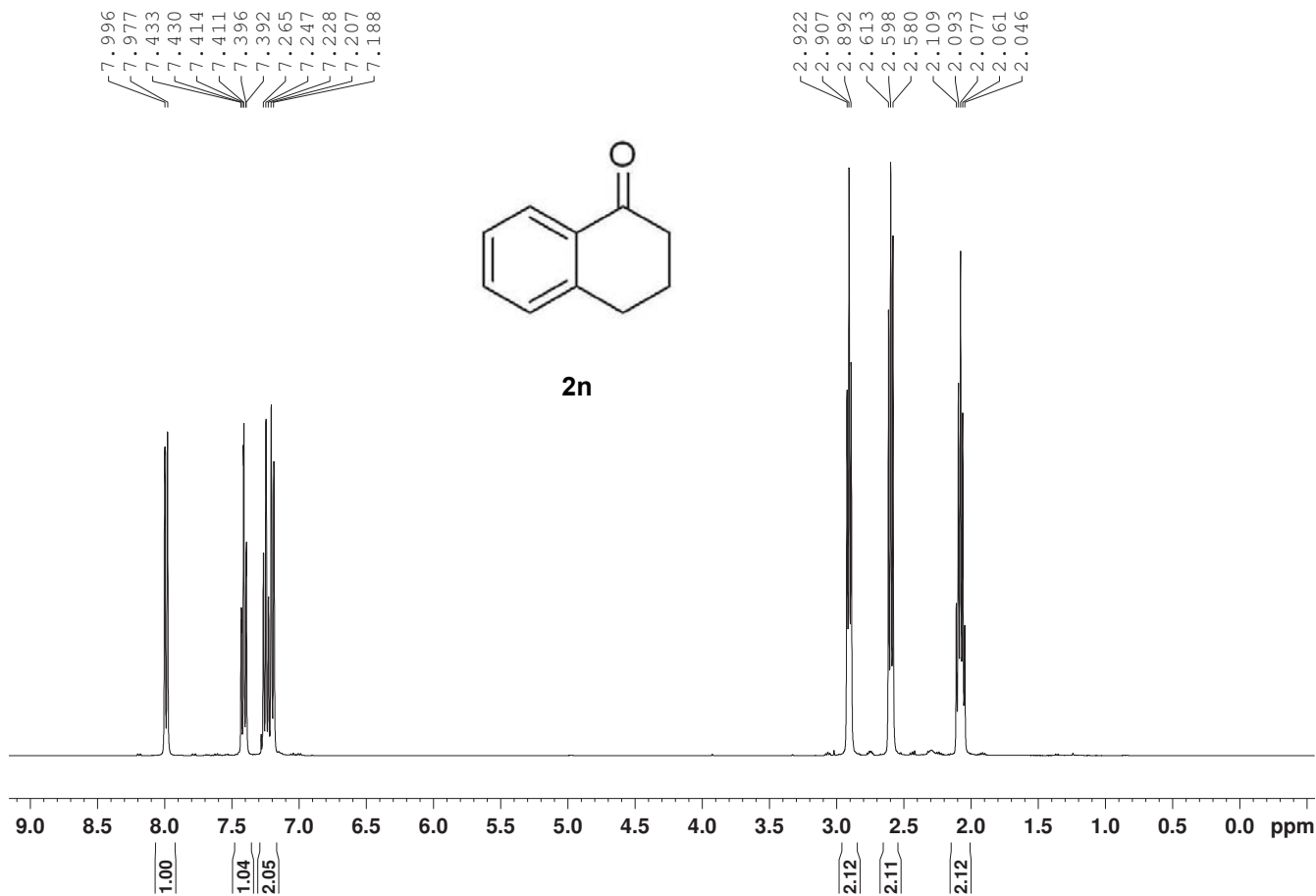
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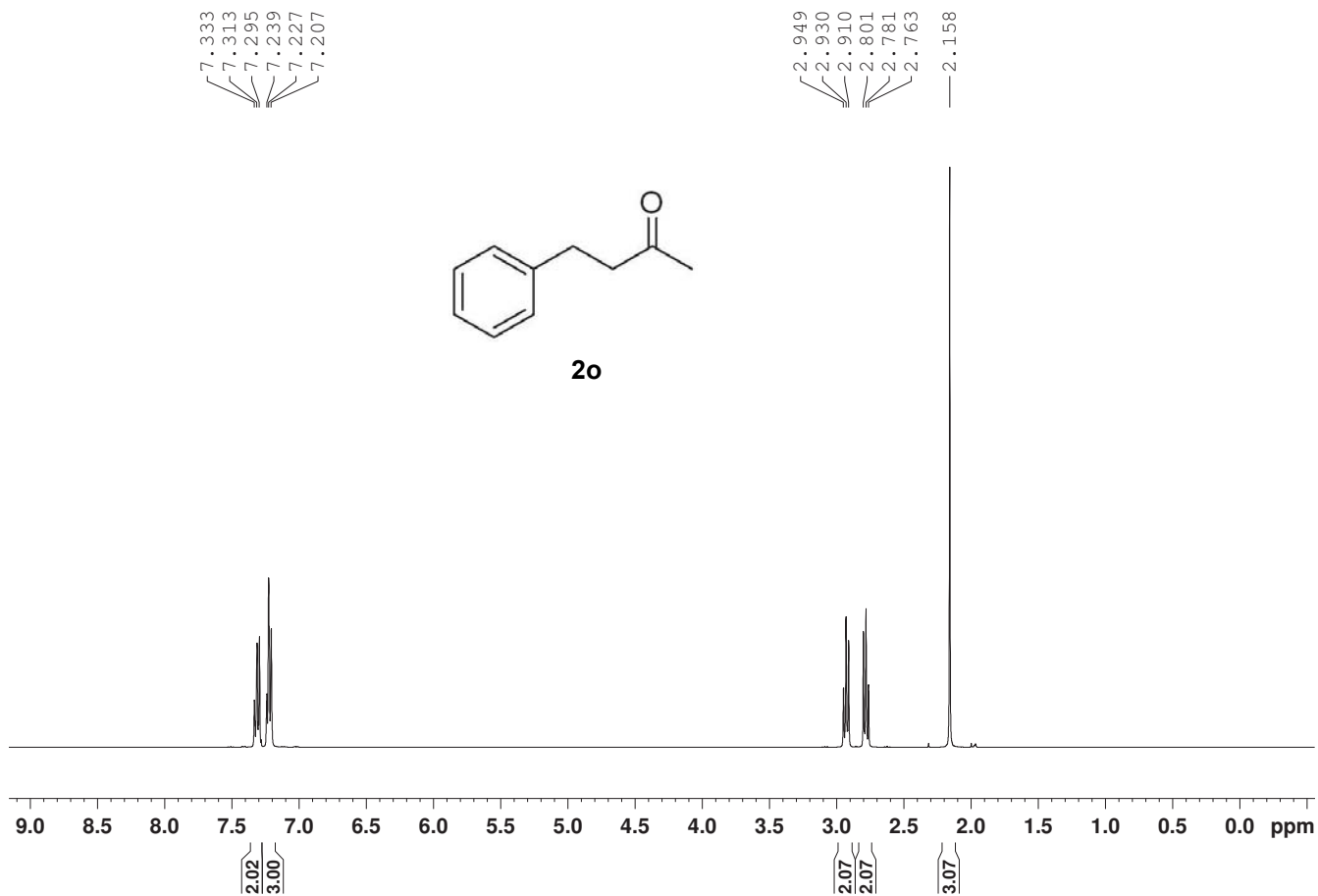


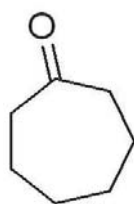
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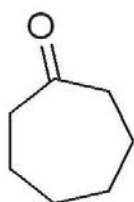
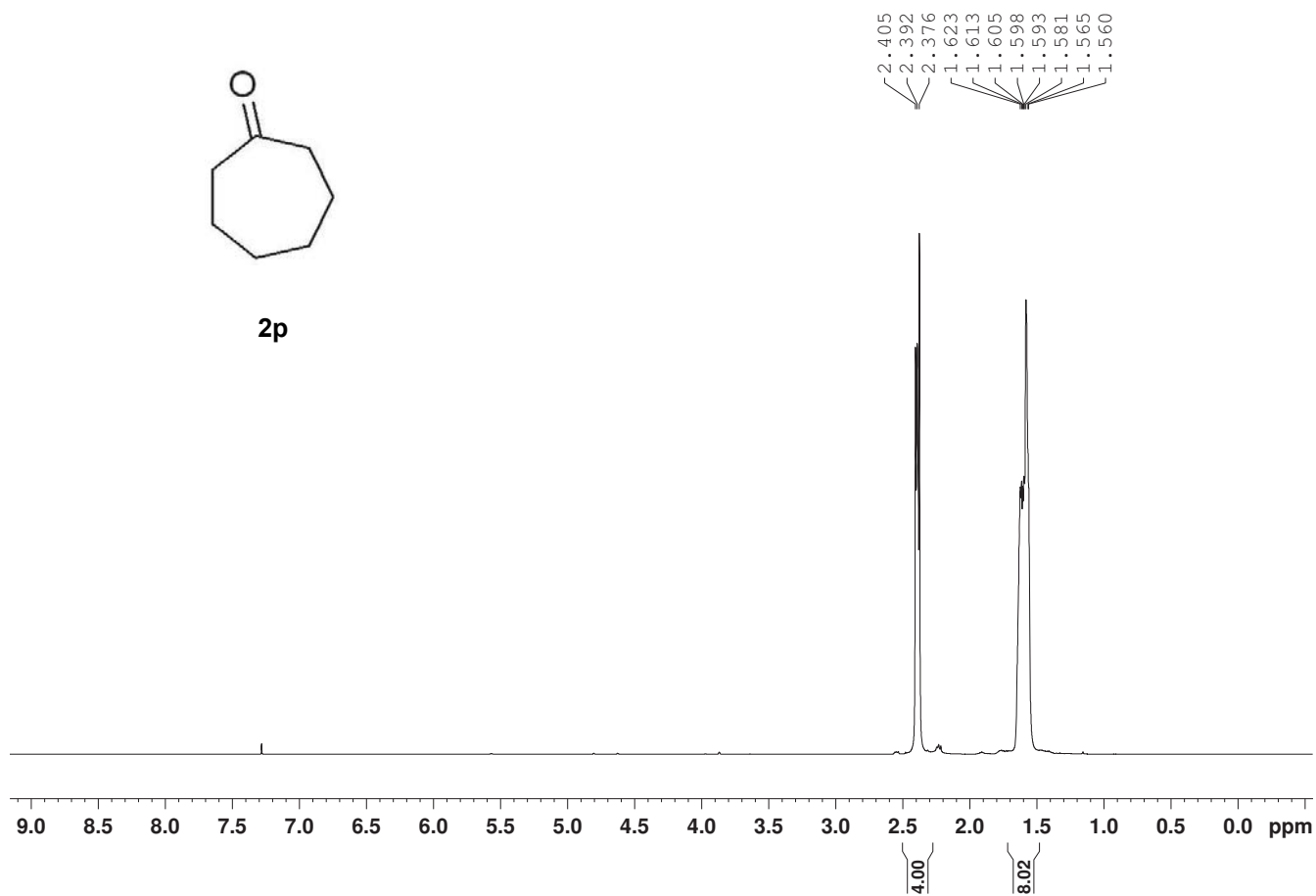




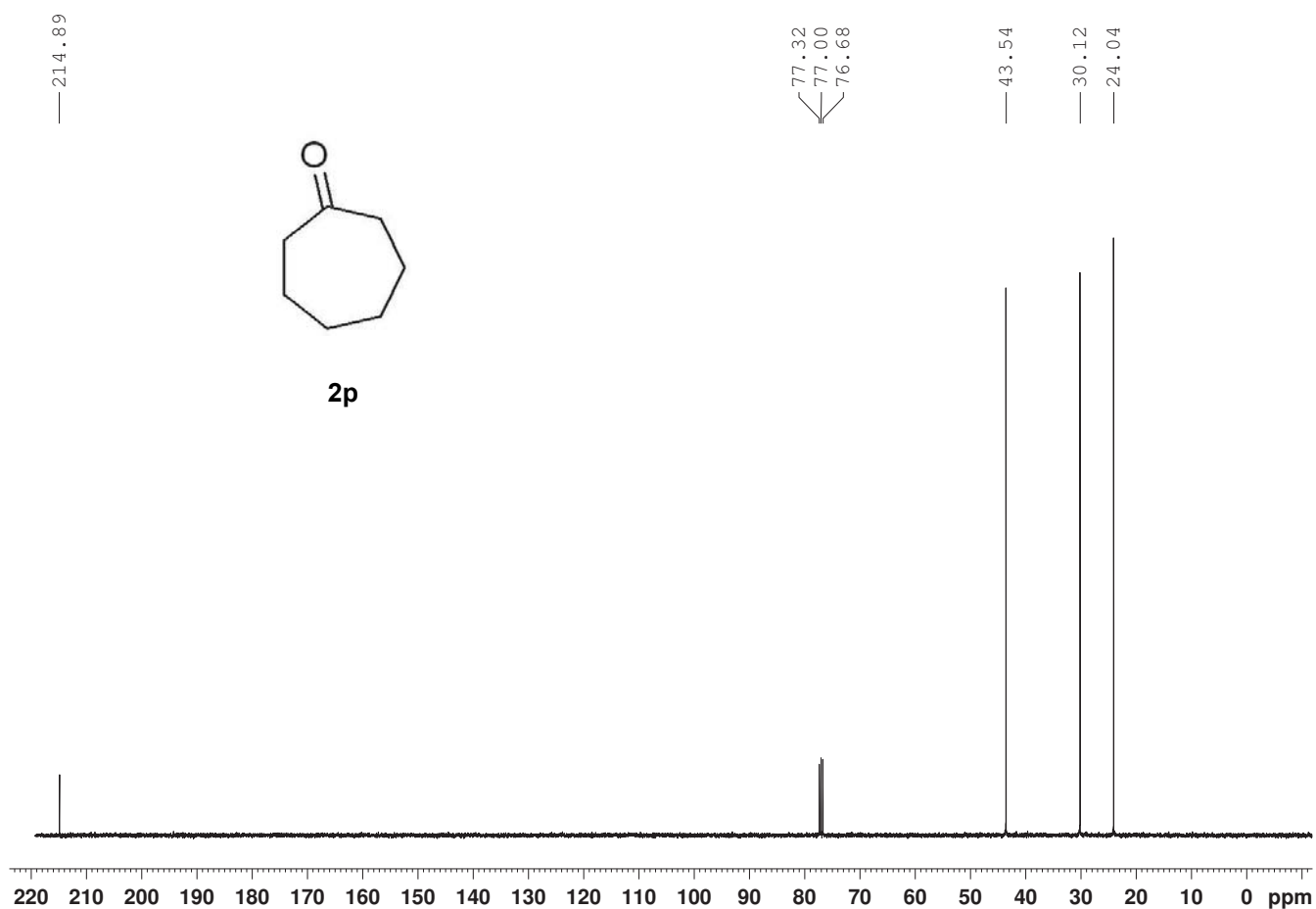




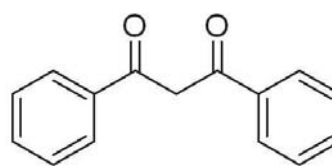
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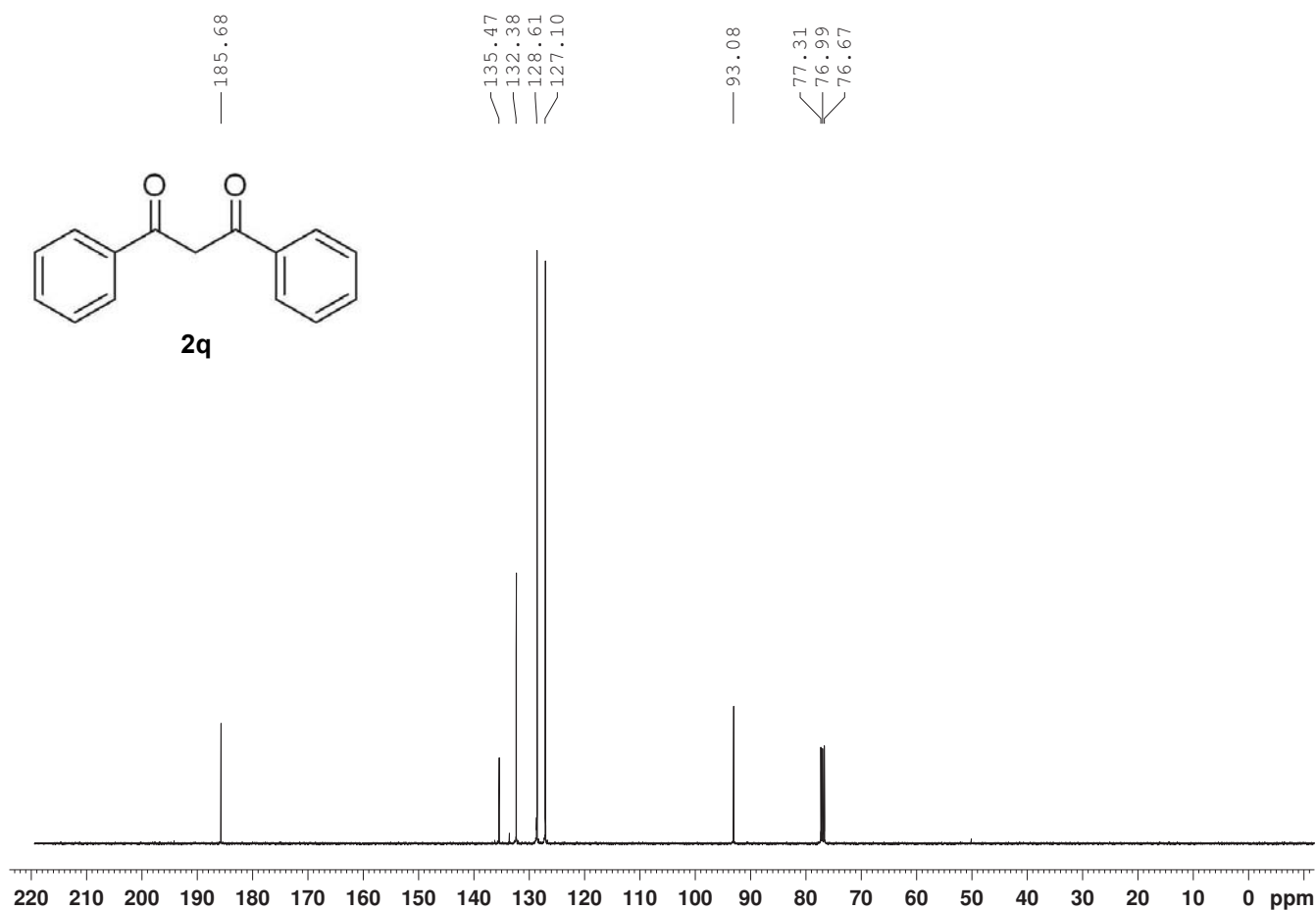
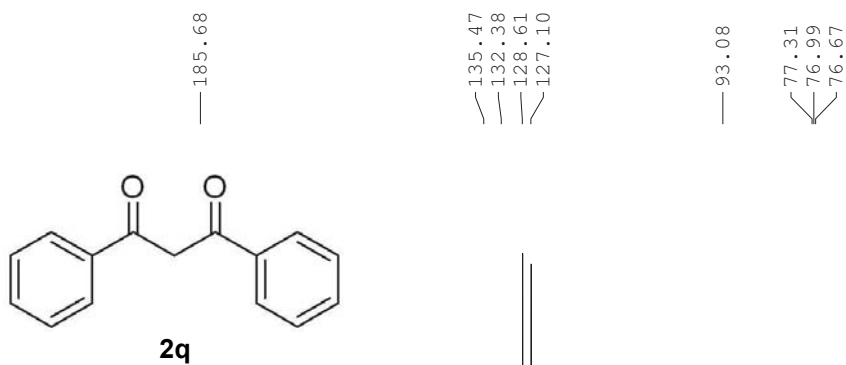
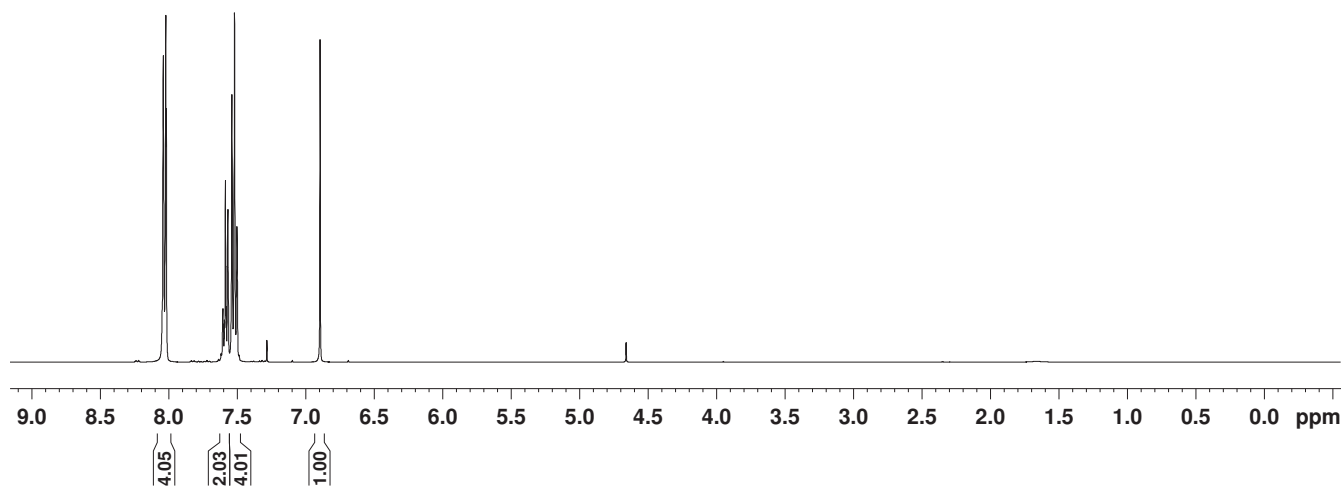
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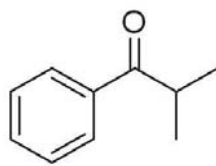
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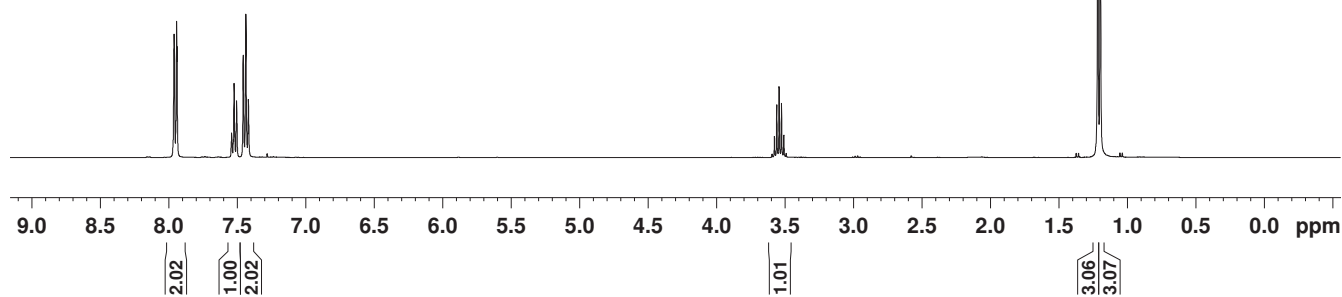
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**2r**



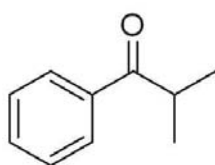
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