

# Supporting Information

## Enantioselective Radical Hydroacylation of Enals with $\alpha$ -Ketoacids Enabled by Photoredox/Amine Cocatalysis

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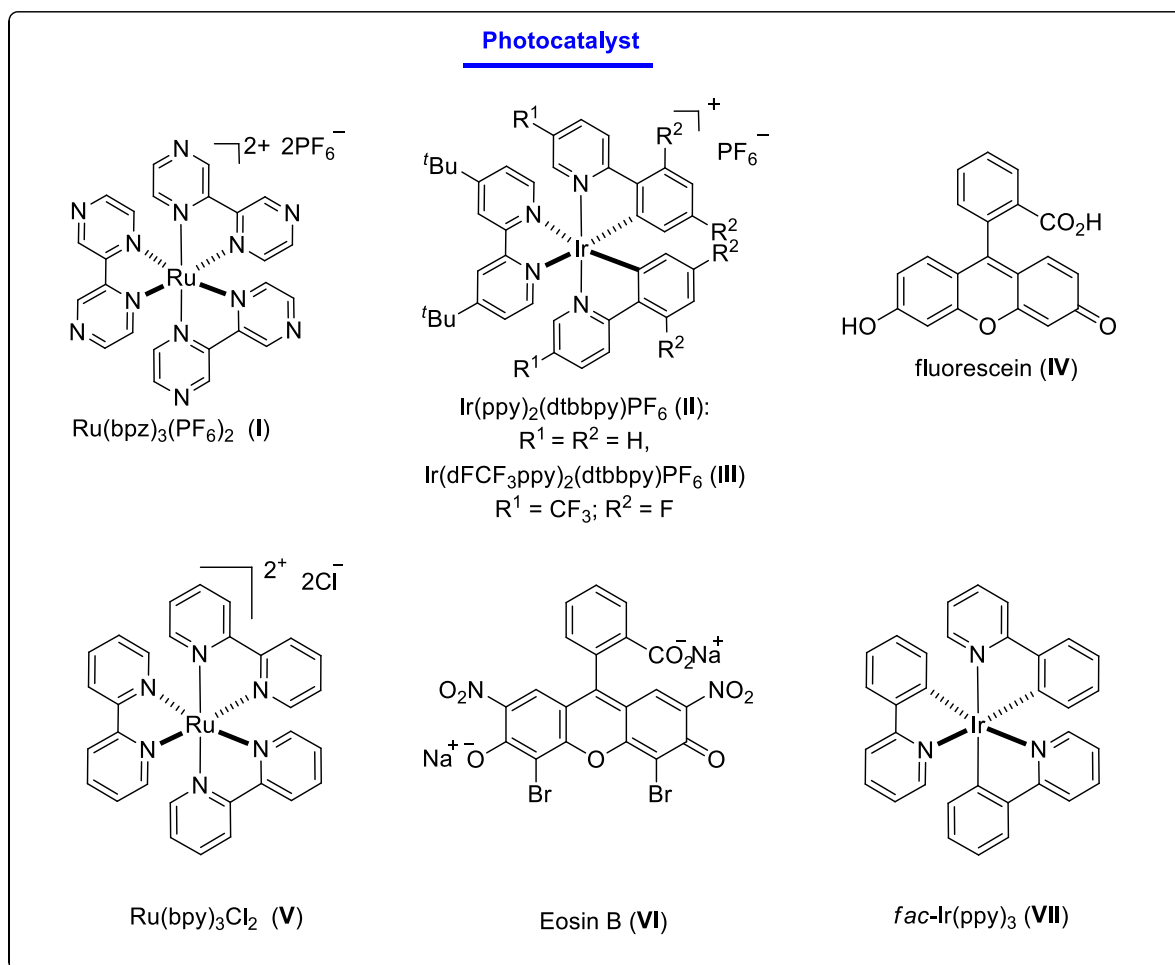
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## 1. General method

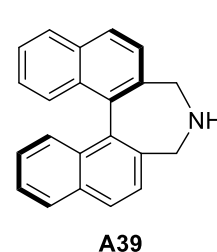
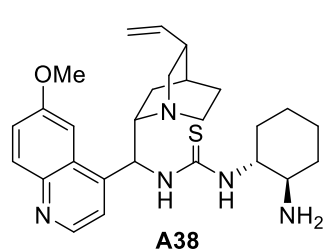
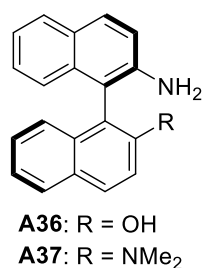
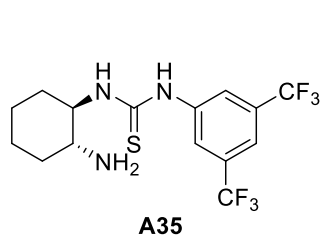
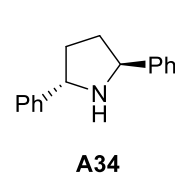
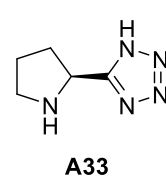
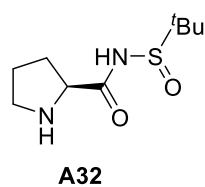
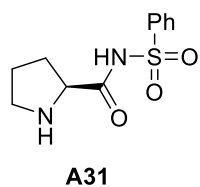
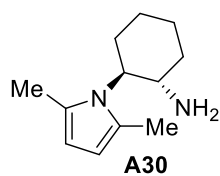
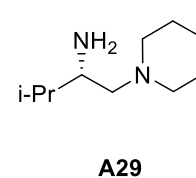
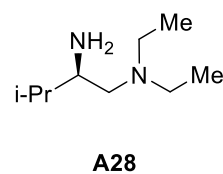
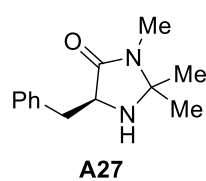
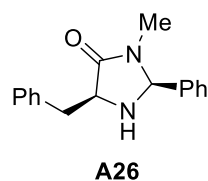
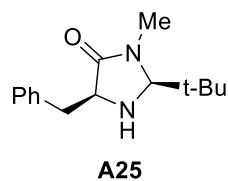
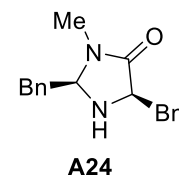
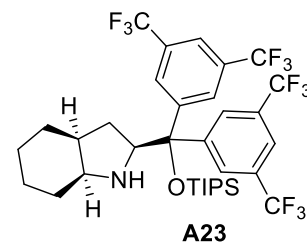
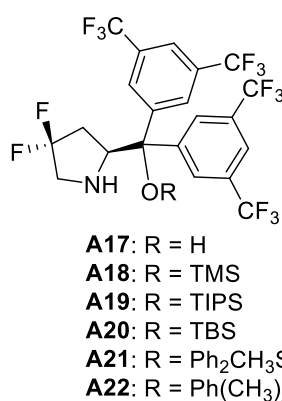
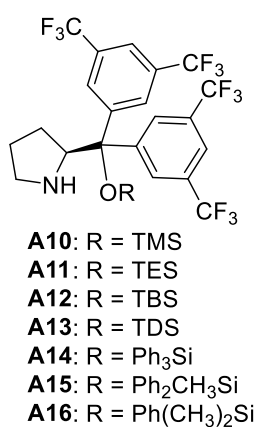
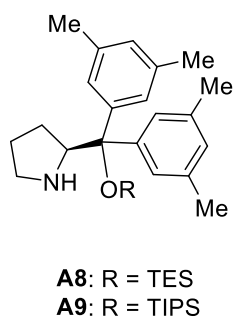
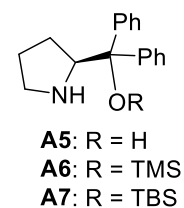
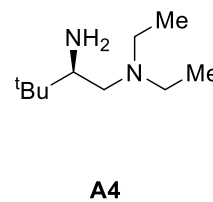
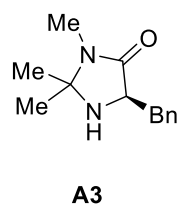
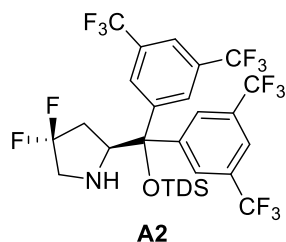
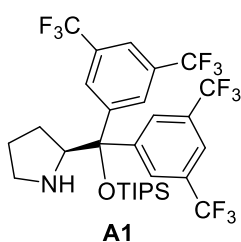
$^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were measured respectively at 400 and 100 MHz, respectively. The solvent used for NMR spectroscopy was  $\text{CDCl}_3$ , using tetramethylsilane as the internal reference. HRMS (ESI) was determined by a micrOTOF-QII HRMS/MS instrument (Bruker). Enantiomeric ratios (*er*) were determined by chiral high-performance liquid chromatography (chiral HPLC). The chiral columns used for the determination of enantiomeric excesses by chiral HPLC were Chiralpak IC and IG-3 columns. Optical rotation values were measured with instruments operating at  $\lambda = 589$  nm, corresponding to the sodium D line at the temperatures indicated. Analytic grade solvents for the chromatography and commercially available reagents were used as received. The substrate  $\alpha$ -ketoacids **1** was prepared according to the literature.<sup>1</sup>

## 2. Numberings and structures of catalysts



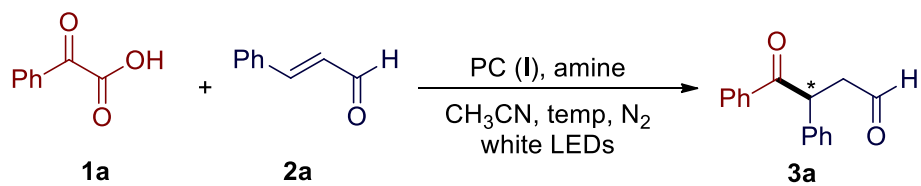
<sup>1</sup> Kuldeep, W.; Yang, C.; West, P. R.; Deming, K. C.; Sanjay, R. C.; Rajarathnam, E. R. *Synth. Commun.* **2008**, *38*, 4434-4444.

# Amine



### 3. Screening of catalysts and condition optimization for 3a

**Table S1: Optimization of reaction conditions<sup>[a]</sup>**



entry	amine	yield/% <sup>[b]</sup>	ee/% <sup>[c]</sup>
1	A1	34	58
2	A3	20	0
3	A4	13	26
4	A5	trace	-
5	A6	6	12
6	A7	trace	-
7	A8	10	22
8	A9	12	2
9	A10	19	38
10	A11	22	46
11	A12	21	58
12	A13	19	58
13	A14	10	60
14	A15	11	58
15	A16	8	48



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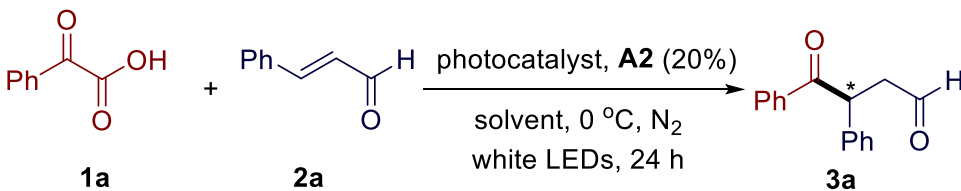
16	<b>A24</b>	14	10
17	<b>A31</b>	trace	-
18	<b>A32</b>	14	2
19	<b>A33</b>	22	0
20	<b>A34</b>	33	24
21	<b>A35</b>	9	6
22	<b>A36</b>	11	6
23	<b>A37</b>	4	26
24	<b>A38</b>	trace	-
25	<b>A39</b>	14	2
26 <sup>[d]</sup>	<b>A1</b>	20	66
27 <sup>[d]</sup>	<b>A2</b>	55	74
28 <sup>[d]</sup>	<b>A17</b>	20	55
29 <sup>[d]</sup>	<b>A18</b>	25	50
30 <sup>[d]</sup>	<b>A19</b>	40	72
31 <sup>[d]</sup>	<b>A20</b>	38	69
32 <sup>[d]</sup>	<b>A21</b>	50	74
33 <sup>[d]</sup>	<b>A22</b>	44	62
34 <sup>[d]</sup>	<b>A23</b>	6	56
35 <sup>[d]</sup>	<b>A25</b>	9	13

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36 <sup>[d]</sup>	<b>A26</b>	12	11
37 <sup>[d]</sup>	<b>A27</b>	15	13
38 <sup>[d]</sup>	<b>A28</b>	22	25
39 <sup>[d]</sup>	<b>A29</b>	19	18
40 <sup>[d]</sup>	<b>A30</b>	trace	-

[a] Reaction conditions: a solution of **1a** (0.15 mmol), **2a** (0.10 mmol), amine (0.02 mmol, 20 mol%), and photocatalyst (0.002 mmol, 2 mol%) in the indicated solvent (1.0 mL) was irradiated by white LED strips for 24 h at ambient temperature. [b] Isolated yield. [c] The *ee* value was determined by HPLC. [d] The reaction temperature is 0 °C.

**Table S2: Optimization of reaction conditions<sup>[a]</sup>**

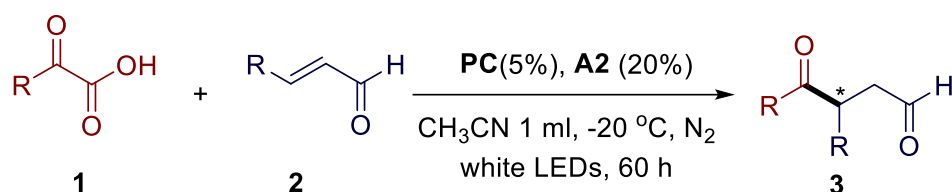
					
entry	PC (x %)	solvent	<b>1a:2a</b>	yield/% <sup>[b]</sup>	<i>ee</i> / % <sup>[c]</sup>
1	<b>I (2%)</b>	CH <sub>3</sub> CN (1 mL)	1.5:1	55	74
2	<b>II (2%)</b>	CH <sub>3</sub> CN (1 mL)	1.5:1	N. R.	-
3	<b>III (2%)</b>	CH <sub>3</sub> CN (1 mL)	1.5:1	21	72
4	<b>IV (2%)</b>	CH <sub>3</sub> CN (1 mL)	1.5:1	18	72
5	<b>V (2%)</b>	CH <sub>3</sub> CN (1 mL)	1.5:1	N. R.	-
6	<b>VI (2%)</b>	CH <sub>3</sub> CN (1 mL)	1.5:1	N. R.	-
7	<b>VII (2%)</b>	CH <sub>3</sub> CN (1 mL)	1.5:1	N. R.	-
8	<b>I (2%)</b>	DCM (1 mL)	1.5:1	26	39
9	<b>I (2%)</b>	PhCF <sub>3</sub> (1 mL)	1.5:1	15	33

10	<b>I (2%)</b>	1,4-dioxane (1 mL)	1.5:1	trace	-
11	<b>I (2%)</b>	EtOAc (1 mL)	1.5:1	trace	-
12	<b>I (2%)</b>	DMSO (1 mL)	1.5:1	trace	-
13	<b>I (2%)</b>	DMF (1 mL)	1.5:1	N.R.	-
14	<b>I (2%)</b>	MeOH (1 mL)	1.5:1	N.R.	-
15	<b>I (2%)</b>	CH <sub>3</sub> CN (1 mL)	3:1	76	74
16	<b>I (2%)</b>	CH <sub>3</sub> CN (1 mL)	1:1	33	74
17	<b>I (2%)</b>	CH <sub>3</sub> CN (1 mL)	1:1.5	35	74
18	<b>I (2%)</b>	CH <sub>3</sub> CN (1 mL)	1:3	37	74
19	<b>I (2%)</b>	CH <sub>3</sub> CN (0.5 mL)	3:1	72	70
20	<b>I (2%)</b>	CH <sub>3</sub> CN (2 mL)	3:1	68	74
21	<b>I (2%)</b>	CH <sub>3</sub> CN (3 mL)	3:1	60	74
22	<b>I (2%)</b>	CH <sub>3</sub> CN (4 mL)	3:1	56	74
23	<b>I (1%)</b>	CH <sub>3</sub> CN (1 mL)	3:1	61	74
24	<b>I (5%)</b>	CH <sub>3</sub> CN (1 mL)	3:1	91	74
25	<b>I (10%)</b>	CH <sub>3</sub> CN (1 mL)	3:1	95	74
26 <sup>[d]</sup>	<b>I (5%)</b>	CH <sub>3</sub> CN (1 mL)	3:1	48	78
27 <sup>[e]</sup>	<b>I (5%)</b>	CH <sub>3</sub> CN (1 mL)	3:1	76	78
28 <sup>[f]</sup>	<b>I (5%)</b>	CH <sub>3</sub> CN (1 mL)	3:1	90	80
29 <sup>[g]</sup>	<b>I (5%)</b>	CH <sub>3</sub> CN (1 mL)	3:1	N.R.	-

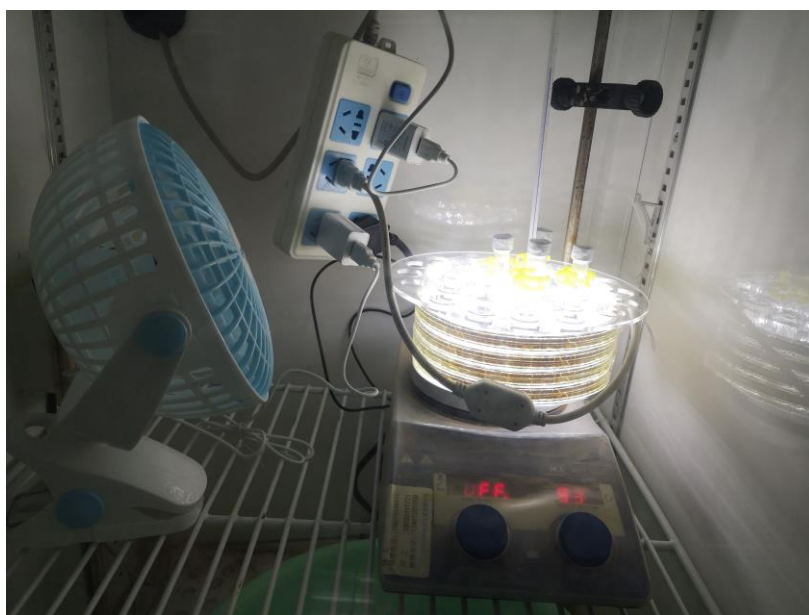
30	-	CH <sub>3</sub> CN (1 mL)	3:1	13	80
31 <sup>[h]</sup>	<b>I (5%)</b>	CH <sub>3</sub> CN (1 mL)	3:1	trace	-
32 <sup>[i]</sup>	<b>I (5%)</b>	CH <sub>3</sub> CN (1 mL)	3:1	88	78

[a] Unless otherwise indicated, the reaction was carried out at the 0.10 mmol scale and irradiated by white LED strips for 24h at 0 °C. [b] Isolated yield. [c] The *ee* value was determined by HPLC. [d] The reaction temperature is -20 °C, reaction time is 24 h. [e] The reaction temperature is -20 °C, reaction time is 48 h. [f] The reaction temperature is -20 °C, reaction time is 60 h. [g] in dark. [h] no amine. [i] The reaction takes place in air.

#### 4. General procedure for the synthesis of products 3



Acetonitrile (CH<sub>3</sub>CN) (1 mL) was added to the mixture of benzoylformic acid **1** (0.3 mmol), trans-cinnamaldehyde **2** (0.1 mmol), catalyst **A2** (0.02 mmol) and **PC** (0.005 mmol) under nitrogen. After being stirred at -20 °C in a refrigerator for 60 h with white LED strips, the reaction mixture was quenched with NaHCO<sub>3</sub> (aq.), extracted with ether, and purified through preparative thin layer chromatography to afford pure products **3**.

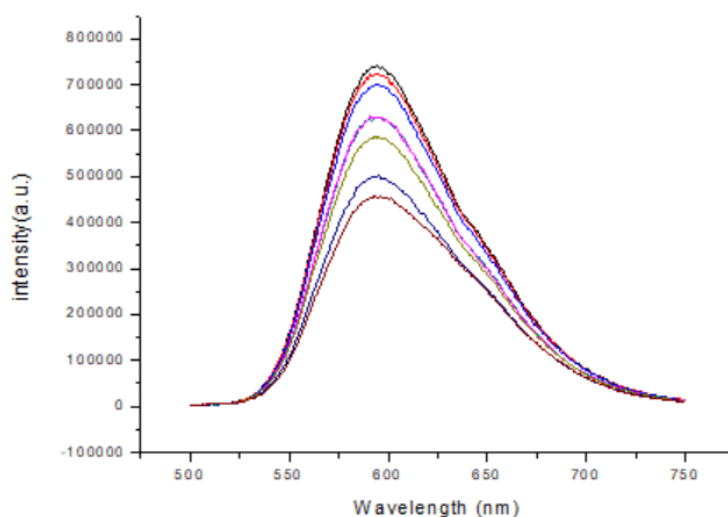


**Figure S1.** The reaction apparatus.

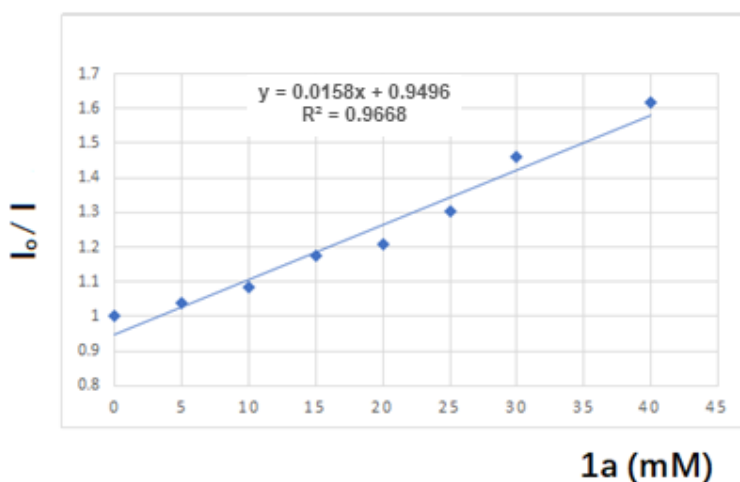
## 5. Mechanism Studies

### a) Quenching experiments and Stern-Volmer analysis.

A Hitachi F-7000 fluorescence spectrometer was used to record the emission intensities. All  $\text{Ru}(\text{bpz})_3(\text{PF}_6)_2$  solutions were excited at 443 nm and the emission intensity at 590 nm was observed.  $\text{CH}_3\text{CN}$  was degassed with a stream of Ar for 30 min. In a typical experiment, the emission spectrum of a  $5 \times 10^{-5}$  M solution of  $\text{Ru}(\text{bpz})_3(\text{PF}_6)_2$  in  $\text{CH}_3\text{CN}$  was collected. Then, appropriate amount of quencher was added to the measured solution in a quartz cuvette and the emission spectrum of the sample was collected.  $I_0$  and  $I$  represent the intensities of the emission in the absence and presence of the quencher at 590 nm.



**Figure S2.** Emission spectra of  $5 \times 10^{-5}$  M  $\text{Ru}(\text{bpz})_3(\text{PF}_6)_2$  at  $\lambda_{\text{ex}} = 443\text{nm}$  showing the quenching effect of increasing of **1a**.



**Figure S3.** The linear relationship over the increasing concentration of **1a**.

## b) Quantum Yield Measurement

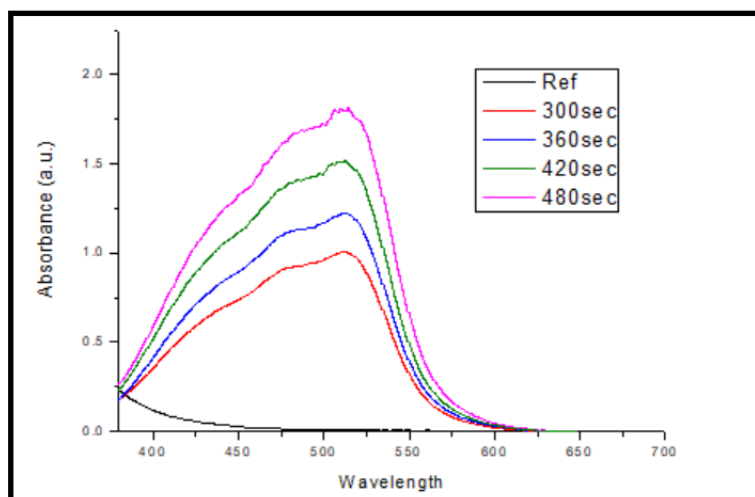
A ferrioxalate actinometry solution was prepared by following the Hammond variation of the Hatchard and Parker procedure outlined in Handbook of Photochemistry. Ferrioxalate actinometer solution measures the decomposition of ferric ions to ferrous ions, which are complexed by 1,10-phenanthroline and monitored by UV/Vis absorbance at 510 nm. The moles of iron-phenanthroline complex formed are related to moles of photons absorbed.

The solutions were prepared and stored in the dark (red light):

- 1) Potassium ferrioxalate solution: 294.8 mg of potassium ferrioxalate (commercially available from Alfa Aesar) and 139  $\mu$ L of sulfuric acid (96%) were added to a 50 mL volumetric flask, and filled to the mark with water (HPLC grade).
- 2) Phenanthroline solution: 0.2% by weight of 1,10-phenanthroline in water (100 mg in 50 mL volumetric flask).
- 3) Buffer solution: to a 50 mL volumetric flask 4.94 g of NaOAc and 0.5 mL of sulfuric acid (96%) were added and filled to the mark with water (HPLC grade).
- 4) Model reaction solution: phenylglyoxylic acid 1a (0.3 mmol), *trans*-cinnamaldehyde 2a (0.1 mmol), amine (20 mol %), and photocatalyst (5 mol %) in the acetonitrile (1.0 mL) was irradiated by white LED strips at  $-20\text{ }^{\circ}\text{C}$ .

The actinometry measurements were done as follows:

- 1) 1 mL of the actinometer solution was added to a quartz cuvette ( $l = 10\text{ mm}$ ). The cuvette was placed along with a sample solution (1 mL in a similar cuvette) whose quantum yield has to be measured (our model reaction). The sample and actinometry solutions (placed 10 cm away from the lamp) were irradiated with 13 W white LED strips for specified time intervals (5, 6, 7, 8) min.
- 2) After irradiation all the actinometer solution was removed and placed in a 25 mL volumetric flask. 0.5 mL of 1,10-phenanthroline solution and 2 mL of buffer solution was added to this flask and filled to the mark with water (HPLC grade).
- 3) The UV-Vis spectra of actinometry samples were recorded for each time interval. The absorbance of the actinometry solution was monitored at 510 nm.



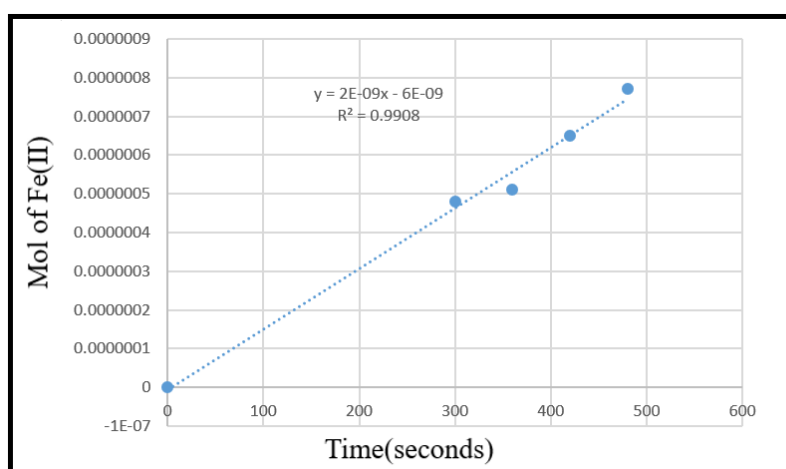
**Figure S4.** The absorbance of the actinometry solution was monitored at 510 nm.

4) The moles of  $\text{Fe}^{2+}$  formed for each sample is determined using Beers' Law:

$$\text{moles Fe}^{2+} = \frac{V_1 \times V_3 \times \Delta A(510 \text{ nm})}{10^3 \times V_2 \times l \times \varepsilon(510 \text{ nm})}$$

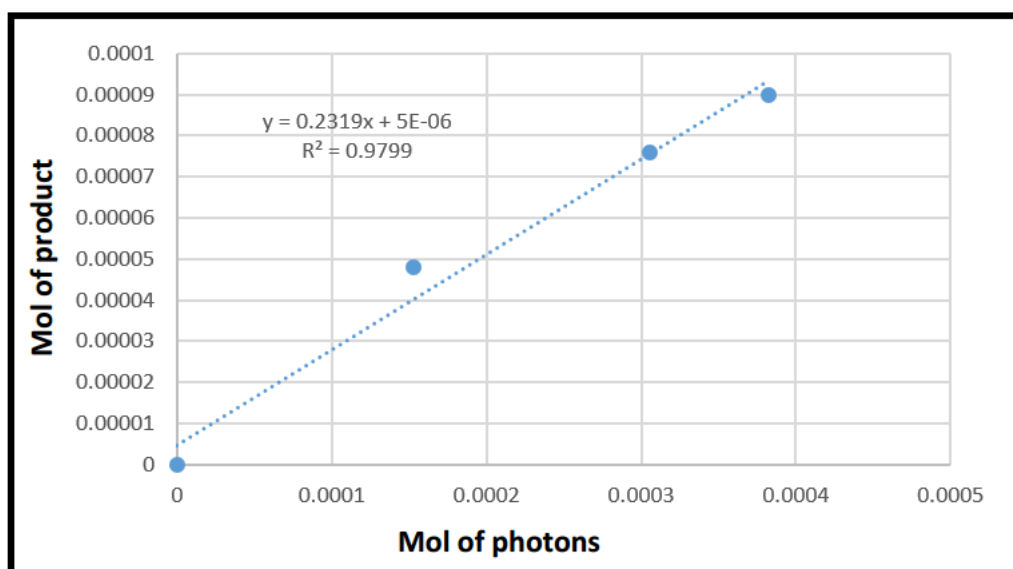
where  $V_1$  is the irradiated volume (1 mL),  $V_2$  is the aliquot of the irradiated solution taken for the determination of the ferrous ions (1 mL),  $V_3$  is the final volume after complexation with phenanthroline (25 mL),  $l$  is the optical path-length of the irradiation cell (1 cm),  $\Delta A(510 \text{ nm})$  the optical difference in absorbance between the irradiated solution and that taken in the dark,  $\varepsilon(510 \text{ nm})$  is that of the complex  $\text{Fe}(\text{phen})_3^{2+}$  ( $11100 \text{ L mol}^{-1} \text{ cm}^{-1}$ ).

5) The moles of  $\text{Fe}^{2+}$  formed ( $N$ ) are plotted as a function of time ( $t$ ). The slope is a product of the photon flux ( $F$ ) and the quantum yield for  $\text{Fe}^{2+}$  ( $\phi_{\text{Fe}^{2+}} = 1.13$ ) at 400 nm as,  $F = N / \phi_{\text{Fe}^{2+}} t$ . The  $F$  was determined to be  $1.77 \times 10^{-9} \text{ einstein s}^{-1}$ .



**Figure S5.** The plot of the moles of  $\text{Fe}^{2+}$  as a function of time ( $t$ ).

6) The moles of products formed for the reaction of interest (done by irradiating the sample alongside the actinometer solution) are described above. The moles of products formed were determined by column chromatography (The product will break down by GC measurement (FID detector)). The number of moles of product per unit time is related to the number of photons absorbed. The slope yields the quantum yield ( $\Phi$ ) of the photoreaction, 0.23.

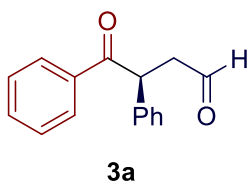


**Figure S6.** The plot of the number of moles of product per unit time as a function of the number of photons absorbed.

The procedure was repeated a second time to provide a similar value, a quantum yield ( $\Phi$ ) of 0.21.

The quantum yield ( $\Phi$ ) was determined to **0.22** (the average of two parallel experiments).

## 6. Characterization data of products 3

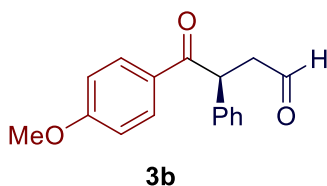


**(S)-4-oxo-3,4-diphenylbutanal (3a):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 90% (21.4 mg); white solid; m.p. 82-83 °C;  $[\alpha]_D^{20} = +155.5$  (c 2.08, CHCl<sub>3</sub>);  $[\alpha]_D^{26} = +120.1$  (c 1.15, CH<sub>2</sub>Cl<sub>2</sub>); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  9.80 (s, 1H), 7.96 (dd,  $J = 5.2, 3.3$  Hz, 2H), 7.52 – 7.44 (m, 1H), 7.37 (dd,  $J = 10.5, 4.7$  Hz, 2H), 7.32 – 7.25 (m, 4H), 7.24 – 7.18 (m, 1H), 5.13 (dd,  $J = 9.6, 4.2$  Hz, 1H), 3.61 (dd,  $J = 18.5, 9.7$  Hz, 1H), 2.83

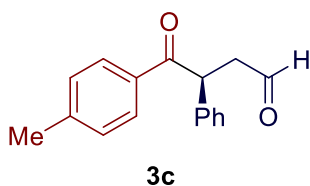


(dd,  $J = 18.6, 4.2$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  200.1, 198.2, 138.3, 136.0, 133.1, 129.3, 128.9, 128.6, 128.1, 127.5, 48.3, 47.6; ESI FTMS exact mass calcd for  $(\text{C}_{16}\text{H}_{14}\text{O}_2+\text{H})^+$  requires  $m/z$  239.1072, found  $m/z$  239.1073; Enantiomeric excess: 80%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min,  $T = 25^\circ\text{C}$ , 220 nm):  $t_R = 17.276$  min (major),  $t_R = 12.359$  min (minor).

The absolute configuration of product **3a** was determined to be *S* by comparing its optical rotation  $[\alpha]_D^{26} = +120.1$  with that of the same known compound  $[\alpha]_D^{26} = +52.9$ .<sup>2</sup> The absolute configurations of other products were assigned by analogy.



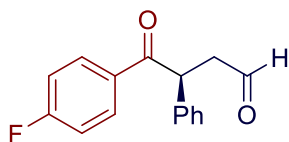
**(S)-4-(4-methoxyphenyl)-4-oxo-3-phenylbutanal (3b):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 93% (24.9mg); pale yellow oil;  $[\alpha]_D^{20} = +145.8$  (c 1.24,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.81 (s, 1H), 7.99 – 7.92 (m, 2H), 7.32 – 7.25 (m, 4H), 7.24 – 7.19 (m, 1H), 6.89 – 6.83 (m, 2H), 5.09 (dd,  $J = 9.5, 4.3$  Hz, 1H), 3.81 (s, 3H), 3.58 (dd,  $J = 18.4, 9.5$  Hz, 1H), 2.81 (dd,  $J = 18.5, 4.3$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  200.2, 196.6, 163.5, 138.8, 131.3, 129.2, 128.9, 128.0, 127.4, 113.7, 55.4, 48.3, 47.4; ESI FTMS exact mass calcd for  $(\text{C}_{17}\text{H}_{16}\text{O}_3+\text{H})^+$  requires  $m/z$  269.1177, found  $m/z$  269.1173; Enantiomeric excess: 68%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min,  $T = 25^\circ\text{C}$ , 220 nm):  $t_R = 31.651$  min (major),  $t_R = 26.172$  min (minor).



**(S)-4-oxo-3-phenyl-4-(p-tolyl)butanal (3c):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 92% (23.4 mg); pale yellow oil;  $[\alpha]_D^{20} = +141.7$  (c 1.16,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.81 (s, 1H), 7.87 (d,  $J = 8.3$  Hz, 2H), 7.32 – 7.24 (m, 4H), 7.23 – 7.19 (m, 1H), 7.17 (d,  $J = 8.0$  Hz, 2H), 5.11 (dd,  $J = 9.5, 4.3$  Hz, 1H), 3.59 (dd,  $J = 18.5, 9.6$  Hz, 1H), 2.82 (dd,  $J = 18.5, 4.3$  Hz, 1H), 2.34 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$

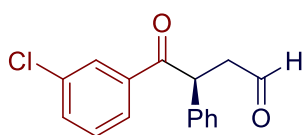
<sup>2</sup>Goti, G.; Bieszczad, B.; Vega-Peñaloza, A.; Melchiorre, P. *Angew. Chem. Int. Ed.* 10.1002/anie.201810798.

200.1, 197.7, 143.9, 138.5, 133.4, 129.2, 129.1, 128.1, 127.4, 48.2, 47.5, 21.6; ESI FTMS exact mass calcd for  $(C_{17}H_{16}O_2+H)^+$  requires  $m/z$  253.1229, found  $m/z$  253.1223; Enantiomeric excess: 68%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 °C, 220 nm):  $t_R$  = 20.145 min (major),  $t_R$  = 15.144 min (minor).



**3d**

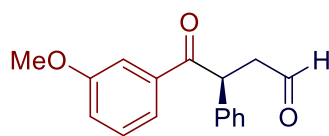
**(S)-4-(4-fluorophenyl)-4-oxo-3-phenylbutanal (3d):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 91% (23.3 mg); pale yellow solid; m.p. 95-96 °C;  $[\alpha]_D^{20}$  = +135.5 (c 1.16,  $CHCl_3$ );  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  9.81 (s, 1H), 8.02 – 7.95 (m, 2H), 7.34 – 7.28 (m, 2H), 7.26 – 7.19 (m, 3H), 7.09 – 7.01 (m, 2H), 5.07 (dd,  $J$  = 9.7, 4.1 Hz, 1H), 3.62 (dd,  $J$  = 18.7, 9.8 Hz, 1H), 2.83 (dd,  $J$  = 18.7, 4.1 Hz, 1H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  199.9, 196.6, 165.6 ( $J$  = 255.1 Hz), 138.1, 132.4, 131.6 ( $J$  = 9.3 Hz), 129.4, 128.0, 127.6, 115.7 ( $J$  = 21.9 Hz), 48.3, 47.6; ESI FTMS exact mass calcd for  $(C_{16}H_{13}FO_2+H)^+$  requires  $m/z$  257.0978, found  $m/z$  257.0977; Enantiomeric excess: 74%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 °C, 220 nm):  $t_R$  = 10.511 min (major),  $t_R$  = 9.625 min (minor).



**3e**

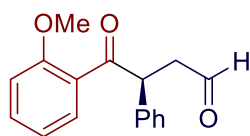
**(S)-4-(3-chlorophenyl)-4-oxo-3-phenylbutanal (3e):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 71% (19.3 mg); pale yellow oil;  $[\alpha]_D^{20}$  = +137.2 (c 0.97,  $CHCl_3$ );  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  9.73 (s, 1H), 7.86 (t,  $J$  = 1.8 Hz, 1H), 7.75 (d,  $J$  = 7.8 Hz, 1H), 7.42 – 7.33 (m, 1H), 7.28 – 7.21 (m, 3H), 7.21 – 7.14 (m, 3H), 4.98 (dd,  $J$  = 9.8, 4.0 Hz, 1H), 3.55 (dd,  $J$  = 18.7, 9.8 Hz, 1H), 2.77 (dd,  $J$  = 18.8, 4.0 Hz, 1H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  198.8, 195.9, 136.6, 133.9, 131.9, 128.8, 128.4, 127.9, 127.0, 126.7, 125.9, 47.3, 46.6; ESI FTMS exact mass calcd for  $(C_{16}H_{13}ClO_2+H)^+$  requires  $m/z$  273.0682, found  $m/z$  273.0680; Enantiomeric excess: 78%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 °C, 220 nm):  $t_R$  = 10.184 min (major),  $t_R$  = 9.157

min (minor).



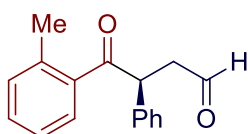
**3f**

**(S)-4-(3-methoxyphenyl)-4-oxo-3-phenylbutanal (3f):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 91% (24.4 mg); pale yellow solid; m.p. 110-111 °C;  $[\alpha]_D^{20} = +144.2$  (c 1.22, CHCl<sub>3</sub>); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.81 (s, 1H), 7.55 (d, *J* = 7.8 Hz, 1H), 7.50 – 7.46 (m, 1H), 7.33 – 7.25 (m, 5H), 7.25 – 7.19 (m, 1H), 7.06 – 7.00 (m, 1H), 5.10 (dd, *J* = 9.6, 4.2 Hz, 1H), 3.79 (s, 3H), 3.60 (dd, *J* = 18.6, 9.7 Hz, 1H), 2.84 (dd, *J* = 18.6, 4.3 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 200.0, 197.9, 159.7, 138.2, 137.3, 129.5, 129.3, 128.1, 127.5, 121.6, 119.7, 113.2, 55.4, 48.3, 47.8; ESI FTMS exact mass calcd for (C<sub>17</sub>H<sub>16</sub>O<sub>3</sub>+H)<sup>+</sup> requires *m/z* 269.1178, found *m/z* 269.1172; Enantiomeric excess: 74%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 °C, 220 nm): *t<sub>R</sub>* = 25.453 min (major), *t<sub>R</sub>* = 19.149 min (minor).



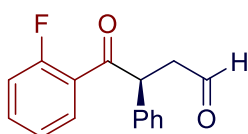
**3g**

**(S)-4-(2-methoxyphenyl)-4-oxo-3-phenylbutanal (3g):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 99% (26.5 mg); yellow oil;  $[\alpha]_D^{20} = +64.2$  (c 1.33, CHCl<sub>3</sub>); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.82 (s, 1H), 7.63 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.41 – 7.33 (m, 1H), 7.26 – 7.21 (m, 2H), 7.21 – 7.16 (m, 3H), 6.95 – 6.89 (m, 1H), 6.84 (d, *J* = 8.3 Hz, 1H), 5.23 (dd, *J* = 9.0, 5.2 Hz, 1H), 3.81 (s, 3H), 3.47 (dd, *J* = 17.9, 9.0 Hz, 1H), 2.77 (dd, *J* = 17.9, 5.2 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 200.6, 200.4, 158.0, 138.4, 133.6, 131.0, 128.7, 128.5, 127.4, 127.1, 120.6, 111.5, 55.4, 51.9, 47.8; ESI FTMS exact mass calcd for (C<sub>17</sub>H<sub>16</sub>O<sub>3</sub>+H)<sup>+</sup> requires *m/z* 269.1178, found *m/z* 269.1174; Enantiomeric excess: 76%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 °C, 220 nm): *t<sub>R</sub>* = 33.029 min (major), *t<sub>R</sub>* = 23.081 min (minor).



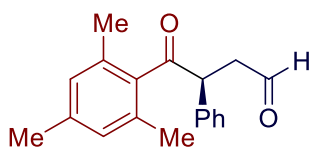
**3h**

**(S)-4-oxo-3-phenyl-4-(o-tolyl)butanal (3h):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 89% (22.4 mg); colorless oil;  $[\alpha]_D^{20} = +59.2$  (c 1.12,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.84 (s, 1H), 7.74 (d,  $J = 7.5$  Hz, 1H), 7.32 – 7.24 (m, 3H), 7.24 – 7.16 (m, 4H), 7.13 (d,  $J = 7.6$  Hz, 1H), 4.96 (dd,  $J = 10.1, 3.8$  Hz, 1H), 3.68 (dd,  $J = 18.7, 10.2$  Hz, 1H), 2.82 (dd,  $J = 18.7, 3.8$  Hz, 1H), 2.26 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  202.0, 200.1, 138.3, 137.9, 137.3, 131.5, 131.1, 129.1, 128.3, 127.5, 125.5, 50.4, 47.7, 20.6; ESI FTMS exact mass calcd for  $(\text{C}_{17}\text{H}_{16}\text{O}_2 + \text{H})^+$  requires  $m/z$  253.1229, found  $m/z$  253.1224; Enantiomeric excess: 80%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min,  $T = 25^\circ\text{C}$ , 220 nm):  $t_R = 11.448$  min (minor),  $t_R = 12.229$  min (major).



**3i**

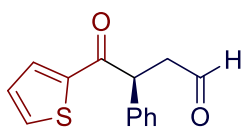
**(S)-4-(2-fluorophenyl)-4-oxo-3-phenylbutanal (3i):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 70% (17.9 mg); pale yellow oil;  $[\alpha]_D^{20} = +46.4$  (c 0.90,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.74 (s, 1H), 7.81 – 7.69 (m, 1H), 7.41 – 7.30 (m, 1H), 7.23 – 7.11 (m, 5H), 7.07 (t,  $J = 7.6$  Hz, 1H), 6.99 – 6.91 (m, 1H), 4.99 (dd,  $J = 9.6, 4.4$  Hz, 1H), 3.50 (dd,  $J = 18.5, 9.6$  Hz, 1H), 2.74 (dd,  $J = 18.5, 4.4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  200.0, 196.7, 161.2 ( $J = 255.5$  Hz), 137.2, 134.6 ( $J = 9.2$  Hz), 131.2 ( $J = 2.4$  Hz), 129.0, 128.5, 127.5, 125.1, 124.4, 116.7 ( $J = 23.9$  Hz), 51.3 ( $J = 6.8$  Hz), 48.1; ESI FTMS exact mass calcd for  $(\text{C}_{16}\text{H}_{13}\text{FO}_2 + \text{H})^+$  requires  $m/z$  257.0978, found  $m/z$  257.0975; Enantiomeric excess: 74%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min,  $T = 25^\circ\text{C}$ , 220 nm):  $t_R = 15.262$  min (major),  $t_R = 12.207$  min (minor).



**3j**

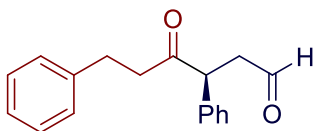
**(S)-4-mesityl-4-oxo-3-phenylbutanal (3j):** Preparative thin layer chromatography, petroleum

ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 97% (27.2 mg); yellow solid; m.p. 121-122 °C;  $[\alpha]_D^{20} = +189.8$  (c 1.37, CHCl<sub>3</sub>); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.86 (s, 1H), 7.25 – 7.19 (m, 3H), 7.12 – 7.07 (m, 2H), 6.71 (s, 2H), 4.70 (dd, *J* = 7.3, 6.7 Hz, 1H), 3.56 (dd, *J* = 18.0, 7.6 Hz, 1H), 3.05 (dd, *J* = 18.0, 6.4 Hz, 1H), 2.22 (s, 3H), 1.94 (s, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 206.6, 199.9, 138.7, 137.4, 135.7, 133.8, 129.0, 128.7, 128.5, 128.3, 127.7, 125.5, 124.8, 53.7, 44.5, 21.1, 19.4; ESI FTMS exact mass calcd for (C<sub>19</sub>H<sub>20</sub>O<sub>2</sub>+H)<sup>+</sup> requires *m/z* 281.1542, found *m/z* 281.1537; Enantiomeric excess: 74%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 °C, 220 nm): *t<sub>R</sub>* = 10.310 min (major), *t<sub>R</sub>* = 12.322 min (minor).



**3k**

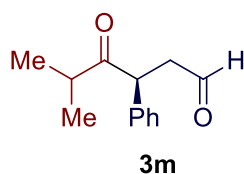
**(S)-4-oxo-3-phenyl-4-(thiophen-2-yl)butanal (3k):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 44% (10.7 mg); yellow solid; m.p. 89-90 °C;  $[\alpha]_D^{20} = +129.8$  (c 0.54, CHCl<sub>3</sub>); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.80 (s, 1H), 7.72 (d, *J* = 3.8 Hz, 1H), 7.57 (d, *J* = 4.9 Hz, 1H), 7.32 (d, *J* = 4.3 Hz, 4H), 7.27 – 7.22 (m, 1H), 7.08 – 7.01 (m, 1H), 4.95 (dd, *J* = 9.5, 4.4 Hz, 1H), 3.59 (dd, *J* = 18.6, 9.5 Hz, 1H), 2.84 (dd, *J* = 18.6, 4.4 Hz, 1H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 199.8, 190.9, 142.9, 138.4, 133.9, 133.0, 129.3, 128.1, 127.7, 48.9, 47.9; ESI FTMS exact mass calcd for (C<sub>14</sub>H<sub>12</sub>O<sub>2</sub>S+H)<sup>+</sup> requires *m/z* 245.0636, found *m/z* 245.0633; Enantiomeric excess: 62%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min, T = 25 °C, 220 nm): *t<sub>R</sub>* = 31.693 min (major), *t<sub>R</sub>* = 21.162 min (minor).



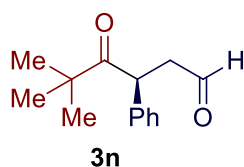
**3l**

**(S)-4-oxo-3,6-diphenylhexanal (3l):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 64% (17.0 mg); colorless oil;  $[\alpha]_D^{20} = +128.9$  (c 0.85, CHCl<sub>3</sub>); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 9.75 (s, 1H), 7.33 – 7.24 (m, 3H), 7.21 (t, *J* = 7.3 Hz, 2H), 7.14 (t, *J* = 7.3 Hz, 3H), 7.05 (d, *J* = 7.1 Hz, 2H), 4.21 (dd, *J* = 9.8, 4.1 Hz, 1H), 3.46 (dd, *J* = 18.6,

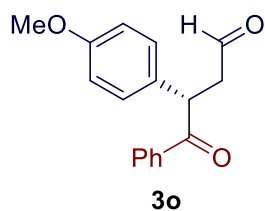
9.9 Hz, 1H), 2.91 – 2.62 (m, 5H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  207.6, 200.1, 140.8, 137.3, 129.3, 128.4, 128.2, 127.7, 126.0, 52.2, 46.7, 42.8, 29.7; ESI FTMS exact mass calcd for  $(\text{C}_{18}\text{H}_{18}\text{O}_2+\text{H})^+$  requires  $m/z$  267.1385, found  $m/z$  267.1381; Enantiomeric excess: 70%, determined by HPLC (Daicel Chiralpak IG-3, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min,  $T = 25\text{ }^\circ\text{C}$ , 192 nm):  $t_R = 9.668$  min (major),  $t_R = 9.111$  min (minor).



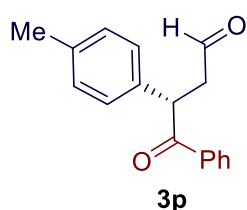
**(S)-5-methyl-4-oxo-3-phenylhexanal (3m):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 75% (15.3 mg); colorless oil;  $[\alpha]_D^{20} = +216.3$  (c 1.07,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.75 (s, 1H), 7.36 – 7.30 (m, 2H), 7.30 – 7.24 (m, 1H), 7.22 – 7.15 (m, 2H), 4.40 (dd,  $J = 9.9, 4.1$  Hz, 1H), 3.45 (dd,  $J = 18.5, 9.9$  Hz, 1H), 2.71 (dt,  $J = 13.8, 6.9$  Hz, 1H), 2.63 (dd,  $J = 18.5, 4.1$  Hz, 1H), 1.16 (d,  $J = 7.1$  Hz, 3H), 0.89 (d,  $J = 6.7$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  212.5, 200.0, 137.7, 129.2, 128.3, 127.6, 50.6, 47.1, 39.4, 19.2, 18.2; ESI FTMS exact mass calcd for  $(\text{C}_{13}\text{H}_{16}\text{O}_2+\text{H})^+$  requires  $m/z$  205.1228, found  $m/z$  205.1229; Enantiomeric excess: 72%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 95/5, flow rate 1.0 mL/min,  $T = 25\text{ }^\circ\text{C}$ , 220 nm):  $t_R = 9.281$  min (major),  $t_R = 11.081$  min (minor).



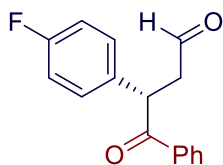
**(S)-5,5-dimethyl-4-oxo-3-phenylhexanal (3n):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 68% (14.8 mg); colorless oil;  $[\alpha]_D^{20} = +190.4$  (c 0.79,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.68 (s, 1H), 7.35 – 7.28 (m, 2H), 7.27 – 7.22 (m, 1H), 7.22 – 7.16 (m, 2H), 4.64 (dd,  $J = 9.9, 4.1$  Hz, 1H), 3.42 (dd,  $J = 18.6, 9.9$  Hz, 1H), 2.65 (dd,  $J = 18.6, 4.1$  Hz, 1H), 1.09 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  214.2, 200.0, 138.1, 129.1, 128.1, 127.4, 49.4, 46.9, 45.1, 27.3; ESI FTMS exact mass calcd for  $(\text{C}_{14}\text{H}_{18}\text{O}_2+\text{H})^+$  requires  $m/z$  219.1385, found  $m/z$  219.1386; Enantiomeric excess: 80%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 95/5, flow rate 1.0 mL/min,  $T = 25\text{ }^\circ\text{C}$ , 220 nm):  $t_R = 7.045$  min (major),  $t_R = 8.062$  min (minor).



**(S)-3-(4-methoxyphenyl)-4-oxo-4-phenylbutanal (3o):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 74% (19.8 mg); pale yellow oil;  $[\alpha]_D^{20} = +112.2$  (c 0.99,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.72 (s, 1H), 7.88 (d,  $J = 7.5$  Hz, 2H), 7.40 (t,  $J = 7.3$  Hz, 1H), 7.30 (t,  $J = 7.5$  Hz, 2H), 7.10 (d,  $J = 8.5$  Hz, 2H), 6.74 (d,  $J = 8.5$  Hz, 2H), 5.00 (dd,  $J = 9.4, 4.3$  Hz, 1H), 3.66 (s, 3H), 3.48 (dd,  $J = 18.5, 9.5$  Hz, 1H), 2.74 (dd,  $J = 18.5, 4.3$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  200.3, 198.4, 158.9, 136.0, 133.0, 130.1, 129.2, 128.9, 128.5, 114.7, 55.2, 48.3, 46.7; ESI FTMS exact mass calcd for  $(\text{C}_{17}\text{H}_{16}\text{O}_3+\text{H})^+$  requires  $m/z$  269.1178, found  $m/z$  269.1176; Enantiomeric excess: 68%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min,  $T = 25^\circ\text{C}$ , 220 nm):  $t_R = 27.571$  min (major),  $t_R = 19.673$  min (minor).

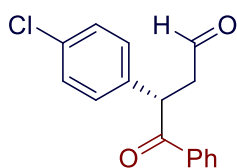


**(S)-4-oxo-4-phenyl-3-(p-tolyl)butanal (3p):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 89% (22.4 mg); pale yellow solid; m.p. 108-109  $^\circ\text{C}$ ;  $[\alpha]_D^{20} = +124.3$  (c 1.12,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.81 (s, 1H), 8.01 – 7.92 (m, 2H), 7.47 (t,  $J = 7.4$  Hz, 1H), 7.37 (t,  $J = 7.6$  Hz, 2H), 7.15 (d,  $J = 8.1$  Hz, 2H), 7.10 (d,  $J = 8.0$  Hz, 2H), 5.09 (dd,  $J = 9.6, 4.3$  Hz, 1H), 3.58 (dd,  $J = 18.5, 9.6$  Hz, 1H), 2.81 (dd,  $J = 18.5, 4.3$  Hz, 1H), 2.27 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  200.2, 198.3, 137.2, 136.0, 135.2, 133.0, 129.9, 128.9, 128.5, 127.9, 48.3, 47.2, 21.0; ESI FTMS exact mass calcd for  $(\text{C}_{17}\text{H}_{16}\text{O}_2+\text{H})^+$  requires  $m/z$  253.1228, found  $m/z$  253.1224; Enantiomeric excess: 72%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/ 10, flow rate 1.0 mL/min,  $T = 25^\circ\text{C}$ , 220 nm):  $t_R = 18.431$  min (major),  $t_R = 13.323$  min (minor).



**3q**

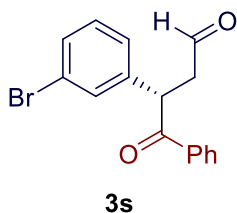
**(S)-3-(4-fluorophenyl)-4-oxo-4-phenylbutanal (3q):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 82% (21.0 mg); colorless oil;  $[\alpha]_D^{20} = +112.7$  (c 1.05,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.72 (s, 1H), 7.94 – 7.78 (m, 2H), 7.42 (t,  $J = 7.4$  Hz, 1H), 7.31 (t,  $J = 7.6$  Hz, 2H), 7.20 – 7.13 (m, 2H), 6.99 – 6.80 (m, 2H), 5.05 (dd,  $J = 9.4, 4.4$  Hz, 1H), 3.51 (dd,  $J = 18.6, 9.5$  Hz, 1H), 2.75 (dd,  $J = 18.6, 4.4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  199.8, 198.1, 162.1 ( $J = 246.7$  Hz), 160.8, 135.8, 133.9 ( $J = 3.3$  Hz), 133.2, 129.7 ( $J = 8.1$  Hz), 128.9, 128.6, 116.2 ( $J = 21.5$  Hz), 48.3, 46.6; ESI FTMS exact mass calcd for  $(\text{C}_{16}\text{H}_{13}\text{FO}_2 + \text{H})^+$  requires  $m/z$  257.0978, found  $m/z$  257.0977; Enantiomeric excess: 70%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min,  $T = 25^\circ\text{C}$ , 220 nm):  $t_R = 16.221$  min (major),  $t_R = 11.609$  min (minor).



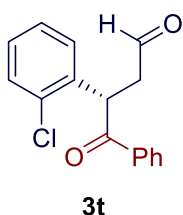
**3r**

**(S)-3-(4-chlorophenyl)-4-oxo-4-phenylbutanal (3r):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 77% (20.9 mg); colorless oil;  $[\alpha]_D^{20} = +97.1$  (c 1.05,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.80 (s, 1H), 7.98 – 7.90 (m, 2H), 7.54 – 7.47 (m, 1H), 7.43 – 7.36 (m, 2H), 7.29 – 7.25 (m, 2H), 7.24 – 7.19 (m, 2H), 5.12 (dd,  $J = 9.4, 4.4$  Hz, 1H), 3.59 (dd,  $J = 18.6, 9.5$  Hz, 1H), 2.83 (dd,  $J = 18.7, 4.4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  199.6, 197.8, 136.7, 135.8, 133.5, 133.3, 129.5, 129.4, 128.9, 128.6, 48.1, 46.7; ESI FTMS exact mass calcd for  $(\text{C}_{16}\text{H}_{13}\text{ClO}_2 + \text{H})^+$  requires  $m/z$  273.0682, found  $m/z$  273.0685; Enantiomeric excess: 76%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min,  $T = 25^\circ\text{C}$ , 220 nm):  $t_R = 14.691$  min (major),  $t_R = 11.123$  min (minor).

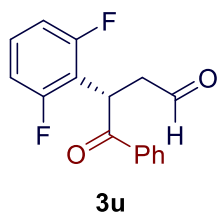




**(S)-3-(3-bromophenyl)-4-oxo-4-phenylbutanal (3s):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 92% (29.1 mg); colorless oil;  $[\alpha]_D^{20} = +247.1$  (c 1.46,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.79 (s, 1H), 8.01 – 7.90 (m, 2H), 7.51 (t,  $J = 7.4$  Hz, 1H), 7.47 – 7.38 (m, 3H), 7.37 – 7.33 (m, 1H), 7.24 – 7.13 (m, 2H), 5.10 (dd,  $J = 9.6, 4.2$  Hz, 1H), 3.60 (dd,  $J = 18.7, 9.6$  Hz, 1H), 2.84 (dd,  $J = 18.7, 4.2$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  199.5, 197.6, 140.5, 135.7, 133.4, 131.0, 130.8, 130.7, 128.9, 128.7, 126.8, 123.2, 48.2, 46.9; ESI FTMS exact mass calcd for  $(\text{C}_{16}\text{H}_{13}\text{BrO}_2 + \text{H})^+$  requires  $m/z$  317.0177, found  $m/z$  317.0180; Enantiomeric excess: 74%, determined by HPLC (Daicel Chiralpak IC, hexane/isopropanol = 90/10, flow rate 1.0 mL/min,  $T = 25$  °C, 220 nm):  $t_R = 14.231$  min (major),  $t_R = 10.801$  min (minor).

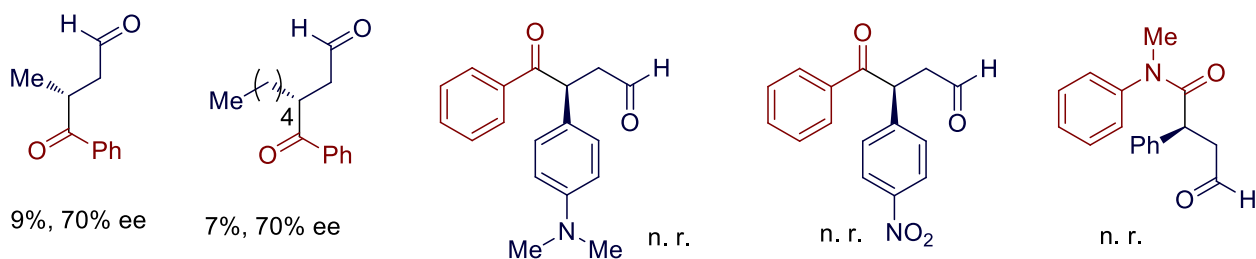


**(S)-3-(2-chlorophenyl)-4-oxo-4-phenylbutanal (3t):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 79% (21.5 mg); pale yellow oil;  $[\alpha]_D^{20} = +170.6$  (c 1.07,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.83 (s, 1H), 7.92 (dd,  $J = 5.2, 3.3$  Hz, 2H), 7.53 – 7.47 (m, 1H), 7.46 – 7.35 (m, 3H), 7.21 – 7.10 (m, 2H), 7.10 – 7.06 (m, 1H), 5.59 (dd,  $J = 10.2, 3.5$  Hz, 1H), 3.48 (dd,  $J = 18.3, 10.2$  Hz, 1H), 2.76 (dd,  $J = 18.3, 3.5$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  199.5, 197.8, 136.0, 135.6, 133.3, 133.0, 130.3, 128.9, 128.8, 128.6, 127.7, 46.5, 44.2; ESI FTMS exact mass calcd for  $(\text{C}_{16}\text{H}_{13}\text{ClO}_2 + \text{H})^+$  requires  $m/z$  273.0682, found  $m/z$  273.0677; Enantiomeric excess: 62%, determined by HPLC (Daicel Chiralpak IC, hexane/isopropanol = 90/ 10, flow rate 1.0 mL/min,  $T = 25$  °C, 220 nm):  $t_R = 14.590$  min (major),  $t_R = 11.179$  min (minor).

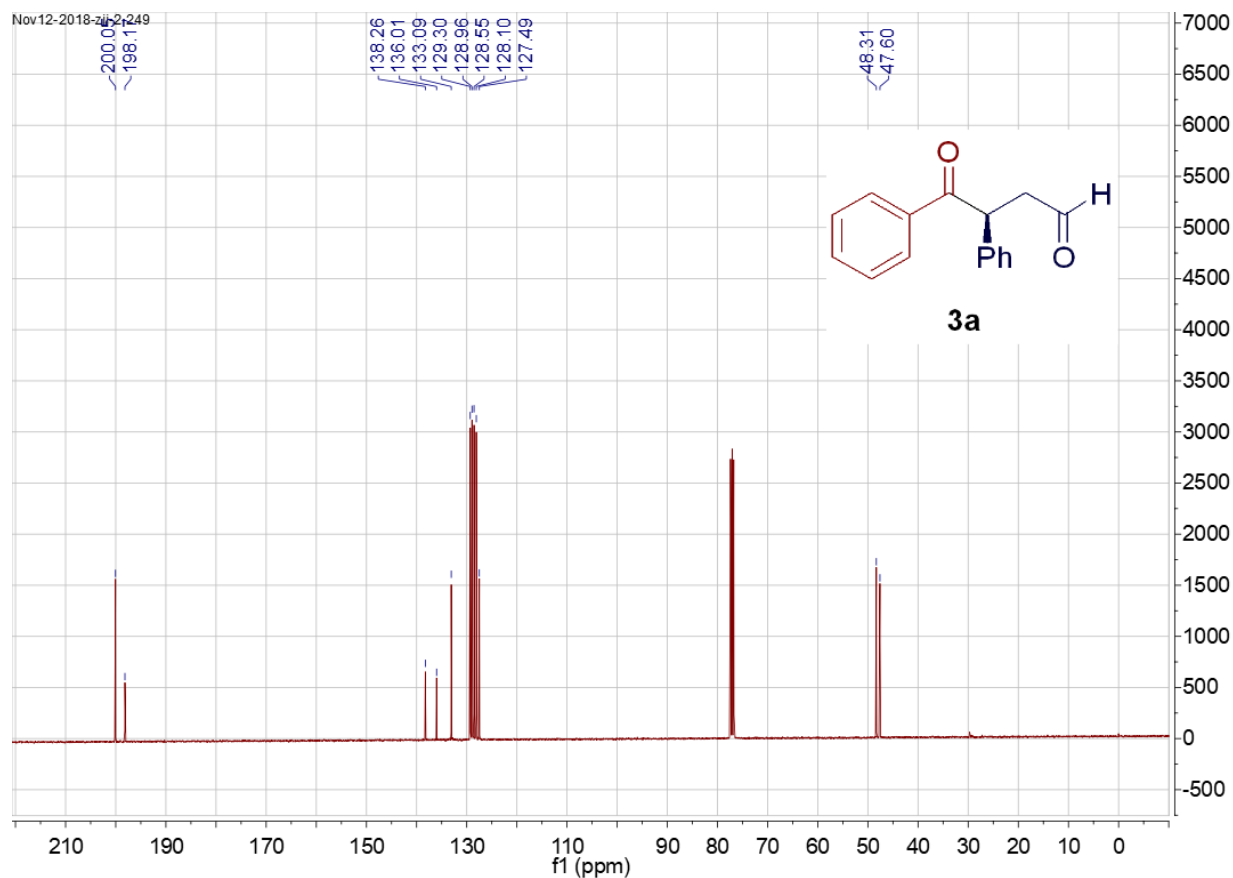
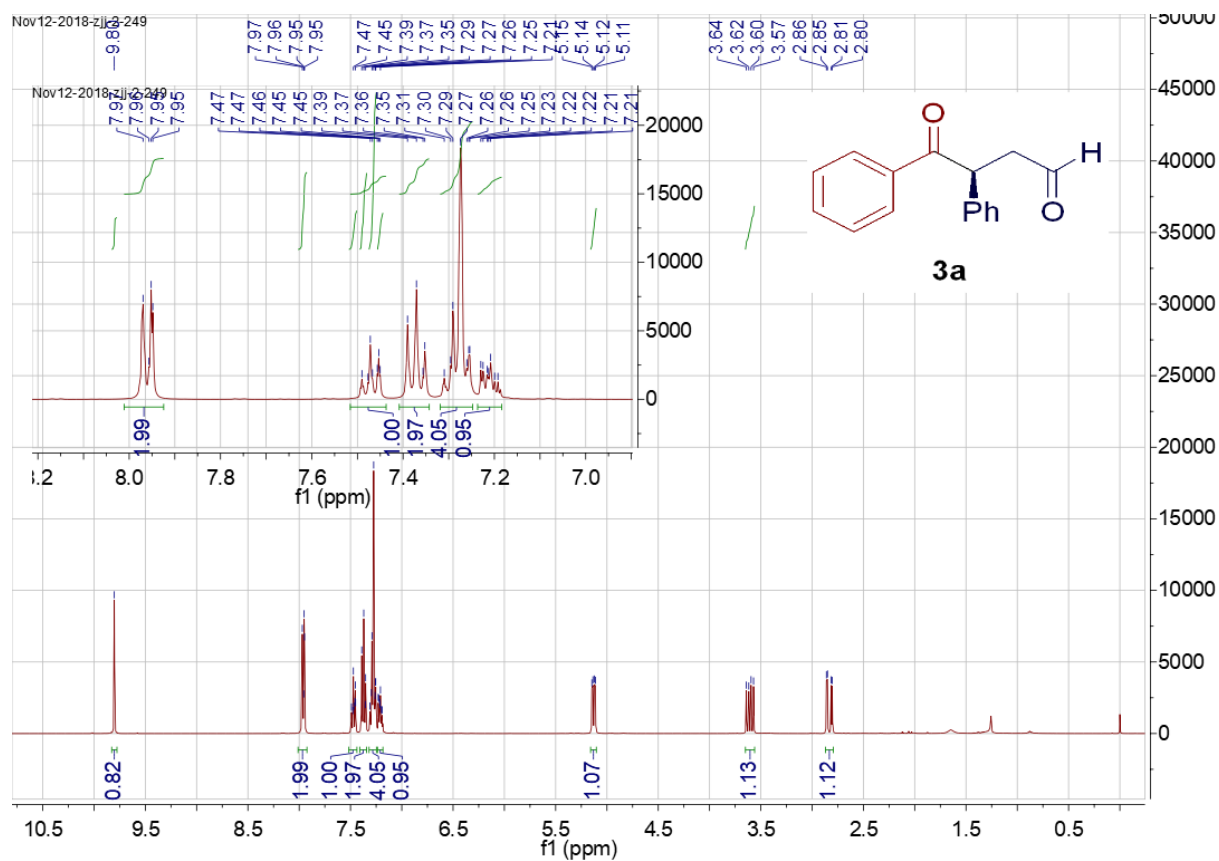


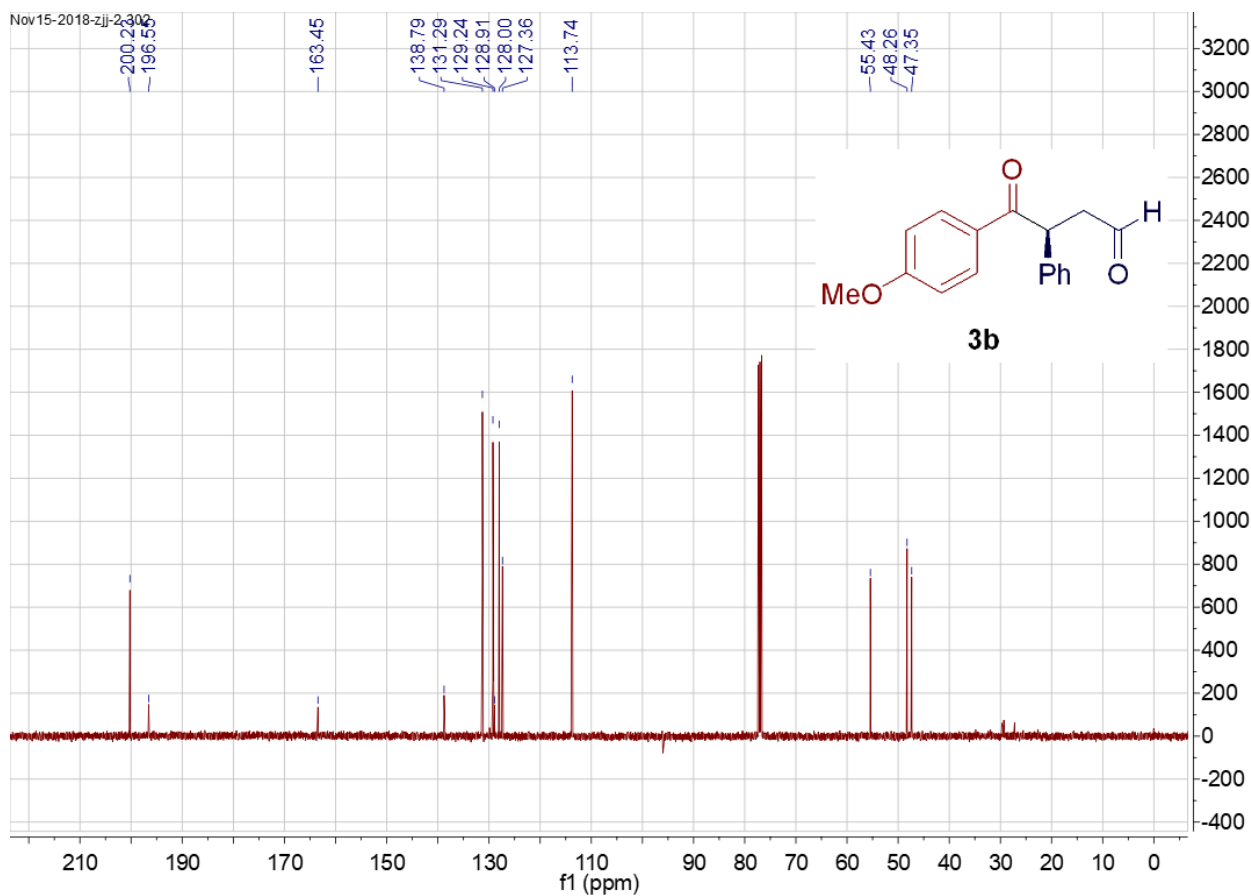
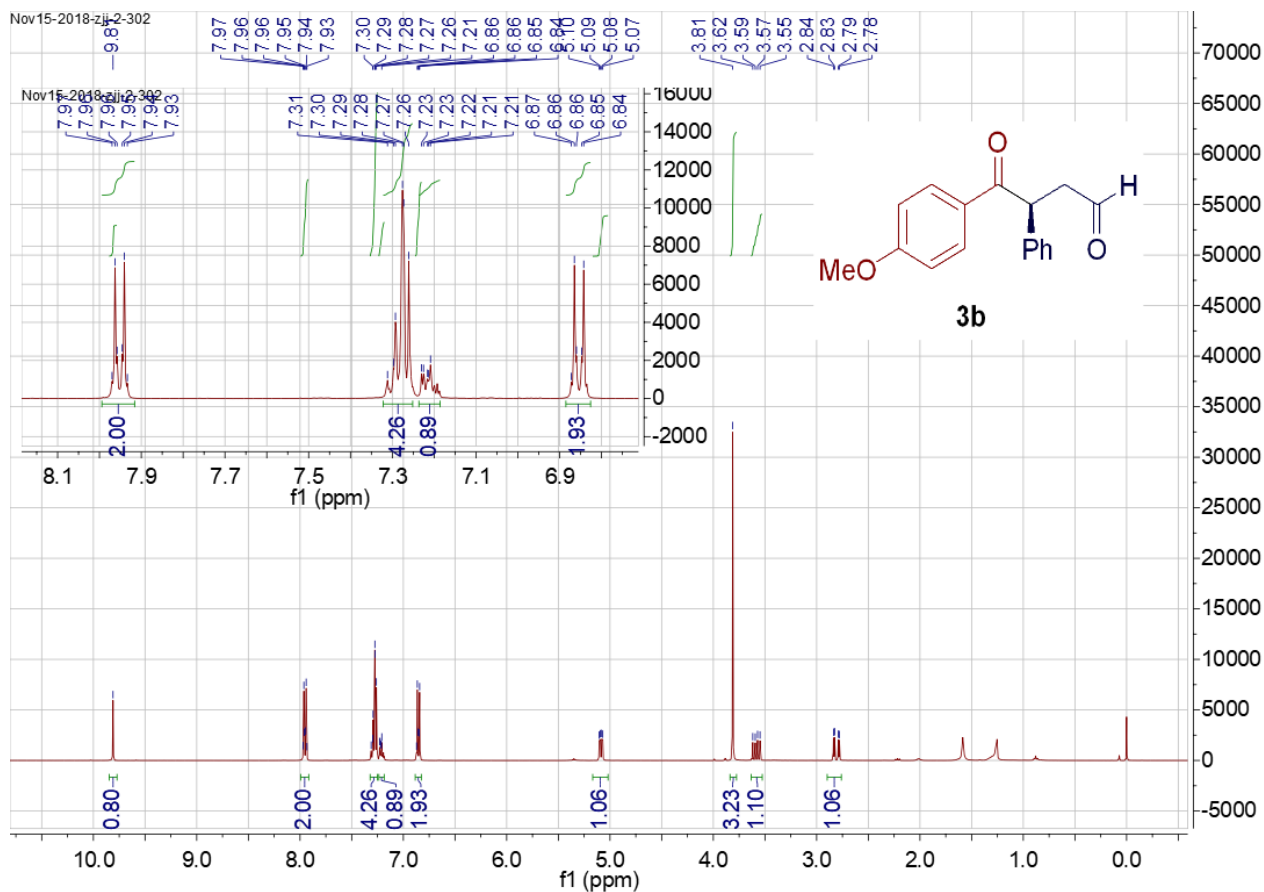
**(S)-3-(2,6-difluorophenyl)-4-oxo-4-phenylbutanal (3u):** Preparative thin layer chromatography, petroleum ether/ethyl acetate = 10/1; reaction time = 60 h; yield: 93% (25.5 mg); colorless oil;  $[\alpha]_D^{20} = +128.1$  (c 1.27,  $\text{CHCl}_3$ );  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  9.83 (s, 1H), 7.79 (d,  $J = 7.3$  Hz, 2H), 7.40 (t,  $J = 7.4$  Hz, 1H), 7.29 (t,  $J = 7.7$  Hz, 2H), 7.15 – 7.05 (m, 1H), 6.77 (t,  $J = 8.2$  Hz, 2H), 5.31 (dd,  $J = 9.1, 4.3$  Hz, 1H), 3.60 (dd,  $J = 18.0, 9.1$  Hz, 1H), 2.63 (dd,  $J = 18.0, 4.4$  Hz, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  198.3, 195.0, 159.6 ( $J = 249.2$  Hz), 158.4, 134.4, 132.1, 128.6 ( $J = 10.4$  Hz), 127.4 ( $J = 17.8$  Hz), 114.3, 111.1, 110.9 ( $J = 25.2$  Hz), 42.7, 36.9; ESI FTMS exact mass calcd for  $(\text{C}_{16}\text{H}_{12}\text{F}_2\text{O}_2 + \text{H})^+$  requires  $m/z$  275.0883, found  $m/z$  275.0878; Enantiomeric excess: 76%, determined by HPLC (Daicel Chiralpak IC, hexane/ isopropanol = 90/10, flow rate 1.0 mL/min,  $T = 25^\circ\text{C}$ , 220 nm):  $t_R = 19.560$  min (major),  $t_R = 13.253$  min (minor).

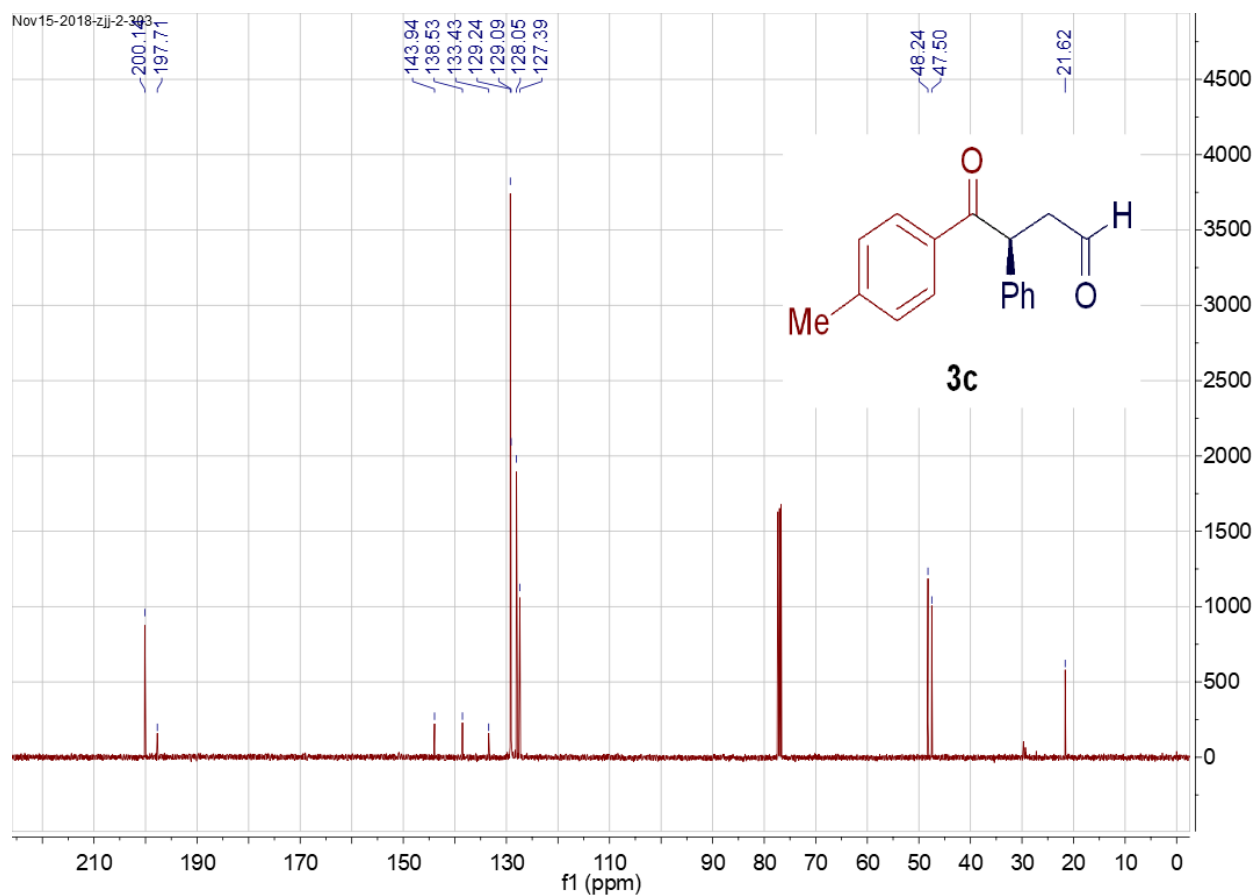
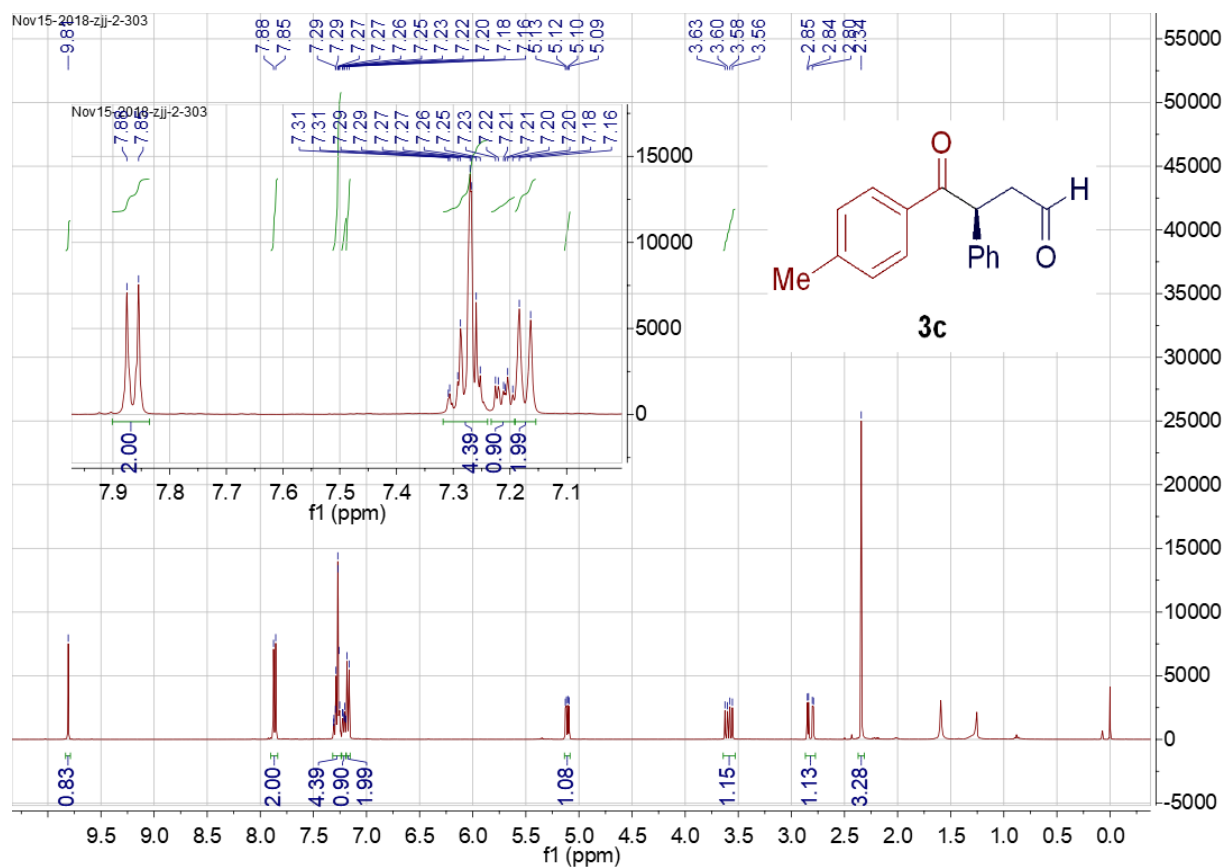
## 7. Unsuccessful Substrates

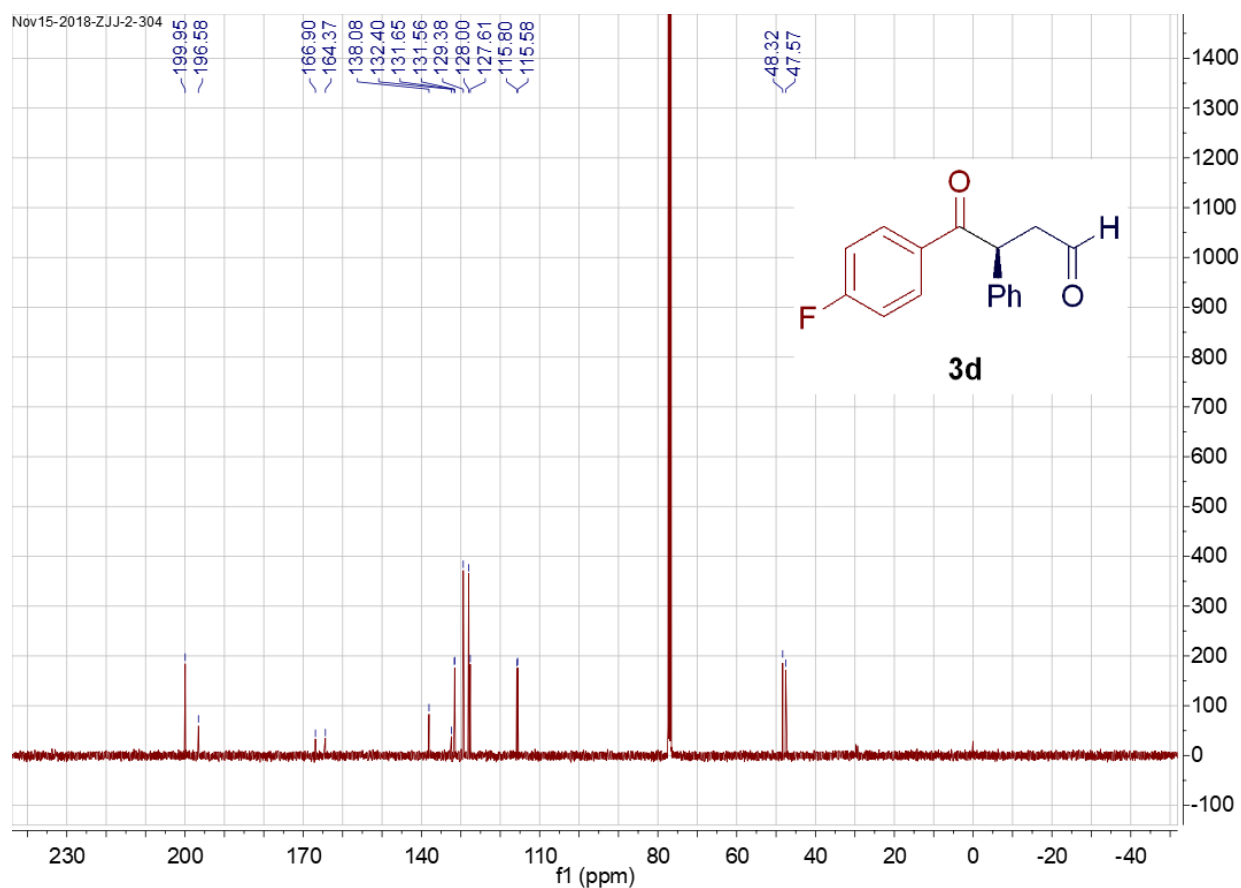
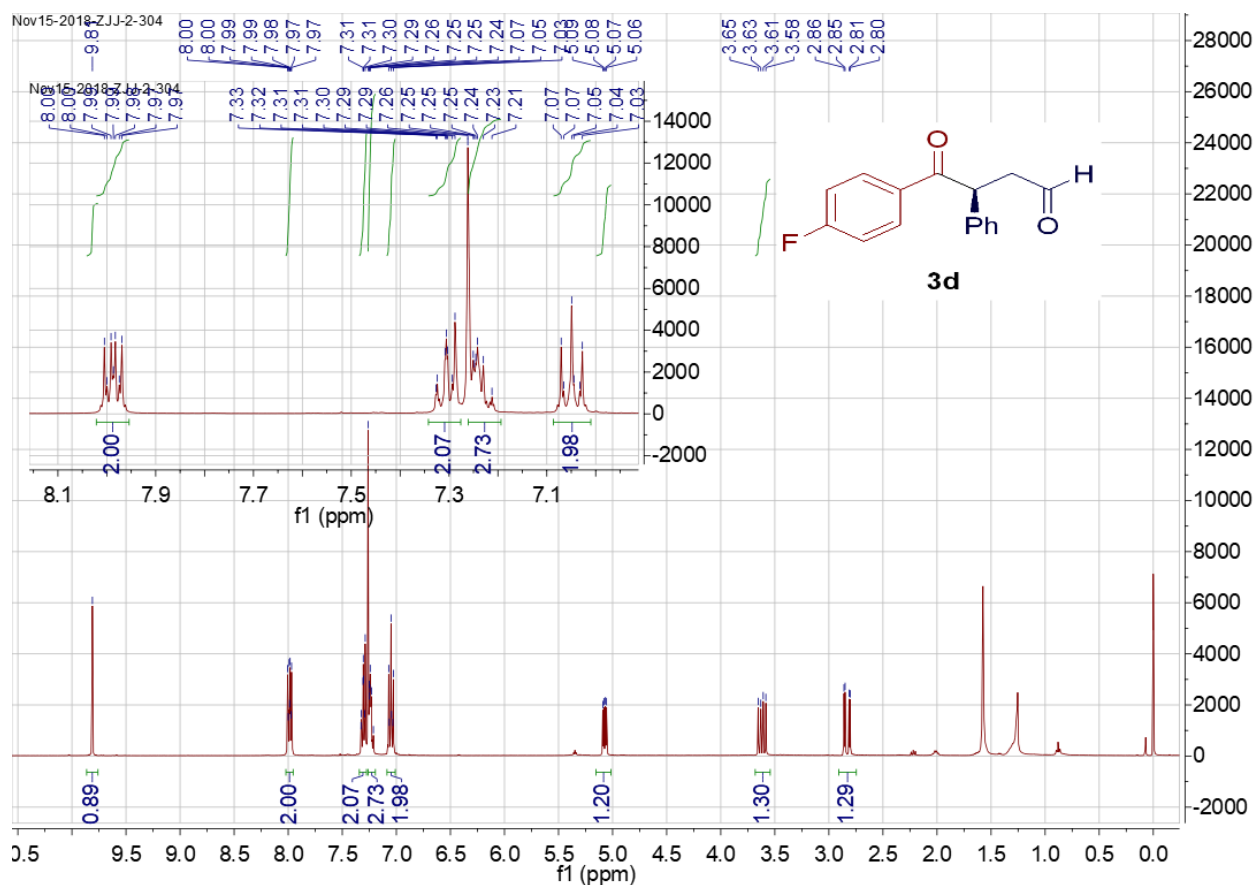


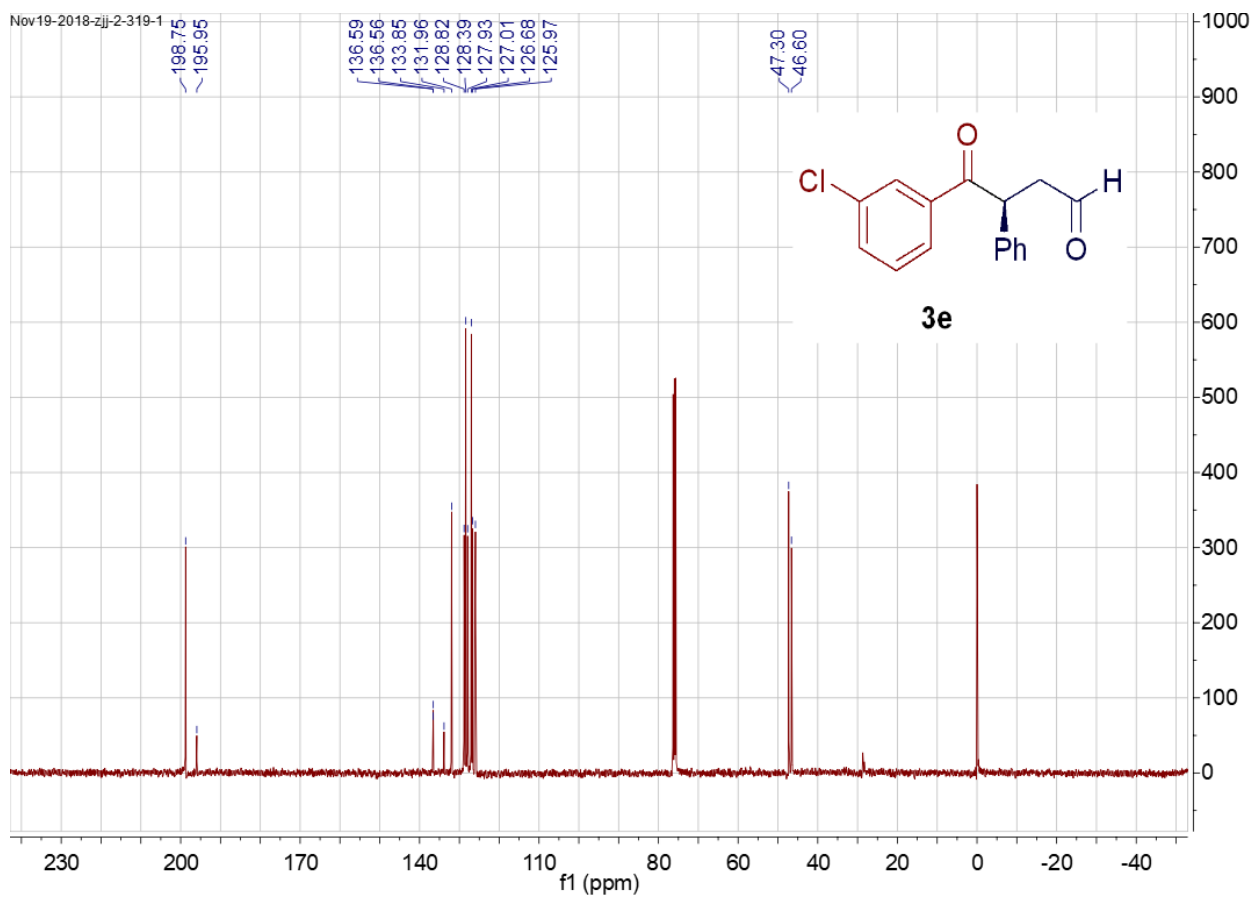
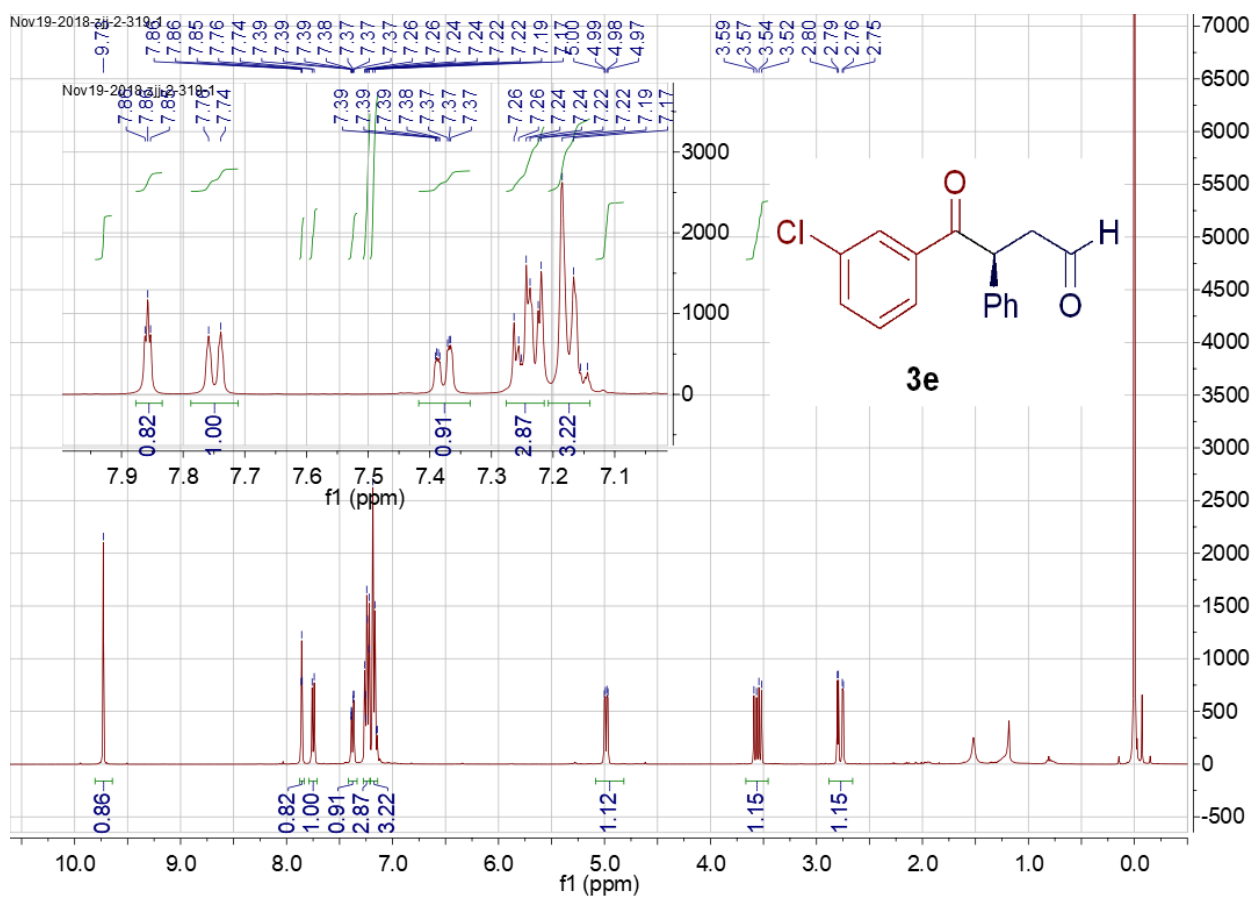
## 8. Copies of NMR spectra for products 3

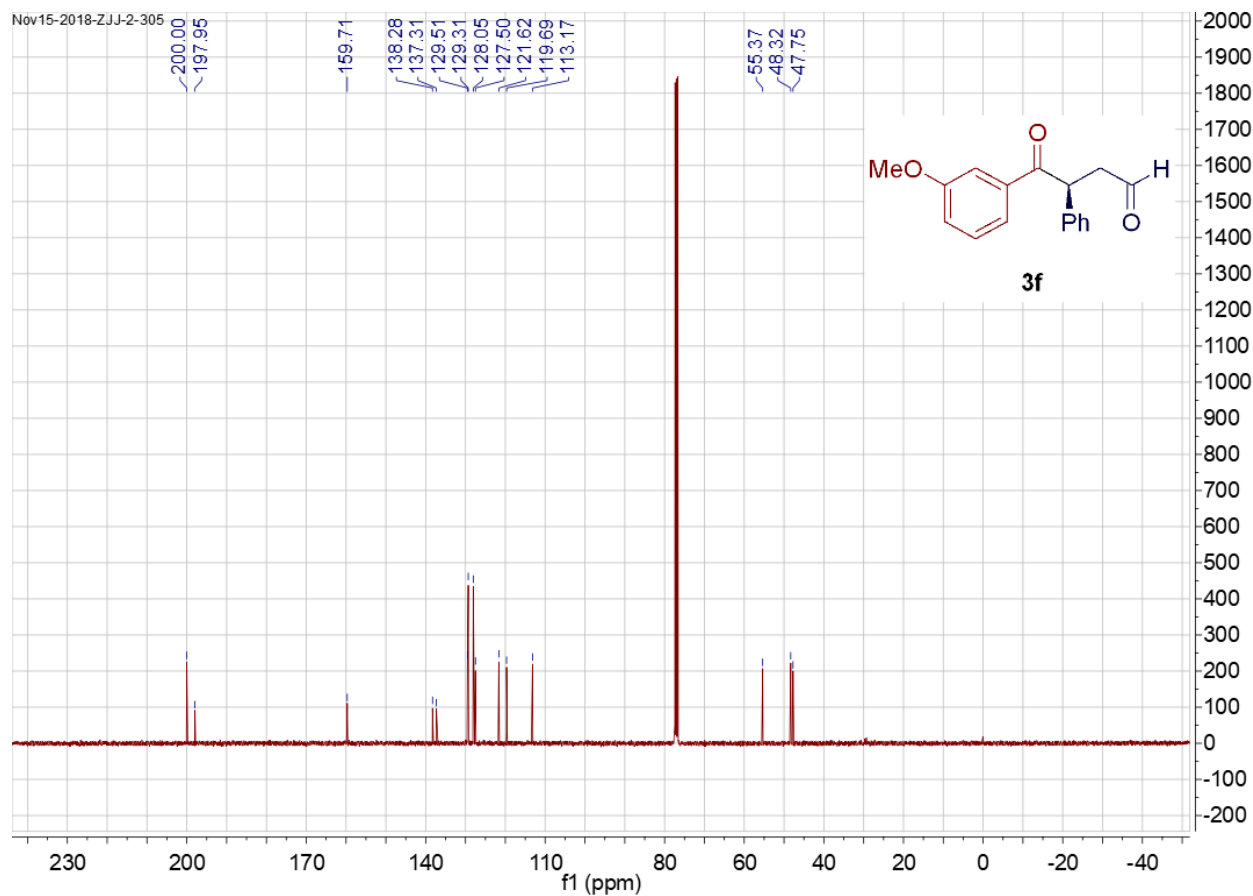
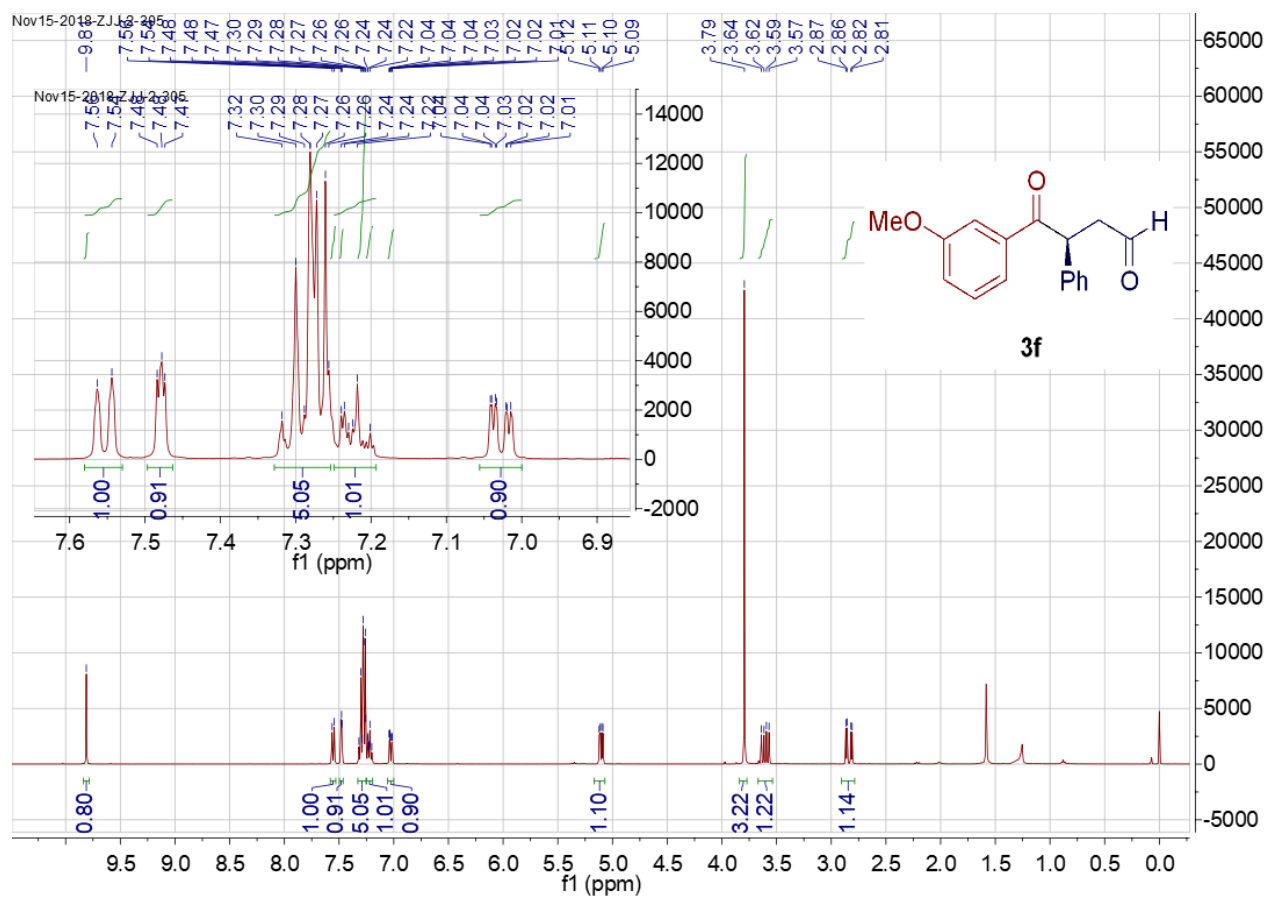




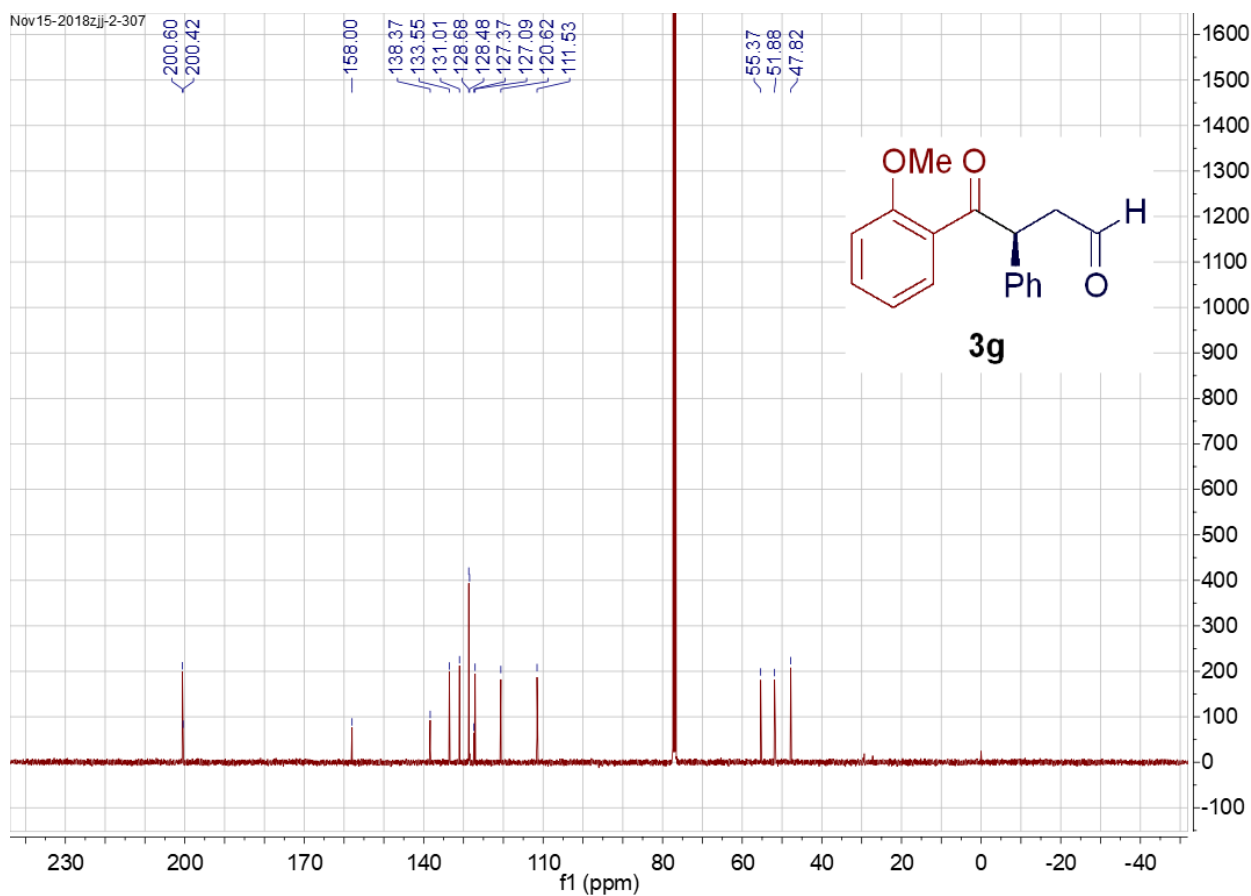
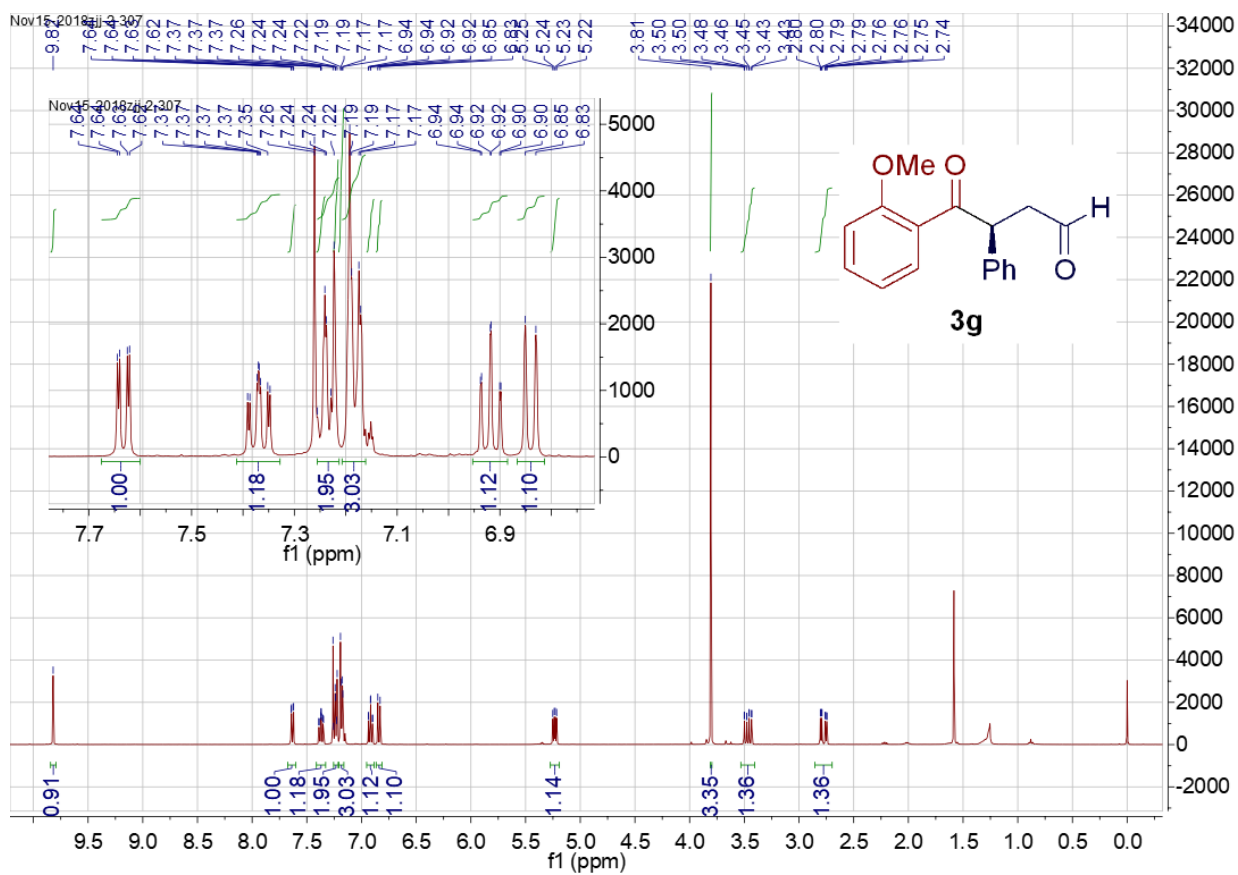


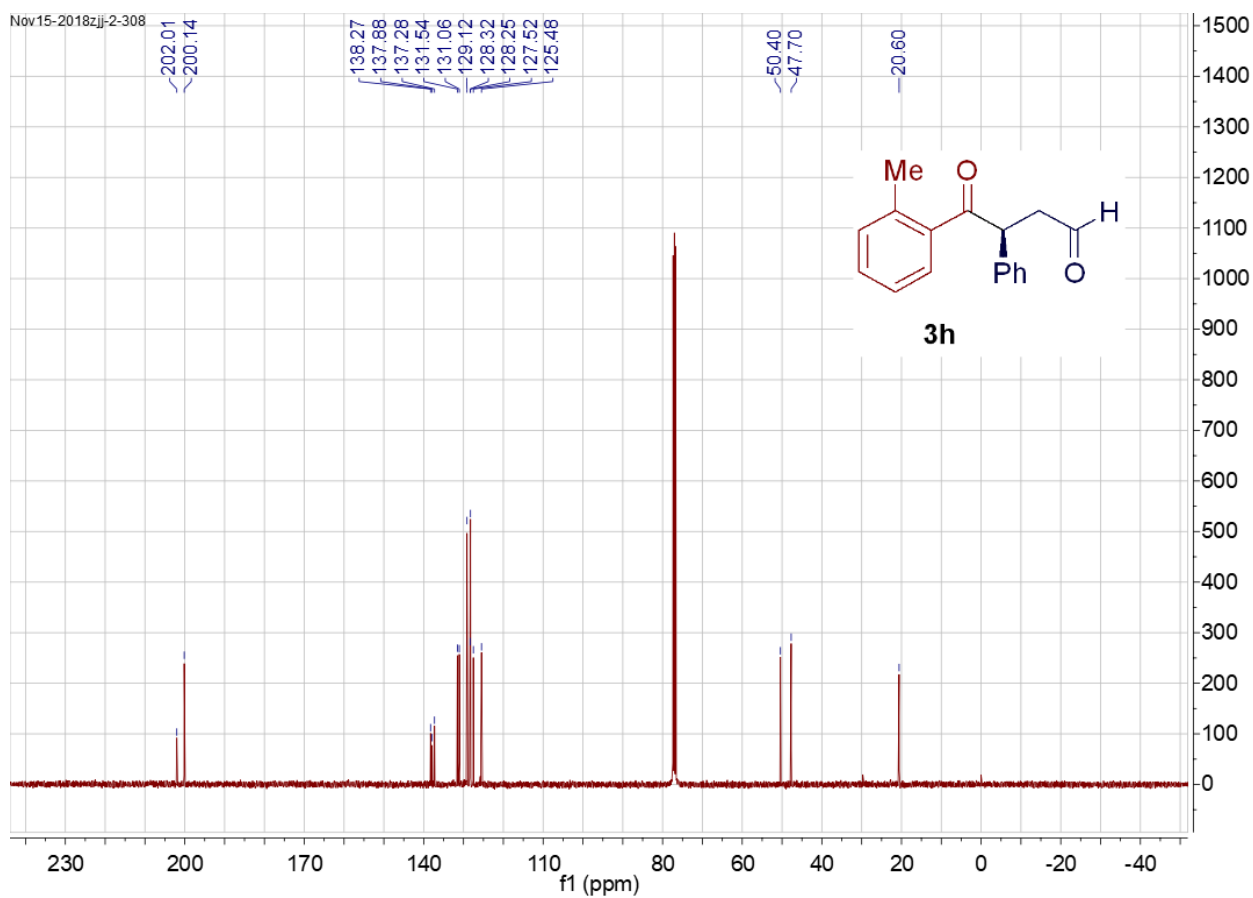
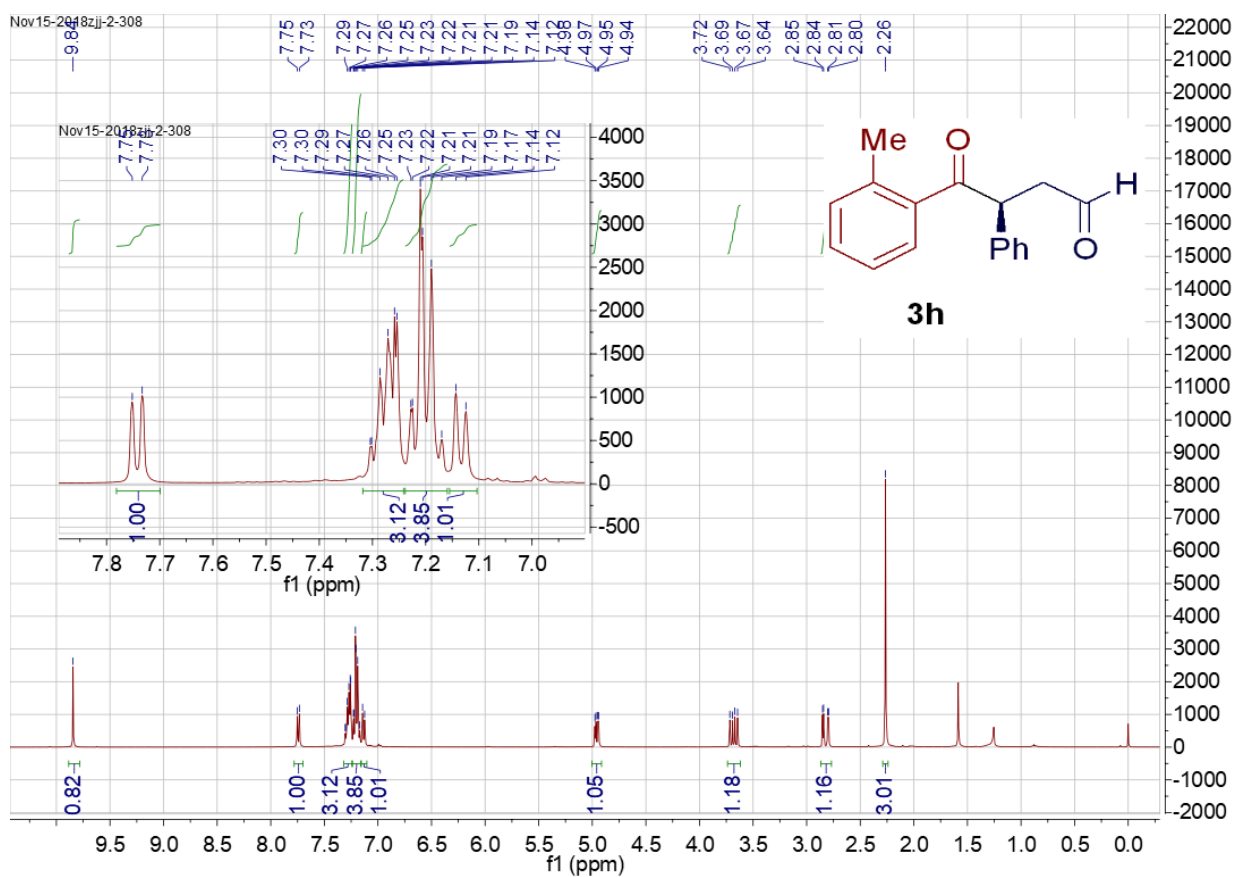


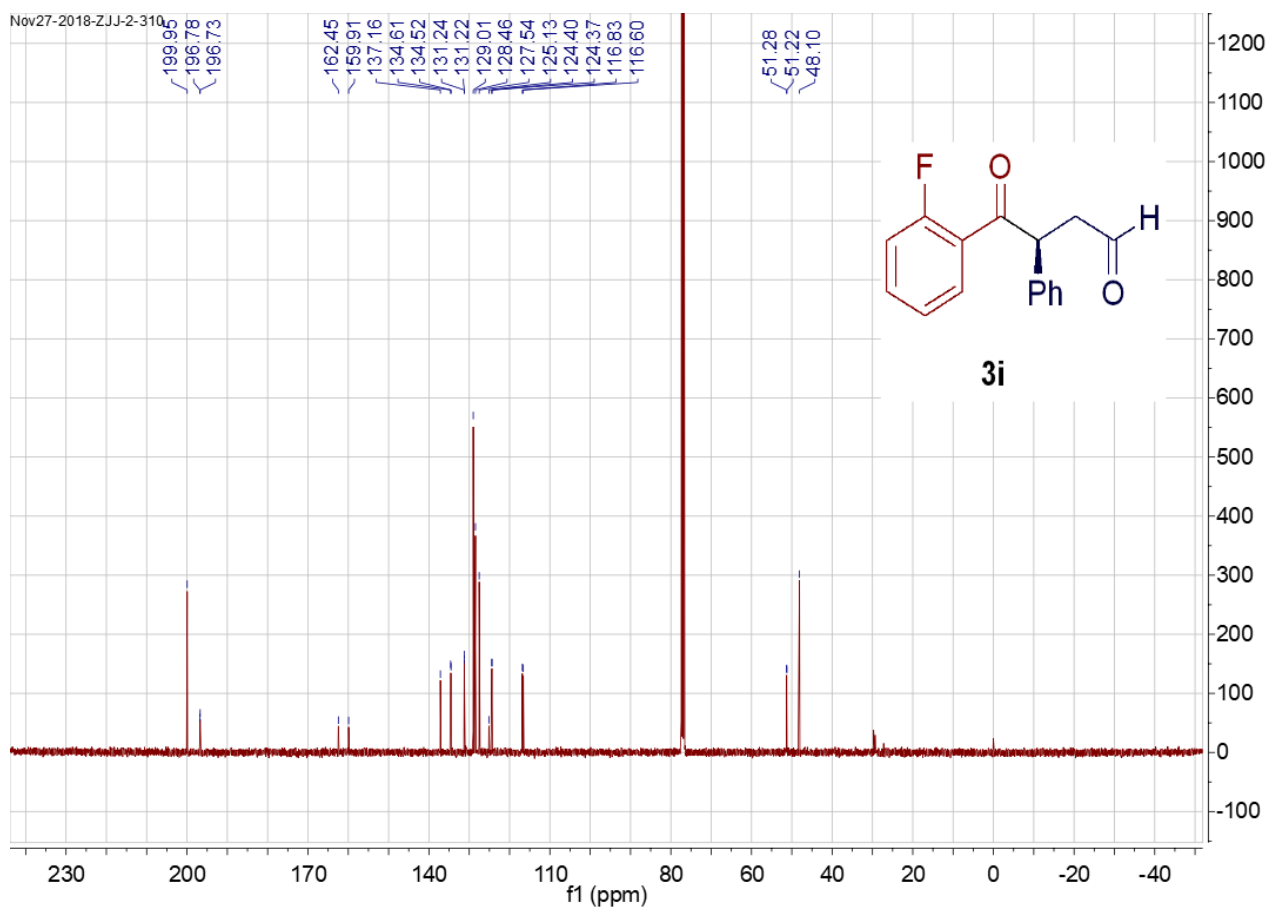
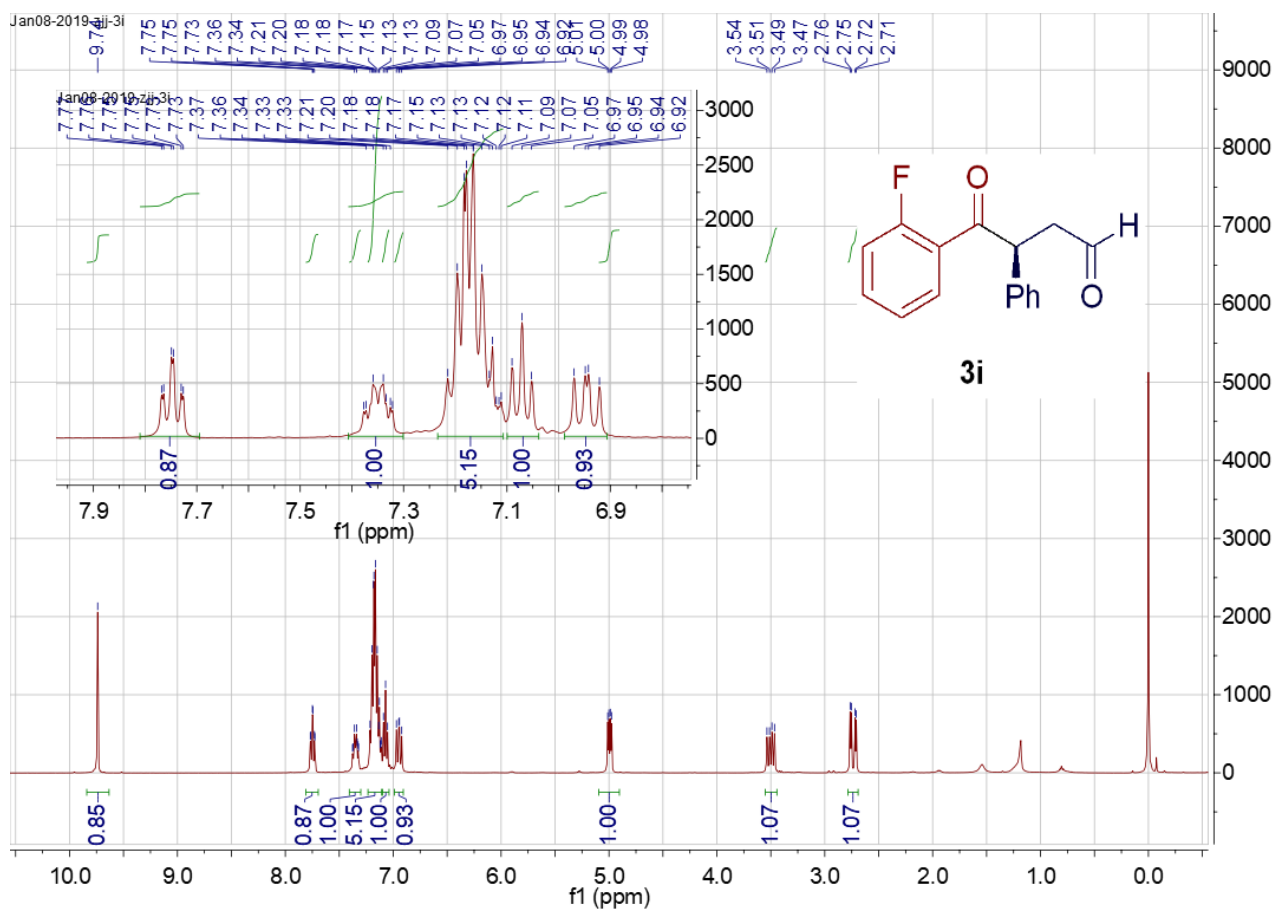


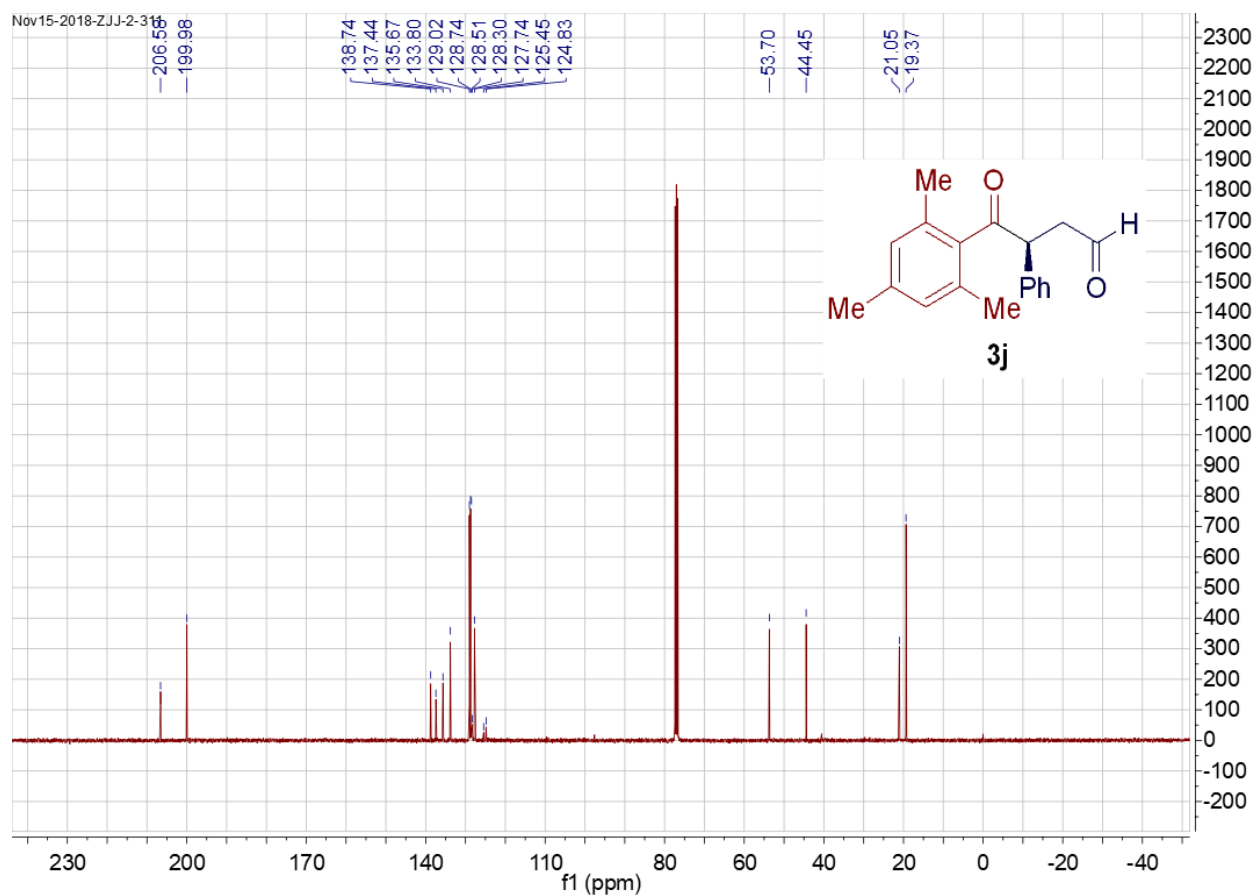
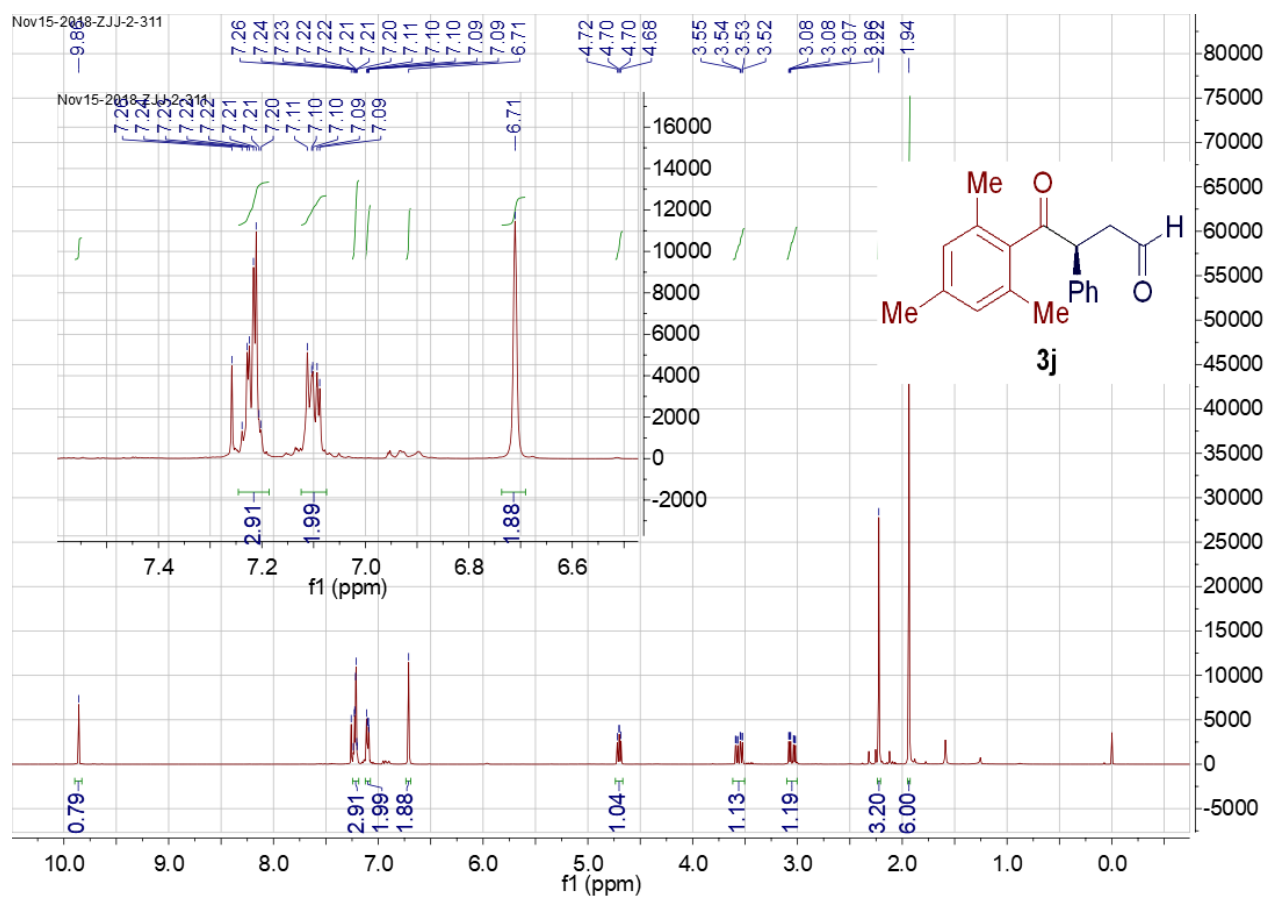


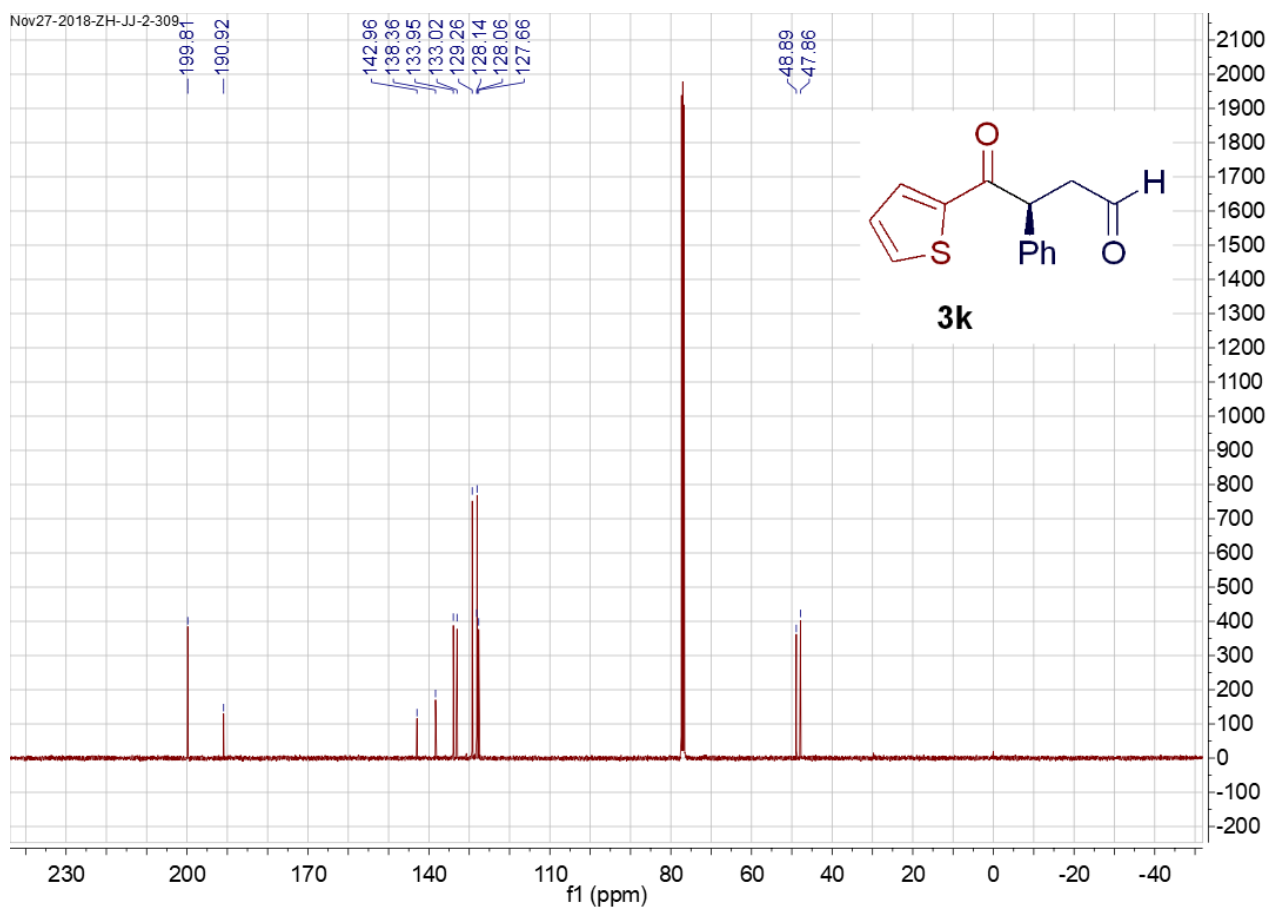
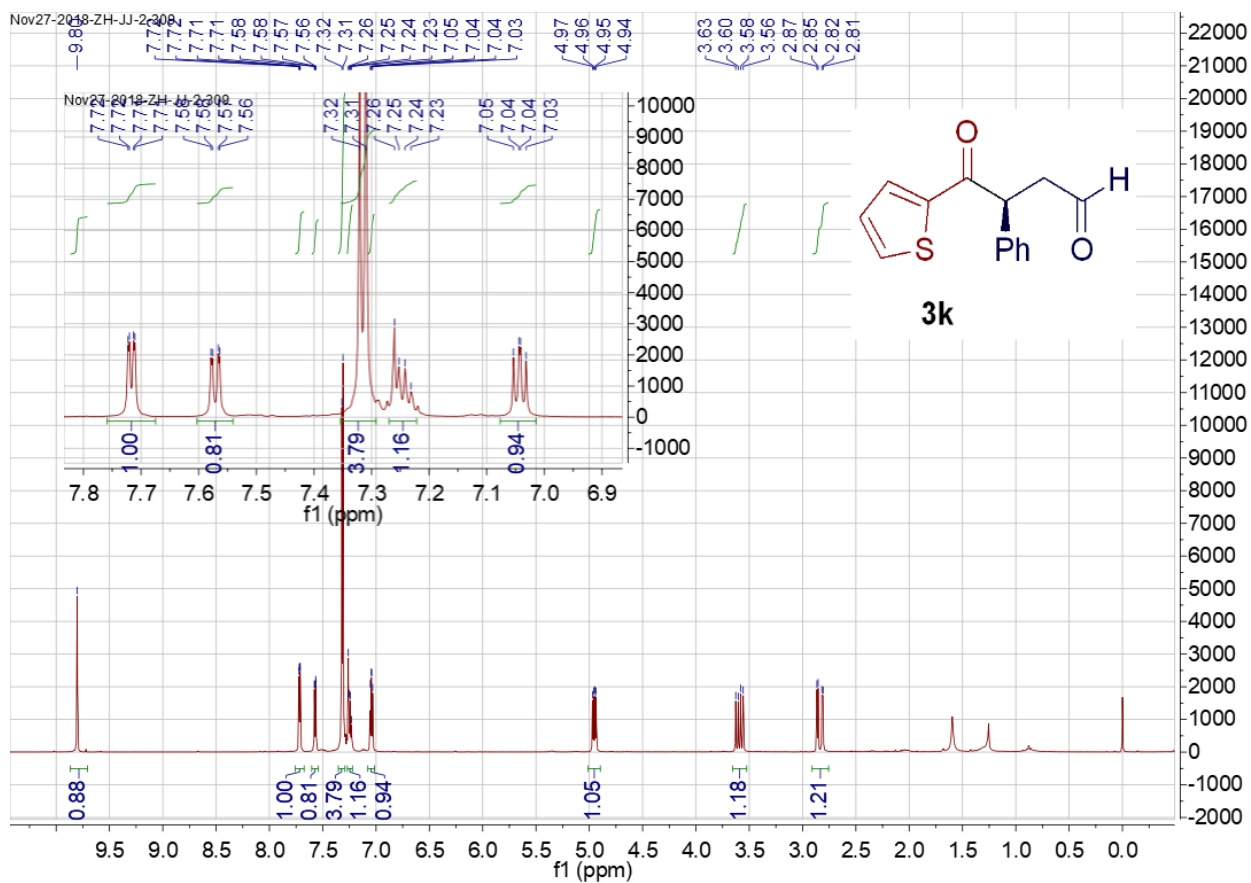


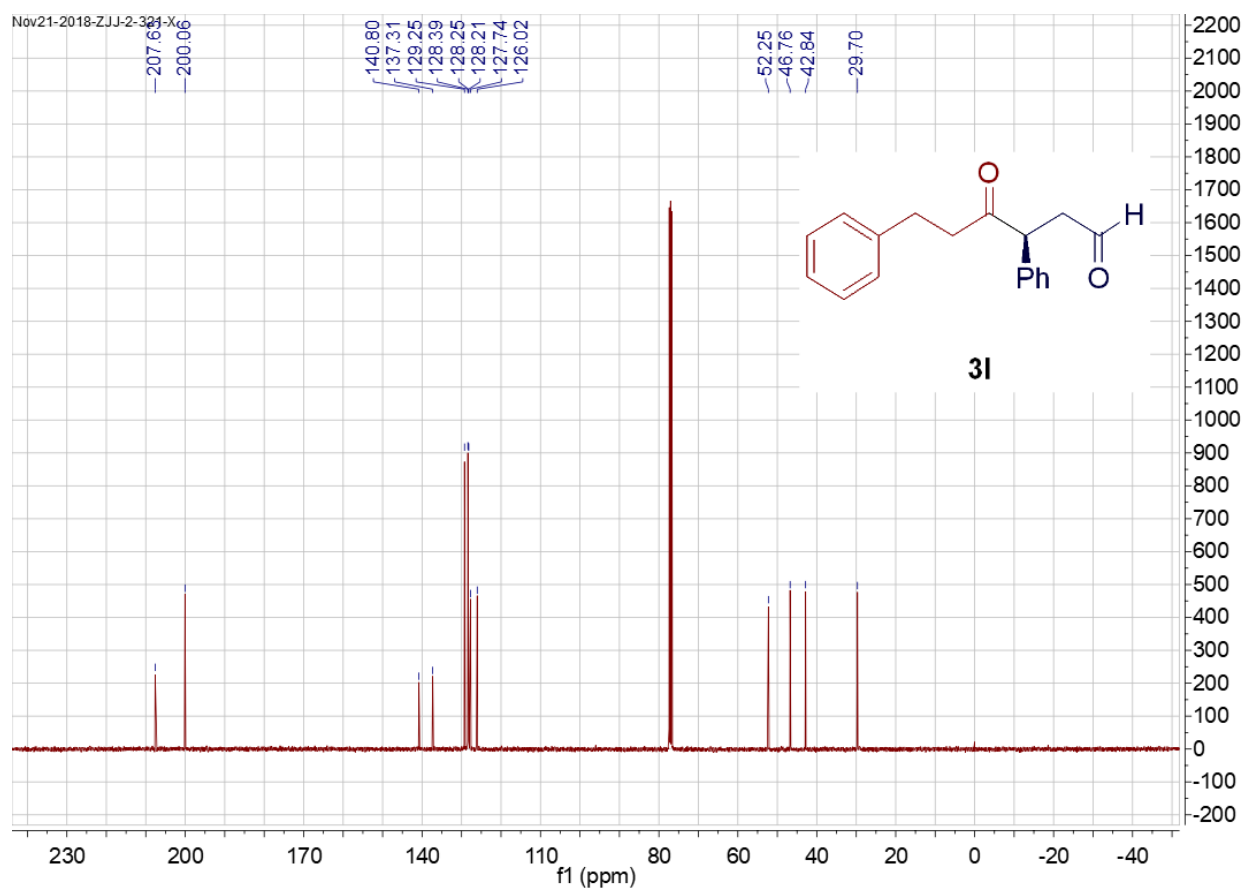
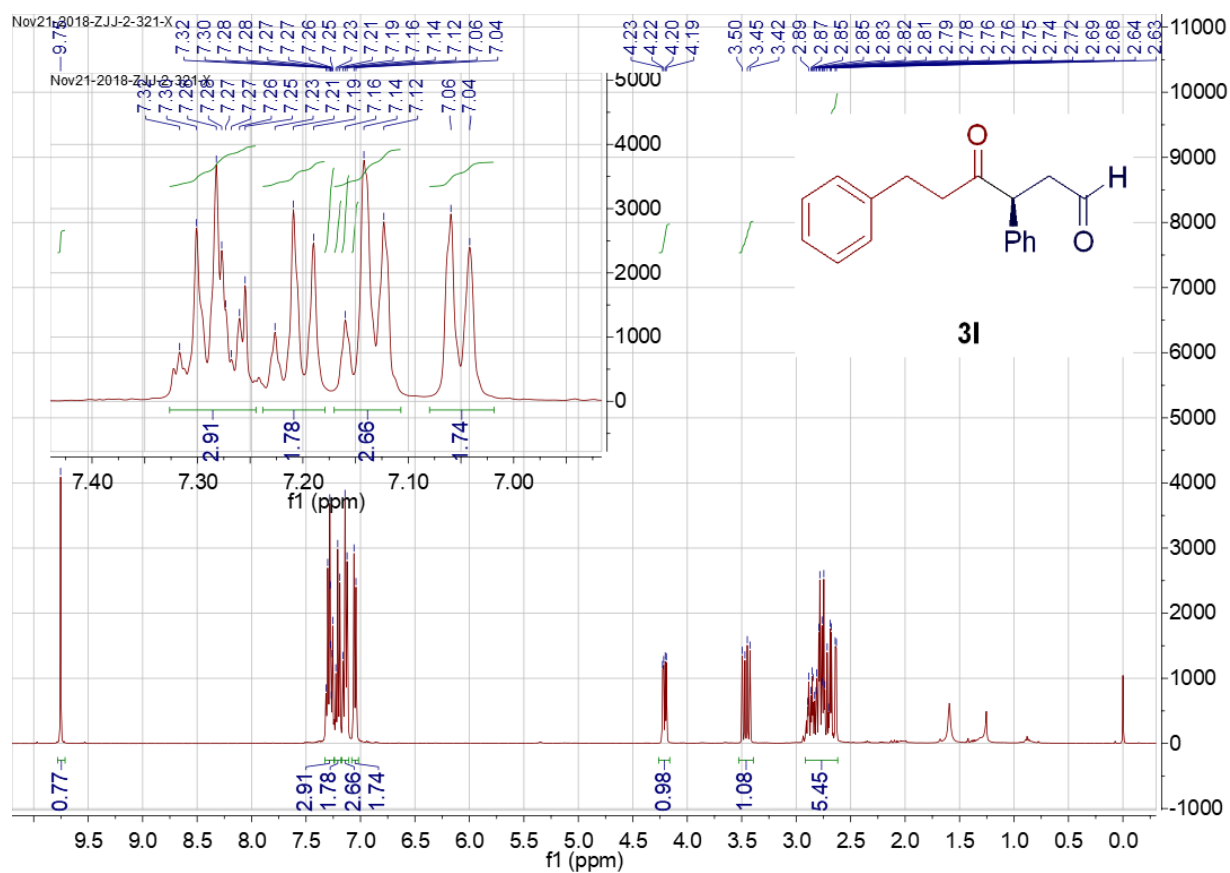


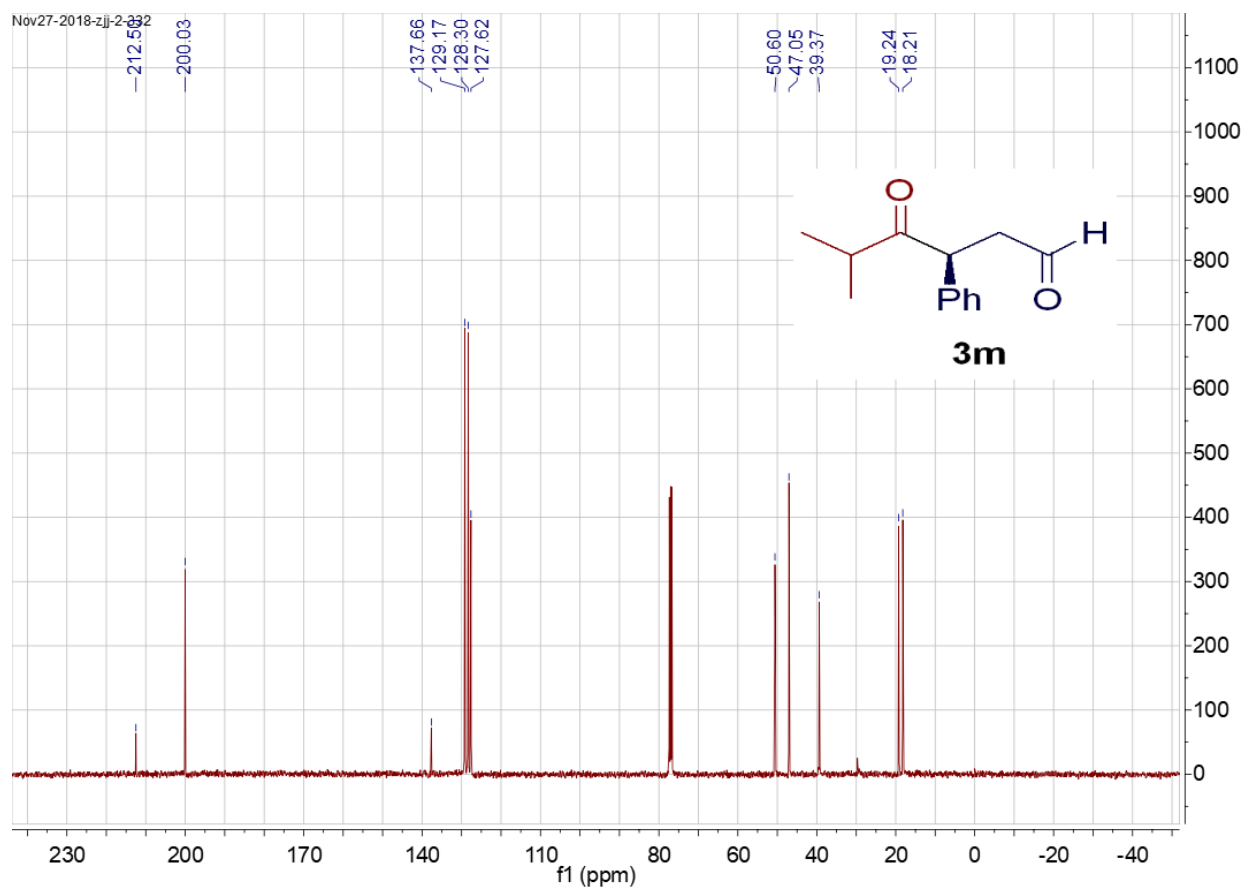
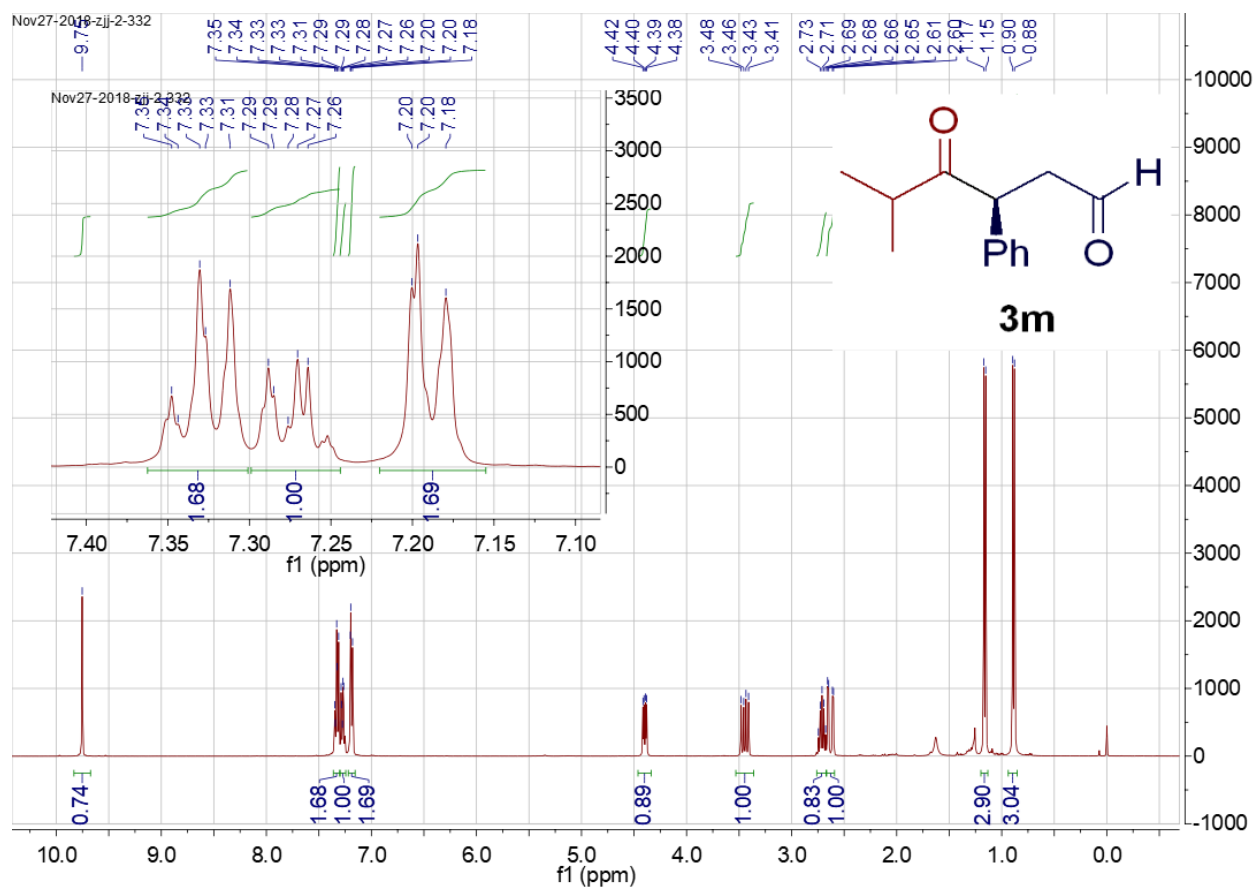


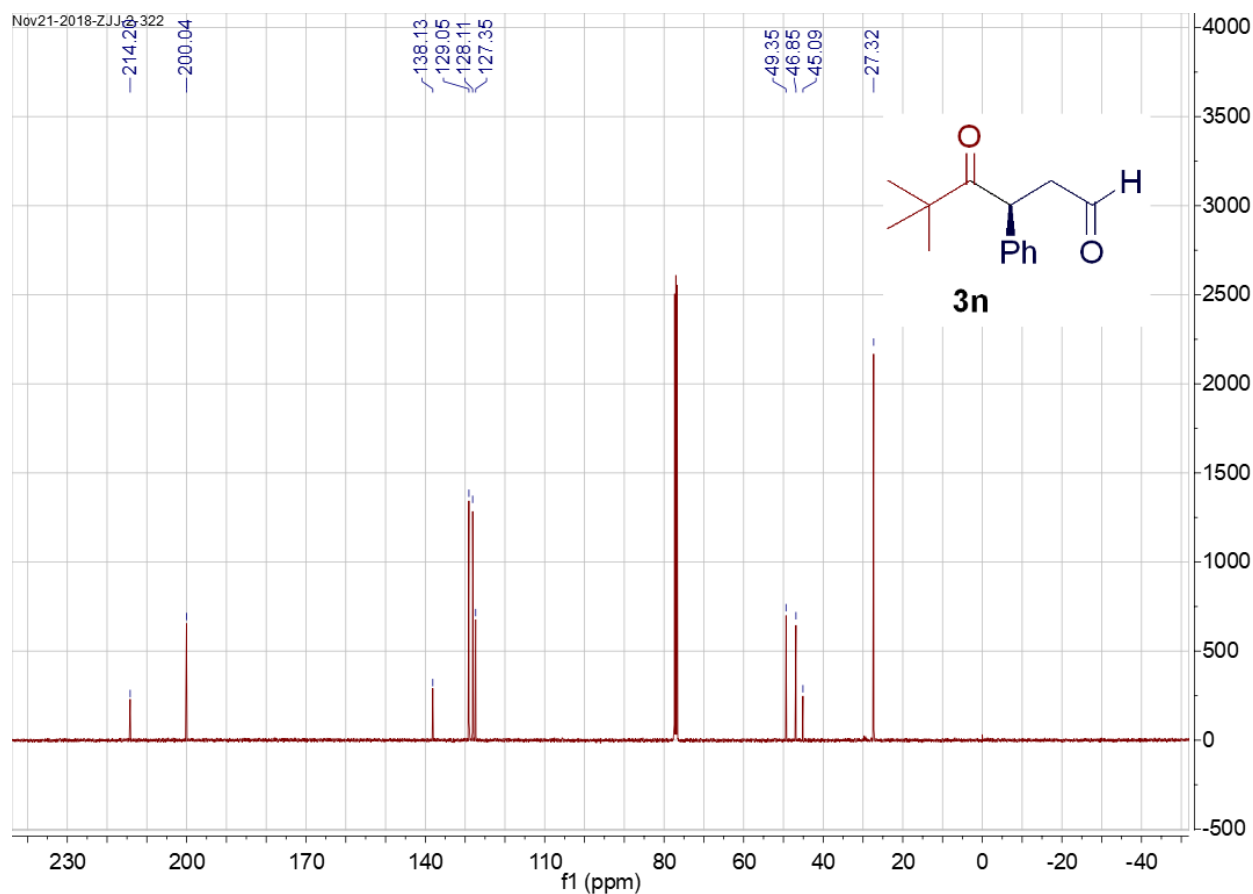
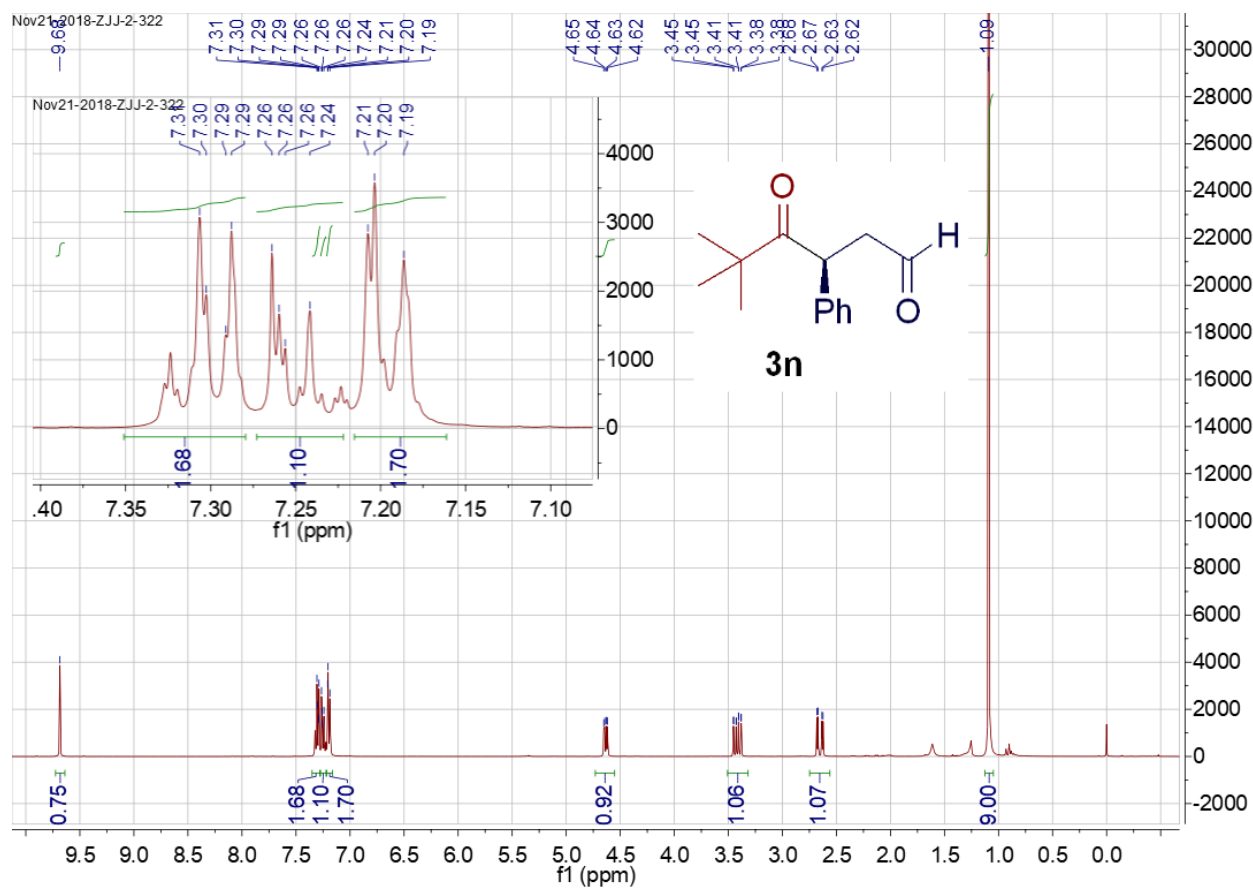




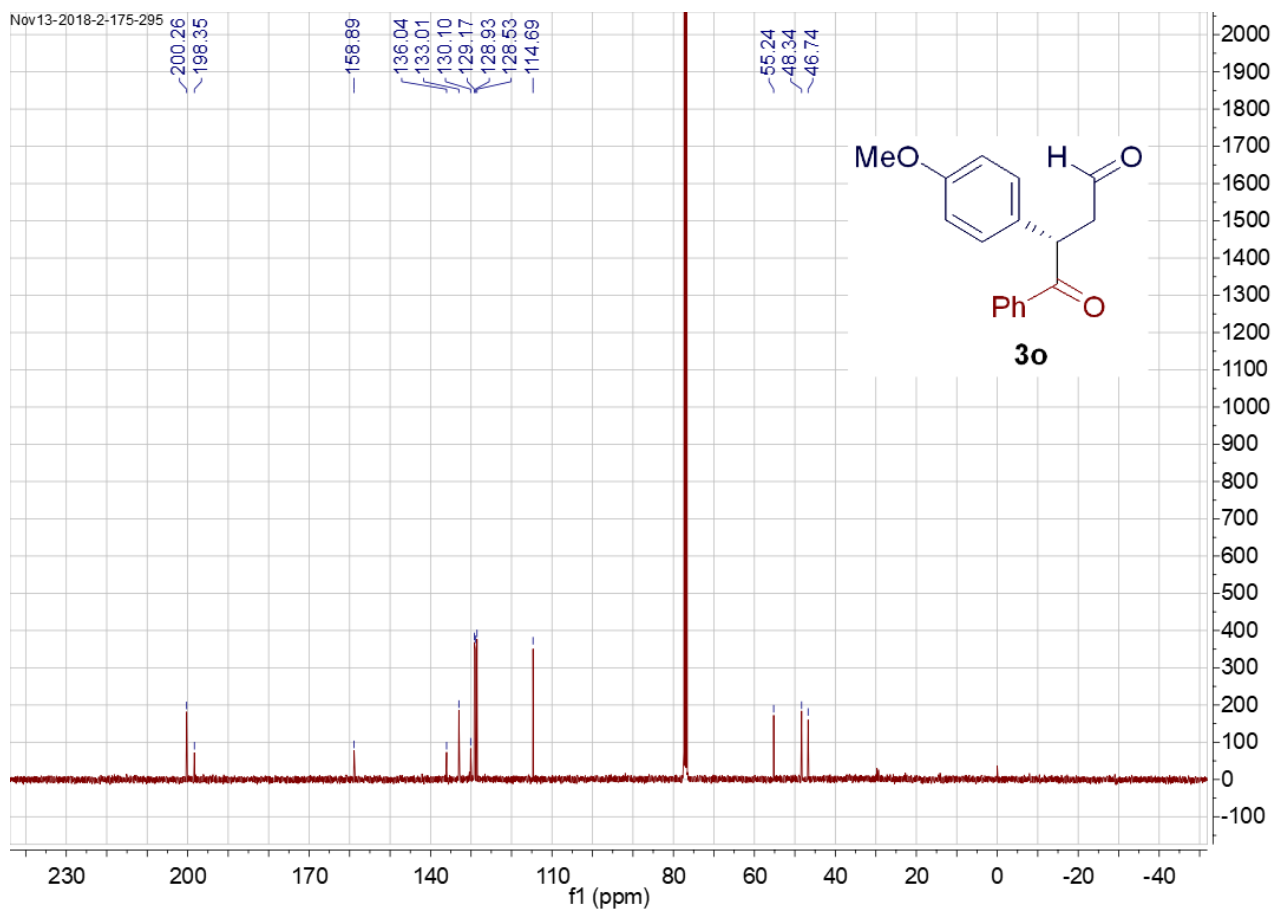
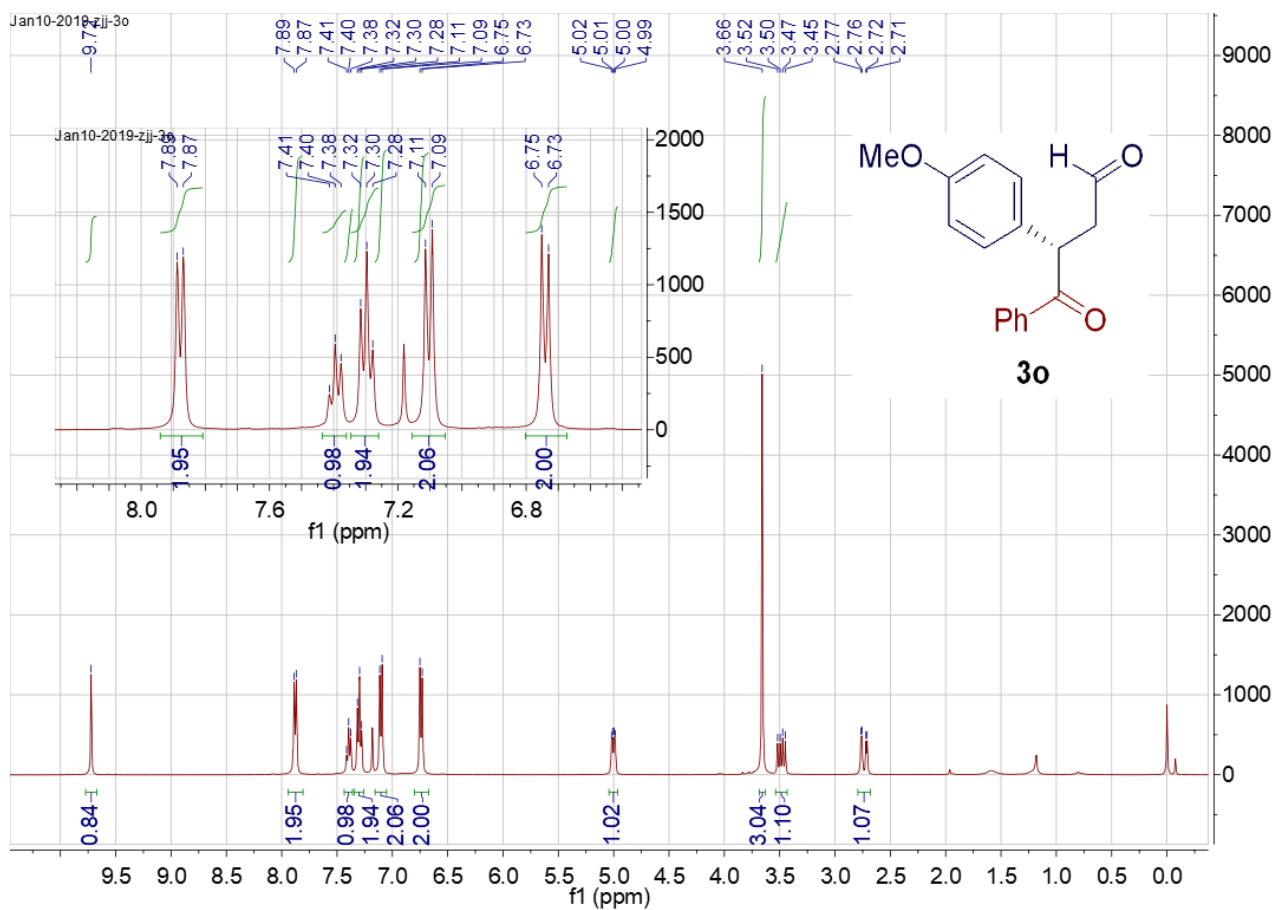


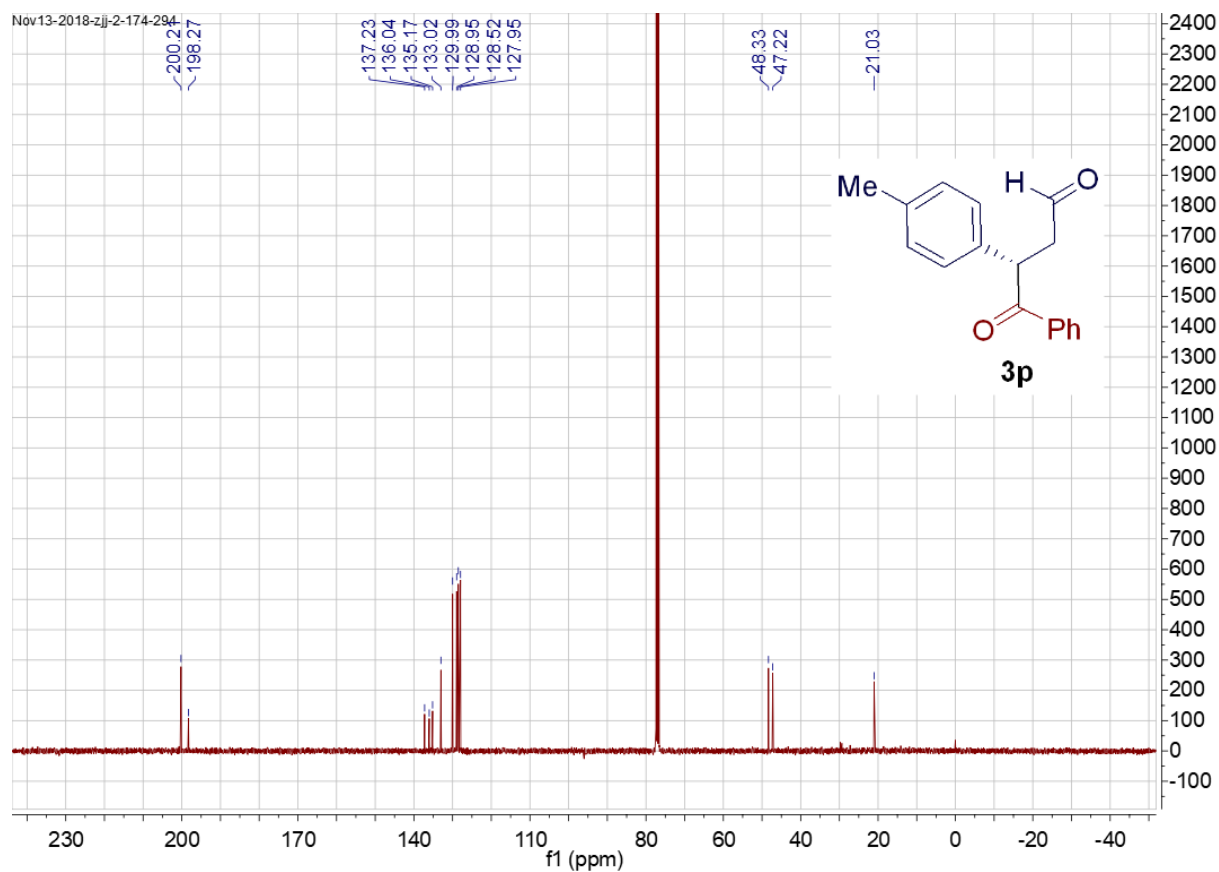
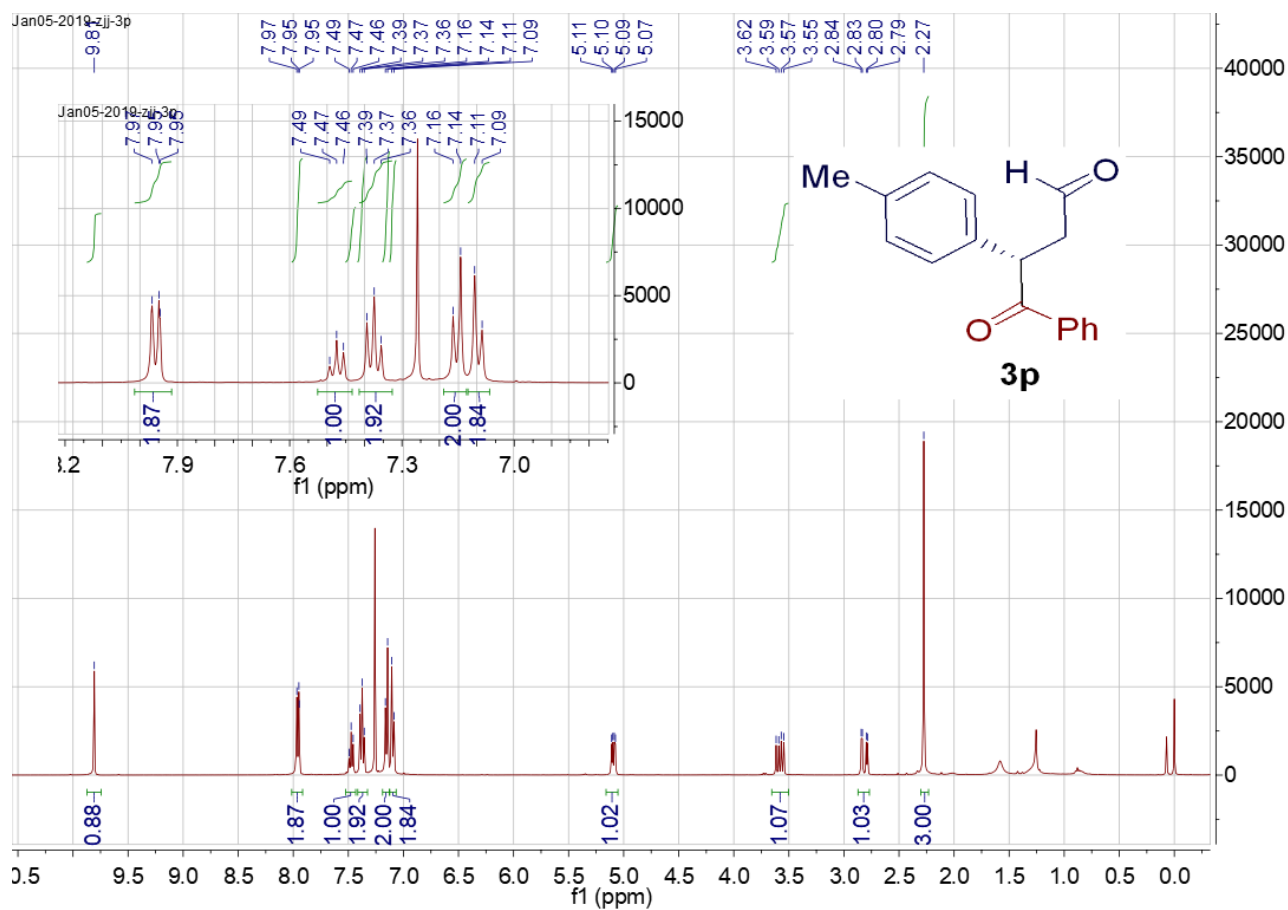


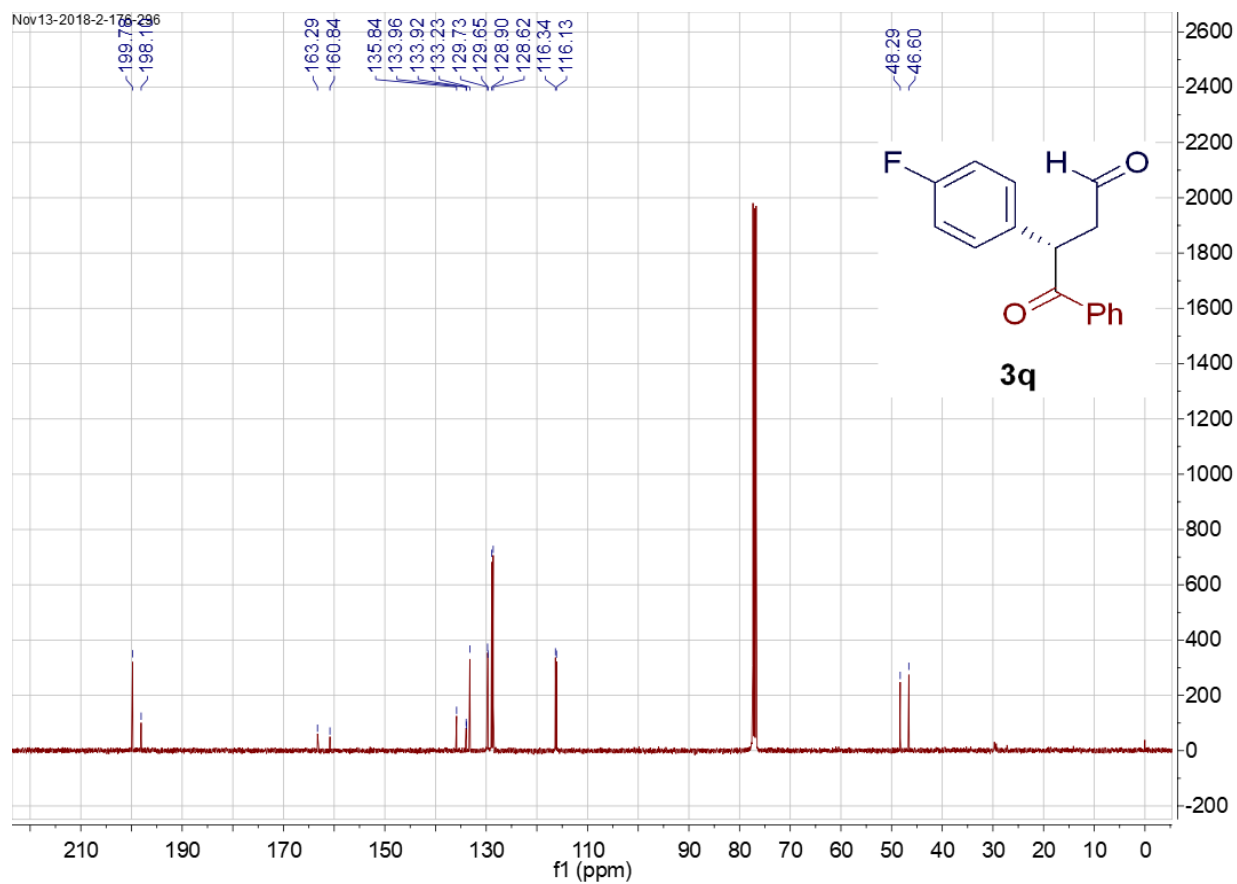
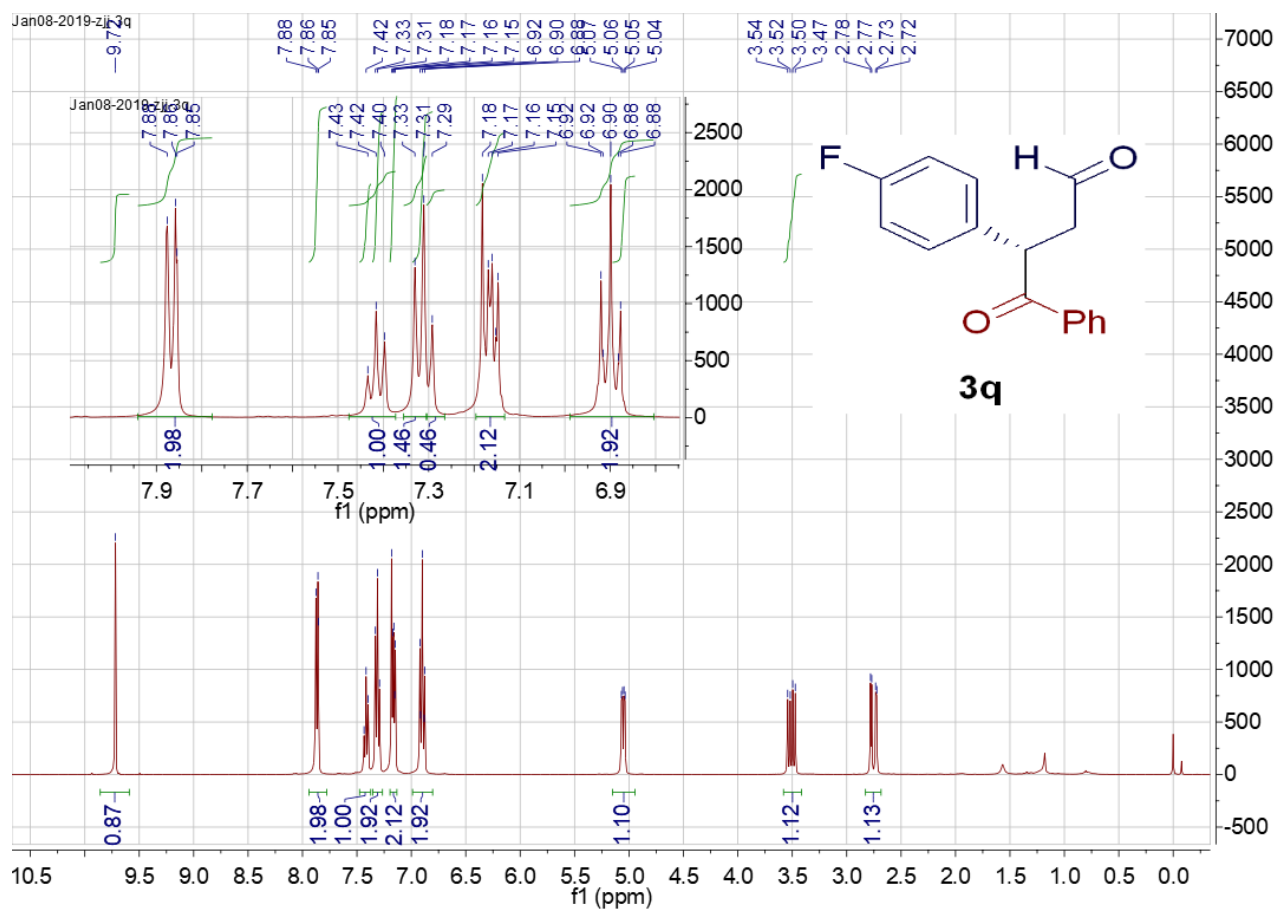


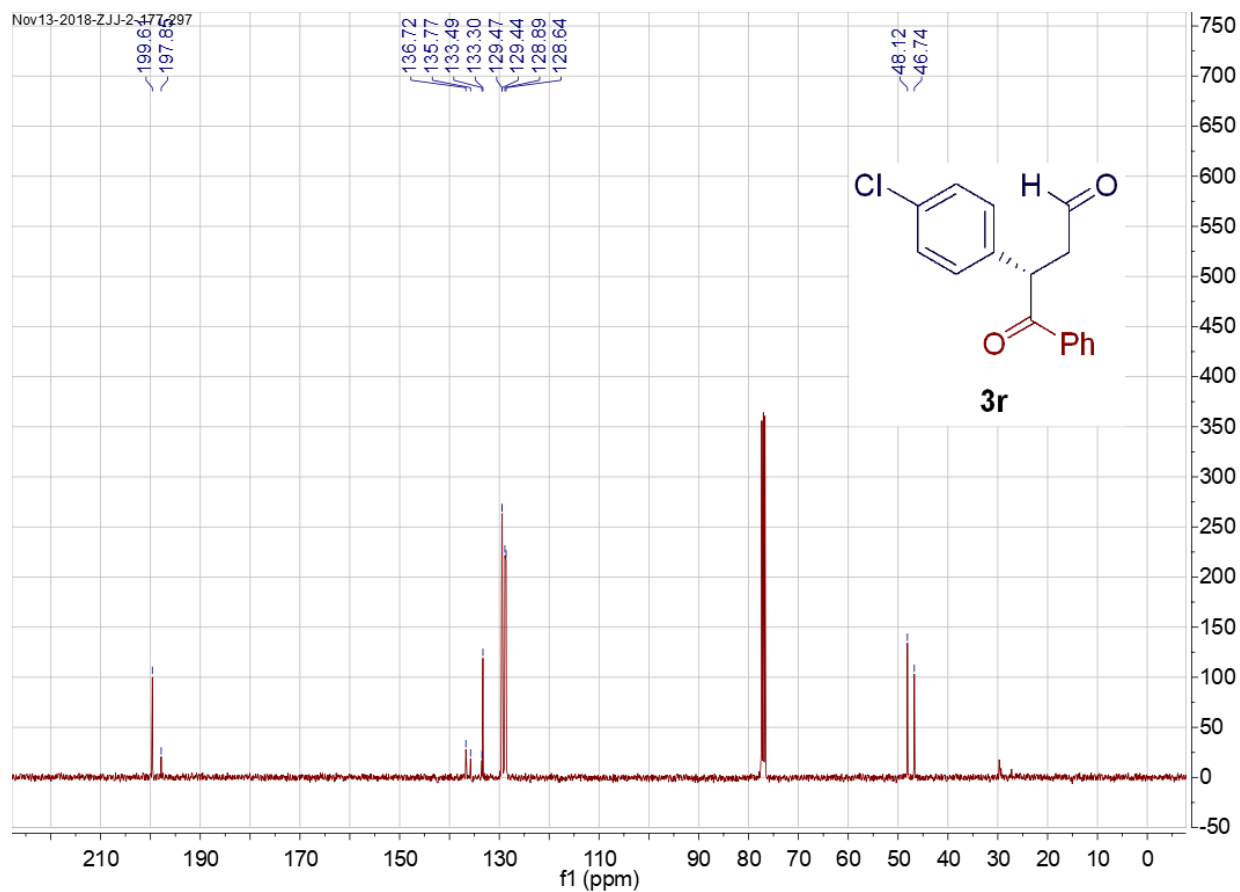
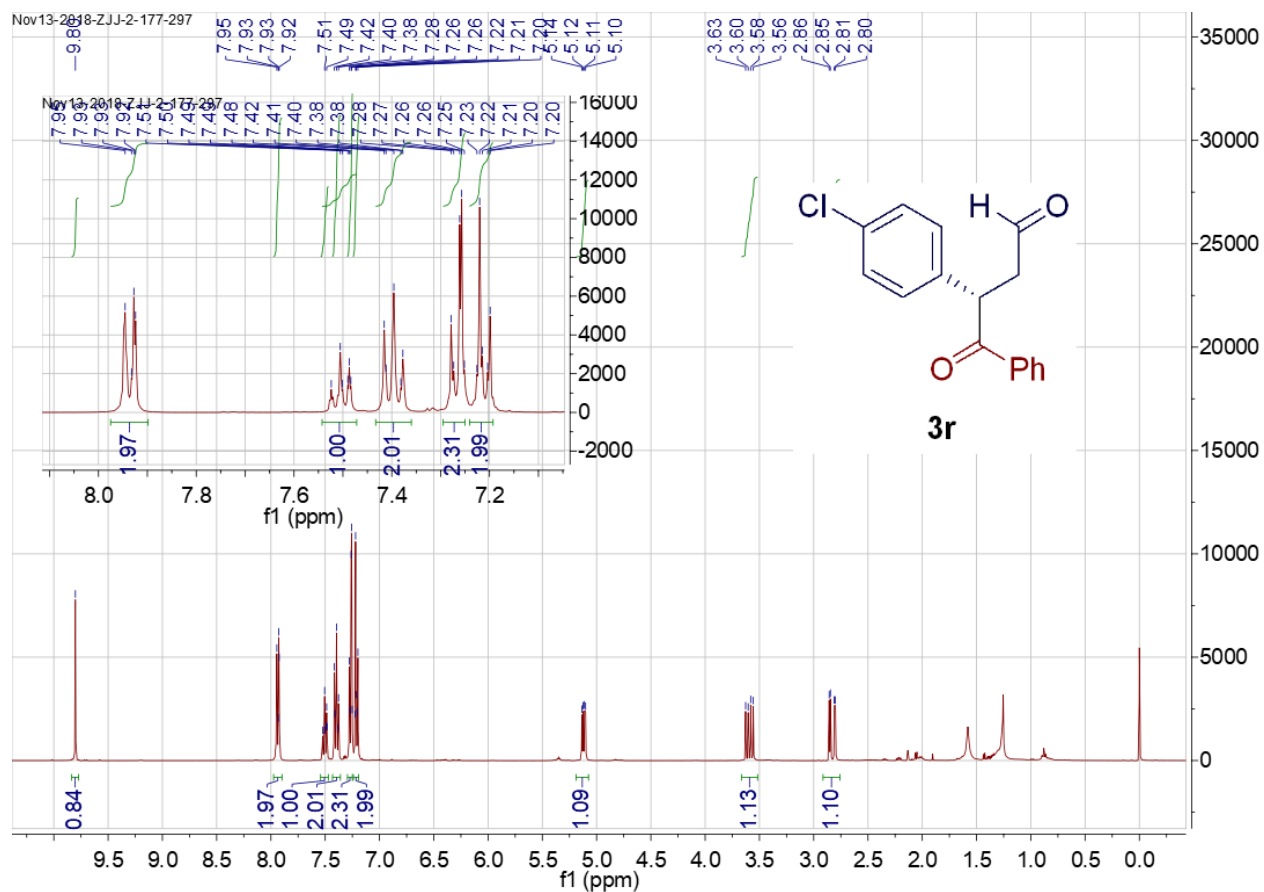


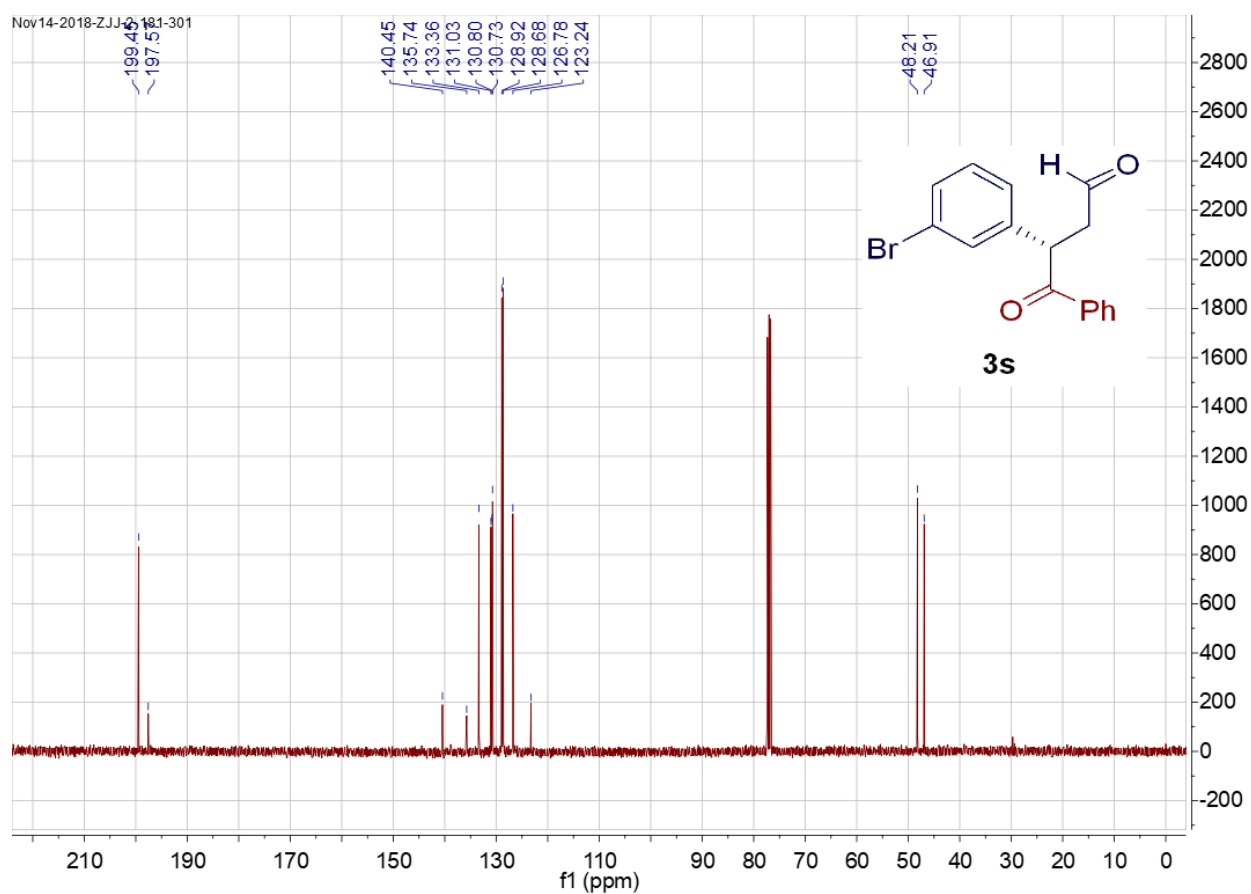
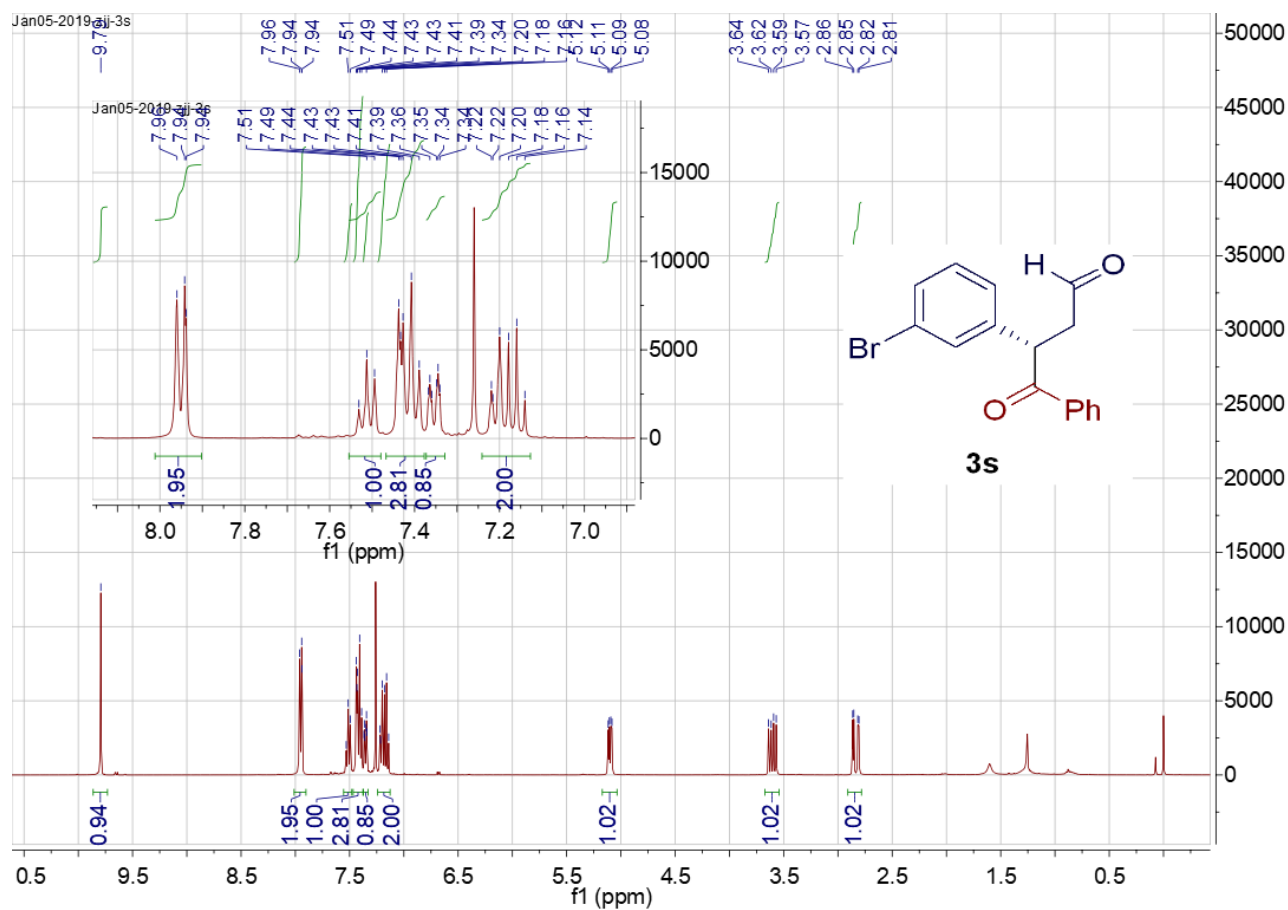


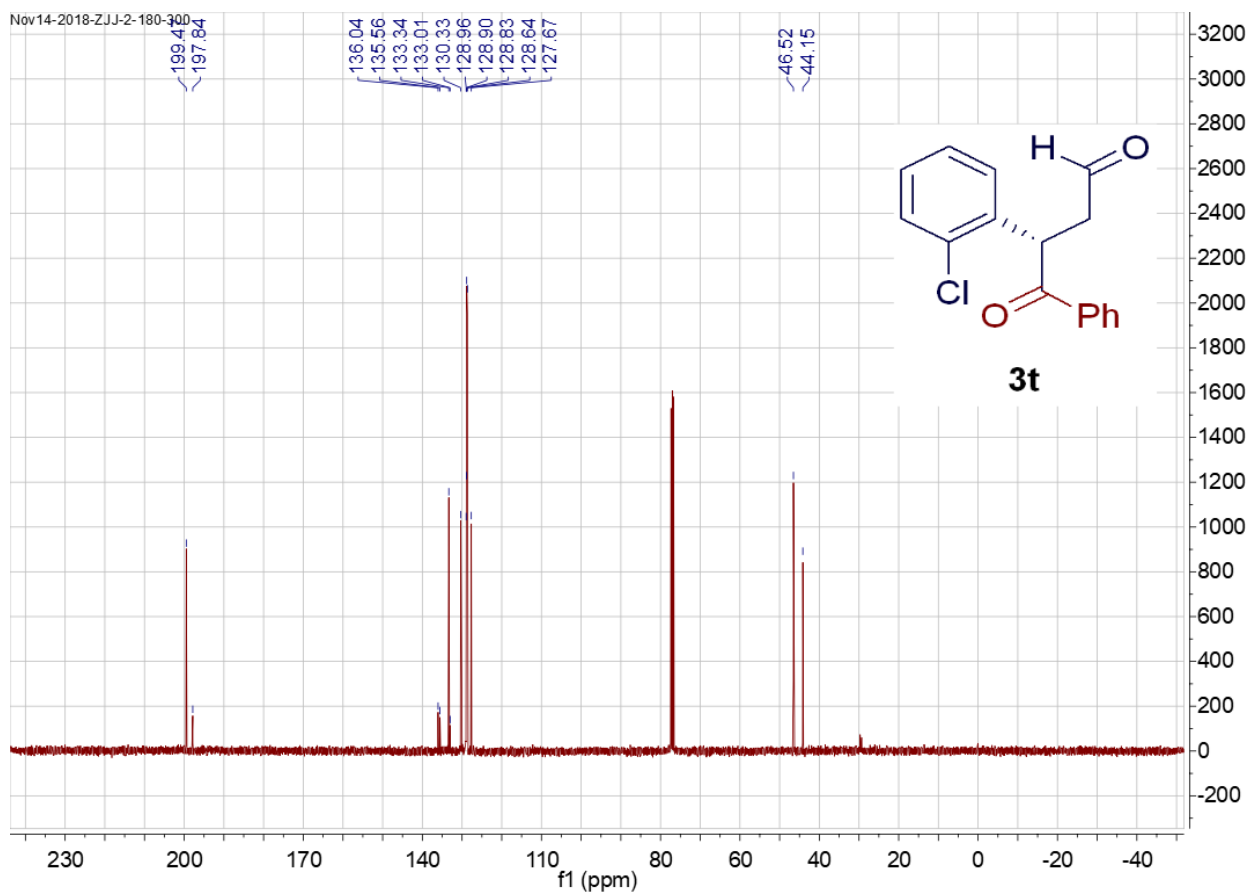
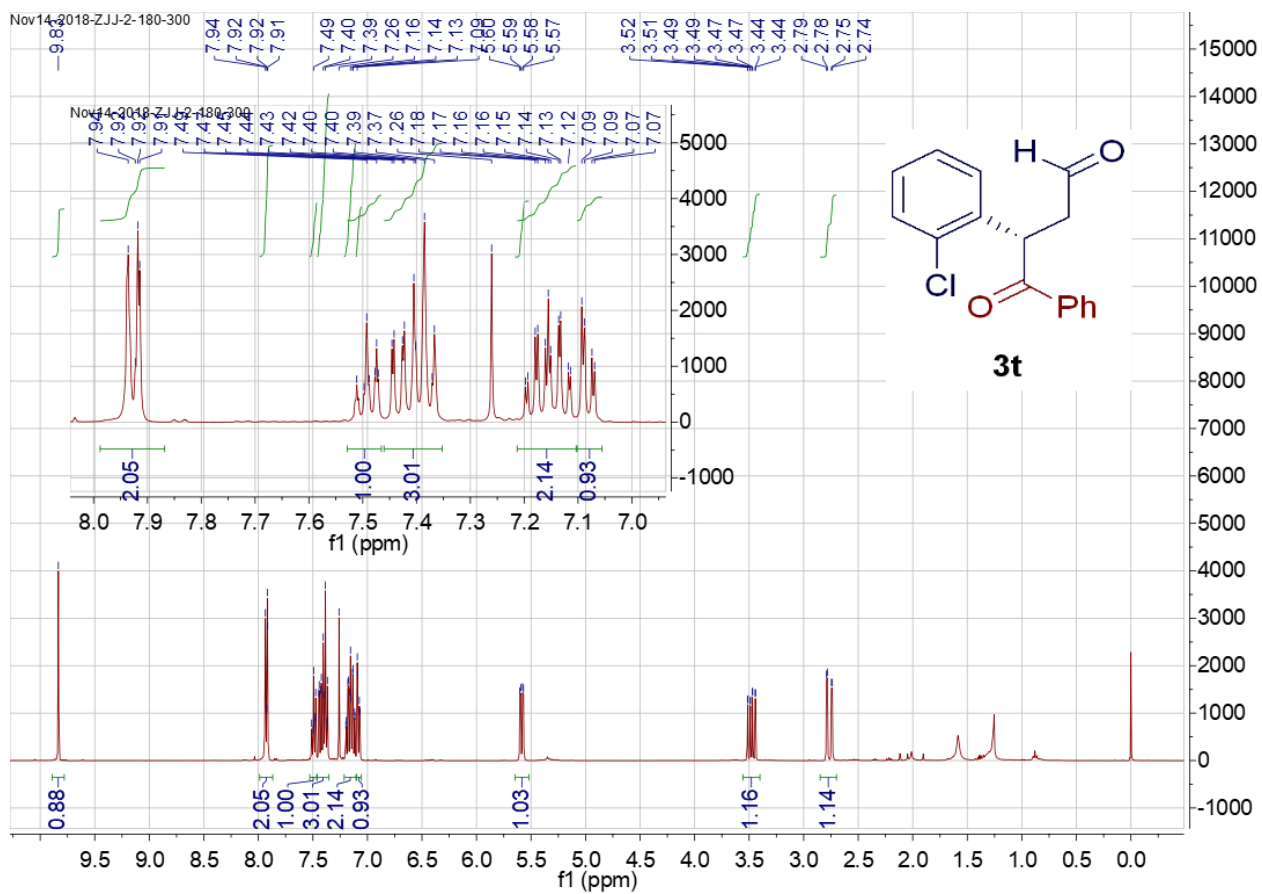


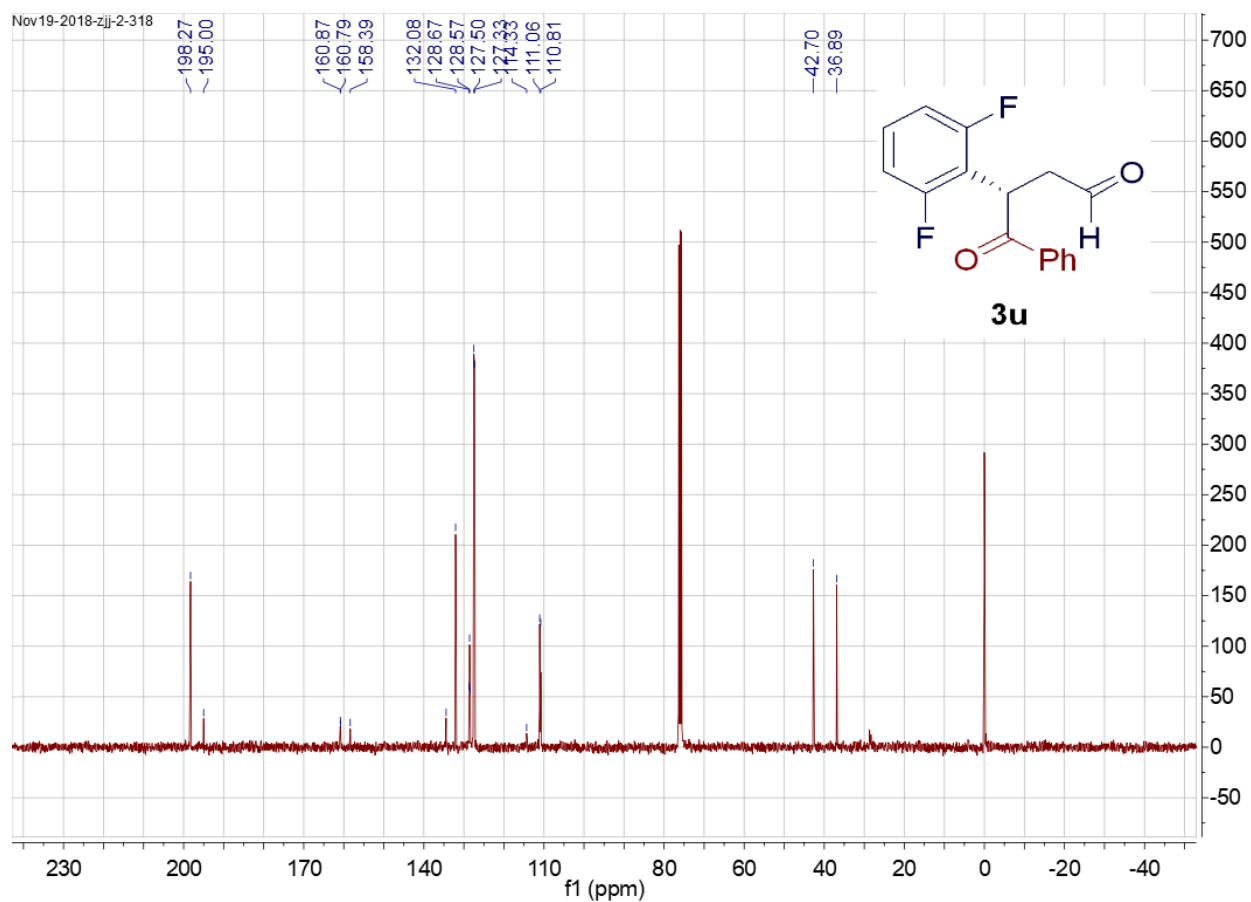
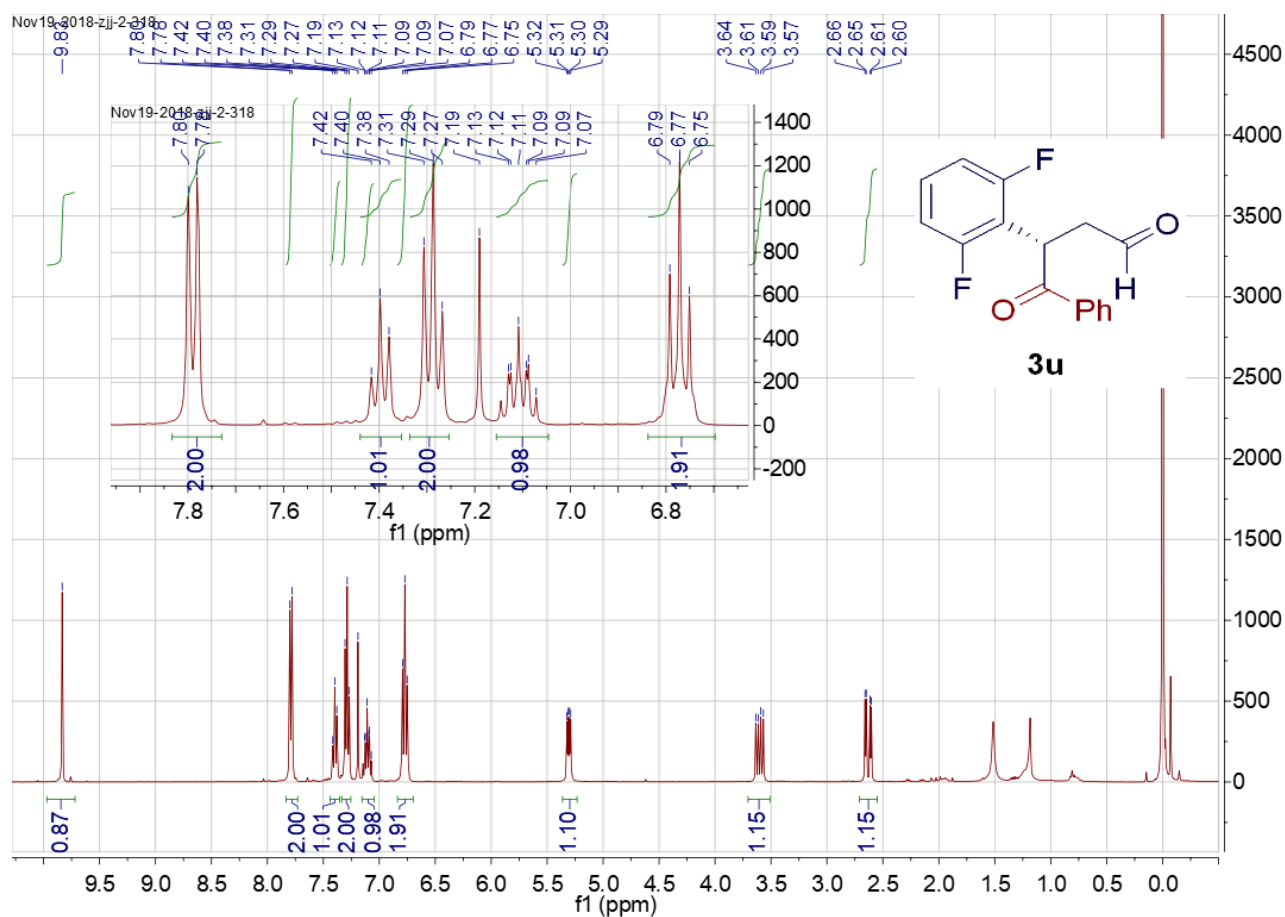




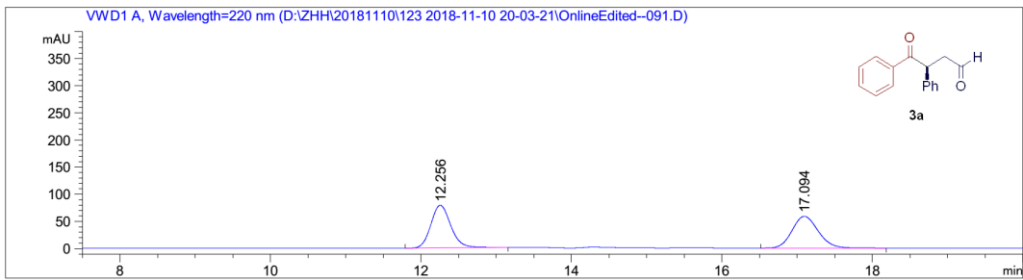








9. Chiral HPLC analyses of products 3



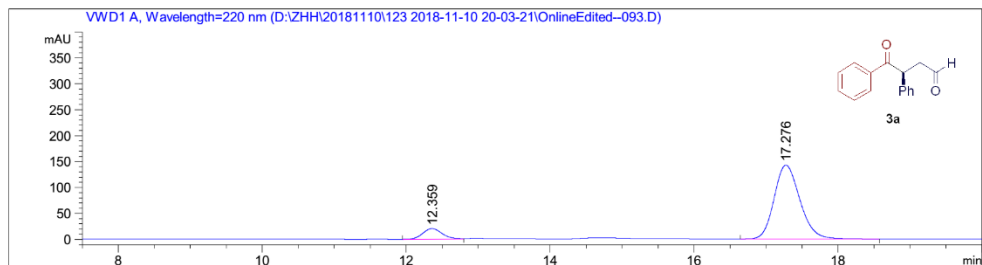
面积百分比报告

排序 : 信号  
乘积因子 : 1.0000  
稀释因子 : 1.0000  
内标中不使用乘积因子和稀释因子

Retention Time			Area		Height	Relative Area
信号 1: VWD1 A, Wavelength=220 nm						
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	12.256	BB	0.2844	1462.15808	78.84209	50.6316
2	17.094	BB	0.3739	1425.68030	58.58331	49.3684

总量 : 2887.83838 137.42540

\*\*\* 报告结束 \*\*\*



面积百分比报告

排序 : 信号  
乘积因子 : 1.0000  
稀释因子 : 1.0000  
内标中不使用乘积因子和稀释因子

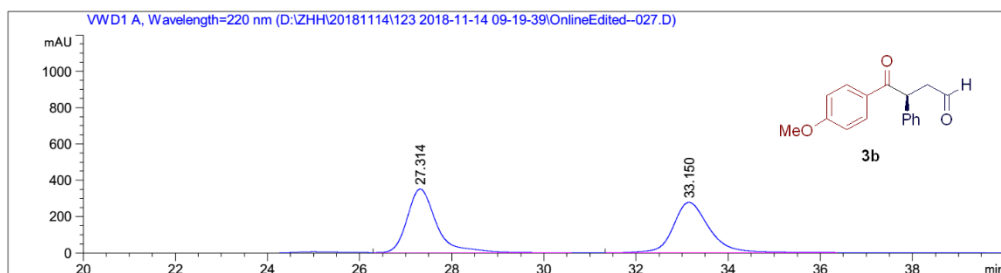
信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	12.359	BV	0.2937	392.45886	20.65601	9.9715
2	17.276	BB	0.3864	3543.35962	142.37236	90.0285

总量 : 3935.81848 163.02837

\*\*\* 报告结束 \*\*\*





面积百分比报告

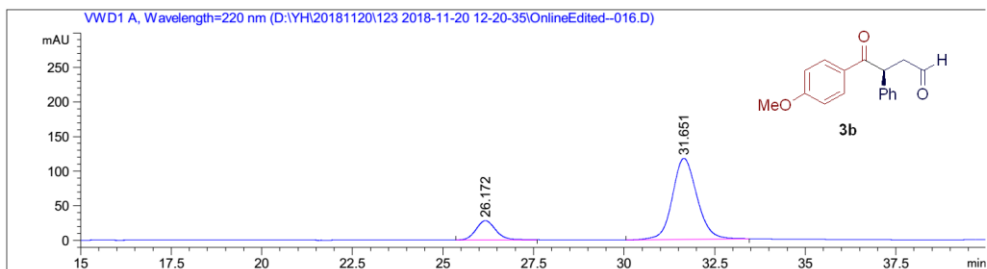
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信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	27.314	VB	0.6784	1.58108e4	351.77167	51.2376
2	33.150	BB	0.8103	1.50469e4	277.17560	48.7624

总量 : 3.08577e4 628.94727

\*\*\* 报告结束 \*\*\*



面积百分比报告

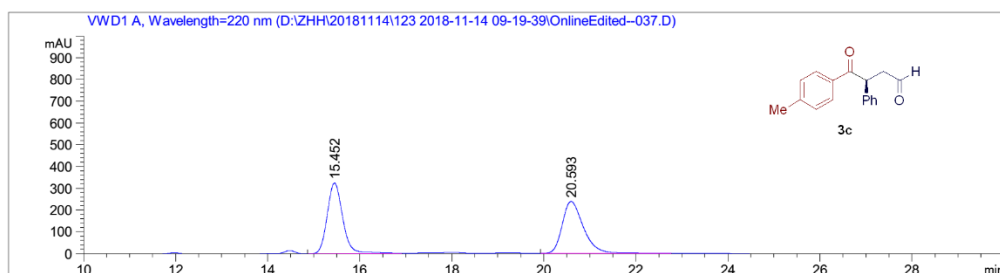
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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	26.172	BB	0.5793	1060.62500	27.85097	16.0627
2	31.651	BB	0.7081	5542.40625	117.33327	83.9373

总量 : 6603.03125 145.18425

\*\*\* 报告结束 \*\*\*



面积百分比报告

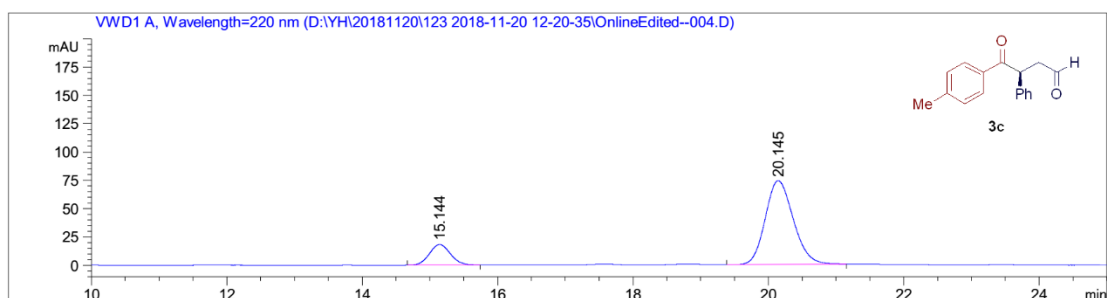
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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.452	VB	0.3621	7602.43799	324.74649	49.9229
2	20.593	VB	0.4837	7625.90967	238.72430	50.0771

总量 : 1.52283e4 563.47079

\*\*\* 报告结束 \*\*\*



面积百分比报告

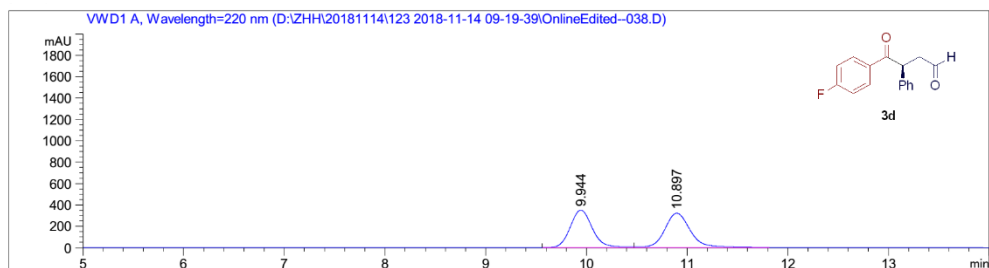
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 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	15.144	BB	0.3339	393.45355	18.15154	15.6096
2	20.145	VB	0.4454	2127.13281	73.98449	84.3904

总量 : 2520.58636 92.13603

\*\*\* 报告结束 \*\*\*



面积百分比报告

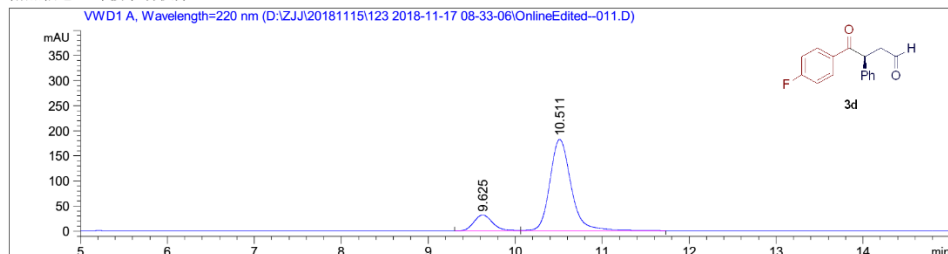
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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.944	VV	0.2376	5370.98584	350.77914	48.4408
2	10.897	VB	0.2720	5716.74902	322.15158	51.5592

总量 : 1.10877e4 672.93073

\*\*\* 报告结束 \*\*\*



面积百分比报告

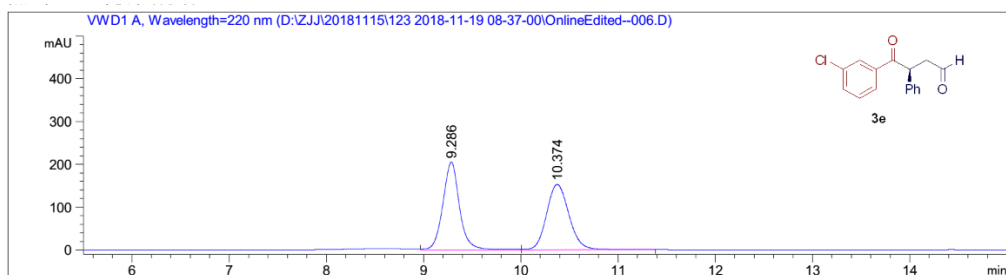
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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.625	BV	0.2327	477.81854	31.72096	13.4006
2	10.511	VB	0.2607	3087.82007	182.19090	86.5994

总量 : 3565.63861 213.91187

\*\*\* 报告结束 \*\*\*



面积百分比报告

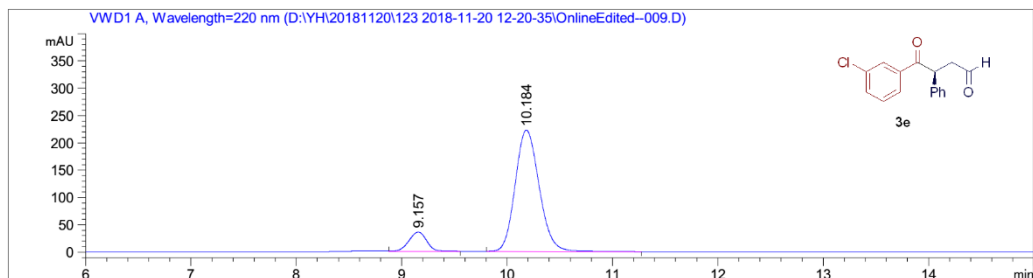
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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.286	VV	0.1792	2441.29517	204.60977	50.0130
2	10.374	VB	0.2499	2440.02783	152.28035	49.9870

总量 : 4881.32300 356.89012

\*\*\* 报告结束 \*\*\*



面积百分比报告

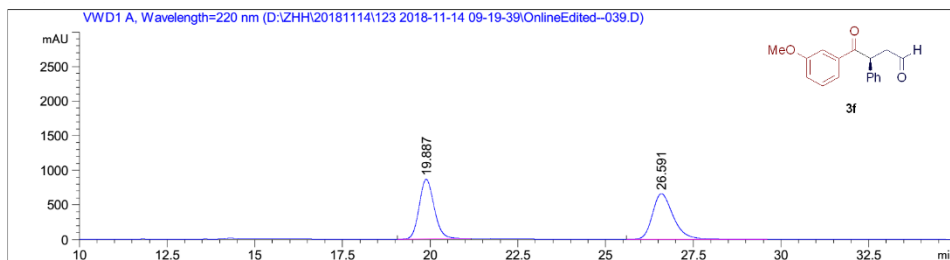
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 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.157	VB	0.1870	417.76932	35.31725	10.6609
2	10.184	BB	0.2460	3500.95313	221.97729	89.3391

总量 : 3918.72244 257.29454

\*\*\* 报告结束 \*\*\*



面积百分比报告

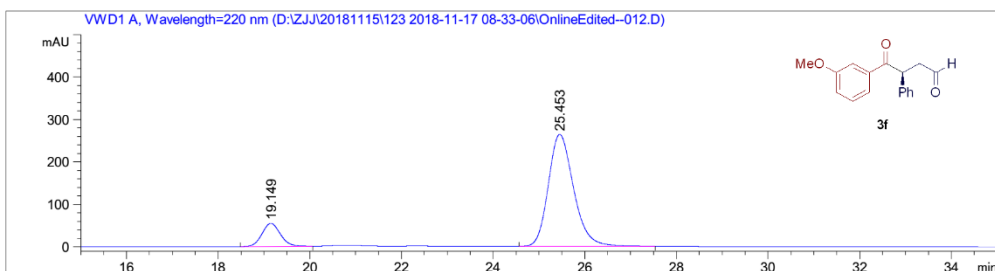
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信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	19.887	VB	0.4576	2.56340e4	862.81165	48.6113
2	26.591	BB	0.6253	2.70986e4	659.60938	51.3887

总量 : 5.27327e4 1522.42102

\*\*\* 报告结束 \*\*\*



面积百分比报告

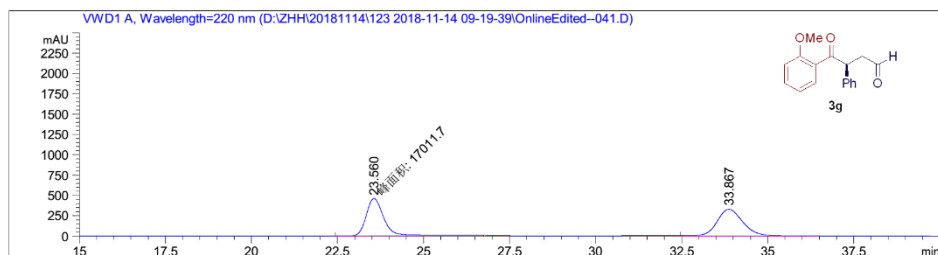
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 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	19.149	BB	0.4366	1523.85132	54.27430	13.1202
2	25.453	BB	0.5813	1.00907e4	263.78049	86.8798

总量 : 1.16146e4 318.05479

\*\*\* 报告结束 \*\*\*



面积百分比报告

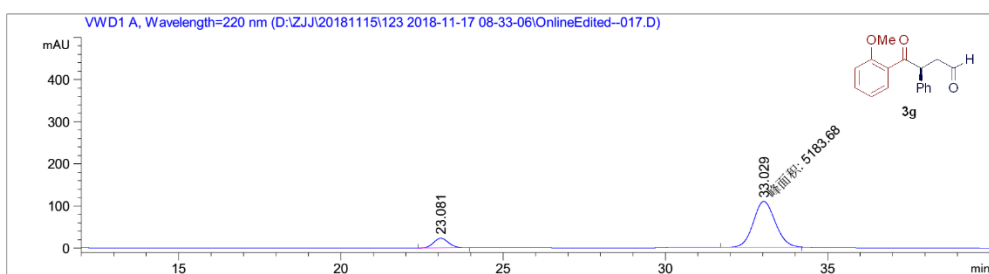
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 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	23.560	MM	0.6166	1.70117e4	459.82669	49.7384
2	33.867	VB	0.7971	1.71906e4	328.75296	50.2616

总量 : 3.42023e4 788.57965

\*\*\* 报告结束 \*\*\*



面积百分比报告

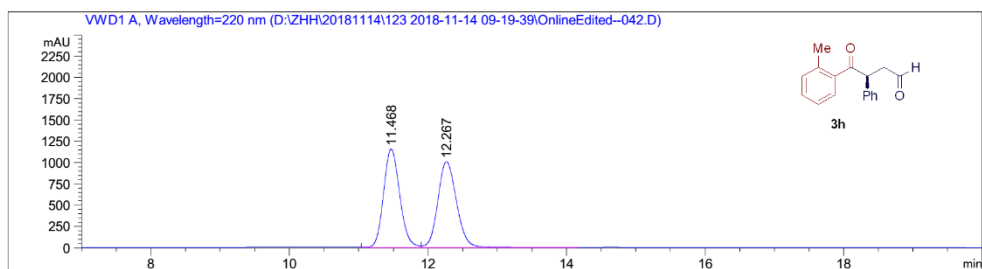
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信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	23.081	BB	0.4761	723.33148	22.79909	12.2453
2	33.029	MF	0.7907	5183.68018	109.26099	87.7547

总量 : 5907.01166 132.06009

\*\*\* 报告结束 \*\*\*



面积百分比报告

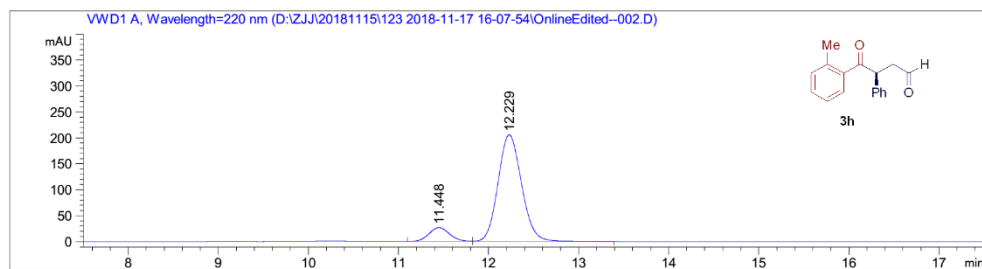
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信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.468	VV	0.2528	1.88340e4	1157.95447	49.7457
2	12.267	VB	0.2936	1.90265e4	1010.65546	50.2543

总量 : 3.78605e4 2168.60992

\*\*\* 报告结束 \*\*\*



面积百分比报告

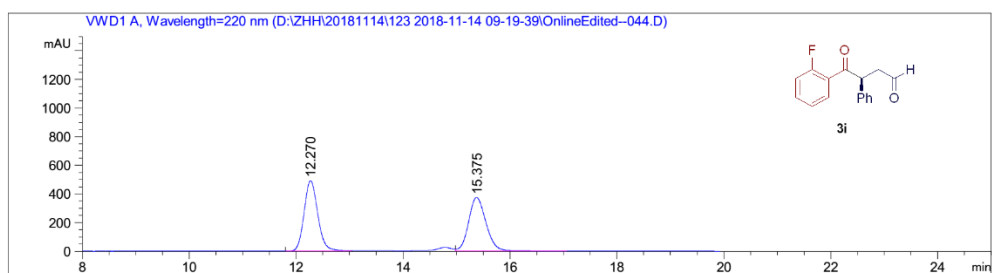
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 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.448	BV	0.2495	423.63541	26.50046	10.1792
2	12.229	VB	0.2842	3738.12012	205.47368	89.8208

总量 : 4161.75552 231.97414

\*\*\* 报告结束 \*\*\*



面积百分比报告

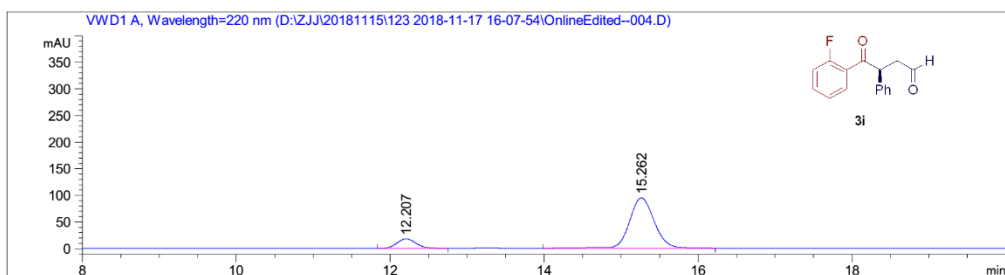
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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	12.270	BV	0.2757	8811.41992	492.49893	50.8321
2	15.375	VB	0.3523	8522.92578	374.84131	49.1679

总量 : 1.73343e4 867.34024

\*\*\* 报告结束 \*\*\*



面积百分比报告

排序 : 信号  
 乘积因子 : 1.0000  
 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

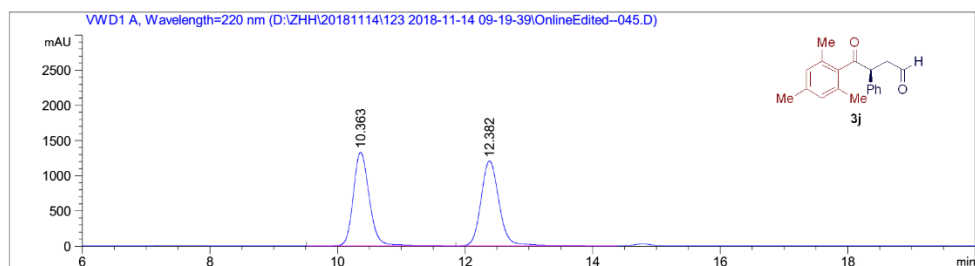
信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	12.207	BB	0.2713	313.14178	17.97318	12.8720
2	15.262	BB	0.3454	2119.59961	94.60391	87.1280

总量 : 2432.74139 112.57709

\*\*\* 报告结束 \*\*\*





面积百分比报告

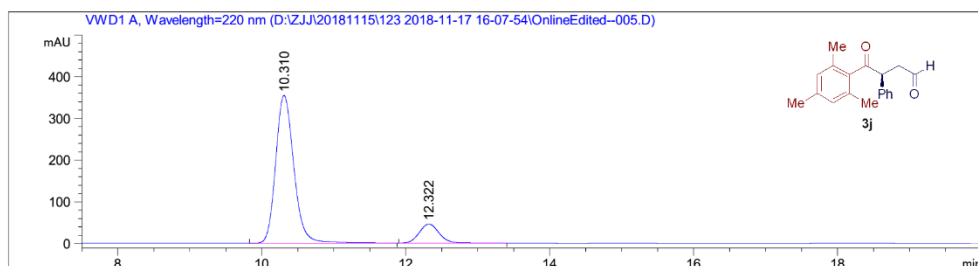
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稀释因子 : 1.0000  
内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.363	VV	0.2685	2.30678e4	1328.99316	49.2711
2	12.382	VB	0.3027	2.37503e4	1206.53027	50.7289

总量 : 4.68181e4 2535.52344

\*\*\* 报告结束 \*\*\*



面积百分比报告

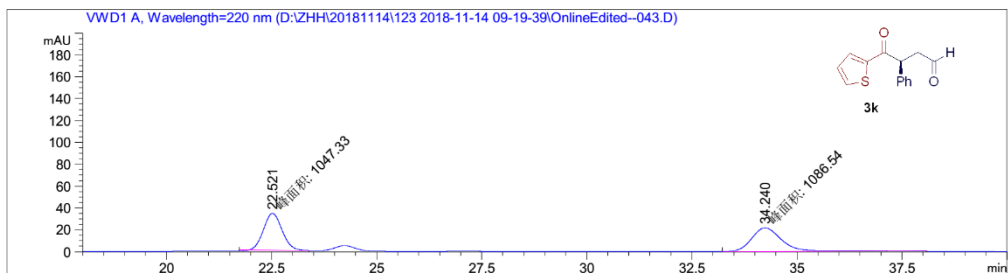
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稀释因子 : 1.0000  
内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.310	BB	0.2751	6265.92188	354.73795	87.3451
2	12.322	BB	0.3037	907.83307	46.10472	12.6549

总量 : 7173.75494 400.84266

\*\*\* 报告结束 \*\*\*



面积百分比报告

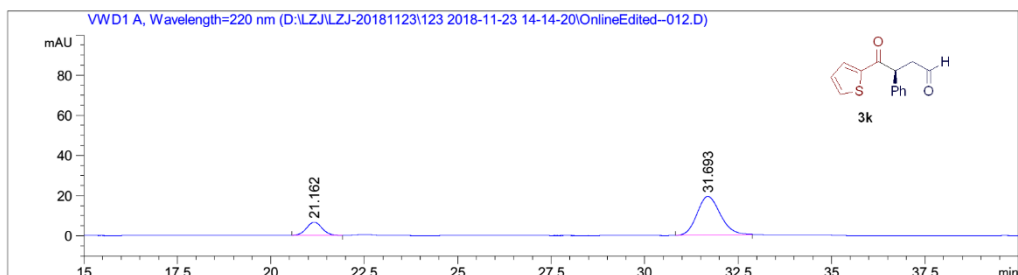
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 稀释因子 : 1.0000  
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信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	22.521	MM	0.5162	1047.33325	33.81378	49.0814
2	34.240	MM	0.8372	1086.53735	21.63116	50.9186

总量 : 2133.87061 55.44494

\*\*\* 报告结束 \*\*\*



面积百分比报告

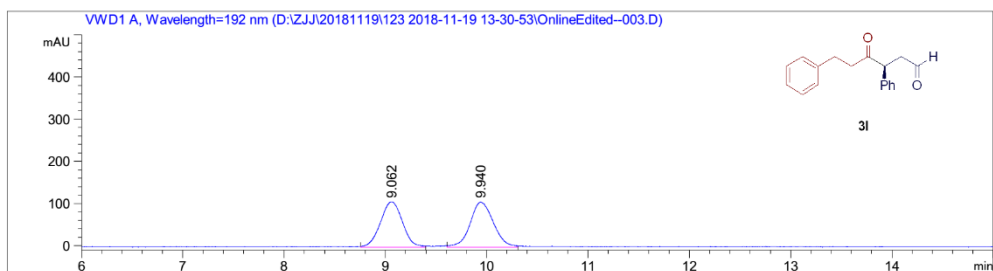
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 稀释因子 : 1.0000  
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信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	21.162	BB	0.3928	190.36230	6.59403	18.6020
2	31.693	BB	0.6243	832.98029	19.23265	81.3980

总量 : 1023.34259 25.82668

\*\*\* 报告结束 \*\*\*



面积百分比报告

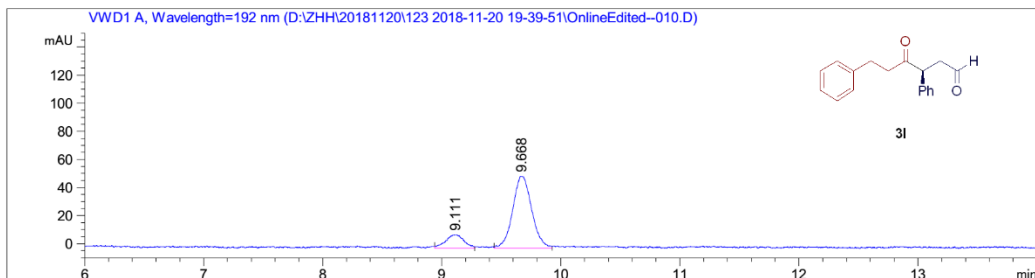
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 稀释因子 : 1.0000  
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信号 1: VWD1 A, Wavelength=192 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.062	VV	0.1882	1616.15564	107.01091	48.7342
2	9.940	VV	0.1983	1700.11230	106.45448	51.2658

总量 : 3316.26794 213.46539

\*\*\* 报告结束 \*\*\*



面积百分比报告

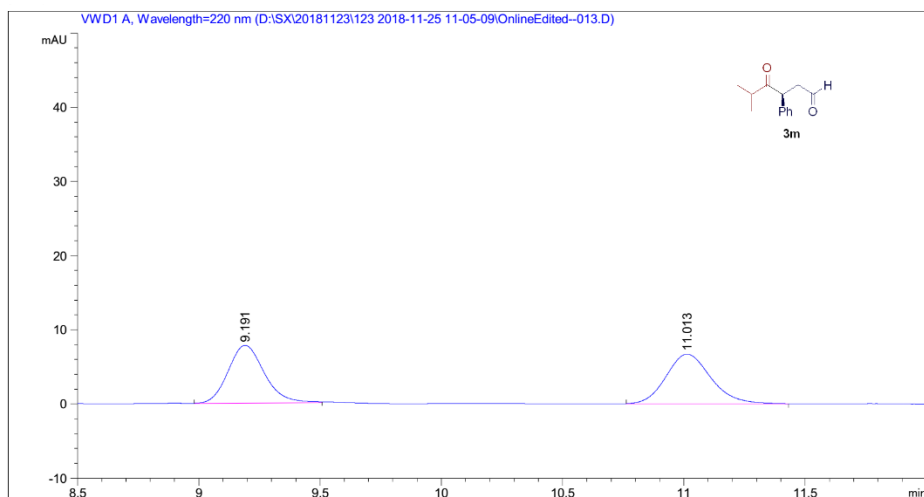
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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=192 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.111	VV	0.1281	103.87579	9.69115	15.3940
2	9.668	VV	0.1370	570.90344	51.21814	84.6060

总量 : 674.77924 60.90929

\*\*\* 报告结束 \*\*\*

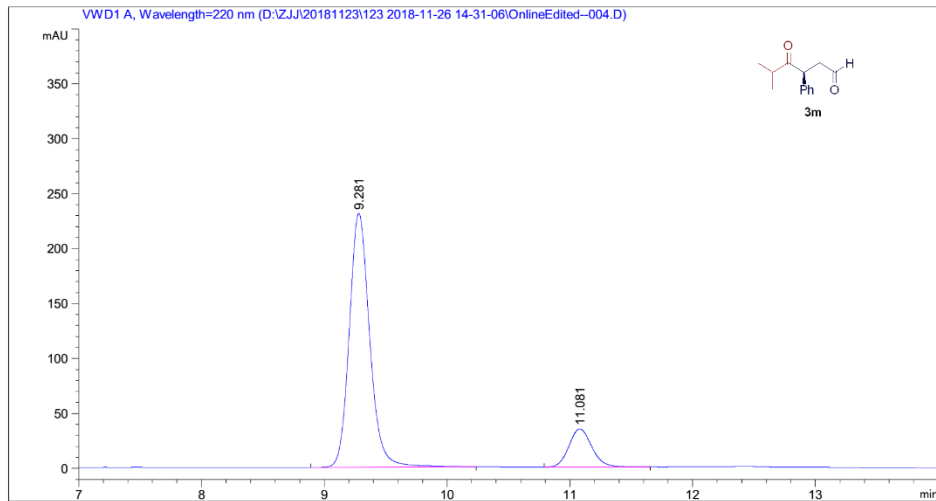


面积百分比报告

排序 : 信号  
 乘积因子 : 1.0000  
 稀释因子 : 1.0000  
 内标使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.191	BB	0.1603	80.97955	7.78597	48.3766
2	11.013	BB	0.1966	86.41460	6.69563	51.6234

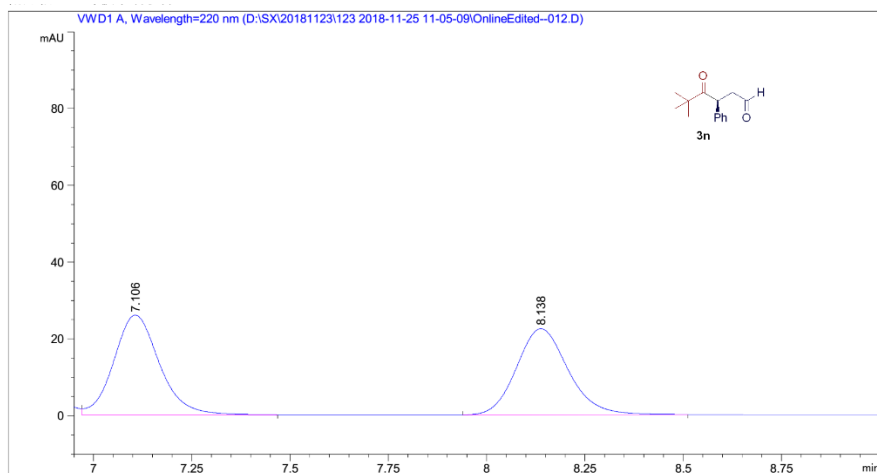


面积百分比报告

排序 : 信号  
 乘积因子 : 1.0000  
 稀释因子 : 1.0000  
 内标使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.281	BB	0.1773	2683.26318	231.31868	85.5935
2	11.081	BB	0.2016	451.62793	34.76586	14.4065

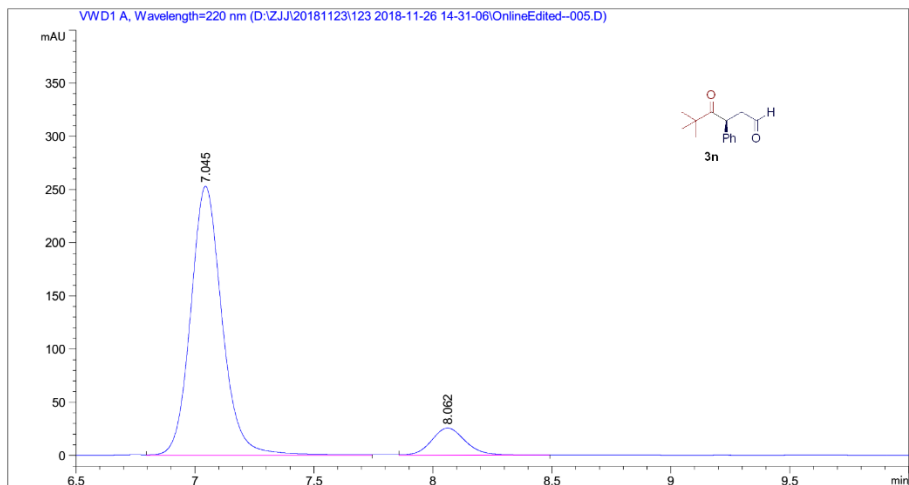


面积百分比报告

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 内标使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	7.106	VB	0.1237	211.32396	26.04159	50.6822
2	8.138	BB	0.1409	205.63533	22.44261	49.3178



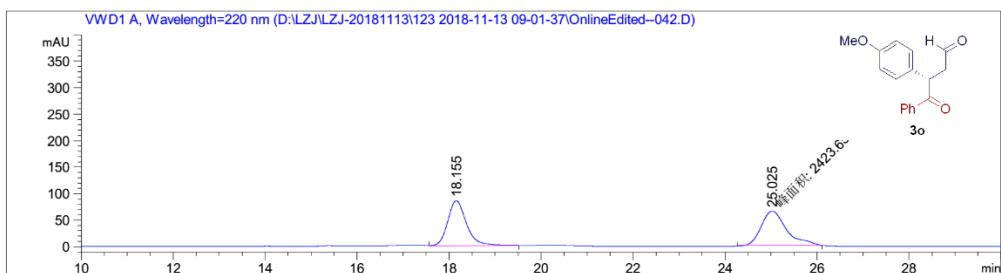
面积百分比报告

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 内标使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	7.045	VB	0.1402	2325.55078	252.99445	90.1513
2	8.062	BB	0.1531	254.05759	25.53905	9.8487

总量 : 2579.60837 278.53349



面积百分比报告

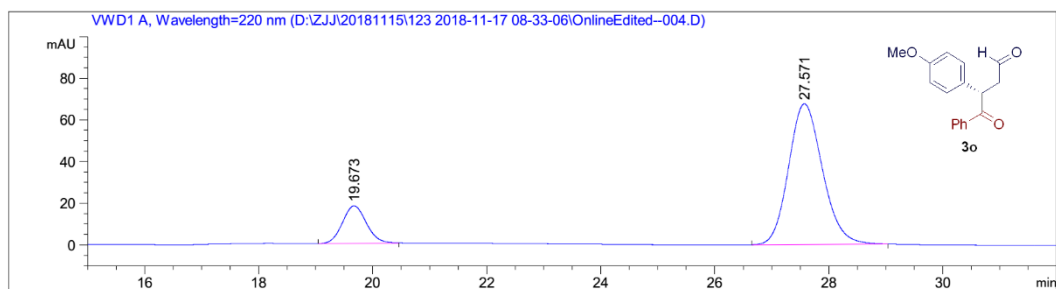
排序 : 信号  
 乘积因子 : 1.0000  
 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	18.155	VB	0.4287	2394.78003	84.99420	49.7004
2	25.025	MM	0.6263	2423.64990	64.49909	50.2996

总量 : 4818.42993 149.49329

\*\*\* 报告结束 \*\*\*



面积百分比报告

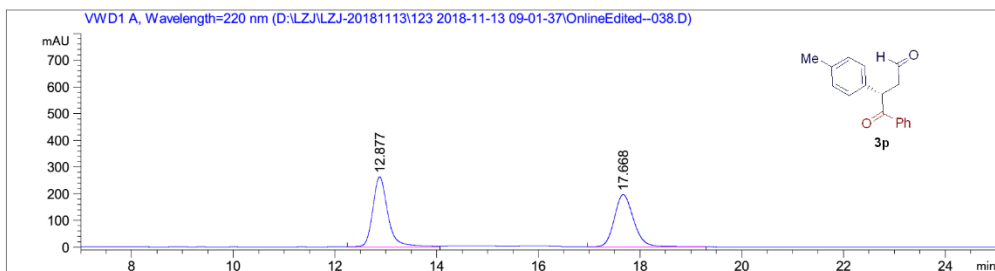
排序 : 信号  
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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	19.673	BB	0.4463	524.18146	17.97072	15.7977
2	27.571	BB	0.6404	2793.90991	67.56318	84.2023

总量 : 3318.09137 85.53390

\*\*\* 报告结束 \*\*\*



面积百分比报告

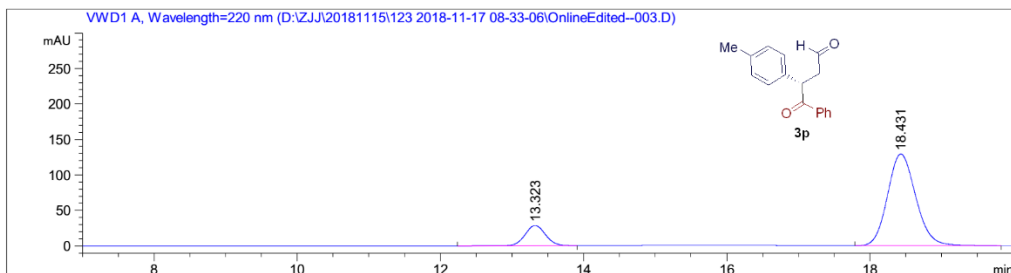
排序 : 信号  
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 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	12.877	VB	0.3170	5465.90234	262.44299	51.2968
2	17.668	BB	0.4079	5189.55273	195.99489	48.7032

总量 : 1.06555e4 458.43788

\*\*\* 报告结束 \*\*\*



面积百分比报告

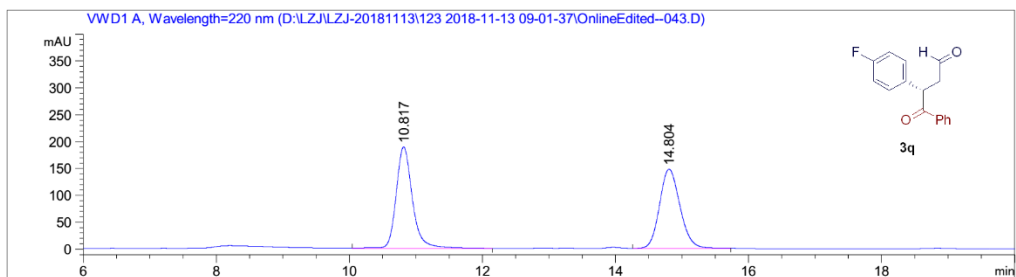
排序 : 信号  
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 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	13.323	BB	0.3161	584.35571	28.63651	14.1316
2	18.431	BB	0.4284	3550.74902	129.27928	85.8684

总量 : 4135.10474 157.91580

\*\*\* 报告结束 \*\*\*



面积百分比报告

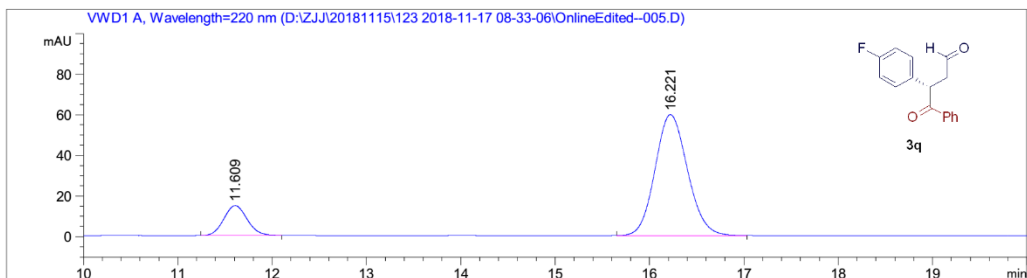
排序 : 信号  
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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.817	VB	0.2606	3227.22998	188.67720	50.6460
2	14.804	VB	0.3290	3144.90771	147.36853	49.3540

总量 : 6372.13770 336.04573

\*\*\* 报告结束 \*\*\*



面积百分比报告

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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

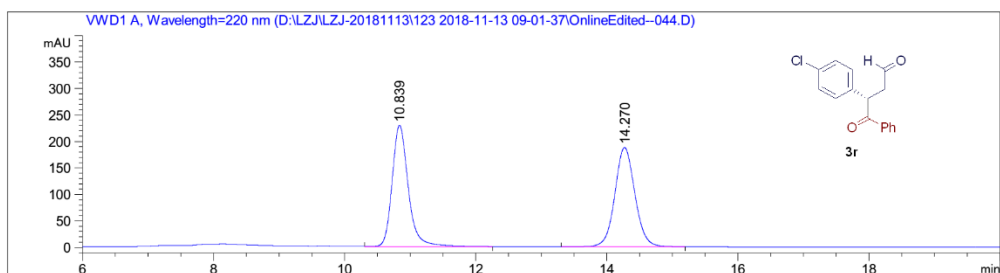
信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.609	BB	0.2674	252.55983	14.70080	15.0582
2	16.221	BB	0.3701	1424.66809	59.56138	84.9418

总量 : 1677.22792 74.26218

\*\*\* 报告结束 \*\*\*





面积百分比报告

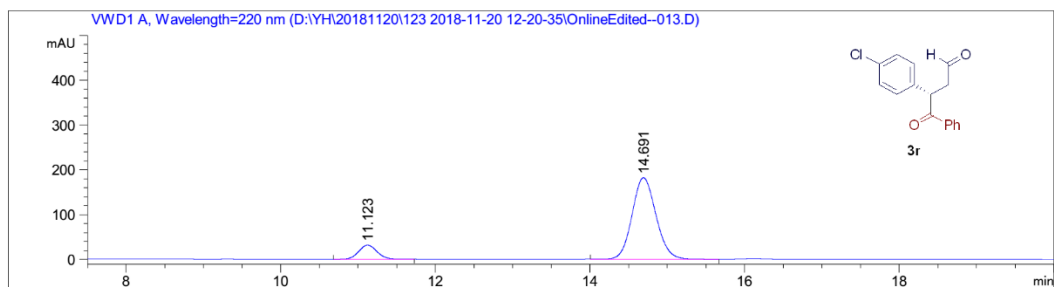
排序 : 信号  
 乘积因子 : 1.0000  
 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.839	VB	0.2622	3954.31177	229.32411	49.8020
2	14.270	BB	0.3273	3985.75781	187.29362	50.1980

总量 : 7940.06958 416.61774

\*\*\* 报告结束 \*\*\*



面积百分比报告

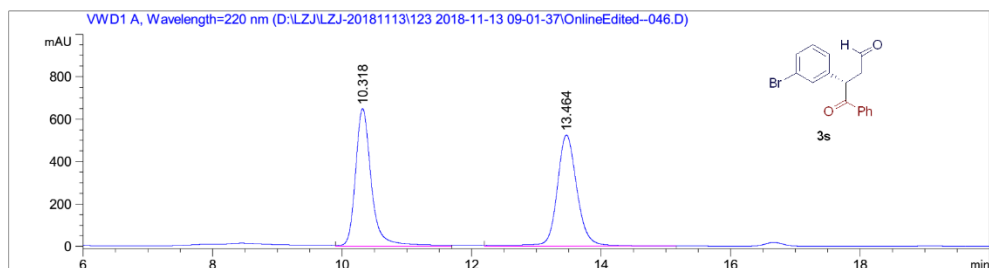
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 乘积因子 : 1.0000  
 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.123	BB	0.2563	521.82898	31.50201	11.7811
2	14.691	BB	0.3358	3907.53369	181.78241	88.2189

总量 : 4429.36267 213.28442

\*\*\* 报告结束 \*\*\*



面积百分比报告

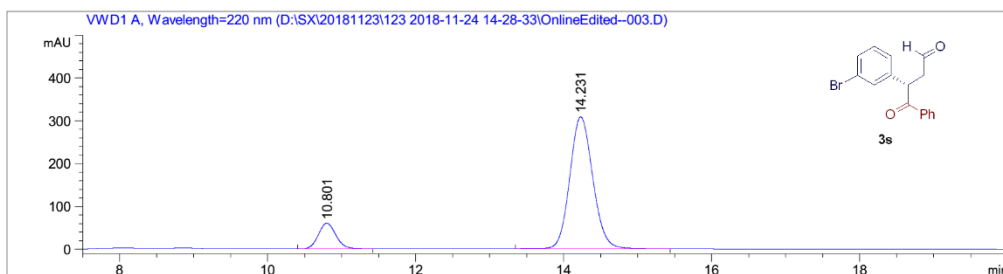
排序 : 信号  
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信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.318	VB	0.2596	1.10947e4	648.37738	49.7446
2	13.464	VB	0.3268	1.12087e4	523.50037	50.2554

总量 : 2.23034e4 1171.87775

\*\*\* 报告结束 \*\*\*



面积百分比报告

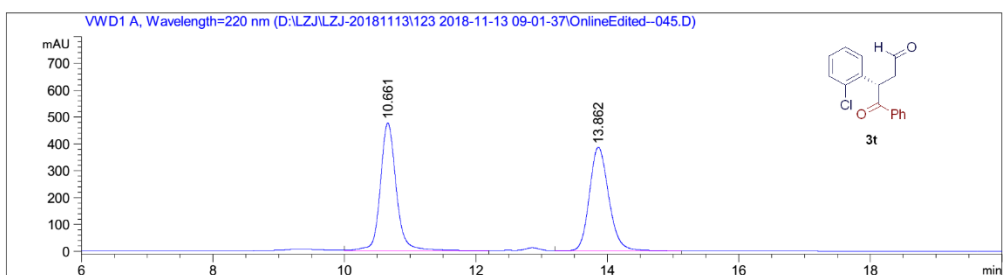
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 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.801	BB	0.2557	982.13690	59.77727	12.8879
2	14.231	BB	0.3336	6638.45654	307.82010	87.1121

总量 : 7620.59344 367.59737

\*\*\* 报告结束 \*\*\*



面积百分比报告

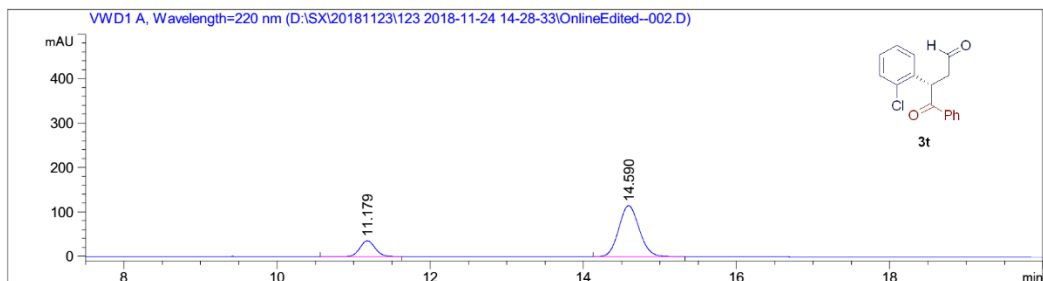
排序 : 信号  
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内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.661	VB	0.2610	8178.63086	477.16788	50.5841
2	13.862	VB	0.3179	7989.73828	386.97763	49.4159

总量 : 1.61684e4 864.14551

\*\*\* 报告结束 \*\*\*



面积百分比报告

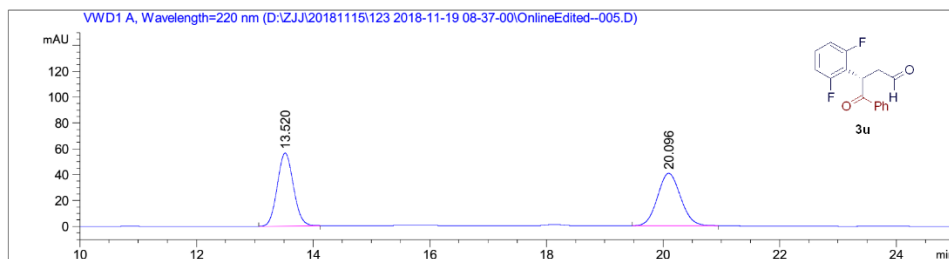
排序 : 信号  
乘积因子 : 1.0000  
稀释因子 : 1.0000  
内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.179	BB	0.2145	493.19757	35.62347	18.7776
2	14.590	BB	0.2887	2133.31641	114.84486	81.2224

总量 : 2626.51398 150.46834

\*\*\* 报告结束 \*\*\*



面积百分比报告

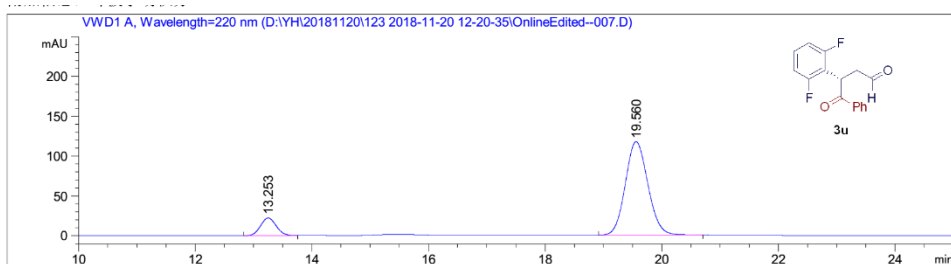
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 稀释因子 : 1.0000  
 内标中不使用乘积因子和稀释因子

信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	13.520	BB	0.2998	1082.82825	56.43952	49.2784
2	20.096	BB	0.4255	1114.54089	40.56545	50.7216

总量 : 2197.36914 97.00497

\*\*\* 报告结束 \*\*\*



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信号 1: VWD1 A, Wavelength=220 nm

峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	13.253	BB	0.2952	419.58060	22.23423	11.7459
2	19.560	BB	0.4209	3152.57861	117.18459	88.2541

总量 : 3572.15921 139.41882

\*\*\* 报告结束 \*\*\*